make exactly 100 mL. Pipet 5 mL of this solution, add water to make exactly 100 mL, then pipet 1 mL of this solution, add water to make exactly 100 mL, and use this solution as the standard solution. Determine immediately the fluorescence intensities, F_T and F_S, of the sample solution and the standard solution at 338 nm as the excitation wavelength and at 427 nm as the fluorescence wavelength as directed under the Fluorometry.

The dissolution rate of Methylergometrine Maleate Tablets in 30 minutes should be not less than 70%.

Dissolution rate (%) with respect to the labeled amount of methylergometrine maleate ($C_{20}H_{25}N_3O_2.C_4H_4O_4$)

$$= W_S \times \frac{F_T}{F_S} \times \frac{V'}{V} \times \frac{1}{C} \times 0.45$$

W_S: Amount (mg) of methylergometrine maleate for assay.

C: Labeled amount (mg) of methylergometrine maleate ($C_{20}H_{25}N_3O_2.C_4H_4O_4$) in 1 tablet.

Content uniformity Transfer 1 tablet of Methylergometrine Maleate Tablets to a brown glass-stoppered centrifuge tube, add 10 mL of water, shake for 10 minutes vigorously, and disintegrate the tablet. Add 3 g of sodium chloride and 2 mL of ammonia solution (28), add exactly 25 mL of chloroform, and after vigorous shaking for 10 minutes, centrifuge for 5 minutes. Discard the water layer, take the chloroform extracts, add chloroform to make exactly V mL of a solution containing about 5 μg of methylergometrine maleate ($C_{20}H_{25}N_3O_2.C_4H_4O_4$) per mL, and use this solution as the sample solution. Separately, weigh accurately about 1.25 mg of Ergometrine Maleate Reference Standard, previously dried in a desiccator (silica gel) for 4 hours, dissolve in water, and add water to make exactly 100 mL. Pipet 10 mL of this solution into a brown glass-stoppered centrifuge tube, and add 3 g of sodium chloride and 2 mL of ammonia solution (28). Add exactly 25 mL of chloroform, and after vigorous shaking for 10 minutes, centrifuge for 5 minutes. Discard the water layer, take the chloroform extract, and use this solution as the standard solution. Pipet 20 mL each of the sample solution and the standard solution into brown glass-stoppered centrifuge tubes, add immediately exactly 10 mL of dilute 4-dimethylaminobenzaldehyde-ferric chloride TS, respectively, and shake for 5 minutes vigorously. Centrifuge these solutions for 5 minutes, take the water layers, and allow to stand for 1 hour. Perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry, using dilute 4-dimethylaminobenzaldehyde-ferric chloride TS 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.

Amount (mg) of methylergometrine maleate ($C_{20}H_{25}N_3O_2.C_4H_4O_4$)
= amount (mg) of Ergometrine Maleate Reference Standard

$$\times \frac{A_T}{A_S} \times \frac{V}{250} \times 1.0318$$

Assay Weigh accurately and powder not less than 20 Methylergometrine Maleate Tablets. Weigh accurately a portion of the powder, equivalent to about 0.3 mg of methylergometrine maleate ($C_{20}H_{25}N_3O_2.C_4H_4O_4$), transfer to a brown separator, add 15 mL of sodium hydrogen carbonate solution (1 in 20), and extract with four 20-mL portions of chloroform. Filter each portion of the chloroform extracts through a pledget of absorbent cotton, previously moistened with chloroform, into another dried, brown separator, combine all the extracts, and use this extract as the sample solution. Separately, weigh accurately about 10 mg of Ergometrine Maleate Reference Standard, previously dried in a desiccator (silica gel) for 4 hours, dissolve in water, and add water to make exactly 100 mL. Pipet 3 mL of this solution, and transfer to a brown separator, proceed in the same manner as the preparation of the sample solution, and use this extract as the standard solution. To each total volume of the sample solution and the standard solution add exactly 25 mL each of dilute *p*-dimethylaminobenzadehyde-ferric chloride TS, and after vigorous shaking for 5 minutes, allow to stand for 30 minutes. Draw off the water layer, centrifuge, and allow to stand for 1 hour. Perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry, using dilute 4-dimethylaminobenzaldehyde-ferric chloride TS 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.

Amount (mg) of methylergometrine maleate ($C_{20}H_{25}N_3O_2.C_4H_4O_4$)
= amount (mg) of Ergometrine Maleate Reference Standard

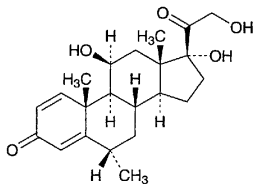
$$\times \frac{A_T}{A_S} \times \frac{3}{100} \times 1.0318$$

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Methylprednisolone

メチルプレドニゾロン



$C_{22}H_{30}O_5$: 374.47

11β,17,21-Trihydroxy-6α-methylpregna-1,4-diene-3,20-dione [*83-43-2*]

Methylprednisolone, when dried, contains not less than 96.0% and not more than 104.0% of $C_{22}H_{30}O_5$.

Description Methylprednisolone occurs as a white, crystalline powder. It is odorless.

It is sparingly soluble in methanol and in 1,4-dioxane, slightly soluble in ethanol (95) and in chloroform, and practically insoluble in water and in diethyl ether.

Melting point: 232 - 240°C (with decomposition).

