Department of Drug Administration
National Medicines Laboratory
ANALYTICAL METHOD VALIDATION COMMITTEE FOR NON PHARMACOPOEIAL PRODUCT

Amlodipine and Atorvastatin Tablet

Analytical Profile No.: AmloAtor 073/074/AP 006

Amlodipine and Atorvastatin Tabletscontain not less than 90% and not more than 110% of the stated amount of Amlodipine andAtorvastatin.

1. Identification:

1.1. Amlodipine Besylate:

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.

1.2. Atorvastatin:

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.

2. Dissolution Test

2.1:Atorvastatin

2.1.1 Dissolution Parameter: Determine by liquid chromatography

Apparatus: Paddle

Medium: 900ml of Phosphate buffer pH 6.8

Speed and Time: 75 RPM &30 minutes

Temperature: 37+/-0.5℃

Withdraw a suitable volume of the medium and filter

2.1.2 Test Solution:

Dilute the filtrate, if necessary, with dissolution medium.

2.1.3 Reference Solution: Weigh accurately 20 mg of Atorvastatin calcium WS and trasfer in 100 ml of voumetric flask, dissolve it with methanol and make up the volume with same solvent and sonicate for 5 minutes. Dilute 5 ml of the resulting solution to 100 ml with dissolution medium.

2.1.4 Procedure:

Use the chromatographic system as described in the Assay.

Inject the reference solution and the test solution.

Calculate the % release of Atorvastatin.

2.1.6 Limit:

D. Not less than 70 % of the stated amount of Atorvastatin.

2.2. Amlodipine Besylate

2.2.1 Dissolution Parameter: Determine by liquid chromatography

Apparatus: Paddle

Medium: 900ml of 0.01 N HCL

Speed and Time: 75 RPM & 45 minutes

Temperature: 37+/-0.5℃

Withdraw a suitable volume of the medium and filter

2.2.2 Test Solution:

Dilute the filtrate, if necessary, with dissolution medium.

2.2.3 Reference Solution: Weigh accurately about 15 mg of Amlodipine Besylate and transfer in 100 ml volumetric flask, add about 70 ml of methanol and sonicate for 5 minutes, cool to room temperature and make up the volume to 100 ml with the methanol. Dilute 2 ml of the resulting solution to 50 ml with the dissolution medium.

2.2.4 Procedure:

Use the chromatographic system as described in the Assay except injection volume of 30 µl.

Inject the reference solution and the test solution.

Calculate the % release of Amlodipine.

2.2.6 Limit:

D. Not less than 70 % of the stated amount of Amlodipine.

3.0 Uniformity of content (if required):

Determine by liquid chromatography, as described in the Assay, using the following solution as the test solution.

3.1 Test Solution:

Weigh 10 tablets individually and transfer each into 100 ml volumetric flask.Add about 70 ml of mobile phase and dissolve by sonicating for about 10 minutes, cool and make up the volume to 100 ml with same solvent.

3.2 Uniformity of Content:

85-115% of the stated amount

4.0 Assay: Amlodipine and Atorvastatin

4.1Test Solution: Weigh 20 tablets individually and crush 20 tablets. Weigh accurately the powder of the sample equivalent to 5 mg of Amlodipine or 10 mgofAtorvastatin in 100 ml of volumetric flask. Add about 70 ml of mobile phase and dissolve by sonicating for about 10 minutes, cool and make up the volume to 100 ml with same solvent.

4.2Reference Solution: Weigh accurately about 25 mg of Amlodipine Besylate WS and 50 mg of Atorvastatin calcium WS in a 100 ml volumetric flask. Add about 60 ml of mobile phase and dissolve by sonicating for about 5 minutes, allow cooling at room temperature and make up the final volume with same solvent. Dilute 5 ml of the resulting solution to 50 ml with mobile phase.

4.3 Chromatographic system:

- Column: C18, (250mm x 4.6mm), 5 micron
- Injection volume: 20 µl
- Flow rate: 1.0 ml / min.
- Wave length: 230 nm
- Column Temperature: 35 °C
- Detector: UV

  Mobile Phase: A mixture of 40 volume of buffer prepared by dissolving 1.54g of ammonium acetate in 1000 ml of water and pH adjusted to 4.0 with acetic acid, 40 volume of methanol and 20 volume of Acetonitrile

4.4 Procedure:

Inject the reference solution. The test is not valid unless the column efficiency is not less than 2000 theoretical plates; the tailing factor is not more than 2.0 and the relative standard deviation for replicate injections in not more than 2.0%.

Calculate the content of Amlodipine & Atorvastatin per tablet.

5. Other tests: As per pharmacopoeial requirements.