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VALIDATED SPECTROPHOTOMETRIC METHOD FOR THE ESTIMATION OF NIMODIPINE IN BULK AND TABLET DOSAGE FORM

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ABSTRACT

Simple, precise and accurate area under curve spectroscopic method has been developed and validated for the estimation of Nimodipine in bulk and pharmaceutical dosage form. The drug shows maximum absorption (λ_{max}) at 235nm in Acetonitrile solution and Area under Curve [AUC] in absorption spectra were measured between the wavelength range 230 to 240nm which obeys Beer's law in the concentration range of 2-12 µg/ml. The linearity study was carried out and regression coefficient was found to be 0.9998 and it has showed good linearity, precision during this concentration range. The % recovery was found to be 98.85-100.91. The LOD and LOQ were found to be 0.042 and 0.12µg/ml. The % relative standard deviation was found to be less than 2. According to ICH guidelines the method has been validated for linearity, precision, accuracy, robustness, ruggedness, LOD and LOQ. The developed and validated method can be successfully applied for routine estimation of Nimodipine in bulk and pharmaceutical dosage form.

KEYWORDS: Nimodipine, Area under curve spectroscopy, validation, pharmaceutical formulations.

INTRODUCTION

Nimodipine, sold under the brand name Nimotop among others, is a calcium channel blocker used in preventing vasospasm secondary to subarachnoid hemorrhage (a form of cerebral hemorrhage). It was originally developed within the calcium channel blocker class as it was used for the treatment of high blood pressure, but is not used for this indication. Because it has some selectivity for cerebral vasculature, nimodipine main use is in the prevention of cerebral vasospasm and resultant ischemia, a complication of subarachnoid hemorrhage (a form of cerebral bleed), specifically from ruptured intracranial berry aneurysms irrespective of the patient's post-ictus neurological condition.[1]

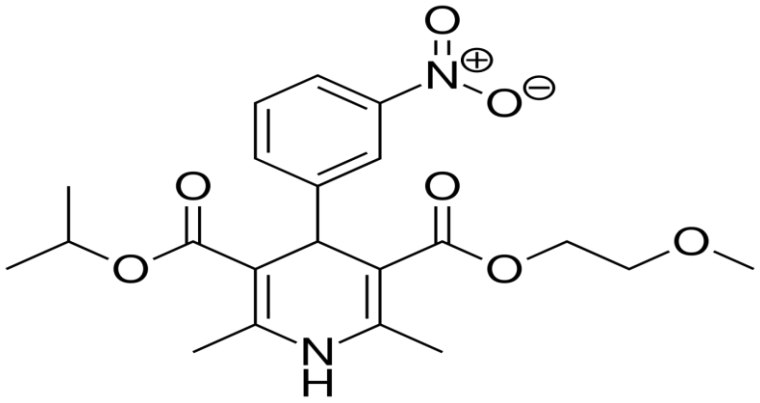

Fig.1: Chemical structure of Nimodipine.

Literature survey revealed that there were few analytical methods have been reported for the determination of Nimodipine in pure drug and pharmaceutical dosage forms by using UV spectrophotometric[2-5], and HPLC[6-11] so far. The aim of present work is to develop and validate a novel, rapid, simple, precise and specific Area

under curve Spectrophotometric method for estimation of Nimodipine in bulk and tablet dosage form.

MATERIALS AND METHODS

Instrument: UV-Visible double beam spectrophotometer, SHIMADZU (model UV-1800) with UV probe software. All weights were taken in analytical balance.

Chemicals: Nimodipine pure drug was obtained as a gift sample from Shree Icon Laboratories Ltd, Vijayawada and its pharmaceutical dosage form Nimodipine 20 tablet labelled claim 30mg from local pharmacy manufactured by USV private Ltd.

Solvent: Acetonitrile is used as a solvent.

Selection of analytical wavelength: Appropriate dilutions of Nimodipine were prepared from standard stock solution and using spectrophotometer solution was scanned in the wavelength range 200-400nm. Area under Curve [AUC] in absorption spectra were measured between the wavelength range 230 to 240nm as the wavelength for detection (Fig-2).

Preparation of standard stock solution: 100mg of Nimodipine was weighed accurately and transferred in to 100ml volumetric flask and diluted in Acetonitrile up to mark. From this, the solution was further diluted into 100µg/ml and pipette out 0.2, 0.4, 0.6, 0.8, 1.0, and 1.2ml into 10ml individual volumetric flask and diluted in Acetonitrile up to mark, this gives 2, 4, 6, 8, 10, and 12µg/ml concentration.

Preparation of sample solution: 20 tablets of Nimodipine marketed formulations were weighed and powdered. A quantity of tablet powder equivalent to 100mg of Nimodipine was transferred into a 100ml of volumetric flask then it was diluted with Acetonitrile and made up to the mark.

METHOD AND VALIDATION: The method was validated according to ICH guidelines.

RESULTS AND DISCUSSION

Method: Area under curve spectroscopy.

