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Readily available phosphine–imine ligands from α-phenylethylamine for highly efficient Pd-catalyzed asymmetric allylic alkylation

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Abstract

A series of novel chiral phosphine–imine ligands have been prepared by a two-step transformation from chiral α-phenylethylamine. The resulting chiral ligands were found to be effective for the palladium-catalyzed asymmetric allylic alkylation of 1,3-diphenylprop-2-en-1-yl pivalate with dimethyl malonate, in which up to 94% ee and 99% conversions were obtained. The results demonstrate that the chirality resided on the chelate ring of P–Pd–N complex is more effective for the transfer of the stereochemical information by comparison with the result obtained by Hashimoto and coworkers' phosphine–imine ligand, in which the chirality lay in the outside of P–Pd–N chelate ring. The effect of solvent, base and substitutent in phosphine–imine ligand on this catalytic reaction is also described.

Keywords: Palladium; Phenylethylamine; Phosphine–imine ligand; Asymmetric; Allylic alkylation

1. Introduction

The asymmetric alkylation of allylic electrophiles catalyzed by palladium complexes has been extensively investigated due to the synthetic potential of the products of these types of reactions [1–3]. A wide variety of ligands have been found to be effective for these transformations. Among them, chiral phosphine–Schiff-based imine ligands have received increasing attention recently due to their flexible coordination behaviors associated with tunable steric and electronic properties [4–14]. By appropriately electronic and steric modification, many phosphine–imine ligands have been developed and shown to be very effective in the Pd-catalyzed asymmetric allylic substitution. Recently, Hashimoto and coworkers reported a series of phosphine–imine ligands **1** derived from 1-arylethylamines and 2-(diphenylphosphino)benzaldehyde and examined their enantioselective induction in Pd-catalyzed asymmetric allylic alkylation [15]. It's found that most of these phosphine–imine ligands showed poor enantioselectivity, and good enantioselectivity could be obtained only when sterically congested (*R*)-1-mesitylethylamine was used as the amine component. This is perhaps not surprising, as these phosphine–imines were derived from 2-(diphenylphosphino)benzaldehyde, with the chirality remote from the reaction center, residing on the conformationally labile amino portion of the imine. We then surmised that if the chirality was placed on the chelate ring of P–Pd–N complex, the transfer of the stereochemical information might be more efficient. A new class of phosphine–imine ligands **2** are then proposed, which seemed ideal species to investigate since the Pd-complex formed by the reaction of these ligands with Pd precursor possess the chirality held in fairly rigid conformations. Our interest was augmented by our ready access to a chiral amino-phosphine precursor, (*S*)-1-[2-(diphenylphosphino)phenyl]ethylamine [(*S*)-DPPNH$_2$, **3**], easily prepared from commercially available, inexpensive chiral 1-phenylethylamine, which are the final intermediates in the synthesis of a variety of recently introduced phosphine–phosphoramidite ligands (PEAPhos) for highly efficient Rh-catalyzed asymmetric hydrogenation [16]. As a result, herein we report our results in the development of this kind of phosphine–imine ligands for highly efficient Pd-catalyzed asym-

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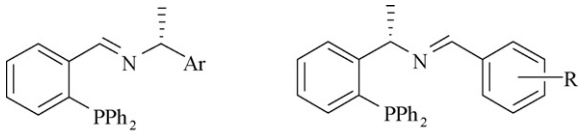


1a : Ar = Ph; **1b** : Ar = mesityl

2

Fig. 1. The structures of phosphine–imine ligands **1** and **2**.

metric allylic alkylation, in which up to 94% ee was obtained (Fig. 1).

2. Experimental

2.1. General methods

Melting points were measured on a Yazawa micro melting point apparatus (uncorrected). Optical rotations were measured on a JASCO P-1020 high sensitive polarimeter. The ^{1}H NMR spectra were recorded on a BRUKER DRX 400 system with TMS as an internal standard. The ^{31}P NMR spectra were recorded using a BRUKER DRX 400 system with 85% phosphoric acid as the external standard. Enantiomeric excesses (% ee) were determined by HPLC analysis using a Chiralpak AD column. All experiments were carried out under an argon atmosphere using standard Schlenk techniques. All solvents were dried using standard procedures. 1,3-Diphenylprop-2-en-1-yl pivalate **7** was prepared following a modified method for the corresponding acetate analogue [17]. All other chemicals were obtained commercially.

