phy ⟨621⟩) coated with a 0.25-mm layer of chromatographic silica gel mixture. Allow the spots to dry, protect the chromatogram from light, and develop in a solvent system consisting of a mixture of alcohol and 1 N hydrochloric acid (95:5). Remove the plate from the developing chamber, mark the solvent front, and allow the solvent to evaporate. Locate the spots on the plate by spraying with Dragendorff's reagent, prepared as directed for *Visualization Technique 3* under *Ordinary Impurities* ⟨466⟩: the R_F value of the principal spot obtained from the test solution corresponds to that obtained from the Standard solution.

Uniformity of dosage units ⟨905⟩: meet the requirements.

Dissolution ⟨711⟩—

Medium: 0.1 N hydrochloric acid; 900 mL.

Apparatus 1: 100 rpm.

Time: 30 minutes.

Solvent A—Mix 300 mL of methanol and 700 mL of 0.1 N hydrochloric acid.

Solvent B—Mix 75 mL of methanol and 25 mL of 0.1 N hydrochloric acid.

Standard solution—Transfer about 100 mg of USP Molindone Hydrochloride RS, accurately weighed, to a 250-mL volumetric flask, and dissolve in and dilute with *Solvent A* to volume. Pipet 5.0 mL of this stock solution into a 250-mL volumetric flask, and dilute with *Solvent A* to volume. Pipet 15.0 mL of the diluted stock solution into a 50-mL volumetric flask, and dilute with *Solvent A* to volume.

Test solution—Withdraw a portion of the solution under test, and filter, discarding the first 3 mL of filtrate. Pipet 15.0 mL of this solution into a 25-mL volumetric flask, and dilute with *Solvent B* to volume.

Mobile phase—Dissolve 1.08 g of sodium 1-octanesulfonate in 480 mL of water. Add 520 mL of methanol, 2.0 mL of acetic acid, and 0.4 mL of triethylamine, and mix. Make adjustments if necessary (see *System Suitability* under *Chromatography* ⟨621⟩).

Chromatographic system (see *Chromatography* ⟨621⟩)—The liquid chromatograph is equipped with a 254-nm UV detector and a 4.6-mm × 25-cm column that contains packing L11. The flow rate is about 1.5 mL per minute.

Procedure—Separately inject equal volumes (about 100 µL) of the *Standard solution* and the *Test solution* into the chromatograph, record the chromatograms, and measure the peak heights. Determine the amount of molindone hydrochloride ($C_{16}H_{24}N_2O_2 \cdot HCl$) dissolved.

Tolerances—Not less than 80% *(Q)* of the labeled amount of $C_{16}H_{24}N_2O_2 \cdot HCl$ is dissolved in 30 minutes.

Assay—

Mobile phase, *Solvent mixture*, *Internal standard solution*, *Standard preparation,* and *Chromatographic system*—Proceed as directed in the *Assay* under *Molindone Hydrochloride*.

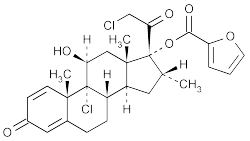
Assay preparation—Accurately weigh not less than 20 Tablets, grind the Tablets to a homogeneous mixture, and transfer an accurately weighed portion, equivalent to about 50 mg of molindone hydrochloride, to a 250-mL conical flask. Add 10.0 mL of *Internal standard solution* and 90.0 mL of *Solvent mixture*, shake for 30 minutes, and filter.

Procedure—Proceed as directed for *Procedure* in the *Assay* under *Molindone Hydrochloride*. Calculate the quantity, in mg, of molindone hydrochloride ($C_{16}H_{24}N_2O_2 \cdot HCl$) in the portion of Tablets taken by the formula:

$$100C(R_U / R_S)$$

in which C is the concentration, in mg per mL, of USP Molindone Hydrochloride RS in the *Standard preparation,* and R_U and R_S are the ratios of the peak response of molindone to that of butylparaben obtained from the *Assay preparation* and the *Standard preparation,* respectively.

Mometasone Furoate



$C_{27}H_{30}Cl_2O_6$ 521.43

Pregna-1,4-diene-3,20-dione, 9,21-dichloro-17-[(2-furanylcarbonyl)oxy]-11-hydroxy-16-methyl-, (11β,16α)-.

9,21-Dichloro-11β,17-dihydroxy-16α-methylpregna-1,4-diene-3,20-dione 17-(2-furoate) [83919-23-7].

» Mometasone Furoate contains not less than 97.0 percent and not more than 102.0 percent of $C_{27}H_{30}Cl_2O_6$, calculated on the dried basis.

Packaging and storage—Preserve in well-closed containers.

USP Reference standards ⟨11⟩—

USP Mometasone Furoate RS

Identification—

A: *Infrared Absorption* ⟨197M⟩.

B: The retention time of the major peak in the chromatogram of the *Assay preparation* corresponds to that of the *Standard preparation*, both relative to the internal standard, as obtained in the *Assay*.

