A New PEG GC Column with Improved Inertness Reliability and Column Lifetime

Agilent J&W DB-WAX Ultra Inert Polyethylene Glycol Column

Competitive Comparison

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Introduction

Current demanding GC and GC/MS applications have an increased focus on sensitive and reproducible qualitative and quantitative analysis of more challenging active analytes. An inert GC flow path is required to prevent the adsorption of the active compounds of interest onto active sites anywhere from injection to detection. Building on our experience developing enhanced surface deactivation chemistries [1], Agilent has successfully improved the inertness of the entire GC flow path. The flow path is directly in contact with critical, active analytes that are susceptible to break down or otherwise inferior chromatographic detectability. Enhancing the flow path deactivation has made significant improvement in the sensitivity and reproducibility of measurements using Ultra Inert (UI) deactivated products. These include Agilent Ultra Inert apolar and midpolar columns [2,3,4], Agilent Ultra Inert liners [3,4], Agilent Ultra Inert Gold Seals, split/splitless top, shell weldments [5], and Agilent Ultimate Plus deactivated fused silica tubing [6]. The knowledge and experience gained developing these products led to the development of an Ultra Inert polar, "WAX column" based on 100% polyethylene glycol (PEG) stationary phase. Agilent J&W WAX type columns have been the industry standard over the last 35 years. These columns are used to analyze a wide variety of compounds with polar functional groups, saturated compounds in flavors and fragrances, and industrial chemical quality control. In this competitive comparison, we present the Agilent DB-WAX Ultra Inert GC column.

DB-WAX UI has been developed and tested through a rigorous inertness testing procedure using the most active probes in today's demanding applications. Performance of DB-WAX UI was evaluated based on a combination of three different perspectives:

- **Inertness performance**: This is evaluated using more demanding test mixes that enable greater scrutiny of column activity. In addition, inertness stability is also investigated using a thermal longevity test at 250 °C, the column's upper temperature limit.
- **Column-to-column consistency**: A set of 20 DB-WAX UI columns were randomly selected from different batches and tested for inertness consistency from column to column.
- **Selectivity testing**: A combination of retention indexes of certain compounds was monitored in QC tests to verify that the same selectivity is achieved between DB-WAX and DB-WAX UI. Retaining the same selectivity as DB-WAX is important for customers who have used the standard DB-WAX for many years [7]. It makes upgrading to the Ultra Inert version easy with minimal revalidation. There is no need to recreate or modify existing compound libraries and methods that are based on DB-WAX.

A benchmarking study was also performed to compare inertness between DB-WAX UI and various non-Agilent WAX columns on the market. All experimental conditions except the columns were kept constant to provide as fair a comparison as possible among the columns. Overall, the DB-WAX UI had superior performance for inertness, thermal stability at the column's upper temperature limit, and column-to-column consistency, thus ensuring the most sensitive and reproducible analytical results for analysis of more critical active analytes.

Results and Discussion

Test methods and standards

QC test probes play a key role in the adequate evaluation of column inertness and column-to-column consistency. Highly active analytes have been known to absorb onto active sites of the column. Therefore, the composition and amount on-column of these probes must be carefully selected to allow sufficient detection of important column activity [8]. An easy QC test mix containing undemanding probes results in poor inertness evaluation because column activity may be insufficiently recognized. Testing WAX columns using demanding test probes ensures consistent column inertness performance. This demanding testing ultimately contributes to improvement in column-to-column consistency and reliability of analytical results. A detailed guideline for choosing suitable and effective QC test probes to evaluate column inertness performance can be found in *Agilent J&W Ultra Inert GC Columns: A New Tool to Battle Challenging Active Analytes*, Technical Overview [2].

Following this guideline, new and demanding test probe mixes were developed for critical assessment of the inertness performance of DB-WAX UI columns. More challenging test probes such as 2-ethylhexanoic acid, ethyl maltol, dicyclohexylamine, 2,3-butanediol, and decanal are present at critical levels. The inertness of the WAX columns was evaluated based on peak shapes of the active analytes of interest.

These analytes are highlighted in two test mixes (DB-WAX UI and a modified Grob mix [9]) shown in Tables 1 and 2. The influence of other components in the GC flow path are minimized using Agilent Ultra Inert liners and Ultra Inert Gold Seals, increasing confidence in injection port inertness during the evaluation of the columns.

Table 1. Agilent DB-WAX UI test mix, in dichloromethane.

Peak no.	Agilent DB-WAX UI	Amount on column (ng)
1	5-Nonanone	3.3
2	Decanal	3.3
3	Propionic acid	3.3
4	Ethylene glycol	3.3
5	Heptadecane	1.65
6	Aniline	3.3
7	Methyl dodecanoate	3.3
8	2-chlorophenol	3.3
9	1-Undecanol	3.3
10	Nonadecane	1.65
11	2-Ethylhexanoic acid	6.6
12	Ethyl maltol	6.6

Analytical conditions for Agilent DB-WAX UI test mix evaluations	
Injector temp:	250 °C
Split:	1:75
Injection volume:	1 µL
Carrier gas flow rate:	1.1 mL/min, H_2
Oven temp:	130 °C isothermal
FID detector:	260 °C

Table 2. Modified Grob test mix, in dichloromethane

Compound	Modified Grob	Amount on column (ng)
1	Decane	2.5
2	Dodecane	2.5
3	Decanal	2.5
4	2,3-Butanediol*	**5**
5	1-Octanol	2.5
6	C10 FAME	2.5
7	*n*C11-FAME	2.5
8	Dicyclohexylamine	**5**
9	*n*C12-FAME	2.5
10	2,6-Dimethylaniline	2.5
11	2,6-Dimethylphenol	2.5
12	2-Ethylhexanoic acid	5
13	Ethyl maltol	5

*2,3-Butanediol is present at two isomers, RR/SS and meso isomers, respectively.

