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Short communication

Ru(III)-catalyzed oxidation of pyridoxine and albuterol in pharmaceuticals

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ABSTRACT

Ru(III) complexes with coordinated amide were synthesized and characterized by elemental, IR, mass, electronic, ESR spectral analysis, magnetic and conductance measurements and octahedral structures have been proposed. These complexes were used as catalysts for the oxidation of pyridoxine and albuterol in pharmaceuticals in presence of hydrogen peroxide. The role of co-oxidant and the effect of reaction time on the yields of oxidation products which were spectrophotometrically determined by condensing them with sulfanilic acid in acid medium were investigated. Structures of the oxidation products were established with the help of IR and NMR spectral analysis.

1. Introduction

The development of ruthenium-catalyzed methods [1] for the oxidation of primary [2] and secondary [3] alcohols to produce carbonyl derivatives remains an important one in pharmaceutical products [4]. But, these methods require either the presence of a co-oxidant [5] or a prolonged oxidation time [6] leading to environmental problems. Pyridoxine [7], the first isolated vitamin B6, is oxidized to pyridoxal in biological system. Albuterol [7] is a beta-adrenergic stimulant which has a highly selective action on the receptors in bronchial smooth muscle, there by causing relaxation of bronchial muscle fibres. In literature, some oxidation methods of pyridoxine as hydrochloride (5-hydroxy-6-methyl-3,4-pyridine dimethanol, hydrochloride, PYE) to pyridoxal (3-hydroxy-5-(hydroxy methyl)2-methyl-4-pyridine carboxaldehyde, PYL) [8] and albuterol (α^1[(*tert*-butyl amino)methyl]4-hydroxy *m*-xylene α,α^1-diol sulfate, 2:1 salt, AB) to its oxidation product (5-[(*tert*-butyl amino)acetyl]-2-hydroxy benzaldehyde, OPAB) [9] were reported. Even though some complexes were synthesized using the $[RuCl_3(PPh_3)_3]$ [10–12], no amide complex of this precursor was synthesized so far. Hence, Ru(III) complexes with coordinated amide of the type $RuCl_2(PPh_3)_2(L_2)$ (Complex-1–12) were synthesized using the precursor $[RuCl_3(PPh_3)_3]$ and used as catalysts for the oxidation of pyridoxine and albuterol (in pure form and pharmaceutical formulations) in presence of hydrogen peroxide with lesser oxidation times without using a co-oxidant. PYL and OPAB were condensed with sulfanilic acid (SA) in acid medium [13] for their spectrophotometric determination.

2. Experimental

2.1. Instruments

The melting points of all the ligands and complexes were determined on a Buchi-510 melting point apparatus. The percentages of carbon, hydrogen, nitrogen were determined using a PerkinElmer CHN analyzer. The IR spectra were recorded in KBr pellets on PerkinElmer-283 spectrophotometer. The scanning rate was 6 min in the range of 4000–200 cm^{-1}. MICROMASS-7070 spectrometer operating at 70 eV using a direct inlet system was used for mass spectra. UV–visible spectra were recorded with Shimadzu UV-160A, a UV–visible double beam spectrophotometer with matched quartz cells of path length 1 cm. ESR spectra were recorded on JEOL-JES-FE-3X spectrometer. Gouy balance calibrated with $Hg[Co(NCS)_4]$ was used for the determination of magnetic susceptibilities of complexes in solid state at room temperature. Conductance measurements were done on 10^{-3} M solution of compounds in dichloromethane at room temperature using Digisun Digital conductivity meter model DL-909.

