



Synthesis and spectroscopic characterization of Cuppe (II) and Silver (II) Metal chelate with 2-(4.5-dihydro-1HPyrazol-5yl) 5-hydroxy Phenol

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Abstracts:-

Now in present work the pyrazol ligand 2-(4.5-dihydro-1HPyrazol-5yl) 5-hydroxy Phenol were prepared by (E)-3-(2-hydroxyphenyl)-N-methyl-N-methylene-3-oxoprop-1-amonium with Phenyl hydrazine were added and the reaction mixture stirred for 10-12 hours at room temperature. TLC Monitoring of the reaction showed complete transformation. The reaction mixture was then cooled with crushed ice. The resulting product having color black were filtered off, washed with water and dried in vacuum. The metal chelate characterized by elemental analysis, UV visible spectra which suggesting ML ratio 1; 1 for silver and copper metal chelate

Keywords:-2-(4, 5 –Dihydro-1H-Pyrazol-5-yl) 5-hydroxy Phenol spectroscopic study: some transition metal ion chelate

Introduction:

There has been enough interest organic ligand containing S, N, and O donor atoms because of the verity of ways in which they are bonded to metal ions. Coordination compounds have been extensively used in industrial, biological, biochemical, analytical, clinical, antimicrobial anticancer activity. The legend plays an important role in complexes formation. Ligand acts as electron pair donors on a single cation. The ligand acts as balding groups to form stable metal complexes.

Coordination chemistry of pyrazol derived ligand has been received much attention primarily due to their biological implication¹⁻³ The transition metal like Zinc, Nickel, Copper, Iron etc playing an important role in the various biological processes occurs in the living organism like human, animals, plants, etc. Hemoglobin carries oxygen to vital areas of body by binding it to iron atom contained within it .Metal ions such as zinc provide the standered 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 that framework of human,⁴ Metals such iron zinc, copper, and manganese are in comported in to catalytic protons which facilitate a number of chemical reactions metal for life one atom other than carbon forms a part of the ring system that it is designated as a heterocyclic compounds⁵ Pyrazol which are five member two nitrogen containing heterocyclic nitrogen, oxygen and sulpher are the most common hetero atom but heterocyclic ring containing O.N.S donor atoms. Because of the verity of way in which they are bonded to meal ions pyrazol derivative which are five members two are nitrogen containing heterocyclic. Pyrazol are aromatic molecules due to their planner conjugated ring structures with six delocalized π -electrons. There fore many important properties of these molecules were analyzed by comparing with properties of

benzene derivatives⁵The metal chelate depends on the affinity of metal ion reacts with towers chelating and its coordination. The rapidly developed field of bioinorganic chemistry is converted on the study of coordination compounds present in living system.

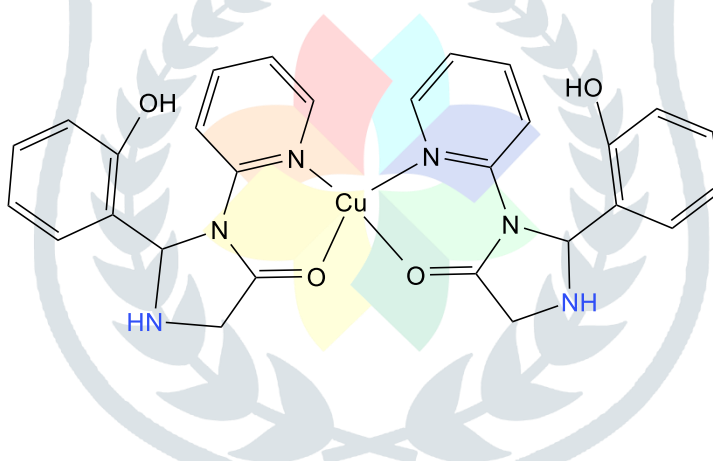
Experimental Preparation of metal complexes

Synthesis of Legend- Bis(2-(4,5,dihydro-1H pyrazol-5yl)4-hydroxy)phenol.

A molar solution of(E)-3-(2-hydroxyphenyl)-N-methyl-N-methylene-3-oxoprop-1-en-1 aminium and phenyl hydrazine (B) are dissolved in a ethanol solution. The reaction mixture was stirred and reflex condensation for 10 to 12 hours. TLC monitoring of the reaction showed complete transformation. After completion of reaction, mixture was then poured in to crushed ice. The resulting product having color black were filtered off, washed with cold water and dried in vacuum.