Analytical conditions for modified Grob test mix	
Injector temp:	250 °C
Split:	1:100
Injection volume:	1 µL
Carrier gas flow rate:	1.35 mL/min, H_2
Oven temp:	Initial temp. 60 °C, Ramp 3 °C/min, Final temp. 200 °C
FID detector:	260 °C.

The DB-WAX UI test mix was created by adding active analytes at critical levels to the current QC test mix of the classic Agilent J&W DB-WAX column. This ensured efficient detection of column activity. The analytes decanal, propionic acid, ethylene glycol, 2-ethylhexanoic acid, and ethyl maltol (highlighted in Table 1) were added. In addition to the DB-WAX UI test mix, a modified Grob test mix containing compounds with various chemical properties was used for extra inertness testing. The additional active probes in this test mix (highlighted in Table 2) are representative compounds of several analyte groups seen in flavor and fragrance applications, where standard DB-WAX columns have been used over 35 years.

Inertness performance

Figure 1 shows representative FID chromatograms of the DB-WAX UI test mix on standard DB-WAX and DB-WAX UI columns after conditioning for one hour at 250 °C. The column activity of the standard DB-WAX column was characterized by tailing peaks and loss of responses of critical analytes of interest, such as propionic acid (peak 3), 2-ethylhexanoic acid (peak 11), and ethyl maltol (peak 12). Good peak shapes and significant improvement in responses for these active probes were obtained with the DB-WAX UI column. These results indicate the excellent inertness performance of the DB-WAX UI column.

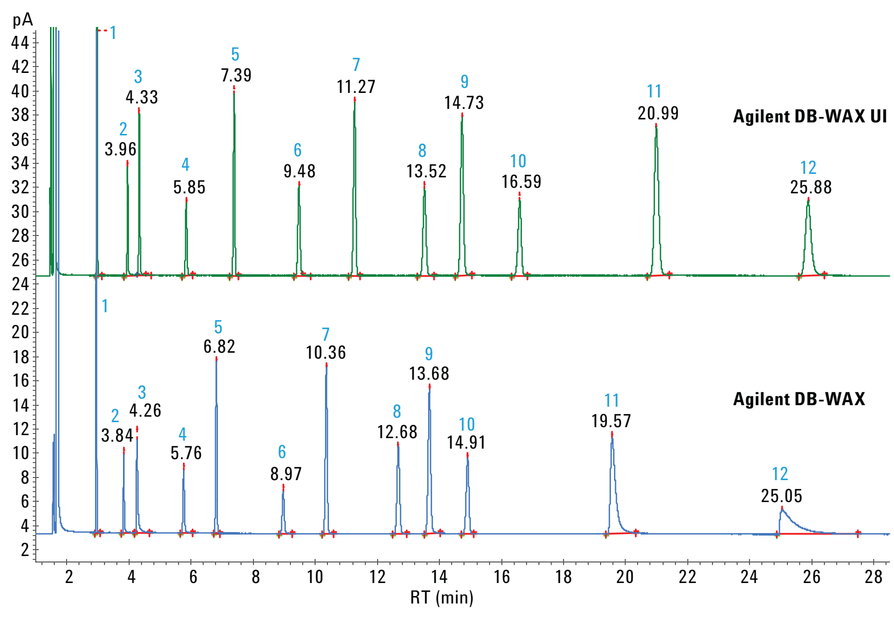

Figure 1. Example FID chromatograms of the Agilent DB-WAX UI test mix on standard Agilent DB-WAX and Agilent DB-WAX UI columns after conditioning for one hour at 250 °C. See Table 1 for GC conditions and peak identification.

Longevity testing at 250 °C was also performed on the DB-WAX UI column to evaluate inertness stability at the column's upper temperature limit. An extended period of 50 hours conditioning at 250 °C was carried out. QC tests were performed every five hours of conditioning at 250 °C. Figure 2 shows example FID chromatograms of the modified Grob test mix on a DB-WAX UI column after conditioning for one hour, and then after 50 hours at 250 °C. The good peak shapes and responses of decanal (peak 3), 2,3-butanediol (peaks 4a and 4b), dicyclohexylamine (peak 8), 2-ethyhexnoic acid (peak 12), and ethyl maltol (peak 13) in this test mix indicate the excellent inertness performance of this column. Good inertness was maintained even after long-term thermal exposure at the upper temperature limit of 250 °C. These results show the superior inertness stability of the DB-WAX UI column, delivering consistent analytical results, and enhancing column lifetime.

