Supplementary Information

Study on the Regioselectivity of Rhodium-Catalyzed Ring opening Reactions of C₁-Substituted 7-Oxabenzonorbornadienes with Boronic Acids

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General Considerations

All ring opening reactions were carried out under inert atmospheric conditions. All glassware was oven dried overnight before use. Flash column chromatography was performed on 230-400 mesh silica gel purchased from Silicycle. Analytical TLC was performed on pre-coated silica gel 250 μm 60 F254 aluminum plates, also purchased from Silicycle. TLC visualization was carried out under UV light and *p*-anisaldehyde stain. ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance 400 MHz spectrometer (CDCl_3 : δ 7.24 ppm (^1H at 400 MHz) or δ 77.0 ppm (^{13}C at 100 MHz)). HRMS analyses were performed at the Queen's Mass Spectrometry and Proteomics Unit, Kingston, Ontario. The samples were ionized by electron impact (EI) or electrospray ionization (ESI) as specified and detection of the ions was performed by time of flight (TOF).

General procedure for Rh catalyzed reaction between 1-substituted-7-oxabenzonorbornadiene and boronic acids.

Inside an inert atmospheric glove box system, $[\text{Rh}(\text{COD})\text{Cl}]_2$ (5-7 mol%), NaHCO_3 (1 equiv.), and boronic acid (1.1 equiv.) were weighed and added to a screw cap vial equipped with a stir bar. To this mixture was charged the substituted oxabenzonorbornadiene (0.11-0.35 mmol) and methanol (2 mL). The vial was then capped and left to stir at room temperature. Upon reaction completion, the vial was removed from the dry box, diluted with DCM, and passed through a silica plug. The crude product was then subjected to flash column chromatography, eluting with mixtures of EtOAc/hexanes to provide the neat product.

(1*S,2*R**)-1,2-dihydro-4-ethyl-2-phenyl-1-naphthalenol (16a)** Yield: 96% (38.2 mg, 0.152 mmol) Light brown oil. R_f = 0.44 (EtOAc-Hexanes, 3:7). IR (CH_2Cl_2); 3564, 3054, 2986, 1602,

1452, 1422, 1265, 896, 739 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 1.17 (t, J = 7.4 Hz, 3H), 1.41 (d, J = 8.4 Hz, 1H), 2.52 (dq, J = 1.3, 7.4 Hz, 2H), 3.76 (dd, 5.8, 4.4 Hz, 1H), 4.81 (dd, J = 8.3, 5.9 Hz, 1H), 5.87 (d, J = 4.4 Hz, 1H), 7.12-7.35 (m, 9H). ^{13}C NMR (100 MHz, CDCl_3): δ 12.9, 25.1, 47.0, 71.5, 122.9, 124.3, 126.6, 127.3, 127.5, 128.1, 128.5, 129.3, 133.6, 136.9, 138.2, 138.5. HRMS (EI) calcd. for $\text{C}_{18}\text{H}_{18}\text{O}$ (M^+): 250.1358; found: 250.1354.

(1S*,2R*)-1,2-dihydro-4-ethyl-2-(4-methylphenyl)-1-naphthalenol (16b) Yield: 96% (85.9 mg, 0.325 mmol). Light brown oil. R_f = 0.35 (EtOAc–hexanes, 1:5). IR (CH_2Cl_2): 3560, 3054, 2986, 1603, 1514, 1422, 1266, 896, 735 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 1.28 (t, J = 7.4 Hz, 3H), 1.55 (s, 1H), 2.35 (s, 3H), 2.61-2.65 (m, 2H), 3.81 – 3.85 (m, 1H), 4.91 (d, J = 5.8 Hz, 1H), 5.96 – 5.97 (m, 1H), 7.14 – 7.17 (m, 4H), 7.27 – 7.31 (m 1H), 7.35 – 7.44 (m, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 13.0, 21.1, 25.2, 46.6, 71.5, 76.8, 77.1, 77.4, 122.9, 124.6, 126.56, 127.6, 128.0, 129.0, 129.3, 133.7, 135.0, 137.1, 138.4. HRMS (ESI) calcd. for $\text{C}_{19}\text{H}_{20}\text{ONa}$ ($\text{M}+\text{Na}^+$): 287.1411; found: 287.1420.