2.2. Materials and reagents

$RuCl_3 \cdot 3H_2O$ (Johnson Matthey & Co. Ltd.), acetone (Qualigens) and diethyl ether (Qualigens) were used as such. The precursor $RuCl_3(PPh_3)_3$ [10] and the 12 amide ligands viz. 2-(anilinocarbonyl) benzoicacid (ACBA); 4-anilino-4-oxo but-2-enoicacid (AOBEA);

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Table 1
Physical and analytical data of Ru(III) complexes with coordinated amide

Complex no.	Complex/formula	Mp (°C)	Color	Yield (g)	*Analysesfound(calculated)*(%)			
					C	H	N	Ru
1.	$RuCl_2(PPh_3)_2$(ACBA), $C_{50}H_{40}Cl_2NO_3P_2Ru$	242	Green	0.269 (72%)	64.06 (64.10)	4.24 (4.27)	1.47 (1.49)	10.82 (10.79)
2.	$RuCl_2(PPh_3)_2$(AOBEA), $C_{46}H_{38}Cl_2NO_3P_2Ru$	235	Green	0.261 (74%)	62.22 (62.30)	4.32 (4.28)	1.60 (1.58)	11.36 (11.39)
3.	$RuCl_2(PPh_3)_2$(AOBA), $C_{46}H_{40}Cl_2NO_3P_2Ru$	231	Green	0.251 (71%)	62.11 (62.16)	4.55 (4.50)	1.59 (1.57)	11.33 (11.37)
4.	$RuCl_2(PPh_3)_2$(NACBA), $C_{54}H_{42}Cl_2NO_3P_2Ru$	249	Brown	0.295 (75%)	65.77 (65.72)	4.29 (4.25)	1.44 (1.41)	10.26 (10.24)
5.	$RuCl_2(PPh_3)_2$(NAOBEA), $C_{50}H_{40}Cl_2NO_3P_2Ru$	245	Brown	0.272 (73%)	64.02 (64.10)	4.31 (4.27)	1.46 (1.49)	10.83 (10.79)
6.	$RuCl_2(PPh_3)_2$(NAOBA), $C_{50}H_{42}Cl_2NO_3P_2Ru$	243	Brown	0.265 (71%)	64.01 (63.96)	4.51 (4.47)	1.49 (1.49)	10.72 (10.76)
7.	$RuCl_2(PPh_3)_2$(BACBA), $C_{51}H_{40}Cl_2N_3O_3P_2Ru$	279	Black	0.288 (74%)	62.63 (62.70)	4.13 (4.09)	4.35 (4.30)	10.33 (10.34)
8.	$RuCl_2(PPh_3)_2$(BAOBEA), $C_{47}H_{38}Cl_2N_3O_3P_2Ru$	275	Black	0.281 (76%)	60.83 (60.90)	4.14 (4.10)	4.56 (4.53)	10.88 (10.90)
9.	$RuCl_2(PPh_3)_2$(BAOBA), $C_{47}H_{40}Cl_2N_3O_3P_2Ru$	271	Black	0.266 (72%)	60.71 (60.77)	4.34 (4.31)	4.56 (4.52)	10.91 (10.88)
10.	$RuCl_2(PPh_3)_2$(PHCBA), $C_{50}H_{41}Cl_2N_2O_3P_2Ru$	242	Green	0.269 (71%)	63.01 (63.09)	4.27 (4.31)	2.96 (2.94)	10.60 (10.62)
11.	$RuCl_2(PPh_3)_2$(OPHBEA), $C_{46}H_{39}Cl_2N_2O_3P_2Ru$	234	Green	0.259 (72%)	61.20 (61.26)	4.36 (4.32)	3.13 (3.10)	11.17 (11.20)
12.	$RuCl_2(PPh_3)_2$(OPHBA), $C_{46}H_{41}Cl_2N_2O_3P_2Ru$	231	Green	0.274 (76%)	61.05 (61.12)	4.57 (4.54)	3.13 (3.10)	11.15 (11.18)

