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Synthesis of cationic rhodium(I) and iridium(I) carbonyl complexes of tetradentate $P(CH_2CH_2PPh_2)_3$ ligand: An implication of steric inhibition and catalytic hydroformylation reaction

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ABSTRACT

New cationic carbonyl complexes of the type [M(CO)L]Cl (**1**) [M = Rh (**a**) and Ir (**b**); L = $P(CH_2CH_2PPh_2)_3$] have been synthesized and characterized by various spectroscopic techniques including the single crystal X-ray diffraction. Both the complexes crystallize in trigonal bipyramidal symmetry with the metal at the centre. Two strong intermolecular hydrogen bonds are observed in **1a**. Despite the high *trans* effect of the CO group, the symmetrical tetradentate phosphine ligand bonded strongly to the metal centre through all sites and thereby expelling the Cl^- ion outside the coordination sphere. The complexes are very stable and inert towards the oxidative addition (OA) of small molecules like CH_3I, C_2H_5I and I_2 at room as well as higher temperature. However, the complexes show catalytic activity towards the hydroformylation of alkene to the corresponding aldehydes under the reaction conditions: pressure 35 ± 2 bar, temperature 80 ± 2 °C, 500 rpm and time 5–8 h. The yields of the aldehyde products are in the ranges 55–75%. The complexes exhibited dissociation of weaker M—P bond under the above reaction conditions to form a vacant coordination site for initiation of the catalytic cycle.

1. Introduction

Steric and electronic properties of ligands play important roles in the reactivity and catalytic activity of organometallic complexes [1–13]. In general, the coordination chemistry of phosphine ligands with transition metals is of considerable interest because of their structural novelty, reactivity and catalytic activity [1–19]. A wide range of transition metal carbonyl complexes of phosphine based ligands show excellent reactivity towards small molecule activation [8–14,17–25]. The chemistry of rhodium(I) and iridium(I) complexes of different ligands is a subject of interest due to their rich chemistry, both in academia as well as industry. Several catalytic systems like rhodium based Monsanto's process of methanol carbonylation [26–29], L-DOPA synthesis [30], etc. and iridium based Cativa process [31] for production of acetic acid are a few notable examples of the existing industrial processes. Apart from these, rhodium complexes are also known to act as efficient catalysts for hydroformylation of alkenes to corresponding aldehydes [32–35]. Though a large number of rhodium complexes are well documented in literature for such process [17,32–35], the studies of iridium complexes in hydroformylation reaction are quite scanty because of their lower efficiency [36–38]. The rhodium(I) and iridium(I) complexes tend to undergo OA reactions which are often the key step in catalytic cycles. A series of rhodium carbonyl complexes of different mono- and multidentate ligands have been reported by us [17–19,28,29,39] for homogeneous catalytic carbonylation reactions. In this work, we report the synthesis and structural characterization of two new cationic pentacoordinated rhodium(I) and iridium(I) carbonyl complexes of tetradentate phosphine ligand $P(CH_2CH_2PPh_2)_3$. The effect of the ligand towards the activation of small molecules and catalytic activity in hydroformylation reactions are also demonstrated.

2. Experimental

2.1. General information

All solvents were distilled under N_2 prior to use. $RhCl_3 \cdot xH_2O$ and $IrCl_3 \cdot xH_2O$ were purchased from M/S Arrora Matthey Ltd., Kolkata, India. The ligand tris[2-(diphenylphosphino)ethyl]phosphine was purchased from M/S Acros Organics, USA and was used without further purification. Elemental analyses were performed on a Perkin-Elmer 2400 elemental analyzer. IR spectra (4000–400 cm^{-1}) were recorded on KBr discs and liquid cells with a Shimadzu IR Affinity-1 FTIR spectrophotometer. ^1H, ^{13}C and ^{31}P NMR spectra were recorded in $CDCl_3$ solution on a Bruker DPX-300 spectrometer, and chemical shifts were reported relative to $SiMe_4$ and

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85% H_3PO_4 as internal and external standard respectively. Mass spectra of complexes were recorded on an ESQUIRE 3000 mass spectrometer. Thermal analyses of the complexes were carried out using a thermal analyzer (TA instrument, model SDQ 600) simultaneous DTA-TGA) under argon atmosphere with a heating rate of 10 °C/min. The hydroformylation reactions of alkene were carried out in a high pressure reactor (Parr-4592, USA) fitted with a pressure gauge and the reaction products were analyzed by GC (Chemito 8510, FID).

