

Antimicrobial and Magnetic Studies Of 2-Hydroxy-5-Methyl-3-Nitro Acetophenone Thiazole Schiff Base Complexes of VO(IV), Zr(IV) and UO₂(VI)

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ABSTRACT

Coordinating metal complexes of VO(IV), Zr(IV) and UO₂(VI) have been synthesized using 2-hydroxy-5-methyl-3-nitro acetophenone thiazole Schiff base ligand was derived from the condensation of 2-hydroxy-5-methyl-3-nitro acetophenone and thiazole. The Schiff bases behaved as charge bidentate ligand. The ligand was characterized by elemental analysis and spectral methods. Metal complexes characterized by elemental analysis, conductance measurements, molecular weight determinations and spectral studies. The Schiff base and their metal complexes have been evaluated for their antibacterial activities The synthesized products are coloured solids, soluble in DMF, DMSO and THF.

Keywords: Schiff base, Magnetic susceptibility, Antimicrobial

I. INTRODUCTION

Schiff bases have often been used as chelating ligands in the field of coordination chemistry and their metal complexes are of great interest for many years. In the development of coordination chemistry and biochemistry the compounds which contain pyridine and its derivatives or Schiff bases as ligands have occupied a central role. The chemical studies of metal complexes with heterocyclic Schiff base ligands containing nitrogen, sulfur, and oxygen has attracted increasing attention. The studies of synthesis optical characterizations and Solar Energy applications of New Schiff Base materials¹. Synthesis, characterization and antifungal activity of manganese (II) complex with Schiff Base derived from acetylacetone and leucine² The newly synthesized Schiff bases, 2-acetylthiophene thiosemicarbazone and thiophene-2-aldehyde thiosemicarbazone and their metal complexes with Co(II), Cu(II), Zn(II) and Ni(II) complexes and Their Schiff bases metal complexes were tested for antibacterial activity³ They are reported the possible use of such systems in biological applications for their antifungal properties and antioxidant activities⁴. 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, electroluminescent 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-

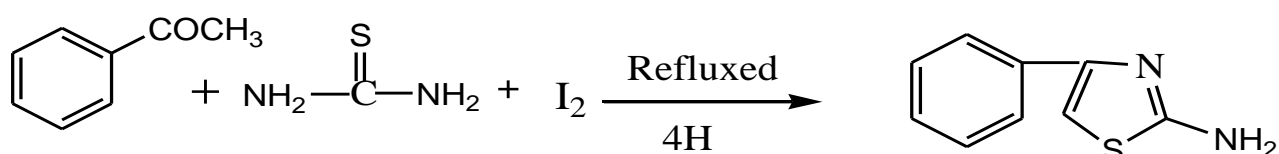
derived Ligand Schiff base and its complexes with Cu (II), Hg (II), Ni (II), Mn (II) and Co (II)⁵. Due to biological potency, pharmacological properties and synthetic flexibility of thiazole Schiff bases. The aim of present investigation is to synthesize various transition metal complexes of Schiff base derived from 2-hydroxy-5-methyl-3-nitro acetophenone and 2-amino-4-phenylthiazole.

II. EXPERIMENTAL

All the chemicals were of A.R. grade and used as received. 2-hydroxy-5-methyl-3-nitro acetophenone (HMNA) and 2-amino-4-phenylthiazole was prepared by known methods⁶⁻⁹. The solvents were purified by standard methods¹⁰.

Synthesis of 2-amino-4-phenylthiazole:

The synthesis of 2-amino-4-phenylthiazole prepared by known method⁷⁻⁹. Yield: (75%); m.p.: 148-150°C



Acetophenone

2-amino-4-phenylthiazole

Synthesis of 2-hydroxy-5-methyl-3-nitroacetophenone 4-phenyl-2 imino thiazole [HMNAT]:

A solution of 4-phenyl-2 imino thiazole (0.02M) in 25ml of ethanol was added to an ethanolic solution(25ml) of 2-hydroxy-5-methyl-3-nitro acetophenone (0.02M) and the reaction mixture was refluxed on a water bath for 4-6h. 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. 310°C

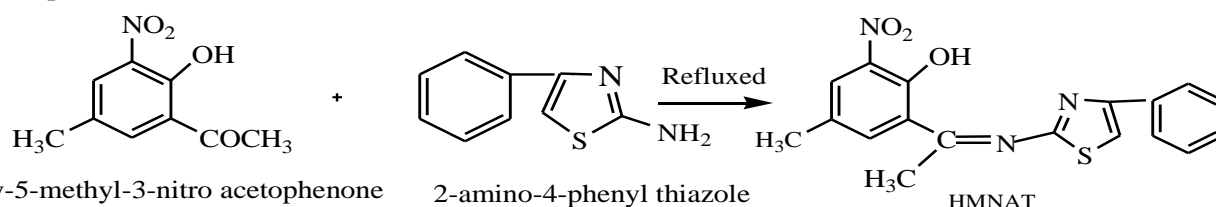


Table1. Analytical data of the Ligands.

