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Highly enantioselective hydrogenation of enamides catalyzed by rhodium-monodentate phosphoramidite complex derived from H_8-BINOL

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Abstract—A series of monodentate phosphoramidite ligands derived from H_8-BINOL have been synthesized. The ligands are used in the Rh-catalyzed asymmetric hydrogenation of enamides to give the acetamides in up to 99% ee.

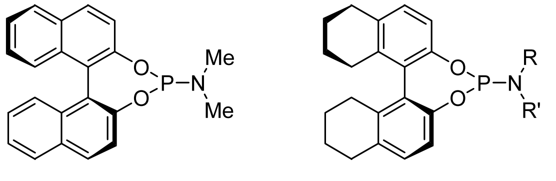
1. Introduction

The asymmetric catalytic hydrogenation of enamides provides a general and convenient access to chiral amides or amine derivatives. Kagan et al. first recognized the feasibility of this reaction in 1975 and achieved up to 90% ee for some substrates by employing a Rh(I)–DIOP system.[1] In 1996, Burk et al. reported an important advance in the enantioselective hydrogenation of arylenamides with Rh catalysts containing Duphos and BPE ligands.[2] Later, a number of structurally diverse but effective ligands, such as H_8-BDPAB,[3] Binaphane and the likes,[4] Pennphos,[5] 1,4-diphosphines,[6] modified Duphos,[7] *P*-stereogenic ligands,[8] and *ortho*-substituted BIPHEP,[9] for this reaction were also reported.

Our interest in catalytic asymmetric synthesis is focused on the development of effective chiral ligands, which can be easily prepared so that the related chemistry can be widely used. That BINOL can be expediently synthesized on a large scale and that H_8-BINOL can be readily obtained from BINOL via hydrogenation rendered H_8-BINOL an interesting starting material for the preparations of chiral ligands.[10] Spurred by a rekindled interest in monodentate ligands[11] and the effectiveness of the octahydrobinaphthyl backbone in asymmetric hydrogenations,[10] we initiated a study of the synthesis of a number of monodentate phosphoramidites derived from H_8-BINOL (Fig. 1) and their applications in the Rh-catalyzed asymmetric hydrogenation of enamides. Herein we report the results of this study in comparison with those obtained using the original Monophos ligands.[11b,j–l]

2. Results and discussions

In our preliminary study, *N*-(1-phenylethenyl) acetamide **3** was used as a model substrate for testing the



1 (*S*)-Monophos

2a: R = R' = Me
2b: R = R' = Et
2c: R = (*R*)-CH(Me)Ph, R' = H
2d: R = (*R*)-CH(Me)Ph, R' = Me
2e: R + R' = -$(CH_2)_4$-

Figure 1.

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catalytic performance of the catalysts containing chiral ligands. Common factors governing the enantioselectivities of the reaction were examined. The results shown in Table 1 indicated that the enantioselectivity of the catalysts at 0°C was sensitive to the choice of solvent when **2a** was chosen as the chiral ligand (THF: 91.2% ee, dichloromethane: 90.4% ee, acetone: 84.5% ee, methanol: 40.0% ee). On the other hand, hydrogen pressure had relatively little effect on the enantioselectivity (entries 4, 9–10). The reaction temperature was also an important factor. Significant increase in enantioselectivity was observed at lower reaction temperatures and the ee reached a plateau at −10°C. Further decrease of temperature did not bring about further enhancement in enantioselectivity. Under the optimal conditions, 96.2% ee was obtained (entry 11). These results compared favorably with the results of Rh-**1** catalyzed asymmetric hydrogenation of enamides, which gave 50–96% ee.[11g] Other listed ligands were also investigated. The results demonstrate that there are no substantial differences between ligands **2b** and **2c** (entries 5 and 6) with **2a**. However, when ligand **2d** or **2e** was used in the reaction, the enantioselectivity decreased dramatically to 50.1% and 62.5% ee (entries 7 and 8), respectively. Considering the relatively simple preparation of ligand **2a**, we selected **2a** for further study of this reaction.

Various enamides were used in the study of the enantioselectivity of H_8-Monophos **2a** under the optimal conditions (Table 2). Ee values were found to be consistently 3% higher in most cases in comparison with those obtained with Rh-**1** as catalyst, in accord with a recent finding.[11f] This is probably attributable to the larger torsional dihedral angle in **2a**. However the ee's decreased abruptly for substrates **10**, **11** (entries 8–10) and **12** (Scheme 1) due possibly to the larger steric bulk of both the octahydrobinaphthyl backbone of **2a** and the substrates. The highest ee (99%) was realised for **6** which contained a p-CF_3 group on the aromatic ring (entry 4).

