velops.

(3) Determine the infrared absorption spectrum of Drostanolone Propionate, 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 Drostanolone Propionate Reference Standard: both spectra exhibit similar intensities of absorption at the same wave numbers. If any difference appears between the spectra, dissolve Drostanolone Propionate and Drostanolone Propionate Reference Standard in chloroform, respectively, then evaporate the chloroform to dryness, and repeat the test on the residues.

Optical rotation $[\alpha]_D^{20}$: +22 – +28° (after drying, 0.2 g, chloroform, 10 mL, 100 mm).

Melting point 129 – 133°C

Purity **(1)** Clarity and color of solution—Dissolve 0.20 g of Drostanolone Propionate in 10 mL of chloroform: the solution is clear and colorless.

(2) Heavy metals—Proceed with 1.0 g of Drostanolone Propionate 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).

(3) Other steroids—Dissolve 0.05 g of Drostanolone Propionate in 5 mL of chloroform, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add chloroform 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 for thin-layer chromatography. Develop the plate with a mixture of heptane and ethyl acetate (9:1) to a distance of about 10 cm, and air-dry the plate. Develop the plate with the same solvent again, and air-dry the plate. Spray vanillin-sulfuric acid TS on the plate, and heat at 105°Cfor 5 minutes: 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, 50°C, 2 hours).

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

Assay Weigh accurately about 0.025 g of Drostanolone Propionate and Drostanolone Propionate Reference Standard, previously dried, dissolve each in exactly 5 mL of the internal standard solution, add chloroform to make 10 mL, and use these solutions as the sample solution and the standard solution, respectively. Perform the test with 2 μL each of the sample solution and the standard solution as directed under the Gas Chromatography according to the following conditions, and calculate the ratios, Q_T and Q_S, of the peak area of drostanolone propionate to that of the internal standard, respectively.

$$\text{Amount (mg) of } C_{23}H_{36}O_3 = \text{amount (mg) of Drostanolone Propionate Reference Standard} \times \frac{Q_T}{Q_S}$$

Internal standard solution—A solution of cholesterol in chloroform (1 in 200).

Operating conditions—

Detector: A hydrogen flame-ionization detector.

Column: A glass column about 3 mm in inside diameter and about 1 m in length, packed with siliceous earth for gas chromatography (125 to 150 μm in particle diameter) coated with 50% phenylmethyl silicon polymer for gas chromatography at the ratio of 3%.

Column temperature: A constant temperature of about 260°C.

Carrier gas: Nitrogen

Flow rate: Adjust the flow rate so that the retention time of drostanolone propionate is about 8 minutes.

Selection of column: Proceed with 2 μL of the standard solution under the above operating conditions, and calculate the resolution. Use a column giving elution of drostanolone propionate and the internal standard in this order with the resolution between these peaks being not less than 3.

Containers and storage Containers—Tight containers.

Storage—Light-resistant.

Drostanolone Propionate Injection

Dromostanolone Propionate Injection

プロピオン酸ドロスタノロン注射液

Drostanolone Propionate Injection is an oily solution for injection. It contains not less than 90% and not more than 110% of the labeled amount of drostanolone propionate ($C_{23}H_{36}O_3$: 360.53).

Method of preparation Prepare as directed under Injections, with Drostanolone Propionate.

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

Identification To a volume of Drostanolone Propionate Injection, equivalent to 0.05 g of drostanolone propionate according to the labeled amount, add 20 mL of diluted methanol (4 in 5), shake for 5 minutes, and centrifuge. Evaporate 5 mL of the supernatant liquid on a water bath to dryness under a current of air. Dissolve the residue in 2 mL of chloroform, and use this solution as the sample solution. Separately, dissolve 5 mg of Drostanolone Propionate Reference Standard in 1 mL of chloroform, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5 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 heptane and ethyl acetate (9:1) to a distance of about 12 cm, and air-dry the plate. Spray evenly vanillin-sulfuric acid TS on the plate, and heat at 105°C for 5 minutes: the principal spot from the sample solution and the spot from the standard solution show the same color tone and *R*f value.

Assay (i) Chromatographic tube: Use a glass tube about 20 mm in inside diameter and about 23 cm in length. Place a small amount of absorbent cotton in the bottom of the tube, and put sea sand 5 mm in height on it.

(ii) Chromatographic column: Moisten 10 g of 4%

hydrated alumina neutral thoroughly with 15 to 20 mL of hexane, and mix gently. Wash with hexane down to the chromatographic tube, and pack by flowing out the solution. Place sea sand 5 mm in height on it, and fill with hexane up to the surface of sea sand.

