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Preparation and catalytic hydrogenation properties of some rhodium and ruthenium complexes with chiral tridentate phosphine ligands (*S*,*S*)-PhP($CH_2CHMeCH_2PPh_2$)$_2$ and (*S*)-$Ph_2PCH_2CH(PPh_2)CH_2CH_2PPh_2$)

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Abstract

Treatment of RhCl(ttp*) (ttp* = (*S*,*S*)-PhP($CH_2CHMeCH_2PPh_2$)$_2$) with $NaBH_4$ produced $Rh(BH_4)$(ttp*). The reaction of $RuCl_2$(ttp*) with $NaBH_4$ produced $RuH(BH_4)$(ttp*). The complexes RhCl(ttp*), $RuCl_2$(ttp*), $Rh(BH_4)$(ttp*), and $RuH(BH_4)$(ttp*) were found to be catalytically active for the hydrogenation of α-acetamidocinnamic acid.

Keywords: Catalytic hydrogenation; Rhodium; Ruthenium; Chiral tridentate phosphines

1. Introduction

Complexes with chiral phosphine ligands have been extensively studied for asymmetric catalysis [1]. Most of the previous investigations are based on complexes with chiral bidentate phosphine ligands. These studies have shown that chiral bidentate phosphines are generally more efficient in inducing asymmetric reactions than chiral monodentate phosphines. The ligand chelation is believed to play an important role by restricting the number of competing asymmetric conformations surrounding a metal center. In this regard, it would be interesting to prepare complexes with chiral tridentate phosphine ligands and to investigate their chemical and catalytic properties. However, such studies are still rather limited [2–11], despite the fact that tridentate phosphines have been widely used in organometallic and coordination chemistry [12]. Closely related chiral tridentate ligands, for example, chiral PCP [13], PNP [14], and PSP [15] (P, C, N, S denote donor atoms) type ligands, have recently been exploited for asymmetric catalysis. Some promising results have been obtained with these chiral tridentate ligand systems. We wish to report here the catalytic hydrogenation properties of some ruthenium and rhodium complexes with chiral tridentate phosphines (*S*,*S*)-PhP($CH_2CHMeCH_2PPh_2$)$_2$ (ttp*) [11] and (*S*)-$Ph_2PCH_2CH(PPh_2)CH_2CH_2PPh_2$ (etp*) [9], and the synthesis of the new hydride complexes $Rh(BH_4)$(ttp*) and $RuH(BH_4)$(ttp*).

2. Results and discussion

2.1. Synthesis and characterization of new ttp* complexes of rhodium and ruthenium

The chiral complexes used in this study include RhCl(ttp*) (**1**), $RuCl_2$(ttp*) (**2**), $RuCl_2$(etp*) (**3**), $Rh(BH_4)$(ttp*) (**4**), and $RuH(BH_4)$(ttp*) (**5**) (see Chart 1 for structures). Complexes **1** [11], **2** [11], and **3** [3] have been reported previously.

The new rhodium hydride complex $Rh(BH_4)$(ttp*) (**4**) was prepared by treating **1** with excess $NaBH_4$ in methanol. The complex was characterized by IR, NMR and elemental analysis. The triphosphine is meridonally coordinated to the rhodium, as indicated by the observation of a large $J(Ph_2P–Ph_2P)$ coupling constant.

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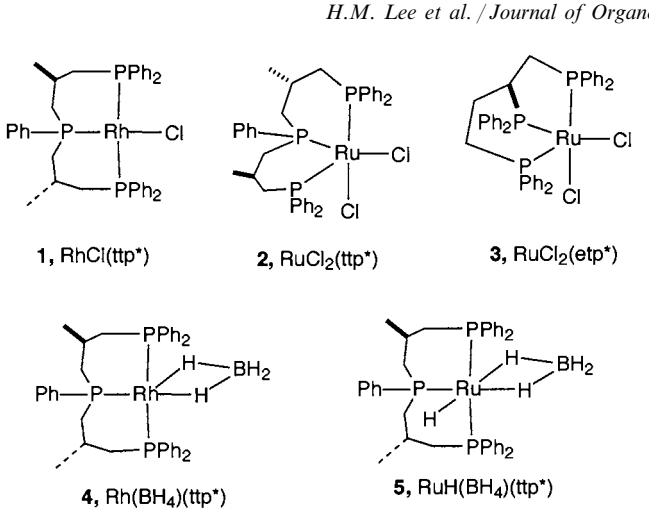


