International Journal of Trend in Scientific Research and Development (IJTSRD)
Volume 6 Issue 5, July-August 2022 Available Online: www.ijtsrd.com e-ISSN: 2456 – 6470

Analytical Method Development and Validation for the Estimation of the Azilsartan Medoxamil by RP-HPLC Method in Bulk & Pharmaceutical Dosage Form

Nakul Arjun*, Vikram Veer, Dr. Ashok Bhosale

Department of Pharmaceutical Quality Assurance, Pune District Education Association's
Shankarrao Ursal College of Pharmaceutical Science & Research Center, Pune, Maharashtra, India

ABSTRACT

In this study, a straightforward, accurate, and sensitive RP-HPLC method was created and validated for the verification of the Azilsartan medoxamil. It is an angiotensin II receptor antagonist used to treat adult patients with high blood pressure.

Results of the analysis were good after being validated for accuracy, precision, LOD, and LOQ.

The suggested procedure is quick, easy, and appropriate for routine analysis.

KEYWORDS: *Azilsartan medoxamil, Methanol: water, Spectroscopy, validation, LOD, LOQ*

How to cite this paper: Nakul Arjun | Vikram Veer | Dr. Ashok Bhosale "Analytical Method Development and Validation for the Estimation of the Azilsartan Medoxamil by RP-HPLC Method in Bulk & Pharmaceutical Dosage Form" Published in International Journal of Trend in Scientific Research and Development (ijtsrd), ISSN: 2456-6470, Volume-6 | Issue-5, August 2022, pp.55-59, URL: www.ijtsrd.com/papers/ijtsrd50399.pdf

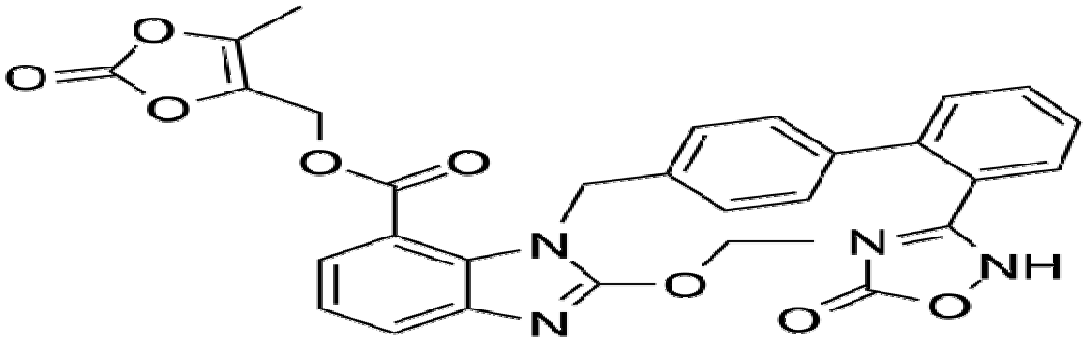
IJTSRD50399

INTRODUCTION:

Azilsartan medoxamil is an antihypertensive medication used to treat high blood pressure. Vasopressor hormone acts on the angiotensin receptor, preventing vasoconstriction and lowering blood pressure. It is mostly used to treat high blood pressure. Azilsartan medoxamil is a benzimazole 4-carboxylic acid that is 2-ethoxy- 3-4-2-5-oxo-2H-1,2,4-oxidizole-3-phenylmethyl. Azilsartan medoxamil is soluble into the methanol and ethanol.

Edarbi tablet (AZL), which was approved by the USA Food and Drug Administration on February 15, 2011, is used to treat adult hypertension. The dose range for Azilsartan medoxamil is 40 mg to 80 mg. Adults should take 80 mg of the recommended dosage.

Figure1: Structure of Azilsartan medoxamil



Literature survey reveals. The azilsartan medoxamil can be estimated by, RP-HPLC and spectroscopic method. The scope of present investigation were to developed and validate spectroscopy method for qualitative and qualification of azilsartan medoxamil in pharmaceutical dosage form.

MATERIALS AND METHOD:

Materials:

Azilsartan medoxamil standard drug was used. Analytical grade chemicals and solvents was supplied by from Loba Chemicals Pvt. Ltd, Mumbai. Distilled water is used to prepare all solutions. freshly prepared solutions are always used

Equipments:

The UV-spectrophotometry (Jasco UV visible detector 2075) with data processing system (Borwin chromatography software (version 1.50) was used. In a quartz cell measuring 200–400 nm, the sample solution was recorded in comparison to a solvent blank. The citizen electronic balance (Shimadzu AY-250) was used for weighing the sample. An ultrasonicator bath (Prama solutions for laboratory) was used for sonicating the drug sample.

