JETIR.ORG ISSN: 2349-5162 | ESTD Year : 2014 | Monthly Issue
JOURNAL OF EMERGING TECHNOLOGIES AND INNOVATIVE RESEARCH (JETIR)
An International Scholarly Open Access, Peer-reviewed, Refereed Journal

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

Dr.Garud. S.B. Dr.Ballal.S.D
Department of Chemistry, Vasantrao Naik College Nanded, Maharashtra.
Department of Chemistry Sharda Mahavidyalaya (Arts & Science) Parbhani

Abstracts:-

Naw in present work the pyrazol ligand 2-(4.5-dihydro-1HPyrazol-5yl) 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.

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

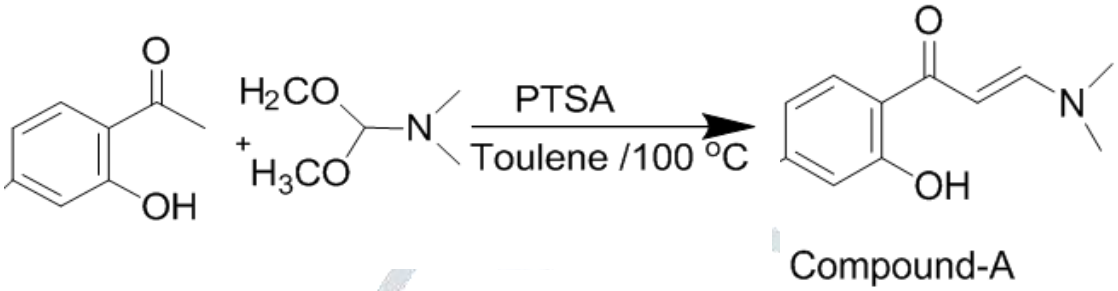
Introduction: There has been enough interest organic ligand containing O, N, S, and Donor atoms because of the verity of ways in which they are bounded to metal ions Coordination compounds have been extensively used in industrial, biological, analytical, biochemical, clinical, antimicrobial, and anticancer activity.

The ligand plays an important role in complex formation. The ligand act as electron pair donor in a single cation. The ligand acts as bridging groups to form stable metal complex. The coordination chemistry of derived ligands has received much attention primarily due to their biological implication [1-4] .The transition metal like Nickel, Copper, Zinc, Iron, etc playing an important role in the various biological processes occurs in the living organism like plants, animals, humans etc. Hemoglobin carries oxygen to vital areas of body by binding it to the iron atom contained within it. Metal ions such as Zinc provide the structure 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, [5(12)] Metals such as Zinc, copper iron, and manganese are incorporated into catalytic protins which facilitate a number of chemical reactions needed for life. Ligand if at least one atom other then carbon forms a part of the ring system that it is designated as a heterolytic compounds, [6(!)] Pyrazol, which are members, two nitrogen containing heterocyclic nitrogen, oxygen and sulpher are the most common hetero atom but heterolytic ring containing O, N, S donor atoms but because of the varity of ways in which they are bonded to metal ion. Pyrazol derivative which are five members two nitrogen containing heterocyclic Pyrazol are aromatic molecules due to their planer conjugated ring structure with six delocalized II-electrons. Therefore many important properties of these molecules were analyzed by comparing with properties of benzene derivative[7(53).The] metal chelate depends on the affinity of metal ion reacts with towers chelating and its coordination[6,] The rapidly developed field of bioinorganic chemistry countered on the study of coordination compounds present in living system

Experimental:

Synthesis of ligands Preparation of starting materials for the pyrazole ligands

In this work acetophenone and diacetyl form amide were dissolved in required amount of toluene with paratoluene sulphonic acid as a catalyst. The reaction mixture in a long neck beaker and exposed to microwave irradiation for about 10-15 min (at interval of 5 min.). The reaction product obtained was compound A



