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Preparation and Some Characterization Analyses of Cobalt (II) Complex with Schiff Base Derived from Acetyleacetone and 2-Aminopyridine

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Abstract:** A Schiff base has been prepared by condensation of acetylacetone and 2-aminopyridine. Metal complex of the Schiff base was prepared by the reaction of the Schiff base and cobalt(II) chloride in methanol. The complex was isolated, washed and dried. The Schiff base and the complex were yellow and purple colors respectively. They were characterized by solubility test, melting point and decomposition temperature, FT-IR., and molar conductivity analysis. Solubility test on the Schiff base and complex showed that they all soluble in DMF and DMSO but insoluble in CCl_4 and ether. Also FT-IR. analysis showed a band at 1674cm^{-1} assigned to azomethine of the Schiff base which shifted to 1644cm^{-1} in the complex. The complex is high spin complex and electrolytic in nature.

***Keywords:** 2-Aminopyridine, Acetylacetone, Cobalt (II) Chloride, Ligand, and Physicochemical Properties.*

1. INTRODUCTION

A substantial amount of bio-inorganic chemistry research has been conducted on the coordination chemistry of transition metal complexes, which has gained momentum in recent years due to their numerous applications in the chemical and medical sciences [1]. Chemical researchers have been motivated by it to create unique metal complexes all around the world. The topic of metal drug interaction chemistry is expanding quickly in the medical and chemical sciences because metal ions form the connection between drug compounds and pathogenic organisms [2]. Depending on the physical and chemical characteristics of the metal ions, microbial interactions with a range of metal ions at different oxidation states can occasionally be advantageous or deleterious [3]. Since the beginning of time, people have sought out natural herbal remedies from nature to alleviate their illnesses. Due to the difficulty of their chemical extraction and their gradual effects on diseases, the usage of pure

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herbal remedies has significantly decreased recently [4]. Because of the discovery of penicillin, pharmaceutical research has increased since the Second World War to include an extensive screening of microbes for novel antibiotics. The main focus of pharmaceutical discovery activities would be the identification of novel metabolites from live organisms given the success of synthetic medicinal chemistry. Today's therapeutic pharmaceutical research goal is the discovery of synthetic medications because the world is running out of antibiotics. However, there are other natural product-based medications in clinical use that still require a lot of research [5]. Hugo Schiff, who has been referred to as one of the inventors of modern chemistry, is best known for his discovery of Schiff base. According to Professor Wohler's research, there is no metaphysical distinction between organic and inorganic substances, ruling out the vis-vitalis idea. Hugo Schiff, Berzelius Wohler, and their tremendous pioneering work altered how people saw organic chemicals. Remember that you are descended from Berzelius since Berzelius taught chemistry to the old Wohler and the old Wohler taught me, according to Professor Schiff, who has committed the phrase to memory. German-born Professor Schiff has been doing research in Italy under the name of an Italian chemist [6]. Following Schiff's groundbreaking breakthrough in 1864, other researchers engaged in this field of study and achieved progress in the synthesis and structural development of this class of chemicals. Cisplatin, which was accidentally discovered to be an effective anticancer drug in the late 19th century, was the first metal-based medication to appear. This finding opened the door for further research into metal-based chemotherapeutic agents. According to [7] and [8], it was the most potent anticancer medication on the market. The hunt for alternative metal-based chemotherapeutic drugs has received a great deal of attention in recent years as a result of the spectacular therapeutic success of cisplatin and its analogs. Since then, coordination chemistry research on metal-drug interactions has been well-focused and is regarded as an active area of study [9].Therefore, it is crucial to find and define novel medications that are more active, selective, bioavailable, and have less side effects than current treatments.

2. MATERIALS AND METHOD

Materials

All glass wares used were thoroughly washed with detergent, soaked in concentrated nitric acid, rinsed with distilled water and dried in an oven at 110^{o}C. The reagent used in this work, were of analytical grade and used without further purification. They include acetylacetone, 2-aminopyridine, methanol, cobalt (II) chloride salt, ether, DMF, DMSO, CCl_4, chloroform acetone and ethanol. Also all weighing were carried out on electrical weighing balance model AB54. IR spectral analysis was recorded on Fourier transform spectrophotometer model IR Genesis series using pallet. Molar conductance of the metal complexes was determined in DMSO using a coronation digital conductivity meter, melting point was determined on scientific melting point apparatus.

