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UV SPECTROPHOTOMETRIC METHOD DEVELOPMENT AND VALIDATION OF AZELNIDIPINE IN BULK AND DOSAGE FORM

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ABSTRACT:

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

Methods: The UV spectrum of Azelnidipine in acetonitrile showed λ max at 251 nm. Beer's law is valid in the concentration range of 5-25µg/ml. This method was validated 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 = 0.056x + 0.0593 and regression correlation coefficient r^2= 0.9993. Moreover, the method was found to be highly sensitive with LOD (0.991µg/ml) and LOQ (3.00µg/ml).

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

KEYWORD: Azelnidipine, UV spectroscopy, method development and validation, λ max.

1. INTRODUCTION:

Hypertension is a condition where blood pressure is elevated to an extent that clinical benefit is obtained from BP lowering. Hypertension is one of the most important risk factor for both coronary artery disease and cardiovascular disease [1]. Azelnidipine (AZEL) (3-[1-(diphenylmethyl)azetidin-3-yl] 5-propan-2-yl 2-amino-6-methyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate) is a new dihydropyridine derivative with calcium antagonistic activity. Azelnidipine is inhibits trans membrane Ca+2 influx through the voltage dependent channels of smooth muscle in vascular walls. They enter the cells through cell membrane, lower peripheral vascular resistance and arterial pressure. It is used for treatment of essential hypertension and angina pectoris [2]. Azelnidipine is Ca+2 channel blocker inhibits trans membrane Ca +2 influx through the voltage dependent channels of smooth muscle in vascular walls. They enter the cells through cell membrane, lower peripheral vascular resistance and arterial pressure.Ca+2 channels are classified into various categories including L-type, T-type, N-type, P/Q- type, R-type Ca+2 channels. Normally, calcium induces smooth muscle contraction, contributing to hypertension. When calcium channels are blocked, the vascular smooth muscle does not contract, resulting in relaxation of vascular smooth muscle walls and decreased BP [3].

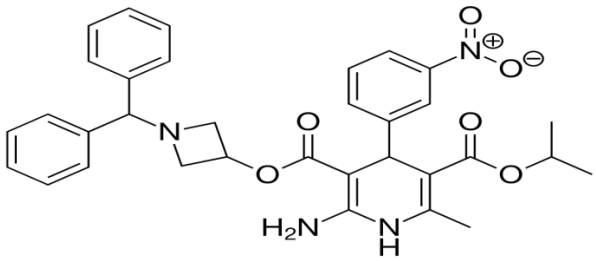

Figure1: Structure of Azelnidipine[4]

The drug is official in Indian Pharmacopiea. A few analytical methods have been reported for the determination of the selected drug. The reported methods for estimation of azelnidipine were UV-Spctrophotometric methods [5,6]. The aim of this study is to give a new, simple, sensitive, precise and reproducible UV spectroscopic method for the estimation of Azelnidipine in bulk and formulation.

2.0 MATERIALS AND METHOD:

2.1 Materials

Azelnidipine was taken as gift sample from Pure Chem Pvt Ltd. . Acetonitrile was taken from Merck Specialities Private Limited.

2.2 Instruments:

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

3.0 Experimental:

3.1 Preparation of standard stock solution:

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

3.2 Procedure for plotting calibration curve:

For calibration curve in a series of 10 ml volumetric flasks, 5-25ml of standard solution was pipetted out separately.The volume was completed to the mark using Acetonitrile. The absorbance was measured at wavelength 251 nm against blank solution.

3.3 Analysis of Azelnidipine in Formulation:

10 mg equivalent Azelnidipine tablet was weighed and transferred to the 50ml volumetric flask and dissolved in acetonitrle as a solvent. After that sonicated for 5min and vortex for 2min. 1 ml of above solution was pipetted out and transferred to the 10ml volumetric flask and make up the volume upto the mark with same solvents and analysed at 251nm. Calculate the % purity of Azelmidipine.

