Edition: BP 2025 (Ph. Eur. 11.6 update)

Tenoxicam Tablets

General Notices

Action and use

Cyclo-oxygenase inhibitor; analgesic; anti-inflammatory.

DEFINITION

Tenoxicam Tablets contain Tenoxicam.

The tablets comply with the requirements stated under Tablets and with the following requirements.

Content of tenoxicam, $C_{13}H_{11}N_3O_4S_2$

92.5 to 105.0% of the stated amount.

IDENTIFICATION

A. In the Assay, the principal peak in the chromatogram obtained with solution (1) has the same retention time as the peak in the chromatogram obtained with solution (2).
B. Carry out the method for *thin-layer chromatography*, Appendix III A, using the following solutions.
(1) Mix with the aid of ultrasound a quantity of the powdered tablets containing 20 mg of Tenoxicam with 20 mL of *dichloromethane* for 15 minutes, centrifuge and use the supernatant liquid.
(2) 0.1% w/v of *tenoxicam BPCRS* in *dichloromethane*.

CHROMATOGRAPHIC CONDITIONS

(a) Use as the coating *silica gel F_{254}* (Merck silica gel 60 F_{254} plates are suitable).
(b) Use the mobile phase as described below.
(c) Apply 10 µL of each solution.
(d) Develop the plate to 10 cm.
(e) After removal of the plate, dry in air and examine under *ultraviolet light (254 nm)*.

MOBILE PHASE

4 volumes of *anhydrous formic acid*, 30 volumes of *acetone* and 70 volumes of *dichloromethane*.

CONFIRMATION

The principal spot in the chromatogram obtained with solution (1) corresponds in position and colour to that in the chromatogram obtained with solution (2).

TESTS

Dissolution

Comply with the requirements for Monographs of the British Pharmacopoeia in the *dissolution test for tablets and capsules*, Appendix XII B1.

TEST CONDITIONS

(a) Use Apparatus 2, rotating the paddle at 50 revolutions per minute.
(b) Use 900 mL of phosphate buffer pH 6.8 prepared in the following manner: dissolve 6.8 g of *potassium dihydrogen orthophosphate* in 500 mL of *water*, add 23 mL of 1M *sodium hydroxide*, dilute to 1000 mL and, if necessary, adjust the pH to 6.8 using either 1M *sodium hydroxide* or a 10% w/v solution of *orthophosphoric acid*, at a temperature of 37°, as the medium.

PROCEDURE

(1) After 45 minutes withdraw a 10 mL sample of the medium and measure the *absorbance* of the filtered sample, suitably diluted with the dissolution medium if necessary, at the maximum at 368 nm, Appendix II B.
(2) Measure the *absorbance* of a suitable solution of *tenoxicam BPCRS*.

DETERMINATION OF CONTENT

Calculate the total content of tenoxicam, $C_{13}H_{11}N_3O_4S_2$, in the medium from the absorbances obtained and using the declared content of $C_{13}H_{11}N_3O_4S_2$ in *tenoxicam BPCRS*.

Related substances

Carry out the method for *liquid chromatography*, Appendix III D, using the following solutions.

(1) Shake a number of whole tablets containing 0.1 g of Tenoxicam with 100 mL of *acetonitrile* (*50%*) for 70 minutes, mixing occasionally with the aid of ultrasound. Allow to stand for 10 minutes, dilute 5 volumes of the clear supernatant liquid to 20 volumes with the mobile phase and filter through a 0.45-µm membrane filter.
(2) Dilute 1 volume of solution (1) to 200 volumes with the mobile phase.
(3) 0.0000625% w/v of *2-pyridylamine* (impurity A) in *acetonitrile* (*50%*).
(4) Dilute 1 volume of a 0.1% w/v solution of *tenoxicam degradation impurity standard BPCRS* in *acetonitrile* (*50%*) to 4 volumes with the mobile phase.

CHROMATOGRAPHIC CONDITIONS

(a) Use a stainless steel column (25 cm × 4 mm) packed with *octylsilyl silica gel for chromatography* (5 µm) (Nucleosil C8 5µ is suitable) and a pre-column packed with *octylsilyl silica gel for chromatography* (10 µm) (Spheri-10 RP8, RP-GU pre-column is suitable).
(b) Use isocratic elution and the mobile phase described below.
(c) Use a flow rate of 0.7 mL per minute.
(d) Use an ambient column temperature.
(e) Use detection wavelengths of 254 nm and 290 nm.
(f) Inject 20 µL of each solution.

Condition the column with the mobile phase for 3 hours.

MOBILE PHASE

Dissolve 0.12 g of *sodium dodecyl sulfate* in 700 mL of *methanol*, mix with 1000 mL of 0.05M *potassium dihydrogen orthophosphate* and adjust the pH to 2.8 with *orthophosphoric acid*.

SYSTEM SUITABILITY

The test is not valid unless the chromatogram obtained with solution (4) closely resembles the reference chromatogram supplied with *tenoxicam degradation impurity standard BPCRS*.

LIMITS

At a detection wavelength of 290 nm In the chromatogram obtained with solution (1):

the area of any peak corresponding to 2-pyridylamine (impurity A) is not greater than the area of the principal peak in the chromatogram obtained with solution (3) (0.25%).

At a detection wavelength of 254 nm In the chromatogram obtained with solution (1):

the area of any *secondary peak* is not greater than the area of the principal peak in the chromatogram obtained with solution (2) (0.5%);

the sum of the areas of any such peaks is not greater than four times the principal peak in the chromatogram obtained with solution (2) (2%).

ASSAY

Carry out the method for *liquid chromatography*, Appendix III D, using the following solutions.

(1) Shake 10 whole tablets with 200 mL of *acetonitrile* (*50%*) for 70 minutes, mixing occasionally with the aid of ultrasound. Allow to stand for 10 minutes, dilute a volume of the clear supernatant liquid with sufficient mobile phase to produce a solution containing 0.025% w/v of Tenoxicam and filter through a 0.45-µm membrane filter.
(2) Dilute 5 mL of a 0.1% w/v solution of *tenoxicam BPCRS* in *acetonitrile* (*50%*) to 20 mL with the mobile phase.
(3) Dilute 1 volume of a 0.1% w/v solution of *tenoxicam degradation impurity standard BPCRS* in *acetonitrile* (*50%*) to 4 volumes with the mobile phase.

CHROMATOGRAPHIC CONDITIONS

The chromatographic conditions described under Related substances may be used with a detection wavelength of 290 nm.

MOBILE PHASE

Dissolve 0.12 g of *sodium dodecyl sulfate* in 700 mL of *methanol*, mix with 1000 mL of 0.05M *potassium dihydrogen orthophosphate* and adjust the pH to 2.8 with *orthophosphoric acid*.

SYSTEM SUITABILITY

The test is not valid unless the chromatogram obtained with solution (3) closely resembles the reference chromatogram supplied with *tenoxicam degradation impurity standard BPCRS*.

DETERMINATION OF CONTENT

Calculate the content of $C_{13}H_{11}N_3O_4S_2$ in the tablets using the declared content of $C_{13}H_{11}N_3O_4S_2$ in *tenoxicam BPCRS*.

IMPURITIES

The impurities limited by the requirements of this monograph include those impurities listed under Tenoxicam.