

Calculate the percentage content of flavonoids, expressed as hyperoside, using the following expression:

$$\frac{A \times 1.25}{m}$$

i.e. taking the specific absorbance of hyperoside to be 500.

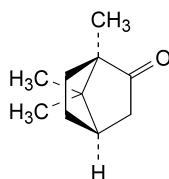
A = absorbance at 425 nm;

m = mass of the drug to be examined, in grams.

01/2008:1400
corrected 6.0

D-CAMPHOR

D-Camphora



$C_{10}H_{16}O$
[464-49-3]

M_r 152.2

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 alcohol 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): + 40.0 to + 43.0, 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, $\emptyset = 0.25$ mm,

– stationary phase: *macrogol 20 000 R* (0.25 μ m).

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
	45 - 55	200
Injection port		220
Detector		250

Detection: flame ionisation.

Injection: 1 μ l.

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).

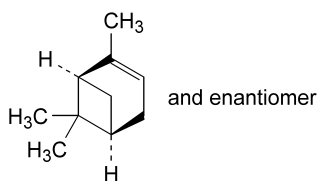
Residue on evaporation (2.8.9): maximum 0.05 per cent.

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.

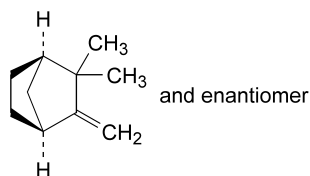
Water. Dissolve 1 g in 10 ml of *light petroleum R*. The solution is clear (2.2.1).

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corrected 6.0

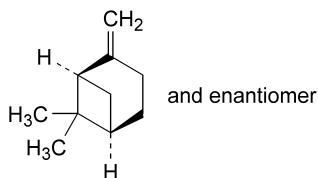
IMPURITIES



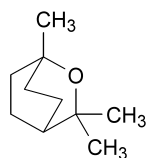
A. 2,6,6-trimethylbicyclo[3.1.1]hept-2-ene (α -pinene),



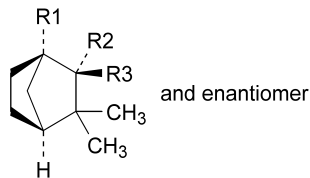
B. 2,2-dimethyl-3-methylenebicyclo[2.2.1]heptane (camphene),



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



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

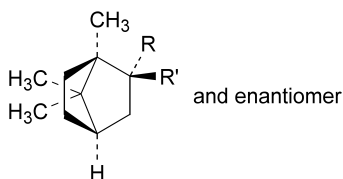


E. R1 = CH₃, R2 + R3 = O: 1,3,3-trimethylbicyclo[2.2.1]heptan-2-one (fenchone),

F. R1 = CH₃, R2 = OH, R3 = H: *exo*-1,3,3-trimethylbicyclo[2.2.1]heptan-2-ol (fenchol),

G. R1 = H, R2 = OH, R3 = CH₃: *exo*-2,3,3-trimethylbicyclo[2.2.1]heptan-2-ol (camphene hydrate),

H. R1 = H, R2 = CH₃, R3 = OH: *endo*-2,3,3-trimethylbicyclo[2.2.1]heptan-2-ol (methylcamphenilol),

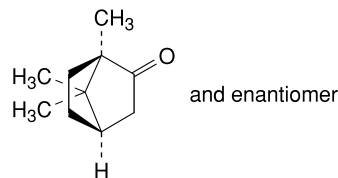


I. R = OH, R' = H: *exo*-1,7,7-trimethylbicyclo[2.2.1]heptan-2-ol (*exo*-borneol),

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

CAMPHOR, RACEMIC

Camphora racemica



C₁₀H₁₆O
[76-22-2]

M_r 152.2

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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° 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.