Pd-Catalyzed Asymmetric Allylic Alkylation of Indoles and Pyrroles by Chiral Alkene-Phosphine Ligands

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Representative procedure for the synthesis of terminal-alkene-phosphine hybrid ligand 2a (Scheme 1)



A single necked flask charged with a solution of (*S*)-BINOL (**3**) (14.3 g, 50 mmol) and Et₃N (15.2 g, 20.9 mL, 150 mmol) in CH₂Cl₂ (125 mL) was cooled to -78 °C, and Tf₂O (31.0 g, 18.5 mL, 110 mmol) was added dropwise. Then the resulting mixture was warmed to 0 °C for 2 h. After removal of the solvent under vacuum, the resulting thick residue was filtrated through a short silica gel column and flushed with petroleum ether/ethyl acetate (5:1, v/v). The filtrate was concentrated under reduce pressure, and the product **4** was obtained as a white solid (27.5 g, >99% yield) and used for the next step without further purification.

Ooi, T.; Kameda, M.; Maruoka, K. J. Am. Chem. Soc. 2003, 125, 5139.

To a schlenk flask charged with **4** (1.10 g, 2.0 mmol), diphenylphosphine oxide (0.808 g, 4 mmol), $Pd(OAc)_2$ (0.0224 g, 0.1 mmol) and dppb (0.0427 g, 0.1 mmol) in DMSO (8.8 mL) was added DIEA (1.03 g, 1.4 mL, 8.0 mmol) under argon. The resulting mixture was stirred at 100 °C for 12 h. Then the mixture was cooled to room temperature, diluted with EtOAc (50 mL), washed with water (20 mL x 3), brine (20 mL), successively. The organic phase was dried over anhydrous Na₂SO₄, filtered, concentrated, and the crude residue was further purified by flash chromatography on silica gel (petroleum ether/ethyl acetate = 1:1, v/v) to afford the desired product **5** as a white solid (1.08 g, 90% yield).

Uozumi, Y.; Tanahashi, A.; Lee, S.-Y.; Hayashi, T. J. Org. Chem. 1993, 58, 1945.

To a dried sealed tube charged with 5 (0.4876 g, 0.81 mmol) in dry toluene (20 mL), Et_3N (0.5727 g, 0.79 mL, 5.67 mmol) and $HSiCl_3$ (0.5488 g, 0.41 mL, 4.05 mmol) were added

successively under argon at 0 °C. The resulting mixture was stirring at 100 °C for 12 h. After cooled to 0 °C, diluted by Et₂O (10 mL), quenched with several drops of saturated Na₂CO₃ solution, the mixture was filtered by a short celite column, and washed with Et₂O (10 mL x 3). The filtrate was dried over anhydrous Na₂SO₄, filtered, concentrated, and the residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate = 20:1, v/v) to afford the desired product **6** as a white solid (0.394 g, 83% yield).

Ref: Uozumi, Y.; Tanahashi, A.; Lee, S.-Y.; Hayashi, T. J. Org. Chem. 1993, 58, 1945.

To a dried schlenk tube charged with **6** (0.0586 g, 0.1 mmol), potassium vinyltrifluoroborate (0.0161 g, 0.12 mmol), and Pd(PPh₃)₄ (0.0046 mg, 0.004 mmol), degassed Et₃N (0.0162 mg, 0.022 mL, 0.16 mmol) and dioxane (2 mL) was added sequentially. The mixture was heated to 104 °C for 3 h (detected by TLC). After cooling to ambient temperature, the mixture was concentrated and purified by flash chromatography on silica gel (petroleum ether/ethyl ether = 50:1, v/v) to afford the desired product **2a** as a white solid (0.0288 g, 62% yield).

Representative procedure for 2a/Pd-catalyzed asymmetric allylic alkylation of indoles. To a dried Schlenk flask charged with $[PdCl(C_3H_5)]_2$ (0.0011 g, 0.003 mmol, 3 mol % Pd), chiral phosphine-alkene ligand **2a** (0.0028 g, 0.006 mmol, 3 mol %), was added distilled CH₂Cl₂ (0.4 mL) under argon. Then the mixture was stirred for 30 minutes at room temperature before cooled to 0 °C. 1,3-Diphenyl-2-propenyl acetate **8** (0.0605 g, 0.24 mmol) in CH₂Cl₂ (0.40 mL), indole **7a** (0.0234 g, 0.2 mmol) and sodium carbonate (0.0424 g, 0.4 mmol), CH₂Cl₂ (0.2 mL) were added sequentially. After stirring at 0 °C for 24 h, the reaction mixture was quenched by water (1 mL) and extracted with CH₂Cl₂ (3 mL x 3). The solvent was removed under reduced pressure and crude residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate = 20:1, v/v) to afford the desired product **9a** as a yellow solid (0.059 g, 95% yield).



