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Rhodium-catalyzed hydroformylation of olefins: Effect of [bis(2,4-di-*tert*-butyl) pentaerythritol] diphosphite (alkanox P-24) on the regioselectivity of the reaction

Jimoh Tijani, Bassam El Ali *

Chemistry Department, KFUPM, 31261 Dhahran, Saudi Arabia

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Abstract

Rhodium (I) associated with [bis(2,4-di-*tert*-butyl) pentaerythritol] diphosphite (**I**) as a ligand represents an active catalyst system for highly regioselective hydroformylation of various alkenes. The commercially available bis(2,4-di-*tert*-butyl)pentaerythritol diphosphite (alkanox P-24) (**I**), which has been used so far as an antioxidant in the stabilization of polymers, was used as a diphosphite ligand for the selective hydroformylation reaction of olefins. Excellent selectivity towards linear aldehydes and excellent conversions were achieved in the hydroformylation of alkenes. The hydroformylation reaction was applied to various olefinic substrates including the internal alkenes.

Keywords: Hydroformylation; Rhodium; Alkanox P-24; 1-Octene; Olefins; Phosphite ligands; Syngas

1. Introduction

The rhodium-catalyzed low-pressure hydroformylation of olefins is, in terms of production volume, one of the most important homogeneous catalysis processes [1]. Since Wilkinson's discovery through addition of phosphine ligands to rhodium-catalysts, the hydroformylation can be now performed at lower temperature and pressure [2]. Phosphite ligands and especially bulky phosphites are very useful in rhodium catalyzed hydroformylation because of the higher reaction rates obtained compared to phosphine ligands [3,4]. An important drawback however is the loss of selectivity, caused by isomerization [5]. One way to improve the selectivity was by changing to a diphosphite system. It was only after the first report from Bryant and coworkers that diphosphites were recognized as a new generation of promising ligands in rhodium catalyzed hydroformylation of alkenes [6]. High regioselectivity with diphosphite ligands (with 2,2′-biphenol backbone) in the rhodium catalyzed hydroformylation of functionalized alkenes was claimed [7,8].

The hydroformylation of 1-octene catalyzed by $Rh(CO)_2(acac)$ with chelated bisphosphites (calix[4]arene backbone) gave 99.5% nonanal at 80–100 °C and 5–20 bars [1]. The naturally occurring cinchona alkanoids, chiconidine, quinine and quinidine were hydroformylated to terminal aldehyde derivatives in yields of 87%, 81% and 85%, respectively, using $Rh(CO)_2(acac)$/tetraphosphite catalyst at 90 °C and 20 bars CO/H_2 in toluene [9].

Sterically constrained diphosphonite ligands with xanthene backbone were used in the rhodium-catalyzed hydroformylation of 1-octene and 2-butene. High activities were obtained for 1-octene, with good selectivity to the linear aldehyde and for the 2-butene the selectivity for the *n*-pentanal was 62% [10].

Bis(2,4-di-*tert*-butyl)pentaerythritol diphosphite (alkanox P-24) (**I**), which was used mainly as an antioxidant in the stabilization of polymers [11], was used in the hydroformylation of internal alkenes or mixture of alkenes [12,13].

* Corresponding author. Tel.: +966 3 8604491; fax: +966 3 8604277.
E-mail address: belali@kfupm.edu.sa (B. El Ali).

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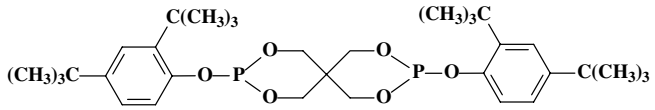
In the present work, a highly regioselective rhodium-catalyzed hydroformylation of 1-alkenes was achieved in the presence of bis(2,4-di-*tert*-butyl)pentaerythritol diphosphite (alkanox P-24) (**I**) as a ligand.

