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MAGNESIUM –PICRATE COMPLEXES AS COUPLING AGENTS TO ENHANCE UV-VISIBLE SPECTROPHOTOMETRIC DRUG ANALYSIS

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ABSTRACT

This research investigates the production of magnesium-picrate complexes and their use as coupling agents to improve UV-Visible spectrophotometric detection and quantification of vital medicinal medicines. The analyzed pharmaceuticals comprise Diclofenac Sodium, Ciprofloxacin HCl, Levoceitrizine 2HCl, Levofloxacin Hemihydrate, Nimesulide, Piroxicam BCD, and Terbinafine HCl. The synthesized magnesium-picrate complexes, upon heating with medication solutions, exhibited substantial increases in absorbance at their specific characteristic wavelengths. The increases were documented as 70.82%, 47.95%, 64.11%, 64.05%, 39.24%, 81.16%, and 87.10%, respectively, reflecting increased molar absorptivity. The 90% yield of magnesium-picrate complexes and their capacity to improve spectrum detection highlight their promise in optimizing UV-Visible spectrophotometric techniques for pharmaceutical quality assurance.

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KEYWORDS: Terbinafine HCl, Absorbance enhancement, Molar absorptivity, Spectrum detection, Pharmaceutical quality assurance, Drug quantification, Yield optimization
Analytical chemistry

1. Introduction:

UV-Visible spectrophotometry is a prevalent analytical technique for quality assurance in the pharmaceutical business, owing to its simplicity, cost-effectiveness, and resilience. The detection and quantification limits of numerous medications are limited by their inherent molar absorptivity, which directly affect sensitivity. [1]

Utilizing coupling agents to enhance these qualities can markedly improve analytical results. Magnesium-picrate complexes, recognized for their robust coordination chemistry and spectroscopic characteristics, present a viable solution. Enhancing the molar absorptivity of target chemicals allows these complexes to augment the sensitivity and precision of spectrophotometric investigations. This work intends to synthesize magnesium-picrate complexes and assess their effectiveness in enhancing the UV-Visible detection thresholds of various pharmaceuticals essential for pharmaceutical quality control.[2]

The findings of this research may facilitate the development of more dependable analytical techniques, thereby guaranteeing enhanced standards in drug safety and efficacy. These innovations would improve detection capabilities and ensure adherence to rigorous regulatory standards in the pharmaceutical sector. This may result in the creation of novel formulations and methods that enhance drug delivery systems, hence improving patient safety and therapeutic efficacy.[3]

Integrating these complexes into current analytical frameworks may enable researchers to provide new insights into drug interactions and stability, therefore enhancing the overall quality assurance processes in pharmaceutical manufacturing.

2. Experimental:

2.1. Sample preparation.

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2.1.1. Preparing the Magnesium Chloride Solution: Measure out 1 mmol of magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$) and dissolve this in 50 mL of deionized water. Stir the solution to ensure that it becomes clear, thus showing that the magnesium chloride is fully ionized.

2.1.2. Picric Acid Solution Preparation: Dissolve 1 mmol of picric acid (2,4,6-trinitrophenol) in 50 mL of ethanol in a separate container and stir the solution to complete dissolution until the result is a homogeneous yellow solution.

2.1.3. Solution Mix and Reaction Conditions: Add picric acid solution slowly to magnesium chloride solution with stirring in such a way that the mix is properly mixed up. Hold the temperature at 70°C for 3 hours in order to ensure formation of desired crystalline complexes. The continuous stirring prevents the local concentration gradients and gives uniform reaction conditions.

2.1.4. Isolation of Precipitate: After the expiry of the reaction time, let the solution cool to a little and filter the mixture leaving the solid precipitate and continue with the liquid. In cases where vacuum filtration is available, use it for more rapid filtration.

2.1.5. Washing and drying: The collected precipitate was washed thoroughly in ethanol, to remove possible impurities or un-reacted materials. Finally the filtered product was dried under an oven at 60°C for 24 hours thus removing all possible remaining solvent present. Thus finally the resultant product would turn out dry and stable.

2.1.6. Yield and product characteristics: Reaction typically gives a product yield of 90% in the form of yellow crystalline complexes. A complex of that sort will tell that, magnesium ions and picric acid molecules have successfully combined. This process will provide a highly pure product that may be characterized or used for applications in areas such as coordination chemistry or material science. [4] The high purity and stability of the resulting yellow crystalline complexes make them suitable candidates for further studies in catalysis, drug development, or as precursors in the synthesis of advanced materials. These applications highlight the versatility of these complexes, paving the way for innovative research and potential breakthroughs in various scientific fields.

2.2. Characterization of Magnesium-Picrate Complexes: The synthesized magnesium-picrate complexes characterized through Fourier-Transform Infrared Spectroscopy (FT-IR) were confirmed to be coordinated between magnesium ions and the picrate molecules. It is a very

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powerful analytical technique used for the identification of functional groups and detecting the formation of any new bonds on the basis of its vibrational frequencies.[5]

2.2.1. Key FT-IR Peaks:

I. **O-H Stretching (~3200 cm^{-1}):** Such broad O-H stretching is diagnostic for OH groups. Although the hydrogen bond, or coordination with magnesium reduces the intensity of this peak, its presence demonstrates some amount of residual hydroxyl remains. [6]

II. **Nitro Group Stretching (1550–1650 cm^{-1}):** The picrate molecule has nitro groups (-NO_2) that cause intense asymmetric and symmetric stretches within this range. This stretching pattern is important in assigning the structure of the picrate ligand in the complex.

III. **Metal-Ligand Coordination (450–500 cm^{-1}):** This new peak in the given region of the spectrum indicates coordination bonding of the magnesium ion with a ligand. It is an indication of a characteristic of metal complexes that coordinates the magnesium ions to the picrate ligand itself.

2.3. Preparation of Drug Solutions: The stock solutions were prepared to investigate the interaction of different pharmaceutical drugs with magnesium-picrate complexes. Drugs used in the experiments are those which are normally used for therapeutic effects and were dissolved in methanol for uniformity and solubility. Each drug was standardized to 10 µg/mL to maintain consistency within experiments. The drugs studied include:

- **Diclofenac Sodium:** This is a nonsteroidal anti-inflammatory drug used widely in pain and inflammation management. The graphical representation of diclofenac sodium with magnesium picrate complexes are given below.

