Probe Selection with Respect to the 5% Phenyl Ultra Inert Columns Offered by Agilent Technologies: Why Are There Two Test Mixtures?

Technical Overview

Introduction

QC test probes serve a vital function in ensuring the quality and reproducibility of modern GC columns. A good test mixture should measure partition ratio, retention indexes, and probe the inertness of the surface. Properly chosen probes should be adequately resolved from each other. Having adequate separation prevents one test analyte from influencing or masking the behavior of a second analyte. During the development of the Über One test mixture [1], it was discovered that one test mixture would not meet this requirement for both the Agilent J&W DB-5ms Ultra Inert and the Agilent J&W HP-5ms Ultra Inert columns.

The Test Mixtures

In order to resolve this issue, two mixtures were developed to test the Agilent J&W family of 5% phenyl GC columns. The DB-5ms Ultra Inert column is tested with the compounds in Table 1. Figure 1 shows that this mixture delivers clear, baseline separation for all of the test components.

Table 1. DB-5ms Ultra Inert Über One Test Mixture

1.	1-Propionic acid	7.	1,2-Pentanediol
2.	1-Octene	8.	n-Propylbenzene
3.	n-Octane	9.	1-Heptanol
4.	4-Methylpyridine	10.	3-Octanone
5.	n-Nonane	11.	n-Decane
6.	Trimethyl phosphate		

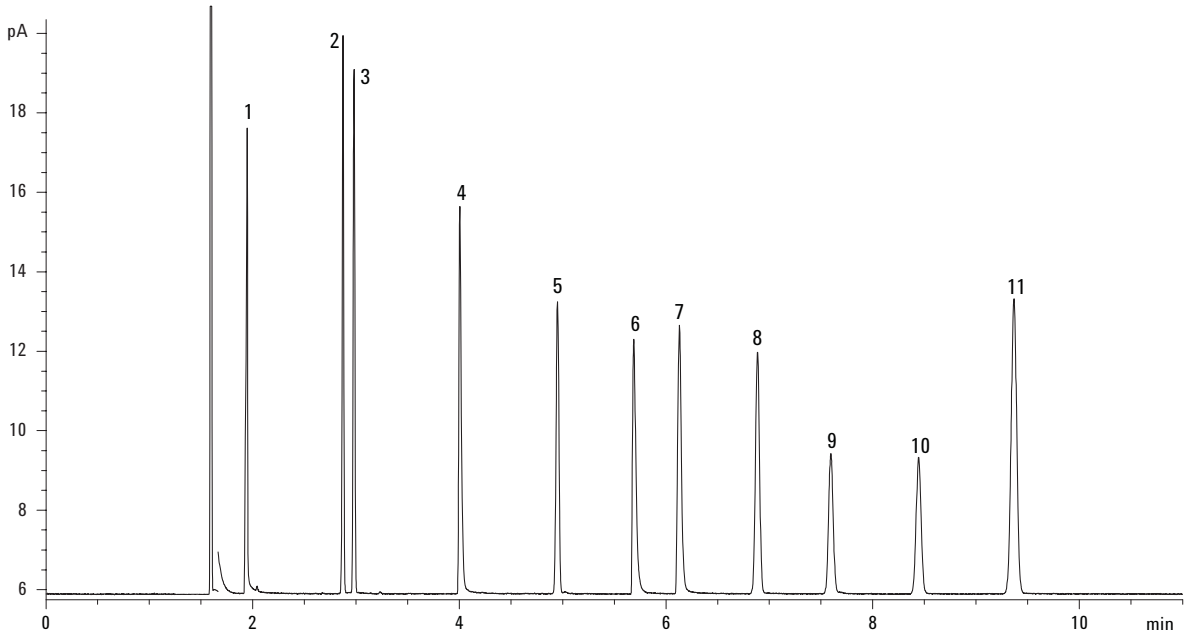


Figure 1. DB-5ms Ultra Inert Über One test mixture on an Agilent J&W DB-5ms Ultra Inert, 30 m × 0.25 mm × 0.25 µm column (Agilent p/n 122-5532UI).

However, when this same mixture is used to test the HP-5ms Ultra Inert column (Figure 2), we find that analytes 6 and 7, trimethyl phosphate and 1,2-pentanediol, respectively, are not baseline resolved. It is no longer possible to evaluate the peak shape of the trimethyl phosphate as it is hidden by the 1,2-pentanediol peak. Furthermore, we are unsure of the contribution of the trimethyl phosphate peak tailing to the 1,2-pentanediol peak shape.

After careful evaluation of a number of analyte candidates, we determined that we could substitute 1,3-propanediol for the 1,2-pentanediol in the HP-5ms Ultra Inert test mixture. The HP-5ms Ultra Inert Über One test mixture (Table 2) maintains the same rigorous test conditions while providing clear, baseline separation of all of the test analytes. In Figure 3 we find the new 1,3-pentanediol peak, D, clearly resolved between n-octane and 4-methylpyridine, peaks C and E, respectively. We also see the timethyl phosphate peak, G, is standing alone, allowing us to evaluate its shape and height.