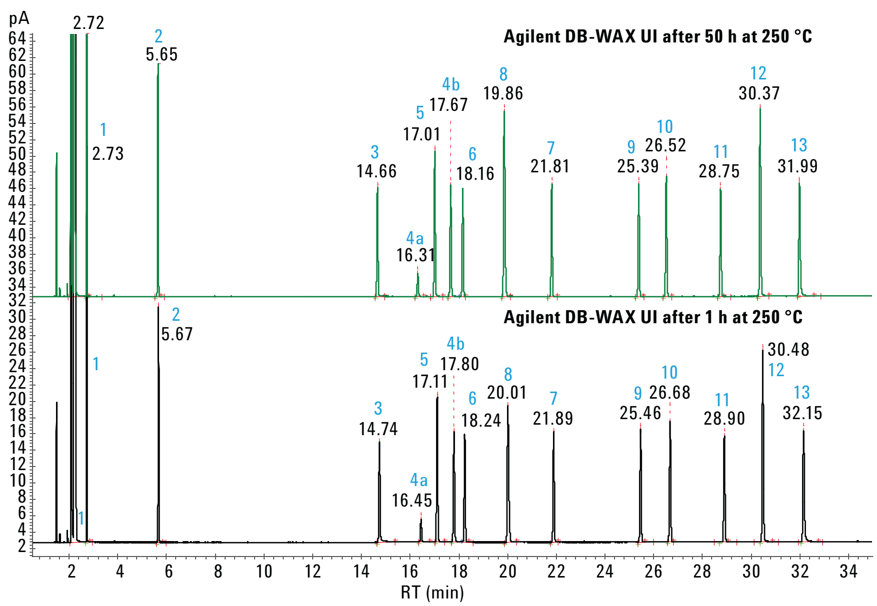

Figure 2. Example FID chromatograms of the modified Grob test mix on an Agilent DB-WAX UI column after conditioning for one hour, and then 50 hours at 250 °C. See Table 2 for GC conditions and peak identification.

Column-to-column consistency is a key requirement for reproducible qualitative and quantitative analyses. A set of 20 DB-WAX UI columns from different batches were randomly selected to evaluate the column inertness consistency. Peak asymmetry at 10% peak height was used to evaluate the peak shapes of active compounds such as propionic acid, decanal, and 2,3-butanediol. Figure 3 shows good column-to-column inertness consistency illustrated by the small variation in peak asymmetry at 10% peak height of these active compounds on 20 random DB-WAX UI columns.

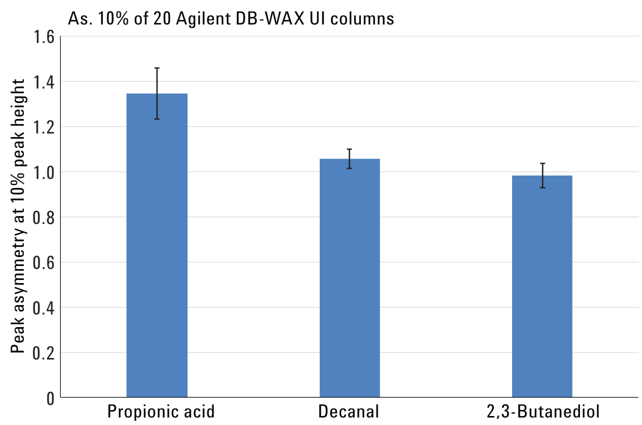

Figure 3. Peak asymmetry at 10% peak height (As. 10%) of propionic acid, decanal, and 2,3-butanediol on 20 Agilent DB-WAX UI columns randomly selected from different batches. QC tests were performed after conditioning for 11 hours at 250 °C.

Identical selectivity

In addition to inertness, selectivity is an important column evaluation parameter. Standard DB-WAX columns have been routinely used in many applications. Similar selectivity between DB-WAX and DB-WAX UI is an important advantage. It ensures an easy, fast, and simple column upgrade, with minimal method revalidation, thus avoiding the risks that come with having to recreate or modify existing compound libraries or analytical methods already based on DB-WAX. Column selectivity is often determined using a combination of retention indexes of some target compounds in QC test mix. Retention indexes of DB-WAX UI columns were measured and compared to current specifications for DB-WAX columns [7] (data not shown). The results indicate no significant differences in retention indexes between DB-WAX UI and DB-WAX columns, thus verifying the same selectivity between the two columns. In addition, the same selectivity between these two columns was also demonstrated in the analysis of fatty acid methyl esters (FAMEs) shown in Figure 4. Here, a DB-WAX UI column is shown to be identical to the DB-WAX, following the parameters established in a retention time locked method for FAMEs and the DB-WAX column [10]. The same selectivity between the two columns was also demonstrated in the analysis of lavender essential oil [11].

