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Synthetic Communications

An International Journal for Rapid Communication of Synthetic Organic Chemistry

ISSN: 0039-7911 (Print) 1532-2432 (Online) Journal homepage: https://www.tandfonline.com/loi/lsyc20

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To cite this article: De-Quan Wei, Xiu-Shuai Chen, Chuan-Jin Hou & Xiang-Ping Hu (2019): Iridium-catalyzed asymmetric hydrogenation of β-keto esters with new phenethylamine-derived tridentate P,N,N-ligands, Synthetic Communications, DOI: 10.1080/00397911.2018.1550203

To link to this article: https://doi.org/10.1080/00397911.2018.1550203

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https://doi.org/10.1080/00397911.2018.1550203

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Iridium-catalyzed asymmetric hydrogenation of β-keto esters with new phenethylamine-derived tridentate P,N,N-ligands

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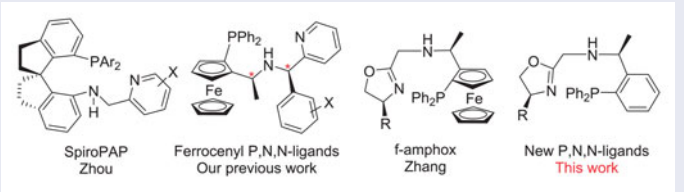
ABSTRACT

A new class of phenethylamine-derived tridentate P,N,N-ligands has been successfully developed. These ligands exhibited good performance in the Ir-catalyzed asymmetric hydrogenation of β-keto esters, affording the corresponding β-hydroxy esters in moderate to good enantioselectivities.

GRAPHICAL ABSTRACT



ARTICLE HISTORY
Received 6 October 2018

KEYWORDS
Iridium; P,N,N- ligands; asymmetric hydrogenation; β-keto esters

Introduction

Over the past few decades, transition metal-catalyzed asymmetric transformations have become extremely powerful tools for the synthesis of optically pure compounds, in which the design and synthesis of chiral ligands is the key issue for achieving high reactivity and selectivity. In 1968, Knowles and Horner reported the asymmetric hydrogenation of prochiral olefins with methylphenylpropylphosphine as a chiral monodentate phosphine ligand.[1] Although very low enantioselectivity (3–15% *ee*) was obtained, it represented the first homogeneous transition-metal catalyzed asymmetric transformation. In 1972, Kagan developed a diphosphine ligand, 2,3-O-Isopropylidene-2,3-dihydroxy-1,4-bis(diphenylphosphino)butane (DIOP), which showed good performance in the Rh-catalyzed asymmetric hydrogenation of dehydro *N*-acylamino acids.[2] A major breakthrough was obtained by Noyori in 1980, who developed an axially chiral bisphosphine ligand, 2,2′-bis (diphenylphosphino)-1,1′-binaphthyl (BINAP).[3] The BINAP represented one of the most

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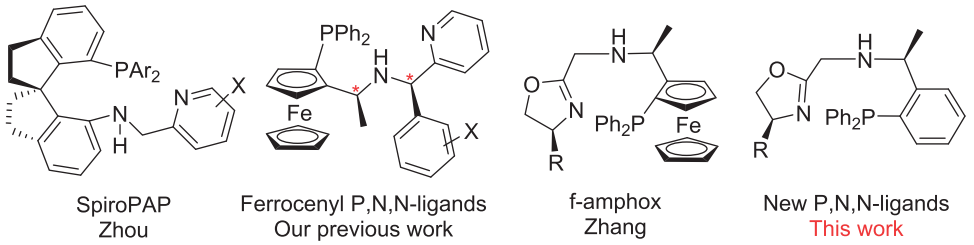


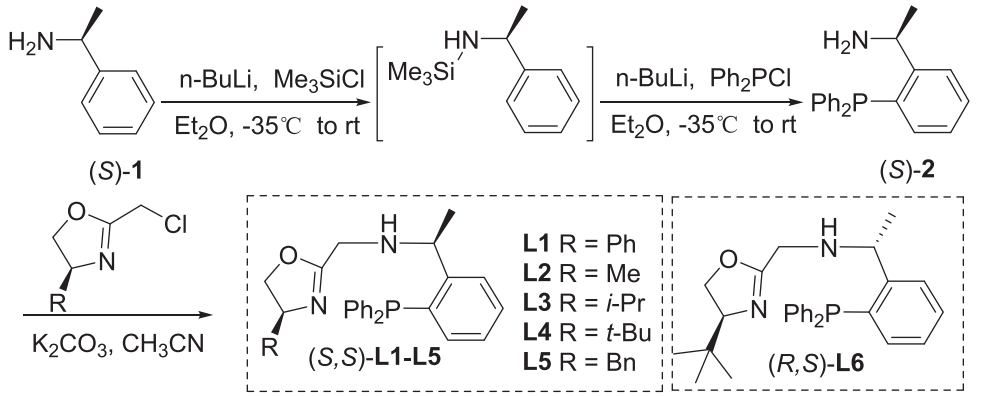
Figure 1. Examples of efficient chiral P,N,N- ligands in asymmetric hydrogenation.

