

Related substances. Examine by thin-layer chromatography (2.2.27), using as the coating substance a suitable octadecylsilyl silica gel with a fluorescent indicator having an optimal intensity at 254 nm.

Test solution (a). Dissolve 0.100 g in 10 ml of *water R*. Immediately add 2 ml of *hydrochloric acid R* and shake with 50 ml of *ether R*. Evaporate the upper layer to dryness and dissolve the residue to 10 ml with *acetone R*.

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

Reference solution (a). Dissolve 34.3 mg of *4-hydroxybenzoic acid R* in *acetone R* and dilute to 100 ml with the same solvent.

Reference solution (b). Dilute 0.5 ml of test solution (a) to 100 ml with *acetone R*.

Reference solution (c). Dissolve 10 mg of *methyl parahydroxybenzoate CRS* in *acetone R* and dilute to 10 ml with the same solvent.

Reference solution (d). Dissolve 10 mg of *ethyl parahydroxybenzoate CRS* in 1 ml of test solution (a) and dilute to 10 ml with *acetone R*.

Apply separately to the plate 5 µl of each solution. Develop over a path of 15 cm using a mixture of 1 volume of *glacial acetic acid R*, 30 volumes of *water R* and 70 volumes of *methanol R*. Allow the plate to dry in air and examine in ultraviolet light at 254 nm. In the chromatogram obtained with test solution (a): any spot due to 4-hydroxybenzoic acid is not more intense than the spot in the chromatogram obtained with reference solution (a) (4 per cent) and any spot, apart from the principal spot and the spot due to 4-hydroxybenzoic acid, is not more intense than the spot in the chromatogram obtained with reference solution (b) (0.5 per cent). The test is not valid unless the chromatogram obtained with reference solution (d) shows two clearly separated principal spots.

Chlorides (2.4.4). To 10 ml of solution S, add 30 ml of *water R* and 1 ml of *nitric acid R* and dilute to 50 ml with *water R*. Shake and filter. Dilute 10 ml of the filtrate to 15 ml with *water R*. The solution complies with the limit test for chlorides (350 ppm). Prepare the standard using 14 ml of *chloride standard solution (5 ppm Cl) R* to which 1 ml of *water R* has been added.

Sulphates (2.4.13). To 25 ml of solution S, add 5 ml of *distilled water R* and 10 ml of *hydrochloric acid R* and dilute to 50 ml with *distilled water R*. Shake and filter. Dilute 10 ml of the filtrate to 15 ml with *distilled water R*. The solution complies with the limit test for sulphates (300 ppm).

Heavy metals (2.4.8). 2.0 g complies with limit test C for heavy metals (10 ppm). Prepare the standard using 2 ml of *lead standard solution (10 ppm Pb) R*.

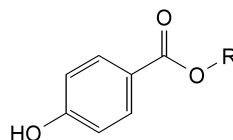
Water (2.5.12). Not more than 5.0 per cent, determined on 0.500 g by the semi-micro determination of water.

ASSAY

Dissolve 0.150 g in 50 ml of *anhydrous acetic acid 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 17.41 mg of $C_8H_7NaO_3$.

IMPURITIES



A. R = H: 4-hydroxybenzoic acid,

B. R = CH_2-CH_3 : ethyl 4-hydroxybenzoate,

C. R = $CH_2-CH_2-CH_3$: propyl 4-hydroxybenzoate,

D. R = $CH_2-CH_2-CH_2-CH_3$: butyl 4-hydroxybenzoate.

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corrected 6.0

SODIUM MOLYBDATE DIHYDRATE

Natrii molybdas dihydricus

$Na_2MoO_4 \cdot 2H_2O$
[10102-40-6]

M_r 241.9

DEFINITION

Content: 98.0 per cent to 100.5 per cent (dried substance).

CHARACTERS

Appearance: white or almost white powder or colourless crystals.

Solubility: freely soluble in water.

IDENTIFICATION

A. Loss on drying (see Tests).

B. Dissolve 0.2 g in 5 ml of a mixture of equal volumes of *nitric acid R* and *water R* and add 0.1 g of *ammonium chloride R*. Add 0.3 ml of *disodium hydrogen phosphate solution R* and heat slowly at 50-60 °C. A yellow precipitate is formed.

C. Dissolve 0.15 g in 2 ml of *water R*, the solution gives reaction (a) of sodium (2.3.1).

TESTS

Solution S. Dissolve 10.0 g in *water R* and dilute to 50 ml with the same solvent.

Appearance of solution. Solution S is clear (2.2.1) and colourless (2.2.2, *Method II*).

Chlorides: maximum 50 ppm.

To 10 ml of a mixture of equal volumes of *nitric acid R* and *water R* add 10 ml of solution S with shaking. Add 1 ml of 0.1 M *silver nitrate*. Any opalescence in the solution is not more intense after 5 min than that of a standard solution prepared at the same time in the same manner with 2 ml of *chloride standard solution (50 ppm Cl) R*.

Phosphates: maximum 200 ppm.

