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Palladium-catalyzed asymmetric allylic nucleophilic substitution reactions using chiral *tert*-butanesulfinylphosphine ligands

Junmin Chen [a,b,c], Feng Lang [a,b], Dong Li [a,b], Linfeng Cun [a], Jin Zhu [a,b], Jingen Deng [a,b], Jian Liao [a,b,*]

[a] National Engineering Research Center of Chiral Drugs and Key Laboratory of Asymmetric Synthesis and Chirotechnology of Sichuan Province, Chengdu Institute of Organic Chemistry, Chinese Academy of Sciences, Chengdu 610041, China
[b] Graduate School of Chinese Academy of Sciences, Beijing 100049, China
[c] College of Chemistry and Chemical Engineering, Jiangxi Normal University, Nanchang 330022, China

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ABSTRACT

The asymmetric allylic alkylation of racemic 1,3-diphenyl-2-propenyl acetate **3** with dimethyl malonate proceeded smoothly in the presence of lithium acetate, BSA (*N*,*O*-bis(trimethylsilyl)acetamide), $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$, and chiral *tert*-butanesulfinylphosphine ligand **2c** to give the allylic alkylation product in good yield and high enantiomeric excess (up to 93% ee), while the enantioselectivities of allylic amination of **3** with various amines were moderate (up to 76% ee).

1. Introduction

Palladium-catalyzed asymmetric allylic substitution has received considerable attention as a useful asymmetric carbon–carbon and carbon–heteroatom bond-forming process. Racemic or achiral allylic substrates can be converted into optically active products in the presence of a palladium complex of a chiral ligand.[1] In order to achieve high enantioselectivity, the development of novel chiral ligands is an essential and challenging task.[2] Some ligands have already been successfully produced and employed in various allylic alkylation reactions. However, the use of a chiral sulfoxide as a ligand is less common. Pioneered by Shibasaki in 1995,[3] (*S*,*S*)-1,2-bis(*p*-tolylsulfinyl)benzene (BTSB) was first applied as a ligand in an asymmetric allylic alkylation (AAA) reaction, which afforded moderate to good yields and moderate enantioselectivities. Since then, many chiral sulfoxide bidentate ligands such as other bis-sulfoxides,[4] sulfoxide–oxazoline,[5] sulfoxide–dialkyl amine/sulfoxide–sulfonamide,[6] and sulfoxide–phosphine,[7] have been applied to palladium-catalyzed asymmetric allylic substitution reaction without satisfactory results. In the case of sulfoxide ligands, sulfoxide–phosphine **1** showed very good potential in AAA reactions (Fig. 1), which generated up to 97% ee, but moderate yield (49%) when using 12 mol % ligand **1**.[7a] Encouraged by the high enantioselectivity with phosphine–sulfoxide ligands and as a part of our ongoing investigations of chiral ligands, we herein report *tert*-butanesulfinylphosphine ligands **2** in AAA reactions.

Recently, we have synthesized a class of chiral *tert*-butanesulfinylphosphine ligands **2a–c** and applied them to catalytic asymmetric diethylzinc addition to imines. High activities and enantioselectivities (up to 94% ee) were achieved.[8] Herein, we report the results of palladium-catalyzed enantioselective allylic substitutions of 1,3-diphenyl-2-propenyl acetate **3** with dimethyl malonate and various amines.

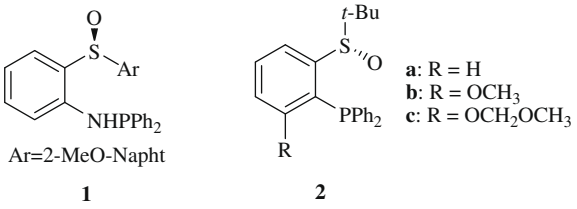

Figure 1.

2. Results and discussion

We first examined chiral ligands **2a–c** under the standard conditions reported by Mino et al.[9] On a 0.5 mmol scale, *rac*-1,3-diphenyl-2-propenyl acetate **3** reacted with 3.0 equiv dimethyl malonate in the presence of 2.0 mol % $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$, 5.2 mol % ligand (*S*)-**2**, 3.0 equiv *N*,*O*-bis(trimethylsilyl)acetamide (BSA), and a catalytic amount of lithium acetate in 1.0 mL acetonitrile. The results are summarized in Table 1. Under these conditions, the optically active substitution product **4** was obtained in quantitative yield when using ligands **2a–c**, and **2c** produced the best results (99% yield and 81% ee, Table 1, entry 3). The absolute configuration of **4** was determined to be (*S*) by comparing the specific rotation with the

* Corresponding author. Tel.: +86 28 85229250; fax: +86 28 85223978.
E-mail address: jliao10@cioc.ac.cn (J. Liao).