Chart 1

The chemical shift of the central P atom in **4** is shifted upfield compared with that in RhCl(ttp*) (**1**). Such a shift is consistent with the change of the chloride ligand in **1** to the hydride ligand in **4**. It has been noted that strong-field ligands cause the *trans* phosphine resonance to occur further upfield, compared with a similar complex with a halide *trans* to the phosphine [16]. In the ^{1}H-NMR spectrum in C_6D_6, a broad signal assignable to BH_4 protons was observed at 0.31 ppm. The assignment was confirmed by ^{2}D-NMR for the sample prepared by reacting **1** with $NaBD_4$. The observation of only one broad signal of the BH_4 ligand suggests that there exists an exchange process making all the BH_4 protons equivalent. Fluxional behavior of BH_4 complexes is well documented [17]. The four BH_4 protons in complex **4** must exchange at a rate faster than the NMR time scale even at 173 K in toluene-d_8, as separation of the broad BH_4 signal could not be achieved at this temperature. The ^{1}H- and ^{31}P-NMR spectroscopic data are consistent with the formulation of the fluxional 18 electron complex Rh(η^2-BH_4)(ttp*), although the alternative structure Rh(η^1-BH_4)(ttp*) could not be excluded. The BH_4 ligands in the related compounds $Rh(BH_4)(CO)(PR_3)_2$ were also assumed to be bidentate [18].

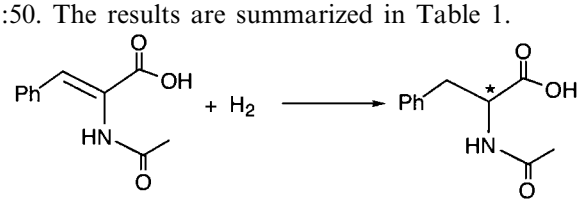
Reactions of RhCl(ttp) (ttp = $PhP(CH_2CH_2CH_2PPh_2)_2$) and RhCl(etp) (etp = $PhP(CH_2CH_2PPh_2)_2$) with $NaBH_4$ in ethanol were reported to give RhH(ttp) and $Rh(BH_3)$(etp), respectively [19]. We have attempted to synthesize the monohydride complex RhH(ttp*) by reactions of RhCl(ttp*) with lithium hydride or ethanolic KOH. However, no pure compound could be obtained from these reactions.

The new ruthenium hydride complex $RuH(BH_4)$(ttp*) (**5**) was obtained from the reaction of $RuCl_2$(ttp*) with excess $NaBH_4$. The spectroscopic data are consistent with a structure in which the ttp* ligand is coordinated in a meridional geometry and the BH_4 ligand in an η^2 fashion. Consistent with the structural assignment, the ^{31}P-NMR spectrum in C_6D_6 exhibited a triplet for the central PPh and two doublets of doublets for the magnetically non-equivalent terminal PPh_2 groups with $J(Ph_2P–Ph_2P)$ of 266 Hz. In the ^{1}H-NMR spectrum, the hydride signal was observed at -15.95 ppm as a doublet of triplet (^2J(P–H) = 21.6, 34.2 Hz). The two bridging BH proton signals were observed at -7.39 and -5.62 ppm. The terminal BH_2 proton signal was observed at 5.39 ppm. Similar ruthenium BH_4 complexes have been reported [20–23], for example, $RuH(BH_4)(PhP(CH_2CH_2CH_2PPh_2)_2)$ [20], $RuH(BH_4)(CH_3C(CH_2PPh_2)_3)$ [21], $RuH(BH_4)(PMe_2Ph)_3$ [22].

Attempts have been made to synthesize RuHCl(ttp*) by reactions of $RuCl_2$(ttp*) with lithium hydride or thallium formate; unfortunately no pure compounds could be obtained.

