01/2008:1522

GINGER

Zingiberis rhizoma

DEFINITION

Dried, whole or cut rhizome of *Zingiber officinale* Roscoe, with the cork removed, either completely or from the wide flat surfaces only.

Content: minimum 15 ml/kg of essential oil (anhydrous drug).

CHARACTERS

Characteristic aromatic odour.

Spicy and burning taste.

IDENTIFICATION

A. The rhizome is laterally compressed, bearing short, flattened, obovate oblique branches on the upper side, each sometimes having a depressed scar at the apex; the whole rhizomes are about 5-10 cm long, 1.5-3 cm or 4 cm wide and 1-1.5 cm thick, sometimes split longitudinally. The scraped rhizome with a light-brown external surface shows longitudinal striations and occasional loose fibres; the outer surface of the unscraped rhizome varies from pale to dark brown and is more or less covered with cork which shows conspicuous, narrow, longitudinal and transverse ridges; the cork readily exfoliates from the lateral surfaces but persists between the branches. The fracture is short and starchy with projecting fibres. The smoothed transversely cut surface exhibits a narrow cortex separated by an endodermis from a much wider stele; it shows numerous, scattered, fibrovascular bundles and abundant scattered oleoresin cells with yellow contents. The unscraped rhizome shows, in addition, an outer layer of dark brown cork.

B. Reduce to a powder (355) (*2.9.12*). The powder is pale yellow or brownish. Examine under a microscope using *chloral hydrate solution R*. The powder shows the following diagnostic characters: groups of large, thin-walled, septate fibres, with one wall frequently dentate; fairly large vessels with reticulate thickening and often accompanied by narrow, thin-walled cells containing brown pigment; abundant thin-walled parenchyma of the ground tissue, some cells containing brown oleoresin; fragments of brown cork, usually seen in surface view. Examine under a microscope using a 50 per cent *V/V* solution of *glycerol R*. The powder shows abundant starch granules, simple, flattened, oblong or oval or irregular, up to about 50 µm long and 25 µm wide, with a small point hilum situated at the narrower end; occasional granules show faint, transverse striations.

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

Test solution. To 1.0 g of the powdered drug (710) (*2.9.12*) add 5 ml of *methanol R*. Shake for 15 min and filter.

Reference solution. Dissolve 10 µl of *citral R* and 10 mg of *resorcinol R* in 10 ml of *methanol R*. Prepare the solution immediately before use.

Plate: *TLC silica gel plate R*.

Mobile phase: *hexane R, ether R* (40:60 *V/V*).

Application: 20 µl, as bands.

Development: in an unsaturated tank, over a path of 15 cm.

Drying: in air.

Detection: spray with a 10 g/l solution of *vanillin R* in *sulphuric acid R* and examine in daylight while heating at 100-105 °C for 10 min.

Results: the chromatogram obtained with the reference solution shows in the lower half an intense red zone (resorcinol) and in the upper half 2 violet zones (citral). The chromatogram obtained with the test solution shows below the zone due to resorcinol in the chromatogram obtained with the reference solution 2 intense violet zones (gingerols) and in the middle, between the zones due to resorcinol and citral in the chromatogram obtained with the reference solution, 2 other less intense violet zones (shogaols). Other zones may be present.

TESTS

Water (*2.2.13*): maximum 100 ml/kg, determined by distillation on 20.0 g of the powdered drug (710) (*2.9.12*).

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 20.0 g of the freshly, coarsely powdered drug, a 1000 ml round-bottomed flask, 10 drops of *liquid paraffin R* or other antifoam, 500 ml of *water R* as distillation liquid and 0.5 ml of *xylene R* in the graduated tube. Distil at a rate of 2-3 ml/min for 4 h.

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corrected 6.0

GINKGO LEAF

Ginkgonis folium

DEFINITION

Whole or fragmented, dried leaf of *Ginkgo biloba* L.

Content: not less than 0.5 per cent of flavonoids, calculated as flavone glycosides (M_r 757) (dried drug).

CHARACTERS

Greyish or yellowish-green or yellowish-brown.

IDENTIFICATION

A. The upper surface of ginkgo leaf is slightly darker than the lower surface. The petioles of the leaf are about 4 cm to 9 cm long. The lamina is about 4 cm to 10 cm wide, fan-shaped, usually bilobate or sometimes undivided. Both surfaces are smooth, and the venation dichotomous, the veins appearing to radiate from the base; they are equally prominent on both surfaces. The distal margin is incised, irregularly and to different degrees, and irregularly lobate or emarginate. The lateral margins are entire and taper towards the base.

B. Reduce to a powder (355) (*2.9.12*). The powder is greyish or yellowish-green or yellowish-brown. Examine under a microscope using *chloral hydrate solution R*. The powder shows irregularly-shaped fragments of the lamina in surface view, the upper epidermis consisting of elongated cells with irregularly sinuous walls, the lower epidermal cells smaller, with a finely striated cuticle and each cell shortly papillose; stomata about 60 µm, large, deeply sunken with 6 to 8 subsidiary cells, are more numerous in the lower epidermis; abundant large cluster crystals of calcium oxalate of various sizes in the mesophyll; fragments of fibro-vascular tissue from the petiole and veins.

