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STUDIES OF TWO COMPLEXES WITH AMPICILLIN

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Abstract: Ni(II) and Cu(II) complex with ampicillin have been synthesized and characterized. On the basis of elemental analysis and molar conductance, formulas $Ni(C_{16}H_{19}N_3O_4S)MoO_4 \cdot 4H_2O$ and $Cu(C_{16}H_{19}N_3O_4S)MoO_4 \cdot 4H_2O$ have been suggested for the complexes under study. The geometries of the complexes have been proposed on the basis of magnetic moment, electronic and infrared spectral data. Thermo-gravimetric analysis (TGA) has been carried out to determine 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 (XRD). The aim of investigation is to study coordination behavior of Ni and Cu having molybdate anion (MoO_4), in the presence of ampicillin(6-[D(-)-α-Amino-α-Phenylacetamido]Penicil-Lanic Acid).
Key words: Ni(II) and Cu(II) complex with Ampicillin.

INTRODUCTION

In continuation of the work being carried out in this laboratory on the metal molybdate with organic ligand, the present note describes two new complexes of nickel(II) and copper(II) with ampicillin ($C_{16}H_{19}N_3O_4S$) having molybdate. The complexes have been synthesized and characterized using analytical and spectral methods

EXPERIMENTAL

The starting material M MoO_4 $4H_20$ [where M = Ni(II) / Cu(II)] was synthesized by reported methods[1,2]. Complexes were isolated by shaking MoO_4 nH_20 (0.01 mole) with a require amount of $C_{16}H_{19}N_3O_4S$ (0.03 mole) in water (~100 ml) The products were filtered, washed 3-4 times with ether and dried. The metals was estimated by usual methods of estimation[3] . Elemental analysis of prepared complexes were carried out at Lab India and ASCHO Lab Mumbai, X-ray diffraction analysis was carried out by Inter University Consortium (I.U.C.)Indore (M.P.), Infrared spectral analysis (FTIR) and Thermo-gravimetric of synthesized complexes were performed at Centre for Advance Technology (CAT) Indore (M.P.), KBr pellets were used in FTIR spectral analyses. The weight loss measured by heating at the rate of 15° C/min up to 950°C. Unico Gouy's balance using $Hg[Co(NCS)_4]$ as standard used for magnetic studies. The molar conductance of prepared complexes was measured in liquid phase using Conductivity Bridge.

RESULTS AND DISCUSSIONS

Table 1 shows Physical and analytical data of the prepared complexes. The nickel(II) and copper(II) complexes found green and brown in color respectively Molecular formula of the complexes has been worked out on the basis of the above data, to M $C_{16}H_{19}N_3O_4SMoO_4 \cdot 4H_2O$ [where M = Ni (II)/ Cu (II)]. Synthesized complexes are insoluble in water and soluble in common organic solvents, indicating non-electrolyte nature of these complexes.

The magnetic moment of the Ni(II) complex is 3.42 B.M. Correspond to two unpaired electrons . In the electronic spectra of the Ni (II) complex shows three distinct bands appears at 838 nm (ν1) , 516 nm (ν2), 316.5 nm (ν3) which may be assigned to $^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$ (ν_1) , $^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$ (ν_2) , $^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ (ν_3) transition respectively . The ligand field parameter Dq (1193.6), B (614) and β(0.56) are in good agreement with those for an octahedral geometry for nickel(II) complexes .

The magnetic moment of the Cu(II) complex is 1.9 B.M. indicates the presence of one unpaired electron .The electronic spectra of the complex shows one broad band in the region 610 nm which may occur due to the overlapping of the $^2B_{1g} \rightarrow {}^2A_{1g}$ band of ligand field transition, suggested a square planer geometry to the complex.

Interpretation of IR bands of the complex have been carried out comparing with the spectrum of IR of ampicillin and related compound have been well studied [4-6].

The bands present in the drug due to νNH_2 (3200 cm^{-1}) amide C = O (1690 cm^{-1}) NH_2 (2860 cm^{-1}) and NH_3^+ (1495 cm^{-1}) remained almost lower side in the complex indicating involvement of these group in chelation a new amino group is produced due to degradation of βLactum carbonyl group by metal ion. This group also take part in chelation in complex vibration due to this new group may be observed at 3100 cm^{-1} .

The band present at 1770 cm^{-1} in the drug was assigned to β. Lactum carbonyl group and was found absent in complexes, because after degradation this group gets converted into – COOH group(ampicillin → ampicillonic acid) carboxylic group present in Ligand displays its vibration due to asymn. and syn. stretching at 1612 cm^{-1} and 1410 cm^{-1} .

Although this group takes part in chelation by deprotonation but at the same time a new carboxylic group is produced by degradation of βLactum carbonyl group. Hence in complexes two new bands appearing at 1590 cm^{-1} and 1370 cm^{-1} were assigned to shifted

asymmetric and symmetric stretch of original carboxylic group, new carboxyl group shown its vibration at normal frequencies (1620 Cm^{-1}) and (1400 Cm^{-1}).

In this complex presence of –OH group is supported by a sharp and strong bond at 3380 cm^{-1} and also by the band present at 1115 cm^{-1}, which indicates Cu-OH bending.

The thermo-gravimetric data in shows the decomposition of complexes in two steps. First step weight loss 300-425 K which indicates the loss of loosely bound water of crystallization . The second step in the thermogram shows the loss of ligand molecules of the complex. Which occurs between 450-950 K . The metal oxide are formed in the both cases.

The X ray diffraction data of the these complexes shows in Table 2. It records 34 and 20 peaks for Ni(II) and Cu(II) respectively clearly indicating the crystalline nature of complexes. The X- ray pattern by trail and error method [7-10]. The unit cell parameters were calculated from indexed data. It is also clear from the data that Ni(II) complexes posses Tetragonal symmetry , whereas Cu(II) complexes posses cubic symmetry. The calculated and experimental values of density of the complexes are good agreement within the limits of experimental error.

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Table 1: Analytical and physical data of the complexes

Mol. formula	**Observed/ calculated %**							
	Colour	**M.W.**	**Metal***	**MoO_4**	**C**	**H**	**N**	**S**
Ni (C_{16} H_{19} N_3 O_4S) MoO_4 $4H_2O$	Green	644.05	9.377 (9.115)	25.600 (25.454)	30.891 (29.811)	4.021 (4.192)	5.965 (6.521)	5.234 (4.968)
Cu (C_{16} H_{19} N_3 O_4S) MoO_4 $4H_2O$	Brown	644.88	9.162 (9.79)	25.982 (25.265)	30.456 (29.589)	3.691 (3.698)	6.537 (6.472)	4.541 (4.931)

Metal* = Ni /Cu

Table 2: Crystal parameters and density of the complexes

Complexes	Crystal lattice Edge (Å)			Cell volume	n	Density obs. / Density calc.	Crystal System
	a	b	C	Å ^{3}			
Ni (C_{16} H_{19} N_3 O_4S) MoO_4 $4H_2O$	17.3869	17.3869	11.9743	3619.9405	10	3.169 / 2.954	Tetragonal
Cu (C_{16} H_{19} N_3 O_4S) MoO_4 $4H_2O$	12.3775	12.3775	12.3775	1896.2941	3	1.699 / 1.702	Cubic

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