Dexamethasone Tablets

> Dexamethasone Tablets contain not less than 90.0 percent and not more than 110.0 percent of the labeled amount of C$_{22}$H$_{29}$FO$_5$.

Packaging and storage—Preserve in well-closed containers.

USP Reference standards (11)—
USP Dexamethasone RS

Identification—Evaporate 10 mL of the methanol extract of Tablets obtained as directed under Assay preparation in the Assay on a steam bath just to dryness, and dissolve the residue in 1 mL of chloroform. Apply 10 µL of this solution and 20 µL of a solution of Dexamethasone RS in chloroform containing 50 µg per mL on a thin-layer chromatographic plate (see System Suitability under Chromatography (621)) coated with a 0.25-mm layer of chromatographic silica gel mixture. Develop the chromatogram in Solvent A as directed under Single-Steroid Assay (511). Mark the solvent front, and locate the spots on the plate by visualizing under short-wavelength UV light: the RF value of the principal spot obtained from the solution under test corresponds to that obtained from the Standard solution.

Dissolution (711)—

Medium: dilute hydrochloric acid (1 in 100); 500 mL.

Apparatus 1: 100 rpm.

Time: 45 minutes.

Standard solution—Prepare as directed for Standard Preparation under Assay for Steroids (351), using USP Dexamethasone RS.

Procedure—Extract a filtered aliquot of Dissolution Medium, equivalent to about 200 µg of dexamethasone, with three 15-mL portions of chloroform. Evaporate the combined chloroform extracts on a steam bath just to dryness, cool, and dissolve the residue in 20 mL of alcohol. Proceed as directed for Procedure under Assay for Steroids (351), except to allow to stand in the dark for 45 minutes. Calculate the portion, in mg, of C$_{22}$H$_{29}$FO$_5$ dissolved by the formula:

$$10(C / V)(A_0 / A_t)$$

in which V is the volume, in mL, of the aliquot extracted with chloroform.

Tolerances—Not less than 70% (Q) of the labeled amount of C$_{22}$H$_{29}$FO$_5$ is dissolved in 45 minutes.

Uniformity of dosage units (905): meet the requirements.

Procedure for content uniformity—

Standard solution—Prepare as directed for Standard Preparation under Assay for Steroids (351), using USP Dexamethasone RS.

Test solution—Place 1 Tablet in a separator with 15 mL of water, and swirl to disintegrate the Tablet completely. Extract with four 10-mL portions of chloroform, filtering each portion through chloroform-washed cotton into a 50-mL volumetric flask, add chloroform to volume, and mix. Pipet a volume of this solution, equivalent to about 200 µg of dexamethasone into a glass-stoppered, 50-mL conical flask, evaporate the chloroform on a steam bath just to dryness, cool, and dissolve the residue in 20.0 mL of alcohol. Use this where Assay Preparation is specified in the Procedure.

Procedure—Proceed as directed for Procedure under Assay for Steroids (351), except to allow to stand in the dark for 45 minutes. Calculate the quantity, in mg, of total steroids, as C$_{22}$H$_{29}$FO$_5$, in the Tablet by the formula:

$$(C / V)(A_0 / A_t)$$

in which V is the volume, in mL, of the aliquot taken to prepare the Test solution.

Assay—

Mobile solvent—Prepare a suitable aqueous solution of acetonitrile, approximately 1 in 3, such that the retention time of dexamethasone is between 3 minutes and 6 minutes.

Standard preparation—Dissolve an accurately weighed quantity of USP Dexamethasone RS in dilute methanol (1 in 2) to obtain a solution having a known concentration of about 0.1 mg per mL.

Assay preparation—Weigh and finely powder not fewer than 10 Tablets. Weigh accurately a portion of the powder, equivalent to about 5 mg of dexamethasone, transfer to a 50-mL volumetric flask, and add 30 mL of dilute methanol (1 in 2). Sonicate the flask for about 2 minutes, shake by mechanical means for 30 minutes, and dilute with the same solvent to volume. Filter a portion of the mixture through a suitable filter to obtain a clear filtrate.

Procedure—Introduce equal volumes (between 5 µL and 25 µL) of the Assay preparation and the Standard preparation into a high-pressure liquid chromatograph (see Chromatography (621)) operated at room temperature, by means of a loop injector, adjusting the specimen size and other operating parameters such that the peak obtained with the Standard preparation is about 0.6 full scale. Typically, the apparatus is fitted with a 4.6-mm × 30-cm column packed with packing L1 and is equipped with an UV detector capable of monitoring absorption at 254 nm and a suitable recorder. In a suitable chromatogram, the coefficient of variation for five replicate injections of a single specimen is not more than 3.0%. Measure the responses of the peaks, at identical retention times, obtained with the Assay preparation and the Standard preparation, Calculate the quantity, in mg, of C$_{22}$H$_{29}$FO$_5$, in the portion of Tablets taken by the formula:

$$50C(r_U / r_S)$$

in which C is the concentration, in mg per mL, of USP Dexamethasone 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.
**Dexamethasone Acetate**

**C₂₄H₃₁FO₆ · H₂O**  452.51

Pregna-1,4-diene-3,20-dione, 21-(acetoxy)-9-fluoro-11,17-dihydroxy-16-methyl-1(11β,16α)-monohydrate. 9-Fluoro-11ß,17ß,21-trihydroxy-16α-methylpregna-1,4-diene-3,20-dione 21-acetate monohydrate  \( [55812-90-3] \).

