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SPECTROCOPIC METHOD DEVELOPMENT AND VALIDATION OF VALSARTAN BY USING PH 6.4 BUFFER

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ABSTRACT

Simple precise accurate UV Spectroscopic method has been developed and validated for estimation of valsartan in bulk and pharmaceutical dosage form. It is approved for the treatment of hypertension. It is an angiotensin II receptor antagonist. UV Spectroscopic method which is based on measurement of absorption of UV light, the spectra of valsartan in Phosphate buffer P^H 6.4 showed maximum wavelength at 252 nm and calibration curve were plotted over the concentrations ranging from 2-20µg/ml of valsartan with correlation coefficient 0.998 validation was performed as per ICH Q2 (R1) guidelines for linearity, accuracy, precision and recovery. The limit of detection (LOD) and limit of quantification (LOQ) were found to be 0.1224 and 0.3785 respectively by simple UV Spectroscopy. The proposed method was validated.

KEYWORDS: Valsartan, Phosphate buffer P^H 6.4, spectrophotometry and validation.

INTRODUCTION

Valsartan is chemically (2S) - 3-methy l-2- [N- ({4- [2 -(2H-1,2,3,4- tetrazol-5-yl)phenyl] phenyl} methyl) pentanamido] butanoic acid. It is white fine powder, slightly soluble in water, soluble in alcohol. It acts as antihypertensive Agent (Angiotensin II Receptor Antagonist). Structure of Valsartan is shown in figure – I. Here calibration curve method was employed by using phosphate buffer for the estimation of Valsartan in bulk and tablet dosage forms. "Phosphate buffer is aqueous solutions which are used to increase the aqueous solubility of another solute (poorly water soluble drug)". In the present investigation, phosphate buffer as solublizing agent, P^H 6.4 was employed to solubilize Valsartan fine

powder and its tablet dosage form to carryout spectrophotometric analysis. UV spectrum of Valsartan in phosphate buffer P^H 6.4 shown in figure-II.

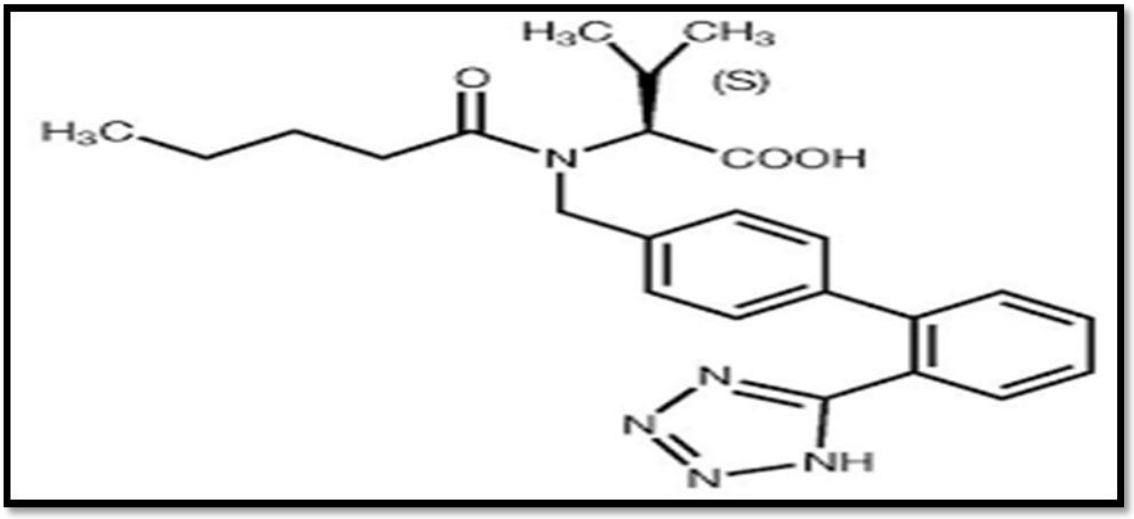

Fig. 1: Structure of Valsartan.

MATERIALS AND METHODS

Materials: Valsartan working standard drug was obtained from Dr. Reddy's Laboratories Ltd. (India), Mumbai, India. Distilled water was used to prepare phosphate buffer. Freshly prepared solutions were always employed.

Equipment: The UV-spectrophotometry **(**Jasco V630**)** with data processing system (UV Probe Software 2.31) was used. The sample solution Mumbai. All analytical grade chemicals and solvents were supplied by S.D. Fine chemicals, was recorded in 1 cm quartz cell against solvent blank over the range 200-400 nm. The citizen electronic balance (Schimadzu 220h) was used for weighing the sample. An ultrasonicator bath (PCI Analytics Pvt. Ltd) was used for sonicating the drug sample.

Preparation of standard stock solution

Standard drug solution of valsartan was prepared by dissolving 10 mg pure valsartan in phosphate buffer P^H 6.4 and transferred into 100 ml volumetric flask to obtain 100 µg/ml of stock solution from which desired concentrations of solutions were prepared.