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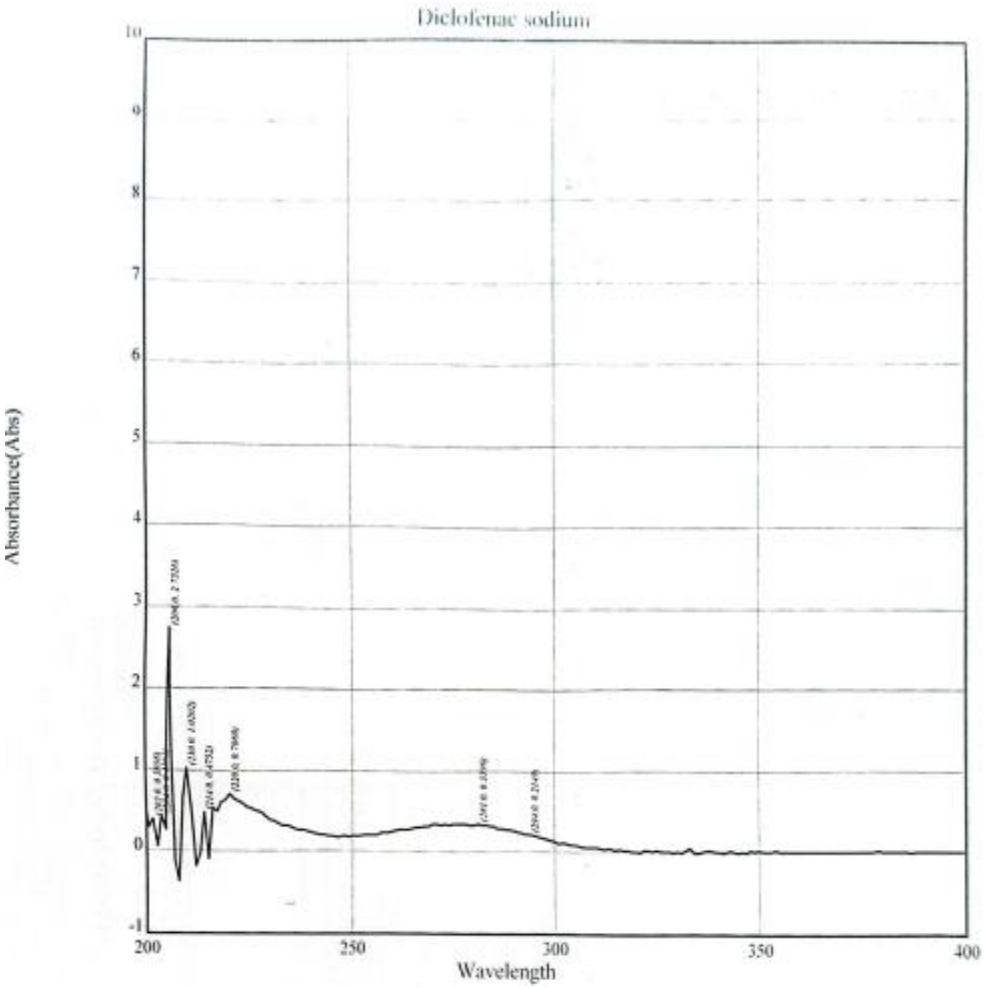

Fig.1.A. Diclofenac sodium without Magnesium picrate complex.

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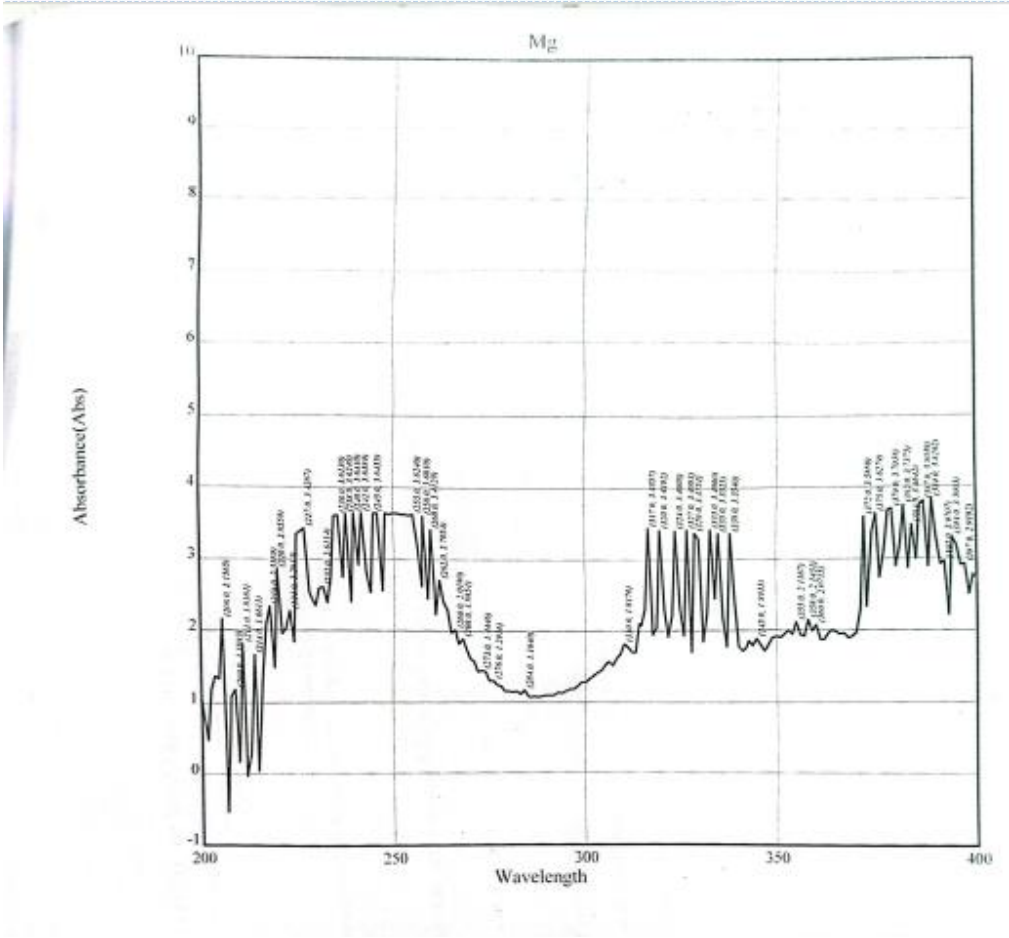

Fig.1.B. Diclofenac sodium with Magnesium picrate complex.

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- **Ciprofloxacin HCl:** It is a fluoroquinolone-class antibiotic with activity against most bacterial infections.

