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

Silodosin Capsules

Analytical Profile No.: SIL 074/075/AP 028

Silodosin Capsules contain not less than 90% and not more than 110% of the stated amount of Silodosin.

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 Silodosin.

2. Dissolution: Determine by liquid chromatography

Medium: 0.1 M HCl

Apparatus: Basket

Speed and time: 50 rpm for 30min

Volume: 500 ml

2.1 Chromatographic system:

Column: C 18, 250 X 4.6 mm

Flow rate: 1.0 ml/min

Wave length: 225 nm

Injection volume: 20 µl

Column Temperature: 35 ºC

Detector: UV

Diluent: 0.1 % Orthophosphoric acid: Methanol (6:4)

Buffer: 0.1 % Ortho Phosphoric Acid

Mobile phase: Buffer: Methanol: Acetonitrile (50:30:20)

Mix buffer and Acetonitrile and Methanol, cool to room temperature, adjust the pH of the solution to 3.5 with potassium hydroxide solution or dilute orthophosphoric acid and filter the solution through 0.45 micron Nylon membrane filter paper using vacuum pump.

2.2 Test Solution:

Filter the resulting solution if necessary.

2.3 Reference Solution:

Weigh accurately about 16 mg of working standard of Silodosin and transfer into 200 ml volumetric flask. Add about 100 ml of diluents and dissolve by sonicating for about 10 minutes and make up the volume to 200 ml with diluent. Dilute 2 ml of the resulting solution to 20 ml with diluents.

2.5 Procedure:

Inject 20 µl of standard preparation five/six times. 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 is not more than 2.0%.

Inject 20 µl of each of the sample solution separately. Measure the absorbance at 272.0 nm using dissolution medium as blank.

Calculate the percentage release of Silodosin.

2.8 Limit:

Not less than 75 % of the stated amount

3. Assay: Determine by Liquid Chromatography

3.1 Test Solution:

Take 20 capsules, determine the average fill weight. Weigh accurately the powder equivalent to 5 mg of Silodosin and transfer into 50 ml volumetric flask. Add about 35 ml of diluents, dissolve by sonicating for about 15 minutes and make up the volume to 50 ml with same diluents. Dilute 2 ml of the resulting solution to 20 ml with diluents. Filter through 0.22 micron nylon membrane filter paper.

3.2 Standard solution:

Weigh accurately about 25 mg of working standard of Silodosin and transfer into 100 ml volumetric flask. Add about 70 ml of diluents, dissolve by sonicating for about 15 minutes and make up the volume to 100 ml with same diluents. Dilute 2 ml of the resulting solution to 50 ml with diluents. Filter through 0.22 micron nylon membrane filter paper.

3.3 Chromatographic system:

Column:	250 X 4.6 mm (C 18)
Flow rate:	1.0 ml/min
Wave length:	225 nm
Detector:	UV
Injection volume:	20 µl
Column Temperature:	35 ºC
Buffer:	0.1 % Ortho Phosphoric Acid
Mobile phase:	Buffer: Methanol: Acetonitrile (50:30:20)

Mix buffer and Acetonitrile and Methanol, cool to room temperature, adjust the pH of the solution to 3.5 with potassium hydroxide solution or dilute orthophosphoric acid and filter the solution through 0.45 micron Nylon membrane filter paper using vacuum pump.

3.4 Procedure:

Inject 20 µl of standard preparation five/six times. 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%.

After the completion of the system suitability test parameter, inject 20 µl of each of the sample solution and chromatograph as per above mentioned chromatographic condition.

Calculate the content of Silodosin per capsule.

4. Uniformity of content

4.1 Perform the test as in Assay except for standard solution.

4.3 Standard Solution:

Place one capsule in a 100 ml volumetric flask; add about 70 ml of diluents. Dissolve by sonicating for about 15 minutes. Cool and make up the volume to 100 ml with diluent. Centrifuge or filter the resulting solution. Dilute 3 ml of the filtrate to 25 ml with diluents. Filter through 0.22 micron nylon membrane filter paper.

4.7 Other tests: As per pharmacopoeial requirement.