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Method Development and Validation of Hypolipidemic Drug by using UV Spectroscopy

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Abstract: *Hypolipidemic drugs are therapeutic agents designed to lower lipid levels, particularly cholesterol and triglycerides, in the blood. These drugs play a crucial role in managing conditions such as hyperlipidemia, cardiovascular diseases, and metabolic disorders. As the global prevalence of such diseases continues to rise, the demand for effective hypolipidemic treatments grows, prompting ongoing research and development in this field. Rosuvastatin, belongs to class of statins. Which are used to treat the high cholesterol and use to prevent the cardiovascular disease. Rosuvastatin chemically is (3R,5S,6E)-7[4-(4-Flurophenyl)-2- (N-Methyl- methanesulfonamido)-6-(Propan-2-ylPyrimidin-5-yl]-3,5-dihydroxyhept-6-enoic acid.[1] Spectroscopy offers a promising alternative due to its rapid, non-destructive nature and minimal sample requirements.*

Keywords: Hypolipidemic drugs

I. INTRODUCTION

Hypolipidemic drugs are therapeutic agents designed to lower lipid levels, particularly cholesterol and triglycerides, in the blood. These drugs play a crucial role in managing conditions such as hyperlipidemia, cardiovascular diseases, and metabolic disorders. As the global prevalence of such diseases continues to rise, the demand for effective hypolipidemic treatments grows, prompting ongoing research and development in this field. Rosuvastatin, belongs to class of statins. Which are used to treat the high cholesterol and use to prevent the cardiovascular disease. Rosuvastatin chemically is (3R,5S,6E)-7[4-(4-Flurophenyl)-2- (N-Methyl- methanesulfonamido)-6-(Propan-2-ylPyrimidin-5-yl]-3,5-dihydroxyhept-6-enoic acid.[1] Spectroscopy offers a promising alternative due to its rapid, non-destructive nature and minimal sample requirements. Techniques such as UV-Visible spectroscopy have gained traction in pharmaceutical analysis due to their ability to provide precise quantitative data with relatively straightforward methodology. The methodology involves the optimization of key parameters, including solvent selection and wavelength determination, followed by the construction of a calibration curve to assess the linearity of the response. Comprehensive validation will be conducted to evaluate the method's accuracy, precision, specificity, and robustness. The objective of the present study is to develop a new simple, sensitive, accurate, rapid and economic method for the estimation of Rosuvastatin.[2]

ULTRAVIOLET SPECTROSCOPY (UV)

UV spectroscopy includes measuring the absorbance of UV light by a test. Atoms retain particular wavelengths of UV light, which permits for the recognizable proof and measurement of substances. UV spectroscopy may be a capable and flexible apparatus that plays a vital part in different logical areas, giving profitable bits of knowledge into the composition and behavior of distinctive substances. Its capacity to quickly and precisely analyze tests makes it a fundamental strategy in both investigate and industry. UV spectroscopy determining the concentration of substances in a solution, such as proteins, nucleic acids, and pharmaceuticals. UV spectroscopy Identify compounds based on their unique absorption spectra.

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INSTRUMENTATION OF UV :

Light Source:

Deuterium Lamp: Emits continuous UV light, typically covering the range of 190 to 400 nm. It's commonly used for UV spectroscopy due to its stability and broad spectrum.

Tungsten-Halogen Lamp: Used for the visible spectrum (320 to 800 nm) and sometimes in conjunction with the deuterium lamp.

Monochromator:

Prism or Grating: Used to disperse light into its component wavelengths. The monochromator selects a specific wavelength to pass through the sample.

Slits: Adjustable slits control the bandwidth of the light beam that enters the sample, affecting resolution and sensitivity.

Sample Holder (Cuvette):

Cuvettes: Typically made of quartz for UV analysis (to avoid absorption by glass) or plastic for visible light. The cuvette holds the sample solution.

Detector:

Photodiode: Commonly used for detecting light intensity; it converts light into an electrical signal.

Photomultiplier Tube (PMT): More sensitive than photodiodes, often used for low concentration measurements.

Data Acquisition System:

Computer/Software Interface: Captures the electrical signal from the detector, processes the data, and displays the results as absorbance or transmittance spectra.

