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Journal of Molecular Catalysis A: Chemical 195 (2003) 95–100

JOURNAL OF MOLECULAR CATALYSIS A: CHEMICAL

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Transfer hydrogenation of a variety of ketones catalyzed by rhodium complexes in aqueous solution and their application to asymmetric reduction using chiral Schiff base ligands

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Received 17 April 2002; received in revised form 17 April 2002; accepted 19 October 2002

Abstract

The rhodium complex [Cp*Rh(bpy)Cl]Cl shows efficient catalytic activity in the transfer hydrogenation of a wide variety of ketones in aqueous formic acid solution under mild reaction conditions. In the asymmetric reduction using chiral Schiff base ligands, the asymmetric induction was observed in reduction of dialkyl ketones as well as aryl ketones, among which *ortho*-chloroacetophenone gave the highest ee of 84%.

Keywords: Transfer hydrogenation; Asymmetric reduction; Rhodium complex; Aqueous reaction; Chiral ligand; Ketones

1. Introduction

Catalytic transfer hydrogenation is useful for the reduction of multiple bonds, due to its operational simplicity and the ready availability of the hydrogen donor. There are many successful examples of the asymmetric transfer hydrogenation catalyzed by chiral transition-metal complexes [1–3]. In particular, ruthenium catalysts bearing *N*-(toluenesulfonyl)-1,2-diphenylethylenediamine were remarkably effective for the asymmetric reduction of aryl ketones in 2-propanol or formic acid–triethylamine mixture [4,5]. On the other hand, the application of the transfer hydrogenation to the asymmetric reduction of dialkyl ketones, which are difficult to be converted into the corresponding alcohols [6,7], has not been reported so far except for a few examples [8–10]. Although formic acid as a hydrogen donor in place of 2-propanol has favorable properties [4,5,11], aqueous formic acid has not been extensively used for in the asymmetric reduction [12,13]. From an environmental standpoint, aqueous-phase organometallic catalysis has been recently receiving much attention ([14] and references cited therein). Westerhausen et al. reported that the rhodium complex $[Cp^*Rh(bpy)(H_2O)]Cl_2$ serves as a catalyst precursors for the reduction of benzylacetone in aqueous sodium formate solution at pH 7, even though the catalytic efficiency is low (turnover number <5, 45 h, 30 °C) [15]. The transfer hydrogenation is believed to involve the formation of the water-soluble hydride complex [16,17]. These attractive features, namely the water-soluble catalyst system and the ability to reduce dialkyl ketones, prompted us to evaluate the possibility

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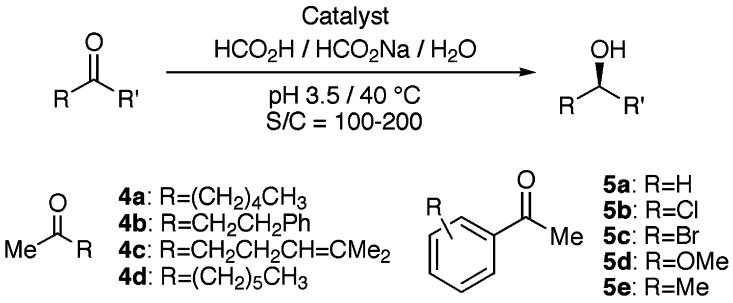
1381-1169/02/$ – see front matter
PII: S1381-1169(02)00576-9



Scheme 1.

of the rhodium-catalyzed transfer hydrogenation [18] and apply it to the asymmetric reduction [19]. Independently, Rhyoo et al. reported the highly enantioselective Ru(II)-catalyzed transfer hydrogenation of aryl ketones with (*S*)-proline amides in aqueous solution [20]. In this paper, we report the rhodium-catalyzed transfer hydrogenation of dialkyl ketones as well as aryl ketones in aqueous solution (Scheme 1).

