CHAPTER-4
SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL STUDIES OF Mn(II), Co(II), Cu(II) COMPLEXES WITH (N-((7-HYDROXY-4-METHYL-2-OXO-2H-CHROMEN-8-YL)METHYLENE)BENZOHYDRAZIDE LIGAND
4.1. INTRODUCTION

Many complexes of hydrazones ligand with metal ions have been investigated as models for active sites of enzymes\textsuperscript{1,2}, DNA-cleavage systems\textsuperscript{3,4}, as antibacterial\textsuperscript{5-7} and anticancer\textsuperscript{8} drugs. They also provide useful magnetic materials and have a wide range of catalytic applications, such as in polymerization\textsuperscript{7}, olefin oxidation\textsuperscript{6} and Suzuki-Miyaura coupling\textsuperscript{7,8}. Mixed N/S/O donor Schiff bases have been used extensively in catalysis\textsuperscript{9}. Most of the N/S/O donor Schiff base ligands have nitrogen with phosphorous\textsuperscript{10} or oxygen\textsuperscript{11} atoms as the biological active ligand units\textsuperscript{12,13}. Several metal ions and their complexes exhibit antidiabetic effects\textsuperscript{15,16}.

A broad area of medicinal applications of metal complexes of N/S/O donor coumarins has been investigated\textsuperscript{17}. It was found that in some cases the metal complexes obtained revealed higher biological activity than their ligands\textsuperscript{18,19}. N, O donor coumarin derivatives possess more than one reactive group (OH, COOH, NO\textsubscript{2}) and act as monodentate, bidentate chelate, mono- and bidentate bridging ligands toward lanthanide and transition metal ions\textsuperscript{20}. This ability has reached an increasing interest since the therapeutic efficacy\textsuperscript{21}, the specific biological activity and pharmacological properties of the coumarin compounds were found to be improved upon complexation with metal ions\textsuperscript{22}.

Recently, Chakravarty and co-workers\textsuperscript{23} have utilized O, N, and S-Schiff bases as ligands for models of copper-containing enzymes. Elzbieta Budzisz et al.,\textsuperscript{24} reported that the medical properties of naturally occurring N, O, S donor compounds such as chromones, flavonoids and coumarins have been well known for many years. However, the discoveries that their complexes with metal ions are more effective than coumarins and flavonoids alone changed the course of drug research have reviewed
systematically. Patel K.D. et al.,\textsuperscript{25} have reported a series of Co(II), Ni(II), Cu(II) and Mn(II) complexes with ligands containing O, O donor atoms of 6-bromo-3-(3-(4-chlorophenyl)acryloyl)-2H-chromen-2-one, Ciprofloxacin and various transition metal are discussed.

In view of these facts, author have studied the synthesise, characterization, alpha-glucosidase, antioxidant and antimicrobial activities of Mn(II), Co(II) and Cu(II) complexes with N, O, O donor (7-hydroxy-4-methyl-2-oxo-2H-chromen-8-yl) methylene) benzohydrazide ligand. The structural characterization of metal complexes have been studied on the basis of elemental analysis, conductivity measurements, magnetic susceptibility, infrared, electronic spectral studies, X-ray diffraction, ESR and thermal analysis.

4.2. EXPERIMENTAL

The details of synthesis and characterization of (7-hydroxy-4-methyl-2-oxo-2H-chromen-8-yl)methylene)benzohydrazide (ligand-$L_1$) have been discussed in chapter-3. In this section, the synthesis and characterization of Mn(II), Co(II) and Cu(II) complexes and their biological studies have been illustrated.

The following metal salts are used for the preparation of various complexes;

1. Manganese(II) chloride (MnCl$_2$.6H$_2$O)
2. Manganese(II) bromide (MnBr$_2$.4H$_2$O)
3. Manganese(II) sulphate (MnSO$_4$.7H$_2$O)
4. Manganese(II) acetate (Mn(Ac)$_2$.4H$_2$O)
5. Manganese(II) perchlorate (Mn(ClO$_4$)$_2$.6H$_2$O)
6. Manganese(II) nitrate (Mn(NO$_3$)$_2$.6H$_2$O)
7. Cobalt(II) chloride (CoCl$_2$.6H$_2$O)
8. Cobalt(II) bromide (CoBr$_2$.4H$_2$O)
9. Cobalt(II) sulphate (CoSO$_4$.7H$_2$O)  
10. Cobalt(II) acetate (Co(CH$_3$COO)$_2$.4H$_2$O)  
11. Cobalt(II) perchlorate (Co(ClO$_4$)$_2$.6H$_2$O)  
12. Cobalt(II) nitrate (Co(NO$_3$)$_2$.6H$_2$O)  
13. Copper(II) chloride (CuCl$_2$.6H$_2$O)  
14. Copper(II) bromide (CuBr$_2$.4H$_2$O)  
15. Copper(II) sulphate (CuSO$_4$.7H$_2$O)  
16. Copper(II) acetate (Cu(CH$_3$COO)$_2$.4H$_2$O)  
17. Copper(II) perchlorate (Cu(ClO$_4$)$_2$.6H$_2$O)  
18. Copper(II) nitrate (Cu(NO$_3$)$_2$.6H$_2$O)

4.3. SYNTHESIS AND CHARACTERIZATION OF COMPLEXES WITH (7-HYDROXY-4-METHYL-2-OXO-2H-CHROMEN-8-YL) METHYLENE) BENZOHYDRAZIDE (LIGAND-L$_1$)

4.3.1. Preparation of Mn(II), Co(II) and Cu(II) complexes with (ligand-L$_1$) 

To the ethanolic solution of (7-hydroxy-4-methyl-2-oxo-2h-chromen-8-yl)methylene)benzohydrazide (1 mmol) were added a hot ethanolic solution (10 ml) of respective metal salts (1 mmol) as depicted in reaction protocol of scheme 4.1. The reaction mixture were refluxed on a water bath for 2-3 hours to get clear colored solution$^{26,27}$. On cooling, the suspended solid complexes were allowed to settle and collected by filtration, washed with sufficient quantity of distilled water and then with little hot ethanol to apparent dryness and dried in a vacuum over anhydrous calcium chloride. The sulphate and acetate complexes were prepared by refluxing in 1:1 (alcoholic: aqueous) media. Yield; 63-91%.
4.4. RESULTS AND DISCUSSION

i) Stoichiometry

All the synthesized complexes are coloured, stable in air, non-hygrosopic, have high melting points and are insoluble in water, but soluble in DMF, DMSO, THF and acetonitrile. The analytical datas are depicted in table 4.1. These data suggest that the metal:ligand stichometry ratio is 1:2 and they can be corresponds to general formulae;

\[ M(L_1)_2X_2 \cdot nH_2O \] (where \( n = 2, 4, 6... \), \( M= \text{Mn(II), Co(II) and Cu(II)} \), \( X = Cl^-, Br^-, NO_3^-, ClO_4^-, Ac^- \) and \( \frac{1}{2}SO_4^{2-} \))

The stichiomteric composition of the Mn(II), Co(II) and Cu(II) complexes with ligand (L_{1}) are depicted as is shown below;
M=Mn(II), Co(II), Cu(II).

Proposed structure of metal complexes with O and N donor atoms.
ii) Electrical conductance measurements

Electric conductivity measurements are helpful to know whether the anions of the metal salts remain inside or outside the coordination sphere of the central metal atom.

The conductivity measurements in organic solvents for the characterization of coordination compounds have been discussed in detail in a review by Geary\textsuperscript{28}. Molar conductance values of different electrolytic systems in different solvents are available as standard references.

