Catalytic Asymmetric Carbalkoxyallylation of Imines Using Chiral Bis- π -allylpalladium Complex

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General information. Most commercially supplied chemicals were freshly distilled or recrystallized before use. Solvents were dried by standard procedures. Dry THF refers to commercial dehydrated THF. Commercially available imines were directly procured, while most were prepared by refluxing equimolar amounts of corresponding aldehyde and amine in toluene or benzene with Dean-Stark removal of water. They were then purified by either high vacuum distillation or recrystallization. The carbethoxyallylstannane **2c** was prepared following literature report.¹ The catalyst **4** was prepared following our earlier reports.² The ¹H NMR and ¹³C NMR were recorded at 400 MHz and 75 MHz respectively. The chemical shifts are reported in δ units relative to internal tetramethylsilane for ¹H NMR and CDCl₃ was used as internal standard (δ = 77.0) for ¹³C NMR. Mass spectra were recorded in EI mode. HPLC was performed using Daicel CHIRALCEL OD column (0.46 cm $\times$ 25 cm).

Typical experimental procedure: Synthesis of 3c:

The imine **1c** (119.64 mg, 0.5 mmol), catalyst **4** (14.56 mg, 0.025 mmol, 5 mol%) and carbethoxyallylstannane **2c** (302 mg, 0.75 mmol, 1.5 equivalent) were taken in a Wheaton microreactor vial (5 mL capacity) and THF (1.5 mL) and MeOH (16 μ L, 1 equivalent) were added. The vial was flushed with argon and stirred at room temperature for 144 h. After completion, water (2 mL) and EtOAc (4 mL) were added. The organic phase was separated and the aqueous phase extracted with EtOAc (2 $\times$ 5 mL). The combined organic extracts were dried (Na₂SO₄) and concentrated. Purification of the residue by silica gel column chromatography

using hexane:EtOAc (3:1) as eluent furnished the carbethoxyhomoallylamine **3c** (122 mg, 69%) as colorless oil.

TABLE: Catalytic asymmetric carbethoxyallylation of imines **1** with **2c** using catalyst **4**.^a

1a R = Ph, R¹ = Bn

1b R = naphthyl, R¹ = Bn

1c R = piperonyl, R¹ = Bn

1d R = Ph, R¹ = -CH₂C₆H₄OMe-4

1e R = cyclohexyl, R¹ = Bn

1f R = 4-MeOC₆H₄, R¹ = Bn

1g R = thiophenyl, R¹ = Bn

1h R = 2-MeOC₆H₄, R¹ = Bn

entry	imine	time (h)	product ^b	%yield	%ee ^c
1	1a	120		3a 81	82
2	1b	120		3b 69	88
3	1c	144		3c 69	84
4	1d	144		3d 63	86

5	1e	120		3e	63	60
6	1f	144		3f	70	89
7	1g	120		3g	77	69
8	1h	144		3h	59	80

^aAll reactions were carried out with **1** (0.5 mmol), **2c** (1.5 equiv.), **4** (5 mol%), MeOH (1 equiv.), THF (1.5 mL) at room temperature for the specified time. ^bAbsolute configuration (*R*)- was determined by converting **3d** to the known α -methylene- γ -lactam.³ ^cEnantiomeric excess was determined by HPLC.

Ethyl 2-[2-(benzylamino)-2-phenylethyl]acrylate, **3a**

Colorless oil; $[\alpha]_D^{22} = +21.3$ (*c* 1.0, CHCl₃); IR (neat) 3333, 3061, 3028, 2980, 2926, 1714, 1661, 1629, 1602, 1494, 1454, 1304, 1192, 1145, 1028, 948, 817, 760 cm⁻¹; ¹H NMR (CDCl₃) δ 7.37–7.22 (m, 10H), 6.13 (d, *J* = 1.2 Hz, 1H), 5.4 (d, *J* = 1.2 Hz, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 3.87–3.83 (m, 1H), 3.68 (d, *J* = 13.2 Hz, 1H), 3.52 (d, *J* = 13.2 Hz, 1H), 2.67–2.63 (m, 2H), 1.7 (brs, 1H), 1.27 (t, *J* = 7.2 Hz, 3H);

