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UV Spectrophotometric Method Development and Validation for the Estimation of Olmesartan in Pure and Commercial Formulations

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ABSTRACT

A new rapid, sensitive UV-spectrophotometric method was described for the determination of olmesartan in pure and commercial formulations. The method is based on the measurement of absorbance of the drug solution in methanol at 257nm. Regression analysis of the Beer's law plot showed a good correlation in the concentration range 8.0 µg/ml to 48 µg/ml with a regression coefficient (r^2) of 0.9964. The limits of detection (LOD) and quantification (LOQ) were calculated to be 3.95 and 13.5µg/ml, respectively. The method was validated for accuracy, precision, selectivity, robustness and ruggedness. The method was applied to formulations with a recovery of 99.95 % respectively. The proposed method can be easily adapted in quality control laboratories of pharmaceutical industries for the assay of the olmesartan in its commercial formulations.

A. INTRODUCTION

Olmesartan [**Fig.1**] (5-methyl-2-oxo-2*H*-1,3-dioxol-4-yl)methyl 4-(2-hydroxypropan-2-yl)-2-propyl-1-({4-[2-(2*H*-1,2,3,4-tetrazol-5-yl) phenyl] phenyl}methyl)-1*H*-imidazole-5-carboxylate[1-3] belongs to the class of angiotensin II receptor blockers (ARBs) . It works by blocking angiotensin II in the body. that causes the blood vessels to tighten. As a result, olmesartan relaxes the blood vessels thereby lowers blood pressure and increases the supply of blood and oxygen to the heart. It is marketed by Unichem labs, India in the brand name of OlSAR (label claim 40mg of Olmesartan).

Literature survey revealed various research articles [4-17] are available regarding determination of olmesartan in pharmaceutical dosage form or in biological fluids. No UV spectrophotometric method has reported in literature for analysis of olmesartan Clonazepam in tablet dosage form. This paper describes very specific UV spectral identification test and simple, precise and accurate UV Spectrophotometric method for estimation of olmesartan in tablet dosage form. The present paper describes the development and validation of a new sensitive UV-spectrophotometric method for olmesartan in pure and commercial formulations as per ICH guidelines [17].

B. MATERIALS AND METHODS

a. Instrumentation: Shimadzu UV/Vis Spectrophotometer (Model-2450) equipped with UV probe software was used in the present assay. For dilutions, various micropipettes of volumes 10-100µL were used. All weighing experiments were done on Shimadzu Digital Analytical Balance (Japan) and standard glassware (Borosil Make) was used for preparing of solution.

b. Chemicals and Reagents: Olmesartan (99.9% Pure) used was supplied by Dr. Reddys Labs, Hyderabad and market formulations in the brand name of OLSAR (Strength: 40mg Olmesartan) manufactured by Unichem Laboratories Ltd, India were purchased from local pharmacy (Apollo). Methanol of analytical grade was purchased from local vendor and was used without further purification.

c. Diluent: Methanol of analytical grade was used as diluent in the present assay. It is used as received.

d. Preparation of Standard Solutions: The standard stock solution of olmesartan (1.0 mg/ml) was prepared by weighing 100mg of the pure drug, taken in 100 mL volumetric flask and was dissolved in 50 mL methanol and then makeup to the mark with distilled water. Working standard solutions of olmesartan were made with distilled water to obtain concentrations ranging from 8.0- 48.0 µg/mL respectively.

e. Preparation of Sample Solution: 10 Tablets of oral dosage form of olmesartan [**OLSAR (Strength: 40mg Olmesartan]** purchased from local pharmacy was weighed and powdered to fine powder. Then powder equivalent to 100 mg was transferred into a 100 ml clean dry volumetric flask, 70 ml of diluent was added to it, and was diluted up to the mark with the same diluent (stock solution concentration:1 mg/ml). This solution was then filtered through Whatman filter paper # 41. Fresh aliquots from standard sample solution were pipetted out and suitably diluted with distilled water to get the final that obey within the beers law limit.

C. RESULTS AND DISCUSSION

a. Method Development: Various solvents like distilled water, 0.1N HCl and methanol were tried to select an appropriate solvent for the assay of olmesartan with good suitability and stability. From these experimental the solvent methanol was selected since, it was soluble and exhibited, minimal interference in the absorbance of olmesartan was observed. The standard solution of olmesartan was scanned in the range of 200 to 400nm against water as blank for obtaining overlain spectra. The overlain UV spectra are shown in Figure.2. The absorbance and absorptivities of serial standard solutions of olmesartan were carried out at selected wavelength 257 nm, respectively. This wavelength of 257 nm was used for the quantification of standard and in dosage forms of olmesartan respectively.

b.Procedure: Working standard solutions of olmesartan in concentration range of 8.0 - 48µg/mL were placed in the cuvette of UV-spectrophotometer by using a precision pipette (Eppendorf). The absorbance of these concentrations were measured at this fixed wavelength (λmax 257.30 nm) and the quantity of olmesartan in standard preparation was calculated. A calibration graph of absorbance versus concentration was plotted.

c. Method Validation: The developed UV-Spectrophotometric method of olmesartan was validated as with International Conference on Harmonization (ICH) guide-lines [18].

i. SPECIFICITY:

a. Blank Interference: The interference of blank at the working wavelength was scanned from 200-800 nm and was observed the non-interference of blank at the working wavelength of 257nm for olmesartan, revealing the specificity of the proposed UV spectrophotometric method for olmesartan.

ii. Linearity: The linearity of the proposed method was made by determining the absorbance of different working concentrations of olmesartan in triplicate over a range of 25% (8.0µg/mL) to 150% (48.0µg/mL). Correspondingly a calibration graph was plotted by plotting the absorbance recorded versus the concentration and was treated by least-squares linear regression analysis.

