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Research Article

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Synthesis, spectral characterisation of Ni(II) complexes with Schiff base ligand MPLTSC: Triaquo(3-hydroxy-5-hydroxymethyl-2-methoxypyridine-4-carbaldehyde thiosemicarbazone) Nickel(II) nitrate [Ni(MPLTSC) $(H_2O)_3$] $(NO_3)_2$

Dr. Somnath Jha

+2 Sarvodaya High School, Gangasagar, Darbhanga - 846004

E-mail: dr.somnathjha@gmail.com

Abstract

The new octahedral Nickel(II) complex has been synthesized from the reaction of aqueous solution of Nickel (II) nitrate with the tridentate Schiff base ligand MPLTSC (3-hydroxy-5-hydroxymethyl-2-methoxy pyridine-4-carbaldehyde thiosemicarbazone) in ethanol. This complex has been characterized by elemental analysis, I.R. electronic spectra.

Keywords: Ni(II) complex, MPLTSC, spectral characteristic.

Introduction

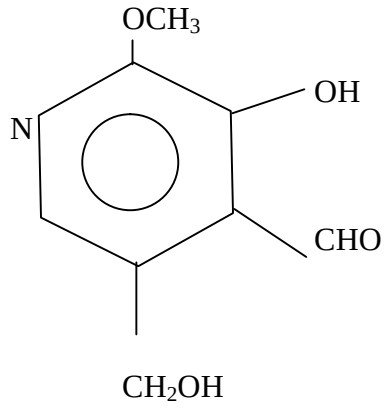
Research based on new Schiff bases and their metal complexes represents one of the most promising areas of material science, catalysis and chemical research[1-3]. The Schiff bases are easily synthesized and form complexes with almost all metal ions. Studies on such complexes of transition metals, including nickel, have received overwhelming attention in recent times due to their important catalytic properties[4-14]. The synthesis of nickel(II) complexes with mixed O, N and S donor sets is the research interest of many groups[15-17]. Furthermore, nickel complexes containing triphenylphosphine ligands play an important role in small molecular catalysts used for the synthesis of fine chemicals and drug molecules[18-22].

Experimental

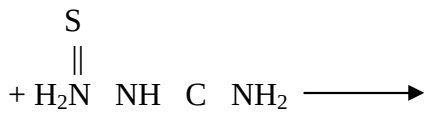
All the chemicals used of research grade chemicals of either B.D.H. Annalar or pure E. Merck quality. Carbon, Hydrogen, Nitrogen analysis were carried form C.D.R.I. Lucknow. The conductivity of the complexes were determined with help of systronics conductivity bridge. The conductivity of complexes were measure in DMF solution. Molar conductivity of complex was calculated by using the formula M = K x 1000 /C were K = conductivity of complex, C = concentration of complex. The magnetic susceptibilities of solid complexes were determined by the Bouy method at room temperature. The magnetic moment data were made available to us by kind courtesy of Patna Science College Laboratory Patna. IR Spectra were taken from Perkin Elmer IR Spectrometer from department of Chemistry I.I.T. New Delhi and by the courtesy of C.D.R.I. Lucknow. Electronic spectra of the compounds were recorded by the courtesy of Chemistry IIT New Delhi and by the courtesy of CDRI Lucknow.

Synthesis of Ligand :-

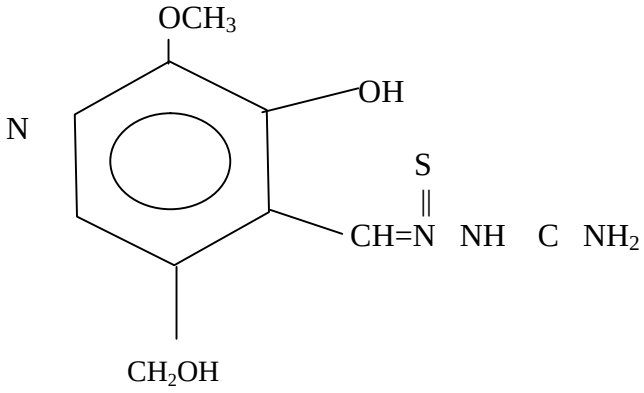
Preparation of 3-Hydroxy-5-Hydroxymethyl - 2- methoxypyridine - 4 - carbaldehyde-thiosemicarbazone (MPLTSC)



3-Hydroxy–5–Hydroxymethyl–
2–methoxypyridine–4–carbaldehyde
(MPL)



Thiosemicarbazide
(TSC)



3–Hydroxy–5–hydroxymethyl–2–methoxypyridine–4–carbaldehyde thiosemicarbazone (MPL TSC)

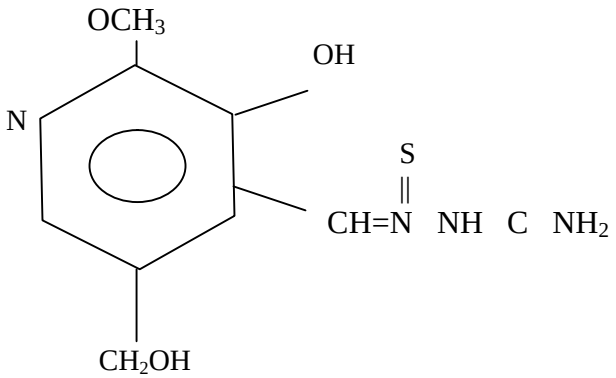
A solution of MPL (1 mmole) in alcohol was mixed with TSC (0.5m mole) in water. The resulting solution was acidified with few drops of HCl and refluxed for an hour on water bath on cooling, yellow solid separated. It was washed thoroughly with dil. HCl and finally with water. The thiosamicarbazone was recrystallised from alcohol upto 218^0C.

