

1 mL of sulfuric acid, add 1 drop of formaldehyde solution, and allow to stand for 5 minutes. To the solution add 5 mL of water: a blue-green suspended substance is produced.

Optical rotation $[\alpha]_D^{20}$: +59.0 – +62.0° (after drying, 1.0 g, ethanol (99.5), 25 mL, 100 mm).

Melting point 200 – 204°C

Purity (1) Odor—To 2.0 g of Ursodeoxycholic Acid add 100 mL of water, and boil for 2 minutes: no odor is perceptible.

(2) Chloride—Dissolve 2.0 g of Ursodeoxycholic Acid in 20 mL of acetic acid (100) with shaking, add water to make 200 mL, shake thoroughly, and allow to stand for 10 minutes. Filter this solution, discard the first 10 mL of the filtrate, and use the subsequent filtrate as the sample solution. To 40 mL of the sample solution add 6 mL of dilute nitric acid and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution as follows: to 0.25 mL of 0.01 mol/L hydrochloric acid VS add 4 mL of acetic acid (100), 6 mL of dilute nitric acid and water to make 50 mL (not more than 0.022%).

(3) Sulfate—To 40 mL of the sample solution obtained in (2) add 1 mL of dilute hydrochloric 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 0.40 mL of 0.005 mol/L sulfuric acid VS add 4 mL of acetic acid (100), 1 mL of dilute hydrochloric acid and water to make 50 mL (not more than 0.048%).

(4) Heavy metals—Proceed with 1.0 g of Ursodeoxycholic Acid 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).

(5) Barium—To the solution obtained in (1) add 2 mL of hydrochloric acid, boil for 2 minutes, cool, filter, and wash with water until the last washing makes 100 mL. To 10 mL of the solution add 1 mL of dilute sulfuric acid: no turbidity is produced.

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

(7) Related substances—Dissolve 0.050 g of Ursodeoxycholic Acid in a mixture of chloroform and ethanol (95) (9:1) to make exactly 25 mL, and use this solution as the sample solution. Separately, dissolve 0.075 g of chenodeoxycholic acid for thin-layer chromatography in a mixture of chloroform and ethanol (95) (9:1) to make exactly 100 mL. To exactly 2 mL of this solution add a mixture of chloroform and ethanol (95) (9:1) to make exactly 50 mL, and use this solution as the standard solution (1). Dissolve 0.025 g of lithocholic acid for thin-layer chromatography in a mixture of chloroform and ethanol (95) (9:1) to make exactly 50 mL. To exactly 1 mL of this solution add a mixture of chloroform and ethanol (95) (9:1) to make exactly 50 mL. To exactly 2 mL of this solution add a mixture of chloroform and ethanol (95) (9:1) to make exactly 10 mL, and use this solution as the standard solution (2). Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μ L each of the sample solution and standard solutions (1) and (2) on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of chloroform, acetone and acetic acid (100) (7:2:1) to a distance of about 10 cm, and air-dry the plate. Dry the plate at 120°C for 30 minutes, spray evenly a solution of phosphomolybdc

acid *n*-hydrate in ethanol (95) (1 in 5) immediately, and heat at 120°C for 2 to 3 minutes: the spot from the sample solution, corresponding to that from the standard solution (1), is not more intense than the spot from the standard solution (1), and the spot other than the principal spot and the above spots from the sample solution are not more intense than the spot from the standard solution (2).

Loss on drying Not more than 1.0% (1 g, 105°C, 2 hours).

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

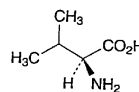
Assay Weigh accurately about 0.5 g of Ursodeoxycholic Acid, previously dried, and dissolve in 40 mL of neutralized ethanol and 20 mL of water. Add 2 drops of phenolphthalein TS, titrate with 0.1 mol/L sodium hydroxide VS, and titrate again after adding 100 mL of freshly boiled and cooled water near the end point.

Each mL of 0.1 mol/L sodium hydroxide VS
= 39.258 mg of C₂₄H₄₀O₄

Containers and storage Containers—Well-closed containers.

L-Valine

L-バリン



C₅H₁₁NO₂: 117.15

(2S)-2-Amino-3-methylbutanoic acid [72-18-4]

L-Valine, when dried, contains not less than 98.5% of C₅H₁₁NO₂.

Description L-Valine occurs as white crystals or crystalline powder. It is odorless or has a faint characteristic odor, and has a slightly sweet taste, which becomes bitter.

It is freely soluble in formic acid, soluble in water, and practically insoluble in ethanol (95).

It dissolves in dilute hydrochloric acid.

Identification Determine the infrared absorption spectrum of L-Valine, 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.