(1S*,2R*)-1,2-dihydro-4-ethyl-2-(3-methylphenyl)-1-naphthalenol (16c) Yield: 97% (78.4 mg, 0.297 mmol). Light brown oil; R_f = 0.35 (EtOAc–hexanes, 1:5). IR (CH_2Cl_2): 3560, 3054, 2986, 1605, 1422, 1264, 1094, 896 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 1.28 (t, J = 7.4 Hz, 3H), 1.56 (s, 1H), 2.36 (s, 3H), 2.63-2.65 (m, 2H), 3.82 – 3.85 (m, 1H), 4.88 (d, J = 5.7 Hz, 1H), 5.96 (d, J = 4.1 Hz, 1H), 7.06-7.45 (m, 8H). ^{13}C NMR (100 MHz, CDCl_3): δ 13.0, 21.5, 25.2, 47.0, 71.6, 123.0, 124.4, 126.2, 126.9, 127.6, 128.1, 128.2, 128.6, 130.1, 133.6, 137.0, 138.2, 138.4, 138.5. HRMS (EI) calcd. for $\text{C}_{19}\text{H}_{20}\text{O}$ (M^+): 264.1514; found: 264.1517.

(1S*,2R*)-1,2-dihydro-4-ethyl-2-(2-methylphenyl)-1-naphthalenol (16d) Yield: 87%. (65.0 mg, 0.245 mmol). Off-white solid, Mp: 95-96 °C; R_f = 0.40 (EtOAc–hexanes, 1:5). IR (CH₂Cl₂): 3562, 3054, 2986, 1602, 1488, 1422, 1265, 896, 739 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 1.16 (t, J = 7.4 Hz, 3H), 1.44 (s, 1H), 2.34 (s, 3H), 2.51 (q, J = 7.4 Hz, 2H), 4.07 – 4.10 (m, 1H), 4.68 (d, J = 7.9 Hz, 1H), 5.78 (d, J = 2.2 Hz, 1H), 7.07 – 7.21 (m, 6H), 7.26 – 7.35 (m, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 13.0, 19.9, 20.0, 25.2, 42.8, 70.1, 122.95, 125.07, 126.8, 127.1, 128.0, 129.29, 133.5, 136.4, 136.7, 137.4, 138. HRMS (EI) calcd. for C₁₉H₂₀O (M⁺): 264.1514; found: 264.1518.

(1S*,2R*)-1,2-dihydro-4-ethyl-2-(4-methoxyphenyl)-1-naphthalenol (16e) Yield: 92% (45.6 mg, 0.163 mmol). Off-white solid, Mp: 63-65 °C; R_f = 0.30 (EtOAc–hexanes, 1:5). IR (CH₂Cl₂): 3444, 2962, 2931, 1641, 1473, 1267, 1132 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 1.23 (t, J = 7.3 Hz, 3H), 1.45 (d, J = 8.6 Hz, 1H), 2.57 (dq, J = 7.3, 1.4 Hz, 2H), 3.73-3.78 (m, 4H), 4.85-4.89 (at, 1H), 5.91 (d, J = 4.6 Hz, 1H), 6.81 (d, J = 8.7 Hz, 2H), 7.11 (d, J = 8.7 Hz, 2H), 7.22-7.26 (m, 4H). ¹³C NMR (100 MHz, CDCl₃): δ 13.0, 25.2, 31.0, 46.1, 55.3, 71.5, 114.0, 118.6, 122.9, 124.8, 126.4, 127.7, 128.0, 129.7, 130.4, 133.7, 137.2, 138.4, 158.9. HRMS (ESI) calcd. for C₁₉H₂₀O₂Na (M+Na⁺): 303.1360; found: 303.1364.

