

SYNTHESIS AND SPECTRAL STUDIES OF SOME CO-ORDINATION COMPOUNDS

Ayesha Durrani¹, Shivaji Jadhav, Megha Rai² and Mazahar Farooqui²

¹Maulana Azad College, Aurangabad (M.S.) 431001

²Dr. Rafiq Zakaria College for Women, Aurangabad (M.S.) 431001

E-mail: simshivv_48@yahoo.com

ABSTRACT

A new series of complexes of Co(II) and Ni(II) with amino acid and 2-amino-iso butyric acid have been synthesized exhibits 1:2 metal-ligand ratio. Characterization of the ligand as well as complexes has been done on the basis of elemental analysis, Infra-red and magnetic susceptibility measurements. The ligand have been found to Co-ordinate through nitrogen and sulphur atoms. The complexes are binary and ternary complexes and are thermally stable.

The present paper describe the formation of complexes of Co(II) and Ni(II) with L-cystein and 2-amino is butyric acid.

Keywords: 2-amino-iso butyric acid, 1:2 metal-ligand ratio, L-cystein.

INTRODUCTION

For present study we have selected amino acid and substituted amino acids because a very less work has been done on synthesis of complexes with transition elements. P.V. Marykutty and Geetha Parameswaran¹ reported synthesis of Fe(II), Co(II), Ni(II) and Mn(II) complexes of Anthracene carboxaldehyde semicarbazone.

Liang-Nian Ji and Jin-Gang Liu² reported that transition metal complexes are possible molecular tools or the study of nucleic acid. Sami A. Zabin and C.R. Tehurkar³ reported fluorescence, antibacterial and pigmentation studies of some binuclear Schiff base complexes. The ligand and their metal Ni(II), Zn(II) complexes shows good antibacterial activities. Praveen Sharma and Surendra N. Dubey⁴ reported Co(II), Ni(II) and Zn(II) complexes with 3 - acetyl - amino - 2 - benzoyl benzofuran. Results shows that Co(II), Ni(II) complexes are polymeric octahedral and the ligand behaves as bidentate in all the complexes. Takaji Yasui Jinsai Hidaka and Yoichi Shimura⁵ studied circular dichorism of Co(II) complexes with L-amino acids. A binary complexes of Fe(III) with amino acids has been reported. Ru Hu, Jia-Yuan, Li Xian-Jun⁶ reported three new crowned bis-Schiff's bases and their cobalt (II) complexes. Sakiyan, Iffet, Gunduz, Neda, Gunduz Turgut⁷ synthesized Mn (III) complexes of Schiff bases derived from amino acid such as glycine, L-alanine, L-histidine etc. Where characterized by elemental analysis, IR and magnetic susceptibility. Y. Jadegound, Omkar B. Ijare, N.N. Mallikarjuna, S.D. Angadi and BHM. Mruthyunjayasyamy⁸ synthesised Cu(II), Co(II) and Ni(II) complexes with Schiff bases in ethanol medium and characterized by IR spectra and magnetic data. Many metal complexes of semicarbazone have been known for their pharmacological properties including activity against tuberculosis⁹ bacterial¹⁰ and viral functions¹¹.

EXPERIMENTAL

Solid complexes are prepared by taking different transition metal nitrate and ligands. The metals used are cobalt nitrate and ferric nitrate of analytical grade and prepared in double distilled water. Ligands used for complex formation are obtained from sd-fine chemicals. The ligands used as L-cystein and 2-aminoisobutyric acid.

Synthesis

The complexes are prepared by using transition metal nitrate and ligands are from sd-fine chemical. 50 ml of nitrate solution and in that 100 ml of ligand solution 1:2 ratio added with constant stirring. The crystals obtained are kept at room temperature for sometime. The coloured crystals obtained are collected and dried in an electric oven.

Elemental analysis

For finding the percentage of constituent elements of complex such as C, H, O and N. The elemental analysis is done by Wockhardt Ltd, Aurangabad.