2.2. Synthesis of starting materials

The starting dimeric rhodium moiety $[Rh(CO)_2Cl]_2$ [40] and iridium moiety $[Ir(COE)_2Cl]_2$ [41] were synthesized by following the literature procedure.

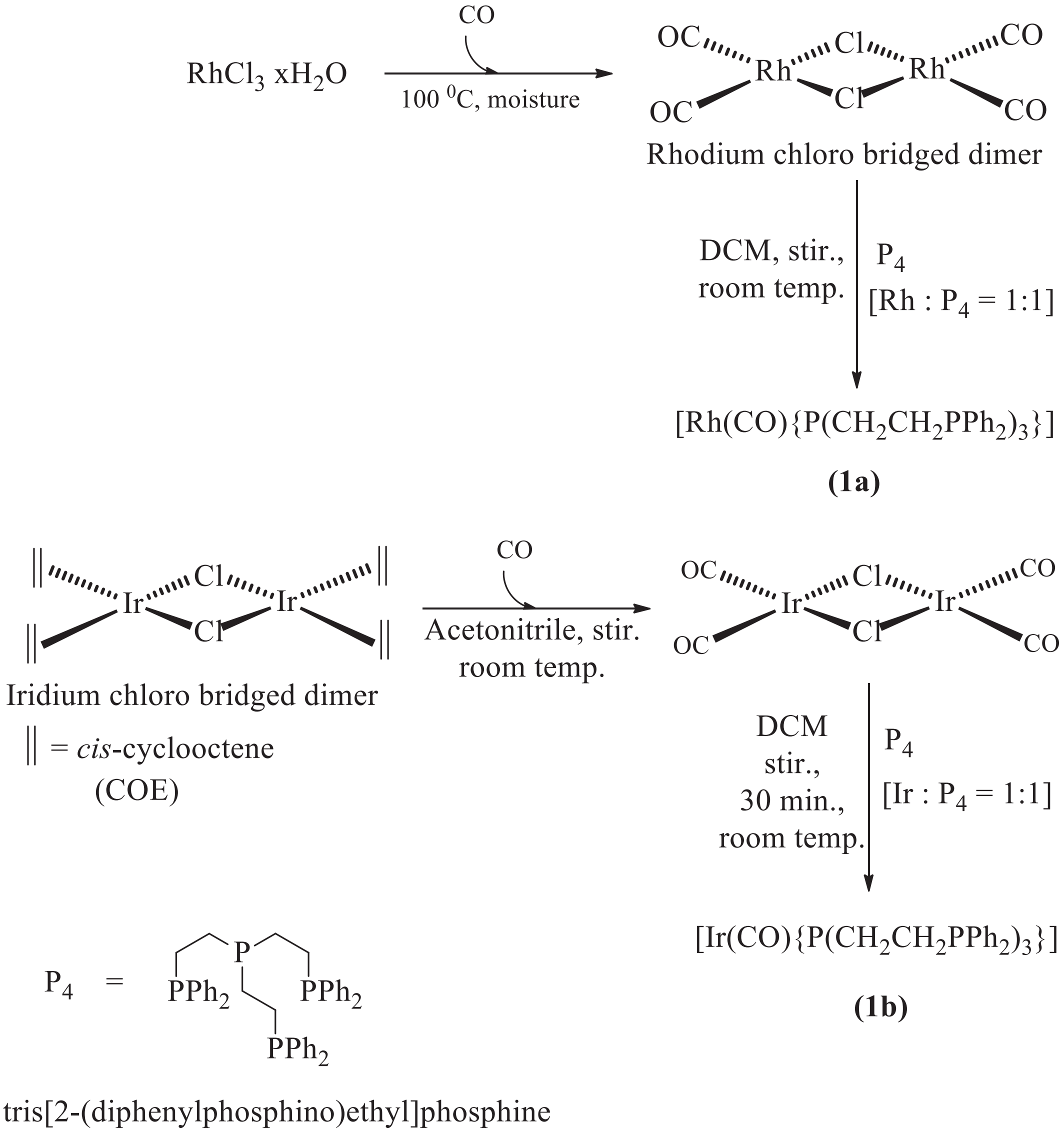
*2.3. Synthesis of the complex $[Rh(CO)\{P^{/}(CH_2CH_2PPh_2)_3\}]Cl$ (**1a**)*

