Journal of Organometallic Chemistry 695 (2010) 1869–1872

Contents lists available at ScienceDirect

Journal of Organometallic Chemistry

journal homepage: www.elsevier.com/locate/jorganchem

Rhodium phosphino-enolate complexes as chemo- and regioselective catalysts for the hydroformylation of styrenes

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ARTICLE INFO

Article history:
Received 12 March 2010
Received in revised form
25 April 2010
Accepted 27 April 2010
Available online 24 May 2010

Keywords:
Hydroformylation
Catalysis
P,O-Ligand
Rhodium
Styrene

ABSTRACT

The catalytic utility of [κ^2-{3-^{i}Pr$_2$*P*-2-*O*-indene}Rh(COD)] (COD = η^4–1,5-cyclooctadiene) **1a** in the hydroformylation of styrenes was examined. Complex **1a** was shown to be an effective pre-catalyst in benzene and tetrahydrofuran, exhibiting good conversions to aldehyde and high branched-to-linear selectivity for styrene, 4-chlorostyrene, and 4-methylstyrene under reasonably mild conditions (1000 psi syngas; 1.8 mol% Rh, 45 °C, 2–5 h). Under analogous conditions, the iridium congener of **1a** proved inactive for hydroformylation. The synthesis and crystallographic characterization of the new complex [κ^2-{2-^{i}Pr$_2$*P*C$_6$H$_4$*O*}Rh(COD)] **2** is also reported; the catalytic performance of **2** in the hydroformylation of styrene was found to be comparable to that of **1a** under similar catalytic conditions.

1. Introduction

The hydroformylation of alkenes is an atom-economical C–C bond-forming reaction that gives rise to synthetically useful aldehyde products [1]. As a result, olefin hydroformylation is currently one of the most important industrial–scale reactions, and is employed in the production of 10^6 tons of aldehydes annually that in turn are used in the synthesis of myriad commodity and fine chemicals, as well as pharmaceuticals [2]. Hundreds of complexes that combine rhodium (among other metals) with various phosphorus-based ligands have been examined as pre-catalysts for olefin hydroformylation [3]; nonetheless, the quest to establish increasing levels of catalyst activity, as well as chemo-, regio-, and/or enantioselectivity, provides the motivation for further explorations of ancillary ligand effects on such catalytic processes [4,5].

In this context, and building on our previous investigations of the 2-indanone-derived complexes **1a** and **1b** (Chart 1) in which these complexes were revealed to be effective pre-catalysts for the hydrosilylation and hydrogenation of alkenes under mild conditions [6], we became interested in surveying the utility of **1a** and **1b** in the hydroformylation of alkenes. We report herein that the rhodium complex **1a** (but not the iridium analogue **1b**) is a competent pre-catalyst under mild conditions for the hydroformylation of styrene substrates, exhibiting high branched-to-linear selectivity. We also report the synthesis and crystallographic characterization of the new and structurally related complex [κ^2-{2-^{i}Pr$_2$*P*C$_6$H$_4$*O*}Rh(COD)] **2** (COD = η^4–1,5-cyclooctadiene), as well as its use in hydroformylation catalysis.

2. Experimental

2.1. General experimental details pertaining to the synthesis of 2

All manipulations were conducted in the absence of oxygen and water under an atmosphere of dinitrogen, either by use of standard Schlenk methods or within a glovebox apparatus, utilizing glassware that was oven-dried (130 °C) and evacuated while hot prior to use. The tetrahydrofuran (THF) and pentane (both purchased from VWR) were deoxygenated and dried by sparging with dinitrogen gas, followed by passage through a double-column solvent purification system purchased from mBraun Inc.; THF was purified over two alumina-packed columns, while pentane was purified over one alumina-packed column and one column packed with copper-Q5 reactant. The purified solvents were stored over activated 3 Å

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doi:10.1016/j.jorganchem.2010.04.032

