1 ml of *0.1 M perchloric acid* is equivalent to 10.51 mg of $C_3H_7NO_3$.

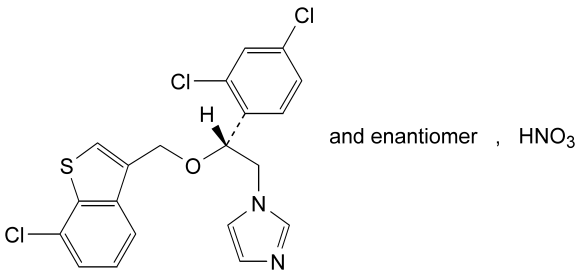
STORAGE

Store protected from light.

01/2008:1148

SERTACONAZOLE NITRATE

Sertaconazoli nitras



$C_{20}H_{16}Cl_3N_3O_4S$ M_r 500.8

[99592-39-9]

DEFINITION

(*RS*)-1-[2-[(7-Chloro-1-benzothiophen-3-yl)methoxy]-2-(2,4-dichlorophenyl)ethyl]-1*H*-imidazole nitrate.

Content: 98.5 per cent to 101.0 per cent (anhydrous substance).

CHARACTERS

Appearance: white or almost white powder.

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

IDENTIFICATION

First identification: A, C.

Second identification: A, B, D, E.

A. Melting point (*2.2.14*): 156 °C to 161 °C.

B. Ultraviolet and visible absorption spectrophotometry (*2.2.25*).

Test solution. Dissolve 0.1 g in *methanol R* and dilute to 100 ml with the same solvent. Dilute 10 ml of this solution to 100 ml with *methanol R*.

Spectral range: 240-320 nm.

Absorption maxima: at 260 nm, 293 nm and 302 nm.

Absorbance ratio: A_{302}/A_{293} = 1.16 to 1.28.

C. Infrared absorption spectrophotometry (*2.2.24*).

Preparation: dry the substances at 100-105 °C for 2 h and examine as discs of *potassium bromide R*.

Comparison: *sertaconazole nitrate CRS*.

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

Solvent mixture: *concentrated ammonia R, methanol R* (10:90 *V/V*).

Test solution. Dissolve 40 mg of the substance to be examined in the solvent mixture and dilute to 10 ml with the solvent mixture.

Reference solution (a). Dissolve 40 mg of *sertaconazole nitrate CRS* in the solvent mixture and dilute to 10 ml with the solvent mixture.

Reference solution (b). Dissolve 20 mg of *miconazole nitrate CRS* in reference solution (a) and dilute to 5 ml with reference solution (a).

Plate: *TLC silica gel G plate R.*

Mobile phase: *concentrated ammonia R, toluene R, dioxan R* (1:40:60 *V/V/V*).

Application: 5 µl.

Development: over a path of 15 cm.

Drying: in a current of air for 15 min.

Detection: expose to iodine vapour for 30 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).

E. About 1 mg gives the reaction of nitrates (*2.3.1*).

TESTS

Appearance of solution. The solution is clear (*2.2.1*) and not more intensely coloured than reference solution Y_5 (*2.2.2, Method II*).

Dissolve 0.1 g in *ethanol (96 per cent) R* and dilute to 10 ml with the same solvent.

Related substances. Liquid chromatography (*2.2.29*).

Test solution. Dissolve 10.0 mg of the substance to be examined in the mobile phase and dilute to 10.0 ml with the mobile phase.

Reference solution (a). Dilute 5.0 ml of the test solution to 100.0 ml with the mobile phase. Dilute 1.0 ml of this solution to 20.0 ml with the mobile phase.

Reference solution (b). Dissolve 5.0 mg of *sertaconazole nitrate CRS* and 5.0 mg of *miconazole nitrate CRS* 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.

Column:

– *size*: *l* = 0.25 m, Ø = 4.0 mm;

– *stationary phase*: *nitrile silica gel for chromatography R1* (10 µm).

Mobile phase: *acetonitrile R*, 1.5 g/l solution of *sodium dihydrogen phosphate R* (37:63 *V/V*).

Flow rate: 1.6 ml/min.

Detection: spectrophotometer at 220 nm.

Injection: 20 µl.

Run time: 1.3 times the retention time of sertaconazole.

Retention time: nitrate ion = about 1 min; miconazole = about 17 min; sertaconazole = about 19 min.

System suitability: reference solution (b):

– *resolution*: minimum 2.0 between the peaks due to miconazole and sertaconazole.

Limits:

– *impurities A, B, C*: for each impurity, not more than the area of the principal peak in the chromatogram obtained with reference solution (a) (0.25 per cent);

– *total*: not more than twice the area of the principal peak in the chromatogram obtained with reference solution (a) (0.5 per cent);

– *disregard limit*: 0.2 times the area of the principal peak in the chromatogram obtained with reference solution (a) (0.05 per cent); disregard the peak due to the nitrate ion.

