(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.

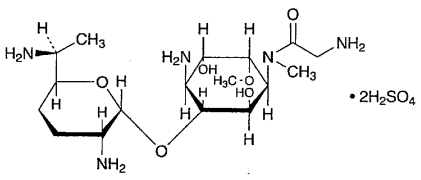
Amount [μg (potency)] of $C_{21}H_{27}N_5O_7S$
= amount [mg (potency)] of Aspoxicillin Reference Standard × $\frac{Q_T}{Q_S}$ × 1000

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*]

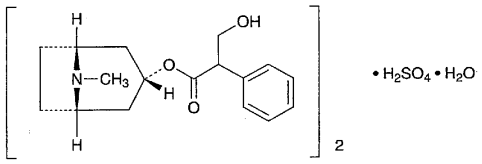
Astromicin Sulfate conforms to the requirements of Astromicin Sulfate in the Requirements for Antibiotic Products of Japan.

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.

(3) Other alkaloids—Dissolve 0.25 g of Atropine Sulfate in 1 mL of diluted hydrochloric acid (1 in 10), add water to make 15 mL, and use this solution as the sample solution.

(i) To 5 mL of the sample solution add 2 to 3 drops of hydrogen hexachloroplatinate (IV) TS: no precipitate is formed.

(ii) To 5 mL of the sample solution add 2 mL of ammonia TS, and shake vigorously: the turbidity of the solution is not greater than that of the following control solution.

Control solution: To 0.30 mL of 0.01 mol/L hydrochloric acid VS add 6 mL of dilute nitric acid and water to make 50 mL. To this solution add 1 mL of silver nitrate TS, and allow 7 mL of the mixture to stand for 5 minutes.

(4) Hyoscyamine—Weigh accurately about 1 g of Atropine Sulfate, previously dried, and dissolve in water to make exactly 10 mL: the specific optical rotation $[\alpha]_D^{20}$ of this solution in a 100-mm cell is between $-0.60°$ and $+0.10°$.

(5) Readily carbonizable substances—Take 0.20 g of Atropine Sulfate, and perform the test: the solution has no more color than Matching Fluid A.

Loss on drying Not more than 4.0% (0.5 g, in vacuum, phosphorus (V) oxide, 110°C, 4 hours).

Residue on ignition Not more than 0.1% (0.5 g).

Assay Dissolve about 0.25 g of Atropine Sulfate, previously dried and accurately weighed, in 30 mL of acetic acid (100). If necessary, dissolve it by warming, and cool. Titrate with 0.05 mol/L perchloric acid VS until the color of the solution changes from purple through blue to blue-green (indicator: 3 drops of crystal violet TS). Perform a blank determination, and make any necessary correction.

Each mL of 0.05 mol/L perchloric acid VS
= 33.841 mg of $(C_{17}H_{23}NO_3)_2.H_2SO_4$

Containers and storage Containers—Tight containers.
Storage—Light-resistant.

Atropine Sulfate Injection

硫酸アトロピン注射液

Atropine Sulfate Injection is an aqueous solution for injection. It contains not less than 93% and not more than 107% of the labeled amount of atropine sulfate $[(C_{17}H_{23}NO_3)_2.H_2SO_4.H_2O$: 694.83].

Method of preparation Prepare as directed under Injections, with Atropine Sulfate.

Description Atropine Sulfate Injection is a clear, colorless liquid.

pH: 4.0 - 6.0

Identification **(1)** Evaporate a volume of Atropine Sulfate Injection, equivalent to 1 mg of Atropine Sulfate according to the labeled amount, on a water bath to dryness. Proceed with the residue as directed in the Identification (1) under Atropine Sulfate.

(2) Heat an exactly measured volume of Atropine Sulfate Injection, equivalent to 5 mg of Atropine Sulfate according to the labeled amount, on a water bath to a volume of 5 mL, and use this solution as the sample solution. Separately, weigh accurately 0.050 g of Atropine Sulfate Reference Standard, dissolve in ethanol (95) to make exactly 10 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 50 μL of the sample solution and 10 μL of the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of chloroform and diethylamine (9:1) to a distance of about 10 cm, and air-dry the plate. Spray evenly hydrogen hexachloroplatinate (IV)-potassium iodide TS on the plate: the spots obtained from the sample solution and the standard solution show a purple color and the same *Rf* value.

(3) Atropine Sulfate Injection responds to the Qualitative Tests for sulfate.

Assay To an exactly measured volume of Atropine Sulfate Injection, equivalent to about 5 mg of atropine sulfate $[(C_{17}H_{23}NO_3)_2.H_2SO_4.H_2O]$, add water to make exactly 100 mL, and use this solution as the sample solution. Separately, dissolve about 5 mg of Atropine Sulfate Reference Standard (determine previously its loss on drying in the same manner as directed under Atropine Sulfate), accurately weighed, in water to make exactly 100 mL, and use this solution as the standard solution. Pipet 4 mL each of the sample solution and standard solution, add 2 mL of sodium hydroxide TS, 10 mL of potassium hydrogen phthalate buffer solution, pH 5.6, and 20 mL of chloroform to each of the solutions, and shake vigorously for 5 minutes. Cool down to about 10°C, centrifuge, and collect the chloroform layer. Perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry using chloroform as the blank. Determine the absorbances, A_T and A_S, of the subsequent solutions of the sample solution and the standard solution at 416 nm, respectively.

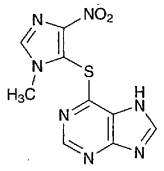
Amount (mg) of atropine sulfate $[(C_{17}H_{23}NO_3)_2.H_2SO_4.H_2O]$
= amount (mg) of Atropine Sulfate Reference Standard, calculated on the dried basis

$$\times \frac{A_T}{A_S} \times 1.0266$$

Containers and storage Containers—Hermetic containers.
Storage—Light-resistant.

Azathioprine

アザチオプリン



$C_9H_7N_7O_2S$: 277.26
6-(1-Methyl-4-nitro-1*H*-imidazol-5-ylthio)purine
[*446-86-6*]