

Synthesis, Characterization and Antimicrobial Activity of some Metal Complexes of Substituted Furoinoximes

P.M.Dahikar

Department of Chemistry

Shri.R.R.Lahoti Science College,Morshi,Dist- Amravati.

Abstract : Recently, the synthesis of furoinoxime, furoinhydrazone, furoinphenylhydrazone, furoinsemicarbazone were synthesized by the interactions of furoinbenzoin with hydroxylamine hydrochloride, hydrazine hydrate, phenyl hydrazine and semicarbazide hydrochloride in presence of aqueous sodium hydroxide in DMF-water (80%) medium respectively. The synthesis of furoinbenzoin were carried out by the known literature method. Metal benzoinoxime complexes were synthesized from substituted benzoinoximes they were characterized by elemental and spectral analysis. The physico-chemical data suggested octahedral geometry for Co(II) and Cu(II) complexes. The synthesized complexes were screened for antimicrobial activity at a concentration of 1000µgm/ml which was serially diluted to determine their MIC values .

Keywords: - Metal complexes, Antimicrobial activity, 4-Dimethylaminobenzoinoxime, Furoinoximes.

I. INTRODUCTION

Benzoin and oxime nucleus containing heterocyclic compounds possess pharmaceutical, medical, agricultural, industrial significance .Synthesis Fe(III) complexes of O-Vanillin oxime and their characterisation by different physico-chemical techniques was carried out by kurup¹.synthesis of mononuclear and binuclear Cr(III) complexes of α -benzoinoxime and their characterization was studied in detailed².synthesis of Fe(III) benzoin complexes and there characterization were carried out by El- agaily³. Luo⁴ synthesized oximes from different carbonyl compounds in a novel ionic liquid or water biphasic system synthesis of Co(II) , Ni(II) Zn(II), Fe(III), Cu(II) complexes with oxime amido and thioamido groups and their characterization were briefly studied by El-Asmy⁵. Five co-ordinated Co(II) complexes possesses both low-spin configuration having three unpaired electrons. These configuration showed trigonal bipyramidal and square pyramidal as well as intermediate configuration⁶⁻⁸. Mohmmed⁹ reported two novel aroyl hydrazone monoxime ligands and their Co(II) complexes. The magnetic moment value for Co(II) complexes are found in the range 4.78-5.04 B.M.suggest octahedral geometry. complexes of Cr(III) and Mn(II) with oximes such as 2-hydroxyacetatophenone oxime, 2-hydroxynaphthaldehyde oxime and salicylaldehyde oxime were synthesized and characterized by Chandra¹⁰.Benzoinoxime are well known for their biological activity, co-ordination compounds containing O,N,S. as donor atoms are reported to possess antimicrobial activity¹¹. synthesis characterization and thermal degradation studies of coordination polymers of ethanone oxime were carried out by wanjar¹².Day¹³, synthesized a large number of Cr(III) complexes and reported the magnetic moment values in the range 3.78-3.99 .Rahangadale¹⁴.synthesized the Schiff base and its complexes were screened for their antimicrobial activities various against bacteria and fungi.

II. Experimental

The benzoinoxime were prepared by refluxing substituted benzoin with hydroxylamine hydrochloride in alkaline medium for 3-4 hours, the reaction mixtures were kept overnight the solid products formed were isolated and wash several times with water alcohol mixture. The purity was checked by TLC paper. Their structural detail were confirmed on the basis of elemental and spectral analysis In order to synthesize the complexes, the equimolar mixture of each of the ligand (0.01M) and metal salts was refluxed on a water bath for 6-8 hours in presence of sodium acetate in ethanol . The reaction mixture was kept overnight. The product formed were isolated ,washed several times with cold water ethanol mixture the characterization of synthesized complexes was made by elemental analysis, IR and UV-VIS spectra.

