Supporting Information

for

IBX Oxidations for the Synthesis of Substituted 2H-Pyrans

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Part 1. General Information

Unless otherwise noted, all reactions were performed in flame-dried or oven-dried glassware under argon atmosphere. All non-volatile samples were pumped to constant weight at ambient temperature (0.2 mmHg) following removal of solvents by rotary evaporation. Non-aqueous solutions were transferred using syringe techniques under argon atmosphere. Bulk grade hexanes and ethyl acetate for chromatography were distilled prior to use. Tetrahydrofuran (THF), dimethylformamide (DMF) and dichloromethane (CH$_2$Cl$_2$) were obtained anhydrous by degassing with argon and then passing through activated alumina columns to remove water. Diisopropylethylamine (DIPEA) was distilled from CaH$_2$ under dry argon immediately before use. Commercial reagents were used as obtained from vendors unless otherwise specified. Air-sensitive reagents were handled inside a glovebox facility.

Reactions were monitored by standard thin-layer chromatography (TLC) techniques using silica gel 60 F254 on pre-coated glass plates (0.25 mm thickness). Following the run, TLC plates were visualized under UV light and/or by application of appropriate stains (p-anisaldehyde or cerium ammonium molybdate or potassium permanganate). Flash column chromatography was performed with Silica-P Flash Silica Gel (ultra-pure 40-63 μm).

Proton nuclear magnetic resonance (1H NMR) spectra were recorded on Varian VXR 400 (400 MHz), Varian INOVA 400 (400 MHz) or Varian 500 (500 MHz) instruments. Carbon nuclear magnetic resonance (13C NMR) spectra were measured using Varian VXR 400 (101 MHz), Varian INOVA 400 (101 MHz) or Varian 500 (125 MHz) instruments. NMR coupling constants and signal patterns are reported as J values in Hz and δ values in parts per million (ppm). 1H and 13C NMR spectra are internally referenced to residual solvent signals (CDCl$_3$ referenced to δ 7.26 and 77.16 ppm respectively). The following abbreviations were used to indicate the multiplicities: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. High resolution mass measurements (HRMS) were obtained by EI/CI or ESI methods. Optical
rotation data were obtained on polarimeter and are reported in terms of degree of rotation of plane-polarized light. IR spectra are reported in terms of frequency of absorption (cm\(^{-1}\)).

**Part 2. A General Procedure for Preparation of Starting 3,5-Hexadien-1-ols**

An Example of the General Experimental Procedure for Preparation of \(\alpha\)-Linked Bisenones of Table 1.

To a dried round bottom flask under argon was added LiCl (0.16 g, 3.9 mmol, 4 equiv). The LiCl was then flame-dried and once cooled to room temperature, Pd\(_2\)dba\(_3\) (0.13 g, 0.14 mmol, 0.15 equiv) was added followed by vinyl iodide A (0.3 g, 0.97 mmol, 1.0 equiv) as a solution in DMF (7 ml). The reaction mixture was allowed to stir for 10 minutes. Stannane B (0.5 g, 0.97 mmol, 1.0 equiv) was then added as a solution in DMF (3 ml) followed by DIPEA (0.33 ml, 1.9 mmol, 2.0 equiv). The reaction mixture was heated to 60 °C, stirred and the progress was followed by thin-layer chromatography. The reaction was quenched with water and diluted with Et\(_2\)O. The organic phase was separated, and aqueous layer was extracted with Et\(_2\)O (3 \(\times\) 4 ml). The combined organic phases were washed with brine, dried over anhydrous MgSO\(_4\), filtered and concentrated under reduced pressure. The crude oil was purified via flash column chromatography on silica gel (20% Et\(_2\)O/hexanes) to yield diene C as a yellow oil (0.28 g, 72%).
Silyl group deprotection was undertaken via mild hydrolysis.

To a solution of silylether C (0.28 mg, 0.67 mmol, 1.0 equiv) in MeOH (1.5 ml) was added pTsOH (25 mg, 0.13 mmol, 0.2 equiv) at room temperature and the reaction mixture was stirred for 2 hours. Concentration under reduced pressure and flash column chromatography (40% EtOAc/hexanes) gave the 3,5-hexadiene-1-ol 6 a colorless oil (140 mg, 70 %): Rf 0.4 (50% EtOAc/hexanes).