(1S*,2R*)-1,2-dihydro-4-ethyl-2-(3-methoxyphenyl)-1-naphthalenol (16f) Yield: 99% (71.8 mg, 0.256 mmol). Light brown oil. R_f = 0.27 (EtOAc–hexanes, 1:5). IR (CH₂Cl₂): 3559, 3054, 2986, 1599, 1486, 1422, 1264, 1155, 1050 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 1.28 (t, J = 7.4 Hz, 3H), 1.59 (s, 1H), 2.60-2.66 (m, 2H), 3.75 (s, 3H), 3.83 (dd, J = 6.5, 4.4 Hz, 1 H), 4.93 (d, J = 6.1, 1 H), 5.96 (d, J = 4.4, 1 H), 6.81 – 6.88 (m, 3H), 7.23 – 7.31 (m, 2H), 7.34 – 7.43 (m, 3H).

^{13}C NMR (100 MHz, CDCl_3): δ 12.9, 25.1, 47.0, 55.1, 71.4, 112.6, 114.8, 121.6, 122.9, 124.2, 126.5, 127.6, 128.0, 129.5, 133.5, 127.0, 138.5, 139.8, 159.7. HRMS (EI) calcd. for $\text{C}_{19}\text{H}_{20}\text{O}_2$ (M^+): 280.1463; found: 280.1465.

(1S*,2R*)-1,2-dihydro-4-ethyl-2-(2-methoxyphenyl)-1-naphthalenol (16g) Yield: 85% (65.0 mg, 0.246 mmol). Off-white solid, Mp: 70-72 °C; R_f = 0.24 (EtOAc–hexanes, 1:5). IR (CH_2Cl_2): 3565, 3054, 2986, 1600, 1491, 1463, 1439, 1422, 1264, 739 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 1.17 (t, J = 7.5 Hz, 3H), 2.02 (s, 1H), 2.5 – 2.55 (m, 2H), 3.79 (s, 3H), 4.3 – 4.33 (m, 1H), 4.8 (d, J = 5.5 Hz, 1H), 5.77 (d, 3.6 Hz, 1H), 6.81 (d, J = 1.0 Hz, 1H), 6.84 (d, J = 7.7 Hz, 1H), 7.12 – 7.20 (m, 3H), 7.25 (dt, J = 7.5, 1.6 Hz, 1H), 7.32 (d, J = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3): δ 13.1, 25.2, 39.7, 55.5, 70.5, 110.5, 120.8, 122.9, 124.3, 126.7, 127.4, 127.5, 128.1, 128.2, 130.2, 133.3, 136.7, 138.4, 157.3. HRMS (EI) calcd. for $\text{C}_{19}\text{H}_{20}\text{O}_2$ (M^+): 280.1463; found: 280.1467.

(1S*,2R*)-1,2-dihydro-4-ethyl-2-(4-chlorophenyl)-1-naphthalenol (16h) Yield: 64% (62.3 mg, 0.219 mmol). Light brown oil, R_f = 0.51 (EtOAc–Hexanes, 1:4). IR (KBr): 3416, 2964, 1488, 1265, 1091, 1027, 1015, 822, 767, 756, 739, 705 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 1.15 (t, J = 7.4 Hz, 3H), 1.40 (d, J = 8.6 Hz, 1H), 2.48 – 2.54 (m, 2H), 3.68 – 3.71 (m, 1H), 4.78 (dd, J = 6.0, 3.4 Hz, 1H), 5.79 (d, J = 4.4 Hz, 1H), 7.05 – 7.35 (m, 8H). ^{13}C NMR (100 MHz, CDCl_3): δ 13.0, 25.2, 46.4, 71.4, 123.1, 123.9, 126.5, 127.9, 128.2, 128.6, 130.7, 133.1, 133.4, 136.8, 138.9, 138.9. HRMS (EI) calcd. for $\text{C}_{18}\text{H}_{17}\text{ClO}$ (M^+): 284.0968; found: 284.0959.

