mol/L perchloric acid VS until the color of the solution changes from purple through blue to blue-green (indicator: 2 to 3 drops of crystal violet TS). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 21.122 mg of $C_{10}H_{13}NO_4$

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Methyldopa Tablets

メチルドパ錠

Methyldopa Tablets contain not less than 90% and not more than 110% of the labeled amount of methyldopa ($C_{10}H_{13}NO_4$: 211.21).

Method of preparation Prepare as directed under Tablets, with Methyldopa.

Identification **(1)** To a quantity of powdered Methyldopa Tablets, equivalent to 0.1 g of Methyldopa according to the labeled amount, add 10 mL of water, and heat in a water bath for 5 minutes with occasional shaking. After cooling, centrifuge for 5 minutes at 2000 rotations per minute, apply 1 drop of the supernatant solution to a filter paper, and dry with warm air. Place 1 drop of ninhydrin TS over the spot, and heat for 5 minutes at 100°C: a purple color develops.

(2) To 0.5 mL of the supernatant liquid obtained in the Identification (1) add 2 mL of 0.05 mol/L sulfuric acid TS, 2 mL of iron (II) tartrate TS and 4 drops of ammonia TS, and shake well: a deep purple color develops.

(3) To 0.7 mL of the supernatant liquid obtained in the Identification (1) add 0.1 mol/L hydrochloric acid TS to make 20 mL. To 10 mL of this solution add 0.1 mol/L hydrochloric acid TS to make 100 mL, and determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 277 nm and 283 nm.

Dissolution test Perform the test with 1 tablet of Methyldopa Tablets at 50 revolutions per minute according to Method 2 under the Dissolution Test, using 900 mL of water as the test solution. Take 30 mL or more of the dissolved solution 60 minutes after start of the test, and filter through a membrane filter with pore size of not more than 0.8 μm. Discard the first 10 mL of the filtrate, pipet the subsequent V mL, add water to make exactly V' mL so that each mL contains about 25 μg of methyldopa ($C_{10}H_{13}NO_4$) according to the labeled amount, and use this solution as the sample solution. Separately, weigh accurately about 0.056 g of methyldopa for assay (its loss on drying is determined, separately, at 125°C for 2 hours), and dissolve in water to make exactly 200 mL. Pipet 10 mL of this solution, add water to make exactly 100 mL, and use this solution as the standard solution. Determine the absorbances, A_T and A_S, of the sample solution and the standard solution at 280 nm as directed under the Ultraviolet-visible Spectrophotometry.

The dissolution rate of Methyldopa Tablets in 60 minutes is not less than 75%.

Dissolution rate (%) with respect to the labeled amount of methyldopa ($C_{10}H_{13}NO_4$)

$$= W_S \times \frac{A'}{A_S} \times \frac{V'}{V} \times \frac{1}{C} \times 45$$

W_S: Amount (mg) of methyldopa for assay, calculated on the dried basis.

C: Labeled amount (mg) of methyldopa ($C_{10}H_{13}NO_4$) in 1 tablet.

Assay Weigh accurately and powder not less than 20 Methyldopa Tablets. Weigh accurately a portion of the powder, equivalent to about 0.1 g of methyldopa ($C_{10}H_{13}NO_4$), add 50 mL of 0.05 mol/L sulfuric acid TS, shake thoroughly for 15 minutes, add 0.05 mol/L sulfuric acid TS to make exactly 100 mL, and filter through a dry filter paper. Discard the first 20 mL of the filtrate, and use the subsequent filtrate as the sample solution. Separately, weigh accurately about 0.11 g of Methyldopa Reference Standard (previously dry at 125°C for 2 hours, and determine the loss on drying), dissolve in 0.05 mol/L sulfuric acid TS to make exactly 100 mL, and use this solution as the standard solution. Pipet 5 mL each of the sample solution and the standard solution, add exactly 5 mL of iron (II) tartrate TS, and add ammonia-ammonium acetate buffer solution, pH 8.5, to make exactly 100 mL. Perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry, using a solution prepared with 5 mL of 0.05 mol/L sulfuric acid TS 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 520 nm, respectively.

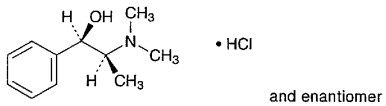
Amount (mg) of methyldopa ($C_{10}H_{13}NO_4$)
= amount (mg) of Methyldopa Reference Standard, calculated on the dried basis

$$\times \frac{A_T}{A_S}$$

Containers and storage Containers—Well-closed containers.

dl-Methylephedrine Hydrochloride

dl-塩酸メチルエフェドリン



and enantiomer

$C_{11}H_{17}NO.HCl$: 215.72
(1*RS*,2*SR*)-2-Dimethylamino-1-phenylpropan-1-ol monohydrochloride [*18760-80-0*]

dl-Methylephedrine Hydrochloride, when dried, contains not less than 99.0% of $C_{11}H_{17}NO.HCl$.

