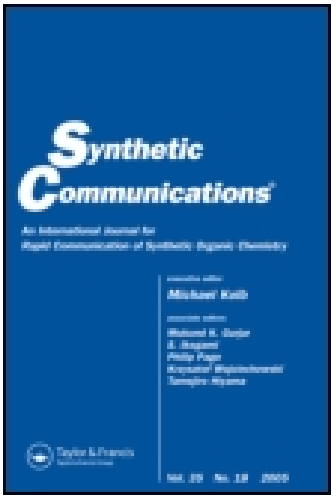

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Asymmetric Allylation of Aldimines with Indium and (+)-Cinchonine

Rongbi Han [a b], Seung Hyuk Choi [a], Kee In Son [a], Young Moo Jun [a], Byung Min Lee [c] & Byeong Hyo Kim [a]

[a] Department of Chemistry, Kwangwoon University, Seoul, South Korea

[b] Department of Chemistry, Yanbian University, Jilin, China

[c] Korea Research Institute of Chemical Technology, Taejon, South Korea

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Asymmetric Allylation of Aldimines with Indium and (+)-Cinchonine

Rongbi Han
Department of Chemistry, Kwangwoon University, Seoul, South Korea
and Department of Chemistry, Yanbian University, Jilin, China

Seung Hyuk Choi, Kee In Son, and Young Moo Jun
Department of Chemistry, Kwangwoon University, Seoul, South Korea

Byung Min Lee
Korea Research Institute of Chemical Technology, Taejon, South Korea

Byeong Hyo Kim
Department of Chemistry, Kwangwoon University, Seoul, South Korea

Abstract: By applying indium and allyl bromide, aldimines were converted into homoallyl amines with excellent yields. The addition of (+)-cinchonine to the reaction mixture yielded enantioselective allylation with 22–44% ee.

Keywords: Enantioselective allylation, indium, aldimines, homoallylamines, chiral ligand

Asymmetric allylations on prochiral faces of carbonyl and imine functionalities have been widely used for the preparation of optically active homoallyl alcohols and amines.[1] Despite their popularity, the preparation of optically active homoallyl amines remains a difficult procedure. To overcome this challenge, a wide variety of metals and stereochemical controlling techniques have been applied to the allylation of imine.[2] Among the many possible candidate metals, indium appears to be the appropriate choice for this

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Address correspondence to Byeong Hyo Kim, Department of Chemistry, Kwangwoon University, Seoul 139-701, South Korea. E-mail: bhkim@daisy.kw.ac.kr

purpose. Compared with other transition metals, such as zinc and heavy metal tin, indium shows incredible stability in a variety of atmospheres and has a relatively low ionization potential.[3] The first ionization potential of indium (5.78 V) is closer to alkali and alkali earth metals, which contrasts with that of zinc (9.39 V) and tin (7.34 V). Particularly, indium is so stable in air and humid conditions that even water can be used as a reaction medium in some cases.[4] Many groups,[5] including the authors of this paper,[6] have recently reported their utilization of indium for the synthetic transformation.

The studies on enantioselective additions to the imine C═N bond with organometallic reagents can be grouped into two categories: 1) the introduction of intrinsic chiral substituents into the substrate imine molecule and 2) the employment of chiral ligands as an external source of chiral auxiliary via complexation. Most of the stereoselective manipulations of imine with allylindium fall under the first category; however, it has a limitation resulting from the difficulty of the proper chiral ligand selection. Catalytic Lewis acid promoters were able to enhance the reactivity in some cases,[7] but they also generated the undesirable reverse reaction of converting imine back to carbonyl and amine functionality.

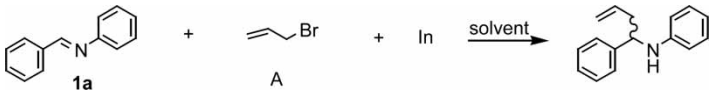
In our previous study, SmI_2-mediated allylation of imines was successful.[8] Enatioselective allylation, however, did not work very well with SmI_2. As a simple approach to synthesizing the optically active homoallyl amines, the results of the stereoselective allylation of aldimines with indium and chinchona alkaloid chiral ligands are herein reported.

