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Title Key: Synthesis And Cherecterization Of Some Trasition ……

Synthesis and Cherecterization of Some Trasition Metail Complexes With Hyadrazone Schif Bases

Bhagat S. M.

Department of chemistry I.C.S. College of Arts, Comm. and Science, Khed.
Dist. Ratnagiri (MH)- 415 709.

Abstract

In this paper authors should explain the methodology used to prove the result. Abstract should be 6-8 lines. The various components of your paper [title, text, heads, etc.] are already defined on the style sheet, as illustrated by the portions given in this document. (Abstract).

Keywords: Metal, chelate, Metal ion chelates

1. Introduction

Metal complexes is one of the most important field due to its versatile application. Certain metal complexes are used in Industries[1,2] , for synthesis, It work as catalyst [3] , they have some application in Agriculture [4]. Particularly hydrazine metal complexes are bio-active compound and can used as anti-tubercular[5], antimicrobial[6]Antifungal[7],anticancer[8],Anti-convconvulasant[9] and antiinflamatori[10] activity. Particularly, the antibacterial and antifungal properties of hydrazone and their complexes with some transition metal ion is reported in letarature. The chelating agent like hydrazine contain C=N linkage is essential for biological activity . Several azomethazines were reported to possess remarkable antibacterial, antifungal, anticancer active.

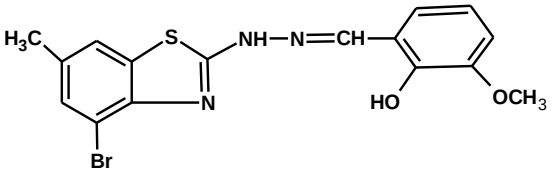
In present study I reported some transition metal ion complex and charecterised them.

Synthesis of ligand

2-(2'-hydroxy-3'-methoxy phenyl)-4-bromo-6-methyl benzothiazolylhydrazones

2.48 of gm 4-bromo-2-hydrazino-6-methyl benzothiazolylhydrazones is dissolved in alcohol and alcoholic solution of 2-hydroxy-3-methoxybenzaldehyde were refluxed for 1 hour using water condenser. The reaction solution were cooled and filtered, the product was recrystallized using alcohol in hot condition. The purity of compound is confirmed by TLC and melting point.

Structure of ligand



2-(2'-hydroxy-3'methoxy phenyl)-4-bromo-6-methyl benzothiazolylhydrazon.

Melting point-175 °C,Empiricalformula-$C_{16}H_{14}N_3BrSO_2$,Exact mass -392

This ligand is referred as HMPBMBTH

Synthesis of complexes.

i) Synthesis of 2-(2'-hydroxy-3'-methoxy phenyl)-4-bromo-6-methyl benzothiazolylhydrazonesFe^{III} chloride complex

100 ml 0.1 M $FeCl_3.4H_2O$ were prepared in alcohol and 2-(2'-hydroxy-3'-methoxy phenyl)-4-bromo-6-methyl benzothiazolylhydrazones 0.2 M solution were prepared in ethyl alcohol. These two solutions

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were mixed and transfer into 500 ml round bottom flask attached water condenser, the pH is of the reaction mixture were adjusted by adding basic buffer solution pH-10. Reaction mixture were refluxed for one hour in water bath. The precipitate was obtained. it is digested, after cooling it is filtered through Buckner funnel , the precipitate of complex were purified by washing with ethyl alcohol, the complex were dried by keeping it in oven. The product was packed into sample bottle.

iv)Synthesis of cobalt complex

Cobalt chloride and ligand 2(2'-hydroxy-3'-methoxy phenyl)-4-bromo-6-methyl benzothiazolylhydrazone (HMPBMBTH) were dissolved separately in ethanol so as to prepare 0.1 molar solution with constant stering. A clear solution of cobalt chloride was mixed in ligand solution in 1:2 proportion and pH is adjusted to 6.5 with buffer solution and refluxed on water bath for one hour and allowed to cool .the contents were diagested for one hour and filtered. Pale pink coloured solid is obtained it washed with ethanol and dried and stored in bottle.

