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Synthesis and characterization of Cu (II), Co (II), Ni (II), Au (III), complexes with (Z)-1-(4-((3-chlorobenzylidene) amino)phenyl)ethan-1-one ligand

Ghufran Sh. Al-Obaidy*, Mohammad F. Mesher, Kaiss R. Ibraheem

ABSTRACT

Background: Schiff bases are considered privileged ligands, as condensation between aldehydes and amines makes them easy to prepare also in the synthetic design, stereogenic centers, or other chirality elements can be introduced. Schiff base ligands can combine and stabilize with several different metals in different oxidation states, allowing Schiff base metal complexes to be used for a wide range of useful catalytic transformations. The reaction in ethanol of 4-aminoacetophenone and 3-chlorobenzaldehyde resulted in the formation of Schiff base (Z)-1-(4-((3-chloro benzylidene)amino)phenyl)ethan-1-one. Ligand contact with different di- and trivalent metal ions were observed and ligand complexes are prepared from the reaction of two ligand (Z)-1-(4-(3-chlorobenzylidene) amino) phenyl) ethan-1-one equivalents with one mole of metal salt as the central ion. Physicochemical and spectroscopic methods were put in place to confirm bonding mode and over all complex structure. These results lead to the preparation of four coordinate complexes, and we suggested the structure of the complexes from the above discussions is tetrahedral except Au complex which is square planar geometry. IR, UV, NMR, mass, conductivity, elemental study, and *magnetic* susceptibility characterized the ligand and its complexes. **Materials and Methods:** The reaction in ethanol of 4-aminoacetophenone and 3-chlorobenzaldehyde with molar ratio (1:1) in absolute ethanol with just a few drops of acetic acid resulted in the formation of Schiff base (Z)-1-(4-((3-chlorobenzylidene)amino) phenyl) ethan-1-one. **Results:** The complexes were prepared from the reaction of the ligand L with metal chloride salts (M) in a 2:1 mole ratio heated under reflux in ethanol, where M= Co⁺², Ni⁺², Cu⁺², and Au⁺³. IR, UV, NMR, mass, conductivity, elemental study, and magnetic susceptibility characterized the ligand and its complexes. These results lead to the preparation of four coordinate complexes: [Co(L)₂(Cl)₂], [Cu(L)₂(Cl)₂], [Ni(L)₂(Cl)₂], and [Au(L)₂(Cl)₂]Cl. **Conclusion:** we concluded that the structure of the complexes from the physicochemical and spectroscopic methods is tetrahedral except the Au+3 complex which is square planar geometry.

KEY WORDS: Imine, Metal complexes synthesis, Spectral characterization

INTRODUCTION

A Schiff base or azomethine is a compound formed from either an aldehyde or an aromatic amine ketone condensation.^[1] It is possible to synthesize Schiff bases from an aromatic amine and a carbonyl compound with a hemiaminal nucleophilic addition, production of amine which was followed by the dehydration.^[2] Schiff bases are a special ligand group with a number of donor atoms exhibiting unique modes of coordination against different metals.^[3] Several types of Schiff base ligands are known and their metal chelates properties have been investigated. Because of their important and interesting properties, these include the ability to bind

toxic and heavy metal atoms, undergo tautomerism, show catalytic reduction and photochromy for Schiff bases and their metal complexes.^[4] Several types of Schiff base ligands are known, and their metal chelate properties have been investigated. As catalysts in various biological systems, polymers, dyes, and medicinal and pharmaceutical fields, these compounds and their metal complexes are very important. We are also identified for use in birth control, food packages, and as an O₂ detector. Schiff bases are versatile ligands which are synthesized from the condensation of an amino compound with carbonyl compounds. These compounds and their metal complexes are very important as catalysts in various biological systems, polymers, dyes, and medicinal and pharmaceutical fields. Their use in birth control, food packages, and as an O₂ detector is also outlined.^[5]

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Reagents

4-aminoacetophenone, 3-chlorobenzaldehyde, and the metal ions were bought from Aldrich. All solvents are analytical grade reagents and purified using standard methods.

