Status: Currently Official on 16-Feb-2025
Official Date: Official as of 01-Aug-2017
Document Type: USP Monographs
DocId: GUID-D9AF17B0-6F60-40AB-99BD-CC04559F682D_1_en-US
DOI: https://doi.org/10.31003/USPNF_M81840_01_01
DOI Ref: 7hf6d

Tetracycline Hydrochloride Ophthalmic Ointment

DEFINITION

Tetracycline Hydrochloride Ophthalmic Ointment contains NLT 90.0% and NMT 125.0% of the labeled amount of tetracycline hydrochloride ($C_{22}H_{24}N_2O_8 \cdot HCl$).

IDENTIFICATION

• **A.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY

• **Procedure**

Solution A: 0.1 M ammonium oxalate

Solution B: 0.2 M dibasic ammonium phosphate

Diluent: Dimethylformamide and *Solution A* (270:680)

Mobile phase: Dimethylformamide, *Solution A*, and *Solution B* (270:680:50). Adjust, if necessary, with 3 N ammonium hydroxide or 3 N phosphoric acid to a pH of 7.6–7.7.

System suitability solution: 100 µg/mL of tetracycline hydrochloride and 25 µg/mL of USP 4-Epianhydrotetracycline Hydrochloride RS in *Diluent*

Standard stock solution: 1 mg/mL of USP Tetracycline Hydrochloride RS in methanol

Standard solution: 0.12 mg/mL of USP Tetracycline Hydrochloride RS from *Standard stock solution* in *Diluent*

Sample stock solution: Nominally 3 mg/mL of tetracycline hydrochloride prepared as follows. Transfer a portion of Ophthalmic Ointment, containing nominally 300 mg of tetracycline hydrochloride, to a glass-stoppered conical flask. Add 20 mL of cyclohexane, and shake. Add 35 mL of methanol, and sonicate for 20 min. Filter this solution into a 100-mL volumetric flask, and rinse the sides of the conical flask with 40 mL of methanol, filtering the rinsing into the volumetric flask. Dilute with methanol to volume.

Sample solution: Nominally 0.12 mg/mL of tetracycline hydrochloride in *Diluent* from *Sample stock solution*

Chromatographic system

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

Mode: LC

Detector: UV 280 nm

Columns

Guard: 4.6-mm × 3-cm; 10-µm packing L7

Analytical: 4.6-mm × 25-cm; 5–10-µm packing L7

Flow rate: 2 mL/min

Injection volume: 20 µL

System suitability

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

[Note—The relative retention times of 4-epianhydrotetracycline and tetracycline are 0.9 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 1.2 between 4-epianhydrotetracycline and tetracycline, *System suitability solution*

Relative standard deviation: NMT 2.0%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of tetracycline hydrochloride ($C_{22}H_{24}N_2O_8 \cdot HCl$) in the portion of Ophthalmic Ointment taken:

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

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

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

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

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

P = potency of USP Tetracycline Hydrochloride RS (µg/mg)

F = conversion factor, 0.001 mg/µg

Acceptance criteria: 90.0%–125.0%

SPECIFIC TESTS

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

• **Other Requirements:** It meets the requirements for *Particulate and Foreign Matter* and *Container Contents* in *Ophthalmic Products—Quality Tests ⟨771⟩, Drug Product Quality, Universal Tests, Particulate and Foreign Matter* and *Container Contents*.

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in collapsible ophthalmic ointment tubes.

• **USP Reference Standards ⟨11⟩**

USP 4-Epianhydrotetracycline Hydrochloride RS	$C_{22}H_{22}N_2O_7 \cdot HCl$	462.88
USP Tetracycline Hydrochloride RS		

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

Topic/Question	Contact	Expert Committee
TETRACYCLINE HYDROCHLORIDE OPHTHALMIC OINTMENT	Documentary Standards Support	SM12020 Small Molecules 1
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM12020 Small Molecules 1

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 39(5)

Current DocID: GUID-D9AF17B0-6F60-40AB-99BD-CC04559F682D_1_en-US

DOI: https://doi.org/10.31003/USPNF_M81840_01_01

DOI ref: 7hf6d