Iron (*2.4.9*): maximum 20 ppm.

Dissolve 2.5 g in 10 ml of *dilute hydrochloric acid R* with heating, evaporate to dryness, with stirring, and dissolve the residue in 25 ml of hot *water R*. Dilute 5 ml of the obtained solution to 10 ml with *water R*. The solution complies with the limit test for iron.

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

12 ml of the solution obtained in the test for iron complies with limit test A. Prepare the standard using *lead standard solution (1 ppm Pb) R*.

ASSAY

Dissolve 0.300 g in 50.0 ml of *water R*. Dilute 10.0 ml of the solution to 50 ml with *water R* and add 10 ml of *dilute sulphuric acid R*. Titrate with *0.02 M potassium permanganate*.

1 ml of *0.02 M potassium permanganate* is equivalent to 7.693 mg of NaH_8BO_7.

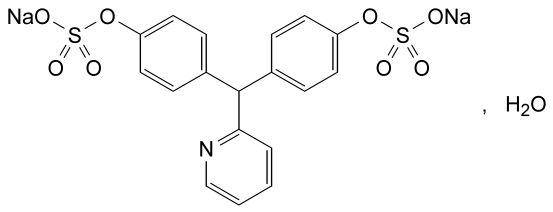
STORAGE

In an airtight container.

01/2008:1031

SODIUM PICOSULFATE

Natrii picosulfas



$C_{18}H_{13}NNa_2O_8S_2,H_2O$ M_r 499.4

DEFINITION

Sodium picosulfate contains not less than 98.5 per cent and not more than the equivalent of 100.5 per cent of 4,4′-(pyridin-2-ylmethylene)bisphenyl bis(sodium sulphate), calculated with reference to the anhydrous substance.

CHARACTERS

A white or almost white, crystalline powder, freely soluble in water, slightly soluble in alcohol.

IDENTIFICATION

First identification: A, E.

Second identification: B, C, D, E.

A. Examine by infrared absorption spectrophotometry (*2.2.24*), comparing with the spectrum obtained with *sodium picosulfate CRS*. Examine the substances prepared as discs.

B. Examine the chromatograms obtained in the test for related substances in ultraviolet light at 254 nm. The principal spot in the chromatogram obtained with test solution (b) is similar in position and size to the principal spot in the chromatogram obtained with reference solution (a).

C. To 5 ml of solution S (see Tests) add 1 ml of *dilute hydrochloric acid R* and heat to boiling. Add 1 ml of *barium chloride solution R1*. A white precipitate is formed.

D. To about 10 mg add 3 ml of *sulphuric acid R* and 0.1 ml of *potassium dichromate solution R1*. A violet colour develops.

E. The solution S gives reaction (a) of sodium (*2.3.1*).

TESTS

Solution S. Dissolve 2.5 g in *distilled water R* and dilute to 50 ml with the same solvent.

Appearance of solution. Solution S is clear (*2.2.1*) and not more intensely coloured than reference solution GY_7 (*2.2.2, Method II*).

Acidity or alkalinity. To 10 ml of solution S add 0.05 ml of *phenolphthalein solution R*. The solution is colourless. Not more than 0.25 ml of *0.01 M sodium hydroxide* is required to change the colour of the indicator to pink.

Related substances. Examine by thin-layer chromatography (*2.2.27*), using *silica gel* GF_{254} *R* as the coating substance.

Test solution (a). Dissolve 0.20 g of the substance to be examined in *methanol R* and dilute to 5 ml with the same solvent.

Test solution (b). Dilute 1 ml of test solution (a) to 10 ml with *methanol R*.

Reference solution (a). Dissolve 20 mg of *sodium picosulfate CRS* in *methanol R* and dilute to 5 ml with the same solvent.

Reference solution (b). Dilute 2 ml of test solution (b) to 100 ml with *methanol R*.

Reference solution (c). Dissolve 0.20 g of the substance to be examined in 2 ml of a 103 g/l solution of *hydrochloric acid R*. Heat rapidly to boiling and maintain boiling for 10 s. Cool in iced water and dilute to 10 ml with *methanol R*.

Apply to the plate 5 µl of each solution. Develop over a path of 10 cm using a mixture of 2.5 volumes of *anhydrous formic acid R*, 12.5 volumes of *water R*, 25 volumes of *methanol R* and 60 volumes of *ethyl acetate R*. Dry the plate in a current of hot air for 15 min and examine in ultraviolet light at 254 nm. Spray with a 200 g/l solution of *hydrochloric acid R* in *methanol R* and heat at 110 °C for 10 min. Spray the hot plate with a freshly prepared solution containing 50 g/l of *ferric chloride R* and 1 g/l of *potassium ferricyanide R*. Examine the wet plate. Any spot in the chromatogram obtained with test solution (a), apart from the principal spot, is not more intense than the spot in the chromatogram obtained with reference solution (b) (0.2 per cent). The test is not valid unless the chromatogram obtained with reference solution (c) shows three clearly separated spots. A fourth spot may be present on the starting-line.

Chlorides (*2.4.4*). Dilute 5 ml of solution S to 15 ml with *water R*. The solution complies with the limit test for chlorides (200 ppm).

Sulphates (*2.4.13*). Dilute 7.5 ml of solution S to 15 ml with *distilled water R*. The solution complies with the limit test for sulphates (400 ppm).

Heavy metals (*2.4.8*). 12 ml of solution S complies with limit test A for heavy metals (20 ppm). Prepare the standard using 10 ml of *lead standard solution (1 ppm Pb) R*.

