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Note

Palladium-catalyzed asymmetric allylic alkylations of cycloalkenyl acetates with planar chiral phosphino-ferrocene carboxylic acids

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Abstract

New phosphino-ferrocene carboxylic acids with only planar chirality (*Sp*)-**3** and (*Sp*)-**4** have been synthesized conveniently from (*S*,*Sp*)-**5** and (*S*,*Sp*)-**7**. They were used as ligands in palladium-catalyzed allylic alkylation of cycloalkenyl acetates. With ligand (*Sp*)-**4**, high yield and good ee were given for reactions of a series of cycloalkenyl acetates and nucleophiles.

Keywords: Alkylation; Asymmetric catalysis; Ferrocene; Planar chirality

1. Introduction

Palladium-catalyzed allylic substitution reactions have been one of the most intense topics in asymmetric synthesis during the past two decades [1]. Various ligands have been synthesized and proved to be powerful in these reactions, especially with 1,3-diphenylprop-2-enyl acetate [2]. On the other hand, substitutions with cyclic allylic substrate are more difficult to achieve high enantioselectivity [3]. Among a few successful systems, chiral phosphinocarboxylic acids, such as **1** and **2** (Fig. 1), have been found to be effective for these types of substrates [4].

In our group, many ferrocene ligands have been synthesized and applied into asymmetric palladium-catalyzed allylic substitution reactions [5,6]. During this process, planar chiral phosphino-ferrocene carboxylic acids (*Sp*)-**3** and (*Sp*)-**4** were important intermediates for synthesizing planar chiral ligands [7,8]. However, we found these two compounds were also useful ligands in palladium-catalyzed allylic alkylation reaction. In this paper, we report the synthesis of ligands with only planar chirality (*Sp*)-**3** and (*Sp*)-**4**, and their applications in palladium-catalyzed asymmetric allylic alkylation of cycloalkenyl acetates.

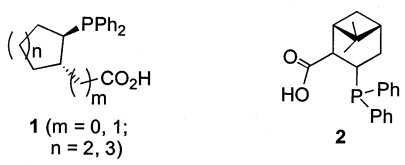

Fig. 1. Ligands **1** and **2**.

Ligands **3** and **4**

2. Results and discussion

The synthesis of ligands (*Sp*)-**3** and (*Sp*)-**4** was shown in Scheme 1 [6e,7a]. (*S*,*Sp*)-**5** and (*S*,*Sp*)-**7** could be synthesized from commercially available ferrocene and enantiopure aminoalcohol. Following Meyer's method [6e,7a,9], the oxazoline was converted conveniently to

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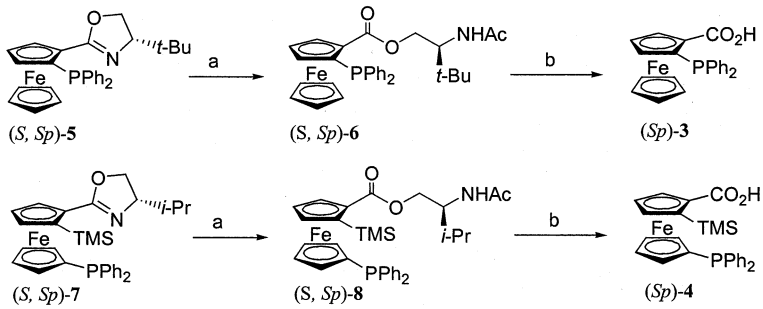


Scheme 1. Condition and reagent. (a) (1) TFA, Na_2SO_4, H_2O; (2) Ac_2O, Pyr. (b) (1) NaOH, MeOH, THF; (2) H_3O^+.

esters (*S*,*Sp*)-**6** and (*S*,*Sp*)-**8**. In the presence of NaOH, (*S*,*Sp*)-**6** was hydrolyzed to (*Sp*)-**3** in high yield after being acidified with HCl. Following the same method, (*Sp*)-**4** was also synthesized from (*S*,*Sp*)-**8** in high yield. Both of them were orange solids and were very stable in air.

To examine the catalytic efficiency of (*Sp*)-**3** and (*Sp*)-**4** in palladium-catalyzed allylic alkylation reactions of cycloalkenyl acetates, the most used condition, reaction of cyclohexenyl acetate with the nucleophile derived from dimethylmalonate was chosen (Scheme 2) and the results were summarized in Table 1.