III. RESULTS AND DISCUSSION

3.1 IR spectral data of ligands and their complexes are given in table -1

Ligand and its complexes	(O-H)	(C=N)	(C-O)	(M-O)	(M-N)
4-DMABO	3423	1660	1385	-	-
[Co(II)(L) ₂ (H ₂ O) ₂] H ₂ O	3318	1645	1380	482	587
[Cu(II)(L) ₂ (H ₂ O) ₂] H ₂ O	3420	1640	1382	485	59
[Mn(II)(L) ₂ (H ₂ O) ₂]	3412	1605	1375	465-	585-
FURO	3395	1651	1420	-	-
[Co(II)(L) ₂ (H ₂ O) ₂] H ₂ O	3383	1604	1396	486	589
[Cu(II)(L) ₂ (H ₂ O) ₂]H ₂ O	3380	1616	1418	487	609
Mn(II)(L) ₂ (H ₂ O) ₂]	3351	1592	1394	478	587

In these complexes 4-DMABO-Co(II), $\nu(\text{O-H})$ is observed at 3318 cm^{-1} $\nu(\text{C=N})$ at 1645 cm^{-1} . Are indicative of linking of oxygen without loss of hand linking of N to the metal ion respectively. These lower values of bands in hydroxyl and oximino stretching as compared to ligand clearly indicates that the coordinate bonding through hydroxyl oxygen and oximino nitrogen atom to the metal ion. In the complexes of 4-DMABO-Cu(II), $\nu(\text{O-H})$ at 3420 cm^{-1} which shows linking of metal oxygen atom without loss of proton similarly, $\nu(\text{C=N})$ is observed at 1645 cm^{-1} . which shows decrease in (C=N) stretching frequency during complexation and hence give clue about linkage. The electronic spectrum of 4-DMABO-Co(II) complexes exhibits their transition in the range $13476, 19002, 22620\text{ cm}^{-1}$

3.2 Magnetic moment and electronic spectral data (cm^{-1}) of the metal complexes as given table-2

Complexes	$\mu_{\text{eff}}(\text{BM})$	$\lambda_{\text{max}}(\text{cm}^{-1})$	$Dq(\text{cm}^{-1})$	$B^1(\text{cm}^{-1})$	B	%Covalency	ν_1/ν_2
$[\text{Co}(\text{L})_2(\text{H}_2\text{O})_2] \text{H}_2\text{O}$	5.15	13476,19002,22620	1476	695	0.716	28.4	1.41
$[\text{Cu}(\text{L})_2(\text{H}_2\text{O})_2] \text{H}_2\text{O}$	1.93	13670,19002,22620	1467	695	0.716	28.37	1.41
$[\text{Mn}(\text{L})_2(\text{H}_2\text{O})_2]$	4.51	13652,19126,23251	1498	728	0.759	24.01	1.40
$[\text{Co}(\text{L})_2(\text{H}_2\text{O})_2] \text{H}_2\text{O}$	5.17	13698,19047,21739	1489	616	0.634	36.06	1.35
$[\text{Cu}(\text{L})_2(\text{H}_2\text{O})_2] \text{H}_2\text{O}$	1.87	13586,19157,22624	1489	616	0.634	36.55	1.35
$[\text{Mn}(\text{L})_2(\text{H}_2\text{O})_2]$	5.51	14180,19575,22935	1546	669	0.629	30.01	1.38

The electronic spectrum of 4-DMABO-Co(II) complexes exhibits three transition in the range, $13476, 19002, 22620\text{ cm}^{-1}$. These spectral bands may be assigned to the following transition ${}^4\text{A}_2 \rightarrow {}^4\text{T}_1$, ${}^4\text{A}_2 \rightarrow {}^4\text{T}_1(\text{F})$, ${}^4\text{A}_2 \rightarrow {}^4\text{T}_1(\text{P})$ characteristic to an octahedral geometry. The magnetic moment of 5.15 BM for Co(II) complexes is consistent with octahedral geometry around central metal ion has high spin configuration. FURO-Cu(II) complexes exhibit absorption bands at $13586, 19157, 22624\text{ cm}^{-1}$.

Which may be assigned to ${}^2\text{B}_{1g} \rightarrow {}^2\text{A}_{1g}$, ${}^2\text{B}_{1g} \rightarrow {}^2\text{E}_g$, ${}^2\text{B}_{1g} \rightarrow {}^2\text{B}_{2g}$ transition respectively suggesting on octahedral geometry around a Cu(II) ion in the complexes under study, further more the magnetic moment measurement recorded at room temperature lies at 1.87 BM. This value indicates of an octahedral geometry of these complexes the calculated values is of ligand field splitting energy ($10Dq$), Racah interelectronic repulsion parameter (β), ν_1/ν_2 and % Covalency as shown in above table.

