

SYNTHESIS, CHARACTERISATION AND BIOLOGICAL STUDIES ON Ni(II) QUINOLINE SCHIFF BASE COMPLEXES

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ABSTRACT

Schiff base play an important role in inorganic chemistry as they easily form stable complexes with most transition metal ions. *The synthesis and characterization of transition metal complexes containing Schiff bases as ligands due to their application as catalyst in many reactions and related to synthetic organic and natural oxygen carriers. Molecules containing donor-acceptors such as Schiff bases have ability to serve as polymeric ultraviolet stabilizers, laser dyes .The present work is focused on the study of co-ordination behaviour of thiosemicarbazone Schiffbase with hydrated Ni(II) chlorides. Analytical and spectral data confirmed the structure of the complexes. Also absorption at 305nm might be due to the extended conjugation of the ring or may be due to the ring residue. Antibacterial and Antifungal activities were carried out using Disc diffusion method and the compounds were found to be active.*

Keywords

Schiff base complexes, Antibacterial activity, Antifungal activity

INTRODUCTION

Coordination compounds have found application in medicine in the treatment and diagnosis of diseases **Fenton (1995)**. Among the transition metals iron, cobalt, nickel and copper complexes are extensively studied because of their application towards novel biological

properties **Crichton (1991)**. Transition metal complexes acts as a homogeneous catalyst in many industrially important reactions such as hydrogenation, hydrosilation, hydroformylation, polymerization, isomerisation, acylation and oxidative hydrolysis of olefins and related to synthetic organic and natural oxygen carriers **Mutterliuer (1975)** and **Brink - Shoemaker et.al (1964)**. Bimetallic coordination complexes may serve as model for variety of biological reactions such as oxygen transport, oxygen activation, photosynthetic water reduction, the study of electron transfer process, metal-metal interactional multi centered catalysis **Lingappa etal (1996)**. Finely divided Nickel is used as a catalyst in the hydro generation of oils and fats **Mowton (1980)**. In coenzyme B¹² cobalt is bound to a tetraazamacrocyclic ligand **Nishiya etal(1986)**. In addition to the varied magnetic property and catalytic activities, the transition metal Schiff base complexes can also serve as efficient models for metalloproteins and enzymes. Molecules containing donor-acceptors such as Schiff bases have ability to serve as their implication in biology **Christou etal(1979)**. Many enzymes, the naturally occurring catalysts that regulate biological processes, are metal complexes

EXPERIMENTAL WORK

All the chemicals used were of analar grade. The solvents used were neat and dried. The TLC Plates were prepared by using silica gel G. Petroleum ether, Ethyl acetate and ethanol were used as irrigants. Microwave oven was used for the irradiation of reactions.

PREPARATION OF LIGANDS:

Preparation of 2-Hydroxy-7-Methyl-quinoline-4-Carboxy Thiosemicarbazone (1):

When equal moles of 7-methyl-quinoline-3-carbonyl chloride and thiosemicarbazone in ethanol was refluxed for 18-22 hours on irradiation give 2-Hydroxy-7-methyl-quinoline-4-carboxy thiosemicarbazone. The completion of the reaction confirmed by thin layer chromatography and the reaction mixture, washed with Petroleum ether and dried.

Melting Point: 171°C.

Yield: 2.2g (47%).

Preparation of 7-Methyl -2- Hydroxy- Quinoline-4-Carboxy Thiosemicarbazone-Schiff base (2):

An ethanolic solution of 0.500g , 7-methyl quinoline thiosemicarbazone was irradiated for 40 seconds with 0.3ml of benzaldehyde which give **7-methyl 2- hydroxy quinoline-4-carboxy thiosemicarbazone-schiff base** the excess solvent was evaporated, washed and recrystallised from ethyl acetate.

Melting Point :138°C.

Yield :1.4. g (39%).

Preparation of Nickel complex (3):

An ethanolic solution of 0.0788 g of nickel chloride was slowly added to the quinoline semicarbazone Schiff base (2) and the mixture was irradiated for 30 seconds. The reaction mixtures were allowed to stand for 2 days at room temperature. A dark green colored needle was obtained.