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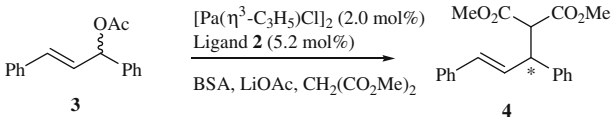
Table 1
The palladium-catalyzed asymmetric allylic alkylation reaction[a]



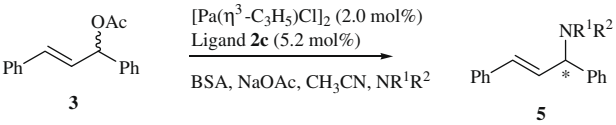
Entry	Ligand	Solvent	Temp. (°C)	Time (h)	Yield[b](%)	ee[c] (%)
1	**2a**	CH_3CN	rt	16	98	62
2	**2b**	CH_3CN	rt	16	99	73
3	**2c**	CH_3CN	rt	16	99	81
4[d]	**2c**	CH_3CN	rt	16	99	78
5[e]	**2c**	CH_3CN	rt	16	93	77
6	**2c**	CH_2Cl_2	rt	16	98	77
7	**2c**	EtOAc	rt	16	91	62
8	**2c**	Toluene	rt	10	94	68
9	**2c**	Et_2O	rt	10	94	64
10	**2c**	CH_3CN	0	24	99	89
11	**2c**	CH_3CN	−20	36	89	90
12	**2c**	CH_3CN	−40	48	79	91
13	**2c**	CH_3CN	−60	48	68	93

[a] Reaction conditions: substrate (0.5 mmol), 2.0 mol % $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$, 5.2 mol % ligand, 1.5 mmol *N,O*-bis(trimethylsilyl)acetamide (BSA), 1.5 mmol dimethyl malonate, 1 mg LiOAc, and 1.0 mL of solvent.
[b] Isolated yield.
[c] Determined by HPLC with a Daicel chiral column.
[d] NaOAc as salt.
[e] KOAc as salt.

literature value.[10] The effects of base and solvent on enantioselectivity were also investigated. Sodium acetate and potassium acetate led to a slight decrease in enantioselectivities (78% ee and 77% ee, respectively, Table 1, entries 4 and 5), and solvents such as dichloromethane, ethyl acetate, toluene, and diethylether also resulted in a decreased ee (Table 1, entries 6–9). To our delight, decreasing the temperature enhanced the enantioselectivity, although the reaction rate slowed (Table 1, entries 10–13). The optimal reaction temperature was hence assigned as −60 °C, which afforded the highest enantioselectivity (93% ee, Table 1, entry 13).

Having achieved enantioselective C–C bond formation, we also evaluated the chiral *tert*-butanesulfinylphosphine ligands in a C–N bond formation reaction. At room temperature, in the presence of ligand **2**, *rac*-1,3-diphenyl-2-propenyl acetate **3** was reacted with benzylamine under conditions similar to those of alkylation described above. We found that ligand **2c** and acetonitrile were also the best ligand and solvent, respectively. In addition, sodium acetate was superior to lithium or potassium salt in this reaction (Table 2, entries 1–3). Improved enantioselectivities (68% and 73% ee) were observed when the temperature was decreased to

Table 2
The palladium-catalyzed asymmetric allylic amination reaction[a]



Entry	Amine	Temp.[b](°C)	Time (h)	Yield[c](%)	ee[d](%)
1[e]	Benzylamine	rt	12	99	53
2[f]	Benzylamine	rt	12	99	62
3	Benzylamine	rt	12	99	63
4	Benzylamine	0	16	99	68
5	Benzylamine	−20	24	99	73
6	Benzylamine	−40	24	75	67
7	Morpholine	rt	10	99	76
8	Pyrrolidine	rt	16	98	52
9	Piperidine	rt	16	98	56
10	Dibenzylamine	rt	16	98	<5
11	Cyclohexylamine	0	16	98	70
12	Phathalimide	0	24	56	55
13	Boc-piperazine	0	24	93	73

[a] Reaction conditions: substrate (0.5 mmol), 2.0 mol % $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$, 5.2 mol % ligand, 1.5 mmol *N,O*-bis(trimethylsilyl)acetamide (BSA), 1.5 mmol amine, 1 mg NaOAc, and 1.0 mL CH_3CN.
[b] Three temperatures (−20 °C, 0 °C and rt) were screened for all substrates and the best results are presented in this table.
[c] Isolated yield.
[d] Determined by HPLC with a Daicel chiral column.
[e] LiOAc as salt.
[f] KOAc as salt.