2a, white foam solid, m.p. 119-121 °C, $[\alpha]^{20}_{D}$ -90.0 (*c* 0.2, CH₂Cl₂). IR (film) 1478, 1434 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, *J* = 8.8 Hz, 1H), 7.90 (d, *J* = 8.4 Hz, 2H), 7.85 (d, *J* = 8.4 Hz, 2H), 7.50-7.45 (m, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.27-7.12 (m, 10H), 7.06 (dd, *J* = 7.2, 6.8 Hz, 2H), 7.50-7.45 (m, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.27-7.12 (m, 10H), 7.06 (dd, *J* = 7.2, 6.8 Hz, 2H), 7.50-7.45 (m, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.27-7.12 (m, 10H), 7.06 (dd, *J* = 7.2, 6.8 Hz, 2H), 7.50-7.45 (m, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.27-7.12 (m, 10H), 7.06 (dd, *J* = 7.2, 6.8 Hz, 2H), 7.50-7.45 (m, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.27-7.12 (m, 10H), 7.06 (dd, *J* = 7.2, 6.8 Hz, 2H), 7.50-7.45 (m, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.27-7.12 (m, 10H), 7.06 (dd, *J* = 7.2, 6.8 Hz, 2H), 7.50-7.45 (m, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.27-7.12 (m, 10H), 7.06 (dd, *J* = 7.2, 6.8 Hz, 2H), 7.50-7.45 (m, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.27-7.12 (m, 10H), 7.06 (dd, *J* = 7.2, 6.8 Hz, 2H), 7.50-7.45 (m, 2H), 7.50-7.45 (m, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.27-7.12 (m, 10H), 7.06 (dd, *J* = 7.2, 6.8 Hz, 2H), 7.50-7.45 (m, 2H), 7.50-7.45 (m, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.27-7.12 (m, 10H), 7.06 (dd, *J* = 7.2, 6.8 Hz, 2H), 7.50-7.45 (m, 2H), 7.50-7.50 (m, 2H), 7.50-7.5

2H), 7.01 (dd, J = 8.4, 6.8 Hz, 1H), 6.88 (d, J = 8.4 Hz, 1H), 6.15 (dd, J = 17.6, 11.2 Hz, 1H), 5.64 (d, J = 17.6 Hz, 1H), 4.87 (d, J = 11.2 Hz, 1H); ³¹P NMR (121 MHz, CDCl₃) δ -13.9; HRMS (EI): Calcd. for C₃₄H₂₅P (M): 464.1694; Found: 464.1699.



2b, white foam solid, m.p. 53-55 °C, $[\alpha]^{20}_{D}$ -29.6 (*c* 0.25, CH₂Cl₂). IR (film) 1456 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.94 (d, *J* = 8.8 Hz, 1H), 7.91-7.82 (m, 4H), 7.54 (dd, *J* = 8.4, 2.8 Hz, 1H), 7.47 (dd, *J* = 7.6, 7.2 Hz, 1H), 7.35-7.29 (m, 1H), 7.26-7.20 (m, 1H), 7.15 (d, *J* = 8.4 Hz, 1H), 7.01 (dd, *J* = 8.0, 7.2 Hz, 1H), 6.89-6.82 (m, 4H), 6.80 (s, 1H), 6.68 (d, *J* = 8.0 Hz, 2H), 6.22 (dd, *J* = 17.6, 11.2 Hz, 1H), 5.68 (d, *J* = 17.6 Hz, 1H), 4.95 (d, *J* = 11.2 Hz, 1H), 2.21 (s, 6H), 2.13 (s, 6H); ³¹P NMR (121 MHz, CDCl₃) δ -12.8; HRMS (EI): Calcd. for C₃₈H₃₃P (M): 520.2320; Found: 520.2325.



2c, white foam solid, m.p. 95-97 °C, $[\alpha]^{20}_{D}$ -82.7 (*c* 0.19, CH₂Cl₂). IR (film) 1499 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, J = 8.8 Hz, 1H), 7.93-7.80 (m, 5H), 7.77 (d, J = 6.8 Hz, 2H), 7.70-7.65 (m, 3H), 7.61 (dd, J = 8.4, 2.4 Hz, 1H), 7.55 (d, J = 7.6 Hz, 1H), 7.53-7.37 (m, 6H), 7.37-7.19 (m, 5H), 6.91-6.83 (m, 2H), 6.23 (dd, J = 17.6, 10.8 Hz, 1H), 5.60 (d, J = 17.6 Hz, 1H), 4.82 (d, J = 10.8 Hz, 1H); ³¹P NMR (121 MHz, CDCl₃) δ -12.4; HRMS (EI): Calcd. for C₄₂H₂₉P (M): 564.2007; Found: 564.2014.



2d, white foam solid, m.p. 97-99 °C, $[\alpha]^{20}_{D}$ +40.9 (*c* 0.43, CH₂Cl₂). IR (film) 1479, 1434 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.01-7.86 (m, 5H), 7.52-7.46 (m, 2H), 7.37 (t, *J* = 7.2 Hz, 1H), 7.27-7.12 (m, 10H), 7.12-7.06 (m, 6H), 7.06-6.94 (m, 4H), 6.52 (d, *J* = 16.4 Hz, 1H); ³¹P NMR

(121 MHz, CDCl₃) δ -14.0; HRMS (FT-ICRMS): Calcd. for C₄₀H₂₉P (M): 540.2007; Found: 540.2001.



2e, white foam solid, m.p. 50-52 °C, $[\alpha]^{20}_{D}$ -46.0 (*c* 0.2, CH₂Cl₂). IR (film) 1477, 1456 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.37 (d, *J* = 8.0 Hz, 1H), 7.27-7.19 (m, 8H), 7.16-7.11 (m, 2H), 7.07 (d, *J* = 8.0 Hz, 1H), 7.04-6.99 (m, 2H), 6.00 (dd, *J* = 17.6, 10.8 Hz, 1H), 5.41 (d, *J* = 17.6 Hz, 1H), 4.78 (d, *J* = 10.8 Hz, 1H), 2.81 (dd, *J* = 6.4, 5.6 Hz, 2H), 2.75 (dd, *J* = 6.4, 6.0 Hz, 2H), 2.21-2.03 (m, 2H), 1.93 (dd, *J* = 6.4, 6.0 Hz, 2H), 1.73-1.50 (m, 6H), 1.50-1.19 (m, 2H); ³¹P NMR (121 MHz, CDCl₃) δ -15.0; HRMS (FT-ICRMS): Calcd. for C₃₄H₃₃P (M): 472.2320; Found: 472.2312.



