

Competitive Column Inertness Analysis for Agilent J&W DB-5ms Ultra Inert Column with Active Basic Compounds

Application Note

Chemical, Foods and Flavors, Environmental, and Forensics

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Abstract

Agilent J&W DB-5ms Ultra Inert columns were compared to similar versions from four other column manufacturers with respect to their chromatographic behaviors for active analytes. Superior results were observed on the DB-5ms columns for basic compounds. Chromatograms depicting peak shape characteristics are presented to show how exceptional inertness leads to a significant reduction in tailing of active basic compounds for more accurate, reliable analytical results. Excellent chromatographic performance was obtained for a diverse group of compounds that included chlorophenols, organophosphorus pesticides and aromatic positional isomers demonstrating the applicability of the DB-5ms column for a wide range of applications.



Agilent Technologies

Introduction

The chromatographic analysis of very active samples by GC can be a challenging task unless the GC flow path is inert toward these types of compounds. A stable, unreactive GC column has a considerable effect on the inertness of the flow path due to its high surface area, and will not have active sites that interact unfavorably with sample compounds. Both low column bleed and inertness are critical to maximizing sensitivity, yet inertness is central to maintaining optimum column performance for these demanding substances. A high level of inertness is needed to achieve sharp, symmetrical peaks especially at trace levels. Because the GC column is such an integral part of the chromatographic system, it is in an analyst's best interest to have the most inactive column possible to consistently achieve high quality data [1-5].

The adverse effects of a non-inert column are displayed as differences in peak deformation (tailing and irreversible adsorption) of active compounds. The column can have acidic characteristics that cause unwanted interactions with basic compounds. A group of active basic compounds was analyzed on the DB-5ms Ultra Inert column and columns of the same chemistry from four different vendors to measure inertness of the columns toward basic compounds and assess the acidic nature of the columns. Both qualitative (peak shape) and quantitative (tailing factor, T_f values) data were used to assess the activity of the columns in the study.

Experimental

An Agilent 6890N network GC system equipped with an Agilent 7683B automatic liquid sampler and a flame ionization detector (FID) was used for this column inertness study. Sample introduction was done by a single split/splitless injection port. Table 1 lists the columns included in the study.

Table 1. Columns Used in Study

Column 1:	Agilent J&W DB-5ms Ultra Inert 30 m × 0.25 mm × 0.25 µm (Agilent p/n 122-5532UI)
Column 2:	Restek Rxi®-5Sil MS 30 m × 0.25 mm × 0.25 µm
Column 3:	Varian VF-5ms 30 m × 0.25 mm × 0.25 µm
Column 4:	Phenomenex Zebron® ZB-5ms 30 m × 0.25 mm × 0.25 µm
Column 5:	GL Sciences InertCap 5ms/Sil 30 m × 0.25 mm × 0.25 µm

Two columns were tested from each vendor to confirm reproducibility of results. All columns used in this comparison were installed in the GC and analyzed in the same manner. The injection of undecane at 0.2% in methanol was done to ensure that a symmetrical peak was obtained thus verifying proper installation. The composition of the basic test mixture consisted of the six compounds listed in Table 2 and was analyzed using the chromatographic conditions included in Table 3. The basic standard compounds were purchased from Sigma-Aldrich (≥ 98% purity) and the mixture prepared in methanol (Burdick & Jackson High Purity Solvent). The same standard mixture was injected onto each column at least five times.

Table 2. Basic Test Mixture in Methanol

1. Triethylamine	0.2%
2. Pyridine	0.2%
3. 4-Methylpyridine	0.2%
4. N,N-Dimethylacetamide	0.5%
5. 2,4-Dimethylaniline	0.1%
6. Dicyclohexylamine	0.15%

Table 3. Chromatographic Conditions for Basic Test Mixture

GC:	Agilent 6890N
Sampler:	Agilent 7683B, 10 µL syringe (Agilent p/n 5181-3360)
Carrier:	Helium in Constant Flow mode, 1.5 mL/min
Inlet:	Split/splitless at 250 °C, Split 50:1, 0.2 µL injected
Inlet liner:	Single taper, MS certified liner with restriction to hold glass wool (Agilent p/n 5188-6576)
Oven Program:	30 °C (0.5 min) to 60 °C (1 min) at 5 °C/min, then to 150 °C (5 min) at 50 °C/min
Detection:	FID at 250 °C; hydrogen 40 mL/min; air 450 mL/min; nitrogen makeup 45 mL/min

Tables 4 and 5 list the acidic and organophosphorus pesticide compounds tested on the DB-5ms column with their respective concentrations. The chlorophenols were purchased from Sigma-Aldrich (≥ 98% purity) and the organophosphorus pesticides were obtained from ChemService. The positional aromatic compounds include o-xylene, m-xylene and p-xylene and were purchased from Sigma-Aldrich (≥ 98% purity) and prepared in methanol at 0.2%. Table 6 includes the chromatographic conditions used for the acids, pesticides and aromatic isomers.

