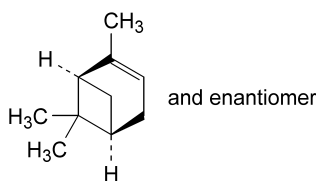


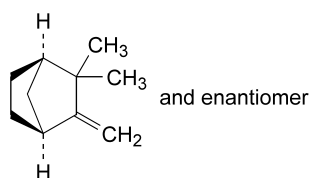
**Water.** Dissolve 1 g in 10 ml of *light petroleum R*. The solution is clear (2.2.1).

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corrected 6.0

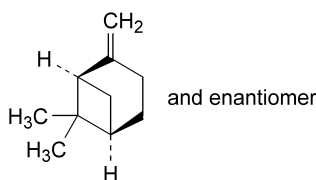
#### IMPURITIES



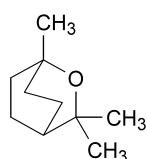
A. 2,6,6-trimethylbicyclo[3.1.1]hept-2-ene ( $\alpha$ -pinene),



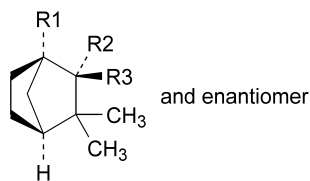
B. 2,2-dimethyl-3-methylenebicyclo[2.2.1]heptane (camphene),



C. 6,6-dimethyl-2-methylenebicyclo[3.1.1]heptane ( $\beta$ -pinene),



D. 3,3-dimethyl-2-oxabicyclo[2.2.2]octane (cineole),

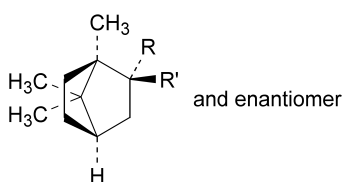


E. R1 = CH<sub>3</sub>, R2 + R3 = O: 1,3,3-trimethylbicyclo[2.2.1]heptan-2-one (fenchone),

F. R1 = CH<sub>3</sub>, R2 = OH, R3 = H: *exo*-1,3,3-trimethylbicyclo[2.2.1]heptan-2-ol (fenchol),

G. R1 = H, R2 = OH, R3 = CH<sub>3</sub>: *exo*-2,3,3-trimethylbicyclo[2.2.1]heptan-2-ol (camphene hydrate),

H. R1 = H, R2 = CH<sub>3</sub>, R3 = OH: *endo*-2,3,3-trimethylbicyclo[2.2.1]heptan-2-ol (methylcamphenilol),

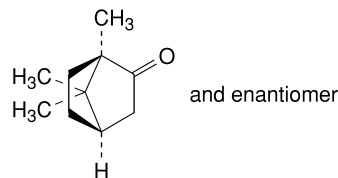


I. R = OH, R' = H: *exo*-1,7,7-trimethylbicyclo[2.2.1]heptan-2-ol (*exo*-borneol),

J. R = H, R' = OH: *endo*-1,7,7-trimethylbicyclo[2.2.1]heptan-2-ol (*endo*-borneol).

## CAMPHOR, RACEMIC

Camphora racemica



C<sub>10</sub>H<sub>16</sub>O  
[76-22-2]

M<sub>r</sub> 152.2

#### DEFINITION

(1*RS*,4*RS*)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-one.

#### CHARACTERS

**Appearance:** white or almost white, crystalline powder or friable, crystalline masses, highly volatile even at room temperature.

**Solubility:** slightly soluble in water, very soluble in ethanol (96 per cent) and in light petroleum, freely soluble in fatty oils, very slightly soluble in glycerol.

#### IDENTIFICATION

**First identification:** A, C.

**Second identification:** A, B, D.

A. Optical rotation (see Tests).

B. Melting point (2.2.14): 172 °C to 180 °C.

C. Infrared absorption spectrophotometry (2.2.24).

**Preparation:** mulls in *liquid paraffin R*.

**Comparison:** *racemic camphor CRS*.

D. Dissolve 1.0 g in 30 ml of *methanol R*. Add 1.0 g of *hydroxylamine hydrochloride R* and 1.0 g of *anhydrous sodium acetate R*. Boil under a reflux condenser for 2 h. Allow to cool and add 100 ml of *water R*. A precipitate is formed. Filter, wash with 10 ml of *water R* and recrystallize from 10 ml of a mixture of 4 volumes of *ethanol (96 per cent) R* and 6 volumes of *water R*. The crystals, dried *in vacuo*, melt (2.2.14) at 118 °C to 121 °C.

#### TESTS

**Carry out the weighings rapidly.**

**Solution S.** Dissolve 2.50 g in 10 ml of *ethanol (96 per cent) R* and dilute to 25.0 ml with the same solvent.

**Appearance of solution.** Solution S is clear (2.2.1) and colourless (2.2.2, *Method II*).

**Acidity or alkalinity.** Dissolve 1.0 g in 10 ml of *ethanol (96 per cent) R* and add 0.1 ml of *phenolphthalein solution RI*. The solution is colourless. Not more than 0.2 ml of 0.1 M *sodium hydroxide* is required to change the colour of the indicator.

**Optical rotation** (2.2.7):  $-0.15^{\circ}$  to  $+0.15^{\circ}$ , determined on solution S.

**Related substances.** Gas chromatography (2.2.28).

**Test solution.** Dissolve 50 mg of the substance to be examined in *hexane R* and dilute to 50.0 ml with the same solvent.

