SYNTHESIS AND MICROBIAL CHARACTERIZATION OF ISONITROSO

PHENYL 2 PROPANONE WITH CU(II), NI(II) & CO(II)

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ABSTRACT

Metal complexes of the formulae M (INP2P) $_2$ where M=Cu (II), Ni (II) and Co (II) with Isonoitroso Phenyl 2 propanone have been synthesized and characterized. The I.R. electronic spectral data in all the complexes, the ligand is coordinated to the central metal ion through nitrogen and oxygen.

Key-words: Magnetic susceptibility, Isonitroso Phenyl 2- propanone, conductivity.

INTRODUCTION

Transition metals and their derivatives with ligands have evoked much interest due to their inherent biopotency, striking structural aspects and unique stereo and magneto chemistry[1-2]. Coordination compounds containing ONS donar are of considerable importance due to their antimicrobial activity[3-4]. A much work have been carried out and studied by synthesizing complexes of transition metal with P-Chloro isonitroso acetophenone, Isonitroso 4-chloro acetophenone and Ethyl1 Isonitrosoacetoacetate. But yet complexes of transition metal with Isonitroso Phenyl 2 Propanone was not studied. In view of the interesting ligating behavior of INP2P we synthesized and characterized complexes of INP2P with *Cu (II), Ni (II) Co (II).*

Preparation of Metal complexes

1. Preparation of Cu(INP2P)$_2$: 0.199gms of copper acetate was dissolved in minimum quantity of alcohol and equal volume of water was added. Similarly 0.326gms of HINP2P was dissolved in minimum quantity of alcohol was added. The copper solution was added to reagent solution drop wise with constant stirring in a beaker. The solid complex formed was digested on water bath for 2 Hrs. On cooling it was filtered through filter paper and washed with 50% alcohol.

2. Preparation of Ni(INP2P)$_2$: 0.326gms of HINP2P was dissolved in minimum volume of alcohol and equal volume of water was added. Similarly 0.237gms Nickel acetate was dissolved in alcohol and water (1:1). The nickel solution was added to reagent solution drop wise with

constant stirring. The pH was adjusted to 5-5.6 with HCl/NH_4OH. A green colour solid complex was formed, separated, recrystalised from chloroform.

3. Preparation of $Co(INP2P)_2$: Aqueous solution of Cobalt nitrate and HINP2P was mixed in the molar ration of 1:2 and pH of solution was maintained 6-6.5 by $HCl/NH4OH$. On refluxing for an hour yellow colour complex was formed, filtered and recrystalised from chloroform.

Physical Measurements:

Magnetic measurements of solid complexes were carried out from room temperature to liquid nitrogen temperature with Gouy's balance. Dimagnetic corrections were calculated by the method given in the literature. The I.R. Spectra were recorded on Perkin Elmer-137 instrument in Nujol mull/KBr Pellets at IPER, Wardha. The conductance measurements were made in nitrobenzene solution.

Results and Discussion: The physical and analytical data in Table-1 indicate that *Cu (II), Ni (II) and Co (II) can be reported as M (INP2P) 2.* The complexes are colored and have good thermal stability. They are insoluble in water and dilute alkali solutions suggesting absence of free oxime group. Due to removal of free oximino proton during complexation supported by I.R. studies. The molar conductance value in nitro benzene indicates their non electrolytic nature.

Magnetic Measurements: The results of magnetic susceptibility measurements in a variable temperature data suggest that the Cu (II), Ni (II) complexes may be diamagnetic with square planner geometries. And a Co (II) complex is diamagnetic which suggest a low spin octahedral coordination. The magnetic moment of Cu(II), Ni(II), Co (II) complexes are in the range of 1.49-2.05B.M, 2-3.0 -3.67 B.M. and 3.90- 5.23 B.M. respectively. These suggestions are in agreement with spectral data[9-10].

Electronic Spectra: The ligand field transition of complexes show a group of three or more absorption band around 7002950 Nm regions. The envelops of this bands are generally unsymmetrical Seeming to encompass several overlapping transition[11]. The electronic spectra of INP2P in methanol shows An intense band at 47.84 kK. It splits into two strong bands at 43.10 kK and 42.79 kK. The electronic Spectrum of Ni (II) complex was consistent with a very broad intense band in the 52.35 kK region and band in the 41.32 kK range. A shoulder also appears in the 50.76 kK region. This region screened by the intense charge transfer

band exhibited broad maxima at 51.81 kK. The electronic spectrum of Cu (II) complex resembles to those of tetrahedral or square planner Cu (II) complexes. A broad band for Co (II) at 49.50 kK in Co (INP2P) $_2$ suggest distorted octahedral geometry. Distorted octahedral species of Co (II) complexes are also characterized by the presence of two main absorption bands[12].

