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Methylergonovine Maleate Tablets

» Methylergonovine Maleate Tablets contain not less than 90.0 percent and not more than 110.0 percent of the labeled amount of methylergonovine maleate ($C_{20}H_{25}N_3O_2 \cdot C_4H_4O_4$).

Packaging and storage—Preserve in tight, light-resistant containers.

USP Reference standards ⟨11⟩—
USP Methylergonovine Maleate RS

Identification—

A: The R_F values of the principal fluorescent spot and the principal blue spot in the chromatogram of the *Test preparation* correspond to those in the chromatogram of the *Standard preparation*, as obtained in the test for *Related alkaloids* under *Ergonovine Maleate*, using the Tablets instead of Ergonovine Maleate.

B: Transfer a quantity of powdered Tablets, equivalent to about 4 mg of methylergonovine maleate, to a separator, add 20 mL of water, and render alkaline to litmus with sodium carbonate solution (1 in 10). Extract with three 20-mL portions of chloroform, filter the combined chloroform extracts into a small evaporating dish, and evaporate on a steam bath to dryness. Dissolve the residue in a mixture of 6 mL of water and 0.3 mL of hydrochloric acid, and filter, if necessary: the solution so obtained exhibits a bluish fluorescence under UV light. To this solution, add 2 mL of a solution of glacial acetic acid in ethyl acetate (1 in 2), and stratify 2 mL of sulfuric acid, by pipetting, under the solution: a bluish purple ring appears at the interface of the two liquids.

Dissolution ⟨711⟩—
Medium: tartaric acid solution (1 in 200); 900 mL.
Apparatus 2: 75 rpm.
Time: 30 minutes.
Procedure—Filter a portion of the solution under test into a flask. Concomitantly determine the fluorescence intensity of this solution in comparison with a Standard solution of USP Methylergonovine Maleate RS in the same medium having a known concentration of about 0.22 µg per mL in a fluorometer at an excitation wavelength of about 327 nm and an emission wavelength of about 428 nm, using tartaric acid solution (1 in 200) as the blank.
Tolerances—Not less than 70% *(Q)* of the labeled amount of $C_{20}H_{25}N_3O_2 \cdot C_4H_4O_4$ is dissolved in 30 minutes.

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

Related alkaloids—[Note—Conduct this test without exposure to daylight and with the minimum exposure to artificial light.]
Solvent mixture—Mix 75 volumes of chloroform, 25 volumes of methanol, and 1 volume of ammonium hydroxide.
Detecting reagent—Cautiously dissolve 800 mg of *p*-dimethylaminobenzaldehyde in a mixture of alcohol and sulfuric acid (101:11).
Test preparation—Transfer a quantity of finely powdered Tablets, equivalent to 5.0 mg of methylergonovine maleate, to a suitable container, add 50 mL of *Solvent mixture*, and stir with the aid of a magnetic stirrer for 40 minutes. Filter, rinsing the container with two 10-mL portions of *Solvent mixture*. Evaporate the combined filtrates in vacuum at 25° to 30°, and dissolve the residue in 2.0 mL of *Solvent mixture*.
Standard stock solution—Transfer 25 mg of USP Methylergonovine Maleate RS to a 10-mL volumetric flask, add *Solvent mixture* to volume, and mix to obtain a solution having a known concentration of 2.5 mg per mL.
Standard preparations A, B, C, and *D*—Dilute accurately measured volumes of *Standard stock solution* quantitatively with *Solvent mixture* (designated below as parts by volume of *Standard stock solution* in total parts by volume of the finished *Standard preparation*) to obtain *Standard preparations*, designated below by letter, having the following concentrations and percentage assignments:
A: (1 in 20); 125 µg per mL (5.0%).
B: (1 in 33); 75 µg per mL (3.0%).
C: (1 in 100); 25 µg per mL (1.0%).
D: (1 in 200); 12.5 µg per mL (0.5%).
Procedure—Apply separately 20 µL of the *Test preparation* and 20 µL of each *Standard preparation* to a suitable thin-layer chromatographic plate (see *Chromatography* ⟨621⟩) coated with a 0.25-mm layer of chromatographic silica gel mixture. Dry the plate with the aid of a stream of cool air. Position the plate in a chromatographic chamber, and develop the chromatograms in *Solvent mixture* 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 in a stream of cool air. Examine the plate under long-wavelength UV light. Mark the principal and any secondary fluorescent spots. Spray the plate with *Detecting reagent*, and mark the principal and secondary blue spots. Compare the intensities of any secondary spots observed in the chromatogram of the *Test preparation* with those of the principal spots in the chromatograms of the *Standard preparations:* the sum of the intensities of secondary spots obtained from the *Test preparation* corresponds to not more than 5.0% of related compounds.

Assay—[NOTE—Conduct this procedure with a minimum exposure to light.]

Mobile phase, Solvent mixture, Standard preparation, and *Chromatographic system*—Proceed as directed in the *Assay* under *Methylergonovine Maleate*.

Assay preparation—Place 10 Tablets in 1500-mL volumetric flask, add 400 mL of *Solvent mixture*, and shake by mechanical means for 15 minutes or until completely disintegrated. Dilute with *Solvent mixture* to volume, and mix. Allow the solution to settle for not less than 30 minutes before use, and then filter to obtain the *Assay preparation*.

Procedure—Separately inject equal volumes (about 10 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 methylergonovine maleate ($C_{20}H_{25}N_3O_2 \cdot C_4H_4O_4$) in the portion of Tablets taken by the formula:

$$(L/D)(C)(r_U/r_S)$$

in which L is the labeled quantity, in mg, of methylergonovine maleate in each Tablet, D is the concentration, in µg per mL, of methylergonovine maleate in the *Assay preparation*, based on the labeled quantity per Tablet and the extent of dilution, C is the concentration, in µg per mL, of USP Methylergonovine Maleate RS in the *Standard preparation*, and r_U and r_S are the responses obtained from the *Assay preparation* and the *Standard preparation*, respectively.

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

Topic/Question	Contact	Expert Committee
METHYLERGONOVINE MALEATE TABLETS	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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