

STORAGE

In an airtight container.



01/2016:0565

SODIUM NITROPRUSSIDE

Natrii nitroprussias

$\text{Na}_2[\text{Fe}(\text{CN})_5(\text{NO})]\cdot 2\text{H}_2\text{O}$
[13755-38-9]

 M_r 298.0

DEFINITION

Sodium pentacyanonitrosylferrate (III) dihydrate.

Content: 99.0 per cent to 100.5 per cent (anhydrous substance).

CHARACTERS

Appearance: reddish-brown powder or crystals.

Solubility: freely soluble in water, slightly soluble in ethanol (96 per cent).

IDENTIFICATION

A. Ultraviolet and visible absorption spectrophotometry (2.2.25).

Test solution. Dissolve 0.700 g in *water R* and dilute to 100.0 mL with the same solvent. Examine the solution immediately after preparation.

Spectral range: 350-600 nm.

Absorption maximum: at 395 nm.

Shoulder: at about 510 nm.

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 sulfide 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 sulfuric acid R*.

Filter the sulfuric acid extracts on a filter paper and collect the filtrates. Render the filtrate acid to *blue litmus paper R* by adding, if necessary, a few drops of *dilute sulfuric 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 sulfuric 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 sulfate 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.

Sulfates: maximum 100 ppm.

Test solution. Dissolve 3.6 g in 120 mL of *distilled water R*, add with mixing 4 mL of *sulfate 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 *sulfate 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 sulfuric acid R*. Titrate with 0.1 M *silver nitrate*, determining the end-point potentiometrically (2.2.20).

1 mL of 0.1 M *silver nitrate* is equivalent to 13.10 mg of $\text{Na}_2[\text{Fe}(\text{CN})_5(\text{NO})]$.

STORAGE

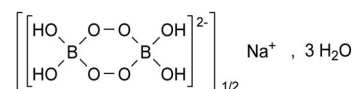
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01/2017:1997

SODIUM PERBORATE, HYDRATED

Natrii perboras hydricus



$\text{NaBO}_3\cdot 4\text{H}_2\text{O}$ or $\text{NaBO}_2\cdot \text{H}_2\text{O}_2\cdot 3\text{H}_2\text{O}$

 M_r 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. Dissolve 20 mg in a mixture of 1 mL of *dilute sulfuric acid R* and 1 mL of *water R*. Add 1 mL of *potassium iodide solution R*. A reddish-brown colour appears.
- B. The mixture obtained by treating about 100 mg with 0.1 mL of *sulfuric 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*.

Sulfates (2.4.13): maximum 1.2 per cent.

Dissolve 0.13 g in 150 mL of *distilled water R*.

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*.

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 sulfuric acid R*. Titrate with 0.02 M *potassium permanganate*.

1 mL of 0.02 M *potassium permanganate* is equivalent to 7.693 mg of NaH₈BO₇.

STORAGE

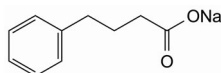
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01/2017:2183
corrected 10.0



SODIUM PHENYLBUTYRATE

Natrii phenylbutyras



C₁₀H₁₁NaO₂
[1716-12-7]

M_r 186.2

DEFINITION

Sodium 4-phenylbutanoate.

Content: 99.0 per cent to 101.0 per cent (anhydrous substance).

CHARACTERS

Appearance: white or yellowish-white powder.

Solubility: freely soluble in water and in methanol, practically insoluble in methylene chloride.

IDENTIFICATION

- A. Infrared absorption spectrophotometry (2.2.24).

Comparison: *sodium phenylbutyrate CRS*.

- B. Dissolve 0.15 g in 2 mL of *water R*. The solution gives reaction (a) of sodium (2.3.1).

TESTS

pH (2.2.3): 6.5 to 7.5.

Dissolve 0.20 g in *carbon dioxide-free water R* and dilute to 10 mL with the same solvent.

Impurity C. Gas chromatography (2.2.28).

Silylation solution. To 2 mL of *N,O-bis(trimethylsilyl)trifluoroacetamide R* add 0.04 mL of *chlorotrimethylsilane R* and mix.

Test solution. Dissolve 50.0 mg of the substance to be examined in 3 mL of *water R* and add 0.5 mL of *hydrochloric acid R*. Extract with 2 quantities, each of 5 mL, of *methylene chloride R*. Evaporate the combined methylene chloride extracts to dryness in a vial with a screw cap and add 0.5 mL of the silylation solution. Seal the vial and heat at 70 ± 5 °C for 20 min.

Reference solution (a). Dissolve 5.0 mg of *sodium phenylbutyrate impurity C CRS* in *methylene chloride R* and dilute to 10.0 mL with the same solvent.

Reference solution (b). Dilute 1.0 mL of reference solution (a) to 10.0 mL with *methylene chloride R*. Place 1.0 mL of this solution in a vial with a screw cap, evaporate to dryness and add 0.5 mL of the silylation solution. Seal the vial and heat at 70 ± 5 °C for 20 min.

Reference solution (c). Dissolve 10 mg of the substance to be examined in 25 mL of *water R*. To 3 mL of this solution add 0.1 mL of *hydrochloric acid R*. Extract with 2 quantities, each of 5 mL, of *methylene chloride R*. Combine the methylene chloride extracts in a vial with a screw cap and add 2 mL of reference solution (a). Evaporate to dryness and add 0.5 mL of the silylation solution. Seal the vial and heat at 70 ± 5 °C for 20 min.

Column:

- **material:** fused silica;
- **size:** *l* = 25 m, Ø = 0.25 mm;
- **stationary phase:** *phenyl(5)methyl(95)polysiloxane R* (film thickness 1.0 µm).

Carrier gas: *helium for chromatography R*.

Flow rate: 0.9 mL/min.

Split ratio: 1:100.

Temperature:

	Time (min)	Temperature (°C)
Column	0 - 5	50
	5 - 27	50 → 270
	27 - 32	270
Injection port		270
Detector		270

Detection: flame ionisation.

Injection: 1 µL.

Relative retention with reference to phenylbutyrate (retention time = about 20 min): impurity C = about 0.98.

System suitability: reference solution (c):

- **resolution:** minimum 3.0 between the peaks due to impurity C and phenylbutyrate.

Limit:

- **impurity C:** not more than the area of the corresponding peak in the chromatogram obtained with reference solution (b) (0.1 per cent).

Related substances. Liquid chromatography (2.2.29).

Test solution. Dissolve 0.20 g of the substance to be examined in 10 mL of *methanol R* and dilute to 50.0 mL with *water R*.

Reference solution (a). Dissolve 4.0 mg of *α-tetralone R* (impurity B) in 10 mL of *methanol R* and dilute to 200.0 mL with the same solvent.