2. Results and discussion

We first examined the pH dependence of the transfer hydrogenation catalyzed by achiral prototype [Cp*Rh(bpy)Cl]Cl **1**. The reaction of acetone with **1** in degassed aqueous formic acid/sodium formate solution at various pH values proceeded at 40 °C with a substrate/catalyst molar ratio (*S*/*C*) of 200 under an argon atmosphere (Table 1). The reactions, as monitored by GC, showed no significant induction period and rapid formation of 2-propanol. The observed initial turnover frequency (TOF, moles of product per mole of catalyst per hour; initial 1 h period) was strongly dependent on the pH of the solution [21–23]. When the reaction was carried out at pH 3.5, the highest TOF was obtained and GC analysis showed almost complete conversion to 2-propanol after 12 h (entry 2). During the course of the reaction, the color of the solution changed from yellow to blue and evolution of CO_2 and H_2 was observed. After complete consumption of acetone, the pH value of this solution slightly increased and CO_2 and H_2 gases continuously evolved. In the absence of sodium formate, the catalytic activity in 1 N aqueous formic acid dramatically decreased (entry 6) [24]. The reactions using the iridium analogue [Cp*Ir(bpy)Cl]Cl **2** or the ruthenium complex [(C_6Me_6)Ru(bpy)Cl]Cl **3**, both of which had the structure isoelectronic to **1** [25–28], were also performed (entries 7, 8). The rate of the reaction was affected by the central metals and decreased in the order Rh ≥ Ir ≫ Ru. This result and the observation of slight gas evolution in the reaction using **3** were possibly due to the relatively slow rate of formation of the ruthenium hydrid from **3**, which is probably the rate limiting step of the overall reaction [16], in contrast to **1** and **2**.

The scope of this reaction was examined with a variety of ketones (Table 2). Water-soluble acetone and cyclohexanone were reduced with almost complete conversion (entries 1, 2). Interestingly, in the case of slightly water-soluble dialkyl ketones **4a**–**c** (entries

Table 1
Transfer hydrogenation of acetone[a]

Entry	Catalyst	pH	Initial TOF[b] (h^{-1})	Yield[c] (%)
1	**1**	3.0	50	97
2	**1**	3.5	66	99
3	**1**	4.0	66	89
4	**1**	5.0	28	78
5	**1**	6.0	18	86
6	**1**	–[d]	3	5
7	**2**	3.5	41	98
8	**3**	3.5	2	13

[a] The reactions were carried out at 40 °C for 12 h under an argon atmosphere using acetone (2.0 mmol) and catalyst (10 μmol) in degassed aqueous formic acid/sodium formate solution at various pH values.
[b] Moles of product per mole of catalyst/h formed for initial 1 h period.
[c] Determined by GC analysis.
[d] The reaction was carried out in 1 N aqueous formic acid.

Table 2
Transfer hydrogenation of a variety of ketones catalyzed by **1**[a]

Entry	Substrate	Time (h)	Yield[b] (%)
1	Acetone	12	>99
2	Cyclohexanone	4	>99 (70)
3	**4a**	72	94[c]
4	**4b**	72	92[c] (90)
5	**4c**	72	93[c] (74)
6	**5a**	5	99 (94)
7	Ethyl pyruvate	1.5	>99
8	**5a**	5	6[d]

[a] The reactions were carried out at 40 °C using ketone (2.0 mmol) with **1** (10 μmol) in aqueous formic acid/sodium formate solution (20 ml) at pH 3.5 under an argon atmosphere, unless otherwise noted.

[b] Determined by GC analysis. Numbers in parentheses indicate isolated yield.

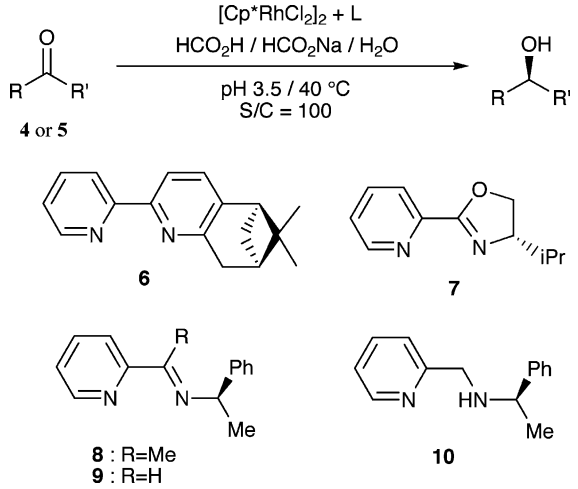
[c] Reaction with $S/C = 100$.

[d] The reactions were carried out at 40 °C using ketone (2.0 mmol) with **1** (10 μmol) and 0.1 N KOH (100 μmol) in 2-propanol (20 ml) under an argon atmosphere.

3–5), the reactions proceeded even under biphasic conditions. High yields (>90%) were obtained with $S/C = 100$ for 72 h. The reductions of acetophenone **5a** and ethyl pyruvate also proceeded with high efficiency (entries 6, 7). Furthermore, we examined the typical transfer hydrogenation of acetophenone catalyzed by **1** in 2-propanol containing KOH. The reaction in 2-propanol proceeded slowly to give 1-phenylethanol in 6% yield after 5 h at 40 °C (entry 8) [29].