0 °C and −20 °C (Table 2, entries 4 and 5). However, decreasing the temperature further (to −40 °C) was harmful to both enantioselectivity and the yield (Table 2, entry 6). With the reaction conditions optimized, other substrates were examined in this reaction. However, −20 °C was not the optimal temperature for several other amines, which we screened at three temperatures (−20 °C, 0 °C, and rt). As shown in Table 2, morpholine, pyrrolidine, piperidine, and dibenzylamine demonstrated excellent activities and moderate enantioselectivities (except dibenzylamine) at room temperature (Table 2, entries 7–10). Cyclohexylamine, phathalimide, and Boc-piperazine showed moderate to high yields and moderate enantioselectivities at 0 °C (Table 2, entries 11–13).

3. Conclusion

In conclusion, we have demonstrated that palladium complex derived from chiral *tert*-butanesulfinylphosphine ligand **2c** was an efficient catalyst for asymmetric allylic substitution of 1,3-diphenylpropenyl acetate **3** with dimethyl malonate (up to 93% ee) and nitrogen nucleophiles such as amines (up to 76% ee). Further studies focusing on the modification of the ligands and applications in other catalytic reactions are currently underway in our laboratory.

4. Experimental

All experiments were carried out under an argon atmosphere. Commercial reagents were used as received without purification. Commercial grade solvents were dried and purified by standard literature procedures. ^{1}H NMR spectra were recorded at 300 MHz and chemical shifts (δ) are reported in ppm relative to $CHCl_3$ (δ = 7.27 ppm). Optical rotation data were recorded on Perkin–Elmer Polarimeter-341. Enantiomeric excess was determined by HPLC analysis on chiral columns in comparison with the authentic racemates. Column chromatography was performed using silica gel (200–300 mesh) eluting with ethyl acetate and petroleum ether. The preparation of the chiral *tert*-butanesulfinylphosphine ligands was carried out according to the reported method.[8]

4.1. Typical procedure for the palladium-catalyzed asymmetric allylic alkylation

Under an argon atmosphere, to a Schlenck flask containing ligand **2c** (11.1 mg, 5.2 mol %) was added CH_3CN (1.0 ml) followed by $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ (3.7 mg, 4.0 mol % Pd). The mixture was stirred at room temperature for 45 min, and then *rac*-1,3-diphenyl-2-propenyl acetate **3** (126 mg, 0.5 mmol) was added to the reaction system via a syringe. The mixture was then cooled to -60 °C, and dimethyl malonate (0.17 ml, 1.5 mmol) was added to the mixture followed by *N*,*O*-bis(trimethylsilyl)acetamide (BSA, 0.37 mL, 1.5 mmol) and lithium acetate (1.0 mg). After 48 h, the reaction was diluted with Et_2O, washed with saturated NH_4Cl(aq), saturated $NaHCO_3$(aq), and brine. The combined aqueous solutions were extracted with CH_2Cl_2. The combined organic solutions were dried over $MgSO_4$, filtered, concentrated in vacuo, and purified by flash chromatography (85% hexane, 15% ethyl acetate) to yield a yellow oil (68% yield); $[\alpha]_D^{25} = -21.4$ (*c* 1.40, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 300 MHz): δ 3.52 (s, 3H), 3.70 (s, 3H), 3.95 (d, *J* = 10.8 Hz, 1H), 4.27 (dd, *J* = 8.7 Hz, *J* = 10.8 Hz, 1H), 6.34 (dd, *J* = 8.4 Hz, *J* = 15.9 Hz, 1H), 6.48 (d, *J* = 15.6 Hz, 1H),7.20–7.34 (m, 10H). The enantiomeric excess was determined by chiral HPLC using a Chiralcel AD-H column, hexane/propan-2-ol = 95/5 (V/V), 1.0 mL/min, 254 nm; 93% ee, (*S*)-isomer t_r = 14.6 min, (*R*)-isomer t_r = 19.8 min. The absolute stereochemistry of the product [(*S*)-isomer] was determined by comparison of the specific rotation to the literature value.[10]