4-anilino-4-oxobutanoicacid (AOBA); 2-[(1-naphthyl amino) carbonyl] benzoic acid (NACBA); 4-(1-naphthylamino)4-oxobut-2-enoicacid (NAOBEA); 4-(1-naphthyl amino)-4-oxobutanoicacid (NAOBA); 2-[(1*H*-benzimidazol-2-yl amino)carbonyl]benzoic acid (BACBA); 4-(1*H*-benzimidazol-2-ylamino)-4-oxobut-2-enoic acid (BAOBEA); 4-(1*H*-benzimidazol-2-ylamino)-4-oxobutanoicacid (BAOBA); 2-[(2-phenylhydrazino) carbonyl] benzoicacid (PHCBA); 4-oxo-4-(2-phenylhydrazino)but-2-enoic acid (OPHBEA); 4-oxo-4-(2-phenyl hydrazino)butanoic acid (OPHBA) were synthesized as previously reported [14]. Hydrogen peroxide solution (Merck, 30%) was used as it is. 0.1N hydrochloric acid solution (Qualigens) was prepared by diluting 9.1 ml of conc. hydrochloric acid solution to 1000 ml with double distilled water. 0.1N sulfanilic acid solution (Merck) was prepared by dissolving 1.071 g in 100 ml of double distilled water.

2.3. Drug solutions

Standard stock solutions of pyridoxine hydrochloride or albuterol sulfate (1 mg/ml) were prepared by dissolving 100 mg of pure pyridoxine hydrochloride or albuterol sulfate in 100 ml of double distilled water. The stock solutions were diluted with double distilled water to get the working pure drug solutions of 100 μg/ml. An accurately weighed amount of tablet powder equivalent to 100 mg of pyridoxine hydrochloride or albuterol sulfate was extracted separately with chloroform (4 × 20 ml) and filtered. The filtrate was evaporated to dryness and the residue was dissolved in 100 ml of double distilled water to achieve a concentration of 1 mg/ml. This solution was diluted with double distilled water to get the working pharmaceutical solutions of 100 μg/ml.

2.4. Recommended procedures

2.4.1. Synthesis of ruthenium(III) catalysts

To $RuCl_3(PPh_3)_3$ solution (0.4 mmol in 20 ml acetone), ligand solution (0.4 mmol in 20 ml acetone) was added and the reaction mixture was stirred magnetically for 3 h. The resulting solution was concentrated to 5 ml under reduced pressure and a few ml of diethylether was added to initiate the crystallization. The precipitate formed was separated by suction filtration, washed with diethylether and dried in vacuum. The crystalline compound obtained was recrystallized using dichloromethane and diethylether mixture.

2.4.2. Ruthenium-catalyzed oxidation method

In a 100 ml round bottom flask, 4 ml of pyridoxine hydrochloride or albuterol sulfate solution (pure or pharmaceutical formulation), hydrogen peroxide (4 ml for PYE and 6 ml for AB) and 0.01 mmol of Ru(III) catalyst were taken. The contents of the flask were refluxed (15 min for PYE and 30 min for AB) at 60 °C. The contents of the flask were cooled and transferred separately into 20-ml-calibrated tubes. Now, sulfanilic acid solution (2 ml for PYL and 4 ml for AB) was added and the tubes were heated for 5 min in boiling water bath. Pink color was developed slowly. The tubes were cooled and the total volumes were made up to 20 ml with double distilled water. The absorbances of the colored solutions were measured at 520 nm against their reagent blanks. The amounts of PYL and OPAB formed during oxidation process were determined from their respective calibration curves.

3. Results and discussion

3.1. Characterization of Ru(III) complexes with coordinated amide

3.1.1. Physical and analytical data

Twelve Ru(III) complexes with coordinated amide were synthesized using the precursor, $RuCl_3(PPh_3)_3$. The percentages of carbon, hydrogen and nitrogen were determined experimentally using CHN analyzer. The percentage of ruthenium in complexes was determined by literature method [15]. The physical and analytical data (Table 1) for the newly synthesized Ru(III) complexes is in good agreement with the proposed molecular formulae viz. $RuCl_2(PPh_3)_2(L_2)$.

