low.

(2) To 1 mL of a solution of Pyridoxine Hydrochloride (1 in 10,000) add 2 mL of a freshly prepared solution of 2,6-dibromo-*N*-chloro-1,4-benzoquinone monoimine in ethanol (95) (1 in 4000) and 1 drop of ammonia TS: a blue color develops. To 1 mL of a solution of Pyridoxine Hydrochloride (1 in 10,000) add 1 mL of a saturated boric acid solution, and proceed as directed in the same manner: no blue color develops.

(3) Add 1 mL of water to 0.5 g of Pyridoxine Hydrochloride, warm to dissolve, cool, add 6 mL of 2,4,6-trinitrophenol TS, and allow to stand for 2 to 3 hours. Filter the crystals, wash with a small amount of ice-water, and dry at 105°C for 2 hours. The crystals so obtained melt between 156°C and 159°C (with decomposition).

(4) A solution of Pyridoxine Hydrochloride (1 in 10) responds to the Qualitative Tests for chloride.

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

(2) Heavy metals—Proceed with 1.0 g of Pyridoxine Hydrochloride according to Method 1, and perform the test. Prepare the control solution with 3.0 mL of Standard Lead Solution (not more than 30 ppm).

Loss on drying Not more than 0.30% (1 g, in vacuum, silica gel, 4 hours).

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

Assay Weigh accurately about 0.2 g of Pyridoxine Hydrochloride, previously dried, add 5 mL of acetic acid (100) and 5 mL of acetic anhydride, dissolve by gentle boiling, cool, add 30 mL of acetic anhydride, 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
= 20.564 mg of $C_8H_{11}NO_3.HCl$

Containers and storage Containers—Tight containers.
Storage—Light-resistant.

Pyridoxine Hydrochloride Injection

Vitamin B_6 Injection

塩酸ピリドキシン注射液

Pyridoxine Hydrochloride Injection is an aqueous solution for injection. It contains not less than 95% and not more than 115% of the labeled amount of pyridoxine hydrochloride ($C_8H_{11}NO_3.HCl$: 205.64).

Method of preparation Prepare as directed under Injections, with Pyridoxine Hydrochloride.

Description Pyridoxine Hydrochloride Injection is a colorless or pale yellow, clear liquid.
It is gradually affected by light.
pH: 3.0 - 6.0

Identification (1) To a volume of Pyridoxine Hydrochloride Injection, equivalent to 0.01 g of Pyridoxine Hydrochloride according to the labeled amount, add water to make 10 mL, and use this solution as the sample solution. Proceed with 1 mL of the sample solution as directed in the Identification (1) under Pyridoxine Hydrochloride.

(2) Dilute 1 mL of the sample solution obtained in (1) with water to make 10 mL. Proceed with 1 mL of this solution as directed in the Identification (2) under Pyridoxine Hydrochloride.

(3) Add 0.5 mL of phosphotungstic acid TS to 1 mL of the sample solution obtained in (1): a white turbidity is produced.

Assay Measure exactly a volume of Pyridoxine Hydrochloride Injection, equivalent to about 0.02 g of pyridoxine hydrochloride ($C_8H_{11}NO_3.HCl$), dilute with water, if necessary, and add water to make exactly 100 mL. Pipet 25 mL of this solution, add water to make exactly 200 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.1 g of Pyridoxine Hydrochloride Reference Standard, previously dried in a desiccator (in vacuum, silica gel) for 4 hours, and dissolve in water to make exactly 100 mL. Pipet 5 mL of this solution, add water to make exactly 200 mL, and use this solution as the standard solution. Pipet 1 mL each of the sample solution and the standard solution, add 2.0 mL of barbital buffer solution, 9.0 mL of 2-propanol and 2.0 mL of a freshly prepared solution of 2,6-dibromo-*N*-chloro-1,4-benzoquinone monoimine in ethanol (95) (1 in 4000), shake well, add 2-propanol to make exactly 25 mL, and allow to stand for 90 minutes. Determine the absorbances, A_T and A_S, of the subsequent sample solution and the subsequent standard solution, respectively, at 650 nm as directed under the Ultraviolet-visible Spectrophotometry, using a solution, prepared in the same manner with 1 mL of water, as the blank.

Amount (mg) of pyridoxine hydrochloride ($C_8N_{11}NO_3.HCl$)
= amount (mg) of Pyridoxine Hydrochloride Reference Standard
$\times \frac{A_T}{A_S} \times \frac{1}{5}$

Containers and storage Containers—Hermetic containers, and colored containers may be used.
Storage—Light-resistant.

Quinidine Sulfate

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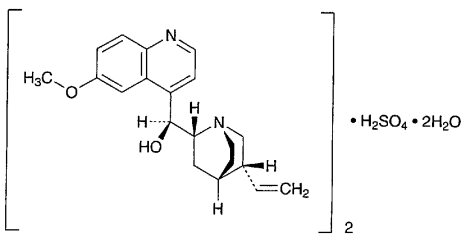

$(C_{20}H_{24}N_2O_2)_2.H_2SO_4.2H_2O$: 782.94
(8*R*,9*S*)-6′-Methoxycinchonan-9-ol hemisulfate monohydrate [*6591-63-5*]

Quinidine Sulfate, when dried, contains not less than 98.5% of $(C_{20}H_{24}N_2O_2)_2.H_2SO_4$: 746.91.

