PENTASODIUM TRIPHOSPHATE

Prepared at the 55th JECFA (2000) and published in FNP 52 Add 8 (2000), superseding tentative specifications prepared at the 20th JECFA (1976) and published in FNS 1B (1977) and in FNP 52 (1992). No ADI was established, but a group MTDI of 70 mg/kg bw, expressed as phosphorus from all food sources, was established at the 26th JECFA (1982).

SYNONYMS Pentasodium tripolyphosphate, Sodium triphosphate, Sodium tripolyphosphate, Triphosphate; INS No. 451(i)

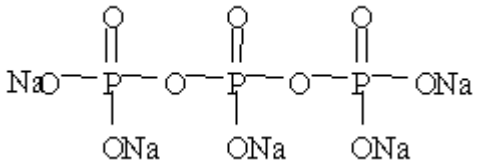
DEFINITION

Chemical names: Pentasodium triphosphate, pentasodium tripolyphosphate

C.A.S. number: 7758-29-4

Chemical formula: $Na_5O_{10}P_3 \cdot x\ H_2O$ (x = 0 or 6)

Structural formula:



Formula weight: Anhydrous: 367.86
Hexahydrate: 475.94

Assay: Anhydrous: not less than 85.0% of $Na_5O_{10}P_3$ and not less than 56.0% and not more than 58.0% of P_2O_5

Hexahydrate: not less than 65.0% of $Na_5O_{10}P_3$ and not less than 43.0% and not more than 45.0% of P_2O_5

DESCRIPTION White, slightly hygroscopic granules or powder

FUNCTIONAL USES Sequestrant, texturizer

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4): Freely soluble in water; insoluble in ethanol

pH (Vol. 4): 9.1 - 10.1 (1 % soln)

Test for phosphate (Vol. 4): Passes test

Test for sodium (Vol. 4): Passes test

PURITY

Loss on drying (Vol. 4): Anhydrous: not more than 0.7% (105°, 1 h)

Hexahydrate: not more than 23.5% (60 °, 1 h, followed by 105°, 4 h)

Water-insoluble matter (Vol. 4) — Not more than 0.1%

Higher polyphosphates — Not detectable
See description under TESTS

Fluoride (Vol. 4) — Not more than 50 mg/kg (Method I or III)

Arsenic (Vol. 4) — Not more than 3 mg/kg (Method II)

Lead (Vol. 4) — Not more than 4 mg/kg
Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, “Instrumental Methods”.

TESTS

PURITY TESTS

Higher polyphosphates

Chromatographic solvent
Mix 75 ml of isopropanol, 10 ml of water, 20 ml of 20% trichloroacetic acid and 0.3 ml of 20% ammonia. Make fresh every week.

Chromatographic spray
Dissolve 1 g of ammonium molybdate in 85 ml of water, 10 ml of N hydrochloric acid and 5 ml of 60% perchloric acid.

Sample solution
Dissolve 1 g of the sample in 50 ml of water.

Reference solution
Dissolve 1 g of a standard sample of pentasodium triphosphate in 50 ml of water.

Procedure
Place 0.01 ml of the sample solution and 0.01 ml of reference solution on the starting line of the chromatographic paper and allow to dry in a stream of warm air. Use ascending chromatography at 18-20° until the solvent has ascended about 25 cm from the starting line (12 - 15 h). Dry at 60° in an oven and spray with the chromatographic spray. Place the paper under an ultraviolet lamp and irradiate until the phosphates are visible as blue spots (about 2 min).

Three spots (one from the monophosphate (R_f = 0.69), a second from the diphosphate (R_f = 0.44) and the third from the triphosphate (R_f = 0.29) are observed, and no other spot is observed.

METHOD OF ASSAY

1. Determination of $Na_5O_{10}P_3$

Reagents and solutions
- Potassium acetate buffer (pH 5.0): Dissolve 78.5 g of potassium acetate

in 1000 ml of water and adjust the pH of the solution to 5.0 with acetic acid. Add a few mg of mercuric iodide to inhibit mould growth.
- 0.3 M Potassium chloride: Dissolve 22.35 g of potassium chloride in water, add 5 ml of potassium acetate buffer, dilute with water to 1000 ml, and mix. Add a few mg of mercuric iodide.
- 0.6 M Potassium chloride: Dissolve 44.7 g of potassium chloride in water, add 5 ml of potassium acetate buffer, dilute with water to 1000 ml, and mix. Add a few mg of mercuric iodide.
- 1 M Potassium chloride: Dissolve 74.5 g of potassium chloride in water, add 5 ml of potassium acetate buffer, dilute to 1000 ml with water, and mix. Add a few mg of mercuric iodide.

Chromatographic Column

Use a standard chromatographic column 20 to 40 cm length, 20 to 28 cm inside diameter, with a sealed-in, coarse porosity fritted disk. If a stopcock is not provided, attach a stopcock having a 3 to 4 mm, diameter bore to the outlet of the column with a short length of flexible vinyl tubing.