Physical Measurements

A Unicam-Mattson 1000 FT-IR spectrometer was used to measure IR (KBr pellets) in the range of 4000–400 cm^{-1} and CsI disks in the range of 400–200 cm^{-1} . Electronic spectra for 10^{-3} M solutions in DMSO using 1.0 cm long quartz cells were obtained at room temperature with UV-Vis Shimadzu 1800 spectrophotometer style from 200–1000 nm. NMR tests were carried out on a 300 MHz spectrometer Spectrospin-Bruker. Samples were dissolved in (CDCl_3). Elemental analyses were conducted on Perkin-Elmer 2400 CHNS elemental analyzer. Atomic absorption (A.A) technique used atomic absorption spectrophotometer Shimadzu (A.A 680 GBC 933 plus) to determine the metal content of the complexes. Measurements of mass spectrometry of solid complexes (70 eV, EI) were performed on a spectrometer Finnigan MAT SSQ 7000. Measurements of conductivity were made on a model 32 YSE conductivity meter. For the measurements, concentration samples of Calcium 1×10^{-3} M in DMSO were used. On a Sherwood magnetic susceptibility balance, magnetic measurements of the solid-state complexes (Gouy method) were reported.

Preparation of the Ligand

A solution of 4-aminoacetophenone (0.675g, 0.005 mol) and 3-chlorobenzaldehyde (0.702 g, 0.005

mol) was mixed together with a molar ratio 1:1 in absolute ethanol with just a few drops of acetic acid and heated to reflux for 3 h. The solid was recrystallized from hot ethanol and dried.^[4]

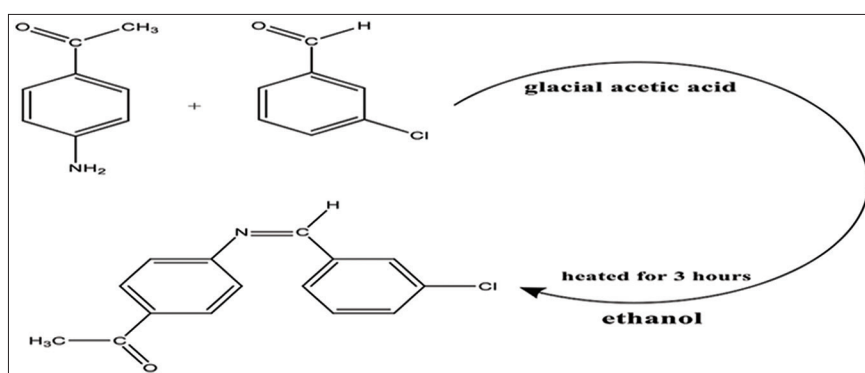
Preparation of Complexes

Complexes were prepared from the reaction of two equivalent of the ligand (Z)-1-(4-((3-chlorobenzylidene) amino) phenyl) ethan-1-one with one mole of metal salt as a central ion. Metal chloride solution in 10 ml absolute ethanol has been added to a round bottom container, volume (100) ml containing a Schiff base ligand solution dissolved in 10 ml ethanol. The mixture of the reaction was stirred and warmed for 4 h under reflux. The complex was collected by filtration, washed with excess methanol and diethyl ether, and dried for 48 h at room temperature.^[5,6]

RESULTS AND DISCUSSION

In this analysis, Schiff base ligand has been identified. L was synthesized by combining a solution of 4-aminoacetophenone and 3-chlorobenzaldehyde together in a 1:1 mole ratio using ethanol as a solvent. Scheme 1 shows that the complexes were prepared from the reaction of the ligand L with metal chloride salts in a 2:1 mole ratio heated under reflux in ethanol to produce pure complexes where isolated in moderate yield. Scheme 2 shows that, Where $M = \text{Co}^{+2}, \text{Ni}^{+2}, \text{Cu}^{+2}, \text{and Au}^{+3}$

All prepared complexes were characterized by physical constant such as melting points and spectroscopic methods (FT-IR, UV-Vis, A.A, and



Scheme 1: Synthesis route for ligand

Table 1: Elemental analysis data and physical properties for ligand and complexes