Physical parameter and elemental analysis .

Melting point of complexes are determined with the help of melting point apparatus by open capillary method. Chlorine is estimated by Mohr's method. Metal ion percentage in a complexes is determined by E.D.T.A. titration. M:L ratio is determined by heating known weight of complexes in platinum crucible. Physical parameter and analytical data of the Fe(II), Co(II),complexes and ligand HMPBMBTHare given in table 2.1. metal ligand ratio and empirical formula were assigned on the basis of TGA measurements and elemental analysis (table 2.2.)

3.5 Characterization of complexes.:-

U.V. and visible spectra of complexes and ligand recorded on U.V. SHIMADZU UV3600 spectrophotometer at range 200-800 nm by using D.M.S.O. solvent at P.G.

department of chemistry Shivaji University Kolhapur. I.R. spectra of ligand were recorded at YeshwantMahavidyalaNanded and I.R. spectra of complexes are recorded at PERKIN ELMER spectrum-100/79720 by KBrplatelate method at Shivaji University Kolhapur. Thermo gravimetric analysis (T.G./D.T.A.) measurement are recorded on thermo gravimetric analyzer on TA model S.T.D-2960 at Shivaji University Kolhapur in Nitrogen atmosphere.XRD pattern of the complexes recorded on PW-3719/1710 Philips –Holland spectrometer at Shivaji University Kolhapur and E.S.R. is recorded at IIT, pawai, Mumbai.

2. Result and discussion

The complexes of Fe(III), Co(II), are prepared with the ligand HMPBMBTH . This complexes are coloured. These complexes are soluble in D.M.S.O. but insoluble in water, alcohol, chloroform, and D.M.F. Decomposition point of complexes are in the range of 240-300°C . It suggest that they have good thermal stability at room temperature.

Table.2.1: physical property of HMPBMBTH metal complexes.

Complex	color	D.P.	Yield %	%Cl
[Fe(HMPBMBTH) 2Cl H_2O] Cl	Faint brown	272-280	68	18.6
[Co(HMPBMBTH)$_2$]$Cl_2.H_2O$	Pale pink	243-249	64	7.61

Electronic spectra *:-*

Theligand 2-(2′-hydroxy-3′-methoxyphenyl)-4-bromo-6-methyl benzothiazolylhydrazones (HMPBMBTH) has exhibited one characteristic maxima in U.V. region at 344 nm while in [Ni(HMPBMBTH) H_2O] Cl_2 complex it is shifted towards blue shift i.e. lower region and observed at 324nm. While in [Cu(HMPBMBTH) Cl] $Cl.H_2O$ complex the band is shifted towards red shift and observed at 352 nm . This shifting of bands indicate that there is complexetion in metal and ligand.

Table.2.2: Percent C,H,N and metal ion in HMPBMBTH metal *complex*

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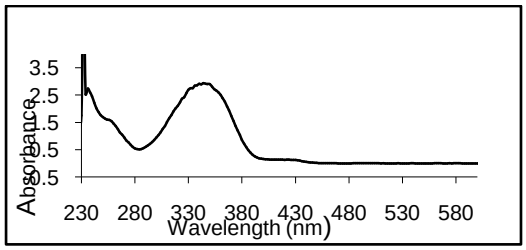
compond	M.wt	Empirical formula	%C	%H	%N	%M
HMPBMBTH	392.17	$C_{16}H_{14}N_3BrSO_2$	49.005	3.569	10.714	-
[Fe(HMPBMBTH)2Cl H_2O] Cl	572.52	$C_{16}H_{16}Cl_3FeN_3SBrO_3$	33.568	2.794	7.339	9.755
[Co(HMPBMBTH)$_2$] Cl_2. H_2O	932.28	$C_{32}H_{30}Cl_2CoN_6S_2Br_2O_5$	44.228	3.217	9.053	6.322

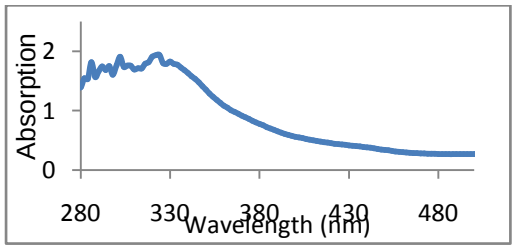
Table.2.3Electronic data of ligand HMPBMBTH metal complexes.

