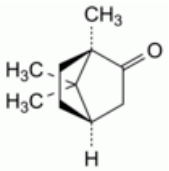
Edition: BP 2025 (Ph. Eur. 11.6 update)

Natural Camphor

General Notices

(D-Camphor, Ph. Eur. monograph 1400)



$C_{10}H_{16}O$ 152.2 *464-49-3*

Ph Eur

DEFINITION

(1*R*,4*R*)-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. Specific optical rotation (see Tests).
B. Melting point (2.2.14): 175 °C to 179 °C.
C. Infrared absorption spectrophotometry (2.2.24).

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*. Filter, wash the precipitate obtained with 10 mL of *water R* and recrystallise from 10 mL of a mixture of 4 volumes of *alcohol 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 and dissolution rapidly.

Solution S

Dissolve 2.50 g in 10 mL of *alcohol 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

To 10 mL of solution S add 0.1 mL of *phenolphthalein solution R1*. 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.

Specific optical rotation (*2.2.7*)

+ 41.0 to + 44.0, determined on solution S.

Related substances

Gas chromatography (*2.2.28*).

Test solution Dissolve 2.50 g of the substance to be examined in *heptane R* and dilute to 25.0 mL with the same solvent.

Reference solution (a) Dilute 1.0 mL of the test solution to 100.0 mL with *heptane R*.

Reference solution (b) Dilute 10.0 mL of reference solution (a) to 20.0 mL with *heptane R*.

Reference solution (c) Dissolve 0.50 g of *borneol R* in *heptane R* and dilute to 25.0 mL with the same solvent. Dilute 5.0 mL of the solution to 50.0 mL with *heptane R*.

Reference solution (d) Dissolve 50 mg of *linalol R* and 50 mg of *bornyl acetate R* in *heptane R* and dilute to 100.0 mL with the same solvent.

Column:

— *size*: *l* = 30 m, Ø = 0.25 mm,

— *stationary phase*: *macrogol 20 000 R* (0.25 µm).

Carrier gas *helium for chromatography R*.

Split ratio 1:70.

Flow rate 45 cm/s.

Temperature:

	Time (min)	Temperature (°C)
Column	0 - 10	50
	10 - 35	50 → 100
	35 - 45	100 → 200

	Time (min)	Temperature (°C)
	45 - 55	200
Injection port		220
Detector		250

Detection Flame ionisation.

Injection 1 µL.

System suitability Reference solution (d).

— *resolution*: minimum 3.0 between the peaks due to bornyl acetate and to linalol.

Limits:

— *borneol*: not more than the area of the principal peak in the chromatogram obtained with reference solution (c) (2.0 per cent),

— *any other impurity*: not more than half of the area of the principal peak in the chromatogram obtained with reference solution (a) (0.5 per cent),

— *total of other impurities*: not more than 4 times the area of the principal peak in the chromatogram obtained with reference solution (a) (4.0 per cent),

— *disregard limit*: 0.1 times the area of the principal peak in the chromatogram obtained with reference solution (b) (0.05 per cent).

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 the 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*).

Residue on evaporation (*2.8.9*)

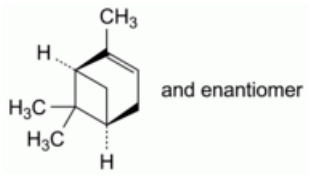
Maximum 0.05 per cent.

Evaporate 2.0 g on a water-bath and dry in an oven at 100-105 °C for 1 h. The residue weighs a maximum of 1 mg.

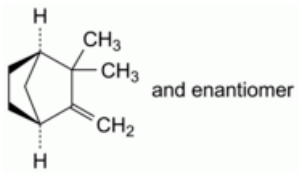
Water

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

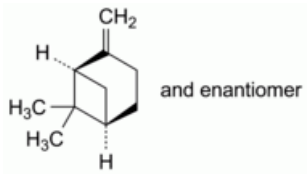
IMPURITIES



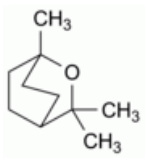
A. 2,6,6-trimethylbicyclo[3.1.1]hept-2-ene (α-pinene),



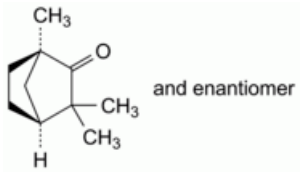
B. 2,2-dimethyl-3-methylenebicyclo[2.2.1]heptane (camphene),



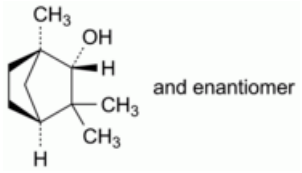
C. 6,6-dimethyl-2-methylenebicyclo[3.1.1]heptane (β-pinene),



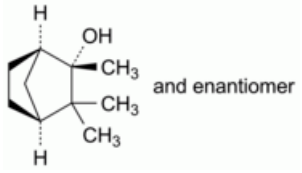
D. 1,3,3-trimethyl-2-oxabicyclo[2.2.2]octane (cineole),



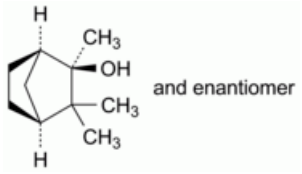
E. 1,3,3-trimethylbicyclo[2.2.1]heptan-2-one (fenchone),



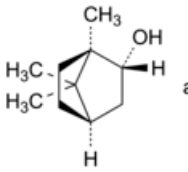
F. *exo*-1,3,3-trimethylbicyclo[2.2.1]heptan-2-ol (fenchol),



G. *exo*-2,3,3-trimethylbicyclo[2.2.1]heptan-2-ol (camphene hydrate),

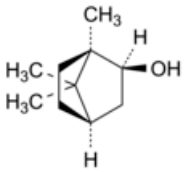


H. *endo*-2,3,3-trimethylbicyclo[2.2.1]heptan-2-ol (methylcamphenilol),



and enantiomer

I. *exo*-1,7,7-trimethylbicyclo[2.2.1]heptan-2-ol (*exo*-borneol),



and enantiomer

J. *endo*-1,7,7-trimethylbicyclo[2.2.1]heptan-2-ol (*endo*-borneol).

Ph Eur