Application Note

Spirits Analysis

Analysis of Distilled Spirits Using an Agilent 8890 Gas Chromatograph System

Author

Brent Casper
Agilent Technologies, Inc.
Wilmington, DE, USA.

Abstract

The analysis of distilled spirits is a challenging application for gas chromatography (GC) because of the large aqueous component of the sample matrix. Water within the sample shortens GC column life, and requires the user to perform continuous inlet and column maintenance. This Application Note demonstrates the capability of an Agilent 8890 GC system with an Agilent J&W DB-WAX UI column to precisely perform the repeated analysis of spirits.

Introduction

Over the past several years, the popularity of American bourbon whiskey has been increasing. To meet this increasing demand, there has been a dramatic growth in distilleries across the United States. This increase in production of bourbon whiskey has provided a need for quick and reproducible analysis of spirits.

Traditionally, analysis of distilled spirits by GC has proven to be difficult because of their high water contents (40 to 80 %)[1]. Normally, the analysis of a sample with such high water content requires significant sample preparation or the use of liquid chromatography (LC)[2]. When the analysis of spirits is performed with GC, a polar polyethylene glycol (PEG) column is typically used for separation. Polar PEG columns tend to degrade, with repeated injections of samples containing matrices with high concentrations of alcohol/water. This requires the user to perform frequent inlet or column maintenance or both[3].

This Application Note demonstrates the use of a J&W DB-WAX UI GC column in an 8890 GC system for the analysis of spirits. The workflow involved repeated injections of neat bourbon whiskey. This demonstrated the ability of this system to analyze complex samples with challenging matrices and maintain retention time and area precision.

Experimental

Test samples

Woodford Reserve Distiller's Select bourbon whiskey was purchased from a local retail provider of spirits. Bourbon whiskey samples were then transferred to a 2-mL autosampler vial with a 250-µL vial insert, and injected neat into the GC.

Instrumentation

Analysis of bourbon whiskey was completed on an 8890 GC with a flame ionization detector (FID). A split/splitless inlet was used in split mode. Helium carrier gas was used in constant flow mode. Table 1 provides detailed method parameters, and Table 2 presents a list of consumables.

Table 1. GC method conditions.

Method parameters	
Gas chromatograph	8890 Series GC
Software	OpenLab CDS 2.2
Automatic liquid sampler	Agilent 7693A automatic liquid sampler (1 µL injection)
Inlet (split/splitless)	250 °C, 50:1 split
Column	J&W DB-WAX UI (p/n 122-7032UI)
Column flow	2.0 mL/min (constant flow)
Oven	40 °C (4 minutes hold), 5 °C/min to 100 °C (no hold), 10 °C/min to 200 °C (no hold) Method time: 26 minutes
FID	250 °C 400 mL/min air 30 mL/min hydrogen 25 mL/min nitrogen

Table 2. List of Agilent consumables used.

Consumables	Part number
Screw caps with septa	5185-5820
2 mL screw top vials	5182-0716
Vial insert (250 µL)	5181-8872
ALS syringe, blue, 10 µL, PTFE plunger	G4513-80203
Inert septa, Advanced Green	5183-4759
Inlet liner O-ring, nonstick	5188-5365
Inert liner, Ultra Inert, split, low pressure drop	5190-2295
J&W DB-WAX Ultra Inert, 30 m × 0.25 mm, 0.25 µL column	122-7032UI
J&W DB-WAX Ultra Inert, 20 m × 0.18 mm, 0.18 µL column	121-7022UI

Results and discussion

Figure 1 shows an example chromatogram of the analysis of neat Woodford Reserve bourbon whiskey by GC/FID. Because of the high ethanol content of the sample (45 %), the ethanol peak (peak 3) is much larger than minor components. Use of the J&W DB-WAX UI column provides excellent peak shape for other challenging analytes of interest. These include alcohols, esters, and organic acids, which are present in much smaller concentrations. These minor constituents of bourbon whiskey help to make up the variety of complex flavors that are present within the spirit[1].

