

Synthesis, Characterization and Biological Activity of Some Mixed Ligand Complexes of 1, 10-Phenanthroline and [4-(2-hydroxy-1,2-diphenylethylideneamino)-N-pyrimidin-2-yl] benzene sulfonamide] with Divalent Metal Ions

Rehab K. Al-Shemary¹ ✉, Inam H. Ibrahim², Nibras A. Al-marsomy²

¹ Department of Chemistry, College of Education for Pure Sciences, Ibn -Al-Haitham, University of Baghdad, IRAQ

² Department of Chemical industries, Technology institute, Baghdad, IRAQ

Email: drrehabalshemary@gmail.com

ArticleInfo ABSTRACT

Received
4/2/2016

Accepted
5/6/2016

Schiff base ligand [4-(2-hydroxy-1, 2-diphenylethylideneamino)-N-pyrimidin-2-yl]benzene sulfonamide] (L) was prepared through a condensation reaction sulfadiazine and benzoin in acidic medium. The prepared ligand has been characterized with different techniques (C.H.N.S, FT-IR, UV-Vis and ¹H&¹N.M.R). Mixed ligand complexes of some divalent metal ions [Co^(II), Mn^(II), Ni^(II), Cu^(II) and Hg^(II)] were prepared by the reaction of [4-(2-hydroxy-diphenylethylidene amino)-N-pyrimidin-2-yl]benzene sulfonamide] (L) and 1, 10-Phenanthroline with the metal ions in basic conditions. The prepared complexes were characterized and their astrochemical structures and geometries were suggested depending upon data of (UV-Vis, FT-IR, atomic absorption, micro elemental analysis). The following general formula was achieved : [M(PHN)(L)₂], where M represents (Co^(II)- Cu^(II)-Mn^(II)- Ni^(II) and Hg^(II)). Electronic spectra supported by magnetic moment's revealed octahedral geometries for all the prepared complexes. The mixed ligand complexes showed resistivity to different bacteria such as (*Pseudomonas aeruginosa*), (*Bacillus subtilis*), (*Escherichia coli*), and (*staphylococcus aureus*).

Keywords: Schiff base, benzoin, Characterization, Mixed ligand complexes.

الخلاصة

Do not write
anything here

حضرت قاعدة شيف الجديدة [4-(2-hydroxy-1,2-diphenyl ethylidene amino) -N-pyrimidin-2-yl] benzene sulfonamide] (L) من خلال تفاعل تكاثف السلفاديازين مع البنزوين في وسط حامضي. حيث جرى تشخيص الليكاند (L) بمختلف التقنيات من (طيف الأشعة فوق البنفسجية، طيف الأشعة تحت الحمراء، طرق تحليل العناصر وطيف الرنين النووي المغناطيسي البروتوني والكربوني) تم تحضير معقدات الليكاندات المختلطة من تفاعل الليكاند (L) [4-(2-hydroxy-1,2-diphenyl ethylidene amino) -N-pyrimidin-2-yl] benzene sulfonamide] مع (1,10-Phenanthroline) مع الأيونات الفلزية ثنائية التكافؤ [Co^(II), Mn^(II), Ni^(II), Cu^(II) and Hg^(II)] في ظروف قاعدية. الليكاندات المختلطة المخلقة تم تشخيصها باستعمال تقنيات مختلفة (طيف الأشعة تحت الحمراء ، طيف الأشعة فوق البنفسجية والمرئية و التحليل الدقيق للعناصر ، التحليل الكمي للفلزات، قياسات الحساسية المغناطيسية ، محتوى الكلور والتوصيلية الكهربائية. واستنادا الى لنتائج هذه التشخيصات التي بينته ان الأشكال الهندسية هي ثمانية السطوح. تم تقويم الفعالية المضادة للبكتريا لليكاند[ومعقداته المختلطة للبكتريا (*Escherichia coli*), (*staphylococcus aureus*), (*Bacillus subtilis*), (*Pseudomonas aeruginosa*)

INTRODUCTION

In recent years, there has been renewed interest in the synthesis and study of mixed ligand transition metal complexes [1]. The utility aspects of these complexes have received their share of attention as these have found applications in diverse fields [2]. Schiff bases compose a significant class of organic compounds in chemistry and undergo to huge number of reactions due to their advantageous chemical and physical characteristics [3]. They are also having big industrial applications and very entertaining pharmacological, biological activities [4]. Schiff bases obtained from aromatic aldehydes and substituted aliphatic amines have a broad assortment of applications in numerous domains, e.g. analytical, inorganic biological and chemistry [5]. Schiff bases and their metal complexes are known to exhibit anticancer, antioxidant and anti-inflammatory activities [6]. It has been reported many important biologically Schiff bases in literature possess anti-inflammatory, antimicrobial, anti-tumor and anxiolytic properties [7]. Preparation and diagnosis of transition metal complexes including ligands such as Schiff bases have been used by investigators on account of their prominence as catalysts in plentiful reactions [8]. These ligand systems have excited great advantage because of their interest as model compounds in bio inorganic works [9]. Large transition metal complexes having nitrogen and oxygen donor atoms of the Schiff bases own special arrangement, sensitive to the molecular environment and structural liability [10]. In few Schiff base complexes, small referents in the design of the ligands including donor/acceptor atoms (O and N) noticeable influence the reactivity of the compounds [11]. Result to the industrial importance of mixed ligand complexes which have been used such as an activity catalyst in some reactions contain oxidative hydrolysis of olefins hydrogenation and carboxylation of methanol hydroxylation [12]. These complexes have also shown catalytic activity in various oxidation reactions of environmental and biological importance [13].

