

Study of Schiff's Base Ligands by The Condensation of α -Amino Aceto Hydroxamic Acid with Diacti Monoxime

Dilip Kumar Ray¹, Dr. Arbind Kumar²

¹Research Scholar, University Department of Chemistry, B. R. A. Bihar University, Muzaffarpur, Bihar, India

²Associate Professor, P. G. Department of Chemistry, L. S. College, Muzaffarpur, B. R. A. Bihar University, Muzaffarpur, Bihar, India

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ABSTRACT

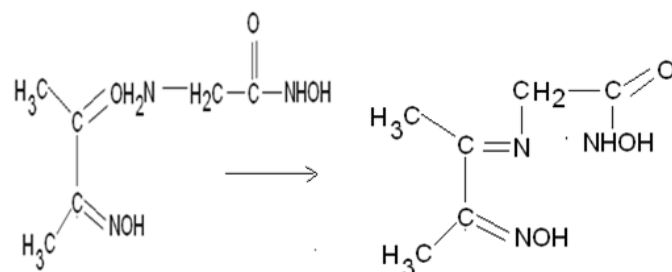
The article proposes Hydroxamic acids and their Schiff base derivatives are the most widely investigated ligands and their metal complexes is comprising worldwide attention.

Keywords: Hydroxamic acids, Ligands, Schiff's base ligands

I. INTRODUCTION

Hydroxamic acids and their Schiff base derivatives are the most widely investigated ligands and their metal complexes is comprising worldwide attention. Amino hydroxamic acids, due to the protonation of the amino group, have intricate acid base properties as shown by Farkas and Kiss. Anions of α -amino hydroxamic acids (such as glycine hydroxamic acid, $\text{NH}_2\text{CH}_2\text{CON}-\text{HOH}=\text{Gha}$) are potentially able to form three kinds of a five member chelate complex using either: 1- the two oxygens of the hydroxamic group, 2 – the two nitrogen atoms or, 3- the nitrogen atom of the amino group and the oxygen of carbonyl group. As a result, they are powerful ligands for a great number of metal ions.

Preparation of Schiff's base ligands by the condensation of α -amino aceto hydroxamic acid with diacti monoxime. The condensation may be represented by the tanoming scheme:



The following analytical results were obtained:

Table 1

Element	Found %	Calculated %
Carbon	41.51	41.62
Hydrogen	6.25	6.36
Nitrogen	24.13	24.28

which corresponds to the formulas, $(\text{C}_7\text{H}_{11}\text{N}_3\text{O}_3)$

II. PREPARATIONS OF LIGAND

III. ANALYTIC METHOD

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.

3.1. COBALT

The compound was ignited for a few minutes, cooled and then treated with a few drops of Conc. HNO₃ acid by which the residual carbon was oxidized and the oxide of cobalt was converted into nitrate. The product was heated to expel nitric acid. Finally excess of Conc. H₂SO₄ acid was added to convert the nitrate into sulphate and then heated between 450-500°C and determined as CoSO₄ by weighing.

3.2. NICKEL

As described in the estimation of cobalt, 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 100-120°C, was then weighed as [Ni (C₄H₇N₂O₂)₂].

3.3. COPPER

The weighed amount of copper complex was decomposed in a pyrex beaker by repeated evaporation with HNO₃ acid and HClO₄ until a clear solution was obtained. The solution was then 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.

3.4. NITROGEN

Nitrogen was determined by semi-micro-Duma's method.

IV. CONDUCTIVITY MEASUREMENTS

Electrical conductivity of solution of complexes was measured by conductivity Meter Bridge manufactured by Wiss-Techen Werch Stathen type-LBR at room temperature in dimethyl formamide. The cell constant at (room temperature) 300c using N\10 and N\100 KCl solution. Pure DMF and conductivity water were used as solvents.

V. U.V.-VISIBLE SPECTRA PHOTOMETRIC MEASUREMENT

The electronic absorption spectra of the complexes were recorded with Hitachi -320 spectrophotometer at C.D.R.I., Lucknow.

VI. I. R. SPECTRA

The infra-red spectra of the complexes and ligand were recorded as Nujol mull on Perkin Eeemer 577 spectrometer at C.D.R.I., Lucknow.

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

VII. CHEMICAL REQUIRED

Acetate\chloride of Co (II), Ni (II), Cu (II) used were either of EDH (A. R) or extra pure E. Merck quality. Quinoline, liquor ammonia, phenylisocynide, different picolines (α, β, γ) methyl anthranilate, 2-mercepto benzaldehyde used were also either of E. Merck extra pure or BDH (A.R.) quality.

VIII. MAGNETIC SUSCEPTIBILITIES

Magnetic Susceptibility of the complexes were determined by Gouys 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 tube used were made of soda glass. Particular care was taken to insure uniform diameter. These were provided with ground glass stoppers to which was attached 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 the bottom of the pans of the balance by means of an adjustable crew device. Thus, the length of the suspension could be adjusted. The pole pieces were fixed at a distance of 1.8 cm and all measurements were made with 5 amperes 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 coincides with the centre. The bottom of the suspension tube was, therefore, always in the region of maximum field even under oscillation during weighing. The field was found to be practically negligible at 11.6 cm above this point. The specimen tube was, therefore, always field upto height off about 11.6 cm. Filling up of the specimen tube requires some skill. Generally, the substance was well powered in a agate mortar. A small amount of substance was then introduced into the tube and rammed with a properly fitting glass rod. In this way the required length was filled in uniformly.

IX. DETERMINATION OF MAXIMUM FIELD STRENGTH

The field was determined by using a number of standard substance 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.

$$H_{\max}^2 = \frac{2hwg}{X_g m} = \frac{2l(\text{in cm}) \times w(\text{in gms}) \times 981}{X_g \times m(\text{in gm})}$$

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

$$X_g = \frac{2l \times w \times 10^{-3} \times 981}{H_{\max}^2 \times m} \\ = \frac{2l \times w \times 10^{-3}}{H_{\max}^2 \times m \times 1.019}$$

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

$$\frac{2l \times w}{H_{\max}^2}$$

is calculated and then the expression applied for different samples.

The magnetic moment μ (in B.M.) is taken calculated using the expression

$$\mu = 2.84 \sqrt{X_m^{corr.} T}$$

where $X_m = X_g \times M$ (M = molecular weight of the sample and $X_m^{corr.}$ is the corrected value of the susceptibility when correction is made for diamagnetism, using Pascal's constants.

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