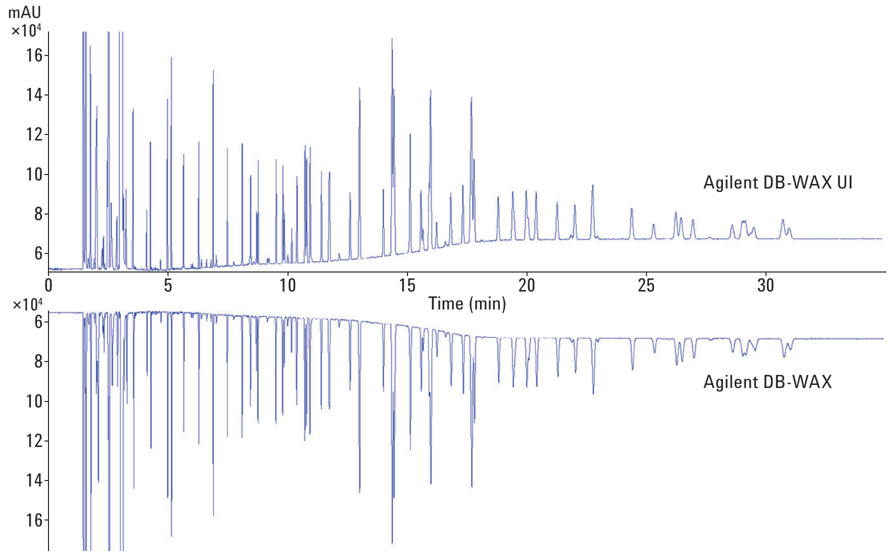

Figure 4. Example FID chromatograms of extended FAMEs Mix of 72 compounds retention time locked on Agilent DB-WAX UI and Agilent DB-WAX columns.

Test conditions	
Column:	Agilent DB-WAX UI, 30 m × 0.25 mm, 0.25 µm (p/n 122-7032UI) or Agilent DB-WAX (p/n 122-7032)
Inlet:	Inert Flow Path Split/splitless weldment (p/n G3970A)
Detector:	FID
Inlet temperature:	250 °C
Injection volume:	1 µL
Split ratio:	1/50
Carrier gas:	Hydrogen
Head pressure:	Methyl stearate is retention time locked to 14.000 min. Constant pressure mode (pressure approximately 53 kPa at 50 °C cm/s, 36 cm/s at 50 °C)
Oven temperature:	50 °C, 1 min, 25 °C/min to 200 °C, 3 °C/min to 230 °C, 18 min hold
Detector temperature:	280 °C
Detector gases:	Hydrogen: 40 mL/min Air: 450 mL/min Helium make-up gas: 30 mL/min
Flow path supplies:	Ultra Inert Low-Pressure drop liner (p/n 5190–2295) UI Gold seal (p/n 5190–6144) Self-tightening column nut (p/n 5190–6194) Graphite-vespel ferrules (p/n 5181–3323 10Pk) Long life septa (p/n 5183–4761)

Benchmarking research

A benchmarking study was performed to compare the inertness performance and thermal stability between DB-WAX UI and selected WAX columns from various column suppliers. Selected WAX columns were Stabilwax, Stabilwax-MS, and ZB-WAXplus. Inertness testing was performed after the columns were conditioned for one hour at 250 °C, using both the DB-WAX UI and modified Grob test mixes. A longevity test at 250 °C for 50 hours tested for column inertness versus modest thermal stress at each column's upper temperature limit. QC tests were performed after each five hours of conditioning at 250 °C. All other analytical conditions were kept constant to provide a fair comparison.

Stabilwax and Stabilwax-MS share a similar inertness performance for propionic acid, 2-ethylhexanoic acid, and ethyl maltol. Peak tailing for these active analytes was also observed for ZB-WAXplus columns using the DB-WAX UI test mix. Figure 5 shows FID example chromatograms of Stabilwax, Stabilwax-MS, and ZB-WAXplus columns compared to DB-WAX UI with one hour of conditioning at 250 °C. In addition, the inertness of these columns rapidly deteriorated during the longevity test at 250 °C. Figure 6 shows FID example chromatograms of these columns compared to DB-WAX UI after 50 hours conditioning at 250 °C using the DB-WAX UI test mix.

