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Method Development and Validation of Atorvastatincalcium in Bulk and Tablet Dosage Form by Uv Spectroscopy.

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ABSTRACT
The objective of this research is to describe the optimization, validation, and application of spectrophotometric techniques for determination of Atorvastatin Calcium in their pharmaceutical formulation(tablets). In this paper simple, rapid, accurate and sensitive spectrophotometric methods have been developed and validated. This method is a direct spectrophotometric analytical method depend on dissolve of atorvastatin calcium in n-butanol. The maximum absorption wavelength for determination ofATV drug was found to be 271 nanometer (nm) for Beer's law was obeyed in the concentration range from 5 to 40 µg/ml for UV-spectrophotometric method

Keywords: Atorvastatincalcium, UV spectroscopy, Tablets.

I. INTRODUCTION

Atorvastatin is mainly used as an antihyperlipidemic agent in cardiovascular risk conditions. Atorvastatin belongs to the class of antihyperlipidemic agents known as statins. It is intended for lowering cholesterol level in the body. It acts by enzyme inhibition mechanism. Atorvastatin act by competitively in hibiting the 3-hydroxy-3-methylglutarylCo-enzyme-A(HMG-CoA) reductase. HMG- CoA reductase is a rate determining enzyme in in the biosynthesis of cholesterolvia mevalonate pathway. This enzyme catalyzes the HMGCoA conversion into mevalonate. Atorvastatin primarily shows its action in liver. It causes the decrease in hepatic cholesterol level, hence hepatic uptake of cholesterol increases and it results in lowering ofplasma cholesterol level. Statins can reduce mortality and morbidity associated with coronary heart disorder. Atorvastatin appears as white crystal line powder. It is practically in soluble in water, slightly soluble in methylene chloride and soluble in methanol.

Atorvastatincalcium is chemically{[R-(R,R*)]-2-(4-flurophenyl)-ß,□-dihydroxy-5-(1-methylethyl) -3- phenyl-4-[phenylamino) carbonyl]-14- pyrrole-1-heptanoic acid, calcium salt (2:1) trihydrat. It is available in commercial pharmaceutical formulations for the treatment of hypercholesterolemia. it is effective to reduce both cholesterol and triglycerides. Main objectiveis to develop and validate UV visible method.

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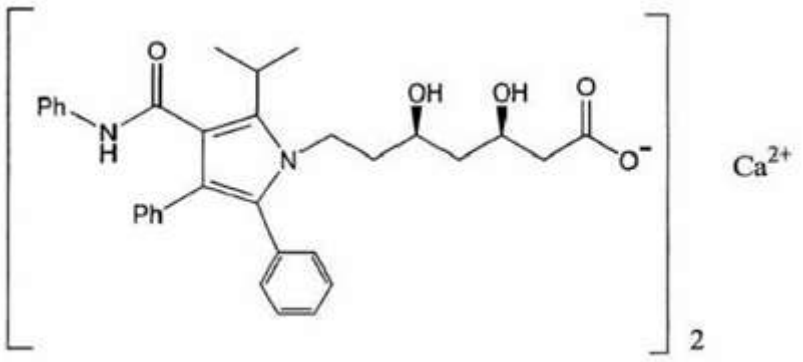
STRUCTURE:



VALIDATION:

Establishing documentation evidence, which provides a high degree of assurance that specific process, will consistently produce a product meeting its predetermined specifications and quality attributes.

System Suitability:

It is a checking of a system to ensure system performance before or during the analysis of unknowns. System solubility tests are an integral part of UV methods and they verify the resolution and reproducibility of the system are adequate for the analysis to be performed %RSD.

Accuracy (%Recovery):

For accuracy determination, three different concentrations were prepared separately i.e. 50%, 100%, and 150% for the concentration of absorbance values are recorded for the same.

II. MATERIALS AND METHODS:

Instrumentation: A Shimadzu UV -1800 240 UV/VISIBLE Spectrophotometer was used having two matched 1 cm matches quartz cell.

Materials: The Atorvastatincalcium 20mg were gifted by Medrich Limited, Bengalore. The solvents used is Butanol.

