



## Original Research Article

# Synthesis, Structural, Thermal and Biological Studies of Ligand Derived from Anthrone with 4-Aminoantipyrine and its Metallic Complexes

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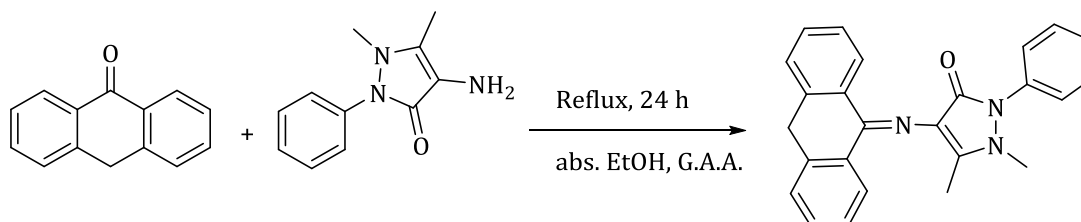
Metal complexes

Biological activity

## ABSTRACT

This study presented the synthesis of ligand 4-(anthracen-9(10H)-ylideneamino)-1,5-dimethyl-2-phenyl-1,2-dihydro-3H-pyrazol-3-one (L2), synthesized from reaction of one equivalent of (Anthrone) with one equivalent for 4-aminoantipyrine and its complexes for metal ions [Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II)]. The ligand and their complexes were characterized by melting point measurement, elemental microanalysis C.H.N, FT-IR, UV, (<sup>1</sup>H, <sup>13</sup>C-NMR and Mass spectroscopy only ligand) along with atomic absorption spectrophotometer, chloride contents, conductivity measurement, magnetic susceptibility and thermal gravimetric analysis only complexes, in addition to evaluating their biological activity against the types of bacteria. Based on data of all techniques suggested an octahedral geometry for complexes except complex [Ni(L)<sub>2</sub>] Cl<sub>2</sub>.H<sub>2</sub>O, the shape square planer and complexes Cd(II), Hg(II) and Zn(II) were shown. The shape tetrahedral appeared. The results showed biological activity against the types of bacteria for most of the synthesized ligand and its complexes.

## GRAPHICAL ABSTRACT



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## Introduction

Heterocyclic compounds are importance in organic chemistry because of their biological activities [1]. Pyrazol is doubly unsaturated 5 membered ring compound with 3 C atoms and 2 N atoms. Many pyrazoline substitution productions are useful in medical [2]. 4-Aminoantipyrine (4-AAP) is one of the heterocyclic compounds containing 2 N atoms in its ring, linked with a more reactive amine and carbonyl functional groups. The existence of hetero atoms affects the redistribution of electrons and thus exhibits aromatic character, which is designated as heteroatom effect [3] that imparts the reactivity, chelating effect, etc. Because of this, it is individually useful in research areas such as analytical, modern organic, bioorganic and medicine chemistry [4, 5]. 4-Aminoantipyrine (4-AAP), an anti-pyretic agent, is one of the pyrazole derives [6]. Many preparation compounds containing pyrazole have drawn attention in the field of medicine chemistry because of their pharmacological, photographic, catalytic and liquid crystal application [7, 8]. Its metallic complexes have some applications in analytic and pharmacologic fields [9]. The transition metals complexes of 4-aminoantipyrine (4-AAP) and its derived have been extensive examined related to their many applications in different areas such as biological, analytical and therapeutically [10].

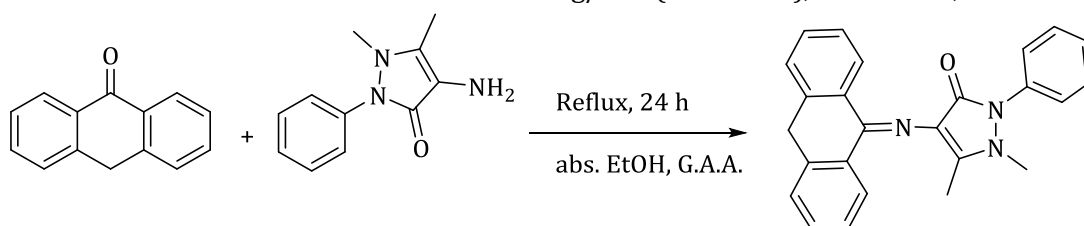
## Materials and Methods

Melting point for prepared compounds were measured by using digital melting point apparatus Stuart Melting Point. IR spectra of the ligand and its complexes were measured using a Shimadzu (FT-IR)-8400S spectrophotometer at range 400-4000  $\text{cm}^{-1}$ . The U.V spectra of the compounds were using U.V-Vis spectrophotometer kind

double beam at range 200-1000 nm, Shimadzu UV160A nm; the standard solutions was 0.001 mol/L in dimethyl sulfoxide solvent and the cell is one cm long, which is made from quartz. The  $^1\text{H}$ -NMR and  $^{13}\text{C}$ -NMR spectra of compounds were recorded on Bruker DRX kind system (500 MHz) in TMS as a standard in Dimethyl sulfoxide- $\text{d}_6$  solution. Mass spectra for ligand was recorded by (EI) mass spectroscopic using MS Model: 5973 Network Mass Selective Detector. TGA analysis of some compounds was carried out using a STA PT-1000 Linseis company /Germany. Molar conductivity measurements for complexes were measured at (27  $^{\circ}\text{C}$ ) of 0.001 mol/ L solution for complexes in DMSO using a Jenway Ltd. 4071 digital conductivity meter. The magnetic sensitiveness for some complexes was checked using in Balance Johnson Matthey. The elemental micro analysis of ligand and its complexes were measured in Euro vector model (EA3000). Metals content of compounds were recorded using Shimadzu atomic absorption spectrophotometer 680G. Potentiometry titrations methods were used to determined chloride content of compounds using a 686-titro processor-665 Dosimat-Metrohm Swiss. Agar diffusion was used to test antibacterial activity.