The molar conductance data (45.3-65.2 mhos mol\textsuperscript{-1} cm\textsuperscript{2}) presented in table 4.1. compared with that of the reported\textsuperscript{29} data, which suggested that all metal complexes in 10\textsuperscript{-3} M DMSO were found to be electrolytic in nature and the anions are not coordinated with metal ions. Thus it may be concluded that the anions are present outside the coordination sphere.
Table 4.1. Elemental analysis, melting point, molecular weight and molar conductance data of ligand and their metal complexes

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Complexes</th>
<th>Yield (%)</th>
<th>Mel.pt. (°C)</th>
<th>Mol. Wt.</th>
<th>Elemental analysis</th>
<th>Calc. (found) (%)</th>
<th>Ohm(^{-1}) cm(^2) mol(^{-1})</th>
</tr>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>M</td>
<td>C</td>
<td>H</td>
</tr>
<tr>
<td>1</td>
<td>[Mn(C(<em>{18}H</em>{14}N_{2}O_{4})]Cl(_2).4H(_2)O</td>
<td>81</td>
<td>220-225</td>
<td>697.41</td>
<td>7.18 (7.20)</td>
<td>61.01 (61.09)</td>
<td>3.86 (3.90)</td>
</tr>
<tr>
<td>2</td>
<td>[Mn(C(<em>{18}H</em>{14}N_{2}O_{4})]Br(_2).4H(_2)O</td>
<td>78</td>
<td>231-233</td>
<td>731.23</td>
<td>6.41 (6.46)</td>
<td>60.99 (61.02)</td>
<td>3.25 (3.26)</td>
</tr>
<tr>
<td>3</td>
<td><a href="SO(_4)">Mn(C(<em>{18}H</em>{14}N_{2}O_{4})</a>.H(_2)O</td>
<td>68</td>
<td>251-253</td>
<td>724.41</td>
<td>6.17 (6.20)</td>
<td>51.15 (51.17)</td>
<td>2.02 (2.04)</td>
</tr>
<tr>
<td>4</td>
<td><a href="ClO(_4)">Mn(C(<em>{18}H</em>{14}N_{2}O_{4})</a>(_2).6H(_2)O</td>
<td>85</td>
<td>261-265</td>
<td>743.40</td>
<td>8.41 (8.45)</td>
<td>56.21 (56.27)</td>
<td>3.41 (3.15)</td>
</tr>
<tr>
<td>5</td>
<td><a href="CH(_3)COO">Mn(C(<em>{18}H</em>{14}N_{2}O_{4})</a>(_2).4H(_2)O</td>
<td>79</td>
<td>270-273</td>
<td>708.31</td>
<td>6.98 (7.05)</td>
<td>61.28 (61.34)</td>
<td>3.06 (3.10)</td>
</tr>
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<td>6</td>
<td><a href="NO(_3)">Mn(C(<em>{18}H</em>{14}N_{2}O_{4})</a>.5H(_2)O</td>
<td>75</td>
<td>259-261</td>
<td>724.81</td>
<td>7.05 (7.11)</td>
<td>60.81 (60.85)</td>
<td>3.81 (3.84)</td>
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<tr>
<td>7</td>
<td>[Co(C(<em>{18}H</em>{14}N_{2}O_{4})]Cl(_2).6H(_2)O</td>
<td>76</td>
<td>247-251</td>
<td>701.02</td>
<td>8.24 (8.27)</td>
<td>55.23 (55.24)</td>
<td>2.44 (2.45)</td>
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<td>[Co(C(<em>{18}H</em>{14}N_{2}O_{4})]Br(_2).H(_2)O</td>
<td>81</td>
<td>286-287</td>
<td>706.42</td>
<td>8.04 (8.09)</td>
<td>59.48 (59.51)</td>
<td>2.95 (3.01)</td>
</tr>
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<td><a href="SO(_4)">Co(C(<em>{18}H</em>{14}N_{2}O_{4})</a>.5H(_2)O</td>
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<td>291-293</td>
<td>715.31</td>
<td>7.85 (7.87)</td>
<td>57.34 (57.37)</td>
<td>2.34 (2.35)</td>
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<td>10</td>
<td><a href="ClO(_4)">Co(C(<em>{18}H</em>{14}N_{2}O_{4})</a>(_2).6H(_2)O</td>
<td>91</td>
<td>278-282</td>
<td>723.12</td>
<td>7.06 (7.11)</td>
<td>61.71 (61.75)</td>
<td>3.04 (3.06)</td>
</tr>
<tr>
<td>No.</td>
<td>Formula</td>
<td>mp (°C)</td>
<td>T (°C)</td>
<td>Yield (%)</td>
<td>KBr (%)</td>
<td>KNO₃ (%)</td>
<td>KSCN (%)</td>
</tr>
<tr>
<td>-----</td>
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</tr>
<tr>
<td>11</td>
<td><a href="CH%E2%82%83COO">Co(C₁₈H₁₄N₂O₄₂)</a>₂·4H₂O</td>
<td>72</td>
<td>255-258</td>
<td>712.06</td>
<td>7.01</td>
<td>52.23</td>
<td>3.14</td>
</tr>
<tr>
<td>12</td>
<td><a href="NO%E2%82%83">Co(C₁₈H₁₄N₂O₄₂)</a>₂·6H₂O</td>
<td>67</td>
<td>267-268</td>
<td>702.24</td>
<td>6.49</td>
<td>51.34</td>
<td>2.87</td>
</tr>
<tr>
<td>13</td>
<td>[Cu(C₁₈H₁₄N₂O₄₂)]Cl₂·6H₂O</td>
<td>76</td>
<td>234-236</td>
<td>705.45</td>
<td>8.18</td>
<td>55.23</td>
<td>3.71</td>
</tr>
<tr>
<td>14</td>
<td>[Cu(C₁₈H₁₄N₂O₄₂)]Br₂·H₂O</td>
<td>68</td>
<td>222-226</td>
<td>735.80</td>
<td>7.21</td>
<td>49.23</td>
<td>3.11</td>
</tr>
<tr>
<td>15</td>
<td><a href="SO%E2%82%84">Cu(C₁₈H₁₄N₂O₄₂)</a>₂·5H₂O</td>
<td>63</td>
<td>248-251</td>
<td>707.21</td>
<td>7.01</td>
<td>48.23</td>
<td>2.71</td>
</tr>
<tr>
<td>16</td>
<td><a href="ClO%E2%82%84">Cu(C₁₈H₁₄N₂O₄₂)</a>₂·6H₂O</td>
<td>73</td>
<td>261-264</td>
<td>722.45</td>
<td>6.85</td>
<td>61.34</td>
<td>2.56</td>
</tr>
<tr>
<td>17</td>
<td><a href="CH%E2%82%83COO">Cu(C₁₈H₁₄N₂O₄₂)</a>₂·3H₂O</td>
<td>78</td>
<td>271-274</td>
<td>718.51</td>
<td>7.58</td>
<td>62.74</td>
<td>2.71</td>
</tr>
<tr>
<td>18</td>
<td><a href="NO%E2%82%83">Cu(C₁₈H₁₄N₂O₄₂)</a>₂·5H₂O</td>
<td>81</td>
<td>267-273</td>
<td>725.50</td>
<td>7.91</td>
<td>59.31</td>
<td>3.05</td>
</tr>
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</table>
iii) Magnetic susceptibility measurements

The magnetic susceptibility measurements of the complexes were obtained at room temperature using Gouy’s balance. Magnetic susceptibility measurements were done in order to find out the effective magnetic moment of each metal atom in the complexes. The number of unpaired electrons ‘n’ possessed by the metal ion can be determined from the effective magnetic moment of the metal ion. From the knowledge of the number of unpaired electrons, it is possible to infer the valence state of the metal ion \( \mu_{\text{eff}} = \left[ \frac{n(n+1)}{2} \right]^{1/2} \text{BM} \) in a complex.

The transition element of manganese having electronic configuration [Ar]3d\(^5\) 4s\(^2\) and Mn(II) ion being a d\(^5\) system has five unpaired electron in 3d shell and its complexes are expected to have magnetic moments close to the spin only value of 5.65-5.91 BM, irrespective of the bond type involved. The magnetic moment of mononuclear Mn(II) complexes usually lies in the range 5.62-5.95 BM have no major interaction between the unpaired electron on different manganese ions and essentially temperature independent.

