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NOTE

Thermal and Spectral Studies of Ni (II) and Cu(II) Complexes with Chloramphenicol

PRANAY GURU
Department of Chemistry, Dr H.S. Gour University, Sagar-470 003, India

Ni(II) and Cu(II) complexes with chloramphenicol have been synthesized and characterized. On the basis of elemental analysis and molar conductance, formulae $Ni(L)MoO_4 \cdot 2H_2O$ and $Cu(L)MoO_4 \cdot 4H_2O$ (L = chloramphenicol) have been suggested for the complexes under study. The geometries of the complexes have been proposed on the basis of magnetic moment, electron and infrared spectral data. TGA studies have also been carried out to know the pattern of their decomposition. The crystal system, lattice parameters, unit cell volume and number of molecules in it have been determined by X-ray diffraction data; various ligand field parameters like Dq, B, etc. have been evaluated. The aim of investigation is to study the coordination behaviour of Ni and Cu in the presence of MoO_4^{2-} anion.

Key Words: Ni (II), Cu(II), Complexes, Chloramphenicol, Thermal and spectral studies.

In continuation of the work being carried out in this laboratory on the metal molybdate with organic ligand[1], the present note describes two new complexes of nickel(II) and copper(II) with chloramphenicol(L) in the presence of molybdate. The complexes have been synthesized and characterized using analytical and spectral methods.

The starting material $M\ MoO_4 \cdot nH_2O$ [where M = Ni(II) and Cu(II); n = 2–4] was synthesized by reported methods[2-4]. Complexes were isolated by shaking $M\ MoO_4 \cdot nH_2O$ (0.01 mol) with the required amount of $C_{11}H_{12}Cl_2N_2O_5$ (0.03 mol) in water (*ca.* 100 mL). The products were filtered, washed 3–4 times with diethyl ether and dried. Elemental analyses of the prepared complexes were carried out at Lab India and ASCHO Laboratory, Mumbai, while X-ray diffractions (XRD) were performed at Inter-University Consortium, Indore. Thermogravimetric and infrared spectral analyses (FTIR) were performed at Centre for Advanced Technology, Indore. KBr pellets were used in the FTIR spectral analyses. The weight loss was measured from room temperature up to 950°C at a heating rate of 15°C per min. The electronic spectra of the solutions of the complexes in water (taken at *ca.* concentration M/500) were recorded on Chemito-2500 UV/Visible spectrophotometer. Electronic spectra were recorded at Forensic Science Laboratory, Sagar in the range of 300–900 nm.

The physical and analytical data of the prepared complexes were presented in Table-1. The nickel(II) and copper(II) complexes were yellow and green in colour respectively. Molecular formulae of the complexes have been worked out as ML $MoO_4 \cdot nH_2O$ [where M = Ni(II) and Cu(II), L = ligand, n = 2–4]. Prepared complexes are insoluble in water and soluble in common organic solvents like dimethyl formamide. The Co(II) complex shows lower value of conductance (41–62 ohm^{-1} cm^2 mol^{-1}). Ni(II) complex also shows lower value of conductance (40–46 ohm^{-1} cm^2 mol^{-1}). The lower conductance values indicate the non-electrolyte nature of these complexes[6].

TABLE-1
ANALYTICAL AND PHYSICAL DATA OF THE COMPLEXES

m.f.	Observed (Calculated) %							
	Colour	M.W.	*Metal	MoO_4	C	H	N	Cl
$Ni(L)MoO_4 \cdot 2H_2O$	Yellow	581.57	9.789 (10.091)	23.895 (23.540)	24.256 (22.690)	3.123 (2.750)	4.787 (4.813)	11.836 (12.204)
$Cu(L)MoO_4 \cdot 4H_2O$	Green	622.58	10.145 (10.206)	25.978 (26.332)	21.983 (21.202)	2.961 (3.212)	4.532 (4.497)	11.743 (11.404)

*Metal = Ni/Cu, L= chloramphenicol

The magnetic moment of the Ni(II) complex is 3.24 B.M. corresponding to two unpaired electrons. Electronic spectra of the Ni(II) complex shows three distinct bands appearing at 10595, 16315 and 26393 cm^{-1} (ν_3). The magnetic moment of the Cu(II) complex is 1.83 B.M. which indicates the presence of one unpaired electron. The electronic spectra of the complex shows one broad band in the region 15613 cm^{-1}.

Interpretations of IR bands of the complex have been carried out by comparing with the IR spectrum of chloramphenicol[7–9]. The NH stretching frequency bond is shifted to lower side at 3340 cm^{-1}. In the present case asymmetrical and symmetrical bands due to NH or NH_2 group in drug and the complex were observed at 3260 cm^{-1}. Band appearing near 1695 cm^{-1} of strong intensity may be due to the strong shifted frequency of this carbonyl group. The band which is due to stretching vibration of hydroxy OH also remained unchanged. The stretching bond observation of nitro group in free ligand appears at 1530 and 1358 cm^{-1} but after complexation these bands are shifted to lower side appearing at 1514 and 1351 cm^{-1}.

The thermogravimetric data show the decomposition of complexes in two steps. In the first step the weight loss is 300–430 K, which indicates the loss of loosely bound water of crystallization while in the second step, the thermogram shows the loss of ligand molecules of the complex between 440–910 K. The metal oxides are formed in both the cases.

The X-ray pattern was obtained by trial and error method[10–12]. The unit cell parameters were calculated from indexed data. It is also clear from the data that Ni(II) complex possesses tetragonal symmetry, whereas Cu(II) complex possesses hexagonal symmetry. The calculated and experimental values of the densities of the complexes are in good agreement within the limits of experimental error.

TABLE-2
CRYSTAL PARAMETERS AND DENSITY OF THE COMPLEXES

Complexes	Crystal lattice edge (Å)			Cell volume (Å)	n	Density observed (Density calcd.)	Crystal system
	a	b	c				
$Ni(L)MoO_4 \cdot 2H_2O$	11.881	11.881	11.231	1585.528	3	1.803 (1.827)	Tetragonal
$Cu(L)MoO_4 \cdot 4H_2O$	18.187	18.187	20.803	6881.018	21	3.289 (3.154)	Hexagonal

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