

Application News

Analysis of Ethanol in Alcoholic Beverages Using the Nexis GC-2060

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User Benefits

- ◆ The Nexis GC-2060, equipped with a newly developed FID, provides excellent linearity for high-concentration samples.
- ◆ With rapid oven heating and cooling, the Nexis GC-2060 delivers accurate, reproducible results during temperature ramping to elute high-boiling compounds.
- ◆ A special liner for aqueous solutions and an Xtra Life Microsyringe enable reliable, repeatable results even with aqueous samples.

Introduction

The Japanese National Tax Agency (NTA) prescribes the official analytical methods for ethanol in alcoholic beverages. Among these, the gas chromatography method specifies an isothermal oven temperature of 50 °C. However, at 50 °C, water and high-boiling compounds in alcoholic beverages do not elute from the column. Therefore, after the target analyte (ethanol) and the internal standard have eluted, the oven temperature must be increased to expel the water and high-boiling compounds from the column.

In GC analysis of samples containing a high water content (e.g., alcoholic beverages), sample vaporization may become unstable, leading to distorted peak shapes and poor repeatability. In addition, when a standard micro-syringe for an AOC autosampler is used with aqueous solutions, the plunger may become heavy during aspiration/dispensing, which can also degrade repeatability.

This article introduces an example of ethanol analysis in alcoholic beverages using the Nexis GC-2060 equipped with a newly developed FID. The Nexis GC-2060 can be configured with the new, highly sensitive FID, which offers excellent linearity. In addition, its rapid oven heating and cooling enable high-boiling compounds to be quickly expelled from the column, thereby shortening the overall analysis time. Furthermore, using a special liner for aqueous solutions and the Xtra Life Microsyringe, both of which are effective for aqueous samples, enabled highly repeatable analysis.

Nexis GC-2060 and the Newly Developed FID

The Nexis GC-2060 gas chromatograph combines world-class analytical performance with workflow optimization based on the latest technologies. Rapid oven heating and cooling shorten analysis cycle times and improve throughput.

The instrument can also be equipped with a newly developed FID (Fig. 1), which offers world-class sensitivity and improved linearity.



Fig. 1 Appearance of the Nexis™ GC-2060 (Left) and the Newly Developed FID (Right)

Special Liner for Aqueous Solutions and Syringe

In this application, a special liner for aqueous solutions (P/N: 227-35015-01, Fig. 2, left) was used. This liner stabilizes sample vaporization, enabling good peak shapes and high repeatability for aqueous samples.

For sample introduction, an Xtra Life Microsyringe (P/N: 227-35400-01, Fig. 2 right) was used. The Xtra Life Microsyringe employs a flexible titanium alloy plunger, enabling stable introduction of aqueous samples.



Fig. 2 The Special Liner for Aqueous Solutions (Left) and Xtra Life Microsyringe (Right)

Sample Preparation and Quantification Method

Standard solutions were prepared by diluting ethanol with water to 5 %, 10 %, 15 %, and 20 % (v/v). In addition, an internal standard solution of isopropyl alcohol was prepared by dissolving it in water to a concentration of 2 % (v/v). 0.9 mL of the internal standard solution was added to 0.1 mL of each ethanol standard solution to prepare standard samples that were analyzed to create the calibration curve.

Commercially available beer, liqueur, fruit wine, and Japanese sake were used as samples. For each, 0.9 mL of the internal standard solution was added to 0.1 mL of the sample as described above. These samples were analyzed, and the ethanol was quantified using the calibration curve.

Analysis Conditions

Table 1 shows the instrument configuration and analysis conditions. Following the isothermal step at 50 °C as specified in the prescribed method, the oven temperature and inlet pressure were increased to elute water and high-boiling components rapidly.

Table 1 Instrument Configuration and Analysis Conditions

Main Unit:	Nexis GC-2060/AOC-30i	
Column:	SH-1 (I.D. 0.53 mm × 30 m, df = 3.00 μm) ^{*1}	
Detector:	FID	
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 (N ₂)	30 mL/min
	H ₂	40 mL/min
	Air	170 mL/min

*1 P/N: 221-75733-30

■ Chromatograms and Calibration Curve of the Standard Samples

The chromatograms of the standard samples are shown in Fig. 3. The ethanol and isopropyl alcohol peaks were completely separated.

Fig. 4 shows the calibration curve prepared using the standard samples. The R^2 value of the calibration curve was 0.9999 or higher, demonstrating excellent linearity and confirming the new FID's high linearity.

