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Desogestrel and Ethinyl Estradiol Tablets

DEFINITION

Desogestrel and Ethinyl Estradiol Tablets contain NLT 90.0% and NMT 110.0% of the labeled amounts of desogestrel ($C_{22}H_{30}O$) and ethinyl estradiol ($C_{20}H_{24}O_2$).

IDENTIFICATION

• **A. Thin-Layer Chromatographic Identification Test ⟨201⟩**

Standard solution: 0.15 mg/mL of USP Desogestrel RS and 0.03 mg/mL of USP Ethinyl Estradiol RS in ether

Sample solution: Transfer a number of Tablets, equivalent to 1.5 mg desogestrel and 0.3 mg ethinyl estradiol, to a suitable container, add 50 mL of water, and sonicate until the Tablets disintegrate (if necessary, remove any coating with water before sonication). Place the sample in a separatory funnel, add 25 mL of ether, and shake well to extract the actives. Using a glass pipet, transfer the ether layer to a clean beaker, and evaporate to about 10 mL.

Chromatographic system

(See *Chromatography ⟨621⟩, Thin-Layer Chromatography*.)

Mode: TLC

Application volume: 30 µL

Developing solvent system: Chloroform and alcohol (96:4)

Spray reagent: Methanol and sulfuric acid (1:1)

Analysis: Proceed as directed in the chapter, and then air-dry. Spray the plate with the *Spray reagent*, place in an oven at 105° for about 5 min, and examine the plate.

Acceptance criteria: Meet the requirements

• **B.** The retention times of the major peaks of the *Sample solution* correspond to those of the *Standard solution*, as obtained in the *Assay*.

ASSAY

• **Procedure**

Buffer: 20 mM potassium phosphate buffer, pH 6.0

Mobile phase: Acetonitrile and *Buffer* (1:1)

Diluent: Acetonitrile and water (1:1)

Standard stock solution A: 0.3 mg/mL of USP Desogestrel RS in methanol

Standard stock solution B: 0.3 mg/mL of USP Ethinyl Estradiol RS in methanol

Standard solution: 0.6 µg/mL of USP Desogestrel RS and 0.12 µg/mL of USP Ethinyl Estradiol RS in *Diluent*, prepared by diluting appropriate aliquots of *Standard stock solution A* and *Standard stock solution B* with *Diluent*

Sample solution: Transfer 20 Tablets into a 200-mL volumetric flask. Add about 120 mL of *Diluent*, and shake for about 30 min. Dilute with *Diluent* to volume, and mix. Centrifuge a portion of the sample, and dilute with *Diluent* to obtain a solution nominally containing 0.6 µg/mL of desogestrel.

Chromatographic system

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

Mode: LC

Detectors

Desogestrel analysis: UV 210 nm

Ethinyl estradiol analysis: Spectrofluorometric detector, excitation at 285 nm and emission at 310 nm

Columns

Guard: 4.6-mm × 12.5-mm; packing L11

Analytical: 4.6-mm × 15-cm; packing L11

Flow rate: 2 mL/min

Injection volume: 200 µL

System suitability

Sample: *Standard solution*

[NOTE—The relative retention times for ethinyl estradiol and desogestrel are about 0.2 and 1.0, respectively.]

Suitability requirements

Tailing factor: NMT 2.0 for both ethinyl estradiol and desogestrel

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of desogestrel ($C_{22}H_{30}O$) and ethinyl estradiol ($C_{20}H_{24}O_2$) in the portion of Tablets taken:

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

r_U = peak response of the relevant analyte from the *Sample solution*

r_S = peak response of the relevant analyte from the *Standard solution*

C_S = concentration of the appropriate USP Reference Standard in the *Standard solution* (mg/mL)

C_U = nominal concentration of the relevant analyte in the *Sample solution* (mg/mL)

