ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT
DEPARTMENT OF DRUG ADMINISTRATION
National Medicines Laboratory

Dabigatran Etexilate Capsules

Analytical Profile No.: DAB075/076/AP037

Dabigatran Etexilate Capsules containnot less than 90% and not more than 110% of the stated amount of Dabigatran Etexilate.

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 Dabigatran Etexilate.

2. Dissolution: Determine by Thin Layer Chromatography

2.1 DissolutionParameters

Apparatus: Basket

Medium: 900 ml, 0.01N HCl

Speed and Time: 100 rpm at 45min

Time: 45 minutes

Temperature: 37˚C ± 0.5˚C

2.2 Chromatographic system:

Column: C18, (250*4.6 mm), 5 µm

Flow rate: 1.0 ml/min

Wavelength: 341 nm

Injection volume: 10 µl

Column Temp.: 27ºC

Detector: **UV**

Mobile phase:

Buffer: Acetonitrile (40:60)

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

2.3 Test Solution

Withdraw a suitable volume of the sample after 45 minutes. Filter the sample.

2.4Reference 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 and make up the volume to 50 ml with dissolution medium. Dilute 2ml of resulting solution to 20ml with dissolution medium.

2.5 Procedure:

Inject the reference solution. The test is not valid unless the column efficiency is not less than 2000 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%. Inject sample and measure the peak responses. Calculate the % release of the drug.

2.6 Limit:

D. NLT 75 % of the stated amount

3. Assay: Determine by Thin Layer Chromatography.

3.1 Test Solution

Weigh accurately the powder eq. to 50 mg of Dabigatran Etexilate in 100ml volumetric flask, add 70 ml of methanol & sonicate for 15 minutes, cool and make volume to 100 ml with methanol.Stir for 15 minutes. Dilute 2ml of resulting solution to 20ml with diluent. Filter the final solution through 0.2 µm membrane filter.

3.2Reference Solution

Weigh accurately about 57.65 mg Dabigatran Etexilate (as Mesylate) working standard in 100 ml volumetric flask. Add about 70 ml of methanol and sonicate for about 10 minutes and make up the volume to 100 ml with same solvent. Dilute 2ml of resulting solution to 20ml with diluent.Filter the final solution through 0.2 µm membrane filter.

3.3 Chromatographic system:

Column: C18, (250*4.6 mm), 5 µm

Flow rate: 1.0 ml/min

Wavelength: 226 nm

Injection volume: 10 µl

Detector: **UV**

Column Temperature: Ambient

Mobile phase:

Buffer: methanol (10:90)

Buffer: 0.1N Ammonium acetate buffer pH 5.0

Diluent:buffer: methanol (40:60)

2.6 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 per capsule.

2.7Other test: As per pharmacopoieal requirement.