3.1.2. Infrared spectral analysis

The infrared spectra of the free amide ligands and precursor are compared with the Ru(III) complexes to elucidate the binding mode of the amide ligands to ruthenium. The non-involvement of amide nitrogen in coordination is confirmed by the consistent stretching frequencies of amide nitrogen in the range of 3375–3264 cm^{-1} in ligand and complexes spectra. However, in the IR spectra of Ru(III) complexes having ligands derived from benzimidazoles viz. BACBA, BAOBEA and BOABA, $\upsilon_{N\text{-}H}$ (benzimidazole) modes are observed at 3360, 3368 and 3365 cm^{-1}, respectively. Similarly, in the IR spectra of complexes having ligands derived from phenylhydrazines viz. PHCBA, OPHBEA and OPHBA, $\upsilon_{N\text{–}H}$ (phenylhydrazine) modes are observed at 3362, 3367 and 3360 cm^{-1}, respectively. Stretching frequencies of amide oxygen of complexes have undergone negative shifts by 30–40 cm^{-1} from 1670 cm^{-1} of free amide ligands indicating the coordination of amide oxygen to ruthenium [16]. In free ligands, strong absorption bands are found around 1710 and 1340 cm^{-1} corresponding to $\upsilon_{C=O}$ stretching and $\delta_{O\text{–}H}$ deformation modes of vibration. In complexes spectra, these bands are not observed, but new bands are observed in the ranges of 1548–1529 and 1388–1348 cm^{-1} corresponding to υ_{COO^-} (asymmetric) and υ_{COO^-} (symmetric) vibrations indicating the participation of oxygen atom of carboxylic group in chelation [12]. Similarly, a strong absorption band in precursor spectrum as well as complexes

Table 2
Infrared spectral data of Ru(III) complexes with coordinated amide

Complex no.	Complex	Selected IR bands (cm^{-1})						
		υ_{N-H} (amide)	$\upsilon_{C=O}$ (amide)	υ_{COO^-} (asy)	υ_{COO^-} (sy)	υ_{Ru-P}	υ_{Ru-Cl}	υ_{Ru-Cl}
1.	$RuCl_2(PPh_3)_2$(ACBA)	3375	1658	1546	1371	541	420	325, 317
2.	$RuCl_2(PPh_3)_2$(AOBEA)	3354	1660	1548	1378	548	418	342, 320
3.	$RuCl_2(PPh_3)_2$(AOBA)	3373	1662	1531	1384	541	440	330, 321
4.	$RuCl_2(PPh_3)_2$(NACBA)	3264	1669	1547	1383	542	432	335, 312
5.	$RuCl_2(PPh_3)_2$(NAOBEA)	3268	1667	1529	1382	539	425	338, 309
6.	$RuCl_2(PPh_3)_2$(NAOBA)	3272	1652	1553	1383	538	415	339, 311
7.	$RuCl_2(PPh_3)_2$(BACBA)	3360	1653	1545	1388	537	416	341, 312
8.	$RuCl_2(PPh_3)_2$(BAOBEA)	3368	1659	1541	1386	538	422	338, 303
9.	$RuCl_2(PPh_3)_2$(BAOBA)	3365	1661	1542	1382	540	430	337, 302
10.	$RuCl_2(PPh_3)_2$(PHCBA)	3362	1662	1541	1386	541	436	331, 301
11.	$RuCl_2(PPh_3)_2$(OPHBEA)	3367	1661	1542	1388	542	423	332, 305
12.	$RuCl_2(PPh_3)_2$(OPHBA)	3360	1652	1541	1348	539	417	343, 306

spectra was found around 540 cm^{-1} due to the presence of Ru–P bond [17]. The coordination of oxygen atom of ligand with ruthenium is indicated by the presence of a band in the range of 440–415 cm^{-1}. Two bands appear around 340 and 320 cm^{-1} in complexes spectra indicating the presence of two chlorides in *cis* position around ruthenium centre. All other characteristic bands due to triphenylphosphine are also present in the expected regions in precursor spectrum and complexes spectra [18] (Table 2). IR spectrum of $RuCl_2(PPh_3)_2$(ACBA) is shown in Fig. 1.

