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Mometasone Furoate Ointment

DEFINITION

Mometasone Furoate Ointment is Mometasone Furoate in a suitable ointment base. It contains NLT 90.0% and NMT 110.0% of the labeled amount of mometasone furoate ($C_{27}H_{30}Cl_2O_6$).

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 Chromatography**

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

Standard solution: 0.6 mg/mL of USP Mometasone Furoate RS in methanol

Sample solution: Transfer the equivalent to 3 mg of mometasone furoate from Ointment to a 50-mL screw-capped centrifuge tube. Pipet 5.0 mL of methanol into the tube, and attach the cap. Heat in a steam bath until the Ointment completely melts, and shake vigorously until the Ointment resolidifies. Place in an ice-water bath for 10 min. Centrifuge, and filter a portion of the supernatant. Extract 1 mL of the filtrate with 1 mL of hexane, and use the lower phase.

Adsorbent: 0.25-mm layer of chromatographic silica gel mixture

Application volume: 10 µL

Developing solvent system A: Methanol

Developing solvent system B: Chloroform and ethyl acetate (3:1)

Analysis

Samples: *Standard solution* and *Sample solution*

Allow the spots to dry, and develop the chromatogram in *Developing solvent system A* until the solvent front has moved 2 cm from the origin. Remove the plate from the developing chamber, and air-dry. Develop the chromatogram in *Developing solvent system B* until the solvent front has moved three-fourths of the length of the plate. Remove the plate from the developing chamber, mark the solvent front, and allow the spots to air-dry. Examine the plate under short-wavelength UV light.

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

ASSAY

• **Procedure**

[Note—Protect from light.]

Diluent A: Tetrahydrofuran and glacial acetic acid (100:1)

Diluent B: Acetonitrile, water, and glacial acetic acid (50:50:1)

Solution A: Water

Solution B: Acetonitrile

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	70	30
2	70	30
45	45	55

Time (min)	Solution A (%)	Solution B (%)
46	70	30
50	70	30

Internal standard solution: 1.4 mg/mL of diethyl phthalate in acetonitrile

Standard stock solution: 0.2 mg/mL of USP Mometasone Furoate RS in *Diluent A*

Standard solution: 0.05 mg/mL of mometasone furoate and 0.35 mg/mL of diethyl phthalate from equal quantities of the *Standard stock solution* and the *Internal standard solution,* in *Diluent B*

Sample solution: Transfer a portion of Ointment, equivalent to 1.0 mg of mometasone furoate, to a 50-mL screw-capped centrifuge tube. Add 5.0 mL of *Diluent A* and a few glass beads, and mix on a vortex mixer. Add 5.0 mL of *Internal standard solution*, and mix. Add 10.0 mL of *Diluent B,* mix on a vortex mixer for 1 min, and centrifuge for 10 min. Pass the aqueous phase through a polypropylene filter of 0.2-µm pore size, discarding the first 1–2 mL of filtrate.

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 25-cm; 5-µm packing L60

Flow rate: 2 mL/min

Injection size: 20 µL

System suitability

Sample: *Standard solution*

[Note—The relative retention times for diethyl phthalate and mometasone furoate are 0.4 and 1.0, respectively.]

Suitability requirements

Tailing factor: NMT 1.5 for the mometasone furoate peak

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of mometasone fuorate ($C_{27}H_{30}Cl_2O_6$) in the portion of Ointment taken:

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

R_U = ratio of the mometasone furoate peak response to the diethyl phthalate peak response from the *Sample solution*

R_S = ratio of the mometasone furoate peak response to the diethyl phthalate peak response from the *Standard solution*

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

C_U = nominal concentration of mometasone furoate in the *Sample solution* (mg/mL)

Acceptance criteria: 90.0%–110.0%

IMPURITIES

• **Organic Impurities**

[Note—Protect from light.]

Diluent A, Solution A, Solution B, Mobile phase, and **Standard stock solution:** Prepare as directed in the *Assay*.

Diluent C: Acetonitrile, water, and glacial acetic acid (30:70:1)

System suitability solution: 0.1 µg/mL of USP Mometasone Furoate RS from *Standard stock solution* in *Diluent C*

Sample solution: Transfer a portion of Ointment, equivalent to 2.0 mg of mometasone furoate, to a 50-mL screw-capped centrifuge tube. Add 5.0 mL of *Diluent A* and a few glass beads, and mix on a vortex mixer. Add 15.0 mL of *Diluent C*, and mix. Centrifuge for 10 min. Pass the aqueous phase through a polypropylene filter of 0.2-µm pore size, discarding the first 1–2 mL of filtrate.

Blank solution: *Diluent C* and *Diluent A* (3:1)

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 25-cm; 5-µm packing L60

Column temperature: 25 ± 5°

Flow rate: 2 mL/min

Injection size: 50 µL

System suitability

Sample: *System suitability solution*

Suitability requirements

Relative standard deviation: NMT 10%

Analysis

Samples: *System suitability solution*, *Sample solution*, and *Blank solution*

[NOTE—Exclude any peak areas less than those from the chromatogram of the *System suitability solution.* Also exclude any peaks with the same retention time as that observed in the chromatogram of the *Blank solution.* Any peaks having a relative retention time of about 1.04 or 1.13 are controlled in the monograph for *Mometasone Furoate*, and therefore are not included in the total specified and unspecified impurities limit.]

Calculate the percentage of each impurity in the portion of Ointment taken:

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

r_U = peak response of each impurity from the *Sample solution*

r_T = sum of all the peak responses from the *Sample solution*

Acceptance criteria: See *Table 2*.

Table 2

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
9α-Chloro-11β,17,21-trihydroxy-16α-methylpregna-1,4-diene-3,20-dione 17-(2-furoate)	0.56	0.2
9α,21-Dichloro-11β,17-dihydroxy-16α-methylpregna-1,4-diene-3,20-dione	0.73	0.2
21-Chloro-17-hydroxy-16α-methylpregna-1,4-diene-3,11,20-trione 17-(2-furoate)	0.88	0.2
21-Chloro-9β,11β-epoxy-17-hydroxy-16α-methylpregna-1,4-diene-3,20-dione 17-(2-furoate)	0.94	1.0
Mometasone furoate	1.0	—
Unspecified individual impurity	—	0.2
Total specified and unspecified impurities	—	1.0

PERFORMANCE TESTS

• **MINIMUM FILL ⟨755⟩:** Meets the requirements

SPECIFIC TESTS

• **MICROBIAL ENUMERATION TESTS ⟨61⟩** and **TESTS FOR SPECIFIED MICROORGANISMS ⟨62⟩:** It meets the requirements of the tests for absence of *Staphylococcus aureus*, *Pseudomonas aeruginosa*, *Escherichia coli*, and *Salmonella* species.

ADDITIONAL REQUIREMENTS

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

  USP Mometasone Furoate RS

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

Topic/Question	Contact	Expert Committee
MOMETASONE FUROATE 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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