Highly Stereoselective Synthesis of Tetrasubstituted Acyclic All-Carbon Olefins via Enol Tosylation and Suzuki–Miyaura Coupling

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### List of Compounds

#### Table S1. Ketones

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**Note:** 1a is commercially available.

#### Table S2. Tosylates

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**Table S3.** Boronic esters and zinc halides (all commercially available)

![Boronic esters and zinc halides](image)

**Table S4.** Tetrasubstituted olefins

![Tetrasubstituted olefins](image)
Materials and Methods

Unless stated otherwise, reactions were performed under an ambient atmosphere of nitrogen in 4-mL vials sealed with Teflon-lined caps. All solvents and commercially obtained reagents were used as received, unless specified otherwise. p-Toluenesulfonic anhydride was purchased from Sigma-Aldrich (catalog number 259764, lot number BCBQ2378V) and used directly. Thin-layer chromatography (TLC) was conducted with EMD silica gel 60 F254 pre-coated plates and visualized using UV light (254 nm). Flash column chromatography was performed with pre-packed RediSep silica gel columns on a CombiFlash ISCO system using i-PrOAc in heptanes as eluent. Specific gradients are listed under experiment procedures. Melting points were measured on a Büchi Melting Point B-540 apparatus. Analytical High Performance Liquid Chromatography (HPLC) analyses were performed with an Agilent 1200 Series or an Agilent 1260 Infinity Series HPLC instrument. Specific methods are listed under experimental procedures; retention times \( t_M \) and \( t_m \) refer to that of major and minor isomers, respectively. IR spectra were recorded on a Bruker Alpha Platinum-neat spectrometer and are reported in frequency of absorption (cm\(^{-1}\)). \(^1\)H NMR spectra were recorded on a Bruker 400 (at 400 MHz) and are reported relative to the residual solvent peak (\( \delta 7.26 \) for CDCl\(_3\)). Data for \(^1\)H NMR spectra are reported as follows: chemical shift (\( \delta \) ppm), multiplicity, coupling constant (Hz), and integration. \(^{13}\)C NMR spectra were recorded on a Bruker 400 (at 101 MHz), and are reported relative the residual solvent peak (\( \delta 77.0 \) for CDCl\(_3\)). Data for \(^{13}\)C NMR spectra are reported in terms of chemical shift (\( \delta \) ppm). \(^{19}\)F NMR spectra were recorded on a Bruker 400 (at 376 MHz). Data for \(^{19}\)F NMR spectra are reported in terms of chemical shift (\( \delta \) ppm). High Resolution Mass Spectroscopy (HRMS) data was obtained on an Agilent 6210 Time-of-Flight instrument, using electrospray ionization in positive or negative mode; HPLC analyses prior to ionization were performed with an Agilent 1290 instrument; the column used was ACE3 C18 HL 3x50mm, particle size 5 um; injection volume 2 \( \mu \)L; temperature 23 °C; flow rate 1 mL/min; mobile phase A = 0.1% formic acid in H\(_2\)O (in positive mode) or 10 mM ammonium acetate (in negative mode); mobile phase B = acetonitrile; gradient: 0–3' = 25% B; 3–7' = 95% B; 7–7.5' = 95–25% B; 7.5–10' = 25% B. Substrates that do not ionize under either ESI+ or ESI– conditions are also noted.
Experimental Procedures and Characterization Data

Tosylate Synthesis

To a nitrogen-purged septum-top 40 mL vial equipped with a stir bar was added LiHMDS (2 equiv, 0.90 M in toluene, 4.4 mL) and N,N-dimethylethylamine (DMEA, 2 equiv, 0.43 mL). The reaction mixture was placed in a 23 °C water bath and a solution of ketone 1 (2.0 mmol, dissolved in 2 mL PhMe) was added via syringe. After stirring for 20 min, a solution of TsO (2 equiv, 4.0 mmol, 1.31 g dissolved in 10 mL DCM) was added dropwise over 5 min. The reaction was stirred vigorously (1500 rpm) for another 20 min, and sampled via HPLC to verify completion. Analytical HPLC analyses were performed with an Agilent 1200 Series HPLC instrument; the column used was ChiralPak AD-H 4.6×150mm, particle size 5 um; injection volume 2 µL; temperature 20 °C; flow rate 1 mL/min; mobile phase A = hexanes; mobile phase B = isopropyl alcohol; gradient: 0–5' = 2–10% B; 5–10' = 10–20% B; 10–12' = 20–2% B; 12–13' = 2% B. After no further increase in conversion, the reaction was then diluted with 10 mL MTBE, 1 mL aq 1N NaOH, and 3 mL H2O, by which point the thick slurry turned clear. The biphasic solution was separated, and the aq layer was extracted with MTBE (3×5 mL). The combined organic layers were washed with brine (5 mL), dried over Na2SO4 (0.5 g), filtered, and concentrated in vacuo (40 °C). The crude residue was purified by silica gel column chromatography using 20–60% i-PrOAc in heptane if product 2 contains nitrogen (2j and 2s), or 0–10% i-PrOAc in heptane otherwise.

(€)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzenesulfonate (2a): Commercially available 1,2-diphenylbutan-1-one (1a, 2.0 mmol, 449 mg) was employed. Compound 2a was obtained as a white solid (668 mg, 88%); mp 95–96 °C; HPLC (tM = 3.66 min, tM = 5.01 min) indicated >99:1 isomer ratio; FTIR (neat, cm⁻¹) 3057, 2968, 2923, 2874, 1597; 1H NMR (400 MHz, CDCl3) δ 7.51–7.41 (m, 2H), 7.18–7.11 (m, 3H), 7.07 (d, J = 8.3 Hz, 2H), 7.04–6.94 (m, 3H), 6.87 (d, J = 4.4 Hz, 4H), 2.68 (q, J = 7.5 Hz, 2H), 2.34 (s, 3H), 0.92 (t, J = 7.5 Hz, 3H); 13C NMR (101 MHz, CDCl3) δ 144.3, 142.6, 138.3, 136.9, 134.4, 133.9, 129.9, 129.4, 129.2, 128.1, 128.0, 127.6, 127.3, 127.0, 26.2, 21.6, 12.0; HRMS (ESI–) m/z calculated for C23H22O3S ([M–H]–), 377.1212; found, 377.1242.

(€)-2-Phenyl-1-((p-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (2b): 2-Phenyl-1-((p-tolyl) butan-1-one (1b, 2.0 mmol, 477 mg) was employed. Compound 2b was obtained as a white solid (645 mg, 82%); mp 91–92 °C; HPLC (tM = 3.71 min, tM = 4.75 min) indicated 98:2 isomer ratio; FTIR (neat, cm⁻¹) 2972, 2924, 2872, 2855, 1595, 1363; 1H NMR (400 MHz, CDCl3) δ 7.53–7.43 (m, 2H), 7.16 (dd, J = 6.0, 1.5 Hz, 3H), 7.08 (d, J = 8.1 Hz, 2H), 7.05 –6.98 (m, 2H), 6.76 (d, J = 8.2 Hz, 2H), 6.69 (d, J = 8.0 Hz, 2H), 2.65 (q, J = 7.5 Hz, 2H), 2.36 (s, 3H), 2.16 (s, 3H), 0.90 (t, J = 7.5 Hz, 3H); 13C NMR (101 MHz, CDCl3) δ 144.2, 142.8, 138.5, 137.4, 136.1, 134.5, 131.0, 129.7, 129.4, 129.1, 128.1, 127.6, 127.3, 127.0, 26.2, 21.6, 12.0; HRMS (ESI–) m/z calculated for C23H20O3S ([M–H]–), 391.1368; found, 391.1368.
(E)-1-(4-Methoxyphenyl)-2-phenylbut-1-en-1-y1 4-methylbenzenesulfonate (2c): 1-(4-Methoxyphenyl)-2-phenylbutan-1-one (1c, 2.0 mmol, 509 mg) was employed. Compound 2c was obtained as a white solid (542 mg, 66%); mp 89–90 °C; HPLC (tM = 4.99 min, tM = 6.04 min) indicated 98:2 isomer ratio; FTIR (neat, cm⁻¹) 3016, 2960, 2932, 1608; ¹H NMR (400 MHz, CDCl₃) δ 7.52–7.45 (m, 2H), 7.20–7.12 (m, 3H), 7.10 (t, J = 8.2 Hz, 2H), 7.08–6.98 (m, 2H), 6.84–6.75 (m, 2H), 3.66 (s, 3H), 2.66 (q, J = 7.5 Hz, 2H), 2.36 (s, 3H), 0.91 (t, J = 7.5 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 158.9, 144.1, 142.6, 138.5, 135.6, 134.5, 131.2, 129.4, 129.1, 128.1, 128.0, 126.9, 126.3, 112.8, 55.0, 26.0, 21.5, 12.0; HRMS (ESI−) m/z calculated for C₂₄H₂₄O₄S ([M–H]−), 407.1317; found, 407.1327.

(OTs)

(E)-1-(4-Chlorophenyl)-2-phenylbut-1-en-1-y1 4-methylbenzenesulfonate (2d): 1-(4-Chlorophenyl)-2-phenylbutan-1-one (1d, 2.0 mmol, 517 mg) was employed. Compound 2d was obtained as a white solid (612 mg, 74%); mp 85–90 °C; HPLC (tM = 3.76 min, tM = 4.99 min) indicated 98:2 isomer ratio; FTIR (neat, cm⁻¹) 3050, 2987, 2923, 1598, 1487, 1347; ¹H NMR (400 MHz, CDCl₃) δ 7.48 (d, J = 8.4 Hz, 2H), 7.18 (dd, J = 5.1, 1.9 Hz, 3H), 7.12 (dt, J = 7.8, 0.7 Hz, 2H), 7.05–6.96 (m, 2H), 6.85 (d, J = 8.7 Hz, 2H), 6.78 (d, J = 8.6 Hz, 2H), 2.67 (q, J = 7.5 Hz, 2H), 2.39 (s, 3H), 0.91 (t, J = 7.5 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 144.7, 141.4, 137.9, 137.7, 134.2, 133.5, 132.5, 131.1, 129.31, 129.27, 128.3, 128.0, 127.6, 127.0, 26.2, 21.6, 11.9; HRMS (ESI−) m/z calculated for C₂₃H₂₁ClO₃S ([M–H]−), 411.0822; found, 411.0853.

(OTs)

(E)-1-(4-Fluorophenyl)-2-phenylbut-1-en-1-y1 4-methylbenzenesulfonate (2e): 1-(4-Fluorophenyl)-2-phenylbutan-1-one (1e, 2.0 mmol, 485 mg) was employed. Compound 2e was obtained as a white solid (617 mg, 78%); mp 74–77 °C; HPLC (tM = 3.78 min, tM = 4.81 min) indicated >99:1 isomer ratio; FTIR (neat, cm⁻¹) 3045, 2988, 2924, 1598, 1368; ¹H NMR (400 MHz, CDCl₃) δ 7.53–7.41 (m, 2H), 7.20–7.09 (m, 5H), 7.04–6.94 (m, 2H), 6.89–6.79 (m, 2H), 6.64–6.49 (m, 2H), 2.67 (q, J = 7.5 Hz, 2H), 2.37 (s, 3H), 0.91 (t, J = 7.5 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 161.9 (d, J = 248.6 Hz), 144.6, 141.5, 138.1, 137.1, 134.4, 131.7 (d, J = 8.4 Hz), 130.1 (d, J = 3.6 Hz), 129.31, 129.28, 128.2, 128.0, 127.2, 114.4 (d, J = 21.9 Hz), 26.1, 21.6, 11.9; ¹⁹F NMR (376 MHz, CDCl₃) δ –113.1; HRMS (ESI−) m/z calculated for C₂₃H₂₁FO₃S ([M–H]−), 395.1117; found, 395.1154.

(OTs)

(2f)

(2f)

(E)-2-Phenyl-1-(4-(trifluoromethyl)phenyl)but-1-en-1-y1 4-methylbenzenesulfonate (2f): 2-Phenyl-1-(4-(trifluoromethyl)phenyl)butan-1-one (1f, 2.0 mmol, 585 mg) was employed. Compound 2f was obtained as a white solid (712 mg, 80%); mp 114–115 °C; HPLC (tM = 3.48 min, tM = 4.56 min) indicated 96:4 isomer ratio; FTIR (neat, cm⁻¹) 2974, 2372, 2345, 1374, 1322; ¹H NMR (400 MHz, CDCl₃) δ 7.48 (d, J = 8.4 Hz, 2H), 7.20–7.09 (m, 5H), 7.04–6.94 (m, 2H), 6.89–6.79 (m, 2H), 6.64–6.49 (m, 2H), 2.67 (q, J = 7.5 Hz, 2H), 2.37 (s, 3H), 0.91 (t, J = 7.5 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 161.9 (d, J = 248.6 Hz), 144.6, 141.5, 138.1, 137.1, 134.4, 131.7 (d, J = 8.4 Hz), 130.1 (d, J = 3.6 Hz), 129.31, 129.28, 128.2, 128.0, 127.2, 114.4 (d, J = 21.9 Hz), 26.1, 21.6, 11.9; ¹⁹F NMR (376 MHz, CDCl₃) δ –113.1; HRMS (ESI−) m/z calculated for C₂₃H₂₁FO₃S ([M–H]−), 395.1117; found, 395.1154.
Ethyl (E)-4-(2-phenylbutanoyl)benzoate (2g): Ethyl 4-(2-phenylbutanoyl)benzoate (1g, 2.0 mmol, 593 mg) was employed. Compound 2g was obtained as a white solid (780 mg, 87%); mp 87–88 ºC; HPLC ($t_M = 4.77$ min, $t_m = 6.18$ min) indicated 98:2 isomer ratio; FTIR (neat, cm$^{-1}$) 3054, 2980, 2923, 2813, 1598, 1577, 1487, 1362; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.60–7.51 (m, 2H), 7.53–7.44 (m, 2H), 7.17 (ddt, $J = 5.5$, 4.0, 2.4 Hz, 3H), 7.09 (d, $J = 8.1$ Hz, 2H), 7.05–6.95 (m, 2H), 6.97–6.87 (m, 2H), 4.31 (q, $J = 7.1$ Hz, 2H), 2.68 (q, $J = 7.5$ Hz, 2H), 2.35 (s, 3H), 1.34 (t, $J = 7.0$ Hz, 3H), 0.91 (t, $J = 7.5$ Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 166.2, 144.9, 141.6, 139.0, 138.7, 137.9, 134.2, 129.8, 129.5, 129.4, 129.3, 128.7, 128.5, 128.2, 127.6, 61.1, 26.4, 21.7, 14.5, 12.0; HRMS (ESI−) $m/z$ calculated for C$_{29}$H$_{26}$O$_5$S ([M−H]$^-$), 449.1423; found, 449.1461.

(E)-1-(3-Methoxyphenyl)-2-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2h): 1-(3-Methoxyphenyl)-2-phenyl-but-1-one (2h, 2.0 mmol, 509 mg) was employed. Compound 2h was obtained as a white solid (712 mg, 87%); mp 106–108 ºC; HPLC ($t_M = 4.28$ min, $t_m = 5.91$ min) indicated 98:2 isomer ratio; FTIR (neat, cm$^{-1}$) 3075, 2980, 2932, 2872, 2836, 1598, 1577, 1487, 1362; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.52–7.42 (m, 2H), 7.23–7.12 (m, 3H), 7.12–6.99 (m, 4H), 6.79 (t, $J = 7.0$ Hz, 1H), 6.58–6.44 (m, 2H), 6.34 (dd, $J = 2.6$, 1.6 Hz, 1H), 3.42 (s, 3H), 2.70 (q, $J = 7.5$ Hz, 2H), 2.35 (s, 3H), 0.93 (t, $J = 7.5$ Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 158.5, 144.3, 142.4, 138.4, 137.0, 134.9, 134.4, 129.3, 129.1, 128.3, 128.2, 128.0, 127.1, 122.5, 114.4, 114.3, 54.8, 26.2, 21.5, 11.9; HRMS (ESI−) $m/z$ calculated for C$_{29}$H$_{25}$NO$_3$S ([M−H]$^-$), 407.1317; found, 407.1317.

(E)-2-Phenyl-1-(o-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (2i): 1-(o-Tolyl)-2-phenyl-butan-1-one (IIi, 2.0 mmol, 477 mg) was employed. Compound 2i was obtained as a white solid (480 mg, 61%); mp 96–98 ºC; HPLC ($t_M = 3.62$ min, $t_m = 4.55$ min) indicated 82:18 isomer ratio; FTIR (neat, cm$^{-1}$) 3055, 2976, 2922, 2863, 1596, 1424, 1356; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.34 (d, $J = 8.4$ Hz, 2H), 7.13–7.07 (m, 3H), 7.03–6.99 (m, 2H), 6.99–6.89 (m, 4H), 6.81–6.74 (m, 2H), 2.88 (dq, $J = 14.9$, 7.5 Hz, 1H), 2.69 (dq, $J = 14.8$, 7.5 Hz, 1H), 2.32 (s, 3H), 2.01 (s, 3H), 1.00 (t, $J = 7.5$ Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 144.1, 142.7, 138.2, 137.4, 136.7, 134.6, 133.1, 132.3, 129.7, 129.14, 129.07, 128.5, 128.0, 127.7, 127.1, 125.0, 25.7, 21.7, 19.9, 12.7; HRMS (ESI−) $m/z$ calculated for C$_{29}$H$_{25}$O$_3$S ([M−H]$^-$), 391.1368; found, 391.1408.

(E)-2-Phenyl-1-(pyridin-2-yl)but-1-en-1-yl 4-methylbenzenesulfonate (2j): 2-Phenyl-1-(pyridin-2-yl)butan-1-one (1j, 2.0 mmol, 451 mg) was employed. Compound 2j was obtained as a white solid (620 mg, 82%); mp 89–93 ºC; HPLC ($t_M = 3.93$ min, $t_m = 5.21$ min) indicated 98:2 isomer ratio; FTIR (neat, cm$^{-1}$) 3055, 2980, 2923, 2855, 1713, 1362; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.22 (ddd, $J = 4.8$, 1.7, 0.9 Hz, 1H), 7.60 (d, $J = 8.5$ Hz, 2H), 7.35–7.09 (m,
6H), 7.07–6.97 (m, 2H), 6.90 (ddd, J = 7.5, 4.8, 1.2 Hz, 1H), 6.77 (d, J = 7.9 Hz, 1H), 2.69 (q, J = 7.5 Hz, 2H), 2.38 (s, 3H), 0.92 (t, J = 7.5 Hz, 3H); 13C NMR (101 MHz, CDCl3) δ 152.8, 148.7, 144.4, 141.4, 139.5, 137.9, 135.1, 134.2, 129.3, 129.2, 128.2, 128.1, 127.4, 126.1, 126.1, 26.1, 21.6, 11.7; HRMS (ESI−) m/z calculated for C22H21NO3S ([M–H]−), 378.1164; found, 378.1145.

