Analysis of Distilled Spirits Using an Agilent J&W DB-WAX Ultra Inert Capillary GC Column

Application Note

Flavors and Fragrances, Food

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Abstract

The Agilent J&W DB-WAX Ultra Inert GC column has excellent stability for distilled spirit samples containing high levels of water or water-ethanol mixture. The high inertness performance of the column ensures good peak shapes for problematic volatile congeners including alcohols, aldehydes, and organic acids.

Introduction

The quality and flavor of alcoholic beverages relate directly to the complex mixture of ingredients in the beverage. Some ingredients convey pleasing taste and aromatic characteristics while other elements can impart undesirable flavor or taste features. Manufacturers and blenders of distillates worldwide have an expert understanding of these factors, balancing their sensory skills with modern analytical tools such as GC and GC/MS. The control of spirit flavor quality and meeting regulatory requirements are necessities as a distillery expands trade, exports, and tries to protect its brand against counterfeiting.

Historically, gas chromatography with polar polyethylene glycol (PEG, wax type) GC phases is one of the tools used to characterize distilled spirits. PEG phase columns exhibit excellent selectivity for the flavor elements found in distillates, generating useful composite information. The analysis of distilled spirits is quite challenging because of the high levels of water in the samples (40–80%). In addition, the levels of some of the target compounds are in the lower parts per million (ppm).

Traditionally, PEG columns have had shortcomings particularly with more chemically active components in the alcohol/water matrix. The analysis of aldehydes, alcohols, and organic acids suffer from a lack of column inertness, which results in tailing peaks and variable, inaccurate results. Some traditional PEG phases can be unstable with aqueous samples, which can lead to column deterioration, poor reproducibility, and decreased lifetime.

In this application note, Agilent J&W DB-WAX UI columns are used to characterize distilled spirits. This study highlights the impact an inert wax column has on the more problematic components in spirits. The unique inertness of this phase delivers sharper, more consistent peak shapes for alcohols, organic acids, and aldehydes found in various standard and premium distillates.

Experimental

Samples were obtained locally from retail providers of spirits in Shanghai, China, and Wilmington, DE USA. Before analysis, each sample was carefully transferred to a 2 mL autosampler vial, and introduced into the GC as neat samples.

Instrumentation

Tables 1 and 2 show the instruments and conditions, and Table 3 lists the flow path consumable supplies.

Table 1. System 1 conditions.

Parameter	Value
GC system:	Agilent 7890B/FID
Column:	Agilent J&W DB-WAX UI, 30 m × 0.25 mm, 0.25 µm (p/n 122-7032UI)
Autosampler:	Agilent 7683B autosampler and sample tray, 5 µL syringe (p/n G4513-80213), 0.5 µL injection volume
Carrier gas:	Helium, constant flow mode, 0.7 mL/min
Inlet:	Inert flow path Split/splitless, 250 °C, split ratio 30:1
Oven:	For sample run 40 °C (4 min), 5 °C/min to 100 °C, 10 °C/min to 200 °C (10 min) (run time 36 min)
	For solvent run 40 °C (4 min), 5 °C/min to 56 °C, 100 °C/min to 200 °C (10 min) (run time 18.64 min)
FID:	250 °C

Table 2. System 2 conditions.

Parameter	Value
GC system:	Agilent 7890B/FID
Column:	Agilent J&W DB-WAX UI, 30 m × 0.25 mm, 0.25 µm (p/n 122-7032UI)
Autosampler:	Agilent 7683B autosampler and sample tray, 5 µL syringe (p/n G4513-80213), 0.5 µL injection volume
Carrier gas:	Hydrogen, constant flow mode 2.5 mL/min
Inlet:	Split/splitless, 225 °C, split ratio 20:1
Oven:	35 °C (5 min), 12 °C/min to 240 °C (hold 5.0 min)
FID:	250 °C

Table 3. Flow path supplies.

Parameter	Value
Vials:	Amber, write-on spot, certified, 2 mL, screw top vial packs (p/n 5182–0554)
Septa:	Nonstick BTO septa (p/n 5183-4757)
Column nut:	Self-tightening, inlet/detector (p/n 5190-6194)
Ferrules:	15% graphite: 85% Vespel, short, 0.4 mm id, for 0.1 to 0.25 mm columns (10/pk, p/n 5181-3323)
Liner:	Agilent Ultra Inert split liner with glass wool (p/n 5190-2295)
Inlet seal:	Ultra Inert, gold-plated, with washer (p/n 5190-6144)

Results and Discussion

Chinese liquor has been made for over 5,000 years [2]. The typical process of liquor production involves fermentation, distillation, and blending. The different raw materials, various microorganisms, and diverse procedures in different production regions mean that the mixtures of components in Chinese liquors are complex, including esters, acids, alcohols, aldehydes, and other trace level flavor compounds [3].

