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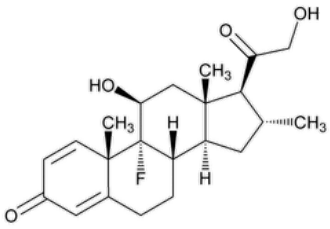
Desoximetasone



$C_{22}H_{29}FO_4$ 376.46

Pregna-1,4-diene-3,20-dione, 9-fluoro-11,21-dihydroxy-16-methyl-, (11β,16α)-;

9-Fluoro-11β,21-dihydroxy-16α-methylpregna-1,4-diene-3,20-dione CAS RN®: 382-67-2; UNII: 4E07GXB7AU.

DEFINITION

Desoximetasone contains NLT 97.0% and NMT 103.0% of desoximetasone ($C_{22}H_{29}FO_4$), calculated on the dried basis.

IDENTIFICATION

Change to read:

• **A.** ▲Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: **197K**▲ (CN 1-May-2020) ▲or ⟨197A⟩▲ (USP 1-Dec-2019)

Delete the following:

▲• **B. Thin-Layer Chromatography**

Diluent: Chloroform and alcohol (3:1)

Standard solution: 10 mg/mL of USP Desoximetasone RS in *Diluent*

Sample solution: 10 mg/mL of desoximetasone in *Diluent*

Chromatographic system

Mode: TLC

Adsorbent: 0.25-mm layer of chromatographic silica gel mixture

Application volume: 20 µL

Developing solvent system: Chloroform and ethyl acetate (1:1)

Spray reagent: 200 mg/mL of *p*-toluenesulfonic acid in alcohol

Analysis

Samples: *Standard solution* and *Sample solution*

Allow the spots to dry, and develop the chromatogram in a saturated chamber containing the *Developing solvent system*. Allow the solvent front to move 10 cm beyond the application point. After drying, examine the plate under UV light at 254 nm. Spray the dried plate with *Spray reagent*.

Acceptance criteria: The major spot from the *Sample solution* corresponds in R_F value (0.25) and appearance to that obtained from the *Standard solution*.▲ (USP 1-Dec-2019)

Add the following:

▲• **B.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.▲ (USP 1-Dec-2019)

ASSAY

Change to read:

• **Procedure**

▲**Buffer:** 1.36 g/L of monobasic potassium phosphate

Solution A: Acetonitrile, tetrahydrofuran, and *Buffer* (23.3:1.5:75). Adjust with 10% phosphoric acid to a pH of 4.0.

Solution B: Acetonitrile and *Buffer* (49.6:50). Adjust with 10% phosphoric acid to a pH of 4.0.

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	95	5
25	5	95
30	5	95
31	95	5
35	95	5

System suitability stock solution: 0.12 mg/mL of USP Desoximetasone Related Compound A RS prepared as follows. Transfer a suitable amount of USP Desoximetasone Related Compound A RS to a suitable volumetric flask. Add about 1 mL of tetrahydrofuran and swirl to dissolve. Dilute with *Solution A* to volume. Sonication may be used to aid dissolution.

System suitability solution: 1 mg/mL of USP Desoximetasone RS and 1.2 µg/mL of USP Desoximetasone Related Compound A RS prepared as follows. Transfer a suitable amount of USP Desoximetasone RS to a suitable volumetric flask. Add about 1 mL of tetrahydrofuran and swirl to dissolve. Add 1% of the flask volume of the *System suitability stock solution* and dilute with *Solution A* to volume. Sonication may be used to aid dissolution.

Standard solution: 0.1 mg/mL of USP Desoximetasone RS prepared as follows. Transfer a suitable amount of USP Desoximetasone RS to a suitable volumetric flask. Add about 1 mL of tetrahydrofuran and swirl to dissolve. Dilute with *Solution A* to volume. Sonication may be used to aid dissolution.

Sample solution: 0.1 mg/mL of Desoximetasone prepared as follows. Transfer a suitable amount of Desoximetasone to a suitable volumetric flask. Add about 1 mL of tetrahydrofuran and swirl to dissolve. Dilute with *Solution A* to volume. Sonication may be used to aid dissolution.