9a, yellow solid, $[\alpha]^{20}_{D}$ +38.5 (*c* 1.90, CH₂Cl₂) (92% ee) [lit.: $[\alpha]^{20}_{D}$ -45.8 (*c* 0.98, CH₂Cl₂) (95% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 7.96 (brs, 1H), 7.42 (d, *J* = 7.6 Hz, 1H), 7.38-7.14 (m, 12H), 7.02 (dd, *J* = 7.6, 7.2 Hz, 1H), 6.90 (s, 1H), 6.72 (dd, *J* = 16.0, 7.2 Hz, 1H), 6.44 (d, *J* = 16.0 Hz, 1H), 5.12 (d, *J* = 7.2 Hz, 1H) ; ¹³C NMR (100 MHz, CDCl₃) δ 143.6, 137.7, 136.9, 132.8, 130.8, 128.7, 128.6, 127.4, 127.0, 126.6, 126.5, 122.8, 122.3, 120.1, 119.6, 118.9, 111.3, 46.4. Cheung, H. Y.; Yu, W.-Y.; Lam, F. L.; Au-Yeung, T. T.-L.; Zhou, Z.; Chan, T. H.; Chan, A. S. C. *Org. Lett.* **2007**, 9, 4295.

Table 2, entry 2



9b, yellow solid, $[\alpha]^{20}{}_{D}$ +75.3 (*c* 1.05, CH₂Cl₂) (82% ee) [lit.: $[\alpha]^{20}{}_{D}$ -71.9 (*c* 1.0, CH₂Cl₂) (92% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 8.06 (brs, 1H), 7.59 (d, *J* = 7.2 Hz, 2H), 7.54-7.38 (m, 9H), 7.38-7.30 (m, 4H), 7.30-7.21 (m, 3H), 7.07 (dd, *J* = 7.6, 7.2 Hz, 1H), 6.97 (dd, *J* = 16.0, 7.2 Hz, 2H)

1H), 6.48 (d, J = 16.0 Hz, 1H), 5.36 (d, J = 7.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 143.7, 137.7, 136.4, 135.8, 133.1, 132.5, 131.3, 129.0, 128.8, 128.7, 128.5, 128.4, 128.2, 128.1, 127.3, 126.5, 126.3, 122.3, 121.4, 119.9, 114.0, 111.2, 45.3.

Cheung, H. Y.; Yu, W.-Y.; Lam, F. L.; Au-Yeung, T. T.-L.; Zhou, Z.; Chan, T. H.; Chan, A. S. C. *Org. Lett.* **2007**, 9, 4295.

Table 2, entry 3



9c, white powder, m.p. 171-173 °C, $[\alpha]^{20}_{D}$ +68.4 (*c* 0.31, CH₂Cl₂) (94% ee). IR (film) 3419 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.96 (brs, 1H), 7.38 (d, *J* = 7.2 Hz, 2H), 7.37-7.27 (m, 6H), 7.27-7.16 (m, 3H), 7.11 (dd, *J* = 8.0, 7.2 Hz, 1H), 6.87-6.83 (m, 2H), 6.78 (dd, *J* = 16.0, 6.4 Hz, 1H), 6.28 (d, *J* = 16.0 Hz, 1H), 5.49 (d, *J* = 6.4 Hz, 1H), 2.57 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 144.3, 137.8, 137.2, 134.1, 131.3, 130.9, 129.1, 128.7, 128.6, 127.3, 126.5, 125.8, 123.7, 122.4, 121.5, 119.2, 109.2, 46.7, 20.6. HRMS (FT-ICRMS): Calcd. for C₂₄H₂₁N (M): 323.1674; Found: 323.1663.

Table 2, entry 4

9d, yellow oil, $[\alpha]^{20}{}_{D}$ +38.1 (*c* 1.35, CH₂Cl₂) (92% ee) [lit.: $[\alpha]^{20}{}_{D}$ -48.3 (*c* 1.01, CH₂Cl₂) (96% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 7.81 (brs, 1H), 7.37-7.16 (m, 12H), 6.99 (d, *J* = 8.4 Hz, 1H), 6.82 (d, *J* = 2.0 Hz, 1H), 6.71 (dd, *J* = 16.0, 7.2 Hz, 1H), 6.41 (d, *J* = 16.0 Hz, 1H), 5.09 (d, *J* = 7.2 Hz, 1H), 2.36 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 143.7, 137.8, 135.2, 132.9, 130.7, 128.9, 128.7, 128.6, 128.5, 127.3, 126.5, 124.0, 123.0, 119.6, 118.4, 111.0, 46.3, 21.7. Cheung, H. Y.; Yu, W.-Y.; Lam, F. L.; Au-Yeung, T. T.-L.; Zhou, Z.; Chan, T. H.; Chan, A. S. C. *Org. Lett.* **2007**, 9, 4295.

Table 2, entry 5

9e, yellow oil, $[\alpha]^{20}{}_{D}$ +32.1 (*c* 0.38, CH₂Cl₂) (90% ee) [lit.: $[\alpha]^{20}{}_{D}$ -38.5 (*c* 0.96, CH₂Cl₂) (94% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 7.90 (brs, 1H), 7.44-7.17 (m, 11H), 6.89-6.82 (m, 3H), 6.72 (dd, *J* = 15.6, 7.6 Hz, 1H), 6.45 (d, *J* = 15.6 Hz, 1H), 5.08 (d, *J* = 7.6 Hz, 1H), 3.72 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 154.0, 143.5, 137.7, 132.7, 132.0, 130.7, 128.7, 128.6, 128.5, 127.4, 127.3, 126.6, 126.5, 123.7, 118.5, 112.3, 112.0, 102.0, 56.0, 46.4.

