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Studies of Spectral and Magnetic Behaviour of Some Complexes of Fe(II) Metals with 2-Hydroxy Benzo-Hydroxamic Acid

J.P. SUDHANSHU*, IRFAN AHMAD SIDDIQUE[1], SANDEEP SUDHANSHU[2] and KAMINI KUMARI[3]

*Department of Chemistry, L.C.S.College, Darbhanga, India.
L.N.Mithila University, Darbhanga, India.

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ABSTRACT

Some complexes of Fe(II) cations with 2-hydroxy benzo- hydroxamic acid has been prepared separately in aqueous and non-aqueous medium. The general molecular formula of the complexes has been found to be $[Fe(L)(B)_4]$, where L= 2-hydroxy benzo-hydroxamic acid and B= bases containing oxygen and nitrogen atoms as their donor sites. On the characterisation of the complexes by usual physico-chemical methods, all the complexes have been found to be non-electrolyte, monomeric and octahedral in geometry.

Key words: Ligand, Mono-meric, Geometry octahedral, Non-electrolyte and complex.

INTRODUCTION

In this paper, we report the synthesis of some complexes of Fe(II) cations with 2-hydroxy benzo-hydroxamic acid[1] in presence of bases like ammonia, quinoline, phenyl-isocyanide, pyridine, and α-picoline.

Chemical required

Acetate / chloride of iron in divalent state, methyl/phenyl salicylate, hydroxylamine hydrochloride, ethanol, methanol and some bases like ammonium hydroxide pyridine, quinoline, phenyl isocyanide and α- picolines.

Preparation of the Ligand[2]

A dry and cold ethanolic solution containing 0.01 mole of methyl / phenyl salicylate and 0.01 mole of hydroxylamine hydrochloride was allowed to react under reflux on a water bath for several hours at room temperature. During the course of reflux, the whole solution was shaken well more than three times. The resulting solution on cooling under ice bath produced a faint green coloured solid which was separated by filteration, washed and then dried over the pellets of KOH placed in a desiccator.

The compound was further analysed and

found to contain C=54.90%, H=4.57% and N=9.15% which correspond the expected molecular formula $C_7H_7NO_3$. The identity of the ligand was verified by infra-red spectroscopy.

Preparation of the Complexes

The ehtanolic solution of Fe(II) acetate / chloride (0.01 mole) and the ligand (0.01 mole) in molar proportion of 1:1 respectively was allowed to react in presence of bases under reflux on water bath for about two hours at room temperature. The resulting solution on crystallization produced the crystals of faint green colour. The solid was then separated by filteration washed with acetone and finally dried over KOH pellets placed in a desiccator. The complexes of Fe(II) cations with the ligand were prepared separately in presence of bases like water, ammonia, quinoline, phenyl isocyanide, pyridine and α-picoline.

Magnetic properties of the Complexes

The magnetic moments of the complexes were measured by Gouy's method using the $[Hg(NCS)_4]$ as a calibrant. The values of magnetic moment obtained in the range of 5.12 to 5.18 B.M. indicate octahedral geometry of all the Fe(II) complexes with ligand 2-hydroxy benzo-hydroxamic acid.

Electronic Spectra of the complexes

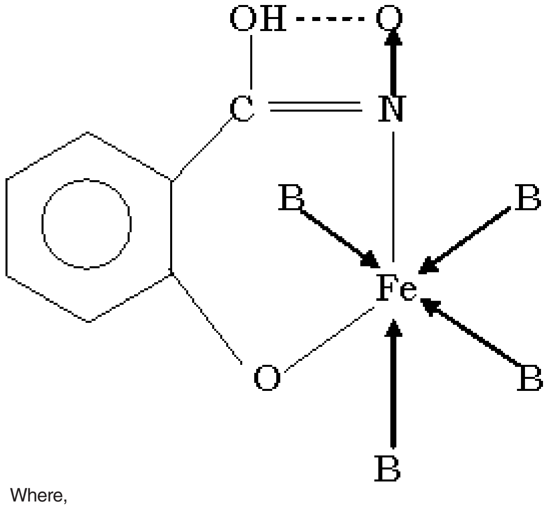
A broad and unsymmetrical band[3] obtained around 10600 to 10900 cm^{-1} due to $^5T_{2g} \rightarrow {}^5E_g$ transitions indicate octahedral geometry of all the Fe(II) complexes. The band obtained is broad and unsymmerical due to Jahn-Teller Distortion.

