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Research Article

SYNTHESIS AND PHYSICO-CHEMICAL STUDIES OF MACROCYCLIC LIGAND AND ITS Co (II), Ni (II), Cu (II) AND Zn (II) COMPLEXES

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ABSTRACT

14 membered tetraazamacrocyclic ligand and its metal complexes of Co(II), Ni(II), Cu(II) and Zn(II) have been synthesized biologically active macrocyclic ligand. The ligand was synthesized by condensation of β - diketone 1-(5-bromo-2-hydroxyphenyl)-3-(furan-2-yl)propane-1,3-dione with 2,3-diaminopyridine. All the complexes were characterised by conductivity, UV-visible, FTIR, ¹H NMR, Mass spectra, powder X-ray diffraction. From the analytical data, stoichiometry of the complexes has been found to be 1:2 (metal:ligand). All the complexes are of high spin type and six coordinated type. On the basis of IR, electronic spectral studies and magnetic behaviour, an octahedral geometry has been assigned to these complexes. The antibacterial and antifungal activities of the ligand and its metal complexes, has been screened in vitro against Gentamycin, Ampicillin, Chloramphenicol, Ciprofloxacin, Norfloxacin, Nystatin and Griseofulvin used as standard drug

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INTRODUCTION

Coordination chemistry of macrocyclic ligand containing a heteroatom is important complexing agents or cations, anions and molecule. The stability of macrocyclic metal complexes depends upon a number of factors such as number and type of donor atoms present in the ligand and their relative positions within the macrocyclic skeleton, as well as the number and size of the chelate ring formed on complexation[1],[2]. The macrocyclic complexes of various β - diketones with 2, 3-diaminopyridine in presence of metal ions in ethanol are prepared by the template condensation method have been already reported. Macrocyclic ligands form more stable complexes as compared to open chain ligands[3]. Schiff base complexes containing phenolic group as chelating moieties in the ligand are considered as models for executing important biological reactions and mimic the catalytic activities metalloenzymes[4]. The various studies reveal azomethine linkage (C = N) in Schiff base, a responsible part for biological activities. Many reports have shown that some drugs have greater activity when overseen as metal complex as that as free organic compound [5], [6].

MATERIALS AND METHODS

The substituted β - diketone was prepared from 2-hydroxyacetophenone and furon 2- carboxylic acid by BekarVankataraman rearrangement. furon 2- carboxylic acid, phosphorus oxychloride, pyridine, potassium hydroxide, 2,3-diaminopyridine of A.R. grade were used for preparation of ligands. A.R. grade meal nitrate was used for synthesis of complexes. The FTIR spectrum was recorded on Shimadzu spectrophotometer using KBr pellets. ¹H NMR spectra was recorded in CDCl₃ using TMS as internal standard. The conductivity of complexes was measured on Elico CM- 180 conductivity meter using 1mM solution in DMSO.

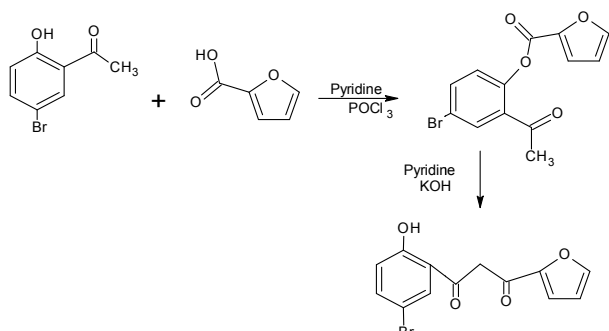
Synthesis of β - diketone step I

Equimolar amount of furon 2- carboxylic acid and 5-bromo-2-hydroxyacetophenone were dissolved in 50 ml pyridine. The reaction mixture was then cooled 0 °C. To this phosphorus oxychloride (0.06 mol) was added drop wise maintaining temperature below 10°C. The reaction mixture was kept overnight at room temperature. It was then poured on crushed ice with vigorous stirring. The crimson coloured solid (ester)

