sorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 231 nm and 235 nm.

Dissolution test Perform the test with 1 tablet of Chlorpropamide Tablets at 50 revolutions per minute according to Method 2 under the Dissolution Test, using 900 mL of diluted phosphate buffer solution, pH 6.8, (1 in 2) as the test solution. Take 20 mL or more of the dissolved solution 45 minutes after starting 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 of the filtrate, add diluted phosphate buffer solution, pH 6.8, (1 in 2) to make exactly V' mL so that each mL contains about 10 μg of chlorpropamide ($C_{10}H_{13}ClN_2O_3S$) according to the labeled amount, and use this solution as the sample solution. Separately, weigh accurately about 0.05 g of chlorpropamide for assay, previously dried at 105°C for 3 hours, dissolve in 10 mL of methanol, and add water to make exactly 50 mL. Pipet 1 mL of this solution, add diluted phosphate buffer solution, pH 6.8, (1 in 2) 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 232 nm as directed under the Ultraviolet-visible Spectrophotometry.

The dissolution rate of Chlorpropamide Tablets in 45 minutes should be not less than 70%.

Dissolution rate (%) with respect to the labeled amount of chlorpropamide ($C_{10}H_{13}ClN_2O_3S$)

$$= W_S \times \frac{A_T}{A_S} \times \frac{V'}{V} \times \frac{1}{C} \times 18$$

W_S: Amount (mg) of chlorpropamide for assay.
C: Labeled amount (mg) of chlorpropamide ($C_{10}H_{13}ClN_2O_3S$) in 1 tablet.

Assay Weigh accurately and powder not less than 20 Chlorpropamide Tablets. Weigh accurately a quantity of the powder, equivalent to about 0.05 g of chlorpropamide ($C_{10}H_{13}ClN_2O_3S$), add 75 mL of the mobile phase, shake for 10 minutes, and add the mobile phase to make exactly 100 mL. Centrifuge this solution, pipet 10 mL of the supernatant liquid, add the mobile phase to make exactly 100 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.05 g of chlorpropamide for assay, previously dried at 105°C for 3 hours, dissolve in the mobile phase to make exactly 100 mL. Pipet 10 mL of this solution, add the mobile phase to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions. Determine the peak areas, A_T and A_S, of chlorpropamide of the sample solution and the standard solution.

Amount (mg) of $C_{10}H_{13}ClN_2O_3S$
= amount (mg) of chlorpropamide for assay
$\times \frac{A_T}{A_S}$

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 240 nm).

Column: A stainless steel column 4.6 mm in inside diameter and 25 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (10 μm in particle diameter).

Column temperature: A constant temperature of about 25°C.

Mobile phase: A mixture of diluted acetic acid (100) (1 in 100) and acetonitrile (1:1).

Flow rate: Adjust the flow rate so that the retention time of chlorpropamide is about 5 minutes.

System suitability—

System performance: When the procedure is run with 20 μL of the standard solution under the above operating conditions, the number of theoretical plates and the symmetry constant of the peak of chlorpropamide are not less than 1500 and not more than 1.5, respectively.

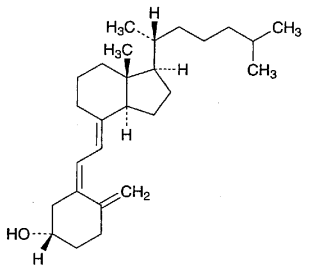
System repeatability: When the test is repeated 6 times with 20 μL of the standard solution under the above operating conditions, the relative standard deviation of the peak areas of chlorpropamide is not more than 1.5%.

Containers and storage Containers—Well-closed containers.

Cholecalciferol

Vitamin D_3

コレカルシフェロール



$C_{27}H_{44}O$: 384.64
(3*S*,5*Z*,7*E*)-9,10-Secocholesta-5,7,10(19)-trien-3-ol
[*67-97-0*]

Cholecalciferol contains not less than 97.0% and not more than 103.0% of $C_{27}H_{44}O$.

Description Cholecalciferol occurs as white crystals. It is odorless.

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

It is affected by air and by light.

Melting point: 84 - 88°C Transfer Cholecalciferol to a capillary tube, and dry for 3 hours in a desiccator (in vacuum at a pressure not exceeding 2.67 kPa). Immediately fireseal the capillary tube, put it in a bath fluid, previously heated to a temperature about 10°C below the expected melting point, and heat at a rate of rise of about 3°C per minute, and read the melting point.

Identification (1) Dissolve 0.5 mg of Cholecalciferol in 5 mL of chloroform, add 0.3 mL of acetic anhydride and 0.1 mL of sulfuric acid, and shake: a red color is produced, and

rapidly changes through purple and blue to green.

(2) Determine the infrared absorption spectrum of Cholecalciferol as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of Cholecalciferol Reference Standard: both spectra exhibit similar intensities of absorption at the same wave numbers.

