DEPARTMENT OF DRUG ADMINISTRATION

National Medicines Laboratory

ANALYTICAL METHOD VALIDATION COMMITTEE

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 test solution should correspond to the peak in the chromatogram obtained with the reference solution of Silodosin.

Tests:

2. Dissolution: *Determine by liquid chromatography*

2.1 Dissolution Parameters:

Apparatus: Basket

Medium: 500 ml 0.1 M HCl

Speed and time: 50 rpm for 30 min

Withdraw a suitable volume of the medium and filter.

2.2 Test Solution: Use the filtrate.

2.3 Reference Solution: Weigh accurately about 16 mg Silodosin reference standard 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.

2.4 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) adjust the pH of the solution to 3.5 with potassium hydroxide solution or dilute orthophosphoric acid

2.5 Procedure: Inject 20 µl of standard preparation five times. 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%. Inject 20 µl of each of the sample solution separately. Calculate the percentage release of Silodosin.

2.6 Limit: Not less than 75 % (D) of the stated amount.

3. Uniformity of content: *Proceed as directed under the Assay using the following solution as test solution.*

3.1 Test 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 0.2 micron membrane filter paper.

3.2 Limit: 85 % - 115 % of the stated amount.

4. Assay: *Determine by Liquid Chromatography*

4.1 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 0.2 micron membrane filter paper.

4.2 Reference solution: Weigh accurately about 25 mg Silodosin reference standard 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 0.2 micron membrane filter paper.

4.3 Chromatographic system:

Column: 250 X 4.6 mm (C 18)

Flow rate: 1.0 ml/min

Wavelength: 225 nm

Detector: UV

Injection volume: 20 µl

Column Temperature: 35 ºC

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

Buffer: 0.1 % Ortho Phosphoric Acid

Mobile phase: Buffer: Methanol: Acetonitrile (50:30:20) adjust the pH of the solution to 3.5 with potassium hydroxide solution or dilute orthophosphoric acid.

4.4 Procedure: Inject 20 µl of standard preparation five times. 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%. 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.

5. Other tests: As per Pharmacopoeial requirement.