Table 2. HP-5ms Ultra Inert Über One Test Mixture

A.	1-Propionic acid	G.	Trimethyl phosphate
B.	1-Octene	H.	n-Propylbenzene
C.	n-Octane	I.	1-Heptanol
D.	1,3-Propanediol	J.	3-Octanone
E.	4-Methylpyridine	K.	n-Decane
F.	n-Nonane		

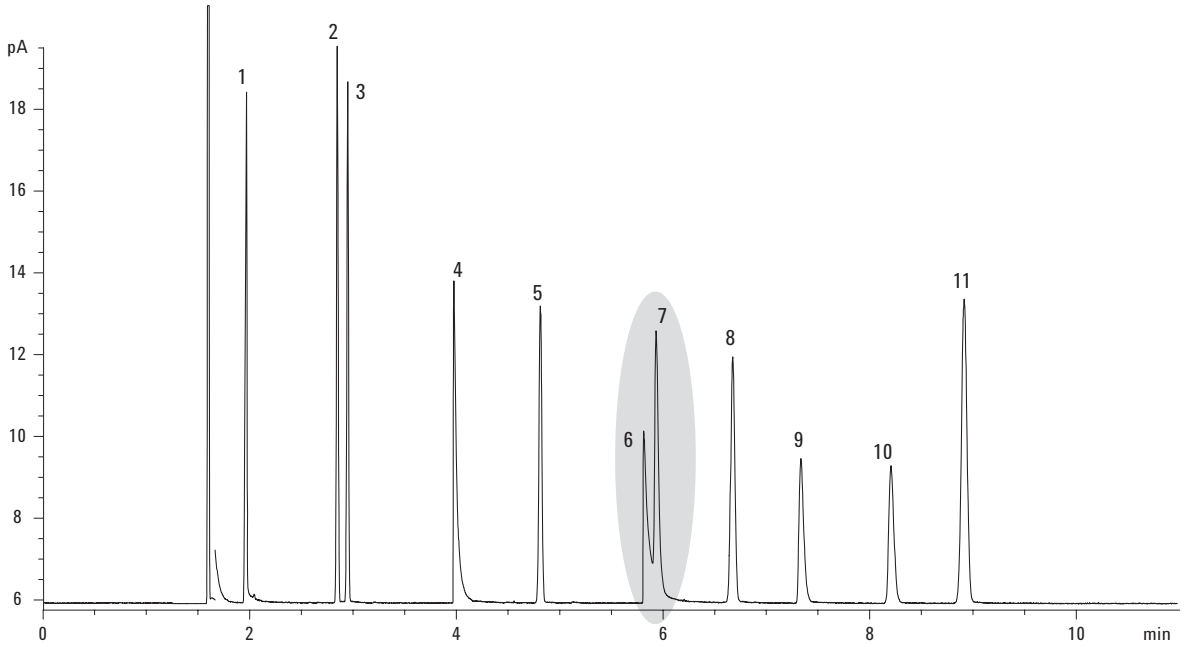


Figure 2. DB-5ms Ultra Inert Über One test mixture on an Agilent J&W HP-5ms Ultra Inert, 30 m × 0.25 mm × 0.25 µm column (Agilent p/n 19091S-433UI).

Table 3. Chromatographic Conditions

GC	Agilent 6890N
Sampler	Agilent 7683B, 0.5-µL syringe (Agilent p/n 5188-5246), 0.02-µL split injection
Carrier	Hydrogen constant pressure 36 cm/sec
Inlet	Split/splitless; 250 °C, 200 mL/min total flow
Inlet liner	Deactivated single taper with glass wool (Agilent p/n 5183-4647)
Figure 1	Agilent J&W DB-5ms Ultra Inert 30 m × 0.25 mm × 0.25 µm (Agilent p/n 122-5532UI)
Figures 2 and 3	Agilent J&W HP-5ms Ultra Inert 30 m × 0.25 mm × 0.25 µm (Agilent p/n 19091S-431UI)
Oven	65 °C isothermal
Detection	FID at 325 °C, 450 mL/min air, 40 mL/min hydrogen, 45 mL/min nitrogen makeup