Procedure

Close the column stopcock, fill the space between the fritted disk and the stopcock with water, and connect a vacuum line to the stopcock. Prepare a 1:1 water slurry of Dowex F x 8, or equivalent, chloride form, 100-200 or 200-400 mesh, styrenedivinylbenzene ion exchange resin, and decant off any fine particles and any foam. Do this two or three times or until no more finely suspended material or foaming is observed. Fill the column with the slurry, and open the stopcock to allow the vacuum to pack the resin bed until the water level is slightly above the top of the resin, then immediately close the stopcock. Do not allow the liquid level to fall below the resin level at any time. Repeat this procedure until the packed resin column is 15 cm above the fritted disk. Place one circle of tightly fitting fibre filter paper on top of the resin bed, then place a perforated polyethylene disk on top of the paper. Alternatively, a loosely packed plug of glass wool may be placed on top of the bed. Close the top of the column with a rubber stopper in which a 7.6 cm length of capillary tubing (1.5 mm i.d., 7 mm o.d.) has been inserted through the centre, so that about 12 mm of the tubing extends through the bottom of the stopper. Connect the top of the capillary tubing to the stem of a 500-ml separator with flexible vinyl tubing, and clamp the separator to a ring stand above the column. Wash the column by adding 100 ml of water to the separator with all stopcocks closed. First open the separator stopcock, then open the column stopcock. The rate of flow should be about 5 ml per min. When the separator is empty, close the stopcock on the column then close the separator stopcock.

Transfer about 500 mg of the sample (a), accurately weighed, into a 250 ml volumetric flask, dissolve and dilute to volume with water, and mix. Transfer 10 ml of this solution into the separator, open both stopcocks and allow the solution to drain into the column, rinsing the separator with 20 ml of water. Discard the eluate. Add 370 ml of 0.3 M Potassium Chloride to the separator, and allow this solution to pass through the column, discarding the eluate. Add 250 ml of 0.6 M Potassium Chloride to the column, allow the solution to pass through the column, and receive the eluate in a 400-ml beaker. (To ensure a clean column for the next run, pass 100 ml of 1 M Potassium Chloride through the column, followed by 100 ml of water. Discard all washings.) To the beaker add 15 ml of nitric acid, mix, and boil

for 15 to 20 min. Add methyl orange TS, and neutralize the solution with stronger ammonia TS. Add 1 g of ammonium nitrate crystals, stir to dissolve, and cool. Add 15 ml of ammonium molybdate TS, with stirring, and stir vigorously for 3 min or allow to stand with occasional stirring for 10 to 15 min. Filter the contents of the beaker with suction through a 6-7 mm paper pulp filter pad supported in a 25 mm porcelain disk. The filter pad should be covered with a suspension of a filtering aid. After the contents of the beaker have been transferred to the filter, wash the beaker with five 10 ml portions of a 1% solution of sodium or potassium nitrate, passing the washings through the filter, then wash the filter with five 5-ml portions of the wash solution. Return the filter pad and the precipitate to the beaker, wash the funnel thoroughly with water into the beaker, and dilute to about 150 ml. Add 0.1 N sodium hydroxide from a buret until the yellow precipitate is dissolved, then add 5 to 8 ml in excess. Add phenolphthalein TS, and titrate the excess alkali with 0.1 N nitric acid. Finally, titrate with 0.1 N sodium hydroxide to the first appearance of the pink colour. The difference between the total volume of 0.1 N sodium hydroxide added and the volume of nitric acid required represents the volume, V, in ml, of 0.1 N sodium hydroxide consumed by the phosphomolybdate complex.

Calculate the $Na_5O_{10}P_3$ content of the sample in % by the formula

$$\%\,Na_5O_{10}P_3 = \frac{0.533 \times 25 \times V}{a} \times 100$$

where
a = the weight of the sample (mg)
2. Determination of P_2O_5

Accurately weigh about 20 g of the sample into a beaker. Add 150 ml water and 20 ml concentrated nitric acid. Introduce anti-bumping granules, cover the beaker with a watch glass and boil gently for 1 h. Cool to room temperature. Quantitatively transfer the solution to a 500-ml volumetric flask, dilute with water, mix well and dilute to the mark with water. Transfer 20.0 ml of the solution to a plastic beaker, dilute to about 50 ml with water and place the beaker in an automatic titrator equipped with a pH meter. Adjust the pH of the solution to between 2.5 and 2.8 with 5 mol/l sodium hydroxide. Titrate the solution with 0.5 mol/l sodium hydroxide. Record the consumed volumes at the inflection points at about pH 4 (V1) and about pH 9 (V2).

Calculate the P_2O_5 content of the sample in % by the formula

% P_2O_5 = [(V2 - V1)/2000] x f x 70.97 x (500/20) x (100/w)
= [(V2 - V1)/w] x f x 88.71

where
w = weight of the sample (g)
f = factor of 0.5 mol/l sodium hydroxide (= actual molarity/0.5)