form the test with 2 μL each of these solutions as directed under the Gas Chromatography according to the following conditions, and calculate the ratios, Q_T and Q_S, of the peak area of *dl*-camphor to that of the internal standard.

$$\text{Amount (mg) of } C_{10}H_{16}O = \text{amount (mg) of } dl\text{-Camphor Reference Standard} \times \frac{Q_T}{Q_S}$$

Internal standard solution—A solution of methyl salicilate in ethanol (99.5) (1 in 25).

Operating conditions—

Detector: A hydrogen flame-ionization detector.

Column: A glass column 3 mm in inside diameter and 3 m in length, which is packed with 10% of polyethylene glycol 20 M for gas chromatography supported on 180- to 250-μm mesh silanized siliceous earth for gas chromatography.

Column temperature: A constant temperature of about 160°C.

Carrier gas: Nitrogen

Flow rate: Adjust the flow rate so that the retention time of *dl*-camphor is about 6 minutes.

System suitability—

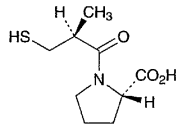
System performance: When the procedure is run with 2 μL of the standard solution under the above operating conditions, *dl*-camphor and the internal standard are eluted in this order with the resolution between these peaks being not less than 7.

System repeatability: When the test is repeated 6 times with 2 μL of the standard solution under the above operating conditions, the relative standard deviation of the ratios of the peak area of *dl*-camphor to that of the internal standard is not more than 1.0%.

Containers and storage Containers—Tight containers.

Captopril

カプトプリル



$C_9H_{15}NO_3S$: 217.29

(2*S*)-1-[(2*S*)-2-Methyl-3-sulfanylpropanonyl]pyrrolidine-2-carboxylic acid [*62571-86-2*]

Captopril contains not less than 98.0% of $C_9H_{15}NO_3S$, calculated on the dried basis.

Description Captopril occurs as white crystals or crystalline powder.

It is very soluble in methanol, freely soluble in ethanol (99.5), and soluble in water.

Identification Determine the infrared absorption spectrum of Captopril 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.

Optical rotation $[\alpha]_D^{25}$: −125 - −134° (after drying, 0.1 g, ethanol (99.5), 10 mL, 100 mm).

Melting point 105 - 110°C

Purity **(1)** Heavy metals—Proceed with 1.0 g of Captopril 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).

(2) Arsenic—Prepare the test solution with 1.0 g of Captopril according to Method 1, and perform the test using Apparatus B (not more than 2 ppm).

(3) Related substances—Dissolve 0.20 g of Captopril in methanol to make exactly 10 mL, and use this solution as the sample solution. Separately, dissolve 0.015 g of 1,1′-[3,3′-dithiobis(2-methyl-1-oxopropyl)]-L-diproline in methanol to make exactly 250 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 with a mixture of toluene and acetic acid (100) (13:7) to a distance of about 15 cm, and air-dry the plate. Place the plate in a chamber filled with iodine vapor, and allow to stand for 30 minutes: the number of the spots other than the spot corresponding to that from the standard solution and the principal spot from the sample solution is not more than two, and they are not more intense than the spot from the standard solution.

(4) 1,1′-[3,3′-Dithiobis(2-methyl-1-oxopropyl)]-L-diproline—Dissolve 0.10 g of Captopril in methanol to make exactly 20 mL, and use this solution as the sample solution. Separately, dissolve 0.025 g of 1,1′-[3,3′-dithiobis(2-methyl-1-oxopropyl)]-L-diproline in methanol to make exactly 250 mL, and use this solution as the standard solution. Perform the test with 20 μL each of these solutions as directed under the Liquid Chromatography according to the following conditions, and calculate the peak area, A_T and A_S, of 1,1′-[3,3′-dithiobis(2-methyl-1-oxopropyl)]-L-diproline of these solutions: A_T is not larger than A_S.

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 220 nm).

Column: A stainless steel column 3.9 mm in inside diameter and 30 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 water, methanol and phosphoric acid (1000:1000:1).

Flow rate: Adjust the flow rate so that the retention time of 1,1′-[3,3′-dithiobis(2-methyl-1-oxopropyl)]-L-diproline is about 10 minutes.

System suitability—

System performance: Dissolve 0.025 g each of Captopril and 1,1′-[3,3′-dithiobis(2-methyl-1-oxopropyl)]-L-diproline in 200 mL of methanol. When the procedure is run with 20 μL of this solution under the above operating conditions, captopril and 1,1′-[3,3′-dithiobis(2-methyl-1-oxopropyl)]-L-diproline are eluted in this order with the resolution between these peaks being not less than 3.

