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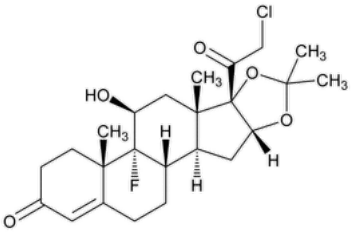
Halcinonide



$C_{24}H_{32}ClFO_5$ 454.96

Pregn-4-ene-3,20-dione, 21-chloro-9-fluoro-11-hydroxy-16,17-[(1-methylethylidene)bis(oxy)]-, (11β,16α)-;

21-Chloro-9-fluoro-11β,16α,17-trihydroxypregn-4-ene-3,20-dione cyclic 16,17-acetal with acetone CAS RN®: 3093-35-4; UNII: SI86V6QNEG.

DEFINITION

Halcinonide contains NLT 97.0% and NMT 102.0% of halcinonide ($C_{24}H_{32}ClFO_5$).

IDENTIFICATION

Change to read:

• **A.** ▲Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: **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 (50:50)

Diluent: Acetonitrile and water (2:1)

Standard solution: 0.3 mg/mL of USP Halcinonide RS in *Diluent*

Sample solution: 0.3 mg/mL of Halcinonide in *Diluent*. [Note—Sonication may be needed to aid in the dissolution of the *Standard solution* and *Sample solution*.]

Chromatographic system

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

Mode: LC

Detector: UV 240 nm

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

Flow rate: 2 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 1.5

Relative standard deviation: NMT 0.85% for six replicate injections

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of halcinonide ($C_{24}H_{32}ClFO_5$) in the portion of Halcinonide 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 Halcinonide RS in the *Standard solution* (mg/mL)

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

Acceptance criteria: 97.0%–102.0%

IMPURITIES

- **Residue on Ignition ⟨281⟩:** NMT 0.2%
- **Organic Impurities**

Solution A: 10 mM of ammonium acetate in water

Solution B: Acetonitrile

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	90	10
20.0	10	90
22.0	10	90
22.1	90	10
25.0	90	10

Diluent 1: Acetonitrile and water (60:30)

Diluent 2: Acetonitrile and water (10:90)

Standard stock solution: 0.2 mg/mL of USP Halcinonide RS in *Diluent 1*

Standard solution: 0.1 µg/mL of USP Halcinonide RS from *Standard stock solution* in *Diluent 2*

Sample stock solution: 0.2 mg/mL of Halcinonide in *Diluent 1*

Sample solution: 0.1 mg/mL of Halcinonide from *Sample stock solution* in *Diluent 2*

Chromatographic system

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

Mode: LC

Detector: UV 240 nm

Column: 2.1-mm × 15-cm; 1.7-µm packing L1

Flow rate: 0.3 mL/min

Injection volume: 10 µL

System suitability

Sample: *Standard solution*

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

Suitability requirements

Relative standard deviation: NMT 2.8%

Signal-to-noise ratio: NLT 50

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of any individual unspecified impurity in the portion of Halcinonide taken:

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

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

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

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

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

Acceptance criteria: See *Table 2*.

Table 2

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
Dihydrotriamcinolone[a]	0.5	—
Halcinonide	1.0	—
Any individual unspecified impurity	—	0.10
Total impurities	—	3.0

[a] 9-Fluoro-11β,16α,17,21-tetrahydroxypregna-4-ene-3,20-dione.

SPECIFIC TESTS

- **Optical Rotation,** *Specific Rotation*⟨781S⟩
  Sample solution: 20 mg/mL in chloroform
  Acceptance criteria: +150° to +160°
- **Loss on Drying** ⟨731⟩
  Analysis: Dry under vacuum at 100° for 3 h.
  Acceptance criteria: NMT 1.0%

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in well-closed containers.
- **USP Reference Standards** ⟨11⟩
  USP Halcinonide RS

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

Topic/Question	Contact	Expert Committee
HALCINONIDE	Documentary Standards Support	SM22020 Small Molecules 2
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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