(E)-1-Phenyl-2-(p-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (2k): 1-Phenyl-2-(p-tolyl) butan-1-one (1k, 2.0 mmol, 477 mg) was employed. Compound 2k was obtained as a white solid (660 mg, 84%); mp 115–117 °C; HPLC (tM = 3.63 min, tm = 4.93 min) indicated 95:5 isomer ratio; FTIR (neat, cm−1) 3046, 2970, 2931, 1595, 1365; 1H NMR (400 MHz, CDCl3) δ 7.49–7.44 (m, 2H), 7.07 (d, J = 8.1 Hz, 2H), 7.02–6.94 (m, 3H), 6.93–6.83 (m, 6H), 2.66 (q, J = 7.5 Hz, 2H), 2.35 (s, 3H), 2.26 (s, 3H), 0.91 (t, J = 7.5 Hz, 3H); 13C NMR (101 MHz, CDCl3) δ 144.2, 142.4, 136.8, 136.7, 135.2, 134.4, 129.9, 129.24, 129.17, 128.8, 128.0, 127.4, 127.3, 26.1, 21.5, 21.1, 12.0; HRMS (ESI−) m/z calculated for C24H24O3S ([M–H]−), 391.1368; found, 391.1388.

(E)-2-(4-Methoxyphenyl)-1-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2l): 2-(4-Methoxyphenyl)-1-phenylbutan-1-one (1l, 2.0 mmol, 509 mg) was employed. Compound 2l was obtained as a white solid (610 mg, 75%); mp 100–101 °C; HPLC (tM = 4.79 min, tm = 6.43 min) indicated 95:5 isomer ratio; FTIR (neat, cm−1) 2995, 2967, 2922, 1606, 1508, 1346; 1H NMR (400 MHz, CDCl3) δ 7.46 (d, J = 8.4 Hz, 2H), 7.07 (d, J = 8.1 Hz, 2H), 7.02–6.85 (m, 7H), 6.77–6.66 (m, 2H), 3.74 (s, 3H), 2.66 (q, J = 7.5 Hz, 2H), 2.35 (s, 3H), 0.91 (t, J = 7.5 Hz, 3H); 13C NMR (101 MHz, CDCl3) δ 158.6, 144.2, 142.3, 136.4, 134.4, 134.1, 130.5, 130.4, 129.9, 129.2, 128.0, 127.4, 127.4, 113.6, 55.1, 26.1, 21.5, 12.0; HRMS (ESI−) m/z calculated for C22H21NO3S ([M–H]−), 391.1368; found, 391.1388.

(E)-2-(4-Chlorophenyl)-1-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2m): 2-(4-Chlorophenyl)-1-phenylbutan-1-one (1m, 2.0 mmol, 517 mg) was employed. Compound 2m was obtained as a white solid (600 mg, 80%); mp 123–125 °C; HPLC (tM = 3.88 min, tm = 4.77 min) indicated 98:2 isomer ratio; FTIR (neat, cm−1) 2975, 2967, 2922, 1606, 1508, 1346; 1H NMR (400 MHz, CDCl3) δ 7.50–7.39 (m, 2H), 7.17–7.10 (m, 2H), 7.07 (d, J = 8.1 Hz, 2H), 7.04–6.82 (m, 7H), 2.68 (q, J = 7.5 Hz, 2H), 2.35 (s, 3H), 0.91 (t, J = 7.5 Hz, 3H); 13C NMR (101 MHz, CDCl3) δ 144.4, 143.0, 136.8, 135.7, 134.3, 133.5, 132.9, 130.8, 129.9, 129.22, 129.20, 128.4, 128.0, 127.8, 127.5, 26.0, 21.6, 11.9; HRMS (ESI−) m/z calculated for C23H21ClO3S ([M–H]−), 407.0822; found, 411.0883.

(E)-2-(4-Fluorophenyl)-1-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2n): 2-(4-Fluorophenyl)-1-phenylbutan-1-one (1n, 2.0 mmol, 485 mg) was employed. Compound 2n was obtained as a white solid (600 mg, 76%); mp 121–122 °C; HPLC (tM = 3.85 min, tm = 4.79 min) indicated 98:2 isomer ratio; FTIR (neat, cm−1) 3041, 2973, 2973, 2939, 2978, 1598, 1507, 1446, 1347; 1H NMR (400 MHz, CDCl3) δ 7.49–7.42 (m, 2H), 7.10–7.04 (m, 2H), 7.03–6.95 (m, 3H), 6.93–6.82 (m, 6H), 2.68 (q, J = 7.5 Hz, 2H), 2.35 (s, 3H), 0.92 (t, J = 7.5 Hz, 3H); 13C NMR (101 MHz, CDCl3) δ 152.8, 148.7, 144.4, 141.4, 139.5, 137.9, 135.1, 134.2, 129.3, 129.2, 128.2, 128.1, 127.4, 126.1, 122.1, 26.1, 21.6, 11.7; HRMS (ESI−) m/z calculated for C22H21NO3S ([M–H]−), 378.1164; found, 378.1145.
MHz, CDCl$_3$) $\delta$ 161.80 (d, $J$ = 246.4 Hz), 144.4, 142.8, 135.9, 134.3, 134.1 (d, $J$ = 3.6 Hz), 133.7, 131.0 (d, $J$ = 8.0 Hz), 129.9, 129.2, 128.0, 127.7, 127.4, 115.2 (d, $J$ = 21.3 Hz), 26.1, 21.6, 11.9; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ –114.9; HRMS (ESI−) $m/z$ calculated for C$_{23}$H$_{21}$FO$_3$S ([M – H$^-$]), 395.1117; found, 395.1202.

$(E)$-1-Phenyl-2-(4-(trifluoromethyl)phenyl)but-1-en-1-yl 4-methylbenzenesulfonate (2o): 1-Phenyl-2-[4-(trifluoromethyl)phenyl]butan-1-one (1o, 2.0 mmol, 585 mg) was employed. Compound 2o was obtained as a white solid (790 mg, 88%); mp 97–98 °C; HPLC ($t_M$ = 3.65 min, $t_m$ = 4.36 min) indicated 98:2 isomer ratio; FTIR (neat, cm$^{-1}$) 3061, 2996, 2969, 2937, 1597, 1324; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.43 (dd, $J$ = 11.5, 8.3 Hz, 4H), 7.19–6.98 (m, 5H), 6.96–6.83 (m, 4H), 2.73 (q, $J$ = 7.5 Hz, 2H), 2.35 (s, 3H), 0.92 (t, $J$ = 7.5 Hz, 3H); $^{13}$C NMR (101 MHz, DMSO-d$_6$) $\delta$ 145.0, 143.0, 142.1, 135.0, 133.1, 132.9, 130.0, 129.7, 129.6, 128.3, 127.65 (q, $J$ = 31.2 Hz), 127.62, 127.55, 125.1 (q, $J$ = 3.8 Hz), 124.0 (q, $J$ = 271.7 Hz), 25.1, 21.0, 11.6; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ –62.6; HRMS (ESI−) $m/z$ calculated for C$_{24}$H$_{21}$F$_3$O$_3$S ([M–H$^-$]), 445.1085; found, 445.1114.

Ethyl $(E)$-4-(1-phenyl-1-(tosyloxy)but-1-en-2-yl)benzoate (2p): Ethyl 4-(1-benzoylpropyl) benzoate (1p, 2.0 mmol, 593 mg) was employed. Compound 2p was obtained as a white solid (860 mg, 95%); mp 63–64 °C; HPLC ($t_M$ = 5.23 min, $t_m$ = 6.17 min) indicated 99:1 isomer ratio; FTIR (neat, cm$^{-1}$) 3065, 2973, 2936, 2875, 1708, 1605, 1372, 1290; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.84 (d, $J$ = 8.6 Hz, 2H), 7.45 (d, $J$ = 8.9 Hz, 2H), 7.14–7.04 (m, 4H), 7.04–6.94 (m, 1H), 6.93–6.83 (m, 4H), 4.33 (q, $J$ = 7.1 Hz, 2H), 2.72 (q, $J$ = 7.5 Hz, 2H), 2.34 (s, 3H), 1.35 (t, $J$ = 7.1 Hz, 3H), 1.09 (t, $J$ = 7.5 Hz, 3H); $^{13}$C NMR (101 MHz, DMSO-d$_6$) $\delta$ 166.3, 144.4, 143.3, 143.2, 136.1, 134.2, 133.4, 129.9, 129.5, 129.4, 129.2, 128.0, 127.9, 127.5, 60.9, 53.4, 25.9, 21.5, 14.3, 11.9; HRMS (ESI−) $m/z$ calculated for C$_{26}$H$_{26}$O$_5$S ([M–H$^-$]), 449.1423; found, 449.1483.

$(E)$-2-(3-Methoxyphenyl)-1-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2q): 2-(3-Methoxyphenyl)-1-phenyl-butan-1-one (1q, 2.0 mmol, 509 mg) was employed. Compound 2q was obtained as a white solid (640 mg, 78%); mp 84–85 °C; HPLC ($t_M$ = 4.41 min, $t_m$ = 5.73 min) indicated 95:5 isomer ratio; FTIR (neat, cm$^{-1}$) 3054, 2977, 2933, 2874, 1712, 1594; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.53–7.42 (m, 2H), 7.07 (dd, $J$ = 8.1, 5.9 Hz, 3H), 7.03–6.96 (m, 1H), 6.90 (d, $J$ = 4.4 Hz, 4H), 6.69 (dd, $J$ = 8.3, 2.7, 0.9 Hz, 1H), 6.60 (dd, $J$ = 7.7, 1.3 Hz, 1H), 6.55 (dd, $J$ = 2.6, 1.5 Hz, 1H), 3.63 (s, 3H), 2.67 (q, $J$ = 7.5 Hz, 2H), 2.35 (s, 3H), 0.93 (t, $J$ = 7.5 Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 159.3, 144.3, 142.6, 139.6, 136.7, 134.3, 133.9, 129.8, 129.2, 129.1, 128.0, 127.6, 127.3, 121.9, 114.9, 112.8, 55.1, 26.1, 21.6, 12.0; HRMS (ESI−) $m/z$ calculated for C$_{23}$H$_{22}$O$_6$S ([M–H$^-$]), 407.1317; found, 407.1200.
(E)-1-Phenyl-2-(o-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (2r): 2-(o-Tolyl)-1-phenyl-butan-1-one (1r, 2.0 mmol, 477 mg) was employed. Compound 2r was obtained as a white solid (390 mg, 50%); mp 85–87 °C; HPLC ($t_M$ = 3.78 min, $t_m$ = 4.73 min) indicated 95:5 isomer ratio; FTIR (neat, cm$^{-1}$) 3023, 2973, 2933, 1597, 1492, 1443, 1360; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.52 (d, $J$ = 8.3 Hz, 2H), 7.18–6.79 (m, 11H), 2.82–2.59 (m, 1H), 2.59–2.44 (m, 1H), 2.35 (s, 3H), 2.05 (s, 3H), 0.91 (t, $J$ = 7.5 Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 144.4, 142.3, 137.4, 136.2, 135.9, 134.2, 134.1, 130.1, 129.7, 129.2, 128.8, 128.1, 127.5, 127.3, 127.2, 125.5, 26.2, 21.5, 19.4, 11.4; HRMS (ESI−) m/z calculated for C$_{24}$H$_{24}$O$_3$S ([M–H]−), 391.1368; found, 391.1441.

(Z)-1-Phenyl-2-(pyridin-2-yl)but-1-en-1-yl 4-methylbenzenesulfonate (2s′): 1-Phenyl-2-(pyridin-2-yl)butan-1-one (1s, 2.0 mmol, 451 mg) was employed. Compound 2s′ was obtained as a white solid (464 mg, 61%); HPLC ($t_M$ = 7.64 min, $t_m$ = 6.59 min) indicated 98:2 isomer ratio (favoring 2s′); mp 135–136 °C; FTIR (neat, cm$^{-1}$) 2961, 2931, 2873, 2373, 2345, 1459, 1432, 1366; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.59 (ddd, $J$ = 4.8, 1.9, 1.0 Hz, 1H), 7.87–7.80 (m, 1H), 7.69 (td, $J$ = 7.4, 1.8 Hz, 1H), 7.64 (dd, $J$ = 7.7, 1.1 Hz, 1H), 7.40–7.36 (m, 2H), 7.34–7.23 (m, 4H), 7.21–7.17 (m, 1H), 7.00 (d, $J$ = 8.0 Hz, 2H), 2.59 (q, $J$ = 7.4 Hz, 2H), 2.34 (s, 3H), 0.86 (t, $J$ = 7.4 Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 144.4, 142.3, 137.4, 136.2, 135.9, 134.2, 134.1, 130.1, 129.7, 129.2, 128.8, 128.1, 127.5, 127.3, 127.2, 125.5, 26.2, 21.5, 19.4, 11.4; HRMS (ESI−) m/z calculated for C$_{22}$H$_{21}$NO$_3$S ([M–H]−), 378.1164; found, 378.1215.

(E)-1,2-Diphenylprop-1-en-1-yl 4-methylbenzenesulfonate (2t): 1,2-Diphenylpropan-1-one (1t, 2.0 mmol, 421 mg) was employed. Compound 2t was obtained as a white solid (341 mg, 47%); mp 91–93 °C; HPLC ($t_M$ = 4.45 min, $t_m$ = 5.77 min) indicated 83:17 isomer ratio; FTIR (neat, cm$^{-1}$) 3060, 3029, 2919, 2870, 1650, 1597, 1372; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.55–7.45 (m, 2H), 7.20–6.85 (m, 11H), 2.35 (s, 3H), 2.20 (s, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 144.4, 143.3, 140.0, 134.0, 134.4, 134.0, 131.0, 129.8, 129.2, 128.8, 128.0, 127.7, 127.4, 127.1, 21.6, 19.9; HRMS (ESI−) m/z calculated for C$_{22}$H$_{20}$O$_3$S ([M–H]−), 363.1055; found, 363.1112.

(E)-1,2-diphenylbut-1-en-1-yl 4-methylbenzenesulfonate (2u): Standard LiHMDS/DMEA conditions lead to decomposition. Alternative, less stereoselective LiO$t$Bu conditions were used to prepare material for subsequent Suzuki coupling studies. To a nitrogen-purged septum-top 20 mL vial equipped with a stir bar was added 1,2-diphenylpent-4-en-1-one (1u, 1.0 mmol, 238 mg) and THF (1 mL). The vial was submerged in a 23 °C water bath, and LiO$t$Bu (1.4 equiv, 1.0 M in THF, 1.4 mL) was added via syringe over 2 min. The resulting yellow solution was then stirred for 30 min. A solution of Ts$_2$O (1.4 equiv, 457 mg) in 2.5 mL THF was added via syringe over 5 min. The reaction was stirred for 3 h and sampled for HPLC analysis which shows 86:14 isomer ratio, favoring the desired E isomer 2u. The reaction was then diluted with 5 mL MTBE and 2 mL 0.5 M aq NaOH. The biphasic solution was separated, and the organic layer was concentrated in vacuo (40 °C). The crude residue was
purified by silica gel column chromatography, eluent conditions 0–15% i-PrOAc in heptane. Compound 2u was obtained as a white solid (104 mg, 27%); mp 137–138 °C; HPLC (t_m = 3.68 min, t_m = 4.73 min) indicated 85:15 isomer ratio; FTIR (neat, cm⁻¹) 3061, 3032, 2969, 2926, 2856, 2373, 2345, 1648, 1373; ¹H NMR (400 MHz, CDCl₃) δ 7.51–7.43 (m, 2H), 7.26 (s, 1H), 7.19–7.12 (m, 2H), 7.07 (d, J = 8.2 Hz, 2H), 7.03–6.79 (m, 7H), 3.51 (hept, J = 6.9 Hz, 1H), 2.34 (s, 3H), 0.99 (d, J = 7.0 Hz, 6H); ¹³C NMR (101 MHz, CDCl₃) δ 144.2, 142.2, 140.5, 135.7, 134.3, 134.0, 130.5, 129.6, 129.2, 128.0, 127.6, 127.4, 127.1, 126.9, 29.2, 21.5, 20.8; HRMS (ESI−) m/z calculated for C₂₄H₂₄O₃S ([M–H]−), 391.1368; found, 391.1412.

(2v): 1,2-Diphenylpenta-1,4-dien-1-yl 4-methylbenzenesulfonate (2v): 1,2-Diphenylpent-4-en-1-one (1v, 2.0 mmol, 473 mg) was employed. Compound 2v was obtained as a white solid (630 mg, 81%); mp 72–74 °C; HPLC (t_M = 3.86 min, t_m = 5.14 min) indicated 95:5 isomer ratio; FTIR (neat, cm⁻¹) 3057, 2982, 2893, 1641, 1597, 1347; ¹H NMR (400 MHz, CDCl₃) δ 7.55–7.43 (m, 2H), 7.20–7.11 (m, 3H), 7.11–7.05 (m, 2H), 7.05–6.97 (m, 3H), 6.89 (d, J = 4.4 Hz, 4H), 5.70 (ddt, J = 16.8, 10.0, 6.7 Hz, 1H), 5.03 (dq, J = 17.1, 1.6 Hz, 1H), 4.97 (dq, J = 10.0, 1.5 Hz, 1H), 3.44 (dt, J = 6.7, 1.5 Hz, 2H), 2.35 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 144.4, 143.7, 138.2, 134.3, 134.1, 133.7, 132.9, 129.9, 129.5, 129.2, 128.1, 128.0, 127.8, 127.4, 127.1, 116.8, 37.5, 21.6; HRMS (ESI−) m/z calculated for C₂₄H₂₂O₃S ([M–H]−), 389.1212; found, 389.1254.

