

Dexamethasone Sodium Phosphate Cream

DEFINITION

Dexamethasone Sodium Phosphate Cream 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

Solution A: Dissolve 0.29 g of dibasic sodium phosphate in 450 mL of water, add 550 mL of alcohol, and mix.

Solution B: 6.9 g/L of monobasic sodium phosphate in water

Mobile phase: Methanol and *Solution B* (52:48)

Standard solution: 33 µg/mL of freshly prepared USP Dexamethasone Sodium Phosphate RS in *Solution A*.

(IRA 1-May-2015)

Sample solution: Nominally 30 µg/mL of dexamethasone phosphate, prepared as follows. Transfer a portion of Cream, equivalent to 3 mg of dexamethasone phosphate, to a 150-mL beaker. Add 65 mL of *Solution A*, and heat just to boiling. Pour the contents of the beaker into a 125-mL separator containing 45 mL of isooctane. After shaking for 1 min, decant the lower layer into a 100-mL volumetric flask with the aid of a glass funnel. Rinse the 150-mL beaker with two 15-mL portions of *Solution A*, extracting the remaining isooctane in the separator with each portion and decanting the lower layer from each extraction into the 100-mL volumetric flask. Dilute with *Solution A* to volume. Pass through a suitable membrane filter before injecting.

Chromatographic system

(See *Chromatography* <621>, *System Suitability*.)

Mode: LC

Detector: UV 254 nm

Column: 4-mm × 30-cm; packing L1

Flow rate: 1.5 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

[NOTE—The retention time for dexamethasone phosphate is about 8.5 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 Cream taken:

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

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* (µg/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%

PERFORMANCE TESTS

- **MINIMUM FILL** <755>: Meets the requirements

SPECIFIC TESTS

- **MICROBIAL ENUMERATION TESTS** <61> and **TESTS FOR SPECIFIED MICROORGANISMS** <62>: Meets the requirements of the tests for absence of *Staphylococcus aureus* and *Pseudomonas aeruginosa*

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in collapsible tubes or tight containers.

Change to read:

- **USP REFERENCE STANDARDS** <11>

USP Dexamethasone RS

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