J. Chemtracks 20(1&2) 41-44, 2018

Abstracted / Indexed in
A division of the American Chemical Society
Chemical Abstract U.S.A.

ISSN 0973-239X
CODEN:JCOHF7
IMPACT FACTOR : 4.950

Synthesis of Fe(II) with Schiff's base chelating ligand 4' (p-hydroxy benzal diamino)-3,5 dimercapto -1,2,4 triazole

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Manuscript received online 16 November 2017, accepted on 13 December 2017

Abstract : The present paper deals with the preparation of shiff's base chelating ligand 4' (p-hyroxy benzal diamino) -3,5 dimercapto -1,2,4 triazole and its complexes with Fe (II). All together five complexes compound is formed of Fe (II) with schiff's base ligand of two donor atom sulphur and nitrogen. On the basis of elemental analysis, IR spectral studies, the complexes have been found to be bidentate.

(Keywords: Elemental analysis, IR spectral studies, chelating ligand)

Introduction

A Schiff's base metal complexes have been widely studied because of their industrial and biological applications[1-2]. It is reported that shiff's base metal complexes have a special role in the development of Inorganic chemistry[3-4].

Shiff's base are typically formed by the condensation of a primary amine with aldehyde/ ketone shiff's base that contains aryl substituents are substantially more stable.

In the present study, we report the formation of Fe (II) with 4'(p-hydroxy benzal diamino)-3,5 dimercapto -1,2,4 triazole and prepared five complexes compound with the help of secondary ligand H_2O, pyridue pyridine oxide, morpholine and α- picoline which has not been reported as yet.

Schiff's base are becoming important bio-chemistry analytical and antimicrobial agent Ramesh Kumar etal[5].

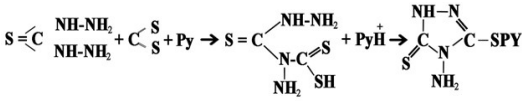
Experimental

Preparation of ligand:

Preparation of 4'-(p-hydroxy benzaldiamino) 3,5 dimercapto -1,2,4 tri-azole requires following steps.

Step I

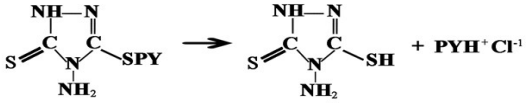
Preparation of monopridinium salt of 4 – amino -3,5 dimercapto -1,2,4 triazoles. Its preparation is carried out by the interaction of thio-carbohydrazide with carbon disulphies in pyridine.



Thio-carbohydrazide (10.6gm, 0.1m) and carbondisulphide (11.41ml, 0.19m) are taken in a round botton flask fitted with liebig condenser containing 100ml pyridine and refluxed with an oil bath for 1 hour.

Step II

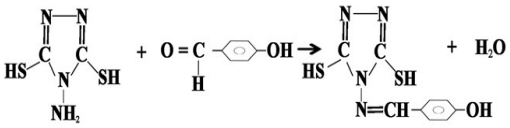
The pyridine salt (4.6 g) obtained as above is dissolved in hot water (20ml) and conc HCl(3ml) is added. The colored solid amino -3,5 dimeracapto – 1,2,4 triazole separated out immediately which was filtered out.The mp of crude product is found to be 216-217°C.



Step III

The preparation of ligand is achieved by refluxing the mixture containing equimolar quanties of 4-amino -3,5 dimercapto -1,2,,4 triazole with p-hydroxy benzaldehyde in ethanol for about one hour[6-7].

The reacton mixture is allowed to stand over night at room temperature, whereby yellow crystals are obtained.



4'(p- hydroxy benzaldiamino)
-3,5 dimercapto -1,2,4 troazole

Preparation of metal complexes:

(i) Preparation of complexes $[Fe\ L_2(H_2O)_2]$
Where L=4'(p – hydroxyl benzal diamino) 3,5 diamcapto -1,2,4 triazole

The complexes of (i) 0.0051M of ferrous chloride and 0.01M if ligand (LH), where dissolved separately in minimum quantity of alcohol and then mixed together[8-9]. The mixture was refluxed on water bath by one and half hour. The pH of the refluxed solution was raised to 8 and diluted with distilled water and finally with alcohol and dried in desiccators over anhydrous calcium chloride.
(ii) preparation of complexes of type $[Fe\ L_2\ \beta_2]$
Where β=(py, Py – o, α- Pico, Mor)

The alcoholic solution of 0.0051M Ferous chloride and the alcoholic solution of 0.01M Ligand were mixed together with about 2ml of appropriate base and the content were refluxed on water both at low temperature on cooling the precipitate separated out. The precipitate was filtered, washed with water, followed by several portion of alcoholic water and quietus base sedition. The precipitate was dried in dessicator over anhydrous $CaCl_2$

Results and Discussion

Table 1.1 shows the quantitative estimation of C,H,N,S and metal ions, in ligands and complexes