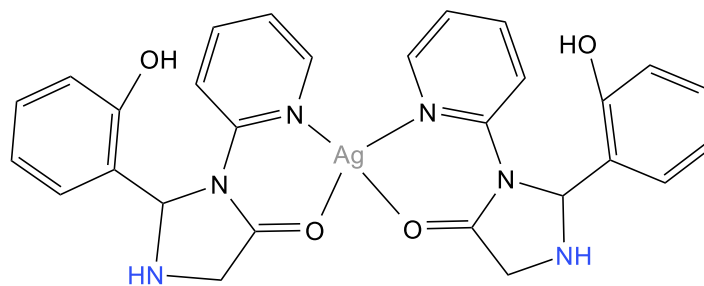
Preparation of Bis (2-(4, 5, dihydro-1Hpyrazole-5yl) 4-hydroxy) phenoxy Cu (II) complexes

A weighted quantity of copper nitrate (0.01mol) of legend Bis (2-(4, 5, dihydro-1H pyrazol-5yl)4-hydroxy) phenol(0.01mol) were separately dissolved in 100ml ethanol solution⁶⁻⁷.Clear solution of metal salt and legend were mixed in stiochemetric ratio1:2 (by volume) solution were mixed thoroughly with constant stirring and adjusted to a PH of 6.8 by adding alcoholic ammonia solution. The resultant mixture was heated under reflux for about two to three house and allowed to cool. Gray black colored precipitated obtained. It was separated after digestion for half an hour, washed with ethanol for three times and then dried in desiccators (Yield 59%)



Preparation of Bis (2-(4, 5, dihydro-1Hpyrazole-5yl) 4-hydroxy) phenoxy Ag (II) complexes

Molar solution of legend(0.01mol) and silver nitrate (0.01mol) were prepared by dissolving in 100ml ethyl alcohol, those solution were mixed in to each other 1:1 ratio and adjust PH of 6.5 by adding alcoholic ammonia solution .present mixture was heated under reflux for about two to three hours, at the end content were allowed to cool. Digested for half hour and filtered, black brown crystals was seperated^{8,9} were dried in desiccators and stored in fresh bottle (Yield 47%)



UV-visible spectral studies: - Analytical data of Cu (II), Ag (II) metal complexness with Bis-(2 (4, 5-dihydro-1H-pyrazol-5yl) 4-hydroxyPhenol

Sr. no	Complex	Color	Yield (%)	Formula	Mol. Wt	Elemental analysis(Calc.)(%)			
						C	H	N	M
1	(2-(4,5,dihydro-1Hpyrazol-5yl)-4-hydroxy)phenol (legend)	Dirty black	65	C ₉ H ₁₀ N ₂ O ₂	162	(66.60)	6.15	17.22	-----
2	Bis-(2-(4,5,dihydro-1Hpyrazol-5yl)4-hydroxy)-4-hydroxy Cu (II) Complex	Gray black	59	Cu C ₁₈ H ₁₈ N ₄ O ₄	417	51.88 (51.73)	4.25 (4.34)	15.65 (15.21)	13.28 (13.41)
3	Bis-(2-(4,5,dihydro-1Hpyrazol-5yl)4-hydroxy)-4-hydroxy Ag(II) complex	Black brown	47	AgC ₁₈ H ₁₈ N ₄ O ₄	462	46.26 (46.77)	3.86 (3.93)	23.22 (23.34)	12.14 (12.12)