(1S*,2R*)-1,2-dihydro-4-ethyl-2-(3-chlorophenyl)-1-naphthalenol (16i) Yield: 98% (96.5 mg, 0.339 mmol). Light brown oil, $R_f = 0.51$ (EtOAc-Hexanes, 1:4). IR (KBr): 3557, 3423, 3061, 2967, 2933, 2876, 1595, 1571, 1475, 1452, 1429, 1378, 1265, 1094, 1079, 1045, 999, 841, 789, 761, 738, 698, 677 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 1.16 (t, $J = 7.4$ Hz, 3H), 1.43 (s, 1H), 2.49 – 2.55 (m, 2H), 3.70 – 3.73 (m, 1H), 4.76 (d, $J = 5.6$ Hz, 1H), 5.80 (d, $J = 4.1$, 1H), 7.03 – 7.34 (m, 8H). ^{13}C NMR (100 MHz, CDCl_3): δ 13.0, 25.2, 46.9, 71.5, 123.2, 123.5, 126.7, 127.4, 127.5, 127.9, 128.4, 129.5, 129.7, 133.3, 134.3, 136.7, 139.1, 141.0. HRMS (EI) calcd. for $\text{C}_{18}\text{H}_{17}\text{ClO}$ (M^+): 284.0968; found: 284.0961.

(1S*,2R*)-1,2-dihydro-4-methyl-2-phenyl-1-naphthalenol (16j) Yield: 94% (54.8mg, 0.219 mmol). Light brown oil, $R_f = 0.49$ (EtOAc-Hexanes, 1:3). ^1H NMR (400 MHz, CDCl_3): δ 1.51 (d, $J = 8.2$ Hz, 1H), 2.22 (t, $J = 1.7$ Hz, 3H), 3.86-3.88 (m, 1H), 4.92 (dd, $J = 10.2$, 6 Hz, 1H), 5.97 (dd, $J = 4.1$, 1.4 Hz, 1H), 7.26-7.40 (m, 9H). ^{13}C NMR (100 MHz, CDCl_3): δ 19.3, 47.3, 71.6, 123.3, 126.4, 126.6, 127.3, 127.8, 128.2, 128.6, 129.3, 133.0, 134.2, 136.58, 138.3. Spectra data are in accord with the literature values.¹

(1S*,2R*)-1,2-dihydro-4-isopropyl-2-phenyl-1-naphthalenol (16l) Yield: 25% (16.6 mg, 0.063 mmol). Light brown oil, $R_f = 0.38$ (EtOAc-Hexanes, 1:5). IR (CH_2Cl_2): 3556, 3432, 3060, 3028, 2962, 2929, 2872, 1601, 1492, 1452, 1382, 1265, 1073, 1032, 771, 757, 737, 700 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 1.15 (d, $J = 6.7$ Hz, 3H), 1.18 (d, $J = 6.7$ Hz, 3H), 1.41 (d, $J = 8.6$ Hz, 1H), 2.99 – 3.02 (m, 1H), 3.74 (t, $J = 4.8$ Hz, 1H), 4.79 (dd, $J = 8.1$, 6.0 Hz, 1H), 4.57 (dd, $J = 4.5$, 0.8 Hz, 1H), 7.13 – 7.38 (m, 9H). ^{13}C NMR (100 MHz, CDCl_3): δ 22.3, 22.4, 28.3, 46.8,

¹ Murakami, M.; Igawa, H. *Chem. Comm.* **2002**, 4, 390-391

71.5, 122.3, 123.0, 126.6, 127.3, 127.5, 128.1, 128.6, 129.4, 133.4, 137.3, 138.3, 143.3. HRMS (EI) calcd. for $C_{19}H_{20}O$ (M^+): 264.1514; found: 264.1523.

