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UV Spetrophotometric Method Development and Validation for determination of Lacidipine in Pharmaceutical dosage form

Nagaraju P.T.*, K. P. Channabasavaraj, Shantha Kumar P .T. , K.Chiranjeevi

Department of Pharmaceutical Analysis, BharathiCollege of Pharmacy, Bharathinagara, Maddur, Karnataka-571422, India.

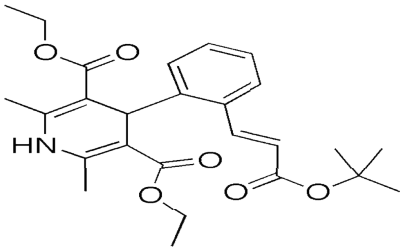
***Corres.author: nraju04@gmail.com**
Mob: 09885755749

Abstract: A simple, precise and economical second order derivative method has been developed for the estmation of Lacidipine in bulk and pharmaceutical formulations. In this method Lacidipine showed sharp peak at 218 nm when n= 1 and linearity was measured at 218 nm. It obeyed Beer's law in the concentration range of 5-30 mcg/ml. The LOD and LOQ were found to be 0.36 mcg/ml and 1.11 mcg/ml respectively. A recovery of Lacidipine in tablet formulation was observed in the range of 98.30-101.09%. The proposed method is precise, accurate and reproducible and can be extended to the analysis of Lacidipine in bulk and pharmaceutical formulations.
Keywords : Lacidipine, Derivative spectrophotometric, Method validation.

1. Introduction

Lacidipine [1] is a calcium channel blocker drug. Lacidipine is a highly vascular selective newer dihydropyridines suitable for once daily administration. It is claimed to attain higher concentration in Vascular smooth muscle membrane; approved only for use as antihypertensive. Chemical name of Lacidipine is (E)-4-[2-[3-(1,1-Dimethylethoxy)-3-oxo-1-propenyl] phenyl]-1,4-dihydro-2,6-dimethyl-3,5-pyridine dicarboxylic acid diethyl ester. It has a molecular formula of $C_{26}H_{33}NO_6$ and a molecular weight of 455.55 g/mol.

Literature survey reveals that several analytical methods have been reported for the estimation of Lacidipine by LC-DAD[2], High Performance Thin Layer Chromatography[3] , HPLC[4], LC-MS[5,6] and UV[7] methods. Apart from above no other spectroscopic methods such as UV/Vis, difference spectrophotometric method, RP-HPLC etc., were reported for this compound.



Lacidipine

In derivative spectroscopy by UV no second order derivative spectrophotometric method was reported for the quantitative determination of Lacidipine in pharmaceutical dosage forms. The developed method was simple, precise, specific and accurate and the statiscal analysis proved that method is reproducible and selective for the analysis of Lacidipine in bulk drug and tablet formulations.

2. Experimental

2.1 Instruments and reagents

A Shimadzu UV - 1800 UV/VIS spectrophotometer was used with 1 cm matched quartz cell.

All the chemicals used were of analytical grade. Methanol A.R. grade was procured from Loba Chem. Ltd., Mumbai. An analytically pure sample of Lacidipine was obtained from Cipla Health Care, Ahmadabad as a gift sample. Tablet of 2 mg were procured from local pharmacy.

2.2 Preparation of working standard drug solution

The standard Lacidipine (100 mg) was weighed accurately and transferred to volumetric flask (100 ml). It was dissolved properly and diluted up to the mark with methanol to obtain final concentration of 1000 mcg/ml and the resulting solution was used as working standard solution.

2.3 Analysis of marketed formulations

For the estimation of Lacidipine in tablets formulations by this method. 20 branded tablets were weighed and triturate to fine powder. Tablet powder equivalent to 10 mg of Lacidipine was weighed and transfer into 100 ml volumetric flask than dissolved with methanol and further diluted with methanol. It was kept for ultrasonication for 30 min; this was filtered through Whatman filter paper No. 41 and then final dilution was made with methanol to get the final stock solution of 100 mcg/ml. From this stock solution, various dilutions of the sample solution were prepared and analysed.