$[Rh(CO)_2Cl]_2$ (100 mg) was dissolved in dichloromethane (DCM) (10 cm^3) and to this solution, a stoichiometric amount (Rh:L = 1:1) of the ligand solution in DCM was added. The reaction mixture was stirred at room temperature (25 °C) for about 30 min and the solvent was evaporated under vacuum. The light green coloured compounds so obtained were washed with hexane as well as diethyl ether and recrystallized from DCM solution. The compound was dried and stored over silica gel in a desiccator.

Analytical data for **1a**: Yield: 85%; IR (KBr, cm^{-1}): 1980 [ν(CO)]. ^{1}H NMR (CDCl$_3$, ppm): δ 2.3, 2.67 (m, 12H, CH$_2$), δ 7.18, 7.22 (m, 30H, Ph), ^{13}C NMR (CDCl$_3$, ppm): δ 30.92, 40.45 (CH$_2$), δ 125.14–141.3 (Ph), δ 199.24 (CO). ^{31}P {^{1}H} NMR (CDCl$_3$, ppm): δ 152.6 [d, J_{Rh-P} = 82.84 Hz P$^{/}$], δ 62.1 [d, J_{Rh-P} = 156.94 Hz, P]. Elemental analyses; Found (Cald for $RhC_{43}H_{42}OP_4$): C, 60.88 (61.48), H, 5.11 (5.24). MS: m/z = 834.6 (M$^+$).

*2.4. Synthesis of the complex $[Ir(CO)\{P^{/}(CH_2CH_2PPh_2)_3\}]Cl$ (**1b**)*

$[Ir(COE)_2Cl]_2$ (100 mg) was dissolved in acetonitrile. The solution was then stirred at room temperature for about 10 min under a N_2 atmosphere. The flow of N_2 was stopped and the CO gas was passed to the solution until a clear greenish yellow solution was obtained. A stoichiometric amount (Ir:L = 1:1) of the ligand solution in DCM was added to the solution and the mixture was stirred for about another 30 min. The solvent was evaporated under vacuum. The greenish yellow coloured compounds so obtained were



Scheme 1. Synthesis of **1a** and **1b**.

washed with hexane as well as diethyl ether and recrystallized from DCM solution. The compounds were dried and stored over silica gel in a desiccator.

Analytical data for **1b**: Yield: 84%; IR (KBr, cm^{-1}): 1965 [ν(CO)]. ^{1}H NMR ($CDCl_3$, ppm): δ 1.56, 2.17 (m, 12H, CH_2), δ 7.45, 7.74 (m, 30H, Ph), ^{13}C NMR ($CDCl_3$, ppm): δ 22.7, 29.37 (CH_2), δ 128–137.1 (Ph), δ 200.63 (CO). ^{31}P {^{1}H} NMR ($CDCl_3$, ppm): δ 129.96 [d, $J_{P-P} = 13.43$ Hz P'], δ 36.79 [d, $J_{P-P} = 13.27$ Hz, P]. Elemental analyses; Found (Cald for $IrC_{43}H_{42}OP_4$): C, 57.32 (57.91), H, 4.29 (4.71). MS: $m/z = 952.2$ (M^+).

2.5. X-ray structural analysis

Single crystals of **1a** and **1b** suitable for X-ray crystallographic analyses were obtained by layering DCM solution of the respective compounds with hexane. Intensity data of **1a** and **1b** were collected on a Bruker Smart-CCD with Mo Kα radiation ($\lambda = 0.71073$ Å) at 110 K. The structures were solved with SHELXS-97 and refined by full-matrix least squares on F^2 using the SHELXL-97 computer program [42]. Hydrogen atoms were idealized by using the riding models.

2.6. Catalytic activity towards hydroformylation reaction

The hydroformylation reactions were carried out in a Parr reactor. 2.5 mmol of the alkene and 0.005 mmol of the catalyst in 5 cm^3 DCM were placed in a 50 cm^3 reaction vessel. The reaction mixture was purged with CO/H_2 (1:1) for about 5 min. The CO/H_2 pressure was increased to 35 bar (actual pressure) at 25 °C in the closed reactor. The hydroformylation reactions were carried out at 80 ± 2 °C for 3–8 h at 35 ± 2 bar. The reaction mixture was analyzed by GC and products were isolated by column chromatography taking hexane and ethyl acetate as eluents.

