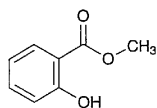


Methyl Salicylate

サリチル酸メチル



$C_8H_8O_3$: 152.15
Methyl 2-hydroxybenzoate [119-36-8]

Methyl Salicylate contains not less than 98.0% of $C_8H_8O_3$.

Description Methyl Salicylate is a colorless to pale yellow liquid. It has a strong, characteristic odor.

It is miscible with ethanol (95) and with diethyl ether.

It is very slightly soluble in water.

Specific gravity d_{20}^{20} : 1.182 – 1.192

Boiling point: 219 – 224°C

Identification Shake 1 drop of Methyl Salicylate thoroughly with 5 mL of water for 1 minute, and add 1 drop of iron (III) chloride TS: a purple color develops.

Purity (1) Acid—Shake 5.0 mL of Methyl Salicylate thoroughly with 25 mL of freshly boiled and cooled water and 1.0 mL of 0.1 mol/L sodium hydroxide VS for 1 minute, add 2 drops of phenol red TS, and titrate with 0.1 mol/L hydrochloric acid VS until the red color disappears: not more than 0.45 mL of 0.1 mol/L sodium hydroxide VS is consumed.

(2) Heavy metals—Shake 10.0 mL of Methyl Salicylate thoroughly with 10 mL of water, add 1 drop of hydrochloric acid, and saturate with hydrogen sulfide by passing it through the mixture: neither the oily layer nor the aqueous layer shows a dark color.

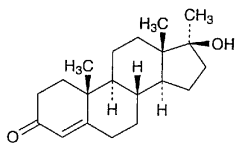
Assay Weigh accurately about 2 g of Methyl Salicylate, add an exactly measured 50 mL of 0.5 mol/L potassium hydroxide-ethanol VS, and heat on a water bath for 2 hours under a reflux condenser. Cool, and titrate the excess potassium hydroxide with 0.5 mol/L hydrochloric acid VS (indicator: 3 drops of phenolphthalein TS). Perform a blank determination.

Each mL of 0.5 mol/L potassium hydroxide-ethanol VS = 76.07 mg of $C_8H_8O_3$

Containers and storage Containers—Tight containers.

Methyltestosterone

メチルテストステロン



$C_{20}H_{30}O_2$: 302.45
17 β -Hydroxy-17 α -methylandroster-4-en-3-one [58-18-4]

Methyltestosterone, when dried, contains not less than 97.0% and not more than 103.0% of $C_{20}H_{30}O_2$.

Description Methyltestosterone occurs as white to pale yellow crystals or crystalline powder. It is odorless.

It is freely soluble in methanol and in ethanol (95), soluble in acetone, sparingly soluble in diethyl ether, and practically insoluble in water.

Identification (1) Dissolve 1 mg of Methyltestosterone in 2 mL of a solution of xanthydrol in acetic acid (100) (1 in 200), add 0.2 mL of sulfuric acid, and heat in a water bath for 20 minutes. After cooling, add 6 mL of water and 5 mL of chloroform, and shake: a green color develops in the chloroform layer. To the chloroform layer add 5 mL of diluted sulfuric acid (4 in 5), and mix: the color of the solution does not change.

(2) Dissolve 0.02 g of Methyltestosterone in 5 mL of methanol, add 3.5 mL of a solution of 0.05 g of hydroxylammonium chloride and 0.05 g of anhydrous sodium acetate in 25 mL of methanol, boil under a reflux condenser for 2 hours, add 15 mL of water, and filter the precipitate by suction. Wash the precipitate with water, and recrystallize from diluted methanol (7 in 10), and dry in a desiccator (in vacuum, phosphorus (V) oxide) for 4 hours: the crystals melt between 212°C and 218°C.

Optical rotation $[\alpha]_D^{20}$: +79 – +85° (after drying, 0.1 g, ethanol (95), 10 mL, 100 mm).

Melting point 163 – 168°C

Purity Other steroids—Dissolve 0.040 g of Methyltestosterone in 2 mL of ethanol (95), and use this solution as the sample solution. Pipet 1 mL of this solution, add ethanol (95) to make exactly 100 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μ L each of the sample solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of chloroform and diethylamine (19:1) to a distance of about 15 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

Loss on drying Not more than 1.0% (0.5 g, in vacuum, phosphorus (V) oxide, 10 hours).

Residue on ignition Not more than 0.1% (0.5 g).

Assay Weigh accurately about 0.01 g of Methyltestosterone, previously dried, and dissolve in ethanol (95) to make exactly 100 mL. To exactly 5 mL of this solution add ethanol (95) to make exactly 50 mL. Determine the absorbance A of this solution at the wavelength of maximum absorption at about 241 nm as directed under the Ultraviolet-visible Spectrophotometry.

$$\text{Amount (mg) of } C_{20}H_{30}O_2 = \frac{A}{536} \times 10000$$

Containers and storage Containers—Tight containers.
Storage—Light-resistant.