Column:

– *size*: *l* = 0.25 m, Ø = 4.6 mm,

– *stationary phase*: spherical *octylsilyl silica gel for chromatography R* (5 µm).

Mobile phase: *glacial acetic acid R, water R, methanol R* (10:190:800 *V/V/V*).

Flow rate: 1 ml/min.

Detection: spectrophotometer at 589 nm.

Injection: 20 µl.

Run time: 2.5 times the retention time of the principal peak.

System suitability: reference solution (a):

– *resolution*: peak due to pentamethyl-*p*-rosanilinium is baseline separated from the peak due to methylrosanilinium.

Locate the peak due to pentamethyl-*p*-rosanilinium using the chromatogram provided with *methylrosanilinium for system suitability CRS*.

Limits:

– *pentamethyl-p-rosanilinium*: maximum 10 per cent.

Related substances. Liquid chromatography (*2.2.29*) as described in the test for pentamethyl-*p*-rosanilinium.

Limits:

– *impurity A*: maximum 1.0 per cent;

– *any other impurity*: for each impurity, maximum 0.1 per cent;

– *sum of impurities other than A*: maximum 1.0 per cent;

– *disregard limit*: the area of the principal peak in the chromatogram obtained with reference solution (b) (0.05 per cent); disregard the peak due to pentamethyl-*p*-rosanilinium.

Substances insoluble in ethanol (90 per cent *V/V*): maximum 0.5 per cent.

In a conical flask introduce 1.0 g and add 50 ml of *ethanol (90 per cent V/V) R*. Boil under a reflux condenser for 1 h. Filter the warm liquid through a weighed sintered glass filter (16) (*2.1.2*) previously dried at 100-105 °C. Wash with hot *ethanol (90 per cent V/V) R* until a colourless filtrate is obtained. Dry at 100-105 °C until constant weight.

Water (*2.5.12*): maximum 10.0 per cent, determined on 0.100 g.

Sulphated ash (*2.4.14*): maximum 1.5 per cent, determined on 1.0 g.

ASSAY

Dissolve 50.0 mg in *ethanol (96 per cent) R* and dilute to 100.0 ml with the same solvent. Dilute 2.0 ml of the solution to 250.0 ml with *ethanol (96 per cent) R*. Measure the absorbance (*2.2.25*) at the maximum at 589 nm.

Calculate the content of $C_{25}H_{30}ClN_3$ taking the specific absorbance to be 2605.

STORAGE

In an airtight container.

IMPURITIES

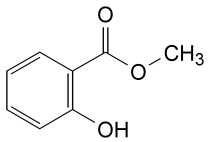
Specified impurities: A.

A. impurity of unknown structure with a relative retention of about 0.7.

01/2008:0230

METHYL SALICYLATE

Methylis salicylas



$C_8H_8O_3$ M_r 152.1

[119-36-860-7]

DEFINITION

Methyl 2-hydroxybenzoate.

Content: 99.0 per cent *m/m* to 100.5 per cent *m/m*.

CHARACTERS

Appearance: colourless or slightly yellow liquid.

Solubility: very slightly soluble in water, miscible with ethanol (96 per cent) and with fatty and essential oils.

IDENTIFICATION

A. Heat 0.25 ml with 2 ml of *dilute sodium hydroxide solution R* on a water-bath for 5 min. Add 3 ml of *dilute sulphuric acid R*. A crystalline precipitate is formed. Filter. The precipitate washed with *water R* and dried at 100-105 °C, melts (*2.2.14*) at 156 °C to 161 °C.

B. To 10 ml of a saturated solution add 0.05 ml of *ferric chloride solution R1*. A violet colour develops.

TESTS

Appearance of solution. The solution is clear (*2.2.1*) and not more intensely coloured than reference solution Y_7 (*2.2.2, Method II*).

To 2 ml add 10 ml of *ethanol (96 per cent) R*.

Acidity. Dissolve 5.0 g in a mixture of 0.2 ml of *bromocresol green solution R* and 50 ml of *ethanol (96 per cent) R* previously neutralised to a blue colour by addition of *0.1 M sodium hydroxide*. Not more than 0.4 ml of *0.1 M sodium hydroxide* is required to restore the blue colour.

Relative density (*2.2.5*): 1.180 to 1.186.

Refractive index (*2.2.6*): 1.535 to 1.538.

ASSAY

Dissolve 0.500 g in 25 ml of *ethanol (96 per cent) R*. Add 0.05 ml of *phenol red solution R* and neutralise with *0.1 M sodium hydroxide*. To the neutralised solution add 50.0 ml of *0.1 M sodium hydroxide* and heat under a reflux condenser on a water-bath for 30 min. Cool and titrate with *0.1 M hydrochloric acid*. Calculate the volume of *0.1 M sodium hydroxide* used in the saponification. Carry out a blank titration.

1 ml of *0.1 M sodium hydroxide* is equivalent to 15.21 mg of $C_8H_8O_3$.

STORAGE

Protected from light.