SUPPORTING INFORMATION

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

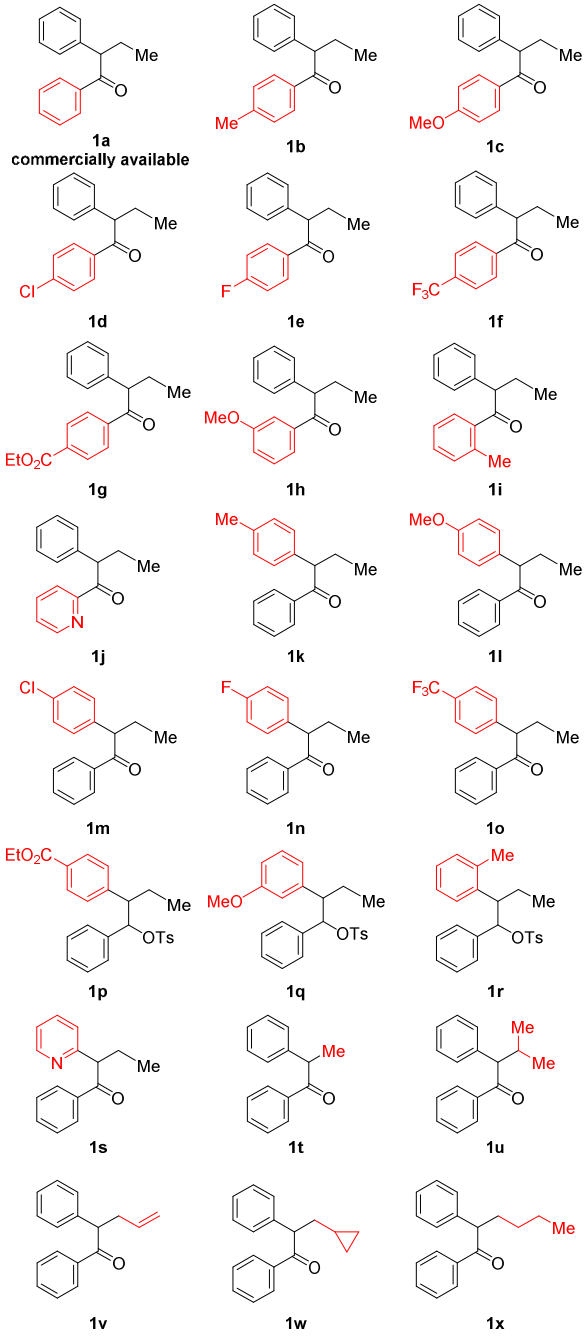


Table S2. Tosylates

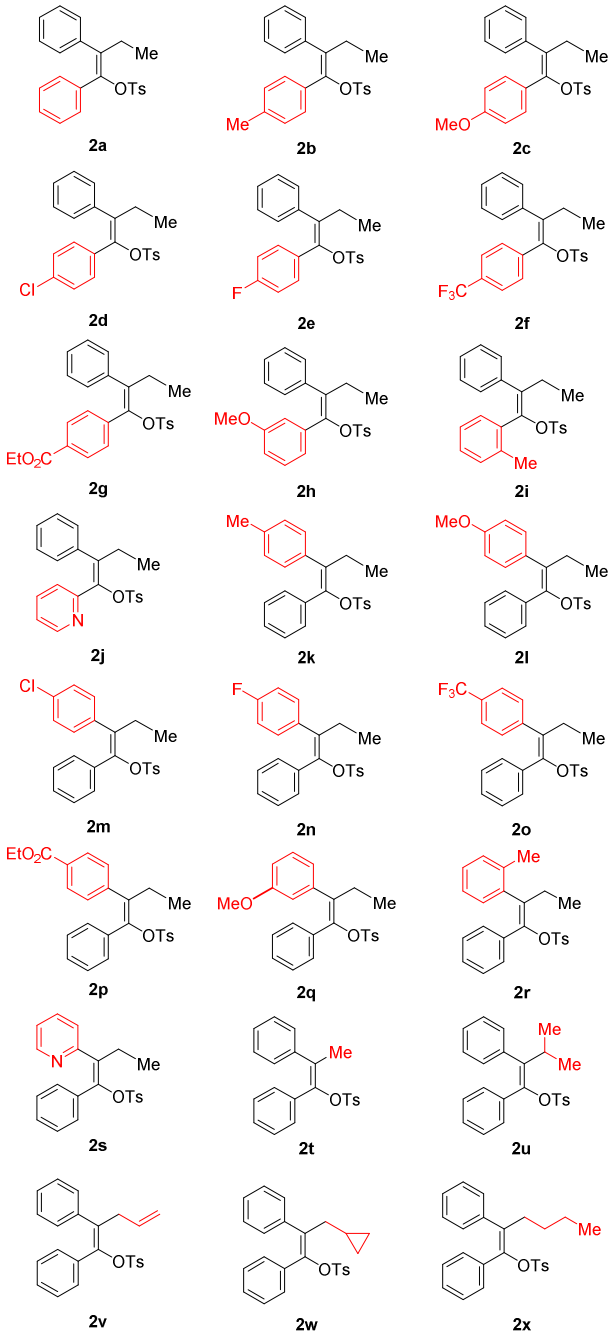


Table S3. Boronic esters and zinc halides (all commercially available)

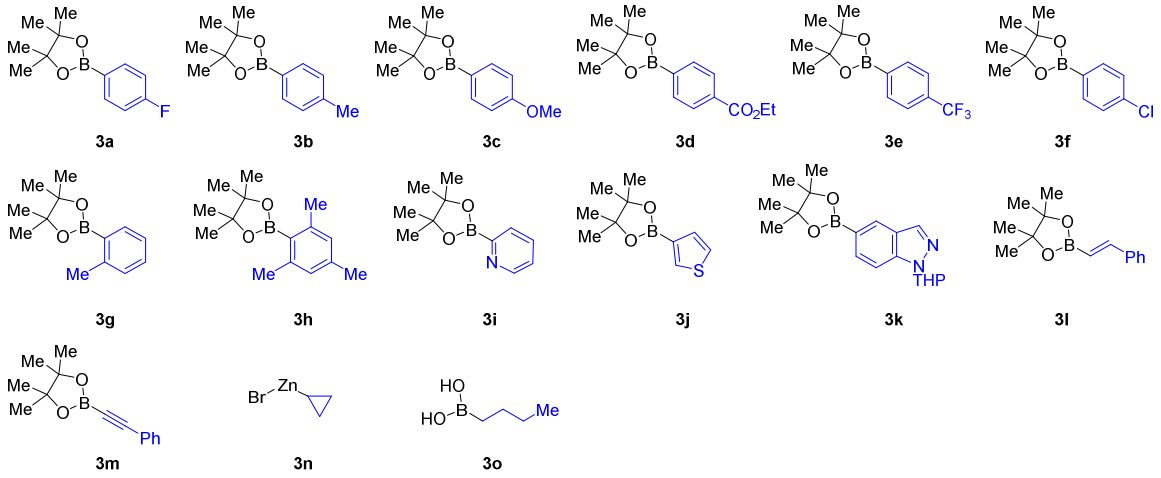
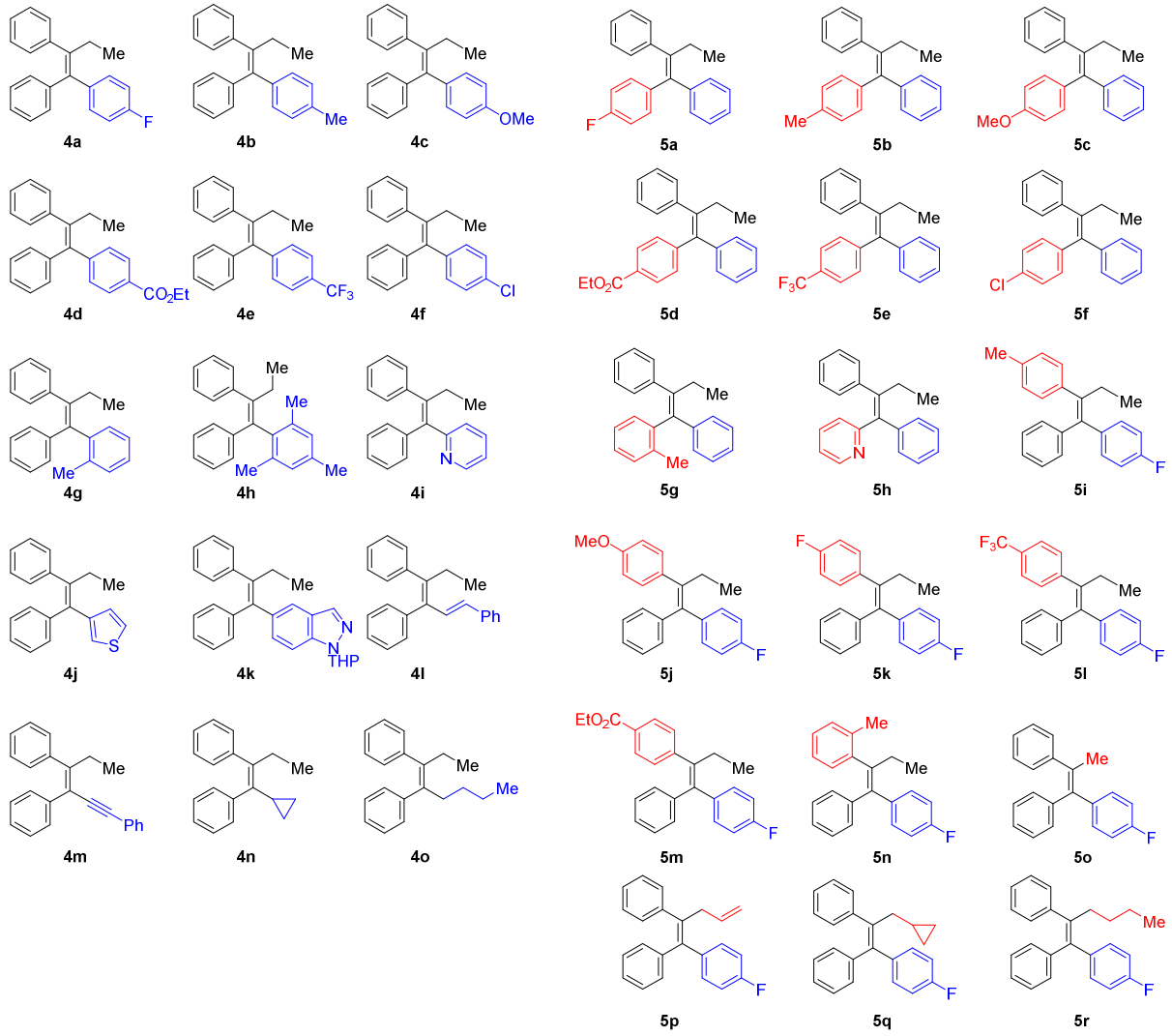


Table S4. Tetrasubstituted olefins

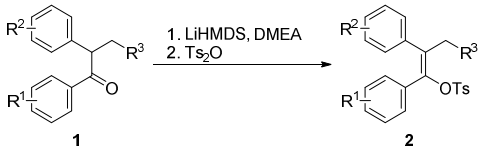


Materials and Methods

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}). ^1H NMR spectra were recorded on a Bruker 400 (at 400 MHz) and are reported relative to the residual solvent peak (δ 7.26 for $CDCl_3$). Data for ^1H NMR spectra are reported as follows: chemical shift (δ 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 (δ 77.0 for $CDCl_3$). Data for ^{13}C NMR spectra are reported in terms of chemical shift (δ 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 (δ 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 μL; temperature 23 °C; flow rate 1 mL/min; mobile phase A = 0.1% formic acid in H_2O (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.

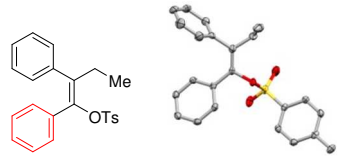
Experimental Procedures and Charaterization Data

Tosylate Synthesis

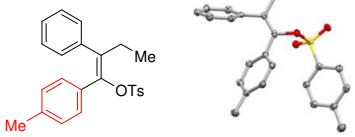


1 2

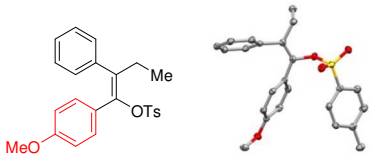
To a nitrogen-purged septum-top 40 mL vial equipped with a stir bar was added LiHMDS (2 equiv, 0.90 M in toluene, 4.4 mL) and *N*,*N*-dimethylethylamine (DMEA, 2 equiv, 0.43 mL). The reaction mixture was placed in a 23 °C water bath and a solution of ketone **1** (2.0 mmol, dissolved in 2 mL PhMe) was added via syringe. After stirring for 20 min, a solution of Ts_2O (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 H_2O, 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 Na_2SO_4 (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.



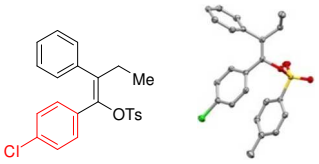
(*E*)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzenesulfonate (2a): Commercially available 1,2-diphenylbutan-1-one (**1a**, 2.0 mmol, 449 mg) was employed. Compound **2a** was obtained as a white solid (668 mg, 88%); mp 95–96 °C; HPLC (t_M = 3.66 min, t_m = 5.01 min) indicated >99:1 isomer ratio; FTIR (neat, cm^{-1}) 3057, 2968, 2923, 2874, 1597; ^1H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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 $C_{23}H_{22}O_3S$ ($[M–H]^-$), 377.1212; found, 377.1242.



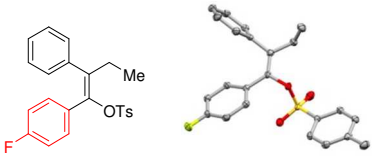
(*E*)- 2-Phenyl-1-(*p*-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (2b): 2-Phenyl-1-(*p*-tolyl) butan-1-one (**1b**, 2.0 mmol, 477 mg) was employed. Compound **2b** was obtained as a white solid (645 mg, 82%); mp 91–92 °C; HPLC (t_M = 3.71 min, t_m = 4.75 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 2972, 2924, 2872, 2855, 1595, 1363; ^1H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.2, 142.8, 138.5, 137.4, 136.1, 134.5, 131.0, 129.7, 129.4, 129.1, 128.1, 128.0, 126.9, 26.1, 21.6, 21.2, 12.0; HRMS (ESI–) *m/z* calculated for $C_{24}H_{24}O_3S$ ($[M–H]^-$), 391.1368; found, 391.1368.



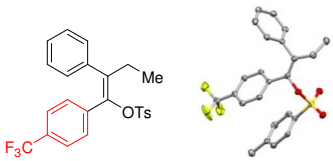
(*E*)-1-(4-Methoxyphenyl)-2-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2c): 1-(4-Methoxyphenyl)-2-phenyl-butan-1-one (**1c**, 2.0 mmol, 509 mg) was employed. Compound **2c** was obtained as a white solid (542 mg, 66%); mp 89–90 °C; HPLC (t_M = 4.99 min, t_m = 6.404 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 3016, 2960, 2932, 1608; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52–7.45 (m, 2H), 7.20–7.12 (m, 3H), 7.10 (d, *J* = 8.2 Hz, 2H), 7.08–6.98 (m, 2H), 6.84–6.75 (m, 2H), 6.47–6.36 (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); ^{13}C NMR (101 MHz, $CDCl_3$) 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 $C_{24}H_{24}O_4S$ ($[M–H]^-$), 407.1317; found, 407.1327.



(*E*)-1-(4-Chlorophenyl)-2-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2d): 1-(4-Chlorophenyl)-2-phenyl-butan-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 (t_M = 3.76 min, t_m = 4.99 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 3050, 2987, 2923, 2874, 1598, 1487, 1347; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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 $C_{23}H_{21}ClO_3S$ ($[M–H]^-$), 411.0822; found, 411.0853.

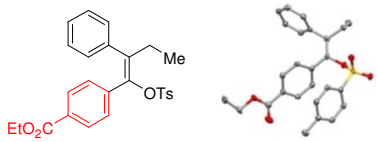


(*E*)-1-(4-Fluorophenyl)-2-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2e): 1-(4-Fluorophenyl)-2-phenyl-butan-1-one (**1e**, 2.0 mmol, 485 mg) was employed. Compound **2e** was obtained as a white solid (617 mg, 78%); mp 74–77 °C; HPLC (t_M = 3.78 min, t_m = 4.81 min) indicated >99:1 isomer ratio; FTIR (neat, cm^{-1}) 3045, 2988, 2924, 1598, 1506, 1368; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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; ^{19}F NMR (376 MHz, $CDCl_3$) δ –113.1; HRMS (ESI–) *m/z* calculated for $C_{23}H_{21}FO_3S$ ($[M–H]^-$), 395.1117; found, 395.1154.

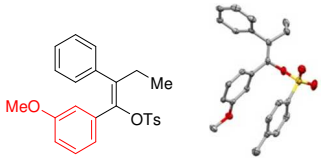


(*E*)-2-Phenyl-1-(4-(trifluoromethyl)phenyl)but-1-en-1-yl 4-methylbenzenesulfonate (2f): 2-Phenyl-1-(4-(trifluoromethyl)phenyl)butan-1-one (**1f**, 2.0 mmol, 585 mg) was employed. Compound **2e** was obtained as a white solid (712 mg, 80%); mp 114–115 °C; HPLC (t_M = 3.48 min, t_m = 4.56 min) indicated 96:4 isomer ratio; FTIR (neat, cm^{-1}) 2974, 2372, 2345, 1374, 1322; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.4 Hz, 2H), 7.23 – 7.14 (m, 3H), 7.14 – 6.99 (m, 6H), 6.93 (dt, *J* = 8.0, 0.8 Hz, 2H), 2.73 (q, *J* = 7.5 Hz, 2H), 2.33 (s, 3H), 0.93 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.8, 140.9, 139.1, 137.6, 137.5, 134.1, 130.0, 129.3, 129.24, 129.19 (q, *J* = 32.3

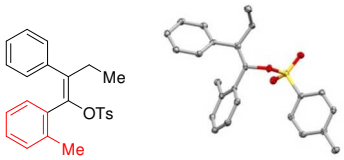
Hz), 129.0, 128.4, 128.0, 127.5, 124.2 (q, J = 2.8 Hz), 123.8 (q, J = 273.7 Hz), 26.3, 21.4, 11.8 ; HRMS (ESI–) m/z calculated for $C_{24}H_{21}F_3O_3S$ ([M–H]$^-$), 446.1163; found, 445.1133.



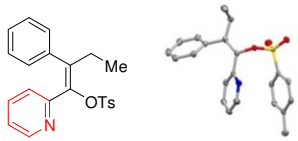
Ethyl (*E*)-4-(2-phenyl-1-(tosyloxy)but-1-en-1-yl)benzoate (2g): Ethyl 4-(2-phenylbutanoyl)benzoate (**1g**, 2.0 mmol, 593 mg) was employed. Compound **2g** was obtained as a white solid (780 mg, 87%); mp 87–88 °C; HPLC (t_M = 4.77 min, t_m = 6.18 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 3054, 2980, 2923, 1713, 1362, 1275; ^{1}H NMR (400 MHz, $CDCl_3$) δ 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 = 7.1 Hz, 3H), 0.91 (t, J = 7.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 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 $C_{26}H_{26}O_5S$ ([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-butan-1-one (**2h**, 2.0 mmol, 509 mg) was employed. Compound **2h** was obtained as a white solid (712 mg, 87%); mp 106–108 °C; 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, $CDCl_3$) δ 7.52–7.42 (m, 2H), 7.23–7.12 (m, 3H), 7.12–6.99 (m, 4H), 6.79 (t, J = 7.9 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, $CDCl_3$) δ 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 $C_{22}H_{21}NO_3S$ ([M–H]$^-$), 407.1317; found, 407.1317.

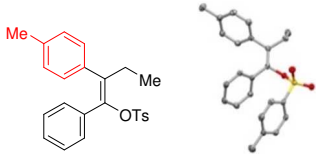


(*E*)-2-Phenyl-1-(*o*-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (2i): 1-(*o*-Tolyl)-2-phenyl-butan-1-one (**1i**, 2.0 mmol, 477 mg) was employed. Compound **2i** was obtained as a white solid (480 mg, 61%); mp 96–98 °C; HPLC (t_M = 3.62 min, t_m = 4.55 min) indicated 82:18 isomer ratio; FTIR (neat, cm^{-1}) 3055, 2976, 2922, 2863, 1596, 1442, 1356; ^{1}H NMR (400 MHz, $CDCl_3$) δ 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, $CDCl_3$) δ 144.1, 142.7, 138.2, 137.74, 137.67, 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 $C_{24}H_{24}O_3S$ ([M–H]$^-$), 391.1368; found, 391.1408.

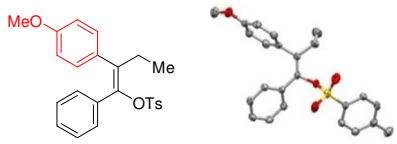


(*E*)-2-Phenyl-1-(pyridin-2-yl)but-1-en-1-yl 4-methylbenzenesulfonate (2j): 2-Phenyl-1-(pyridin-2-yl)butan-1-one (**1j**, 2.0 mmol, 451 mg) was employed. Compound **2j** was obtained as a white solid (620 mg, 82%); mp 89–93 °C; HPLC (t_M = 3.93 min, t_m = 5.21 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 3055, 2980, 2923, 2855, 1713, 1362; ^{1}H NMR (400 MHz, $CDCl_3$) δ 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,

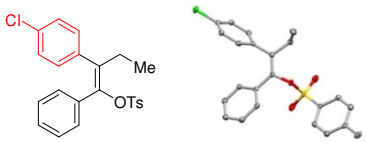
6H), 7.07–6.97 (m, 2H), 6.90 (ddd, J = 7.5, 4.8, 1.2 Hz, 1H), 6.77 (d, J = 7.9 Hz, 1H), 2.69 (q, J = 7.5 Hz, 2H), 2.38 (s, 3H), 0.92 (t, J = 7.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.8, 148.7, 144.4, 141.4, 139.5, 137.9, 135.1, 134.2, 129.3, 129.2, 128.2, 128.1, 127.4, 126.1, 122.1, 26.1, 21.6, 11.7; HRMS (ESI–) *m/z* calculated for $C_{22}H_{21}NO_3S$ ([M–H]$^-$), 378.1164; found, 378.1145.



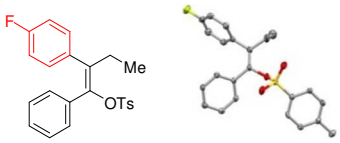
(*E*)-1-Phenyl-2-(*p*-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (2k): 1-Phenyl-2-(*p*-tolyl) butan-1-one (**1k**, 2.0 mmol, 477 mg) was employed. Compound **2k** was obtained as a white solid (660 mg, 84%); mp 115–117 °C; HPLC (t_M = 3.63 min, t_m = 4.93 min) indicated 95:5 isomer ratio; FTIR (neat, cm^{-1}) 3046, 2970, 2931, 1595, 1365; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.2, 142.4, 136.8, 136.7, 135.2, 134.4, 134.1, 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 $C_{24}H_{24}O_3S$ ([M–H]$^-$), 391.1368; found, 391.1388.



(*E*)-2-(4-Methoxyphenyl)-1-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2l): 2-(4-Methoxyphenyl)-1-phenyl-butan-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 (t_M = 4.79 min, t_m = 6.43 min) indicated 95:5 isomer ratio; FTIR (neat, cm^{-1}) 2995, 2967, 2922, 1606, 1508, 1346; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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 $C_{22}H_{21}NO_3S$ ([M–H]$^-$), 407.1317; found, 407.1284.

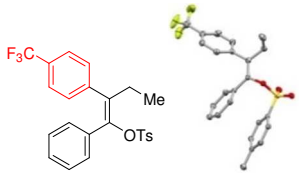


(*E*)-2-(4-Chlorophenyl)-1-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2m): 2-(4-Chlorophenyl)-1-phenyl-butan-1-one (**1m**, 2.0 mmol, 517 mg) was employed. Compound **2m** was obtained as a white solid (600 mg, 80%); mp 123–125 °C; HPLC (t_M = 3.88 min, t_m = 4.77 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 2975, 2961, 2932, 1596, 1487, 1444, 1347; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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 $C_{23}H_{21}ClO_3S$ ([M–H]$^-$), 411.0822; found, 411.0883.

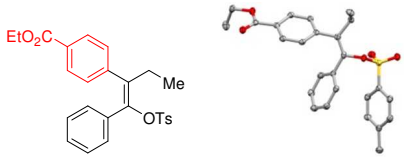


(*E*)-2-(4-Fluorophenyl)-1-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2n): 2-(4-Fluorophenyl)-1-phenyl-butan-1-one (**1n**, 2.0 mmol, 485 mg) was employed. Compound **2n** was obtained as a white solid (600 mg, 76%); mp 121–122 °C; HPLC (t_M = 3.85 min, t_m = 4.79 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 3041, 2973, 2939, 2876, 1598, 1507, 1446, 1347; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101

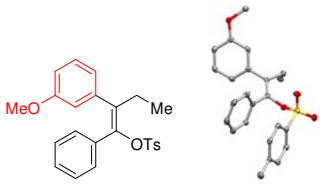
MHz, $CDCl_3$) δ 161.80 (d, *J* = 246.4 Hz), 144.4, 142.8, 135.9, 134.3, 134.1 (d, *J* = 3.6 Hz), 133.7, 131.0 (d, *J* = 8.0 Hz), 129.9, 129.2, 128.0, 127.7, 127.4, 115.2 (d, *J* = 21.3 Hz), 26.1, 21.6, 11.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ –114.9; HRMS (ESI–) *m/z* calculated for $C_{23}H_{21}FO_3S$ ([M – H]$^-$), 395.1117; found, 395.1202.



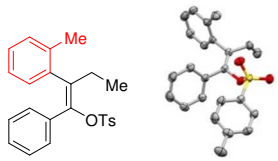
(*E*)-1-Phenyl-2-(4-(trifluoromethyl)phenyl)but-1-en-1-yl 4-methylbenzenesulfonate (2o): 1-Phenyl-2-[4-(trifluoromethyl)phenyl]butan-1-one (**1o**, 2.0 mmol, 585 mg) was employed. Compound **2o** was obtained as a white solid (790 mg, 88%); mp 97–98 °C; HPLC (t_M = 3.65 min, t_m = 4.36 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 3061, 2996, 2969, 2937, 1597, 1324; ^{1}H NMR (400 MHz, $CDCl_3$) δ 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) δ 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, $CDCl_3$) δ –62.6; HRMS (ESI–) *m/z* calculated for $C_{24}H_{21}F_3O_3S$ ([M–H]$^-$), 445.1085; found, 445.1114.



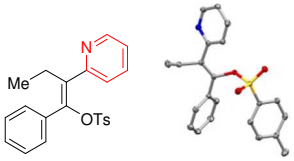
Ethyl (*E*)-4-(1-phenyl-1-(tosyloxy)but-1-en-2-yl)benzoate (2p): Ethyl 4-(1-benzoylpropyl) benzoate (**1p**, 2.0 mmol, 593 mg) was employed. Compound **2p** was obtained as a white solid (860 mg, 95%); mp 63–64 °C; HPLC (t_M = 5.23 min, t_m = 6.17 min) indicated 99:1 isomer ratio; FTIR (neat, cm^{-1}) 3065, 2973, 2936, 2875, 1708, 1605, 1372, 1290; ^{1}H NMR (400 MHz, $CDCl_3$) δ 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), 0.91 (t, *J* = 7.5 Hz, 3H).; ^{13}C NMR (101 MHz, DMSO-d_6) δ 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 $C_{26}H_{26}O_5S$ ([M–H]$^-$), 449.1423; found, 449.1483.



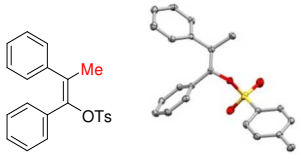
(*E*)-2-(3-Methoxyphenyl)-1-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (2q): 2-(3-Methoxyphenyl)-1-phenyl-butan-1-one (**1q**, 2.0 mmol, 509 mg) was employed. Compound **2q** was obtained as a white solid (640 mg, 78%); mp 84–85 °C; HPLC (t_M = 4.41 min, t_m = 5.73 min) indicated 95:5 isomer ratio; FTIR (neat, cm^{-1}) 3054, 2977, 2933, 2874, 1712, 1594; ^{1}H NMR (400 MHz, $CDCl_3$) δ 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 (ddd, *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, $CDCl_3$) δ 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 $C_{24}H_{24}O_4S$ ([M–H]$^-$), 407.1317; found, 407.1200.



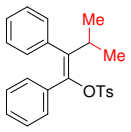
(*E*)-1-Phenyl-2-(*o*-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (2r): 2-(*o*-Tolyl)-1-phenyl-butan-1-one (**1r**, 2.0 mmol, 477 mg) was employed. Compound **2r** was obtained as a white solid (390 mg, 50%); mp 85–87 °C; HPLC (t_M = 3.78 min, t_m = 4.73 min) indicated 95:5 isomer ratio; FTIR (neat, cm^{-1}) 3023, 2973, 2933, 1597, 1492, 1443, 1360; ^{1}H NMR (400 MHz, $CDCl_3$) δ 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, $CDCl_3$) δ 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 $C_{24}H_{24}O_3S$ ($[M–H]^-$), 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, $CDCl_3$) δ 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, $CDCl_3$) δ 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 $C_{22}H_{21}NO_3S$ ($[M–H]^-$), 378.1164; found, 378.1215.

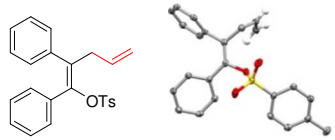


(*E*)-1,2-Diphenylprop-1-en-1-yl 4-methylbenzenesulfonate (2t): 1,2-Diphenylpropan-1-one (**1t**, 2.0 mmol, 421 mg) was employed. Compound **2t** was obtained as a white solid (341 mg, 47%); mp 91–93 °C; HPLC (t_M = 4.45 min, t_m = 5.77 min) indicated 83:17 isomer ratio; FTIR (neat, cm^{-1}) 3060, 3029, 2919, 2870, 1650, 1597, 1372; ^{1}H NMR (400 MHz, $CDCl_3$) δ 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, $CDCl_3$) δ 144.4, 143.3, 140.0, 134.4, 134.0, 131.0, 129.8, 129.2, 128.8, 128.2, 128.0, 127.7, 127.4, 127.1, 21.6, 19.9; HRMS (ESI–) *m/z* calculated for $C_{22}H_{20}O_3S$ ($[M–H]^-$), 363.1055; found, 363.1112.

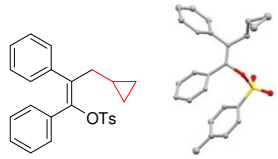


(*E*)-3-Methyl-1,2-diphenylbut-1-en-1-yl 4-methylbenzenesulfonate (2u): Standard LiHMDS/DMEA conditions lead to decomposition. Alternative, less stereoselective Li*O*t*B*u conditions were used to prepare material for subsequent Suzuki coupling studies. To a nitrogen-purged septum-top 20 mL vial equipped 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 Li*O*t*B*u (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_2O (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

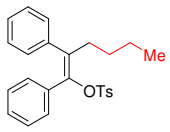
purified by silica gel column chromatography, eluent conditions 0–15% *i*-PrOAc in heptane. Compound **2u** was obtained as a white solid (104 mg, 27%); mp 137–138 °C; HPLC (t_M = 3.68 min, t_m = 4.73 min) indicated 85:15 isomer ratio; FTIR (neat, cm^{-1}) 3061, 3032, 2969, 2926, 2856, 2373, 2345, 1648, 1373; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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 $C_{24}H_{24}O_3S$ ([M–H]$^-$), 391.1368; found, 391.1412.



(*E*)-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; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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 $C_{24}H_{22}O_3S$ ([M–H]$^-$), 389.1212; found, 389.1254.

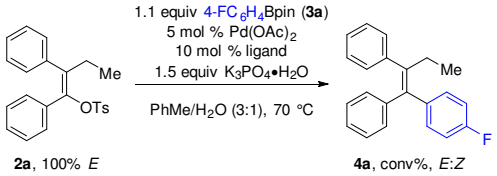


(*E*)-3-Cyclopropyl-1,2-diphenylprop-1-en-1-yl 4-methylbenzenesulfonate (2w): 3-Cyclopropyl-1,2-diphenyl-propan-1-one (**1w**, 2.0 mmol, 501 mg) was employed. Compound **2w** was obtained as a white solid (660 mg, 82%); mp 113–114 °C; HPLC (t_M = 3.73 min, t_m = 5.08 min) indicated 95:5 isomer ratio; FTIR (neat, cm^{-1}) 3066, 2981, 2920, 1597, 1362; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51–7.42 (m, 2H), 7.22–7.12 (m, 3H), 7.12–7.03 (m, 4H), 7.03–6.93 (m, 1H), 6.88 (d, *J* = 4.4 Hz, 4H), 2.57 (d, *J* = 7.0 Hz, 2H), 2.35 (s, 3H), 0.71–0.55 (m, 1H), 0.40–0.27 (m, 2H), 0.13–0.03 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) 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 $C_{25}H_{24}O_3S$ ([M–H]$^-$), 403.1368; found, 403.1414.



(*E*)-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; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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, 127.0, 32.6, 29.3, 22.4, 21.5, 13.9; HRMS (ESI–) *m/z* calculated for $C_{25}H_{26}O_3S$ ([M–H]$^-$), 405.1525; found, 405.1506.

High-throughput Screening

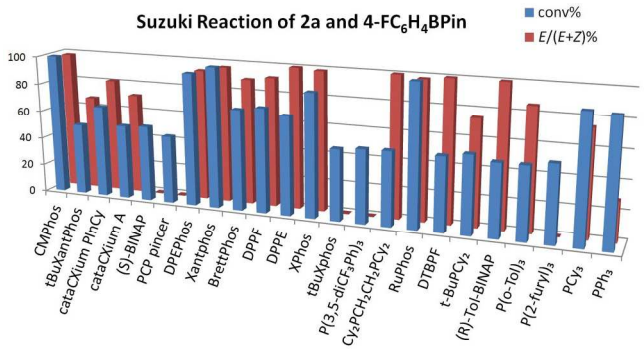


2a, 100% *E* **4a**, conv%, *E*:*Z*

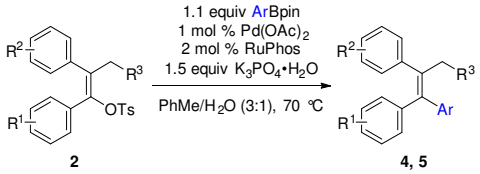
The high-throughput screening of the Suzuki-Miyaura coupling was conducted employing tosylate **2a** (20 mg, 53 μmol), boronic ester **3a** (1.1 equiv), $Pd(OAc)_2$ (5 mol %), ligand (10 mol %), K_3PO_4•H_2O (1.5 equiv), PhMe/H_2O (3:1, 0.20 mL, 100 mg/mL) at 70 °C for 18 h. The results are shown below.

entry	ligand	conv%	*E*/(*E*+*Z*)%	entry	ligand	conv%	*E*/(*E*+*Z*)%
1	CMPhos	100	98	13	*t*-BuXPhos	50	0[a]
2	*t*-BuXantPhos	51	67	14	P(3,5-diCF_3Ph)$_3$	52	0[a]
3	cataCXium PInCy	65	81	15	$Cy_2PCH_2CH_2PCy_2$	52	100
4	cataCXium A	53	71	16	RuPhos	100	98
5	(*S*)-BINAP	54	0[a]	17	DTBPF	52	100
6	PCP pincer	48	0[a]	18	*t*-BuPCy_2	55	75
7	DPEPhos	94	93	19	(*R*)-Tol-BINAP	51	100
8	XantPhos	100	97	20	P(o-Tol)$_3$	51	86
9	BrettPhos	72	89	21	P(2-furyl)$_3$	54	0[a]
10	DPPF	74	92	22	PCy_3	89	75
11	DPPE	70	100	23	PPh_3	88	29
12	XPhos	88	99				

[a]None of the *E*- or *Z*- olefin isomer was produced in the reaction.



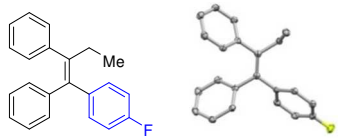
Olefin Synthesis



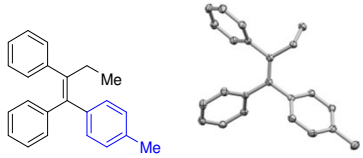
2 **4, 5**

To a 4 mL septum-top vial equipped with a stir bar was added *E*-tosylate (**2**, 1.0 mmol), boronic ester **3** (1.1 equiv), 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×). Degassed PhMe (1.5 mL) and degassed aqueous K_3PO_4 (1.5 mmol, 364 mg, 0.54 mL[1]) were added via syringes. Then the vial was heated to 70 °C while

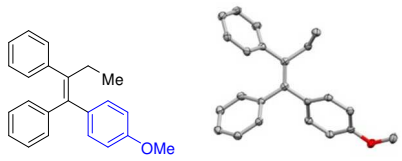
stirring vigorously (1500 rpm). The reaction was sampled by HPLC at 1 h and overnight (16–24 h). Analytical HPLC analyses were performed with an Agilent 1260 Infinity Series HPLC instrument; the column used was ACE Excel 3 C18 HL, 3×50mm; particle size 3 um; injection volume 2 µL; temperature 35 °C; flow rate 1 mL/min; mobile phase A = 0.05% trifluoroacetic acid in H_2O, 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 H_2O, 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 Na_2SO_4 (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.



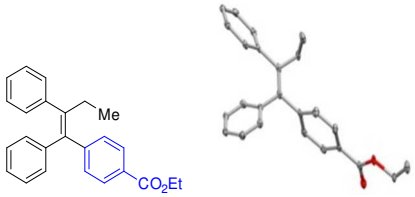
(*E*)-(1-(4-Fluorophenyl)but-1-ene-1,2-diyl)dibenzene (4a): (*E*)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (**2a**, 2 mmol, 757 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (**3a**, 1.1 equiv, 448 mg) were employed. Amounts of other reagents and solvents were scaled accordingly. Compound **4a** was obtained as a white solid (590 mg, 98%); mp 71–72 °C; HPLC (t_M = 8.73 min, t_m = 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, $CDCl_3$) δ 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); ^{13}C NMR (101 MHz, $CDCl_3$) δ 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; ^{19}F NMR (376 MHz, $CDCl_3$) δ –115.8; HRMS was not collected because compound **4a** does not ionize under both positive and negative modes.



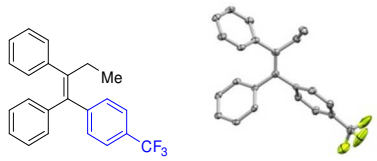
(*E*)-(1-(*p*-Tolyl)but-1-ene-1,2-diyl)dibenzene (4b): (*E*)-1,2-Diphenylbut-1-en-1-yl 4-methyl benzene sulfonate (**2a**, 1 mmol, 379 mg) and 4,4,5,5-tetramethyl-2-(*p*-tolyl)-1,3,2-dioxaborolane (**3b**, 1.1 equiv, 240 mg) were employed. Compound **4b** was obtained as a white solid (292 mg, 98%); mp 90–91 °C; HPLC (t_M = 10.78 min, t_m = 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, $CDCl_3$) δ 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); ^{13}C NMR (101 MHz, $CDCl_3$) δ 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.



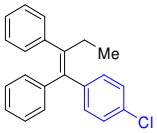
(*E*)-(1-(4-Methoxyphenyl)but-1-ene-1,2-diyl)dibenzene (4c): (*E*)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (**2a**, 1 mmol, 379 mg) and 2-(4-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (**3c**, 1.1 equiv, 263 mg) were employed. Compound **4c** was obtained as a white solid (301 mg, 96%); mp 89–90 °C; HPLC (t_M = 7.73 min, t_m = 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, $CDCl_3$) δ 7.20–7.03 (m, 7H), 7.01–6.91 (m, 3H), 6.90–6.84 (m, 4H), 3.78 (s, 3H), 2.51 (q, *J* = 7.4 Hz, 2H), 0.94 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.4, 143.4, 142.5, 142.0, 138.5, 136.1, 130.9, 130.7, 129.8, 127.9, 127.8, 127.4, 126.1, 125.7, 55.3, 29.1, 13.7; HRMS (ESI+) *m/z* calculated for $C_{23}H_{23}O$ ($[M+H]^+$), 315.1749; found, 315.1755.



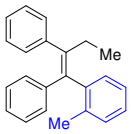
Ethyl (*E*)-4-(1,2-diphenylbut-1-en-1-yl)benzoate (4d): (*E*)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (**2a**, 1 mmol, 379 mg) and ethyl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (**3d**, 1.1 equiv, 304 mg) were employed. Compound **4d** was obtained as a white solid (340 mg, 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, 1604, 1475, 1442, 1365; ^{1}H NMR (400 MHz, $CDCl_3$) δ 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, $CDCl_3$) δ 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.



(*E*)-(1-(4-(Trifluoromethyl)phenyl)but-1-ene-1,2-diyl)dibenzene (4e): (*E*)-1,2-Diphenylbut-1-en-1-yl 4-methyl benzene sulfonate (**2a**, 1 mmol, 379 mg) and 4,4,5,5-tetramethyl-2-(4-(trifluoromethyl)phenyl)-1,3,2-dioxaborolane (3**e**, 1.1 equiv, 305 mg) were employed. Compound **4e** was obtained as a white solid (343 mg, 97%); mp 96–97 °C; HPLC (t_M = 10.69 min, t_m = 10.59 min) indicated >99:1 isomer ratio; FTIR (neat, cm^{-1}) 2977, 2872, 1712, 1604, 1489, 1442, 1402, 1363; ^{1}H NMR (400 MHz, $CDCl_3$) δ 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), 6.86 (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, $CDCl_3$) δ 147.3, 143.5, 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* = 272.7 Hz), 29.1, 13.6. ^{19}F NMR (376 MHz, $CDCl_3$) δ –62.3; HRMS (ESI–) *m/z* calculated for $C_{23}H_{19}F_3$ ([M–H]$^-$), 351.1361; found, 351.1362.

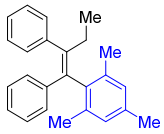


(*E*)-(1-(4-Chlorophenyl)but-1-ene-1,2-diyl)dibenzene (4f): (*E*)-1,2-Diphenylbut-1-en-1-yl 4-methyl benzene sulfonate (**2a**, 1 mmol, 379 mg) and 4-chlorophenylboronic acid pinacol ester (**3f**, 2.1 equiv, 512 mg) were employed. Palladium(II) acetate and 2-dicyclohexylphosphino-2',6'-diisopropoxy-1,1'-biphenyl (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 = 11.39 min, t_m = 11.30 min) indicated 99:1 isomer ratio; FTIR (neat, cm^{-1}) 2973, 2872, 1488, 1441; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37–7.27 (m, 1H), 7.26–7.05 (m, 8H), 7.04–6.92 (m, 3H), 6.90–6.79 (m, 2H), 2.47 (q, *J* = 7.4 Hz, 2H), 0.93 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.9, 142.6, 142.0, 141.9, 137.7, 132.5, 130.9, 130.7, 129.6, 128.4, 127.9, 127.5, 126.3, 126.0, 29.0, 13.5; HRMS was not collected because compound **4f** does not ionize under both positive and negative modes.

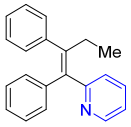


(*E*)-(1-(*o*-Tolyl)but-1-ene-1,2-diyl)dibenzene (4g): (*E*)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (**2a**, 1 mmol, 379 mg) and 4,4,5,5-tetramethyl-2-(*o*-tolyl)-1,3,2-dioxaborolane (**3g**, 1.1 equiv, 458 mg) were employed.

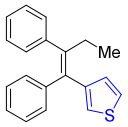
Palladium(II) acetate and 2-dicyclohexylphosphino-2',6'-diisopropoxy-1,1'-biphenyl (RuPhos) loadings were doubled to 2 mol % and 4 mol %, respectively. Other reagents remained unchanged from general procedure. Compound **4g** was obtained as an opaque oil (258 mg, 86%); HPLC (t_M = 10.24 min, t_m = 9.29 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 3055, 3017, 2930, 2871, 1597, 1490, 1441, 1078, 1030; ^{1}H NMR (400 MHz, $CDCl_3$) δ 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, $CDCl_3$) δ 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.



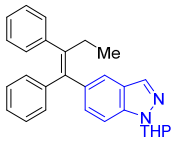
(*E*)-(1-Mesitylbut-1-ene-1,2-diyl)dibenzene (4h): (*E*)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (**2a**, 1 mmol, 379 mg) and 4,4,5,5-tetramethyl-2-(2,4,6-trimethylphenyl)-1,3,2-dioxaborolane (**3k**, 1.5 equiv, 369 mg) were employed. Reaction showed <5% conversion under standard conditions.



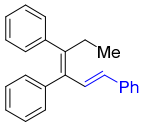
(*E*)-2-(1,2-Diphenylbut-1-en-1-yl)pyridine (4i): (*E*)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (**2a**, 1 mmol, 379 mg) and pyridine-2-boronic acid pinacol ester (**3i**, 1.1 equiv, 233 mg) were employed. Reaction showed <5% conversion under standard conditions.



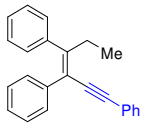
(*E*)-3-(1,2-Diphenylbut-1-en-1-yl)thiophene (4j): (*E*)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (**2a**, 1 mmol, 379 mg) and 4,4,5,5-tetramethyl-2-(3-thienyl)-1,3,2-dioxaborolane (**3j**, 1.5 equiv, 315 mg) were employed. Palladium(II) acetate and 2-dicyclohexylphosphino-2',6'-diisopropoxy-1,1'-biphenyl (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_M = 7.83 min, t_m = 8.75 min) indicated 97:3 isomer ratio; FTIR (neat, cm^{-1}) 3094, 3028, 2923, 2865, 1486, 1371; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37–7.29 (m, 1H), 7.24 (dd, *J* = 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, *J* = 7.5 Hz, 2H), 0.98 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) 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+) *m/z* calculated for $C_{20}H_{18}S$ ($[M+H]^+$), 291.1207; found, 291.1176.



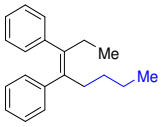
(*E*)-5-(1,2-Diphenylbut-1-en-1-yl)-1-(tetrahydro-2H-pyran-2-yl)-2,3-dihydro-1H-indazole (4k): (*E*)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (**2a**, 1 mmol, 379 mg) and 1-tetrahydropyran-2-yl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)indazole (**3k**, 1.1 equiv, 361 mg) were employed. Compound **4k** was obtained as a white solid (409 mg, 84%); mp 94–97 °C; HPLC (t_M = 8.06 min, t_m = 8.29 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 2961, 2848, 1504, 1440; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (s, 1H), 7.65–7.60 (m, 1H), 7.54 (d, *J* = 8.7 Hz, 1H), 7.26 (dd, *J* = 8.6, 1.5 Hz, 1H), 7.19–7.05 (m, 3H), 7.01–6.85 (m, 6H), 5.70 (dd, *J* = 9.6, 2.6 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 (ddt, *J* = 25.6, 13.0, 2.6 Hz, 2H), 1.82–1.58 (m, 3H), 0.95 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) 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+) *m/z* calculated for $C_{28}H_{28}N_2O$ ($[M+H]^+$), 409.2280; found, 409.2226.



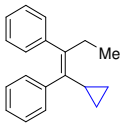
((1*E*,3*Z*)-Hexa-1,3-diene-1,3,4-triyl)tribenzene (4l): (*E*)-1,2-Diphenylbut-1-en-1-yl 4-methyl benzene sulfonate (**2a**, 1 mmol, 379 mg) and 4,4,5,5-tetramethyl-2-[(*E*)-styryl]-1,3,2-dioxaborolane (**3l**, 1.50 equiv, 345 mg) were employed. Palladium(II) acetate and 2-dicyclohexylphosphino-2',6'-diisopropoxy-1,1'-biphenyl (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_M = 10.72 min, t_m = 12.90 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 3055, 3025, 2966, 2931, 2872, 1598, 1489, 1442;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 15.8 Hz, 1H), 7.40–7.34 (m, 2H), 7.29 (dd, *J* = 8.4, 6.8 Hz, 2H), 7.22–6.93 (m, 11H), 6.18 (d, *J* = 15.9 Hz, 1H), 2.82 (q, *J* = 7.5 Hz, 2H), 1.13 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) 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+) *m/z* calculated for $C_{24}H_{22}$ ($[M+H]^+$), 311.1800; found, 311.1840.



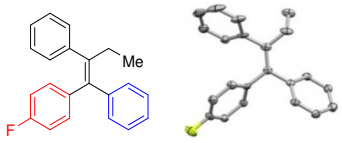
(*E*)-Hex-3-en-1-yne-1,3,4-triyltribenzene (4m): (*E*)-1,2-Diphenylbut-1-en-1-yl 4-methylbenzene sulfonate (**2a**, 1 mmol, 379 mg) and 4,4,5,5-tetramethyl-2-(2-phenylethynyl)-1,3,2-dioxaborolane (**3m**, 2.1 equiv, 479 mg) were employed. Compound **4m** was obtained as a yellow oil (188 mg, 61%); HPLC (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; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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 $C_{24}H_{20}$ ([M+H]$^+$), 309.1643; found, 309.1581.



(*Z*)-Oct-3-ene-3,4-diyldibenzene (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; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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.

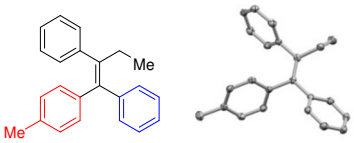


(*Z*)-(1-Cyclopropylbut-1-ene-1,2-diyl)dibenzene (4n): To a 8 mL septum-top vial equipped with a stir bar was added (*E*)-1,2-diphenylbut-1-en-1-yl 4-methyl benzenesulfonate compound (**2a**, 1.0 mmol, 379 mg), palladium(II) acetate (1 mol %, 2.3 mg), and 2-dicyclohexylphosphino-2',6'-diisopropoxy-1,1'-biphenyl (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 H_2O, 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 Na_2SO_4 (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; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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.

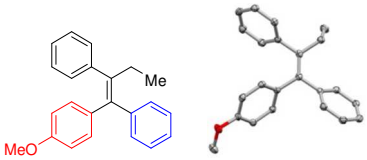


(*Z*)-(1-(4-Fluorophenyl)but-1-ene-1,2-diyl)dibenzene (5a): (*E*)-1-(4-Fluorophenyl)-2-phenylbut-1-en-1-yl 4-methyl benzenesulfonate (**2e**, 1 mmol, 204 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were

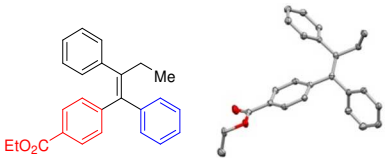
employed. Compound **5a** was obtained as a white solid (144 mg, 93%); mp 74–75 °C; HPLC (t_M = 8.41 min, t_m = 8.73 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 3057, 2971, 2928, 2870, 1894, 1599, 1503, 1441; ^1H NMR (400 MHz, $CDCl_3$) δ 7.36–7.29 (m, 2H), 7.28–7.20 (m, 3H), 7.19–7.05 (m, 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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; ^{19}F NMR (376 MHz, $CDCl_3$) δ –116.6; HRMS was not collected because compound **5a** does not ionize under both positive and negative modes.



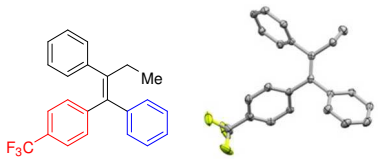
(*Z*)-(1-(*p*-Tolyl)but-1-ene-1,2-diyl)dibenzene (5b): (*E*)-2-Phenyl-1-(*p*-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (**2b**, 1 mmol, 330 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were employed. Compound **5b** was obtained as a white solid (280 mg, 94%); mp 77–78 °C; FTIR (neat, 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, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.8, 142.4, 141.7, 140.0, 138.7, 135.2, 130.6, 129.7, 129.5, 128.1, 127.8, 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.