Linearity: The linearity of an analytical method is its dimensions to show the test results that are directly proportional to the concentration of the analyte in the sample within the range. The linearity was established in the range of 2-12µg/ml and Area under Curve [AUC] in absorption spectra were measured between the wavelength of 230 to 240nm as absorbance values are shown in table-1 (Fig-3). The calibration curve was prepared by plotting graph against the concentration and absorbance and therefore the graph shown in (Fig-4). Statistical variables like slope, intercept, regression equation, correlation coefficient and Sandell's sensitivity were determined. (table-2).

Precision: The precision of an analytical method expresses the closeness of a series of individual analyte measurements obtained from multiple sampling of the equivalent sample. Precision was determined by intra-day and inter-day study. Intra-day precision was determined by analysing the same concentration for six times in a same day. Inter-day precision was determined by analysing the same concentration daily for six days. (table-3).

Accuracy: The accuracy of an analytical method says that closeness of test results obtained by that method to the true value. To assess the accuracy of the developed method, recovery studies were carried out at three different levels as 50%, 100% and 150%. In which the formulation concentration kept constant and varied pure drug concentration. (table-4).

Ruggedness: The ruggedness is defined as the reproducibility of results when the method is performed under the variation in conditions. This includes different analyst, laboratories, instruments, temperature etc. Ruggedness was determined between different analyst; the value of %RSD was found to be less than 2. (table-5).

LOD and LOQ: The limit of detection is an individual analytical method is the smallest amount of analyte in a sample which can be reliably detected by the analytical method. The limit of quantitation is an individual analytical procedure is the smallest amount of analyte in a sample which can be quantitatively determined. LOD and LOQ were calculated by utilizing fallowing formula.

LOD = 3.3(SD)/S and LOQ = 3(LOD)

LOD and LOQ value of were found Rosuvastatin calcium be 0.042 and 0.12µg/ml.

Table 1: Results of calibration curve at 230-240nm by Area under curve method.

Sl no	Concentration in µg/ml	Absorbance ± Standard deviation*
1	0	0
2	2	0.085±0.000816
3	4	0.173±0.001886
4	6	0.255±0.002672
5	8	0.336±0.001772
6	10	0.428±0.001951
7	12	0.506± 0.002357

*Average of six determinations.

Table 2: Regression parameter for Nimodipine at 230-240nm by Area under curve method.

Regression parameter	Results
Range(µg/ml)	2-18
Detection Wavelengths (nm)	230/240
Regression Equation	Y= 0.0423x+0.0011
Slope(b)	0.0423
Intercept(a)	0.0011
Correlation coefficient(r^2)	0.9998
Sandell's equation	0.023
Limit of detection(µg/ml)	0.042
Limit of quantitation(µg/ml)	0.12

Table 3: Determination of precision results for Nimodipine at 230-240nm by Area under curve method.

Concentration (µg/ml)	Intra-day Absorbance ±Standard deviation*	%RSD**	Inter-dayAbsorbance ±Standard deviation*	%RSD**
2	0.085±0.00157	1.76	0.086±0.0008	0.93
4	0.174±0.002	1.14	0.175±0.0033	1.94
6	0.254±0.002	0.78	0.256±0.0027	1.05
8	0.336±0.001	0.29	0.337±0.0015	0.44
10	0.428±0.0017	0.39	0.427±0.0019	0.44
12	0.508± 0.0018	0.35	0.507± 0.0021	0.41

*****Average of six determinations, **percentage relative standard deviation.

Table 4: Determination of Accuracy results for Nimodipine at 230-240nm by Area under curve method.

Spiked Levels	Amount of Sample (µg/ml)	Amount of Standard (µg/ml)	Amount Recovered	% Recovery ±Standard deviation*	%RSD**
50	6	3	8.91	99.09±0.380	0.383
100	6	6	11.87	98.85±0.361	0.365
150	6	9	15.13	100.91±0.230	0.227

*****Average of six determinations, **percentage relative standard deviation.

Table 5: Determination of Ruggedness results for Nimodipine at 230-240nm by Area under curve method.

Analysts	Analyst 1	Analyst 2
Mean absorbance	0.254	0.255
±Standard deviation*	0.0014	0.0015
%RSD	0.55	0.58

*Average of six determinations, **percentage relative standard deviation.