2.4 Second order derivative spectroscopic method

The second order derivative spectra showed sharp peak at 218 nm when n=1 and linearity was measured at 218 nm (Fig 1). The absorbance difference at n=1 ($dA/d\lambda$) is calculated which was directly proportional to the concentration of the standard solution. The standard drug solution was diluted so as to get the final concentration in the range of 5-30 mcg/ml and scanned in the second order derivative spectra. The calibration curve of $dA/d\lambda$ against concentration of the drug showed linearity. Similarly absorbance of sample solution was measured and amount of Lacidipine was determined from standard calibration curve.

3. Results and Discussion

As the drug Lacidipine showed a broad spectrum, the derivative spectroscopy method applied has the advantage that it locate the hidden peak in the normal spectrum, when the spectrum is not sharp and it also eliminate the interference caused by the excipients and the degradation products present, if any, in the formulation.

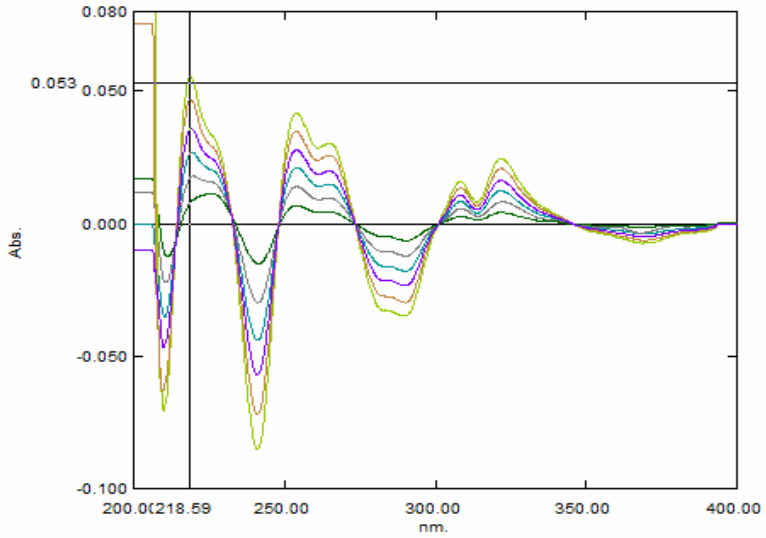


Figure1: Second order derivative spectrum of Lacidipine with n=1

Table 1:Calibration Parameters

Parameters	**Second order Derivative Method**
λ_{max} (nm)	218
Beer's law limits (µg/ml)	5-30
Molar extinction coefficient (mol^{-1} cm^{-1})	0.0018 $X10^4$
Sandell's sensitivity (µg/cm^2-0.001 absorbance units)	0.555
Regression equation (Y*)	Y= 0.0019 X - 0.0017
Slope (b)	0.0019
Intercept (a)	0.0017
Correlation coefficient(r^2)	0.9997
% RSD**	0.565
Limit of detection (µg/ml)	0.368
Limit of quantitation (µg/ml)	1.1157

*Y= b X + a where X is the concentration of Lacidipine in µg/ml and Y is the absorbance at the respective $\lambda_{max.}$

**Average of six determinations.

Table 2 : Recovery study Data

Brand name	**Amount of sample (µg/ml)**	**Amount of drug added (µg/ml)**	**Amount Recovered** (µg/ml)**	**% Recovery± SD****
Sinopil	20	15	15.20	101.09 ± 0.70
Sinopil	20	20	19.66	98.30 ± 0.67
Sinopil	20	25	25.06	99.61 ± 0.93

**Average of six determinations.

Table 3:Analysis of tablet formulation

Tablet	**Label claim**	**Amount found (mg)**	**%Recovery ± S.D****
Sinopil	2	1.98	99.21 ± 0.028

**Average of six determinations.

The second order derivative spectra showed sharp peak at 218 nm when n=1 and linearity was measured at 218 nm. The polynomial regression data for the calibration plots showed good linear relationship in the concentration range of 5-30 mcg/ml with r^2= 0.9997 and given in Table 1.

Recovery studies were carried out at three different levels i.e. 80%, 100% and 120% by adding the pure drug to the previously analysed tablet powder sample and shown in Table 2. The percentage recovery value indicates non interference of the excipients used in formulation.

The marketed formulation analysis are shown in Table 3. The reproducibility and accuracy of the method were found to be good, which was evidenced by low standard deviation.

4. Conclusion

A spectrophotometric method for quantifying Sinopil in formulation samples has been developed and validated. The proposed method is precise, accurate and reproducible and can be extended to the analysis of Lacidipine in bulk and tablet formulations.

.**5. Acknowledgement**

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6. References

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