

Synthesis and Electrical Conductivity of Mn(III), Fe(III), VO(IV), Zr(IV) and UO₂(IV) Complexes derived from thiazole Schiff base.

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ABSTRACT

A newly thiazole Schiff base has been prepared by the condensation of 2-hydroxy-5-chloro-3-nitroacetophenone and thiazole. The ligand was characterized by elemental analysis and spectral methods. The coordinating ability of the ligand is investigated by preparing its metal complexes with, Mn(III), Fe(III), VO(IV), Zr(IV) and UO₂(VI) have been prepared and characterized by elemental analysis, molecular weight determinations, conductance measurements, spectral and thermal studies.. The isolated products are coloured solids, soluble in DMF, DMSO and THF. All the complexes have been studied and evaluated by electrical conductivity

Keywords: Schiff base, Magnetic susceptibility, Electrical conductivity.

INTRODUCTION

The electrical conductivity of metal complexes varies with their nature and temperature. The variation of electrical conductivity of the metal complexes with temperature is the basis of their classification as semiconductors or metallic conductors. The electrical conductivity of metallic conductors decreases with increasing temperature (i.e. temperature coefficient is negative) and their resistivity ranges from 10⁻⁶ to 10⁻³. Thermal Stability and Bacterial Activity of Schiff Bases Derived From Selective Amino Acid and Their Complexes.[1]

The present Research paper focus on synthesis, characterisation and various methods of Schiff base derived from sulphanilic acid and salicylaldehyde and Comparative study of Schiff base using various synthesis methods and their theoretical prediction of activities.[2] The electrical conductivity of metal complexes varies with their nature and temperature. The variation of electrical conductivity of the metal complexes with temperature is the basis of their classification as semiconductors or metallic conductors. They are reported the possible use of such systems in biological applications for their antifungal properties and antioxidant activities.[3] The Schiff base prepared by using variety of aldehydes and amines or any other amines possessed antitubercular, antitumor, anticancer, fungicidal medicinal and agrochemical activities. Schiff base and their metal complexes are becoming increasingly important in recent years due to their biological activity and their used as catalysts photoluminescent, electro-luminescent properties Antimicrobial screening, biological great significance of Schiff base metal complexes research and play a significant role in the area of coordination chemistry. Antimicrobial evaluation of 2-amino pyridine-derived Ligand Schiff base and its complexes with Cu (II), Hg (II), Ni (II), Mn (II) and Co (II).[4] Synthesis and Characterisation of new Heterocyclic Schiff base ligand derived from 4-Amino Antipyrine.[5]

METHODOLOGY

All the chemicals were of A.R. grade and used as received. 2-hydroxy-5-chloro-3-nitro acetophenone (HCNA) and 4-(p-hydroxyphenyl)-2 amino thiazole was prepared by known methods [6-9]. The solvents were purified by standard methods.[10]

Synthesis of 2-hydroxy-5-chloro-3-nitro acetophenone 4-(p-hydroxyphenyl)-2 imino thiazole [HCNAT]: A solution of 4-(p-hydroxyphenyl)-2 imino thiazole (0.02M) in 25ml of ethanol was added to an ethanolic solution(25ml) of 2-hydroxy-5-chloro-3-nitro acetophenone (0.02M) and the reaction mixture was refluxed on a water bath for 4h. After cooling a pale yellow coloured crystalline solid was separated out. It was filtered and washed with ethanol, crystallized

from DMF and dried under reduced pressure at ambient temperature. The purity of ligand was checked by elemental analysis and m.p. It was also characterized by IR and ^1H NMR spectral studies. Yield:70%; m.p. 310⁰C

Preparation of complexes:

All the metal complexes were prepared in a similar way by following method. To a hot solution of ligand HCNAT (0.02M) in 25ml of ethanol a suspension of respective metal salts was added drop wise with constant stirring. The reaction mixture was refluxed on a water bath for 4-5 h. The precipitated complexes were filtered, washed with ethanol followed by ether and dried over fused calcium chloride. Yield : 50-55%

