Government of Nepal
Ministry of Health and Population
Department of Drug Administration
National Medicines Laboratory
Quality and Method Validation Section

Analytical profile of Daclatasvir Tablets

Analytical Profile No.: DAC 075/076/AP040

Daclatasvir Tablets contains not less than 90.0% and not more than 110.0% of the stated amount of Daclatasvir.

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.

2.0 Dissolution: *Determine by liquid chromatography*

2.1 Dissolution Parameters:

Apparatus: Paddle

Medium: 1000 ml of Phosphate buffer pH 6.8 (Dissolve 6.8 g of potassium dihydrogen phosphate in 1000ml of water and adjust the pH to 6.8 with 0.2M sodium hydroxide and then add 7.5g of Brij 35)

Speed and Time: 75 rpm and 45 minutes

Withdraw a suitable volume of the medium and filter.

2.2 Test Solution: Dilute 1ml of the filtrate to 20ml with methanol.

2.3 Reference Solution: Weigh accurately about 33.0 mg Daclatasvir dihydrochloride WS in 100 ml volumetric flask. Add about 70 ml of dissolution medium, sonicate to dissolve and make up the volume to 100 ml with dissolution medium. Dilute 5ml of resulting solution to 50ml with dissolution medium. Further dilute 2 ml of this solution to 20 ml with methanol. (0.003mg/ml)

2.4 Procedure: Use the chromatographic system as described in the Assay. Inject the reference solution and the test solution.

Calculate the percent release of Daclatasvir.

2.5 Limit: Not less than 80% (D) of the stated amount

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3. Assay:

3.1 Test Solution: Weigh individually 20 tablets & crush the tablet into fine powder. Weigh accurately the powder eq. to 30 mg of Daclatasvir in 100 ml volumetric flask, add 70 ml of methanol, sonicate to dissolve with intermittent shaking at 20°C to 25°C temperature and make volume to 100 ml with same solvent. Stir for 10 minutes. Dilute 5ml of resulting solution to 50ml with same solvent. Further dilute 10ml of this solution to 25ml with same solvent & mix. (0.012 mg/ml)

3.2 Reference Solution: Weigh accurately about 33.0 mg Daclatasvir dihydrochloride WS in 100 ml volumetric flask. Add about 70 ml of methanol and sonicate for about 5 minutes and make up the volume to 100 ml with same solvent. Dilute 5ml of resulting solution to 50ml with methanol. Further dilute 10ml of this solution to 25ml with methanol & mix. (0.012 mg/ml)

3.3 Chromatographic system:

Column: Octyldecylsilane (C18), (250*4.6 mm), 5 µm

Flow rate: 1.0 ml/min

Wavelength: 315 nm

Injection volume: 5 µl

Column Temperature: 25°C

Mobile Phase: Methanol: Water (98:02)

3.4 Procedure: Inject the reference solution five/six times and test 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 Daclatasvir.

NOTE: 110 mg Daclatasvir dihydrochloride eq. to 100 mg Daclatasvir

4. Other tests: As per pharmacopoeial requirement.