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Enantioselective palladium catalyzed allylic substitution using chiral P,N,O Schiff base ligands

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Abstract

Palladium complexes prepared in situ from $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ and a number of chiral Schiff ligands having hard and soft donor atoms were evaluated as catalysts for allylic substitution reaction. Enantioselectivity of up to 92% was observed.

Keywords: Enantioselectivity; Schiff ligand; Allylic

1. Introduction

Chiral Schiff base derivatives have attracted great interest because of their utility as chiral ligands in metal complexes for enantioselective catalysis [1–6]. However, chiral Schiff base ligands having both hard and soft donor atoms are rare and their uses in catalytic reactions are still in their infancy [7–10]. These ligands have been shown to produce unusual coordination environment, to form bifunctional mononuclear or binuclear metal complex catalysts [8,9] and to impart unusual reactivity in catalysis [10–15]. We recently demonstrated efficient enantioselective ruthenium-catalyzed catalytic transfer hydrogenation using a chiral tridentate P,N,O Schiff base ligand [16]. This type of ligand has the advantage that large structural variations are possible and they are very easy to prepare. To increase the scope of this type of ligand, herein, we report the results of catalytic enantioselective allylic substitutions of 1,3-diphenyl-2-propenyl acetate with different nucleophiles by the complexes formed in situ from allylpalladium chloride dimer $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ and chiral P,N,O Schiff bases. Palladium-catalyzed allylic substitution is a very useful process in organic synthesis [17–20]. Substitution of rac-1,3-diphenyl-2-propenyl acetate serves as a model substrate to compare the efficiencies of different ligands. The asymmetric versions of this reaction using *P,P*-chelate, *N,N*-chelate and more recently *P,N*-chelate chiral ligands have been reported [21–35]. Our results here show that highly enantioselective catalysts can be prepared using these very simple chiral P,N,O Schiff base ligands.

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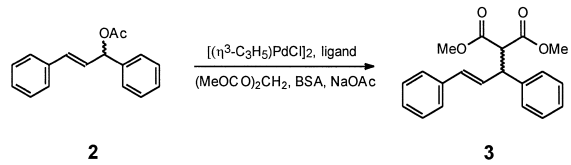
2. Results and discussion

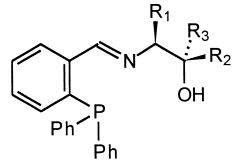
The Schiff base ligands **1a**–**f** used in this study were prepared by treating different readily available optically pure aminoalcohols with 2-diphenylphosphino-benzaldehyde in ethanol under reflux (Scheme 1) [9]. Evaporation of the solvent under vacuum gave the ligands in good yields. These ligands were characterized by IR, ^{1}H, ^{13}C, ^{31}P NMR and MS. In the ^{1}H NMR spectra, the imine proton appears as a doublet at about 8–9 ppm due to coupling to the phosphorus atom. As expected, the ^{13}C NMR spectra shows the CH=N carbon as a doublet at about 159–162 ppm. The ^{31}P NMR spectra show a peak at about δ −9.0 to −10.0 ppm. The IR spectra show a strong band at 3300–3500 cm^{-1} assigned to ν(OH) and a band at 1630–1650 cm^{-1} assigned to ν(C=N).

In the process of developing these ligands for catalytic reactions, we found that the complex generated in situ by reacting **1a** with $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ in CH_2Cl_2 is an effective catalyst for asymmetric allylic substitution. The species shows an UV/VIS absorption band at 320 nm and a ^{31}P NMR peak at 33.8 ppm. Under the standard conditions previously reported by Mino et al. (2 mol% $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ and a mixture of *N,O*-bis(trimethylsilyl)acetamide (BSA) and catalytic amounts of NaOAc as base) [27], racemic 1,3-diphenyl-2-propenyl acetate **2** reacted with dimethyl malonate to give optically active substitution product **3** (Scheme 2). Table 1 summarizes the results with a variety of ligands and conditions. Several observations are apparent from these results. The reaction conditions affected the results quite significantly. We found that CH_2Cl_2 gave the best result in terms of e.e. and yield (Table 1, entries 1–5). Changing the additive from NaOAc to KOAc dropped the yield and e.e. slightly (Table 1, entry 6). Good to excellent yields of products (78–99%) were obtained in a few hours with most ligands except ligand **1g** which gave only 52% yield after 40 h (Table 1, entry 15). The best enantioselection of 70% was obtained by **1a** at a Pd/ligand ratio of 2.5 (Table 1, entry 7). Ligands **1b** to **1g** give e.e. in the range of 17 to 66%. Increasing the ligand amounts increased the e.e. (Table 1, entries 7, 12 and 14). Because of the relatively high enantioselectivity of **1a**,

