



Schiff base complexes derived from thiosemicarbazone. Synthesis, characterization & their antifungal activity

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ABSTRACT

The Schiff bases bis (P-Methoxy benzaldehyde) thiosemicarbazone (MBTSCZ), bis (N, N¹, dim ethyl benzaldehyde) Thiosemicarbazone (DMACTSCZ), bis (N, N¹-dimethyl benzaldehyde) thiosemicarbazone (DMABTSCZ), bis (Pyridine-2-aldehyde)thiosemicarbazone (PATSCZ), bis (P-chlorabenzaldehyde) thiosemicarbazone (CBTSCZ) were synthesized & characterization by elemental analysis melting point & I.R. spectra. Their complexes with Nickel (ii) have been synthesized & characterized by elemental analysis. electrical conductance, Magnetic susceptibility, I.R. spectra & electronic spectra, thiosemicarbazones & their Ni (ii) complexes have also been screen by antifungal activities.

Key word: - Thiosemicarbazone, Schiff base, Ni (ii), antifungal activity.

Introduction

The co-ordination compound showed different type of geometry, literature survey reveal's few report regarding complexion behavior of schiff base. containing thiosemicarbazide^{1,2,3}, No wok has-been done on the Schiff base derived from p-methoxy benzaldehyde, N, N¹ demethylcinnamaldehyde N, N¹-dimethyl benzaldehyde, Pyridine-2-aldehyde & p-chlorabenzaldehyde Also not much

work done on their antifungal activity we have synthesized these ligands and also characterized by T.L.C, Melting point & I.R. spectra. The complexing behavior of these ligands has been studied and their structure has been established. Using analytical data, magnetic moment, I.R. spectra & electronic spectral data. The result obtained is in good agreement with the ligand field splitting energy ($10 Dq$). The antifungal activity of Schiff base and their corresponding complexes have been studied.

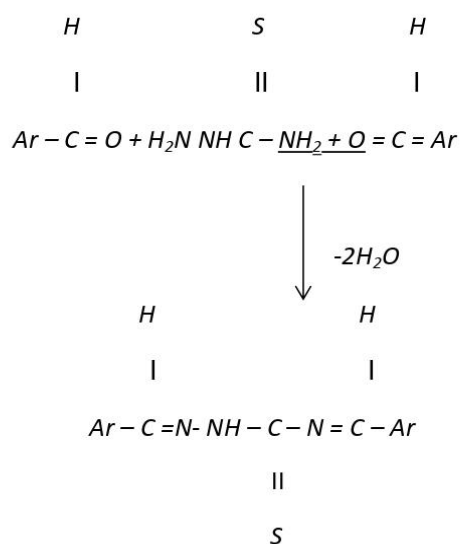
Material & Method

A.R. grade chemicals are used in the synthesis of ligand & their complexes. Schiff bases prepared by the different aldehyde & thiosemicarbazide. Above chemicals were collected from different Pharmaceutical's such as Aldrich, Fluka, Sigma, Glaxo, B.DH etc. In the preparation of complexes metal salt nickel (ii) chloride is used.

I.R. spectra, elemental analyses were carried out R.S.I.C. department CDRI Lucknow, conductivity measurement were carried out by Philip conductivity bridge model. PR 9500 with deuterium conductivity cell. The conductance of the complexes was measured in MeOH, DMF & DMSO at 10^{-3} M dilution at 30°C . Magnetic susceptibility of the complexes was determined by the Goy's method. The sample tube was calibrated with CuSO_4 . The diamagnetic correction was made for the ligand. T.G.A. were carried out in G.N.D. University.

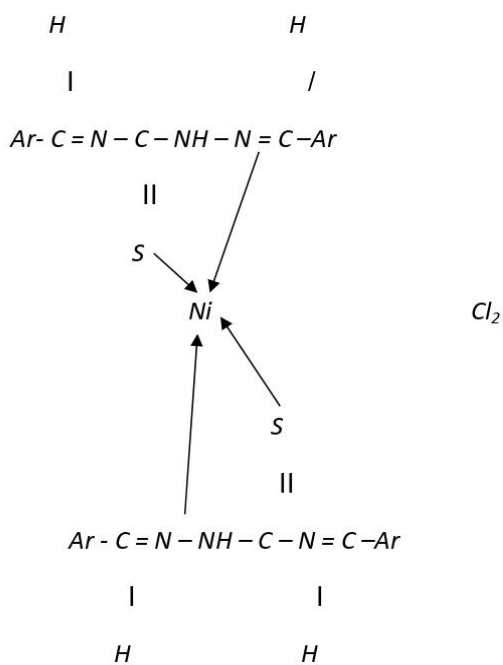
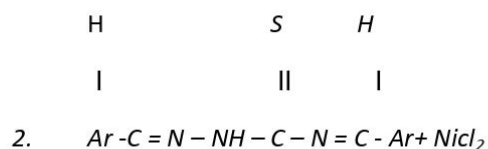
Preparation of Schiff bases

The Schiff bases were prepared by the condensation of respective aldehyde & thiosemicarbazide in 2:1 ratio. The thiosemicarbazide was dissolved in ethanol & refluxed for half an hour. The requisite amount of respective aldehyde was added in the flask. The mixture was refluxed for six hours. The reaction mixture was kept for 24 hours. The crystals of the Schiff base were obtained which were purified by recrystallization. The purity of ligands was checked by melting point determination, elemental analysis. TLC & spectroscopic studies were also recorded.



