ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT

DEPARTMENT OF DRUG ADMINISTRATION

Itopride Sustained Release Capsules

Itopride Sustained Release Capsules 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 2 ml of Orthophosphoric acid in 1400 ml water, adjust pH 3.0 ± 0.05 with Triethylamine

2.2.2 Mobile phase: Buffer: Acetonitrile (1400:600 v/v)

2.3 Chromatographic system

Column: Octyldecylsilane (C18), (150*4.6 mm), 5 µm

Flow rate: 1.0 ml/min

Detector: UV Detector

Wavelength: 258 nm

Injection volume: 20 µl

Oven temperature: 30 °C

2.4 Standard Solution:

Weigh about 33 mg of Itopride RS in 50 ml volumetric flask and add mobile phase in it. Sonicate for about 10 min and make volume with mobile phase. Dilute 2 ml of the filtrate to 50 ml with mobile phase.

2.5 Sample Preparation:

Weigh individually 20 tablets & crush the tablet into fine powder. Weigh powder eq. to 5 g of Itopride pellets in a mortar and pestle and crush. Transfer about 50 mg of powder into 50 ml volumetric flask and add mobile phase in it. Sonicate for about 15 min and make volume with mobile phase. Filter and dilute 2 ml of the filtrate to 50 ml with mobile phase.

Note: Adjust the concentration of the standard preparation and sample preparation depending upon the strength of Itopride HCl capsules.

2.6 Chromatographic Procedure:

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

2.7 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 : 2 (basket)

RPM : 100

Time : 2nd hr, 6th hr and 10th hr

3.3 Standard Solution:

Weigh about 25 mg of Itopride RS in 50 ml volumetric flask and add mobile phase in it. Sonicate for about 10 min and make volume with mobile phase. Dilute 5 ml of the filtrate to 100 ml with mobile phase.

3.4 Sample Solution:

Transfer one capsule into each vessel and run the apparatus for 10 hours. After completion of the specific time interval (2nd hr, 6th hr and 10th hour) samples are taken by auto sampler. Dilute 5 ml of this solution to 25 ml with mobile phase.

3.5 Mobile Phase: Same as Assay

3.6 Chromatographic System: Same as Assay

3.7 Chromatographic Procedure:

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

3.8 Calculation:

$$\%\ release = \frac{Spl\ Peak\ Area}{Std\ Peak\ Area} X \frac{Std\ wt}{Std\ Dil} X \frac{1000}{Label\ Claim} X \frac{100}{1} x Std.Potency\ (\%) X \frac{100 - LOD}{100} X\ 100\ \%$$

3.9 Tolerance Limit: 2nd hr. (NLT 25% and NMT 55% of the stated amount)
6th hr. (NLT 55% and NMT 85% of the stated amount)
10th hr. (NLT 75% of the stated amount)