Absorbance $E^{1\%}_{1\,cm}$ (265 nm): 450 - 490 (0.01 g, ethanol (95), 1000 mL).

Optical rotation $[\alpha]_D^{20}$ +103 - +112° (0.05 g, ethanol (95), 10 mL, 100 mm). Prepare the solution without delay, using Cholecalciferol from a container opened not longer than 30 minutes, previously, and determine the rotation within 30 minutes after the solution has been prepared.

Purity 7-Dehydrocholesterol—Dissolve 0.010 g of Cholecalciferol in 2.0 mL of diluted ethanol (95) (9 in 10), add a solution prepared by dissolving 0.020 g of digitonin in 2.0 mL of diluted ethanol (95) (9 in 10), and allow the mixture to stand for 18 hours: no precipitate is formed.

Assay Dissolve separately about 0.03 g each of Cholecalciferol and Cholecalciferol Reference Standard, accurately weighed, in isooctane to make exactly 50 mL. Pipet 10 mL each of these solutions, add 3 mL each of the internal standard solution, then add the mobile phase to make 50 mL, and use these solutions as the sample solution and the standard solution. Perform the test with 10 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the ratios, Q_T and Q_S, of the peak area of cholecalciferol to that of the internal standard. Proceed with the operation avoiding contact with air or other oxidizing agents and using light-resistant containers.

Amount (mg) of $C_{27}H_{44}O$
= amount (mg) of Cholecalciferol Reference Standard
$\times \frac{Q_T}{Q_S}$

Internal standard solution—A solution of dimethyl phthalate in isooctane (1 in 100).

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 254 nm).

Column: A stainless steel column about 4 mm in inside diameter and 10 to 30 cm in length, packed with silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Column temperature: Ordinary temperature.

Mobile phase: A mixture of hexane and *n*-amylalcohol (997:3).

Flow rate: Adjust the flow rate so that the retention time of cholecalciferol is about 25 minutes.

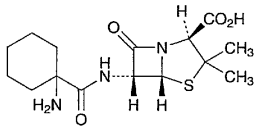
Selection of column: Dissolve 0.015 g of Cholecalciferol Reference Standard in 25 mL of isooctane. Transfer this solution to a flask, heat under a reflux condenser in an oil bath for 2 hours, and cool to room temperature rapidly. Transfer this solution to a quartz test tube, and irradiate under a short-wave lamp (main wavelength: 254 nm) and a long-wave lamp (main wavelength: 365 nm) for 3 hours. To this solution add the mobile phase to make 50 mL. Proceed with 10 μL of this solution under the above operating conditions. Use a column with the ratios of the retention time of previtamin D_3, trans-vitamin D_3 and tachysterol$_3$ to that of cholecalciferol being about 0.5, about 0.6 and about 1.1, respectively, and with resolution between previtamin D_3 and trans-vitamin D_3, and that between cholecalciferol and tachysterol$_3$ being not less than 1.0.

Containers and storage Containers—Hermetic containers.

Storage—Light-resistant, under nitrogen atmosphere, and in a cold place.

Ciclacillin

シクラシリン



$C_{15}H_{23}N_3O_4S$: 341.43
(2*S*,5*R*,6*R*)-6-[(1-Aminocyclohexanecarbonyl)amino]-3,3-dimethyl-7-oxo-4-thia-1-azabicyclo[3.2.0]heptane-2-carboxylic acid [*3485-14-1*]

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

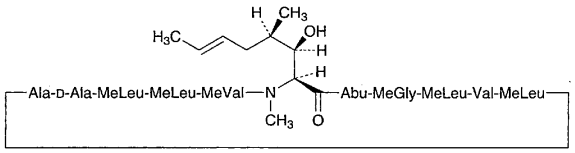
Description Ciclacillin occurs as a white to light yellowish white, crystalline powder.

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

Ciclosporin

Ciclosporin A

シクロスポリン



Abu = (2*S*)-2- Abu=(2*S*)-2-Aminobutyric acid
MeGly = *N*- MeGly=*N*-Methylglycine
MeLeu = *N*- MeLeu=*N*-Methylleucine
MeVal = *N*- MeVal=*N*-Methylvaline

$C_{62}H_{111}N_{11}O_{12}$: 1202.61
cyclo{-[(2*S*,3*R*,4*R*,6*E*)-3-Hydroxy-4-methyl-2-methylaminooct-6-enoyl]-L-2-aminobutanoyl-*N*-methylglycyl-*N*-methyl-L-leucyl-L-valyl-*N*-methyl-L-leucyl-L-alanyl-D-alanyl-*N*-methyl-L-leucyl-*N*-methyl-L-leucyl-*N*-methyl-L-valyl-} [*59865-13-3*]

Ciclosporin contains not less than 98.5% and not more than 101.5% of $C_{62}H_{111}N_{11}O_{12}$, calculated on