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SYNTHESIS AND STRUCTURAL ELUCIDATION OF A COBALT(II) COMPLEX DERIVED FROM 2,2′-((1E,1′E)-(ETHANE-1,2-DIYLBIS(AZANYLYLIDENE))BIS(3-PHENYLPROPAN-1-YL-1-YLIDENE))DIPHENOL AND ITS ANTIBACTERIAL AND ANTIFUNGAL ASSESSMENT

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ABSTRACT

Preparation of Metal Complex Of newly synthesized Schiff base that 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol using 2-hydrxy-3- phenylpropiophenone and ethylenediamine and then react with cobalt chloride hexahydrate salt to form unique metal complex were characterized by different physicochemical studies like FTIR, H1& C13 NMR,mass spectroscopy, P-XRD, determine the metal content by using ICP-OES. Examination of the antibacterial and antifungal properties of the metal complex of schiff base. They exhibit promising activity. The Successful Synthesis, structural characterization and notable biological activities of the compound underscore its pharmaceutical potential. Additional research is necessary to enhance its bioactivity and investigate its therapeutic applications.

KEYWORDS: Schiff base, metal complex, 2-hydroxy-phenylpropiophenone, Cobalt chloride hexahydrate, ethylenediamine, antibacterial activity, Antifungal anctivity.

1. INTRODUCTION

The discovery of Schiff bases dates back to 1864 when the German chemist Hugo Schiff first reported their formation through the condensation of primary amines with carbonyl compounds. These compounds, characterized by the azomethine (-C=N-) functional group, have since become a central focus in coordination chemistry due to their structural versatility and ability to act as effective ligands. Schiff bases readily coordinate with metal ions, especially when derived from substituted aromatic compounds containing functional groups such as hydroxyl (–OH) or thiol (–SH), which significantly enhance their chelating abilities.[1-6]

Among the various Schiff base ligands, those synthesized from 2-hydroxyaryl ketones and ethylenediamine have attracted considerable interest. The presence of both hydroxyl and imine groups allows for the formation of stable bidentate or tetradentate complexes with a wide range of metal ions.[7-11] These metal complexes often exhibit improved thermal and chemical stability, as well as enhanced biological activity compared to the uncoordinated Schiff base ligands.[12] The increased lipophilicity and improved membrane permeability of the metal chelates contribute to their superior biological performance.

Schiff base metal complexes have shown a broad spectrum of biological applications, including anti-inflammatory, analgesic, anticancer, antiviral, antifungal, pesticidal, bactericidal, insecticidal, herbicidal, and growth-regulating activities.[13-21] Beyond biological significance, Schiff bases also play important roles in materials science and industrial applications, such as in the manufacture of semiconductors, corrosion inhibitors, cross-linked polymers, antiglare mirrors, deodorants,

dental materials, and perfumes. Their ability to function as efficient catalysts under diverse conditions, including high temperatures and moisture, has made them valuable in organic transformations like carbonylation, hydroformylation, oxidation, epoxidation, and hydrolysis.

In recent decades, Schiff base complexes have gained increasing attention in the fields of medicinal and bioinorganic chemistry, especially for their anticancer, antifungal, antiviral, antitubercular, and herbicidal properties. Their strong chelating ability, structural tunability, and potential multifunctionality make them promising candidates for the development of new therapeutic agents and functional materials. This research focuses on the synthesis, characterization, and evaluation of metal complexes derived from Schiff bases of 2-hydroxyaryl ketones and ethylenediamine, with an emphasis on their antimicrobial activity against selected pathogenic strains.

2. EXPERIMENTAL

2.1. Materials

Make of 2-hydrxy-3- phenylpropiophenone is sigma Aldrich, ethylene diamine from SD fine, ethanol, AR Grade Cobalt chloride, DM water.