The model PU 2080 plus intelligent HPLC pump was used, along with Rheodyne sample injection port with 50 microlitter loop is used.

Method development:

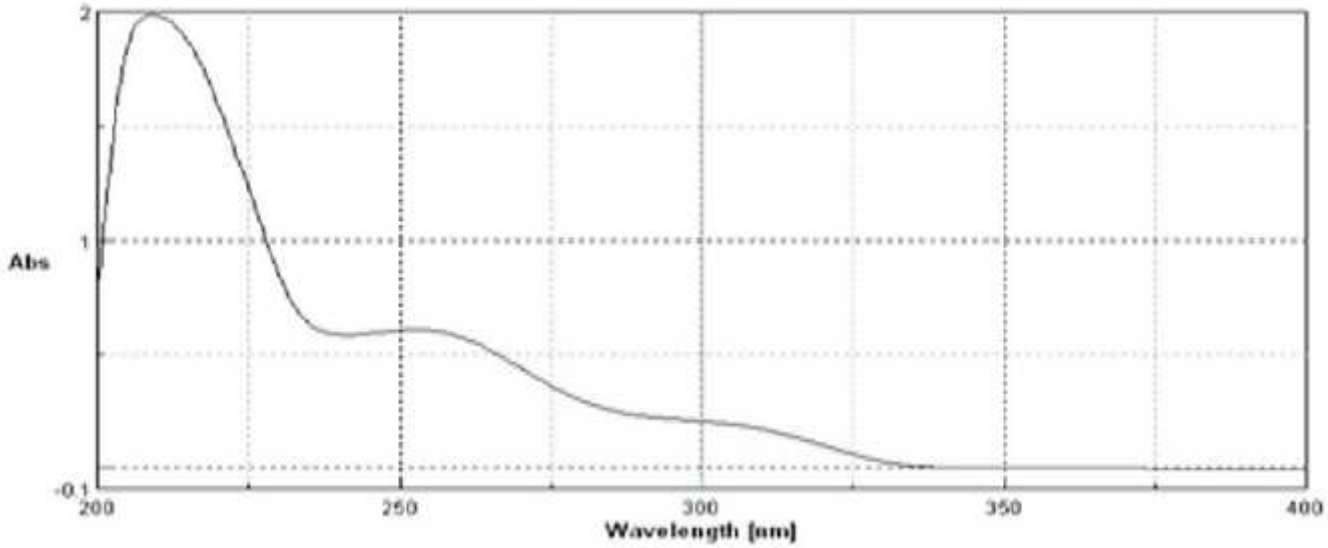
Preparation of standard stock solution:

Making a standard stock solution The standard stock solution of the medication was made by mixing 10 ml of methanol with 10 mg of the drug to make a 1000 g/ml concentration (A). A working standard solution containing 100 g/ml of azilsartan medoxomil in methanol was created from the equivalent standard stock solution (B). To obtain the final solution of Azilsartan Medoxomil (10 g/ml), additional dilution in methanol was made from this.

Selection of Detection Wavelength:

From the standard stock solution further dilutions were done using methanol and scanned over the range of 200 - 400 nm and the spectra was obtained (Fig. 2). It was observed that drug showed considerable absorbance at 249 nm.