3.1.3. Mass spectral analysis

The proposed molecular formula of Ru(III) complex was confirmed by the mass spectral analysis by comparing its molecular formula weight with *m*/*z* value. The mass spectra contain molecular ion peaks at *m*/*z* (M+) 936 (Complex-1), 886 (Complex-2), 888 (Complex-3), 987 (Complex-4), 936 (Complex-5), 938 (Complex-6), 976 (Complex-7), 927 (Complex-8), 928 (Complex-9), 951 (Complex-10), 902 (Complex-11) and 903 (Complex-12). This data is in good agreement with the respective molecular formulae. Mass spectrum of $RuCl_2(PPh_3)_2$(ACBA) is presented in Fig. 2.

3.1.4. Electronic spectral analysis

Electronic spectra of all the complexes in dichloromethane showed two to four bands in the 240–560 nm region. The ground state of Ru(III) ($t^5{}_{2g}$ configuration) is $^2T_{2g}$ and the first excited doublet levels in the order of increasing energy are $^2A_{2g}$ and $^2T_{1g}$ which arise from the $t^4{}_{2g}e^1{}_g$ configuration. In most of the Ru(III) complexes the electronic spectra show only charge transfer bands. Those at 480–540 nm have been assigned to the $^2T_{2g} \rightarrow {}^2A_{2g}$ transition, in conformity with assignments made for similar octahedral Ru(III) complexes. The other bands in the 220–280 nm region have been assigned to charge transfer transitions [11].

3.1.5. ESR spectral analysis

The ESR spectra of all the powdered samples of Ru(III) complexes were recorded at room temperature. They exhibit normal axial distortion with three *g* values. The peak high field side is more intense than the peak at low field side. The *g* values calculated are in the range of 1.84–2.40, which are within the literature range [19]. The average *g* values of these complexes are very close indicating that all of them have similar type of distortion [20].

3.1.6. Magnetic and conductance measurements

The magnetic susceptibility measurements have been carried out for Ru(III) complexes and the values lie between 1.7 and 1.9 BM indicating $t^5{}_{2g}$ configuration and a low spin 3+ oxidation state for ruthenium in all these complexes. Hence, the compounds are paramagnetic in nature [11]. Molar conductance measurements made for all the Ru(III) complexes in dichloromethane reveals that all the compounds are non-electrolytes.

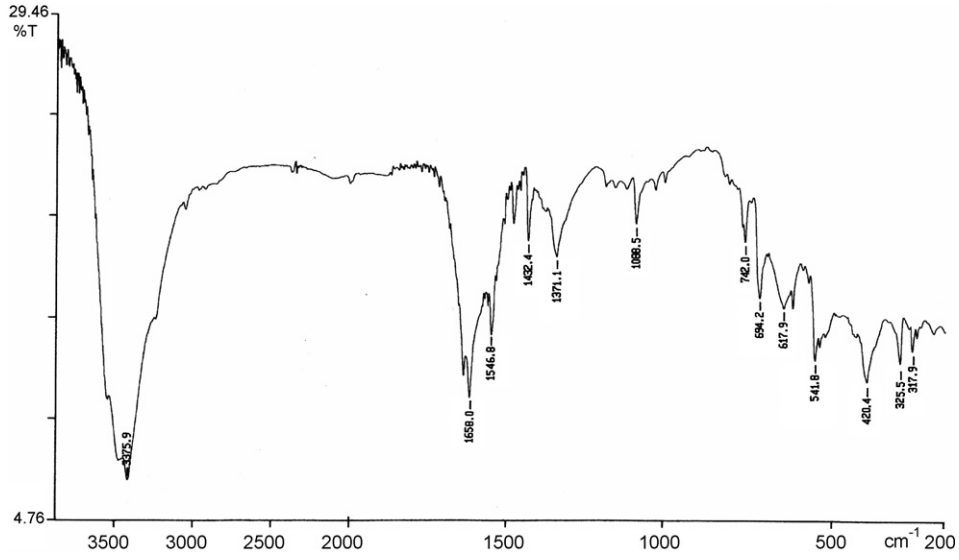


Fig. 1. IR spectrum of $RuCl_2(PPh_3)_2$(ACBA).

