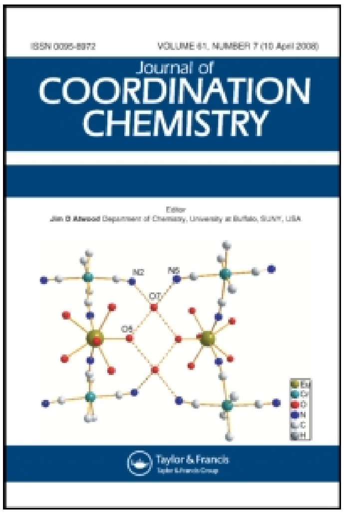

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Synthesis, Characterization and reactivities of Schiff-base complexes of Ruthenium(III)

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SYNTHESIS, CHARACTERIZATION AND REACTIVITIES OF SCHIFF-BASE COMPLEXES OF RUTHENIUM(III)

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Schiff-base complexes of ruthenium (**1**–**5**) have been synthesized using Schiff-base ligands derived by condensation of either 1,2-phenylenediamine with aldehydes (salicyldehyde, 2-pyridinecarboxaldehyde) or acetylacetone with amines (2-aminophenol, 2-aminomethylpyridine). All complexes were characterized by analytical, spectroscopic, conductance, magnetic moment and electrochemical studies. At room temperature, complexes **1**–**5** catalyze the oxidation of both saturated and unsaturated hydrocarbons using *tert*-butylhydroperoxide (*t*-BuOOH). A mechanism involving formation of and transfer from a reactive high valency Ru(V)-oxo species as the catalytic intermediate is proposed for the processes.

Keywords: Ruthenium complex; Schiff-base ligands; *t*-BuOOH; Oxo transfer catalysis

INTRODUCTION

The chemistry of metal complexes containing *salen*-type Schiff-base ligands derived from condensation of aldehydes and amines is of enduring significance, since they have common features with metalloporphyrins with respect to their electronic structures and catalytic activities that mimic enzymatic hydrocarbon oxidation [1]. Catalytic activity of such metal complexes has been highlighted in the past few decades [2–12]. Although a variety of polypyridyl-, macrocyclic- and pac (polyaminopolycarboxylate)-based ruthenium complexes are known [13–29] to catalyze olefin epoxidation and many other organic oxidations in the presence of O_2, H_2O_2, ClO^-, pyridine-*N*-oxide, iodosyl benzene, oxone or *t*-BuOOH, reports on studies with ruthenium complexes containing salen-type Schiff-base ligands in the catalysis of hydrocarbon oxidation are noticeably few in the literature [30,31]. As a part of our interest in developing non-porphyrinic ruthenium(III) catalyst complexes [32–37] that exhibit selectivity toward oxo-functionalization of saturated and unsaturated hydrocarbons, we have undertaken the present investigation with the objective of realizing the catalytic properties of ruthenium(III)–Schiff-base complexes towards hydrocarbon oxidation

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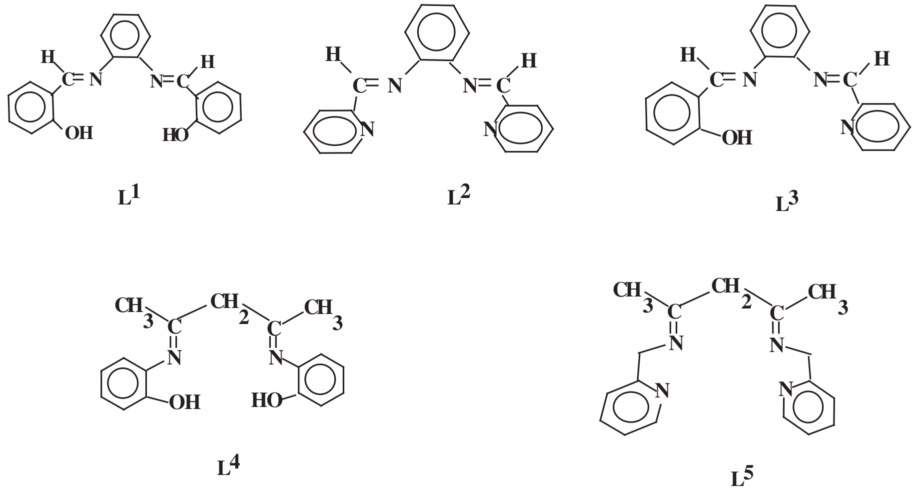

