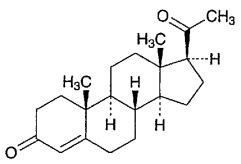
Assay Weigh accurately and powder not less than 20 Prochlorperazine Maleate Tablets using an agate mortar. Weigh accurately a portion of the powder, equivalent to about 0.016 g of prochlorperazine maleate ($C_{20}H_{24}ClN_3S.2C_4H_4O_4$), transfer to a glass-stoppered centrifuge tube, add exactly 25 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

($C_{21}H_{30}O_2$: 314.46).

Method of preparation Prepare as directed under Injections, with Progesterone.

Description Progesterone Injection is a clear, colorless to pale yellow, oily liquid.

Identification Transfer a volume of Progesterone Injection, equivalent to 0.02 g of progesterone according to the labeled amount, to a separator. Add 40 mL of hexane, and mix thoroughly, then extract with three 20-mL portions of diluted ethanol (99.5) (9 in 10). Evaporate the combined extracts on a water bath to dryness. Add 0.075 g of 2,4-dinitrophenylhydrazine and 30 mL of ethanol (95) to the residue, and boil for 15 minutes under a reflux condenser. Add 1 mL of hydrochloric acid, and heat for 15 minutes. Cool, and collect the precipitate on a glass filter (G4). Wash the precipitate with five 10-mL portions of hexane and three 5-mL portions of ethanol (95). Then wash with diluted hydrochloric acid (1 in 20) until the washings become colorless, and dry at 105°C for 3 hours: the residue melts between 269°C and 275°C.

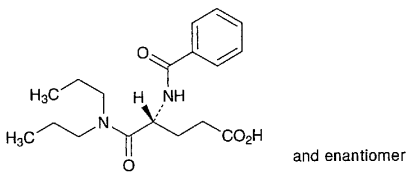
Assay Measure exactly a volume of Progesterone Injection, equivalent to about 0.05 g of progesterone ($C_{21}H_{30}O_2$), and dissolve in chloroform to make exactly 100 mL. To exactly measured 3 mL of this solution add chloroform to make exactly 50 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.05 g of Progesterone Reference Standard, previously dried in a desiccator (in vacuum, phosphorus (V) oxide) for 4 hours, and prepare the standard solution in the same manner as directed for the preparation of the sample solution. Pipet 5 mL each of the sample solution and the standard solution, add exactly measured 10 mL of isoniazid TS and methanol to make exactly 20 mL, respectively. Allow to stand for 45 minutes, and perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry, using a solution, prepared with 5 mL of chloroform in the same manner, as the blank. Determine the absorbances, A_T and A_S, of the subsequent solutions of the sample solution and the standard solution at 380 nm.

Amount (mg) of progesterone ($C_{21}H_{30}O_2$)
= amount (mg) of Progesterone Reference Standard
$\times \frac{A_T}{A_S}$

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

Proglumide

プログルミド



and enantiomer

$C_{18}H_{26}N_2O_4$: 334.41

(*RS*)-4-Benzoylamino-*N*,*N*-dipropylglutaramic acid
[*6620-60-6*]

Proglumide, when dried, contains not less than 98.5% of $C_{18}H_{26}N_2O_4$.

Description Proglumide occurs as white crystals or crystalline powder. It is freely soluble in methanol, soluble in ethanol (95), sparingly soluble in diethyl ether, and very slightly soluble in water.

A solution of Proglumide in methanol (1 in 10) shows no optical rotation.

Identification **(1)** Put 0.5 g of Proglumide in a round bottom tube, add 5 mL of hydrochloric acid, seal the tube, and heat the tube carefully at 120°C for 3 hours. After cooling, open the tube, filter the content to collect crystals separated out, wash the crystals with 50 mL of water, and dry at 100°C for 1 hour: the melting point of the crystals is between 121°C and 124°C.

(2) Determine the infrared absorption spectrum of Proglumide, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

Absorbance $E^{1\%}_{1cm}$ (225 nm): 384 - 414 (after drying, 4 mg, methanol, 250 mL)

Melting point 148 - 150°C

Purity **(1)** Heavy metals—Proceed with 1.0 g of Proglumide according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(2) Arsenic—To 1.0 g of Proglumide add 10 mL of a solution of magnesium nitrate hexahydrate in ethanol (95) (1 in 10) and 1.5 mL of hydrogen peroxide solution, burn the ethanol, and prepare the test solution according to Method 3, and perform the test using Apparatus B (not more than 2 ppm).

(3) Related substances—Dissolve 0.10 g of Proglumide in 5 mL of methanol, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add methanol to make exactly 200 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 cyclohexane, ethyl acetate, acetic acid (100) and methanol (50:18:5:4) to a distance of about 10 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 0.10% (1 g, reduced pressure, phosphorus (V) oxide, 60°C, 3 hours).

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

Assay Weigh accurately about 0.16 g of Proglumide, previously dried, dissolve in 40 mL of methanol, add 10 mL of water, and titrate with 0.1 mol/L sodium hydroxide VS (potentiometric titration). Perform a blank determination,