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Table 2, entry 6



9f, yellow oil, $[\alpha]^{20}{}_{D}$ +30.5 (*c* 0.2, CH₂Cl₂) (90% ee) [lit.: $[\alpha]^{20}{}_{D}$ -30.2 (*c* 1.0, CH₂Cl₂) (94% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 7.87 (brs, 1H), 7.40-7.16 (m, 16H), 6.93-6.86 (m, 3H), 6.69 (dd, *J* = 16.0, 7.2 Hz, 1H), 6.42 (d, *J* = 16.0 Hz, 1H), 5.04 (d, *J* = 7.2 Hz, 1H), 4.95 (s, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 153.2, 143.5, 137.8, 137.7, 132.7, 132.2, 130.8, 128.7, 128.6, 127.9, 127.8, 127.4, 127.3, 126.6, 126.5, 123.7, 118.6, 113.2, 112.0, 103.7, 71.1, 46.4.

Cheung, H. Y.; Yu, W.-Y.; Lam, F. L.; Au-Yeung, T. T.-L.; Zhou, Z.; Chan, T. H.; Chan, A. S. C. *Org. Lett.* **2007**, 9, 4295.

Table 2, entry 7



9g, light yellow oil, $[\alpha]^{20}_{D}$ +36.2 (*c* 1.41, CH₂Cl₂) (92% ee). IR (film) 3427 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.91 (brs, 1H), 7.43-7.23 (m, 11H), 7.11 (d, *J* = 8.8 Hz, 1H), 6.99-6.93 (m, 2H), 6.75 (dd, *J* = 16.0, 7.2 Hz, 1H), 6.49 (d, *J* = 16.0 Hz, 1H), 5.10 (d, *J* = 7.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 157.8 (d, *J*_{C-F} = 233.0 Hz), 143.2, 137.6, 133.4, 132.3, 131.0, 128.7, 128.6, 127.5, 127.4, 127.3, 126.7, 126.5, 124.6, 119.0 (d, *J*_{C-F} = 4.7 Hz), 111.9 (d, *J*_{C-F} = 9.6 Hz), 110.7 (d, *J*_{C-F} =

26.2 Hz), 104.9 (d, $J_{C-F} = 23.5$ Hz), 46.3. HRMS (FT-ICRMS): Calcd. for $C_{23}H_{18}FN$ (M): 327.1423; Found: 327.1407.

Table 2, entry 8

9h, colorless oil, [α]²⁰_D+38.4 (*c* 0.23, CH₂Cl₂) (92% ee) [lit.: [α]²⁰_D -37.0 (*c* 0.94, CH₂Cl₂) (96% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 7.98 (brs, 1H), 7.43-7.38 (m, 3H), 7.37-7.24 (m, 9H), 7.15 (dd, *J* = 8.4, 2.0 Hz, 1H), 6.93 (d, *J* = 2.0 Hz, 1H), 6.72 (dd, *J* = 16.0, 7.2 Hz, 1H), 6.45 (d, *J* = 16.0 Hz, 1H), 5.09 (d, *J* = 7.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 143.1, 137.5, 135.2, 132.3, 131.0, 128.7, 128.6, 128.1, 127.5, 126.8, 126.5, 125.4, 124.2, 122.7, 119.4, 118.7, 112.3, 46.1. Cheung, H. Y.; Yu, W.-Y.; Lam, F. L.; Au-Yeung, T. T.-L.; Zhou, Z.; Chan, T. H.; Chan, A. S. C. *Org. Lett.* **2007**, 9, 4295.

Table 2, entry 9

9i, light yellow oil, $[\alpha]^{20}_{D}$ +28.1 (*c* 1.80, CH₂Cl₂) (92% ee) [lit.: $[\alpha]^{20}_{D}$ -35.1 (*c* 1.0, CH₂Cl₂) (94% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 8.02 (brs, 1H), 7.55 (d, *J* = 1.2 Hz, 1H), 7.38 (d, *J* = 7.2 Hz, 2H), 7.35-7.29 (m, 5H), 7.29-7.20 (m, 5H), 6.92 (d, *J* = 1.6 Hz, 1H), 6.69 (dd, *J* = 16.0, 7.2 Hz, 1H), 6.42 (d, *J* = 16.0 Hz, 1H), 5.07 (d, *J* = 7.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 143.1, 137.6, 135.5, 132.3, 131.1, 128.8, 128.7, 128.6, 128.5, 127.5, 126.8, 126.6, 125.3, 124.0, 122.5, 118.7, 113.0, 112.8, 46.1.

Cheung, H. Y.; Yu, W.-Y.; Lam, F. L.; Au-Yeung, T. T.-L.; Zhou, Z.; Chan, T. H.; Chan, A. S. C. *Org. Lett.* **2007**, 9, 4295.

Table 2, entry 10



9j, white solid, m.p. 135-137 °C, $[\alpha]_{D}^{20}$ +33.4 (*c* 1.34, CH₂Cl₂) (94% ee). IR (film) 3424 cm⁻¹; ¹H

NMR (400 MHz, CDCl₃) δ 7.94 (brs, 1H), 7.50 (s, 1H), 7.39 (d, J = 8.0 Hz, 2H), 7.35-7.21 (m, 9H), 7.15 (dd, J = 8.4, 1.6 Hz, 1H), 6.88 (d, J = 2.0 Hz, 1H), 6.72 (dd, J = 16.0, 7.2 Hz, 1H), 6.45 (d, J = 16.0 Hz, 1H), 5.10 (d, J = 7.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 143.2, 137.7, 137.5, 132.3, 131.0, 128.8, 128.7, 128.6, 127.5, 126.8, 126.5, 125.9, 123.4, 123.0, 121.4, 119.2, 115.9, 114.2, 46.3. HRMS (FT-ICRMS): Calcd. for C₂₃H₁₈BrN (M): 387.0623; Found: 387.0606.



