

red color develops, then add 2.0 mL of Standard Lead Solution, 2 mL of dilute acetic acid and water to make 50 mL (not more than 20 ppm).

(3) Related substances—Dissolve 0.010 g of Pentobarbital Calcium in 100 mL of water, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add water to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 20 μ L each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the areas of each peak by the automatic integration method: the area of any peak other than the peak of pentobarbital from the sample solution is not bigger than 3/10 of the peak area of pentobarbital from the standard solution, and the total of these peak area is not bigger than the peak area of pentobarbital from the standard solution.

Operating conditions—

Detector, column, column temperature, mobile phase, and flow rate: Proceed as directed in the operating conditions in the Assay.

Time span of measurement: About 3 times as long as the retention time of pentobarbital after the solvent peak.

System suitability—

Test for required detection: Pipet 2 mL of the standard solution, add water to make exactly 20 mL, and confirm that the peak area of pentobarbital obtained from 20 μ L of this solution is equivalent to 5 to 15% of that of pentobarbital obtained from 20 μ L of the standard solution.

System performance: Proceed as directed in the system performance in the Assay.

System repeatability: When the test is repeated 6 times with 20 μ L of the standard solution under the above operating conditions, the relative standard deviation of the peak areas of pentobarbital is not more than 5%.

Loss on drying Not more than 7.0% (1 g, 105°C, 5 hours).

Assay Weigh accurately about 0.02 g of Pentobarbital Calcium, dissolve in 5 mL of water, add exactly 5 mL of the internal standard solution and water to make 50 mL. To 5 mL of this solution add water to make 20 mL. To 2 mL of this solution add water to make 20 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.018 g of Pentobarbital Reference Standard, previously dried at 105°C for 2 hours, dissolve in 10 mL of acetonitrile, add exactly 5 mL of the internal standard solution and water to make 50 mL. To 5 mL of this solution add water to make 20 mL. To 2 mL of this solution add water to make 20 mL, and use this solution as the standard solution. Perform the test with 20 μ L each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the ratios, Q_T and Q_S , of the peak area of pentobarbital to that of the internal standard.

$$\begin{aligned} & \text{Amount (mg) of } C_{22}H_{34}CaN_4O_6 \\ &= \text{amount (mg) of Pentobarbital Reference Standard} \\ & \times \frac{Q_T}{Q_S} \times 1.0841 \end{aligned}$$

Internal standard solution—Dissolve 0.2 g of isopropyl parahydroxybenzoate in 20 mL of acetonitrile, and add water to make 100 mL.

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 210 nm).

Column: A stainless steel column 4.6 mm in inside diameter and 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μ m in particle diameter).

Column temperature: A constant temperature of about 40°C.

Mobile phase: Dissolve 1.36 g of potassium dihydrogenphosphate in 1000 mL of water, and adjust to pH 4.0 with diluted phosphoric acid (1 in 10). To 650 mL of this solution add 350 mL of acetonitrile.

Flow rate: Adjust the flow rate so that the retention time of pentobarbital is about 7 minutes.

System suitability—

System performance: When the procedure is run with 20 μ L of the standard solution under the above operating conditions, pentobarbital and the internal standard are eluted in this order with the resolution between these peaks being not less than 5.

System repeatability: When the test is repeated 6 times with 20 μ L of the standard solution under the above operating conditions, the relative standard deviation of the ratios of the peak area of pentobarbital to that of the internal standard is not more than 1.0%.

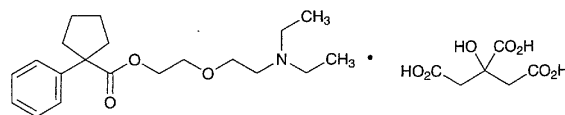
Containers and storage Containers—Well-closed containers.

Pentoxiverine Citrate

Carbetapentane Citrate

Carbetapentene Citrate

クエン酸ペントキシベリン



$C_{20}H_{31}NO_3 \cdot C_6H_8O_7$: 525.59
2-[2-(Diethylamino)ethoxy]ethyl
1-phenylcyclopentanecarboxylate monocitrate
[23142-01-0]

Pentoxiverine Citrate, when dried, contains not less than 98.5% of $C_{20}H_{31}NO_3 \cdot C_6H_8O_7$.