(2w): 3-Cyclopropyl-1,2-diphenylprop-1-en-1-yl 4-methylbenzenesulfonate (2w): 3-Cyclopropyl-1,2-diphenylprop-1-one (1w, 2.0 mmol, 501 mg) was employed. Compound 2w was obtained as a white solid (660 mg, 82%); mp 113–114 °C; HPLC (t_M = 3.73 min, t_m = 5.08 min) indicated 95:5 isomer ratio; FTIR (neat, cm⁻¹) 3066, 2981, 2920, 1597, 1362; ¹H NMR (400 MHz, CDCl₃) δ 7.47 (d, J = 8.5 Hz, 2H), 7.17–7.10 (m, 3H), 7.07 (d, J = 6.7 Hz, 1H), 7.04–6.93 (m, 1H), 6.88 (d, J = 4.4 Hz, 4H), 2.64 (d, J = 7.3 Hz, 2H), 2.34 (s, 3H), 0.71–0.55 (m, 1H), 0.40–0.27 (m, 2H), 0.13–0.03 (m, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 144.3, 142.8, 138.9, 135.5, 134.3, 133.9, 129.9, 129.5, 129.2, 128.0, 127.5, 127.3, 126.9, 37.5, 21.6, 9.3, 4.6; HRMS (ESI−) m/z calculated for C₂₅H₂₄O₃S ([M–H]−), 403.1368; found, 403.1414.

(2x): 1,2-Diphenylhex-1-en-1-yl 4-methylbenzenesulfonate (2x): 1,2-Diphenylhexan-1-one (1x, 2.0 mmol, 505 mg) was employed. Compound 2x was obtained as a white solid (665 mg, 82%); mp 101–102 °C; HPLC (t_M = 3.45 min, t_m = 4.69 min) indicated >99:1 isomer ratio; FTIR (neat, cm⁻¹) 2953, 2921, 2859, 1598, 1444, 1362; ¹H NMR (400 MHz, CDCl₃) δ 7.47 (d, J = 8.5 Hz, 2H), 7.17–7.10 (m, 3H), 7.07 (d, J = 6.7 Hz, 1H), 7.04–6.93 (m, 4H), 6.88 (d, J = 4.4 Hz, 4H), 2.64 (d, J = 7.3 Hz, 2H), 2.34 (s, 3H), 1.37–1.18 (m, 4H), 0.83 (t, J = 7.0 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 144.3, 143.1, 138.6, 135.6, 134.4, 134.0, 129.9, 129.4, 129.2, 128.1, 128.0, 127.5, 127.3, 126.9, 32.6, 29.3, 22.4, 21.5, 13.9; HRMS (ESI−) m/z calculated for C₂₅H₂₆O₃S ([M–H]−), 405.1525; found, 405.1506.
High-throughput Screening

The high-throughput screening of the Suzuki-Miyaura coupling was conducted employing tosylate \( \text{2a} \) (20 mg, 53 \( \mu \)mol), boronic ester \( \text{3a} \) (1.1 equiv), \( \text{Pd(OAc)}_2 \) (5 mol %), ligand (10 mol %), \( \text{K}_3\text{PO}_4 \cdot \text{H}_2\text{O} \) (1.5 equiv), PhMe/H\(_2\)O (3:1, 0.20 mL, 100 mg/mL) at 70 °C for 18 h. The results are shown below.

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\(^a\)None of the \( E\)- or \( Z\)- olefin isomer was produced in the reaction.

Olefin Synthesis

To a 4 mL septum-top vial equipped with a stir bar was added \( E\)-tosylate \( \text{2} \) (1.0 mmol), boronic ester \( \text{3} \) (1.1 equiv), palladium(II) acetate (1 mol %, 2.3 mg), and 2-dicyclohexylphosphino-2',6'-diisoproxy-1,1'-biphenyl (RuPhos, 2 mol %, 9.8 mg). The vial was evacuated and back-filled with nitrogen (3x). Degassed PhMe (1.5 mL) and degassed aqueous \( \text{K}_3\text{PO}_4 \) (1.5 mmol, 364 mg, 0.54 mL\(^1\)) were added via syringes. Then the vial was heated to 70 °C while
stirring vigorously (1500 rpm). The reaction was sampled by HPLC at 1 h and overnight (16–24 h). Analytical HPLC analyses were performed with an Agilent 1260 Infinity Series HPLC instrument; the column used was ACE Excel 3 C18 HL, 3×50mm; particle size 3 um; injection volume 2 µL; temperature 35 °C; flow rate 1 mL/min; mobile phase A = 0.05% trifluoroacetic acid in H₂O, mobile phase B = 0.05% trifluoroacetic acid in acetonitrile, gradient: 0–0.3’ = 5% B, 0.3–3’ = 5–60% B, 3–4’ = 60–90% B, 4–6’ = 90% B, 6–6.1’ = 5% B, 6.1–7.5’ = 5% B. After no further increase in conversion, the biphasic mixture was diluted with 2 mL of H₂O, and then separated into organic and aqueous layers. The aqueous layer was extracted with PhMe (3×1 mL), and the combined organic layers were washed with brine (1 mL), dried over Na₂SO₄ (0.25 g), filtered, and concentrated in vacuo (50°C). The crude residue was purified by silica gel column chromatography using 20–40% i-PrOAc in heptane if product contains nitrogen or sulfur (4i–k, 5h), or 0–10% i-PrOAc in heptane otherwise.

\[(E)-(1-(4-Fluorophenyl)but-1-ene-1,2-diyl)dibenzene \text{ (4a)}: \text{ (E)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (2a, 2 mmol, 757 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3a, 1.1 equiv, 448 mg) were employed. Amounts of other reagents and solvents were scaled accordingly. Compound 4a was obtained as a white solid (590 mg, 98%); mp 71–72 °C; HPLC (tₘ = 8.73 min, tₚ = 8.41 min) indicated 98:2 isomer ratio; FTIR (neat, cm⁻¹) 3053, 2977, 2957, 2926, 2869, 1597, 1502, 1459, 1441; \(^1\)H NMR (400 MHz, CDCl₃) \(\delta \) 7.32–7.24 (m, 2H), 7.24–7.13 (m, 5H), 7.12–7.00 (m, 5H), 6.98–6.88 (m, 2H), 2.54 (q, \(J = 7.5 \) Hz, 2H), 1.00 (t, \(J = 7.5 \) Hz, 3H); \(^1\)C NMR (101 MHz, CDCl₃) \(\delta \) 161.7 (d, \(J = 247.5 \) Hz), 142.9, 142.7, 142.1, 139.4 (d, \(J = 3.0 \) Hz), 137.8, 131.1 (d, \(J = 7.1 \) Hz), 129.7, 127.9, 127.5, 126.3, 125.9, 115.1 (d, \(J = 21.1 \) Hz), 29.0, 13.6; \(^1\)F NMR (376 MHz, CDCl₃) \(\delta \) –115.8; HRMS was not collected because compound 4a does not ionize under both positive and negative modes.\]

\[(E)-(1-(p-Tolyl)but-1-ene-1,2-diyl)dibenzene \text{ (4b): } (E)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (2a, 1 mmol, 379 mg) and 4,4,5,5-tetramethyl-2-(p-tolyl)-1,3,2-dioxaborolane (3b, 1.1 equiv, 240 mg) were employed. Compound 4b was obtained as a white solid (292 mg, 98%); mp 90–91 °C; HPLC (tₘ = 10.78 min, tₚ = 10.36 min) indicated 98:2 isomer ratio; FTIR (neat, cm⁻¹) 2958, 2929, 2871, 1596, 1508, 1488, 1451, 1363; \(^1\)H NMR (400 MHz, CDCl₃) \(\delta \) 7.18–7.04 (m, 8H), 7.01–6.92 (m, 4H), 6.90–6.84 (m, 2H), 2.49 (q, \(J = 7.4 \) Hz, 2H), 2.35 (s, 3H), 0.93 (t, \(J = 7.4 \) Hz, 3H); \(^1\)C NMR (101 MHz, CDCl₃) \(\delta \) 143.3, 142.4, 142.0, 140.7, 138.9, 136.2, 130.8, 129.8, 129.4, 128.9, 127.8, 127.4, 126.1, 125.7, 29.1, 21.3, 13.7; HRMS was not collected because compound 4b does not ionize under both positive and negative modes.\]

\[(E)-(1-(4-Methoxyphenyl)but-1-ene-1,2-diyl)dibenzene \text{ (4c): } (E)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (2a, 1 mmol, 379 mg) and 2-(4-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3c, 1.1 equiv, 263 mg) were employed. Compound 4c was obtained as a white solid (301 mg, 96%); mp 89–90 °C; HPLC (tₘ = 7.73 min, tₚ = 7.96 min) indicated 98:2 isomer ratio; FTIR (neat, cm⁻¹) 3040, 2955, 2870, 2835, 1604, 1571, 1460, 1440; \(^1\)H NMR (400 MHz, CDCl₃) \(\delta \) 7.20–6.70 (m, 7H), 7.01–6.92 (m, 4H), 6.90–6.84 (m, 2H), 2.49 (q, \(J = 7.4 \) Hz, 2H), 2.35 (s, 3H), 0.93 (t, \(J = 7.4 \) Hz, 3H); \(^1\)C NMR (101 MHz, CDCl₃) \(\delta \) 143.3, 142.4, 142.0, 140.7, 138.9, 136.2, 130.8, 129.8, 129.4, 128.9, 127.8, 127.4, 126.1, 125.7, 29.1, 21.3, 13.7; HRMS (ESI+) \(m/z \) calculated for \(C_{23}H_{23}O ([M+H]^+)\), 315.1749; found, 315.1755.\]
**Ethyl (E)-4-(1,2-diphenylbut-1-en-1-yl)benzoate (4d):** (E)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (2a, 1 mmol, 379 mg) and ethyl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (3d, 1.1 equiv, 304 mg) were employed. Compound 4d was obtained as a white solid (340 mg, 95%); mp 91–92 °C; HPLC ($t_M$ = 9.41 min, $t_m$ = 9.66 min) indicated 98:2 isomer ratio; FTIR (neat, cm$^{-1}$) 3078, 2977, 2871, 1711, 1475, 1442, 1365; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.10–7.98 (m, 2H), 7.39–7.27 (m, 2H), 7.22–7.05 (m, 4H), 7.04–6.92 (m, 4H), 6.91–6.82 (m, 2H), 4.37 (q, $J$ = 7.1 Hz, 2H), 2.46 (q, $J$ = 7.3 Hz, 2H), 1.37 (t, $J$ = 7.1 Hz, 3H), 0.94 (t, $J$ = 7.5 Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 166.5, 148.4, 143.2, 142.4, 141.8, 138.1, 130.8, 129.6, 129.6, 128.8, 128.4, 128.0, 127.6, 126.5, 126.1, 60.9, 29.1, 14.4, 13.6; HRMS (ESI−) $m/z$ calculated for C$_{25}$H$_{24}$O$_2$ ([M–H$^-$]), 355.1698; found, 355.1677.

**MeCl(E)-(1-(4-Chlorophenyl)but-1-ene-1,2-diyl)dibenzene (4f):** (E)-1,2-Diphenylbut-1-en-1-yl 4-methyl benzene sulfonate (2a, 1 mmol, 379 mg) and 4-chlorophenylboronic acid pinacol ester (3f, 2.1 equiv, 512 mg) were employed. Palladium(II) acetate and 2-dicyclohexylphosphino-2',6'-diisopropoxy-1,1'-biphenyl (RuPhos) loadings were increased to 2 mol % and 4 mol %, respectively. Other reagents were unchanged from general procedure. Compound 4f was obtained as a white solid (96 mg, 30%); mp 88–90 °C; HPLC ($t_M$ = 10.69 min, $t_m$ = 10.59 min) indicated >99:1 isomer ratio; FTIR (neat, cm$^{-1}$) 2973, 2872, 1488, 1441; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.37–7.27 (m, 1H), 7.26–7.05 (m, 8H), 7.04–6.92 (m, 4H), 6.86 (dd, $J$ = 7.6, 2.0 Hz, 2H), 2.45 (q, $J$ = 7.4 Hz, 2H), 0.94 (t, $J$ = 7.5 Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 142.9, 142.6, 142.3, 141.7, 137.7, 130.8, 129.9, 129.6, 128.9 (q, $J$ = 32.3 Hz), 128.0, 127.7, 126.6, 126.2, 125.3 (q, $J$ = 4.0 Hz), 124.4 (q, $J$ = 27.2 Hz), 29.1, 13.6; HRMS was not collected because compound 4f does not ionize under both positive and negative modes.

**MeMe(E)-(1-(o-Tolyl)but-1-ene-1,2-diyl)dibenzene (4g):** (E)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (2a, 1 mmol, 379 mg) and 4,4,5,5-tetramethyl-2-(o-tolyl)-1,3,2-dioxaborolane (3g, 1.1 equiv, 458 mg) were employed.
Palladium(II) acetate and 2-dicyclohexylphosphino-2',6'-diisopropoxy-1,1'-biphenyl (RuPhos) loadings were doubled to 2 mol % and 4 mol %, respectively. Other reagents remained unchanged from general procedure. Compound 4g was obtained as an opaque oil (258 mg, 86%); HPLC ($t_M = 10.24$ min, $t_m = 9.29$ min) indicated 98:2 isomer ratio; FTIR (neat, cm$^{-1}$) 3055, 3017, 2930, 2871, 1597, 1490, 1441, 1078, 1030; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.34–7.28 (m, 1H), 7.26–7.07 (m, 7H), 6.99–6.85 (m, 6H), 2.38–2.23 (m, 2H), 2.16 (s, 3H), 0.84 (t, $J = 7.5$ Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 142.6, 142.3, 142.0, 141.4, 137.8, 136.2, 130.5, 130.4, 129.9, 129.8, 128.0, 127.4, 127.1, 126.4, 125.8, 125.7, 29.3, 20.0, 13.0; HRMS was not collected because compound 4g does not ionize under both positive and negative modes.

(\(E\)-(1-Mesitylbut-1-ene-1,2-diyl)dibenzene (4h)): \(E\)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (2a, 1 mmol, 379 mg) and 4,4,5,5-tetramethyl-2-(2,4,6-trimethylphenyl)-1,3,2-dioxaborolane (3k, 1.5 equiv, 369 mg) were employed. Reaction showed <5% conversion under standard conditions.

(\(E\)-2-(1,2-Diphenylbut-1-en-1-yl)pyridine (4i)): \(E\)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (2a, 1 mmol, 379 mg) and pyridine-2-boronic acid pinacol ester (3l, 1.1 equiv, 233 mg) were employed. Reaction showed <5% conversion under standard conditions.
(E)-3-(1,2-Diphenylbut-1-en-1-yl)thiophene (4j): (E)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (2a, 1 mmol, 379 mg) and 4,4,5,5-tetramethyl-2-(3-thienyl)-1,3,2-dioxaborolane (3j, 1.5 equiv, 315 mg) were employed. Palladium(II) acetate and 2-dicyclohexylphosphino-2',6'-diisopropoxy-1,1'-biphenyl (RuPhos) loadings were doubled to 2 mol % and 4 mol %, respectively. Other reagents remained unchanged from general procedure. Compound 4j was obtained as a white solid (194 mg, 48%); mp 94–98 °C; HPLC (t<sub>M</sub> = 7.83 min, t<sub>m</sub> = 8.75 min) indicated 97:3 isomer ratio; FTIR (neat, cm<sup>−1</sup>) 3094, 3028, 2923, 2865, 1486, 1371; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.37–7.29 (m, 1H), 7.24 (dd, <i>J</i> = 5.0, 3.0 Hz, 1H), 7.16–7.04 (m, 6H), 7.03–6.94 (m, 2H), 6.93–6.85 (m, 4H), 2.60 (q, <i>J</i> = 7.5 Hz, 2H), 0.98 (t, <i>J</i> = 7.4 Hz, 3H); <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 143.6, 143.0, 142.8, 142.2, 133.5, 130.6, 129.7, 129.4, 127.8, 127.4, 126.2, 125.9, 124.7, 122.9, 29.3, 13.7; HRMS (ESI+) <i>m/z</i> calculated for C<sub>20</sub>H<sub>18</sub>S ([M+H]<sup>+</sup>), 291.1207; found, 291.1176.

(E)-5-(1,2-Diphenylbut-1-en-1-yl)-1-(tetrahydro-2H-pyran-2-yl)-2,3-dihydro-1H-indazole (4k): (E)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (2a, 1 mmol, 379 mg) and 1-tetrahydropyran-2-yl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)indazole (3k, 1.1 equiv, 361 mg) were employed. Compound 4k was obtained as a white solid (409 mg, 84%); mp 94–97 °C; HPLC (t<sub>M</sub> = 8.06 min, t<sub>m</sub> = 8.29 min) indicated 98:2 isomer ratio; FTIR (neat, cm<sup>−1</sup>) 2961, 2848, 1504, 1440; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.00 (s, 1H), 7.65–7.60 (m, 1H), 7.54 (d, <i>J</i> = 8.7 Hz, 1H), 7.26 (dd, <i>J</i> = 8.6, 1.5 Hz, 1H), 7.19–7.05 (m, 3H), 7.01–6.85 (m, 6H), 5.70 (dd, <i>J</i> = 9.6, 2.6 Hz, 1H), 4.04 (d, <i>J</i> = 11.9 Hz, 1H), 3.80–3.67 (m, 1H), 2.65–2.54 (m, 1H), 2.49 (q, <i>J</i> = 7.5 Hz, 2H), 2.11 (ddt, <i>J</i> = 25.6, 13.0, 2.6 Hz, 2H), 1.82–1.58 (m, 3H), 0.95 (t, <i>J</i> = 7.4 Hz, 3H); <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 143.3, 142.5, 142.3, 138.8, 138.6, 136.9, 134.2, 130.9, 129.8, 129.0, 127.9, 127.4, 126.2, 125.8, 124.9, 121.3, 109.8, 85.5, 67.6, 29.5, 29.1, 25.2, 22.8, 13.7; HRMS (ESI+) <i>m/z</i> calculated for C<sub>28</sub>H<sub>28</sub>N<sub>2</sub>O ([M+H]<sup>+</sup>), 409.2280; found, 409.2226.