System repeatability: When the test is repeated 5 times with 20 μL of the standard solution under the above operating conditions, the relative standard deviation of the peak areas of 1,1′-[3,3′-dithiobis(2-methyl-1-oxopropyl)]-L-diproline is not more than 2.0%.

Loss on drying Not more than 1.0% (1 g, in vacuum, 80°C, 3 hours).

Residue on ignition Not more than 0.20% (1 g).

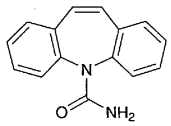
Assay Weigh accurately about 0.3 g of Captopril, dissolve in 100 mL of water, add 20 mL of dilute sulfuric acid and 1 g of potassium iodide, and shake. Titrate with 1/60 mol/L potassium iodate VS (indicator: 2 mL of starch TS). Perform a blank determination in the same manner, and make any necessary correction.

Each mL of 1/60 mol/L potassium iodate VS
= 21.729 mg of $C_9H_{15}NO_3S$

Containers and storage Containers—Tight containers.

Carbamazepine

カルバマゼピン



$C_{15}H_{12}N_2O$: 236.27
5*H*-Dibenz[*b*,*f*]azepine-5-carboxamide [*298-46-4*]

Carbamazepine, when dried, contains not less than 97.0% and not more than 103.0% of $C_{15}H_{12}N_2O$.

Description Carbamazepine occurs as a white to slightly yellowish white powder. It is odorless and tasteless at first, and leaves a slightly bitter aftertaste.

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

Identification (1) To 0.1 g of Carbamazepine add 2 mL of nitric acid, and heat on a water bath for 3 minutes: an orange-red color is produced.

(2) To 0.1 g of Carbamazepine add 2 mL of sulfuric acid, and heat on a water bath for 3 minutes: a yellow color is produced with a green fluorescence.

(3) Examine Carbamazepine under ultraviolet light: the solution shows an intense blue fluorescence.

(4) Determine the absorption spectrum of the solution obtained in the Assay 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.

Melting point 189 - 193°C

Purity (1) Clarity and color of solution—Dissolve 1.0 g of Carbamazepine in 10 mL of chloroform: the solution is clear and colorless to pale yellow.

(2) Acid—To 2.0 g of Carbamazepine add exactly 40 mL of water, stir well for 15 minutes, and filter through a glass filter (G3). To 10 mL of this filtrate add 1 drop of phenolphthalein TS and 0.50 mL of 0.01 mol/L sodium hydroxide VS: a red color is produced.

(3) Alkali—To 10 mL of the filtrate obtained in (2) add 1 drop of methyl red TS and 0.50 mL of 0.01 mol/L hydrochloric acid VS: a red color is produced.

(4) Chloride—Dissolve 0.25 g of Carbamazepine in 30 mL of acetone, add 6 mL of dilute nitric acid and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution as follows: to 0.20 mL of 0.01 mol/L hydrochloric acid VS add 30 mL of acetone, 6 mL of dilute nitric acid and water to make 50 mL (not more than 0.028%).

(5) Heavy metals—Proceed with 2.0 g of Carbamazepine according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

(6) Related substances—Dissolve 0.25 g of Carbamazepine in 10 mL of chloroform, and use this solution as the sample solution. Separately, dissolve 5.0 mg of iminodibenzyl in chloroform 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 toluene and methanol (19:1) to a distance of about 10 cm, and air-dry the plate. Spray evenly potassium dichromate-sulfuric acid TS on the plate: the spots other than the principal spot obtained from the sample solution is not more intense than the spot from the standard solution.

Loss on drying Not more than 0.5% (1 g, 105°C, 2 hours).

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

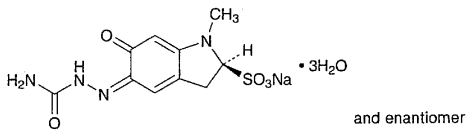
Assay Dissolve about 0.05 g of Carbamazepine, previously dried and accurately weighed, in ethanol (95) to make exactly 250 mL. Dilute 5 mL of this solution with ethanol (95) to exactly 100 mL. Perform the test as directed under the Ultraviolet-visible Spectrophotometry, and determine the absorbance A of this solution at the wavelength of maximum absorption at about 285 nm.

$$\text{Amount (mg) of } C_{15}H_{12}N_2O = \frac{A}{490} \times 50{,}000$$

Containers and storage Containers—Tight containers.

Carbazochrome Sodium Sulfonate

カルバゾクロムスルホン酸ナトリウム



and enantiomer

$C_{10}H_{11}N_4NaO_5S.3H_2O$: 376.32
Monosodium (*RS*)-2,3,5,6-tetrahydro-1-methyl-6-oxo-5-semicarbazonoindole-2-sulfonate trihydrate
[*51460-26-5*, anhydride]