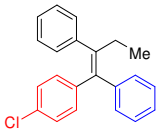
(*Z*)-(1-(4-Methoxyphenyl)but-1-ene-1,2-diyl)dibenzene (5c): (*E*)-1-(4-Methoxy phenyl)-2-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (**2c**, 1 mmol, 340 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were employed. Compound **5c** was obtained as a beige solid (240 mg, 92%); mp 114–116 °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, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.5, 143.9, 142.5, 141.3, 138.3, 135.5, 131.9, 129.7, 129.5, 128.1, 127.9, 126.5, 126.0, 112.8, 55.0, 29.1, 13.6; HRMS (ESI+) *m/z* calculated for $C_{23}H_{23}O$ ($[M+H]^+$), 315.1749; found, 315.1703.



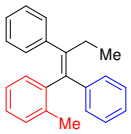
Ethyl (*Z*)-4-(1,2-diphenylbut-1-en-1-yl)benzoate (5d): Ethyl (*E*)-4-(2-phenyl-1-(tosyloxy)but-1-en-1-yl)benzoate (**2g**, 1 mmol, 451 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were employed. Compound **5d** was obtained as a yellow solid (300 mg, 84%); mp 88–92 °C; HPLC (t_M = 9.66 min, t_m = 9.41 min) indicated 96:4 isomer ratio; FTIR (neat, cm^{-1}) 3053, 2976, 2933, 1711, 1605, 1439, 1365; ^1H NMR (400 MHz, $CDCl_3$) 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), 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); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.6, 148.0, 143.9, 142.8, 141.7, 138.1, 130.7, 129.6, 129.5, 128.7, 128.3, 128.0, 126.9, 126.6, 60.7, 29.1, 24.9, 14.3, 13.5; HRMS (ESI–) *m/z* calculated for $C_{24}H_{24}O_3S$ ($[M–H]^-$), 355.1698; found, 355.1728.



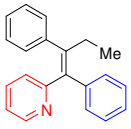
(*Z*)-(1-(4-(Trifluoromethyl)phenyl)but-1-ene-1,2-diyl)dibenzene (5e): (*E*)-2-Phenyl-1-(4-(trifluoromethyl)phenyl) but-1-en-1-yl 4-methylbenzenesulfonate (**2f**, 1 mmol, 447 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were employed. Compound **5e** was obtained as a white solid (339 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; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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; ^{19}F NMR (376 MHz, $CDCl_3$) 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-diyl)dibenzene (5f): (*E*)-1-(4-Chlorophenyl)-2-phenylbut-1-en-1-yl 4-methyl benzenesulfonate (**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; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.1, 143.0, 141.9, 141.5, 137.6, 132.1, 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.

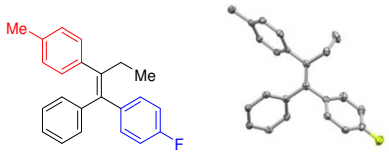


(*Z*)-(1-(*o*-Tolyl)but-1-ene-1,2-diyl)dibenzene (5g): (*E*)-2-Phenyl-1-(*o*-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (**2i**, 1 mmol, 352 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were employed. Compound **5g** was obtained as a white solid (263 mg, 98%); mp 69–71 °C; HPLC (t_M = 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; ^{1}H NMR (400 MHz, $CDCl_3$) 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, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) 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.

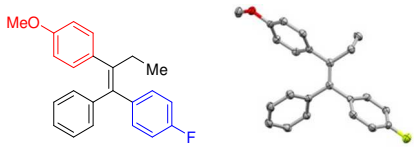


(*Z*)-2-(1,2-Diphenylbut-1-en-1-yl)pyridine (5h): (*E*)-2-Phenyl-1-(pyridin-2-yl)but-1-en-1-yl 4-methylbenzene sulfonate (**2j**, 1 mmol, 380 mg) and phenylboronic acid pinacol ester (1.1 equiv, 231 mg) were employed. Palladium(II) acetate and 2-dicyclohexylphosphino-2',6'-diisopropoxy-1,1'-biphenyl (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; ^{1}H NMR indicated 96:4 isomer ratio; FTIR (neat, cm^{-1}) 3053, 2926, 2868, 1584, 1561, 1465, 1440, 1425; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (ddd, *J* = 4.8, 2.0, 1.0 Hz, 1H), 7.40–7.23 (m, 6H), 7.18–7.05 (m, 5H), 6.91–6.80 (m, 2H), 2.54 (q, *J* = 7.4 Hz, 2H), 0.96 (t, *J*

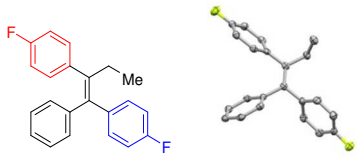
= 7.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.5, 149.0, 144.5, 141.9, 141.7, 139.1, 135.2, 129.4, 129.4, 128.2, 127.8, 126.8, 126.4, 125.9, 120.6, 28.5, 13.4; HRMS (ESI–) *m/z* calculated for $C_{21}H_{19}N$ ([M–H]$^-$), 284.1439; found, 284.1462.



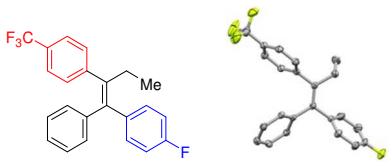
(*E*)-1-Fluoro-4-(1-phenyl-2-(*p*-tolyl)but-1-en-1-yl)benzene (5i): (*E*)-1-Phenyl-2-(*p*-tolyl)but-1-en-1-yl 4-methyl benzenesulfonate (**2k**, 1 mmol, 393 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 224 mg) were employed. Compound **5i** was obtained as a white solid (313 mg, 99%); mp 130–132 °C; HPLC (t_M = 10.58 min, t_m = 11.03 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 2970, 2929, 2872, 1739, 1601, 1508, 1457, 1443; ^{1}H NMR (400 MHz, $CDCl_3$) 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), 0.92 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.7 (d, *J* = 246.4 Hz), 142.9, 139.7 (d, *J* = 3.0 Hz), 138.9, 137.5, 135.8, 131.1 (d, *J* = 8.1 Hz), 130.8, 129.5, 128.6, 127.5, 125.8, 115.1 (d, *J* = 20.2 Hz), 29.0, 24.9, 21.2, 13.6; ^{19}F NMR (376 MHz, $CDCl_3$) δ –115.9; HRMS was not collected because compound **5i** does not ionize under both positive and negative modes.



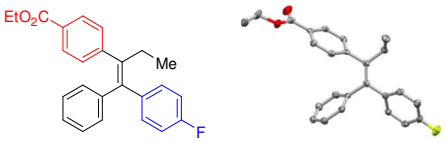
(*E*)-1-Fluoro-4-(2-(4-methoxyphenyl)-1-phenylbut-1-en-1-yl)benzene (5j): (*E*)-2-(4-Methoxyphenyl)-1-phenyl but-1-en-1-yl 4-methylbenzenesulfonate (**2l**, 1 mmol, 409 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 224 mg) were employed. Compound **5j** was obtained as a white solid (326 mg, 98%); mp 118–119 °C; HPLC (t_M = 7.82 min, t_m = 8.10 min) indicated 97:3 isomer ratio; FTIR (neat, cm^{-1}) 2968, 2931, 1741, 1606, 1508, 1462, 1442; ^{1}H NMR (400 MHz, $CDCl_3$) δ 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, *J* = 7.3 Hz, 2H), 1.33 (s, 1H), 0.93 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.7 (d, *J* = 246.4 Hz), 158.0, 143.2, 142.1, 139.7, 137.3, 134.1, 131.1 (d, *J* = 8.1 Hz), 130.8, 130.7, 127.5, 125.8, 115.1 (d, *J* = 21.2 Hz), 113.3, 55.1, 28.9, 24.9, 13.6; ^{19}F NMR (376 MHz, $CDCl_3$) δ –116.0; HRMS (ESI+) *m/z* calculated for $C_{23}H_{21}FO$ ([M + H]$^+$), 333.1655; found, 333.1682.



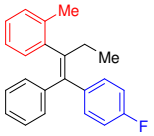
(*E*)-4,4'-(1-Phenylbut-1-ene-1,2-diyl)bis(fluorobenzene) (5k): (*E*)-2-(4-Fluorophenyl)-1-phenylbut-1-en-1-yl 4-methylbenzenesulfonate (**2n**, 1 mmol, 397 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 224 mg) were employed. Compound **5k** was obtained as a white solid (275 mg, 86%); mp 91–93 °C; HPLC (t_M = 8.40 min, t_m = 8.58 min) indicated 98:2 isomer ratio; FTIR (neat, cm^{-1}) 2970, 2932, 2873, 1739, 1602, 1507; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24–7.15 (m, 2H), 7.10–6.94 (m, 7H), 6.91–6.78 (m, 4H), 2.45 (q, *J* = 7.4 Hz, 2H), 0.93 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.7 (d, *J* = 246.2 Hz), 161.4 (d, *J* = 246.4 Hz), 142.7, 141.5, 139.3, 138.3, 137.8, 131.1 (d, *J* = 7.1 Hz), 131.0 (d, *J* = 8.1 Hz), 130.7, 127.6, 126.0, 115.1 (d, *J* = 21.1 Hz), 114.9 (d, *J* = 21.1 Hz), 29.0, 13.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ –115.7, –116.1; HRMS (ESI–) *m/z* calculated for $C_{21}H_{19}N$ ([2M+MeCN]$^-$), 681.3019; found, 681.2966.



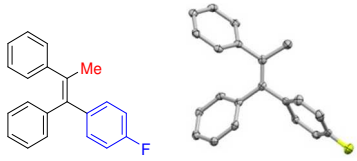
(*E*)-1-Fluoro-4-(1-phenyl-2-(4-(trifluoromethyl)phenyl)but-1-en-1-yl)benzene (5l): (*E*)-1-Phenyl-2-(4-(trifluoro methyl)phenyl)but-1-en-1-yl 4-methylbenzene sulfonate (**2o**, 1 mmol, 447 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 224 mg) were employed. Compound **5l** was obtained as a white solid (346 mg, 93%); mp 126–127 °C; ^{1}H NMR indicated >99:1 isomer ratio (HPLC does not resolve the isomers); FTIR (neat, cm^{-1}) 2971, 1739, 1615, 1602, 1506, 1325; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.2 Hz, 2H), 7.27–7.16 (m, 4H), 7.09–6.95 (m, 5H), 6.89–6.81 (m, 2H), 2.50 (q, *J* = 7.5 Hz, 2H), 0.92 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.9 (d, *J* = 247.5 Hz), 146.1, 142.2, 141.3, 139.4, 138.9 (d, *J* = 3.0 Hz), 131.0 (d, *J* = 8.1 Hz), 130.7, 130.0, 128.4 (q, *J* = 32.3 Hz), 127.7, 126.4, 124.9 (q, *J* = 4.0 Hz), 124.3 (q, *J* = 272.7 Hz), 115.2 (d, *J* = 21.1 Hz), 28.9, 13.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ –62.4, –115.3; HRMS (ESI–) *m/z* calculated for $C_{21}H_{19}N$ ([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 (t_M = 9.34 min, t_m = 9.21 min) indicated >99:1 isomer ratio; FTIR (neat, cm^{-1}) 2971, 2930, 2873, 1715, 1603, 1506, 1366; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.4 Hz, 2H), 7.26–7.13 (m, 4H), 7.09–6.96 (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); ^{13}C NMR (101 MHz, $CDCl_3$) 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; ^{19}F NMR (376 MHz, $CDCl_3$) δ –115.4; HRMS (ESI–) *m/z* calculated for $C_{21}H_{19}N$ ([M–H]$^-$), 373.1604; found, 373.1557.

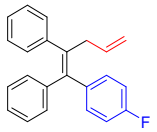


(*E*)-1-(1-(4-Fluorophenyl)-1-phenylbut-1-en-2-yl)-2-methylbenzene (5n): (*E*)-1-Phenyl-2-(*o*-tolyl)but-1-en-1-yl 4-methylbenzenesulfonate (**2r**, 0.67 mmol, 262 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 163 mg) were employed. Amounts of other reagents and solvents were scaled accordingly. Compound **5n** was obtained as a white solid (206 mg, 98%); mp 87–88 °C; HPLC (t_M = 9.28 min, t_m = 9.53 min) indicated 97:3 isomer ratio; FTIR (neat, cm^{-1}) 3054, 3017, 2969, 2930, 2872, 1739, 1601, 1505, 1442, 1375; ^{1}H NMR (400 MHz, $CDCl_3$) 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), 2.39 (q, *J* = 7.5 Hz, 2H), 2.09 (s, 3H), 0.92 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) 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; ^{19}F NMR (376 MHz, $CDCl_3$) δ –115.9; HRMS was not collected because compound **5n** does not ionize under both positive and negative modes.

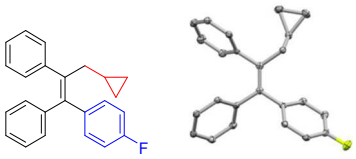


(*E*)-(1-(4-Fluorophenyl)prop-1-ene-1,2-diyl)dibenzene (5o): (*E*)-1,2-Diphenyl prop-1-en-1-yl 4-methylbenzene sulfonate (**2t**, 0.64 mmol, 232 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 156 mg) were employed. Amounts of other reagents and solvents were scaled accordingly. Compound **5o** was obtained as a white solid (165 mg, 90%); mp 106–107 °C; HPLC (t_M = 7.19 min, t_m = 7.90 min) indicated 97:3 isomer ratio; FTIR (neat, cm^{-1}) 3054, 2913, 2855, 1740, 1599, 1506, 1442, 1375; ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (101 MHz, $CDCl_3$) 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,

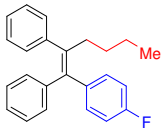
126.0, 115.1 (d, J = 22.2 Hz), 23.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ –115.7; HRMS was not collected because compound **5o** does not ionize under both positive and negative modes.



(*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.

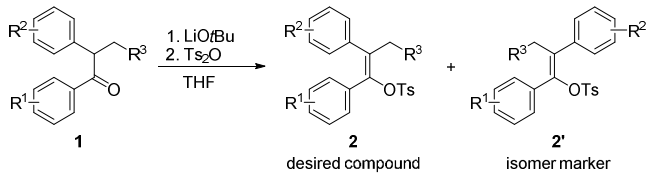


(*E*)-(3-Cyclopropyl-1-(4-fluorophenyl)prop-1-ene-1,2-diyl)dibenzene (5q): (*E*)-3-Cyclopropyl-1,2-diphenylprop-1-en-1-yl 4-methylbenzenesulfonate (**2w**, 1 mmol, 405 mg) and 2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.1 equiv, 224 mg) were employed. Compound **5q** was obtained as 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.

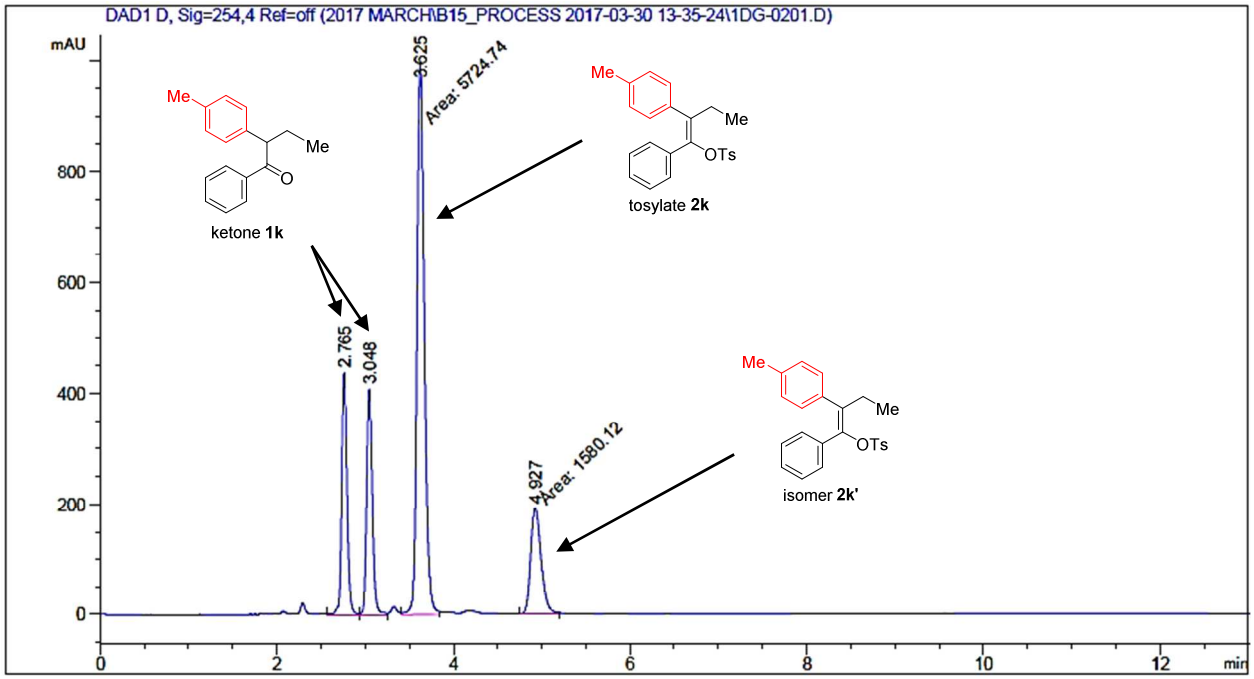
Tosylate isomer marker synthesis (for HPLC)



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.

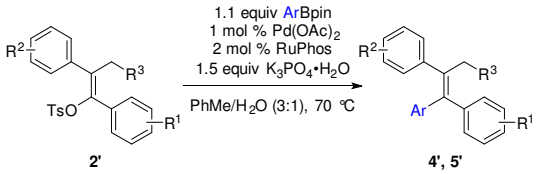
To a septum-top 10 mL vial equipped with a stir bar was added ketone **1** (1 mmol). The vial was vacuumed and back-filled with nitrogen (3×), then anhydrous THF (1 mL) was added via syringe, followed by dropwise addition of LiO*t*-Bu (1.4 equiv, 1 M in THF, 1.4 mL). After stirring for 30 min, a solution of Ts_2O (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'**.

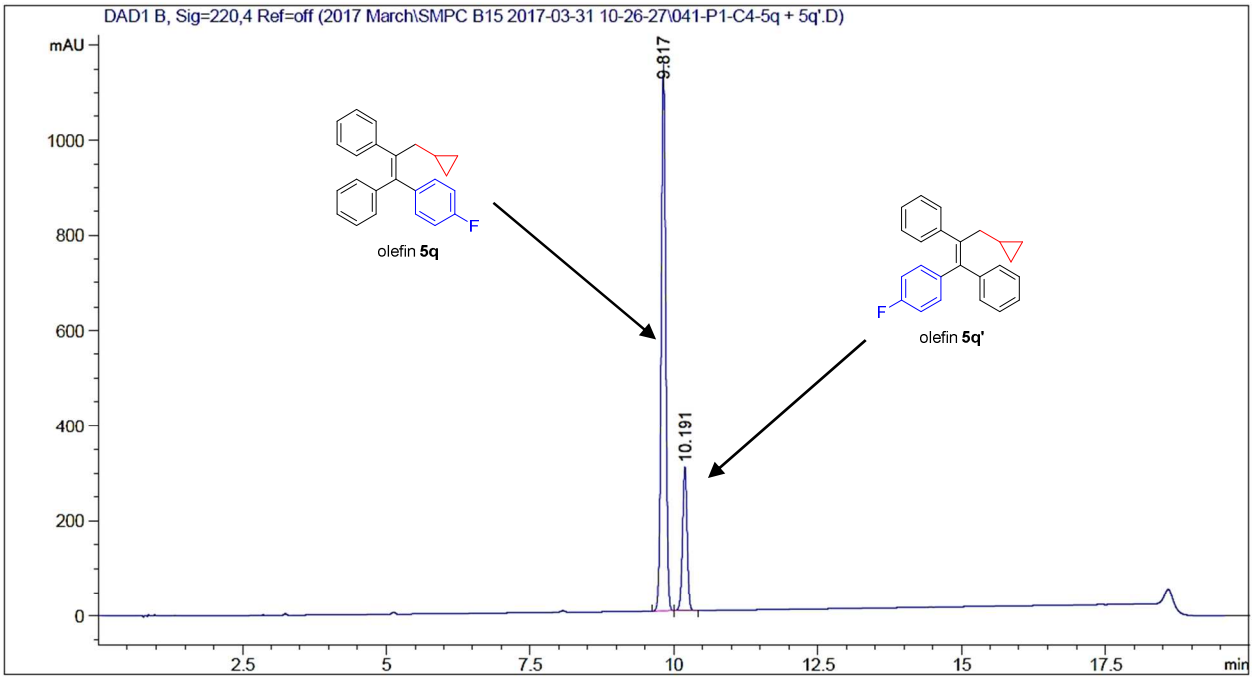


Olefin isomer marker synthesis (for HPLC)

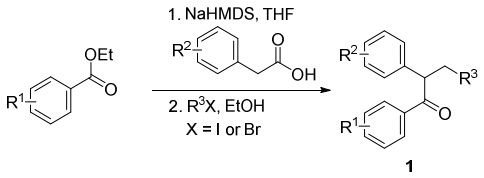
Except for **4a–g** and **5a–g** which were synthesized as a pair of isomers by judicious choice of coupling partners, all other olefin isomers were synthesized by employing the same general Suzuki procedure described in previous sections using the corresponding tosylate isomers. These olefin isomers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



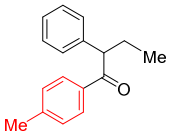
Analytical HPLC analyses were performed with an Agilent 1260 Infinity Series HPLC instrument; the column used was ACE Excel 3 C18 HL, 3x50mm; particle size 3 um; injection volume 2 µL; temperature 35 °C; flow rate 1 mL/min; mobile phase A = 0.05% trifluoroacetic acid in H_2O, mobile phase B = 0.05% trifluoroacetic 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.



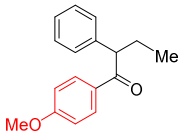
Ketone Synthesis



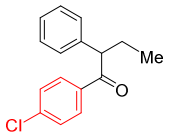
Ketones were synthesized according to a literature procedure.[2] To a stirring solution of NaHMDS (2.5 equiv, 1.0 M in THF) at 23 °C was slowly added arylacetic acid (1 equiv, 1.5 M in THF) over 1 h. The resulting mixture was stirred for 1 h, and benzoate (0.77–0.95 equiv, 1.0 M in THF) was added dropwise. 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 H_2O (20 mL/g) and filtered through Celite. The filtrate was extracted with DCM (3×15 mL/g). The combined organic layers were washed with H_2O (3×15 mL/g), dried over Na_2SO_4 (as needed), filtered, and concentrated *in vacuo*. The crude residue was purified by silica gel column chromatography, then by preparative HPLC if necessary.



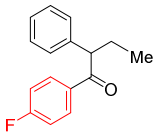
2-Phenyl-1-(*p*-tolyl)butan-1-one (1b): Ethyl 4-methylbenzoate (0.95 equiv, 69.8 mmol, 11.4 g), 2-phenylacetic acid (1.0 equiv, 73.5 mmol, 10.0 g), and ethyl iodide (2.0 equiv, 147 mmol, 22.9 g) were employed. After extraction, the crude residue was purified via preparative HPLC. Ketone **1b** was obtained as a light yellow oil (6.00 g, 34%). ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (400 MHz, $CDCl_3$) 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.



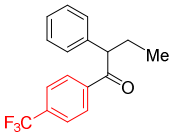
1-(4-Methoxyphenyl)-2-phenylbutan-1-one (1c): Methyl-4-methoxylbenzoate (0.80 equiv, 61.0 mmol, 10.1 g), 2-phenylacetic acid (1.0 equiv, 76.0 mmol, 10.3 g), and ethyl iodide (1.9 equiv, 144 mmol, 22.5 g) were employed. After extraction, the crude residue was purified via silica gel column chromatography 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%). ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (400 MHz, $CDCl_3$) δ 198.6, 163.3, 140.1, 130.9, 130.1, 128.8, 128.2, 126.9, 113.7, 55.4, 55.1, 27.2, 12.4.



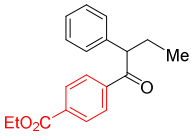
1-(4-Chlorophenyl)-2-phenylbutan-1-one (1d): Methyl 4-chlorobenzoate (0.77 equiv, 87.9 mmol, 15.0 g), 2-phenylacetic acid (1.0 equiv, 114 mmol, 15.6 g), and ethyl iodide (1.9 equiv, 214 mmol, 33.3 g) were employed. After extraction, the crude residue was purified via preparatory HPLC. Ketone **1d** was obtained as a light yellow oil (13.6 g, 60%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, 2H, *J* = 8.8 Hz), 7.14 (m, 6H), 7.04 (m, 1H), 4.2 3 (t, 1H, *J* = 7.0 Hz), 2.05 (m, 1H), 1.71 (m, 1H), 0.75 (t, 3H, *J* = 7.4 Hz); ^{13}C NMR (400 MHz, $CDCl_3$) 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.



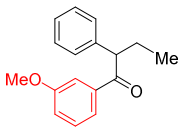
1-(4-Fluorophenyl)-2-phenylbutan-1-one (1e): Ethyl 4-fluorobenzoate (0.77 equiv, 179 mmol, 30.0 g), 2-phenylacetic acid (1.0 equiv, 233 mmol, 32.0 g), and ethyl iodide (2.5 equiv, 448 mmol, 69.0 g) were employed. After extraction, the crude residue was purified via silica gel column chromatography using 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, $CDCl_3$) δ 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, $CDCl_3$) δ 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.



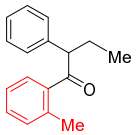
2-Phenyl-1-(4-(trifluoromethyl)phenyl)butan-1-one (1f): Methyl-4-(trifluoromethyl)benzoate (0.95 equiv, 69.8 mmol, 14.2 g), 2-phenylacetic acid (1.0 equiv, 73.5 mmol, 10.0 g), and ethyl iodide (2.0 equiv, 147 mmol, 22.9 g) were employed. After extraction, the crude residue was purified by preparatory HPLC. Ketone **1f** was obtained as a light yellow oil (7.92 g, 37%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, *J* = 8.0Hz, 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, $CDCl_3$) δ 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.



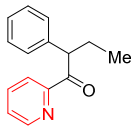
Ethyl 4-(2-phenylbutanoyl)benzoate (1g): Diethyl terephthalate (0.95 equiv, 69.8 mmol, 19.6 g), 2-phenylacetic acid (1.0 equiv, 73.5 mmol, 10.0 g), and ethyl iodide (2.4 equiv, 176 mmol, 27.5 g) were employed. After extraction, the crude residue was purified by preparatory HPLC. Ketone **1g** was obtained as a light yellow solid (7.20 g, 33%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, 2H, *J* = 8.3Hz), 8.01 (d, 2H, *J* = 8.5 Hz), 7.32 (m, 4H), 7.22 (m, 1H), 4.41 (m, 3H), 2.23 (m, 1H), 1.89 (m, 1H), 1.40 (t, 3H, *J* = 7.2Hz), 0.94 (t, 3H, *J* = 7.3 Hz); ^{13}C NMR (400 MHz, $CDCl_3$) δ 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.



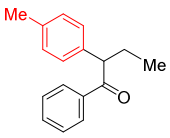
1-(3-Methoxyphenyl)-2-phenylbutan-1-one (1h): Methyl-3-methoxylbenzoate (0.80 equiv, 59.0 mmol, 9.8 g), 2-phenylacetic acid (1.0 equiv, 73.5 mmol, 10.0 g), and ethyl iodide (1.9 equiv, 140 mmol, 21.8 g) were employed. After extraction, the crude residue was purified via silica gel column chromatography 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, $CDCl_3$) δ 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, $CDCl_3$) δ 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.



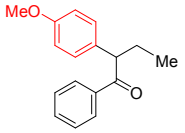
2-Phenyl-1-(*o*-tolyl)butan-1-one (1i): Methyl 2-methylbenzoate (0.77 equiv, 240 mmol, 36.0 g), 2-phenylacetic acid (1.0 equiv, 312 mmol, 42.5 g), and ethyl iodide (2.1 equiv, 580 mmol, 89.9 g) were employed. After extraction, the crude product was dissolved in THF (100 mL) and aq LiOH (13 g monohydrate 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%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 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); ^{13}C NMR (400 MHz, $CDCl_3$) 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.



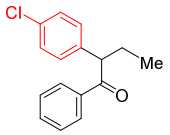
2-Phenyl-1-(pyridine-2-yl)butan-1-one (1j): Methyl picolinate (0.80 equiv, 137 mmol, 11.6 g), 2-phenylacetic acid (1.0 equiv, 110 mmol, 15.0 g), and ethyl iodide (2.4 equiv, 201 mmol, 31.5 g) were employed. After extraction, the crude residue was purified by preparatory HPLC. Ketone **1j** was obtained as a light yellow oil (6.20 g, 32%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.70 (d, 1H, J = 4.3 Hz), 8.02 (d, 1H, *J* = 8.1 Hz), 7.77(td, 1H, *J* = 7.7, 1.4Hz), 7.41 (m, 3H), 7.28 (t, 2H, *J* = 7.5 Hz), 7.19 (m, 1H), 5.29 (t, 1H, *J* = 7.5Hz), 2.22 (m, 1H), 1.95 (m, 1H), 0.94 (t, 1H, *J* = 7.4Hz); ^{13}C NMR (400 MHz, $CDCl_3$) δ 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.



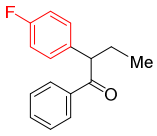
1-Phenyl-2-(*p*-tolyl) butan-1-one (1k): Methyl benzoate (0.83 equiv, 100 mmol, 13.6 g), 2-*p*-tolylacetic acid (1.0 equiv, 120 mmol, 18.0 g), and ethyl iodide (2.1 equiv, 250 mmol, 39.0 g) were employed. After extraction, the crude residue was purified via silica gel column chromatography using eluent conditions 100% petroleum ether. Ketone **1k** was obtained as a white solid (10.7 g, 45%). ^{1}H NMR (400 MHz, $CDCl_3$) 7.99–7.92 (m, 2H), 7.45–7.29 (m, 3H), 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); δ ^{13}C NMR (400 MHz, $CDCl_3$) δ 200.2, 137.2, 136.7, 136.6, 132.7, 129.6, 128.7, 128.5, 128.2, 55.1, 27.2, 21.0, 12.4.



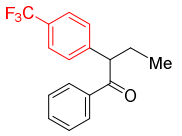
2-(4-Methoxyphenyl)-1-phenylbutan-1-one (1l): Methyl benzoate (0.83 equiv, 150 mmol, 20.4 g), 2-(4-methoxyphenyl)acetic acid (1.0 equiv, 180 mmol, 29.9 g), and ethyl iodide (2.1 equiv, 375 mmol, 58.5 g) were employed. After reaction completion, the reaction was quenched with H_2O (200 mL), then concentrated *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%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00–7.92 (m, 2H), 7.46–7.35 (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); ^{13}C NMR (400 MHz, $CDCl_3$) 200.3, 158.6, 137.1, 132.7, 131.7, 129.3, 128.6, 128.5, 114.3, 55.1, 54.6, 27.1, 12.3.



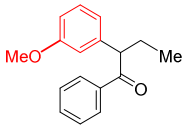
2-(4-Chlorophenyl)-1-phenylbutan-1-one (1m): Ethyl benzoate (0.77 equiv, 45 mmol, 6.80 g), 2-(4-chlorophenyl)acetic acid (1.0 equiv, 59 mmol, 10.0 g), and ethyl iodide (2.1 equiv, 109 mmol, 16.9 g) were employed. The crude residue was purified via silica gel column chromatography, eluent conditions 0 – 5% *i*PrOAc in heptanes. Ketone **1m** was obtained as a yellow oil (6.6 g, 63%). ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (400 MHz, $CDCl_3$) 199.7, 138.2, 136.8, 133.0, 132.9, 129.7, 129.0, 128.6 (2C), 54.6, 27.2, 12.3.



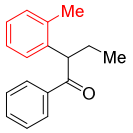
2-(4-Fluorophenyl)-1-phenylbutan-1-one (1n): Methyl benzoate (0.77 equiv, 184 mmol, 25.0 g), *p*-fluorophenylacetic acid (1.0 equiv, 239 mmol, 36.6 g), and ethyl iodide (2.1 equiv, 460 mmol, 71.7 g) were employed. After extraction, the crude product was dissolved in THF (100 mL) and aq LiOH (13 g monohydrate in 80 mL H_2O), 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%). ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (400 MHz, $CDCl_3$) 200.1, 136.9, 135.3, 132.9, 129.8, 128.6, 128.6, 115.8, 115.6, 54.5, 27.2, 12.2.



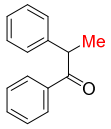
1-Phenyl-2(4-(trifluoromethyl)phenyl)butan-1-one (1o): Methyl benzoate (1.0 equiv, 196 mmol, 26.7 g), 2-(4-(trifluoromethyl)phenyl)acetic acid (1.0 equiv, 196 mmol, 40.0 g), and ethyl iodide (2.5 equiv, 490 mmol, 76.0 g) were employed. After extraction, the crude residue was purified via silica gel column chromatography using eluent conditions 100% petroleum ether. Ketone **1o** was obtained as a yellow oil (6.5 g, 11%). ^{1}H NMR (400 MHz, $CDCl_3$) 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, 1H), 2.26 (m, 1H), 0.93 (m, 3 H); ^{13}C NMR (400 MHz, $CDCl_3$) 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.



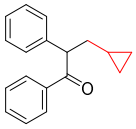
2-(3-Methoxyphenyl)-1-phenylbutan-1-one (1q): Methyl benzoate (0.83 equiv, 147 mmol, 20.0 g), 2-(3-methoxyphenyl)acetic acid (1.0 equiv, 180 mmol, 30.0 g), and ethyl iodide (2.1 equiv, 375 mmol, 58.5 g) were employed. After reaction completion, the reaction was quenched with H_2O (100 mL), then concentrated *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%). ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (400 MHz, $CDCl_3$) 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.



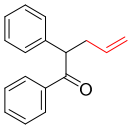
1-Phenyl-2-(*o*-tolyl)butan-1-one (1r): Methyl benzoate (0.77 equiv, 147 mmol, 20.0 g), 2-*o*-tolylacetic acid (1.0 equiv, 191 mmol, 28.7 g), and ethyl iodide (2.1 equiv, 353 mmol, 55.1 g) were employed. After extraction, the crude product was dissolved in THF (100 mL) and aq LiOH (13 g monohydrate in 80 mL H_2O), 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%). ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (400 MHz, $CDCl_3$) 200.5, 138.5, 137.5, 135.2, 132.7, 131.0, 128.5, 128.4, 127.3, 126.9, 126.7, 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 H_2O), 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%). ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (400 MHz, $CDCl_3$) 200.3, 141.5, 136.5, 132.8, 129.0, 128.8, 128.5, 127.8, 126.9, 47.9.

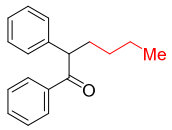


3-Cycolpropyl-1,2-diphenylpropan-1-one (1v): Methyl benzoate (0.77 equiv, 147 mmol, 20.0 g), 2-phenylacetic acid (1.0 equiv, 191 mmol, 26.0 g), and allyl bromide (2.4 equiv, 353 mmol, 42.7 g) were employed. After extraction, the crude product was dissolved in THF (80 mL) and aq LiOH (13 g monohydrate in 80 mL H_2O), 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%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (m, 2H), 7.48 (m, 1H), 7.38 (m, 2H), 7.29 (m, 4H), 7.2 (m, 1H), 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); ^{13}C NMR (400 MHz, $CDCl_3$) 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.

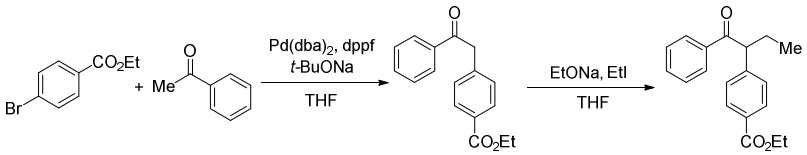


1,2-Diphenylpent-4-en-1-one (1w): Methyl benzoate (0.77 equiv, 147 mmol, 20.0 g), 2-phenylacetic acid (1.0 equiv, 191 mmol, 26.0 g), and bromomethyl cyclopropane (2.4 equiv, 353 mmol, 47.7 g) were employed. After extraction, the crude product was dissolved in THF (80 mL) and aq LiOH (13 g monohydrate in 80 mL H_2O), 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%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02–7.93 (m, 2H), 7.51–7.44 (m, 1H), 7.43–7.23 (m,

5H), 7.22–7.14 (m, 1H), 4.68 (t, J = 7.2 Hz, 1H), 2.06 (dt, J = 14.2, 7.2 Hz, 1H), 1.75 (dt, J = 13.9, 6.9 Hz, 1H), 0.71–0.54 (m, 1H), 0.46–0.30 (m, 2H), 0.14– −0.04 (m, 1H); ^{13}C NMR (400 MHz, $CDCl_3$) 200.2, 139.9, 137.0, 132.8, 128.8, 128.7, 128.5, 128.2, 126.9, 54.1, 39.2, 9.4, 4.7.

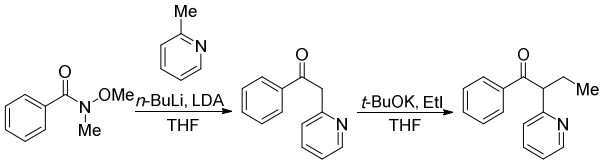


1,2-Diphenylhexan-1-one (1x): Methyl benzoate (1.0 equiv, 125 mmol, 17.0 g), 2-phenylacetic acid (1.0 equiv, 125 mmol, 17.0 g), and *n*-butyl iodide (2.5 equiv, 250 mmol, 46.0 g) were employed. After extraction, the crude product was dissolved in THF (80 mL) and aq LiOH (13 g monohydrate in 80 mL H_2O), 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%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 8.00 (m, 2H), 7.45–7.52 (m, 1H), 7.36–7.43 (m, 2H), 7.25–7.34 (m, 4H), 7.17–7.24 (m, 1H), 4.54 (t, J = 7.28 Hz, 1H), 2.12–2.26 (m, 1H), 1.77–1.90 (m, 1H), 1.56 (s, 1H), 1.14–1.42 (m, 4H), 0.87 (t, J = 7.15 Hz, 3H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 200.2, 139.9, 137.1, 132.8, 128.9, 128.7, 128.5, 128.2, 126.9, 53.7, 33.8, 30.0, 22.7, 14.0.



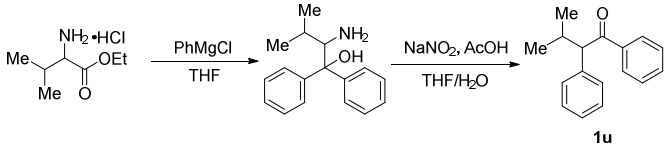
1p

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 jacked reactor was added ethyl 4-bromobenzoate (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 (3×), 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 offwhite 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_2O (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.



1s

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-Methylpridine (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 20mL 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 H_2O (100 mL). The biphasic mixture was concentrated to remove THFand 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 jacked reactor equipped with mechanical stirrer was added THF (400mL), 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 H_2O (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 reslurried in 10% EtOAc in petroleum ether to give the product ketone **1s** as a light yellow solid (7.2 g, 16 %). ^{1}H NMR (400 MHz, $CDCl_3$) 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); ^{13}C NMR (400 MHz, $CDCl_3$) 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.



1u

3-methyl-1,2-diphenylbutan-1-one (1u)[2]: 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 in 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 1h and quenched with MeOH (350 mL) and H_2O (250 mL). The reaction was filtered and washed with THF (1000 mL). The filtrate was concentrated to approximately 150 mL, then diluted with H_2O (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 H_2O (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 $NaNO_2$ (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 H_2O (2×200 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%). ^{1}H NMR (400 MHz, $CDCl_3$) 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, $CDCl_3$) δ 200.6, 138.6, 137.7, 132.8, 128.8, 128.7, 128.5 (2C), 127.0, 61.4, 31.9, 22.0, 20.5.

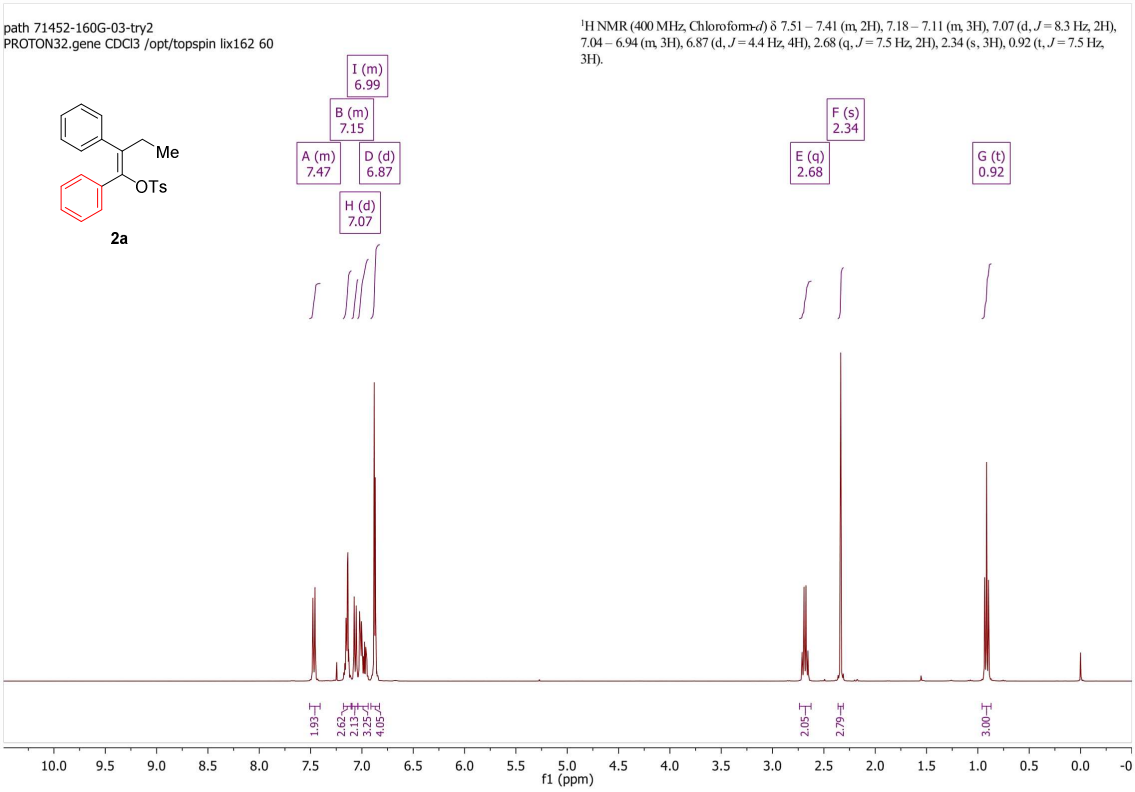
References

(1) Stock solution of aqueous K_3PO_4 for 20 reactions was prepared from dissolving K_3PO_4•H_2O (30 mmol, 7.28 g) in 10 mL H_2O 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_3PO_4•H_2O.

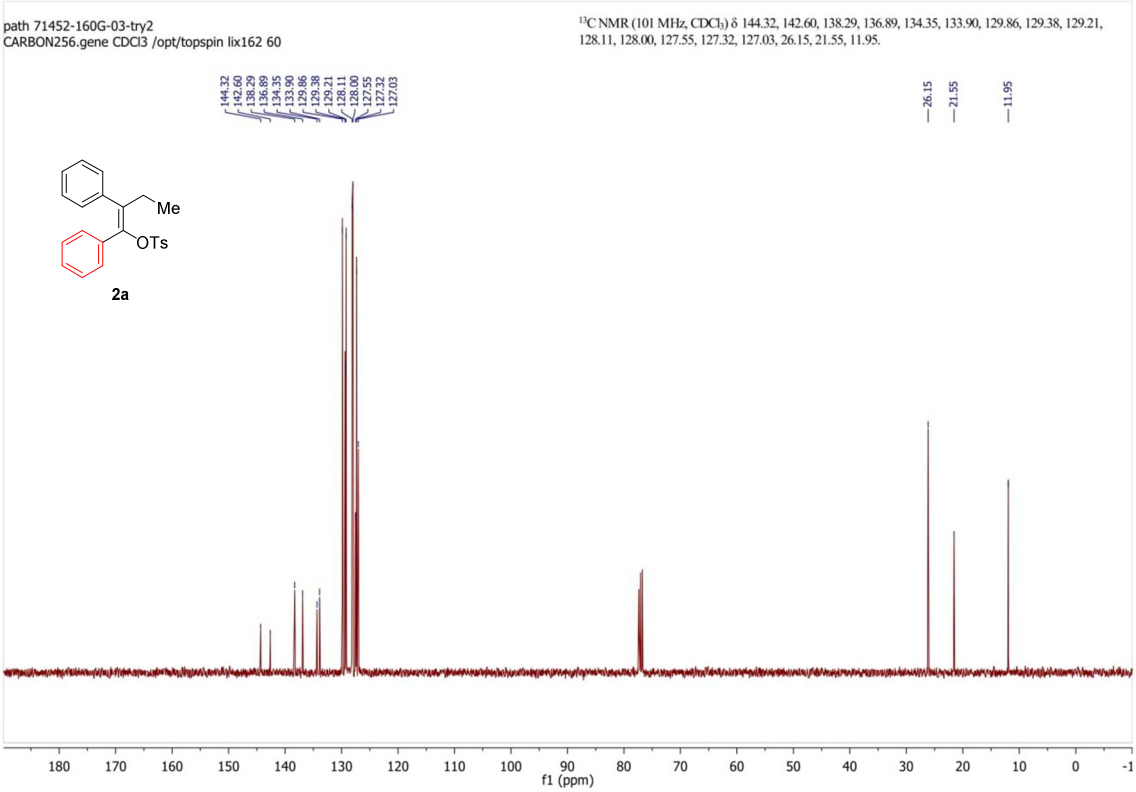
(2) Wu, G.; Yin, W.; Shen, H. C.; Huang, Y. *Green Chem.* **2012**, *41*, 580.

(3) Chung, J. Y. L.; Steinhuebel, D.; Krska, S. W.; Hartner, F. W.; Cai, C.; Rosen, J.; Mancheno, D. E.; Pei, T.; DiMichele, L.; Ball, R. G.; Chen, C.-y.; Tan, L.; Alorati, A. D.; Brewer, S. E.; Scott, J. P. *Org. Process Res. Dev.* **2012**, *16*, 1832.

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



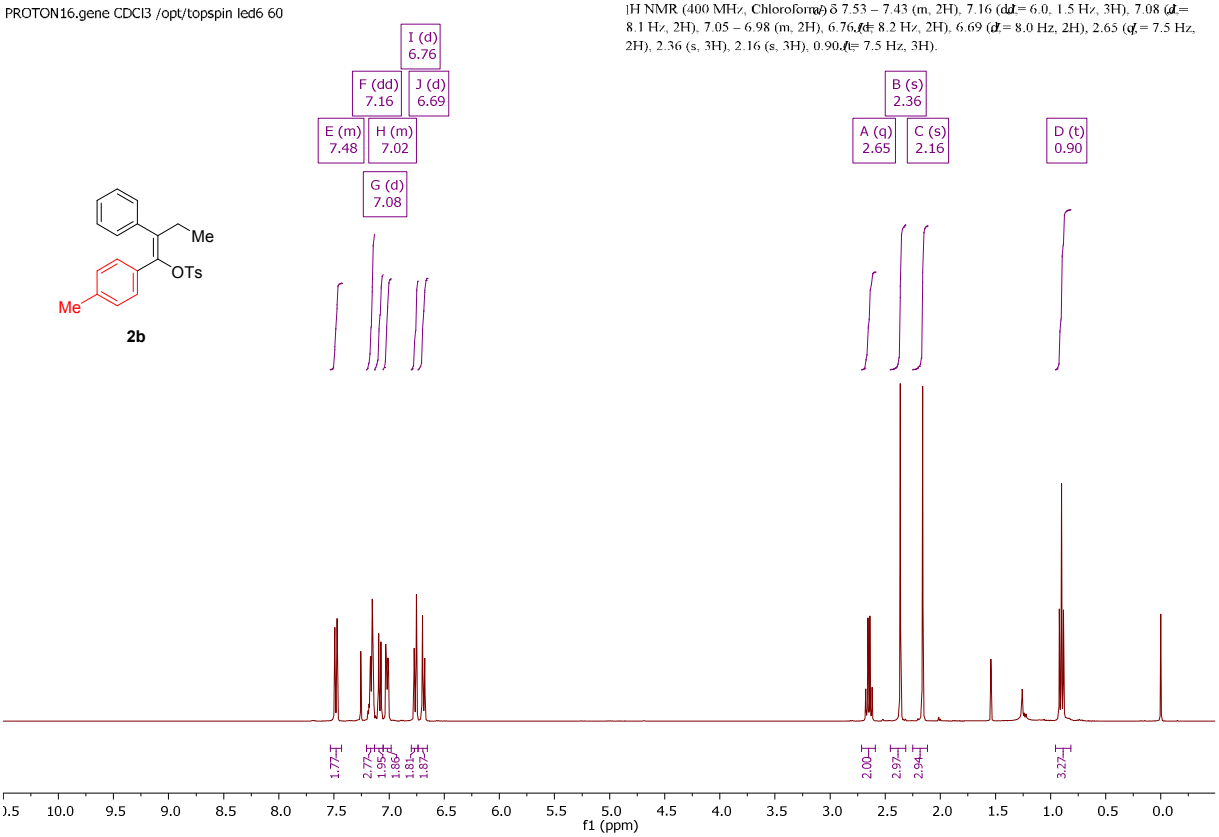
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.51 – 7.41 (m, 2H), 7.18 – 7.11 (m, 3H), 7.07 (d, *J* = 8.3 Hz, 2H), 7.04 – 6.94 (m, 3H), 6.87 (d, *J* = 4.4 Hz, 4H), 2.68 (q, *J* = 7.5 Hz, 2H), 2.34 (s, 3H), 0.92 (t, *J* = 7.5 Hz, 3H).



^{13}C NMR (101 MHz, $CDCl_3$) δ 144.32, 142.60, 138.29, 136.89, 134.35, 133.90, 129.86, 129.38, 129.21, 128.11, 128.00, 127.55, 127.32, 127.03, 26.15, 21.55, 11.95.

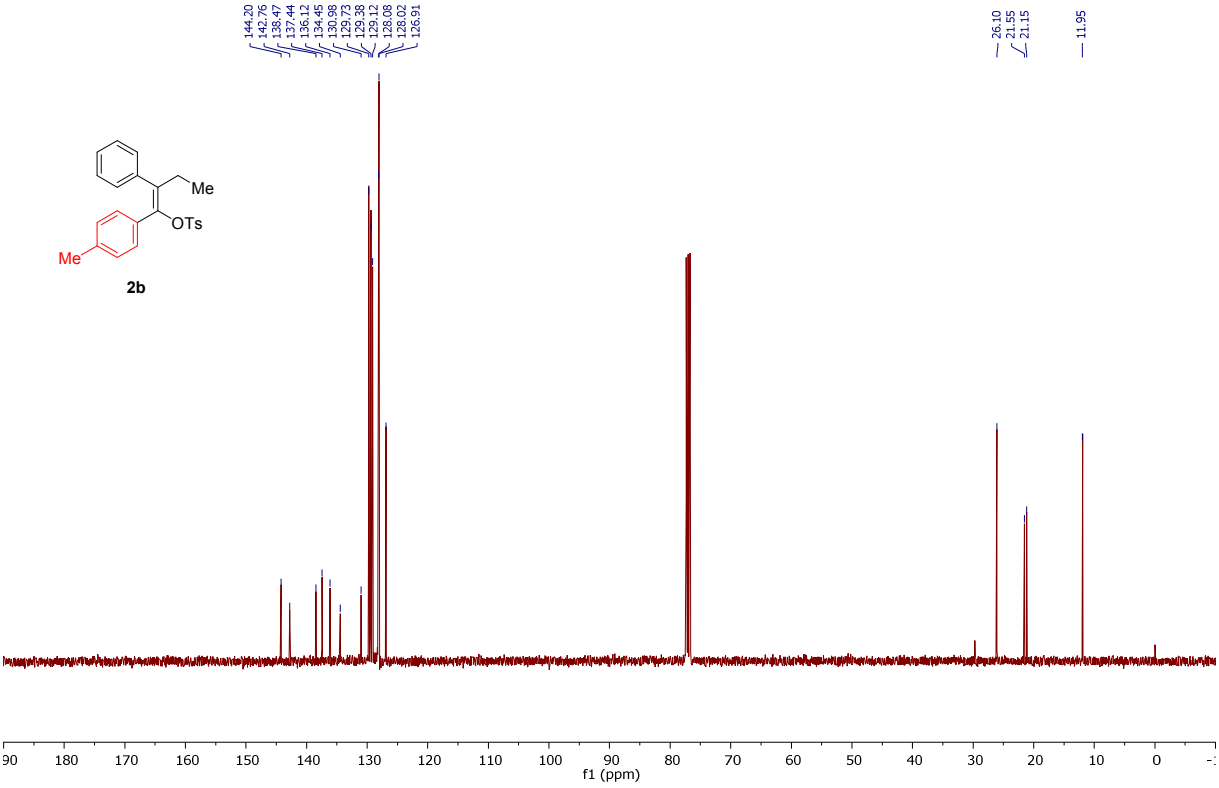
PROTON16.gene CDCl3 /opt/topspin led6 60

^{1}H NMR (400 MHz, Chloroform-d) 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).