In this paper we focus on the synthesis of mixed ligand complexes of $\text{Co}^{(II)}$, $\text{Mn}^{(II)}$, $\text{Ni}^{(II)}$, $\text{Cu}^{(II)}$ and $\text{Hg}^{(II)}$ complexes with bidentate Schiff base [4-(2-hydroxy-1,2-diphenylethylideneamino)-N-pyrimidin-2-yl]benzenesulfonamide] ligand and 1,10-Phenanthroline (PHN), as well as to study the biological activity of the prepared Schiff base and their mixed ligand complexes.

MATERIALS AND METHODS

The following compounds were available commercially and used without further purification: benzoic acid (98 %, Merck Co.), sulfadiazine (97 %, Merck Co.), 1,10-phenanthroline (99%, Merck Co.), hydrochloric acid HCl (99.5%, B.D.H Co.), Cobalt (II) chloride hexahydrate $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ (99%, Merck Co.), Copper(II) chloride dihydrate $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (98%, B.D.H Co.), Manganese (II) chloride tetrahydrate $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ (98%, B.D.H Co.), Nickel (II) chloride hexahydrate (98%, B.D.H Co.), Mercury (II) chloride HgCl_2 (98%, B.D.H Co.), Potassium hydroxide (solid) KOH (98%, Fluka Co.). The solvents: Acetone (98%, Fluka Co.), Ethanol absolute (99.8%, GCC Co.), DMSO (98%, Fluka Co.) and DMF (99%, Fluka Co.).

Methods:

^{13}C -NMR and ^1H spectra of the compounds were registered using spectropin (Bruker) ultra-shield instrument 300 MHz magnets by (TMS) tetramethylsilane as a typical internal standard and (DMSO- d_6) as a solvent. A FT-IR spectrum was recorded on SHIMADZU FTIR-8400 spectrophotometer as KBr disc. Electronic spectra were recorded using U.V-Vis. spectrophotometer type CECIL, England, with quartz cell of (1cm) path length in range (200-1000) nm in 10^{-3}M solution of ethanol at room temperature. Chloride content for the prepared complexes was determined using potentiometric titration method on (686-Titro Processor-665 Dosim A-Metrohm/Swiss). Magnetic susceptibility measurements were obtained using (BM6) Bruker at (298)°K. The Microelemental (C, H, and N %) analysis for prepared compounds obtained on Perkin Elmer by (CHN Analyzer) 2400 series II.

Study of Biological Activity:

Four bacteria chosen type were applied included (*Escherichia coli*) and (*Staphylococcus aureus*), as gram (-) bacteria, (*Bacillus subtilis*) and (*Pseudomonas aeruginosa*) as gram (+) bacteria nutrient as agar medium, and applied a solvent and control (DMSO). All the compounds have concentration about (10^{-3} M) of disc sensibility test exposure. These processes implicated the zone inhibition exposure of diffusion (micro-organism) on plate agar. The plate incubated (24hr.) at (37) °C.

Synthesis of Schiff base ligand (L):

The solution of benzoin (0.21 gram, 0.1m mole) in methanol (10 ml), added a solution of Sulfadiazine (0.25 gm, 1mmole) with continuous stirring, (2 drops of con. HCl was added) [14]. The reaction mixture was heated under reflux for about 4-5 hours. Then, the volume of reaction mixture was reduced by slow evaporation at room temperature. The

isolated compound was purified by recrystallization from diethyl ether to get a yellow product of the ligand, Yield: 0.15gm, 70% Mp: 230°C. (Figure1).

Synthesis of the mixed-ligand [L] and 1,10-phenanthroline complexes with some metal ions:

To a solution of metal salt (0.001mmol) in ethanol 15ml, was added a solution of Schiff base ligand (0.89 g, 0.002mmole) in (12) ml ethanol. The pH of the solution was adjusted to 6-8 using (15%) ethanolic solution of KOH. Finally a solution of 1,10-phenanthroline (0.18 g, 0.001 mmole) in (7) ml ethanol was also added, by reflux in (1) h. Then filtered mixture and the washed precipitate with ethanol an amount excess and recrystallized from acetone solvent. (see Figure 2).

