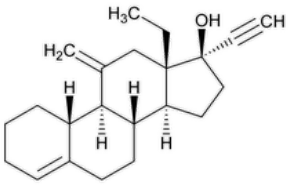


Status: Currently Official on 14-Feb-2025
Official Date: Official as of 01-May-2020
Document Type: USP Monographs
DocId: GUID-4D35F093-ED87-4B0B-8BDA-993ACFC9E49A_2_en-US
DOI: https://doi.org/10.31003/USPNF_M22960_02_01
DOI Ref: 5j06t

Desogestrel



$C_{22}H_{30}O$ 310.47

18,19-Dinorpregn-4-en-20-yn-17-ol, 13-ethyl-11-methylene-, (17α)-;

13-Ethyl-11-methylene-18,19-dinor-17α-pregn-4-en-20-yn-17-ol CAS RN®: 54024-22-5.

DEFINITION

Desogestrel contains NLT 98.0% and NMT 102.0% of desogestrel ($C_{22}H_{30}O$), calculated on the dried basis.

IDENTIFICATION

Change to read:

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

• **B.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY

• **Procedure**

Mobile phase: Acetonitrile and water (73:27)

System suitability solution: 400 µg/mL of USP Desogestrel RS and 0.4 µg/mL each of USP Desogestrel Related Compound A RS and USP Desogestrel Related Compound D RS prepared as follows. Dissolve the material in acetonitrile equivalent to 50% of the volume of a suitable volumetric flask and dilute with water to volume.

Standard solution: 400 µg/mL of USP Desogestrel RS prepared as follows. Dissolve the material in acetonitrile equivalent to 50% of the volume of a suitable volumetric flask and dilute with water to volume.

Sample solution: 400 µg/mL of Desogestrel prepared as directed in the *Standard solution*

Chromatographic system

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

Mode: LC

Detector: UV 205 nm

Column: 4.6-mm × 25-cm; 5-µm packing L1

Column temperature: 50°

Flow rate: 1 mL/min

Injection volume: 15 µL

Run time: 1.5 times the retention time of desogestrel

System suitability

Samples: *System suitability solution* and *Standard solution*

[Note—See *Table 1* for relative retention times.]

Suitability requirements

Resolution: NLT 1.3 between desogestrel and desogestrel related compound A, *System suitability solution*

Tailing factor: NMT 2.0, *Standard solution*

Relative standard deviation: NMT 0.73%, *Standard solution*

Peak-to-valley ratio: NLT 2.0 between desogestrel and desogestrel related compound D, *System suitability solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of desogestrel ($C_{22}H_{30}O$) in the portion of Desogestrel taken:

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

r_U = peak response of desogestrel from the *Sample solution*

r_S = peak response of desogestrel from the *Standard solution*

C_S = concentration of USP Desogestrel RS in the *Standard solution* (µg/mL)

C_U = concentration of Desogestrel in the *Sample solution* (µg/mL)

Acceptance criteria: 98.0%–102.0% on the dried basis

IMPURITIES

- **Residue on Ignition ⟨281⟩:** NMT 0.1%
- **Organic Impurities**

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

Diluent: Acetonitrile and water (50:50)

Standard stock solution: 0.04 mg/mL each of USP Desogestrel RS, USP Desogestrel Related Compound B RS, and USP Desogestrel Related Compound C RS, and 0.08 mg/mL each of USP Desogestrel Related Compound A RS and USP Desogestrel Related Compound D RS prepared as follows. Dissolve the materials in acetonitrile equivalent to 50% of the volume of a suitable volumetric flask and dilute with water to volume.

Standard solution: 0.4 µg/mL each of USP Desogestrel RS, USP Desogestrel Related Compound B RS, and USP Desogestrel Related Compound C RS, and 0.8 µg/mL each of USP Desogestrel Related Compound A RS and USP Desogestrel Related Compound D RS in *Diluent*, from *Standard stock solution*

System suitability

Samples: *System suitability solution* and *Standard solution*

[Note—See *Table 1* for relative retention times.]

Suitability requirements

Resolution: NLT 1.3 between desogestrel and desogestrel related compound A, *System suitability solution*

Relative standard deviation: NMT 5.0% for each corresponding peak present in the *Standard solution*

Peak-to-valley ratio: NLT 2.0 between desogestrel and desogestrel related compound D, *System suitability solution*

Analysis

Samples: *Sample solution* and *Standard solution*

Calculate the percentage of desogestrel related compound A, desogestrel related compound B, desogestrel related compound C, or desogestrel related compound D in the portion of Desogestrel taken:

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

r_U = peak response of desogestrel related compound A, desogestrel related compound B, desogestrel related compound C, or desogestrel related compound D from the *Sample solution*

r_S = peak response of the corresponding Reference Standard from the *Standard solution*

C_S = concentration of USP Desogestrel Related Compound A RS, USP Desogestrel Related Compound B RS, USP Desogestrel Related Compound C RS, or USP Desogestrel Related Compound D RS in the *Standard solution* (µg/mL)

C_U = concentration of Desogestrel in the *Sample solution* (µg/mL)

Calculate the percentage of 11-methylene lynestrenol and any other individual unspecified impurity in the portion of Desogestrel taken:

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

r_U = peak response of 11-methylene lynestrenol or any other individual unspecified impurity from the *Sample solution*

r_S = peak response of desogestrel from the *Standard solution*

C_S = concentration of USP Desogestrel RS in the *Standard solution* (µg/mL)

C_U = concentration of Desogestrel in the *Sample solution* (µg/mL)

Acceptance criteria: See *Table 1*. Disregard peaks less than 0.05%.

Table 1

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
Desogestrel related compound B	0.16	0.10
Desogestrel related compound C	0.19	0.1
11-Methylene lynestrenol[a]	0.71	0.2
Desogestrel related compound A	0.96	0.2
Desogestrel	1.0	—
Desogestrel related compound D	1.06	0.2
Any other individual unspecified impurity	—	0.10
Total impurities	—	0.5

[a] 11-Methylene-19-nor-17α-pregn-4-en-20-yn-17-ol.

SPECIFIC TESTS

- **Optical Rotation** ⟨781S⟩, *Procedures, Specific Rotation*

  Sample solution: 10 mg/mL of Desogestrel in absolute alcohol

  Acceptance criteria: +53° to +57° (dried substance)

- **Loss on Drying** ⟨731⟩

  Analysis: Dry under vacuum at a pressure not exceeding 15 mm of mercury at room temperature to constant weight.

  Acceptance criteria: NMT 0.5%

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight, light-resistant containers, and store at controlled room temperature.
- **USP Reference Standards** ⟨11⟩

USP Desogestrel RS

USP Desogestrel Related Compound A RS

13-Ethyl-11-methylene-18,19-dinor-5α,17α-pregn-3-en-20-yn-17-ol;
Desogestrel Δ3-isomer.

$C_{22}H_{30}O$ 310.47

USP Desogestrel Related Compound B RS

13-Ethyl-3-hydroxy-11-methylene-18,19-dinor-17α-pregn-4-en-20-yn-17-ol.

$C_{22}H_{30}O_2$ 326.48

USP Desogestrel Related Compound C RS

13-Ethyl-11-methylene-18,19-dinor-17α-pregn-4-en-20-yn-17-ol-3-one.

$C_{22}H_{28}O_2$ 324.46

USP Desogestrel Related Compound D RS

13-Ethyl-11-methylenegon-4-en-17-one.

$C_{20}H_{28}O$ 284.44

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

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

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 41(6)

Current DocID: GUID-4D35F093-ED87-4B0B-8BDA-993ACFC9E49A_2_en-US
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