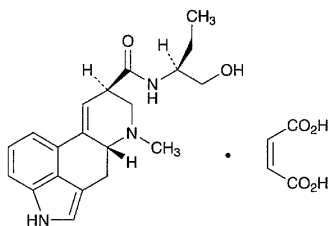


## Methylergometrine Maleate

マレイン酸メチルエルゴメトリン



$C_{20}H_{25}N_3O_2 \cdot C_4H_4O_4$ : 455.50  
(8*S*)-9,10-Didehydro-*N*-[(1*S*)-1-(hydroxymethyl)propyl]-6-methylergoline-8-carboxamide monomaleate [7054-07-1]

Methylergometrine Maleate, when dried, contains not less than 95.0% and not more than 105.0% of  $C_{20}H_{25}N_3O_2 \cdot C_4H_4O_4$ .

**Description** Methylergometrine Maleate occurs as a white to pale yellow, crystalline powder. It is odorless.

It is slightly soluble in water, in methanol and in ethanol (95), and practically insoluble in diethyl ether.

It gradually changes to yellow by light.

Melting point: about 190°C (with decomposition).

**Identification (1)** A solution of Methylergometrine Maleate (1 in 200) shows a blue fluorescence.

(2) The solution obtained in the Assay develops a deep blue in color. Determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelengths.

(3) To 5 mL of a solution of Methylergometrine Maleate (1 in 500) add 1 drop of potassium permanganate TS: the red color of the test solution fades immediately.

**Optical rotation**  $[\alpha]_D^{20}$ : +44 – +50° (after drying, 0.1 g, water, 20 mL, 100 mm).

**Purity** Related substances—Conduct this procedure without exposure to daylight, using light-resistant vessels. Dissolve 8 mg of Methylergometrine Maleate in 2 mL of a mixture of ethanol (95) and ammonia solution (28) (9:1), and use this solution as the sample solution. Pipet 1 mL of the sample solution, add a mixture of ethanol (95) and ammonia solution (28) (9:1) to make exactly 100 mL, and use this solution as the standard solution. Perform the test immediately with these solutions as directed under the Thin-layer Chromatography. Spot 10  $\mu$ L each of the sample solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography, and immediately develop the plate with a mixture of chloroform, methanol and water (75:25:3) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

**Loss on drying** Not more than 2.0% (0.2 g, in vacuum, phosphorus (V) oxide, 4 hours).

**Assay** Weigh accurately about 0.01 g of Methylergometrine Maleate, previously dried, add water to make exactly 250 mL, and use the solution as the sample solution. Separately, weigh accurately about 0.01 g of Ergometrine Maleate Reference Standard, previously dried over silica gel for 4 hours, add water to make exactly 250 mL, and use the solution as the standard solution. Pipet 2 mL each of the sample solution and the standard solution into brown glass-stoppered test tubes, add 4.0 mL each of 4-dimethylaminobenzaldehyde-ferric chloride TS in ice water, and after heating for 10 minutes at 45°C, allow to stand for 20 minutes at room temperature. Perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry, using a solution, prepared with 2 mL of water in the same manner, as the blank. Determine the absorbances,  $A_T$  and  $A_S$ , of the subsequent solutions of the sample solution and the standard solution at 545 nm, respectively.

$$\begin{aligned} & \text{Amount (mg) of methylergometrine} \\ & \text{maleate } (C_{20}H_{25}N_3O_2 \cdot C_4H_4O_4) \\ & = \text{amount (mg) of Ergometrine Maleate} \\ & \text{Reference Standard} \\ & \times \frac{A_T}{A_S} \times 1.0318 \end{aligned}$$

**Containers and storage** Containers—Tight containers.  
Storage—Light-resistant.

## Methylergometrine Maleate Tablets

マレイン酸メチルエルゴメトリン錠

Methylergometrine Maleate Tablets contain not less than 90% and not more than 110% of the labeled amount of  $C_{20}H_{25}N_3O_2 \cdot C_4H_4O_4$ : 455.50.

**Method of preparation** Prepare as directed under Tablets, with Methylergometrine maleate.

**Identification (1)** The sample solution obtained in the Assay shows a blue fluorescence.

(2) The colored solution obtained in the Assay shows a deep blue color. Determine the absorption spectrum of the colored solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits maxima between 543 nm and 547 nm and between 620 nm and 630 nm.

**Dissolution test** Perform the test with 1 tablet of Methylergometrine Maleate Tablets at 100 revolutions per minute according to Method 2 under the Dissolution Test, using 900 mL of water as the test solution. Take 20 mL or more of the dissolved solution 30 minutes after starting the test, and filter through a membrane filter with a pore size of not more than 0.8  $\mu$ m. Discard the first 10 mL of the filtrate, and use the subsequent as the sample solution, or to exactly  $V$  mL of the subsequent add water to make exactly  $V'$  mL so that each mL contains about 0.13  $\mu$ g of methylergometrine maleate ( $C_{20}H_{25}N_3O_2 \cdot C_4H_4O_4$ ) according to the labeled amount, and use this solution as the sample solution. Separately, weigh accurately about 0.025 g of methylergometrine maleate for assay, previously dried in a desiccator for 4 hours (in vacuum, phosphorus (V) oxide), and dissolve in water to