CARBON2K.gene CDCl3 /opt/topspin led6 60

^{13}C NMR (101 MHz, CDCl$_3$) δ 144.20, 142.76, 138.47, 137.44, 136.12, 134.45, 130.98, 129.73, 129.38, 129.12, 128.08, 128.02, 126.91, 26.10, 21.55, 21.15, 11.95.



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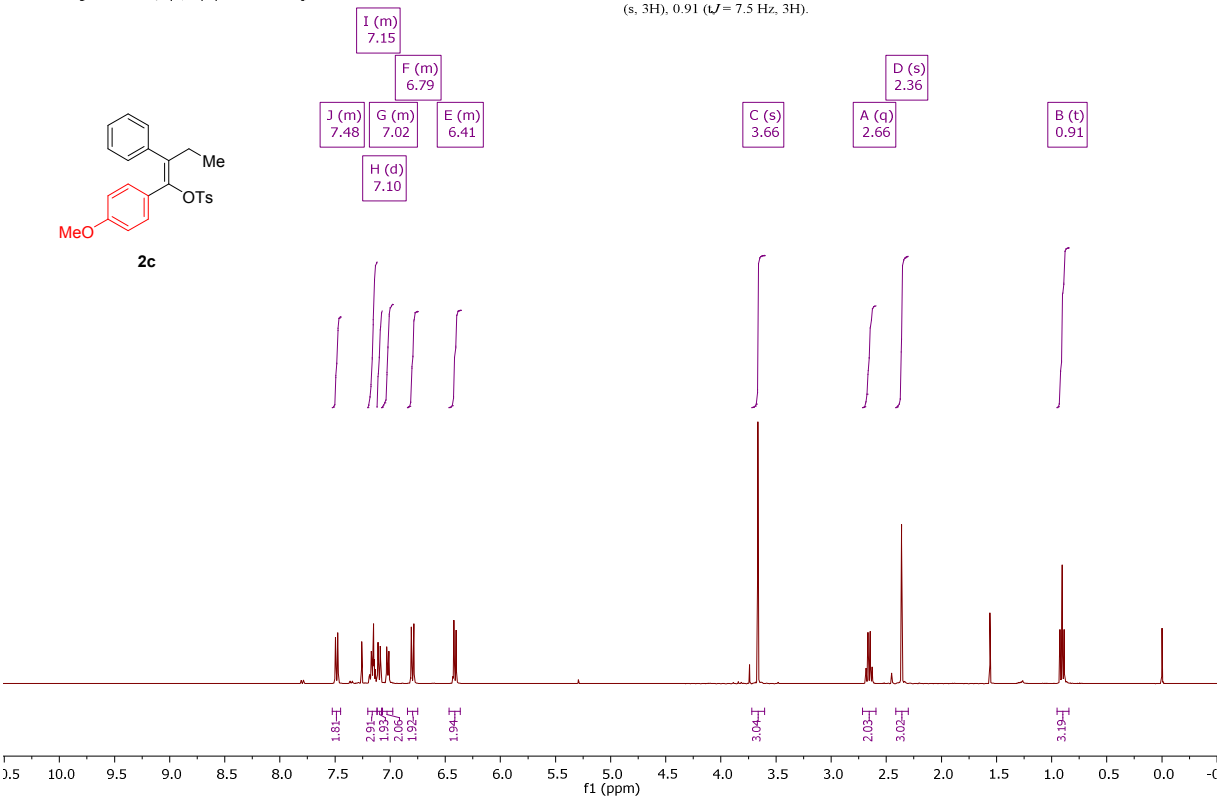
PROTON16.gene CDCl3 /opt/topspin3.2.5 zhangh34 82

^{1}H NMR (400 MHz, Chloroform-d) δ 7.52 – 7.45 (m, 2H), 7.20 – 7.12 (m, 3H), 7.10 (d, J = 8.2 Hz, 2H), 7.08 – 6.98 (m, 2H), 6.84 – 6.75 (m, 2H), 6.47 – 6.36 (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).



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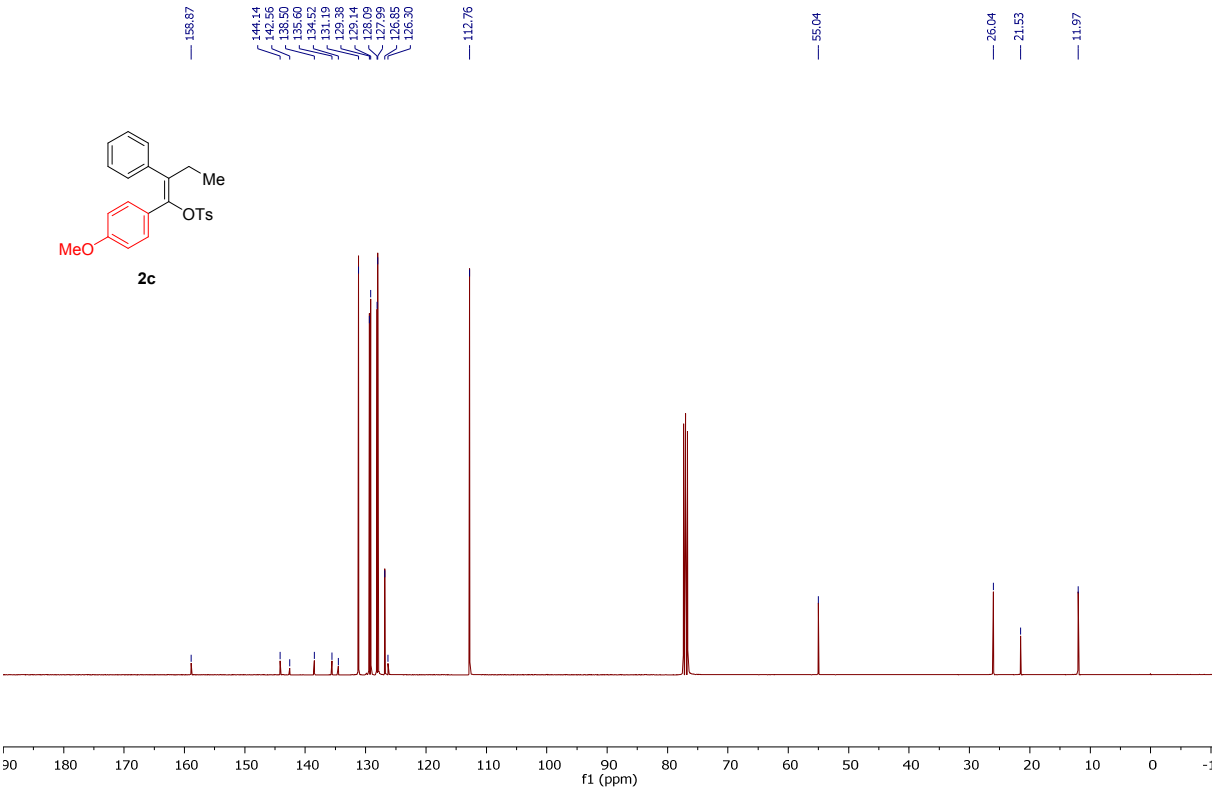
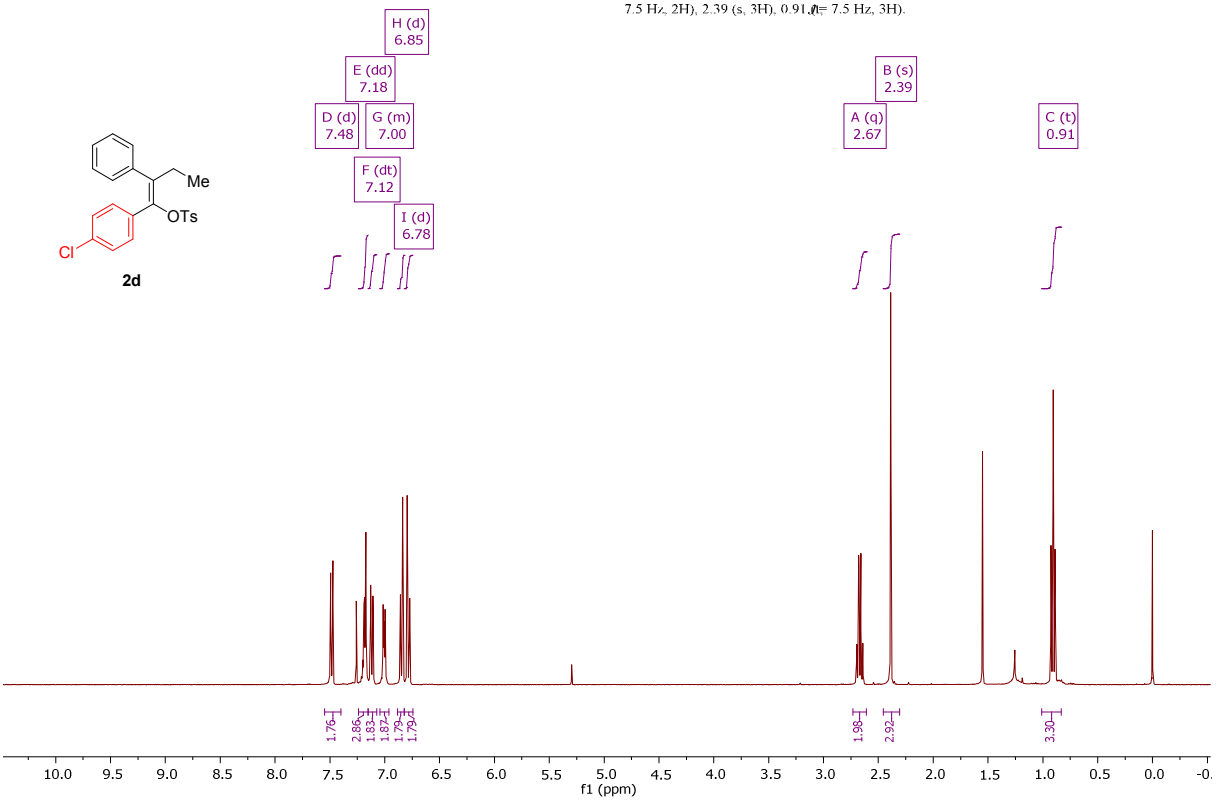
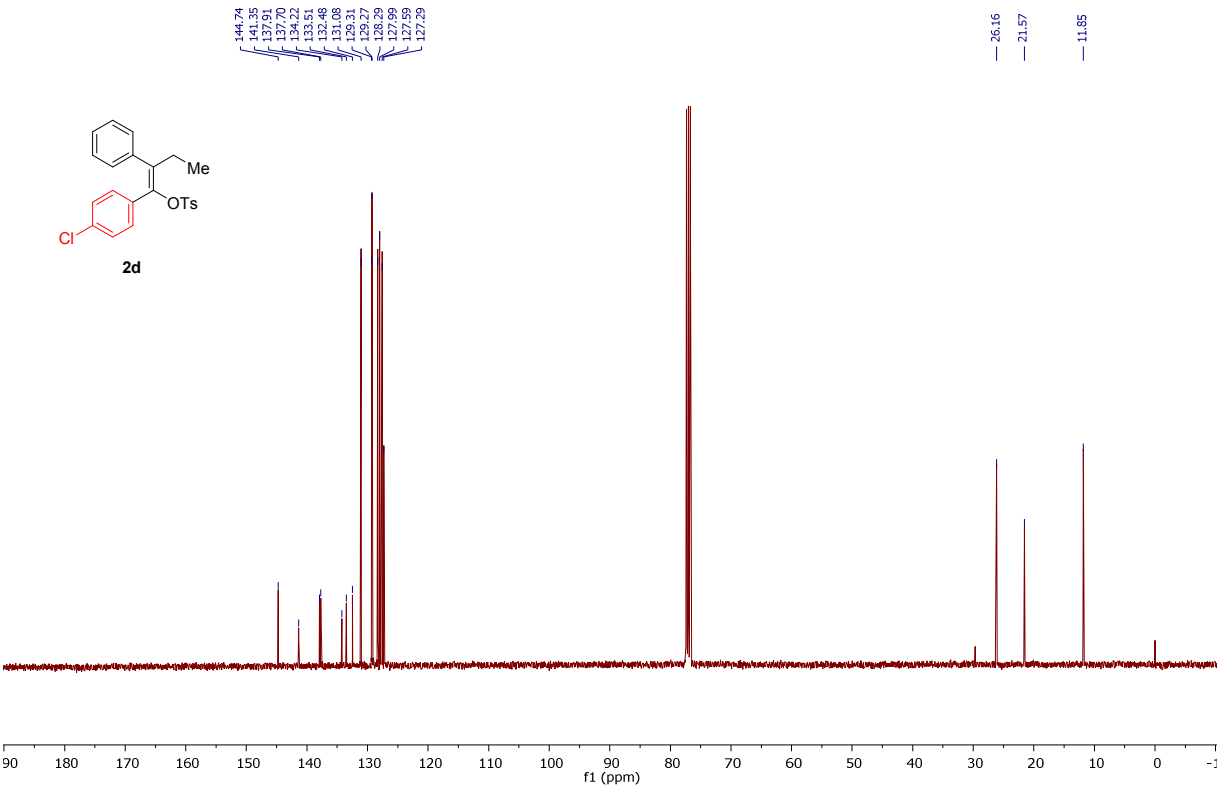
^{13}C NMR (101 MHz, CDCl$_3$) δ 158.87, 144.14, 142.56, 138.50, 135.60, 134.52, 131.19, 129.38, 129.14, 128.09, 127.99, 126.85, 126.30, 112.76, 55.04, 26.04, 21.53, 11.97.



PROTON16.gene CDCl3 /opt/topspin led6 89

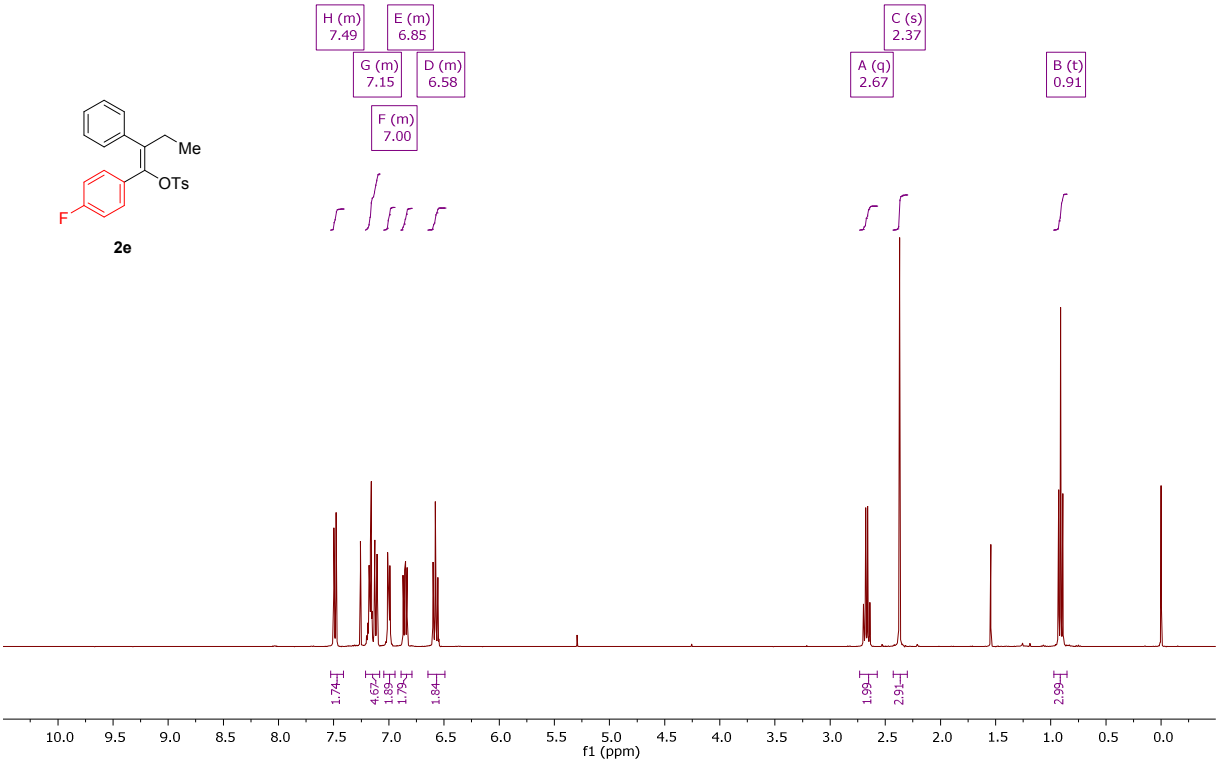


CARBON8K.gene CDCl3 /opt/topspin led6 89



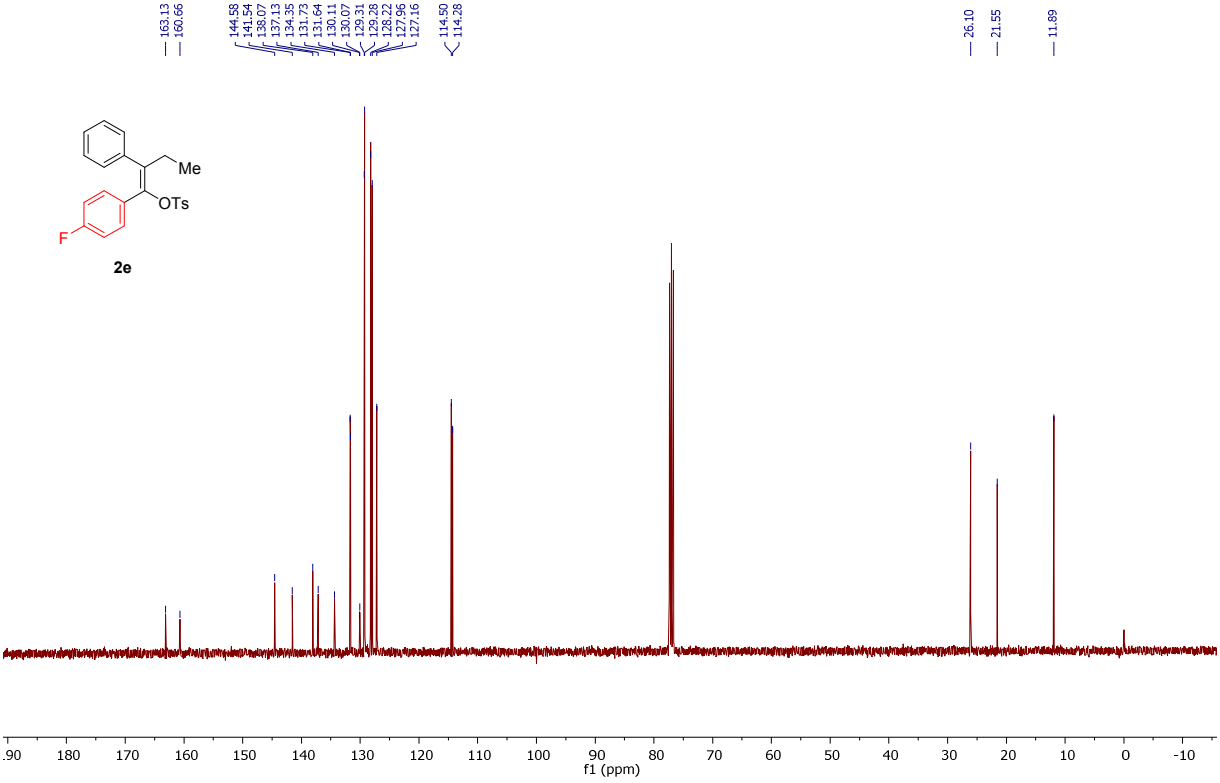
68863-191-1
PROTON16.gene CDCl3 /opt/topspin zhangh34 82

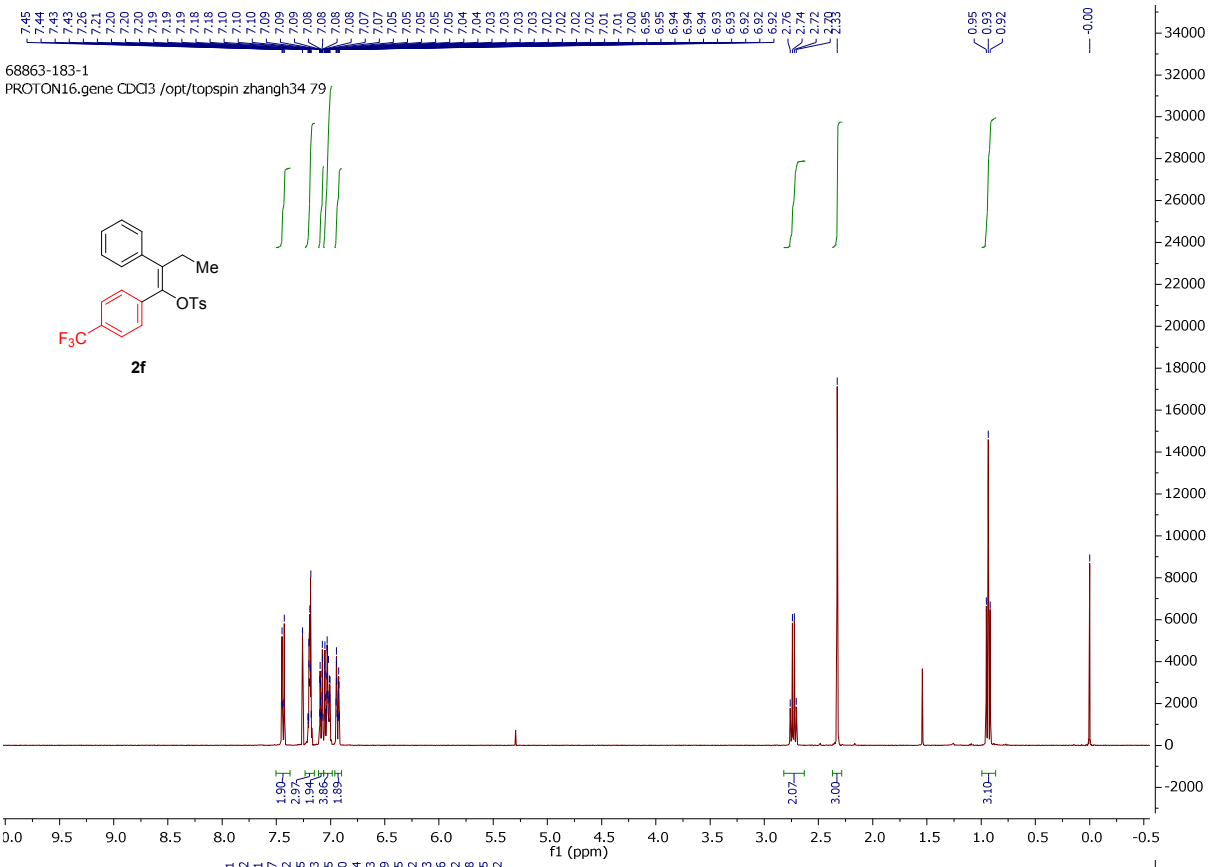
^{1}H NMR (400 MHz, Chloroform-d) δ 7.53 – 7.41 (m, 2H), 7.21 – 7.08 (m, 5H), 7.04 – 6.94 (m, 2H), 6.89 – 6.79 (m, 2H), 6.64 – 6.49 (m, 2H), 2.67 (q, J = 7.5 Hz, 2H), 2.37 (s, 3H), 0.91 (t, J = 7.5 Hz, 3H).

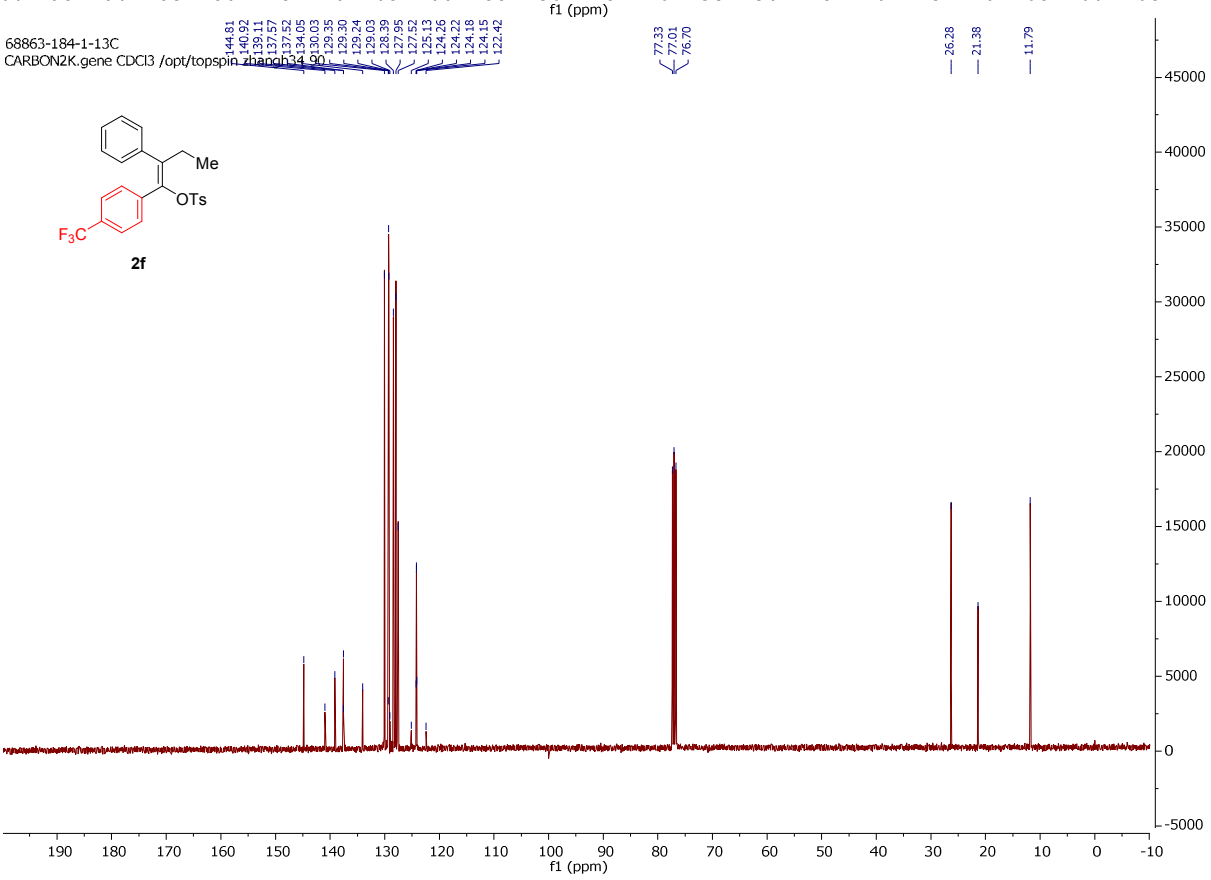


68863-191-1
CARBON2K.gene CDCl3 /opt/topspin zhangh34 82

^{13}C NMR (101 MHz, CDCl$_3$) δ 163.13, 160.66, 144.58, 141.54, 138.07, 137.13, 134.35, 131.73, 131.64, 130.11, 130.07, 129.31, 129.28, 128.22, 127.96, 127.16, 114.50, 114.28, 26.10, 21.55, 11.89.

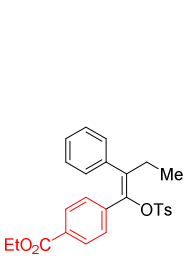



68863-183-1
PROTON16.gene CDCl3 /opt/topspin zhangh34 79
Me
OTs
F3C
2f
f1 (ppm)


68863-184-1-13C
CARBON2K.gene CDCl3 /opt/topspin zhangh34 90
Me
OTs
F3C
2f
f1 (ppm)

PROTON16.gene CDCl3 /opt/topspin led6 69

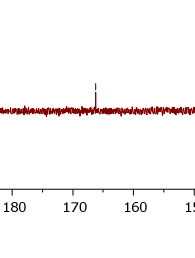
^{1}H NMR (400 MHz, Chloroform-d) δ 7.60 – 7.51 (m, 2H), 7.53 – 7.44 (m, 2H), 7.17 (ddt, J = 5.5, 4.0, 2.4 Hz, 3H), 7.09 (d, J = 8.1 Hz, 2H), 7.05 – 6.95 (m, 2H), 6.97 – 6.87 (m, 2H), 4.31 (q, J = 7.1 Hz, 2H), 2.68 (q, J = 7.5 Hz, 2H), 2.35 (s, 3H), 1.34 (t, J = 7.1 Hz, 3H), 0.91 (t, J = 7.5 Hz, 3H).



2g

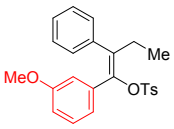
CARBON8K.gene CDCl3 /opt/topspin led6 55

^{13}C NMR (101 MHz, CDCl$_3$) δ 166.21, 144.93, 141.56, 138.99, 138.65, 137.94, 134.20, 129.79, 129.48, 129.41, 129.34, 128.67, 128.47, 128.17, 127.58, 61.08, 26.44, 21.67, 14.45, 11.96.



2g

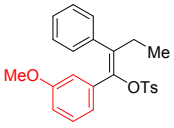
PROTON16.gene CDCl3 /opt/topspin led6 56



2h

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 – 7.42 (m, 2H), 7.23 – 7.12 (m, 3H), 7.12 – 6.99 (m, 4H), 6.79 (t, *J* = 7.9 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).

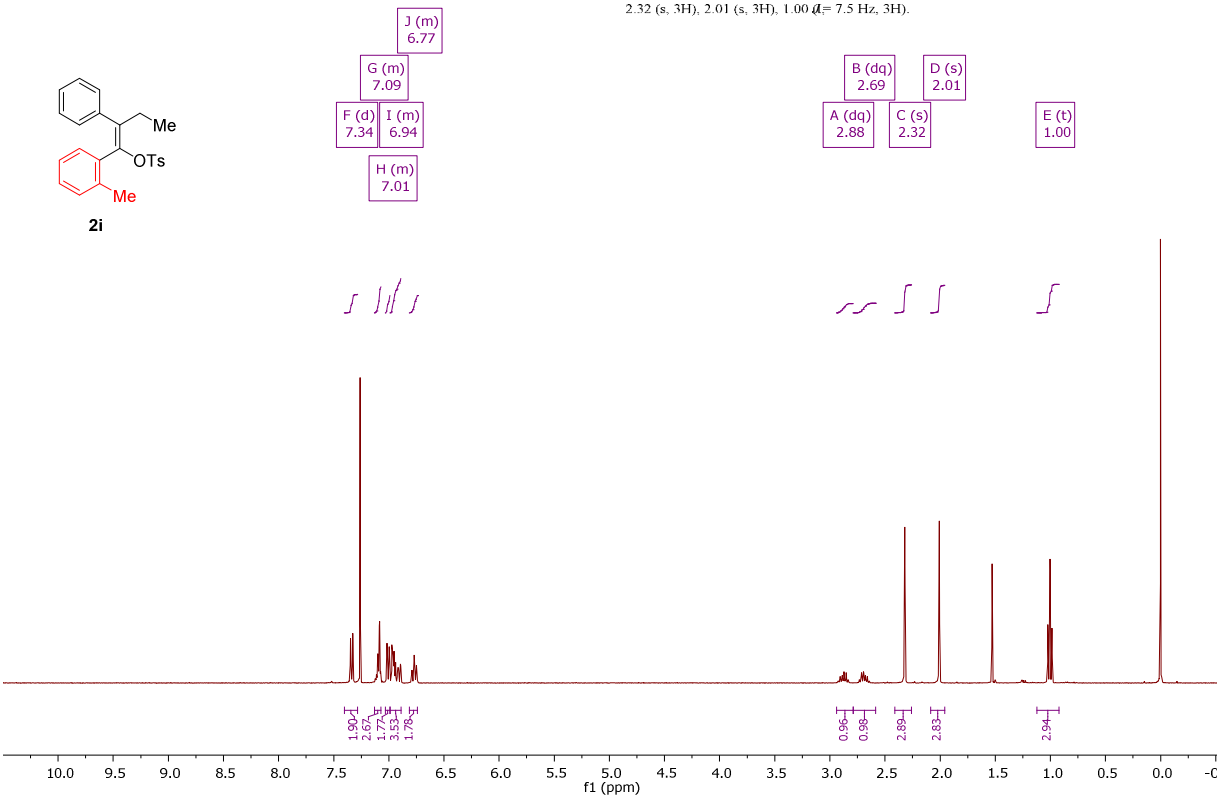
CARBON2K.gene CDCl3 /opt/topspin led6 56



2h

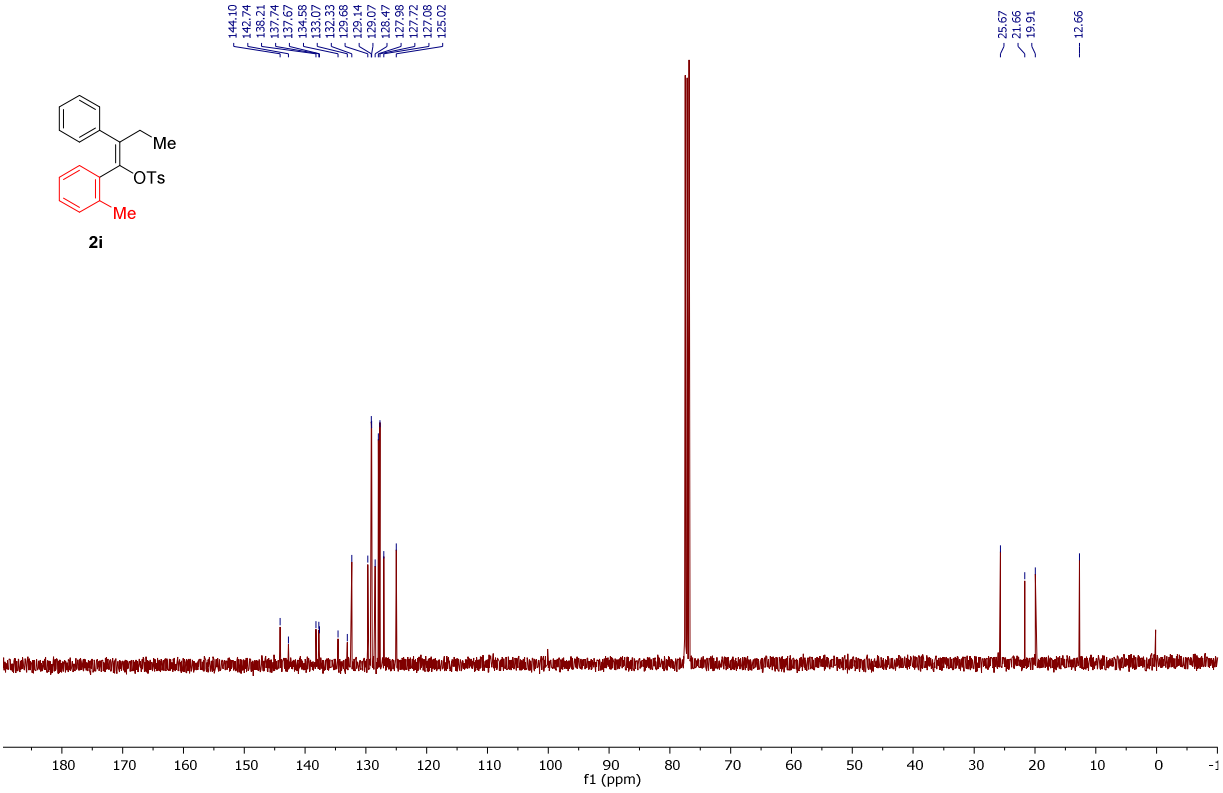
^{13}C NMR (101 MHz, CDCl$_3$) δ 158.46, 144.32, 142.42, 138.41, 137.02, 134.92, 134.40, 129.29, 129.13, 128.28, 128.15, 128.01, 127.06, 122.54, 114.38, 114.31, 54.80, 26.20, 21.52, 11.91.

PROTON16.gene CDCl3 /opt/topspin led6 3

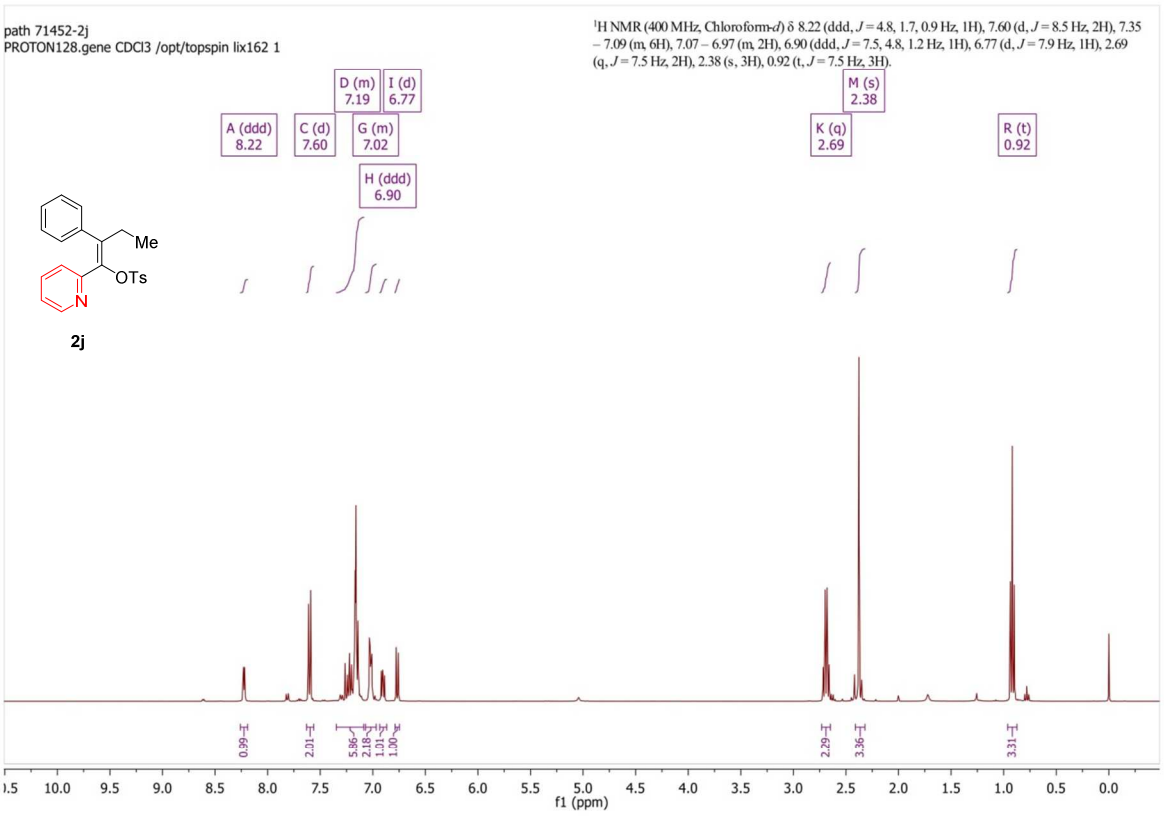


^{1}H NMR (400 MHz, Chloroform-d) δ 7.34 (d, J = 8.4 Hz, 2H), 7.13 – 7.07 (m, 3H), 7.03 – 6.99 (m, 2H), 6.99 – 6.89 (m, 4H), 6.81 – 6.74 (m, 2H), 2.88 (dq, J = 14.9, 7.5 Hz, 1H), 2.69 (dq, J = 14.8, 7.5 Hz, 1H), 2.32 (s, 3H), 2.01 (s, 3H), 1.00 (t, J = 7.5 Hz, 3H).

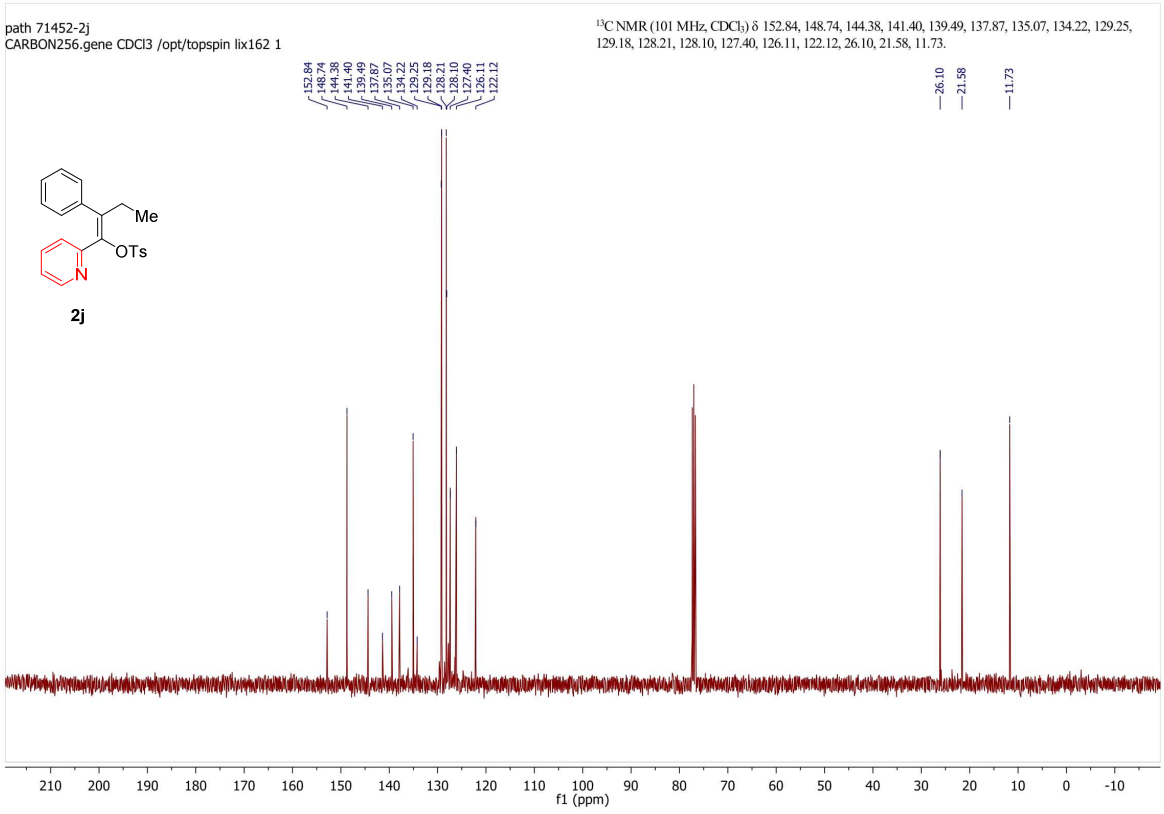
CARBON2K.gene CDCl3 /opt/topspin led6 38



^{13}C NMR (101 MHz, CDCl$_3$) δ 144.10, 142.74, 138.21, 137.74, 137.67, 134.58, 133.07, 132.33, 129.68, 129.14, 129.07, 128.47, 127.98, 127.72, 127.08, 125.02, 25.67, 21.66, 19.91, 12.66.



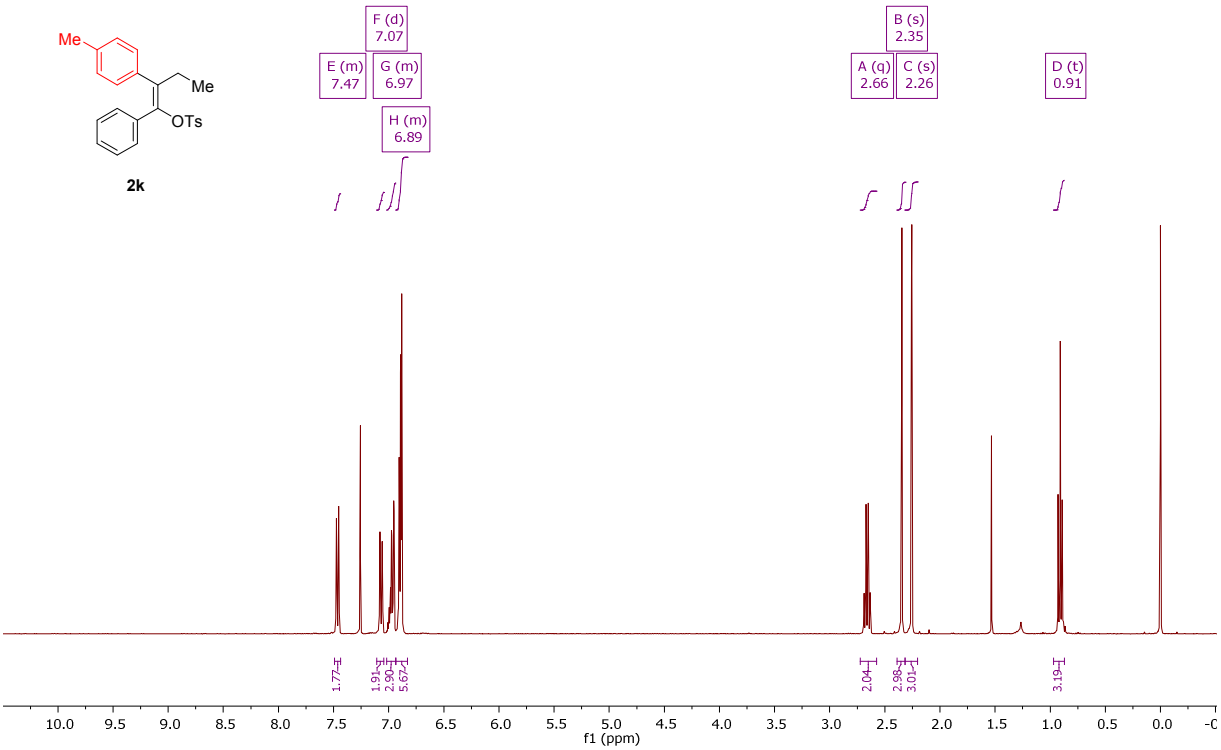
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.22 (ddd, *J* = 4.8, 1.7, 0.9 Hz, 1H), 7.60 (d, *J* = 8.5 Hz, 2H), 7.35 – 7.09 (m, 6H), 7.07 – 6.97 (m, 2H), 6.90 (ddd, *J* = 7.5, 4.8, 1.2 Hz, 1H), 6.77 (d, *J* = 7.9 Hz, 1H), 2.69 (q, *J* = 7.5 Hz, 2H), 2.38 (s, 3H), 0.92 (t, *J* = 7.5 Hz, 3H).



^{13}C NMR (101 MHz, $CDCl_3$) δ 152.84, 148.74, 144.38, 141.40, 139.49, 137.87, 135.07, 134.22, 129.25, 129.18, 128.21, 128.10, 127.40, 126.11, 122.12, 26.10, 21.58, 11.73.

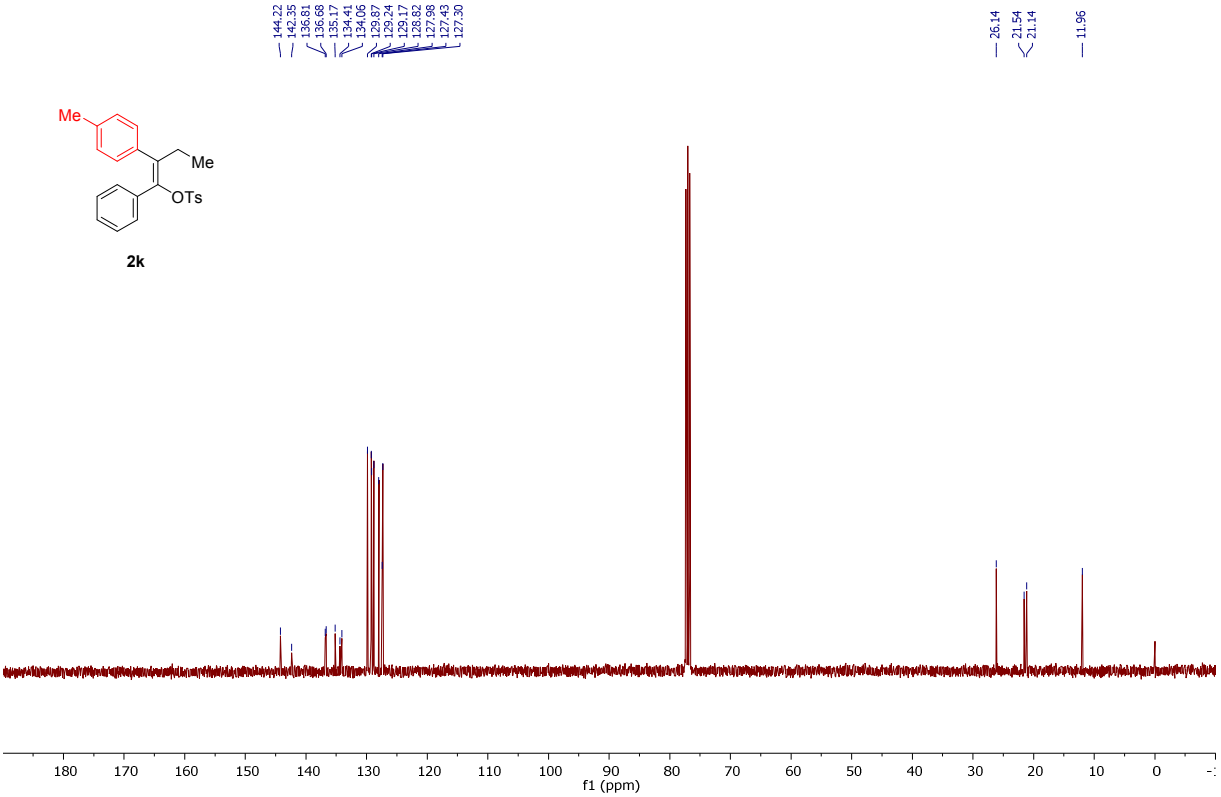
PROTON16.gene CDCl3 /opt/topspin led6 23

^{1}H NMR (400 MHz, Chloroform-d) 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).