Description Quinidine Sulfate occurs as white crystals. It is odorless, and has a very bitter taste.

It is freely soluble in ethanol (95) and in boiling water, sparingly soluble in water, and practically insoluble in diethyl ether. Quinidine Sulfate, previously dried, is freely soluble in chloroform.

It darkens gradually by light.

Specific rotation $[\alpha]_D^{20}$: +275 – +287° (after drying, 0.5 g, 0.1 mol/L hydrochloric acid VS, 25 mL, 100 mm).

Identification (1) Dissolve 0.01 g of Quinidine Sulfate in 10 mL of water and 2 to 3 drops of dilute sulfuric acid: a blue fluorescence is produced.

(2) To 5 mL of an aqueous solution of Quinidine Sulfate (1 in 1000) add 1 to 2 drops of bromine TS, then add 1 mL of ammonia TS: a green color develops.

(3) To 5 mL of an aqueous solution of Quinidine Sulfate (1 in 100) add 1 mL of silver nitrate TS, stir with a glass rod, and allow to stand for a short interval: a white precipitate is produced, and it dissolves on addition of nitric acid.

(4) Dissolve 0.4 g of Quinidine Sulfate in 20 mL of water and 1 mL of dilute hydrochloric acid: the solution responds to the Qualitative Tests for sulfate.

pH Dissolve 1.0 g of Quinidine Sulfate in 100 mL of freshly boiled and cooled water: the pH of this solution is between 6.0 and 7.0.

Purity (1) Chloroform-ethanol-insoluble substances—Warm 2.0 g of Quinidine Sulfate with 15 mL of a mixture of chloroform and ethanol (99.5) (2:1) at about 50°C for 10 minutes. After cooling, filter through a tared glass filter (G4) by gentle suction. Wash the residue with five 10-mL portions of a mixture of chloroform and ethanol (99.5) (2:1), and dry at 105°C for 1 hour: the mass of the residue is not more than 2.0 mg.

(2) Related substances—Dissolve 0.020 g of Quinidine Sulfate in the mobile phase to make exactly 100 mL, and use this solution as the sample solution. Separately, dissolve 0.025 g of cinchonine in the mobile phase to make exactly 100 mL. Pipet 2 mL of this solution, add the mobile phase to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 50 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Determine each peak area of the sample solution by the automatic integration method, and calculate their amount by the area percentage method: the amount of hydroquinidine sulfate is not more than 15.0%, and those of quinine sulfate and dihydroquinine sulfate are not more than 1.0%. The total area of the peaks other than the principal peak and the above peaks is not larger than the peak area of cinchonine from the standard solution.

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 235 nm).

Column: A column about 4 mm in inside diameter and about 25 cm in length, packed with octadecylsilanized silica gel (10 μm in particle diameter).

Temperature: Room temperature.

Mobile phase: A mixture of water, acetonitrile, methanesulfonic acid TS and a solution of diethylamine (1 in 10) (43:5:1:1).

Flow rate: Adjust the flow rate so that the retention time of quinidine is about 10 minutes.

Selection of column: Dissolve 0.01 g each of Quinidine Sulfate and quinine sulfate in 5 mL of methanol, and add the mobile phase to make 50 mL. Proceed with 50 μL of this solution under the above operating conditions, and calculate the resolution. Use a column giving elution of quinidine, quinine, dihydroquinidine and dihydroquinine in this order with a resolution between quinidine and quinine and that between quinine and dihydroquinidine being not less than 1.2, respectively.

Detection sensitivity: Adjust the detection sensitivity so that the peak height of cinchonine obtained from 50 μL of the standard solution is between 5 mm and 10 mm.

Time span of measurement: About twice as long as the retention time of quinidine after the solvent peak.

(3) Readily carbonizable substances—Take 0.20 g of Quinidine Sulfate and perform the test: the solution has no more color than Matching fluid M.

Loss on drying Not more than 5.0% (1 g, 130 °C, 3 hours).

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

Assay Weigh accurately about 0.5 g of Quinidine Sulfate, previously dried, dissolve in 20 mL of acetic acid (100), and add 80 mL of acetic anhydride, and titrate ith 0.1 mol/L perchloric acid VS until the color of the solution changes from purple through blue to blue-green (indicator: 3 drops of crystal violet TS). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 24.898 mg of $(C_{20}H_{24}N_2O_2)_2.H_2SO_4$

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Quinine Ethyl Carbonate

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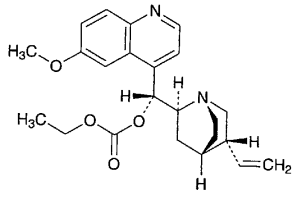

$C_{23}H_{28}N_2O_4$: 396.48
Ethyl (8*S*,9*R*)-6′-methoxycinchonan-9-yl carbonate [*83-75-0*]

Quinine Ethyl Carbonate contains not less than