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was obtained which was filtered and washed several times with ice-cold water. Ester was then crystallized with distilled ethanol. Purity of the compound was checked by the TLC. Ester was subjected to well-known Baker-Venkatraman transformation. Ester (0.003mol) was dissolved in 15 ml dry pyridine. To this mixture, powdered KOH (1g) was added and the reaction mixture was stirred on magnetic stirrer at room temperature for 5 h. Then it was poured over crushed ice and acidified with concentrated hydrochloric acid. Finally yellow coloured product was obtained which was recrystallized from ethanol (Yield 55-58%). Purity of all synthesized β -diketonewas checked by TCL using silica gel and melting points (see Scheme 1).



Scheme 1 Synthesis of β -diketone

Synthesis of Ligand: A hot ethanolic solution of 30ml of β -diketone (0.02 mol) and a hot ethanolic solution 30ml of 2,3-diaminopyridine (0.02mol) were mixed slowly with constant stirring. This reaction mixture was then refluxed at 80-90°C for 14-15 hrs. in the presence few drops of conc. HCl (pH= 3-4) on cooling a solid yellow precipitate was formed, which was filtered, washed with cold ethanol and dried under vacuum over P_4O_{10} (yield- 59-60%).

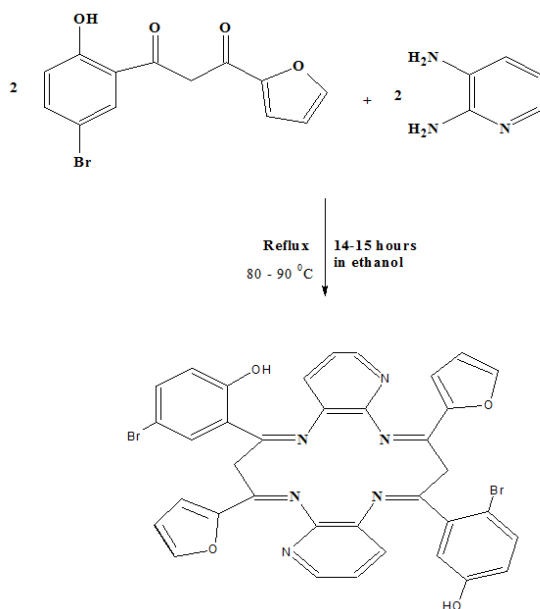


Figure 1 Synthesis of ligand

Synthesis of Metal Complexes: A hot ethanolic (20ml) solution of the ligand (0.01mol) and a hot ethanolic (20ml) solution of corresponding metal nitrate (0.01mol) were mixed together with constant stirring. The pH of the mixture was adjusted in the range of 7-8 by adding 10% alcoholic ammonia solution. The reaction mixture was refluxed for 7-8 hrs. at 80-90°C on

cooling, a coloured precipitate was formed. It was filtered and wash with cold ethanol and dried under vacuum over P_4O_{10} (yield 55-57%).

RESULTS AND DISCUSSION

Physical characteristic molar conductance data of ligand and metal complexes are given in Table 1. The analytical data of complexes reveals 1:2 molar ratio (metal: ligand). The X-ray diffraction data suggest cubic crystal system for Co(II) and Ni(II) and orthorhombic crystal system for Cu(II) complexes. The metal chelate solution in DMSO shows low conductance which support non-electrolyte nature of metal chelate.

¹H NMR Spectra of Ligand

¹H NMR spectra of the ligand was recorded in DMSO. It shows following signals at 1.7 (s, 4H, -CH₂), 6.7 (s, 2H, Ar-OH), 6.6-8.4 corresponding to phenyl ring protons (m, 18H).

Magnetic measurements and electronic spectra

Cobalt(II) complexes. The magnetic moments of the cobalt (II) complexes were measured at room temperature and were found in the range 4.90 μ_B , which is consistent with three unpaired electrons. The solution spectra of Co(II) complexes shows 13547 cm^{-1} and 17503 cm^{-1} were assigned $^4T_{1g}(F) \rightarrow ^4A_{2g}(F)$ and $^4T_{1g}(F) \rightarrow ^4T_{1g}(P)$ respectively. Suggesting octahedral geometry around the Co(II) ion [7].

