Composition of fatty acids (*2.4.22, Method C*). The fatty-acid fraction of the substance has the following composition:

- *palmitic acid*: not more than 12.0 per cent,
- *stearic acid*: not more than 6.0 per cent,
- *oleic acid*: not less than 60.0 per cent,
- *linoleic acid*: not more than 35.0 per cent,
- *linolenic acid*: not more than 2.0 per cent,
- *arachidic acid*: not more than 2.0 per cent,
- *eicosenoic acid*: not more than 2.0 per cent.

Water (*2.5.12*). Not more than 1.0 per cent, determined on 1.00 g by the semi-micro determination of water. Use as the solvent a mixture of equal volumes of *anhydrous methanol R* and *methylene chloride R*.

Total ash (*2.4.16*). Not more than 0.1 per cent, determined on 1.00 g.

ASSAY

Determine the free glycerol content and the mono-, di- and triacylglycerol contents by size-exclusion chromatography (*2.2.30*).

Test solution. Into a 15 ml flask, weigh about 0.2 g (*m*), to the nearest 0.1 mg. Add 5 ml of *tetrahydrofuran R* and shake to dissolve. Reweigh the flask and calculate the total mass of solvent and substance (*M*).

Reference solutions. Into four 15 ml flasks, respectively weigh, to the nearest 0.1 mg, about 2.5 mg, 5 mg, 10 mg and 20 mg of *glycerol R*. Add 5 ml of *tetrahydrofuran R* and shake until well mixed. Weigh the flasks again and calculate the concentration of glycerol in milligrams per gram for each reference solution.

The chromatographic procedure may be carried out using:

- a gel-permeation column 0.6 m long and 7 mm in internal diameter packed with *styrene-divinylbenzene copolymer R* (particle diameter 5 µm and porosity 10 nm),
- as mobile phase at a flow rate of 1 ml/min *tetrahydrofuran R*,
- a differential refractive index detector.

Inject 40 µl of each solution. When the chromatograms are recorded in the prescribed conditions, the retention times relative to glycerol are about 0.85 for the monoacylglycerols, about 0.79 for the diacylglycerols and about 0.76 for the triacylglycerols. From the calibration curve obtained with the reference solutions determine the concentration (*C*) in milligrams per gram of glycerol in the test solution.

Calculate the percentage content of free glycerol in the substance to be examined using the following expression:

$$\frac{C \times M}{m \times 10}$$

Calculate the percentage content of mono-, di- and triacylglycerols in the substance to be examined by the normalisation procedure.

STORAGE

Store in an airtight container, protected from light.

LABELLING

The label states:

- the nominal content of monoacylglycerol,
- the name and concentration of any added antioxidant.

01/2005:0495

GLYCEROL MONOSTEARATE 40-55

Glyceroli monostearas 40-55

DEFINITION

Glycerol monostearate 40-55 is a mixture of monoacylglycerols, mainly monostearoylglycerol, together with variable quantities of di- and triacylglycerols. It contains 40.0 per cent to 55.0 per cent of monoacylglycerols, 30.0 per cent to 45.0 per cent of diacylglycerols and 5.0 per cent to 15.0 per cent of triacylglycerols, obtained by partial glycerolysis of vegetable oils mainly containing triacylglycerols of palmitic or stearic acid or by esterification of glycerol with stearic acid 50 (type I), stearic acid 70 (type II) or stearic acid 95 (type III) (see *Stearic acid (1474)*). The fatty acids may be of vegetable or animal origin.

CHARACTERS

A hard, waxy mass or unctuous powder or flakes, white or almost white, practically insoluble in water, soluble in alcohol at 60 °C.

IDENTIFICATION

A. Melting point (*2.2.15*): 54 °C to 64 °C. Introduce the melted substance into the capillary tubes and allow to stand for 24 h in a well-closed container.

B. Examine by thin-layer chromatography (*2.2.27*), using a *TLC silica gel plate R*.

Test solution. Dissolve 1.0 g of the substance to be examined in *methylene chloride R*, with gentle heating, and dilute to 20 ml with the same solvent.

Reference solution. Dissolve 1.0 g of *glycerol monostearate 40-55 CRS* in *methylene chloride R*, with gentle heating, and dilute to 20 ml with the same solvent.

Apply to the plate 10 µl of each solution. Develop over a path of 15 cm using a mixture of 30 volumes of *hexane R* and 70 volumes of *ether R*. Allow the plate to dry in air. Spray with a 0.1 g/l solution of *rhodamine B R* in *alcohol R* and examine in ultraviolet light at 365 nm. The spots in the chromatogram obtained with the test solution are similar in position to those in the chromatogram obtained with the reference solution.

C. It complies with the test for composition of fatty acids according to the type stated on the label (see Tests).

D. It complies with the limits of the assay (monoacylglycerol content).

TESTS

Acid value (*2.5.1*). Not more than 3.0, determined on 1.0 g, using a mixture of equal volumes of *alcohol R* and *toluene R* as solvent and heating gently.

Iodine value (*2.5.4*). Not more than 3.0.

Saponification value (*2.5.6*): 158 to 177, determined on 2.0 g. Carry out the titration with heating.

Free glycerol. Not more than 6.0 per cent, determined as described under Assay.