Figure 1 is an example chromatogram profiling some of the major components found in a Chinese liquor. The DB-WAX UI column shows excellent resolution and peak shape for the more challenging elements typical in Chinese liquor samples. Butyl acetate (peak 11) is used as an internal standard. As shown in Figure 1, ethyl acetate (peak 3) is baseline separated from acetal (peak 4). 2-pentanol (peak 13) and ethyl valerate (peak 14) cannot be separated well on some vendors' WAX type columns. However, they have good resolution on the DB-WAX UI column. Inertness performance and selectivity of DB-WAX UI ensures that furfural is resolved from acetic acid (peak 24) and isoamyl caproate (25). Ethanol (38% v/v) in this sample is severely overloaded but shows excellent peak shape. Methanol, *n*-propanol, isobutanol, 2-pentanol, 1-butanol, isoamyl alcohol, 2-heptanol, and 1-hexanol all elute with sharp symmetrical peaks. Aldehydes in distilled alcoholic beverages can also be problematic, often showing tailing peaks with variable retention times. Here, on the inert wax column, the aldehyde peaks are sharp and easy to integrate. Arguably, the most problematic class of compounds found in the samples is the organic acids. These compounds typically show severe peak tailing, making accurate quantification all but impossible on the less inert wax columns. Using the DB-WAX UI column however, organic acid peaks are sharp and symmetrical, enabling consistent integration and reliable quantification.

Repeatability of the analysis and column performance stability was tested with 200 injections of neat Chinese liquor or the solvent blanks (ethanol:water, 38:62 v/v). Every 10 analyses, a solvent blank was injected.

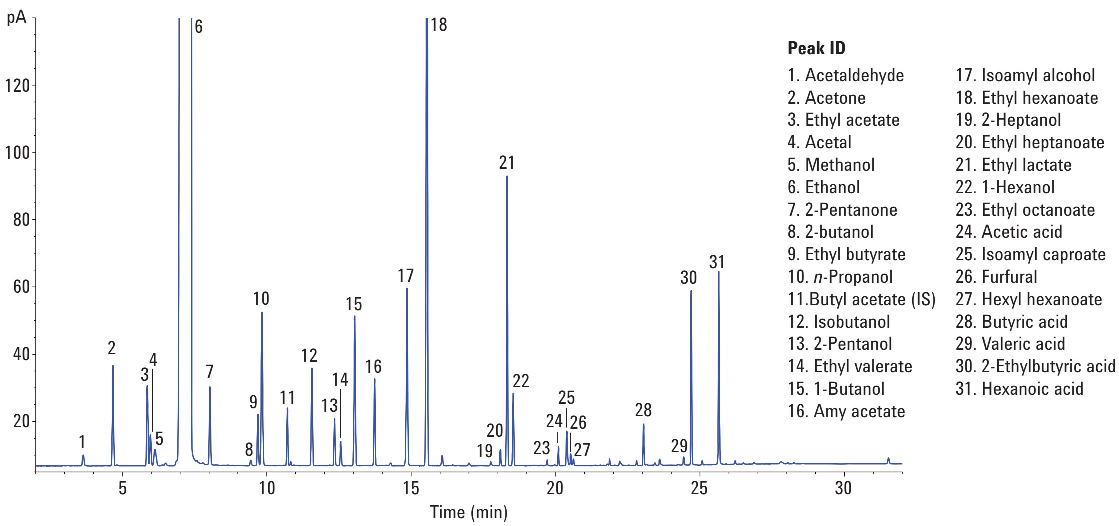

Figure 1. GC/FID chromatogram of a Chinese liquor sample using an Agilent J&W DB-WAX Ultra Inert 30 m × 0.25 mm, 0.25 µm GC column (p/n 122-7032UI).

Figure 2 illustrates retention time stability over the course of 200 replicate injections. Given that this neat Chinese liquor sample is predominately water (62% v/v), there is no retention time stability drift resulting from repeated injections of aqueous samples. Peak shapes are also maintained over the course of the study. The consistent retention times and peak shapes over the 200 injections are clear indications of the stability and inertness of the DB-WAX UI column, for the analysis of complex aqueous alcoholic beverages.

Figure 3 is an overlay of the first and 50th injections of neat Maker's Mark bourbon whiskey. Alcohol, aldehyde, and organic acid peaks are sharp and well-defined. Retention time reproducibility was excellent over the course of 50 largely aqueous (60% by volume) injections of a neat whiskey sample. Peak shapes for problematic analytes were symmetrical and consistent throughout the study. The DB-WAX UI capillary GC column showed excellent inertness and phase stability with the Marker's Mark sample.