Table 4. Acid Test Mix in Methanol

1. 2,4-Dichlorophenol	0.5%
2. 2,4,6-Trichlorophenol	0.5%
3. Pentachlorophenol	0.5%

Table 5. Organophosphorus Pesticides Test Mix in Methanol

1. Dichlorovos	100 ppm
2. Dimethoate	139 ppm
3. Methyl parathion	109 ppm
4. Malathion	100 ppm
5. Chlorpyrifos	109 ppm
6. Parathion	100 ppm

Table 6 Chromatographic Conditions for Acids, Pesticides and Aromatic Isomers

Chlorophenols

Injection:	0.2 µL, Split, 100:1
Injection temperature	250 °C
Carrier:	Helium at 1.5 mL/min in Constant Flow mode
Oven:	50 °C (1 min); 30 °C to 300 °C (5 min)
Detection:	FID at 250 °C; hydrogen 40 mL/min; air 450 mL/min; nitrogen makeup 45 mL/min

Organophosphorus Pesticides

Injection:	1 µL, Split, 10:1
Injection temperature	250 °C
Carrier:	Helium in Constant Pressure mode at 25 psi
Oven:	30 °C (0.2 min); 30 °/min to 240 °C (4 min)
Detection:	FID at 250 °C; hydrogen 40 mL/min; air 450 mL/min; nitrogen makeup 45 mL/min

Aromatic Isomers (o-,m- and p-Xylene, 0.2% Methanol)

Injection:	0.2 µL, Split, 300:1
Injection temperature	250 °C
Carrier:	Helium at 1.5 mL/min in Constant Flow mode
Oven:	30 °C isothermal
Detection:	FID at 250 °C; hydrogen 40 mL/min; air 450 mL/min; nitrogen makeup 45 mL/min

Results and Discussion

A distinct difference in column inertness was observed with the basic compound group highlighting significant variations in the acidic nature of the columns. Visually, there is noticeable tailing with the first four peaks in the chromatograms for the Restek, Varian, Phenomenex and GL Sciences columns as shown in Figure 1. Excellent peak shapes were obtained for

the compounds of interest with the DB-5ms column. A measurement of the peak tailing was done using the US Pharmacopeia tailing factor (T_f). This is calculated using the following formula [6].

$$T_f = W_{5.0} / (T_w \times 2)$$

where: T_w = distance between peak front and retention time of peak (T_R) at 5% of peak height, units are the same as used for $W_{5.0}$.

$W_{5.0}$ = width at 5% of height

The tailing factors (T_f) are listed in Table 7 for all the basic compounds and the T_f values for the first four peaks are included in the chromatograms in Figure 2. When compared to other columns, the DB-5ms Ultra Inert column shows a significant reduction in the tailing factors for four of the bases. There is also incomplete resolution of 4-methylpyridine and N,N-dimethylacetamide in the chromatograms for the Rxi®-5Sil MS, Zebron® ZB-5ms P and VF-5ms columns. This observation shows how the lack of column inertness can negatively affect resolution, and consequently selectivity, since resolution is a measure of the degree of separation (selectivity) of two compounds. The selectivity of the other vendor's columns is not sufficient with the experimental conditions used, to separate 4-methylpyridine and N,N-dimethylacetamide due to the effects of column activity.

A diverse group of compounds that included acids (chlorophenols), organophosphorus pesticides and positional aromatic isomers was analyzed on the DB-5ms column. Excellent chromatographic results were obtained for these compounds, in addition to the bases, with the Agilent J&W DB-5ms Ultra Inert column. The separation of the aromatic isomers demonstrates that the column retains the unique selectivity to separate isomers that is not compromised as a result of the enhanced inertness. The chromatograms in Figure 3 depict the very favorable chromatographic results attained for this wide-ranging group of compounds and illustrate the versatility of the DB-5MS Ultra Inert column for a variety of applications.