(1S*,2R*)-1,2-dihydro-2,4-diphenyl-1-naphthalenol (16l) Yield: 96% (45.70 mg, 0.153 mmol). Colourless oil, R_f = 0.34 (EtOAc-Hexanes, 1:5). IR (CH_2Cl_2): 3551, 3424, 3058, 3027, 2906, 1600, 1493, 1451, 1265, 1157, 1112, 1074, 1032, 1006, 861, 773, 759, 737, 700 cm^{-1} . 1H NMR (400 MHz, $CDCl_3$): δ 3.89 (dd, J = 5.4, 4.6 Hz, 1H), 4.89 (d, J = 5.7 Hz, 1H), 6.08 (d, J = 4.4 Hz, 1H), 7.05 – 7.37 (m, 15H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 47.5, 71.6, 126.1, 126.8, 127.5, 128.1, 128.2, 128.3, 128.4, 128.9, 129.4, 133.5, 137.1, 137.8, 139.8, 140.4. HRMS (EI) calcd. for $C_{22}H_{18}O$ (M^+): 298.1358; found: 298.1351.

(3R*,4S*)-4-hydroxy-3-phenyl-3,4-dihydro-1-naphthalenecarboxylic acid methyl ester (16n) Yield: 77% (55.9 mg, 0.199 mmol). Light brown oil, R_f = 0.33 (EtOAc-Hexanes, 1:4). IR (CH_2Cl_2): 3465, 3060, 3029, 2951, 1717, 1493, 1452, 1436, 1286, 1265, 1198, 1171, 1075, 1034, 1010, 887, 785, 773, 755, 736, 702 cm^{-1} . 1H NMR (400 MHz, $CDCl_3$): δ 1.58 (m, 1H), 3.77 (s, 3H), 3.81 (t, J = 4.7 Hz, 1H), 4.74 (t, J = 6.1 Hz, 1H), 7.11 – 7.30 (m, 9H), 7.87 (d, J = 7.7 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 47.5, 52.1, 71.1, 126.6, 127.0, 127.7, 128.6, 128.7, 128.8, 129.3, 129.4, 130.6, 136.4, 136.9, 140.1, 166.5. HRMS (EI) calcd. for $C_{18}H_{16}O_3$ (M^+): 280.1099; found: 280.1108.

(3R*,4S*)-4-hydroxy-3-phenyl-3,4-dihydro-1-naphthalenecarboxylic acid (16o) Yield: 66% (41.2 mg, 0.155 mmol). Clear oil, R_f = 0.48 (EtOAc-Hexanes, 2:5). IR (CH_2Cl_2): 3062, 3028, 2948, 1728, 1493, 1460, 1222, 1026, 759, 735, 701 cm^{-1} . 1H NMR (400 MHz, $CDCl_3$): δ 2.24

(dd, $J = 11.5, 4.4$ Hz, 1H), 2.40 (dd, $J = 11.9, 8.5$ Hz, 1H), 2.94 (dd, $J = 8.4, 4.4$ Hz, 1H), 5.32 (s, 1H), 7.17 – 7.36 (m, 8H), 7.50 – 7.52 (m, 1H), 10.45 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3): δ 42.2, 47.3, 85.5, 86.4, 119.2, 119.5, 126.9, 127.3, 127.6, 127.8, 128.8, 143.1, 143.6, 145.0, 174.6. HRMS (EI) calcd. for $\text{C}_{17}\text{H}_{14}\text{O}_3$ (M^+): 266.1722; found: 266.0954.

(1S*,2R*)-1,2-dihydro-4-(3-hydroxypropan-2-yl)-2-diphenyl-1-naphthalenol (16p) Yield: 55% (33.6 mg, 0.120 mmol). Pink oil, $R_f = 0.63$ (EtOAc-Hexanes, 1:1). IR (CH_2Cl_2): 3454, 3062, 3026, 2983, 1493, 1460, 1371, 1181, 1156, 1030, 1015, 950, 896, 759, 747, 701, 659 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 1.46 (s, 3H), 1.52 (s, 3H), 1.93 (dd, $J = 8.8, 2.8$ Hz, 1H), 2.17 – 2.22 (m, 2H), 2.87 (dd, 8.7, 4.0 Hz, 1H), 5.15 (s, 1H), 7.12 – 7.33 (m, 9H). ^{13}C NMR (100 MHz, CDCl_3): δ 25.7, 26.7, 37.7, 48.2, 71.2, 84.6, 94.6, 119.1, 120.0, 126.5, 126.6, 126.7, 127.6, 128.6, 145.0, 145.4, 147.3. HRMS (ESI) calcd. for $\text{C}_{19}\text{H}_{20}\text{O}_2$ (M-H): 279.1385; found 279.1375.

