β -Alkyl- α -allylation of Michael Acceptors through the Palladium Catalyzed Three Component Coupling between Allylic Substrates, Trialkylboranes and Activated Olefins

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General Procedure.

The preparation of **3a** is representative. Into a argon purged screw capped vial containing **1a** (50 mg, 0.3182 mmol), and Pd(PPh₃)₄ (19 mg, 0.0159 mmol) were added successively THF (2.0 mL), Et₃B (0.38 mL of 1 M hexane solution, 0.3818 mmol), and allyl acetate (38 mg, 0.3818 mmol) via syringe. The reaction mixture was stirred at 40 °C for 6 h, during which the reaction was monitored by means of TLC. After dilution with ethyl acetate (30 mL), the mixture was washed with sat. NaHCO₃ and with brine. The organic layer was dried (Na₂SO₄) and concentrated in vacuo, and the residue was subjected to column chromatography over silica gel (EtOAc-hexane, 1/9) to give **3a** in 81% yield (56 mg).

(3a). Yield = 81%; IR (neat) 2255, 1625 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 7.40-7.3 (m, 5H), 5.91-5.80 (m, 1H), 5.36 (d, *J* = 10.3 Hz, 1H), 5.27 (dt, *J* = 16.8, 1.5 Hz, 1H), 2.88 (dd, *J* = 11.9, 3.4 Hz, 1H), 2.46 (dd, *J* = 14.2, 7.6 Hz, 1H), 2.34 (ddd, *J* = 14.2, 7.1, 1.0 Hz, 1H), 2.26-2.04 (m, 2H), 0.80 (t, *J* = 7.6 Hz, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 135.1, 128.9, 128.8, 128.7, 128.7, 122.7, 115.4, 114.5, 53.1, 43.5, 40.5, 25.0, 11.9; HRMS calcd for C₁₅H₁₆N₂ (M⁺) 224.1313, found 185.1103, 184.0997, 154.0546, 129.0599, 128.0493, 127.0457.

(3b). Yield = 78%; IR (neat) 2250, 1630 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 7.11 (bs, 4H), 5.85 (m, 1H), 5.29 (d, *J* = 10.2 Hz, 1H), 5.23 (dd, *J* = 17.0, 1.3 Hz, 1H), 2.78 (dd, *J* = 12.0, 3.4 Hz, 1H), 2.39 (dd, *J* = 13.9, 7.6 Hz, 1H), 2.29 (bs, 4H), 2.18-1.96 (m, 2H), 0.74 (t, *J* = 7.5 Hz, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 138.5, 131.9, 129.6, 128.8, 128.7, 122.7, 115.5, 114.6, 52.8, 43.6,

40.5, 24.9, 21.1, 11.9; HRMS calcd for $C_{16}H_{18}N_2$ (M^+) 238.1470, found 239.1522, 238.1466, 168.0698, 140.0475, 134.1072, 133.1003.

(3c). Yield = 79%; IR (neat) 2254, 1632 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz) δ 7.22 (d, J = 8.3 Hz, 2H), 6.90 (d, J = 8.3 Hz, 2H), 5.90-5.80 (m, 1H), 5.35 (d, J = 9.2 Hz, 1H), 5.28 (dd, J = 16.9, 1.2 Hz, 1H), 3.80 (s, 3H), 2.85 (d, J = 11.9, 3.4 Hz, 1H), 2.44 (dd, J = 14.0, 7.3 Hz, 1H), 2.35 (d, J = 14.0, 7.1 Hz, 1H), 2.22-2.01 (m, 2H), 0.80 (t, J = 7.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.7, 129.8, 128.8, 126.8, 122.7, 115.5, 114.6, 114.3, 55.2, 52.4, 43.7, 40.4, 24.9, 11.9; HRMS calcd for $C_{16}H_{18}N_2O$ (M^+) 254.1419, found 277.1311 ($M+Na$).

(3d). Yield = 61%; IR (neat) 2245, 1621 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz) δ 8.26 (d, J = 8.8 Hz, 2H), 7.54 (d, J = 8.8 Hz, 2H), 5.91-5.81 (m, 1H), 5.41 (d, J = 10.3 Hz, 1H), 5.31 (d, J = 16.8 Hz, 1H), 3.01 (d, J = 12.0, 3.7 Hz, 1H), 2.53 (dd, J = 14.0, 7.3 Hz, 1H), 2.42 (dd, J = 14.0, 7.3 Hz, 1H), 2.29-2.02 (m, 2H), 0.81 (t, J = 7.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 148.1, 142.7, 129.9, 128.1, 124.2, 123.5, 114.6, 113.9, 52.4, 43.1, 40.4, 24.7, 11.8; HRMS calcd for $C_{15}H_{15}N_3O_2$ (M^+) 269.1164, found 292.1056 ($M+Na$).

(3e). Yield = 66%; IR (neat) 2255, 1633 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz) δ 7.37 (s, 1H), 6.33 (s, 2H), 5.86-5.75 (s, 1H), 5.36 (d, J = 10.2 Hz, 1H), 5.27 (dd, J = 17.1, 3.4 Hz, 1H), 3.04 (dd, J = 11.0, 4.4 Hz, 1H), 2.48-2.34 (m, 2H), 2.09-1.97 (m, 2H), 0.83 (t, J = 7.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 148.9, 142.9, 128.6, 122.9, 114.9, 114.1, 110.6, 109.9, 46.8, 42.8, 40.0, 24.1, 11.8; HRMS calcd for $C_{13}H_{14}N_2O$ (M^+) 214.1106, found 237.0998 ($M+Na$).

