weigh it as the mass of phenytoin ($C_{15}H_{12}N_2O_2$: 252.27).

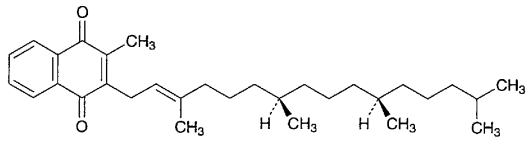
Amount (mg) of phenytoin sodium ($C_{15}H_{11}N_2NaO_2$) = amount (mg) of phenytoin ($C_{15}H_{12}N_2O_2$) × 1.0871

Containers and storage Containers—Hermetic containers.

Phytonadione

Phytomenadione
Vitamin K_1

フィトナジオン



$C_{31}H_{46}O_2$: 450.70
2-Methyl-3-[(2*E*,7*R*,11*R*)-3,7,11,15-tetramethylhexadec-2-en-1-yl]-1,4-naphthoquinone [*84-80-0*]

Phytonadione contains not less than 97.0% and not more than 102.0% of $C_{31}H_{46}O_2$.

Description Phytonadione is a clear yellow to orange-yellow, viscous liquid. It is odorless.

It is miscible with diethyl ether and with isooctane.

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

It decomposes gradually and darkens by light.

Specific gravity d_{20}^{20}: about 0.967

Identification **(1)** Dissolve 0.05 g of Phytonadione in 10 mL of ethanol (95), and add 1 mL of a solution of potassium hydroxide in ethanol (95) (1 in 10): a blue color develops and changes to purple, then to brown upon standing.

(2) Dissolve 0.05 g of Phytonadione in 10 mL of a mixture of methanol and diethyl ether (1:1), add a freshly prepared solution of 0.75 g of sodium hydrosulfite in 2 mL of warm water, and shake vigorously: a yellow color disappears immediately.

(3) Determine the absorption spectrum of a solution of Phytonadione in isooctane (1 in 100,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum 1: both spectra exhibit similar intensities of absorption at the same wavelengths. Separately, determine the absorption spectrum of a solution of Phytonadione in isooctane (1 in 10,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum 2: both spectra exhibit similar intensities of absorption at the same wavelengths.

Refractive index n_D^{20}: 1.525 - 1.529

Purity **(1)** Clarity and color of solution—Dissolve 1.0 g of Phytonadione in 10 mL of isooctane: the solution is clear, and shows a yellow color.

(2) Ratio of absorbances—Determine the absorbances, A_1, A_2 and A_3, of a solution of Phytonadione in isooctane (1 in 100,000) at 248.5 nm, 253.5 nm and 269.5 nm, respectively: the ratio A_2/A_1 is between 0.69 and 0.73, and the ratio A_2/A_3 is between 0.74 and 0.78. Determine the absorbances, A_4 and A_5, of a solution of Phytonadione in isooctane (1 in 10,000) at 284.5 nm and 326.0 nm, respectively: the ratio A_4/A_5 is between 0.28 and 0.34.

(3) Heavy metals—Carbonize 1.0 g of Phytonadione by gentle heating. Cool, add 10 mL of a solution of magnesium nitrate hexahydrate in ethanol (95) (1 in 10), and ignite the ethanol to burn. Cool, add 1 mL of sulfuric acid, proceed according to Method 4, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(4) Menadione—Dissolve 0.020 g of Phytonadione in 0.5 mL of a mixture of water and ethanol (95) (1:1), add 1 drop of a solution of 3-methyl-1-phenyl-5-pyrazolone in ethanol (95) (1 in 20) and 1 drop of ammonia solution (28), and allow to stand for 2 hours: no blue-purple color develops.

Assay Perform the test quickly under the protection from sunlight. Weigh accurately about 0.1 g of Phytonadione, dissolve in isooctane to make exactly 100 mL. Measure exactly 10 mL of this solution, and add isooctane to make exactly 100 mL. Pipet 10 mL of this solution, and add isooctane to make exactly 100 mL. Determine the absorbance A of this solution at the wavelength of maximum absorption at about 248.5 nm, as directed under the Ultraviolet-visible Spectrophotometry, adjusting the slit of the spectrophotometer to a band width of 0.5 nm.

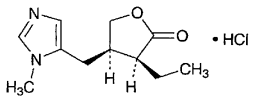
$$\text{Amount (mg) of } C_{31}H_{46}O_2 = \frac{A}{422} \times 100{,}000$$

Containers and storage Containers—Tight containers.

Storage—Light-resistant.

Pilocarpine Hydrochloride

塩酸ピロカルピン



$C_{11}H_{16}N_2O_2$.HCl: 244.72
(3*S*,4*R*)-3-Ethyldihydro-4-(1-methyl-1*H*-imidazol-5-ylmethyl)furan-2(3*H*)-one monohydrochloride [*54-71-7*]

Pilocarpine Hydrochloride, when dried, contains not less than 99.0% of $C_{11}H_{16}N_2O_2$.HCl.

