

(3) Aldehyde—To 3 mL of a mixture of equal volumes of silver nitrate TS and aldehyde free-ethanol add ammonia TS dropwise until the precipitate first formed is redissolved. Add 1.0 mL of Amyl Nitrite, and warm between 60°C and 70°C for 1 minute: a brown to black color is not produced.

(4) Residue on evaporation—Evaporate 10.0 mL of Amyl Nitrite on a water bath in a draft, carefully protecting from flame, and dry the residue at 105°C for 1 hour: the mass of the residue is not more than 1.0 mg.

Assay Weigh accurately a volumetric flask containing 10 mL of ethanol (95), add about 0.5 g of Amyl Nitrite, and weigh accurately again. Add exactly 25 mL of 0.1 mol/L silver nitrate VS, then add 15 mL of potassium chlorate solution (1 in 20) and 10 mL of dilute nitric acid, stopper the flask immediately, and shake it vigorously for 5 minutes. Dilute with water to make exactly 100 mL, shake, and filter through dry filter paper. Discard the first 20 mL of the filtrate, measure exactly 50 mL of the subsequent filtrate, and titrate the excess silver nitrate with 0.1 mol/L ammonium thiocyanate VS (indicator: 2 mL of ammonium iron (III) sulfate TS). Perform a blank determination.

$$\begin{aligned} \text{Each mL of 0.1 mol/L silver nitrate VS} \\ = 35.144 \text{ mg of } C_5H_{11}NO_2 \end{aligned}$$

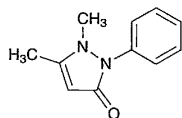
Containers and storage Containers—Hermetic containers not exceeding 10-ml capacity.

Storage—Light-resistant, in a cold place, and remote from fire.

Antipyrine

Phenazone

アンチピリン



$C_{11}H_{12}N_2O$: 188.23
1,5-Dimethyl-2-phenyl-1,2-dihydropyrazol-3-one
[60-80-0]

Antipyrine, when dried, contains not less than 99.0% of $C_{11}H_{12}N_2O$.

Description Antipyrine occurs as colorless or white crystals, or a white, crystalline powder. It is odorless, and has a slightly bitter taste.

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

A solution of Antipyrine (1 in 10) is neutral.

Identification (1) To 5 mL of a solution of Antipyrine (1 in 100) add 2 drops of sodium nitrite TS and 1 mL of dilute sulfuric acid: a deep green color develops.

(2) To 2 mL of a solution of Antipyrine (1 in 100) add 4 drops of dilute iron (III) chloride TS: a yellow-red color develops. Then add 10 drops of dilute sulfuric acid: the color changes to light yellow.

(3) To 5 mL of a solution of Antipyrine (1 in 100) add 2 to 3 drops of tannic acid TS: a white precipitate is produced.

(4) To 0.1 g of Antipyrine add 0.1 g of vanillin, 5 mL of water and 2 mL of sulfuric acid, boil the mixture, and cool: a yellow-red precipitate is produced.

Melting point 111 – 113°C

Purity (1) Chloride—Perform the test with 1.0 g of Antipyrine. Prepare the control solution with 0.40 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.014%).

(2) Heavy metals—Proceed with 1.0 g of Antipyrine 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).

(3) Readily carbonizable substances—Perform the test with 0.5 g of Antipyrine: the solution remains colorless.

Loss on drying Not more than 0.5% (1 g, silica gel, 4 hours).

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

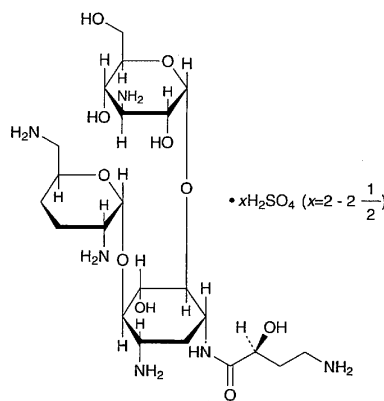
Assay Dissolve about 0.2 g of Antipyrine, previously dried and accurately weighed, in 20 mL of sodium acetate TS, add exactly 30 mL of 0.05 mol/L iodine VS, and allow to stand for 20 minutes with occasional shaking. Dissolve the precipitate in 10 mL of chloroform, and titrate the excess iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 3 mL of starch TS). Perform a blank determination.

$$\begin{aligned} \text{Each mL of 0.05 mol/L iodine VS} \\ = 9.411 \text{ mg of } C_{11}H_{12}N_2O \end{aligned}$$

Containers and storage Containers—Well-closed containers.

Arbekacin Sulfate

硫酸アルベカシン



$C_{22}H_{44}N_6O_{10} \cdot xH_2SO_4$ ($x = 2 - 2\frac{1}{2}$)
O-3-Amino-3-deoxy- α -D-glucopyranosyl-(1 \rightarrow 6)-O-[2,6-diamino-2,3,4,6-tetra-deoxy- α -D-erythro-hexopyranosyl-(1 \rightarrow 4)]-1-N-[(2S)-4-amino-2-hydroxybutanoyl]-2-deoxy-D-streptomine sulfate [51025-85-5, Arbekacin]

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

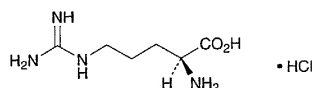
Description Arbekacin Sulfate occurs as a white powder.