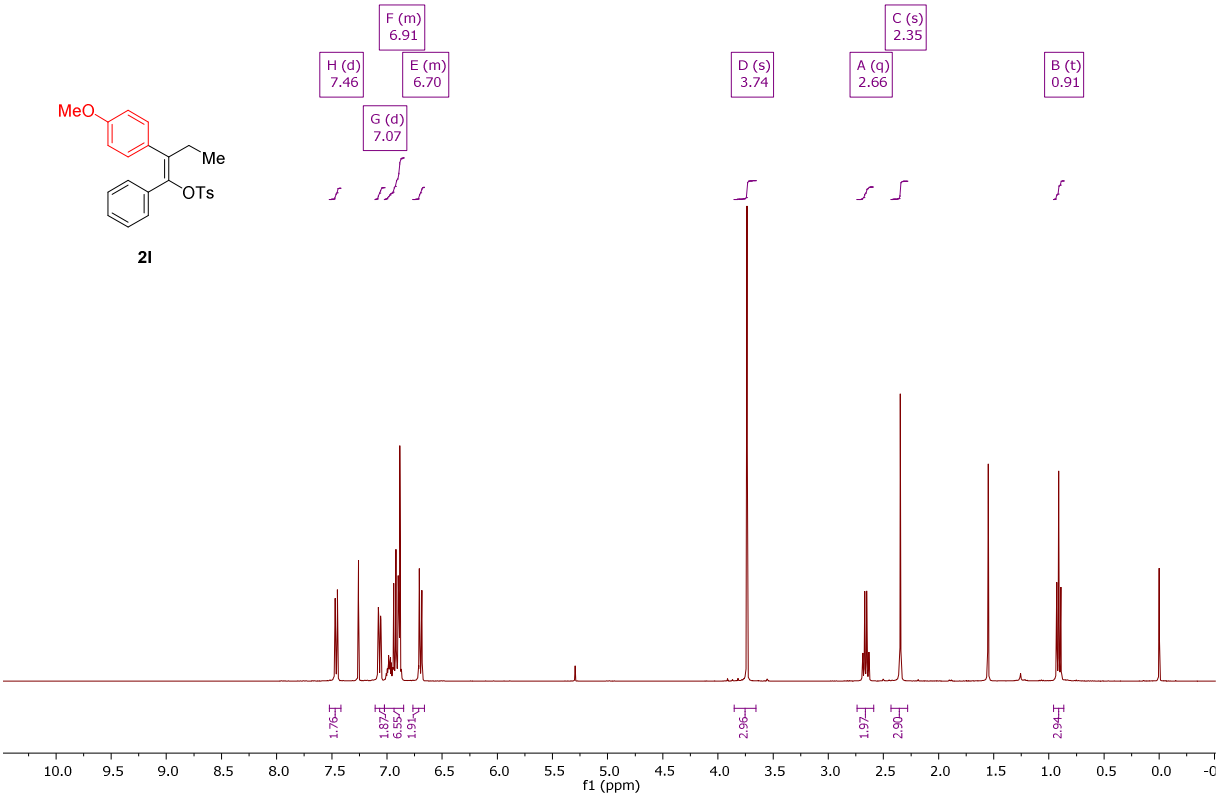
CARBON2K.gene CDCl3 /opt/topspin led6 23

^{13}C NMR (101 MHz, CDCl3) δ 144.22, 142.35, 136.81, 136.68, 135.17, 134.41, 134.06, 129.87, 129.24, 129.17, 128.82, 127.98, 127.43, 127.30, 26.14, 21.54, 21.14, 11.96.



PROTON16.gene CDCl3 /opt/topspin led6 18

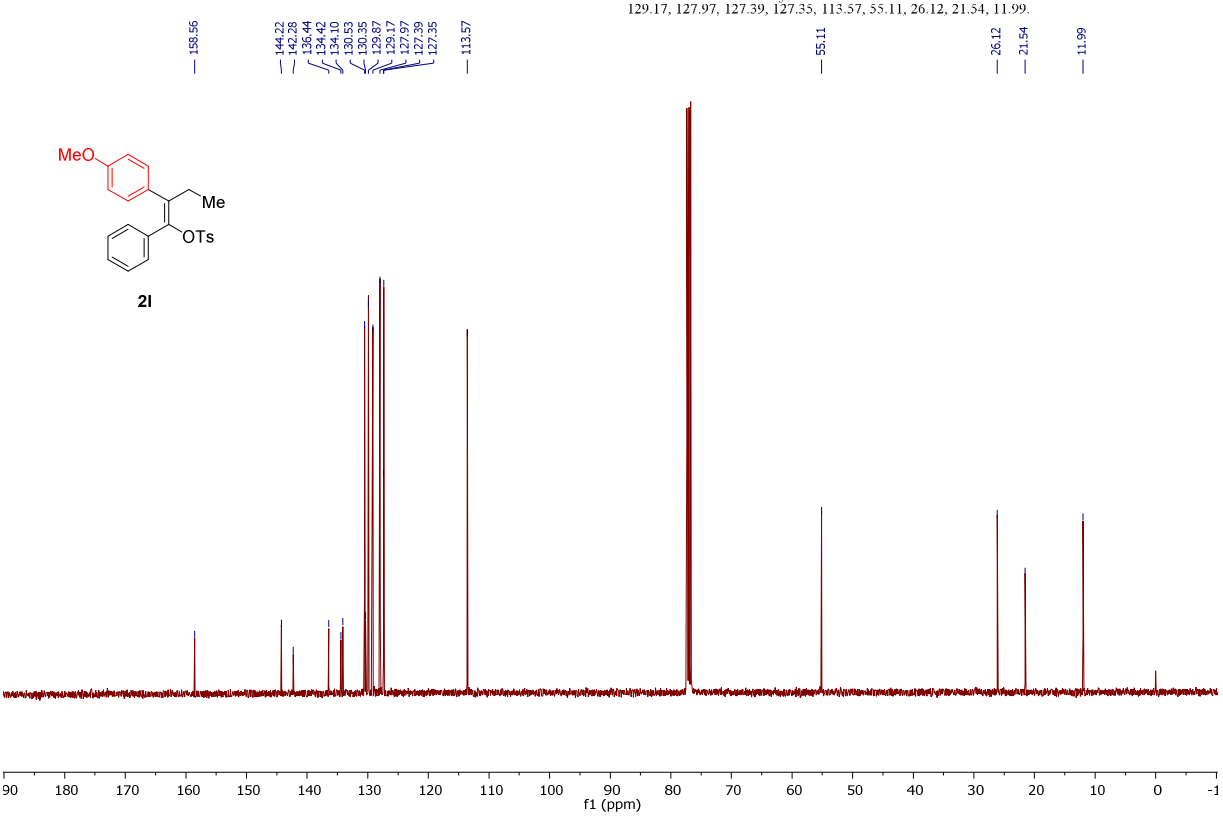
^{1}H NMR (400 MHz, Chloroform-d) δ 7.46 (d, J = 8.4 Hz, 2H), 7.07 (d, J = 8.1 Hz, 2H), 7.02 – 6.85 (m, 7H), 6.77 – 6.66 (m, 2H), 3.74 (s, 3H), 2.66 (q, J = 7.5 Hz, 2H), 2.35 (s, 3H), 0.91 (t, J = 7.5 Hz, 3H).



2l

CARBON8K.gene CDCl3 /opt/topspin led6 18

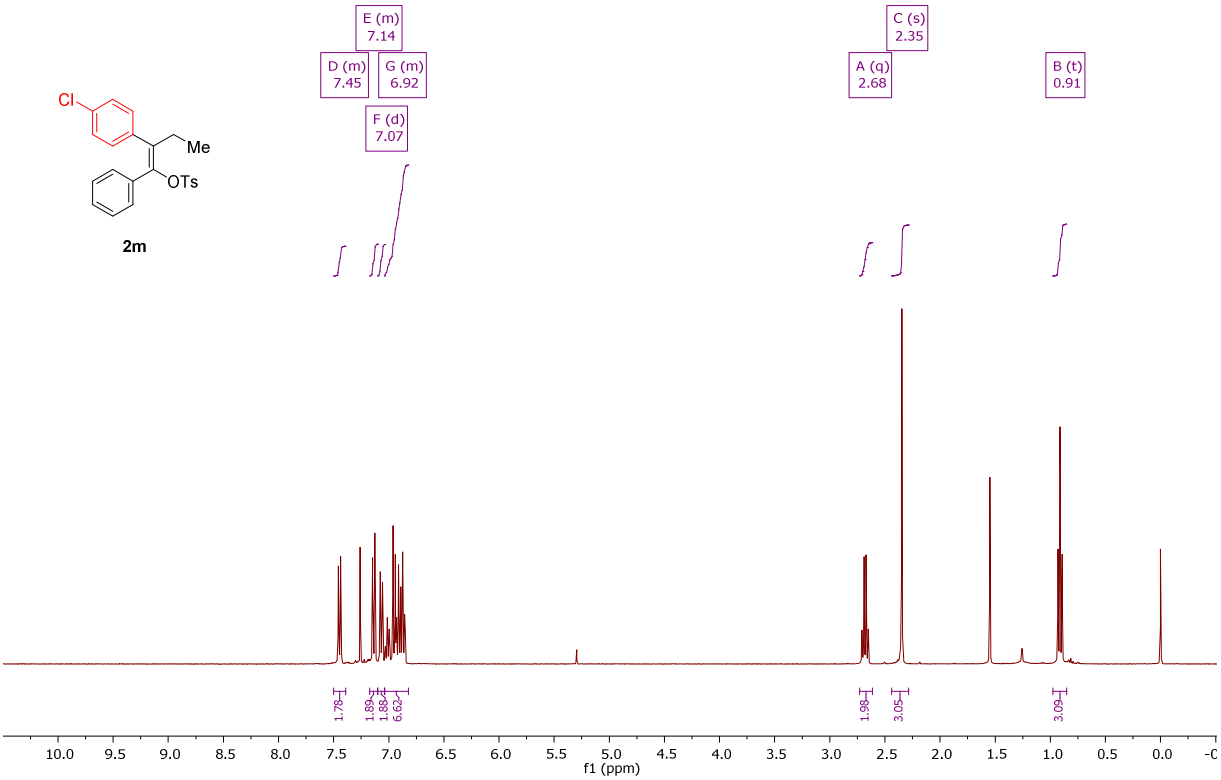
^{13}C NMR (101 MHz, CDCl$_3$) δ 158.56, 144.22, 142.28, 136.44, 134.42, 134.10, 130.53, 130.35, 129.87, 129.17, 127.97, 127.39, 127.35, 113.57, 55.11, 26.12, 21.54, 11.99.



2l

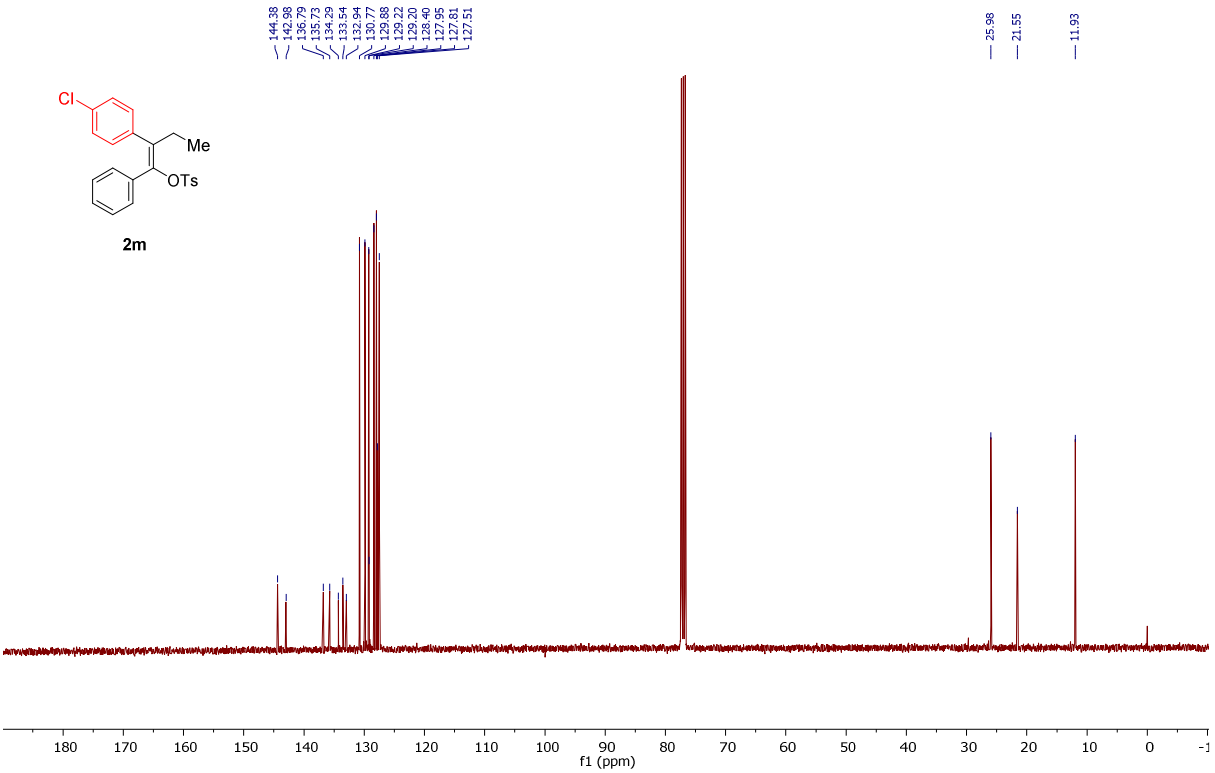
PROTON16.gene CDCl3 /opt/topspin led6 56

^{1}H NMR (400 MHz, Chloroform-d) 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).



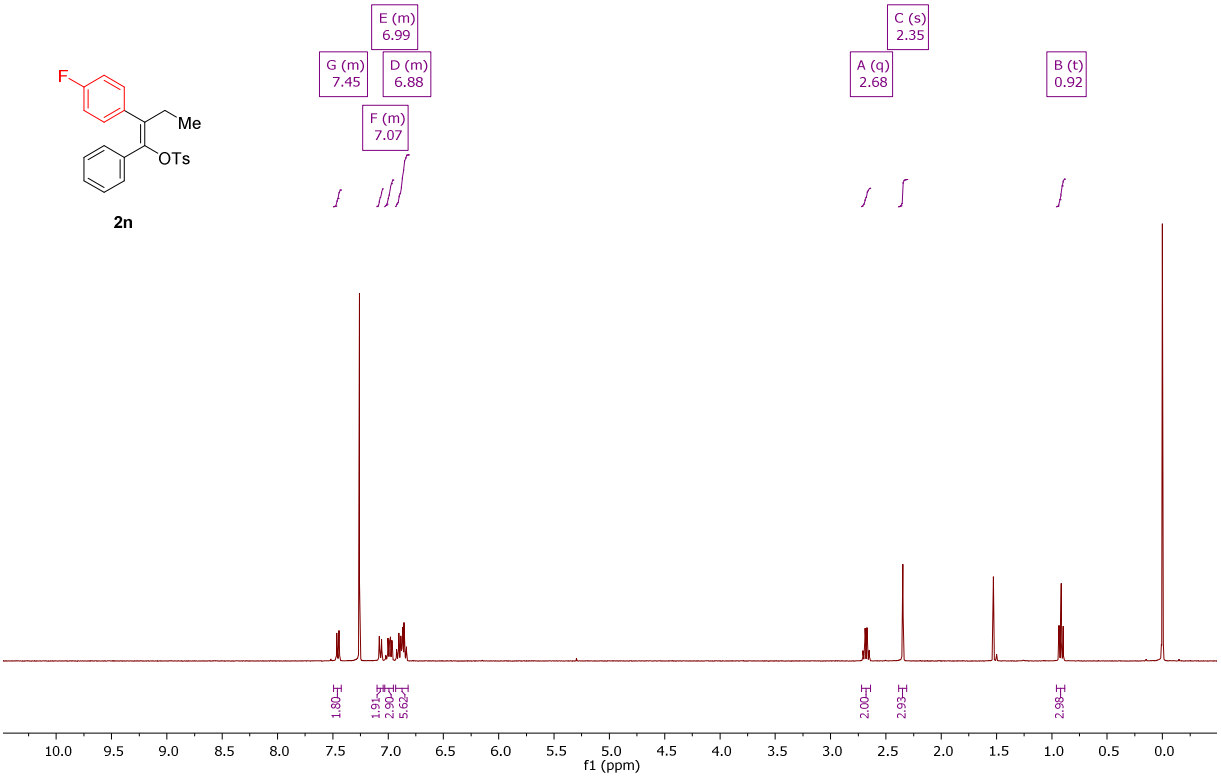
CARBON8K.gene CDCl3 /opt/topspin led6 56

^{13}C NMR (101 MHz, CDCl$_3$) δ 144.38, 142.98, 136.79, 135.73, 134.29, 133.54, 132.94, 130.77, 129.88, 129.22, 129.20, 128.40, 127.95, 127.81, 127.51, 25.98, 21.55, 11.93.

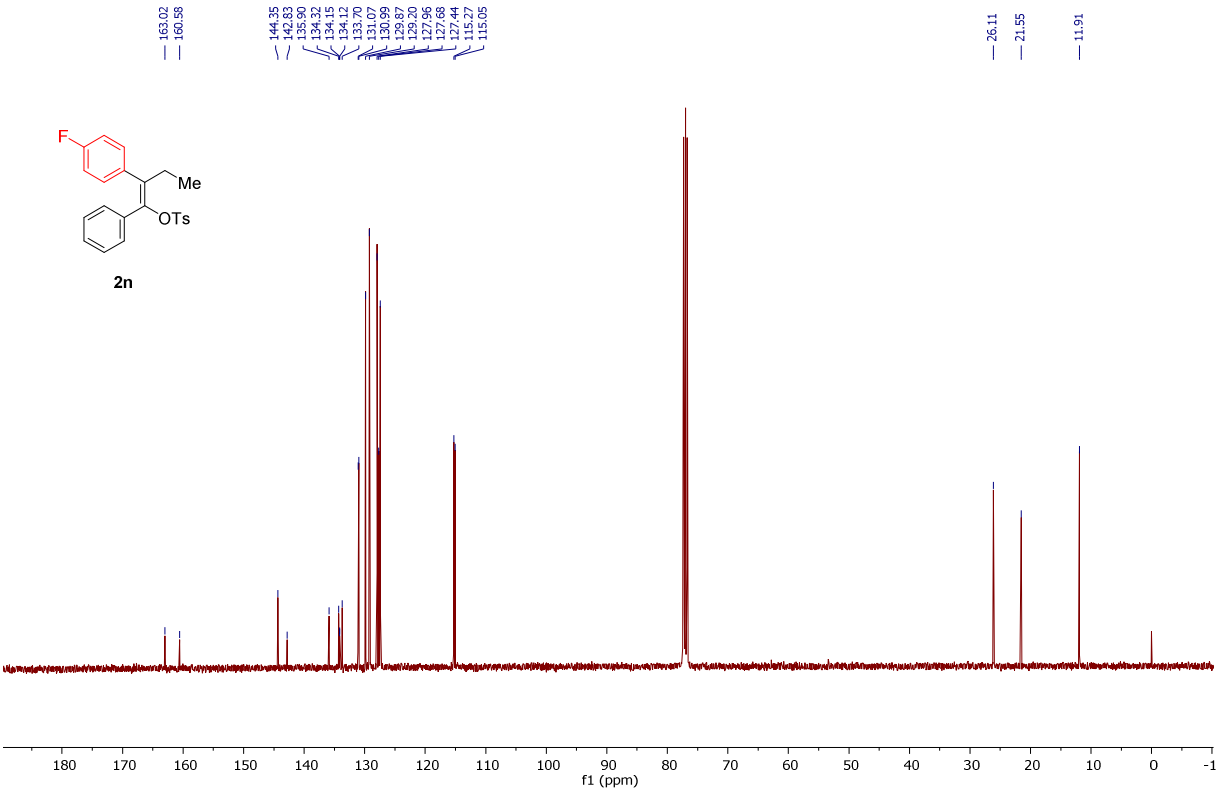


PROTON16.gene CDCl3 /opt/topspin led6 35

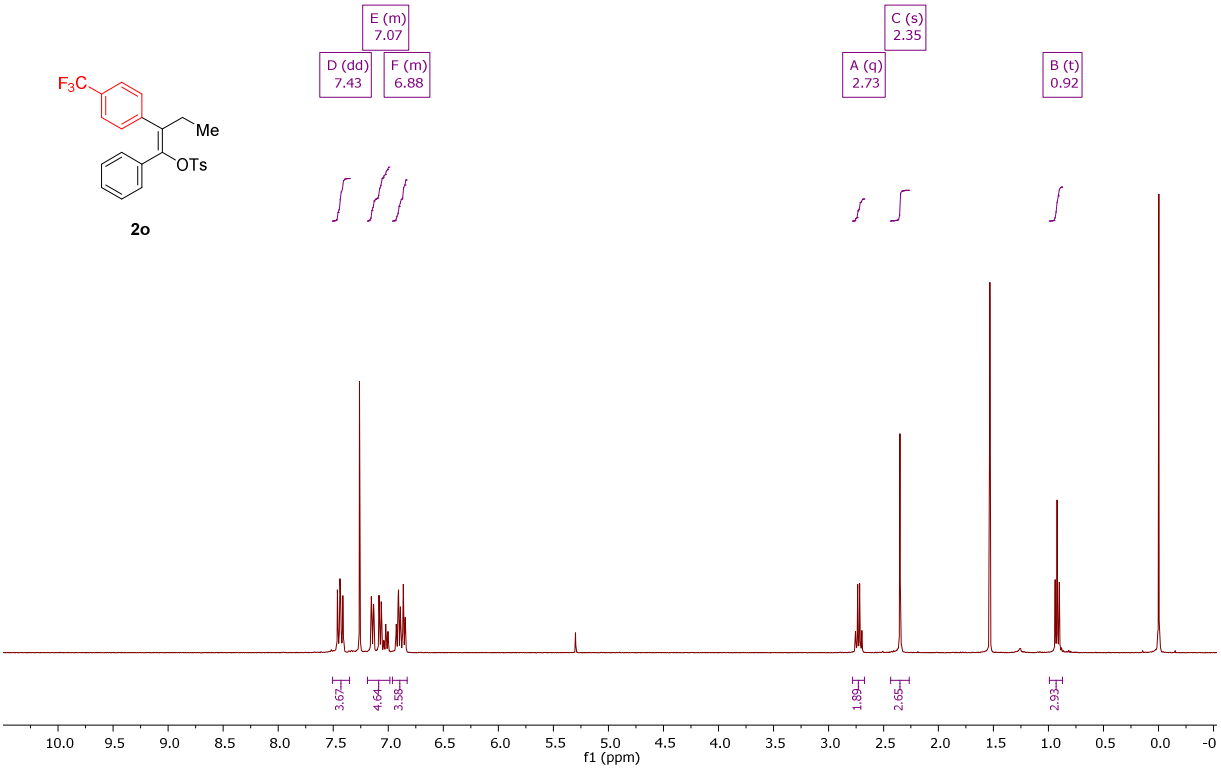
^{1}H NMR (400 MHz, Chloroform-d) δ 7.49 – 7.42 (m, 2H), 7.10 – 7.04 (m, 2H), 7.03 – 6.95 (m, 3H), 6.93 – 6.82 (m, 6H), 2.68 (q, J = 7.5 Hz, 2H), 2.35 (s, 3H), 0.92 (t, J = 7.5 Hz, 3H).



^{13}C NMR (101 MHz, CDCl$_3$) δ 163.02, 160.58, 144.35, 142.83, 135.90, 134.32, 134.15, 134.12, 133.70, 131.07, 130.99, 129.87, 129.20, 127.96, 127.68, 127.44, 115.27, 115.05, 26.11, 21.55, 11.91.

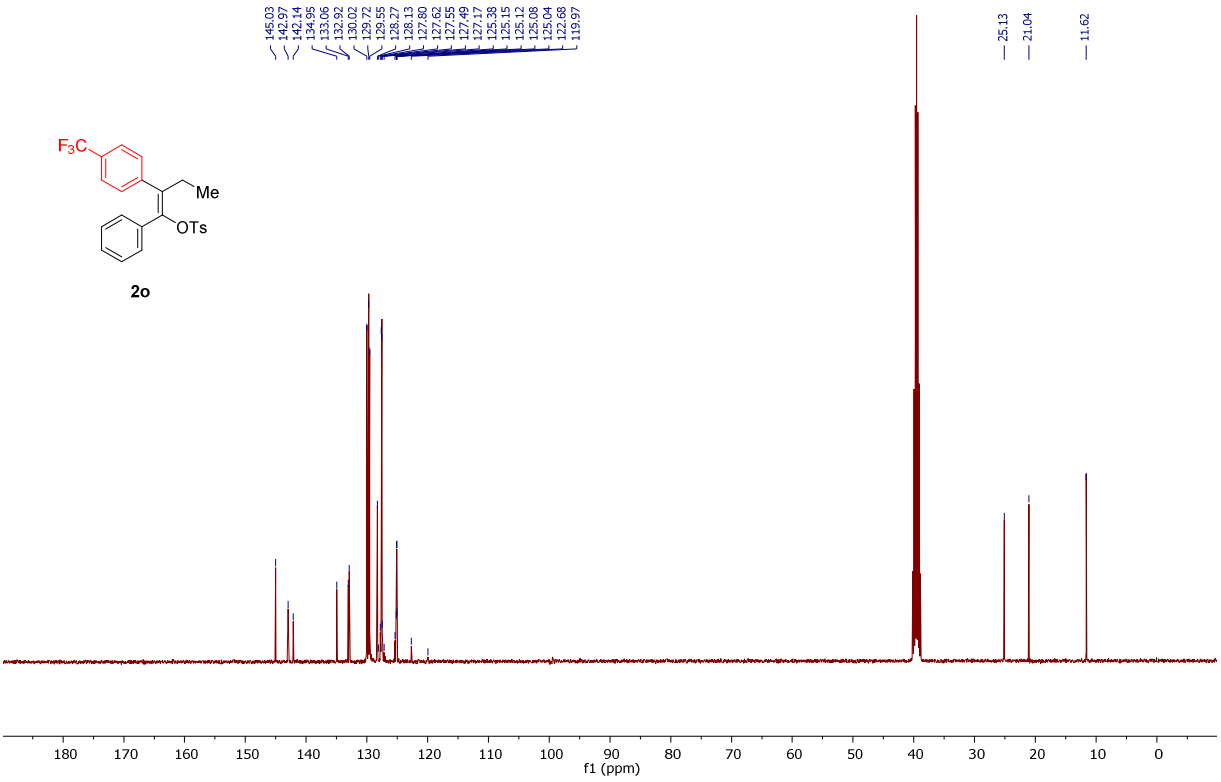


PROTON16.gene CDCl3 /opt/topspin led6 82

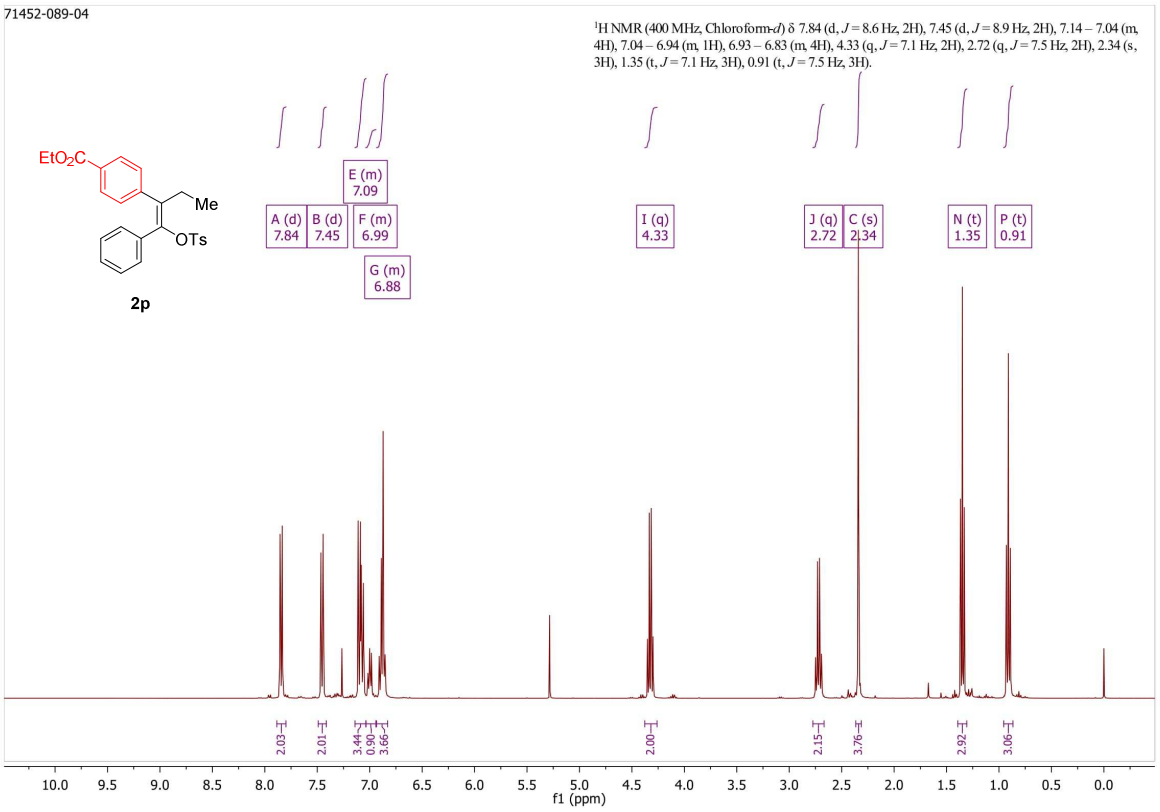


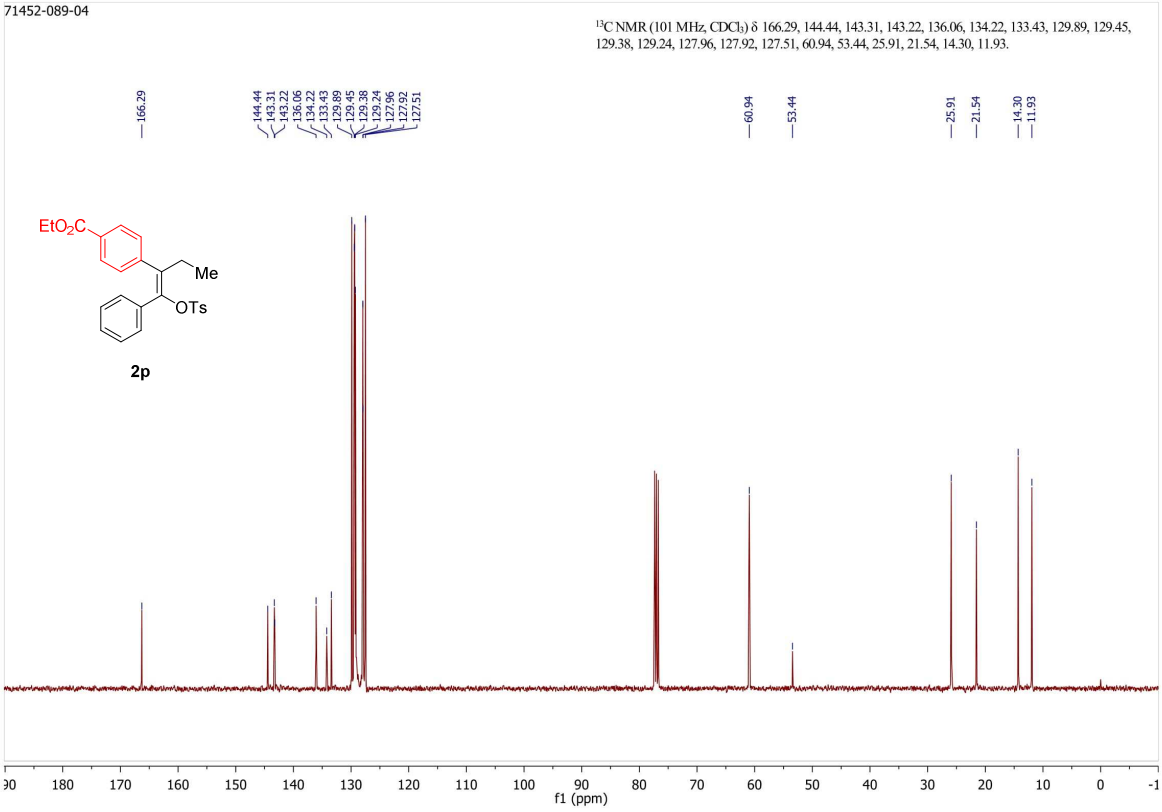
^{1}H NMR (400 MHz, Chloroform-d) δ 7.43 (dd, J = 11.5, 8.3 Hz, 4H), 7.19 – 6.98 (m, 5H), 6.96 – 6.8
(m, 4H), 2.73 (q, J = 7.5 Hz, 2H), 2.35 (s, 3H), 0.92 (t, J = 7.5 Hz, 3H).

CARBON8K.gene DMSO /opt/topspin led6 20

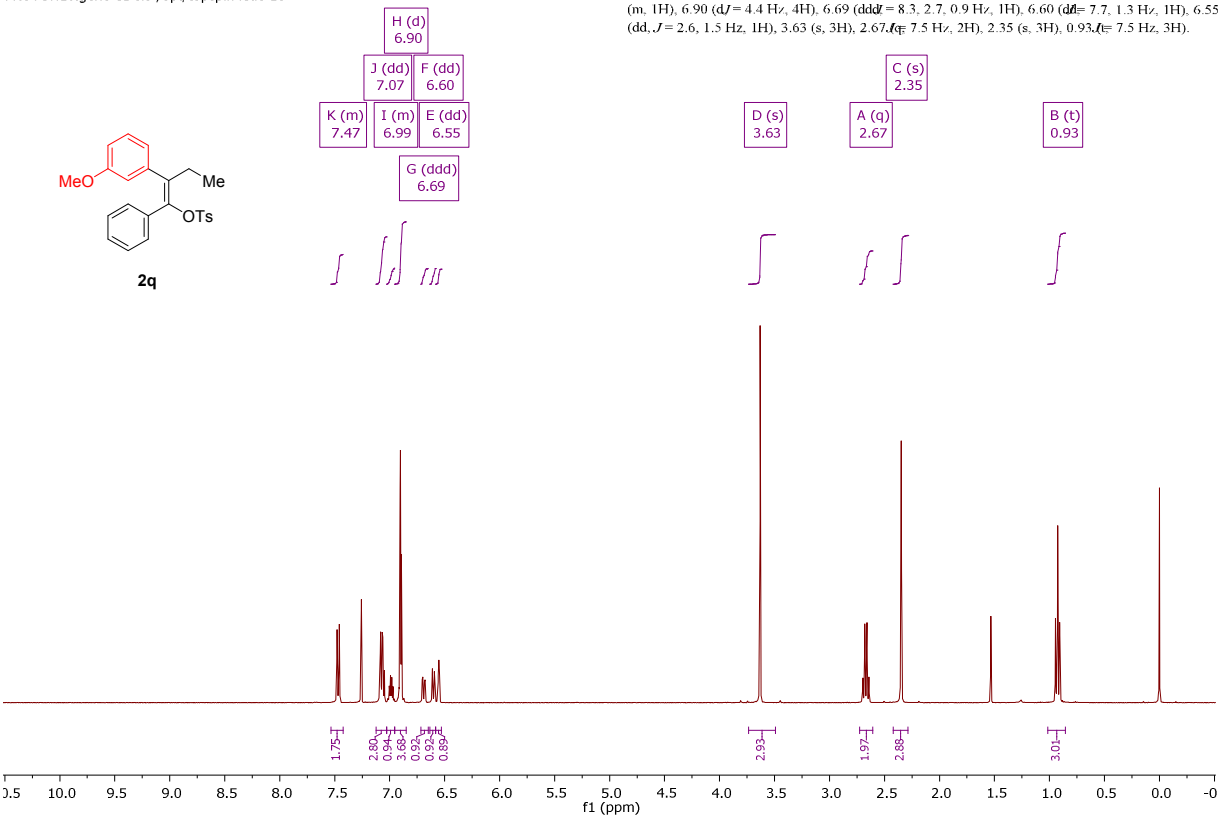


^{13}C NMR (101 MHz, DMSO-d_6) δ 145.03, 142.97, 142.14, 134.95, 133.06, 132.92, 130.02, 129.72, 129.
128.27, 128.13, 127.80, 127.62, 127.55, 127.49, 127.17, 125.38, 125.15, 125.12, 125.08, 125.04, 1
119.97, 25.13, 21.04, 11.62.

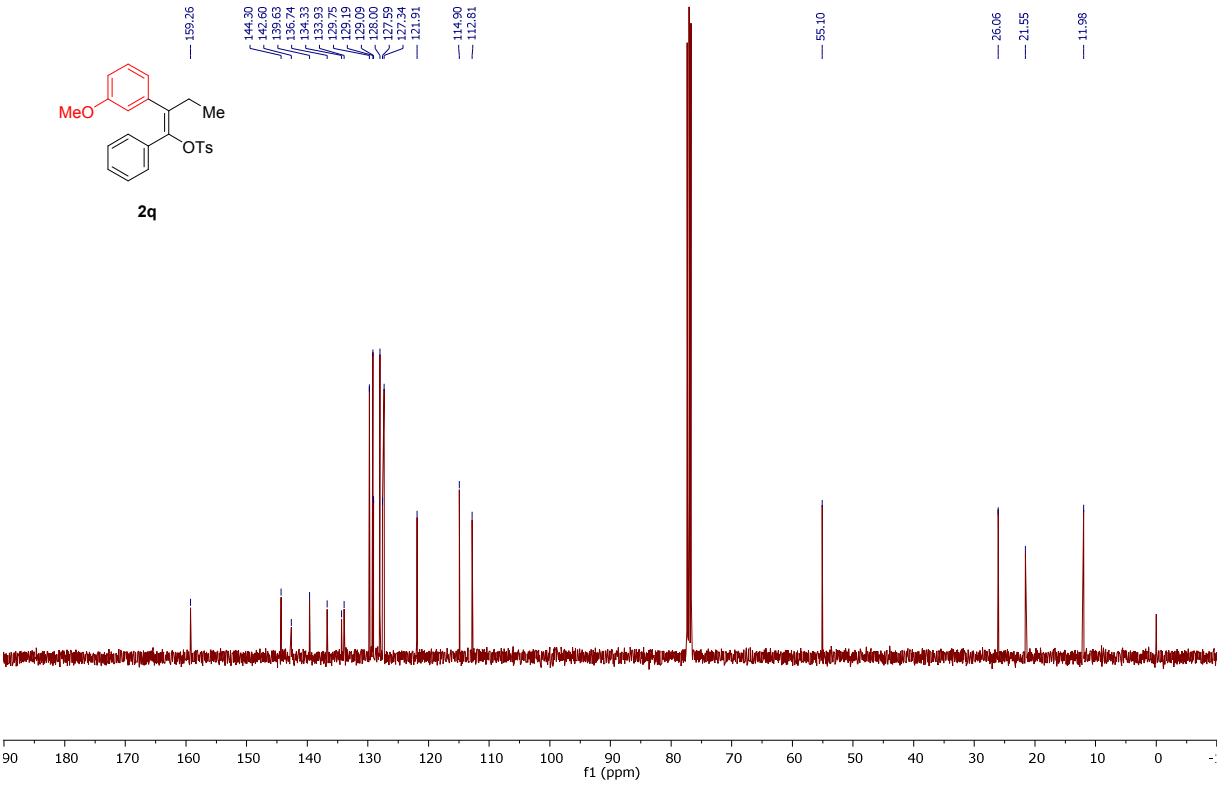

71452-089-04
^{1}H NMR (400 MHz, Chloroform-d) δ 7.84 (d, J = 8.6 Hz, 2H), 7.45 (d, J = 8.9 Hz, 2H), 7.14 – 7.04 (m, 4H), 7.04 – 6.94 (m, 1H), 6.93 – 6.83 (m, 4H), 4.33 (q, J = 7.1 Hz, 2H), 2.72 (q, J = 7.5 Hz, 2H), 2.34 (s, 3H), 1.35 (t, J = 7.1 Hz, 3H), 0.91 (t, J = 7.5 Hz, 3H).
EtO2C
Me
OTs
2p
A (d) 7.84
B (d) 7.45
E (m) 7.09
F (m) 6.99
G (m) 6.88
I (q) 4.33
J (q) 2.72
C (s) 2.34
N (t) 1.35
P (t) 0.91
2.03
2.01
3.44
0.90
3.66
2.00
2.15
3.76
2.92
3.06
f1 (ppm)


71452-089-04
^{13}C NMR (101 MHz, $CDCl_3$) δ 166.29, 144.44, 143.31, 143.22, 136.06, 134.22, 133.43, 129.89, 129.45, 129.38, 129.24, 127.96, 127.92, 127.51, 60.94, 53.44, 25.91, 21.54, 14.30, 11.93.
166.29
144.44
143.31
143.22
136.06
134.22
133.43
129.89
129.45
129.38
129.24
127.96
127.92
127.51
60.94
53.44
25.91
21.54
14.30
11.93
EtO2C
Me
OTs
2p
f1 (ppm)

PROTON16.gene CDCl3 /opt/topspin led6 16

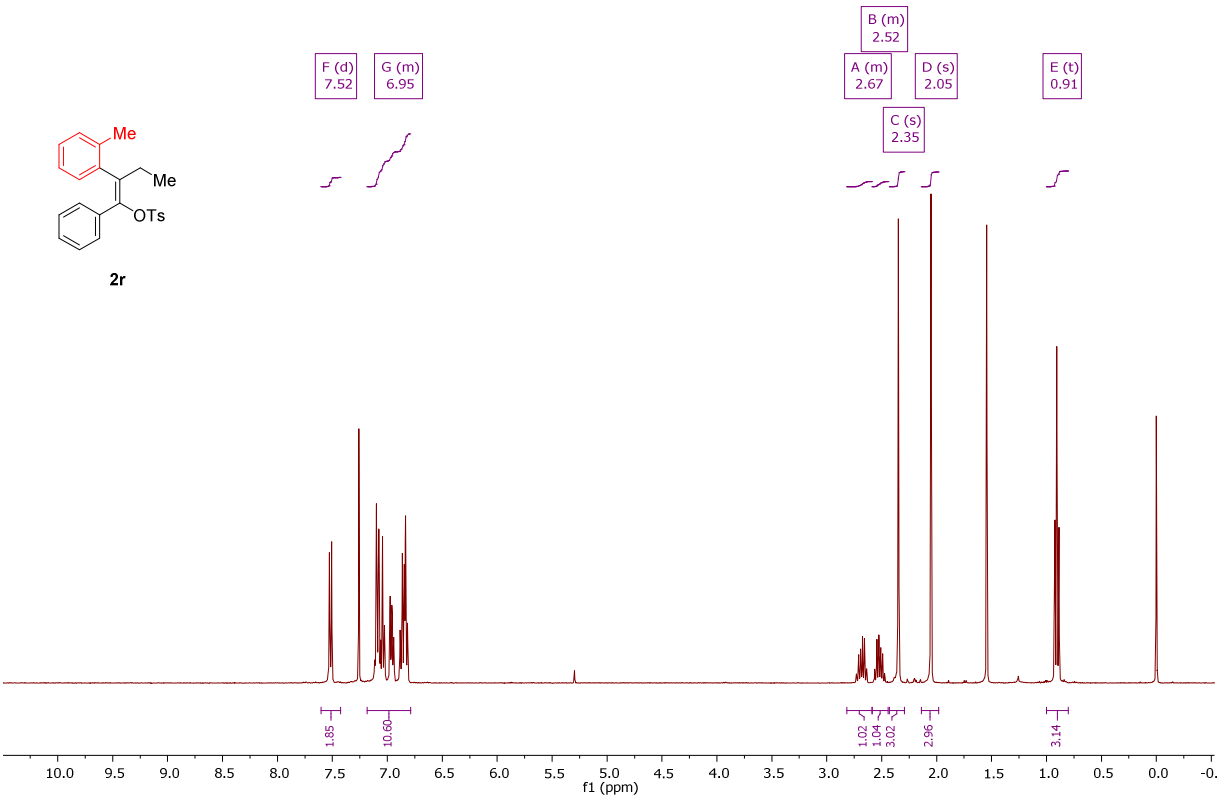


CARBON2K.gene CDCl3 /opt/topspin led6 16



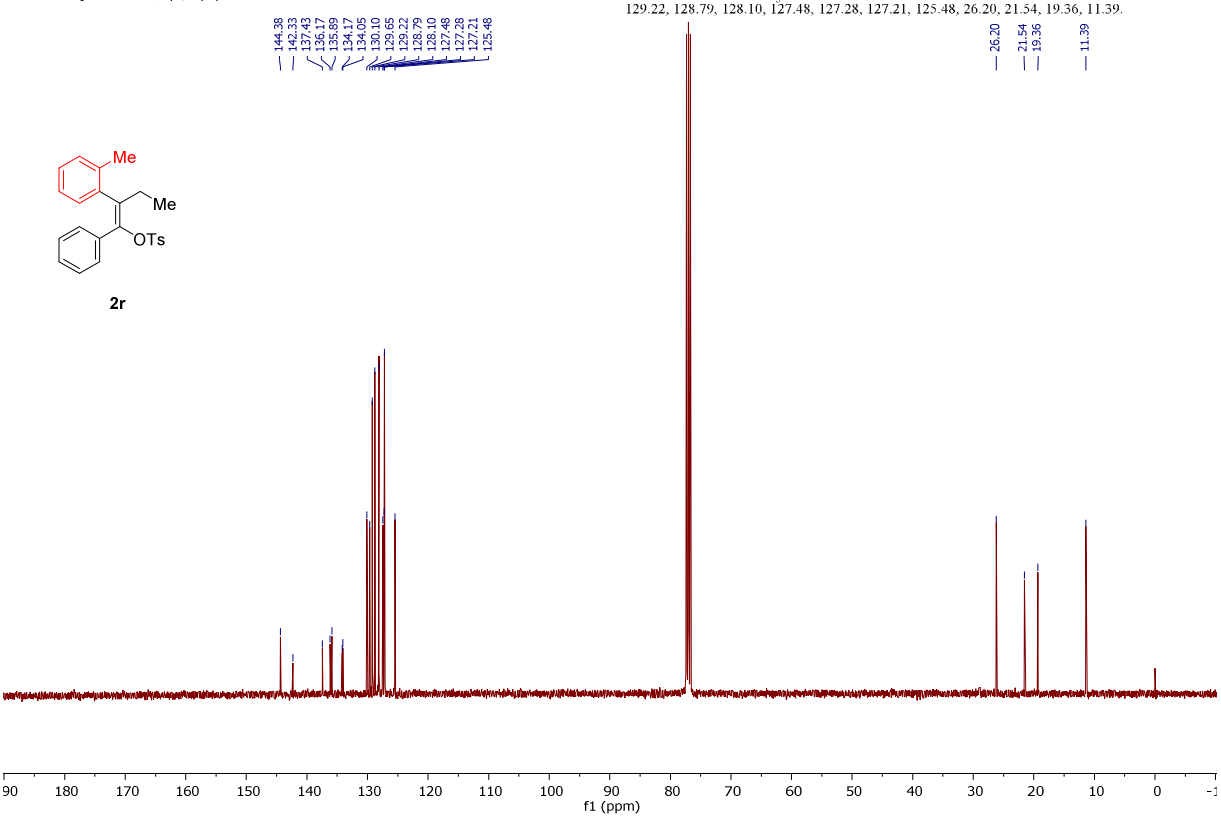
PROTON16.gene CDCl3 /opt/topspin led6 19

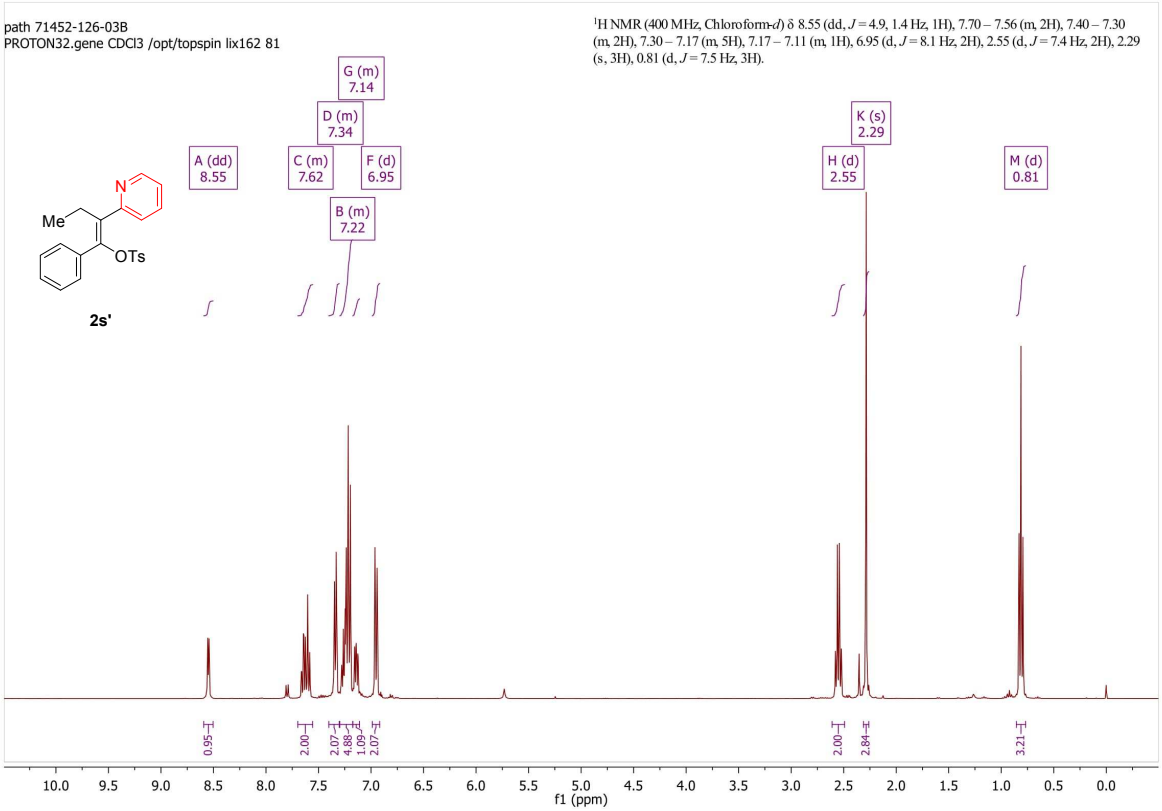
^{1}H NMR (400 MHz, Chloroform-d) δ 7.52 (d, J = 8.3 Hz, 2H), 7.18 – 6.79 (m, 11H), 2.82 – 2.59 (m, 1H), 2.59 – 2.44 (m, 1H), 2.35 (s, 3H), 2.05 (s, 3H), 0.91 (t, J = 7.5 Hz, 3H).



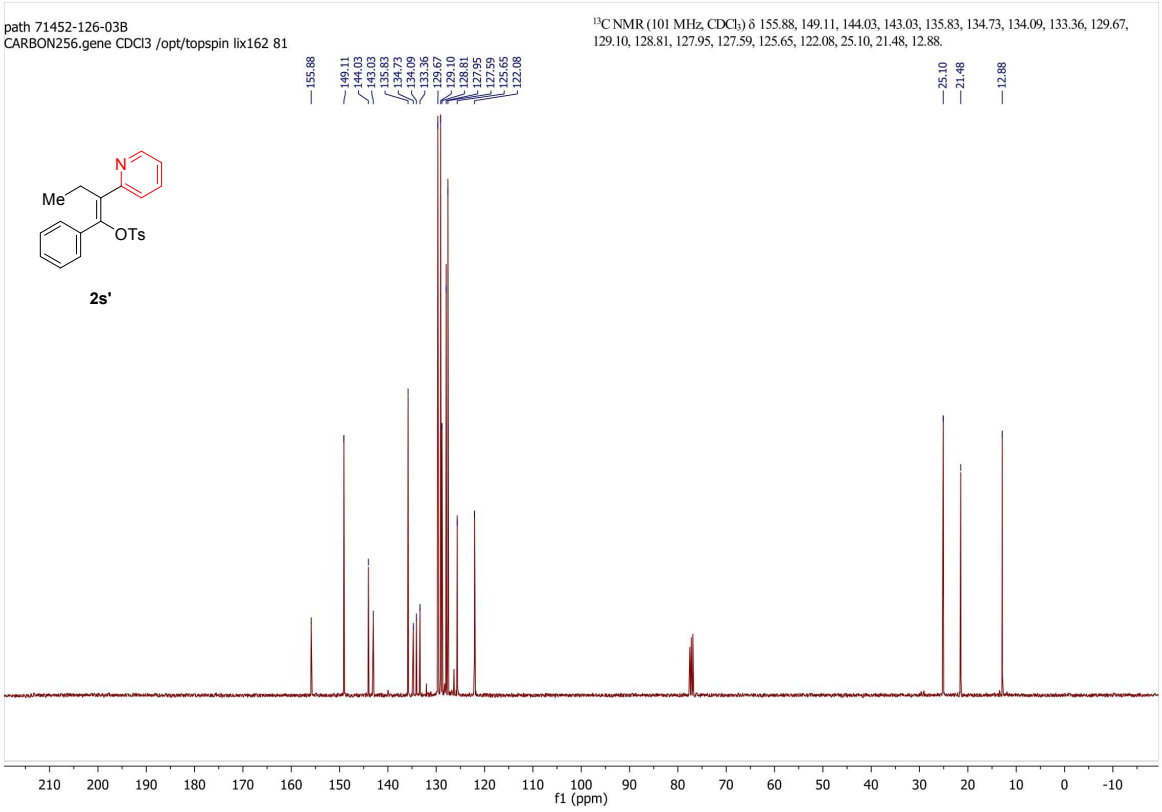
CARBON8K.gene CDCl3 /opt/topspin led6 19

^{13}C NMR (101 MHz, CDCl3) δ 144.38, 142.33, 137.43, 136.17, 135.89, 134.17, 134.05, 130.10, 129.65, 129.22, 128.79, 128.10, 127.48, 127.28, 127.21, 125.48, 26.20, 21.54, 19.36, 11.39.