2.7. IR study

The course of the hydroformylation reactions were monitored by FTIR spectroscopy in a solution cell (CaF_2 windows, 1.0 mm path length). The reaction mixtures were scanned in the range 1550–2200 cm^{-1} at different time intervals to investigate the differences in the corresponding spectra.

3. Results and discussion

3.1. Synthesis and characterization

The dimeric precursor $[Rh(CO)_2Cl]_2$ reacts with 2 mol equivalents of the ligand P_4 [Dimer:Ligand = 1:2] in DCM to afford the complex $[Rh(CO)P(CH_2CH_2PPh_2)_3]Cl$ (**1a**); while in situ generated dimeric precursor $[Ir(CO)_2Cl]_2$ reacts with 2 mol equivalents of the ligand P_4 [Dimer:Ligand = 1:2] in DCM to yield the complex $[Ir(CO)P(CH_2CH_2PPh_2)_3]Cl$ (**1b**) (Scheme 1). Elemental analyses and mass spectrometric data of the complexes support the observed molecular composition of **1a** and **1b**. The IR spectra of **1a** and **1b** exhibit intense ν(CO) bands at 1980 and 1965 cm^{-1} respectively, indicating the formation of monocarbonyl complexes. The appearance of relatively lower ν(CO) in IR spectra reveals the presence of electron rich metal centres in both the complexes. The $^{31}P\{^{1}H\}$ NMR spectra of the complexes show two distinct doublets at δ 152.6 ($J_{Rh-P} = 82.84$ Hz), 62.1 ($J_{Rh-P} = 156.94$ Hz) ppm (**1a**) and δ 129.96 ($J_{P-P} = 13.43$ Hz), 36.79 ($J_{P-P} = 13.27$ Hz) ppm (**1b**) for two different types of P-atoms in the complexes. The intensity ratio of two chemical shifts in $^{31}P\{^{1}H\}$ NMR spectra are around 1:3, which indicates that out of four phosphorus centres, three are in the similar chemical environment. The significant downfield shift in **1a** compared to **1b** indicates a stronger interaction of the P-atoms with the metal centre in the former complex. The ^{1}H NMR spectra of the complexes show characteristic resonances for methylene and phenylic protons in the range 1.56–2.67 and 7.18–7.74 ppm respectively. In the ^{13}C NMR spectra of **1a** and **1b**, weak signals for the carbonyl carbons appeared in the range 199.24–200.63 ppm. The thermogravimetric analyses (TGA) data (Fig. 1) indicate that both the complexes are thermally stable up to around 390 °C. The mass loss of ~9% at around 45 °C is due to the decomposition of DCM as solvent of crystallization. Both the complexes decompose at around 400 °C with mass loss of ~50%.

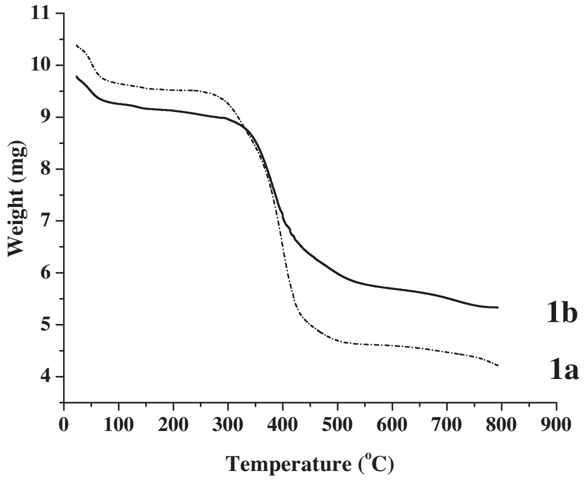


Fig. 1. TGA plots of **1a** and **1b**.

