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.

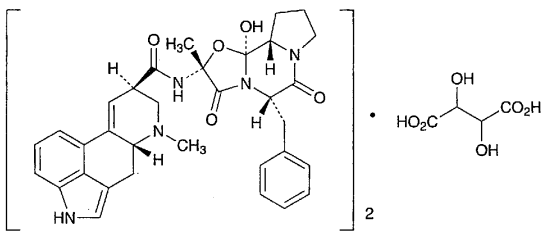
Amount (mg) of ergometrine maleate ($C_{19}H_{23}N_3O_2.C_4H_4O_4$)
= amount (mg) of Ergometrine Maleate Reference Standard
$\times \frac{A_T}{A_S}$

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.

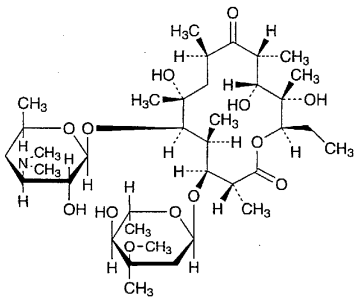
Each mL of 0.05 mol/L perchloric acid VS
= 32.836 mg of $(C_{33}H_{35}N_5O_5)_2.C_4H_6O_6$

Containers and storage Containers—Tight containers.

Storage—Light-resistant, and almost well-filled, or under nitrogen atmosphere, and not exceeding 5°C.

Erythromycin

エリスロマイシン



$C_{37}H_{67}NO_{13}$: 733.93

(2*R*,3*S*,4*S*,5*R*,6*R*,8*R*,10*R*,11*R*,12*S*,13*R*)-5-(3,4,6-Trideoxy-3-dimethylamino-β-D-*xylo*-hexopyranosyloxy)-3-(2,6-dideoxy-3-*C*-methyl-3-*O*-methyl-α-L-*ribo*-hexopyranosyloxy)-6,11,12-trihydroxy-2,4,6,8,10,12-hexamethyl-9-oxopentadecan-13-olide
[*114-07-8*]

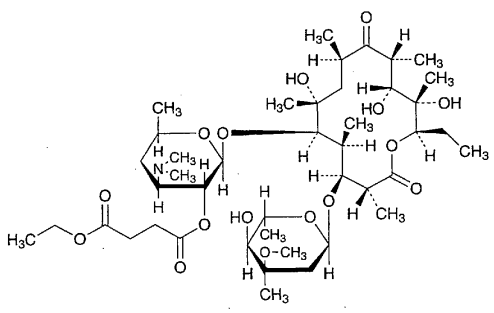
Erythromycin conforms to the requirements of Erythromycin in the Minimum Requirements for Antibiotic Products of Japan.

Description Erythromycin occurs as white to light yellowish white powder. It has a bitter taste.

It is freely soluble in methanol, in ethanol (95) and in acetone, and soluble in diethyl ether, and very slightly soluble in water.

Erythromycin Ethylsuccinate

エチルコハク酸エリスロマイシン



$C_{43}H_{75}NO_{16}$: 862.05

(2*R*,3*S*,4*S*,5*R*,6*R*,8*R*,10*R*,11*R*,12*S*,13*R*)-5-[3,4,6-Trideoxy-2-*O*-(3-ethoxycarbonylpropanoyl)-3-dimethylamino-β-D-*xylo*-hexopyranosyloxy]-3-(2,6-dideoxy-3-*C*-methyl-3-*O*-methyl-α-L-*ribo*-hexopyranosyloxy)-6,11,12-trihydroxy-2,4,6,8,10,12-hexamethyl-9-oxopentadecan-13-olide
[*41342-53-4*]

Erythromycin Ethylsuccinate contains not less than 780 μg (potency) per mg, calculated on the anhydrous basis. The potency of Erythromycin Ethylsuccinate is expressed as mass (potency) of erythromycin ($C_{37}H_{67}NO_{13}$: 733.93).

Description Erythromycin Ethylsuccinate occurs as a white powder.

It is freely soluble in methanol and in acetone, soluble in ethanol (95), and practically insoluble in water.

Identification (1) Dissolve 3 mg of Erythromycin Ethylsuccinate in 2 mL of acetone, and add 2 mL of hydrochloric acid: an orange color develops and is immediately changed to red to deep purple.

(2) Determine the infrared absorption spectrum of Erythromycin Ethylsuccinate, previously dried in a desiccator (reduced pressure, silica gel) for 24 hours, as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

Water Not more than 5.0% (0.5 g, volumetric titration, direct titration).

Assay Perform the test according to the Cylinder-plate method as directed under the Microbial Assay for Antibiotics according to the following conditions.

(1) Test organism—*Staphylococcus aureus* ATCC 6538 P

(2) Culture medium—Use the medium i in 3) Medium for other organisms under (1) Agar media for seed and base layer. Adjust the pH of the medium so that it will be 7.8 to 8.0 after sterilization.

(3) Standard solution—Weigh accurately an amount of Erythromycin Reference Standard equivalent to about 0.05 g (potency), dissolve in 50 mL of methanol, add 0.1 mol/L phosphate buffer solution, pH 8.0 to make exactly 100 mL, and use this solution as the standard stock solution. Keep the standard stock solution at 5°C or below and use within 7 days. Take exactly a suitable amount of the standard stock solution before use, add 0.1 mol/L phosphate buffer solution, pH 8.0 to make solutions so that each mL contains 20 μg (potency) and 5 μg (potency), and use these solutions as the high concentration standard solution and the low concentration standard solution, respectively.

(4) Sample solution—Weigh accurately an amount of Erythromycin Ethylsuccinate equivalent to about 0.05 g (potency), dissolve in 50 mL of methanol, and add 0.1 mol/L phosphate buffer solution, pH 8.0 to make exactly 100 mL. Take exactly a suitable amount of the solution, add 0.1 mol/L phosphate buffer solution, pH 8.0 to make solutions so that each mL contains 20 μg (potency) and 5 μg (potency), and use these solutions as the high concentration sample solution and the low concentration sample solution, respectively.

Containers and storage Containers—Tight containers.