

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 μ L 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 %.

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

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 μ m in thickness.

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

Injection port temperature: A constant temperature of about 220 °C.

Detector temperature: A constant temperature of about 250 °C.

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 1 μ L 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 1 μ L 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 μ L 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 %.

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

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 μ m in thickness.

Column temperature: Inject at a constant temperature of about 100 $^{\circ}$ C, raise at the ratio of 7.5 $^{\circ}$ C per minute to 220 $^{\circ}$ C, and maintain at a constant temperature of about 220 $^{\circ}$ C.

Injection port temperature: A constant temperature of about 220 $^{\circ}$ C.

Detector temperature: A constant temperature of about 250 $^{\circ}$ C.

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 1 μ L 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 1 μ L 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 %.