Ligand	Molecular Formula	Formula Weight	Color and nature	Elemental Analysis		
				C% found (Cal.)	H% Found (Cal.)	S% Found (Cal.)
HMNAT	C ₁₈ H ₁₅ N ₃ O ₃ S	353.1	Yellow Crystalline	60.42 (61.17)	04.04 (04.24)	08.84 (09.06)

Preparation of complexes: All the metal complexes were prepared in a similar way by following method. To a hot solution of ligand HMNAT (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 3-5 h. The precipitated complexes were filtered, washed with ethanol followed by ether and dried over fused calcium chloride. Yield : 55-60%

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^{11,12} 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 400-4000cm⁻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 [HgCo(SCN)₄] as the calibrant. The molecular weights of the complexes were determined by Rast method.

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 ($\Omega^{-1} \text{cm}^2$ mol ⁻¹)
			M%	C%	H%		
[VOL ₂]	771.2	Green	6.50 (6.60)	55.55 (56.01)	3.17 (3.89)	1.5	11.8
[ZrL ₂ (OH) ₂] 2H ₂ O	865.4	Yellow	9.88 (10.53)	49.14 (49.91)	3.34 (3.92)	Dia	17.2
[UO ₂ L ₂]	974.3	Orange	23.53 (24.43)	43.64 (44.33)	2.93 (3.07)	Dia	13.2

III. RESULT AND DISSCUTION

The Schiff base HMNAT and its complexes have been characterized on the basis of ¹H NMR, IR spectral data, elemental analysis, molar conductance, magnetic susceptibility measurements and thermogravimetric analysis data . All these values and analytical data is consistent with proposed molecular formula of ligand . 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(10⁻³M) solution at room temperature (Table2) shows all the complexes are non electrolytes. The ¹H NMR spectra of ligand HMNAT shows signals at δ 12.24,(1H, s phenolic OH), δ 7.60, 7.74, 7.63 and 7.72 (4H, m, phenyl) δ 6.87, 6.88, and 6.72(3H, s Phenyl), 6.78 (1H s thiophene), and 2.66(3H, s, methyl) ^{11,13-15}.

IR spectra of ligand and metal complexes shows $\nu(\text{C}=\text{N})$ peaks at 1626 cm⁻¹ and absence of C=O peak at around 1700 – 1740 cm⁻¹ indicates the Schiff base formation¹⁶⁻¹⁹.

Table 3. IR spectra of ligand and metal complexes

Compound	$\nu(\text{O-H})$ hydrogen bonded	$\nu(\text{C=N})$ imine	$\nu(\text{C=O})$ phenolic	$\nu(\text{M-O})$	$\nu(\text{M-N})$	$\nu(\text{C-S})$
HMNAT	3085	1626	1520	--	--	1128
[VOL ₂]	--	1599	1502	514	445	1094
[ZrL ₂ (OH) ₂] 2H ₂ O	--	1601	1488	445	412	1102
[UO ₂ L ₂]	--	1588	1442	550	480	1084

IV. ANTIMICROBIAL ACTIVITY

Antimicrobial activity assay depends upon a comparison of the inhibition of growth of microorganism by measuring the concentration of the sample to be examined with the known concentration of standard antibiotic. For the antimicrobial analysis the agar diffusion method has been employed. In this study the ligand and their metal complexes were tested for their effect on certain human pathogenic bacteria such as Gram-positive.

The ligand HMNAT and its complexes²⁰⁻²⁷ are found to show considerable bacteriocidal activity against *E. coli*, *A. aerogenes*, *S. aureus* and *B. subtilis* and are almost inactive against *B. megatherium*, *P. vulgaris* and *P. fluorescens*. The ligand inhibits the growth of *S. aureus* more than all its complexes. The results reveal that the sensitivity of ligand HMNAT and its complexes is shown in Table 4.

