Desoxycorticosterone Pivalate Injectable Suspension

DEFINITION

Desoxycorticosterone Pivalate Injectable Suspension is a sterile suspension of Desoxycorticosterone Pivalate in an aqueous medium. It contains NLT 90.0% and NMT 110.0% of the labeled amount of desoxycorticosterone pivalate ($C_{26}H_{38}O_4$).

IDENTIFICATION

Change to read:

- **A.**
  Sample: Centrifuge a portion of Injectable Suspension, decant the supernatant, wash the residue by stirring with several successive portions of water, centrifuging and decanting each time, and finally dry the residue at 105°. The residue so obtained, meets the following requirements.
  Analysis 1: Melting point
  Acceptance criteria 1: Melts at 198°–•206°• (RB 1-Feb-2015)
  Analysis 2: Dissolve 5 mg in 2 mL of sulfuric acid.
  Acceptance criteria 2: The solution is yellowish, with a greenish fluorescence.
  Analysis 3: Dilute the solution obtained from *Analysis 2* with 2 mL of water.
  Acceptance criteria 3: The color changes to a dark red-blue, and on further dilution with 2 mL of water, the color is discharged.

ASSAY

- **PROCEDURE**
  Mobile phase: Methanol and water (4:1)
  Internal standard solution: 2 mg/mL of desoxycorticosterone acetate in methanol
  Standard solution: 0.5 mg/mL of USP Desoxycorticosterone Pivalate RS in methanol, prepared as follows. Transfer 12.5 mg of USP Desoxycorticosterone Pivalate RS to a 25-mL volumetric flask, and add 20 mL of methanol. Add 2.5 mL of the *Internal standard solution*, and dilute with methanol to volume.
  Sample solution: Nominally 0.5 mg/mL of desoxycorticosterone pivalate in methanol, prepared as follows. Transfer a nominal equivalent of 125 mg of desoxycorticosterone pivalate from Injectable Suspension to a 250-mL volumetric flask. Add 200 mL of methanol, and sonicate to dissolve. Add 25.0 mL of *Internal standard solution*, and dilute with methanol to volume. Centrifuge a 20-mL portion at high speed for 5 min. Filter the supernatant through a 5-µm disk, discarding the first 5 mL of the filtrate.
  Chromatographic system
  (See *Chromatography* ⟨621⟩, *System Suitability*.)
  Mode: LC
  Detector: UV 254 nm
  Column: 4.6-mm × 25-cm; packing L7
  Flow rate: 1.5 mL/min
  Injection volume: 25 µL
  System suitability
  Sample: *Standard solution*
  [NOTE—The relative retention times for desoxycorticosterone acetate and for desoxycorticosterone pivalate are about 0.5 and 1.0, respectively.]
  Suitability requirements
  Resolution: NLT 2.0 between desoxycorticosterone acetate and desoxycorticosterone pivalate
  Relative standard deviation: NMT 1.5% for the peak response ratio of desoxycorticosterone pivalate to the internal standard
  Analysis
  Samples: *Standard solution* and *Sample solution*
  Calculate the percentage of the labeled amount of desoxycorticosterone pivalate ($C_{26}H_{38}O_4$) in the portion of Injectable Suspension taken:

$$\text{Result} = (R_U/R_S) \times (C_S/C_U) \times 100$$

R_U = peak response ratio of desoxycorticosterone pivalate to the internal standard from the *Sample solution*
R_S = peak response ratio of desoxycorticosterone pivalate to the internal standard from the *Standard solution*
C_S = concentration of USP Desoxycorticosterone Pivalate RS in the *Standard solution* (mg/mL)
C_U = nominal concentration of desoxycorticosterone pivalate in the *Sample solution* (mg/mL)

Acceptance criteria: 90.0%–110.0%

SPECIFIC TESTS

- **pH ⟨791⟩:** 5.0–7.0
- **OTHER REQUIREMENTS:** It meets the requirements in *Injections and Implanted Drug Products* ⟨1⟩.
- **BACTERIAL ENDOTOXINS TEST ⟨85⟩:** It contains NMT 2.78 USP Endotoxin Units/mg of desoxycorticosterone pivalate.

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in single-dose or multiple-dose containers, preferably of Type I glass, protected from light.
- **LABELING:** Label Suspension to indicate that it is for veterinary use only.
- **USP REFERENCE STANDARDS ⟨11⟩**
  USP Desoxycorticosterone Pivalate RS
  USP Endotoxin RS