

## SHORT NOTE ON PREPARATION OF THE LIGAND AND METHODS FOR ANALYSIS

RAKESH KUMAR RANJAN

Email ID rakeshkrra@gmail.com

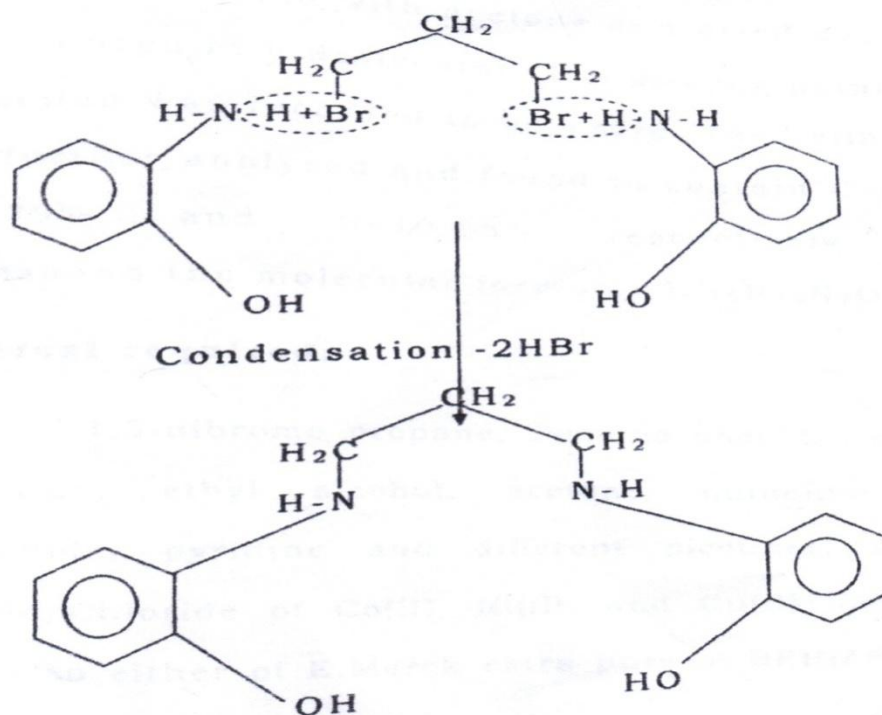
Research scholar, J. P. University Chapra (Bihar)

**ABSTRACT :-** This paper deals with preparation of the ligand (LH<sub>2</sub>) and the methods for elemental analysis of the ligand and the complexes the schiff base ligand (LH<sub>2</sub>) , 1, 3-bis (o-hydroxy) aniline) propane has been prepared by the condensation of 1, 3-dibromo propane and anilino) propane has been prepared by the condensation of 1, 3-dibromo propane and 2-amino phenol. The methods for the elemental analysis measurement of magnetic moment, electrical conductance, U.V. and I.R. Spectra have also been referred to this paper.

**Keywords :-** Ligand, Analysis, Conductivity, spectra magnetic, determination

### 1. Preparation of the ligand :-

The Schiff base ligand, 1, 3-bis (o-hydroxy aniline) propane has been synthesized by the condensation of 1, 3-dibromo propane and 2-amino phenol. The expected chemical reaction taking place during the course of condensation is given below:-



1, 3-bis (o-hydroxy anilino) propane

Fig-1

2. **Procedure :-** An ethanolic solution of 1.12 grams (0.01 mole) of 1, 3-dibromo propane was gradually added to alcoholic solution of 1.1 gram (0.01 mole) of 2- amino phenol with regular shaking. The resulting solution was heated on water bath for 3-hours with regular shaking. A yellow solid separated out on cooling under ice bath. The solid was separated by filtration. Washed with acetone and dried over fused CaCl<sub>2</sub> placed in a desiccator. The melting point of the compound was recorded to be 132<sup>o</sup>c. The yellow solid was further analysed and found to contain C=68.70%, H= 8.79% and N= 10.68% respectively which correspond to the molecular formulae [C<sub>15</sub>H<sub>18</sub>N<sub>2</sub>O<sub>2</sub>]

### 3. Chemical required :-

1, 3-dibromo propane, 2-amino phenol, ammonium hydroxide, ethyl alcohol, acetone, quinoline, phenyl isocyanide, pyridine and different picolines, methanol, Acetate/Chloride of Co (II), Ni(II) and Cu (II) metals used were also either of E. Merck extra pure of BKH(AR) quality They were used without further purification.

