DEVELOPMENT OF TEST METHOD FOR AMLODIPINE AND VALSARTAN TABLET

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Abstract

Product Name:Amlodipine + Valsartan Tablet (5mg + 160mg, 5mg + 320mg, 10 mg + 160mg, 10mg + 320mg)
Test:Assay of Amlodipine + Valsartan tablet (By HPLC)

Use the standard as such and use % potency on as is basis for calculations. Keep The container tightly closed. Preserve in well – closed, light – resistant containers, And store at controlled room temperature.

Keywords-Amlodipine ,Valsartan ,HPLC.Assay

Valsartan working standard:

Use the standard as such and use % potency on as is basis for calculations. Keep The container tightly closed. Preserve in well – closed, light – resistant Containers, and store at controlled room temperature.

1.0 **Reagents:**

Water (Milli Q or equivalent)

Ortho Phosphoric Acid 88% (GR Grade, Merck)

Methanol (HPLC Gradient Grade, Merck)

Acetonitrile (HPLC Gradient Grade, Rankem)

Triethylamine (Chromatography Grade, Merck)

2.0 Preparation of Buffer pH 3.0:

Pipette out 7 mL of Triethyl amine and transfer into 1000 mL water. Adjust the pH to 3.0 with Ortho Phosphoric acid. Filter through 0.45µ nylon Membrane filter.

3.0 Preparation of Mobile Phase:

Prepare a mixture of Buffer: Acetonitrile: Methanol in the ratio 52:15:33 v/v. Mix and Degas.

4.0 Preparation of Standard Solution:

4.1 Preparation of Amlodipine standard stock solution:

Weigh accurately about 70 mg of Amlodipine besylate working standard and transfer it into 50mL amber colored volumetric flask. Add about 30 mL of mobile phase and sonicate to dissolve. Make up to the mark with mobile phase and mix.

4.2 Preparation of Valsartan standard stock solution:

Weigh accurately about 50 mg of Valsartan working standard and transfer it into a 50mL amber colored volumetric flask. Add about 30mL of mobile phase and sonicate to dissolve. Make up to the mark with mobile phase and mix.

4.3 **Preparation of Standard solution:**

Dilute 4 mL of amlodipine standard stock solution + 5 mL of Valsartan standard stock solution in 100 mL amber colored volumetric flask. Make up to the mark with mobile phase and mix.

5.0 Preparation of Sample Stock Solution:

5.1 For 5mg + 160 mg:

Transfer 10 tablets of Amlodipine + Valsartan into 250mL amber colored volumetric flask.Add about 200 mL mobile phase and sonicate for 15 minutes with intermittent shaking. Check whether all tablets are dispersed; if not then allow it for more sonication for 5 min. Cool to room temperature and then make up to the mark with mobile phase.

5.2 For 10mg + 160 mg:

Transfer 10 tablets of Amlodipine + Valsartan into 250mL amber colored volumetric flask. Add about 200 mL mobile phase and sonicate for 15 minutes with intermittent shaking. Check whether all tablets are dispersed; if not then allow it for more Sonication for 5 min. Cool to room temperature and then make up to the mark with mobile phase.

5.3 For 5 mg + 320 mg:

Transfer 10 tablets of Amlodipine + Valsartan into 500 mL amber colored volumetric flask. Add about 400 mL mobile phase and sonicate for 15 minutes with intermittent shaking. Check whether all tablets are dispersed; if not then allow it for more sonication for 5 min. Cool to room temperature and then make up to the mark with mobile phase.

5.4 For 10mg + 320 mg:

Transfer 10 tablets of Amlodipine + Valsartan into 500 mL amber colored volumetric flask. Add about 400 mL mobile phase and sonicate for 15 minutes with intermittent shaking. Check whether all tablets are dispersed; if not then allow it for more sonication for 5 min. Cool to room temperature and then make up to the mark with mobile phase.

6.0 Preparation of Amlodipine Sample solution:

6.1 For 5mg + 160 mg, 10 mg+ 320 mg

Transfer 10 mL of sample stock solution into 50 mL amber colored volumetric flask and make up to the mark with mobile phase and mix. Filter it through 0.45µ Teflon + Glass membrane syringe filter.