Appearance: Needles

Color: Dark green

Melting point:216°C.

RESULTS AND DISCUSSION

The present work is focused on the study of co-ordination behavior of thiosemicarbazone Schiffbase with hydrated Ni(II) chlorides.

The complex thiosemicarbazone Schiff base was obtained via 7-methyl quinoline-4-carboxy-thiosemicarbazone synthesized by refluxing equal moles of 7-methylquinoline -4-carbonylchloride and thiosemicarbazide in ethanol for 18-22 hrs. The 7-methyl quinoline -4-carboxythiosemicarbazone and benzaldehyde in equal moles were refluxed in ethanol for 18hrs at 80°C.

IR spectrum of the **compound (1)** showed absorption peaks at 1695cm^{-1} (CO), 1680 cm^{-1} (CO), 1635 cm^{-1} (CN of quinoline ring), 1626 cm^{-1} (CH=N,) 1214 cm^{-1} (C=S) **fig(1)**. UV spectrum of the compound showed absorption at 290nm, 281nm, 237nm **fig(2)**. .

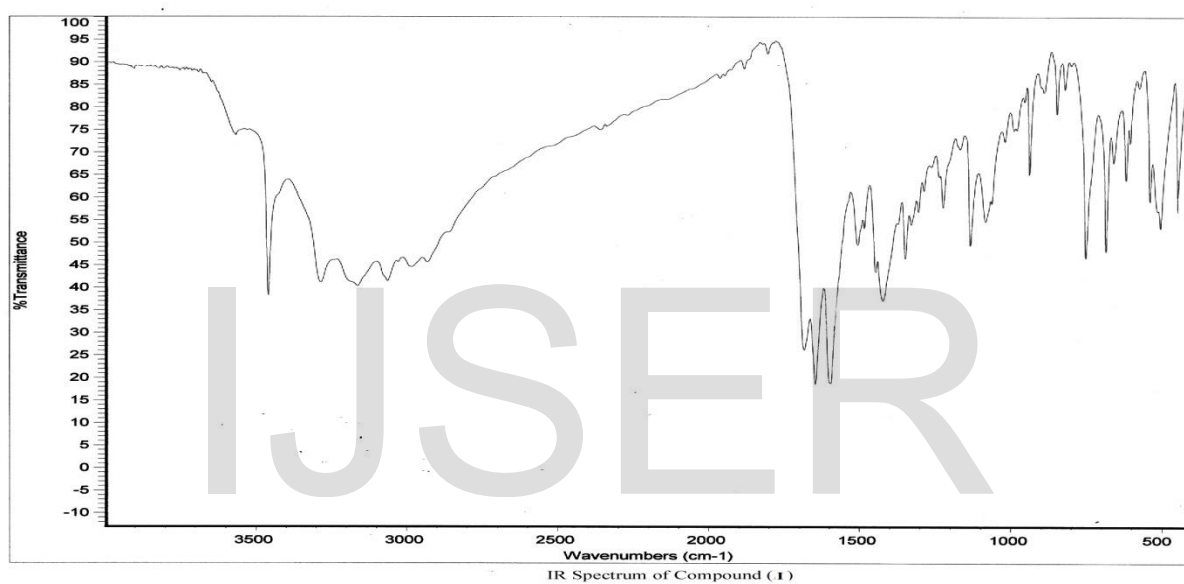


FIGURE (1)