Because of the variety of analytes of interest present within bourbon, an inert GC flowpath is required when repeat analyses are performed. Use of a J&W DB-WAX UI column enables the analysis of multiple injections of neat bourbon whiskey without repeated column maintenance.

A stability study was conducted with repeated injections of bourbon whiskey. Figure 2 shows the stacked FID chromatograms of the 1st and 400th injection of the bourbon sample. Figure 2 shows that retention time stability and peak shape were held constant over the 400 injections. This demonstrates the robustness of the 8890 GC system and J&W DB-WAX UI column. Minimal peak tailing was also observed from chromatographically challenging compounds, such as acetic acid (peak 7) after 400 repeated injections.

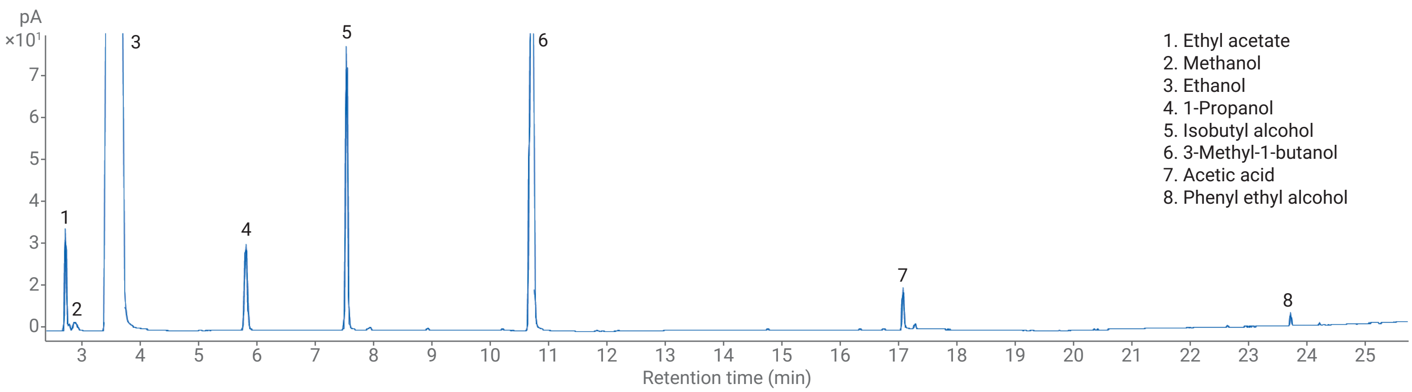

Figure 1. Example chromatogram of the analysis of Woodford Reserve Distiller's Select bourbon whiskey.

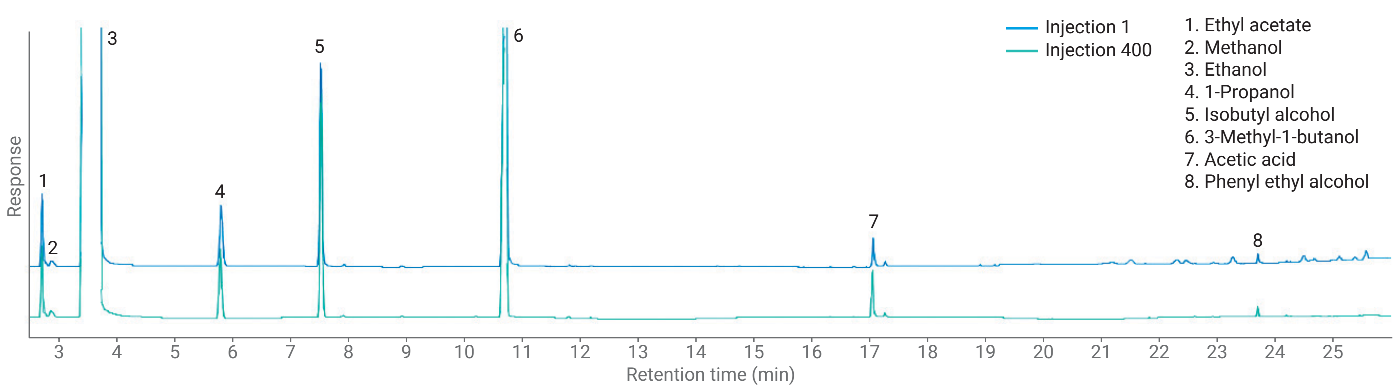

Figure 2. Example stacked chromatogram of the 1st and 400th injection of Woodford Reserve Distiller's Select bourbon.

