

## Synthesize characterization and antimicrobial activity study of some transition metal complexes derived from (*E*)-*N*-[(6-bromo-2-hydroxyquinoline-3-yl) methylene]-3-chlorobenzo[b]thiophene-2-carbohydrozide

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### ABSTRACT

A series of coordination complexes of Cu(II), Co(II), Ni(II), Zn(II), Cd(II) and Hg(II) with the Schiff base, (*E*)-*N*-[(6-bromo-2-hydroxyquinoline-3-yl)methylene]-3-chlorobenzo [b] thiophene-2-carbohydrozide have been synthesized and characterized by elemental analyses, electrical conductance, IR spectra, <sup>1</sup>H NMR, thermal, electronic spectra, ESR spectra and magnetic susceptibility measurement. The Schiff base behaves as tridentate ONO donor ligand and forms the complexes of the type ML (metal – ligand). All the complex are colored and non-electrolytes. It is found that Cu(II), Co(II), Ni(II), Zn(II), Cd(II) and Hg(II) complexes have exhibited octahedral geometry. The ligand and their metal complexes have been screened for their antimicrobial activity against *E.coli* and *S.aureus*. Antifungal activity against, *A.niger* and *C.albicans*.

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### KEYWORDS

Schiff base;  
Antimicrobial activity;  
Benzo (b) thiophene.

### INTRODUCTION

The transition metal complexes with potentially tridentate and tetra dentate ligands have evoked much interest in coordination chemistry<sup>[1]</sup>. Several Schiff base metal complexes have been studied because of their industrial and biological application.<sup>[2-4]</sup> Schiff base complexes of transition metals have played a prominent role in coordination chemistry<sup>[5]</sup>. Schiff bases containing poly functional groups offer many practical advantages

and unique structural environmental for complexes<sup>[6]</sup>. The study of transition metal thiophene compounds is an area of great current interest. This is due to the importance of thiophene compounds in hydrodesulphurization reaction in the petroleum industry<sup>[7-9]</sup>. The literature survey reveals that many benzothiophenes<sup>[10-12]</sup> and quinolines<sup>[13-16]</sup> possess good biological activity like antimicrobial, anti-inflammatory, analgesic, and antiviral activities<sup>[17]</sup>. In view of these findings and in continuation of our research work on coor-

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dination chemistry<sup>[18-20]</sup>, we are reporting the synthesis, characterization and antimicrobial activity studies of some transition metal complexes of N [1E]-6-bromo-2-hydroxyquinoline-3-yl] methylene)-3-chloro-4,5-dihydro-1-benzo(b) thiophene-2-carbohydrazide (HL)

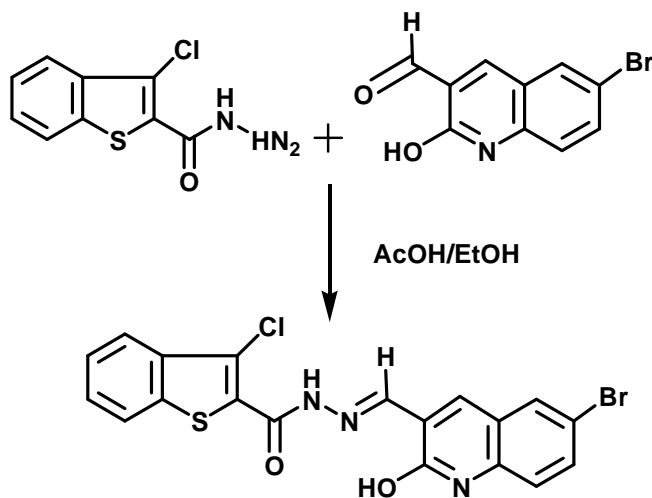


Figure 1

## RESULT AND DISCUSSION

The analytical data of the synthesized ligand HL and its Cu(II), Co(II), Ni(II), Zn(II), Cd(II) and Hg(II) complexes are given in the TABLE 1. The molar conductance of the complexes was measured in DMF at  $10^{-3}$ M concentration. Measured conductance values of these complexes are too low to account for their electrolytic behaviour.