The Barbier-type allylation of imine to produce homoallyl amine was carried out in an *N*-(benzylidene)aniline system to preclude potential complications during the reaction process, such as enamination or enolization within the substrates. Among many reaction variables, the optimum molar ratio was obtained when the excessive allyl bromide (6 equiv.), indium (6 equiv.) and limiting amounts of the substrate were used in THF as shown in Table 1 (entry 3). The reaction in the cosolvent (THF–hexane = 3:1) exhibited an excellent yield similar to the reaction in the THF solution; however, the reaction time was extended up to 12 h. The relatively mild reaction condition produced a better yield than the cold temperature and was distinctively noticeable even though the subsequent temperature elevation did not help improve the reaction yield (Table 1, entries 5, 6).

With the optimized reaction condition, simple allylation of various *N*-(benzylidene)aniline derivatives were examined prior to the asymmetric allylation application. As shown in Table 2, excellent yields of homoallyl amine, ranging from 81 to 97%, were obtained within 2–3 h. The yield was not significantly affected by each substituent attachment to the rings, regardless of its position.

For the asymmetric allylation of aldimines, the application of chiral ligands to the reaction system was examined based on the results of the allylation of conventional allylation that was described previously. It was observed that the application of ligands to the reaction system slowed down the reaction

Table 1. Controlled reactions for the formation of *N*-phenyl-α-2-propenyl-benzenemethanamine under various reaction conditions

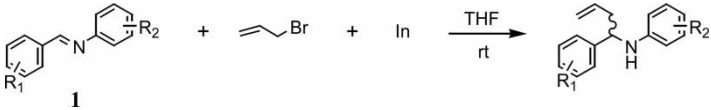


Entry	Molar ratio **1**[a]:A:In	Solvent	Temp. (°C)	Time (h)	Yield (%)[b]
1	1:6:4	THF	rt	11	51
2	1:6:5	THF	rt	10	53
3	1:6:6	THF	rt	2	98
4	1:6:6	THF/hexane[c]	rt	12	96
5	1:6:6	THF	−78°C (2 h) → rt	4	37
6	1:6:6	THF	0°C (2 h) → rt	4	33

[a]0.5 mmol of substrate was used.
[b]GC yield with an internal standard (n-octane).
[c]3:1 v/v ratio.

process. As shown in Table 3, the additions of (+)-cinchonine, (−)-cinchonidine and *N*-(4-trifluoromethylbenzyl)cinchoninium bromide all required a considerably prolonged reaction time, compared to the reaction without. Screening of the performance of some of the suitable chiral ligands indicated that the (+)-cinchonine produced the best result in terms of the

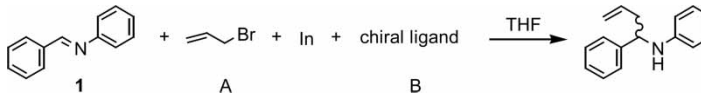
Table 2. Allyation of *N*-(benzylidene)aniline derivatives by indium in optimal condition, substrate (1 equiv.)/allyl bromide (6 equiv.)/indium (6 equiv.) in THF at room temperature



Entry	Substrate	Time (h)	Product	Yield (%)[a]
1	**1a**; R_1 = H, R_2 = H	2	**2a**	96
2	**1b**; R_1 = 4-chloro, R_2 = H	2	**2b**	83
3	**1c**; R_1 = 4-ethoxy, R_2 = H	2	**2c**	81
4	**1d**; R_1 = 3-bromo, R_2 = H	2	**2d**	89
5	**1e**; R_1 = 4-methyl, R_2 = H	3	**2e**	88
6	**1f**; R_1 = 4-isopropyl, R_2 = H	2	**2f**	97
7	**1g**; R_1 = 3-chloro, R_2 = H	2	**2g**	93
8	**1h**; R_1 = H, R_2 = 2-methoxy	2	**2h**	75

[a]Isolated yield.