The Compounds	Molecular Wt.	Yield (%)	M. P (°C)	Color	Found (calcd.) (%)			
					C	H	N	M
C ₁₅ H ₁₂ ClNO (L)	257.72	92.9	65	Brown	69.88 (69.91)	4.73 (4.69)	5.37 (5.44)	
[Co (L) ₂ (Cl) ₂]	645.27	84.3	83	Black	55.73 (55.84)	3.87 (3.75)	4.45 (4.34)	9.06 (9.13)
[Cu (L) ₂ (Cl) ₂]	649.88	77.1	90	Nutty	55.52 (55.45)	3.86 (3.72)	4.26 (4.31)	9.89 (9.78)
[Ni (L) ₂ (Cl) ₂]	645.03	71.4	96	Maroon	55.82 (55.86)	3.81 (3.75)	4.29 (4.34)	9.01 (9.10)
[Au(L) ₂ (Cl) ₂]Cl	818.75	80	110	Dark brown	43.95 (44.01)	3.01 (2.95)	3.34 (3.42)	23.90 (24.06)

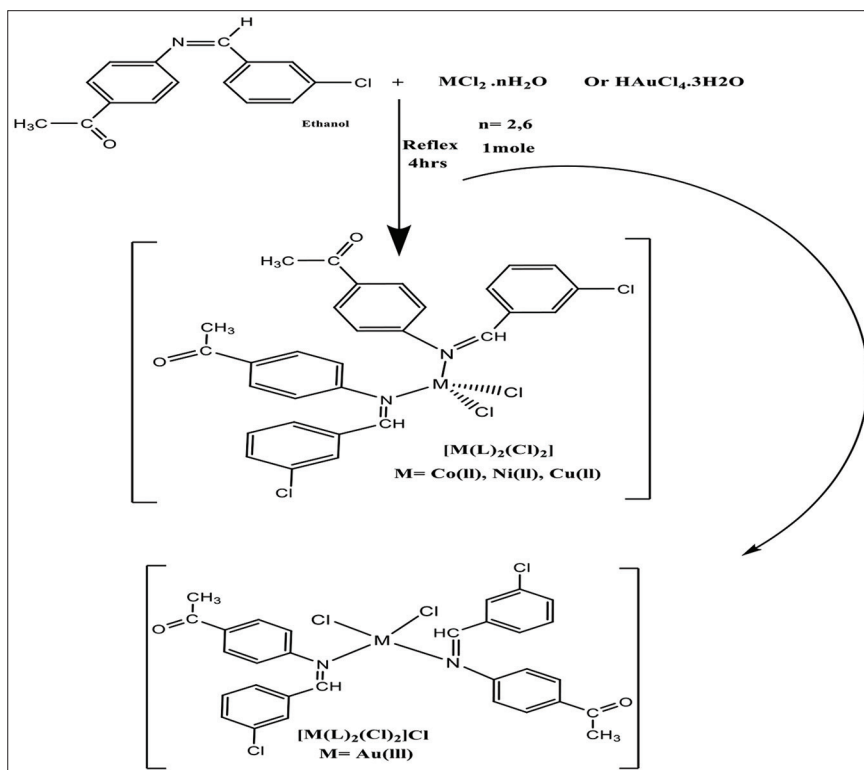
m.p.: Melting point; Calc.: Calculated

mass spectroscopy), elemental microanalysis, also magnetic susceptibility, and molar conductance measurements. Table 1 describes the ligand and complexes of physical properties.

The ¹H-NMR, ¹³C-NMR, and Mass Spectrum for the Ligand

The ¹H-NMR spectrum for the ligand [Figure 1] shows the feature below chemical shift (the solvent CDCl₃): The spectrum of singlet signal was displayed at δ = 2.51

ppm attributed to the one proton for CH₃.^[7] The doublet signal at δ = 6.67 ppm is specified for proton CH(3) and also to CH(3') in the opposite direction (benzene ring). The doublet chemical shifts at δ = 7.44 ppm are referred to proton for CH(6) (benzylidenimin), The triplet chemical shifts at δ = 7.45 ppm are referred to proton for CH(7) (benzylidenimin). The doublet chemical shifts at δ = 7.83 ppm are referred to proton for CH(8) (benzylidenimin). The singlet signal at δ = 7.96 ppm is specified for proton CH(5) (benzylidenimin)