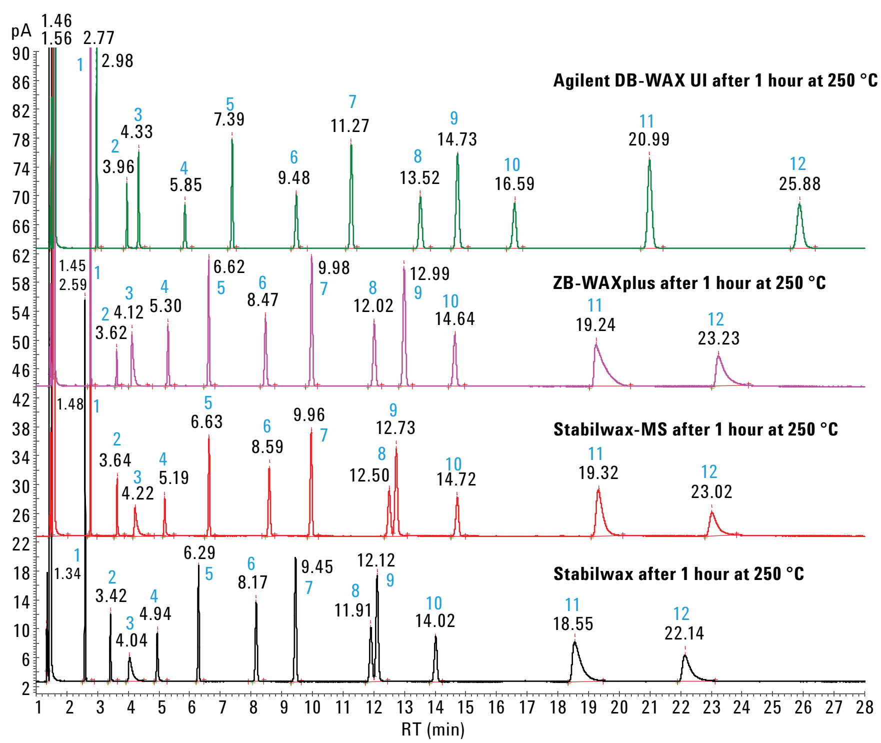

Figure 5. Example FID chromatograms of the Agilent DB-WAX UI test mix on an Agilent DB-WAX UI, Stabilwax, Stabilwax-MS, and ZB-WAXplus columns after conditioning for one hour at 250 °C. See Table 1 for GC conditions and peak identification.

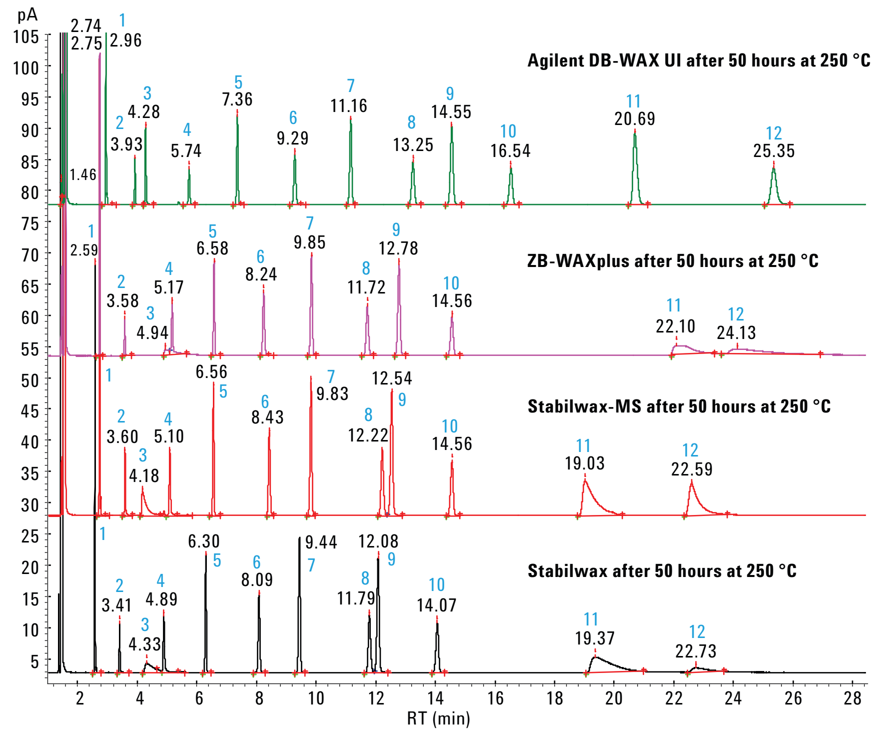

Figure 6. Example FID chromatograms of the Agilent DB-WAX UI test mix on an Agilent DB-WAX UI, Stabilwax, Stabilwax-MS, and ZB-WAXplus columns after conditioning for 50 hours at 250 °C. See Table 1 for GC conditions and peak identification.

Table 3 shows a summary of the inertness performance comparison for DB-WAX UI, Stabilwax, Stabilwax-MS, and ZB-WAXplus using peak asymmetry measured at 10% of peak height of most active probes in the modified Grob test mix. QC test results are one hour at 250 °C (Table 3A) and 50 hours at 250 °C (Table 3B).

Table 3. Summary of peak asymmetry values measured at 10% of peak height on Agilent DB-WAX UI, Stabilwax, Stabilwax-MS, and ZB-WAXplus columns using the modified Grob test mix. Peak asymmetry of active analytes was ranged in three colors: green, yellow, and red.

A. 1 hour at 250 °C: Green: As (10%) ≤ 1.5; Yellow: 1.5 < As (10%) ≤ 2; Red: As (10%) >2

Column	2-Ethylhexanoic acid	2,3-Butanediol	Ethyl maltol	Decanal	Dicyclohexylamine
DB-WAX UI					
Stabilwax					
Stabilwax-MS					
ZB-WAXplus					

B. 50 hours at 250 °C: Green: As (10%) ≤ 1.5; Yellow: 1.5 < As (10%) ≤ 2; Red: As (10%) >2

Column	2-Ethylhexanoic acid	2,3-Butanediol	Ethyl Maltol	Decanal	Dicyclohexylamine
DB-WAX UI					
Stabilwax					
Stabilwax-MS					
ZB-WAXplus					

