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Synthesis and Characterization of Complexes of Mn(Ii) And Co (Ii) With N,N'-Orthophenylene Bis - (1-Phenyl-1,2-Butadione-2-Oxime)

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ABSTRACT

Complexes of Mn(II) and Co(II) ion with N,N'-Orthophenylene bis-(1-phenyl-1 butadione-2-Oxime) were synthesized and characterized by analyzing by I electronic spectra and magnetic moment susceptibility. Complexes of the ty $M(H_2L)X_2$ where M=Co(II) & Mn(II) and X=Cl^-, Br^-, I^-, No_3^- and ClO_4^- we obtained.

Keywords : IR, Electronic Spectra, Magnetic moment, Complexes of M $(H_2L)X_2$.

I. INTRODUCTION

Synthesis of tetradentate, pentadentate and hexadentate Schiff base ligands of varying ring size in which N donor atom or mixed donor atoms such as N,O and N S are reported to have bonded with metal ions. Both mononuclear and binuclear complexes with bridging groups have been characterized and their paramagnetic behaviour have been studied during last few decades. This kinetic liability and electrochemical behaviour of complexes formed by macrocyclic ligands have appeared in literature[1-5].

In this present work we have reported to synthesis and characterization of metal complexes of Mn(II) and Co(II) with the tetradentate Schiff base ligand H_2L. orthophenylene bis-(1-phenyl-1,2-butane dione-2-oxime) which behaves as a hexadentate ligand, complexes of the type $M(H_2L)X_2$ where M=Co(II) & Mn(II) and X= =Cl^-, Br^-, I^-, No_3^- and ClO_4^- have been synthesized where the organic ligand co-ordinates with the metal ions in their uncharged state. These complexes have been obtained by treating metal (II) salt with ligand in ethanolic solution in 1:1 molar ratio.

II. EXPERIMENTAL

We have prepared ligand H_2L and the metal complexes by using chemicals of reagent grade of Loba, Aldrich , E. Merck. As the reagents were of G.R. and A.R. grade hence used without further purification. Almost all reactions were carried out in alcoholic medium. The constituent part of each compounds have been estimated by established analytical method[6-7].

2.1 Preparation of ligand N-N'-Ortho-Phenylene-bis-(1-phenyl-1,2-butanedione-2-oxime. [H_2L]

Orthophenylene (0.05 mol) was mixed with 1-phenyl-1,2-butanedione-2-oxime(1 mol) with the help of agate mortar and pestle and titurating them together for thirty minutes. The whole amount of this solution was transferred into a beaker and was stirred vigorously for half an hour. During the course of stirring the reaction mixture gradually became more and more viscous. It was slightly cooled and viscous liquid solidified and dried. The M.P of compound was found to be 184°C.

2. 2 Preparation of metal complexes of type $M(H_2L)X_2$.

Where M= Co(II) & Mn(II)

X= =Cl^-, Br^-, I^-, No_3^- and ClO_4^-

An alcoholic solution of N,N'-Orthophenylene-bis-(1-phenyl-1,2-butanedione-2-oxime). H_2L were added slowly into an alcoholic solution of metal(II) salts with constant stirring. The whole mixture was refluxed on hot water bath for an hour. Crystals of metal complexes were obtained which filtered, washed and dried. These were analysed for their constituents.

Table 1

Colour and analytical data of the complexes of Co(II) Ni(II) Cu(II) with Ligand [H_2L]

