Glycerin
グリセリン

Change the Purity (11) to Purity (12) and add the following next to Purity (10)

Purity (11) Diethylene glycol and related substances-Weigh accurately about 5.88 g of Glycerin, mix in methanol, add methanol to make exactly 100 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.1 g of Diethylene glycol, mix in methanol, and add methanol to make exactly 100 mL. Pipet 5 mL of this solution, add methanol to make exactly 100mL, and use this solution as the standard solution. Perform the test with 1 μL each of the sample solution and standard solution as directed under Gas Chromatography<2.02> according to the following conditions. Determine each peak area of the sample solution and standard solution by the automatic integration method, and determine the peak areas, A_T and A_S, of diethylene glycol in each solution. Calculate the amount of diethylene glycol by the following equation: not more than 0.1 %. Calculate the amount of the related substances in the sample solution by the area percentage method: each amount of the related substances other than glycerin and diethylene glycol is not more than 0.1 % and the total amount of peaks other than glycerin is not more than 1.0 %.

$$\text{Amount(\%) of diethylene glycol} = (W_S/W_T)\times(A_T/A_S)\times 5/0.85$$

W_S: Amount(g) of diethylene glycol

W_T: Amount(g) of glycerin

Operating conditions-

Detector: A hydrogen flame-ionization detector.

Column: A fused-silica column 0.32 mm in inside diameter and 30 m in length, coated the inner surface with 14 % cyanopropylphenyl / 86 % dimethylsilicon polymer for gas chromatography 1 μm in thickness.

Column temperature: Inject at a constant temperature of about 100 ºC, raise at the ratio of 7.5 ºC per minute to 220 ºC, and maintain at a constant temperature of about 220 ºC.

Injection port temperature: A constant temperature of about 220 ºC.

Detector temperature: A constant temperature of about 250 ºC.

Carrier gas: Helium

Flow rate: About 38 cm per second.

Split ratio: 1:20

Time span of measurement: About 3 times as long as the retention time of glycerin, beginning after the solvent peak.

System suitability-

System performance: Weigh 0.05 g of Diethylene glycol and Glycerin, and mix in 100 mL of methanol. When the procedures is run with 1μL of this solution under the above operating conditions, diethylene

glycol and glycerin are eluted in this order with the resolution between these peaks being not less than 7.0.

System repeatability: When the test is repeated 6 times with 1μL of the standard solution under the above operating conditions, the relative standard deviation of the peak area of diethylene glycol is not more than 15 %.

Concentrated Glycerin
濃グリセリン

Change the Purity (11) to Purity (12) and add the following next to Purity (10)

Purity (11) Diethylene glycol and related substances-Weigh accurately about 5 g of Concentrated Glycerin, mix in methanol, add methanol to make exactly 100 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.1 g of Diethylene glycol, mix in methanol, and add methanol to make exactly 100 mL. Pipet 5 mL of this solution, add methanol to make exactly 100mL, and use this solution as the standard solution. Perform the test with 1 μL each of the sample solution and standard solution as directed under Gas Chromatography<2.02> according to the following conditions. Determine each peak area of the sample solution and standard solution by the automatic integration method, and determine the peak areas, A_T and A_S, of diethylene glycol in each solution. Calculate the amount of diethylene glycol by the following equation: not more than 0.1 %. Calculate the amount of the related substances in the sample solution by the area percentage method: each amount of the related substances other than glycerin and diethylene glycol is not more than 0.1 % and the total amount of peaks other than glycerin is not more than 1.0 %.

$$\text{Amount(\%) of diethylene glycol} = (W_S/W_T)\times(A_T/A_S)\times 5$$

W_S: Amount(g) of diethylene glycol

W_T: Amount(g) of concentrated glycerin

Operating conditions-

Detector: A hydrogen flame-ionization detector.

Column: A fused-silica column 0.32 mm in inside diameter and 30 m in length, coated the inner surface with 14 % cyanopropylphenyl / 86 % dimethylsilicon polymer for gas chromatography 1 μm in thickness.

Column temperature: Inject at a constant temperature of about 100 ºC, raise at the ratio of 7.5 ºC per minute to 220 ºC, and maintain at a constant temperature of about 220 ºC.

Injection port temperature: A constant temperature of about 220 ºC.

Detector temperature: A constant temperature of about 250 ºC.

Carrier gas: Helium

Flow rate: About 38 cm per second.

Split ratio: 1:20

Time span of measurement: About 3 times as long as the retention time of glycerin, beginning after the solvent peak.

System suitability-

System performance: Weigh 0.05 g of Diethylene glycol and Glycerin, and mix in 100 mL of methanol. When the procedures is run with 1μL of this solution under the above operating conditions, diethylene

glycol and glycerin are eluted in this order with the resolution between these peaks being not less than 7.0.

System repeatability: When the test is repeated 6 times with 1μL of the standard solution under the above operating conditions, the relative standard deviation of the peak area of diethylene glycol is not more than 15 %.