Scheme 2: Synthesis route of $[M(L)_2(Cl)_2]$ and $[Au(L)_2(Cl)_2]Cl$ complexes

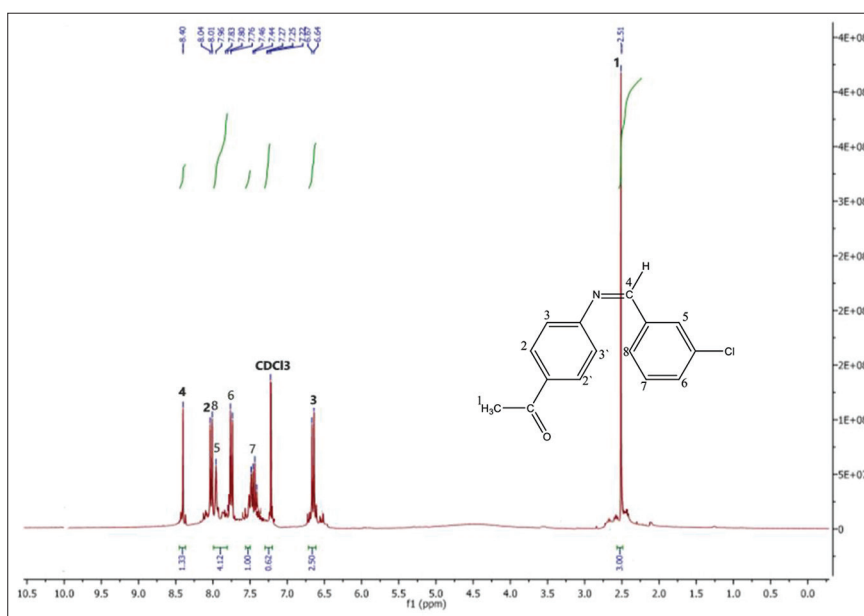


Figure 1: ¹H-NMR spectra for ligand in CDCl₃ as solvent

and doublet chemical shifts at $\delta = 8.04$ ppm are assigned to proton for CH(2) and also to CH(2') in the opposite direction (benzene ring).^[8,9] The singlet signal at $\delta = 8.40$ ppm attributed to the one proton for HC=N(4) (benzylidenimin).^[10] The signal at $\delta = 7.27$ is specified to CDCl₃. The ¹³C-NMR spectrum, in CDCl₃ solvent, shows the number of ligand-assigned signals [Figure 2], construe as below: the forming of the ligand has been detected by tracking signal at $\delta = 196.83$ ppm, which is due to carbon atom C₂ for C=O.^[11] The signal at $\delta = 160.19$ ppm can be attributed to carbon atom C₉ for HC=N group. The carbon C₆ rebounds with the signal at $\delta = 155.82$ ppm which is due to carbon atom C₉ for C-N,^[12] the carbon atoms C₅ and C₇ rebound with the signal at $\delta = 123.17$ ppm and the carbon atoms C₁₁ and C₁₅ rebound with the chemical shifts at $\delta = 126.71$ and 127.51 ppm, respectively. The aromatic ring is assigned to these signals, and the carbon atoms C₄ and C₈ rebound with the signal at $\delta = 128.48$ ppm and the carbon atoms C₁₄, C₁₃, C₁₂, C₁₀, and C₃ rebound with the signal at $\delta = 130.82, 131.85, 133.83, 134.96,$

and 137.52 ppm, respectively.^[13] These signals are attributed to aromatic carbons too. The signal at $\delta = 26.08$ ppm can be attributed to carbon atom C₁ for CH₃.^[11] Finally, the signal at $\delta = 77.80$ ppm refers to CDCl₃. The electrospray (+) mass spectra for L is offered in Figure 3. The spectra display successive fragments linked to ligand structure. The parent ion peak is spotted for the ligand at $m/z = 257$ which corresponds to (M+H)⁺ (%) for C₁₅H₁₂ClNO, requires = 257.72.

The Mass Spectrum for Complexes

The mass spectra of some complexes are offered in Figures 4 and 5. The spectrum exhibits successive fragments concerning to complexes structure. The parent ion peak is observed at $m/z = 649$ and 645 which corresponds to (M+H)⁺ (%) for [Cu (L)₂(Cl)₂], [Ni (L)₂(Cl)₂], requires = 649.88, 645.03, respectively.

