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Synthesis and characterization of Bis - (2, 4 pentanedione dihydrazone) nickel(II) chloride hexahydrate

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Abstract

The complex Bis-(2,4-pentanedione dihydrazone) copper(II)chloride hexahydrate was synthesized "insitu" and characterized by infrared spectroscopy, conductivity measurement, decomposition temperature and elemental analyses. Present method of continuous variation shows 1:2 metal to ligand ratio. The solubility of the complex in different solvents such as ethanol, methanol, dimethylsulphoxide (DMSO) and ether was also established.

Keywords: Dihydrazone, acetylacetone, ligand, coordination

Introduction

The ligand, 2, 4 – pentanedione dihydrazone is a schiff base, a chemical compound containing azomethine group, -R-C==N-, usually obtained by the condensation of acetylacetone with hydrazine hydrate. It has been known that a variety of metal ions on interaction with schiff bases yield chelates. Schiff bases are used as, rubber accelerators, dyes, reaction intermediates and also as liquid crystals in electronic display systems. Transition metal schiff base complexes are applicable in areas such as electrochemistry, catalysis, bioinorganic, metallic deactivators, separation technique and environmental chemistry. This paper reports the synthesis and characterization of bis-(2,4-pentanedione dihydrazone) nickel (II) chloride hexahydrate complex.

Method of prepareation

Preparation of Bis (2, 4-pentanedione dihydrazone) nickel (II) chloride hexahydrate Complex Ethanolic solution of nickel (II) Chloride dehydrate (0.05 moles) was added, drop wise with constant stirring, into a mixture of 10cm^3 of acetyl acetone and 20cm^3 ethanol in a 250cm^3 conical flask. The resulting mixture was heated to boiling and 10cm^3 of hydrazine hydrate was added drop wise with constant stirring, before being refluxed over water bath for 2 hours. The precipitate that formed was cooled, filtered, washed successively (with ethanol and diethyl ether) and dried at room temperature to constant weight within 4 days.

Results and discussion

The ligand was prepared as reported by Williams *et al*. (1989), it is in the form of pale yellow crystals with 77.3% yield (Table 1). The prepared complex is a green crystalline solid with 52.12% yield (Table 1). The decomposition temperature (Table 1) of nickel (II) Schiff base complex is 173ºC, which presents a fairly stable complex compound. The value is in agreement with the stability reported for the first row transition metal complex compounds. The mean percentages of Cl^-, Ni^{+2}, water of crystallization, the ligand and their ratios in the complex were 13.41 and 13.72, 23.34 and 49.65%, respectively, which support $[Ni(PDH)_2]Cl_2.6H_2O$ composition for the complex 1:1 metal to ligand ratio. The complex is insoluble in water and common organic solvents, but is readily soluble in dimethylsulphoxide (DMSO), ethanol, methanol and ether. The molar conductance value (Table 2) of the nickel (II) schiff base complex in 1 x 10^{-3}M ethanol solution is 71 ohm^{-1}cm^2 mol^{-1}, suggesting a 1:2 electrolytic type. The IR spectrum (Table 3) of the ligand shows a very sharp band in the region 1625 – 1640cm^{-1} attributable to ν(C==N) vibration mode, Which shifted in the complex compound to the range 1590 – 1620cm^{-1}. This negative shift suggests coordination of the imine group (C==N) to the metal. The ligand also shows broad doublets around 3300 and 3200cm^{-1}, assignable to ν(N—H) mode of primary and secondary amine groups. A broad band appears around 3200 – 3400 cm^{-1} in the complex compound that provides evidence for the presence of water of crystallization. The far IR spectrum of the complex compound shows a strong band at 340cm^{-1}, assignable to ν (M—N) vibration mode. The average number of ligands that were coordinated to nickel (II) ion at varying mole fractions determined from a plot of absorbance versus mole fractions of the schiff base, revealed a 1:1 complex. From the analytical results of the complex compound, the structure below (Fig. 1) is proposed;

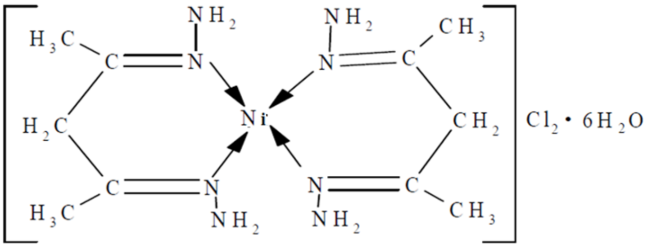

Fig 1: The Proposed Structure of the Comolex compound prepared.

Table 1: Some properties of the Ligand and the Complex compounds

Compound	Colour	Yield (%)	Decomposition Temp (ºC)
Ligand	Yellow	77.3	-
$[Ni(PDH)_2]Cl_2.6H_2O$	Green	52.12	173

Table 2: Conductivity Measurement of the Complex in 1x10^{-3}M Ethanol

Compound	Specific Conductance(K)	Molar Conductance (Ohm^{-1}cm^{2}mol^{-1})
$[Ni(PDH)_2]Cl_2.6H_2O$	7.1x10^{-5}	71.0

Table 3: Infrared data for the Ligand and the Complex Compounds

Compound	ν(C==N) cm^{-1}	ν (N—H) cm^{-1}	ν (O—H) cm^{-1}	ν (M—N) cm^{-1}
Ligand	1625-1640	3200, 3300	-	-
$[Ni(PDH)_2]Cl_2.6H_2O$	1590-1620	3200, 3300	3300-3400	340

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