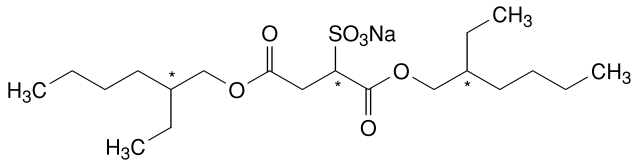
01/2008:1418

DOCUSATE SODIUM

Natrii docusas



$C_{20}H_{37}NaO_7S$ M_r 444.6
[577-11-7]

DEFINITION

Sodium 1,4-bis[(2-ethylhexyl)oxy]-1,4-dioxobutane-2-sulphonate.

Content: 98.0 to 101.0 per cent (anhydrous substance).

CHARACTERS

Appearance: white or almost white, waxy masses or flakes, hygroscopic.

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

IDENTIFICATION

A. Infrared absorption spectrophotometry (*2.2.24*).

Preparation: place about 3 mg of the substance to be examined on a sodium chloride plate, add 0.05 ml of *acetone R* and immediately cover with another sodium chloride plate. Rub the plates together to dissolve the substance to be examined, slide the plates apart and allow the acetone to evaporate.

Comparison: *Ph. Eur. reference spectrum of docusate sodium*.

B. In a crucible, ignite 0.75 g in the presence of *dilute sulphuric acid R*, until an almost white residue is obtained. Allow to cool and take up the residue with 5 ml of *water R*. Filter. 2 ml of the filtrate gives reaction (a) of sodium (*2.3.1*).

TESTS

Alkalinity. Dissolve 1.0 g in 100 ml of a mixture of equal volumes of *methanol R* and *water R*, previously neutralised to *methyl red solution R*. Add 0.1 ml of *methyl red solution R*. Not more than 0.2 ml of *0.1 M hydrochloric acid* is required to change the colour of the indicator to red.

Related non-ionic substances. Gas chromatography (*2.2.28*).

Internal standard solution. Dissolve 10 mg of *methyl behenate R* in *hexane R* and dilute to 50 ml with the same solvent.

Test solution (a). Dissolve 0.10 g of the substance to be examined in 2.0 ml of the internal standard solution and dilute to 5.0 ml with *hexane R*. Pass the solution, at a rate of about 1.5 ml/min, through a column 10 mm in internal diameter, packed with 5 g of *basic aluminium oxide R* and previously washed with 25 ml of *hexane R*. Elute with 5 ml of *hexane R* and discard the eluate. Elute with 20 ml of a mixture of equal volumes of *ether R* and *hexane R*. Evaporate the eluate to dryness and dissolve the residue in 2.0 ml of *hexane R*.

Test solution (b). Prepare as described for test solution (a) but dissolving 0.10 g of the substance to be examined in *hexane R*, diluting to 5.0 ml with the same solvent, and using a new column.

Reference solution. Dilute 2.0 ml of the internal standard solution to 5.0 ml with *hexane R*.

Column:

– *material*: glass,
– *size*: *l* = 2 m, Ø = 2 mm,
– *stationary phase*: *silanised diatomaceous earth for gas chromatography R* impregnated with 3 per cent *m/m* of *polymethylphenylsiloxane R*.

Carrier gas: *nitrogen for chromatography R*.

Flow rate: 30 ml/min.

Temperature:

– *column*: 230 °C,
– *injection port and detector*: 280 °C.

Detection: flame ionisation.

Injection: 1 µl.

Run time: 2.5 times the retention time of the internal standard.

System suitability: there is no peak with the same retention time as the internal standard in the chromatogram obtained with test solution (b).

Limits: test solution (a):

– *any impurity*: for each impurity, not more than the area of the peak due to the internal standard (0.4 per cent).

Chlorides: maximum 350 ppm.

Dissolve 5.0 g in 50 ml of *ethanol (50 per cent V/V) R* and add 0.1 ml of *potassium dichromate solution R*. Not more than 0.5 ml of *0.1 M silver nitrate* is required to change the colour of the indicator from yellow to orange.

Sodium sulphate: maximum 2 per cent.

Dissolve 0.25 g in 40 ml of a mixture of 20 volumes of *water R* and 80 volumes of *2-propanol R*. Adjust to pH between 2.5 and 4.0 using *perchloric acid solution R*. Add 0.4 ml of *naphtharson solution R* and 0.1 ml of a 0.125 g/l solution of *methylene blue R*. Not more than 1.5 ml of *0.025 M barium perchlorate* is required to change the colour of the indicator from yellowish-green to yellowish-pink.

Heavy metals (*2.4.8*): maximum 10 ppm.

Dissolve 4.0 g in *ethanol (80 per cent V/V) R* and dilute to 20 ml with the same solvent. 12 ml of the solution complies with test B. Prepare the reference solution using lead standard solution (2 ppm Pb) obtained by diluting *lead standard solution (100 ppm Pb) R* with *ethanol (80 per cent V/V) R*.

Water (*2.5.12*): maximum 3.0 per cent, determined on 0.250 g.

ASSAY

To 1.000 g in a 250 ml conical flask fitted with a reflux condenser add 25.0 ml of *0.5 M alcoholic potassium hydroxide* and heat on a water-bath under reflux for 45 min. Allow to cool. Add 0.25 ml of *phenolphthalein solution R1* and titrate with *0.5 M hydrochloric acid* until the red colour disappears. Carry out a blank titration.

1 ml of *0.5 M alcoholic potassium hydroxide* is equivalent to 0.1112 g of $C_{20}H_{37}NaO_7S$.

STORAGE

In an airtight container.