The structures of complexes **1a** and **1b** determined by single crystal X-ray diffraction (Fig. 2) show that the metal atom occupies the centre of a distorted trigonal bipyramidal geometry formed by four P atoms of the ligand P_4 and a C (of CO) atom. In **1a**, intermolecular hydrogen type bond (2.682 Å) is observed between the oxygen of carbonyl group and a hydrogen of phenyl ring (Fig. 3), while such interaction is absent in **1b**. The CO group occupies one of the axial positions trans to central P atom of the ligand P_4. The P(1)-M-P(3) angles in the equatorial plane of **1a** and **1b** are 123.28° and 119.97° respectively which are very close to the normal value i.e. 120° for the angles of the attributed due to the symmetric distribution of the ligand around the metal centre. The bulky tripodal ligand forms capped complexes with the metal centres, leaving very little space for other donors to attach with. Despite the high *trans* effect of the CO group, the highly symmetrical phosphine ligand bonded strongly to the metal centre through all sites, resulting the expulsion of the Cl^- ion outside the coordination sphere to generate cationic complexes. It appears (Fig. 2) that all P atoms are not at equal distances from the central metal centre in the complexes. In **1a**, the bond distance (2.287 Å) of central P atom from the metal centre is slightly smaller compared to the other three P atoms. On the other hand, the Rh—P(4) bond distance (2.346 Å) is the highest among all Rh—P bonds, which may dissociate preferably compared to others during a catalytic cycle. Similarly, the Ir—P(4) bond distance is the longest among all Ir—P bonds in **1b**, indicating that the dissociation may likely to occur at the Ir—P(4) bond.

3.2. Reactivity

The reactivity of the complexes were evaluated towards the OA reaction of small molecules like I_2, CH_3I and C_2H_5I at different temperatures. But, the complexes were found inert under the reaction condition, which may be due to the steric effect of the phosphine ligand around the metal centres (Fig. 2). The bulkiness of the P_4 ligand around the metal centres in both the complexes allow no

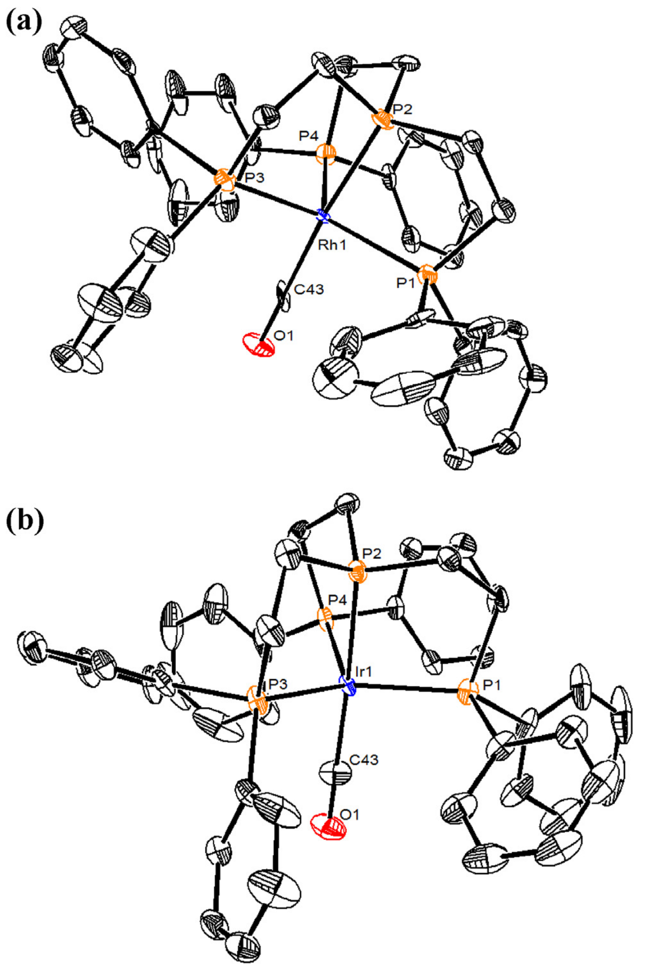


Fig. 2. Ortep diagram of **1a** and **1b** (thermal ellipsoids are drawn at 50% probability).

room to the incoming electrophile for coordination to the metal centre.

