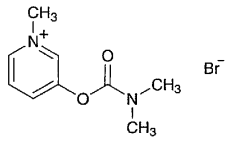
nation, and make any necessary correction.

Each mL of 0.05 mol/L sulfuric acid VS
= 12.311 mg of $C_5H_5N_3O$

Containers and storage Containers—Well-closed containers.

Pyridostigmine Bromide

臭化ピリドスチグミン



$C_9H_{13}BrN_2O_2$: 261.12
3-Dimethylcarbamoyloxy-1-methyl-pyridinium bromide
[101-26-8]

Pyridostigmine Bromide, when dried, contains not less than 98.5% of $C_9H_{13}BrN_2O_2$.

Description Pyridostigmine Bromide occurs as a white, crystalline powder. It is odorless or has a slightly characteristic odor.

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

The pH of a solution of Pyridostigmine Bromide (1 in 10) is between 4.0 and 6.0.

It is deliquescent.

Identification **(1)** Dissolve 0.02 g of Pyridostigmine Bromide in 10 mL of water, add 5 mL of Reinecke salt TS: a light red precipitate is produced.

(2) To 0.1 g of Pyridostigmine Bromide add 0.6 mL of sodium hydroxide TS: the unpleasant odor of dimethylamine is perceptible.

(3) Determine the absorption spectrum of a solution of Pyridostigmine Bromide in 0.1 mol/L hydrochloric acid TS (1 in 30,000) 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) A solution of Pyridostigmine Bromide (1 in 50) responds to the Qualitative Tests for Bromide.

Melting point 153 - 157°C

Purity **(1)** Clarity and color of solution—Dissolve 1.0 g of Pyridostigmine Bromide in 10 mL of water: the solution is clear and colorless.

(2) Heavy metals—Proceed with 1.0 g of Pyridostigmine Bromide according to Method 1, 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 Pyridostigmine Bromide according to Method 1, and perform the test using Apparatus B (not more than 2 ppm).

(4) Related substances—Dissolve 0.10 g of Pyridostigmine Bromide in 10 mL of ethanol (95), and use this solution as the sample solution. Pipet 2 mL of the sample solution, and add ethanol (95) to make exactly 10 mL. Pipet 1 mL of this solution, add ethanol (95) to make exactly 25 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μL each of the sample solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of methanol, chloroform and ammonium chloride TS (5:4:1) to a distance of about 12 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 in color.

Loss on drying Not more than 2.0% (1 g, in vacuum, phosphorus (V) oxide, 100°C, 5 hours).

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

Assay Weigh accurately about 0.3 g of Pyridostigmine Bromide, previously dried, dissolve in 10 mL of acetic acid (100), add 40 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.

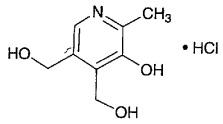
Each mL of 0.1 mol/L perchloric acid VS
= 26.112 mg of $C_9H_{13}BrN_2O_2$

Containers and storage Containers—Hermetic containers.

Pyridoxine Hydrochloride

Vitamin B_6

塩酸ピリドキシン



$C_8H_{11}NO_3$.HCl: 205.64
5-Hydroxy-6-methylpyridine-3,4-dimethanol monohydrochloride *[58-56-0]*

Pyridoxine Hydrochloride, when dried, contains not less than 98.0% of $C_8H_1NO_3$.HCl.

Description Pyridoxine Hydrochloride occurs as a white to pale yellow, crystalline powder. It is odorless, and has a bitter, acid taste.

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

The pH of a solution of Pyridoxine Hydrochloride solution (1 in 50) is between 2.5 and 3.5.

It is gradually affected by light.

Melting point: about 206°C (with decomposition).

Identification **(1)** To 1 mL of a solution of Pyridoxine Hydrochloride (1 in 1000) add 1 drop of iron (III) chloride TS: an orange-brown color is produced. Then add 1 drop of hydrochloric acid to the solution: the color changes to yel-

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

硫酸キニジン