

Purity Heavy metals—Proceed with 2.0 g of Diethylcarbamazine Citrate according to Method 4, and perform the test. Prepare the control solution with 4.0 mL of Standard Lead Solution (not more than 20 ppm).

Loss on drying Not more than 1.0% (2 g, 105°C, 4 hours).

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

Assay Weigh accurately about 0.75 g of Diethylcarbamazine Citrate, previously dried, dissolve in 50 mL of acetic acid (100) by warming, cool, 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
= 39.142 mg of $C_{10}H_{21}N_3O \cdot C_6H_8O_7$

Containers and storage Containers—Tight containers.

Diethylcarbamazine Citrate Tablets

クエン酸ジエチルカルバマジン錠

Diethylcarbamazine Citrate Tablets contain not less than 95% and not more than 105% of the labeled amount of diethylcarbamazine citrate ($C_{10}H_{21}N_3O \cdot C_6H_8O_7$; 391.42).

Method of preparation Prepare as directed under Tablets, with Diethylcarbamazine Citrate.

Identification (1) To a quantity of powdered Diethylcarbamazine Citrate Tablets, equivalent to 0.5 g of Diethylcarbamazine Citrate according to the labeled amount, add 10 mL of water, shake, and filter. Add 10 mL of sodium hydroxide TS to the filtrate, and proceed as directed in the Identification (1) under Diethylcarbamazine Citrate.

(2) To a quantity of powdered Diethylcarbamazine Citrate Tablets, equivalent to 0.8 g of diethylcarbamazine citrate according to the labeled amount, add 10 mL of water, shake, centrifuge, and filter the supernatant liquid. To 5 mL of the filtrate add 5 mL of sodium hydroxide TS, and extract with two 20-mL portions of chloroform. Separate the aqueous layer, and neutralize with dilute hydrochloric acid: the solution responds to the Qualitative Tests (2) and (3) for citrate.

Assay Weigh accurately and powder not less than 20 Diethylcarbamazine Citrate Tablets. Weigh accurately a portion of the powder, equivalent to about 0.05 g of diethylcarbamazine citrate ($C_{10}H_{21}N_3O \cdot C_6H_8O_7$), add 10 mL of water, shake well, add 5 mL of sodium hydroxide TS, then add exactly 20 mL of the internal standard solution, and shake vigorously for 10 minutes. Centrifuge, discard the aqueous layer, and use the chloroform layer as the sample solution. Separately, weigh accurately about 0.05 g of Diethylcarbamazine Citrate Reference Standard, previously dried at 105°C for 4 hours, dissolve in 10 mL of water, add 5 mL of sodium hydroxide TS, proceed in the same manner as the preparation of the sample solution, and use the chloroform layer as the standard solution. Perform the test with 2 μ L of the sample solution and the standard solution as directed under the Gas Chromatography according to the following conditions, and calculate the ratios, Q_T and Q_S , of the peak

area of diethylcarbamazine to that of the internal standard, respectively.

$$\begin{aligned} & \text{Amount (mg) of diethylcarbamazine citrate} \\ & (C_{10}H_{21}N_3O \cdot C_6H_8O_7) \\ & = \text{amount (mg) of Diethylcarbamazine Citrate} \\ & \quad \text{Reference Standard} \\ & \quad \times \frac{Q_T}{Q_S} \end{aligned}$$

Internal standard solution—A solution of *n*-octadecane in chloroform (1 in 1250).

Operating conditions—

Detector: A hydrogen flame-ionization detector.

Column: A glass tube 3 mm in inside diameter and 1 to 2 m in length, having methylphenyldimethyl silicone polymer coated at the ratio of 3% on silanized siliceous earth for gas chromatography (180 to 250 μ m in particle diameter).

Column temperature: A constant temperature of about 145°C.

Carrier gas: Nitrogen.

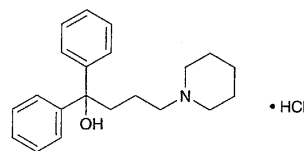
Flow rate: Adjust the flow rate so that the retention time of the internal standard is 8 to 11 minutes.

Selection of column: Proceed with 2 μ L of the standard solution under the above operating conditions, and calculate the resolution. Use a column giving elution of diethylcarbamazine and the internal standard in this order with the resolution between these peaks being not less than 5.

Containers and storage Containers—Well-closed containers.

Difenidol Hydrochloride

塩酸ジフェニドール



$C_{21}H_{27}NO \cdot HCl$; 345.91
1,1-Diphenyl-4-piperidin-1-ylbutan-1-ol
monohydrochloride [3254-89-5]

Difenidol Hydrochloride, when dried, contains not less than 98.5% of $C_{21}H_{27}NO \cdot HCl$.

Description Difenidol Hydrochloride occurs as white crystals or crystalline powder. It is odorless.

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

Melting point: about 217°C (with decomposition).

Identification (1) Dissolve 0.01 g of Difenidol Hydrochloride in 1 mL of sulfuric acid: an orange-red color develops. To this solution add carefully 3 drops of water: the solution becomes yellowish brown, and colorless on the addition of 10 mL of water.

(2) To 5 mL of a solution of Difenidol Hydrochloride (1 in 100) add 2 mL of Reinecke salt TS: a light red precipitate