Synthesis, Characterization and X-Ray Structure of Mononuclear Cu(II) Complex of The Schiff Base 2,2'-[1,1'-(Ethane-1,2-Diyl)bis(nitriloethylidyne)]diphenol

ABSTRACT

The reactions of the Schiff base 2,2'-[1,1'-(ethane-1,2-diyl)bis(nitriloethylidyne)]diphenol (H_2L), which was synthesised by condensation of 2-hydroxyacetophenone and ethylenediamine in 2:1 ratio, with acetate copper (II) salt afforded a new mononuclear complex formulated as $[Cu(L)]\cdot(H_2O)_{0.5}$. The ligand and the complex have been characterized by elemental analysis, FTIR, UV-Vis and ^{1}H and ^{13}C NMR spectroscopies. The characterization of the copper complex was completed by molar conductivity and room temperature magnetic measurements. The structure of the complex has been resolved by X-ray crystallography technic. The mononuclear copper (II) complex crystallises in the monoclinic space group C2/c with the following unit cell parameters a = 26.131(6) Å, b = 7.293(5) Å, c = 17.256(4) Å, β = 106.10(5)°, V = 3160(2) Å^3, Z = 4, R_1 = 0.077 and wR_2 = 0.145. In the structure of the complex the ligand acts in tetradentate fashion and the coordination environment of the copper atom is best described as distorted square planar geometry.

Keywords: 2-hydroxyacetophenone, Schiff base, Copper, Mononuclear, FTIR, XRD

1. INTRODUCTION

Schiff bases are organic ligands widely studied in coordination chemistry. They are derived from the condensation of amine derivatives and ketone or aldehyde derivatives and constitute a wide range of organic ligands (Haj et al., 2020; Mighani, 2020; Segura et al., 2016). These ligands, which can have biological or physical properties, are often used in the preparation of transition metal complexes that can enhance the properties of the free ligands (Al Zoubi et al., 2018; El-Sonbati et al., 2019; Gul et al., 2024; Hosny et al., 2024; Venkatesh et al., 2024). Schiff bases can generate mononuclear (Middya et al., 2025; Paul et al., 2024), dinuclear (A. H et al., 2024; Huang et al., 2024) or polynuclear complexes (Chakraborty et al., 2024; Mahapatra et al., 2024; Nowicka et al., 2024). The interest of chemists in these compounds has increased considerably in recent years because of the exceptional properties observed for these molecules. Some coordination compounds derived from Schiff bases have been tested for their biological activities and have been shown to be antibacterial (Anacona et al., 2021; Kargar et al., 2022), antifungal (Amirthaganesan et al., 2022; Frei et al., 2021), anticancer (Alorini et al., 2022; Jiang et al., 2024), or

antituberculosis (Bufarwa et al., 2024; Malav & Ray, 2024). In these compounds, the presence of several metal ions can induce cooperation between the metal centers and improve physical properties such as magnetism (Chen et al., 2024), fluorescence (Dong et al., 2024) or optics (Turan et al., 2024) and catalytic properties (Parada et al., 2024; Rezaie et al., 2024). Schiff bases and their coordination compounds have also been used in the field of biomimetic chemistry to understand the behavior of some metalloenzymes, (Mandal et al., 2020; Mureseanu et al., 2024; Shahraki, 2022). In particular, hydroxyacetophenone is a keto-precursor that enables the synthesis of Schiff bases used in the preparation of biomimetic complexes (Banerjee & Paine, 2020; Chandra, 1984; J. M. Mir & Maurya, 2018; Nasr-Esfahani et al., 2009). In our previous studies, we reported the preparation and use of 2-hydroxyacetophenone-derived Schiff bases to prepare transition metal complexes with novel structures and interesting properties such as antioxidation (Fall et al., 2020; Faye et al., 2021; Kébé et al., 2021; Mamour et al., 2018). This study aims to isolate and characterize a copper(II) Schiff base complex obtained by the condensation reaction between 2-hydroxyacetophenone and ethylenediamine.