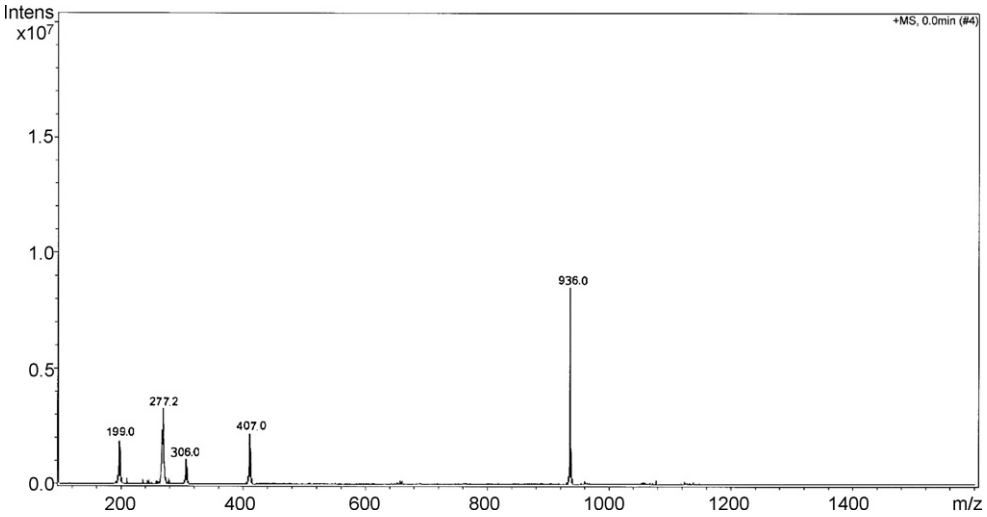


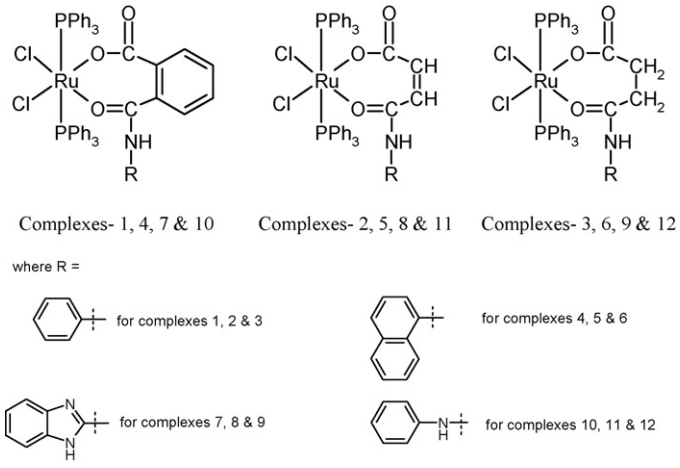
Fig. 2. Mass spectrum of $RuCl_2(PPh_3)_2$(ACBA).

On the basis of analytical and spectral data, octahedral structures (Scheme 1) have been proposed for all the Ru(III) complexes with coordinated amide.

