



Research Article

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Synthesis and Structural Investigation of Nickel Metal- Ligand (Thiourea Derivative) Complexes

M. S. Rathod and S. Z. Jadhao

Department of Chemistry, Institute of Science, Nagpur, India

ABSTRACT

Studies of the complexes of metal ion with thiourea derivatives. Complexes has been carried out on the basis of elemental analysis molar conductance, IR, NMR, electronic spectra etc. Thiourea derivatives used as ligand and their Ni²⁺. Complexes have been synthesized (Diethyl N Benzoyl thiourea derivative).

Keywords : Spectral Ni²⁺ complexes Diethyl N Benzoyl thiourea derivative.

INTRODUCTION

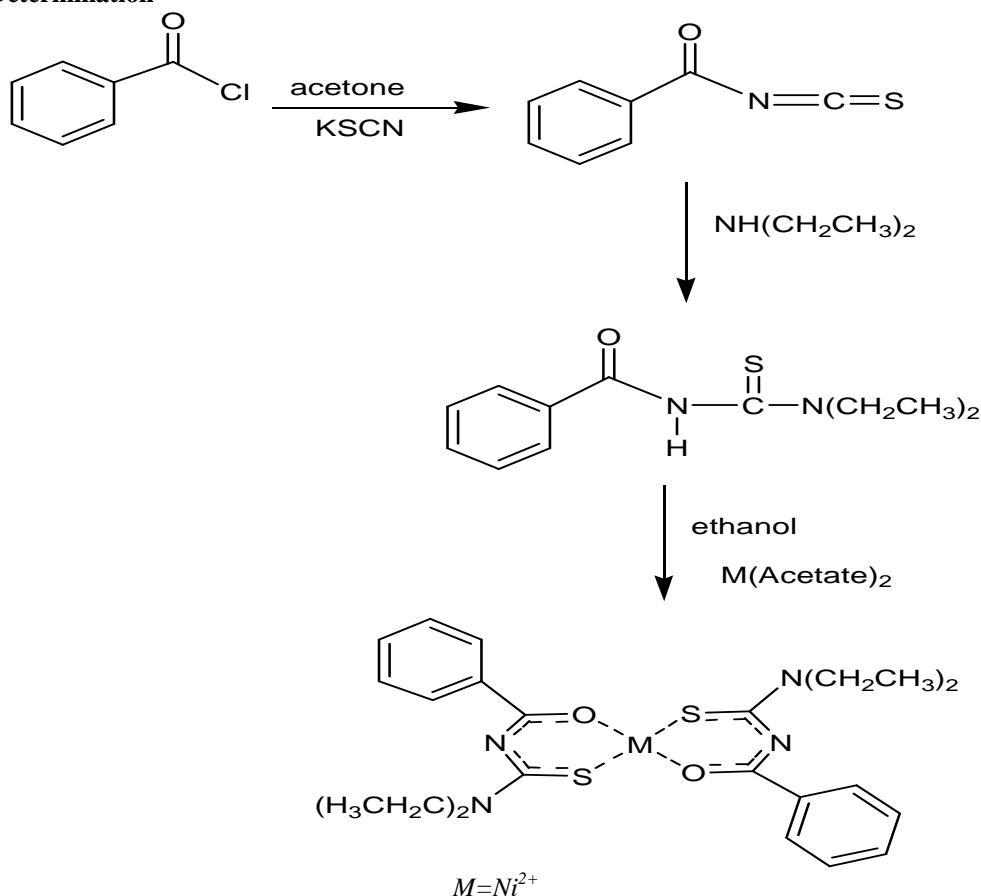
Industrial production and the use of Fe, Co, Ni, Ag, Pb elements can cause environmental pollutions. On the other hand, some of these metals are present in trace amount as essential elements for the biological systems and these metal ions also play an important role in bio-inorganic chemistry. In order to understand the role of these metal ions in biological systems, structural studies of the biological compounds and their metal complexes are extremely important. Compound containing carbonyl and thiocarbonyl groups occupy an important position among organic reagents as potential donor ligands for transition metal ions, among these thio derivatives are potentially very versatile ligands, able to coordinate to range of metal centre as a neutral ligands, mono ions or dianions. The oxygen, nitrogen and sulphur donor atoms of thiourea derivative provide multitude of bonding possibilities. Both the ligand and their metal complexes display a wide range of biological activities[6].

