Piperazine citrate tablets (Piperazini citratis compressi)

Category. Intestinal anthelminthic drug.

Storage. Piperazine citrate tablets should be kept in a tightly closed container.

Additional information. Available strength: 500 mg.

Requirements

Comply with the monograph for Tablets.

Piperazine citrate tablets contain not less than 93.0% and not more than 107.0% of the amount of \((\text{C}_4\text{H}_{10}\text{N}_2)_3\cdot\text{C}_6\text{H}_8\text{O}_7\) stated on the label.

Identity tests

A. See the test described below under "Related substances". Examine the chromatograms after spraying with the two triketohydrindene hydrate reagent solutions. The principal spot obtained with solution B corresponds in position, appearance and intensity with that obtained with solution C.

B. Mix a quantity of the powdered tablets equivalent to 0.2 g of Piperazine citrate with 5 mL of hydrochloric acid (~70 g/l) TS and filter. To the filtrate add 0.5 g of sodium nitrite R and shake to dissolve. Cool in ice for 15 minutes, stir if necessary to induce crystallization, filter, wash with 10 mL of ice-water and dry the precipitate at 105 °C; melting temperature, about 158 °C (N,N’-dinitrosopiperazine).

C. To a quantity of the powdered tablets equivalent to 0.5 g of Piperazine citrate add 10 mL of water, shake and filter. The filtrate yields reaction A described under 2.1 General identification tests as characteristic of citrates.

Related substances

Carry out the test as described under 1.14.1 Thin-layer chromatography using silica gel R1 as the coating substance and a freshly prepared mixture of 2 volumes of ammonia (~260 g/l) TS and 8 volumes of acetone R as the mobile phase. Prepare 500 mL of the following mixture of solvents for solutions B, C, D, E and F: add to 2 volumes of ammonia (~260 g/l) TS 3 volumes of ethanol (~750 g/l) TS. Apply separately to the plate 5 μl of each of the following six solutions. For solution (A) shake a quantity of the powdered tablets equivalent to 1 g of Piperazine citrate with a mixture of 6 mL of ammonia (~260 g/l) TS diluted to 10 mL with water, filter and use the clear filtrate. For solution (B) dilute 7 mL of solution A to 10 mL with the mixture of solvents. For solution (C) dissolve 0.1 g of piperazine citrate RS in sufficient mixture of solvents to produce 10 mL. For solution (D) dissolve 25 mg of ethylenediamine R in sufficient mixture of solvents to produce 100 mL. For solution (E) dissolve 25 mg of triethylenediamine R in sufficient mixture of solvents to produce 100 mL. For solution (F) dissolve 12.5 mg of triethylenediamine R in 5 mL of test solution A and dilute to 50 mL with the mixture of solvents. After removing the plate from the chromatographic chamber allow to dry at 105 °C and spray successively with a 3 mg/mL solution of triketohydrindene hydrate R dissolved in a mixture of 3 volumes of glacial acetic acid R and 100 volumes of 1-butanol R and then with a 1.5 mg/mL solution of triketohydrindene hydrate R in ethanol (~750 g/l) TS. Dry the plate at 105 °C for 10 minutes and examine the chromatogram in daylight.

Any spot obtained with solution A, other than the principal spot, is not more intense than that obtained with solution D. Spray the plate with iodine (0.05 mol/l) VS, allow to stand for 10 minutes and examine the chromatogram in daylight.

Any spot corresponding to triethylenediamine obtained with solution A is not more intense than that obtained with solution E. The test is valid only if the chromatogram obtained with solution F shows two clearly separated spots. Disregard any spot remaining on the line of application.

Assay

Weigh and powder 20 tablets. To a quantity of the powder equivalent to about 0.2 g of Piperazine citrate, accurately weighed, add 10 mL of a mixture of 1 part of hydrochloric acid (~70 g/l) TS and 3 parts of water and shake for 1 hour. Filter and wash the residue with two portions, each of 10 mL, of water. To the combined extract and washings add 100 mL of trinitrophenol (7 g/l) TS, heat on a water-bath for 15 minutes and allow to stand for 1 hour. Filter, wash the residue with successive quantities of trinitrophenol (7 g/l) TS, using 10 mL each time, and finally wash with dehydrated ethanol R. Dry the residue to constant mass at 105 °C.

Each g of residue is equivalent to 393.5 mg of \((\text{C}_4\text{H}_{10}\text{N}_2)_3\cdot\text{C}_6\text{H}_8\text{O}_7\).