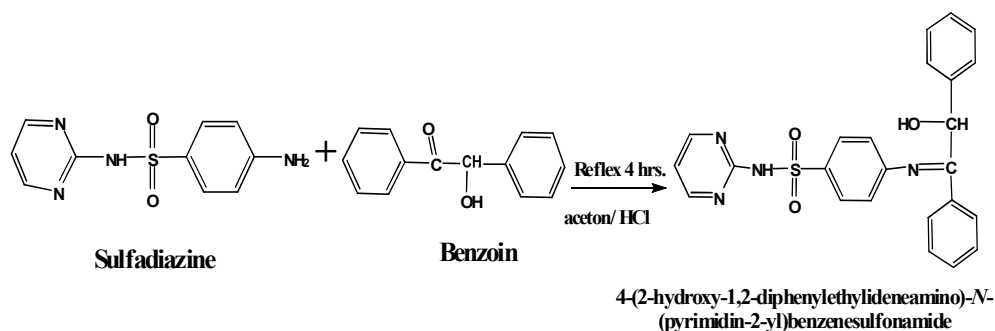


Figure 1: Synthesis of Schiff base ligand

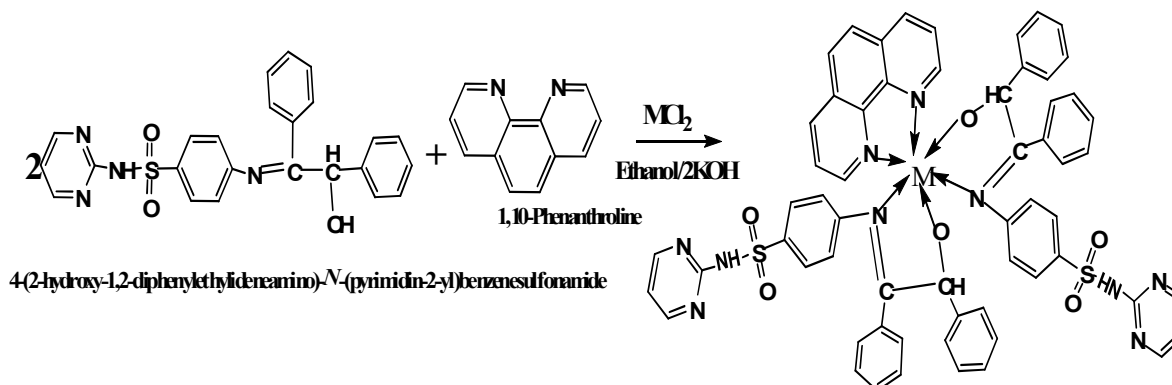


Figure 2: Synthesis diagram for the preparation of complexes

RESULTS AND DISCUSSION

Characterization of Mixed Ligand Complexes:

The complexes Mostly, were synthesized by reacting ligands to metal salts with the using (1:2:1) mole ratio, i.e. one metal chloride mole: two moles L and one mole 1,10-phenanthroline

(PHN). The solubility tests of the prepared complexes in various solvents showed that all complexes are soluble in acetone, (DMF and DMSO) while they are insoluble in water and common organic solvents. The experimental and calculated values of proportion metal complexes are in fair approval as shown in Table (1). The AgNO_3 test for (chloride ion) with (Nil %) mean solution was negative that pointed to there is no chloride ion outside the coordination sphere of the central metal [2]. Molar conductance (Λ_m) of 10^{-3}M solutions of the complexes in DMSO lie in very low range (5.1-14) $\text{ohm}^{-1}\text{cm}^2\text{mol}^{-1}$ supporting their non-electrolytic behavior [1].

NMR spectrum for Schiff base ligand (L):

The ^1H and ^{13}C NMR, spectra of Schiff base ligand [L] are consistent with structure. In the ^1H NMR spectrum, shown in [Fig 3], the singlet signal was observed at ($\delta=2.07\text{ppm}$) is assigned to the proton of O-H group. The singlet signal at ($\delta=3.3\text{ ppm}$) refers to the N-CH₃ proton. The spectrum also showed the singlet signal at ($\delta=4.1\text{ ppm}$) which can be assigned to (NH) proton of enamine group [2]. The multiple signal were observed at ($\delta=6.3-8.3\text{ppm}$) are assigned to protons of aromatic rings.

In the ^{13}C NMR spectra, shown in [Figure 4], the singlet signal was observed at ($\delta=78.29\text{ ppm}$) is assigned to the carbon of C-OH group [3]. The multiple signal were observed at the range ($\delta=115.18-158.79\text{ppm}$) are assigned to carbons of aromatic rings. The signal at ($\delta=164.43\text{ ppm}$) refers to the N=C carbon of azomethine group [4]. The signal was observed at ($\delta=169.16\text{ ppm}$) is assigned to the carbon of N=C-N group in pyridine ring.

Infrared Spectral Analysis:

The FT-IR spectrum for the ligand (L), [fig 3] displayed absorption at $(3350)\text{ cm}^{-1}$ due to stretching vibration (O-H) group. A band sharp at $(3257)\text{ cm}^{-1}$ is attributed to $\nu(\text{N-H})$ stretching vibration [9]. Bands at $(3035)\text{ cm}^{-1}$ and $(2935)\text{ cm}^{-1}$ were referred to aromatic and aliphatic stretching vibrations (C-H) groups, respectively. The band strong at $(1651)\text{ cm}^{-1}$ referred to azomethine $\nu(\text{C=N})$ stretching vibration [5]. The absorption at $(1575)\text{ cm}^{-1}$

was referred to the (C=C) stretching vibration. The assignments of bands properties are listed in [Table 2].

