Supporting Information

for

# IBX Oxidations for the Synthesis of Substituted 2*H*-Pyrans

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## Part 1. General Information

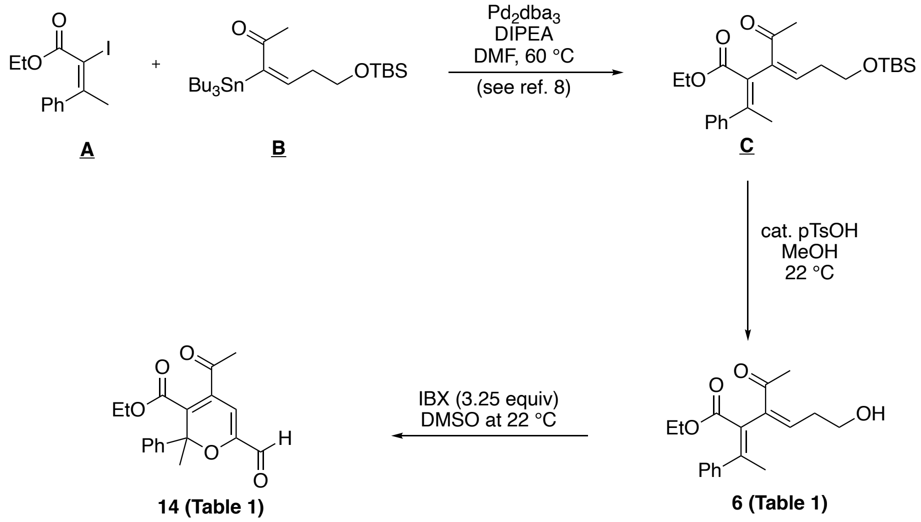
Unless otherwise noted, all reactions were performed in flame-dried or oven-dried glassware under argon atmosphere. All non-volatile samples were pumped to constant weight at ambient temperature (0.2 mmHg) following removal of solvents by rotary evaporation. Non-aqueous solutions were transferred using syringe techniques under argon atmosphere. Bulk grade hexanes and ethyl acetate for chromatography were distilled prior to use. Tetrahydrofuran (THF), dimethylformamide (DMF) and dichloromethane ($CH_2Cl_2$) 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 $\mu$m).

Proton nuclear magnetic resonance ($^1$H 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 ($^{13}$C 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 $\delta$ values in parts per million (ppm). $^1$H and $^{13}$C NMR spectra are internally referenced to residual solvent signals ($CDCl_3$ referenced to $\delta$ 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 α-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, $Pd_2dba_3$(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 $Et_2O$. The organic phase was separated, and aqueous layer was extracted with $Et_2O$ (3 × 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% $Et_2O$/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 %): R*f* 0.4 (50% EtOAc/ hexanes).

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

Compound **6**. R*f* 0.4 in 50% EtOAc/hexanes; IR (neat) 3436, 2980, 2937, 2875, 1693, 1669, 1373, 1301, 1275, 1222, 1014, 726, 701cm$^{-1}$; $^1$H NMR (500 MHz, $CDCl_3$) δ 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, $CDCl_3$) δ 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 $C_{18}H_{22}O_4Na$ $[M+Na]^+$ 325.1410, found: 325.1408.

Compound **7**. R*f* 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, $CDCl_3$) δ 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, $CDCl_3$) δ 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 $C_{22}H_{28}O_6Na$ $[M+Na]^+$ 411.1778, found: 411.1778.

Compound **8**. R*f* 0.4 in 30% EtOAc/hexanes; IR (neat) 3435, 2958, 2926, 1766, 1721, 1674, 1367, 1194, 1026 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 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, $CDCl_3$) δ 198.0, 168.1, 143.6, 141.4, 128.3, 126.3, 61.2, 61.0, 33.9, 33.0, 29.7, 26.7, 13.9; (ESI) Calcd for $C_{15}H_{24}O_4Na$ $[M+Na]^+$ 291.1567, found: 291.1567.

Compound **9**. R*f* 0.2 in 30% EtOAc/hexanes; $[\alpha]^{22}_D$ + 36.7 (c = 1 $CHCl_3$); IR (neat) 3467, 2960, 2931, 2858, 1716, 1672, 1427, 1368, 1215, 1108, 1079, 703 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) 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), 1.25 (t, *J* = 7.1 Hz, 1H), 1.06 (s, 9H), 1.04 (t, *J* = 7.1 Hz, 3H); $^{13}C$ NMR (100 MHz, $CDCl_3$) 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 $C_{29}H_{38}O_5SiNa$ $[M+Na]^+$ 517.2381, found: 517.2378.

Compound **10**. R*f* 0.2 in 30% EtOAc/hexanes; IR (neat) 3432, 2977, 2937, 2875, 1714, 1682, 1371, 1205, 1035 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) 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); $^{13}C$ NMR (100 MHz, $CDCl_3$) 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 $C_{14}H_{22}O_4Na$ $[M+Na]^+$ 277.1410, found: 277.1410.