2.2. Catalytic hydrogenation reactions

To evaluate the catalytic properties of metal complexes with ttp* and etp*, hydrogenation of the prochiral substrate, α-acetamidocinnamic acid, was carried out (Eq. (1)). The catalytic reactions were performed in an autoclave under a hydrogen pressure of 50 bar at room temperature with the catalyst to substrate ratio of 1:50. The results are summarized in Table 1.



(1)

The experimental results show that RhCl(ttp*), $RuCl_2$(ttp*), $Rh(BH_4)$(ttp*), and $RuH(BH_4)$(ttp*) are all catalytically active for hydrogenation of α-acetamidocinnamic acid. Quantitative production of *N*-acetylphenylalanine was achieved after 24 h in MeOH under 50 bars of hydrogen pressure. The optical yields, however, are moderate. The tripodal tridentate phosphine complex $RuCl_2$(etp*) has a poor catalytic activity for the hydrogenation of α-acetamidocinnamic acid. Under similar conditions, the percentage of conversion was just 11% producing a nearly racemic mixture (entry 10).

It is of interest to note that the enantioselectivity is different with Ru and Rh complexes containing the ttp* ligand. The Ru complexes promote the production of *N*-acetylphenylalanine with (*S*)-configuration in excess, while the Rh complexes promote the production of *N*-acetylphenylalanine with the (*R*)-enantiomer predominantly. Such an inversion of enantioselectivity has also been observed in the hydrogenation of α-acetamidocinnamates by Ru [24] and Rh [25] complexes containing BINAP.

As seen in Table 1, the catalytic activity of RhCl(ttp*) was influenced by solvents. For example,

quantitative conversion was obtained in MeOH (entry 2), but only 16% conversion could be obtained in toluene (entry 3). The observation implies that dissociation of the chloride ligand might occur in the catalytic reactions, at least in polar solvents such as methanol.

There are reports that the presence of cyclodextrins in catalytic reaction mixtures could influence catalytic activity and stereoselectivity [26]. Thus we have tried to carry the catalytic reaction in the presence of excess β-cyclodextrin using RhCl(ttp*) as the catalytic precursor, hoping that the added β-cyclodextrin can increase the optical yield. However no significant change in optical yield was observed (entry 4).

Both $RuCl_2$(ttp*) and $RuH(\eta^2\text{-}BH_4)$(ttp*) were found to be catalytically active for hydrogenation of α-acetamidocinnamic acid in methanol in the presence of NEt_3. In the absence of NEt_3, the catalytic activity decreased. Apparently, the NEt_3 helped to generate the active species. In the case of $RuCl_2$(ttp*), RuHCl(ttp*) is likely the active species. The reaction of $RuCl_2(PPh_3)_3$ with H_2 in the presence of NEt_3 to give $RuHCl(PPh_3)_3$ has been reported [27]. In the case of $RuH(BH_4)$(ttp*), RuH_2(ttp*) is likely the active species. The closely related hydride complex RuH_2(ttp) has been suggested as the active species for the hydrogenation of 1-octene using $RuH(BH_4)$(ttp)–NEt_3 as the catalytic precursor [20]. It is noted that addition of NEt_3 to the reaction mixture in MeOH has no appreciable effect on the catalytic activity and steroselectivity (entry 1).

Generally the enantioselectivity induced by the Rh and Ru complexes of ttp* is low compared with ruthenium and rhodium complexes with chiral bidentate ligands such as BINAP, DIOP [28]. The poorer enantioselectivity could be related to the fact that the methyl groups at the stereogenic centers of the ligand backbone is small. Therefore, steric interaction between the substrate and substituents of the ligand is not very effective. In addition, it has been accepted that in the hydrogenation of dehydroamino acids with chiral bidentate phosphine metal complexes, the substrate binds to the metal center in a bidentate manner through the amide carboxyl group and the double bond [28,29]. This kind of tight chelate locates the substrate precisely within the coordination sphere. In our tridentate phosphine metal complex systems, this bidentate interaction of substrate with the metal centers may be limited, as there are not enough coordination sites for such binding without dissociation of one of the phosphine groups.