L^1 = 1,2-diaminophenyl-N,N'-bis(salicyaldimine)
L^2 = 1,2-diaminophenyl-N,N'-bis-(2-pyridinecarboxaldimine)
L^3 = 1,2-diaminophenyl-N-(salicyaldimine),N'-(2-pyridinecarboxaldimine)
L^4 = N,N'-bis (2-hydroxyphenylene)dimineacetylacetonate
L^5 = N,N'-bis (2-aminomethylpyridine)dimineacetylacetonate

FIGURE 1 Pictorial representation of the Schiff-base ligands.

in a comprehensive manner. We report herein the syntheses, characterization and efficacy of ruthenium(III)-complexes towards catalyzing hydrocarbon oxidation with regards to solvent and terminal oxidants. Schiff-base ligands (Fig. 1) have been derived by condensation of either 1,2-phenylenediamine with aldehydes (salicyldehyde, 2-pyridinecarboxaldehyde) or acetylacetone with amines (2-aminophenol, 2-aminomethylpyridine).

EXPERIMENTAL

Synthesis of Ruthenium Catalyst Complexes

Syntheses of ruthenium(III) complexes were carried out by interacting $RuCl_3$ and the appropriate ligand in a stoichiometric ratio under reflux conditions.

Dichloro-1,2-diaminophenyl-N,N′-bis(salicyaldimine)ruthenate(III) (1)

The Schiff-base ligand *saloph* (1,2-diaminophenyl-*N*,*N′*-bis-salicyaldimine) was synthesized by condensation of salicyaldehyde and *o*-phenylenediamine in a 2 : 1 stoichiometric ratio and collected as a yellow solid. A solution of 1 mmol (0.316 g) of saloph in alkaline ethanol (2 mmol of KOH in 25 cm^3 of ethanol) was added dropwise to an ethanolic solution (25 cm^3) of $RuCl_3$ (1 mmol, 0.207 g). Equivalent amounts of

KOH were added to deprotonate the phenolic OH group of the Schiff-base ligand. The resultant mixture was stirred for 30 min and then kept under reflux for 8 h. The greenish-brown solution that formed was evaporated to dryness over a waterbath, the solid mass collected, washed with 1 : 9 methanol–water to remove salts, diethylether, petroleum ether and dried over calcium chloride in a desiccator (yield: 70%). Anal. Calcd. (found) for $KRuC_{20}H_{14}N_2O_2Cl_2$ (%): C, 45.7 (45.3); H, 2.6 (2.3); N, 5.3 (5.1).

Dichloro-1,2-diaminophenyl-N,N′-bis-(2-pyridinecarboxaldimine)ruthenium(III) (2)

To a solution of *o*-phenylene diamine (1 mmol, 0.108 g) in dry ethanol (25 cm^3) was added a solution of 2-pyridine carboxaldehyde (2 mmol, 0.2 cm^3) in ethanol (25 cm^3) and the resultant mixture was magnetically stirred at room temperature for 1 h. The Schiff-base ligand 1,2-diaminophenyl-*N*,*N′*-bis-(2-pyridinecarboxaldimine) so formed was added dropwise to the ethanolic solution (50 cm^3) of $RuCl_3$ (1 mmol) and the reaction mixture was refluxed for 10 h. The resultant solution was evaporated to dryness, solid mass collected, washed with 1 : 9 methanol–water and petroleum ether and dried in a desiccator loaded with calcium chloride (yield: 78%). Anal. Calcd. (found) for $RuC_{18}H_{14}N_4Cl_3$ (%): C, 43.8 (44.1); H, 2.8 (2.5); N, 11.3 (11.5).