Nickel(II) complexes. The magnetic moments of the nickel (II) complexes were measured at room temperature and were found in the range 2.94 μ_B , which is consistent with two unpaired electrons. The solution spectra of Ni(II) complexes shows 15503 cm^{-1} and 23041 cm^{-1} and 27173 cm^{-1} were assigned $^3A_{2g} \rightarrow ^3T_{2g}$, $^3A_{2g} \rightarrow ^3T_{1g}(F)$, and $^3A_{2g} \rightarrow ^3T_{1g}(P)$ respectively. Suggesting octahedral geometry around the Ni(II) ion [8].

Copper(II) complexes. The magnetic moments of the copper (II) complexes were measured at room temperature and were found in the range 1.82 μ_B , which is consistent with one unpaired electron. The solution spectra of Cu(II) complexes shows 13386 cm^{-1} and 17730 cm^{-1} were assigned $^2B_{1g} \rightarrow ^2B_{2g}$ and $^2B_{1g} \rightarrow ^2A_{2g}$ respectively. Suggesting octahedral geometry around the Cu(II) ion [9].

FTIR Spectra

The IR spectrum of the macrocyclic ligand (L) and its complexes shows a $\nu(C=N)$ peak at 1655 cm^{-1} , [10] and the absence of $\nu(C=O)$ peak at 1700 cm^{-1} and $\nu(NH_2)$ peak at 3250 cm^{-1} is indicative of Schiff base condensation [11]. The IR spectrum of ligand, appearance of a new strong absorption band at 1645 cm^{-1} , attributable to the characteristic stretching frequencies of the imino linkage $\nu(C=N)$, which provides strong evidence for the presence of cyclic product. On complete formation the $\nu(C=N)$ shifted towards lower side by 11-10 cm^{-1} hence the ligand is tetradentate, nitrogen donor coordinates through nitrogen of $\nu(C=N)$ group. The band in the 418-426 cm^{-1} regions may be assigned to $\nu(M-N)$ vibration [12]-[14]. The $\nu(OH)$ vibration of phenolic proton appears as a broad band in the region 3200-3600 cm^{-1} probably due to the overlapping of the symmetric and antisymmetric OH stretching vibration of lattice water [15]. The spectrum also shows the band at 1477, 640, 426 due to pyridine ring deformation, in plane ring deformation and out of plane ring

deformation, respectively. The absorption bands show the positive shift in complex which indicates that the nitrogen of pyridine ring is involved in coordination[16], [17]. The appearance of strong band in the range 1354-1394 cm^{-1} indicate the presence of ionic nitrate[18].

Mass Spectrum of the Ligand (L)

The spectrum of the ligand L shows the peaks at $m/z = 764$. The peak at $m/z = 747, 620, 582$ and 291 are due to the stepwise fragmentation of the macrocyclic ligand. The intensity of the peak at 291 is 100% because it corresponds to the macrocyclic moiety. Some other peaks also present in the spectrum at 246 and 185 due to the other fragmented ions [19]. The peaks area provides an idea of abundance of these ions.

Mass Spectrum of the [CoL (NO₃)₂2H₂O]

The ESI mass spectrum of the complex shows $[M + 1]^{+1}$ peak at 983 , which is equivalent to their molecular weights. The M [Co (C₃₆H₂₈Br₂N₈O₁₂)] molecular ion peak recorded at m/z 983 under goes fragmentation by 966 [Co (C₃₆H₂₆Br₂N₈O₁₁)], 948 [Co (C₃₆H₂₄Br₂N₈O₁₀)], 885 [Co (C₃₆H₂₄Br₂N₇O₇)], 747 [Co (C₃₁H₂₄Br₂N₆O₃)], 604 [Co (C₂₈H₂₄BrN₆O₂)], 465 [Co (C₂₅H₂₂N₆)], 360 [Co (C₁₇H₁₄N₆)], 290 (C₁₆H₁₄N₆).

