

AGILENT INTUVO 9000

Gas Chromatographic Analysis of Fusel Oils in Distilled Alcoholic Beverages



Introduction

Consumer alcoholic beverages are made by naturally fermenting grains or fruits. In addition to the desired ethanol formation, natural fermentation also produces smaller quantities of C₃ to C₅ alcohols collectively known as fusel oils. Specifically, fusel oils are *n*-propanol, *n*-butanol, isobutanol, and 2-methyl-1-butanol (amyl alcohol). The presence of fusel oils, along with methanol and ethyl acetate, in an alcoholic beverage indicates a naturally fermented product. The absence of these compounds means the product may not be naturally sourced. This distinction is important when assessing taxes or tariffs during commercial transactions. In addition to regulatory concerns, the presence of small quantities of fusel oil can contribute to the flavor profile of the beverages. Higher concentrations are thought to contribute to the intensity of hangovers, the adverse physiological effects associated with excess alcohol consumption.

The analysis of fusel oils in distilled alcoholic beverages is performed using gas chromatography (GC). The United States Treasury's Alcohol and Tobacco Tax Bureau publishes method TM:200 for the capillary GC measurement of fusel oils¹. This method is scoped to measure not only fusel oil, but other naturally occurring compounds found in alcoholic beverages as well as some illegal adulterants. This paper describes the analysis of fusel oils, methanol, and ethyl acetate in distilled alcoholic beverages.

TM:200 is a simple GC method in both configuration and operation. However, since fusel oils are very polar compounds, quality results require an inert system that will not cause excess peak tailing. The Agilent Intuvo 9000 Gas Chromatograph is ideally suited for this application due to the Agilent Intuvo Inert Flow path incorporated into the instrument. Combined with the simple user interface and advanced built-in diagnostics, the Intuvo 9000 GC ensures high quality results when measuring fusel oil components in a variety of alcoholic beverages.

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Methodology

Sample preparation

Four calibration standards containing varying amounts of ethyl acetate, *n*-propanol, *i*-butanol, *n*-butanol, amyl alcohol, and methanol were made according to the requirements of the US Treasury TM:200 method. All standards were prepared in a solvent mixture of 40 volume percent (vol%) ethanol/water. These standards were used to quantify fusel oils in five different distilled spirits: Kentucky bourbon whiskey, premium Kentucky bourbon whiskey, American rye whiskey, Russian vodka, and Chinese baijiu. Both Kentucky whiskeys were made from corn and wheat, and all whiskey samples were aged in oak barrels for 2 to 10 years. The Russian vodka was made from wheat/rye, and the Chinese baijiu was made from sorghum. These two spirits were not aged in wood. All samples were analyzed neat without any additional preparation.

Results and Discussion

Figure 1 shows the fusel oil content in three different distilled spirits. Figure 2 shows a comparison of the fusel oil content in three American aged whiskeys. Table 1 lists the retention times for each target compound. After calibration, the individual fusel oil compounds were quantified and reported according to the requirements of the TM:200 method. Table 2 shows the results.

From the results shown in Figures 1 and 2, and Tables 1 and 2, several interesting features are observed. First, no fusel oil components could be detected in the vodka sample using this method. This result could allow this vodka sample to be classified as a neutral spirit by the US Treasury Department for the imposition of tariffs and taxes.

GC Instrument conditions

Parameter	Value
Inlet	split/splitless 220 °C, 25:1 split
Injection volume	1 µL
Column	Agilent HP-Innowax, 30 m × 0.25 mm, 0.25 µm
Constant column flow	1.8 mL/min hydrogen
Column temperature	40 °C for 5 minutes 12 °C/min to 215 °C 215 °C hold 5 minutes
Detector	flame ionization 250 °C

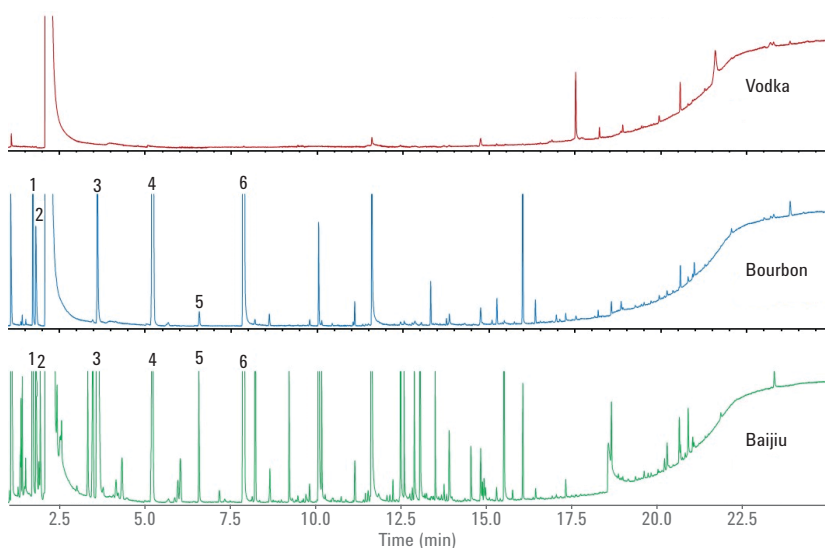


Figure 1. Fusel oil content in three types of distilled spirits.

