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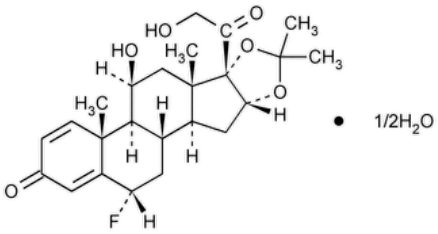
Flunisolide



$C_{24}H_{31}FO_6 \cdot ½H_2O$ 443.51

Pregna-1,4-diene-3,20-dione, 6-fluoro-11,21-dihydroxy-16,17-[(1-methylethylidene)bis(oxy)]-, hemihydrate, (6α,11β,16α)-.

6α-Fluoro-11β,16α,17,21-tetrahydroxypregna-1,4-diene-3,20-dione cyclic 16,17-acetal with acetone, hemihydrate CAS RN®: 77326-96-6; UNII: QK4DYS664X.

Anhydrous 434.51 CAS RN®: 3385-03-3.

» Flunisolide contains not less than 97.0 percent and not more than 102.0 percent of $C_{24}H_{31}FO_6$, calculated on the anhydrous basis.

Packaging and storage—Preserve in well-closed containers.

USP Reference standards ⟨11⟩—

USP Flunisolide RS

Identification—

Change to read:

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

Change to read:

B: ▲*Spectroscopic Identification Tests ⟨197⟩, Ultraviolet-Visible Spectroscopy: 197U*▲ *(CN 1-May-2020)* —

Solution: 10 µg per mL.

Medium: methanol.

Specific rotation ⟨781S⟩: between +103° and +111°.

Test solution: 10 mg per mL, in chloroform.

Loss on drying ⟨731⟩—Dry it in vacuum at 60° for 3 hours: it loses not more than 1.0% of its weight.

Water Determination, *Method I* ⟨921⟩ —The anhydrous form contains not more than 1.0%. The hemihydrate form contains between 1.8% and 2.5% (determined on a dried specimen).

Residue on ignition ⟨281⟩: not more than 0.1% from 250 mg.

Chromatographic purity—

Standard solutions—Prepare a solution of USP Flunisolide RS in acetone to contain 10 mg per mL (*Standard solution A*). Dilute 1 mL of *Standard solution A* with acetone to 100 mL (*Standard solution B*).

Test preparation—Prepare a solution of Flunisolide in acetone to contain 10 mg per mL.

Procedure—Apply 10-µL volumes of *Standard solution A*, *Standard solution B*, and the *Test preparation* to a suitable thin-layer chromatographic plate (see *Chromatography* ⟨621⟩) coated with a 0.25-mm layer of chromatographic silica gel mixture. Place the plate in a suitable chromatographic chamber previously equilibrated with a mixture of toluene and alcohol (90:10), seal the chamber, and develop the chromatogram until the solvent front has moved three-fourths of the length of the plate. Remove the plate, allow the solvent to evaporate, and examine the plate under short-wavelength UV light: the R_F value of the principal spot obtained from the *Test preparation* corresponds to that obtained from *Standard solution A*. No secondary spot exhibits an intensity greater than that of the principal spot from *Standard solution B*.

Assay—

Mobile phase—Prepare a suitable degassed solution of water and acetonitrile (3:2) such that at an approximate flow rate of 1.6 mL per minute, the retention time of Flunisolide is about 6 minutes.

Standard preparation—Dissolve an accurately weighed quantity of USP Flunisolide RS in *Mobile phase* to obtain a solution having a known concentration of about 0.2 mg per mL.

Assay preparation—Using 20 mg of Flunisolide, accurately weighed, proceed as directed for *Standard preparation*.

Chromatographic system (see *Chromatography ⟨621⟩*)—The liquid chromatograph is equipped with a 254-nm detector and a 4-mm × 25-cm column that contains 5- to 10-µm packing L7. The flow rate is about 1.6 mL per minute. Chromatograph the *Standard preparation*, and record the peak response as directed for *Procedure:* the column efficiency is not less than 2700 theoretical plates; the tailing factor for the flunisolide peak is not more than 1.7; and the relative standard deviation for replicate injections is not more than 1.0%.

Procedure—Separately inject equal volumes (between 15 µL and 30 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the quantity, in mg, of $C_{24}H_{31}FO_6$ in the portion of Flunisolide taken by the formula:

$$(434.51/443.51)100C(r_U/r_S)$$

in which 434.51 and 443.51 are the molecular weights of $C_{24}H_{31}FO_6$ and $C_{24}H_{31}FO_6 \cdot ½H_2O$, respectively; C is the concentration, in mg per mL, of USP Flunisolide RS in the *Standard preparation;* and r_U and r_S are the peak responses obtained from the *Assay preparation* and the *Standard preparation*, respectively.

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

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

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. Information currently unavailable

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