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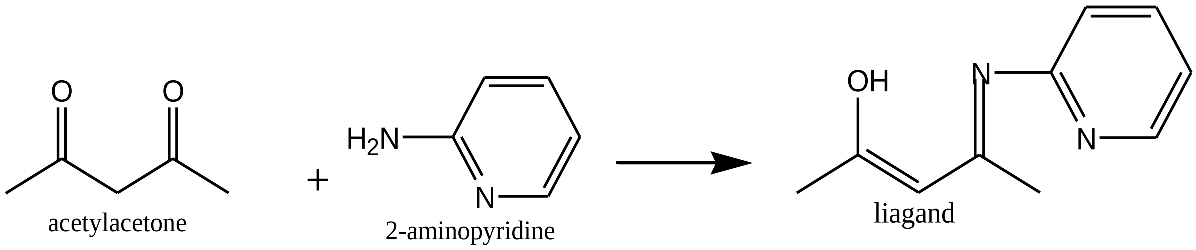
Method

Preparation of Schiff Base (Ligand)

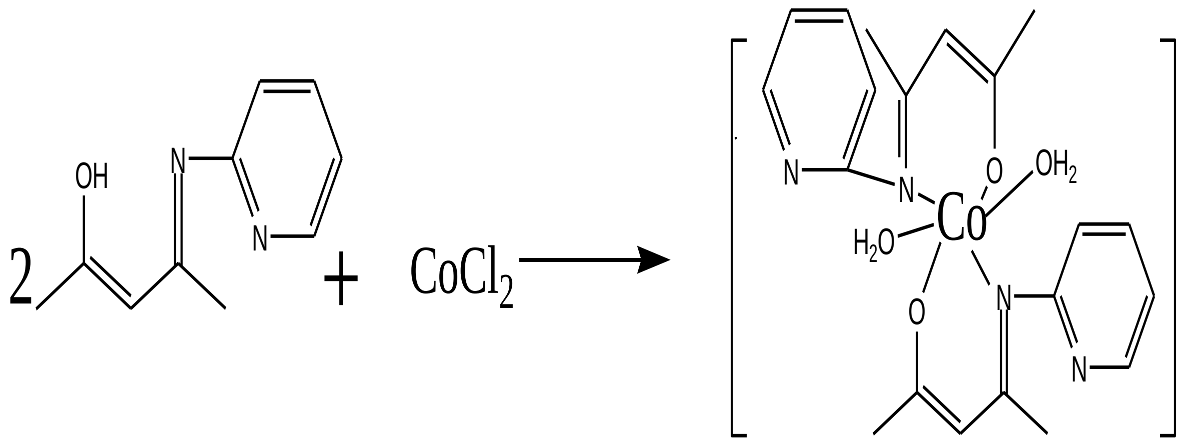
The Schiff base (ligand) was prepared using literature procedure [10] by equimolar ratio (0.05mol) of 2-aminopyridine (4.7055g) and (0.05mol) acetylacetone (5.2ml) in 20ml alcoholic solution each, in a round bottom flask. The mixture was subjected to reflux for 3 hours with constant stirring after which the resulting solution was concentrated on water bath and allowed to cool at room temperature. The pale yellow solid was filtered and dried. The ligand was stored in the airtight container till its further use.



Scheme 1: Preparation of Schiff base (ligand).

Preparation of Cobalt (II) Complex

The complex was prepared by dissolving (1.18g) of $CoCl_2$ salt in 20ml methanol and (1.76g) of Schiff base in 20ml methanol in a round bottom flask. The mixture was then refluxed for 3 hours at 70°C with constant stirring after which the resulting solution was concentrated on water bath and allowed to cool at room temperature. From then, the purple colored crystal was filtered and dried in a desiccator after which was stored in an airtight vial till it further use.



Scheme 2: preparation of cobalt (II) complex

Determination of Melting Point and Decomposition Temperature

The melting point and the decomposition temperature were determined by taking small amount of the Schiff base and the complex respectively in a capillary tubes, and then the tubes were inserted into a gallenkamp apparatus where the temperature at which were melt and decompose were recorded.

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Solubility Test

The solubility test of both the ligand and the complex were carried out in the following solvent; water, ethanol, methanol, chloroform, tetrachloromethane, ether, DMF, and DMSO. The test was successful by taking little amount of the ligand and the complex into a test tube of 5ml solvent each.

3. RESULTS AND DISCUSSION

2-aminopyridine and acetylacetone were refluxed to give a yellow crystalline Schiff base with percentage yield and melting point of 53.47% and 54^{0}C respectively. The Schiff base reacts with cobalt(II)chloride in methanolic solution to form a complex with purple color. The color of the complex was due to transition between lower t_{2g} and e_g orbital by absorption of visible light [11].The molar conductance of the complex was measured in DMSO at concentration of 0.001M, and the observed conductance value was measured to be 100Scm^{-2}mol^{-1} as shown in the table below.

