Microncosmos International Journal of Research (MIJR)

www.mijr.org

A Novel Route of Synthesis for Schiff bases derived from 4-Chloro Benzaldehyde with 2 Amino 4-(P-Ethoxy Phenyl) Oxazole

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Abstract

Transition metals of 3d series are of immense importance due to their role in biological activity. A number of 3d metals are generally toxic in nature. For example cu having concentration more than 10-9 gm is toxic, it has been observed the toxicity of metal complexes can be reduced by the formation of their transition metal complexes. Keeping above fact in mind a novel route detected for the synthesis of Schiff bases of 3d metal series with 4-chloro benzaldehyde with 2-amino (4-ethoxy phenyl oxazole. The characterization of ligand as well as their metal complexes has been made on the basis of elemental analysis, magnetic susceptibility measurement, Infrared spectra and magnetic moment data.

Article Details

Submission: 27-09-2015
Re-submission: 30-09-2015
Publication: 31-10-2015

Key Words:
Co (II), Ni (II), Mn (II) and Zn (II) complexes, Schiff base

Introduction

The derivative of oxazoline has been found to show a wide range of biological activity1.They are mainly used as asanalgesic, nematocides, bactericides, fungicides etc. In the present day therapy these oxazolines are also popular for their radio protective activities. The versatility of oxazoline and their derivative is demonstrated by the fact that some of these compounds possess antimalarial2, anthalmintic3, antifungal4,5, antimalarial6, antiplasmodic7 and antitubercular8 activities. These compounds are also used as local anaesthatic9 anti radiation drugs10 anti viral and anti neoplastic agent11and above all vulcanization accelerators12 in the rubber industry. Hence the role of oxazoline and their derivative cannot be avoided in medicinal as well as industrial field. Keeping above fact in mind we go for the synthesis of metal complexes of oxazoline and also evaluated their medicinal property.

Experimental

The entire reagent used were of AR grade otherwise purified before used. Experiment was performed in two steps (I)Synthesis of ligand i.e. Schiff base and (II) Synthesis of 3d transition metal complexes with Schiff base. Both step was performed as follows:

Synthesis of Schiff base from 4-chloro benzaldehyde and 2-Amino 4(p-ethoxy phenyl) oxazole

The Schiff base was prepared by the condensation of 4-chloro benzaldehyde with 2-amino 4(p-ethoxy phenyl) oxazole in 1:1 using liquid bromine as condensing agent. First 4-chloro benzaldehyde 0.02 mole was added in 50 ml absolue ethanol and another solution of 0.02 mole of 2-amino 4(p-ethoxy phenyl) oxazole in 50 ml. absolute ethanol separately. Both of the solution was mixed together in round bottom flask at room temperatue and shaked well. Further the reaction mixture was refluxed for four hours in a round bottom flask using water condenser fitted with anhydrous silica gel guard tube at the top of the condenser. After the reaction approximately 50% of the alcohols were distilled off and the reaction mixture was cooled by immersing the flask in ice. The precipitate obtained was filtered, washed three times by minimum amount of absolute ethanol (ice cooled) and dried in electric oven at 60ºC-65 ºC

Synthesis of 3d transition metal complexes with Schiff bases derived from4-chloro benzaldehyde and 2-Amino 4 (p-ethoxy phenyl) oxazole

The complexes was synthesized using 1:2 metal and ligand solution in ethanol. In order to prepare 0.02 mole Schiff base solution 0.547 gm ligand was dissolved in 100 ml absolute ethanol and similarly the solution of transition metal salt was prepared in absolute ethanol for the concentration of 0.01 mole. In the same manner the solutions of other transition salt of 3d series such as [Mn(II),Co(II),Ni(II) and Zn(II)] was prepared in same solvent. Excess amount of ligand solution was used in reaction mixture. The reaction mixture is now refluxed in

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Citation: Joshi, A, Lal, N G Kumar, R. (2015). A Novel Route of Synthesis for Schiff bases derived from 4-Chloro Benzaldehyde with 2 Amino 4-(P-Ethoxy PhenyL) Oxazole. *Microcosmos International Journal of Research*, 1(2):12-14.

TABLE 1
COLOUR, ANALYTICAL, CONDUCTANCE AND MAGNETIC MOMENT DATA OF METAL COMPLEXES

Complex	Color of the complex	Elemental analysis % calculated (found)						Ωcm/mol	$?_{eff}$(BM)
		M	C	H	N	O	Cl		
$[Mn(C_{12}H_{11}N_2O_2Cl_2)_2](NO_3)_2$	Light Yellow	8.08	42.35	3.23	12.35	23.53	10.44	59.2	5.4
		8.06	42.33	3.21	12.33	23.51	10.41		
$[Co(C_{12}H_{11}N_2O_2Cl_2)_2](NO_3)_2$	Buff	8.61	42.11	3.21	12.28	23.39	10.38	58.4	2.91
		8.59	42.09	3.19	12.26	23.37	10.36		
$[Ni(C_{12}H_{11}N_2O_2Cl_2)_2](NO_3)_2$	Dull Green	8.58	42.12	3.21	12.28	23.40	10.38	61.4	3.01
		8.56	42.1	3.19	12.26	23.38	10.36		
$[Zn(C_{12}H_{11}N_2O_2Cl_2)_2](NO_3)_2$	cream	9.47	41.71	3.18	12.16	23.17	10.28	55.2	2.76
		9.45	41.69	3.16	12.14	23.15	10.26		

In above table M=Metal C= carbon H= Hydrogen N= Nitrogen O= Oxygen Cl= chlorine.

a round bottom flask for one an hour at 80-90 0c temperature .