Preparation of Complexes

The preparation of complexes were prepared by the adding the nickel chloride solution to the solution of ligand. In THF & DMSO in 1:2 ratio. The precipitation of the complex thus obtained was washed with THF & DMSO & dried over fused $CaCl_2$.

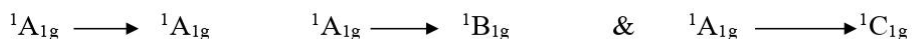


Result & Discussion

The elemental analyses indicate that the complexes have the composition [Ni (Ligand)₂Cl₂]. The molar conductance value of Ni (ii) complexes in three solvents (MeOH, DMF & DMSO) in 10⁻³ M dilution at room temp are reported around 1:2 electrolyte complexes.

The study of magnetic moment of the complexes shows the diamagnetic character. The diamagnetic complexes are square planner. Diamagnetic is consequences of eight electron are paired in the four lower lying d-orbital Ni (ii) is prone to form for co-ordinate square planner complexes. Especially with ligand containing S-donar atom.

All diamagnetic nickel (ii) complexes shows the three electron spectral band near at 17650, 20880, 26450. cm^{-1} . These band suggest that. these -



Transition respectively.³

The I.R. spectra. of schiff base of Thiosemicarbazide. Shows a band b/w 750-920 cm^{-1} . This band may be assigned the $\text{V C} = \text{S}$ vibration. This band is shifted to b/w 810 cm^{-1} – 920 cm^{-1} in spectra of complexes suggesting that co-ordination through. S-atom of thiogroup. Another Important band in the I.R. spectra of the schiff base appear in b/w 1550-1750 cm^{-1} Which shifted in b/w 1570-1750 cm^{-1} in the spectra of the complexes. This shift suggested that co-ordination of nitrogen atom of azomethane group.

Table - 1

Sr. No	Name of Molecular formula	Colour	M. P	Elemental analyses					Molar Conductance			M. moment
				C	H	N	S	M	MeOH	DMF	DMSO	
1	[Ni(MBTSCZ) ₂].Cl ₂ (C ₁₇ H ₁₇ N ₃ O ₂ S) ₂ NiCl ₂	Radish	285	52.04	4.33	10.71	8.16	7.53	180	140	75	2.99
		Yellow		(51.03)	(5.13)	(11.64)	(8.16)	(6.41)				
	[Ni(DMACTSCZ) ₂].Cl ₂ (C ₂₃ H ₂₇ N ₅ S) ₂ NiCl ₂	Light	296	58.72	5.74	14.89	6.80	6.27	195	160	65	2.94
		Yellow		(57.36)	(4.28)	(12.69)	(7.17)	(4.92)				
	[Ni(DMABTSCZ) ₂].Cl ₂ (C ₁₃ H ₁₁ N ₅ S) ₂ NiCl ₂	Yellowish	305	54.54	5.50	16.75	7.65	7.05	202	155	88	3.02
	[Ni(PATSCZ) ₂].Cl ₂ (C ₁₃ S ₁₃ N ₅ S) ₂ NiCl ₂	Redish Brown	275	46.70	3.29	20.95	9.58	8.83	270	170	58	3.01
	[Ni(CBTSCZ) ₂].Cl ₂ (C ₁₅ H ₁₁ N ₃ Cl ₂ S) ₂ NiCl ₂	Yellow	285	44.88	2.74	10.47	7.98	7.35	205	195	55	2.97

Antifungal Activity of Schiff bases & Their Complexes

Their Complexes

The metal complexes of schiff bases are widely used as fungicides, an septic & disinfectant. The metal complexes shows antimicrovial activity. Pechiney prugel⁵ prepared the schiff base having sheolic & halogen group by condensing. ^{6,7} dichloro & dibromo salicyaldelyde with 2 amino – 2 – ethyl – 1,3 propanediol which were used as agriculture fungicides. The schiff derived from thiosemicarbazide & their metal complexion was tasted against verious fungi & bacteria. certain schiff base containg NH₂ group d group against b. subtilis, E. coli, A. fumigates & A. Niger at 50 gm. mi-1 condensation by single disc method. ^{8,9} It is suggest that the schiff bases & their metal complexes having antimicrobial activity may be either by the killing the microbe or by blocking active ste of microbe. ^{6, 7, 8}

Table-2

Antifungal activity of Schiff base & their Nickel (ii) complexes

Sr. No	Name of Compound	A. niger		A. fumigates	
		0.02	0.002	0.02	0.002
1	Thiosemicarbazide	-	-	-	-
2	[MBTSCZ]	++	+++	++	++
3	[DMACTSCZ]	+++	+	++	+++
4	[DMABTSCZ]	++	+++	+	+++
5	[PATSCZ]	+++	+++	++	++
6	[CBTSCZ]	+++	+++	++	+++
7	[Ni(MBTSCZ) ₂]Cl ₂	+++	++	+	++
8	[Ni(DMACTSCZ)]Cl ₂	++	+	++	+
9	[Ni(DMABTSCZ) ₂]Cl ₂	+++	++	++	++
10	[Ni(PATSCZ) ₂]Cl ₂	+++	++	+	++
11	[Ni(CBTSCZ) ₂]Cl ₂	++++	++	+++	++

Inhibition Diameter of Zone.

-	less than 11MM
+	11-14 MM
++	15-18 MM
+++	19-22 MM
++++	23-25 MM
+++++	More than 25MM

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