Dissolve 2.0 g by heating in 13 ml of *water R*. In the still hot solution, dissolve 8.0 g of *ammonium nitrate R1*. Add this solution to 27 ml of a mixture of equal volumes of *nitric acid R* and *water R*. Any yellow colour or opalescence in the solution is not more intense within 3 h than that in a standard solution prepared at the same time as follows: dissolve 1.0 g in 12 ml of *water R* and add 1 ml of *phosphate standard solution (200 ppm PO_4) R*.

Ammonium (2.4.1, Method B): maximum 10 ppm, determined on 0.10 g.

Prepare the standard using 1 ml of *ammonium standard solution* (1 ppm NH_4) R.

Heavy metals: maximum 10 ppm.

To 10 ml of solution S, add 2 ml of *water R*, 6 ml of a 168 g/l solution of *sodium hydroxide R* and 2 ml of *concentrated ammonia R* (solution A). To 0.5 ml of *thioacetamide reagent R* add a mixture of 15 ml of solution A and 5 ml of *water R*. Any coloration of the solution is not more intense after 2 min than that of a reference solution prepared at the same time as follows: to 0.5 ml of *thioacetamide reagent R* add a mixture of 5 ml of solution A, 1 ml of *lead standard solution* (10 ppm Pb) R and 14 ml of *water R*.

Loss on drying (2.2.32): 14.0 per cent to 16.0 per cent, determined on 1.000 g by drying in an oven at 140 °C for 3 h.

ASSAY

Dissolve 0.100 g in 30 ml of *water R*, add 0.5 g of *hexamethylenetetramine R* and 0.1 ml of a 250 g/l solution of *nitric acid R*. Heat to 60 °C. Titrate with 0.05 M *lead nitrate* using 4-(2-pyridylazo)resorcinol monosodium salt R as indicator.

1 ml of 0.05 M *lead nitrate* is equivalent to 10.30 mg of Na_2MoO_4 .

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SODIUM NITRITE

Natrii nitris

NaNO_2
[7632-00-0]

M_r 69.0

DEFINITION

Content: 98.5 per cent to 100.5 per cent (dried substance).

CHARACTERS

Appearance: colourless crystals or mass or yellowish rods, hygroscopic.

Solubility: freely soluble in water, soluble in alcohol.

IDENTIFICATION

- Dilute 1 ml of solution S1 (see Tests) to 25 ml with *water R*. To 0.1 ml of the solution add 1 ml of *sulphanilic acid solution R1*. Allow to stand for 2-3 min. Add 1 ml of β -*naphthol solution R* and 1 ml of *dilute sodium hydroxide solution R*. An intense red colour develops.
- To 1 ml of the solution prepared for identification test A add 3 ml of a 20 g/l solution of *phenazone R* and 0.4 ml of *dilute sulphuric acid R*. An intense green colour develops.
- To 0.15 ml of solution S1, add 0.35 ml of *water R*. The solution gives reaction (b) of sodium (2.3.1).

TESTS

Solution S1. Dissolve 2.5 g in *carbon dioxide-free water R* and dilute to 50 ml with the same solvent.

Solution S2. Dissolve 3 g in *distilled water R*. Cautiously add 10 ml of *nitric acid R* and evaporate to dryness. Dissolve the residue in 10 ml of *distilled water R*, neutralise with *dilute sodium hydroxide solution R* and dilute to 30 ml with *distilled water R*.

Appearance of solution. Solution S1 is clear (2.2.1) and not more intensely coloured than reference solution B₆ (2.2.2, Method II).

Acidity or alkalinity. To 10 ml of solution S1, add 0.05 ml of *phenol red solution R*. Add 0.1 ml of 0.01 M *sodium hydroxide*. The solution is red. Add 0.3 ml of 0.01 M *hydrochloric acid*. The solution is yellow.

Chlorides (2.4.4): maximum 50 ppm.

Dilute 10 ml of solution S2 to 15 ml with *water R*. The solution complies with the limit test for chlorides.

Sulphates (2.4.13): maximum 200 ppm.

Dilute 7.5 ml of solution S2 to 15 ml with *distilled water R*. The solution complies with the limit test for sulphates.

Heavy metals (2.4.8): maximum 20 ppm.

Dilute 10 ml of solution S2 to 20 ml with *water R*. 12 ml of the solution complies with limit test A. Prepare the standard using *lead standard solution* (1 ppm Pb) R.

Loss on drying (2.2.32): maximum 1.0 per cent, determined on 1.000 g by drying *in vacuo*.

ASSAY

Dissolve 0.400 g in 100.0 ml of *water R*. Introduce 20.0 ml of the solution, while stirring continuously and keeping the tip of the pipette below the surface of the liquid, into a conical flask containing 30.0 ml of 0.1 M *cerium sulphate*. Immediately stopper the flask and allow to stand for 2 min. Add 10 ml of a 200 g/l solution of *potassium iodide R* and 2 ml of *starch solution R*.

While stirring continuously, titrate with 0.1 M *sodium thiosulphate* until the blue colour disappears. Carry out a blank titration.

1 ml of 0.1 M *cerium sulphate* is equivalent to 3.45 mg of NaNO_2 .

STORAGE

In an airtight container.

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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

- 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.