4.0 RESULTS AND DISCUSSION:

The absorption spectrum shows λ max of Azelnidipine at 251nm. Optimization parameters of Azelnidipine were as follows :
Maximum Wavelength : 251nm.
Beer's Law : 5-25 µg/ml
Correlation Coefficient (r^2) : 0.9993
Regression Equation : y=0.056x + 0.0593
Slope (m) : 0.056
Intercept (c) : 0.0593

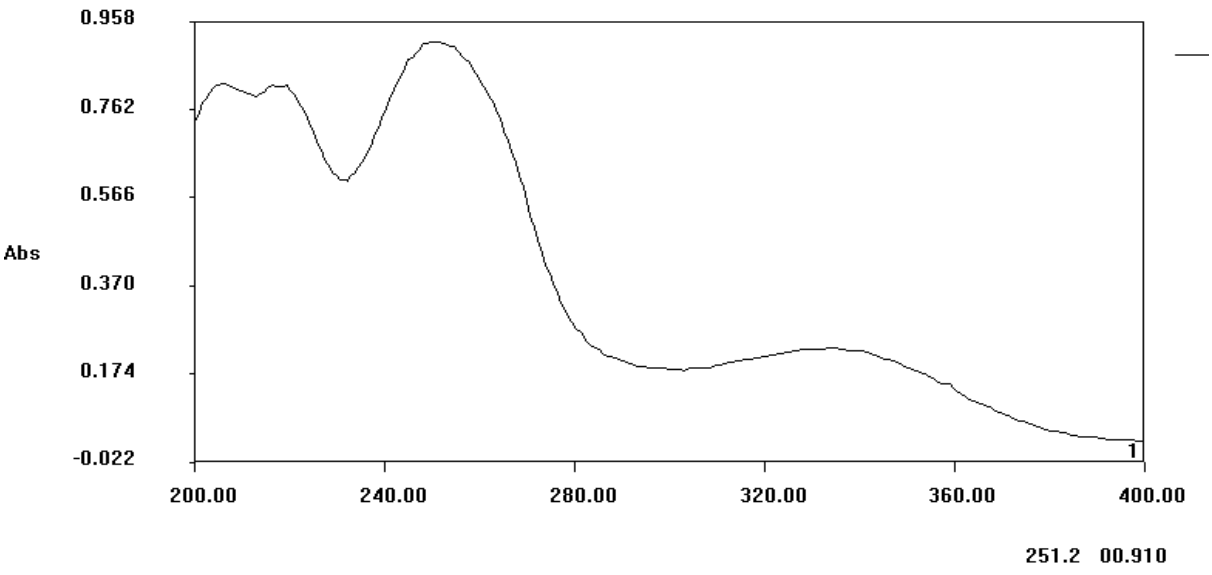

Figure 2: UV spectrum of Azelnidipine.

The proposed method was validated according to ICH Q28 R1 guidelines for validation of analytical procedure[7].

4.1 Linearity:

Five different concentrations of Azelnidipine were prepared and analysed at wavelength 251nm. The regression coefficient was found to be 0.9993. The absorbance was found in limit i.e. 0-2. Hence the analysed parameter was found to be validated .

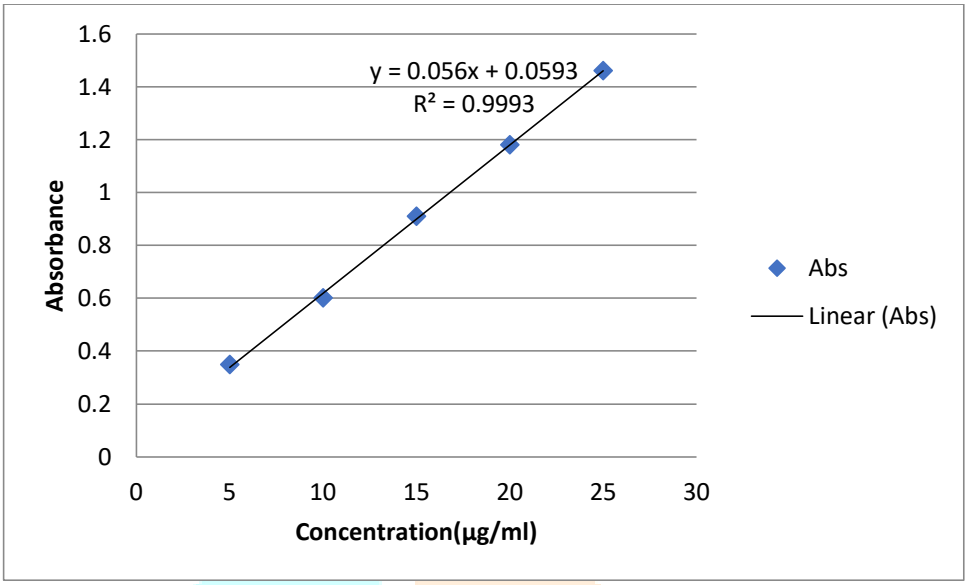

Figure 3: Calibration curve for Azelnidipne (Conc.vs.Abs.)

