Injection: 40 µl.

Relative retention with reference to glycerol (retention time = about 15.6 min): triacylglycerols = about 0.76; diacylglycerols = about 0.79; monoacylglycerols = about 0.85.

Calculations:

– *free glycerol*: from the calibration curve obtained with the reference solutions determine the concentration (*C*) of glycerol in milligrams per gram in the test solution and calculate the percentage content of free glycerol in the substance to be examined using the following expression:

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

– *mono-, di- and triacylglycerols*: calculate the percentage content of mono-, di- and triacylglycerols using the normalisation procedure.

STORAGE

In an airtight container, protected from light.

LABELLING

The label states the nominal content of monoacylglycerol.

01/2008:0495

GLYCEROL MONOSTEARATE 40-55

Glyceroli monostearas 40-55

DEFINITION

Mixture of monoacylglycerols, mainly monostearoylglycerol, together with variable quantities of di- and triacylglycerols. It is obtained by partial glycerolysis of vegetable oils mainly containing triacylglycerols of palmitic (hexadecanoic) or stearic (octadecanoic) acid or by esterification of glycerol with stearic acid. The fatty acids may be of vegetable or animal origin.

Content:

– *monoacylglycerols*: 40.0 per cent to 55.0 per cent;
– *diacylglycerols*: 30.0 per cent to 45.0 per cent;
– *triacylglycerols*: 5.0 per cent to 15.0 per cent.

CHARACTERS

Appearance: hard, waxy mass or unctuous powder or flakes, white or almost white.

Solubility: practically insoluble in water, soluble in ethanol (96 per cent) at 60 °C.

IDENTIFICATION

First identification: C, D.

Second identification: A, B.

A. Melting point (*2.2.15*): 54 °C to 66 °C.

Introduce the melted substance into the capillary tubes and allow to stand for 24 h in a well-closed container.

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

Test solution. Dissolve 0.5 g of the substance to be examined in *methylene chloride R*, with gentle heating, and dilute to 10 ml with the same solvent.

Reference solution. Dissolve 0.5 g of *glycerol monostearate 40-55 CRS* in *methylene chloride R*, with gentle heating, and dilute to 10 ml with the same solvent.

Plate: *TLC silica gel plate R.*

Mobile phase: *hexane R, ether R* (30:70 *V/V*).

Application: 10 µl.

Development: over a path of 15 cm.

Detection: spray with a 0.1 g/l solution of *rhodamine B R* in *ethanol (96 per cent) R* and examine in ultraviolet light at 365 nm.

Suitability system: reference solution:

– the chromatogram shows 4 clearly separated spots.

Results: 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. Composition of fatty acids (see Tests) according to the type stated on the label.

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

TESTS

Acid value (*2.5.1*): maximum 3.0, determined on 1.0 g.

Use a mixture of equal volumes of *ethanol (96 per cent) R* and *toluene R* as solvent and heat gently.

Iodine value (*2.5.4, Method A*): maximum 3.0.

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

Free glycerol: maximum 6.0 per cent, determined as described under Assay.

Composition of fatty acids (*2.4.22, Method C*). Use the mixture of calibrating substances in Table 2.4.22.-1.

Composition of the fatty-acid fraction of the substance:

Glycerol monostearate 40-55	Composition of fatty acids
Type I	*Stearic acid*: 40.0 per cent to 60.0 per cent
	Sum of the contents of palmitic and stearic acids: minimum 90.0 per cent
Type II	*Stearic acid*: 60.0 per cent to 80.0 per cent
	Sum of the contents of palmitic and stearic acids: minimum 90.0 per cent
Type III	*Stearic acid*: 80.0 per cent to 99.0 per cent
	Sum of the contents of palmitic and stearic acids: minimum 96.0 per cent

Nickel (*2.4.31*): maximum 1 ppm.

Water (*2.5.12*): maximum 1.0 per cent, determined on 1.00 g. Use *pyridine R* as the solvent and heat gently.

Total ash (*2.4.16*): maximum 0.1 per cent.

ASSAY

Size-exclusion chromatography (*2.2.30*).

Test solution. Into a 15 ml flask, weigh 0.200 g (*m*). Add 5.0 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 2.5 mg, 5.0 mg, 10.0 mg and 20.0 mg of *glycerol R*, and add 5.0 ml of *tetrahydrofuran R* to each flask. Weigh the flasks again and calculate the concentration of glycerol in milligrams per gram for each reference solution.

Column:

– *size*: *l* = 0.6 m, Ø = 7 mm;
– *stationary phase*: *styrene-divinylbenzene copolymer R* (5 µm) with a pore size of 10 nm.

Mobile phase: *tetrahydrofuran R.*

Flow rate: 1 ml/min.

Detection: differential refractometer.

Injection: 40 µl.

Relative retention with reference to glycerol (retention time = about 15 min): triacylglycerols = about 0.75; diacylglycerols = about 0.80; monoacylglycerols = about 0.85.

Calculations:

– *free glycerol*: from the calibration curve obtained with the reference solutions, determine the concentration (*C*) in milligrams per gram in the test solution and calculate the percentage content in the substance to be examined using the following expression:

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

– *mono-, di- and triacylglycerols*: calculate the percentage contents by the normalisation procedure.

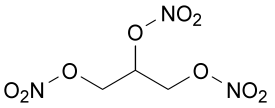
LABELLING

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

01/2008:1331

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.

TESTS

Upon diluting glyceryl trinitrate solution, care must be taken always to 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.

Appearance of solution. If necessary dilute the solution to be examined to a concentration of 10 g/l with *ethanol R*. The solution is not more intensely coloured than reference solution Y_7 (*2.2.2, Method II*).

Inorganic nitrates. Thin-layer chromatography (*2.2.27*).

Test solution. If necessary dilute the solution to be examined to a concentration of 10 g/l with *ethanol R*.

Reference solution. Dissolve 5 mg of *potassium nitrate R* in 1 ml of *water R* and dilute to 100 ml with *alcohol R*.

Plate: *TLC silica gel plate R.*

Mobile phase: *glacial acetic acid R, acetone R, toluene R* (15:30:60 *V/V/V*).

Application: 10 µl.

Development: over 2/3 of the plate.

Drying: in a current of air until the acetic acid is completely removed.

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

Limit:

– *nitrate ion*: any spot corresponding to the nitrate ion in the chromatogram obtained with the test solution is not more intense than the spot in the chromatogram obtained with the reference solution (0.5 per cent of the content of glyceryl trinitrate calculated as potassium nitrate).

Related substances. Liquid chromatography (*2.2.29*).

Test solution. Dissolve a quantity of the substance to be examined corresponding to 2 mg of glyceryl trinitrate in the mobile phase and dilute to 20.0 ml with the mobile phase.

Reference solution (a). Dissolve 0.10 g of *glyceryl trinitrate solution CRS* and a quantity of *diluted pentaerythrityl tetranitrate CRS* equivalent to 1.0 mg of pentaerythrityl tetranitrate in the mobile phase and dilute to 100.0 ml with the mobile phase. Sonicate and filter if necessary.