The complexes are soluble in DMSO and DMF but insoluble in water and common organic solvents. The metal chloride content of complexes were analyzed by standard methods.[12] The ^1H NMR spectra of ligand was recorded and obtained from RSIC Chandigarh. IR spectra of the compounds were recorded on Perkin Elmer 842 spectrophotometer in the region 400-4000 cm^{-1} , Carbon, Hydrogen and Nitrogen analysis were carried out at RSIC, Punjab University, Chandigarh. The molar conductance of the complexes at 10^{-3} M dilution in DMF were determined using equiptronic digital conductivity meter EQ-660 with a cell constant 1.00 cm^{-1} at room temperature. The magnetic moment measurement were made on a Gouy balance at room temperature using $[\text{HgCo}(\text{SCN})_4]$ as the calibrant. The thermogravimetric analysis were performed on laboratory set up apparatus in air atmosphere at 10^0 C min^{-1} heating rate. The molecular weights of the complexes were determined by Rast method.

RESULTS AND DISCUSSION

The Schiff base HCNAT and its complexes have been characterized on the basis of ^1H NMR, IR spectral data, elemental analysis, molar conductance, magnetic susceptibility measurements and thermo gravimetric analysis data. All these values and analytical data is consistent with proposed molecular formula of legend. All the compounds are coloured solid and stable in air. They are insoluble in water but soluble in

coordinating solvents like DMF and DMSO. The molar conductance values in DMF (10^{-3} M) solution at room temperature (Table2) shows all the complexes are non-electrolytes. The ^1H NMR spectra of ligand HCNAT shows signals at δ 12.11, (1H, s phenolic OH), δ 9.52 (1H, s, phenolic OH), δ 7.56, 7.54, 7.53 and 7.52

(4H, m, phenyl) δ 6.81, 6.80, and 6.78(3H, s Phenyl), 6.68 (1H s thiophene), and 2.56(3H, s, methyl). [11,13-15] IR spectra of ligand and metal complexes shows $\nu(\text{C}=\text{N})$ peaks at 1620 cm^{-1} and absence of $\text{C}=\text{O}$ peak at around $1700 - 1750\text{ cm}^{-1}$ indicates the Schiff base formation.[16-19]

Table1. Analytical data of the Ligand.

Ligand	Molecular Formula	Formula Weight	Color and nature	Elemental Analysis			
				C% found (Cal.)	H% Found (Cal.)	Cl% Found (Cal.)	S% Found (Cal.)
HCNAT	$\text{C}_{17}\text{H}_{13}\text{N}_3\text{O}_4\text{S}$	390.6	Yellow Crystalline	52.34 (52.22)	03.26 (03.32)	9.02 (9.08)	08.12 (08.21)

Table 2. Analytical data and molar conductance of the compounds.

Ligand	Formula weight g mole^{-1}	Colour	Elemental Analysis Found(Calcd.)				μ_{eff} B.M	Λ_{M} ($\Omega^{-1}\text{ cm}^2\text{ mol}^{-1}$)
			M%	C%	H%	Cl%		
$[\text{MnL}_2(\text{OAc})] \text{H}_2\text{O}$	929.1	Brown	5.40 (5.90)	46.15 (46.49)	3.16 (3.33)	7.32 (7.64)	4.6	18.4
$[\text{FeL}_2(\text{H}_2\text{O})\text{Cl}] \text{H}_2\text{O}$	906.6	Black	6.02 (6.16)	44.81 (45.00)	3.02 (3.08)	11.41 (11.74)	5.2	22.2
$[\text{VOL}_2]$	846.2	Green	5.63 (6.02)	48.01 (48.21)	2.15 (2.83)	8.32 (8.39)	1.4	12.4
$[\text{ZrL}_2(\text{OH})_2] 2\text{H}_2\text{O}$	940.4	Yellow	9.48 (9.69)	43.13 (43.38)	3.06 (3.19)	7.26 (7.54)	Dia	16.2
$[\text{UO}_2\text{L}_2]$	1049.3	Orange	22.43 (22.69)	38.51 (38.88)	2.11 (2.28)	6.32 (6.76)	Dia	14.2

