Flow rate: 2.4 mL/min.

Temperature:

	Time (min)	Temperature (°C)
Column	0 - 2	70
	2 - 36	70 → 240
	36 - 41	240
Injection port		220
Detector		260

Detection: flame ionisation.

Injection: 1 µL.

Relative retention with reference to methyl stearate (retention time = about 40 min): methyl palmitate = about 0.88.

System suitability: reference solution:

– *resolution*: minimum 5.0 between the peaks due to methyl stearate and methyl palmitate.

Calculate the content of stearic acid and palmitic acid.

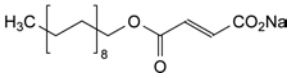
STORAGE

In an airtight container, protected from light.

07/2010:1567

SODIUM STEARYL FUMARATE

Natrii stearylis fumaras



$C_{22}H_{39}NaO_4$ M_r 390.5
[4070-80-8]

DEFINITION

Sodium octadecyl (*E*)-butenedioate.

Content: 99.0 per cent to 101.5 per cent (anhydrous substance).

CHARACTERS

Appearance: white or almost white, fine powder with agglomerates of flat, circular particles.

Solubility: practically insoluble in water, slightly soluble in methanol, practically insoluble in acetone and in anhydrous ethanol.

IDENTIFICATION

Infrared absorption spectrophotometry (*2.2.24*).

Comparison: *sodium stearyl fumarate CRS*.

TESTS

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

Silylation solution. To 2 mL of *N,O-bis(trimethylsilyl)trifluoroacetamide R* add 0.02 mL of *chlorotrimethylsilane R* and mix.

Test solution. Introduce 15.0 mg of the substance to be examined in a vial with a screw cap and add 1 mL of the silylation solution. Seal the vial and heat at about 70 °C for 1 h. After the reaction a precipitate remains in the vial; filter the solution through a nylon filter (pore size 0.45 µm).

Reference solution. Introduce 1.0 mg of *sodium stearyl maleate CRS* and 1.0 mg of *sodium stearyl fumarate CRS* into a vial with a screw cap and add 1 mL of the silylation solution. Seal the vial and heat at about 70 °C for 1 h.

Column:

– *material*: fused silica;

– *size*: *l* = 15 m, Ø = 0.53 mm;

– *stationary phase*: *poly(dimethyl)siloxane R* (film thickness 0.15 µm).

Carrier gas: *helium for chromatography R*.

Flow rate: 2 mL/min.

Split ratio: 1:25.

Temperature:

	Time (min)	Temperature (°C)
	0 - 1	180
Column	1 - 21	180 → 320
	21 - 26	320
Injection port		250
Detector		320

Detection: flame ionisation.

Injection: 2 µL.

Relative retention with reference to stearyl trimethylsilyl fumarate (retention time = about 9 min): stearyl alcohol = 0.30; stearyl trimethylsilyl ether = 0.35; palmityl trimethylsilyl fumarate = 0.80; heptadecyl trimethylsilyl fumarate = 0.85; stearyl trimethylsilyl maleate = 0.90; nonadecyl trimethylsilyl fumarate = 1.05; eicos-11-enyl trimethylsilyl fumarate = 1.15; distearyl fumarate = 2.25.

System suitability:

– *resolution*: minimum 1.5 between the peaks in the chromatogram obtained with the reference solution.

Limits:

– *any impurity*: maximum 0.5 per cent;

– *total*: maximum 5.0 per cent.

Water (*2.5.12*): maximum 5.0 per cent, determined on 0.250 g.

ASSAY

Dissolve 0.250 g, accurately weighed, in 10 mL of *methylene chloride R* and add 30 mL of *anhydrous acetic acid R*. Titrate with *0.1 M perchloric acid*, determining the end-point potentiometrically (*2.2.20*).

1 mL of *0.1 M perchloric acid* is equivalent to 39.05 mg of $C_{22}H_{39}NaO_4$.

FUNCTIONALITY-RELATED CHARACTERISTICS

This section provides information on characteristics that are recognised as being relevant control parameters for one or more functions of the substance when used as an excipient (see chapter 5.15). This section is a non-mandatory part of the monograph and it is not necessary to verify the characteristics to demonstrate compliance. Control of these characteristics can however contribute to the quality of a medicinal product by improving the consistency of the manufacturing process and the performance of the medicinal product during use. Where control methods are cited, they are recognised as being suitable for the purpose, but other methods can also be used. Wherever results for a particular characteristic are reported, the control method must be indicated.

The following characteristics may be relevant for sodium stearyl fumarate used as a lubricant in tablets and capsules.

Particle-size distribution (*2.9.31*).

Specific surface area (*2.9.26, Method I*).

01/2008:0099
corrected 6.0

SODIUM SULFATE, ANHYDROUS

Natrii sulfas anhydricus

Na_2SO_4 M_r 142.0
[7757-82-6]