The transition element of copper having electronic configuration [Ar]3d\(^{10}\) 4s\(^1\) and Cu(II) ion being a d\(^9\) system has one unpaired electron in 3d shell and its complexes are expected to have magnetic moments close to the spin only value of 1.70–2.20 BM, irrespective of the bond type involved. The magnetic moment of mononuclear copper(II) complexes usually lies in the range 1.70–2.20 BM have no major interaction between the unpaired electron on different copper ions and essentially temperature independent.

The cobalt having electronic configuration [Ar]3d\(^7\) 4s\(^2\) and Co(II) ion being a d\(^7\) system has three unpaired electron in 3d shell and its complexes are expected to
have magnetic moments close to the spin only value of 4.3–5.0 BM, irrespective of the bond type involved. In all other cases the symmetry will be lowered due to Jahn-Teller distortion prevailing in the cobalt(II) ion. The magnetic moment of mononuclear cobalt(II) complexes usually lies in the range 4.3–5.0 BM. It can be seen from the data presented in table 4.2.

iv) Electronic spectral measurements

Electronic spectral studies data of complexes are presented in table 4.2.

Manganese(II) ion which has five d electrons and The ground term arising from the $t^3_{2g} e^2_g$ configuration in an octahedral field is $^4T_{1g}$ and having three spin allowed d-d transitions as follows:

\[ \nu_1 = ^4T_{1g} (F) \rightarrow ^4T_{2g} (F), \]
\[ \nu_2 = ^4T_{1g} \rightarrow ^4A_{2g} (F), \]
\[ \nu_3 = ^4T_{1g} (F) \rightarrow ^4T_{1g} (P). \]

The lowest energy transition $^4T_{1g} (F) \rightarrow ^4T_{2g}$ was assigned near infrared region and the band in visible region near 18000-17500 cm$^{-1}$ is assigned to the $^4T_{1g} (F) \rightarrow ^4T_{1g} (P)$. The $^4T_{1g} \rightarrow ^4A_{2g}$ transition is due to a band near 18000 cm$^{-1}$. The $^4A_{2g}$ state is derived from a $t^3_{2g} e^4_g$ configuration. The $^4T_{1g} \rightarrow ^4A_{2g} (F)$ essentially a two electron process and for this region it should be weaker than the other transition. This weakness combined with closeness of the $^4T_{1g} (F) \rightarrow ^4T_{1g} (P)$ band results in the $^4T_{1g} \rightarrow ^4A_{2g}$ transition being unobserved and the spectra as shown in figure 4.2.

The electronic spectral data of the new manganese(II) complexes are depicted in Table-4.3 and Figure-4.4. the band located in the region 17150-19550 cm$^{-1}$ are assigned to $^4T_{1g} \rightarrow ^4A_{2g} (F)$ and $^4T_{1g} (F) \rightarrow ^4T_{1g} (P)$ transition respectively. These results revels that the manganese ion complexes is in octahedral field with ligand.
Manganese(II) complexes are shown three expected transition in tetrahedral nature these three transitions are as follows;

\[ ^4A_2 \rightarrow ^4T_2, \quad ^4A_2 \rightarrow ^4T_1 \text{ (F)} \text{ and } \quad ^4A_2 \rightarrow ^4T_1 \text{ (P)}. \]

The cobalt element having the ground term arising from the \( t^3 \) \( 2g \) \( e^2 \) \( g \) configuration in an octahedral field is \( ^4T_{1g} \) and having three spin allowed d-d transitions as follows;

\[ v_1= ^4T_{1g} \text{ (F)} \rightarrow ^4T_{2g} \text{ (F)} \]
\[ v_2= ^4T_{1g} \rightarrow ^4A_{2g} \text{ (F)} \]
\[ v_3= ^4T_{1g} \text{ (F)} \rightarrow ^4T_{1g} \text{ (P)} \]

The lowest energy transition \( ^4T_{1g} \text{ (F)} \rightarrow ^4T_{2g} \) was assigned near infrared region and the band in visible region near 17000-17500 cm\(^{-1}\) is assigned to the \( ^4T_{1g} \text{ (F)} \rightarrow ^4T_{1g} \text{ (P)} \). The \( ^4T_{1g} \rightarrow ^4A_{2g} \) transition is due to a band near 14000 cm\(^{-1}\). The \( ^4A_{2g} \) state is derived from a \( t^3 \) \( 2g \) \( e^2 \) \( g \) configuration. The \( ^4T_{1g} \rightarrow ^4A_{2g} \text{ (F)} \) essentially a two electron process and for this region it should be weaker than the other transition. This weakness combined with closeness of the \( ^4T_{1g} \text{ (F)} \rightarrow ^4T_{1g} \text{ (P)} \) band results in the \( ^4T_{1g} \rightarrow ^4A_{2g} \) transition being unobserved.

The electronic spectral data of the new cobalt(II) complexes are depicted in table-4.2 and spectra in figure 4.4, the band located in the region 15150-15950 cm\(^{-1}\) are assigned to \( ^4T_{1g} \rightarrow ^4A_{2g} \text{ (F)} \) and \( ^4T_{1g} \text{ (F)} \rightarrow ^4T_{1g} \text{ (P)} \) transition respectively. These results revels that the cobalt(II) ion complexes is in octahedral field with lignad\(^{32}\).

Cobalt(II) complexes are shown three expected transition in tetrahedral nature these three transitions are as follows;

\[ ^4A_2 \rightarrow ^4T_2, \quad ^4A_2 \rightarrow ^4T_1 \text{ (F)} \text{ and } \quad ^4A_2 \rightarrow ^4T_1 \text{ (P)}. \]
The copper(II) ion have the ground term arising from the \( t_{2g}^6 e_{g}^3 \) configuration in an octahedral field is \( ^2E_g \) and the energy level diagram of a six coordinated copper(II) ion is as follows;

![Energy level diagram](image)

The copper(II) complexes display three bands in the visible region, the bands at 14150-15550, 19500-21000 and 25000-25650 cm\(^{-1}\) can be attributed to \( ^2B_{1g} \rightarrow ^2A_{1g} \), \( ^2B_{1g} \rightarrow ^2B_{2g} \) and \( ^2B_{1g} \rightarrow ^2E_g \) transitions. The rest of copper complexes show two bands in visible region at 14000-15650 cm\(^{-1}\) being assigned for the \( ^2B_{1g} \rightarrow ^2A_{1g} \) and \( ^2B_{1g} \rightarrow ^2B_{2g} \) transitions\(^3\), the electronic spectra of complex was shown in Figure 4.5.

**Electronic transitions of complexes**

<table>
<thead>
<tr>
<th>Complexes</th>
<th>( \lambda_{max} \text{ cm}^{-1} )</th>
<th>Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mn(II) complexes</td>
<td>14390-15674</td>
<td>( ^2T_{2g} \leftrightarrow ^2E_g )</td>
</tr>
<tr>
<td></td>
<td>24531-26041</td>
<td>Charge transfer</td>
</tr>
<tr>
<td></td>
<td>34246-35758</td>
<td>ligand</td>
</tr>
<tr>
<td>Co(II) complexes</td>
<td>14706-16357</td>
<td>( ^2T_{2g} \leftrightarrow ^2E_g )</td>
</tr>
<tr>
<td></td>
<td>24691-25874</td>
<td>Charge transfer</td>
</tr>
<tr>
<td></td>
<td>32250-35174</td>
<td>ligand</td>
</tr>
<tr>
<td>Cu(II) complexes</td>
<td>14925-15687</td>
<td>( ^2T_{2g} \leftrightarrow ^2E_g )</td>
</tr>
<tr>
<td></td>
<td>24000-25897</td>
<td>Charge transfer</td>
</tr>
<tr>
<td></td>
<td>31948-34516</td>
<td>ligand</td>
</tr>
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</table>
**Table 4.2.** Electronic spectral and magnetic susceptibility data of metal complexes