*Reference solution (a).* Dissolve 50 mg of the substance to be examined and 50 mg of *bornyl acetate R* in *hexane R* and dilute to 50.0 ml with the same solvent.

*Reference solution (b).* Dilute 1.0 ml of the test solution to 200.0 ml with *hexane R*.

*Column:*

- *size:*  $l = 2\text{ m}$ ,  $\varnothing = 2\text{ mm}$ ;
- *stationary phase:* *diatomaceous earth for gas chromatography R* impregnated with 10 per cent *m/m* of *macrogol 20 000 R*.

*Carrier gas:* *nitrogen for chromatography R*.

*Flow rate:* 30 ml/min.

*Temperature:*

- *column:* 130 °C;
- *injection port and detector:* 200 °C.

*Detection:* flame ionisation.

*Injection:* 1 µl.

*Run time:* 3 times the retention time of camphor.

*System suitability:*

- *resolution:* minimum 1.5 between the peaks due to camphor and bornyl acetate in the chromatogram obtained with reference solution (a);
- *signal-to-noise ratio:* minimum 5 for the principal peak in the chromatogram obtained with reference solution (b).

*Limits:*

- *any impurity:* for each impurity, not more than 2 per cent of the area of the principal peak;
- *total:* not more than 4 per cent of the area of the principal peak;
- *disregard limit:* the area of the principal peak in the chromatogram obtained with reference solution (b).

**Halogens:** maximum 100 ppm.

Dissolve 1.0 g in 10 ml of *2-propanol R* in a distillation flask. Add 1.5 ml of *dilute sodium hydroxide solution R* and 50 mg of *nickel-aluminium alloy R*. Heat on a water-bath until the *2-propanol R* has evaporated. Allow to cool and add 5 ml of *water R*. Mix and filter through a wet filter previously washed with *water R* until free from chlorides. Dilute the filtrate to 10.0 ml with *water R*. To 5.0 ml of this solution, add *nitric acid R* dropwise until the precipitate which forms is redissolved and dilute to 15 ml with *water R*. The solution complies with the limit test for chlorides (2.4.4).

**Water.** Dissolve 1 g in 10 ml of *light petroleum R*. The solution is clear (2.2.1).

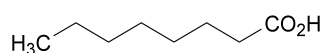
**Residue on evaporation:** maximum 0.05 per cent.

Evaporate 2.0 g on a water-bath and dry at 100-105 °C for 1 h. The residue weighs not more than 1 mg.

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## CAPRYLIC ACID

Acidum caprylicum



$C_8H_{16}O_2$   
[124-07-2]

$M_r$  144.2

### DEFINITION

Octanoic acid.

*Content:* 99.0 per cent to 100.5 per cent (anhydrous substance).

### CHARACTERS

*Appearance:* clear, colourless or slightly yellowish, oily liquid.

*Solubility:* very slightly soluble in water, very soluble in acetone and in ethanol (96 per cent). It dissolves in dilute solutions of alkali hydroxides.

### IDENTIFICATION

A. Relative density (see Tests).

B. Examine the chromatograms obtained in the test for related substances.

*Results:* the principal peak in the chromatogram obtained with the test solution is similar in retention time and size to the principal peak in the chromatogram obtained with reference solution (a).

### TESTS

**Appearance.** The substance to be examined is clear (2.2.1) and not more intensely coloured than reference solution  $Y_5$  (2.2.2, Method II).

**Relative density (2.2.5):** 0.909 to 0.912.

**Related substances.** Gas chromatography (2.2.28): use the normalisation procedure.

*Test solution.* Dissolve 0.10 g of the substance to be examined in *ethyl acetate R* and dilute to 10.0 ml with the same solvent.

*Reference solution (a).* Dissolve 0.10 g of *caprylic acid CRS* in *ethyl acetate R* and dilute to 10.0 ml with the same solvent.

*Reference solution (b).* Dilute 1.0 ml of the test solution to 100.0 ml with *ethyl acetate R*. Dilute 5.0 ml of this solution to 50.0 ml with *ethyl acetate R*.

*Column:*

- *material:* fused silica;
- *size:*  $l = 30\text{ m}$ ,  $\varnothing = 0.25\text{ mm}$ ;
- *stationary phase:* *macrogol 20 000 2-nitroterephthalate R* (film thickness 0.25 µm).

*Carrier gas:* *helium for chromatography R*.

*Flow rate:* 1.5 ml/min.

*Split ratio:* 1:100.

*Temperature:*

	Time (min)	Temperature (°C)
Column	0 - 1	100
	1 - 25	100 → 220
	25 - 35	220
Injection port		250
Detector		250

*Detection:* flame ionisation.

*Injection:* 1 µl.

*System suitability:* reference solution (b):

- *signal-to-noise ratio:* minimum 5 for the principal peak.

*Limits:*

- *any impurity:* for each impurity, maximum 0.3 per cent;
- *total:* maximum 0.5 per cent;
- *disregard limit:* 0.5 times the area of the principal peak in the chromatogram obtained with reference solution (b) (0.05 per cent).

**Heavy metals (2.4.8):** maximum 10 ppm.

Dissolve 2.0 g in *ethanol (96 per cent) R* and dilute to 20 ml with the same solvent. 12 ml of the solution complies with test B. Prepare the reference solution using 1 ml of