### Synthesis of Schiff base ligand (L2)

0.203 g was dissolved of 4-AAP (1 mmol) in 20 mL absolute ethanol and added to a solution of anthrone (0.194 g, 1 mmol) mixed with 60 mL absolute ethanol and 60 mL glacial acetic acid. The mixture was refluxing for 24 h with stirring. Brown precipitate formed which was washed by ethanol and recrystallized with methanol to get a pure product and dried at room temperature [11]. The melting point of Schiff base ligand was prepared at 205-207  $^{\circ}\text{C}$ , molecular weight: 379 g/mol ( $\text{C}_{25}\text{H}_{21}\text{N}_3\text{O}$ ), Yield: 93%, Scheme 1.

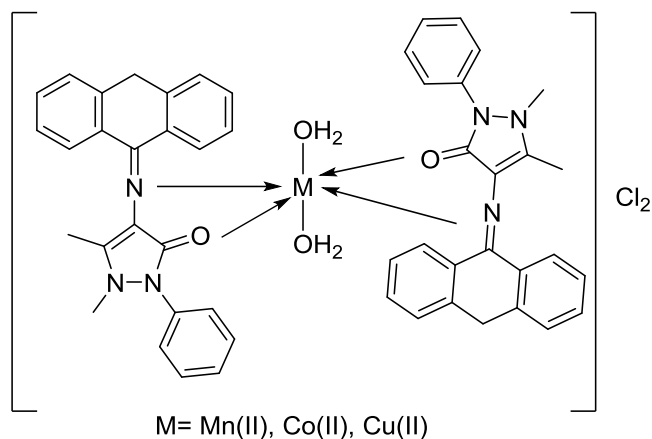


**Scheme 1:** Synthetic route for ligand L2

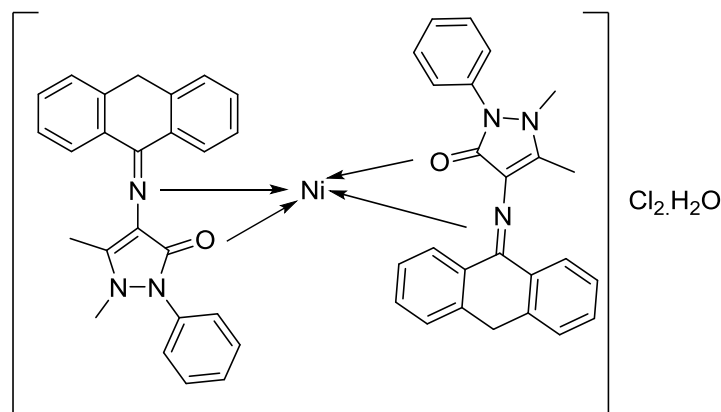
### Preparation of L2 complexes

0.179 g was dissolved of manganese (II) chloride tetra hydrate (1 mmol) in 20 mL of absolute ethanol and added drop wise to a solution of L2 dissolved in 25 mL of absolute ethanol. The mixture was refluxed for 1 h with stirring. Brown

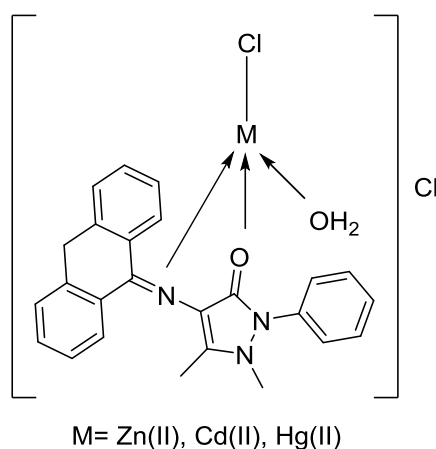
precipitate was produced, washed several times with absolute ethanol to get a pure sample and dried [12]. Using a similar method of Mn(II) complex, we synthesized the complexes  $MCl_2 \cdot nH_2O$ ,  $M(II)=[Co (n=6), Cu (n=2) ions, Ni (n=6) ion, Zn (n=0), Cd (n=2) and mercury (n=0) ions]$  (Schemes 2-4).



**Scheme 2:** Structure of complexes of Manganese (II), Cobalt (II) and Copper (II) ions



**Scheme 3:** Structure of complex for Ni(II) ion



**Scheme 4:** structure of complexes for Zn(II), Cd (II) and Hg(II)) ions

## Results and discussion

The physical properties, C.H.N analysis, metal content and chloride content data are listed in Table 1.

### Characterization of ligand L2

#### FT-IR spectra

The FT-IR spectrum of L2 (Figure S1) shows new peak at  $1658\text{ cm}^{-1}$  due to imine group (C=N) stretching vibration [13]. The ligand spectrum did not show the appearance of two bands of  $\nu_{\text{asym}}(\text{NH}_2)$  and  $\nu_{\text{sym}}(\text{NH}_2)$  for 4-amino antipyrine and band of C=O of anthrone and appearance of imine group indicate the formation of Schiff base L2 [14].

#### Electronic spectrum

The Ultraviolet-visible (UV-Vis) spectrum of L2 (Figure S2) exhibited two absorption peaks; the first strong peak at 301 nm and  $332\text{ cm}^{-1}$  refers to  $\pi \rightarrow \pi^*$  electronic transition. The other weak band at 400 nm ( $25000\text{ cm}^{-1}$ ) is attributed to  $n \rightarrow \pi^*$  electronic transitions [15].

#### $^1\text{H}$ -NMR spectrum

$^1\text{H}$ -NMR spectrum for L2 is displayed the resonance at chemical shift  $\delta_{\text{H}} = 7.03\text{--}7.98\text{ ppm}$  related to protons of aromatic ring Ar-CH. The spectrum displayed chemical shifts at  $\delta_{\text{H}} = 4.48\text{ ppm}$  attributed to protons of  $\text{CH}_2$  group of

anthrone [11]. The manifestation of these protons as a multi are attributed to mutual coupling. The spectrum showed chemical shifts at  $\delta_{\text{H}} = 2.49\text{--}2.51\text{ ppm}$  and  $3.15\text{ ppm}$  pointing to Dimethyl sulfoxide and existence of water molecule HOD in solvent, respectively [16]. The spectrum showed chemical shifts at  $\delta_{\text{H}} = 2.39$  and  $3.19\text{ ppm}$ , which are assignable to protons of C- $\text{CH}_3$  and N- $\text{CH}_3$  groups, respectively (Figure S3) [13].