2.3. Summary

We have prepared the new hydride complexes $Rh(BH_4)$(ttp*) and $RuH(BH_4)$(ttp*). RhCl(ttp*), $RuCl_2$(ttp*), $Rh(BH_4)$(ttp*), and $RuH(BH_4)$(ttp*) were found to be catalytically active for hydrogenation of α-acetamidocinnamic acid. The optical yields, however, are not as good as expected. The etp* complex $RuCl_2$(etp*) was even poor in terms of both catalytic efficiency and stereoselectivity.

3. Experimental

All manipulations were carried out under a dinitrogen atmosphere using standard Schlenk techniques. Solvents were distilled under dinitrogen over sodium-benzophenone (hexane, diethyl ether, THF), or calcium hydride (dichloromethane). Complexes **1** [11], **2** [11], and **3** [3] were prepared according to literature methods. All other reagents were used as purchased from Aldrich or Strem Chemical Co., USA.

Microanalyses were performed by MHW lab (Phoenix, AZ, USA). Optical rotation was measured

Table 1
Catalytic hydrogenation of α-acetamidocinnamic acid by Ru and Rh chiral complexes [a]

Entry	Catalysts	NEt_3	Solvent	Conversion (%)	$[\alpha]_D$ (c, EtOH)	%ee [b]	Configuration
1	RhCl(ttp*)	y	MeOH	100	−19 (2.1)	41	(*R*)
2	RhCl(ttp*)	no	MeOH	100	−20 (4.1)	44	(*R*)
3	RhCl(ttp*)	no	Toluene	16	−23 (14)	50	(*R*)
4	RhCl(ttp*)/CD [c]	y	MeOH	100	−19.4 (5.2)	42	(*R*)
5	$Rh(BH_4)$(ttp*)	no	MeOH	100	−16 (3.8)	35	(*R*)
6	$RuCl_2$(ttp*)	no	MeOH	30	0.98 (1.5)	2	(*S*)
7	$RuCl_2$(ttp*)	y	MeOH	100	22 (3.5)	47	(*S*)
8	$RuH(BH_4)$(ttp*)	no	MeOH	83	3.8 (3.6)	8	(*S*)
9	$RuH(BH_4)$(ttp*)	y	MeOH	100	6.1 (5.7)	13	(*S*)
10	$RuCl_2$(etp*)	y	MeOH	11	0.98 (0.61)	2	(*S*)

[a] Reaction conditions: α-acetamidocinnamic acid, 1.5 mmol; substrate/catalyst = 50; triethylamine if added (indicated by the letter 'y'), 1.8 mmol; H_2, 50 bar; r.t.; 24 h.

[b] The optical yield was determined based on the specific rotation of pure enantiomer *N*-acetyl-(*S*)-phenylalanine ($[\alpha]_D^{26} = +46.0°$ (c 1.0 in EtOH)).

[c] CD = β-cyclodextrin.

with a Perkin–Elmer 241 polarimeter. ^{1}H-, and ^{31}P{^{1}H}-NMR spectra were collected on a Bruker ARX-300 spectrometer. ^{1}H-NMR chemical shifts are relative to TMS and ^{31}P-NMR chemical shifts relative to 85% H_3PO_4.

3.1. $Rh(BH_4)(ttp^*)$ (**4**)

A mixture of RhCl(ttp*) (0.29 g, 0.40 mmol) and $NaBH_4$ (0.15 g, 3.97 mmol) in ethanol (10 ml) was refluxed for 5 min to give an orange precipitate. The mixture was allowed to cool down to room temperature (r.t.). The solid was collected on a filter frit and washed with a small amount of cold ethanol. Yield: 0.23 g, 82%. Anal. Calc. for $C_{38}H_{45}BP_3Rh$: C, 64.43; H, 6.40. Found: C, 64.19; H, 6.23%. ^{1}H-NMR (300.13 MHz, C_6D_6): δ 0.31 (br, BH_4), 0.71 (d, J(HH) = 5.3 Hz, 3H, CH_3), 0.89 (d, J(HH) = 6.3 Hz, 3H, CH_3), 1.33–2.35 (m, 10H, $2CH_2CHCH_2$), 7.04–8.18 (m, 25H, aromatic protons). ^{31}P{^{1}H}-NMR (121.51 MHz, C_6D_6, ABCX (X = Rh) spin system): δ 15.8 (B, PPh_2), 24.2 (C, PPh_2), 17.5 (A, PPh); J(Rh–PPh) = 166.5 Hz, J(Rh–PPh_2) = 131.1 Hz, J(PPh–PPh_2) = 38.5 and 51.6 Hz, J(PPh_2–PPh_2) = 304.5 Hz.