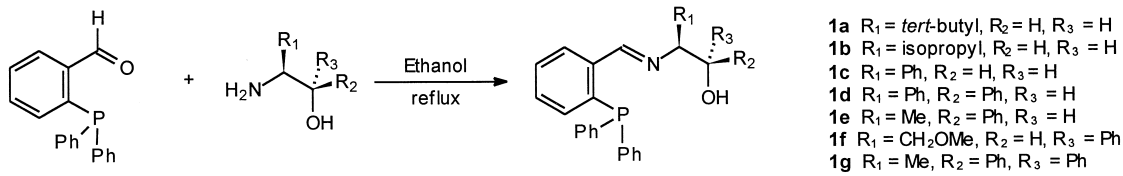


2 **3**

Scheme 2.



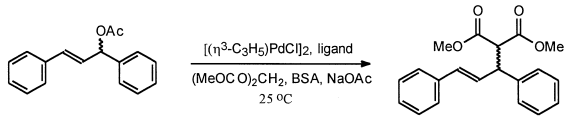
Ligand **1**



Ligand **1**

Scheme 1.

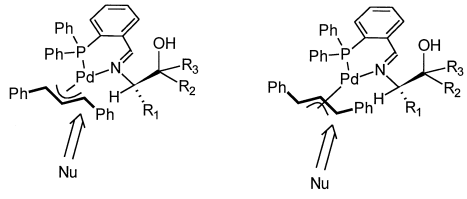
Table 1
Catalytic asymmetric allylic substitution with chiral palladium catalyst



Entry	Ligand L	Conditions	Solvent	Time (h)[a]	Percentage yield[b]	Percentage e.e.[c] (conf.)[d]
1	**1a**	5 mol% L	CH_2Cl_2	4	99	60(*S*)
2	**1a**	5 mol% L	THF	5	93	38(*S*)
3	**1a**	5 mol% L	CH_3CN	3	84	48(*S*)
4	**1a**	5 mol% L	Benzene	20	72	51(*S*)
5	**1a**	5 mol% L	Toluene	21	77	54(*S*)
6	**1a**	5 mol% L	CH_2Cl_2	4	75	55(*S*)
7	**1a**	10 mol% L	CH_2Cl_2	4	79	70(*S*)
8	**1b**	5 mol% L	CH_2Cl_2	2.5	99	42(*S*)
9	**1c**	5 mol% L	CH_2Cl_2	1.5	78	17(*S*)
10	**1d**	5 mol% L	CH_2Cl_2	1.5	91	19(*R*)
11	**1e**	5 mol% L	CH_2Cl_2	1	85	41(*R*)
12	**1e**	10 mol% L	CH_2Cl_2	10	64	66(*R*)
13	**1f**	5 mol% L	CH_2Cl_2	4	89	36(*R*)
14	**1f**	10 mol% L	CH_2Cl_2	7	81	38(*R*)
15	**1g**	5 mol% L	CH_2Cl_2	40	52	25(*R*)

[a] Reactions were monitored by TLC.
[b] Isolated yield after chromatography.
[c] Determined by HPLC analysis using a chiral column (Chiralcel-OD).
[d] Determined by comparison with literature data [36].

the effect of ligand concentration on the enantioselectivity was then studied using it. The result was illustrated in Fig. 1. The e.e. increased gradually as the ligand to palladium ratio increased from 1:1.25 to 1:6. This result suggested that the palladium–ligand complex is in equilibrium with unbound palladium species at a **L**/Pd ratio of 1.25. We believe that increasing the ligand concentration increases the bound and unbound ratio of palladium which, in turn, affects the e.e.