<table>
<thead>
<tr>
<th>Sl.No.</th>
<th>Complexes</th>
<th>Electronic spectra (cm(^{-1}))</th>
<th>Racah parameters</th>
<th>eff(\mu) (BM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>[Mn(C(<em>{18})H(</em>{14})N(<em>{2})O(</em>{4}))(_2)]Cl(_2).4H(_2)O</td>
<td>15100 21000 28986 35660</td>
<td>876 0.91</td>
<td>5.52</td>
</tr>
<tr>
<td>2</td>
<td>[Mn(C(<em>{18})H(</em>{14})N(<em>{2})O(</em>{4}))(_2)]Br(_2).4H(_2)O</td>
<td>15100 21500 28906 35562</td>
<td>855 0.87</td>
<td>5.41</td>
</tr>
<tr>
<td>3</td>
<td><a href="SO(_4)">Mn(C(<em>{18})H(</em>{14})N(<em>{2})O(</em>{4}))(_2)</a>H(_2)O</td>
<td>15886 21375 28674 35580</td>
<td>786 0.68</td>
<td>5.08</td>
</tr>
<tr>
<td>4</td>
<td><a href="ClO(_4)">Mn(C(<em>{18})H(</em>{14})N(<em>{2})O(</em>{4}))(_2)</a>(_2).6H(_2)O</td>
<td>15931 17550 29761 36705</td>
<td>809 0.94</td>
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Figure 4.2. Electronic spectrum of complex (1)

Figure 4.3. Electronic spectrum of complex (2)
Figure 4.4. Electronic spectrum of complex (7)

Figure 4.5. Electronic spectrum of complex (13)
v) Infrared spectral studies

Generally, a comparison of the infrared spectrum of the ligand and its metal complexes will be of much help to find out the ligand through which the atoms are attached to the metal atom and an attention has been focused on a limited number of bands which provide considerable structural information in order to suggest the most probable manner of coordination of the ligands with metal ions. When the anion of the metals are polyatomic like, perchlorides, sulphates etc., the IR spectral studies gives useful information regarding the anionic groups are coordinated with metal atoms are not and if coordinated, to determine the particular atom involved in coordination.

The important IR bands of ligand exhibited a broad band at 3525-3550 cm\(^{-1}\) which is attributed to the presence of \(\nu\)(O-H) and strong band at 3425-3450 cm\(^{-1}\) which is represents the presence of \(\nu\)(N-H) group. The remaining strong band at 1325, 1597 and 1475 cm\(^{-1}\) assigned to \(\nu\)(C-O), amide \(\nu\)(C=O) carbonyl and \(\nu\)(C=N) vibrations respectively.

In the spectrum of manganese complexes of ligand (L\(_1\)), a strong band at 3425-3450 cm\(^{-1}\) which is represents the presence of \(\nu\)(N-H) group in free ligand moiety which is shifted towards the higher frequency region with large extent of decrease in intensity it is due to the coordination with metal ion through amino group. The broad band at 3560-3515 cm\(^{-1}\) which is due to \(\nu\)(O-H) group present in the ligand is shifted to higher frequency of about 50-75 cm\(^{-1}\) this changes of frequencies revels that the \(\nu\)(O-H) group involved in coordination with metal atom. The new bands at 465-435 cm\(^{-1}\) and 565-530 cm\(^{-1}\) are assign to \(\nu\)(Mn-N) and \(\nu\)(Mn-O) respectively\(^{34}\). The important IR spectrum of complexes are presented in figure 4.6 and figure 4.7.
In the spectrum of cobalt(II) complexes of ligand \((L_1)\), a strong band at 3225-3350 cm\(^{-1}\) which represents the presence of \(\nu(\text{N-H})\) group in free ligand moiety which is shifted towards the higher frequency region with large extent of decrease in intensity it is due to the coordination with metal ion through amino group. Cobalt complexes show a broad peak in the region 3521-3555 cm\(^{-1}\) which is due to hydroxyl group and followed by a sharp peak in the range 1621-1627 cm\(^{-1}\). These peaks can be assigned to \(\nu(\text{O-H})\) stretching vibrations. This is also supported by thermogravimetric analysis. The new peak appeared in the far infra-red spectrum of the complexes at 550-500 cm\(^{-1}\) and 480-470 cm\(^{-1}\) is assigned to \(\nu(\text{Co-N}), \nu(\text{Co-O})\) respectively, confirms the bonding of nitrogen of azomethine group and oxygen atom of the hydroxyl group to metal ions. The important infrared frequencies exhibited by the ligands and its metal complexes are shown in table 4.3 and infrared spectrum is presented in figure 4.8.

In the spectrum of copper(II) complexes of ligand \((L_1)\), a strong band at 3200-3350 cm\(^{-1}\) which represents the presence of \(\nu(\text{N-H})\) group in free ligand moiety which is shifted towards the higher frequency region with large extent of decrease in intensity it is due to the coordination with metal ion through amino group. Copper complexes show a broad peak in the region 3505-3550 cm\(^{-1}\) which is due to hydroxyl group and followed by a sharp peak in the range 1625-1627 cm\(^{-1}\). These peaks can be assigned to \(\nu(\text{O-H})\) stretching. This is also supported by thermogravimetric analysis. The new peak appeared in the far infra-red spectrum of the complexes at 495-485 cm\(^{-1}\) and 475-460 cm\(^{-1}\) is assigned to \(\nu(\text{Cu-N}), \nu(\text{Cu-O})\) respectively, confirms the bonding of nitrogen of azomethine group and oxygen atom of the hydroxyl group to metal ions. The spectrum of complex is presented in figure 4.9. The important infrared frequencies of the complexes are presented in table 4.3.


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<td>[Mn(C(<em>{18})H(</em>{14})N(_2)O(_4))(_2)] (NO(_3))(_2).4H(_2)O</td>
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Figure 4.6. IR spectrum of complex (1)

Figure 4.7. IR spectrum of complex (4)
Figure 4.8. IR spectrum of complex (8)

Figure 4.9. IR spectrum of complex (15)
vi) X-ray powder diffraction studies

X-ray diffraction study is useful for elucidate crystal structure of metal complexes. Generally the crystal structure of a substance determine the diffraction pattern of that substance or more specifically, the shape and size of the unit cell determines the angular positions of the relative intensities of the lines. Since structure determines the diffraction pattern, from the diffraction pattern one can arrive at the structure.

The X-ray diffraction pattern of the complexes [Co(C$_{18}$H$_{14}$N$_2$O$_4$)$_2$]Cl$_2$.6H$_2$O and [Cu(C$_{18}$H$_{14}$N$_2$O$_4$)$_2$]Cl$_2$.6H$_2$O were recorded between 5-50° and 6-70°C. All the peaks have been indexed and their $\sin^2\theta$ values compared with calculated ones and also the unit cell parameters like a, b, c, $\alpha$, $\beta$ and $\gamma$ have been calculated.

The X-ray diffraction data of the complexes are presented in table 4.4. The diffractogram records 21 reflections as shown in figure 4.10. between 5 and 50° (2$\theta$) with maximum reflection $2\theta = 19.75^\circ$ which corresponds to $d = 4.3561$ Å with hkl values of (340) all the peaks have been indexed$^{35,36}$ and their $\sin^2\theta$ values compared with calculated ones, which reveal that there is a good agreement between calculated and observed values of $\sin^2\theta$. The unit cell has been determined by the trial error method$^{37-39}$.

The unit parameters obtained for [Co(C$_{18}$H$_{14}$N$_2$O$_4$)$_2$]Cl$_2$.6H$_2$O complex are $a = 7.2560$ Å, $b = 26.8604$ Å, $c = 17.5204$ Å, $\alpha = 90^\circ$, $\beta = 90^\circ$ and $\gamma = 90^\circ$ with the cell volume $V = 3817.2461$ Å$^3$. Substitution of the cell volume in the following formula gives the theoretical value of density equal to 1.1245 g cm$^{-3}$. For calculating density we have used the following equation:

$$\text{Density} = \frac{nxM_w}{NxV},$$
Where, \( n = 6 \)

\( M_w = \) Molecular weight of the complex,

\( N = \) Avagadro’s number, \( 6.023 \times 10^{23} \),

\( V = \) Cell volume.