2.2. Synthesis of (S)-1-[2-(diphenylphosphino)phenyl] ethylamine [(S)-DPPNH$_2$] (**3**)

To a solution of (*S*)-1-phenylethylamine **4** (1.21 g, 10.0 mmol) in 10 ml of ether at −35 °C was added 6.25 ml (10.0 mmol) of a 1.6 M solution of *n*-BuLi in hexane dropwise. The resulting solution was stirred at −35 °C for 30 min, and then 1.39 ml (11.0 mmol, 1.1 equiv.) of Me_3SiCl was added slowly at the same temperature. The reaction mixture was stirred for another 2 h. Then 18.8 ml (30.0 mmol, 3.0 equiv.) of a 1.6 M solution of *n*-BuLi was added dropwise and the reaction mixture was allowed to slowly warm to room temperature during 5 h and stirred overnight. The reaction mixture was cooled to −35 °C, and a solution of 1.80 ml (10.0 mmol) of chlorodiphenylphosphine in 10 ml of ether was added dropwise during 1 h. The reaction mixture was stirred for another 3 h at the same temperature, and then warmed to room temperature. After stirring for another 4 h, a solution of 1.0 M aqueous HCl was added slowly until the reaction mixture became clear in both phases. The aqueous phase was extracted with ether (3 × 10 ml). Combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexanes/acetate, 8/1) to give 1.22 g (40% yield) of the targeted (*S*)-DPPNH$_2$ **3** as a white solid; mp 80–82 °C; $[\alpha]_D^{13}$ 56.7 (c 0.53, $CHCl_3$); ^{1}H NMR (DMSO-d^6) δ 1.23 (d, J = 6.8 Hz, 3H), 1.38 (s, 2H), 4.90 (m, 1H), 6.83–7.59 (m, 14H); ^{31}P NMR (DMSO-d^6) δ −16.3; HRMS (APCI) calcd. for $C_{20}H_{20}NP$ (M + 1) 306.1406, found 306.1376.

2.3. General procedure for the synthesis of phosphine–imine ligands (**2**)

To a solution of (*S*)-DPPNH$_2$ **3** (305 mg, 1.0 mmol) in 5.0 ml of ethanol was added the corresponding benzaldehyde (1.0 mmol) and anhydrous $MgSO_4$ (600 mg). The reaction mixture was heated to reflux. After the reaction was complete (detected by TLC), the reaction mixture was diluted with CH_2Cl_2. $MgSO_4$ was removed by filtration and the filtrate was concentrated under reduced pressure. The residue was purified by column chromatography.

2.3.1. (S)-N-(3-Nitrobenzylidene)-1-[2-(diphenylphosphino)phenyl]ethylamine (**2a**)

Green crystal; 62% yield; mp 99–101 °C; $[\alpha]_D^{14}$ 48.8 (c 1.10, $CHCl_3$); ^{1}H NMR (DMSO-d^6) δ 1.38 (d, J = 6.4 Hz, 3H), 5.40 (m, 1H), 6.76–8.28 (m, 18H), 8.38 (s, 1H); ^{31}P NMR (DMSO-d^6) δ −17.6; HRMS (APCI) calcd. for $C_{27}H_{23}N_2O_2P$ (M + 1) 439.1569, found 439.1554.

2.3.2. (S)-N-(3-Trifluoromethylbenzylidene)-1-[2-(diphenylphosphino)phenyl]ethyl-amine (**2b**)

White solid; 34% yield; mp 85–87 °C; $[\alpha]_D^{14}$ 34.2 (c 1.01, $CHCl_3$); ^{1}H NMR (DMSO-d^6) δ 1.46 (d, J = 6.4 Hz, 3H), 5.47 (m, 1H), 6.85–7.99 (m, 18H), 8.22 (s, 1H); ^{31}P NMR (DMSO-d^6) δ −17.6; HRMS (APCI) calcd. for $C_{28}H_{23}F_3NP$ (M + 1) 462.1593, found 462.1610.

