

amount of camphor, chloral hydrate or thymol: the mixture liquefies.

(2) Shake 1 g of *dl*-Menthol with 20 mL of sulfuric acid: the mixture becomes turbid with a yellow-red color. Allow to stand for 3 hours: a clear, oily layer possesses no aroma of menthol is separated.

**Congealing point** 27 – 28°C

**Optical rotation**  $[\alpha]_D^{20}$ : –2.0 – +2.0° (2.5 g, ethanol (95), 25 mL, 100 mm).

**Purity (1)** Non-volatile residue—Volatilize 2.0 g of *dl*-Menthol on a water bath, and dry the residue at 105°C for 2 hours: the residue weighs not more than 1.0 mg.

(2) Thymol—Add 0.20 g of *dl*-Menthol to a cold mixture of 2 mL of acetic acid (100), 6 drops of sulfuric acid and 2 drops of nitric acid: no green to blue-green color immediately develops.

(3) Nitromethane or nitroethane—To 0.5 g of *dl*-Menthol placed in a flask add 2 mL of a solution of sodium hydroxide (1 in 2) and 1 mL of strong hydrogen peroxide, connect a reflux condenser to the flask, and boil the mixture gently for 10 minutes. After cooling, add water to make exactly 20 mL, and filter. Take 1 mL of the filtrate in a Nessler tube, add water to make 10 mL, neutralize with dilute hydrochloric acid, then add 1 mL of dilute hydrochloric acid, and cool. To the mixture add 1 mL of a solution of sulfanilic acid (1 in 100), allow to stand for 2 minutes, and then add 1 mL of a solution of *N*-(1-naphthyl)-*N'*-diethylethylenediamine oxalate (1 in 1000) and water to make 25 mL: no red-purple color immediately develops.

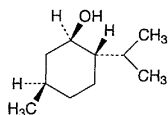
**Assay** Weigh accurately about 2 g of *dl*-Menthol, add exactly 20 mL of a mixture of dehydrated pyridine and acetic anhydride (8:1), connect a reflux condenser, and heat on a water bath for 2 hours. Wash down the condenser with 20 mL of water, and titrate with 1 mol/L sodium hydroxide VS (indicator: 5 drops of phenolphthalein TS). Perform a blank determination, and make any necessary correction.

Each mL of 1 mol/L sodium hydroxide VS  
= 156.27 mg of C<sub>10</sub>H<sub>20</sub>O

**Containers and storage** Containers—Tight containers.  
Storage—In a cold place.

## *l*-Menthol

*l*-メントール



C<sub>10</sub>H<sub>20</sub>O: 156.27  
(1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexanol  
[2216-51-5]

*l*-Menthol contains not less than 98.0% of C<sub>10</sub>H<sub>20</sub>O.

**Description** *l*-Menthol occurs as colorless crystals. It has a characteristic and refreshing odor and a burning taste, fol-

lowed by a cool taste.

*l*-Menthol is very soluble in ethanol (95) and in diethyl ether, and very slightly soluble in water.

*l*-Menthol sublimates gradually at room temperature.

**Identification (1)** Triturate *l*-Menthol with an equal amount of camphor, chloral hydrate or thymol: the mixture liquefies.

(2) Shake 1 g of *l*-Menthol with 20 mL of sulfuric acid: the mixture becomes turbid with a yellow-red color. Allow to stand for 3 hours: a clear, oily layer which possesses no aroma of menthol is separated.

**Optical rotation**  $[\alpha]_D^{20}$ : –45.0 – –51.0° (2.5 g, ethanol (95), 25 mL, 100 mm).

**Melting point** 42 – 44°C

**Purity (1)** Non-volatile residue—Volatilize 2.0 g of *l*-Menthol on a water bath, and dry the residue at 105°C for 2 hours: the residue weighs not more than 1.0 mg.

(2) Thymol—Add 0.20 g of *l*-Menthol to a cold mixture of 2 mL of acetic acid (100), 6 drops of sulfuric acid and 2 drops of nitric acid: no green to blue-green color immediately develops.

(3) Nitromethane or nitroethane—To 0.5 g of *l*-Menthol placed in a flask add 2 mL of sodium hydroxide solution (1 in 2) and 1 mL of strong hydrogen peroxide, connect a reflux condenser to the flask, and boil the mixture gently for 10 minutes. After cooling, add water to make exactly 20 mL, and filter. Take 1 mL of the filtrate in a Nessler tube, add water to make 10 mL, neutralize with dilute hydrochloric acid, add another 1 mL of dilute hydrochloric acid, and cool. To the mixture add 1 mL of a solution of sulfanilic acid (1 in 100), allow to stand for 2 minutes, and then add 1 mL of a solution of *N*-(1-naphthyl)-*N'*-diethylethylenediamine oxalate (1 in 1000) and water to make 25 mL: no red-purple color immediately develops.

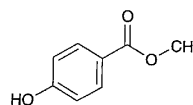
**Assay** Weigh accurately about 2 g of *l*-Menthol, add exactly 20 mL of a mixture of dehydrated pyridine and acetic anhydride (8:1), connect a reflux condenser, and heat on a water bath for 2 hours. Wash the condenser with 20 mL of water, and titrate with 1 mol/L sodium hydroxide VS (indicator: 5 drops of phenolphthalein TS). Perform a blank determination and make any necessary correction.

Each mL of 1 mol/L sodium hydroxide VS  
= 156.27 mg of C<sub>10</sub>H<sub>20</sub>O

**Containers and storage** Containers—Tight containers.  
Storage—In a cold place.

## Methyl Parahydroxybenzoate

パラオキシ安息香酸メチル



C<sub>8</sub>H<sub>8</sub>O<sub>3</sub>: 152.15  
Methyl 4-hydroxybenzoate [98-76-3]