FT-IR spectrum for 1,10-phenanthroline

The spectrum of 1, 10-phenanthroline [fig 4] appeared the absorption at $(1620)\text{cm}^{-1}$ referred to $\nu(\text{C=N})$ stretching vibration. Also the band at $(1549)\text{ cm}^{-1}$ was referred to the stretching vibration for (C=C) group [6]

FT-IR spectrum for the complexes

The binding style used to study the of 1,10-phenanthroline and the prepared Schiff base ligand to metal ions, a comprised with FT-IR spectra for ligands and mixed ligand-complexes prepared. The ligand IR spectrum offered wide band at (3350 cm^{-1}) , which was due to the $\nu(\text{OH})$ stretching vibration [7]. This absorption was missing in all synthesized complexes spectra, that signaled participation oxygen of the enol in coordination and deprotonation [8]. The (FT-IR) spectrum for ligand (L) exhibited stretching vibration band at $(1651)\text{ cm}^{-1}$ due to of azomethine group $\nu(\text{C=N})$. this absorption has been shifted to lower frequencies $(1628), (1636), (1622), (1638)$ and $(1626)\text{ cm}^{-1}$ in the spectra of the Ni(II), Co(II), Cu(II), Mn(II) [Figure. 5] and Hg(II) complexes respectively [9]. This shift can be attributed to the coordination of nitrogen atom of the azomethine group to metal atom. The spectrum of 1,10-phenanthroline exhibit band at $(1620)\text{cm}^{-1}$ due to the stretching vibration of azomethine group $\nu(\text{C=N})$ which has been shifted to lower frequencies $(1607), (1610), (1612), (1610)$ and $(1608)\text{cm}^{-1}$ for complexes (Co(II), Ni(II), Cu(II), Mn(II), and Hg(II)) complexes respectively [10], indicating the coordination through nitrogen atom of $\nu(\text{C=N})$ group. The new bands at $(567, 452), (558, 422), (562, 441), (551, 449)$ and $(554, 443)\text{cm}^{-1}$ were referred to $\nu(\text{M-N})$ and $\nu(\text{M-O})$ for complexes $\text{Co}^{(\text{II})}, \text{Ni}^{(\text{II})}, \text{Cu}^{(\text{II})}, \text{Mn}^{(\text{II})}$ and $\text{Hg}^{(\text{II})}$ respectively, signaling that the oxygen and the nitrogen of azomethine group of ligand [L] are implicated in chelation with metal ions together with nitrogen of 1,10-phenanthroline. Additional bands and their assignments can be found in [Table 2].

Magnetic moment and electronic spectra:

The UV-Vis spectral data for the free ligands and all metal complexes are listed in (Table 3). The UV-Vis spectrum of the ligand (L) [Figure 6] showed two peaks at $\{259 \text{ nm}\} \{(38610) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=1982 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ and $\{335 \text{ nm}\} \{(29850) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=687 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ assigned to $\{\pi \rightarrow \pi^*\}$ and $\{n \rightarrow \pi^*\}$ electronic transitions [11]. The 1,10-phenanthroline spectrum, display absorption peaks at $\{202 \text{ nm}\} \{(49504) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=789 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$, $\{228 \text{ nm}\} \{(43859) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=1992 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ which assigned to $\{\pi \rightarrow \pi^*\}$ and peaks at $\{264 \text{ nm}\} \{(37878) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=134 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ which assigned to $\{n \rightarrow \pi^*\}$ transition respectively [12], the data are illustrated in [Table 3]. The (UV-Vis) spectrum of **Co(II)** complex, displayed high five peaks, the first at $\{268 \text{ nm}\} \{(37313) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=1987 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ is due to the ligand field while second peak at $\{347 \text{ nm}\} \{(28818) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=97 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ is due to the charge transfer transition. The third peak at $\{v_3=602 \text{ nm}\} \{(16611) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=231 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$, the fourth at $\{v_2=701 \text{ nm}\} \{(16611) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=112 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ and the fifth $\{v_1=810 \text{ nm}\} \{(14265) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=88 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ which assigned to the (d-d) electronic transition type $\{^4T_{1g(F)} \rightarrow ^4T_{1g(P)}\}$, $\{^4T_{1g(F)} \rightarrow ^4A_{2g(F)}\}$ and $\{^4T_{1g(F)} \rightarrow ^4T_{2g(F)}\}$ respectively suggested "an octahedral" structure around the $\text{(Co}^{II})$ ion [13]

The (UV-Vis) spectrum of **Ni(II)** complex, displayed four peaks, the first absorption at $\{271 \text{ nm}\} \{(36900) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=1250 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ referred to L.F and the second absorption at $\{378 \text{ nm}\} \{(26455) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=1345 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ referred to the C.T transition. The third and fourth peaks at $\{v_2=632 \text{ nm}\} \{(15822) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=168 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ and $\{v_1=803 \text{ nm}\} \{(12453) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=151 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ were referred to the (d-d) electronic transition type $\{^3A_{2g(F)} \rightarrow ^3T_{1g(F)}\}$ and $\{^3A_{2g(F)} \rightarrow ^3T_{2g(F)}\}$ suggested an octahedral structure around the $\text{(Ni}^{II})$ ion [14]. The (U.V-Vis) spectrum of **Cu(II)** complex, displayed three peaks, the first absorption at $\{277 \text{ nm}\} \{(36101) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=1403 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ was referred to L.F

transition and the second absorption at $\{369 \text{ nm}\} \{(37174) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=1118 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ was referred to C.T transition. The third peak at $\{605 \text{ nm}\} \{(16528) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=89 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ was referred to the (d-d) electronic transition type $\{^4E_g \rightarrow ^4T_{2g}\}$ suggested an octahedral structure around the $\text{(Cu}^{II})$ ion [15].