Anhydrous 434.51 \([1177-87-3]\).

- Dexamethasone Acetate contains one molecule of water of hydration or is anhydrous. It contains not less than 97.0 per cent and not more than 102.0 percent of \( C₂₄H₃₁FO₆ \), calculated on the dried basis.

**Packaging and storage**—Preserve in well-closed containers. Store at 25°, excursions permitted between 15° and 30°.

**Labeling**—Label it to indicate whether it is hydrous or anhydrous.

**USP Reference standards** (11)—

USP Dexamethasone Acetate RS

**Identification**—

A: Infrared Absorption (197M).

B: Ultraviolet Absorption (197U)—

Solution: 15 μg per mL.

Medium: methanol.

Absorptivities at 239 nm, calculated on the dried basis, do not differ by more than 3.0%.

**Specific rotation** \( (781S) \); between +82° and +88°.

**Test solution**—10 mg per mL in dioxane.

**Loss on drying** (731)—Dry it in vacuum at 105° for 3 hours; the hydrous form loses between 3.5% and 4.5%, and the anhydrous form not more than 0.4%, of its weight.

**Residue on ignition** (281); not more than 0.1%.

**Heavy metals**, Method II (231); not more than 0.002%.

**Chromatographic purity**—

- Mobile phase—Prepare a filtered and degassed mixture of Formate buffer and acetonitrile (3:2). Make adjustments if necessary (see System Suitability under Chromatography (621)).

- Test solution—Transfer about 200 mg of Dexamethasone Acetate, accurately weighed, to a 100-mL volumetric flask. Add 100 mL of Diluent, and sonicate until a clear solution is obtained. Dilute with Diluent to volume, and mix.

- System Suitability—The liquid chromatograph is equipped with a 254-nm detector and a 4.6-mm × 25-cm column containing 10-μm packing L1. The flow rate is about 1 mL per minute. Chromatograph the Standard preparation, and record the peak responses as directed for Procedure: the capacity factor, \( k' \), is not less than 2.0; the column efficiency is not less than 1500 theoretical plates; the tailing factor is not more than 2.0; and the relative standard deviation for replicate injections is not more than 2.0%.

- Procedure—Inject a volume (about 10 μL) of the Test solution into the chromatograph, record the chromatogram, and measure the peak responses and retention times. The mobile phase is a mixture of acetonitrile and water. The mobile phase is mixed with 1 N sodium hydroxide (1:1). Obtain the test solution by dissolving 100 mg of Dexamethasone Acetate in Water for Injection. It contains an amount of dexamethasone acetate monohydrate \( (C₂₄H₃₁FO₆ · H₂O) \) equivalent to not less than 90.0 percent and not more than 110.0 per cent of the labeled amount of dexamethasone acetate in the portion of Dexamethasone Acetate taken by the formula:

\[
250(c_r / t_r)
\]

in which \( c \) is the concentration, in mg per mL, of USP Dexamethasone Acetate RS in the Standard preparation; and \( t_r \) and \( r_\text{is} \) are the peak responses and retention times.

**Dexamethasone Acetate Injectable Suspension**

- Dexamethasone Acetate Injectable Suspension is a sterile suspension of Dexamethasone Acetate in Water for Injection. It contains an amount of dexamethasone acetate monohydrate \( (C₂₄H₃₁FO₆ · H₂O) \) equivalent to not less than 90.0 percent and not more than 110.0 per cent of the labeled amount of dexamethasone acetate in the portion of Dexamethasone Acetate taken by the formula:

\[
250(c_r / t_r)
\]

in which \( c \) is the concentration, in mg per mL, of USP Dexamethasone Acetate RS in the Standard preparation; and \( t_r \) and \( r_\text{is} \) are the peak responses and retention times.

**Packaging and storage**—Preserve in single-dose or multiple-dose containers, preferably of Type I glass.

**USP Reference standards** (11)—

USP Dexamethasone Acetate RS USP Endotoxin RS

**Identification**, Infrared Absorption (197M)—Obtain the test specimen as follows. Transfer the contents of a well-shaken container of Injectable Suspension to a fine-porosity, sintered-glass vacuum filter, filter, and wash with several 10-mL portions of water. Remove the powder from the filter and allow to air-dry. [NOTE—Do not use heat to dry y the specimen. Total or partial dehydration may occur. Use a similar undried preparation of USP Dexamethasone Acetate RS.]

**Bacterial endotoxins** (85)—It contains not more than 21.7 USP Endotoxin Units per mg of dexamethasone acetate.

**pH** (791); between 5.0 and 7.5.

**Other requirements**—It meets the requirements under *Injections* (1).