From the results listed in Table 1, we found the reaction ran very slowly with ligand (*Sp*)-**3** at ambient temperature. However, the reaction could be completed in 1 day by raising the temperature to 50 °C. The product **10** was given in 96% yield and 30.0% ee. Ligand (*Sp*)-**4** was shown to be more effective in this reaction. Even at ambient temperature, the reaction was completed in 1 h with 50.1% ee and 98% yield. On considering both reactivity and enantioselectivity, ligand (*Sp*)-**4** is better.

With ligand (*Sp*)-**4**, different cycloalkenyl acetates and nucleophiles were investigated (Scheme 3). The results are summarized in Table 2.

All reactions proceeded smoothly and the products were given in high yield. In previous works [3,4], the ee of the products were determined mainly by using optical rotation, ^{1}H-NMR chiral shifting reagents or GC. In addition to these methods, in this work, the ee's were also determined by separating the products using chiral HPLC.

In order to investigate how the steric demanding of the nucleophiles affects the enantioselectivity, more steric demanding nucleophiles like sodium diethylmalonate and sodium di-*tert*-butylmalonate were used in the reaction of cyclohexenyl acetate. We found that the ee of the products decreased a little. The result was also unsatisfactory for cyclopentenyl acetate, only 31.8% ee was given. However, the highest ee of 65.5 was obtained for the seven-member ring substrate, cycloheptenyl acetate.

With these two ligands, the enantioselectivity of the products was determined only by the planar chirality [10]. As part of our program for studying the role of planar chirality [6,7,11], the results here were very interesting. It indicated also that the ferrocene modified planar chiral pocket could show better asymmetric induction in some reaction than the phenyl derived chiral pocket ligands [7,8], for that a moderate enantioselectivity could be caused by the planar chirality of phosphino-ferrocene carboxylic acid moiety.

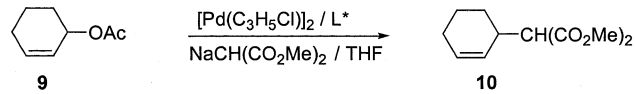
3. Conclusions

New planar chiral ferrocene phosphino-carboxylic acids (*Sp*)-**3** and (*Sp*)-**4** have been synthesized and used as ligands in palladium-catalyzed allylic alkylation with cycloalkenyl acetates. With 1,1′-disubstituted ligand (*Sp*)-**4**, high yield and moderate to good ee could be achieved for a series of cycloalkenyl acetates and nucleophiles.

4. Experimental

4.1. General methods

All reactions were performed under an atmosphere of either Ar or N_2 using oven-dried glassware. Solvents were treated prior to use according to the standard method. The commercially available reagents were used as received without further purification. Melting points were uncorrected. ^{1}H-NMR spectra were recorded in a Bruker AMX-400 (400 MHz) or AMX-300 (300 MHz) spectrometer in $CDCl_3$ at room temperature (r.t.). ^{31}P-NMR spectra were recorded in a Bruker AMX-400 (162 MHz) spectrometer and the chemical shifts were



Scheme 2.

Table 1
Palladium-catalyzed allylic alkylation of **9** with different ligands [a]

Entry	Ligand	Temperature	Time	Yield (%) [b]	ee (%) [c]
1	(*Sp*)-**3**	r.t.	2 days	<10%	–
2	(*Sp*)-**3**	50	1 day	96	30.0 (*R*)
3	(*Sp*)-**4**	r.t.	1 h	98	50.1 (*R*)

[a] Reactions were performed with molar ratio: $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$–ligand–**9**–$NaCH(CO_2Me)_2$ = 2:8:100:200.
[b] Isolated yield based on **9**.
[c] Determined by HPLC (chiracel OD column) and the absolute configuration of product was assigned through comparison of the sign of specific rotations with the literature data [3e].

