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### Synthesis Characterization and Biological Studies on Drug Metal Complexes

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#### Abstract

Transition metal complexes of gabapentin {1-(aminomethyl) cyclohexane acetic acid (1-AMCA)}, acebutolol {N-(p-acetyl-4) 2-hydroxy-3-((1-methyl ethyl) amino)propoxy phenyl-butanamide, N-(PA-4-2-HMEPPB)} and t-leucine (TL) were prepared and characterized by elemental analysis, conductance, magnetic moments and spectroscopic data. In these complexes ligand chelates through O-H, C=O and N-H functional group of the drug molecule. The complexes were formulated as  $ML_2$  (1:2) and  $M_2L_2$  (2:2) type. All the complexes were found to be six-coordinated. The geometry appears to be simple octahedral. The drug molecules and complexes were tested for their antimicrobial and antifungal activity against *Escherichia coli*, *Staphylococcus aureus*, *Aspergillus niger* and *Aspergillus flavous*.

Key- Words: Transition metal complexes, antimicrobial, gabapentin (1-AMCA), acebutolol N-(PA-4-2-HMEPPB) and t-leucine (TL)

#### Introduction

Transition metal complexes derived from drugs have been among the most widely studied coordination compounds<sup>1-5</sup>, since they are becoming increasingly important as biochemical<sup>6</sup>, analytical<sup>7</sup> and antimicrobial reagents<sup>8</sup>. These complexes containing metal ions are active in many biological processes. Copper, iron, zinc and cobalt exhibit great biological activity when complexes with certain metal-proteins, such complexes participating in oxygen transport, electron transfer reactions or the storage of ions<sup>9</sup>, have created enormous interest in the study of systems containing these metals<sup>10</sup>. In some drug-metal chelates, it has been observed that minor changes in the structure of the drugs containing hard soft donor atoms, viz., nitrogen, sulphur and/or oxygen markedly affected the activity of the complexes<sup>11-12</sup>.

Gabapentin, i.e., (1-(aminomethyl) cyclohexaneacetic acid, has been introduced as an anti-epileptic drug<sup>13</sup>. It has been advanced for the treatment of neuropathic pain<sup>14</sup>. Acebutolol is a cardio selective beta blocker with intrinsic sympathomimetic activity.

It is therefore; more suitable than non cardio selective beta blocker, if a patient with asthma bronchiole or chronic obstructive pulmonary disease needs treatment with a beta blocker.

It was found that the regulation of the arterial hypertension is also a copper(II) and zinc(II) controlled process<sup>15</sup>. It was assumed that the effect of some of the most effective antihypertensive agents could be related to their ability for complexation with two metals mentioned above<sup>15</sup>. From chemical point of view the beta-adrenergic blocking drugs i.e., Gabapentin (1-AMCA), Acebutolol, N-(PA-4-2-HMEPPB) and t-Leucine (TL) (Figure 1a, b & c) are derivatives of aromatic amino alcohols, containing adjacent hydroxo- and amino- groups capable of chelate formation with Cu(II), Co(II) and Zn(II)<sup>16</sup>.

In this paper, we report the preparation of metal complexes of drugs viz., gabapentin, acebutolol and t-leucine as a chelating agent with Co(II), Ni(II) and Cu(II). Probable structures have been proposed on the basis of the elemental analysis, infrared, electron spin resonance and electronic spectra, magnetic susceptibilities and molar conductivity measurements. Thermal properties of the complexes have also been studied by DTA and TGA techniques. The antimicrobial activities of the ligands and their complexes have been tested against *Escherichia coli*,

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*Staphylococcus aureus*, *Aspergillus niger* and *Aspergillus flavous*.

