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METHOD DEVELOPMENT AND VALIDATION OF UV SPECTROSCOPIC METHOD FOR THE DETERMINATION OF LERCANIDIPINE

Dr. M. Sunitha Reddy*, Navya Sai.K, S. Muhammad Fazal Ul Haq

Department of Pharmaceutics, Centre for Pharmaceutical Sciences, Jntuh, Hyderabad, Telangana, India.

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***Correspondence for Author**
Dr. M. Sunitha Reddy
Department of Pharmaceutics, Centre for Pharmaceutical Sciences, Jntuh, Hyderabad, Telangana, India.

ABSTRACT

Lercanidipine is a calcium channel antagonist used as antihypertensive agent. A suitable spectroscopic method which is specific,accurate,and precise, has been developed for the determination of lercanidipine.The solvent used is methanol and λmax is found to be 239nm.The method is highly sensitive and linearity is observed at 5ppm to 25ppm.Regression equation is found to be y = 0.03566 x + 0.00620. The method is validated for various parameters like precision, accuracy, robustness,detection and quantification limits. The results shows that method is precise accurate, reproducible,simple,cheap and less time cosuming. This method can be suitable for determination of lercanidipine in bulk formulation.

KEY WORDS: lercanidipine, method development, spectroscopic method, validation.

INTRODUCTION

Lercanidipine is a calcium channel antagonist used as antihypertensive agent.chemically it is 3-{1-[(3,3-diphenylpropyl)(methyl)amino]-2-methylpropan-2-yl} 5-methyl 2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate . A few methods are available for determination of lercanidipine in uv-spectroscopy.Present work shows method is precise accurate and sensitive method for the determination of lercanidipine .The method is also validated for various parameters.

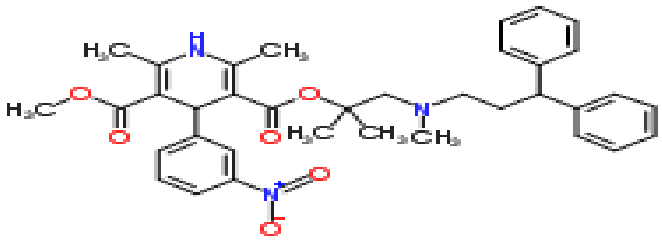

Figure 1: structure of lercanidipine

MATERIALS AND METHOD

- **Instruments**

Analytical balance

Uv-spectroscopy

Glass ware

- **Reagents and chemicals**

Lercanidipine

Methanol

Acetonitrile

Triple distilled water.

WAVELENGTH SELECTION

Different concentration of lercanidipine 5 to 25 ppm was prepared in methanol.They are scanned under uv within 200-400nm wavelength range by using methanol as blank.The absorption maxima from spectra was noted as 239nm.It is shown in fig.2.

PREPARATION OF STOCK SOLUTION

100mg of lercanidipine was dissolved in 100 ml of methanol in 100 ml vol.flask.The resultant concentration is 1000ppm.

PREPARATION OF WORKING STANDARD

From the stock solution 1000ppm,10 ml was withdrawn and taken in 100ml vol.flask.Make it upto 100ml with methanol.This gives concentration of 100ppm.

PREPARATION OF SAMPLE AND CALIBRATION CURVE

From the working standard 5,10.15,20,25 ppm solutions were prepared by taking 0.5ml,1ml,1.5ml,2ml,2.5ml and made upto 10 ml with methanol.The absorbance of these solutions were measured at λmax 239nm by using methanol as blank. Calibration curve was shown in fig.3.

METHOD VALIDATION

Accuracy

For determining accuracy of the proposed method, drug concentrations were prepared from independent stock solution as 50%,100%,150% of the 20ppm concentration and analyzed . Accuracy was assessed as %RSD.

Precision

Repeatability was determined by using different levels of drug concentrations (same concentration levels taken in accuracy study i,e,. 20ppm), prepared from independent stock solution and analyzed. Inter-day and intra-day variation and instrument variations were taken to determine intermediate precision of the proposed methods. Different levels of drug concentrations in triplicates were prepared three different times in a day and studied for intra-day variation. Same protocol was followed for three different days to study inter-day variation.