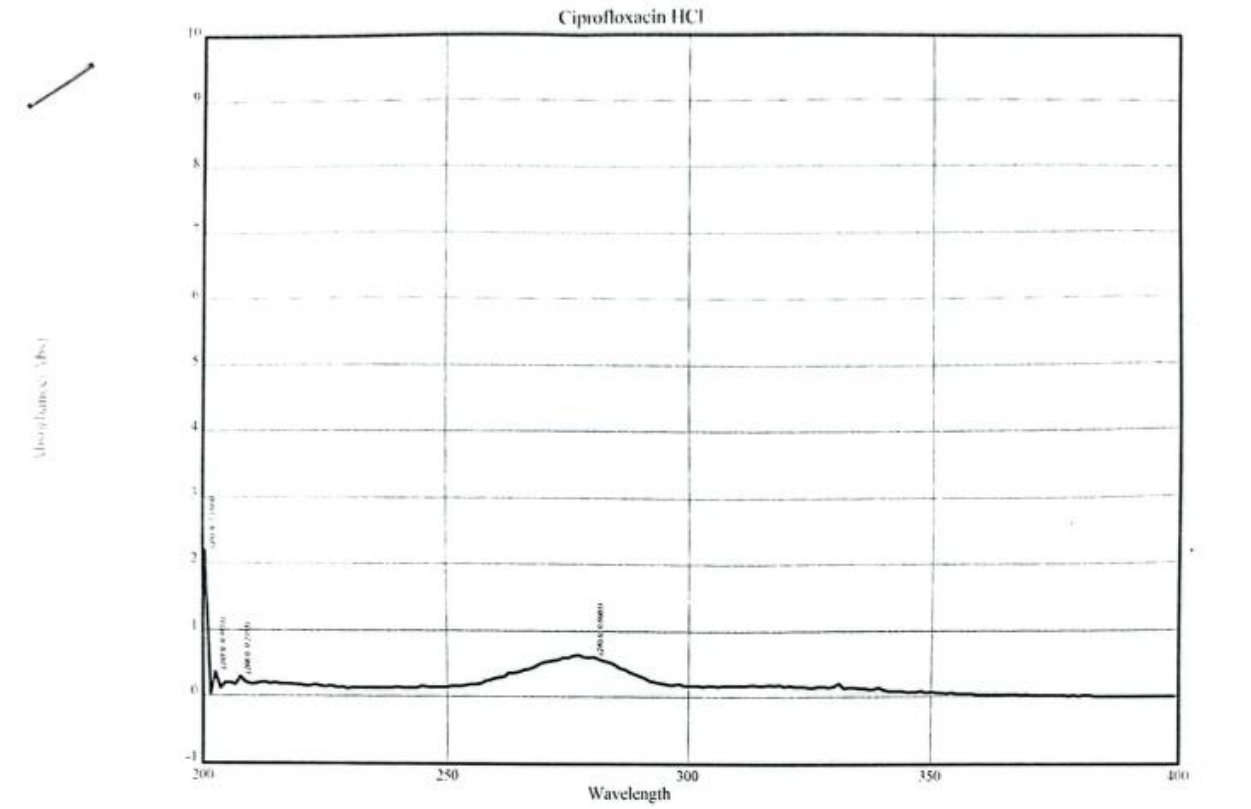

Fig.2.A. ciprofloxacin without magnesium picrate complex

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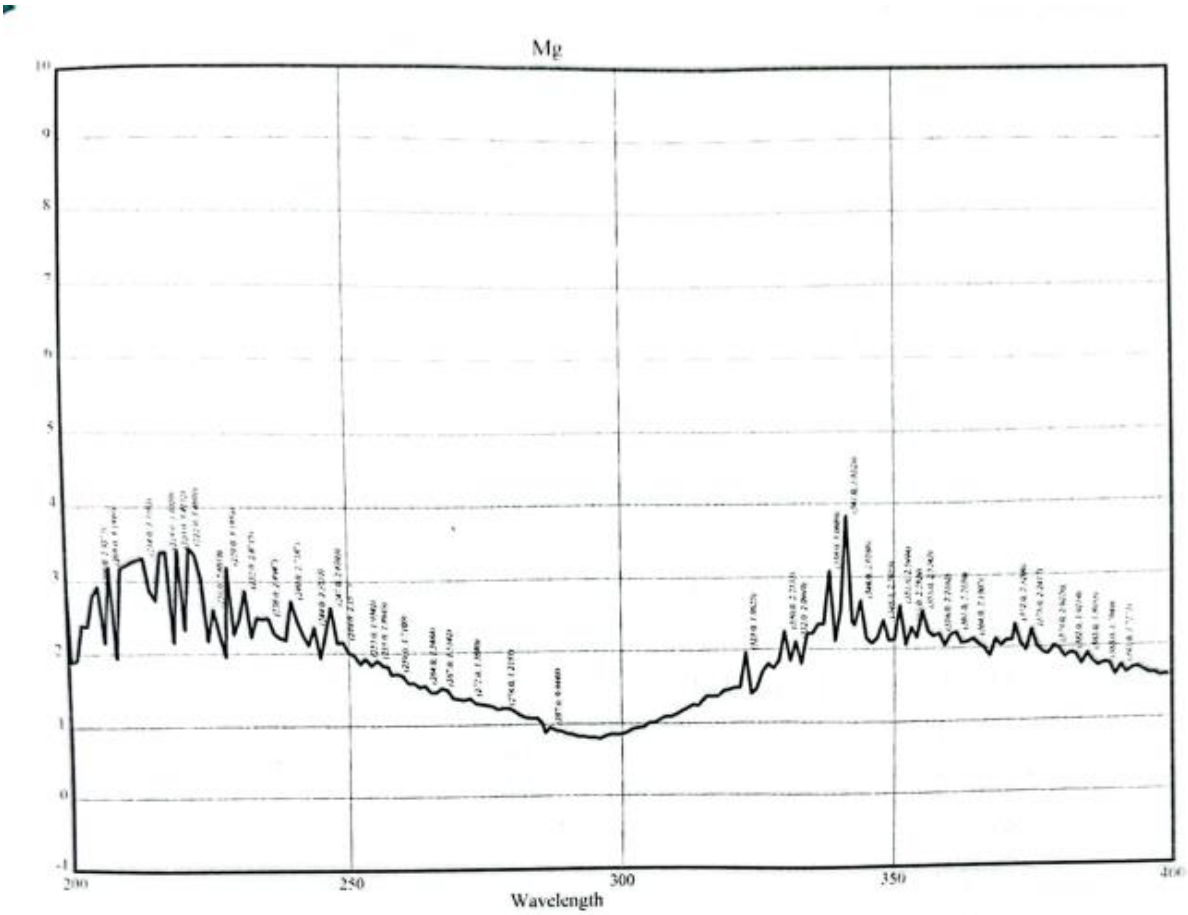

Fig.2.B. ciprofloxacin with magnesium picrate complex

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- **Levocetirizine Dihydrochloride (Levoceitrizine 2HCl):** Antihistaminic drug used in allergic conditions.

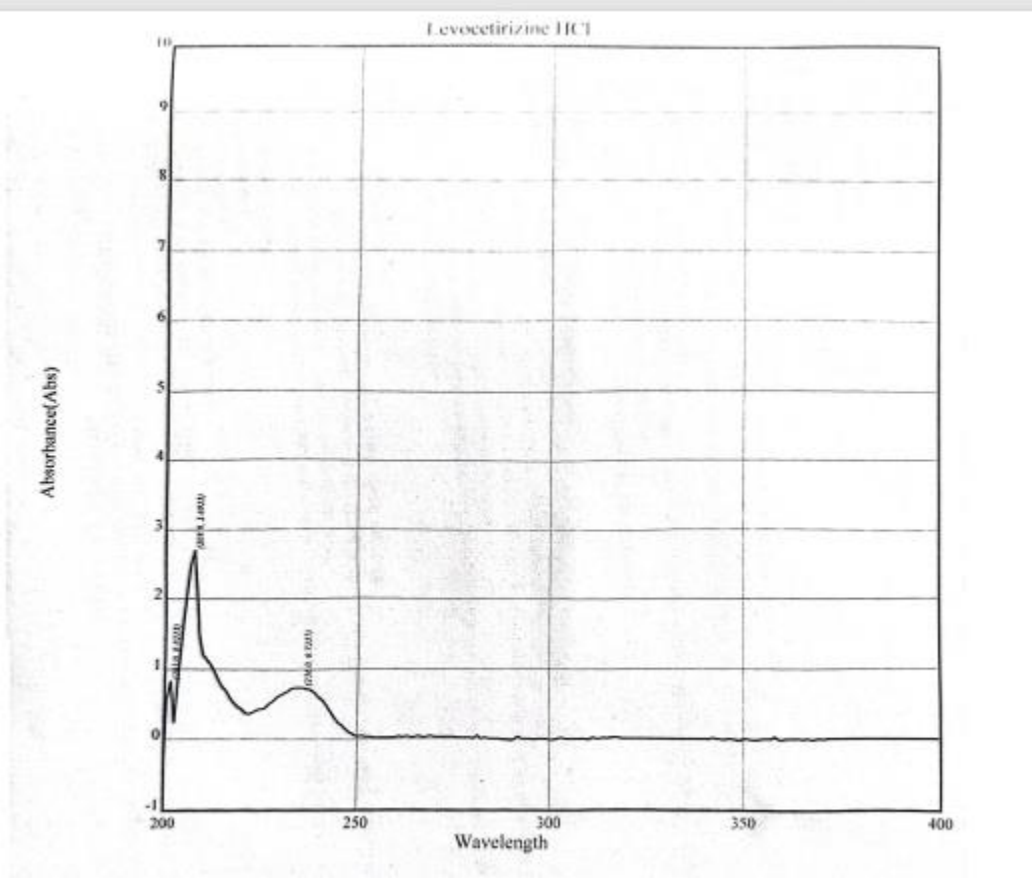

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Fig.3.A. Levocetirizine Dihydrochloride without magnesium picrate complex

