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Synthesis and spectral studies on monometallic ruthenium (III) complexes of N-(2-hydroxysalicyliden-1-yl)methylenebenzoylhydrazide



N.K. Kar^{a,*}, M.K. Singh^a, R.A. Lal^b

^a Department of Chemistry, Tripura University, Suryamaninagar-799130, Tripura, India

^b Department of Chemistry, North-Eastern Hill University, Shillong-793022, Meghalaya, India

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KEYWORDS

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Abstract A novel Schiff base ligand (H_2L) derived from the condensation of benzoyl hydrazine and salicylaldehyde and its metal complexes with Ru (III) ion has been synthesized and characterized by elemental analyses, thermal analysis (TGA), conductance measurements, magnetic moments IR, UV-vis spectra. The complexes were given the formulae $[Ru(L-H)Cl_2(H_2O)](1), [Ru(L-H)Cl_2(py)]$ (2), [Ru(L-H)Cl₂(2-pic)] (3), [Ru(L-H)Cl₂(3-pic)] (4), [Ru(L-H)Cl₂(4-pic)] (5). Molar conductance in DMF (N,N-dimethyl formamide) solution indicates that the complexes are non-electrolytes. Magnetic susceptibility measurements indicate that all the complexes are mononuclear and one-electron paramagnetic. Electronic spectral studies suggest six coordinate metal ions in its complexes. IR spectra reveal that H₂L ligand coordinates in keto-form to ruthenium metal ion in its complexes. ESR studies of the complexes are also reported.

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1. Introduction

Synthesis and structural characterization studies of ruthenium complexes have become one of the most attractive research fields in modern inorganic chemistry due to its varied properties and applications (Sharma et al., 2006; Mishra et al., 2007; Fouda et al., 2008; Beves et al., 2009; Singh et al., 2008,

Corresponding author. Tel.: +91 9436504154.

E-mail address: nirmalkantikar@gmail.com (N.K. Kar). Peer review under responsibility of King Saud University.



2009a,b; El-Tabl et al., 2010; Thilagavathi et al., 2010; Muthukumar and Viswanathamurthi, 2010; Kar et al., 2012). Ruthenium complexes can form the core of new polymetallic systems that may have applications in excited state energy and electron transfer reactions (Peterson et al., 1985). The oxo complexes of ruthenium have been shown to act as selective oxidants; polypyridyl complexes of ruthenium electrocatalytically oxidize alcohols, phenols, aldehydes and cyclic ketones, even certain C-H bonds such as catalysts in oxidation (Bressan et al., 2000), reduction (Ebadh et al., 2001), and as organic conductors (Pohmer et al., 1996). Ruthenium complexes can act as antioxidant reagents and show high bioactivity (Xu et al., 2012; Huang et al., 2011; Liu et al., 2011). The versatility of ruthenium complexes is due to redox properties, intense electronic absorption bands, and stability,

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Figure 1 Keto- and enol-forms of *N*-(2-hydroxysalicyliden-1-yl)methylenebenzoylhydrazide.

which can be significantly perturbed and advantageously manipulated by subtle changes to the axial ligands (Rawling et al., 2007). As properties are dependent mostly on the coordination environment around the metal centre, complexation of ruthenium by ligand of selected type is of significant importance. H₂L is also an important Schiff base derived from the condensation of benzoylhydrazine and salicylaldehyde. The high affinity for the complexation of the Schiff bases towards the transition metal ions is utilized in preparing their solid complexes. Ruthenium draws particular attention because it presents the four oxidation portions of the popular [Ru(b $py_{3}^{2^{+}}$ sensitized photosystem (Berber and Anderson, 1994). Complexes of ruthenium ion with the titled ligand have been reported by Volkov et al. (2004), Raveendran et al. (2007) and Orysyc et al. (2010) but there is no report on complexes with Ru (III) ion of the present communication type. We are reporting herein the use of ligand N-(2-hydroxysalicyliden-1yl)mehylene-

benzoylhydrazide in preparing octahedral ruthenium (III) complexes. The main target of the present article is to study the coordination behaviour of H_2L Schiff base that incorporates several binding sites towards ruthenium ion. The structure of the ligand used in the present study is shown below in Fig. 1.