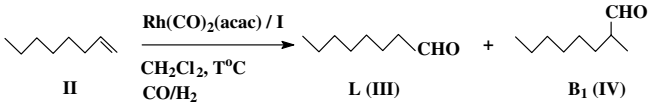


Bis(2,4-di-tert-butylphenyl)pentaerythritol diphosphite (I)

2. Results and discussion

2.1. Hydroformylation of 1-octene by $Rh(CO)_2(acac)$-I: effect of the temperature and the type of solvent

Hydroformylation of 1-octene, chosen as a model substrate, into 1-nonanal (**L**) and 2-methyl octanal ($\mathbf{B_1}$) occurred smoothly at relatively mild conditions (120 °C, 300 psi ($CO/H_2 = 1{:}2$), and 1 h), with $Rh(CO)_2(acac)$ used as a catalyst and the diphosphite alkanox (**I**) used as a ligand



(1)

A systematic study on the influence of the temperature on the regioselectivity and the catalytic activity in the hydroformylation of 1-octene in dichlomethane as a solvent was carried out at a variety of temperatures ranging from 80 °C to 130 °C. The results are shown in Table 1. The formation of the linear aldehyde as a major product prevailed in all cases at all temperatures.

The results of the reaction after an hour showed a very low conversion (18%) at 80 °C (Table 1, entry 1) with 84% linear aldehyde. The conversion increased to 73% and 99% at 100 °C and 120 °C, respectively. The selectivity for the linear isomer increases from 87% at 100 °C to 95% at 120 °C (Table 1, entries 2,3). It was also observed that the isomerization of 1-octene increases with the temperature. For instance, only 5% of isomerized octenes were detected in the products of the reaction at 80 °C, while the percentage of these isomers increased to 18% at 130 °C. In fact, it was reported that branched Rh-alkyl intermediate forms faster than the linear isomer intermediate. However, the first isomer was shown to be reversible through β-elimination to form 2-octene, while the second is irreversible and can only evolve to the linear aldehyde. The β-elimination of the branched Rh-alkyl intermediate significantly increases with temperature, thus forming more linear aldehyde and also more products of isomerization [14].

In addition, it is important to note that the percentages of octane, which is the hydrogenated product, were generally low (1–2%).

Table 1
Hydroformylation of 1-octene by $Rh(CO)_2(acac)$/alkanox (**I**). Effect of the temperature and the type of solvent[a]

Entry	T °C	Solvent	Conv.[b], %	Products distribution[b], %		
				Aldehydes[b] %	B_1/L[c], %	Octene isomers[b,d], %
1	80	CH_2Cl_2	18	94	16/84	5
2	100	CH_2Cl_2	73	93	13/87	7
3	120	CH_2Cl_2	98	82	5/95	16
4	130	CH_2Cl_2	99	80	7/93	18
5	120	Toluene	96	91	12/88	8
6	120	DMSO	75	82	7/93	17
7[e]	120	THF	98	77	21/49	19
8	120	*n*-Heptane	77	86	10/90	12
9	120	Propylene carbonate	66	89	8/92	9

[a] Reaction conditions: $Rh(CO)_2(acac)$ (0.005 mmol), ligand **I** (0.03 mmol), 1-octene (5.0 mmol), solvent (5 ml), 300 psi ($CO/H_2 = 1/2$), 1 h.
[b] Determined by GC.
[c] Determined by GC and ^1H NMR.
[d] Octene isomers include *cis*- and *trans*-2-octene, *cis*- and *trans*-3-octene, and *cis*- and *trans*-4-octene and were determined by GC–MS.
[e] Other branched aldehydes B_2 and B_3 ($B_2 = 22$, $B_3 = 8$) were formed.

The study of the effect of the type of solvent on the hydroformylation of 1-octene by the catalytic system $Rh(CO)_2$-(acac)/alkanox (**I**) is also shown in Table 1 (entries 5–9). Among the tested solvents, toluene, DMSO, *n*-heptane, and propylene carbonate led to total aldehydes yield of 91%, 82%, 86%, and 89%, with 88%, 93%, 90%, and 92% linear aldehydes, respectively (Table 1, entries 5–9). For THF, the selectivity to the linear isomer is 70%, and that of branched isomers 2-methyl octanaldehyde (IV) and 2-ethyl heptanaldehyde (V) (Eq. (2)) are 22% and 8%, respectively (Table 1, entry 7).

