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Synthesis and Characterization of Mixed Ligand Complexes With Ethyl -□- Isonitrosoacetoacetate and Dienes

Anita Taksande[1] R. D. Raut[2] M. D. Choudhary[3]
[1]S.D.College of Engineering, Wardha, India, [2]J.B.College of Science, Wardha, India, [3]B.D.College of Engineering, Wardha, India

Abstract: *Mixed ligand Co(II), Ni(II) and Cu(II) complexes $Co(EINA)_2(PPD)$, $Ni(EINA)_2(PPD)$ and $Cu(EINA)_2(PPD)$, formed with ethyl-α-isonitrosoacetoacetate and dienes have been synthesized and characterized by their elemental analysis, infra red spectra and magnetic measurements. Ethyl-α-isonitrosoacetoacetate shows a tridentate behavior with coordination occurring through two oximino nitrogen atoms and further two coordination sides of metal ion are satisfied by oxygen atom. Diene acts as a bidentate ligand coordinating through the two nitrogen of the amino group.*

Keywords: *characterization, Dienes Co (II), Cu (II) and Ni (II), Ethyl-α-isonitrosoacetoacetate. Synthesis.*

I. Introduction

The role of mixed ligand complexes in biological systems is well known[1]. Synthesis and charecterization of some bivalent simple metal complexes of isonitrosoacetophenon have been reported[2-5]. Ethyl-*α*-isonitrosoacetoacetate (EINA) is bidentate ligand containing two corboxylate groups and NH group. It can coordinate with the metal ion through the only two oximino nitrogen atom acting as a bidentate ligand. It also coordinates through the corboxylate oxygen atom and the nitrogen of the amino group and thus acts as a bidentate ligand. Mixed ligand complexes of Co (II) and Cu (II) and Ni (II) has been reported[6-8]. The present paper outlines the synthesis and characterization of mixed ligand complexes formed with ethyl-*α*-isonitrosoacetoacetate and *p*-phenylenediamine (PPD).

II. Experimental

All the chemicals used were of analytical grade.

2.1 Preparation of Mixed Ligand Complexes

A warm ethanolic solution of metal salts (0.01 M) was added to ethanalic solution of ligand (0.02 M) and *p*-phenylenediamine (0.01 M). The resulting solution was refluxed for about six hours. The complex thus formed was filtered and washed with alcohol and dried in vacuum over fused $CaCl_2$. The metal estimation was carried out by standard method and nitrogen by Kjeldahl method. The conductance was measured in DMF and DMSO solvent on and Elico CM-82 Conductivity Bridge. The magnetic susceptibility measurement at room temperature was made on Gouy's balance. The IR spectra were recorded on Perkin-Elmer-137 instrument in Nujol mull/KBr pellets ^{1}H NMR spectra were recorded on a Bruker WP 80 SY spectrometer.

III. Results And Discussion

The elemental analysis shown in Table 1 indicates that all the metal complexes have 1:2 stoichiometry and are dark colour substances, soluble in DMF and DMSO. The molar conductance values obtained for these complexes at the concentration of 10^{-3} are in the range of 2.9 to1.07 ohm^{-1} mo1 cm^2. These values are too low to account for any dissociation of the complexes can be regarded as non-electrolytes. The magnetic moment values for Cu(II) complexes are in the range of 1.58 BM, Ni(II) complexes 2.46 BM and Co(II) 4.28 BM having octahedral structure.

Table-1: Elemental Analysis and Magnetic Moment of the Complexes

Sr. No	Empirical Formula	% of Carbon Found Calc.	% of Hydrogen Found Calc.	% of Nitrogen Found Calc.	% of Metal Found Calc.	Molar conductance concentration $=10^{-3}$	Magnetic moment µBM
1	$Cu(EINA)_2PPD$	44.35 (44.30)	5.40 (5.33)	11.50 (11.48)	13.30 (13.33)	1.07	1.58

2	Ni $(EINA)_2$ PPD	44.80 (44.72)	5.45 (5.38)	11.62 (11.59)	12.25 (12.20)	1.87	2.46
3	$Co(EINA)_2PPD$	44.70 (44.74)	5.42 (5.38)	11.62 (11.60)	12.18 (12.15)	2.9	4.28

The infrared spectra of HEINA, PPD and its metal complexes with Co (II), Ni(II) and Cu(II) have been recorded. And the significant infra red bands are summarized in Table 2. Coordination sites of the HEINA, PPD have been determined by careful comparison of infra red spectra of the complexes. The infra red spectra of HEINA and PPD gave a strong band at 3410 cm^{-1} which may be assigned to OH and N-OH group. Which was absent in metal complexes.

Table-2:

Assignments	**HEINA**	**$Ni(EINA)_2PPD$**	**$CO(EINA)_2PPD$**	**$Cu(EINA)_2PPD$**
OH &N-OH	3410	-----	-----	---
-O-H	-----	3216	3326	3220
C=O	1620	----	----	-----
C=N	-----	1590	1604	1610

The infra red spectra of HEINA gave a strong band at 1620 cm^{-1} which may be attributed to the ester C=O. The infra red spectra of metal complexes gave two new band at 3216 cm^{-1} and 1590 cm^{-1} which is due to O-H and –C=N group take part in coordination of ligand.

The strong band due to υC = O seen at 1620 cm^{-1} in (HEINA) (PPD) is absent in all the complexes indicating a successful attachment of carboxyl oxygen to metal ion. The υC = N band appear in the 1590-1628/cm reason for metal complexes of (HEINA) (PPD).

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