UV SPECTROPHOTOMETRIC METHOD DEVELOPMENT AND VALIDATION OF NIFEDIPINE IN BULK AND FORMULATION

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ABSTRACT:

Objective: A new, simple, sensitive, precise and reproducible UV spectroscopic method was developed for the estimation of Nifedipine in bulk and Formulation.

Methods: The UV spectrum of Nifedipine in phosphate buffer ph7.4 showed λ max at 213nm. Beer's law is valid in the concentration range of 5-25µg/ml. This process was authenticated for linearity, accuracy, precision, ruggedness and robustness.

Results: The method has demonstrated excellent linearity over the range of 5-25µg/ml with regression equation y = 0.025x + 0.014and regression correlation coefficient r = 0.996. Moreover, the method was found to be highly sensitive with LOD (5.64µg/ml) and LOQ (1.86µg/ml).

Conclusion: Depending on results the given method can be successfully applied for assay of Nifedipine in Semisolid formulation.

Keyword: Nifedipine, UV spectroscopy, method development and validation, Phosphate buffer ph7.4, Formulation.

INTRODUCTION:

Nifedipine is a used for the treatment of calcium channel blocker used as an anti-hypertensive agent. The anti-hypertensive agent prevents the complication of high blood pressure, such as stroke and myocardial infarction. Nifedipine is the first generic medication. It was discovered by Bayer in 1972. Nifedipine is a BCS class II drug having low solubility and high permeability. Nifedipine is almost completely absorbed in the gastrointestinal tract.

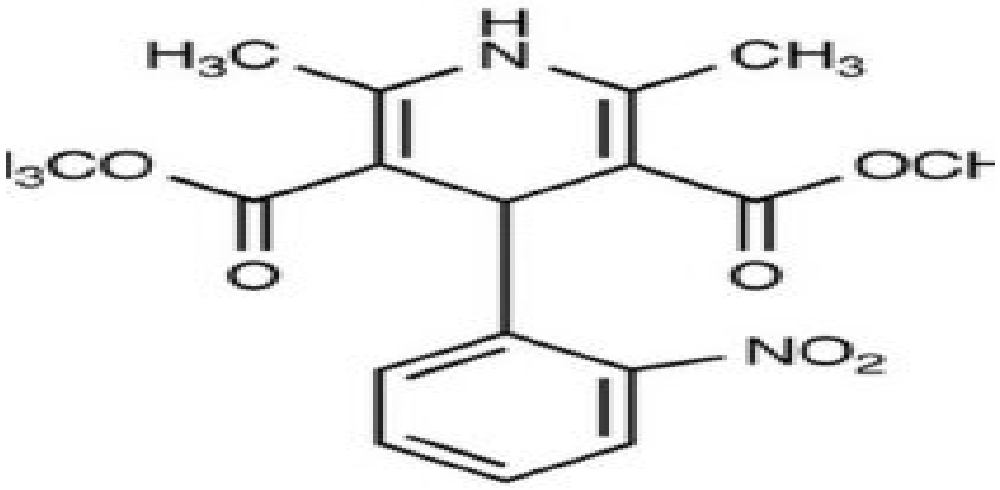

Figure1: Structure of Nifedipine

The Chemical name of Nifedipine is 3,5-dimethyl 2,6-dimethyl-4-(2-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate. The molecular formula of Nifedipine is $C_{17}H_{19}CIN_2O_6$ and molecular weight is 382.8 gm/mol. It is freely soluble in acetone and in chloroform; sparingly soluble in ethanol; The aim of this study is to give a new, simple, sensitive, precise and reproducible UV spectroscopic method was developed for the estimation of Nifedipine in bulk and formulation.

MATERIALS AND METHOD:

Materials

Nifedipine was taken as gift sample from Sun Pharma, Solapur. RO water was taken from local market.

Instruments:

Analytical balance (Shimadzu AY220), Sonicator(Microclean-1103), UV-Visible spectrophotometer (Systronic 2201).

Experimental:

Preparation of standard stock solution:

Accurately weighed 10mg of Nifedipine transferred to 100ml volumetric flask. It was dissolved in RO water & sonicated for 5 minutes. The volume was made up to mark with same diluent to make up final strength.

