Palladium(0)-catalyzed cross-benzannulation between conjugated enynes. Reactivity-controlled synthesis of multifunctionalized benzenes

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Supporting information

Analytical and Spectral Data for 3a and 9-14.

3a: yellow oil: ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.53 (d, 2H, *J* = 8.3 Hz), 7.14 (d, 2H, *J* = 8.3 Hz), 6.88 (d, 1H, *J* = 12.8 Hz), 5.87 (d, 1H, *J* = 12.7 Hz), 4.16 (ddd, 2H, *J* = 7.2, 7.2, 7.2 Hz), 2.58 (t, 2H, *J* = 7.7 Hz), 1.61-1.54 (m, 2H) 1.28-1.21 (m, 9H), 0.86 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.4, 144.3, 143.2, 132.1, 130.0, 128.1, 118.7, 60.2, 35.8, 31.7, 31.2, 29.0, 22.6, 14.1; IR (neat) 2956, 2929, 2856, 1722, 1625, 1608, 1564, 1512, 1463, 1443, 1412, 1387, 1177, 1123, 1096, 1031, 949, 853 725 cm^{-1}; Anal. Calcd for $C_{17}H_{24}O_2$: C, 78.42; H, 9.29. Found: C, 78.71; H, 9.58. HRMS Calcd for $C_{17}H_{24}O_2$: 260.1776. Found: 260.1771.

9: yellow oil: ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.65 (d, 1H, *J* = 16.0 Hz), 7.42 (d, 2H, *J* = 8.1 Hz), 7.17 (d, 2H, *J* = 8.1 Hz), 6.37 (d, 1H, *J* = 16.0 Hz), 4.24 (ddd, 2H, *J* = 7.1, 7.1, 7.1 Hz), 2.60 (t, 2H, *J* = 8.1 Hz), 1.61-1.54 (m, 2H) 1.37-1.28 (m, 9H), 0.86 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.2, 145.7, 144.6, 131.9, 128.9, 128.0, 117.1, 60.4, 35.9, 31.7, 31.2, 28.9, 22.6, 14.3, 14.1; IR (neat) 2956, 2929, 2865, 1716, 1634, 1608, 1568, 1512, 1465, 1446, 1366, 1310, 1173, 1039, 983, 826 cm^{-1}; Anal. Calcd for $C_{17}H_{24}O_2$: C, 78.42; H, 9.29. Found: C, 78.37; H, 9.19. HRMS Calcd for $C_{17}H_{24}O_2$: 260.1776. Found: 260.1775.

10: yellow oil: ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.53 (d, 2H, *J* = 8.3 Hz), 7.17 (d, 2H, *J* = 8.1 Hz), 6.88 (d, 1H, *J* = 12.6 Hz), 5.88 (d, 1H, *J* = 12.6 Hz), 4.16 (t, 1H, *J* = 5.4 Hz), 2.72-2.66 (m, 2H), 1.79-1.73 (m, 2H), 1.32-1.22 (m, 12H), 5.81); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.3, 143.8, 143.1, 132.3, 130.1, 128.0, 118.9, 70.8, 60.2, 45.4, 30.6, 29.3, 14.1; IR (neat) 3419, 3072, 3024, 2972, 2937, 2868, 1716, 1625, 1608, 1564, 1512, 1465, 1444, 1409, 1377, 1272, 1178, 1099, 1029, 985, 916, 854, 833, 785 cm^{-1}; HRMS Calcd for $C_{16}H_{22}O_3$: 262.1569. Found: 262.1570.

