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:

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

– *monoacylglycerols*: calculate the percentage content of monoacylglycerols using the following expression:

$$A - D$$

A = percentage content of monoacylglycerols determined by the normalisation procedure,

D = percentage content of free fatty acids.

Calculate the percentage content of free fatty acids using the following expression:

$$\frac{I_A \times 340}{561.1}$$

I_A = acid value.

– *diacylglycerols, triacylglycerols*: determine the percentage content of each by the normalisation procedure.

01/2008:1428

GLYCEROL DISTEARATE

Glyceroli distearas

DEFINITION

Mixture of diacylglycerols, mainly distearoylglycerol, together with variable quantities of mono- and triacylglycerols. It is obtained by partial glycerolysis of vegetable oils 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*: 8.0 per cent to 22.0 per cent;

– *diacylglycerols*: 40.0 per cent to 60.0 per cent;

– *triacylglycerols*: 25.0 per cent to 35.0 per cent.

CHARACTERS

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

Solubility: practically insoluble in water, soluble in methylene chloride, partly soluble in hot ethanol (96 per cent).

IDENTIFICATION

First identification: C, D.

Second identification: A, B.

A. Melting point (*2.2.14*): 50 °C to 60 °C (types I and II), 50 °C to 70 °C (type III).

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 distearate 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.

System suitability: 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 (diacylglycerol content).

TESTS

Acid value (*2.5.1*): maximum 6.0, 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*): 165 to 195, determined on 2.0 g. Carry out the titration with heating.

Free glycerol: maximum 1.0 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 distearate	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.

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 three 15 ml flasks, respectively weigh 2.0 mg, 5.0 mg and 10.0 mg of *glycerol R* and add 5.0 ml of *tetrahydrofuran R* to each flask. Into a 4th flask, weigh about 2.0 mg of *glycerol R* and add 10.0 ml of *tetrahydrofuran R*. 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 distearate.

01/2008:2213

GLYCEROL MONOCAPRYLATE

Glyceroli monocaprylas

DEFINITION

Mixture of monoacylglycerols, mainly mono-*O*-octanoylglycerol, containing variable quantities of di- and triacylglycerols, obtained by direct esterification of glycerol with caprylic (octanoic) acid, followed by a distillation step in the case of glycerol monocaprylate (type II).

Content:

– *glycerol monocaprylate (type I)*:
  – *monoacylglycerols*: 45.0 per cent to 75.0 per cent;
  – *diacylglycerols*: 20.0 per cent to 50.0 per cent;
  – *triacylglycerols*: maximum 10.0 per cent;
– *glycerol monocaprylate (type II)*:
  – *monoacylglycerols*: minimum 80.0 per cent;
  – *diacylglycerols*: maximum 20.0 per cent;
  – *triacylglycerols*: maximum 5.0 per cent.

CHARACTERS

Appearance: colourless or slightly yellow, oily liquid or soft mass.

Solubility: practically insoluble in water, very soluble in ethanol (96 per cent) and freely soluble in methylene chloride.

IDENTIFICATION

A. Composition of fatty acids (see Tests).

B. It complies with the limits of the assay (monoacylglycerols).

TESTS

Acid value (*2.5.1*): maximum 3.0.

Composition of fatty acids (*2.4.22, Method C*). Use the mixture of calibrating substances in Table 2.4.22.-2.

Composition of the fatty acid fraction of the substance:

– *caproic acid*: maximum 1.0 per cent;
– *caprylic acid*: minimum 90.0 per cent;
– *capric acid*: maximum 10.0 per cent;
– *lauric acid*: maximum 1.0 per cent;
– *myristic acid*: maximum 0.5 per cent.

Free glycerol: maximum 3.0 per cent.

Dissolve 1.20 g in 25.0 ml of *methylene chloride R*. Heat to about 50 °C then allow to cool. Add 100 ml of *water R*. Shake and add 25.0 ml of *periodic acetic acid solution R*. Shake again and allow to stand for 30 min. Add 40 ml of a 75 g/l solution of *potassium iodide R* and allow to stand for 1 min. Add 1 ml of *starch solution R*. Titrate with *0.1 M sodium thiosulfate* until the aqueous phase becomes colourless. Carry out a blank titration.

1 ml of *0.1 M sodium thiosulfate* is equivalent to 2.3 mg of glycerol.

Water (*2.5.12*): maximum 1.0 per cent, determined on 1.00 g.

Total ash (*2.4.16*): maximum 0.5 per cent.

ASSAY

Gas chromatography (*2.2.28*): use the normalisation procedure.

Test solution. To 0.25 g of the substance to be examined, add 5.0 ml of *tetrahydrofuran R* and shake to dissolve.

Reference solution (a). To 0.25 g of *glycerol monocaprylate CRS*, add 5.0 ml of *tetrahydrofuran R* and shake to dissolve.

Reference solution (b). To 50 mg of *glycerol 1-octanoate R* and 50 mg of *glycerol 1-decanoate R*, add 2.5 ml of *tetrahydrofuran R* and shake to dissolve.

Column:

– *size*: *l* = 10 m, Ø = 0.32 mm;
– *stationary phase*: *poly(dimethyl)(diphenyl)siloxane R* (film thickness 0.1 µm).

Carrier gas: *helium for chromatography R*.

Flow rate: 2.3 ml/min.

Split ratio: 1:50.

Temperature:

	Time (min)	Temperature (°C)
Column	0 - 3	60
	3 - 38	60 → 340
	38 - 50	340
Injection port		350
Detector		370

Detection: flame ionisation.

Injection: 1 µl.

Identification of peaks: use the chromatogram supplied with *glycerol monocaprylate CRS* and the chromatogram obtained with reference solution (a) to identify the peaks due to mono-, di- and triacylglycerols.

System suitability: reference solution (b):

– *resolution*: minimum 5 between the peaks due to glycerol 1-octanoate and glycerol 1-decanoate.