make exactly 100 mL, and use this solution as the standard solution (2). Perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry. Determine the absorbances, A_{T1} and A_{S1}, of the sample solution and the standard solution (1) at 278 nm, and absorbances, A_{T2} and A_{S2}, of these solution, at 308 nm, respectively. Then determine the absorbance A_{S3} of the standard solution (2) at 278 nm.

$$\text{Amount (mg) of aspirin } (C_9H_8O_4) = \text{amount (mg) of aspirin for assay} \times \left[\frac{A_{T1} - \frac{A_{T2} \times A_{S1}}{A_{S2}}}{A_{S3}}\right]$$

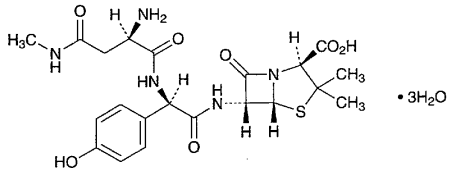
(2) Aluminum—Weigh accurately about 0.4 g of Aspirin Aluminum, and dissolve in 10 mL of sodium hydroxide TS. Add dropwise 1 mol/L hydrochloric acid TS to adjust the solution to a pH of about 1, add 20 mL of acetic acid-ammonium acetate buffer solution, pH 3.0, and 0.5 mL of Cu-PAN TS, and heat. While boiling, titrate with 0.05 mol/L disodium dihydrogen ethylenediamine tetraacetate VS until the color of the solution changes from red to yellow and persists for 1 minute. Perform a blank determination, and make any necessary correction.

Each mL of 0.05 mol/L disodium dihydrogen ethylenediamine tetraacetate VS
= 1.3491 mg of Al

Containers and storage Containers—Well-closed containers.

Aspoxicillin

アスポキシシリン



$C_{21}H_{27}N_5O_7S.3H_2O$: 547.58
(2*S*,5*R*,6*R*)-6-[(2*R*)-2-[(2*R*)-2-Amino-3-methylcarbamoylpropanoylamino]-2-(4-hydroxyphenyl)acetylamino]-3,3-dimethyl-7-oxo-4-thia-1-azabicyclo[3.2.0]heptane-2-carboxylic acid trihydrate
[*63358-49-6*, anhydride]

Aspoxicillin contains not less than 950 μg (potency) per mg, calculated on the anhydrous basis. The potency of Aspoxicillin is expressed as mass (potency) of aspoxicillin ($C_{21}H_{27}N_5O_7S$: 493.53).

Description Aspoxicillin occurs as a white, crystals or crystalline powder.

It is freely soluble in *N,N*-dimethylformamide, sparingly soluble in water, and practically insoluble in acetonitrile, in methanol and in ethanol (95).

Identification **(1)** Determine the absorption spectrum of a solution of Aspoxicillin (1 in 4000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of Aspoxicillin Reference Standard: both spectra exhibit similar intensities of absorption at the same wavelength.

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

Optical rotation $[\alpha]_D^{20}$: +170 – +185° (0.2 g calculated on the anhydrous bases, water, 20 mL, 100 mm).

pH Dissolve 0.1 g of Aspoxicillin in 50 mL of water: the pH of the solution is between 4.2 and 5.2.

Purity **(1)** Clarity and color of solution—Dissolve 1.0 g of Aspoxicillin in 50 mL of water: the solution is clear and colorless.

(2) Heavy metals—Proceed with 2.0 g of Aspoxicillin 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).

(3) Arsenic—Prepare the test solution with 2.0 g of Aspoxicillin according to Method 5, and perform the test using Apparatus B (not more than 1 ppm).

(4) Related substances—Dissolve 0.05 g of Aspoxicillin in 10 mL of the mobile phase, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add the mobile phase to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of these solutions as directed under the Liquid Chromatography according to the following conditions, and calculate the areas of each peak by the automatic integration method: the area of each peak other than aspoxicillin from the sample solution is not more than 3/10 of the peak area of aspoxicillin from the standard solution, and the total of peak areas other than aspoxicillin from the sample solution is not more than the peak area of aspoxicillin from the standard solution.

Operating conditions—

Detector, column, column temperature, mobile phase, and flow rate: Proceed as directed in the operating conditions in the Assay.

Time span of measurement: About 6 times as long as the retention time of aspoxicillin.

System suitability—

Test for required detection: Pipet 2 mL of the standard solution, add the mobile phase to make exactly 10 mL. Confirm that the peak area of aspoxicillin obtained from 10 μL of this solution is equivalent to 15 to 25% of that of aspoxicillin obtained from 10 μL of the standard solution.

System performance: Proceed as directed in the system suitability in the Assay.

System repeatability: When the test is repeated 6 times with 10 μL of the standard solution under the above operating conditions, the relative standard deviation of the peak areas of aspoxicillin is not more than 5%.

Water Not less than 9.5% and not more than 13.0% (0.2 g, volumetric titration, direct titration).