Fig 2 spectrum of Azilsartan medoxamil by using methanol

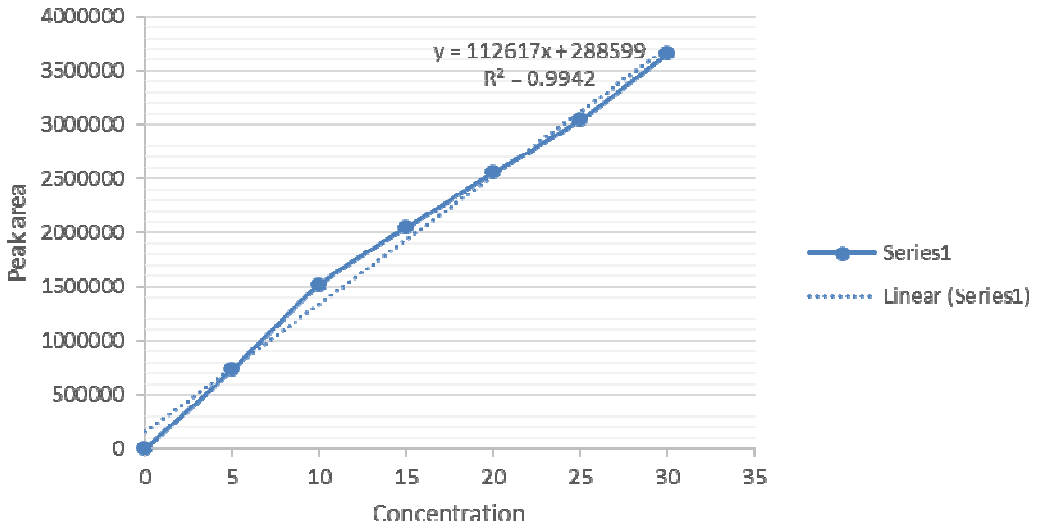


Preparation of calibration curve:

From the standard stock solution fresh sample is pipette out suitable diluted with Methanol: water, Methanol: sodium hydrogen phosphate, Methanol: sodium dihydrogen phosphate.

The solution was scanned under spectrum mode for 200 -400 nm. After few trials methanol: Sodium dihydrogen phosphate buffer in the ratio of 90:10 v/v was chosen as as the mobile phase which have the good resolution and acceptable peak parameters. A calibration graph of the absorbance versus the concentration of the drug was ploted and represented in figure 3.

Fig 3 calibration curve of Azilsartan medoxamil at 249 nm



Method validation:

Linearity:

The validation of Azilsartan medoxamil was validated according to the ICH guidelines

The developed method validates as per the ICH guidelines. The plot of absorbance verses concentration is shown in figure 3. It can be seen that plot is linear in concentration rane 5 -25 µg/ml with correlation coefficient (R^2) of 0.9942.

Precision:

The precision is calculated by using intraday and interday precision. It was determined by measurement of the absorbance for the three times on same day and on three different days The relative standard deviation for replicates of sample solution was less than 2% which meet accepatance criteria. The obtained results are as follows.

Intra-day Precision:

Table 1 Intraday precision

Conc (µg/ml)	Area	Amount recovered (µg/ml)	% Recovery	Average % Recovery	SD	%RSD
10	1413430.19	9.988	99.881	100.694	0.571	0.567
10	1422582.89	10.069	100.694			
10	1410184.07	9.959	99.593			
20	2563761.89	20.203	101.013	99.219	1.269	1.279
20	2523351.84	19.844	99.219			
20	2578556.87	20.334	101.670			
25	3085154.67	24.832	99.330	99.022	0.634	0.640
25	3076488.15	24.755	99.022			
25	3110821.41	25.060	100.241			

Inter-day Precision

Table 2 Inter-day Precision

Conc (µg/ml)	Area	Amount recovered (µg/ml)	% Recovery	Average % Recovery	SD	%RSD
10	1412445.79	9.979	99.794	99.763	0.149	0.149
10	1412096.29	9.976	99.763			
10	1409381.038	9.952	99.522			
20	2534307.62	19.941	99.706	101.099	0.960	0.950
20	2565701.83	20.220	101.099			
20	2524234.73	19.852	99.258			
25	3114242.98	25.091	100.363	100.339	0.368	0.367
25	3102893.60	24.990	99.960			
25	3123578.29	25.174	100.695			

LOD AND LOQ:

The limit of detection and limit of quantification of the drug were separately determined based on method of the intercept and the average value of the slope (0.652 for LOD and 1.997 for LOQ)

LOD and LOQ are calculated from the formula: -

$$LOD = \frac{3.3\ \sigma}{S} \qquad LOQ = \frac{10\ \sigma}{S}$$

Where, σ = standard deviation of Y intercept = 22265.29
S = slope of the calibration curve = 112616.67
LOD = 0.652 µg/ml
LOQ = 1.977 µg/ml

Accuracy:

To check accuracy of the method, recovery studies were carried by spiking the standard drug to the Azilceda – 40 tablet sample solution, at three different levels around 50, 100 and 150 %. Basic concentration of sample solution chosen was 10 µg/ml. % recovery was determined from linearity equation. The results obtained are shown in table 4.