The experimental value of density of the complex has been found (1.6547 g of the complex occupies 1.4 cm\(^3\) of volume) to be 1.1483 g cm\(^3\) which is in good agreement within the limits of experimental error. Based upon these data, the complex is suggested to be prthorhombic in nature.

The X-ray diffraction pattern of the complexes \([Cu(C_{18}H_{14}N_{2}O_{4})_2]Cl_2.6H_2O\) are presented in figure 4.11. This complex were recored 23 reflections between 6 and 70\(^\circ\) with maximum reflections at \(\theta = 27.45\) which corresponds to \(d = 3.0456 \text{ Å}\). All the peaks have been indexed\(^{40,41}\) and their \(\sin^2\theta\) values compared with the calculated one with the help of computer. The unit cell parameters found to be \(a = 3.189 \text{ Å}, b = 4.831 \text{ Å}, c = 3.6524 \text{ Å}, \alpha = 90.78^\circ, \beta = 84.62^\circ\) and \(\gamma = 89.40^\circ\) and the unit cell volume \(V = 81.1247 \text{ Å}^3\). Based upon data presented in table 4.5, the complex suggested to be orthorhombic in nature.
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Figure 4.10. X-ray diffraction pattern of [Co(C\textsubscript{18}H\textsubscript{14}N\textsubscript{2}O\textsubscript{4})\textsubscript{2}]Cl\textsubscript{2}.6H\textsubscript{2}O

Figure 4.11. X-ray diffraction pattern of [Cu(C\textsubscript{18}H\textsubscript{14}N\textsubscript{2}O\textsubscript{4})\textsubscript{2}]Cl\textsubscript{2}.6H\textsubscript{2}O
### Table 4.5. X-ray data of \([\text{Cu(C}_{18}\text{H}_{14}\text{N}_{2}\text{O}_{4})_{2}]\text{Cl}_2\cdot\text{6H}_2\text{O}\) complexes

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vii) Thermogravimetric analysis

The thermal behavior of metal complexes was presented in figure 4.12-4.13. The thermogravimetric and the temperature characterization data such as $T_0$ (temperature of onset of decomposition), $T_{10}$ (temperature for 10% weight loss) and IPDT (Integral Procedure Decomposition Temperature) of the degradation process of complexes are represented as following:

**Thermal degradation of manganese(II) complexes**

The TG curve of Mn(II) complexes shows three significant temperature transitions of weight loss termed at three stages of thermal degradation process indicating dehydration, decomposition of organic moiety and inorganic ligands. The percentage, weight losses, degradation temperature and nature of the decomposition products are compiled in table 4.6.

**First stage of Thermal degradation**

The manganese(II) complexes (1-6) starts decompositions in the temperature range 95-200$^\circ$ C upto 210$^\circ$ C, the weight loss was very gradual is termed as first stage of degradation. The TGA values of complexes show weight loss in the temperature range 12.25-13.35% corresponding to loss of two water molecules$^{42}$. The expulsion of water molecules in this temperature range indicate that they are present outside the coordination sphere$^{43,44}$.

**Second stage of thermal degradation**

The weight loss in the range 380-480$^\circ$ C in TGA curve of complexes is termed as the second stage of thermal degradation. The weight loss in the range 54.46-55.12% which may be attributed to the oxidation of organic moiety ($L_1$). The DTG peak of this stage is observed in the range 420$^\circ$ C the height of the DTG peaks at this
temperature is also indicating the rate of mass change in the degradation process as shown in figure 4.12.

**Third stage of thermal degradation**

The weight loss in the range 550-640° C in TGA curve of the complexes is termed as the third stage of thermal degradation. The percentage of weight loss may be attributed to the anions of the inorganic ligand (X) after this stage the TGA curve become a level line. The final product was respective oxide. In the basis of weight loss percentage the three significant steps of thermal degradation can be formulated as:

\[
[Mn(L_1)_2]_2(X)_2(Y)_2 \xrightarrow{95-180^\circ C} [Mn(L)](X)_2 \xrightarrow{380-480^\circ C} Mn(X)_2 \xrightarrow{550-640^\circ C} MnO
\]

**Thermal degradation of cobalt(II) complexes**

The TG curve of Co(II) complexes(7-12) of L_1 shows three significant three stages of thermal degradation process indicating dehydration, decomposition of organic moiety an inorganic ligands. The percentage, weight losses, degradation temperature and nature of the decomposition products are compiled in table 4.6.

**First stage of thermal degradation**

The weight loss occurs in the range 98-198° C upto 210° C. In TGA curve of complexes is termed as the first stage of thermal degradation. In these steps the weight loss is in the range 09.24-10.24%. The weight loss corresponds due to dehydration of uncoordinated water molecule and has been found to be equivalent to 2 moles of water as shown in figure 4.13. The DTG peaks of this stage is observed in the range 240° C and height of the DTG peaks at about temperature gives the rate of mass change is in agreement with the theoretical values of the complexes.
Second stage of thermal degradation

The weight loss in the range 350-480° C upto 500° C in TGA curve of complexes is termed as the second stage of thermal degradation. The weight loss in the range 53.47-54.17% which may be attributed to the oxidation of organic moiety (L₁)⁴⁷. the DTG peak of this stage is observed in the range 425° C, the height of the DTG peaks at this temperature is also indicating the rate of mass change in the degradation process.

Third stage of thermal degradation

The third stage of degradation occurs in the range 550-620° C in this stage the percentage of weight loss is in the range of 20.14-21.04% which may be attributed to loss of unbounded inorganic ligand to cobalt(II) ion, where TG curve become a level line. In all stages the experimental values of weight loss are in agreement with the experimental value. Thus the thermal studies of complexes revels that the three stages of degradation in all Co(II) complexes of L₁ which are confirmed the presence of non-coordinated water in the complexes⁴⁸ and also show complexes posses 1:2 (metal:ligand) stoichiometry.

Thermal degradation of copper(II) complexes

The TG curve of Cu(II) complexes of L₁ shows three significant temperature transitions of weight loss termed at three stages of thermal degradation process indicating dehydration, decomposition of organic moiety an inorganic ligands. The percentage, weight losses, degradation temperature and nature of the decomposition products are compiled in table 4.6.
First stage of thermal degradation

The weight loss occurs in the range 100-205° C. In TGA curve of complexes is termed as the first stage of thermal degradation. In these steps the weight loss is in the range 8.24-9.07%. The weight loss corresponds due to dehydration of uncoordinated water molecule and has been found to be equivalent to 2 moles of water. The DTG peaks of this stage is observed in the range 150-180° C and height of the DTG peaks at about temperature gives the rate of mass change is in agreement with the theoretical values of the complexes.

Second stage of thermal degradation

The weight loss in the range 365-490° C in TGA curve of complexes is termed as the second stage of thermal degradation. The weight loss in the range 51.28-52.11% which may be attributed to the oxidation of organic moiety (L1). The DTG peak of this stage is observed in the range 440° C, the height of the DTG peaks at this temperature is also indicating the rate of mass change in the degradation process.

