

# A SCHIFF BASE DERIVATION FROM 1,2-DIAMINO BENZENE AND CEPHA CLOR AS WELL AS ITS TRANSITION METAL COMPLEXES, WERE SYNTHESIS, MAGNETIC STUDIES AND SPECTROSCOPIC STUDIES

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## ABSTRACT

M(II) co-ordination complexes containing Mn(II), Fe(II), Co(II), Ni(II), Cu (II), and Zn (II), were synthesised and described using a Schiff base obtained from addition of 1,2-diaminobenzene with cephaclor antibiotic. The Cl<sup>-</sup> ions Coordinate to M ions, according to analytic and molar conductivity values. The generic equations  $[M(L)Cl(H_2O)]$  (M(II) = Mn(II), Fe(II), Co(II), Ni(II), Cu (II), and Zn(II), are provided for complexes based on these research. The ligand HL acts as a cleaving substance for mono-anionic tetra-dentate NNNO.

**Keywords:** Complexes of Schiff base metals, Magnetic research, Spectroscopy research and Synthesis.

## INTRODUCTION

The condensation of carbonyl with primary amine to form Schiff base material with an amine set (-RC=N-). Schiff bases are types of a synthetic particle have a wide range of biological properties. Medicinal field are presently interest in developing a new Schiff base chemotherapy<sup>1</sup>. The biological properties of Schiff bases have been studied extensively, including, antibacterial<sup>2</sup>, anticancer<sup>3</sup> antifungal, and herbicidal properties. Schiff bases generate from carbonyl and amine variants have been shown to have genotoxic, antibacterial<sup>4</sup>, and antifungal nature<sup>5</sup>.

High stability under a variety of oxidation / reduction climates, as well as fact imine ligands are borderline with both soft and hard Lewis bases, Schiff bases have play, over the years, they've playin main group and transition metal (T-M) co-ordination science, they have a distinctive function as chelating ligands<sup>6</sup>, because of enormous diversity of potential ligand configurations depending on amine and aldehyde employed, the study of Schiff base

complexes is rapidly evolving. Transition-metal compounds with a Schiff base ligand are gaining popularity due to well-known science and biological properties, catalytic and medicinal uses<sup>7</sup>. We present isolation and characterisation of M (II) compounds a Schiff base ligand derive from addition of 1,2-diaminobenzene and cephaclor antibiotic, a first-generation cephalosporin, in order to see if complex formation affects the ligand's medicinal uses and to learn more about antibiotic action<sup>8</sup>. The chemical composition of cephaclor is seen in Figure 1.

## EXPERIMENTAL

### MATERIALS AND PROCEDURES

During the syntheses and handling of compounds, all required measures were followed to keep oxygen and moisture out. For all tests, analytically grade substances were use as receive, with a Perkin-Elmer Series 2000 equipment, FTIR spectrum of metal and ligand as potassium bromide pellets were acquire in spectral spectrum 4005-4025 cm<sup>-1</sup>. A Bruker IFS 66V spectro-photo-meter was

use to record FT-IR spectra of polyethylene pellets between 460 and 130  $\text{cm}^{-1}$ . A Bruker ECS 106 spectrometer operating in X-band as use to record EPR spectra (9.76 GHz). The g marker was the DPPH free radical. A Cary Recording Spectro-photometer Model 17D as use to measure d-d transitions in vis. and near IR areas, while a Perkin-Elmer spectrophotometer as utilize to record visible and ultraviolet regions. A LECO CHNS 932 model micro-analytical organ was use to examine contents of C, H, N, and S, after breakdown with a combination of  $\text{HNO}_3$  and HCl followed by  $\text{H}_2\text{SO}_4$ , the compounds were examined by using a Perkin-Elmer atomic absorption analyzer, determine the metal content. The chlorine content of the solid compound (30 mg) was evaluated by burning it in presence of an oxygen flask of a  $\text{KOH-H}_2\text{O}_2$  solution. The halide concentration as measure by using diphenylcarbazone as indicator and a normal  $\text{Hg}(\text{NO}_3)_2$  fix. Magnet properties as measure at RT by using  $\text{HgCo}(\text{NCS})_4$  as a on a Johnson calibrant Magnetic Matthey Susceptible proportion, in DMSO,  $^1\text{H}$  NMR spectra as record at 100 MHz on a Varian instrument versus such an internal reference of tetramethylsilane (TMS). Thermo-grams as acquire on a Perkin Elmer STA-6000 simultaneous thermal analyzer at a rate of heat of 4  $^\circ\text{C}/\text{min}$  to 210  $^\circ\text{C}$ .