^{13}C NMR (CDCl_3) δ 166.8, 143.3, 140.4, 137.7, 128.5, 128.5, 128.3, 128.2, 128.1, 127.9, 127.2, 127.03, 127.00, 126.7, 126.6, 60.8, 60.6, 51.3, 41.3, 14.2;

MS (EI) m/z (relative intensity) 263 [$M^+ - C_2H_5OH$] (2.1), 236 (4.0), 203 (3.6), 196 (40.7), 172 (31.3), 129 (5.5), 91 (100); Anal. Calcd for $C_{20}H_{23}NO_2$ (309.41): C, 77.64; H, 7.49; N, 4.52. Found: C, 77.34; H, 7.58; N, 4.68. The enantiomeric excess was determined by converting to trifluoroacetamide form and using CHIRALCEL OD column, hexane/*i*-PrOH = 300/1, flow rate = 1 mL/min, UV detection at 254 nm, t_R = 12.71 min (major enantiomer), t_R = 17.28 min (minor enantiomer); 82% ee.

Ethyl 2-[2-(benzylamino)-2-(2-naphthyl)ethyl]acrylate, 3b

Colorless oil; $[\alpha]_{\text{D}}^{22} = +18.0$ (c 1.0, CHCl_3); IR (neat) 3332, 3057, 3026, 2979, 2926, 1713, 1630, 1601, 1508, 1495, 1453, 1370, 1314, 1244, 1194, 1144, 1028, 950, 858, 820, 747 cm^{-1} ; ^1H NMR (CDCl_3) δ 7.83–7.79 (m, 4H), 7.53–7.43 (m, 3H), 7.29–7.21 (m, 5H), 6.11 (d, $J = 1.0$ Hz, 1H), 5.41 (d, $J = 1.0$ Hz, 1H), 4.14 (q, $J = 7.0$ Hz, 2H), 4.02–4.01 (m, 1H), 3.66 (d, $J = 13.2$ Hz, 1H), 3.54 (d, $J = 13.2$ Hz, 1H), 2.73–2.70 (m, 2H), 1.75 (brs, 1H), 1.25 (t, $J = 7.0$ Hz, 3H);

^{13}C NMR (CDCl_3) δ 166.8, 140.7, 140.4, 137.6, 133.3, 132.8, 128.6, 128.2, 128.1, 127.9, 127.7, 127.5, 127.1, 126.7, 126.1, 126.0, 125.8, 125.4, 125.1, 60.9, 60.7, 51.4, 41.2, 14.2;

MS (EI) m/z (relative intensity) 313 [$\text{M}^+ - \text{C}_2\text{H}_5\text{OH}$] (6.6), 246 (100), 222 (11.5), 194 (6.7), 179 (14.3), 165 (6.4), 141 (5.4), 91 (72.1); Anal. Calcd for $\text{C}_{24}\text{H}_{25}\text{NO}_2$ (359.46): C, 80.19; H, 7.01; N, 3.89. Found: C, 79.82; H, 7.28; N, 3.68. The enantiomeric excess was determined by converting to trifluoroacetyl amide form and using CHIRALCEL OD column, hexane/*i*-PrOH = 300/1, flow rate = 1 mL/min, UV detection at 254 nm, t_R = 18.17 min (major enantiomer), t_R = 28.18 min (minor enantiomer); 88% ee.

Ethyl 2-[2-(benzylamino)-2-piperonylethyl]acrylate, 3c

Colorless oil; $[\alpha]_D^{22} = +27.7$ (c 1.0, CHCl_3); IR (neat) 3336, 3063, 3030, 2904, 1715, 1630, 1595, 1503, 1488, 1446, 1417, 1387, 1322, 1281, 1246, 1167, 1098, 1079, 1038, 930, 861, 732 cm^{-1} ; ^1H NMR (CDCl_3) δ 7.31–7.21 (m, 5H), 6.9 (s, 1H), 6.77–6.75 (m, 2H), 6.13 (d, $J = 1.2$ Hz, 1H), 5.97 (s, 2H), 5.41 (d, $J = 1.2$ Hz, 1H), 4.18 (q, $J = 7.2$ Hz, 2H), 3.79–3.76 (m, 1H), 3.68 (d, $J = 13.4$ Hz, 1H), 3.51 (d, $J = 13.4$ Hz, 1H), 2.60–2.59 (m, 2H), 1.7 (brs, 1H), 1.25 (t, $J = 7.2$ Hz, 3H);