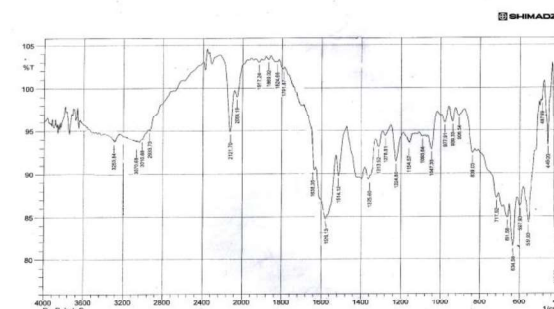
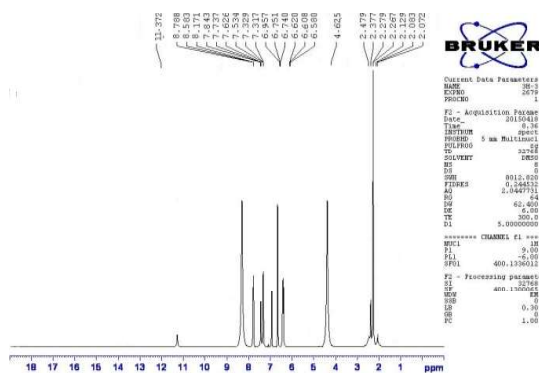
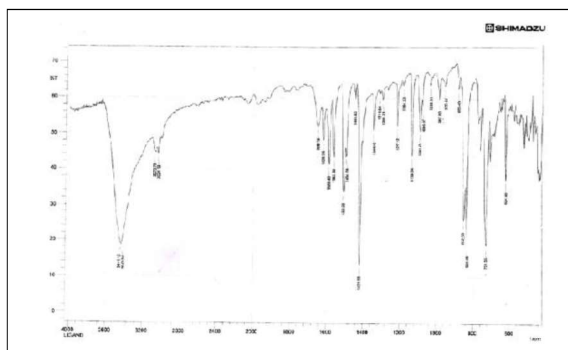
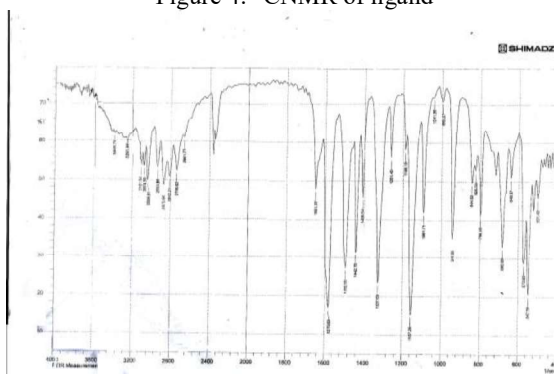
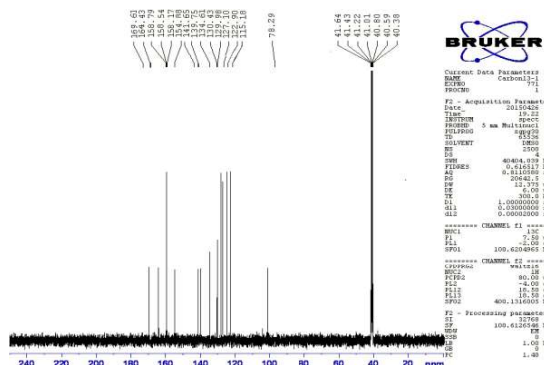
The (UV-Vis) spectrum of **Mn(II)** complex Figure(7) displayed four peaks, first absorption $\{280 \text{ nm}\} \{(35714) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=998 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ was referred to L.F transition and second absorption at $\{360 \text{ nm}\} \{(27777) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=602 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ was due to the C.T transition. These peaks were shifted to lower frequency in comparison with the spectrum of the free ligands. The third and four peaks at $\{v_2=410 \text{ nm}\} \{(24390) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=162 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ and $\{v_1=732 \text{ nm}\} \{(13661) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=82 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ which can be assigned to the (d-d) electronic transitions type $\{^6A_{1g} \rightarrow ^4T_{2g(G)}\}$ and $\{^6A_{1g} \rightarrow ^4T_{1g(G)}\}$ suggested an octahedral structure around the $\text{(Mn}^{II})$ ion [16].