Table 4. Antimicrobial activity

Ligand and its complexes	<i>B. subtilis</i> (mm)	<i>P. vulgaris</i> (mm)	<i>S. aureus</i> (mm)	<i>E. coli</i> (mm)	<i>P. fluorescens</i> (mm)	<i>A. aerogenes</i> (mm)	<i>B. megatherium</i> (mm)
HMNAT	R	S ₆	R	S ₁₂	R	S ₉	R
VO- HMNAT	R	R	S ₁₃	S ₁₁	R	S ₁₁	S ₉
Zr- HMNAT	S ₁₁	S ₁₀	S ₁₄	R	R	S ₁₁	R
UO ₂ -HMNAT	S ₈	R	S ₈	S ₁₃	R	S ₉	R

S-Sensitive, R-Resistant

V. CONCLUSIONS

In conclusion, we have synthesized new ligand 2-hydroxy-5-methyl-3-nitro acetophenone-2-imino thiazole and their metal complexes. The newly synthesized Metal complexes were confirmed by the spectral analysis

and further evaluated for their antimicrobial activity. It is observed structural changes in metal complexes have marked effect on the sensitivity and sensitivity varies with organisms. The antibacterial activity revealed that most of the compounds showed moderate to good activity.

VI. REFERENCES

- [1] . Sobhi M.G., Hoda A., Mohamed S., Tariq Z.A., Muna S.K. and Khalid A.A., *Materials J.*, 2021, 14, 3718
- [2] . S. M . Aishatu., A.Fatima and E. A Abigail., *American J. Nano Res. and Appl.*, 2017, 5(6), 110.
- [3] . E. Katsoulakou, M. Tiliakos and G. Papaefstathiou, *Journal of Inorganic Biochemistry*, 2008, 102(7), 1397.
- [4] . Siham Slassi, Adeline Fix-Tailler, Gérald Larcher, Amina Amine and Abdelkrim El-Ghayoury, *J. of Heteroatom Chem.*, 2019, 6862170,1
- [5] . Rehab K A.S., Wurood A J., and Ali N N., *J. Pharma, Bio and Chem Sci.*, 8(3), 2017, 174.
- [6] . Aswar A., Bahad P.,Pardhi A. and Bhawe N., *J. Poym. Mater.*, 1988, 5, 232.
- [7] . Pattan S.,Ali M, Pattan J., Purohit S., Reddy V. and Nataraj B., *Indian J. Chem.*, 2006, 45B, 1929.
- [8] . Khrustalev D., Suleimenova A. and Fazylov S., *Russian J. App. chem.*, 2008, 81(5), 900.
- [9] . Maradiya H. and Patel V., *J. Fibers and poly.*, 2002, 3(1), 43.
- [10] . Furniss B., Hannaford A., Smith P. & Tatchell A., *Vogel's practical organic chemistry 5 thEd.* (Logman Scientific Technical, John Wiley and Sons), 1989.
- [11] . Sadigova S., Magerramov A. and Allakhverdiev M., *Russian J. Org. chem.*, 2008, 81(5), 900.
- [12] . Vogel AI, "A Text book of quantitative inorganic chemistry"3thEd., (ELBS,London),1961.
- [13] . Campbell E and Nguyen S, *J. Tetrahedron*, 2001, 42, 1221.
- [14] . Pietikainen P and Haikarainen A, *J. Mole. Catalysis.*, 2002, 180, 59.
- [15] . Kidwai M, Poddar P and Singhal K, *Indian J. Chem.*, 2009, 48B, 59.
- [16] . Sonwane S, Srivastava S and Srivastava S, *Indian J. Chem.*, 2008, 47B, 633.
- [17] . Patel K and Mehata A, *E. J. Chem.*, 2006, 3(13), 267.
- [18] . Maurya R, Antony D, Gopinathan S, Puranic V, Tavale S and Gopinathan C, *Bull. Chem. Soc. Jpn.*, 1995, 68, 2847.
- [19] . Boghaei D and Mohebi S, *J. Tetrahedron*, 2002, 58, 5357.
- [20] . Sutariya B, Mohan S, Sambasiva S and Rao S, *Indian j. Chem.*, 2007, 46B, 884.
- [21] . Baluja S, Solanki A and Kachhadia N, *J. Iranian Chem. Soc.*, 2006 3(4), 312.
- [22] . Venkatesh P, *Asian J. Pharm. Hea. Sci.*, 2011, 1(1), 8.
- [23] . Singh UI, Singh RK, Devi WR and Singh CH, *J. Chem. Pharm. Res.*, 2012, 4(2), 1130.
- [24] . Adeoye IO, Adelowo OO, and Onawumi OO, *J. Chem. Pharm. Res.*, 2012, 4(1), 1
- [25] . Chauhan N, Vyas K, Nimavat K and Joshi K, *J. Chem. Pharm. Res.*, 2012, 4(2),1106
- [26] . Barry AL, *The Antimicrobial Susceptibility Test, Principle and Practice*, Illus, Lea, and Febiger, Philadelphia,Pa, USA, 1976; 180.
- [27] . Raj CI, Christudhas M and Raj GA, *J. Chem. Pharm. Res.*, 2011, 3(6), 127