#### $^{13}\text{C}$ -NMR spectrum

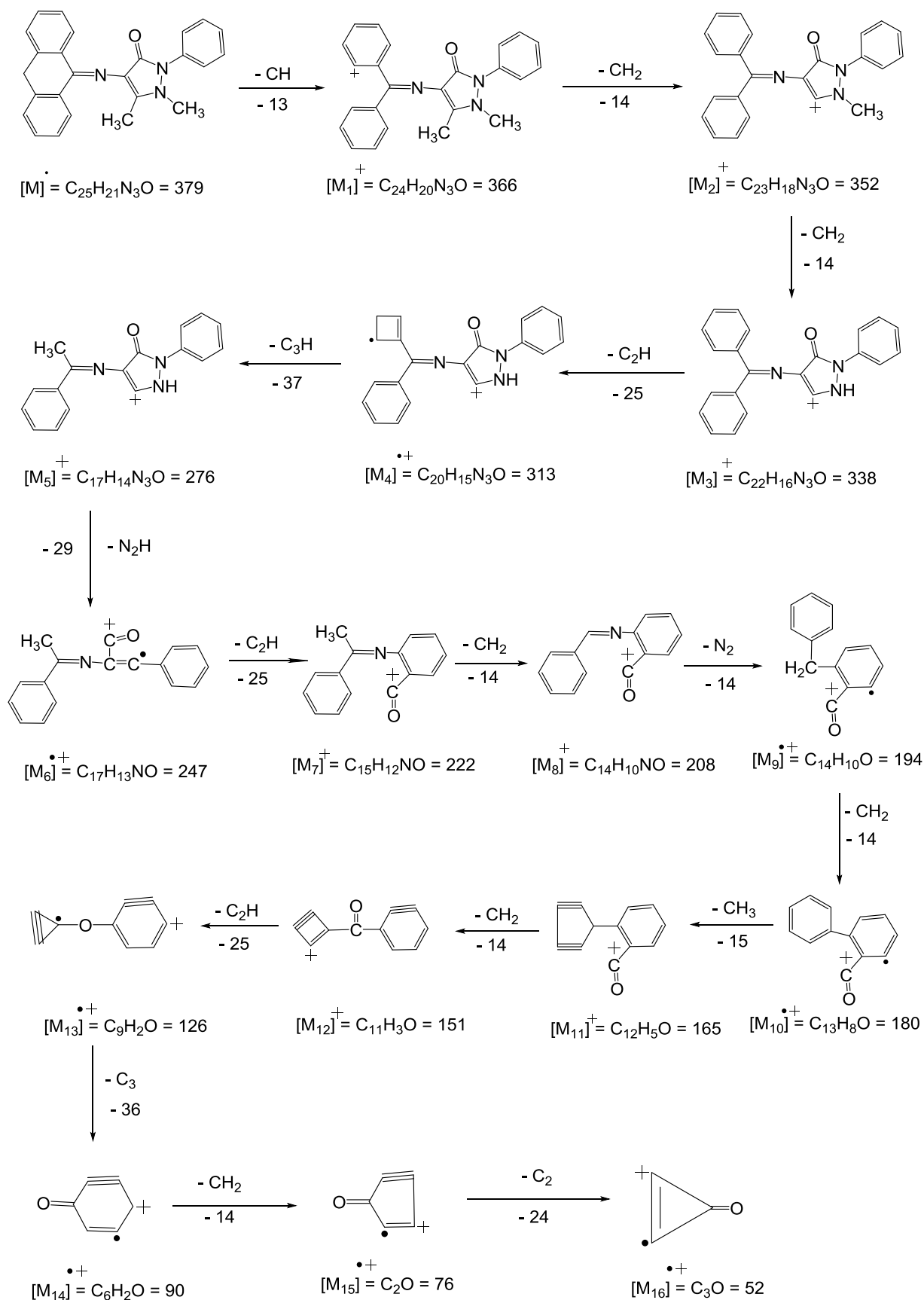
The  $^{13}\text{C}$ -NMR spectrum of L2 in dimethyl sulfoxide- $d_6$  solvent showed chemical shift at range  $\delta = 124.1\text{--}129.8\text{ ppm}$  pointing to aromatic carbons atoms [11]. The chemical shifts at  $\delta = 182.9\text{ ppm}$  were due to C=O carbon atom ( $\text{C}_{16}$ ), while the chemical shifts at  $\delta = 160\text{ ppm}$  were due to C=N carbon atom ( $\text{C}_{13}$ ) [17]. The chemical shifts at  $\delta = 32.5\text{ ppm}$  were due to methylene group ( $\text{C}_{14}$ ). The chemical shifts at  $\delta = 133.4\text{ ppm}$  were due to ( $\text{C}_{23}$ ) [11]. Chemical shifts at  $\delta = 10.7\text{ ppm}$  and  $32.5\text{ ppm}$  were assigned to methyl group carbon atoms ( $\text{C}_{20}, 22$ ), respectively (Figure S4) [18].

#### Mass spectra

The mass spectrum for L2 is shown in Figure S5. The molecular ion peak of ligand appearing at  $m/z^+ = 379\text{ [M]}^+ \text{C}_{25}\text{H}_{21}\text{N}_3\text{O}$  requires = 379 [19].