((1E,3Z)-Hexa-1,3-diene-1,3,4-triyl)tribenzene (4l): (E)-1,2-Diphenylbut-1-en-1-yl 4-methyl benzene sulfonate (2a, 1 mmol, 379 mg) and 4,4,5,5-tetramethyl-2-[(E)-styryl]-1,3,2-dioxaborolane (3l, 1.50 equiv, 345 mg) were employed. Palladium(II) acetate and 2-dicyclohexylphosphino-2',6'-diisopropoxy-1,1'-biphenyl (RuPhos) loadings were doubled to 2 mol % and 4 mol %, respectively. Other reagents remained unchanged from general procedure. Compound 4l was obtained as a yellow oil (248 mg, 80%); HPLC (t<sub>M</sub> = 10.72 min, t<sub>m</sub> = 12.90 min) indicated 98:2 isomer ratio; FTIR (neat, cm<sup>−1</sup>) 3055, 3025, 2966, 2931, 2872, 1598, 1489, 1442; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.51 (d, <i>J</i> = 15.8 Hz, 1H), 7.40–7.34 (m, 2H), 7.29 (dd, <i>J</i> = 8.4, 6.8 Hz, 2H), 7.22–6.93 (m, 11H), 6.18 (d, <i>J</i> = 15.9 Hz, 1H), 2.82 (q, <i>J</i> = 7.5 Hz, 2H), 1.13 (t, <i>J</i> = 7.5 Hz, 3H); <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 144.2, 143.1, 140.3, 138.0, 136.6, 132.3, 131.2, 129.4, 128.6, 128.2, 127.5, 127.4, 127.3, 126.4, 126.1, 125.9, 27.7, 13.7; HRMS (ESI+) <i>m/z</i> calculated for C<sub>24</sub>H<sub>22</sub>N<sub>2</sub>O ([M+H]<sup>+</sup>), 311.1800; found, 311.1840.
Me

(E)-Hex-3-en-1-yn-1,3,4-triyltribenzene (4m): (E)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (2a, 1 mmol, 379 mg) and 4,4,5,5-tetramethyl-2-(2-phenylethynyl)-1,3,2-dioxaborolane (3m, 2.1 equiv, 479 mg) were employed. Compound 4m was obtained as a yellow oil (188 mg, 61%); HPLC (t_M = 10.72 min, t_m = 10.46 min) indicated 99:1 isomer ratio; FTIR (neat, cm⁻¹) 3056, 3028, 2967, 2930, 2871, 2197, 1597, 1441.96; ¹H NMR (400 MHz, CDCl₃) δ 7.52–7.45 (m, 2H), 7.41–7.26 (m, 3H), 7.23–7.00 (m, 10H), 2.99 (q, J = 7.5 Hz, 2H), 1.10 (t, J = 7.5 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 151.9, 140.7, 139.0, 131.5, 129.9, 129.2, 128.3, 128.01, 127.95, 127.7, 126.9, 126.6, 123.9, 120.1, 93.7, 90.3, 31.4, 12.7; HRMS (ESI+) m/z calculated for C₂₄H₂₀([M+H]+), 309.1643; found, 309.1581.

(Z)-Oct-3-ene-3,4-diyl dibenzene (4o): (E)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (2a, 1 mmol, 379 mg) and n-butylboronic acid (3o, 1.1 equiv, 116 mg) were employed. Compound 4o was obtained as a clear oil (259 mg, 98%); HPLC (t_M = 12.22 min, t_m = 13.53 min) indicated 99:1 isomer ratio; FTIR (neat, cm⁻¹) 2958, 2870, 1440, 1066; ¹H NMR (400 MHz, CDCl₃) δ 7.02 (tq, J = 8.1, 1.9 Hz, 4H), 6.98–6.88 (m, 6H), 2.63–2.47 (m, 4H), 1.32 (dt, J = 7.3, 3.6 Hz, 4H), 0.95 (t, J = 7.5 Hz, 3H), 0.91–0.81 (m, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 143.6, 143.3, 139.8, 137.9, 129.9, 129.83 (2C), 127.45 (2C), 125.5, 34.1, 30.9, 27.6, 22.9, 14.2, 13.3; HRMS was not collected because compound 4o does not ionize under both positive and negative modes.

(Z)-(1-Cyclopropylbut-1-ene-1,2-diyl)dibenzene (4n): To a 8 mL septum-top vial equipped with a stir bar was added (E)-1,2-diphenylbut-1-en-1-yl 4-methylbenzenesulfonate compound (2a, 1.0 mmol, 379 mg), palladium(II) acetate (1 mol %, 2.3 mg), and 2-dicyclohexylphosphino-2',6'-diisopropoxy-1,1'-biphenyl (RuPhos, 2 mol %, 9.8 mg). The vial was evacuated and back-filled with nitrogen (3×). Cyclopropylzinc bromide (3n, 1.1 equiv, 0.36 M in THF, 3.1 mL) was added to the reaction via syringe, followed by LiCl (1.1 equiv, 0.5 M in THF, 2.2 mL). Then the vial was heated to 60 °C while stirring vigorously (1500 rpm). It was sampled by HPLC at 1 h and overnight (16–24 h). After no further increase in conversion, the biphasic mixture was quenched with 4 mL of H₂O, and then separated into organic and aqueous layers. The aqueous layer was extracted with i-PrOAc (3×1 mL), and the combined organic layers were washed with brine (1 mL), dried over Na₂SO₄ (0.25 g), filtered, and concentrated in vacuo (50 °C). The crude residue was purified by silica gel column chromatography using 0–10% i-PrOAc in heptane. Compound 4n was obtained as a clear oil (236 mg, 95%); HPLC (t_M = 8.25 min, t_m = 10.09 min) indicated 98:2 isomer ratio; FTIR (neat, cm⁻¹) 3078, 3054, 3018, 2964, 2930, 2871, 1597, 1489, 1440; ¹H NMR (400 MHz, CDCl₃) δ 7.07–6.97 (m, 4H), 6.97–6.87 (m, 4H), 6.86–6.79 (m, 2H), 2.73 (d, J = 7.5 Hz, 2H), 2.00–1.88 (m, 1H), 1.04 (d, J = 7.8 Hz, 3H), 0.76–0.63 (m, 2H), 0.33–0.19 (m, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 143.7, 141.3, 140.0, 137.9, 130.8, 129.5, 127.3, 127.0, 125.6, 125.4, 27.5, 13.8, 13.0, 5.8; HRMS was not collected because compound 4n does not ionize under both positive and negative modes.

(Z)-(1-(4-Fluorophenyl)but-1-ene-1,2-diyl)dibenzene (5a): (E)-1-(4-Fluorophenyl)-2-phenylbut-1-en-1-yl 4-methyl benzenesulfonate (2e, 1 mmol, 204 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were
employed. Compound 5a was obtained as a white solid (144 mg, 93%); mp 74–75 °C; HPLC (t_M = 8.41 min, t_m = 8.73 min) indicated 98:2 isomer ratio; FTIR (neat, cm⁻¹) 3057, 2971, 2928, 2870, 1894, 1599, 1503, 1441; ¹H NMR (400 MHz, CDCl₃) δ 7.36–7.29 (m, 2H), 7.28–7.20 (m, 3H), 7.19–7.05 (m, 5H), 6.88–6.79 (m, 2H), 6.72–6.62 (m, 2H), 2.46 (d, J = 7.5 Hz, 2H), 0.93 (t, J = 7.5 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 161.0 (d, J = 246.4 Hz), 143.4, 142.6, 142.1, 139.0 (d, J = 4.0 Hz), 137.9, 132.3 (d, J = 7.1 Hz), 129.5, 128.3, 128.0, 126.8, 126.3, 114.3 (d, J = 21.2 Hz), 29.0, 13.5; ¹⁹F NMR (376 MHz, CDCl₃) δ –116.6; HRMS was not collected because compound 5a does not ionize under both positive and negative modes.

(Z)-(1-(p-Tolyl)but-1-ene-1,2-diyl)dibenzene (5b): (E)-2-Phenyl-1-(p-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (2b, 1 mmol, 330 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were employed. Compound 5b was obtained as a white solid (280 mg, 94%); mp 77–78 °C; FTIR (neat, cm⁻¹) 3078, 3051, 2977, 2957, 2917, 2870, 1509, 1490, 1441; HPLC (t_M = 10.36 min, t_m = 10.78 min) indicated 96:4 isomer ratio; ¹H NMR (400 MHz, CDCl₃) δ 7.38–7.29 (m, 2H), 7.28–7.21 (m, 2H), 7.20–7.07 (m, 5H), 6.84–6.71 (m, 5H), 2.47 (q, J = 7.4 Hz, 2H), 2.17 (s, 3H), 0.92 (d, J = 7.4 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 143.8, 142.4, 141.7, 140.0, 138.7, 135.2, 130.6, 129.7, 129.5, 128.1, 127.8, 126.5, 126.1, 29.1, 21.1, 13.6; HRMS was not collected because compound 5b does not ionize under both positive and negative modes.

(Z)-(1-(4-Methoxyphenyl)but-1-ene-1,2-diyl)dibenzene (5c): (E)-1-(4-Methoxy phenyl)-2-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2c, 1 mmol, 340 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were employed. Compound 5c was obtained as a beige solid (240 mg, 92%); mp 114–116 °C; HPLC (t_M = 7.96 min, t_m = 7.73 min) indicated 93:7 isomer ratio; FTIR (neat, cm⁻¹) 2929, 2832, 1604, 1505, 1464, 1439; ¹H NMR (400 MHz, CDCl₃) δ 7.37–7.30 (m, 2H), 7.29–7.21 (m, 3H), 7.20–7.07 (m, 5H), 6.82–6.73 (m, 2H), 6.58–6.51 (m, 2H), 3.66 (s, 3H), 2.45 (d, J = 7.4 Hz, 2H), 0.91 (d, J = 7.4 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 157.5, 143.9, 142.5, 141.3, 138.3, 135.5, 131.9, 129.7, 129.5, 128.1, 127.9, 127.8, 126.5, 126.1, 55.0, 29.1, 13.6; HRMS (ESI+) m/z calculated for C₂₃H₂₃O ([M+H]+), 315.1749; found, 315.1703.

Ethyl (Z)-4-(1,2-diphenylbut-1-en-1-yl)benzoate (5d): Ethyl (E)-4-(2-phenyl-1-(tosyloxy)but-1-en-1-yl)benzoate (2g, 1 mmol, 451 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were employed. Compound 5d was obtained as a yellow solid (300 mg, 84%); mp 88–92 °C; HPLC (t_M = 9.66 min, t_m = 9.41 min) indicated 96:4 isomer ratio; FTIR (neat, cm⁻¹) 2929, 2932, 2832, 1604, 1505, 1464, 1439; ¹H NMR (400 MHz, CDCl₃) δ 7.73–7.70 (m, 2H), 7.39–7.31 (m, 2H), 7.31–7.19 (m, 3H), 7.19–7.06 (m, 5H), 6.98–6.91 (m, 2H), 4.27 (q, J = 7.1 Hz, 2H), 2.49 (q, J = 7.4 Hz, 2H), 1.31 (d, J = 7.2 Hz, 3H), 0.93 (t, J = 7.5 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 166.6, 148.0, 143.9, 142.8, 141.7, 138.1, 130.7, 129.6, 129.5, 128.5, 128.3, 128.0, 126.9, 126.6, 60.7, 29.1, 24.9, 14.3, 13.5; HRMS (ESI–) m/z calculated for C₂₄H₂₂O₃S ([M–H]−), 355.1698; found, 355.1728.
(Z)-(1-(4-(Trifluoromethyl)phenyl)but-1-ene-1,2-diyldibenzene (5e): (E)-2-Phenyl-1-(4-(trifluoromethyl)phenyl) but-1-en-1-yl 4-methylbenzenesulfonate (2f, 1 mmol, 447 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were employed. Compound 5e was obtained as a white solid (339 mg, 96%); mp 59–62 °C; HPLC (t_M = 10.59 min, t_m = 10.69 min) indicated >99:1 isomer ratio; FTIR (neat, cm⁻¹) 2975, 2930, 2872, 1614, 1489, 1442, 1406, 1323; ¹H NMR (400 MHz, CDCl₃) δ 7.46–7.37 (m, 2H), 7.37–7.27 (m, 4H), 7.27–7.11 (m, 6H), 7.05 (d, J = 8.2 Hz, 2H), 2.55 (q, J = 7.4 Hz, 2H), 1.00 (t, J = 7.5 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 146.8, 144.1, 142.8, 141.5, 137.6, 131.0, 129.6, 128.4, 128.1, 127.7 (q, J = 32.3 Hz), 127.0, 126.7, 124.4 (q, J = 3.0 Hz), 124.3 (q, J = 273.7 Hz), 29.2, 13.5; ¹⁹F NMR (376 MHz, CDCl₃) δ –62.4; HRMS was not collected because compound 5e does not ionize under both positive and negative modes.

(Z)-(1-(4-Chlorophenyl)but-1-ene-1,2-diyldibenzene (5f): (E)-1-(4-Chlorophenyl)-2-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2d, 1 mmol, 413 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were employed. Compound 5f was obtained as a clear oil (265 mg, 83%); HPLC (t_M = 11.30 min, t_m = 11.39 min) indicated 98:2 isomer ratio; FTIR (neat, cm⁻¹) 3077, 3054, 3020, 2967, 2871, 1597, 1487; ¹H NMR (400 MHz, CDCl₃) δ 7.34 (dt, J = 6.8, 1.1 Hz, 2H), 7.30–7.20 (m, 3H), 7.20–7.06 (m, 5H), 6.99–6.90 (m, 2H), 6.84–6.73 (m, 2H), 2.46 (d, J = 7.4 Hz, 2H), 0.93 (d, J = 7.5 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 143.1, 143.0, 141.9, 141.5, 137.6, 131.2, 131.6, 129.6, 129.5, 128.3, 128.1, 127.6, 126.9, 126.4, 29.1, 13.5; HRMS was not collected because compound 5f does not ionize under both positive and negative modes.

(Z)-(1-(o-Tolyl)but-1-ene-1,2-diyldibenzene (5g): (E)-2-Phenyl-1-(o-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (2i, 1 mmol, 352 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were employed. Compound 5g was obtained as a white solid (263 mg, 98%); mp 69–71 °C; HPLC (t_M = 9.29 min, t_m = 10.24 min) indicated 95:5 isomer ratio; FTIR (neat, cm⁻¹) 3059, 3018, 2956, 2869, 1597, 1490, 1440, 1377; ¹H NMR (400 MHz, CDCl₃) δ 7.32–7.22 (m, 3H), 7.22–7.14 (m, 1H), 7.11–6.98 (m, 5H), 6.98–6.85 (m, 5H), 2.73–2.45 (m, 2H), 2.05 (s, 3H), 1.00 (t, J = 7.4 Hz, 2H), 0.96 (t, J = 7.5 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 142.8, 142.5, 142.3, 138.4, 136.1, 131.5, 129.9, 129.2, 128.9, 128.0, 127.6, 126.5, 126.3, 125.1, 28.0, 20.4, 14.0; HRMS was not collected because compound 5g does not ionize under both positive and negative modes.

(Z)-(2-(1,2-Diphenylbut-1-en-1-yl)pyridine (5h): (E)-2-Phenyl-1-(pyridin-2-yl)but-1-en-1-yl 4-methylbenzenesulfonate (2j, 1 mmol, 380 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were employed. Palladium(II) acetate and 2-dicyclohexylphosphino-2',6'-diisopropoxy-1,1'-biphenyl (RuPhos) loadings were doubled to 2 mol % and 4 mol %, respectively. Other reagents remained unchanged from general procedure. Compound 5h was obtained as a yellow solid (185 mg, 65%); mp 81–82 °C; ¹H NMR indicated 96:4 isomer ratio; FTIR (neat, cm⁻¹) 3053, 2926, 2868, 1584, 1561, 1465, 1440, 1425; ¹H NMR (400 MHz, CDCl₃) δ 8.39 (ddd, J = 4.8, 2.0, 1.0 Hz, 1H), 7.40–7.23 (m, 3H), 7.18–7.05 (m, 5H), 6.91–6.80 (m, 2H), 2.54 (q, J = 7.4 Hz, 2H), 0.96 (t, J
\[ \text{S20} \]

\[ \text{MeF} \]

\[ \text{MeO} \]

\[ \text{F} \]

\[ \text{C} \]

\[ \\]

\[ \text{[Image]} \]

\[ \text{1H NMR (400 MHz, CDCl}_3\text{)} \delta 7.27–7.14 (m, 2H), 7.07–6.91 (m, 9H), 6.87 (d, \text{J} = 6.1 \text{ Hz}, 2H), 2.45 (q, \text{J} = 7.4 \text{ Hz}, 2H), 2.24 (s, 3H), 0.92 (t, \text{J} = 7.4 \text{ Hz}, 3H); \text{13C NMR (101 MHz, CDCl}_3\text{)} \delta 161.7 (d, \text{J} = 246.4 \text{ Hz}), 142.9, 139.7 (d, \text{J} = 3.0 \text{ Hz}), 138.9, 137.5, 135.8, 131.1 (d, \text{J} = 8.1 \text{ Hz}), 130.8, 129.5, 128.6, 127.5, 125.8, 115.1 (d, \text{J} = 20.2 \text{ Hz}), 29.0, 24.9, 21.2, 13.6; \text{19F NMR (376 MHz, CDCl}_3\text{)} \delta -115.9; \text{HRMS was not collected because compound 5i does not ionize under both positive and negative modes.} \]

\[ \text{MeF} \]

\[ \text{MeO} \]

\[ \text{F} \]

\[ \text{C} \]

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\[ \text{[Image]} \]

\[ \text{1H NMR (400 MHz, CDCl}_3\text{)} \delta 7.23–7.15 (m, 2H), 7.08–6.92 (m, 7H), 6.90–6.83 (m, 2H), 6.73–6.65 (m, 2H), 3.72 (s, 3H), 2.44 (q, \text{J} = 7.3 \text{ Hz}, 2H), 1.33 (s, 1H), 0.93 (t, \text{J} = 7.4 \text{ Hz}, 3H); \text{13C NMR (101 MHz, CDCl}_3\text{)} \delta 161.7 (d, \text{J} = 246.4 \text{ Hz}), 158.0, 143.2, 142.1, 139.7, 137.3, 134.1, 131.1 (d, \text{J} = 8.1 \text{ Hz}), 130.8, 130.7, 127.5, 125.8, 115.1 (d, \text{J} = 21.2 \text{ Hz}), 113.3, 55.1, 28.9, 24.9, 13.6; \text{19F NMR (376 MHz, CDCl}_3\text{)} \delta -116.0; \text{HRMS (ESI+)} m/z calculated for C\text{23H}_{21}\text{FO ([M + H]^+), 333.1655; found, 333.1682.} \]

\[ \text{MeF} \]

\[ \text{MeO} \]

\[ \text{F} \]

\[ \text{C} \]

\[ \\]

\[ \text{[Image]} \]

\[ \text{1H NMR (400 MHz, CDCl}_3\text{)} \delta 7.24–7.15 (m, 2H), 7.10–6.94 (m, 7H), 6.91–6.78 (m, 2H), 6.73–6.65 (m, 2H), 3.72 (s, 3H), 2.45 (q, \text{J} = 7.4 \text{ Hz}, 2H), 0.93 (t, \text{J} = 7.4 \text{ Hz}, 3H); \text{13C NMR (101 MHz, CDCl}_3\text{)} \delta 161.7 (d, \text{J} = 246.4 \text{ Hz}), 158.0, 143.2, 142.1, 139.7, 137.3, 134.1, 131.1 (d, \text{J} = 8.1 \text{ Hz}), 130.8, 130.7, 127.5, 125.8, 115.1 (d, \text{J} = 21.2 \text{ Hz}), 113.3, 55.1, 28.9, 24.9, 13.6; \text{19F NMR (376 MHz, CDCl}_3\text{)} \delta -116.0; \text{HRMS (ESI−)} m/z calculated for C\text{21H}_{19}\text{N ([2M+MeCN]^−), 681.3019; found, 681.2966.} \]
(E)-1-Fluoro-4-(1-phenyl-2-(4-(trifluoromethyl)phenyl)but-1-en-1-yl)benzene (5l): (E)-1-Phenyl-2-(4-(trifluoromethyl)phenyl)but-1-en-1-yl 4-methylbenzenesulfonate (2o, 1 mmol, 447 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 122 mg) were employed. Amounts of other reagents and solvents were scaled accordingly. Compound 5l was obtained as a white solid (346 mg, 93%); mp 126–127 °C; 1H NMR indicated >99:1 isomer ratio (HPLC does not resolve the isomers); FTIR (neat, cm−1) 2971, 1739, 1615, 1602, 1325; 1H NMR (400 MHz, CDCl3) δ 7.40 (d, J = 8.2 Hz, 2H), 7.27–7.16 (m, 4H), 6.89–6.81 (m, 5H), 7.09–6.95 (m, 5H), 6.89–6.81 (m, 2H), 2.12 (s, 3H); 13C NMR (101 MHz, CDCl3) δ 161.9 (d, J = 7.5 Hz, 3H); 19F NMR (376 MHz, CDCl3) δ −62.4, −115.3; HRMS (ESI−) m/z calculated for C21H19F1 (M−H)−, 369.1267; found, 369.1282.