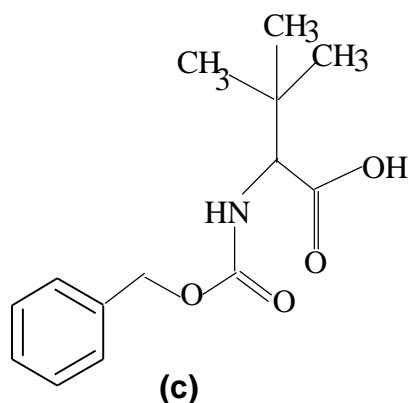
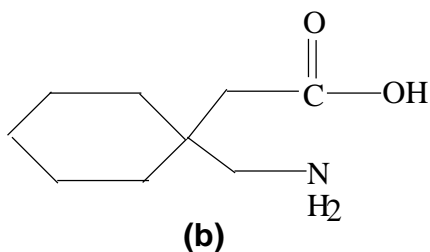
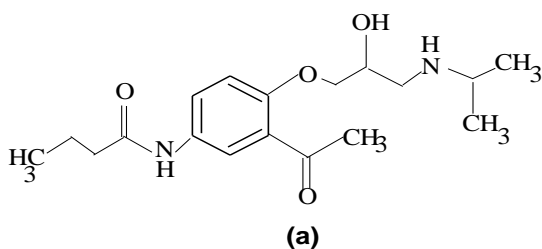


Fig. 1: (a) Acebutolol (b) Gabapentin (c) T-Leucine

### Material and Methods

Analytical grade metal chlorides, viz., NiCl<sub>2</sub>, CoCl<sub>2</sub>, and CuCl<sub>2</sub> of Lancaster and Lobachemie were used without any purification and drugs received were recrystallised from absolute alcohol.

Conductivity measurements were made using an Elico CM – 82 Conductivity Bridge. Electronic spectra of the complexes were taken in DMSO on Systronic spectrophotometer in the range of 200 – 900 nm. The magnetic susceptibilities were measured at room temperature using Gouy balance. IR spectra of the ligands and complexes were recorded in KBr matrix using Parkin Elmer 1000 FTIR spectrometer in the range of 4000 – 250 cm<sup>-1</sup>. The electron spin resonance spectra of Cu(II) complexes in poly crystalline state

were recorded on Varian X – band ESR spectrometer using diphenylpicrylhydrazine (DPPH) as a reference standard free radical. The electronic spectra were recorded on a Varian-Cary/2390 spectrophotometer. The thermal analysis studies were performed on a model DT-40 Shimadzu thermal analyzer using 40 mg sample. The DTA and TG curves were obtained at a heating rate of 10° C min<sup>-1</sup> in the 20-800° C temperature range in dry nitrogen atmosphere. Nitrogen was determined by the Dumas method and sulphur was estimated by the Messenger's method. The bio-efficacies of the synthesized compounds were tested *in vitro* and *in vivo*. *In vitro* tests were conducted for growth inhibitory activity against the common microorganisms are used viz., *Escherichia coli*, *Staphylococcus aureus*, *Aspergillus niger* and *Aspergillus flavous*. The radial growth and the disc methods<sup>17</sup> were employed to evaluate fungicidal and bactericidal activities, respectively.

### Preparation of metal complexes

Metal chlorides (0.01 mol) and ligand (0.02 mol) in ethanol were refluxed for about one hour in the presence of few drops of concentrated hydrochloric acid. On cooling, the complex separated was filtered and washed several times with alcohol and dried *in vacuo* over phosphorus pentoxide.

### Results and Discussion

The analytical data in Table-1 indicate that Co(II), Ni(II) and Cu(II) form 1:2 complexes of the type ML<sub>2</sub> (where L = 1-AMCA, TL) and 2:2 complexes of the type M<sub>2</sub>L<sub>2</sub> (L<sup>1</sup> = N-(PA-4-2-HMEPPB). Co(II) complexes are brown in color and Ni(II) complexes are yellow green in color, whereas, Cu(II) complexes are dark brown in color. All the complexes are soluble in DMSO and partially soluble in DMF. The molar conductance of all the complexes was determined in DMSO at 10<sup>-3</sup> molar concentration. The observed molar conductance values are in the range of 10 to 30 ohm<sup>-1</sup> cm<sup>2</sup> mol<sup>-1</sup>. These values are too low to account for any electrolytic behavior. Hence, these complexes are non – electrolytic in nature.