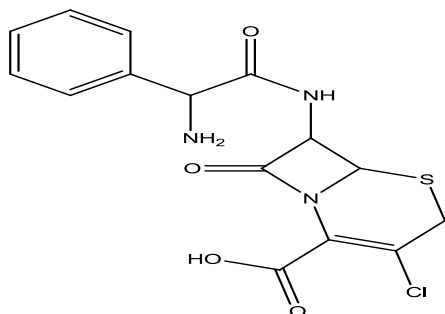


Fig. 1. Chemical structure of cephaclor.

### Synthesis of Schiff- base ligand (HL)

1 mmol of 1,2-diaminobenzene was added to 1 mmol of cephaclor in (100) mL of boiling  $\text{C}_2\text{H}_5\text{OH}$ , the solution as reflux for 25 minutes at 50-60 $^\circ\text{C}$  in a nitrogen environment, yielding a dark yellow Ppt. This substance as filterer out and wash in ether and ethanol before being under moist air to dry, purification was achieved by re-crystallization from the same solvent (yield 75%), at a temperature of 165-166  $^\circ\text{C}$ .

### Made of Schiff base compounds

The same method as use to make  $\text{Mn}(\text{II})$ ,  $\text{Fe}(\text{II})$ ,  $\text{Co}(\text{II})$ ,  $\text{Ni}(\text{II})$ ,  $\text{Cu}$ , and  $\text{Zn}$ , compounds. A fix of 1 mmol of HL in 15 mL of  $\text{CH}_3\text{OH}$  was gently add with burning to a hot fix of 1 mmol of suitable  $\text{MCl}_2$  M-salt in 25 mL of  $\text{CH}_3\text{OH}$ . The crystalline solids of HL's chloride metal(II) materials were isolate from reaction mixture and wash multiple times with  $\text{CH}_3\text{OH}$  and ether before being dry under reduce pressure at RT (yield 35-40%), m.p. > 240  $^\circ\text{C}$ .

### DISCUSSION AND RESULTS

The ligand and  $\text{M}(\text{II})$  compounds as purify from  $\text{CH}_3\text{OH}$  in high yields and come in a variety of hues, the ligand and  $\text{Mn}(\text{II})$  compound are yellow, whereas  $\text{Mn}(\text{II})$ ,  $\text{Fe}(\text{II})$ ,  $\text{Co}(\text{II})$ ,  $\text{Ni}(\text{II})$ , ( $\text{Cu}$ , and  $\text{Zn}$ , complexes are red wine and grey, respectively. When heated to 250 $^\circ\text{C}$ , none of the compounds melted or decomposed. Scheme.1 shows the HL ligand's synthesis pathway. Table.1 shows analyses of ligand and compounds at atomic level, which show that all of the compounds correspond well with a 1:1:1:1 M: HL: chloride: coordinate water stoichiometry. The materials are hydrophobic in nature and additional organic solvents such as  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ ,  $\text{C}_2\text{H}_5\text{OH}$ ,  $\text{C}_3\text{H}_6\text{O}$ , DMF, acetonitrile,  $\text{CH}_2\text{Cl}_2$ , and ether, however DMSO is soluble. Thermo-grams of hydrate transition metals show distortions endothermic in 165-173  $^\circ\text{C}$  range due to loss of one water molecule, as well as, fact that compounds are stable in the absence of hydration water and solvent particles, under aforementioned circumstances, attempts to form complexes with  $\text{Co}(\text{II})$ ,  $\text{Cu}(\text{II})$ , and  $\text{Zn}(\text{II})$  ions with a well-define stoichiometry were unsuccessful<sup>9</sup>. The non-electrolytic character of chloro compounds is revealed by conductance molar values obtain in DMSO at ambient temperature, which range from 12.5 to 14.5  $\text{S cm}^2 \text{mol}^{-1}$ , implying that Cl<sup>-</sup> is coordinate to  $\text{M}(\text{II})$  ions (Table 1 & 2).