4.2. Typical procedure for the palladium-catalyzed asymmetric allylic amination

Under an argon atmosphere, to a Schlenck flask containing ligand **2c** (11.1 mg, 5.2 mol %) was added CH_3CN (1 ml) followed by $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ (3.7 mg, 4.0 mol % Pd). The mixture was stirred at room temperature for 45 min, and then *rac*-1,3-diphenyl-2-propenyl acetate **3** (126 mg, 0.5 mmol) was added to the reaction via a syringe. The mixture was cooled to −20 °C, benzylamine (0.16 mL, 1.5 mmol) was added to the reaction, followed by *N*,*O*-bis(trimethylsilyl)acetamide (BSA, 0.37 mL, 1.5 mmol) and sodium acetate (1.0 mg). After 24 h, the reaction was diluted with Et_2O, washed with saturated NH_4Cl(aq), saturated $NaHCO_3$(aq), and brine. The combined aqueous solutions were extracted with CH_2Cl_2. The combined organic solutions were dried over $MgSO_4$, filtered, concentrated in vacuo, and purified by flash chromatography (85% hexane, 15% ethyl acetate) to yield a yellow oil (99% yield); $[\alpha]_D^{25} = -7.6$ (*c* 0.48, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 300 MHz): δ 3.80 (s, 2H), 4.42 (d, *J* = 7.2 Hz, 1H), 6.36 (dd, *J* = 7.2 Hz, *J* = 15.9 Hz, 1H), 6.60 (d, *J* = 15.9 Hz, 1H), 7.22–7.47 (m, 15H). The enantiomeric excess was determined by chiral HPLC using a Chiralcel AD-H column, hexane/propan-2-ol = 90/10 (V/V), 1.0 mL/min, 254 nm; 73% ee, (*R*)-isomer t_r = 9.9 min, (*S*)-isomer t_r = 12.0 min. The absolute stereochemistry of the product [(*R*)-isomer] was determined by comparison of the specific rotation to the literature value.[11]

4.2.1. (*R*)-*N*-[(*E*)-1,3-Diphenylallyl]cyclohexanamine

Colorless oil, 98% yield, $[\alpha]_D^{25} = -8.5$ (*c* 0.46, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 300 MHz): δ 0.87–1.27 (m, 6H), 1.69 (br, 1H), 2.00 (br, 2H), 1.89–1.97 (m, 2H), 2.44–2.48 (m, 1H), 4.58 (d, *J* = 7.4 Hz, 1H), 6.34 (dd, *J* = 8.1 Hz, *J* = 15.9 Hz, 1H), 6.54 (d, *J* = 15.9 Hz, 1H), 7.20–7.42 (m, 10H). The enantiomeric excess was determined by chiral HPLC using a Chiralcel AD-H column, hexane/propan-2-ol = 99/1 (V/V) (0.1% Et_3N), 0.5 mL/min, 254 nm; 70% ee, (*R*)-isomer t_r = 7.9 min, (*S*)-isomer t_r = 7.0 min. Absolute stereochemistry of the product [(*R*)-isomer] was determined by comparison of the specific rotation to the literature value.[11]

4.2.2. (*R*)-*N*-[(*E*)-1,3-Diphenylprop-2-enyl]morpholine

Pale solid, 99% yield, $[\alpha]_D^{25} = -7.4$ (*c* 0.34, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 300 MHz): δ 2.36–2.57 (m, 4H), 3.70–3.73 (m, 4H), 3.79 (d, *J* = 9.0 Hz, 1H), 6.36 (dd, *J* = 8.6 Hz, *J* = 15.8 Hz, 1H), 6.58 (d, *J* = 15.8 Hz, 1H), 7.21–7.42 (m, 10H). The enantiomeric excess was determined by chiral HPLC using a Chiralcel OD-H column, hexane/propan-2-ol = 90/10 (V/V), 1.0 mL/min, 254 nm; 76% ee, (*R*)-isomer t_r = 12.2 min, (*S*)-isomer t_r = 6.3 min. The absolute stereochemistry of the product [(*R*)-isomer] was determined by comparison of the specific rotation to the literature value.[11]

4.2.3. (*R*)-*N*-[(*E*)-1,3-Diphenylprop-2-enyl]pyrrolidine

Pale solid, 98% yield, $[\alpha]_D^{25} = -2.6$ (*c* 1.0, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.79–1.84 (m, 4H), 2.43–2.58 (m, 4H), 3.76 (d, *J* = 8.4 Hz, 1H), 6.36 (dd, *J* = 8.4 Hz, *J* = 15.8 Hz, 1H), 6.58 (d, *J* = 15.8 Hz, 1H), 7.12–7.44 (m, 10H). The enantiomeric excess was determined by chiral HPLC using a Chiralcel OD-H column, hexane/propan-2-ol = 70/30 (V/V), 1.0 mL/min, 254 nm; 52% ee, (*R*)-isomer t_r = 7.5 min, (*S*)-isomer t_r = 16.1 min. The absolute stereochemistry of the product [(*R*)-isomer] was determined by comparison of the specific rotation to the literature value.[12]