Dichloro-1,2-diaminophenyl-N-(salicyaldimine)-N′-2-pyridinecarboxaldimine) ruthenium(III) (3)

A similar synthetic procedure to that described for complex **2** was adopted for the synthesis of the Schiff-base ligand 1,2-diaminophenyl-*N*-(salicyaldimine)-*N′*-(2-pyridinecarboxaldimine). Magnetic stirring (1 h at room temperature) of the reaction mixture containing *o*-phenylenediamine (1 mmol), salicyaldehyde (1 mmol) and 2-pyridinecarboxaldehyde (1 mmol) produced the desired Schiff base. Equivalent amounts of KOH (1 mmol) were added to the ligand solution prior to the addition of $RuCl_3$. Complex **3** was obtained after 10 h reflux of the ligand solution containing 1 mmol of ruthenium chloride. Work-up was effected using the procedures described above (yield: 75%). Anal. Calcd. (found) for $RuC_{19}H_{14}N_3OCl_2$ (%): C, 48.3 (47.9); H, 2.9 (3.1); N, 8.9 (9.1).

Dichloro-N,N′-bis-(2-hydroxyphenylene)diimineacetylacetonatoruthenate(III) (4)

Acetylacetone (1 mmol, 0.1 cm^3) was added dropwise to a methanolic solution of 2 mmol (0.2183 g) of 2-aminophenol, and the mixture was stirred at room temperature for 1 h. The Schiff-base ligand *N*,*N′*-bis-(2-hydroxyphenylene)dimineacetylacetone so formed was added dropwise to an ethanolic solution (50 cm^3) of $RuCl_3$ (1 mmol) and the reaction mixture was refluxed for 10 h (2 mmol of KOH was added during that period to deprotonate the phenolic OH groups of the ligand). A dark-brown solution resulted and this was then evaporated to dryness over a waterbath. The brown solid mass was then collected, washed with 1 : 9 methanol–water and petroleum ether and dried over calcium chloride in a desiccator (yield: 70%). Anal. Calcd. (found) for $KRuC_{17}H_{16}N_2O_2Cl_2$ (%): C, 41.5 (40.8); H, 3.25 (3.15); N, 5.7 (5.5).

Dichloro-N,N′-bis-(2-aminomethylpyridine)diimineacetylacetonatoruthenium(III) (5)

Complex **5** was synthesized in a similar manner except that 2-aminomethylpyridine was used in place of 2-aminophenol. The brown solid product was washed with 1 : 9 methanol–water and petroleum ether and dried over calcium chloride in a desiccator. (yield: 70%). Anal. Calcd. (found) for $RuC_{17}H_{20}N_4Cl_3$ (%); C, 41.8 (41.2); H, 4.1 (3.9); N, 11.5 (11.8).

All other chemicals and solvents were of AR grade and used as obtained. Doubly distilled water was used throughout the experiments.

Instrumentation

Electronic absorption spectra were measured with a GBC Cintra 10 spectrophotometer. Infrared spectra were recorded on a Perkin Elmer 783 spectrometer using KBr pellets. Electrochemical studies were carried out in acetonitrile using tetraethylammonium perchlorate (TEAP) as supporting electrolyte. A PAR electrochemical unit (Model 174A) equipped with a platinum working electrode and Ag/AgCl as reference electrode was used for this purpose. All potentials are expressed against SCE. Magnetic susceptibility was measured using a PAR-155 vibrating sample magnetometer. A Perkin-Elmer 240C instrument was used to collect microanalytical data (C, H, N).

Procedure for Catalytic Studies

In a typical experiment 0.01 mmol of catalyst complex, 0.1 mmol of benzyltributylammonium chloride (phase transfer catalyst, PTC) and 1.0 mmol of substrate in 10 cm^3 of CH_2Cl_2 were stirred with 1.00 mmol of oxidant at room temperature for 4 h. Aliquots of the CH_2Cl_2 layer were withdrawn and subjected to gas chromatographic (GC) analysis for product(s). Gas chromatographic analyses were performed with a Carlo Erba GC 8000 series instrument on a Tenax column with an FID detector. GC parameters were quantified by authentic samples prior to analysis.

