Edition: BP 2025 (Ph. Eur. 11.6 update)

Macrogol Isotridecyl Ether

General Notices

(Ph. Eur. monograph 2730)

Ph Eur

DEFINITION

Mixture of ethers of mixed macrogols with linear and branched fatty alcohols, mainly $C_{13}H_{28}O$. It contains a variable quantity of free $C_{13}H_{28}O$ and it may contain free macrogols. The number of moles of ethylene oxide reacted per mole of branched $C_{13}H_{28}O$ is 3 or 4 (nominal value).

CHARACTERS

Appearance

Colourless liquid.

Solubility

Practically insoluble in water, soluble or dispersible in ethanol (96 per cent), practically insoluble in light petroleum.

IDENTIFICATION

A. Gas chromatography (*2.2.28*).

Test solution Dissolve 0.200 g of the substance to be examined in *acetone R* and dilute to 10.0 mL with the same solvent.

Reference solution Dissolve 0.35 g of *lauryl alcohol R*, 0.35 g of *tridecyl alcohol R*, 0.15 g of *myristyl alcohol R* and 0.15 g of *ethylene glycol monododecyl ether R* in *acetone R* and dilute to 100.0 mL with the same solvent. Dilute 1.0 mL of the solution to 10.0 mL with *acetone R*.

Column:

— *material*: fused silica;

— *size*: *l* = 30 m, Ø = 0.25 mm;

— *stationary phase*: methylpolysiloxane R (film thickness 0.25 µm).

Carrier gas: hydrogen for chromatography R or helium for chromatography R.
Flow rate: 1 mL/min.

Split ratio 1:50.

Temperature:

	Time (min)	Temperature (°C)
Column	0 - 1	120
	1 - 24	120 → 350
	24 - 34	350
Injection port		300
Detector		350

Detection Flame ionisation.

Injection 1 µL.

Retention time Lauryl alcohol = about 6.7 min; tridecyl alcohol = about 7.8 min; myristyl alcohol = about 9 min; ethylene glycol monododecyl ether = about 9.3 min.

System suitability Reference solution:

— resolution: minimum 5 between the peaks due to myristyl alcohol and ethylene glycol monododecyl ether.

Results The sum of the areas of the peaks eluting between the peak due to lauryl alcohol and the peak due to tridecyl alcohol is greater than the area of the peak due to tridecyl alcohol and the area of the peak due to lauryl alcohol.

B. Hydroxyl value (see Tests).
C. Iodine value (see Tests).
D. Saponification value (see Tests).
E. Dissolve or disperse 0.1 g in 5 mL of ethanol (96 per cent) R. Add 10 mL of dilute hydrochloric acid R, 10 mL of barium chloride solution R1 and 10 mL of a 100 g/L solution of phosphomolybdic acid R. A precipitate is formed.

TESTS

Appearance of solution

The solution is not more intensely coloured than reference solution BY_5 (2.2.2, Method II).

Dissolve 5.0 g in ethanol (96 per cent) R and dilute to 50 mL with the same solvent.

Alkalinity

Dissolve 2.0 g in a hot mixture of 10 mL of ethanol (96 per cent) R and 10 mL of water R. Add 0.1 mL of bromothymol blue solution R1. Not more than 0.5 mL of 0.1 M hydrochloric acid is required to change the

colour of the indicator to yellow.

Acid value (*2.5.1*)

Maximum 1.0, determined on 5.0 g.

Hydroxyl value (*2.5.3, Method A*)

See Table 2730.-1.

Table 2730.-1

Ethylene oxide units per molecule (nominal value)	Hydroxyl value
3	165 - 185
4	145 - 160

Iodine value (*2.5.4*)

Maximum 2.0.

Saponification value (*2.5.6*)

Maximum 3.0, determined on 10.0 g.

Ethylene oxide and dioxan (*2.4.25*)

Maximum 1 ppm of ethylene oxide and 10 ppm of dioxan.

Water (*2.5.12*)

Maximum 3.0 per cent, determined on 2.00 g.

Sulfated ash (*2.4.14*)

Maximum 0.2 per cent.

Ignite a silica crucible at 600 ± 50 °C for 30 min, allow to cool in a desiccator over a suitable desiccant and weigh. Evenly distribute 1.0 g in the crucible and weigh. Dry at 100-105 °C for 1 h and ignite in a muffle furnace at 600 ± 25 °C until the substance is thoroughly charred. Carry out the test for sulfated ash (*2.4.14*) on the residue obtained, starting from “Moisten the substance to be examined...”.

STORAGE

In an airtight container.

LABELLING

The label states the number of moles of ethylene oxide reacted per mole of $C_{13}H_{28}O$ (nominal value).

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