Description *dl*-Methylephedrine Hydrochloride occurs as colorless crystals or a white, crystalline powder. It is odorless, and has a bitter taste.

It is freely soluble in water, soluble in ethanol (95), slightly soluble in acetic acid (100), and practically insoluble in acetic anhydride and in diethyl ether.

A solution of *dl*-Methylephedrine Hydrochloride (1 in 20) shows no optical rotation.

Identification (1) To 2 mL of a solution of *dl*-Methylephedrine Hydrochloride (1 in 100) add 1 drop of copper (II) sulfate TS and 2 mL of sodium hydroxide TS: a blue-purple color is produced. To the mixture add 1 mL of diethyl ether, and shake: a red-purple color develops in the diethyl ether layer, and a blue-purple color develops in the water layer.

(2) To 1 mL of a solution of *dl*-Methylephedrine Hydrochloride (1 in 20) add sodium hydroxide TS to make alkaline. To the mixture add 2 to 3 drops of potassium permanganate TS, and heat: the odor of benzaldehyde is perceptible, and the gas evolved changes moistened red litmus paper to blue.

(3) Dissolve 0.1 g of *dl*-Methylephedrine Hydrochloride in 1 mL of water, add 10 mL of 2,4,6-trinitrophenol TS, and allow to stand for 2 hours with occasional shaking. Collect the precipitate, recrystallize from dilute ethanol, and dry in a desiccator (in vacuum, silica gel) for 5 hours: the crystals melt between 124°C and 128°C.

(4) A solution of *dl*-Methylephedrine Hydrochloride (1 in 10) responds to the Qualitative Tests for chloride.

Melting point 207 - 211°C

Purity Acid or alkali—Dissolve 2.0 g of *dl*-Methylephedrine Hydrochloride in 40 mL of water, add 2 drops of methyl red TS, and use this solution as the sample solution.

(i) To 20 mL of the sample solution add 0.10 mL of 0.01 mol/L sulfuric acid VS: a red color develops.

(ii) To 20 mL of the sample solution add 0.20 mL of 0.02 mol/L sodium hydroxide VS: a yellow color develops.

Loss on drying Not more than 0.5% (1 g, 105°C, 3 hours).

Residue on ignition Not more than 0.10% (1 g).

Assay Weigh accurately about 0.4 g of *dl*-Methylephedrine Hydrochloride, previously dried, dissolve in 80 mL of a mixture of acetic anhydride and acetic acid (100) (7:3), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 21.572 mg of $C_{11}H_{17}NO.HCl$

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

10% *dl*-Methylephedrine Hydrochloride Powder

dl-Methylephedrine Hydrochloride Powder

dl-塩酸メチルエフェドリン散 10%

10% *dl*-Methylephedrine Hydrochloride Powder contains not less than 9.3% and not more than 10.7% of *dl*-methylephedrine hydrochloride ($C_{11}H_{17}NO.HCl$: 215.72).

Method of preparation

dl-Methylephedrine Hydrochloride	100 g
Starch, Lactose or their mixture	a sufficient quantity
To make	1000 g

Prepare as directed under Powders, with the above ingredients.

Description To 2.5 g of 10% *dl*-Methylephedrine Hydrochloride Powder add 30 mL of ethanol (99.5), and allow to stand for 10 minutes at 40°C with occasional shaking. Cool, and filter: the filtrate shows no optical rotation.

Identification To 10 g of 10% *dl*-Methylephedrine Hydrochloride Powder add 120 mL of ethanol (99.5), and warm at 40°C for 10 minutes with occasional shaking. Cool, filter, evaporate the filtrate on a water bath to dryness, and proceed with the residue as directed in the Identification under *dl*-Methylephedrine Hydrochloride.

Assay Weigh accurately about 0.6 g of 10% *dl*-Methylephedrine Hydrochloride Powder, add 80 mL of water, shake, add water to make exactly 100 mL, and filter. Pipet 5 mL of the filtrate, dilute with water to make exactly 50 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.06 g of *dl*-methylephedrine hydrochloride for assay, previously dried at 105°C for 3 hours, and dissolve in water to make exactly 100 mL. Pipet 5 mL of the solution, add water to make exactly 50 mL, and use as the standard solution. To exactly 2 mL each of the sample solution and the standard solution add 1 mL of sodium carbonate TS and 2 mL of a solution of potassium hexacyanoferrate (III) (3 in 100), allow to stand for 40 minutes, then add exactly 20 mL of hexane for ultraviolet-visible spectrophotometry, and shake. Separate the hexane layer, filter, discard the first 10 mL of each filtrate, and determine the absorbances, A_T and A_S, of the subsequent filtrates at 241 nm, respectively, as directed under the Ultraviolet-visible Spectrophotometry, using a solution, prepared with 2 mL of water in the same manner, as the blank.

Amount (mg) of *dl*-methylephedrine hydrochloride ($C_{11}H_{17}NO.HCl$)
= amount (mg) of *dl*-methylephedrine hydrochloride for assay
$\times \frac{A_T}{A_S}$

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.