^{13}C NMR (CDCl_3) δ 167.8, 148.2, 147.4, 138.6, 136.0, 134.3, 128.5, 128.43, 128.41, 128.0, 127.4, 120.3, 116.1, 108.2, 106.3, 101.1, 58.1, 57.8, 44.5, 34.6, 14.6;

MS (EI) m/z (relative intensity) 307 [$\text{M}^+ - \text{C}_2\text{H}_5\text{OH}$] (38.9), 262 (13.4), 216 (98.1), 202 (20.9), 186 (21.1), 173 (66.0), 158 (32.9), 148 (19.2), 144 (100), 135 (25.0), 116 (22.3), 91 (24.0); Anal. Calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_4$ (353.42): C, 71.36; H, 6.56; N, 3.96. Found: C, 71.28; H, 6.88; N, 3.68. The enantiomeric excess was determined by converting to trifluoroacetyl amide form and using CHIRALCEL OD column, hexane/*i*-PrOH = 100/1, flow rate = 0.8 mL/min, UV detection at 254 nm, t_R = 18.35 min (major enantiomer), t_R = 24.27 min (minor enantiomer); 84% ee.

Ethyl 2-[2-(4-methoxybenzylamino)-2-phenylethyl]acrylate, 3d

Colorless oil; $[\alpha]_{\text{D}}^{22} = +13.8$ (c 1.0, CHCl₃); IR (neat) 3329, 3068, 3028, 2931, 2836, 1712, 1635, 1611, 1513, 1439, 1412, 1303, 1281, 1248, 1176, 1110, 1032, 924, 820, 760, 703 cm⁻¹; ¹H NMR (CDCl₃) δ 7.36–7.28 (m, 4H), 7.27–7.22 (m, 1H), 7.04 (d, $J = 8.4$ Hz, 2H), 6.84 (d, $J = 8.4$ Hz, 2H), 6.12 (d, $J = 1.6$ Hz, 1H), 5.4 (d, $J = 1.6$ Hz, 1H), 4.18 (q, $J = 7.2$ Hz, 2H), 3.85–3.81 (m, 1H), 3.78 (s, 3H), 3.62 (d, $J = 13.2$ Hz, 1H), 3.46 (d, $J = 13.2$ Hz, 1H), 2.67–2.63 (m, 2H), 1.67 (brs, 1H), 1.26 (t, $J = 7.2$ Hz, 3H);

^{13}C NMR (CDCl_3) δ 166.9, 158.3, 143.4, 140.7, 138.9, 137.7, 129.9, 129.1, 128.9, 128.1, 127.2, 127.0, 126.6, 116.0, 113.6, 60.8, 55.2, 50.7, 41.3, 34.7, 14.2;

MS (EI) m/z (relative intensity) 293 [$M^+ - C_2H_5OH$] (21.2), 264 (8.1), 249 (5.2), 226 (15.8), 202 (11.6), 185 (39.5), 172 (100), 131 (8.5), 121 (91.0), 91 (30.4); Anal. Calcd for $C_{21}H_{25}NO_3$ (339.43): C, 74.31; H, 7.42; N, 4.12. Found: C, 74.39; H, 7.68; N, 3.88. The enantiomeric excess was determined by converting to trifluoroacetamide form and using CHIRALCEL OD column, hexane/*i*-PrOH = 300/1, flow rate = 1 mL/min, UV detection at 254 nm, t_R = 19.5 min (major enantiomer), t_R = 27.93 min (minor enantiomer); 86% ee.

