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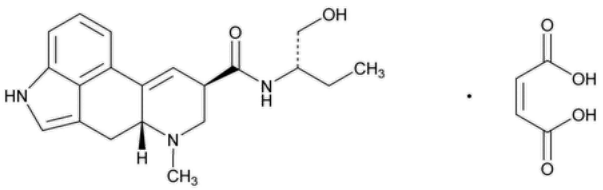
Methylergonovine Maleate



$C_{20}H_{25}N_3O_2 \cdot C_4H_4O_4$ 455.50

Ergoline-8-carboxamide, 9,10-didehydro-*N*-[1-(hydroxy methyl)propyl]-6-methyl-, [8β(*S*)]-, (*Z*)-2-butenedioate (1:1) (salt); 9,10-Didehydro-*N*-[(*S*)-1-(hydroxymethyl)propyl]-6-methylergoline-8β-carboxamide maleate (1:1) (salt) CAS RN®: 57432-61-8; UNII: IR84JPZ1RK.

DEFINITION

Methylergonovine Maleate contains NLT 97.0% and NMT 103.0% of methylergonovine maleate ($C_{20}H_{25}N_3O_2 \cdot C_4H_4O_4$), calculated on the dried basis.

IDENTIFICATION

Change to read:

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

• **B.** The R_F values of the principal fluorescent spot and the principal blue spot of the *Sample solution* correspond to those of the *Standard stock solution*, as obtained in the test for *Related Alkaloids*.

ASSAY

• **Procedure**

Conduct this procedure with a minimum exposure to light.

Mobile phase: Acetonitrile and 2.0 g/L of monobasic potassium phosphate (1:4)

Diluent: 2.5 mg/mL of tartaric acid prepared as follows. Transfer a suitable amount of tartaric acid to a suitable volumetric flask, add 50% of the flask volume of water, and mix with shaking. Dilute with methanol to volume. Allow the mixture to cool before use.

Standard stock solution: 0.1 mg/mL of USP Methylergonovine Maleate RS in *Diluent*. Shake by mechanical means for 15 min.

Standard solution: 4 µg/mL of USP Methylergonovine Maleate RS from the *Standard stock solution* in *Diluent*

Sample stock solution: 0.2 mg/mL of Methylergonovine Maleate in *Diluent*. Shake by mechanical means for 15 min or until completely dissolved.

Sample solution: 4 µg/mL of Methylergonovine Maleate from the *Sample stock solution* in *Diluent*

Chromatographic system

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

Mode: LC

Detector: Fluorescence with excitation at 315 nm and emission at 423 nm

Column: 4.6-mm × 25-cm; packing L7

Temperature: 30°

Flow rate: 2 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 2.0

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of methylergonovine maleate ($C_{20}H_{25}N_3O_2 \cdot C_4H_4O_4$) in the portion of Methylergonovine Maleate taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

C_S = concentration of USP Methylergonovine Maleate RS in the *Standard solution* (µg/mL)

C_U = concentration of Methylergonovine Maleate in the *Sample solution* (µg/mL)

Acceptance criteria: 97.0%–103.0% on the dried basis

IMPURITIES

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

• **Related Alkaloids**

Conduct this test promptly, without exposure to daylight and with minimum exposure to artificial light. Solutions containing methylergonovine maleate should be prepared immediately before use.

Diluent: Alcohol and ammonium hydroxide (9:1)

Standard stock solution: 10 mg/mL of USP Methylergonovine Maleate RS in *Diluent*

Standard solution A: 0.20 mg/mL of USP Methylergonovine Maleate RS from the *Standard stock solution* in *Diluent*

Standard solution B: 0.10 mg/mL of USP Methylergonovine Maleate RS from the *Standard stock solution* in *Diluent*

Standard solution C: 0.05 mg/mL of USP Methylergonovine Maleate RS from the *Standard stock solution* in *Diluent*

Sample solution: 10 mg/mL of Methylergonovine Maleate in *Diluent*

Chromatographic system

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

Mode: TLC

Adsorbent: 0.25-mm layer of chromatographic silica gel mixture

Application volume: 5 µL

Developing solvent system: Chloroform, methanol, and water (75:25:3), equilibrated for 30 min

Spray reagent: 10 mg/mL of *p*-dimethylaminobenzaldehyde in a cooled mixture of alcohol and hydrochloric acid (1:1)

Analysis

Samples: *Standard stock solution*, *Standard solution A*, *Standard solution B*, *Standard solution C*, and *Sample solution*

Proceed as directed in the chapter. Locate the spots on the plate by spraying thoroughly and evenly with *Spray reagent*. Immediately dry in a stream of nitrogen for 2 min.

Acceptance criteria: The R_F value of the principal spot of the *Sample solution* corresponds to that of the *Standard stock solution*. Estimate the concentration of any other spots observed from the *Sample solution* by comparison with *Standard solution A*, *Standard solution B*, and *Standard solution C*. The spots from *Standard solution A*, *Standard solution B*, and *Standard solution C* are equivalent to 2.0%, 1.0%, and 0.50% of impurities, respectively. The sum of the impurities is NMT 2.0%.

SPECIFIC TESTS

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

Sample solution: 5 mg/mL of Methylergonovine Maleate in water

Acceptance criteria: +44° to +50°

• **pH ⟨791⟩**

Sample solution: 0.2 mg/mL of Methylergonovine Maleate in water

Acceptance criteria: 4.4–5.2

• **Loss on Drying ⟨731⟩**

Analysis: Dry under vacuum at 80° to constant weight.

Acceptance criteria: NMT 2.0%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight, light-resistant containers, and store in a cold place.

• **USP Reference Standards ⟨11⟩**

USP Methylergonovine Maleate RS

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

Topic/Question	Contact	Expert Committee
METHYLERGONOVINE MALEATE	Documentary Standards Support	SM52020 Small Molecules 5

Chromatographic Database Information: Chromatographic Database

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