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Application News

No.G282A

Gas Chromatography

Analysis of Ethylene Glycol and Diethylene Glycol in Propylene Glycol

An amendment (Supplement Ⅱ) to the Japanese Pharmacopoeia 16th Edition (the Japanese Ministry of Health, Labour and Welfare (MHLW) Notification No. 65, 2011) was announced in the MHLW Notification No. 47. The change became effective on the same day.
With this partial revision of the Pharmacopoeia, the Purity section was amended in monographs pertaining to propylene glycol, and specifications related to ethylene glycol, diethylene glycol and related substances were added.
In this Application News, we introduce an analysis of ethylene glycol, diethylene glycol and related substances in propylene glycol in accordance with this supplement to the Japanese Pharmacopoeia.

System Suitability

The Japanese Pharmacopoeia 16th Edition Supplement Ⅱ stipulates the system performance verification as follows:
System performance: Mix 50 mg each of ethylene glycol, diethylene glycol and propylene glycol for gas chromatography with 100 mL of methanol. When the procedure is run with 1 mL of this mixture under the conditions listed in Table 1, ethylene glycol, propylene glycol and diethylene glycol are eluted in this order, and the resolution between the peaks of ethylene glycol and propylene glycol is not less than 5, and that between the peaks of propylene glycol and diethylene glycol is not less than 50.
Fig. 1 shows the chromatogram obtained using a 1 µL injection of this solution. As indicated in the chromatogram, the degree of resolution between the peaks of ethylene glycol and propylene glycol is greater than 5, and that of propylene glycol and diethylene glycol is greater than 50.

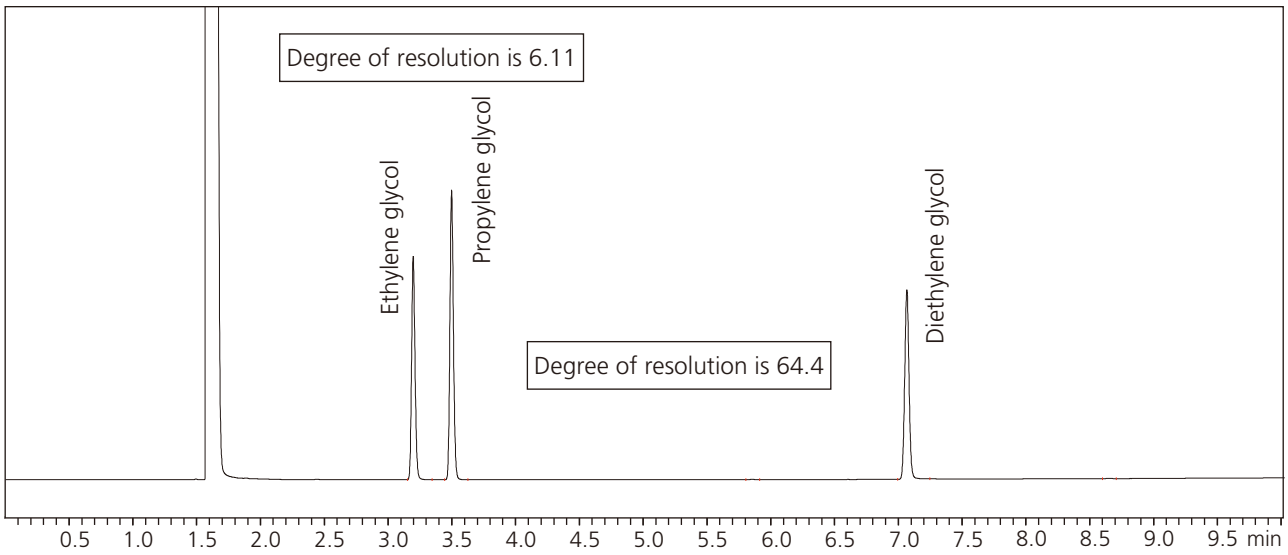

Fig. 1 Chromatogram Using System Suitability Test Solution

Table 1 Analytical Conditions

Model	: GC-2010 Plus AF/AOC-20i	Inj. Temp.	: 220 ˚C
Column	: SH-1701 (30 m × 0.32 mm I.D. df = 1.0 µm) *1	Det. Temp.	: 250 ˚C
Column Temp.	: 100 ˚C - 7.5 ˚C/min – 220 ˚C (4 min) Carrier	Split Ratio	: 1:20
Carrier Gas	: He, 38 cm/sec	Inj. Volume	: 1 µL

*1 P/N: 221-75782-30

Supplement Ⅱ stipulates the system repeatability requirements as follows:
System repeatability: Accurately weight about 0.1 g each of ethylene glycol and diethylene glycol, and mix with methanol to prepare a solution of exactly 100 mL. Pipet 5 mL of this solution, and transfer to a 100 mL volumetric flask. Separately, weigh 5.0 g of propylene glycol for gas chromatography, mix with a suitable amount of methanol and transfer this to the 100 mL volumetric flask. Bring this solution to volume with methanol, and use this as the standard solution. When six consecutive repeat analyses are performed using 1 mL of this standard solution, the relative standard deviation of the peak area of ethylene glycol and diethylene glycol is not to exceed 10 %.
The chromatogram obtained using a 1 µL injection of this standard solution is shown in Fig. 2. The peak area repeatability values for ethylene glycol and diethylene glycol obtained based on six consecutive repeat analyses are shown in Table 2. The relative standard deviations were each less than 10 %.

