IJPPR
INTERNATIONAL JOURNAL OF PHARMACY & PHARMACEUTICAL RESEARCH
An official Publication of Human Journals
ISSN 2349-7203
HUMAN

Human Journals
Research Article
May 2018 Vol.:12, Issue:2

A Validated Analytical Method Development for Rosuvastatin Calcium by Difference Spectroscopy

IJPPR
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ISSN 2349-7203
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Submission: 20 April 2018
Accepted: 27 April 2018
Published: 31 May 2018

Keywords: Rosuvastatin calcium, methanol, UV spectroscopy

ABSTRACT

In this study, the development and validation of UV spectroscopy (difference spectroscopy) for determination of Rosuvastatin calcium used to preventing the stroke and heart attack Rosuvastatin calcium in bulk drug and pharmaceutical formulation is described. Method development was done and Beers lamberts concentration range was found to be (2 to 20µg/ml) the regression equation was found to be r^2 =0.992 with correlation coefficient value the solvent used was methanol. validation of developed analytical method is going on. Parameters studied till dated are linearity, precision and Robustness result of validation parameter were found within the acceptance limits LOD and LOQ for the given method was found to be 7.86 and 23.822 respectively.

INTRODUCTION

Rosuvastatin Calcium [1] is official in Indian pharmacopeia. It is synthetically (E)- (3R,5S)-7-{4-(4- fluorophenyl)-6-isopropyl-2-{methyl(methyl sulphonyl amino)]pyrimidin-5-yl}-3,5-dihydroxyhepten-6-oic corrosive calcium. It is utilized as a lipid bringing down operator act by restraint of 3-hydroxy-3-methylglutaryl-coenzyme (HMG-CoA) reductase[2]. Rosuvastatin calcium is orally regulated as the calcium salt. The structure of Rosuvastatin calcium is shown in fig. 1

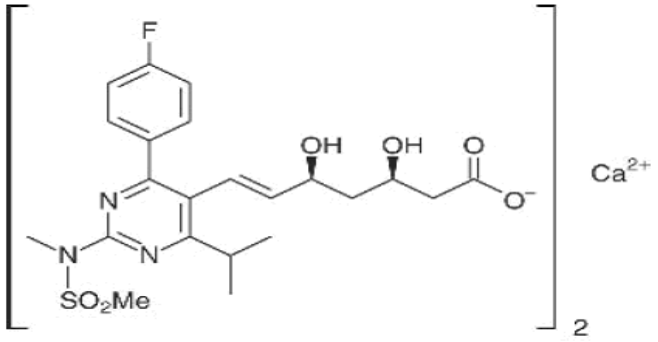

Fig no: 1 Structure of Rosuvastatin calcium

OBJECTIVE

Rosuvastatin calcium shows improved absorbing interference by the technique of different spectrophotometry[3]. Thus the objective of the present study was to develop new analytical difference spectrophotometry method and its validation parameters for the proposed method according to ICH guidelines for the estimation of Rosuvastatin calcium[4].

MATERIALS AND METHODS

The bulk drugs of Rosuvastatin calcium and were procured available in ltd and mankind pharmaceutical Ltd. Chemicals and reagents used for the study were of an Analytical grade.

Instrumentation

A Shimadzu 1800 UV/VIS double beam spectrometer (Japan) equipped with 1 cm matched quartz cell was used for all special measurement.

Selection of common solvents

1N HCl and 1N NaOH were selected as a common solvent for developing spectral characteristics of the drug.

Preparation of solution

The standard stock solution containing Rosuvastatin calcium prepared by dissolving 25 mg in 25 ml of methanol then diluted with 1N NaOH and 1N HCl separately to get series of dilution ranging from 2-20 µg/ml absorbance's recorded at 229.3 nm and 235.7 nm respectively against reagent blank. The calibration curve was prepared by plotting concentration versus the difference in absorbance and found to be linear in the concentration range of 2-20 µg/ml.

VALIDATION

The proposed method was validated according to ICH (Q2) B guidelines for validation of analytical procedures. As per the ICH guidelines, the method validation parameters checked were Selectivity, linearity, precision, and accuracy[5].

Selectivity

The selectivity of the method was assessed by analyzing standard drug, and pharmaceutical product, comparing the maxima and minima of the standard with that of the sample to determine whether the pharmaceutical product and excipient lead to interfering in the estimation [6].

Limit of Detection and Limit of Quantification

The Limit of Detection (LOD) is the smallest concentration of the analyte that gives the measurable response[7]. LOD was calculated using the following formula

$$LOD = 3.3\ \sigma / S$$

The Limit of Quantification (LOQ) is the smallest concentration of the analyte [8], which gives the response that can be accurately quantified. LOQ was calculated using the following formula

$$LOQ = 10\ \sigma / S$$

Where σ is a standard deviation of the response and S is the slope of the calibration curve. LOD& LOQ of Rosuvastatin calcium was found to be7.86 µg/ml &23.822µg/ml respectively.