Strongly active analytes normally give lower peak heights and responses because of their adsorption onto the active sites throughout the flow path from injector to detector. In this test, all analytical conditions, except the columns, were kept constant. Therefore, the differences in peak heights or responses of critical probes can be attributed to the inertness of the columns tested. Figure 7 shows peak height ratios between some active compounds and an inert analyte (n-C19) in the DB-WAX UI test mix on DB-WAX UI, Stabilwax, Stabilwax-MS, and ZB-WAXplus columns. Peak height ratios on DB-WAX UI were higher than those ratios on other WAX columns for propionic acid, 2-ethylhexanoic acid, and ethyl maltol at both one and 50 hours of conditioning at 250 °C. These results are strong evidence of the increased inertness of the DB-WAX UI compared to other WAX columns, with clear improvement in the peak height ratios of highly active analytes relative to an inert alkane. As a result, a significant improvement of sensitivity was achieved. Figure 7 also shows that the peak height ratios of these probes are stable after 50 hours at 250 °C on the DB-WAX UI column. This result strongly indicates excellent thermal stability at the DB-WAX UI column's upper temperature limit, providing great improvements in delivering consistent analytical results and column lifetimes. In contrast, a significant reduction in those peak height ratios after 50 hours of conditioning at 250 °C was observed for Stabilwax-MS and ZB-WAXplus columns. As a result, approximately two times loss in sensitivity is observed for Stabilwax-MS, and more than four times for ZB-WAXplus columns.

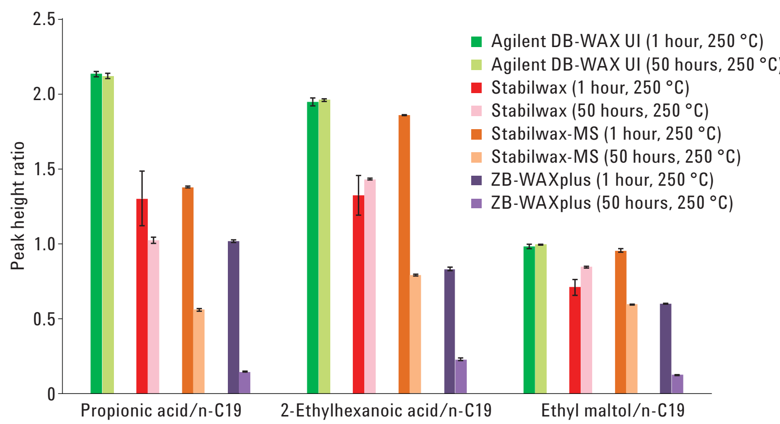

Figure 7. Peak height ratios between active compounds propionic acid, 2-ethylhexanoic acid, ethyl maltol, and an inert compound (n-C19) in the Agilent DB-WAX UI test mix on Agilent DB-WAX UI, StabilWAX, Stabilwax-MS, and ZB-WAXplus columns.

Inertness evaluations were performed on three recently introduced WAX columns from brands, seen here as G, TK, and M. Inertness performance and thermal longevity test at 250 °C were carried out using the DB-WAX UI and modified Grob test mixes in the manner described before.

The brand G column exhibits peak tailing of 2,3-butanediol (peaks 4a and 4b) using the modified Grob test mix shown in Figure 8.

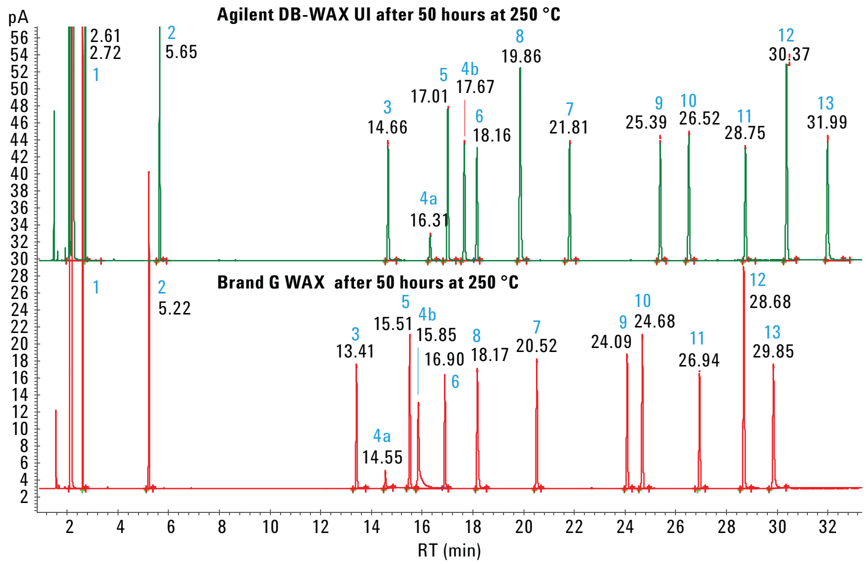

Figure 8. Example FID chromatograms of the modified Grob mix on Agilent DB-WAX UI and Brand G WAX columns, after 50 hours conditioning at 250 °C. See Table 2 for GC conditions and peak identification.

Figure 9 shows the inertness performance of the TK column after conditioning for one hour and 50 hours at 250 °C using the DB-WAX UI test mix. A reasonable inertness was observed for this column after one hour of conditioning at 250 °C. However, inertness performance significantly deteriorated during the longevity test at 250 °C. After 50 hours of conditioning at 250 °C, severely tailing peaks were observed for more active compounds, including propionic acid (peak 3), 2-ethylhexanoic acid (peak 11), and ethyl maltol (peak 12). A similar trend for significant reduction in inertness performance of acids and ethyl maltol on this column was also found using the modified Grob test mix (Figure 10). As a result of the wide variety of chemical properties in the modified Grob test mix, tailing peaks were observed for other active analytes such as decanal (peak 3), 2,3-butanediol (peaks 4a and 4b), and dicyclohexylamine (peak 8).

