Mentha Oil

Oleum Menthae Japonicae

ハッカ油

Mentha Oil is the essential oil which is distilled with steam from the terrestrial parts of *Mentha arvensis* Linné var. *piperascens* Malinvaud (*Labiatae*), and from which solids are removed after cooling.

It contains not less than 30.0% of menthol ($C_{10}H_{20}O$: 156.27).

Description Mentha Oil is a colorless or pale yellow, clear liquid. It has a characteristic, pleasant aroma and has a pungent taste, followed by a cool aftertaste.

It is miscible with ethanol (95), with ethanol (99.5), with warm ethanol (95), and with diethyl ether.

It is practically insoluble in water.

Refractive index n_D^{20}: 1.455 - 1.467

Optical rotation α_D^{20}: −17.0 - −36.0° (100 mm).

Specific gravity d_{25}^{25}: 0.885 - 0.910

Acid value Not more than 1.0.

Purity **(1)** Clarity and color of solution—To 1.0 mL of Mentha Oil add 3.5 mL of diluted ethanol (7 in 10), and shake: Mentha Oil dissolves clearly. To the solution add 10 mL of ethanol (95): the solution is clear or has no more turbidity, if any, than the following control solution.

Control solution: To 0.70 mL of 0.01 mol/L hydrochloric acid VS add 6 mL of dilute nitric acid and water to make 50 mL, add 1 mL of silver nitrate TS, and allow to stand for 5 minutes.

(2) Heavy metals—Proceed with 1.0 mL of Mentha Oil according to Method 2, and perform the test. Prepare the control solution with 4.0 mL of Standard Lead Solution (not more than 40 ppm).

Assay Weigh accurately about 5.0 g of Mentha Oil, and dissolve in ethanol (95) to make exactly 20 mL. Pipet 10 mL of this solution, add exactly 10 mL of the internal standard solution, and use this solution as the sample solution. Separately, weigh accurately about 10.0 g of *l*-menthol for assay, and dissolve in ethanol (95) to make exactly 100 mL. Pipet 10 mL of this solution, add exactly 10 mL of the internal standard solution, and use this solution as the standard solution. Perform the test with 1 μL each of the sample solution and the standard solution as directed under the Gas Chromatography according to the following conditions. Calculate the ratios, Q_T and Q_S, of the peak area of menthol to that of the internal standard.

$$\text{Amount (mg) of } C_{10}H_{20}O = \text{amount (mg) of } l\text{-menthol for assay} \times \frac{Q_T}{Q_S}$$

Internal standard solution—A solution of *n*-ethyl caprylate in ethanol (95) (4 in 100).

Operating conditions—

Detector: A hydrogen flame-ionization detector.

Column: A glass column about 3 mm in inside diameter and about 2 m in length, packed with 25% of polyethylene glycol 6000 for gas chromatography supported on acid-washed 180 - 250 μm siliceous earth for gas chromatography.

Column temperature: A constant temperature of about 150°C.

Carrier gas: Nitrogen.

Flow rate: Adjust the flow rate so that the retention time of the internal standard is about 10 minutes.

Selection of column: Proceed with 1 μL of the standard solution under the above operating conditions, and calculate the resolution. Use a column giving elution of the internal standard and *l*-menthol in this order with the resolution between these peaks being not less than 5.

Containers and storage Containers—Tight containers.

Storage—Light-resistant.

Mentha Water

ハッカ水

Method of preparation

Mentha Oil	2 mL
Purified Water	a sufficient quantity
	To make 1000 mL

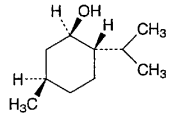
Prepare as directed under Aromatic Waters, with the above ingredients.

Description Mentha Water is a clear, colorless liquid, having the odor of mentha oil.

Containers and storage Containers—Tight containers.

dl-Menthol

dl-メントール



and enantiomer

$C_{10}H_{20}O$: 156.27

(1*RS*,2*SR*,5*RS*)-2-Isopropyl-5-methylcyclohexanol

[*15356-70-4*]

dl-Menthol contains not less than 98.0% of $C_{10}H_{20}O$.

Description *dl*-Menthol occurs as colorless crystals. It has a characteristic and refreshing odor and a burning taste, followed by a cool taste.

It is very soluble in ethanol (95) and in diethyl ether, and very slightly soluble in water.

It sublimes gradually at room temperature.

Identification **(1)** Triturate *dl*-Menthol with an equal

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.

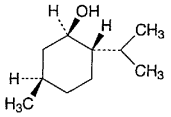
Assay Weigh accurately about 2 g of *dl*-Menthol, add exactly 20 mL of a mixture of dehydrated pyridine and acetic anhydride (8:1), connect a reflux condenser, and heat on a water bath for 2 hours. Wash down the condenser with 20 mL of water, and titrate with 1 mol/L sodium hydroxide VS (indicator: 5 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 $C_{10}H_{20}O$

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

l-Menthol

l-メントール



$C_{10}H_{20}O$: 156.27
(1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexanol
[*2216-51-5*]

l-Menthol contains not less than 98.0% of $C_{10}H_{20}O$.

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

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

l-Menthol sublimes 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.

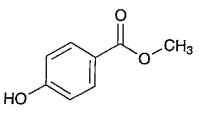
Assay Weigh accurately about 2 g of *l*-Menthol, add exactly 20 mL of a mixture of dehydrated pyridine and acetic anhydride (8:1), connect a reflux condenser, and heat on a water bath for 2 hours. Wash the condenser with 20 mL of water, and titrate with 1 mol/L sodium hydroxide VS (indicator: 5 drops 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 $C_{10}H_{20}O$

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



$C_8H_8O_3$: 152.15
Methyl 4-hydroxybenzoate [*98-76-3*]