Infra-Red Spectra

Transition metal complexes with amino acid and substituted amino acids were prepared and characterized by Infra-red spectra. The spectra are recorded using IR- spectropotometer 8700 FTIP by Wockhardt Ltd.

Magnetic Susceptibility

It is measured on Gouy balance. Standard substance used in mercury tetra thiocyanato cobalt. The Xg value is 16.44×10^{-6} cgs unit. The tube is suspended to the arms of Gouy balance and reading are recorded when current is ON and OFF of each complex.

RESULTS AND DISCUSSION

Synthesis of solid complexes are very useful since some transition metal complexes are found to possess biological activity against bacteria, fungi and certain type of tumors. Studies on metal complexes of nitrogen containing ligands, especially heteroaromatic nitrogen bases are of much interest¹².

Elemental analysis of the complexes obtained are stable at room temperature. The analytical data obtained by using thermal conductivity detector and eager 200 software.

Table-I: Analytical Data

Sr. No	Complex	Colour	% Analysis Found (Calculated)						Molecular weight
			C	H	N	O	S	M	
1	Co(II)-cystein	Pink	24.316 (22.696)	3.761 (3.782)	9.525 (7.880)	24.421 (25.218)	19.793 (20.212)	18.184 (18.576)	312.05
2	Ni(II)-cystein	Light green	23.303 (24.080)	4.396 (4.347)	9.164 (9.364)	24.034 (21.404)	19.479 (21.444)	19.624 (19.632)	299.82
3	Ni(II)-2-amino-isobutyric acid	Light blue	28.786 (29.662)	7.188 (6.797)	9.644 (8.651)	15.855 (19.775)	0.00 (0.00)	38.527 (36.274)	163.7
4	Co(II)-2-amino isobutyric acid	Violet	41.933 (46.490)	11.334 (12.326)	12.448 (10.559)	5.165 (6.033)	0.00 (0.00)	29.12 (22.22)	269.93
5	Co(II)-cystein-2-amino iso butyric acid	Pink	23.901 (23.859)	4.001 (3.728)	9.297 (10.435)	28.044 (27.836)	19.137 (15.936)	15.62 (14.93)	346.05
6	Ni(II)-Cystein-2-amino iso butyric acid	Light green	29.217 (29.841)	5.872 (6.217)	11.334 (10.444)	13.994 (15.915)	25.943 (23.918)	13.64 (14.59)	405.88

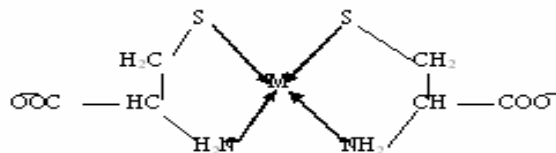
Infra Red Spectra

IR-Spectroscopy is used to know the Co-ordination site of the ligand. IR spectra are also very diagnostic¹³. The most reliable feature being a C - N stretching band which is sharp 2000cm^{-1} for S-band complex, which are broad and below 2000cm^{-1} for N-bonded complexes.

L - cystein has three potential Co-ordination sites (S, N and O) and IR spectra, the Zn(II) complex shows number ν_{SH} and its carboxylate frequency indicates the presence of a free Coo^- group. The following structure was proposed¹⁴.

The IR-spectrum of cystein shows bands at 3369.4cm^{-1} which are assigned to N - H, C - H stretching vibrations position of this band changes indicating that NH_2 group in complexation cystein also shows IR bands at 1569.9cm^{-1} which may be due to vibration of $\nu_{(\text{Coo}^-)}$ band which shifted to 1589 in case of Co(II)

and 1604 for Ni(II). The weak bands in the range of 400-490 cm^{-1} may be assigned to $\nu_{(M-O)}$ vibrations. The sharp intense band at 738 cm^{-1} is referring to $\nu_{(C-S)}$. The co-ordination through O, S and N donor atom are confirmed by occurrence of three bands in the far IR region of 570 – 520 cm^{-1} for $\nu_{(M-O)}$ ¹⁵. The IR – spectrum of 2-amino iso butyric acid show band at 3031 – 2317 cm^{-1} for N – H and C – H stretching. The IR spectrum of cystein shows band at 3369 – 2885 cm^{-1} which assigned to N – H and C – H stretching. The sharp intense band at 732.9 cm^{-1} for Co(II) 779.2 cm^{-1} for Ni(II).



