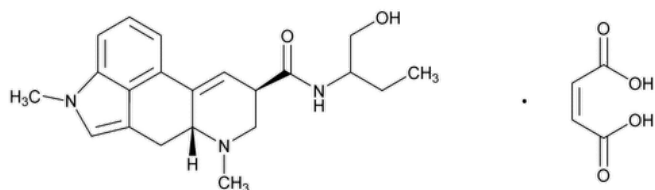


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Methysergide Maleate



$C_{21}H_{27}N_3O_2 \cdot C_4H_4O_4$ 469.53

Ergoline-8-carboxamide, 9,10-didehydro-*N*-[1-(hydroxy methyl)propyl]-1,6-dimethyl-, (8β)-, (*Z*)-2-butenedioate (1:1) (salt);
 9,10-Didehydro-*N*-[1-(hydroxymethyl)propyl]-1,6-dimethylergoline-8β-carboxamide maleate (1:1) (salt) CAS RN®: 129-49-7; UNII: 2U7H1466GH.

DEFINITION

Methysergide Maleate contains NLT 97.0% and NMT 103.0% of methysergide maleate ($C_{21}H_{27}N_3O_2 \cdot C_4H_4O_4$), calculated on the dried basis.

IDENTIFICATION

Change to read:

- **A.** ▲ [SPECTROSCOPIC IDENTIFICATION TESTS \(197\)](#), [Infrared Spectroscopy: 197K](#) ▲ (CN 1-MAY-2020)
- **B. THIN-LAYER CHROMATOGRAPHY**

Conduct this test without exposure to daylight and with minimum exposure to artificial light.

Standard solution: 5 mg/mL of [USP Methysergide Maleate RS](#) in methanol

Sample solution: 5 mg/mL of Methysergide Maleate in methanol

Chromatographic system

(See [Chromatography \(621\)](#), [Thin-Layer Chromatography](#).)

Adsorbent: 0.25-mm layer of chromatographic silica gel

Application volume: 25 µL

Developing solvent: Chloroform and methanol (20:1)

Spray reagent: 8 mg/mL of *p*-dimethylaminobenzaldehyde in a cooled mixture of alcohol and sulfuric acid (8:2)

Analysis

Samples: *Standard solution* and *Sample solution*

In the chromatographic chamber, place a volume of the *Developing solvent* sufficient to develop the chromatogram. Place a beaker containing 25 mL of ammonium hydroxide in the chamber, cover, and allow to equilibrate for 30 min. Apply the *Samples*, and develop the chromatogram 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 allow the solvent to evaporate. Locate the spots on the plate by lightly spraying with *Spray reagent*. Allow the plate to dry, then expose it briefly to fumes of a mixture of nitric and hydrochloric acids.

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

ASSAY

PROCEDURE

Sample solution: 200 mg of Methysergide Maleate in 30 mL of glacial acetic acid. Add 1 drop of crystal violet TS.

Analysis: Titrate with 0.1 N perchloric acid VS to a blue endpoint. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N perchloric acid is equivalent to 46.95 mg of methysergide maleate ($C_{21}H_{27}N_3O_2 \cdot C_4H_4O_4$).

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

IMPURITIES

- [ORDINARY IMPURITIES \(466\)](#)

Standard solution and **Sample solution:** Methanol

Eluant: Use the *Developing solvent* from *Identification* test B.

Visualization: 1

Acceptance criteria: Meets the requirements

SPECIFIC TESTS

- [OPTICAL ROTATION, Specific Rotation\(781S\)](#).
Sample solution: 2.5 mg/mL in water
Acceptance criteria: +35° to +45°
- [pH \(791\)](#).
Sample solution: 1 in 500 in carbon dioxide-free water
Acceptance criteria: 3.7–4.7
- [LOSS ON DRYING \(731\)](#).
Analysis: Dry a sample under vacuum at 120° for 2 h.
Acceptance criteria: NMT 7.0%

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight, light-resistant containers, in a cold place.
- [USP REFERENCE STANDARDS \(11\)](#).
[USP Methysergide Maleate RS](#)

Auxiliary Information - Please [check for your question in the FAQs](#) before contacting USP.

Topic/Question	Contact	Expert Committee
METHYSERGIDE MALEATE	Documentary Standards Support	SM42020 Small Molecules 4

Chromatographic Database Information: [Chromatographic Database](#)

Most Recently Appeared In:

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