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UV SPECTROPHOTOMETRIC METHOD DEVELOPMENT AND VALIDATION FOR THE ESTIMATION OF VALSARTAN IN BULK AND TABLET FORMULATION

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ABSTRACT

A simple, accurate, specific and precise UV Spectrophotometric method has been developed for estimation of valsartan in pure and pharmaceutical formulation. The λmax of valsartan in 0.1N NaOH and water was found to be 250 nm. 0.1N NaOH and water is used as diluent in equal proportion. The drug exhibited the linearity in the concentration range of 5-25µg/ml with correlation coefficient of 0.999. The % recovery of the drug for the proposed was found to be 99.25 %. The limit of detection and limit of quantification was found to be 0.91% and 2.77% respectively. No interference was observed in the presence of common pharmaceutical excipient. The method was validated as per ICH guidelines. The developed method was successfully employed for the estimation of valsartan in pharmaceutical dosage form.

KEYWORDS: Valsartan, UV Spectroscopy, Method development, Validation, Estimation.

1. INTRODUCTION

Valsartan is chemically N-(1-Oxopentyl)-N-[[2'-(1H-tetrazol-5-yl) [1, 1'-biphenyl]-4-yl] methyl]-L-valine (Figure 1). Valsartan is potent Angiotensin II receptor blocker. It is mainly used as anti-hypertensive drug. It is used for treatment of high blood pressure, of congestive heart failure (CHF), and post-myocardial infarction (MI). By blocking the action of angiotensin, valsartan dilates blood vessels and reduces blood pressure.[1-2]

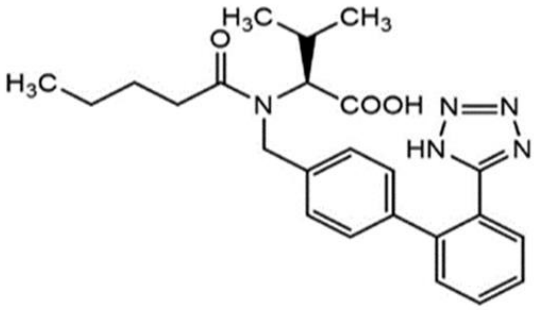

Fig. 1: Chemical structure of valsartan.

From the literature survey, it was found that valsartan was estimated by analytical methods such as Spectrophotometry[3-9], HPLC[10-13] in single form or combination with other drugs. The objective of this study was to develop and validate a simple, precise and accurate spectrophotometric method for the estimation of valsartan in pharmaceutical dosage form as per ICH guidelines.

2. MATERIALS AND METHODS

2.1 Instruments and reagents

A.UV-Spectrophotometer

LAB INDIA 3000 + Shimadzu balance.

B. Reference samples, chemicals and Reagent

Valsartan, 0.1 N Sodium Hydroxide, Distilled Water.

2.2 Sample Preparation

Take a 10 mg of Valsartan in 100ml of volumetric flask. Dissolve in 0.1N NaOH and distilled water (50:50) to obtain stock solution.

2.3 Selection of solvent

The solubility of Valsartan is determined in variety of solvents as per pharmacopeia standard. Solubility test was carried out in different solvents like distilled water, methanol, ethanol, 0.1N sodium hydroxide. From the solubility studies it was found that valsartan is soluble in distilled water, methanol and 0.1N sodium hydroxide. In this study 0.1N NaOH and distilled water (50:50) were selected as solvent.

2.4 Preparation of Standard Stock Solution

10 mg of Valsartan Standard was weigh and transferred to a 10 ml volumetric flask & diluted with 0.1N NaOH and distilled water (50:50) which gives 1000 µg/ml stock solution. Then from this stock 10 ml solution was transferred to 100 ml volumetric flask and diluted with distilled water which gives 100 µg/ml solution.

2.5 Determination of λmax

The standard solution of valsartan (10ug/ml) was scanned in UV region (200-400 nm) and the spectrum was recorded. Solvent was used as blank. It was seen that at 250 nm maximum absorbance was found, shown in figure 2. Therefore that 250 nm was selected for this study.

