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**COLORIMETRIC DETERMINATION AND VALIDATION OF AMLODIPINE  
BESYLATE IN PURE AND TABLET DOSAGE FORM**

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**ABSTRACT**

Amlodipine besylate is a commonly used antihypertensive drug acting as calcium antagonist. In this study, a colored ion-pair complex formation reaction among Amlodipine and acid-dye bromothymolblue blue at pH 6.0 was used for the colorimetric determination of the drug. The complex formed was extracted into chloroform and the maximum absorbance of the solution was measured at 414 nm against blank. The calibration curve calculated obeys Beer's law over the concentration range of 1-3 $\mu$ g/ml and the regression equation was  $A=0.299x+0.010$  ( $r=0.998$ ). The recovery of the drug from a commercial tablet was 104.6 % of the label claim with a relative standard deviation of 0.21 %. The results were compared with those of the spectrophotometric method currently used by the manufacturer of the tablets and no significant difference was found.

**KEYWORDS**

Arnlodipine, Bromothymolblue and Ion-pair complex.

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**INTRODUCTION**

Amlodipine besylate (ADB) (Figure No.1) is a calcium channel blocking agent with vasodilators activity similar to that of nifedipine. It is mainly used for its antiarrhythmic, antianginal and antihypertensive activity. It is chemically known as 2-[(2 aminoethoxy) methyl]-4-(2-chloroprienyl)-1, 4-dihydro-6-methyl-3,5-pyridine dicarboxylic acid, 3-ethyl, 5 methylesterbesylate. It has been determined in pharmaceutical formulations by

Spectrophotometry in human plasma by HPLC and used the instrumental methods of choice in industrial laboratories for quality control of the drugs and pharmaceutical formulations, a simple and specific colorimetric method based on ion-pair complex formation among the drug and bromothymolblue (BTB) has been proposed<sup>6-12</sup>.

## **MATERIAL AND METHOD**

### **Experimental**

A Labindia Visible Spectrophotometer/ colorimeter with 1 cm matched quartz cells was used for all spectral measurements. All chemicals used were of A.R.grade from S.D. Fine-chem, Merck, Fischer scientific, and Spectrochem, Mumbai. Authentic drug sample of Amlodipine besylate was given as a gift sample by Hetero drugs Ltd., Hyderabad. Tablets of Amlodipine besylate were procured from local market.

### **SOLUTIONS**

#### **Reagent solution**

Bromothymolblue.

#### **Buffer solution: (pH- 6)**

Dissolve 100mg of ammonium acetate in 300ml of water and add 4.1ml of glacial acetic acid adjust the pH if necessary using 10M ammonia {or}5M acetic acid and dilute water with to 500ml.

#### **Stock solution**

Stock Solution of Amlodipine besylate: 50 mg of ADB was weighed accurately into a 100-ml volumetric flask. 5 ml of ethanol and 10 ml of water were added and the mixture was shaken until ADB was dissolved completely. Then the solution was diluted to 100 ml with water (500µg/ml).

### **RECOMMENDED PROCEDURE**<sup>13-17</sup>

1-3 ml of standard solution of ADB was transferred to stoppered 25-ml test tubes. 1.0 ml of buffer

LC<sup>1-6</sup>. Since spectrophotometric methods have been solution and 1.0 ml of BTB solution were added to each tube and the tubes were shaken for 5 min. 15ml aliquots of chloroform were added to the tubes and the mixtures were shaken for 2 min and allowed to stand for 5 min for separation of the chloroform layer. The absorbance of the chloroform phase was measured after an equilibrium time of 10 min in 1-cm quartz cells at 414 nm against blank solution, which was prepared similarly calibration curve, was plotted using absorbance values concentration.

### **Assay procedure for tablets**

Twenty tablets were weighed and pulverized to a fine powder. An aliquot equivalent to about 50 mg of ADB was transferred into a 100-ml volumetric flask. A suspension of the drug with 5 ml ethanol and 50ml water was shaken for 10 min and filtered to a second 100-ml volumetric flask. The first flask was rinsed with 3x10 ml water, which were transferred through the same filter paper. Final solution was diluted to 100 ml with water. 1.5 ml of the filtrate was preceded as stated in recommended procedure.

## **RESULTS AND DISCUSSION**

The colorimetric determination of Amlodipine (Figure No.2 and 3) was based on the absorbance measurement of the yellow ion-pair complex at 414 nm (Table No.1). The formation of the colored complex is based on the basic nature of the Amlodipine, which forms at pH 6 under specified experimental conditions an ion-association complex with the acid-base indicator, bromothymolblue. The optimum conditions for formation of the colored complex with respect to the pH of the aqueous phase and volume of the reagent added were investigated (Table No.2). Proper extraction solvent for the complex was also investigated.

