Ciprofloxacin and Tinidazole Tablets

Ciprofloxacin Hydrochloride and Tinidazole Tablets

Ciprofloxacin and Tinidazole Tablets contain Ciprofloxacin Hydrochloride equivalent to not less than 90.0 per cent and not more than 110.0 per cent of the stated amount of Ciprofloxacin, $C_{17}H_{18}FNO_3$ and Tinidazole $C_8H_{13}N_3O_4S$.

Usual strengths. Ciprofloxacin 250 mg and Tinidazole 300 mg; Ciprofloxacin 500 mg and Tinidazole 600 mg;

Identification

In the Assay, the principal peaks in the chromatogram obtained with the test solution correspond to the principal peaks in the chromatogram obtained with the reference solution.

Tests

Dissolution (2.5.2).

Apparatus No. 1,
Medium. 900 ml of *water,*
Speed and time. 50 rpm and 30 minutes.

Withdraw a suitable volume of the medium and filter.

Determine by liquid chromatography (2.4.14),

Test solution. Dilute the filtrate, if necessary with the dissolution medium.

Reference solution. A solution containing 0.01 per cent w/v of *ciprofloxacin hydrochloride RS* and 0.012 per cent w/v of *tinidazole RS* in the dissolution medium.
Chromatographic system

- a stainless steel column 15 cm x 3.9 mm, packed with octadecylsilane bonded to porous silica (4 µm),
- mobile phase: a mixture of 83 volumes of buffer solution prepared by dissolving 3.03 g of *heptane sulphonic acid sodium salt* in 1000 ml with *water*, add 1 ml *trimethylamine*, adjusted to pH 2.5 with *orthophosphoric acid* and 17 volumes of *acetonitrile.*
- flow rate: 1.5 ml per minute,
- spectrophotometer set at 300 nm,
- injection volume: 20 µl.

Inject the reference solution. The test is not valid unless the column efficiency is not less than 2000 theoretical plates, the tailing factor is not more than 2.0 and the relative standard deviation for replicate injections is not more than 2.0 per cent for both the peaks.

Inject the reference solution and the test solution.

Calculate the contents of $C_{17}H_{18}FN_3O_3$ and $C_8H_{13}N_3O_4S$ in the medium.

D. Not less than 70 per cent of the stated amount of $C_{17}H_{18}FN_3O_3$ and $C_8H_{13}N_3O_4S$.

Related substances. Determine by liquid chromatography (2.4.14).

Solvent mixture. A mixture of 83 volumes of mobile phase A and 17 volumes of mobile phase B.

Test solution. Disperse a quantity of the powdered tablets containing 300 mg of Tinidazole in 25 ml of mobile phase B with the aid of ultrasound for 10 minutes and dilute to 250.0 ml with the solvent mixture and filter.

Reference solution. Dissolve about 55 mg of *ciprofloxacin hydrochloride RS* and 50 mg of *tinidazole RS* in 10 ml of mobile phase B with the aid of ultrasound for 10 minutes and dilute to 100.0 with the solvent mixture. Dilute 5.0 ml of the solution to 50.0 ml with the solvent mixture. Further dilute 10.0 ml of the solution to 50.0 ml with the solvent mixture.

Chromatographic system

- a stainless steel column 15 cm x 4.6 mm, packed with octadecylsilane bonded to porous silica (5 µm) (Such as YMC Paek ODS-AM),
- column temperature: 40º,
- mobile phase: A. a buffer solution prepared by dissolving 2.45 g of *orthophosphoric acid* in 1000 ml of *water*, adjusted to a pH 3.0 with *trimethylamine*,
  B. A mixture of 95 volumes of *methanol* and 5 volumes of *tetrahydrofuran*,
- a gradient programme using the conditions given below,
- flow rate: 1 ml per minute,
- spectrophotometer set at 300 nm,
- injection volume: 10 µl.

Time (in min.)	Mobile phase A (per cent v/v)	Mobile phase B (per cent v/v)
0	95	5

15	88	12
35	88	12
37	40	60
48	40	60
50	95	5
65	95	5

Name	Relative retention time
Tinidazole impurity A[1]	0.17
Ciprofloxacin impurity E[2]	0.42
Ciprofloxacin	1.0

[1]2-Methyl-5-Nitroimidazole,
[2]Decarbboxylated analogue.

Inject the reference solution. The test is not valid unless the column efficiency is not less than 10000 theoretical plates, the resolution between the peaks due to tinidazole and ciprofloxacin is not less than 2.0, the tailing factor is not more than 2.0 and the relative standard deviation for replicate injections is not more than 5.0 per cent for both the peaks.

Inject the reference solution and the test solution. In the chromatogram obtained with the test solution, the area of the any peak corresponding to tinidazole impurity A is not more than 0.5 times the area of the ciprofloxacin peak in the chromatogram obtained with the reference solution (0.5 per cent), the area of the any peak corresponding to ciprofloxacin impurity E is not more than 0.2 times the area of the ciprofloxacin peak in the chromatogram obtained with the reference solution (0.2 per cent), the area of any other secondary peak is not more than 0.2 times the area of the ciprofloxacin peak in the chromatogram obtained with reference solution (0.2 per cent) and the sum of the areas of all the secondary peaks is not more than the area of the ciprofloxacin peak in the chromatogram obtained with the reference solution (1.0 per cent). Ignore any peak with an area less than 0.05 times the area of the ciprofloxacin 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)

Test solution. Weigh and powder 20 tablets. Disperse a quantity of the powder containing 100 mg of Ciprofloxacin in 10 ml of 7 per cent v/v solution of *orthophosphoric acid*, add 50.0 ml of the mobile phase, with the aid of ultrasound and dilute to 100.0 ml with the mobile phase, filter. Dilute 1.0 ml of the solution to 10.0 ml with the mobile phase.

Reference solution. A 0.01 per cent w/v solution of *ciprofloxacin hydrochloride RS* and 0.012 per cent w/v solution of *tinidazole RS* in the solvent mixture.

Use the chromatographic system as described under the Dissolution.

Inject the reference solution. The test is not valid unless the column efficiency is not less than 2000 theoretical plates, the tailing factor is not more than 2.0 and the relative standard deviation for replicate injections is not more than 2.0 per cent for both the peaks..

Inject the reference solution and the test solution.

Calculate the contents of $C_{17}H_{18}FN_3O_3$and $C_8H_{13}N_3O_4S$ in the tablets.

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