2. Experimental

2.1. Materials and reagents

Ruthenium (III) chloride trihydrate (98%), ethyl benzoate (99%), hydrazine hydrate (80%) and salicylaldehyde (98%) were GR (E-Merck) or equivalent grade reagents. Commercial grade ruthenium trichloride was activated by dissolving in concentrated hydrochloric acid and then evaporating to dryness

over steam bath before using it for the synthesis of complexes. Organic solvents used included absolute ethylalcohol, diethylether, methanol, dimethyl- sulphoxide (DMSO) and dimethyl formamide (DMF). Reagent grade organic solvents were purified and dried by recommended procedures (Chan et al., 1995). Hydrochloric and nitric acids (E-Merck) were used. De-ionized water collected from all glass equipments were usually used in all preparations Table 5.

2.2. Instruments

Thermo gravimetric data were obtained in air at 10 °C min⁻¹ using Shimadzu TGA-50H analyser. The molar conductance values of the complexes at 10^{-3} M in DMF solution were measured on a Systronics Direct Reading Conductivity meter-303 with a dip type conductivity cell at room temperature. Magnetic susceptibility measurements were carried out on a Vibrating Sample Magnetometer. Electronic spectra of the complexes were recorded in DMF solution (10^{-3} M) on a Perkin–Elmer Lambda 25 UV-vis spectrophotometer. Infrared spectra were recorded on a Perkin-Elmer IR spectrophotometer in the range $4000-500 \text{ cm}^{-1}$ in KBr discs. The ESR spectra of the complexes were recorded at X-band frequency on a Varian E-112E-line century series ESR spectrometer using TCNE (g = 2.00277) as an internal field marker. Micro analytical (C, H & N) data were obtained with an Elementar Vario EL 1108 at SAIF, Central Drug Research Institute, Lucknow, India. For determining Ru, the complexes were decomposed with aqua regia till all the organic moieties were oxidized. The solution was evaporated to dryness and treated with concentrated HCl. The concentrated HCl solution was also evaporated to dryness and finally the solution was made in distilled water and was used for the estimation of Ru by spectrophotometric method (Snell and Snell, 1959).

2.3. Synthesis of the ligand (H_2L)

The ligand, [*N*-(2-hydroxysalicyliden-1-yl)methylenebenzoylhydrazide] (H₂L) was prepared in two steps. In the first step benzoylhydrazide was prepared by the reaction of ethyl benzoate (48.0 mL) and hydrazine hydrate (16.3 mL) in 1:1 molar ratio under reflux for 4 h. The product thus isolated was recrystallized from hot benzene. Yield: 80%. In the second step, benzoyl hydrazide (5.0 g, 36.746 mmol) in ethanol (50 mL) solu-

Table 1 The analytical data and physical properties of the ligand and its complexes.								
Ligand/complex (colour)	% Yield (dec. temp. °C)	Elementa	al analysis	found (ca	$\mu_{\rm B}$ (B.M)	$\Lambda_{\rm M}~(\Omega^{-1}~{\rm cm}^2~{\rm mol}^{-1})$		
		Ru	С	Н	Ν	Cl		
$H_{2}L(C_{14}H_{12}N_{2}O_{2})$	85.00	-	70.38	5.02	11.58	-	-	-
(light yellow)	(162)		(70.00)	(5.00)	(11.66)			
$[Ru (L-H) Cl_2 (H_2O)] (1)$	78.00	23.24	39.54	3.05	6.80	16.54	1.70	10.00
(brown)	(242)	(23.48)	(39.06)	(3.02)	6.51)	(16.51)		
[Ru (L-H) Cl ₂ (py)] (2)	74.00	20.22	46.50	3.30	8.62	14.80	1.72	12.00
(dark brown)	(240)	(20.57)	(46.43)	(3.25)	(8.55)	(14.46)	1.75	12.8
[Ru (L-H) Cl ₂ (2-pic)] (3)	80.00	19.58	48.00	3.60	8.40	14.20		
(dark brown)	(230)	(20.00)	(47.52)	(3.56)	(8.31)	(14.05)		
[Ru (L-H) Cl ₂ (3-pic)] (4)	75.00	19.68	47.48	3.56	8.36	14.00	1.74	15.00
(dark brown)	(250)	(20.00)	(47.52)	(3.56)	(8.31)	(14.05)		
[Ru (L-H) Cl ₂ (4-pic)] (5)	78.00	19.68	47.65	3.58	8.34	14.25	1.72	14.00
(dark brown)	(248)	(20.00)	(47.52)	(3.56)	(8.31)	(14.05)		

