Synthesis, UV Visible Spectroscopic Characterization and Antimicrobial Activity of Ag (II) Metal complex with Pyrazole ligand 4-(4, 5-dihydro-1H-pyrazol-5-yl) benzene-1,3-diol.

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ABSTRACT :-

In the present work the entitled Pyrazole ligand 4-(4, 5-dihydro-1H-pyrazol-5-yl)benzene-1,3-diol is prepared by (E)-1-(2,4-dihydroxyphenyl)-3-(dimethylamino)prop-2-en-1-one and phenyl hydrazine while its biologically active Silver metal complexes (M = Ag) are prepared by refluxing in ethanol solution. The metal complex is characterised by UV Visible spectra, which suggesting M; L ration 1:1 for Ag(II) metal ion chelate.

KEY WORDS: - Silver, Pyrazole, Metal Complex, Spectral Study, Antimicrobial activity.

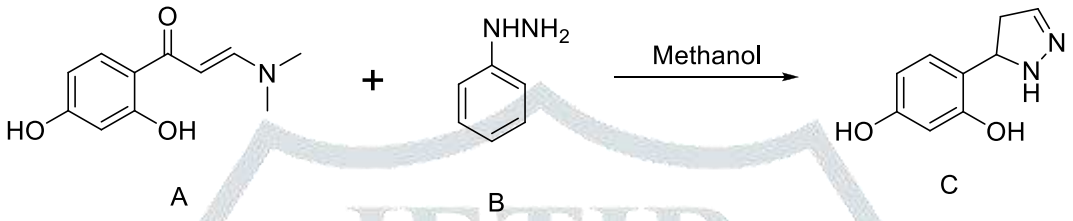
1.0] INTRODUCTION:-

The coordination chemistry of pyrazole derived ligands has received much attention, primarily due to their biological implications[14-17]. The Transition metals like Nickel, Copper, Zinc, Iron etc playing an important role in the various biological process occurs in the living organism like plants, animals, human etc. Haemoglobin carries oxygen to vital areas of body by binding it to the iron atom contained within it. Metal ions such as zinc provide the structural framework for the zinc fingers that regulates the functions of the genes in the nuclei of cells minerals containing calcium are the bases of bones, the framework of human body[12]. Metals such as zinc, copper iron and manganese are incorporated into catalytic proteins which facilitate a number of chemical reactions needed for life1.There has been enough interest organic ligand if at least one atom other than carbon forms a part of the ring system that it is designated as a heterocyclic compound[1]. Parasol, which are five members two nitrogen containing heterocycle. Nitrogen, oxygen and sulphur are the most common hetero atom but heterocyclic rings containing O, N, S, donor atoms because of the Varity of ways in which they are bonded to metal ion. Benzimidazole, pyrazole, isoquinoline, derivatives are the different types of heterocyclic used as anthelmintics. Albendazole is the most active benzamidazole antihelminitic drug[10]. Coordination compounds have been extensively used in industrial, biological, analytical, biochemical, clinical, antimicrobial[2], analgesic[3], antibacteral[4], antihypersensive[5] anticancer, antifungal and antitumor activity. The ligand plays an important role in complex formation, ligand act as electron donor to a single cation, they also acts as bridging groups to form stable metal chelates. The metal Chelates depends on the affinity of metal ion reacts with towards chelating and its coordination[5]. The rapidly developing field of bioinorganic chemistry is centered on the study of coordination compounds present in living systems.

2.0] EXPERIMENTAL :-

2.1. Synthesis of Ligand

In the synthesis of 4-(4,5-dihydro-1H-pyrazol-5-yl)benzene-1,3-diol (C), the equimolar mixture of (E)-1-(2,4-dihydroxyphenyl)-3-(dimethylamino)prop-2-en-1-one (A) and phenyl hydrazine (B) are dissolved in a 30 ml Methanol solution. The reaction mixture was stirred under reflux condition for 10 to 12 hour. TLC monitoring of the reaction showed complete transformation. After completion of reaction, mixture was poured into crushed ice. The resulting product having black colur were filtered off, washed with cold water and dried in vacuum.