The proposed chemical structure for the transition metal complex

Table-2:IR Spectral Band in cm^{-1}

Sr. No.	Compound	Band Vibrations						
		$\nu_{(N-H)}$	$\nu_{(C-H)}$	$\delta_{(N-H)}$	$\nu_{(M-O)}$	$\nu_{(C-S)}$	$\nu_{(S-H)}$	$\nu_{(M-S)}$
1	Co(II)-cystein	3340.5	2923.9	1604.0	547.7	786.9	2360.7	447.5
2	Ni(II)-cystein	3348.0	--	1604.0	540.0	709.8	2098.4	455.0
3	Co(II)-2-amino-sio butyric acid	3031.9	2815.9	1581.5	547.7	786.9	--	--
4	Ni(II)-2-amino isobutyric acid	3178.5	2985.6	1589.2	524.6	786.0	--	--
5	Co(II)-cystein-2-amino iso butyric acid	3340.5	2977.9	1596.9	547.7	732.9	2584.4	447.5
6	Ni(II)-cystein-2-amino iso butyric acid	3348.2	2916.2	1620.3	540.0	779.2	2584.4	455.2

Magnetic Susceptibility:

Measurement of effective magnetic moment μ_{eff} can be used to estimate the number of band type or oxidation state of a Co-ordination metal ion. At its simplest mean using the so called “spin-only” formula.

$$\mu_{\text{eff}} = \sqrt{n(n+2)} \text{ BM} \quad \text{Where, BM =Bohr magnetons.}$$

For present study effective magnetic moments has been studied at room temperature. The value of μ_{eff} are:

Table-3: Results of Magnetic Susceptibility

Sr. No.	Complexes	Colour	Solubility	μ_{eff} (BM)
1	CO(II)-cystein	Pink	CCl_4	1.66
2	Ni(II)-cystein	Light green	Methanol	1.80
3	Ni(II)-2-amino isobutyric acid	Light blue	Ethanol	1.69
4	Co(II)-2-amino isobutyric acid	Violet	Hot water	1.73
5	Co(II)-cystein-2 amino isobutyric acid	Pink	Chloroform	1.84
6	Ni(II)-cystein-2-amino isobutyric acid	Light green	Dioxane	1.85

J.K. Nag, D. Das, S. Pal and C. Sinha¹⁶ reported synthesis and magnetic moments of copper and Schiff base. The magnetic moments found are (1.7– 1.8 BM) deviated slightly from the spin values. The homodinuclear copper complex shows very low magnetic moment, 0.80 BM per copper centre. In case of Co(II) complexes the observed moment for the spin free octahedral and tetragonal distorted octahedral

complexes which are in excess of the spin-only value of 0.8 to 1.3 BM. The spin spread Co(II) complexes having moments in the range 2.2 – 2.9 BM are more certainly square planar in structure¹⁷. The magnetic moment values suggest that the Co(II) complexes are of low spin type with one unpaired electrons¹⁸. If the ligand have only a weak field the splitting is small, and only when each of the t_{2g} and e_g level contains one such complexes are called as high spin or spin free. The magnetic moment value of 0.78 BM in close to the range observed for same square planar Ni(II) complexes in which the partial paramagnetic (0.94 – 1.24 BM) considered to be caused by spin crossover from singlet to triplet state¹⁹. Xishi Tai et al²⁰ reported synthesis of transition metal complexes of Novel Schiff base ligand. The elemental analysis indicates the complex of H_2L with Co(II), Cu(II) can be formulated as Metal-ligand. The IR band appearing at $420 - 430\text{ cm}^{-1}$ and $541 - 560\text{ cm}^{-1}$ are assigned to $\nu_{(M-N)}$ vibrations. It also suggested that the complexes are nearly square planar coordinated accordingly. Different worker²¹⁻²² reported in the synthesis of Trioxime, Hydrazone complexes with some metal like Ni^{2+} , Cu^{2+} , Co^{2+} etc. the magnetic susceptibility measurements of Ni(II) – complex indicate that this complex is diamagnetic and magnetic moments of Co(II) complex are 1.76 BM. Bayazeed H. Abdullah²³ showed the complex formation of Cd(II), Ni(II) and Cu(II) with N-phenyl-N(2-pyrimidyl) thiourea shows BM 3.06, 2.15 susceptibility measured at room temperature.