The calibration graph for standard olmesartan was linear, within the working range of 8.0µg/ml to 48µg/ml that obeyed Beer- Lambert's law. The regression line equation for the graph was found to be y = 0.0278x + 0.0246, with a regression coefficient (r^2) of 0.9964 ensuring an accurate interpolation of the prepared test concentrations of olmesartan samples within the working range [Table.1 & Figure.3**]**

iii. LOD and LOQ: The LOD and LOQ for olmesartan were 3.95 and 13.5µg/ml, respectively that elaborated the sufficient sensitivity of the proposed method.

iv. Method Precision: The precision of the present proposed method was performed in six replicates of fixed concentration of olmesartan and the percentage relative standard deviation (% RSD) was measured. The %RSD of 0.30 tabulated in Table.2 was less than 2.0% revealing good precision of the proposed method.

v. Accuracy: The recovery studies of the proposed method was analyzed in triplicate preparations on composite blend collected from 20 tablets of olmesartan as per the proposed method at three different levels and the results of percentage recoveries were reported **Table.3.** The percent recovery at each level was ranged from 99.93% - 101.22% respectively, indicating insignificant interference from the excipients.

vi. Ruggedness: Ruggedness of the proposed method was determined by analyzing aliquots from homogenous slot by different analyst using similar operational and environmental

conditions and data is presented in Table.4. The %RSD obtained in this study were found to be not more than 2.0% (Table.4) making the current method rugged.

vii. Solution Stability: In this study, the absorbance of the same standard and sample solutions of olmesartan in triplicate at intervals of 6 hours, 12 hours, and 24 hours were recorded and the cumulative % recovery at each interval was determined. The % recoveries tabulated in Table.5 revealed the stability of the proposed method.

viii. Assay of Dosage Forms: Pharmaceutical assay was carried out on available brand of olmesartan (**OLSAR; Strength: 40mg**) procured from local pharmacy using the developed UV spectrophotometric method and the % assay was calculated and the results were tabulated (**Table.6**). The assay showed 99.95% contents of olmesartan, respectively, which is non-significantly different from the stated purity for olmesartan formulation.

D. CONCLUSION

The UV-Spectrophotometric method discussed in the present paper provides a simple, accurate, economical and convenient method for the analysis of olmesartan using UV-Spectrophotometry. The λ_{max} selected for quantitation of olmesartan was 257.30 nm with a linearity observed in the concentration of 8.0 - 48mcg/ml. The validation studies results demonstrated that the proposed procedure was accurate, precise and reproducible and moreover the proposed method utilized easily available and cheap solvent for analysis of and hence the method was also economical for the estimation of olmesartan in tablet dosage form and hence, it is concluded that the proposed UV-Spectrophotometric method could be regarded as a suggestive method for routine analysis of olmesartan in commercial formulations for quality control laboratories.

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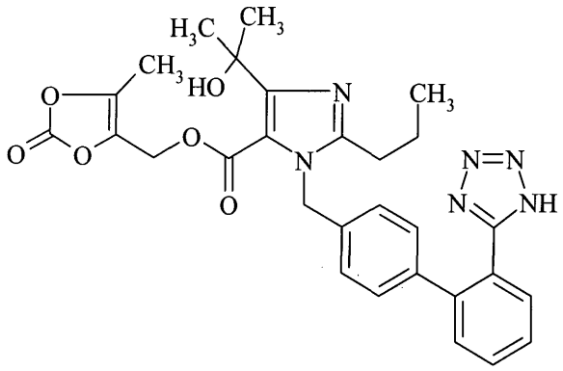