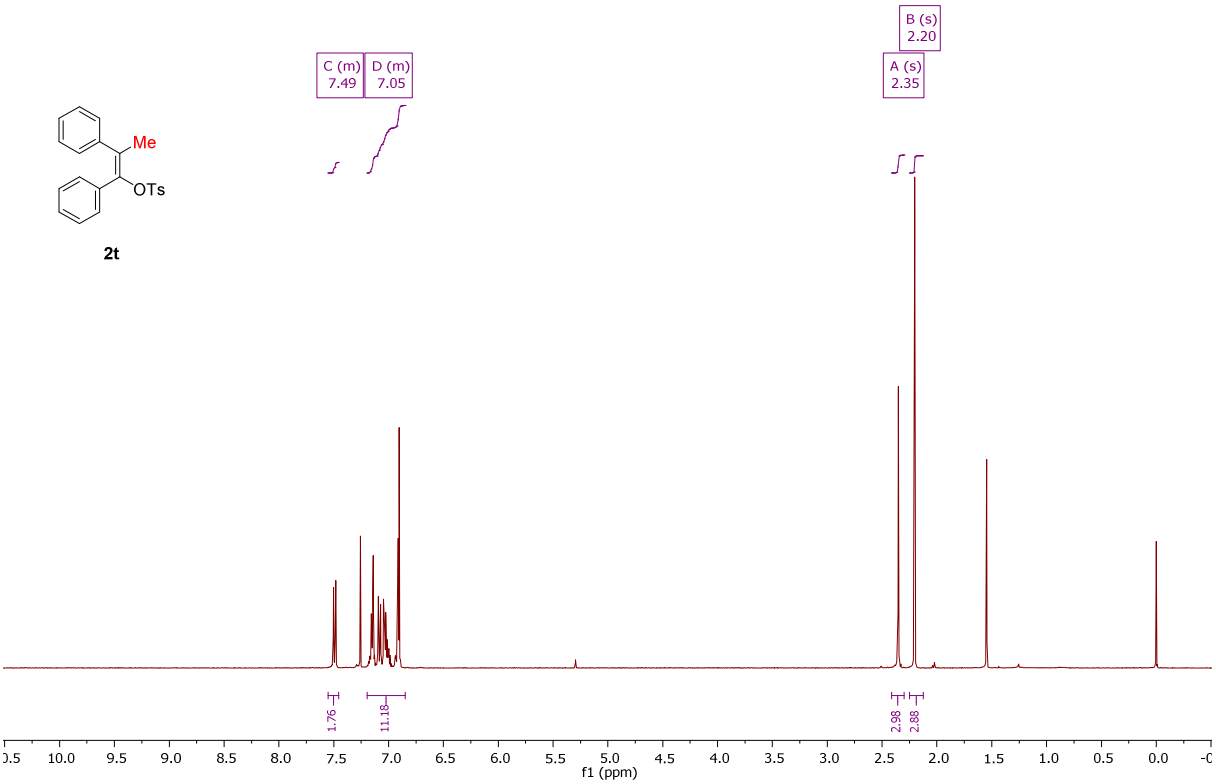
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.55 (dd, *J* = 4.9, 1.4 Hz, 1H), 7.70 – 7.56 (m, 2H), 7.40 – 7.30 (m, 2H), 7.30 – 7.17 (m, 5H), 7.17 – 7.11 (m, 1H), 6.95 (d, *J* = 8.1 Hz, 2H), 2.55 (d, *J* = 7.4 Hz, 2H), 2.29 (s, 3H), 0.81 (d, *J* = 7.5 Hz, 3H).



^{13}C NMR (101 MHz, $CDCl_3$) δ 155.88, 149.11, 144.03, 143.03, 135.83, 134.73, 134.09, 133.36, 129.67, 129.10, 128.81, 127.95, 127.59, 125.65, 122.08, 25.10, 21.48, 12.88.

PROTON16.gene CDCl3 /opt/topspin led6 17

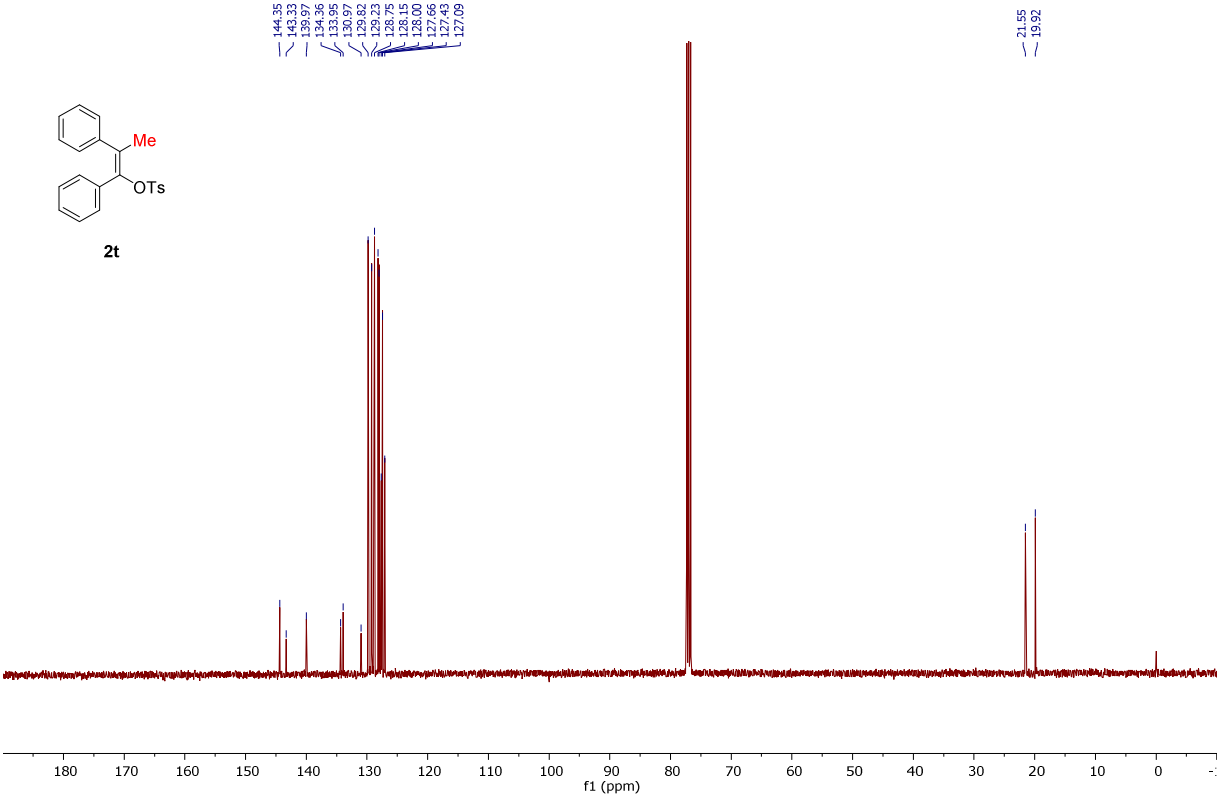
^{1}H NMR (400 MHz, Chloroform-d) δ 7.55 – 7.45 (m, 2H), 7.20 – 6.85 (m, 11H), 2.35 (s, 3H), 2.20 (s, 3H).



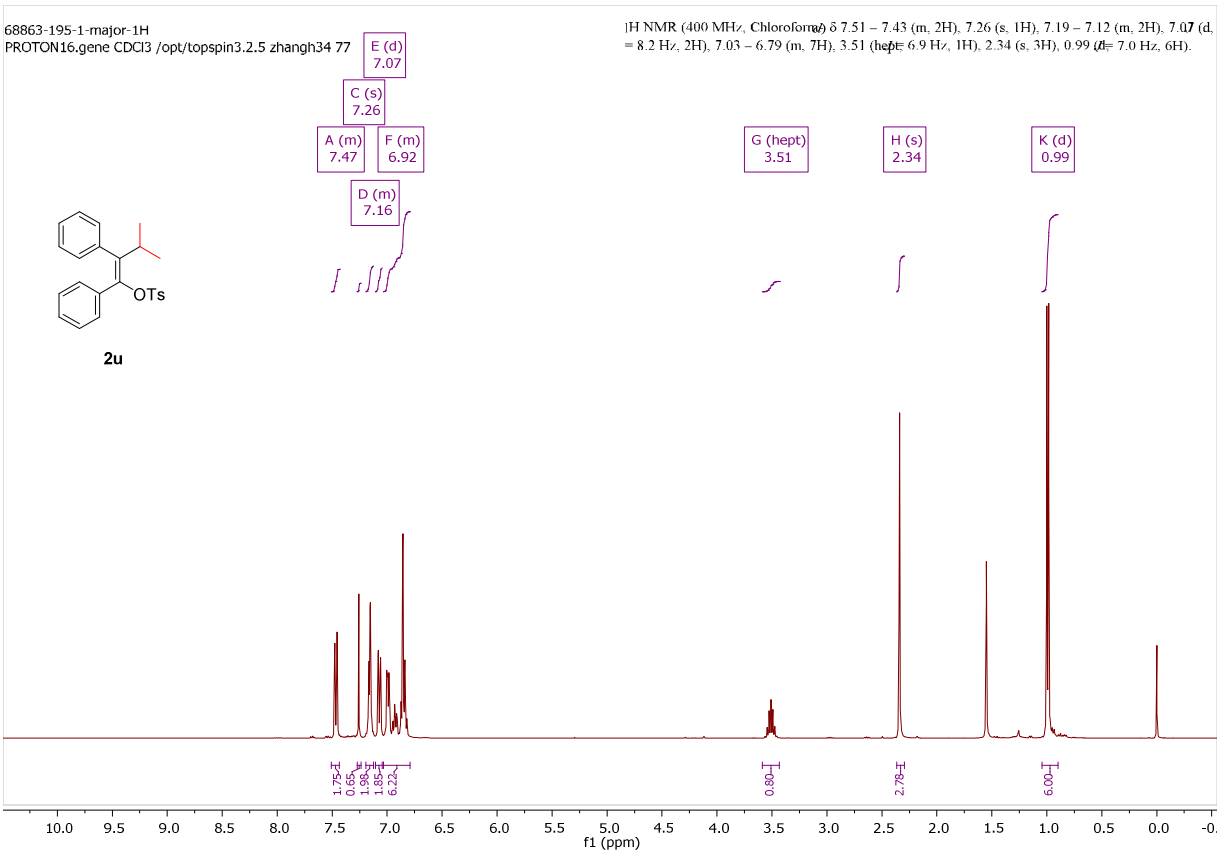
2t

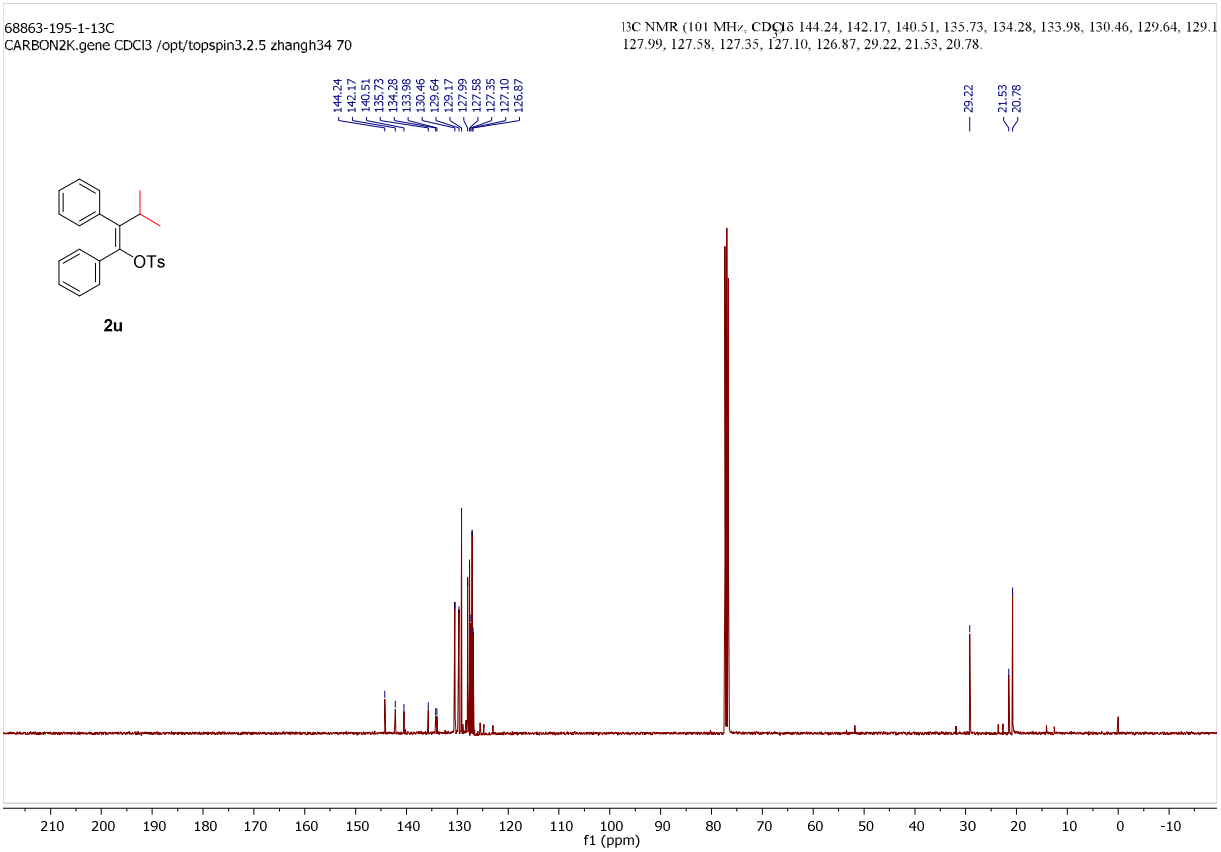
CARBON8K.gene CDCl3 /opt/topspin led6 17

^{13}C NMR (101 MHz, CDCl) δ 144.35, 143.33, 139.97, 134.36, 133.95, 130.97, 129.82, 129.23, 128.7, 128.15, 128.00, 127.66, 127.43, 127.09, 21.55, 19.92.



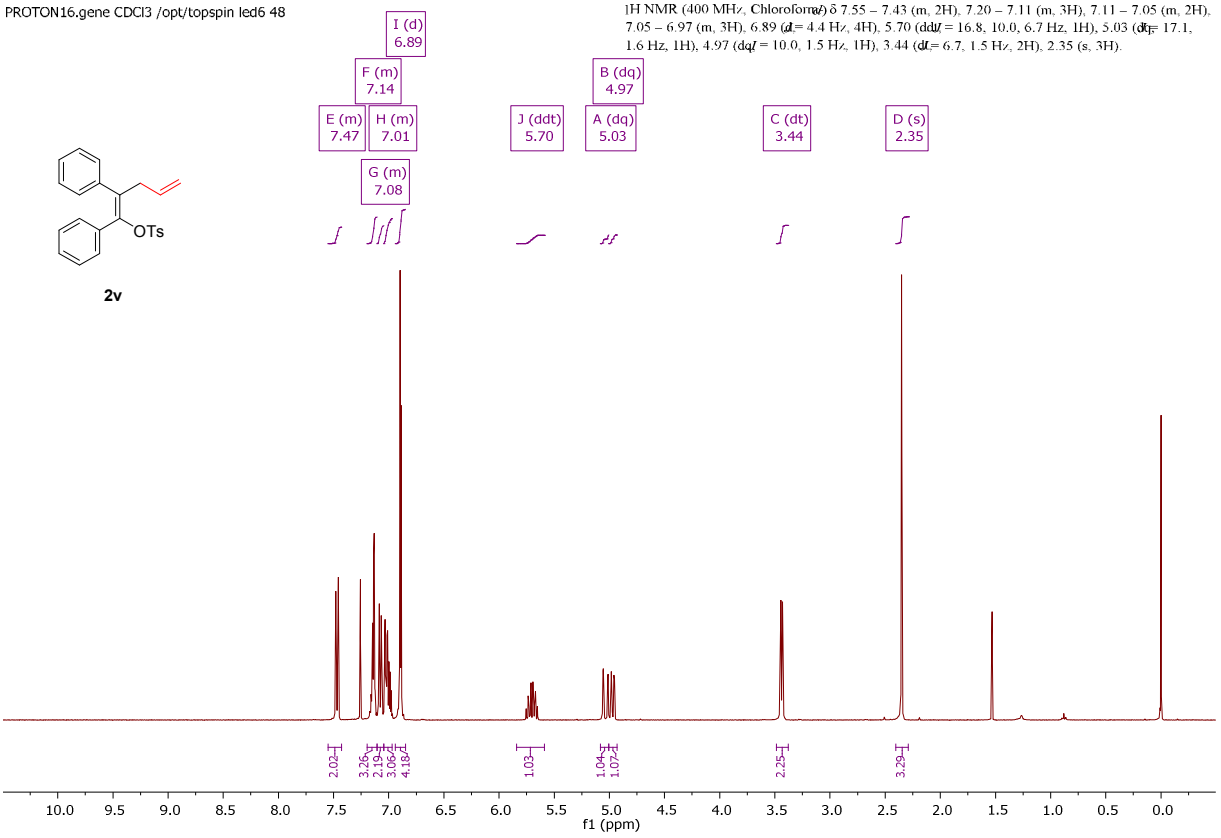
2t


68863-195-1-major-1H
PROTON16.gene CDCl3 /opt/topspin3.2.5 zhangh34 77
E (d) 7.07
C (s) 7.26
A (m) 7.47
F (m) 6.92
D (m) 7.16
G (hept) 3.51
H (s) 2.34
K (d) 0.99
OTs
2u
1.75
0.65
1.98
1.85
6.22
0.80
2.78
6.00
f1 (ppm)


68863-195-1-13C
CARBON2K.gene CDCl3 /opt/topspin3.2.5 zhangh34 70
144.24
142.17
140.51
135.73
134.28
133.98
130.46
129.64
129.17
127.99
127.58
127.35
127.10
126.87
29.22
21.53
20.78
OTs
2u
f1 (ppm)

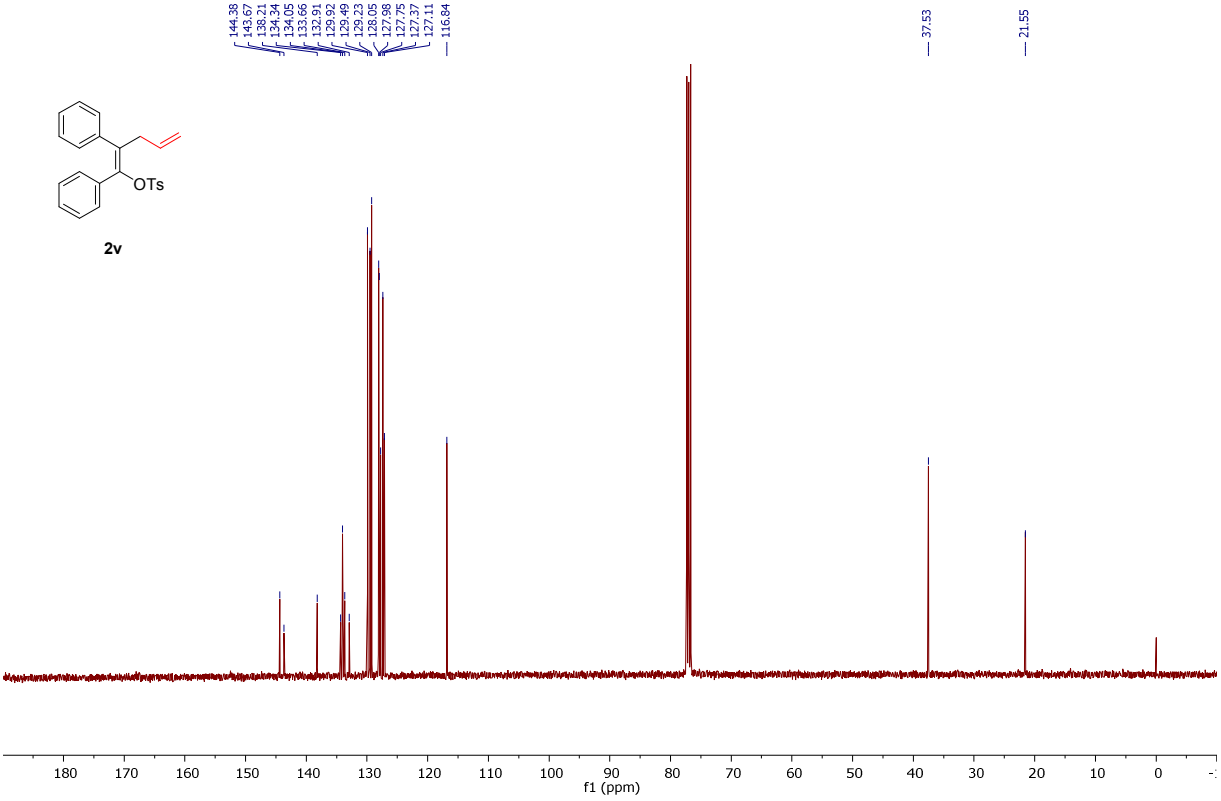
PROTON16.gene CDCl3 /opt/topspin led6 48

^{1}H NMR (400 MHz, Chloroform-d) 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 (ddd, 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).

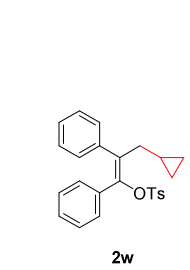


CARBON8K.gene CDCl3 /opt/topspin led6 48

^{13}C NMR (101 MHz, CDCl$_3$) δ 144.38, 143.67, 138.21, 134.34, 134.05, 133.66, 132.91, 129.92, 129.49, 129.23, 128.05, 127.98, 127.75, 127.37, 127.11, 116.84, 37.53, 21.55.



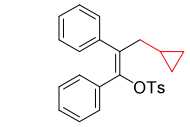
PROTON16.gene CDCl3 /opt/topspin led6 73



2w

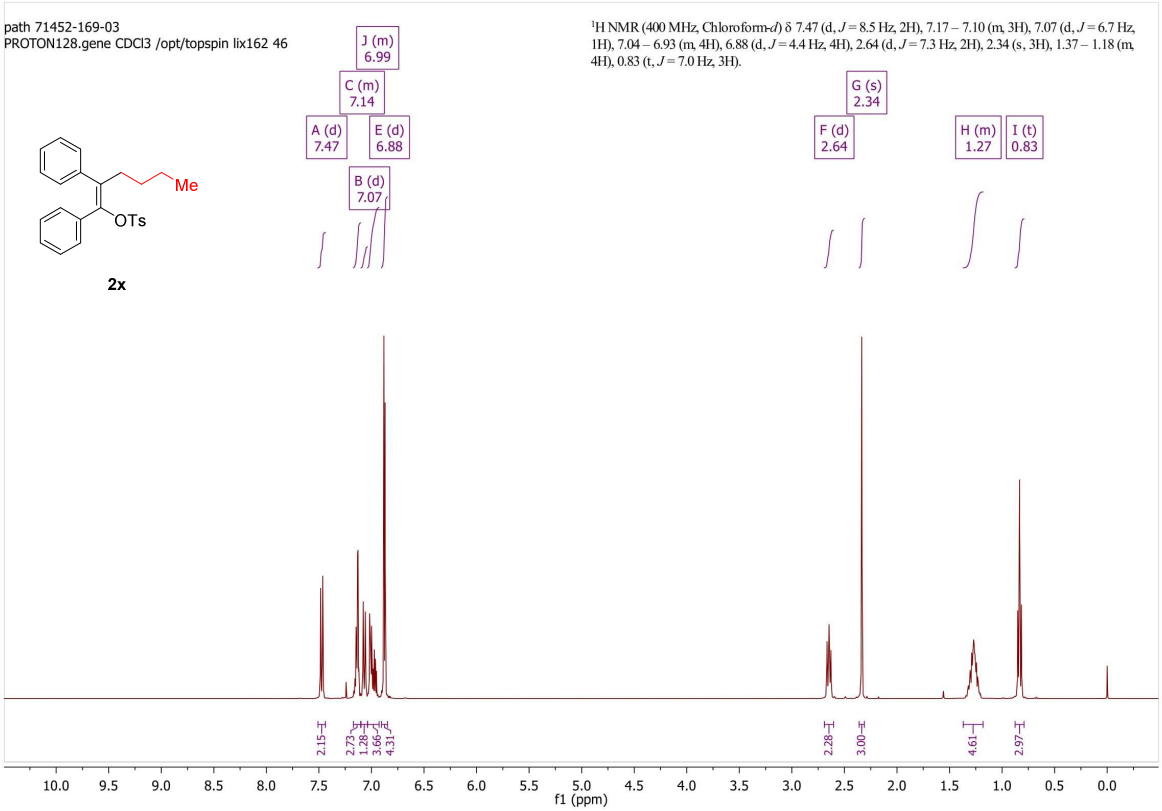
1H NMR (400 MHz, Chloroform-*d*) δ 7.51 – 7.42 (m, 2H), 7.22 – 7.12 (m, 3H), 7.12 – 7.03 (m, 4H), 7.03 – 6.93 (m, 1H), 6.88 (*d*, *J* = 4.4 Hz, 4H), 2.57 (*d*, *J* = 7.0 Hz, 2H), 2.35 (s, 3H), 0.71 – 0.55 (m, 1H) 0.40 – 0.27 (m, 2H), 0.13 – 0.03 (m, 2H).

CARBON8K.gene CDCl3 /opt/topspin led6 73

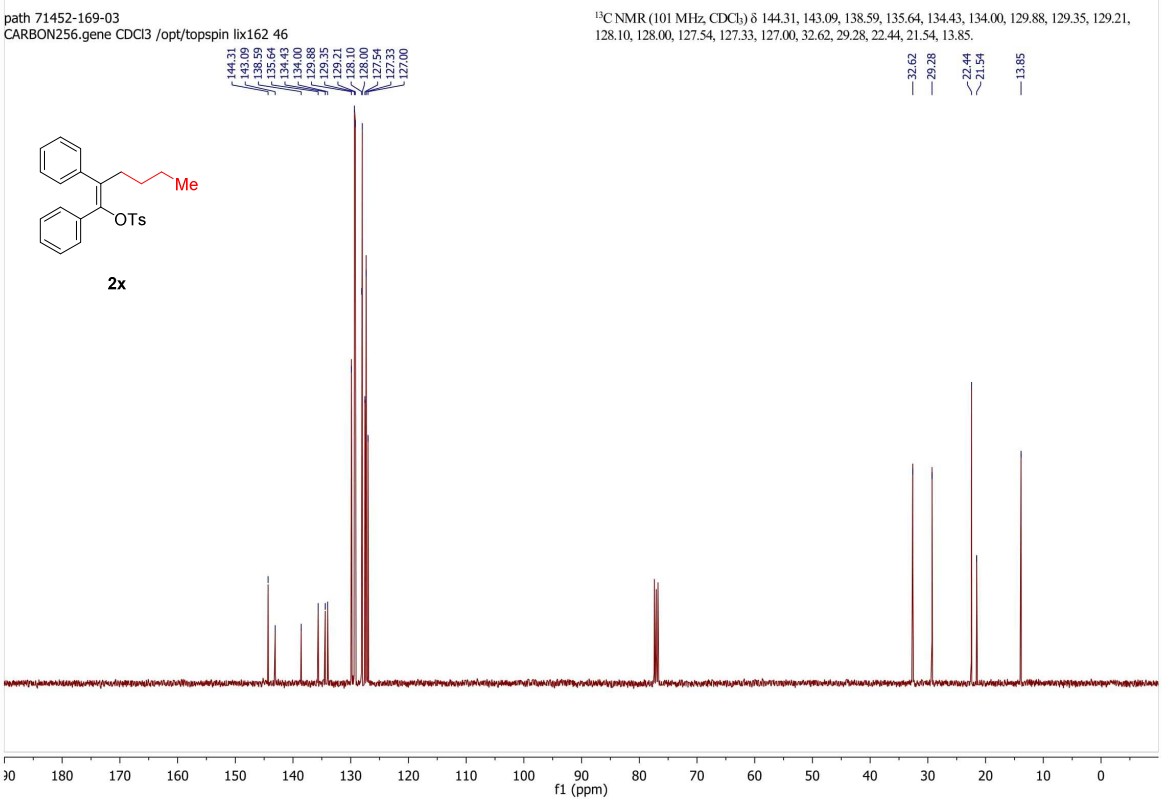


2w

13C NMR (101 MHz, CDCl3) δ 144.29, 142.81, 138.87, 135.49, 134.33, 133.85, 129.88, 129.45, 129.1 128.00, 127.53, 127.29, 126.93, 37.53, 21.55, 9.30, 4.58.



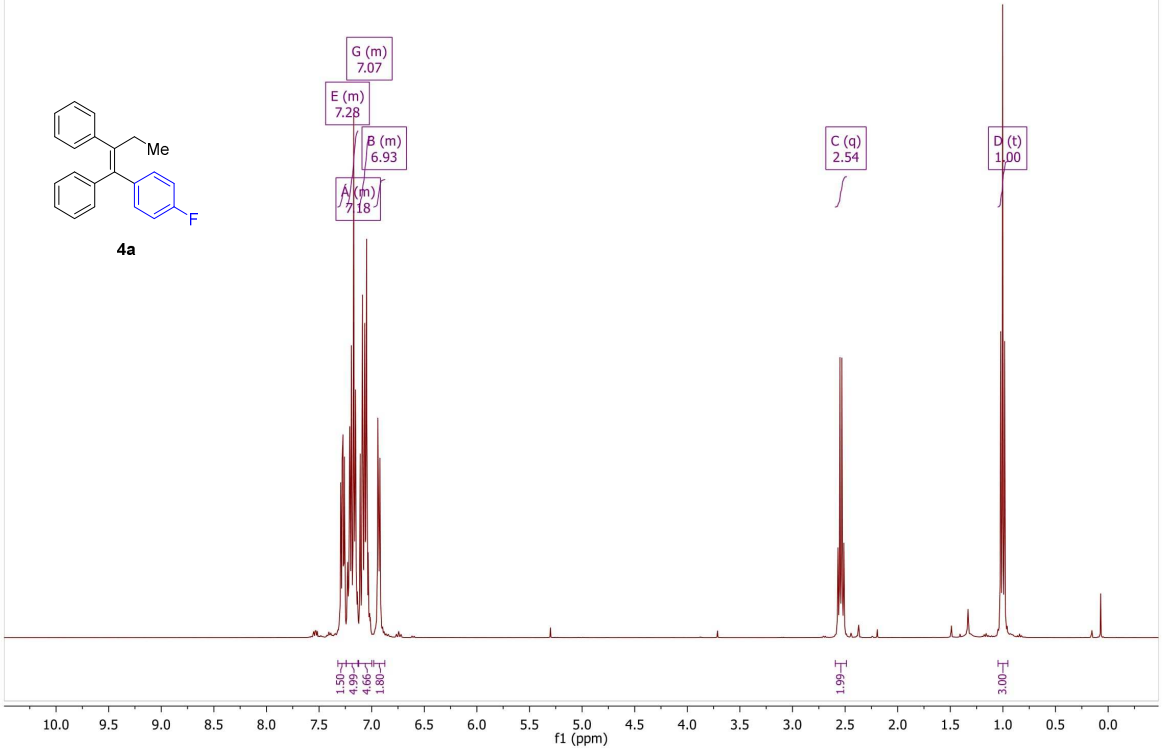
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 (d, *J* = 8.5 Hz, 2H), 7.17 – 7.10 (m, 3H), 7.07 (d, *J* = 6.7 Hz, 1H), 7.04 – 6.93 (m, 4H), 6.88 (d, *J* = 4.4 Hz, 4H), 2.64 (d, *J* = 7.3 Hz, 2H), 2.34 (s, 3H), 1.37 – 1.18 (m, 4H), 0.83 (t, *J* = 7.0 Hz, 3H).



^{13}C NMR (101 MHz, $CDCl_3$) δ 144.31, 143.09, 138.59, 135.64, 134.43, 134.00, 129.88, 129.35, 129.21, 128.10, 128.00, 127.54, 127.33, 127.00, 32.62, 29.28, 22.44, 21.54, 13.85.

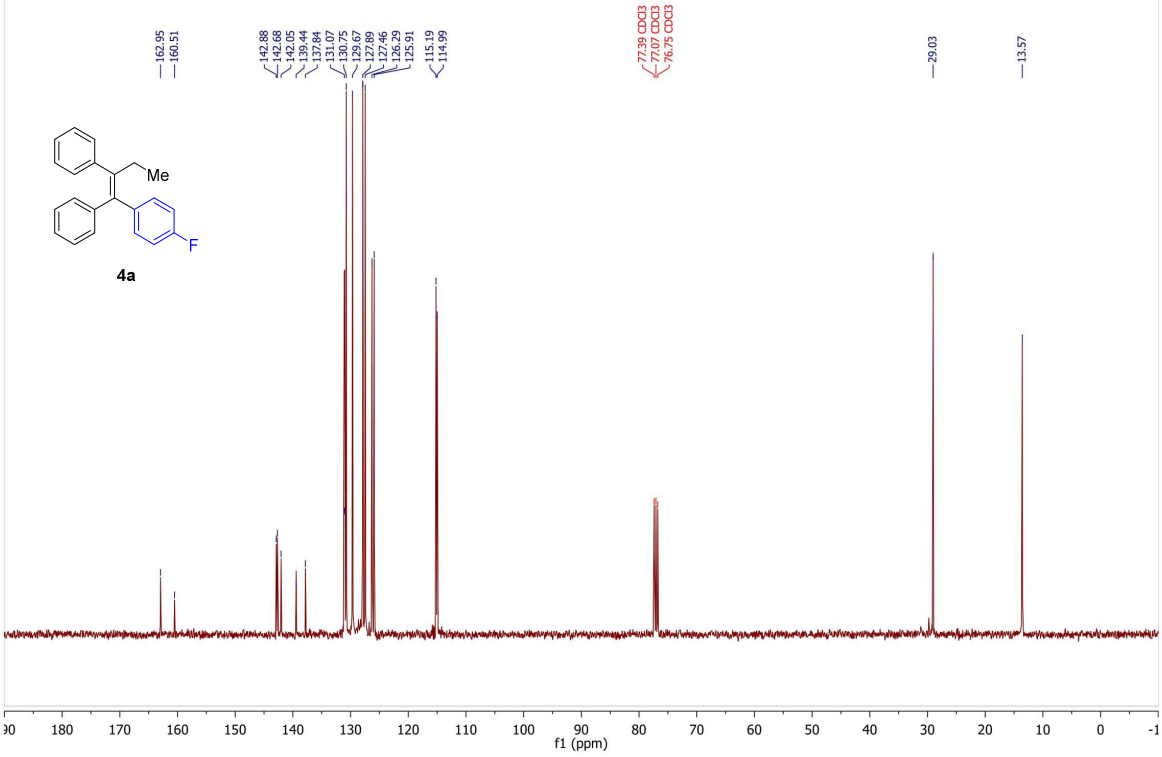
path 71452-112A-05
PROTON32.gene CDCl3 /opt/topspin lix162 35

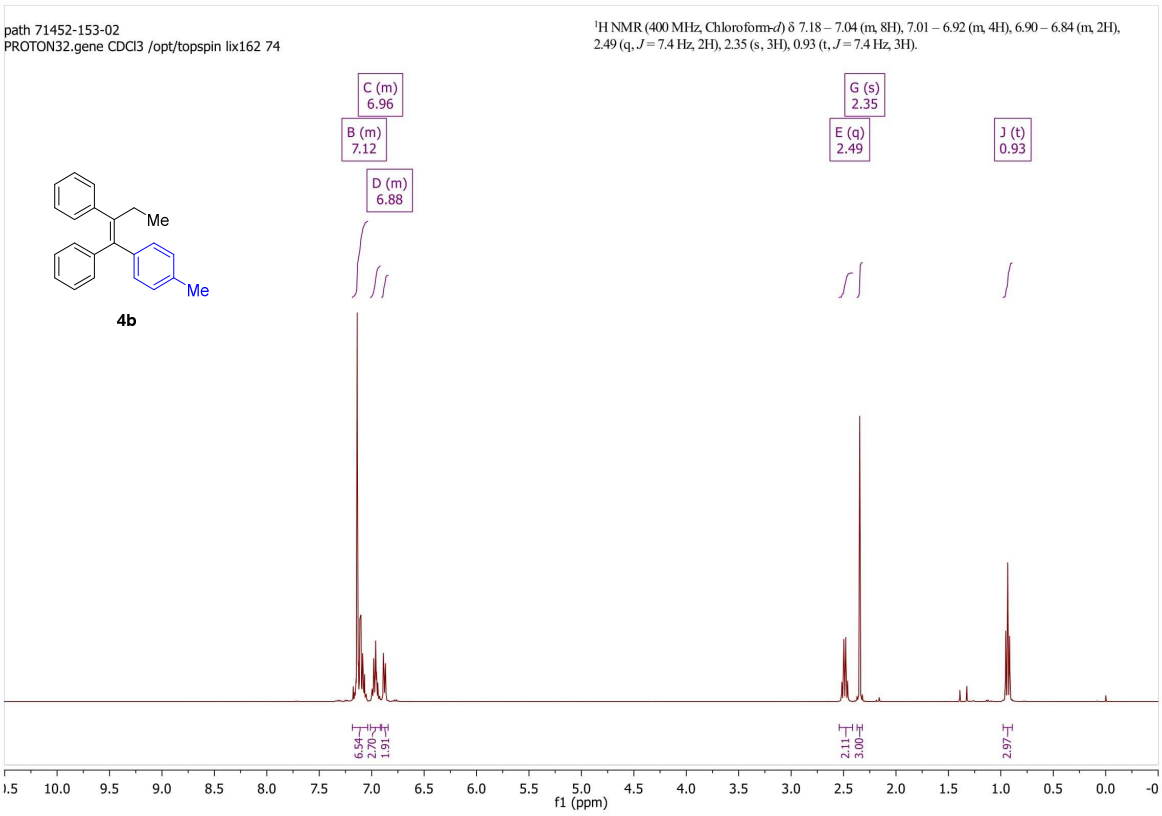
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32 – 7.24 (m, 2H), 7.24 – 7.13 (m, 5H), 7.12 – 7.00 (m, 5H), 6.98 – 6.88 (m, 2H), 2.54 (q, *J* = 7.5 Hz, 2H), 1.00 (t, *J* = 7.5 Hz, 3H).



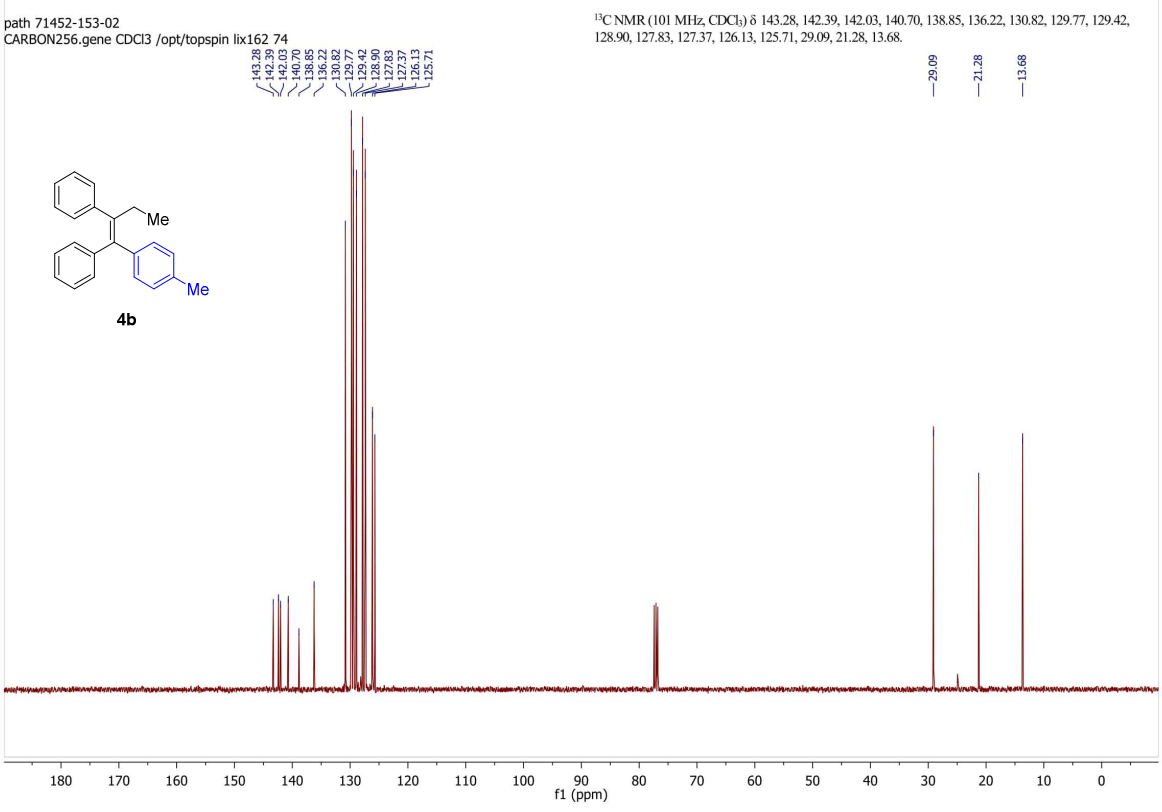
path 71452-112A-05
CARBON256.gene CDCl3 /opt/topspin lix162 35

^{13}C NMR (101 MHz, $CDCl_3$) δ 162.95, 160.51, 142.88, 142.68, 142.05, 139.44, 137.84, 131.07, 130.75, 129.67, 127.89, 127.46, 126.29, 125.91, 115.19, 114.99, 29.03, 13.57.

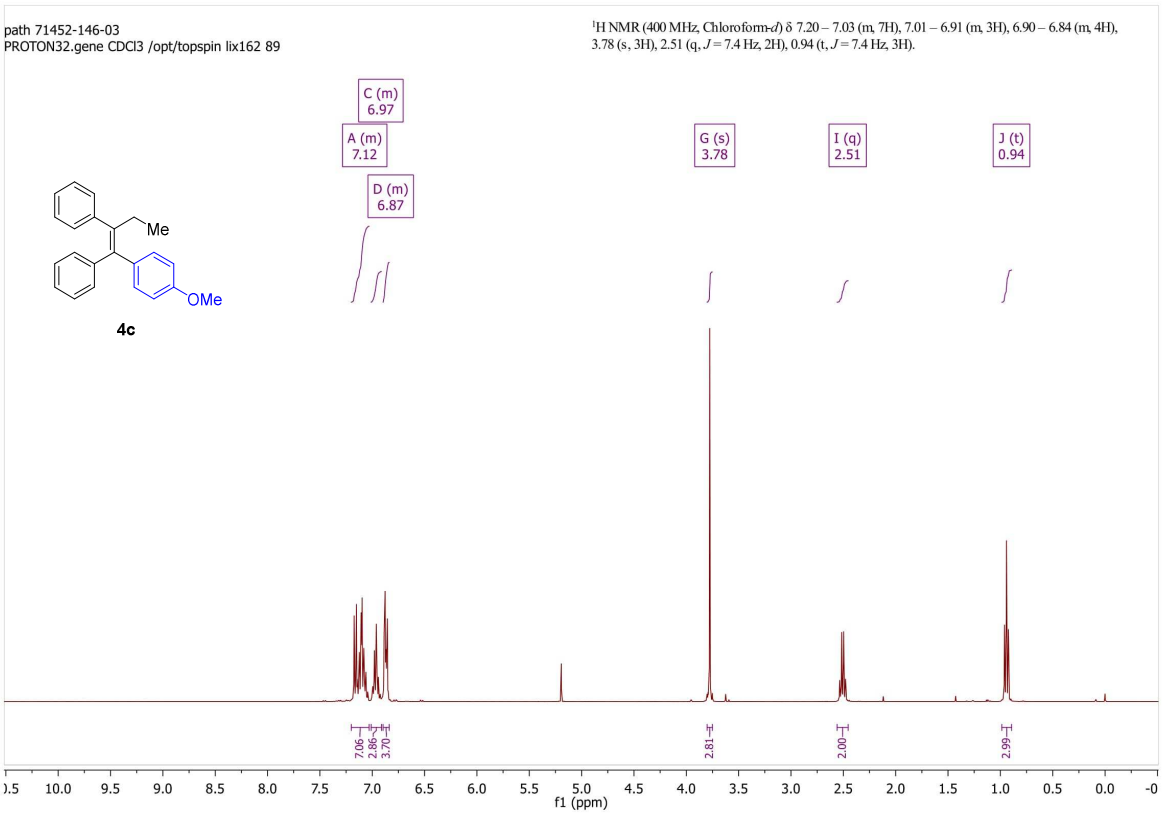




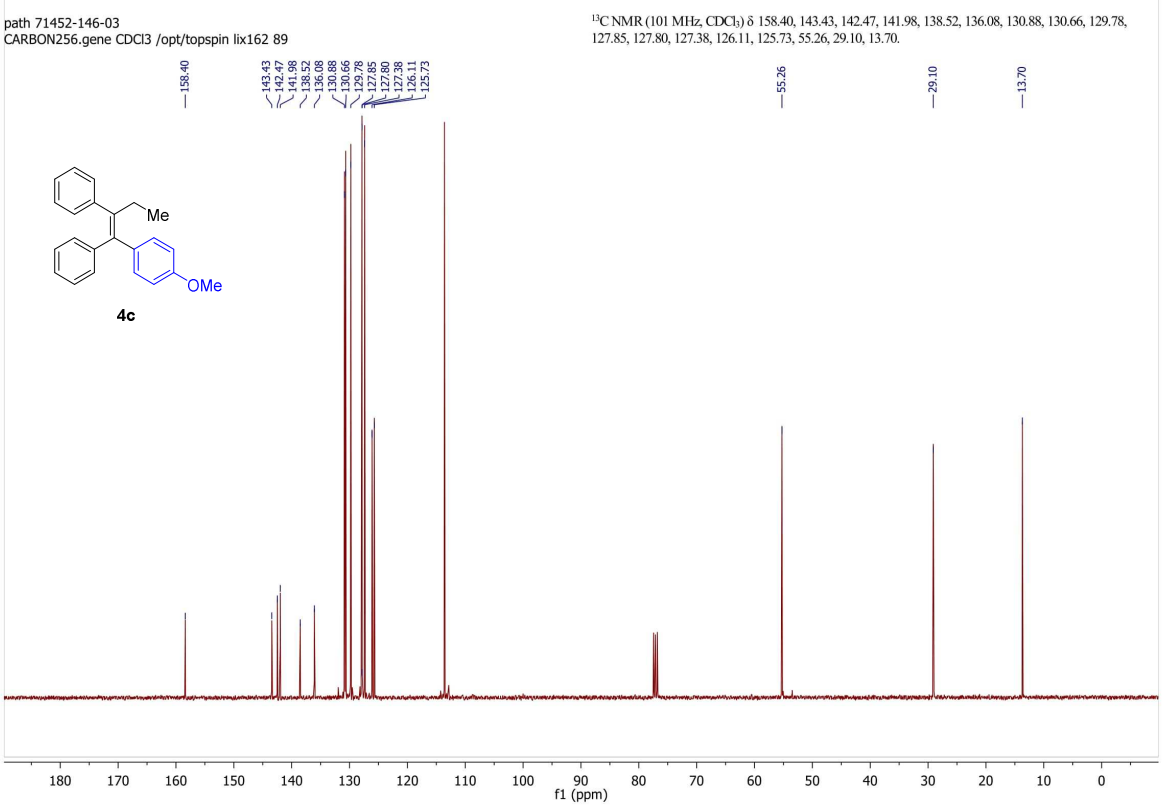
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.18 – 7.04 (m, 8H), 7.01 – 6.92 (m, 4H), 6.90 – 6.84 (m, 2H), 2.49 (q, *J* = 7.4 Hz, 2H), 2.35 (s, 3H), 0.93 (t, *J* = 7.4 Hz, 3H).



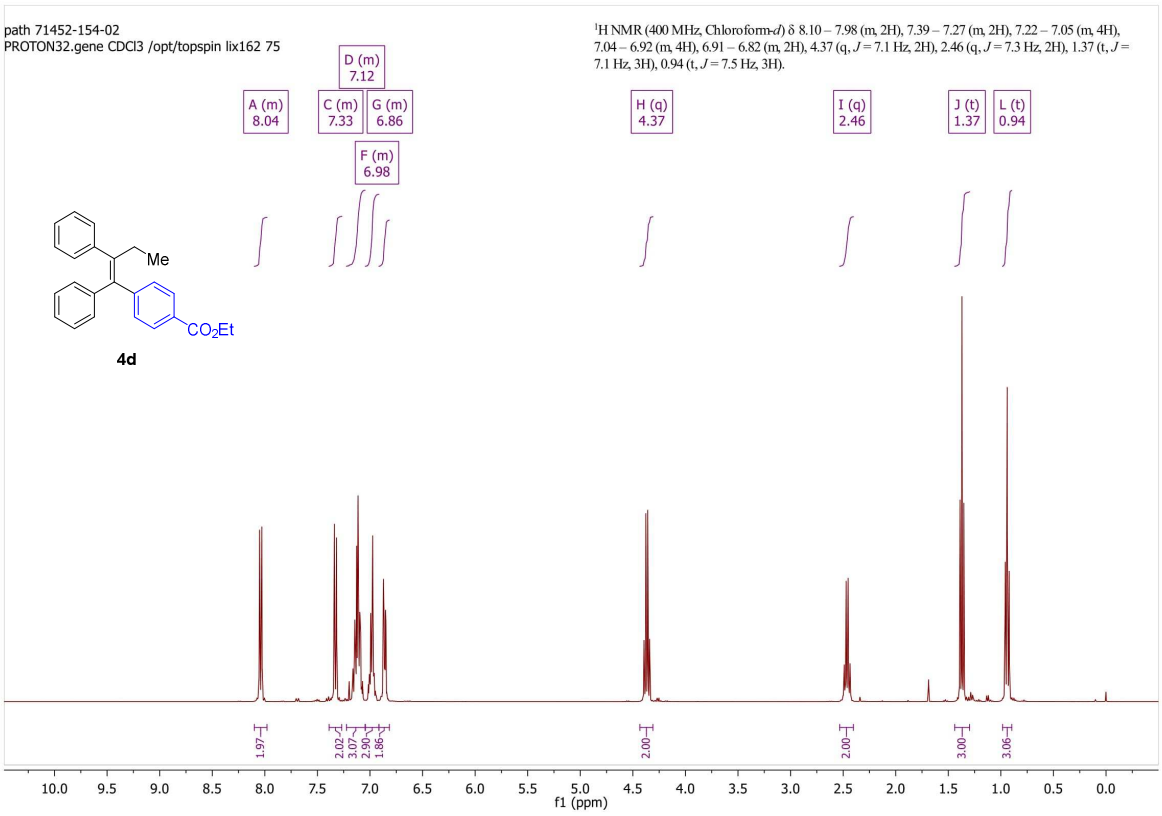
^{13}C NMR (101 MHz, $CDCl_3$) δ 143.28, 142.39, 142.03, 140.70, 138.85, 136.22, 130.82, 129.77, 129.42, 128.90, 127.83, 127.37, 126.13, 125.71, 29.09, 21.28, 13.68.



