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Research Article

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Development and validation of new UV-spectrophotometric assay method for valsartan in pure and in formulations

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ABSTRACT

New simple, accurate and economical UV-spectrophotometric method has been developed for estimation of valsartan in pure and pharmaceutical formulation. The λ_{max} of valsartan in methanol and water was found to be 250.80nm. The drug exhibited the linearity in the concentration range of 5.0-30µg/ml with correlation coefficient of 0.996.The accuracy of the method was made by recovery experiment performed at three different levels i.e., 50%, 100% and 150 %. The % recovery was found to be in the range 99.26% - 100.7%. The low values of % R.S.D. are indicative of the high accuracy and reproducibility of the proposed method. The precision studies of the method revealed results of % R.S.D.values less than 2 indicating that the developed method is precise. The proposed method was applied to pharmaceutical formulation and % amount of drug estimated 99.97% was found in good agreement with the label claim. The developed method was a rapid and cost-effective for routine analysis of valsartan in pure and in pharmaceutical dosage form.

Keywords: Valsartan, UV-Spectrophotometry, validation

INTRODUCTION

Valsartan[1-4],N-[p-(o-1H-Tetrazol-5-ylphenyl) benzyl]-N-valeryl-L-valine1(Fig I) is an angiotensin II receptor antagonist, used in the treatment of hypertension.

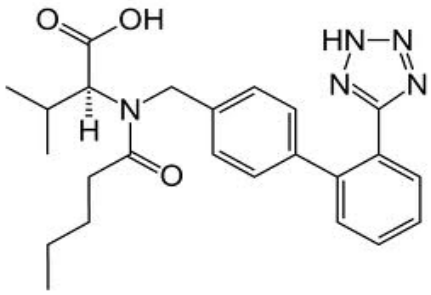


Fig I: Structure of Valsartan

Few HPLC methods[5-9] have been reported for the estimation of valsartan either in single or combined form in biological fluids and tablet forms. Among the various anlalytical methods available for the determination of drugs, spectrophotometry continues to be very popular, because of their simplicity and low cost. Accordingly, the objective of this study was to develop and validate the UV-spectrophotometric method for the estimation of valsartan in pure

and pharmaceutical formulation as per ICH guidelines (2004). This paper presents new UV-spectrophotometric method for the determination of valsartan in pure and pharmaceutical formulations

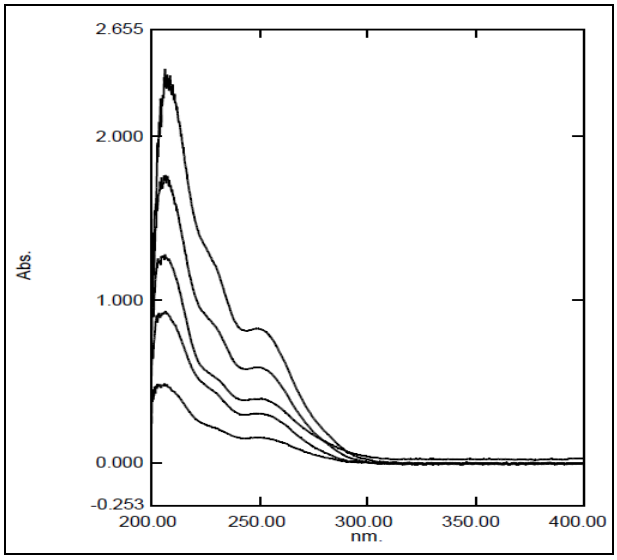
EXPERIMENTAL SECTION

Apparatus& chemicals:
Spectral and absorbance measurements were carried out by using Shimadzu UV/Vis spectrophotometer model UV-2450 equipped with 1.0cm thickness matched quartz cells were used for the entire experimental work. Valsartan was a gift sample from Torrent Pharmaceutical Limited, Ahmedabad. All chemicals and reagents used were of analytical grade and purchased from Qualigens Fine Chemicals, Mumbai,India.

Preparation of standard stock solution:
Weigh and transfer 50mg of Valsartan working standard into 50ml volumetric flask, add 40ml of diluent[Methanol] and sonicate to dissolve and dilute to volume with diluent. The volume was adjusted with the same up to the mark to give final strength i.e. 100µg/ml.

Selection of wavelength for analysis of Valsartan:
Appropriate volume 2.0ml of standard stock solution of valsartan was transferred into 100 ml volumetric flask, diluted to mark with distilled water to give concentration of 20µg/ml. The resulting solution was scanned in UV range (200nm- 400nm). In spectrum valsartan showed absorbance maximum at 250.80nm (**Fig.II**).

Fig.II:Absorption spectras of Five different concentrations(5.0-25µg/ml)of Valsartan



Validation of the method: The method was validated in terms of linearity, accuracy and precision respectively.