Methods:

PREPARATION OF STANDARD STOCK SOLUTION:

10 mg of Atorvastatin calcium was accurately weighted and transferred into 10 ml volumetric flask. About 7ml of butanol was added and sonicated to dissolve it completely. Then made volume up to the mark with the same solvent. The concentration of the resultant 1000µg/ml. Further standard stock solution was prepared by diluting it with butanol to get 100µg/ml. Again solution was dilution has made with the same solvent to get a concentration of 10µg/ml. The solution was scanned between 200-400 nm ranges against blank. From the UV spectrum,271nm was selected as absorption maximum for the drug Atorvastatin calcium

METHOD VALIDATION OF UV SPECTROSCOPY

The method was developed and validated according to the analytical procedure as per the ICH guidelines for validation of analytical procedures in order to determine linearity, accuracy, precision, robustness, ruggedness, LOD, LOQ.

- **Linearity:**

The analytical method should be line ari-e, should be a direct relationship between the concentration of the analyte (s) and the signal produced

Linearity is usually evaluated by analysing sample containing the analyte at5 different concentration level. the correlation coefficient, Y-intercept, slope of the regression line and residual sum of square should be calculated.

- **Accuracy:**

Accuracy is the closeness of the test result to the true or theoretical value accuracy is assessed by using minimum of a determination over a minimum of 3 concentration levels

Accuracy is then reported as a percentage recovery of the theoretical amount of analyte in the sample together with confidence interval.

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➢ **Precision:**

a.) Inter-day precision:

It was done by analysing the solution by same analyte on alternate day till 5th day result indicatethatthesolutionisstableupto1day.Thereafterd egradationmayhavetakenplace leading lower percent label claim.

b.) Intra-day precision:

It was done by analyzing the solution by same analyst within a day result indicates that the solution is stable up to 1daythereafter degradation may have taken place in the solution.

➢ **Limit Of Quantitation (LOQ):**

LOQ is the lowest amount of an analyte that can be quantitated with suitable accuracy and precision.

➢ **Limit of Detection(LOD):**

LOD is the lowest amount of an analyte that can be detection but not necessary quantitation

➢ **Robustness:**

The robustness of the proposed assessed method was with changes in the analytical wavelength (271±1nm). Robustness was carried out at two different concentration levels (2 and 20µgmL-1) the results was expressed as standard deviation and relative standard deviation and are compiled the results revealed that the slight changes in the analytical wavelength did not adversely influence the absorbance intensity and indicate acceptable robustness of the proposed method.

➢ **Ruggedness:**

Ruggedness of the proposed method was evaluated by comparison of the absorbance of reaction that have been measured by two different analyte. In the same laboratory. Ruggedness carried out at two different concentration level (2and 20µg/ml-1). The results are expressed as standard deviation and relative standard deviation.

III. RESULTS AND DISCUSION

Solubility studies

The solubility of drug indifferent solvents were studied. Since, the drug is polar in nature, different polar solvents like Methanol, Ethanol, Butanol, Aniline and water were studied. From the solubility studies, it was found that the drug Atorvastatin calcium is soluble in Butanol. The results are given in Table.

S.no	Solvents used	Parts of solvent required for parts of solute (mg/ml)	Descriptive
1	Methanol	50-100	Freely soluble
2	Aniline	50-100	Soluble
3	Butanol	10-50	Sparingly soluble
4	Ethanol	10-50	Sparingly soluble
5	Water	<0.1	Insoluble

TABLE1: SOLUBILITY STUDIES IDENTIFICATION OF DRUG

MELTING POINT:

Instrument	Drug name	Standard values	Observed value
Melting point apparatus-Laboratory setup	Atorvastatin calcium	164-180	172^0 c

TABLE2: MELTING POINT

International Journal of Pharmaceutical Research and Applications
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IR SPECTROSCOPIC METHOD