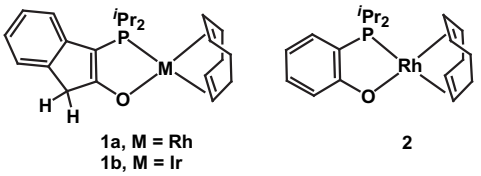


Chart 1.

molecular sieves. C_6D_6 (Cambridge Isotopes) was degassed by using three repeated freeze-pump-thaw cycles, and dried over 3 Å molecular sieves for 24 h prior to use. $[(COD)RhCl]_2$ was purchased from Strem Chemicals and was dried under vacuum for 24 h prior to use. The characterization of **2** was carried out at 300 K on a Bruker AV-500 spectrometer operating at 500.1, 125.8, and 202.5 MHz for ^1H, ^{13}C and ^{31}P (respectively) with chemical shifts reported in parts per million downfield of $SiMe_4$ (for ^1H and ^{13}C) or 85% H_3PO_4 in D_2O (for ^{31}P).

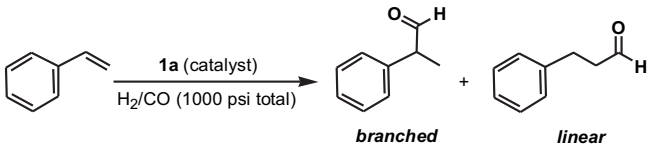
2.2. Synthesis of 2

Using a synthetic protocol similar to that used for the preparation of the analogous iridium complex [κ^2-{2-^{i}Pr$_2$*P*C$_6$H$_4$O}Ir(COD)] [6], to a magnetically stirred solution of 2-^{i}Pr$_2$PC$_6$H$_4$OH (0.148 g, 0.702 mmol) in THF (2 mL) was added a THF solution of $[(COD)RhCl]_2$ (0.173 g, 0.351 mol). After 10 min of stirring, NEt_3 (98 μL, 0.702 mmol) was added causing the reaction mixture to turn bright yellow in color. After 3 h of stirring at ambient temperature, ^{31}P NMR analysis of the reaction mixture indicated the complete consumption of 2-^{i}Pr$_2$PC$_6$H$_4$OH and the presence of a new phosphorus-containing species. The reaction mixture was concentrated under vacuum and the remaining solid residue was extracted into pentane (4 × 2 mL). The pentane fractions were combined and removal of the solvent afforded **2** as an analytically pure orange solid (0.192 g, 0.456 mmol, 65%). Anal. Calcd for $C_{20}H_{30}PORh$: C 57.13, H 7.20 N 0.00. Found: C 56.99 H 7.16N < 0.3. ^1H NMR (C_6D_6): 7.20 (m, 1H, Ar–H), 7.15 (m, 1H, Ar–H), 6.93 (dt, $^3J_{HH} = 8.0$ Hz, $^4J_{HH} = 1.5$ Hz, 1H, Ar–H), 6.57 (m, 1H, Ar–H), 5.58 (m, 2H, C*H*(COD)), 3.67 (m, 2H, C*H*(COD)), 2.24 (m, 2H, CH_2(COD)), 2.12 (m, 2H, CH_2(COD)), 1.97–1.84 (m, 4H, P(C*H*Me$_2$)$_2$ and CH_2(COD)), 1.72 (m, 2H, CH_2(COD)), 1.03 (dd, $^3J_{PH} = 16.5$ Hz, $^3J_{HH} = 7.0$ Hz, 6H, P(CH*Me*$_a$Me$_b$)$_2$, 0.96 (dd, $^3J_{PH} = 14.0$ Hz, $^3J_{HH} = 6.5$ Hz, 6H, P(CHMe$_a$*Me*$_b$)$_2$); $^{13}C\{^1H\}$ NMR (C_6D_6): 179.2 (d, $J = 25.2$ Hz, Ar-quat.), 132.5 (d, $J = 1.8$ Hz, Ar–*C*H), 130.2 (Ar–*C*H), 118.3 (dd, $J = 10.7$, 2.1 Hz, Ar–*C*H), 113.5 (d, $J = 5.9$ Hz, Ar–*C*H), 113.7 (d, $J = 35.2$ Hz, Ar-quat.), 102.9 (dd, $J = 10.4$, 7.5 Hz, *C*H(COD)), 64.2 (dd, $J = 11.3$, 1.4 Hz, *C*H(COD)), 32.8 (d, $J = 2.3$ Hz, *C*H_2(COD)), 27.2 (d, $J = 1.5$ Hz, *C*H_2(COD)), 23.4 (dd, $J = 22.4$, 2.0 Hz, P(*C*H(Me$_2$)$_2$), 17.5 (d, $J = 5.4$ Hz, P(CH*Me*$_a$Me$_b$)$_2$), 16.7 (P(CHMe$_a$*Me*$_b$)$_2$); $^{31}P\{^1H\}$ NMR (C_6D_6): 48.6 (d, $^1J_{RhP} = 160$ Hz). Crystals suitable for X-ray crystallography were grown from a concentrated solution of **2** in pentane at −35 °C.

