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Development and statistical validation of spectrophotometric methods for the estimation of Valsartan in tablet dosage form

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ABSTRACT

Three new simple, economic spectrophotometric methods were developed and validated for the estimation of Valsartan in bulk and tablet dosage form. First method includes determination of Valsartan at absorption maxima 250 nm, second method applied was area under curve for analysis of Valsartan in the wavelength range of 245-255 nm and third method was second order derivative. Beer law obeyed in the concentration range of 5-50 µg/mL for all three methods. The correlation coefficients were found to be 0.999, 0.999 and 0.979 by absorption maxima, area under curve and second order derivative spectra. Results of analysis were validated statistically and by performing recovery studies. The mean percent recoveries were found satisfactory for all three methods. The developed methods were also compared statistically using one way ANOVA. The proposed methods have been successfully applied for the estimation of Valsartan in bulk and pharmaceutical tablet dosage form.

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KEYWORDS

UV spectrophotometric;
Valsartan;
Area under curve method;
Derivative spectroscopy.

INTRODUCTION

Valsartan chemically is N-[p-(o-1H-Tetrazol-5-ylphenyl)benzyl]-N-valeryl-L-valine^[1] Figure 1. It is an angiotensin II receptor antagonist, effective in the treatment of hypertension^[2]. It is also effective when used alone or in combination with other drugs for the treatment of high blood pressure. It is not official in any of the pharmacopoeia.

Literature survey revealed that several analytical techniques like spectrophotometric methods^[3,4] and HPLC5-9 Methods have been reported for the

determination of Valsartan and its combination with other drugs. The proposed research work describes three new UV-spectrophotometric methods for the estimation of Valsartan in bulk and tablet dosage form.

EXPERIMENTAL

Shimadzu UV-1800 double beam spectrophotometer with 1 cm path length supported by Shimadzu UV-Probe software, version 2.35 was used for all spectrophotometric estimations.

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Shimadzu balance (BL-220H) was used for all weighing. Valsartan was obtained from ISP Hongkong Ltd., Hyderabad, India. Formulation of Valsartan in tablet dosage form was purchased from local market.

Standard stock solution

Solution containing 200 $\mu\text{g/mL}$ of pure drug was prepared by dissolving 20 mg of Valsartan in sufficient methanol to produce 100 mL solution in volumetric flask. From this aliquot solution was pipetted out and diluted with methanol to obtained working standard stock solution of 100 $\mu\text{g/mL}$.

Analysis of the tablet formulation

Twenty tablets were accurately weighed and powdered. A portion of tablet powder equivalent to 20 mg of Valsartan was accurately weighed and transferred into a 100 mL volumetric flask and then added 25 mL of methanol to dissolve contents of tablet formulation. Then, solution was sonicated for 20 minutes and filtered through Whatman filter paper 41. The final volume was made up to 100 mL with methanol to obtain concentration of 200 $\mu\text{g/mL}$ Valsartan. From this aliquot solution was pipetted out and suitably diluted with methanol to obtained working standard stock solution of 100 $\mu\text{g/mL}$. Various dilutions of the tablet solution were prepared and analyzed for five times and the concentration was calculated by using the calibration curve for three methods.

Recovery

A recovery study was carried out by addition of known amount of standard drug in the preanalysed tablet formulation, in 80%, 100% and 120% of label claim. At each level of amount three determinations were performed.

RESULTS AND DISCUSSION

Absorption maxima method

For selection of analytical wavelength 20 $\mu\text{g/mL}$ solution of Valsartan was prepared by appropriate dilution of standard stock solution and scanned in the spectrum mode from 400-200 nm. From the spectra λ_{max} of Valsartan 250nm was selected for the analysis Figure 1 and Figure 2.

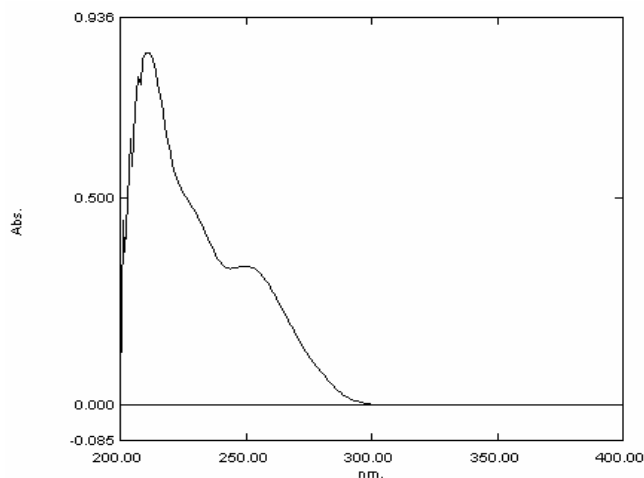


Figure 1 : Zero order UV-spectrum of Valsartan (10 $\mu\text{g/mL}$) showing absorption maxima at 250 nm.