Identification (1) Add 2 mL of sulfuric acid to 2 mg of Methylprednisolone: a deep red color develops with no fluorescence. Then add 10 mL of water to this solution: the

color fades, and a gray, flocculent precipitate is produced.

(2) Dissolve 0.01 g of Methylprednisolone in 1 mL of methanol, add 1 mL of Fehling's TS, and heat: a red precipitate is produced.

(3) Determine the absorption spectrum of a solution of Methylprednisolone in methanol (1 in 100,000) 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.

Optical rotation $[\alpha]_D^{20}$: +79 – +86° (after drying, 0.1 g, 1,4-dioxane, 10 mL, 100 mm).

Purity Other steroids—Dissolve 0.050 g of Methylprednisolone in 5 mL of a mixture of chloroform and methanol (9:1), and use this solution as the sample solution. Pipet 1 mL of this solution, add a mixture of chloroform and methanol (9:1) to make exactly 200 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 dichloromethane, diethyl ether, methanol and water (385:75:40:6) to a distance of about 12 cm, and air-dry the plate. Then heat at 105°C for 10 minutes, cool, and spray evenly alkaline blue tetrazolium 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 1.0% (0.5 g, 105°C, 3 hours).

Residue on ignition Not more than 0.2% (0.2 g).

Assay Weigh accurately about 0.01 g of Methylprednisolone, previously dried, and dissolve in methanol to make exactly 100 mL. To exactly 5 mL of this solution add methanol to make exactly 50 mL, and determine the absorbance A at the wavelength of maximum absorption at about 243 nm as directed under the Ultraviolet-visible Spectrophotometry.

$$\text{Amount (mg) of } C_{22}H_{30}O_5 = \frac{A}{400} \times 1000$$

Containers and storage Containers—Tight containers.

Methylrosanilinium Chloride

Crystal Violet

塩化メチルロザニリン

$C_{25}H_{30}ClN_3$: 407.98

Methylrosanilinium Chloride is hexamethylpararosaniline chloride, and is usually admixed with pentamethylpararosaniline chloride and tetramethylpararosaniline chloride.

It contains not less than 96.0% of methylrosanilinium chloride [as hexamethylpararosaniline chloride ($C_{25}H_{30}ClN_3$)], calculated on the dried basis.

Description Methylrosanilinium Chloride occurs as green fragments having a metallic luster or a dark green powder. It is odorless or has a slight odor.

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

Identification (1) To 1 mL of sulfuric acid add 1 mg of Methylrosanilinium Chloride: it dissolves, and shows an orange to red-brown color. To this solution add water dropwise: the color of the solution changes from brown through green to blue.

(2) Dissolve 0.02 g of Methylrosanilinium Chloride in 10 mL of water, add 5 drops of hydrochloric acid, and use this solution as the sample solution. To 5 mL of the sample solution add tannic acid TS dropwise: an intense blue precipitate is formed.

(3) To 5 mL of the sample solution obtained in (2) add 0.5 g of zinc powder, and shake: the solution is decolorized. Place 1 drop of this solution on filter paper, and apply 1 drop of ammonia TS adjacent to it: a blue color is produced at the zone of contact of the both solutions.

Purity (1) Ethanol-insoluble substances—Weigh accurately about 1 g of Methylrosanilinium Chloride, previously dried at 105°C for 4 hours, heat with 50 mL of ethanol (95) under a reflux condenser for 15 minutes in a water bath, and filter the mixture through a tared glass filter (G4). Wash the residue on the filter with warm ethanol (95) until the last washing does not show a purple color, and dry at 105°C for 2 hours: the mass of the residue is not more than 1.0%.

(2) Heavy metals—Proceed with 1.0 g of Methylrosanilinium Chloride according to Method 2, and perform the test. Prepare the control solution with 3.0 mL of Standard Lead Solution (not more than 30 ppm).

(3) Zinc—To 0.10 g of Methylrosanilinium Chloride add 0.1 mL of sulfuric acid, and incinerate by ignition. After cooling, boil with 5 mL of dilute hydrochloric acid, 0.5 mL of dilute nitric acid and 4 mL of water, add 5 mL of ammonia TS, boil again, and filter. To the filtrate add 2 to 3 drops of sodium sulfide TS: no turbidity is produced.

(4) Arsenic—Prepare the test solution with 0.40 g of Methylrosanilinium Chloride, according to Method 3, and perform the test using Apparatus B (not more than 5 ppm).

Loss on drying Not more than 7.5% (1 g, 105°C, 4 hours).

Residue on ignition Not more than 1.5% (0.5 g).

Assay Transfer about 0.4 g of Methylrosanilinium Chloride, accurately weighed, to a wide-mouthed, conical flask, add 25 mL of water and 10 mL of hydrochloric acid, dissolve, and add exactly 50 mL of 0.1 mol/L titanium (III) chloride VS while passing a stream of carbon dioxide through the flask. Heat to boil, and boil gently for 15 minutes, swirling the liquid frequently. Cool while passing a stream of carbon dioxide through the flask, titrate the excess titanium (III) chloride with 0.05 mol/L ammonium iron (III) sulfate VS until a faint, red color is produced (indicator: 5 mL of ammonium thiocyanate TS). Perform a blank determination.

Each mL of 0.1 mol/L titanium (III) chloride VS
= 20.399 mg of $C_{25}H_{30}ClN_3$

Containers and storage Containers—Tight containers.