tion was allowed to react with salicylaldehyde (5 mL) in ethanol (20 mL) in 1:1 molar ratio over hot plate at 70 °C with constant gentle stirring for about 30–45 min. The light yellow poly crystalline precipitate thus obtained was purified by repeated washing with hot ethanol and dried over anhydrous CaCl₂. [Yield: ~85%]. (m. p. 162 °C) (Found: C, 70.38; H, 5.02; N, 11.58; Calcd. for C₁₄H₁₂ O₂N₂, C, 70.00; H, 5.0; N, 11.66 (%). λ_{max} (nm), 285 (7200 dm³ mol⁻¹ cm⁻¹), 320 (5030 dm³ mol⁻¹ cm⁻¹) and 378 (4615 dm³ mol⁻¹ cm⁻¹); v (cm⁻¹) 3500–300 (sbr), 3468 (S), 3307 (s), (OH + NH); 1676 (s), 1648 (m) (C=O); 1623 (m), 1613 (m) (C=N); 1581 (m), 1532 (s) [amide II(CO + NH) + (C–O)(phenolic)].

2.4. Synthesis of monometallic complexes

[Ru(L-H)Cl₂(H₂O)](1): To a 20 mL methanolic suspension of the ligand, H₂O (0.458 g, 1.90 mmol), 20 mL methanolic solution of RuCl₃·3H₂O (0.5 g, 1.91 mmol) was added slowly with stirring and refluxing for 3 h. The colour of the reaction mixture changed from reddish to brown and it was filtered. The undissolved material was removed and the filtrate was kept for crystallization which yielded brown precipitate after 5 days. The precipitate so formed was suction filtered, washed with methanol and ether and dried over anhydrous CaCl₂.

[Ru(L-H)Cl₂(A)] [where, A = py (2), 2-pic (3), 3-pic (4), 4-pic (5)]: The complexes 2–5 were isolated by essentially following the same procedure used for the preparation of complex 1 by adding pyridine bases such as, pyridine (py), 2-picoline (2-pic), 3-picoline (3-pic) and 4-picoline (4-pic) to the reaction mixture containing H₂L, RuCl₃·3H₂O and pyridine base in 1:1:5 molar ratio and refluxing for 3 h.

3. Results and discussion

3.1. Composition and structures of the monometallic complexes

All the complexes are insoluble in common organic solvents but soluble in DMF and DMSO and melt with decomposition in the temperature range 230–250 °C. The analytical data and stoichiometry of the complexes reveal complexes of compositions [Ru(L-H)Cl₂(H₂O)] (1), [Ru(L-H)Cl₂(py)](2), [Ru(L-H)Cl₂(2-pic)] (3), [Ru(L-H)Cl₂(3-pic)] (4), [Ru(L-H)Cl₂(4-pic)] (5). Thermal analyses (TGA) carried out under N₂ atmosphere giving information about the stability of metal chelates and decide to some extent whether the water molecules are inside or outside the coordination sphere. In TGA analysis, the complexes do not display any weight loss up to 165 °C indicating the non existence of lattice water molecule. In all the complexes, decomposition involved two steps: the first step indicated loss of two chloride ions in the temperature range 270–320 °C, while the second was observed at 380– 480 °C range giving Ru₂O₃, consistent with +3 oxidation state of ruthenium. The isolated complexes obtained from Ru (III) ion with Schiff base H₂L were subjected to elemental analyses (C, H, N and metal content), molar conductance, magnetic studies, IR, electronic and ESR, to identify their tentative formulae in a trial to elucidate their molecular structures. The analytical data and stoichiometries of the complexes are summarized in Table 1.