Table 3. Formation of *N*-phenyl-α-2-propenyl-benzenemethanamine with various chiral ligands



Entry	Molar ratio **1**:**A**:In:**B**	Chiral ligand	Temp.	Time (h)	Yield (%)[a]	ee (%)
1	1:6:4:2	(+)-cinchonine	rt	22	73	24
2	1:6:6:2	(+)-cinchonine	rt	20	76	28
3	1:6:6:2	(+)-cinchonine	0°C (1 h) → rt	18	60	22
4	1:6:6:2	(−)-cinchonidine	rt	20	75	0
5	1:6:6:2	*N*-(4-trifluoro-methylbenzyl) cinchoninium bromide	rt	18	79	6
6	1:6:6:2	(+)-cinchonine[b]	rt	4	50	16
7	1:6:6:3	(+)-cinchonine	rt	48	30	32

[a]GC yield with an internal standard(n-octane).
[b]$InCl_3$ was added.

enantiomeric excess (ee), whereas (−)-cinchonidine exhibited absolutely no enantiomeric excess (Table 3).

This result was somewhat surprising because when the substrates had carbonyl functionality, (−)-cinchonidine demonstrated the best catalytic activity rather than the (+)-cinchonine, which is quite contrary to the imine case.[9] The anticipated chiral ligand, *N*-(4-trifluoromethylbenzyl)cinchoninium bromide, gave only unsatisfactory results in view of its enantiomeric excess (Table 3, entry 5).

Given (+)-cinchonine as a catalytic ligand and other variables, the optimal reaction condition was reached as illustrated in Table 3, when the

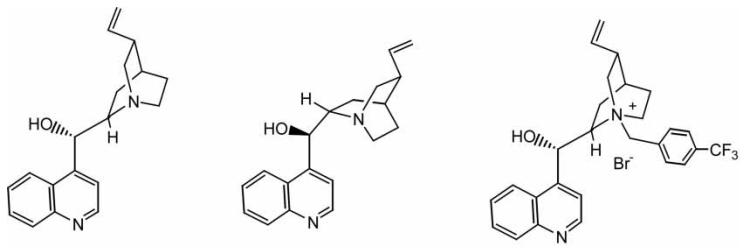


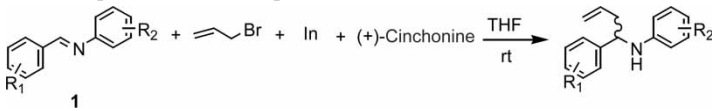
Figure 1. The chiral ligands tested in this study.

ratio of the substrate (1 equiv.), allylbromide (6 equiv.), indium (6 equiv.), and the ligand (2 equiv.) produced the best result. It is noteworthy that the addition of a Lewis acid promoter such as indium(III) chloride did not improve the prospects; rather, it deteriorated both the chemical yield and the ee (Table 3, entry 6). Lower temperature also lowered the yields, even though the temperature was raised from the low initial temperature application (Table 3, entry 3). The attempted experiment of increasing the chiral ligand concentration (3 equiv.) gave rise to only a doubled reaction time and lowered chemical yields and ee (Table 3, entry 7).

The optimized reaction condition was applied to various *N*-(benzylidene)aniline derivatives for the asymmetric allylation application. As shown in Table 4, moderate ee was observed in most cases. The yield and ee were not significantly affected by each substituent attachment to the rings, regardless of its position.

There are some reports that the mixed solvent system drastically improved both the chemical yields and the ee in the case of the enantioselective allylation of aldehydes and ketones with base additives.[10] Different combinations of hydrocarbon mixed solvents did not significantly improve the ee, and neither did the application of ether lower the ee. The optimal condition for the use of the mixed solvent for the allylation of *N*-(benzylidene)aniline was the

Table 4. Allylation of various *N*-(benzylidene)aniline derivatives by indium in optimal condition, substrate (1 equiv.)/allyl bromide (6 equiv.)/indium (6 equiv.)/(+)-cinchonine (2 equiv.) at room temperature



Entry	Substrate	Solvent	Time (h)	Product	Yield (%)[a]	ee (%)
1	**1a**; $R_1 = H$, $R_2 = H$	A	20	**2a**	76	28
2		B	26	**2a**	89	33
3	**1b**; $R_1 = 4$-Cl, $R_2 = H$	A	17	**2b**	86	28
4		B	25	**2b**	91	30
5	**1c**; $R_1 = 4$-OEt, $R_2 = H$	A	26	**2c**	84	24
6		B	22	**2c**	68	44
7	**1d**; $R_1 = 3$-Br, $R_2 = H$	A	26	**2d**	88	20
8	**1e**; $R_1 = 4$-Me, $R_2 = H$	A	22	**2e**	84	26
9	**1f**; $R_1 = 4$-*i*-Pr, $R_2 = H$	A	20	**2f**	83	24
10	**1g**; $R_1 = 3$-Cl, $R_2 = H$	A	22	**2g**	93	24
11	**1h**; $R_1 = H$, $R_2 = 2$-OMe	A	22	**2h**	70	34

[a]Isolated yield.

