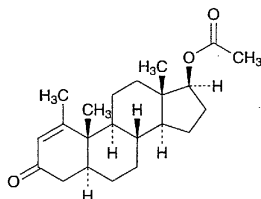


$$\begin{aligned} & \text{Amount (mg) of } C_{21}H_{26}O_2 \\ &= \text{amount (mg) of Mestranol Reference Standard} \\ & \quad \times \frac{A_T}{A_S} \end{aligned}$$

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

Metenolone Acetate

酢酸メテノロン



$C_{22}H_{32}O_3$: 344.49
1-Methyl-3-oxo-5 α -androst-1-en-17 β -yl acetate [434-05-9]

Metenolone Acetate, when dried, contains not less than 97.0% and not more than 103.0% of $C_{22}H_{32}O_3$.

Description Metenolone Acetate occurs as a white to pale yellowish white, crystalline powder. It is odorless.

It is freely soluble in acetone, in 1,4-dioxane and in chloroform, soluble in ethanol (95) and in methanol, sparingly soluble in diethyl ether and in sesame oil, slightly soluble in hexane and in petroleum ether, and practically insoluble in water.

Identification (1) Dissolve 1 mg of Metenolone Acetate in 5 mL of a mixture of ethanol (95) and sulfuric acid (1:1), and heat for 30 minutes in a water bath: a red-brown color develops.

(2) To 0.01 g of Metenolone Acetate add 0.5 mL of dilute sodium hydroxide-ethanol TS, and heat for 1 minute on a water bath. After cooling, add 0.5 mL of diluted sulfuric acid (1 in 2), and boil gently for 1 minute: the odor of ethyl acetate is perceptible.

(3) Dissolve 0.05 g of Metenolone Acetate in 3 mL of methanol, add 0.3 mL of a solution of potassium carbonate (1 in 6), and boil for 2 hours under a reflux condenser. After cooling, add this solution gradually to 50 mL of cold water, and stir for 15 minutes. Filter the precipitate so obtained by suction through a glass filter (G4), wash with 10 mL of water, and dry at 105°C for 1 hour: it melts between 157°C and 161°C.

(4) Determine the infrared absorption spectrum of Metenolone Acetate, 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 $[\alpha]_D^{20}$: +39 – +42° (after drying, 0.2 g, chloroform, 10 mL, 100 mm).

Melting point 141 – 144°C

Purity (1) Clarity and color of solution—Dissolve 0.50 g

of Metenolone Acetate in 10 mL of 1,4-dioxane: the solution is clear and colorless to pale yellow.

(2) Heavy metals—Proceed with 2.0 g of Metenolone Acetate according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

(3) Other steroids—Dissolve 0.035 g of Metenolone Acetate in 20 mL of chloroform, and use this solution as the sample solution. Pipet 1 mL of the sample solution, dilute with chloroform to exactly 250 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μ L 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 and ethyl acetate (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 0.5% (0.5 g, 105°C, 3 hours).

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

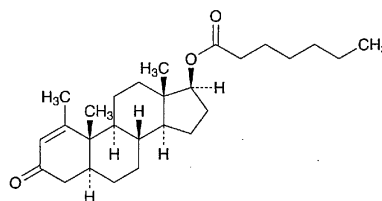
Assay Weigh accurately about 0.01 g of Metenolone Acetate, previously dried, and dissolve in methanol to make exactly 100 mL. Pipet 5 mL of this solution, and dilute with methanol to exactly 50 mL. Determine the absorbance A of this solution at the wavelength of maximum absorption at about 242 nm.

$$\text{Amount (mg) of } C_{22}H_{32}O_3 = \frac{A}{391} \times 10,000$$

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

Metenolone Enanthate

エナント酸メテノロン



$C_{27}H_{42}O_3$: 414.62
1-Methyl-3-oxo-5 α -androst-1-en-17 β -yl heptanoate [303-42-4]

Metenolone Enanthate, when dried, contains not less than 97.0% and not more than 103.0% of $C_{27}H_{42}O_3$.

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

It is very soluble in ethanol (95), in acetone, in 1,4-dioxane and in chloroform, freely soluble in methanol, in ethyl acetate, in diethyl ether, in cyclohexane, in petroleum

ether and in toluene, soluble in sesame oil, and practically insoluble in water.

Identification (1) Heat 1 mg of Metenolone Enanthate with 5 mL of a mixture of ethanol (95) and sulfuric acid (1:1) on a water bath for 30 minutes: a red-brown color develops.