## IR spectra

The important bands in the IR spectra of the ligand as well as complexes together with their assignments are listed in the TABLE 2. The free ligand showed a broad band at  $3432\text{ cm}^{-1}$  due to  $\nu$  (OH) of the ligand which disappeared in all the complexes indicating its involvement in the coordination with the metal ion via deprotonation. The ligand showed medium intensity band at  $3204\text{ cm}^{-1}$  assigned to  $\nu$ (NH) vibration of amide function which is observed in the region  $3298\text{--}3206\text{ cm}^{-1}$  in case of all the complexes indicating the non-involvement of NH in coordination and no enolization of C=O bond during complexation. Strong intensity bands at  $1647\text{ cm}^{-1}$  and  $1562\text{ cm}^{-1}$  are assigned to  $\nu$ (C=O) and  $\nu$ (C=N) of the hydrazone functions of the ligand respectively<sup>[21-22]</sup>. The ligand showed an intense strong band at  $1515\text{ cm}^{-1}$  due to benzo(b)thiophene ring vibration<sup>[23]</sup> and a strong band at  $1279\text{ cm}^{-1}$  due to phenolic  $\nu$ (C-O) vibration respectively. In case of all the complexes the band due to  $\nu$ (C=O) was observed in the region  $1644\text{--}1608\text{ cm}^{-1}$ . The shift of this band towards lower wave number side by  $3\text{--}39\text{ cm}^{-1}$  indicates coordination of the  $\nu$ (C=O) of the amide function to the metal ions.<sup>[24]</sup> The absorption band due to  $\nu$ (C=N) has been shifted to the region of lower wave number by  $4\text{--}27\text{ cm}^{-1}$  and observed in the range  $1558\text{--}1535\text{ cm}^{-1}$  in case of all the complexes can be attributed to coordination of nitrogen of azomethene nitrogen to the metal ions. The band due to phenolic C-O observed at  $1279\text{ cm}^{-1}$

TABLE 1 : Physical and analytical, magnetic susceptibility and molar conductance data HL and its complexes

Compound/Complex	Mol.Wt	M.P. °C	Elemental analysis(%), Found (Cala)					$\mu_{\text{eff}}$ (BM)	$\lambda_{\text{m}}$ ( $\text{ohm}^{-1}\text{ cm}^2\text{ Mol}^{-1}$ )
			M	C	H	N	Cl		
$\text{C}_{19}\text{H}_{11}\text{BrClN}_3\text{O}_2\text{S}$	459	307	-	49.53 (49.56)	2.41 (2.39)	9.12 (9.13)	7.69 (7.60)	-	-
$[\text{Cu}(\text{C}_{19}\text{H}_{14}\text{BrCl}_2\text{N}_3\text{O}_4\text{S})]$	592.9	>300	10.6 (10.62)	38.37 (38.44)	2.37 (2.36)	7.07 (7.08)	11.92 (11.80)	1.95	26
$[\text{Co}(\text{C}_{19}\text{H}_{14}\text{BrCl}_2\text{N}_3\text{O}_4\text{S})]$	588.83	289	9.99 (10.00)	38.67 (38.71)	2.39 (2.37)	7.12 (7.13)	12.02 (11.88)	4.96	25
$[\text{Ni}(\text{C}_{19}\text{H}_{14}\text{BrCl}_2\text{N}_3\text{O}_4\text{S})]$	588.69	>300	9.95 (9.96)	38.71 (38.73)	2.39 (2.37)	7.12 (7.13)	12.02 (11.89)	3.54	23
$[\text{Zn}(\text{C}_{19}\text{H}_{14}\text{BrCl}_2\text{N}_3\text{O}_4\text{S})]$	594.91	285	10.96 (10.98)	38.25 (38.29)	2.37 (2.35)	7.04 (7.05)	11.88 (11.75)	Dia*	16
$[\text{Cd}(\text{C}_{19}\text{H}_{14}\text{BrCl}_2\text{N}_3\text{O}_4\text{S})]$	641.9	>300	17.47 (17.49)	35.46 (35.49)	2.19 (2.17)	6.53 (6.53)	11.02 (10.89)	Dia*	25
$[\text{Hg}(\text{C}_{19}\text{H}_{14}\text{BrCl}_2\text{N}_3\text{O}_4\text{S})]$	729.91	270	27.41 (27.45)	31.18 (31.20)	1.93 (1.91)	5.74 (5.74)	9.69 (9.58)	Dia*	23