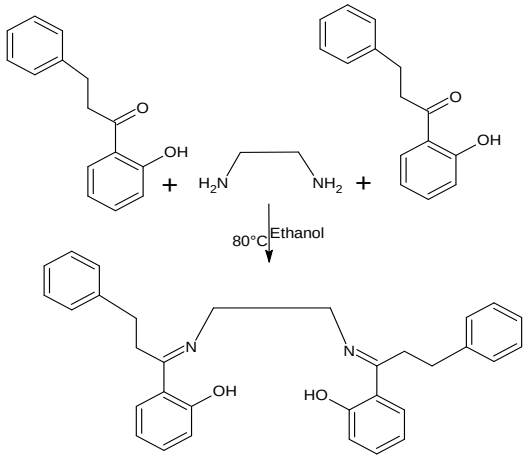
2.2. Physical Measurements

Fourier-transform infrared (FT-IR) spectra were recorded using a SHIMADZU FT-IR 8300 spectrophotometer in the range of 4000–200 cm^{-1} to identify characteristic vibrationalmodes of functional groups. Proton and carbon-13 nuclear magnetic resonance (^{1}H and ^{13}C NMR) spectra were acquired using a BRUKER FT-NMR spectrometer operating at 400 MHz and 75.45 MHz, respectively. Tetramethylsilane (TMS) was used as an internal standard, and spectra were recorded in DMSO-d_6. Elemental analysis (C, H, N, S) was performed using a Thermo Fisher Scientific CHNSAnalyzer (FLASH SH 1112 Series) to confirm the composition and purity of thecompounds. Electrospray ionization mass spectra (ESI-MS) were recorded on an Agilent LC-MS instrument to determine molecular masses and fragment patterns. Metal content was determined via inductively coupled plasma optical emission spectroscopy (ICP-OES) using standard protocols for elemental quantification.

Powder X-ray diffraction (P-XRD) analysis was conducted using a BRUKER AXS diffractometer (Germany) equipped with Cu $K\alpha_1$ radiation (λ = 1.5406 Å), to assess the crystalline nature and phase composition of the synthesized samples.

2.3. Preparations of 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol (L1)

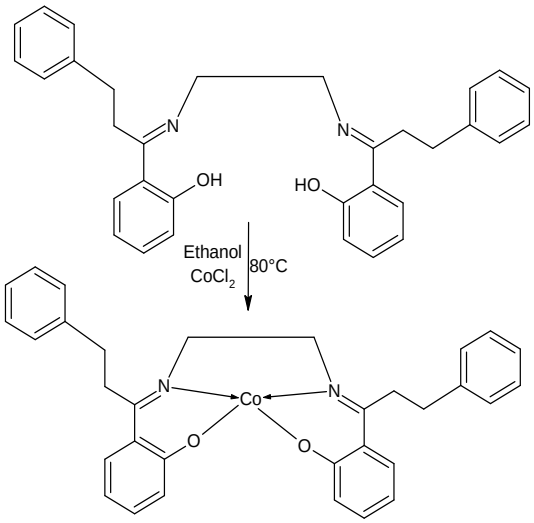
Place 2.5 gm of 2-hydroxy-3-phenylpropiophenone in a clean 500 ml round-bottom flask, add 25 ml of ethanol, and heat it up to 85 degrees Celsius. Once the temperature is maintained at 85 degrees, add ethylenediamine mixed with 10 ml of ethanol slowly within 30 min at the same temperature. Maintain the reaction till all 2-hydroxy-3-phenylpropiophene gets converted into 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol. Check TLC after 4 hrs for completion of the reaction. After 4 hrs, cool the reaction mass at room Temperature gradually started crystal formation near to room temperature, then chilled it up to 20 degrees for 1 hr, filtered it, and washed it with chilled ethanol to obtain crystalline 2-2' ((1E,1'E-(ethane-1,2-diylbis(azanylylidene))bis(3-phenylpropan-1-yl-1-ylidene)) diphenol.