The results obtained showed clearly that the highest ratio ($L/B = 19$) of linear to branched aldehyde was obtained at 120 °C in CH_2Cl_2 as a solvent. DMSO is the other polar solvent that gave almost similar results to CH_2Cl_2 compared to THF and propylene carbonate that gave either lower selectivity in linear aldehyde or low conversion of 1-octene. The stability of the active rhodium intermediates $Rh_x(CO)_yI_z$ depends strongly on the type of solvent and on the temperature of the reaction.

2.2. Hydroformylation of 1-octene by $Rh(CO)_2(acac)$-I: effect of CO/H_2 pressure and reaction time

The improvement of the results of the reaction required further optimization of the reaction conditions. For instance, the effect of the ratio of CO/H_2 and the reaction time were studied with the catalytic system $Rh(CO)_2(acac)$/alkanox (**I**) (Table 2). At a total pressure of 300 psi of CO/H_2, the selectivity for the linear aldehydes decreases from 96% at $CO/H_2 = 50/250$, to 95% at $CO/H_2 = 100/200$ and finally to 84% at $CO/H_2 = 200/100$. It was observed that the percentage of the isomerized olefins decreases with

Table 2
Hydroformylation of 1-octene by $Rh(CO)_2(acac)$-I. Effect of the reaction time and the CO/H_2 partial and total pressures[a]

Entry	Time, h	CO/H_2, psi	Conv.[b] %	Products distribution[b], %		
				Aldehydes[b], %	B_1/L[c], %	Octene isomers[b,d], %
1	1	50/250	98	63	4/96	29
2	1	100/200	98	82	5/95	16
3	1	150/150	98	90	11/89	9
4	1	200/100	84	94	16/84	6
5	1	500/100	91	95	21/79	5
6	1	300/300	98	94	16/84	6
7	1	100/500	98	88	10/90	10
8	0.25	100/200	74	86	10/90	13
9	0.5	100/200	81	80	6/94	18
10	3	100/200	98	75	6/94	21
11[e]	6	100/200	100	85	15/67	11

[a] Reaction conditions: $Rh(CO)_2(acac)$ (0.005 mmol), ligand **I** (0.03 mmol), 1-octene (5.0 mmol), CH_2Cl_2 (5 ml), 120 °C.
[b] Determined by GC (products are aldehydes and isomerized octenes).
[c] Determined by GC and ^1H NMR.
[d] Octene isomers include *cis*- and *trans*-2-octene, *cis*- and *trans*-3-octene, and *cis*- and *trans*-4-octene and were determined by GC–MS.
[e] Branched aldehydes B_1 and B_2 ($B_1 = 13$, $B_2 = 5$) were formed.

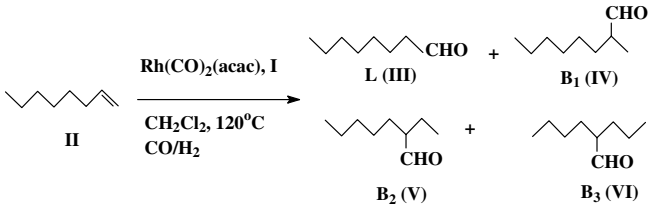
the increase of CO partial pressure (Table 2, entries 1–4). On the other hand, when the total pressure was increased to 600 psi an increase in both the conversion of 1-octene and the selectivity of the linear aldehydes was observed. At 600 psi, a decrease in the partial pressure of CO from 500 ($CO/H_2 = 5/1$) to 300 ($CO/H_2 = 1/1$) and to 100 psi ($CO/H_2 = 1/5$) maintains the conversions of 1-octene high (91%, 98%, and 98%, respectively), but with the increase an increase in the percentage of the linear isomer from 79 to 84 and finally to 90%, respectively (Table 2, entries 6–8). No products were obtained in the absence of hydrogen.

