

SOME ASPECTS OF STUDY OF STRUCTURE, IRON(II) COMPLEXES WITH ACETAZOLAMIDE, A SCHIFF BASE.

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Abstract:

The paper reflects FE(II) complexes of Schiff bases were synthesized separately in the presence of various bases containing "O" and "N" atom as their donor sites. On characterization by usual physicochemical methods each of the complexes generated were non-electrolytic, mono-magic and paramagnetic nature. A six co-ordinated octahedral geometry of every complexes were proposed. The main aim of this work being discovery of safe and effective therapeutic agents for the treatment of bacterial infections.

Keywords:

Schiff base, Acetazolamide, Characterization, Octahedral, Paramagnetic, Non-electrolyte, therapeutic.

Introduction:

In this paper, we report the preparation of some complexes of divalent iron with the ligand schiff base acetazolamide in presence of bases like water, ammonia, quinoline, phenyl iso-cyanide, pyridine and picolines. This paper presents the synthesis spectral and biological studies of some metal chalets of bidentate Schiff base derived from acetazolamide. The ligand and the complex of Fe(II) have been synthesized with the Schiff base ligand, acetazolamide. Schiff base metal chalets have played a central role in the development of co-ordination chemistry. In the present paper, it was reported that preparation, spectroscopic, and biological studies of Fe(II) complexes with acetazolamide (5- acetamide-1-3-4-thiadiazole-2- sulphonamide) a diuretic drug. The bacteriological activities of ligand and Fe(II) complexes have also been studied.

Material and Methods:

Materials- The chemicals and solvents used in this research work were of analytical grade sourced from sigma chemical company. Synthesis of Schiff base ligand was carried out in pure solvent using standard literature methods. The chemical Fe(II) acetate/chloride. Acetazolamide, 2- hydroxy benzaldehyde ethanol, methanol and some bases like ammonium hydroxide pyridine, quinoline, phenyl iso-cyanide and picolines.

Experimental:

Preparation of the ligand:

The schiff base ligand acetazolamide has been prepared by the condensation of 5-acetamide-1,3,4 thiadiazole-2 sulphonamide and o-hydroxy benzaldehyde in presence of dilute acid.

Procedure :

About 22.2 grams (0.01 mole) of 5-acetamide -1,3,4- thiadiazole-2 sulphonamide was completely dissolved in ethyl alcohol and 12.2 grams (0.01 mole) 2- hydroxy benzaldehyde was also completely dissolved in ethyl alcohol. Both the solutions were gradually mixed together and shaken well. About 60ml. Of dilute hydrochloric acid was added in the resulting solution to make it acidic. The resulting solution was refluxed on water bath for about three hours.

The solution on cooling, under tap-water, produced yellowish solid which was separated by filtration. The solid was recrystallized with acetone and washed with methanol. The melting point of the compound was recorded to be 196 °C. The compound was further analysed and found to contain, C=40.10%, H=70.20% and S=19.56% which corresponds to the molecular formulae $C_{11}H_{10}N_4O_4S_2$. The chemical reaction taking place during the course of condensation reaction is given below:

Preparation of Fe(II) complexes:

A amount of 0.65 gram (0.002 mole) of the schiff base ligand 5-acetamide-2-sulphoimide salicylaldehydically-1,3,4-thiadiazole was completely dissolved in a minimum volume of ethanol and 0.23 gram (0.001 mole) of Fe chloride hexahydrate was also completely dissolved in aqueous ethanolic solution. Both the solutions were mixed together. The ratio of the metal and the ligand was always kept in 1:2 respectively. The resulting solution was heated under reflux over a hot water bath for two hours. The complexes of divalent iron with the ligand were prepared separately in presence of ammonia, quinoline, phenyl iso-cyanide pyridine and picolines. When a crystalline solid of different colour was obtained which was separated by filtration. The solid was washed with small quantity of alcohol followed by ether and dried in oven. The compound was further analysed and found to contain.

Elemental analysis of Fe(II) complexes:

Compound	Colour	M.M B.M	Metal	C	H	N	S
1. $[Fe(C_{11}H_9N_4O_4S_4)_2(H_2O)_2]$	Light Green	5.16	7.40 (7.54)	35.20 (35.57)	2.94 (2.96)	15.10 (15.09)	17.00 (17.25)
2. $[Fe(C_{11}H_9N_4O_4S_4)_2(NH_3)_2]$	Faint Green	5.24	7.80 (7.92)	35.40 (35.53)	3.20 (3.23)	18.90 (18.84)	17.20 (17.22)
3. $[Fe(C_{11}H_9N_4O_4S_4)_2(C_9H_7N)_2]$	Brownish Green	5.14	5.68 (5.80)	49.60 (49.79)	3.32 (3.31)	14.56 (14.52)	17.20 (17.27)
4. $[Fe(C_{11}H_9N_4O_4S_4)_2(C_9H_7N)_2]$	Dirty Green	5.18	6.10 (6.14)	47.24 (47.36)	3.10 (3.07)	15.40 (15.35)	14.10 (14.03)
5. $[Fe(C_{11}H_9N_4O_4S_4)_2(C_5H_5N)_2]$	Blackish Green	5.12	6.32 (6.48)	44.30 (44.44)	3.26 (3.24)	16.24 (16.20)	14.84 (14.81)
6. $[Fe(C_{11}H_9N_4O_4S_4)_2(C_6H_4NCH_3)_2]$	Dirty Green	5.32	6.20 (6.27)	45.70 (45.73)	3.60 (3.58)	15.76 (15.79)	14.30 (14.34)

