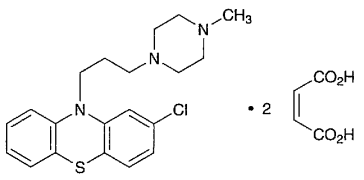
Prochlorperazine Maleate

マレイン酸プロクロルペラジン



$C_{20}H_{24}ClN_3S.2C_4H_4O_4$: 606.09
2-Chloro-10-[3-(4-methylpiperazin-1-yl)propyl]phenothiazine dimaleate [*84-02-6*]

Prochlorperazine Maleate, when dried, contains not less than 98.0% of $C_{20}H_{24}ClN_3S.2C_4H_4O_4$.

Description Prochlorperazine Maleate occurs as a white to light yellow powder. It is odorless, and has a slightly bitter taste.

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

It gradually acquires a red tint by light.

Melting point: 195 - 203°C (with decomposition).

Identification (1) Dissolve 5 mg of Prochlorperazine Maleate in 5 mL of sulfuric acid: a red color develops, which darkens slowly on standing. Warm a half of the solution: the color changes to red-purple. To the remainder add 1 drop of potassium dichromate TS: a green-brown color develops, which changes to brown on standing.

(2) Dissolve 0.2 g of Prochlorperazine Maleate in 5 mL of a solution of sodium hydroxide (1 in 10), and extract with three 3-mL portions of diethyl ether [reserve the aqueous layer, and use for test (4)]. Evaporate the combined diethyl ether extracts on a water bath to dryness, dissolve the residue in 10 mL of methanol by warming, and pour into 30 mL of a solution of 2,4,6-trinitrophenol in methanol (1 in 75), previously warmed to 50°C. Allow to stand for 1 hour, collect the crystals, wash with a small amount of methanol, and dry at 105°C for 1 hour: the crystals melt between 252°C and 258°C (with decomposition).

(3) Boil 0.5 g of Prochlorperazine Maleate with 10 mL of hydrobromic acid under a reflux condenser for 10 minutes. After cooling, add 100 mL of water, and filter through glass filter (G4). Wash the residue with three 10-mL portions of water, and dry at 105°C for 1 hour: it melts between 195°C and 198°C (with decomposition).

(4) To the aqueous layer reserved in (2) add boiling chips, and heat on a water bath for 10 minutes. Cool, add 2 mL of bromine TS, heat on a water bath for 10 minutes, and heat the solution to boil. After cooling, add 2 drops of this solution to 3 mL of a solution of resorcinol in sulfuric acid (1 in 300), and heat on a water bath for 15 minutes: a red-purple color is produced.

Purity Heavy metals—Proceed with 1.0 g of Prochlorperazine Maleate according to Method 2, and perform the test. Prepare the control solution with 1.0 mL of Standard Lead Solution (not more than 10 ppm).

Loss on drying Not more than 1.0% (1 g, 105°C, 3 hours).

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

Assay Weigh accurately about 0.3 g of Prochlorperazine Maleate, previously dried, dissolve in 60 mL of acetic acid (100) while stirring and warming. Cool, and titrate with 0.05 mol/L perchloric acid VS until the color of the solution changes from orange to green (indicator: 0.5 mL of *p*-naphtholbenzein TS). Perform a blank determination, and make any necessary correction.

Each mL of 0.05 mol/L perchloric acid VS
= 15.152 mg of $C_{20}H_{24}ClN_3S.2C_4H_4O_4$

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

Prochlorperazine Maleate Tablets

マレイン酸プロクロルペラジン錠

Prochlorperazine Maleate Tablets contain not less than 95% and not more than 105% of the labeled amount of prochlorperazine maleate ($C_{20}H_{24}ClN_3S.2C_4H_4O_4$: 606.09).

Method of preparation Prepare as directed under Tablets, with Prochlorperazine Maleate.

Identification (1) Weigh a quantity of powdered Prochlorperazine Maleate Tablets, equivalent to 5 mg of Prochlorperazine Maleate according to the labeled amount, add 15 mL of acetic acid (100), shake, and filter. To 5 mL of the filtrate add 3 mL of sulfuric acid, and shake: a light red color develops. To this solution add 1 drop of potassium dichromate TS: a green-brown color is produced and changes to brown on standing.

(2) Weigh a quantity of powdered Prochlorperazine Maleate Tablets, equivalent to 0.08 g of Prochlorperazine Maleate according to the labeled amount, add 15 mL of methanol and 1 mL of dimethylamine, shake, centrifuge, and use the supernatant liquid as the sample solution. Separately, dissolve 0.08 g of Prochlorperazine Maleate Reference Standard in 16 mL of a mixture of methanol and dimethylamine (15:1), and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μL each of the sample solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of 1-butanol and ammonia TS (15:2) to a distance of about 10 cm, and air-dry the plate. Spray evenly palladium (II) chloride TS on the plate: the spots obtained from the sample solution and the standard solution show a red-purple color, and has the same *R*f value.

