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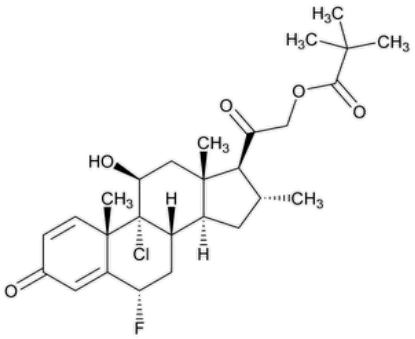
Clocortolone Pivalate



$C_{27}H_{36}ClFO_5$ 495.03

Pregna-1,4-diene-3,20-dione, 9-chloro-21-(2,2-dimethyl-1-oxopropoxy)-6-fluoro-11-hydroxy-16-methyl-, (6α,11β,16α)-;

9-Chloro-6α-fluoro-11β,21-dihydroxy-16α-methylpregna-1,4-diene-3,20-dione 21-pivalate CAS RN®: 34097-16-0; UNII: QBL8IZH14X.

DEFINITION

Change to read:

Clocortolone Pivalate contains ▲NLT 98.0% and NMT 102.0%▲ (USP 1-Aug-2021) of clocortolone pivalate ($C_{27}H_{36}ClFO_5$), calculated on the dried basis.

IDENTIFICATION

Change to read:

• **A.** **Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*:** 197M ▲or 197A▲ (USP 1-Aug-2021)

Change to read:

• **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-Aug-2021)

ASSAY

Change to read:

• **Procedure**

▲**Mobile phase:** Acetonitrile and water (60:40)

Standard solution: 1 mg/mL of USP Clocortolone Pivalate RS in acetonitrile

Sample solution: 1 mg/mL of Clocortolone Pivalate in acetonitrile

Chromatographic system

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

Mode: LC

Detector: UV 240 nm

Column: 4.0-mm × 25-cm; 5-µm packing L1

Column temperature: 30°

Flow rate: 1 mL/min

Injection volume: 10 µL

Run time: NLT 2 times the retention time of clocortolone pivalate

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 1.5

Relative standard deviation: NMT 0.73%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of clocortolone pivalate ($C_{27}H_{36}ClFO_5$) in the portion of Clocortolone Pivalate taken:

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

r_U = peak response of clocortolone pivalate from the *Sample solution*

r_S = peak response of clocortolone pivalate from the *Standard solution*

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

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

Acceptance criteria: 98.0%–102.0% on the dried basis ▲(USP 1-Aug-2021)

IMPURITIES

• **Residue on Ignition ⟨281⟩**

Sample: 100 mg

Acceptance criteria: NMT 0.2%

Change to read:

• **Organic Impurities**

▲**Mobile phase:** Prepare as directed in the *Assay*.

System suitability solution: 0.5 mg/mL each of USP Clocortolone RS and USP Clocortolone Pivalate RS in acetonitrile

Sensitivity solution: 0.00125 mg/mL of USP Clocortolone Pivalate RS in acetonitrile

Standard solution: 0.0125 mg/mL of USP Clocortolone Pivalate RS in acetonitrile

Sample solution: 2.5 mg/mL of Clocortolone Pivalate in acetonitrile

Chromatographic system: Proceed as directed in the *Assay* except for the following.

Injection volume: 20 µL

Run time: NLT 3.3 times the retention time of clocortolone pivalate

System suitability

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

Suitability requirements

Resolution: NLT 1.5 between clocortolone and clocortolone pivalate, *System suitability solution*

Relative standard deviation: NMT 2.0%, *Standard solution*

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

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of clocortolone or any unspecified impurity in the portion of Clocortolone Pivalate taken:

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

r_U = peak response of clocortolone or any unspecified impurity from the *Sample solution*

r_S = peak response of clocortolone pivalate from the *Standard solution*

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

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

Acceptance criteria: See *Table 1*. The reporting threshold is 0.05%.

Table 1

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
Clocortolone	0.25	0.1
Clocortolone pivalate	1.0	—
Any unspecified impurity	—	0.10
Total impurities	—	0.5▲ (USP 1-Aug-2021)

SPECIFIC TESTS

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

Sample solution: 40 mg/mL, in chloroform

Acceptance criteria: +125° to +135°

• **Loss on Drying** ⟨731⟩

Analysis: Dry at 105° for 3 h.

Acceptance criteria: NMT 1.0%

Delete the following:

▲• **Color and Clarity of Solution:**

Sample solution: 1-in-100 solution of clocortolone pivalate in chloroform

Acceptance criteria: Solution is clear and practically colorless.▲ (USP 1-Aug-2021)

ADDITIONAL REQUIREMENTS

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

Change to read:

• **USP Reference Standards** ⟨11⟩

▲ USP Clocortolone RS▲ (USP 1-Aug-2021)

USP Clocortolone Pivalate RS

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

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

Chromatographic Database Information: Chromatographic Database

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