ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT

DEPARTMENT OF DRUG ADMINISTRATION

National Medicines Laboratory

Empagliflozin Tablets

Analytical Profile No.: EMPA075/076/AP044

Empaglifozin Tablet contains 90% to 110% of empaglifozin of stated amount.

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 Empagliflozin.

2. Dissolution:

2.1 Dissolution Parameters

Apparatus: Paddle

Medium: 900ml of pH 6.8 phosphate buffer

Speed and Time: 75rpm for 45min

2.2 Chromatographic system:same as assay

2.3 Mobile Phase: same as assay

Buffer:0.01M Potassium Dihydrogen Phosphate buffer, pH4.0

2.4 Test Solution:

Withdraw a suitable volume of the sample after 45 minutes. Dilute the filtrate, if necessary, with the dissolution medium. Filter the final solution through 0.2 µm membrane filter.

2.4 Standard Solution:

Weigh accurately about 25 mg Empagliflozin WS in 100 ml volumetric flask. Add 5 ml methanol, sonicate for 2 min to dissolve and make up the volume to 100 ml with dissolution medium. Further dilute 2 ml of this solution to 50 ml with dissolution medium. Filter the final solution through 0.2 µm membrane filter.

2.5 Procedure:

Proceed the process as described in assay methodand obtain the respective chromatograms. Measure the peak responses and calculate the % release of the drug.

2.6 Limit:

D.NLT 75% of the stated amount

3.0 Uniformity of Content (if required):

3.1 Chromatographic condition: same as assay

3.2 Procedure:

Weigh 10 tablets individually and put each in volumetric flask. Proceed the process as described in assay methodexcept for test solution.

3.3 Test Solution:

Place a tablet in a 50ml volumetric flask, add 30ml of diluents, sonicate for 15 minutes. Cool and make up the volume to 50ml with diluents. Filter it through 0.2 µm membrane filter.

4. Assay:

4.1Chromatographic system:

Column:	C18, 150*4.6 mm, 5 µm
Flow rate:	1.5 ml/min
Wavelength:	227nm
Injection volume:	20 µl
Column Temperature:	30°C
Detector:	UV
Solvent Mixture	Equal volume of water and methanol
Mobile Phase:	Methanol: Buffer (50:50)

4.2Standard Solution:

Weigh accurately about 25 mg EmpagliflozinWSin 100 ml volumetric flask. Add about 70 ml of diluents and sonicate for about 10 minutes to dissolve and make up the volume to 100 ml with same solvent. Filter the solution through 0.2 µm membrane filter.

4.3Test Solution:

Weigh individually 20 tablets& crush the tablet into fine powder. Weigh a quantity of powder equivalent to 25 mg of Empagliflozin in 100ml volumetric flask, add 70 ml of diluents, sonicate to dissolve with intermittent shaking and make volume to 100 ml with same solvent.Filter the final solution through 0.2 µm membrane filter.

4.4Procedure: Inject the reference solution five/six times and sample solutions. 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 isnot more than 2.0%. Measure the peak responses.Calculate the content of Empaglifozin per tablet.

4.5 Other Tests: As per pharmacopoieal requirement.