



SYNTHESIS AND CHARACTERISATION OF SCHIFF BASE CO (II) AND CU (II) COMPLEXES DERIVED FROM CIPROFLOXACIN AND P-NITROANILINE

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ABSTRACT

The complexes of Co(II) and Cu(II) with Schiff base derived from ciprofloxacin and p-nitroaniline have been synthesized. The ligand and the complexes were characterized on the basis of physical properties conductance measurement and infrared spectroscopic analysis. The ligand melting point and decomposition temperatures for the complexes are high. The molar conductance values of the complexes are low. The I.R data of the complexes showed absorption bands ranging from (500–800) cm^{-1} indicating coordination of the ligand to the metal ions.

Keywords: Schiff base, Ciprofloxacin, P-nitroaniline, Molar conductance, Tetrahedral stereochemistry, Coordination.

Contribution/ Originality

The paper's primary contribution is finding that ciprofloxacin and p-nitroaniline can be used to synthesize a new Schiff base and its metal complexes by, characterizing the complexes according to their physical properties, conductance measurement and infrared spectroscopy. The results of which were found to be in conformity to the literature.

1. INTRODUCTION

Schiff bases are typically formed by the condensation of a primary amine and a ketone or an aldehyde [1]. The resultant functional group $\text{R}'\text{C}=\text{NR}''$ is called an imine and it is used particularly for binding metal ions via the N-atom lone pair especially when in combination with one or more donor atom to form polydentate chelating ligand [2-5].

Conventionally, Schiff bases have been prepared by refluxing mixtures of the amine and the carbonyl compound in an organic solvent, for example ethanol and methanol [6], but variations are known such as treatment of the same mixture at room temperature, refluxing the mixture in heptanes in the presence of acetic acid [7] or azeotroping the mixture with benzene in a Dean

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Starkapparatus in the presence of an acid [8]. Schiff bases belong to a widely used group of organic intermediates important for production of special chemicals such as pharmaceuticals and rubber additives and as amino protective groups in organic synthesis [9]. They also have uses as liquid crystal and in analytical, medicinal and polymer chemistry [10]. Metal complexes of Schiff bases have been extensively studied due to synthetic flexibility, selectivity and sensitivity towards a variety of metal atoms [11]. They are found useful in catalysis, in medicine as antibiotics and anti-inflammatory agents and in industry as anti-corrosion agents [12]. There is paucity of information on Schiff base derived from ciprofloxacin and its metal complexes. In light of the foregoing, this paper reports the synthesis and characterization of a new complex compound formed by Co(II) and Cu(II) ions with Schiff base derived from ciprofloxacin and p-nitroaniline.

2. MATERIALS AND METHODS

The Solvents and chemicals used in the work were of analar grade. All glass wares used were washed thoroughly with distilled water. Ciprofloxacin was obtained from Gemini Pharmaceuticals Limited, Nigeria and was used without further purification. Weighing were carried out using metler balance model AB54, melting and decomposition temperatures were determined on Gallekamp melting point apparatus. Electrical conductivity measurements were carried out using conductivity meter model 4010. While the I.R spectral analysis were recorded using FTIR NICOLET 100 at the range of 4000 cm^{-1} - 400 cm^{-1} .

2.1. Synthesis of Ciprofloxacin-Imine

A mixture of methanolic solution of ciprofloxacin (3.313(g), 0.01mol) and p-nitroaniline (1.381(g), 0.01mol) was refluxed in the presence of glacial acetic acid for 4hrs. The resulting solution was concentrated to 8 cm^3 in a water bath and allowed to cool at 0°C in an ice bath. A yellow solid was filtered, washed with methanol and ethanol, then dried at room temperature.

2.2. Synthesis of the Complex

The Schiff base (9.02(g), 0.02 mol) dissolved in methanol (25 cm^3) was mixed with respective transition metal (II) chloride (0.01mol) in methanol (20 cm^3). The reaction mixture was each refluxed for 2-3 hrs. Each of the resulting solution was concentrated in a water bath, cooled in an ice bath, filtered and washed with methanol. The precipitate was further recrystallized with methanol and dried in a desiccator over P_2O_{10} .

