

**CASCARA
FOR HOMOEOPATHIC PREPARATIONS**

**CASCARA SAGRADA
FOR HOMOEOPATHIC PREPARATIONS**

Rhamni purshianae cortex ad praeparationes homoeopathicas

DEFINITION

The herbal drug complies with the requirements of *Cascara (0105)*.

STOCK

DEFINITION

Cascara mother tincture complies with the requirements of the general technique for the preparation of mother tinctures (see *Homoeopathic Preparations (1038)* and French Pharmacopoeia Authority Supplement). The mother tincture is prepared with *ethanol (65 per cent V/V)*, using the dried whole bark or fragmented bark of *Rhamnus purshianus* D.C. (*Frangula purshiana (D.C.)* A. Gray ex J.C. Cooper).

Contents: minimum 0.20 per cent *m/m* hydroxy-anthracene heterosides, with minimum 60 per cent *m/m* cascarosides, both groups being expressed as cascaroside A ($C_{27}H_{32}O_{14}$; M_r 580.5).

CHARACTERS

Appearance: dark orange-brown liquid.

IDENTIFICATION

Thin-layer chromatography (2.2.27).

Test solution. Mother tincture.

Reference solution (a). Dissolve 10 mg of *barbaloin R* in 20 mL of *methanol R*.

Reference solution (b). Dissolve 1 mg of *emodin R* in 6 mL of a mixture of 2 volumes of *methanol R* and 1 volume of *methylene chloride R*.

Plate: TLC silica gel plate *R*.

Mobile phase: *water R*, *methanol R*, *ethyl acetate R* (13:17:100 V/V/V).

Application: 20 mL, as bands.

Development: over a path of 10 cm.

Drying: in air.

Detection A: examine in ultraviolet light at 365 nm.

The General Chapters and General Monographs of the European Pharmacopoeia and Preamble of the French Pharmacopoeia apply.

Results A: see below the sequence of fluorescent zones present in the chromatograms of the reference solution and the test solution. Furthermore other fluorescent zones may be present in the chromatogram obtained with the test solution.

| Top of the plate | |
|----------------------------------|--|
| Emodin: an orange zone | An orange zone An orange-brown zone A green-blue zone An orange zone An orange zone A blue zone |
| Barbaloin: a central orange zone | A central orange zone (barbaloin) A light green zone A blue zone An orange zone |
| Reference solution | Test solution |

Detection B: spray the plate with a 50 g/L solution of *potassium hydroxide R* in *ethanol (96 per cent) R*. Examine in ultraviolet light at 365 nm.

Results B: see below the sequence of fluorescent zones present in the chromatograms of the reference solution and the test solution. Furthermore other fluorescent zones may be present in the chromatogram obtained with the test solution.

| Top of the plate | |
|----------------------------------|--|
| Emodin: an orange-brown zone | An orange-brown zone A dark purple zone A blue zone A blue zone An orange-yellow zone A yellow-green zone |
| Barbaloin: an orange-yellow zone | An orange-yellow zone (barbaloin) A yellow zone |
| Reference solution | Test solution |

TESTS

Ethanol (2.9.10): 60 per cent V/V to 70 per cent V/V.

Dry residue (2.8.16): minimum 1.8 per cent *m/m*.

ASSAY

Visible absorption spectrophotometry (2.2.25).

Carry out the assay away from bright light and within a day.

The General Chapters and General Monographs of the European Pharmacopoeia and Preamble of the French Pharmacopoeia apply.

Test solution. Place 10.00 g of mother tincture in a 250 mL round-bottomed flask. Eliminate the ethanol on a water-bath. Transfer to a 100.0 mL volumetric flask and dilute to 100.0 mL with *water R*. To 10.0 mL of this solution, add 0.1 mL of 1 M *hydrochloric acid* then extract with 2 quantities, each of 20 mL, of a mixture of 1 volume of *ether R* and 3 volumes of *hexane R*. Combine the organic solutions and wash with 5 mL of *water R*. Discard the organic phase and add the washings to the aqueous phase. Combine the aqueous phases and extract with 4 quantities, each of 30 mL, of *ethyl acetate R*, extemporaneously saturated with *water R* (*water R*, *ethyl acetate R* (15:150 V/V), shake for 3 min then, allow to stand). Each time, allow to stand until the organic phase is clear. Combine the organic phases. Use the aqueous phase for the assay for cascarosides and the organic phase for the assay for hydroxy-anthracene heterosides other than cascarosides.

Hydroxy-anthracene heterosides other than cascarosides. Place the organic phase in a flask; eliminate the solvent by distillation, then evaporate almost to dryness. Dissolve the residue in 0.3-0.5 mL of *methanol R*. Transfer to a 50.0 mL volumetric flask. Wash the first flask with hot *water R*; add the washings to the methanolic solution. Allow to cool and dilute to 50.0 mL with *water R*. Place 20.0 mL of the solution in a 100 mL round-bottomed flask with a ground glass neck, containing 2 g of *ferric chloride R* and 12 mL of *hydrochloric acid R*. Fit a reflux condenser to the flask and place it in a water-bath so that the water level is above that of the liquid in the flask. Heat for 4 h. Allow to cool. Place the solution in a separating funnel. Wash the flask first with 3-4 mL of 1 M *sodium hydroxide*, then with 3-4 mL of *water R*. Add the washings to the content of the separating funnel. Extract the content of the separating funnel with 3 quantities, each of 30 mL, of a mixture of 1 volume of *ether R* and 3 volumes of *hexane R*.

Combine the organic solutions. Wash with 2 quantities, each of 10 mL, of *water R* and discard the washings. Transfer the organic phase to a 100.0 mL volumetric flask and dilute to 100.0 mL with the mixture of *ether R* and *hexane R*. Carefully evaporate 20.0 mL to dryness on a water-bath. Dissolve the residue in 10.0 mL of a 5 g/L solution of *magnesium acetate R* in *methanol R*.

Compensation liquid. Methanol R.

Measure the absorbance of the solution at 515 nm, in comparison with the compensation liquid.

Calculate the percentage content *m/m* of hydroxy-anthracene heterosides, expressed as cascaroside A, from the expression:

$$\frac{A \times 6.95}{m}$$

i.e. taking the specific absorbance of cascaroside A at 515 nm to be 180.

A = absorbance of the test solution at 515 nm,

m = mass of the mother tincture sample, in grams.

Measure the absorbance of the test solution at 440 nm. If the ratio of the absorbance measured at 515 nm and 440 nm is less than 2.4, ignore the results and start again.

Cascarosides. Use the aqueous phase set aside for this assay, and dilute to 50.0 mL with *water R*. Proceed with 20.0 mL of the solution as described in the assay for hydroxy-anthracene heterosides other than cascarosides.

Calculate the percentage content *m/m* of cascarosides, expressed as cascaroside A, from the expression:

The General Chapters and General Monographs of the European Pharmacopoeia and Preamble of the French Pharmacopoeia apply.

$$\frac{A \times 6.95}{m}$$

i.e. taking the specific absorbance to be 180.

A = absorbance of the test solution at 515 nm,

m = mass of the mother tincture sample, in grams.

Measure the absorbance of the test solution at 440 nm. If the ratio of the absorbance measured at 515 nm and 440 nm is less than 2.7, ignore the results and start again.