ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT

DEPARTMENT OF DRUG ADMINISTRATION

Itopride HCl Tablet

Itopride HCl Tablet contain not less than 90 % and not more than 110 % of the stated amount of Itopride HCl.

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 Itopride.

2. Assay:

2.1 Reagents Required:

1. Acetonitrile (HPLC Grade)
2. Orthophosphoric acid (HPLC Grade)
3. Triethylamine
4. HPLC Grade water

2.2 Preparation of reagent solution:

2.2.1 Buffer: Prepared by adding 1 ml of Orthophosphoric acid in 1000 ml water, adjust pH to 3.0 ± 0.05 with Triethylamine

2.2.2 Mobile phase: Buffer: Acetonitrile (70:30)

2.3 Chromatographic system

Column: Octyldecylsilane (C18), (250*4.6 mm), 5 µm

Flow rate: 1.0 ml/min

Detector: UV Detector

Wavelength: 220 nm

Injection volume: 20 µl

Oven temperature: 30 °C

2.4 Standard Solution:

Weigh accurately about 20 mg of Itopride HCl RS and transfer in 100 ml volumetric flask. Add 70 ml mobile phase and sonicate for about 10 min and make volume to 100 ml with same solvent. Centrifuge or filter the solution. Dilute 5 ml of the resulting solution to 50 ml with same solvent. Filter the resulting solution through 0.2 micron nylon membrane filter. (20 ppm)

2.5 Sample Preparation:

Weigh and powder 20 tablets. Weigh powder eq. to 50 mg of Itopride HCl, dissolve with mobile phase by sonicating for about 10 minutes and make the volume to 100 ml with same solvent. Filter or centrifuge the resulting solution and dilute 2 ml of the filtrate to 50 ml with same solvent. Filter the resulting solution through 0.2 micron nylon membrane filter. (20 ppm)

2.6 Chromatographic Procedure:

Inject 20 µl of standard and sample solution separately and obtain the respective chromatogram. Measure the peak responses.

2.7 System suitability:

Inject 20 µl of standard solution and sample solution of Itopride as per above mentioned chromatographic condition. In the chromatogram obtained from the standard preparation, the column efficiency determined from the major peak should not be less than 2000 theoretical plates, the tailing factor should be not more than 2.0 and the relative standard deviation of five replicate injections should not more be than 2.0 %. Inject 20 µl of the sample preparation and chromatograph as per above mentioned chromatographic condition. Calculate the result from the formula given below.

2.8 Calculations:

Content of Itopride HCl per Tablet:

$$\frac{\textbf{Spl Peak Area}}{\textbf{Std Peak Area}} \textbf{ X } \frac{\textbf{Std wt}}{\textbf{Std Dil.}} \textbf{ X } \frac{\textbf{Spl Dil.}}{\textbf{Spl wt}} \textbf{ X Average wt X Std Potency } (\%) \textbf{X} \frac{(\textbf{100} - \textbf{Std LOD})}{\textbf{100}}$$

3.0 Dissolution:

3.1 Dissolution Medium: 0.1N Hydrochloric Acid

3.2 Dissolution test condition

Volume : 900 ml

Apparatus : 1 (Paddle)

RPM : 50

Time : 30 minutes

3.3 Standard Solution:

Weigh accurately about 20 mg of Itopride HCl RS in 100 ml volumetric flask and add dissolution medium in it. Sonicate for about 10 min and make volume with same solvent. Dilute 5 ml of the filtrate to 100 ml with same solvent. (10 ppm)

3.4 Sample Solution:

Transfer one tablet into each six dissolution vessels. Precede the test as prescribed in the Dissolution test condition. Withdraw a suitable volume of sample from the dissolution vessel after completion of test, filter the solution. Dilute 5 ml of this solution to 25 ml with dissolution medium.

Measure the absorbance of both standard and sample solution at about 258 nm taking dissolution medium as blank. Calculate the % release by comparison.

3.5 Calculation:

$$\%\ \boldsymbol{release} = \frac{\boldsymbol{Spl\ Absorbance}}{\boldsymbol{Std\ Absorbance}} \boldsymbol{X} \frac{\boldsymbol{Std\ wt}}{\boldsymbol{Std\ Dil}} \boldsymbol{X} \frac{\mathbf{900}}{\boldsymbol{Label\ Claim}} \boldsymbol{X} \frac{\mathbf{25}}{\mathbf{5}} \boldsymbol{x Std.\ Potency}\ (\%) \boldsymbol{X} \frac{\mathbf{100} - \boldsymbol{LOD}}{\mathbf{100}} \boldsymbol{X}\ \mathbf{100}\ \%$$

3.6 Tolerance Limit: NLT 75% D of the stated amount

Note

1. Weight variation and friability test should be as per the Pharmacopoeia recognized by drug advisory committee.