Alclometasone Dipropionate Cream

DEFINITION
Alclometasone Dipropionate Cream contains NLT 90.0% and NMT 110.0% of the labeled amount of alclometasone dipropionate ($C_{28}H_{37}ClO_7$) in a suitable cream base.

IDENTIFICATION
- **A.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, both relative to the internal standard, as obtained in the *Assay*.
- **B. Thin-Layer Chromatographic Identification Test ⟨201⟩**

Standard solution: 0.08 mg/mL of USP Alclometasone Dipropionate RS in methanol

Sample solution: Place a quantity of Cream, equivalent to 1.25 mg of alclometasone dipropionate, in a 50-mL centrifuge tube, and add 15 mL of methanol. Insert a stopper securely into the tube, and place the tube in a water bath maintained at 60° until the semisolid components melt. Remove the tube from the bath, shake vigorously until the specimen components resolidify, and place the tube in an ice–methanol bath for 15 min. Remove the tube from the bath, and centrifuge at 2500 rpm for 5 min. Transfer the clear supernatant to a vial, and allow to equilibrate to room temperature.

Chromatographic system
(See *Chromatography ⟨621⟩, Thin-Layer Chromatography.*)
Adsorbent: 0.25-mm layer of chromatographic silica gel mixture
Application volume: 20 µL
Developing solvent system: Chloroform and acetone (7:1)

Analysis
Samples: *Standard solution* and *Sample solution*
Dry the applications with the aid of a stream of nitrogen, and develop the chromatograms in a saturated, unlined chromatographic chamber. When the solvent front has moved three-fourths of the length of the plate, remove the plate from the chamber, mark the solvent front, and allow the solvent to evaporate. Observe the plate under short-wavelength UV light.

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

ASSAY
- **Procedure**

Buffer: 6.80 g/L of monobasic potassium phosphate (0.05 M)
Mobile phase: Methanol and *Buffer* (2:1)
Internal standard solution: 0.4 mg/mL of betamethasone dipropionate in methanol
Standard stock solution: 0.25 mg/mL of USP Alclometasone Dipropionate RS in methanol
Standard solution: 0.08 mg/mL of USP Alclometasone Dipropionate RS obtained by combining, in a small stoppered flask, 5.0 mL of *Standard stock solution*, 5.0 mL of methanol, and 5.0 mL of *Internal standard solution*
Sample solution: Transfer a quantity of Cream, equivalent to 1.25 mg of alclometasone dipropionate, to a 50-mL centrifuge tube. Add 5.0 mL of *Internal standard solution* and 10.0 mL of methanol. Insert a stopper securely into the tube, and place it in a water bath maintained at 60° until the semisolid components melt. Remove the tube from the bath, shake vigorously until the specimen components resolidify, and return the tube to the 60° water bath until the semisolid components melt. Remove the tube from the bath, shake vigorously until the specimen components resolidify, and place the tube in an ice–methanol bath for 15 min. Remove the tube from the bath, and centrifuge at 2500 rpm for 5 min. Transfer the clear supernatant to a small stoppered flask, and allow to equilibrate to room temperature.

Chromatographic system
(See *Chromatography ⟨621⟩, System Suitability.*)
Mode: LC
Detector: UV 254 nm
Column: 4-mm × 30-cm; packing L1
Flow rate: 1.2 mL/min
Injection volume: 20 µL

System suitability
Sample: *Standard solution*
[NOTE—The relative retention times for alclometasone dipropionate and betamethasone dipropionate are about 0.7 and 1.0, respectively.]
Suitability requirements
Resolution: NLT 3.0 between the analyte and internal standard peaks
Relative standard deviation: NMT 2%

Analysis
Samples: *Standard solution* and *Sample solution*
Calculate the percentage of the labeled amount of alclometasone dipropionate ($C_{28}H_{37}ClO_7$) in the portion of Cream taken:

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

R_U = peak height ratio of alclometasone dipropionate to the internal standard from the *Sample solution*
R_S = peak height ratio of alclometasone dipropionate to the internal standard from the *Standard solution*
C_S = concentration of USP Alclometasone Dipropionate RS in the *Standard solution* (mg/mL)
C_U = nominal concentration of alclometasone dipropionate in the *Sample solution* (mg/mL)

Acceptance criteria: 90.0%–110.0%

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, and store at controlled room temperature.
- **USP Reference Standards ⟨11⟩**
  USP Alclometasone Dipropionate RS

Alclometasone Dipropionate Ointment

DEFINITION
Alclometasone Dipropionate Ointment contains NLT 90.0% and NMT 110.0% of the labeled amount of alclometasone dipropionate ($C_{28}H_{37}ClO_7$) in a suitable ointment base.

IDENTIFICATION
- **A.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, both relative to the internal standard, as obtained in the *Assay*.