2.3. Experimental details and representative protocol for hydroformylation experiments

Under inert atmosphere of dinitrogen, solvents were dried via distillation from the drying agents listed as following – hexane from sodium metal; dichloromethane and benzene from calcium hydride. CH_3CN (≥99.9%) was purchased from Sigma Aldrich. All air and moisture sensitive reactions were conducted under an inert atmosphere (nitrogen or argon passed through activated drierite and potassium hydroxide) in flame-dried glassware using standard Schlenk techniques. All catalytic reactions were set up in MBraun or Vacuum Atmosphere gloveboxes under dinitrogen. High-pressure experiments were carried out in a Parr stainless steel apparatus equipped with an oven-dried glass liner. All gaseous reagents H_2/CO (50 ± 5% mix, 99.999% H_2, 99.9% CO) and CO_2 (99.9999% SFC/SFE grade) were purchased from Praxair and used directly. All reactions were performed using 31 mL high pressure steel vessels. Styrene was passed through a pre-packed aluminium oxide column before use to remove inhibitor; this substrate was not purified further. 4-Chlorostyrene and 4-methoxystyrene were purified by distillation under partial vacuum and percolated through a column of basic alumina. All vessels and glassware were washed, dried at 160 °C overnight, cooled under vacuum and then stored in a glovebox before use. THF was obtained from Fisher Scientific and passed through a double-column solvent purification system purchased from Innovative Technologies, Inc. prior to use as solvent for hydroformylations. The solvent and styrene substrates were

Table 1
Hydroformylation of styrene using **1a**.[a]



Entry	Solvent	Loading (mol % 1a)	Additive	Temp.	Time (h)	Conversion (%)[b]	B: L[b]
1	C_6H_6	1.8	–	80 °C	5	>99	83: 17
2	C_6H_6	1.8	–	45 °C	5	>99	95: 5
3	C_6H_6	1.8	–	20 °C	5	14	97: 3
4	C_6H_6	1.8	–	45 °C	2	90	95: 5
5	C_6H_6	1.8	–	45 °C	1.5	81	95: 5
6	C_6H_6	1.8	1eq PPh_3	45 °C	1.5	9	94: 6
7	THF	1.8	–	45 °C	2	98	95: 5
8	THF	1.8	–	45 °C	1.5	93	95: 5
9	THF	1.8	1eq PPh_3	45 °C	1.5	11	94: 6
10	THF	1.8	–	20 °C	5	24	97: 3
11	THF	1.8	CO_2 (60 bar)	20 °C	5	37	96: 4
12	THF	0.35	–	45 °C	5	79	94: 6
13	CH_2Cl_2	1.8	–	45 °C	1.5	37	95: 5
14	Hexanes	1.8	–	45 °C	1.5	32	95: 5
15	CH_3CN	1.8	–	45 °C	1.5	24	95: 5

