Absorption minimum: at 370 nm.
Specific absorbance at the absorption maximum: 0.65 to 0.80.

B. Dissolve about 20 mg in 2 ml of water R and add 0.1 ml of sodium sulphide solution R. A deep violet-red colour is produced.

C. Dissolve 50 mg in 1 ml of water R and acidify the solution by the addition of hydrochloric acid R. Place a drop of the solution in an oxidising flame. A persistent yellow colour is produced.

TESTS

Insoluble matter: maximum 100 ppm.

Dissolve 10 g without heating in 50 ml of water R. Allow to stand for 30 min and filter through a sintered-glass filter (16) (2.1.2). Wash the filter with cold water R until the filtrate is colourless. Dry the residue on the filter at 105 °C. The residue weighs a maximum of 1 mg.

Chlorides (2.4.4): maximum 200 ppm.

In a metallic crucible (nickel) mix 1.0 g with 8 ml of a 200 g/l solution of sodium hydroxide R. Heat slowly and evaporate carefully to dryness over a small flame, then heat to a dull red colour for 30 min. Allow to cool and transfer the solid residue with 3 quantities, each of 8 ml, of dilute sulphuric acid R. Filter the sulphuric acid extracts on a filter paper and collect the filtrates. Render the filtrate acid to litmus paper R by adding, if necessary, a few drops of dilute sulphuric acid R. Wash the crucible and the filter paper with 3 quantities, each of 10 ml, of water R, add the washings to the main sulphuric acid solution and dilute to 60 ml with water R. Mix.

Ferricyanides: maximum 200 ppm.

Dissolve 1.25 g in acetate buffer solution pH 4.6 R and dilute to 50.0 ml with the same buffer solution. Use three 50 ml volumetric flasks (A, B, C). To flask B add 1.0 ml of ferricyanide standard solution (50 ppm Fe(CN)6 R). To flasks A and B add 1 ml of a 5 g/l solution of ferrous ammonium sulphate R. To the 3 flasks add 10.0 ml of the solution of the substance to be examined. Dilute the contents of each flask to 50.0 ml with water R. Allow to stand for 30 min. The absorbance (2.2.25) of the solution in flask A measured at 720 nm using the solution in flask C as the compensation liquid is not greater than the absorbance of the solution in flask B measured at 720 nm using the solution in flask A as the compensation liquid.

Ferrocyanides: maximum 200 ppm.

Dissolve 4.0 g in water R and dilute to 100.0 ml with the same solvent. Use three 50 ml volumetric flasks (A, B, C). To flask B add 2.0 ml of ferrocyanide standard solution (100 ppm Fe(CN)6 R). To flasks A and B add 1 ml of ferric chloride solution R2. To the 3 flasks add 25.0 ml of the solution of the substance to be examined. Dilute the contents of each flask to 50.0 ml with water R. Allow to stand for 30 min. The absorbance (2.2.25) of the solution in flask A measured at 695 nm using the solution in flask C as the compensation liquid is not greater than the absorbance of the solution in flask B measured at 695 nm using the solution in flask A as the compensation liquid.

Sulphates: maximum 100 ppm.

Test solution. Dissolve 3.6 g in 120 ml of distilled water R, add with mixing 4 ml of sulphate standard solution (10 ppm SO4 R) and 20 ml of a 250 g/l solution of cupric chloride R and dilute to 150.0 ml with distilled water R. Allow to stand for 16 h and filter or centrifuge until a clear light-blue solution is obtained.

Reference solution. To 40 ml of sulphate standard solution (10 ppm SO4 R) add 80 ml of distilled water R and 12.13 ml of a 250 g/l solution of cupric chloride R. Dilute to 150.0 ml with distilled water R. The volume of cupric chloride solution added is such that the colour of the final solution matches that of the test solution. Allow the solutions to stand. Filter both solutions, discarding the first 25 ml of filtrate. To 100 ml of each filtrate, add 0.5 ml of acetic acid R. Mix and add 2 ml of a 250 g/l solution of barium chloride R and mix again. The test solution is not more opalescent than the reference solution.

Water (2.5.12): 9.0 per cent to 15.0 per cent, determined on 0.250 g.

ASSAY

Dissolve 0.250 g in 100 ml of water R and add 0.1 ml of dilute sulphuric acid R. Titrate with 0.1 M silver nitrate, determining the end-point potentiometrically (2.2.20) with a silver-mercurous sulphate electrode system.

1 ml of 0.1 M silver nitrate is equivalent to 13.10 mg of Na2[Fe(CN)5(NO)].

STORAGE

Protected from light.

01/2008:1997 corrected 6.0

SODIUM PERBORATE, HYDRATED

Natrii perboras hydricus

NaBO3.H2O or NaBO3.H2O.3H2O

M, 153.9

DEFINITION

Content: 96.0 per cent to 103.0 per cent.

CHARACTERS

Appearance: colourless, prismatic crystals or white or almost white powder, stable in the crystalline form.

Solubility: sparingly soluble in water, with slow decomposition. It dissolves in dilute mineral acids.

IDENTIFICATION

A. Mix 1 ml of a saturated solution with a mixture of 1 ml of dilute sulphuric acid R and 0.2 ml of a 70 g/l solution of potassium dichromate R, shake with 2 ml of ether R and allow to stand. A blue colour is produced in the ether layer.

B. The mixture obtained by treating about 100 mg with 0.1 ml of sulphuric acid R and 5 ml of methanol R burns with a greenish flame when ignited.

C. It gives reaction (a) of sodium (2.3.1).

TESTS

Chlorides (2.4.4): maximum 330 ppm.

Dissolve 0.15 g in 15 ml of water R. The solution complies with the limit test for chlorides.

Sulphates (2.4.13): maximum 1.2 per cent.

Dissolve 0.13 g in 150 ml of distilled water R. 15 ml of the solution complies with the limit test for sulphates.
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 NaH₂BO₃.

STORAGE
In an airtight container.

01/2008:1031

SODIUM PICOSULFATE
Natrii picosulfas

C₁₈H₁₃NNa₂O₈S₂.H₂O  M, 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 GY₁ (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 GF₂₅₄ 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 105 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 methanol 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 C₁₈H₁₃NNa₂O₈S₂.