The dependence of B/L ratio on the CO partial pressure can be explained by the fact that the isomerization of alkene occurs only when β-elimination of the branched Rh-alkyl compound takes place. This β-H elimination reaction, facilitated at low CO pressures [15], converts the branched alkylrhodium into the 2-octenes. Thus, at low pressures, a substantial amount of the branched alkylrhodium complex does not proceed to form the branched aldehydes and consequently (at the incomplete conversions), the percentage of the linear aldehydes increases. However, the decrease of the isomerization rate at high CO pressures cannot totally account for the loss of selectivity (Table 2, entries 4,6,7). The presence of rhodium complexes that contain only one or even no rhodium–phosphorous bonds may lead to this result, because these complexes are expected to react with low selectivity and high rate [16].

Interestingly, at longer reaction time (6 h) the selectivity towards the linear aldehyde dropped to 82% compared to 95% and 94% at 3 h, 1 h and 0.5 h, respectively (Table 2, entries 2, 10–11). No major changes were observed between 0.5 h and 1 h; however, the high selectivity in linear aldehyde was maintained after 1 h of reaction. The results can be explained by the low stability and the decomposition of the alkanox ligand or Rh-alkanox intermediates at longer reaction time.

2.3. Hydroformylation of 1-octene by $Rh(CO)_2(acac)$-I: effect of ligand to rhodium complex molar ratio

The alkanox **I** as a ligand was applied in the rhodium-catalyzed hydroformylation of 1-octene. The reaction was carried out by adding the required amount of the catalyst $Rh(CO)_2(acac)$ and ligand **I**, CO (100 psi) and H_2 (200 psi) at 120 °C for 1 h. The results are shown in Table 3. A very low selectivity for the linear aldehydes (43%) was obtained in the absence of the ligand, due to the contribution of other side reactions such as hydrogenation and isomerization of 1-octene. In the absence of ligand $Rh(CO)_2(acac)$ is converted to $HRh(CO)_4$, which is a good isomerization and hydrogenation catalyst [17]. Interestingly, the addition of 0.01 mmol of **I** (i.e. L/[Rh] = 2) significantly improved the selectivity for the linear aldehydes to 62%. At a ratio of L/[Rh] of 6, maximum selectivity for the linear aldehydes of 95% obtained, further addition of I (L/[Rh] = 12) resulted in a decreased in linearity (89%).



(2)

Table 3
Hydroformylation of 1-octene catalyzed by $Rh(CO)_2(acac)$-I. Effect of the ligand to rhodium complex molar ratio[a]

Entry	Ligand/catalyst ratio	Conv.[b], %	Products distribution[b], %			
			Aldehydes[b], %	B_1/L[c], %	Octene isomers[b,d]	*n*-Octane[b], %
1[e]	0	99	24	37/43	30	46
2[f]	1	99	46	34/49	39	15
3[g]	2	99	61	25/62	25	14
4[h]	4	99	80	13/85	19	1
5	6	98	82	5/95	16	2
6	12	98	90	11/89	9	0

[a] Reaction conditions: $Rh(CO)_2(acac)$ (0.005 mmol), ligand **I** (0.0–0.06 mmol), 1-octene (5.0 mmol), CH_2Cl_2 (5 ml), 300 psi ($CO/H_2 = 1/2$), 120 °C, 1 h.
[b] Determined by GC.
[c] Determined by GC and ^1H NMR.
[d] Octene isomers include *cis*- and *trans*-2-octene, *cis*- and *trans*-3-octene, and *cis*- and *trans*-4-octene and were determined by GC–MS.
[e] Other branched aldehydes B_2 and B_3 ($B_2 = 11\%$, $B_3 = 9\%$) were formed.
[f] Other branched aldehydes B_2 and B_3 ($B_2 = 10\%$, $B_3 = 7\%$) were formed.
[g] Other branched aldehyde B_2 (12%) (13%) was formed.
[h] Other branched aldehyde B_2 (2%) was formed.

