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

Macrogol Lauryl Ether

General Notices

(Ph. Eur. monograph 1124)

Action and use

Non-ionic surfactant.

Ph Eur

DEFINITION

Mixture of ethers of mixed macrogols with linear fatty alcohols, mainly $C_{12}H_{26}O$. It contains a variable quantity of free $C_{12}H_{26}O$ and may contain free macrogols. The number of moles of ethylene oxide reacted per mole of $C_{12}H_{26}O$ is 3 to 23 (nominal value).

CHARACTERS

— Macrogol lauryl ethers with 3 to 5 units of ethylene oxide per molecule.

Appearance

Colourless liquid.

Solubility

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

— Macrogol lauryl ethers with 9 units of ethylene oxide per molecule.

Appearance

White or almost white, unctuous, hygroscopic mass, melting at 24 °C into a colourless or yellowish, viscous liquid.

Solubility

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

— Macrogol lauryl ethers with 10 to 23 units of ethylene oxide per molecule.

Appearance

White or almost white, waxy mass.

Solubility

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

IDENTIFICATION

Carry out tests A, B, C, D, E for macrogol lauryl ethers with 3 to 5 units of ethylene oxide per molecule.

Carry out tests A, B, C, D, F for macrogol lauryl ethers with 9 to 23 units of ethylene oxide per molecule.

A. Hydroxyl value (see Tests).
B. Iodine value (see Tests).
C. Saponification value (see Tests).
D. 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.
E. 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 0.15 g of *ethylene glycol monododecyl ether R*, 0.15 g of *lauryl alcohol R* and 0.15 g of *myristyl alcohol R* in *acetone R* and dilute to 100.0 mL with the same solvent.

Column:

— *material*: fused silica;

— *size*: *l* = 30 m, Ø = 0.25 mm;

— *stationary phase*: *phenyl(5)methyl(95)polysiloxane R* (film thickness 0.25 µm).

Carrier gas *helium for chromatography R* or *hydrogen 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 1.0 µL.

Identification of peaks Use the chromatogram obtained with the reference solution to identify the peaks due to ethylene glycol monododecyl ether, lauryl alcohol and myristyl alcohol.

Retention time Lauryl alcohol = about 6.7 min; myristyl alcohol = about 8.9 min; ethylene glycol monododecyl ether = about 9.3 min.

System suitability Reference solution:

— *resolution*: minimum 5.0 between the peaks due to myristyl alcohol and ethylene glycol monododecyl ether.

Results The sum of the areas of the peaks eluted before the peak due to lauryl alcohol is less than 10 per cent of the area of the peak due to lauryl alcohol; disregard the peak due to the solvent.

F. Gas chromatography (*2.2.28*).

Test solution Into a 50 mL round-bottomed flask fitted with a reflux condenser introduce 0.300 g of the substance to be examined, 15 mL of *hydriodic acid R*, 2.5 mL of a 603 g/L solution of *phosphorous acid R* and 2 or 3 anti-bumping granules. Heat to 140 °C using an oil bath or an electric heating mantle, then boil for 2 h. Allow to cool to room temperature and rinse the condenser with 5 mL of *ethanol (96 per cent) R*. Add the rinsings to the flask and transfer to a separating funnel, rinsing the flask with 2 quantities, each of 5 mL, of a mixture of equal volumes of *ethanol (96 per cent) R* and *water R*.

Extract with 15 mL of *heptane R* and wash the upper layer with 5 mL portions of a mixture of equal volumes of *ethanol (96 per cent) R* and *water R* until the pH of the lower layer is greater than 3 using a *pH indicator strip R*.

Transfer the upper layer into a 20 mL glass vial with a screw cap and shake with 5 g of *anhydrous sodium sulfate R*. Allow to settle and transfer about 1 mL of the clear supernatant to an autosampler vial.

Reference solution Prepare as described for the test solution using 0.150 g of *2-butyloctanol R*, 0.150 g of *lauryl alcohol R* and 0.150 g of *myristyl alcohol R* instead of the substance to be examined.

Column:

— *material*: fused silica;

— *size*: *l* = 30 m, Ø = 0.32 mm;

— *stationary phase*: *phenyl(5)methyl(95)polysiloxane R* (film thickness 1.0 µm).

Carrier gas *helium for chromatography R* or *hydrogen for chromatography R*.

Flow rate 3 mL/min.

Split ratio 1:30.

Temperature:

	Time (min)	Temperature (°C)
Column	0 - 5	100
	5 - 55	100 → 300
	55 - 70	300
Injection port		250
Detector		310

Detection Flame ionisation.

Injection 1.0 µL.

Identification of peaks Use the chromatogram obtained with the reference solution to identify the peaks due to 2-butyloctyl iodide, lauryl iodide and myristyl iodide.

Retention time 2-butyloctyl iodide = about 22.7 min; lauryl iodide = about 25.6 min; myristyl iodide = about 31.1 min.

System suitability Reference solution:

— *resolution*: minimum 5.0 between the peaks due to 2-butyloctyl iodide and lauryl iodide.

Results The sum of the areas of the peaks eluted before the peak due to lauryl iodide is less than 10 per cent of the area of the peak due to lauryl iodide; disregard the peak due to the solvent.

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*)

Ethylene oxide units per molecule (nominal value)	Hydroxyl value
3	165 - 180
4	145 - 165
5	130 - 140
9	90 - 100
10	85 - 95
12	73 - 83
15	64 - 74
20 - 23	40 - 60

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 maximum 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 and weigh. Evenly distribute 1.0 g in the crucible and weigh again. 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 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 $C_{12}H_{26}O$ (nominal value).

Ph Eur