TABLE 2 : IR Spectra data (in cm<sup>-1</sup>) ligand (HL) and its complexes

Ligand/Complex	OH/ H <sub>2</sub> O	$\nu_{\text{NH}}$	$\nu_{\text{C=O}}$	$\nu_{\text{C=N}}$	Phenolic $\nu_{\text{C-O}}$	$\nu_{\text{C-S-C}}$	$\nu_{\text{M-O}}$	$\nu_{\text{M-N}}$	$\nu_{\text{M-Cl}}$
Ligand (HL)	3432	3234	1647	1562	1279	1515	--	--	-
Cu-Complex	3422	3251	1631	1540	1314	1517	541	407	317
Co-Complex	3422	3206	1638	1555	1317	1518	512	471	318
Ni-Complex	3381	3266	1640	1558	1316	1517	534	473	316
Zn-Complex	3424	3266	1644	1554	1315	1516	562	467	317
Cd-Complex	3431	3298	1608	1539	1313	1520	550	473	312
Hg - Complex	3431	3270	1609	1535	1314	1516	512	430	317

in the case of ligand has been shifted to higher wave number side by 38-29 cm<sup>-1</sup> and observed in the region 1317-1308 cm<sup>-1</sup> suggesting the coordination of the phenolate ion with the metal ions via deprotonation<sup>[25]</sup>. The presence of broad stretching vibrations in the region 3431-3381 cm<sup>-1</sup> is due to presence of coordinated or lattice water in all the complexes. The new bands appeared in the complexes in the region 516-518 cm<sup>-1</sup> and 407-473 cm<sup>-1</sup> have been assigned to  $\nu(\text{M-O})$  and  $\nu(\text{M-N})$  respectively. The bands in the region 317-312 cm<sup>-1</sup> have been assigned to (M-Cl) bands in all complexes (TABLE 2).

### Electronic spectra

Electronic spectral data of the Co(II), Ni(II) and Cu(II) complexes of the ligand HL were carried out in DMF at 10<sup>-3</sup> molar concentration. The electronic spectrum of Co(II) complex showed three bands observed at 11756 cm<sup>-1</sup>, 16946 cm<sup>-1</sup> and 20655 cm<sup>-1</sup>. due to the  ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{2g}(\text{F})$  ( $\nu_1$ ),  ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{A}_{2g}(\text{F})$  ( $\nu_2$ ) and  ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{2g}(\text{P})$  ( $\nu_3$ ) transitions respectively. These transitions suggests octahedral geometry for Co(II) complex, These assignment are in good agreement with the reported values.<sup>[26]</sup> The electronic spectra of Ni(II) complex under the present investigation exhibited three bands at 11524 cm<sup>-1</sup>, 16354 cm<sup>-1</sup> and 25231 cm<sup>-1</sup> respectively in DMF solution. These bands are assigned to  ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{2g}(\text{F})$  ( $\nu_1$ ),  ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{F})$  ( $\nu_2$ ) and  ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{P})$  ( $\nu_3$ ) transition respectively. The band around 10516 cm<sup>-1</sup> is attributed to a d-d component of Ni(II) complex there by suggesting the octahedral geometry for the Ni(II) complex. The light green colored Cu(II) complex exhibits asymmetric band in the region 16321 – 13459 cm<sup>-1</sup> with maxima at 14366 cm<sup>-1</sup> in an distorted octahedral geometry<sup>[27]</sup>. The broad-

ness of the band may be due to dynamic Jahn-Teller distortion and assigned to  ${}^2\text{T}_{2g} \rightarrow {}^2\text{E}_g$  transition.