A = THF, B = THF/hexane (3/1, v/v).

THF–hexane (3:1) system, which gave an 89% chemical yield and a 33% ee (Table 4, entry 2), which was better than in the THF case. In the case of 4-isopropyl-*N*-(benzylidene)aniline, the allylation reaction in the THF/hexane solution produced 44% ee with a low chemical yield (Table 4, entry 6), whereas the allylation reaction in THF produced 24% ee with a high yield (Table 4, entry 5). Except in these cases, the use of the THF/hexane cosolvent system showed the chemical yield and ee results that were similar to or worse than when the THF solvent was used.

In conclusion, the synthesis of homoallyl amine from aldimine with allyl bromide in the presence of indium has presented excellent chemical yields ranging from 75% to 97%. The stereoselectivity in terms of the ee in the presence of a (+)-cinchonine chiral ligand turned out to be 22–44% ee. Further efforts to improve the ee, including pursuit of new chiral ligands and employment of other possible methods, are underway.

EXPERIMENTAL

Typical Procedure for Asymmetric Allylation

To a mixture of indium powder (344 mg, 3 mmol), (+)-chinchonine (294 mg, 1 mmol), and allyl bromide (260 mg, 3 mmol) in 2 ml of THF was added *N*-(benzylidene)aniline (0.5 mmol) in 1 mL THF at room temperature. The mixture was then stirred for 17–26 h, quenched with water, and extracted with CH_2Cl_2 (3×20 mL). The combined organic layer was washed with brine solution and then dried over $MgSO_4$, filtered, and concentrated. The residue was chromatographed over silica gel (hexane/EtOAc, 99:1) to give the allylated product. The product's ee was determined with HPLC with Welk-O chiral column.

N-Phenyl-α-2-propenylbenzenemethanamine (2a)

Liquid, ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.32–2.54 (m, 2H), 4.05 (bd s, 1H), 4.27 (dd, 1H, $J = 5.1$, 8.0 Hz), 5.02–5.11 (m, 2H), 5.57–5.73 (m, 1H), 6.39 (d, 2H, $J = 8.0$ Hz), 6.51–6.57 (m, 1H), 6.94–7.00 (m, 2H), 7.08–7.15 (m, 1H) 7.16–7.28 (m, 4H); IR (KBr) 3450, 3068, 3055, 2924, 1571, 1267 cm^{-1}; GC-MS m/z (rel. intensity) 223 (3, M^+), 182 (100), 104 (16), 91 (4), 77 (19).

4-Chloro-*N*-phenyl-α-2-propenylbenzenemethanamine (2b)

Liquid, ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.42–2.57 (m, 2H), 4.18 (bd s, 1H), 4.34 (dd, 1H, $J = 5.1$, 8.1 Hz), 5.13–5.21 (m, 2H), 5.66–5.77 (m, 1H), 6.45 (d, 2H, $J = 7.5$ Hz), 6.63–6.68 (m, 1H), 7.04–7.10 (m, 2H), 7.29 (bd s, 4H); IR (KBr) 3419, 3081, 3059, 3020, 2981, 2913, 2846 cm^{-1}; GC-MS m/z (rel. intensity) 257 (3, M^+), 216 (100), 180 (7), 104 (16), 77 (25).

4-Ethoxy-*N*-phenyl-α-2-propenylbenzenemethanamine (2c)

White solid, mp 49–50°C, ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.30 (t, 3H, $J = 7.0$ Hz), 2.38–2.52 (m, 2H), 3.91 (q, 2H, $J = 7.0$ Hz), 4.15 (bd s, 1H), 4.24 (dd, 1H, $J = 5.5$, 7.5 Hz), 5.02–5.11 (m, 2H), 5.58–5.74 (m, 1H), 6.42 (d, 2H, $J = 7.9$ Hz), 6.53–6.59 (m, 1H), 6.76 (d, 2H, $J = 8.6$ Hz), 6.96–7.02 (m, 2H), 7.17 (d, 2H, $J = 8.6$ Hz); IR (KBr) 3416, 3081, 3055, 3021, 2982, 2928, 2878 cm^{-1}; GC-MS m/z (rel. intensity) 267 (1, M^+), 226 (100), 198 (18), 104 (7), 77 (6).