The FTIR Spectrum for Ligand and Complexes

The spectrum of (L) shows that no bands (disappeared) around 3332 cm^{-1} and 3396 cm^{-1} were assigned to ν_{as}

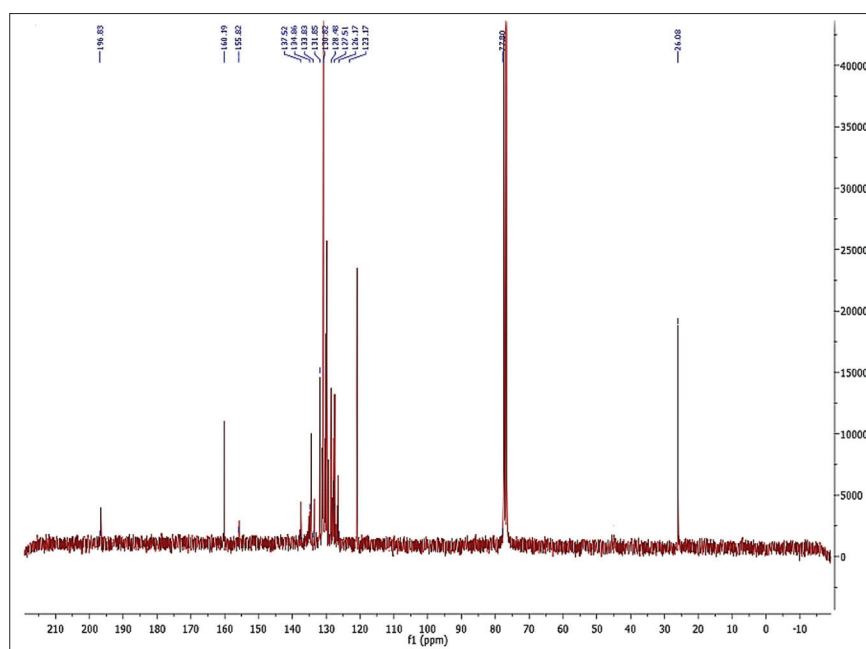


Figure 2: ¹³C-NMR spectra for ligand in CDCl₃ as solvent

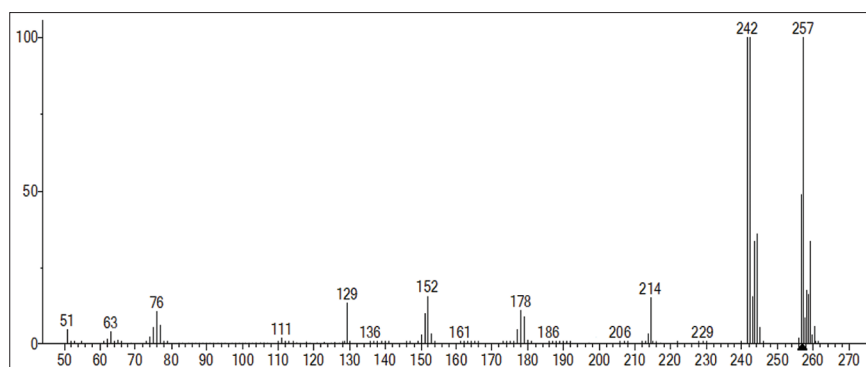


Figure 3: Mass spectra for ligand

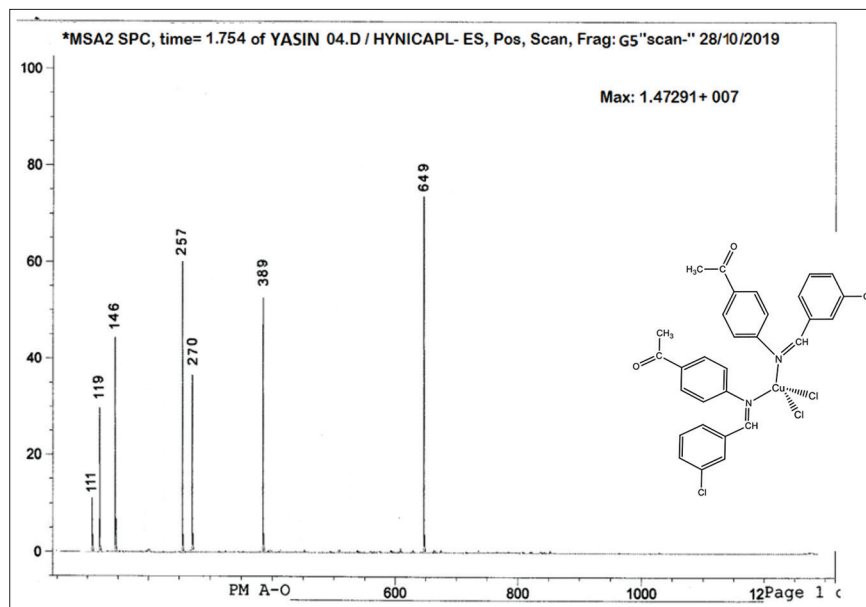


Figure 4: Mass spectrum of complex $[\text{Cu}(\text{L})_2(\text{Cl})_2]$

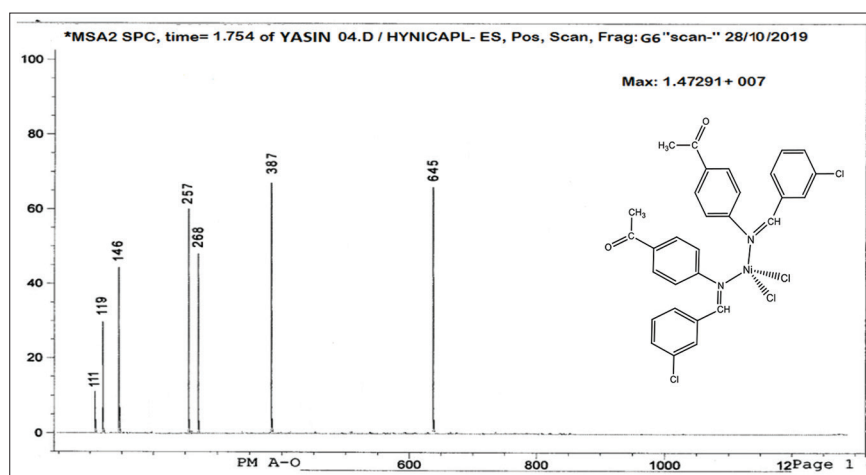


Figure 5: Mass spectrum for complex $[\text{Ni}(\text{L})_2(\text{Cl})_2]$

(NH_2) and $\nu_s(\text{NH}_2)$ stretching vibration, respectively.^[14] The FTIR spectrum reveals that new band at 1674 cm^{-1} can be attributed to $\nu(\text{HC}=\text{N})$ imine group.^[15] The band of imines found to be moved to lower wave numbers after complexation ($1662\text{--}1604\text{ cm}^{-1}$) suggesting their participation in metal coordination.^[16] The bands at 3064 cm^{-1} attributed to the $\nu(\text{C}-\text{H})$ for an aromatic ring, and the band at 1595 cm^{-1} was assigned to $\nu_{ar}(\text{C}=\text{C})$ for the aromatic ring.^[17] The band observed at 1272 cm^{-1} and 684 cm^{-1} recorded to $\nu(\text{C}-\text{N})$ and $\nu(\text{C}-\text{Cl})$ stretching vibration, respectively.^[18,19] The band of the range $308\text{--}435\text{ cm}^{-1}$ is assigned to the $\nu(\text{M}-\text{N})$ vibration where it is absent in the ligand but exhibited as a result of coordination [Table 2].^[20]