**Table 1:** Analysis data and Physical properties for ligand and its metals complexes

Molecular formula of complexes	m.p ( $^{\circ}\text{C}$ )	M.Wt (g/mol)	found/(calc.)%				
			C	H	N	Cl	Metal
[Mn (C <sub>25</sub> H <sub>21</sub> N <sub>3</sub> O) <sub>2</sub> (OH <sub>2</sub> ) <sub>2</sub> ]Cl <sub>2</sub>	219-221	920	65.05	4.87	9.27	7.45	5.79
			(65.21)	(5.00)	(9.13)	(7.71)	(5.97)
[Co(C <sub>25</sub> H <sub>21</sub> N <sub>3</sub> O) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub>	220-222	924	64.72	4.72	8.69	7.31	6.19
			(64.93)	(4.97)	(9.09)	(7.68)	(6.38)
[Ni(C <sub>25</sub> H <sub>21</sub> N <sub>3</sub> O) <sub>2</sub> ]Cl <sub>2</sub> . H <sub>2</sub> O	210-212	905.7	66.09	4.68	9.09	7.59	6.33
			(66.24)	(4.85)	(9.27)	(7.83)	(6.48)
[Cu(C <sub>25</sub> H <sub>21</sub> N <sub>3</sub> O) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub>	219-221	928.5	64.45	4.73	8.88	7.37	6.75
			(64.62)	(4.95)	(9.04)	(7.44)	(6.83)
[Zn(C <sub>25</sub> H <sub>21</sub> N <sub>3</sub> O)(H <sub>2</sub> O)(Cl)]Cl	221-223	533.4	56.16	4.43	7.65	13.05	12.08
			(56.24)	(4.61)	(7.87)	(13.31)	(12.26)
[Cd(C <sub>25</sub> H <sub>21</sub> N <sub>3</sub> O)(H <sub>2</sub> O)(Cl)]Cl	225-227	580.4	51.44	4.14	7.12	12.09	19.11
			(51.68)	(4.30)	(7.23)	12.23	(19.36)
[Hg(C <sub>25</sub> H <sub>21</sub> N <sub>3</sub> O)(H <sub>2</sub> O)(Cl)]Cl	210-212	668.6	44.71	3.59	6.11	10.42	29.87
			(44.86)	(3.73)	(6.28)	(10.61)	(30.00)



**Scheme 5:** The suggested mass fragmentation of (L2)

The other peaks detected at  $m/z^+ = 366-52$  correspond to  $[C_{24}H_{20}N_3O]^+ - [C_3O]^+$ . The suggested mass fragmentation of L2 is shown in Scheme 5.

### Characterization of complexes

#### FT-IR spectra

The assignment of the characteristic peaks for functional group of complexes are presented in Table 2 (Figure 1 and 2). The peak at  $1635\text{ cm}^{-1}$  is assigned to the stretching frequency for azomethine group  $C=N$  of the free ligand. This band was shifted to lower or higher frequency at range  $1589-1650\text{ cm}^{-1}$  in spectra of all prepared complexes; this shift may be due to involved nitrogen atom of azomethine group in

coordination with metal ions [11]. The peak at  $1678\text{ cm}^{-1}$  stretching vibration refers to  $C=O$  for carbonyl group of free ligand was shifted at range  $1649-1674\text{ cm}^{-1}$  in spectra of all complexes, displaying coordination between oxygen atom of this group and metal ions [20]. At the lower frequency region, the IR spectra of all synthesized complexes showed new bands, not present in the spectrum of the free ligand; these bands are located at  $495-505\text{ cm}^{-1}$ ,  $455-464\text{ cm}^{-1}$  due to  $M-N$ ,  $M-O$ , respectively [21, 22]. The band at  $3365\text{ cm}^{-1}$  in spectrum of complex  $[Ni(L_2)]Cl_2 \cdot H_2O$  is due to  $H_2O$  hydrated [23], while the stretching bands at range  $3062-3431\text{ cm}^{-1}$  and at range  $910-920\text{ cm}^{-1}$  in spectrum for complexes were due to coordination of  $H_2O$  (aqua) [24].