Specific rotation ⟨781S⟩: between +56° and +62°.

Test solution: 5 mg per mL in dioxane.

Loss on drying ⟨731⟩—Dry it at 105° for 3 hours: it loses not more than 0.5% of its weight.

Residue on ignition ⟨281⟩: not more than 0.1%.

Heavy metals, *Method II* ⟨231⟩: 30 ppm.

Chromatographic purity—

Standard solutions—Dissolve an accurately weighed quantity of USP Mometasone Furoate RS, and dilute quantitatively with dichloromethane to obtain a solution containing 10 mg per mL. Dilute portions of this solution with dichloromethane to obtain *Standard solutions A*, *B*, *C*, *D*, and *E* containing 0.5 (5%), 0.2 (2%), 0.1 (1%), 0.02 (0.2%), and 0.01 (0.1%) mg per mL, respectively.

Test solution—Prepare a solution of Mometasone Furoate in dichloromethane containing 10 mg per mL.

Procedure—Separately apply 40 µL of the *Test solution*, and *Standard solutions A*, *B*, *C*, *D*, and *E* to a thin-layer chromatographic plate (see *Chromatography* ⟨621⟩) coated with a 0.25-mm layer of chromatographic silica gel. Develop the chromatogram in a chamber, previously equilibrated with a solvent system consisting of a mixture of chloroform and ethyl acetate (3:1), until the solvent front has moved about three-fourths of the length of the plate. Remove the plate from the developing chamber, mark the solvent front, and air-dry. Examine the plate under short-wavelength UV light. Compare the intensities of any secondary spots observed in the chromatogram of the *Test solution* with those of the principal spots in the chromatogram of the *Standard solutions:* no secondary spot from the chromatogram of the *Test solution* is larger or more intense than the principal spot obtained from *Standard solution C*, and the sum of the intensities of the secondary spots obtained from the *Test solution* is not more than 2.0%.

Assay—

Mobile phase—Prepare a filtered and degassed mixture of methanol and water (65:35). Make adjustments if necessary (see *System Suitability* under *Chromatography* ⟨621⟩).

Diluting solution—Prepare a solution consisting of a mixture of methanol, water, and acetic acid (65:35:0.2).

Internal standard solution—Transfer about 40 mg of beclomethasone dipropionate to a 100-mL volumetric flask, dilute with *Diluting solution* to volume, and mix.

Standard preparation—Dissolve an accurately weighed quantity of USP Mometasone Furoate RS in methanol, and dilute quantitatively, and stepwise if necessary, with *Diluting solution* to obtain a solution having a known concentration of about 0.1 mg per mL. Pipet equal amounts of this solution and the *Internal standard solution*, and dilute quantitatively, and stepwise if necessary, with *Diluting solution* to obtain a solution having a known concentration of about 0.02 mg per mL for mometasone furoate and 0.08 mg per mL for beclomethasone dipropionate.

Assay preparation—Dissolve an accurately weighed quantity of Mometasone Furoate in methanol, and dilute quantitatively, and stepwise if necessary, with *Diluting solution* to obtain a solution having a concentration of about 0.1 mg per mL. Pipet 10.0 mL of this solution and 10.0 mL of the *Internal standard solution* into a 50-mL volumetric flask, dilute with *Diluting solution* to volume, and mix.

Chromatographic system (see *Chromatography* ⟨621⟩)—The liquid chromatograph is equipped with a 254-nm detector and a 4.6-mm × 25-cm column that contains packing L7. The flow rate is about 1.7 mL per minute. Chromatograph the *Standard preparation*, and record the peak responses as directed under *Procedure*: the relative retention times are about 1.6 for beclomethasone dipropionate and 1.0 for mometasone furoate, the resolution, *R,* between the mometasone furoate and beclomethasone dipropionate peaks is not less than 4.0, the tailing factor for the mometasone furoate peak is not more than 1.8, and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure—Separately inject equal volumes (about 20 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the quantity, in mg, of $C_{27}H_{30}Cl_2O_6$ in the portion of Mometasone Furoate taken by the formula:

$$1000C(R_U / R_S)$$

in which C is the concentration, in mg per mL, of USP Mometasone Furoate RS in the *Standard preparation*, and R_U and R_S are the ratios of the mometasone furoate peak to the internal standard peak obtained from the *Assay preparation* and the *Standard preparation*, respectively.

Mometasone Furoate Cream

DEFINITION

Mometasone Furoate Cream is Mometasone Furoate in a suitable cream 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 CHROMATOGRAPHIC IDENTIFICATION TEST ⟨201⟩**
  Standard solution: 0.2 mg/mL of USP Mometasone Furoate RS in acetonitrile
  Sample solution: 0.2 mg/mL of mometasone furoate from Cream in acetonitrile
  Developing solvent system: Chloroform and ethyl acetate (3:1)
  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
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 Cream, 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 furoate ($C_{27}H_{30}Cl_2O_6$) in the portion of Cream 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)