Ethyl 2-[2-(benzylamino)-2-cyclohexylethyl]acrylate, 3e

Colorless oil; $[\alpha]_D^{22} = -11.2$ (c 1.0, CHCl_3); IR (neat) 3328, 3063, 3030, 2904, 1712, 1633, 1603, 1503, 1488, 1446, 1417, 1387, 1322, 1281, 1246, 1167, 1098, 1079, 1038, 930, 861, 732 cm^{-1} ; ^1H NMR (CDCl_3) δ 7.27–7.15 (m, 5H), 6.13 (d, $J = 1.6$ Hz, 1H), 5.49 (d, $J = 1.6$ Hz, 1H), 4.13 (q, $J = 7.2$ Hz, 2H), 3.74 (d, $J = 13.2$ Hz, 1H), 3.67 (d, $J = 13.2$ Hz, 1H), 2.54–2.46 (m, 2H), 2.26–2.20 (m, 1H), 1.73–1.63 (m, 5H), 1.50–1.43 (m, 1H), 1.24 (t, $J = 7.2$ Hz, 3H), 1.23–1.02 (m, 6H);

^{13}C NMR (CDCl_3) δ 167.1, 139.7, 136.4, 128.5, 128.1, 128.09, 127.4, 126.6, 115.0, 60.6, 51.8, 40.5, 34.1, 29.0, 26.7, 26.5, 26.2, 25.7, 24.1, 14.2;

MS (EI) m/z (relative intensity) 269 [$M^+ - C_2H_5OH$] (15.0), 232 (7.4), 215 (2.0), 202 (7.7), 186 (100), 158 (1.7), 110 (4.1), 91 (24.4); Anal. Calcd for $C_{20}H_{29}NO_2$ (315.45): C, 76.15; H, 9.26; N, 4.44. Found: C, 75.91; H, 9.49; N, 4.68. The enantiomeric excess was determined by converting to trifluoroacetyl amide form and using CHIRALCEL OD column, hexane/*i*-PrOH = 400/1, flow rate = 0.8 mL/min, UV detection at 254 nm, t_R = 15.23 min (major enantiomer), t_R = 20.56 min (minor enantiomer); 60% ee.

Ethyl 2-[2-(benzylamino)-2-(4-methoxyphenyl)ethyl]acrylate, 3f

Colorless oil; $[\alpha]_D^{22} = +22.6$ (c 1.0, CHCl_3); IR (neat) 3330, 3061, 3027, 2980, 2933, 2835, 1714, 1628, 1610, 1585, 1512, 1455, 1369, 1301, 1246, 1192, 1146, 1033, 949, 831, 736 cm^{-1} ; ^1H NMR (CDCl_3) δ 7.31–7.19 (m, 7H), 6.88 (d, $J = 8.2$ Hz, 2H), 6.12 (d, $J = 1.4$ Hz, 1H), 5.39 (d, $J = 1.4$ Hz, 1H), 4.17 (q, $J = 7.2$ Hz, 2H), 3.83–3.80 (m, 1H), 3.78 (s, 3H), 3.67 (d, $J = 13.2$ Hz, 1H), 3.51 (d, $J = 13.2$ Hz, 1H), 2.63–2.59 (m, 2H), 1.73 (brs, 1H), 1.26 (t, $J = 7.2$ Hz, 3H);

^{13}C NMR (CDCl_3) δ 166.8, 158.5, 140.4, 137.7, 135.2, 128.5, 128.4, 128.2, 128.1, 127.9, 127.8, 126.9, 126.8, 126.6, 113.6, 60.6, 60.2, 55.1, 51.2, 41.3, 14.2;

MS (EI) m/z (relative intensity) 293 [$M^+ - C_2H_5OH$] (20.3), 249 (11.3), 226 (60.0), 202 (47.7), 188 (15.8), 174 (16.7), 159 (41.2), 145 (13.8), 121 (17.2), 91 (100); Anal. Calcd for $C_{21}H_{25}NO_3$ (339.43): C, 74.31; H, 7.42; N, 4.12. Found: C, 74.59; H, 7.28; N, 3.86. The enantiomeric excess was determined by converting to trifluoroacetylamide form and using CHIRALCEL OD column, hexane/*i*-PrOH = 400/1, flow rate = 0.8 mL/min, UV detection at 254 nm, t_R = 23.07 min (major enantiomer), t_R = 28.02 min (minor enantiomer); 89% ee.