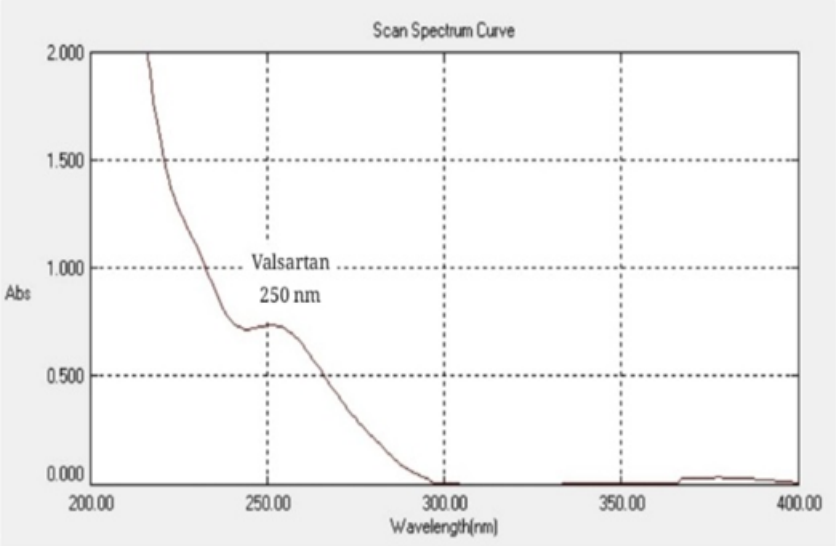

Fig. 2: Absorbance spectra of valsartan.

3. METHOD VALIDATION

The objective of method validation is to demonstrate that the method is suitable for its intended purpose. The method was validated for linearity, precision, accuracy, robustness, ruggedness, LOD, LOQ, and specificity as per ICH guidelines.

3.1 Linearity

From the standard stock solution, the various dilutions in the concentration of 5, 10, 15, 20 and 25 were prepared. The solutions were scanned at 250 nm and the absorbance was recorded, given in table 1. From this calibration curve was obtained by plotting absorbance versus concentration of valsartan and the linearity graph was represented in figure 3. The correlation coefficient was found to be 0.999.

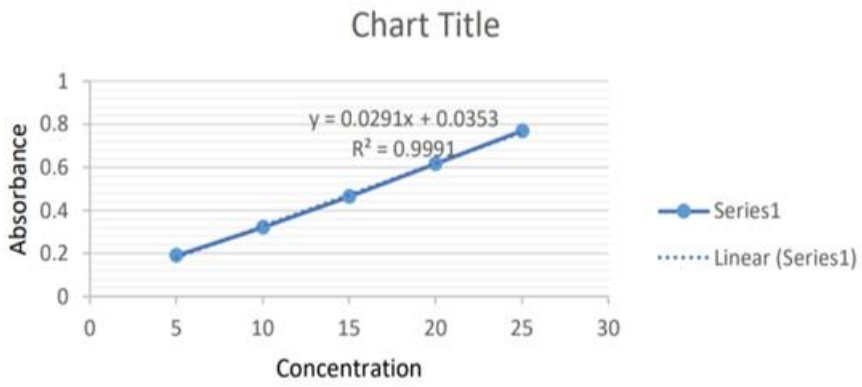

Fig. 3: Calibration curve of valsartan.

Table 1: Linearity parameter for valsartan.

Concentration(µg\ml)	Absorbance(nm)
5	0.189
10	0.321
15	0.464
20	0.616
25	0.769

3.2 Precision

Repeatability of the method was checked by scanning 10 µg/ml solution for 6 times. Intra-day precision was determined by checking the absorbance of (10ug/ml) on the same day. Inter-day precision was determined by checking the absorbance of (10ug/ml) on three different days. The %RSD was found to be 1.03% for intra-day and 1.27% inter-day as shown table 2.

Table 2: Precision parameter for valsartan.

