

## Preparation of Some Complexes of Nickel (II) and Copper (II) Metals with Macrocyclic Ligand, 1,7,10,16- tetraaza-8, 9, 17, 18- dibenzo - 3,4,5,12,13,14 -Difuranyl Cyclo Octadeca - 1, 6, 10, 15-Tetraene

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### ABSTRACT

The complexes of Nickel(II) and Copper(II) salts have been prepared with the macrocyclic ligand-1,7,10,16-tetraaza-8, 9, 17, 18-dibenzo - 3,4,5,12,13,14-difuranylcyclooctadeca- 1, 6, 10, 15-tetraene. On the basis of characterization of complexes formed with the macrocyclic ligand by usual physico-chemical methods such as elemental analysis, measurement of electrical conductance, magnetic moment and electronic transition spectra of the complexes and I.R. spectral behaviour of both the ligand and the complexes. All the complexes of both the metals have been found to be non-electrolyte, mono-meric, paramagnetic with octahedral geometry . The general molecular formula of the complexes have been found to be  $[M(L)(X)_2]$  Where M= Nickel(II) and Copper(II) Cations, L= macrocyclic ligand and X= monodentate anion.

**Keywords :** Macrocyclic Ligand, Complex, Paramagnetic, Magnetic Moment, Mono-Meric, Octahedral Geometry.

### I. INTRODUCTION

A considerable amount of research work in the field of coordination compounds with macrocyclic ligand has been carried out. But at least work has been done with the macrocyclic ligand which contains 2,5-furandicarboxaldehyde in its moiety. In this paper, we report the formation of complexes of Nickel(II) and Copper(II) metals with a such macrocyclic ligand which contains 2,5-furandicarboxaldehyde in its moiety.

#### Preparation of the complexes:

**Procedure:-** Equimolar mixture (0.001) of Nickel(II) chloride hexahydrate, o-phenylene diamine and 2,5-furandicarboxaldehyde was completely dissolved in minimum volume of ethyl alcohol and the resulting solution was filtered and the filtrate was collected in small round bottom flask. The resulting solution was refluxed continuously under a condenser fitted with a running water tap for 5 to 6 hours . Then the solution was cooled and transferred in a petridish and allowed to stand for over night for slow evaporation which after cooling produced light green crystalline

solid. The solid separated by filtration and washed with a small amount of cold water followed by a small amount of acetone. Then the solid was dried over KOH pellets placed in a desiccator. There compound was found to be insoluble in cold water and common organic solvents. The ratio of metal, o-phenyldiamine and 2,5-furandicarboxaldehyde was always kept into 1:2:2 respectively. The similar procedure was adopted for the preparation of Bro-

mide, nitrate, acetate and perchlorate salts of Nickel and Copper metals. In case of copper metal, the colour of the complexes was found to quite different than those of Nickel metal.

The compound was further analysed and found to contain the general molecular formula  $[M](L)(X)_2$  Where M= divalent Nickel(II) and Copper(II) metals.

L=  $C_{24}H_{16}N_4O_2$  = macrocyclic ligand X= Unidentate anions.

#### Chemical required:

Acetates/ Chlorides, Bromide, Nitrate and Perchlorate of Nickel(II) and Copper(II) metals, Dimethylformamide, ethyl alcohol, sodium hydroxide, 2,5-difurandicarboxaldehyde, O-phenylene di- amine, calcium chloride, Common organic solvents, silver nitrate, etc. All chemicals were taken either of BDH (A.R.) quality or E.Merk extra pure. They were used without further purification.

#### ANALYTICAL METHODS:

The estimation of metals and non-metals present in the complexes has been done by standard methods:-

- (i) Nickel :- Nickel has been estimated by gravimetrically by dimethoyl glyoximate method
- (ii) Copper has been estimated by iodimetrically.
- (iii) Hydrogen, Carbon and Nitrogen:- Hydrogen, Carbon and Nitrogen was estimated by semi-macro-duma's method.

(iv) Chlorine- Chlorine was gravimetrically estimated as AgCl

(v) Bromine-Bromine was estimated gravimetrically as AgBr

#### CONDUCTIVITY MEASUREMENT:

The measurement of electrical conductivity of the solutions of the complexes was done by conductivity meter bridge manufactured by Wiss-Techen Wearch Stathen type-LBR-at room temperature. Pure DMF and conductivity water were used as solvent.

#### U.V. -VISIBLE SPECTRA PHOTOMETRIC MEASUREMENT:-

Hitachi-320 spectrophotometer were used to record the electronic absorption spectra of the complexes.