RESULTS AND DISCUSSION

Characterization of Ruthenium Complexes

Electronic spectra of complexes **1**–**5** in CH_3OH are characterized by charge-transfer bands. Bands below 300 nm are intraligand charge-transfer transitions; bands in the visible region are assigned to ligand-to-metal charge transfer ($\pi \rightarrow t_{2g}$ origin). The basis of assignment is earlier reports [30,38] of data for ruthenium(III) complexes containing Schiff-base ligands. IR spectra of the complexes displayed bands typical of coordinated Schiff-base ligands (viz, 1620–1640 cm^{-1} for C=N stretch). Magnetic moments of complexes **1**–**5** conform to low-spin d^5 configurations (idealized t_{2g}^5, $S = 1/2$). Conductance data established the 1 : 1 electrolyte nature of complexes **1**, **2**, **4** and **5** whereas complex **3** is a non-electrolyte in solution.

Complexes **1**–**5** were electrochemically examined at a platinum working electrode in acetonitrile solution. Cyclic voltammograms of complexes **1**–**4** in acetonitrile displayed *quasi*-reversible Ru^{III}/Ru^{II} redox couples with peak-to-peak separations (ΔE_p) in the range 80–120 mV. $E_{1/2}$ values corresponding to Ru^{III}/Ru^{II} redox couples estimated

TABLE I Spectroscopic, conductivity and magnetic susceptibility data for complexes **1**–**5**

Complex	*UV-vis* λ_{max}, nm (e_{max}, $M^{-1}cm^{-1}$)	*IR* (cm^{-1})	Λ_M *in* H_2O *at* 25°C (Ω^{-1} mol^{-1} cm^2)	μ_{eff} (BM)	$E_{1/2}(Ru^{III}/Ru^{II})$ (*V vs Ag/AgCl*)
1	485 (4500), 358 (8373), 279 (11550)	1660, 1601, 1520, 1458, 1306, 1149, 1028, 926, 752, 667, 536, 469, 305	104	1.96	−0.68
2	480 (3365), 320 (9670), 243 (8376)	3421, 1604, 1475, 1442, 1324, 1282, 1228, 1151, 1058, 1012, 748, 690, 647, 457, 325	111	1.98	−0.48
3	482 (2315), 324 (8150), 242 (19000)	1602, 1475,1303, 1149, 1012, 750, 451	65	1.96	−0.55
4	528 (3305), 392 (9650), 279 (13 100)	2923, 1747, 1660, 1602, 1520, 1475, 458, 1303, 1149, 1012, 750, 459, 320	82	1.97	−0.73
5	500 (3151), 358 (9300), 270 (24 000)	3408, 1872, 1699, 1468, 1429, 1228, 1155, 764, 451, 324	85	1.98	−0.56

from cyclic voltammetric studies are summarized in Table I. $E_{1/2}$ values of complexes containing phenolato donors (**1** and **3**) are more cathodic than for complexes containing pyridyl-*N* donors (**2** and **4**). Coordination of phenolato O atoms makes the metal centre more electron rich and shifts the oxidation potential towards more negative values as observed in the present case.

Catalysis of Hydrocarbon Oxidation

The catalytic properties of complexes **1**–**5** were examined under a standard set of conditions (see Experimental Section for reaction conditions). Preliminary experiments ascertained the necessity of each component to observe catalytic activity. The influence of solvent on product yield was evaluated for CH_2Cl_2, CH_3CN and 1,4-dioxane. It has been found that a coordinating solvent (CH_3CN) or larger solvent molecules suppress reactivity by inhibiting coordination of *t*-BuOOH, and compete for the Ru^V=O group through their own intrinsic C–H reactivity. The lower yield observed in the case of dioxane in comparison to CH_2Cl_2 is presumably associated with solvation effects of the larger dioxane molecules which impinge on the accessibility of the Ru(V)-oxo moiety to substrate approach. The effect of change of precursor oxidants (PO), often referred to in the catalytic literature as terminal oxidants (TO), has been studied using various O-atom transfer agents (*t*-BuOOH, H_2O_2, NaOCl, $KHSO_5$ and pyridinium-*N*-oxide). The results of the preliminary experiments suggest that catalytic efficiency of the **1**–**5**/**PO** system is best when *t*-BuOOH is used as terminal oxidant. H_2O_2 was found to be less reactive, while NaOCl and $KHSO_5$ were found to be inert. Complexes **1**–**5** undergo decomposition (probably base hydrolysis coupled with oxidative degradation) at high pH in the presence of NaOCl or $KHSO_5$. Slow decomposition of **1**–**5** was also observed in the case of H_2O_2. A Fenton-type process might be operative in the case of hydrogen peroxide, resulting in the formation of hydroxyl radicals that attack the coordinated Schiff-base ligands causing oxidative