U.V. of HMPBMBTH U.V. of Fe(III) complex wit HMPBMBTH

Compound	Wavelength	Log E
Ligand HMPBMBTH	344	2.942
[Fe(HMPBMBTH) 2Cl H_2O] Cl	324	1.936
[Co(HMPBMBTH)$_2$] Cl_2. H_2O	352	2.009



Co complex with (HMPBMBTH)



I.R. Spectra :-

The ligand 2(2′-hydroxy-3′-methoxy phenyl)-4-bromo-6-methyl benzothiazolylhydrazones exhibit a sharp strong band at 3533 cm-1in I.R. spectra which may be assign to phenolic hydroxyl group In [Fe(HMPBMBTH) 2Cl H2O] Clcomplex borad strong is observed at 3435 and in [[Co(HMPBMBTH)2] Cl2. H2O complex broad band is observed at 3456 cm-1 . The shifting of band indicate that the coordination of metal ion through 'O' of phenolic OH group. In I.R. spectra of one band is observed at 1616 cm-1 it is due to C=N (ring) but in but in [Fe(HMPBMBTH) 2Cl H2O] Clit is observed at 1600 cm-1 this shifting of band in complex clearly indicate that Nitrogen of benzothiazine ring is involved in the complex formation. The band is observed at 1605 cm-1 in [Co(HMPBMBTH)$_2$] Cl_2. H_2O this shifting of band is indicate that 'N' of thiazol ring is involved in the complex formation .One band isobserved at 1580 cm-1 in ligand it may be due to the C=N (azomethine) group this band is shifted to lower region in [Fe(HMPBMBTH)$_2$Cl H_2O] Cland observed at 1560cm-1 it indicate that azomethine nitrogen is involve in the formation of coordinate bond with Fe^{+3}. In [Co(HMPBMBTH)$_2$] Cl_2. H_2O complex band is observed at lower region and it appear at 1530 cm-1 is indicate that the azomethine nitrogen is involved in the complex formation. Another band is observed at 3163 in I.R. spectra of ligand which may assign N-H stretching but in [Fe(HMPBMBTH)2Cl H2O] Cland [Co(HMPBMBTH)2] Cl2. H2O it is not observe because it may merged in the broad peak of OH group and water molecule this

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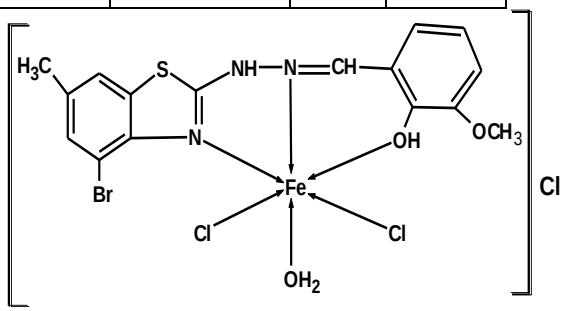
indicate that N-H is not involve in the complex formation. Another band is 595 in $[Fe(HMPBMBTH)_2Cl\ H_2O]\ Cl]\ Cl_2$ and $[Co(HMPBMBTH)_2]\ Cl_2.\ H_2O$ complexes but not observed in ligand it indicate that there is formation of M-O bond in both complexes.