Procedure for plotting calibration curve:

For calibration curve in a series of 10 ml volumetric flasks, 2-10ml of standard solution was pipetted out separately. The volume was completed to the mark using RO water. The absorbance was measured at wavelength 213 nm against blank solution.

RESULTS AND DISCUSSION:

The absorption spectrum shows λ max of Nifedipine at 213nm.

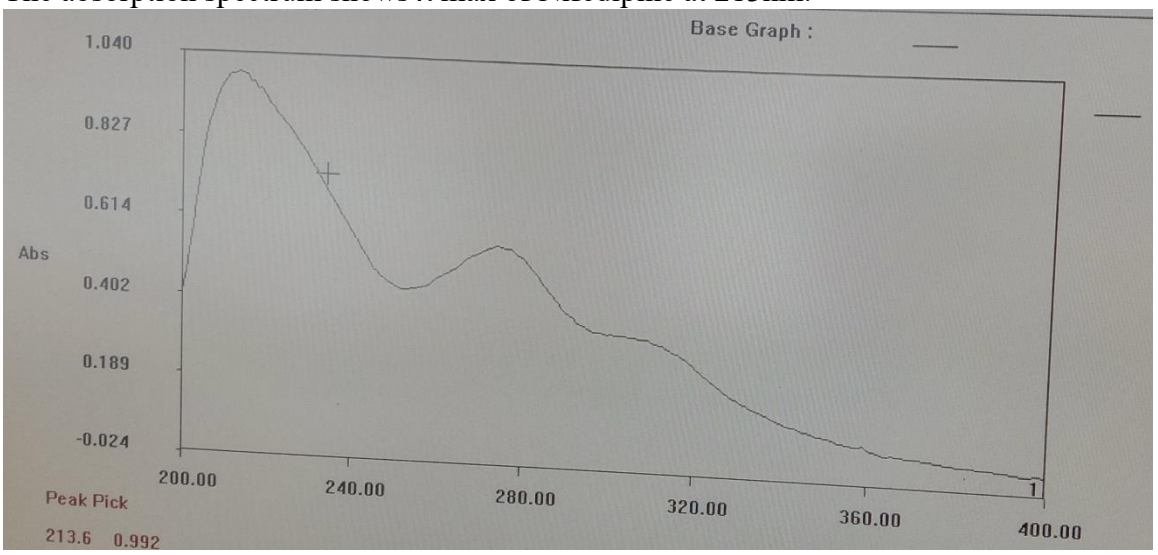

Figure 2: UV spectrum of Nifedipine

The proposed method was validated according to ICH Q28 R1 guidelines for validation of analytical procedure.

Linearity:

Five different concentrations of Nifedipine were prepared and analyzed at wavelength 213 nm. The regression coefficient was found to be 0.996. The absorbance was found in limit i.e., 0-2. Hence the analysed parameter was found to be validated (table 1).

Table 1: Results of Linearity

Sr.no.	Concentration(µg/ml)	Absorbance
1	5	0.155
2	10	0.26
3	15	0.401
4	20	0.531
5	25	0.629