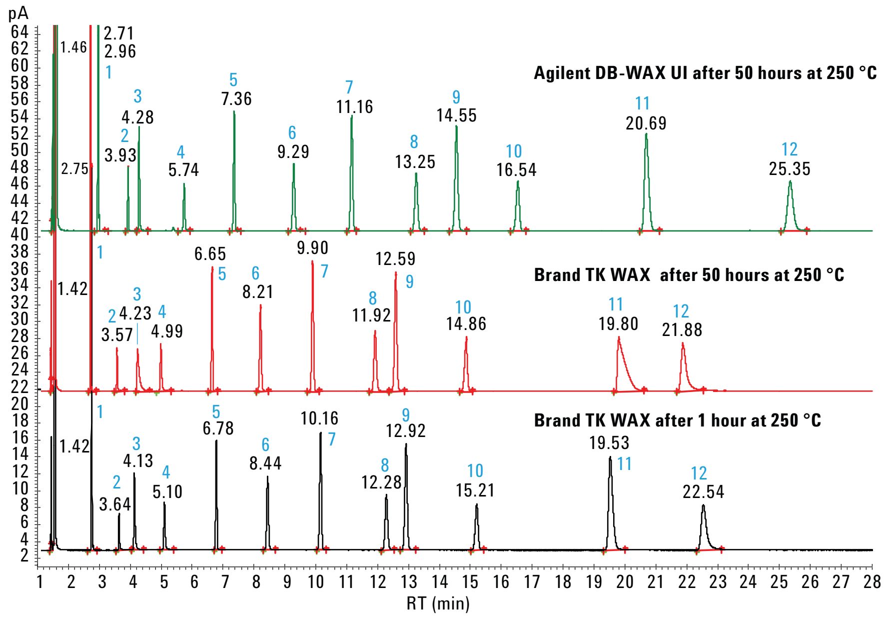

Figure 9. Example FID chromatograms of the Agilent DB-WAX UI test mix on a Brand TK WAX column after one hour and 50 hours of conditioning at 250 °C compared to an Agilent DB-WAX UI after 50 hours of conditioning at 250 °C. See Table 1 for GC conditions and peak identification.

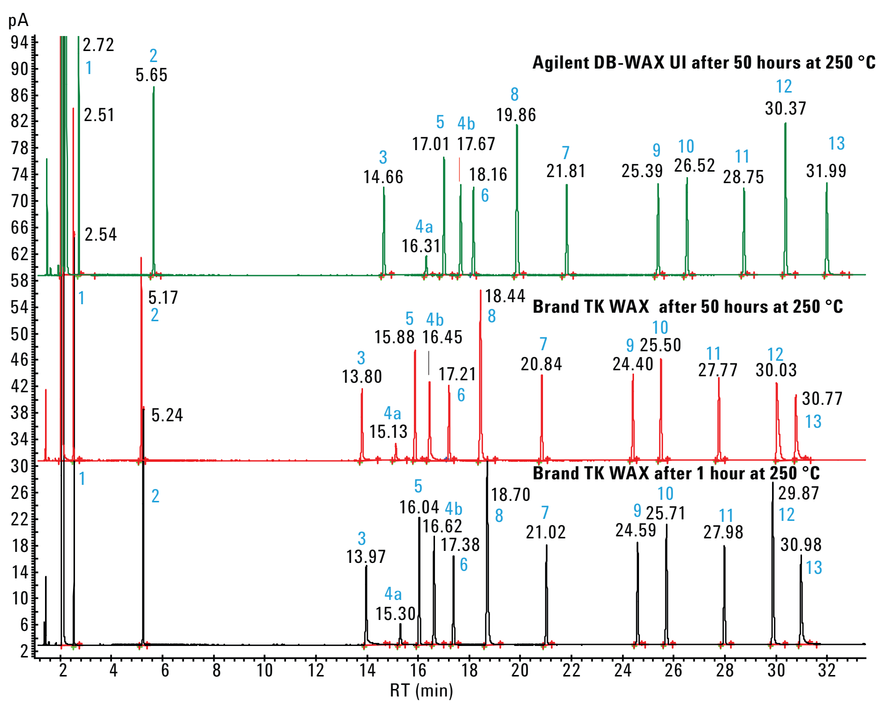

Figure 10. Example FID chromatograms of the modified Grob test mix on Brand TK WAX column after one hour and 50 hours of conditioning at 250 °C compared to Agilent DB-WAX UI after 50 hours of conditioning at 250 °C. See Table 2 for GC conditions and peak identification.

Figure 11 shows example FID chromatograms for the inertness performance and longevity test after 50 hours of conditioning at 250 °C on a Brand M column using the modified Grob test mix. Peak tailing is observed for several active analytes including decanal (peak 3), 2,3-butanediol (peaks 4a and 4b), dicyclohexylamine (peak 8), 2-ethylhexanoic acid (peak 12), and ethyl maltol (peak 13).