[a] Reactions employing 1000 psi syngas.
[b] Conversion to aldehyde and branched-to-linear ratio (B : L) determined on the basis of ^1H NMR data using hexamethylbenzene as an internal standard.

separately degassed using standard techniques (freeze-pump-thaw), and then stored at −20 °C in the glovebox until use. The catalyst (1.8 mol%) and substrate were weighed out in separate 4 dram vials; 2 mL of THF was added to the substrate and then the resulting solution was transferred to the vial containing the catalyst. The mixture was stirred with a stir bar for 10 min. The entire vial was then placed into the steel vessel, which was assembled and tightened before being removed from the glovebox. The vessel equilibrated in a 45 °C water bath for 40 min. The vessel was then re-tightened and pressurized to 70 bar with a 1:1 mix of CO and H_2. The reaction was stirred using a stir bar at 650 rpm and 45 °C for 5 h. The vessel was moved to an ice bath for 1 h and then depressurized. The solvent was allowed to evaporate slowly to prevent the loss of product. Samples of the residue were evaluated by use of NMR spectroscopic methods in $CDCl_3$.

3. Results and discussion

The hydroformylation of styrene was selected as a test reaction for evaluating the catalytic utility of **1a** (Table 1). Complex **1a** displayed good activity and high chemoselectivity for aldehydes, with the expected branched aldehyde being favored in reactions conducted at 80 °C in benzene (entry 1); the alkene insertion step leading to the formation of the sterically disfavored branched product is thought to be promoted by the favorable formation of (η^3-benzyl)Rh species [1,3]. Notably, full conversion and increased regioselectivity were also achieved when employing **1a** as a catalyst under similar conditions at 45 °C (entry 2), in keeping with previously observed trends in rhodium-catalyzed hydroformylation [7]. The analogous iridium complex **1b** exhibited negligible catalytic activity for the hydroformylation of styrene under similar conditions (12 h, 45 °C, 1.8 mol% Ir). While increased regioselectivity (97:3) was achieved in reactions conducted at 20 °C using **1a**, the conversion to aldehyde under these conditions proved to be unacceptably low (entry 3); by comparison, Breit and co-workers [4a] have disclosed a rhodium catalyst system that allows for the regioselective hydroformylation of alkenes under ambient temperature and pressure conditions.

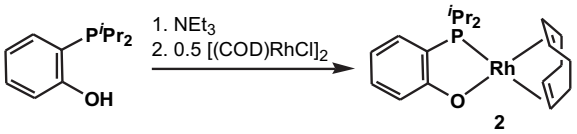
Whereas the addition of phosphine and phosphite co-ligands has been shown to augment the catalytic performance of some rhodium hydroformylation catalysts [3], the inclusion of triphenylphosphine into the catalytic system resulted in a dramatic loss in catalytic activity in both benzene and tetrahydrofuran (entry 5 vs. entry 6; entry 8 vs. entry 9).

Solvent effects on the reaction performance were particularly evident at 20 °C because of the lower conversion observed at that temperature. Reactions conducted in tetrahydrofuran at 20 °C afforded improved (albeit modest) conversion to the aldehyde with high regioselectivity (entry 10) relative to analogous experiments conducted in benzene (entry 3). The inclusion of liquid CO_2 afforded further gains in conversion (entry 11). At 45 °C, the use of **1a** in tetrahydrofuran enabled reactions to be conducted at lower loadings than could be achieved in benzene (entry 12); conversely, dichloromethane, hexanes, and acetonitrile each proved to be a less suitable reaction medium (entries 13–15).