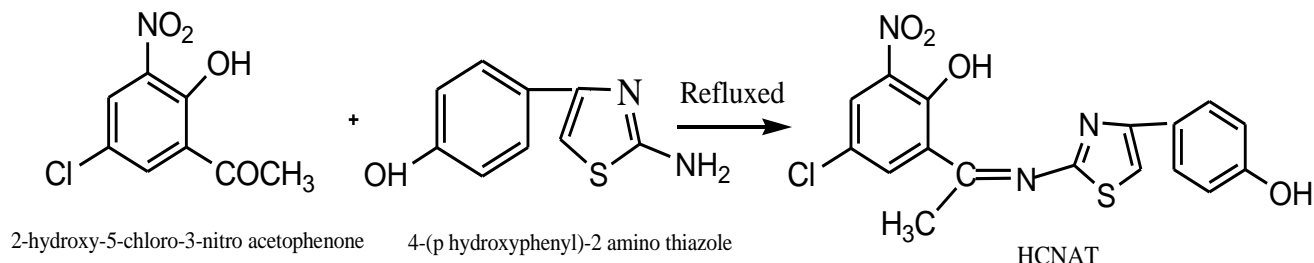
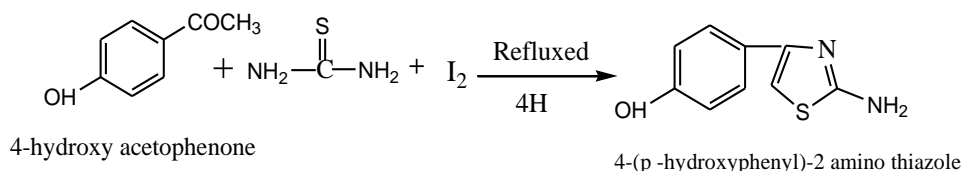
Table 3. IR spectra of ligand and metal complexes

Compound	$\nu(\text{O}-\text{H})$ hydrogen bonded	$\nu(\text{C}=\text{N})$ imine	$\nu(\text{C}=\text{O})$ phenolic	$\nu(\text{M}-\text{O})$	$\nu(\text{M}-\text{N})$	$\nu(\text{C}-\text{S})$
HCNAT (LH)	3119	1620	1514	--	--	1122
$[\text{MnL}_2(\text{OAc})] 2\text{H}_2\text{O}$	--	1562	1461	496	422	1090
$[\text{FeL}_2(\text{H}_2\text{O})\text{Cl}] \text{H}_2\text{O}$	--	1602	1502	510	441	1080
$[\text{VOL}_2]$	--	1598	1505	512	446	1098
$[\text{ZrL}_2(\text{OH})_2] 2\text{H}_2\text{O}$	--	1600	1496	446	414	1108
$[\text{UO}_2\text{L}_2]$	--	1585	1440	548	480	1082

Table 4. Electrical Conductivity (σ) at 373 K and Activation Energy (E_a) of the complexes

Metal		Mn(III)	Fe(III)	VO(IV)	Zr(IV)	$\text{UO}_2(\text{VI})$
HCNAT	σ ($\Omega^{-1}\text{ cm}^{-1}$)	1.97×10^{-8}	4.32×10^{-7}	1.14×10^{-8}	2.66×10^{-8}	6.67×10^{-7}
	E_a (eV)	0.0114	0.1626	0.0791	0.0642	0.2113

Synthesis of 4-(hydroxyphenyl)-2-aminothiazole;



Electrical conductivity:

The electrical conductivity and activation energy of HCNAT complexes are shown in table 4. [20-24] Electrical conductivity of the complexes lies in the range of 2.10×10^{-9} to $6.69 \times 10^{-7} \text{ } \Omega^{-1}\text{cm}^{-1}$ at 373 K. The electrical conductivity of these complexes at 373 K follows the order $\text{UO}_2 > \text{Fe} > \text{VO} > \text{Mn} > \text{Zr}$. The activation energy of electrical conduction of the complexes has been found to increase in the order $\text{Mn} < \text{Zr} < \text{VO} < \text{Fe} < \text{UO}_2$.

CONCLUSION

In conclusion, we have synthesized new ligand 2-hydroxy-5-chloro-3-nitroacetophenone 4-(p-hydroxyphenyl)-2-iminothiazole and their metal complexes. All the complexes indicating their semiconducting behavior. Ligand was found to bind the metal ion monobasic (ON) bidentate manner.

Conflicts of interest: The authors stated that no conflicts of interest.

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