(2) Dissolve 0.05 g of Metenolone Enanthate in 3 mL of methanol, add 0.3 mL of a solution of potassium carbonate (1 in 6), boil under a reflux condenser for 2 hours, cool, add slowly this solution to 50 mL of cold water, and stir for 15 minutes. Filter the produced precipitate by suction through a glass filter (G4), wash with water until the washings become neutral, and dry at 105°C for 1 hour: it melts between 156°C and 162°C.

Optical rotation $[\alpha]_D^{20}$: +39 – +43° (after drying, 0.2 g, chloroform, 10 mL, 100 mm).

Melting point 67 – 72°C

Purity (1) Clarity and color of solution—Dissolve 0.5 g of Metenolone Enanthate in 10 mL of 1,4-dioxane: the solution is clear and colorless.

(2) Heavy metals—Proceed with 2.0 g of Metenolone Enanthate according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

(3) Other steroids—Dissolve 0.020 g of Metenolone Enanthate in exactly 10 mL of chloroform, and use this solution as the sample solution. Perform the test with the sample solution as directed under the Thin-layer Chromatography. Spot 10 μ L of the sample solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of cyclohexane and ethyl acetate (1:1) to a distance of about 15 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): any spot other than the principal spot does not appear.

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 0.1 g of Metenolone Enanthate, previously dried, and dissolve in methanol to make exactly 100 mL. Pipet 10 mL of this solution, and dilute with methanol to make exactly 100 mL. Pipet 10 mL of this solution, and dilute again with methanol to make exactly 100 mL. Determine the absorbance A of this solution at the wavelength of maximum absorption at about 242 nm as directed under the Ultraviolet-visible Spectrophotometry.

$$\text{Amount (mg) of } C_{27}H_{42}O_3 = \frac{A}{325} \times 100,000$$

Containers and storage Containers—Tight containers.

Storage—Light-resistant.

Metenolone Enanthate Injection

エナント酸メテノロン注射液

Metenolone Enanthate Injection is an oily solution

for injection. It contains not less than 90% and not more than 110% of the labeled amount of metenolone enanthate ($C_{27}H_{42}O_3$; 414.62).

Method of preparation Prepare as directed under Injections, with Metenolone Enanthate.

Description Metenolone Enanthate Injection is a clear, pale yellow, oily liquid.

Identification (1) Measure a volume of Metenolone Enanthate Injection, equivalent to 0.1 g of Metenolone Enanthate according to the labeled amount, add 20 mL of petroleum ether, and extract with three 20-mL portions of diluted acetic acid (31) (5 in 7). Combine the extracts, wash with 20 mL of petroleum ether, add 300 mL of cold water while cooling in an ice bath, and stir sufficiently. Filter the produced precipitate by suction through a glass filter (G4), wash with water until the last washing becomes neutral, and dry in a desiccator (in vacuum, phosphorus (V) oxide) for 6 hours. With this sample, proceed as directed in the Identification (1) under Metenolone Enanthate.

(2) Measure a volume of Metenolone Enanthate Injection, equivalent to 0.01 g of Metenolone Enanthate according to the labeled amount, dissolve in 10 mL of chloroform, and use this solution as the sample solution. Separately dissolve 0.01 g of metenolone enanthate in 10 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 10 μ L 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 toluene to a distance of about 15 cm, and air-dry the plate. Again develop this plate with a mixture of cyclohexane and ethyl acetate (1:1) to a distance of about 15 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the principal spot from the sample solution and the spot from the standard solution show the same R_f value.

Assay To an exactly measured volume of Metenolone Enanthate Injection, equivalent to about 0.1 g of metenolone enanthate ($C_{27}H_{42}O_3$), add chloroform to make exactly 100 mL. Pipet 5 mL of this solution, add chloroform to make exactly 50 mL, and use this solution as the sample solution. Weigh accurately about 0.1 g of metenolone enanthate for assay, 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 3 mL each of the sample solution and the standard solution, and treat each solution as follows: add 10 mL of isoniazid TS, exactly measured, add methanol to make exactly 20 mL, and allow to stand for 60 minutes. Determine the absorbances, A_T and A_S , of the solutions from the sample solution and the standard solution, respectively, at 384 nm as directed under the Ultraviolet-visible Spectrophotometry, using a solution obtained by proceeding with 3 mL of chloroform as the blank.

$$\begin{aligned} \text{Amount (mg) of metenolone enanthate } (C_{27}H_{42}O_3) \\ = \text{amount (mg) of metenolone enanthate for assay} \\ \times \frac{A_T}{A_S} \end{aligned}$$

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