In view of this, we focused on synthesis, characterization of complexes on the basis of electronicspectra [19] analysis, magnetic measurements and spectral analysis. Infra red spectra of complexes indicate that bonding through, oxygen and sulphur

RESULTS AND DISCUSSION

Synthesis:

The ligands were prepared by procedure to that reported in the literature [1].A solution of benzoyl chloride (0.05 mole) in acetone (30 mL) was added dropwise to a suspension of potassium thiocyanate (0.05 mole) in acetone (30 mL). The reaction mixture was heated(30 °C) under reflux for 30 min., and then cooled to room temp.A solution of secondary amine (0.05 mole) in acetone (30 mL) was added and the resulting mixture was stirred for 2 hour, hydrochloric acid (0.1N , 100 mL) was added and the solution is filtered. The solid product was washed with water and purified by recrystallisation from an ethanol– dichloromethanol mixture (1:2). Metallic complexes were prepared according to the method described in the literature [2]A solution of metallic acetate (0.05 mole) in ethanol (30 mL) was added dropwise to a solution of the ligand in ethanol (30 mL), 1:2 ratio for all metal with a small excess of ligand at room temp. And the resulting mixture was stirred 30 min. The solid complexes were filtered and re-crystallized from a 1:2 ethanol– dichloromethane mixture[7].

Structure Determination

The molar conductance values for the complexes in DMF and DMFSDO at 10^{-3} m dilution are in the range of 3.2 -6.8 $\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$ [8], suggesting non electronic nature of these complexes. Ligand has four co-ordinating sites[3]. The result showed that two ligand and molecules might co-ordinate to the metal ion[13]. In order to identify the structure of complexes, IR, NMR, Spectra were also investigated[4].

IR spectra of the ligands showed strong band at $1715\text{-}1730 \text{ cm}^{-1}$, which is the characteristic peak of carbonyl group(C=O).while in complexes this band shifted to the region $1650\text{-}1670 \text{ cm}^{-1}$, indicating the co-ordination of carbonyl oxygen atom of ligand with metal ion. Meanwhile strong band at $820\text{-}840 \text{ cm}^{-1}$ in the free ligand, which was assigned to the C=S gp, significantly disappeared in the complexes, indicating that sulphur, atom contributed to the formation of the complexes[17].

The spectra of complexes showed only the peak at δ 11.73 ppm for NH, while peak at δ 12.81 ppm for NH between C=S, C=O group disappeared, indicating the removal of this proton and formation of C=N group. Aromatic skeleton vibration at 1425 cm^{-1} [14]. C-O stretching 1307 cm^{-1} [15]. Symmetrical six member structure [16].

Aromatic C-H, $3060(\text{w})$ Aromatic C-H, $710(\text{m})$ Aromatic skeleton vibration, $1425(\text{w})$ C-N, $1425(\text{M})$, C=N $1610(\text{m})$, C-O $1307(\text{m})$, C=S $2012(\text{m})$, M-O $542(\text{m})$ t(CH₃), 1.2, q(CH₂), 1.9

Electronic spectral data of metal complex

Complex	Absorption spectra	Wave no (k k)
Ni (diethyl thiourea deriva.) ₂	206	37.99
	232	36.67
	233	42.80

A Broad band for Ni 36.67 KK in Ni (diphenyl thiourea derivative)₂[12] Suggest Tetrahedral geometry[9,10]. All the complexes shows group of three more bands in the 45.44 KK range which can be assigned as $\pi\text{-}\pi^*$ ligand-Metal transition[19].

Fundamental infrared bands (cm⁻¹) of metal complex

Assignment	Ligand (in cm ⁻¹)	ML2(in cm ⁻¹)
Aromatic C-H	3060cm ⁻¹	3064cm ⁻¹
Aromatic (monosubstituted benzene)	690cm ⁻¹ -750 cm ⁻¹	710cm ⁻¹
Aromatic skeletal vibration	1425cm ⁻¹	1445cm ⁻¹
C=N stretching	1610cm ⁻¹	-
Amide (N-H stretching)	3425cm ⁻¹	-
C=O Amide	1670-1725cm ⁻¹	-
C-N stretching	1425cm ⁻¹	1430cm ⁻¹
C=S(asymmetric stretching)	1350cm ⁻¹	-
C-H Stretching	2950cm ⁻¹ -3000cm ⁻¹	3100cm ⁻¹
C-O Stretching	1307cm ⁻¹	1300cm ⁻¹
M-O stretching	-	542cm ⁻¹

NMR Spectra for metal ligand complex

Assignment	Ligand	ML2
Benzene (m)	δ 8.5 ppm	δ 7.2 ppm
N-H(amide proton)	δ 1-5 ppm	-
-CH ₂ -(t)	δ 1.3 ppm	δ 1.2 ppm
-CH ₃ (q)	δ 0.9 ppm	δ 1.2 ppm

CONCLUSION

Above the N.M.R ,I.R data shows that successfully synthesized Nickel Metal Ligand (Thiourea Derivative) Complexes

This study has been of preliminary nature with the objective of establishing the N.M.R,I.R electronic spectra. We are understanding of extension study that is application of these complex ,work is in progress

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