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Analytical method development and validation for the estimation of nimodipine in pure and marketed formulation by UV-Spectrophotometric method

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Abstract
A Simple, specific, rapid, precise and accurate UV Spectrophotometric method was developed and for the estimation of Nimodipine in tablet dosage form. The optimum conditions for the analysis of the drug were established.The wavelength maxima for Nimodipine was found to be 239nm.Beer's law was obeyed in the concentration range of 5-25μg/ml having line y=0.033x + 0.021 with correlation coefficience of 0.9995.The slope, intercept, correlation coefficient, detection and quantization limits were also calculated. Results of the analysis were validated satistically and by recovery study. The proposed method can be applied for the routine analysis of Nimodipine from the tablet formulation.

Keywords: nimodipine, UV spectrophotometer, ICH guidelines

Introduction
Nimodipine is cardio selective calcium channel blocker, an Anti-hypertensive drug being used for cerebrospinal hemorrhage. Nimodipine is well known for its significant action on cerebral blood vessels and its potential cytoprotective effects by reducing calcium influx into nerve. The IUPAC name is 3, 5-Pyridinedicarboxylic acid, 1, 4-dihydro-2, 6-dimethyl-4-(3- nitro phenyl)-, 2-Methoxyethyl 1-Methylether ester.

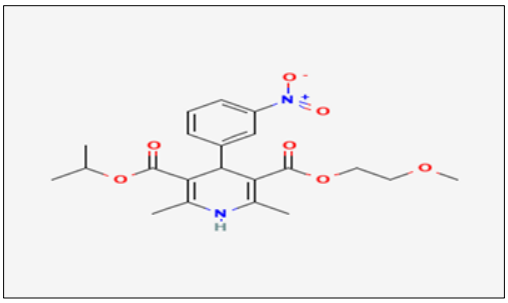

Fig 1: structure of Nimodipine

Methodology
Selection of Solvent
The solubility of Nimodipine was determined in a variety of solvent as per Indian Pharmacopoeia standards. Solubility test was carried out in different polar and non-polar solvents from the solubility studies. Methanol and Water (60:40) V/V was selected as suitable solvent for proposed method.

Preparation of Standard Stock Solution
Standard drug solution of Nimodipine was prepared by dissolving 10mg of Nimodipine in 10ml of methanol: water (60:40v/v) in volumetric flask to give stock solution of 1000 µg/ml.1ml of stock solution was withdrawn and future diluted with 10ml of methanol: water(60:40)v/v in volumetric flask to give stock solution of 100 µg/ml concentration.

Preparation of sample solution
Accurately weighed of 10 tablets, Average weight is taken and powerded amount equivalent to 10 µg/ml weighed and transferred into 10ml of volumetric flask and made upto mark to make mobile phase. This solution was filtered through Whatsmann filter paper number 40.From the above solution 1ml is taken and future diluted in 10 ml volumetric flask with mobile phase to acqurate acceleration of 100 µg/ml of Nimodipine.

Determination of Absorbance Maxima (λ max)
The standared solution having concentration 10 µg/ml was scanned at 200-400nm with dilutent and blank to detect maximum wavelength. After through the wavelength 239 nm was seen from above spectra of Nimodipine wavelength maxima identified of 239nm (λ max) as shown in Fig-2.

Determination of concentration range
Five levels of five different concentrations, liquate of standared solutions of sutable concentrations of Nimodipine were transferred into a series of 10ml standared volumetric flask and volumes are made upto the mark with methanol: water(60:40v/v). Five dilution concentrations were prepared into range 5-25 µg/ml and the absorbance were measured at 239nm against diluent as blank. The uptained values are plotted against the concentration of Nimodipine to get the calibration curve.

Precision
The precision of an analytical method is the degree of agreement among individual test results when the method is applied repeatedly to multiple sarplay of homogenous samples.

Intra-day and inter-day precision

A variation of results within the same day (intra- day), variation of results between days (inter- day) was analyzed. Intraday precision was determined by analyzing Nimodipine (15 µg/ml) For six times in the same day at 239nm. Inter day precision was determined by analyzing the drug daily twice for three days at 239 nm.

Recovery studies

Accuracy of the method was studied by recovery experiments. Recovery experiments were performed by adding known amount of tablet. Recovery studies carried out by addition of standard drug solution (50%, 100% and 150 %) to the sample at 3 different concentration levels.

Aliquots of 1.5 ml of sample drug solution of 100 mg/ml were pipetted into each of three 10ml volumetric flasks (15 ug/ml), to all the 3 volumetric flask 0.75ml (7.5 ug/ml), 1.5ml (15 ug/ml) and 2.25 ml (22.5 ug/ml) of standard solution of 100mg/ml was added respectively, the volume was made up to 10ml with mobile phase solution and the absorbance was measured at 239nm against dilute as blank.

Limit of Detection (LOD) and Limit of Quantification (LOQ)

Limit of detection and quantification limit were calculated by the method based on the standared on the standared dilution (σ) and slope of the calibration plot, usinf the formula.