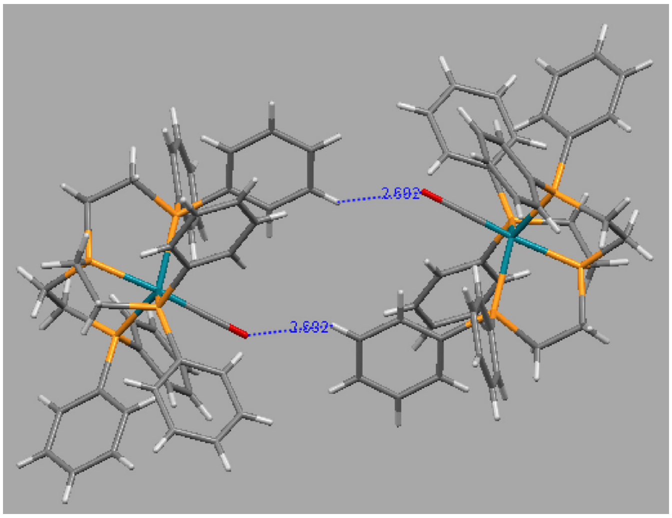
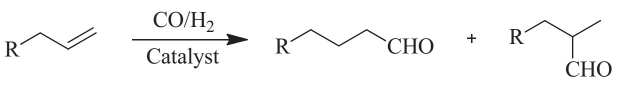


Fig. 3. Intermolecular hydrogen bonding in **1a**.

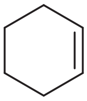
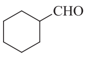
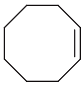
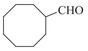
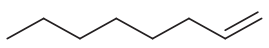
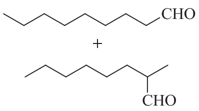


Scheme 2. Formation of aldehydes by the hydroformylation of terminal alkenes.

3.3. Catalytic activity

The catalytic activity of the complexes was investigated in carbonylation of methanol under the reaction conditions: temperature 130 ± 2 °C, pressure 30 ± 2 bar, 450 rpm and time 1 h, but no product could be detected, which may be due the inertness of **1a** and **1b** towards the OA of CH_3I, a key step of carbonylation reaction. The complexes were further investigated towards the hydroformylation of alkenes under the reaction conditions: 35 ± 2 bar, 80 ± 2 °C, 500 rpm, 5–8 h (Scheme 2) and good results are obtained (Table 1). The complex **1a** shows better catalytic activity over **1b**, under the given reaction conditions. The reaction products separated by column chromatography were identified by ^{1}H NMR spectroscopy. The reaction mixture was analyzed by GC to determine the conversion and regioselectivity. The hydroformylation of 1-octene yielded 71% nonanal with a regioselectivity (n:iso) of 62:35 in the presence of **1a** as catalyst, while the complex **1b** yielded 62% nananal with a regioselectivity of 55:32 (Table 1, entry 3). The bulkiness of the P_4 ligand around the metal centres in both the complexes results lower turnover frequency (TOF) for hydroformylation of 1-octene compared to the previously reported rhodium-based catalytic systems [43]. The

Table 1
Catalytic efficiency of **1a** and **1b** for hydroformylation of alkenes.[a]

Sl. no	Substrate	Product	Catalyst	Time (h)	Isolated yield[b] (%)	Conversion (%)[c]		TOF (h^{-1})	
						n	*iso*	*n*	*iso*
1		 	**1a** **1b**	5 8	75 60	75 63	– –	75 39.4	– –
2		 	**1a** **1b**	5 8	69 55	64 52	– –	64 33	– –
3		 	**1a** **1b**	5 8	71 62	62 55	35 32	62 34.4	35 20

[a] Reaction conditions: 2.5 mmol of the alkene, 0.005 mmol of catalyst, 35 ± 2 bar, 80 ± 2 °C, 500 rpm, 5–8 h.

[b] Isolated yields were calculated with respect to the substrate taken after separating the product by column chromatography.

[c] Conversion and *n:iso* ratio was determined by GC.