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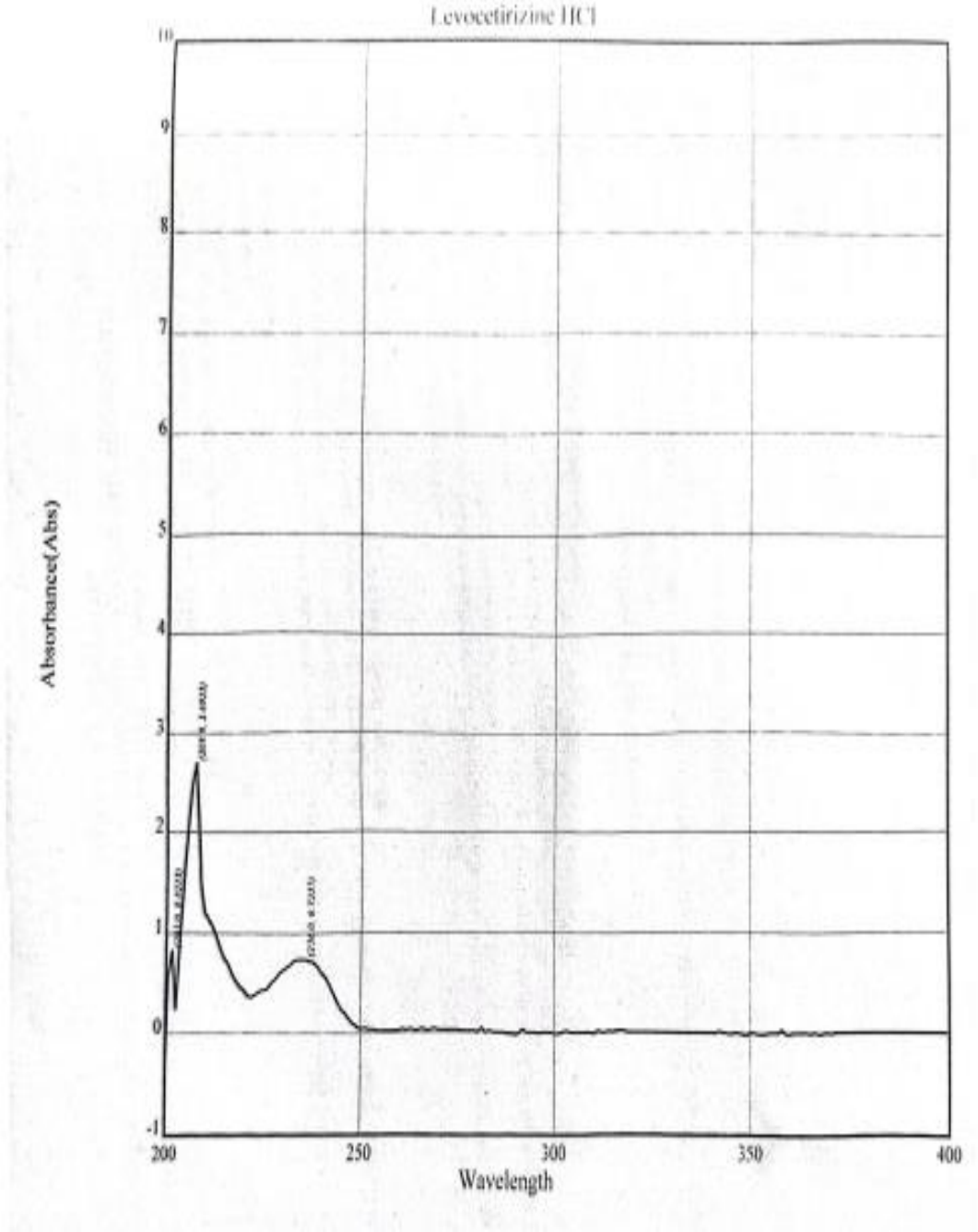

Fig.3.B. Levocetirizine Dihydrochloride with magnesium picrate complex

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- **Levofloxacin Hemihydrate:** Another fluoroquinolone antibiotic used in treating respiratory and urinary tract infections.