9k, light yellow oil, $[\alpha]^{20}{}_{D}$ +37.3 (*c* 1.00, CH₂Cl₂) (75% ee) [lit.: $[\alpha]^{20}{}_{D}$ -40.7 (*c* 1.0, CH₂Cl₂) (96% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 7.89 (brs, 1H), 7.42- 7.30 (m, 9H), 7.30 - 7.21 (m, 2H), 7.04-6.97 (m, 2H), 6.92 (d, *J* = 1.6 Hz, 1H), 6.78 (d, *J* = 15.6, 7.2 Hz, 1H), 6.49 (d, *J* = 15.6 Hz, 1H), 5.16 (d, *J* = 7.2 Hz, 1H), 2.51 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 143.7, 137.8, 136.5, 132.8, 130.8, 128.7, 128.6, 128.5, 127.3, 126.6, 126.5, 122.9., 122.5, 120.4, 119.9, 119.4, 117.9, 46.5, 16.8.

Cheung, H. Y.; Yu, W.-Y.; Lam, F. L.; Au-Yeung, T. T.-L.; Zhou, Z.; Chan, T. H.; Chan, A. S. C. *Org. Lett.* **2007**, 9, 4295.

Table 2, entry 12



91, yellow oil, $[\alpha]^{20}_{D}$ +21.1 (*c* 0.55, CH₂Cl₂) (80% ee). IR (film) 3449, 3416, 1489 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.97 (brs, 1H), 7.43 (d, *J* = 7.6 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.35-7.20 (m, 9H), 7.10 (dd, *J* = 7.6, 7.2 Hz, 1H), 6.90 (d, *J* = 1.6 Hz, 1H), 6.69 (dd, *J* = 16,0, 7.2 Hz, 1H), 6.39 (dd, *J* = 16.0, 0.8 Hz, 1H), 5.12 (d, *J* = 7.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 141.8, 136.8, 135.9, 133.1, 132.8, 132.4, 130.0, 129.9, 128.8, 128.7, 127.7, 126.7, 122.8, 122.5, 119.9, 119.8, 118.1, 111.4, 45.7. HRMS (TOF-EI): Calcd. for C₂₃H₁₇Cl₂N (M): 377.0738; Found: 377.0742.

Table 2, entry 13



9m, light yellow form, m.p. 47-49 °C, $[\alpha]^{20}_{D}$ +32.8 (*c* 0.5, CH₂Cl₂) (86% ee). IR (film) 3419, 1510, 1456 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.91 (brs, 1H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.21 (t, *J* = 7.6 Hz, 1H), 7.18-7.10 (m, 4H), 7.07 (dd, *J* = 7.6, 7.2 Hz, 1H), 6.90 (d, *J* = 1.6 Hz, 1H), 6.71 (dd, *J* = 15.6, 7.2 Hz, 1H), 6.45 (d, *J* = 15.6 Hz, 1H), 5.12 (d, *J* = 7.2 Hz, 1H), 2.38 (s, 3H), 2.37 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 140.7, 137.0, 136.8, 136.0, 135.0, 131.9, 130.4, 129.4, 129.3, 128.5, 127.0, 126.4, 122.7, 122.2, 120.1, 119.6, 119.2, 111.3, 46.0, 21.4, 21.3. HRMS (TOF-EI): Calcd. for C₂₅H₂₃N (M): 337.1830; Found: 337.1835.

Table 2, entry 14



9n, light yellow oil, $[\alpha]^{20}_{D}$ +35.6 (*c* 1.74, CH₂Cl₂) (89% ee). IR (film) 3448, 3416, 1593, 1569, 1473 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.04 (brs, 1H), 7.41 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 2.4 Hz, 1H), 7.32 (s, 1H), 7.27-7.17 (m, 7H), 7.06 (t, *J* = 7.6 Hz, 1H), 6.93 (d, *J* = 1.6 Hz, 1H), 6.72 (dd, *J* = 15.6, 7.2 Hz, 1H), 6.37 (d, *J* = 15.6 Hz, 1H), 5.10 (d, *J* = 7.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 145.4, 139.3, 136.9, 134.7, 134.5, 133.4, 130.1, 130.0, 128.7, 127.5, 127.0, 126.9, 126.7, 126.4, 124.8, 122.8, 122.6, 119.9, 119.8, 117.8, 111.5, 46.0. HRMS (TOF-EI): Calcd. for C₂₃H₁₇Cl₂N (M): 377.0738; Found: 377.0743.

Scheme 3



13a, pink oil, $[\alpha]^{20}_{D}$ +19.7 (*c* 0.89, CH₂Cl₂) (98% ee, dr = 8.5/1). IR (film) 3439, 3422, 1599, 1449

cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.56 (brs, 1H), 7.37-7.18 (m, 20H), 6.56 (d, *J* = 15.6, 7.6 Hz, 2H), 6.39 (d, *J* = 15.6 Hz, 2H), 5.89 (d, *J* = 2.4 Hz, 2H), 4.82 (d, *J* = 7.6 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 142.4, 137.3, 132.9, 131.4, 131.3, 128.8, 128.7, 128.6, 127.6, 127.0, 126.6, 107.1, 48.3. HRMS (TOF-EI): Calcd. for C₃₄H₂₉N (M): 451.2300; Found: 451.2305.



13b, light yellow oil, $[\alpha]^{20}_{D}$ -11.5 (*c* 1.89, CH₂Cl₂) (86% ee). IR (film) 3423, 1598, 1492 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.60 (brs, 1H), 7.46-7.22 (m, 10H), 6.65 (dd, *J* = 15.6, 7.6 Hz, 1H), 6.49 (d, *J* = 15.6 Hz, 1H), 5.91-5.88 (m, 2H), 4.88 (d, *J* = 7.6 Hz, 1H), 2.61 (q, *J* = 7.6 Hz, 2H), 1.26 (t, *J* = 7.6 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 142.5, 137.3, 134.1, 131.6, 131.5, 131.2, 128.8, 128.7, 128.6, 127.6, 127.0, 126.6, 106.8, 104.3, 48.5, 21.1, 13.7. HRMS (TOF-EI): Calcd. for C₂₁H₂₁N (M): 287.1674; Found: 287.1678.

