

# Spectrophotometric Method for Determination of Lercanidipine in Tablets

G. Mubeen, Damanjit Singh Rao, Kadri Uvesh\*

Department of Quality Assurance, Al-Ameen College of Pharmacy, Near Lalbagh Main Gate, Bangalore – 560027, India.

\*Corres. Author: [kadriuvesh@yahoo.co.in](mailto:kadriuvesh@yahoo.co.in)  
+91-9979971053

**ABSTRACT:** Lercanidipine is calcium channel antagonist, used as an antihypertensive agent. A simple, rapid, precise and accurate spectrophotometric method has been developed and validated for determination of Lercanidipine in tablet formulation. The method is based on diazotization of reduced Lercanidipine with nitrous acid followed by its coupling with  $\beta$ -naphthol in an alkaline medium to form red color chromophore with absorption maximum at 553 nm. Linearity was observed in the concentration range of 2-14  $\mu\text{g/mL}$ . The assay result was found to be in good agreement with label claim. The recovery studies were carried out at three different levels. The method was validated statistically and by recovery studies.

**Key words:** Lercanidipine, Spectrophotometry, Validation

## INTRODUCTION

Lercanidipine<sup>1</sup> is chemically 2-[(3,3-diphenylpropyl) methylamino]-1,1-dimethylethyl, methyl-1,4-dihydro-2,6-dimethyl-4-(3-nitrophenyl)-3,5-pyridine dicarboxylic ester with molecular formula  $\text{C}_{36}\text{H}_{41}\text{N}_3\text{O}_6$ . It is a new third generation 1,4-dihydropyridine calcium channel antagonist used as antihypertensive agent<sup>2</sup>. Literature review revealed that few methods<sup>3-7</sup> are available for the determination of Lercanidipine in bulk, formulation and in plasma.

The present study describes accurate, precise and reproducible spectrophotometric method for estimation of Lercanidipine in tablet formulation. The method was validated by using various parameters as per ICH guidelines.

## EXPERIMENTAL

### Instruments and Materials:

Pure Lercanidipine was obtained as gift samples from Healthon, division of Glenmark Pharmaceutical Limited, Mumbai, India. The Shimadzu UV-Visible spectrophotometer 1601 was used with spectral bandwidth 3 nm and wavelength accuracy (with automatic wavelength correction) 0.5 nm. All the apparatus and instruments were calibrated and validated before starting the experimental work.

## MATERIALS AND METHODS

### Preparation of standard solution:

Standard solution was prepared by dissolving 100 mgs of Lercanidipine hydrochloride in 20 ml of methanol and treated with 5 g of zinc dust and 4 ml of concentrated Hydrochloric acid and kept at room temperature ( $25 \pm 2^\circ\text{C}$ ) for 1 hour, the solution was filtered through Whatman filter paper and residue was washed with 3 x 10 ml portions of methanol and diluted to 100 ml with distilled water. 10 ml of this solution is further diluted to 100 ml with distilled water to get a concentration of 100  $\mu\text{g/ml}$ . Different aliquot was taken from standard solution to obtain series of concentrations 1-100  $\mu\text{g/ml}$  in 10 ml volumetric flask, then 1 ml of 1 N HCl, 1 ml of 1% w/v sodium nitrite solution was added and allowed to stand for 10 minutes at  $0 - 5^\circ\text{C}$ , 1 ml of ammonium sulphamate solution was further added, mixed and allowed to stand for 2 minutes, 0.5 ml of alkaline  $\beta$ -naphthol solution was added mixed well and the final volume was made up to 10 ml with distilled water and the absorbance was recorded at 553 nm against reagent blank using Shimadzu UV-Visible 1601 Spectrophotometer.

**Validation of Analytical method:**

Validation is the process of establishing documented evidence which provides a high degree of assurance that a specific process will consistently produce a product meeting its predetermined specifications and quality attributes.

**1. Linearity**

The linearity of Lercanidipine was found to be 2-14 µg/mL. The linear regression value was found to be  $R^2 = 0.9981$  (Table 1).

**2. Precision**

Precision is the degree of agreement among individual test results when the procedure is applied repeatedly to multiple samplings of a homogeneous sample. Precision is usually expressed as the standard deviation or relative standard deviation. In the present study, developed method was validated for method, system, inter-day and intra-day precision.

As the values of % RSD of all precision study were within the acceptable limits (less than 2 %), both the method as well as the system provides good precision and reproducibility (Table 1).