Ethyl 2-[2-(benzylamino)-2-(2-thiophenyl)ethyl]acrylate, 3g

Colorless oil; $[\alpha]_D^{22} = +22.6$ (c 1.0, CHCl_3); IR (neat) 3328, 3063, 3028, 2980, 2835, 1714, 1629, 1602, 1494, 1454, 1369, 1313, 1198, 1028, 949, 850, 819 cm^{-1} ; ^1H NMR (CDCl_3) δ 7.34–7.22 (m, 6H), 6.97–6.88 (m, 2H), 6.16 (d, $J = 1.6$ Hz, 1H), 5.44 (d, $J = 1.6$ Hz, 1H), 4.20–4.14 (m, 1H), 4.16 (q, $J = 7.2$ Hz, 2H), 3.81 (d, $J = 13.2$ Hz, 1H), 3.61 (d, $J = 13.2$ Hz, 1H), 2.76–2.67 (m, 2H), 1.73 (brs, 1H), 1.25 (t, $J = 7.2$ Hz, 3H);

^{13}C NMR (CDCl_3) δ 166.6, 148.4, 140.0, 137.2, 128.4, 128.1, 127.9, 127.6, 127.4, 126.7, 126.2, 124.4, 123.9, 60.6, 56.5, 51.2, 42.0, 14.1;

MS (EI) m/z (relative intensity) 269 [$\text{M}^+ - \text{C}_2\text{H}_5\text{OH}$] (18.2), 242 (5.3), 202 (56.4), 106 (3.8), 91 (100); Anal. Calcd for $\text{C}_{18}\text{H}_{21}\text{NO}_2\text{S}$ (315.43): C, 68.54; H, 6.71; N, 4.44; S, 10.16. Found: C, 68.89; H, 6.48; N, 4.23; S, 10.01. The enantiomeric excess was determined by converting to trifluoroacetamide form and using CHIRALCEL OD column, hexane/*i*-PrOH = 300/1, flow rate = 1 mL/min, UV detection at 254 nm, t_R = 19.42 min (major enantiomer), t_R = 25.38 min (minor enantiomer); 69% ee.

Ethyl 2-[2-(benzylamino)-2-(2-methoxyphenyl)ethyl]acrylate, 3h

Colorless oil; $[\alpha]_D^{22} = +14.7$ (c 1.0, CHCl_3); IR (neat) 3337, 3158, 3010, 2935, 2836, 1715, 1630, 1600, 1491, 1464, 1368, 1303, 1240, 1189, 1146, 1106, 1029, 942, 817, 754 cm^{-1} ; ^1H NMR (CDCl_3) δ 7.26–7.12 (m, 7H), 6.89–6.77 (m, 2H), 5.98 (d, $J = 1.6$ Hz, 1H), 5.25 (d, $J = 1.6$ Hz, 1H), 4.18–4.14 (m, 1H), 4.10 (q, $J = 7.6$ Hz, 2H), 3.71 (s, 3H), 3.60 (d, $J = 13.2$ Hz, 1H), 3.48 (d, $J = 13.2$ Hz, 1H), 2.74–2.69 (m, 1H), 2.59–2.53 (m, 1H), 1.81 (brs, 1H), 1.18 (t, $J = 7.2$ Hz, 3H);

^{13}C NMR (CDCl_3) δ 167.2, 157.4, 140.7, 138.3, 130.8, 128.5, 128.3, 128.0, 127.9, 127.7, 127.3, 126.5, 126.1, 120.5, 110.3, 60.5, 55.2, 55.1, 51.5, 39.1, 14.2;

MS (EI) m/z (relative intensity) 293 [$\text{M}^+ - \text{C}_2\text{H}_5\text{OH}$] (22.3), 249 (21.3), 226 (70.9), 188 (5.3), 174 (31.9), 145 (15.3), 121 (7.2), 91 (100); Anal. Calcd for $\text{C}_{21}\text{H}_{25}\text{NO}_3$ (339.43): C, 74.31; H, 7.42; N, 4.12. Found: C, 74.45; H, 7.24; N, 4.04. The enantiomeric excess was determined by converting to trifluoroacetylamide form and using CHIRALCEL OD column, hexane/*i*-PrOH = 100/1, flow rate = 0.8 mL/min, UV detection at 254 nm, t_R = 15.24 min (major enantiomer), t_R = 17.66 min (minor enantiomer); 80% ee.

