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Method Development and Validation of Rosuvastatin Calcium by Using Uv-Spectrophotometry

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ABSTRACT: Statins are the treatment of choice for controlling hypercholesterolemia in patients with cardiovascular risk. Although all statins are HMG-CoA reductase inhibitors, they possess different chemical structure, pharmacokinetic profile and lipid modifying efficacy. From the published literature, it is known that Rosuvastatin calcium is more effective in lowering bad cholesterol and raising good cholesterol compared to rest of statins. Extensive analytical methods for the determination of Rosuvastatin are more complex, expensive and time-consuming. So the present study is focused on development and validation of a reliable, simple and economic method for the estimation of Rosuvastatin Calcium in bulk and formulation. Analytical method development being a vital part of pre-formulation and formulation, research and development obviates the need to develop reliable and economical methods for the estimation of drugs in bulk and formulation. UV Spectroscopy is one of the earliest and widely used methods in drug analysis despite the availability of chromatographic and hyphenated techniques. A simple, precise, sensitive, accurate and economical method has been developed for the estimation of Rosuvastatin Calcium in bulk and formulation. The drug exhibits an absorption maximum at a wavelength of 247nm with distilled water as solvent. Beer's law is obeyed in the concentration range of 5-30µg/ml and percentage purity is found to be 98.90%. LOD and LOQ values are found to be 0.75 and 2.27µg/ml. The developed method is validated statistically as per ICH guidelines and the results obtained are within acceptance criteria related to linearity, accuracy and precision.

Keywords: Rosuvastatin Calcium, Distilled water, Hypercholesterolemia, Accuracy, Precision**.**

I.INTRODUCTION

Rosuvastatin calcium is used as an antihyperlipidemic, which is a HMG-CoA reductase inhibitor, a rate-limiting enzyme in cholesterol biosynthesis. It is used in the treatment of dyslipidemia, which is effective at low doses and its half-life is more compared to other statins. Chemically it is, calcium salt of (3R,5S,6e)-7-(4-(4-Fluorophenyl)-6-(1-methylethyl)- 2-(ethyl(methylsulfonyl)amino)-5-pyrimidinyl)-3,5-dihydroxy-6-heptenoic acid (fig:1) and molecular formula is (C22H27FN3O6S)2Ca [1]. Extensive literature survey reveals that a few spectrometric [2-9] were available for the estimation of Rosuvastatin calcium in bulk and formulations. The objective of the present study is to develop a new simple, sensitive, accurate, rapid and economic method for the estimation of Rosuvastatin Calcium in bulk and tablet formulation.

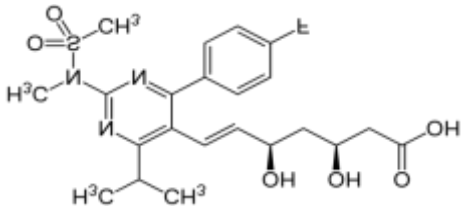

Fig-1: Structure of Rosuvastatin

II.MATERIALS AND METHODS:

Instrument

Spectrophotometric analysis was performed using double beam UV-Visible spectrophotometer (SHIMADU), (model no.UV-1900) with 1cm pathlength supported by UV-WIN Software.

Chemicals

Rosuvastatin Calcium was obtained as gift sample from, Arti chemicals; Mumbai, distilled water (AR Grade) were used for analysis. Rosuvastatin tablets 10mg were purchased from a local pharmacy.

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METHOD:

Determination of lambda max

Solution of Rosuvastatin Calcium was prepared using water and scanned on UV-Visible spectrometer between 200-400nm against water as blank.

Preparation of standard stock solutions:

Standard stock solution of Rosuvastatin were prepared by dissolving 100 mg of drug in a 100ml volumetric flask and dissolved in distilled water to get a concentration of 1000µg/ml.

Preparation of working standard solutions:

The working standard solution of Rosuvastatin were prepared by diluting 10 ml of the standard stock solution to 100 ml with distilled water in a 100ml volumetric flask to get the concentration of 100µg/ml.

Preparation of calibration standards

Accurately 0.5-3 ml of working standard solution of Rosuvastatin Calcium was transferred to a series of 10ml volumetric flasks and the volume was made up to the mark with distilled water to produce 5-30µg/ml solutions and the absorbance of the resulted solutions was measured at 247nm. The calibration curve was constructed by plotting absorbance against concentration.

Preparation of sample solutions:

rosuvas (M/s Ranbaxy Ltd.) were purchased from local market. The average weight of each tablet (before and after removing coating) was calculated by weighing 20 tablets. Ten tablets were powdered finely in a glass mortar. Powder equivalent to 50 mg of rosuvastatin was successively extracted with distilled water and the extracts transferred quantitatively into 100 mL volumetric flask after filtering through Whatman No. 1 filter paper. The volume was then made up distilled water with (500 µ g/mL). Then this solution was further diluted with distilled water to get working standard solution of 50 µ g/mL. Suitable volume of this solution was taken in 10 mL volumetric flask and volume was made up with distilled water. Absorbances were read and concentrations of rosuvastatin determined using the calibrationcurve. Calculations were then made with the dilution factor to find out the concentration of the drug in tablets. The
experiments were repeated six times to check its reproducibility.

Validation of the Developed Method

The developed method was validated for accuracy, precision, linearity, limit of detection, limit of quantitation and robustness as per ICH guidelines.