Scheme 1: 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol (L1)

2.4. Preparation of Cobalt complex of 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol (L1M1)

Place 2.0 g of 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol (0.00419 mol) was placed in a clean and dry round-bottom flask (RBF). To this, 25 mL of ethanol was added. The reaction mixture was heated to 85 °C until a clear solution was obtained. Subsequently, 0.004 mol of $CoCl_2.6H_2O$ pre-dissolved in 10 mL of demineralized (DM) water was added dropwise to the reaction mixture. A precipitate of the metal complex began to form within 30 minutes. The reaction was stirred continuously for 4 hours at the same temperature. After completion of the reaction, the mixture was allowed to cool at room temperature and then further chilled to 20 °C for 2 hours. The solid product was filtered and washed with 10 mL of chilled ethanol. The crude solid was then recrystallized using DMSO as the solvent. The purified product was suitable for further characterization by NMR, IR & XRD and other analytical technique.



Scheme 2: Cobalt complex of 2-2'((1E,1'E-(ethane-1,2-diylbis(azanylylidene)) bis(3-phenylpropan-1-yl-1-ylidene)) diphenol (L1M1).

3. RESULTS AND DISCUSSION

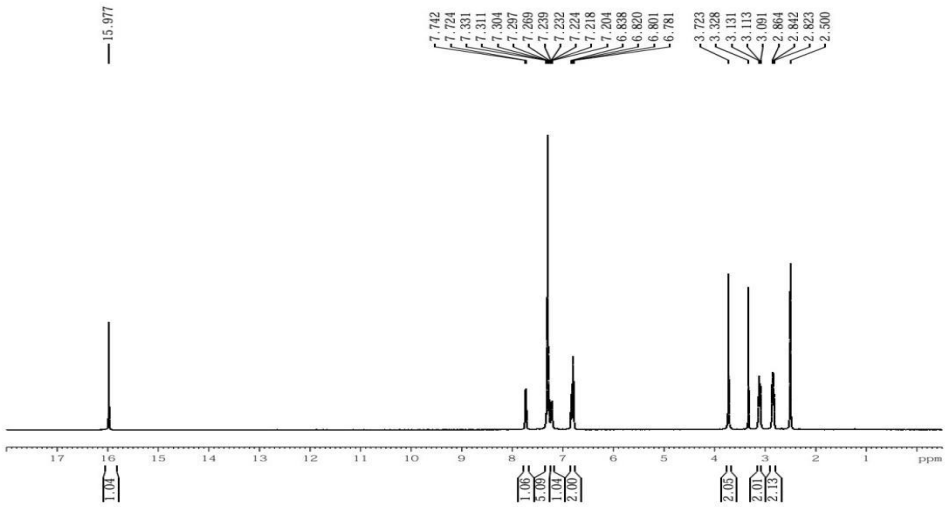
INFRARED SPECTROSCOPIC STUDY

The mode of binding between the ligands and the metal salts in complexes was investigated by comparing the IR spectrum of the free ligand and those of the complexes. The FT-IR spectra of L_1 was characterized by bands at (1405), (3010), (1295, 1078), (1631), and (3250) cm^{-1}. These can be assigned to (νAr-C-C), (νAr C-H), (νCH_2N), (νC=N), (νC-O) and (νO-H) respectively. In the L_1M_1 complex a negative shift with reduced intensity were observed for (C=N) and (CH_2N) stretching bands. The characteristic phenolic ν(OH), due to the presence of a hydroxy group at the o-position was observed in L_1 at 3250cm^{-1}, and a band at 1455 cm^{-1} due to ν(C–O) of the phenolic group was also observed in ligand L_1. The sharp band due to the ligand's phenolic (OH) is absent in the complex L_1M_1 and monometallic indicate the coordination of the phenolic oxygen to the metal ion after deprotonation. Such coordination is also supported by shifting the phenolic ν(C–O) band to lower wave numbers at 1440 cm^{-1} in the complex L_1M_1. The coordination of the azomethine nitrogen and phenolic oxygen in metallic complex L_1M_1 is further supported by the appearance of two non-ligand bands at 510–600 and 400–460 cm^{-1} due to ν(M–O) and ν(M–N), respectively.