Ethyl (E)-4-(1-(4-fluorophenyl)-1-phenylbut-1-en-2-yl)benzoate (5m): Ethyl (E)-4-(1-phenyl-1-(tosyloxy)but-1-en-2-yl)benzoate (2p, 0.5 mmol, 225 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 122 mg) were employed. Amounts of other reagents and solvents were scaled accordingly. Compound 5m was obtained as a white solid (156 mg, 83%); mp 121–122 °C; HPLC (tM = 9.34 min, tR = 9.21 min) indicated >99:1 isomer ratio; FTIR (neat, cm−1) 2971, 2930, 2873, 1715, 1603, 1506, 1366; 1H NMR (400 MHz, CDCl3) δ 7.84 (d, J = 8.4 Hz, 2H), 7.26–7.13 (m, 4H), 7.09–7.06 (m, 5H), 7.84 (d, J = 8.4 Hz, 2H), 7.12–6.93 (m, 5H), 6.89–6.81 (m, 2H), 4.33 (q, J = 7.2 Hz, 2H), 2.50 (q, J = 7.5 Hz, 2H), 1.35 (t, J = 7.2 Hz, 3H), 0.92 (t, J = 7.4 Hz, 3H); 13C NMR (101 MHz, CDCl3) δ 166.5, 161.8 (d, J = 247.5 Hz), 147.2, 142.4, 141.7, 139.0 (d, J = 4.0 Hz), 131.0 (d, J = 8.1 Hz), 130.7, 129.7, 129.2, 128.3, 127.6, 126.3, 115.2 (d, J = 21.1 Hz), 60.8, 28.8, 24.9, 14.3, 13.5; 19F NMR (376 MHz, CDCl3) δ −115.4; HRMS (ESI−) m/z calculated for C21H19F1 (M−H)−, 373.1604; found, 373.1557.

(E)-1-(1-(4-Fluorophenyl)-1-phenylbut-1-en-2-yl)-2-methylbenzene (5n): (E)-1-Phenyl-2-(o-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (2r, 0.67 mmol, 262 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 163 mg) were employed. Amounts of other reagents and solvents were scaled accordingly. Compound 5n was obtained as a white solid (206 mg, 98%); mp 87–88 °C; HPLC (tM = 9.28 min, tR = 9.53 min) indicated 97:3 isomer ratio; FTIR (neat, cm−1) 3054, 3017, 2969, 2930, 2872, 1739, 1601, 1505, 1442, 1375; 1H NMR (400 MHz, CDCl3) δ 7.28–7.20 (m, 2H), 7.16 (dd, J = 7.3, 1.8 Hz, 1H), 7.12–6.92 (m, 8H), 6.87–6.79 (m, 2H), 4.33 (q, J = 7.5 Hz, 2H), 2.09 (s, 3H), 0.92 (t, J = 7.5 Hz, 3H); 13C NMR (101 MHz, CDCl3) δ 161.7 (d, J = 246.4 Hz), 142.5, 141.3, 141.1, 139.1 (d, J = 4.0 Hz), 138.1, 135.6, 131.1 (d, J = 8.1 Hz), 130.0, 129.8, 129.7, 127.3, 126.5, 126.0, 125.2, 115.1 (d, J = 21.2 Hz), 29.8, 19.8, 12.8; 19F NMR (376 MHz, CDCl3) δ −115.9; HRMS was not collected because compound 5n does not ionize under both positive and negative modes.

(E)-(1-(4-Fluorophenyl)prop-1-ene-1,2-diyldibenzene (5o): (E)-1,2-Diphenyl prop-1-en-1-yl 4-methylbenzene sulfonate (2t, 0.64 mmol, 232 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 156 mg) were employed. Amounts of other reagents and solvents were scaled accordingly. Compound 5o was obtained as a white solid (165 mg, 90%); mp 106–107 °C; HPLC (tM = 7.19 min, tR = 7.90 min) indicated 97:3 isomer ratio; FTIR (neat, cm−1) 3054, 2913, 2855, 1740, 1599, 1506, 1442, 1375; 1H NMR (400 MHz, CDCl3) δ 7.27–7.17 (m, 2H), 7.17–7.05 (m, 4H), 7.05–6.94 (m, 5H), 6.90–6.83 (m, 2H), 2.12 (s, 3H); 13C NMR (101 MHz, CDCl3) δ 161.6 (d, J = 246.4 Hz), 143.9, 142.9, 139.4 (d, J = 4.0 Hz), 138.4, 136.1, 131.6 (d, J = 8.1 Hz), 130.8, 129.3, 127.7, 126.3,
126.0, 115.1 (d, J = 22.2 Hz), 23.4; $^{19}$F NMR (376 MHz, CDCl$_3$) δ −115.7; HRMS was not collected because compound 5o does not ionize under both positive and negative modes.

(E)-(1-(4-Fluorophenyl)penta-1,4-diene-1,2-diyl)dibenzene (5p): (E)-1,2-Diphenylpenta-1,4-dien-1-yl 4-methyl benzenesulfonate (2v, 1 mmol, 391 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 224 mg) were employed. Compound 5p was obtained as a yellow solid (305 mg, 97%); mp 55–59 °C; HPLC (t$_M$ = 8.55 min, t$_m$ = 8.31 min) indicated 97:3 isomer ratio; FTIR (neat, cm$^{-1}$) 3056, 3020, 2971, 2927, 1739, 1600, 1507, 1443; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.29–7.18 (m, 2H), 7.16–7.05 (m, 4H), 7.04–6.95 (m, 6H), 6.92–6.84 (m, 2H), 5.80–5.65 (m, 1H), 5.04–4.89 (m, 2H), 3.25 (dt, J = 6.3, 1.6 Hz, 2H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 161.9 (d, J = 247.5 Hz), 142.8, 142.1, 139.7, 139.1 (d, J = 4.0 Hz), 138.2, 136.2, 131.3 (d, J = 8.1 Hz), 130.8, 129.8, 127.9, 127.6, 126.4, 126.2, 116.0, 115.1 (d, J = 21.2 Hz), 40.5, 24.9; $^{19}$F NMR (376 MHz, CDCl$_3$) δ −115.4; HRMS (ESI−) m/z calculated for C$_{21}$H$_{19}$N ([M+MeCN]$^-$) 355.1736; found, 355.1232.

(E)-(3-Cyclopropyl-1-(4-fluorophenyl)prop-1-ene-1,2-diyl)dibenzene (5q): (E)-3-Cyclopropyl-1,2-diphenylprop-1-en-1-yl 4-methylbenzenesulfonate (2w, 1 mmol, 405 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 224 mg) were employed. Compound 5q was obtained as an off-white solid (316 mg, 96%); mp 92–93 °C; HPLC (t$_M$ = 9.70 min, t$_m$ = 10.07 min) indicated 96:4 isomer ratio; FTIR (neat, cm$^{-1}$) 3067, 2950, 2850, 1739, 1597, 1505, 1489, 1442; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.31–7.19 (m, 5H), 7.19–7.06 (m, 5H), 0.46–0.36 (m, 2H), 0.01 (dt, J = 5.9, 4.4 Hz, 2H), 7.41–7.33 (m, 2H), 7.05–6.99 (m, 2H), 2.49 (d, J = 6.6 Hz, 2H), 0.87–0.73 (m, 1H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 161.7 (d, J = 246.4 Hz), 142.80, 142.76, 141.2, 139.2 (d, J = 4.0 Hz), 138.2, 131.2 (d, J = 7.1 Hz), 130.6, 129.8, 127.8, 127.5, 126.2, 125.9, 115.1 (d, J = 21.2 Hz), 40.1, 10.4, 4.9; $^{19}$F NMR (376 MHz, CDCl$_3$) δ −115.8; HRMS was not collected because compound 4n does not ionize under both positive and negative modes.

(E)-(1-(4-Fluorophenyl)hex-1-ene-1,2-diyl)dibenzene (5r): (E)-1,2-Diphenylhex-1-en-1-yl 4-methylbenzene sulfonate (2x, 1 mmol, 407 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 244 mg) were employed. Compound 5r was obtained as white solid (327 mg, 99%); mp 101–102 °C; HPLC (t$_M$ = 12.30 min, t$_m$ = 12.74 min) indicated 98:2 isomer ratio; FTIR (neat, cm$^{-1}$) 3054, 3020, 2956, 2858, 1739, 1598, 1506, 1442, 1376; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.26–7.18 (m, 2H), 7.17–7.06 (m, 5H), 7.06–6.93 (m, 5H), 6.89–6.82 (m, 2H), 2.46–2.38 (m, 2H), 1.37–1.14 (m, 4H), 0.78 (t, J = 7.2 Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 161.7 (d, J = 246.4 Hz), 142.9, 142.4, 141.6, 139.4 (d, J = 4.0 Hz), 138.0, 131.1 (d, J = 8.1 Hz), 130.7, 129.5, 127.8, 127.4, 126.2, 125.8, 115.0 (d, J = 22.2 Hz), 35.7, 31.1, 22.8, 13.9; $^{19}$F NMR (376 MHz, CDCl$_3$) δ −116.0; HRMS (ESI+) m/z calculated for C$_{24}$H$_{23}$F ([M+H]$^+$), 331.1862; found, 331.1891.
Tosylate isomer marker synthesis (for HPLC)

The mixture of E- and Z-tosylates 2 and 2* was prepared by a less selective reaction conditions employing LiO-t-Bu and subjected to HPLC analysis along with isolated E-tosylates 2 from the selective tosylation reaction, to establish HPLC retention times and isomer ratios. A general preparation of a mixture of E- and Z-tosylates is shown below.

To a septum-top 10 mL vial equipped with a stir bar was added ketone 1 (1 mmol). The vial was vacuumed and back-filled with nitrogen (3×), then anhydrous THF (1 mL) was added via syringe, followed by dropwise addition of LiO-t-Bu (1.4 equiv, 1 M in THF, 1.4 mL). After stirring for 30 min, a solution of Ts₂O (1.4 equiv, 1.4 mmol, 457 mg dissolved in 3 mL THF) was added dropwise via syringe. The reaction was continued to stir vigorously (1500 rpm) for another 1h, then sampled by HPLC.

Analytical HPLC analyses were performed with an Agilent 1200 Series HPLC instrument; the column used was ChiralPak AD-H 4.6×150mm, particle size 5 um; injection volume 2 µL; temperature 20 °C; flow rate 1 mL/min; mobile phase A = hexanes; mobile phase B = isopropyl alcohol; gradient: 0–5' = 2–10% B; 5–10' = 10–20% B; 10–12' = 20–2% B; 12–13' = 2% B. Below is a sample chromatogram for tosylate 2k and 2k’.
Olefin isomer marker synthesis (for HPLC)

Except for 4a–g and 5a–g which were synthesized as a pair of isomers by judicious choice of coupling partners, all other olefin isomers were synthesized by employing the same general Suzuki procedure described in previous sections using the corresponding tosylate isomers. These olefin isomers 4' and 5' were subjected to HPLC analysis along with isolated olefins products 4 or 5 to establish HPLC retention times and isomer ratios.

Analytical HPLC analyses were performed with an Agilent 1260 Infinity Series HPLC instrument; the column used was ACE Excel 3 C18 HL, 3x50mm; particle size 3 um; injection volume 2 µL; temperature 35 °C; flow rate 1 mL/min; mobile phase A = 0.05% trifluoroacetic acid in H2O, mobile phase B = 0.05% trifluoroacetic acid in acetonitrile, gradient: 0–0.3' = 5% B, 0.3–3' = 5–60% B, 3–4' = 60–90% B, 4–6' = 90% B, 6–6.1' = 5% B, 6.1–7.5' = 5% B. Below is a sample chromatogram for olefins 4d and 5d with which we identify retention times.
Ketone Synthesis

Ketones were synthesized according to a literature procedure. To a stirring solution of NaHMDS (2.5 equiv, 1.0 M in THF) at 23 °C was slowly added arylacetic acid (1 equiv, 1.5 M in THF) over 1 h. The resulting mixture was stirred for 1 h, and benzoate (0.77–0.95 equiv, 1.0 M in THF) was added dropwise. After stirring at 23 °C for another 2 h, EtOH (5.0 equiv) was added, followed by alkyl halide (1.9–2.5 equiv), and the reaction was stirred overnight (16–20 h). The reaction was quenched with H₂O (20 mL/g) and filtered through Celite. The filtrate was extracted with DCM (3×15 mL/g). The combined organic layers were washed with H₂O (3×15 mL/g), dried over Na₂SO₄ (as needed), filtered, and concentrated in vacuo. The crude residue was purified by silica gel column chromatography, then by preparative HPLC if necessary.

2-Phenyl-1-(p-tolyl)butan-1-one (1b): Ethyl 4-methylbenzoate (0.95 equiv, 69.8 mmol, 11.4 g), 2-phenylacetic acid (1.0 equiv, 73.5 mmol, 10.0 g), and ethyl iodide (2.0 equiv, 147 mmol, 22.9 g) were employed. After extraction, the crude residue was purified via preparative HPLC. Ketone 1b was obtained as a light yellow oil (6.00 g, 34%). ¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, J = 8.0 Hz, 2H), 7.35 (m, 4H), 7.23 (m, 3H), 4.50 (t, J = 7.4 Hz, 1H), 2.37 (s, 1H), 2.28 (m, 1H), 1.93 (m, 1H), 0.97 (t, J = 7.4 Hz, 3H); ¹³C NMR (400 MHz, CDCl₃) δ 199.8, 143.6, 139.9, 134.6, 129.2, 128.9, 128.3, 126.9, 55.4, 27.2, 21.6, 12.4.

1-(4-Methoxyphenyl)-2-phenylbutan-1-one (1c): Methyl-4-methoxybenzoate (0.80 equiv, 61.0 mmol, 10.1 g), 2-phenylacetic acid (1.0 equiv, 76.0 mmol, 10.3 g), and ethyl iodide (1.9 equiv, 144 mmol, 22.9 g) were employed. After extraction, the crude residue was purified via silica gel column chromatography using eluent conditions 100% petroleum ether. The residue was then purified by preparatory HPLC. Ketone 1c was obtained as a white solid (11.6 g, 77%). ¹H NMR (400 MHz, CDCl₃) δ 8.00–7.91 (m, 2H), 7.34–7.22 (m, 4H), 7.20–7.13 (m, 1H), 6.87–6.78 (m, 2H), 4.40 (t, J = 7.3 Hz, 1H), 3.76 (s, 3H), 2.19 (dd, J = 13.8, 7.3 Hz, 1H), 1.83 (dd, J = 13.6, 7.4 Hz, 1H), 0.89 (t, J = 7.3 Hz, 3H); ¹³C NMR (400 MHz, CDCl₃) δ 198.6, 163.3, 140.1, 130.9, 130.1, 128.8, 126.9, 113.7, 55.4, 55.1, 27.2, 12.4.

1-(4-Chlorophenyl)-2-phenylbutan-1-one (1d): Methyl 4-chlorobenzoate (0.77 equiv, 87.9 mmol, 15.0 g), 2-phenylacetic acid (1.0 equiv, 114 mmol, 15.6 g), and ethyl iodide (1.9 equiv, 214 mmol, 33.3 g) were employed. After extraction, the crude residue was purified via preparatory HPLC. Ketone 1d was obtained as a light yellow oil (13.6 g, 60%). ¹H NMR (400 MHz, CDCl₃) δ 7.74 (d, 2H, J = 8.8 Hz), 7.14 (m, 6H), 7.04 (m, 1H), 4.23 (t, 1H, J = 7.0 Hz), 2.05 (m, 1H), 1.71 (m, 1H), 0.75 (t, 3H, J = 7.4 Hz); ¹³C NMR (400 MHz, CDCl₃) δ 198.9, 139.4, 139.2, 135.3, 130.1, 129.0, 128.8, 128.2, 127.2, 55.6, 27.1, 12.3.
1-(4-Fluorophenyl)-2-phenylbutan-1-one \((1e)\): Ethyl 4-fluorobenzoate (0.77 equiv, 179 mmol, 30.0 g), 2-phenylacetic acid (1.0 equiv, 233 mmol, 32.0 g), and ethyl iodide (2.5 equiv, 448 mmol, 69.0 g) were employed. After extraction, the crude residue was purified via silica gel column chromatography using eluent conditions 100% petroleum ether. The residue was then purified by preparatory HPLC. Ketone \(1e\) was obtained as a light yellow oil (13.6 g, 60%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.00 (m, 2H), 7.29 (d, 4H), 7.22 (m, 1H), 7.05 (m, 2H), 4.40 (t, 3H, \(J = 7.2\) Hz), 2.20 (m, 1H), 1.85 (m, 1H), 0.91 (t, 3H, \(J = 7.4\) Hz); \(^{13}\)C NMR (400 MHz, CDCl\(_3\)) \(\delta\) 198.4, 166.7, 164.2, 139.5, 133.4, 131.3, 128.9, 128.2, 127.1, 115.6, 55.5, 27.1, 12.2.