Table 1: Physicochemical Properties of the Schiff Base and the Complex

Compound	**Color**	**% Yield**	**D. tp. / M. Pt ^{0}C**	**K (µScm^{-1})**	**Λ_m (Scm^{-2}mol^{-1})**
Ligand	Yellow	53.47	54	-	-
Complex	Purple	86.147	70	110	100

Table 2: Solubility Tests of Schiff Base and Complex

Compound:	**Water**	**CHCL$_4$**	**Ethano**	**Acetone**	**CCl$_4$**	**DMF**	**DMSO**	**Ether**	**CH$_3$OH**
Ligand	S	SS	S	IS	S	S		IS	S
Complex	S	SS	S	IS	S	S		IS	S

Keys: IS = Insoluble, S = Soluble SS = Slightly Soluble

Table 2 above shows the solubility test carried out on the ligand and the complex in which the ligand was found to be insoluble in carbon tetrachloride and petroleum ether and slightly soluble in chloroform while soluble in water, methanol, ethanol, acetone, DMF and DMSO, therefore methanol was used as solvent for the synthesis of the complex. Similarly, the

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complex was found to be insoluble in carbontetrachloride, petroleum ether and slightly soluble in chloroform while soluble in water, methanol, ethanol, acetone, DMF and DMSO.

Table 3: Infrared Analyses of Ligand And Complex

Compound	**(OH)(cm^{-1})**	**(C=N)(cm^{-1})**	**(Co-O)(cm^{-1})**
Ligand	3305	1674	—
Complex	—	1644	718

The infrared spectra provide information regarding the nature of the functional group attached to the compound. The ligand and metal complex were characterized mainly using azomethine with absorption band *v*(1674cm^{-1}) in the ligand which was shifted to *v*(1644cm^{-1}) in the complex due to coordination with the metal complex and thus this correspond to what [12] reported for (C=N) vibrations. Also the absorption band *v*(3305cm^{-1}) due to OH stretch in the ligand was found been shifted to *v*(3309) in the complex, confirming the coordination of ligand to metal complex shown above at table 3.