### <sup>1</sup>H NMR spectrum of the ligand HL

In the <sup>1</sup>H NMR spectrum (in  $\delta$ ) of the ligand HL, the three singlets observed at 8.72, 12.00 and 12.45 ppm are due to a proton of azomethine function, a proton of amide NH and a proton of OH function attached to quinoline moiety respectively. A multiplet due to eight aromatic protons has appeared in the region 7.15-8.50 ppm.

### <sup>1</sup>H NMR spectrum of Cd (II) complex of the ligand HL

In the <sup>1</sup>H NMR spectrum (in  $\delta$  ppm) of the Zn (II) complex of the ligand HL, the disappearance of peak at 12.45 ppm due to phenolic proton when compared to that of ligand, clearly confirms the deprotonation of the phenolic OH during complexation, A fine singlet appeared at 12.10 ppm is due to NH proton of amide function. The proton due to azomethine function has appeared at 8.7. The aromatic protons have resonated in the region 7.8 – 8.70 ppm. The downfield shift of all the signals indicates the complexation of the ligand with metal ion.

### Mass spectrum of the ligand HL

The ligand molecule in its mass spectrum showed molecular ion peak at  $M^+_{\text{obs}}$  459 (3%), 461 (5%), 463(2%). The molecular ion on simultaneous loss of hydrogen and isocyanate radicals gave a fragment ion peak at  $m/z$  416 (3%), 418 (5%), 420 (2%), which on simultaneous loss of hydrogen and chloride radicals gave another fragment ion peak at  $m/z$  380(4%), 382(6%). This fragment ion on elimination of bromide radical

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gave peak at  $m/z$  301(100%) which is also a base peak. This ion on simultaneous expulsion of  $C_8H_4$  species, HCN molecule and NCO radical gave a fragment ion peak recorded  $m/z$  132(6%). This fragmentation pattern of the ligand is in consistency with its structure.(Figure 2).

### Magnetic susceptibility data

Magnetic susceptibility measurements of the complexes were performed at room temperature. The magnetic moment for Cu(II) complex of the ligand HL is 1.86 BM. The reported values for the mononuclear Cu(II) having no major spin interac-

tion is 1.75-2.20 BM<sup>[28-29]</sup>. Thus the present Cu(II) complex is devoid of any spin interaction with distorted octahedral geometry. In octahedral Co(II) complex the ground state is  $4T_{1g}$  and a large orbital contribution to the magnetic moment is expected. The mixing of the singlet states lowers the magnetic moment. The reported magnetic moment values for the various Co(II) complexes is in the range 4.7- 5.2 BM for octahedral complexes. In the present investigation the observed magnetic moment value for Co(II) complex is 4.96 BM indicates octahedral geometry for this Co(II) complex. For Ni(II) complex the observed magnetic moment value is 3.54 BM which is well within the expected range 2.83-4.0 BM<sup>[30-31]</sup>. for Ni(II) complex with octahedral stereochemistry. The Zn(II), Cd(II), and Hg(II) complexes showed a diamagnetic behavior having a  $d^{10}$  system which is as per expectation.

### ESR spectral studies of the Cu(II) complex of the ligand HL

The X-band ESR spectrum of the powder Cu(II) complex was recorded at room temperature using DPPH as reference standard. One unpaired electron in Cu(II) complex with  $^2B_{1g}$  as ground state lies in  $dx^2-y^2$  orbital and follows the trends  $g_{||} > g_{\perp} > g_e$  ( $g_e = 2.0036$ ) free ion value. The observed  $g_{||} = 2.2867$ ,  $g_{\perp} = 2.0384$  values of the Cu(II) complex under the present study followed the same trend  $g_{||} > g_{\perp} > g_e$  which suggest the presence of unpaired electron in  $dx^2-y^2$  orbital giving octahedral geometry<sup>[32]</sup>. The observed  $G = 4.73$  for the complex under present study evidenced the monomeric nature of the complex<sup>[33]</sup>. This fact is in agreement with its electronic spectral data for octahedral geometry.