4.2 Accuracy:

The concentration 12,15,18µg/ml was taken as 80,100,120% and % recovery was found to be in range 99%-102%. Hence the parameter was found to be validated.

Table 1: Results of Accuracy

Name of Drug	Recovery Level in %	Theoretical Concentration	Obtained Concentration	% recovery
Azelnidipine	80	12µg/ml	12.28	102.3
	100	15µg/ml	14.99	99.9
	120	18µg/ml	18.42	02.2

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

4.4 Precision:

In precision intra-day and inter-day precision were performed at concentration (20µg/ml). The obtained results were found within limit i.e. less than 2%RSD.

Table 2: Results of Inter-day and Intra-day precision

Sr.no.	Concentration	Absorbance	
	(20µg/ml)	Inter-day	Intra-day
1		1.192	1.183
2		1.183	1.194
3		1.194	1.193
4		1.19	1.185
5		1.192	1.198
6		1.193	1.192
	SD	**0.003**	**0.005**
	%RSD	**0.33**	**0.479**

4.5 Limit of Detection (LOD):

The limit of detection was found to be 0.991 µg/ml .

4.6 Limit of Quantification (LOQ):

The limit of quantification was found to be 3.00µg/ml .

4.7 Ruggedness:

The change in analyst with same concentration and environmental condition didn't affect the results.

Table 3: Results of Ruggedness

Concentration	Absorbance (Analyst1)	Absorbance (Analyst2)
20µg/ml	1.193	1.192
	1.192	1.198
	1.19	1.185
	1.192	1.193
	1.194	1.194
	1.183	1.183
Average	**1.1906**	**1.1908**
SD	**0.003**	**0.005**

4.8 Robustness:

The change in wavelength (249nm and 253nm) didn't affect the results.

Table 4: Results of Robustness

Wavelength	249nm	253nm
Concentration	20µg/ml	20µg/ml
Absorbance	1.188	1.181
	1.185	1.181
	1.188	1.178
Average	**1.187**	**1.18**
SD	**0.0017**	**0.0017**

4.9 Assay:

The assay was performed by using Azovas 16mg Tablet at concentration 20µg/ml. The % purity was found to be 97.35%.

5.0 Acknowledgement:

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6.0 Conclusion: An analytical UV spectrophotometric method was developed & validated thoroughly for quantitative determination of Azelnidipine 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 Azelnidipine.

7.0 References:

1. Walker R and Whittlesea C. Clinical Pharmacy and Therapeutics; 5thEdn; Elsevier (2007): 295-307.
2. Chen B., et al. "Clinical use of azelnidipine in the treatment of hypertension in Chinese patients". Therapeutics and Clinical Risk Management 11 (2015): 309-318.
3. Tamargo J and Ruilope LM. "Investigational calcium channel blockers for the treatment of hypertension". Expert Opinion on Investigational Drug (2016): 1-51.
4. Azelnidipine image [Internet]. wikipidia [cited 2020 Nov 6]. Available from: https://en.wikipedia.org/wiki/Azelnidipine#/media/File:Azelnidipine_structure.svg
5. Rajan R. Spectrophotometric estimation of azelnidipine in bulk and pharmaceutical dosage form by second order derivative methods. J Chem Pharm Res. 2014;6(8):198-202.
6. Kunti R, Mrunali P, Anandkumari C. Uv-Spectrophotometric Method Development and Validation for Determination of Azelnidipine in Pharmaceutical Dosage Form. Int J Pharm Pharm Sci. 2012;4(1):238-240.
7. ICH Q2 (R1) validation of analytical procedures: twext and methodology, International Conference on Harmonization, Nov,1996.