Part 3. Characterization Data for the 3,5-Hexdien-1-ols of Table 1

Compound 6. Rf 0.4 in 50% EtOAc/hexanes; IR (neat) 3436, 2980, 2937, 2875, 1693, 1669, 1373, 1301, 1275, 1222, 1014, 726, 701 cm\(^{-1}\); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.31 (m, 3H), 7.23 (m, 2H), 6.98 (t, \(J = 7.5\) Hz, 1H), 3.84 (m, 4H), 2.61 (t, \(J = 5.0\) Hz, 1H), 2.55 (m, 2H), 2.38 (s, 3H), 1.98 (s, 3H), 0.80 (t, \(J = 7.5\) Hz, 3H); \(^13\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 197.7, 168.6, 149.2, 143.0, 142.6, 141.2, 128.2, 127.8, 126.9, 126.2, 60.9, 60.9, 33.4, 26.6, 23.2, 13.4; HRMS (ESI) Calcd for C\(_{18}\)H\(_{22}\)O\(_4\)Na [M+Na]\(^{+}\) 325.1410, found: 325.1408.

Compound 7. Rf 0.3 in 30% EtOAc/hexanes; IR (neat) 3468, 2957, 2927, 2854, 1719, 1716, 1387, 1201, 1096, 1026, 759, 700 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.52–7.42 (m, 2H), 7.42–7.31 (m, 3H), 6.72 (t, \(J = 7.8\) Hz, 1H), 6.24 (s, 1H), 5.55 (s, 1H), 4.43 (d, \(J = 11.0\) Hz, 2H), 4.17 (q, \(J = 7.1\) Hz, 2H), 3.70 (d, \(J = 11.0\) Hz, 2H), 3.37 (q, \(J = 5.8\) Hz, 2H), 2.48 (q, \(J = 6.4\) Hz, 2H), 2.30 (s, 3H), 1.80 (t, \(J = 6.6\) Hz, 1H), 1.26–1.21 (m, 3H), 1.23 (s, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 198.1, 171.1, 166.1, 150.1, 144.6, 142.4, 129.4, 128.8, 128.1, 125.6, 101.1, 77.1, 61.0, 60.8, 37.0, 33.7, 26.1, 17.8, 13.9; HRMS (ESI) Calcd for C\(_{22}\)H\(_{28}\)O\(_6\)Na [M+Na]\(^{+}\) 411.1778, found: 411.1778.

Compound 8. Rf 0.4 in 30% EtOAc/hexanes; IR (neat) 3435, 2958, 2926, 1766, 1721, 1674, 1367, 1194, 1026 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 6.79 (t, \(J = 7.6\) Hz, 1H), 5.63 (s, 1H), 4.18 (q, \(J = 7.1\) Hz, 2H), 3.77 (t, \(J = 6.1\) Hz, 2H), 2.56 (q, \(J = 6.6\) Hz, 2H), 2.31 (s, 3H), 1.28 (t, \(J = 7.2\) Hz, 3H), 1.25–1.23 (m, 1H), 1.19 (s, 9H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 198.0,

Compound 9. Rƒ 0.2 in 30% EtOAc/hexanes; [α]²²D + 36.7 (c = 1 CHCl₃); IR (neat) 3467, 2960, 2931, 2858, 1716, 1672, 1427, 1368, 1215, 1108, 1079, 703 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.65 (m, 4H), 7.45–7.30 (m, 6H), 6.75 (t, J = 7.5 Hz, 1H), 6.03 (d, J = 7.8 Hz, 1H), 5.32 (dq, J = 7.8, 6.2 Hz, 1H), 3.96 (q, J = 7.1 Hz, 2H), 3.65 (t, J = 6.1 Hz, 2H), 2.28 (m, J = 7.5, J = 6.1 Hz, 2H), 2.22 (s, 3H), 1.33 (d, J = 6.2 Hz, 3H), 1.06 (s, 9H), 1.04 (t, J = 7.1 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 190.8, 175.1, 151.9, 145.0, 130.3, 129.2, 128.0, 119.0, 113.7, 72.4, 68.7, 55.2, 31.8, 31.5, 29.9, 29.1, 27.0; HRMS (ESI) Calcd for C₂₉H₃₈O₅SiNa [M+Na]⁺ 517.2381, found: 517.2378.