### 4. Analytical Methods :-

The estimation of metals in the complexes was carried out by standard methods described below. The complexes were first decomposed with a view to bringing the metals in their proper ionic solution and then they were quantitatively analysed.

#### 4.1 Cobalt :-

The compound was ignited for a few minutes, cooled and treated with a few drops of conc. Nitric acid by which the residual cobalt oxidized and the oxide of cobalt can be converted into nitrate. The product was heated to expel nitric acid. Finally excess of conc. Sulfuric acid was added to convert the nitrate into sulphate and then heated between 450-450<sup>o</sup>c and determined as CoSO<sub>4</sub> by weighing.

#### 4.2 Nickel :-

A weighed amount of nickel complex was decomposed and brought into ionic solution as chloride. Nickel was precipitated as nickel dimethyl glyoximate in slightly ammoniacal solution. The precipitate was quantitatively transferred to previously weighed sintered glass crucible the precipitate dried at 110<sup>o</sup>C - 120<sup>o</sup>C and then weighed as Ni(C<sub>4</sub>H<sub>7</sub>O<sub>2</sub>N<sub>2</sub>)<sub>2</sub>

#### 4.3 Copper :-

The weighed amount complex was decomposed in a pyrex beaker by requested evaporation with HNO<sub>3</sub> and HClO<sub>4</sub> until a clear solution was obtained. The solution was further boiled with HCl and the volume was reduced to 10 to 15 ml. the clear solution was diluted with water and then copper was determined iodometrically.

#### 4.4 Nitrogen :-

It was determined by semi-macro Duma's method.

### 5. Conductivity Measurement :-

Electrical Conductivity of solutions of complexes were measured by conductivity meter bridge manufactured by wiss-Techen wearch stathen type LBR at room temperature in DMF. The Cell constant was measured at (Room temperature ) 30<sup>o</sup>c using N/10 and N/100 KCl Solution. Pure DMF and conductivity water were used as solvents.

#### 5.1 U.V. Visible spectra photometric measurement :-

The electronic absorption spectra of the complexes were recorded with Hitachi-320 spectrophotometer at CDRI, Lucknow.

#### 5.2 I.R Spectra :-

The infra red spectra of complexes and ligand were recorded in nujol mull on parkin Elemer 577 spectrophotometer at C.D.R.I. Lucknow.

Nujol gives characteristic sharp peaks at 2960 cm<sup>-1</sup> to 2800 cm<sup>-1</sup> and at 1460 cm<sup>-1</sup> to 1380 cm<sup>-1</sup> and a small peak at about 700 cm<sup>-1</sup>. The I.R. bands of complexes taken in nujol mull range has been excluded for the nujol mull while recording the I.R. spectra for Complexes.

### 6. Magnetic Susceptibilities :-

Magnetic susceptibilities of the complexes were determined by Gouy's method. A short description of the apparatus and its calibration is given below :

The apparatus consists of an electromagnet and a semi-micro balance. The current was taken from the 110 D.C. main through an adjustable resistance and an ammeter. Specimen tubes used were made of soda glass. Particular care was taken to ensure uniform diameter. These were provided with ground glass stoppers to which was attached with a glass hook for suspension. A very fine silk thread was used for suspending the tube in the field. The other end of the suspension was attached to bottom of one of the pans of the balance by means of an adjustable screw device. Thus the length of the suspension could be adjusted.