Compound-A

General method for the preparation pyrazol ligands

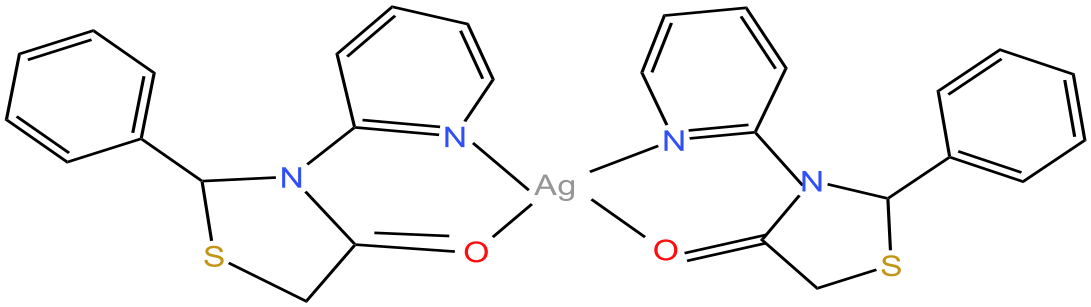
From the literature1,2 the compound A obtained from the above reaction was further refluxed under toluene solvent with phenyl hydrazine it gives a Pyrazol ligands.

1) Synthesis of 2-(4,5-dihydro-1H-pyrazol-5-yl)phenol

A solution of (E)-3-(dimethylamino)-1-(2- hydroxyphenyl) prop-2-en-1-one with phenyl hydrazine were added and the reaction mixture stirred for 10-12 hours under reflux condition. TLC monitoring of the reaction showed complete transformation. The reaction mixture was then cooled with crushed ice. The resulting product having black color were filtered off, washed with cold water and dried in vacuum

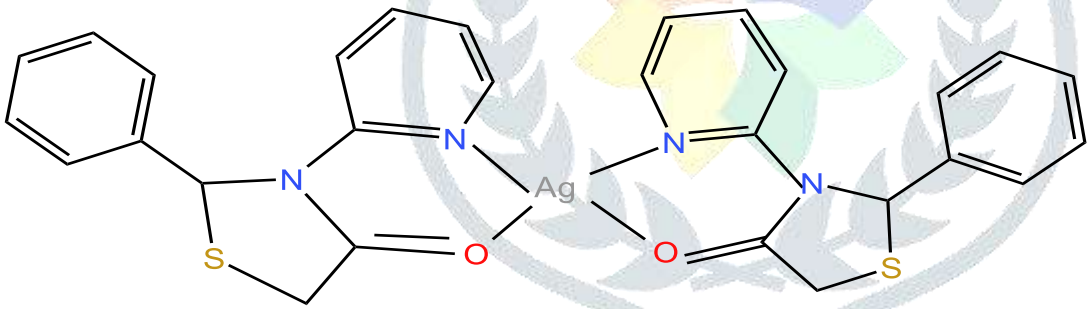
Synthesis of metal chelate;

Preparation of bis (2-(4,5 dihydro -1HPyrazol-5yl) Phenoxy Copper: A weighted quantity of ligand 2-(4,5 dihydro-1HPyrazol-5yl) Phenol (0.02) in slight excess was taken in a round bottomed flask containing anhydrous ethanol (30ml) and refluxed for few minutes with constant stirring to ensure complete dissolution. A solution of (0.01mol) appropriate metal salt (Copper chloride) in anhydrous ethanol (20ml) was then added drop by drop with constant stirring in the hot solution of ligand. The contents were refluxed for two hours. After cooling, the pH of the mixture was maintained by the addition of dilute alcoholic ammonia solution. The complexes of different metals were precipitated at various pH ranges. The resultant precipitate was digested for one hour. The precipitate was filtered, washed with hot ethanol (40 to 60^0) and dried in vacuum desiccators over anhydrous granular calcium chloride and stored in a air tight glass bottle.



