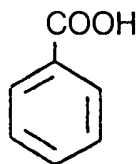


## D. MONOGRAPHS

### Benzoic Acid



$C_7H_6O_2$

benzenecarboxylic acid

Mol.Wt.122.12

[ 65-85-0 ]

**Content** Benzoic Acid, when dried, contains not less than 99.5% of benzoic acid ( $C_7H_6O_2$ ).

**Description** Benzoic Acid occurs as white laminar crystals or needles. It is odorless or has a slight odor of benzaldehyde.

**Identification** Dissolve 1 g of Benzoic Acid in 20 ml of sodium hydroxide solution (1 : 25). The solution responds to test (2) for Benzoate as described in the Qualitative Tests.

**Purity** (1) Melting point 121 - 123 °C.

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

**Test Solution** Weigh 2.0 g of Benzoic Acid, 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 ml of diluted acetic acid (1 : 20), and water to make 50 ml.

(3) Arsenic Not more than 4.0 µg/g as  $As_2O_3$  (0.50 g, Method 3, Apparatus B).

(4) Readily oxidizable substances Add 1.5 ml of sulfuric acid to 100 ml of water, add dropwise 0.02 mol/l potassium permanganate while boiling until the pink color persists for 30 seconds. Weigh 1.0 g of Benzoic Acid, and dissolve in the solution. Titrate with 0.02 mol/l potassium permanganate at about 70 °C until the pink color persists for 15 seconds. The amount is not more than 0.5 ml.

(5) Chlorinated compounds Not more than 0.014% as Cl.

**Test Solution** Weigh 0.50 g of Benzoic Acid and 0.7 g of calcium carbonate, transfer together in a porcelain crucible, add a small amount of water, mix, dry at 100 °C, and heat at about 600 °C for 10 minutes. Cool, add 20 ml of diluted nitric acid (1 : 10) to dissolve the residue, and filter. Wash the insoluble substances with about 15 ml of water, combine the washings and the filtrate, add water to make 50 ml.

**Control Solution** Weigh 0.7 g of calcium carbonate, dissolve in 20 ml of diluted nitric acid (1 : 10), filter if necessary, add 0.20 ml of 0.01 mol/l hydrochloric acid and water to make 50 ml.

**Procedure** Add 0.5 ml of silver nitrate solution (1 : 50) to both solutions, shake well, and allow to stand for 5 minutes. The test solution is not more turbid than the

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control solution.

(6) Phthalic acid Not more than 50 µg/g.

*Test Solution* Weigh 1.0 g of Benzoic Acid, dissolve in 20 ml of methanol, add diluted acetic acid (1 : 100) to make exactly 50 ml.

*Control Solution* Weigh 10.0 mg of phthalic acid, dissolve in 30 ml of methanol, add diluted acetic acid (1 : 100) to make exactly 100 ml. Measure 1.0 ml of this solution, add methanol-diluted acetic acid (1 : 100) mixture (2 : 3) to make exactly 100 ml. solution as the control solution.

*Procedure* Measure 20 µl each of the test solution and the control solution, and proceed as directed under Liquid Chromatography under the operating conditions given below. The peak height of the phthalic acid of the test solution does not exceed that of the control solution.

### *Operating Conditions*

Detector: Detector for absorbances in the ultraviolet region (determination wavelength: 228 nm).

Column packing material: 7-µm octadecylsilanized silica gel.

Column: Stainless steel tube 4.6 mm in internal diameter and 250 mm in length.

Column temperature: 40 °C.

Mobile phase: Mixture of methanol and diluted acetic acid (1 : 100) (3 : 7).

Flow rate: 1 ml/minute.

**Loss on Drying** Not more than 0.50% (3 hours).

**Assay** Weigh accurately about 0.25 g of Benzoic Acid, previously dried, dissolve in 25 ml of 50% (vol) ethanol neutralized with 0.1 mol/l sodium hydroxide, and titrate with 0.1 mol/l sodium hydroxide (indicator: 3 drops of phenol red TS).

1 ml of 0.1 mol/l sodium hydroxide = 12.212 mg of C<sub>7</sub>H<sub>6</sub>O<sub>2</sub>