Linearity

The linearity is established for the proposed method. separate series of solutions of the drug (5-25 µg/ ml in methanol medium) were prepared from the stock solutions and analyzed. Least square regression analysis is done for the obtained data.

Detection limit and quantitation limit

Detection limit (DL) and quantitation limit (QL) for the developed method is determined by using calibration curve standards. DL and QL were calculated as 3.3r/S and 10r/S, respectively, where S is the slope of the calibration curve and r is the standard deviation of y-intercept of regression equation.

Robustness

Robustness of the proposed method is determined by (a) changing the media (acetonitrile) and (b)changing the wavelength (±3nm). Different concentrations were prepared in both media. %RSD was determined.

RESULTS AND DISCUSSIONS

After optimization methanol is selected as media which shows high sensitivity, less cost and ease of preparation.calibration curve is shown in fig. It shows λmax of 239nm. Regression equation is found to be Y=0.03566 X+0.00620 at 239 nm with regression coefficient 0.99976.

ANALYTICAL VALIDATION

Accuracy

%Recovery values are excellent and has low standard deviation values.This shows that the method is accurate.Mean percentage recoveries for the concentrations were found to be in the range of 95.165% to 97.4% and the %RSD ranges from 0.000423 to 0.000665.This shows that small change in concentrations of the sample can be accurately determined by this method.

Presicion

Repetability results were seen under same conditions for a short time intervals and in different days with laboratory variations.values are noted for 20 ppm.% RSD values were within the acceptance range which shows that the method has repeatability and it is precise.

linearity

In methanol medium the linearity range was found to be 5–25 µg ml–1 at 239 nm. Lower values of parameters like standard error of slope and intercept indicated high precision of the proposed methods. The mean slope and intercept values are within confidence limits.

LOD AND LOQ

In methanol LOD and LOQ values were found to be 0.086618 and 0.2624 respectively.

Robustness

Variation in media by Acetonitrile and change in wavelength by ±3% do not show any significant change in absorbance.

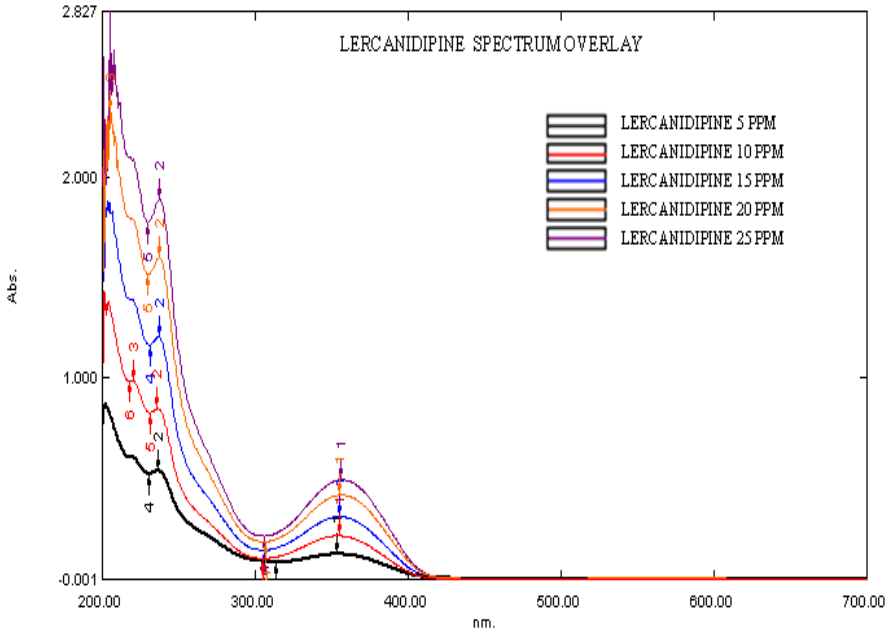

Figure 2 absorption spectrum of lercanidipine

Table 1: standard table for calibration curve.