3.2. Catalytic applications

3.2.1. Development of catalytic oxidation method

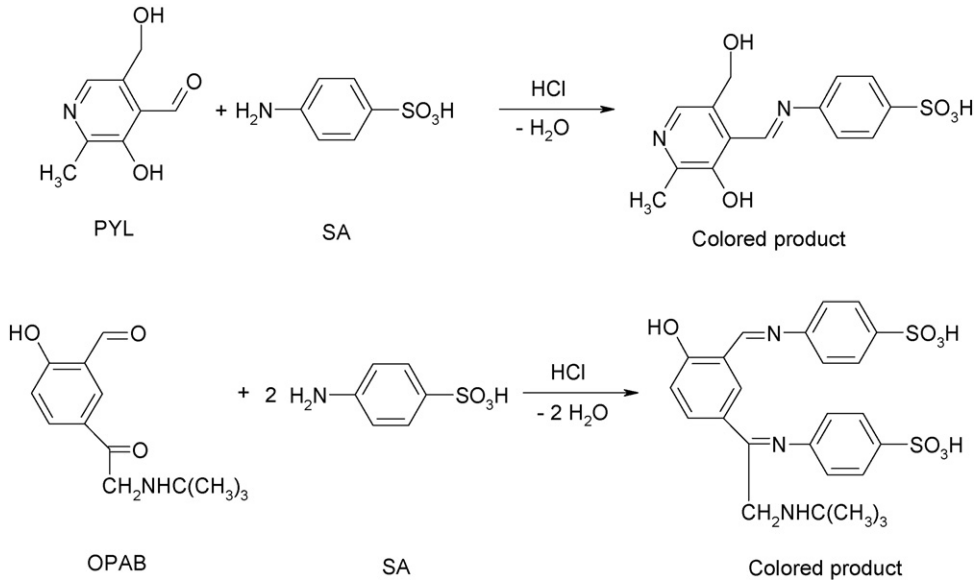
The oxidation of pyridoxine and albuterol using hydrogen peroxide was carried out in the presence of ruthenium catalyst. To produce a high yield of oxidation products, a continuous feed of hydrogen peroxide was employed. 4 ml of hydrogen peroxide for pyridoxine and 6 ml of hydrogen peroxide for albuterol were found to be optimum for oxidation. To assess whether any direct oxidation occurred upon addition of hydrogen peroxide, it was added to pyridoxine and albuterol separately in the absence of ruthenium catalyst at 60 °C. The oxidation of pyridoxine was started at about 30 min, completed after 2 h. The yield of oxidation product, i.e. pyridoxal was found to be 82.62%. But, in the presence of ruthenium catalyst (Complex-1), the oxidation was started at about 10 min, completed within 20 min and the yield of the oxidation product was found to be 96.66%. Similarly, the oxidation of albuterol was started at about 45 min, completed after 3 h and the percent yield of oxidation product was found to be 85.83% in the absence of ruthenium catalyst. But, in the presence of ruthenium catalyst (Complex-1), the oxidation was stated at about 15 min and completed within 30 min and the percent yield of the product was found to be 98.18%. Almost similar tendencies were observed with the remaining 11 Ru(III) catalysts (Complex-2–12). 0.01 mmol of ruthenium catalyst is found to be sufficient for the reaction. The Ru(III) catalyst can be used three times after its recovery. The best results were obtained when the temperatures of reaction mixtures were set to 60 °C. The role of co-oxidant, i.e. *N*-methyl morpholine-N-oxide, which was generally used in the oxidation of benzyl alcohol [2], was also investigated. The yields of oxidation products in the presence as well as in the absence of co-oxidant were found to be one and the same. Hence, presence of co-oxidant was not necessary when present catalysts are used for oxidation. After completion of the oxidation of drugs, the mixtures were cooled. The residual hydrogen peroxide after competition of oxidation is removed by adding 1 ml of 0.01 M potassium permanganate solution. The condensation of oxidation products of pyridoxine and albuterol were carried out with five amino group compounds viz. sulfanilic acid [13], 2-thio barbituric acid [21], thiosemicarbazide [22], *o*-toluidine [23] and diphenylamine [24]. Since, sulfanilic acid produces either high molar absorptivity it was chosen for condensation of oxidation products. The adequate volumes of sulfanilic acid required for condensation are found to be 2 ml for pyridoxal and 4 ml for the oxidation product albuterol. Maximum wavelength of both the colored products were found to be 520 nm and were stable up to 1 h.



Scheme 1. Structures of Ru(III) complexes with coordinated amide.

