ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT

DEPARTMENT OF DRUG ADMINISTRATION

Silodosin Capsules

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

1. Identification:

1.1 Silodosin

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 Test:

2.1 Dissolution Parameter:

2.1.1 Medium : 0.1 M HCl
2.1.2 Apparatus : Basket
2.1.3 Rotation : 50 RPM
2.1.4 Temperature : 37˚C ± 0.5˚C
2.1.5 Time : 30 minutes
2.1.6 Volume : 500 ml

2.1.7. Dissolution Medium Preparation: Dissolve 8.5 ml of hydrochloric acid to 1000 ml with water.

2.2 Chromatographic system:
2.2.1 Column: 250 X 4.6 mm (C 18)
2.2.2 Flow rate: 1.0 ml/min
2.2.3 Wave length: 225 nm
2.2.4 Injection volume: 20 µl
2.2.5 Column Oven Temperature: 35 ºC

2.3 Mobile Phase Preparation:
2.3.1 Buffer: 0.1 % Ortho Phosphoric Acid
2.3.2 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.

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

2.5 Standard Preparation:
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.

2.6 Sample Preparation

Place 1 tablet in each dissolution vessel and run the apparatus as per above condition and collect the sample solution from each jar at specified time. After the completion of the dissolution, filter the resulting solution.

2.7 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 separately. Calculate the percentage release of Silodosin in dissolution medium by using the following formula**:**

$$\frac{Peak\ Area\ of\ spl}{Peak\ Area\ of\ std} \times \frac{conc.of\ std}{conc.of\ spl} \times std\ potency\ \% \times \frac{100 - LOD/WC}{100}\ \ x\ \mathbf{100}\ \%$$

Result: Silodosin in %

2.8 Tolerence Limit: More than 75 % D of the stated amount

3. Assay: Silodosin Capsule

3.1 Chromatographic system:
3.1.1 Column: 250 X 4.6 mm (C 18)
3.1.2 Flow rate: 1.0 ml/min
3.1.3 Wave length: 225 nm
3.1.4 Injection volume: 20 µl
3.1.5 Column Oven Temperature: 35 ºC

3.2 Mobile Phase Preparation:
3.2.1 Buffer: 0.1 % Ortho Phosphoric Acid
3.2.2 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.

3.3 Standard Preparation: 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.

3.4 Sample Preparation: 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.

3.5 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 in not more than 2.0%. After the completion of the system suitability test parameter, inject 20 µl of each of the sample solution separately. Calculate the content of Silodosin in each tablet by using the following formula**:**

$$\frac{Peak\ Area\ of\ spl}{Peak\ Area\ of\ std} \times \frac{conc.of\ std}{conc.of\ spl} \times std\ potency\ \% \times \frac{100 - LOD/WC}{100}\ \ x\ \boldsymbol{Average\ wt.}$$

4.0 Uniformity of content

4.1 Mobile phase, Diluent, Standard solution & Chromatographic conditions – Same as Assay

4.2 Sample Preparation:

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.

4.3 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 10 µl of each of the sample solution separately. Calculate the content of Illaprazole in each tablet by using following formula:

Silodosin per capsule:

$$\frac{\boldsymbol{Spl\ Peak\ Area}}{\boldsymbol{Std\ Peak\ Area}}\ \boldsymbol{X}\ \frac{\boldsymbol{Std\ wt}}{\boldsymbol{Std\ Dil.}}\ \boldsymbol{X}\ \frac{\boldsymbol{Spl\ Dil.}}{\boldsymbol{Spl\ wt}}\ \boldsymbol{X\ Avg\ wt\ X\ Std\ Pot.}\ (\%)\boldsymbol{X}\ \frac{\boldsymbol{100 - Std\ W.C}}{\boldsymbol{100}}\boldsymbol{X}\frac{\boldsymbol{1}}{\boldsymbol{Label\ Claim}}\boldsymbol{X}\ \boldsymbol{100}\%$$