2.3.3. (S)-N-(3-Methoxybenzylidene)-1-[2-(diphenylphosphino)phenyl]ethylamine (**2c**)

White solid; 54% yield; mp 110–112 °C; $[\alpha]_D^{14}$ 62.9 (c 0.98, $CHCl_3$); ^{1}H NMR (DMSO-d^6) δ 1.32 (d, J = 6.4 Hz, 3H), 3.77 (s, 3H), 5.29 (m, 1H), 6.99–7.79 (m, 18H), 7.96 (s, 1H); ^{31}P NMR (DMSO-d^6) δ −17.5; HRMS (APCI) calcd. for $C_{28}H_{26}NOP$ (M + 1) 424.1824, found 424.1813.

2.3.4. (S)-N-(2-Methoxybenzylidene)-1-[2-(diphenylphosphino)phenyl]ethylamine (**2d**)

White crystal; 43% yield; mp 140–142 °C; $[\alpha]_D^{14}$ −15.1 (c 0.66, $CHCl_3$); ^{1}H NMR (DMSO-d^6) δ 1.31 (d, J = 6.4 Hz, 3H), 3.80 (s, 3H), 5.26 (m, 1H), 6.94–7.80 (m, 18H), 8.39 (s, 1H); ^{31}P NMR (DMSO-d^6) δ −17.4; HRMS (APCI) calcd. for $C_{28}H_{26}NOP$ (M + 1) 424.1824, found 424.1798.

2.3.5. (S)-N-(2-Chlorobenzylidene)-1-[2-(diphenylphosphino)phenyl]ethylamine (**2e**)

White crystal; 58% yield; mp 88–90 °C; $[\alpha]_D^{14}$ −34.8 (c 0.8, $CHCl_3$); ^{1}H NMR (DMSO-d^6) δ 1.31 (d, J = 6.4 Hz, 3H), 5.36 (m, 1H), 6.77–7.89 (m, 18H), 8.38 (s, 1H); ^{31}P NMR (DMSO-d^6) δ −17.1; HRMS (APCI) calcd. for $C_{27}H_{23}NPCl$ 427.1257, found 427.1253.

*2.3.6. (S)-N-(2-Methybenzylidene)-1-[2-(diphenylphosphino)phenyl]ethylamine (**2f**)*

White crystal; 49% yield; mp 82–84 °C; $[\alpha]_D^{14}$ −57.5 (c 0.83, $CHCl_3$); ^{1}H NMR (DMSO-d^6) δ 1.36 (d, J = 6.4 Hz, 3H), 2.32 (s, 3H), 5.28 (m, 1H), 6.75–7.83 (m, 18H), 8.27 (s, 1H); ^{31}P NMR (DMSO-d^6) δ −17.0; HRMS (APCI) calcd. for $C_{28}H_{26}NP$ 407.1803, found 407.1796.

*2.3.7. (S)-N-(4-Methoxybenzylidene)-1-[2-(diphenylphosphino)phenyl]ethylamine (**2g**)*

Yellow solid; 57% yield; mp 48–50 °C; $[\alpha]_D^{14}$ 87.2 (c 0.99, $CHCl_3$); ^{1}H NMR (DMSO-d^6) δ 1.40 (d, J = 6.4 Hz, 3H), 3.88 (s, 3H), 5.33 (m, 1H), 6.84–7.88 (m, 18H), 8.01 (s, 1H); ^{31}P NMR (DMSO-d^6) δ −17.3; HRMS (APCI) calcd. for $C_{28}H_{26}NOP$ (M + 1) 424.1824, found 424.1848.

