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Estimation of nifedipine by reverse phase high performance liquid chromatography tablet dosage form

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Abstract

A simple, selective, rapid, precise, sensitive and accurate HPLC method has been developed for the estimation of nifedipine using methanol and water (70:30 v/v) as mobile phase; pH was adjusted to 3.0 with orthophosphoric acid. Detection was carried out using UV detector at 238 nm. The method was carried out on a Shim-Pack CLC, ODS (C-18), 5 µ column and dimension of column was 250×4.6mm. The retention time of nifedipine was 3.401 min. The developed method was validated in terms of linearity, precision, accuracy, precision, limit of detection and limit of quantitation. The method was found to be linear in the range of 5-40 µg/ml. In the linearity study, regression equation and coefficient of correlation was found to be y = 361627x + 338075, r^2= 0.9996.

Key-Words: Nifedipine, RP-HPLC, Methanol, Water.

Introduction

Nifedipine is chemically known as dimethyl1,4-dihydro-2,6-dimethyl-4 (2-nitrophenyl) pyridine-3,5 di carboxylate , it is a dihydropyridine calcium channel antagonist widely used in the treatment of angina pectoris, hypertension,and other vascular disorders suchas Ray-naud's phenomenon[1,2].

Literature reveals that several works have been reported on nifedipine determination in plasma, formulations adopting gas chromatographic techniques with an electron capture detector or HPLC, using reverse phase columns and UV or fluorometric detection[3-17]. The present RP-HPLC method was validated following the ICH guidelines[18].

Material and Methods

Reference standard of Nifedipine was obtained as gift sample by Medrich Pharmaceuticals Pvt. Ltd., Banglore. Commercial Preparation of Nifedipine was purchased from local market. HPLC grade methanol was procured from Rankem (Mumbai, India). othophosphoric acid AR grade was procured from Qualigens fine chemicals, Mumbai. Double distilled water, prepared in our laboratory was used throughout the experiment. Mobile phase was filtered using 0.45µ nylon filters made by mllipore (USA).

Apparatus and chromatographic conditions

Chromatographic separation was performed on a Shimadzu® liquid chromatographic system equipped with a LC-10AT-vp solvent delivery system (pump), UV detector (Set at 238nm), Rheodyne 7725i injector with 20 µl loop volume. A Shim-Pack CLC, ODS (C-18), (250X4.6 mm i.d., 5 µ) was used for the separation. The mobile phase was composed of mixture of methanol and water (70:30 v/v) and pH 3.0 was adjusted with orthophosphoric acid. It was filtered through a 0.45 µ membrane filter and degassed for 10 mins. Detecetion was carried out at 238 nm

Preparation of standard solution

Standard solution of the pure drug was prepared by dissolving 10 mg of working standard of Nifedipine in a 50 ml volumetric flask using small amount of methanol. Then the volume made up to the mark with the mobile phase. Further dilute 5 ml of this solution to 50 ml with mobile phase. The mixture was sonicated for 5 min or until the reference solution completely dissolves.

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Preparation of sample solution

Twenty tablets were accurately weighed and their average weight was calculated. The tablets were ground using pestle and mortar to a homogenized powder. A quantity of tablet powder equivalent to 10 mg of nifedipine was weighed and transferred into a 50 ml volumetric flask. 30 ml of mobile phase was added and sonicated for 30 minutes for extracting all the drugs from the excipients and to ensure complete solublization of drug and solution make up to 50ml with mobile phase. The excipeints were separated by filteration through a 0.45µm membrane filter. Discard initial few ml and after that 5 ml of filtered solution was diluted up to 50ml with mobile phase.

Before injection, both standard and sample solution was filtered through 0.45µm membrane filter. Inject separately 20 µl of the standard and sample solutions in 3 replicates and measure the response of major peak due to nifedipine. A typical chromatogram obtained from a sample solution is shown in Fig. 1.

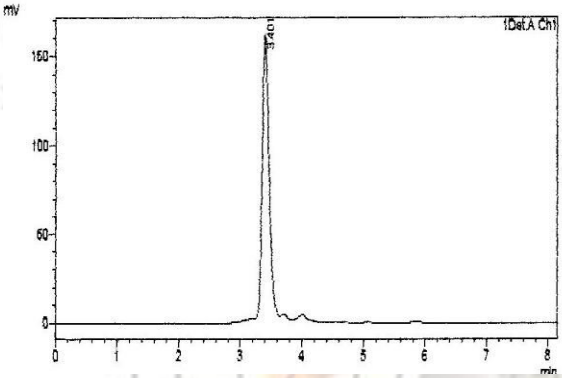

Fig. 1: Typical chromatogram obtained from Nifedipine

Results and Conclusion

Linearity

Linearity was assessed with the aid of serially diluted calibration solutions as mentioned above. The standards were injected separately. Calibration graphs were plotted on the basis of triplicate analysis of each calibration solutions. Linear calibration plot for the proposed method were obtained by analyzing seven solutions in the concentration range of 5-35 µg/ml. The peak area of drugs was potted against the corresponding concentration to construct a linear regression equation and to calculate the value of correlation coefficient. Linear correlations was found to be $y = 361627x + 338075$, ($r^2 = 0.9996$).

Precision

The precision of the method was done by triplicate analysis of tablet preparations. The precision was also studied in terms of intraday changes in peak area of drug solution on the same day and on three different days over a period of one week. The intra-day and inter-day variation was calculated in terms of percentage relative standard deviation and the result was found to be 0.534 and 0.642 for intraday and intra-day respectively.

Accuracy

Accuracy was performed by the method of standard addition at three different levels, by multiple level recovery studies. Three levels of solution were made which correspond to 80, 100 and 120% of the nominal analytical concentration. Each level was made in triplicate. These solutions were then analyzed for recovery studies and consistent values by replicated injections cum analysis. The recovery was found to be 98.85-100.89 % with the value of % RSD less than indicating that the proposed method was accurate.

Limit of Detection and Quantification

Limit of Detection (LOD) and Limit of Quantification (LOQ) were estimated from signal to noise ratio. LOD and LOQ were found to be 0.029 µg/ml and 0.187µg/ml respectively.

Specificity

Specificity was tested against standard compounds and against potential interferences in the presence of placebo. The comparison of the chromatograms of the synthetic placebo mixture and the spiked drug solution revealed that there was no interference of placebo with the peaks of nifedipine in sample solution. No interference from placebo was observed at the retention time of the drugs. Therefore, it was concluded that the method is specific.

System suitability

System suitability tests are an integral part of chromatographic method. They were used to verify that the reproducibility of the chromatographic system is adequate for the analysis. The application of the method was checked by analyzing the nifedipine in commercial tablets. The results are given in table 1 which shows high recovery and low RSD (%) values.

Table 1: System Suitability Data

Parameters	Obtained Value
Tailing Factor	1.374
Retention Time	3.401
No. of Theoretical Plates	3997.311

A RP-HPLC method has been developed for the simultaneous estimation of Nifedipine in pharmaceutical dosage forms, using the UV detector. The proposed RP-HPLC method for assay of nifedipine in tablets dosage forms is simple, precise, specific and highly accurate and less time consumption for analysis could be recorded. So, it can be employed for the routine analysis as method for estimation of nifedipine. Hence this RP-HPLC method is suitable for quality

control of raw materials and formulations, and also for stability studies and dissolution studies.

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