Compound 10. Rƒ 0.2 in 30% EtOAc/hexanes; IR (neat) 3432, 2977, 2937, 2875, 1714, 1682, 1371, 1205, 1035 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 6.09 (m, 1H), 4.20 (m, 2H), 3.78 (m, 2H), 2.82 (q, J = 6.4 Hz, 2H), 2.33 (q, J = 7.6 Hz, 1H), 2.18 (s, 3H), 2.07 (q, J = 7.6 Hz, 1H), 1.99 (s, 2H), 1.71 (s, 1H), 1.26 (m, 3H), 1.07 (t, J = 7.6 Hz, 1H), 0.97 (t, J = 7.6 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 201.6, 201.3, 167.3, 150.8, 150.4, 144.4, 144.2, 135.3, 134.9, 133.0, 132.8, 129.4, 61.7, 61.7, 61.0, 32.9, 32.9, 30.4, 30.4, 30.2, 28.7, 27.2, 20.8, 19.7, 14.2, 13.05, 12.1; HRMS (ESI) Calcd for C₁₄H₂₂O₄Na [M+Na]⁺ 277.1410, found: 277.1410.

Compound 11. Rƒ 0.3 in 30% EtOAc/hexanes; IR (neat) 3461, 2939, 2865, 1714, 1672, 1514, 1245, 1207, 1101, 1037 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.24 (d, J = 8.0 Hz, 2H), 6.86 (d, J = 8.0 Hz, 2H), 6.76 (t, J = 7.6 Hz, 1H), 6.08 (t, J = 7.2 Hz, 1H), 4.43 (s, 2H), 4.16 (q, J = 7.2 Hz, 2H), 3.79 (s, 3H), 3.66 (t, J = 6.4 Hz, 2H), 3.62 (t, J = 5.6 Hz, 2H), 2.95 (m, 2H), 2.45 (m, 2H), 2.29 (s, 3H), 1.84 (br, 1H), 1.23 (t, J = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 198.3, 166.3, 159.3, 145.5, 143.7, 141.3, 130.3, 129.4, 128.7, 113.9, 72.6, 69.2, 61.3, 60.7, 55.4, 33.5, 30.1, 26.3, 14.2; HRMS (ESI) Calcd for C₂₁H₂₈O₆Na [M+Na]⁺ 399.1784, found: 399.1773.
Compound 12. R<sub>f</sub> 0.3 in 30% EtOAc/hexanes; IR (neat) 3452, 2968, 2865, 1720, 1666, 1514, 1251, 1207, 1091, 1037 cm<sup>–1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.24 (d, J = 8.0 Hz, 2H), 6.86 (d, J = 8.0 Hz, 2H), 6.81 (t, J = 7.6 Hz, 1H), 6.06 (t, J = 7.2 Hz, 1H), 4.44 (s, 2H), 4.15 (q, J = 7.2 Hz, 2H), 3.89 (m, 1H), 3.80 (s, 3H), 3.61 (t, J = 6.4 Hz, 2H), 2.96 (m, 2H), 2.37 (m, 2H), 2.29 (s, 3H), 1.86 (br, 1H), 1.23 (t, J = 7.2 Hz, 3H), 1.17 (d, J = 6.0 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 198.4, 166.2, 159.2, 145.3, 143.3, 141.0, 130.3, 129.4, 128.7, 113.8, 72.5, 69.0, 67.0, 60.6, 55.3, 39.4, 30.1, 26.2, 23.4, 14.1; HRMS (ESI) Calcd for C<sub>22</sub>H<sub>30</sub>O<sub>6</sub>Na [M+Na]<sup>+</sup> 413.1924, found: 413.1925.

Compound 13. R<sub>f</sub> 0.4 in 50% EtOAc/hexanes; IR (neat) 3480, 2980, 2897, 1706, 1666, 1488, 1445, 1259, 1241, 1445, 1259, 1241, 1209, 1037, 929 cm<sup>–1</sup>; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 6.95 (t, J = 7.6 Hz, 1H), 6.79–6.72 (m, 2H), 6.69 (dd, J = 7.9, 1.7 Hz, 1H), 5.95 (s, 2H), 3.91 (q, J = 7.1 Hz, 2H), 3.81 (t, J = 6.1 Hz, 2H), 2.56 (s, 1H), 2.51 (d, J = 6.1 Hz, 2H), 2.36 (s, 3H), 1.93 (s, 3H), 0.93 (t, J = 7.1 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 197.6, 168.6, 148.3, 147.4, 147.2, 142.8, 141.1, 136.2, 126.0, 120.5, 108.0, 107.7, 101.1, 60.8, 60.8, 33.3, 26.5, 23.0, 13.6. HRMS (ESI) Calcd for C<sub>19</sub>H<sub>22</sub>O<sub>6</sub>Na [M+Na]<sup>+</sup> 369.1416, found: 369.1417.