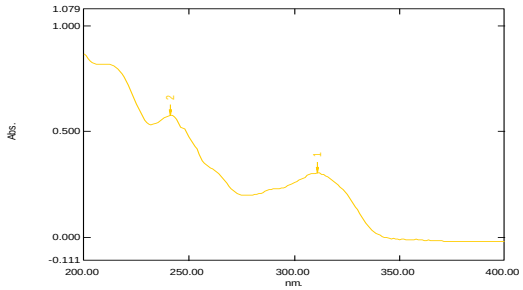

Table 1: Analysis of marketed formulation

Tablet formulation	Label claim	Amount taken	Assay (Amount found)	%rsd
Rosustat	10 mg	10 mg	9.89 ± 0.00115	1.402

Table 2: Absorbance values of Rosuvastatin with distilled water

S.NO	Concentration (µg / ml)	Absorbance at 247nm
1	0.5	0.229
2	1.0	0.352
3	1.5	0.443

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4	2.0	0.518
5	2.5	0.619
6	3.0	0.777

Table 3: Absorbance values of Precision at intraday

Concentration (µg/ml)	Absorbance (nm)			Mean	Standard Deviation	% Relative standard Deviation	Average of % RSD
	1	2	3				
10	0.341	0.360	0.360	0.337	0.005219	1.455505	0.295168
15	0.528	0.498	0.457	0.521	0.006034	1.214124	
20	0.637	0.672	0.678	0.632	0.004509	0.670687	

Table 4: Absorbance values of Precision at interday

Concentration (µg/ml)	Absorbance (nm)			Mean	Standard Deviation	% Relative standard Deviation	Avg % of RSD
	1	2	3				
10	0.360	0.360	0.360	0.360	6.79	1.455505	0.754566
15	0.494	0.495	0.494	0.495	0.001	1.214124	
20	0.668	0.666	0.666	0.666	0.173205	0.670687	

Table 5: Accuracy results for Rosuvastatin

No of Preparation	Concentration (µg/ml)		Percent Recovery	Mean %Recovery ±SD	% RSD	Mean RSD
	Formulation	Pure Drug				
1: 80%	10	8	100.00	100.09±0.362	0.15	0.14
2: 80%	10	8	100.00			
3: 80%	10	8	100.28			
1:100%	10	10	100.90	100.64± 0.454	0.12	
2:100%	10	10	100.90			
3:100%	10	10	100.13			
1:120%	10	12	100.18	100.18± 0.539	0.11	
2:120%	10	12	100.18			
3: 120%	10	12	100.18			

Table 6: LOD and LOQ values for Rosuvastatin at 247 nm

S. no	Parameters	Standard deviation	Formula	Calculation (µg/ml)
1	LOD	0.010	$3.3(S.D/b)$	0.75
2	LOQ	0.010	$10(S.D/b)$	2.27

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Table 7: Validation results for UV method of Rosuvastatin

Sr.NO	PARAMETERS	RESULTS
1	Absorption maxima(nm)	247
2	Correlation coefficient (R2)	0.999
3	Linearity Range (µg/ml)	5-30
4	Interday Precision	0.754566
5	Intraday Precision	0.295168
6	Assay in percentage(n=6)	100.79±0.0037
7	Accuracy (Mean RSD) (n=3)	0.14
8	LOD (µg/ml)	0.75
9	LOQ (µg/ml)	2.27

III.RESULTS AND DISCUSSION:

METHOD DEVELOPMENT:

This study was focused on development of a new spectrometric method for the analysis of Rosuvastatin in bulk drug and tablet dosage form. Spectrophotometric analysis was performed using double beam UV-Visible spectrophotometer (UV-1900) with 1cm path length supported by UV-WIN Software.

Linearity:

The linear regression data for the calibration curves showed good linear relationship over the concentration range 5–30 µg/ml for Rosuvastatin**.** Linear regression equation was found to be Y =0.246 (r^2 = 0.999). The result is expressed in Table 2.

Precision:

The precision of the developed method was expressed in terms of % relative standard deviation (% RSD). These results show reproducibility of the assay. The % RSD values found to be less than 2 that indicate this method precise for the determination of both the drugs in formulation [Table 3&Table 4].

Accuracy:

The solutions were reanalyzed by the proposed method; results of recovery studies are reported in Table 4 which showed that the % amount found was between 100.09% and 100.64 % with % RSD > 2 drugs in formulation [Table 5].

LOD & LOQ:

The LOQ and LOD for Rosuvastatin were found to be 0.75 µg and 2.27 µg, respectively. The result is expressed in Table 6.

IV.CONCLUSION

This UV-spectrophotometric technique is quite simple, accurate, precise, reproducible, sensitive and economical quantitative analytical method for determination of Rosuvastatin. The UV method has been developed for quantification of Rosuvastatin in tablet formulation.This method was validated as per ICH Q2 (R1) guideline. The validation procedure confirms that this is an appropriate method for their quantification in the formulation. It is also used in routine quality control of the formulations containing this entire compound**.**

The sample recoveries in all formulations were in good agreement with their respective label claims and they suggested no interference of formulation excipients in the estimation. Hence, the method can be easily and conveniently adopted for routine estimation ofRosuvastatin in tablet dosage form.

V.ACKNOWLEDGEMENT:

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