effective chiral bisphosphine ligands, which exhibited high enantioselectivity in several asymmetric reactions. Since then, thousands of chiral ligands were developed and applied in transition-metal catalyzed asymmetric transformations.[4] Because of their milestone contributions to the asymmetric hydrogenation, Knowles[5] and Noyori[6] were awarded the Nobel Prize in 2001.

The P,N,N-ligands represent a new type of tridentate ligand developed in the past decade. Indeed, these ligands have been found to display wide utility in asymmetric catalysis, and given excellent results in Ru-catalyzed asymmetric cyclopropanation,[7] Ru-catalyzed asymmetric transfer hydrogenation,[8] Pd-catalyzed asymmetric allylic substitution,[9] Cu-catalyzed asymmetric 1,4-addition,[10] Cu-catalyzed asymmetric propargylic substitution,[11] Cu-catalyzed asymmetric decarboxylative propargylic substitution,[12] Cu-catalyzed asymmetric Friedel-Crafts propargylic alkylation,[13] Cu-catalyzed asymmetric [3 + 2] cycloaddition,[14] [3 + 3] cycloaddition[15] and [4 + 2] cycloaddition[16]. Despite these achievements, development of chiral P,N,N-ligands in asymmetric hydrogenation remains still slowly, although asymmetric hydrogenation is one of the most direct and convenient methods for the synthesis of chiral compounds. In 2011, Zhou[17] developed a chiral spiro P,N,N-ligand, SpiroPAP (Fig. 1), which showed excellent enantioselectivity in the Ir-catalyzed asymmetric hydrogenation of various ketones. Inspired by SpiroPAP ligand, we[18] have disclosed that the chiral ferrocenyl P,N,N-ligand was efficient for the Ir-catalyzed asymmetric hydrogenation of ketones and β-keto esters. More recently, Zhang[19] have successfully developed a tridentate ferrocene aminophosphoxazoline P,N,N-ligand, f-amphox, and applied them in the Ir-catalyzed asymmetric hydrogenation of ketones with excellent enantioselectivity. Based on our success in the asymmetric catalysis with chiral phenethylamine-derived ligands, we introduced a chiral phenethylamine motif to replace the ferrocenylethylamine motif in f-amphox ligands, forming a new type of phenethylamine-derived tridentate P,N,N-ligands. Herein we would like to report the synthesis of the new tridentate P,N,N-ligands and application in the iridium catalyzed asymmetric hydrogenation of β-keto esters.

Results and discussion

The novel phenethylamine-derived tridentate P,N,N-ligands were easily prepared *via* a simple synthetic route (Scheme 1). Starting from commercially available (*S*)-α-phenylethylamine **1**, the aminophosphine (*S*)-**2** was easily obtained and subsequently reacted with various (*S*)-2-chloromethyloxazoline in the presence of K_2CO_3 generating the desired ligands (*S*,*S*)-**L1**-**L5** in moderate yields. In order to investigate the relationship between the



Scheme 1. Synthesis of P,N,N-ligands **L1**-**L6**.

stereoselectivity and configuration of the ligand, we also synthesized another ligand (*R*,*S*)-**L6**, which is the diastereoisomer of ligand (*S*,*S*)-**L4**.

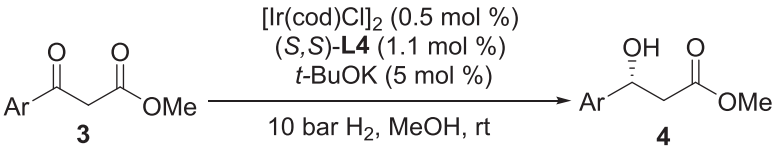
With the newly developed ligands **L1**-**L6** in hand, we began to evaluate them for the iridium catalyzed asymmetric hydrogenation of methyl 3-oxo-3-phenylpropanoate (**3a**) serving as the model substrate with the catalyst generated *in situ* by mixing $[Ir(cod)Cl]_2$ with ligands **L1**-**L6** in MeOH. As shown in Table 1, ligand (*S*,*S*)-**L1**-**L5** showed high reactivities and moderate to good enantioselectivities (entries 1–5). The ligand screening results demonstrated that the substituents on the oxazoline showed an important effect on the enantioselectivity. Ligand (*S*,*S*)-**L4** with *t*-Bu substituent afforded the best result with 82% *ee* (entry 4). In sharp contrast, low enantioselectivity was obtained when the ligand (*R*,*S*)-**L6** was applied to this transformation (entry 6). It was shown that the configuration of the ligand was also very critical for the enantioselectivity and the (*S*,*S*)-configuration was the matched ligand configuration. The solvent screen results showed that MeOH was the best choice for the hydrogenation, while other solvents showed low reactivities and enantioselectivities (entries 7–9).