### IR spectra of ligands and complexes

Important IR bands of ligands and complexes and their assignments are listed in Table 2. Ligands exhibit bands in the region 1776-1705 cm<sup>-1</sup> attributed to ν(C=O). The bands in the region 3482-3414 cm<sup>-1</sup> are assigned to ν(O-H) and bands in the region 3082-3000 cm<sup>-1</sup> are assigned to ν(N-H) vibrational frequencies as per previous reports<sup>17</sup>.

In metal complexes ν(C=O) band in the region 1719-1655 cm<sup>-1</sup>, ν(N-H) band in the region 3054-2926 cm<sup>-1</sup> and ν(O-H) observed in the region 3545-3441 cm<sup>-1</sup>. The shift in these IR bands indicates that metal ions

coordinates through C=O, N-H and O-H groups respectively.

In addition to these IR bands in the region 526-490  $\text{cm}^{-1}$  and 455-430  $\text{cm}^{-1}$  are observed and are assigned to  $\nu(\text{M-N})$  and  $\nu(\text{M-O})$  respectively on analogy with the previous reports<sup>18</sup>.

#### Magnetic and electronic spectral studies

Electronic spectra of the complexes were taken in DMSO solution ( $1 \times 10^{-3}$  M). The electronic spectra of Co(II) complexes of ligands 1-AMCA, N-(PA-4-2-HMEPPB) and TL exhibited bands in the region 10108.02-10388.49  $\text{cm}^{-1}$ , 13156.83-18188.87  $\text{cm}^{-1}$  and 23927.44 -25000.90  $\text{cm}^{-1}$  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  ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{1g}(\text{P})$  ( $\nu_3$ ) transitions respectively. These transitions suggest that the octahedral geometry for the Co(II) complexes<sup>19</sup>. In the present study magnetic moment values are in the range of 4.5-4.9 B.M. This support an octahedral geometry around the metal ion for Co(II) complexes<sup>20</sup>.

Electronic spectra of Ni(II) complexes of all the ligands, 1-AMCA, N-(PA-4-2-HMEPPB) and TL, exhibited three bands in the region 11236.95 – 11429.57, 13881.88 – 15157.51 and 20839.33 – 23804.52  $\text{cm}^{-1}$  in DMSO. 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  ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{P})$  ( $\nu_3$ ) transitions respectively. All these observations favour octahedral geometry for Ni(II) complexes<sup>12</sup>. The observed magnetic moment values for Ni(II) complexes of ligands 1-AMCA, N-(PA-4-2-HMEPPB) and TL fall in the range 3.27-3.43 B.M which are well within the range expected for octahedral geometry around the central metal ion<sup>21,22</sup>.

The Cu(II) complexes of ligands 1-AMCA, N-(PA-4-2-HMEPPB) and TL showed broad band at 16122.00, 12156.52 and 12820.51  $\text{cm}^{-1}$ . The observed broad band can be assigned to  ${}^2\text{B}_{1g} \rightarrow {}^2\text{E}_g$ , and  ${}^2\text{B}_{1g} \rightarrow {}^2\text{E}_{2g}$  transitions in distorted octahedral geometry<sup>23</sup> of Cu(II).

The Cu(II) complexes of ligands 1-AMCA, N-(PA-4-2-HMEPPB) and TL exhibit magnetic moment values in the range of 1.70-1.88 B.M. These values are attributable to spin only value, and clearly indicates distorted octahedral<sup>24</sup> configurations for Cu(II).

#### ESR spectra

The 'g' values obtained from the spectra are presented in Table 3. When the monomeric species having axial symmetry and identical sites, the 'g' values also change due to the change in symmetry<sup>25</sup>. From the observed 'g' values  $g_{\parallel} > g_{\perp} > g_e$  (2.002), it is evident that the unpaired electron lies predominantly in the  $d_{x^2-y^2}$  orbital of Cu(II) ion<sup>26</sup>. The  $g_{\parallel} > g_{\perp} > g_e$  (2.009) observed in the 'g' values suggest the presence of an

unpaired electron in the  $d_{x^2-y^2}$  orbital. The values of the  $\sigma$  – bonding parameter,  $\alpha^2$ , show appreciable covalence character in the metal –ligand bond. These observations suggest Cu(II) complexes may have octahedral geometry<sup>26</sup>.

