and the same *Rf* value, and any spot from the sample solution other than that corresponding to the spot from the standard solution does not appear.

Loss on drying Not more than 2.0% (0.2 g, silica gel, 4 hours).

Assay Weigh accurately about 0.01 g each of Ergometrine Maleate and Ergometrine Maleate Reference Standard, previously dried in a desiccator (silica gel) for 4 hours, dissolve in water to make exactly 250 mL, and use these solutions as the sample solution and the standard solution, respectively. Pipet 2 mL of each solution into a separate brown glass-stoppered tube. To each tube add 4 mL of 4-dimethylaminobenzaldehyde-ferric chloride TS, exactly measured, while cooling in an ice bath, then warm at 45°C for 10 minutes. Allow to stand at room temperature for 20 minutes, and 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 550 nm, respectively.

Amount (mg) of $C_{19}H_{23}N_3O_2.C_4H_4O_4$
= amount (mg) of Ergometrine Maleate Reference Standard
$\times \frac{A_T}{A_S}$

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

Assay Transfer an exactly measured volume of Ergometrine Maleate Injection, equivalent to about 2 mg of ergometrine maleate ($C_{19}H_{23}N_3O_2.C_4H_4O_4$), and add sodium chloride in a ratio of 0.3 g to 1 mL of the solution. To this mixture add 20 mL of diethyl ether and 2 mL of ammonia TS, shake, and extract. Further, extract with three 15-mL portions of diethyl ether, combine all the extracts, add 5 g of anhydrous sodium sulfate, filter through a pledget of absorbent cotton, and wash with three 5-mL portions of diethyl ether. Add the washings to the filtrate, shake with 5 mL of dilute sulfuric acid, evaporate the diethyl ether by warming in a current of nitrogen, to the remaining solution add water to make exactly 50 mL, and use this solution as the sample solution. Weigh accurately about 2 mg of Ergometrine Maleate Reference Standard, previously dried in a desiccator (silica gel) for 4 hours, add water to make exactly 50 mL, and use this solution as the standard solution. Transfer 2 mL each of the sample solution and the standard solution, accurately measured, to separate glass-stoppered test tubes, and proceed as directed in the Assay under Ergometrine Maleate.

Amount (mg) of ergometrine maleate ($C_{19}H_{23}N_3O_2.C_4H_4O_4$)
= amount (mg) of Ergometrine Maleate Reference Standard
$\times \frac{A_T}{A_S}$

Containers and storage Containers—Hermetic containers, and colored containers may be used.
Storage—Light-resistant, and in a cold place.

Ergometrine Maleate Injection

マレイン酸エルゴメトリン注射液

Ergometrine Maleate Injection is an aqueous solution for injection. It contains not less than 90% and not more than 110% of the labeled amount of ergometrine maleate ($C_{19}H_{23}N_3O_2.C_4H_4O_4$: 441.48).

Method of preparation Prepare as directed under Injections, with Ergometrine Maleate.

Description Ergometrine Maleate Injection is a clear, colorless to pale yellow liquid.
pH: 2.7 - 3.5

Identification **(1)** Measure a volume of Ergometrine Maleate Injection, equivalent to 3 mg of Ergometrine Maleate according to the labeled amount, if necessary, dilute with water or evaporate on a water bath to make 15 mL, and use this solution as the sample solution. The sample solution shows a blue fluorescence.

(2) To 1 mL of the sample solution obtained in (1) add 1 mL of ammonia TS, and extract with 20 mL of diethyl ether. To the diethyl ether extract add 1 mL of dilute sulfuric acid, shake, and warm to remove diethyl ether in a water bath. Cool, to the residue obtained add 2 mL of 4-dimethylaminobenzaldehyde-ferric chloride TS, and allow to stand for 5 to 10 minutes: a deep blue color develops.

(3) To 5 mL of the sample solution obtained in (1) add 1 drop of potassium permanganate TS: a red color disappears immediately.

Ergometrine Maleate Tablets

マレイン酸エルゴメトリン錠

Ergometrine Maleate Tablets contain not less than 90% and not more than 110% of the labeled amount of ergometrine maleate ($C_{19}H_{23}N_3O_2.C_4H_4O_4$: 441.48).

Method of preparation Prepare as directed under Tablets, with Ergometrine Maleate.

Identification To a quantity of powdered Ergometrine Maleate Tablets, equivalent to 3 mg of Ergometrine Maleate according to the labeled amount, add 15 mL of warm water, shake, and filter: the filtrate shows a blue fluorescence. Proceed with this solution as directed in the Identification (2) and (3) under Ergometrine Maleate.

Content uniformity Transfer 1 tablet of Ergometrine Maleate Tablets to a glass-stoppered centrifuge tube, and add a solution of L-tartaric acid (1 in 100) to make exactly *V* mL of a solution containing about 0.04 mg of ergometrine maleate ($C_{19}H_{23}N_3O_2.C_4H_4O_4$) per ml. Stopper the tube, shake for 30 minutes vigorously, centrifuge, and use the supernatant liquid as the sample solution. Separately, weigh accurately about 4 mg of Ergometrine Maleate Reference Standard, previously dried in a desiccator (silica gel) for 4 hours, dissolve in water to make exactly 100 mL, and use this solution as the standard solution. Pipet 4 mL each of the sample solution and the standard solution into separate brown glass-stoppered test tubes, add exactly 8 mL each of 4-

dimethylaminobenzaldehyde-ferric chloride TS while cooling in an ice bath, after shaking, and allow to stand for 1 hour at ordinary temperature. Perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry, using a solution, prepared with 4 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 550 nm, respectively.