Third stage of thermal degradation

The third stage of degradation occurs in the range 560-635° C in this stage the percentage of weight loss is in the range of 15.71-16.22% which may be attributed to loss of unbounded inorganic ligand to copper(II) ion, where TG curve become a level line. In all stages the experimental values of weight loss are in agreement with the experimental value. Thus the thermal studies of complexes revels that the three stages of degradation in all Cu(II) complexes of L1 which are confirmed the presence of non-coordinated water in the complexes and also show complexes posses 1:2 (metal:ligand) stoichiometry.
Table 4.6. Thermogravimetric characteristics of metal complexes

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<th>Process of degradation</th>
<th>Temp. Range(°C)</th>
<th>DTG peak Temp.</th>
<th>Product Degradised</th>
<th>No. of moles</th>
<th>Weight Cal.</th>
<th>Weight Expt.</th>
<th>Residue(MO)</th>
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<td>H₂O</td>
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<tr>
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<td>165</td>
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<td>Dehydration</td>
<td>98-198</td>
<td>165</td>
<td>H₂O</td>
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<td>11.01</td>
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<tr>
<td><a href="NO%E2%82%83">Co(C₁₈H₁₄N₂O₄)₂</a>₂·6H₂O</td>
<td>Dehydration</td>
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<td>09.14</td>
<td>08.87</td>
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Table 4.6. Continued……..

| Decomposition of L₁ | 350-445 | 410 | L₁ | 2 | 47.34 | 46.98 |
| Decomposition of SO₄²⁻ | 530-625 | 598 | NO₃⁻ | 2 | 19.89 | 19.05 |

| [Cu(C₁₈H₁₄N₂O₄)₂]Cl₂.6H₂O | Dehydration | 115-187 | 170 | H₂O | 2 | 08.24 | 08.01 | 10.97 | 10.24 | CuO |
| Decomposition of L₁ | 335-450 | 387 | L₁ | 2 | 51.28 | 50.34 |
| Decomposition of SO₄²⁻ | 550-648 | 605 | Cl⁻ | 2 | 15.71 | 15.05 |

| [Cu(C₁₈H₁₄N₂O₄)₂]Br₂.5H₂O | Dehydration | 115-198 | 160 | H₂O | 2 | 09.56 | 09.01 | 10.05 | 09.87 | CuO |
| Decomposition of L₁ | 325-420 | 359 | L₁ | 2 | 48.21 | 47.58 |
| Decomposition of SO₄²⁻ | 540-632 | 580 | Br⁻ | 2 | 18.47 | 18.24 |

| [Cu(C₁₈H₁₄N₂O₄)₂](SO₄)₂.5H₂O | Dehydration | 120-205 | 165 | H₂O | 2 | 10.24 | 09.87 | 09.02 | 08.65 | CuO |
| Decomposition of L₁ | 350-437 | 380 | L₁ | 2 | 47.21 | 46.24 |
| Decomposition of SO₄²⁻ | 520-620 | 578 | SO₄²⁻ | 2 | 20.37 | 19.54 |

| [Cu(C₁₈H₁₄N₂O₄)₂](ClO₄)₂.6H₂O | Dehydration | 105-185 | 165 | H₂O | 2 | 09.64 | 09.04 | 10.14 | 09.74 | CuO |
| Decomposition of L₁ | 329-420 | 395 | L₁ | 2 | 47.14 | 46.31 |
| Decomposition of SO₄²⁻ | 510-638 | 617 | ClO₄⁻ | 2 | 15.24 | 14.89 |

| [Cu(C₁₈H₁₄N₂O₄)₂](CH₃COO)₂.3H₂O | Dehydration | 105-195 | 170 | H₂O | 2 | 11.24 | 10.54 | 10.74 | 10.05 | CuO |
| Decomposition of L₁ | 326-440 | 410 | L₁ | 2 | 48.57 | 48.05 |
| Decomposition of SO₄²⁻ | 548-658 | 597 | Ac⁻ | 2 | 21.37 | 20.87 |

| [Cu(C₁₈H₁₄N₂O₄)₂](NO₃)₂.5H₂O | Dehydration | 100-190 | 180 | H₂O | 2 | 10.25 | 09.78 | 09.80 | 09.12 | CuO |
| Decomposition of L₁ | 330-450 | 397 | L₁ | 2 | 49.54 | 49.01 |
| Decomposition of SO₄²⁻ | 560-665 | 598 | NO₃⁻ | 2 | 19.89 | 19.10 |
Table 4.7. Temperature characteristics and integral procedure decomposition temperature (IPDT) of metal complexes

<table>
<thead>
<tr>
<th>Sl.No.</th>
<th>Complexes</th>
<th>Temperature in °C</th>
<th>IPDT</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$T_0$</td>
<td>$T_{10}$</td>
</tr>
<tr>
<td>1</td>
<td>[Mn(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$]Cl$_2$·4H$_2$O</td>
<td>94</td>
<td>195</td>
</tr>
<tr>
<td>2</td>
<td>[Mn(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$]Br$_2$·4H$_2$O</td>
<td>115</td>
<td>207</td>
</tr>
<tr>
<td>3</td>
<td><a href="SO$_4$">Mn(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$</a>$_2$·H$_2$O</td>
<td>108</td>
<td>198</td>
</tr>
<tr>
<td>4</td>
<td><a href="ClO$_4$">Mn(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$</a>$_2$·6H$_2$O</td>
<td>107</td>
<td>214</td>
</tr>
<tr>
<td>5</td>
<td><a href="CH$_3$COO">Mn(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$</a>$_2$·4H$_2$O</td>
<td>120</td>
<td>247</td>
</tr>
<tr>
<td>6</td>
<td><a href="NO$_3$">Mn(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$</a>$_2$·4H$_2$O</td>
<td>109</td>
<td>201</td>
</tr>
<tr>
<td>7</td>
<td>[Co(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$]Cl$_2$·6H$_2$O</td>
<td>100</td>
<td>225</td>
</tr>
<tr>
<td>8</td>
<td>[Co(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$]Br$_2$·H$_2$O</td>
<td>120</td>
<td>230</td>
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<tr>
<td>9</td>
<td><a href="SO$_4$">Co(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$</a>$_2$·5H$_2$O</td>
<td>98</td>
<td>227</td>
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<tr>
<td>10</td>
<td><a href="ClO$_4$">Co(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$</a>$_2$·6H$_2$O</td>
<td>105</td>
<td>220</td>
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<tr>
<td>11</td>
<td><a href="CH$_3$COO">Co(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$</a>$_2$·4H$_2$O</td>
<td>108</td>
<td>207</td>
</tr>
<tr>
<td>12</td>
<td><a href="NO$_3$">Co(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$</a>$_2$·6H$_2$O</td>
<td>107</td>
<td>214</td>
</tr>
<tr>
<td>13</td>
<td>[Cu(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$]Cl$_2$·6H$_2$O</td>
<td>120</td>
<td>241</td>
</tr>
<tr>
<td>14</td>
<td>[Cu(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$]Br$_2$·H$_2$O</td>
<td>109</td>
<td>205</td>
</tr>
<tr>
<td>15</td>
<td><a href="SO$_4$">Cu(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$</a>$_2$·5H$_2$O</td>
<td>105</td>
<td>225</td>
</tr>
<tr>
<td>16</td>
<td><a href="ClO$_4$">Cu(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$</a>$_2$·6H$_2$O</td>
<td>120</td>
<td>230</td>
</tr>
<tr>
<td>17</td>
<td><a href="CH$_3$COO">Cu(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$</a>$_2$·H$_2$O</td>
<td>101</td>
<td>220</td>
</tr>
<tr>
<td>18</td>
<td><a href="NO$_3$">Cu(C$<em>{18}$H$</em>{14}$N$_2$O$_4$)$_2$</a>$_2$·5H$_2$O</td>
<td>120</td>
<td>230</td>
</tr>
</tbody>
</table>
Figure 4.12. TGA and DTA spectrum of complex (1)

Figure 4.13. TGA and DTA spectrum of complex (7)
viii) ESR spectral studies

A representative spectra of the \([\text{Cu(C}_{18}\text{H}_{14}\text{N}_{2}\text{O}_{4})_2]\text{Cl}_2\cdot6\text{H}_2\text{O}\) as shown in figure 4.14 and ESR data are illustrated in table 4.8. A fine structure observed for this complexes with \(g||, g\perp, g_{iso}\) and \(G\) values have been found to be 2.113, 2.104, 2.041 and 2.1024 respectively. It is evident that the unpaired electron lies predominantly in the \(d_{x^2−y^2}\) orbital of Cu(II) ion. Based on this observation Cu(II) complex has distorded octahedral geometry\(^{51}\).