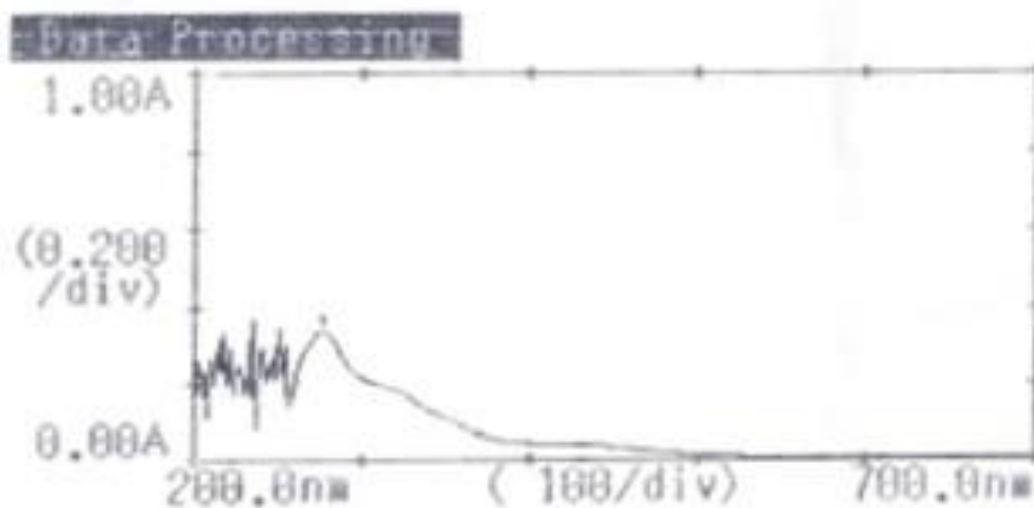


Fig.NO1: UV Spectrum of [2-(4, 5dihydro-1Hpyrazol-5yl)-4-hydroxy phenol

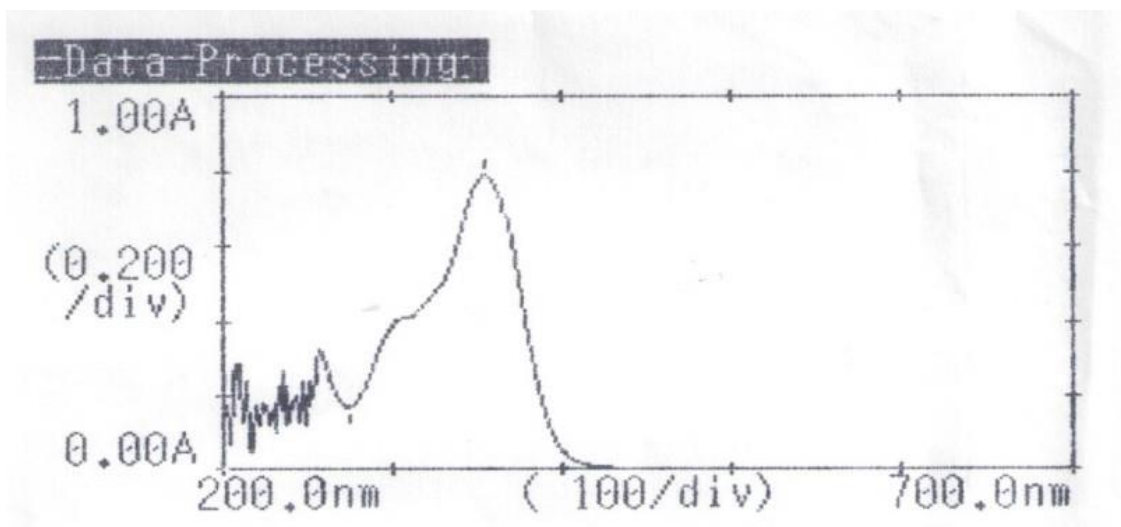


Fig.NO.2 UV Spectrum of Bis-2-(4,5 dihydro-1Hpyrazol-5yl)-4-hydroxy phenoxy Cu(II) Complex

