STUDIES ON SYNTHESIS OF TETRA DENTATE SCHIFF BASES DERIVED FROM O-PHTHALALDEHYDE AND THEIR RU (II) COMPLEXES

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ABSTRACT

This article summarizes the method for the synthesis of tetra dentate Schiff base ligands by condensation of phthalaldehyde and 2-amino benzyl alcohol, 2-amino-2-methyl-1-propanol and 2-aminobenzohydrazine respectively. Later these ligands (L1-L3) were reacted with $[RuCl_2(DMSO)_4]$ (Dichlorotetrakis(dimethyl sulfoxide) ruthenium(II)) in methanol to give corresponding Ru(II) complexes. The synthesized ligands and complexes are characterized by elemental analysis, IR, ^{1}H-NMR and mass spectral studies.

Keywords: Schiff base ligands, Ru (II) complexes, o-Phthaladehyde, Ru(II) complex

INTRODUCTION

Schiff bases are an important class of ligands in coordination chemistry and find extensive applications in different fields. Schiff bases are derived from aromatic carbonyl compounds and have been widely studied in connection with metalloprotein models and asymmetric catalysis, due to versatility of their steric and electronic properties. Schiff bases and their biologically active complexes have been often used as chelating ligands in the coordination chemistry of transition metals, radiopharmaceuticals for cancer targeting, agrochemicals, model systems for biological macromolecules, catalysts and as dioxygen carriers. Tetra dentate Schiff base ligands have been used as chelating agents, these are playing vital role in coordination chemistry and its metal complexes are great attention for several years. These complexes are also used as catalysts for wide range of organic transformations such as C-H bond activation and oxidation reactions.

The chemistry of ruthenium has been receiving considerable current attention largely because of the interesting chemical properties and biological activities exhibited by these complexes. The main objective of the present work has been to synthesize complexes of ruthenium, incorporating the ligands BDMAB (butyldimethylammonium bicarbonate) (3a), BDMAZ (3b) and BDMAM (3c). This Schiff base ligand is abbreviated as H_2L, where H_2 stands for the two potentially dissociable protons, viz. the O–H proton and the N–H proton. All the Ru(II) complexes of Schiff base ligands are investigated for their antibacterial activity.

MATERIALS AND METHODS

All the chemicals like $[RuCl_2(DMSO)_4]$, phthalaldehyde, 2-amino benzyl alcohol, 2-amino-2-methyl-1-propanol; 2-amino benzo hydrazides were purchased from Aldrich, USA. The percentage of carbon, hydrogen, nitrogen and ruthenium Schiff base metal complexes are determined using a Perkin–Elmer CHN analyzer. Infrared spectra in KBr/CsI pellets were recorded with a Perkin-Elmer 283 spectrophotometer. ^{1}H NMR spectra were recorded on a Jeol 300 MHz FT-NMR spectrometer in DMSO-d_6. Mass spectra were recorded on CEC-21-110B and Finnegan MAT-1210 mass spectrometers.

General procedure for the synthesis of Ru (L1-L3) Cl_2] complexes: Schiff base ligands (L1-L3) prepared from reported method [16] (1.0 mmol) was taken in methanol (20 ml) and the solution was purged with a stream of nitrogen for 5 min. Then a solution of $[RuCl_2(DMSO)_4]$ (0.5 mmol) in methanol was added slowly. After being stirred for 2 h at room temperature, the solvent was evaporated under vacuo. Then 10 ml of diethyl ether was added to the residue, after which the orange crystalline solid was collected by filtration using a fine sintered-glass filter washed with ether, dried and recrystallized from mixture of DCM and diethylether.

RESULTS AND DISCUSSION

The physical and analytical data of Ru(II) compounds are depicted in Table.1. The IR spectra of all the Schiff base ligands and complexes are presented Table.2.

The ^{1}H NMR spectral data of ligands L1-L3 and Ru(II) complexes is listed in Table.3. The mass spectra of Ru(II) complexes shows molecular ion peaks at m/z (M^+) 515 (for compd-4a), 447 (for comp-4b) and 588 (for comp-4c)..