Thermogravimetric Analysis :-

An analysis of TG curves shows that 4-DMABO and its Co(II) complexes decompose in three stages, the Cu(II) and Mn(II) complexes decompose in two stages, the complexes of Co(II) are stable up to 140°C . The presence of water molecule (lattice or co-ordinated) in Co(II) Complexes suggested from IR spectra is confirmed by the weight loss observed in first decomposition step of these complexes the Co(II) complexes lose their weights up to 150°C corresponding to one lattice water molecule [% wt. loss obs./calcd : Co(II) : 5.53/5.46 and further up to 220°C corresponding to two coordinated water molecules [% wt. loss obs./calcd : Co(II) : 8.30/8.20]. In the case of Cu(II) and Mn(II) complexes 24 weight loss at 150°C corresponds to two lattice water molecule [% wt. loss obs./calcd : Cu(II) : 5.65/5.60, Mn(II):5.73/5.66]. These complexes are stable up to 250°C . The organic moiety decomposes in the temperature range of 545°C to 650°C leaving behind their respective metal oxides¹⁵. The decomposition rate of each metal complex is faster than that of the ligand and which be due to catalytic action of metal ion present in them.

An analysis of TG curves shows that FURO and its Co(II) and Mn(II) complexes decompose in three stages, the Cu(II) complexes decompose in two stages, the complexes of Co(II) are stable up to 140°C . The presence of water molecule (lattice or co-ordinated) in Co(II) Complexes suggested from IR spectra is confirmed by the weight loss observed in first decomposition step of these complexes the Co(II) complexes lose their weights up to 150°C corresponding to two lattice water molecule [% wt. loss obs./calcd : Cu(II) : 7.03/6.93 and further up to 220°C corresponding to one coordinated water molecules [% wt. loss obs./calcd : Co(II) : 3.42/3.40, Mn(II):3.45/3.42]. These complexes are stable up to 250°C . The organic moiety decomposes in the temperature range of 650°C leaving behind their respective metal oxides.

3.3 On the basis of elemental analysis the complexes were assigned the composition as shown in table-3

Complexes	Colour	M. Wt.	Decomposition Temp $^\circ\text{C}$	$\Delta m \Omega^1 \text{ cm}^2 \text{ mol}^{-1}$
$[\text{Co}(\text{L})_2(\text{H}_2\text{O})_2] \text{H}_2\text{O}$	Red	650.93	278	10.5
$[\text{Cu}(\text{L})_2(\text{H}_2\text{O})_2] \text{H}_2\text{O}$	Dark Brown	637.54	273	10.5
$[\text{Mn}(\text{L})_2(\text{H}_2\text{O})_2]$	Brown	628.93	282	10
$[\text{Co}(\text{L})_2(\text{H}_2\text{O})_2] \text{H}_2\text{O}$	Grey	524.93	290	10.8
$[\text{Cu}(\text{L})_2(\text{H}_2\text{O})_2] \text{H}_2\text{O}$	Pale Brown	511.54	281	10.2
$[\text{Mn}(\text{L})_2(\text{H}_2\text{O})_2]$	Dark Brown	520.93	325	10.4

3.4 Elemental analysis :-Table-4

Complexes	Elemental analysis found/(calculated)%			
	C	H	N	M
[Co (L) ₂ (H ₂ O) ₂] H ₂ O	58.03 (58.99)	5.30 (6.14)	7.71 (8.60)	8.98 (9.05)
[Cu-(L) ₂ (H ₂ O) ₂] H ₂ O	59.07 (60.23)	4.88 (5.96)	8.78 (8.78)	8.90 (9.96)
[Mn (L) ₂ (H ₂ O) ₂]	60.11 (61.05)	5.13 (6.04)	8.89 (8.90)	7.81 (8.73)
[Co (L) ₂ (H ₂ O) ₂]	44.81 (45.72)	4.00 (4.91)	5.33 (5.33)	11.22 (11.22)
[Cu(L) ₂ (H ₂ O) ₂] H ₂ O	45.88 (46.91)	2.86 (3.90)	5.47 (5.47)	11.37 (14.42)
[Mn (L) ₂ (H ₂ O) ₂]	45.11 (46.07)	3.32 (4.22)	5.37 (5.37)	9.60 (10.54)