(1S*,2R*)-1,2-dihydro-4-trimethylsilane-2-diphenyl-1-naphthalenol (16q): Yield: 54% (24.5 mg, 0.083 mmol). Light brown oil, $R_f = 0.53$ (EtOAc-Hexanes, 3:7). IR (NaCl): 3558, 3439, 306, 3026, 2954, 2897, 1591, 1492, 1451, 1380, 1250, 1192, 1073, 1033, 997, 977, 952, 835, 771, 51, 700 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 0.15 (s, 9H), 1.29 (d, $J = 8.2$ Hz, 1H), 3.63 (dd, $J = 4.3, 5.8$ Hz, 1H), 4.66 (dd, $J = 5.6, 8.4$ Hz, 1H), 6.28 (d, $J = 4.0$ Hz, 1H), 7.21-7.06 (m, 9H). ^{13}C NMR (100 MHz, CDCl_3): δ 0.3, 48.2, 71.6, 127.0, 127.2, 127.5, 127.6, 128.3, 129.5, 135.2, 136.5, 138.3, 139.0, 139.9. HRMS (ESI) calcd. for $\text{C}_{19}\text{H}_{22}\text{OSi}$ (M+Na): 317.1338; found: 317.1348.

400 MHz ¹H NMR Spectrum of **16a** in CDCl₃

100 MHz ^{13}C NMR Spectrum of **16a** in CDCl_3

400 MHz ^1H NMR Spectrum of **16b** in CDCl_3

100 MHz ¹³C NMR Spectrum of **16b** in CDCl₃

400 MHz ^1H NMR Spectrum of **16c** in CDCl_3

100 MHz ^{13}C NMR Spectrum of **16c** in CDCl_3

400 MHz ^1H NMR Spectrum of **16d** in CDCl_3

400 MHz ^1H NMR Spectrum of **16e** in CDCl_3

100 MHz ¹³C NMR Spectrum of **16e** in CDCl₃

400 MHz ^1H NMR Spectrum of **16f** in CDCl_3

100 MHz ^{13}C NMR Spectrum of **16f** in CDCl_3

400 MHz ¹H NMR Spectrum of **16g** in CDCl₃

100 MHz ^{13}C NMR Spectrum of **16g** in CDCl_3

400 MHz ¹H NMR Spectrum of **16h** in CDCl₃

100 MHz ^{13}C NMR Spectrum of **16h** in CDCl_3

400 MHz ^1H NMR Spectrum of **16i** in CDCl_3

100 MHz ^{13}C NMR Spectrum of **16i** in CDCl_3

400 MHz ^1H NMR Spectrum of **16j** in CDCl_3

100 MHz ^{13}C NMR Spectrum of **16j** in CDCl_3

400 MHz ^1H NMR Spectrum of **16k** in CDCl_3

100 MHz ¹³C NMR Spectrum of **16k** in CDCl₃

400 MHz ^1H NMR Spectrum of **16l** in CDCl_3

100 MHz ^{13}C NMR Spectrum of **16l** in CDCl_3

400 MHz ^1H NMR Spectrum of **16n** in CDCl_3

100 MHz ^{13}C NMR Spectrum of **16n** in CDCl_3

400 MHz ¹H NMR Spectrum of **16o** in CDCl₃

100 MHz ^{13}C NMR Spectrum of **16o** in CDCl_3

400 MHz ¹H NMR Spectrum of **16p** in CDCl₃

100 MHz ^{13}C NMR Spectrum of **16p** in CDCl_3

400 MHz ^1H NMR Spectrum of **16q** in CDCl_3