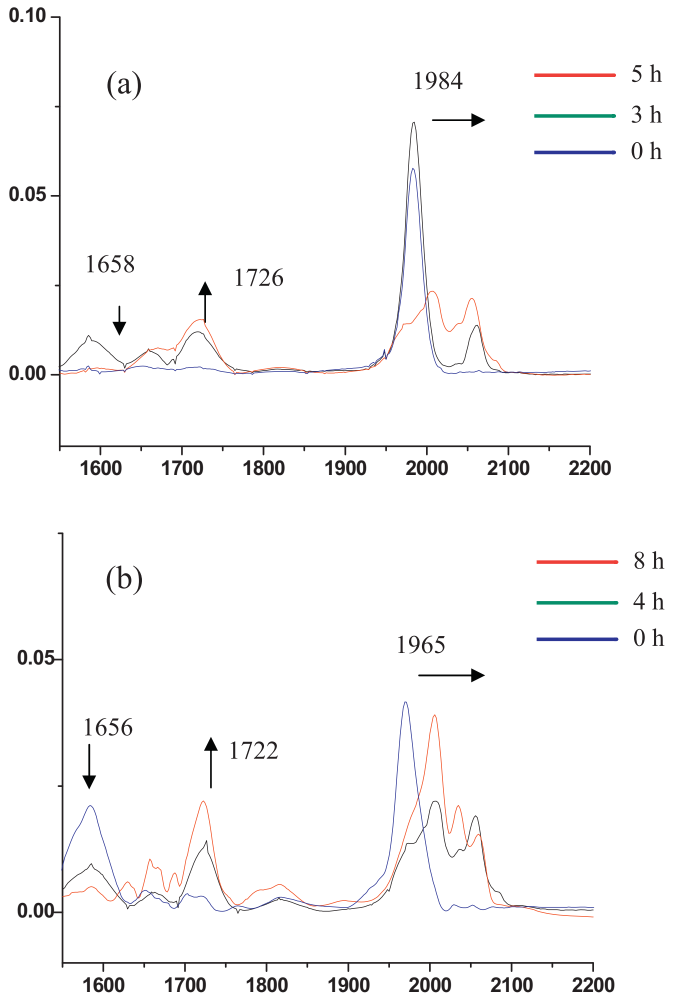


Fig. 4. Series of IR spectra illustrating the progress of reaction for the substrate cyclohexene in presence of **1a** (a) and **1b** (b).

catalytic behaviour of the complexes was studied by monitoring the hydroformylation reaction mixture of cyclohexene by IR spectroscopy at different time intervals. It is obserevd (Fig. 4a) that as the reaction proceeds, the initial peaks at 1658 and 1984 cm^{-1} due to stretching bands of the C=C of cyclohexene and CO of **1a** respectively show a significant change indicating formation of new products. The appearance of new peak at 1726 cm^{-1} infers the formation of aldehydic carbonyl group with gradual decrease in the intensity of C=C peak. A similar behaviour was also observed (Fig. 4b) with **1b**, in which the intensity of a peak at 1722 cm^{-1} gradually increases with the shifting of the CO peak towards higher value. Unlike carbonylation, the activity of the complexes towards hydroformylation may be due to the OA of smaller H_2 molecule into the crowded metal centres, which initiates the catalytic cycle. ^{1}H NMR spectrum of the reaction mixture containing **1a** shows a peak at −40.0 ppm, which may be due to the formation of a hydride complex during the reaction. The higher activity of **1a** over **1b** may be attributed to the relatively facile dissociation of one of the Rh—P bonds in **1a** than the one of the Ir—P bonds in **1b** during the course of the catalytic reaction. The dissociation of a M—P bond during the catalytic reaction was confirmed by ^{31}P NMR spectroscopy on analysing the organometallic residue of **1a** after reaction [**1a**$'$: δ 34.66, 31.49, −27.07 ppm]. Bianchini and coworkers also reported [13] dissociation of one phosphine arm of the bulky P_4 ligand was necessary for the insertation of a chemical moiety at the metal centres.

4. Conclusions

Two new cationic rhodium(I) and iridium(I) carbonyl complexes of the type $[M(CO)P(CH_2CH_2PPh_2)_3]Cl$ (M = Rh, Ir) have been synthesized and structurally characterized by different spectroscopic techniques including single crystal X-ray diffraction. Both the complexes exhibit a trigonal bipyramidal structure where the CO group occupies an axial position. The complexes were found to be unreactive towards the oxidative addition of small molecules i.e. I_2, CH_3I and C_2H_5I at room as well as elevated temperature. However, both the complexes were found to be good catalysts for the hydroformylation reaction under the experimental conditions 35 ± 2 bar, 80 ± 2 °C, 500 rpm, 5–8 h.

Supplementary material

CCDC-876707 (**1a**) and 876708 (**1b**); contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Acknowledgements

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.molcata.2013.10.016.

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