The reaction mixture was concentrated approximately half of its total volume and then cooled in ice. Different colored precipitates for different metal ions are obtained. The precipitate were filtered washed with ice cold absolute ethanol and dried in electric oven at 85-95 ºC. Elemental analysis and spectral data are shown in table.

Results and Discussion

Yield (Calculated/observed) = 9.06/7.86 gm Percentage =87% Melting point= 189 ºC

Analytical data suggested 1:1 stoichiometry for the binary complexes. The complexes were soluble in common organic solvents *i.e.* DMSO(Dimethyl silane organic solvent) and DMF. (Dimethyl furane)

The magnetic moment values of complexes are found to be 2.76 - 5.40 B.M. The spectra of these complexes exhibit three bands at 10900, 16100 and 22400 cm-1 corresponding to the transition 3A2g(F) 3T2g(F), 3A2g(F) 3T1g(F) and 3A2g(F) 3T2g(P) respectively, which corresponds to octahedral geometry7. Magnetic moment value of the present Mn(II) complexes was 5.80BM indicating that 6A1g as ground state for d5 configuration in high spin octahedral stereochemistry. The electronic spectra of Mn(II) complexes exhibit three bands at 15650-15500, 18630-18400 and 23650-23400 cm-1 which can be assigned to the transition 6A1g 4T1g, 6A1g 4T2g and 6A1g 4Eg, 4A1g(G) respectively. From a careful comparison of the infrared spectra of metal complexes with those of ligands, it is inferred that a band at 1200cm-1 due to C=N bonding disappears during chelate formation. This indicates complex formation between the metal cations and the ligand leading to the generation of a new entity. In the chelate the band observed around 500cm-1 correspond to M-O vibration suggests that phenolic groups are involved in bond formation with metal ions. The M-N stretching frequency in the Schiff base complex is obtained at higher wave number because of the character of M-N due to M-N ? interaction. Nakamato7-8 has shown that M-N stretching frequency undergo coupling with other stretching, vibration resulting in a number of bands. The bands around 730 and 610

TABLE 2 ZONE INHIBITION (mm)

Complexes	Solvent	E.Coli 604	S.aureus 86	B.subtilis 430	S.typhi 510	A.niger 261
[Mn(C12H11N2O2Cl2)2](NO3)2	Carbon tetra chloride	_	_	_	_	0.4
[Co(C12H11N2O2Cl2)2](NO3)2	Carbon tetra chloride	_	_	45	_	14
[Ni(C12H11N2O2Cl2)2](NO3)2	Carbon tetra chloride	_	_	34	15	10
[Zn(C12H11N2O2Cl2)2](NO3)2	DMSO	_	_	_	_	32

cm-1 may correspond to the coupled ?(M-N). From these results it is concluded that the primary ligand is being utilized with various species showing absence of CHO group and generation of new entities. Further weight loss in the complexes at 140-180°C corresponds to a coordinated water molecule.

Bacterial Screening

The anti microbial activity of the ligands and synthesized ternary complexes were evaluated by the paper disc plate methods. The MTCC (Microbial type culture collection) culture of E.coli, S.aureus, B. Subtilis. S.typhi and A.niger were taken for the antimicrobial screening. The result of the antibacterial screening in terms of zone of inhibition is shown in table 2. The entire synthesized compounds along with the parent compound were screened for their antibacterial activities. DMSO and chloroform were taken as controlled standard. From the antibacterial screening it is observed that Ni-complexes was found to be more active against

B.subtilis, S.Typhii and A.niger as gram positive bacteria only whereas with gram negative bacteria no significant activity has been observed. Against A.niger all compounds were found active amongst the synthesized complexes. Only Mn-complexes show active against gram negative bacteria whereas other found inactive.

From the above results it has been observed that amongst the synthesized complexes were found active against S.aureus. Thus it can be concluded that it is worthwhile to per sue further investigation by modifying the structure as well as concentration ratio.

REFERENCES

1. B.N. Figgis, *Introduction to ligand field*, Willey eastern Ltd.New delhi p.279(1976)
2. R.P. Bhamaria, R.A. Bellare and C.V. DellwalaJ. *Synthesisi of mix ligand complexes Exp*. boil.6, 62(1968).
3. Sinha,C.; Trans. Met. Chem.,14(1994)
4. Sharma,C.L., Mishra, V. and Narvi, S.S.;Synth. React. Inorg. Met-Org. Chem.,(169) (2),243 (1986)
5. R.B. Pathak, B. Thana and S.C. Bahel, Antibacterial sources of mixed ligand complexes J. anti bact., Antifun. Agents, 8,12 (1980)
6. R.L. carlin & A.J. van drynevelidt, magnetic properties of transition metal compounds, springer- verlag New York (1977).
7. K. Nakamoto, infrared and raman spectra of inorganic coordination compounds Wille interscience new York p.308(1978).
8. Hankare, P.P., et al; Indian J. Chem., 43A, 2578 (2004)
9. Mishra, A.P. and Pandey, L.R.; *Indian J. Rehm*., 44A, 94 (2005)
10. A.K. Mishra, Niranjan lal. Synthesis and Characterization of '3D' Metal Complexes with 2-(Furan -2- Formylamino) *Sulphanilamldo thiazole asian J. Chem.* 20, 8, 6612, (2008)
11. A.K. Mishra, Niranjan lal Chemical Analysis of River water Collected from Differentsites of kali nadiOriental J. Chem. 24 1, 319, (2008).
12. A.K. Mishra, Niranjan lal Synthesis and Characterization of Cu(II), Ni(II) Co(II) andZn(II) Complexes with 2- Amino-4-p-bromophenyl Oxazole Asian J. Chem. 20, 7, 419, (2008).