Description Pilocarpine Hydrochloride occurs as colorless crystals or white powder. It is odorless, and has a slightly bitter taste.

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

The pH of a solution of Pilocarpine Hydrochloride (1 in 10) is between 3.5 and 4.5.

It is hygroscopic.

It is affected by light.

Identification **(1)** Dissolve 0.1 g of Pilocarpine

Hydrochloride in 5 mL of water, add 1 drop of dilute nitric acid, 1 mL of hydrogen peroxide TS, 1 mL of chloroform and 1 drop of a potassium dichromate solution (1 in 300), and shake the mixture vigorously: a violet color develops in the chloroform layer while no color or a light yellow color is produced in the aqueous layer.

(2) To 1 mL of a solution of Pilocarpine Hydrochloride (1 in 20) add 1 mL of dilute nitric acid and 2 to 3 drops of silver nitrate TS: a white precipitate or opalescence is produced.

Melting point 200 - 203°C

Purity **(1)** Sulfate—Dissolve 0.5 g of Pilocarpine Hydrochloride in 20 mL of water, and use this solution as the sample solution. To 5.0 mL of the sample solution add 1 mL of dilute hydrochloric acid and 0.5 mL of barium chloride TS: no turbidity is produced.

(2) Nitrate—To 2.0 mL of the sample solution obtained in (1) add 2 mL of iron (II) sulfate TS, and superimpose the mixture upon 4 mL of sulfuric acid: no dark brown color develops at the zone of contact.

(3) Related substances—Dissolve 0.3 g of Pilocarpine Hydrochloride in 10 mL of methanol, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add methanol to make exactly 100 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, methanol and ammonia TS (85:14:2) to a distance of about 13 cm, and dry the plate at 105°C for 10 minutes. Cool, and spray evenly bismuth potassium iodide 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.

(4) Readily carbonizable substances—Take 0.25 g of Pilocarpine Hydrochloride, and perform the test: the solution has no more color than Matching Fluid B.

Loss on drying Not more than 3.0% (1 g, 105°C, 2 hours).

Residue on ignition Not more than 0.5% (0.1 g).

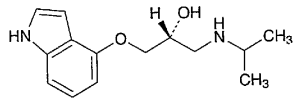
Assay Weigh accurately about 0.5 g of Pilocarpine Hydrochloride, previously dried, dissolve in 50 mL of a mixture of acetic anhydride and acetic acid (100) (7:3), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 24.472 mg of $C_{11}H_{16}N_2O_2$.HCl

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

Pindolol

ピンドロール



and enantiomer

$C_{14}H_{20}N_2O_2$: 248.32
(*RS*)-1-(1*H*-Indol-4-yloxy)-3-isopropylaminopropan-2-ol
[*13523-86-9*]

Pindolol, when dried, contains not less than 98.5% of $C_{14}H_{20}N_2O_2$.

Description Pindolol occurs as a white, crystalline powder. It has a slight, characteristic odor.

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

It dissolves in dilute sulfuric acid and in acetic acid (100).

Identification **(1)** To 1 mL of a solution of Pindolol in methanol (1 in 10,000) add 1 mL of a solution of 1-(4-pyridyl)-pyridinium chloride hydrochloride (1 in 1000) and 1 mL of sodium hydroxide TS, then add 1 mL of hydrochloric acid: a blue to blue-purple color, changing to red-purple, is produced.

(2) Dissolve 0.05 g of Pindolol in 1 mL of dilute sulfuric acid, and add 1 mL of Reinecke salt TS: a light red precipitate is produced.

(3) Determine the absorption spectrum of a solution of Pindolol in methanol (1 in 50,000) 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.

(4) Determine the infrared absorption spectrum of Pindolol, previously dried, 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.

Absorbance $E^{1\%}_{1\,cm}$ (264 nm): 333 - 350 (0.01 g, methanol, 500 mL).

Melting point 169 - 173°C

Purity **(1)** Clarity and color of solution—Dissolve 0.5 g of Pindolol in 10 mL of acetic acid (100), and observe immediately: the solution is clear, and has no more color than the following control solution.

Control solution: Measure accurately 4 mL of Matching Fluid A, add exactly 6 mL of water, and mix.

(2) Heavy metals—Proceed with 1.0 g of Pindolol according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(3) Arsenic—Prepare the test solution with 1.0 g of Pindolol according to Method 3, and perform the test using Apparatus B (not more than 2 ppm).

(4) Related substances—Dissolve 0.10 g of Pindolol in 10 mL of methanol, and use this solution as the sample solu-