

System suitability: reference solution:

- **resolution:** minimum 7 between the peaks due to the iron complex of impurity A and the iron complex of edetic acid,
- **signal-to-noise ratio:** minimum 50 for the peak due to impurity A.

Limit:

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

Iron (2.4.9): maximum 80 ppm.

Dilute 2.5 ml of solution S to 10 ml with *water R*. Add 0.25 g of *calcium chloride R* to the test solution and the standard before the addition of the *thioglycollic acid R*.

Heavy metals (2.4.8): maximum 20 ppm.

1.0 g complies with test F. Prepare the reference solution using 2 ml of *lead standard solution (10 ppm Pb) R*.

ASSAY

Dissolve 0.300 g in *water R* and dilute to 300 ml with the same solvent. Add 2 g of *hexamethylenetetramine R* and 2 ml of *dilute hydrochloric acid R*. Titrate with *0.1 M lead nitrate*, using about 50 mg of *xylene orange triturate R* as indicator.

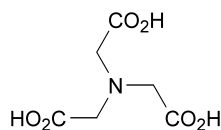
1 ml of *0.1 M lead nitrate* is equivalent to 37.22 mg of $\text{C}_{10}\text{H}_{14}\text{N}_2\text{Na}_2\text{O}_8 \cdot 2\text{H}_2\text{O}$.

STORAGE

Protected from light.

IMPURITIES

Specified impurities: A.



A. nitrilotriacetic acid.

01/2008:1509
corrected 6.0

DISODIUM PHOSPHATE, ANHYDROUS

Dinatrii phosphas anhydricus

Na_2HPO_4
[7558-79-4]

M_r 142.0

DEFINITION

Content: 98.0 per cent to 101.0 per cent (dried substance).

CHARACTERS

Appearance: white or almost white powder, hygroscopic.

Solubility: soluble in water, practically insoluble in ethanol (96 per cent).

IDENTIFICATION

- Solution S (see Tests) is slightly alkaline (2.2.4).
- Loss on drying (see Tests).
- Solution S gives reaction (b) of phosphates (2.3.1).
- Solution S gives reaction (a) of sodium (2.3.1).

TESTS

Solution S. Dissolve 5.0 g in *distilled water R* and dilute to 100.0 ml with the same solvent.

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

Reducing substances. To 10 ml of solution S add 5 ml of *dilute sulphuric acid R* and 0.25 ml of *0.02 M potassium permanganate* and heat on a water-bath for 5 min. The solution retains a slight red colour.

Monosodium phosphate: maximum 2.5 per cent.

From the volume of *1 M hydrochloric acid* (25 ml) and of *1 M sodium hydroxide* (n_1 ml and n_2 ml) used in the assay, calculate the following ratio:

$$\frac{n_2 - 25}{25 - n_1}$$

This ratio is not greater than 0.025.

Chlorides (2.4.4): maximum 200 ppm.

Dilute 5 ml of solution S to 15 ml with *dilute nitric acid R*.

Sulphates (2.4.13): maximum 500 ppm.

To 6 ml of solution S add 2 ml of *dilute hydrochloric acid R* and dilute to 15 ml with *distilled water R*.

Arsenic (2.4.2, *Method A*): maximum 2 ppm, determined on solution S.

Iron (2.4.9): maximum 20 ppm, determined on solution S.

Heavy metals (2.4.8): maximum 10 ppm.

12 ml of solution S complies with test A. Prepare the reference solution using 5 ml of *lead standard solution (1 ppm Pb) R* and 5 ml of *water R*.

Loss on drying (2.2.32): maximum 1.0 per cent, determined on 1.000 g by drying in an oven at 105 °C for 4 h.

ASSAY

Dissolve 1.600 g (m) in 25.0 ml of *carbon dioxide-free water R* and add 25.0 ml of *1 M hydrochloric acid*. Carry out a potentiometric titration (2.2.20) using *1 M sodium hydroxide*. Read the volume added at the 1st inflexion point (n_1 ml). Continue the titration to the 2nd inflexion point (total volume of *1 M sodium hydroxide* required, n_2 ml).

Calculate the percentage content of Na_2HPO_4 from the following expression:

$$\frac{1420 (25 - n_1)}{m (100 - d)}$$

d = percentage loss on drying.

STORAGE

In an airtight container.

01/2008:0602

DISODIUM PHOSPHATE DIHYDRATE

Dinatrii phosphas dihydricus

$\text{Na}_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$
[10028-24-7]

M_r 178.0

DEFINITION

Content: 98.0 per cent to 101.0 per cent (dried substance).

CHARACTERS

Appearance: white or almost white powder or colourless crystals.

Solubility: soluble in water, practically insoluble in ethanol (96 per cent).