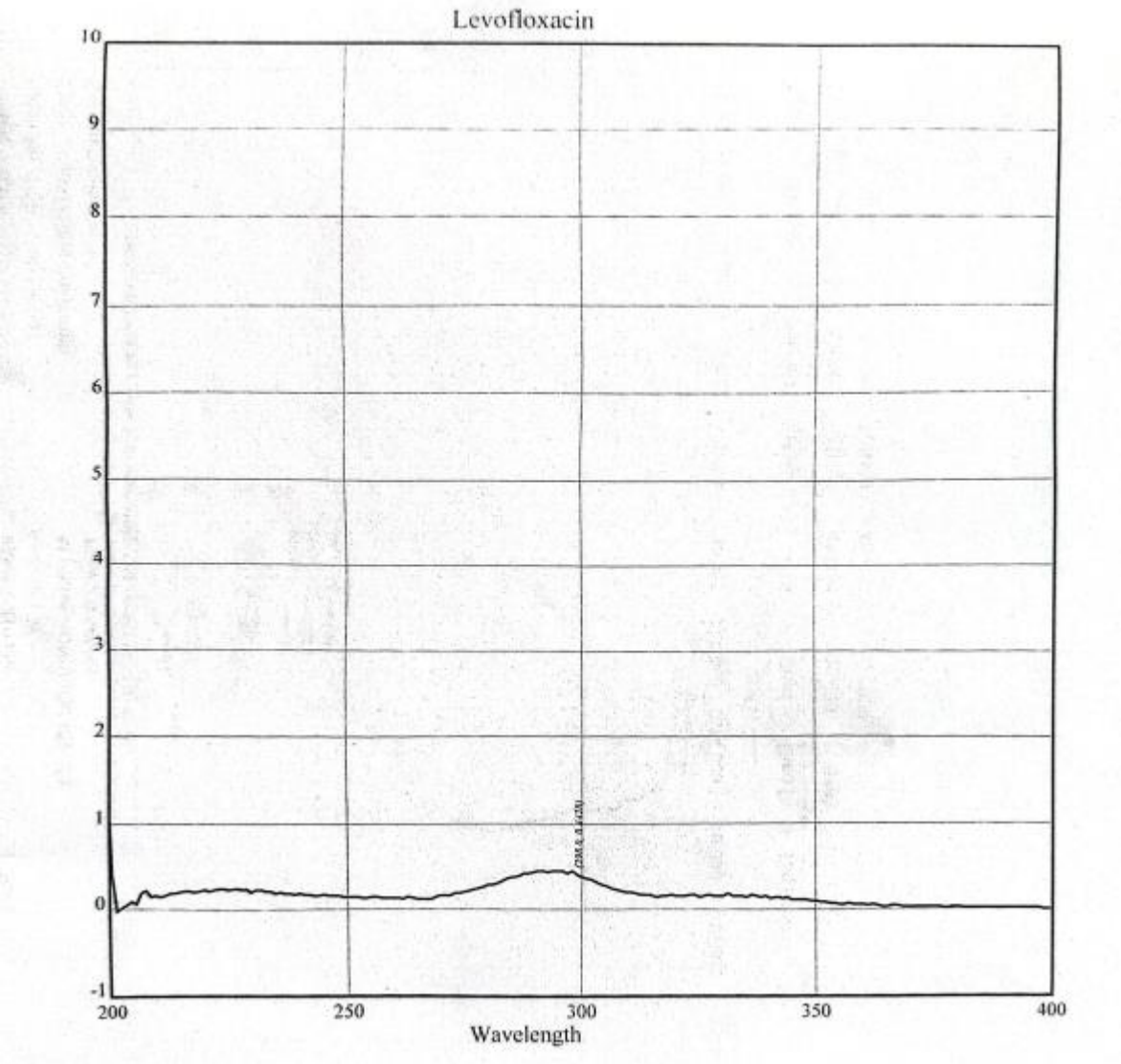

Fig.4.A. Levofloxacin Hemihydrate without magnesium picrate complex

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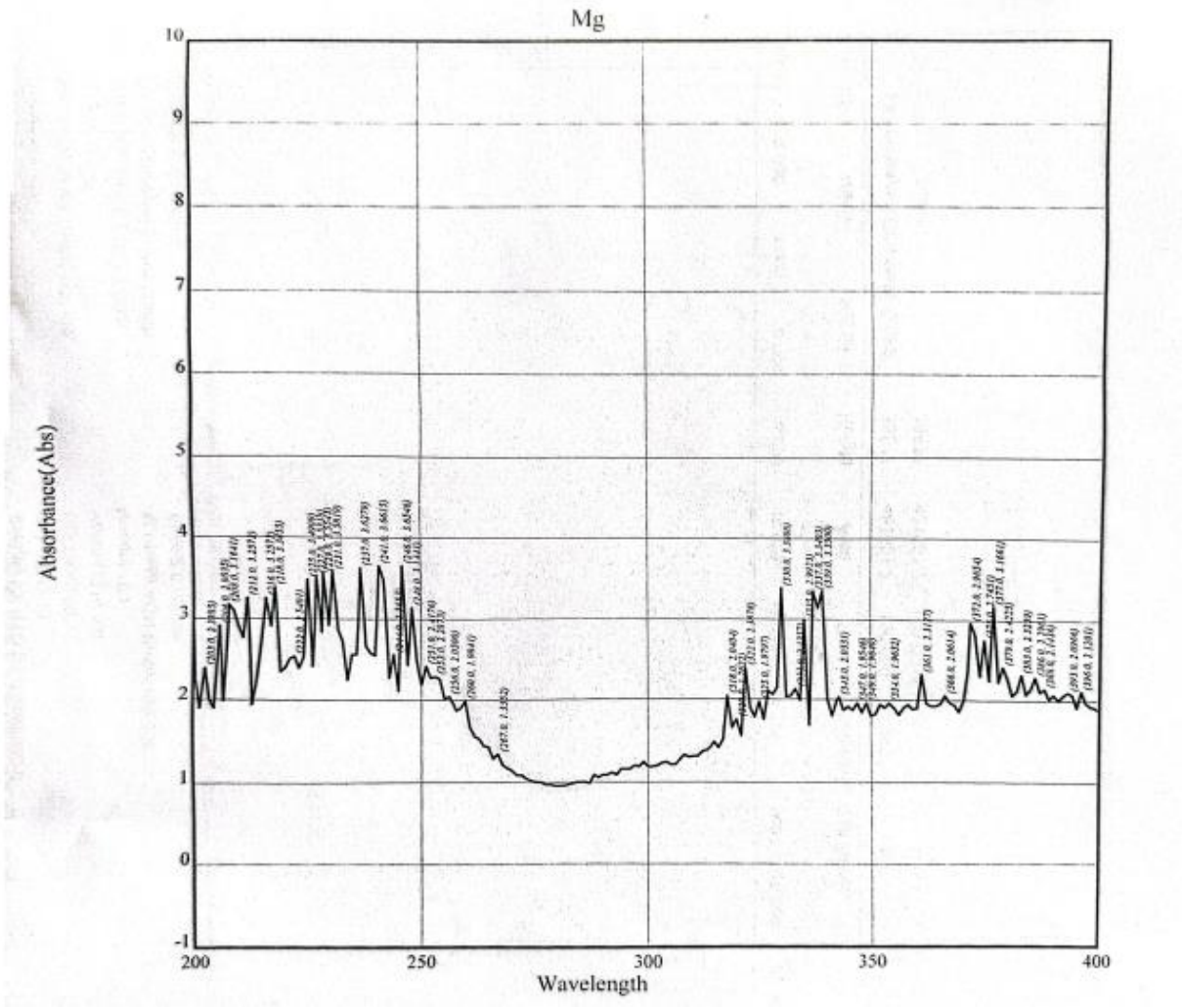

Fig.4.B. Levofloxacin Hemihydrate with magnesium picrate complex

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- **Nimesulide:** An NSAID with anti-inflammatory, analgesic, and antipyretic properties.

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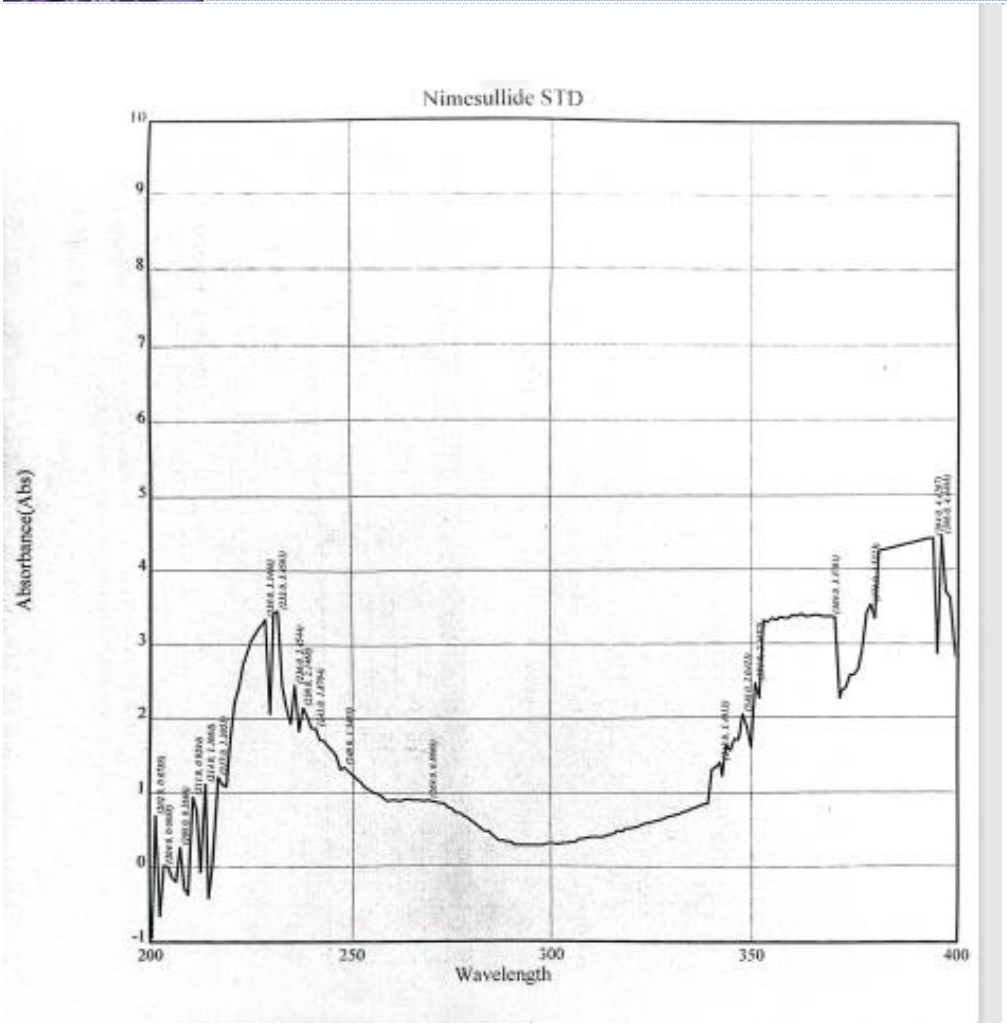