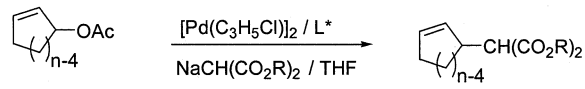
referenced to external 85% H_3PO_4. Chemical shifts were given in parts per million relative to Me_4Si as an internal standard. Optical rotations were measured using a Perkin–Elmer 241 MC polarimeter with a thermally jacketed 10 cm cell at 25 °C (concentration *c* given as g 100 ml^{-1}). IR spectra were measured in cm^{-1}, using a Shimadzu IR-440 infrared spectrophotometer. Mass spectra and high-resolution mass spectra were taken using HP5989A and Finnigan MAT mass spectrometers, respectively. Elemental analyses were performed in a Foss-Heraeus Vario EL instrument. The ee values were determined by chiral HPLC on a Chiralcel AS column.

4.2. (S,Sp)-2-(Diphenylphosphino)-[1-(N-acetyl)-2-tert-butyl-2-amino ethoxycarboxyl]-1-ferrocene (S,Sp)-6

A solution of (*S*,*Sp*)-**5** (1.98 g, **4** mmol) in THF (40 ml) was treated with powdered Na_2SO_4 (29 g, 60 mmol), water (4 ml, 220 mmol) and trifluoroacetic acid (1.74 ml, 22 mmol), The suspension was stirred for 3 days, and anhydrous Na_2SO_4 (8.4 g, 60 mmol) was added. Filtration and concentration at < 30 °C afforded the unstable ammonium salt, which was dissolved in CH_2Cl_2 (70 ml), cooled in an ice bath, and treated sequentially with Ac_2O (14 ml, 15 mmol) and Py (22 ml, 27 mmol). The reaction mixture was allowed to warm to ambient temperature over 7 h. The solution was washed with cold 3 N HCl (3 × 100 ml) and saturated $NaHCO_3$ (100 ml). The organic layer was dried and the solvent was removed in vacuo. The crude product was purified by column chromatography (EtOAc–petroleum ether = 1:3) to afford **6** (1.40 g, 63%) as a yellow solid: m.p. 188–189 °C (dec); $[\alpha]_D^{20}$ = −231 (*c* 0.33, $CHCl_3$). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.52 (m, 2H), 7.39–7.41 (m, 3H), 7.22–7.24 (m, 3H), 7.14–7.15 (m, 2H), 5.92 (d, *J* = 9.4 Hz, 1H), 5.08 (m, 1H), 4.64 (dd, *J* = 6.8 Hz, 11.5 Hz, 1H), 4.48 (t, *J* = 2.6 Hz, 1H), 4.17 (s, 5H), 4.09 (m, 1H), 3.95 (dd, *J* = 4.0 Hz, 11.5 Hz, 1H), 3.80 (m, 1H), 1.74 (s, 3H), 1.01 (s, 9H). EIMS; *m*/*z* (relative intensity): 555 [M^+, 100], 490 (14), 414 (73), 369 (51), 213 (26). IR (KBr, cm^{-1}) 3332 (m), 3057 (w), 2966 (m), 1702 (s), 1652 (s), 1434 (m), 1254 (s), 1165 (s), 696 (m). Anal. Found: C, 66.57; H, 6.23; N, 2.32. Calc. for $C_{31}H_{34}FeNO_3P$: C, 67.00; H, 6.13; N, 2.52%.

4.3. (Sp)-2-(Diphenylphosphino)-1-ferrocene carboxylic acid (Sp)-3

Under Ar, to a solution of (*S*,*Sp*)-**6** (555 mg, 1 mmol) in 30 ml THF and 10 ml MeOH, 4 ml 2.5 N NaOH (10 mmol) was added. The mixture was refluxed for 2 h, cooled to r.t. and the solvent was removed. With an ice bath the mixture was acidified with 1 N HCl to pH 1. The resulting solution was extracted with CH_2Cl_2, the organic layer was dried and the solvent was removed in vacuo. The crude product was purified by column chromatography (EtOAc–petroleum ether = 1:2) to afford (*Sp*)-**3** (392 mg, 95%) as an orange solid: m.p. 158–160°C; $[\alpha]_D^{20}$ = −296 (*c* 0.31, $CHCl_3$). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.49–7.51 (m, 2H), 7.37–7.39 (m, 3H), 7.17–7.26 (m, 5H), 5.09 (m, 1H), 4.94 (t, *J* = 2.5 Hz, 1H), 4.24 (s, 5H), 3.79 (m, 1H); ^{31}P-NMR (161.92 MHz, $CDCl_3$) δ −17.46. EIMS; *m*/*z* (relative intensity): 414 [M^+, 100], 368 (86), 303 (39),



Scheme 3.

