

tromethorphan Hydrobromide 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) To 50 mL of a solution of Dextromethorphan Hydrobromide (1 in 100) add 2 drops of phenolphthalein TS and sodium hydroxide TS until a red color develops. Add 50 mL of chloroform, shake, and add 5 mL of dilute nitric acid to 40 mL of the water layer. This solution responds to the Qualitative Tests for bromide.

Optical rotation $[\alpha]_D^{20}$: +26 – +30° (0.34 g, calculated on the anhydrous basis, water, 20 mL, 100 mm).

pH Dissolve 1.0 g of Dextromethorphan Hydrobromide in 100 mL of water: the pH of this solution is between 5.2 and 6.5.

Purity (1) Clarity and color of solution—Dissolve 0.20 g of Dextromethorphan Hydrobromide in 20 mL of water: the solution is clear and colorless.

(2) *N,N*-dimethylaniline—To 0.50 g of Dextromethorphan Hydrobromide add 20 mL of water, and dissolve by heating on a water bath. After cooling, add 2 mL of dilute acetic acid, 1 mL of sodium nitrite TS and water to make 25 mL: the solution has no more color than the following control solution.

Control solution: Dissolve 0.10 g of *N,N*-dimethylaniline in 400 mL of water by warming on a water bath, cool, and add water to make 500 mL. Pipet 5 mL of this solution, and add water to make 200 mL. To 1.0 mL of this solution add 2 mL of dilute acetic acid, 1 mL of sodium nitrite TS and water to make 25 mL.

(3) Heavy metals—Proceed with 1.0 g of Dextromethorphan Hydrobromide according to Method 4, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(4) Phenolic compounds—Dissolve 5 mg of Dextromethorphan Hydrobromide in 1 drop of dilute hydrochloric acid and 1 mL of water, add 2 drops of iron (III) chloride TS and 2 drops of potassium hexacyanoferrate (III) TS, shake, and allow to stand for 15 minutes: no blue-green color develops.

(5) Related substances—Dissolve 0.25 g of Dextromethorphan Hydrobromide 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 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 silica gel for thin-layer chromatography. Develop the plate with a mixture of toluene, ethyl acetate, methanol, dichloromethane and 13.5 mol/L ammonia TS (55:20:13:10:2) to a distance of about 15 cm, and air-dry the plate. Spray evenly bismuth potassium iodide TS on the plate, and then spray evenly hydrogen peroxide TS on the plate: the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

Water 4.0 – 5.5% (0.2 g, back titration).

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

Assay Weigh accurately about 0.5 g of Dextromethorphan

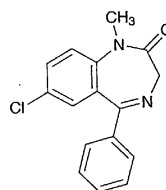
Hydrobromide, dissolve in 10 mL of acetic acid (100) and add 40 mL of acetic anhydride. Titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 35.231 mg of $C_{18}H_{25}NO.HBr$

Containers and storage Containers—Well-closed containers.

Diazepam

ジアゼパム



$C_{16}H_{13}ClN_2O$: 284.74
7-Chloro-1,3-dihydro-1-methyl-5-phenyl-2H-1,4-benzodiazepin-2-one [439-14-5]

Diazepam, when dried, contains not less than 98.0% of $C_{16}H_{13}ClN_2O$.

Description Diazepam occurs as a white to light yellow, crystalline powder. It is odorless, and has a slightly bitter taste.

It is freely soluble in acetone, soluble in acetic anhydride and in ethanol (95), sparingly soluble in diethyl ether, slightly soluble in ethanol (99.5), and practically insoluble in water.

Identification (1) Dissolve 0.01 g of Diazepam in 3 mL of sulfuric acid, and observe under ultraviolet light (main wavelength: 365 nm): the solution shows a yellow-green fluorescence.

(2) Dissolve 2 mg of Diazepam in 200 mL of a solution of sulfuric acid in ethanol (99.5) (3 in 1000). Determine the absorption spectrum of the solution 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.