The absolute configuration of **3** was determined by HPLC and confirmed by comparison with literature data [36]. Based on previous results obtained by other groups [37–40], we believe that the possible active intermediates are **4** and **5**, in which the P,N,O Schiff base function as a bidentate P,N ligand. The relative ratio of **4** and **5** formed by different ligands and the

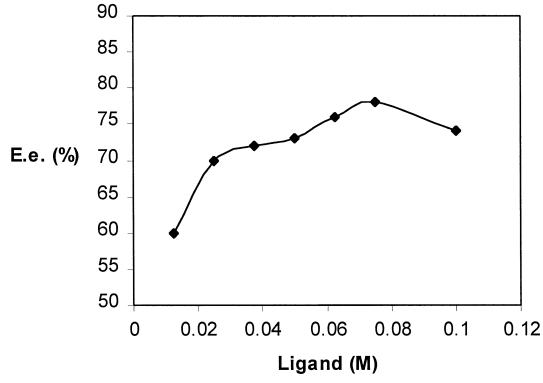
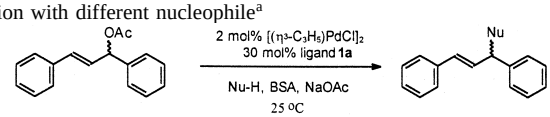
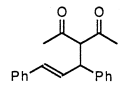
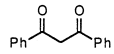
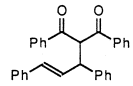
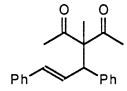
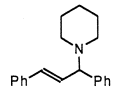
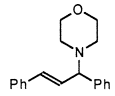
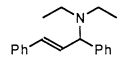
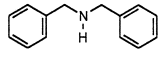
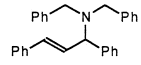


Fig. 1. Dependence of e.e. allylic substitution product **3** on the concentration of ligand **1a**. Reaction condition: $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ (0.01 mmol), chiral ligand **1a** (0.025–0.2 mmol), racemic 1,3-diphenyl-2-propenyl acetate **2** (0.5 mmol), dimethyl malonate (1.5 mmol), BSA (1.5 mmol), NaOAc (0.012 mmol), and CH_2Cl_2 (2 ml).

rate they reacted with nucleophile then determined the configuration of the product in the reaction. According to the model proposed by Spritz et al. [38], which suggests that trans influence directs nucleophilic addition to the allyl terminus trans to the phosphorus atom, the e.e. would therefore be determined by the relative amounts of **4** and **5**, and this would in turn depends on the ligand used. Ligand **1a** to **1c** favored **4** whereas **1d** to **1g** because of the increase in bulk of the alcohol carbon favored **5**. With ligand **1a** being the best ligand, we have investigated substitution with different nucleophiles under the optimum conditions. The results are summarized in Table 2. The carbon nucleophiles give e.e. from 33 to 63% while the *N,N*-dialkylamine nucleophiles give slightly faster reaction and better enantioselectivities (89–92%). However, *N*-monoalkylamine nucleophile, like benzyl amine, does not give any e.e. under the reaction condition. The absolute configuration of the *N,N*-dibenzylamine-de-

Table 2
Catalytic asymmetric allylic substitution with different nucleophile[a]



Entry	Nucleophile (Nu-H)	Product	Time (h)[b]	% yield[c]	% ee[d]
1			7	64	63
2			7	36	33
3			8	49	62
4			4	54	89
5			4	65	92
6			4	32	89
7			6	67	90(R)[e]

[a] Reactions with nitrogen nucleophile were run with no BSA.
[b] Reactions were monitored by TLC.
[c] Isolated yield after chromatography.
[d] Determined by HPLC analysis using a chiral column (Chiralcel-OD and Chiralpak AD).
[e] Absolute configuration was determined by comparing the elution order of enantiomers in HPLC of *N,N*-dibenzylamine-derived sample prepared from benzylation of *N*-benzyl-(1,3-diphenyl-2-propenyl)amine having a known configuration [41,42]. The absolute configuration of the other products has not been determined.

rived product was determined to be R by comparing the elution order of enantiomers in HPLC of sample prepared from benzylation of *N*-benzyl-(1,3-diphenyl-2-propenyl)amine of known configuration [41,42]. This result indicates that the nitrogen nucleophile approaches the intermediate the same way as carbon nucleophile.

In summary, palladium catalyst based on a very simple and easily prepared chiral P,N,O ligand such as **1a** can be highly enantioselective in allylic substitution. Further studies aiming at the synthesis and application of new chiral Schiff base ligands are underway.