3.2.2. Product analysis

In order to establish the structures of oxidation products, IR and NMR spectra of pyridoxine and albuterol were compared with the spectra of their oxidation products. The infrared spectra of pyridoxine and albuterol show a broad peak around 3455 cm^{-1} due to O–H stretching. They do not show any peak around 1680 cm^{-1} indicating the absence of carbonyl group. The appearance strong absorption band in PYL and OPAB around 1680 cm^{-1} indicates the presence of carbonyl group due to C=O stretching after oxidation. This supports the oxidation of primary alcoholic group to aldehyde group in PYL and oxidation of primary and secondary alcoholic groups to aldehyde and ketone groups, respectively, in OPAB. This fact was further supported by the disappearance of the broad peak around 3055 cm^{-1} due to O–H stretching in OPAB. However, the broad peak at 3500 cm^{-1} was retained in PYL, since the alcoholic group at *meta* position of the pyridine ring does not undergo oxidation. Hence, in PYE, the alcoholic group at *para* position to pyridine ring which has relatively less steric hindrance when compared to alcoholic group



Scheme 2. Colored products formed between PYL or OPAB and SA.

Table 3
Yields of PYL and OPAB formed by using Ru(III) complexes with coordinated amide

Complex no.	Complex	Yield (%)	
		PYL	OPAB
1.	$RuCl_2(PPh_3)_2$(ACBA)	96.66	98.18
2.	$RuCl_2(PPh_3)_2$(AOBEA)	97.27	98.84
3.	$RuCl_2(PPh_3)_2$(AOBA)	96.02	97.58
4.	$RuCl_2(PPh_3)_2$(NACBA)	97.29	98.55
5.	$RuCl_2(PPh_3)_2$(NAOBEA)	97.82	98.95
6.	$RuCl_2(PPh_3)_2$(NAOBA)	95.03	96.78
7.	$RuCl_2(PPh_3)_2$(BACBA)	97.54	98.88
8.	$RuCl_2(PPh_3)_2$(BAOBEA)	98.25	99.65
9.	$RuCl_2(PPh_3)_2$(BAOBA)	98.15	98.85
10.	$RuCl_2(PPh_3)_2$(PHCBA)	96.54	97.90
11.	$RuCl_2(PPh_3)_2$(OPHBEA)	97.85	98.54
12.	$RuCl_2(PPh_3)_2$(OPHBA)	96.35	97.85

at *meta* position may be oxidized to aldehyde [25]. Oxidation of alcoholic groups to carbonyl groups is further confirmed by NMR spectral analysis. The NMR spectra of pyridoxine show peaks at 8.37, 5.94, 5.13 and 2.86 δ corresponding to aromatic, methylene, alcoholic and alkyl protons, respectively, where as its oxidation product shows one additional peak at 9.50 δ. The NMR spectra of albuterol shows peaks at 6.55, 5.83, 4.66, 3.61 and 1.12 δ corresponding to aromatic, alcoholic, imino, methylene and methyl protons, respectively, where as its oxidation product show one additional peak at 9.60 δ. The peak at 5.83 was not shown in the spectra of OPAB, indicating the absence of alcoholic proton. The appearance of peaks at 9.50 or 9.60 δ in both the spectra of oxidation products, indicates the presence of aldehyde protons in the oxidation products and there by confirms the oxidation of alcoholic groups to aldehyde groups.

3.2.3. Chemistry of colored species and yields of oxidation products

The pink colored products are formed due to the condensation between the newly formed carbonyl groups in PYL and OPAB and amino group of sulfanilic acid in acid medium with the elimination of water molecule(s) (Scheme 2). The yields of oxidation products of pyridoxine and albuterol with all the 12 ruthenium catalysts were determined spectrophotometrically. The yields of OPAB are high when compared to PYL (Table 3), which may be due to the presence of two alcoholic groups in AB.

4. Conclusions

Ru(III) complexes with coordinated amide ligands were synthesized from $RuCl_3(PPh_3)_3$. Octahedral structures were assigned to these complexes based on elemental and spectral data. These complexes were found to be efficient for the oxidation of alcoholic drugs. This catalytic method is environmentally friendly, simple to set-up, requires short reaction times and produces high product yields.

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