Preparation of bis (2-(4,5 dihydro -1HPyrazol-5yl) Phenoxy Silver: A weighted quantity of ligand 2-(4,5 dihydro-1HPyrazol-5yl) Phenol (0.02) in slight excess was taken in a round bottomed flask containing anhydrous ethanol (30ml) and refluxed for few minutes with constant stirring to ensure complete dissolution. A solution of (0.01mol) appropriate metal salt (Silver Nitrate) in anhydrous ethanol (20ml) was then added drop by drop with constant stirring in the hot solution of ligand. The contents were refluxed for two hours. After cooling, the pH of the mixture was maintained by the addition of dilute alcoholic ammonia solution. The complexes of different metals were precipitated at various pH ranges. The resultant precipitate was digested for one hour.

The precipitate was filtered, washed with hot ethanol (40 to 60^0) and dried in vacuum desiccators over anhydrous granular calcium chloride and stored in a air tight glass bottle.

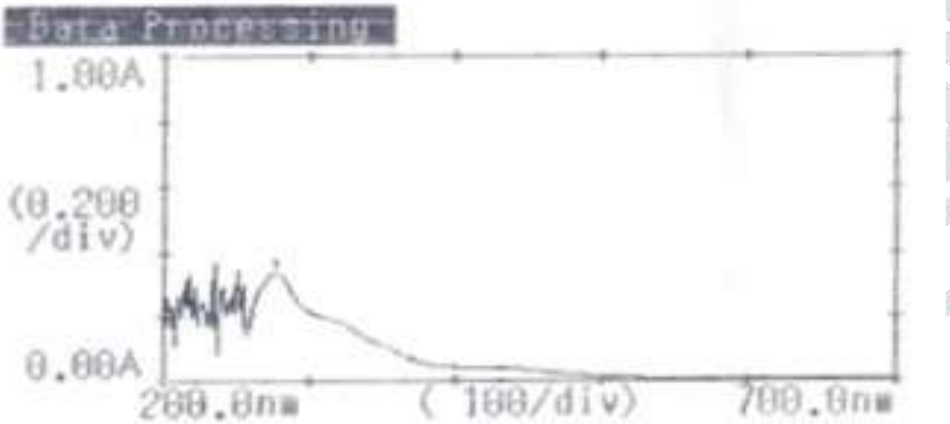


Analytical data of Cu(II), Ag(II) metal chelate with 2 (4,5-dihydro-1H-pyrazol-5yl) Phenol.

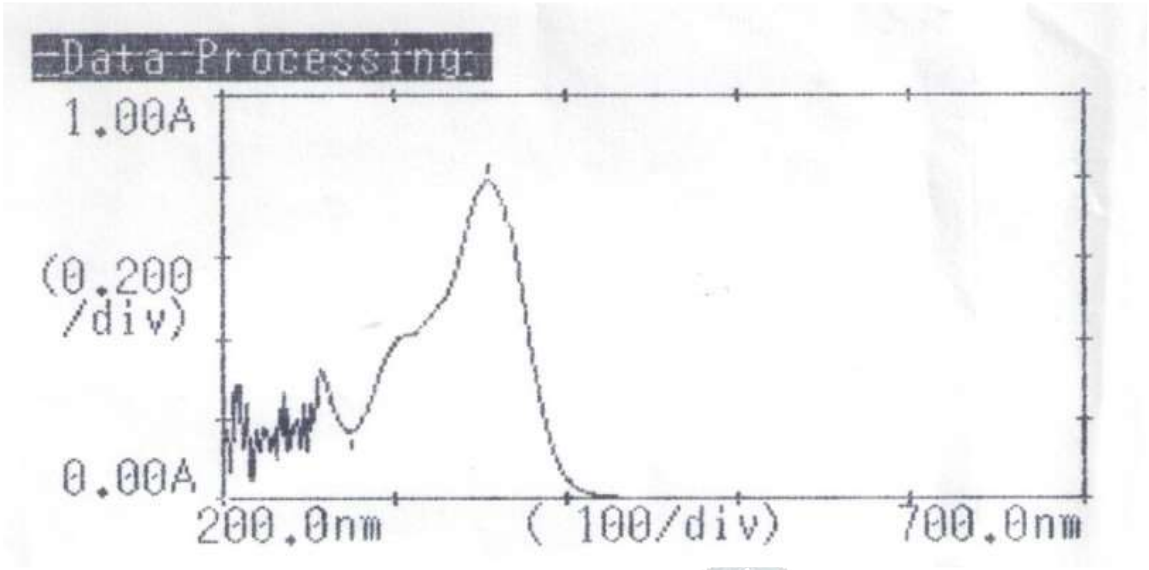
Ligand / Metal chelate Empirical formula	Mol.Wt D.P ^{0}c	ML Ratio	Molar conductance	Elemental analysis				
				C	H	N	M	
$C_9H_{10}N_2O$	162			66.60 (66.65)	6.15 (6.21)	17.22 (17.27)		
$C_{18}H_{18}CuN_4O_2$	385	1;2	16.5	56.96 (56.02)	4.61 (4.70)	14.63 (14.52)	16.35 (16.47	
$C_{18}H_{18}AgN_4O_2$	429	1;2	13.21	50.1 (50.25	4.46 (4.22)	13.10 (13.02)	24.98 (25.01)	