3. Experimental

All air-sensitive manipulations were carried out under an atmosphere of dry dinitrogen. Vacuum-line and syringe techniques were utilized throughout where appropriate. 2-Diphenylphosphinobenzaldehyde, $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ and chiral aminoalcohols for ligands **1a**–**1g**, were purchased from Aldrich. All other chemicals were of reagent grade quality and were used as commercially obtained. Solvents were purified following the standard procedures and stored under nitrogen [43]. IR spectra in the range 500–4000 cm^{-1} as Nujol matrix or KBr plates were recorded on a Perkin-Elmer Model FTIR-1600 spectrometer. UV spectra were recorded on a Perkin-Elmer Lambda 19 UV spectrometer. ^1H, ^{13}C and ^{31}P NMR spectra were recorded on Jeol 270 MHz FT-NMR or Varian 300 MHz Mercury instruments. Positive ion FAB mass spectra as 3-nitrobenzylalcohol matrix were recorded on a Finnagin MAT 95 spectrometer. Elemental analyses were performed on a Vario EL elemental analyser.

*3.1. General procedure for the synthesis of P,N,O, ligand **1a–f***

Chiral aminoalcohol (0.8 mmol) in absolute ethanol (2 ml) was added to a solution of 2-diphenylphosphinobenzaldehyde (0.233 g, 0.8 mmol) in absolute ethanol (3 ml). The solution was heated to reflux for 2.5 h under N_2. The solvent was removed by rotatory evaporator to produce a yellow liquid. Diethyl ether (3 ml) was then added and rotatory evaporated to about 1 ml and the product was dried in vacuum. All the other ligands were synthesized and characterized in a similar manner. The yellow solid obtained was characterized by IR, ^1H, ^{13}C, ^{31}P NMR, MS and CHN elemental analysis.

Ligand **1a**. (*S*)-*tert*-Leucinol was used. Yield: 0.196 g (63%). IR (Nujol): 3381.3s, 1640.9s cm^{-1}. ^1H NMR ($CDCl_3$): δ 0.68 (s, 9H), δ 2.00 (s, 1H), 2.80 (m, 1H), 3.52 (m, 2H), 6.80–7.74 (m, 14H), 8.53 (d, $J_{PH} = 3.9$ Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 160.6 (d, C=N); ^{31}P NMR ($CDCl_3$) δ −10.02 (s); Positive ion FAB mass spectrum: m/z 390 ($M^+ + H$). Anal. Calcd. for $C_{27}H_{24}PNO \cdot (1/2)H_2O$: C, 77.50; H, 6.02; N, 3.35. Found: C, 77.20; H, 5.90; N, 3.13.

Ligand **1b**. (*S*)-2-Amino-3-methyl-1-butanol was used. Yield: 0.225 g (75%). IR (Nujol): 3402.9m, 1640.9m cm^{-1}. ^1H NMR ($CDCl_3$): δ 0.50 (d, $J = 6.6$ Hz, 3H), δ 0.73 (d, $J = 6.9$ Hz, 3H), 1.59 (m, 1H), 1.87 (s, 1H), 2.70 (m, 1H), 3.50 (m, 2H), 6.79–7.73 (m, 14H), 8.53 (d, $J_{PH} = 3.9$ Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 160.0 (d, C=N); ^{31}P NMR ($CDCl_3$) δ −9.74 (s); Positive ion FAB mass spectrum: m/z 376 ($M^+ + H$). Anal. Calcd. for $C_{24}H_{26}PNO$: C, 76.78; H, 6.98; N, 3.73. Found: C, 76.40; H, 6.91; N, 3.35.

Ligand **1c**. (*S*)-2-Phenylglycinol was used. Yield: 0.262 g (80%). IR (KBr): 3381.4s, 1639.4s cm^{-1}. ^1H NMR ($CDCl_3$): δ 3.03 (s, 1H), δ 3.83 (m, 2H), 4.56 (dd, $J = 6.8$, 6.2 Hz, 1H), 6.88–7.93 (m, 19H), 8.82 (d, $J_{PH} = 3.6$ Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 161.1 (d, C=N); ^{31}P NMR ($CDCl_3$) δ −9.94 (s); Positive ion FAB mass spectrum: m/z 410 ($M^+ + H$). Anal. Calcd. for $C_{25}H_{28}PNO$: C, 77.10; H, 7.25; N, 3.60. Found: C, 76.78; H, 7.50; N, 3.65.