FIGURES

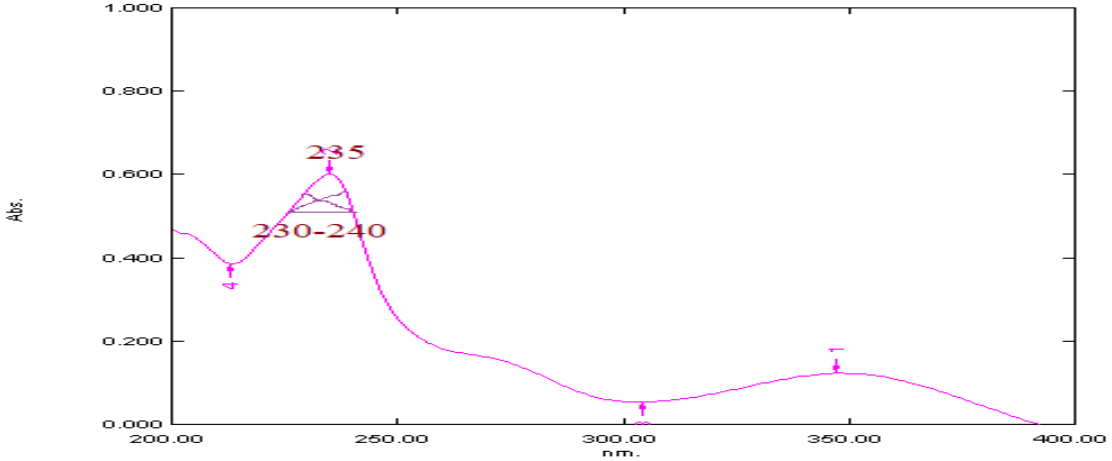

Fig.2: Area under curve spectrum of Nimodipine at 230-240nm.

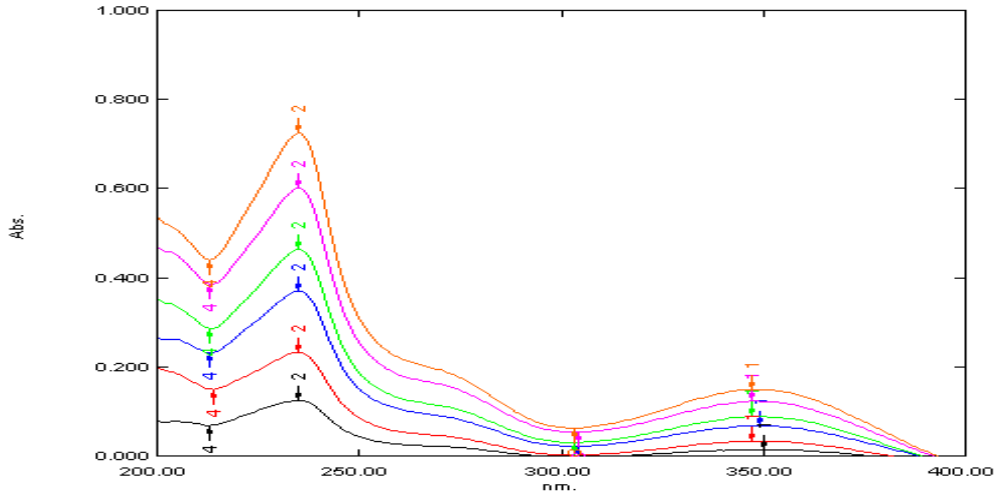

Fig.3: Area under curve overlain spectra of Nimodipine showing absorbance at 230-240nm.

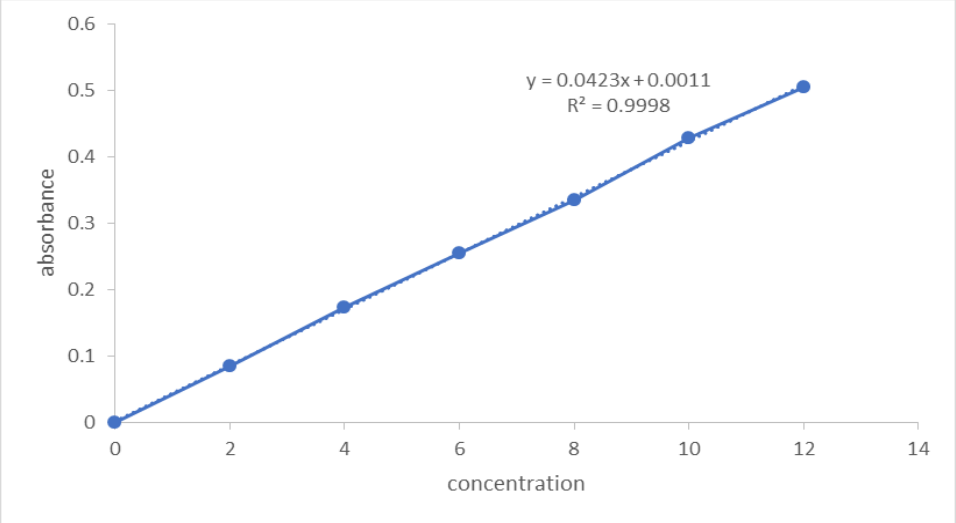

Fig.4: Calibration curve of Nimodipine at 230-240nm by Area under curve.

CONCLUSION

As per ICH guidelines, the developed analytical method meets the acceptance criteria. It was concluded that the method is simple, specific, accurate, economical, sensitive and can be used for routine analysis of Nimodipine in bulk drug and in pharmaceutical dosage form.

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