**I.R. Spectra:-** Perkin Elemer 577 spectrophotometer was used to record the infra-red spectra of the complexes and the ligand in nujol mull.

Characteristics peaks shown by Nujol mull as such sharp peaks

at  $2960\text{ cm}^{-1}$  to  $2800\text{ cm}^{-1}$  and at  $1460\text{ cm}^{-1}$  and a small peak at about  $700\text{ cm}^{-1}$  has been excluded while recording the I.R. spectra for the complexes and the ligand.

#### MAGNETIC SUSCEPTIBILITIES:

For the measurement of the magnetic susceptibilities of the complexes was done by Gouy's method by using Mercury tetra thio cyanato cobaltate,  $(\text{Hg Co}(\text{SCN})_4)$  as calibrant.

#### Result and discussion:

The values of electrical conductance obtained for the Nickel(II) and Copper(II) complexes have been found to be in the range of 14-32 Am and 18-36 Am respectively which clearly indicates the non-electro-lyte nature of all the complexes.

The values of magnetic moment obtained for Nickel(II) and Copper(II) complexes have been found to be in the range of 3.20-3.38 BM and 1.88-2.10 BM

respectively clearly indicates distorted octahedral geometry for all the complexes of Nickel(II) and Copper(II) cations.

The values of electronic transition spectra obtained for Copper(II) complexes in the range of 14140-14350  $\text{cm}^{-1}$  which without any fail indicate the distorted octahedral geometry of all the complexes of Copper(II) cations. The nature of the band was broad and unsymmetrical which support the distorted octahedral geometry.

In case of Nickel(II) complexes, three bands have been obtained due to the following

$$\nu_1 = {}^3A_{2g}(F) \rightarrow {}^3T_{2g}(F), 8100-8250 \text{ cm}^{-1}$$

$$\nu_2 = {}^3A_{2g}(F) \rightarrow {}^3T_{1g}(F), 16250-17100 \text{ cm}^{-1} \text{ and}$$

$$\nu_3 = {}^3A_{2g}(F) \rightarrow {}^3T_{1g}(P), 21260-23180 \text{ cm}^{-1}$$

transitions which clearly indicate the distorted octahedral geometry of all the complexes.

***I.R. spectra of the macrocyclic ligand, 1,7,10,16-tetraaza-8, 9, 17, 18-dibenzo-3, 4,5,12,13,14-difuranlcyclooctadeca-1, 6, 10, 15 tetraene.***

For the formation of complexes with this macrocyclic ligand, (L<sub>1</sub>) 2,5-furandicarboxaldehyde, o-phenylenediamine and metal (II) salt have been taken in equimolar proportion. 2,5-furan dicarboxaldehyde contains which should give a sharp and medium band in the range of 1750-1780  $\text{cm}^{-1}$  and o-phenylenediamine contains primary carbonyl group (>C=O) which should provide a sharp and medium band in the range of 3450-3500  $\text{cm}^{-1}$  due to the vibration of carbonyl group (>C=O) and amino group (NH<sub>2</sub>) respectively indicating the presence of primary amino group in the ligand molecule. But in the I.R. spectra of macrocyclic ligand, no bands around 1750-1780  $\text{cm}^{-1}$  due to Carbonyl group (>C=O) and 3450-3500  $\text{cm}^{-1}$  due to the vibration of amino (-NH<sub>2</sub>) and have been obtained. It indicates the complete condensation of carbonyl group (>C=O) and amino group (-NH<sub>2</sub>) with the formation of

azomethimine group. The appearance of a new broad and medium band in the range of 1630-1640  $\text{cm}^{-1}$  due to the vibration of azomethine group. The formation of this band support the complete condensation of carbonyl and amino group. The band position obtained due to vibration of azomethine group has been reduced by 40-50  $\text{cm}^{-1}$  in the complexes, This reduction in the band position indicates that Nitrogen atom of azomethine group takes part in the complex formation. Thus a comparison of I.R. spectra of constituents of free macrocyclic ligand and the complexes, it has been found that the macrocyclic ligand 1,7,10,16-tetraaza-8, 9, 17, 18-dibenzo-3,4,5,12,13,14-difuranlcyclooctadeca-1, 6, 10, 15-tetraene are tetradentate molecule. In this molecule there are four azomethine group obtained due to the complete condensation for carbonyl groups and four primary amino groups. Out of six coordination sites of the metal cations, four coordination sites (secondary valencies) are satisfied by the four nitrogen atoms of four azomethine group.