Linearity

Different volumes of stock solutions were suitably diluted with the corresponding medium to get the desired concentrations. Each solution was analyzed in triplicate. The amplitude values were plotted against the corresponding concentrations to obtain the linear calibration curve[9].

Range

2-20 µg/ml.

Precision

The precision of analytical methods was expressed in relative standard deviation (RSD)The proposed method was validated according to ICH (Q2) B guidelines for validation of analytical procedures[10]. As per the ICH guidelines, the method validation parameters checked were Selectivity, linearity, precision, and accuracy.

Selectivity

The selectivity of the method was assessed by analyzing standard drug, and pharmaceutical product, comparing the maxima and minima of the standard with that of the sample to determine whether the pharmaceutical product and excipient lead to interfering in the estimation[11].

Robustness

It is the measure of the capacity of an assay to remain unaffected by small but deliberate variation in method parameter and provide an indication of its reliability in normal usage. Degradation and variation in a chromatography column, mobile phase, and inadequate method development are column causes of lack of robustness [12].

Simultaneous Equation Method

The absorption spectrum shows that Rosuvastatin calcium has at 229.3nm respectively. For the simultaneous equation method, two wavelengths i.e. λmax of these drugs were selected and the absorbance, as well as the absorptivity values, were calculated from their individual

spectra [8]. Absorbance was noted against each concentration at 229.3nm and 235.7nm for these drugs from their individual spectra and their absorptivity values were calculated

Analysis of marketed formulation

Twenty tablets of the formulation were accurately weighed and powdered. A quantity of powder equivalent to 10mg of both drugs was weighed and dissolves in 100ml of methanol. The mixture was ultrasonicated for 20 minutes[13]; the solution is filtered through Whatman filter paper no.4 then final dilution was made with methanol to get final concentration.

Determination or estimation of overlay:

The concentration range of 8-16 µg/ml The drug Rosuvastatin calcium working standard solution was scanned at 2 different λmax 229.3nm and 235.7nm with UV spectrophotometer (Japan) equipped with 1cm matched quartz cell and solvent used Methanol(AR grade), manufactured by Merck pharmaceutical, shown in figure no:2 and 3

Determination of λ_{max} by UV

Each working standard solution was scanned between the range 200-400 nm. The calibration curves for RSV calcium were prepared in the concentration range of 8-16 µg/ml respectively. The Linearity graphs of Rosuvastatin calcium[14].

Estimation of linearity

The linearity of the response of the drug Rosuvastatin calcium was observed in the concentration range from 8-16 µg/ml were scanned at 2 different λmax 229.3 and 235.7 and different insolvent i.e. 1N HCl and 1N NaOH and this graph show a difference in absorbance and it obeyed Beers law[15]. Linearity in the response was obtained in the range of 8µg/ml to 16µg/ml. The regression equation was calculated as Y=0.001×+0.011. correlation coefficient value for Rosuvastatin calcium was R^2 =0.992**,** shown in table no 1 and 2.and graph is shown in fig no 4.

Estimation of Precision:

The developed UV-spectroscopic method was found to be precise as the %RSD values for system precision was found to be Rosuvastatin calcium at 2 different λmax 229.3nm and

235.7nm in 1N HCl %RSD is 0.3964 and 1N NaOH %RSD is 0.2921 are shown in table no: 3

Estimation LOD and LOQ:

The detection limits and quantitation limits for altretamine were found to be 7.86and23.822 µg/ml respectively. These results prove that drug sample can also be determined accurately and precisely shown in table no: 2.

Estimation of Robustness:

The evaluation of robustness study of Rosuvastatin calcium in a change in temperature, change in pipette and change in analyst is shown in table no 4.

Estimation of Recovery study:

The estimation of % recovery study of Rosuvastatin calcium is 3µg/ml is 471.4, 6µg/ml is 157.13 and 9µg/ml is 28.56 is shown in table no: 5

RESULTS AND DISCUSSION

The optical characteristics such as Beer's law limits, percent relative standard deviation and percent range of error were found to be within the limit and satisfactory. All of the analytical validation parameter for the proposed method was determined according to ICH guidelines .the method was found to provide a high degree of precision and reproducibility. The recovery studies showed that the result was within the limit indicating no interference. The proposed method is simple, sensitive, accurate and precise and can be successfully employed for the routine analysis of the Rosuvastatin calcium.

CONCLUSION

The proposed method is simple, accurate, precise and selective for the estimation of Rosuvastatin calcium. The method is economical, rapid and does not require any sophisticated instruments contrast to chromatographic method.it can be effectively applied for the routine analysis of Rosuvastatin calcium.

ACKNOWLEDGMENTS

We are grateful to Prof. Dr. Chanchal deep Kaur, Mr.Ajeet Pandey, and his staff at of pharmacy Rawatpura Sarkar Institute of pharmaceutical science for the providing laboratory facilities and providing an opportunity for performing the work in college.