Encouraged by these results, a study of the reaction with various *β*-aryl-*β*-keto esters **3** were performed to evaluate the scope and limitation of the reaction, and the results were summarized in Table 2. The results indicated that in the presence of $[Ir(cod)Cl]_2$ and ligand (*S*,*S*)-**L4**, a series of *β*-keto esters **3** can be hydrogenated smoothly to afford various chiral *β*-hydroxy esters **4** with good enantioselectivities (entries 1-11). The substrates containing electron-donating groups and electron-withdrawing groups on the phenyl ring performed well with good results (entries 1-8). In addition, 2-naphthyl substrate **3i** served well and gave the hydrogenation product **4i** with 72% *ee* (entry 9). Meanwhile, the heterocyclic substrate **3j** and **3k** also proceeded efficiently with 74% and 78% *ee*, respectively (entries 10-11).

Conclusion

In conclusion, we have successfully developed a series of phenethylamine-derived tridentate P,N,N-ligands for the Ir-catalyzed asymmetric hydrogenation of *β*-keto esters. Under the optimized conditions, a wide range of *β*-keto esters can be hydrogenated

Table 1. Asymmetric hydrogenation of Methyl 3-oxo-3-phenylpropanoate (**3a**): ligand screening[a].

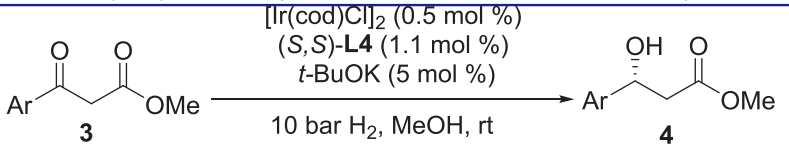
[


]

entry	ligand	yield (%)[b]	ee (%)[c]
1	(*S,S*)-**L1**	95	42
2	(*S,S*)-**L2**	95	28
3	(*S,S*)-**L3**	95	31
4	(*S,S*)-**L4**	95	82
5	(*S,S*)-**L5**	95	32
6	(*R,S*)-**L6**	95	33
7[d]	(*S,S*)-**L4**	90	22
8[e]	(*S,S*)-**L4**	60	69
9[f]	(*S,S*)-**L4**	70	46

[a]Hydrogenation was carried out with 0.5 mol % of $[Ir(cod)Cl]_2$, 1.1 mol % of ligands and 5 mol % of *t*-BuOK under 10 bar of H_2 at room temperature for 12h.
[b]isolated yield.
[c]Enantiomeric excesses were determined by HPLC using a chiral stationary phase.
[d]EtOH was used as solvent and the corresponding ethyl ester was obtained.
[e]Toluene was used as solvent.
[f]$ClCH_2CH_2Cl$ was used as solvent.

Table 2. Asymmetric hydrogenation of β-keto esters **3** with Ir/(*S,S*)-**L4**[a].Table Layout



entry	substrate	Ar	yield (%)[b]	ee (%)[c]
1	**3a**	C_6H_5	95	82
2	**3b**	2-ClC_6H_4	96	62
3	**3c**	3-ClC_6H_4	90	77
4	**3d**	4-ClC_6H_4	93	74
5	**3e**	4-BrC_6H_4	98	75
6	**3f**	4-FC_6H_4	92	76
7	**3g**	4-MeC_6H_4	92	79
8	**3h**	4-$MeOC_6H_4$	92	61
9	**3i**	2-Naphthyl	94	72
10	**3j**	2-Furyl	90	74
11	**3k**	2-Thienyl	91	78

[a]Hydrogenation was carried out with 0.5 mol % of $[Ir(cod)Cl]_2$, 1.1 mol % of ligands (*S,S*)-**L4** and 5 mol % of *t*-BuOK under 10 bar of H_2 at room temperature for 12h.
[b]isolated yield.
[c]Enantiomeric excesses were determined by HPLC using a chiral stationary phase and the absolute configuration was determined by comparison of the sign of the specific rotation with the reported data.

smoothly and good enantioselectivities have been achieved. Further application of these ligands in asymmetric hydrogenation is in progress.