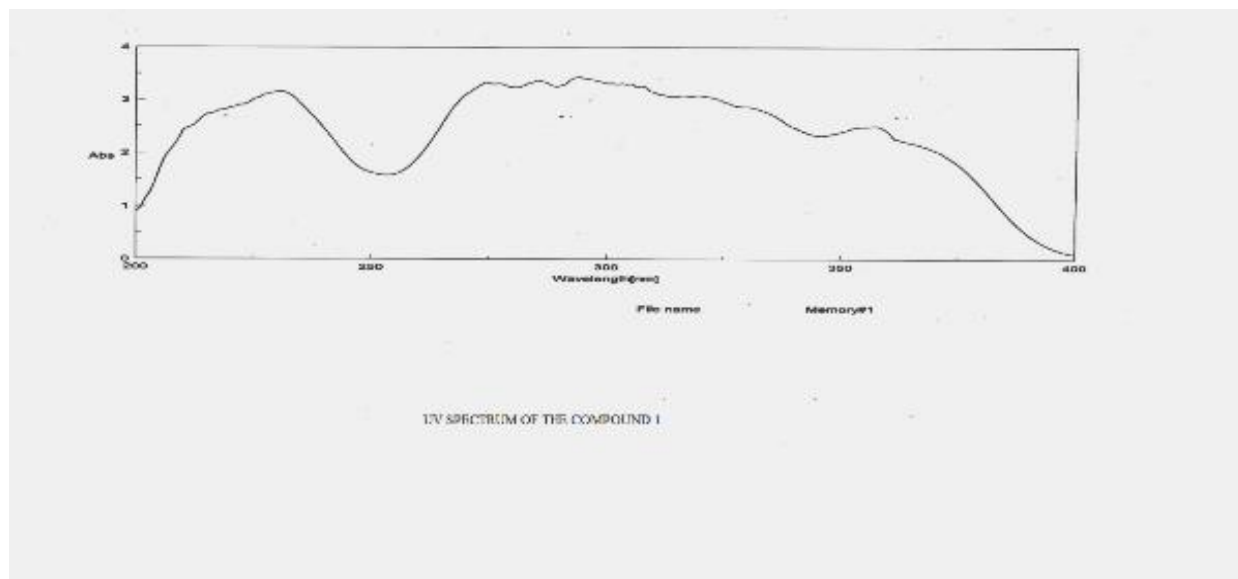


FIGURE (2)

The 7-methyl 2 hydroxy quinoline-4-carboxy thiosemicarbazone-schiff base was then subjected to salt to coordinate with Nickel form the nickel complex.

To a hot ethanolic solution of 0.500g(0.00031mol) of Schiff base(2) and 0.0756 g (0.0031mol) of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ in ethanol was added and irradiated in the microwave oven for 50 seconds a dark green colored needle was obtained.