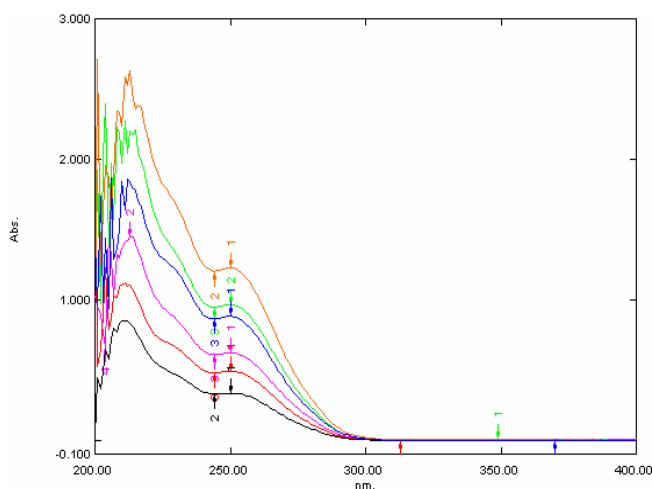


Figure 2 : Overlain spectra of Valsartan.

The calibration curve was prepared in the concentration range of 5-50 $\mu\text{g/mL}$ at 250 nm. By using calibration curve, the concentration of the sample solution was determined.

Area under curve method

For selection of analytical wavelength, 20 $\mu\text{g/mL}$ solution of Valsartan was prepared by appropriate dilution of standard stock solution and scanned in the spectrum mode from 400-200 nm. From the spectra of drug, area under the curve in the range of 245-255 nm was selected for the analysis Figure 3. The calibration curve was prepared in the concentration range of 5-50 $\mu\text{g/mL}$ at their respective AUC range. By using calibration curve, the concentration of the sample solution can be determined.

Second order derivative spectroscopic method

In this method, 20 $\mu\text{g/mL}$ solution of Valsartan

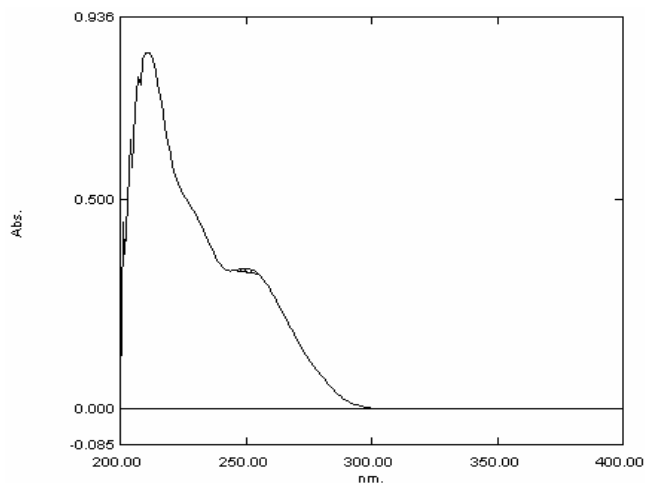


Figure 3 : Zero order UV-spectrum of area under curve method for Valsartan 10 µg/mL.

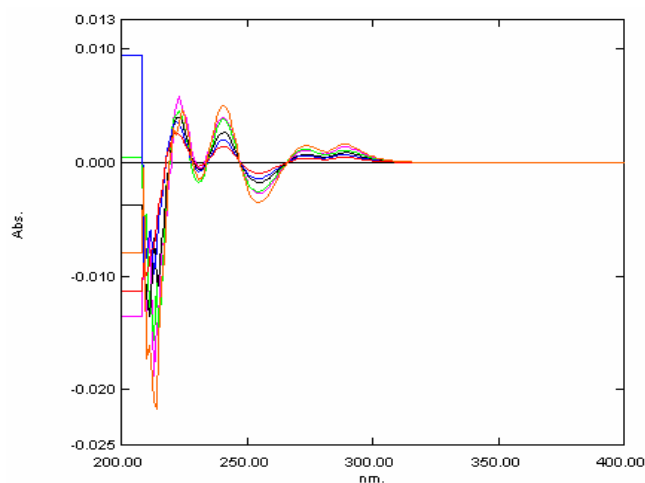


Figure 5 : Second order derivative overlain spectra of valsartan.