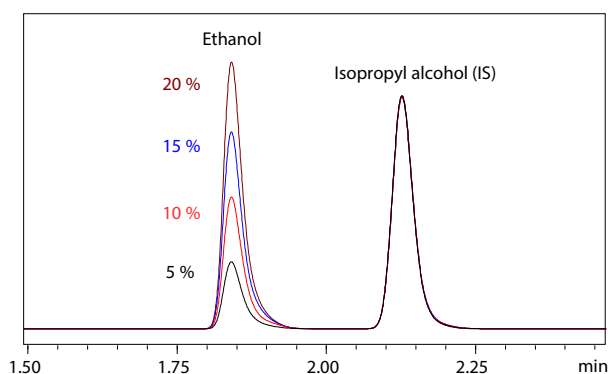


Fig. 3 Chromatograms of the Standard Samples

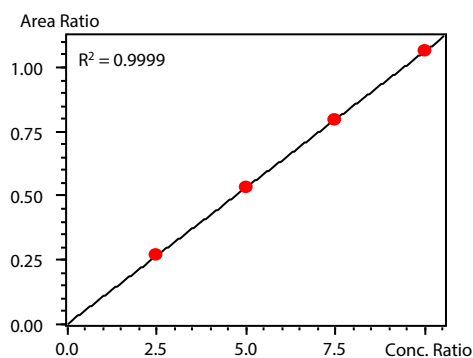


Fig. 4 Calibration Curve

■ Chromatograms of Actual Samples

Fig. 5 shows chromatograms of the actual liquor samples. After elution of ethanol and isopropyl alcohol, many peaks were observed, confirming that high-boiling compounds in each liquor were eluted from the column.

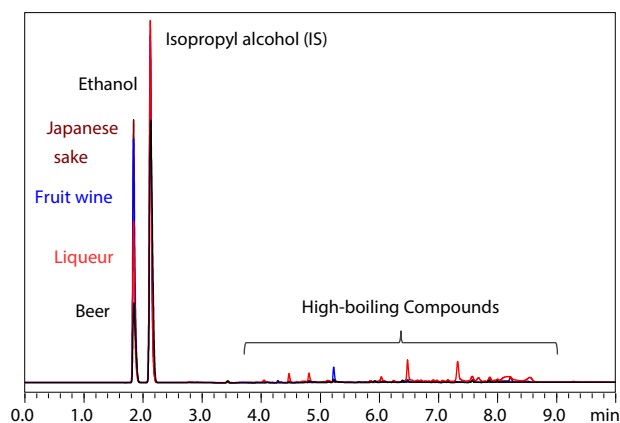


Fig. 5 Chromatograms of Actual Samples

■ Quantitative Results and Repeatability

Table 2 shows the ethanol concentrations (v/v%) quantified for each liquor sample and the repeatability of quantitative values (%RSD, $n = 10$). Excellent repeatability was obtained regardless of the alcoholic beverage type, with %RSD ranging from 0.038 to 0.15. Although repeatability tends to deteriorate in aqueous solution analysis, high repeatability was achieved in this application by using a liner and micro-syringe suitable for aqueous samples.

In the Japanese National Tax Agency's designated method for ethanol analysis, accuracy is specified as follows (excluding samples around 1 v/v% alcohol content):

- Perform approximately 10 replicate measurements and keep the coefficient of variation of quantitative values within 1 %.

Based on the results in Table 2, it was confirmed that these criteria were sufficiently satisfied.

Table 2 Ethanol Concentration (v/v%) and Repeatability in Each Alcoholic Beverage Sample

Run	Beer	Liqueur	Fruit wine	Japanese sake
1	5.315	7.648	12.219	14.193
2	5.323	7.660	12.226	14.202
3	5.326	7.663	12.230	14.194
4	5.330	7.665	12.234	14.185
5	5.332	7.664	12.232	14.194
6	5.335	7.670	12.232	14.187
7	5.336	7.666	12.229	14.191
8	5.337	7.674	12.240	14.196
9	5.339	7.673	12.242	14.193
10	5.342	7.678	12.247	14.201
Average Concentration Value (%)	5.332	7.666	12.233	14.194
%RSD	0.15	0.11	0.067	0.038

■ Conclusion

This article demonstrated the analysis of ethanol in alcoholic beverages using the Nexis GC-2060. The calibration curve created from the standard samples showed an R^2 value greater than 0.9999, confirming the excellent linearity of the newly developed FID. In addition, rapid oven heating enabled the removal of unwanted high-boiling components, enabling analysis to be completed in a short time. For the quantification of ethanol in commercially available alcoholic beverages, repeatability met the precision criteria of the designated method, confirming the effectiveness of the special liner for aqueous solutions and the Xtra Life Microsyringe.

Note: 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 exercise caution when changing the analytical method.

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