Preparation of test solution

20 Tablets were weighed and its average weight was determined. An accurately weighed tablet power equivalent to 10 mg of valsartan transferred into 100 ml volumetric flask

dissolved in 100 ml of phosphate buffer and sonicated for 15 min and volume was made upto the mark and solution was filtered using whattman filter paper to obtain 100µg/ml stock solution.

Determination of λmax

10 µg/ml solution of valsartan was prepared and scanned in UV range of 200-400nm and spectrum was obtained. The λmax was found to be at 252 nm wavelength where absorbance was maximum at this wavelength. Hence this is considered as absorbance maxima (λmax) shown in figure 1.

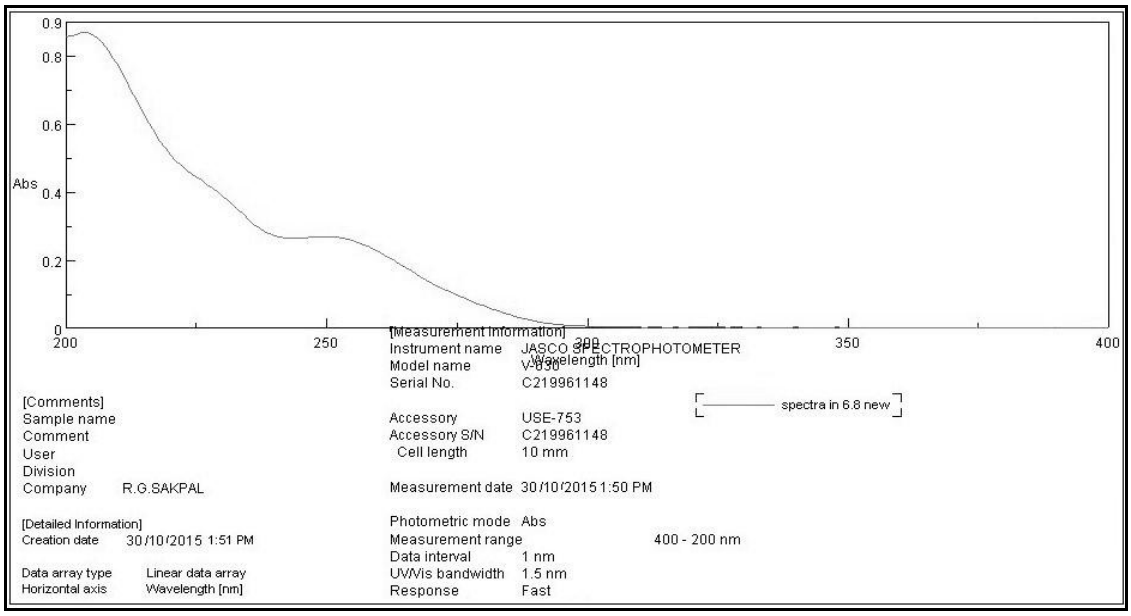

Fig. 2: Determination of λmax.

Preparation of calibration curve

Standard stock solution was suitably diluted with phosphate buffer to obtain concentrations ranging from 2-20 µg/ml. Absorbance of these solutions was measured at 252 nm (λmax valsartan) using UV, calibration curve was obtained by plotting graph between concentration and absorbance shown in figure 2.

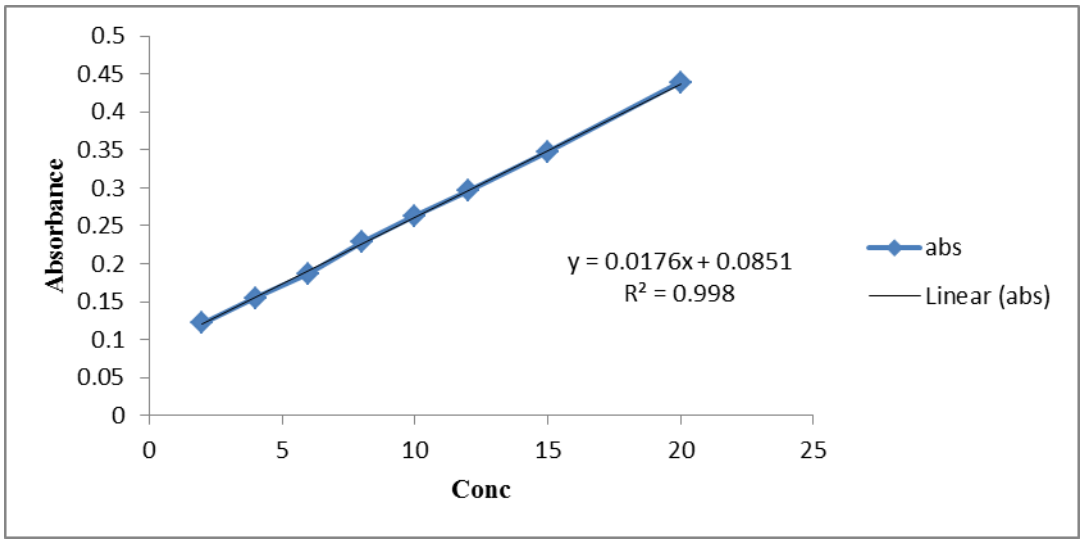

Fig. 3: Calibration Curve of Valsartan In Phosphate Buffer pH 6.4.

