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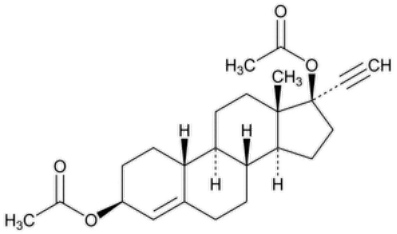
Ethynodiol Diacetate



$C_{24}H_{32}O_4$ 384.51

19-Norpregn-4-en-20-yne-3,17-diol, diacetate, (3β,17α)-;

19-Nor-17α-pregn-4-en-20-yne-3β,17-diol diacetate CAS RN®: 297-76-7; UNII: 62H10A1236.

DEFINITION

Ethynodiol Diacetate contains NLT 97.0% and NMT 102.0% of ethynodiol diacetate ($C_{24}H_{32}O_4$).

IDENTIFICATION

Change to read:

• **A.** ▲Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: **197K**▲ (CN 1-May-2020)

ASSAY

• **Procedure**

Mobile phase: Acetonitrile and water (41:59)

Standard stock solution: 2.5 mg/mL of USP Ethynodiol Diacetate RS, prepared as follows. Transfer a sufficient amount of USP Ethynodiol Diacetate RS into a suitable volumetric flask and dissolve, by sonication, in a volume of acetonitrile equivalent to 50% of the flask volume. Dilute with water to volume.

Standard solution: 0.25 mg/mL of USP Ethynodiol Diacetate RS in *Mobile phase* from *Standard stock solution*

Sample stock solution: 2.5 mg/mL of Ethynodiol Diacetate, prepared as follows. Transfer a sufficient amount of Ethynodiol Diacetate into a suitable volumetric flask and dissolve, by sonication, in a volume of acetonitrile equivalent to 50% of the flask volume. Dilute with water to volume.

Sample solution: 0.25 mg/mL of Ethynodiol Diacetate in *Mobile phase* from *Sample stock solution*

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: LC

Detector: UV 200 nm

Column: 4.6-mm × 15-cm; packing L11

Column temperature: 40°

Flow rate: 2 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

[Note—The retention time for ethynodiol diacetate is NLT 18 min.]

Suitability requirements

Tailing factor: 0.75–2.0

Relative standard deviation: NMT 0.7%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of ethynodiol diacetate ($C_{24}H_{32}O_4$) in the portion of Ethynodiol Diacetate taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

C_S = concentration of USP Ethynodiol Diacetate RS in the *Standard solution* (mg/mL)

C_U = concentration of Ethynodiol Diacetate in the *Sample solution* (mg/mL)

Acceptance criteria: 97.0%–102.0%

IMPURITIES

• **Procedure 1**

Mobile phase, Sample stock solution, Sample solution, Chromatographic system, and **System suitability:** Proceed as directed in the *Assay*.

Analysis

Sample: *Sample solution*

Calculate the area percentages of the individual impurities in the portion of Ethynodiol Diacetate taken:

$$\text{Result} = (r_U/r_T) \times 100$$

r_U = peak area of each individual peak between the solvent front and the ethynodiol diacetate peak

r_T = sum of the areas of all peaks appearing after the solvent front

Acceptance criteria: See *Table 1*.

Table 1

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
α-Ethynodiol diacetate	0.87	1.5
Ethynodiol diacetate	1.0	—
Any other individual impurity	—	0.5
Total impurities	—	2.0

• **Procedure 2: Limit of Conjugated Diene**

Sample solution: 0.5 mg/mL in methanol

Blank: Methanol

Instrumental conditions

(See *Ultraviolet-Visible Spectroscopy* ⟨857⟩.)

Mode: UV

Analytical wavelength: about 236 nm

Cell: 1 cm

Acceptance criteria: Absorbance is NMT 0.500.

SPECIFIC TESTS

• **Optical Rotation,** *Specific Rotation* ⟨781S⟩

Sample solution: 10 mg/mL in chloroform

Acceptance criteria: −70° to −76°

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed, light-resistant containers.

• **USP Reference Standards** ⟨11⟩

USP Ethynodiol Diacetate RS

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
ETHYNODIOL DIACETATE	Documentary Standards Support	SM52020 Small Molecules 5

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. Information currently unavailable
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