**Part 4. A General Procedure for IBX Oxidations to 2H-Pyrans**

To a solution of alcohol 6 (50 mg, 0.16 mmol, 1.0 equiv) in DMSO (1.6 ml) was added IBX (153 mg, 0.54 mmol, 3.25 equiv) at room temperature. The reaction was stirred for approximately 8 hours and progress was followed by thin-layer chromatography. Reactions were quenched by addition of H<sub>2</sub>O (5 ml) and diluted with Et<sub>2</sub>O (2 ml). The combined organic layers were dried over anhydrous MgSO<sub>4</sub>, filtered and concentrated under reduced pressure and purified via flash column chromatography to yield pyran 14 (42 mg, 81%): R<sub>f</sub> 0.9 (1:1 EtOAc/ hexanes).
Part 5. Characterization Data for the 2H-Pyrans of Table 1

Compound 14. Rf 0.9 in 50% EtOAc/hexanes; IR (neat) 3069, 2984, 2937, 2851, 2747, 1719, 1690, 1364, 1296, 1256, 1177, 1046, 700 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 9.31 (s, 1H), 7.55 (d, J = 6.5 Hz, 2H), 7.31 (m, 3H), 6.36 (s, 1H), 4.19 (m, 2H), 2.40 (s, 3H), 1.98 (t, J = 7.5 Hz, 3H), 1.94 (s, 3H); ¹⁳C NMR (100 MHz, CDCl₃) δ 197.1, 185.3, 165.3, 151.6, 141.6, 135.4, 132.6, 128.6, 128.2, 126.6, 114.0, 82.2, 62.1, 28.8, 25.9, 13.7; HRMS (ESI) Calcd for C₁₈H₁₈O₅Na [M+Na]⁺ 337.1046, found: 337.1044.

Compound 15. Rf 0.5 in 50% EtOAc/hexanes; IR (neat) 2922, 2851, 1692, 1457, 1389, 1262, 1174, 1099, 1016, 758, 699 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 9.29 (s, 1H), 7.50–7.41 (m, 2H), 7.41–7.30 (m, 3H), 6.02 (s, 1H), 5.97 (s, 1H), 5.46 (s, 1H), 4.44 (dd, J = 11.9, 3.0 Hz, 1H), 4.15 (ddt, J = 16.2, 9.2, 5.0 Hz, 3H), 3.57 (dd, J = 12.2, 7.9 Hz, 2H), 2.18 (s, 3H), 1.15 (t, J = 7.2 Hz, 3H), 0.90 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 200.8, 184.5, 164.7, 153.5, 142.6, 137.8, 128.8, 127.9, 126.1, 120.3, 112.5, 101.9, 77.1, 73.0, 72.4, 61.9, 41.6, 29.6, 29.0, 16.6, 13.4; HRMS (ESI) Calcd for C₂₂H₂₄O₇Na [M+Na]⁺ 423.1414, found: 423.1415.

Compound 16. Rf 0.5 in 30% EtOAc/hexanes; IR (neat) 3050, 2958, 2931, 2893, 1693, 1365, 1259, 1171, 1090, 1030, 835 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 9.29 (s, 1H), 5.97 (s, 1H), 5.19 (s, 1H), 4.24 (m, J = 7.3 Hz, 2H), 2.39 (s, 3H), 1.30 (t, J = 7.2 Hz, 3H), 0.96 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 201.0, 184.6, 164.0, 159.3, 134.1, 129.7, 127.5, 119.4, 112.2, 78.5, 69.7, 61.8, 25.1, 13.8; HRMS (ESI) Calcd for C₁₅H₂₀O₅Na [M+Na]⁺ 303.1203, found: 303.1203.

Compound 17 (major diastereomer). Rf 0.5 in 30% EtOAc/hexanes; [α]²²D + 1.5 (c = 1 CHCl₃); IR (neat) 3071, 2958, 2931, 2893, 1695, 1427, 1258, 1107, 907, 702, 505 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 9.26 (s, 1H), 7.66 (m, 4H), 7.53 (m, 6H), 6.06 (s, 1H), 5.34 (d, J = 6.5 Hz, 1H), 4.20 (m, J = 9.3 Hz, 2H), 3.96 (p, J = 9.1, 6.5 Hz, 1H), 2.43 (s, 3H), 1.33 (d, J = 9.1, 3H), 1.26 (s, 9H), 1.03 (t, J = 9.3 Hz, 3H), 0.99 (s, 9H); ¹³C NMR (125 MHz, CDCl₃) δ 201.0, 184.6, 164.0, 152.7, 143.2, 135.7, 134.1, 129.7, 127.5, 119.4, 112.2, 78.5, 69.7, 61.8,

