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Synthesis and characterization of schiff base salicylaldehyde and thiohydrazones and its metal complexes

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ABSTRACT

The ligand salicylaldehyde & thiohydrazones was synthesized & characterized by elemental analyses & IR spectra. Its metal complexes with the metals Ti (III), V (III), VO (IV), CO (II) & MN (III) were synthesized & characterized by the determination of mp, molar conductance, magnetic susceptibilities, electronics & IR spectral studies, Based on the studied & octahedral geometry has been proposed for all the synthesized complexes

INTRODUCTION

Metal complexes of Schiff bases have played a central role in the development of co-ordination chemistry. Schiff bases offer a versatile and flexible series of ligand capable of bind with various metal ions to give complexes with suitable properties for theoretical or practical application. During the past two decades, considerable attention has been paid to the chemistry of the metal complexes of schiff base containing nitrogen and other donors. This may be attributed to their application in many fields.



MATERIALS AND METHODS

Preparation of ligand:

The Schiff bases were prepared by the condensation of respective aldehyde & amino compound the salicylaldehyde morpholine N-thio hydrazone was prepared by refluxing salicylaldehyde and morpholine thio-hydrazone in 1:1 molar ratio in ethanol 2.44 gm of the aldehyde was dissolved in ethanol and refluxed for half an hour. 2.10gm of morpholine thiohydrazone was added and the mixture was refluxed for 6 hours. The solution was kept for 24 hour. Crystal of ligand were obtained and purified by recrystallisation.

Structure:



Salicylaldehyde Morpholine N-thiohydrazone

Characterization of ligand:

The melting point of the ligand was determined by open capillary method & uncorrected. The ligand was subjected to elemental analyses for C, H, N to ascertain its molecular composition the IR spectra of the ligand was recorded in KBr phase.

S. No.	Name & Molecular formula of the ligands	Colors	M.P/ D.T	Elemental analysis					
				%	%	%	%	%	Stability
				of C	of H	of N	of O	of S	
1.	Salicyaldehyde Morpholine N- thiohydrazone	Light Yellow	180	45.90	3.25	20.10	14.88	14.88	DMF
				(44.80)	(3.20)	(19.20)	(13.90)	(13.80)	

Preparation of complexes

The metal complexes were prepared by adding the solution of the ligand in appropriate solvent to the solution of the respective metal. A Slight excess of the ligand was added to ensure complete precipitation of the metal complexes. The precipitate so obtained was filtered washed and dried in vacuum desicater.

Characterzation of metal complexes

The colours of complexes were noted and their melting points were determined. The complexes were subjected to elemental analyses. The metal was estimated gravimetrically. The molar conductance of the complexes was measured at 10^{-3} M dilution in DMSO/ methanol or both. The magnetic properties of complexes were studied & infrared and visible spectra were recorded. The complexes were also subjected to the thermo-gravimetric analyses.



Salicylaldehyde morpholine N- thiohydrazone vanadium (III) chloride

Elemental analyses

It was carried out at R.S.I.C., CDRT, Lucknow.

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IR Studies

The IR studies of the complexes were recorded with perkin Elmer spectrophotometer model 651 in KBr or Nujol phase from 4000 cm⁻¹ to 200 cm⁻¹ at R.S.I.C, CRDI, Lucknow.

Conductivity measurements:

Conductivity measurements were carried out by philips at department of chemistry, Bareilly College Bareilly. The conductance of the complex were measured in methanol and DMSO.

Magnetic Susceptibility:

Magnetic susceptibility of the complexes was determined by the Gouy method at the department of chemistry, Bareilly College Bareilly. The sample tube was calibrated with $CuSO_4$. The diamagnetic corrections were made for the ligands.

Thermo-gravimetric Analysis (TGA):

This was done at the Chemistry Department, GND University Amritsar.

Visible Spectra:

Visible spectra were recorded with Beckman DU-2-spectrophotometer in the range of 750 cm⁻¹ to 300cm⁻¹ at the department of chemistry, Bareilly College Bareilly.

RESULTS AND DISCUSSION

The colour of complexes were noted the melting point of the complexes were determined was open capillary and compose with the ligand to ascertain the formation of the complexes. The elemental analysis suggested 1:1 metal ligand sterieometric for all the synthesized complexes the determination of molar conductance at room temperature and 10^{-3} M dilution. DMSO suggested 1:1 electrolytic nature for all the complexes. The comparison of IR spectra of the ligand with metal suggested dibasic tridentate nature of the ligand.

The IR spectra of the ligand and the metal complexes have been recorded in kBr phase. The comparison of the spectra indicate that both phenolic –OH and v(C=S) disappeared and a new band appeared at 755cm⁻¹, suggesting co-ordination of metal ion through thiolsulphur and phenolic oxygen via deprotonation. Along with this the lowering of v (C=N) by 15 cm⁻¹ indicates co-ordination of azo-methane nitrogen atom, which is further substaintated by the appearance of azine chromophore v(>C=N-N=C<) around 1600 cm⁻¹. Thus in this complex the ligand functions in a diabasic tridentatae fashion bonding through O, N and S atom.

The IR spectra of the complexes showed some additional bands in the region of 3480-3510 cm⁻¹ assigned to ν –OH of co-ordinated water, in the region of 840-850 due to rocking mode and 740-755 attributable to wagging mode of co-ordinated water molecules. TGA also supported the finding of IR spectra regarding co-ordenated nature of water molecules. The thermogram of all the complexes showed loss of 3 water molecules except of VO (IV) complexes which lost two water molecules in the temperature range of 170-190 0 C.

REFERENCES

[1] S.S. Djebbar, B.O. Benali and J.P. Deloume *Polyhedron*, **1997**, 16, 21, 75.

[2] C.M. Liu, R.G. Xiong, X.Z. you, Y.J.Liu and K.K.Cheung, Polyhedron, 1996, 15, 4565.

[3] P. Venkateshwar Rao and A Venkata Narasaiah, *International Journal of Chemistry, August* 2003 vol. 42A, pp. 1896-1899.

[4] Sarswati Ravi and Suman Bala, Oriental Journal of chemistry, 2012 28, pp. 1685-90.

[5] Karuna Mahajan, Nighat Fahmi & Ran Uir Singh, Indian J Chemistry, 2007 46A: 1221.

[6] Jayabalakrishnan c. and Natarajan K. synt. React. Inorg. Met.org. Chemistry, 2001 31, 98.

[7] Masoud salavai-Nissari & Ahmad Amiri, journal of Molecular Catalysis A: Chemical 2005 235, 114-121.

[8] Kamal Mi ibrahim, Sahar I Mostafa, Nagwa Nawar & Zeinab A Younis, *Indian journal of Chemistry*, **2004**, 43A, 2294-2300.

[9] Rahul K Rastogi, Poonam Garg & Shamim Ahmad, Asian J Chemistry, 2009, 21, 6144.

[10] Samik K Gupta, S.Roy, T.N.Mandal, K.Das & S.Roy J. Chemitry Sci., 2010 122, 239.

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