BOC-derivatives



Light yellow oil, $[\alpha]^{20}_{D}$ +36.1 (*c* 1.65, CH₂Cl₂) (92% ee) [lit.: $[\alpha]^{20}_{D}$ -38.8 (*c* 1.01, CH₂Cl₂) (95% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 8.14 (d, *J* = 7.6 Hz, 1H), 7.39 (d, *J* = 7.6 Hz, 4H), 7.37-7.20 (m, 9H), 7.15 (t, *J* = 7.6 Hz, 1H), 6.71 (dd, *J* = 16.0, 7.2 Hz, 1H), 6.46 (d, *J* = 16.0 Hz, 1H), 5.06 (d, *J* = 7.2 Hz, 1H), 1.69 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 150.1, 142.3, 137.4, 136.0, 131.5, 131.4, 130.1, 128.8, 128.7, 128.6, 127.6, 126.9, 126.6, 124.6, 124.0, 123.3, 122.6, 120.3, 115.5, 83.9, 46.2, 28.4.

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Colorless oil, $[\alpha]^{20}_{D}$ +33.6 (*c* 1.56, CH₂Cl₂) (82% ee) [lit.: $[\alpha]^{20}_{D}$ -39.6 (*c* 1.0, CH₂Cl₂) (92% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 8.28 (d, *J* = 8.8 Hz, 1H), 7.45-7.35 (m, 5H), 7.33-7.14 (m, 12H), 7.09 (t, *J* = 7.6 Hz, 1H), 6.74 (dd, *J* = 15.6, 7.6 Hz, 1H), 6.30 (d, *J* = 15.6 Hz, 1H), 4.84 (d, *J* = 7.6 Hz, 1H), 1.22 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 150.4, 142.8, 137.6, 137.2, 136.8, 134.5, 131.8, 130.9, 130.3, 128.7, 128.5, 128.3, 128.2, 128.1, 128.0, 127.5, 126.5, 126.4, 124.5, 122.7, 121.5, 121.1, 115.6, 83.3, 45.1, 27.7.

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White powder, m.p. 138-140 °C, $[\alpha]^{20}_{D}$ +45.6 (*c* 0.31, CH₂Cl₂) (94% ee). IR (film) 1731 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.02 (d, *J* = 8.0 Hz, 1H), 7.36-7.14 (m, 12H), 6.90 (d, *J* = 7.2 Hz, 1H), 6.68 (dd, *J* = 16.0, 6.8 Hz, 1H), 6.26 (d, *J* = 16.0 Hz, 1H), 5.33 (d, *J* = 6.8 Hz, 1H), 2.46 (s, 3H), 1.64 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 150.0, 142.9, 137.5, 136.5, 132.8, 131.5, 131.4, 129.0, 128.8, 128.7, 127.5, 126.9, 126.6, 125.1, 125.0, 124.6, 123.6, 113.3, 83.9, 46.7, 28.4, 20.5. HRMS (FT-ICRMS): Calcd. for C₂₉H₂₉NO₂ (M): 423.2198; Found: 423.2191.

White powder, m.p. 139-141 °C, $[\alpha]^{20}_{D}$ +45.8 (*c* 0.26, CH₂Cl₂) (92% ee) [lit.: $[\alpha]^{20}_{D}$ -41.2 (*c* 1.05, CH₂Cl₂) (96% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 8.00 (d, *J* = 7.2 Hz, 1H), 7.40-7.20 (m, 11H), 7.13 (dd, *J* = 8.8, 8.0 Hz, 2H), 6.70 (dd, *J* = 16.0, 7.2 Hz, 1H), 6.44 (d, *J* = 16.0 Hz, 1H), 5.03 (d, *J* = 7.2 Hz, 1H), 2.37 (s, 3H), 1.68 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 150.1, 142.4, 137.5, 134.2, 132.1, 131.6, 131.4, 130.3, 128.8, 128.7, 128.6, 127.5, 126.9, 126.6, 126.0, 124.1, 123.0, 120.1, 115.1, 83.7, 46.0, 28.5, 21.6.

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White solid, m.p. 109-111 °C, $[\alpha]^{20}{}_{D}$ +34.7 (*c* 2.25, CH₂Cl₂) (90% ee) [lit.: $[\alpha]^{20}{}_{D}$ -37.6 (*c* 0.95, CH₂Cl₂) (94% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 8.04 (brs, 1H), 7.43-7.22 (m, 11H), 6.94 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.82 (d, *J* = 2.0 Hz, 1H), 6.72 (dd, *J* = 16.0, 7.6 Hz, 1H), 6.49 (d, *J* = 16.0 Hz, 1H), 5.03 (d, *J* = 7.6 Hz, 1H), 3.75 (s, 3H), 1.69 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 155.8, 150.0, 142.2, 137.4, 131.5, 131.3, 130.9, 130.7, 128.8, 128.7, 128.6, 127.6, 126.9, 126.6, 124.6, 123.0, 116.1, 112.9, 103.3, 83.7, 55.8, 46.2, 28.4.