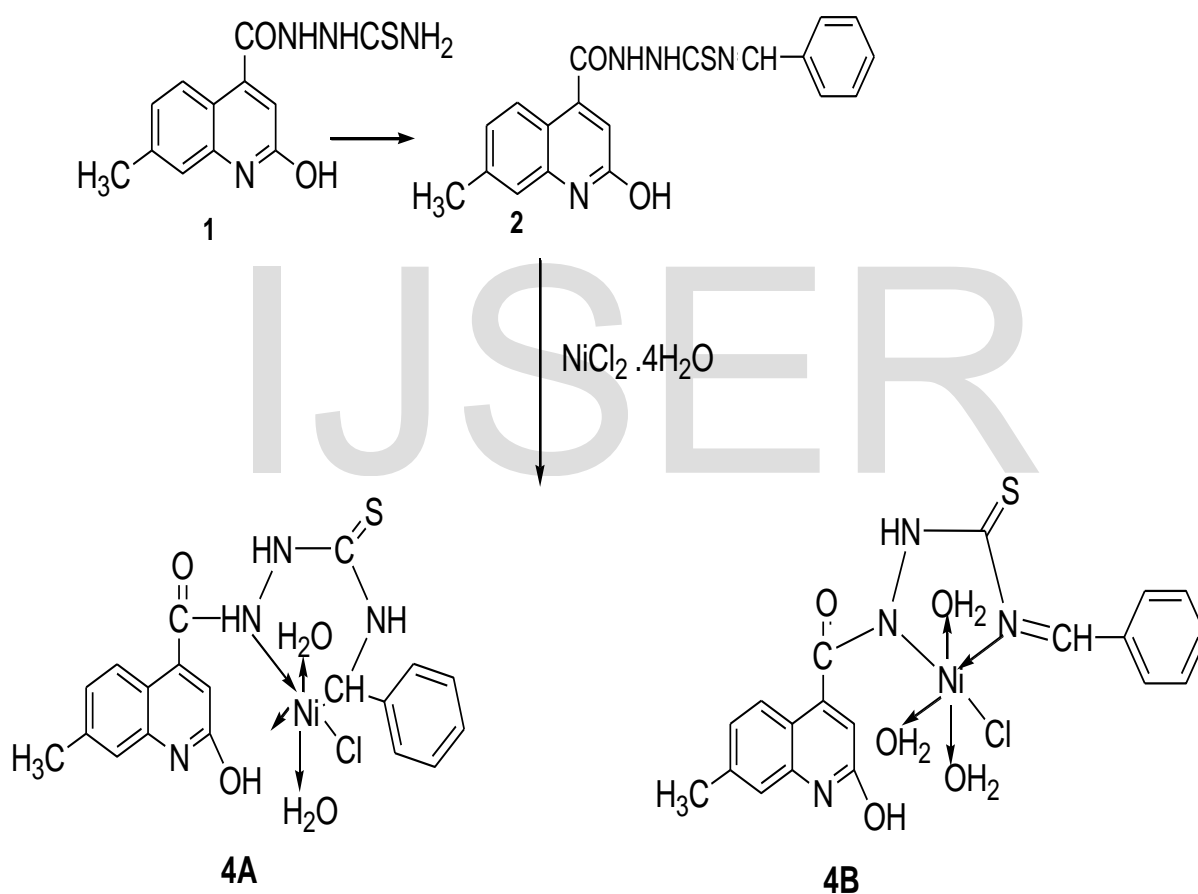
IR spectrum of the compound **2-Hydroxy-7-methyl-quinoline-4-carboxy thiosemicarbazone** showed absorption peaks at 1715 cm^{-1} (CO), 1635 cm^{-1} (CN of quinoline ring); 1611 cm^{-1} (CH=N); 3298 cm^{-1} ($\text{NH}=\text{C}=\text{S} \longleftrightarrow \text{N}=\text{C}-\text{SH}$); 1217 cm^{-1} and 781 cm^{-1} ($-\text{C}=\text{S} \longleftrightarrow \text{C}-\text{SH}$). **fig(3).**

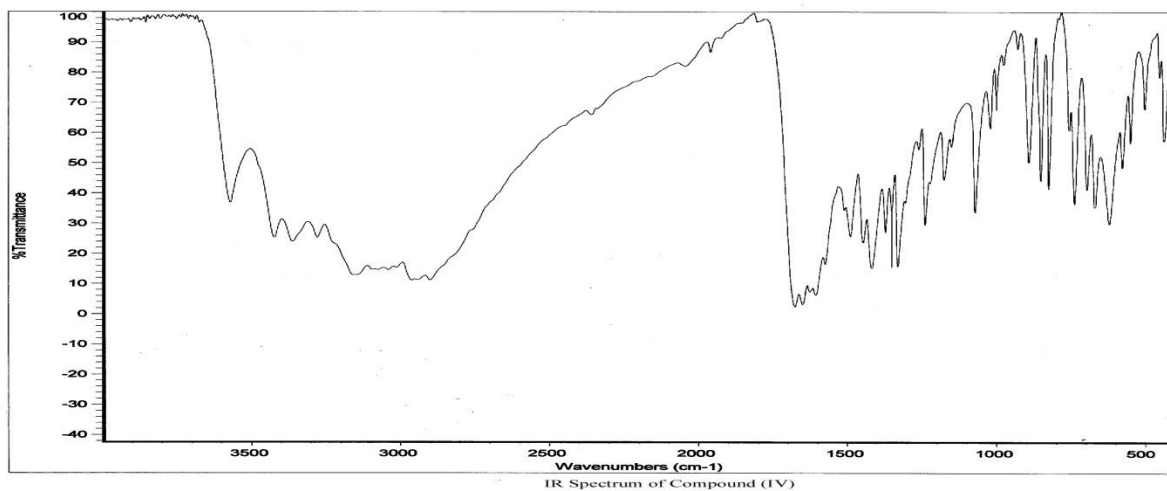
UV spectrum of the compound showed absorption at 305nm, 290nm, 281nm, 237nm Presence of absorption max at 305nm **fig(4).**

The tautomerism in the IR spectrum showed the $\text{NH}=\text{C}$ moiety is not disturbed during coordination.

