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Synthesis and characterization of mixed ligand complexes of Cr (II), Cu (II) and Ni (II) metals containing DMG and dicarboxylic acids as ligands

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ABSTRACT

The organic ligand contain nitrogen, oxygen and carbonyl group were used for preparation of metal ligand mixed chelated complexes. Mixed ligand complexes of nickel (II), copper (II) and Chromium (II) metals with DMG and dicarboxylic acid acids such as oxalic acid & phthalic acid. The formed metal ligand mixed complexes were characterized by molar conductivity measurements, micro elemental analysis, UV- visible spectroscopy, Infra red spectroscopy of these complexes. The complexes are of (1:1:1) (Metal: Ligand: Ligand) Stoichiometry. The structural analysis indicates a four co-ordinated [M (DMG) (Oxalate)] & [M (DMG) (PTH)] Chromophore and distorted tetrahedral geometry and may be square planer geometry metal and ligand molecules. The magnetic properties were determined by using Gouy balance method. The magnetic moment was found to be in between 3.20 BM to 4.88 BM. These value is calculated by using spin only formula.

Keywords: DMG, Oxalic acid, Pthalic acid, chromium, Nickel, Copper etc.

INTRODUCTION

In chemistry a complexes are formed by the combination of metal and ligands. It is also called as co-ordination compound or metal complex. The structure of these complexes is depends upon the central metal atom and surrounding ligand molecule they are connected to the metal atom by coordinate bond. Transition metal ions are playing important role in human body [1,2] for Ex. Copper. Nickel, Chromium etc. These metals are obtained in human body. They are found in the form of different enzyme molecule [3,4] The organic bidentade ligands, is the important class of ligand in coordination chemistry and find extensive application in different fields [5] the ligands contain donor atoms like Nitrogen. Oxygen and carbonyl groups as like organic acids such as oxalic Acid and Phthalic acid contain carbonyl group therefore its interaction with metal atom ions and gives complexes of different geometries and are found to be biological activity [6,7] In recent years , there has been renewed interest in the synthesis and study of mixed ligand transition metal complexes. The utility aspects of these complexes have received their share of attention as these applications in divers fields [8] some metal ligand complexes are found to catalyze different organic reactions such as Oxidation, reduction, oxidative cleavage, hydroformylation etc and it shows catalyses like activity in decomposition of hydrogen peroxide [9] the coordination chemistry of copper(II) attracts much attention because of its biological relevance and its own interesting coordination chemistry such as geometry, flexible redox property , and oxidation state[10-11].

The Cu (I) complexes are biologically relevant because they are able to reductively activate molecular oxygen (O_2). How ever, due to the labiality of most Cu (I) complexes, they typically lack sufficient kinetic stability for radiopharmaceutical applications. We report here the synthesis, characterization and biological activity of some new chromium, copper and nickel metal ligand complexes containing DMG organic acids such as oxalic acid and Pthalic acid as a ligand. The synthesized complexes are characterized by elemental, IR & electronic spectroscopic analysis.

MATERIALS AND METHODS

Metal salts such as chromium chloride ,copper sulphate, nickel chloride and ligand molecules such as Dimethylglyoxime (DMG), Oxalic acid and Pthalic acid and solvent such as DMF, Alcohol, DMSO, Alcoholic KOH solution all were purchased of A.R. Grade quality and were from BDH and Babaji traders Parbhani. The double glass distill water was used.

Synthesis of [Cr (DMG) (Phth)] Cl_2

Prepare 1 mmol aqueous solution of metal salt in distilled water and dissolve ligand DMG (1mmol) and pthalic acid (1mmol) in ethanol. Than prepare 1:1 ligand solution of DMG & pthalic acid. This mixer of ligand solution was added into metal salt solution drop wise with constant steering up to 30 min. stir this reaction mixture 1 hours using magnetic stirrer at room temperature. Filtered the obtained complex, washed with ethanol first than acetone and hot water and dried in vacuum discicater. This solid complex was recrystalise by using ethanol and measure yield & physical constant of complex.

Reaction: $CrCl_2$ + DMG + Pthalic acid → [Cu (DMG) (Phth)] Cl_2

Synthesis of [Cu (DMG) (oxalate)] $So4^{2-}$

Firstly dissolve the 1 mmol of cuso4 in distilled water and dissolve ligand DMG (1mmol) and oxalic acid (1mmol) in ethanol. Than prepare 1:1 ligand solution of DMG & Oxalic acid. This miscible solution was added into Cuso4 solution drop wise with constant steering up to 30 min. stir this reaction mixture 50-55 min using magnetic stirrer at room temperature. Filtered the obtained complex, washed with ethanol first than acetone and hot water and dried in vacuum discicater. This solid complex was recrystalise by using ethanol and measure yield & physical constant of complex.

Reaction: $CuSo_4$ + DMG + $C_2H_2O_4$ → [Cu (DMG) (oxalate)] $So4^{2-}$

Synthesis of [Ni (DMG) (Phth)] $So4^{2-}$

Firstly prepare the aqueous solution of $Niso_4$ (1mmol) & dissolve the ligand molecules DMG (1mmol) & Phthalic acid (1mmol) in ethanol. Add one after another into metal solution and stir this reaction mixture unto 90 min It gives the formation of radish colored complex is formed. Filtered it washed it by using ethanol and acetone , dried it and recrystalise by using ethanolic solution , measure the yield of complex and note down melting point of complex. Remaining complexes was synthesized by using same method mentioned above.

