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Short Communication

Development and Validation of UV Spectrophotometric Method of Nimodipine in Bulk and Tablet Formulation

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ABSTRACT

A simple, sensitive and precise UV spectrophotometric method was developed for the estimation of Nimodipine in tablet dosage form. The optimum conditions for the analysis of the drug were established. The wavelength maxima (λmax) for Nimodipine was found to be 238.5 nm. Beer's law was obeyed in the concentration range of 5-30 mcg/mL having line equation y = 0.033x + 0.020 with correlation coefficient of 0.9981. The slope, intercept, correlation coefficient, detection and quantization limits were also calculated. Results of the analysis were validated statistically and by recovery study. The proposed method can be applied for the routine analysis of Nimodipine from tablet formulation.

Keywords: Nimodipine, UV spectrophotometer, ICH Guidelines.

INTRODUCTION:

Nimodipine is antihypertensive, calcium channel blocker drug. Chemically nimodipine (NM) is isopropyl-2-methoxyethyl-1,4-dihydro-2,6-dimethyl-4-(3-nitrophenyl)-3,5–pyridine dicarboxylate, a dihydropyridine calcium antagonist. It is used mainly in treatment of cerebrospinal haemorrhgae. It is known for its preferential action on cerebral blood vessels and its potential cytoprotective effects by reducing calcium influx into nerve cells [1,2]

Analysis is an important procedure in the formulation and development of any drug molecule. A suitable and validated method must be available for the analysis of drug in the bulk, in drug delivery systems, for dissolution studies as well as *in-vivo* studies.

Nimodipine is official in USP and is determined by liquid chromatographic method with UV detection. Various other analytical methods for analysis are also available includes HPLC[3], LC-MS[4] and GC with electron capture detection[5] but all these analytical techniques requires expensive set up. Quantification of drug also reported in therapeutics includes AAS[6], Spectrophotometry[7], polarography[8] but most of this methods reported are costly, time consuming and less accurate (Eg. Paleography). In addition methods based upon charge transfer complexation reaction using metol-di-chromate[9] and diazotization of reduced nimodipine with nitrous acid followed by coupling with acetylacetone [1] are reported in literature. Considering these demerits, present study was undertaken to develop and validate a simple sensitive, accurate, precise and reproducible UV method for Nimodipine.

MATERIAL AND METHOD:

Nimodipine pure drug was obtain from Cipla Ltd, Mumbai, as a gift sample. Instrument used was Jasko double beam UV/Visible Spectrophotometer and Schimadzu analytical balance. All other chemicals used were of analytical grade.

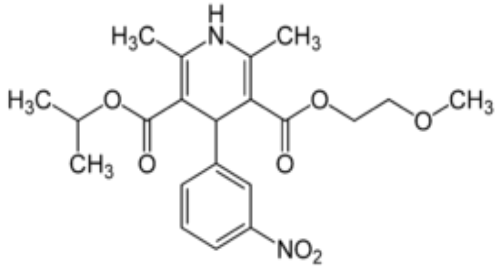

Fig 1. Chemical Structure of Nimodipine

Preparation of standard stock solution:

Standard drug solution of Nimodipine was prepared by dissolving 10 mg of Nimodipine in 10ml of pH 6.8 Phosphate buffer containing 0.05 % w/v of SLS in volumetric flask to give stock solution of 1000 µg/ml. 1ml of stock solution was withdrawn and further diluted with pH 6.8 Phosphate buffer containing 0.05 % w/v of SLS solution to give stock solution of 100 µg/ml concentration.

Preparation of calibration curve:

Calibration curve was prepared in pH 6.8 Phosphate buffer containing 0.05 % w/v of SLS solution at λmax of 238.5 nm by using Jasko UV-Visible spectrophotometer. For this stock solution of 100 µg/ml was prepared. Serial dilutions of 5, 10, 15, 20, 25 and 30 µg/ml were prepared and absorbance was taken at 238.5 nm. The solutions were scanned in the range of 200-400 nm against blank. The calibration curve was plotted. The optical characteristics are summarized in Table no. 1

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Sr. no.	Concentration (µg/ml)	Absorbance
1	5	0.169
2	10	0.362
3	15	0.523
4	20	0.668
5	25	0.854
6	30	0.997

Table no. 1: Calibration Curve

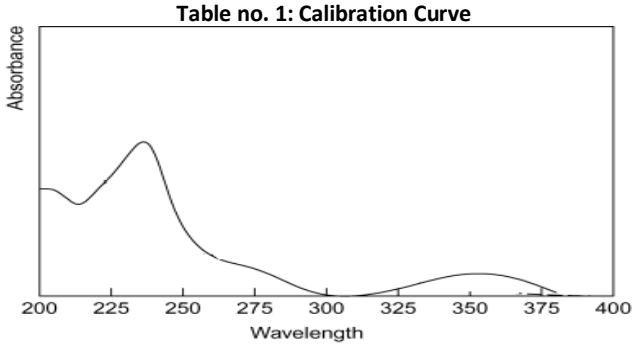

Fig 2: Determination of λmax of Nimodipine by UV spectrophotometer

Analytical method validation[10,11]

Precision:

The reproducibility of the proposed method was determined by performing at different time intervals on the same day (intra-day precision) and on three different days (inter-day precision).The results of intra-day and inter-day precisions were expressed in % RSD. The % RSD for intra-day precision was found to be 0.978 and inter-day precision was 0.775.