2. EXPERIMENTAL SECTION

2.1. Starting materials and Instrumentations

All chemicals and solvents were of analytical reagent grade and were used directly without further purification. Elemental analyses of C, H and N were recorded on a VxRio EL Instrument. Infrared spectra were obtained on a FTIR Spectrum Two of Perkin Elmer spectrometer in the 4000-400 cm^{-1} region. The UV-Visible spectra were recorded on a Perkin Elmer Lambda UV-Vis spectrophotometer. The ^{1}H NMR spectra were recorded at 300 MHz and $^{13}C\{^{1}H\}$ NMR spectra at 75 MHz on a Bruker AC–300 instrument. The molar conductance of 1×10^{-3} M in DMF solutions of the metal complex was measured at 25 °C using a WTW LF-330 conductivity meter with a WTW conductivity cell. Room temperature magnetic susceptibility of the powdered sample was measured using a Johnson Mattey scientific magnetic susceptibility balance (Calibrant: $Hg[Co(SCN)_4]$).

2.2. Synthesis of 2,2'-[1,1'-(ethane-1,2-diyl)bis(nitriloethylidyne)]diphenol (H_2L)

The Schiff base H_2L was synthesized by mixing 2-hydroxyacetophenone (6.02 mL, 0.050 mmol) and ethylenediamine (1.70 mL, 0.025 mmol) in 50 mL of absolute ethanol. The reaction mixture was heated under reflux for four hours. After cooling the mixture at room temperature, yellowish powder appears. The compound was filtered and recrystallized from ethanol. Yield, 83 %, M. P. 199 °C. Anal. Calc for $C_{18}H_{20}N_2O_2$: C, 72.95; H, 6.80; N, 9.45. Found: C, 72.91; H, 6.77; N, 9.40.

IR (cm^{-1}): 3423 ($\nu_{O—H}$), 1612 ($\nu_{C=N}$), 1285 (δ_{C-O}), 1550-1450 ($\nu_{C=C}$). ^{1}H NMR (DMSO-d_6, δ(ppm)): 2.52 (S, 6H, -N=C-C**H**$_3$); 3.99 (S, 4H, -C**H**$_2$-); 6.80-7.55 (m, 8H, =C**H**$_{Ar}$); 12.93 (s, 2H, **H**O-). ^{13}C NMR (DMSO-d_6, δ(ppm)): 14.80 (-**C**H_3), 50.12 (-**C**H_2-), 117.49 (**C**$_{Ar}$), 118.53 (**C**$_{Ar}$), 119.72 (**C**$_{Ar}$), 132.39 (**C**$_{Ar}$), 132.79 (**C**$_{Ar}$), 163.63 (**C**$_{Ar}$-OH), 172.39 (**C**=N). UV/Vis (DMF, λ (nm)): 243, 324.

2.3. Synthesis of the copper(II) complex

In a 100 mL round bottomed flask containing 20 mL of methanol, H_2L (0.2961 g; 1 mmol) was introduced with stirring. To this solution, a 10 mL methanolic solution containing $Cu(OAC)_2 \cdot 4(H_2O)$ (0.2 g; 1 mmol) was added. The mixture was heated to reflux for two hours. After cooling, the brown solution was filtered, and the filtrate was left to evaporate slowly for two weeks. Brown single crystals suitable for XRD are collected by filtration. Yield, 67%. Anal. Calc for $C_{18}H_{19}N_2O_{2.5}Cu$: C, 58.92; H, 5.22; N, 7.64. Found: C, 58.88; H, 5.17; N, 7.59. IR (cm^{-1}): 3458 (ν_{O-H}), 1588 ($\nu_{C=N}$), 1220 (δ_{C-O}). Conductance Λ ($S \cdot cm^2 \cdot mol^{-1}$): 10 (fresh solution) et 12 (two weeks after). UV/Vis (DMF, λ (nm)): 230, 279, 372, 554. Magnetic moment : μ_{eff} = 1.76 μ_B.

