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Dexamethasone Tablets

To view the Notice from the Expert Committee that posted in conjunction with this accelerated revision, please click https://www.uspnf.com/rb-dexamethasone-tabs-20230929.

DEFINITION
Dexamethasone Tablets contain NLT 90.0% and NMT 110.0% of the labeled amount of dexamethasone ($C_{22}H_{29}FO_5$).

IDENTIFICATION

• **A. Thin-Layer Chromatography**

Standard solution: 500 µg/mL of USP Dexamethasone RS in chloroform

Sample solution: Nominally, 1 mg/mL of dexamethasone prepared as follows. Evaporate 10 mL of the *Sample solution* as directed under the *Assay* on a steam bath just to dryness, and dissolve the residue in 1 mL of chloroform.

Chromatographic system

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

Adsorbent: 0.25-mm layer of chromatographic silica gel mixture

Application volume

Sample solution: 10 µL

Standard solution: 20 µL

Analysis

Samples: *Standard solution* and *Sample solution*

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.

Acceptance criteria: The R_F value of the principal spot of the *Sample solution* corresponds to that of the *Standard solution*.

ASSAY

• **Procedure**

Diluent: Methanol and water (50:50)

Mobile phase: Acetonitrile and water (33:66)

Standard solution: 0.1 mg/mL of USP Dexamethasone RS in *Diluent*

Sample solution: Nominally 0.1 mg/mL of dexamethasone prepared as follows. Transfer the equivalent of 5 mg of dexamethasone from finely powdered Tablets (NLT 10) to a 50-mL volumetric flask, and add 30 mL of *Diluent*. Sonicate the flask for 2 min, shake by mechanical means for 30 min, and dilute with *Diluent* to volume. Pass a portion of the mixture through a suitable filter to obtain a clear filtrate.

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 30-cm; packing L1

Injection volume: 5–25 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Relative standard deviation: NMT 3.0%, for five replicate injections

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of dexamethasone ($C_{22}H_{29}FO_5$) in the portion of Tablets 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 Dexamethasone RS in the *Standard solution* (mg/mL)

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

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

Change to read:

• **Dissolution** ⟨711⟩

Test 1

Medium: Dilute 1 mL of hydrochloric acid with water to 100 mL; 500 mL

Apparatus 1: 100 rpm

Time: 45 min

Standard solution: Prepare as directed for *Standard Preparation* in *Assay for Steroids* ⟨351⟩, using USP Dexamethasone RS.

Sample solution: Extract a filtered aliquot of *Medium*, equivalent to 0.2 mg 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.

Analysis: Proceed as directed for *Procedure* in *Assay for Steroids* ⟨351⟩, except allow it to stand in the dark for 45 min.

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of dexamethasone ($C_{22}H_{29}FO_5$) dissolved:

$$\text{Result} = (A_U/A_S) \times 20 \times C_S \times (V/V_S) \times (1/L) \times 100$$

A_U = absorbance of the *Sample solution*

A_S = absorbance of the *Standard solution*

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

V = volume of *Medium*, 500 mL

V_S = volume of *Medium* extracted with chloroform (mL)

L = label claim (mg/Tablet)

Tolerances: NLT 70% (*Q*) of the labeled amount of dexamethasone ($C_{22}H_{29}FO_5$) is dissolved.

Test 2: If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 2*.

Medium: 0.1 N hydrochloric acid VS; 500 mL

Apparatus 1: 100 rpm

Time: 30 min

Solution A: 25% (v/v) phosphoric acid in water prepared as follows. Transfer 25 mL of phosphoric acid to a 100-mL volumetric flask containing about 50 mL of water. Cool and dilute with water to volume.

Buffer: 1.36 g/L of monobasic potassium phosphate in water. Add 1.0 mL of triethylamine to each liter of solution, and adjust with *Solution A* to a pH of 3.0.

Mobile phase: Methanol and *Buffer* (50:50)

Standard stock solution: 0.5 mg/mL of USP Dexamethasone RS in methanol. Sonicate to dissolve as needed.