In conclusion, H_8-Monophos **2a** was found to be an effective ligand for the Rh-catalyzed asymmetric hydrogenation of enamides.

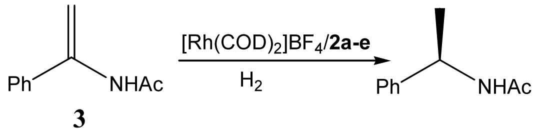
3. Experimental

All experiments were carried out under a nitrogen atmosphere using standard Schlenk techniques or in a glovebox. Flash column chromatography was performed on basic alumina (Merck Kieselgel 60, particle size 0.032–0.063 mm). Tetrahydrofuran (THF) was distilled from sodium benzophenone ketyl prior to use. Methanol was distilled from magnesium/iodine. Dichloromethane and acetone was distilled from CaH_2 and P_2O_5, respectively. ^1H, ^{31}P and ^{13}C NMR were recorded on a Varian AS 500 at room temperature using $CDCl_3$ as solvent. Enantiomeric excesses were determined by gas chromatographic analyses conducted on a HP 4890A or HP 5890 series II system.

3.1. General procedure for the synthesis of 2b–e

Phosphorus trichloride (0.3 mL, 3.4 mmol) and triethylamine (1.2 mL, 8.6 mmol) were transferred via a syringe into a 100 mL round-bottomed flask containing H_8-BINOL (0.88 g, 3.0 mmol) in toluene (30 mL) at 0°C under a nitrogen atmosphere. After the mixture was kept stirring for 6 h, the volatiles were removed under vacuum. A solution of the amine (3.9 mmol) and triethylamine (0.6 mL, 4.3 mmol) in toluene (30 mL) was added to the above flask and the mixture was allowed to react overnight. The resulting mixture was purified through a Al_2O_3-packed column with toluene

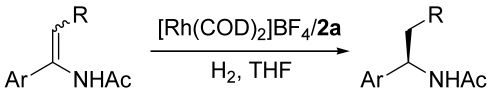
Table 1. Asymmetric hydrogenation of enamide **3** catalyzed by Rh-monodentate phosphoramidite ligands derived from (S)-H_8-BINOL



Entry	Ligands	Solvent	H_2 (psi)	T (°C)	Time (h)	Ee (%)[a]
1	**2a**	CH_2Cl_2	300	0	3	90.4
2	**2a**	MeOH	300	0	3	40.0
3	**2a**	Acetone	300	0	3	84.5
4	**2a**	THF	300	0	3	91.2
5	**2b**	THF	300	0	3	91.0
6	**2c**	THF	300	0	3	91.1
7	**2d**	THF	300	0	3	50.1
8	**2e**	THF	300	0	3	62.5
9	**2a**	THF	150	0	3	87.1
10	**2a**	THF	500	0	3	89.1
11	**2a**	**THF**	**300**	**−10**	**6**	**96.2**
12	**2a**	THF	300	−20	8	96.1

[a] The ee's were determined by chiral GC analysis using a Chrompack chiral fused silica gel 50 m×0.25 mm chirasil-L-VAL column; all products were found with a R configuration based on the comparison of the experimental results with published data.[11k]

Table 2. Asymmetric hydrogenation of enamides catalysed by Rh-(*S*)-H_8-Monophos[a]

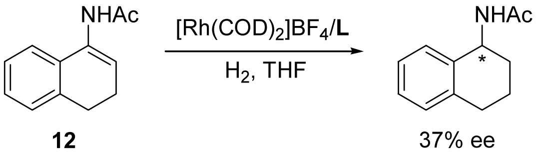


3: Ar = C_6H_5, R = H
4: Ar = *p*-CH_3-C_6H_4, R = H
5: Ar = *p*-CH_3O-C_6H_4, R = H
6: Ar = *p*-CF_3-C_6H_4, R = H
7: Ar = *p*-Br-C_6H_5, R = H
8: Ar = *m*-$CH_3OC_6H_5$, R = H
9: Ar = *m*-$CH_3C_6H_5$, R = H
10: Ar = 1-naphthyl, R = H
11: Ar = C_6H_5, R = CH_3 (E/Z = 3/2)

Entry	Substrate	S/C (mol/mol)	*T* (°C)	Time (h)	Conversion (%)	Ee (%)[b]
1	**3**	100	−10	8	>99	96
2	**4**	100	−10	8	>99	95
3	**5**	100	−10	8	>99	92
4	**6**	**100**	**−10**	**6**	**>99**	**99**
5	**7**	100	−10	8	>99	98
6	**8**	100	−10	8	>99	98
7	**9**	100	−10	8	>99	95
8	**10**	50	0	24	>99	15
9	**10**	50	−10	18	20	23
10	**11**	100	−10	18	>99	42

[a] Hydrogen pressure was 300 psi in all reactions.

[b] The ee's were determined by chiral GC analysis using a Chrompack chiral fused silica gel 50 m×0.25 mm chirasil-L-VAL column; all products were found with a *R* configuration based on the comparison of the experimental results with published data.[11i]



Scheme 1.

as an eluent. The eluates were concentrated in vacuum to afford the phosphoramidite as solids.

3.1.1. *N*,*N*-Diethyl-(*S*)-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-bi-2-naphthyl phosphoramidite 2b. The general procedure was followed with diethylamine as starting material. Yield: 81.2%. Analytical data: $[\alpha]_D^{20}=+219$ (*c* 0.55, CH_2Cl_2). ^1H NMR (500 MHz, $CDCl_3$): δ 6.81–7.06 (m, 4H), 2.93–2.97 (m, 2H), 2.77–2.91 (m, 6H), 2.61–2.75 (m, 2H), 2.22–2.33 (m, 2H), 1.73–1.80, m, 6H), 1.52–1.59 (m, 2H), 1.00–1.03 (t, *J*=7 Hz, 6H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 148.91, 148.36, 148.33, 137.90, 137.88, 137.39, 133.87, 132.78, 128.10–129.36 (m), 128.09, 118.41, 118.39, 118.36, 37.94–38.11 (m), 29.09–29.21(m), 27.69–27.80 (m), 22.56–22.84 (m), 14.67–14.74 (m). ^{31}P NMR (200 MHz, $CDCl_3$): δ 144.8. HRMS calcd for mass: 396 (M^++1, 100%). HRMS calcd for C_{24} $H_{30}NO_2P$: 395.2014. Found: 395.2011.

3.1.2. (*R*)-*N*-1-Phenylethyl-(*S*)-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-bi-2-naphthyl phosphoramidite 2c. The general procedure was followed with (*R*)-phenylethylamine as starting material. Yield: 71.6%. Analytical data: $[\alpha]_D^{20}=+85$ (*c* 0.55, $CHCl_3$). ^1H NMR (500 MHz, $CDCl_3$): δ 6.20–7.41 (m, 9H), 4.38–4.40 (m, 1H), 2.61–2.80 (m, 4H), 2.50–2.60 (m, 2H), 2.18–2.22 (m, 2H), 1.60–1.71 (m, 8H), 1.42–1.51 (d, *J*=7.5 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 148.51, 146.07, 145.25, 137.94, 137.53, 134.13, 133.30, 129.37, 129.28, 128.85, 128.53, 127.07, 126.18, 119.12, 118.52, 51.15, 50.93, 29.19, 29.16, 27.79, 27.71, 25.44, 25.38, 22.77, 22.72, 22.59, 22.52. ^{31}P NMR (200 MHz, $CDCl_3$): δ 145.8. Anal. calcd for $C_{28}H_{30}O_2NP$ (%): C, 75.83; H, 6.82; N, 3.16; P, 6.98. Found: C, 75.58; H, 6.86; N, 3.01; P, 6.81.

3.1.3. (*R*)-*N*-Methyl-*N*-1-phenylethyl-(*S*)-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-bi-2-naphthyl phosphoramidite 2d. The general procedure was followed with (*R*)-*N*-methylphenethylamine as starting material. Yield: 76.9%. Analytical data: $[\alpha]_D^{20}=+92$ (*c* 0.60, CH_2Cl_2). ^1H NMR (500 MHz, $CDCl_3$): δ 7.45–7.47 (m, 2H), 7.37–7.41 (m, 2H), 7.27–7.30 (m, 1H), 7.03–7.08 (m 2H), 6.82–6.84 (d, *J*=7.5 Hz, 1H), 6.50–6.51 (d, *J*=8.0 Hz, 1H), 4.74–4.78 (m, 1H), 2.59–2.83 (m, 6H), 2.21–2.30 (m, 2H), 1.97–1.98 (d, *J*=4.5 Hz, 3H), 1.72–1.78 (m, 6H), 1.54–1.55 (d, *J*=7.5 Hz, 3H), 1.50–1.53 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 148.81, 148.55, 148.52, 142.09, 142.07, 138.02, 138.00, 137.30, 134.07, 132.81, 129.31–129.41 (m), 129.18, 128.10–128.26 (m), 127.67–127.76 (m), 127.05–127.15 (m), 118.76 (m), 118.36–118.42 (m), 55.21–55.66 (dd), 29.00–29.33 (m), 27.73–27.84 (m), 26.61–26.63 (m), 22.60–22.83 (m), 18.00–18.04 (m). ^{31}P NMR (200 MHz, $CDCl_3$): δ 142.9. HRMS calcd for mass: 458 (M^++1, 100%). HRMS calcd for $C_{29}H_{32}NO_2P$: 457.2171. Found: 457.2168.

3.1.4. *N*-(1-Pyrrolidinyl)-(*S*)-5,5′,6,6′,7,7′,8,8′-octahydro-1,1′-bi-2-naphthyl phosphoramidite 2e. The general procedure was followed with pyrrolidine as starting material. Yield: 77.3%. Analytical data: $[\alpha]_D^{20}=+238$ (*c* 0.43, CH_2Cl_2). ^1H NMR (500 MHz, $CDCl_3$): δ 7.02–

7.10 (d, $J=40.5$ Hz, 2H), 6.95–6.97 (d, $J=10.0$ Hz, 2H), 6.81–6.83 (d, $J=8.5$ Hz, 1H), 3.04–3.59 (m, 2H), 2.72–2.95 (m, 6H), 2.61–2.68 (m, 2H), 2.19–2.30 (m, 2H), 1.43–1.91 (m, 12H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 148.95, 148.47, 148.44, 137.73, 137.31, 133.67, 132.75, 129.11–129.27 (m), 128.85–128.91 (m), 128.32, 128.31, 118.37 (m), 45.17–45.29 (m), 28.94–29.18 (m), 27.52–27.70 (m), 25.71–25.74 (m), 22.49–22.76 (m). ^{31}P NMR (200 MHz, $CDCl_3$): δ 144.4. HRMS calcd for $C_{24}H_{28}NO_2P$: 393.1858. Found: 393.1851.

3.2. Typical procedure for the hydrogenation of enamides

A stock catalyst solution was prepared in situ by mixing $[Rh(COD)_2]BF_4$ (2.0 mg, 0.005 mmol) and the phosphoramidite **2a**–**e** (0.01 mmol) in THF (1.0 mL). The mixture was stirred for 10 min. An appropriate amount of this catalyst solution (ca. 0.2 mL) was transferred into a 50 mL stainless steel autoclave with a glass liner containing a predetermined amount of the enamide substrate (1.0 mmol) and a magnetic stirring bar. Hydrogen gas was charged into the reactor at the required temperature and pressure and the solution was stirred for a specified period of time. After the reaction was complete, the hydrogen gas was released and the ee value of the product was measured directly from the reaction mixture by GC without further purification.

The enantiomeric excesses of the products were determined by chiral GC analysis using a Chrompack chiral fused silical gel 50 m×0.25 mm chirasil-L-VAL column.

***N*-(1-Phenylethyl)acetamide**
Oven temperature: 150°C, $t_R=30.7$ min (*R*), 31.8 min (*S*).

***N*-[1-(4-Methylphenyl)ethyl]acetamide**
Oven temperature: 165°C, $t_R=30.7$ min (*R*), 31.7 min (*S*).

***N*-[1-(4-Methoxyphenyl)ethyl]acetamide**
Oven temperature: 170°C, $t_R=28.4$ min (*R*), 29.3 min (*S*).

***N*-[1-(4-Bromophenyl)ethyl]acetamide**
Oven temperature: 175°C, $t_R=32.2$ min (*R*), 33.5 min (*S*).

***N*-[1-[4-(Trifluoromethyl)phenyl]ethyl]acetamide**
Oven temperature: 155°C, $t_R=32.2$ min (*R*), 33.5 min (*S*).

***N*-[1-(3-Methylphenyl)ethyl]acetamide**
Oven temperature: 170°C, $t_R=28.9$ min (*R*), 29.8 min(*S*).

***N*-[1-(3-Methoxyphenyl)ethyl]acetamide**
Oven temperature: 175°C, $t_R=30.6$ min (*R*), 31.5 min (*S*).

***N*-[1-(1-Naphthalenyl)ethyl]acetamide**
Oven temperature: 165°C, $t_R=41.8$ min (*R*), 43.8 min (*S*).

***N*-(1-Phenylpropyl)acetamide**
Oven temperature: 150°C, $t_R=31.2$ min (*R*), 32.7 min (*S*).

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