11: pale yellow oil: ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.53 (d, 2H, *J* = 8.0 Hz), 7.14 (d, 2H, *J* = 8.2 Hz), 6.88 (d, 1H, *J* = 12.6 Hz), 5.87 (d, 1H, *J* = 12.8 Hz), 4.67 (ddd, 1H, *J* = 5.3, 5.3, 5.3 Hz), 4.16 (ddd, 2H, *J* = 7.0, 7.0, 7.0 Hz), 3.68-3.34 (m, 4H), 2.58 (t, 2H, *J* = 7.5 Hz) 1.54-1.51 (m, 2H), 1.29-1.16 (m, 23H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.3, 144.3, 143.2, 132.1, 129.9, 128.0, 118.7, 99.5, 65.3, 60.6, 60.2, 35.8, 31.2, 29.9, 29.6, 29.4, 29.3, 19.9, 15.3, 14.1; IR (neat) 3853, 3751, 3735, 3649, 2977, 2929, 2854, 1722, 1625, 1558, 1510, 1456, 1444, 1386, 1379, 1338, 1299, 1257, 1174, 1134, 1099, 1060, 1031, 981, 948, 854, 732 cm^{-1}; Anal. Calcd for $C_{23}H_{40}O_4$: C, 74.22; H, 9.97. Found: C, 74.01; H, 9.96. HRMS Calcd for $C_{23}H_{40}O_4$: 404.2927. Found: 404.2941.

12: yellow oil: ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.81 (d, 1H, *J* = 1.8 Hz), 7.71 (dd, 1H, *J* = 8.1, 1.8 Hz), 7.27 (d, 1H, *J* = 8.3 Hz), 6.85 (d, 1H, *J* = 22.7Hz), 5.99 (d, 1H, *J* = 12.7 Hz), 4.16 (ddd, 1H, *J* = 7.2, 7.2, 7.2 Hz), 2.81 (t, 2H, *J* = 7.9 Hz), 1.70-1.60 (m, 2H) 1.36-1.30 (m, 4H),), 1.24 (t, 3H, *J* = 7.2 Hz) 0.88 (t, 3H, *J* = 7.0 Hz); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.5, 147.4, 140.8, 133.9, 133.1, 129.1, 121.4, 117.8, 112.2, 60.6, 34.4, 31.3, 30.4, 22.4, 14.1, 13.9; IR (neat) 2957, 2931, 2861, 2226, 1717, 1635, 1496, 1467, 1387, 1310, 1182, 1029, 853 cm^{-1}; Anal. Calcd for $C_{20}H_{21}NO_2$: C, 75.25; H, 7.80; N, 5.16. Found: C, 75.14; H, 7.92; N, 5.16. HRMS Calcd for $C_{20}H_{21}N$: 271.1572. Found: 271.1568.

13: yellow oil: ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.31 (d, 2H, *J* = 8.2 Hz), 7.18 (d, 2H, *J* = 8.2 Hz), 5.47 (s, 1H), 2.83 (t, 2H, *J* = 7.9 Hz), 2.60 (t, 2H, *J* = 8.1 Hz), 1.59-1.29 (m, 14H), 0.88-0.81 (m, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.0, 145.4, 134.8, 128.9, 126.2, 117.8, 94.5, 35.7, 33.7, 31.6, 31.4, 31.2, 28.9, 28.3, 22.6, 22.3, 14.1, 13.9; IR (neat) 2956, 2929, 2858, 2213, 1599, 1560, 1512, 1465, 1413, 1379, 1189, 1018, 809 cm^{-1}; HRMS Calcd for $C_{20}H_{29}N$: 283.2300. Found: 283.2309.

14: yellow oil: ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.35 (dd, 2H, *J* = 6.4, 1.8 Hz), 7.20 (d, 2H, *J* = 8.1 Hz), 5.30 (t, 1H, *J* = 1.1 Hz), 2.60 (t, 2H, *J* = 7.9 Hz), 2.52 (t, 2H, *J* = 7.1 Hz), 1.63-1.55 (m, 2H) 1.40-1.22 (m, 12H), 0.89-0.81 (m, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.0, 144.8, 134.6, 128.6, 127.2, 117.9, 94.3, 38.0, 35.8, 31.7, 31.2, 29.0, 27.4, 22.6, 22.3, 14.1, 13.9; IR (neat) 3029, 2955, 2858, 2215, 1609, 1512, 1466, 1378, 1310, 1187, 1122, 1018, 822 cm^{-1}; Anal. Calcd for $C_{20}H_{29}N$: C, 84.75; H, 10.31; N, 4.94. Found: C, 84.66; H, 10.05; N, 4.90. HRMS Calcd for $C_{20}H_{29}N$: 283.2300. Found: 283.2306.