1-(4-Methoxybenzyl)-3-methylene-5-phenyl-2-pyrrolidinone, 8

To a solution of **3d** (0.3 g, 0.883 mmol) in toluene (4 mL) was added *p*-TsOH (catalytic) and stirred at 60°C for 8 h. Toluene was evaporated on rotavapor and the residue was purified by silica gel column chromatography using hexane:EtOAc (4:1) to give **8** (0.22 g, 85%) as colorless oil. $[\alpha]_D^{22} = -131.8$ (*c* 1.0, CHCl₃); ¹H NMR (CDCl₃) δ 7.39–7.32 (m, 3H), 7.25–7.11 (m, 2H), 7.02 (d, *J* = 8.2 Hz, 2H), 6.82 (d, *J* = 8.2 Hz, 2H), 6.13–6.12 (m, 1H), 5.38–5.37 (m, 1H), 5.14–5.1 (m, 1H), 4.39 (dd, *J* = 8.8, 3.6 Hz, 1H), 3.79 (s, 3H), 3.49 (d, *J* = 14.4 Hz, 1H), 2.64 (ddt, *J* = 17.2, 8.8, 2.6 Hz, 1H), 2.65–2.59 (m, 1H);

¹³C NMR (CDCl₃) δ 168.0, 159.0, 140.7, 138.9, 129.9, 129.0, 128.2, 128.1, 126.6, 116.0, 113.9, 57.9, 55.2, 44.0, 34.6;

MS (EI) m/z (relative intensity) 293 [M^+] (22.6), 172 (56.3), 121 (100); Anal. Calcd for $C_{19}H_{19}NO_2$ (293.36): C, 77.79; H, 6.52; N, 4.77. Found: C, 77.56; H, 6.58; N, 4.64.

3-methylene-5-phenyl-2-pyrrolidinone, **9**

To a solution of **8** (0.2 g, 0.681 mmol) in CH_2Cl_2 (4 mL) was added H_2O (1 mL) and cooled to $0^\circ C$. DDQ^4 (0.232 g, 1.022 mmol) was added in portions over 30 min. The reaction mixture was stirred at room temperature for 48 h. It was then diluted with CH_2Cl_2 (15 mL) and quenched with sat. aq. $NaHCO_3$ (5 mL). The organic layer was separated and washed with sat. aq. $NaHCO_3$ (1 ×

5 mL), dried (Na₂SO₄) and concentrated. Purification of the residue by silica gel column chromatography using hexane:EtOAc (4:1) as eluent gave the unreacted starting material (54 mg). Further elution with hexane:EtOAc (2:1) gave **9** (0.41 g, 48% based on recovered starting material) as a colorless solid; m.p. 188–189°C; $[\alpha]_D^{22} = -14.9$ (c 0.45, CHCl₃); ¹H NMR (CDCl₃) δ 7.39–7.24 (m, 6H), 6.14 (t, *J* = 2.8 Hz, 1H), 5.39 (t, *J* = 2.8 Hz, 1H), 4.4 (dd, *J* = 8.48, 4.0 Hz, 1H), 3.16–3.09 (m, 1H), 2.66–2.60 (m, 1H);

¹³C NMR (CDCl₃) δ 168.0, 140.7, 138.9, 129.9, 129.0, 128.2, 128.1, 126.6, 55.2, 34.6;

MS (EI) m/z (relative intensity) 173 [M^+] (100), 96 (23.3), 77 (10.9); Anal. Calcd for $C_{11}H_{11}NO$ (173.21): C, 76.27; H, 6.40; N, 8.08. Found: C, 76.51; H, 6.28; N, 7.88.

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