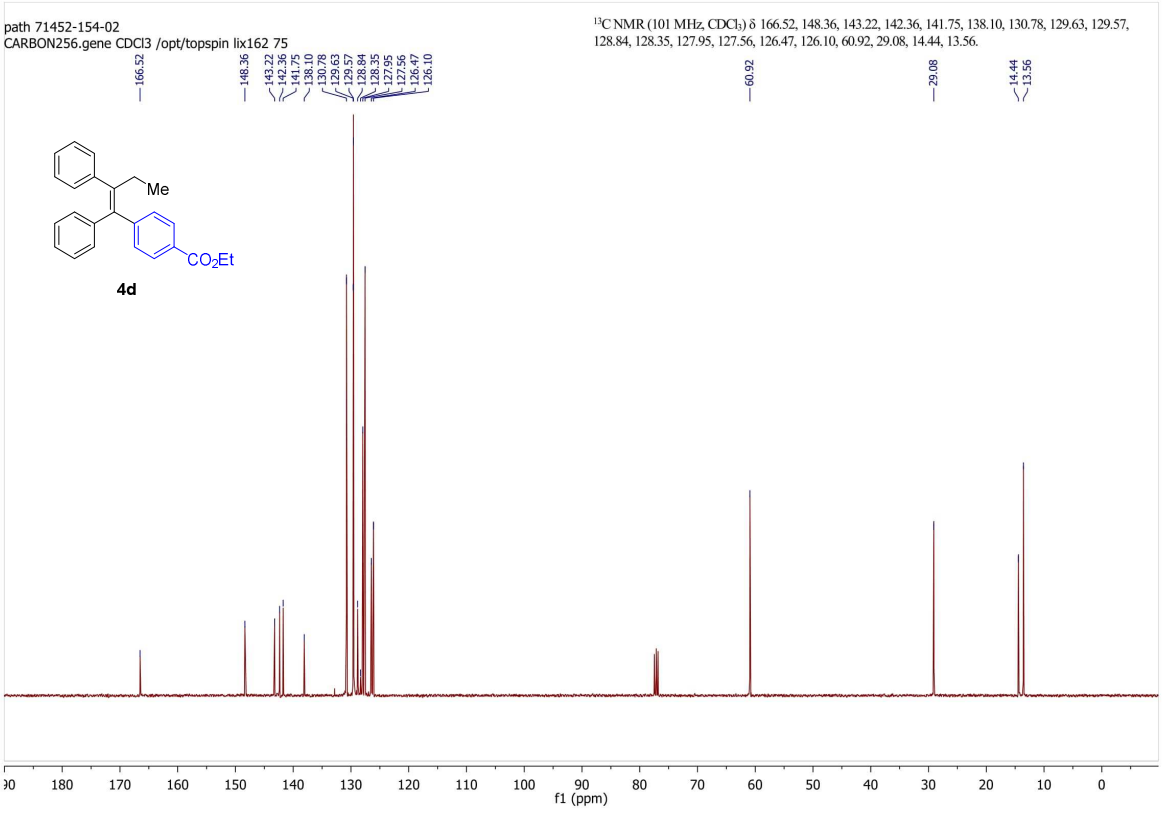
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.20 – 7.03 (m, 7H), 7.01 – 6.91 (m, 3H), 6.90 – 6.84 (m, 4H), 3.78 (s, 3H), 2.51 (q, *J* = 7.4 Hz, 2H), 0.94 (t, *J* = 7.4 Hz, 3H).



^{13}C NMR (101 MHz, $CDCl_3$) δ 158.40, 143.43, 142.47, 141.98, 138.52, 136.08, 130.88, 130.66, 129.78, 127.85, 127.80, 127.38, 126.11, 125.73, 55.26, 29.10, 13.70.



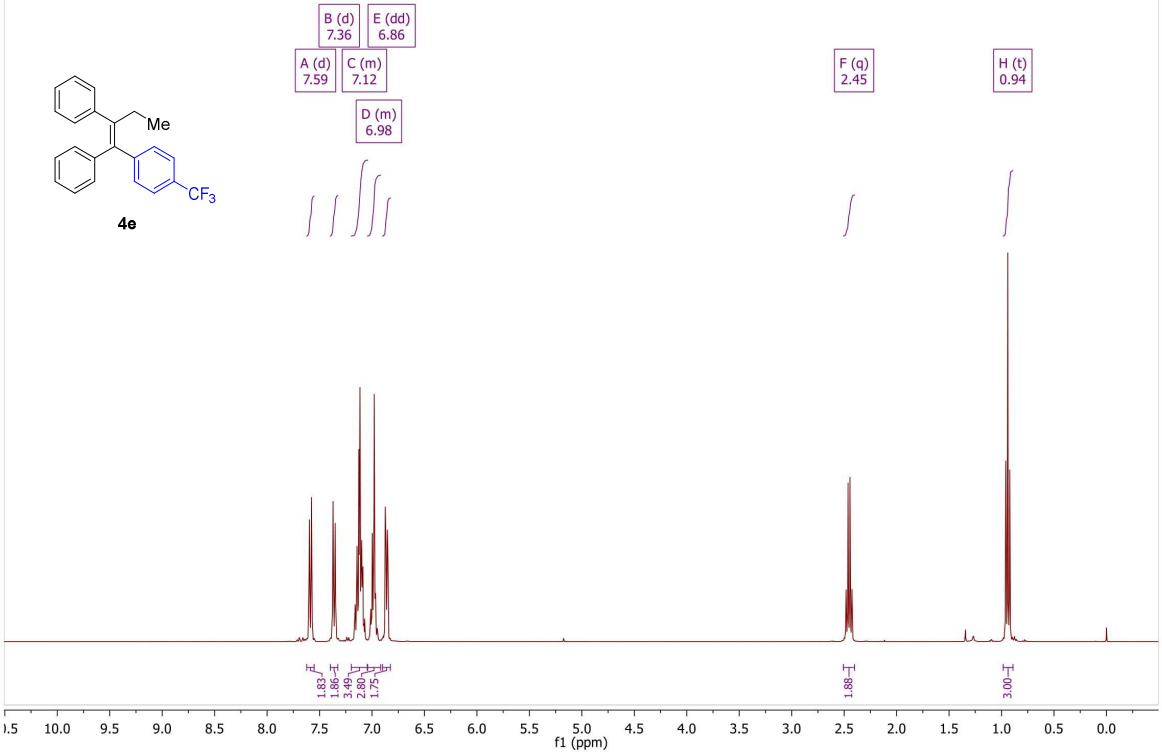
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.10 – 7.98 (m, 2H), 7.39 – 7.27 (m, 2H), 7.22 – 7.05 (m, 4H), 7.04 – 6.92 (m, 4H), 6.91 – 6.82 (m, 2H), 4.37 (q, *J* = 7.1 Hz, 2H), 2.46 (q, *J* = 7.3 Hz, 2H), 1.37 (t, *J* = 7.1 Hz, 3H), 0.94 (t, *J* = 7.5 Hz, 3H).



^{13}C NMR (101 MHz, $CDCl_3$) 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, 60.92, 29.08, 14.44, 13.56.

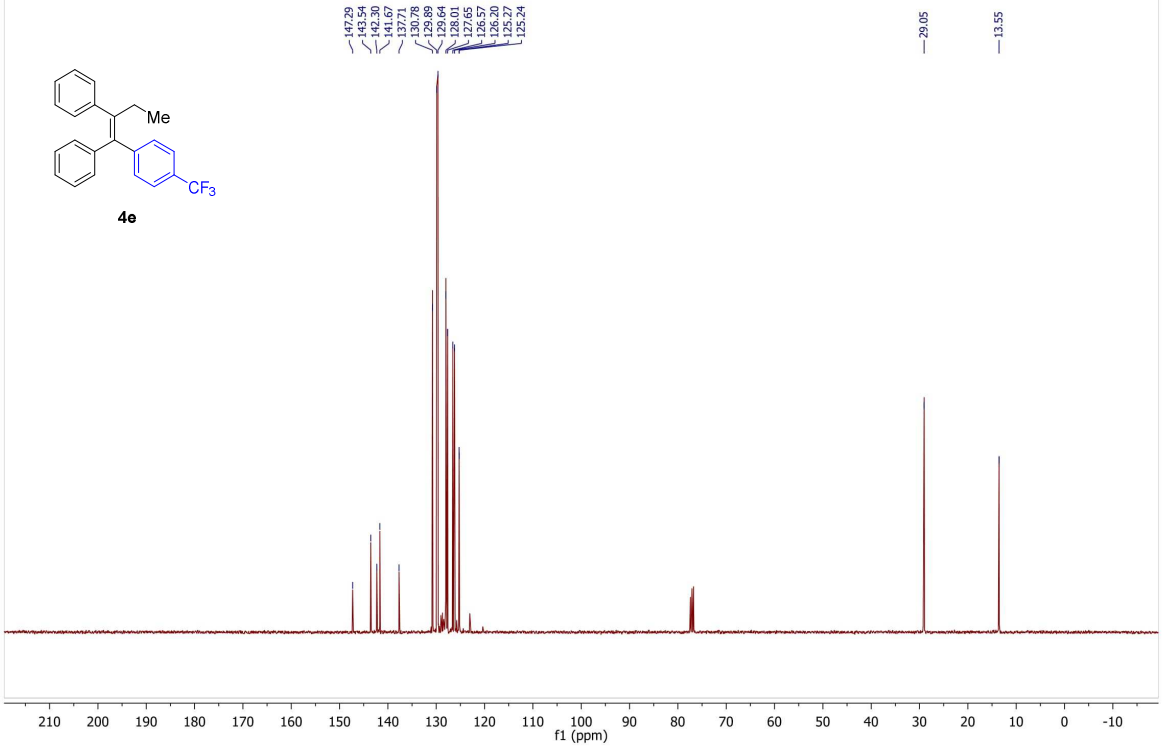
71452-162-03
PROTON32.gene CDCl3 /opt/topspin lix162 71

^{1}H NMR (400 MHz, Chloroform-*d*) δ 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), 6.86 (dd, *J* = 7.6, 2.0 Hz, 2H), 2.45 (q, *J* = 7.4 Hz, 2H), 0.94 (t, *J* = 7.5 Hz, 3H).



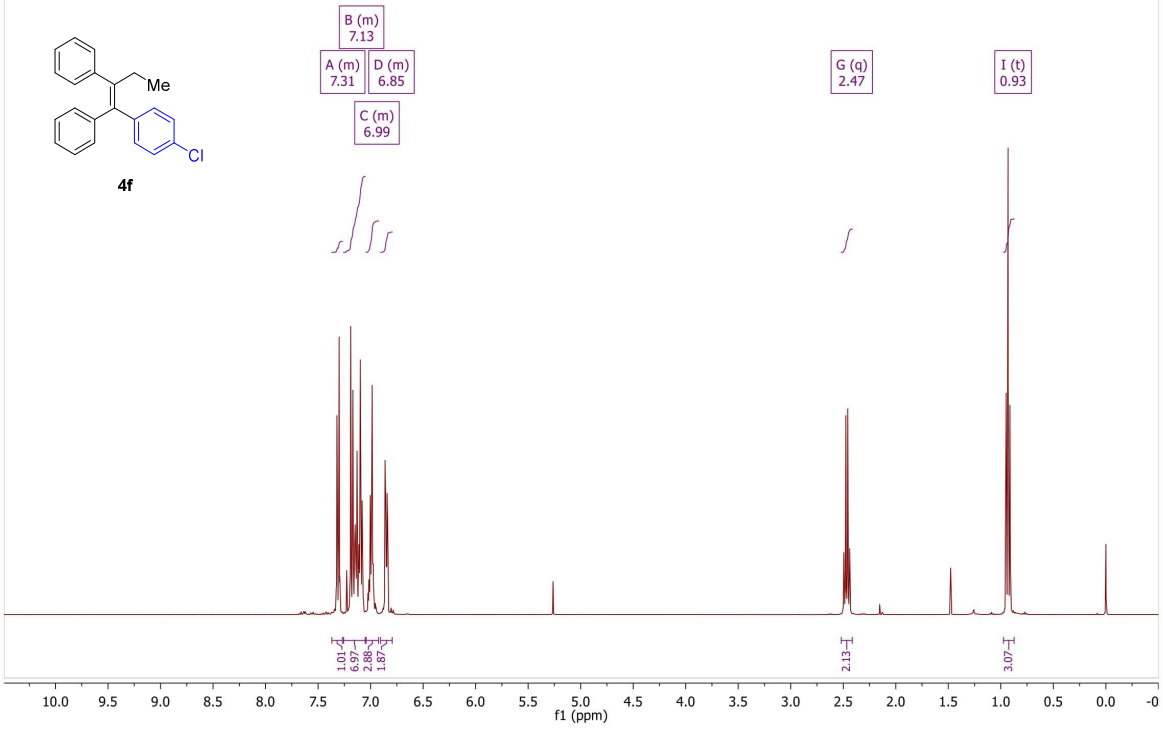
71452-162-03
CARBON256.gene CDCl3 /opt/topspin lix162 71

^{13}C NMR (101 MHz, $CDCl_3$) δ 147.29, 143.54, 142.30, 141.67, 137.71, 130.78, 129.89, 129.64, 128.01, 127.65, 126.57, 126.20, 125.27, 125.24, 29.05, 13.55.



path 71452-163A-05
PROTON128.gene CDCl3 /opt/topspin lix162 32

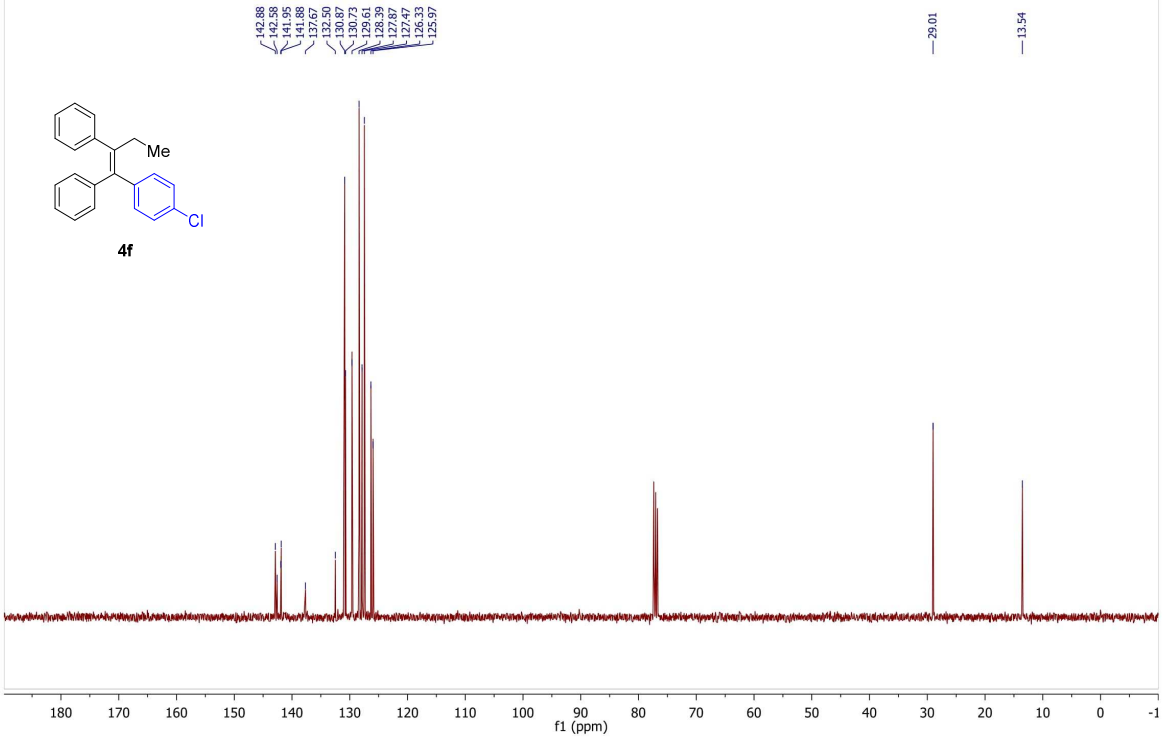
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 – 7.27 (m, 1H), 7.26 – 7.05 (m, 8H), 7.04 – 6.92 (m, 3H), 6.90 – 6.79 (m, 2H), 2.47 (q, *J* = 7.4 Hz, 2H), 0.93 (t, *J* = 7.4 Hz, 3H).



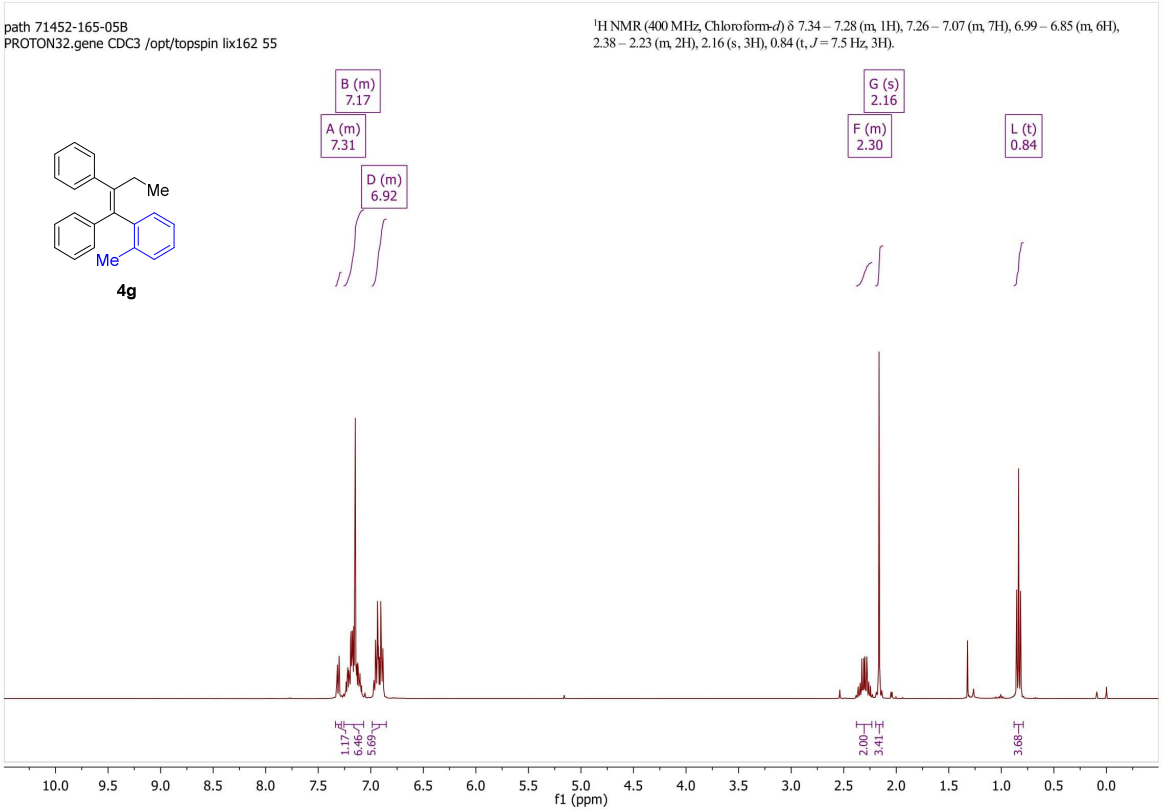
4f

path 71452-163A-05
CARBON256.gene CDCl3 /opt/topspin lix162 32

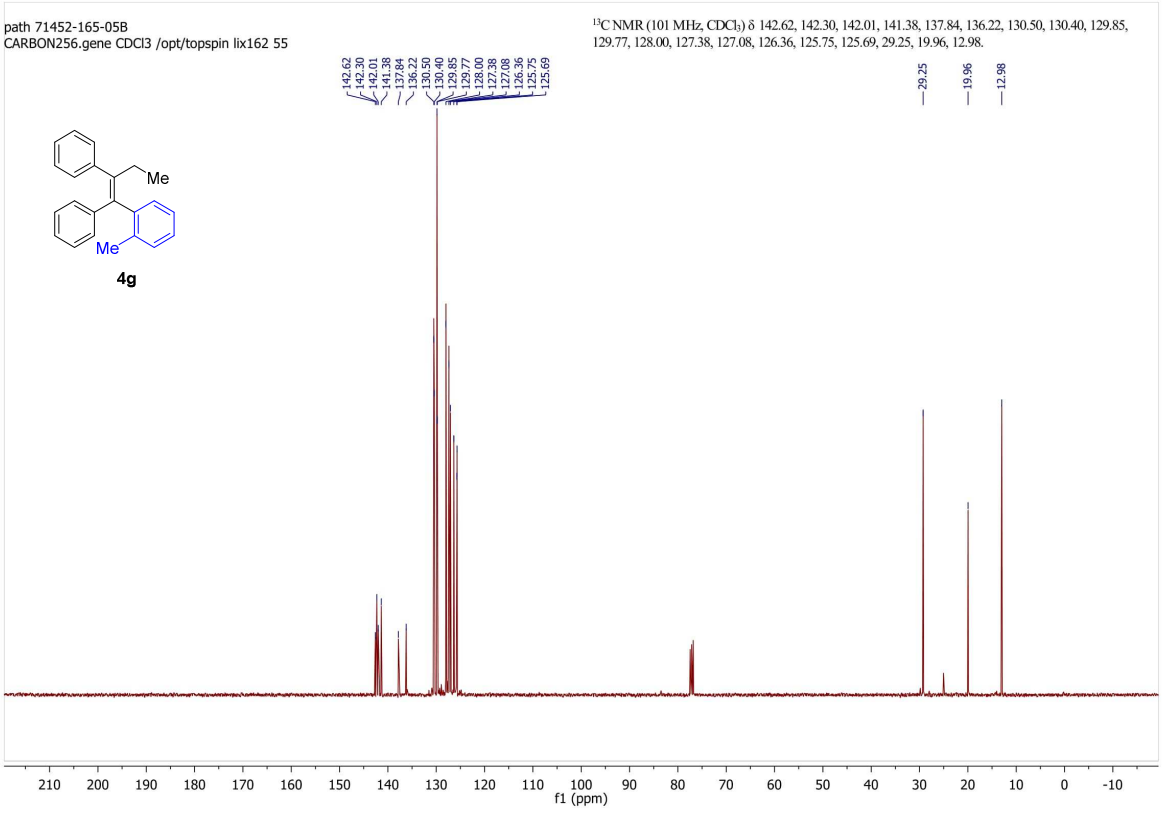
^{13}C NMR (101 MHz, $CDCl_3$) δ 142.88, 142.58, 141.95, 141.88, 137.67, 132.50, 130.87, 130.73, 129.61, 128.39, 127.87, 127.47, 126.33, 125.97, 29.01, 13.54.



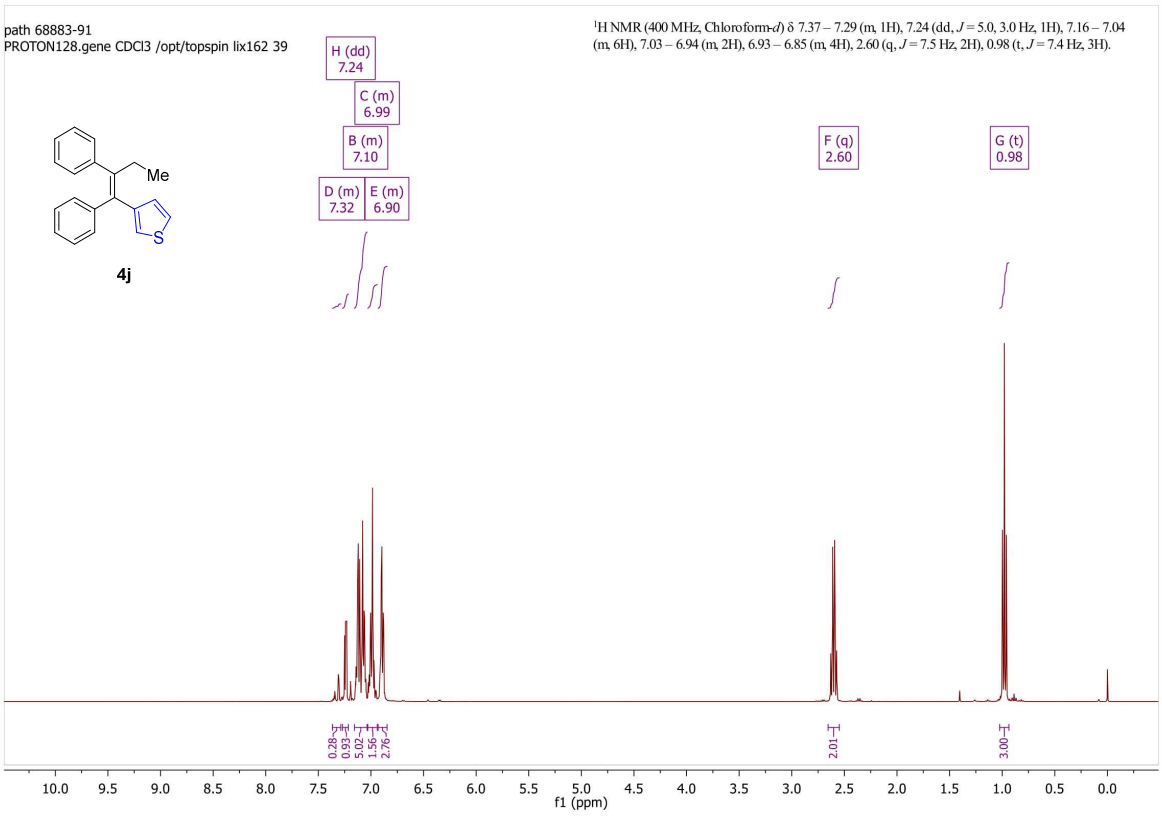
4f



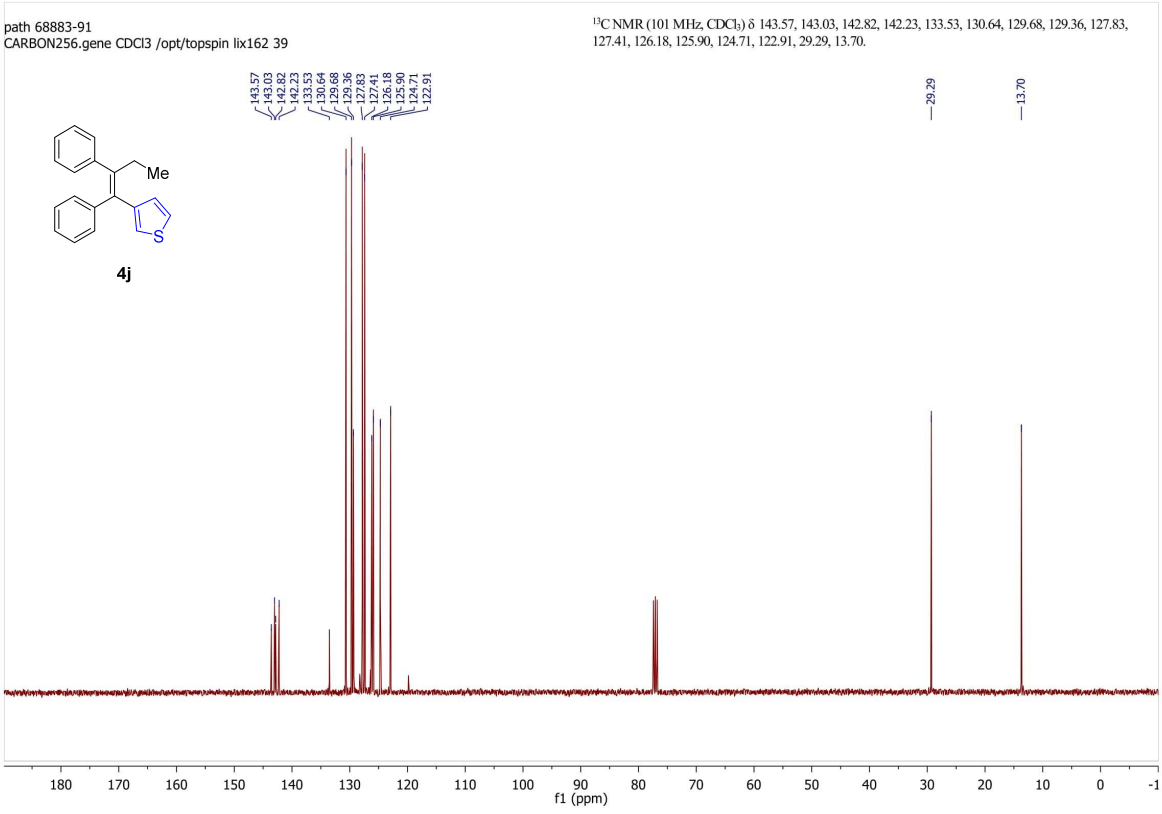
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 – 7.28 (m, 1H), 7.26 – 7.07 (m, 7H), 6.99 – 6.85 (m, 6H), 2.38 – 2.23 (m, 2H), 2.16 (s, 3H), 0.84 (t, *J* = 7.5 Hz, 3H).



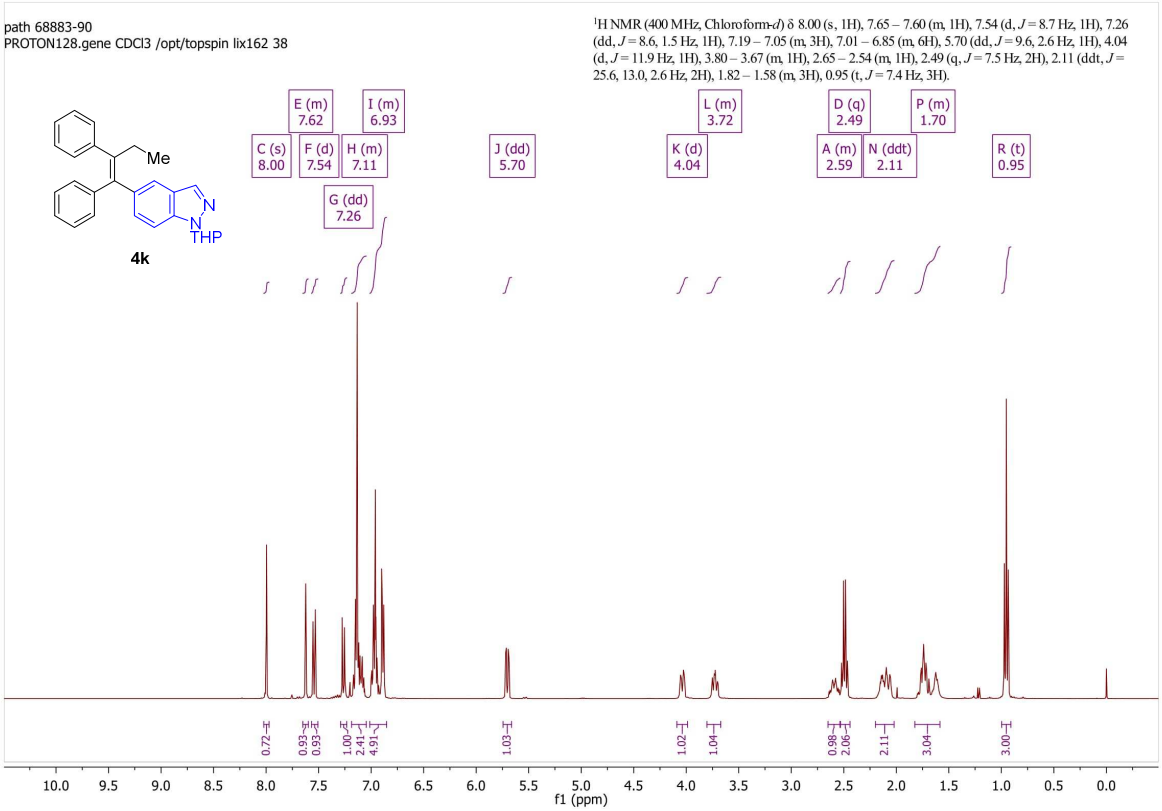
^{13}C NMR (101 MHz, $CDCl_3$) δ 142.62, 142.30, 142.01, 141.38, 137.84, 136.22, 130.50, 130.40, 129.85, 129.77, 128.00, 127.38, 127.08, 126.36, 125.75, 125.69, 29.25, 19.96, 12.98.



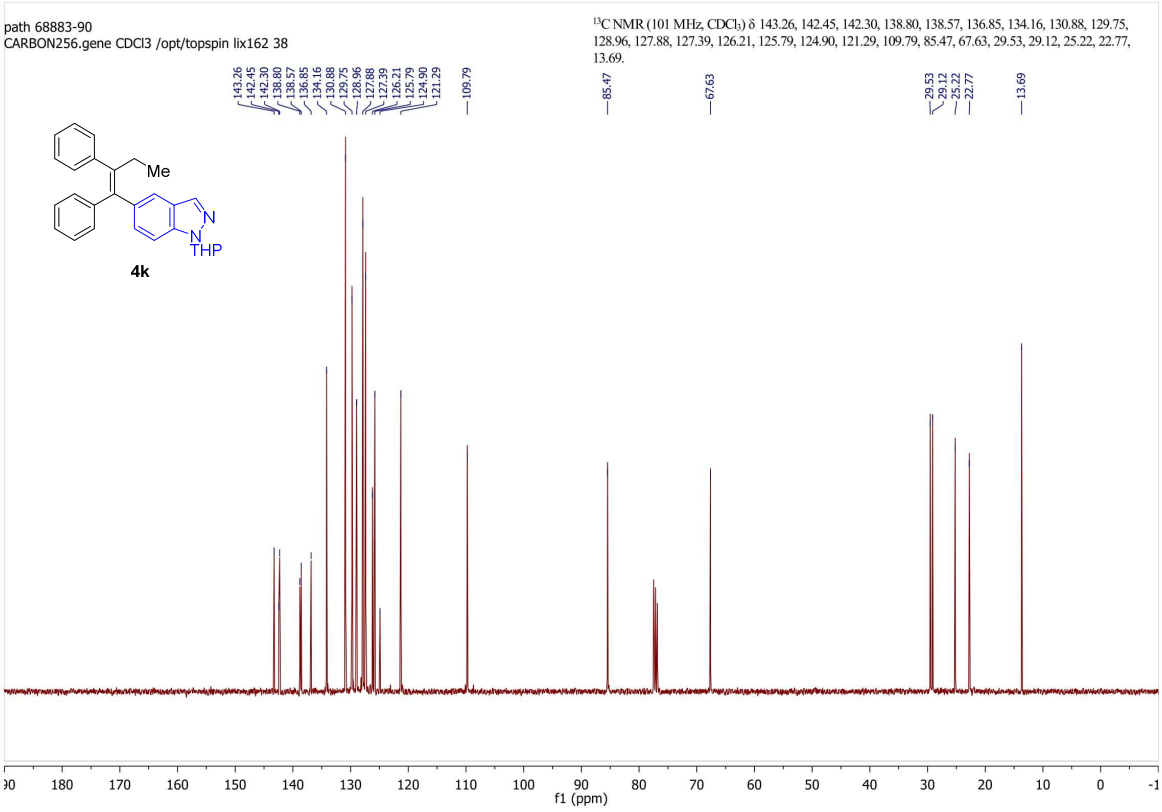
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 – 7.29 (m, 1H), 7.24 (dd, *J* = 5.0, 3.0 Hz, 1H), 7.16 – 7.04 (m, 6H), 7.03 – 6.94 (m, 2H), 6.93 – 6.85 (m, 4H), 2.60 (q, *J* = 7.5 Hz, 2H), 0.98 (t, *J* = 7.4 Hz, 3H).



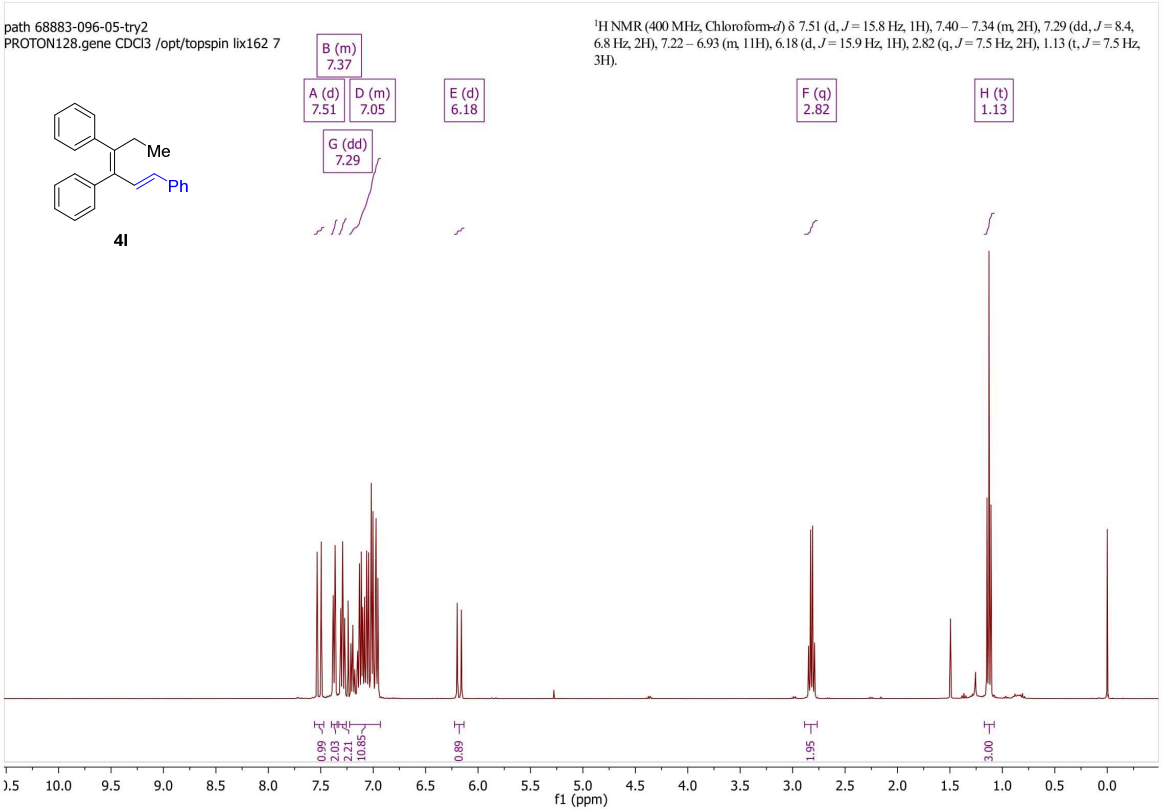
^{13}C NMR (101 MHz, $CDCl_3$) δ 143.57, 143.03, 142.82, 142.23, 133.53, 130.64, 129.68, 129.36, 127.83, 127.41, 126.18, 125.90, 124.71, 122.91, 29.29, 13.70.



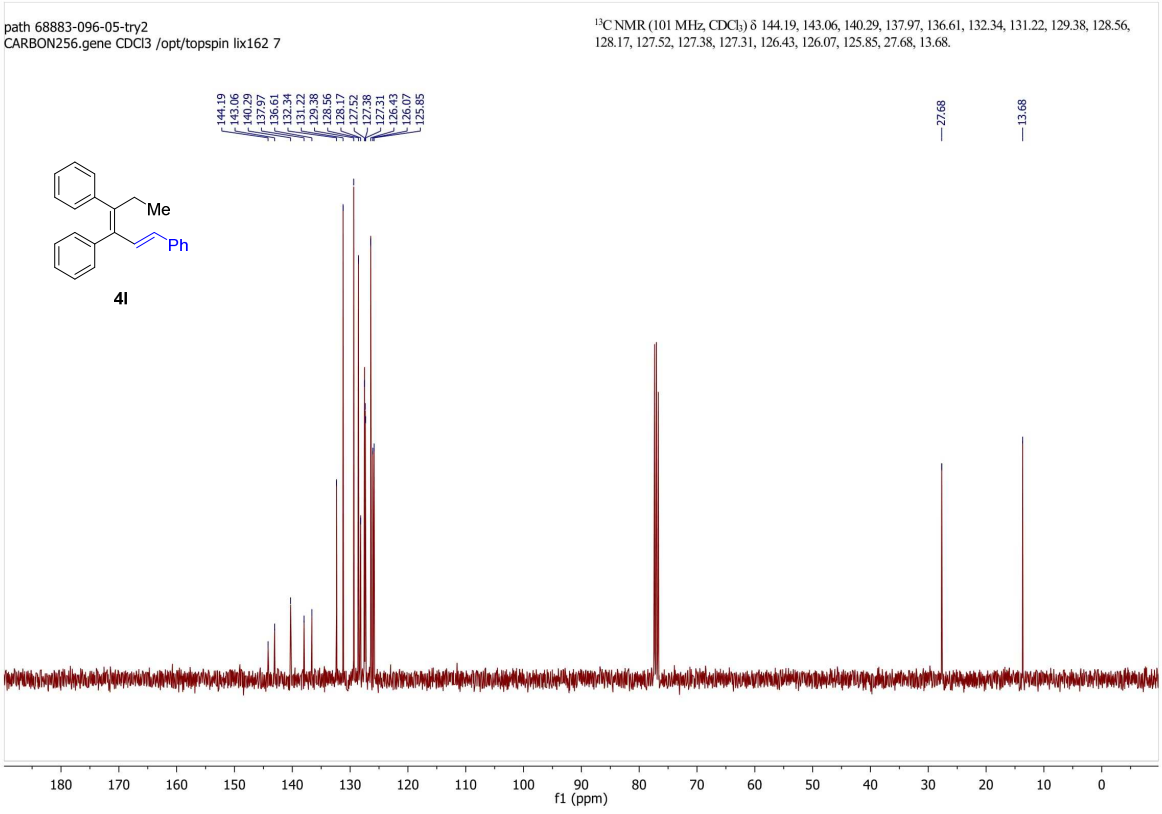
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.00 (s, 1H), 7.65 – 7.60 (m, 1H), 7.54 (d, *J* = 8.7 Hz, 1H), 7.26 (dd, *J* = 8.6, 1.5 Hz, 1H), 7.19 – 7.05 (m, 3H), 7.01 – 6.85 (m, 6H), 5.70 (dd, *J* = 9.6, 2.6 Hz, 1H), 4.04 (d, *J* = 11.9 Hz, 1H), 3.80 – 3.67 (m, 1H), 2.65 – 2.54 (m, 1H), 2.49 (q, *J* = 7.5 Hz, 2H), 2.11 (ddt, *J* = 25.6, 13.0, 2.6 Hz, 2H), 1.82 – 1.58 (m, 3H), 0.95 (t, *J* = 7.4 Hz, 3H).



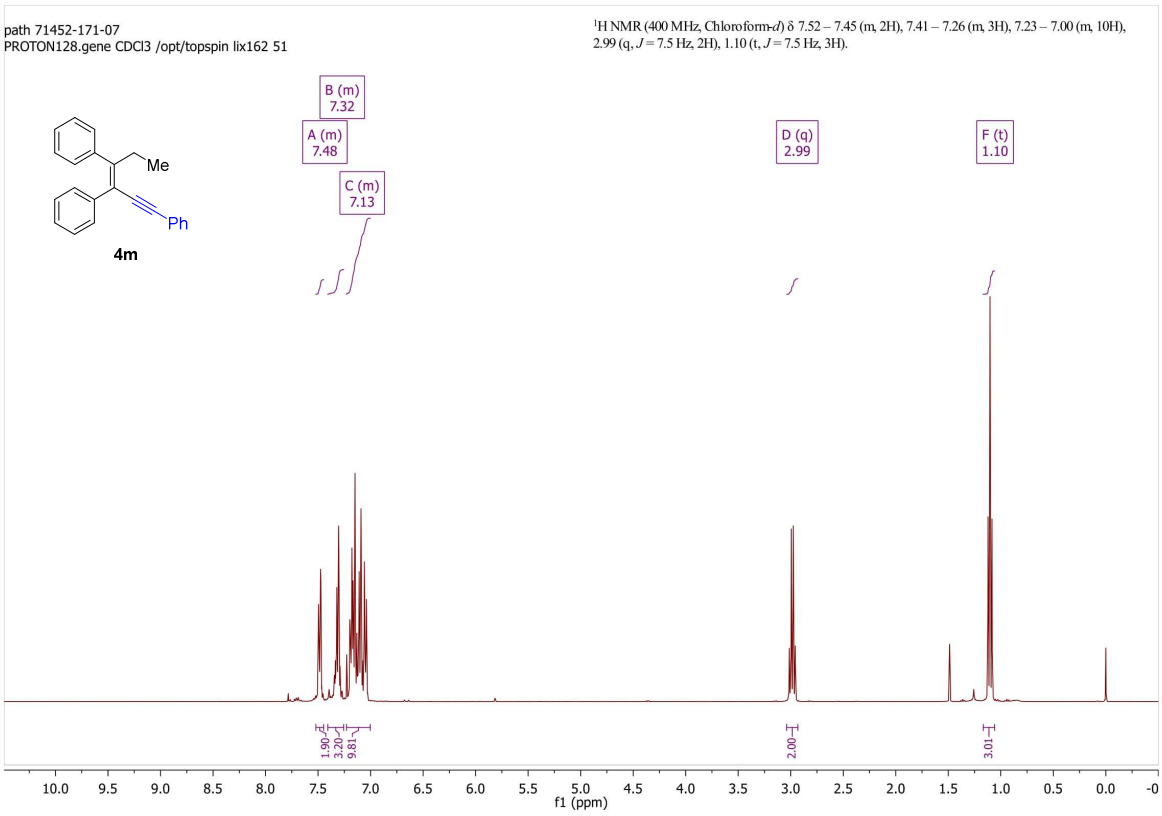
^{13}C NMR (101 MHz, $CDCl_3$) δ 143.26, 142.45, 142.30, 138.80, 138.57, 136.85, 134.16, 130.88, 129.75, 128.96, 127.88, 127.39, 126.21, 125.79, 124.90, 121.29, 109.79, 85.47, 67.63, 29.53, 29.12, 25.22, 22.77, 13.69.



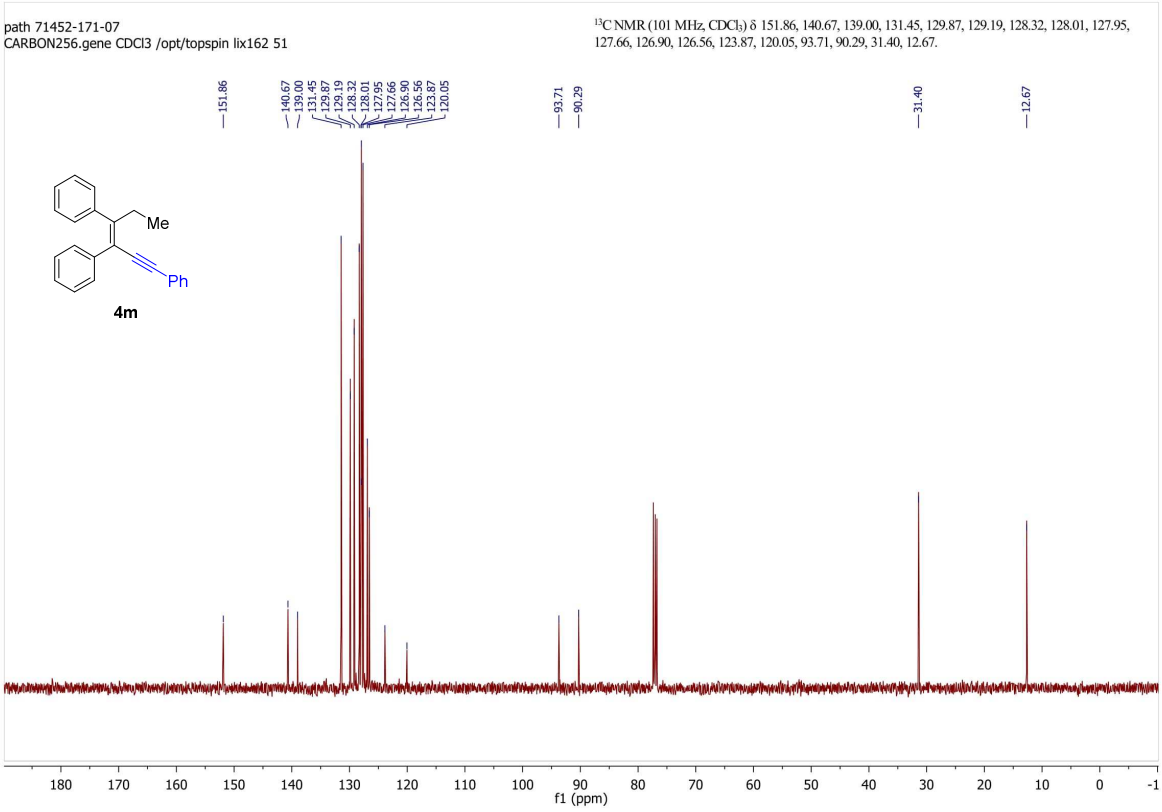
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.51 (d, *J* = 15.8 Hz, 1H), 7.40 – 7.34 (m, 2H), 7.29 (dd, *J* = 8.4, 6.8 Hz, 2H), 7.22 – 6.93 (m, 11H), 6.18 (d, *J* = 15.9 Hz, 1H), 2.82 (q, *J* = 7.5 Hz, 2H), 1.13 (t, *J* = 7.5 Hz, 3H).



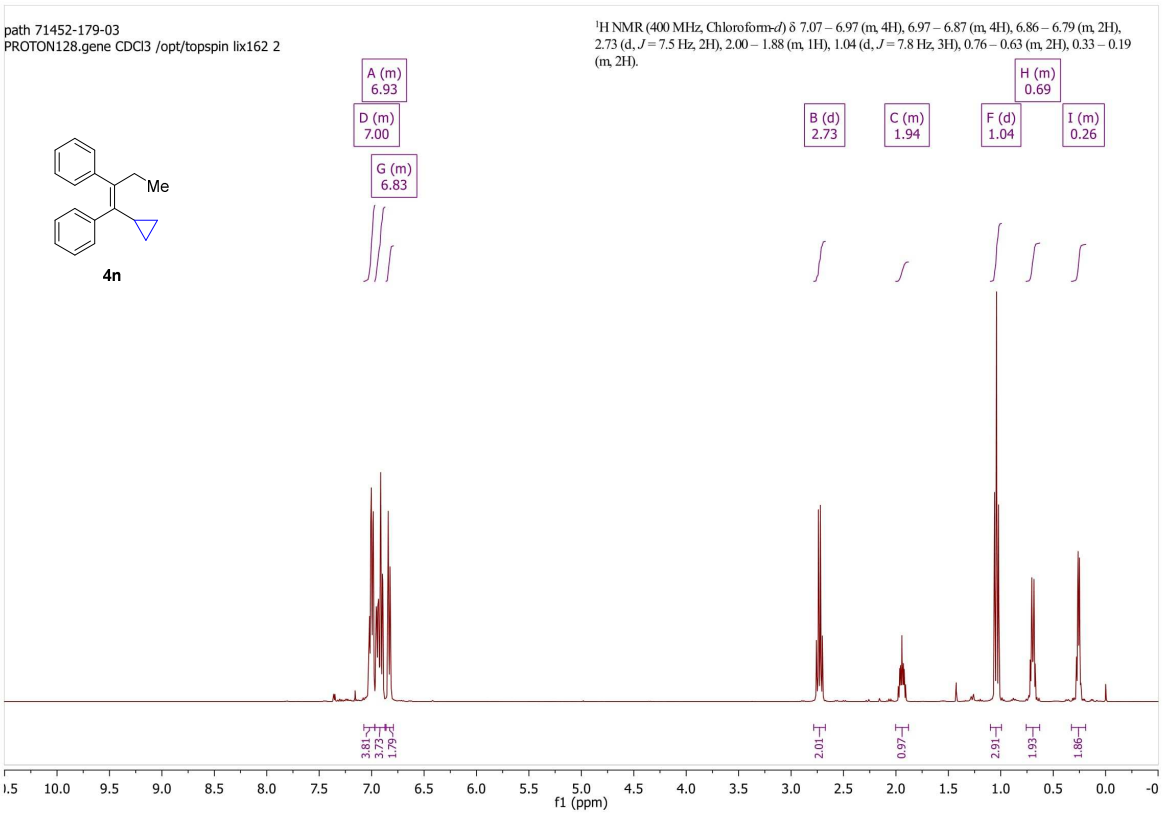
^{13}C NMR (101 MHz, $CDCl_3$) δ 144.19, 143.06, 140.29, 137.97, 136.61, 132.34, 131.22, 129.38, 128.56, 128.17, 127.52, 127.38, 127.31, 126.43, 126.07, 125.85, 27.68, 13.68.