It has been found that a 10-fold amount of bulky diphosphite with 2,2′-biphenol backbone was required to reach a total conversion with Rh(COD)(acac) although only one phosphite ligand can coordinate to the rhodium center [14]. Further addition of the diphosphite does not affect the rate of the reaction. Similarly, it was found that a ligand-to-rhodium ratio of 5 is the minimum amount of ligand needed. The use of lower L/[Rh] resulted into the formation of a large amount of 2-octene and other aldehyde isomers [16].

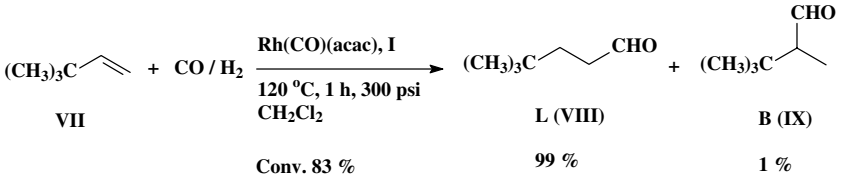
2.4. Hydroformylation of 1-octene by $Rh(CO)_2(acac)$-I: effect of the type of ligand

Other phosphine ligands have been considered in the reaction of the hydroformylation of 1-octene during the optimization process (Table 4). Most of monophosphines such as PPh_3, $P(OPh)_3$, and 2,4-di-*tert*-butyl triphenylphosphite gave excellent conversions (98%, 99%, and 99%, respectively), but the selectivity for the linear aldehydes was very poor (73%, 76% and 49%, respectively), because of the isomerization of 1-octene (Table 4, entry 5–8). In addition, the diphosphine ligands such as dppp and dppb (Table 4, entries 2, 3) gave very low conversion (55% and 14%, respectively). The experimental results indicated that **I** was by far the most active among the tested phosphine and phosphite ligands for this reaction. A maximum conversion (98%) and selectivity for nonanaldehydes (95%) was obtained in CH_2Cl_2 as a solvent and with $Rh(CO)_2(acac)$ as a catalyst and alkanox **I** base ligand.

It is important to note that both conversion and selectivity into linear aldehydes (98% and 95%, respectively) were much higher than those reported earlier using the same ligand (conversion = 69.4%, selectivity = 62%) [12].

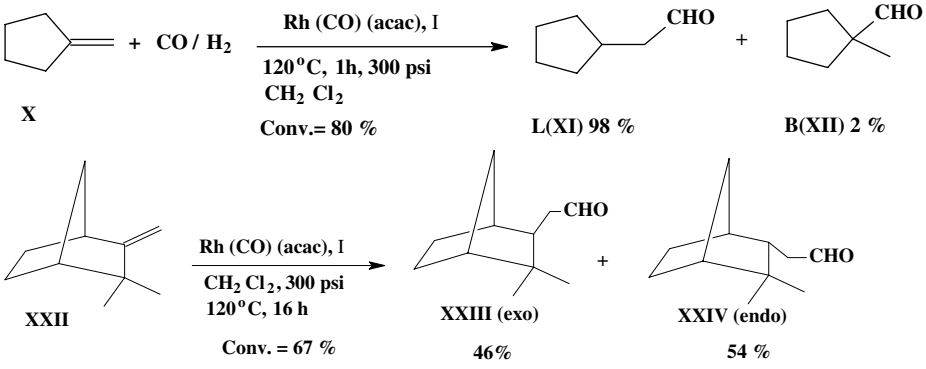
2.5. Hydroformylation of various olefins by $Rh(CO)_2(acac)$-I

The hydroformylation of a variety of alkenes occurred smoothly at relatively mild conditions [120 °C, CO/H_2(100/200 psi), 1 h] with $Rh(CO)_2(acac)$ as a catalyst precursor and alkanox **I** as a ligand in dichloromethane as a solvent. For example, alkyl alkenes containing tertiary carbon atom bonded to the vinyl group, such as 3,3-dimethyl-1-butene (VII), showed excellent regioselectivity toward the linear aldehyde (VIII) (Eq. (3)). The main reason for such behavior was the steric effect, which prevents the addition of CHO moiety on the internal carbon of the double bond.