Control Unit:

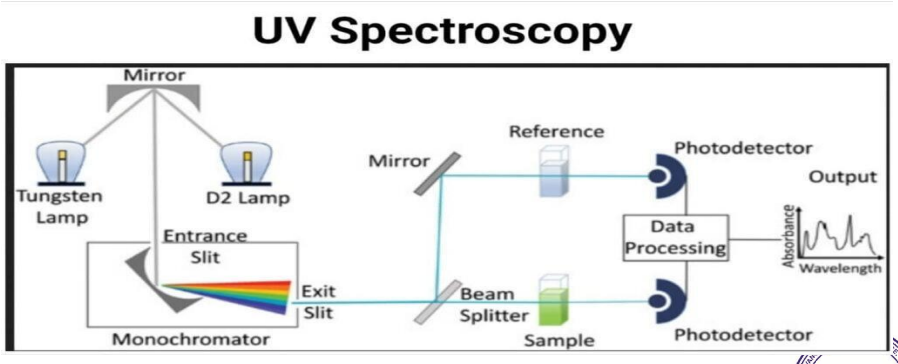
User Interface: Allows users to set parameters such as wavelength, scan speed, and data collection methods. It also manages calibration and data storage.

Temperature Control (Optional):

Some spectrophotometers include temperature control for the sample, which can be important for stability and reproducibility in certain analyses.

PRINCIPLE OF UV SPECTROSCOPY:

UV- Vi's spectroscopy is based on the interaction between light and matter. When light passes through or is absorbed by a molecule, it can cause the molecule to vibrate. The wavelength of light that is most strongly absorbed by a molecule is called the absorption maximum. By measuring the absorbance of light at different wavelengths, it is possible to identify and characterize molecules.



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The development of UV has achieved great success in providing following

Features:

- Wavelength Range
- Absorbance Measurement
- Qualitative Analysis
- Quantitative Analysis
- Speed and Efficiency
- Non destructive efficacy
- Sensitivity
- Ease of use
- Wide Range of Application
- Compatible with various sample types
- Integration with other Techniques

Advantages of UV:

- High Sensitivity
- Rapid Analysis
- Non-Destructive
- Quantitative Capabilities
- Qualitative Identification
- Versatile Applications
- Minimal Sample Preparation
- Wide Range of Sample Types
- Ease of Use
- Cost-Effective
- Integration with Other Techniques

II. LITERATURE REVIEW

B. Sailaja.al., (2016) Develop a new, simple, rapid validated UV method for the estimation of rosuvastatin calcium spectrophotometric analysis was performed using double beam UV visible spectrometer (LABINDIA3000) the solvent selected for the study was 0.1N NAOH and the drug showed absorption maxima at 240nm.Several methods used for the validation of the developed method such as accuracy, precision, limit of detection, limit of quantitation. The method was validated according to ICH guidelines.

Aika Gupta*al., (2008) Proposed method for determination of slowed molar absorptivity of 7.2345 into 10 to the power 4 L/mol.cm by using a GBC cintra-10 double beam UV visible spectrophotometer (Australia) and Sartorius analytical balance was used. The chemical that was used methanol (AR) (Qualigens) in present study Thus it can be said that the proposed method is precise, accurate, and economical which can be very well applied to the marketed.

Sireesha. D al., (2017): Develop a new method for the simultaneous estimation and validation of rosuvastatin and ezetimibe in pharmaceutical dosage form by UV. The method was carried out by using Shimadzu double beam UV model UV1800s. The solvent selected for the method was distilled water. Rosuvastatin showed absorption maxima at 223 nm and Ezetimibe showed absorptivity at 220 nm. The method was validated by various validation parameters such as accuracy, precision, linearity, sensitivity as per the ICH guidelines.

Asma Afroz al., (2011): proposed accurate and reproducible spectrophotometric method has been developed and validated by using a double beams SHIMADZU (Kyoto, Japan) UV visible spectrophotometer model uv-1601pc and the wavelength was selected from the overlain spectra. The methanol and sodium hydroxide were analytical reagent grade and purchased from E. Merck, Darmstadt, Germany. Water was deionized and double distilled.

Suvarna G. Bhokare al., (2018): A sensitive, fast UV spectroscopy method was developed and validated for estimation of rosuvastatin in pharmaceutical dosage form the analysis was performed by using the UV. The UV (w-1800

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Shimadzu Japan) the solvent use in method was phosphate buffer of PH range 7.4 the solution was scanned in UV over the 7 P. Wadhwani College of Pharmacy, Yavatmal range 200 – 400nm rosuvastatin should be absorption should absorption maximum at 246nm. The methods used for the validation where linearity and range, accuracy, precision and accurate way for estimation of rosuvastatin in pharmaceuticals dosage form. This analytical method for estimation of rosuvastatin has been validation protocol of ICH guidelines.