3. RESULTS

Table-1. Physical Properties of the Schiff Base and the Complexes

Serial	Compounds	Molecular Weight(g)	Colour	% Yield	M.P (°C)	Decomposition Temperature (°C)
1.	Ligand	451.43	Yellow	71.06	190 ^o	-
2.	[Co(cip-imine) ₂]	989.41	Yellowish-Green	25.00	-	240
3.	[Cu(cip-imine) ₂]	969.41	Green	22.90	-	270

Table-2. Conductivity Measurement of the Compounds

Serial	Compound	Concentration (mol dm ⁻³)	Electrical Conductivity ohm ⁻¹ cm ⁻¹	Molar Conductivity Ohm ⁻¹ cm ² mol ⁻¹
1.	[Co(cip-imine) ₂]	1.0x10 ⁻³	9.03x10 ⁻⁵	90.3
2.	[Cu(cip-imine) ₂]	1.0x10 ⁻³	7.50x10 ⁻⁵	75.0

Table-3. Percentage Composition of Each Metal Present

Serial	Compound	Percentage of metal calculated	Percentage of Metal observed	Percentage of Ligand
1.	[Co(cip-imine) ₂]	6.14	5.83	94.17
2.	[Cu(cip-imine) ₂]	6.58	6.11	93.89

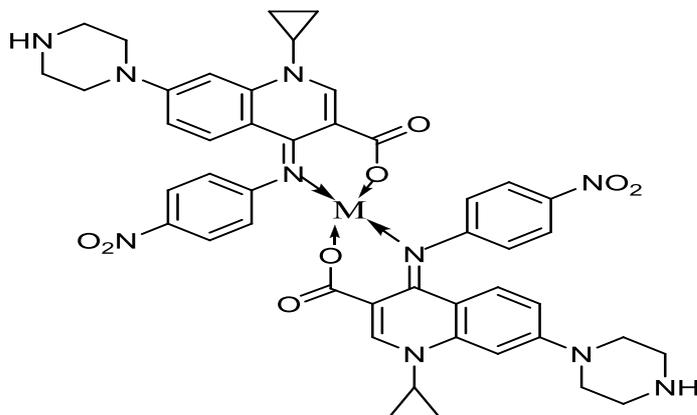
Table-4. IR Data of the Compounds

Serial	Compound	V(C=N)	V(M-O)	V(M-N)
1.	Ligand	1588	-	-
2.	[Co(cip-imine) ₂]	1583	540	709.16
3.	[Cu(cip-imine) ₂]	1596.25	548.95	750

3.1. Discussion of Results

The interaction between ciprofloxacin (1-cyclopropyl-6-fluoro-1,4-dihydro-4-oxo(7-1-piperazinyl-3) quinolone carboxylic acid and p-nitro aniline gave a yellow solid powder. The physical properties of the Schiff base and its complexes were summarized in Table 1. The molar conductance values of the complexes determined (Table 2) are quite low, suggesting the complexes are non electrolytic in nature [13]. The Percentages of metal observed and the Schiff base in each complex showed 1:2 metal to ligand ratio for the complexes establishing the general molecular formula [ML₂] (Table 3). The I.R spectral data of the ligand and the complexes showed bands in the region (1583-1596.25) cm⁻¹ which falls within the range (1560-1600)cm⁻¹ assigned to V(C=N) group stretching vibrational mode by Boucher and Day [14]. The absorption bands in Co(II) and Cu(II) complexes at 540cm⁻¹ and 548.95cm⁻¹ due to V(M-O) and 709.16cm⁻¹ and 750cm⁻¹ due to V(M-N) falls within the ranges (500-700) cm⁻¹ and (600-800)cm⁻¹ assigned to V(M-O) and V(M-N) stretching vibrations respectively by Abdulsalamath, et al. [15]. These bands confirmed the coordination of the ligand to the respective metal ion as shown in Table 4.

The correlation of the experimental data allows assigning a tetrahedral stereochemistry to the complexes. The proposed structural representation of the complexes is presented below:



Ciprofloxacin imine metal (II) complex

4. CONCLUSION

The Schiff base ligand and its metal (II) complexes were prepared and characterized by melting point/decomposition temperature, molar conductance measurement and I.R spectroscopy. The ligand melting point and decomposition complexes are high. The molar conductance of the complexes are low revealing their non-electrolytic nature, this is because of the incomplete dissociation of the complexes to produce ions that can conduct electricity. The I.R data of the complexes showed bands in the range of $(500-800) \text{ cm}^{-1}$ indicating coordination of the ligand to the metal ions.

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