### Schiff base ( $^1\text{H}$ NMR)

The chemical shifts found were same to those reported in the literature for Schiff base ligands<sup>10</sup>. One peak attributable to the  $\text{Ph-CH-C=O}$ ,  $\text{Ph-NH}_2$ , and  $\text{CO-OH}$  sets occurred at 2.48, 3.45, and 9.95 ppm, approximately, in  $^1\text{H}$  NMR spectra of Schiff base L.CO-CH and CH-S on -lactam ring, as well as NH, produced three sets of double peaks at 6.65, 7.15, and 6.86 ppm, respectively. In the 3.27-3.39 ppm range, one set of four signs of resonance compatible with an AB network assign to S- $\text{CH}_2$  on di-hydro-thiazine ring as found, with a coupling constant of 16.5 Hz for JAB. Furthermore, there was no discernible connection between  $\text{NH}_2$  and the surrounding

CH, and at 6.00 ppm, there was a wide single signal owing to NH<sub>2</sub> protons. The presence of phenyl ring protons caused a multiplet in range 6.53–7.58 ppm (m, 9H, CH aromatic). The <sup>1</sup>H-NMR spectra of the compounds could not be produced since all of them are paramagnetic.

### IR spectra

Table 2 shows primary IR spectral bands of ligand and transition metals. In spectra of cephaclor, lactam  $\nu_{(C=O)}$  band appears at 1765 cm<sup>-1</sup>. There are no absorption bands in IR spectra of Schiff base ligand that may be ascribed to lactam  $\nu_{(C=O)}$  vibration zone originating from cephaclor. The lack of such absorbing, in addition to development of a new peak at 1632 cm<sup>-1</sup> attributable to  $\nu_{(C=N)}$  vibration, suggests that product is (Sb)<sup>11,12</sup>. The metal compounds' infrared spectra show IR absorption bands in 1617–1625 cm<sup>-1</sup>, which may be attributed to founder ligand's C=N extending frequency (HL). On complexation, the band shifts towards lower wave numbers, indicating that azomethane N is founder to metal centre. The Schiff base's terminal NH<sub>2</sub> sets produce N-H tremors at 3350 and 3230 cm<sup>-1</sup>. The amine sets are present in complexes, and founder is also accomplished through NH<sub>2</sub> sets, as seen by shift in N-H bending and stretching bands. In addition, the loss of stretching attributed to  $\nu_{(COOH)}$  in ligand at 1715 cm<sup>-1</sup>, as well as the development in compounds of new  $\nu_{asy}$  and  $\nu_{sy}$  dimensions of (-COO<sup>-</sup>) set at 1615–1583 and 1385–1383 cm<sup>-1</sup>. These results, together with the  $\Delta\nu$  value ( $\nu_{Asy} - \nu_{sy}$ ) > 210 cm<sup>-1</sup>, the carboxylate group is monodentately coordinated to the metal atoms. As a result of coordination, the remaining carboxylate bands,  $\omega(COO)$ ,  $\rho(COO)$ ,  $\nu_{sym}(COO)$ ,  $\gamma(COO)$ , which were formerly at 613, 535 cm<sup>-1</sup>, 1415, and 786, shift as well. Moreover, the emergence of new lines in the 455–495 cm<sup>-1</sup> regions in spectra of compounds (missing in free ligand) suggest that C=N and NH<sub>2</sub> moiety might be bound to M-ion through N particle.  $\nu_{(M-O)}$  vibrations are provisionally ascribed to bands in 355–405 cm<sup>-1</sup> area detected in compounds but lacking in free Schiff base ligand. Bands in 1425–1463, 1073–1105, and 723–746 cm<sup>-1</sup> ranges may be attributed to phenyl ring tremors in [M(L)Cl(H<sub>2</sub>O)] compounds. Aliphatic  $\nu_{(C-H)}$  stretches may be found in 2832–2955 cm<sup>-1</sup> range, whereas aromatic  $\nu_{(C-H)}$  stretches can be found in 3005–3110 cm<sup>-1</sup> region. These findings imply that coordination is mediated by azomethine-N, two amino sets, and