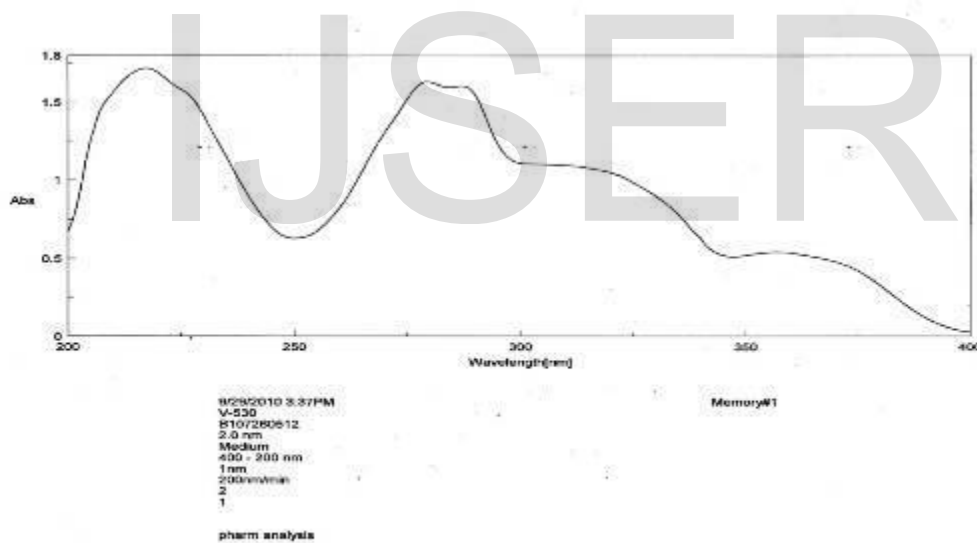
Absorption at 305nm might be due to the extended conjugation of the ring or may be due to the ring residue. This shows that the the coordination is formed between the lone pair of nitrogen and hence a 5-membered -6-coordination was confirmed.

From the spectral and analytical data the structure of the compound formed was confirmed to be 4B and not 4A.





FIGURE(3)



FIGURE(4)

BIOLOGICAL ACTIVITY

The disc diffusion method uses filter paper discs, 6.0mm in diameter, charged with appropriate concentrations of the drugs. The disc are stored dry in cold. A suitable dilution of a broth culture or a broth suspension of the test bacterium is flooded on the surface of a solid medium (Mueller-Hinton agar). Compounds **1**, **2** and **4B** were tested against the bacterias E.Coli and staphylococcus albus and the fungi candida species and Aspergillus niger at various concentrations 100µg/L, 50µg/L, 25µg/L.

ANTIBACTERIAL & ANTIFUNGAL ACTIVITIES :

Antibacterial and antifungal activities were carried by Kirby-Bauer Method (Disc diffusion method). The media for antibacterial study is Muller Hintan Agar(MHA). The media for antifungal study is Sabouraul Dextrose Agar(SDA). The standard used was Gentamycin for antibacterial studies. The standard used for antifungal studies was Ketocandizole.

Table-1 shows the antibacterial activity of the compounds 1, 2 and 4B against E.coli. It was found that all the compounds were active. Among the four compounds 3A and 4B are active than their precursors.

Table-2 shows the the antibacterial activity of the compounds 1, 2 and 4B against Staphylococcus albus . It was found that all the compounds were active. Among the four compounds 3A and 4B are active than their precursors.

