ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT

DEPARTMENT OF DRUG ADMINISTRATION

Tapentadol Capsules

Tapentadol Capsules contain not less than 90 % and not more than110 % of the stated amount of Tapentadol.

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 Tapentadol.

2. Assay:

2.1 Reagents Required:

1. Hydrochloric acid (AR Grade)
2. Methanol (HPLC Grade)
3. Triethylamine (HPLC Grade)
4. Orthophosphoric acid (HPLC Grade)
5. HPLC Grade water
6. Potassium Dihydrogen orthophosphate (AR Grade)

2.2 Preparation of reagent solution:

2.2.1 Buffer solution: Dissolve 2.72 gm of potassium dihydrogen orthophosphate in 1000 ml of water: add 2 ml of triethylamine, and mix. Adjust the pH to 2.5 with orthophosphoric acid.

2.2.2 Mobile phase: Prepare a suitable mixture of Buffer and methanol (75:25), and pass through a membrane filter having a 0.45 µm or finer porosity.

2.3 Chromatographic system

Column: Octylsilane (C8), (150*4.6 mm), 5 µm

Flow rate: 1.0 ml/min

Detector: UV Detector

Wavelength: 215 nm

Tapentadol Capsules

Injection volume: 20 µl

Oven temperature: 40 °C

2.4 Standard Solution:

Weigh accurately about 25 mg of working standard of Tapentadol hydrochloride and transfer into 25 ml volumetric flask. Dissolve with mobile phase and make up the volume to 25 ml with mobile phase. Dilute 5 ml of the resulting solution to 25 ml with mobile phase. Filter the resulting solution through 0.2 µm membrane filter paper.

2.5 Sample Preparation: Weigh individually 20 capsules & remove the content of each capsule. Mix the content of all the capsules. Weigh powder eq. to 50 mg of tapentadol and transfer into 50 ml volumetric flask. Add about 70 ml of mobile phase and dissolve by sonicating for about 10 minutes. Filter the resulting solution and dilute 5 ml of the filtrate to 25 ml with mobile phase. Again filter the resulting solution through 0.2 µm membrane filter paper.

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 20µl of standard solution of tapentadol 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 replicate injections should not more be than 2.0 %. Inject 20µl 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 Tapentadole per Tablet:

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

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3.0 Dissolution (Tapentadol 100 mg):

3.1 Dissolution Medium: 0.1 M Hydrochloric acid

3.2 Dissolution test condition

Volume : 900 ml
Apparatus : 2 (Basket)
RPM : 75
Time : 60 minutes

3.3 Standard Solution:

Weigh accurately about 27.5 mg of working standard of Tapentadol hydrochloride and transfer into 100 ml volumetric flask. Dissolve with dissolution medium and make up the volume to 100 ml with dissolution medium. Dilute 5 ml of the filtrate to 25 ml with dissolution medium.

3.4 Sample solution:

Place the stated volume of dissolution medium, free from dissolved air, into the vessel of the apparatus. Assemble the apparatus and warm the dissolution medium to 36.5 º to 37.5 º. Operate the apparatus immediately at the time and speed as specified. Within the time interval specified, or at each of the times stated, withdraw a specimen from a zone midway between the surface of the dissolution medium and top of the rotating blade. Filter the sample solution promptly through filter paper or SS filter. Discard the first few ml of the filtrate. Dilute 10 ml of this solution to 20 ml with dissolution medium.

3.5 Empty shell solution: To one of the dissolution vessel, add an empty capsule shell, proceed the test similar to the sample, filter and collect the filtrate. Dilute 10 ml of this solution to 20 ml with dissolution medium.

3.6 Procedure:

Measure the absorbance at 272.0 nm using dissolution medium as blank. Subtract the absorbance value of empty capsule shell from the test sample solution. Calculate the content of release of tapentadol in each tablet by using the following formula:

3.7 Calculation:

$$\%\ release = \frac{Spl\ Peak\ Area.}{Std\ Peak\ Area}\ X\ \frac{Std\ wt}{Std\ Dil}\ X\ \frac{900}{Label\ Claim}\ X\ \frac{20}{10}\ x\ Std.\ Potency\ (\%) X\ \frac{100 - LOD}{100}\ X\ 100\ \%$$

3.8 Tolerence Limit: D. NLT 80 % of the stated amount