DEPARTMENT OF DRUG ADMINISTRATION
National Medicines Laboratory
ANALYTICAL METHOD VALIDATION COMMITTEE

Analytical profile of Ticagrelor Tablets

Ticagrelor Tablets contains not less than 90.0% and not more than 110.0% of the stated amount of Ivabradine.

Usual Strength: Ticagrelor 60 mg, 90 mg

1. Identification:

In the Assay, the principle peak in the chromatogram obtained with the test solution corresponds to the peak in the chromatogram obtained with the reference solution.

Tests:

2. Dissolution: *Determine by liquid chromatography*

2.1 Dissolution Parameters:

Apparatus: Paddle

Medium: 900 ml of 0.2 % w/v polysorbate-80 in water

Speed and Time: 75 rpm and 45 minutes

Withdraw a suitable volume of the medium and filter.

Determine by liquid chromatography.

2.2 Test Solution: Use the filtrate, dilute if necessary with dissolution medium.

2.3 Reference Solution: Weigh accurately and transfer about 50 mg of Ticagrelor WS in to a 100 ml volumetric flask. Add about 70 ml solvent mixture, sonicate to dissolve, cool to room temperature and make up the volume with same solvent. Dilute 5 ml of the solution to 25 ml with dissolution medium and mix well.

2.4 Procedure: Use the chromatographic system as described in the Assay. Inject the reference solution and the test solution using injection volume of 10 µl.

2.5 Limit: Not less than 70 percent (D) of the stated amount of Ticagrelor.

3. Assay: *Determine by liquid chromatography*

3.1 Solvent Mixture: A solution containing equal volumes of Solution A and Acetonitrile

3.2 Test Solution: Weigh individually 20 tablets and crush the tablet into fine powder. Weigh accurately powder equivalent to 25 mg of Ticagrelor in 100 ml volumetric flask, add about 70 ml

of solvent mixture, sonicate to dissolve, cool and make up the volume to 100 ml with same solvent.

3.3 Reference Solution: Weigh accurately about 25 mg Ticagrelor RS into 100 ml volumetric flask. Add about 70 ml of solvent mixture and sonicate to dissolve, cool and make up the volume to 100 ml with same solvent.

3.4 Chromatographic system:

- **Column:** 15 cm x 4.6 mm, 5µ particle size, packed with phenyl group bonded to porous silica
- **Flow rate:** 1.0 ml per minute
- **Wavelength:** 270 nm
- **Injection volume:** 5 µl
- **Detector:** UV/PDA
- **Column temperature:** ambient
- **Mobile Phase:** A mixture containing 45 volumes of Solution A and 55 volumes of Solution B
- **Solution A:** A buffer solution prepared by dissolving 1.2 g of sodium dihydrogen phosphate in 1000 ml of water, adjusted to pH 3.0 with orthophosphoric acid.
- **Solution B:** A solution containing equal volumes of Acetonitrile and Methanol.

3.5 Procedure: Inject the reference solution. 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%.
Calculate the content of Ticagrelor in the tablet.

4. Other tests: As per pharmacopoeial requirement.