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

Analytical profile of Dabigatran Etexilate Capsules

Analytical Profile No.: DAB 075/076/AP 037

Dabigatran Etexilate Capsules contains not less than 90.0% and not more than 110.0% of the stated amount of Dabigatran Etexilate.

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:

2.1 Dissolution Parameters:

Apparatus: Basket

Medium: 900 ml of 0.01N HCl

Speed and Time: 100 rpm and 45 minutes

Withdraw a suitable volume of the medium and filter.

2.2 Test Solution: After completion of the test withdraw a specimen from the dissolution medium & filter.

2.3 Reference Solution: Weigh accurately about 48.0 mg Dabigatran Etexilate (as Mesylate) working standard in 50 ml volumetric flask. Add about 30 ml of dissolution medium and sonicate for about 15 minutes, cool to room temperature and make up the volume to 50 ml with dissolution medium. Dilute 2ml of resulting solution to 20ml with dissolution medium.

2.4 Chromatographic system:

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

Flow rate: 1.0 ml/min

Wavelength: 341 nm

Injection volume: 10 µl

Column Temp.: 27ºC

Mobile phase: A mixture of 40 volume of Buffer & 60 volume of Acetonitrile

Buffer: Take 5ml Triethylamine in 1000ml of water, adjust pH to 3.0 with orthophosphoric acid

2.5 Procedure: Inject the reference solution and the test solution. 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 and calculate the % release of the drug by using following formula:

2.6 Limit: D. NLT 75 % of the stated amount

3. Assay:

3.1 Solvent Mixture: Buffer:Methanol (40:60)

3.2 Test Solution: Weigh accurately the powder eq. to 50 mg of Dabigatran Etexilate in 100 ml volumetric flask, add 70 ml of methanol & sonicate for 15 minutes, cool to room temperature and make volume to 100 ml with methanol. Stir for 15 minutes. Dilute 2ml of resulting solution to 20ml with solvent mixture.

3.3 Reference Solution: Weigh accurately about 57.65 mg Dabigatran Etexilate (as Mesylate) WS in 100 ml volumetric flask. Add about 70 ml of methanol and sonicate for about 10 minutes, cool to room temperature and make up the volume to 100 ml with same solvent. Dilute 2ml of resulting solution to 20ml with solvent mixture.

3.4 Chromatographic system:

Column: C18, (250 x 4.6 mm), 5 µm

Flow rate: 1.0 ml/min

Wavelength: 226 nm

Injection volume: 10 µl

Mobile phase: A mixture of 10 volume of Buffer & 90 volume of Methanol

Buffer (0.1N Ammonium acetate buffer pH 5.0): Weigh 7.71g ammonium acetate in 200ml water, add 1ml glacial acetic acid and dilute to 1000ml with water and adjust pH 5.0 with sodium hydroxide solution or orthophosphoric acid.

3.5 Procedure: Inject the reference solution five/six 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 in not more than 2.0%. Measure the peak responses. Calculate the content of Dabigatran Etexilate in Dabigatran Etexilate Capsules.

4. Other tests: As per Pharmacopoeial requirements.