Analysis	:	$C_9H_{12}O_3N_4S$
Calculated	:	C– 42.18, H – 4.70, N – 21.87%
Found	:	C– 42.06, H – 5.12, N – 21.52%

I.R. Bands of 3-Hydroxy-5-hydroxymethyl-2-methoxypyridine-4-carbaldehyde thiosemicarbazone (MPLTSC)

Bands (cm^{-1})	Assignment
3460	ν N–H streching
3200	ν O–H (H–bonded) phenolic
2850	ν $(N–H)^+$ streching
1395	ν C=S streching
1634	ν C=N (Pyridine ring)
1440	ν N–H bending

The IR and elemental analysis suggests the following structure for ligand:



3-Hydroxy–5–Hydroxymethyl–2–methoxypyridine–4–Carbaldehyde thiosemicarbazone (MPL TSC)

Synthesis of Complex:-

Triaquo(3–hydroxy–5–hydroxymethyl–2–methoxypyridine–4–carbaldehydethiosemi carbazone) Nickel(II) nitrate :

$[Ni(MPLSC)\ (H_2O)_3]\ (NO_3)_2$

The alcoholic solution of the ligand (MPLSC) (1 mmole) was mixed with aqueous solution of Nickel (II) nitrate (0.713 mmole). The whole solution was refluxed about three hours and left to stand for an hour. The red coloured precipitate obtained was filtered and washed thoroughly with water and ethanol and dried in air.

The compound was analysed chemically
.

Elemental analysis :

Calculated		
	Ni	11.92%
	C	21.92%
	H	3.65%
	N	17.04%
	S	6.49%
Found		
	Ni	11.35%
	C	21.05%
	H	4.01%
	N	16.98%
	S	6.03%

The compound was found to be insoluble in water, methanol and ethanol but dissolves in DMF.

The compound is decomposed when heated to above 250^0C.

Magnetic susceptibility:

The μ_{eff} value of the complex is 2.98 BM.

I.R. Bands of [Ni(MPLSC) $(H_2O)_3$] $(NO_3)_2$

Bands position (cm^{-1})	Assignment
3460	ν N–H stretching
3000	ν C–H stretching
1640	ν C=O stretching
1580	ν C=N stretching
1440	ν N–H stretching
1290	ν C–O (phenolic)
920–930	ν C–N bending
490	ν M–N bending
520	ν M–O bending

Results and Discussion

Triaquo(3–hydroxy–5–hydroxymethyl–2–methoxy–pyridine–4–carbaldehyde-thiosemicarbazone) Nickel(II) Nitrate (Ni(MPLTSC) $(H_2O)_3](NO_3)_2$

This Ni(II) complexes show four bands in their reflectance spectra favouring an octahedral stereochemistry[23] out of these four bands three can be assigned to spin allowed d–d transition of $^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$, $^3A_{2g}(F) \rightarrow {}^3T_{ig}$ (F) and $^3A_{2g}$ (F) $\rightarrow {}^3T_{1g}$ (F)

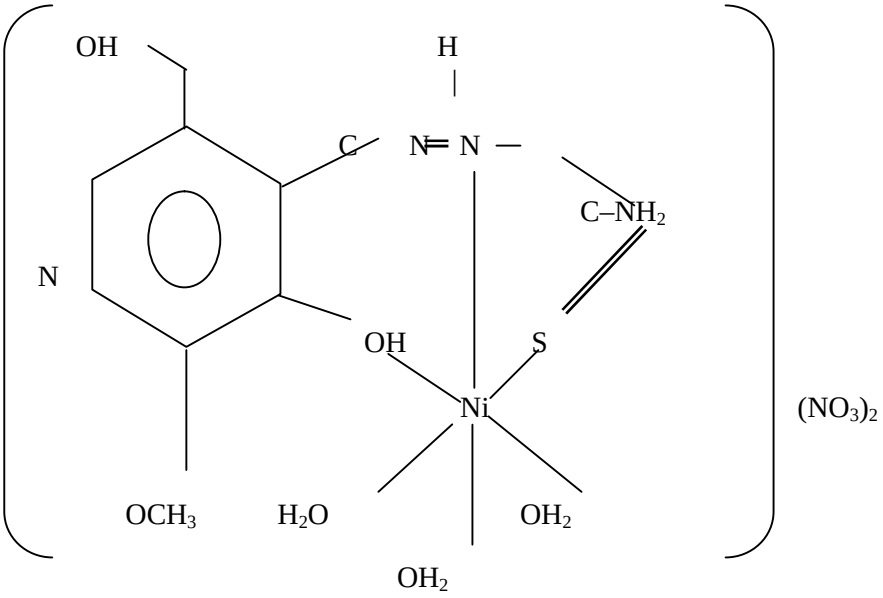
The fourth band near 10752 cm^{-1} is probably spin forbidden[24] and may be assigns to $^2B_{1g} \rightarrow {}^2E_g$, transition.

Electronic spectra;

The diffused reflectance spectra of Ni(II) complex gives four bands in their reflectance spectra at 11240 cm^{-1}, 16000 cm^{-1}, 24690 cm^{-1} and 10752 cm^{-1} out of which first three are assigned as $^3A_{2g}$ (F) $\rightarrow {}^3T_{2g}$ (F), $^3A_2$ (F) $\rightarrow {}^3T_{1g}$ (F) and $^3A_2$ (F) $\rightarrow {}^3T_{1g}$ (P) transition respectively and the last one is a spin forbidden transition.

The μ effective values of these complexes (2.98 BM are in good agreement with an octahedral geometry having two unpaired electrons. These complexes are found to be electrolytic in nature.

Now it is clear that these complex of Ni(II) have octahedral structure given below :-



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