Chromatographic system

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

Mode: LC

Detector: UV 240 nm

Column: 4.6-mm × 15-cm; 3.5-µm packing L1

Flow rate: 1.2 mL/min

Injection volume: 20 µL

System suitability

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

[NOTE—See *Table 2* for the relative retention times.]

Suitability requirements

Resolution: NLT 1.5 between desoximetasone and desoximetasone related compound A, *System suitability solution*

Relative standard deviation: NMT 1.10%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of desoximetasone ($C_{22}H_{29}FO_4$) in the portion of Desoximetasone 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 Desoximetasone RS in the *Standard solution* (mg/mL)

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

▲ (USP 1-Dec-2019)

Acceptance criteria: 97.0%–103.0% on the dried basis

IMPURITIES

• **Residue on Ignition ⟨281⟩:** NMT 0.2%

Add the following:

▲• **Organic Impurities**

Buffer, Solution A, Solution B, Mobile phase, System suitability solution, and **Chromatographic system:** Proceed as directed in the *Assay*.

Standard solution: 0.001 mg/mL of USP Desoximetasone RS prepared as follows. Transfer a suitable amount of USP Desoximetasone RS to a suitable volumetric flask. Add about 1 mL of tetrahydrofuran and swirl to dissolve. Dilute with *Solution A* to volume. Sonication may be used to aid dissolution.

Sensitivity solution: 0.2 µg/mL of USP Desoximetasone RS in *Solution A* from the *Standard solution*

Sample solution: 1 mg/mL of Desoximetasone prepared as follows. Transfer a suitable amount of Desoximetasone to a suitable volumetric flask. Add about 1 mL of tetrahydrofuran and swirl to dissolve. Dilute with *Solution A* to volume. Sonication may be used to aid dissolution.

System suitability

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

[Note—See *Table 2* for the relative retention times.]

Suitability requirements

Resolution: NLT 1.5 between desoximetasone and desoximetasone related compound A, *System suitability solution*

Relative standard deviation: NMT 5.0%, *Standard solution*

Signal-to-noise ratio: NLT 10, *Sensitivity solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of each individual impurity in the portion of Desoximetasone taken:

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

r_U = peak response of each individual impurity from the *Sample solution*

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

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

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

Acceptance criteria: See *Table 2*. The reporting threshold is 0.02%.

Table 2

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
Desoximetasone diacetal[a]	0.88	0.15
Desoximetasone	1.00	—
Desoximetasone related compound A	1.10	0.15
Desoximetasone acid[b]	1.18	0.15
Any individual unspecified impurity	—	0.10
Total impurities	—	0.5▲ (USP 1-Dec-2019)

[a] 9-Fluoro-11β,21,21-trihydroxy-16α-methylpregna-1,4-diene-3,20-dione.

[b] 9-Fluoro-11β-hydroxy-16α-methyl-3-oxoandrosta-1,4-diene-17β-carboxylic acid.

SPECIFIC TESTS

Delete the following:

▲• ***Melting Range or Temperature*** ⟨741⟩**:** 206°–218°; but the range between beginning and end of melting does not exceed 4°▲ (USP 1-Dec-2019)

Change to read:

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

Sample solution: 5 mg/mL of Desoximetasone in ▲alcohol▲ (USP 1-Dec-2019)

Acceptance criteria: ▲+123° to +129°▲ (USP 1-Dec-2019)

• **Loss on Drying** ⟨731⟩

Analysis: Dry at 105° to constant weight.

Acceptance criteria: NMT 1.0%

ADDITIONAL REQUIREMENTS

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

Change to read:

• **USP Reference Standards** ⟨11⟩

USP Desoximetasone RS

▲ USP Desoximetasone Related Compound A RS

Dihydrodesoximetasone;
9-Fluoro-11β,21-dihydroxy-16α-methylpregna-4-ene-3,20-dione.

$C_{22}H_{31}FO_4$ 378.48▲ (USP 1-Dec-2019)

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

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

Chromatographic Database Information: Chromatographic Database

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