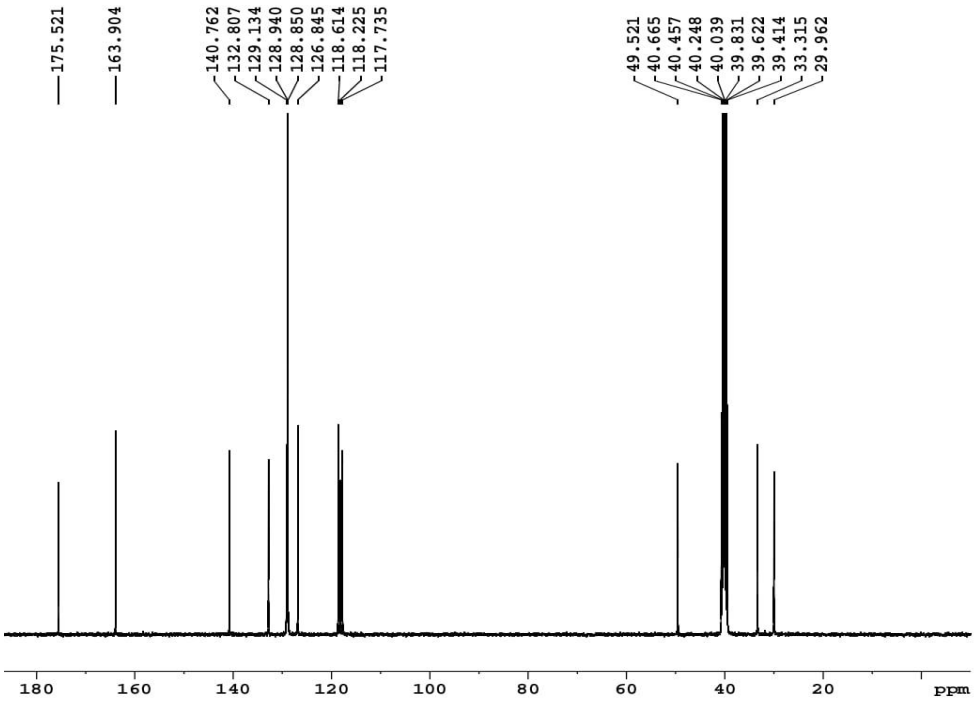
Complex/ Ligand	ʋ Ar C=C	ʋ Ar C-H	ʋ CH2-N	ʋ C=N	ʋ N=O	ʋ C-O	ʋ M-O	ʋ M-N	ʋ O-H
(L1)	1405 (s)	3010 (m)	1295, 1078(s)	1631 (s)	-	1455 (s)	-	-	3250 (m)
(L1M1)	1451.17 (s)	3002.2 (m)	1280,1066 (s)	1617.02 (s)	-	1440 (s)	510–600 (m)	400–460 (m)	-

NMR SPECTROSCOPY

Schiff base ^{1}H NMR were recordet as (400 MHz, DMSO-d_6) δ 15.9 (s, 1H, -OH), 7.73 (d, J = 8 Hz, 2H, Ar-H), 7.33–7.20 (m, 10H, Ar-H), 3.7 2 (s, 4H, –CH_2N–), 3.13–3.09 (t, 4H, Ar-CH_2–), 2.86–2.82 (t, 4H, Ar-CH_2–CH_2–).

