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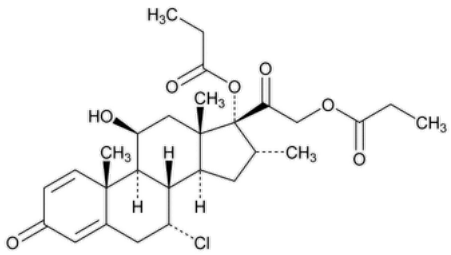
Alclometasone Dipropionate



$C_{28}H_{37}ClO_7$ 521.04

Pregna-1,4-diene-3,20-dione, 7-chloro-11-hydroxy-16-methyl-17,21-bis(1-oxopropoxy)-, (7α,11β,16α)-;

7α-Chloro-11β,17,21-trihydroxy-16α-methylpregna-1,4-diene-3,20-dione 17,21-dipropionate CAS RN®: 66734-13-2; UNII: S56PQL4N1V.

DEFINITION

Alclometasone Dipropionate contains NLT 97.0% and NMT 102.0% of $C_{28}H_{37}ClO_7$, calculated on the dried basis.

IDENTIFICATION

Change to read:

• **A.** ▲**Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: 197M**▲ (CN 1-May-2020)

• **B.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, both relative to the *Internal standard solution*, as obtained in the *Assay*.

ASSAY

• **Procedure**

Solution A: 6.80 mg/mL of monobasic potassium phosphate (0.05 M)

Mobile phase: Methanol and *Solution A* (2:1)

Internal standard solution: 2 mg/mL of betamethasone dipropionate in methanol

Standard stock solution: 1.2 mg/mL of USP Alclometasone Dipropionate RS in methanol

Standard solution: 4.0 mL of *Standard stock solution* and 4.0 mL of *Internal standard solution*. Dilute with methanol to 25 mL. [Note—This solution contains approximately 0.2 mg/mL of USP Alclometasone Dipropionate RS.]

Sample stock solution: 1.2 mg/mL of Alclometasone Dipropionate in methanol

Sample solution: 4 mL of *Sample stock solution* and 4 mL of *Internal standard solution*. Dilute with methanol to 25 mL.

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 size: 10 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 the internal standard peaks

Relative standard deviation: NMT 2%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of $C_{28}H_{37}ClO_7$ in the portion of Alclometasone Dipropionate taken:

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

R_U = peak height ratio from the *Sample solution*

R_S = peak height ratio from the *Standard solution*

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

C_U = concentration of the *Sample solution* (mg/mL)

Acceptance criteria: 97.0%–102.0% on the dried basis

IMPURITIES

Inorganic Impurities

- **Residue on Ignition ⟨281⟩:** NMT 0.1%

Organic Impurities

- **Procedure**

Mobile phase: Acetonitrile and water (3:2)

Diluent: Acetonitrile and water (2:1)

System suitability solution: 1.5 mg/mL of USP Alclometasone Dipropionate RS and 0.015 mg/mL of USP Alclometasone Dipropionate Related Compound A RS in *Diluent*

Sample solution: 1.5 mg/mL of Alclometasone Dipropionate in *Diluent*

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 15-cm; 5-µm packing L1

Flow rate: 1 mL/min

Injection size: 5 µL

Run time: Three times the retention time of alclometasone

System suitability

Sample: *System suitability solution*

Suitability requirements

Tailing factor: NMT 1.5 for alclometasone dipropionate

Relative standard deviation: NMT 2.0% for alclometasone dipropionate

Resolution: NLT 2.0 between alclometasone dipropionate and alclometasone dipropionate related compound A

Analysis

Sample: *Sample solution*

Calculate the percentage of each impurity in the portion of Alclometasone Dipropionate taken:

$$\text{Result} = (r_U/r_T) \times (1/F) \times 100$$

r_U = peak area for each impurity from the *Sample solution*

r_T = sum of all the peaks from the *Sample solution*

F = relative response factor (see *Impurity Table 1*)

Acceptance criteria

Individual impurities: See *Impurity Table 1*.

Total impurities: NMT 2.0%

Impurity Table 1

Name	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (%)
Alclometasone dipropionate	1.0	—	—
Alclometasone dipropionate related compound A[a]	1.2	0.93	1.0
2-Bromo aclometasone dipropionate[b]	1.7	0.91	0.5
Any individual, unspecified impurity	—	1.0	0.10

[a] 11β,17,21-Trihydroxy-16α-methylpregna-1,4-diene-3,20-dione 17,21-dipropionate.

[b] 2-Bromo-7α-chloro-11β,17,21-trihydroxy-16α-methylpregna-1,4-diene-3,20-dione 17,21-dipropionate.

SPECIFIC TESTS

- **Optical Rotation,** *Specific Rotation*⟨781S⟩

  Sample solution: 30 mg/mL in dioxane

  Acceptance criteria: +21° to +25°

- **Loss on Drying** ⟨731⟩**:** Dry a sample in a vacuum at a pressure not exceeding 5 mm of mercury at 105° for 3 h: it loses NMT 0.5% of its weight.

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight containers, and store at controlled room temperature.
- **USP Reference Standards** ⟨11⟩

  USP Alclometasone Dipropionate RS

  USP Alclometasone Dipropionate Related Compound A RS

  11β,17,21-Trihydroxy-16α-methylpregna-1,4-diene-3,20-dione 17,21-dipropionate.
  $C_{28}H_{38}O_7$ 486.60

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

Topic/Question	Contact	Expert Committee
ALCLOMETASONE DIPROPIONATE	Documentary Standards Support	SM52020 Small Molecules 5
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM52020 Small Molecules 5

Chromatographic Database Information: Chromatographic Database

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