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Application News

No. G332

Gas Chromatography

Packed Column Analysis of Alcohol Number Determination Conforming to Japanese Pharmacopoeia Using Nexis™ GC-2030 (FID)

The methods of alcohol number determination provided in the Japanese Pharmacopoeia (JP) are Method 1 Distilling method and Method 2 Gas chromatography as for the tincture or other preparations containing ethanol. In these methods, the alcohol number is determined by reading the number of milliliters (mL) of ethanol distillate obtained from 10 mL of a sample measured at 15 °C. In the gas chromatography method, a packed column is used.

This article introduces an example of alcohol number determination as provided in the JP using a Nexis GC-2030 gas chromatograph, which now also supports packed column analysis.

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Packed Column Support System of GC-2030

As previously announced in Application News No. G320, 321, 322 and 323, the Nexis GC-2030 now supports packed column analysis. The compatible detectors are the flame ionization detector (FID) and the thermal conductivity detector (TCD).

It is possible to install the glass columns used with the GC-17 and GC-2010. If an SUS column is to be used, it is possible to install all SUS columns used with the GC-17, GC-2010, GC-14 and GC-2014, and the parts can also be shared.

The FID-2030 can also be switched to packed column use or capillary column use by a simple modification using the FID-2030Packed kit (P/N: S221-85191-41).

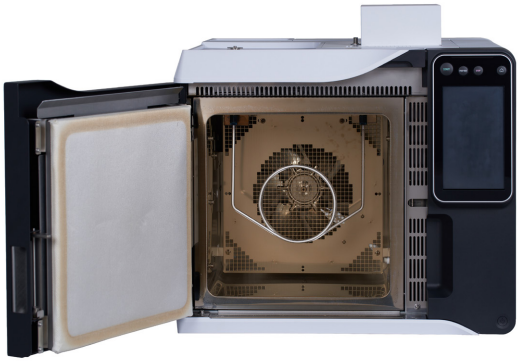

Fig. 1 Example of Installation of Glass Column

Support System for Analysis of Ethanol for Disinfection with One Instrument

As analyses of ethanol for disinfection, the JP provides methods for alcohol number determination using a glass packed column and purity testing of ethanol for disinfection using a capillary column. Because the Nexis GC-2030 supports both capillary analysis and packed analysis, it is possible to support both types of analysis of ethanol for disinfection with a single instrument, thereby saving installation space while also contributing to higher productivity.

Sample Preparation

Samples for analysis by gas chromatography were prepared in accordance with the JP. For details, please refer to the Japanese Pharmacopoeia, "General Tests, Processes and Apparatus, 1.01 Alcohol Number Determination."

In preparing the sample solution, water was added to a volume of sample equivalent to 5 mL of ethanol to make 50 mL, 10 mL of an internal standard solution (aqueous acetonitrile) was added to 25 mL of the ethanol aqueous solution, and water was then added to make 100 mL.

The standard solution was prepared by adding water to 5 mL of ethanol to make 50 mL, adding 10 mL of the internal standard solution to 25 mL of this solution, and then adding water to make 100 mL.

The vials containing the sample solution and the standard solution were immersed for 30 min in water, which was allowed to stand at room temperature for 1 h or more, and 1 mL of the headspace was injected into the GC.

In this measurement, the alcohol number was obtained by measuring simulated samples prepared with ethanol concentrations of 60, 70 and 80 (vol%).

Analysis Conditions

Table 1 shows the instrument configuration and analysis conditions in this measurement.

As provided in the JP, the column flow rate was adjusted so that the retention time of ethanol was 5 to 10 min, and a column with resolution of not less than 2.0 for ethanol and the internal standard substance (acetonitrile) was used.

Table 1 Instrument Configuration and Analysis Conditions

Model	: Nexis GC-2030 +SINJ-2030+FID-2030Packed Kit
Injection mode	: Direct
Injection volume	: 1.0 mL
Injection temp.	: 110 °C
Carrier gas	: N_2
Carrier gas control	: 35 mL/min (reference value) *
Column	: Porapak™ Q 80/100 (1.5 m × 3 mm I.D.)
Column temp.	: 110 °C
Detector	: Flame Ionization Detector (FID)
Detector temp.	: 150 °C
Detector gas	: H_2 32.0 mL/min, Air 200 mL/min Make up: 5 mL/min

* The flow rate will vary depending on the condition of the column.