**Table No.1: Results of calibration curve at 414 nm for Amlodipine besylate**

S.No	Concentration (mcg / ml)	Absorbance at 414 nm
1	1	0.308
2	1.5	0.475
3	2	0.625
4	2.5	0.754
5	3	0.895

**Table No.2: Optimum conditions, Optical characteristics and Statistical data of the Regression equation in UV method**

S.No	Parameter	UV method
1	$\lambda_{\max}$ (nm)	414
2	Beer's law limits (mcg/ml)	1-10
3	Regression equation (Y*)	Y = 0.0299X + 0.010
4	Slope (b)	0.0299
5	Intercept (a)	+ 0.010
6	Correlation coefficient( $r^2$ )	0.998
7	% RSD**	< 2%

**Table No.3: Assay of Amlodipine besylate formulation**

S.No	Formulation	Label claimed (mg/tab)	Amount found (mg) (n=10) Mean $\pm$ SD	Assay	%RSD
1	Norvas	5	137 $\pm$ 0.000983	104.6%	0.2109

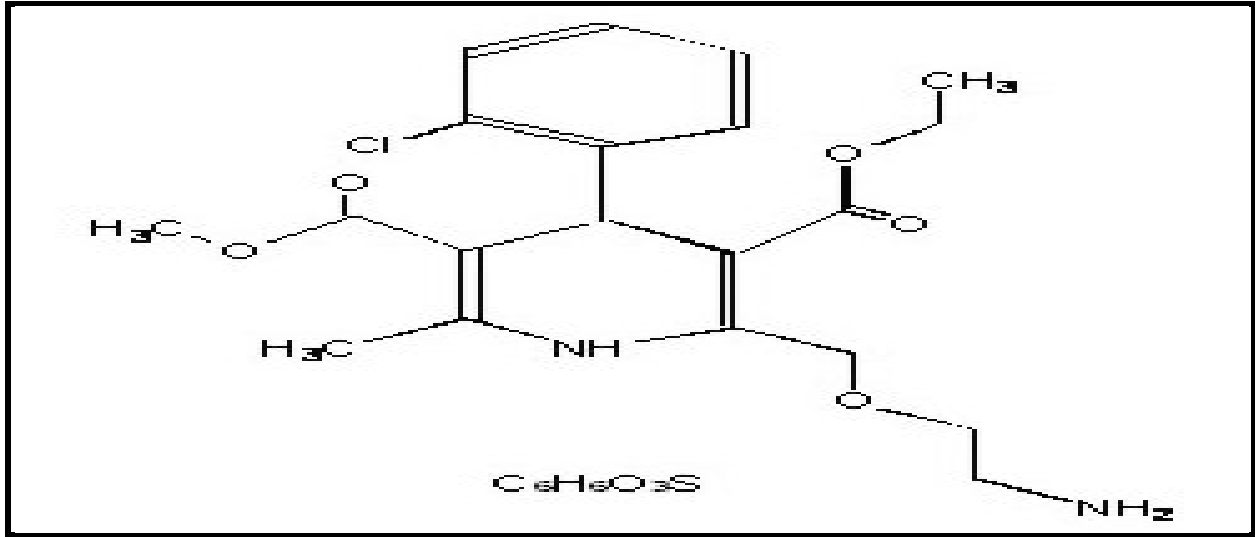


Figure No.1: Structure of Amlodipine besylate

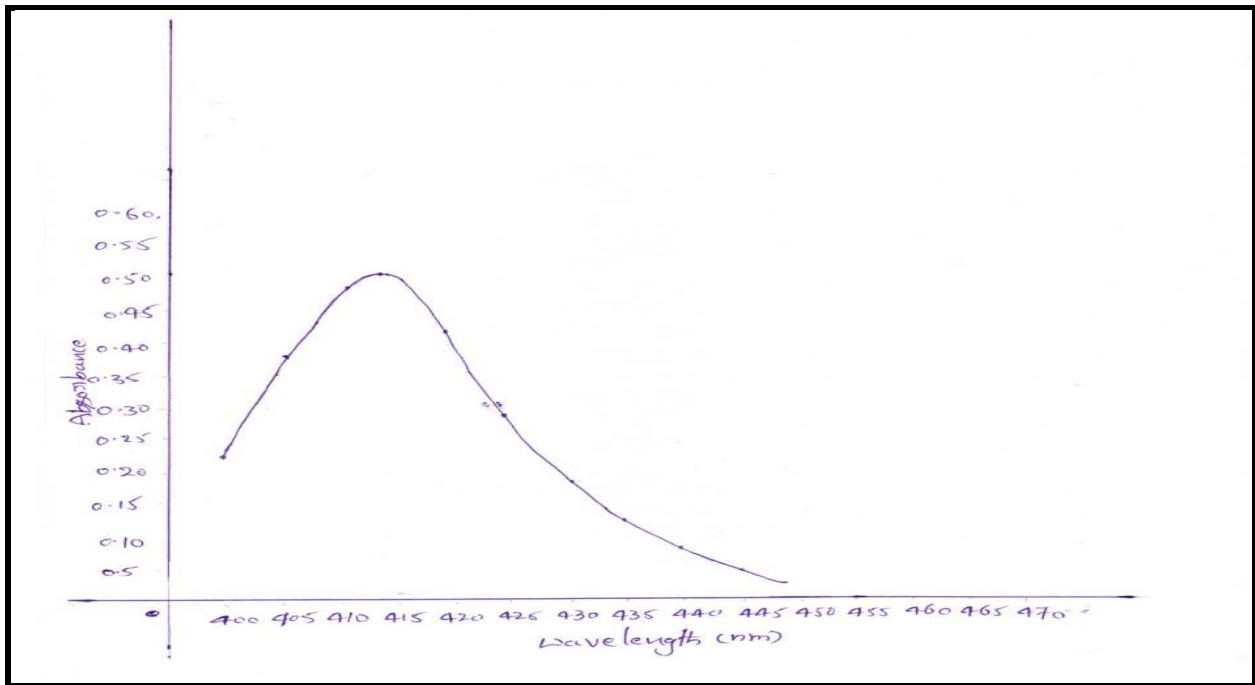


Figure No.2: Colorimetric determination of Amlodipine

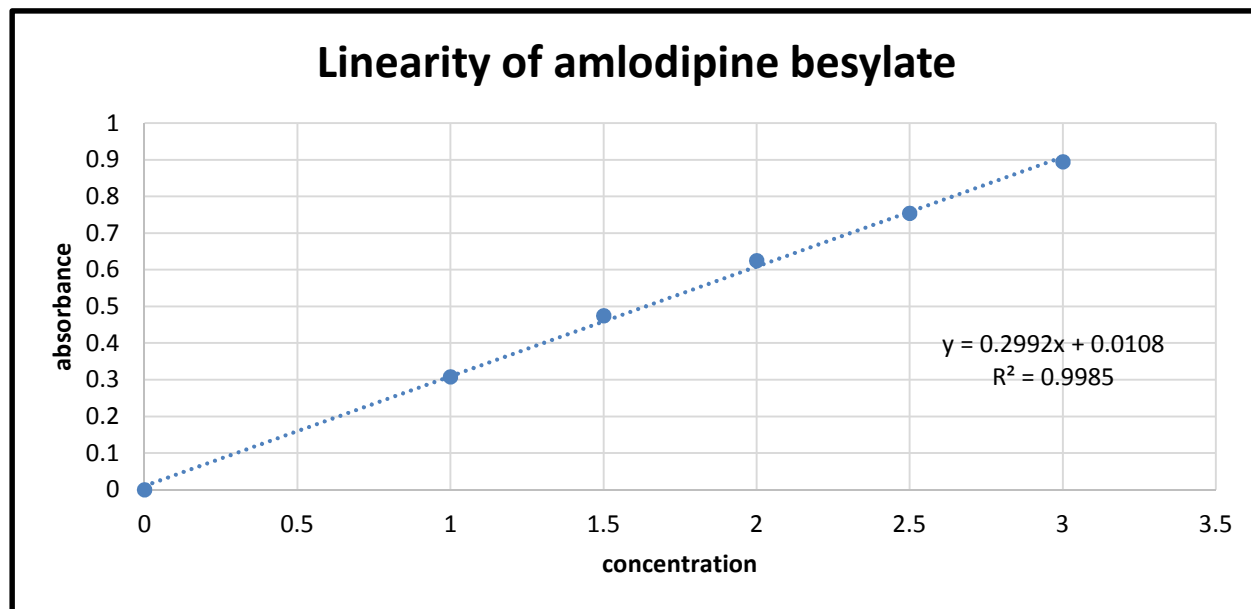


Figure No.3: Linearity curve for Amlodipine besylate at 414 nm

#### CONCLUSION

Amlodipine besylate is a commonly used drug to treat Hypertension currently marketed as Norvasc. The proposed analytical method is simple, reliable, rapid, sensitive and accurate for the determination of Amlodipine besylate. The method adopted for our studies is Colorimetric method. A new Colorimetric method was developed for both bulk drug and formulation. The proposed method uses the solvent as Chloroform. The content of the drug present in the formulation was found to be 5 mg. All the above studies do not suffer from any interference due to common excipients. Therefore it was shown that the proposed method could be successfully applied to estimate commercial pharmaceutical products containing Amlodipine besylate. Thus the above studies and findings will enable the quantification of drug for future investigation in the field of analytical chemistry. Among the established analytical methods Colorimetric method was found to be more precise and accurate. The % RSD (Table No.3) calculated for this method was very less. Hence the proposed method can be applied for regular analysis of Amlodipine besylate from the bulk drug and its dosage form.

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#### CONFLICT OF INTEREST

We declare that we have no conflict of interest.

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