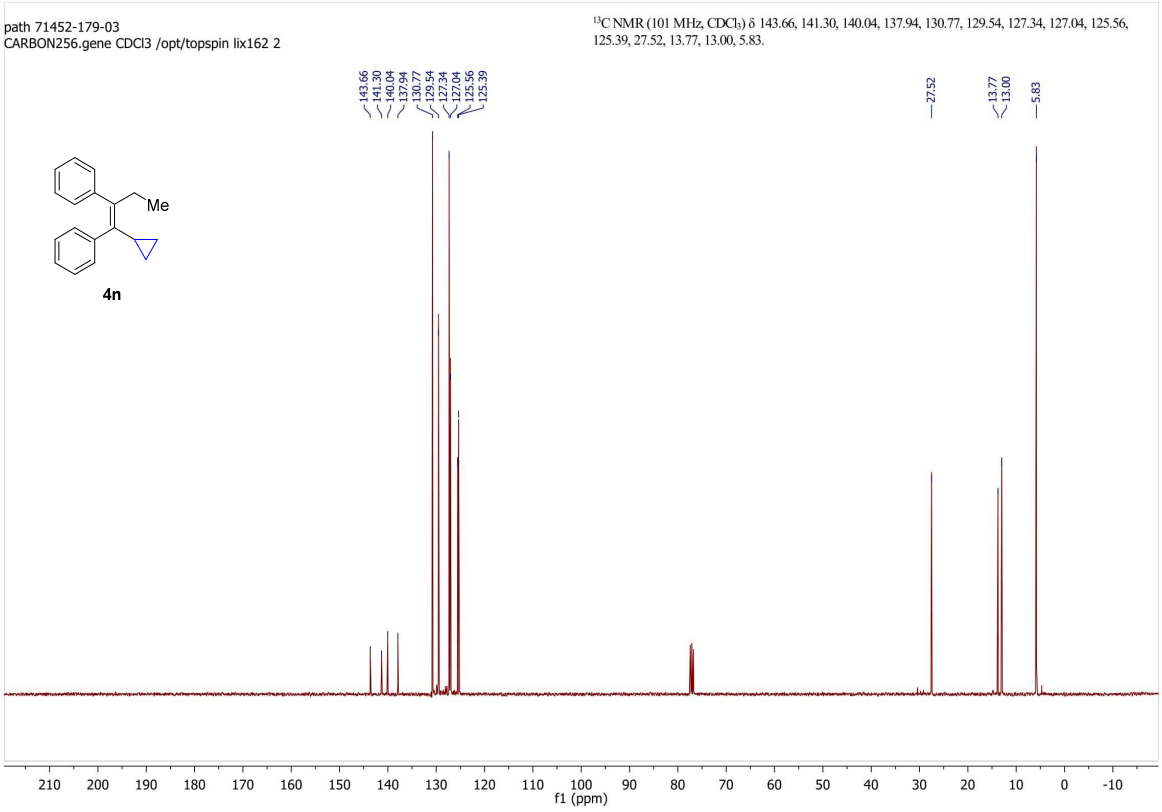
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 – 7.45 (m, 2H), 7.41 – 7.26 (m, 3H), 7.23 – 7.00 (m, 10H), 2.99 (q, *J* = 7.5 Hz, 2H), 1.10 (t, *J* = 7.5 Hz, 3H).



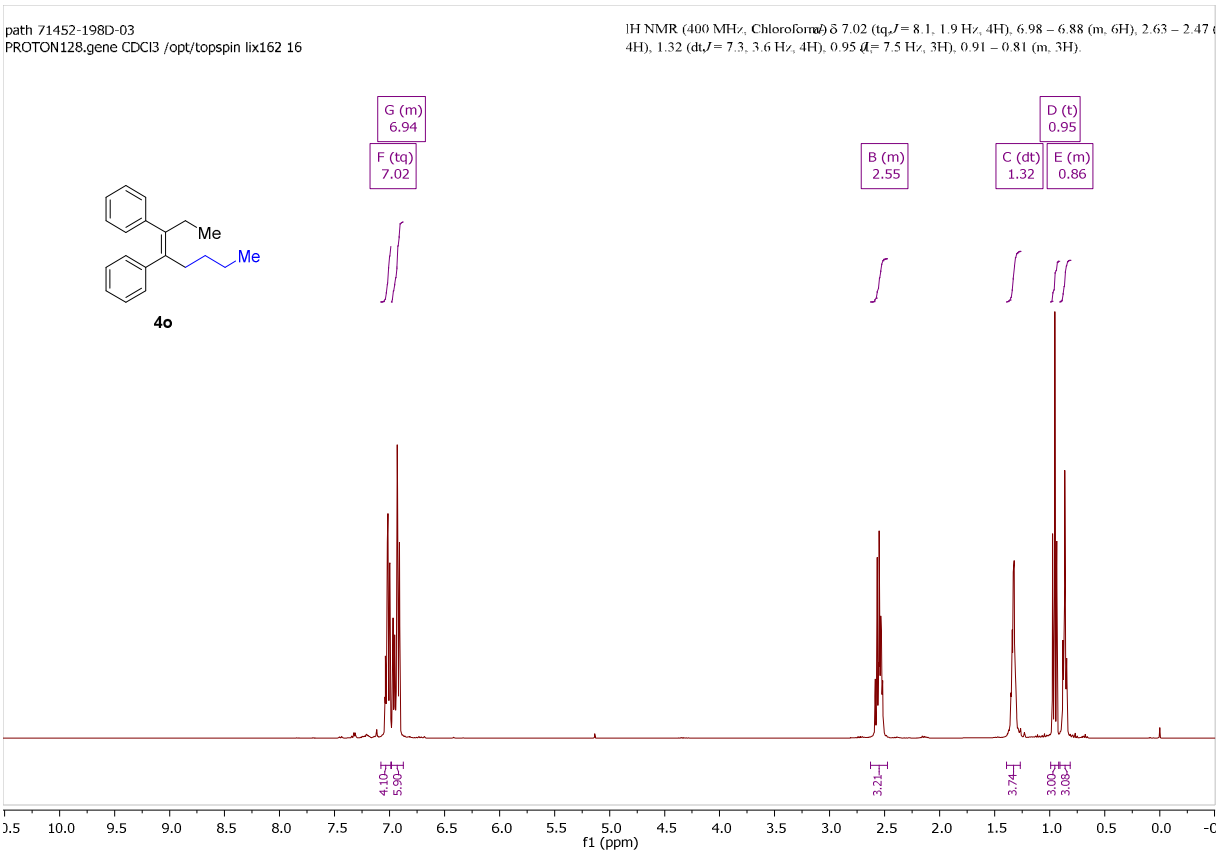
^{13}C NMR (101 MHz, $CDCl_3$) δ 151.86, 140.67, 139.00, 131.45, 129.87, 129.19, 128.32, 128.01, 127.95, 127.66, 126.90, 126.56, 123.87, 120.05, 93.71, 90.29, 31.40, 12.67.

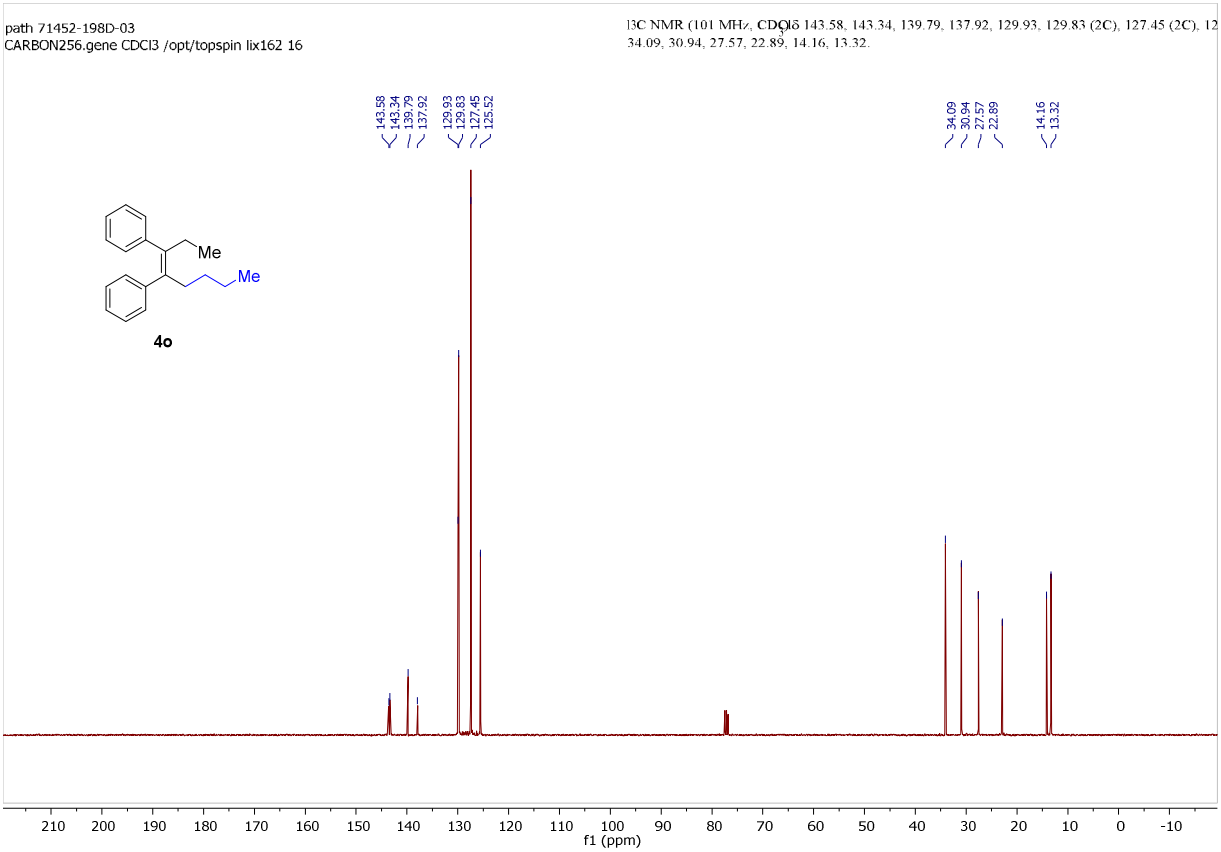


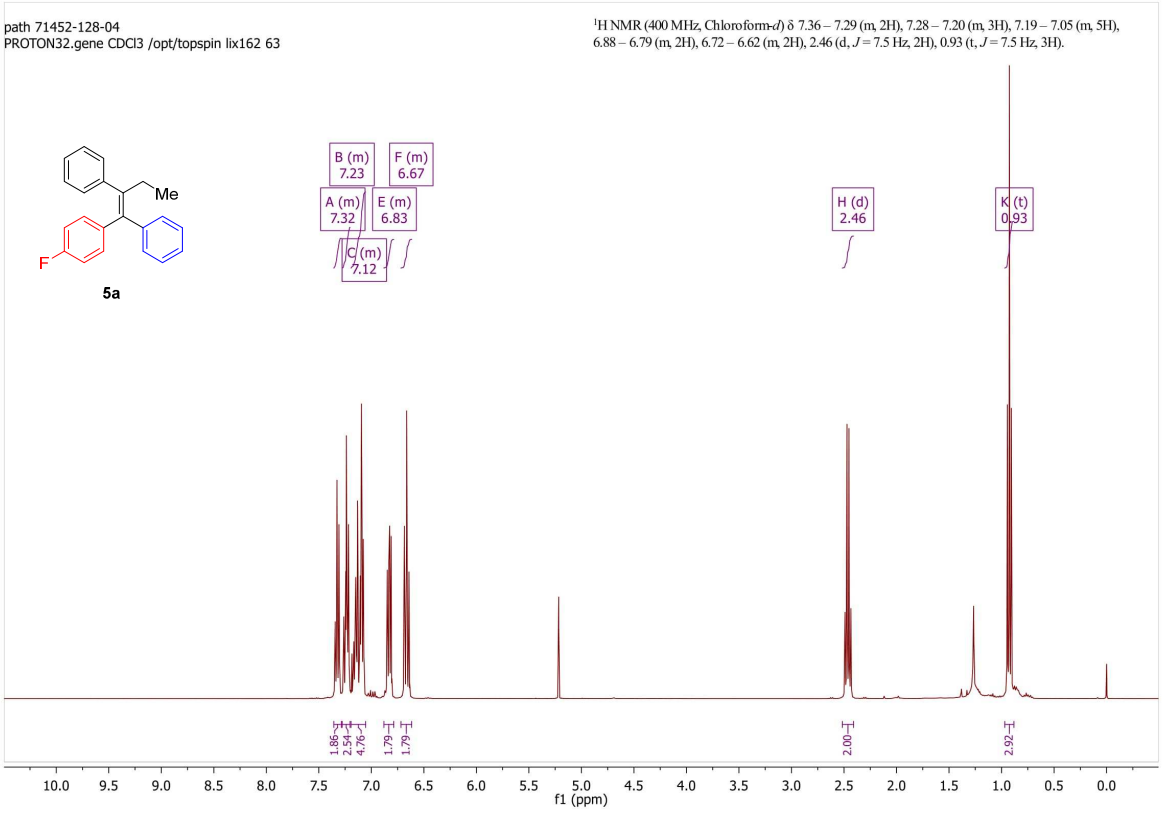
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.07 – 6.97 (m, 4H), 6.97 – 6.87 (m, 4H), 6.86 – 6.79 (m, 2H), 2.73 (d, *J* = 7.5 Hz, 2H), 2.00 – 1.88 (m, 1H), 1.04 (d, *J* = 7.8 Hz, 3H), 0.76 – 0.63 (m, 2H), 0.33 – 0.19 (m, 2H).



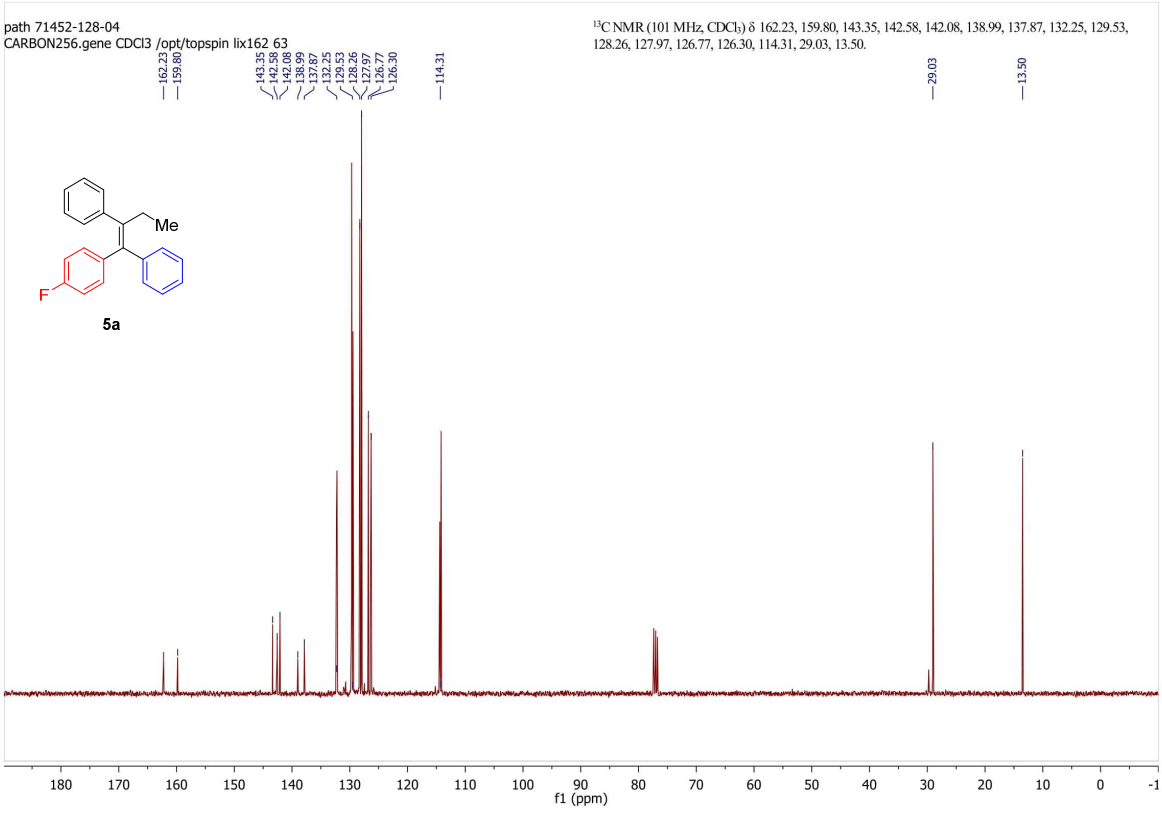
^{13}C NMR (101 MHz, $CDCl_3$) δ 143.66, 141.30, 140.04, 137.94, 130.77, 129.54, 127.34, 127.04, 125.56, 125.39, 27.52, 13.77, 13.00, 5.83.


path 71452-198D-03
PROTON128.gene CDCl3 /opt/topspin lix162 16
G (m)
6.94
F (tq)
7.02
B (m)
2.55
D (t)
0.95
C (dt)
1.32
E (m)
0.86
Me
Me
4o
4.10
5.90
3.21
3.74
3.00
3.08
f1 (ppm)


path 71452-198D-03
CARBON256.gene CDCl3 /opt/topspin lix162 16
143.58
143.34
139.79
137.92
129.93
129.83
127.45
125.52
34.09
30.94
27.57
22.89
14.16
13.32
Me
Me
4o
f1 (ppm)



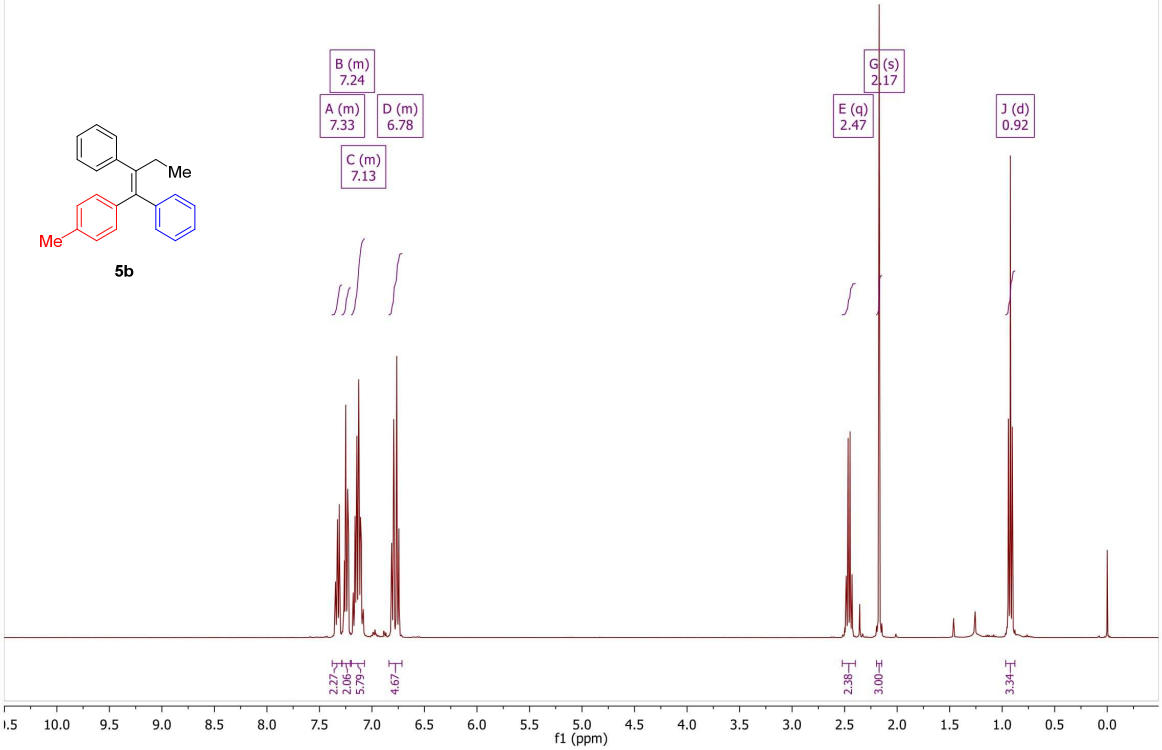
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 – 7.29 (m, 2H), 7.28 – 7.20 (m, 3H), 7.19 – 7.05 (m, 5H), 6.88 – 6.79 (m, 2H), 6.72 – 6.62 (m, 2H), 2.46 (d, *J* = 7.5 Hz, 2H), 0.93 (t, *J* = 7.5 Hz, 3H).



^{13}C NMR (101 MHz, $CDCl_3$) δ 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, 114.31, 29.03, 13.50.

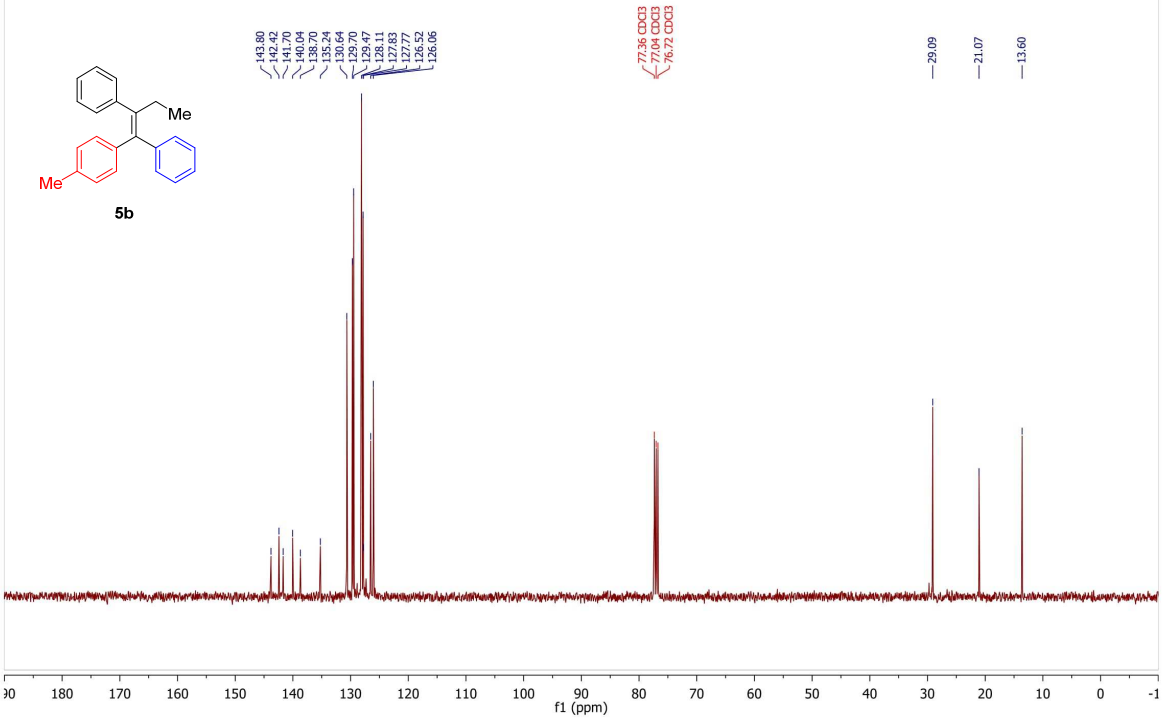
path 71452-123-04
PROTON32.gene CDCl3 /opt/topspin lix162 63

^{1}H NMR (400 MHz, Chloroform-*d*) 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).



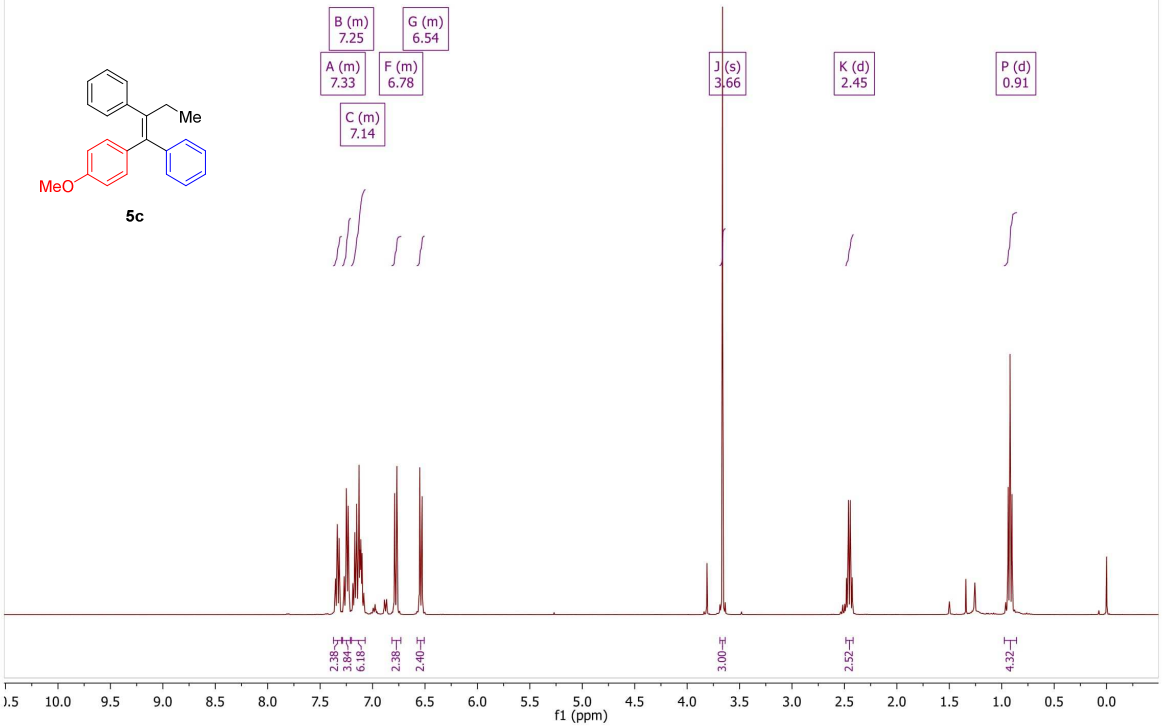
path 71452-123-04
CARBON256.gene CDCl3 /opt/topspin lix162 63

^{13}C NMR (101 MHz, $CDCl_3$) δ 143.80, 142.42, 141.70, 140.04, 138.70, 135.24, 130.64, 129.70, 129.47, 128.11, 127.83, 127.77, 126.52, 126.06, 29.09, 21.07, 13.60.



71452-124-04
PROTON32.gene CDCl3 /opt/topspin lix162 64

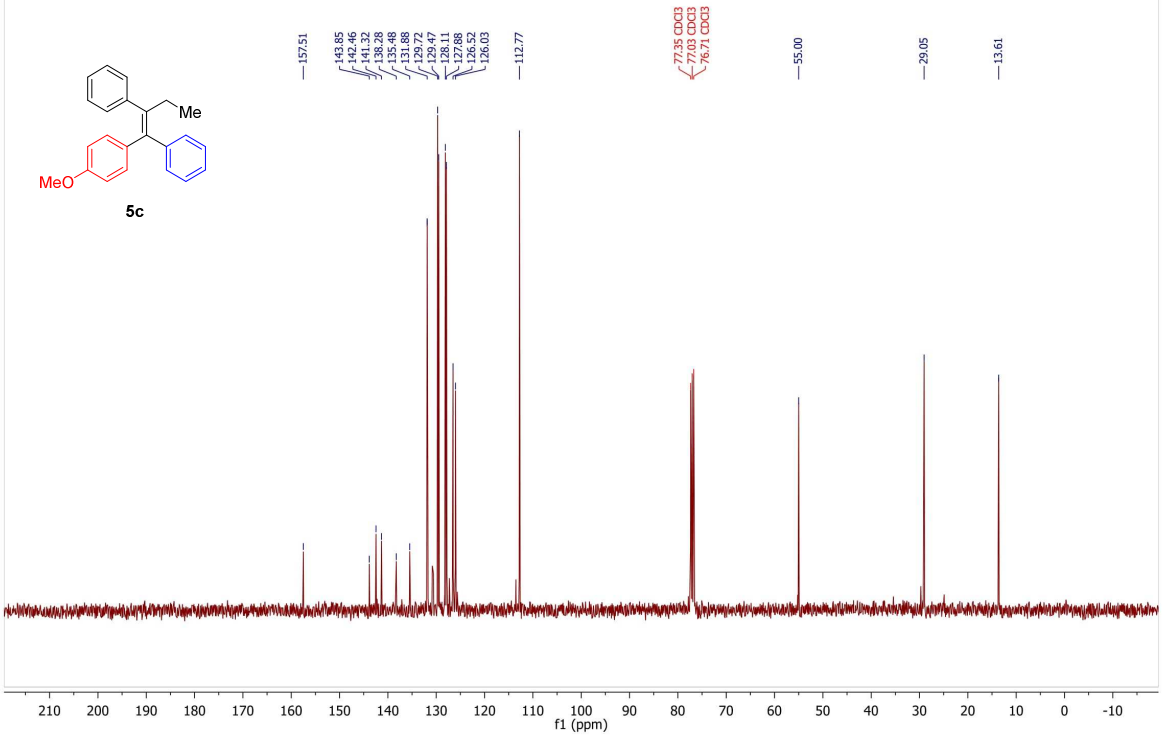
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 – 7.30 (m, 2H), 7.29 – 7.21 (m, 3H), 7.20 – 7.07 (m, 5H), 6.82 – 6.73 (m, 2H), 6.58 – 6.51 (m, 2H), 3.66 (s, 3H), 2.45 (d, *J* = 7.4 Hz, 2H), 0.91 (d, *J* = 7.4 Hz, 3H).



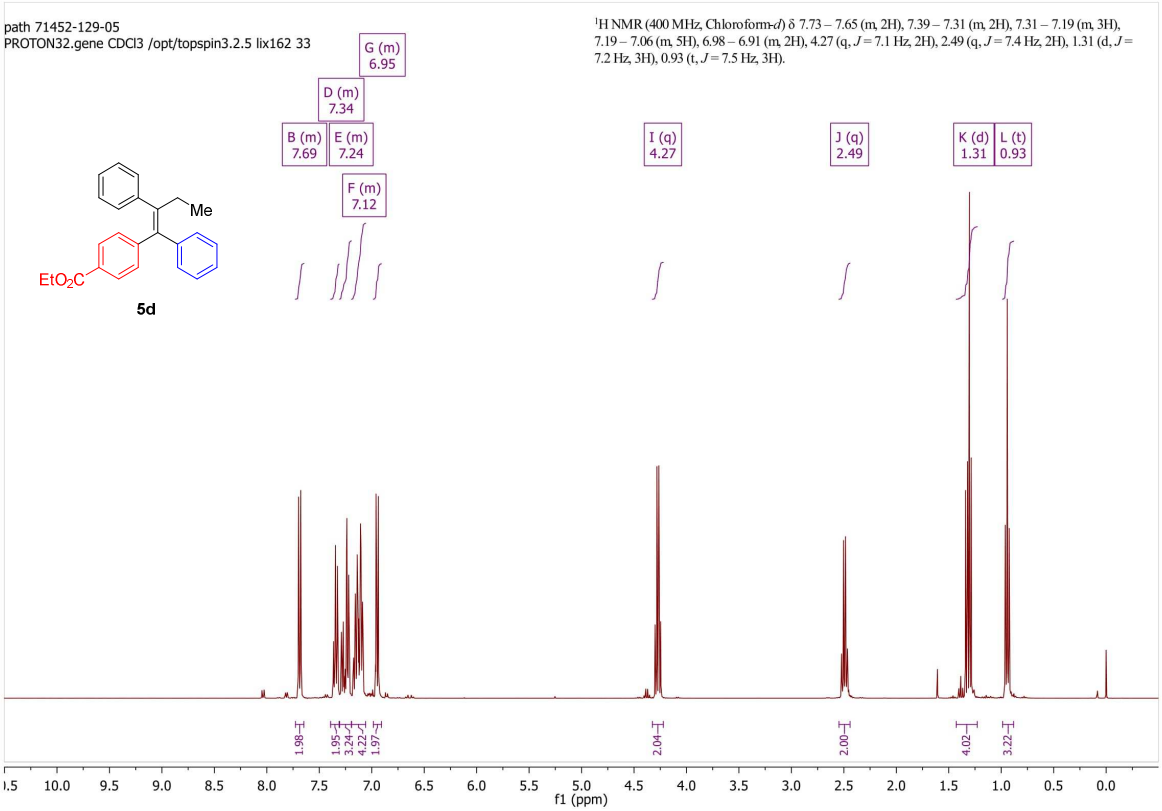
5c

71452-124-04
CARBON256.gene CDCl3 /opt/topspin lix162 64

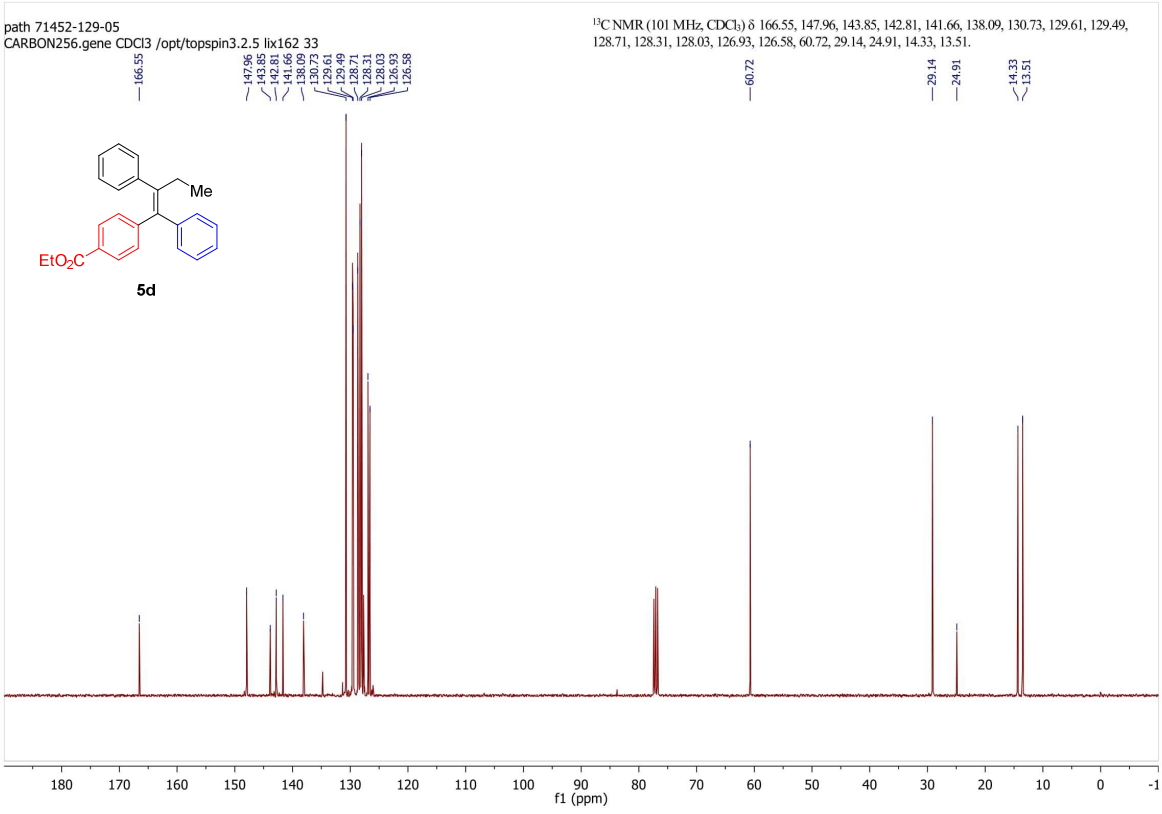
^{13}C NMR (101 MHz, $CDCl_3$) δ 157.51, 143.85, 142.46, 141.32, 138.28, 135.48, 131.88, 129.72, 129.47, 128.11, 127.88, 126.52, 126.03, 112.77, 55.00, 29.05, 13.61.



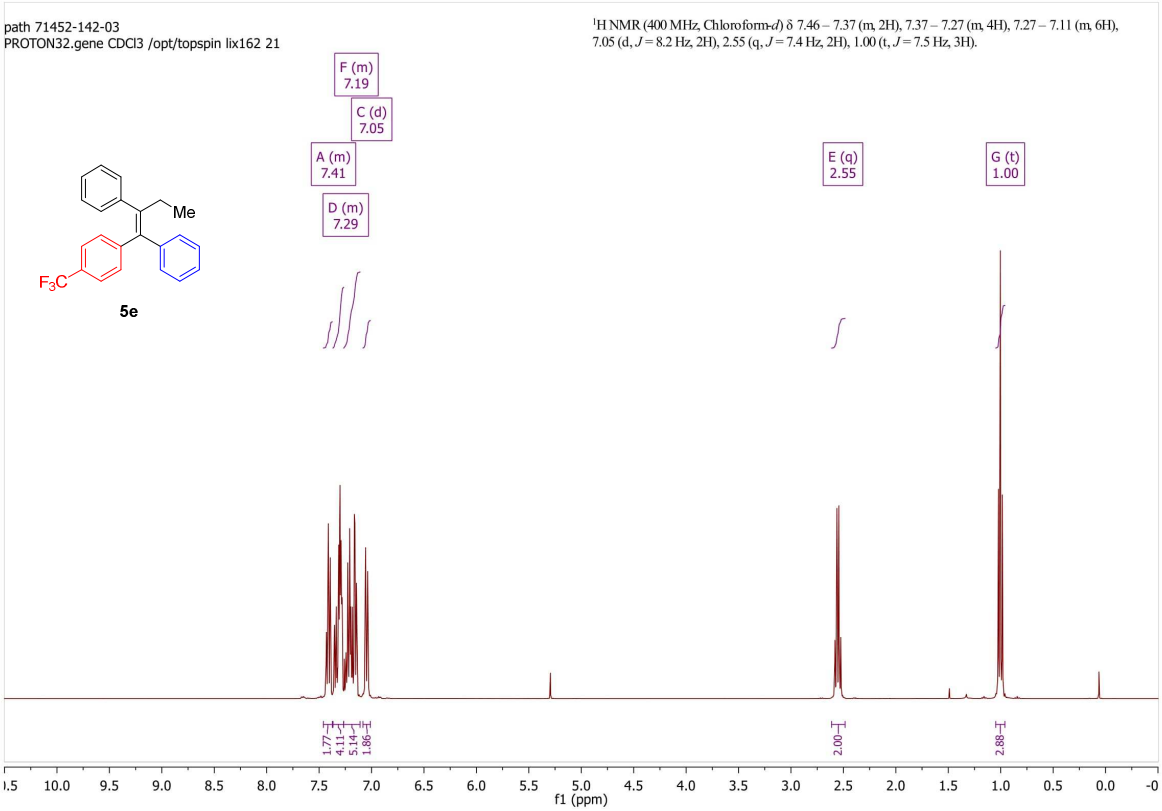
5c



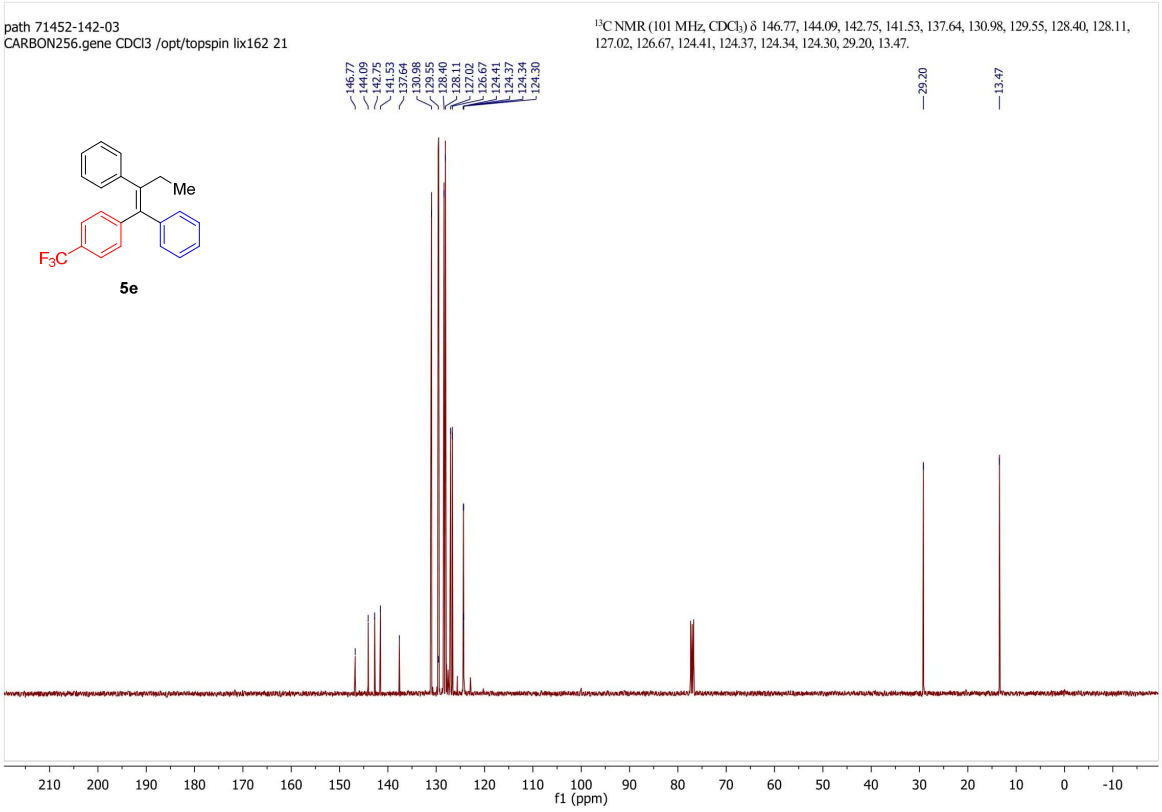
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.73 – 7.65 (m, 2H), 7.39 – 7.31 (m, 2H), 7.31 – 7.19 (m, 3H), 7.19 – 7.06 (m, 5H), 6.98 – 6.91 (m, 2H), 4.27 (q, *J* = 7.1 Hz, 2H), 2.49 (q, *J* = 7.4 Hz, 2H), 1.31 (d, *J* = 7.2 Hz, 3H), 0.93 (t, *J* = 7.5 Hz, 3H).



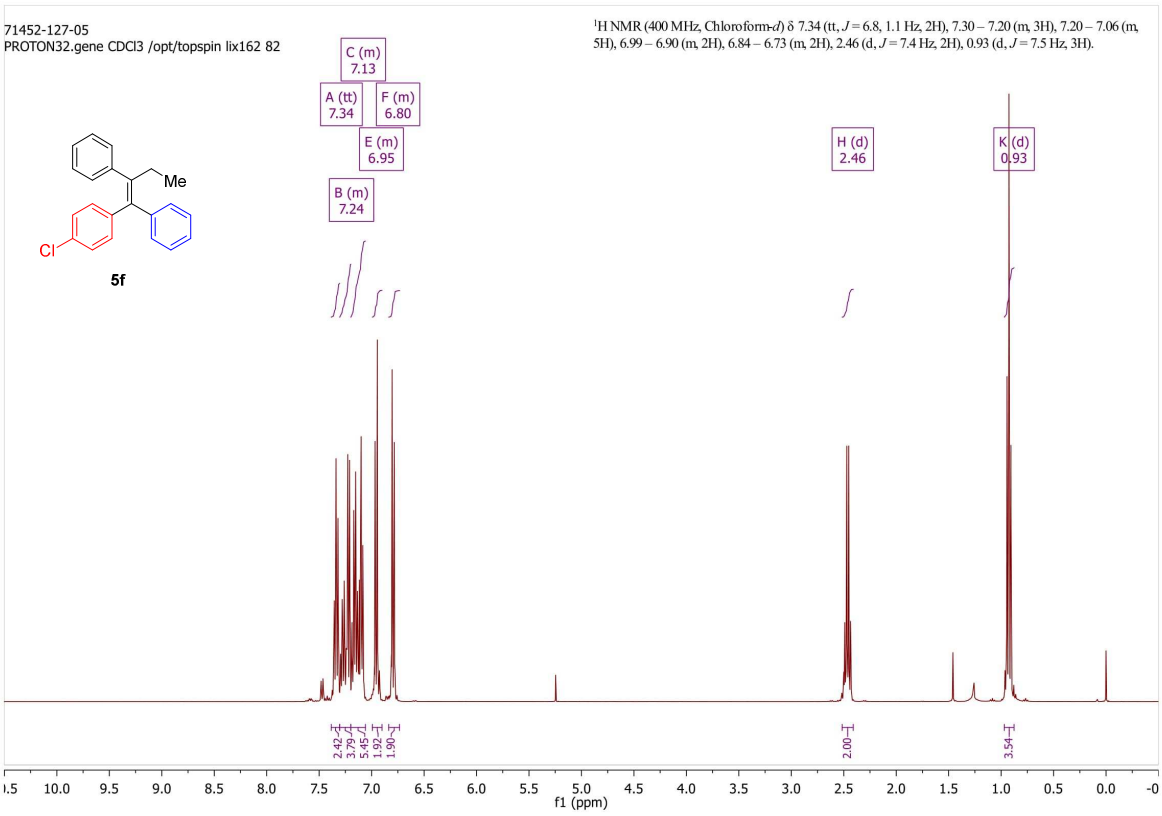
^{13}C NMR (101 MHz, $CDCl_3$) δ 166.55, 147.96, 143.85, 142.81, 141.66, 138.09, 130.73, 129.61, 129.49, 128.71, 128.31, 128.03, 126.93, 126.58, 60.72, 29.14, 24.91, 14.33, 13.51.



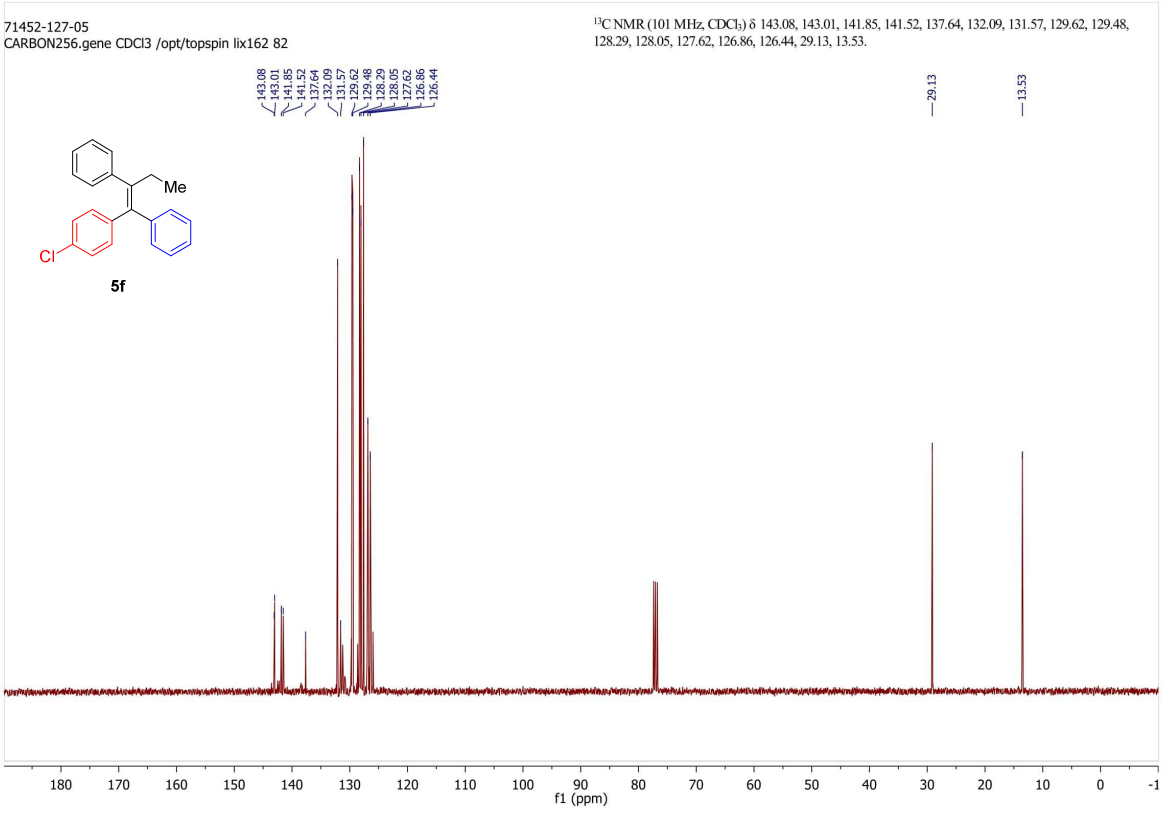
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46 – 7.37 (m, 2H), 7.37 – 7.27 (m, 4H), 7.27 – 7.11 (m, 6H), 7.05 (d, *J* = 8.2 Hz, 2H), 2.55 (q, *J* = 7.4 Hz, 2H), 1.00 (t, *J* = 7.5 Hz, 3H).



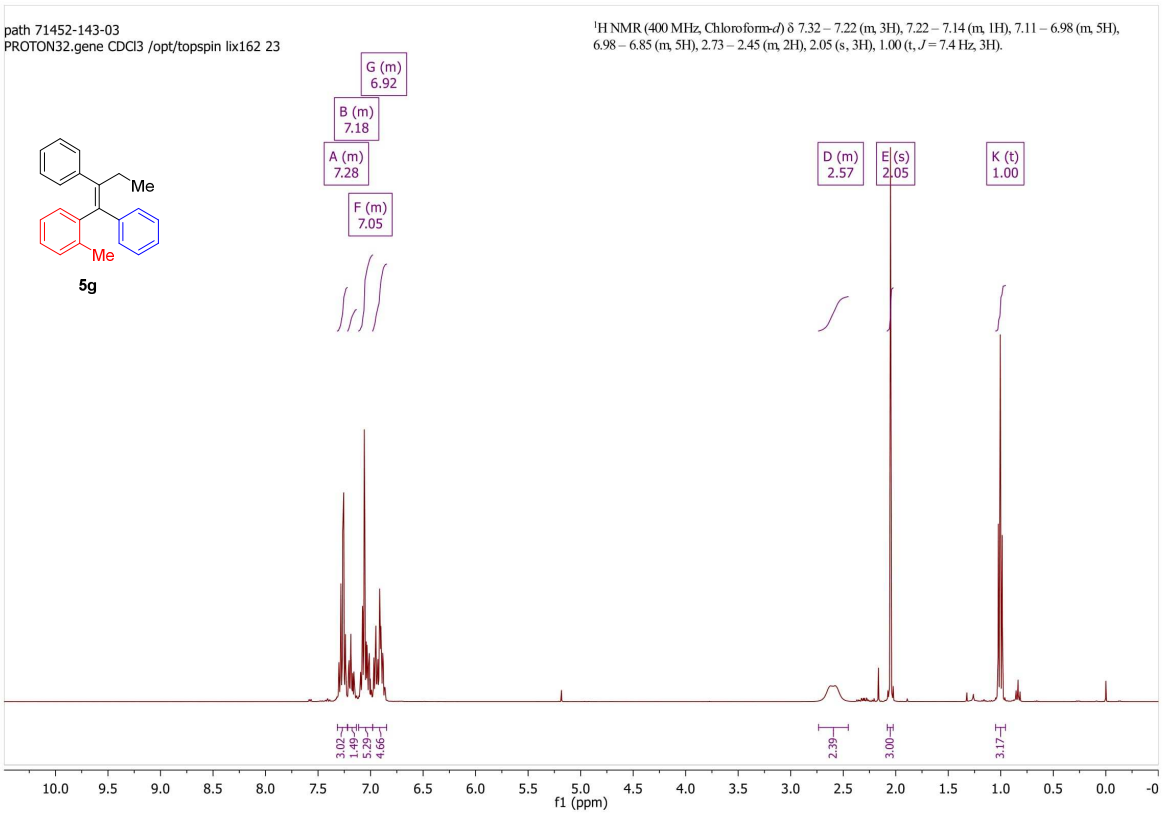
^{13}C NMR (101 MHz, $CDCl_3$) δ 146.77, 144.09, 142.75, 141.53, 137.64, 130.98, 129.55, 128.40, 128.11, 127.02, 126.67, 124.41, 124.37, 124.34, 124.30, 29.20, 13.47.



