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Application News

No. G315A

Gas Chromatography

Analysis of Ethanol in Liquors Using Nexis™ GC-2030

The Japanese National Tax Agency (NTA) prescribes analytical methods for ethanol in liquors, which are official methods. Various analytical methods are included, and the prescribed GC method is isothermal gas chromatography with a column oven condition at 50 °C. However, since the water and high-boiling-point components in the liquor are not eluted from the column during isothermal analysis at 50 °C, it is not possible to perform a favorable analysis without expelling them by raising the oven temperature after the elution of ethanol and internal standard substances. In addition, since the major component of liquors is moisture, the amount and position of the wool in the insert need to be fine-tuned. In this article, the ethanol in samples of beer, liqueur and Japanese sake was quantified using a method that made it possible to perform analysis in accordance with the NTA's ethanol analysis favorably in a short time.

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Reagent Preparation and Quantification Method

As the standard solutions, ethanol was diluted with water and prepared at concentrations of 5%, 10%, 15%, and 20% (v/v). In addition, as an internal standard solution, isopropyl alcohol was dissolved in water and prepared at 2% (v/v). 0.9 mL of the internal standard solution was added to 0.1 mL of each ethanol standard solution to make the standard samples that were analyzed to create the calibration curve.

Actual samples of beer, liqueur, and Japanese sake available on the market were prepared and, in the same manner as above, 0.9 mL of the internal standard solution was added to 0.1 mL of each actual sample. These samples were analyzed, and the ethanol was quantified using the created calibration curve.

Wool in Insert, and Syringe

When analyzing samples containing a lot of water, such as alcoholic beverages, it can be difficult to analyze them with good repeatability. By making the amount of silica wool in the insert greater than normal (10 mg) and setting the wool packing position slightly above the standard position (22 mm from the top) as shown in the figure below, it is expected that repeatability can be improved. In addition, when samples in aqueous solution are analyzed with a standard syringe for AOC, the plunger motion may become slow during analysis, which affects the repeatability. Using an elastic syringe for AOC (P/N: 221-49548) equipped with a plunger made of titanium enables stable sample introduction.

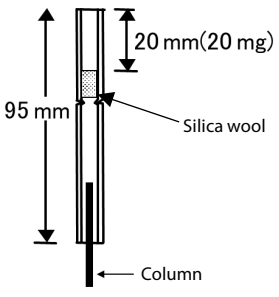

Fig. 1 Position and Quantity of Silica Wool in the Insert

Analysis Conditions

Table 1 lists the configuration of the instrument used for analysis and the analysis conditions.

Table 1 Instrument Configuration and Analysis Conditions

Main Unit	:	Nexis GC-2030 /AOC-20i plus
Column	:	SH-1 (I.D. 0.53 mm × 30 m, df=3.00 µm) *1
Detector	:	FID-2030
Injection Volume	:	1 µL
Injection Mode	:	Split
Split Ratio	:	1 : 40
Injection Unit Temp	:	250 °C
Carrier Gas	:	He
Carrier Gas Control	:	Pressure
Pressure Program	:	28 kPa (3 min) - 300 kPa/min - 90 kPa (6.79 min)
Column Temp.	:	50 °C (3 min) - 40 °C/min - 200 °C - 25 °C/min - 245 °C (1.45 min)
Detector Temp.	:	250 °C
Detector Gas	:	Make up (He) 24 mL/min H_2 32 mL/min Air 200 mL/min

*1 P/N: 221-75733-30

Chromatograms and Calibration Curve for Standard Samples

The chromatograms of the standard samples are shown in Fig. 2, and the calibration curve is shown in Fig. 3.