Exceptional Peak Shape for Basic Compounds Evident on Agilent J&W DB-5ms Ultra Inert Column

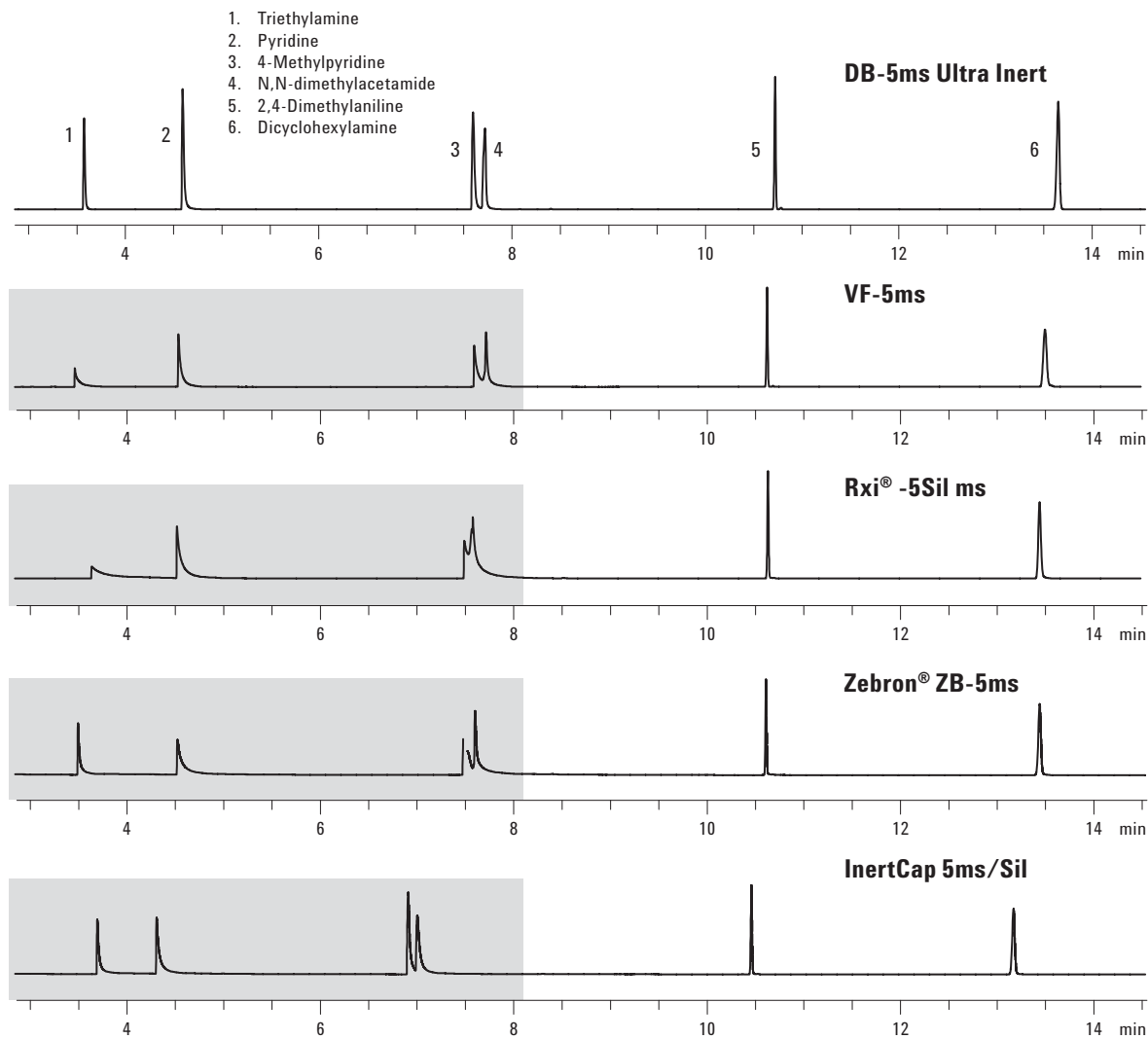


Figure 1. Chromatograms of basic compounds on different vendor's columns. Chromatographic conditions are listed in Table 3.