3-Bromo-*N*-phenyl-α-2-propenylbenzenemethanamine (2d)

Liquid, ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.39–2.63 (m, 2H), 4.13 (bd s, 1H), 4.32 (dd, 1H, $J = 5.0$, 8.1 Hz), 5.15–5.22 (m, 2H), 5.66–5.80 (m, 1H), 6.47 (d, 2H, $J = 7.7$ Hz), 6.64–6.70 (m, 1H), 7.06–7.38 (m, 5H), 7.52(s, 1H); IR (KBr) 3413, 3081, 3055, 3014, 2984, 2907, 2835 cm^{-1}; GC-MS m/z (rel. intensity) 301 (6, M^+), 260 (100), 180 (38), 104 (27), 77 (39).

4-Methyl-*N*-phenyl-α-2-propenylbenzenemethanamine (2e)

Liquid, ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.31 (s, 1H), 2.41–2.62 (m, 2H), 4.12 (bd s, 1H), 4.34 (dd, 1H, $J = 5.3$, 7.9 Hz), 5.11–5.22 (m, 2H), 5.68–5.82 (m, 1H), 6.48 (d, 2H, $J = 8.6$ Hz), 6.59–6.65 (m, 1H), 7.03–7.12 (m, 4H), 7.23 (d, 2H, $J = 8.0$ Hz); IR (KBr) 3420, 3054, 2985, 2919, 1263 cm^{-1}; GC-MS m/z (rel. intensity) 237 (3, M^+),196 (100), 104 (16), 77 (19).

4-(1-Methylethyl)-*N*-phenyl-α-2-propenylbenzenemethanamine (2f)

Liquid, ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.21 (d, 6H, $J = 7.0$ Hz), 2.47–2.57 (m, 2H), 2.83–2.89 (m, 1H), 4.34 (dd, 1H, $J = 5.3$, 7.9 Hz), 5.09–5.19 (m, 2H), 5.69–5.82 (m, 1H), 6.50 (d, 2H, $J = 8.6$ Hz), 6.60–6.65 (m, 1H), 7.03–7.27 (m, 6H); IR (KBr) 3412, 3081, 3059, 3015, 2966, 2928, 2867 cm^{-1}; GC-MS m/z (rel. intensity) 265 (1, M^+), 224 (100), 104 (10), 77 (9).

3-Chloro-*N*-phenyl-α-2-propenylbenzenemethanamine (2g)

Liquid, ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.42–2.65 (m, 2H), 4.12 (bd s, 1H), 4.33 (dd, 1H, $J = 5.3$, 7.9 Hz), 5.13–5.21 (m, 2H), 5.64–5.80 (m, 1H), 6.49 (d, 2H, $J = 7.7$ Hz), 6.65–6.71 (m, 1H), 7.05–7.12 (m, 2H), 7.17–7.26 (m, 3H), 7.35 (s, 1H); IR (KBr) 3419, 3081, 3059, 3020, 2981, 2913, 2846 cm^{-1}; GC-MS m/z (rel. intensity) 257 (2, M^+), 216 (100), 180 (5), 104 (12), 77 (21).

(2-Methoyphenyl-1-phenylbut-3-enyl)amine (2h)

Liquid, ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.52–2.62 (m, 2H), 3.87 (s, 3H), 4.37 (s, 1H), 4.79 (s, 1H), 5.10–5.21 (m, 2H), 5.71–5.85 (m, 1H), 6.28–6.31 (dd, 1H, $J = 1.7$, 6 Hz), 6.56–6.77 (m, 3H), 7.19–7.37 (m, 5H); IR (KBr) 3420, 3080, 2946, 1596, 1505, 1456, 1231 cm^{-1}; GC-MS m/z (rel. intensity) 253 (4, M^+), 212 (100), 196 (14), 120 (16), 91 (9), 77 (4).

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