Composition of fatty acids (*2.4.22, Method C*). The fatty acid fraction of the substance to be examined has the following composition:

	Fatty acid used for production by esterification	Composition of fatty acids
Glycerol monostearate 40-55 type I	Stearic acid 50	*Stearic acid*: 40.0 per cent to 60.0 per cent *Sum of the contents of palmitic and stearic acids*: not less than 90.0 per cent
Glycerol monostearate 40-55 type II	Stearic acid 70	*Stearic acid*: 60.0 per cent to 80.0 per cent *Sum of palmitic and stearic acids*: not less than 90.0 per cent
Glycerol monostearate 40-55 type III	Stearic acid 95	*Stearic acid*: 90.0 per cent to 99.0 per cent *Sum of the contents of palmitic and stearic acids*: not less than 96.0 per cent

Nickel (*2.4.27*). Not more than 1 ppm of Ni.

Water (*2.5.12*). Not more than 1.0 per cent, determined on 1.00 g by the semi-micro determination of water. Use *pyridine R* as the solvent and heat gently.

Total ash (*2.4.16*). Not more than 0.1 per cent, determined on 1.00 g.

ASSAY

Determine the free glycerol content and the mono-, di- and triacylglycerol contents by size-exclusion chromatography (*2.2.30*).

Test solution. Into a 15 ml flask, weigh about 0.2 g (*m*), to the nearest 0.1 mg. Add 5 ml of *tetrahydrofuran R* and shake to dissolve. Reweigh the flask and calculate the total mass of solvent and substance (*M*).

Reference solutions. Into four 15 ml flasks, respectively weigh, to the nearest 0.1 mg, about 2.5 mg, 5 mg, 10 mg and 20 mg of *glycerol R*. Add 5 ml of *tetrahydrofuran R* and shake until well mixed. Weigh the flasks again and calculate the concentration of glycerol in milligrams per gram for each reference solution.

The chromatographic procedure may be carried out using:

– a gel-permeation column 0.6 m long and 7 mm in internal diameter packed with *styrene-divinylbenzene copolymer R* (particle diameter 5 µm and porosity 10 nm),
– as mobile phase at a flow rate of 1 ml/min *tetrahydrofuran R*,
– a differential refractive index detector.

Inject 40 µl of each solution. When the chromatograms are recorded in the prescribed conditions, the retention times relative to glycerol are about 0.86 for the monoacylglycerols, about 0.81 for the diacylglycerols and about 0.77 for the triacylglycerols. From the calibration curve obtained with the reference solutions determine the concentration (*C*) in milligrams per gram of glycerol in the test solution.

Calculate the percentage content of free glycerol in the substance to be examined using the following expression:

$$\frac{C \times M}{m \times 10}$$

Calculate the percentage content of mono-, di- and triacylglycerols in the substance to be examined by the normalisation procedure.

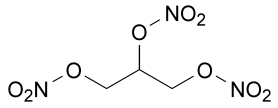
LABELLING

The label states the type of glycerol monostearate 40-55.

01/2005:1331

GLYCERYL TRINITRATE SOLUTION

Glyceroli trinitratis solutio



$C_3H_5N_3O_9$ M_r 227.1

DEFINITION

Ethanolic solution of glyceryl trinitrate.

Content: 1 per cent *m/m* to 10 per cent *m/m* of propane-1,2,3-triyl trinitrate and 96.5 per cent to 102.5 per cent of the declared content of glyceryl trinitrate stated on the label.

CHARACTERS

Appearance: clear, colourless or slightly yellow solution.

Solubility: miscible with acetone and with ethanol.

Solubility of pure glyceryl trinitrate: practically insoluble in water, freely soluble in ethanol, miscible with acetone.

IDENTIFICATION

First identification: A, C.

Second identification: B, C.

Upon diluting glyceryl trinitrate solution, care must be taken to always use anhydrous ethanol, otherwise droplets of pure glyceryl trinitrate may precipitate from the solution.

After examination, the residues and the solutions obtained in both the identification and the test sections must be heated on a water-bath for 5 min with dilute sodium hydroxide solution R.

A. Infrared absorption spectrophotometry (*2.2.24*).

Preparation: place 50 µl of a solution diluted, if necessary, with *ethanol R*, to contain 10 g/l of glyceryl trinitrate, on a disc of *potassium bromide R* and evaporate the solvent *in vacuo*.

Comparison: *Ph. Eur. reference spectrum of glyceryl trinitrate.*

B. Thin-layer chromatography (*2.2.27*).

Test solution. Dilute a quantity of the substance to be examined corresponding to 50 mg of glyceryl trinitrate to 100 ml with *acetone R*.

Reference solution. Dilute 0.05 ml of *glyceryl trinitrate solution CRS* to 1 ml with *acetone R*.

Plate: *TLC silica gel G plate R.*

Mobile phase: *ethyl acetate R, toluene R* (20:80 *V/V*).

Application: 5 µl.

Development: over 2/3 of the plate.

Drying: in air.

Detection: spray with freshly prepared *potassium iodide and starch solution R*. Expose the plate to ultraviolet light at 254 nm for 15 min. Examine in daylight.

Results: the principal spot in the chromatogram obtained with the test solution is similar in position, colour and size to the principal spot in the chromatogram obtained with the reference solution.

C. It complies with the limits of the assay.