2.4. Crystal structure determination

Details of the X-rays crystal structure solution and refinement are given in Table 1. Diffraction data were collected using an ENRAF NONIUS Kappa CCD diffractometer with graphite monochromatized MoKα radiation (λ = 0.71073 Å). All data were corrected for Lorentz and polarization effects. No absorption correction was applied. Complex scattering factors were taken from the program package SHELXTL (Sheldrick, 2015b). The structures were solved by direct methods which revealed the position of all non-hydrogen atoms. All the structures were refined on F^2 by a full-matrix least-squares procedure using anisotropic displacement parameters for all non-hydrogen atoms (Sheldrick, 2015a). The hydrogen atoms of water molecules and NH groups were located in the Fourier difference maps and refined. Others H atoms (CH and CH_3 groups) were geometrically optimized and refined as riding models by AFIX instructions. Molecular graphics were generated using ORTEP-3 (Farrugia, 2012).

Table 1. **Crystallographic data and refinement parameter for the complex.**

Chemical formula	$[2(C_{18}H_{18}CuN_2O_2)] \cdot H_2O$
M_r	733.80
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	293
a (Å)	26.131 (6)
b (Å)	7.293 (5)
c (Å)	17.256 (4)
β (°)	106.10 (5)
V (Å^3)	3160 (2)

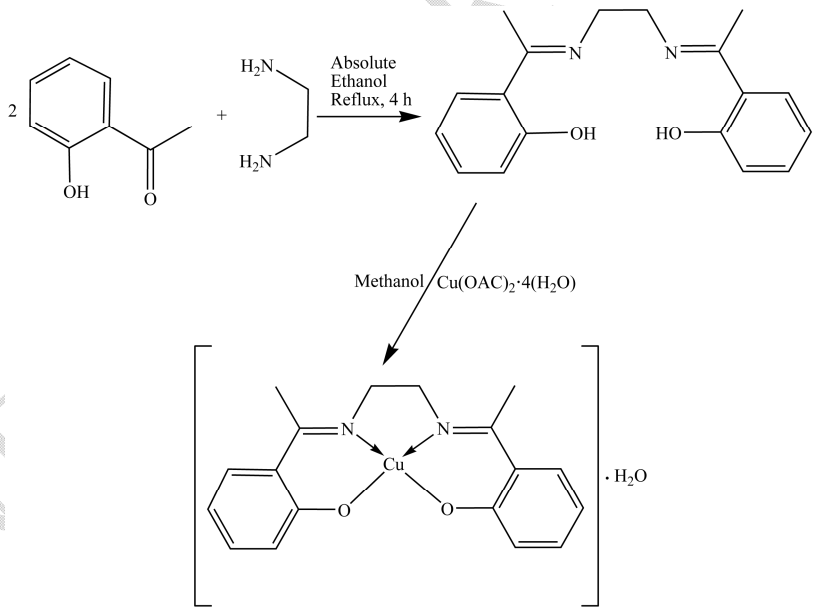
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.40
Crystal size (mm)	0.19 × 0.08 × 0.05
No. of measured reflections	16310
No. of independent reflections	4504
No. of observed [$I > 2\sigma(I)$] reflections	2135
R_{int}	0.077
$R[F^2 > 2\sigma(F^2)]$	0.077
$wR(F^2)$	0.145
GOF	1.11
No. of reflections	4504
No. of parameters	215
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e $Å^{-3}$)	1.03, −0.72

3. RESULTS AND DISCUSSION

3.1. General study

The symmetrical Schiff base ligand 2,2'-[1,1'-(ethane-1,2-diyl)bis(nitriloethylidyne)]diphenol (H_2L) was prepared by refluxing acetophenone and ethylene diamine in 2:1 ratio as shown in Scheme 1. The ^{1}H NMR spectrum of the ligand exhibits a broad signal pointed at 12.93 ppm attributed to the phenolic protons. The signals of the aromatic protons appear in the range 6.80-7.55 ppm as a complex multiplet. The signals due to the methylene and the methyle protons appear, respectively, at 3.99 ppm and 2.52 ppm. The ^{13}C NMR spectrum reveal two signals at 172.39 and 163.63 ppm attributed to the carbon atoms of the azomethine group and the C_{ipso}-OH of the aromatic ring. The signals due to the other aromatic carbon atoms appear in the range 117.49-132.79 ppm. The signals due to the methyle and the methylene carbon atoms are pointed, respectively, at 14.80 and 50.12 ppm. The FTIR of the free ligand shows a broad band at 3423 cm^{-1} attributed to the ν_{O-H} stretching of the phenolic group. The $\nu_{C=N}$ stretching band is pointed at 1612 cm^{-1}. The ν_{C-O} stretching appears at 1285 cm^{-1}. The bands which appear in the range 1550-1450 cm^{-1} are attributed to the C=C of the aromatic ring. The electronic spectrum of the free ligand shows two bands pointed at 243 nm and 324 nm which are attributed to the $\pi\rightarrow\pi^*$ and $\pi\rightarrow n^*$ of the