Description Pentoxiverine Citrate occurs as a white, crystalline powder.

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

Identification (1) Dissolve 0.1 g of Pentoxiverine Citrate in 10 mL of water, and add 10 mL of Reinecke salt TS: a light red precipitate is formed.

(2) Determine the infrared absorption spectrum of Pentoxiverine Citrate, previously dried, as directed in the paste method under the Infrared Spectrophotometry, and com-

pare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

(3) A solution of Pentoxiverine Citrate (1 in 10) responds to the Qualitative Tests (1) and (2) for citrate.

Melting point 92 – 95°C

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

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

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

(4) Related substances—Dissolve 0.20 g of Pentoxiverine Citrate in 10 mL of ethanol (95), and use this solution as the sample solution. Pipet 1 mL of the sample solution, add ethanol (95) 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 15 μ L each of the sample solution and the standard solution on a plate of silica gel for thin-layer chromatography. Immediately after air-drying, develop the plate with a mixture of chloroform, methanol, ethyl acetate and ammonia solution (28) (25:10:10:1) to a distance of about 10 cm, and air-dry the plate. Allow to stand in iodine vapor for 10 minutes: 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, in vacuum, phosphorus (V) oxide, 60°C, 4 hours).

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

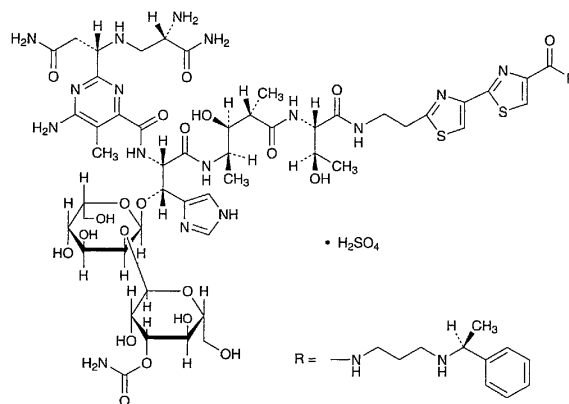
Assay Weigh accurately about 0.5 g of Pentoxiverine Citrate, previously dried, dissolve in 30 mL of acetic acid (100), add 30 mL of acetic anhydride, and titrate with 0.1 mol/L of perchloric acid VS until the color of the solution changes from purple through blue-green to green (indicator: 3 drops of crystal violet TS). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 52.56 mg of $C_{20}H_{31}NO_3 \cdot C_6H_8O_7$

Containers and storage Containers—Well-closed containers.

Peplomycin Sulfate

硫酸ペプロマイシン



$C_{61}H_{88}N_{18}O_{21}S_2 \cdot H_2SO_4$: 1571.67

*N*¹-{3-[(1*S*)-(1-Phenylethyl)amino]propyl}bleomycinamide monosulfate [70384-29-1]

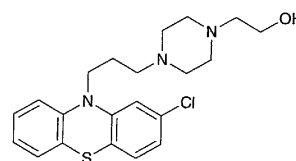
Peplomycin Sulfate conforms to the requirements of Peplomycin Sulfate in the Requirements for Antibiotic Products of Japan.

Description Peplomycin Sulfate occurs as a white to light yellowish white powder.

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

Perphenazine

ペルフェナジン



$C_{21}H_{26}ClN_3OS$: 403.97

2-[4-[3-(2-Chlorophenothiazin-10-yl)propyl]piperazin-1-yl]ethanol [58-39-9]

Perphenazine, when dried, contains not less than 98.5% of $C_{21}H_{26}ClN_3OS$.

Description Perphenazine occurs as white to light yellow crystals or crystalline powder. It is odorless, and has a bitter taste.

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

It dissolves in dilute hydrochloric acid.

It is gradually colored by light.

Identification (1) Dissolve 5 mg of Perphenazine in 5 mL of sulfuric acid: a red color, changing to deep red-purple upon warming, is produced.