#### Thermal studies

The TGA and DTA curve for the gabapentin - cobalt complex [1-AMCA -Co] exhibited a three-stage decomposition pattern. The first stage decomposition is at about 90°C attributed to the liberation of one water molecule, while, the second at about 240°C is due to liberation of coordinated water molecules. According to Nikolaev<sup>27</sup> et al., water eliminated above 150°C can be considered as coordinated water. Two endothermic peaks in DTA showed these steps. The inflexion points of the endotherms were located at 110°C and at 180°C. Above 325°C there was complete decomposition of the complex and the mass loss consideration confirmed the products to be corresponding to metal oxides (CoO). In case of acebutolol nickel complex [N-(PA-4-2-HMEPPB)-Ni] the decomposition started at 140°C. The first stage decomposition between 140°C-220°C with a weight loss of 5.01 % (Calc. 5.12 %) indicating the complete removal of coordinated water molecules<sup>27</sup>. In the second stage it shows a weight loss of 58.37 % (Calc. 58.0 %) between 250°C-390°C due to the decomposition of the ligand molecule. Finally, at 420°C-550°C the TGA curve represents the complete oxidation of the remaining organic molecule with the formation of a stable metal oxide (NiO) as the final product with a weight loss of 16.67 % (Calc. 16.69 %).

#### Antimicrobial activity

Antimicrobial activity of ligand and their metal complexes are given in Table-4. Better activities of some of the metal complexes as compared to the pure compound can be explained by chelation theory. The theory explains that decrease in polarizability of the metal could enhance the lipophilicity of the complexes which leads to breakdown of permeability of the cells resulting in interference with normal cell process<sup>28</sup>.

#### Antibacterial activities

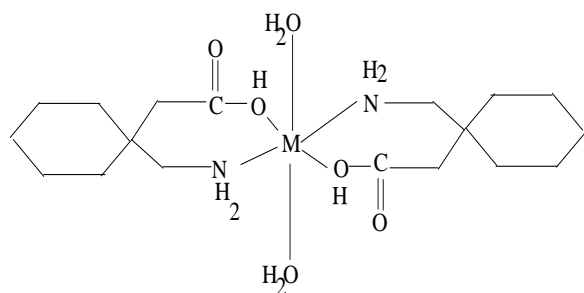
Gabapentin, acebutolol and t- leucine showed negligible antibacterial activity, but the activities of complexes were considerable against *E coli* and *S aureus*. Cu(II) complexes showed more activities against both the organisms when compared with the standard drug and the ligands.

#### Antifungal activity

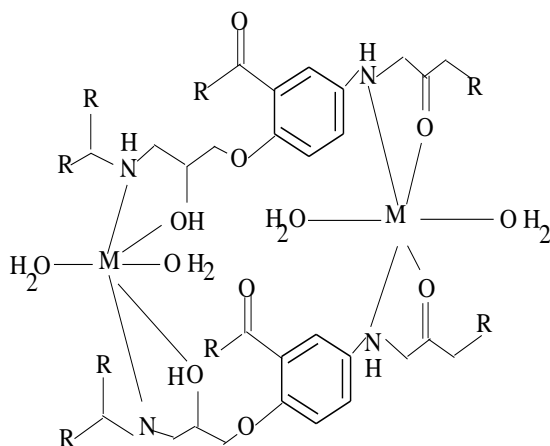
The complexes of Ni(II) exhibit less active whereas, metal complexes of Co(II) are significantly active towards *A niger* and *A flavous*.