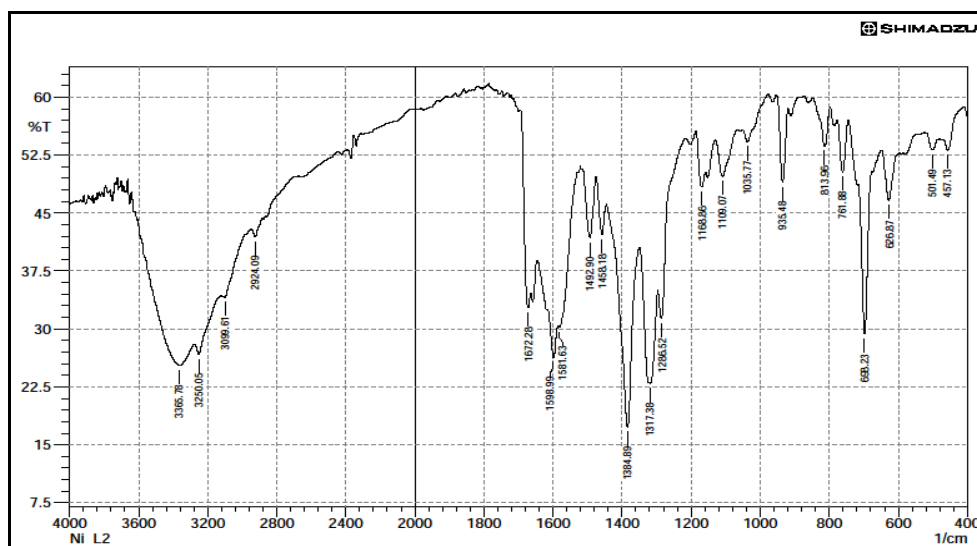


Figure 1: FT-IR spectrum for  $[Ni(L^2)_2]Cl_2 \cdot H_2O$

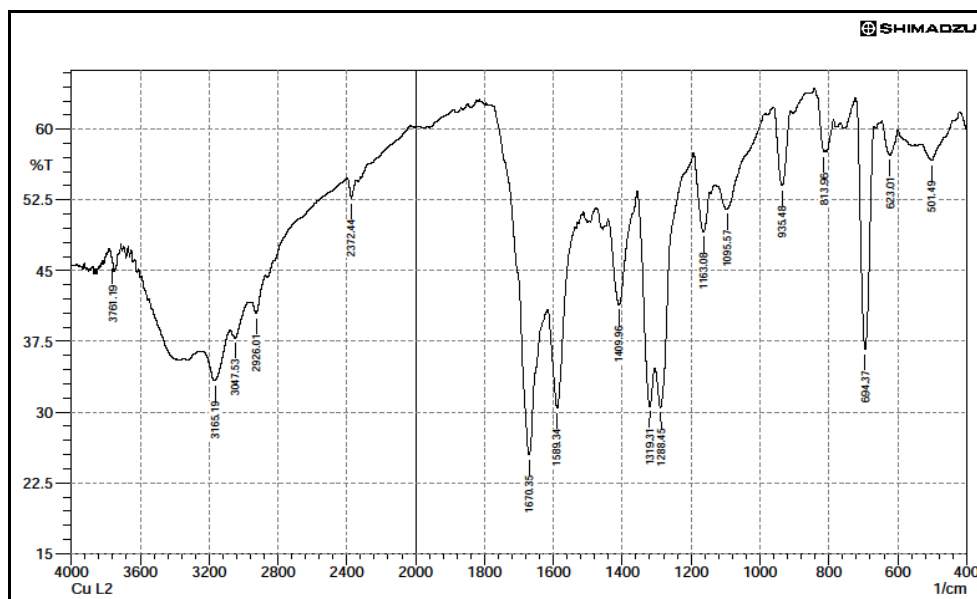


Figure 2: FT-IR spectrum for  $[Cu(L^2)_2(H_2O)_2]Cl_2$

**Table 2:** FT-IR data (cm<sup>-1</sup>) of Schiff base (L2) complexes

Compounds	$\nu(\text{C=O})$	$\nu(\text{C=N})$ imin	M-N	M-O
[Mn(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub>	1649	1597	495	460
[Co(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub>	1674	1620	505	464
[Ni(L) <sub>2</sub> ] Cl <sub>2</sub> .H <sub>2</sub> O	1672	1650	501	457
[Cu(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub>	1670	1589	501	460
[Zn(L) (H <sub>2</sub> O)(Cl)]Cl	1656	1637	501	464
[Cd(L) (H <sub>2</sub> O)(Cl)]Cl	1658	1593	505	455
[Hg(L) (H <sub>2</sub> O)(Cl)]Cl	1658	1631	501	462

### Electronic Spectra

The electronic spectra data of synthetic complexes are listed in Table 3 with electronic transition and suggestion geometries (Figure S6 and S7). The electronic spectra of prepared complexes showed

two-four absorption bands at range 272-393 nm and 36765-25445 cm<sup>-1</sup> which were attributed to the intra-ligand [25]. New absorption peak at range 392-420 nm and 25510-23810 cm<sup>-1</sup> in spectra for complexes can be assigned to MLCT [23].

**Table 3:** Electronic spectral data for complexes

Compounds	$\lambda$ (nm)	$\nu$ (cm <sup>-1</sup> )	$\epsilon_{\text{max}}$ (M <sup>-1</sup> .cm <sup>-1</sup> )	Assignment	Suggested Structure
[Mn(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub>	299	33445	2223	Intra-ligand	Oh
	393	25445	311	Intra-ligand	
	415	24096	240	MLCT + ( <sup>6</sup> A <sub>1g</sub> → <sup>4</sup> T <sub>2g</sub> (G))	
	500	20000	30	( <sup>6</sup> A <sub>1g</sub> → <sup>4</sup> T <sub>1g</sub> (G))	
[Co(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub>	298	33557	2256	Intra-ligand	Oh
	371	26954	306	Intra-ligand	
	392	25510	304	MLCT	
	614	16287	42	( <sup>4</sup> T <sub>1g</sub> (F) → <sup>4</sup> T <sub>1g</sub> (P))	
	682	14663	63	( <sup>4</sup> T <sub>1g</sub> (F) → <sup>4</sup> A <sub>2g</sub> (F))	
	849	11779	5	( <sup>4</sup> T <sub>1g</sub> (F) → <sup>4</sup> T <sub>2g</sub> (F))	
[Ni(L) <sub>2</sub> ] Cl <sub>2</sub> . H <sub>2</sub> O	276	36232	1786	Intra-ligand	Sq.planer
	301	33223	1783	Intra-ligand	
	357	28011	176	Intra-ligand	
	392	25510	142	MLCT	
	500	20000	18	( <sup>1</sup> A <sub>1g</sub> → <sup>1</sup> B <sub>1g</sub> )	
	967	10341	2	( <sup>1</sup> A <sub>1g</sub> → <sup>1</sup> E <sub>g</sub> )	
[Cu(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]Cl <sub>2</sub>	272	36765	1589	Intra-ligand	Dist. Oh
	302	33113	1246	Intra-ligand	
	318	31447	1184	Intra-ligand	
	356	28090	876	Intra-ligand	
	396	25253	402	MLCT	
	12719	13908	6	<sup>2</sup> B <sub>1g</sub> → <sup>2</sup> A <sub>1g</sub>	
	805	12422	5	<sup>2</sup> B <sub>1g</sub> → <sup>2</sup> B <sub>2g</sub>	
[Zn(L) (H <sub>2</sub> O)(Cl)]Cl	286	34965	1908	Intra-ligand	td
	392	25510	203	Intra-ligand	
	416	24038	164	MLCT	
[Cd(L) (H <sub>2</sub> O)(Cl)]Cl	272	36765	1603	Intra-ligand	td
	392	25510	152	Intra-ligand	
	420	23810	158	MLCT	
[Hg(L) (H <sub>2</sub> O)(Cl)]Cl	279	35842	1830	Intra-ligand	td
	393	25445	181	Intra-ligand	
	416	24038	162	MLCT	



### Molar conductance

The molar conductivity values of Schiff base ligand complexes in DMSO ( $10^{-3}$ M solution) at room temperature are listed in Table 4. The molar conductivity values of complexes  $[\text{Mn}(\text{L}2)_2(\text{H}_2\text{O})_2]\text{Cl}_2$ ,  $[\text{Co}(\text{L}2)_2(\text{H}_2\text{O})_2]\text{Cl}_2$  and  $[\text{Cu}(\text{L}2)_2(\text{H}_2\text{O})_2]\text{Cl}_2$  are 1:2 electrolytic natures. The molar conductivity values of complexes  $[\text{Zn}(\text{L}2)(\text{H}_2\text{O})(\text{Cl})]\text{Cl}$ ,  $[\text{Cd}(\text{L}2)(\text{H}_2\text{O})(\text{Cl})]\text{Cl}$  and  $[\text{Hg}(\text{L}2)(\text{H}_2\text{O})(\text{Cl})]\text{Cl}$  are 1:1 electrolytic natures.

