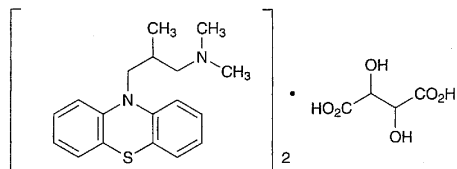


Alimemazine Tartrate

酒石酸アリメマジン



(C₁₈H₂₂N₂S)₂·C₄H₆O₆: 746.98

N,N-Dimethyl-*N*-[2-methyl-3-(phenothiazin-10-yl)propyl]amine hemitartrate [41375-66-0]

Alimemazine Tartrate, when dried, contains not less than 98.0% of (C₁₈H₂₂N₂S)₂·C₄H₆O₆.

Description Alimemazine Tartrate occurs as a white powder. It is odorless, and has a bitter taste.

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

The pH of a solution of Alimemazine Tartrate (1 in 50) is between 5.0 and 6.5.

It is gradually colored by light.

Identification (1) To 2 mL of a solution of Alimemazine Tartrate (1 in 100) add 1 drop of iron (III) chloride TS: a red-brown color is produced, and immediately a yellow precipitate is formed.

(2) Dissolve 1 g of Alimemazine Tartrate in 5 mL of water, add 3 mL of sodium hydroxide TS, extract with two 10-mL portions of diethyl ether [use the aqueous layer obtained in the Identification (4)]. Shake the combined diethyl ether extracts with 3 g of anhydrous sodium sulfate, filter, and evaporate the diethyl ether with the aid of a current of air. Dry the residue in a desiccator (in vacuum, phosphorus (V) oxide) for 16 hours: it melts between 66°C and 70°C.

(3) Determine the absorption spectrum of a solution of Alimemazine Tartrate (1 in 100,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelengths.

(4) The aqueous layer, obtained in the identification (2), when neutralized with dilute acetic acid, responds to the Qualitative Tests (1) and (2) for tartrate.

Melting point 159 – 163°C

Purity (1) Clarity and color of solution—Dissolve 1.0 g of Alimemazine Tartrate in 20 mL of water: the solution is clear and colorless.

(2) Heavy metals—Proceed with 1.0 g of Alimemazine Tartrate according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(3) Arsenic—Prepare the test solution with 1.0 g of Alimemazine Tartrate according to Method 3, and perform the test using Apparatus B. Use a solution of magnesium nitrate hexahydrate in ethanol (95) (1 in 5) (not more than 2 ppm).

Loss on drying Not more than 0.5% (1 g, 105°C, 3 hours).

Residue on ignition Not more than 0.10% (1 g).

Assay Weigh accurately about 0.8 g of Alimemazine Tartrate, previously dried, dissolve in 50 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS until the color of the solution changes from red through brown to green-brown (indicator: 2 mL of *p*-naphtholbenzein TS). Perform a blank determination, and make any necessary correction.

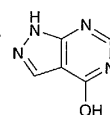
Each mL of 0.1 mol/L perchloric acid VS
= 37.350 mg of (C₁₈H₂₂N₂S)₂·C₄H₆O₆

Containers and storage Containers—Tight containers.

Storage—Light-resistant.

Allopurinol

アロプリノール



C₅H₄N₄O: 136.11

1*H*-Pyrazolo[3,4-*d*]pyrimidin-4-ol [315-30-0]

Allopurinol, when dried, contains not less than 98.0% of C₅H₄N₄O.

Description Allopurinol occurs as white to pale yellowish white crystals or crystalline powder. It is odorless.

It is slightly soluble in *N,N*-dimethylformamide, very slightly soluble in water, and practically insoluble in ethanol (95) and in diethyl ether.

It dissolves in sodium hydroxide TS and in ammonia TS.

Melting point: not lower than 320°C (with decomposition).

Identification (1) Dissolve 0.1 g of Allopurinol in 50 mL of water by warming. To 5 mL of this solution add 1 mL of ammonia TS and 1 mL of silver nitrate TS: a white precipitate is produced.

(2) Dissolve 0.1 g of Allopurinol in 50 mL of water by warming. To 5 mL of this solution add 0.5 mL of copper (II) sulfate TS: a blue precipitate is produced.

(3) Determine the absorption spectrum of a solution of Allopurinol (1 in 200,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelengths.