*2.3.8. (S)-N-(4-Chlolbenzylidene)-1-[2-(diphenylphosphino)phenyl]ethylamine (**2h**)*

White crystal; 49% yield; mp 108–110 °C; $[\alpha]_D^{14}$ −57.5 (c 0.83, $CHCl_3$); ^{1}H NMR (DMSO-d^6) δ 1.33 (d, J = 6.4 Hz, 3H), 5.31 (m, 1H), 6.77–7.78 (m, 18H), 8.01 (s, 1H); ^{31}P NMR (DMSO-d^6) δ −17.3; HRMS (APCI) calcd. for $C_{27}H_{23}NPCl$ 427.1257, found 427.1246.

*2.3.9. (S)-N-benzylidene-1-[2-(diphenylphosphino)phenyl] ethylamine (**2i**)*

White solid; 74% yield; mp 102–104 °C; $[\alpha]_D^{14}$ 44.1 (c 1.02, $CHCl_3$); ^{1}H NMR (DMSO-d^6) δ 1.42 (d, J = 6.2 Hz, 3H), 5.38 (m, 1H), 6.85–7.71 (m, 19H), 8.09 (s, 1H); ^{31}P NMR (DMSO-d^6) δ −17.4, HRMS (APCI) calcd. for $C_{27}H_{24}NP$ (M + 1) 394.1719, found 394.1690.

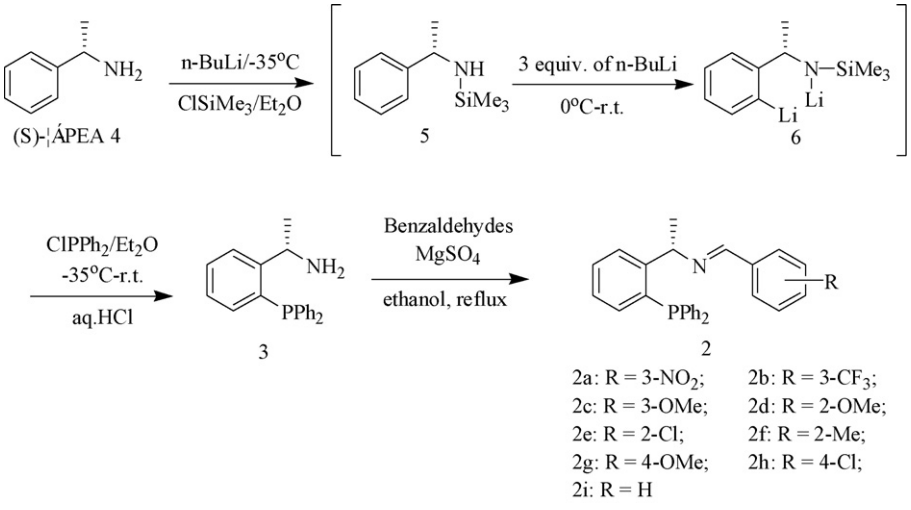
2.4. General procedure for Pd-catalyzed asymmetric allylic alkylation

A solution of $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ (3.7 mg, 0.01 mmol) and chiral phosphine–imine **2** (0.025 mmol) in toluene (1.5 ml) was stirred under argon atmosphere at room temperature for 1 h. To this Pd-catalyst was added allylic pivalate **7** (0.50 mmol) in toluene (1.5 ml), followed by dimethyl malonate (0.17 ml, 1.5 mmol), *N*,*O*-bis(trimethylsilyl)acetamide (BSA, 0.37 ml, 1.5 mmol), and a catalytic amount of Cs_2CO_3 sequentially. After stirring at room temperature for 24 h, the reaction mixture was quenched with a saturated solution of aqueous NH_4Cl and diluted with CH_2Cl_2. The organic layer was separated, dried over $MgSO_4$, and concentrated under reduced pressure. The residue was purified by column chromatography (hexanes:ethyl acetate, 8:1) to afford the pure product **8**. The enantiomeric excess was determined by HPLC (Chiralpak AD, hexane:2-propanol = 90:10, 1.0 ml/min). The absolute configuration was determined by comparing the specific rotation with a literature value.