The ligand 2(4, 5-dihydro-1H-pyrazol-5yl) Phenol is used to prepared metal chelate of Cu(II), Ag(II). After preparation of metal chelate it is characterized by color, decomposition point, conductance, and elemental analysis, physical, chemical and spectral properties are determined.

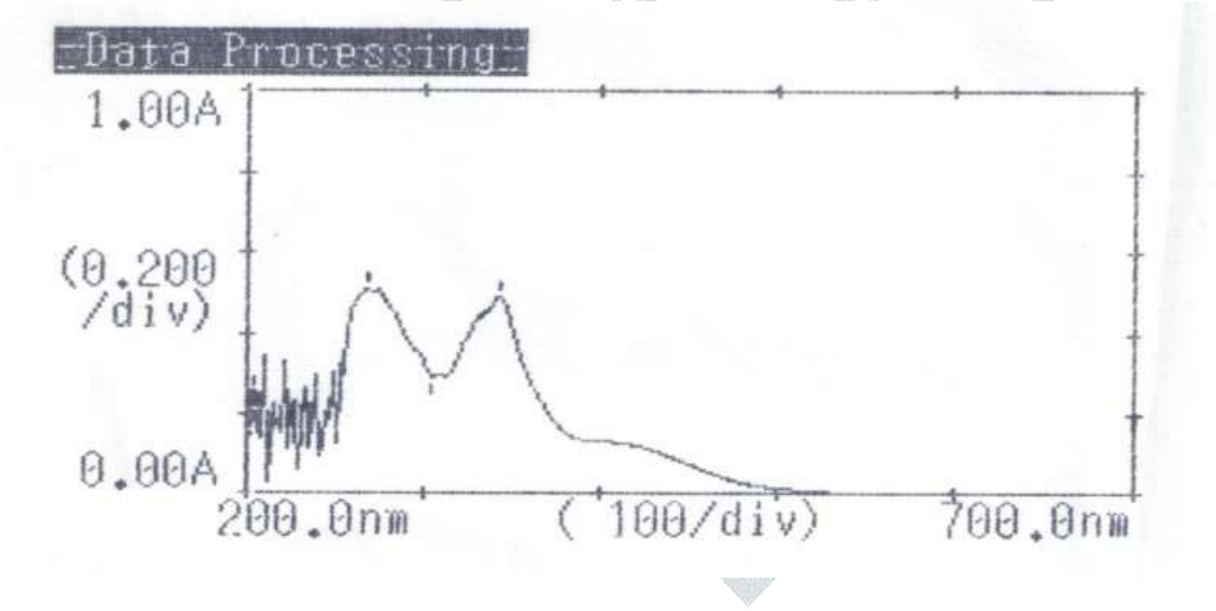
L1

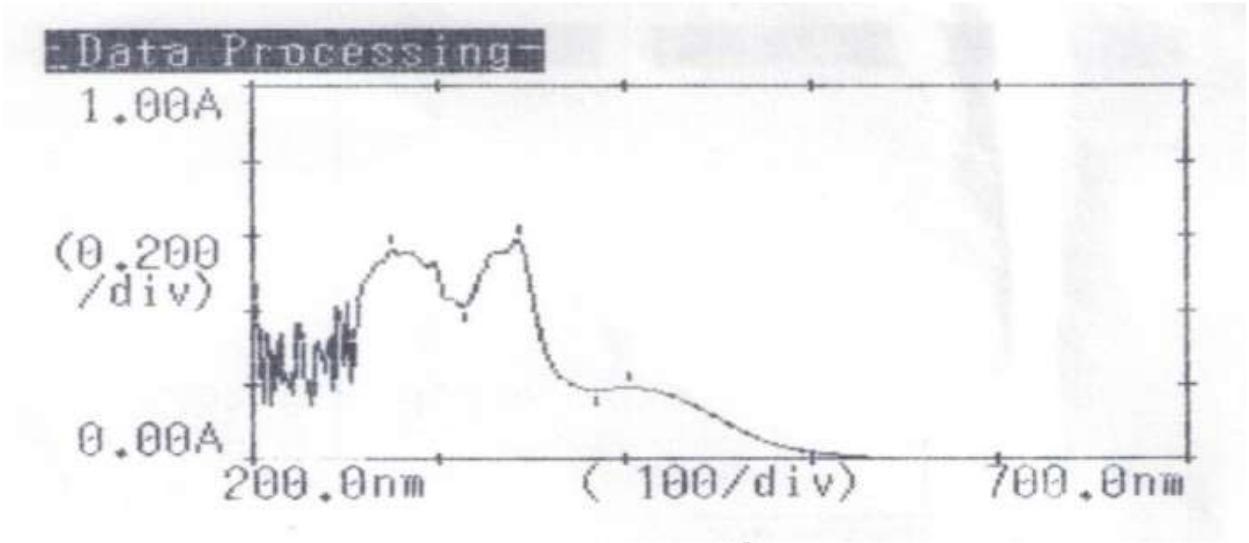


1


Data Processing
1.00A
(0.200
/div)
0.00A
200.0nm
(100/div)
700.0nm

L2


Data Processing
1.00A
(0.200
/div)
0.00A
200.0nm
(100/div)
700.0nm



U.V. Visible Spectral Studies:-Electronic spectra of ligand and mononuclear complexes of the copper and silver complexes have been recorded in the DMSO solution in the range 200-800 nm. The electronic spectrum of ligand shows strong bands in the ultraviolet region (260-401 nm) this shows that the transition ∏--∏* and n—∏* of the aromatic ring (Benzene) or diazomethane (-CN) group for free ligand [8] .Electronic Spectral of metal complexes the bands of high or low wavelength side shows to its free ligand in different solutions. The absorption bands between 261nm and 402nm in the free ligand changed in the metal complexes. The absorption shift and the intensity change in the spectra of metal complexes changed because of conjugation and delocalization of the whole electronic system and result in the energy change of the ∏--∏*and n—∏* of transition of conjugation chromospheres [9-10] in case of copper complexes the absorption bands in the visible region are 410-623nm similarly for silver complexes the absorption bands 403--579nm the nature of Cu (II) and Ag (II) affects the position of absorption bands.

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 position [11] and many of the complex with less bulky N-alkyl group or N-substituent are planar [12-13]

(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 Gyarfas E.C., *Chem. Rev*., 54, 959. **1954.**
(7) Morsey, A.M. Abou, Y., Raja D. and Varslav 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.**