E59 - PROPYLENE GLYCOL

Propylenglycolum

$C_3H_8O_2$ M_r 76.1

DEFINITION

(*RS*)-Propane-1,2-diol.

Content : minimum 99.7 per cent.

CHARACTERS

Appearance: viscous, clear, colourless, hygroscopic liquid.

Solubility: miscible with water and with ethanol (96 per cent).

IDENTIFICATION

Infrared absorption spectrophotometry.

Preparation: as a film between sodium chloride plates.

Comparison: *propylene glycol reference standard* (USP/EP).
propylene glycol reference spectrum (JP).

TESTS

Appearance.

It is clear (its clarity is the same as that of water or its opalescence is not more pronounced than that of reference suspension I) and colourless as water or not more intensely coloured than the reference solution.

Primary solutions:
- *Ferric chloride primary solution:* a 45.0 g/L solution of ferric chloride ($FeCl_3$, $6H_2O$).
- *Cobalt chloride primary solution:* a 59.5 g/L solution of cobalt chloride ($CoCl_2$, $6H_2O$).
- *Copper sulphate primary solution:* a 62.4 g/L solution of copper sulpfate ($CuSO_4$, H_2O).

Reference solution:
Immediately before use, mix 3.0 mL of ferric chloride primary solution, 3.0 mL of cobalt chloride primary solution, and 2.4 mL of cupric sulfate primary solution with 1.6 mL of hydrochloric acid (10 g/L HCl). Dilute 1.0 mL of this solution to 100.0 mL with hydrochloric acid (10 g/L HCl).

Acidity. maximum 100 mg/l, calculated as CH_3COOH.

To 50 ml of *water*, add 1 ml of *phenolphthalein solution*, then add *0.01 M sodium hydroxide* until the solution remains pink for 30 s. Add 50.0 ml of the substance to be examined and titrate with *0.01 M*

sodium hydroxide until the colour turns back to pink and remains for more than 30 s.

1 ml of *0.01 M sodium hydroxide* is equivalent to 0.6 mg of CH_3COOH.

Oxidising substances. To 10 ml add 5 ml of *water*, 2 ml of *potassium iodide solution* and 2 ml of *dilute sulphuric acid* and allow to stand in a ground-glass-stoppered flask protected from light for 15 min. Titrate with *0.05 M sodium thiosulphate*, using 1 ml of *starch solution* as indicator. Not more than 0.2 ml of *0.05 M sodium thiosulphate* is required (170 ppm expressed as H_2O_2).

Aldehydes. maximum 20 ppm, expressed as HCHO.

Stock solution. To 0.300 g of *formaldehyde methanol solution* add *deionised distilled water* and dilute to 100.0 ml with the same solvent. Dilute 10.0 ml of this solution to 1000.0 ml with *deionised distilled water*. Dilute 20.0 ml of this solution to 500.0 ml with *deionised distilled water*.

Test solution. Introduce 1.00 g of the substance to be examined into a volumetric flask and add about 5 ml of *deionised distilled water*. Then proceed as described below.

Reference solutions. Introduce into volumetric flasks, respectively, 1.0 ml, 3.0 ml, 5.0 ml, 10.0 ml, 15.0 ml and 25.0 ml of stock solution. Then proceed as described below.

To each flask, add 2 ml of a freshly prepared 5 g/l solution of *methylbenzothiazolone hydrazone hydrochloride* adjusted to pH 4.0 using *sodium hydroxide 0.02 M*. Allow the solutions to stand for 30 min. Add 5 ml of a freshly prepared 7 g/l solution of *ferric chloride*. Cap and swirl the flasks. Allow to stand for 5 min. Add *methanol* to each flask and dilute to 50.0 ml with the same solvent. Mix thoroughly then allow to stand for 1 min.

Blank solution. Prepare in the same manner as for the reference solutions but omitting the stock solution.

Measure the absorbance of the solutions at 655 nm. Calculate the content of aldehydes expressed as HCHO in the substance to be examined from the calibration curve obtained using the reference solutions.

Ethylene glycol and Diethylene glycol. Gas chromatography.

Test solution. Dilute 5.0 g of the substance to be examined to 75 mL with methanol. In a second flask dilute 200.0 mg of 2,2,2-trichloroethanol to 100.0 mL with methanol. Transfer 5.0 mL of the solution to the propylene glycol solution and dilute to 100.0 mL with methanol.

Reference solution. Dilute 200.0 mg of propylene glycol to 75 mL with methanol. In a second flask dissolve 200.0 mg of 2,2,2-trichloroethanol, 100.0 mg of ethylene glycol and 100.0 mg of diethylene glycol 100.0 mL with methanol. Transfer 5.0 mL of the solution to the propylene glycol solution and dilute to 100.0 mL with methanol.

Column:

- material: fused silica
- *size*: *l* = 30 m, Ø = 0.53 mm;
- *stationary phase*: p*oly(cyanopropylphenyl)(6)(dimethyl)siloxane (94) R* (film thickness: 3 µm)

Carrier gas: *helium for chromatography*.

Flow rate: 4.5 ml/min.

Split ratio: 1:10.

Temperature:

	Time (min)	Temperature (°C)
Column	0 - 4 4 - 4.4 4.4 - 14.4 14.4 - 16.4 16.4 - 22.4	100 100 - 120 120 120 - 220 220
Injection port		220
Detector		250

Detection: flame ionisation.