VII — Conv. 83 % — L (VIII) 99 % — B (IX) 1 % (3)

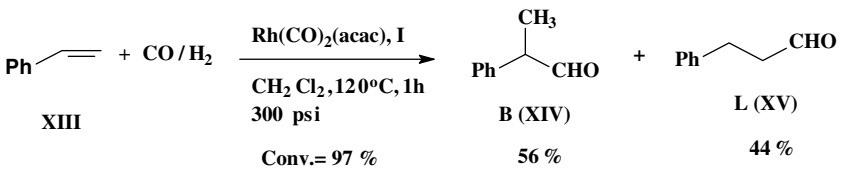
Similarly, methylene cylopentane (X) was hydroformylated to give predominately the linear aldehyde, 2-cylopentyl ethanal L (XI), with only traces of the branched aldehyde, 1,1-cyclopentyl methyl methanal B (XII) (Eq. (4)). Camphene (XXII) also undergoes the hydroformylation by the catalytic system $Rh(CO)_2(acac)$/alkanox (**I**) to form the two linear aldehydes isomers XXIII (*endo*) and XXIV (*exo*). A conversion of 67% was achieved after 16 h. with a ratio of *endo*/*exo* of 1.2 (Eq. (5)).



(4)

(5)

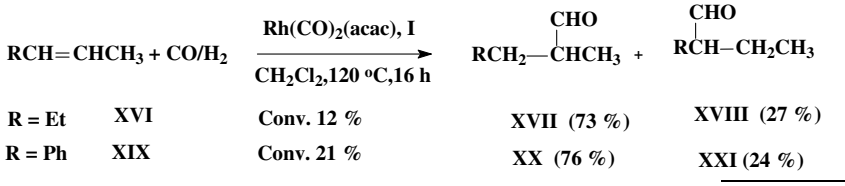
The hydroformylation of styrene (XIII) occurred smoothly at the same experimental conditions in dichlomethane (Eq. (6)). As expected, the n/i ratio was poor (0.8) due to the preference for a branched alkyl rhodium intermediate or electronically stabilized η^3-benzyl intermediate [15].



(6)

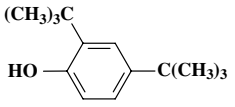
The catalyst system $Rh(CO)_2(acac)$/alkanox (**I**) was also applied for the hydroformylation of internal alkenes such as 2-pentene and β-methylstyrene (Eq. (7)) and showed a lower activity, with the conversions of 12% and 21%, respectively after 16 h. Similarly, low conversion (25%) and moderate selectivity into linear aldehyde (60%) were reported using $PtCl_2(cod)/SnCl_2$/alkanox (**I**) catalytic system in the hydroformylation of 2-butene [13].

and pentaerythriol which occurs at longer reaction time (>1 h) [11]. In fact, the reaction of the hydroformylation of 1-octene in the presence of the catalytic system $Rh(CO)_2(acac)$/alkanox P-24 (**I**) (0.03 mmol) was carried out in CH_2Cl_2 at 120 °C for 1 h. The analysis of the products of the reaction by GC–MS indicated clearly the formation of 0.03 mmol of 2,4-*tert*-butyl phenol (**XXII**) ($m/z = 206$) that corresponds of the hydrolysis of the ligand **I** [11,18].



