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Application News

GC Nexis™ GC-2030

Importance of Liner Selection in Aqueous Solution Analysis

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User Benefits

- A special liner has been developed for aqueous solutions that is effective for GC analysis using water as a solvent.
- By using the special liner for aqueous solutions, it is possible to obtain data with good repeatability.

■ Introduction

When a sample containing a lot of water is analyzed by GC, peak shapes become abnormal, and it can be difficult to analyze with good repeatability. This phenomenon is related to sample vaporization, and it is known that the analysis results differ depending on the type of liner used. Therefore, a special liner has been developed for aqueous solutions (P/N: 227-35015-01) that is optimal for analysis using water as a solvent (Fig. 1).

Examples of aqueous solution analysis include measurement of ethanol concentration in sanitizer gel in accordance with USP<611> (Application News No. G333) and analysis of ethanol in liquor (Application News No. G315). This article introduces the effectiveness of a special liner for aqueous solutions that was confirmed by two types of analyses.

Fig. 1 Special Liner for Aqueous Solutions

■ Special Liner for Aqueous Solutions

The way the sample vaporizes in the GC sample injection unit depends on the characteristics of the sample and the solvent used. In addition, the type of liner used (liner shape and material, wool material and filling amount, etc.) affects the analysis results because the thermal conductivity during sample vaporization differs depending on the material of wool at the vaporization point.

In the newly developed special liner for aqueous solutions, the amount of glass wool in the liner (20 mg) is greater than the normal 10 mg, and the wool packing position is set 20 mm from the top, which is slightly above the standard position (22 mm from the top) as shown in Fig. 2. It is expected that sample vaporization is stable, and repeatability can be improved.

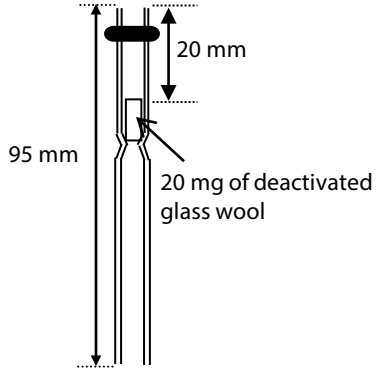

Fig. 2 Position and Quantity of Wool in the Liner

■ Evaluation Method and Analysis Conditions

With reference to the analysis of ethanol concentration in sanitizer gel (Application News No. G333) and ethanol in liquor (Application News No. G315), the special liner for aqueous solutions was evaluated by the following procedure.

○ Evaluation A

Using a standard liner of GC-2030 (P/N: 227-35007-01) and a special liner for aqueous solutions, peak shapes and the separation of the target compound and water (solvent) were compared. Unlike the actual analysis, the detector used was a TCD, which can detect water as a peak. (Detector used for the actual analysis: FID)

○ Evaluation B

The results of the above two types of analysis were compared using standard and special liners.

For sample preparation and analysis conditions, each application news was referred to. Table 1 shows the equipment configuration and analysis conditions.

Table 1 Instrument Configuration and Analysis Conditions

Common instrument configuration and analysis conditions	
Model	: Nexis GC-2030 + AOC™-30i
Evaluation A (TCD)	
Detector	: Thermal conductivity detector (TCD)
Current	: 50 mA
Evaluation B (FID)	
Detector	: Flame ionization detector (FID)
Detector Gas	: H_2 32 mL/min , Air 200mL/min
Make up Gas	: He 24 mL/min
Syringe	: Shimadzu Xtra Life Microsyringe 10 µL (P/N: 227-35400-01)*1
<Determination of Alcohol Concentration in Sanitizer Gel>	
Column	: SH-624 (0.53 mm I.D. × 30m, d.f.= 3 µm)
Column Temperature	: 50 °C (5 min) – 10 °C/min – 200 °C (4 min)
Injection Temperature	: 210 °C
Injection Mode	: Split (split ratio 5)
Carrier Gas Controller	: Liner velocity (He)
Liner Velocity	: 34 cm/sec
Detector Temperature	: FID 280 °C, TCD 210 °C
Injection Volume	: 0.2 µL
<Analysis of Ethanol in Liquor>	
Column	: SH-1 (0.53 mm I.D. × 30m, d.f.= 3 µm)
Column Temperature	: 50 °C (3 min) – 40 °C/min – 200 °C – 25 °C/min – 245 °C (1.45 min)
Injection Temperature	: 250 °C
Injection Mode	: Split (split ratio 40)
Carrier Gas Controller	: Pressure (He)
Pressure Program	: 28 kPa (3 min) - 300 kPa/min - 90 kPa (6.79 min)
Detector Temperature	: FID 250 °C, TCD 250 °C
Injection Volume	: 1 µL

*1 When samples in aqueous solution are analyzed with a standard syringe for AOC, the plunger motion may become dull during analysis, which affects repeatability. Using an Xtra Life Microsyringe equipped with a plunger made of titanium enables stable sample introduction.

■ Evaluation A Results

Using a standard liner and a special liner for aqueous solutions, the peak shapes detected were confirmed using TCD in two types of aqueous solutions analysis.

Examples of measurement results are shown in Figs. 3 and 4.

(1) shows the results when using a special liner for aqueous solutions, and (2) shows the results when using a standard liner. Using the standard liner, the irregular peak shape (tailing) was observed as shown in (2), and the peak shape of water (solvent) eluted at this time was found to be wider than normal. The occurrence of such irregular peak shapes can lead to poor repeatability.

