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

Dutasteride Tablets

Analytical Profile No.: DUT 075/076/AP036

Dutasteride Tablets contain not less than 90% and not more than 110% of the stated amount of Dutasteride.

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: Determine by liquid chromatography

2.1 Dissolution Parameters:

Apparatus: Paddle

Medium: 450 ml of medium A for first 25 minutes followed by addition of 450 ml of medium B.

Medium A: To 1000 ml of 0.1 M hydrochloric acid, add and dissolve 1.6 g of Pepsin (label activity 1:3,000)

Medium B: To 1000 ml of 0.1 M hydrochloric acid, add and dissolve 40 g of sodium lauryl sulphate

Speed and Time: 50 rpm and 60 minutes

Withdraw a suitable volume of the medium and filter

2.2 Solvent Mixture: 75 volumes of acetonitrile and 25 volumes of water

2.3 Test Solution:

Dilute the filtrate, if necessary, with a mixture of equal volumes of medium A and medium B.

2.4 Reference Solution:

A 0.005 percent w/v solution of dutasteride RS in the solvent mixture. Dilute 1.0 ml of this solution to 100 ml with a mixture of equal volumes of medium A and medium B.

2.4 Chromatographic system

Column: C18 (15 cm X 4.6 mm)

Flow rate: 1.5 ml/min

Wavelength: 215 nm

Injection volume: 100 µl

Column Temperature: 50˚ C

Detector: UV

Mobile Phase:

Mobile Phase A: 0.1% v/v orthophosphoric acid

Mobile Phase B: Acetonitrile

Gradient programme using the conditions given below,

Time (in min)	**Mobile Phase A (percent v/v)**	**Mobile Phase B (percent v/v)**
0	50	50
14	50	50

15	20	80
20	20	80
22	50	50

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 the reference solution and the test solution.

Calculate the content of Dutasteride.

2.6 Limit:

D. Not less than 80.0 percent of the stated amount of Dutasteride.

3. Uniformity of Content(if required):

Determine by liquid chromatography, as described in the Assay, using the following solution as the test solution.

3.1 Test Solution:

Weigh 10 tablets individually and place one tablet individually in 50 ml volumetric flask. Disperse in 12.5 ml of water, add 25 ml of acetonitrile and sonicate for 10 minute and dilute to 50 ml with acetonitrile.

4. Assay: Determine by liquid chromatography

4.1 Solvent Mixture:

75 volumes of acetonitrile and 25 volumes of water

4.2 Test Solution:

Place 5 intact tablets into 50 ml volumetric flask. Disperse in 12.5 ml of water, add 25 ml of acetonitrile and sonicate for 10 minute and dilute to 50 ml with acetonitrile.

4.3 Reference Solution:

A 0.005 percent w/v solution of dutasteride RS in the solvent mixture.

4.4 Chromatographic system

Column: C18 (15 cm X 4.6 mm)

Flow rate: 1.5 ml/min

Wavelength: 275 nm

Injection volume: 20 µl

Column temperature: 35˚ C

Detector: UV

Mobile Phase

Mobile PhaseA: 0.1% v/v orthophosphoric acid

Mobile Phase B: Acetonitrile

Gradient programme using the conditions given below

Time (in min)	**Mobile Phase A (percent v/v)**	**Mobile Phase B (percent v/v)**
0	45	55

10	45	55
11	20	80
15	20	80
16	45	55
22	45	55

4.5 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 the reference solution and the test solution.

Calculate the content of Dutasteride in the tablets.

5. Other tests: As per pharmacopoeial requirements.