2-Phenyl-1-(4-(trifluoromethyl)phenyl)butan-1-one \((1f)\): Methyl-4-(trifluoromethyl)benzoate (0.95 equiv, 69.8 mmol, 14.2 g), 2-phenylacetic acid (1.0 equiv, 73.5 mmol, 10.0 g), and ethyl iodide (2.0 equiv, 147 mmol, 22.9 g) were employed. After extraction, the crude residue was purified by preparatory HPLC. Ketone \(1f\) was obtained as a light yellow oil (7.92 g, 37%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.09 (d, \(J = 8.0\) Hz, 2H), 7.66 (d, \(J = 8.4\) Hz, 2H), 7.34 (m, 4H), 7.26 (m, 1H), 4.47 (t, 1H, \(J = 7.2\) Hz), 2.26 (m, 1H), 1.93 (m, 1H), 0.96 (t, 3H, \(J = 7.2\) Hz); \(^{13}\)C NMR (400 MHz, CDCl\(_3\)) \(\delta\) 199.1, 139.8, 139.0, 134.1, 133.8, 133.5, 129.1, 129.0, 128.3, 127.3, 125.6, 125.6, 122.3, 119.6, 56.0, 27.0, 12.1.

Ethyl 4-(2-phenylbutanoly)benzoate \((1g)\): Diethyl terephthalate (0.95 equiv, 69.8 mmol, 19.6 g), 2-phenylacetic acid (1.0 equiv, 73.5 mmol, 10.0 g), and ethyl iodide (2.4 equiv, 176 mmol, 27.5 g) were employed. After extraction, the crude residue was purified by preparatory HPLC. Ketone \(1g\) was obtained as a light yellow solid (7.20 g, 33%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.06 (d, 2H, \(J = 8.3\) Hz), 8.01 (d, 2H, \(J = 8.5\) Hz), 7.32 (m, 4H), 7.22 (m, 1H), 4.41 (m, 3H), 2.23 (m, 1H), 1.40 (t, 3H, \(J = 7.2\) Hz), 0.94 (t, 3H, \(J = 7.3\) Hz); \(^{13}\)C NMR (400 MHz, CDCl\(_3\)) \(\delta\) 199.6, 165.7, 140.2, 139.1, 133.8, 129.6, 128.9, 128.5, 128.3, 127.1, 77.0, 76.7, 61.3, 56.0, 26.9, 14.2, 12.2.

1-(3-Methoxyphenyl)-2-phenylbutan-1-one \((1h)\): Methyl-3-methoxybenzoate (0.80 equiv, 59.0 mmol, 9.8 g), 2-phenylacetic acid (1.0 equiv, 73.5 mmol, 10.0 g), and ethyl iodide (1.9 equiv, 140 mmol, 21.8 g) were employed. After extraction, the crude residue was purified via silica gel column chromatography using eluent conditions 100% petroleum ether. The residue was then purified by preparatory HPLC. Ketone \(1h\) was obtained as a light yellow oil (5.30 g, 35%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.59 (d, 2H, \(J = 7.5\) Hz), 7.53 (s, 1H), 7.32 (m, 5H), 7.23 (m, 1H), 7.05 (dd, 1H, \(J = 8.2, 2.1\) Hz), 4.46 (t, 1H, 7.3 Hz), 3.82 (s, 3H), 2.23 (m, 1H), 1.90 (m, 1H), 0.94 (t, 3H, \(J = 7.4\) Hz); \(^{13}\)C NMR (400 MHz, CDCl\(_3\)) \(\delta\) 199.9, 159.8, 139.8, 138.5, 129.5, 128.9, 128.3, 127.0, 121.3, 119.2, 113.1, 55.6, 55.3, 27.2, 12.4.
2-Phenyl-1-(α-tolyl)butan-1-one (1i): Methyl 2-methylbenzoate (0.77 equiv, 240 mmol, 36.0 g), 2-phenylacetic acid (1.0 equiv, 312 mmol, 42.5 g), and ethyl iodide (2.1 equiv, 580 mmol, 89.9 g) were employed. After extraction, the crude product was dissolved in THF (100 mL) andaq LiOH (13 g monohydrate in 80 mL), and refluxed for 16 h at 75 °C to ensure complete hydrolysis of the ester. The mixture was extracted once more according to general procedure. The crude residue was purified by recrystallization in n-hexane. Ketone 1i was obtained as a white solid (31.5 g, 55%).

\[ \text{H NMR (400 MHz, CDCl}_3\text{):} \delta 7.52 (dd, J = 7.7, 1.4 Hz, 1H), 7.28–7.09 (m, 8H), 4.26 (t, J = 7.3 Hz, 1H), 2.30 (s, 3H), 2.28–2.16 (m, 1H), 1.85 (dt, J = 13.8, 7.3 Hz, 1H), 0.92 (t, J = 7.4 Hz, 3H); \text{C NMR (400 MHz, CDCl}_3\text{):} \delta 204.5, 139.2, 138.9, 137.8, 131.6, 130.7, 128.7, 128.4, 127.8, 127.0, 125.4, 58.8, 26.5, 20.7, 12.4.\]

2-Phenyl-1-(pyridine-2-yl)butan-1-one (1j): Methyl picolinate (0.80 equiv, 137 mmol, 11.6 g), 2-phenylacetic acid (1.0 equiv, 110 mmol, 15.0 g), and ethyl iodide (2.4 equiv, 201 mmol, 31.5 g) were employed. After extraction, the crude residue was purified by preparatory HPLC. Ketone 1j was obtained as a light yellow oil (6.20 g, 32%).

\[ \text{H NMR (400 MHz, CDCl}_3\text{):} \delta 8.70 (d, J = 4.3 Hz, 1H), 8.02 (d, J = 8.1 Hz, 1H), 7.77 (td, J = 7.7, 1.4Hz), 7.41 (m, 3H), 7.28 (t, 2H, J = 7.5 Hz), 7.19 (m, 1H), 5.29 (t, J = 7.5Hz), 2.22 (m, 1H), 1.95 (m, 1H), 0.94 (t, J = 7.4Hz); \text{C NMR (400 MHz, CDCl}_3\text{):} \delta 201.3, 153.0, 148.7, 139.2, 137.1, 129.0, 128.5, 127.0, 126.8, 122.8, 52.6, 26.2, 12.3.\]

1-Phenyl-2-(p-tolyl) butan-1-one (1k): Methyl benzoate (0.83 equiv, 100 mmol, 13.6 g), 2-p-tolylacetic acid (1.0 equiv, 120 mmol, 18.0 g), and ethyl iodide (2.1 equiv, 250 mmol, 39.0 g) were employed. After extraction, the crude residue was purified via silica gel column chromatography using eluent conditions 100% petroleum ether. Ketone 1k was obtained as a white solid (10.7 g, 45%).

\[ \text{H NMR (400 MHz, CDCl}_3\text{):} \delta 7.99–7.92 (m, 2H), 7.45–7.35 (m, 1H), 7.37–7.28 (m, 2H), 7.27–7.17 (m, 2H), 6.86–6.75 (m, 2H), 4.39 (t, J = 7.3 Hz, 1H), 3.66 (s, 3H), 2.17 (dt, J = 13.7, 7.2 Hz, 1H), 1.91–1.75 (m, 1H), 0.89 (t, J = 7.4 Hz, 3H); \text{C NMR (400 MHz, CDCl}_3\text{):} \delta 200.3, 158.6, 137.1, 132.7, 129.6, 128.7, 128.5, 128.2, 55.1, 27.2, 21.0, 12.4.\]

2-(4-Methoxyphenyl)-1-phenylbutan-1-one (1l): Methyl benzoate (0.83 equiv, 150 mmol, 20.4 g), 2-(4-methoxyphenyl)acetic acid (1.0 equiv, 180 mmol, 29.9 g), and ethyl iodide (2.1 equiv, 375 mmol, 58.5 g) were employed. After reaction completion, the reaction was quenched with H2O (200 mL), then concentrated in vacuo. The aqueous residue was extracted with MTBE, and the organic layers were combined and concentrated in vacuo. THF (100 mL) and 10% aq LiOH (100 mL) were added to ensure complete hydrolysis of the ester. The mixture was concentrated and extracted with DCM (200 mL). After extraction, the crude residue was purified via silica gel column chromatography using eluent conditions 100% petroleum ether. Ketone 1l was obtained as a colorless oil (18.0 g, 47%).

\[ \text{H NMR (400 MHz, CDCl}_3\text{):} \delta 8.00–7.92 (m, 2H), 7.22–7.15 (m, 2H), 7.07 (d, J = 8.1 Hz, 2H), 4.40 (t, J = 7.3 Hz, 1H), 2.24 (s, 3H), 2.19 (dt, J = 13.7, 7.3 Hz, 1H), 1.84 (dp, J = 13.6, 7.4 Hz, 1H), 0.89 (t, J = 7.4 Hz, 3H); \text{C NMR (400 MHz, CDCl}_3\text{):} \delta 200.3, 158.6, 137.1, 132.7, 129.6, 128.7, 128.5, 128.2, 55.1, 27.2, 21.0, 12.4.\]
2-(4-Chlorophenyl)-1-phenylbutan-1-one (1m): Ethyl benzoate (0.77 equiv, 45 mmol, 6.80 g), 2-(4-chlorophenyl)acetic acid (1.0 equiv, 59 mmol, 10.0 g), and ethyl iodide (2.1 equiv, 109 mmol, 16.9 g) were employed. The crude residue was purified via silica gel column chromatography, eluent conditions 0 – 5% iPrOAc in heptanes. Ketone 1m was obtained as a yellow oil (6.6 g, 63%).

1H NMR (400 MHz, CDCl3) δ 7.99–7.91 (m, 2H), 7.48–7.39 (m, 1H), 7.39–7.30 (m, 2H), 7.23 (s, 4H), 4.44 (t, J = 7.3 Hz, 1H), 2.17 (dq, J = 14.5, 7.2 Hz, 1H), 1.91–1.75 (m, 1H), 0.88 (t, J = 7.4 Hz, 3H); 13C NMR (400 MHz, CDCl3) 199.7, 138.2, 136.8, 133.0, 132.9, 129.7, 129.0, 128.6 (2C), 54.6, 27.2, 12.3.

2-(4-Fluorophenyl)-1-phenylbutan-1-one (1n): Methyl benzoate (0.77 equiv, 184 mmol, 25.0 g), p-fluorophenylacetic acid (1.0 equiv, 239 mmol, 36.6 g), and ethyl iodide (2.1 equiv, 460 mmol, 71.7 g) were employed. After extraction, the crude product was dissolved in THF (100 mL) and aq LiOH (13 g monohydrate in 80 mL H2O), and refluxed for 16 h at 75 °C to ensure complete hydrolysis of the ester. The mixture was extracted once more according to general procedure. The crude residue was purified by recrystallization in n-hexane. Ketone 1n was obtained as a white solid (13.5 g, 30%).

1H NMR (400 MHz, CDCl3) δ 7.95 (dd, J = 8.4, 1.3 Hz, 2H), 7.51–7.23 (m, 5H), 6.96 (t, J = 8.7 Hz, 2H), 4.44 (t, J = 7.3 Hz, 1H), 2.18 (dt, J = 13.8, 7.2 Hz, 1H), 1.91–1.75 (m, 1H), 0.89 (t, J = 7.4 Hz, 3H); 13C NMR (400 MHz, CDCl3) 200.1, 136.9, 135.3, 132.9, 129.8, 128.6, 128.6, 115.8, 115.6, 54.5, 27.2, 12.2.

1-Phenyl-2(4-(trifluoromethyl)phenyl)butan-1-one (1 o): Methyl benzoate (1.0 equiv, 196 mmol, 26.7 g), 2-(4-(trifluoromethyl)phenyl)acetic acid (1.0 equiv, 196 mmol, 40.0 g), and ethyl iodide (2.5 equiv, 490 mmol, 76.0 g) were employed. After extraction, the crude residue was purified via silica gel column chromatography using eluent conditions 100% petroleum ether. Ketone 1 o was obtained as a yellow oil (6.5 g, 11%).

1H NMR (400 MHz, CDCl3) δ 7.99 (d, J = 7.5, 2H), 7.57 (d, J = 8.3, 2 H), 7.5 (m, 3H), 7.42 (m, 2H), 4.58 (t, J = 7.4 Hz, 1H), 2.26 (m, 1H), 0.93 (m, 3 H); 13C NMR (400 MHz, CDCl3) δ 199.4, 143.7, 136.7, 133.2, 128.7, 125.8, 125.8, 125.7, 125.7, 55.0, 27.2, 12.2.

2-(3-Methoxyphenyl)-1-phenylbutan-1-one (1q): Methyl benzoate (0.83 equiv, 147 mmol, 20.0 g), 2-(3-methoxyphenyl)acetic acid (1.0 equiv, 180 mmol, 30.0 g), and ethyl iodide (2.1 equiv, 375 mmol, 58.5 g) were employed. After reaction completion, the reaction was quenched with H2O (100 mL) and concentrated in vacuo. MeOH (100 mL) and 10% aq LiOH (100 mL) were added to ensure complete hydrolysis of the ester. The mixture was concentrated and extracted with i-PrOAc (200 mL). After extraction, the crude residue was purified via silica gel column chromatography using eluent conditions 0–5% EtOAc in petroleum ether. Ketone 1q was obtained as a colorless oil (16.0 g, 43%).

1H NMR (400 MHz, CDCl3) δ 8.00–7.92 (m, 2H), 7.47–7.38 (m, 1H), 7.38–7.29 (m, 2H), 7.17 (d, J = 8.0 Hz, 1H), 6.93–6.82 (m, 2H), 6.71 (ddd, J = 8.3, 2.5, 1.0 Hz, 1H), 4.41 (t, J = 7.2 Hz, 1H), 3.71 (s, 3H), 2.19 (dt, J = 13.7, 7.2 Hz, 1H), 1.92–1.80 (m, 1H), 0.90 (t, J = 7.4 Hz, 3H); 13C NMR (400 MHz, CDCl3) δ 199.91, 160.0, 141.2, 137.1, 132.8, 129.8, 128.6, 128.5, 120.8, 114.0, 112.2, 55.5, 55.1, 27.1, 12.3.
1-Phenyl-2-(o-tolyl)butan-1-one (1r): Methyl benzoate (0.77 equiv, 147 mmol, 20.0 g), 2-o-tolylacetic acid (1.0 equiv, 191 mmol, 28.7 g), and ethyl iodide (2.1 equiv, 353 mmol, 55.1 g) were employed. After extraction, the crude product was dissolved in THF (100 mL) and aq LiOH (13 g monohydrate in 80 mL H₂O), and refluxed for 16 h at 75 °C to ensure complete hydrolysis of the ester. The mixture was extracted once more according to general procedure. The crude residue was purified via silica gel column chromatography using eluent conditions 100% petroleum ether. Ketone 1r was obtained as a yellow oil (8.3 g, 24%).

1H NMR (400 MHz, CDCl₃) δ 7.87–7.80 (m, 2H), 7.43–7.33 (m, 1H), 7.33–7.24 (m, 2H), 7.18–7.00 (m, 4H), 4.60 (dd, J = 7.9, 6.1 Hz, 1H), 2.48 (s, 3H), 2.22 (dt, J = 13.8, 7.5 Hz, 1H), 1.82–1.67 (m, 1H), 0.94 (t, J = 7.4 Hz, 3H); 13C NMR (400 MHz, CDCl₃) 200.5, 138.5, 137.5, 135.2, 132.7, 131.0, 128.5, 128.4, 127.3, 126.9, 51.5, 26.9, 20.0, 12.7.

1,2-Diphenylpropan-1-one (1t): Methyl benzoate (0.77 equiv, 147 mmol, 20.0 g), 2-phenylacetic acid (1.0 equiv, 191 mmol, 26.0 g), and methyl iodide (2.4 equiv, 353 mmol, 50.0 g) were employed. After extraction, the crude product was dissolved in THF (80 mL) and aq LiOH (13 g monohydrate in 80 mL H₂O), and refluxed for 3 h at 75 °C to ensure complete hydrolysis of the ester. The mixture was extracted once more according to general procedure. The crude residue was purified preparatory HPLC. Ketone 1t was obtained as a yellow oil (10.0 g, 32%).

1H NMR (400 MHz, CDCl₃) δ 7.95 (m, 2H), 7.48 (m, 1H), 7.38 (m, 2H), 7.29 (m, 4H), 7.2 (m, 1H), 4.69 (m, 1H, J = 6.9 Hz), 1.54 (d, 3H, J = 6.8Hz); 13C NMR (400 MHz, CDCl₃) 200.3, 141.5, 136.5, 132.8, 129.0, 128.8, 128.5, 127.8, 126.9, 47.9.

3-Cyclopropyl-1,2-diphenylpropan-1-one (1v): Methyl benzoate (0.77 equiv, 147 mmol, 20.0 g), 2-phenylacetic acid (1.0 equiv, 191 mmol, 26.0 g), and allyl bromide (2.4 equiv, 353 mmol, 42.7 g) were employed. After extraction, the crude product was dissolved in THF (80 mL) and aq LiOH (13 g monohydrate in 80 mL H₂O), and refluxed for 5 h at 75 °C to ensure complete hydrolysis of the ester. The mixture was extracted once more according to general procedure. The crude residue was purified via silica gel column chromatography, then recrystallized in n-hexane. Ketone 1v was obtained as a white solid (9.8 g, 27%).

1H NMR (400 MHz, CDCl₃) δ 7.98 (m, 2H), 7.51–7.44 (m, 1H), 7.43–7.23 (m, 4H), 5.78 (m, 1H), 5.0 (m, 2 H), 4.6 (t, 1H, J = 14.8 Hz), 2.9 (m, 1H), 2.5 (m, 1H); 13C NMR (400 MHz, CDCl₃) 199.2, 139.1, 136.8, 136.0, 132.9, 128.9, 128.7, 128.5, 128.3, 127.1, 116.7, 53.7, 38.2.