Standard solution: (*L*/500) mg/mL of USP Dexamethasone RS from *Standard stock solution* in *Medium*, where *L* is the label claim in mg/Tablet

Sample solution: Pass a portion of the solution under test through a suitable filter of 0.45-µm pore size and discard the first 5 mL of the filtrate.

Chromatographic system

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

Mode: LC

Detector: UV 240 nm

Column: 4.6-mm × 5.0-cm; 3.5-µm packing L1

Flow rate: 1 mL/min

Injection volume: 50 µL

Run time: NLT 1.3 times the retention time of dexamethasone

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 2.0

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of dexamethasone ($C_{22}H_{29}FO_5$) dissolved:

$$\text{Result} = (r_U/r_S) \times C_S \times V \times (1/L) \times 100$$

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

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

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

V = volume of the *Medium*, 500 mL

L = label claim (mg/Tablet)

Tolerances: NLT 80% (Q) of the labeled amount of dexamethasone ($C_{22}H_{29}FO_5$) is dissolved.

▲**Test 3:** If the product complies with this test, the labeling indicates that it meets USP *Dissolution Test 3*.

Medium: 0.1 N hydrochloric acid; 500 mL, deaerated

Apparatus 2: 75 rpm

Time: 30 min

Mobile phase: Acetonitrile and water (350:600)

Solution A: Methanol and water (50:50)

Standard stock solution A: 0.5 mg/mL of USP Dexamethasone RS in *Solution A*

Standard stock solution B: 0.05 mg/mL of USP Dexamethasone RS from *Standard stock solution A* in *Medium*

Standard solution: (L/500) mg/mL of USP Dexamethasone RS from *Standard stock solution B* in *Medium*, where L is the label claim in mg/Tablet

Sample solution: Pass a portion of the solution under test through a suitable filter of 0.45-µm pore size, discarding the first 1 mL of the filtrate.

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 10-cm; 5-µm packing L1

Flow rate: 1 mL/min

Injection volume

For Tablets labeled to contain 1, 1.5, 2, 4, and 6 mg of dexamethasone: 50 µL

For Tablets labeled to contain 0.5 and 0.75 mg of dexamethasone: 100 µL

Run time: NLT 1.5 times the retention time of dexamethasone

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: 0.8–1.5

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of dexamethasone ($C_{22}H_{29}FO_5$) dissolved:

$$\text{Result} = (r_U/r_S) \times C_S \times V \times (1/L) \times 100$$

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

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

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

V = volume of the *Medium*, 500 mL

L = label claim (mg/Tablet)

Tolerances: NLT 80% (Q) of the labeled amount of dexamethasone ($C_{22}H_{29}FO_5$) is dissolved.▲ (RB 12-Sep-2023)

- **Uniformity of Dosage Units ⟨905⟩**

Procedure for content uniformity

Standard solution: Prepare as directed for *Standard Preparation* in *Assay for Steroids ⟨351⟩*, using USP Dexamethasone RS.

Sample 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, and add chloroform to volume. Pipet a volume of this solution, equivalent to 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 *Assay for Steroids ⟨351⟩, Procedure*.

Analysis

Samples: *Standard solution* and *Sample solution*

Proceed as directed in *Assay for Steroids ⟨351⟩, Procedure*, except allow it to stand in the dark for 45 min.

Calculate the percentage of total steroids, as dexamethasone ($C_{22}H_{29}FO_5$), in the Tablet:

$$\text{Result} = (A_U/A_S) \times C_S \times V \times (1/L) \times 100$$

A_U = absorbance of the *Sample solution*

A_S = absorbance of the *Standard solution*

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

V = volume of the chloroform extract (mL) used to prepare the *Sample solution*

L = label claim (mg/Tablet)

Acceptance criteria: Meets the requirements

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in well-closed containers.
- **Labeling:** When more than one *Dissolution* test is given, the labeling states the *Dissolution* test used only if *Test 1* is not used.
- **USP Reference Standards ⟨11⟩**
  USP Dexamethasone RS

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

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

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