(3) To a quantity of powdered Prochlorperazine Maleate Tablets, equivalent to 0.04 g of Prochlorperazine Maleate according to the labeled amount, add 10 mL of 1 mol/L hydrochloric acid TS and 20 mL of diethyl ether, shake, and centrifuge. Transfer the diethyl ether layer to a separator, wash with 5 mL of 0.05 mol/L sulfuric acid TS, and evaporate on a water bath to dryness. Dissolve the residue in 5 mL of sulfuric acid TS, filter, if necessary, and add 1 to 2 drops of potassium permanganate TS: the red color of the test solution is discharged immediately.

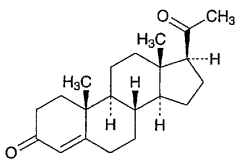
Assay Weigh accurately and powder not less than 20 Prochlorperazine Maleate Tablets using an agate mortar. Weigh accurately a portion of the powder, equivalent to about 0.016 g of prochlorperazine maleate ($C_{20}H_{24}ClN_3S.2C_4H_4O_4$), transfer to a glass-stoppered centrifuge tube, add exactly 25 mL of a mixture of *N,N*-dimethylformamide and dimethylamine (100:1), stopper tightly, shake vigorously for 15 minutes, and centrifuge. Use the supernatant liquid as the sample solution. Separately, weigh accurately about 0.064 g of Prochlorperazine Maleate Reference Standard, previously dried in a desiccator (in vacuum, silica gel) for 4 hours, dissolve in a mixture of *N,N*-dimethylformamide and dimethylamine (100:1) to make exactly 100 mL, and use this solution as the standard solution. Pipet 4 mL each of the sample solution and the standard solution into glass-stoppered centrifuge tubes, add exactly 10 mL of boric acid-potassium chloride-sodium hydroxide buffer solution, pH 9.0, and 20 mL of cyclohexane, stopper tightly, and centrifuge after shaking vigorously for 5 minutes. Pipet 10 mL each of the cyclohexane layer of these solutions into glass-stoppered centrifuge tubes, add exactly 20 mL of palladium (II) chloride TS and 5 mL of *N,N*-dimethylformamide, stopper tightly, and centrifuge after shaking vigorously for 15 minutes. Determine the absorbances, A_T and A_S, of the water layers obtained from the sample solution and the standard solution at 495 nm as directed under the Ultraviolet-visible Spectrophotometry, using palladium (II) chloride TS as the blank.

Amount (mg) of prochlorperazine maleate ($C_{20}H_{24}ClN_3S.2C_4H_4O_4$)
= amount (mg) of Prochlorperazine Maleate Reference Standard
$\times \frac{A_T}{A_S} \times \frac{1}{4}$

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

Progesterone

プロゲステロン



$C_{21}H_{30}O_2$: 314.46
Pregn-4-ene-3,20-dione [*57-83-0*]

Progesterone, when dried, contains not less than 97.0% and not more than 103.0% of $C_{21}H_{30}O_2$.

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

It is soluble in methanol, in ethanol (95), in ethanol (99.5) and in 1,4-dioxane, sparingly soluble in diethyl ether, and practically insoluble in water.

Identification (1) To 0.05 g of progesterone add a solution of 0.05 g of hydroxylammonium chloride and 0.05 g of anhydrous sodium acetate in 5 mL of ethanol (95). Boil for 2 hours under a reflux condenser, evaporate the ethanol to 3 mL, and add 10 mL of water. Filter by suction, and wash the precipitate on the filter with a small amount of water. Recrystallize from dilute ethanol, and dry at 105°C for 1 hour: the dried crystals melt between 235°C and 240°C.

(2) Determine the infrared absorption spectrum of Progesterone, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of previously dried Progesterone Reference Standard: both spectra exhibit similar intensities of absorption at the same wave numbers. If any difference appears between the spectra, dissolve Progesterone and Progesterone Reference Standard in ethanol (95), respectively, then evaporate the ethanol to dryness, and repeat the test on the residues.

Optical rotation $[\alpha]_D^{20}$: +174 – +182° (after drying, 0.2 g, 1,4-dioxane, 10 mL, 100 mm).

Melting point 128 – 133°C or 120 – 122°C

Purity Other steroids—Dissolve 0.080 g of Progesterone in 2 mL of methanol, and use this solution as the sample solution. Pipet 1 mL of this solution, add methanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μL each of the sample solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of chloroform and diethylamine (19:1) to a distance of about 15 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): 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% (0.5 g, in vacuum, phosphorus (V) oxide, 4 hours)

Residue on ignition Not more than 0.1% (0.5 g).

Assay Weigh accurately about 0.01 g of Progesterone, previously dried, and dissolve in ethanol (99.5) to make exactly 100 mL. To 5 mL of this solution, exactly measured, add ethanol (99.5) to make exactly 50 mL, and determine the absorbance A at the wavelength of maximum absorption at about 241 nm as directed under the Ultraviolet-visible Spectrophotometry.

$$\text{Amount (mg) of } C_{21}H_{30}O_2 = \frac{A}{540} \times 10{,}000$$

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

Progesterone Injection

プロゲステロン注射液

Progesterone Injection is an oily solution for injection. It contains not less than 90% and not more than 110% of the labeled amount of progesterone