Reference solution. Mix 80 mg of *cineole CRS* with *alcohol R* and dilute to 10 ml with the same solvent.

Plate: *TLC silica gel plate R*.

Mobile phase: *ethyl acetate R, toluene R* (10:90 *V/V*).

Application: 2 µl.

Development: over 2/3 of the plate.

Drying: in a current of cold air.

Detection: spray with *anisaldehyde solution R*, heat at 100-105 °C for 5 min.

Results: the principal spot in the chromatogram obtained with the test solution is similar in position, colour and size to the principal spot in the chromatogram obtained with the reference solution.

C. To 0.1 ml add 4 ml of *sulphuric acid R*. An orange-red colour develops. Add 0.2 ml of *formaldehyde solution R*. The colour changes to deep brown.

TESTS

Solution S. Dilute 2.00 g to 10.0 ml with *alcohol R*.

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

Chiral impurities. The optical rotation (*2.2.7*) of solution S is – 0.10° to + 0.10°.

Refractive index (*2.2.6*): 1.456 to 1.460.

Related substances. Gas chromatography (*2.2.28*).

Internal standard solution. Dissolve 1.0 g of *camphor R* in *heptane R* and dilute to 200 ml with the same solvent.

Test solution (a). Dissolve 2.5 g of the substance to be examined in *heptane R* and dilute to 25.0 ml with the same solvent.

Test solution (b). Dissolve 2.5 g of the substance to be examined in *heptane R*, add 5.0 ml of the internal standard solution and dilute to 25.0 ml with *heptane R*.

Reference solution (a). To 2.0 ml of test solution (a) add 20.0 ml of the internal standard solution and dilute to 100.0 ml with *heptane R*.

Reference solution (b). Dissolve 50 mg of *1,4-cineole R* and 50 mg of the substance to be examined in *heptane R* and dilute to 50.0 ml with the same solvent.

Column:

– *size*: *l* = 30 m, Ø = 0.25 mm,
– *stationary phase*: *macrogol 20 000 R* (film thickness 0.25 µm).

Carrier gas: *helium for chromatography R*.

Linear velocity: 45 cm/s.

Split-ratio: 1:70.

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 µl.

System suitability: reference solution (b):

– *resolution*: minimum 10 between the peaks due to impurity A and to cineole.

Limits:

– *total*: calculate the ratio (*R*) of the area of the peak due to cineole to the area of the peak due to the internal standard from the chromatogram obtained with reference solution (a); from the chromatogram obtained with test solution (b), calculate the ratio of the sum of the areas of any peaks, apart from the principal peak and the peak due to the internal standard, to the area of the peak due to internal standard: this ratio is not greater than *R* (2 per cent),
– *disregard limit*: 0.025 times the area of the principal peak in the chromatogram obtained with reference solution (a) (0.05 per cent).

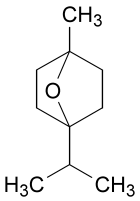
Residue on evaporation: maximum 0.1 per cent.

To 2.0 g add 5 ml of *water R*, evaporate to dryness on a water-bath and dry at 100-105 °C for 1 h. The residue weighs a maximum of 2 mg.

STORAGE

In an airtight container, protected from light.

IMPURITIES



A. 1-methyl-4-(1-methylethyl)-7-oxabicyclo[2.2.1]heptane (1,4-cineole).

01/2008:0387

CINNAMON

Cinnamomi cortex

DEFINITION

Dried bark, freed from the outer cork and the underlying parenchyma, and shoots grown on cut stock of *Cinnamomum zeylanicum* Nees.

Content: minimum 12 ml/kg of essential oil.

CHARACTERS

Characteristic, aromatic odour.

IDENTIFICATION

A. The bark is about 0.2-0.8 mm thick and occurs in closely-packed compound quills made up of single or double quills. The outer surface is smooth, yellowish-brown with faint scars marking the position of leaves and axillary buds and has fine, whitish and wavy longitudinal striations. The inner surface is slightly darker and longitudinally striated. The fracture is short and fibrous.

B. Reduce to a powder (355) (*2.9.12*). The powder is yellowish or reddish-brown. Examine under a microscope using *chloral hydrate solution R*. The powder shows the following diagnostic characters: groups of rounded sclereids with pitted, channelled and moderately thickened walls; numerous colourless single fibres, often whole with narrow lumen and thickened, lignified walls and few pits; small acicular crystals of calcium oxalate. Cork fragments are absent or very rare. Examine under a microscope using a 50 per cent *V/V* solution of *glycerol R*; the powder shows abundant starch granules.

C. Thin-layer chromatography (*2.2.27*).

Test solution. Shake 0.1 g of powdered drug (500) (*2.9.12*) with 2 ml of *methylene chloride R* for 15 min. Filter and evaporate the filtrate carefully almost to dryness on a water-bath. Dissolve the residue in 0.4 ml of *toluene R*.

Reference solution. Dissolve 50 µl of *cinnamic aldehyde R* and 10 µl of *eugenol R* in *toluene R*, then dilute to 10 ml with the same solvent.