Fig.5.A. Nimesulide without magnesium picrate complex

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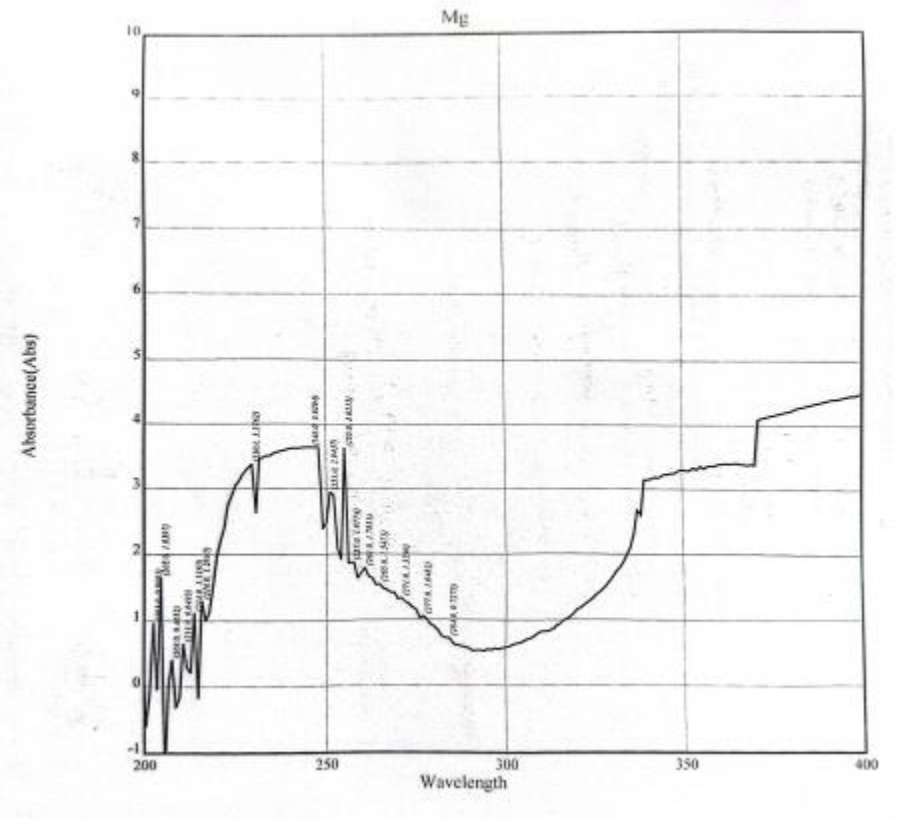

Fig.5.B. Nimesulide with magnesium picrate complex

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- **Piroxicam BCD:** A cyclodextrin-based formulation of piroxicam to enhance solubility and thus bioavailability.

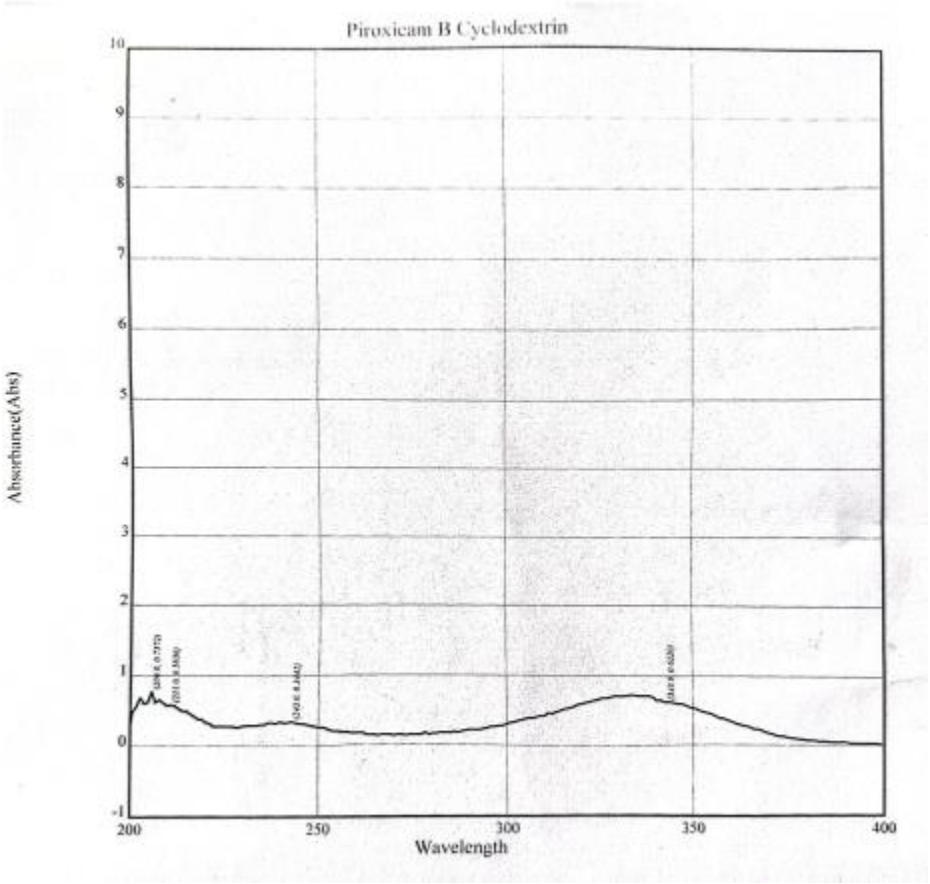

Fig.6.A.Piroxicam BCD without magnesium picrate complex Piroxicam BCD

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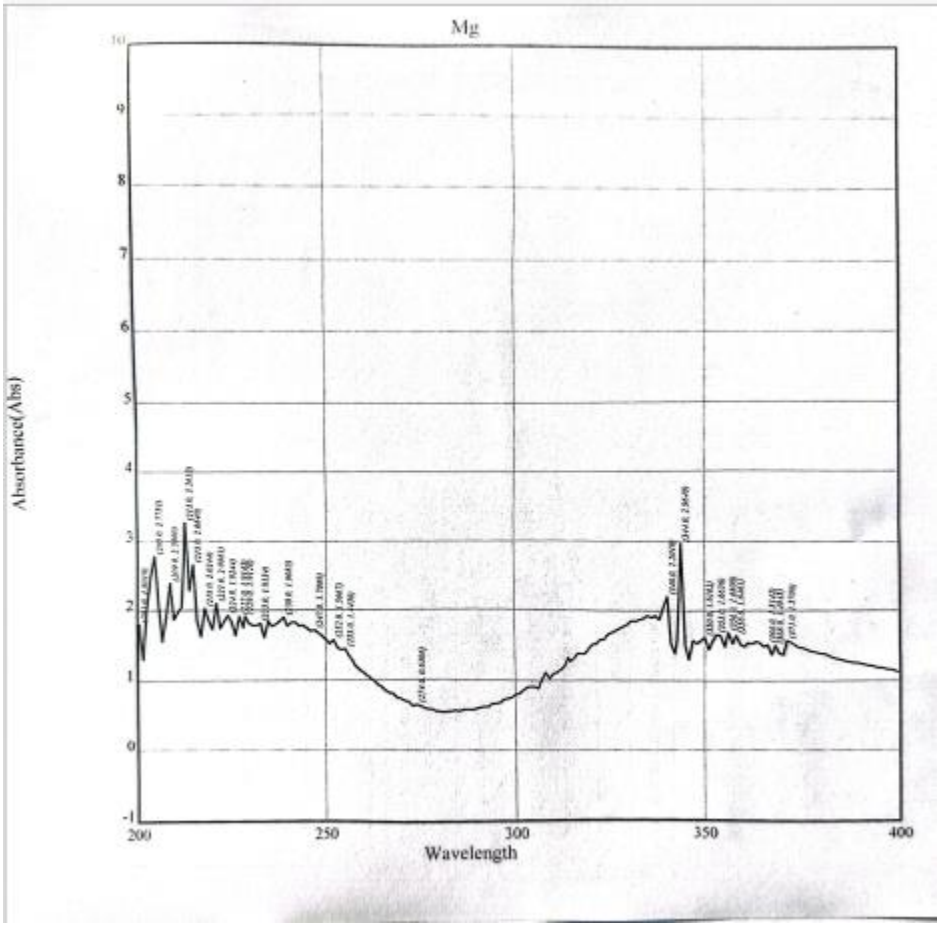

Fig.6.B.Piroxicam BCD with magnesium picrate complex Piroxicam BCD

- **Terbinafine HCl:** An antifungal that is mainly used in dermal infections.