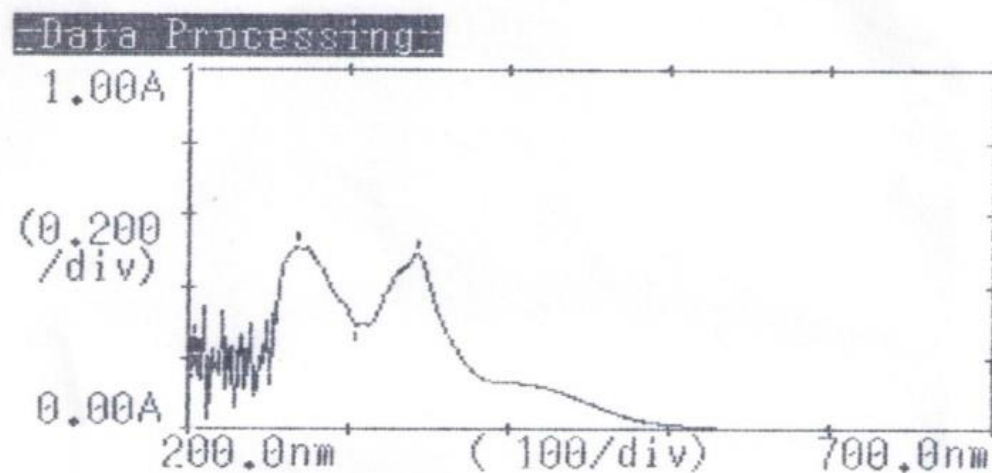


Fig.NO1: UV Spectrum of [2-(4, 5dihydro-1Hpyrazol-5yl)-4-hydroxy) phenoxy Ag (II) Complex

Result and discussion :-The ligand [2-4,5-dihydro-1HPyrazol-5yl)-4-hydroxy)Phenol is used to prepare metal chelate of Cu (II), Ag (II) These Chelate are colored and only soluble in ethanol. Decomposition points of these metal ions chelate are very high suggesting good thermal stability

U.V. Visible Spectral Studies:-Electronic absorption data of compound in DMSO are given in the table. In the spectrum of legend, the band at 260nm is attributed to the solution in the $\pi-\pi^*$ transition of the spectra of legend and 402nm assigned to the $n-\pi^*$ transition N=N group in free legend¹⁰. The spectra of metal complex the less intense band 265nm is observed and that may be due to the charge transfer. LMCT (legend to metal

bond) transition. The electronic absorption spectrum of Bis-2-(4, 5dihydro-1Hpyrazol-5yl)-4-hydroxy) phenoxy Cu (II) Complex show 421nm may be assigned to the $n-\pi^*$ transition.

The absorption shift and intensity change in the spectra of metal complex change because of conjugation and delocalization of the whole electronic system and result in the energy change of $\pi-\pi^*$ and $n-\pi^*$ of transition of conjugation chromophore^{11,12}. In case of copper complex, the absorption bands in the visible region are 410nm-623nm similarly for silver complexes the absorption band 403-579nm the nature of Cu (II) Ag (II) affects the position of absorption band.

The observation spectra of transition metal complex can be very well interpreted using ligand field theory. Most of the Cu (II) complexes with square planer geometry have weakly coordinately groups in axial position¹³ and many of the complex with less bulky N-alkyl group or N-substituent are planar¹⁴ A wide range of pent coordinated Cu (II) complexes of both trigonal bipyramidal and square pyramidal types have been reported

Reference:-

- 1) Bishop, M. N., Merinbaum K.D. and Emeribe. U., *Euro. Jour. of Clin. Endocrinol. Metab.* 94, 2975, **2009**.
- 2) Dowle, M.D. and Coates, I.H., DE 332052, **1983**.
- 3) Chen, C. Y., Liberman, R. and Reider. J. P. *Bioog. Tetrahedron Lett.* 6981, **1994**.
- 4) Heinzelman, R.V. and Szmuszkovicz, J., *Prog. Drug Res.* 6, 75, **1963**.
- 5) Molina, J., Romanha, A., M. F. and Urbina J., *Jour. Ame Society for Micr.* 44(1), 150, **2000**.
- 6) Brandht, W. W., Dwyer F. P. and Gyarfes E.C., *Chem. Rev.*, 54, 959. **1954**.
- 7) Morsey, A.M. Abou, Y., Raja D. and Varoslav L., *Inorg. chem.*46, 5893, **2007**.
- 8) Bethe, H., *Ann. Physik.* 5, 3133, **1929**.
- 9) Van Vleck, J. H. Z., *Physik.* 73, 565, **1931**.
- 10) Kumar, K. N. and Ramaesh R., *Polyhydron*, 24(14), 1985, **2005**.
- 11) Holm, R. H., *Prog. Inorg. Chem. Jour.* 151, **1966**
- 12) Sacconi, L. and Ciampolini, M. *Jour. Chem. Soc.* 2, 761, **1964**.
- 13) Sacconi, L. and Ciampolini, M. *Jour. Chem. Soc.* 4, 407, **1965**.
- 14) Huheey, J. E., *Inorganic Chemistry*, Harper and Row, New York, 447, **1983**.