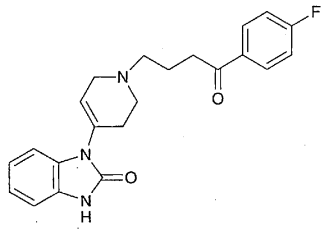
Droperidol

ドロペリドール



$C_{22}H_{22}FN_3O_2$: 379.43
1-{1-[4-(4-Fluorophenyl)-4-oxobutyl]-1,2,3,6-tetrahydropyridine-4-yl}-1,3-dihydro-2*H*-benzimidazol-2-one
[*548-73-2*]

Droperidol, when dried, contains not less than 98.0% of $C_{22}H_{22}FN_3O_2$.

Description Droperidol occurs as a white to light yellow powder.

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

It is gradually colored by light.

Identification (1) To 10 mL of a solution of Droperidol in chloroform (1 in 10,000) add 5 mL of bromophenol blue-potassium biphthalate TS, shake, and allow to stand: a yellow color develops in the chloroform layer.

(2) Put 0.03 g of Droperidol in a brown volumetric flask, and dissolve in 10 mL of 0.1 mol/L hydrochloric acid TS and ethanol (95) to make 100 mL. Transfer 5 mL of the solution to a brown volumetric flask, and add 10 mL of 0.1 mol/L hydrochloric acid TS and ethanol (95) to make 100 mL. Determine the absorption spectrum of the solution 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 Droperidol, 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.

Melting point 141 - 145°C

Purity (1) Heavy metals—Proceed with 1.0 g of Droperidol in a platinum crucible 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) Related substances—Conduct this procedure without exposure to daylight, using light-resistant vessels. Dissolve 0.050 g of Droperidol in 5 mL of dichloromethane, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add dichloromethane 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 ethyl acetate, chloroform, methanol and acetic acid-sodium acetate buffer solution, pH 4.7, (54:23:18:5) 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 3.0% (0.5 g, in vacuum, silica gel, 70°C, 4 hours).

Residue on ignition Not more than 0.20% (1 g, platinum crucible).

Assay Weigh accurately about 0.5 g of Droperidol, previously dried, dissolve in 50 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

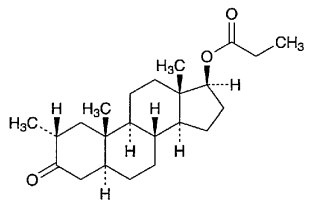
Each mL of 0.1 mol/L perchloric acid VS
= 37.943 mg of $C_{22}H_{22}FN_3O_2$

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

Drostanolone Propionate

Dromostanolone Propionate

プロピオン酸ドロスタノロン



$C_{23}H_{36}O_3$: 360.53
2α-Methyl-3-oxo-5α-androstan-17β-yl propionate
[*521-12-0*]

Drostanolone Propionate, when dried, contains not less than 97.0% and not more than 103.0% of $C_{23}H_{36}O_3$.

Description Drostanolone Propionate occurs as a white to yellowish white, crystalline powder. It is odorless, or has a faint, characteristic odor.

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

Identification (1) Dissolve 0.02 g of Drostanolone Propionate in 1 mL of ethanol (95), add 1 mL of alkaline hydroxylamine TS, allow to stand for 10 minutes, and add 1 mL of hydrochloric acid-ethanol TS and 1 mL of iron (III) chloride TS: a dark red color develops.

(2) To 0.01 g of Drostanolone Propionate add 10 mL of freshly prepared vanillin-sulfuric acid TS, and dissolve by heating in a water bath for 5 minutes: a red-purple color de-

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%