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Light yellow powder, m.p. 50-51 °C, $[\alpha]^{20}_{D}$ +30.1 (*c* 1.05, CH₂Cl₂) (90% ee) [lit.: $[\alpha]^{20}_{D}$ -28.2 (*c* 0.97, CH₂Cl₂) (94% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 7.98 (d, *J* = 6.8 Hz, 1H), 7.40-7.17 (m, 16H), 6.96 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.85 (d, *J* = 2.4 Hz, 1H), 6.64 (dd, *J* = 15.6, 7.2 Hz, 1H), 6.41 (d, *J* = 15.6 Hz, 1H), 4.95 (d, *J* = 7.2 Hz, 1H), 4.94 (s, 2H), 1.64 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 154.9, 150.0, 142.2, 137.4, 131.6, 131.3, 130.9, 128.8, 128.7, 128.6, 128.5, 128.0, 127.7, 127.6, 126.9, 126.6, 124.7, 123.0, 116.2, 113.9, 104.8, 83.7, 70.8, 46.2, 28.4.

Cheung, H. Y.; Yu, W.-Y.; Lam, F. L.; Au-Yeung, T. T.-L.; Zhou, Z.; Chan, T. H.; Chan, A. S. C. *Org. Lett.* **2007**, 9, 4295.



Light yellow powder, m.p. 49-51 °C, $[\alpha]^{20}{}_{D}$ +37.9 (*c* 1.56, CH₂Cl₂) (92% ee). IR (film) 1733 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.10 (brs, 1H), 7.45-7.42 (m, 11H), 7.06-6.99 (m, 2H), 6.69 (dd, *J* = 15.6, 7.2 Hz, 1H), 6.47 (d, *J* = 15.6 Hz, 1H), 5.00 (d, *J* = 7.2 Hz, 1H), 1.69 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 159.2 (d, *J*_{C-F} = 238 Hz), 149.8, 141.9, 137.3, 132.4, 131.7, 131.1, 131.0, 128.9, 128.8, 128.5, 127.7, 127.1, 126.6, 125.5, 123.0 (d, *J*_{C-F} = 4.7 Hz), 116.4 (d, *J*_{C-F} = 9.6 Hz), 112.3 (d, *J*_{C-F} = 26.2 Hz), 105.9 (d, *J*_{C-F} = 23.5 Hz), 84.1, 46.1, 28.4. HRMS (FT-ICRMS): Calcd. for



White solid, m.p. 136-138 °C, $[\alpha]^{20}_{D}$ +36.2 (*c* 1.50, CH₂Cl₂) (92% ee) [lit.: $[\alpha]^{20}_{D}$ -36.1 (*c* 0.95, CH₂Cl₂) (96% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 8.07 (d, *J* = 8.4 Hz, 1H), 7.42-7.23 (m, 13H), 6.68 (dd, *J* = 15.6, 7.6 Hz, 1H), 6.45 (d, *J* = 15.6 Hz, 1H), 5.01 (d, *J* = 7.6 Hz, 1H), 1.69 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 149.7, 141.9, 137.3, 134.4, 131.8, 131.3, 131.0, 128.9, 128.7, 128.5, 128.4, 127.7, 127.1, 126.6, 125.2, 124.8, 122.7, 119.8, 116.5, 84.3, 46.0, 28.4.

Cheung, H. Y.; Yu, W.-Y.; Lam, F. L.; Au-Yeung, T. T.-L.; Zhou, Z.; Chan, T. H.; Chan, A. S. C. *Org. Lett.* **2007**, 9, 4295.



Light yellow solid, m.p. 128-130 °C, $[\alpha]^{20}_{D}$ +35.4 (*c* 0.26, CH₂Cl₂) (92% ee) [lit.: $[\alpha]^{20}_{D}$ -32.9 (*c* 1.0, CH₂Cl₂) (94% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 8.00 (d, *J* = 8.4 Hz, 1H), 7.46 (s, 1H), 7.39-7.20 (m, 12H), 6.65 (dd, *J* = 15.6, 7.2 Hz, 1H), 6.42 (d, *J* = 15.6 Hz, 1H), 4.99 (d, *J* = 7.2 Hz, 1H), 1.66 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 149.7, 141.9, 137.3, 134.8, 131.8, 131.7, 131.0, 128.9, 128.8, 128.6, 127.7, 127.5, 127.1, 126.6, 125.1, 122.8, 122.6, 116.9, 116.1, 84.4, 45.9, 28.4. Cheung, H. Y.; Yu, W.-Y.; Lam, F. L.; Au-Yeung, T. T.-L.; Zhou, Z.; Chan, T. H.; Chan, A. S. C. *Org. Lett.* **2007**, 9, 4295.



White powder, m.p. 138-140 °C, $[\alpha]^{20}_{D}$ +37.0 (*c* 1.49, CH₂Cl₂) (94% ee). IR (film) 1733 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.37 (brs, 1H), 7.41-7.24 (m, 12H), 7.19 (d, *J* = 8.4 Hz, 1H), 6.68 (dd, *J* = 15.6, 7.2 Hz, 1H), 6.46 (d, *J* = 15.6 Hz, 1H), 5.03 (d, *J* = 7.2 Hz, 1H), 1.70 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 149.6, 142.0, 137.3, 136.8, 131.7, 131.0, 128.9, 128.8, 128.6, 127.7, 127.1, 126.6, 125.9, 124.4, 123.1, 121.4, 118.7, 118.4, 84.5, 46.1, 28.4. HRMS (FT-ICRMS): Calcd. for

C₂₈H₂₆BrNO₂ (M): 487.1147; Found: 487.1151.