The antibacterial activity of the compound was almost closer to the standard gentamycin.

Table-3 shows the antifungal activity of the compounds 1, 2 and 4B against Candida species . It was found that all the compounds were active. Among the four compounds 3A and 4B are active than their precursors.

Table-4 shows the antifungal activity of the compounds 1, 2 and 4B against Aspergillus niger. It was found that all the compounds were active. Among the four compounds 3A and 4B are active than their precursors.

The Fungi *Candida* species was found to be active than *Aspergillus niger*. *Aspergillus niger* shows only moderate activity

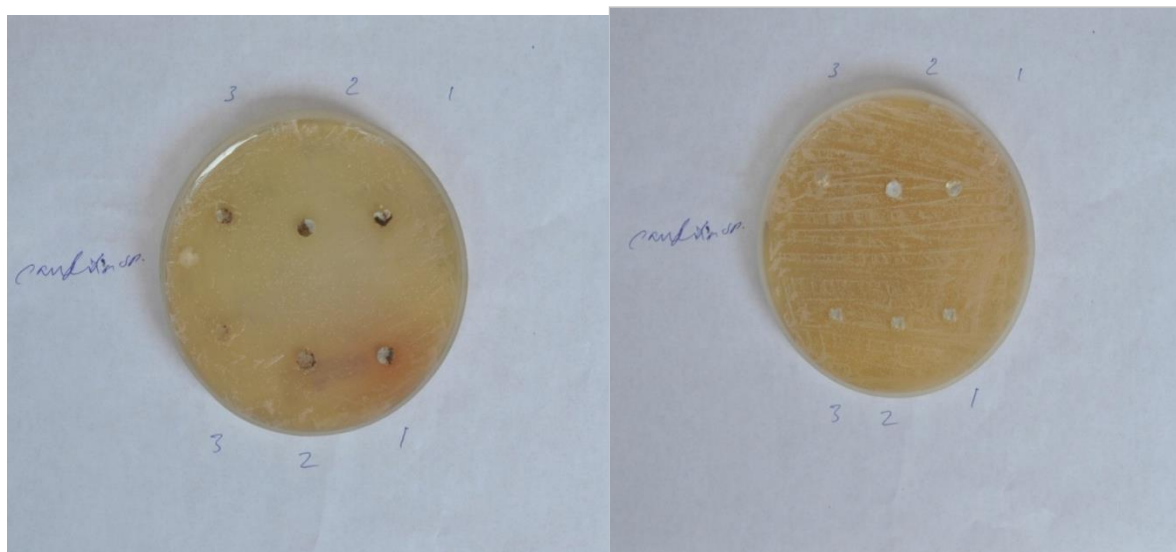
The antifungal activity of the compound was almost closer to the standard Ketocandazole.

Table-1 Antibacterial Activity

Samples	Concentrations (100µg/l) (50µg/L) (25µg/L) in mm			Concentrations (100µg/l) (50µg/L) (25µg/L) in mm		
	E.Coli			Staphylococcus albus		
Compound 4A	7	5	4	7	4	2
Compound 4B	7	3	2	8	6	2
Gentamycin	10	8	8	12	10	8



Samples	Concentrations (100µg/l) (50µg/L) (25µg/L) in mm			Concentrations (100µg/l) (50µg/L) (25µg/L) in mm		
	Candida species			Aspergillums niger		
Compound 4A	5	3	3	6	2	2
Compound 4B	8	6	2	6	2	2
Ketocandizale	10	8	8	8	6	6



CONCLUSION

Co-ordination behavior of Ni(II) complex and the antibacterial activity and antifungal activity were studied in the present work. The acid chloride(1) was reacted with thiosemicarbazide at 80°C for 16-22 hours and to obtained thiosemicarbazone(2). Equal moles thiosemicarbazone(2) and $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ were irradiated under microwave oven to obtain the Ni complex(4B). Analytical and spectral data confirmed the structure of the complex as 4B. Antibacterial and Antifungal activity were carried out using Disc diffusion method and the compounds were found to be active.

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