TABLE II Results of catalytic oxidation of organic substrates with *t*-BuOOH[a]

Substrate	*Product*	*1*	*2*	*3*	*4*	*5*
Cyclohexene	Cyclohexene-1-ol	28	36	30	19	25
	Cyclohexene-1-one	20	26	22	13	17
	Cyclohexene epoxide	2	2	3	2	3
Cyclohexane	Cyclohexanol	17	23	21	11	14
	Cyclohexanone	13	15	14	7	12
Cyclohexanol	Cyclohexanone	32	39	31	28	35
Toluene	Benzyl alcohol	21	35	28	26	31
	Benzaldehyde	18	21	20	18	20
Benzyl alcohol	Benzaldehyde	48	53	47	50	55
Tetrahydrofuran	γ-Butyro-lactone	36	48	38	37	44

[a] See Experimental Section for reaction conditions.

decomposition of the complexes. It is noted that while H_2O_2 is a cheap, easy-to-handle and environmentally acceptable oxidant, reports on the use of H_2O_2 as an oxidant for hydrocarbon oxidation catalyzed by ruthenium complexes are conspicuously absent, except for polypyridyl-based ruthenium complexes; these complexes are stable and do not undergo oxidative degradation in the presence of H_2O_2 [13,15].

Oxidation of Unsaturated and Saturated Hydrocarbons with *t*-BuOOH Catalyzed by 1–5

Based on the above experimental facts, CH_2Cl_2 and *t*-BuOOH were chosen as optimum solvent and terminal oxidant, respectively, for carrying out catalyzed oxidation of the organic substrates. It is worth mentioning here that safety and environmental concerns have attached special importance to the catalytic scheme with alkyl hydroperoxides. Results of catalytic oxidation are summarized in Table II. The most significant aspect of the present catalytic system is that it selectively oxo-functionalizes the C–H bond of both unsaturated and saturated hydrocarbons. Cyclohexene is converted to cyclohexene-1-ol in appreciable yields. It is capable of oxidizing cyclohexane to cyclohexanol, toluene to benzylalcohol and tetrahydrofuran to γ-butyrolactone. It also readily oxidizes cyclohexanol to cyclohexanone and benzyl alcohol to benzaldehyde. Comparison of hydroxyalated products noted in the present studies with epoxide formation reported during olefin oxidation by the Ru(III)-Schiff base/PhIO system [30] obviously suggests that the nature of catalytically active intermediate in the two cases is different. Though an Ru(V)-oxo species had been proposed earlier as an active intermediate in the Ru(III)-salen/PhIO catalytic system [30], formation of an Ru-O-I-Ph adduct akin to that reported [39,40] in the case of the manganese porphyrin/PhIO system could not explicitly be ruled out by the authors [30]. Ru^V(Schiff-base)-oxo species seem to be labile as attempts to isolate them in the crystalline state have not succeeded. However, formation of an Ru(V)-oxo species in the present work is supported by IR studies. IR spectra of the solid mass obtained by evaporation of the solution obtained by interacting the catalyst complex and *t*-BuOOH exhibited characteristic Ru(V)-oxo bands [28,41–43] in the range 860–872 cm^{-1}, and which were not found in IR spectra of precursor complexes **1**–**5**. However, other data were found to be similar to those noted in the IR spectra of the precursor complexes. This essentially indicates that the coordinated Schiff-base ligands in complexes **1**–**5** remain unchanged during the oxidation under specified conditions. The same solid mass (Ru^V-oxo species) as obtained above was allowed to interact with cyclohexene in