**Conclusion**

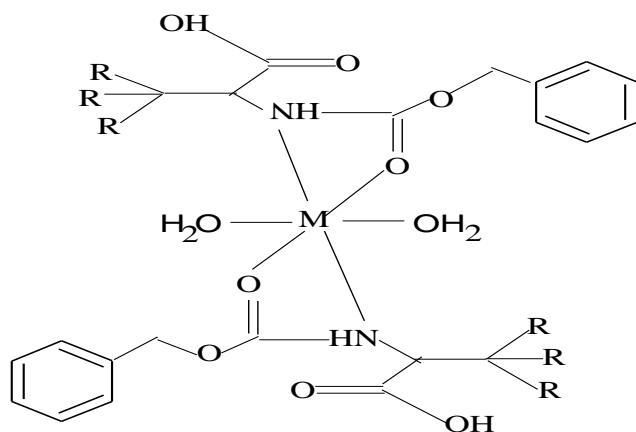
Based on analytical, IR, electronic, magnetic, ESR, TGS/DTA, conductance measurements, we propose the following octahedral structure for metal complexes.



**Fig. 2(a): Metal complexes of gabapentin**  
Where M= Co(II), Ni(II) and Cu(II)



**Fig. 2 (b): Metal complexes of acebutolol**  
Where M = Co(II), Ni(II) and Cu(II) ; R = CH<sub>3</sub>



**Fig. 2(c): Metal complexes of T-leucine**  
Where M = Co(II), Ni(II) and Cu(II) ; R

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Table 1: Elemental analysis, melting point and conductance data of ligands and metal complexes

S/No.	Ligand/Complex	Analysis% Found (Calculated)				M.P. (°C)	Molar Conductance ( $\Omega^{-1}\text{cm}^2\text{Mol}^{-1}$ )
		C	H	N	M		
1	1-AMCA	63.23 (63.06)	9.93 (9.92)	8.18 (8.17)	-	143	-
2	N-(PA-4-2-HMEPPB)	64.26 (64.24)	8.39 (8.32)	8.33 (8.32)	-	231	-
3	TL	69.92 (69.98)	9.48 (9.40)	6.27 (6.26)	-	252	-
4	Co(1-AMCA) <sub>2</sub> ·(H <sub>2</sub> O) <sub>2</sub>	48.30 (49.60)	7.20 (7.34)	6.20 (6.42)	13.41 (13.53)	220	10.23
5	Co <sub>2</sub> (N-(PA-4-2-HMEPPB) <sub>2</sub> (H <sub>2</sub> O) <sub>4</sub> )	56.39 (55.67)	7.11 (7.03)	7.32 (6.68)	7.75 (7.88)	>300	22.38
6	Co(TL) <sub>2</sub> ·(H <sub>2</sub> O) <sub>2</sub>	53.22 (53.85)	6.43 (6.73)	4.30 (4.49)	9.03 (9.30)	>300	21.22
7	Ni(1-AMCA) <sub>2</sub> ·(H <sub>2</sub> O) <sub>2</sub>	49.68 (49.63)	7.42 (7.35)	6.45 (6.43)	13.52 (13.49)	>300	18.98
8	Ni <sub>2</sub> (N-(PA-4-2-HMEPPB) <sub>2</sub> (H <sub>2</sub> O) <sub>4</sub> )	56.21 (56.47)	7.10 (7.04)	7.36 (6.67)	7.87 (7.98)	>300	16.13
9	Ni(TL) <sub>2</sub> ·(H <sub>2</sub> O) <sub>2</sub>	53.22 (53.79)	6.45 (6.72)	4.27 (4.48)	9.00 (9.40)	>300	13.89
10	Cu(1-AMCA) <sub>2</sub> ·(H <sub>2</sub> O) <sub>2</sub>	49.00 (50.88)	7.22 (4.95)	6.33 (6.60)	14.53 (14.97)	>300	09.23
11	Cu <sub>2</sub> (N-(PA-4-2-HMEPPB) <sub>2</sub> (H <sub>2</sub> O) <sub>4</sub> )	55.89 (55.95)	7.05 (6.99)	7.20 (7.28)	8.35 (8.23)	>300	22.45
12	Cu(TL) <sub>2</sub> ·(H <sub>2</sub> O) <sub>2</sub>	53.92 (53.37)	6.46 (6.67)	4.68 (4.45)	10.40 (10.10)	280	19.19