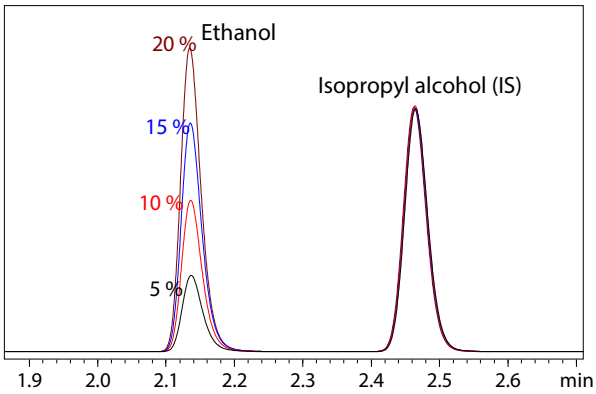

Fig. 2 Chromatograms of Standard Samples

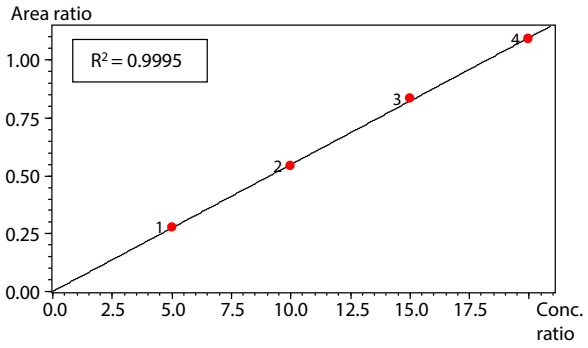

Fig. 3 Calibration Curve

Analysis of Actual Samples and Ethanol Quantification Results

The chromatograms of the actual samples are shown in Fig. 4, and the quantitative results and repeatability (n=10) are shown in Table 2. It was confirmed that the high-boiling-point components in each liquor were eluted after the elution of ethanol and isopropyl alcohol.

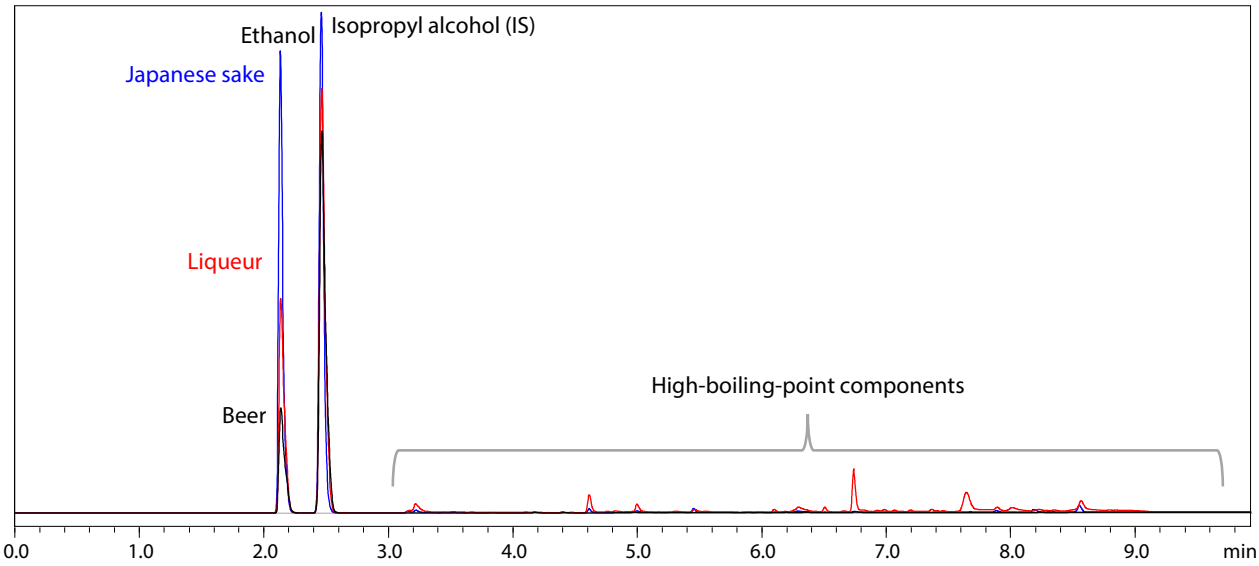

Fig. 4 Chromatograms of Actual Samples (Japanese Sake, Liqueur, Beer)

Table 2 Quantitative Results and Repeatability

	Beer	Liqueur	Japanese sake
Concentration value for first analysis (%)	4.699	8.785	15.446
Concentration value for second analysis (%)	4.701	8.789	15.443
Concentration value for third analysis (%)	4.699	8.783	15.442
Concentration value for fourth analysis (%)	4.700	8.783	15.444
Concentration value for fifth analysis (%)	4.695	8.782	15.447
Concentration value for sixth analysis (%)	4.699	8.778	15.459
Concentration value for seventh analysis (%)	4.697	8.781	15.435
Concentration value for eighth analysis (%)	4.695	8.786	15.455
Concentration value for ninth analysis (%)	4.691	8.772	15.437
Concentration value for tenth analysis (%)	4.697	8.760	15.439
Average concentration value (%)	4.697	8.780	15.445
%RSD	0.073	0.096	0.048

Conclusion

Analysis conditions for quickly expelling unnecessary components after the elution of ethanol and isopropyl alcohol (IS) allowed us to analyze ethanol in beer, liqueur, and Japanese sake in a short period of time with good repeatability.

* Attention
In the ethanol analysis prescribed by the National Tax Agency, the principles of analysis are different between the GC method and the other analytical methods, so the quantitative values may be different. Please be careful when changing the analytical method.

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