Loss on drying (*2.2.32*). Not more than 0.5 per cent, determined on 1.000 g by drying in an oven at 105 °C.

Sulphated ash (*2.4.14*). Not more than 0.1 per cent, determined on 1.0 g.

ASSAY

In order to avoid overheating in the reaction medium, mix thoroughly throughout and stop the titration immediately after the end-point has been reached.

Dissolve 0.200 g in 3.0 ml of *anhydrous formic acid R* and add 50.0 ml of *acetic anhydride R*. Titrate with *0.1 M perchloric acid* determining the end-point potentiometrically (*2.2.20*).

1 ml of *0.1 M perchloric acid* is equivalent to 23.82 mg of $C_{10}H_{14}N_4O_3$.

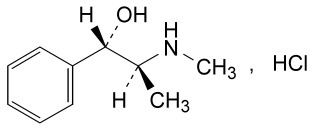
STORAGE

Store protected from light.

01/2008:1367
corrected 6.0

PSEUDOEPHEDRINE HYDROCHLORIDE

Pseudoephedrini hydrochloridum



$C_{10}H_{16}ClNO$ M_r 201.7
[345-78-8]

DEFINITION

(1*S*,2*S*)-2-(Methylamino)-1-phenylpropan-1-ol hydrochloride.

Content: 99.0 per cent to 101.0 per cent (dried substance).

CHARACTERS

Appearance: white or almost white, crystalline powder or colourless crystals.

Solubility: freely soluble in water and in ethanol (96 per cent), sparingly soluble in methylene chloride.

mp: about 184 °C.

IDENTIFICATION

First identification: A, B, D.

Second identification: A, C, D.

A. Specific optical rotation (see Tests).

B. Infrared absorption spectrophotometry (*2.2.24*).

Preparation: discs.

Comparison: *pseudoephedrine hydrochloride CRS*.

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

Test solution. Dissolve 20 mg of the substance to be examined in *methanol R* and dilute to 10 ml with the same solvent.

Reference solution (a). Dissolve 20 mg of *pseudoephedrine hydrochloride CRS* in *methanol R* and dilute to 10 ml with the same solvent.

Reference solution (b). Dissolve 10 mg of *ephedrine hydrochloride CRS* in reference solution (a) and dilute to 5 ml with reference solution (a).

Plate: *TLC silica gel plate R*.

Mobile phase: *methylene chloride R, concentrated ammonia R, 2-propanol R* (5:15:80 *V/V/V*).

Application: 10 µl.

Development: over a path of 15 cm.

Drying: in air.

Detection: spray with *ninhydrin solution R* and heat at 110 °C for 5 min.

System suitability: reference solution (b):

– the chromatogram shows 2 clearly separated spots.

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 reference solution (a).

D. Solution S (see Tests) gives reaction (a) of chlorides (*2.3.1*).

TESTS

Solution S. Dissolve 1.25 g in *carbon dioxide-free water 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. Dilute 2 ml of solution S to 10 ml with *carbon dioxide-free water R*. Add 0.1 ml of *methyl red solution R* and 0.1 ml of *0.01 M sodium hydroxide*; the solution is yellow. Add 0.2 ml of *0.01 M hydrochloric acid*; the solution is red.

Specific optical rotation (*2.2.7*): + 61.0 to + 62.5 (dried substance), determined on solution S.

Related substances. Liquid chromatography (*2.2.29*).

Test solution. Dissolve 50.0 mg of the substance to be examined in the mobile phase and dilute to 25.0 ml with the mobile phase.

Reference solution (a). Dissolve 20.0 mg of *ephedrine hydrochloride CRS* (impurity A) in the mobile phase and dilute to 20.0 ml with the mobile phase. Dilute 1.0 ml of this solution to 50.0 ml with the mobile phase.

Reference solution (b). Dilute 1.0 ml of the test solution to 200.0 ml with the mobile phase.

Reference solution (c). Dissolve 10 mg of *ephedrine hydrochloride CRS* (impurity A) in 5 ml of the test solution and dilute to 100 ml with the mobile phase.

Column:

– *size*: *l* = 0.25 m, Ø = 4.6 mm;

– *stationary phase*: *phenylsilyl silica gel for chromatography R* (5 µm).

Mobile phase: mix 6 volumes of *methanol R* and 94 volumes of an 11.6 g/l solution of *ammonium acetate R* adjusted to pH 4.0 with *glacial acetic acid R*.

Flow rate: 1 ml/min.

Detection: spectrophotometer at 257 nm.

Injection: 20 µl.

Run time: 1.5 times the retention time of pseudoephedrine.

System suitability: reference solution (c):

– *resolution*: minimum 2.0 between the peaks due to impurity A and pseudoephedrine; if necessary, reduce the content of methanol in the mobile phase.

Limits:

– *impurity A*: not more than the area of the principal peak in the chromatogram obtained with reference solution (a) (1 per cent);

– *any other impurity*: for each impurity, not more than the area of the principal peak in the chromatogram obtained with reference solution (b) (0.5 per cent);

Monographs P

– *sum of impurities other than A*: not more than twice the area of the principal peak in the chromatogram obtained with reference solution (b) (1 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).

Loss on drying (*2.2.32*): maximum 0.5 per cent, determined on 1.000 g by drying in an oven at 105 °C.

