

International Journal of ChemTech Research CODEN(USA): IJCRGG ISSN : 0974-4290 Vol.1, No.3, pp 552-554, July-Sept 2009

STUDIES OF NICKEL COMPOUND WITH CHLORAMPHENICOL

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ABSTRACT : The present paper deals with the spectral, x-ray studies of the Ni complex with Chloramphenicol. On the basis of elemental analysis and molar conductance, formula $Ni(C_{11}H_{12}Cl_2N_2O_5)VO_3 \cdot 3H_2O$ has been suggested for the complex under study. The geometries of the complex 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. The aim of investigation is to study coordination behavior of Ni in the presence of VO_3 anion.

Keyword - Nickel, Thermo-gravimetric analysis, Infrared spectroscopy, X-ray Powder Diffraction

INTRODUCTION

The ancient man used the raw extracts of plants for the cure of human sufferings without knowing their chemical constituents and active principles. The importance of plant products, their utility encouraged human mind to find out the mysteries and marvels of biogenic processes. For attaining this aim, drugs have been thoroughly investigated throughout the ages and it was noticed that several metal complexes with a drug possess more medicinal importance as compared to the corresponding drug.

In continuation of the work being carried out in laboratory on the metal vanadate with organic ligand, the present note describes Nickel(II) with Chloramphenicol $(C_{11}H_{12}Cl_2N_2O_5)$ in the presence of vanadate. The complex has been synthesized and characterized using analytical and spectral methods





EXPERIMENTAL

The starting material $MVO_3 \cdot 3H_20$ [where M = Ni] were synthesized by reported methods¹⁻³. Complex were isolation by shaking MVO₃•nH₂0 (10 mmol, 0.25 g) with a required amount of ligand (30 mmol, 0.75 g) in water (~100 mL). The products were filtered, washed 3-4 times with diethyl ether and dried. The metals were estimated by usual methods of estimation⁴. Elemental analyses of the prepared complexes were carried out by Lab India and Ascho Laboratory Mumbai; X-ray diffraction (XRD) of the prepared complex was carried out by the Inter University Consortium (IUC), Indore, India. Thermogravimetric and Infrared spectral analyses (FTIR) of synthesized complexes were performed at Centre for Advanced Technology (CAT) Indore, India, 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 minute. The molar conductance of prepared complexes was measured in liquid phase using conductivity Bridge.

RESULTS AND DISCUSSIONS

Analytical and physical data of the prepared complex tabulated Table 1. The Nickel(II) complex found light purple in colour. Molecular formula of the complexes has been worked out on the basis of the above data. Prepared complexes are insoluble in water and soluble in common organic solvents, indicating non-electrolyte nature of this complex.



Magnetic Measurements and Electronic spectra

The magnetic moment of the Ni(II) complex is 4.53 B.M. correspond to two unpaired electrons . Electronic spectra of the Ni(II) complex shows two distinct bands appearing at 18,779 cm⁻¹ (v2), 22,100 cm⁻¹ (v3) which may be assigned to ${}^{4}T_{1g}(F) \rightarrow {}^{4}A_{2g}$ (F)(v₂) and ${}^{4}T_{1o}(F) \rightarrow$ (v_3) transition respectively. The ligand field ${}^{4}T_{2o}(P)$ parameter are in good agreement with those for an octahedral geometry of the Nickel(II) complex ^{5,6}.

Infrared Spectra

Interpretation of IR bands of the complex have been carried out by comparing with the IR spectrum of Chloramphenicol $^{7-9}$. The NH stretching frequency a bond sifted lower side at ~3310 Cm⁻¹. In the present case asymmetrical and symmetrical bands due to NH or NH₂ group in drug and the complex were observed at 3190cm⁻¹. In complex near band appearing near 1690 cm⁻¹ of strong intensity may be due to strong shifted frequency of this carbonyl group .The band observation in Chloramphenicol and complex near 3345 are due to stretching vibration and deformation of alcoholic- OH mode.

The band, which is due to stretching vibration of hydroxyl OH, also remained unchanged. The stretching

Purple

| Table 1: Analytical and physical data of the complex | | | | | | | |
|--|------------------------|--------|--------|-----------------|--------|-------|-------|
| Mol. formula | Observed/ calculated % | | | | | | |
| | Colour | M.W. | Metal* | VO ₃ | С | Н | Ν |
| $Ni(L)VO_3 \cdot 3H_2O$ | Light | 599.97 | 10.713 | 22.332 | 32.525 | 2.956 | 5.121 |

(20.929)

(31.998)

(2.836)

(4.978)

(10.101)





Figure 2. Representative structure of the complexes.

bond observation of Nitro group in free ligand appears at 1545 & 1350 but after formation of complex symmetric bands is shifted to lower frequencies by 10-15 cm⁻¹ and 7-10 cm⁻¹ respectively.

X-ray Powder Diffraction Studies (XRD)

The X-ray diffraction data of the these complex shows 15 peaks for Ni(II) complex clearly indicating the crystalline nature of complex. The X-ray pattern by trail and error method¹⁰⁻¹¹. The unit cell parameters were calculated from indexed data. It is also clear from the data that Ni(II) complex posses tetragonal symmetry. The calculated and experimental values of density of the complexes are good agreement within the limits of experimental error.

Thermogravimetric Studies (TGA)

The thermo gravimetric data shows of complex in two steps. First step weight loss 325-450 K, which indicates the loss of loosely, bound water of crystallization. The second step in the thermo gram shows the loss of ligand molecules of the complex. Which occurs between 490-925 K. The metal oxides are formed in the both cases.

ACKNOWLEDGMENT

The authors are thankful to Dr A. K. Guru, Ex Director, State Forensic Science Laboratory Sagar (M.P.) for providing necessary laboratory facilities and valuable suggestions. The authors are also thankful to Dr. Bhawalkar, Director and Dr. V.K. Wadhawan, Head

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Laser and material division , Center for Advance Technology (CAT) Indore, India and Dr. Ajay Gupta, Centre Director, Inter University Consortium (I.U.C.) Indore India. for granting permission to use Instrumental facilities at their laboratory

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