3.2. Molar conductivity measurements

The complexes were dissolved in DMF and the molar conductivities of 10^{-3} M of their solutions at room temperature were measured. The molar conductance values of the complexes fall in the range 10-15 S mol⁻¹ cm² indicating the non-electrolytic nature of the complexes (Singh et al., 2008).

3.3. Magnetic susceptibility measurements

Ruthenium, being a metal of second transition series will always give a low spin complex with μ_{eff} 1.80 B.M (Das et al., 2000). The μ_{eff} values for the synthesized complexes lie in the range 1.70–1.75 B.M. with d^5 configuration ($S = \frac{1}{2}$) (Singh et al., 2009a,b). Thus, the μ_{eff} values for the complexes are in accord with the +3 oxidation of ruthenium.

3.4. Electronic spectral measurements

The nature of the ligand field around the metal ion has been deduced from the electronic spectra. UV-vis spectra of the ligand and its complexes were recorded in DMF solution (10^{-3} M) and relevant data are given in Table 2. In the present study the ligand shows four absorption bands at 232 (7720), 285 (7640), 320 (5200) and 378 nm (4714 L mol⁻¹ cm⁻¹) due to carbonyl and imino chromophores in conjugation with phenyl group. Complexes 1-5 show two bands in the region 430-450 and 660-680 nm, respectively, in addition to intra-ligand bands with high molar extinction coefficients. The band in the range 430-450 nm may be assigned to ligand to metal charge transfer (LMCT) transition (Singh et al., 2009a,b) while the bands in the region 660-680 nm have tentatively been assigned to spin allowed ${}^{1}A_{1g} \rightarrow {}^{1}T_{1g}$ transition (Dharamraj and Natarajan, 1994). The *d*-*d* transition bands ${}^{2}T_{2g} \rightarrow {}^{4}T_{1g}$, $^{2}T_{2g} \rightarrow ^{4}T_{2g}$ and $^{2}T_{2g} \rightarrow ^{2}T_{1g}$ are masked by strong LMCT bands (Lever, 1999).

3.5. Infrared spectral studies

Infrared spectral data of the Schiff base ligand and its metal complexes are listed in Table 3. IR spectrum of the H_2L ligand

Table 2 UV-vis spectral data of the ligand and its complexes.

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Ligand/complex	Wave length (λ_{max} , nm) (ε_{max} , L mol ⁻¹ cm ⁻¹)
$H_{2}L(C_{14}H_{12}N_{2}O_{2})$	232 (7720), 285 (7640), 320 (5200), 378 (4714)
$[Ru (L-H) Cl_2 (H_2O)] (1)$	675(3500), 440(2450), 378 (5400), 322 (4100), 282 (6250)
[Ru (L-H) Cl ₂ (py)](2)	660 (3600), 432 (2600), 390 (6100), 320 (5320)
[Ru (L-H) Cl ₂ (2-pic)] (3)	665 (2840), 450(2450), 380 (6230), 290 (7400)
[Ru (L-H) Cl ₂ (3-pic)] (4)	670 (3100), 445(2380), 384 (6250), 296 (7350)
[Ru (L-H) Cl ₂ (4-pic)] (5)	680 (2800), 436(2550), 3392 (6200), 300 (7300)