Table 2
Palladium-catalyzed allylic alkylation of cycloalkenyl acetates with (*Sp*)-**4** [a]

Entry	Substrate	R	Yield (%) [b]	ee (%) [c]
1	*n* = 6	Me	98	50.1
2	*n* = 6	Et	95	40.3
3	*n* = 6	^{t}Bu	91	48.4
4	*n* = 5	Me	95	31.8
5	*n* = 7	Me	94	65.5

[a] Reactions were performed with molar ratio: $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$–(*Sp*)-**4**–cycloalkenyl acetates–$NaCH(CO_2R)_2$ = 2:8:100:200.
[b] Isolated yield based on cycloalkenyl acetates.
[c] Determined by HPLC.

121 (19), 57 (42), 44 (59). IR (KBr, cm^{-1}) 3400 (w), 3054 (m), 2923 (m), 1705 (s), 1437 (s), 1156 (s), 1069 (w). HRMS Found: 414.0469. Calc. for $C_{23}H_{19}O_2PFe$: 414.0466.

4.4. (S,Sp)-1-Diphenylphosphino-1′-[N-acetyl-2-isopropyl-2-aminoethoxycarbonyl]-2′-(trimethylsilyl)-ferrocene (S,Sp)-8

By a similar procedure as for (*S*,*Sp*)-**6**, (*S*,*Sp*)-**8** was obtained in 54% yield (10 mmol scale) from (*S*,*Sp*)-**7** as a yellow solid: m.p. 85–86 °C; $[\alpha]_D^{20} = -132.0°$ (*c* 0.32, $CHCl_3$). ^{1}H-NMR δ 7.24–7.40 (m, 10H), 6.05 (d, $J = 8.9$ Hz, 1H), 4.85 (m, 1H), 4.45 (s, 1H), 4.35 (m, 1H), 4.07–4.28 (m, 7H), 1.99 (m, 1H), 1.87 (s, 3H), 1.03 (d, $J = 6.8$ Hz, 3H), 1.01 (d, $J = 6.8$ Hz, 3H), 0.27 (s, 9H); ^{31}P-NMR (161.92 MHz, $CDCl_3$) δ −17.29. MS; *m*/*z* (relative intensity): 613 [M^+, 27], 486 (100), 442 (29), 321 (28), 170 (25), 86 (18). IR (KBr, cm^{-1}) 3285, 2960, 1714, 1649, 1550, 1434, 1247, 1157, 835, 696. Anal. Found: C, 64.02; H, 6.46; N, 2.19. Calc. for $C_{33}H_{40}NO_3PSiFe$: C, 64.60; H, 6.57; N, 2.28%.

4.5. (Sp)-1-Diphenylphosphino-2′-(trimethylsilyl)-1′-ferrocene carboxylic acid (Sp)-4

A similar procedure as for (*Sp*)-**3** gave (*Sp*)-**4** in 83% yield (1 mmol scale) from (*S*,*Sp*)-**8**: $[\alpha]_D^{20} = +36.5°$ (*c* 0.19, $CHCl_3$). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.25–7.38 (m, 10H), 4.90 (dd, $J = 1.4$, 2.3 Hz, 1H), 4.47 (m, 1H), 4.43 (s, 1H), 4.33 (t, $J = 2.5$ Hz, 1H), 4.29 (m, 1H), 4.23 (s, 1H), 4.19 (s, 1H), 0.28 (s, 9H); ^{31}P-NMR (161.92 MHz, $CDCl_3$) δ −17.52. EIMS; *m*/*z* (relative intensity): 486 [M^+, 100], 471 (13), 442 (26), 321 (13), 226 (19). IR (KBr, cm^{-1}) 2500–3000 (w), 1376 (s), 1459 (s), 1247 (m), 836 (s). Anal. Found: C, 63.98; H, 5.53. Calc. for $C_{26}H_{27}O_2SiPFe$: C, 64.20; H, 5.59%.