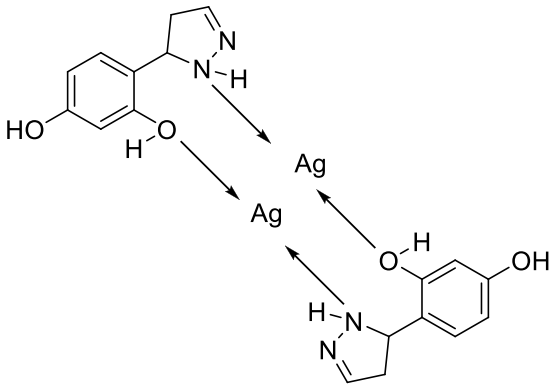
2.2] Synthesis of metal complex (Ag_2L_2)

The Metal complex (Ag_2L_2) i.e. bis(2-(4,5-dihydro-1H-pyrazol-5-yl)-5-hydroxyphenoxy)silver (D) is prepared by following method. A weighed quantity of ligand (C) (0.01 mol) and metal chelate i.e. AgNO3 (0.01mol) were separately dissolved in 100 ml of ethanol solution. Clear solution of Silver Nitrate and ligand (C) solution were mixed in stoichiometric ration 1:1, the solution were reflux for two hours with constant steering. The pH of solution was adjusted 6.0 to 6.5 by alcoholic ammonia. After cooling the resultant precipitate was digested for one hour. The precipitate was filtered, washed with hot ethanol (40-60 ^{0}C) and dried in vacuum desiccators over anhydrous granular calcium chloride and stored in a air tight glass bottle.

3.0] RESULT & ANALYSIS:

Table 1: Analytical Data of 4-(4, 5-dihydro-1H-pyrazol-5-yl)benzene-1,3-diol and Ag(II) metal Complexs bis(2-(4,5-dihydro-1H-pyrazol-5-yl)-5-hydroxyphenoxy)silver.

Ligand / Complex	Colour	Yield (%)	Formula	Mol. Wt.	%Analysis Found(Calculated)			
					C	H	N	M
4-(4,5-dihydro-1H-pyrazol -5-yl)benzene-1,3-diol	Black	68	$C_9H_{10}N_2O_2$	178	66.60 (66.65)	6.15 (6.21)	17.22 (17.27)	
bis(2-(4,5-dihydro-1H-pyrazol-5-yl)-5-hydroxyphenoxy) silver	Black brown	47	$C_{18}H_{20}Ag_2N_4O_4$	572	46.26 (46.77)	3.86 (3.93)	12.14 (12.12)	23.22 (23.34)



Structure (D):- bis(2-(4,5-dihydro-1H-pyrazol-5-yl)-5-hydroxyphenoxy)silver

3.1 U.V. Visible Spectral studies:-

Electronic spectral data of the silver complex have been recorded in the DMSO solution in the range 200nm to 700nm. For the silver complex, the absorption bands in the visible region are 343nm to 416nm, the nature of Ag(II) affects the position of absorption bands.

Table 2 : U.V. Visible Spectral data of transiton metal Chelate

Metal Complex	Mol. Wt. of Complex	Absorbance	Transition
(Ag_2L_2)	$C_{18}H_{20}Ag_2N_4O_4$	343nm 416nm 402nm	$\pi \longrightarrow \pi^*$ $\pi \longrightarrow \pi^*$ LMCT