Mass Spectrum of the [NiL (NO₃)₂2H₂O]

The ESI mass spectrum of the complex shows $[M + 1]^{+1}$ peak at 983 , which is equivalent to their molecular weights. The M [Ni (C₃₆H₂₈Br₂N₈O₁₂)] molecular ion peak recorded at m/z 983 under goes fragmentation by 966 [Ni(C₃₆H₂₆Br₂N₈O₁₁)], 948 [Ni (C₃₆H₂₄Br₂N₈O₁₀)], 885 [Ni (C₃₆H₂₄Br₂N₇O₇)], 747 [Ni(C₃₁H₂₄Br₂N₆O₃)], 604 [Ni (C₂₈H₂₄BrN₆O₂)], 465 [Ni(C₂₅H₂₂N₆)], 360 [Ni (C₁₇H₁₄N₆)], 290 (C₁₆H₁₄N₆).

Mass Spectrum of the [CuL (NO₃)₂2H₂O]

The ESI mass spectrum of the complex shows $[M + 1]^{+1}$ peak at 987 , which is equivalent to their molecular weights. The M [Cu (C₃₆H₂₈Br₂N₈O₁₂)] molecular ion peak recorded at m/z 987 under goes fragmentation by 952 [Cu (C₃₆H₂₄Br₂N₈O₁₀)], 890 [Cu (C₃₆H₂₄Br₂N₇O₇)], 827 [Cu (C₃₆H₂₄Br₂N₆O₄)], 747 [Cu (C₃₂H₂₄Br₂N₆O₂)], 685 [Cu (C₂₇H₂₂Br₂N₆O₂)], 604 [Cu (C₂₇H₂₀BrN₆O₂)], 577 [Cu (C₂₆H₂₀BrN₆O)], 465 [Cu (C₂₄H₂₀N₆O)], 445 [Cu (C₂₂H₁₈N₆O)], 429 [Cu (C₂₂H₁₈N₆)], 393 [Cu (C₁₉H₁₈N₆)], 379 [Cu (C₁₉H₁₈N₆)], 290 (C₁₉H₁₈N₆).

Mass Spectrum of the [ZnL (NO₃)₂]

The ESI mass spectrum of the complex shows $[M + 1]^{+1}$ peak at 953 , which is equivalent to their molecular weights. The M [Zn(C₃₆H₂₄Br₂N₈O₁₀)] molecular ion peak recorded at m/z 953 under goes fragmentation by 892 [Zn(C₃₆H₂₄Br₂N₇O₇)], 814 [Zn (C₃₆H₂₂Br₂N₆O₃)], 747 [Zn(C₃₂H₂₂Br₂N₆O₂)], 604 [Zn(C₂₇H₁₈Br₂N₆O₂)], 383 [Zn (C₁₈H₁₈N₆)], 355 [Zn (C₁₈H₁₈N₆)], 290 (C₁₉H₁₈N₆).

Table I Molecular formula, melting point, molar conductance and wavelength data of ligand and its metal complexes

compound	f.w	m.p ⁰ c	colour	molar conductance mho. $\text{cm}^2\text{mol}^{-1}$
L [C ₃₆ H ₂₄ Br ₂ N ₈ O ₄]	764	210	Brown	-
CoL. (NO ₃) ₂ .2H ₂ O	983	245	Dark-red	10.12
NiL. (NO ₃) ₂ .2H ₂ O	982	216	Greenish- yellow	11.10
CuL. (NO ₃) ₂ .2H ₂ O	987	219	Green	12.15
ZnL. (NO ₃) ₂ .	953	210	Dark- white	10.07

Table II IR Spectra (cm^{-1}) of ligand and its complexes

compound	(OH)	ν (C=N)	(C-Br)	(M-N)
L [C ₃₆ H ₂₄ Br ₂ N ₈ O ₄]	3267	1655	536	----
CoL (NO ₃) ₂ .2H ₂ O	3240	1656	536	418
NiL. (NO ₃) ₂ .2H ₂ O	3254	1654	536	428
CuL. (NO ₃) ₂ .2H ₂ O	3247	1658	536	418
ZnL. (NO ₃) ₂ .	3230	1645	536	426

Powder X- Ray Diffraction

The X-ray diffraction of representative metal complexes was scanned in the range $20 - 80^\circ$ at wavelength 1.560 \AA .