REFERENCES

1. P.V. Mary Kutty and Geetha Parameswaran, *Asian J. Chem.*, **13(9)**, 905(2001).
2. Liang – Nian Ji and Jim Gang Liu, *J. Ind. Chem. Soc.*, **78(10-12)**, 565, (2001).
3. Sami A. Zabin and Tejurkar, *Asian J. Chem.*, **7(3)**, 542,(1995).
4. Parveen Sharma and Surendra N. Dubey, *Proc. Indian Acad. Sci.* **106(1)**, 23,(1994).
5. Takaji Yasai, Jinsai Hidaka and Yoichi Shimura, *J. Indian Chem. Soc.* **99(12)**, 921,(2002).
6. Ru-Hu-Hai, Yuan, Li and Xian-Jun, *Chemical abstract* 136, 5, 7523,(2002).
7. Zakiyan, Iffet. Gunduz, Neela Turgul. *Mereel-dekker. Inc.* ,**31(7)**, 1175, 1187,(2001).
8. Y. Jadegoud, Omkar B. Ijare, N.N. Mallikarjuna, S.D. Angadi and BHM Mruthuyjayasyamy, *J. Indian Chem. Soc.* **79**,12, 921-924,(2002).
9. N.N. Orlova, V.A. Akseveva, V.A. Seliolovkin, N.S. Bogdanova and G.N. Pershin, *Russ. Pharm. Toxicol.*, 348, (1988).
10. G. Dognagk, R. Bebrisch, F. Mietzsch and H. Schmuadt, *Naturwissen Schaten*, **33**, 315, (1946).
11. U. Shrivastava, R.B. Pathak and S.C. Bahel, *J. Indian Chem. Soc.* **58**, 822,(1981).
12. K. Hussain Reddy, *J. Indian Chem. Soc.* **80**, 67-78,(2003).
13. K. Nakamoto – Infrared Spectra of Inorg. and Co-ord. Comps, John Wiley and Sons NY,(1963).
14. H. Shinde and T.C. Brown, *J. Am. Chem. Soc.* **87**, 1904(1965).
15. S.N. Chube, J.P. Shreevastava and L.K. Mishra, *Inorg. Chim Acta*, **23**, 1,(1977).
16. J.K. Nag, D. Das, S. Pal and C. Sinha, *Proc. Ind. Acad. Sci. (Chem. Sci.)* **113(01)**, 17(2001).
17. M.C. Day and J. Selbin, “Theoretical Inorg. Chem.” 2nd Edn. EWP. 487 – 494.
18. H.C. Busch and A. Mitra, *Indian J. Phya.* **26**, 393,(1952).
19. P.R. Shukla, V.K. Singh and A.M. Jaiswal, *J. Indian Chem. Soc.* **60**, 321,(1983).
20. Xishi Tai, Xianghong Yin, Qiang Chen and Minyu Tan, *Molecule* **8**, 439 – 443,(2003).
21. N. Sarikavkhi and G. Irez, *Turk J. Chem;* **29**, 107-115 (2005).
22. E. Capolat and M. Kaye, *J. Co-ord. Chem.* **58**, 14, 1217, 1224,(2005).
23. Bayazeed H. Abdullah, **19**, 5, 3903-3910,(2007).

(Received: 22 January 2010

Accepted: 12 February 2010

RJC-513)