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ESTIMATION OF NISOLDIPINE BY RP-HPLC IN ORAL SOLID DOSAGE FORM

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ABSTRACT
A reverse phase high performance liquid chromatographic method has been developed for the estimation of nisoldipine in tablet formulation. The separation was achieved by Phenomenex Gemini C_{18} column (250x4.60mm, particle size 5ì) using water, acetonitrile and methanol (40:40:20) as mobile phase, at a flow rate of 1.5mL/min. Detection was carried out at 235 nm. Retention time of NIS was found to be 5.3min. The method has been validated for linearity, accuracy and precision. Linearity observed was in the range of 80-120µg/mL. The developed method was found to be accurate, precise, selective and rapid for estimation of nisoldipine in tablet dosage form.

Keywords: *Nisoldipine, (NIS), RP-HPLC.*

INTRODUCTION

Nisoldipine[1] is chemically 1,4-dihydro-2,6-dimethyl-4-(2-nitrophenyl)-3,5-pyridine dicarboxylic acid methyl 2-methylpropyl ester. The drug is not official in any Pharmacopoeia. It has a molecular weight of 388.41g/mol and the molecular formula is $C_{20}H_{24}N_2O_6$ (Fig- 1). It is a dihydropyridine calcium channel blocker which is used alone or with an angiotension converting enzyme inhibitor to treat hypertension and angina pectoris. The drug is similar to other peripheral vasodilators and inhibits the influx of extracellular calcium across the myocardial and vascular smooth muscle cell membranes. Literature review reveals that HPLC methods have been reported for hydrolytic degradation[3] studies and enantioselective assay[4] of nisoldipine in biological fluids by chiral high performance liquid chromatography. Estimation for nisoldipine (NIS) in bulk and formulation by RP-HPLC[2] has not been reported.

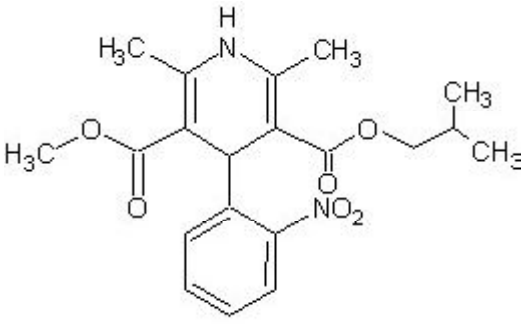

Fig. 1

EXPERIMENTAL

Instrumentation

Shimadzu UFLC, UV/VIS detector SPD 20A, LC 20AT pump system and reverse phase Phenomenex Gemini C_{18} column (250x4.60mm, particle size 5µ) was used.

Chromatographic conditions

The mobile phase consists of mixture of water, acetonitrile and methanol in the ratio 40:40:20 v/v and was filtered through 0.45 µ membrane filter and sonicated before use. The flow rate of mobile phase was maintained at 1.5 mL/min. The temperature is ambient and the volume of injection was 20µL. The eluent was monitored at 235nm.

Preparation of mobile phase

A quantity of 40mL of HPLC grade water and 40mL of HPLC grade acetonitrile and 20mL of HPLC grade methanol were mixed well and the solution was filtered using 0.45µ membrane filter.

Preparation of standard stock solution

The standard solution was prepared by dissolving 50mg of drug in mobile phase and made up to 100mL in a volumetric flask; subsequent dilution of this solution ranging from 80-120µg/mL were prepared using the mobile phase.

Preparation of sample solution

Twenty tablets were accurately weighed and powdered. The powder equivalent to 25mg was accurately weighed and transferred into a 50mL volumetric flask. The contents were dissolved in 25mL of the mobile phase and sonicated for 15min. Then the solution was made up to volume with mobile phase. The resulting solution was filtered through a 0.45µ membrane filter and the filtrate was used.

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ASSAY PROCEDURE

20µL of standard of varied concentrations were injected and their retention time was determined. The peak area versus concentration was plotted as calibration graph. The sample solution was analyzed by injecting 20µL of the solution and the peak area was determined. Evaluation of the drug was done at 235nm. The amount of NIS present in commercial tablets was calculated by plotting a calibration graph using peak area and the concentrations of the standard drug.

RESULTS AND DISCUSSION

In above method, C_{18} column and reverse mode were used for analysis. The mobile phase used was water, acetonitrile and methanol in the ratio 40:40:20 at a flow rate of 1.5mL/min. The eluent was monitored at 235nm and the retention time was found to be 5.3mins. A calibration graph was constructed using peak area versus concentration. The LOD and LOQ were found to be 7.853 and 23.79µg/mL. The %RSD was less than 1 and the percent recovery was found to be greater than 98% which showed the reproducibility and accuracy of the method. The system suitability parameters are shown in Table 1. The results of analysis and recovery studies are shown in Table 2.

Table 1: *System suitability parameters*

S. No.	Parameters	Results obtained
1.	No. of theoretical plates	6859
2.	Linearity in µg/mL	80-120
3.	Accuracy (% recovery)	98.73
4.	LOD (µg/mL)	7.853
5.	LOQ (µg/mL)	23.79
6.	Asymmetry factor	1.86
7.	Correlation coefficient	0.9999
8.	Slope (m)	11.000
9.	Intercept	1.96

Table 2: *Assay and recovery of nisoldipine and its formulations*

FORMULATION	LABEL CLAIM (mg)	AMOUNT FOUND BY PROPOSED METHOD	%RECOVERY BY THE PROPOSED METHOD
TABLET-I	8.5mg	8.32mg	98.11%
TABLET-II	34.0mg	35.36mg	98.37%

* *Each value is an average of three determinations*

REFERENCES

1. The Merck Index, Whitehouse Station, NJ: Merck & Co. Inc., 13th edn, 2001;1136.
2. Sethi PD. High Performance Liquid Chromatography Quantitative analysis of Pharmaceutical formulations, New Delhi: CBS Publishers & Distributors, 60-61, 173-210.
3. A Alvarez-Lueje, *et al*., J Pharm Biomed Anal. 2002;28:887-95.
4. Marques MP, *et al*. J Chromatogr B Biomed Sci Appl. 2001; Oct 5; 762(1):87-95.