  These stock solutions thus served as a platform for interaction studies from the magnesium-picrate complexes

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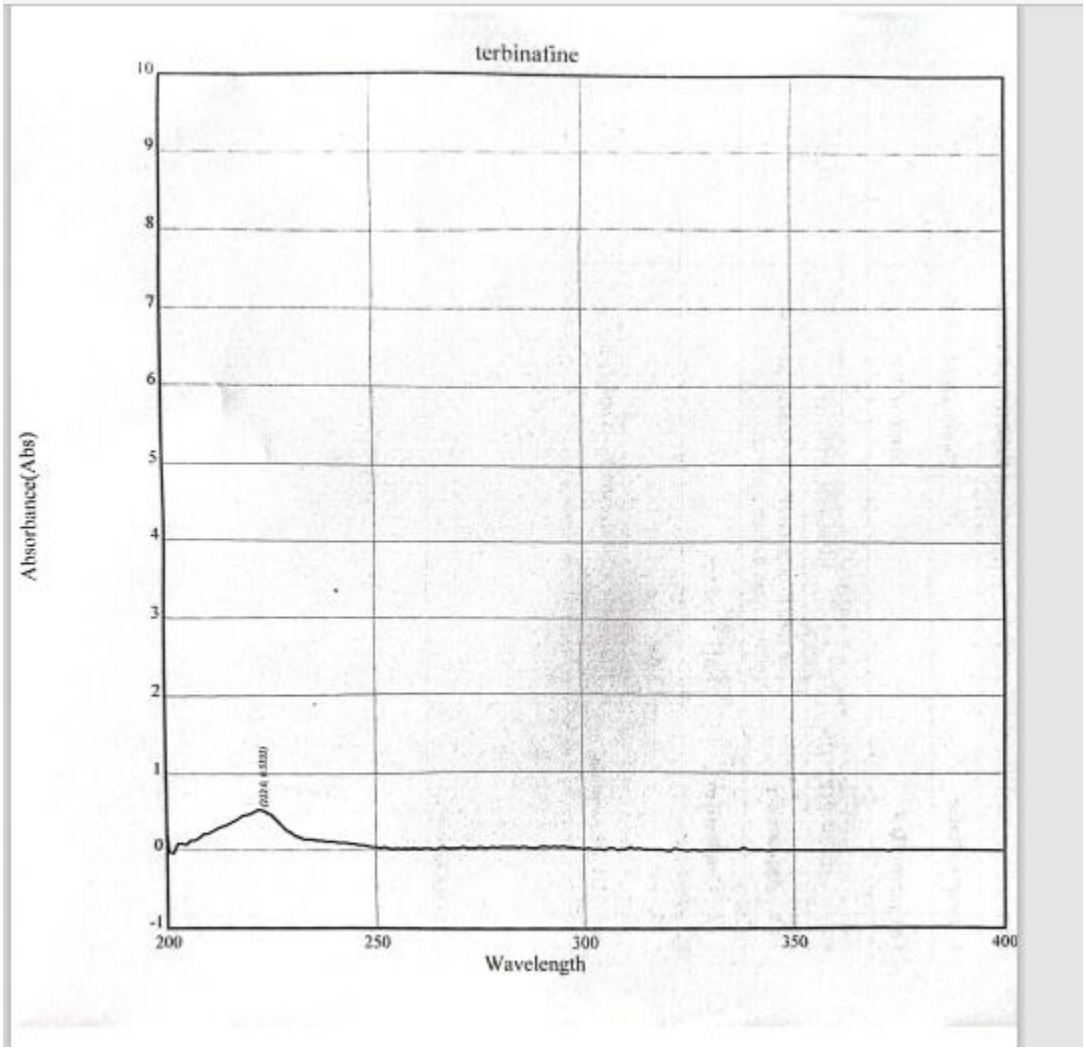

Fig.6.B. Terbinafine HCl with magnesium picrate complex.

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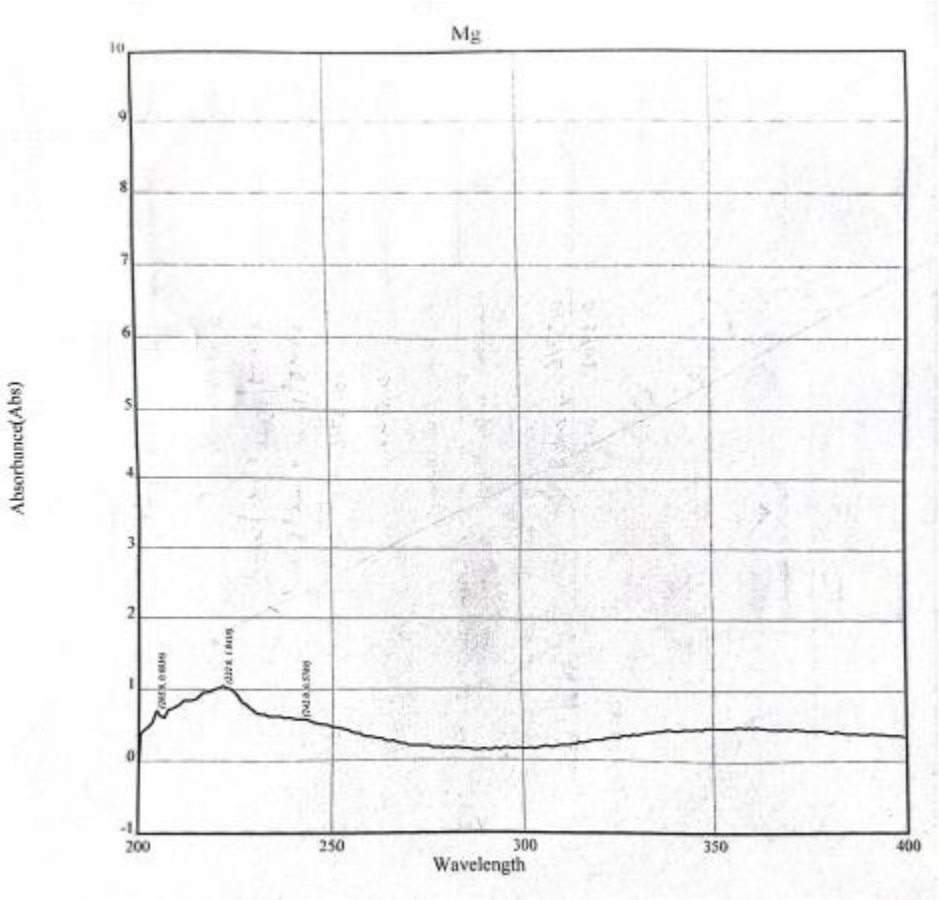

Fig.6.B. Terbinafine HCl with magnesium picrate complex.

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2.4. Modified UV-Visible Spectrophotometric Method:

A modified UV-Visible spectrophotometric technique was used in the investigation of the interaction between the drugs and magnesium-picrate complexes. It is a good technique in probing the changes in absorbance due to the new complex formed or the formation of any new complexes interacting with drugs and magnesium-picrate complexes.

2.4.1. Procedure:

2.4.1.1. Mixing: Mixtures of each drug solution and the magnesium-picrate complex at a 1:1 molar ratio were prepared. In choosing the 1:1 molar ratio, equimolar interaction is ensured, thus guaranteeing clear observation of either binding or complexation events.

2.4.1.2. Heating: The mixtures were heated to 70°C for 30 minutes to facilitate and accelerate the interaction between the drug molecules and the magnesium-picrate complexes. This temperature ensures enough energy for complex formation without degrading the components.

2.4.1.3. Absorbance Determination: The absorbance of the final solutions was determined using a UV-Visible spectrophotometer at the exact wavelengths of each drug. Since the spectrophotometer determines the minute changes in the absorbance that take place as a result of new bond formation or a change in the electronic environment, that is, an interaction did occur between the drugs and the magnesium-picrate complexes.