Table 2
Hydroformylation of alternative alkene substrates using **1a**.[a]

Loading (mol % 1a)	Substrate	Time (h)	Conversion (%)[b]	B: L[b]
0.5	4-chlorostyrene	12	71	95: 5
0.5	4-methylstyrene	12	87	94: 6
0.5	6-methoxy-2-vinylnapthalene	12	10	99: 1
1.8	1-decene	2	53[c]	1: 1.6

[a] Reactions employing 1000 psi syngas in THF at 45 °C.
[b] Conversion to aldehyde and branched-to-linear ratio (B : L) determined on the basis of ^{1}H NMR data using hexamethylbenzene or 1,4-dimethoxybenzene as an internal standard.
[c] Trace amounts of alkene isomerization product observed.



Scheme 1. Preparation of 2.

Having established the catalytic utility of **1a** in the hydroformylation of styrene, alternative olefinic substrates were examined (Table 2). While the branched regioselectivity remained high for reactions employing 4-chlorostyrene, 4-methylstyrene, and 6-methoxy-2-vinylnapthalene, longer reaction times were required and in the case of the last substrate, low conversions were achieved. Efforts to extend these highly regioselective hydroformylations to the aliphatic substrate 1-decene were unsuccessful; while the conversion was reasonable, the regioselectivity was poor.

In an effort to evaluate the possible influence of the indene backbone in **1a** on catalytic performance, the alternative [(COD)Rh(κ^2-*P,O*)] complex **2** was prepared in 65% isolated yield from the corresponding ortho-substituted phenol and $[(COD)RhCl]_2$ in the presence of triethylamine (Scheme 1). The structure of **2** is in keeping with the proposed connectivity, and is supported by single-crystal X-ray diffraction data. An ORTEP [8] diagram of **2** is presented in Fig. 1, while X-ray experimental data are collected in Table 3. The overall structural features in **2** compare well with those observed in both the analogous Ir complex [6], as well as in **1a** [9]. Notably, the Rh-alkene distances *trans* to P (2.198(3) and 2.191(3) Å) are statistically longer than the Rh-alkene distances *trans* to O (2.116(3) and 2.126(3) Å), in keeping with the greater *trans*-influence predicted for a phosphine fragment relative to an alkoxy donor on rhodium [10].

Hydroformylation experiments employing **2** as a pre-catalyst under conditions analogous to those featured in Table 1, entry 7 for **1a** afforded catalytic results for **2** that were only slightly more selective (B:L ratio of 97:3) than those obtained by using **1a**, thereby suggesting that the κ^2-*P,O* backbone of the phosphino-enolate ancillary ligand has little influence over the catalytic behavior of the associated rhodium center. Nonetheless, the desirable catalytic performance exhibited by **1a** and **2** is in contrast to structurally

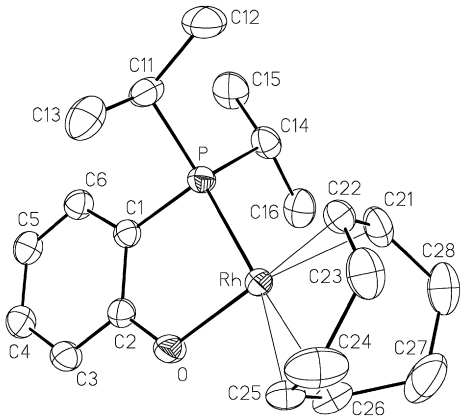


Fig. 1. The crystallographically determined structure of 2, depicted with 50% thermal ellipsoids and with the hydrogen atoms removed for clarity. Selected interatomic distances (Å) for 2: Rh-P 2.2938(8); Rh-O 2.048(2); Rh-C21 2.116(3); Rh-C22 2.126(3); Rh-C25 2.198(3); Rh-C26 2.191(3).

Table 3
Crystallographic data for 2.