Ligand **1d**. (1*S*,2*R*)-2-Amino-1,2-diphenylethanol was used. Yield: 0.318 g (82%). IR (KBr): 3411.2s, 1634.1vs cm^{-1}. ^1H NMR ($CDCl_3$): δ 3.00 (s, 1H), δ 4.35 (d, $J = 4.8$ Hz,

1H), 4.80 (d, $J = 4.8$ Hz, 1H), 6.76–7.74 (m, 24H), 8.59 (d, $J_{PH} = 3.9$ Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 160.5 (d, C=N); ^{31}P NMR ($CDCl_3$) δ −10.37 (s); Positive ion FAB mass spectrum: m/z 486 ($M^+ + H$). Anal. Calcd. for $C_{28}H_{26}PNO \cdot (1/2)H_2O$: C, 77.76; H, 6.29; N, 3.24. Found: C, 77.41; H, 6.17; N, 3.04.

Ligand **1e**. (1*R*,2*S*)-Norephedrine was used. Yield: 0.264 g (78%). IR (KBr): 3411.2s, 1634.2s cm^{-1}. ^1H NMR ($CDCl_3$): δ 0.69 (d, $J = 6.6$ Hz, 3H), 3.43 (m, 1H), 4.56 (d, J = 3.9 Hz, 1H), 6.80–7.76 (m, 19H), 8.65 (d, $J_{PH} = 4.2$ Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 158.8 (d, C=N); ^{31}P NMR ($CDCl_3$) δ −10.15 (s); Positive ion FAB mass spectrum: m/z 424 ($M^+ + H$). Anal. Calcd. for $C_{33}H_{26}PNO \cdot (1/2)H_2O$: C, 80.14; H, 5.71; N, 2.83. Found: C, 79.85; H, 5.71; N, 2.60.

Ligand **1f**. (1*S*,2*S*)-2-Amino-3-methoxy-1-phenyl-1-propanol was used. Yield: 0.243 g (67%). IR (Nujol): 3390.0m, 1644.8m cm^{-1}. ^1H NMR ($CDCl_3$): δ 3.10 (s, 3H), δ 3.37 (d, $J = 1.5$ Hz, 2H), 3.45 (m, 1H), 4.86 (m, 1H), 6.94–7.67 (m, 19H), 8.32 (d, $J_{PH} = 3.9$ Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 162.6 (d, C=N); ^{31}P NMR ($CDCl_3$) δ −7.4 (s); Positive ion FAB mass spectrum: m/z 454 ($M^+ + H$). Anal. Calcd. for $C_{29}H_{28}PNO$: C, 76.80; H, 6.22; N, 3.09. Found: C, 76.58; H, 6.10; N, 2.83.

Ligand **1g**. (*S*)-2-Amino-1,1-diphenyl-1-propanol was used. Yield: 0.224 g (56%). IR (Nujol): 3506.7s, 1639.4vs cm^{-1}. ^1H NMR ($CDCl_3$): δ 0.82 (d, $J = 6.6$ Hz, 3H), δ 1.84 (s, 1H), 4.38 (m, 1H), 6.76–8.11 (m, 24H), 8.77 (d, $J_{PH} = 3.9$ Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 159.8 (d, C=N); ^{31}P NMR ($CDCl_3$) δ −10.7 (s); Positive ion FAB mass spectrum: m/z 500 ($M^+ + H$). Anal. Calcd. for $C_{34}H_{30}PNO \cdot (1/2)H_2O$: C, 80.29; H, 6.14; N, 2.75. Found: C, 79.82; H, 6.02; N, 2.63.

3.2. General procedure for the allylic substitution with carbon nucleophile

Palladium complex $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ (4 mg, 0.01 mmol) and chiral ligand **1** (0.025–0.2 mmol) were dissolved in dry CH_2Cl_2 (1 ml) under N_2 and stirred at room temperature for 1 h. Racemic 1,3-diphenyl-2-propenyl acetate **2** (0.126 g, 0.5 mmol) in dry CH_2Cl_2 (1 ml) was added dropwise into the reaction mixture. Nucleophile (1.5 mmol), BSA (0.37 ml, 1.5 mmol) and NaOAc (1 mg, 0.012 mmol) were added sequentially to the reaction mixture. The reaction mixture was stirred at room temperature and monitored by TLC. After the reaction, the mixture was diluted with H_2O and CH_2Cl_2. The organic layer was separated, washed with brine and dried over Na_2SO_4. The filtrate was concentrated with a rotary evaporator and the residue was purified by column chromatography. Enantiomeric excess was determined by HPLC using a Daicel chiralcel-OD or chiralpak AD column using 2-propanol and hexane as mobile phase.

3.3. General procedure for the allylic substitution with nitrogen nucleophile

The procedure was the same as the one with carbon nucleophile except that all reactions were run with no BSA.

Acknowledgements

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