Purity (1) Clarity and color of solution—Dissolve 0.5 g of Allopurinol in 10 mL of sodium hydroxide TS: the solution is clear, and has no more color than Matching Fluid D.

(2) Sulfate—To 2.0 g of Allopurinol add 100 mL of water, and boil for 5 minutes. Cool, add water to make 100 mL, and filter. To 25 mL of the filtrate add 1 mL of dilute hydrochloric acid and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid VS (not more than 0.038%).

(3) Heavy metals—Proceed with 1.0 g of Allopurinol according to Method 2, and perform the test. Prepare the con-

trol solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(4) Arsenic—Prepare the test solution with 1.0 g of Allopurinol according to Method 3, and perform the test using apparatus B (not more than 2 ppm).

(5) Related substances—Dissolve 0.05 g of Allopurinol in 10 mL of ammonia TS, and use this solution as the sample solution. Pipet 1 mL of this solution, add ammonia TS to make exactly 200 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5 μ L each of the sample solution and the standard solution on a plate of cellulose with fluorescent indicator for thin-layer chromatography. Develop the plate with ammonia TS-saturated 1-butanol to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

Loss on drying Not more than 0.5% (1 g, 105°C, 4 hours).

Residue on ignition Not more than 0.10% (1 g).

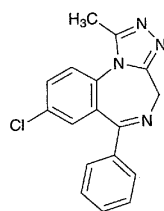
Assay Weigh accurately about 0.16 g of Allopurinol, previously dried, dissolve in 70 mL of *N,N*-dimethylformamide by warming. Cool, and titrate with 0.1 mol/L tetramethylammonium hydroxide VS (potentiometric titration). To 70 mL of *N,N*-dimethylformamide add 12 mL of water, perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L tetramethylammonium hydroxide VS
= 13.611 mg of C₅H₄N₄O

Containers and storage Containers—Tight containers.

Alprazolam

アルプラゾラム



C₁₇H₁₃ClN₄: 308.76
8-Chloro-1-methyl-6-phenyl-4*H*-[1,2,4]triazolo[4,3-*a*][1,4]benzodiazepine [28981-97-7]

Alprazolam, when dried, contains not less than 98.5% of C₁₇H₁₃ClN₄.

Description Alprazolam occurs as white crystals or crystalline powder.

It is freely soluble in chloroform, soluble in methanol and in ethanol (95), sparingly soluble in acetic anhydride, and practically insoluble in water.

It dissolves in dilute nitric acid.

Identification (1) Determine the absorption spectrum of a solution of Alprazolam in ethanol (95) (1 in 200,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelength.

(2) Dissolve 0.05 g of Alprazolam in 0.7 mL of deuteriochloroform for nuclear magnetic resonance spectroscopy, and determine the spectrum of this solution using tetramethylsilane for nuclear magnetic resonance spectroscopy as an internal reference compound, as directed under the Nuclear Magnetic Resonance Spectroscopy (¹H): it exhibits a single signal A at around δ 2.6 ppm, doublet signals B and C at around δ 4.0 ppm and δ 5.4 ppm, and a broad signal D between δ 7.1 ppm and 7.9 ppm. The ratio of integrated intensity of each signal, A:B:C:D, is about 3:1:1:8.

(3) Perform the test with Alprazolam as directed under the Flame Coloration Test (2): a green color appears.

Melting point 228 – 232°C

Purity (1) Chloride—Dissolve 0.5 g of Alprazolam in 10 mL of dilute nitric acid, and add water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 0.20 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.014%).

(2) Heavy metals—Proceed with 2.0 g of Alprazolam according to Method 4, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

(3) Related substances—Dissolve 0.050 g of Alprazolam in 10 mL of methanol, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add methanol to make exactly 100 mL, then pipet 1 mL of this solution, add methanol to make exactly 10 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 20 μ L each of the sample solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop with a mixture of acetone, hexane, ethyl acetate and ethanol (95) (4:2:2:1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

Loss on drying Not more than 0.5% (1 g, reduced pressure not exceeding 0.67 kPa, 60°C, 4 hours).

Residue on ignition Not more than 0.10% (1 g).

Assay Weigh accurately about 0.25 g of Alprazolam, previously dried, dissolve in 100 mL of acetic anhydride, and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination in the same manner, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 15.438 mg of C₁₇H₁₃ClN₄

Containers and storage Containers—Well-closed containers.