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Structural Studies of Some Complexes of Divalent Transition Metals with α(*o*-Hydroxy Aceto Phenone) Imino Aceto Hydroxamic Acid

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ABSTRACT: Some complexes of divalent Iron metal have been prepared with Schiff base ligand α(o-hydroxy aceto phenone) imino aceto hydroxamic acid in presence of bases containing nitrogen and oxygen atoms as donor sites. The ligand behaves as bi-anionic tridentate molecule. On characterisation of the ligand and the complexes by usual physico-chemical methods such as elemental analysis, measurement of electrical conductance, magnetic moment, electronic and IR spectra of the ligand and the complexes, all the complexes have been found to be nonelectrolyte, monomeric, paramagnetic and octahedral in geometry with general molecular formula $[Fe(L)(B)_3]$ where L = ligand and B = bases.

KEY WORDS: Solution, condensation, ligand, complex, divalent transition metal, separation, filtration, crystallisation, cation, mono-meric, octahedral, geometry.

INTRODUCTION: A lot of research work has been carried out in the field of coordination chemistry with Schiff bases having nitrogen and oxygen donor atoms as ligands. In the absence of Schiff bases as ligands, the field of coordination chemistry may be certainly shortened. But at least research work has been carried out for the formation of complexes of divalent transition metals with such Schiff bases which contain hydroxamic acid group in its moiety. Therefore, in this paper, I report the formation of some complexes of divalent

Iron with the Schiff base ligand, α(o-hydroxy aceto phenone) imino aceto hydroxamic acid in presence of bases like water, ammonia, quinoline, phenyl isocyanide, pyridine and different picolines. Such complexes have wide application in the field of agriculture and medicines for the treatment of curable and incurable diseases. They are good source to increase the level of haemoglobin in the human body.

PREPARATION OF IRON(II) COMPLEXES:

Procedure: About 2.10g (0.01 mole) of the ligand was completely dissolved in methyl alcohol. About 2.65g (0.01 mole) of Ferrous (II) sulphate heptahydrate was completely dissolved in about 20ml of aqueous ethyl alcohol. Both the solutions were gradually mixed together with regular shaking. Then, the resulting solution was refluxed on water-bath for about two and half an hour. During the course of reflux, the colour of the solution gradually changed and faint green and in some cases dirty and brownish green colour crystals separated out in the solution by keeping the solution in the cup-board for overnight. The product was separated by filtration and washed with a small amount of acetone and then dried over

KOH pellets placed in a desiccator.

The complexes of Fe(II) metal with the ligand have been prepared separately in case of each base like water, ammonia, quinoline, phenyl isocyanide, pyridine and different picolines.

Analytical Date of Fe(II) Complexes with the ligand in the presence of bases:

Table – 1

S.N.	Compounds	Metal	Carbon	Hydrogen	Nitrogen
1.	$[Fe(C_{10}H_{10}N_2O_3)(H_2O)_3]$	17.40 (17.72)	37.32 (37.97)	5.08 (5.06)	8.90 (8.86)
2.	$[Fe(C_{10}H_{10}N_2O_3)(NH_3)_3]$	17.44 (17.89)	38.10 (38.33)	6.10 (6.07)	22.42 (22.36)
3.	$[Fe(C_{10}H_{10}N_2O_3)(C_6H_5NC)_3]$	9.44 (9.80)	64.84 (65.14)	4.40 (4.37)	12.42 (12.35)
4.	$[Fe(C_{10}H_{10}N_2O_3)(C_9H_7N)_3]$	8.40 (8.62)	68.10 (68.41)	4.80 (4.77)	10.86 (10.78)
5.	$[Fe(C_{10}H_{10}N_2O_3)(C_5H_5N)_3]$	10.96 (11.22)	59.80 (60.12)	5.04 (5.01)	14.10 (14.02)
6.	$[Fe(C_{10}H_{10}N_2O_3)(C_6H_4NCH_3)_3]$ α-picoline	10.10 (10.35)	61.76 (62.10)	5.76 (5.73)	13.00 (12.93)
7.	$[Fe(C_{10}H_{10}N_2O_3)(C_6H_4NCH_3)_3]$ β-picoline	10.12 (10.35)	61.80 (62.10)	5.75 (5.73)	12.04 (12.93)
8.	$[Fe(C_{10}H_{10}N_2O_3)(C_6H_4NCH_3)_3]$ γ-picoline	10.06 (10.35)	61.70 (62.10)	5.74 (5.73)	12.06 (12.93)

CHEMICALS REQUIRED: Ethyl alcohol, acetone, Fe(II) sulphate heptahydrate, DMF, ammonia, phenyl isocyanide, quinoline, pyridine, different picolines etc. All the chemicals taken were either of E. Merck extra pure or BDH (AR) quality.

ANALYTICAL METHODS: The estimation of metals and non-metals present in the ligand and the complexes has been done by standard methods:

(i) **Iron:** Iron is volumetrically estimated by dichromate titration method.

