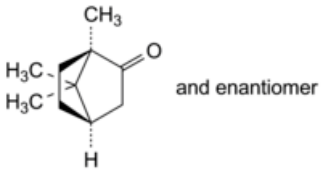
Edition: BP 2025 (Ph. Eur. 11.6 update)

Racemic Camphor

General Notices

(Ph. Eur. monograph 0655)



$C_{10}H_{16}O$ 152.2 *76-22-2*

Action and use

Counter-irritant.

Preparations

Camphorated Opium Tincture

Concentrated Camphorated Opium Tincture

Concentrated Camphor Water

Ph Eur

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 recrystallise 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 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.

Optical rotation (2.2.7)

-0.15° to + 0.15°, 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 m, Ø = 2 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.