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

• **DISSOLUTION** ⟨711⟩

Test 1

Medium: 0.05% sodium lauryl sulfate with an assay content of NLT 95%; 500 mL

Apparatus 2: 50 rpm

Time: 30 min

Buffer: 20 mM potassium phosphate buffer, pH 6.0

Mobile phase: Acetonitrile and *Buffer* (1:1)

Standard stock solution A: 0.005 mg/mL of USP Desogestrel RS in *Medium* prepared as follows. Dissolve a sufficient quantity of USP Desogestrel RS in methanol to obtain a solution containing 0.25 mg/mL of USP Desogestrel RS. Dilute 1.0 mL of this solution with *Medium* to 50.0 mL.

Standard stock solution B: 0.005 mg/mL of USP Ethinyl Estradiol RS in *Medium* prepared as follows. Dissolve a sufficient quantity of USP Ethinyl Estradiol RS in methanol to obtain a solution containing 0.25 mg/mL of USP Ethinyl Estradiol RS. Dilute 1.0 mL of this solution with *Medium* to 50.0 mL.

Standard solution: 0.3 µg/mL of USP Desogestrel RS and 0.06 µg/mL of USP Ethinyl Estradiol RS in *Medium*, from *Standard stock solution A* and *Standard stock solution B*

Sample solution: Sample per *Dissolution* ⟨711⟩. Centrifuge a portion of the dissolution sample, and use the clear supernatant.

Chromatographic system

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

Mode: LC

Detectors

Desogestrel analysis: UV 210 nm

Ethinyl estradiol analysis: Spectrofluorometric detector, excitation at 285 nm and emission at 310 nm

Columns

Guard: 4.6-mm × 12.5-mm; packing L11

Analytical: 4.6-mm × 15-cm; packing L11

Flow rate: 2 mL/min

Injection volume: 200 µL

System suitability

Sample: *Standard solution*

[NOTE—The relative retention times for ethinyl estradiol and for desogestrel are about 0.2 and 1.0, respectively.]

Suitability requirements

Relative standard deviation: NMT 3.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Determine the amounts of desogestrel ($C_{22}H_{30}O$) and ethinyl estradiol ($C_{20}H_{24}O_2$) dissolved:

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

r_U = peak response of the relevant analyte from the *Sample solution*

r_S = peak response of the relevant analyte from the *Standard solution*

C_S = concentration of the appropriate USP Reference Standard in the *Standard solution* (mg/mL)

L = label claim (mg/Tablet)

V = volume of medium, 500 mL

Tolerances: NLT 80% (*Q*) of each of the labeled amounts of desogestrel ($C_{22}H_{30}O$) and ethinyl estradiol ($C_{20}H_{24}O_2$) is dissolved.

Test 2: If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 2*.

Medium: 0.3% sodium lauryl sulfate; 500 mL

Apparatus 2: 100 rpm

Time: 30 min

Analysis: Determine the amounts of desogestrel ($C_{22}H_{30}O$) and ethinyl estradiol ($C_{20}H_{24}O_2$) dissolved by the chromatographic method used in *Test 1*.

Tolerances: NLT 80% (*Q*) of each of the labeled amounts of desogestrel ($C_{22}H_{30}O$) and ethinyl estradiol ($C_{20}H_{24}O_2$) is dissolved.

• **Uniformity of Dosage Units ⟨905⟩:** Meet the requirements for *Content Uniformity* for both desogestrel and ethinyl estradiol

ADDITIONAL REQUIREMENTS

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

• **Labeling:** When more than one *Dissolution* test is given, the labeling states the *Dissolution* test used only if *Test 1* is not used.

• **USP Reference Standards ⟨11⟩**

USP Desogestrel RS

USP Ethinyl Estradiol RS

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

Topic/Question	Contact	Expert Committee
DESOGESTREL AND ETHINYL ESTRADIOL TABLETS	Documentary Standards Support	SM52020 Small Molecules 5

Chromatographic Database Information: Chromatographic Database

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