Table 1.1

S. No	Compound	% Fe Calculated (found)	% C Calculated (found)	% H Calculated (found)	% N Calculated (found)	% S Calculated (found)
01	$[FeL_2(H_2O)_2]$	9.21 (9.10)	36.62 (36.74)	3.98 (3.87)	18.92 (18.80)	19.36 (19.12)
02	$[FeL_2(PY)_2]$	8.90 (8.54)	32.12 (32.24)	4.52 (4.39)	18.75 (18.62)	17.92 (17.80)
03	$[FeL_2(PY\text{-}0)_2]$	7.98 7.66	44.38 (44.48)	4.24 (4.00)	19.15 (19.02)	15.39 (15.26)
04	$[FeL_2(Mor)_2]$	8.02 (7.96)	42.38 (42.21)	4.36 (4.21)	18.96 (18.88)	14.87 (14.69)
05	$[FeL_2(\alpha\text{-}Pico)_2]$	7.96 (7.81)	44.36 (44.46)	3.87 (3.71)	16.92 (16.79)	13.95 (13.82)

Where
LH = 4'-(p- hydroxy benzal diamino) -3,5-dimercapto – 1,2,4 – triazole
PY-O- Pyridinium oxide PY- Pyridine
Mor – Morpholine α- Pico – α- Picoline

Estimation of Iron:

Iron was estimated as ferric 8 – hyroxy quinolinate (oxinate) complex. Accurately 0.5gm of iron complex was converted into solution by decomposition of the complex with con HNO_3 and cone $HClO_4$ repeatedly[10-11]. The iron quantity was calculated from weight and the chemical factor for ion is 0.11437.

Estimation of Sulphur

The accurate weight of the complex weight 0.2gm was placed in a 500ml capacity round bottom flask containing 2-3ml liquid bromine and 20-30ml concHNO_3. The mouth of the flask was covered with conical funnel[11-12]. The solution contains sulphate by oxidation of adding excess of $BaCl_2$ solution. The barium sulphate preapitate was filtered, washed well with water, dried and weighted as barium sulphate. The gravimetric factor for sulphate is 0.13736.

IR spectral studies of complexes & Ligands

Table 1.2 shows. Important absorption bound in I.R spectrum of the Ligand and their assignment.

Table 1.2

S. No.	Frequency (cm^{-1})	Assignment
01	3260	t(> N-H) stretching
02	3150	t(O-H) Phenolic
03	3020	t(> C-H) stretching
04	2560	t(-S-H) stretching
05	1520, 1455	Skeletal vibration of Benzene ring
06	750	δ(C-H)
07	690	ν(C-S)

Table 1.3 show important IR band of LH and metal complexes where L = 4' (p – hydroxybenzal diamino) -3,5 – dimercapto – 1,2,4- trizole

Table - 1.3

S.No	Complexes	NH (cm^{-1})	OH (cm^{-1})	SH (cm^{-1})	M-N (cm^{-1})	M-S (cm^{-1})	M-O (cm^{-1})
01	[Fe L_2 $(H_2O)_2$]	3255	2850	-	300	285	450
02	[Fe L_2 $(PY)_2$]	3240	-	-	315	280	-
03	[Fe L_2 $(PY\text{-}o)_2$]	3255	-	-	300	285	435
04	[Fe L_2 $(Mor)_2$]	3245	-	-	315	275	-
05	[Fe L_2 $(\alpha\text{- Pico})_2$]	3250	-	-	320	280	450
06	LH	3260	-	2560	-	-	-

From the Table 1.2 Infrared spectra of phenolic (-OH) group appears near 3150-3050cm^{-1} indicating appreciable hydrogen bonding in phenolic –OH group; the N – H for free ligand appears as strong intensity at 3260 which is in the range of N-H for free ligand appears as strong intensity at 3260 which is in the range of N-H stretching vibration of primary and secondary amides 3570-3125cm^{-1} region. The weak band found 2560cm^{-1} in the I.R spectrum of the ligand may be contributed to S – H stretching vibration. The C – S group is obtained at 690cm^{-1} a weak absorption band in the region of 700 – 600cm^{-1} owing to C – S , stretching vibration .

Skeletal vibration of benzene ring structure in the 1600 – 1450cm^{-1} region[13]. The remaining two

bands 1520cm^{-1}and 1455cm^{-1}have been observed at identical values within the experimental limits.

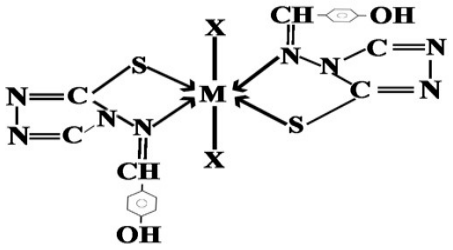
It has been reported in the literature that other and para disubshted compounds have a single strong band in the region 770 – 760cm^{-1}. The band appearing at 760cm^{-1} in the spectoum is assignable to C – H deformation Vibration.

Conclusion

Infra red spectoum of ligand and complex have been studied and the band position of important group and those of M – N, M – S, M – O have been assigned on the basis of work reported earlier[13-15]. New bands in the lower range near 450,315,275cm^{-1} are assigned to M – O, M – N, M – S respectively.

Thus the ligand behaves as bidentate monoamine co-ordinating through one deprotonated mercapto sulphur and azomethine nitrogen and thus behaving as heterochalting agent.

The structure of complexes on the basis of above discussion is assigned to



M^{+2} = Fe (II)

X = H_2O, Py, Py – O, Morpholine, α – picoline

The presence of some new band in IR spectra of complexes indicates the presence of pyridine, pyridine oxide, morpholine and α - picoline in co-ordinate sphere.

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Abstracted / Indexed in : Chemical Abstract U.S.A.
JC- 1275/2K18 (ISSN:0973-239X), INDIA
Journal Chemtracks Vol. 20 (1&2), 41-44, January to December, 2018