1,2-Diphenylpent-4-en-1-one (1w): Methyl benzoate (0.77 equiv, 147 mmol, 20.0 g), 2-phenylacetic acid (1.0 equiv, 191 mmol, 26.0 g), and bromomethyl cyclopropane (2.4 equiv, 353 mmol, 47.7 g) were employed. After extraction, the crude product was dissolved in THF (80 mL) and aq LiOH (13 g monohydrate in 80 mL H₂O), and refluxed for 16 h at 75 °C to ensure complete hydrolysis of the ester. The mixture was extracted once more according to general procedure. The crude residue was purified via silica gel column chromatography using eluent conditions 2% EtOAc in petroleum ether; the product was then recrystallized in n-hexane. Ketone 1w was obtained as a white solid (3.0 g, 8%).

1H NMR (400 MHz, CDCl₃) δ 8.02–7.93 (m, 2H), 7.51–7.44 (m, 1H), 7.43–7.23 (m,
1,2-Diphenylhexan-1-one (1x): Methyl benzoate (1.0 equiv, 125 mmol, 17.0 g), 2-phenylacetic acid (1.0 equiv, 125 mmol, 17.0 g), and n-butyl iodide (2.5 equiv, 250 mmol, 46.0 g) were employed. After extraction, the crude product was dissolved in THF (80 mL) and aq LiOH (13 g monohydrate in 80 mL H$_2$O), and refluxed for 16 h at 45 °C to ensure complete hydrolysis of the ester. The mixture was extracted once more according to general procedure. The crude residue was purified via silica gel column chromatography using eluent conditions 0–10 % EtOAc in petroleum ether. Ketone 1x was obtained as a white solid (9.8 g, 31%).

Ethyl 4-(1-oxo-1-phenylbutan-2-yl) benzoate (1p): Compound 1p was synthesized according to a modified literature procedure.$^3$ To a 250 mL jacketed reactor was added ethyl 4-bromo benzoate (1.0 equiv, 100 mmol, 22.9 g), acetophenone (1.0 equiv, 100 mmol, 12.0 g), Pd(dba)$_2$ (7.5 mol %, 7.5 mmol, 4.30 g), dppf (9 mol %, 9 mmol, 4.90 g), t-BuONa (1.5 equiv, 110 mmol, 10.5 g), and 120 mL of anhydrous THF. The reactor was vacuumed and back-filled with nitrogen (3x), then the reaction mixture stirred at 70–75°C for 5 h. Upon reaction completion as assessed by HPLC, the reaction was cooled, concentrated, and purified by silica gel column chromatography using EtOAc in petroleum ether (1:5) to give ethyl 4-(2-oxo-2-phenylethyl) benzoate as an off-white solid (10.0 g, 37%). To a solution of ethyl 4-(2-oxo-2-phenylethyl) benzoate (1.0 equiv, 10.0 g, 37.3 mmol) in 100mL of THF was added EtONa (3.0 equiv, 112 mmol, 7.60 g) under nitrogen. The mixture was stirred at 20–30 °C for 30 min, followed by addition of EtI (2.5 equiv, 92.5 mmol, 14.4 g). The reaction mixture was stirred at 35–40 °C for 16 h, then cooled down to 20 °C. The reaction was diluted with H$_2$O (100 mL). The biphasic mixture was concentrated to remove THF, then extracted with ethyl acetate (200 mL). The organic layer was concentrated, and then purified by silica gel column chromatography to give an off-white solid. Further purification by recrystallization in 20 mL heptane afforded ketone 1p as a white solid (7.0 g, 64%). $^1$H NMR (400 MHz, CDCl$_3$) δ 8.01–7.90 (m, 4H), 7.54–7.43 (m, 1H), 7.43–7.34 (m, 4H), 4.51 (t, $J = 7.3$ Hz, 1H), 4.34 (q, $J = 7.1$ Hz, 2H), 2.22 (dp, $J = 14.5$, 7.3 Hz, 1H), 1.87 (dt, $J = 13.7$, 7.4 Hz, 1H), 1.35 (t, $J = 7.1$ Hz, 3H), 0.91 (t, $J = 7.4$ Hz, 3H); $^{13}$C NMR (400 MHz, CDCl$_3$) δ 199.4, 166.3, 144.7, 136.8, 133.0, 130.1, 129.3, 128.6 (2C), 128.3, 60.9, 55.4, 27.1, 14.3, 12.2.
1-Phenyl-2-(pyridine-2-yl) butan-1-one (1s): To a 250 mL round bottom flask was added LDA (1.2 equiv, 113 mmol, 12.1 g) dissolved in anhydrous THF at 0–5 °C, followed by n-BuLi (1.2 equiv, 120 mmol, 96 mL, 2.5 M in THF). The reaction mixture stirred at this temperature for 30 min and cooled to –60 °C. 2-Methylpyridine (1.0 equiv, 100 mmol, 9.3 g) in 20 mL of THF was added to the mixture at dropwise. The resulting mixture was stirred at this temperature for another 1 h and N-methoxy-N-methylbenzamide (1 equiv, 100 mmol, 16.5 g) in 20 mL of THF was slowly added. After addition, the mixture was stirred at –50 to –60 °C for another 1 h, and then was quenched with H₂O (100 mL). The biphasic mixture was concentrated to remove THF and extracted with ethyl acetate (200 mL). The organic layer was concentrated to give 20 g 1-phenyl-2-(pyridine-2-yl)ethanone as oil. To a 500 mL jacket reactor equipped with mechanical stirrer was added THF (400 mL), 1-phenyl-2-(pyridine-2-yl)ethanone (1 equiv, 102 mmol, 20 g), and followed by t-BuOK (1.2 equiv, 122 mmol, 18.4 g) in portions at 0–5 °C. The mixture was stirred at this temperature for 30 min, and EtI (1.1 equiv, 112 mmol, 17.4 g) was added. After addition, the mixture gradually warmed to 25 °C and stirred until completion. The reaction was quenched with H₂O (100 mL). The biphasic mixture was concentrated to remove THF and extracted with ethyl acetate (200 mL). The organic layer was concentrated, and then purified by silica gel column chromatography using 5% EtOAc in petroleum ether. The resulting light yellow oil was resublimed in 10% EtOAc in petroleum ether to give the product ketone 1s as a light yellow solid (7.2 g, 16%). ¹H NMR (400 MHz, CDCl₃) δ 8.58 (ddd, J = 5.0, 1.9, 0.9 Hz, 1H), 8.12–8.04 (m, 2H), 7.68 (td, J = 7.8, 1.8 Hz, 1H), 7.52–7.45 (m, 1H), 7.43–7.35 (m, 3H), 7.19 (ddd, J = 7.5, 5.0, 1.2 Hz, 1H), 4.90 (t, J = 7.4 Hz, 1H), 2.27 (dt, J = 13.6, 7.3 Hz, 1H), 2.07–1.91 (m, 1H), 0.94 (t, J = 7.4 Hz, 3H); ¹³C NMR (400 MHz, CDCl₃) δ 198.9, 158.9, 148.3, 138.0, 136.7, 133.1, 128.9, 128.6, 122.8, 122.3, 57.0, 26.5, 12.1.

3-methyl-1,2-diphenylbutan-1-one (1u): To a 2 L jacket reactor under nitrogen was added PhI (4.0 equiv, 1762 mmol, 359.4 g) and anhydrous THF (500 mL) at 20 °C. i-PrMgCl (5.5 equiv, 1211 mL, 2.0 M in THF) was added dropwise at –10 to 0 °C, and the reaction mixture was stirred for 30 min. To a separate reactor under nitrogen was added ethyl 2-amino-3-methylbutanoate (1.0 equiv, 440 mmol, 80.0 g) and anhydrous THF (300 mL) at –5 °C. The prepared Grignard reagent was slowly transferred at –5 to 10 °C, and the reaction mixture was stirred at –5 °C for 1 h and quenched with MeOH (350 mL) and H₂O (250 mL). The reaction was filtered and washed with THF (1000 mL). The filtrate was concentrated to approximately 150 mL, then diluted with H₂O (500 mL). The biphasic mixture was extracted with MTBE (1000 mL) and EtOAc (500 mL). The combined organic layers were concentrated to 100 mL. n-Heptane (1000 mL) was added and the mixture was concentrated again to 150 mL. The residue was stirred at 0–5 °C for 1 h and the solid was collected by filtration. The wet cake was dried in air for 2 h to give 2-amino-3-methyl-1,1-diphenylbutan-1-ol as a yellow solid (22.5 g, 20%). To as stirred solution of THF (200 mL) and H₂O (200 mL) was added 2-amino-3-methyl-1,1-diphenylbutan-1-ol (1.0 equiv, 165 mmol, 43 g). The solution was cooled to 0–5 °C and NaNO₂ (5.1 equiv, 842 mmol, 58.1 g) was added in portions, followed by slow addition of AcOH (10.1 equiv, 1671 mmol, 101 g). After addition, the mixture was continued to stir for 30 min at this temperature, then at 15–20 °C for 1 h. The reaction mixture was concentrated in vacuo to remove THF, and the residue was diluted with EtOAc (200 mL). The aqueous and organic layers were separated, and the organic layer was washed with H₂O (2x200 mL) and concentrated in vacuo. The residue was purified via silica gel column chromatography using 100% petroleum ether. The oil obtained was diluted with 100 mL n-heptane and stirred at 5–10 °C for 30 min. The solid was filtered and dried to obtain ketone 1u as an off-white solid (11.5 g, 55%). ¹H NMR (400 MHz, CDCl₃) δ 8.02–
7.94 (m, 2H), 7.51–7.44 (m, 1H), 7.43–7.31 (m, 4H), 7.31–7.24 (m, 2H), 7.23–7.15 (m, 1H), 4.21 (d, J = 10.1 Hz, 1H), 2.59 (hept, J = 10.1, 6.7 Hz, 1H), 1.01 (d, J = 6.4 Hz, 3H), 0.75 (d, J = 6.7 Hz, 3H); \(^{13}\)C NMR (400 MHz, CDCl\(_3\)) \(\delta\) 200.6, 138.6, 137.7, 132.8, 128.8, 128.7, 127.0, 61.4, 31.9, 22.0, 20.5.

References

(1) Stock solution of aqueous K\(_3\)PO\(_4\) for 20 reactions was prepared from dissolving K\(_3\)PO\(_4\)•H\(_2\)O (30 mmol, 7.28 g) in 10 mL H\(_2\)O and sparging with nitrogen for 15 min. The 0.54 mL volume added to each reaction was accounted for volume change in water after dissolving K\(_3\)PO\(_4\)•H\(_2\)O.


$^1$H and $^{13}$C NMR Spectra

$^1$H NMR (400 MHz, Chloroform-$d$): $\delta$ 7.51 – 7.41 (m, 2H), 7.18 – 7.11 (m, 3H), 7.07 (d, $J = 8.3$ Hz, 2H), 7.04 - 6.94 (m, 3H), 6.87 (d, $J = 4.4$ Hz, 4H), 2.68 (q, $J = 7.5$ Hz, 2H), 2.34 (s, 3H), 0.92 (t, $J = 7.5$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): 144.32, 142.48, 138.29, 136.89, 134.35, 133.90, 129.06, 129.38, 129.21, 128.11, 128.06, 127.59, 127.32, 127.03, 20.13, 21.55, 11.05.
$^1$H NMR (400 MHz, CDCl$_3$) δ 7.52 – 7.45 (m, 2H), 7.20 – 7.12 (m, 3H), 7.16 (d, 2 Hz, 2H), 7.08 – 6.98 (m, 2H), 6.85 – 6.75 (m, 2H), 6.47 – 6.36 (m, 2H), 3.66 (s, 3H), 2.66 (s, 3H), 2.50 (s, 3H), 2.91 (d, 8 Hz, 2H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ 128.97, 144.14, 142.26, 128.35, 133.60, 134.12, 131.19, 129.23, 129.19, 127.99, 126.85, 126.76, 112.78, 25.04, 26.04, 24.32, 11.97.
$^1$H NMR (400 MHz, Chloroform-d) δ 7.53 – 7.41 (m, 2H), 7.21 – 7.08 (m, 5H), 7.04 – 6.94 (m, 2H), 6.89 – 6.79 (m, 2H), 6.64 – 6.49 (m, 2H), 2.67 (q, 7.5 Hz, 3H), 2.57 (t, 7.5 Hz, 3H).

$^13$C NMR (100 MHz, Chloroform-d) 163.13, 160.66, 144.28, 141.54, 128.07, 127.15, 124.25, 131.73, 131.11, 130.07, 129.21, 128.28, 128.22, 127.96, 127.16, 114.30, 114.28, 26.16, 21.55, 11.89.
**1H NMR (400 MHz, CDCl3):** 7.50 – 7.39 (m, 3H), 7.17 – 7.10 (m, 2H), 2.04 (s, 3H, 2H), 2.55 (s, 3H, 3H), 7.04 – 6.82 (m, 7H), 2.04 (s, 3H), 7.54 (s, 1H).

**13C NMR (101 MHz, CDCl3):** 141.38, 142.98, 150.79, 133.72, 134.20, 133.54, 132.99, 129.77, 129.12, 127.62, 127.39, 127.19, 22.29, 21.36, 11.95.
PROTON 1H NMR (400 MHz, Chloroform-d) 6.40 – 7.42 (m, 2H), 7.10 – 7.04 (m, 2H), 7.03 – 6.95 (m, 3H), 6.90 – 6.82 (m, 6H), 2.04 (t, $^1$J = 7.5 Hz, 3H), 2.35 (s, 3H), 0.95 (s, 3H).

$^1$H NMR (400 MHz, CDCl$_3$) 103.02, 100.58, 144.35, 142.83, 135.90, 134.22, 134.15, 124.12, 123, 114.97, 110.95, 120.87, 120.74, 127.96, 127.08, 127.04, 113.27, 113.03, 26.11, 21.25, 11.91.

S46
$\text{H NMR (400 MHz, Chloroform-$d$)}$ 6.73 (dd, $J_F = 13.5, 8.5$ Hz, 4H), 7.19 – 6.98 (m, 5H), 6.96 – 6.87 (m, 4H), 2.73 (q, $J_F = 7.5$ Hz, 2H), 2.55 (t, 3H), 0.95 (t, $J_F = 7.5$ Hz, 3H).

$\text{C NMR (101 MHz, DMSO-$d_6$)}$ 145.02, 142.07, 142.14, 154.09, 155.06, 152.02, 138.02, 127.72, 129.12, 128.13, 127.80, 127.62, 127.51, 127.49, 127.47, 125.39, 125.13, 125.13, 125.08, 123.04, 1, 119.97, 25.13, 21.04, 11.62.

$\text{PRONTO}_{16}$ gene CCDC3 /opt/taprin ledI B2
$^1$H NMR (400 MHz, Chloroform-d): δ 7.34 (d, J = 8.6 Hz, 2H), 7.45 (d, J = 8.9 Hz, 2H), 7.14 – 7.04 (m, 4H), 7.01 – 6.94 (m, 3H), 6.97 – 6.93 (m, 3H), 4.33 (q, J = 7.1 Hz, 2H), 2.72 (q, J = 7.3 Hz, 2H), 2.34 (q, J = 7.1 Hz, 3H), 1.35 (t, J = 7.1 Hz, 3H), 0.91 (t, J = 7.5 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCls): δ 166.29, 144.44, 143.31, 143.22, 136.06, 134.22, 133.43, 129.09, 128.45, 129.38, 129.24, 127.96, 127.92, 127.51, 126.44, 25.91, 21.54, 14.30, 11.93.
**PROTON NMR (CDCl3, Chloroform-d):**
- 7.57 - 7.42 (m, 2H), 7.07 (d, J= 8.1 Hz, 3H), 7.05 - 6.96 (m, 3H), 6.90 (d, J= 4.4 Hz, 2H), 6.90 (d, J= 8.3, 7.7, 0.5 Hz, 2H), 6.60 (d, J= 7.7, 1.3 Hz, 2H), 6.55 (d, J= 2.6, 1.0 Hz, 2H), 3.64 (s, 3H), 2.67 (t, J= 7.5 Hz, 2H), 2.35 (s, 3H), 1.93 (s, 3H), 1.36 (s, 3H), 0.93 (s, 3H).

**CARBON13 NMR (CDCl3, Chloroform-d):**
- 129.26, 144.20, 142.60, 139.03, 136.74, 154.23, 133.95, 129.72, 129.13, 128.09, 127.29, 127.34, 121.91, 114.90, 112.31, 55.18, 20.36, 21.25, 11.98.
S$^4$ NMR (400 MHz, C$_{6}$D$_{6}$): 7.52 (d, $J = 8.5$ Hz, 2H). 7.18 - 6.79 (m, 11H). 2.82 - 2.59 (m, 2H). 2.59 - 2.44 (m, 1H). 2.35 (s, 3H). 2.05 (s, 3H). 0.91 (d, $J = 6.5$ Hz, 3H).

DC NMR ($^{13}$C): 142.28, 142.23, 137.45, 136.17, 125.89, 134.17, 134.05, 130.10, 129.22, 128.79, 128.19, 127.18, 127.28, 127.21, 121.48, 26.29, 21.34, 19.35, 11.39.

PROTON $^6$-DCl $^2$ /opt/topspin load 19

CARBON $^4$-DCl $^2$ /opt/topspin load 19

$Zr$
$^{1}H$ NMR (400 MHz, Chloroform-d$_{6}$): δ 4.55 (dd, $J = 4.9, 1.4$ Hz, 1H), 7.70 - 7.56 (m, 2H), 7.40 - 7.30 (m, 2H), 7.30 - 7.17 (m, 3H), 7.17 - 7.11 (m, 1H), 6.94 (d, $J = 8.1$ Hz, 2H), 5.55 (d, $J = 7.4$ Hz, 2H), 2.29 (s, 3H), 0.81 (d, $J = 7.5$ Hz, 3H).

$^{13}$C NMR (101 MHz, CDCl$_{3}$): δ 155.88, 140.11, 144.03, 145.03, 135.83, 134.73, 134.09, 133.36, 129.67, 129.10, 128.81, 127.95, 127.59, 125.65, 122.06, 25.10, 21.48, 12.88.
\textbf{S53}

\textbf{PROTON 1H NMR (400 MHz, Chloroform-$d$):} 7.51 – 7.43 (m, 2H), 7.26 (s, 1H), 7.19 – 7.12 (m, 2H), 7.07 (d, $J = 8.2$ Hz, 2H), 7.05 – 7.01 (m, 2H), 5.31 (s, 2H), 2.34 (s, 3H), 0.99 (dt, $J = 7.8$ Hz, 6H).

\textbf{CARBON 13 NMR (100 MHz, Chloroform-$d$):} 144.24, 142.17, 140.31, 133.73, 134.28, 133.98, 129.48, 129.04, 129.12, 127.98, 127.35, 127.70, 126.87, 29.22, 21.53, 20.78.

