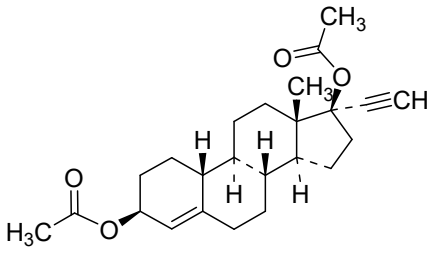
Ethynodiol Diacetate



$C_{24}H_{32}O_4$ Mol. Wt. 384.5

Ethynodiol Diacetate is 19-Norpregn-4-en-20-yne-3,17-diol, diacetate, (3β,17α).

Ethynodiol Diacetate contains not less than 97.0 per cent and not more than 102.0 per cent of $C_{24}H_{32}O_4$.

Description. A white, crystalline powder.

Identification

A. Determine by infrared absorption spectrophotometry (2.4.6). Compare the spectrum with that obtained with *ethynodiol diacetate IPRS* or with the reference spectrum of ethynodiol diacetate.

B. In the Assay, the principal peak in the chromatogram obtained with the test solution corresponds to the peak in the chromatogram obtained with the reference solution.

Tests

Optical rotation (2.4.22). -76.0° to -70.0°, determined on 1 per cent w/v solution in *chloroform.*

Related substances. Determine by liquid chromatography (2.4.14).

Test solution. Dissolve 0.25 g of the substance under examination in 50 ml of *acetonitrile,* with the aid of ultrasound and dilute to 100.0 ml with *water*. Dilute 5.0 ml of the solution to 50.0 m1 with the mobile phase.

Reference solution. Dissolve 125 mg of *ethynodiol diacetate IPRS* in 25 ml of *acetonitrile,* with the aid of ultrasound and dilute to 50.0 ml with *water*. Dilute 5.0 ml of the solution to 50.0 m1 with the mobile phase.

Chromatographic system
- a stainless steel column 15 cm × 4.6 mm, packed with phenyl group (5 µm) (Such as Adsorbosphere Phenyl),
- column temperature: 40°,
- mobile phase: a mixture of 59 volumes of *water* and 41 volumes of *acetonitrile*,
- flow rate: 2 ml per minute,
- spectrophotometer set at 200 nm,
- injection volume: 20 µl.

Name	Relative retention time
α-Ethynodiol diacetate	0.87
Ethynodiol diacetate (Retention time: about 18 minute)	1.0

Inject the reference solution. The test is not valid unless the tailing factor is not less than 0.75 and not more than 2.0 and the relative standard deviation for replicate injections is not more than 1.0 per cent.

Inject the test solution. The area of any peak corresponding to α-ethynodiol diacetate is not more than 1.5 per cent, the area of any other secondary peak is not more than 0.5 per cent and the sum of the areas of all the secondary peaks is not more than 2.0 per cent, calculated by area normalization.

Limit of Conjugated Diene. The absorbance of a 0.05 per cent w/v solution in *methanol* at the maximum at about 236 nm (2.4.7) is not more than 0.50.

Heavy metals (2.3.13). 1.0 g complies with the limit test for heavy metals, Method B (20 ppm).

Assay. Determine by liquid chromatography (2.4.14), as described under Related substances.

Inject the reference solution and the test solution.

Calculate the content of $C_{24}H_{32}O_4$.

Storage. Store protected from light and moisture, at a temperature not exceeding 30°.

2.4.26. Solubility. Very soluble in *chloroform*; freely soluble in *ether*; soluble in *ethanol*; sparingly soluble in fixed oils; insoluble in *water*.