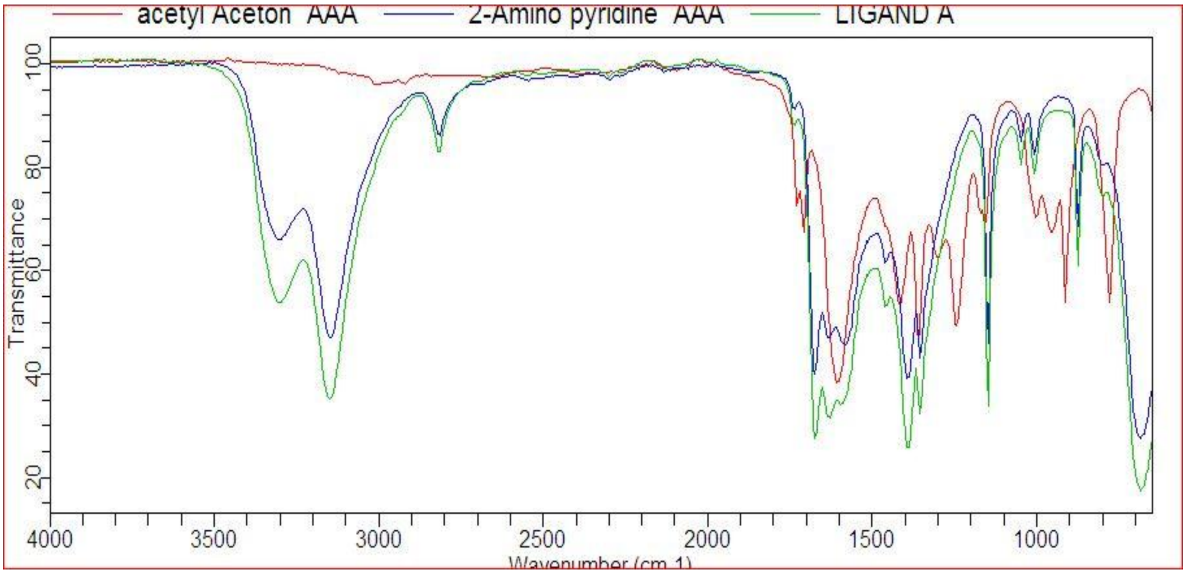

Fig. 1: Superimposed structures of ligand with starting materials

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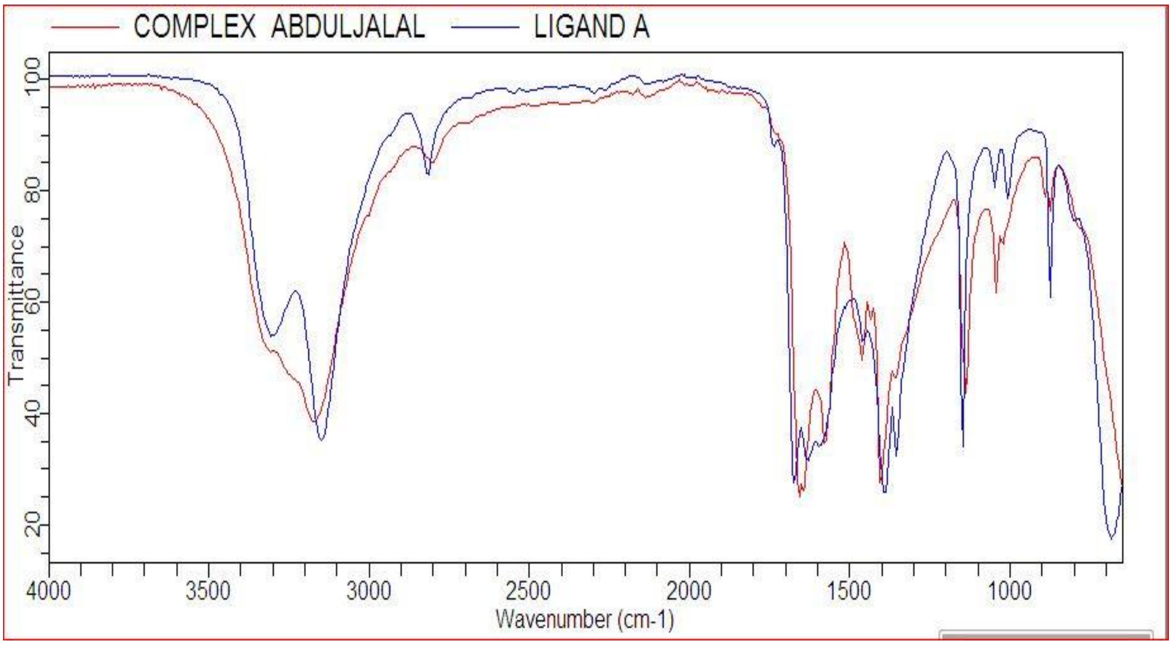