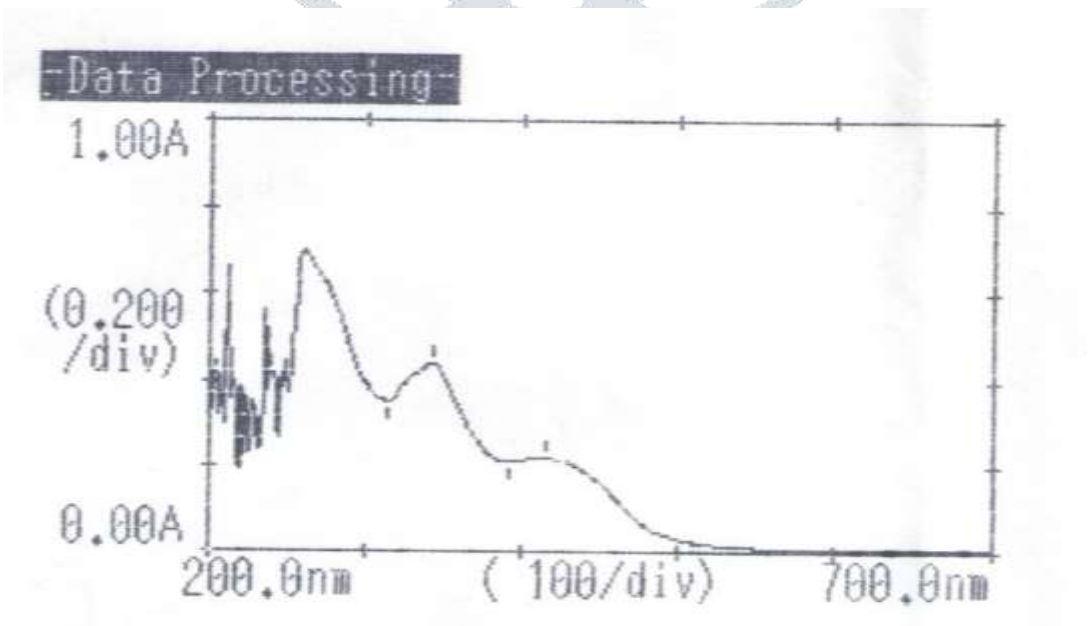


Figure 1: Electronic specra of (Ag_2L_2)

3.2. Antimicrobial Activity

3.2.1.Preparation of culture of media

The antimicrobial activity of test compounds was tested by disc diffusion method. The test compound (1 mg/mL) was dissolved in dimethyl sulphoxide and loaded on a sterile filter paper disc of 6 mm diameter. The petriplates containing nutrient agar medium (HiMedia) were spread with 100µL of actively growing both culture of the test bacteria using sterile cotton swab and allowed to dry for 10 min. For fungal species, 100 µL of active culture was spreaded on CzapekDox agar (HiMedia).Then the impregnated discs were placed on the surface of inoculated agar medium.

Discs loaded with dimethyl sulphoxide (Sd Fine Chemicals) were served as control. Streptomycin and fluconazole (HiMedia) discs were used as positive control for bacterial and fungal species respectively18. The nutrient agar plates were incubated at 37 °C for 24 h and CzapekDox agar plates at 30 °C for 7 days. The development of inhibition zone around the disc was recorded in terms of mm and compared with controls.

3.2.2. Observation

Microscopic organism, Bacteria and fungus are responsible for various diseases. They are also responsible for the some biochemical reactions. The previous studies indicate that the heterocyclic ligands with transition metal ions increases or retards the antibacterial and antifungal activity. Complex formation reduces the polarity of the metal ion due to the partial sharing of its positive charge with the donor groups and delocalization of π electrons. This process increases the lipophilic nature of the central metal atom, which is responsible for increasing the hydrophobic character and liposolubility of the molecule in crossing cell membrane of the microorganism, and hence enhances antibacterial activity[13].

The growth of ***Aspergillus niger*** and ***Escherichiacoli*** was effectively inhibited by (Ag_2L_2). The antimicrobial activity of Metal Complex is summarized in the following table.

Sr. No.	Compound Code	Zone of Inhibition in mm	
		Fungal species	Bacterial species
		Aspergillus niger	***Escherichia coli***
02	$(Ag_2L_2)_2$	21	12

Figure 2: Antimicrobial Activity of Ag_2L_2

4.0 Conclusions:-

The Silver(II) complexes were synthesized and characterized by UV-Visible Spectroscopy and biological studies were carried out. The complexes are soluble in DMF and DMSO. The observation shows that silver complexes are positively antimicrobial in action. They seem to be anti-bacterial and anti-fungicidal. However their toxic effects are to be tested. They can also be used against plant pathogens as plant protection agents.

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