Drug	Conc.	Intra-day [N=3]		Inter-day [N=3]	
		Mean Abs.	% RSD	Mean Abs.	% RSD
Valsartan (0.1N NaOH & Water)	5	0.178	1.992	0.186	1.918
	10	0.337	1.360	0.336	1.404
	15	0.481	0.731	0.482	1.142
	20	0.669	0.960	0.670	1.045
	25	0.815	0.495	0.794	1.893
	30	0.938	0.651	0.938	0.268
Mean		0.5696	1.031	0.567	1.278

3.3 Accuracy

Accuracy study was conducted by spiking at three different concentration levels (80%, 100%120%). At each level samples were scanned and from the absorbance recovery percentage was determined and presented in table 3.

Table 3: Accuracy parameter for valsartan.

Validation Parameter	Mean % Recovery	SD	%RSD
80%	99.27	0.002	0.375
100%	98.79	0.0005	0.095
120%	99.70	0.002	0.309
Mean		0.0015	0.259

3.4 Robustness

To determine robustness of the method two parameters (wavelength and diluent composition) were made slightly different from the selected wavelength and diluent composition. No significant difference was found in the absorbance and hence the proposed method was considered as robust which is shown in table 4.

Table 4: Robustness parameter for valsartan.

Conc.	Wavelengths			SD	% RSD
	248 nm	250 nm	252 nm		
5	0.149	0.151	0.152	0.0015	1.014
10	0.300	0.303	0.302	0.0015	0.506
15	0.450	0.454	0.451	0.0020	0.461
20	0.612	0.616	0.613	0.0020	0.339
25	0.770	0.774	0.770	0.0023	0.299
Mean				0.00186	0.5238
Mean				0.00186	0.5238

3.5 Ruggedness

The ruggedness of the developed method was checked by analysing the samples by different analysts at different days at similar operational condition. The statistical analysis showed no significant differences were observed between results obtained employing different analysts, given in table 5.

Table 5: Ruggedness parameter for valsartan.

Conc.	Absorbance		SD	% RSD
	Analyst-1	Analyst -2		
5	0.151	0.152	0.0007	0.467
10	0.300	0.302	0.0014	0.470
15	0.450	0.454	0.0028	0.626
20	0.616	0.612	0.0028	0461
25	0.772	0.770	0.0014	0.183
Mean			0.00182	0.4414

3.6 Limit of detection and Limit of quantification

Limit of detection is the lowest amount of an analyte in a sample that can be detected, but not necessarily quantified, under the stated experimental conditions Limit of quantification is the lowest amount of analyte in a sample that can be quantified under state experimental conditions. The LOD and LOQ for valsartan was found to be 0.91ug/ml, 2.77ug/ml.

4. RESULTS AND DISCUSSION

The method was validated and developed as per ICH guidelines. The method was validated in terms of linearity, precision, accuracy, robustness, ruggedness, LOD, LOQ and specificity. Beer's law is obeyed over the concentration range of 5-25 µg/ml, using regression analysis the linear equation Y=0.0291x + 0.0353 with correlation coefficient of r2 = 0.999. The precision results shows % RSD less than 2 at each level, which indicate clearly that the method is precise enough for the analysis of valsartan. The accuracy of the method was checked by recovery studies. The high recovery with values indicated the accuracy of the developed method. The robustness and ruggedness studies reveals that the method is enough robust and rugged. The LOD and LOQ values indicate sensitivity of the method. There was no interference observed from the excipients present in the formulation, indicated that the method is specific. Determination of valsartan in tablet formulation of two brands showed, content of valsartan were very close to the labelled amount. The %RSD values in all the parameters were within the acceptable limit. The optical characteristics of the method are represented in the table 6.

Table 6: Optical characteristics of the proposed developed method.

Parameter	Value
Λmax	250 nm
Beer's Range	5-25µg/ml
Correlation Coefficient (r2)	0.9991
Regression Equation	Y=0.0291x + 0.0353
Intercept (C)	0.0353
Slope (m)	0.0291
LOD (µg/ml) & LOQ(µg/ml)	0.91 & 2.77
Precision (%RSD)	1.2783

5. CONCLUSION

A validated UV Spectrophotometric method has been developed for the estimation of valsartan in bulk as well as pharmaceutical dosage form. The developed method was found to be simple, accurate, precise, specific, reproducible and linear over the concentration range studied. The proposed method can be used for routine analysis of valsartan in bulk as well as pharmaceutical formulations.

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