White powder, m.p. 102-104 °C, $[\alpha]^{20}_{D}$ +34.9 (*c* 0.90, CH₂Cl₂) (75% ee) [lit.: $[\alpha]^{20}_{D}$ -44.8 (*c* 1.05, CH₂Cl₂) (96% ee)]. ¹H NMR (400 MHz, CDCl₃) δ 7.38 (d, *J* = 7.2 Hz, 2H), 7.35-7.22 (m, 9H), 7.19 (d, *J* = 8.4 Hz, 1H), 7.11-7.04 (m, 2H), 6.70 (dd, *J* = 16.0, 7.2 Hz, 1H), 6.45 (d, *J* = 16.0 Hz, 1H), 5.03 (d, *J* = 7.2 Hz, 1H), 2.65 (s, 3H), 1.63 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 149.9, 142.4, 137.5, 135.7, 131.5, 131.4, 131.3, 128.7, 128.6, 128.0, 127.5, 126.9, 126.6, 126.2, 125.6, 123.1, 122.8, 117.9, 83.6, 46.1, 28.3, 22.4.

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White solid, m.p. 65-67 °C, $[\alpha]^{20}_{D}$ +35.3 (*c* 1.9, CH₂Cl₂) (80% ee). IR (film) 1732, 1490, 1452 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.13 (d, *J* = 7.2 Hz, 1H), 7.38 (s, 1H), 7.36-7.21 (m, 10H), 7.15 (t, *J* = 7.6 Hz, 1H), 6.63(dd, *J* = 15.6, 7.2 Hz, 1H), 6.36 (d, *J* = 15.6 Hz, 1H), 5.01 (d, *J* = 7.2 Hz, 1H), 1.69 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 150.0, 140.5, 136.0, 135.6, 133.4, 132.8, 131.5, 130.7, 130.0, 129.7, 129.0, 128.9, 127.8, 124.8, 124.0, 122.8, 122.5, 120.1, 115.6, 84.1, 45.4, 28.4. HRMS (TOF-EI): Calcd. for C₂₈H₂₅Cl₂NO₂ (M): 477.1262; Found: 477.1267.



White solid, m.p. 54-56 °C, $[\alpha]^{20}_{D}$ +40.6 (*c* 0.51, CH₂Cl₂) (86% ee). IR (film) 1732, 1542, 1452 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.17 (d, *J* = 6.8 Hz, 1H), 7.44-7.37 (m, 2H), 7.36-7.28 (m,

3H), 7.28-7.23 (m, 2H), 7.21-7.11 (m, 5H), 6.67 (dd, J = 16.0, 7.2 Hz, 1H), 6.45 (d, J = 16.0 Hz, 1H), 5.03 (d, J = 7.2 Hz, 1H), 2.38 (s, 3H), 2.37 (s, 3H), 1.71 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 150.1, 139.4, 137.3, 136.3, 136.0, 134.7, 131.1, 130.6, 130.2, 129.4, 129.3, 128.5, 126.5, 124.5, 123.9, 123.6, 122.6, 120.4, 115.4, 83.8, 45.8, 28.4, 21.4, 21.3. HRMS (TOF-EI): Calcd. for C₃₀H₃₁NO₂ (M): 437.2355; Found: 437.2359.



White solid, m.p. 49-51 °C, $[\alpha]^{20}_{D}$ +41.6 (*c* 1.41, CH₂Cl₂) (89% ee). IR (film) 1732, 1593, 1569, 1474 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.13 (d, *J* = 7.2 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.34-7.27 (m, 3H), 7.27-7.19 (m, 6H), 7.16 (t, *J* = 7.6 Hz, 1H), 6.67 (dd, *J* = 16.0, 7.2 Hz, 1H), 6.37 (d, *J* = 16.0 Hz, 1H), 5.02 (d, *J* = 7.2 Hz, 1H), 1.69 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 150.0, 144.1, 139.0, 136.0, 134.8, 134.7, 132.1, 130.8, 130.1, 130.0, 129.7, 128.7, 127.7, 127.3, 126.8, 126.5, 124.8, 124.7, 124.1, 122.8, 122.1, 120.0, 115.6, 84.1, 45.7, 28.4. HRMS (TOF-EI): Calcd. for C₂₈H₂₅Cl₂NO₂ (M): 477.1262; Found: 477.1268.



X-ray structure of Boc-protected 9j

Table 1. Crystal data and structure refinement for Boc-protected **9j**.

Identification code	sa1001	
Empirical formula	C28 H26 Br N O2	
Formula weight	488.41	
Temperature	173(2) K	
Wavelength	0.71073 A	
Crystal system, space group	Monoclinic, C2	
Unit cell dimensions	a = 23.306(7) A alpha = 90 deg.	
	b = 5.8518(19) A beta = 91.215(5) deg.	
	c = 17.515(6) A gamma = 90 deg.	
Volume	2388.2(13) A^3	
Z, Calculated density	4, 1.358 Mg/m^3	
Absorption coefficient	1.746 mm^-1	
F(000)	1008	
Crystal size	0.43 x 0.13 x 0.12 mm	
Theta range for data collection	2.88 to 27.45 deg.	
Limiting indices	-30<=h<=30, -6<=k<=7, -22<=l<=22	
Reflections collected / unique	14599 / 5303 [R(int) = 0.0399]	
$Completeness to theta = 27.45 \qquad 99.6 \%$		
Absorption correction	Numerical	
Max. and min. transmission	0.8179 and 0.5206	
Refinement method Full-matrix least-squares on F ²		
Data / restraints / parameters	5303 / 1 / 292	
Goodness-of-fit on F^2	1.193	
Final R indices $[I>2sigma(I)]$ R1 = 0.0411, wR2 = 0.0973		
R indices (all data) $R1 = 0.0465, wR2 = 0.1078$		
Absolute structure parameter-0.005(8)		
Largest diff. peak and hole	0.618 and -0.631 e.A^-3	

The chromatography for the determination of enantiomeric excess

















HPLC Conditions: Column: Chiralpak AD-H, Daicel Chemical Industries, Ltd., Eluent: Hexanes/IPA (99/1); Flow rate: 0.8 mL/min; Detection: UV 254 nm





















S59

S65