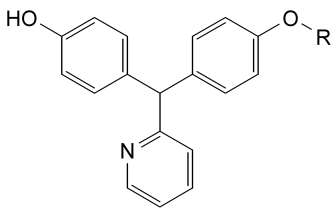
Water (*2.5.12*): 3.0 per cent to 5.0 per cent, determined on 0.500 g by the semi-micro determination of water.

ASSAY

Dissolve 0.400 g in 80 ml of *methanol 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 48.14 mg of $C_{18}H_{13}NNa_2O_8S_2$.

IMPURITIES



A. R = SO_3Na: 4-[(pyridin-2-yl)(4-hydroxyphenyl)methyl]phenyl sodium sulphate,

B. R = H: 4,4′-[(pyridin-2-yl)methylene]bisphenol.

01/2008:1909
corrected 6.0

SODIUM POLYSTYRENE SULPHONATE

Natrii polystyrenesulfonas

DEFINITION

Polystyrene sulphonate resin prepared in the sodium form.

Exchange capacity: 2.8 mmol to 3.4 mmol of potassium per gram (dried substance).

Content: 9.4 per cent to 11.0 per cent of Na (dried substance).

CHARACTERS

Appearance: almost white or light brown powder.

Solubility: practically insoluble in water, in alcohol and in methylene chloride.

IDENTIFICATION

A. Infrared absorption spectrophotometry (*2.2.24*).

Preparation: discs using finely ground substance.

Comparison: *Ph. Eur. reference spectrum of sodium polystyrene sulphonate.*

B. Suspend 0.1 g in *water R*, add 2 ml of a 150 g/l solution of *potassium carbonate R*, and heat to boiling. Allow to cool and filter. To the filtrate add 4 ml of *potassium pyroantimonate solution R* and heat to boiling. Allow to cool in iced water and if necessary rub the inside of the test-tube with a glass rod. A dense white precipitate is formed.

TESTS

Styrene. Liquid chromatography (*2.2.29*).

Test solution. Shake 10.0 g of the substance to be examined with 10 ml of *acetone R* for 30 min, centrifuge and use the supernatant liquid.

Reference solution. Dissolve 10 mg of *styrene R* in *acetone R* and dilute to 100 ml with the same solvent. Dilute 1 ml of this solution to 100 ml with *acetone R*.

Column:

– *size*: *l* = 0.25 m, Ø = 4 mm,

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

Mobile phase: *acetonitrile R, water R* (1:1 *V/V*).

Flow rate: 2 ml/min.

Detection: spectrophotometer at 254 nm.

Injection: 20 µl.

Limit:

– *styrene*: not more than the area of the principal peak in the chromatogram obtained with the reference solution (1 ppm).

Calcium: maximum 0.10 per cent.

Atomic emission spectrometry (*2.2.22, Method I*).

Test solution. To 1.10 g add 5 ml of *hydrochloric acid R*, heat to boiling, cool and add 10 ml of *water R*. Filter, wash the filter and residue with *water R* and dilute the filtrate and washing to 25.0 ml with *water R*.

Reference solutions. Prepare the reference solutions using *calcium standard solution (400 ppm Ca) R*, diluted as necessary with *water R*.

Wavelength: 422.7 nm.

Potassium: maximum 0.10 per cent.

Atomic emission spectrometry (*2.2.22, Method I*).

Test solution. To 1.10 g add 5 ml of *hydrochloric acid R*, heat to boiling, cool and add 10 ml of *water R*. Filter, wash the filter and residue with *water R* and dilute the filtrate and washings to 25.0 ml with *water R*.

Reference solutions. Prepare the reference solutions using *potassium standard solution (100 ppm K) R*, diluted as necessary with *water R*.

Wavelength: 766.5 nm.

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

Treat 1.0 g as described in limit test F. After the addition of the *buffer solution pH 3.5 R* and of the *thioacetamide reagent R*, dilute to 50 ml with *water R* and continue as described in limit test E, beginning at the words “mix and allow to stand for 10 min...”.

Prepare the standard using 10 ml of *lead standard solution (1 ppm Pb) R*.

Loss on drying (*2.2.32*): maximum 7.0 per cent, determined on 1.000 g by drying in an oven at 105 °C.

Microbial contamination (*2.6.13*): not more than 10^2 enterobacteria and certain other gram-negative bacteria per gram.

ASSAY

Sodium. Atomic emission spectrometry (*2.2.22, Method I*).

Test solution. In a platinum crucible moisten 0.90 g with a few drops of *sulphuric acid R*, ignite very gently and allow to cool. Moisten with a few drops of *sulphuric acid R* again, ignite at 800 ± 50 °C until a carbon-free ash is obtained and allow to cool.

Add 20 ml of *water R* to the crucible, warm gently on a water-bath until dissolution, cool, transfer quantitatively to a 100 ml graduated flask and dilute to 100.0 ml with *water R*. Dilute 5 ml of this solution to 1000.0 ml with *water R*.

Reference solutions. Prepare the reference solutions using *sodium standard solution (200 ppm Na) R*, diluted as necessary with *water R*.

Wavelength: 589 nm.

Exchange capacity. Atomic emission spectrometry (*2.2.22, Method I*).

Solution A. 9.533 g/l solution of *potassium chloride R*.

Test solution. To 1.6 g of the substance to be examined in a dry 250 ml ground-glass-stoppered flask add 100 ml of solution A, stopper and shake for 15 min. Filter, discard the first 20 ml of the filtrate and dilute 4 ml of the filtrate to 1000 ml with *water R*.