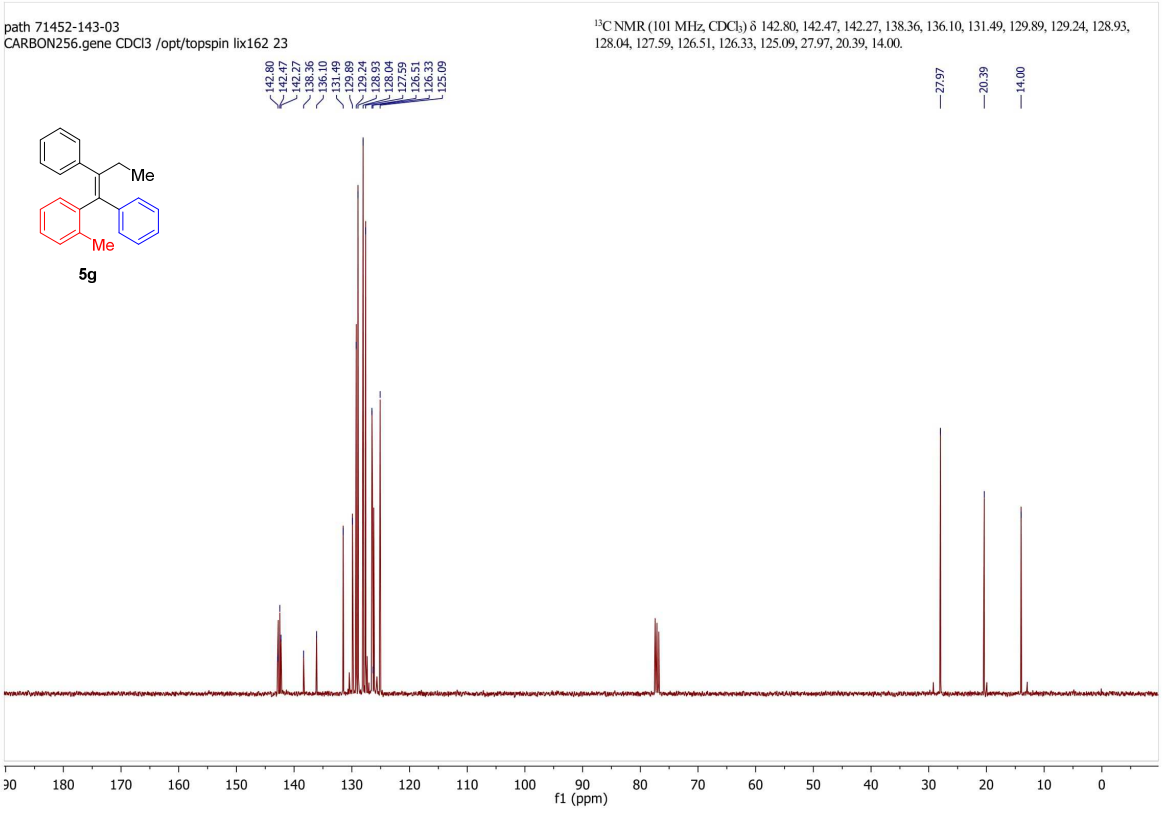
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 (tt, *J* = 6.8, 1.1 Hz, 2H), 7.30 – 7.20 (m, 3H), 7.20 – 7.06 (m, 5H), 6.99 – 6.90 (m, 2H), 6.84 – 6.73 (m, 2H), 2.46 (d, *J* = 7.4 Hz, 2H), 0.93 (d, *J* = 7.5 Hz, 3H).



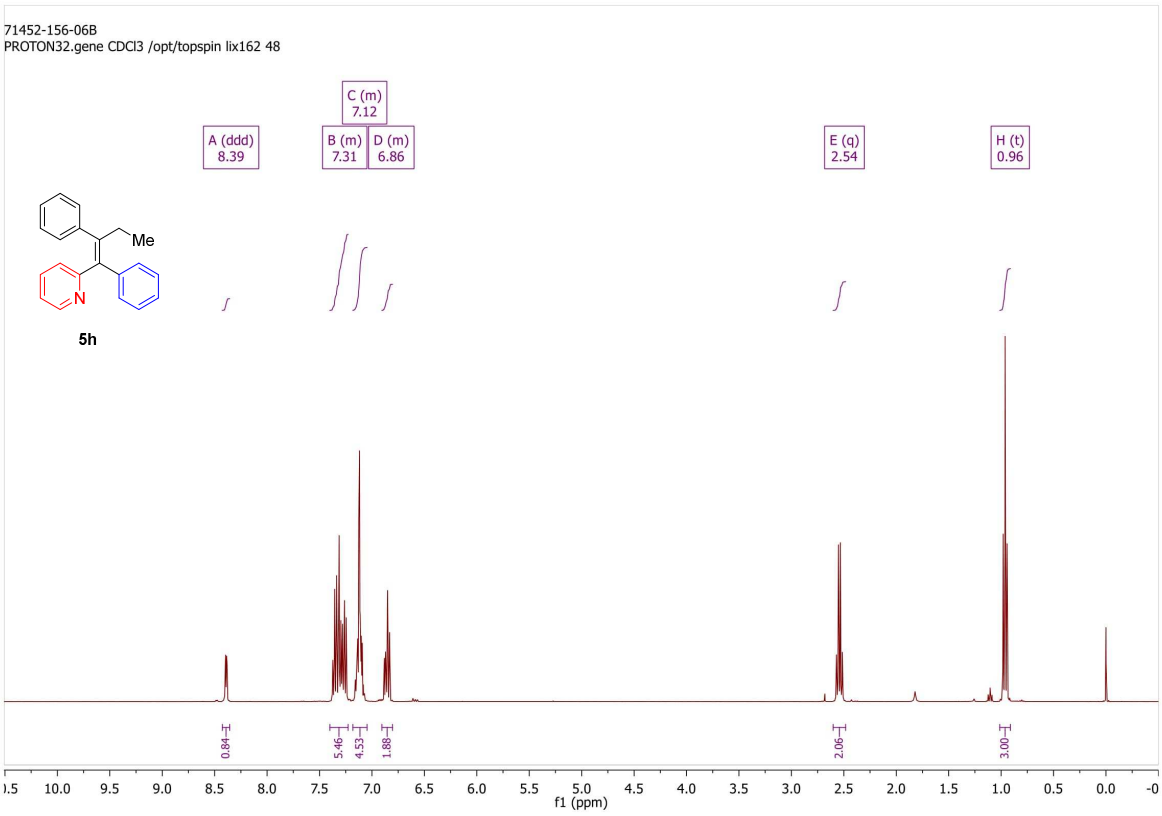
^{13}C NMR (101 MHz, $CDCl_3$) δ 143.08, 143.01, 141.85, 141.52, 137.64, 132.09, 131.57, 129.62, 129.48, 128.29, 128.05, 127.62, 126.86, 126.44, 29.13, 13.53.

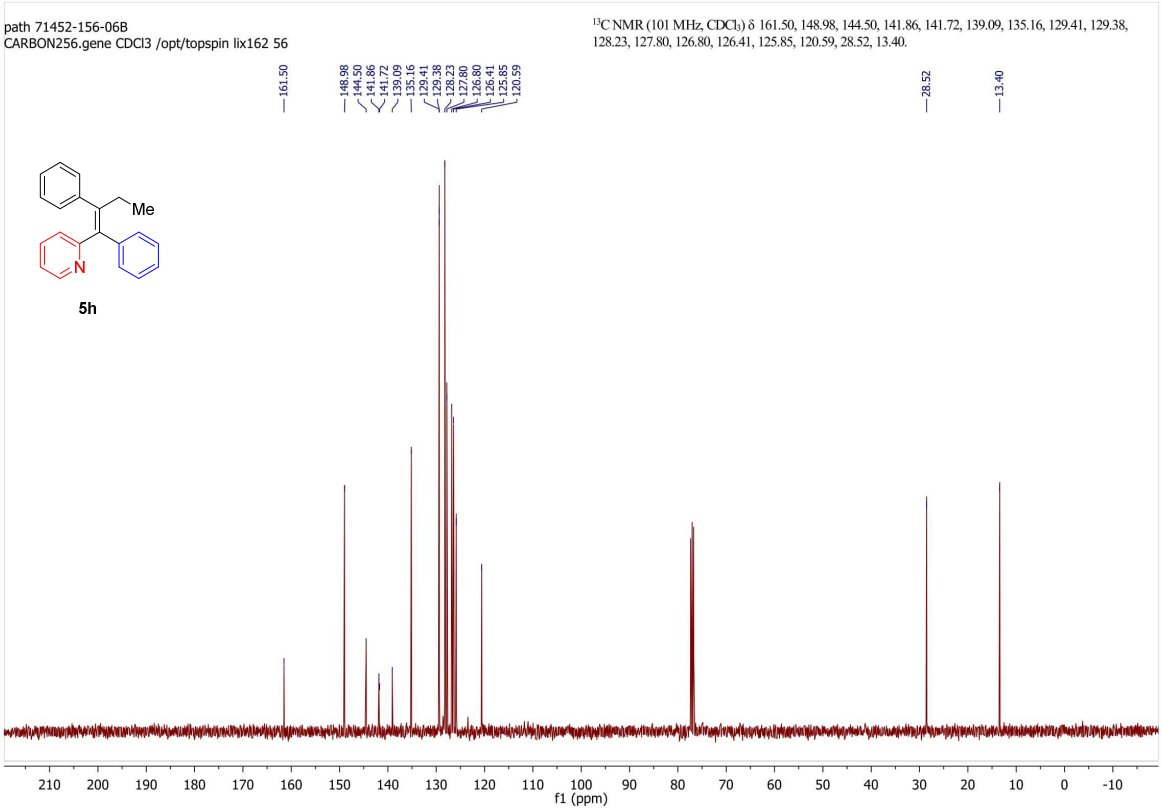


^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32 – 7.22 (m, 3H), 7.22 – 7.14 (m, 1H), 7.11 – 6.98 (m, 5H), 6.98 – 6.85 (m, 5H), 2.73 – 2.45 (m, 2H), 2.05 (s, 3H), 1.00 (t, *J* = 7.4 Hz, 3H).



^{13}C NMR (101 MHz, $CDCl_3$) δ 142.80, 142.47, 142.27, 138.36, 136.10, 131.49, 129.89, 129.24, 128.93, 128.04, 127.59, 126.51, 126.33, 125.09, 27.97, 20.39, 14.00.

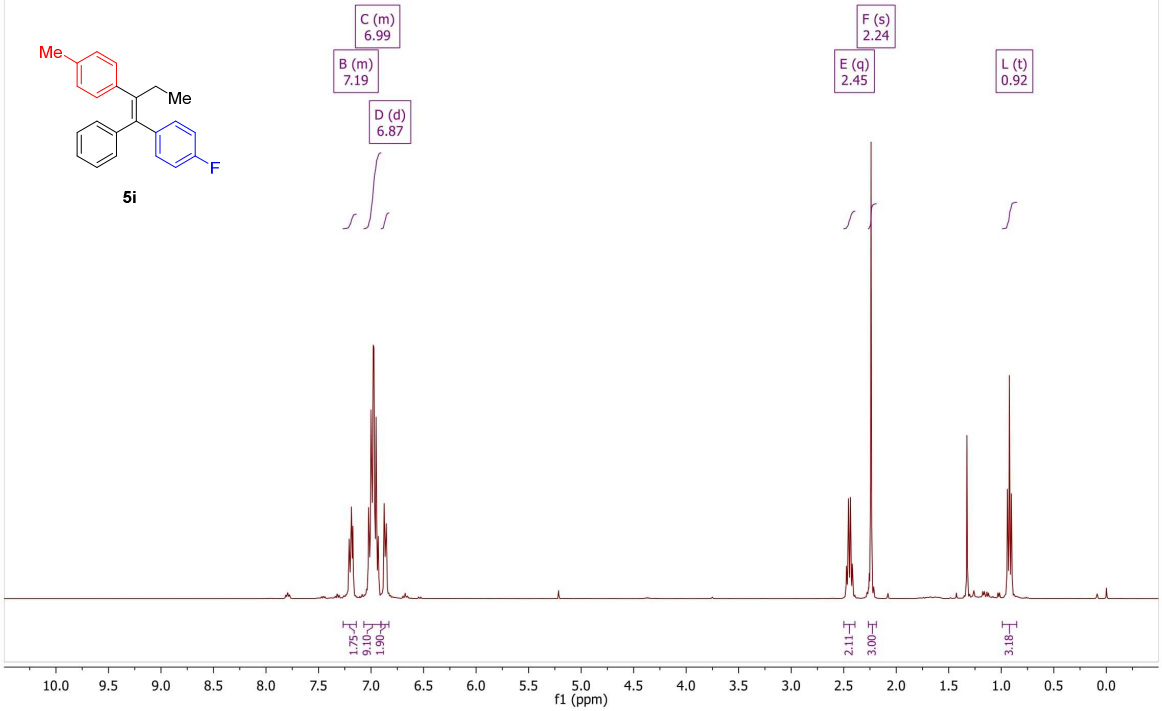




^{13}C NMR (101 MHz, $CDCl_3$) δ 161.50, 148.98, 144.50, 141.86, 141.72, 139.09, 135.16, 129.41, 129.38, 128.23, 127.80, 126.80, 126.41, 125.85, 120.59, 28.52, 13.40.

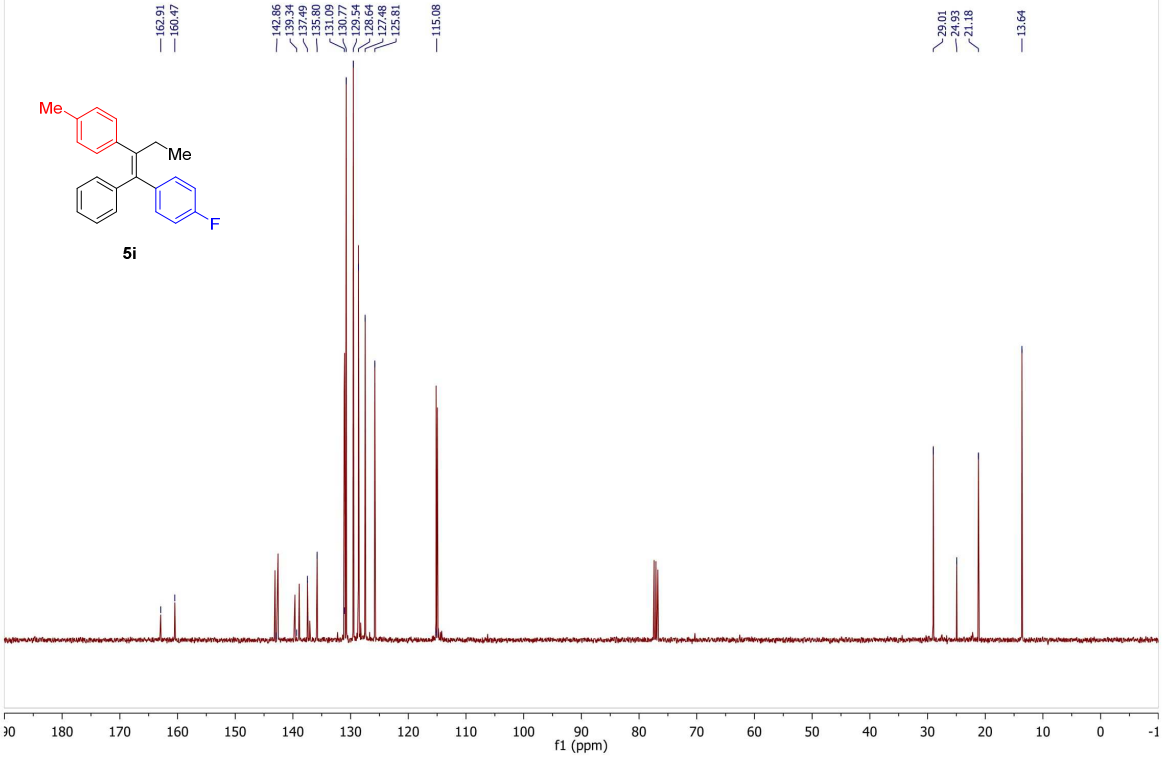
71452-147-03
PROTON32.gene CDCl3 /opt/topspin lix162 90

^{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), 0.92 (t, *J* = 7.4 Hz, 3H).



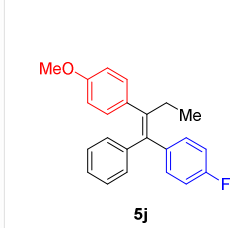
71452-147-03
CARBON256.gene CDCl3 /opt/topspin lix162 90

^{13}C NMR (101 MHz, $CDCl_3$) δ 162.91, 160.47, 142.86, 139.34, 137.49, 135.80, 131.09, 130.77, 129.54, 128.64, 127.48, 125.81, 115.08, 29.01, 24.93, 21.18, 13.64.



path 71452-132B-03
71452-132B-03
PROTON32.gene CDCl3 /opt/topspin lix162 94

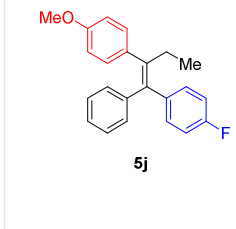
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.23 – 7.15 (m, 2H), 7.08 – 6.92 (m, 7H), 6.90 – 6.83 (m, 2H), 6.73 – 6.65 (m, 2H), 3.72 (s, 3H), 2.44 (q, *J* = 7.3 Hz, 2H), 1.33 (s, 1H), 0.93 (t, *J* = 7.4 Hz, 3H).



5j

path 71452-132B-03
71452-132B-03
CARBON256.gene CDCl3 /opt/topspin lix162 94

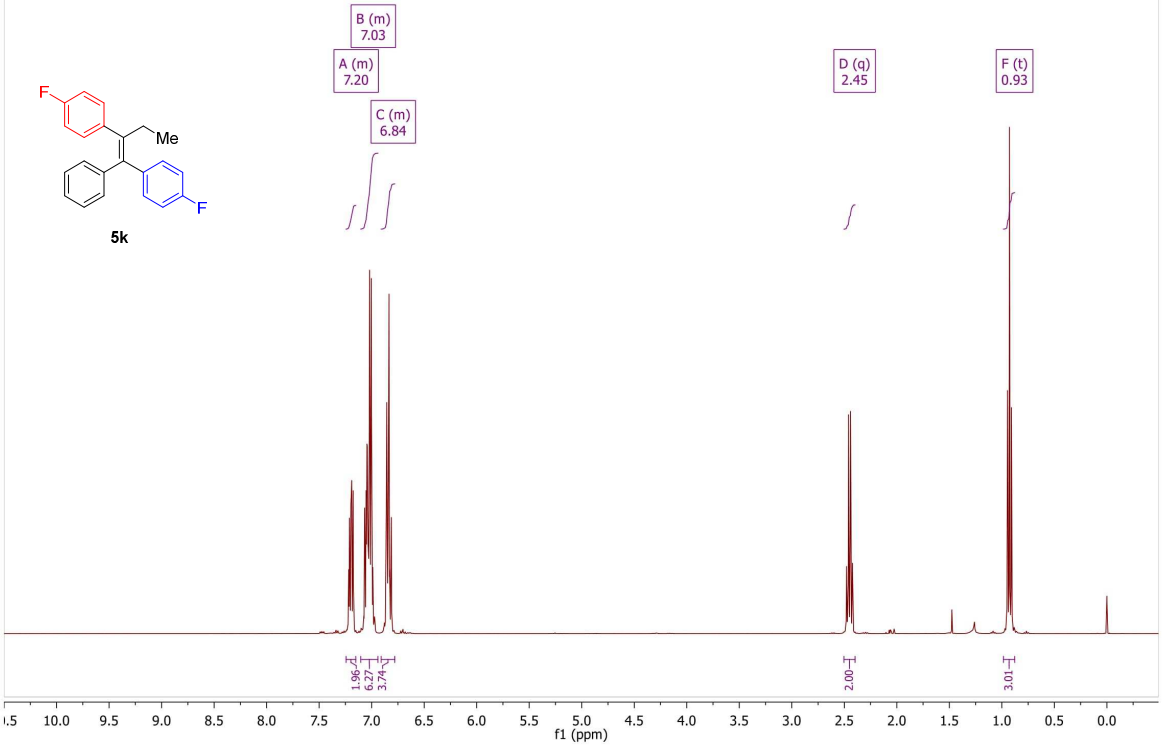
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.87, 160.43, 158.02, 142.72, 139.71, 137.31, 134.14, 131.10, 130.78, 127.50, 125.75, 115.15, 113.33, 55.08, 28.93, 24.90, 13.63.



5j

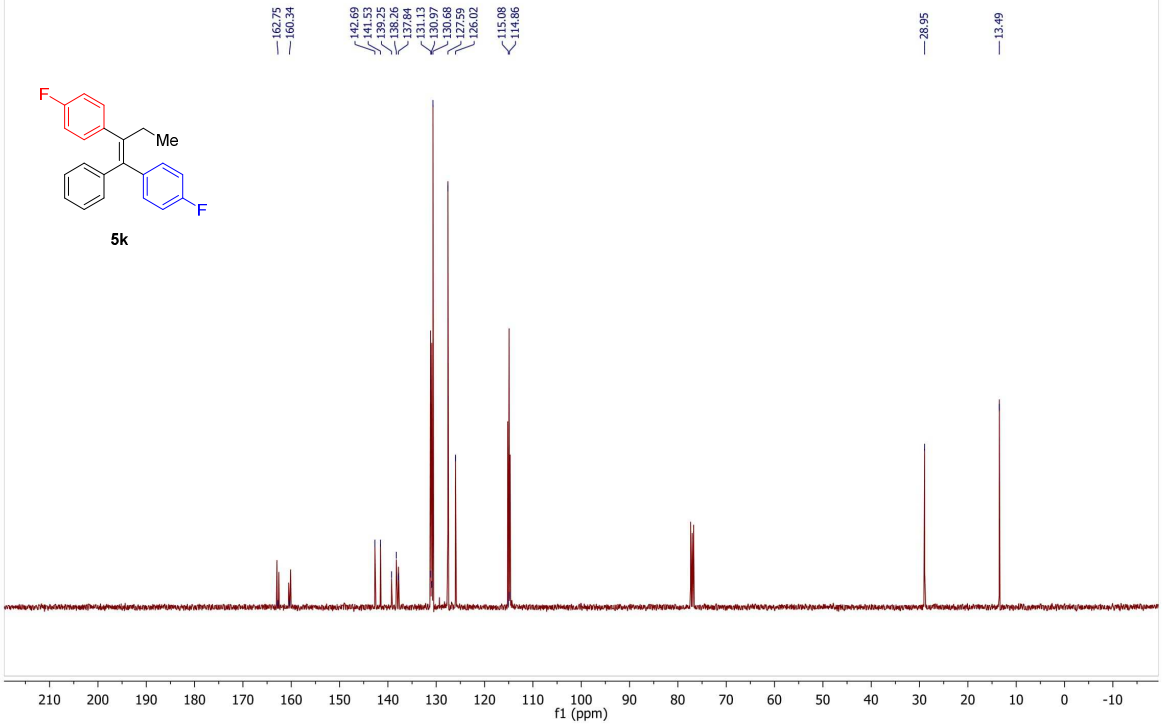
path 71452-131B-03
PROTON32.gene CDCl3 /opt/topspin lix162 93

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24 – 7.15 (m, 2H), 7.10 – 6.94 (m, 7H), 6.91 – 6.78 (m, 4H), 2.45 (q, *J* = 7.4 Hz, 2H), 0.93 (t, *J* = 7.4 Hz, 3H).



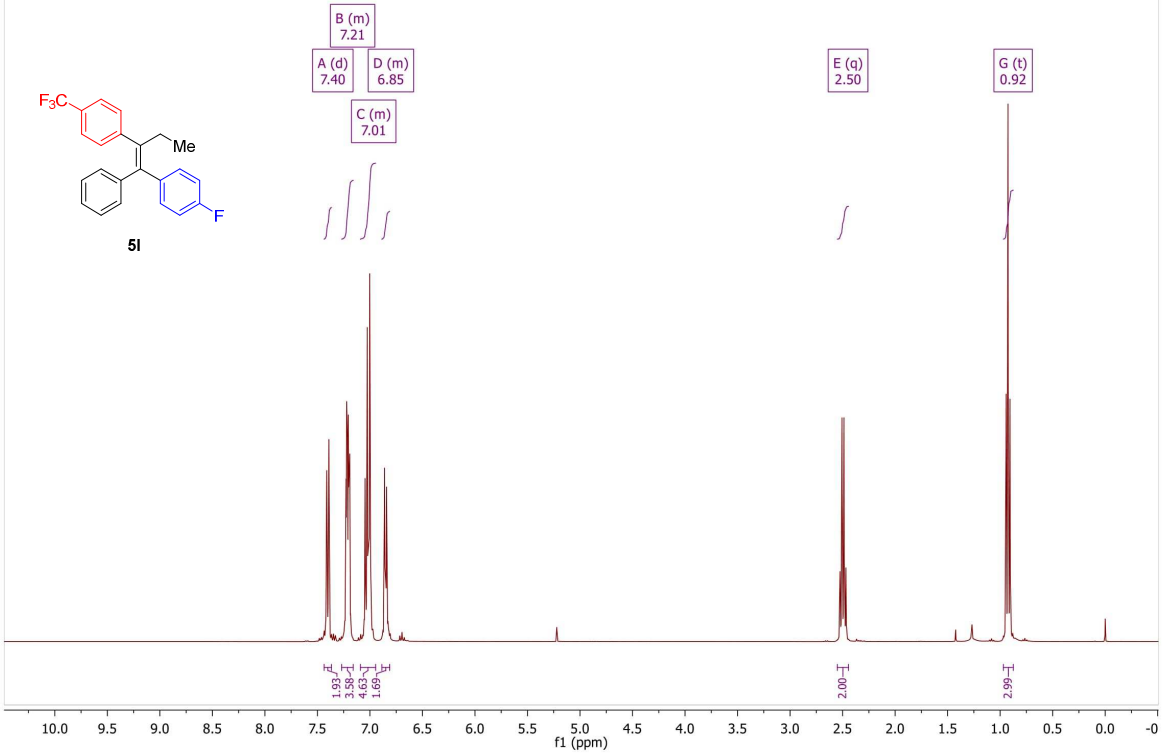
path 71452-131B-03
CARBON256.gene CDCl3 /opt/topspin lix162 93

^{13}C NMR (101 MHz, $CDCl_3$) δ 162.75, 160.34, 142.69, 141.53, 139.25, 138.26, 137.84, 131.13, 130.97, 130.68, 127.59, 126.02, 115.08, 114.86, 28.95, 13.49.



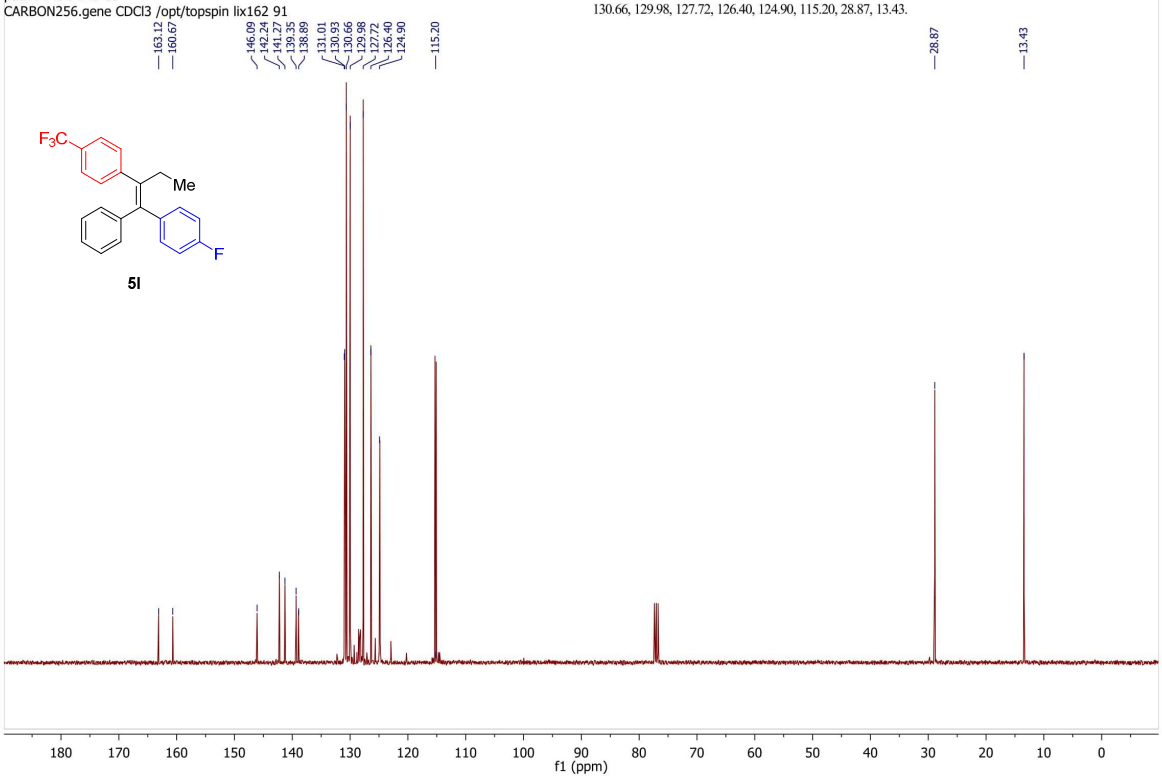
path 71452-148-03
PROTON32.gene CDCl3 /opt/topspin lix162 91

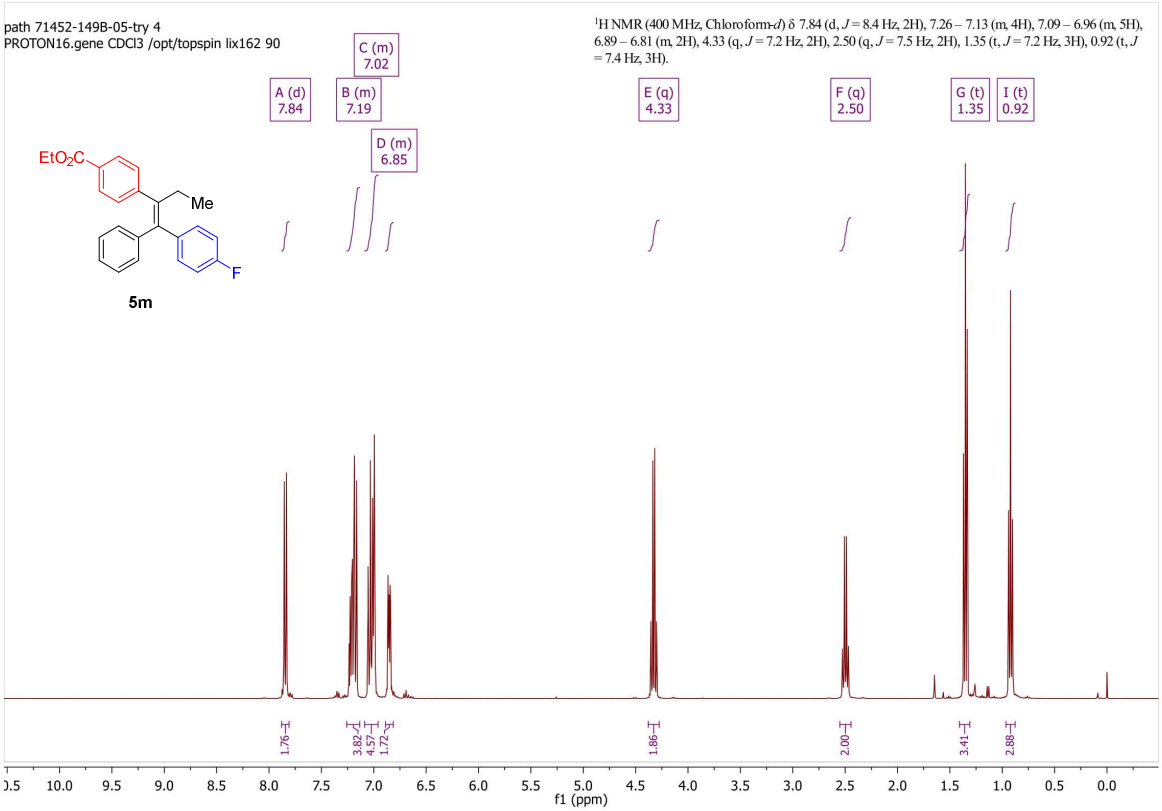
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.40 (d, *J* = 8.2 Hz, 2H), 7.27 – 7.16 (m, 4H), 7.09 – 6.95 (m, 5H), 6.89 – 6.81 (m, 2H), 2.50 (q, *J* = 7.5 Hz, 2H), 0.92 (t, *J* = 7.5 Hz, 3H).



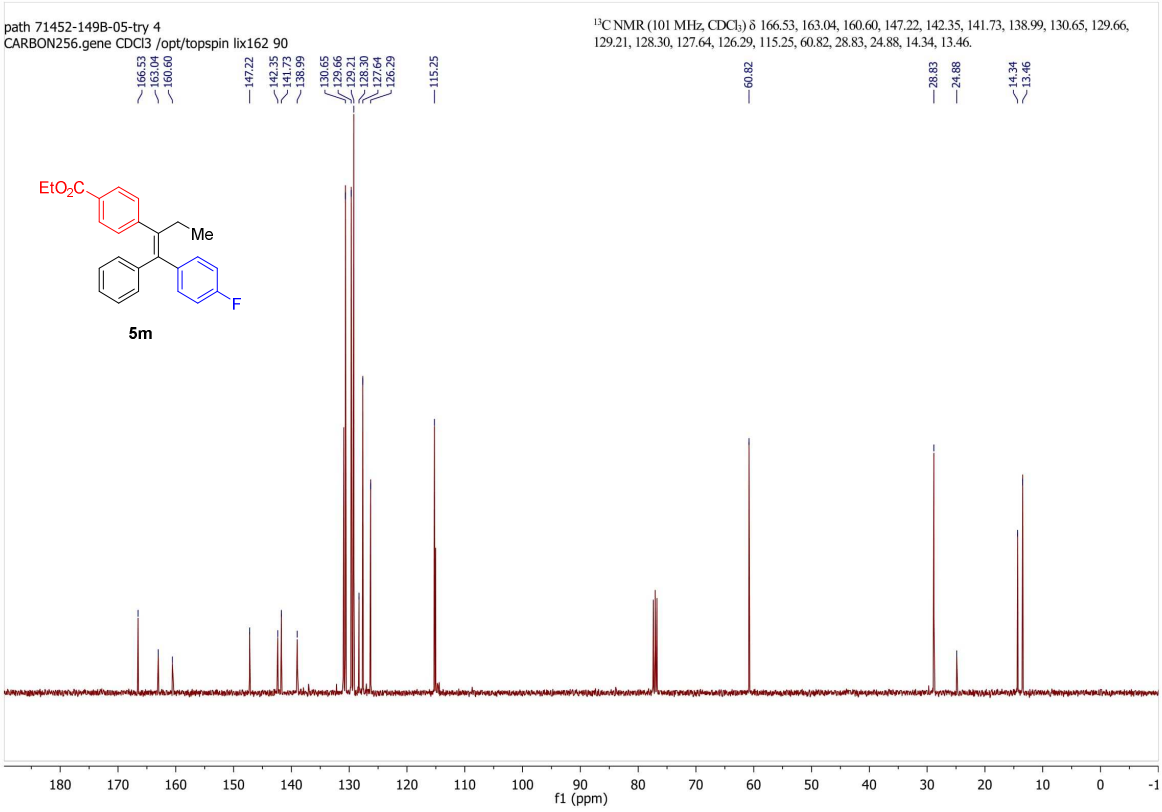
path 71452-148-03
CARBON256.gene CDCl3 /opt/topspin lix162 91

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.12, 160.67, 146.09, 142.24, 141.27, 139.35, 138.89, 131.01, 130.93, 130.66, 129.98, 127.72, 126.40, 124.90, 115.20, 28.87, 13.43.





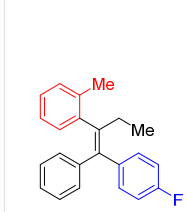
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.84 (d, *J* = 8.4 Hz, 2H), 7.26 – 7.13 (m, 4H), 7.09 – 6.96 (m, 5H), 6.89 – 6.81 (m, 2H), 4.33 (q, *J* = 7.2 Hz, 2H), 2.50 (q, *J* = 7.5 Hz, 2H), 1.35 (t, *J* = 7.2 Hz, 3H), 0.92 (t, *J* = 7.4 Hz, 3H).



^{13}C NMR (101 MHz, $CDCl_3$) δ 166.53, 163.04, 160.60, 147.22, 142.35, 141.73, 138.99, 130.65, 129.66, 129.21, 128.30, 127.64, 126.29, 115.25, 60.82, 28.83, 24.88, 14.34, 13.46.

71452-150-03-try4
PROTON16.gene CDCl3 /opt/topspin lix162 91

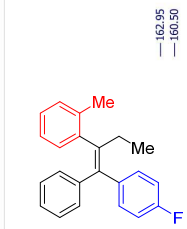
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.28 – 7.20 (m, 2H), 7.16 (dd, *J* = 7.3, 1.8 Hz, 1H), 7.12 – 6.92 (m, 8H), 6.87 – 6.79 (m, 2H), 2.39 (q, *J* = 7.5 Hz, 2H), 2.09 (s, 3H), 0.92 (t, *J* = 7.5 Hz, 3H).



5n

71452-150-03-try4
CARBON256.gene CDCl3 /opt/topspin lix162 91

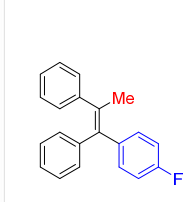
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.95, 160.50, 142.47, 141.25, 139.15, 138.12, 135.61, 131.14, 131.06, 129.83, 129.73, 127.25, 126.52, 125.98, 125.21, 115.12, 29.75, 19.76, 12.80.



5n

path 71452-138-03
PROTON32.gene CDCl3 /opt/topspin lix162 59

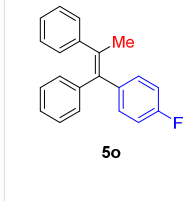
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.27 – 7.17 (m, 2H), 7.17 – 7.05 (m, 4H), 7.05 – 6.94 (m, 5H), 6.90 – 6.83 (m, 2H), 2.12 (s, 3H).



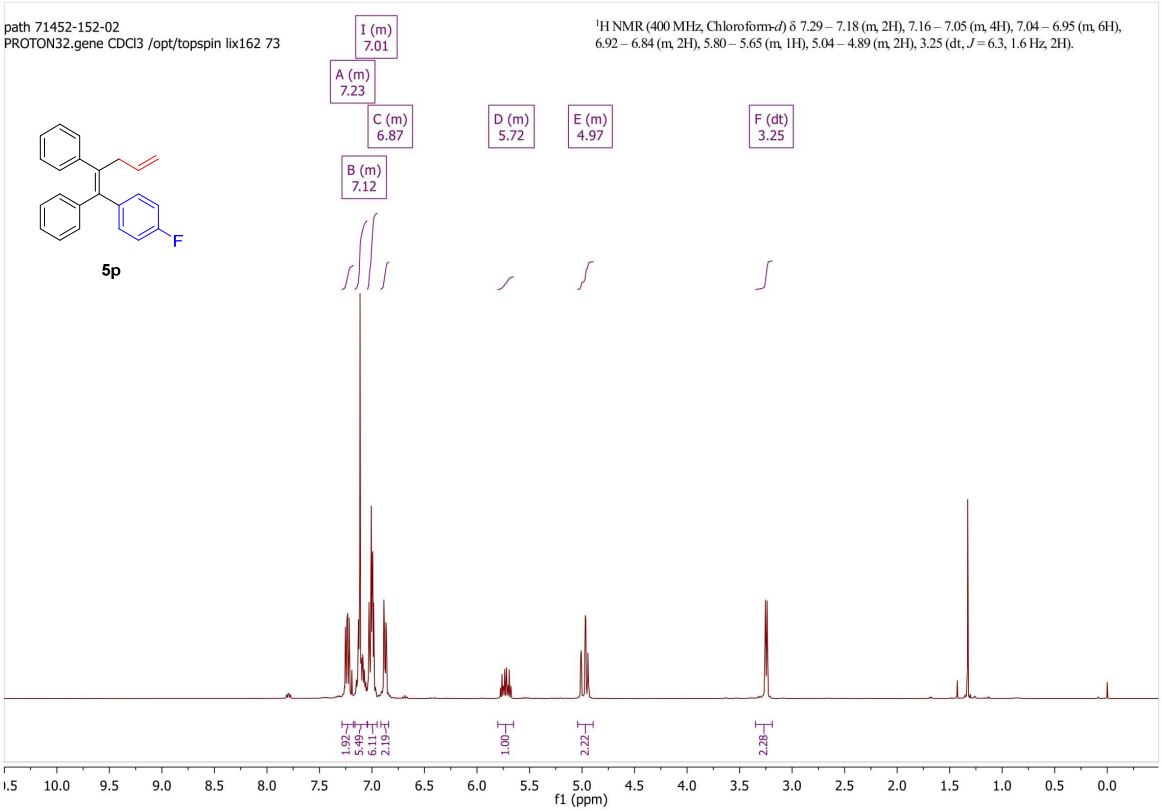
5o

path 71452-138-03
CARBON256.gene CDCl3 /opt/topspin lix162 59

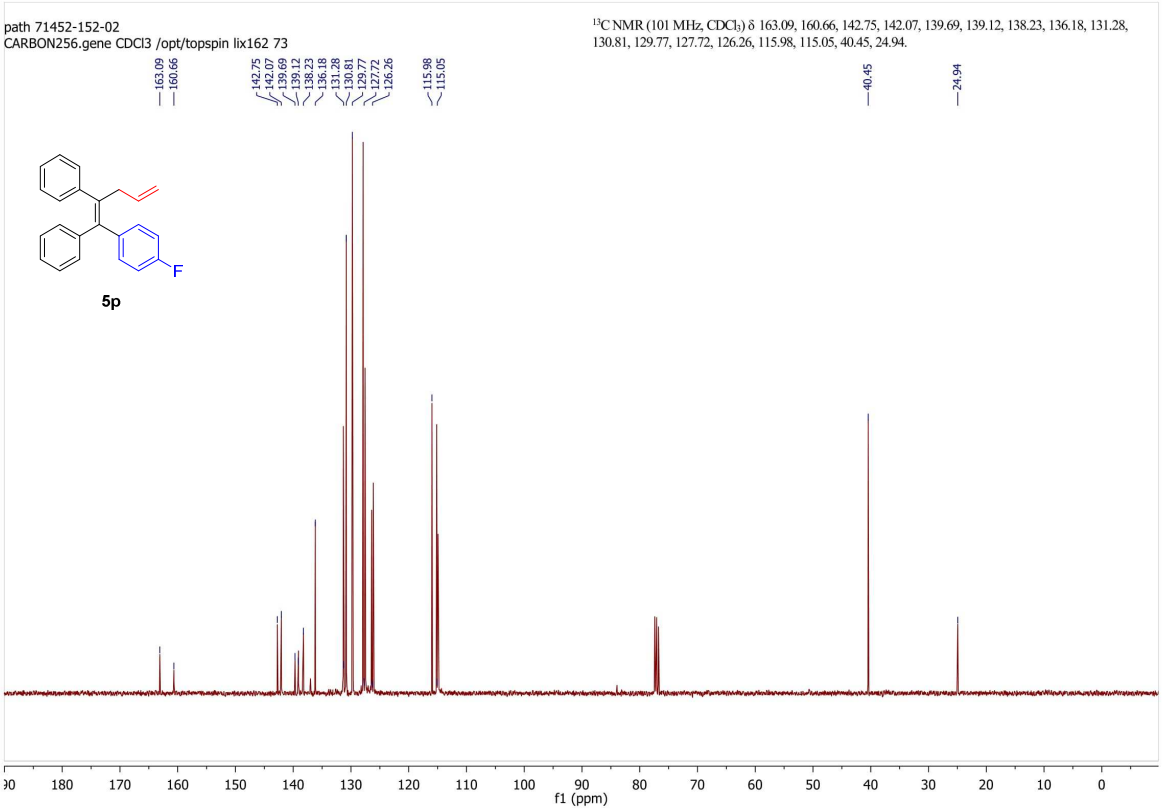
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.86, 160.42, 143.89, 142.94, 139.44, 138.37, 136.12, 131.63, 130.84, 129.28, 127.74, 126.34, 126.01, 115.08, 23.39.



5o



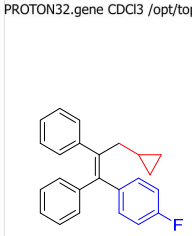
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.29 – 7.18 (m, 2H), 7.16 – 7.05 (m, 4H), 7.04 – 6.95 (m, 6H), 6.92 – 6.84 (m, 2H), 5.80 – 5.65 (m, 1H), 5.04 – 4.89 (m, 2H), 3.25 (dt, *J* = 6.3, 1.6 Hz, 2H).



^{13}C NMR (101 MHz, $CDCl_3$) δ 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.

71452-151-02
PROTON32.gene CDCl3 /opt/topspin lix162 72

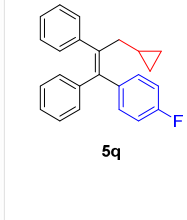
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.31 – 7.19 (m, 5H), 7.19 – 7.06 (m, 5H), 0.46 – 0.36 (m, 2H), 0.01 (dt, *J* = 5.9, 4.4 Hz, 2H), 7.41 – 7.33 (m, 2H), 7.05 – 6.99 (m, 2H), 2.49 (d, *J* = 6.6 Hz, 2H), 0.87 – 0.73 (m, 1H).



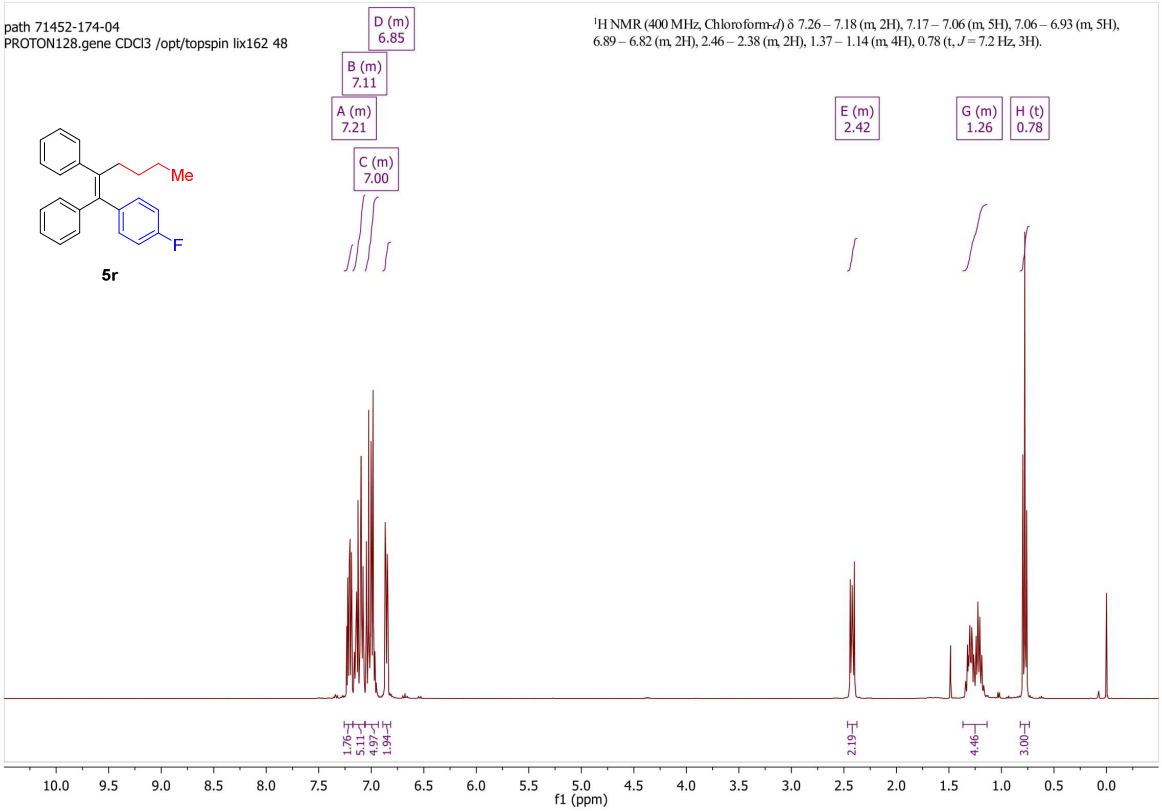
5q

71452-151-02
CARBON256.gene CDCl3 /opt/topspin lix162 72

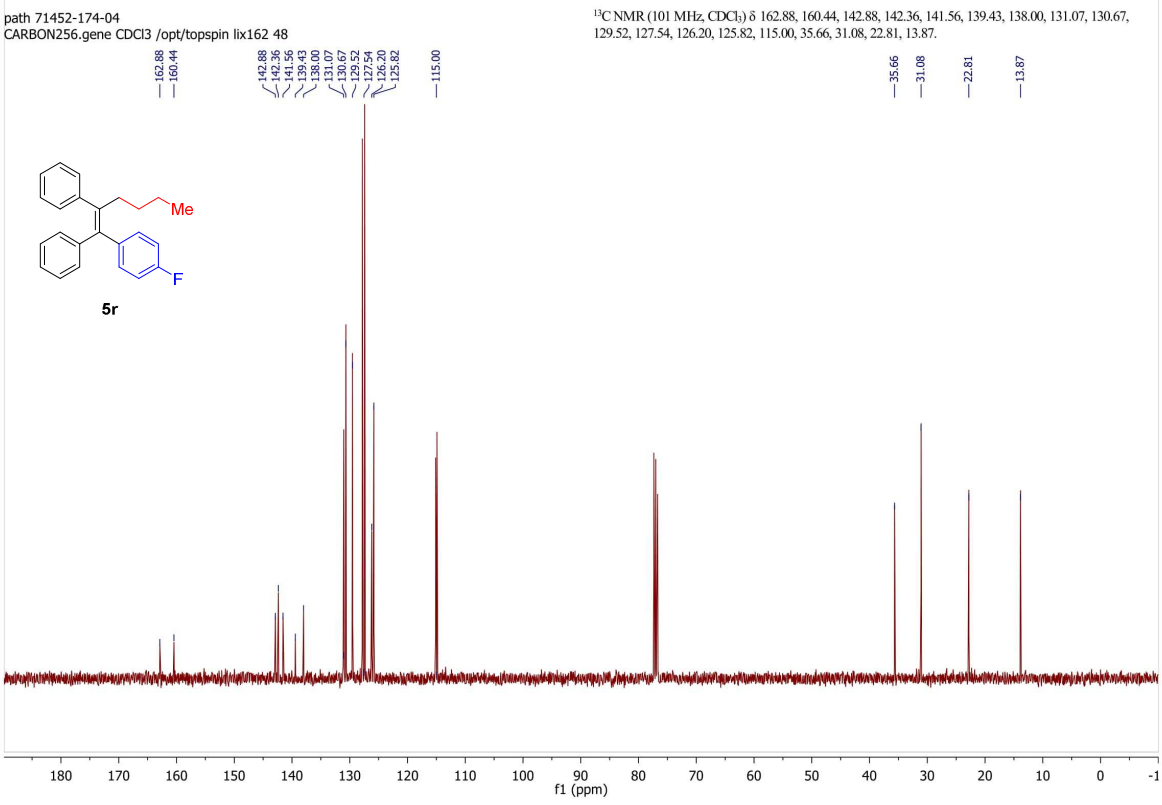
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.95, 142.76, 141.21, 139.24, 138.23, 131.26, 130.61, 129.76, 127.78, 127.48, 126.20, 125.91, 115.08, 40.14, 10.43, 4.85.



5q



^{1}H NMR (400 MHz, Chloroform-*d*) 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 162.88, 160.44, 142.88, 142.36, 141.56, 139.43, 138.00, 131.07, 130.67, 129.52, 127.54, 126.20, 125.82, 115.00, 35.66, 31.08, 22.81, 13.87.