SL.NO	Complexes	Colour	Metal % Calculated (Found)	Carbon % Calculated (Found)	Nitrogen % Calculated (Found)	Hydrogen % Calculated (Found)	Anions % Calculated (Found)
1	H_2L	---	---	73.239 (72.75)	13.146 (12.68)	6.103 (5.64)	---
2	CoH_2LCl_2	Violet	10.60 (9.87)	56.122 (55.74)	10.073 (9.46)	4.676 (4.13)	12.771 (12.04)
3	CoH_2LBr_3	Pink	9.137 (8.75)	48.377 (47.74)	8.683 (7.95)	4.031 (3.26)	24.808 (24.18)
4	CoH_2LI_2	Blue	7.975 (7.02)	42.223 (41.66)	7.578 (6.91)	3.518 (3.01)	34.377 (33.78)
5	$CoH_2L(ClO_4)_2$	Pinkish	8.616 (8.12)	45.618 (45.14)	8.187 (7.54)	3.801 (3.16)	10.381 (9.87)
6	MnH_2LCl_2	Yellow Pink	9.846 (9.36)	55.919 (55.26)	10.036 (9.55)	4.659 4.12	12.725 (12.18)
7	MnH_2LBr_2	Orange	8.571 (8.01)	48.678 (48.01)	8.737 (7.14)	4.056 (3.26)	24.963 (24.23)
8	MnH_2LI_2	Pinkish Yellow	7.475 (6.64)	42.452 (41.28)	7.619 (16.86)	3.537 (2.95)	34.56 (33.75)
9	$MnH_2L(NO_3)_2$	Pink	9.081 (8.45)	51.575 (50.64)	13.885 (13.24)	4.297 (3.64)	---
10	$MnH_2L(ClO_4)_2$	Deep Pink	8.080 (7.24)	45.886 (45.01)	8.236 (7.66)	3.823 (3.04)	10.442 (9.87)

Table2. Significant IR Spectral bands (cm-1) in the spectra of type[M(H2L)X2] where M=Mn(II) Co(II)

SL.NO	Complexes	ν O – H	S N – OH	νC=N azomethine	ν C=N oxime	ν N – O	νM – X
1	$Mn(H_2L)Cl_2$	3380 (m, b)	1685 (m)	1605 (m)	1500 (s)	1100 (s)	620 (s)
2	$Mn(H_2L)Br_2$	3385 (m, b)	1675 (m)	1600 (m)	1490 (s)	1105 (s)	615 (s)
3	$Mn(H_2L)I_2$	3385 (m, b)	1680 (m)	1600 (m)	1495 (s)	1100 (s)	620 (s)
4	$Mn(H_2L)(NO_3)_2$	3375 (m, b)	1680 (m)	1595 (m)	1490 (s)	1100 (s)	615 (s)
5	$Mn(H_2L)(ClO_4)_2$	3380 (m, b)	1685 (m)	1590 (m)	1495 (s)	1105 (s)	610 (s)
6	$Co(H_2L)Cl_2$	3385 (m, b)	1670 (m)	1590 (m)	1490 (s)	1105 (s)	610 (s)
7	$Co(H_2L)Br_2$	3380 (m, b)	1680 (m)	1600 (m)	1495 (s)	1100 (s)	620 (s)
8	$Co(H_2L)I_2$	3380 (m, b)	1680 (m)	1605 (m)	1500 (s)	1095 (s)	610 (s)
9	$Co(H_2L)(NO_3)_2$	3385 (m, b)	1685 (m)	1595 (m)	1495 (s)	1100 (s)	615 (s)
10	$Co(H_2L)(ClO_4)_2$	3380(m, b)	1680 (m)	1600 (m)	1505 (s)	1080 (s)	610 (s)

Table 3. Position of Absorption bands and magnetic moment values of the type $M(H_2L)X_2$ in cm^{-1}

Complexes	$^6A_1g \rightarrow {}^4T_1g$	$^6A_1g \rightarrow {}^4T_2g$	$^6A_1g \rightarrow {}^4Eg$	$^6A_1g \rightarrow {}^4A_1g(G)$	μ eff in B.M
$Mn(H_2L)Cl_2$	16200	23000	37000	45.000	5.80
$Mn(H_2L)Br_2$	16150	23000	37150	45.200	5.85
$Mn(H_2L)I_2$	16300	23200	37300	45.300	5.80
$Mn(H_2L)(NO_3)_2$	16450	23700	37200	45.200	5.90
$Mn(H_2L)(ClO_4)_2$	16600	23700	37400	45.300	5.90
	^4T_1g (F) $\rightarrow {}^4T_1g$ (P)	----	Charge Transfer bands	----	μ eff in B.M
$Co(H_2L)Cl_2$	16000		24000		5.10
$Co(H_2L)Br_2$	16100		23800		5.14
$Co(H_2L)I_2$	16200		23500		5.20
$Co(H_2L)(NO_3)_2$	16100		22700		5.16
$Co(H_2L)(ClO_4)_2$	15800		22900		5.20