### Magnetic properties

The  $X_M$ ,  $X_A$ , and  $\mu_{\text{eff}}$  of the complex were calculated using the  $X_g$  value obtained and theoretically calculated D. The magnetic moments of complexes are listed in Table 4.

### Thermal Analysis

#### Thermal Analysis of $[\text{Mn}(\text{L}2)_2(\text{H}_2\text{O})_2]\text{Cl}_2$

The thermo gram for  $[\text{Mn}(\text{L}2)_2(\text{H}_2\text{O})_2]\text{Cl}_2$  is shown in Figure S8. In TGA, peak recognized at 106.25 °C is pointing to loss ( $\text{H}_2\text{O}$ ) portions, (W.t = 0.1890 mg, 1.95 %). The second step at 317.291 °C that specific to loss ( $\text{H}_2\text{O}$ ,  $\text{Cl}_2$ ,  $2\text{CH}_3$ ,  $\text{C}_6\text{H}_5$ ,  $\text{C}_2\text{CON}_2$ ) fragment, (W.t = 2.90 mg, 30 %). The third step at 546.708 °C pertains to loss ( $\text{C}_{14}\text{H}_{10}\text{N}$ ) fragment, (W.t = 2.02 mg, 20.86 %). The fourth step at 899.958 °C is designated to the loss of ( $\text{CH}_4$ ,  $\text{H}_2$ ,  $\text{C}_6\text{H}_5$ ,  $\text{CO}$ ) fragment, (W.t = 1.43 mg, 14.78 %). The final remainder of the compound that appeared

above 900 °C is assignable to  $(\text{MnC}_{17}\text{H}_{10}\text{N}_2)$ , (W.t = 3.14 mg, 32.41 %) [26].

#### Thermal Analysis of $[\text{Zn}(\text{L}2)(\text{H}_2\text{O})(\text{Cl})]\text{Cl}$

The thermo gram for  $[\text{Zn}(\text{L}2)(\text{H}_2\text{O})(\text{Cl})]\text{Cl}$  is shown in Figure S9. In TGA, band recognized at 334.125 °C is specific to loss ( $\text{H}_2\text{O}$ ,  $\text{Cl}_2$ ,  $2\text{CH}_3$ ,  $\text{C}_6\text{H}_5$ ) portions, (det. = 2.20 mg, 36.74 %). The second step at 899.958 °C pertains to loss ( $\text{C}_{14}\text{H}_6$ ) fragment, (obs. = 1.95 mg, 32.63 %). The final remainder for compound that appeared above 900 °C is specific to  $(\text{ZnC}_4\text{H}_6\text{N}_2\text{O})$ , (W.t = 1.83, 30.63 %) [27].

### Biological activity of Schiff base (L2) and its complexes

The prepared of ligand L2 and its metal complexes of this study were tested against types from bacteria gram negative such as *Bacillus* (Figure 3) and *Escherichia coli* (Figure 4) and gram positive such as *Pseudomonas auroginosa* (Figure 5) and *Staphylococcus aureus* (Figure 6). The ligand and its complexes showed activity against both gram-positive and gram-negative bacteria; some complexes showed better antibacterial activity than the identical ligand [28], but there are other reports in which the ligand has had higher activity than its complexes [29]. The higher activity for complexes is usually expressed mainly based on the Tweedy's chelation theory [30]; the measured areas of inhibition against the growth of various microorganisms are shown in Table 5.

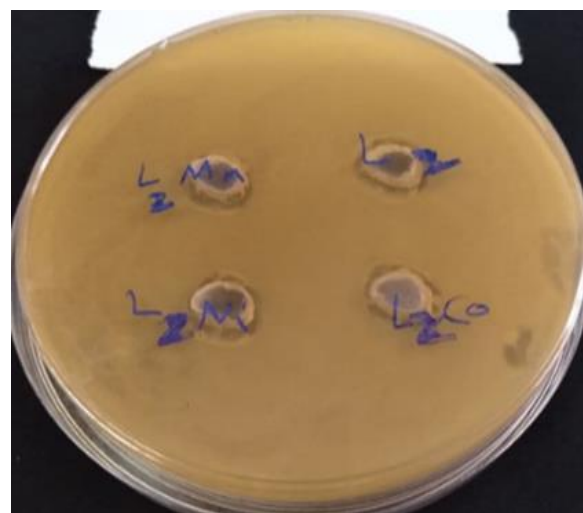
**Table 4:** The molar conductivity of Schiff bases ligand complexes

Complexes	$\Lambda$ s.cm <sup>2</sup> .m ol <sup>-1</sup>	ratio	$X_g \times 10^{-6}$	$X_M \times 10^{-6}$	$X_A \times 10^{-6}$	$\mu_{\text{eff}}$ (B.M)	NO. of unpaired electrons
$[\text{Mn}(\text{L}2)_2(\text{H}_2\text{O})_2]\text{Cl}_2$	75.71	2:1	11.410	10497.93	10832.61	5.10	5
$[\text{Co}(\text{L}2)_2(\text{H}_2\text{O})_2]\text{Cl}_2$	72.36	2:1	8.881	8206.04	8540.72	4.53	3
$[\text{Ni}(\text{L}2)]\text{Cl}_2 \cdot \text{H}_2\text{O}$	72.51	2:1	0.00	0.00	0.00	0.00	0
$[\text{Cu}(\text{L}2)_2(\text{H}_2\text{O})_2]\text{Cl}_2$	71.63	2:1	1.240	1151.34	1486.02	1.89	1
$[\text{Zn}(\text{L}2)(\text{H}_2\text{O})(\text{Cl})]\text{Cl}$	30.41	1:1	-	-	-	0	0
$[\text{Cd}(\text{L}2)(\text{H}_2\text{O})(\text{Cl})]\text{Cl}$	36.30	1:1	-	-	-	0	0
$[\text{Hg}(\text{L}2)(\text{H}_2\text{O})(\text{Cl})]\text{Cl}$	30.42	1:1	-	-	-	0	0