carboxylate-O set, and Schiff base acts as a tetra-dentate ligand mono-anionic NNNO cleaving substance<sup>13-20</sup>.

### Magnetic properties

Using Pascal's constants, corrected magnetic properties as derive by using molar magnetic susceptibility values. The paramagnetic complexes' magnetic moments are within ranges associate high spin ions with no spin in octahedron fields, implying mononuclear species. The magnetic moment of Fe(II) compound is 4.82  $\mu_B$ , which is characteristic of d<sup>6</sup> setups with high spin with 4 un-paired electrons and a ground level of S = 2. When compared to report values for octahedron material of Co (II) (4.72–5.23  $\mu_B$ ), the Co (II) compound has a moment of attraction of 5.04  $\mu_B$ , which is indicative of a d<sup>7</sup> mechanism with three un-paired electrons suggesting a condition of quartet in an octahedron configuration around metal<sup>21</sup>. The magnetic moment of nickel(II) compound is 3.18  $\mu_B$ , which is higher than spin-only value, because of orbital contribution coming from movement of electron from d<sub>x<sup>2</sup>-y<sup>2</sup></sub> orbital to d<sub>xy</sub> orbital. As a result, compound's octahedral shape is most likely deformed.

The magnet's effective magnetic moment [Mn(L)Cl(H<sub>2</sub>O)] compound in condition of matter is  $\mu_{eff} = 3.35 \mu_B$  at room temperature. This value represents the [Mn(L)Cl(H<sub>2</sub>O)] intermediate-spin compound with three un-paired electrons in molecule coming from ground state design of (a<sub>1g</sub>)<sup>2</sup> (E<sub>g</sub>)<sup>2</sup> (b<sub>2g</sub>)<sup>1</sup>. This number is, though, somewhat less than 3.87  $\mu_B$  predicted magnetic moment with simply a spin for S = 3/2, because [Mn(L)Cl(H<sub>2</sub>O)] compound is relatively airborne stability and contains a few di-nuclear material Mn(III) -oxo-complex, [Mn(L)Cl(H<sub>2</sub>O)]<sub>2</sub>O, could be present, less value of  $\mu_{eff}$  than calculate maybe as a result of fact that [Mn(L)Cl(H<sub>2</sub>O)] a good example use for magnetic properties as not completely pure. Due to perfect spin pairing, this di-nuclear Mn(III) -oxo-compound with four electrons on Mn(III) cation shows dia-magnetic property, which is rare for this ion but feasible under two conditions: Anti-ferromagnetic connection between oxygen bridge Mn(III) ions, which are common for -oxo-compounds of Mn(III), and (II) electron pairing on d<sub>xz</sub>, d<sub>yz</sub> orbitals, which are lowest position of Mn(III) d-orbital's, as propose<sup>22</sup>.

Figure. 2 shows RT X-Ray EPR spectra of a sample of powder of [Mn(L)Cl(H<sub>2</sub>O)] record at ambient heat. An overlapping six-line Mn hyper-fine pattern centred at g = 2.017, A = 70 G as seen in room temperature spectrum, which is predicted of an odd un-paired electron system l = ±5/2, ml = ±5/2, ±3/2, ±1/2) and (S

$\pm 5/2$ ,  $mS = \pm 5/2, \pm 3/2, \pm 1/2$ . As a result of allowed transitions ( $mS = \pm 1, m_l = \pm 0$ ), The computed g-factor value of roughly 2 is typical for a 6S state ion like Mn(II) or Fe(III) ( $S = \pm 5/2$ ) and corresponds well with prior outcomes for Mn(II) in various six co-ordinate shapes<sup>23</sup>.

