Piperazine adipate (Piperazini adipas)

Molecular formula. \( \text{C}_4\text{H}_{10}\text{N}_2\text{C}_6\text{H}_{10}\text{O}_4 \) or \( \text{C}_{10}\text{H}_{20}\text{N}_2\text{O}_4 \)

Relative molecular mass. 232.3

Graphic formula.

Chemical name. Piperazine hexanedioate (1:1); hexahydro-1,4-diazine adipate (1:1); CAS Reg. No. 142-88-1.

Description. Colourless crystals or a white, crystalline powder; odourless.

Solubility. Soluble in water; practically insoluble in ethanol (~750 g/l) TS and ether R.

Category. Anthelmintic.

Storage. Piperazine adipate should be kept in a well-closed container.

Requirements

Definition. Piperazine adipate contains not less than 98.0% and not more than 101.0% of \( \text{C}_4\text{H}_{10}\text{N}_2\text{C}_6\text{H}_{10}\text{O}_4 \), calculated with reference to the dried substance.

Identity tests

A. Dissolve 0.1 g in 5 mL of water, add 0.5 g of sodium hydrogen carbonate R, 0.5 mL of freshly prepared potassium ferricyanide (50 g/l) TS, and 0.1 mL of mercury R. Shake vigorously for 1 minute, and allow to stand for 20 minutes; a reddish colour slowly develops.

B. Dissolve 0.5 g in 10 mL of water and add 5 mL of hydrochloric acid (~250 g/l) TS. Extract 3 times with ether R, using 10 mL each time, and keep the aqueous layer for test C. Evaporate the ether extracts to dryness and dry at 105°C; melting temperature, about 152°C (adipic acid).

C. Cautiously heat the aqueous layer obtained from test B to eliminate any dissolved ether. Cool and add 0.5 g of sodium nitrite R. Heat to boiling and cool in ice for 15 minutes, stirring 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).

Heavy metals. Use 1.0 g for the preparation of the test solution as described under 2.2.3 Limit test for heavy metals, Procedure 1; determine the heavy metals content according to Method A; not more than 20 μg/g.

Sulfated ash. Not more than 1.0 mg/g.

Loss on drying. Dry to constant weight at 105°C; it loses not more than 5.0 mg/g.

pH value. pH of a 0.05 g/mL solution, 5.0-6.0.

Primary amines. For the preparation of the test solution dissolve 0.25 g in sufficient water to produce 50 mL. Transfer 0.5 mL of this solution to a test-tube. Separately transfer to a second test-tube 0.5 mL of a solution containing 10 μg/mL of ethylenediamine R to serve as a reference solution. To both tubes add 0.5 mL of ethanol (~750 g/l) TS, 1 mL of diethoxytetrahydrofuran/acetic acid TS, heat on a water-bath at 80°C for 30 minutes, cool in ice, and add 3 mL of 4-dimethylaminobenzaldehyde TS4. Measure the absorbance at about 570 nm, 7-10 minutes after the addition of the last reagent, against a solvent cell containing the reagents prepared in a similar manner. The absorbance of the test solution is not more intense than that of the reference solution.

Assay. Dissolve about 0.20 g, accurately weighed, in 3.5 mL of sulfuric acid (0.5 mol/l) VS and 10 mL of water; 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, until the washings are free from sulfates. Finally, wash with dehydrated ethanol R, and dry the residue to constant weight at 105°C. Each g of residue is equivalent to 426.8 mg of \( \text{C}_4\text{H}_{10}\text{N}_2\text{C}_6\text{H}_{10}\text{O}_4 \).