The diffractogram of Co(II) complex consists of 35 reflections with maxima at $2\theta = 21.660^\circ$ and its intensity 74.39 a.u. corresponding to d value 4.0995 \AA . The unit cell of Co (II) complex yielded values of lattice constant and unit cell volume $V = 1648.80$. In concurrence with these cell parameters, conditions such as $a = 9.714$, $b = 7.193$, $c = 23.72$ and $\alpha = 90^\circ$, $\beta = 95.950$, $\gamma = 90^\circ$ required for monoclinic sample are tested and found to be satisfactory. Hence, it can be concluded that the Co(II) complex belong to monoclinic crystal system.

The diffractogram of Ni(II) complex consists of 38 reflections with maxima at $2\theta = 17.520^\circ$ and its intensity 101.26 a. u. corresponding to d value 5.0578 \AA . The unit cell of Ni (II) complex yielded values of lattice constant and unit cell volume $V = 1758.94$. In concurrence with these cell parameters, conditions such as $a = 13.55$, $b = 14.89$, $c = 8.718$ and $\alpha = \beta = \gamma = 90^\circ$ required for orthorhombic sample are tested and found to be satisfactory. Hence, it can be concluded that the Ni(II) complex belong to orthorhombic crystal system.

The diffractogram of Cu(II) complex consists of 25 reflections with maxima at $2\theta = 23.540^\circ$ and its intensity 59.49 a. u. corresponding to d value 3.776 \AA .

The unit cell of Cu (II) complex yielded values of lattice constant and unit cell volume $V = 4047.27$. In concurrence with these cell parameters, conditions such as $a = 17.07$, $b = 18.48$, $c = 12.83$ and $\alpha = \beta = \gamma = 90^\circ$ required for orthorhombic sample are tested and found to be satisfactory. Hence, it can be concluded that the Cu(II) complex belong to orthorhombic crystal system.

The diffractogram of Zn(II) complex had 29 reflections with maxima at $2\theta = 17.500^\circ$ and its intensity 95.79 a.u. corresponding to d value 5.0635 \AA .

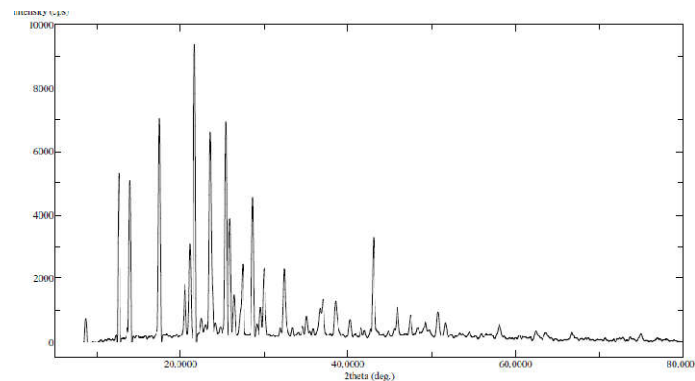


Fig 2 X-Ray Diffraction pattern of Co L (NO₃)₂ . 2H₂O

The X- ray diffraction pattern of these complexes with respect to major peaks having relative intensity greater than 10 % has been indexed by using computer programme[20]-[22]. The

above indexing method also yields Miller indices (hkl), unit cell parameters and unit cell volume.