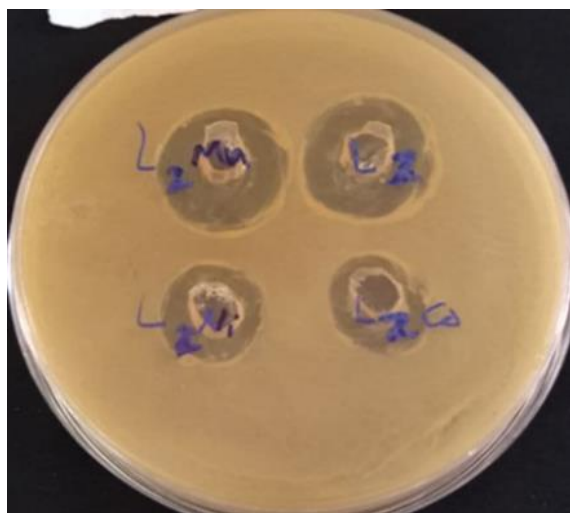
**Figure 3:** Biological activity of Schiff bases ligand and their complexes against *Bacillus bacteria*



**Figure 4:** Biological activity of Schiff bases ligand and their complexes against *Escherichia coli bacteria*



**Figure 5:** Biological activity of Schiff bases ligand and their complexes against *Pseudomonas auroginosa bacteria*



**Figure 6:** Biological activity of Schiff bases ligand and their complexes against *Staphylococcus aureus* bacteria

**Table 5:** Bacterial activity for ligand (L2) and its complexes

Compounds	<i>Bacillus</i>	<i>Escherichia coli</i>	<i>Pseudomonas auroginosa</i>	<i>Staphylococcus aureus</i>
DMSO	-	-	-	-
L2	18	13	16	21
[Mn(L2) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ] Cl <sub>2</sub>	19	12	16	21
[Co(L2) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ] Cl <sub>2</sub>	20	13	17	15
[Ni(L2) <sub>2</sub> ] Cl <sub>2</sub> . H <sub>2</sub> O	20	12	17	16
[Cu(L2) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ] Cl <sub>2</sub>	14	14	17	22
[Zn(L2) (H <sub>2</sub> O)] Cl	17	16	12	25
[Cd(L2) (H <sub>2</sub> O)] Cl	13	15	19	20
[Hg(L2) (H <sub>2</sub> O)] Cl	16	16	20	20

## Conclusion

1. The ligand (L2) acted in the form of bi dentate ligand through a nitrogen in imine and oxygen in (C=O) grope with metallic ions M(II): Manganese, Cobalt, Nickel, Cupper, Zink, Cadmium and mercury .
2. The octahedral shape structure is suggested for prepared complexes except complex [Ni(L)<sub>2</sub>] Cl<sub>2</sub>.H<sub>2</sub>O showing the shape square planer and complexes Cd(II), Hg(II) and Zn(II). It showed the shape tetrahedral.
3. The synthesized ligand and its complexes have had biological activity against some types of bacteria such as *Bacillus*, *Escherichia coli*, *Pseudomonas auroginosa* and *Staphylococcus aureus*.

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## Authors' contributions

All authors contributed toward data analysis, drafting and revising the paper and agreed to responsible for all the aspects of this work.

## Conflict of Interest

We have no conflicts of interest to disclose.