Optical rotation $[\alpha]_D^{20}$: +26.5 – +29.0° (after drying, 2 g, 6 mol/L hydrochloric acid TS, 25 mL, 100 mm).

pH Dissolve 0.5 g of L-Valine in 20 mL of water: the pH of this solution is between 5.5 and 6.5.

Purity (1) Clarity and color of solution—Dissolve 0.5 g of L-Valine in 20 mL of water: the solution is clear and colorless.

(2) Chloride—Perform the test with 0.5 g of L-Valine. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.021%).

(3) Sulfate—Perform the test with 0.6 g of L-Valine. Pre-

pare the control solution with 0.35 mL of 0.005 mol/L sulfuric acid VS (not more than 0.028%).

(4) Ammonium—Perform the test with 0.25 g of L-Valine. Prepare the control solution with 5.0 mL of Standard Ammonium Solution (not more than 0.02%).

(5) Heavy metals—Proceed with 1.0 g of L-Valine according to Method 1, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(6) Arsenic—Proceed with 1.0 g of L-Valine, prepare the test solution according to Method 2, and perform the test using Apparatus B (not more than 2 ppm).

(7) Other amino acids—Dissolve 0.10 g of L-Valine in 25 mL of water, and use this solution as the sample solution. Pipet 1 mL of the sample solution, and add water to make exactly 50 mL. Pipet 5 mL of this solution, add water to make exactly 20 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5 μ L 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 1-butanol, water and acetic acid (100) (3:1:1) to a distance of about 10 cm, and dry the plate at 80°C for 30 minutes. Spray evenly a solution of ninhydrin in acetone (1 in 50) on the plate, and heat at 80°C for 5 minutes: the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

Loss on drying Not more than 0.30% (1 g, 105°C, 3 hours).

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

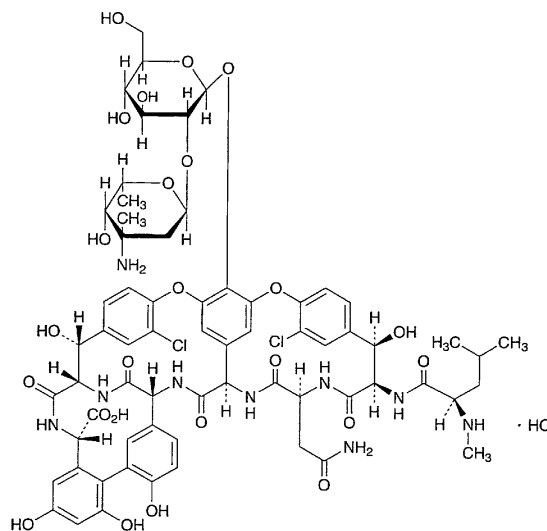
Assay Weigh accurately about 0.12 g of L-Valine, previously dried, and dissolve in 3 mL of formic acid, add 50 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 11.715 mg of C₅H₁₁NO₂

Containers and storage Containers—Tight containers.

Vancomycin Hydrochloride

塩酸バンコマイシン



C₆₆H₇₅Cl₂N₉O₂₄·HCl: 1485.71

(1*S*,2*R*,18*R*,19*R*,22*S*,25*R*,28*R*,40*S*)-50-[2-*O*-(3-Amino-2,3,6-trideoxy-3-*C*-methyl- α -*L*-lyxo-hexopyranosyl)- β -D-glucopyranosyloxy]-22-carbamoylmethyl-5,15-dichloro-2,18,32,35,37-pentahydroxy-19-[(2*R*)-4-methyl-2-(methylamino)pentanoylamino]-20,23,26,42,44-pentaoxo-7,13-dioxo-21,24,27,41,43-pentaazaocetacyclo-[26,14,2,2,3,6,2,14,17,1,8,12,1,29,33,0,10,25,0,34,39]pentaconta-3,5,8,10,12(50),14,16,29,31,33(49),34,36,38,45,47-pentadecaene-40-carboxylic acid monohydrochloride [1404-93-9]

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

Description Vancomycin Hydrochloride occurs as a white powder.

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

Vasopressin Injection

バソプレシン注射液

Vasopressin Injection is an aqueous solution for injection. It contains synthetic vasopressin or the pressor principle, vasopressin, obtained from the posterior lobe of the pituitary of healthy cattles and pigs, from which the majority of the oxytocic principle, oxytocin, has been removed.

It contains not less than 85% and not more than 120% of the labeled vasopressin Units.

Method of preparation Prepare as directed under Injections, with vasopressin prepared by synthesis or obtained from the posterior lobe of the pituitary.