(7)

The reasons for not being able to reach full conversion of camphene (XXII) and the low conversion of the internal alkenes (XVI and XIX) is affected by the loss of the coordinative ability of ligand (**I**) due to its partial or complete hydrolysis into 2,4-*tert*-butyl phenol, phosphorous acid



XXII

Table 4
Hydroformylation of 1-octene. Effect of the type of ligand[a]

Entry	[Ligand] mmol	Conversion[b], %	Products distribution[b], %		
			Aldehydes[b], %	B_1/L[c], %	Octene Isomers[b,d], %
1	**Alkanox** (0.03)	98	82	5/95	16
2	**dppp** (0.03)	55	53	25/75	47
3	**dppb** (0.03)	14	100	27/73	0
4[e]	**PPh_3** (0.06)	98	84	25/73	16
5[f]	**$P(OPh)_3$** (0.06)	99	70	18/76	25
6	**PBu_3** (0.06)	14	97	28/72	3
7[g]	**$P(2,4\text{-di}^{-}Bu^tC_6H_4O)_3$** (0.06)	99	69	32/49	26

[a] Reaction conditions: $Rh(CO)_2(acac)$ (0.005 mmol), 1-octene (5.0 mmol), CH_2Cl_2 (5 ml), 100/200 ($CO/H_2 = 1/2$), 120 °C, 1 h.
[b] Determined by GC.
[c] Determined by GC and ^1H NMR.
[d] Octene isomers include *cis*- and *trans*-2-octene, *cis*- and *trans*-3-octene, and *cis*- and *trans*-4-octene and were determined by GC–MS.
[e] Other branched aldehyde B_2 (2%) was formed.
[f] Other branched aldehydes B_2 and B_3 ($B_2 = 5\%$, $B_3 = 1\%$) were formed.
[g] Other branched aldehydes B_2 and B_3 ($B_2 = 11\%$, $B_3 = 8\%$) were formed.

3. Conclusions

The hydroformylation of 1-octene catalyzed by $Rh(CO)_2(acac)$ and bis(2,4-di-*tert*-butyl)pentaerythritol diphosphite (alkanox P-24) (**I**) catalyst system gives excellent selectivity in linear aldehydes and an acceptable turnover in rhodium. The regioselectivity is caused by the steric repulsion of the diphosphite that coordinates to the rhodium center. The same catalyst showed very low activity and selectivity for the internal alkenes, due to the possible hydrolysis of the ligand that occurred at longer reaction time and in the presence of the rhodium catalyst.

4. Experimental

Rhodium complexes were purchased from Strem Company and used as received. Alkenes were purified by passing through neutral alumina and all other solvents were purchased from Sigma–Aldrich and were purified by distillation prior to use. Propylene carbonate was purchased from BDH chemicals and used without purification. ^1H and ^{13}C NMR spectra were recorded on a 500 MHz Joel 150 NMR

machine. Chemical shifts were reported in ppm relative to tetramethyl silane (TMS) using $CDCl_3$. Gas chromatography analyses were realized on a HP-6890-plus GC equipped with 30 m capillary column (HP-1).

4.1. General experimental procedure for the hydroformylation of alkenes

A typical experimental procedure is as follows: 5.0 mmol of 1-octene, 0.005 mmol of $Rh(CO)_2(acac)$ and 0.030 mmol of ligand were dissolved in 5.0 ml of dichloromethane and placed in the glass liner of a 45 ml Parr autoclave. The autoclave was purged three times with carbon monoxide, pressurized with 300 psi of $CO + H_2$ (1:2) and then heated in an oil bath with temperature controller fixed at 120 °C. After 1.0 h the reaction mixture was cooled to room temperature and the reaction mixtures were identified by GC and GC–MS using *n*-decane as an internal standard. The ^1H and ^{13}C NMR of the products gave excellent spectral data compared to authentic samples.

The available of a relatively small volume glass liner (45 ml) with the magnetic stirrer of average efficiency could be the main reason behind the lower turnover frequency of the rhodium catalyst. A larger reactor with a scale-up of the experiments may lead to significant improvement in the rhodium catalytic efficiency.

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