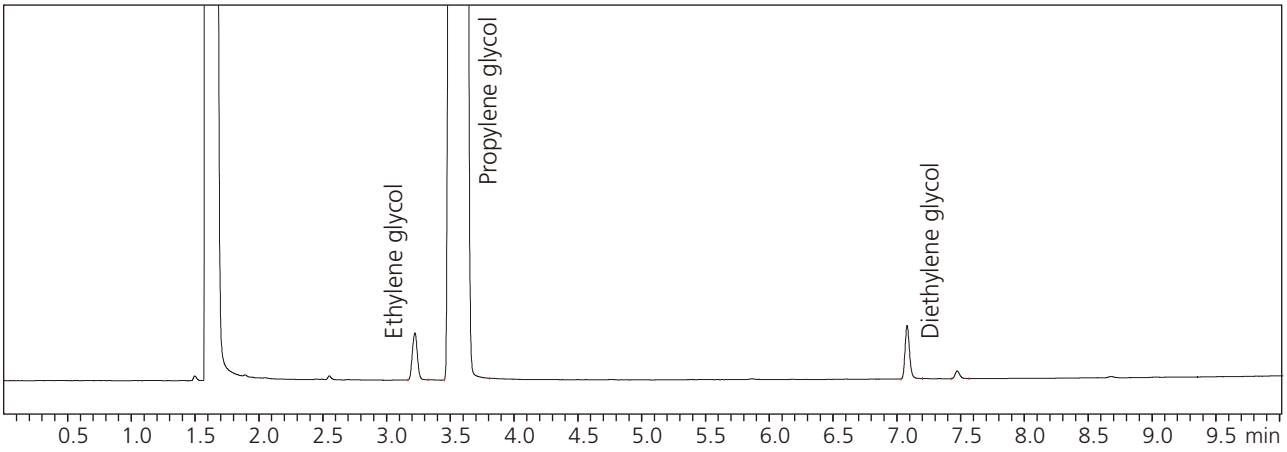

Fig. 2 Chromatogram of Standard Solution

Table 2 Repeatability of Peak Area of Ethylene Glycol and Diethylene Glycol (n=6)

	No.1	No.2	No.3	No.4	No.5	No.6	Mean Value	Standard Deviation	Relative Standard Deviation (%)
Ethylene Glycol	8449	8566	8553	8498	8590	8468	8521	57	0.67
Diethylene Glycol	8659	8760	8775	8597	8741	8711	8707	68	0.78

Note: The area values and relative standard deviations are reference values, not guaranteed values.

Analysis of a Sample Solution

A sample solution was prepared by mixing approximately 5 g of commercially available propylene glycol in 100 mL of methanol. The chromatogram obtained using a 1 µL injection is shown in Fig. 3.
Supplement Ⅱ also specifies as follows:
The amounts of ethylene glycol and diethylene glycol present in a sample solution are not to exceed 0.1 % respectively based on the standard solution area values. Following elution of the solvent, the area values are to be measured over a range about three times the retention time of propylene glycol. The peak areas of peaks other than propylene glycol, ethylene glycol and diethylene glycol in the sample solution must be no greater than 0.1 %, respectively, calculated by the area percentage method, and the total peak area of all peaks other than propylene glycol must be no greater than 1.0 %.
For more information, including the quantitative calculation method, please refer to the MHLW Notification No. 47 (February 28, 2014).

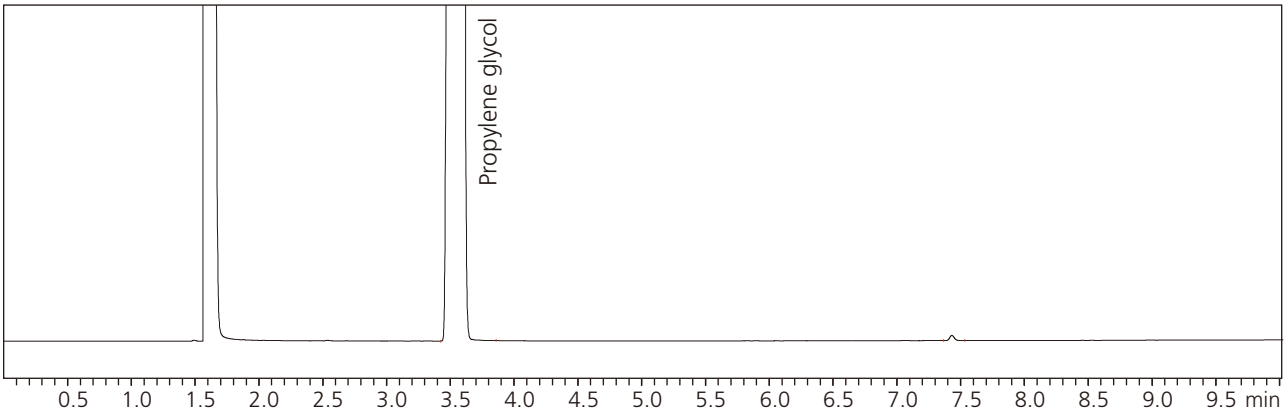

Fig. 3 Chromatogram of Sample Solution (Propylene Glycol 50 g/L)

Reference : The MHLW Notification No. 47 (February 28, 2014)

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