Table.1.Physical and analytical data of Schiff bases and their metal complexes

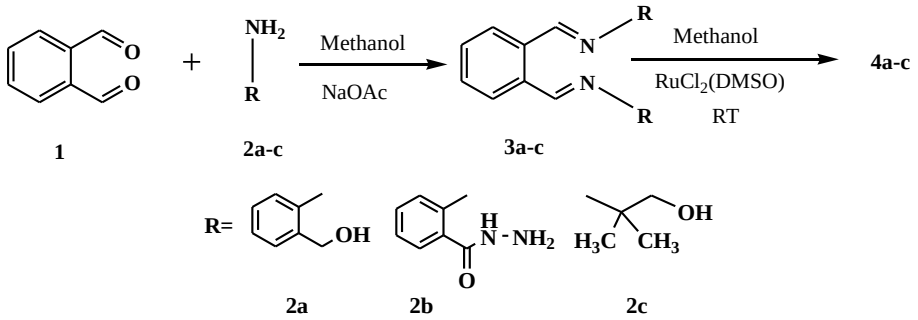
Ligand /complex, Color	M.F (M. Wt.)	Yield %	Calcd./ (Found)%			
			C	H	N	Ru
L1=(BDMAB) lightyellow	$C_{22}H_{20}N_2O_2$ (344)	(72)	76.69(76.72)	5.83(5.85)	8.19(8.13)	Nil
L2= (BDMAZ) white	$C_{22}H_{20}N_6O_2$ (401)	(75)	65.81(65.99)	4.96(5.03)	20.89(20.99)	Nil
L3=(BDMAM) yellow	$C_{16}H_{24}N_2O_2$ (276)	(70)	69.55(69.58)	8.68(8.75)	10.10(10.14)	Nil
$[Ru(L1)Cl_2]$	$C_{22}H_{18}Cl_2N_2O_2Ru$ (515)	(68)	51.37(51.28)	3.53(3.49)	5.45(5.38)	16.65 (16.59)
$[Ru(L2)Cl_2]$	$C_{22}H_{18}Cl_2N_2O_2Ru$ (588)	(72)	47.03(46.98)	3.95(3.89)	14.31(14.25)	17.21 (17.16)
$[Ru(L3)Cl_2]$	$C_{16}H_{22}Cl_2N_2O_2Ru$ (447)	(74)	43.06(43.00)	4.97(4.88)	6.28(6.21)	22.64 (22.58)

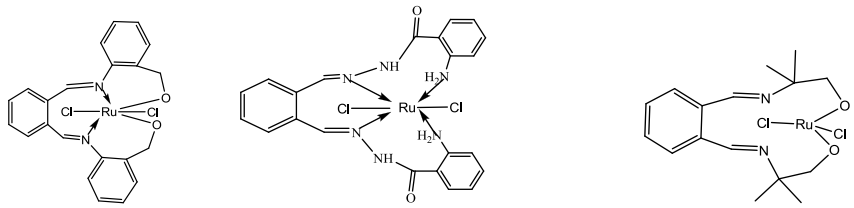
Table.2.IR Spectral data of ligands and its metal complexes (cm^{-1})

Ligand / Complex	$\upsilon_{C=N}$	$\upsilon_{C=O}$	$\upsilon_{NH}/\upsilon_{N-N*}$	υ_{M-N}	υ_{OH}	υ_{M-O}
L1	1639	Nil	Nil	Nil	3435	Nil
L2	1642	1687	3444	Nil	Nil	Nil
L3	1677	Nil	Nil	Nil	3448	Nil
$[Ru(L1)Cl_2]$	1576	Nil	Nil	529	Nil	430
$[Ru(L2)Cl_2]$	1596	1696	3359/1167	507	Nil	331
$[Ru(L3)Cl_2]$	1597	Nil	Nil	511	Nil	423

Table.3.^{1}H-NMR Spectral data of ligand and its metal complexes (δ ppm)

Ligand/Complex	^{1}H-NMR Spectra
L1	8.31(2H,s,- CH=N), 6.82-7.40 (12H, m, Ar-H), 4.6 (4H, s, $-CH_2$-O), 2.80 (2H, s, -OH)
L2	8.10 (2H,s, -CH=N), 8.0 (2H, s,-NH-CO), 6.61-7.94 (12H, m, Ar-H), 4.2 (4H, s, Ar-NH_2)
L3	8.42 (2H,s, -CH=N),7.50-7.86 (4H, m, Ar-H), 3.84 (4H, s,-O-CH_2), 2.52 (2H, s, OH), 1.41 (12H, s,-CH_3)
$[Ru(L1)Cl_2]$	8.47 (2H, s, -CH=N), 6.58-7.70 (12H, m, Ar-H), 4.66(4H, s, $-CH_2$)
$[Ru(L2)Cl_2]$	8.35 (2H, s, CH=N), 8.12 (2H, s, NH- CO), 6.72-8.18 (12H, m, Ar-H), 4.92 (4H, s, Ar-NH_2) 2.21 (6H, $OCOCH_3$)
$[Ru(L3)Cl_2]$	8.46 (2H, s,-CH=N), 7.62-7.92 (4H, m, Ar-H), 4.02 (4H, m, $-CH_2$), 1.74 (12H, s, CH_3)





4a: $[RuL1Cl_2]$ **4b: $[RuL2Cl_2]$** **4c: $[RuL3Cl_2]$**

Scheme.1.Synthesis of Schiff bases and their metal complexes 4(a-c)

CONCLUSION

A simple and efficient method has been developed for the synthesis of tetra dentate Schiff base ligands are obtained from condensation of phthalaldehyde and 2-amino benzyl alcohol, 2-amino-2-methyl-1-propanol and 2-aminobenzohydrazine respectively. These ligands (L1-L3) were reacted with $[RuCl_2(DMSO)_4]$ (Dichlorotetrakis(dimethyl sulfoxide) ruthenium(II)) in methanol to give corresponding Ru(II) complexes. The synthesized ligands and complexes are characterized by elemental analysis, IR, ^{1}H-NMR and mass spectral studies.

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