Department of Drug Administration
National Medicines Laboratory
ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT

Aceclofenac Sustained Release Tablet

Analytical Profile No.: AST 074/075/ AP 032
Aceclofenac SR tablet contains 90-110% of Aceclofenac of stated amount.

1. Identification:

In the assay, the principle peak in the chromatogram obtained with the sample solution should correspond to the peak in the chromatogram obtained with the reference standard solution of Aceclofenac.

2. Dissolution: Determine by Liquid Chromatography

2.1 Dissolution Parameters

- **Apparatus:** Paddle
- **Medium:**900 ml, Phosphate buffer pH 7.5
- **Speed:**50 rpm
- **Time:** 1st hour, 4th hour, 8th hour and 16 th hour

2.2Test Solution:

Place 1 tablet in each dissolution vessel and run the apparatus as per above condition and collect the sample solution from each jar at specified time. Withdraw a suitable volume of sample at the determined time interval, filter, and replace the withdrawn volume with same medium. Dilute 2 ml of the filtrate to 50 ml with dissolution medium.

2.3Reference Solution:

Weigh accurately about 44.4 mg of Aceclofenac RS in 100 ml volumetric flask and add about 70 ml dissolution medium and sonicate for about 10 min and make volume to 100 ml with dissolution medium. Dilute 5 ml of the solution to 100 ml with dissolution medium.

2.4 Chromatographic System:

Use the chromatographic system as described in the Assay.

2.5Procedure:

Proceed as described in assay, using 20 µl injection volumes. Calculate the release of the drug in each tablet.

2.6 Limit:
1st hour - NMT 25%
4 thhour - 20 to 50 %
8 th hour - 50 to80 %
16 th hour - NLT 80 % of the stated amount

3. Assay:

3.1 Diluent: Water: Acetonitrile (45:55)

3.2TestSolution:

Weigh and powder 20 tablets.Weighpowder eq. to 100 mg of Aceclofenac in 100ml volumetric flask. Add about 70ml of Aceclofenac and sonicate for 10 minutes, make up the volume to 100 ml with Aceclofenac. Filter and dilute 5 ml of the filtrate to 50 ml with diluent. Filter the resulting solution through 0.22 micron nylon membrane filter.

3.3Reference Solution:

Weigh accurately about 25 mg of Aceclofenac RS in 25 ml volumetric flask and add about 15 ml of Acetonitrile and sonicate for about 10 min. Cool and make volume to 25 ml with Acetonitrile. Dilute 5 ml of the solution to 50 ml with diluents. Filter the resulting solution through 0.22 micron nylon membrane filter.

3.4 Chromatographic system

- **Column:** C18, (150*4.6 mm), 5 µm
- **Flow rate:** 1.5 ml/min
- **Injection volume:** 10 µl
- **Wavelength:** 275 nm
- **Column temperature:** Ambient
- **Detector:** UV Detector

Mobile phase: Buffer:Acetonitrile (55:45)

Buffer:1 ml of glacial acetic acid in 1000 ml of water

Mix buffer and Acetonitrile, cool to room temperature and filter the solution through 0.2 micron filter paper using vacuum pump.

3.5 Procedure:

Inject 10µl of standard preparation five/six times. The test is not valid unless the column efficiency is not less than 2500 theoretical plates. The tailing factor is not more than 2.0 and the relative standard deviation for replicate injections in not more than 2.0%. After the completion of the system suitability test parameter, inject 10µl of each of the sample solution separately. Calculate the content of Aceclofenac in each tablet.

4. Other tests: As per pharmacopoeial requirement.