### X-ray diffraction studies

The ligand L and its Cd(II) complex have characterized by powder XRD studies with a view to find type of the crystal system. The ligand HL and its Cd(II) complex are chosen for powder XRD study. The XRD data of the ligand and its Cd(II) complex are chosen for powder XRD study. The XRD data of the ligand HL and its Cd(II) given in the (TABLE 3 and 4) respectively. There are 14 reflections ( $2\theta$ ) between 5.166 and 31.247 with maximum at  $2\theta = 5.166$  and  $d$

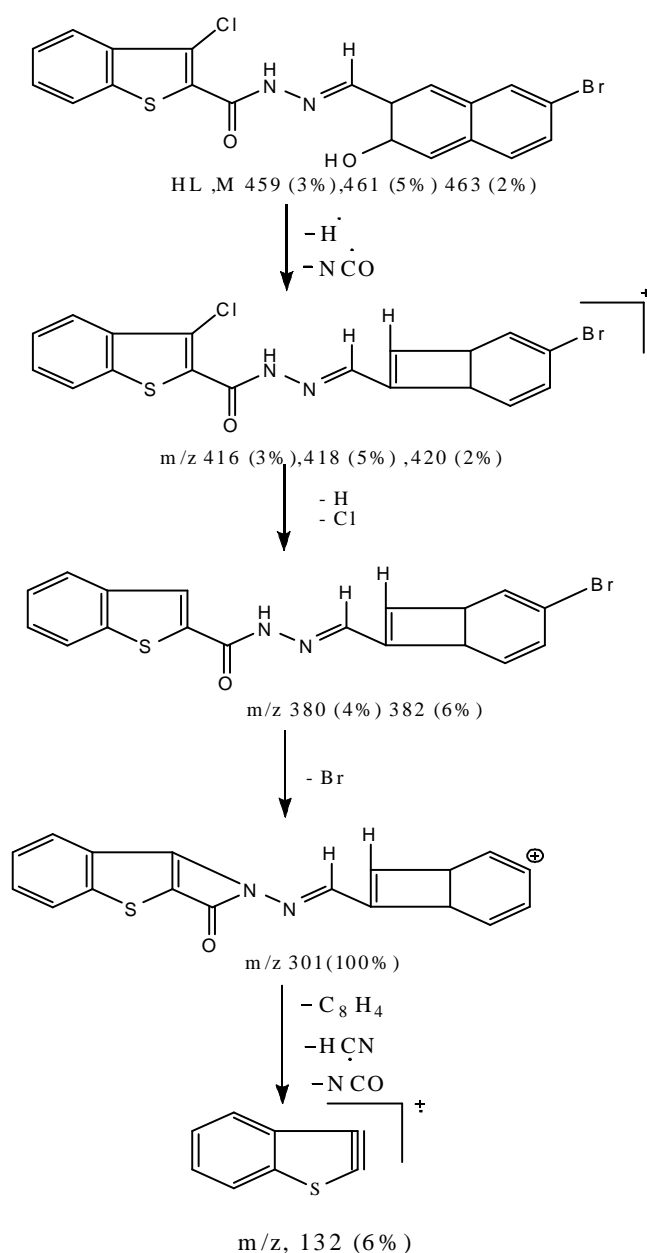


Figure 2

TABLE 3 : Powder x-ray diffraction data of ligand (HL)