We then tried the asymmetric transfer hydrogenation catalyzed by chiral complexes in place of the bipyridine complex **1** (Scheme 2). The chiral catalysts were prepared by mixing the appropriate ligands **6**–**10** with $[Cp^*RhCl_2]_2$ (Rh atom:ligand mole ratio = 1:1.05) in methanol for 12 h at 40 °C, which were used for the catalytic reduction without further purification. The ^{1}H NMR analysis of the complex prepared from pyridyloxazoline ligand **7** confirmed the diastereoselective formation of a single stereoisomer [30]. The ^{1}H NMR analysis of other complexes indicated that a mixture of two complexes was present. Treatment of complexes from **8** or **9** with lithium perchlorate followed by crystallization from CH_3CN–Et_2O gave the major isomers **11** and **12** as the perchlorate salts, respectively.

The reductions of acetophenone **5a** using the prepared chiral rhodium complexes (20 μmol, S/C =



Scheme 2.

100) were conducted for 24 h under the same reaction conditions (Table 3). The catalyst prepared from the chiral bipyridine ligand **6** showed high reactivity without enantioselectivity (entry 1). The Use of the pyridyloxazoline ligand **7** resulted in poor enantioselectivity (entry 2). The best result was obtained when the reduction using the chiral Schiff base ligand **9** was carried out to give (*S*)-1-phenylethanol with 51% ee in 99% yield (entry 4). Interestingly, a similar result was obtained when the isolated major isomer **12** was used (96% yield, 49% ee). When a solution of **12** in CD_3CN was left at room temperature overnight, ^{1}H NMR spectra showed the formation of the original mixture of two isomers in the same ratio (ca. 3:2), implying an equilibrium between the two isomers.

Table 3
Asymmetric transfer hydrogenation of acetophenone **5a** catalyzed by chiral rhodium complexes[a]

Entry	Ligand	Yield[b] (%)	ee[c] (%)
1	**6**	>99 (96)	Rac.
2	**7**	28	12
3	**8**	60 (54)	34
4	**9**	>99 (95)	51
5	**10**	>99 (96)	50

[a] The reactions were carried out at 40 °C for 24 h.

[b] Determined by GC analysis. Numbers in parentheses indicate isolated yield.

[c] Determined by HPLC analysis using DAICEL CHIRALCEL OB. The absolute configuration of the all products was determined to be *S* from its optical rotation except for entry 1.

However, the actual structure of the active catalyst was not clear. Noyori and co-workers suggested that an N–H moiety of the chiral ligand played an important role in the asymmetric transfer hydrogenation using their diamine-based ruthenium catalysts [5,31]. The catalyst from **10** having an N–H moiety showed a reactivity and stereoselectivity similar to those of the catalyst from **9** (entry 5).

A variety of ketones were also examined using the catalyst prepared from **9** (Table 4). In the case of acetophenone derivatives, the enantioselectivity was affected by the position and electronic properties of the ring substituent. The *ortho*-substituted acetophenones gave higher enantioselectivity than the *meta*- and *para*-substituted ones. The best result was obtained in the reduction of *ortho*-chloroacetophenone *ortho*-**5b** (>99% yield, 84% ee). Furthermore, the asymmetric reductions of dialkyl ketones were also carried out. The alkyl methyl ketones **4b–d** were transformed to the corresponding secondary alcohols with ee values ranging from 26% to 27% (entries 10–12). When 2,2-dimethylcyclohexanone was employed as a substrate, somewhat higher enantioselectivity was observed (entry 13). Although there is still great room for improvement in enantioselectivity, this system would provide good possibilities for the asymmetric transfer hydrogenation of a variety of ketones in aqueous media.