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## Supporting Information

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## References

- [1]. Elemike E.E., Oviawe A.P., Otuokere I.E., Potentiation of the antimicrobial activity of 4-benzylimino-2, 3-dimethyl-1-phenylpyrazol-5-one by metal chelation, *Research Journal of Chemical Sciences*, 2011, **1**:6 [[Google Scholar](#)]
- [2]. Bhutani S.P., *Organic Chemistry*, 1st, Ed., Ane Books, India, 2007, 112 [[Google Scholar](#)]
- [3]. Matczak P., Domagała M., Heteroatom and solvent effects on molecular properties of formaldehyde and thioformaldehyde symmetrically disubstituted with heterocyclic groups C<sub>4</sub>H<sub>3</sub>Y (where Y = O–Po), *Journal of molecular modeling*, 2017, **23**:268 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [4]. Joule J.A., Mills K., *Heterocyclic Chemistry*, 5th ed., John Wiley & Sons, 2008 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [5]. Tyrell J.A., Quin L.D., *Fundamentals of Heterocyclic chemistry: Importance in Nature and in the Synthesis of Pharmaceuticals*, John Wiley & Sons, 2010 [[Google Scholar](#)], [[Publisher](#)]
- [6]. Acheson R.M., *Introduction to Heterocyclic compounds*, 4th, Ed., John Wiley and Sons, New York, 2009 [[Google Scholar](#)]
- [7]. Agarwal R.K., Singh I., Sharma D.K., Synthesis, spectral, and biological properties of copper (II) complexes of thiosemicarbazones of Schiff bases derived from 4-aminoantipyrine and aromatic aldehydes, *Bioinorganic Chemistry and Applications*, 2006, **2006** [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [8]. Tharmaraj P., Kodimunthiri D., Sheela C.D., Shanmugapriya C.S., Synthesis, spectral studies and antibacterial activity of Cu (II), Co (II) and Ni (II) complexes of 1-(2-hydroxyphenyl)-3-phenyl-2-propen-1-one, N<sub>2</sub>-[(3, 5-dimethyl-1H-pyrazol-1-yl) methyl] hydrazine, *Journal of the Serbian Chemical Society*, 2009, **74**:927 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [9]. Raman N., Sakthivel A., Rajasekaran K., Synthesis and spectral characterization of antifungal sensitive Schiff base transition metal complexes, *Mycobiology*, 2007, **35**:150 [[Google Scholar](#)], [[Publisher](#)]
- [10]. Cunha S., Oliveira S.M., Rodrigues Jr M.T., Bastos R.M., Ferrari J., de Oliveira C.M.A., Kato L., Napolitano H.B., Vencato I., Lariucci C., Structural Studies of 4-Aminoantipyrine Derivatives, *Journal of Molecular Structure*, 2005, **752**:32 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [11]. Nadia A.S., Synthesis of new heterocyclic compounds derived from anthrone and evaluation of their biological activity, *Um-Salama Science Journal*, 2008, **5**:627 [[Google Scholar](#)], [[Publisher](#)]
- [12]. Shaju K.S., Joby T.K., Vidhya T.K., Vinod P.R., Comparative Cyclic Voltammetric studies of (s)-2-(anthracen-9 (10H)-ylideneamino)-3-phenylpropanoic acid and its Cu (II) complex, 2018, **7**:73 [[Google Scholar](#)]
- [13]. Hassan S.A., *Spectroscopic and Structural Study of New Schiff Bases and Their Metal Complexes and Evaluation of Their Biological Activity*, thesis, 2018
- [14]. Hassan S.A., Lateef S.M., Synthesis, structural, thermal and biological studies for new Schiff base derived from Isoniazid and it's complexes with metal ions. *Egyptian Journal of Chemistry*, 2021, **64**:3235 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [15]. Palanimurugan A., Sudharkani V., Kulandaisamy A., Synthesis, spectral, redox and antimicrobial activities of mixed ligand complexes derived from 1-phenyl-2, 3-dimethyl-4-imino-benzylidene-pyrazol-5-one and dimethylglyoxime, *Journal of Nanoscience and Technology*, 2016, **2**:204 [[Google Scholar](#)]
- [16]. Sahib S.K., Synthesis, Characterization and Biological Activity Study of New Schiff Bases Ligands and their Complexes with some Metal Ions, thesis, 2019
- [17]. Ghorab M.M., El-Gazzar M.G., Alsaid M.S., Synthesis, characterization and anti-breast cancer activity of new 4-aminoantipyrine-based heterocycles. *International Journal of Molecular Sciences*, 2014, **15**:7539 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [18]. Muthukkumar M., Malathy M., Rajavel R., Synthesis and spectral characterization of new mononuclear Schiff base complexes derived from 4-aminoantipyrine, 2, 3-butadione and o-

- phenylenediamine. *Der Chem. Sin.*, 2015, **6**:12 [[Google Scholar](#)]
- [19].Světlik J., Prónayová N.A., Frečer V., Ciež D., Three-component reaction and organocatalysis in one: Synthesis of densely substituted 4-aminochromanes, *Tetrahedron*, 2016, **72**:7620 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [20].Al-Shareefi A.N., Salih H.K., Waleed A.S., Synthesis and study of Fe (III), Co (II), Ni (II) and Cu (II) complexes of new Schiff's base ligand derived from 4-amino antipyrine, *Journal of Applicable Chemistry*, 2013, **2**:438 [[Google Scholar](#)]
- [21].Mangaiyarkkarasi P., Antony S.A., Synthesis, Characterization And Biological Significance of Some Novel Schiff Base Transition Metal Complexes Derived from 4-Aminoantipyrine And Dihydropyrimidine of Vanillin. *Journal of Applicable Chemistry*, 2014, **3**:997 [[Google Scholar](#)]
- [22].Mohanambal D., Antony S.A., *Int. J. Pharm. Bio Sci.*, 2014, **5**:600
- [23].Abdulkhader Jailani N.M., Xavier A., Mumtaj A., *Synthesis and Characterisation of Transition Metal Complexes with new Schiff base Ligand. Journal of Advanced Applied Scientific* 2017, **1**:39 [[Google Scholar](#)], [[Publisher](#)]
- [24].Anupama B., Kumari C.G., *Int. J. Res. Chem. Environ.*, 2013, **3**:172 [[Google Scholar](#)],
- [25].Hassan S.A., Lateef S.M., Majeed I.Y., Structural, Spectral and Thermal studies of new bidentate Schiff base ligand type (NO) derived from Mebendazol and 4- Aminoantipyrine and its metal complexes and evaluation of their biological activity, *Research Journal of Pharmacy and Technology*, 2020, **13**:3001 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [26].Onwudiwe D.C., Ajibade P.A., ZnS, CdS and HgS nanoparticles via alkyl-phenyl dithiocarbamate complexes as single source precursors. *International Journal of Molecular Sciences*, 2012, **13**:9502 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [27].Pal T.K., Alam M.A., Hossain M.D., Paul S., Miyatake R., Sheikh M.C., 2, 4-Dichloro-6-[(2-hydroxy-5-methylanilino) methylidene] cyclohexa-2, 4-dienone. *IUCrData*, 2019, **4**:x191401 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [28].Far B.S., Grivani G., Khalaji A.D., Khorshidi M., Gholizadeh A., A new six coordinated oxidovanadium (IV) Schiff base complex: Synthesis, characterization, crystal structure, thermal study and antibacterial activity, *Journal of Molecular Structure*, 2019, **1197**:361 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [29].Joseph V.A., Pandya J.H., Jadeja R.N., Syntheses, crystal structure and biological evaluation of Schiff bases and copper complexes derived from 4-formylpyrazolone, *Journal of Molecular Structure*, 2015, **1081**:443 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]
- [30].Dharamaraj N., Viswanathamurthi P., Natarajan K., Ruthenium (II) complexes containing bidentate Schiff bases and their antifungal activity, *Transition Metal Chemistry*, 2001, **26**:105 [[Crossref](#)], [[Google Scholar](#)], [[Publisher](#)]

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