

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-dipropylene 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).

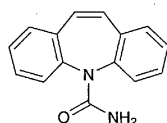
Assay Weigh accurately about 0.3 g of Carbamazepine, dissolve in 100 mL of water, add 20 mL of dilute sulfuric acid and 1 g of potassium iodide, and shake. Titrate with 1/60 mol/L potassium iodate VS (indicator: 2 mL of starch TS). Perform a blank determination in the same manner, and make any necessary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

Each mL of 1/60 mol/L potassium iodate VS
= 21.729 mg of $\text{C}_9\text{H}_{15}\text{NO}_3\text{S}$

Containers and storage Containers—Tight containers.

Carbamazepine

カルバマゼピン



$\text{C}_{15}\text{H}_{12}\text{N}_2\text{O}$: 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 $\text{C}_{15}\text{H}_{12}\text{N}_2\text{O}$.

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).

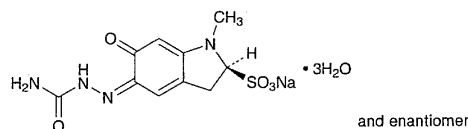
Assay Dissolve about 0.05 g of Carbamazepine, previously dried and accurately weighed, in ethanol (95) to make exactly 250 mL. Dilute 5 mL of this solution with ethanol (95) to exactly 100 mL. Perform the test as directed under the Ultraviolet-visible Spectrophotometry, and determine the absorbance *A*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

$$\text{Amount (mg) of } \text{C}_{15}\text{H}_{12}\text{N}_2\text{O} = \frac{A}{490} \times 50,000$$

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



$\text{C}_{10}\text{H}_{11}\text{N}_4\text{NaO}_5\text{S} \cdot 3\text{H}_2\text{O}$: 376.32
Monosodium (*RS*)-2,3,5,6-tetrahydro-1-methyl-6-oxo-5-semicarbazonoindole-2-sulfonate trihydrate
[51460-26-5, anhydride]