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Sulfacetamide Sodium and Prednisolone Acetate Ophthalmic Ointment

DEFINITION

Sulfacetamide Sodium and Prednisolone Acetate Ophthalmic Ointment is a sterile ointment containing NLT 90.0% and NMT 110.0% of the labeled amounts of sulfacetamide sodium ($C_8H_9N_2NaO_3S \cdot H_2O$) and prednisolone acetate ($C_{23}H_{30}O_6$).

IDENTIFICATION

• **A.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the tests for *Sulfacetamide Sodium* and *Prednisolone Acetate* in the *Assay*.

• **B.** The UV absorption spectra of the major peak of the *Sample solution* and that of the *Standard solution* exhibit maxima and minima at the same wavelengths, as obtained in the tests for *Sulfacetamide Sodium* and *Prednisolone Acetate* in the *Assay*.

ASSAY

• **Sulfacetamide Sodium**

Diluent: Dilute methanol (1 in 5)

Mobile phase: Methanol, glacial acetic acid, and water (100:10:890), filtered and degassed

Standard solution: Transfer about 50 mg of USP Sulfacetamide Sodium RS to a 40-mL centrifuge tube. Add 10.0 mL of *Diluent*, insert the stopper in the tube, and mix using a vortex mixer for about 3 min to dissolve. Add 7.5 mL of heptane, insert the stopper in the tube, and mix using a vortex mixer for another 3 min. Centrifuge to effect separation of the phases. Withdraw and discard the upper heptane layer. Transfer 3.0 mL of the bottom layer to a 500-mL volumetric flask, add *Diluent* to volume, and mix.

System suitability solution: Dissolve 3 mg of sulfanilamide in 100 mL of the *Standard solution*, and mix.

Sample solution: Transfer a quantity of Ophthalmic Ointment nominally equivalent to about 100 mg of sulfacetamide sodium to a 40-mL centrifuge tube. Add 15.0 mL of heptane, insert the stopper in the tube, and mix using a vortex mixer for about 3 min to dissolve the Ophthalmic Ointment. Add 20.0 mL of *Diluent*, insert the stopper in the tube, and mix using a vortex mixer for 3 min. Centrifuge to effect separation of the phases. Withdraw and discard the upper heptane layer. Transfer 3.0 mL of the bottom layer to a 500-mL volumetric flask, dilute with *Diluent* to volume, and mix.

Chromatographic system

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

Mode: LC

Detector: 254-nm diode array

Column: 4.6-mm × 25-cm; packing L1

Flow rate: 1.5 mL/min

Injection volume: 90 µL

System suitability

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

Suitability requirements

Resolution: NLT 3 between the sulfacetamide and sulfanilamide peaks, *System suitability solution*

Column efficiency: NLT 1500 theoretical plates, *Standard solution*

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

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of sulfacetamide sodium ($C_8H_9N_2NaO_3S \cdot H_2O$) in the portion of Ophthalmic Ointment taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times (M_{r1}/M_{r2}) \times 100$$

r_U = peak response of sulfacetamide sodium from the *Sample solution*

r_S = peak response of sulfacetamide sodium from the *Standard solution*

C_S = concentration of USP Sulfacetamide Sodium RS, calculated on the anhydrous basis, in the *Standard solution* (mg/mL)

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

M_{r1} = molecular weight of sulfacetamide sodium monohydrate, 254.24

M_{r2} = molecular weight of anhydrous sulfacetamide sodium, 236.23

Acceptance criteria: 90.0%–110.0%

• **Prednisolone Acetate**

Diluent: Dilute methanol (9 in 10)

Mobile phase: Acetonitrile and water (400:600), filtered and degassed

Internal standard solution: 0.7 mg/mL of norethindrone in *Diluent*

Standard stock solution: 0.8 mg/mL of USP Prednisolone Acetate RS in *Diluent*

Standard solution: 0.04 mg/mL of USP Prednisolone Acetate RS prepared as follows. Transfer 5.0 mL of *Standard stock solution* to a 100-mL volumetric flask, add 5.0 mL of *Internal standard solution*, dilute with *Diluent* to volume, and mix.

Sample solution: Transfer a quantity of Ophthalmic Ointment nominally equivalent to about 4 mg of prednisolone acetate to a 50-mL centrifuge tube. Add 10.0 mL of heptane, and mix using a vortex mixer for about 2 min to dissolve the Ophthalmic Ointment. Add 5.0 mL of *Internal standard solution* and 20.0 mL of *Diluent*, and mix using a vortex mixer for 2 min. Centrifuge to effect separation of the phases. Withdraw and discard the upper heptane layer. Transfer the lower layer to a 100-mL volumetric flask. Add *Diluent* to volume, and mix.

Chromatographic system

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

Mode: LC

Detector: 254-nm diode array

Column: 3.9-mm × 30-cm; packing L1

Flow rate: 1.5 mL/min

Injection volume: 40 µL

System suitability

Sample: *Standard solution*

[Note—The relative retention times for prednisolone acetate and norethindrone are about 1.0 and 1.5, respectively.]

Suitability requirements

Resolution: NLT 4.5 between the prednisolone and norethindrone peaks

Column efficiency: NLT 3000 theoretical plates for the prednisolone peak

Tailing factor: NMT 2.5 for the prednisolone peak

Relative standard deviation: NMT 1.5% for the peak response ratio of prednisolone acetate to norethindrone

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of prednisolone acetate ($C_{23}H_{30}O_6$) in the portion of Ophthalmic Ointment taken:

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

R_U = peak response ratio of prednisolone acetate to the internal standard peak from the *Sample solution*

R_S = peak response ratio of prednisolone acetate to the internal standard peak from the *Standard solution*

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

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

Acceptance criteria: 90.0%–110.0%

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 that are tamper-proof so that sterility is assured at time of first use.
- **USP Reference Standards** ⟨11⟩

  USP Prednisolone Acetate RS

  USP Sulfacetamide Sodium RS

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

Topic/Question	Contact	Expert Committee
SULFACETAMIDE SODIUM AND PREDNISOLONE ACETATE OPHTHALMIC OINTMENT	Documentary Standards Support	SM22020 Small Molecules 2
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

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