Sample no	Inter-day precision	Intra-day precision
1	1.015	0.775
2	0.839	1.180
3	0.470	0.980
Average %RSD	0.775	0.978

Table no. 2 Determination of Precision

Accuracy

Accuracy of the method was studied by recovery experiments. Recovery experiments were performed by adding known amount to tablet. The recovery was performed at three levels, 80%, 100% and 120% of Nimodipine standard concentration. The recovery samples were prepared in mentioned procedure. Three samples were prepared for each recovery level. The solutions were then analyzed, and the percentage recoveries were calculated from the calibration curve. The recovery values for Nimodipine ranged from 101.001±1.01 (Table no.3).

Drug	Amount (mg)	Level of Addition (%)	Amount added (mg)	Drug found (µg/ml)	% Recovery	Average % Recovery
Nimodipine (Nimodip)	30	80	24	16.31	101.93	101.001
	30	100	30	20.23	101.15	
	30	120	36	23.98	99.64	

Table no. 3 Determination of Accuracy by percentage recovery method

Linearity:

The linearity of the response of the drug was verified at 5 to 40 µg/ml concentrations, but linearity was found to be between 5-30 µg/ml concentrations. The calibration graphs were obtained by plotting the absorbance versus the concentration data and were treated by linear regression analysis (Table no. 4). The equation of the calibration curve for nimodipine obtained y = 0.033x +0.020, the calibration curve was found to be linear in the above mentioned concentrations. The correlation coefficient (r^2) of determination was 0.9981.

Limit of detection (LOD) and Limit of quantitation (LOQ):

The LOD and LOQ of Nimodipine were determined by using standard deviation of the response and slope approach as defined in International Conference on Harmonization (ICH) guidelines 6.The LOD and LOQ was found to be as in table no.4.

Determination of Active Ingredients in Tablets:

The validated method was applied to the determination of Nimodipine in Tablets. Ten tablets were assayed and the results are shown in (Table no. 3) indicating that the amount of drug in tablet samples met with requirements (99–102% of the label claim).

Sr. No.	Parameter	Result
1	Absorption maxima(nm)	238.5
2	Linearity Range (µg/ml)	5-30
3	Standard Regression Equation	y = 0.033x +0.020
4	Correlation Coefficient (r2)	0.9981
5	Molar absorptivity	0.72*104
6	Accuracy (% recovery ±SD)	101.001
7	Precision	0.775(Inter-day precision), 0.978 (Inter-day precision)
8	LOD (µg/ml)	0.7469
9	LOQ (µg/ml)	2.26
10	Sandell's Sensitivity	0.029

Table no.4. Validation Parameters

CONCLUSION:

The developed method was found to be simple, sensitive, accurate, precise, reproducible, and can be used for routine quality control analysis of nimodipine in bulk and pharmaceutical formulation.

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REFERNCES:

1. Hosakere D., Shiramahally M., Hemavathi N. and Kanakapura B., Spectrophotometric Methods for the determination of Nimodipine in Pure and in Pharmaceutical Preparations, Jordan Journal of Chemistry Vol. 6 No.4, 2011, pp. 413-422.
2. Nimotop FDA Approved labeling text, NDA 18-869/S-014.
3. Yang, D. B.; Zhu, J. B.; Lv, R. Q.; Hu, Z. G.; Shen, J. Q., *Anal. Lett.,* 2008, 41, 533-542.
4. Mueck, W. M., *J. Chromatogr.,* 1995, 712, 45-53.

5. Jakobsen, P.; Mikkelsen, E. O.; Laursen, J.; Jensen F., *J. Chromatogr*., 1986, 374, 383–387.
6. Canlica, M.; Islimyeli, S., *Turk. J. Chem.,* 2005, 29, 141-146.
7. Squella, J. A.; Sturm, J. C.; Lenac, R.; Nunez-Vergara, L. J., *Anal. Lett.,* 1992, 25 281-292.
8. Bharathi, S. N.; Prakash, M. S.; Nagarajan, M.; Kumar, K. A., *Indian Drugs*, 1999, 36,661-662.
9. Hosakere D., Shiramahally M., Hemavathi N. and Kanakapura B, Spectrophotometric Determination of Nitrazepam and Nimodipine in pure and the Tablet Dosage Forms, Asian Journal of Biochemical and Pharmaceutical Research Issue 1 (Vol. 1) 2011.
10. Panzade P.,Puranik P., Mogal V.and Patnni M., Development and Validation of UV spectrophotometric Method of Granisetron Hydrochloride in Bulk and Pharmaceutical Formulation, Asian J. Research Chem. 3(3): July- Sept. 2010.
11. Validation of Analytical Procedures, Methodoly, ICH Harmonised tripartite guidelines; 1996:1.

Conflict of Interest: None Declared