**Table 4.8. ESR data of copper(II) complexes**

| Complexes                          | \(g||\) | \(g\perp\) | \(g_{iso}\) | \(G\)  |
|------------------------------------|---------|------------|-------------|--------|
| \([\text{Cu(C}_{18}\text{H}_{14}\text{N}_{2}\text{O}_{4})_2]\text{Cl}_2\cdot6\text{H}_2\text{O}\) | 2.113   | 2.104      | 2.041      | 2.1024 |
| \([\text{Cu(C}_{18}\text{H}_{14}\text{N}_{2}\text{O}_{4})_2]\text{Br}_2\cdot\text{H}_2\text{O}\)  | 2.051   | 2.082      | 2.15       | 2.20   |
| \([\text{Cu(C}_{18}\text{H}_{14}\text{N}_{2}\text{O}_{4})_2]\text{SO}_4\cdot5\text{H}_2\text{O}\) | 2.047   | 2.058      | 2.17       | 2.14   |

**Figure 4.14.** ESR spectrum of \([\text{Cu(C}_{18}\text{H}_{14}\text{N}_{2}\text{O}_{4})_2]\text{Br}_2\)
4.4. BIOLOGICAL STUDIES

i) **In-vitro α-glucosidase inhibition activity**

Alpha-Glucosidase inhibition data (percentage inhibition at 50, 100 and 200 μM and IC₅₀ values) for complexes and ligand are presented in table 4.9. The results of inhibition of α-glucosidase by newly synthesized ligand and their complexes at various concentrations are also presented graphically in figure 4.15. A perusal of the data reveals that the complexes of 7, 13, and 14 shows effective alpha-glucosidase activity and L₁, 2, 10, 16 and 17 exhibits good activity. The remaining complexes of 4, 6, 9, 3, 5, 8, 11, 12, 15 and 18 show less activity than the ligand. Complexes of 7, 13 and 14 show effective α-glucosidase inhibition activity⁵²,⁵³ compared to the standard compound (acarbose) and corresponding ligand-L₁⁵⁴.

The reason of this effective changes in alpha-glucosidase activity is electrostatic interactions between the positively charged complexes molecules and the negatively charged N/S/O containing ligand moiety would bring the complex molecules into close proximity, resulting in aggregation of the complex molecules through metal–metal and/or π–π interaction. As the glucose concentration increases, more ligand moieties would be converted into the anionic form⁵⁵, leading to an increase in local density of the anionic sites, and hence the increased electrostatic property of the complexes molecules that led to the growth of the MLCT absorption shoulder and the drop in the absorption bands centered at different absorption wavelength⁵⁶,⁵⁷.
### Table 4.9. α-glucosidase activity of the ligand and its metal complexes

<table>
<thead>
<tr>
<th>Sl.No.</th>
<th>Compounds</th>
<th>IC$_{50}$ (µM)</th>
<th>50 µM</th>
<th>100 µM</th>
<th>150 µM</th>
<th>200 µM</th>
</tr>
</thead>
<tbody>
<tr>
<td>L1</td>
<td>C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>[Mn(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$<em>{2}$]Cl$</em>{2}$.4H$_{2}$O</td>
<td>0.090</td>
<td>0.102</td>
<td>0.0198</td>
<td>0.321</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>[Mn(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$<em>{2}$]Br$</em>{2}$.4H$_{2}$O</td>
<td>0.091</td>
<td>0.108</td>
<td>0.191</td>
<td>0.266</td>
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</tr>
<tr>
<td>3</td>
<td><a href="SO$_%7B4%7D$">Mn(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$_{2}$</a>$<em>{2}$.H$</em>{2}$O</td>
<td>0.013</td>
<td>0.095</td>
<td>0.101</td>
<td>0.179</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td><a href="ClO$_%7B4%7D$">Mn(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$_{2}$</a>$<em>{2}$.6H$</em>{2}$O</td>
<td>0.014</td>
<td>0.071</td>
<td>0.105</td>
<td>0.215</td>
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</tr>
<tr>
<td>5</td>
<td><a href="CH$_%7B3%7D$COO">Mn(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$_{2}$</a>$<em>{2}$.4H$</em>{2}$O</td>
<td>0.090</td>
<td>0.102</td>
<td>0.101</td>
<td>0.114</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td><a href="NO$_%7B3%7D$">Mn(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$_{2}$</a>$<em>{2}$.4H$</em>{2}$O</td>
<td>0.041</td>
<td>0.100</td>
<td>0.157</td>
<td>0.198</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>[Co(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$<em>{2}$]Cl$</em>{2}$.6H$_{2}$O</td>
<td>0.102</td>
<td>0.185</td>
<td>0.371</td>
<td>0.454</td>
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</tr>
<tr>
<td>8</td>
<td>[Co(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$<em>{2}$]Br$</em>{2}$.H$_{2}$O</td>
<td>0.154</td>
<td>0.0971</td>
<td>0.058</td>
<td>0.031</td>
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<td>9</td>
<td><a href="SO$_%7B4%7D$">Co(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$_{2}$</a>$<em>{2}$.5H$</em>{2}$O</td>
<td>0.041</td>
<td>0.098</td>
<td>0.157</td>
<td>0.198</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td><a href="ClO$_%7B4%7D$">Co(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$_{2}$</a>$<em>{2}$.6H$</em>{2}$O</td>
<td>0.090</td>
<td>0.109</td>
<td>0.181</td>
<td>0.124</td>
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<td><a href="CH$_%7B3%7D$COO">Co(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$_{2}$</a>$<em>{2}$.4H$</em>{2}$O</td>
<td>0.081</td>
<td>0.100</td>
<td>0.105</td>
<td>0.181</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td><a href="NO$_%7B3%7D$">Co(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$_{2}$</a>$<em>{2}$.6H$</em>{2}$O</td>
<td>0.078</td>
<td>0.187</td>
<td>0.183</td>
<td>0.204</td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>[Cu(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$<em>{2}$]Cl$</em>{2}$.6H$_{2}$O</td>
<td>0.112</td>
<td>0.156</td>
<td>0.277</td>
<td>0.457</td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>[Cu(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$<em>{2}$]Br$</em>{2}$.H$_{2}$O</td>
<td>0.098</td>
<td>0.121</td>
<td>0.241</td>
<td>0.429</td>
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<td>15</td>
<td><a href="SO$_%7B4%7D$">Cu(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$_{2}$</a>$<em>{2}$.5H$</em>{2}$O</td>
<td>0.087</td>
<td>0.0981</td>
<td>0.198</td>
<td>0.241</td>
<td></td>
</tr>
<tr>
<td>16</td>
<td><a href="ClO$_%7B4%7D$">Cu(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$_{2}$</a>$<em>{2}$.6H$</em>{2}$O</td>
<td>0.092</td>
<td>0.118</td>
<td>0.267</td>
<td>0.345</td>
<td></td>
</tr>
<tr>
<td>17</td>
<td><a href="CH$_%7B3%7D$COO">Cu(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$_{2}$</a>$<em>{2}$.H$</em>{2}$O</td>
<td>0.098</td>
<td>0.119</td>
<td>0.249</td>
<td>0.351</td>
<td></td>
</tr>
<tr>
<td>18</td>
<td><a href="NO$_%7B3%7D$">Cu(C$<em>{18}$H$</em>{14}$N$<em>{2}$O$</em>{4}$)$_{2}$</a>$<em>{2}$.5H$</em>{2}$O</td>
<td>0.075</td>
<td>0.086</td>
<td>0.157</td>
<td>0.274</td>
<td></td>
</tr>
<tr>
<td>Std</td>
<td>Acarbose</td>
<td>0.091</td>
<td>0.114</td>
<td>0.251</td>
<td>0.427</td>
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</tbody>
</table>
ii) Antioxidant Activity

Evaluation of antioxidant activity for the newly synthesized complexes was done by using in vitro assays such as 2,2-diphenyl-1-picryl-hydrazyl (DPPH) radical scavenging activity. The antioxidant properties were expressed as 50% inhibitory concentration (IC$_{50}$) values are illustrated in Table 4.10.