s.no	Concentration (ppm)	Absorbance at 239nm
1	5	0.169
2	10	0.389
3	15	0.537
4	20	0.711
5	25	0.899

Table 1: different parameters and values

Parameter	Values
Λmax	239nm
Beer's law limit(ug/ml)	5-25
Regression equation(Y=mX+C)	y = 0.03566 x + 0.00620
Slope(m)	0.03566
Intercept(c)	0.00620
Correlation coefficient(r2)	0.99976
Limit of detection(ug/ml)	0.086618
Limit of quantification(ug/ml)	0.2624

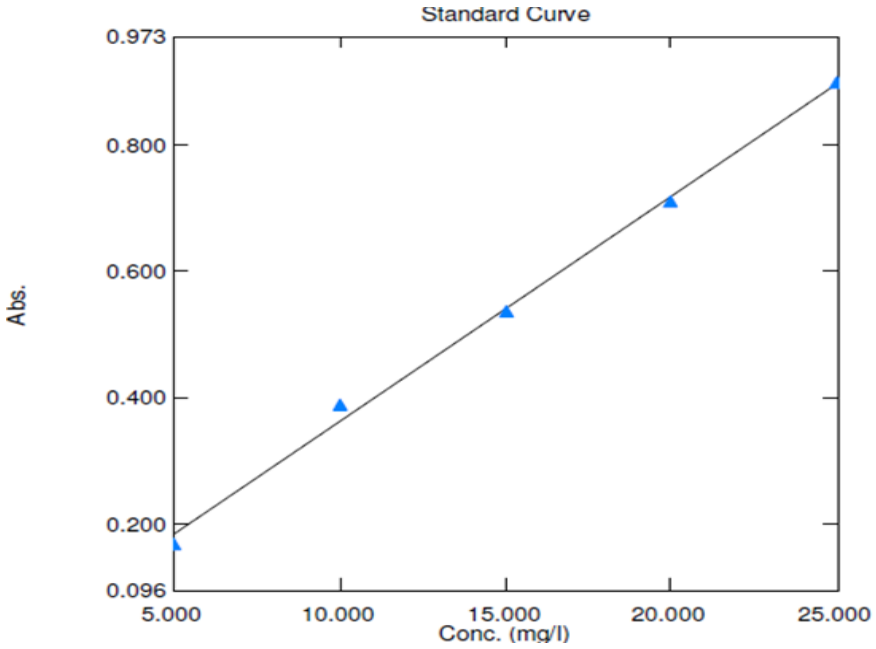

Figure 3 calibration curve of lercanidipine

ACCURACY

Table 2: accuracy table.

s.no	Concentration (ug/ml)	Mean absorbance	SD	%RSD	Concentration Obtained	%recovery
1	30	1.049	0.000577	0.000551	29.233	97.44
2	40	1.364	0.000577	0.000423	38.066	95.165
3	50	1.736	0.001155	0.000665	48.508	97.016

PRECISION

Table 3: precision table.

s.no	concentration	Precision(day)	absorbance±SD	%RSD
1	20ppm	Normal day	0.709±0.0	0
2	20ppm	Intra day	0.697±0.001	0.143472
3	20ppm	Inter day	0.74±0.000548	0.074054

Where n=3

DETECTION LIMIT AND QUANTIFICATION LIMIT

Table 4: LOD AND LOQ.

s.no		Values	LOD	LOQ
1	Slope	0.03566	0.086618	0.2624
2	Intercept	0.00620		
3	R^2	0.99976		

ROBUSTNESS

Table 5: ROBUSTNESS.

s.no	Parameter	Concentration	absorbance	SD	%RSD
1	Solvent change ACN+methanol	20ppm	0.747	0	0
2	Low wavelength (236nm)	20ppm	0.782	0.000447	0.057161
3	High wavelength (242nm)	20ppm	0.667	0.000447	0.067016

CONCLUSION

The proposed method was simple ,precise, accurate reliable and sensitive.the method is specific and is useful for determination of lercanidipine in pure samples and in formulations.

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