**Compound 18.** R_f 0.8 in 30% EtOAc/hexanes; IR (neat) 3090, 2977, 2933, 2857, 2748, 1721, 1694, 1369, 1308, 1232, 1199, 1018 cm\(^{-1}\); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 9.29 (s, 1H), 6.57 (s, 1H), 4.27 (q, \(J = 7.5\) Hz, 2H), 2.42 (s, 3H), 1.98 (m, 1H), 1.71 (m, 1H), 1.50 (s, 3H), 1.33 (t, \(J = 7.5\) Hz, 3H), 0.99 (t, \(J = 7.5\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 202.0, 185.5, 163.0, 150.5, 150.2, 121.4, 114.5, 82.2, 62.3, 32.0, 30.5, 22.8, 14.1, 7.6; HRMS (ESI) Calcd for C\(_{18}\)H\(_{22}\)O\(_4\)Na [M+Na]^+ 325.1410, found: 325.1408.

**Compound 19.** R_f 0.4 in 30% EtOAc/hexanes; IR (neat) 2983, 2860, 1714, 1699, 1514, 1265 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 9.29 (s, 1H), 7.24 (d, \(J = 8.0\) Hz, 2H), 6.86 (d, \(J = 8.0\) Hz, 2H), 6.11 (s, 1H), 5.55 (dd, \(J = 10.0, 4.0\) Hz, 1H), 4.42 (s, 2H), 4.19 (m, 2H), 3.79 (s, 3H), 3.59 (m, 1H), 3.52 (m, 1H), 2.37 (s, 3H), 2.12 (m, 1H), 1.88 (m, 1H), 1.84 (br, s, 1H), 1.26 (t, \(J = 7.2\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 201.4, 185.2, 163.4, 159.2, 152.0, 142.6, 130.3, 129.3, 121.6, 113.8, 112.4, 72.8, 72.7, 65.0, 62.0, 55.4, 32.0, 29.7, 13.9; HRMS (ESI) Calcd for C\(_{21}\)H\(_{24}\)O\(_7\)Na [M+Na]^+ 411.1419, found: 411.1403.

**Compound 20.** R_f 0.7 in 50% EtOAc/hexanes; IR (neat) 2935, 2871, 1708, 1608, 1514, 1369, 1265, 1091 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.25 (d, \(J = 8.0\) Hz, 2H), 6.87 (d, \(J = 8.0\) Hz, 2H), 6.18 (s, 1H), 5.55 (dd, \(J = 10.0, 4.0\) Hz, 1H), 4.46 (d, A of AB, \(J_{AB} = 12.0\) Hz, 1H), 4.39 (d, B of AB, \(J_{AB} = 12.0\) Hz, 1H), 4.22 (q, \(J = 7.2\) Hz, 2H), 4.19 (m, 2H), 3.80 (s, 3H), 3.57 (m, 1H), 3.51 (m, 1H), 2.36 (s, 3H), 2.22 (s, 3H), 2.15 (m, 1H), 1.85 (m, 1H), 1.26 (t, \(J = 7.2\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 202.0, 193.7, 163.5, 159.3, 151.5, 144.0, 130.3, 129.3, 118.7, 113.9, 103.3, 72.8, 72.6, 65.1, 61.7, 55.4, 31.5, 29.7, 25.8, 14.0; HRMS (ESI) Calcd for C\(_{22}\)H\(_{26}\)O\(_7\)Na [M+Na]^+ 425.1576, found: 425.1573.

**Compound 21.** R_f 0.7 in 50% EtOAc/hexanes; IR (neat) 3078, 2985, 2915, 2848, 1719, 1692, 1488, 1248, 1038 cm\(^{-1}\); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 9.29 (s, 1H), 7.04 (d, \(J = 2.0\) Hz,
1H), 7.00 (dd, J = 8.2, 2.0 Hz, 1H), 6.71 (d, J = 8.2 Hz, 1H), 6.33 (s, 1H), 5.92 (s, 2H), 4.27–4.08 (m, 2H), 2.38 (s, 3H), 1.88 (s, 3H), 1.21 (t, J = 7.2 Hz, 3H); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 196.9, 185.1, 165.4, 151.3, 147.7, 147.6, 135.4, 132.3, 119.9, 113.6, 107.6, 106.9, 101.2, 81.9, 62.0, 28.7, 25.9, 13.7; HRMS (ESI) Calcd for \(\text{C}_{19}\text{H}_{18}\text{O}_{7}\text{Na}^{+}\) [M+Na] \(+\) 381.0945, found: 381.0945.