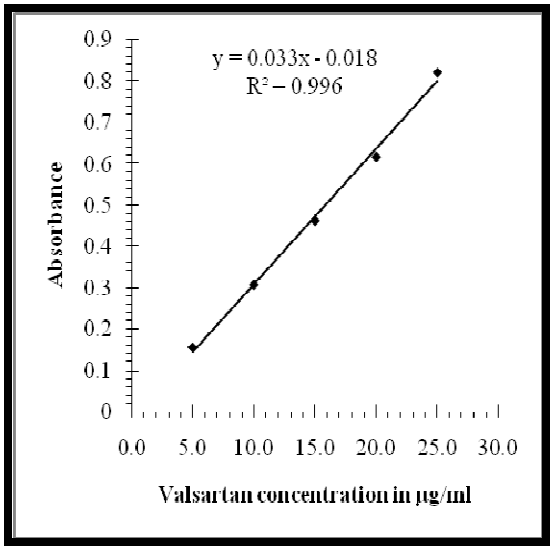
Linearity study: Different aliquots of valsartan in range 0.5-2.5ml were transferred into series of 100ml volumetric flasks and the volume was made up to the mark with distilled water to get concentrations 5, 10, 15, 20 and 25µg/ml, respectively. The solutions were scanned on spectrophotometer in the UV range 200 - 400 nm. The absorption spectrum was recorded at 250.80nm(Fig II). The calibration plot was constructed as concentration vs. amplitude (Fig III).

Sensitivity: The sensitivity of measurements of valsartan by the use of the proposed method was estimated in terms of the Limit of Quanfication (LOQ) and Limit of Detection (LOD). The LOQ and LOD were calculated using equation LOD = 3.3 x N/B and LOQ = 10 x N/B, where, 'N' is standard deviation of the peak areas of the drugs (n = 3), taken as a measure of noise, and 'B' is the slope of the corresponding calibration curve.

Accuracy: To the preanalysed sample solutions, a known amount of standard stock solution was added at different levels i.e. 50%, 100% and 150 %. The solutions were reanalyzed by proposed method.

Precision: The precision of each proposal methods was ascertained from the absorbance values obtained by actual determination of six replicates of a fixed amount of valsartan (20µg/ml) in total solution

Fig.III: Calibration plot of Valsartan



Application of proposed method for pharmaceutical formulation: For analysis of commercial twenty tablets were weighed; average weight was determined and finely powdered. An accurately weighed quantity of tablet powder equivalent to 50mg was transferred into 50ml volumetric flask add 40ml of diluent, sonicate to dissolve for 10mins and dilute to volume with diluent. The solution was then filtered through whatman filter paper no.45. From this 2.0ml was taken and transferred to 100ml volumetric flask and volume was made up to the mark with distilled water to give 20µg/ml concentration. It was scanned on spectrophotometer in the UV range 200 - 400nm. The spectrum was recorded at 250.80nm. The concentrations of the drug were calculated from linear regression equation.

RESULTS AND DISCUSSION

The proposed method was validated as per ICH guidelines. The solutions of the drugs were prepared as per the earlier adopted procedure given in the experiment.

The linear regression data for the calibration curves showed good linear relationship over the concentration range 5.0-30.0µg/ml for valsartan. Linear regression equation was found to be Y = 0.033X - 0.018 (r^2=0.996). The results of this analysis are repersented in Table.I. The linearity equation was found to be Y = 0.033X - 0.018. The LOQ and LOD for valsartan were found to be 1.79µg/ml and 5.95µg/ml, respectively (Table.I).

Table - I: Calibration results data

Parameter	Results
Regression equation; Slope (b)	-0.018
Intercept (a)	0.033
Correlation coefficient	0.996
Standard deviation on intercept(S_a)	0.0195
Standard deviation on slope (S_b)	0.00117
Standard error on estimation(S_e)	0.0186
Limits of Detection (LOD)[µg/ml]	1.79
Limits of Quantification [LOQ)[µg/ml]	5.95

The precision of the developed method was expressed in terms of % relative standard deviation (%RSD). The % R.S.D. values found to be less than 2, indicating that the proposed method is precise for the determination of valsartan in formulations (Table-II).

Table - II: Results of accuracy by the proposed method by standard addition method

Amount taken in µg/ml	Amount added in µg/ml	Amount recovered	%Recovery
10	5	14.89	99.26
10	10	19.97	99.85
10	15	25.18	100.7

The solutions were reanalyzed by proposed method; results of recovery studies are reported in Table-III which showed that the % amount found was between 99.26% to 100.7% with %R.S.D. >2.

Table - III: Results of precision by the proposed method

S.No	Precision	Absorbance
1	Scan-1	0.308
2	Scan-2	0.305
3	Scan-3	0.303
4	Scan-4	0.306
5	Scan-5	0.305
6	Scan-6	0.305
Avg		0.305
Std Dev		0.00163
% RSD		**0.53**

Absorbance was measured for same concentration solutions, six times in two days. The results are given in Table-IV and are in the acceptable range for valsartan. The results showed the % R.S.D. was less than 2% respectively.

Table -IV: Assay of Valsartan in pharmaceutical formulation

Pharmaceutical Formulation	Labelled Amount (mg)	Amount found** (mg) ±S.D by the Proposed Method	Found by reference method[9]±S.D	% recovery by proposed methods
DIOVAN	80	79.97 ± 0.21	79.99 ± 0.15	99.97

*** Average of six determinations*

From the results of validation obtained it is concluded that the proposed UV spectrophotomertic method developed by the author is relatively simple, rapid, and cost effective and therefore, could be applied as alternative method for routine quality control assay of valsartan in pharmaceutical raw material and dosage forms.

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