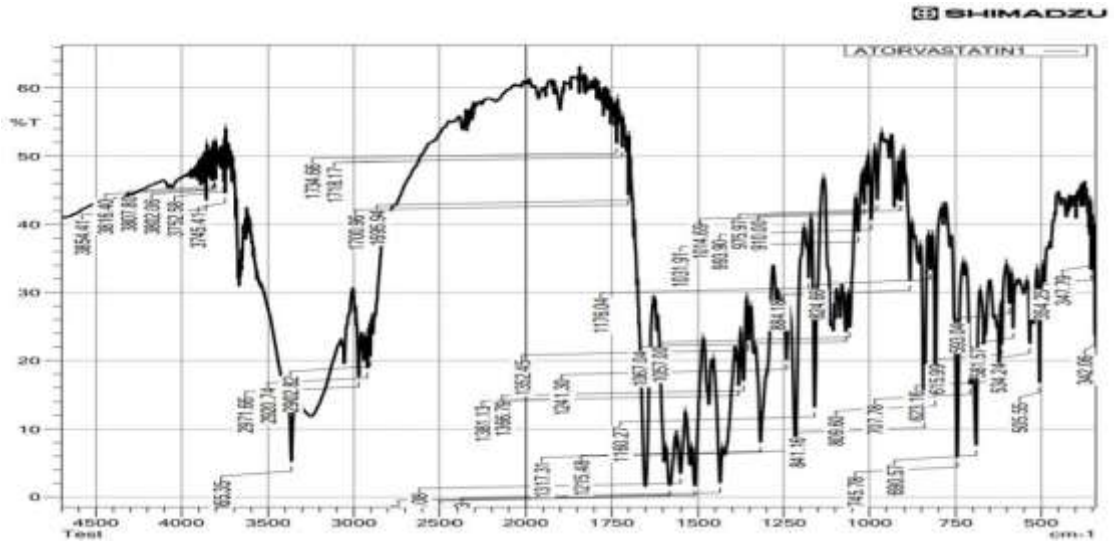

TABLE3: INTERPRETATION OF IR SPECTRUM OF ATORVASTATINCALCIUM

S.NO	Frequency	Mode	Functional group
1	3365.35	O-Hstretch	Hydroxy group
2	2971.66,2902.82	C-H stretch	Alkyl halides
3	1735.35,1712.08	C=O stretch	Carbonyl group
4	1635.54	C=N stretch	Imidazole
5	1274.09,965.45	C-Ostretch	Ester
6	446	Ca-Ostretch	Calcium salt

METHODDEVELOPMENT OF UV SPECTROSCOPY:

SNO	**P/V**	**WAVE LENGTH**	**ABSORBACE**
1		**271.50**	**0.321**
2		**224.50**	**0.050**

TABLE4: LAMBDAM AXPROFILE OF ATORVASTATINCALCIUM

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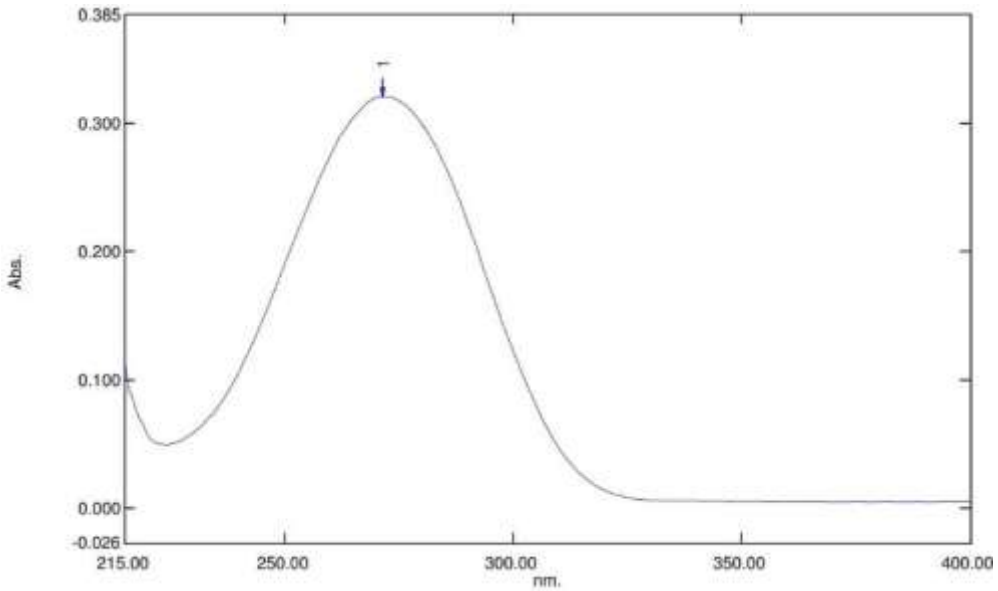

FIGURE2: ABSORPTION SPECTRA OF ATORVASTATINCALCIUM

DETERMINATION OF PERCENT AGEPURITY:

S.NO	Standard absorbance	Sample absorbance	Percentage purity (%)	Average% purity	SD	%RSD
1	**0.847**	**0.854**	**99.43%**			
2	**0.851**	**0.831**	**99.83%**			
3	**0.857**	**0.895**	**100.08%**	**98.76%**		
4	**0.838**	**0.811**	**95.82%**		**0.0173**	**1.7533**
5	**0.842**	**0.860**	**98.64%**			