Compound **11**. R*f* 0.3 in 30% EtOAc/hexanes; IR (neat) 3461, 2939, 2865, 1714, 1672, 1514, 1245, 1207, 1101, 1037 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) 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); $^{13}C$ NMR (100 MHz, $CDCl_3$) 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 $C_{21}H_{28}O_6Na$ $[M+Na]^+$ 399.1784, found: 399.1773.

Compound **12**. R*f* 0.3 in 30% EtOAc/hexanes; IR (neat) 3452, 2968, 2865, 1720, 1666, 1514, 1251, 1207, 1091, 1037 cm$^{-1}$; $^{1}$H NMR (400 MHz, $CDCl_3$) 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); $^{13}$C NMR (100 MHz, $CDCl_3$) 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 $C_{22}H_{30}O_6Na$ $[M+Na]^+$ 413.1924, found: 413.1925.

Compound **13**. R*f* 0.4 in 50% EtOAc/hexanes; IR (neat) 3480, 2980, 2897, 1706, 1668, 1488, 1445, 1259, 1241, 1445, 1259, 1241, 1209, 1037, 929 cm$^{-1}$; $^{1}$H NMR (500 MHz, $CDCl_3$) 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); $^{13}$C NMR (125 MHz, $CDCl_3$) 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 $C_{19}H_{22}O_6Na$ $[M+Na]^+$ 369.1416, found: 369.1417.

## Part 4. A General Procedure for IBX Oxidations to 2*H*-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 $H_2O$ (5 ml) and diluted with $Et_2O$ (2 ml). The combined organic layers were dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure and purified via flash column chromatography to yield pyran **14** (42 mg, 81%): R*f* 0.9 (1:1 EtOAc/ hexanes).

## Part 5. Characterization Data for the 2*H*-Pyrans of Table 1

Compound **14**. R*f* 0.9 in 50% EtOAc/hexanes; IR (neat) 3069, 2984, 2937, 2851, 2747, 1719, 1690, 1364, 1296, 1256, 1177, 1046, 700 cm$^{-1}$; $^1$H NMR (500 MHz, $CDCl_3$) 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); $^{13}$C NMR (100 MHz, $CDCl_3$) 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 $C_{18}H_{18}O_5Na$ $[M+Na]^+$ 337.1046, found: 337.1044.

Compound **15**. R*f* 0.5 in 50% EtOAc/hexanes; IR (neat) 2922, 2851, 1692, 1457, 1389, 1262, 1174, 1099, 1016, 758, 699 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) 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); $^{13}$C NMR (100 MHz, $CDCl_3$) 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 $C_{22}H_{24}O_7Na$ $[M+Na]^+$ 423.1414, found: 423.1415.

Compound **16**. R*f* 0.5 in 30% EtOAc/hexanes; IR (neat) 2956, 2923, 2852, 1693, 1365, 1259, 1171, 1090, 1030, 835 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) 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); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 200.8, 184.8, 165.1, 153.9, 143.4, 119.0, 111.8, 82.2, 61.8, 40.2, 29.5, 25.1, 13.8; HRMS (ESI) Calcd for $C_{15}H_{20}O_5Na$ $[M+Na]^+$ 303.1203, found: 303.1203.

Compound **17** (major diastereomer). R*f* 0.5 in 30% EtOAc/hexanes; $[\alpha]^{22}_D$ + 1.5 (c = 1 $CHCl_3$); IR (neat) 3071, 2958, 2931, 2893, 1695, 1427, 1258, 1107, 907, 702, 505 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) 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); $^{13}$C NMR (125 MHz, $CDCl_3$) 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, 29.6, 26.7, 19.7, 19.1, 13.7; HRMS (ESI) Calcd for $C_{29}H_{34}O_6NaSi$ $[M+Na]^+$ 529.2017, found: 529.2017.

Compound **18**. R*f* 0.8 in 30% EtOAc/hexanes; IR (neat) 3090, 2977, 2933, 2857, 2748, 1721, 1694, 1369, 1308, 1232, 1199, 1018 $cm^{-1}$; $^1H$ NMR (500 MHz, $CDCl_3$) δ 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, $CDCl_3$) δ 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 $C_{18}H_{22}O_4Na$ $[M+Na]^+$ 325.1410, found: 325.1408.

Compound **19**. R*f* 0.4 in 30% EtOAc/hexanes; IR (neat) 2983, 2860, 1714, 1699, 1514, 1265 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 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, $CDCl_3$) δ 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 $C_{21}H_{24}O_7Na$ $[M+Na]^+$ 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 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 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, $CDCl_3$) δ 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 $C_{22}H_{26}O_7Na$ $[M+Na]^+$ 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 $cm^{-1}$; $^1H$ NMR (500 MHz, $CDCl_3$) δ 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, $CDCl_3$) δ 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 $C_{19}H_{18}O_7Na$ $[M+Na]^+$ 381.0945, found: 381.0945.