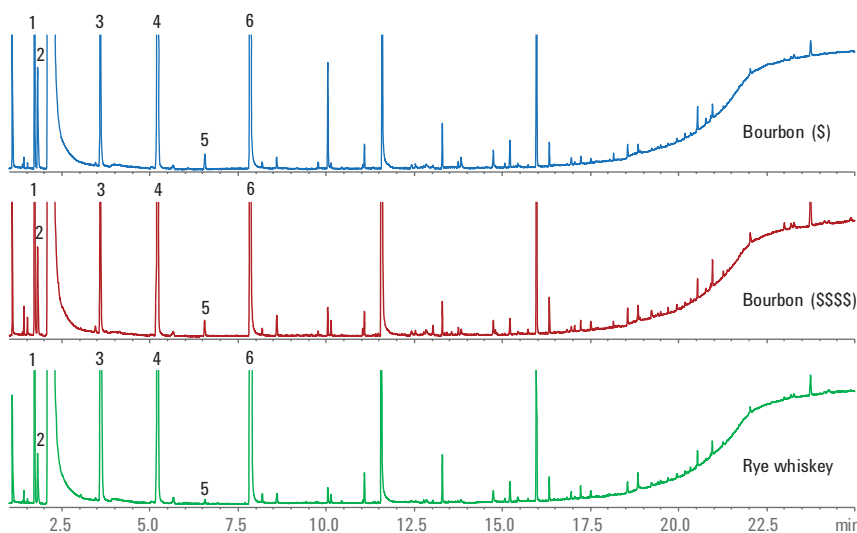


Figure 2. Fusel oil content in three types of American whiskey.

Table 1. Retention times.

Peak no.	Compound	RT (min.)
1	ethyl acetate	1.716
2	methanol	1.815
3	<i>n</i> -propanol	3.603
4	<i>i</i> -butanol	5.240
5	<i>n</i> -butanol	6.572
6	amyl-alcohol	7.850

Table 2. Fusel oil results for five distilled spirits.

	Bourbon (\$)	Bourbon (\$\$\$\$)	Rye	Baijui	Vodka
ethyl acetate (g/100 L)	41.0	67.6	41.8	224.2	0.0
methanol (vol %)	0.001	0.001	0.001	0.002	0.000
<i>n</i> -propanol (g/100 L)	11.2	10.0	20.2	148.2	0.0
<i>i</i> -butanol (g/100 L)	18.8	32.9	53.7	19.1	0.0
<i>n</i> -butanol (g/100 L)	0.5	0.6	0.2	5.7	0.0
amyl alcohol (g/100 L)	151.0	162.4	138.2	49.1	0.0
total fusel oil (g/100 L)	181.6	205.9	212.3	222.1	0.0

The next observation is the overall complexity of the Chinese baijui sample compared to the other samples in regards to the volatile compounds detected by this analysis. While the baijui sample contained similar amounts of total fusel oil, the amount of *n*-propanol and ethyl acetate was considerably higher, but the amount of amyl alcohol was considerable lower compared to the whiskey samples.

Finally, despite the difference in costs, the American whiskey samples were remarkably similar to each other in regards to the fusel oil content and the overall profile of volatile compounds detected. While fusel oils may contribute to the flavor profile of these whiskeys, other nonvolatile compounds not detected by this method could well contribute to the perceived differences in quality of these whiskeys.

Conclusion

The simple analysis of fusel oils using the US Treasury Method TM:2000 is easily done using the Agilent Intuvo 9000 GC system. The Agilent Intuvo Inert Flow path provides excellent peak shape for very polar compounds, such as methanol and fusel oil alcohols, thus ensuring consistent, accurate results.

Reference

1. SSD: TM:200, Capillary GC Analysis of Fusel Oils and Other Components of Interest, revision 7, United States Department of the Treasury, Alcohol and Tobacco Tax and Trade Bureau, Scientific Services Division, October 14, **2014**.

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Published in USA, March 1, 2017
5991-7897EN



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