phenyl ring and -C=N- bond (Kenđel et al., 2020), respectively. The copper complex was synthesized by mixing H_2L ligand in 15 mL of methanol with copper acetate in a molar ratio 1:1. Elemental analyses gives result in agreement with the chemical formula obtained from X-ray diffraction study. The FTIR spectrum of the complex reveals the absence of the band due to ν_{O-H} which appeared in the spectrum of the free ligand at 3423 cm^{-1}. This observation is indicative of the deprotonation of the phenolic groups. The band due to $\nu_{C=N}$ stretching shifts to low frequencies and appears at 1588 cm^{-1}. This fact is indicative of the involvement of the nitrogen atom of the azomethine moiety in the coordination. The band due to ν_{C-O}, which appears at 1285 cm^{-1} in the spectrum of the free ligand is pointed at 1220 cm^{-1}, suggesting the coordination of the phenolate oxygen to the copper(II). The electronic spectrum of the complex shows four bands. The bands at 230 and 279 nm are attributed to the $\pi \rightarrow \pi^*$ and $\pi \rightarrow n^*$ of the aromatic ring and the -C=N- bond. The band at 372 nm is due to the MLCT (Saha et al., 2014). The electronic spectrum displays also a d→d band at 554 nm which is attributed to $^2B_{1g} \rightarrow {}^2A_{1g}$ transition (Dey et al., 2021). The magnetic moment value of 1.76 μ_B of the copper(II) complex is in good agreement with paramagnetic copper(II) complexes (Srivastava et al., 2021) having one unpaired electron with d^9 system. The conductimetry measurement (10 $S \cdot cm^2 \cdot mol^{-1}$) showed that the copper (II) complex is neutral electrolyte type (Geary, 1971). On standing for two weeks the conductance remains quite constant, indicating good stability of the complex in DMF solution.



Scheme 1. Chemical diagrams for the ligand and the complex.

3.2. Description of the structure of the complex $[Cu(C_{18}H_{18}O_2N_2)] \cdot (H_2O)_{0.5}$

The molecular structure of the cooper(II) complex presented in this work was solved by X-ray diffraction and is illustrated Fig. 1. The main bond lengths and angles are shown in Table 2. The asymmetric unit is

composed of one copper(II) ion, one dinegative ligand molecule and a half uncoordinated water molecule. The ligand is coordinated through two nitrogen atoms of azomethine moieties and two phenolate oxygen atoms, yielding a tetracoordinated copper(II) ion which is situated in an N_2O_2 environment. For elucidating the geometry around the metal ion in tetracoordinated complexes, the trigonality parameter τ_4 (τ_4 = [360 º-(α + β)]/141 (α and β are the largest angles around the metal center) is used : τ_4 = 1 define a perfect tetrahedral geometry and τ_4 = 0 describe a perfect square planar geometry) (Singh et al., 2017). The τ_4 value of 0.068 is indicative of a distorted square planar geometry around the copper(II) for the title complex. The *cissoid* angles which are in the range [86.83 (14)° - 92.99 (15)°] deviate from the ideal value of 90°. The *transoid* angles values of 174.91(16)° [N1-Cu1-O2] and 175.55 (17)° [N2-Cu1-O1] deviate from the ideal value of 180°. The sum of the angles subtended by the atoms defining the square planar geometry is 360.39°. The coordinating atoms N1/N2/O1/O2, from the ligand are, approximately, in the same plane (planar rms deviation of 0.0785 Å) and the copper atom is located 0.004 Å out of this plane. Upon coordination of the ligand to the copper(II), one five-membered ring and two six-membered rings were formed. The atoms defining the five membered-ring Cu1/N1/C9/C10/N2 are coplanar with rms deviation of 0.0835 Å. The atoms defining the six membered-ring Cu1/O1/C1/C6/C7/N1 and Cu1/O2/C18/C13/C11/N2 are coplanar with rms deviation of 0.0676 Å and 0.0462 Å, respectively. The mean plane of the five-membered ring forms dihedral angle of 5.852(2)° and 4.591(2)° with the means planes of the two six-membered-rings, respectively. The means planes of the two six-membered-rings form a dihedral angle of 4.472(1)°. The Cu-N bond lengths [Cu1-N1 = 1.931 (4) Å and Cu1-N2 = 1.934 (4) Å] are longer than the Cu-O [Cu1-O1 = 1.878 (3) Å and Cu1-O2 = 1.876 (3) Å] distances. These values are close proximity to the values reported for the complex Chlorido(2-{1-[(2-morpholinoethyl)-imino]ethyl}phenolato-κ^3*N*,*N*',O)-copper(II) (Ikmal Hisham et al., 2011). The bond length of 1.299(5) Å for C1-O1 and 1.303(5) for C18-O2 are compatible with data reported for C-$O_{phenolate}$ distances (Zhang et al., 2009; Zhao et al., 2008). Additionally, C7-N1 [1.286 (5) Å] and C11-N2 [1.292 (6) Å] have double bond character and the values of 1.471(5) Å and 1.461(5) Å for C9-N1 and C10-N2 are indicative of single bond character (Seck et al., 2020). No hydrogen-bonds are found in the structure. The packing diagram of the structure is illustrated in Fig. 2.

