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Application of first derivative spectrophotometric method for the determination of Rilpivirine in pure and tablet formulations

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ABSTRACT

A simple, accurate and reproducible spectrophotometric method was established for the estimation of Rilpivirine by first derivative spectrophotometry. Methanol was used as the solvent. The proposed method obeys beer's law in the concentration range of 6-16 µg/ml with maximum absorbance at 306 nm on the first derivative spectrum. The method was validated for accuracy, precision, ruggedness and robustness. The numerical values for all the validation parameters were within the acceptable limits. The results of rilpivirine were determined by linear regression equation with slope; 0.0047, intercept; 0.00066 and correlation coefficient of 0.9998. The recovery studies confirmed the accuracy of the proposed method and low values of standard deviation confirmed precision of the method. Spectral interference by excipients was eliminated by first order derivative spectrophotometric method. The proposed method was found to be suitable for regular analysis.

Keywords: Rilpivirine, first derivative, methanol, validation.

INTRODUCTION

Rilpivirine is a non nucleoside reverse transcriptase inhibitor used in the treatment of HIV. Chemically it is 4-{[4-({4-[(*E*2-cyanovinyl]-2,6-dimethylphenyl}amino)pyrimidin-2-yl]amino}benzonitriles. It is practically insoluble in water over a wide pH range and soluble in methanol. Earlier reports reveal that there were spectrophotometric[1,2], LC[3,4] methods developed for the estimation in methanol but there were no derivative methods developed for the estimation of rilpivirine.

The present study focuses on development of simple, accurate derivative spectrophotometic method for the estimation of rilpivirine in dosage forms.

MATERIALS AND METHODS

Preparation of stock solution:
A solution of (1mg/mL) rilpivirine was prepared by dissolving 100 mg of rilpivirine in 100 ml of methanol as a solvent.

Preparation of calibration standard solutions:
Sample solutions of 6-16 µg/mL of rilpivirine in methanol were prepared from the suitable dilutions of stock solution.

Spectral scan
The solutions were scanned from 200-400 nm. The zero order spectrum was converted to first order derivative and λ_{max} was selected. The overlain first order spectra were shown in fig-1.

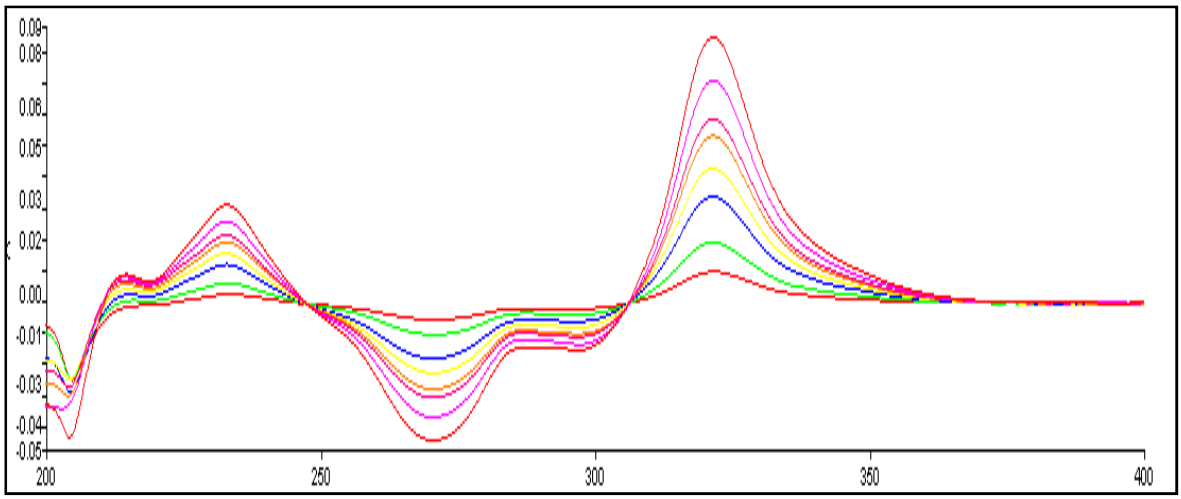

Fig. 1. Overlay First Derivative Spectra of Rilpivirine

Assay:
Twenty commercial tablets were taken and weighed accurately. Tablet powder equivalent to 100 mg of rilpivirine was weighed and dissolved in methanol to make 100 ml. It was suitably diluted within the linearity range and observed the absorbance of the sample at the 306 nm of the first derivative spectrum.