Water (*2.5.12*): maximum 1.0 per cent, determined on 0.50 g.

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

ASSAY

Dissolve 0.400 g in 50 ml of a mixture of equal volumes of *anhydrous acetic acid R* and *methyl ethyl ketone R*. Titrate with *0.1 M perchloric acid,* determining the end-point potentiometrically (*2.2.20*). Carry out a blank titration.

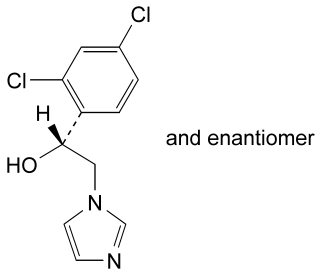
1 ml of *0.1 M perchloric acid* is equivalent to 50.08 mg of $C_{20}H_{16}Cl_3N_3O_4S$.

STORAGE

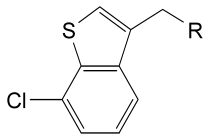
Protected from light.

IMPURITIES

Specified impurities: *A, B, C.*



A. (1*RS*)-1-(2,4-dichlorophenyl)-2-(1*H*-imidazol-1-yl)ethanol,



B. R = Br: 3-(bromomethyl)-7-chloro-1-benzothiophen,

C. R = OH: (7-chloro-1-benzothiophen-3-yl)methanol.

01/2008:0433

SESAME OIL, REFINED

Sesami oleum raffinatum

DEFINITION

Fatty oil obtained from the ripe seeds of *Sesamum indicum* L. by expression or extraction. It is then refined. Improved colour and odour may be obtained by further refining. It may contain a suitable antioxidant.

CHARACTERS

Appearance: clear, light yellow liquid, almost colourless.

Solubility: practically insoluble in ethanol (96 per cent), miscible with light petroleum.

Relative density: about 0.919.

Refractive index: about 1.473.

It solidifies to a butter-like mass at about – 4 °C.

IDENTIFICATION

First identification: A.

Second identification: B.

A. Composition of triglycerides (see Tests).

B. Carry out the identification of fatty oils by thin-layer chromatography (*2.3.2*). The chromatogram obtained is similar to the typical chromatogram for sesame oil.

TESTS

Acid value (*2.5.1*): maximum 0.5, determined on 10.0 g; maximum 0.3 if intended for use in the manufacture of parenteral dosage forms.

Peroxide value (*2.5.5*): maximum 10.0; maximum 5.0 if intended for use in the manufacture of parenteral dosage forms.

Unsaponifiable matter (*2.5.7*): maximum 2.0 per cent, determined on 5.0 g.

Alkaline impurities (*2.4.19*). It complies with the test for alkaline impurities in fatty oils.

Cottonseed oil. Mix 5 ml in a test-tube with 5 ml of a mixture of equal volumes of *pentanol R* and a 10 g/l solution of *sulphur R* in *carbon disulphide R*. Warm the mixture carefully until the carbon disulphide is expelled, and immerse the tube to 1/3 of its depth in boiling *saturated sodium chloride solution R*. No reddish colour develops within 15 min.

Composition of triglycerides. Liquid chromatography (*2.2.29*).

Test solution. Dilute 50.0 mg of the substance to be examined to 10.0 ml with a mixture of equal volumes of *acetone R* and *methylene chloride R*.

Reference solutions. Dissolve 80.0 mg of *triolein R* in a mixture of equal volumes of *acetone R* and *methylene chloride R* and dilute to 50.0 ml with the same mixture of solvents. Prepare 5 reference solutions by dilution of this solution so as to cover concentrations ranging from the disregard limit (0.5 per cent) to the upper limit for OLL (30.0 per cent).

Plot the logarithm of the area of the peak due to triolein against the logarithm of the mass of triolein in the reference solution, in milligrams.

Column: 2 columns coupled in series:

– *size of each column*: *l* = 0.25 m, Ø = 4 mm;

– *stationary phase*: *octadecylsilyl silica gel for chromatography R* (4 µm).

Mobile phase:

– *mobile phase A*: *acetone R, methylene chloride R, acetonitrile R* (5:15:80 *V/V/V*);

– *mobile phase B*: *acetone R, acetonitrile R, methylene chloride R* (20:20:60 *V/V/V*);

Time (min)	Mobile phase A (per cent *V/V*)	Mobile phase B (per cent *V/V*)
0 - 15	100 → 75	0 → 25
15 - 25	75	25
25 - 70	75 → 0	25 → 100
70 - 75	0 → 100	100 → 0
75 - 80	100	0

Flow rate: 1.0 ml/min.

Detection: evaporative light-scattering detector; the following settings have been found to be suitable; if the detector has different setting parameters, adjust the detector settings so as to comply with the system suitability criterion: