INTERNATIONAL JOURNAL OF PHARMACEUTICAL, CHEMICAL AND BIOLOGICAL SCIENCES

Available online at www.ijpcbs.com

Research Article

UV SPECTROSCOPIC METHOD FOR ESTIMATION OF AMLODIPINE BESYLATE IN TABLETS

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ABSTRACT

A simple, sensitive, specific, and validated UV method has been developed for the quantitative determination of Amlodipine besylate in pure and tablet dosage form. The λmax was found to be 366nm for assay. The linearity was found in concentration range of 5-25 µg/ml. The correlation coefficient was found 0.999. The regression equation was found as Y=0.0238C- 0.0048. The method was validated for linearity, accuracy, precision and ruggedness. The LOD and LOQ for estimation of Amlodipine besylate were found as 0.136, 0.400 respectively. Recovery of Amlodipine besylate was found to be 99.80%.

Keywords: Amlodipine besylate, UV Spectrophotometry, Validation, Beer's law.

INTRODUCTION

Amlodipine besylate is chemically 3-Ethyl 5-methyl (4RS)-2-[(2-aminoethoxy) methyl]-4-(2-chlorophenyl)-6-methyl-1,4-dihydropyridine-3,5- dicarboxylate benzene sulphonate. Amlodipine is used in the management of hypertension[1] and coronary artery disease[2]. It is available in several officials in Pharmacopoeia[3-5]. Literature survey reveals that, Spectrophotometric methods[6-20], HPLC[21-23], HPTLC[24], UPLC[25]. In the present study, an attempt has been made to develop UV Spectrophotometric method for the determination of Amlodipine besylate in bulk and marketed formulations using 0.01% O-phosphoric acid. The developed method was found to be simple, sensitive and reproducible.

MATERIALS AND METHODS

Instrumentation

The present work was carried out on ElicoSL164 UV- Visible spectrophotometer having double beam detector configuration. The absorption spectra of reference and test solution were carried out in a 1 cm quartz cell over the range of 200-800 nm.

Chemicals

All chemicals of analytical grade used as it is.

Preparation of standard solution

Standard stock solution was prepared by dissolving accurately weighed 100 mg of amlodipine besylate in 0.01% O-phosphoric acid and the volume was made up to 100 ml with 0.01% O-phosphoric acid. (Stock solution-I, 1000 mcg/ml). 10 ml of stock solution-I was diluted to 100 ml with distilled water. (Stock solution-II, 100 mcg/ml). 1 ml of stock solution-II was diluted to 10 ml with distilled water, so that to produce the concentration 10 mcg/ml. The absorbance of resulting solution was measured against respective blank solution in the UV region of 200-400 nm, which shows maximum absorbance at 366 nm.

Preparation of sample solutions

20 tablets of one brand of amlodipine besylate was took, and all the tablets were crushed to fine powder by using pestle and mortar. Powder equivalent to 25 mg of amlodipine besylate was weighed accurately and transferred into a 25 ml standard volumetric flask. The contents were dissolved in 0.01% O-phosphoric acid and sonicated for five minutes. This solution was

filtered through 0.45 µm whatsmann filter paper. 5 ml of the filtrate was diluted to 50 ml with distilled water to get the solution of 100 mcg/ml. An aliquot of 1 ml of test solution was diluted to 10 ml with distilled water so that to produce the concentration 10 mcg/ml.

PROCEDURE

Aliquots of standard solution of amlodipine besylate ranging from 0.5-2.5 ml (1 ml = 100 mcg) were transferred into a series of 10 ml volumetric flasks. The volume in each flask was made up to 10 ml with distilled water and the absorbances were measured at 366 nm against solvent blank. The obtained absorbance values when plotted against the concentration of amlodipine besylate give the calibration graph.

VALIDATION

Validation of the developed method was done according to ICH guidelines[26].

Linearity

The linearity of the method was demonstrated over the concentration range of 5-25 mcg/ml of the target concentration. Accurately weighed 100 mg of pure drug was taken in clean, dry 100 ml volumetric flask and dissolved in small volume of 0.01% O-phosphoric acid and made up the volume to 100 ml with 0.01% O-phosphoric acid. This gave 1000 mcg/ml of drug concentration (Stock solution-I). From this 10 ml of solution was pipetted out into 100 ml volumetric flask and volume was made upto the mark with distilled water (Stock solution-II, 100 mcg/ml).

Concentrations of 5, 10, 15, 20, and 25 mcg/ml were prepared from above prepared Stock solution-II, calibration curve was plotted and the correlation coefficient was calculated.

Precision

Correlation coefficient of the linearity were found for method and reported in table No.1 The precision of an analytical method is the degree of agreement among individual test results when the method is applied repeatedly to multiple samplings of homogenous samples. It provides an indication of random error results and was expressed as coefficient of variation (CV).

Intra and inter-day precision

A variation of results within the same day (intra-day), variation of results between days (inter-day) was analyzed. Intra-day precision was determined by analyzing amlodipine besylate for five times in the same day at 366 nm. Inter-day precision was determined by analyzing the drug daily once for five days at 366 nm.

Accuracy

Accuracy is the closeness of the test results obtained by the method to the true value. The recovery technique was performed to judge the accuracy of the proposed method. For this, known quantities of the amlodipine besylate solution were mixed with definite amounts of pre-analyzed formulations and the mixtures were analyzed. The total amount of amlodipine besylate was determined by using the proposed method and the amount of added drug was calculated by the difference.

Ruggedness and Robustness

The solutions were prepared and analyzed with change in the analytical conditions like different laboratory conditions and different analysts.

RESULT AND DISCUSSIONS

The optimum conditions for UV spectroscopy method has been established by varying the parameters one at a time and keeping the other parameters fixed and observing the effects of products on the absorbance of the sample and colored species. Beer's law limits, molar absorbivity, Sandal's sensitivity, %range of error and % relative standard deviation are summarized in Table 1. The regression analysis using the method of least squares was made for the slope (b), intercept(a) and correlation coefficient (r)obtained from different concentrations are given in Table 1. The results showed that the method have reasonable precision. To evaluate the validity and reproducibility of the methods, known amounts of pure drug were added to the previously analyzed pharmaceutical dosage forms and the mixtures were analyzed by the proposed methods. The percentage recoveries are given in Table.3. The interference studies veiled that the common excipients and other additives that are usually present in the injection dosage forms did not interfere at their regularly added levels.

CONCLUSIONS

From the results the method described in this paper for the determination of Amlodipine besylate from tablet formulation is simple, accurate, sensitive and reproducible. The proposed method could be applied for routine analysis in quality control laboratories.

ACKNOWLEDGEMENTS

The authors are thankful to management of Sir I.P.V.V.A.S educational society for providing laboratory facilities.

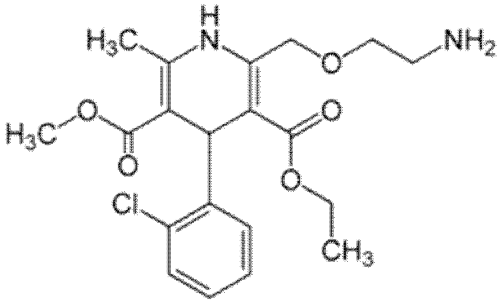

Fig. 1: Structure of Amlodipine besylate

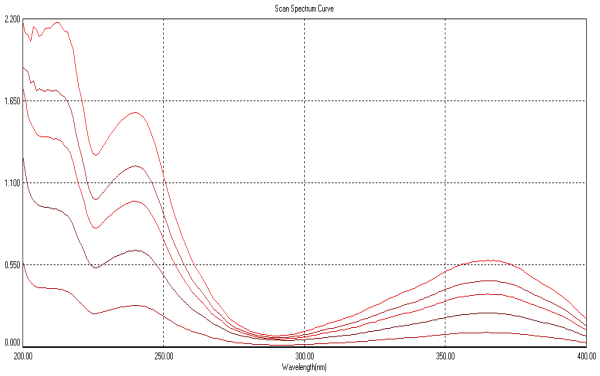

Fig. 2: Overlay spectrum of amlodipine besylate in 0.01% O-phosphoric acid (5-25mcg/ml)

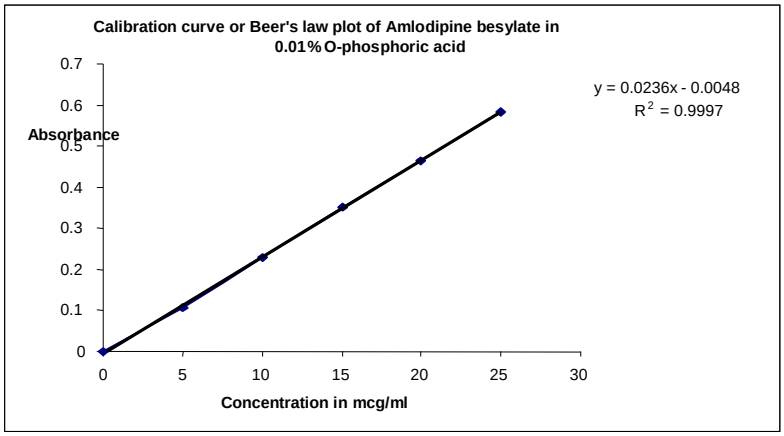

Fig. 3: Calibration curve of amlodipine besylate in 0.01% O-phosphoric acid (5-25mcg/ml)

Table 1: Optimum conditions, Optical characteristics and Statistical data of the regression equation in UV method with 0.01% O-phosphoric acid

Parameter	UV Method with 0.01% O-phosphoric acid
λ_{max} (nm)	366
Beer's law limits (mcg/ml)	5-25
Molar extinction coefficient ($mol^{-1} cm^{-1}$)	0.0138 X10^4
Sandell's sensitivity (mcg/cm^2-0.001 absorbance units)	0.080
Regression equation (Y*)	Y=0.0238C- 0.0048
Slope (b)	0.0238
Intercept (a)	0.0048
Correlation coefficient(r^2)	0.9997
% RSD**	0.96
Limit of detection (mcg/ml)	0.136
Limit of quantitation (mcg/ml)	0.400

Table 2: Analysis of formulation

Drug	Amount (mg/tablet)		% label claim	%RSD
	labelled	Found		
Amlodipine	10	9.977	99.77	0.86

Table 3: Recovery Studies

Drug	Labeled claim (mg/Tablet)	Estimated amount (mg/Tablet)	Spike level (%)	Amount of drug added (mg)	Amount of drug recovered (mg)	Percentage recovery ± SD*
Amlodipine	10	9.977	50	2mg	1.996	99.80±1.130
			100	4mg	3.998	99.95±1.586
			150	6mg	5.994	99.90±1.258

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