Peak No.	2 $\theta$	$\theta$	Sin $\theta$	Sin <sup>2</sup> $\theta$	h <sup>2</sup> +k <sup>2</sup> +l <sup>2</sup>	hkl	D		a in Å <sup>0</sup>
							Calc	Obser	
1	5.166	2.583	0.045066	0.002030	1 0 0	1	17.08	17.093	17.11
2	5.924	2.962	0.051673	0.002670	1 0 0	1	14.901	14.9067	17.09
3	8.451	4.2255	0.073682	0.005429	1 1 0	2	10.450	10.4546	17.09
4	9.752	4.876	0.084999	0.007224	1 1 1	3	9.0589	9.06201	17.16
5	10.252	5.126	0.089346	0.007982	2 0 0	4	8.6181	8.62156	17.13
6	13.542	6.771	0.117901	0.013900	- - -	7	6.5389	6.53320	17.11
7	17.561	8.7805	0.152649	0.023301	3 1 1	11	5.0442	5.04619	17.13
8	19.837	9.9185	0.172247	0.029669	3 2 1	14	4.4703	4.47212	17.11
9	21.947	10.9735	0.190354	0.036235	4 1 1	18	4.5199	4.04669	17.16
10	24.814	12.407	0.214854	0.046162	3 3 2	22	3.5838	3.58527	17.16
11	26.833	13.415	0.232028	0.053837	5 1 0	26	3.3185	3.31990	17.11
12	27.450	13.725	0.237262	0.056293	3 3 3	27	3.2404	3.24664	17.13
13	29.779	14.8895	0.256955	0.066026	4 4 0	32	2.9966	2.99781	17.13
14	31.247	15.6235	0.269314	0.072530	5 3 1	35	2.8591	2.86019	17.11

=17.093 Å in case of ligand HL whereas Cd (II) complex consist 5 reflections (2q) between 11.040 and 26.089 with maxima at  $2q = 23.705$  corresponding to the value of  $d = 3.75038\text{Å}$ . The inter planar spacing (d) has been calculated from the position of intense peaks using Bragg's relation  $n\lambda = 2d \sin q$  (where  $\lambda =$  wave length of X-ray used  $\text{Cu K}\alpha = 1.5406\text{Å}$ ). The observed and calculated values of d are quite consistent (TABLE 3 and 4). The unit cell calculations have been carried out for the cubic system<sup>[34,35]</sup>. The cell parameters have been calculated by using the equation for the cubic system  $\sin^2 q = \lambda^2/4a^2 (h^2 + k^2 + l^2)$ , where  $\lambda^2/4a^2$  is common factor. In the present case, ligand HL and its Cd (II) complex have  $\lambda^2/4a^2 = 0.002030$  and  $0.002925$  respectively. The  $h^2+k^2+l^2$  values are given in the (TABLE 3 and 4). The absence of forbidden numbers 6 in case of ligand and Cd (II) complex indicate that they may belong to hexagonal or tetragonal respectively.

### Thermo gravimetric analysis

The decomposition studies of the Ni(II) complex of ligand HL has been carried out in the thermogram of the  $[\text{Ni}(\text{C}_{19}\text{H}_{14}\text{BrCl}_2\text{N}_3\text{O}_4\text{S})]$ . Loss of  $2\text{H}_2\text{O}, \text{Cl}$  was observed as indicated by inflexion curve at  $113^\circ\text{C}$  with 12.16% weight loss which is in accordance with the theoretical weight loss 12.06%. The resultant intermediate complex under went further degradation and gave another break at  $350^\circ\text{C}$  with weight loss of 25.50% which corresponds to the expulsion of Br, Cl and  $\text{NH}_2$  species from the intermediate complex. The theoretical weight loss for this decomposition was 25.30% agreeing well with the observed value. Third inflection occurred at  $436^\circ\text{C}$  with weight loss of 80.69% which accounts for the loss of  $-\text{C}_{19}\text{H}_8\text{N}_2\text{OS}$  species. This practical weight loss 80.69% of the third stage decomposition is in accordance with the theoretical weight loss of 80.60%. Thereafter the complex showed a gradual