RESULTS AND DISCUSSION

Method development
The method was developed by proper selection of the solvent and suitable wavelength for absorbance measurement of the sample. Several solvents like water, 0.1 M sodium hydroxide, 0.1 M hydrochloric acid, methanol, ethanol and chloroform were tried for solubility test. The drug was insoluble in all the solvents except methanol. Hence methanol was chosen as the solvent. First order derivative spectrum was chosen to avoid interference from excipients and the wavelength of 306 nm was chosen for linearity studies. The effect of temperature on λ_{max} was studied and found that the results were precise at room temperature. The results of the optical and regression parameters were presented in table-1.

Table 1: Regression characteristics of proposed method of Rilpivirine

Parameters	Results
$\lambda_{max,}$ nm	306
Molar absorptivity, L/mol.cm	5×10^{-3}
Sandell's sensitivity, µg/cm^2/0.001 absorbance unit	0.01
LOD(µg/mL)	1.21
LOQ (µg/mL)	4.72
Regression equation Y= a+bc Slope (b) Intercept (a) Correlation coefficient (r)	 47×10^{-3} 66×10^{-4} 0.9998

Validation of method:
The method was validated for linearity, accuracy, precision, LOD, LOQ, ruggedness and robustness as per ICH guidelines and the results were found to be satisfactory. The overlain derivative spectra were shown in fig 1. Linearity was assessed by measuring the absorbances of the several concentrations of rilpivirine prepared by using methanol. The linearity of the method was proportional to the concentration of the analyte within the range 6-16 µg/mL and was presented in table 2 and pictured in fig 2.

Table 2: Linearity of derivative spectrum of Rilpivirine

Conc. µg/mL	Absorbance
6	0.031
8	0.040
10	0.049
12	0.058
14	0.067
16	0.075

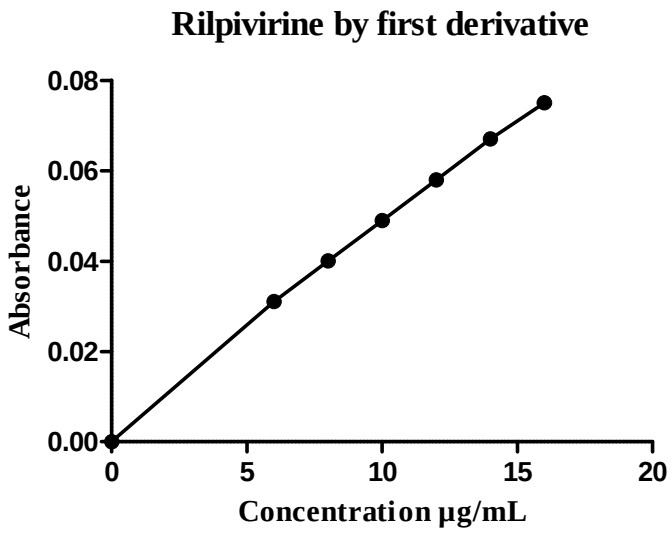

Fig.1. Linearity plot of Rilpivirine

The Intraday and interday precision of the method was found by measuring the average of the absorbance of 6 samples containing 10 µg/mL of drug for three times in a day and three successive days and the results obtained were found to be satisfactory and given in table 3.

Table 3: Precission studies of Rilpivirine

Parameter	Concentration, µg/mL	Mean absorbance, nm	SD	% RSD
Inter day	10	0.051	0.01928	0.041
Intra day	10	0.048	0.01918	0.038

Accuracy of the proposed method was estimated at 80%, 100% and 120% level of drug spiked to the known estimated amount of drug and the results were tabulated in table 4.

Table 4: Analysis of tablet formulation

Label claim (mg)	**%Amount found*±SD**	**% RSD***	**% Recovery***
50	49.72	0.02	99.89
100	99.69	0.04	99.91

**Mean of six determinations*

The % RSD of the recovery studies were less than two. Robustness studies were performed by using 95% and 90 % methanol and The similarity of the results is obvious evidence that minor change in solvent strength do not cause any significant change in results. Change in temperature has not shown much change in solubility and difference in absorbance.

CONCLUSION

The proposed derivative spectrophotometric method is simple and sensitive with reasonable precision, accuracy and constitute better alternative to the existing ones for the routine determination of rilpivirine in bulk and pharmaceutical formulation. The method was found to be simple, precise and accurate and can be used for routine analysis.

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