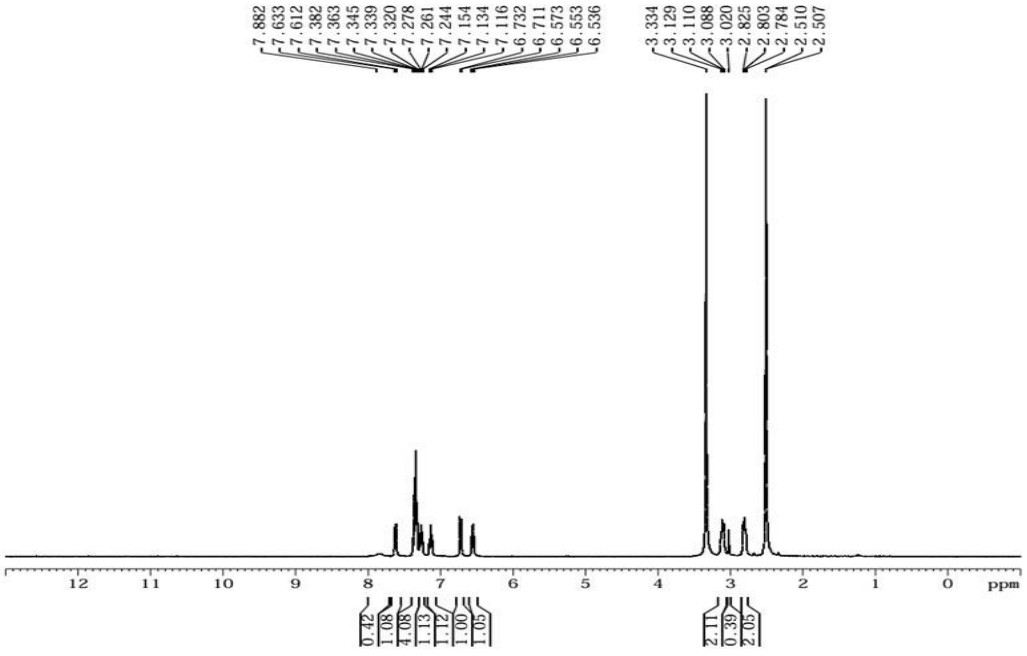


^{13}C NMR (100 MHz, DMSO-d_6): δ = 175.52, 163.90 (C=N and phenolic C–O carbons), 140.76, 132.80, 129.13, 128.94, 128.50, 126.85, 118.24, 117.73 (Ar-C), 49.52, 40.66, 40.45, 40.03, 39.62, 33.41, 29.96 (Ar-CH_2 –CH_2– carbons).



Cobalt Meatal complex ¹H NMR were recordet as (400 MHz, DMSO-d_6).

δ 7.70 (d,J=8.0Hz Ar–H, 2H), 7.63-7.11 (m, Ar–H, 12H), 6.72 (d, Ar–H, 2H), 6.71–6.53 (m, Ar–H, 2H), 3.33 (s, –N–CH_2–, 4H), 3.12–3.08 (t, –CH_2–, 4H), 2.82–2.78 (t, Ar–CH_2–CH_2–, 4H).The disappearance of the phenolic –OH signal at 13–15 ppm indicates coordination of the phenolic oxygen to the metal ion, confirming complex formation.



POWDER X-RAY DIFFRACTION (PXRD)

Powder X-ray diffraction (PXRD) measurements for complex 1 were performed at room temperature to evaluate its crystalline nature and phase homogeneity. The XRD patterns were collected using Cu Kα radiation (λ = 1.5405 Å) at 45 kV and 40 mA, with a scan rate of 5° (2θ) min^{-1}. Powder X-ray Diffraction (PXRD) analysis utilized Shimadzu XR-6100 instrument with Cu (Kα= 1.54 Å) scan rate of 2 deg/min with 0.15 mm receiving data. The obtained diffraction profile displayed sharp and intense peaks, which are characteristic of a well-defined crystalline phase, thereby confirming the successful formation of the complex in pure form.