UV-Vis Spectral Data for DTC Ligand and Complexes

In DMSO solution, the UV-Vis spectrum of L includes two peaks of absorption at $267\text{ nm} = 37453\text{ cm}^{-1}$ and

$\epsilon_{\text{max}} = 1416\text{ molar}^{-1}\text{ cm}^{-1}$ and at $301\text{ nm} = 33222\text{ cm}^{-1}$ and $\epsilon_{\text{max}} = 1764\text{ molar}^{-1}\text{ cm}^{-1}$ which refer to " $\pi \rightarrow \pi^*$ " and " $n \rightarrow \pi^*$ " transitions, respectively,^[21] as shown in Table 3. Most of transition metal complexes are colored. Such colors vary from ligands and transition metal salts, so these colors are considered essential indicators of coordination, so the colored complexes show different bands of absorption in location and strength.^[22]

Molar Conductivities and Magnetic Susceptibility

The values of molar conductivities in DMSO of Co^{II} , Ni^{II} , and Cu^{II} complexes are 5.3, 9.1, and $10.8\text{ ohm}^{-1}\cdot\text{cm}^2\cdot\text{mol}^{-1}$, respectively, be a sign of the prepared complexes are nonelectrolytes,^[23] while conductance measurement values of Au^{III} complexes are $35.2\text{ ohm}^{-1}\cdot\text{cm}^2\cdot\text{mol}^{-1}$. These values indicate the ionic nature and the type 1:1 electrolytes.^[24] The Co-complex magnetic moment measurements show μ_{eff} value in 4.41 B.M. This value is characteristically