Plate: *TLC silica gel GF_{254} plate R*.

Mobile phase: *methylene chloride R*.

Application: 10 µl as bands of 20 mm by 3 mm.

Development: over a path of 10 cm.

Drying: in air.

Detection A: examine in ultraviolet light at 254 nm and mark the quenching zones, then, examine in ultraviolet light at 365 nm and mark the fluorescent zones.

Results A: in ultraviolet light at 254 nm, the chromatograms obtained with the test solution and the reference solution show a quenching zone due to cinnamaldehyde in the median part and just above it a weaker quenching zone due to eugenol. In ultraviolet light at 365 nm, the chromatogram obtained with the test solution shows a zone of light blue fluorescence due to *o*-methoxy-cinnamaldehyde just below the zone due to cinnamaldehyde.

Detection B: spray with *phloroglucinol solution R*.

Results B: the zone due to cinnamaldehyde is yellowish-brown and the zone due to *o*-methoxycinn-amaldehyde is violet.

TESTS

Total ash (*2.4.16*): maximum 6.0 per cent.

ASSAY

Carry out the determination of essential oils in herbal drugs (*2.8.12*). Use a 500 ml flask, 200 ml of *0.1 M hydrochloric acid* as the distillation liquid and 0.50 ml of *xylene R* in the graduated tube. Powder the drug (710) (*2.9.12*) and proceed immediately with the determination, using 20.0 g. Distil at a rate of 2.5-3.5 ml/min for 3 h.

01/2008:1501

CINNAMON BARK OIL, CEYLON

Cinnamomi zeylanicii corticis aetheroleum

DEFINITION

Ceylon cinnamon bark oil is obtained by steam distillation of the bark of the shoots of *Cinnamomum zeylanicum* Nees (*C. Verum* J.S. Presl.).

CHARACTERS

A clear, mobile, light yellow liquid becoming reddish over time, with a characteristic odour reminiscent of cinnamic aldehyde.

IDENTIFICATION

First identification: B.

Second identification: A.

A. Examine by thin-layer chromatography (*2.2.27*), using a *TLC silica gel plate R*.

Test solution. Dissolve 1 ml of the substance to be examined in *acetone R* and dilute to 10 ml with the same solvent.

Reference solution. Dissolve 50 µl of *trans-cinnamic aldehyde R*, 10 µl of *eugenol R*, 10 µl of *linalol R* and 10 µl of *β-caryophyllene R* in *alcohol R* and dilute to 10 ml with the same solvent.

Apply to the plate, as bands, 10 µl of each solution. Develop over a path of 15 cm using a mixture of 10 volumes of *methanol R* and 90 volumes of *toluene R*. Allow the plate to dry in air and spray with *anisaldehyde solution R*. Examine in daylight while heating at 100-105 °C for 5-10 min. The chromatogram obtained with the test solution shows zones similar in position and colour to those in the chromatogram obtained with the reference solution.

B. Examine the chromatograms obtained in the test for chromatographic profile. The retention times of the principal peaks in the chromatogram obtained with the test solution are similar to those in the chromatogram obtained with the reference solution. Safrole, coumarin and cineole may be absent from the chromatogram obtained with the test solution.

TESTS

Relative density (*2.2.5*): 1.000 to 1.030.

Refractive index (*2.2.6*): 1.572 to 1.591.

Optical rotation (*2.2.7*): – 2° to + 1°.

Chromatographic profile. Examine by gas chromatography (*2.2.28*).

Test solution. The substance to be examined.

Reference solution. Dissolve 10 µl of *cineole R*, 10 µl of *linalol R*, 10 µl of *β-caryophyllene R*, 10 µl of *safrole R*, 100 µl of *trans-cinnamic aldehyde R*, 10 µl of *eugenol R*, 20 mg of *coumarin R*, 10 µl of *trans-2-methoxycinnamaldehyde R* and 10 µl of *benzyl benzoate R* in 1 ml of *acetone R*.

The chromatographic procedure may be carried out using:

– a fused-silica column 60 m long and about 0.25 mm in internal diameter coated with *macrogol 20 000 R* as the bonded phase,
– *helium for chromatography R* as the carrier gas at a flow rate of 1.5 ml/min,
– a flame-ionisation detector,
– a split ratio of 1:100,

with the following temperature programme:

	Time (min)	Temperature (°C)	Rate (°C/min)	Comment
Column	0 - 10	60	–	isothermal
	10 - 75	60 → 190	2	linear gradient
	75 - 200	190		isothermal
Injection port		200		
Detector		240		

Inject 0.2 µl of the reference solution. When the chromatogram is recorded in the prescribed conditions, the components elute in the order indicated in the composition of the reference solution. Depending on the operating conditions and the state of the column, coumarin may elute before or after *trans*-2-methoxycinnamaldehyde. Record the retention times of these substances.

The test is not valid unless the resolution between the peaks corresponding to linalol and β-caryophyllene is at least 1.5.