The (UV-Vis) spectrum of **Hg (II)** complex, two peaks displayed, the first peak at $\{285 \text{ nm}\} \{(35087) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=165 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ are referred to L.F transition. This peak was shifted to lower frequency in comparison with the spectrum of the free ligands, while the third peak at second $\{410 \text{ nm}\} \{(3490) \text{ cm}^{-1}\} \{(\epsilon_{\text{max}}=230 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1})\}$ is referred to the C.T transition [17]. Since the metal ion of compounds belongs to (d^{10}) system, these peaks were assigned to C.T transition and suggested an octahedral structure around the (Hg) ions. The effective magnetic moments (Table 3) of the complexes lie in the range (1.83-5.76) BM. These values refer to para magnetic which (high spin) have been for some researched octahedral arrangement. In **Hg (II)** case complex of the magnetic moment ($\mu=0$) is diamagnetic on account of filled d-d orbital [18]. The biological activity: The biological activity of the ligand [L] and their complexes were studied by using inhibition method for four types of pathogenic bacteria: *Staphylococcus aureu* and *Bacillus subtilis* of (gram positive) (*Escherichia coli*) and

(*Pseudomonas aeruginosa*) (gram negative). Both the prepared Schiff base ligand and its mixed ligand complexes are show inhibition diameter against all the types of bacterial, the products signal that the mixed ligand-complexes show more activity than the free Schiff base ligand alone reaction under like experiential statuses, Figure(8) Table (4). This may be due to the chelation reduces considerably [19].

CONCLUSION

Schiff base ligand [4-(2-hydroxy-1,2-diphenylethylideneamino)-N-pyrimidin-2-yl] benzene sulfonamide] (L) was prepared through a condensation reaction of sulfadiazine and benzoin in acidic medium .The prepared ligand has been characterized with different techniques (FT-IR ,C.H.N.S, UV-Vis and ¹H and ¹³C-N.M.R). The prepared complexes were characterized and their astrochemical structures and geometries were suggested depending upon data of (UV-Vis, FT-IR, atomic absorption, micro elemental analysis, magnetic susceptibility, chloride content and molar conductance) techniques. The following general formula was achieved: [M(PHN)(L)₂], metal = {Ni^(II), Co^(II), Mn^(II), Cu^(II) and Hg^(II)}. Electronic spectra supported by magnetic moment revealed octahedral geometries for all the prepared complexes. The mixed complexes showed antibacterial activity to (*Pseudomonas aeruginosa*), (*staphylococcus aureus*) (*Escherichia coli*) and (*Bacillus subtilis*)



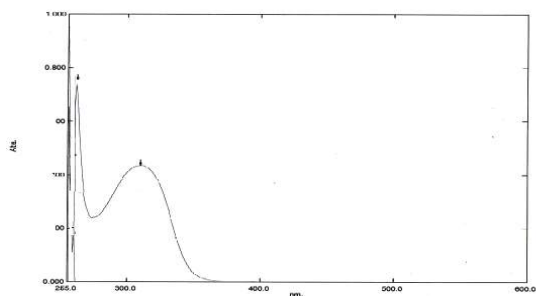


Figure 8: Electronic spectrum of the ligand

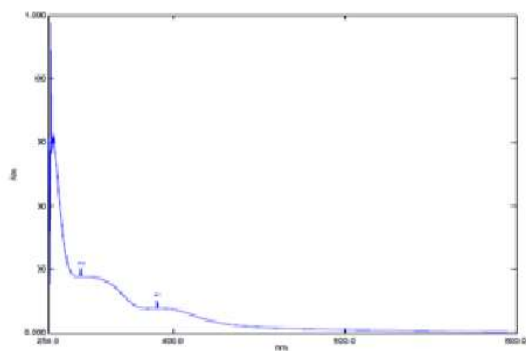
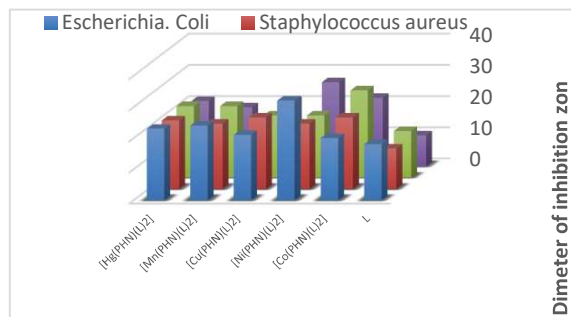
Figure 9: Electronic spectrum of [Mn(PHN)(L)₂]

Figure 10: Difference between the antimicrobial activity of ligand (L) & metal complexes

Table 1: Physical properties and elemental analysis of Schiff base and its complexes.