Another set of experiments was aimed at increasing throughput by reducing the GC run time required for the analysis of spirits. To decrease analytical time, two different approaches were investigated. First, a column with smaller internal diameter (id) and similar stationary phase was installed. Second, hydrogen carrier gas was used. Table 3 provides the method parameters used with the smaller id column with both helium and hydrogen carrier gas. Method translator was used to aid in transferring the method from a 30 m × 0.25 mm, 0.25 µm column to a 20 m × 0.18 mm, 0.18 µm column.

Figure 3 shows the results for the analysis of Woodford Reserve bourbon whiskey with helium carrier gas on the smaller 0.18 mm id column. The original 26 minute analysis time has now been reduced to 19 minutes using the smaller id column. Good peak shapes for the analytes of interest were obtained even with this shorter analysis time. An example is the organic acid indicated as peak 7 in Figure 3.

Switching from helium to hydrogen carrier gas further reduced the analysis time of the spirits to 12.8 minutes. Figure 4 shows the chromatogram using a 20 m × 0.18 mm, 0.18 µm column with hydrogen carrier gas. Changing to hydrogen carrier gas and a smaller id column allows the analysis time to be reduced to half (26 to 13 minutes) of the original analysis time (Figure 1), while still maintaining peak shape of the analytes of interest.

Table 3. Method conditions for 0.180 mm id column analysis.

	Helium carrier gas	Hydrogen carrier gas
Inlet (split/splitless)	250 °C, 200:1 split	250 °C, 200:1 split
Column	J&W DB-WAX UI (p/n 121-7022UI)	J&W DB-WAX UI (p/n 121-7022UI)
Column flow	1.0 mL/min (constant flow)	1.2 mL/min (constant flow)
Oven	40 °C (4 minutes hold); 8.8 °C/min to 100˚C (no hold); 17 °C/min to 200 °C (2.3 minutes hold) Method time: 19.01 minutes	40 °C (2.67 minutes hold); 13 °C/min to 100 °C (no hold); 25 °C/min to 200 °C (1.54 minutes hold) Method time: 12.83 minutes
FID	250 °C 400 mL/min air 30 mL/min hydrogen 25 mL/min nitrogen	250 °C 400 mL/min air 30 mL/min hydrogen 25 mL/min nitrogen