Ligand/complex	ν (OH) + ν (NH)	ν (C==O)	ν (C==N)	AmideII + v (C–O) (phenolic)	β (C–O)	ν (M–O)	v (M–N) pyridinebase
H ₂ L (C ₁₄ H ₁₂ N ₂ O ₂)	3500–3100 (sbr), 3468 (s)	1676 (s), 3307 (s)	1623 (m), 1648 (m)	1581 (m), 1613 (m)	1352 (s), 1532 (s)	– 1276 (s)	-
[Ru (L-H) Cl ₂ (H ₂ O)] (1)	3500–3200 (sbr), 3465 (s), 3130 (sbr)	1797 (s), 1645 (s)	1595 (s)	1510 (s)	1375 (vs) 1245 (s)	645 (m)	-
$[Ru (L-H) Cl_2 (py)] (2)$	3500–3200 (sbr), 3460 (s), 3120 (s)	1790 (s), 1623 (s)	1580 (s)	1479 (vs)	1275 (s) 1211 (s)	650 (s)	1025 (s)
[Ru (L-H) Cl ₂ (2-pic)] (3)	3500–3200 (sbr), 3450 (s), 3116 (s)	1760 (vs), 1630 (s)	1585 (s)	1490 (s)	1310 (s) 1240 (m)	620 (m)	1022 (s)
[Ru (L-H) Cl ₂ (3-pic)] (4)	3500–3200 (sbr), 3445 (s), 3104 (s)	1756 (vs), 1640 (s)	1590 (s)	1520 (s)	1350 (vs) 1240 (m)	670 (s)	1020 (m)
[Ru (L-H) Cl ₂ (4-pic)] (5)	3400–3200 (sbr), 3458 (s), 3119 (s)	1748 (vs), 1646 (s)	1591 (vs) 1500 (s)	1373 (vs)	690 (vs) 1330 (m)	1020 (s)	
s strong vs verv strong shr strong broad m medium							

Table 3 FT-IR (KBr, cm^{-1}) spectral data of the ligand and its complexes.

 Table 4
 ESR parameters of monometallic complexes.

Complex	g-Values ((LNT powder)			g-Values	g-Values (LNT DMF)			
	g_1	g_2	g_3	g_{av}	g_1	g_2	g_3	g_{av}	
[Ru (L-H) Cl ₂ (H ₂ O)] (1)	-	_	_	_	2.260	2.082	1.864	2.068	
[Ru (L-H) Cl ₂ (3-pic)] (4)	3.001	2.404	1.980	2.128	2.500	2.048	1.750	2.099	

 Table 5
 Thermal analysis (TGA) results of complexes.

Complex	Temp. range (°C)	Mass loss% Found (calcd.)	Assignment	Metallic residue
[1] [Ru(L-H)Cl ₂ (H ₂ O)]	270–320	20.88 (21.21)	Loss of H ₂ O and 2HCl	–
	380–480	55.22 (55.47)	Loss of L	Ru ₂ O ₃
2. [Ru(L-H)Cl ₂ (py)]	270–320 380–480	14.70 (14.89) 64.50 (64.69)	Loss of 2HCl Loss of L and py	$-Ru_2O_3$
3. [Ru(L-H)Cl ₂ (2-pic)]	270–320 380–480	14.50 (14.48) 65.55 (65.67)	Loss of 2HCl Loss of L and 2-pic	$-Ru_2O_3$
4. [Ru(L-H)Cl ₂ (3-pic)]	270–320	14.44 (14.48)	Loss of 2HCl	-
	380–480	65.50 (65.67)	Loss of L and 3-pic	Ru ₂ O ₃
5. [Ru(L-H)Cl ₂ (4-pic)]	270–320	14.42 (14.48)	Loss of 2HCl	-
	380–480	65.60 (65.67)	Loss of L and 3-pic	Ru ₂ O ₃