Compounds	Formula	Molecular Weight gm/mol	Colour	Metal salt	Weight gm/mol Metal salt	Weight gm/mol Yield%	M.P. °C	%Elemental Analysis Found % (Calculated)					
								C	H	N	S	O	M
L	C ₂₄ H ₂₀ N ₄ O ₃ S	444	Light Yellow	-	-	0.20g 70%	230	63.98 (64.85)	4.09 (4.54)	12.120 (12.60)	6.87 (7.21)	10.45 (10.80)	-
[Co(PHN)(L) ₂]	C ₆₀ H ₄₆ CoN ₁₀ O ₆ S ₂	1126	Brown	CoCl ₂ ·6H ₂ O	0.238	0.31g, 75%	260	63.23 (63.99)	3.82 (4.12)	11.86 (12.44)	5.08 (5.69)	8.00 (8.52)	4.76 (5.23)
[Ni(PHN)(L) ₂]	C ₆₀ H ₄₆ NiN ₁₀ O ₆ S ₂	1124	Purple	NiCl ₂ ·6H ₂ O	0.237	0.31g, 84%	250	63.67 (64.01)	3.77 (4.12)	12.10 (12.44)	5.22 (5.70)	7.68 (8.53)	6.54 (7.21)
[Cu(PHN)(L) ₂]	C ₆₀ H ₄₆ CuN ₁₀ O ₆ S ₂	1130	Deep purple	CuCl ₂ ·2H ₂ O	0.175	0.26g, 65%	265	63.12 (63.73)	4.28 (4.10)	12.87 (12.39)	5.01 (5.67)	7.87 (8.49)	5.07 (5.62)
[Mn(PHN)(L) ₂]	C ₆₀ H ₄₆ MnN ₁₀ O ₆ S ₂	1121	brown	MnCl ₂ ·4H ₂ O	0.198	0.25g, 79%	247	64.47 (64.22)	3.85 (4.13)	13.84 (12.48)	5.45 (5.71)	7.98 (8.55)	5.05 (4.90)
[Hg(PHN)(L) ₂]	C ₆₀ H ₄₆ HgN ₁₀ O ₆ S ₂	1268	Off-White	HgCl ₂	0.271	0.31g, 86%	256	56.48 (56.48)	3.66 (3.66)	11.05 (11.05)	4.78 (5.56)	7.87 (7.57)	5.43 (5.22)

Table 2: Infrared data for the ligand and its complexes (wave number ν) cm⁻¹

Compound	ν (OH)	ν (NH)	ν (C=N)	ν (O=S=O)	ν (O=S=O)	ν (C=C)	ν (M-N)	ν (M-O)
L	3350	3255	1651	1327	1156	1575	-	-
1,10-Phenanthroline	-	-	1620	-	-	1549	-	-
[Co(PHN)(L) ₂]	-	3252	1636 1607	1326	1155	1577	567	452
[Ni(PHN)(L) ₂]	-	3251	1628 1610	1325	1156	1574	558	422
[Cu(PHN)(L) ₂]	-	3248	1622 1612	1323	1154	1575	562	441
[Mn(PHN)(L) ₂]	-	3253	1638 1610	1325	1154	1576	551	449
[Hg(PHN)(L) ₂]	-	3244	1626	1324	1155	1575	554	443

Table3:Electronic spectral data of ligands and its metal complexes

Compound	μ_{eff}	$\lambda_{\text{m S.Cm}}^2 \text{ molar}^{-1}$	λ_{nm}	ν -wave number cm^{-1}	$\epsilon_{\text{max}} \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$	Assignments	Geometry
L	-	-	259	38610	1982	$\pi \rightarrow \pi^*$	-
			335	29850	687	$n \rightarrow \pi^*$	
			202	49504	789	$\pi \rightarrow \pi^*$	
1,10-phenanthroline	-	-	228	43859	1992	$\pi \rightarrow \pi^*$	-
			264	37878	1345	$n \rightarrow \pi^*$	
			268	37313	1987	L.F	
			347	28818	97	C.T	
			602	16611	231	${}^4\text{T}_{1\text{g}(\text{F})} \rightarrow {}^4\text{T}_{1\text{g}(\text{P})}$	
[Co(PHN)(L) ₂]	3.98	8.3	701	14265	122	${}^4\text{T}_{1\text{g}(\text{F})} \rightarrow {}^4\text{A}_{2\text{g}(\text{F})}$	octahedral
			810	12345	88	${}^4\text{T}_{1\text{g}(\text{F})} \rightarrow {}^4\text{T}_{2\text{g}(\text{F})}$	
			271	36900	1250	L.F	
			378	26455	1345	C.T	
[Ni(PHN)(L) ₂]	3.57	5.1	632	15822	168	${}^3\text{A}_{2\text{g}(\text{F})} \rightarrow {}^3\text{T}_{1\text{g}(\text{F})}$	octahedral
			803	12453	151	${}^3\text{A}_{2\text{g}(\text{F})} \rightarrow {}^3\text{T}_{2\text{g}(\text{F})}$	
			277	36101	1403	L.F	
			369	37174	1118	C.T	
[Cu(PHN)(L) ₂]	1.83	13	605	16528	89	${}^2\text{E}_{\text{g}} \rightarrow {}^2\text{T}_{2\text{g}}$	octahedral
			280	35714	988	L.F	
			360	27777	602	C.T	
[Mn(PHN)(L) ₂]	5.76	14	410	24390	162	${}^6\text{A}_{1\text{g}(\text{F})} \rightarrow {}^4\text{T}_{2\text{g}(\text{G})}$	octahedral
			732	13661	82	${}^6\text{A}_{1\text{g}(\text{F})} \rightarrow {}^4\text{T}_{1\text{g}(\text{G})}$	
			285	35087	165	L.F	
			410	24390	230	C.T	
[Hg(PHN)(L) ₂]	-	10					octahedral

Table 4:Diameter inhibition zone(mm)of ligand and its mixed complexes.