The DPPH radical scavenging evaluation is a standard assay in antioxidant activity studies and offers a rapid technique for screening the radical scavenging activity (RSA) of specific compounds$^{58}$. The reaction of synthesized complexes with stable DPPH free radical indicates their free radical scavenging ability. Initially, ligand (L$_1$) exhibited considerable activity, this could be due to the presence of hydroxyl and electron donating group in ligand moiety$^{59,60}$. Further, coordination with metal ions gave the significant enhancement of activity. Majority of the tested complexes (1-18) showed moderate to high activity. Dominant RSA was observed for copper complexes (13), (14) and (18) this may be presence of copper metal ion which is effective metal for antioxidant activity$^{61}$, presence of electron withdrawing groups.
present in complex. Antioxidant activity of these compounds is related with their electron- or hydrogen-donating ability to DPPH radical, so that it becomes stable diamagnetic molecules. The graphical represented as shown in figure 4.16.

**Table 4.10.** Antioxidant activity (IC₅₀) of the ligand and its metal complexes

<table>
<thead>
<tr>
<th>Compounds</th>
<th>IC₅₀ (µM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>L₁</td>
<td>89±0.03</td>
</tr>
<tr>
<td>1</td>
<td>79±0.13</td>
</tr>
<tr>
<td>2</td>
<td>67±0.09</td>
</tr>
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<td>3</td>
<td>42±0.53</td>
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<tr>
<td>4</td>
<td>54±0.21</td>
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<td>5</td>
<td>84±0.04</td>
</tr>
<tr>
<td>6</td>
<td>95±0.31</td>
</tr>
<tr>
<td>7</td>
<td>51±0.11</td>
</tr>
<tr>
<td>8</td>
<td>78±0.21</td>
</tr>
<tr>
<td>9</td>
<td>50±0.34</td>
</tr>
<tr>
<td>10</td>
<td>48±0.04</td>
</tr>
<tr>
<td>11</td>
<td>64±0.11</td>
</tr>
<tr>
<td>12</td>
<td>84±0.16</td>
</tr>
<tr>
<td>13</td>
<td>105±0.08</td>
</tr>
<tr>
<td>14</td>
<td>114±0.17</td>
</tr>
<tr>
<td>15</td>
<td>65±0.10</td>
</tr>
<tr>
<td>16</td>
<td>74±0.13</td>
</tr>
<tr>
<td>17</td>
<td>69±0.17</td>
</tr>
<tr>
<td>18</td>
<td>127±0.03</td>
</tr>
<tr>
<td>Ascorbic acid</td>
<td>59±0.05</td>
</tr>
</tbody>
</table>
iii) **Antimicrobial activity**

The antibacterial activities of newly synthesized compounds (L₁, 1 - 18) were determined by well plate method. In this work, Escherichia coli ATCC 25922, Staphylococcus aureus ATCC 25923, and Pseudomonas aeruginosa ATCC 27853 were used to investigate the activity.

The test compounds were dissolved in dimethyl sulfoxide (DMSO) at concentrations of 1 and 0.5 mg/mL. The antibacterial screening revealed that some of the tested complexes showed good inhibition against various tested microbial strains as illustrated in table 4.11. Complexes 4, 7, 9 and 15 possessed considerable antibacterial activity. Whereas, compounds of L₁, 2, 5, 11, 14 and 18 exhibited moderate activity.

After complexation with ligand accounted for the enhanced activity. The results indicated that among the tested compounds 4, 7, 9 and 15 showed excellent activity against P. aeruginosa and E. coli at concentrations of 1 and 0.5 mg/mL compared to standard drug Choroamphenicol.
The activity is considerably affected by halogens substituents and electron withdrawing capacity of compounds. compounds L₁, 4, 10, 14 and 17 showed similar activity as that of standard, against E. coli at 1 and 0.5 mg/mL concentrations. The remaining compounds 1, 3, 6, 8, 12, 13 and 16 very least activity against all of the three tested bacterial strains. The antibacterial figure and bar graph represented in figure 4.17 and figure 4.18 respectively.

**Table 4.11.** Antibacterial activity of the ligand and its metal complexes
(minimum inhibitory concentration $\times 10^{-2}$ M) (in mm)

<table>
<thead>
<tr>
<th>Compounds</th>
<th>E. coli</th>
<th>S. aureus</th>
<th>P. aeruginosa</th>
</tr>
</thead>
<tbody>
<tr>
<td>L₁</td>
<td>5.4</td>
<td>7.1</td>
<td>4.9</td>
</tr>
<tr>
<td>1</td>
<td>0.5</td>
<td>1.0</td>
<td>0.6</td>
</tr>
<tr>
<td>2</td>
<td>4.1</td>
<td>11.0</td>
<td>0.5</td>
</tr>
<tr>
<td>3</td>
<td>0.4</td>
<td>-</td>
<td>0.2</td>
</tr>
<tr>
<td>4</td>
<td>7.0</td>
<td>10.2</td>
<td>7.2</td>
</tr>
<tr>
<td>5</td>
<td>5.1</td>
<td>7.2</td>
<td>4.2</td>
</tr>
<tr>
<td>6</td>
<td>1.4</td>
<td>1.8</td>
<td>0.9</td>
</tr>
<tr>
<td>7</td>
<td>0.81</td>
<td>10.8</td>
<td>11.5</td>
</tr>
<tr>
<td>8</td>
<td>0.59</td>
<td>1.58</td>
<td>1.34</td>
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**Figure 4.17.** Antibacterial activity of the ligand and its metal complexes

**Figure 4.18.** Antibacterial activity of the ligand and its metal complexes
Antifungal activity

Newly synthesized compounds (L₁, 1-18) are also screened for their antifungal activity against Aspergillus flavus MTCC 3306, Chrysosporium keratinophilum MTCC 3017 and Candida albicans MTCC 2827.

The compounds were dissolved in dimethyl sulphoxide (DMSO) and antifungal activity was determined by well plate method at concentration of 1 and 0.5 mg/mL. Among the tested compounds, compound L₁, 1, 7, 13 possessing the enhanced antifungal activity against C. albicans compared with standard, fluconazole and as well as other compounds table 4.12. Whereas compounds 4-6, 8-12 and 14-18 showed moderate to good activity against C. albicans but slightly less than that of standard.

The enhanced activity of the complexes over the ligand can be explained on the basis of chelation theory. It is known that chelation makes the ligand a more powerful and potent bactericidal agent, thus killing more of the bacteria than the ligand.

The enhancement in the activity may be rationalized on the basis that ligands mainly possess an azomethine (C=N) bond. It has been suggested that ligands with hetero donor atoms (nitrogen and oxygen) inhibit enzyme activity, since the enzymes that require these groups for their activity appear to be especially more susceptible to deactivation by metal ions on coordination.

It is observed that, in a complex, the positive charge of the metal ion is partially shared with the hetero donor atoms (nitrogen and oxygen) present in the ligand, and there may be π-electron delocalization over the whole chelating
The antifungal figure and bar graph represented in Figure 4.19 and Figure 4.20 respectively.

**Table 4.12.** Antifungal activity of the ligand and its metal complexes (minimum inhibitory concentration × 10⁻² M)(in mm)

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<th>Compounds</th>
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<th>C. albicans</th>
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**Figure 4.19.** Antifungal activity of the ligand and its metal complexes

**Figure 4.20.** Antifungal activity of the ligand and its metal complexes
CONCLUSION

The hydrazone ligand is a tridentate, coordinated through the azomethine nitrogen, phenolic oxygen and benzo-hydrazide oxygen, confirmed by analytical, spectral, magnetic, and thermal studies. On the basis of various physic-chemical techniques, it is tentatively proposed that, the metal complexes have octahedral geometry. The newly synthesized compounds were evaluated for their in-vitro \( \alpha \)-glucosidase inhibition activity, antioxidant and antimicrobial activity. Among the complexes 1b, 1g, and 1h demonstrated effective \( \alpha \)-glucosidase inhibition activity. Almost all complexes exhibits good antioxidant activity. Whereas in antifungal studies of ligand and complexes exhibits moderate activity in comparison with their corresponding parent ligand and standard drug.
REFERENCES