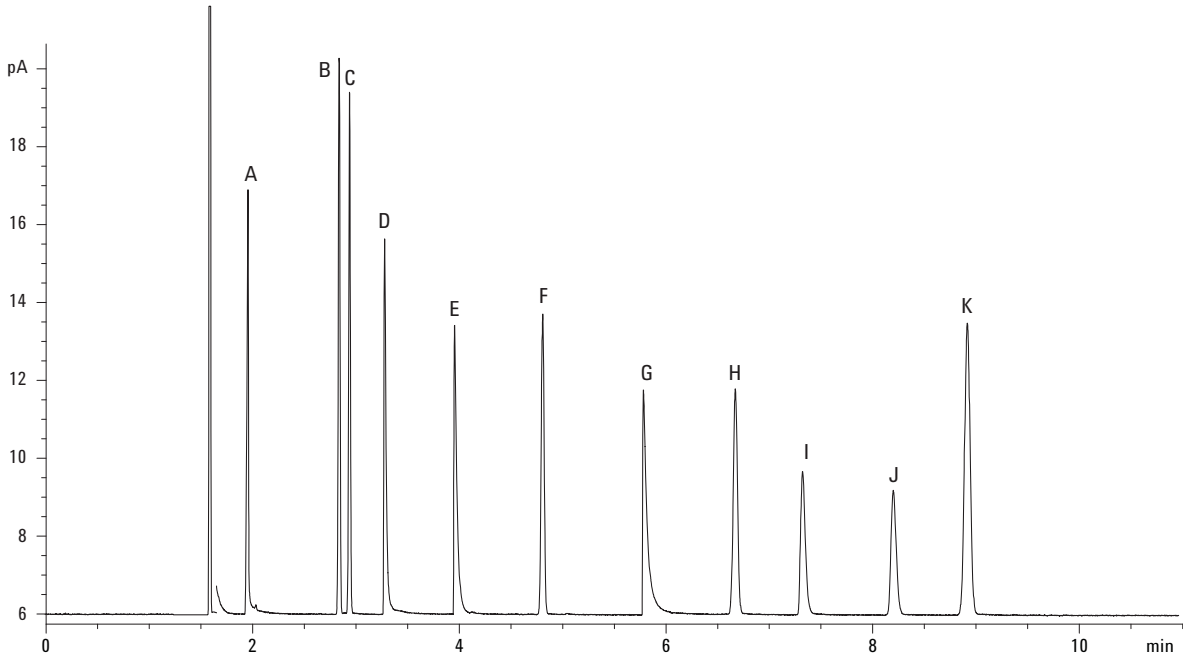
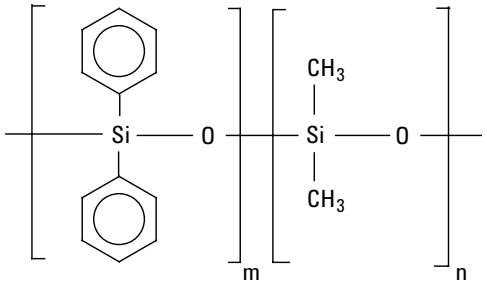


Figure 3. HP-5ms Ultra Inert Über One test mixture on an Agilent J&W HP-5ms Ultra Inert, 30 m x 0.25 mm x 0.25 µm column (Agilent p/n 19091S-433UI).

Differences in Selectivity

Historically a 5% phenyl stationary phase was composed of 5% diphenyl, 95% dimethyl polysiloxane. The backbone linkage of these polymers is the -[-Si-O-]$_n$- siloxane repeating unit. These types of siloxane polymers (structure shown in Figure 4) are the basis to the HP-5ms Ultra Inert columns.

About 20 years ago we began investigating mechanisms to inhibit the thermodynamic decomposition of the siloxanes. The byproduct of this decomposition, cyclic siloxanes, is what one sees as column bleed. One mechanism to decrease this decomposition is to "stiffen" the backbone of the chain by moving the phenyl rings from the pendant group position into the siloxane backbone. These silylarylene types of polymers (structure shown in Figure 4) are the basis to the DB-5ms Ultra Inert columns.



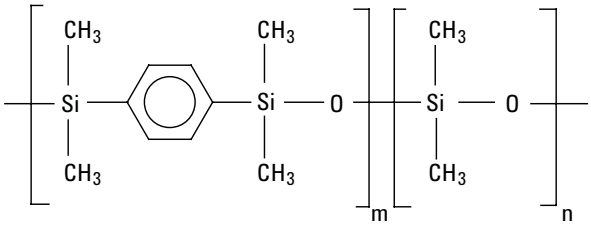


Figure 4. Siloxane composition **Silylarylene composition**

The silylarylene-based stationary phases have been designed to have nearly identical selectivity to the pure siloxane-based stationary phases. In most cases these stationary phases are a direct substitution for each other. However, moving the phenyl rings into the polymer chain causes some slight changes in the electron density distribution in the phenyl rings. This change in electron density may change the stationary phase's interaction with some analytes. It is this difference in selectivity that necessitated the modification of the original Über One test mixture.

Conclusions

Today's demanding analysis of trace active analytes demands the most inert columns available. Slight differences in the selectivity between the DB-5ms and the HP-5ms Ultra Inert columns require a component change to ensure the rigorous testing these columns require. The DB-5ms and HP-5ms Ultra Inert Über One test mixtures have been designed to be very demanding tests of a column's activity, providing analysts the most consistent column inertness performance, and hence the utmost confidence in the analytical results.

Reference

1. "Agilent J&W Ultra Inert GC Columns: A New Tool to Battle Challenging Active Analytes," Agilent Technologies publication 5989-8665EN
2. W. Jennings, "Addressing Concerns in QC Tests for GC Columns," Agilent Technologies *Separation Times*, July 2008, Vol. 2, No. 3

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