Which corresponds to the molecular formula $[Fe(L)_2(B)_2]$
Where, L= Ligand & B= Base

Magnetic properties of the complexes:

The magnetic moments of the complexes were measured by Gouy's method using the $Hg(NCS)_4 \cdot 2H_2O$ as a calibrant. The values of magnetic moment obtained in the range of 5.14 to 5.32 B.M indicate octahedral geometry of all the Fe(II) complexes.

Electronic spectra of the complexes:

Broad and unsymmetrical band⁵ obtained around 12850 to 13120 cm^{-1} due to ${}^5T_{2g} \rightarrow {}^5E_g$ transitions indicate octahedral geometry of the Fe(II) complexes.

I.R. Spectra of the ligand and the complexes:

The frequencies due to the vibrations of phenolic -OH group⁶ and azomethine group⁷ ($>C=N-$) have been appreciable Lee changed after the complex formation.

In almost all the complexes, a strong and sharp band obtained in the Legend molecule at 3420cm^{-1} due to the vibration of phenolic -OH group this appears in all the complexes indicates the donation of phenolic -OH group in the complex formation in almost all the complexes are strong and served and obtained in the Legend molecule at due to the vibration of phenolic -OH group disappears in all the complexes indicates deprotonation of phenolic -OH group in the complex is the definition of phenolic group has been suggested due to the presence of a medium and sharp bend obtained in the range of $1510-1540\text{cm}^{-1}$ the phenolic(C-O) has been shifted to 1530 to 1540cm^{-1} in the complexes. This increase in the bond length of phenolic(C-O) indicates the death deprotonation of phenolic -OH group.

A broad and sharp band obtained in the Legend molecule at 1640cm^{-1} due to (C-N) direction has been reduced to 1590cm^{-1} in the complexes. This reduction in the frequency of (C-N) group suggests the party suppression of aldimino (azomethine) nitrogen in the bond formation with metal ion.

By the comparison I.R spectral behavior of the ligand and the complexes, it is concluded that the ligand behaves as mono-anionic, bi-denate molecule. Thus one aldimino nitrogen and one phenolic oxygen atoms are the bonding sites of the legand molecule.

In our present investigation, complexes of Fe(II) metal have been prepared with the Schiff base acetazolamide in presence of bases containing oxygen and nitrogen donors atoms like water, ammonia, quinoline, phenyl iso-cyanide, pyridine and different picolines.

In case of aquo complexes, seprate band for co-ordinated h_2o is not obtained as it is overlapped by (OH) and (NH) vibrations. The frequencies obtained in the range of $720-790\text{cm}^{-1}$ in aquo-complexes of Fe(II) cation may be attributed to be rocking mode of Co-ordinating water.

The amino complexes $[\text{Fe}(\text{L})_2(\text{NH}_3)_2]$ displays on broad and strong band around 3460cm^{-1} assignable to NH stretching vibration of Co-ordinated ammonia molecule. In case of phenyl iso-cyanide complexes there is a increase in the $\nu_{(C=N)}$ vibration of the ligand (2225cm^{-1}) indicating the involvement of iso-cyanide (N-C) group in Co-ordination. In quinoline complexes a medium and broad band around 1430cm^{-1} obtained due to ring vibration (ring breathing) indicates the involvement of N-atom of the quinoline in the complex formation.

A sharp and medium band, obtained in the range of $390-410\text{cm}^{-1}$ in the complexes due to (M-N) further confirm the co-ordination of element nitrogen atoms in the bond formation with the metal cations. A and medium band obtained in the range of $440-455\text{cm}^{-1}$ in the complexes due to (M-O) vaberation further confirm the coordination of phenolic oxygen atom in the bond formation with the metal cation. The frequency obtained due to the vibrations of (M-O) bond is greater than that of (M-N) bond because (M-O) bond is more ionic than (M-N) bond.

Thus on the basis of elemental analysis, values of electrical conductance and magnetic moment and electronic spectra and interpretation of the the I.R spectra of the ligand and the complexes, the probable structure of $[\text{Fe}(\text{L})_2(\text{B})_2]$ complexes are suggested to be Mono-meric electrolyte and octahedral in nature.

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