The pole pieces were fixed as distance of 1.8 cm and all measurements were made with 5 ampere current.

The centre of the maximum field between the pole pieces were determined by a topographical survey with the tube containing a column of paramagnetic substance like ferrous ammonium sulphate. The tube containing the specimen for measurement was always suspended between the poles in such a way that its lower end coincided with the centre. The maximum field strength was within a length for 7 cm below or above the point. The cotton of the suspension

tube was, therefore, always in the region of maximum. The field was found to be practically negligible at  $11.6 \text{ cm}^{-1}$  above this point. The specimen tube was therefore, always filled up to height to about 11.6 cm.

Filling up of the specimen tube requires some skill. Generally the substance was well powdered in a agate mortar. A small amount of the substance was then introduced into the tube and rammed with properly fitting glass rod. In This way the required length was filled in uniformly.

### 7. Determination of Maximum field strength :-

The field was determined by using a number of standard substances like copper sulphate, ferrous ammonium sulphate accurate values of their mass susceptibilities are known.

The field strength was calculated with the help of the equation :-

$$H^2_{\text{max}} = \frac{21g}{Xg.M} = \frac{21(\text{in cm})X W (\text{in gams})z 98}{XzXm (\text{In gm})}$$

Some people prefer w in milligram and m in gram so that.

$$X_g = \frac{21xwx10^3 \times 98}{H^2_{\text{max}} xm}$$

$$= \frac{21xw}{H^2_{\text{max}} xmx1.019}$$

Where ; L=Length of the tube

W=change in weight of the sample in milligram

M=weight of the sample in gram

$X_g$  =gram susceptibility of the substance

Using the known value for  $X_g$  of standard substances the tube calibration constant

$$\frac{21xw}{H^2_{\text{max}}}$$

Is calculated and then the expression applied for different samples.

The magnetic moment  $\mu$ (in B.M ) is then calculated using the expression

$$\mu_s = 2.84 \sqrt{XmC_{\text{corr}} T}$$

Where ,  $X_M = X_g \times M$  ( $M$ =molecular weight of sample and  $X_M^{\text{CORR}}$  is the corrected value of the susceptibility when correction is made of diamagnetism, using pascal's constants.

### Conclusion :-

The ligand has different doner sites and it can form chelate ring in various ways. The probable mode of attachment for the bond formation of the ligand molecule with the metal cations has been decided on the basis of the perusal of I.R. spectral behaviour of the ligand molecule.

### References :-

1. P.Lumbe : Sumen Kimistilchch, 32, 203 (1959).
2. N.K. Singh and N.R. Agrawal : Indian J. Chem. Fee, A37, 76(1998).
3. K.Nonayama, H.Ojima and M.Nonayama : Inorg. Chem. Acta, 59, 275(1982)
4. A. Ramchandraith, P. Nageshwara Rao and M.Ramaiah : Indian Journal of Chemistry, 28A, 309(1989)
5. H. Schiff. : Annal Phys, 150, 193 (1869)
6. Drago, R.S. : Phy.methods in Inorg. Chem., P. 395
7. B.N. Figgis and J.Lewis : Prog. Inorganic chem. 6, 211(1964)
8. C.N. Rao : Chemical Application of IR spectroscopy "Academic press" (1964)
9. F.G.Mann & B.C. Saunders : Practical Chemistry (1985)
10. B.N. Figgis : Introduction of Ligand Field, Willey Eastern, New Yark (1976).
11. Van Vleck, J.H. : The theory of electric and magnetic Susceptibilities, Oxford University Press, London, pp. 296-297, (1952)
12. Bose, I. : Ind. J. Phys. Phys., 22-23(1948)
13. L.J. Bellamy : The Infrared spectra of complex molecules " Methun & Colld" London, pp. 92-96.
14. S.N. Vidyarthi et al. : Journal of Physical Sc. 2(2010), 35-39.
15. S.N. Thakur : Asian Journal of Chem. 1, (283-287), (2002).