Table 4
Asymmetric transfer hydrogenation of ketones catalyzed by rhodium complex bearing chiral Schiff base ligand **9**[a]

Entry	Substrate	Yield[b] (%)	ee[c] (%)
1	*ortho*-**5b**	>99 (97)	84
2	*ortho*-**5c**	51 (48)	72[d]
3	*ortho*-**5d**	>99 (99)	68
4	*meta*-**5b**	>99 (99)	67[d]
5	*meta*-**5d**	98 (95)	62
6	*para*-**5b**	>99 (99)	65
7	*para*-**5c**	95 (91)	62[d]
8	*para*-**5e**	87 (86)	55
9	Propiophenone	81 (77)	63
10	**4b**	35 (33)	26[e]
11	**4c**	38 (24)	26[d]
12	**4d**	47 (35)	27[d]
13	2,2-Dimethylcyclohexanone	48 (38)	31[d]

[a] The reactions were carried out at 40 °C for 24 h.
[b] Determined by GC analysis. Numbers in parentheses indicate isolated yield.
[c] Determined by HPLC analysis using DAICEL CHIRALCEL OB unless otherwise specified. The absolute configuration of the all products was determined to be *S* from its optical rotation.
[d] Determined by optical rotation (see Section 4).
[e] Determined by HPLC analysis using DAICEL CHIRALCEL OD-R.

3. Conclusion

We have demonstrated the rhodium-catalyzed transfer hydrogenation of a wide variety of ketones in aqueous solution. The catalytic performance of the achiral transfer hydrogenation was satisfactory even under biphasic conditions. These results suggest that water soluble rhodium hydrides are generated in aqueous solution and the hydride transfer to ketones proceeds smoothly. In the asymmetric reduction using the chiral Schiff base ligands, the asymmetric induction was observed in the reduction of dialkyl ketones as well as aryl ketones. Further efforts are directed toward the design and development of catalysts for efficient asymmetric reduction and characterization of the catalytically active species.

4. Experimental

4.1. General

The NMR spectra were recorded on a Varian UNITY INOVA400 spectrometer (400 MHz) with Me_4Si as an internal standard. Optical rotation was measured with a Jasco DIP-1000 polarimeter at ambient temperature. The HPLC analysis were carried out on a CHIRALCEL OB or OD-R column (Daicel Chemical Industry). Elemental analysis were performed by using an Eager 200 instrument. $[Cp^*RhCl_2]_2$ and $[Cp^*IrCl_2]_2$ were purchased from Aldrich Chemical Co. $[C_6Me_6RuCl_2]_2$ was purchased from Tokyo Kasei Kogyo Co. Ltd. The gas samples were analyzed on a GL Sciences GC-390 gas chromatograph with an active carbon column. The pH of aqueous solution was adjusted by addition of formic acid (99%) to 2 M aqueous sodium formate.

4.2. Synthesis of chiral rhodium complexes *11* and *12*

A mixture of Schiff base ligand **8** or **9** (0.42 mmol) and $[Cp^*RhCl_2]_2$ (124 mg, 0.2 mmol) in 20 ml of ab-

solute methanol was stirred at 40 °C for 12 h. $LiClO_4$ (0.24 mmol) was then added and the resulting mixture was stirred for 5 h to afford the mixture of two complexes in a ratio of ca. 3:2. The resultant precipitate was recrystallized from acetonitrile-ethyl ether to give the major isomer **11** and **12**, respectively.

11: mp 258.6–259.2 °C (dec). ^{1}H NMR (CD_3CN) δ: 8.86 (br d, J = 5.5 Hz, 1 H, 6-PyH), 8.18 (td, J = 7.6, 1.4 Hz, 1 H, 4-PyH), 8.02 (d, J = 7.5 Hz, 1 H, 3-PyH), 7.84 (ddd, J = 7.6, 5.5, 1.3 Hz, 1 H, 5-PyH), 7.50–7.31 (m, 3 H, Ph), 5.84 (q, J = 7.1 Hz, 1 H, CMe*H*), 2.27 (s, 3 H, N=C*Me*), 2.04 (d, J = 7.1 Hz, 3 H, CH*Me*), 1.63 (s, 15 H, C_5Me_5). Anal. Calcd. for $C_{25}H_{31}Cl_2N_2O_4Rh$: C, 50.27; H, 5.23; N, 4.69%. Found: C, 50.47; H, 5.19; N, 4.70%.

12: mp 212.4–213.0 °C (dec). ^{1}H NMR (CD_3CN) δ: 8.84 (d, J = 5.5 Hz, 1 H, 6-PyH), 8.13 (td, J = 7.6, 1.4 Hz, 1 H, 4-PyH), 8.09 (bs, 1 H, N=C*H*), 7.88 (d, J = 7.5 Hz, 1 H, 3-PyH), 7.80 (ddd, J = 7.6, 5.5, 1.5 Hz, 1 H, 5-PyH), 7.56–7.46 (m, 3 H, Ph), 5.63 (q, J = 7.1 Hz, 1 H, CMe*H*), 1.84 (d, J = 7.1 Hz, 3 H, CH*Me*), 1.71 (s, 15 H, C_5Me_5). Anal. Calcd. for $C_{24}H_{29}Cl_2N_2O_4Rh$: C, 49.42; H, 5.01; N, 4.80%. Found: C, 49.29; H, 4.95; N, 4.60%.

When a solution of **12** in $CDCN_3$ was left at room temperature overnight, ^{1}H NMR spectra showed the formation of the original mixture of two isomers in the same ratio (ca. 3:2). The signals due to the minor isomer in the ^{1}H NMR spectra (CD_3CN) appeared at δ: 8.82 (d, J = 5.5 Hz, 1 H, 6-PyH), 8.69 (bs, 1 H, N=C*H*), 8.18 (td, J = 7.7, 1.5 Hz, 1 H, 4-PyH), 8.03 (d, J = 7.7 Hz, 1 H, 3-PyH), 7.81 (ddd, J = 7.7, 5.5, 1.5 Hz, 1 H, 5-PyH), 7.59–7.31 (m, 3 H, Ph), 5.65 (q, J = 7.1 Hz, 1 H, CMe*H*), 1.90 (d, J = 7.1 Hz, 3 H, CH*Me*), 1.68 (s, 15 H, C_5Me_5).

4.3. Representative procedure for transfer hydrogenation of 2′-chloroacetophenone (ortho-**5b**) catalyzed by the chiral rhodium complexes from **9**

A mixture of Schiff base ligand **9** (2.2 mg, 21 μmol) and $[Cp^*RhCl_2]_2$ (6.2 mg, 10 μmol) in 10 ml of absolute methanol was stirred at 40 °C for 12 h. The solvent was removed under reduced pressure to give a pale yellow solid. A mixture of the crude complex and *ortho*-**5b** (309 mg, 2.0 mmol) in degassed aqueous formic acid/sodium formate solution (20 ml, pH 3.5) was stirred vigorously at 40 °C for 24 h under an argon atmosphere. The reaction mixture was extracted with ethyl acetate. The organic layer was dried over $MgSO_4$, and concentrated under reduced pressure. The crude product was purified by flash chromatography on silica gel using 10% ethyl acetate/hexane to afford (*S*)-1-phenylethanol (304 mg, 97%) in 84% ee. $[\alpha]_D^{25.8} = -50.9°$ (c 1.55 in $CHCl_3$) [32] $[\alpha]_D^{25} = -62.7°$ (c 0.894 in $CHCl_3$) >99% ee (*S*).

4.4. Optical rotation data of hydrogenation products

4.4.1. (S)-1-(2-Bromophenyl)ethanol from ortho-**5c**

$[\alpha]_D^{24} = -39.2°$ (c 1.01 in $CHCl_3$) [32] $[\alpha]_D^{24} = -54.6°$ (c 1.23 in $CHCl_3$) >99% ee (*S*).

4.4.2. (S)-1-(3-Chlorophenyl)ethanol from meta-**5b**

$[\alpha]_D^{25} = -29.0°$ (c 1.00 in $CHCl_3$) [32] $[\alpha]_D^{25} = -43.5°$ (c 1.08 in $CHCl_3$) >99% ee (*S*).

4.4.3. (S)-1-(4-Bromophenyl)ethanol from para-**5c**

$[\alpha]_D^{22.4} = -23.5°$ (c 0.99 in $CHCl_3$) [32] $[\alpha]_D^{24} = -37.9°$ (c 1.13 in $CHCl_3$) >99% ee (*S*).

4.4.4. (S)-6-Methyl-5-hepten-2-ol (Sulcatol) from **4c**

$[\alpha]_D^{27.5} = +4.1°$ (c 0.98 in EtOH) [33] $[\alpha]_D^{24.5} = -16.0°$ (c 1.1 in EtOH) (*R*).

4.4.5. (S)-2-Octanol from **4d**

$[\alpha]_D^{22} = +2.7°$ (c 0.93 in EtOH) [34] $[\alpha]_D^{20} = +10.0°$ (c 1 in EtOH) >99% ee (*S*).

4.4.6. (S)-2,2-Dimethylcyclohaxanol

Determined by optical rotation of its 3,5-dinitrobenzoate derivative, which was prepared according to the reported procedure [35]. $[\alpha]_D^{22} = -13.0°$ (c 0.72 in $CHCl_3$) [35] $[\alpha]_D^{20} = -42.5°$ (c 1.73 in $CHCl_3$) 99% ee (*S*).

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