(ii) **Carbon, Hydrogen and Nitrogen** have been estimated by semi-micro combustion method.

RESULT AND DISCUSSION: The values of electrical conductivity for Iron(II) complexes have been obtained in the range of 16-24Am respectively which indicate clearly non-electrolyte nature of all the complexes.

The values of magnetic moment of Fe(II) complexes have been obtained 4.96-5.08BM indicating the octahedral nature of all the complexes.

In case of Iron(II) complexes, a broad and unsymmetrical band obtained in the range of 10,450-10,750cm_{-1} due to ${}_5T_{2g}\rightarrow{}_5E_g$ transition indicates octahedral geometry for all the complexes.

INFRA RED SPECTRA OF THE LIGAND AND THE COMPLEXES:

By the perusal of infra-red spectra of the ligand and the complexes, we have come to the conclusion that there are appreciable changes in $\nu_{(OH)}$, $\nu_{(C=N)}$, and

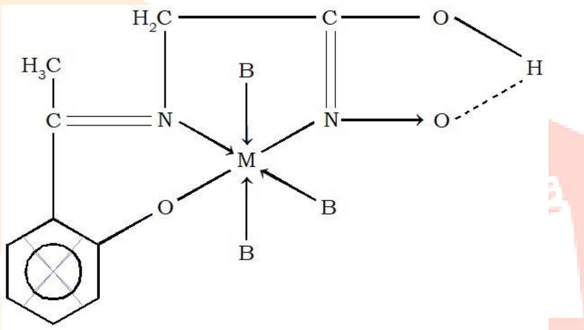
$\nu_{(N\text{-}O)}$ frequencies of the ligand molecule after the complex formation. Remaining frequencies due to the vibration of different groups present in the ligand molecule were intact after complex formation. The highest band around 3,480cm$_{-1}$ has been obtained due to combined $\nu_{(NH+OH)}$ vibrations. A strong and sharp band obtained at 3,410cm$_{-1}$ in the ligand molecule due to the vibration of phenolic –OH group, disappears in all the complexes indicating the deprotonation of phenolic – OH group. A medium and sharp band obtained at 1,520cm$_{-1}$ has been assigned due to the vibration of phenolic C-O group in the ligand molecule. This band position has been shifted to 1,550cm$_{-1}$ suggesting increase in bond order of (C-O) group in the complexes. This means that oxygen atom of phenolic –OH group

–OH group

takes part in the bond formation with the metal cations. In the ligand molecule, a sharp band around 3,180cm$_{-1}$ in the ligand molecule has been obtained due to strong hydrogen bonding. The frequency of this group has been increased to 3,230cm$_{-1}$ due to weaker hydrogen bonding. This means that there is reduction in the strength of hydrogen bonding after complex formation. A broad and strong band obtained at 1,640cm$_{-1}$ in the ligand molecule and 1,580cm$_{-1}$ in the complexes is due to $\nu_{(C=N)}$ vibration. This reduction in the frequency obtained due to $\nu_{(C=N)}$ vibration indicate the participation of aldimino nitrogen atoms as well as hydroxamino nitrogen atoms in the complex formation. The increase in the frequency by 30cm$_{-1}$ due to $\nu_{(N\text{-}O)}$ vibration after complex formation also support the participation of one hydroxamino nitrogen atom in the complex formation. There is an increase by 20cm$_{-1}$ in the frequency obtained due to $\nu_{(C=N)}$ vibration also support the participation of one aldimino nitrogen atoms in the complex formation. One aldimino nitrogen, one hydroxamino nitrogen and one phenolic oxygen atom are the boding sites of the ligand. A sharp and weak band around 2,160cm$_{-1}$ in the ligand and 2,230cm$_{-1}$ in the complexes indicate the participation of nitrogen atom of phenyl-isocyanide in the complex formation. A separate band for coordinated H_2O is not obtained due to overlap of $\nu_{(OH)}$ vibration. Two weak bands around 960cm$_{-1}$ and 610cm$_{-1}$ are obtained due to vibration of pyridine and picoline molecules. A medium and sharp band obtained 1430cm$_{-1}$ due to the ring vibration of quinoline indicates the presence of quinoline in the complexes.

The complexes containing pyridine and different picolines shows a number of bands lying in the finger print and far infra-red region.

A broad and strong band obtained in the complexes at 440cm$_{-1}$due to the vibration of (M-N) bond further confirms the coordination of nitrogen atoms in the bond formation. A weak band obtained in the complexes at 540cm$_{-1}$due to the vibration of (M-O) bond further confirms the participation of the oxygen atoms in the complex formation. Thus on the basis of elemental analysis, magnetic moment, electrical conductivity, electronic transition and IR spectral behaviour, all the complexes of Fe(II) metal with the ligand α(o-hydroxy acetophenone) imino aceto hydroxamic acid in presence of various bases have been suggested to be octahedral in nature as show below:



Where M = Fe(II) cation, L = ligand and B = bases.

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