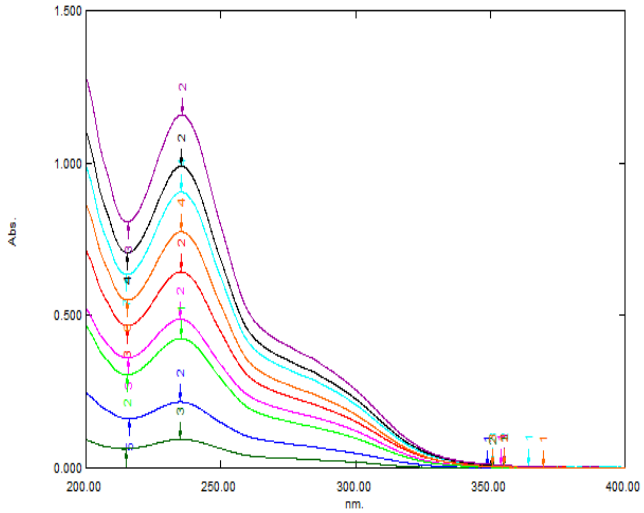

Fig no: 2 depicting the graph for rosuvastatin calcium (Hcl)

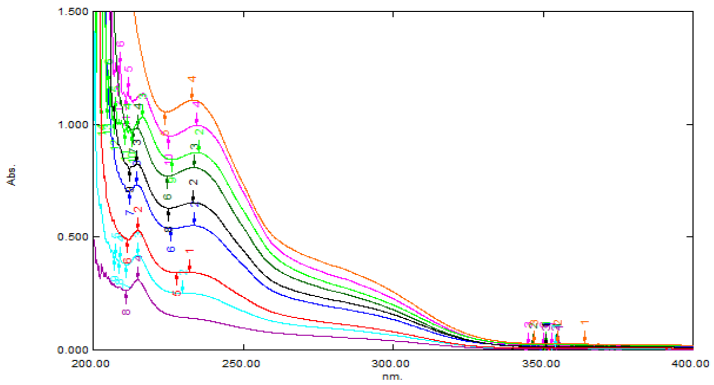

Fig no: 3 depicting the graph for Rosuvastatin calcium (NaOH)

Table no. 1: Linearity of Rosuvastatin Calcium By Difference Spectroscopy-

Sr. No.	Conc. of Rosuvastatin calcium(µg/ml)	Absorbance at 229.3 nm(1N NaOH)	Absorbance at235.7nm (1N HCl)	Difference in absorbance
1.	8	0.425	0.426	0.001
2.	10	0.543	0.546	0.003
3.	12	0.640	0.647	0.007
4.	14	0.792	0.802	0.010
5.	16	0.860	0.873	0.013

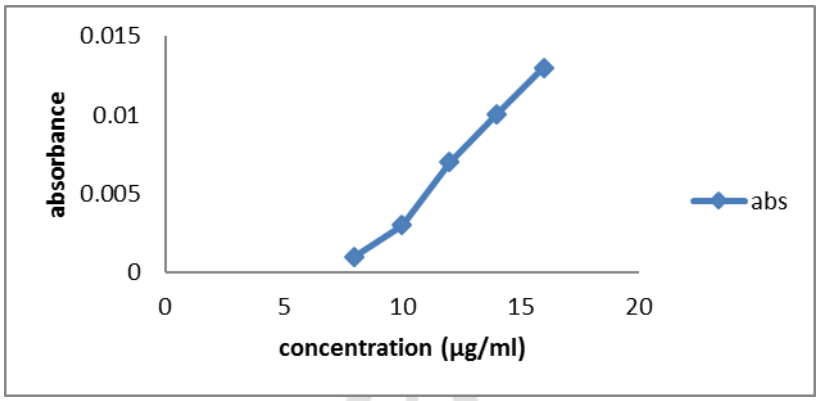

Fig no: 4 depicting the linear graph for Rosuvastatin calcium

Linearity in the response was obtained in the range of 8µg/ml to 16µg/ml. The regression equation was calculated as Y=0.001×+0.011. correlation coefficient value for Rosuvastatin calcium was R^2 =0.992.

Table no. 2: characteristics and validation of Rosuvastatin calcium

Parameters	Values
Beers law limit (µg/ml)	8-16
Registration equation (y=a+bc)	0.001×+0.011
Coefficient correlation	0.992
LOD(µg/ml)	7.86
LOQ(µg/ml)	23.822

Table no. 3: System Precision

Solvent	Absorbance	SD	%RSD
1N HCl	235.7nm	0.001673	0.3964
1N NaOH	229.3nm	0.001414	0.2921

Table no. 4: Robustness

Condition varied	% RSD
Analyst -1	0.304
Analyst-2	1.875
Normal temp.	1.06
Warm temp.	0
Cold temp.	0.275
5ml pipette	0.859
2ml pipette	1.785

Table no. 5: Recovery Study

Sr. No.	Conc. of std. µg/ml	Conc. of test µg/ml	Amount obtained	Amount recovery	% recovery
1	6	3	17.142	14.142	471.4
2	6	6	15.428	9.428	157.13
3	6	9	8.571	2.571	28.56

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