TABLE 4 : Powder x-ray diffraction data of Cd (II) Complexes

Peak No.	2 $\theta$	$\theta$	Sin $\theta$	Sin <sup>2</sup> $\theta$	h <sup>2</sup> +k <sup>2</sup> +l <sup>2</sup>	hkl	D		a in Å <sup>0</sup>
							Calc	Obser	
1	11.040	5.52	0.09619	0.002925	1 0 0	1	8.0049	8.00716	14.23
2	15.300	7.65	0.13312	0.017721	2 1 1	6.05	5.7842	5.78614	14.16
3	16.093	8.0465	0.13997	0.019593	4 2 1	6.69	5.5009	5.50519	14.16
4	23.705	11.8525	0.20539	0.04218	3 2 1	14.42	3.7489	3.75038	14.21
5	26.089	13.0445	0.22570	0.05094	4 1 0	17.41	3.411	3.41277	14.16

TABLE 5 : Thermal decomposition of Ni (II) Complex of ligand HL.

Complex	Stage	Peak temp TG (°C)	Loss of mass (in %)		Probable assignments
			Practical	Theoretical	
Ni(C <sub>19</sub> H <sub>14</sub> C <sub>12</sub> N <sub>3</sub> O <sub>4</sub> Br S)	I	97 °C	12.16	12.06	Ni(C <sub>19</sub> H <sub>14</sub> C <sub>12</sub> N <sub>3</sub> O <sub>4</sub> Br S)
	II	350 °C	25.50	25.30	↓ -2H <sub>2</sub> O, Cl Ni(C <sub>19</sub> H <sub>10</sub> ClN <sub>3</sub> O <sub>2</sub> Br S)
	III	436 °C	80.69	80.60	↓ -Br, Cl NH <sub>2</sub> Ni(C <sub>19</sub> H <sub>8</sub> N <sub>2</sub> O <sub>2</sub> S) ↓ -C <sub>19</sub> H <sub>8</sub> N <sub>2</sub> OS NiO

decomposition up to 822<sup>0</sup>C and onwards. The weight of residue corresponds to the formation of NiO. (TABLE 5).

### Antimicrobial activity

Antimicrobial activity was carried out by the cup-plate method<sup>[36]</sup>. The ligand HL and its Cu (II), Co(II), Ni(II), Zn(II), Cd(II), and Hg(II) complexes have been tested for their antimicrobial activity against *E.coli*, *S.aureus*, and antifungal activity against *A.Niger*, *C.albicans* respectively, at 1mg/mL concentration. The results of the antimicrobial activity have been presented in TABLE 6. The antimicrobial activity ligand HL and its Cu (II), Co(II), Ni(II), Zn(II), Cd(II), and Hg (II) complexes were found to show weak activity with 12-15 mm inhibition against *E.coli* and *S.aureus* whereas Cd(II) complex showed moderate activity with 16 mm and 17 mm inhibition

against the same organisms when compared to the standard drug streptomycin which showed 21 mm and 23 mm inhibition against *E.coli* and *S.aureus*, respectively at the same concentration as that of the test compounds. The antifungal activity results of the ligand and its complexes revealed that the ligand HL showed moderate activity against *A. Niger* and *C.albicans* with 12 mm and 16 mm inhibition respectively. The Cu(II) Co(II), Cd(II), Ni (II), and Hg(II) complexes showed good antifungal activity with 17 mm - 20 mm inhibition against both *A.Niger* and *C.albicans* whereas Ni (II) Zn(II) complex showed moderate activity against both *A.Niger* and *C.albicans* with 09 mm and 12 mm inhibition compared to the standard drug Fluconazole which showed 24 mm and 25 mm inhibition against *A. Niger* and *C.albicans* respectively at the same concentration as that of test compounds.