Injection: 1 µl.

Injection liner: packed with deactivated glass wool.

Relative retention with reference to propylene glycol (retention time = about 4 min):

Ethylene glycol = about 0.8, 2,2,2-trichloroethanol= about 1.7, diethylene glycol = about 2.4.

System suitability: reference solution:

resolution: minimum 5.0 between the peaks due to ethylene glycol and propylene glycol.

Limits:

Ethylene glycol:

Calculate the ratio (R1) of the area of the peak due to ethylene glycol to the area of the peak due to 2,2,2-trichloroethanol from the chromatogram obtained with the reference solution; from the chromatogram obtained with the test solution, calculate the ratio of the area of the peak due to ethylene glycol to the area of the peak due to 2,2,2-trichloroethanol: this ratio is not greater than R1 (0.10 per cent)

Diethylene glycol:

Calculate the ratio (R2) of the area of the peak due to diethylene glycol to the area of the peak due to 2,2,2-trichloroethanol from the chromatogram obtained with the reference solution; from the chromatogram obtained with the test solution, calculate the ratio of the area of the peak due to diethylene glycol to the area of the peak due to 2,2,2-trichloroethanol: this ratio is not greater than R2 (0.10 per cent).

Water. maximum 0.2 per cent, determined on 5.00 g by Karl Fischer.

ASSAY

Gas chromatography: use the normalisation procedure.

Test solution. The substance to be examined.

Reference solution. To 1 g of substance to be examined and 1 g of *diethylene glycol* add *anhydrous ethanol* and dilute to 20 ml with the same solvent. Dilute 1 ml of this solution to 50 ml with *anhydrous ethanol*.

Column:

— *size*: *l* = 30 m, Ø = 0.32 mm;

— *stationary phase*: *poly(dimethyl)(diphenyl)siloxane* (film thickness 0.5 µm).

Carrier gas: *helium for chromatography*.

Flow rate: 1.4 ml/min.

Split ratio: 1:70.

Temperature:

— *column*: 150 °C;

— *injection port and detector*: 250 °C.

Detection: flame ionisation.

Injection: 1 µl.

Run time: 2.5 times the retention time of propylene glycol.

Relative retention with reference to propylene glycol (retention time = about 2 min): diethylene glycol = about 1.2.

System suitability: *reference solution:*

- *resolution*: minimum 5.0 between the peaks due to propylene glycol and diethylene glycol.

STORAGE

In an airtight container.

Reagents

Hydrazine sulphate solution. Dissolve 1.0 g of hydrazine sulphate in water and dilute to 100.0 ml with the same solvent. Allow to stand for 4-6 h.

Hexamethylenetetramine solution. In a 100 ml ground-glass-stoppered flask, dissolve 2.5 g of hexamethylenetetramine in 25.0 ml of water.

Primary opalescent suspension (formazin suspension). To the hexamethylenetetramine solution in the flask add 25.0 ml of the hydrazine sulphate solution. Mix and allow to stand for 24 h. This suspension is

stable for 2 months, provided it is stored in a glass container free from surface defects. The suspension must not adhere to the glass and must be well mixed before use.

Standard of opalescence. Dilute 15.0 ml of the primary opalescent suspension to 1000.0 ml with water. This suspension is freshly prepared and may be stored for up to 24 h.

Reference suspension I. To 5.0 ml of standard of opalescence add 95.0 ml of water. Mix and shake before use.

Formaldehyde methanol solution.
A solution containing respectively 37 per cent *m/m* of formaldehyde and 10-15 per cent *m/m* of methanol in water [1].
Assay. To 2.0 g of *formaldehyde methanol solution*, add 100 ml of a freshly prepared 100 g/l solution of *sodium sulphite* in *deionised distilled water*. Add 0.1 ml of *phenolphthalein solution*. Titrate with *0.25 M sulphuric acid* until the colour changes from pink to colourless.
Carry out a blank titration.
Calculate the percentage content of formaldehyde in *formaldehyde methanol solution* using the following expression:

$$\frac{(V - B) \times 2M \times 3.0}{m}$$

V = volume of *0.25 M sulphuric acid* used in the assay, in millilitres,
B = volume of *0.25 M sulphuric acid* used in the blank, in millilitres,
M = molarity of the titrant,
m = mass of sample, in grams.

Diethylene glycol. $C_4H_{10}O_3$. (M_r 106.1). [111-46-6]. 2,2′-Oxydiethanol.
Content: minimum 99.5 per cent *m/m*.
Clear, colourless liquid, hygroscopic, miscible with water, with acetone and with ethanol (96 per cent).
$d_{20,20}$: about 1.118.
$n_{20,D}$: about 1.447.
bp: 244 °C to 246 °C.
Storage: in an airtight container.

Poly(dimethyl)(diphenyl)siloxane.
Stationary phase for gas chromatography.
Contains 95 per cent of methyl groups and 5 per cent of phenyl groups.

Poly(cyanopropylphenyl)(6)(dimethyl)siloxane (94)
Stationary phase for chromatography.
Contains 6 per cent of cyanopropylphenyl groups and 94 per cent of methyl groups.

Phenolphthalein solution
A 10 g/L solution in ethanol (96 per cent)

Potassium iodide solution
A 166 g/L solution.

[1] available from Fischer Scientific