

Methyl Parahydroxybenzoate, when dried, contains not less than 99.0% of $C_8H_8O_3$.

Description Methyl Parahydroxybenzoate, occurs as colorless crystals or a white, crystalline powder. It is odorless and tasteless, and numbs the tongue.

It is freely soluble in ethanol (95), in acetone and in diethyl ether, soluble in hot water, and slightly soluble in water.

Its saturated solution is slightly acid.

Identification (1) Dissolve 0.25 g of Methyl Parahydroxybenzoate in 5 mL of dilute ethanol, and add 1 drop of iron (III) chloride TS: a red-purple color develops.

(2) Boil 0.5 g of Methyl Parahydroxybenzoate with 10 mL of sodium hydroxide TS for about 30 minutes, allowing the solution to evaporate to about 5 mL. After cooling, acidify with dilute sulfuric acid, collect the precipitate formed, wash thoroughly with a small amount of water, and dry in a desiccator (silica gel): the precipitate melts between 213°C and 217°C.

(3) To 0.05 g of Methyl Parahydroxybenzoate add 2 drops of acetic acid (31) and 5 drops of sulfuric acid, and heat the mixture for 5 minutes: the odor of methyl acetate is perceptible.

Melting point 125 – 128°C

Purity (1) Chloride—Heat 2.0 g of Methyl Parahydroxybenzoate with 50 mL of water, allow to stand in ice water for 1 hour with occasional shaking, then add water to make 100 mL, and filter. Perform the test with 25 mL of the filtrate. Prepare the control solution with 0.50 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.035%).

(2) Sulfate—Perform the test with 40 mL of the filtrate obtained in Purity (1). Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid VS (not more than 0.024%).

(3) Heavy metals—Dissolve 1.0 g of Methyl Parahydroxybenzoate in 25 mL of acetone, add 2 mL of dilute acetic acid and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution as follows: to 2.0 mL of Standard Lead Solution add 25 mL of acetone, 2 mL of dilute acetic acid, and water to make 50 mL (not more than 20 ppm).

(4) Parahydroxybenzoic acid and salicylic acid—Dissolve 0.50 g of Methyl Parahydroxybenzoate in 30 mL of diethyl ether, shake with 20 mL of a solution of sodium hydrogen carbonate (1 in 100), wash the separated aqueous layer with two 20-mL portions of diethyl ether, shake the aqueous layer with 5 mL of dilute sulfuric acid and 30 mL of diethyl ether, and allow to stand. Shake gently the separated diethyl ether layer with 10 mL of water, remove the aqueous layer after allowing the mixture to stand, filter the diethyl ether solution, wash the vessel and the filter paper with a small amount of diethyl ether, evaporate the diethyl ether from the combined filtrate and washings on a water bath, and dry the residue in a desiccator (silica gel) to constant mass: the mass of the residue is not more than 5.0 mg. Warm the residue with 5 mL of water, filter, and to the filtrate add 2 to 3 drops of dilute iron (III) chloride TS: no purple color develops.

(5) Readily carbonizable substances—Perform the test with 0.50 g of Methyl Parahydroxybenzoate. The solution has no more color than Matching Fluid D.

Loss on drying Not more than 0.5% (2 g, silica gel, 3 hours).

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

Assay Weigh accurately about 2 g of Methyl Parahydroxybenzoate, previously dried, add exactly 40 mL of 1 mol/L sodium hydroxide VS, and boil for 30 minutes. Cool, and titrate the excess sodium hydroxide with 0.5 mol/L sulfuric acid VS until the solution shows the same color as that of phosphate buffer solution, pH 6.5, to which the same indicator has been added (indicator: 5 drops of bromothymol blue TS). Perform a blank determination.

Each mL of 1 mol/L sodium hydroxide VS
= 152.15 mg of $C_8H_8O_3$

Containers and storage Containers—Well-closed containers.

Compound Methyl Salicylate Spirit

複方サリチル酸メチル精

Method of preparation

Methyl Salicylate	40 mL
Capsicum Tincture	100 mL
<i>d</i> - or <i>dl</i> -Camphor	50 g
Ethanol	a sufficient quantity
To make 1000 mL	

Prepare as directed under Medicated Spirits, with the above ingredients.

Description Compound Methyl Salicylate Spirit is a reddish yellow liquid, having a characteristic odor and a burning taste.

Identification (1) Shake 1 mL of Compound Methyl Salicylate Spirit with 5 mL of dilute ethanol, and add 1 drop of iron (III) chloride TS: a purple color is produced (methyl salicylate).

(2) Shake thoroughly 0.5 mL of Compound Methyl Salicylate Spirit with 10 mL of chloroform, and use this solution as the sample solution. Dissolve 0.04 g of methyl salicylate in 10 mL of chloroform, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5 μ L each of the sample solution and the standard solution on the plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of hexane and chloroform (4:1) to a distance of about 10 cm, air-dry the plate, and examine under ultraviolet light (main wavelength: 254 nm): the spots from the sample solution and the standard solution show the same *R_f* value. Spray evenly iron (III) chloride TS upon the plate: the spot from the standard solution and the corresponding spot from the sample solution reveal a purple color.

Containers and storage Containers—Tight containers.