Divalent mononuclear manganese EPR spectra frequently exhibit splitting into six atomic ingredients spaced by roughly 70–100 G. The six hyperfine components overlap if the inherent line width is greater than the splitting, resulting in a single (over 800 G) EPR line. The line width ( $H = 425$  G) in this scenario is twice as narrow as that value at ambient temperature. This rule out isolated paramagnetic manganese(II) ions as the source of the observed signal. It's probable that the magnetic exchange interactions are significantly coupling the manganese (II) centres (ferromagnetic or antiferromagnetic). The hyperfine splitting might be suppressed by large exchange narrowing effects<sup>24,25</sup>. The powder EPR spectra acquired at liquid nitrogen temperature shows no difference from the spectrum recorded at ambient temperature. The dipolar and transfer connection between Mn ions should explain why the line width does not vary greatly with lowering temperature.

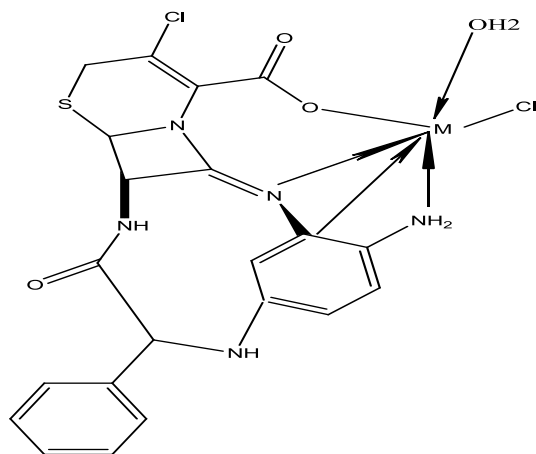


Fig. 3. Proposed structure of metal complexes.

### Electronic spectra

To ensure the consistency of novel compounds in DMSO remedy, a long-term UV–Vis research was conducted. In comparison to ligands, it's worth noting that the absorption wavelengths of novel

compounds didn't change much for up to a month, indicating that they were stable in DMSO remedy. In  $10^{-3}$  M DMSO remedy, the electronic spectrum of (Sb) as well as compounds revealed two wide bands at 263–285 nm and 300–330 nm. The absorption of ( $1Lb \rightarrow 1A1$ ) of benzene ring is ascribed to the first band, whereas second band is assign to azomethine group's  $\pi-\pi^*$  ( $C=N$ -) transition, which is move from high to lower wavelength on co-ordination<sup>26,27</sup>.

The absorptions at 405–805nm are due to L to Mfee transmission and  $d \rightarrow d$  variations bands of metal in compounds, according to spectra of metal compounds, due to the concurrence of fee transmission,  $d \rightarrow \pi^*$ ,  $L \rightarrow M$ , and intra-ligand  $n \rightarrow \pi^*$  variations, the Mn(II) compound has a weak absorption at 363nm. A wide band at 420 nm can be ascribed to  $5T2g \rightarrow 5Eg$  transition in electronic spectra of Fe(II) compound<sup>28</sup>.

Two more bands at 423 and 465 nm in the visible area spectra of the cobalt(II) complex are attribute to ligand-metal charge and  $4T1(F) \rightarrow 4T2g$  (F) transition, implying octahedron stereo-chemistry around the M ion<sup>31</sup>. The UV–Vis spectra of Ni(II) material shows two large absorption at 483 and 405 nm due to  $d-d$  bands, so that might be ascribe to changes  $3A2g \rightarrow 3T1g$  (F) and  $3A2g \rightarrow 3T1g$  (P) correspondingly, given that the metal's immediate coordination sphere has Oh symmetry. The  $3A2g \rightarrow 3T2g$  changes would occur near infrared<sup>29-32</sup>.

