Dexamethasone Sodium Phosphate Ophthalmic Solution

DEFINITION

Dexamethasone Sodium Phosphate Ophthalmic Solution is a sterile, aqueous solution of Dexamethasone Sodium Phosphate. It contains an amount of dexamethasone sodium phosphate ($C_{22}H_{28}FNa_2O_8P$) equivalent to NLT 90.0% and NMT 115.0% of the labeled amount of dexamethasone phosphate ($C_{22}H_{30}FO_8P$).

IDENTIFICATION

- **A. Thin-Layer Chromatography**

Solution A: Dissolve 3.1 g of boric acid, 203 mg of magnesium chloride, and 860 mg of sodium hydroxide in enough water to make 1000 mL.

Solution B: 1 mg/mL of alkaline phosphatase enzyme in *Solution A*

Standard solution: 300 µg/mL of USP Dexamethasone RS in methylene chloride

Sample solution: Transfer 5 mL of *Solution B* to a glass-stoppered, 50-mL tube containing 5 mL of the *Sample solution* from the *Assay*. Incubate at 37° for 45 min, then add 25 mL of methylene chloride and shake for 2 min. Evaporate 15 mL of the methylene chloride extract on a steam bath to dryness, and dissolve the residue in 1 mL of methylene chloride.

Chromatographic system
(See *Chromatography* ⟨621⟩*, Thin-Layer Chromatography*.)

Adsorbent: 0.25-mm layer of chromatographic silica gel mixture (20- × 20-cm plate)

Application volume: 5 µL

Developing solvent system: Chloroform, acetone, and water (50:50:1)

Spray reagent: Dilute sulfuric acid (1 in 2)

Analysis

Samples: *Standard solution* and *Sample solution*

Allow the spots to dry, and develop the chromatogram using the *Developing solvent system* in a tank completely lined with filter paper until the solvent front has moved about three-fourths of the length of the plate. Remove the plate from the developing tank, mark the solvent front, and allow the spots to dry. Spray the plate with *Spray reagent*, and heat at 105° until brown or black spots appear.

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

Add the following:

•• **B.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.• (IRA 1-May-2015)

ASSAY

Change to read:

- **Procedure**

Mobile phase: 0.01 M monobasic potassium phosphate in a mixture of methanol and water (1:1)

Standard solution: •0.09 mg/mL of freshly prepared USP Dexamethasone Sodium Phosphate RS in *Mobile phase*• (IRA 1-May-2015)

Sample solution: Nominally 0.08 mg/mL of dexamethasone phosphate from Ophthalmic Solution in *Mobile phase*

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

Mode: LC

Detector: UV 254 nm

Column: 4-mm × 30-cm; packing L1

Flow rate: 1.6 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

[NOTE—The retention time for dexamethasone phosphate is about 5 min.]

Suitability requirements

Relative standard deviation: NMT 1.5%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of dexamethasone phosphate ($C_{22}H_{30}FO_8P$) in the portion of Ophthalmic Solution taken:

$$\text{•Result} = (r_U/r_S) \times (C_S/C_U) \times (M_{r1}/M_{r2}) \times 100\text{•}$$ (IRA 1-May-2015)

r_U = peak response from the *Sample solution*
r_S = peak response from the *Standard solution*
C_S = concentration of •USP Dexamethasone Sodium Phosphate RS• (IRA 1-May-2015) in the *Standard solution* (µg/mL)
C_U = nominal concentration of dexamethasone phosphate in the *Sample solution* (mg/mL)
M_{r1} = molecular weight of dexamethasone phosphate, 472.44
M_{r2} = molecular weight of dexamethasone sodium phosphate, 516.40• (IRA 1-May-2015)

Acceptance criteria: 90.0%–115.0%

SPECIFIC TESTS

- **pH** ⟨**791**⟩**:** 6.6–7.8
- **Sterility Tests** ⟨**71**⟩**:** Meets the requirements

ADDITIONAL REQUIREMENTS

Change to read:

- **Packaging and Storage:** Preserve in tight, light-resistant containers. •Store between 15° and 25°.• (IRA 1-May-2015)

Change to read:

- **USP Reference Standards** ⟨**11**⟩

USP Dexamethasone RS

•USP Dexamethasone Sodium Phosphate RS• (IRA 1-May-2015)