TABLE 6 : Antimicrobial activity screening data of the ligand (HL) and its complexes

Compound/ complexes	Antimicrobial activity (Zone of inhibition in mm)*			
	Antibacterial activity		Antifungal activity	
	<i>E.coli</i>	<i>S.aureus</i>	<i>A.niger</i>	<i>C. albicans</i>
HL3	13	18	15	16
Cu – complex	13	15	16	15
Co – complex	12	18	17	14
Ni – complex	13	09	10	12
Zn – complex	14	11	09	16
Cd – complex	16	17	15	17
Hg –complex	15	22	20	12
Streptomycin	21	20	-	
Fluconazole	-	-	25	24
DMF (Control)	0	0	0	0

## EXPERIMENTAL MATERIAL AND METHOD

All the chemicals are of reagent grade. Solvents were dried and distilled before use according to standard procedure<sup>[37]</sup>. The precursor 3-chloro-1-benzo(b)thiophene-2-carbohydrazide<sup>[38]</sup> and 6-bromo-2-hydroxyquinoline-3-carboxaldehyde were prepared by literature methods<sup>15</sup>. The metal chlorides used were in their hydrated form.

### Synthesis of the ligand (HL).

An equimolar mixture of 3-chlorobenzo(b)thiophene-2-carbohydrazide (0.001mol) and 6-bromo-2-hydroxyquinoline-3-carboxaldehyde (0.001mol) in ethanol (30mL) were refluxed in presence of catalytic amount of glacial acetic acid (1-2 drops) for about 6 h on water bath. The reaction mixture was cooled to room temperature, the separated Schiff base (HL) was collected by filtration, washed with ethanol, dried and recrystallized from absolute ethanol.

### Preparation of Cu(II),Co(II),Ni(II), Zn(II),Cd(II) and Hg(II) complexes of ligand HL.

To the hot solution of (*E*)-*N*-[(6-bromo-2-hydroxyquinoline-3-yl)methylene]-3-chlorobenzo [b]thiophene-2-carbohydrozide (HL) (0.002 mol) in ethanol (30mL) was added a hot ethanolic solution (10mL) of respective metal(II) chloride (0.002 mol), the reaction mixture was refluxed on a steam bath for 4h, then sodium acetate (0.5 g) was added to it and refluxed for further 2 h. It was then poured in to distilled water. The resulting solid complexes were collected by filtration, washed with sufficient quantity of distilled water, then with hot ethanol to apparent dryness and dried in a vacuum over anhydrous calcium chloride in a desiccator (yield 60-70 %) (TABLE 1).

### Physical measurements

IR Spectra of the synthesized ligand and its complexes were recorded as KBr pellets on Perkins-Elmer Spectrum One FT-IR spectrometer. <sup>1</sup>H NMR spectra were recorded on a Bruker Avance 400 MHz spectrometer. UV-vis spectra of the complexes were recorded on Elico-SL 164

spectrometer in the range 200-1000 nm in DMF solution ( $1 \times 10^{-3}$ M). Mass spectrum of ligand acquired on MASPEC system and. Elemental analysis was obtained from HERAEUS C, H, N,O rapid analyzer and metal analyses were carried out by following the standard methods. ESR measurement was carried out on a BRUKER Bio Spin GmbH spectrometer working at microwave frequency of 9.903 GHz. The experiment was carried out by using diphenylpicrylhydrazyl (DPPH) as reference with field set at 3200 gauss. Magnetic susceptibility was determined by the Faraday method using a model 300 Lewis coil force magnetometer of tesla field strength at room temperature. The instrument was calibrated with Hg [Co(SNC)<sub>4</sub>]<sup>[39]</sup>.

## CONCLUSION

In the light of above discussion we have proposed octahedral geometry for Cu(II), Co(II), Ni(II), Zn(II), Cd(II) and Hg(II) complexes. The ligand behaves as ONO tridentate chelating agent coordinating through the deprotonation of hydroxyl group, carbonyl group and azomethene nitrogen of ligand in all the complexes. The analytical data, electronic, spectral, magnetic susceptibility, IR, <sup>1</sup>H NMR, ESR and TGA spectral data revealed mononuclear nature of the complexes. The ligand as well as its metal complexes were found to be less active against the bacteria whereas moderately active against the fungi. All the complexes showed good activity as compared with their ligand.

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