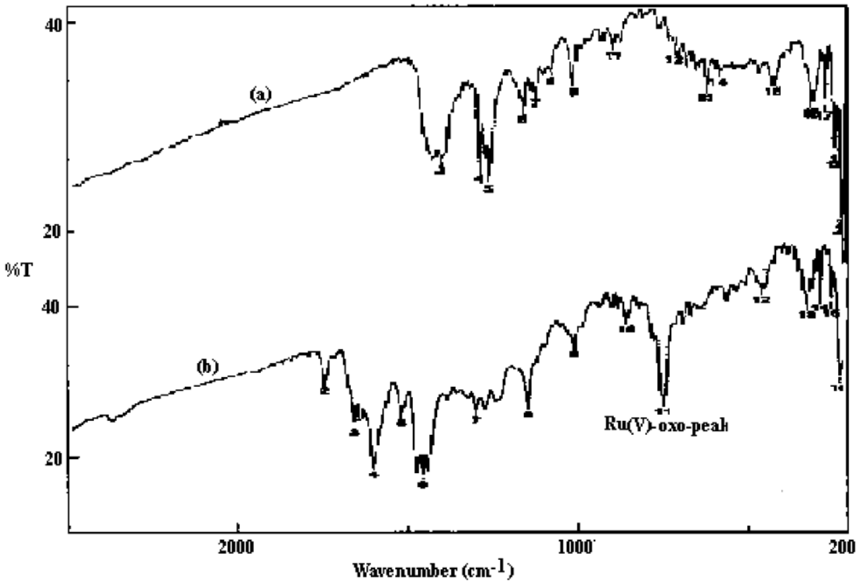

FIGURE 2 IR spectra of (a) complex **2** and (b) the solid mass obtained by interacting complex **2** with *t*-BuOOH.

CH_2Cl_2 for 4 h at room temperature and gas chromatographic analysis of the resultant mixture revealed the formation of cyclohexene-1-ol/1-one product(s). Since both $[Ru^{III}LCl_2]$/*t*-BuOOH catalytic system and $[Ru^{V}(L)(O)]$ (L = Schiff base) oxidize cyclohexene to cyclohexene-1-ol, it seems that the two systems react similarly and that the $[Ru^{V}(L)(O)]$ complex species is the active intermediate. Further, the stable Ru(V)-oxo complex is mostly selective towards hydroxylation of the C–H bond, rather than epoxidizing the C=C bond, as substantiated in several other reports [8,43,44]. For example, $Ru^{V}(N_4O)(O)]^{2+}$ hydroxylates cyclohexene to produce cyclohexene-1-ol/cyclohexene-1-one [43], whereas the $Ru^{III}(N_4O)(H_2O)]^{2+}$-PhIO system selectively epoxidizes olefins [21]. Ru-O-I-Ph adduct species as probable catalytic intermediates have been proposed in the latter case.

The results in Table II show that catalyst complexes **2** and **5** (N4 coordination environment) are slightly more active than **1** and **4** (N2O2 coordination environment). Action of the phenolato donors might imply a less oxidizing "Ru^{V}=O" in complexes **1** and **4**. An important consequence of a less oxidizing Ru centre is that the tendency for reduction via outer-sphere electron transfer is reduced. Thus there is less tendency for radical pathways in the redox mechanisms for O transfer. However, the Ru-O unit in **1** and **4** may still attack the C–H bond of cyclohexene with a "side-on" approach [45].

Comparison of catalytic activity of M-Salen/PhIO systems (M = Mn [5], Ni [6], Cr [4], Ru [30]) reveals that Mn complexes are the most efficient epoxidation catalysts, whereas the Ni-complex is completely inactive with PhIO [6]. The efficiency of the Ru-salen/PhIO system [30] is poor compared to its Mn analogue. However, in all cases [3,4,6,30], formation of hydroxylated product was negligible. However, the results of the present studies clearly reveal that the use of *t*-BuOOH as the terminal oxidant significantly changes the catalytic activity of Ru(III)–Schiff-base complexes. The catalytic system becomes highly selective with regards to C–H bond activation of both unsaturated and saturated hydrocarbons by producing hydroxylated products in appreciable yield. The results are quite prospective for reaching the level of selectivity achieved by cytochrome P-450 in oxidative metabolism.

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