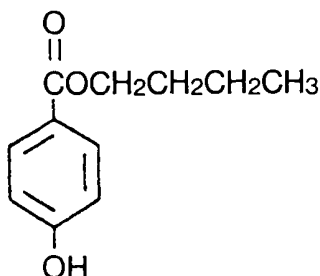


D. MONOGRAPHS

Butyl *p*-Hydroxybenzoate



C₁₁H₁₄O₃

Mol. Wt. 194.23

butyl 4-hydroxybenzoate

[94-26-8]

Content Butyl *p*-Hydroxybenzoate, when dried, contains not less than 99.0% of butyl *p*-hydroxybenzoate (C₁₁H₁₄O₃).

Description Butyl *p*-Hydroxybenzoate occurs as colorless crystals or as a white crystalline powder. It is odorless.

Identification (1) To 0.5 g of Butyl *p*-Hydroxybenzoate, add 10 ml of sodium hydroxide solution (1 : 25), boil for 30 minutes, evaporate to about 5 ml. Cool, acidify with diluted sulfuric acid (1 : 20), collect the formed precipitate by filtration, wash thoroughly with water, and dry at 105 °C for 1 hour. The melting point is 213 - 217 °C.

(2) To 0.05 g of Butyl *p*-Hydroxybenzoate, add 2 drops of acetic acid and 5 drops of sulfuric acid, and warm for 5 minutes. An odor of butyl acetate is evolved.

Purity (1) Melting point 69 - 72 °C

(2) Free acid Not more than 0.55% as *p*-hydroxybenzoic acid.

Weigh 0.75 g of Butyl *p*-Hydroxybenzoate, add 15 ml of water, heat in a boiling water bath for 1 minute, cool, and filter. The filtrate is acidic or neutral. Measure 10 ml of the filtrate, and add 0.20 ml of 0.1 mol/l sodium hydroxide and 2 drops of methyl red TS. The color of the solution changes to yellow.

(3) Sulfate Not more than 0.024% as SO₄.

Sample Solution Weigh 1.0 g of Butyl *p*-Hydroxybenzoate, add 100 ml of boiling water, and heat for 5 minutes while shaking well. After cooling, add water to make 100 ml, filter, and measure 40 ml of the filtrate.

Control Solution Prepare the control solution with 0.20 ml of 0.005 mol/l sulfuric acid.

(4) Heavy metals Not more than 10 µg/g as Pb.

Test Solution Weigh 2.0 g of Butyl *p*-Hydroxybenzoate, dissolve in 25 ml of acetone, and add 2 ml of diluted acetic acid (1 : 20) and water to make 50 ml.

Control Solution To 2.0 ml of Lead Standard Solution, add 25 ml of acetone, 2

D. MONOGRAPHS

ml of diluted acetic acid (1 : 20), and water to make 50 ml.

(5) Arsenic Not more than 4.0 $\mu\text{g/g}$ as As_2O_3 (0.50 g, Method 3, Apparatus B).

Loss on Drying Not more than 0.50% (5 hours).

Residue on Ignition Not more than 0.10% .

Assay Weigh accurately about 2 g of Butyl *p*-Hydroxybenzoate, previously dried, add 40 ml of 1 mol/l sodium hydroxide, exactly measured, and boil for 30 minutes. After cooling, titrate the excess alkali with 0.5 mol/l sulfuric acid (indicator: 5 drops of bromothymol blue TS). The color at the end point is that of the solution prepared by adding the same indicator to phosphate buffer (pH 6.5). Perform a blank test in the same manner.

1 ml of 1 mol/l sodium hydroxide = 194.23 mg of $\text{C}_{11}\text{H}_{14}\text{O}_3$