3. Results and discussion

*3.1. Synthesis of chiral phosphine–amine precursor **3** and phosphine–imine ligands **2***

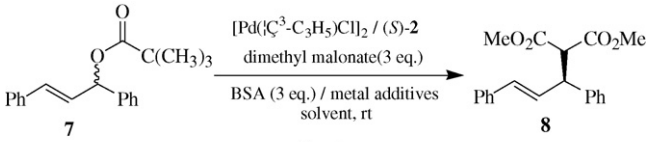
As we have reported [16], the synthesis of chiral phosphine–amine precursor (*S*)-$DPPNH_2$ **3** is straightforward, which is outlined in Scheme 1. In the initial step, commercially available (*S*)-α-phenylethylamine **4** was monosilylated with *n*-BuLi followed by Me_3SiCl to form *N*-trimethylsilylamine intermediates **5** [18], which need not be separated and purified for the next step. The resultant silylamine **5** was dilithiated by use of 3 equiv. of *n*-BuLi according to the general method of Corriu and coworkers [19]. The treatment of *N*-lithio-2-lithiosilylamine **6** with chlorodiphenylphosphine at −35 °C to room temperature afforded the targeted (*S*)-$DPPNH_2$ **3** in the total yields of 40% from (*S*)-α-PEA **1**. The resulting (S)-$DPPNH_2$ **3** was then converted into the corresponding phosphine–imine ligands **2** in the moderate to good yields by reaction with a variety of benzaldehydes in refluxing ethanol in the presence of anhydrous $MgSO_4$.



Scheme 1.

3.2. Pd-catalyzed asymmetric allylic alkylation

We then chose Pd-catalyzed asymmetric allylic alkylation of 1,3-diphenylprop-2-en-1-yl pivalate **7**, one of the model reactions for ligand evaluation, as the tested reaction to investigate the efficiency of our new developed phosphine–imine ligands in the asymmetric catalysis (Eq. (1)):



Eq. 1.

(1)

Initially, the optimization of reaction conditions was examined by use of phosphine–imine ligand **2a** with a meta-NO_2 group, and the results are summarized in Table 1. The reaction was performed at room temperature for 24 h in the presence of 2.0 mol% of $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$, 5.0 mol% of chiral ligand and 3 equiv. of *N*,*O*-bis(trimethylsilyl)acetamide (BSA) as base. The influence of solvents in the reaction was first examined and a significant variation in the catalytic activity and enantioselectivity on the nature of solvents was observed. Thus, when the reaction was carried out in toluene, an ee value of 87% with full conversions was obtained (entry 1). However, the reaction proceeded very slowly in ether, which gave the allylic product in only 19% yield (entry 2). Using CH_2Cl_2 as solvent, the enantioselectivity was dramatically dropped to 58% ee with the reduced yields (entry 3). When the reaction was carried out in THF, full conversions were observed but the enantioselectivity was decreased to 69% ee (entry 4). We then used toluene as solvent for evaluating the effect of the metal additives on the catalytic activity and enantioselectivity. Using NaOAc instead of KOAc gave the allylic product in the slightly decreased enantioselectivity (entry 6), while the use of LiOAc or CsOAc decreased the enantioselectivity dramatically to 59% ee and 71% ee, respectively (entries 5 and 7). It was found that using metal carbonate as metal additives was superior to the corresponding metal acetate. Thus, up to 92% ee with full conversions was obtained by use of Cs_2CO_3 as additives, while using the corresponding CsOAc only gave the allylic product in 71% ee with 80% yield (entry 9 *versus* entry 7).

Table 1
Pd-catalyzed asymmetric allylic alkylation of 1,3-diphenylprop-2-en-1-yl pivalate **7** using phosphine–imine ligand **2a**

Entry	Solvent	Base	Yield (%)[a]	ee (%) (configuration)[b]
1	Toluene	KOAc	99	87 (*S*)
2	Et_2O	KOAc	19	75 (*S*)
3	CH_2Cl_2	KOAc	62	58 (*S*)
4	THF	KOAc	99	69 (*S*)
5	Toluene	LiOAc	98	59 (*S*)
6	Toluene	NaOAc	98	84 (*S*)
7	Toluene	CsOAc	80	71 (*S*)
8	Toluene	K_2CO_3	99	90 (*S*)
9	Toluene	Cs_2CO_3	99	92 (*S*)

The reaction was carried out in the presence of 2.0 mol% of $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$, 5.0 mol% of ligand **2a**, 3.0 equiv. of dimethyl malonate, 3.0 equiv. of BSA and a catalytic amount of metal salt at room temperature for 24 h.