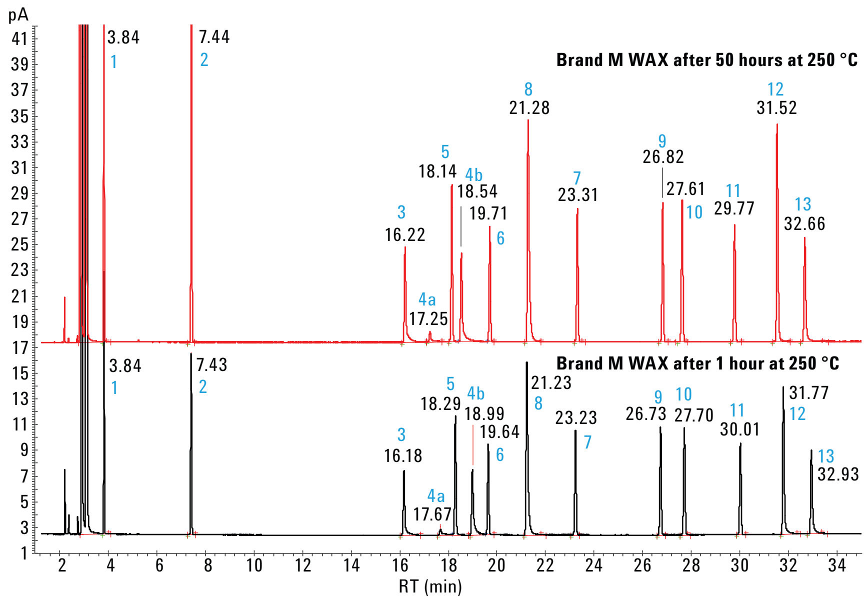

Figure 11. Example FID chromatograms of the modified Grob test mix on a Brand M WAX column after one hour and 50 hours of conditioning at 250 °C. See Table 2 for GC conditions and peak identification.

Using demanding QC test probes, column activity was detected allowing for better classification of inertness performance of WAX columns in this benchmark study. No WAX column on the market shows absolutely perfect inertness for all active analytes of interest in both the DB-WAX UI and modified Grob test mixes. However, compared to other WAX columns, DB-WAX Ultra Inert columns show a far better inertness performance. This improved inertness performance strongly contributes to improved sensitivity and reproducibility for analyses of a wide variety of active analytes of interest at critically low levels. The improved thermal longevity of the DB-WAX UI at the column's upper temperature limit provides better column durability and more consistent analytical results over its lifetime.

Conclusions

In comparison to other WAX columns evaluated in this study, the Agilent DB-WAX Ultra Inert column shows overall superior performance for inertness, thermal stability, and column-to-column inertness consistency. In addition, the same selectivity as the Agilent J&W DB-WAX column facilitates a simplified upgrade to this enhanced, Ultra Inert version with minimal revalidation. For current applications, there is no requirement to recreate or modify existing compound libraries or methods that are based on the J&W DB-WAX. The use of the DB-WAX UI is highly recommended for any of today's demanding GC or GC/MS applications dealing with more active analytes at critical levels. With its excellent performance, the DB-WAX UI is a confident choice to obtain the most sensitive, reproducible, and reliable analytical results.

Table 4. Agilent DB-WAX Ultra Inert ordering guide. Various inner diameters, lengths, and film thickness are available.

Part number	Description
121-7022UI	Agilent DB-WAX Ultra Inert 20 m, 0.18 mm, 0.18 µm
121-7023UI	Agilent DB-WAX Ultra Inert 20 m, 0.18 mm, 0.30 µm
122-7012UI	Agilent DB-WAX Ultra Inert 15 m, 0.25 mm, 0.25 µm
122-7032UI	Agilent DB-WAX Ultra Inert 30 m, 0.25 mm, 0.25 µm
122-7033UI	Agilent DB-WAX Ultra Inert 30 m, 0.25 mm, 0.50 µm
122-7062UI	Agilent DB-WAX Ultra Inert 60 m, 0.25 mm, 0.25 µm
122-7063UI	Agilent DB-WAX Ultra Inert 60 m, 0.25 mm, 0.50 µm
123-7012UI	Agilent DB-WAX Ultra Inert 15 m, 0.32 mm, 0.25 µm
123-7032UI	Agilent DB-WAX Ultra Inert 30 m, 0.32 mm, 0.25 µm
123-7033UI	Agilent DB-WAX Ultra Inert 30 m, 0.32 mm, 0.50 µm
123-7062UI	Agilent DB-WAX Ultra Inert 60 m, 0.32 mm, 0.25 µm
123-7063UI	Agilent DB-WAX Ultra Inert 60 m, 0.32 mm, 0.50 µm
125-7012UI	Agilent DB-WAX Ultra Inert 15 m, 0.53 mm, 1.00 µm
125-7031UI	Agilent DB-WAX Ultra Inert 30 m, 0.53 mm, 0.25 µm
125-7032UI	Agilent DB-WAX Ultra Inert 30 m, 0.53 mm, 1.00 µm
125-7037UI	Agilent DB-WAX Ultra Inert 30 m, 0.53 mm, 0.50 µm
125-7062UI	Agilent DB-WAX Ultra Inert 60 m, 0.53 mm, 1.00 µm
127-7012UI	Agilent DB-WAX Ultra Inert 10 m, 0.10 mm, 0.10 µm
128-7022UI	Agilent DB-WAX Ultra Inert 25 m, 0.20 mm, 0.20 µm

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