

**ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON
PHARMACOPOEIAL PRODUCT**

DEPARTMENT OF DRUG ADMINISTRATION

Aceclofenac Sustained release Tablets

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. Assay:

2.1 Reagents Required:

1. Acetonitrile (HPLC Grade)
2. Glacial acetic acid
3. HPLC Grade water

2.2 Preparation of reagent solution:

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

2.2.2 Mobile phase: Buffer: Acetonitrile (55:45)

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

2.2.3 Diluent: Water: Acetonitrile (45:55)

2.3 Chromatographic system

2.3.1 Column: Octyldecylsilane (C18), (150*4.6 mm), 5 µm

2.3.2 Flow rate: 1.5 ml/min

2.3.3 Detector: UV Detector

2.3.4 Wavelength: 275 nm

2.3.5 Injection volume: 10 µl

2.3.6 Oven temperature: Ambient

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2.4 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 diluents. Filter the resulting solution through 0.22 micron nylon membrane filter.

2.5 Sample Preparation: 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 diluent. Filter the resulting solution through 0.22 micron nylon membrane filter.

2.6 Procedure

Inject 10 µl 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 is not more than 2.0%. After the completion of the system suitability test parameter, inject 10 µl of each of the sample solution separately. Calculate the content of Aceclofenac in each tablet by using following formula:

2.7 Calculations:

Content of Aceclofenac per Tablet:

$$\frac{Spl\ Peak\ Area}{Std\ Peak\ Area} \times \frac{Std\ wt}{Std\ Dil.} \times \frac{Spl\ Dil.}{Spl\ wt} \times Average\ wt \times Std\ Potency\ (\%) \times \frac{(100 - Std\ LOD)}{100}$$

3. Dissolution:

3.1 Dissolution Medium: Phosphate buffer pH 7.5 (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)

3.2 Dissolution test condition

3.2.1 Volume : 900 ml

3.2.2 Apparatus : 1 (Paddle)

3.2.3 RPM : 50

3.2.4 Time : 1st hour, 4th hour, 8th hour and 16th hour

3.3 Standard Solution:

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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.

3.4 Sample 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.

Procedure:

Measure the absorbance of standard and sample solution at about 273 nm using dissolution medium as blank. Calculate the percentage release of Aceclofenac in each tablet by using following formula:

3.5 Calculation:

$$\begin{aligned} & \% \text{ Release} \\ &= \frac{\text{Spl Abs}}{\text{Std Abs}} \times \frac{\text{Std wt}}{\text{Std Dil}} \times \frac{900}{\text{Label Claim}} \times \text{Spl dilution} \times \text{Std. Potency (\%)} \times \frac{100 - LOD}{100} \times 100 \% \end{aligned}$$