Recovery studies of Azilsartan Medoxomil

Table 4: accuracy study

50%	10	5	1976627.83	14.988	99.924 ± 0.233
			1981105.24	15.028	
			1972265.63	14.949	
100%	10	10	2562436.23	20.190	100.641 ± 0.997
			2530212.26	19.904	
			2573761.89	20.291	
150%	10	15	3085114.64	24.831	99.906 ± 0.649
			3121154.67	25.151	
			3098172.55	24.947	

Optical Parameters:

Table 5: Optical Parameters

Sr.no	Parameter	Data
1	λ max	249nm
2	Correlation coefficient	0.9942
3	Slope	112616.67
4	Intercept	22265.29
5	Limit of detection	0.652 µg/ml
6	Limit of quantification	1.977 µg/ml

Result and discussion:

In the process of method development for the Azilsartan medoxamil different mobile composition, different buffers. The satisfactory result with mobile phase composition methanol: Sodium dihydrogen phosphate buffer pH (5) in the ratio of 90:10 v/v. The column Hi Q silc $_{18}$ 250 mm with the flow rate 1ml/min. The above method satisfied all the system solubility parameters like, resolution, tailing factor, plate count within range. Calibration plot was done and obtained linearity equation was y =112617 +288599.

Conclusion:

The simple RP-HPLC method for the determination of the Azilsartan medoxamil have been developed and validated as per the ICH guidelines. The developed method is found to be sensitive, accurate, and reproducible and can be used for the quality analysis of the Azilsartan medoxamil in bulk and pharmaceutical dosage form.

ACKNOWLEDGEMENT:

The authors are thankful and would like to show sincere gratitude to PDEA'S Shankarrao Ursal College Of Pharmaceutical Science & Research Center, Kharadi Pune, for providing all required facilities to complete this research work.

REFERENCES:

[1] Pradeepthi. J, Masthanamma. SK, Alekhya. G, Validated Spectrophotometric Method for Determination of Azilsartan Medoxomil in Pharmaceutical Dosage Form, Journal of

Scientific Research in Pharma. Nov. 2013; 2(4): 7-10.

[2] Srivastava M, editor. High-performance thin-layer chromatography (HPTLC). Springer Science & Business Media;2010 Nov 15. [4] Gandhi S, Mittal P, Pahade A, Rege S.

[3] K. Lavanya, V. Srinivasa Rao, P. Sunitha, K. Pavani Sai Rama. Analytical RP- HPLC Method Development and Validation for Simultaneous Estimation of AzilsartanMedoxomil and Chlorthalidone in Pharmaceutical Dosage Form, American Journal of PharmTech Research, 2017; 7(1), 599,606.

[4] Drug Profile, Azilsartan Medoxomil. https://pubchem.ncbi.nlm.nih.gov/compound/Azilsartan

[5] The Indian Pharmacopoeia, Ministry of Health and family welfare., Indian Pharmacopoeia commission, Ghaziabad, 2018; 8: 1616-1617.

[6] Drug Information, "Central Drugs Standard Control Organisation Directorate General of Health Services Ministry of Health and family Welfare https://cdsco.gov.in/opencms/opencms/system/modules/CDSCO.WEB/elements/downloadfile

[7] Chatwal GR, Anand SH. Instrumental Methods of Chemical Analysis.5th Ed., New Delhi, 2002; 2: 167- 2.172

[8] Deshmukh S, Vanjari S, Patil R. Application of Design of Experiment Based innovative Approach in Method Development and Validation of RP-HPLC for Estimation of Azilsartan in Bulk and Pharmaceutical Tablet Dosage Form. Indian Journal of Pharma Educational Research, 2020; 54(3): 657-666.

[9] Rathod N, Patel P. Development and validation of Spectrophotometric Methods for Simultaneous Estimation of Azilsartan medoxomil and AmJ. Pharm Res, 2019; 8(11): 1023-1035.

[10] Hinge M, Desai D. Chavda R, Patel D, Shing R, Patel E. Analytical method development and validation for simultaneous estimation of Cilnidipine and Metoprolol succinate by RP-HPLC. 2015; 7(5) 333-34.

[11] Center For Drug Evaluation And Research, Chemistry Reviews, Edarbi (Azilsartan Medoxomil) Tablets, 2011.

[12] Srinivasan R, Chandra J.K, Kumar D.R, Kumar N.D. Stability indicating RP-HPLC method for determination of Azilsartan medoxomil in bulk and its dosage form, International Journal of Pharmacy and Analytical Research 3(4), 2014, 445-452.

[13] Gorla R, Venkata R, Sreenivasulu B, Sreenivas N, Srinivas N.K et.al. Development and Validation of HPTLC Method for Estimation of Azilsartan Medoxomil in Bulk and Pharmaceutical Dosage Forms, International Journal of Pharmacy and Pharmaceutical Research, 2(3), 2015, 146-153.

[14] Kasimala M.B, Kasimala B.B. Reverse Phase-HPLC Method development and Validation For the Simultaneous estimation of Azilsartan Medoxomil and Chlortalidone in Pharmaceutical Dosage Forms, Journal of Atoms.