4.2.4. (*R*)-*N*-[(*E*)-1,3-Diphenylprop-2-enyl]piperidine

Pale solid, 98% yield, $[\alpha]_D^{25} = -11.7$ (*c* 0.32, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.26–1.61 (m, 6H), 2.31–2.45 (m, 4H), 3.81 (d, *J* = 8.4 Hz, 1H), 6.36 (dd, *J* = 8.1 Hz, *J* = 15.8 Hz, 1H), 6.53 (d, *J* = 15.8 Hz, 1H), 7.21–7.42 (m, 10H). The enantiomeric excess was determined by chiral HPLC using a Chiralcel AD-H column,

hexane/propan-2-ol = 70/30 (V/V), 0.5 mL/min, 254 nm; 56% ee, (*R*)-isomer t_r = 6.9 min, (*S*)-isomer t_r = 7.3 min. The absolute stereochemistry of the product [(*R*)-isomer] was determined by comparison of the specific rotation to the literature value.[11]

4.2.5. (2*R*,*E*)-*N*,*N*-Dibenzyl-1,4-diphenylbut-3-en-2-amine

Colorless oil, 98% yield, $[\alpha]_D^{25} = -2.9$ (*c* 0.50, $CHCl_3$); ^1H NMR ($CDCl_3$, 300 MHz): δ 3.62 (d, *J* = 13.8 Hz, 2H), 3.74 (d, *J* = 13.8 Hz, 2H), 4.44 (d, *J* = 6.9 Hz, 1H), 6.50–6.52 (m, 2H), 7.21–7.57 (m, 20H). The enantiomeric excess was determined by chiral HPLC using a Chiralcel AD-H column, hexane/propan-2-ol = 70/30 (V/V), 0.5 mL/min, 254 nm; 3% ee, (*R*)-isomer t_r = 6.7 min, (*S*)-isomer t_r = 7.0 min. The absolute stereochemistry of the product [(*R*)-isomer] was determined by comparison of the specific rotation to the literature value.[13]

4.2.6. (*R*)-*N*-[(*E*)-1,3-Diphenyl-2-propenyl]phthalimide

Colorless oil, 56% yield, $[\alpha]_D^{25} = -36.5$ (*c* 0.50, $CHCl_3$); ^1H NMR ($CDCl_3$, 300 MHz): δ 6.14 (d, *J* = 8.7 Hz, 1H), 6.74 (d, *J* = 15.9 Hz, 1H), 7.04 (dd, *J* = 8.4 Hz, *J* = 15.9 Hz, 1H), 7.26–7.86 (m, 14H). The enantiomeric excess was determined by chiral HPLC using a Chiralcel OD-H column, hexane/propan-2-ol = 90/10 (V/V), 1.0 mL/min, 254 nm; 55% ee, (*R*)-isomer t_r = 7.9 min, (*S*)-isomer t_r = 7.0 min. The absolute stereochemistry of the product [(*R*)-isomer] was determined by comparison of the specific rotation to the literature value.[11]

4.2.7. (*R*)-*N*-[(*E*)-1,3-Diphenylprop-2-enyl)]-*N*-Boc-piperazine

White solid, 93% yield, $[\alpha]_D^{25} = -16.0$ (*c* 0.50, $CHCl_3$); ^1H NMR ($CDCl_3$, 300 MHz): δ 1.45 (s, 9H), 2.36–2.54 (m, 4H), 3.43 (br, 4H), 3.82 (d, *J* = 9.0 Hz, 1H), 6.26 (dd, *J* = 8.1 Hz, *J* = 15.9 Hz, 1H), 6.55 (d, *J* = 15.9 Hz, 1H), 7.19–7.41 (m, 10H). The enantiomeric excess was determined by chiral HPLC using a Chiralcel AD-H column, hexane/propan-2-ol = 90/10 (V/V), 1.0 mL/min, 254 nm; 73% ee, (*R*)-isomer t_r = 5.9 min, (*S*)-isomer t_r = 5.4 min. The absolute stereochemistry of the product [(*R*)-isomer] was determined by comparison of the specific rotation to the literature value.[12]

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