\textbf{2u}
**S54**

**PROTON 1H, g=6.80 MHz, Chloroform-d** 6.75 - 7.43 (m, 3H), 7.20 - 7.11 (m, 3H), 7.11 - 7.03 (m, 2H), 7.05 - 6.90 (m, 3H), 6.89 (s, 4H, =4.4 Hz, =0H), 5.70 (s, s, 1H), 10.0, 6.7 Hz, 1H), 3.05 (s, s, 17.1, 1.6 Hz, 1H), 4.09 (s, =10.0, 1.5 Hz, 1H), 3.44 (s, s, 6.7, 1.5 Hz, 2H), 2.75 (s, 3H)

**OTs**

2v

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**CARBON 13, g=6.80 MHz, Chloroform-d** 129.58, 125.07, 129.26, 127.43, 124.03, 123.96, 124.91, 129.02, 120.23, 124.80, 127.98, 127.75, 127.37, 127.71, 116.88, 37.33, 21.13

**OTs**

2v

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**f1 (ppm)**
**H NMR (400 MHz, Chloroform-d):**
- 7.51 – 7.42 (m, 2H)
- 7.22 – 7.12 (m, 3H)
- 7.12 – 7.03 (m, 4H)
- 7.03 – 6.93 (m, 4H)
- 6.88 (dd, 4.4 Hz, 4H)
- 2.35 (s, 3H)
- 2.22 (s, 3H)
- 1.97 – 1.89 (m, 2H)
- 0.80 (d, 10.8 Hz, 3H)
- 0.53 – 0.55 (m, 3H)
- 0.37 – 0.39 (m, 3H)
- 0.27 (m, 3H)
- 0.17 (m, 3H)

**C NMR (100 MHz, CDCl3):**
- 141.29, 142.81, 138.87, 135.49, 134.33, 133.85, 129.38, 129.42, 129.30, 127.53, 127.29, 126.83, 125.53, 121.33, 9.39, 4.78
\[ \text{H NMR (400 MHz, Chloroform-d):} \delta \text{ (d, J = 8.3 Hz, 2H)} 7.47 \text{ (d, J = 7.8 Hz, 2H), 7.17 - 7.10 (m, 3H), 7.07 (d, J = 7.7 Hz, 2H), 7.04 - 6.90 (m, 4H), 6.90 (d, J = 4.4 Hz, 4H), 6.64 (d, J = 7.3 Hz, 2H), 2.81 (s, 2H), 1.37 - 1.30 (m, 8H), 0.83 (t, J = 7.0 Hz, 3H).} \]

\[ \text{C NMR (101 MHz, CDC3):} \delta \text{ 144.31, 143.08, 138.99, 135.86, 134.44, 134.01, 129.68, 129.35, 129.21, 129.10, 128.00, 127.54, 127.33, 127.00, 32.62, 29.28, 22.44, 21.54, 13.85.} \]

\[ \text{Zx} \]
$^1$H NMR (400 MHz, Chloroform-d): δ 7.32 – 7.24 (m, 2H); 7.24 – 7.13 (m, 3H); 7.12 – 7.00 (m, 5H); 6.98 – 6.88 (m, 3H); 2.54 (q, J = 7.3 Hz, 2H); 1.00 (t, J = 7.5 Hz, 3H).

$^{13}$C NMR (100 MHz, Chloroform-d): δ 162.95, 160.51, 142.88, 142.68, 142.05, 139.84, 137.84, 131.02, 130.75, 129.67, 127.89, 127.46, 126.29, 125.91, 115.19, 114.99, 29.03, 13.57.
$^1$H NMR (400 MHz, Chloroform-d): δ 7.30 – 7.00 (m, 7H), 7.01 – 6.91 (m, 3H), 6.89 – 6.84 (m, 4H), 3.76 (s, 3H), 2.31 (q, J = 7.4 Hz, 2H), 0.94 (s, J = 7.4 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl3): δ 158.40, 143.43, 143.47, 141.98, 138.52, 136.08, 130.38, 130.66, 129.78, 127.85, 127.80, 127.38, 126.11, 125.73, 55.26, 29.10, 13.70.
$^1$H NMR (400 MHz, Chloroform-d) δ 8.10 – 7.98 (m, 2H), 7.30 – 7.27 (m, 2H), 7.22 – 7.16 (m, 4H), 7.18 – 7.15 (m, 4H), 7.03 – 7.00 (m, 2H), 4.37 (q, $J = 7.1$ Hz), 2.36 (q, $J = 7.1$ Hz), 2.37 (s, $J = 7.1$ Hz, 3H), 0.94 (t, $J = 7.5$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl₃) δ 166.52, 148.36, 143.22, 142.36, 141.75, 138.10, 130.78, 129.63, 129.57, 128.84, 128.35, 127.95, 127.56, 126.47, 126.10, 60.02, 29.08, 14.44, 13.56.


**S61**

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PROTON NMR (CDCl₃) (opt/optipip IX162 71)

- **4e**

- **A (6)**: 7.59, **B (6)**: 7.36, **C (m)**: 7.12, **D (m)**: 6.88, **E (s)**: 2.45, **F (s)**: 2.04, **G (s)**: 1.94, **H (t)**: 1.35

- **H NMR (400 MHz, Chloroform-d):** 7.59 (d, J = 8.1 Hz, 2H), 7.36 (d, J = 8.1 Hz, 2H), 7.20 – 7.04 (m, 4H), 7.04 – 6.92 (m, 4H), 6.88 (dd, J = 7.4 Hz, 2H), 2.45 (s, J = 7.4 Hz, 2H), 1.94 (s, J = 7.4 Hz, 2H)

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CARBON NMR (CDCl₃) (opt/optimip IX162 71)

- **4e**

- **6**

- **13C NMR (100 MHz, CDCl₃):** 147.29, 143.54, 142.30, 141.67, 137.71, 130.78, 129.99, 129.64, 128.01, 127.65, 126.57, 126.20, 125.27, 125.24, 29.05, 13.55

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S61
$^1$H NMR (400 MHz, Chloroform-d$_2$) δ 7.37 – 7.27 (m, 1H), 7.26 – 7.15 (m, 1H), 7.04 – 6.92 (m, 3H), 6.90 – 6.79 (m, 3H), 2.47 (q, $J = 7.4$ Hz, 2H), 0.95 (t, $J = 7.4$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ 142.88, 142.58, 141.95, 141.88, 137.67, 132.50, 130.87, 130.73, 129.81, 128.39, 127.97, 127.67, 126.33, 125.97, 29.01, 13.54.
$^1$H NMR (400 MHz, Chloroform-d) δ 7.54 - 7.28 (m, 11H), 7.20 - 7.07 (m, 7H), 6.99 - 6.85 (m, 4H), 2.38 - 2.23 (m, 2H), 2.14 (s, 3H), 0.84 (s, 3H), / (7.51 Hz, 3H).

$^{13}$C NMR (101 MHz, CDCl₃) δ 142.62, 142.30, 142.01, 141.38, 137.84, 136.22, 130.59, 130.40, 129.85, 129.77, 128.00, 127.38, 127.08, 126.36, 125.75, 125.60, 29.25, 19.96, 12.96.
$^1$H NMR (400 MHz, Chloroform-d): δ 7.97 – 7.29 (m, 1H), 7.24 (dd, $J = 5.8$, 3.0 Hz, 1H), 7.16 – 7.04 (m, 4H), 7.03 – 6.94 (m, 2H), 6.93 – 6.83 (m, 4H), 2.66 (q, $J = 7.5$ Hz, 2H), 1.98 (d, $J = 7.4$ Hz, 3H).

$^1$C NMR (101 MHz, CDCl3): δ 143.37, 143.03, 142.82, 142.23, 133.53, 130.64, 129.68, 129.36, 127.83, 127.41, 126.18, 125.90, 124.71, 122.91, 29.29, 13.70.
$^1$H NMR (400 MHz, Chloroform-d): δ 4.00 (s, 1H), 7.65 – 7.67 (m, 1H), 7.54 (d, J = 8.7 Hz, 1H), 7.26 (d, J = 8.6, 1H), 7.19 – 7.05 (m, 3H), 7.01 – 6.98 (m, 0H), 3.70 (d, J = 9.4 Hz, 1H), 4.04 (d, J = 11.9 Hz, 1H), 3.80 – 3.67 (m, 1H), 2.65 – 2.54 (m, 1H), 2.49 (q, J = 7.5 Hz, 2H), 2.11 (dd, J = 25.6, 13.0, 2.6 Hz, 2H), 1.82 – 1.38 (m, 3H), 0.95 (t, J = 7.4 Hz, 3H).


**Diagram:**

- **Path:** 68883-90
- **Proton NMR (CDC3):**
- **Carbon NMR (CDC3):**
\[ \text{PROTON}\]128\_gene\_CDCl3\_\text{/opt/tepsjin\_ics162\_2} \\

\[ ^1H\ \text{NMR}\ (400\ \text{MHz, CDCl}_3)\; \delta 7.07 - 6.97 (m, 4H), 6.97 - 6.87 (m, 4H), 6.66 - 6.79 (m, 2H), 2.73 (d, J = 7.5 Hz, 2H), 2.00 - 1.88 (m, 1H), 1.04 (d, J = 7.4 Hz, 3H), 0.70 - 0.63 (m, 2H), 0.33 - 0.19 (m, 2H). \]

\[ \text{CARBON}\_256\_\text{gene\_CDCl3\_\text{/opt/tepsjin\_ics162\_2}} \]

\[ ^13C\ \text{NMR}\ (101\ \text{MHz, CDCl}_3)\; \delta 143.66, 141.30, 140.04, 137.94, 130.77, 129.54, 127.34, 127.04, 125.56, 125.39, 27.52, 13.77, 13.00, 5.83. \]
$^1$H NMR (400 MHz, Chloroform-d$_6$): 7.36 – 7.29 (m, 2H), 7.28 – 7.20 (m, 3H), 7.19 – 7.05 (m, 5H), 6.98 – 6.79 (m, 2H), 6.72 – 6.62 (m, 2H), 2.46 (d, $J$ = 7.5 Hz, 2H), 0.93 (t, $J$ = 7.5 Hz, 3H).

$^{13}$C NMR (101 MHz, CDCl$_3$): 162.23, 159.80, 143.35, 142.58, 142.08, 138.99, 137.87, 132.25, 129.53, 128.26, 127.97, 126.77, 126.30, 114.51, 29.03, 13.50.
\[ ^1H\text{NMR (400 MHz, Chloroform-}d\text{)}: 7.37 – 7.30 (m, 2H), 7.29 – 7.22 (m, 3H), 7.20 – 7.07 (m, 5H), 6.82 – 6.73 (m, 3H), 6.58 – 6.51 (m, 2H), 3.66 (s, 1H), 2.45 (d, \text{j} = 7.4 Hz, 2H), 0.91 (d, \text{j} = 7.4 Hz, 3H). \]

\[ ^13C\text{NMR (101 MHz, CDCl}_3\text{)}: 157.51, 143.85, 142.46, 141.32, 138.28, 135.48, 131.88, 129.72, 129.47, 128.11, 127.88, 126.52, 126.03, 112.77, 55.00, 29.05, 13.61. \]
$^1$H NMR (400 MHz, Chloroform-d): δ 7.73 – 7.65 (m, 2H), 7.39 – 7.31 (m, 2H), 7.31 – 7.19 (m, 3H), 7.19 – 7.06 (m, 5H), 7.06 – 6.91 (m, 2H), 4.27 (q, $J = 7.1$ Hz, 2H), 2.40 (q, $J = 7.4$ Hz, 2H), 1.33 (q, $J = 7.2$ Hz, 3H), 0.93 (t, $J = 7.5$ Hz, 3H).

$^{13}$C NMR (101 MHz, CDCl$_3$): δ 166.35, 147.96, 143.85, 142.81, 141.66, 130.73, 129.73, 129.49, 128.71, 128.31, 128.03, 126.93, 126.90, 60.72, 29.14, 28.91, 14.33, 13.51.
$^{1}H$ NMR (400 MHz, Chloroform-d): δ 7.46 – 7.37 (m, 2H), 7.37 – 7.27 (cm, 4H), 7.27 – 7.11 (m, 4H), 7.05 (d, J = 8.2 Hz, 2H), 5.55 (q, J = 7.4 Hz, 2H), 1.00 (t, J = 7.3 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): δ 146.77, 144.09, 142.75, 141.53, 137.64, 130.98, 129.55, 128.40, 128.11, 127.02, 126.67, 124.41, 124.37, 124.34, 124.30, 29.20, 13.47.
**PROTON NMR** (DCDCl₃) 0.75: 0.74 (t, J = 6.8, 1.1 Hz, 3H), 7.58 – 7.30 (m, 3H), 7.20 – 7.06 (m, 5H), 6.69 – 6.90 (m, 2H), 6.44 – 6.73 (m, 2H), 2.46 (q, J = 7.4 Hz, 2H), 1.93 (t, J = 7.2 Hz, 3H).

**CARBON NMR** (DCDCl₃): 0 132.63, 143.06, 143.01, 141.85, 141.52, 137.64, 132.09, 131.57, 129.62, 129.48, 128.39, 128.05, 127.62, 126.46, 126.44, 29.13, 13.53.
$^1$H NMR (400 MHz, Chloroform-d$_2$): δ 7.32 – 7.22 (m, 3H), 7.22 – 7.14 (m, 1H), 7.11 – 6.98 (m, 5H), 6.96 – 6.85 (m, 5H), 2.73 – 2.45 (m, 2H), 2.05–s (s, 3H), 1.00–s, J = 7.4 Hz, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$): δ 142.30, 142.47, 142.27, 138.36, 136.10, 134.49, 128.98, 128.89, 129.24, 128.83, 129.04, 127.93, 126.51, 126.33, 125.00, 27.97, 20.30, 14.00.
$^1$H NMR (400 MHz, Chloroform-d) δ 7.27 - 7.14 (m, 2H), 7.07 - 6.91 (m, 9H), 6.87 (d, J = 6.1 Hz, 2H), 2.45 (q, J = 7.4 Hz, 2H), 2.24 (s, 3H), 1.90 (q, J = 7.4 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ 162.91, 160.47, 142.86, 139.34, 137.49, 135.80, 131.09, 130.77, 128.54, 126.64, 127.48, 125.81, 115.08, 20.01, 24.93, 21.18, 13.64.

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\[ ^{1}H\text{ NMR (400 MHz, Chloroform-}d_6\text{): 7.23 – 7.15 (m, 2H), 7.06 – 6.92 (m, 7H), 6.90 – 6.83 (m, 2H), 6.73 – 6.65 (m, 2H), 3.72 (s, 3H), 2.44 (q, } J = 7.3\text{ Hz, } 2\text{H), 1.33 (t, } J = 7.4\text{ Hz, } 3\text{H).} \]

\[ ^{13}C\text{ NMR (100 MHz, CDCl3): 162.87, 160.45, 158.02, 142.72, 139.71, 137.31, 134.14, 131.10, 130.78, 127.50, 125.75, 115.15, 113.33, 55.08, 28.93, 24.90, 13.63.} \]
$^1$H NMR (400 MHz, Chloroform-d): δ 7.94 (d, J = 8.4 Hz, 2H), 7.26 – 7.13 (m, 4H), 7.09 – 6.96 (m, 3H), 6.89 – 6.81 (m, 2H), 4.33 (q, J = 7.2 Hz, 2H), 3.90 (q, J = 7.5 Hz, 2H), 1.43 (t, J = 7.2 Hz, 3H), 0.93 (t, J = 7.4 Hz, 3H).

$^{13}$C NMR (101 MHz, CDCl₃): δ 166.5, 163.04, 169.00, 147.22, 142.35, 141.73, 139.09, 130.65, 129.96, 129.21, 129.30, 127.64, 132.29, 115.25, 90.82, 28.83, 28.88, 14.34, 13.46.
$^1$H NMR (400 MHz, Chloroform-d$_6$, δ 7.28 – 7.30 (m, 2H), 7.16 (d, J = 7.3, 3H, 1H), 7.12 – 6.02 (m, 9H), 6.79 (m, 2H), 2.39 (s, J = 7.5 Hz, 2H), 2.09 (s, 3H), 0.92 (s, J = 7.5 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$, δ 162.95, 160.90, 142.47, 141.25, 139.13, 138.12, 135.61, 131.34, 131.06, 129.83, 129.73, 127.25, 126.82, 125.98, 125.21, 115.12, 29.75, 19.76, 12.40.
\( ^1\text{H}NMR (400 \text{ MHz, CDCl}_3) \delta 7.27 - 7.17 (m, 2H), 7.37 - 7.05 (m, 4H), 7.05 - 6.94 (m, 8H), \\
6.90 - 6.83 (m, 2H), 2.12 (s, 3H). \)

\( ^{13}\text{C}NMR (101 \text{ MHz, CDCl}_3) \delta 162.86, 160.42, 143.89, 142.94, 139.44, 138.37, 136.12, 131.65, 130.84, \\
129.28, 127.74, 126.34, 126.05, 115.08, 23.39. \)
$^{1}$H NMR (400 MHz, Chloroform-d): δ 7.29 – 7.18 (m, 2H), 7.16 – 7.05 (m, 4H), 7.04 – 6.99 (m, 4H), 6.90 – 6.84 (m, 2H), 5.80 – 5.60 (m, 1H), 5.04 – 4.80 (m, 2H), 3.25 (s, $J = 6.5$ Hz, 1H).

$^{13}$C NMR (101 MHz, CDCl3): δ 163.09, 160.66, 142.75, 142.07, 139.69, 139.12, 138.23, 136.18, 131.28, 130.81, 129.77, 127.72, 126.26, 115.98, 115.05, 40.45, 24.94.

Path: 71452-152-02
PROTON32.gene (CD3Cl / opt/orcinol 1x162 73)

Path: 71452-152-02
CARBON256.gene (CD3Cl / opt/orcinol 1x162 73)
$^1$H NMR (400 MHz, Chloroform-d): δ 7.31 – 7.19 (m, 2F), 7.19 – 7.06 (m, 3H), 7.00 – 6.90 (m, 2H), 6.01 (d, $J = 5.8$ Hz, 2H), 7.41 – 7.33 (m, 2H), 7.25 – 6.99 (m, 2H), 2.49 (d, $J = 6.6$ Hz, 2H), 0.87 – 0.72 (m, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$): δ 162.95, 142.76, 134.21, 139.24, 138.25, 131.26, 130.61, 129.76, 127.78, 127.48, 126.30, 125.91, 115.08, 40.14, 10.43, 4.85.