Comp.	L	[Co(PHN)(L) ₂]	[Ni(PHN)(L) ₂]	[Cu(PHN)(L) ₂]	[Mn(PHN)(L) ₂]	[Hg(PHN)(L) ₂]
<i>Escherichia. Coli</i>	18	20	32	21	24	23
<i>Staphylococcus aureus</i>	13	23	21	23	21	22
<i>Bacillus</i>	15	28	20	20	23	23
<i>Pseudomonas</i>	10	22	27	21	19	21

REFERENCES

- [1] A.A Emara, A Azza, A Abou-Hussen ", Spectroscopic studies of bimetallic complexes derived from tridentate or tetradentate Schiff bases of some di- and tri-valent transition metals," *Spectrochim. Acta Part A*, p. 1010–1024, 2006.
- [2] J Taswar and AS Makode, "Synthesis, characterization, biological and thermal properties of some new Schiff base complexes derived from 2-hydroxy-5-chloro-acetophenone and S-methyl dithiocarbazate,," *Indian J. chem*, vol. ; 43, no. A, pp. 2120-2125., 2004.
- [3] M Ravanasiddappa, T Sureshg, K Syed, S C Radhavendray, C Basavaraja and S DAngadi, "Transition Metal Complexes of 1, 4 (2-Hydroxyphenyl -1-yl) Diimino Azine, Synthesis, Characterization and Antimicrobial Studies," *E-J. chem*, vol. 5, no. 2, pp. 395-403, 2008.
- [4] M. Raju, "Synthetic, structural and biological Properties of binuclear complexes with some Schiff bases," vol. 64, no. 4, pp. :345-353, 2007.
- [5] K.Tahir, S.Minnat and A, Transition metal ion directed bimetallic macrocyclic complexes, *Trans. Met. Chem .*, vol. ; 24, pp. : 669 -671, 1999.
- [6] JBalsells, LMejorado, M. Phillips, F Ortega, G Aguirre, RSomanathan and P.J

- Walsh, "Synthesis of Chiral Sulfonamide/Schiff Base Ligands," *Tetrahedron Asymm*, vol. 9, 1998.
- [7] M Kojima, H. Taguchi, M. Tsuchimoto and K Nakajima, *Coordination Chemistry Reviews*, vol. 237, 2003, pp. 183-196.
- [8] NJ Long, "Organometallic compounds for nonlinear optics: the search for enlightenment *Angewandte*," *Chem. Inter.Ed*, vol. 34, no. 21, 1995.
- [9] MLD, Rotondo and E A Rotondo, "NMR Study of L-Shaped Quinoxaline Platinum(II) Complexes-Crystal structure of $[Pt(DMeDPQ)(bipy)](PF_6)_2$," *Eur. J. Inorg. Chem*, pp. 4710 - 4717, 2004;.
- [10] E. G. a. M. LG, *Advances in Inorganic Biochemistry Models in Inorganic Chemistry*, PTR prentice-Hall, Inc, 1994.
- [11] K. Burger "Coordination Chemistry Experimental Methods," 1973.
- [12] E Canpolat and M Kaya, "Studies on mononuclear chelates derived from substituted Schiff-base ligands (part 2) synthesis and characterization of a new 5-bromosalicylidene p-aminoacetophenone oxime and its complexes with Co(II), Ni(II), Cu(II) and Zn(II)".
- [13] A R Chakravarty, AP Nreddy, BK Santra and AM Thomas, "Copper Complexes as Chemical Nucleases," *J. Indian. Chem. Sci.*, Vols. 114,, pp. 319-401., 2002.
- [14] BP. Lever, *Inorganic Electronic spectroscopy*, Elsevier, 1984.
- [15] S. S Chandra and J Sangeetika, "EPR and electronic spectrum studies on copper(II) complexes of some N-O donor ligands," *J. Indian Chem. Soc*, no. 81, pp. 203-206, 2004.
- [16] J. Sima, "Mechanism of Photoredox Reactions of Iron(III) Complexes Containing Salen-type Ligands," *Crao. Chem. Acta*, vol. 593, p. ., 2001.
- [17] S. Ren, R. Wang, K. Komastu, P. BKrause, Y. Zyrianov, C. EMckenna, C. Csipke, Z. A. Tokes and E. J. Lien S, "Synthesis, biological evaluation, and quantitative structure-activity relationship analysis of new Schiff bases of hydroxy semicarbazide as potential antitumor agents," *J. Med. Chem.*, vol. 45, no. 410, 2002.
- [18] A. M. A. S. N. W. W. Y. K. S. T. N. U. C. K. A. Tarafder MTH, "Coordination chemistry and biological activity of nickel(II) and copper(II) ion complexes with nitrogen-sulphur donor ligands derived from S-benzyl dithio carbazate (SBDTC)," *Transition Met Chem* 2001, vol. 26, no. 613.
- [19] T. S. ., K. S. ., S. C. R. ., C. B. a. S. D. A. M Ravanasiddappa, ", "Transition Metal Complexes of 1, 4 (2-Hydroxyphenyl -1-yl) Diimino Azine Synthesis, Characterization and Antimicrobial Studies," *E-J. chem*, vol. 2, pp. 395-403, 2008.