Reaction: $Niso_4$ + DMG + Phthalic Acid ⟶ [Ni (DMG) (Phth)] $So4^{2-}$

RESULTS AND DISCUSSION

Instrumentation:

The complexes were analyzed for the elements contains C,H & O using standard procedure for the determination of metal content determined by complex metric EDTA titration [12]. Analyses for carbon & hydrogen were carried out at the micro analytical methods in laboratory.

The molar conductance value were measured in ethanol at the range of 10^{-2} M concentration on a model M-180 Elico digital conductivity cell fitted with a platinum electrode (cell constant =1.0cm^{-1}). The magnetic moment property of complexes were measured by a Gouy balance Hg [Co $(SCN)_4$] as the calibrant. Effective diamagnetic correction for the ligand components using Pascal's constant [13].

The specific optical rotation values for the prepared complexes were determined in ethanol solution (0.02%) on a Jusco DIP-140 polarimeter. Electronic absorption spectra in ultraviolent range in ethanol at 10^{-3} M concentration are

measured on Era UV-160 Spectrophotometrical. The physical and elemental analysis of synthesized complexes shown in table-1.

Table 1: Physical properties and elemental analysis of mixed ligand complexes

Sr.No	Compound	color	M.P(0^c)	Yield	Elemental Analysis				
	-	-	-	-	C	O	N	H	M
01	PTH (Pthalic acid)	White	223	-	-	-	-	-	-
02	DMG (Dimethylglyoxime)	White	241	-	-	-	-	-	-
03	Cr[(DMG)(PTH)2H_2O]	Brown	196	68%	30.50 (30.00)	25.60 (26.10)	10.12 (10.00)	18.20 (18.60)	15.20 (15.10)
04	Cr[(DMG)(oxalate)2H_2O]	Gray brown	210	65%	24.80 (24.60)	26.30 (26.50)	10.20 (10.00)	18.80 (19.00)	15.18 (15.10)
05	Cu[(DMG)(PTH)2H_2O]	Blue wish	170	72%	25.40 (25.00)	27.28 (27.40)	11.30 (11.50)	18.20 (18.60)	14.15 (14.07)
06	Cu[(DMG)(oxalate)2H_2O]	Light blue	235	70%	24.80 (24.60)	27.10 (27.40)	11.40 (11.50)	18.60 (18.40)	14.20 (14.07)
07	Ni[(DMG)(PTH)2H_2O]	Radish brown	190	76%	29.50 (30.00)	28.20 (28.60)	13.60 (13.80)	18.20 (18.60)	16.75 (16.80)
08	Ni[(DMG)(oxalate)2H_2O]	brown	210	72%	24.80 (24.60)	28.40 (28.60)	13.40 (13.80)	19.80 (20.00)	16.84 (16.80)

IR Spectra:

From the evidence of IR spectra (Table no-2) taken the absorption band is obtained in complex containing Pthalic acid as a ligand they show –C=C- stretching at 1600-1470 cm^{-1} i.e. aromatic (-C=C-) stretching but this peak is absent in the complex containing oxalic acid as a ligand. The carbonyl peak at 1645-1655 cm-1 i.e. (C=o) stretching in carboxylic acid group. The C-H stretching in DMG ligand is at 3100-3010 cm-1 & C=N stretching at 1422 cm-1 and O-H starching of water molecule is 3210-3300 cm-1.

Table 2: IR Spectra of synthesized complexes

Sr. No.	Complex	(C-H) Stre.	Ar (C=C)	(C=N)	(C=O)	M-O
01	Cr[(DMG)(PTH)2H_2O]	3040	1440-1600	1502	1654	530
02	Cr[(DMG)(oxalate)2H_2O]	3060	-	1498	1648	522
03	Cu[(DMG)(PTH)2H_2O]	2998	1455-1610	1498	1648	530
04	Cu[(DMG)(oxalate)2H_2O]	3020	-	1500	1645	560
05	Ni[(DMG)(PTH)2H_2O]	3060	1430-1600	1502	1650	515
06	Ni[(DMG)(oxalate)2H_2O]	3040	-	1500	1655	588

Electronic Spectra of synthesized complexes:

The electronic spectra of synthesized complexes is measured in DMSO solvent (Table no-3). It is observed in the range 200-700 nm at room temperature. The electronic spectra of mixed metal ligand complexes the absorption band at 270 nm were assigned it indicates the complex containing (C=C) in benzene ring such as phthalic acid but this absorption band is absent in the complex of oxalic acid as a ligand. The transition band at 330-400 nm is associated due to (C=0) n⟶ $\Pi*$ transition in carbonyl group of carboxylic acid present in ligand molecule.

Table-3 Electronic Spectra of mixed metal ligand complexes

Sr. No.	Complex	Electronic Spectra (nm)		
		$\Pi \longrightarrow \Pi*$	n⟶ $\Pi*$	L⟶ *M*
01	Cr[(DMG)(PTH)2H_2O]	245	380	475
02	Cr[(DMG)(oxalate)2H_2O]	-	345	470
03	Cu[(DMG)(PTH)2H_2O]	275	365	460
04	Cu[(DMG)(oxalate)2H_2O]	-	340	450
05	Ni[(DMG)(PTH)2H_2O]	280	350	448
06	Ni[(DMG)(oxalate)2H_2O]	-	365	440

CONCLUSION

The complexes were obtained good yield at room temperature using magnetic stirrer. The synthesized complexes were characterized using elemental analysis, UV spectra and IR spectra and confirmed by tacking TCL and physical constant. The complexes were obtained as colored powder form and they were stable at room temperature. The possible geometry of synthesized complexes is square planer and it is four coordinated metal ligand complexes.

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