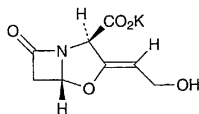


Potassium Clavulanate

クラブラン酸カリウム



$C_8H_8KNO_5$: 237.25

Monopotassium (2*R*,5*R*)-3-[(1*Z*)-2-hydroxyethylidene]-7-oxo-4-oxa-1-azabicyclo[3.2.0]heptane-2-carboxylate [61177-45-5]

Potassium Clavulanate contains not less than 755 μ g (potency) per mg, calculated on the anhydrous basis. The potency of Potassium Clavulanate is expressed as mass (potency) of clavularic acid ($C_8H_9NO_5$; 199.16).

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

It is very soluble in water, soluble in methanol, and slightly soluble in ethanol (95).

It is hygroscopic.

Identification (1) To 1 mL of a solution of Potassium Clavulanate (1 in 50,000) add 5 mL of imidazole TS, warm in a water bath at 30°C for 12 minutes. After cooling, determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 311 nm and 315 nm.

(2) Determine the infrared absorption spectrum of Potassium Clavulanate 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.

(3) Potassium Clavulanate responds to the Qualitative Test (1) for potassium salt.

Optical rotation $[\alpha]_D^{20}$: +55 – +60° (0.5 g calculated on the anhydrous basis, water, 50 mL, 100 mm).

pH Dissolve 0.1 g of Potassium Clavulanate in 10 mL of water: the pH of the solution is between 6.0 and 8.0.

Purity (1) Heavy metals—Proceed with 2.0 g of Potassium Clavulanate according to Method 2, and perform the test. Prepare the control solution with 4.0 mL of Standard Lead Solution (not more than 20 ppm).

(2) Arsenic—Prepare the test solution with 2.0 g of Potassium Clavulanate according to Method 3, and perform the test using Apparatus B (not more than 2 ppm).

Water Not more than 1.5% (5 g, volumetric titration, direct titration).

Assay Weigh accurately an amount of Potassium Clavulanate and Lithium Clavulanate Reference Standard, equivalent to about 12.5 mg (potency), dissolve each in 30 mL of water, add exactly 5 mL of the internal standard solution and water to make 50 mL, and use these solutions as the sample solution and the standard solution, respectively. Perform the test with 5 μ L 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 clavularic acid to that of the internal standard.

Amount [μ g (potency)] of clavularic acid ($C_8H_9NO_5$)
= amount [mg (potency)] of Lithium Clavulanate

$$\text{Reference Standard} \times \frac{Q_T}{Q_S} \times 1000$$

Internal standard solution—Dissolve 0.3 g of sulfanilamide in 30 mL of methanol, and add water to make 100 mL.

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 230 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 (5 μ m in particle diameter).

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

Mobile phase: Dissolve 1.36 g of sodium acetate trihydrate in 900 mL of water, adjust to pH 4.5 with diluted acetic acid (31) (2 in 5), and add 30 mL of methanol and water to make 1000 mL.

Flow rate: Adjust the flow rate so that the retention time of clavularic acid is about 6 minutes.

System suitability—

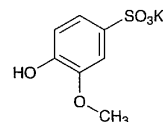
System performance: When the procedure is run with 5 μ L of the standard solution under the above operating conditions, clavularic acid and the internal standard are eluted in this order with the resolution between these peaks being not less than 4.

System repeatability: When the test is repeated 6 times with 5 μ L of the standard solution under the above operating conditions, the relative standard deviation of the ratios of the peak area of clavularic acid to that of the internal standard is not more than 1.0%.

Containers and storage Containers—Tight containers.

Potassium Guaiacolsulfonate

ゲアヤコールスルホン酸カリウム



$C_7H_7KO_5S$: 242.29

Monopotassium 4-hydroxy-3-methoxybenzenesulfonate [1321-14-8]

Potassium Guaiacolsulfonate contains not less than 98.5% of $C_7H_7KO_5S$, calculated on the anhydrous basis.

Description Potassium Guaiacolsulfonate occurs as white crystals or crystalline powder. It is odorless or has a slight, characteristic odor and a slightly bitter taste.

It is freely soluble in water and in formic acid, soluble in