Fig. 1: Superimpose structure of ligand with cobalt(II) complex

From the above analyses, the proposed structures are

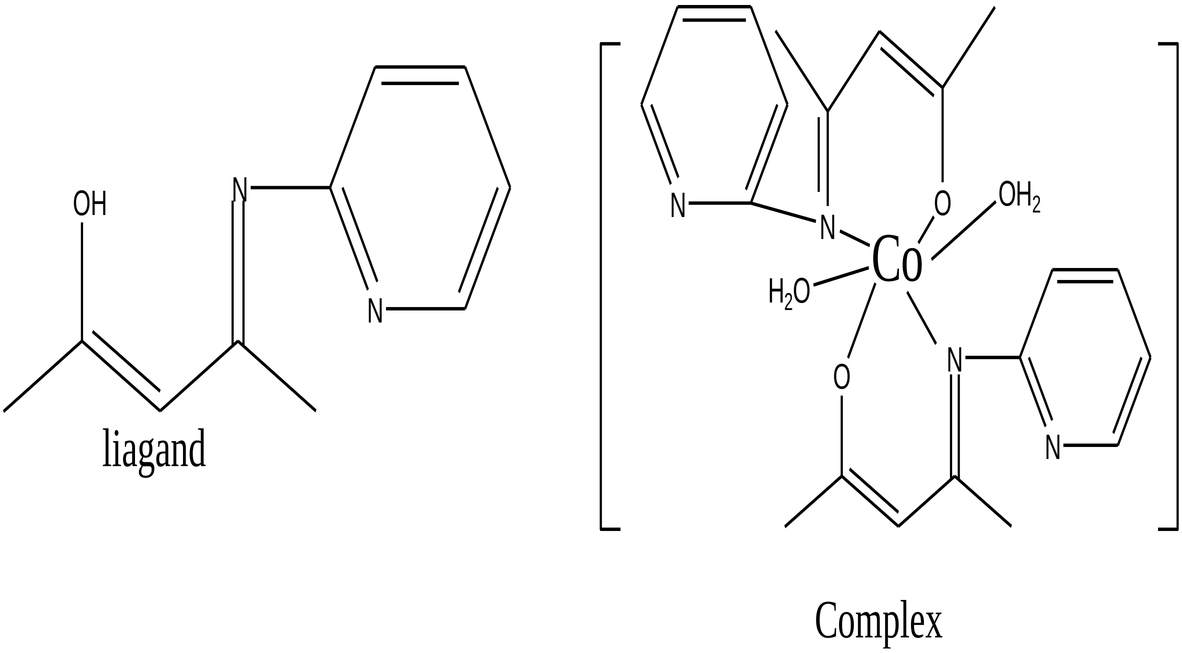

Fig. 2: Proposed Chemical Structure of Ligand and Complex

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4. CONCLUSION

A Schiff base derived from 2-aminopyridine and acetylacetone were prepared and analysed with cobalt (II) to form a Schiff base complex. The ligand and the complex were analysed based on solubility, FT-IR., conductivity, melting point and decomposition temperature. The complex was found to be soluble in DMSO and DMF while insoluble in distil water.

5. REFERENCES

1. Glaser, F., and O. S. Wenger, (2020).Recent progress in the development of transition-metal based photoredoxcatalysts, Coord. Chem Rev. 405:213-129. http://dx.doi.org/10.1016/j.ccr.2019.213129
2. Yang,Y., X. Wu, C. He, J. Huang, S. Yin, M. Zhou, L. Ma, W. Zhao, L. Qiu, C. Cheng, C. Zhao, (2020).Metalorganic framework/Ag-based hybrid nanoagents for rapid and synergistic bacterial eradication, ACSAppl. Mater. Interfaces. 12, 13698–13708.
3. Dias, D., A.,S. Urban, and U., Roessner, (2012). A historical overview of natural products in drug discovery, Metabolites. 2: 303–336. https://doi.org/10.3390/metabo2020303
4. Abdallah, S., M., G., G. Mohamed, M., A., Zayed, M., S. and Abou El-Ela, (2009). Spectroscopic study of molecular structures of novel Schiff base derived from ophthaldehyde and 2-aminophenol and its coordination compounds together with their biological activity Spectrochim. Acta. A. Mol. Biomol. Spectrosc. 73: 833–840.
5. Bérdy, J., (2012). Thoughts and facts about antibiotics: where we are now and where we are heading, J. Antibiot. 65, 385–395. https://doi.org/10.1038/ja.2012.27.
6. Qin, W., S. Long, M. Panunzio, S. Biondi, (2013). Schiff bases: a short survey on an evergreen chemistry tool, Molecules. 18, 12264–12289. https://doi.org/10.3390/molecules181012264.
7. Rani, Ai, M. Kumar, R. Khare, H.S. Tuli, (2015). Schiff bases as an antimicrobial agent: A review, J. Biol. Chem. Sci. 2, 62–91.
8. Vederas,J.W.-H. Li, J.C., (2009). Drug discovery and natural products: end of an era or an endless frontia, J. of Science. 325, 161–165. https://doi.org/10.1126/science.1168243 Romero-CaneloI,N..J.Sadler,(2013).Next-generationmetal anticancer complexes: multitargeting via redox modulation, inorg. chem. 52, 12276- 12291. https://doi.org/10.1021/ic400835n
9. Gibson, D., (2016). Platinum(IV) anticancer prodrugs -hypotheses and facts, Dalt. Trans.45:12983-12991. https://doi.org/10.1039/C6DT01414C.
10. Chakraborty, A., P., Kumar, K. Ghosh, and P., Roy, (2010). Evaluation of a Schiff base copper complex compound as potent anticancer molecule with multiple targets of action, Eur. J. of Pharmacol.,647 1-12 https://doi.org/10.1016/j.ejphar.2010.08.003.
11. Habila, A. N., Putaya, Naomi, P., Ndahi, Hauwa, S. Bello, Grema, Mala, Adekunle, A., Osanlaja and Hussaini Garba, (2020). Synthesis, characterization and antimicrobial analysis of Schiff bases of o-phenylenediamine and 2-aminopyridine-3-carboxylic acid with ofloxacin and their metal(II)complexes Int.J.Biol.Chem.Sci.,14(1):263-278. https://dx.doi.org/10.4314/ijbcs.v14i1.22

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12. Sulaiman, Z., Na'aliya, J. and Umar, A. A., (2019). solid state synthesis, characterization and antimicrobial of Co(II) and Ni(II) metal complexes with schiff base derived from L-Valine and Vanilin ;**.** Bayero Journal of Pure and Applied Sciences, 12(1): 638-6350 http://dx.doi.org/10.4314/bajopas.v12i1.96S.