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

Absorbance $E_{1\text{cm}}^{1\%}$ (285 nm): 425 – 445 [after drying, 2 mg, a solution of sulfuric acid in ethanol (99.5) (3 in 1000), 200 mL].

Melting point 130 – 134°C

Purity (1) Clarity of solution—Dissolve 0.10 g of Diazepam in 20 mL of ethanol (95): the solution is clear.

(2) Chloride—To 1.0 g of Diazepam add 50 mL of water, allow to stand for 1 hour, with occasional shaking, and filter. To 25 mL of the filtrate add 6 mL of dilute nitric acid and 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%).

(3) Heavy metals—Proceed with 1.0 g of Diazepam 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).

(4) Related substances—Dissolve 1.0 g of Diazepam in 10 mL of acetone, and use this solution as the sample solution. Pipette 1 mL of the sample solution, and add acetone to make exactly 100 mL. Pipet 1 mL of this solution, add acetone 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 5 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 the plate with a mixture of ethyl acetate and hexane (1:1) 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.

Loss on drying Not more than 0.5% (1 g, 105°C, 2 hours).

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

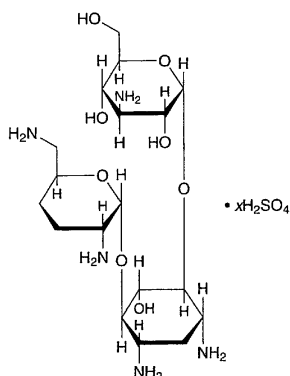
Assay Weigh accurately about 0.6 g of Diazepam, previously dried, dissolve in 60 mL of acetic anhydride, and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 28.475 mg of $C_{16}H_{13}ClN_2O$

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

Dibekacin Sulfate

硫酸ジベカシン



$C_{18}H_{37}N_5O_8 \cdot xH_2SO_4$

O-3-Amino-3-deoxy- α -D-glucopyranosyl-(1 \rightarrow 6)-*O*-[2,6-diamino-2,3,4,6-tetrahydroxy- α -D-erythro-hexopyranosyl-(1 \rightarrow 4)]-2-deoxy-D-streptomine sulfate [58580-55-5]

Dibekacin Sulfate conforms to the requirements of

Dibekacin Sulfate in the Requirements for Antibiotic Products of Japan.

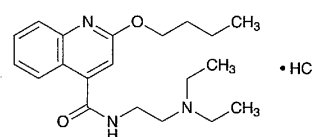
Description Dibekacin Sulfate occurs as a white to yellowish white powder.

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

Dibucaine Hydrochloride

Cinchocaine Hydrochloride

塩酸ジブカイン



$C_{20}H_{29}N_3O_2 \cdot HCl$: 379.92

2-Butyloxy-*N*-(2-diethylaminoethyl)-4-quinolinecarboxamide monohydrochloride [61-12-1]

Dibucaine Hydrochloride, when dried, contains not less than 98.0% of $C_{20}H_{29}N_3O_2 \cdot HCl$.

Description Dibucaine Hydrochloride occurs as white crystals or crystalline powder.

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

It is hygroscopic.

Identification (1) Determine the absorption spectrum of a solution of Dibucaine Hydrochloride in 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.

(2) Determine the infrared absorption spectrum of Dibucaine Hydrochloride, previously dried, 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 Dibucaine Hydrochloride (1 in 10) responds to the Qualitative Tests for chloride.

Melting point 95 – 100°C Charge Dibucaine Hydrochloride into a capillary tube for melting point determination, and dry in vacuum over phosphorus (V) oxide at 80°C for 5 hours. Seal immediately the open end of the tube, and determine the melting point.

pH Dissolve 1.0 g of Dibucaine Hydrochloride in 50 mL of water: the pH of this solution is between 5.0 and 6.0.

Purity (1) Clarity and color of solution—Dissolve 1.0 g of Dibucaine Hydrochloride in 20 mL of water: the solution is clear and colorless. Determine the absorbance of this solution at 430 nm as directed under the Ultraviolet-visible Spectrophotometry, using water as the blank: it is not more than 0.03.