Antibacterial Activity

All the synthesized macrocyclic metal complexes were tested against pathogenic clinically isolated standard bacterial strains *E. coli* (MTCC 442), *P. aeruginosa* (MTCC 441), *S. aureus* (MTCC 96), and *S. Pyogenus* (MTCC 443) using broth dilution method (Table-III). Gentamycine, Ampicillin, Chloramphenicol, Ciprofloxacin and Norfloxacin were used as a reference drug for bacteria. Complex Zn(II) showed strong activity against *E. coli* than Ampicillin. Co(II), Ni(II) and Cu(II) complexes showed moderate antibacterial activity against the *E. coli*, *P. aeruginosa*, *S. aureus* and *S. Pyogenus*.

Table III Antibacterial activity of compounds

Sr.No	Code No	Minimum Inhibition Concentration ($\mu\text{g/ml}$)			
		E.Coli MTCC 442	P. Aeruginos MTCC 441	S. Aureus MTCC 96	S.Pyogenus MTCC 443
1	CoL. (NO ₃) ₂ .2H ₂ O	250	250	250	250
2	NiL. (NO ₃) ₂ .2H ₂ O	500	250	250	500
3	CuL. (NO ₃) ₂ .2H ₂ O	125	250	500	250
4	Zn L. (NO ₃) ₂ .2H ₂ O	50	250	250	250
5	Gentamycine	0.05	1	0.25	0.5
6	Ampicillin	100	100	250	100
7	Chloramphenicol	50	50	50	50
8	Ciprofloxacin	25	25	50	50
9	Norfloxacin	10	10	10	10

Antifungal Activity

All the synthesized macrocyclic metal complexes were tested against pathogenic clinically isolated standard bacterial strains *C. Albicans* (MTCC 227), *A.Niger* (MTCC 282), *A.Clavatus* and (MTCC 1323), using broth dilution method (Table-IV). The Co(II), Ni(II) metal complexes showed better antifungal activity against *C. Albicans* than Greseofulvin. Co(II), Ni(II), Cu(II) and Zn(II), metal complexes showed moderate antifungal activity against *A.Niger*, *A.Clavatus*.

Table IV Antifungal activity of compound

Sr.No	Code No	Minimum Inhibition Concentration ($\mu\text{g/ml}$)		
		C. Albicans MTCC 227	A.Niger MTCC 282	A.Clavatus MTCC 1323
1	CoL. (NO ₃) ₂ .2H ₂ O	250	500	1000
2	NiL. (NO ₃) ₂ .2H ₂ O	250	500	500
3	CuL. (NO ₃) ₂ .2H ₂ O	500	500	1000
4	Zn L. (NO ₃) ₂ .2H ₂ O	500	500	500
5	Nystatin	100	100	100
6	Greseofulvin	500	100	100

CONCLUSION

In this paper, we describe the synthesis and characterisation of hexadentate macrocyclic ligand and its Co²⁺, Ni²⁺, Cu²⁺ and Zn²⁺ complexes. Based on conductance, infrared electronic visible, mass spectral data, and X-ray powder diffraction pattern and biological activity, all these complexes exhibit coordination number six. The FTIR spectral data suggest that ligand behaves as a tetradentate ligand with N₄ donor atoms towards central metal ion

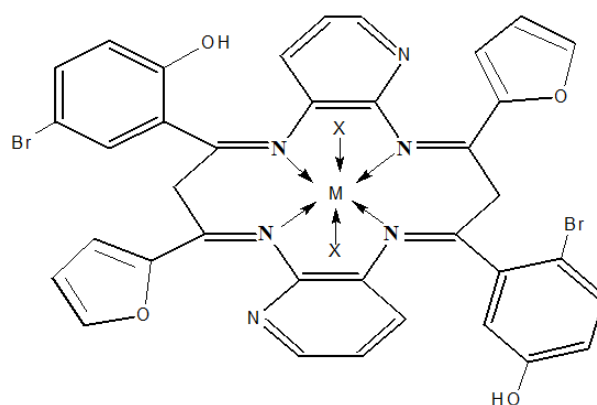


Fig.3 Proposed Structure of Complex

Where M= Co(II), Ni(II), Cu(II) and Zn(II) X= NO₃, H₂O

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