Rekha Rajeev Kumar al., (2012): A straightforward, particular and financial spectroscopic strategy has been created for the estimation of Rosuvastatin calcium in bulk and tablet measurement frame. Within the created strategy water was utilized as the dissolvable. The assimilation most extreme of the medicate was found to be 241nm. The medicate takes after a direct Lambert-Lager law relationship with regard to the sedate concentration within the run of 5-30ug/mL, with linearity coefficient of 0.9998. Factual investigation and recuperation ponder approved the strategy. The proposed strategy was found to be fast, exact and exact and can be connected for the schedule estimation of Rosuvastatin Calcium within the research facility.

AIM & OBJECTIVES

Aim:

To develop and validate hypolipidemic drug for assessing, efficacy, safety and compatibility in development by using UV spectroscopy.

Objectives:

Primary Objectives:

- Validate the method according to regulatory guidelines (e.g., ICH, FDA), assessing parameters such as specificity, linearity, accuracy, precision, limit of detection (LOD), and limit of quantification (LOQ).
- To obtain consistent, reliable and accurate data from the experimentation.
- Determine the optimal wavelength for maximum absorbance and minimal interference.

Secondary Objectives:

- Validate the developed method according to standard criteria, including:
- Specificity
- Linearity
- Precision
- Accuracy
- Limit of Detection (LOD) and Limit of Quantification (LOQ)
- Robustness

Drug profile

NAME OF DRUG: ROSUVASTATIN

MOLECULAR FORMULA: C22H28FN3O6S

IUPAC NOMENCLATURE: [3R,5S,6E]-7-[4-(4-Flurophenyl)–2-(N-Methyl-methanesulfonamido)-6-(propan-2-yl) – 3,5-dihydroxyhept – 6- enoic acid

MOLECULAR MASS: 481.539 g\mol

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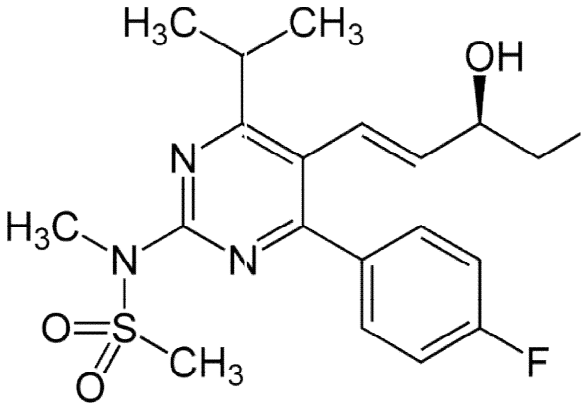
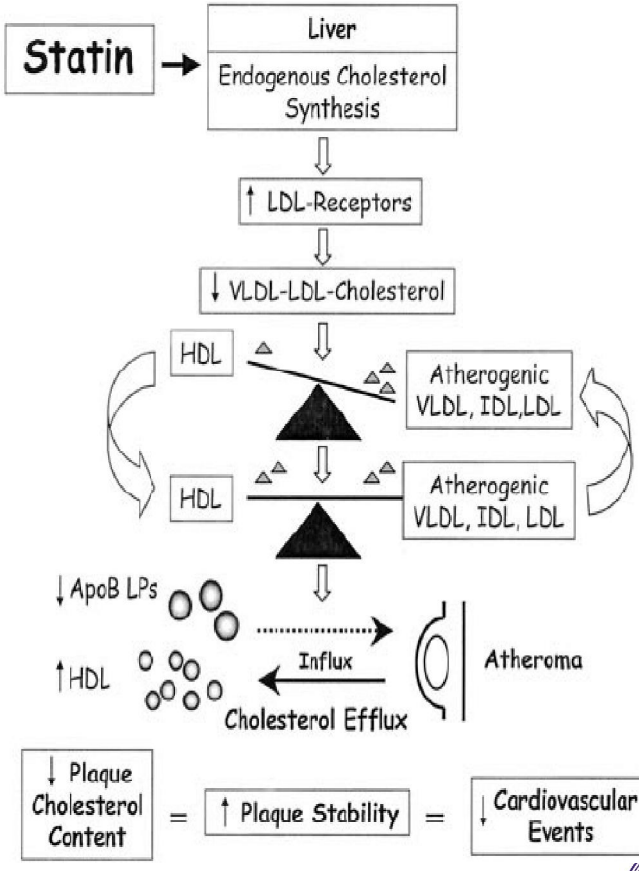
STUCTURE