(3f). Yield = 70%; IR (neat) 2249, 1622 cm^{-1} ; 1H NMR ($CDCl_3$, 400 MHz) δ 5.91-5.81 (m, 1H), 5.37-5.31 (m, 2H), 2.68 (dd, J = 13.6, 7.0 Hz, 1H), 2.59 (dd, J = 13.6, 7.0 Hz, 1H), 2.22-2.14 (m, 1H), 1.69-1.47 (m, 3H), 1.06-1.00 (m, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 128.9, 122.8, 115.6, 115.2, 50.6, 41.9, 40.9, 29.7, 22.4, 19.9, 16.9, 14.9; HRMS calcd for $C_{12}H_{18}N_2$ (M^+) 190.1470, found 175.1228, 161.1050, 149.1082, 148.0998, 147.0913.

(3g). Yield = 66%; IR (neat) 2256, 1618 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 5.95-5.85 (m, 1H), 5.39-5.32 (m, 2H), 2.77 (dd, $J = 13.9, 9.5$ Hz, 1H), 2.59 (dd, $J = 13.9, 9.5$ Hz, 1H), 1.74-1.64 (m, 1H), 1.48-1.34 (m, 2H), 1.12 (s, 9H), 1.08 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 128.9, 123.0, 116.5, 115.7, 55.6, 42.5, 40.1, 35.8, 28.9, 22.7, 16.8; HRMS calcd for $\text{C}_{13}\text{H}_{20}\text{N}_2$ (M^+) 204.1626, found 190.1426, 189.1387, 148.1039, 147.0922, 134.0991, 133.0791, 121.0893, 120.0785.

(3h). Yield = 81%; IR (neat) 2258, 1617 cm^{-1} ; ^1H NMR and ^{13}C NMR shows a mixture of diastereomers (see page S-19 and S-20); HRMS calcd for $\text{C}_{17}\text{H}_{21}\text{NO}_2$ (M^+) 271.1572, found 294.1465 ($\text{M}+\text{Na}$).

(3i). Yield = 82%; IR (neat) 2255, 1630 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 7.43-7.24 (m, 10H), 6.54 (d, $J = 15.6$ Hz, 1H), 6.17 (dt, $J = 15.6, 7.6$ Hz, 1H), 2.92 (dd, $J = 12.0, 3.7$ Hz, 1H), 2.65-2.50 (m, 2H), 2.27-2.03 (m, 2H), 0.8 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 137.3, 135.7, 135.1, 128.9, 128.8, 128.7, 128.5, 128.2, 126.5, 119.4, 115.6, 114.6, 53.1, 43.8, 39.9, 25.1, 11.9; HRMS calcd for $\text{C}_{21}\text{H}_{20}\text{N}_2$ (M^+) 300.1626, found 323.1622 ($\text{M}+\text{Na}$).

(3j). Yield = 62%; IR (neat) 2260, 1621 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 7.33-7.24 (m, 5H), 5.80-5.75 (m, 1H), 5.30 (d, $J = 10.2$ Hz, 1H), 5.22 (dd, $J = 17.1, 1.2$ Hz, 1H), 2.90 (t, $J = 7.8$ Hz, 1H), 2.39 (dd, $J = 14.2, 7.3$ Hz, 1H), 2.30-2.20 (m, 1H), 2.11-2.02 (m, 2H), 1.32-1.15 (m, 2H), 1.08-1.01 (m, 1H), 0.94-0.85 (m, 1H), 0.77 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 135.3, 128.9, 128.7, 122.7, 115.4, 114.5, 51.4, 43.6, 39.7, 31.4, 28.8, 22.2, 13.8; HRMS calcd for $\text{C}_{17}\text{H}_{20}\text{N}_2$ (M^+) 252.1626, found 275.1624 ($\text{M}+\text{Na}$).

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3c (^{13}C NMR, 100MHz)

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3e (^{13}C NMR, 100MHz)

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3f (¹³C NMR, 100MHz)

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3a (¹H NMR, 400MHz)

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NAME 13C

PROB 13C

EXFO BCM

ORFRO 100.40 MHz

ORSET 125.00 KHz

ORFIN 10500.00 Hz

POINT 32768

FREQ 27173.90 Hz

SCANS 200

ACQTM 1.2059 sec

PUL 1.1940 sec

PRG 5.20 usec

IRNUC 1H 24.2 c

CTEMP CDCL3

SLVIT 77.00 ppm

EXREF 0.12 Hz

BF 23

11.8869

25.1356

39.9104

43.7546

53.0765

76.6783

77.0000

77.2062

77.3135

114.5763

115.5663

119.3775

126.4968

128.1880

128.5097

128.7324

128.8232

128.9882

135.1340

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 OBSCT 124.00 MHz
 CAPIN 10500.0 Hz
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 FREQ0 7993.6 Hz
 SCANS 8
 ACQTH 4.081 sec
 PD 2.901 sec
 PULP 5.5 us
 INPRC 1H
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 EXREF 0.00 ppm
 RF 0.12 Hz
 PGM39 23

2a (7H NMR, 400 MHz)

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 OFERQ 100.40 MHz
 OBSET 125.00 KHz
 OFFTN 10500.00 Hz
 POINT 32768
 FREQU 27173.90 Hz
 SCANS 169
 ACQTM 1.2059 sec
 PD 1.1940 sec
 PWR 5.20 usec
 1H 25.7 °C
 CDCL3 77.00 ppm
 EXREF 0.12 Hz
 BF 23
 RGAIN

3j (¹³C NMR, 100MHz)