Phenobarbital Tablets

フェノバルビタール錠

Phenobarbital Tablets contain not less than 95.0% and not more than 105.0% of the labeled amount of phenobarbital ($C_{12}H_{12}N_2O_3$: 232.24).

Method of preparation Prepare as directed under Tablets, with Phenobarbital.

Identification To a quantity of powdered Phenobarbital Tablets, equivalent to 20 mg of Phenobarbital, add 20 mL of boric acid-potassium chloride-sodium hydroxide buffer solution (pH 9.6), shake, and centrifuge. To 1 mL of the supernatant liquid add boric acid-potassium chloride-sodium hydroxide buffer solution (pH 9.6) to make 100 mL. Determine the absorption spectrum of this solution as directed under Ultraviolet-visible Spectrophotometry *<2.24>*: it exhibits a maximum between 238 nm and 242 nm.

Uniformity of dosage units *<6.02>* Perform the Mass variation test, or the Content uniformity test according to the following method: it meets the requirement.

To 1 tablet of Phenobarbital Tablets add exactly V mL of a mixture of water and acetonitrile (1:1) so that each mL contains about 1 mg of phenobarbital ($C_{12}H_{12}N_2O_3$), sonicate to disintegrate, shake for 10 minutes, and centrifuge. Then, proceed as directed in the Assay.

Amount (mg) of phenobarbital ($C_{12}H_{12}N_2O_3$)
$= M_S \times A_T / A_S \times V / 30$

M_S: Amount (mg) of phenobarbital for assay taken

Dissolution *<6.10>* When the test is performed at 50 revolutions per minute according to the Paddle method, using 900 mL of water as the dissolution medium, the dissolution rate in 30 minutes of Phenobarbital Tablets is not less than 75%.

Start the test with 1 tablet of Phenobarbital Tablets, withdraw not less than 20 mL of the medium at the specified minute after starting the test, and filter through a membrane filter with a pore size not exceeding 0.45 μm. Discard the first 10 mL of the filtrate, pipet V mL of the subsequent filtrate, and add water to make exactly V' mL so that each mL contains about 33 μg of phenobarbital ($C_{12}H_{12}N_2O_3$). Pipet 5 mL of this solution, add exactly 10 mL of boric acid-potassium chloride-sodium hydroxide buffer solution (pH 9.6), and use this solution as the sample solution. Separately, weigh accurately about 17 mg of phenobarbital for assay, previously dried at 105ºC for 2 hours, and dissolve in water to make exactly 100 mL. Pipet 5 mL of this solution, and add water to make exactly 25 mL. Pipet 5 mL of this solution, add exactly 10 mL of boric acid-potassium chloride-sodium hydroxide buffer solution (pH 9.6), and use this solution as the standard solution. Determine the absorbances, A_T and A_S, at 240 nm of the sample solution and standard solution as directed under Ultraviolet-visible Spectrophotometry *<2.24>*, using a mixture of boric acid-potassium chloride-sodium hydroxide buffer solution (pH 9.6) and water (2:1) as the blank.

Dissolution rate (%) with respect to the labeled amount of phenobarbital ($C_{12}H_{12}N_2O_3$)
$= M_S \times A_T / A_S \times V' / V \times 1 / C \times 180$

M_S: Amount (mg) of phenobarbital for assay taken
C: Labeled amount (mg) of phenobarbital ($C_{12}H_{12}N_2O_3$) in 1 tablet

Assay Weigh accurately the mass of not less than 20 tablets of Phenobarbital Tablets, and powder. Weigh accurately a portion of the powder, equivalent to about 30 mg of phenobarbital ($C_{12}H_{12}N_2O_3$), add exactly 30 mL a mixture of water and acetonitrile (1:1), shake for 10 minutes, and centrifuge. Pipet 1 mL of the supernatant liquid, add a mixture of water and acetonitrile (1:1) to make exactly 20 mL, and use this solution as the sample solution. Separately, weigh accurately about 30 mg of phenobarbital for assay, previously dried at 105ºC for 2 hours, and dissolve in a mixture of water and acetonitrile (1:1) to make exactly 30 mL. Pipet 1 mL of this solution, add a mixture of water and acetonitrile (1:1) to make exactly 20 mL, and use this solution as the standard solution. Perform the test with exactly 10 μL each of the sample solution and standard solution as directed under Liquid Chromatography *<2.01>* according to the following conditions, and determine the peak areas, A_T and A_S, of phenobarbital in each solution.

Amount (mg) of phenobarbital ($C_{12}H_{12}N_2O_3$)
$= M_S \times A_T / A_S$

M_S: Amount (mg) of phenobarbital for assay taken

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 210 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 45ºC.

Mobile phase: A mixture of water and acetonitrile for liquid chromatography (11:9).

Flow rate: Adjust so that the retention time of phenobarbital 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, the number of theoretical plates and the symmetry factor of the peak of phenobarbital are not less than 3000 and not more than 1.5, respectively.

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 area of phenobarbital is not more than 1.0%.

Containers and storage Containers—Well-closed containers.