Experimental

The hydrogenation reaction was carried out in glove-box by use of a stainless steel autoclave. Solvents were purified by standard procedure and commercial reagents were used

without further purification.[1]HNMR spectra were recorded on a Bruker 400 MHz spectrometer. Chemical shifts were expressed in δ value (ppm) using tetramethylsilane (TMS) as an internal standard. HPLC analysis was performed on an Agilent 1100 series instrument with a chiralpak AS-H, chiralcel OD-H, chiralcel AD-H or chiralcel OJ-H column. Optical rotations were recorded on a JASCO P-1020 polarimeter. The absolute configurations of the products were determined by comparing optical rotation with the reported data.

General procedure for ligand synthesis

The ligands **L1**-**L6** were synthesized as follow: To a solution of aminophosphine **2** (1.0 mmol) and K_2CO_3 (2.5 mmol) in anhydrous CH_3CN (7.0 mL) was added 2-chloromethyloxazoline (1.1 mmol) under a N_2 atmosphere. The reaction mixture obtained was stirred at 85 °C for several hours. The solvent was removed under the cacuum. The mixture was diluted with water and extracted with ethyl acetate. The organic layer was dried with anhydrous Na_2SO_4 and concentrated in vacuo to afford the crude product, which was purified by chromatography to give the corresponding ligands **L1**-**L6** with moderate yields.

(S)-1-(2-(diphenylphosphino)phenyl)-N-(((S)-4-phenyl-4,5-dihydrooxazol-2-yl)methyl)ethanamine L1

Colorless viscous liquid, 31% yield, $[\alpha]_D^{20}$ = –69.3 (*c* 1.0, CH_2Cl_2);^{1}H NMR (400 MHz, DMSO) δ 7.74–7.65 (m, 1H), 7.44–7.30 (m, 9H), 7.29–7.13 (m, 8H), 6.88-6.68 (m, 1H), 5.12 (t, J = 9.3 Hz, 1H), 4.83–4.63 (m, 1H), 4.60 (q, J = 8.4 Hz, 1H), 3.84 (t, J = 8.4 Hz, 1H), 3.20 (d, J = 15.9 Hz, 1H), 3.04 (d, J = 15.9 Hz, 1H), 1.09 (d, J = 6.4 Hz, 3H);^{31}P NMR (162 MHz, DMSO) δ –19.08; ^{13}C NMR (100 MHz, DMSO) δ 167.0, 150.4 (d, J = 22.4 Hz), 143.1, 137.3 (d, J = 11.6 Hz), 136.7 (d, J = 10.9 Hz), 135.2 (d, J = 13.5 Hz), 134.2, 134.0, 133.7, 133.5, 133.4, 130.1, 129.5, 129.3, 129.2, 129.1, 128.9, 127.6, 127.5, 127.2, 126.5 (d, J = 5.1 Hz), 74.6, 69.0, 54.1 (d, J = 26.4 Hz), 43.9, 24.3; HRMS Calcd for $C_{30}H_{30}N_2OP$ $[M+H]^+$: 465.2096, found: 465.2097.

General procedure for asymmetric hydrogenation of β-keto esters 3

In a nitrogen-filled glovebox, a stainless steel autoclave was charged with $[Ir(cod)Cl]_2$ (3.4 mg, 0.005 mmol) and (*S*,*S*)-**L4** (4.9 mg, 0.011 mmol) in 1.0 mL of dry MeOH. After stirring for 1h at room temperature, a solution of the substrates **3** (1.0 mmol) and *t*-BuOK (5.6 mg, 0.05 mmol) in 2.0 mL of MeOH was added to the reaction mixture, and then the hydrogenation was performed at room temperature under an H_2 pressure of 10 bar for 12 h. The solvent was evaporated and the residue was purified by flash column chromatography to give the corresponding hydrogenation product, which was analyzed by chiral HPLC to determine the enantiomeric excesses.

(R)-methyl 3-hydroxy-3-phenylpropanoate 4a

Colorless oil was obtained in 95% yield. 82% *ee* was determined by chiral HPLC (chiralcel OD-H, *n*-hexane/*i*-PrOH =90/10, 0.8 mL/min, 210 nm, 40 °C): t_R (minor) = 11.1 min, t_R (major) = 15.2 min. $[\alpha]_D^{20}$ = +20.4 (*c* 1.0, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41–7.25 (m, 5H), 5.13 (dd, J = 8.9, 4.0 Hz, 1H), 3.72 (s, 3H), 3.05 (br), 2.82–2.67 (m, 2H). EI-MS ($C_{10}H_{12}O_3$) m/z (%): 180 (16), 120 (14), 107 (100), 77 (57), 51 (12).

Acknowledgements

The authors are grateful for the financial support from the National Natural Science Foundation of China (Nos. 21403022, 21572226 and 21772196), Program of Star of Dalian Youth Science and Technology (No 2016RQ062), the Fundamental Research Funds for the Higher Education of Liaoning Province (2017J027), and Liaoning BaiQianWan Talents Program (201892153).

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