PHYSICAL CHARACTERISTICS AND ELEMENTAL ANALYSIS

Complex/ ligand	MW (g mol-1)	COLOUR	%YIELD	% C	% H	% O	%N	% Co
80				Found (Calculated)				
(L1)	476.60	Yellow	75%	80(80.63)	6.5(6.71)	6.68(6.71)	2.91(2.93)	-
(L1M1)	533.28	Dark brown	85%	71.2(72.04)	5.5(5.67)	5.75(6.0)	5.0(5.25)	10.8(11.05)

ANTI-BACTERIAL

We tested the synthesized Co(II) complex's ability to kill bacteria in vitro against four harmful strains: Bacillus subtilis, Staphylococcus aureus, Salmonella typhi, and Escherichia coli. Streptomycin (1 mg/mL) was the standard reference drug for the study, and DMSO was the negative control.We tested the complex's ability to kill germs at two different levels, 5 mg/mL and 10 mg/mL, to see how it reacted to different amounts. The compound showed a clear dose-dependent pattern, with larger areas of inhibition at higher doses. The inhibition zones were 10 mm for S. aureus, 1 mm for S. typhi, 13 mm for E. coli, and 04 mm for B. subtilis at a dose of 5 mg/mL. When the dose was raised to 10 mg/mL, the zones of inhibition got a lot better. They were 15 mm, 02 mm, 16 mm, and 17 mm against S. aureus, S. typhi, E. coli, and B. subtilis, respectively. Streptomycin, a common antibiotic, worked well against all types of bacteria (25–30 mm), but DMSO did not work at all. This showed that the molecule being tested was picky.The findings indicate that the synthesized Co(II) complex exhibits moderate to robust antibacterial activity, particularly against S. aureus, E. coli, and B. subtilis. The response to S. typhi is not very good.The overall results show that the chemical works better when there is more of it. It might be a good place to start making new antimicrobial drugs that work better against more types of bacteria.

Antibacterial Activities of samples against S. typhi, S. aureus, B. subtilis, E. coli.

Sr no	Sample/concentration	S.aureus (mm)	S. Typhi (mm)	E. coli. (mm)	B. Subtilis (mm)
1	Control(DMSO)	-	-	-	-
2	Streptomycin (1 mg/mL)	25	30	30	28
3	Com/G-04/25(5mg/mL)	10	1	13	04
4	Com/G-04/25(10mg/mL)	15	02	16	17

ANTI-FUNGAL

The Agar Well Diffusion Method was used to see how well Sample CO worked against fungi. The Sabouraud Dextrose Agar (SDA) medium was made and then sterilized for 15 minutes at 121°C. About 25 mL of melted SDA (45°C) was put into sterile Petri dishes and permitted to harden. Fungal suspensions of Candida albicans and Aspergillus niger) were prepared in sterile normal saline and standardized using a haemocytometer. Wells of 6 mm diameter were bored aseptically using a sterile cork borer. 100 µL of Sample CO solution at concentrations of 5 mg/mL and 10 mg/mL were added into respective wells. Fluconazole (1 mg/mL) was used as standard, and DMSO served as negative control. The plates were incubated at 20–25°C for 72 hours. After incubation, the diameter of the clear zone of inhibition was measured in millimeters (mm).

Antibacterial Activities of samples agains Candida albicans & Aspergillus niger

Sr. No	Sample	Concentration	Candida albicans (mm)	Aspergillus niger (mm)
1	Standard (Fluconazole)	1 mg/mL	25	30
2	Sample CO	5mg/mL	15	03
3	Sample CO	10mg/mL	19	10

4. CONCLUSION

The cobalt metal complex was synthesized. This synthesized metal complex is characterized with FT-IR, ^{1}H NMR, elemental analysis, and P-XRD. This cobalt metal complex is tested against bacterial strains such as Staphylococcus aureus, Salmonella Typhi, Escherichia coli, and Bacillus subtilis and is used to check the antibacterial activities of the cobalt metallic complex. The biological activity data showed that the metal complex exhibits good antibacterial activities. So that metal complex possesses nice antimicrobial activity; it can be used to prevent various bacterial growth. This cobalt metal complex demonstrated concentration-dependent antifungal activity. It inhibited Candida albicans moderately at low concentrations and had minimal activity against Aspergillus niger. The antifungal activity increased at higher concentrations, with more inhibition of Candida albicans and Aspergillus niger. In general, the cobalt metal complex shows good antifungal activity, especially against Candida albicans, and works better at higher concentrations. This suggests that it could be useful for more research in the pharmaceutical and biological fields.

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