3.5 Antimicrobial activity :-

The compounds were assayed for their antimicrobial activities¹⁶ Against for test organisms. E. Coli S.aureus, P.aeruginosa and B.subtilis, at a concentration of 1000 µgm/ml by agar well technique¹⁷. Further their MIC value against these. Organisms were determined by serial dilution method using DMF as a solvent, the results obtained are given in the following table

MIC Values in µgm/ml of compounds-Table-5

Complex	E. coil	S. aureus	P. aeruginosa	B. subtilis
[Co (L) ₂ (H ₂ O) ₂] H ₂ O	63	125	125	250
[Cu(L) ₂ (H ₂ O) ₂] H ₂ O	125	125	250	250
[Mn (L) ₂ (H ₂ O) ₂]	125	125	125	125
[Co (L) ₂ (H ₂ O) ₂]	250	125	125	125
[Cu (L) ₂ (H ₂ O) ₂] H ₂ O	125	125	250	125
[Mn (L) ₂ (H ₂ O) ₂]	125	125	125	63

On the basis of MIC Values, the complexes 4-MABO-Co(II) FURO-Co(II) is found to be most effective antimicrobial agent followed by FURO-Cu(II) and DMABO-Cu(II). The enhance antimicrobial activity in case of the compounds FURO-Mn(II) may be attributed to the presence of furnayl group.

IV. ACKNOWLEDGMENT

PMD thankful to Shri R. R. Lahoti Science College, Morshi for providing necessary facility.

REFERENCES

- [1] M.R.P. Kurup ; E. Lukose; K. Mraleedharan ; J. Thermal analysis and calorimetry, 59, 815-825, 2000
- [2] A.S. Attia, S.F. El- Mashtouly and M.E. El-Shahat, Synthesi and reactivity in Inorganic and metal-organic chemistry, Vol-32 issue 3, 2002
- [3] M.M. El-Ayaily; A.A. Maihub; M.A. Abuzwida; M.M. Aboukrisha, A.A. Amar; E.A. Asaib, Asian J. Chem. 19 (1), 781-783, 2007.
- [4] H.M. Luo. Y.Q. Li; W.J. Zheng, Chinese Chemical Letterr, 16 (7), 906-908, 2005
- [5] A.A. El-Asmy; M.E. Khalifa; M.M. Hassanian, Ind. J. Chem; 43A, 92-97, 2004 6
- [6] D.H.Sutaria;J.R.Patel;M.N.Patel,J.Ind.Chem.Soc.,73,309-312,1996
- [7] S.Suma;M.R.Sundarsanakumar;C.G.R.Nair;C.P.Prabhakaran,Ind.J.Chem.,33(A),1107-1109,1994
- [8]L.Sacooni,coor.Chem.Rev.,1,192,1967
- [9] Mohammed M.Al-Neaimi,Moudar M.Al-Khuder,Salim Mohammed,Ref.J.Sci.,23(4),51-69,2012
- [10] S. Chandra, P.Pipil; S.D. Sharma, Synthesis and Reactivity in Inorganic, Met-org. and Nano-Metal Chem. 31 (5), 779-797, 2001
- [11] A.kriza;C.Spinu;M.Plencicenu,J.Ind.Chem.Soc.,77,93,2000
- [12] Pettering H.G. and crime J.A. Cancer Res, 27, 1278, 1967
- [13] N. Wanjari, L.J. paliwal M.B. Bagade J. Atoms and Molecules. 2 (6), 410-420, 2012
- [14] K. Dey. K. Chakrabarty, Ind J. Chem. 39 (A) ,1140, 2000
- [15] M.Rahangdale, Gaurav pethe,AmitYaul,Anand Aswar,Pharceumaceutical, Biological and chemical and chemical sciences,2(3),343,2011
- [16] V.N. Patange and B.R. Arbad. J. Serb. Chem Soc. 76 (9), 1237-1245, 2011
- [17] C.D. Donald and A.R. William, In Assay methods of antibiotics A. Lab Manual medical Encyclopedia INC. 1955.