IDENTIFICATION

01/2008:0118

- A. Solution S (see Tests) is slightly alkaline (2.2.4).
 B. Loss on drying (see Tests).
 C. Solution S gives reaction (b) of phosphates (2.3.1).
 D. Solution S gives reaction (a) of sodium (2.3.1).

TESTS

Solution S. Dissolve 5.0 g in *distilled water R* and dilute to 100 ml with the same solvent.

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

Reducing substances. To 5 ml of solution S add 5 ml of *dilute sulphuric acid R* and 0.25 ml of 0.02 M *potassium permanganate* and heat on a water-bath for 5 min. The solution retains a slight red colour.

Monosodium phosphate: maximum 2.5 per cent.

From the volume of 1 M *hydrochloric acid* (25 ml) and of 1 M *sodium hydroxide* (n_1 ml and n_2 ml) used in the assay, calculate the following ratio:

$$\frac{n_2 - 25}{25 - n_1}$$

This ratio is not greater than 0.025.

Chlorides (2.4.4): maximum 400 ppm.

To 2.5 ml of solution S add 10 ml of *dilute nitric acid R* and dilute to 15 ml with *water R*.

Sulphates (2.4.13): maximum 0.1 per cent.

To 3 ml of solution S add 2 ml of *dilute hydrochloric acid R* and dilute to 15 ml with *distilled water R*.

Arsenic (2.4.2, *Method A*): maximum 4 ppm, determined on 5 ml of solution S.

Iron (2.4.9): maximum 40 ppm

Dilute 5 ml of solution S to 10 ml with *water R*.

Heavy metals (2.4.8): maximum 20 ppm.

12 ml of solution S complies with test A. Prepare the reference solution using *lead standard solution* (1 ppm *Pb*) *R*.

Loss on drying (2.2.32): 19.5 per cent to 21.0 per cent, determined on 1.000 g by drying in an oven at 130 °C.

ASSAY

Dissolve 2.000 g (m) in 50 ml of *water R* and add 25.0 ml of 1 M *hydrochloric acid*. Carry out a potentiometric titration (2.2.20) using 1 M *sodium hydroxide*. Read the volume added at the 1st inflexion point (n_1 ml). Continue the titration to the 2nd inflexion point (total volume of 1 M *sodium hydroxide* required, n_2 ml).

Calculate the percentage content of Na_2HPO_4 from the following expression:

$$\frac{1420 (25 - n_1)}{m (100 - d)}$$

d = percentage loss on drying.

DISODIUM PHOSPHATE DODECAHYDRATE

Dinatrii phosphas dodecahydricus

$\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$
[7782-75-6]

M_r 358.1

DEFINITION

Content: 98.0 per cent to 101.0 per cent (anhydrous substance).

CHARACTERS

Appearance: colourless, transparent crystals, very efflorescent.

Solubility: very soluble in water, practically insoluble in ethanol (96 per cent).

IDENTIFICATION

- A. Solution S (see Tests) is slightly alkaline (2.2.4).
 B. Water (see Tests).
 C. Solution S gives reaction (b) of phosphates (2.3.1).
 D. Solution S gives reaction (a) of sodium (2.3.1).

TESTS

Solution S. Dissolve 5.0 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 colourless (2.2.2, *Method II*).

Reducing substances. To 5 ml of solution S add 5 ml of *dilute sulphuric acid R* and 0.25 ml of 0.02 M *potassium permanganate* and heat on a water-bath for 5 min. The solution retains a slight red colour.

Monosodium phosphate: maximum 2.5 per cent.

From the volume of 1 M *hydrochloric acid* (25 ml) and of 1 M *sodium hydroxide* (n_1 ml and n_2 ml) used in the assay, calculate the following ratio:

$$\frac{n_2 - 25}{25 - n_1}$$

This ratio is not greater than 0.025.

Chlorides (2.4.4): maximum 200 ppm.

To 2.5 ml of solution S add 10 ml of *dilute nitric acid R* and dilute to 15 ml with *water R*.

Sulphates (2.4.13): maximum 500 ppm.

To 3 ml of solution S add 2 ml of *dilute hydrochloric acid R* and dilute to 15 ml with *distilled water R*.

Arsenic (2.4.2, *Method A*): maximum 2 ppm, determined on 5 ml of solution S.

Iron (2.4.9): maximum 20 ppm.

Dilute 5 ml of solution S to 10 ml with *water R*.

Heavy metals (2.4.8): maximum 10 ppm.

12 ml of solution S complies with test A. Prepare the reference solution using *lead standard solution* (1 ppm *Pb*) *R*.

Water (2.5.12): 57.0 per cent to 61.0 per cent, determined on 50.0 mg. Use a mixture of 10 volumes of *anhydrous methanol R* and 40 volumes of *formamide R* as solvent.