6.2 For 10 mg + 160 mg

Transfer 5 mL of sample stock solution into 50 mL amber colored volumetric flask and make up to the mark with mobile phase and mix. Filter it through 0.45µ Teflon + Glass membrane syringe filter.

6.3 For 5 mg+320 mg

Transfer 20 mL of sample stock solution into 50 mL amber colored volumetric flask and make up to the mark with mobile phase and mix. Filter it through 0.45µ Teflon + Glass membrane syringe filter.

7.0 Preparation of Valsartan Sample solution:

Transfer 4mL of sample stock solution into 100mL amber colored volumetric flask and make up to the mark with mobile phase and mix. Further take 10 mL of this solution into 50 mL amber colored volumetric flask and make upto the mark with mobile phase. Filter it through 0.45µ Teflon + Glass membrane

8.0 Chromatographic Condition:

Column	:	Akzo Noble, Kromasil C_{18}, 50 x 4.6 mm, 5µm or equivalent
Flow Rate	:	2.0 mL/min
Injection Volume	:	30µl for Amlodipine & Valsartan
Detection	:	237nm for Amlodipine
	:	248nm for Valsartan
Column Temp.	:	30°C
Sample Temp.	:	15°C
Run Time	:	15 minutes

9.0 Evaluation of System Suitability:

Inject the standard solution five times. The relative standard deviation of five replicate injections should not be more than 2.0%. The USP tailing factor for Valsartan peak and Amlodipine peak should not be more than 2.0.

10.0 Procedure:

Separately inject equal volumes of Blank and Sample preparation (in duplicate). The retention time of Amlodipine is about 2.5 minutes and that of Valsartan is about 7.0 minutes.

11.0 Calculation:

Calculate the amount of Amlodipine present in the tablets as per given formula:

For Amlodipine 5 mg +160 mg:

$$\%\ \text{Assay} = \frac{AT}{AS} \times \frac{WS}{50} \times \frac{4}{100} \times \frac{250}{10\ tab} \times \frac{50}{10} \times \frac{P}{100} \times \frac{100}{LC} \times 0.7211$$

For Amlodipine 10 mg +320 mg :

$$\%\ \text{Assay} = \frac{AT}{AS} \times \frac{WS}{50} \times \frac{4}{100} \times \frac{500}{10\ tab} \times \frac{50}{10} \times \frac{P}{100} \times \frac{100}{LC} \times 0.7211$$

For Amlodipine 5 mg + 320 mg :

$$\%\ \text{Assay} = \frac{AT}{AS} \times \frac{WS}{50} \times \frac{4}{100} \times \frac{500}{10\ tab} \times \frac{50}{20} \times \frac{P}{100} \times \frac{100}{LC} \times 0.7211$$

For Amlodipine 10 mg +160 mg :

$$\%\ \text{Assay} = \frac{AT}{AS} \times \frac{WS}{50} \times \frac{4}{100} \times \frac{250}{10\ tab} \times \frac{50}{5} \times \frac{P}{100} \times \frac{100}{LC} \times 0.7211$$

11.1 Calculate the amount of Valsartan present in the tablets as per given formula:

For Valsartan 160 mg:

$$\%\ \text{Assay} = \frac{AT}{AS} \times \frac{WS}{50} \times \frac{5}{100} \times \frac{250}{10\ tab} \times \frac{100}{4} \times \frac{50}{10} \times \frac{P}{100} \times \frac{100}{LC}$$

For Valsartan 320 mg:

$$\%\ \text{Assay} = \frac{AT}{AS} \times \frac{WS}{50} \times \frac{5}{100} \times \frac{500}{10\ \text{tab}} \times \frac{100}{4} \times \frac{50}{10} \times \frac{P}{100} \times \frac{100}{LC}$$

Where,

AT	:	Area of peak due to Active Ingredient in sample preparation.	Avg. area count of Acti of sample solution.
AS	:	Mean Area of peak due to Active Ingredient in standard preparation.	Avg. area count of Acti of standard solution.
WS		Weight of Active Ingredient standard in mg.	
P	:	Label claim of Active Ingredient in mg.	% Potency of Active In basis.
LC	:	Potency of Active Ingredient standard on as is basis.	Label Claim of Active In