2.4.1.4. Control Experiments: For attaining a comparative baseline, results were confirmed through control experiments. Thus, the very same conditions of measuring drug absorbance without magnesium-picrate complex added into drug solutions continued in control experiments.

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The values of absorbance are compared between the drug solution before and after interaction with magnesium-picrate complexes. This suggests the amount of interaction or formation of complexes. Significant shift or changes in absorption represent proper binding, while a little or no change may imply slight to no interaction. These control experiments exclude false positives and confirm the specific interaction, providing a complete explanation for how magnesium picrate-based complexes affect spectral properties across many drugs.

2.5. Result and discussion:

2.5.1. Synthesis and Characterization of Magnesium Picrate Complex:

The synthesis of magnesium picrate complex is a simple and efficient method, yielding approximately 90% of the desired product. These high yields demonstrate the effectiveness of the reaction conditions, including proper temperature control, use of appropriate solvents, and optimal reaction times. This efficiency is important in coordination chemistry, where the reaction products often determine the ability to scale up the synthesis for industrial applications. The reaction procedure involves dissolving 1 mmol $MgCl_2 \cdot 6H_2O$ in 50 mL deionized water and adding 1 mmol picric acid dissolved in 50 mL ethanol with continuous stirring. The mixture was heated to 70°C for 3 hours, the key parameter promoting the formation of stable magnesium complexes. The aqueous phase was filtered, washed with ethanol to remove impurities, and dried at 60°C for 24 hours to obtain a pure and stable crystalline product.

2.6. UV-Visible Absorbance Enhancement:

Significant increases in absorbance were observed for all drugs upon treatment with magnesium-picrate complexes. The results are summarized in the table below. The results are summarized in the table below:

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Drug	Wavelength (nm)	Untreated Absorbance	% Increase	Enhanced Absorbance
Diclofenac Sodium	276	0.3396	70.82%	0.5802
Ciprofloxacin HCl	278	0.6339	47.95%	0.9374
Levoceitrizine 2HCl	236	0.7337	64.11%	1.2045
Levofloxacin Hemihydrate	298	0.4428	64.05%	0.7263
Nimesulide	295	0.3325	39.24%	0.4628
Piroxicam BCD	242	0.3442	81.16%	0.6232
Terbinafine HCl	237	0.1352	87.10%	0.2529

2.7. Analysis of Result:

The analysis of interactions between various drugs and the magnesium picrate complex using UV-Vis spectrophotometry revealed significant findings. These results indicate that the coupling agent not only enhances the absorption of the drug but also improves the sensitivity and accuracy of spectrophotometric drug measurement. This enhancement suggests that magnesium-picrate complexes could play a crucial role in the development of more effective analytical methods for drug testing and quality control in pharmaceutical formulations. [7]

2.7.1. Molar Absorbance Increase:

One of the most striking results of this study was the increase in absorbance, which is related to the increase in molar absorbance. The molar absorbance, or molar absorptivity, is a measure of the ability of a substance to absorb light at a particular wavelength. The combination of the drug and the magnesium picrate complex appears to enhance these properties, increasing the absorption values. The presence of magnesium picrate complexes can change the electronic environment of the drug molecules, making them more suitable for absorbing light in the UV-visible spectrum. The increase in molar mass significantly increases drug detection and quantification

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limits, allowing for more accurate identification of low drug concentrations. In practice, this means that even if only a small amount of drug is detected, this method can be very useful for drug quality control and analytical testing. [8]

2.7.2. Drug specific trend:

The absorption rate varied for the various drugs tested, indicating that the interaction between the drug and the magnesium picrate complex is influenced by the chemical structure and functional groups. Terbinafine HCl:

This drug showed an increase in absorption of 87.10%, indicating a strong interaction with the magnesium picrate complex. This may be due to the presence of functional groups in terbinafine hydrochloride, which can promote effective adsorption through hydrogen bonding or coordination with magnesium ions.

Nimesulide: on the other hand, showed the lowest increase in absorption of 39.24%. This relatively low interaction may stem from its structural features, such as steering inhibition or the lack of suitable binding sites, which limit its ability to form strong bonds with magnesium picrate complexes. Other drugs, such as diclofenac sodium, ciprofloxacin HCl, levofloxacin hemihydrate, and piroxicam BCD, showed a slight increase in uptake, indicating a wide range of interactions based on their molecular structure and functional groups. This trend emphasizes the importance of molecular structure in determining the strength of interactions with complexes.

2.8. Effect on UV-visible spectrum:

The inclusion of magnesium picrate complex as a coupling agent significantly influences the UV-visible spectral behavior of the drug solution. The coupling agent shifts the reaction equilibrium and increases the total light absorption of the drug molecule. This shift leads to increased sensitivity in spectrophotometric readings, making it easier to detect and measure small changes in concentration. Therefore, this method provides greater accuracy and reliability in drug measurement. This is particularly useful for pharmaceutical analysis, where accurate measurement of drug concentration is essential for determining the correct dose and potency of the drug. In

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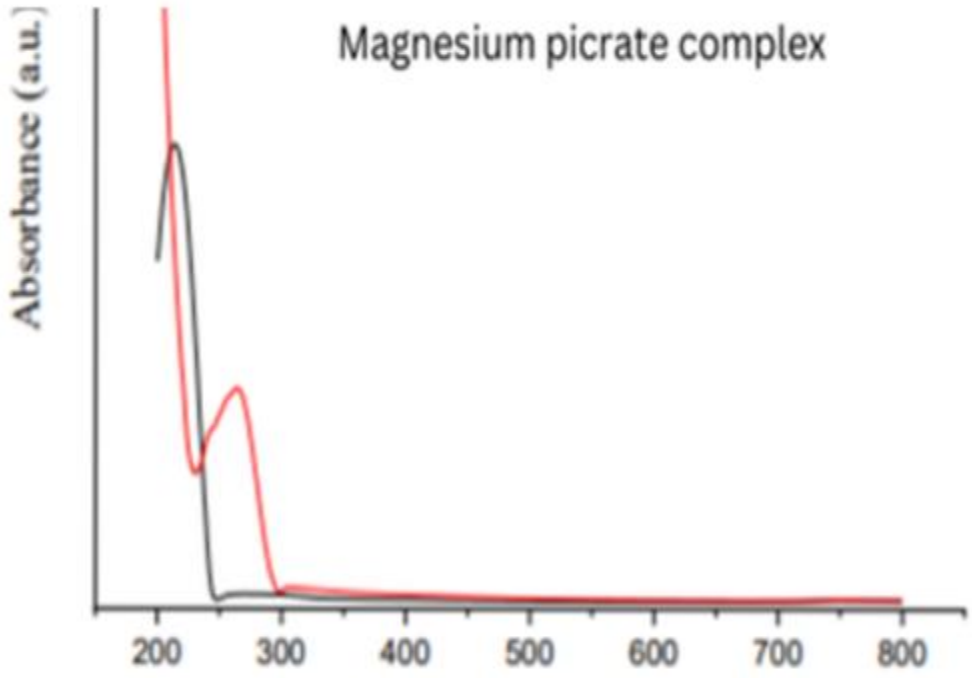
addition, increasing the absorption helps to produce more accurate calibration curves, reduces measurement errors, and improves the reproducibility of multiple samples. [9]



2.9. Conclusion:

The synthesis of magnesium-picrate complexes and their application as coupling agents significantly enhance the UV-Visible spectrophotometric analysis of key pharmaceutical drugs. The observed increases in absorbance improve detection and quantification limits, making the technique more sensitive and effective for quality assurance. The high yield of the synthesis process and the successful FT-IR characterization further validate the practicality of these complexes. Future work should focus on expanding the range of analyte and exploring the underlying mechanisms of interaction.

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