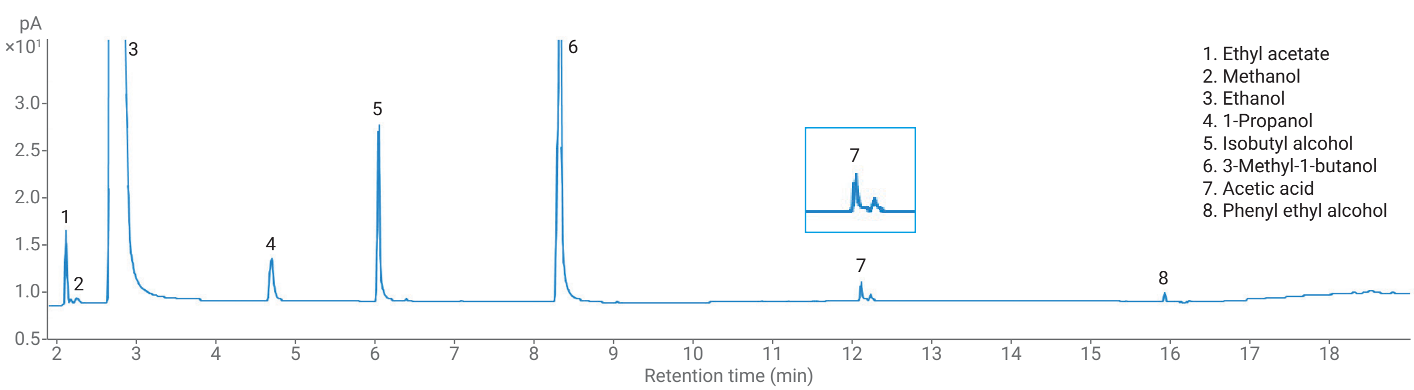

Figure 3. Analysis of Woodford Reserve bourbon whiskey on a 0.180 mm id column with helium carrier gas.

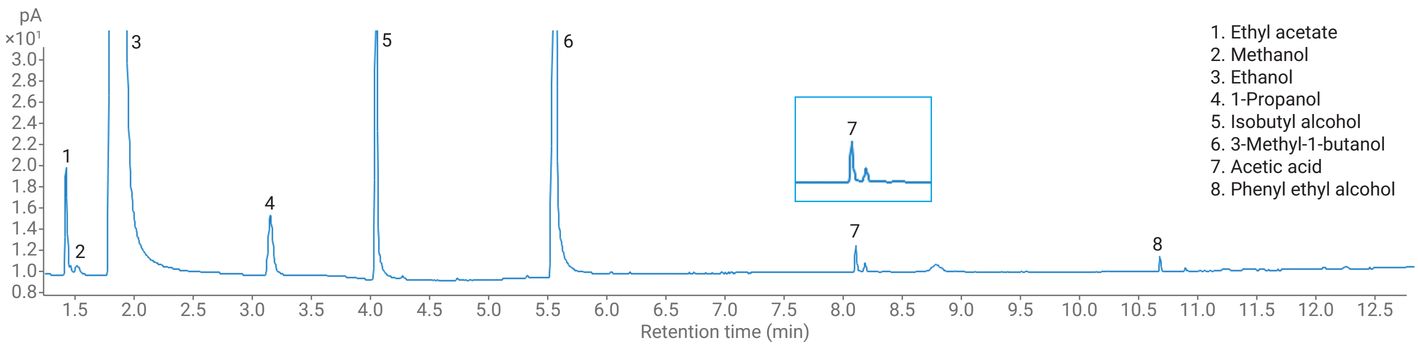

Figure 4. Analysis of Woodford Reserve bourbon whiskey on a 0.180 mm id column with hydrogen carrier gas.

Conclusion

Analysis of aqueous samples, such as distilled spirits, provides a unique challenge to GC. The 8890 GC system with a J&W DB-WAX UI column demonstrates the capability of an inert flowpath to deliver repeatable results over the course of 400 injections of aqueous samples. Simple steps can also be taken with the aid of method translation and a smaller id column to help reduce analysis time and improve sample throughput.

References

1. Analysis of Distilled Spirits Using an Agilent J&W DB-WAX Ultra Inert Capillary GC Column. *Agilent Technologies Application Note*, publication 5991-6638EN (2016)
2. Ng, L.; Lafontaine, P.; Harnois, J. Gas Chromatographic-Mass Spectrometric Analysis of Acids and Phenols in Distilled Alcohol Beverages. Application of Anion-Exchange Disk Extraction Combined with In-Vial Solution and Silylation, *J. Chromatogr. A* **2000**, *873(1)*, 29–38.
3. MacNamara, K.; Lee, M.; Robbat Jr., A. Rapid Gas Chromatographic Analysis of Less Abundant Compounds in Distilled Spirits by Direct Injection with Ethanol-Water Venting and Mass Spectrometric Data Deconvolution. *J. Chromatogr. A* **2010**, *1217(1)*, 136–142.
4. Fitzgerald, G.; *et al.* Characterization of Whiskeys Using Solid-Phase Microextraction with Gas Chromatography-Mass Spectrometry. *J. Chromatogr. A* **2000**, *896(1–2)*, 351–359.

www.agilent.com/chem

Printed in the USA, December 7, 2018
5994-0487EN

Agilent
Trusted Answers