heat on a water bath for 5 minutes with shaking. After cooling, add water to make 100 mL, and filter. To 20 mL of the filtrate add water to make 50 mL. Proceed the test using this solution as the test solution. Prepare the control solution with 0.45 mL of 0.005 mol/L sulfuric acid VS (not more than 0.144%).

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

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

(5) Related substances—The procedure should be performed under protection from direct sunlight in light-resistant vessels. Dissolve 0.10 g of Pyrantel Pamoate in 10 mL of *N,N*-dimethylformamide, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add *N,N*-dimethylformamide 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 silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, water and acetic acid (100) (3:1:1) 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 spot of pyrantel and the spot of pamoic acid from the sample solution are not more intense than the spot of pyrantel (*Rf* value: about 0.3) from the standard solution.

Loss on drying Not more than 1.0% (1 g, 105°C, 2 hours).

Residue on ignition Not more than 0.30% (1 g).

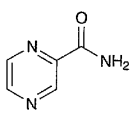
Assay Weigh accurately about 0.5 g of Pyrantel Pamoate, previously dried, add 25 mL of chloroform and 25 mL of sodium hydroxide TS, shake for 15 minutes, and extract. Extract further with two 25-mL portions of chloroform. Filter each extract through 5 g of anhydrous sodium sulfate on a pledget of absorbent cotton. Combine the chloroform extracts, add 30 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS (indicator: 2 drops of crystal violet TS). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 59.47 mg of $C_{11}H_{14}N_2S.C_{23}H_{16}O_6$

Containers and storage Containers—Tight containers.

Pyrazinamide

ピラジナミド



$C_5H_5N_3O$: 123.11
Pyrazine-2-carboxamide [*98-96-4*]

Pyrazinamide, when dried, contains not less than 99.0% of $C_5H_5N_3O$.

Description Pyrazinamide occurs as white crystals or crystalline powder. It is odorless, and has a slightly bitter taste.

It is sparingly soluble in water, slightly soluble in ethanol (95), and very slightly soluble in diethyl ether.

The pH of a solution of Pyrazinamide (1 in 100) is between 5.0 and 7.0.

Identification (1) Dissolve 0.1 g of Pyrazinamide in 10 mL of water, and add 1 mL of iron (II) sulfate TS: an orange-red color develops, and it changes to blue on the addition of 1 mL of sodium hydroxide TS.

(2) Boil gently 0.5 g of Pyrazinamide with 5 mL of sodium hydroxide TS: the gas evolved changes moistened red litmus paper to blue.

(3) Determine the absorption spectrum of a solution of Pyrazinamide in 0.1 mol/L hydrochloric acid TS (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.

Melting point 188 - 193°C

Purity (1) Sulfate—Perform the test with 0.6 g of Pyrazinamide. Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid VS (not more than 0.032%).

(2) Heavy metals—Proceed with 1.0 g of Pyrazinamide 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 Pyrazinamide according to Method 3, and perform the test using Apparatus B (not more than 2 ppm).

(4) Readily carbonizable substances—Perform the test with 0.20 g of Pyrazinamide: the solution has no more color than Matching Fluid A.

(5) Monocarboxylic acid and dicarboxylic acid—Dissolve 0.10 g of Pyrazinamide in 10 mL of water by warming, and add 0.5 mL of 0.05 mol/L potassium iodate VS and 0.5 g of potassium iodide. Then add 1 mL of chloroform, and shake vigorously: the chloroform layer has no more color than the following control solution.

Control solution: Proceed as directed above, but without Pyrazinamide.

Loss on drying Not more than 0.5% (1 g, in vacuum, silica gel, 4 hours).

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

Assay Weigh accurately about 0.3 g of Pyrazinamide, previously dried, in a 500 mL Kjeldahl flask, add 200 mL of water and 50 mL of a solution of sodium hydroxide (2 in 5), and connect the flask to a distillation apparatus having a spray trap. Dip the lower end of the condenser into 40 mL of a solution of boric acid (1 in 25) contained in an absorption flask. Boil gently for 20 minutes, then boil strongly, and continue the distillation until the distillate measures 200 mL. Cool the Kjeldahl flask, add 75 mL of water, repeat the distillation, and receive 70 mL of the distillate into the same absorption flask. Rinse the lower end of the condenser with a small quantity of water, combine the rinsings with the distillate, and titrate with 0.05 mol/L sulfuric acid VS until the color of the solution changes from green through pale grayish blue to light red-purple (indicator: 3 drops of bromocresol green-methyl red TS). Perform a blank determi-

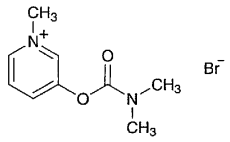
nation, and make any necessary correction.

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.

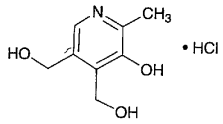
Each mL of 0.1 mol/L perchloric acid VS
= 26.112 mg of $C_9H_{13}BrN_2O_2$

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-