### Coordination sites

There are various possible donor atoms in Schiff base HL in cephaclor that might be engaged in coordination with M ions. The idea that Schiff base ligand's coordination arises due to carboxylate,  $C=N$ , and  $NH_2$  section suggests that it behaves as a tetra-dentate ligand. The M ions in  $[M(L)Cl(H_2O)]$  material including one coordinate hexacoordinates are formed by chloride anion and one  $H_2O$  particle, and octahedral geometries are likely to be deformed (Fig. 3). We attempt to develop metal chelation single crystals, but due to insolubility in organic solvents. These findings will aid future crystallographic investigations, which are hindered by difficulty of producing X-ray grade crystals of cephalosporin compounds.

Table 1: Elemental analyses for M-compounds

Comp.	Found (Calc.)					
	C	N	H	S	Cl	M
[(HL)]	56.1	14.2	5.7	7.3	6.9	--
(C <sub>21</sub> H <sub>20</sub> N <sub>5</sub> O <sub>3</sub> SCI)]	(54.1)	(16.3)	(4.4)	(7.0)	(7.7)	---
[Mn(L)Cl(H <sub>2</sub> O)]	45.3	13.5	3.3	4.8	12.4	10.10
[Mn(C <sub>21</sub> H <sub>21</sub> N <sub>5</sub> O <sub>4</sub> SCI <sub>2</sub> )]	(43.6)	(11.4)	(4.7)	(6.7)	(12.5)	(9.7)
[Co(L)Cl(H <sub>2</sub> O)]	43.5	11.3	4.6	6.2	14.7	10.2
[Co(C <sub>21</sub> H <sub>21</sub> N <sub>5</sub> O <sub>4</sub> SCI <sub>2</sub> )]	(43.3)	(11.3)	(4.7)	(6.6)	(12.5)	(10.4)
[Fe(L)Cl(H <sub>2</sub> O)]	43.8	11.5	4.8	6.3	12.6	9.4
[Fe(C <sub>21</sub> H <sub>21</sub> N <sub>5</sub> O <sub>4</sub> SCI <sub>2</sub> )]	(43.5)	(11.4)	(4.7)	(6.7)	(12.5)	(9.9)
[Ni(L)Cl(H <sub>2</sub> O)]	43.8	11.3	4.4	6.9	12.6	10.7
[Ni(C <sub>21</sub> H <sub>21</sub> N <sub>5</sub> O <sub>4</sub> SCI <sub>2</sub> )]	(43.3)	(11.3)	(4.7)	(6.6)	(12.5)	(10.3)
[Cu(L)Cl(H <sub>2</sub> O)]	43.6	11.3	4.4	6.9	12.6	10.7
[Cu(C <sub>21</sub> H <sub>21</sub> N <sub>5</sub> O <sub>4</sub> SCI <sub>2</sub> )]	(43.3)	(11.3)	(4.8)	(6.8)	(12.7)	(10.5)
[Cu(L)Cl(H <sub>2</sub> O)]	43.6	11.3	4.5	6.9	12.7	10.7
[Cu(C <sub>21</sub> H <sub>21</sub> N <sub>5</sub> O <sub>4</sub> SCI <sub>2</sub> )]	(43.3)	(11.3)	(4.9)	(6.8)	(12.8)	(10.5)

Table 2: Wave numbers of vibration of metal compounds (cm<sup>-1</sup>)

Comp.	$\nu$ C=O	$\nu$ C=O	$\nu$ C=N	$\nu$ COO	$\nu$ COO	$\Delta\nu$
	lactam	amide	imino	Asy	Sym	
Cephaclor	1755	1695	--	---	---	----
[(HL)]	---	1695	1635	--	---	--
[Mn(L)Cl(H <sub>2</sub> O)]	---	1695	1625	1595	1385	215
[Fe(L)Cl(H <sub>2</sub> O)]	---	1695	1625	1590	1385	210
[Co(L)Cl(H <sub>2</sub> O)]	---	1695	1610	1590	1385	210
[Ni(L)Cl(H <sub>2</sub> O)]	---	1695	1625	1595	1385	215
[Cu(L)Cl(H <sub>2</sub> O)]	--	1695	1625	1595	1385	215
[Zn(L)Cl(H <sub>2</sub> O)]	--	1695	1625	1595	1385	215

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