Amount (mg) of ergometrine maleate($C_{19}H_{23}N_2O_2.C_4H_4O_4$)
= amount (mg) of Ergometrine Maleate Reference Standard
$\times \frac{A_T}{A_S} \times \frac{V}{100}$

Assay Weigh accurately, and powder not less the 20 Ergometrine Maleate Tablets. Weigh accurately a portion of the powder, equivalent to about 2 mg of ergometrine maleate ($C_{19}H_{23}N_3O_2.C_4H_4O_4$), transfer to a glass filter (G4), add 10 mL of a solution of L-tartaric acid (1 in 100), and filter with thorough shaking. Repeat the procedures 3 times, combine the filtrates, add a solution of L-tartaric acid (1 in 100) to make exactly 50 mL, and use this solution as the sample solution. Separately, weigh accurately about 2 mg of Ergometrine Maleate Reference Standard, previously dried in a desiccator (silica gel) for 4 hours, dissolve in a solution of L-tartaric acid (1 in 100) to make exactly 50 mL, and use this solution as the standard solution. Pipet 2 mL each of the sample solution and the standard solution, and proceed as directed in the Assay under Ergometrine Maleate.

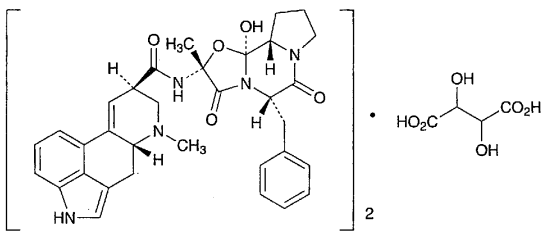
Amount (mg) of ergometrine maleate ($C_{19}H_{23}N_3O_2.C_4H_4O_4$)
= amount (mg) of Ergometrine Maleate Reference Standard
$\times \frac{A_T}{A_S}$

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Ergotamine Tartrate

酒石酸エルゴタミン



$(C_{33}H_{35}N_5O_5)_2.C_4H_6O_6$: 1313.41

(5′*S*)-5′-Benzyl-12′-hydroxy-2′-methylergotaman-3′,6′,18-trione hemitartrate [*379-79-3*]

Ergotamine Tartrate contains not less than 98.0% of $(C_{33}H_{35}N_5O_5)_2.C_4H_6O_6$, calculated on the dried basis.

Description Ergotamine Tartrate occurs as colorless crystals, or a white to pale yellowish white or grayish white, crystalline powder.

It is slightly soluble in water and in ethanol (95).

Melting point: about 180°C (with decomposition).

Identification (1) Dissolve 1 mg of Ergotamine Tartrate in 10 mL of a mixture of acetic acid (100) and ethyl acetate (1:1). To 0.5 mL of this solution add slowly 0.5 mL of sulfuric acid, with shaking in cold water, and allow to stand: a purple color develops. To this solution add 0.1 mL of diluted iron (III) chloride TS (1 in 12): the color of the solution changes to blue to blue-purple.

(2) Dissolve 1 mg of Ergotamine Tartrate in 5 mL of a solution of L-tartaric acid (1 in 100). To 1 mL of this solution add 2 mL of 4-dimethylaminobenzaldehyde-ferric chloride TS, and shake: a blue color develops.

Optical rotation Ergotamine base $[\alpha]_D^{20}$: -155 – $-165°$. Dissolve 0.35 g of Ergotamine Tartrate in 25 mL of a solution of L-tartaric acid (1 in 100), add 0.5 g of sodium hydrogen carbonate, shake gently and sufficiently, and extract with four 10-mL portions of ethanol-free chloroform. Filter the extracts successively through a small filter paper, moistened with ethanol-free chloroform, into a 50-mL volumetric flask. Allow the flask to stand in a water bath at 20°C for 10 minutes, and determine the optical rotation in a 100-mm cell. Separately, pipet 25 mL of this solution, evaporate to dryness under reduced pressure at a temperature not higher than 45°C, dissolve the residue in 25 mL of acetic acid (100), and titrate with 0.05 mol/L perchloric acid VS (indicator: 1 drop of crystal violet TS). Perform a blank determination, and make any necessary correction. Calculate the specific rotation of the ergotamine base from the consumed volume of 0.05 mol/L perchloric acid VS and the optical rotation.

Each mL of 0.05 mol/L perchloric acid VS
= 29.084 mg of $C_{33}H_{35}N_5O_5$

Purity Related substances—Conduct this procedure without exposure to daylight, using light-resistant vessels. To 0.040 g of Ergotamine Tartrate add 10 mL of a solution of L-tartaric acid in diluted methanol (1 in 2) (1 in 1000), dissolve with thorough shaking, and use this solution as the sample solution. Pipet 1 mL of this solution, add a solution of L-tartaric acid in diluted methanol (1 in 2) (1 in 1000) to make exactly 50 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μL each of the sample solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of chloroform and methanol (9:1) to a distance of about 10 cm, and air-dry the plate. Spray evenly 4-dimethylaminobenzaldehyde TS on the plate: 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 5.0% (0.1 g, in vacuum, 60°C, 4 hours).

Assay Weigh accurately about 0.2 g of Ergotamine Tartrate, dissolve in 15 mL of a mixture of acetic acid (100) and acetic anhydride (50:3), and titrate with 0.05 mol/L perchloric acid VS (indicator: 1 drop of crystal violet TS). Perform a blank determination, and make any necessary correction.