3.2. $Rh(BD_4)(ttp^*)$

The compound was prepared similarly except that $NaBD_4$ was used instead. ^{2}D-NMR (61.25 MHz, C_6H_6): δ 0.3 (br, BD_4).

3.3. $RuH(\eta^2\text{-}BH_4)(ttp^*)$ (**5**)

A mixture of $RuCl_2$(ttp*) (0.15 g, 0.20 mmol) and 0.10 g of $NaBH_4$ (2.6 mmol) in ethanol (10 ml) was refluxed for 5 min to give an orange solution. The solution was allowed to cool down to r.t. and the solvent was removed completely under vacuum. The residue was extracted with 50 ml of benzene. The solvent was removed completely to give a red solid. The solid was collected on a filter frit and dried under vacuum overnight. Yield: 85 mg, 61%. Anal. Calc. for $C_{38}H_{46}BP_3Ru$: C, 64.50; H, 6.55. Found: C, 64.70; H, 6.37%. ^{1}H-NMR (300.13 MHz, C_6D_6): δ −15.95 (dt, J(PH) = 34.2, 21.6 Hz, 1H, RuH), −7.39 (br, 1H, Ru–H–B), −5.62 (br, 1H, Ru–H–B), 0.76 (d, J(HH) = 5.1 Hz, 3H, CH_3), 1.00 (m, 3H, CH_3), 1.42–1.60 (m, 3H, $2CH_2CHCH_2$), 1.92–1.14 (m, 3H, $2CH_2CHCH_2$), 2.25–2.60 (m, 3H, $2CH_2CHCH_2$), 3.39 (q, J(HH) = 6.8 Hz, 1H, $2CH_2CHCH_2$), 5.39 (br, 2H, BH_2), 7.07–8.10 (m, 25H, aromatic protons). ^{31}P{^{1}H}-NMR (121.49 MHz, C_6D_6, ABM spin system): δ 32.9 (A, PPh_2), 38.7 (B, PPh_2), 43.9 (M, PPh); J(PPh–PPh_2) = 39.7 Hz, J(PPh_2–PPh_2) = 266.0 Hz).

3.4. Asymmetric hyrdrogenations

In a typical experiment, a mixture of 0.31 g of α-acetamidocinnamic acid (1.5 mmol), 0.25 ml of triethylamine (1.8 mmol) and 0.03 mmol of catalyst in 10 ml of solvent (as listed in Table 1) was placed in a 25 ml stainless steel autoclave. The loaded autoclave was closed, thoroughly evacuated, and flushed at least three times with hydrogen to ensure a completely oxygen-free environment. The evacuated autoclave was filled with hydrogen and the reaction mixture was stirred under 50 bars of hydrogen at r.t. for 24 h. After completion of the reaction, the solvent was removed under vacuum. The residue was dissolved in aqueous sodium hydroxide, and washed with dichloromethane. The aqueous layer was acidified with hydrochloric acid, and extracted three times with diethyl ether. The extracts were combined, dried with anhydrous $MgSO_4$, and evaporated to give the products. The integrations of *N*-acetyl peaks on the product and starting material were used to determine the percentage of conversion. The optical yield was determined by polarimetry based on the specific rotation of pure enantiomer *N*-acetyl-(*S*)-phenylalanine ($[\alpha]_D^{26} = +46.0°$ (*c* 1.0 in EtOH)) [20b].

Acknowledgements

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