Table 7. Tailing Factors (T_f) for Basic Compounds on -5ms Columns

	Tailing Factor (T_f)				
	DB-5MS	Zebron® ZB-5ms	InertCap 5ms/Sil	Rxi®-5Sil MS	VF-5ms
Triethylamine	1.5	7.1	7.7	44	24
Pyridine	2.4	17	5.1	10	9.7
4-Methylpyridine	1.7	4.2	4.7	6.1	7.3
N, N-dimethylacetamide	0.95	3.9	4.6	1.5	1.6
2, 4-Dimethylaniline	0.95	1.0	1.0	0.99	0.95
Dicyclohexylamine	0.87	0.98	1.1	1.0	1.0

Significantly Lower T_f Values Reveal the Extraordinary Inertness of the DB-5ms Ultra Inert Column

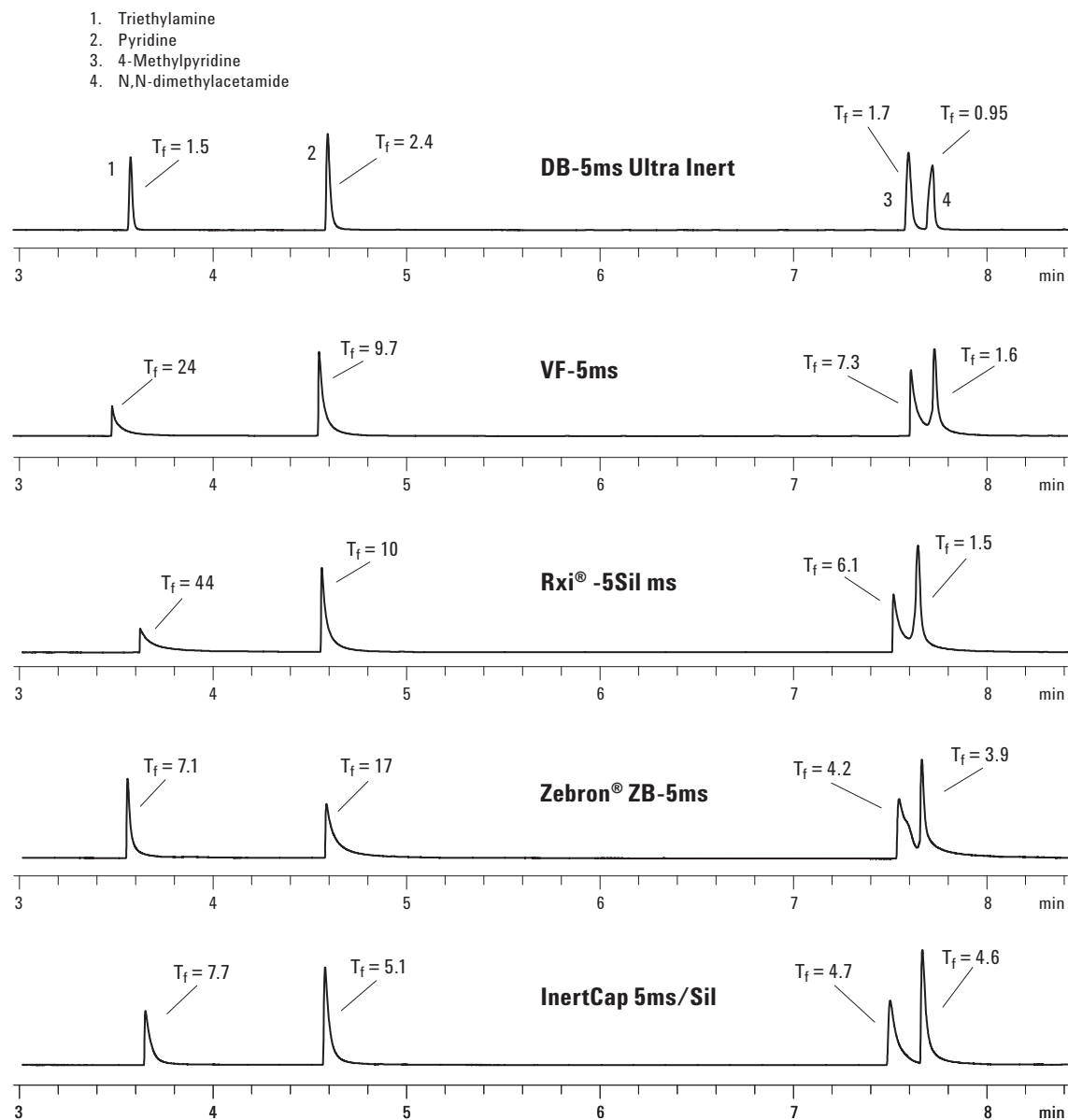


Figure 2. Tailing factors (T_f values) for four basic compounds on different vendor's columns. Chromatographic conditions are listed in Table 3.