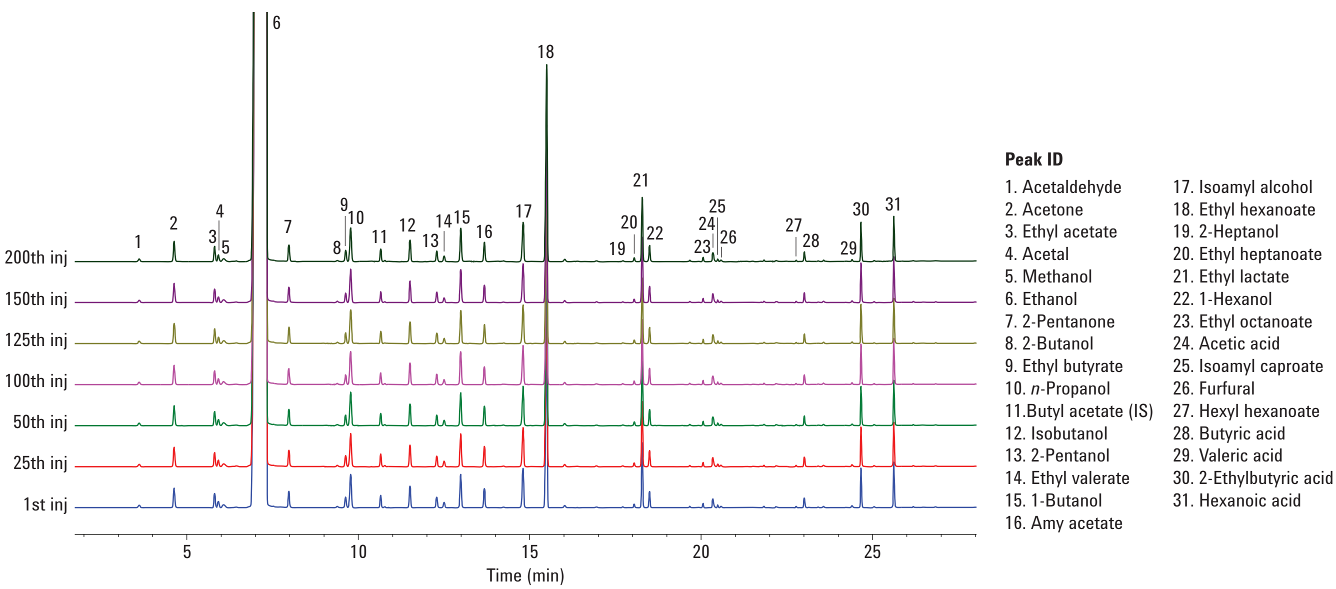

Figure 2. Overlaid GC/FID chromatograms of repeat injections of the same Chinese liquor shown in Figure 1. Produced using an Agilent J&W DB-Wax Ultra Inert 30 m × 0.25 mm, 0.25 µm GC column (p/n 122-7032UI).

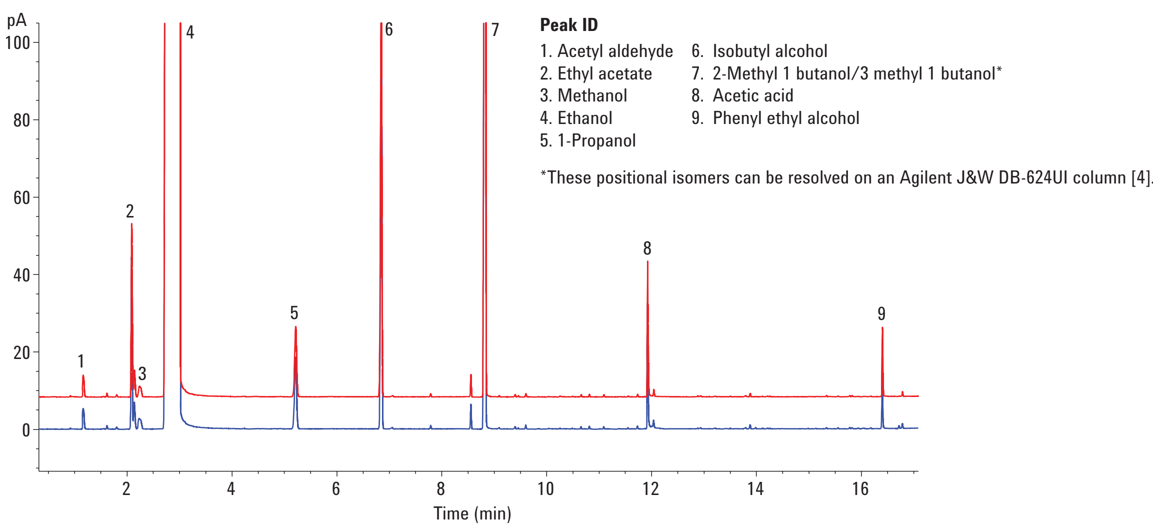

Figure 3. Overlaid FID chromatograms of the first and 50th injections of neat Maker's Mark Whiskey. Conditions as in Table 2.

Conclusions

This application note shows clearly the positive impact the Agilent J&W DB-WAX UI column has on the quantification of problematic volatile compounds found in distilled spirits. Samples were injected repeatedly, demonstrating phase stability for largely aqueous injections. Retention times and peak shapes for alcohols, aldehydes, and organic acids were consistent over the course of 200 direct sample injections of a premium Chinese liquor. The Inert Wax column demonstrates both superior inertness and consistency for the analysis of distilled spirits.

References

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