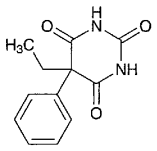
Phenobarbital

フェノバルビタール



$C_{12}H_{12}N_2O_3$: 232.24
5-Ethyl-5-phenylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione
[*50-06-6*]

Phenobarbital, when dried, contains not less than 99.0% of $C_{12}H_{12}N_2O_3$.

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

It is very soluble in *N,N*-dimethylformamide, freely soluble in ethanol (95), in acetone and in pyridine, soluble in diethyl ether, and very slightly soluble in water.

It dissolves in sodium hydroxide TS and in ammonia TS.

The pH of a saturated solution of Phenobarbital is between 5.0 and 6.0.

Identification (1) Boil 0.2 g of Phenobarbital with 10 mL of sodium hydroxide TS: the gas evolved changes moistened red litmus paper to blue.

(2) Dissolve 0.1 g of Phenobarbital in 5 mL of diluted pyridine (1 in 10), shake the solution with 0.3 mL of copper (II) sulfate TS, and allow to stand for 5 minutes: a light red-purple precipitate is produced. Shake the mixture with 5 mL of chloroform: the chloroform layer remains colorless. Dissolve 0.1 g of Phenobarbital in a mixture of 2 to 3 drops of ammonia-ammonium chloride buffer solution, pH 10.7, and 5 mL of diluted pyridine (1 in 10), then add 5 mL of chloroform and 0.3 mL of copper (II) sulfate TS: a light red-purple precipitate is produced in the water layer. Shake again: the chloroform layer remains colorless.

(3) Shake 0.4 g of Phenobarbital with 0.1 g of anhydrous sodium carbonate and 4 mL of water, and add a solution of 0.3 g of 4-nitrobenzyl chloride in 7 mL of ethanol (95). Heat on a water bath for 30 minutes under a reflux condenser, and allow to stand for 1 hour. Filter the crystals, wash with 7 mL of sodium hydroxide TS, then with a small amount of water, recrystallize from a mixture of ethanol (95) and chloroform (1:1), and dry at 105°C for 30 minutes: the crystals melt between 181°C and 185°C.

(4) Dissolve 0.1 g of Phenobarbital in 2 mL of sulfuric acid, shake the solution with 5 to 6 mg of potassium nitrate, and allow to stand for 10 minutes: a yellow to yellow-brown color develops.

Melting point 175 - 179°C

Purity (1) Clarity and color of solution—Dissolve 0.5 g of Phenobarbital in 5 mL of sodium hydroxide TS: the solution is clear and colorless.

(2) Chloride—Dissolve 0.30 g of Phenobarbital in 20 mL of acetone, and 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: take 0.30 mL of 0.01 mol/L hydrochloric acid VS, 20 mL of acetone and 6 mL of dilute nitric acid, and add water to make 50 mL (not more than 0.035%).

(3) Sulfate—Dissolve 0.40 g of Phenobarbital in 20 mL of acetone, and add 1 mL of dilute hydrochloric acid and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution as follows: take 0.40 mL of 0.005 mol/L sulfuric acid VS, 20 mL of acetone, and 1 mL of dilute hydrochloric acid, and add water to make 50 mL (not more than 0.048%).

(4) Heavy metals—Proceed with 1.0 g of Phenobarbital 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) Phenylbarbituric acid—Boil 1.0 g of Phenobarbital with 5 mL of ethanol (95) for 3 minutes: the solution is clear.

(6) Readily carbonizable substances—Perform the test with 0.5 g of Phenobarbital. The solution has not more color than Matching Fluid A.

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

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

Assay Weigh accurately about 0.5 g of Phenobarbital, previously dried, dissolve in 50 mL of *N,N*-dimethylformamide, and titrate with 0.1 mol/L potassium hydroxide-ethanol VS until the color of the solution change from yellow to yellow-green (indicator: 1 mL of alizarin yellow GG-thymolphthalein TS). Perform a blank determination using a mixture of 50 mL of *N,N*-dimethylformamide and 22 mL of ethanol (95), and make any necessary correction.

Each mL of 0.1 mol/L potassium hydroxide-ethanol VS
= 23.224 mg of $C_{12}H_{12}N_2O_3$

Containers and storage Containers—Well-closed containers.

10% Phenobarbital Powder

Phenobarbital Powder

フェノバルビタール散 10%

10% Phenobarbital Powder contains not less than 9.3% and not more than 10.7% of phenobarbital ($C_{12}H_{12}N_2O_3$: 232.24).

Method of preparation

Phenobarbital	100 g
Starch, Lactose or their mixture	a sufficient quantity
	To make 1000 g

Prepare as directed under Powders, with the above ingredients.

Identification Shake thoroughly 5 g of 10% Phenobarbital Powder with 20 mL of hexane, and filter. Collect the residue, and dry on a water bath, then extract with four 30-mL portions of chloroform. Filter the combined chloroform extracts, and evaporate the filtrate to dryness. Dry the residue at 105°C for 1 hour: the residue so obtained