$$\text{Limite of detection} = \frac{\sigma \times 10}{s}$$

$$\text{Limit of Quantification} = \frac{\sigma \times 3.3}{s}$$

σ= standared deviation of response

S= slope of the calibration curve

Result and Discussions

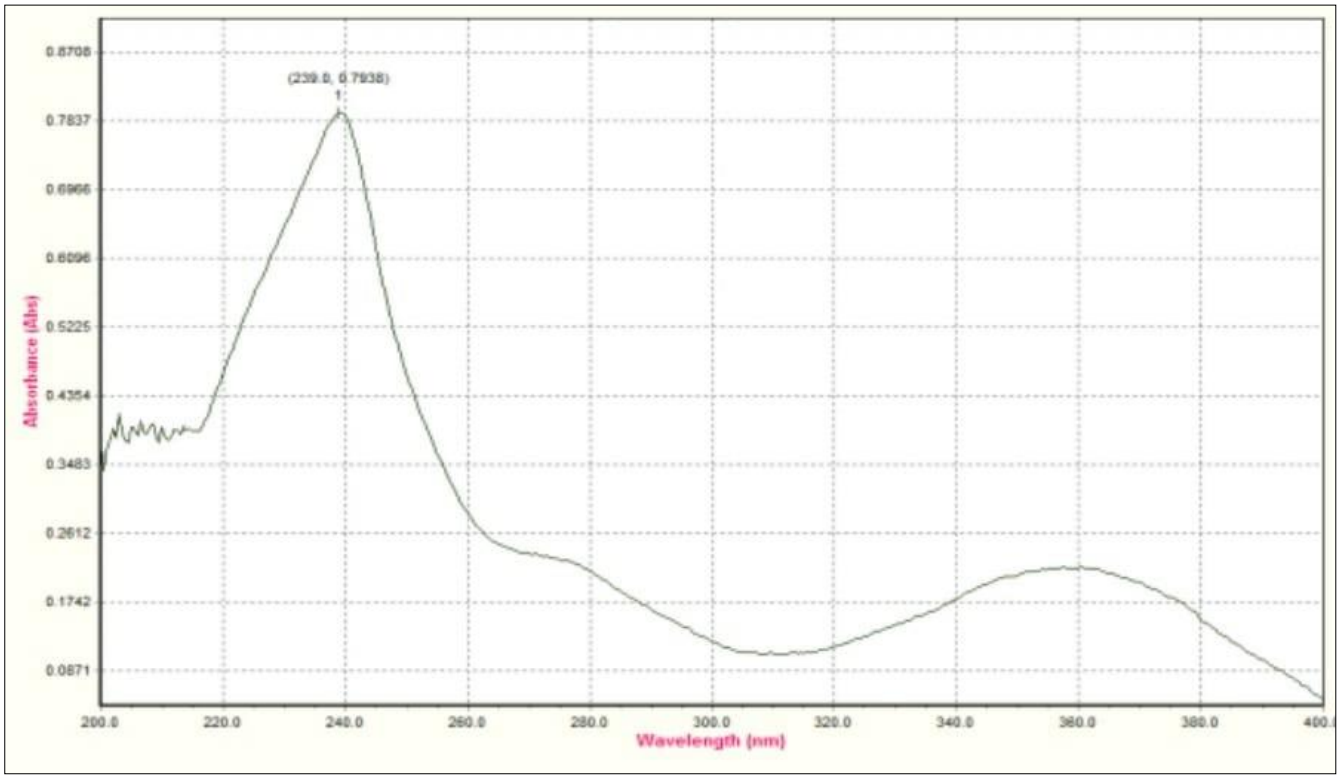

Fig 2: Estimation of Absorption spectra of Nimodipine

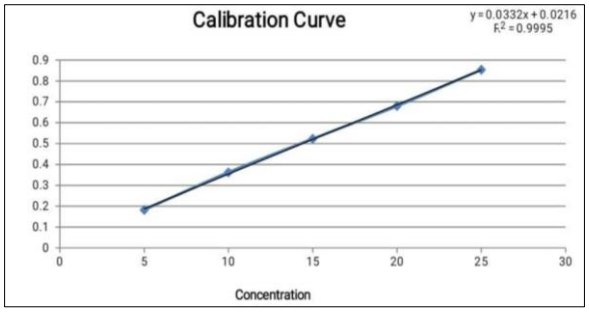

Fig 3: Calibration curve of Nimodipine

Table 1: Optimized Chromatographic conditions

Parameters	Value
λ max (nm)	239
Beers iaw limit (***µ***g/ml)	5-25
Correlation coefficient(r2)	0.9995
Regression equation(Y)	Y=0.033× +0.021
Slope(m)	0.033
Interecept (c)	0.021
LOD (µg/ml)	1.2
LOQ (µg/ml)	3.6

Table 2: Inter-day Precision Data

S.no	Amount (µg/ml)	Amount found	% Recovery	Average % Recovery	SD	RSD
1	15	15.3	102	100.1	0.132	0.88
2		14.7	98.3			
3		15.1	100.6			
4		14.9	99.3			
5		14.9	99.3			
6		15.1	100.6			

Table 3: Recovery Data

S.no	% Added	Amount (µg/ml)	Amount Added	Amount found	% Recovered	Avg % Recovered	SD	RSD
1	50	15	7.5	7.6	108.4	100.2	0.0 28	0.325
2			7.5	7.3	98.1			
3			7.5	7.5	100.6			
1	100		15	15.1	100.1	99.3	0.048	0.322
2			15	14.8	98.6			
3			15	14.9	99.5			
1	150		22.5	22.43	99.7	99.1	0.024	0.108
2			22.5	22.15	98.4			
3			22.5	22.35	99.2			

Table 4: LOD&LOQ

S. No	Parameter	Concentration
1	LOD	1.2
2	LOQ	3.6

Table 5: Assay

S.no	Label claim	Amount found	percentage
1	30mg	29.6mg	98.6

Conclusion

In this study a simple, precise, accurate and sensitive UV-spectroscopy methods were developed for estimation of Nimodipine bulk and in tablet dosage form. The Correlation coefficient (γ) values of the proposed method was close to 1.0, it indicate that the concentration used for plotting calibration curve were obeying Beer's law strictly. Additives and impurities commonly present in the dosage forms but did not show any interference in the proposed method. Statistical validation was done it shows that the method was reproducible and accurate. Also the various parameters were calculated such as standard deviation and percentage relative standard deviation. The values are complies all the limit as per ICH guidelines

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