Empirical formula	$C_{20}H_{30}O_1P_1Rh_1$
Formula weight	420.32
Crystal dimensions	$0.39 \times 0.18 \times 0.08$ mm^3
Color, habit	yellow, prism
Crystal system	monoclinic
Space group	$P2_1/n$
a (Å)	9.9409(10)
b (Å)	15.9331(15)
c (Å)	12.0772(12)
β (deg)	94.5317(12)
V (Å^3)	1906.9(3)
Z	4
ρcalcd (g cm^{-3})	1.464
μ (mm^{-1})	0.982
Range of transmission	0.9256–0.7007
2θ limit (deg)	54.94
	$-12 \le h \le 12$
	$-20 \le k \le 20$
	$-15 \le l \le 15$
Total reflections	14 626
Independent reflections	4354
R(int)	0.0412
Observed reflections	3459
Data/restraints/parameters	4354/0/208
Goodness-of-fit	1.042
$R1$ [$Fo^2 \ge 2\sigma(Fo^2)$]	0.0346
$wR2$ [$Fo^2 \ge -3\sigma(Fo^2)$]	0.0840
Largest peak, hole (eÅ−3)	0.773, −0.465

related complexes of the type [Rh(PO)(CO)L] (PO = κ^2-{2-$Ph_2PC_6H_4O$}; L = $HOC_6H_4PPh_2$ or PPh_3), which proved inactive in the hydroformylation of 1-alkenes [10]. Given that the addition of PPh_3 to **1a** resulted in diminished catalytic performance (vide supra) and that **1a** [present work] and [Rh(PO)(P{OPh}$_3$)$_2$] [10] are capable of mediating the hydroformylation of 1-alkenes, it is plausible that the poor performance of the [Rh(PO)(CO)L] complexes reported by Trzeciak et al. [10] can be attributed to the presence of the triphenylphosphine co-ligand, rather than to the phosphino-enolate ancillary ligand as proposed by the authors. Mechanistic studies of alternative Rh(κ^2-*P,O*) catalysts under hydroformylation conditions reveal that Rh-O cleavage [11], Rh-P cleavage [12], and complete loss of the P,O-ligand [10] are all feasible under catalytic hydroformylation conditions; in the absence of such mechanistic data, we are unable to comment definitively regarding the fate of **1a** and **2** under the catalytic conditions reported herein.

4. Summary and conclusions

The results reported herein confirm that the known rhodium complex **1a**, as well as the newly reported complex [κ^2-{2-^{i}Pr$_2$*P*-C_6H_4O}Rh(COD)] **2**, are competent pre-catalysts for the hydroformylation of styrene substrates under mild conditions, exhibiting high branched-to-linear selectivity under relatively mild conditions. In contrast, the iridium analogue of **1a** (i.e. **1b**) proved ineffective in mediating the hydroformylation of styrene under comparable conditions. Encouraged by the performance of the rhodium complexes featured herein, future work will target chiral variants of **1a** and **2**, for use in mediating asymmetric olefin hydroformylation reactions.

Acknowledgement

Acknowledgment is made to Dalhousie University and the Natural Sciences and Engineering Research Council of Canada (including Discovery Grants for M. S. and P. G. J. and a Canada Graduate Scholarship for K. D. H.). We also thank Dr. Michael Lumsden (NMR3, Dalhousie) for assistance in the acquisition of NMR data. P. G. J. also thanks the Canada Research Chairs program.

Appendix. Supplementary data

Crystallographic Solution and Refinement Details for 2. Crystallographic data were obtained at 193(±2) K on a Bruker PLATFORM/SMART 1000 CCD diffractometer using a graphite-monochromated Mo Kα ($\lambda = 0.71073$ Å) radiation, employing a sample that was mounted in inert oil and transferred to a cold gas stream on the diffractometer. Programs for diffractometer operation, data collection, data reduction, and multi-scan absorption correction (including SAINT and SADABS) were supplied by Bruker. The structure was solved by use of a Patterson search/structure expansion, and the refinement was carried out employing full-matrix least-squares procedures on F^2. Anisotropic displacement parameters were employed throughout for the non-hydrogen atoms, and all hydrogen atoms were added at calculated positions and refined by use of a riding model employing isotropic displacement parameters based on the isotropic displacement parameter of the attached atom. Crystallographic data for the structural analysis has been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 767857 for compound **2**.

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2010.04.032

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