Infrared Spectra: I.R. Spectra (4000-400cm^{-1}) of the complexes are practically identical. The frequency Of some significant band of the free ligand and those of the metal complexes are reported in Table No.2. The observed frequencies of different groups in the metal complexes have been assigned on the basis of literature data. The vO-H of the oxime group observed at 3163 cm^{-1} in (INP2P) is absent in the spectra of Complexes suggesting replacement of the oxime proton by the metal ion during complexation[13]. INP2P reveals two peaks at 1435cm^{-1} and 1480 which may attributed to the ester C=O. The band near 1567 cm-1 may be assigned to the C=N stretching frequency which is usually appears in the spectra of oxime in the range of 1685-1650 cm-1. The N-O frequency in simple oxime appears between 1071-1085 cm-1. The peak near 1074-1080 cm-1 and in INP2P may therefore attribute to N-O stretching mode[14-16].

¹HNMR: NMR Spectrum of Cu(INP2P)2, Ni(INP2P)2 & Co(INP2P)2 in DMSO solution exhibit peak due to –CH, methylene and aromatic ring protons & do not show any proton signal due to =N-OH. This suggest that these complexes have been formed by replacement of =N-OH group by the metal ion. It is interesting to note that the peaks Due to methylene proton in Cu(INP2P)2 appear at lower value compared to that of methylene proton in the reagent INP2P.

Antimicrobial Activity : The in vitro antibacterial activity was performed against gram positive bacteria including staphylococcus aureus , streptococcus pyogenes and gram negative bacteria including Escherichia coli, Pseudomonas aeruginosa. Yeast including candida albicans and fungi Aspergillus clavatus were used to test antifungal activity. Known antibiotics like Amplicilline and Chloramphenicol and Fluconazole were used for comparison as a reference drug.

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

Compound	% C	% H	% N	%Metal	☐

INP2P	66.4 (66.25)	5.70 (5.52)	8.31 (8.53)	------	------
Cu(INP2P)$_2$	55.94 (55.74)	4.22 (4.12)	7.11 (7.22)	16.12 (16.38)	1.72
Ni(INP2P)$_2$	56.32 (56.44)	4.12 (4.18)	7.32 (7.31)	15.15 (15.33)	2.86
Co(INP2P)$_2$	56.00 (56.40)	4.02 (4.17)	7.13 (7.31)	15.12 (15.39)	4.66

* Theoretical calculated value in parenthesis

Table-2: Infra Red Spectra of INP2P and Complexes.

Assignments	INP2P	Cu(INP2P)$_2$	Ni(INP2P)$_2$	Co(INP2P)$_2$
OH, Ar-H	3163	-----	-----	----
Ar-H	----	3623	3620	3660
C=O	1774	----	----	----
C=N, C=O	----	1435	1480	1456
N- Oxide	1071	1075	1080	1074
N-O	----	802	840	842

Table-3: NMR Spectra of INP2P and Complexes

Assignments	INP2P	Cu(INP2P)$_2$	Ni(INP2P)$_2$	Co(INP2P)$_2$
=N-OH	8.50 δ	-----	-----	-----
-CH group	6.23 δ	6.17 δ	6.17 δ	6.17 δ
-CH$_2$ group	3.40 δ	3.38 δ	3.38 δ	3.38 δ
Aromatic Ring	7.22 δ	7.18 δ	7.18 δ	7.18 δ

Table-4: Antibacterial & Antifungal activity of INP2P & Complexes

Compound	Antibacterial activity				Antifungal activity	
	Gram + ve		Gram -ve		C. Albicans	A. Clavatus
	S.pyrogens	S. Aureus	E.Coli	P.Aeruginosa		
INP2P	-	++	++	-	++	++
Cu(INP2P)$_2$	++	+++	+	-	++	++
Ni(INP2P)$_2$	+	++	-	+	++	++
Co(INP2P)$_2$	-	+	-	+	++	++
Ampicilin	++	++	++	++	-	-
Chloramphenicol	+++	+++	++	++	-	-
Fluconazole	-	-	-	-	+++	+++

+++: Good   ++: Moderate   +: Poor   - : Non Effective

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