SOLUBILITY OF DRUG: Ethanol, Methanol, Water **DRUG PHYSICAL FORM:** Powder form **COLOUR:** White
STATE: An amorphous powder
MELTING POINT: 122 degrees Celsius
CHEMICAL CLASS: HMG CoA reductase inhibitor; Statin
DOSAGE FORM: Film coated tablet
MECHANISM OF ACTION: Rosuvastatin are hypolipidemic drug which absorbed in gut then it inhibits hydroxy methyl glutaryl-coenzyme which is responsible formation cholesterol help in form lipid so decrease level of lipid, fat in body



PLAN OF WORK

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II. LITERATURE REVIEW

Collect necessary solvents and reagents (e.g., methanol, buffer solutions).
Determination of λmax by UV – spectroscopy.

METHOD DEVELOPMENT.

- Selection of Wavelength
- Preparation of Solutions
- Calibration Curve
- Linearity
- Accuracy
- Precision
- Specificity
- Robustness

Methodology:

Linearity: In UV spectroscopy refers to the relationship between the absorbance of a sample and its concentration. This relationship is crucial for quantitative analysis, as it allows for the accurate determination of the concentration of an analyte in a solution. The principle underlying linearity is encapsulated in the Beer-Lambert Law, which states that absorbance (A) is directly proportional to the concentration (c) of the absorbing species and the path length (l) of the light through the sample.

$$A=\varepsilon \cdot c \cdot l$$

Where:

A= Absorbance (no units, as it is a logarithmic ratio)

ε = Molar absorptivity ($L \cdot mol^{-1} \cdot cm^{-1}$), a constant that indicates how strongly a substance absorbs light at a given wavelength

c = Concentration of the absorbing species ($mol \cdot L^{-1}$)

L = Path length of the sample (cm), typically the width of the cuvette.

- **Accuracy:** In UV spectroscopy, accuracy refers to how close the measured absorbance values (and, by extension, the calculated concentrations of the analyte) are to the true or actual values. It reflects the reliability of the method in producing results that correctly represent the concentration of the substance being analyzed.
- **Precision:** In UV spectroscopy, precision refers to the degree of consistency and reproducibility of measurements when analyzing the same sample under identical conditions. It indicates how closely related the results of multiple measurements are to each other, regardless of whether those measurements are close to the true value
- **Specificity:** In UV spectroscopy, specificity refers to the ability of a method to accurately measure the analyte of interest in the presence of other components, such as impurities, excipients, or other substances in a sample. It indicates how well the method can distinguish the target compound from other substances that may absorb light at similar wavelengths.
- **Robustness:** In UV spectroscopy, robustness refers to the ability of an analytical method to remain unaffected by small, deliberate variations in method parameters while still producing reliable and consistent results. It indicates the method's reliability and stability under different conditions, which is essential for ensuring that the analysis remains valid in real-world applications.
- **Sensitivity:** In UV spectroscopy, sensitivity refers to the ability of a method to detect and quantify low concentrations of an analyte in a sample. It indicates how effectively the instrument can respond to small changes in absorbance corresponding to changes in concentration. High sensitivity means that even minute amounts of the analyte can be accurately measured

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Limit of Detection (LOD): 3.3 N|B
Where:
N= Standard deviation of the peak areas of the drugs. B= Slope of the corresponding calibration curve
Limit of Quantification (LOQ): 10 N|B

EXPECTED OUTCOMES

- **Safety Assessment:** Contaminant Detection: Establish a method to detect potential contaminants or degradation products in rosuvastatin formulations that could affect safety.
- **Efficacy Evaluation:** Concentration Analysis: Accurately quantify rosuvastatin concentrations in pharmaceutical preparations and biological samples to ensure that effective doses are maintained.
- **Mechanism of Action:** Pharmacokinetic Studies: Use the validated UV method to analyze rosuvastatin levels in biological matrices (like plasma) over time, providing insights into its pharmacokinetics and mechanism of action in lipid regulation.
- **Side Effects:** Detection of Toxic Metabolites: Use the developed method to monitor metabolites associated with adverse effects, contributing to a better understanding of the drug's safety profile.
- **Compatibility:** Formulation Studies: Assess the compatibility of rosuvastatin with various excipients and formulations to ensure stability and effectiveness in final pharmaceutical products.
- **Method Development Outcomes: Standard Operating Procedures (SOPs):** Creation of detailed SOPs for sample preparation, instrument calibration, and data analysis.
- **Method Validation Outcomes:** Linearity and Range: Establishment of a linear relationship between absorbance and concentration over a specified range, confirming the method's reliability

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