In case of metal (II) chloride, a band obtained in the range of 315-320  $\text{cm}^{-1}$  due to the vibration of (M-Cl) bond indicates the coordination of chloride ion in the bond formation with the metal cation. In case of metal (II) bromide a sharp and medium band has been obtained in the range of 280-300  $\text{cm}^{-1}$  due to the vibration of M-Br bond. This indicates the participation of bromide ion in the complex formation with metal ion.

I.R. spectra of the nitrate complexes display three (N-O) stretching bands in the range of 1420-1460  $\text{cm}^{-1}$  ( $\nu_3$ ) 1310-1320  $\text{cm}^{-1}$  ( $\nu_2$ ) and 1020-1030  $\text{cm}^{-1}$  ( $\nu_1$ ). The separation of two highest frequencies bands ( $\nu_3 - \nu_2$ ) suggest that both the nitrate groups are coordinated in a unidentate manner. This has been supported by the presence of bands obtained in the range of 215-225  $\text{cm}^{-1}$  due to the vibration of (M-O) bonds in the all nitrate

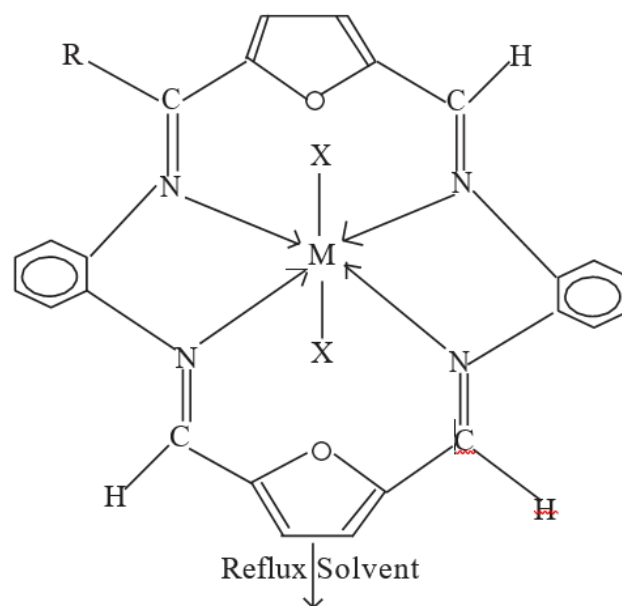
complexes. Thus it is concluded that bond formation between the metal ion and the ligand take place through one oxygen atom of nitrate ion.

In case of acetate complexes two bonds have been obtained in the range of  $1635-1645\text{ cm}^{-1}$  ( $\nu$ ) and  $1385-1395\text{ cm}^{-1}$  ( $\nu$ ). This indicates that acetate group is coordinated to the metal cation in a unidentate manner.

In case of perchlorate complexes two bands have been obtained in the range of  $1210-1220\text{ cm}^{-1}$  ( $\nu$ )  $1125-1135\text{ cm}^{-1}$  ( $\nu$ ). The presence of these two bands indicate that in perchlorate complexes perchlorate groups are coordinated to the metal cation in a unidentate manner.

Thus by the perusal of I.R. spectra of anions such as chloride ion ( $\text{Cl}^-$ ), bromide ion ( $\text{Br}^-$ ), nitrate ion ( $\text{NO}_3^-$ ), acetate ion ( $\text{CH}_3\text{COO}^-$ ) and perchlorate ion, ( $\text{ClO}_4^-$ ) all the anions are coordinated to the metal cation in a unidentate manner to satisfy the two primary valencies (charge of the metal cation) of the metal cation in the complex formation in the macrocyclic complexes.

Thus on the basis of elemental analysis, measurement of electrical conductance, magnetic moment and electronic transition spectra of the complexes and I.R. spectra behaviour of both ligand and the complexes, octahedral geometry of Nickel(II) and Copper(II) complexes has been suggested with both the macrocyclic ligand complexes and have been found to be non-electrolyte mono-meric, paramagnetic with their general molecular formula  $[\text{M}(\text{L})(\text{X})_2]$ . M= Nickel(II) and Copper(II) cations, L= Macrocylic ligands and X= Monodentate anions.



M=Nickel (II) & Copper (II) metals

X = Monodentate anions such as

Macrocylic ligand- 1, 7, 10, 16- tetraaza-8, 9,17, 18-dibenzo-3, 4, 5, 12, 13, 14- ifuranylcyclootadeca-1,6,10,15 -tetraene

$\text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{NO}_3^-$ ,  $\text{CH}_3\text{COO}^-$  and  $\text{ClO}_4^-$   
 $\text{X}' = -(\text{C}_6\text{H}_4)-$

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