**3. Sensitivity**

Absorbance of standard solutions of Lercanidipine was measured at 553 nm. Sandell's sensitivity<sup>8</sup> for Lercanidipine drugs was calculated from formula.

$$(\mu\text{gcm}^{-3} \text{ AU}) = \frac{\text{Conc. of drug } (\mu\text{g}100 \text{ mL}^{-1})}{\text{Absorbance}} \times 0.001$$

The Sandell's sensitivity for Lercanidipine at 553 nm was found to be 0.0151 µgcm<sup>-3</sup>AU.

**4. Accuracy**

Accuracy of the method was determined in the terms of % recovery of standard Lercanidipine. Recovery studies were carried out by addition of standard drug solution at 3 different levels to the preanalyzed sample. Results of the recovery study were found to be within the acceptance criteria 100 ± 10 %, indicates sensitivity of the method towards detection of Lercanidipine and non interference of excipients in the method (Table 1).

**Procedure for the analysis of tablet formulation:**

Sample: Lercanidipine hydrochloride Tablet  
Brand name: Lerez (Lercanidipine solution = 10 mg)  
Manufacturer: Healthon, division of Glenmark Pharmaceutical Limited, Mumbai, India.

**Preparation of sample solution:**

Twenty tablets were accurately weighed, their average weight determined, crushed to fine powder. An accurately weighed quantity of tablet powder equivalent to 100 mgs of Lercanidipine hydrochloride was transferred to 100 ml volumetric flask, dissolved in 20 ml of methanol and treated with 5 g of zinc dust and 4 ml of concentrated hydrochloric acid. After keeping at room temperature (25 ± 2°C) for 1 hour, the solution was filtered through Whatman filter paper and the residue was washed with 3 x 10 ml portion of methanol and diluted to 100 ml with distilled water. 10 ml of this solution was further diluted to 100 ml with water to get a solution of strength 100 µg/ml. To 1 ml of above sample solution in 10 ml volumetric flask, 1 ml of 1 N HCl, 1 ml of 1% w/v sodium nitrite solution was added, allowed to stand for 10 minutes at 0 - 5°C then 1 ml of ammonium sulphamate solution was added, mixed, kept for 2 minutes, then treated with 0.5 ml of alkaline β-naphthol solution mixed well and the final volume was made up to 10 ml with distilled water and the absorbance was recorded at 553 nm against reagent blank and the concentration of Lercanidipine was determined.

**Recovery studies:**

To check the accuracy of the proposed method, recovery studies were carried out at three different levels i.e 100 %, 120 % and 140 %. The standard bulk drug was added at 3 different levels to the preanalyzed sample solution and then reanalyzed.

**RESULT AND DISCUSSION**

Lercanidipine is calcium channel antagonist used as antihypertensive agent; it showed maximum absorbance at 553 nm. Linearity was obeyed in concentration range of 2-14 µg/mL. The percentage label claim was found to be in good agreement. The % recovery was found to be in range of 97-102 % w/w. The % RSD less than 2 indicated that the method was accurate and precise. The method was found to sensitive with respect to Sandell's sensitivity. The developed method was found to be simple, linear, accurate, precise and reproducible.

**CONCLUSION**

A Spectrophotometric method has been developed for the determination of Lercanidipine in tablet formulation. The method was validated based on ICH analytical method validation guidelines. The method was found to be accurate, linear, precise and reproducible. Hence the method can be used for routine analysis of Lercanidipine in bulk and in tablet formulation.

**Table 1: Validation Parameters**

SI. No.	Parameters	Lercanidipine	
1.	Linearity	$\mu\text{g/mL}$	2 – 14
		Regression eq <sup>n</sup>	0.1592x – 0.1388
		R <sup>2</sup>	0.9981
2.	Precision (% RSD)	Method	0.8625
		System	1.1911
		Inter-day	0.6364
		Intra-day	1.2010
3.	Sensitivity $\mu\text{g/cm}^{-3}$ / AU	At 553 nm	0.0151
4.	Accuracy (%Recovery)	N=3	97-101 %

**Table 2: Analysis of tablet formulation**

Label claim mg/tablet	Amount found*	% Amount found	SD	% RSD
Lerez 10 mgs	10.008	100.08	0.0056	0.0565

\*Average of six determinations

**Table 3: Recovery studies**

Sl. No.	Conc. of standard drug ( $\mu\text{g/ml}$ ) (A)	Conc. of marketed sample ( $\mu\text{g/ml}$ ) (B)	Total drug conc ( $\mu\text{g/ml}$ ) (A+B)	Absorbance* at 553 nm	Total conc of lercanidipine from standard curve ( $\mu\text{g/ml}$ )	Amount of sample ( $\mu\text{g/ml}$ )	% Recovery
1	8	2	10	0.660	10.00	8.00	100.00
2	8	4	12	0.825	12.10	8.10	101.25
3	8	6	14	0.955	13.83	7.83	97.87

\*Average of three readings.

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