Method Validation

Validation of the Proposed Method

The proposed method was validated according to the (ICH) guidelines.

Linearity

The linearity of the proposed UV methods were evaluated by analysing different concentration of standard solution of Valsartan and by plotting Area under curve of analyte against concentration of analyte. Beer's law was obeyed for the method in the concentration range 2-20µg/ml. Graph was plotted for concentration and absorbance. A good linear relationship (R^2 = 0.998) was observed between the concentrations of Valsartan and corresponding Area under curve. The regression analysis was made for slope, intercept and correlation coefficient values. The equation of calibration curve obtained was Y=0.0176x+0.0851.

Precision

Precision is the measure of closeness of values between each concentration under same analytical conditions. It is determined by performing Interday and intraday precision studies. In intraday studies three standard raplicates injection of three different concentration were injected on same day and same standard different concentration were injected on three successive days in inter day precision studies. Where, the % RSD was found to be within limit (≤ 2). Given in table 1.

Table 1: Precision Study.

Concentration %	Absorbance Mean	Standard Deviation	% Relative Standard Deviation
Interday Precision(n=3)			
80	0.2627	0.0002645	0.1006
100	0.2625	0.0003162	0.1204
120	0.2628	0.0003605	0.1371
Intraday Precision(n=3)			
80	0.2624	0.0004582	0.1746
100	0.2642	0.0006557	0.2481
120	0.2631	0.0007106	0.2699

Accuracy

The accuracy of an analytical procedure expresses the closeness of agreement between the value which is accepted either as a conventional true value or an accepted reference value and the value found. The accuracy of the method was determined by performing recovery studies at three different levels of standard additions. Accuracy was checked by adding 80, 100 and 120% amount of Valsartan to pre-analyzed sample. Result are shown in Table 2.

Table 2: Accuracy Study.

Recovery	Conc. of Sample	Recovery in (µg/ml)	% Recovery
80%	8	8.11	99.91
100%	10	10.07	99.84
120%	12	11.97	99.96

LOD and LOQ

The limit of detection (LOD) and limit of quantification (LOQ) of the drug were separately determined based on method of the intercept and the average value of slope. (i.e. 3.3 for LOD and 10 for LOQ) ratio using the following equations designated by ICH guideline.

$LOD = 3.3\ \sigma/S$ $LOQ = 10\ \sigma\ /S.$

Where, σ = the standard deviation of the response, S = slope of the calibration curve.

Table 3:

LOD (µg/ml)	LOQ (µg/ml)
0.1224	0.3785

Table 4: Summary of Validation Parameters.

Sr. No.	Parameter	Data
1	λ-max	252 nm
2	Linearity range	2-20 µg/mL
3	Correlation coifficient	0.998
4	Slope	0.0176
5	Intercept	0.0851
6	Interday precision (n=3)	0.1006-0.1371
7	Intraday precision (n=3)	0.1746-0.2699
8	Accuracy (%)	99.91-99.96
9	Repeatability (RSD, n=3)	0.097-0.10
10	Limit of detection	0.1224 µg/mL
11	Limit of quantitation	0.3785 µg/mL

RESULTS AND DISCUSSION

Beer's law is obeyed over the concentration range of 2-20 µg/ml, using regression analysis the linear equation y = 0.0176x + 0.0851 with a correlation coefficient of 0.998. The limit of detection was found to be 0.1224 µg/mL. The limit of quantification was found to be 0.3785 µg/ml. Precision was calculated with intra and interday variation. Recovery study was performed on formulations and % RSD was found. The optical parameters such as Beer's law limit, slope, and intercept values were calculated and given in table 3. Method was validated for accuracy and precision. The accuracy of method was proved by performing recovery studies in prepared formulation. The results were given in table 2 and shows relative standard deviation was observed for analysis of three replicate samples, indicating precision and reproducibility.

CONCLUSION

The organic solvents such as ethanol, methanol, acetonitrile used widely in spectrophotometric analysis of poorly water soluble drugs are toxic in nature, costlier and responsible for pollution. Inaccuracy in spectrophotometric analysis due to volatility of organic solvents is another drawback. These problems are maximum minimized by development of UV method with phosphate buffer. It has UV cut off value 252 nm, since it do not interfere above 252 nm. The results concluded that the developed spectrophotometric method for determination of Valsartan in bulk and formulations using phosphate buffer P^H 7.4 is reliable, accurate, precise, sensitive and ecofriendly. This method can be successfully employed in the routine analysis of Valsartan in bulk drug and dosage formulations.

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