Excellent Chromatographic Performance Exhibited for a Diverse Group of Compounds

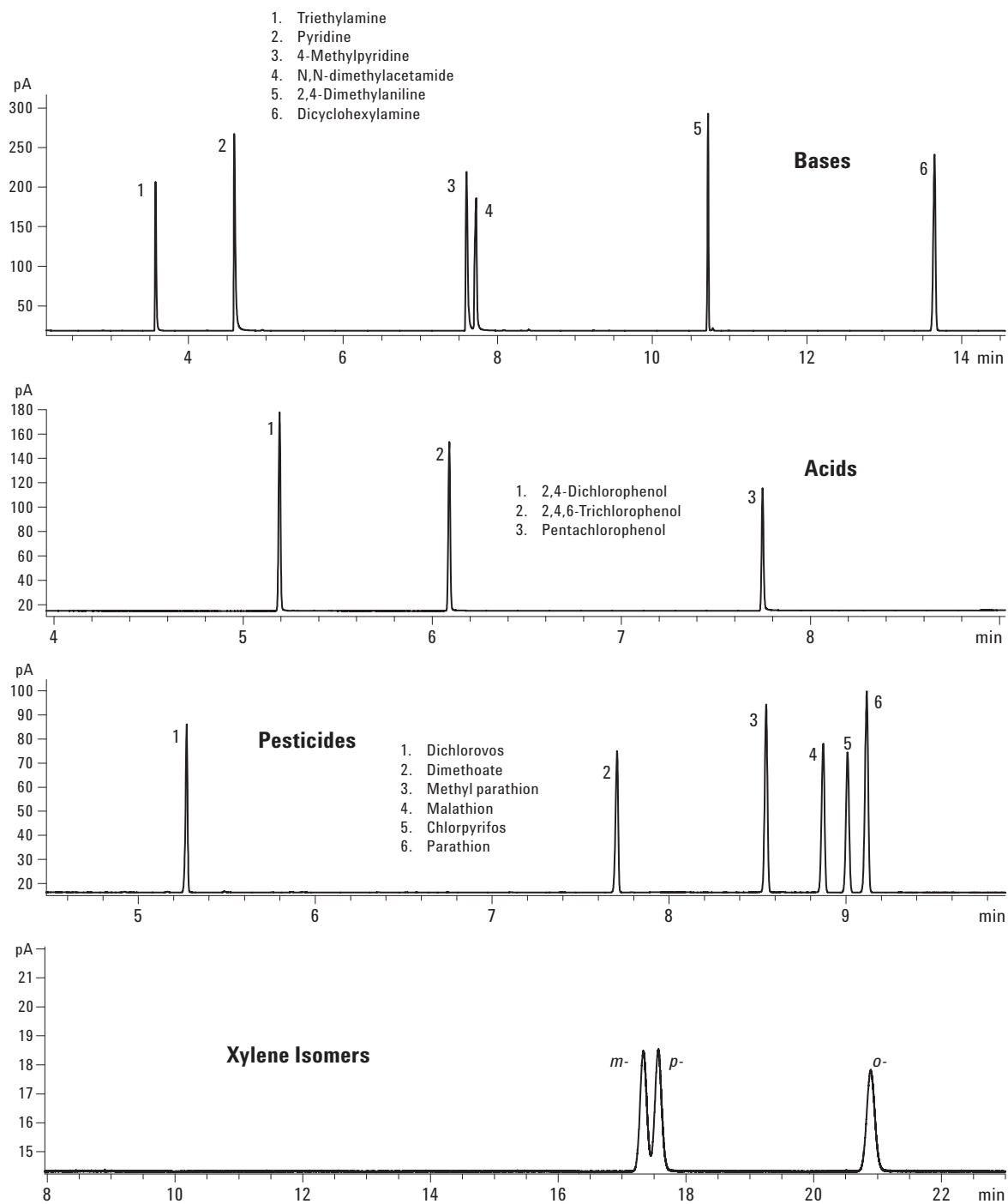


Figure 3. Chromatograms for bases, chlorophenols, organophosphorus pesticides and xylene isomers on an Agilent J&W DB-5ms Ultra Inert column. Chromatographic conditions are included in Tables 3 and 6.

Conclusions

Agilent J&W DB-5ms Ultra Inert columns are superior to other vendor's columns in the same category for the analysis of basic compounds. The high level of inertness results in a better peak shape for active compounds and is visually apparent and verified by tailing factor (T_f) calculations. A high level of sensitivity that translates to lower detection limits is achieved because of the excellent peak shape obtained with the highly inert DB-5ms Ultra Inert column. Excellent chromatographic performance for a varied group of compounds, including chlorophenols, aromatic isomers and pesticides, was clearly demonstrated.

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