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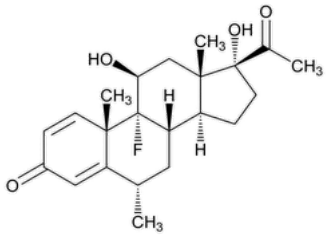
Fluorometholone



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

Pregna-1,4-diene-3,20-dione, 9-fluoro-11,17-dihydroxy-6-methyl-, (6α,11β)-;

9-Fluoro-11β,17-dihydroxy-6α-methylpregna-1,4-diene-3,20-dione CAS RN®: 426-13-1; UNII: SV0CSG527L.

DEFINITION

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

IDENTIFICATION

Change to read:

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

• **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: Mix 425 g (538 mL) of methanol and 400 g (400 mL) of water. Adjust with phosphoric acid to a pH of 2.4.

Standard stock solution: 0.25 mg/mL of USP Fluorometholone RS prepared as follows. Transfer a suitable amount of USP Fluorometholone RS to a suitable volumetric flask and dissolve in 2% of the final volume of tetrahydrofuran. Dilute with *Mobile phase* to volume.

Standard solution: 50 µg/mL of USP Fluorometholone RS in *Mobile phase* from *Standard stock solution*

Sample stock solution: 0.25 mg/mL of Fluorometholone prepared as follows. Transfer a suitable amount of Fluorometholone to a suitable volumetric flask and dissolve in 2% of the final volume of tetrahydrofuran. Dilute with *Mobile phase* to volume.

Sample solution: 50 µg/mL of Fluorometholone in *Mobile phase* from *Sample stock 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: 0.6 mL/min

Injection volume: 20 µL

Run time: NLT 1.6 times the retention time of fluorometholone

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 1.3

Relative standard deviation: NMT 0.73%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of fluorometholone ($C_{22}H_{29}FO_4$) in the portion of Fluorometholone taken:

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

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

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

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

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

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

IMPURITIES

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

• **Organic Impurities**

Mobile phase: Proceed as directed in the *Assay*.

System suitability stock solution: 0.5 mg/mL each of USP Fluorometholone RS and USP Fluorometholone Related Compound A RS prepared as follows. Transfer a suitable amount of USP Fluorometholone RS and USP Fluorometholone Related Compound A RS to a suitable volumetric flask and dissolve in 4% of the final volume of tetrahydrofuran. Dilute with *Mobile phase* to volume.

System suitability solution: 0.005 mg/mL each of USP Fluorometholone RS and USP Fluorometholone Related Compound A RS in *Mobile phase* from *System suitability stock solution*

Standard stock solution: 0.5 mg/mL of USP Fluorometholone RS prepared as follows. Transfer a suitable amount of USP Fluorometholone RS to a suitable volumetric flask and dissolve in 4% of the final volume of tetrahydrofuran. Dilute with *Mobile phase* to volume.

Standard solution: 0.5 µg/mL of USP Fluorometholone RS in *Mobile phase* from *Standard stock solution*

Sample solution: 0.5 mg/mL of Fluorometholone prepared as follows. Transfer a suitable amount of Fluorometholone to a suitable volumetric flask and dissolve in 4% of the final volume of tetrahydrofuran. Dilute with *Mobile phase* to volume.

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

Run time: NLT 2.1 times the retention time of fluorometholone

System suitability

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

Suitability requirements

Resolution: NLT 3 between fluorometholone and fluorometholone related compound A, *System suitability solution*

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

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of each impurity in the portion of Fluorometholone taken:

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

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

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

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

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

Acceptance criteria: See *Table 1*. Disregard any peaks below 0.02%.

Table 1

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
Fluorometholone	1.0	—
Fluorometholone related compound A	1.2	0.5
Individual unspecified impurities	—	0.10
Total impurities	—	1.0

SPECIFIC TESTS

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

Sample solution: 10 mg/mL in dimethyl sulfoxide

Acceptance criteria: +62° to +70°

• **Loss on Drying ⟨731⟩**

Analysis: Dry under vacuum at 60° for 3 h.

Acceptance criteria: NMT 1.0%

ADDITIONAL REQUIREMENTS

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

USP Fluorometholone RS

USP Fluorometholone Related Compound A RS

11β,17α-Dihydroxy-6α-methylpregna-1,4-diene-3,20-dione.

$C_{22}H_{30}O_4$ 358.47

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

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

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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