4.6. General procedure for the palladium-catalyzed allylic alkylation of cycloalkenyl acetate

$[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ (3.7 mg, 0.01 mmol) and ligand (0.04 mmol) were dissolved in dry THF (2 ml), and then stirred for 30 min at r.t. under Ar. To this solution were successively added cycloalkenyl acetate (0.5 mmol), sodium dimethylmalonate (1.0 mmol, prepared in situ). The reaction mixture was stirred at r.t. and monitored by TLC. After completion, the reaction mixture was diluted with Et_2O (20 ml) and washed twice with ice-cold saturated aqueous NH_4Cl. The organic phase was dried over anhydrous Na_2SO_4 and then concentrated under reduced pressure. The residue was purified by column chromatography (EtOAc–petroleum ether = 1:10) to afford the pure product. The enantiomeric purities were determined by HPLC analysis.

4.6.1. Propanedioic acid, 2-cyclohexen-1-yl-dimethyl ester

^{1}H-NMR (300 MHz, $CDCl_3$) δ 5.74–5.82 (m, 1H), 5.52 (dd, $J = 2.3$, 10.2 Hz, 1H), 3.75 (s, 6H), 3.29 (d, $J = 9.2$ Hz, 1H), 2.86–2.95 (m, 1H), 1.96–2.06 (m, 2H), 1.26–1.81 (m, 4H). Chiralcel As, flow rate: 0.7 ml min^{-1}, *n*-hexane–*i*-PrOH = 100:4, 226 nm, 10.0 min (*S*), 11.2 min (*R*).

4.6.2. Propanedioic acid, 2-cyclohexen-1-yl-diethyl ester

^{1}H-NMR (300 MHz, $CDCl_3$) δ 5.73–5.80 (m, 1H), 5.53–5.57 (m, 1H), 4.16–4.24 (m, 4H), 3.24 (d, $J = 9.4$ Hz, 1H), 2.90 (m, 1H), 1.97–2.02 (m, 2H), 1.71–1.77 (m, 2H), 1.57–1.63 (m, 1H), 1.37–1.41 (m, 1H), 1.27 (t, $J = 7.1$ Hz, 6H). Chiralcel As, flow rate: 0.7 ml min^{-1}, *n*-hexane–*i*-PrOH = 95:5, 226 nm, t_R 7.99, 9.44 min.

4.6.3. Propanedioic acid, 2-cyclohexen-1-yl-di-tert-butyl ester

^{1}H-NMR (300 MHz, $CDCl_3$) δ 5.71–5.76 (m, 1H), 5.58–5.62 (m, 1H), 3.03 (d, $J = 9.2$ Hz, 1H), 2.79 (m, 1H), 1.96–2.02 (m, 2H), 1.47 (s, 18H), 1.40–1.82 (m, 4H). Chiralcel As, flow rate: 0.7 ml min^{-1}, *n*-hexane–*i*-PrOH = 100:0.1, 226 nm, t_R 16.8, 19.0 min.

4.6.4. Propanedioic acid, 2-cyclopenten-1-yl-dimethyl ester

^{1}H-NMR (300 MHz, $CDCl_3$) δ 5.82–5.85 (m, 1H), 5.64–5.67 (m, 1H), 3.75 (s, 6H), 3.26–3.37 (m, 2H), 2.30–2.38 (m, 2H), 2.09–2.16 (m, 1H), 1.54–1.63 (m, 1H). Chiralcel As, flow rate: 0.7 ml min^{-1}, *n*-hexane–*i*-PrOH = 90:10, 226 nm, t_R 9.18, 9.99 min.

4.6.5. Propanedioic acid, 2-cyclohepten-1-yl-dimethyl ester

^{1}H-NMR (300 MHz, $CDCl_3$) δ 5.81–5.85 (m, 1H), 5.57–5.62 (m, 1H), 3.74 (s, 6H), 3.47 (d, $J = 8.5$ Hz, 1H), 3.05 (m, 1H), 2.12–2.18 (m, 1H), 1.92–1.97 (m, 1H), 1.59–1.69 (m, 3H), 1.33–1.41 (m, 2H). Chiralcel As, flow rate: 0.7 ml min^{-1}, *n*-hexane–*i*-PrOH = 90:10, 226 nm, t_R 8.63, 9.59 min.

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