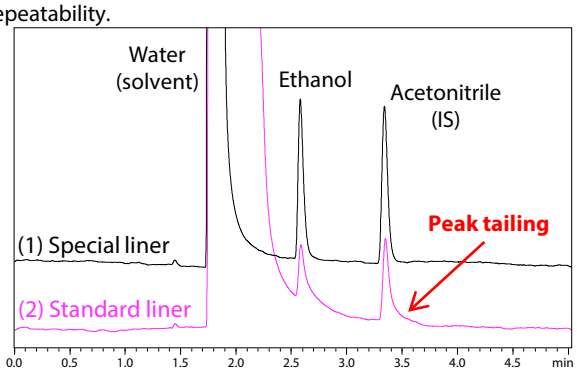

Fig. 3 Standard Solution Results for Alcohol Concentration (TCD)

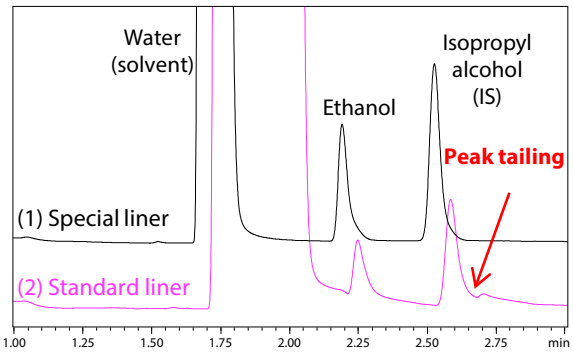

Fig. 4 Results for 10 % Standard Solution in Analysis of Ethanol in Liquor (TCD)

■ Evaluation B Results: Measurement of Alcohol Concentration

System suitability requirements and repeatability were verified using standard and special liners. Fig. 5 shows the chromatograms of the standard and sample solution using the special liner for aqueous solutions, and the calibration curve is shown in Fig. 6. The measurement results for each liner are summarized in Table 2.

When using the special liner for aqueous solutions, system suitability requirements were satisfied, and good repeatability was obtained.

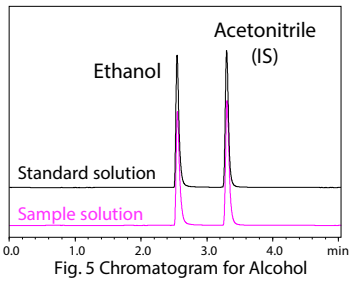

Fig. 5 Chromatogram for Alcohol Concentration Measurement (FID)

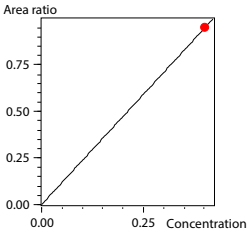

Fig. 6 Calibration Curve

Table 2 Analysis Results for Alcohol Concentration Measurement*2

	Special liner	Standard liner*3	System Suitability
Standard solution			
Area %RSD (n=10)	1.441	16.339	—
Area ratio %RSD (n=6)	0.148	4.102	4.0 % or less (n=6)
Resolution (USP)	8.264	9.708	4 or more
Tailing factor	1.742	1.706	2.0 or less
Sample solution			
Area %RSD (n=10)	1.685	4.098	—
Area ratio %RSD (n=10)	0.305	1.312	—

*2 The values shown are reference values, not guaranteed values. The nonconformity values of the system suitability test and the %RSD value of 2 or more are indicated in red.

*3 The result using the standard liner is an example, and loss of repeatability may not always occur.

■ Evaluation B Results: Ethanol in Liquors

Repeatability was confirmed using standard and special liners. Fig. 7 shows the chromatogram of the standard solution using the special liner, Fig. 8 shows the calibration curve, and Fig. 9 shows the chromatogram of the sample solution. The analysis results for each liner are shown in Table 3.

The special liner for aqueous solutions showed good repeatability for all samples.

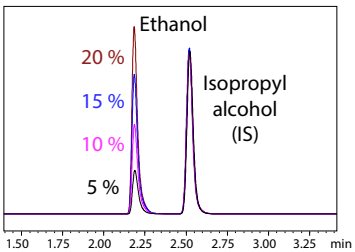

Fig. 7 Chromatogram of Standard Solution (FID)

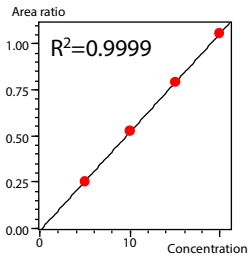

Fig. 8 Calibration Curve

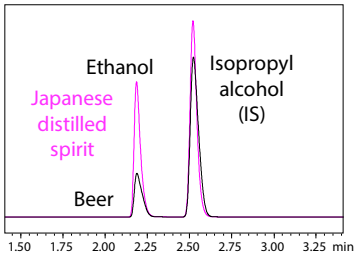

Fig. 9 Chromatogram of Sample Solution (FID)

Table 3 Analysis Results of Ethanol in Liquors *2

			Special liner	Standard liner*3
Area %RSD (n=10)	Standard solution	5 %	0.671	2.096
		10 %	1.061	9.358
		15 %	0.815	0.486
		20 %	1.575	0.891
	Sample solution	Beer	0.483	2.421
		Japanese distilled spirit	0.419	1.184
Area ratio %RSD (n=10)	Standard solution	5 %	0.075	0.116
		10 %	0.079	3.624
		15 %	0.078	0.138
		20 %	0.110	0.106
	Sample solution	Beer	0.332	0.223
		Japanese distilled spirit	0.061	0.108

■ Conclusion

Stable analysis can be performed by using a special liner for aqueous solutions. When using a special liner for aqueous solutions repeatedly, it is recommended that it is replaced after 50 to 100 injections.

01-00328-EN First Edition: Mar. 2022

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