Results of Analysis of Standard Solution

Fig. 2 shows the chromatogram of the standard solution.
Resolution for ethanol and acetonitrile was 2.56.

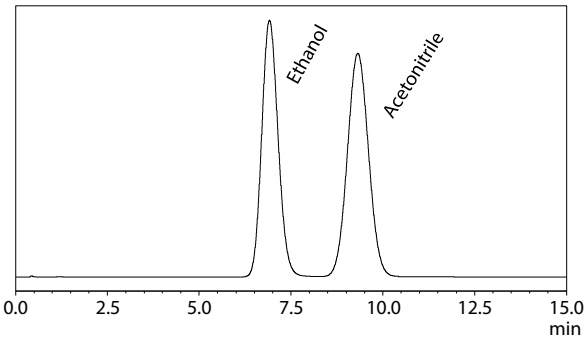

Fig. 2 Chromatogram of Standard Solution

Table 2 shows the ratio Q_S of the peak height of ethanol to the peak height of the internal standard solution (acetonitrile) obtained as a result of 5 continuous measurements.

Table 2 Q_S of 5 Measurements

n=1	1.149
n=2	1.148
n=3	1.149
n=4	1.149
n=5	1.149

Results of Analysis of Sample Solutions

Fig. 3 shows the overwritten chromatograms of the sample solutions with ethanol contents of 60 %, 70 % and 80 %.

Note) Because the sampled amount according to the JP is "a volume of sample equivalent to 5 mL of ethanol," the intensities of the peaks after preparation of the sample solutions are comparatively similar.

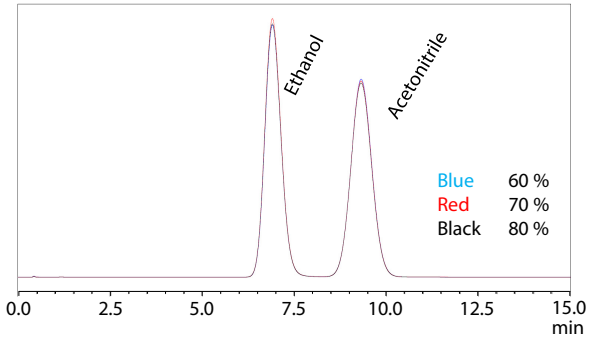

Fig. 3 Chromatogram of Sample Solutions

Alcohol Number

Table 3 shows the alcohol number of each sample solution obtained from the measurement results.

Conclusion

Alcohol number determination using a packed column as provided in the Japanese Pharmacopoeia was carried out with a Shimadzu Nexis GC-2030.

Because the FID of the Nexis GC-2030 can support both packed column analysis and capillary column analysis with a simple modification, it is possible to support both ethanol purity testing (using a capillary column) and alcohol number determination (using a packed column) as provided in the JP with a single instrument.

For ethanol purity testing, please refer to Application News No. G331, "Analysis of Volatile Impurities in Anhydrous Ethanol and Ethanol for Disinfection in Accordance with the Purity Test set by the Pharmacopoeias (JP, USP, EP)."

Table 3 Alcohol Number of Sample Solutions

Ethanol concentration of sample solution	Q_T *1	Q_S Average value (n = 5)	Sample volume (mL)	Ethanol content (vol%) *2	Alcohol number *3
60 %	1.273	1.149	9	99.5	6.51
70 %	1.318	1.149	8	99.5	7.59
80 %	1.306	1.149	7	99.5	8.59

*1 Q_T is the ratio of the peak height of ethanol to the peak height of the internal standard substance (acetonitrile) of the sample solution.
*2 Ethanol content (vol%) indicates the content of ethanol for alcohol number determination used in preparation of the standard solution.
*3 The alcohol number is obtained by the following equation.

$$\text{Alcohol number} = \frac{Q_T}{Q_S} \times \frac{5(mL)}{\text{Volume of sample}(mL)} \times \frac{\text{Content}(\text{vol}\%)\text{of ethanol}(C_2H_5OH)\text{in ethanol for alcohol number determination}}{9.406}$$

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