Table 2: FTIR spectral data (wave number) cm^{-1} of ligand and its complexes

Compounds	$\nu(\text{C-H})$; aromatic	$\nu(\text{C=C})$; aromatic	$\nu(\text{C=N})$	$\nu(\text{C-N})$	$\nu(\text{C-Cl})$	$\nu(\text{M-N})$
(L)	3064	1595	1674	1272	684	-
$[\text{Co}(\text{L})_2(\text{Cl})_2]$	3062	1595	1656	1276	688	414
$[\text{Cu}(\text{L})_2(\text{Cl})_2]$	3066	1598	1658	1271	686	432
$[\text{Ni}(\text{L})_2(\text{Cl})_2]$	3060	1598	1662	1278	690	426
$[\text{Au}(\text{L})_2(\text{Cl})_2]\text{Cl}$	3056	1589	1604	1315	687	385

Table 3: Uv-Vis spectra of the ligand and complexes in 10^{-3} M DMSO

The complex	λ (nm)	ϵ (cm^{-1})	$\epsilon_{\text{max}} \cdot \text{Mol}^{-1}\text{Cm}^{-1}$	Assignment	Suggested formula	
(L)	267	37453	1416	$\pi \rightarrow \pi^*$		
	301	33222	1764	$n \rightarrow \pi^*$		
	$[\text{Co}(\text{L})_2(\text{Cl})_2]$	266	37593	1491	L.F	Tetrahedral
		290	34482	1763	L.F	
		445	22471	39	C.T overlap	
$[\text{Ni}(\text{L})_2(\text{Cl})_2]$	671	14903	23	${}^4\text{A}_2(\text{F}) \rightarrow {}^4\text{T}_1(\text{P})$	Tetrahedral	
	267	37453	1495	${}^4\text{A}_2(\text{F}) \rightarrow {}^4\text{T}_1(\text{F})$		
	301	33222	2250	L.F		
	410	24390	61	C.T		
	800	12500	52	${}^3\text{T}_1(\text{F}) \rightarrow {}^3\text{T}_1(\text{P})$		
	906	11037	55	${}^3\text{T}_1(\text{F}) \rightarrow {}^3\text{A}_2(\text{F})$		
$[\text{Cu}(\text{L})_2(\text{Cl})_2]$	963	10384	55	${}^3\text{T}_1(\text{F}) \rightarrow {}^3\text{T}_2(\text{F})$	Tetrahedral	
	267	37453	1316	L.F		
	301	33222	1864	L.F		
	399	25062	38	C.T		
$[\text{Au}(\text{L})_2(\text{Cl})_2]\text{Cl}$	550	18181	24	${}^2\text{T}_2 \rightarrow {}^2\text{E}$	Square-planar	
	266	37593	1499	L.F		
	300	33333	2301	L.F		
	410	24390	61	C.T overlap		
	671	14903	23	${}^1\text{A}_1\text{g}(\text{F}) \rightarrow {}^1\text{B}_1\text{g}(\text{F})$		
				${}^1\text{A}_1\text{g}(\text{F}) \rightarrow {}^1\text{A}_2\text{g}(\text{F})$		

assigned in Co^{II} complex to tetrahedral geometries. Ni-complex gave 3.79 B.M. values. The m_{eff} value is included in the tetrahedral array for this complex. The Cu-complex magnetic moment measurements show a value in 1.95 B.M, which can be assigned to the tetrahedral geometry around Cu^{II} ion. The Au^{III} complex is diamagnetic which can be attributed to the square planar geometry.

CONCLUSION

The ligand complexes are prepared from the reaction of two ligand (*Z*)-1-(4-(3-chlorobenzylidene) amino) phenyl ethan-1-one equivalents with one mole of metal salt as the central ion. Physicochemical and spectroscopic methods were put in place to confirm bonding mode and over all complex structure. These results lead to the preparation of four coordinate complexes, and we suggested the structure of the complexes from the above discussions is tetrahedral except Au complex which is square planar geometry.

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