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

Test solution. To 2.0 g of the powdered drug (710) (*2.9.12*) add 10 ml of *methanol R*. Heat in a water-bath at 65 °C for 10 min. Shake frequently. Allow to cool to room temperature and filter.

Reference solution. Dissolve 1.0 mg of *chlorogenic acid R* and 3.0 mg of *rutin R* in 20 ml of *methanol R*.

Plate: *TLC silica gel plate R*.

Mobile phase: *anhydrous formic acid R, glacial acetic acid R, water R, ethyl acetate R* (7.5:7.5:17.5:67.5 *V/V/V/V*).

Application: 20 µl, as bands.

Development: over a path of 17 cm.

Drying: at 100-105 °C.

Detection: spray the warm plate with a 10 g/l solution of *diphenylboric acid aminoethyl ester R* in *methanol R*. Subsequently spray with the same volume of a 50 g/l solution of *macrogol 400 R* in *methanol R*. Allow the plate to dry in air for about 30 min. Examine in ultraviolet light at 365 nm.

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

Top of the plate	
	A yellowish-brown fluorescent zone
	A green fluorescent zone
	2 yellowish-brown fluorescent zones
	An intense light blue fluorescent zone sometimes overlapped by a greenish-brown fluorescent zone
Chlorogenic acid: a light blue fluorescent zone	
	A green fluorescent zone
Rutin: a yellowish-brown fluorescent zone	2 yellowish-brown fluorescent zones
	A green fluorescent zone
	A yellowish-brown fluorescent zone
Reference solution	**Test solution**

TESTS

Foreign matter (*2.8.2*): maximum 5 per cent of stems and 2 per cent of other foreign matter.

Loss on drying (*2.2.32*): maximum 11.0 per cent, determined on 1.000 g of the powdered drug (355) (*2.9.12*) by drying in an oven at 105 °C for 2 h.

Total ash (*2.4.16*): maximum 11.0 per cent.

ASSAY

Flavonoids. Liquid chromatography (*2.2.29*).

Test solution. Heat 2.500 g of the powdered drug (710) (*2.9.12*) in 50 ml of a 60 per cent *V/V* solution of *acetone R* under a reflux condenser for 30 min. Filter and collect the filtrate. Extract the drug residue a second time in the same manner, using 40 ml of a 60 per cent *V/V* solution of *acetone R* and filter. Collect the filtrates and dilute to 100.0 ml with a 60 per cent *V/V* solution of *acetone R*. Evaporate 50.0 ml of the solution to eliminate the acetone and transfer to a 50.0 ml vial, rinsing with 30 ml of *methanol R*. Add 4.4 ml of *hydrochloric acid R1*, dilute to 50.0 ml with *water R* and centrifuge. Place 10 ml of the supernatant liquid in a 10 ml brown-glass vial. Close with a rubber seal and an aluminium cap and heat on a water-bath for 25 min. Allow to cool to room temperature.

Reference solution. Dissolve 10.0 mg of *quercetin dihydrate R* in 20 ml of *methanol R*. Add 15.0 ml of *dilute hydrochloric acid R* and 5 ml of *water R* and dilute to 50.0 ml with *methanol R*.

Column:

– *stationary phase*: *octadecylsilyl silica gel for chromatography R* (5 µm),

– *size*: l = 0.125 m, Ø = 4 mm,

– *temperature*: 25 °C.

Mobile phase:

– *mobile phase A*: 0.3 g/l solution of *phosphoric acid R* adjusted to pH 2.0,

– *mobile phase B*: *methanol R*,

Time (min)	Mobile phase A (per cent *V/V*)	Mobile phase B (per cent *V/V*)
0 - 1	60	40
1 - 20	60 → 45	40 → 55
20 - 21	45 → 0	55 → 100
21 - 25	0	100

Flow rate: 1.0 ml/min.

Detector: spectrophotometer at 370 nm.

Injection: 10 µl.

Relative retention with reference to quercetin (retention time = about 12.5 min): kaempferol = about 1.4; isorhamnetin= about 1.5.

System suitability:

– *resolution*: minimum 1.5 between the peaks due to kaempferol and to isorhamnetin.

Do not take into account peaks eluting before the quercetin peak or after the isorhamnetin peak in the chromatogram obtained with the test solution.

Calculate the percentage content of flavonoids, expressed as flavone glycosides, from the expression:

$$2 \times \frac{F_1 \times m_1 \times 2.514 \times p}{F_2 \times m_2}$$

F_1 = sum of the areas of all the considered peaks in the chromatogram obtained with the test solution,

F_2 = area of the peak corresponding to quercetin in the chromatogram obtained with the reference solution,

m_1 = mass of quercetin used to prepare the reference solution, in grams,

m_2 = mass of the drug to be examined used to prepare the test solution, in grams,

p = percentage content of anhydrous quercetin in *quercetin dihydrate R*.