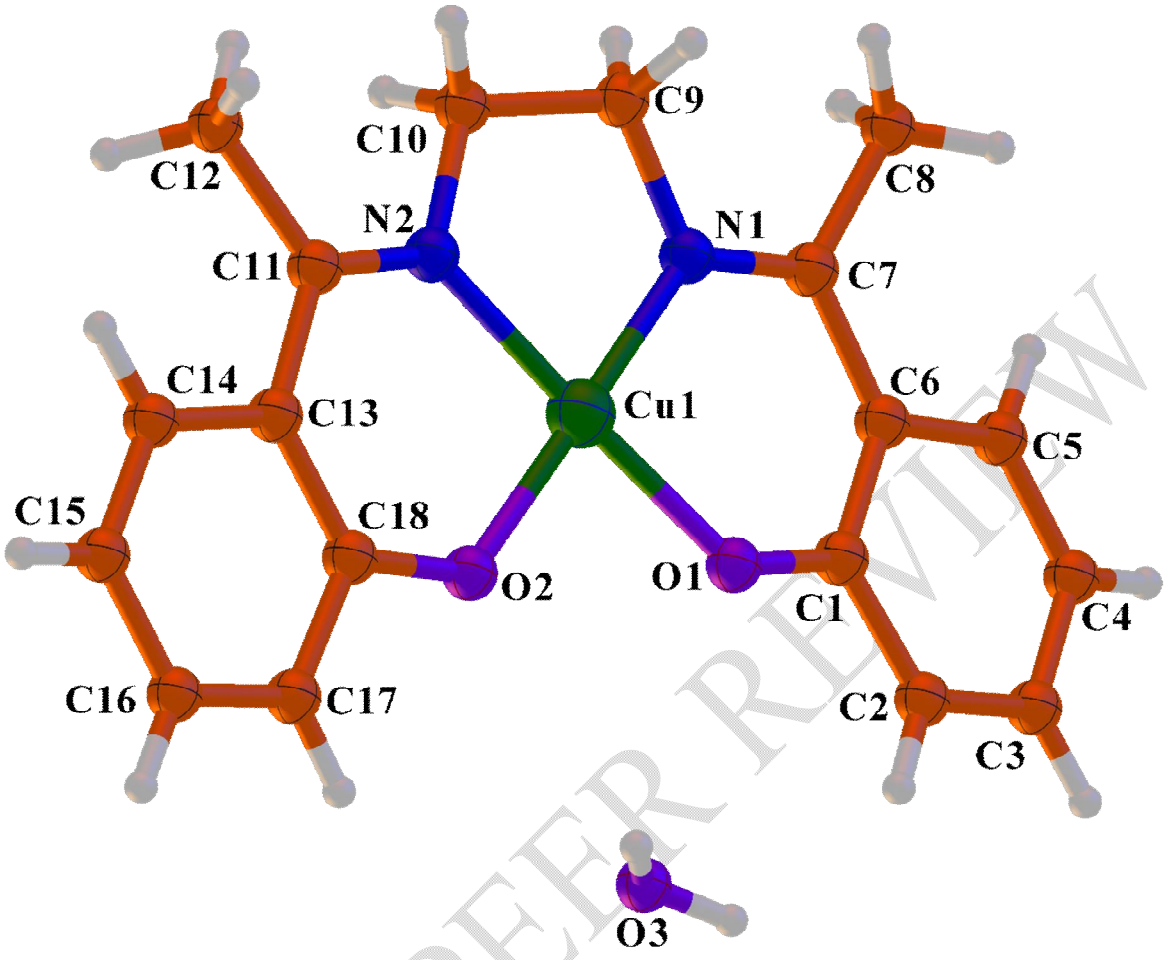


Fig. 1. Crystal structure of the mononuclear copper (II) complex.

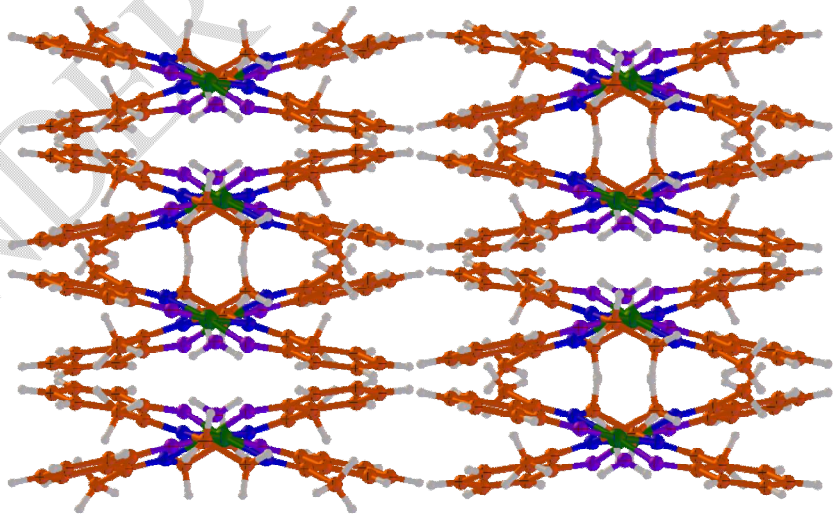


Fig. 2. The packing of the complex in the crystal structure.

Table 2. Selected geometric parameters (Å, °).

Cu1—O2	1.876 (3)	O1—C1	1.299 (5)
Cu1—O1	1.878 (3)	N2—C11	1.292 (6)
Cu1—N1	1.931 (4)	N2—C10	1.461 (5)
Cu1—N2	1.934 (4)	N1—C7	1.286 (5)
O2—C18	1.303 (5)	N1—C9	1.471 (5)
O2—Cu1—O1	86.83 (14)	O2—Cu1—N2	92.99 (15)
O2—Cu1—N1	174.91 (16)	O1—Cu1—N2	175.55 (17)
O1—Cu1—N1	92.54 (15)	N1—Cu1—N2	88.03 (15)

4. CONCLUSION

The prepared organic ligand H_2L coordinates to copper(II) ion yielding a mononuclear complex. The compounds are characterized by elemental analysis, FTIR, UV-Vis and NMR spectroscopies, room temperature magnetic moment measurement and molar conductivity. The structure of the mononuclear copper (II) complex is established by single-crystal X-ray diffraction. The complex is non-ionic electrolyte in DMF solution. The organic molecule H_2L acts in its dideprotonated form as tetradentate ligand through two nitrogen azomethine atoms and two phenolate oxygen atoms. Considering the magnetic moment, infrared spectra and the electronic spectrum data for Cu(II) complex, square planar geometry is proposed for the Cu(II) complex as observed in X-ray diffraction study.

5. SUPPORTING INFORMATION

CCDC-1844618 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via https://www.ccdc.cam.ac.uk/structures/, or by, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK;.

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