

(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.

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

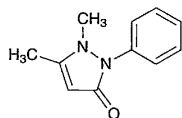
Containers and storage Containers—Hermetic containers not exceeding 10-ml capacity.

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).

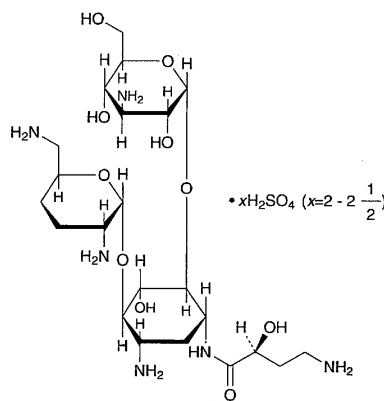
Assay Dissolve about 0.2 g of Antipyrine, previously dried and accurately weighed, in 20 mL of sodium acetate TS, add exactly 30 mL of 0.05 mol/L iodine VS, and allow to stand for 20 minutes with occasional shaking. Dissolve the precipitate in 10 mL of chloroform, and titrate the excess iodine with 0.1 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-O-acetyl- 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.