

## NOTE

## Studies on Complexes of Nickel(II) and Copper(II) with Ampicillin

PRANAY GURU

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

Tel: (91)(7582)222170, (91)9826313501 (Mobile)

E-mail: pranayguru@rediffmail.com

Ni(II) and Cu(II) complexes with ampicillin with the general formula  $M(C_{16}H_{19}N_3O_4S)WO_4 \cdot 4H_2O$  ( $M = Ni$  or  $Cu$ ) have been synthesized and characterized on the basis of elemental analysis and molar conductance. The geometries of the complexes have been investigated by magnetic, electronic and infrared spectral data. TGA studies have also been carried out to know the pattern of their decomposition.

**Key Words:** Nickel(II), Copper(II), Complexes, Ampicillin.

In continuation of the work being carried out in this laboratory on the metal tungstate with organic ligand<sup>1, 2</sup> the present note describes two new complexes of nickel(II) and copper(II) with 6-[D(-)- $\alpha$ -amino- $\alpha$ -phenylacetamido] penicillanic acid ( $C_{16}H_{19}N_3O_4S$ ) having tungstate. The complexes have been synthesized and characterized using analytical and spectral methods.

The starting material  $MWO_4 \cdot 4H_2O$  [where  $M = Ni(II)/Cu(II)$ ] was synthesized by reported methods<sup>1-3</sup>. Complexes were isolated by shaking  $MWO_4 \cdot 4H_2O$  (0.01 mol) with the required amount of  $C_{16}H_{19}N_3O_4S$  (0.03 mol) in water (*ca.* 100 mL). The products were filtered, washed 3–4 times with ether and dried. The metals were estimated by reported methods<sup>4, 5</sup>. Elemental analyses of prepared complexes were carried out by Lab India and ASCHO Lab, Mumbai. Thermogravimetric and Infrared spectral analyses (FTIR) of synthesized complexes were performed at Centre for Advance Technology (CAT), Indore (M.P.), India. KBr pellets was used in FTIR spectral analyses. The weight loss was measured from room temperature up to 950°C at a heating rate of 15°C per minute.

Table-I shows physical and analytical data of the prepared complexes. Synthesized complexes are insoluble in water and soluble in common organic solvents, indicating non-electrolyte nature of these complexes.<sup>5</sup>

The magnetic moment of the Ni(II) complex is 3.70 BM corresponding to two unpaired electrons. The electronic spectrum of the Ni(II) complex shows two distinct bands appearing at  $16693\text{ cm}^{-1}$  ( $\nu_2$ ),  $26316\text{ cm}^{-1}$  ( $\nu_3$ ) which may be assigned to  ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$  ( $\nu_2$ ),  ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$  ( $\nu_3$ ) transitions respectively. The ligand field parameters  $Dq$  (1035),  $B$  (779.12) and  $\beta$  (0.72) are in good agreement with those for an octahedral geometry for nickel(II) complexes.

The magnetic moment of the Cu(II) complex is 1.98 BM indicating the presence of one unpaired electron. The electronic spectra of the complex show one broad band in the region  $12396\text{ cm}^{-1}$  which may occur due to the overlapping

of the  ${}^2B_{1g} \rightarrow {}^2A_{1g}$  band of ligand field transition, suggesting a square-planar geometry for the complex.

TABLE-I  
ANALYTICAL AND PHYSICAL DATA OF THE COMPLEXES

m.f.	Colour	m.w.	Observed/Calculated (%)					
			M*	WO <sub>4</sub>	C	H	N	S
Ni(L)WO <sub>4</sub> ·4H <sub>2</sub> O	Green	727.96	8.229 (8.066)	33.850 (34.048)	27.012 (26.376)	3.579 (3.709)	6.013 (5.769)	4.538 (4.396)
Cu(L)WO <sub>4</sub> ·4H <sub>2</sub> O	Brown	732.75	8.801 (8.666)	34.012 (33.825)	26.898 (26.203)	3.532 (3.685)	5.996 (5.732)	4.425 (4.368)

L = C<sub>16</sub>H<sub>19</sub>N<sub>3</sub>O<sub>4</sub>S

Interpretations of IR bands of the complex have been carried out comparing with the spectrum of IR of ampicillin and the related compounds<sup>6-8</sup>. The bands present in the drug due to  $\nu(\text{NH}_2)$  (3235 cm<sup>-1</sup>), amide C=O (1695 cm<sup>-1</sup>), NH<sub>2</sub> (2860 cm<sup>-1</sup>) and NH<sub>3</sub><sup>+</sup> (1495 cm<sup>-1</sup>) decreased on complexation with metal ions indicating involvement of these groups in chelation. A new amino group is produced due to degradation of  $\beta$ -lactam carbonyl group by metal ion. This group also takes part in chelation in complex vibration due to this new group may be observed.

The band present at 1770 cm<sup>-1</sup> in the drug was assigned to  $\beta$ -lactam carbonyl group and was found absent in complexes, because after degradation this group gets converted into —COOH group (ampicillin  $\rightarrow$  ampicillonic acid). The carboxylic group present in the ligand displays its vibration due to asymmetric and symmetric stretching at 1612 and 1410 cm<sup>-1</sup> respectively.

The thermogravimetric analysis prove the decomposition of complexes in two steps. First step shows a weight loss at 300–410 K indicating the loss of loosely bound water of crystallization. The second step in the thermogram represents the loss of ligand molecules of the complex, which occurs between 425–960 K. Metal oxides are formed in both the cases.

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