– *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.

For the calculation of the contents of mono-, di- and triacylglycerols, disregard the peaks with a retention time less than that of the monoacylglycerols, which are due to impurities of the solvent and to the free fatty acids.

Calculate the percentage content of free fatty acids (*C*) using the following expression:

$$\frac{I_A \times 144}{561.1}$$

I_A = acid value of glycerol monocaprylate.

Calculate the content of mono-, di- and triacylglycerols using the following equations:

$$\text{Monoacylglycerols content} = \frac{X \times (100 - A - B - C)}{100}$$

$$\text{Diacylglycerols content} = \frac{Y \times (100 - A - B - C)}{100}$$

$$\text{Triacylglycerols content} = \frac{Z \times (100 - A - B - C)}{100}$$

A = percentage content of free glycerol (see Tests);

B = percentage content of water;

X = monoacylglycerols content obtained by normalisation;

Y = diacylglycerols content obtained by normalisation;

Z = triacylglycerols content obtained by normalisation.

LABELLING

The label states the type of glycerol monocaprylate (type I or II).

01/2008:2392

GLYCEROL MONOCAPRYLOCAPRATE

Glyceroli monocaprylocapras

DEFINITION

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

Content:

- *glycerol monocaprylocaprate (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 monocaprylocaprate (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 3.0 per cent;
- *caprylic acid*: 50.0 per cent to 90.0 per cent;
- *capric acid*: 10.0 per cent to 50.0 per cent;
- *lauric acid*: maximum 3.0 per cent;
- *myristic acid*: maximum 1.0 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 and 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 thiosulphate* until the aqueous phase becomes colourless. Carry out a blank titration.

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

Water (*2.5.12*): maximum 0.5 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 monocaprylocaprate 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.