Sulphated ash (*2.4.14*): maximum 0.1 per cent, determined on 1.0 g.

ASSAY

Dissolve 0.170 g in 30 ml of *ethanol (96 per cent) R*. Add 5.0 ml of *0.01 M hydrochloric acid*. Carry out a potentiometric titration (*2.2.20*), using *0.1 M sodium hydroxide*. Read the volume added between the 2 points of inflexion.

1 ml of *0.1 M sodium hydroxide* is equivalent to 20.17 mg of $C_{10}H_{16}ClNO$.

STORAGE

Protected from light.

IMPURITIES

Specified impurities: *A*.

A. ephedrine.

01/2008:0858
corrected 6.0

PSYLLIUM SEED

Psyllii semen

DEFINITION

Ripe, whole, dry seeds of *Plantago afra* L. (*Plantago psyllium* L.) or *Plantago indica* L. (*Plantago arenaria* Waldstein and Kitaibel).

CHARACTERS

Sweet taste.

IDENTIFICATION

P. afra seeds are light brown to very dark brown but never black, smooth and shiny having an elliptical oblong shape. They are 2-3 mm long and 0.8-1.0 mm wide, one end being wider than the other. Towards the middle of the dorsal surface there is a fairly marked transverse constriction of light colour. On the ventral surface, there is a linear lighter-coloured groove in the middle of which is a clear spot corresponding to the hilum and bounded by swollen edges.

P. indica seeds are almost identical to the seeds of *P. afra*, but a little less shiny; they are 2-3 mm long and have a maximum diameter of 1.5 mm.

TESTS

Swelling index (*2.8.4*): minimum 10.

Foreign matter (*2.8.2*): maximum 1.0 per cent, determined on 10.0 g of the drug, including greenish unripe seeds. Psyllium seed does not contain seeds having a dark central spot on the groove (*Plantago lanceolata* L. and *P. major* L.) or seeds with brownish-grey or pinkish outer coats (*P. ovata* Forssk. and *P. sempervirens* Crantz).

Loss on drying (*2.2.32*): maximum 14.0 per cent, determined on 1.000 g of drug by drying in an oven at 105 °C for 2 h.

Total ash (*2.4.16*): maximum 4.0 per cent.

STORAGE

Store protected from moisture.

01/2008:1823
corrected 6.0

PURPLE CONEFLOWER HERB

Echinaceae purpureae herba

DEFINITION

Dried, whole or cut flowering aerial parts of *Echinacea purpurea* (L.) Moench.

Content: minimum 0.1 per cent for the sum of caftaric acid ($C_{13}H_{12}O_9$; M_r 312.2) and cichoric acid ($C_{22}H_{18}O_{12}$; M_r 474.3) (dried drug).

IDENTIFICATION

First identification: A, B, C.

Second identification: A, B, D.

A. The herbaceous perennial plant is 60-150 cm, rarely up to 180 cm high. The stem is green to red, upright and slightly branched. The leaves are alternate, ovate to ovate-lanceolate, irregularly serrate, rugose on both surfaces, dark green with prominent light green veins; the lamina is thick and shiny. The involucral bracts of the large capitulum are arranged in 2 or 3 rows. The solid receptacle is slightly convex. Each of the outer violet ligulate florets (4-6 cm) and of the inner violet-pink tubular florets is attached to a reddish acute and coriaceous bract, which overtops the tubular florets. The calyx is reduced to a very short crown, one of the sepals is up to 1 mm long.

B. Reduce to a powder (355) (*2.9.12*). The powder is green. Examine under a microscope using *chloral hydrate solution R*. The powder shows the following diagnostic characters: whitish-green groups of fibres, 150-200 µm in length, 10-15 µm in diameter, sometimes with black deposits; fragments of leaves in surface view showing anomocytic or anisocytic stomata (*2.8.3*) (about 35-40 µm in length); uniseriate covering trichomes or fragments thereof consisting mainly of 3 or 4 thick-walled cells of which the apical cell is markedly longer than the others; fragments of leaves with rosette-like arranged epidermal cells around the base of the covering trichomes; uniseriate glandular trichomes composed of very thin-walled cells; pitted parenchymatous cells from the pith of the stem as well as pitted elongated cells from the mesocarp of the achenes; fragments of parenchyma from the seeds with oil droplets; fragments of the epidermis of ligulate florets composed of red to violet papillous cells; spheroidal pollen grains, 30-40 µm in diameter, with a spiny exine.

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

Test solution. To 1.0 g of the powdered drug (355) (*2.9.12*) add 10 ml of *methanol R* and sonicate for 5 min. Centrifuge and use the supernatant solution.

Reference solution. Dissolve 0.5 mg of *caffeic acid R* and 0.5 mg of *chlorogenic acid R* in 5.0 ml of *methanol R*.

Plate: *TLC silica gel plate R* (5-40 µm) [or *TLC silica gel plate R* (2-10 µm)].

Mobile phase: *anhydrous formic acid R, water R, methyl ethyl ketone R, ethyl acetate R* (3:3:9:15 *V/V/V/V*).

Application: 25 µl [or 5 µl] of the test solution and 10 µl [or 2 µl] of the reference solution, as bands.

Development: over a path of 15 cm [or 5 cm].