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

L-Arginine Hydrochloride

Arginine Hydrochloride

塩酸 L-アルギニン



$C_6H_{14}N_4O_2 \cdot HCl$: 210.66

(2*S*)-2-Amino-5-guanidinopentanoic acid monohydrochloride [1119-34-2]

L-Arginine Hydrochloride, when dried, contains not less than 98.5% of $C_6H_{14}N_4O_2 \cdot HCl$.

Description L-Arginine Hydrochloride occurs as white crystals or crystalline powder. It is odorless, and has a slight, characteristic taste.

It is freely soluble in water and in formic acid, and very slightly soluble in ethanol (95).

Identification (1) Determine the infrared absorption spectrum of L-Arginine Hydrochloride, 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.

(2) A solution of L-Arginine Hydrochloride (1 in 50) responds to the Qualitative Tests for chloride.

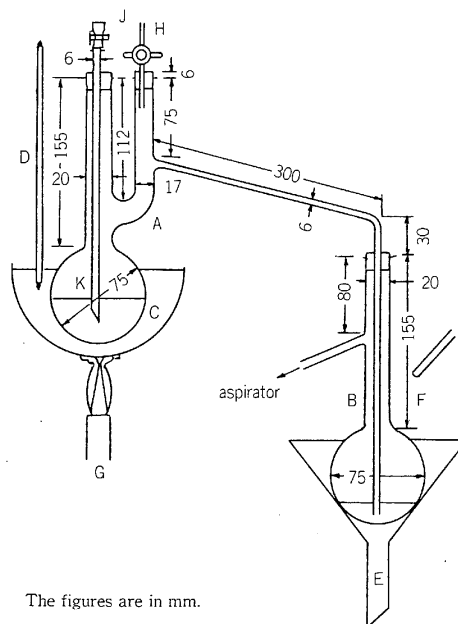
Optical rotation $[\alpha]_D^{20}$: +21.5 – +23.5° (after drying, 2 g, 6 mol/L hydrochloric acid TS, 25 mL, 100 mm).

pH Dissolve 1.0 g of L-Arginine Hydrochloride in 10 mL of water: the pH of this solution is between 4.7 and 6.2.

Purity (1) Clarity and color of solution—Dissolve 1.0 g of L-Arginine Hydrochloride in 10 mL of water: the solution is clear and colorless.

(2) Sulfate—Perform the test with 0.6 g of L-Arginine Hydrochloride. Prepare the control solution with 0.35 mL of 0.005 mol/L sulfuric acid VS (not more than 0.028%).

(3) Ammonium—(i) Apparatus: Use the apparatus illustrated in the figure. It is thoroughly constructed of hard glass, and ground glass surfaces may be used for joints. All rubber parts used in the apparatus should be boiled for 10 to 30 minutes in sodium hydroxide TS and for 30 to 60 minutes in water, and finally washed thoroughly with water before use.



The figures are in mm.

- A: Vacuum distilling flask (200 mL)
- B: Receiver (200-mL flask)
- C: Water bath
- D: Thermometer
- E: Funnel
- F: Cooling water
- G: Gas burner
- H: Glass cock
- J: Rubber tube with screw cock
- K: Glass tube for bumping prevention

(ii) Procedure: Take 0.25 g of L-Arginine Hydrochloride in a vacuum distilling flask A, add 70 mL of water and 1 g of magnesium oxide, and connect the flask with a vacuum distilling apparatus. Place 20 mL of a solution of boric acid (1 in 200) as an absorbing liquid in receiver B, immerse the end of the branch of the vacuum distilling flask in the absorbing liquid, and distil in a water bath of 60°C, adjusting the degree of vacuum so that the distillatory rate is between 1 mL and 2 mL per minute, until 30 mL of distillate is obtained. During distillation, cool the bulb of receiver B with water. Lift the end of the branch from the absorbing liquid, rinsing the end with a small quantity of water, and add water to make exactly 100 mL. Perform the test using this solution as the test solution. Prepare the control solution as follows: take 5.0 mL of Standard Ammonium Solution in vacuum distilling flask A, and proceed in the same manner as the preparation of the test solution (not more than 0.02%).

(4) Heavy metals—Proceed with 1.0 g of L-Arginine Hydrochloride 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).

(5) Arsenic—Prepare the test solution with 1.0 g of L-Arginine Hydrochloride according to Method 1, and perform the test using Apparatus B (not more than 2 ppm).

(6) Other amino acids—Dissolve 0.20 g of L-Arginine Hydrochloride in 10 mL of water, and use this solution as the sample solution. Pipet 1 mL of the sample solution, and add water to make exactly 10 mL. Pipet 1 mL of this solu-