Diclofenac Potassium Tablets

Diclofenac Potassium Tablets contains not less than 90.0 per cent and not more than 110.0 per cent of the stated amount of diclofenac potassium, $C_{14}H_{10}Cl_2KNO_2$.

Usual strengths. 25 mg; 50 mg.

Identification

In the Assay, the principal peak in the chromatogram obtained with the test solution corresponds to the peak in the chromatogram obtained with the reference solution.

Tests

Dissolution (2.5.2).
Apparatus No. 2 (Paddle),
Medium. 900 ml of *intestinal fluid, simulated* (without enzyme),
Speed and time. 50 rpm and 60 minutes.

Withdraw a suitable volume of the medium and filter. Measure the absorbance of the filtrate, suitably diluted with medium, if necessary, at the maximum at about 276 nm (2.4.7). Calculate the content of $C_{14}H_{10}Cl_2KNO_2$in the medium from the absorbance obtained from a solution of known concentration *diclofenac potassium IPRS* in the dissolution medium.

Q. Not less than 75 per cent of the stated amount of $C_{14}H_{10}Cl_2KNO_2$.

Related substances. Determine by liquid chromatography (2.4.14).

NOTE - Prepare the solutions, protected from light.

Solvent mixture. Equal volumes of *acetonitrile* and *water*.

Test solution. Disperse a quantity of powdered tablets containing 100 mg of Diclofenac Potassium in 80 ml of the solvent mixture and dilute to 100.0 ml with the solvent mixture.

Reference solution. A solution containing 0.0001 per cent w/v, each of, *diclofenac potassium IPRS* and *diclofenac related compound A IPRS* in the solvent mixture.

Chromatographic system
- a stainless steel column 10 cm x 2.0 mm, packed with octadecylsilane bonded to porous silica (1.9 µm).
- column temperature: 35°,
- mobile phase: A. *0.01 M ammonium acetate*, adjusted to pH 5.3 with *glacial acetic acid*,
  B. *acetonitrile*,
- flow rate: 0.3 ml per minute,
- spectrophotometer set at 254 nm,
- injection volume: 1 µl.

Time (in min.)	Mobile phase A (per cent v/v)	Mobile phase B (per cent v/v)
0	70	30
0.5	70	30
8.5	5	95
10	5	95
10.1	70	30
15	70	30

Name	Relative retention time
Oxindole [1*]	0.4
Diclofenac	1.0
Diclofenac related compound D[2*]	1.04
Diclofenac related compound A[3]	1.48
Diclofenac alcohol analog[4*]	1.55
Diclofenac benzaldehyde analog[5*]	1.81

[*] Process-related impurities, included for identification only, not to be included in total impurities.
[1]1,3-Dihydro-2H- indol-2-one,
[2]2-{2[(2-Bromo-6-chlorophenyl)amino]phenyl}acetic acid,
[3]N-(2,6-Dichlorophenyl)indolin-2-one (diclofenac lactum),
[4]{2-[(2,6-Dichlorophenyl)amino]phenyl}methanol,
[5]2-[(2,6-Dichlorophenyl)amino]benzaldehyde.

Inject the reference solution. The test is not valid unless the relative standard deviation for replicate injections is not more than 5.0 per cent and the signal to noise ratio for the principal peak is not less than 10.

Inject the reference solution and the test solution. In the chromatogram obtained with the test solution, the area of any peak corresponding to diclofenac related compound A is not more than 5 times the area of diclofenac related compound A peak in the chromatogram obtained with the reference solution (0.5 per cent), the area of any other secondary peak is not more than 5 times of the area of the principal peak in the chromatogram obtained with the reference solution (0.5 per cent) and the sum of the areas of all the secondary peaks is not more than 15 times the area of the principal peak in the chromatogram obtained with the reference solution (1.5 per cent). Ignore any peak with an area less than 0.5 times the area of the principal peak in the chromatogram obtained with the reference solution (0.05 per cent).

Other tests. Comply with the tests stated under Tablets.

Assay. Determine by liquid chromatography (2.4.14), as described under Related substances with the following modifications.

Test solution. Weigh and powder 20 tablets. Disperse a quantity of the powder containing 20 mg of Diclofenac Potassium in 80 ml of the solvent mixture and dilute to 100.0 ml with the solvent mixture.

Reference solution. A 0.02 per cent w/v solution of *diclofenac potassium IPRS* in the solvent mixture.

– spectrophotometer set at 280 nm,

Inject the reference solution. The test is not valid unless the tailing factor is not more than 1.2 and the relative standard deviation for replicate injections is not more than 1.0 per cent.

Inject the reference solution and the test solution.

Calculate the content of $C_{14}H_{10}Cl_2KNO_2$ in the tablets.

Storage. Store protected from light and moisture, at a temperature not exceeding 30°.