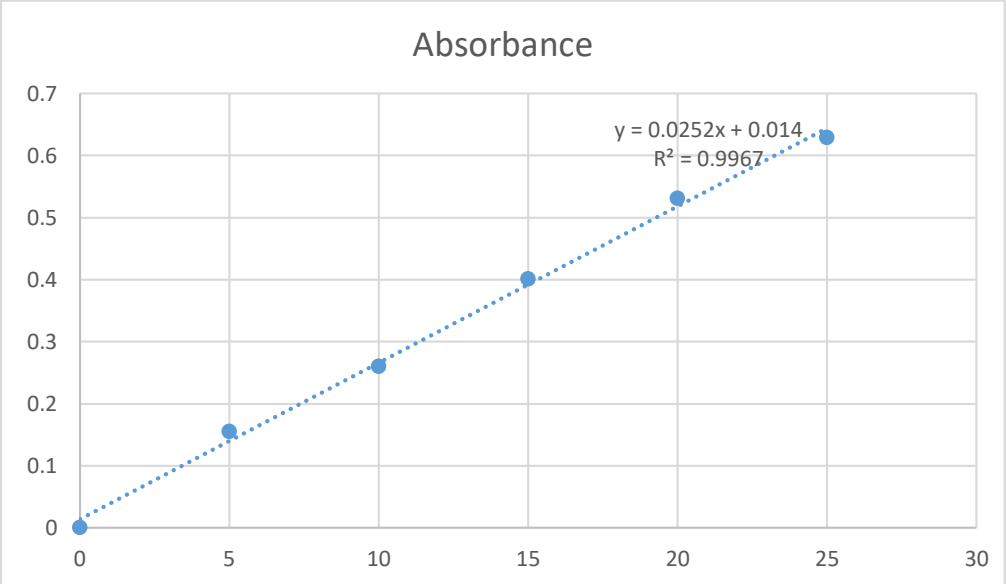

Figure 3: Calibration curve for Nifedipine (Conc.vs.Abs.) Table 2: Optimization parameters of Nifedipine

Parameters	**Method values**
Maximum Wavelength	213nm
Beer's Law	5-25µg/ml
Correlation Coefficient (r^2)	0.996
Regression Equation	y = 0.025x+0.014
Slope (m)	0.025
Intercept (c)	0.014

Accuracy:

The concentration 20,40,60µg/ml was taken as 50,100,150% and % recovery was found to be in range 99%-101%. Hence the parameter was found to be validated.

Table 3: Results of Accuracy

Name of Drug	Recovery Level in %	Concentration	Amount Recovered	% recovery with SD
Nifedipine	50	20µg/ml	20.02	100.02±0.25
	100	40µg/ml	40.03	100.03±0.7
	150	60µg/ml	59.01	99.01±0.29

Range: Range is an interval between highest and lowest concentration limit of the analyte i.e., 5-25µg/ml.

Precision:

In precision intra-day and inter-day precision were performed at concentration (15µg/ml). The obtained results were found within limit i.e., less than 2%RSD.

Table 4: Results of Intra-day Precision

Sr. no.	Concentration	Absorbance
1	(15µg/ml)	0.401
2		0.402
3		0.401
4		0.400
5		0.401
6		0.402
	SD	**0.000753**
	%RSD	**0.187646**

Table 5: Results of Inter-day precision

Sr.no.	Concentration	Absorbance (Day1)	Absorbance (Day2)
1	(15µg/ml)	0.401	0.402
2		0.402	0.401
3		0.401	0.400
4		0.402	0.401
5		0.402	0.402
6		0.401	0.401
	SD	**0.000548**	**0.000753**
	%RSD	**0.136419**	**0.187646**

Limit of Detection (LOD):

The limit of detection was found to be 5.64µg/ml (table 6).

Limit of Quantification (LOQ):

The limit of quantification was found to be 1.86µg/ml (table 6).

Table 6: Results of LOD and LOQ

LOD	5.64µg/ml
LOQ	1.86µg/ml

Ruggedness:

The revolution in analyst with similar concentration and environmental condition didn't affect the results.

Table 7: Results of Ruggedness

Concentration	Absorbance (Analyst1)	Absorbance (Analyst2)
5µg/ml	0.155	0.156
	0.156	0.154
	0.155	0.157
	0.157	0.155
	0.154	0.156
	0.156	0.155
Average	**0.1555**	**0.1555156**
SD	**0.001049**	**0.001049**

Assay:

Sample solution of concentration 50µg/ml was analyzed at wavelength 213nm and the %purity was calculated.

Table 9: Results of Assay

Formulation	Labeled Amount	Amount obtained	% purity
Nicardia Retard	1 gm	97.98	97.98%

CONCLUSION:

An analytical UV spectrophotometric method was developed & validated thoroughly for quantitative determination of Nifedipine in bulk drug and formulation. The presented method was found to be simple, precise, accurate, rugged, reproducible and gives an acceptable recovery of the analyte, which can be directly easily applied to the analysis of pharmaceutical formulation of Nifedipine.

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