exhibits the most characteristic bands at 3468 cm^{-1} v (OH), $3307 \text{ cm}^{-1} \text{ v} (\text{N-H}), 1776 \text{ cm}^{-1} \text{ v} (\text{C=O}), 1623 \text{ cm}^{-1} \text{ v} (\text{C=N})$ and 1581 cm⁻¹ v (C–O). All the metal complexes showing a broad band in the region $3500-3200 \text{ cm}^{-1}$ may be assigned to either v (O-H) or v (N-H). A medium intensity band at 796 cm⁻¹ suggesting the presence of coordinated water in Ru (III) complex 1. The amide-I bands for H₂L (Kar, 2011) observed at 1776 cm^{-1} shifted to higher frequency by 21 cm⁻¹ in complex 1 while shifted to lower frequencies $(16-32 \text{ cm}^{-1})$ in complexes 2-5 suggest the bonding of carbonyl oxygen. The band 1623 cm⁻¹ due to the azomethine group of Schiff base (Singh et al., 2008) has shifted to lower frequency (1591-1595 cm⁻¹) after complexation, indicating the bonding of nitrogen of the azomethine group to the metal ion (Husain et al., 1991) that can be explained by the donation of electrons from nitrogen to the empty *d*-orbital of the metal atom. The amideII band in H_2L is observed at 1581 cm⁻¹ undergoing negative shift in all the complexes suggesting coordination of >C=Oto the metal (Singh et al., 2009a,b). The phenolic C–O stretching vibration that appeared at 1532 cm^{-1} in Schiff base shift towards higher frequency $(20-32 \text{ cm}^{-1})$ in the complexes, indicating the participation of oxygen in the C-O-M bond. Reaction of ruthenium (III) chloride with ligand liberates HCl reducing its $p^{\rm H}$, which suppresses enolization allowing coordination in keto-form to ruthenium. Complexes show a new strong band at 1020–1025 cm⁻¹ probably arising from ring breathing suggesting the presence of pyridine, 2-picoline, 3-picoline or 4-picoline in the complexes. Non-ligand band observed in the region 645-690 cm⁻¹ has been assigned to M-O mode in these complexes (Percy, 1975). The bands at 344 and 333 cm⁻¹ are assigned to Ru-Cl bond. The IR spectra reveal that, the H₂L act as an anionic tridentate ligand towards Ru (III).



Figure 2 The proposed structure of $[Ru(L-H)Cl_2(A)]$ [where $A = H_2O(1)$, py(2), 2-pic(3), 3-pic(4), 4-pic(5)].

3.6. Electron spin resonance spectra

ESR Spectra of the complexes 1 and 4 were studied as representative case at RT (room temperature) and LNT (liquid nitrogen temperature) in polycrystalline state as well as at LNT in DMF and the data are listed in Table 4. ESR spectrum of 1 at RT (Powder) consists of a single isotropic resonance with un-resolved hyperfine structure due to ruthenium with g value equal to 2.054 but at LNT (Powder), lines slightly narrowed and a slight change in the g-parameter (2.052) with no g anisotropy was observed. But complex 4 shows poorly resolved features at LNT (Powder) but are featureless at RT (Powder). Complex 1 shows well-resolved spectral features at LNT in DMF glass. Thus, in complex 1 the spin exchange may not be the main reason for isotropy while the occupancy of unpaired electron is in a low spin state with a degenerate or nearly degenerate ground state, prone to Jahn-Teller instability (Jahn and Teller, 1937). Complex 4 shows well-resolved spectral features at LNT in DMF solution. The g_{av} values vary with axial ligand. The nature of the spectra is consistent with the non-degenerate t_{2g} orbital. Hyperfine coupling due to ruthenium was resolved on g1 components in DMF solution at LNT which is equal to 74G. The above results indicate that the complexes belong to a low spin $4d^5$ configuration with Ru (III) in octahedral symmetry (Dasgupta et al., 2008).

4. Conclusion

The stoichiometries and physico-chemical studies reveal the formation of monometallic complexes of Ru (III) metal ion. The ligand coordinates to the metal centre in keto-form and acts as an anionic tridentate ligand towards metal ion. Octahedral stereochemistry around the metal ion has also been tentatively proposed as shown in Fig. 2.

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