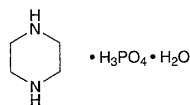


Piperazine Phosphate

リン酸ピペラジン

C₄H₁₀N₂·H₃PO₄·H₂O: 202.15

Piperazine monophosphate monohydrate [18534-18-4]

Piperazine Phosphate contains not less than 98.5% of C₄H₁₀N₂·H₃PO₄ (mol. wt.: 184.13), calculated on the anhydrous basis.

Description Piperazine Phosphate occurs as white crystals or crystalline powder. It is odorless, and has a slightly acid taste.

It is soluble in formic acid, sparingly soluble in water, very slightly soluble in acetic acid (100), and practically insoluble in methanol, in ethanol (95) and in diethyl ether.

It dissolves in dilute hydrochloric acid.

Melting point: about 222°C (with decomposition).

Identification (1) To 3 mL of a solution of Piperazine Phosphate (1 in 100) add 3 drops of Reinecke salt TS: a light red precipitate is formed.

(2) Determine the infrared absorption spectrum of Piperazine Phosphate as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

(3) A solution of Piperazine Phosphate (1 in 100) responds to the Qualitative Tests (1) and (3) for phosphate.

pH Dissolve 1.0 g of Piperazine Phosphate in 100 mL of water: the pH of the solution is between 6.0 and 6.5.

Purity (1) Chloride—To 0.5 g of Piperazine Phosphate add 6 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution, and perform the test. Prepare the control solution with 0.25 mL of 0.01 mol/L hydrochloric acid (not more than 0.018%).

(2) Heavy metals—To 2.0 g of Piperazine Phosphate add 5 mL of dilute hydrochloric acid, 30 mL of water and 2 mL of dilute acetic acid, and dissolve. Add sodium hydroxide TS, adjust the pH of the solution to 3.3, and add water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

(3) Arsenic—Dissolve 2.0 g of Piperazine Phosphate in 5 mL of dilute hydrochloric acid, and use this solution as the test solution. Perform the test using Apparatus B (not more than 1 ppm).

(4) Related substances—Dissolve 0.050 g of Piperazine Phosphate in 10 mL of water, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add water to make exactly 100 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5 μL each of the sample solution and the standard solution on a plate of cellulose for thin-layer chromatography. Develop

the plate with a mixture of ethyl acetate, acetone, ammonia solution (28) and ethanol (99.5) (8:3:3:2) to a distance of about 13 cm, and air-dry the plate. Spray evenly 4-dimethylaminocinnamaldehyde TS, and allow to stand for 15 minutes: the spots other than the principal spot and the spot on the starting line from the sample solution are not more intense than the spot from the standard solution.

Water 8.0–9.5% (0.3 g, direct titration).

Assay Weigh accurately about 0.15 g of Piperazine Phosphate, dissolve in 10 mL of formic acid, add 60 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 9.207 mg of C₄H₁₀N₂·H₃PO₄

Containers and storage Containers—Well-closed containers.

Piperazine Phosphate Tablets

リン酸ピペラジン錠

Piperazine Phosphate Tablets contain not less than 95% and not more than 105% of the labeled amount of piperazine phosphate (C₄H₁₀N₂·H₃PO₄·H₂O: 202.15).

Method of preparation Prepare as directed under the Tablets, with Piperazine Phosphate.

Identification Take a quantity of Piperazine Phosphate Tablets equivalent to 0.1 g of Piperazine Phosphate according to the labeled amount, previously powdered, add 10 mL of water, shake while warming for 10 minutes, allow to cool, and filter. To 3 mL of the filtrate add 3 drops of Reinecke salt TS: a light red precipitate is formed.

Disintegration test When perform the test for 10 minutes, Piperazine Phosphate Tablets meet the requirements of the Disintegration test.

Assay Weigh accurately not less than 20 Piperazine Phosphate Tablets, and powder. Weigh accurately a quantity of the powder, equivalent to about 0.15 g of piperazine phosphate (C₄H₁₀N₂·H₃PO₄·H₂O) according to the labeled amount. Add 5 mL of formic acid, shake for 5 minutes, centrifuge, and collect the supernatant liquid. To the residue add 5 mL of formic acid, shake for 5 minutes, centrifuge, and collect the supernatant liquid. Repeat twice the same procedure with 5 mL each of acetic acid (100), combine all the supernatant liquids, add 50 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 10.107 mg of C₄H₁₀N₂·H₃PO₄·H₂O

Containers and storage Containers—Tight containers.