(iii) Standard solution: Weigh accurately about 0.025 g of Drostanolone Propionate Reference Standard, previously dried in a desiccator (in vacuum, phosphorus (V) oxide, 50°C) for 2 hours, and dissolve in exactly 10 mL of the internal standard solution.

(iv) Sample stock solution: Pipet a volume of Drostanolone Propionate Injection, equivalent to about 0.05 g of drostanolone propionate ($C_{23}H_{36}O_3$), and add hexane to make exactly 20 mL.

(v) Procedure: Pipet 5 mL of the sample stock solution into the previously prepared chromatographic column, and elute to the surface of sea sand. Wash the inner side of the chromatographic tube with two 5-mL portions of hexane, elute to the surface of sea sand, then elute 120 mL of a mixture of hexane and ethyl acetate (50:1) at the rate of 7 to 8 mL per minute, and discard the effluent solution. Elute 150 mL of a mixture of hexane and ethyl acetate (20:1) at the rate of 7 to 8 mL per minute, and collect the effluent solution. After elution, wash the bottom part of the chromatographic tube with a small quantity of hexane, combine the washing with the effluent solution, and evaporate the solvent at below 30°C. To the residue add exactly 5 mL of the internal standard solution, and use this solution as the sample solution. Proceed with 2 μL each of the sample solution and the standard solution as directed in the Assay under Drostanolone Propionate.

Amount (mg) of drostanolone propionate ($C_{23}H_{36}O_3$)
= amount (mg) of Drostanolone Propionate Reference Standard
$\times \frac{Q_T}{Q_S} \times 2$

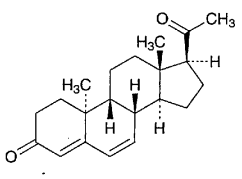
Internal standard solution—A solution of cholesterol in chloroform (1 in 400).

Containers and storage Containers—Hermetic containers, and colored containers may be used.

Storage—Light-resistant.

Dydrogesterone

ジドロゲステロン



$C_{21}H_{28}O_2$: 312.45
9β,10α-Pregna-4,6-diene-3,20-dione [*152-62-5*]

Dydrogesterone, when dried, contains not less than 98.0% and not more than 102.0% of $C_{21}H_{28}O_2$.

Description Dydrogesterone occurs as white to light yellowish white crystals or crystalline powder. It is odorless.

It is freely soluble in chloroform, soluble in acetonitrile, sparingly soluble in methanol and in ethanol (95), slightly soluble in diethyl ether, and practically insoluble in water.

Identification **(1)** To 5 mg of Dydrogesterone add 5 mL of 4-methoxybenzaldehyde-acetic acid TS and 2 to 3 drops of sulfuric acid, and heat in a water bath for 2 minutes: an orange-red color develops.

(2) Determine the absorption spectrum of a solution of Dydrogesterone in methanol (1 in 200,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelengths.

(3) Determine the infrared absorption spectrum of Dydrogesterone, 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.

Optical rotation $[\alpha]_D^{20}$: -470 – $-500°$ (after drying, 0.1 g, chloroform, 10 mL, 100 mm).

Melting point 167 – 171°C

Purity **(1)** Heavy metals—Proceed with 1.0 g of Dydrogesterone 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) Other steroids—Dissolve 0.010 g of Dydrogesterone in 200 mL of the mobile phase, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add the mobile phase to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Determine each peak area of these solutions by the automatic integration method: the total area of peaks other than the peak of dydrogesterone from the sample solution is not larger than the peak area of dydrogesterone from the standard solution.

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 280 nm).

Column: A stainless steel column about 4 mm in inside diameter and about 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (3 μm in particle diameter).

Column temperature: A constant temperature of about 40°C.

Mobile phase: A mixture of water, ethanol (95) and acetonitrile (53:26:21).

Flow rate: Adjust the flow rate so that the retention time of dydrogesterone is about 12 minutes.

Selection of column: Dissolve 1 mg each of Dydrogesterone and progesterone in 20 mL of the mobile phase. Proceed with 10 μL each of these solutions under the above operating conditions, and calculate the resolution. Use a column giving elution of dydrogesterone and progesterone in this order with the resolution between these peaks being not less than 8. Wavelength is 265 nm.

Detection sensitivity: Adjust the detection sensitivity so that the peak height of dydrogesterone obtained from 10 μL of the standard solution is between 5 mm and 10 mm.

Time span of measurement: About twice as long as the