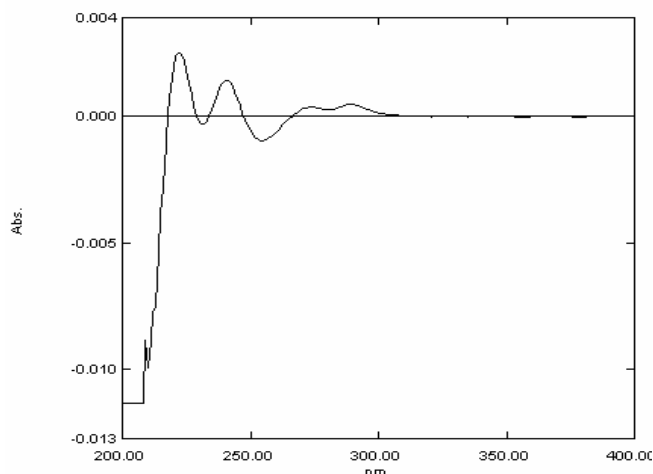


Figure 4 : Second order derivative spectrum of Valsartan (10 µg/mL) method.

was prepared by appropriate dilution of standard stock solution and scanned in the spectrum mode from 400-200 nm. The absorption spectra obtained was derivatized to second order derivative spectra. The second order derivative spectra show maxima and minima at 241 nm and 255 nm respectively Figure 4, 5. The absorption difference is calculated which was directly proportional to the concentration of the standard solution. The calibration curve for Valsartan was plotted in the concentration range of 5-50 µg/mL and scanned in the Second order derivative spectra. The calibration curve of $d^2A/d\lambda^2$ against concentration of drug showed linearity.

Method validation

Linearity

From standard stock solution of 200 µg/mL of

Valsartan 1, 2, 4, 6, 8, 10 mL were transferred into series of 10 mL volumetric flasks to obtain concentration range of 5 to 50 µg/mL for Valsartan (TABLE 1).

TABLE 1 : Statistical parameters of linearity of Valsartan.

Parameters	Absorption Maxima	Area Under Curve	Second Order Derivative Spectroscopy
Beer's law range	5-50 µg/mL	5-50 µg/mL	5-50 µg/mL
Coefficient of Correlation (r^2)	0.999	0.999	0.979
Slope(m)	0.031423	0.005603	0.000171
Intercept(c)	0.007792	0.00107	-0.00025
LOD, µg/mL	0.25	0.53	1.01
LOQ, µg/mL	1.09	0.81	1.75

$Y=mx+c$

Accuracy and precision

Precision of the method was evaluated by using tablet powder equivalent to 100% of the label claim of Valsartan. Method repeatability was obtained from R.S.D. value by repeating assay of four replicates of single concentration three times in a same day. Intermediate precision was assessed by assay of four replicates of single concentration of Valsartan on three consecutive days. The accuracy of the methods was assessed by recovery studies at three different levels, 80%, 100% and 120%. The values of standard deviation and recovery studies were found satisfactory (TABLE 2).

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TABLE 2 : Results of analysis of valsartan in tablet formulation and recovery studies

Method	Label Claim mg/tab	% estimated*	% Recovery*	S.D.	% RSD	SEM
Absorption Maxima	40 mg	99.21	100.59	±0.9358	0.9394	0.5402
Area Under Curve	40 mg	99.80	99.40	±0.8324	0.8335	0.4805
Second Order Derivative	40 mg	99.60	100.33	±0.6639	0.6712	0.3832

*denotes n=4; Valzaar-40 (torrent pharmaceuticals, sikkim)

Limit of detection and limit of quantisation

The detection limit and quantisation limit was computed for lower limit of detection and minimum quantity of analyte measured and was found to be satisfactory by proposed spectrophotometric methods.

Statistical evaluation

The developed methods statistically compared using one way ANOVA and indicate no significant difference between three methods. Hence these methods can be useful in routine analysis of Valsartan in bulk drug and tablet formulation (TABLE 3).

TABLE 3 : Results of one way ANOVA tukey-kramer multiple comparison test).

Comparison	Mean Difference	q Value	P Value
Absorption Maxima vs. AUC	0.5232	6.241	P<0.001
Absorption maxima vs. Second order Derivative spectroscopic	0.6328	7.548	P<0.001
AUC vs. Second order Derivative Spectroscopic	0.1096	1.307	P>0.05

CONCLUSION

The developed new three methods proved to be simple in procedure and it produced more accurate results. Hence all three methods effective for the routine analysis of Valsartan in bulk and tablet dosage form.

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