Status: Currently Official on 17-Feb-2025
Official Date: Official as of 01-May-2019
Document Type: USP Monographs
DocId: GUID-BD6D73B0-7181-4C28-879A-B92D9EE682E1_2_en-US
DOI: https://doi.org/10.31003/USPNF_M23330_02_01
DOI Ref: 8rcx4

Dexamethasone Ophthalmic Suspension

DEFINITION

Dexamethasone Ophthalmic Suspension is a sterile, aqueous suspension of dexamethasone containing a suitable antimicrobial preservative. It may contain suitable buffers, stabilizers, and suspending and viscosity agents. It contains NLT 90.0% and NMT 110.0% of the labeled amount of dexamethasone ($C_{22}H_{29}FO_5$).

IDENTIFICATION

Delete the following:

▲• **Thin-Layer Chromatography**

Adsorbent: 0.25-mm layer of chromatographic silica gel mixture.

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

Sample solution: Transfer a volume of Ophthalmic Suspension, equivalent to about 2.5 mg of dexamethasone, to a test tube, add 5 mL of chloroform, shake, and centrifuge.

Application volume: 10 µL of the chloroform layer

Developing solvent system: *Single-Steroid Assay ⟨511⟩, Solvent A* as directed under *Single-Steroid Assay ⟨511⟩*

Analysis: Develop the chromatogram, mark the solvent front, and locate the spots on the plate by spraying with a 1 in 5 solution of *p*-toluenesulfonic acid in a mixture of 9 volumes of alcohol and 1 volume of propylene glycol, and heating until spots appear.

Acceptance criteria: The R_F value of the principal spot of the *Sample solution* corresponds to that of the *Standard solution*.▲ *(USP 1-May-2019)*

Add the following:

▲• **A.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.▲ (USP 1-May-2019)

Add the following:

▲• **B.** The UV spectrum of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.▲ (USP 1-May-2019)

ASSAY

Change to read:

• **Procedure**

▲**Solution A:** 3.4 g/L of monobasic potassium phosphate in water. Adjust with phosphoric acid to a pH of 3.0.

Solution B: Acetonitrile

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0.0	76	24
10	76	24
15	45	55
16	10	90
16.1	76	24
20.0	76	24

Diluent: Acetonitrile and water (20:80)

System suitability solution: 0.06 mg/mL of USP Dexamethasone RS and 4 µg/mL of USP Betamethasone RS in *Diluent*. Sonicate to dissolve as needed.

Standard solution: 0.06 mg/mL of USP Dexamethasone RS in *Diluent*. Sonicate to dissolve as needed.

Sample solution: Prior to sampling, sonicate the drug product in the original container to achieve optimum homogeneity of the sample to be tested. Nominally 0.06 mg/mL of dexamethasone from Ophthalmic Suspension in *Diluent*. Vortex for 10–15 s and then sonicate for 2 min to dissolve.

Chromatographic system

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

Mode: LC

Detector: UV 240 nm. For *Identification B*, use a diode array detector in the range of 210–400 nm.

Column: 2.1-mm × 10-cm; 1.7-µm packing L1

Column temperature: 35°

Flow rate: 0.4 mL/min

Injection volume: 10 µL

System suitability

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

[NOTE—See *Table 2* for the relative retention times.]

Suitability requirements

Resolution: NLT 1.5 between betamethasone and dexamethasone, *System suitability solution*

Tailing factor: NMT 2.0, *Standard solution*

Relative standard deviation: NMT 1.0%, *Standard solution*

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 Ophthalmic Suspension 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)▲ (USP 1-May-2019)

Acceptance criteria: 90.0%–110.0%

IMPURITIES

Add the following:

▲• **ORGANIC IMPURITIES**

Solution A, Solution B, Mobile phase, Diluent, System suitability solution, and **Chromatographic system:** Proceed as directed in the *Assay*.

Standard solution: 0.6 µg/mL of USP Dexamethasone RS in *Diluent*. Sonicate to dissolve as needed.

Sample solution: Prior to sampling, sonicate the drug product in the original container to achieve optimum homogeneity of the sample to be tested. Nominally 300 µg/mL of dexamethasone from Ophthalmic Suspension in *Diluent*. Vortex for 10–15 s and then sonicate for 2 min to dissolve.

System suitability

[NOTE—See *Table 2* for the relative retention times.]

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

Suitability requirements

Resolution: NLT 1.5 between betamethasone and dexamethasone, *System suitability solution*

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

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of any individual degradation product in the portion of Ophthalmic Suspension taken:

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

r_U = peak response of any individual degradation product 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* (µg/mL)

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

Acceptance criteria: See *Table 2*.

Table 2

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
16α-Methylprednisone[a]	0.87	—[b]
Betamethasone	0.94	—[b]
Dexamethasone	1.00	—[b]
Dexamethasone 7,9-diene[c]	1.39	—[b]
Desoximetasone	1.55	—[b]
Dexamethasone acetate	1.72	—[b]
Any unspecified degradation product	—	0.2
Total degradation products	—	0.5▲ (USP 1-May-2019)

[a] 17,21-Dihydroxy-16α-methylpregna-1,4-diene-3,11,20-trione.

[b] Process impurity included in the table for identification only. Process impurities are controlled in the drug substance and are not to be reported or included in the total degradation products for the drug product.

[c] 17,21-Dihydroxy-16α-methylpregna-1,4,7,9(11)-tetraene-3,20-dione.

SPECIFIC TESTS

• **pH** ⟨791⟩**:** 5.0–6.0

• **Sterility Tests** ⟨71⟩**:** Meets the requirements

ADDITIONAL REQUIREMENTS

Change to read:

• **Packaging and Storage:** Preserve in tight containers. ▲Store upright at 8°–27°.▲ (USP 1-May-2019)

Change to read:

• **USP Reference Standards** ⟨11⟩

▲USP Betamethasone RS▲ (USP 1-May-2019)

USP Dexamethasone RS

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

Topic/Question	Contact	Expert Committee
DEXAMETHASONE OPHTHALMIC SUSPENSION	Documentary Standards Support	SM52020 Small Molecules 5
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM52020 Small Molecules 5

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 43(3)

Current DocID: GUID-BD6D73B0-7181-4C28-879A-B92D9EE682E1_2_en-US

DOI: https://doi.org/10.31003/USPNF_M23330_02_01

DOI ref: 8rcx4