[a] Isolated yield.

[b] Determined by HPLC analysis using a Chiralpak AD column (eluent:hexane/2-propanol = 9/1, 1.0 ml/min). The (*S*)-configuration was confirmed by comparing the specific rotation with a literature value [19].

Table 2
Pd-catalyzed asymmetric allylic alkylation of 1,3-diphenylprop-2-en-1-yl pivalate **7** using phosphine–imine ligand **2**

Entry	Ligand	Yield (%)[a]	ee (%) (configuration)[b]
1	**2a**	99	92 (*S*)
2	**2b**	86	94 (*S*)
3	**2c**	99	92 (*S*)
4	**2d**	80	94 (*S*)
5	**2e**	80	92 (*S*)
6	**2f**	99	92 (*S*)
7	**2g**	80	87 (*S*)
8	**2h**	99	88 (*S*)
9	**2i**	99	94 (*S*)
10	**1a**	85	14 (−)

The reaction was carried out in toluene at room temperature for 24 h in the presence of 2.0 mol% of $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$, 5.0 mol% of ligand **2**, 3.0 equiv. of dimethyl malonate, 3.0 equiv. of BSA and a catalytic amount of Cs_2CO_3.

[a] Isolated yield.

[b] Determined by HPLC analysis using a Chiralpak AD column (eluent:hexane/2-propanol = 9/1, 1.0 ml/min). The (*S*)-configuration was confirmed by comparing the specific rotation with a literature value [19].

From the reaction conditions screening experiments, we then selected toluene as solvent, and Cs_2CO_3 as a base additive for the following investigation of the influence of ligand structure on the catalytic activity and enantioselectivity, and the results are summarized in Table 2. The results indicated that all of phosphine–imine ligands displayed the excellent enantioselectivity, and the substituents in the phenyl ring have some influence in the catalytic activity and enantioselectivity. Compared to the *meta*- and *ortho*-substituted analogues, *para*-substituted ligands exhibited lower enantioselectivity. Thus, **2c** with an *meta*-OMe substitutent and **2d** with an *ortho*-OMe substitutent provided 92% ee and 94% ee of the allylic product, respectively (entries 3 and 4); while ligand **2g** carrying a *para*-OMe group gave the allylic product in only 87% ee (entry 7). The phosphine–imine ligand **2i** without any substituents in the phenyl ring showed the best results, in which over 94% ee with full conversions was achieved (entry 9). The configuration of allylic alkylation product **8** from these reactions proved to be *S* by comparing the specific rotation with the literature values [20]. In sharp contrast, Hashimoto and coworkers' phosphine–imine ligand **1a** without any substituents in the phenyl ring only gave the allylic product in about 10% ee (entry 10). These results clearly demonstrate that the chirality placed on the P–Pd–N chelate ring is more effective for the transfer of the stereochemical information.

4. Conclusions

In conclusion, we have developed a series of new phosphine–imine ligands by the reaction of (*S*)-$DPPNH_2$ with

a variety of benzaldehydes, which were successfully applied in the palladium-catalyzed asymmetric allylic alkylation. The results indicated that the substituents in the phenyl ring have some influence in the catalytic activity and enantioselectivity. The best result (over 94% ee and full conversions) was obtained when phosphine–imine ligand **2i** without any substituents in the phenyl ring was used in the reaction. The results also demonstrate that the chirality resided on the P–Pd–N chelate ring is more effective for the transfer of the stereochemical information by comparison with the result obtained by Hashimoto and coworkers' phosphine–imine ligand. Further studies on the applications of (*S*)-DPPNH$_2$ in the development of new chiral ligands for asymmetric catalysis are still in progress.

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