Table.2.4 I.R. data of ligand HMPBMBTH metal complex

One band is observed at 515 cm-1 in both

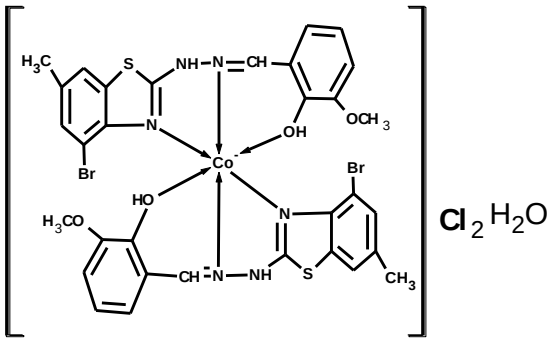
compound	O-H cm^{-1}	N -H cm^{-1}	C=N cm^{-1} ring	C=N cm^{-1} azomethine	M-O cm^{-1}	M-N cm^{-1}
HMPBMBTH	3533	3163	1515	1580	--	--
$[Fe(HMPBMBTH)_2Cl\ H_2O]Cl$	3435	-	1600	1560	595	515
$[Co(HMPBMBTH)_2]\ Cl_2.H_2O$	3458	-	1605	1530	658	523

complex which is absent in ligand this indicate that the formation of M-N coordinate band.Thus 2-(2'-hydroxy-3'-methoxy phenyl)-4-bromo-6-methyl benzothiazolylhydrazones act as tridentate in both complexes and coordinate through ring nitrogen, azomethine nitrogen and oxygen of phenolic OH . I.R. spectral data with probable is given in the table 2.4



Electron spin Resonance Spectroscopy.

The X-band E.S.R. spectrum of the powder Fe(II) and Co(II) complexes was recorded at room temperature. The calculated values of Fe(II) is $g_{\|}$, $g_{\perp}$, $g_{avg,}$ and G are 2.227,2.03674,2.10016, 4.26374 respectively. And Co(II) is $g_{\|}$, $g_{\perp}$, $g_{avg,}$ and G are 2.23474, 2.01126, 2. 0857533, 4.246 respectively. The



$[Fe(HMPBMBTH)_2Cl\ H_2O]\ Cl$
$[Co(HMPBMBTH)_2]\ Cl_2.\ H_2O$

values are typical for one unpaired electron in an orbital of mostly d_{xy} character. If $g_{\|}$ value is less than 2.3 the compound is covalent and $g_{\|}$ value is greater than 2.3 then it is ionic . Present values indicate that the complexes are covalent. G value is greater than 4 it indicate that theligand is weak field ligand.

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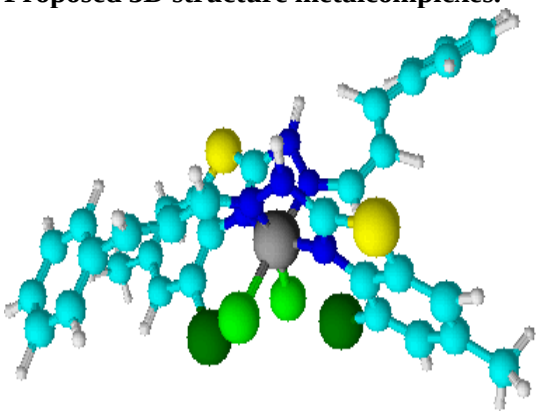
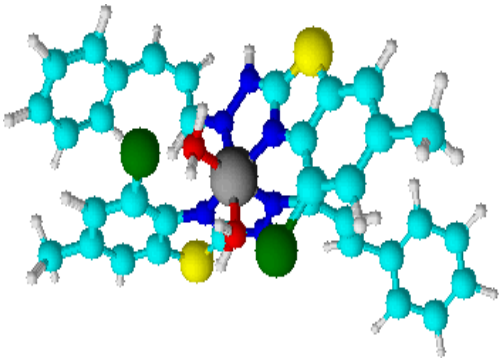
Proposed 3D structure metalcomplexes.





$[Fe(HMPBMBTH)_2 Cl H_2O] Cl$
$[Co(HMPBMBTH)_2] Cl_2. H_2O$

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