Fig.1: Structure of Olmesartan

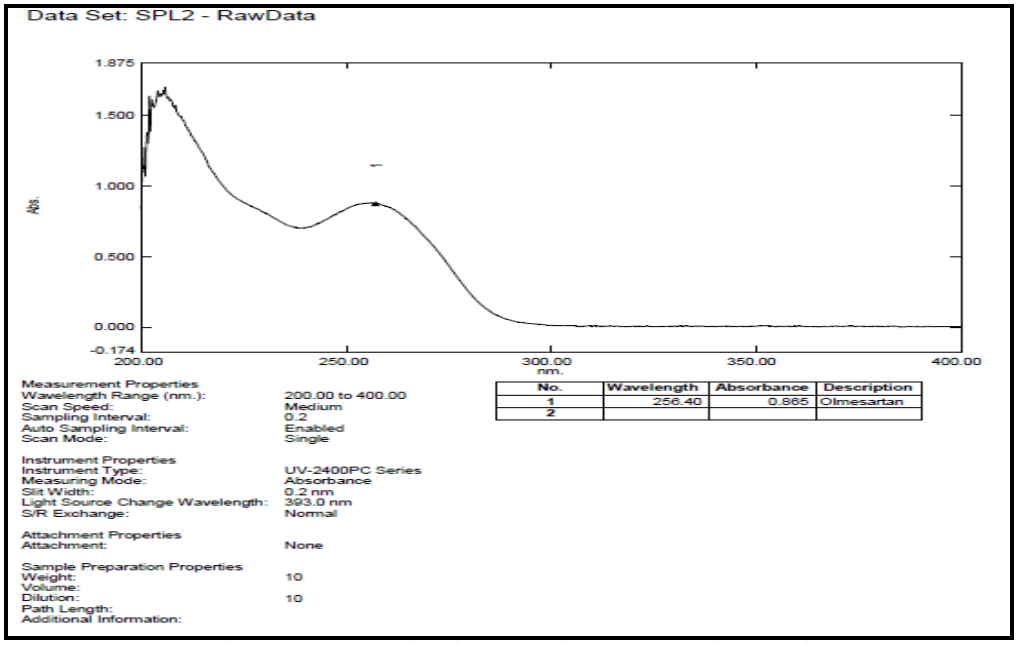

Fig: 2 - Validative UV Spectra of Olmesartan

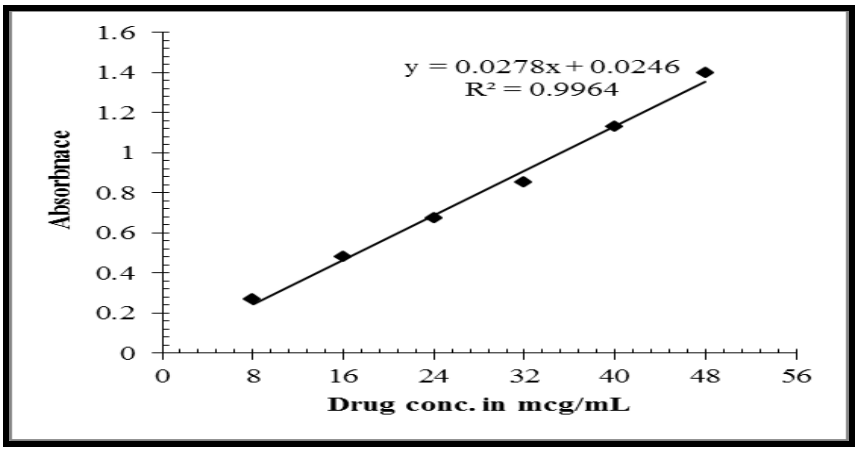

Fig: 3 - Linearity Plot of Olmesartan

Table.1. Results of statistical parameters for UV- spectrophotometric determination of olmesartan

% Level	Conc	Absorbance
25	8.0	0.271
50	16.0	0.480
75	24.0	0.676
100	32.0	0.854
125	40.0	1.132
150	48.0	1.399
Slope, b		0.0278
Intercept, a		0.0246
Correlation, r^2		0.9964
LOD		3.95
LOQ		13.5

Table.2: Results of method precision studies for Olmesartan

Sr. No.	Name	Absorbance
1	Solution-1	0.868
2	Solution -2	0.870
3	Solution -3	0.874
4	Solution -4	0.872
5	Solution-5	0.875
6	Solution-6	0.871
Avg*		0.872
STD DEV*		0.0026
% RSD*		0.30

*Average of six determinations considered

Table.3: Results of Recovery Studies for Olmesartan

Accuracy Level	**50%**	**100%**	**150%**
Sr. No.	**ABS**	**ABS**	**ABS**
Injection-1	0.671	0.894	1.127
Injection-2	0.673	0.898	1.123
Injection-3	0.67	0.894	1.125
AVG*	0.671	0.895	1.125
AMT. Recovered*	48.85	98.33	149.67
% Recovery*	97.70	98.33	99.78

*Average of six determinations considered

Table.4: Results of Ruggedness Studies for Olmesartan

By the Proposed Method

Sr. No	**Name**	**Analyst -1**	**Analyst -2**
		Absorbance	**Absorbance**
1	Scan-1	0.868	0.866
2	Scan-2	0.870	0.862
3	Scan-3	0.874	0.869
4	Scan-4	0.872	0.872
5	Scan-5	0.875	0.865
6	Scan-6	0.871	0.861
Avg*		0.872	0.865
Std Dev*		0.0026	0.004
% RSD*		0.30	0.48

*Average of six determinations considered

Table.5: Results of Stability Studies of Standard and Sample Solutions of Olmesartan with the Proposed Method

Time Interval (Hrs)	%Recovery	
	Standard (n=3)	Sample(n=3)
6	99.87	99.95
12	99.95	99.90
24	99.99	99.89

TABLE.6: Assay of Olmesartan in Marketed Brands

Marketed Brand of the Drug	Amount taken mg	Amount Found in mg	% Assay*
OLSAR	40	39.98	99.95

*Average of three determinations considered