Table 2: Important IR frequencies in (cm<sup>-1</sup>) and their assignments

S/No.	Ligand /complex	v(O-H)	v(N-H)	v(C=O)	v(M-N)	v(M-O)
1	1-AMCA	3414	3037	1776	-	-
2	N-(PA-4-2-HMEPPB)	3454	3000	1765	-	-
3	TL	3482	3080	1705	-	-
4	Co(1-AMCA) <sub>2</sub> ·(H <sub>2</sub> O) <sub>2</sub>	3447	2926	1689	526	388
5	Co <sub>2</sub> (N-(PA-4-2-HMEPPB) <sub>2</sub> (H <sub>2</sub> O) <sub>4</sub> )	3545	3025	1655	490	455

6	Co( TL) <sub>2</sub> .(H <sub>2</sub> O) <sub>2</sub>	3523	2958	1701	508	430
7	Ni( 1-AMCA ) <sub>2</sub> .(H <sub>2</sub> O) <sub>2</sub>	3497	2924	1698	486	438
8	Ni <sub>2</sub> ( N-(PA-4-2-HMEPPB) <sub>2</sub> (H <sub>2</sub> O) <sub>4</sub>	3467	2961	1673	495	449
9	Ni(TL) <sub>2</sub> .(H <sub>2</sub> O) <sub>2</sub>	3479	2949	1696	510	450
10	Cu(1-AMCA ) <sub>2</sub> .(H <sub>2</sub> O) <sub>2</sub>	3448	2926	1688	520	449
11	Cu <sub>2</sub> (N-(PA-4-2-HMEPPB) <sub>2</sub> .(H <sub>2</sub> O) <sub>4</sub>	3439	3054	1702	497	416
12	Cu(TL) <sub>2</sub> .(H <sub>2</sub> O) <sub>2</sub>	3441	2964	1719	501	423

Table 3: EPR spectral data of Cu (II) complex

Complex	g <sub>  </sub>	g <sub>e</sub>	g <sub>⊥</sub>	g <sub>av</sub>	G	α <sup>2</sup>
Cu(1-AMCA) <sub>2</sub> .(H <sub>2</sub> O) <sub>2</sub>	2.208	2.11	2.113	2.16	1.86	0.46

Table 4: Antibacterial and Antifungal activities of drugs and their metal complexes

S/No.	Sample Name	Antibacterial		Antifungal	
		<i>Escherichia coli</i> (in mm)	<i>Staphylococcus aureus</i> (in mm)	<i>A. Niger</i> (in mm)	<i>A. Flavous</i> (in mm)
1	1-AMCA,	10	13	16	09
2	N-(PA-4-2-HMEPPB)	06	11	17	12
3	TL	15	13	14	10
4	Co(1-AMCA) <sub>2</sub> . (H <sub>2</sub> O) <sub>2</sub>	14	18	20	18
5	Co <sub>2</sub> (N-(PA-4-2-HMEPPB) <sub>2</sub> .(H <sub>2</sub> O) <sub>4</sub>	16	17	16	17
6	Co( TL) <sub>2</sub> . (H <sub>2</sub> O) <sub>2</sub>	19	14	19	20
7	Ni(1-AMCA) <sub>2</sub> .(H <sub>2</sub> O) <sub>2</sub>	17	15	16	13
8	Ni <sub>2</sub> (N-(PA-4-2-HMEPPB) <sub>2</sub> . (H <sub>2</sub> O) <sub>4</sub>	14	18	17	14
9	Ni( TL) <sub>2</sub> .(H <sub>2</sub> O) <sub>2</sub>	16	19	14	16
10	Cu(1-AMCA) <sub>2</sub> . (H <sub>2</sub> O) <sub>2</sub>	18	14	22	17

11	$\text{Cu}_2(\text{N}-(\text{PA}-4-2\text{-HMEPPB})_2 \cdot (\text{H}_2\text{O})_4)$	22	22	19	18
12	$\text{Cu}(\text{TL})_2 \cdot (\text{H}_2\text{O})_2$	19	21	21	23
	Oxytetracyclin, Nistatin(Standard)	18	20	19	18

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