TRIMAGNESIUM PHOSPHATE

Prepared at the 17th JECFA (1973), published in FNP 4 (1978) and in FNP 52 (1992). Metals and arsenic specifications revised at the 57th JECFA (2001). A group MTDI of 70 mg/kg bw, as phosphorus from all food sources, was established at the 26th JECFA (1982).

SYNONYMS Magnesium phosphate, tribasic, tertiary magnesium phosphate; INS No. 343(iii)

DEFINITION May contain 4, 5 or 8 molecules of water of hydration. The article of commerce can be specified further as to titration value.

Chemical names Trimagnesium orthophosphate

C.A.S. number 7757-87-1

Chemical formula $Mg_3(PO_4)_2$ (various hydrates)

Formula weight 262.86 (anhydrous)

Assay Not less than 98% of $Mg_3(PO_4)_2$ after ignition at 425^o

DESCRIPTION White, odourless crystalline powder

FUNCTIONAL USES Anticaking agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Practically insoluble in water; insoluble in ethanol, soluble in dilute mineral acids

Test for phosphate To a warm solution of the sample in a slight excess of nitric acid add ammonium molybdate TS. A yellow precipitate of ammonium phosphomolybdate forms which is soluble in ammonia TS.

Test for magnesium Dissolve about 100 mg of the sample in 0.7 ml of dilute acetic acid TS and 20 ml of water. Add 1 ml of ferric chloride TS, let stand for 5 min., and filter. Add ammonium chloride TS and ammonium carbonate TS. No precipitated is formed Add sodium phosphate TS. A white crystalline precipitate is formed which is insoluble in ammonia TS.

PURITY

Loss on ignition (Vol. 4) Tetrahydrate: Between 15% and 23% (425^o to constant weight)
Pentahydrate: Between 20% and 27% (425^o to constant weight)
Octahydrate: Between 30% and 37% (425^o to constant weight)

Fluoride (Vol. 4)

Not more than 5 mg/kg
See description under TESTS

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

Fluoride (Vol. 4)

Weigh 5 g of the sample to the nearest mg, and transfer into a 200-ml distilling flask connected with a condenser and carrying a thermometer and a dropping funnel equipped with a stopcock. Dissolve in 25 ml of a 1 in 2 sulfuric acid solution, add 6 glass beads, and connect the apparatus for distillation, using a 600-ml beaker to collect the distillate. Add 40 ml of the diluted sulfuric acid to the flask through the dropping funnel, then fill the funnel with water, heat the solution to boiling, and continue heating until the temperature reaches 165°. Adjust the stopcock of the dropping funnel so that the temperature is maintained at 165±5°, and continue the distillation until about 300 ml has been collected. Rinse the condenser and condenser arm with water, collecting the rinsings in the beaker. Add sodium hydroxide TS to the distillate to make it alkaline to litmus paper, and then add 5 ml in excess. Add 5 ml of 30% hydrogen peroxide and 6 glass beads to the beaker, boil until a volume of about 30 ml is reached, and cool. Transfer the condensed distillate, including the glass beads, into a 125 ml distilling flask connected with a condenser and carrying a thermometer and a capillary tube, both of which must extend into the liquid. Add 30 ml of perchloric acid and continue as directed under the Fluoride Limit Test (Method I Thorium Nitrate Colorimetric method), beginning with "Connect a small dropping funnel or a steam generator to the capillary tube."

METHOD OF ASSAY

Weigh to the nearest 0.1 mg, 200 mg of the ignited sample. Dissolve in a mixture of 25 ml of water and 10 ml of dilute nitric acid TS. Filter, if necessary, wash any precipitate, then dissolve the precipitate by the addition of 1 ml of dilute nitric acid TS. Adjust the temperature to about 50°, add 75 ml of ammonium molybdate TS, and maintain the temperature at about 50° for 30 min., stirring occasionally. Wash the precipitate once or twice with water by decantation, using from 30 to 40 ml each time. Transfer the precipitate to a filter, and wash with a 1 in 100 potassium nitrate solution until the last washing is not acid to litmus paper. Transfer the precipitate and filter to the precipitation vessel, add 40.0 ml of N sodium hydroxide, agitate until the precipitate is dissolved, add 3 drops of phenolphthalein TS and then titrate the excess alkali with N sulfuric acid. Each ml of N sodium hydroxide corresponds to 5.715 mg of $Mg_3(PO_4)_2$.