Assay Weigh accurately an amount of Aspoxicillin and Aspoxicillin Reference Standard, equivalent to about 0.1 g

(potency), dissolve each in a suitable amount of water, add exactly 10 mL of the internal standard solution, 6.5 mL of acetonitrile and water to make 50 mL, and use these solutions as the sample solution and the standard solution, respectively. Perform the test with 10 μL each of these solutions 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 aspoxicillin to that of the internal standard of each solution.

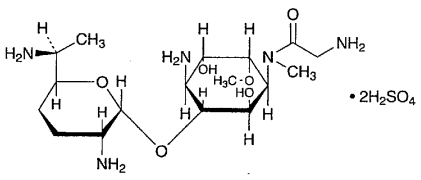
Amount [μg (potency)] of $C_{21}H_{27}N_5O_7S$
= amount [mg (potency)] of Aspoxicillin Reference Standard × $\frac{Q_T}{Q_S}$ × 1000

Internal standard solution—A solution of *N*-(3-hydroxyphenyl)acetamide (1 in 1000).
Operating conditions—
Detector: An ultraviolet absorption photometer (wavelength: 280 nm).
Column: A stainless steel column 4.6 mm in inside diameter and 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).
Column temperature: A constant temperature of about 40°C.
Mobile phase: To 130 mL of acetonitrile add potassium dihydrogenphosphate TS, pH 3.0 to make 1000 mL.
Flow rate: Adjust the flow rate so that the retention time of aspoxicillin is about 3 minutes.
System suitability—
System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, aspoxicillin and the internal standard are eluted in this order with the resolution between these peaks being not less than 8.
System repeatability: When the test is repeated 6 times with 10 μL of the standard solution under the above operating conditions, the relative standard deviation of the ratios of the peak area of aspoxicillin to that of the internal standard is not more than 0.8%.

Containers and storage Containers—Tight containers.

Astromicin Sulfate

硫酸アストロマイシン



$C_{17}H_{35}N_5O_6.2H_2SO_4$: 601.65
4-Amino-1-(2-amino-*N*-methylacetylamino)-1,4-dideoxy-3-*O*-(2,6-diamino-2,3,4,6,7-pentadeoxy-β-L-*lyxo*-heptopyranosyl)-6-*O*-methyl-1L-*chiro*-inositol disulfate
[*72275-67-3*]

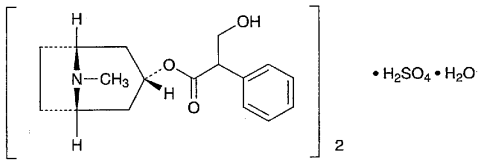
Astromicin Sulfate conforms to the requirements of Astromicin Sulfate in the Requirements for Antibiotic Products of Japan.

Description Astromicin Sulfate occurs as white to light yellowish white powder or masses.

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

Atropine Sulfate

硫酸アトロピン



$(C_{17}H_{23}NO_3)_2.H_2SO_4.H_2O$: 694.83
(1*R*,3*r*,5*S*)-8-Methyl-8-azabicyclo[3.2.1]oct-3-yl [(*RS*)-3-hydroxy-2-phenyl]propanoate hemisulfate hemihydrate
[*5908-99-6*]

Atropine Sulfate, when dried, contains not less than 98.0% of $(C_{17}H_{23}NO_3)_2.H_2SO_4$ (mol. wt.: 676.82).

Description Atropine Sulfate occurs as colorless crystals or a white, crystalline powder. It is odorless.

It is very soluble in water and in acetic acid (100), freely soluble in ethanol (95), and practically insoluble in diethyl ether.

Melting point: 188 – 194°C (with decomposition). Introduce a capillary tube charged with dried sample into a bath previously heated to 180°C, and continue to heat at a rate of rise of about 3°C per minute.

It is affected by light.

Identification **(1)** To 1 mg of Atropine Sulfate add 3 drops of fuming nitric acid, and evaporate the mixture on a water bath to dryness. Dissolve the residue in 1 mL of *N,N*-dimethylformamide, and add 5 to 6 drops of tetraethylammonium hydroxide TS: a red-purple color develops.

(2) To 2 mL of a solution of Atropine Sulfate (1 in 50) add 4 to 5 drops of hydrogen tetrachloroaurate (III) TS: a lusterless, yellowish white precipitate is formed.

(3) To 5 mL of a solution of Atropine Sulfate (1 in 25) add 2 mL of ammonia TS, and allow to stand for 2 to 3 minutes. Collect the precipitate, wash with water, and dry in a desiccator (in vacuum, silica gel) for 4 hours: it melts between 115°C and 118°C.

(4) A solution of Atropine Sulfate (1 in 20) responds to the Qualitative Tests for sulfate.

Purity **(1)** Clarity and color of solution —Dissolve 0.5 g of Atropine Sulfate in 10 mL of water: the solution is clear and colorless.

(2) Acid—Dissolve 1.0 g of Atropine Sulfate in 20 mL of water, and add 0.30 mL of 0.02 mol/L sodium hydroxide VS and 1 drop of methyl red-methylene blue TS: a green color develops.