I. R. Spectra of the ligand and the complexes

In almost all complexes of Fe(II) cation, the frequencies of (OH), aldimine $^{\upsilon}$(C=N) and $^{\upsilon}$(N-O) of the ligand molecule are changed appreciably after complex formation. The strong and sharp band obtained at 3280 cm^{-1} due to vibration of (-OH) and (N-H) groups of the ligand molecule. The band obtained at 3280 cm^{-1} in the ligand molecule disappears in all the complexes and a new broad band[4-8] appears in the complexes at about 3430 cm^{-1} confirming the presence of at least on free –OH group even in the complexes with hydrogen bonding. The medium but sharp band located at 1240 cm^{-1} in the ligand has been obtained due to the deformation vibration of phenolic (OH), disappears in all the complexes supporting the deprotonation of phenolic (OH) and coordination of phenolic oxygen atom to the metal atom. A medium and sharp band obtained in the range of 1450-1470 cm^{-1} due to $\upsilon_{(C-O)}$ vibration of phenolic (C-O) group shifts to higher frequency in the range of 1520-1535 cm^{-1} in the complexes suggesting increase of bond order of (C-O) group in complexes as noticed by sin *et. al.,*[9]. The disappearance of the strong band at 3240 cm^{-1} further suggests the deprotonation of the (OH) group i.e. (N-OH) proton located at hydroxamic acid moiety. The deprotonation of (N-OH) proton is further confirmed by the reduction in the frequency of (N-O) group of

Table 1: Colour, magnetic moment and elemental analysis of the complexes

Compound	Colour	B.M	Metal	Carbon	Hydrogen	Nitrogen
1. $[Fe(C_7H_5NO_3)(H_2O)_4]$	Light green	5.16	19.88 (20.07)	29.90 (30.10)	4.66 (4.65)	5.06 (5.01)
2. $[Fe(C_7H_5NO_3)(NH_3)_4]$	Faint green	5.12	20.12 (20.36)	30.32 (30.54)	6.20 (6.18)	25.25 (25.45)
3. $[Fe(C_7H_5NO_3)(C_6H_5NC)_4]$ (Ph-isocyanide)	Light green	5.18	7.62 (7.74)	71.12 (71.36)	4.58 (4.56)	9.70 (9.68)
4. $[Fe(C_7H_5NO_3)(C_9H_7N)_4]$ Quinoline	Faint green	5.14	8.92 (9.04)	67.66 (67.85)	4.05 (4.03)	11.36 (11.30)
5. $[Fe(C_7H_5NO_3)(C_5H_5N)_4]$ Pyridine	Light green	5.16	10.56 (10.70)	61.76 (61.95)	4.80 (4.78)	13.44 (13.38)
6. $[Fe(C_7H_5NO_3)(C_6H_4NCH_3)_4]$ α-picoline	Faint green	5.12	9.56 (9.67)	63.92 (64.24)	5.70 (5.69)	12.12 (12.08)



Where,

B= Water, ammonia, quinoline, phenyl isocyanide and pyridine

Scheme 1:

the ligand and the complexes by 20-30 cm^{-1} due to bonding mode of vibration of ^{u}(OH) group disappears in the complexes indicating the deprotonation of (OH) group.

The deprotonation of (N-OH) and phenolic (–OH) protons and coordination of hydroxamino nitrogen (C=N) atom and phenolic oxygen atom are further confirmed due to the appearance of two new bands around 450 cm^{-1} and 560 cm^{-1} due to $^{\upsilon}$(M-N)[10] and $^{\upsilon}$(M-O)[11] vibrations respectively.

In case of aquo-complexes, separate band for coordinated H_2O is not obtained as it is overlapped by $^{\upsilon}$(OH) and $^{\upsilon}$(NH) vibrations. The frequencies obtained in the range of 720-820 cm^{-1} in aquo-complexes of Fe(II) ions may be attributed to rocking mode of coordinating water.

The amine complex displays one broad and strong band around 3300 cm^{-1} assignable to (NH) stretching vibration of coordinated ammonia molecule. In case of phenyl isocyanide complexes there is a increase in the $^{\upsilon}$(C ° N) vibration of the ligand (2180 cm^{-1}) and the complexes (2230 cm^{-1}) indicating the involvement of (NC) group in coordination. In quinoline complexes, a medium and broad band around 1440 cm^{-1} obtained due to ring vibration (ring breathing) indicates the involvement of N-atom of the quinoline in the complex formation.

The pyridine and picoline complexes display bands in finger print and for infra red region. The pyridine ring breathing mode of vibration located at 930-1060 cm^{-1} in the complexes have been regarded as the characteristic vibrations of the coordinated pyridine and picolines molecules. The I.R band at 560-570 cm^{-1} in the complexes is also characteristic of presence of pyridine molecule in the complexes.

Thus on the basis of elemental analysis, values of electrical conductance and magnetic moments, electronic and I.R spectra of the ligand and the complexes, the probable structure for $[Fe(L)B_4]$ complexes has been suggested to be octahedral monomeric and non-electrolyte in nature

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