TABLE 5: %PURITY

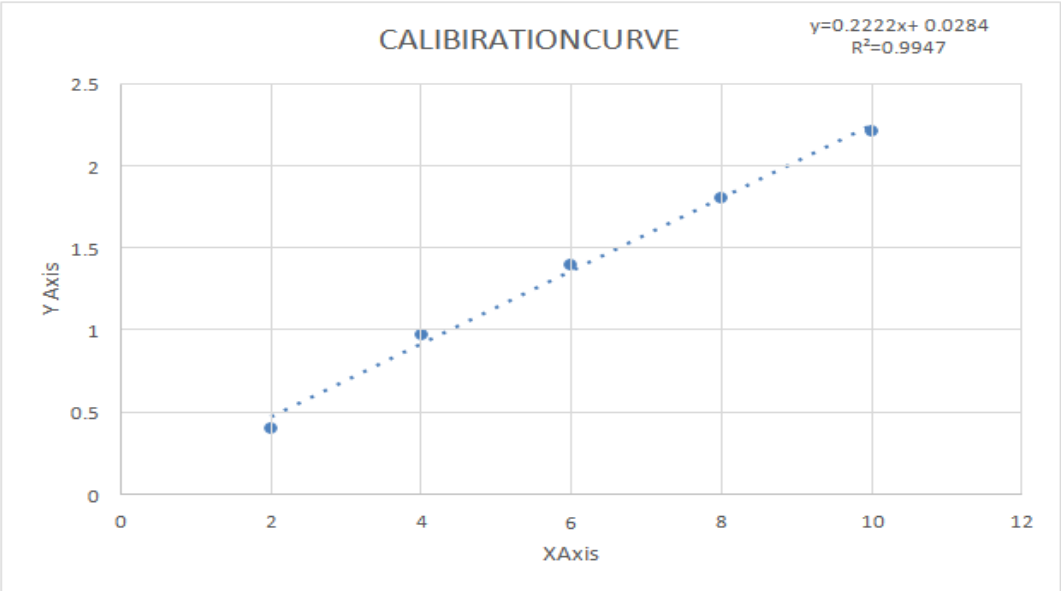

FIGURE3: CALIBRATI ON CURVE

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Sno	Conc	Absorption	Average	Correlation	LOD	LOQ	Slope	intercept
	µg/ml		absorption	coefficient				
1 2 3 4 5	2 4 6 8 10	0.408 0.977 1.401 1.807 2.215	1.3616	0,9993	2.064	6.256	0.222	0.028

TABLE7: LINEARITY

SE of intercept =0.06228 SD of intercept=0.13890 LOD =2.064796
LOQ =6.256958
Slope= 0.2222
Intercept=0.0284 Y = ax + b
Slope = a Intercept=b
The number of testes =N
SE of intercept Excel function (data analysis regression) SD of intercept = SE of intercept* √N
LOD-3.3×(SD of intercept/slope) LOQ=10*(SD of intercept/slope)

PRECISION

A.) Inter day precision

S.No	Absorbance	Average	SD	%RSD
1 **2** **3** **4** **5**	**0.447** **0.441** **0.438** **0.432** **0.431**	**0.437**	**0.0066**	**1.509**

TABLE8: INTER DAY PRECISION

B.) Intra day precision:

S.no	Absorbance	Average	SD	%RSD
1 2 3 4 5	0.448 0.446 0.442 0.436 0.432	0.4408	0.0067	1.525

RUGGEDNESS:

S.NO	Analysts	Conc(µg/ml)	Absorbance	SD	%RSD
1	Analysts-1	2µg/ml 2µg/ml 2µg/ml 2µg/ml 2µg/ml	0.489 0.486 0.498 0.488 0.496	0.00527	1.0729
2	Analysts-2	2µg/ml 2µg/ml	0.488 0.499	0.00645	1.3103

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		2µg/ml	0.484		
		2µg/ml	0.496		
		2µg/ml	0.497		

TABLE10: RUGGEDNESS

ROBUSTNESS:

S.no	Wavelength	Absorbance
1	233	0.858
2	235	0.845
3	237	0.842

TABLE11: ROBUSTNESS

IV. CONCLUSION:

The method were found to be rapid, economical, accurate and precise for the determination of Atorvastatin calcium in bulk drug in tablet by UV-Spectrophotometer methods produce comparable results can be used for precise and accurate analysis of Atorvastatin calcium in its pure and tablet dosage form. The values of % recovery was close to 100% indicating reproducibility and accuracy of the proposed method successfully employed as a quality control toolfor the analysis of Atorvastatin calciumin its tablet dosage formand in bulk drug.

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