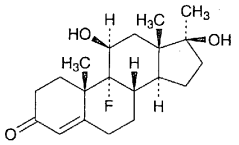
sodium hydroxide TS and 20 mL of water as the absorbing liquid.

Containers and storage Containers—Tight containers.

Fluoxymesterone

フルオキシメステロン



$C_{20}H_{29}FO_3$: 336.44

9-Fluoro-11β,17β-dihydroxy-17-methylandrost-4-en-3-one [*76-43-7*]

Fluoxymesterone, when dried, contains not less than 97.0% and not more than 102.0% of $C_{20}H_{29}FO_3$.

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

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

Identification (1) Dissolve 5 mg of Fluoxymesterone in 2 mL of sulfuric acid: a yellow color develops.

(2) Prepare the test solution with 0.01 g of Fluoxymesterone as directed under the Oxygen Flask Combustion Method, using a mixture of 0.5 mL of 0.01 mol/L sodium hydroxide TS and 20 mL of water as an absorbing liquid: the test solution responds to the Qualitative Tests (2) for fluoride.

(3) Determine the absorption spectrum of a solution of Fluoxymesterone in ethanol (95) (1 in 100,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of a solution of Fluoxymesterone Reference Standard prepared in the same manner as the sample solution: both spectra exhibit similar intensities of absorption at the same wavelengths.

(4) Determine the infrared absorption spectrum of Fluoxymesterone, 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 Fluoxymesterone Reference Standard: both spectra exhibit similar intensities of absorption at the same wave numbers. If any difference appears between the spectra, dissolve Fluoxymesterone and Fluoxymesterone Reference Standard in ethanol (99.5), respectively, then evaporate the ethanol to dryness, and repeat the test on the residues.

Optical rotation $[\alpha]_D^{20}$: +104 – +112° (after drying, 0.1 g, ethanol (95), 10 mL, 100 mm).

Purity (1) Heavy metals—Proceed with 0.5 g of Fluoxymesterone according to Method 2, and perform the test. Prepare the control solution with 1.5 mL of Standard Lead Solution (not more than 30 ppm).

(2) Other steroids—Dissolve 0.03 g of Fluoxymesterone in 10 mL of methanol, and use this solution as the sample solution. Pipet 1 mL of the sample 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 toluene, ethanol (95) and ethyl acetate (3:1:1) to a distance of about 12 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 1.0% (1 g, 105°C, 3 hours).

Residue on ignition Not more than 0.2% (0.5 g, platinum crucible).

Assay Weigh accurately about 0.025 g each of Fluoxymesterone and Fluoxymesterone Reference Standard, previously dried, dissolve each in the internal standard solution to make exactly 100 mL, and use these solutions as the sample solution and the standard solution, respectively. 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, and calculate the ratios, Q_T and Q_S, of the peak area of fluoxymesterone to that of the internal standard, respectively.

$$\text{Amount (mg) of fluoxymesterone } (C_{20}H_{29}FO_3) = \text{amount (mg) of Fluoxymesterone Reference Standard} \times \frac{Q_T}{Q_S}$$

Internal standard solution—A solution of methylprednisolone in a mixture of chloroform and methanol (19:1) (1 in 5000).

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 254 nm).

Column: A stainless steel column 4.6 mm in inside diameter and 30 cm in length, packed with silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 25°C.

Mobile phase: A mixture of *n*-butyl chloride, water-saturated *n*-butyl chloride, tetrahydrofuran, methanol and acetic acid (100) (95:95:14:7:6).

Flow rate: Adjust the flow rate so that the retention time of fluoxymesterone is about 9 minutes.

System suitability—

System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, fluoxymesterone and the internal standard are eluted in this order with the resolution between these peaks being not less than 6.

System repeatability: When the test is repeated 6 times with 10 μL of the standard solution under the above operating conditions, the relative standard deviation of the ratios of the peak area of fluoxymesterone to that of the internal standard is not more than 1.5%.

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.