

DEPARTMENT OF DRUG ADMINISTRATION
National Medicines Laboratory
ANALYTICAL METHOD VALIDATION COMMITTEE

Aceclofenac Sustained release Tablets

Analytical Profile No.: AST 074/075/032

Aceclofenac Sustained release Tablets contains not less than 90.0 % and not more than 110.0 % of the stated amount of Aceclofenac.

1. Identification:

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

2. Dissolution:

2.1 Dissolution Parameters:

Apparatus: Paddle

Medium: 900ml of pH 7.5 phosphate buffer

Speed and Time: 50 rpm and 1, 4, 8 & 16 hours

[Phosphate buffer pH 7.5: Dissolve 6.8 g of monobasic potassium phosphate and 1.4 g sodium hydroxide in 1000 ml water and adjust the pH to 7.5 ± 0.05 with sodium hydroxide solution.]
Withdraw a suitable volume of sample after 1 hr, 4 hrs, 8hrs & 16 hrs consecutively by replacing the volume after each withdrawal with the same dissolution medium maintained at same temperature.

2.2 Test Solution: Dilute 5 ml of the filtrate to 50 ml with dissolution medium.

2.3 Reference Solution: Weigh accurately about 44.4 mg of Aceclofenac WS 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 Procedure: Measure the absorbance of reference and test solution at about 273 nm using dissolution medium as blank. Calculate the percentage release of Aceclofenac in each tablet.

2.5 Limit:

In 1st hour - NMT 25%

In 4th hour – NLT 20% & NMT 50 %

In 8th hour – NLT 50% & NMT 80 %

In 16th hour - NLT 80 % of the stated amount

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3. Assay:

3.1 Solvent Mixture: Water: Acetonitrile (45:55)

3.2 Test Solution: Weigh and powder 20 tablets. Weigh powder eq. to 100 mg of Aceclofenac in 100ml volumetric flask. Add about 70ml of Acetonitrile and sonicate for 10 minutes, make up the volume to 100 ml with Acetonitrile. Filter and dilute 5 ml of the filtrate to 50 ml with solvent mixture.

3.3 Standard 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 solvent mixture.

3.4 Chromatographic system:

- **Column:** Octyldecylsilane (C18), (150 x 4.6 mm), 5 µm
- **Flow rate:** 1.5 ml/min
- **Detector:** UV
- **Wavelength:** 275 nm
- **Injection volume:** 10 µl
- **Column Temperature:** Ambient
- **Mobile Phase:** A mixture of 55 volumes of buffer and 45 volumes of Acetonitrile
- **Buffer solution:** 1 ml of glacial acetic acid in 1000 ml of water

3.5 Procedure: Inject the reference solution five times and sample solutions. The test is not valid unless the column efficiency is not less than 2000 theoretical plates, tailing factor is not more than 2.0 and the relative standard deviation for replicate injections is not more than 2.0%.

Measure the peak responses. Calculate the content of Aceclofenac in the tablets.

4. Other tests: As per pharmacopoeial requirements.