

and use this solution as the sample solution. Separately, weigh accurately about 0.01 g of perphenazine for assay, previously dried in vacuum over phosphorus (V) oxide at 65°C for 4 hours, dissolve in methanol to make exactly 250 mL. Pipet 5 mL of this solution, add methanol to make exactly 50 mL, and use this solution as the standard solution. Determine the absorbances,  $A_T$  and  $A_S$ , of the sample solution and the standard solution at 258 nm as directed under the Ultraviolet-visible Spectrophotometry.

$$\begin{aligned} & \text{Amount (mg) of perphenazine (C}_{21}\text{H}_{26}\text{ClN}_3\text{OS)} \\ &= \text{amount (mg) of perphenazine for assay} \\ & \times \frac{A_T}{A_S} \times \frac{V}{25} \times \frac{1}{x} \end{aligned}$$

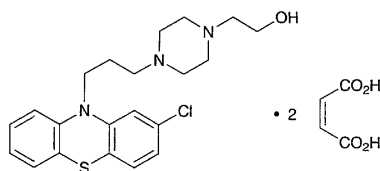
**Assay** Weigh accurately and powder not less than 20 Perphenazine Tablets. Weigh accurately a portion of the powder, equivalent to about 4 mg of perphenazine ( $\text{C}_{21}\text{H}_{26}\text{ClN}_3\text{OS}$ ), add 70 mL of methanol, shake well, and add methanol to make exactly 100 mL. Filter the solution, and discard the first 20 mL of the filtrate. Pipet 5 mL of the subsequent filtrate, add methanol to make exactly 50 mL, and use this solution as the sample solution. Weigh accurately about 0.01 g of perphenazine for assay, previously dried in vacuum over phosphorus (V) oxide at 65°C for 4 hours, and dissolve in methanol to make exactly 250 mL. Pipet 5 mL of this solution, add methanol to make exactly 50 mL, and use this solution as the standard solution. Determine the absorbances,  $A_T$  and  $A_S$ , of the sample solution and the standard solution at 258 nm as directed under the Ultraviolet-visible Spectrophotometry.

$$\begin{aligned} & \text{Amount (mg) of perphenazine (C}_{21}\text{H}_{26}\text{ClN}_3\text{OS)} \\ &= \text{amount (mg) of perphenazine for assay} \\ & \times \frac{A_T}{A_S} \times \frac{2}{5} \end{aligned}$$

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

## Perphenazine Maleate

マレイン酸ペルフェナジン



$\text{C}_{21}\text{H}_{26}\text{ClN}_3\text{OS} \cdot 2\text{C}_4\text{H}_4\text{O}_4$ ; 636.11  
2-[4-[3-(2-Chlorophenothiazin-10-yl)propyl]piperazin-1-yl]ethanol dimaleate [58-39-9, Perphenazine]

Perphenazine Maleate, when dried, contains not less than 98.0% of  $\text{C}_{21}\text{H}_{26}\text{ClN}_3\text{OS} \cdot 2\text{C}_4\text{H}_4\text{O}_4$ .

**Description** Perphenazine Maleate occurs as a white to light yellow powder. It is odorless.

It is sparingly soluble in acetic acid (100), slightly soluble in water and in ethanol (95), and practically insoluble in chloroform.

It dissolves in dilute hydrochloric acid.

It is gradually colored by light.

Melting point: about 175°C (with decomposition).

**Identification (1)** Dissolve 8 mg of Perphenazine Maleate in 5 mL of sulfuric acid: a red color is produced, which becomes deep red-purple on warming.

(2) Dissolve 0.3 g of Perphenazine Maleate in 3 mL of dilute hydrochloric acid, add 2 mL of water and 3 mL of ammonia solution (28), shake, and extract with three 10-mL portions of chloroform. [Reserve the aqueous layer, and use for test (5)]. Evaporate the combined chloroform extracts on a water bath to dryness, dissolve the residue in 20 mL of methanol, and pour into 10 mL of a warm solution of 2,4,6-trinitrophenol in methanol (1 in 25). Allow to stand for 4 hours, collect the crystals, wash with a small amount of methanol, and dry at 105°C for 1 hour: the crystals melt between 237°C and 244°C (with decomposition).

(3) Determine the absorption spectrum of a solution of Perphenazine Maleate (1 in 20,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum 1: both spectra exhibit similar intensities of absorption at the same wavelengths. Separately, to 10 mL of the solution add 30 mL of water. Determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum 2: both spectra exhibit similar intensities of absorption at the same wavelengths.

(4) Perform the test with Perphenazine Maleate as directed under the Flame Coloration Test (2): a green color appears.

(5) Evaporate the aqueous layer reserved in (2) to dryness. To the residue add 1 mL of dilute sulfuric acid and 5 mL of water, and extract with four 25-mL portions of diethyl ether. Combine the diethyl ether extracts, and evaporate in a water bath at about 35°C with the aid of a current of air: the residue melts between 128°C and 136°C.

**Purity (1)** Heavy metals—Proceed with 2.0 g of perphenazine maleate 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).

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

**Loss on drying** Not more than 0.5% (1 g, 105°C, 3 hours).

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

**Assay** Weigh accurately about 0.5 g of Perphenazine Maleate, previously dried, dissolve in 70 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS until the color of the solution changes from purple through blue to blue-green (indicator: 3 drops of crystal violet TS). Perform a blank determination, and make any necessary correction.

$$\begin{aligned} & \text{Each mL of 0.1 mol/L perchloric acid VS} \\ &= 31.806 \text{ mg of } \text{C}_{21}\text{H}_{26}\text{ClN}_3\text{OS} \cdot 2\text{C}_4\text{H}_4\text{O}_4 \end{aligned}$$

**Containers and storage** Containers—Well-closed containers.

Storage—Light-resistant.