III. RESULT AND DISCUSSION

3.1 Interpretation of IR Spectra

The IR absorption spectra of the compounds have been taken on backmann I.R.-20 spectrometer. From the studies of IR spectra it is clear that in the high frequency region the ligand and complexes of the type $M(H_2L)X_2$ show a strong and broad band in the spectra which lies near 3380 cm^{-1}. The band can be ascribed to νO-H of the oxime group (N-O-H). The width of the band manifests intramolecular hydrogen bonding. The spectra of all the metal complexes show two sharp and strong bands in the vicinity of 1600-1500cm^{-1} which have been attributed to >C=N stretching of azomethine and oxime groups respectively. The characteristic behaviour of these bands respectively towards red and blue regions of the spectra. The shift of νC=N band and the existence of a hydrogen bonded OH group as evidenced by the infrared spectra for the series of metal complexes of the type $M(H_2L)X_2$ with uncharged ligand clearly manifest a hydrogen bonded cyclic ring structure.

3.2 Electronics spectra and magnetic moments:-

Mn(II) d^5 has five unpaired electrons in weak octahedral and tetrahedral field. The μ_s=5.92 BM shows 65 free ion ground term which transforms as $^6A_{1g}$ in octahedral and $^6A_1$ in tetrahedral symmetry. In strong octahedral crystal field there is only one unpaired electron which ground term splitting is as $^2T_{2g}$. The spin free planar complexes has μ_s=5.92 BM, where as μ_s=3.87 BM for partial spin paired complexes; in the present investigation of Mn(II) complexes of type $M(H_2L)X_2$ show magnetic moment in range 5.80-5.90 BM at room temperature. The spectra consists of four bands one in the 16200-16600 cm^{-1}, the next higher in the region 23000-23700 cm^{-1} followed by two intense bands in the region 3700 and 45000-45300 cm^{-1}.

These are assigned as

$^6A_{1g} \rightarrow {}^4T_{1g}$

$^6A_{1g} \rightarrow {}^4T_{2g}$

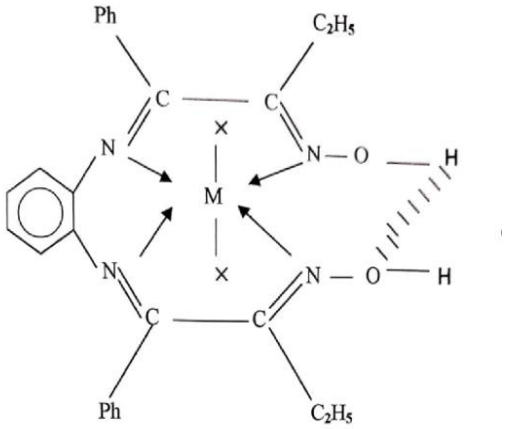
$^6A_{1g} \rightarrow {}^4E_g$ and

$^6A_{1g} \rightarrow {}^4A_{1g}$ (G)

Suggested octahedral structure.

The Co(II) ion has a d^7 system which give rise to the ground state, 4F and which excited state 4p located above ≥ 14500 cm^{-1} under the influence of an octahedral field, the ground state splits into three levels the orbital triplet $^4T_{1g}$, $^4T_{2g}$ and $^4A_{2g}$.

The present series of cobalt(II) complexes of the type $Co(H_2L)X_2$ posses magnetic moment 5.1-5.2 BM which correspond to an octahedral arrangement about the metal ion having the ground state 4F which splits into $^4T_{1g}$, $^4T_{2g}$ and $^4A_{2g}$. whose energy level increases successively one of the important features of the spectra is the appearance of a strong charge transfer band above29000 cm^{-1} and has a few cases impeded clear resolution of the spectra in the vicinity of this region. The cobalt(II) complexes of the type $M(H_2L)X_2$ exhibits a multiplet band structure in the region 15800-16100cm^{-1} and possess magnetic moment between 5.10-5.20 BM which corresponds to an octahedral arrangements. The band can be assigned to the transition $^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$. Thus these spectral features support the structure of metal (II) complexes of type $M(H_2L)X_2$ as



$M(H_2L)X_2$

Where M=Co(II), M_n(II)

X=Cl^-, Br^-, I, NO_3^-, ClO_4^-

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