Electronic Supplementary Information

Palladium-Catalyzed Oxidative Borylation of Conjugated Enynones through Carbene Migratory Insertion: Synthesis of Furyl-Substituted Alkenylboronates

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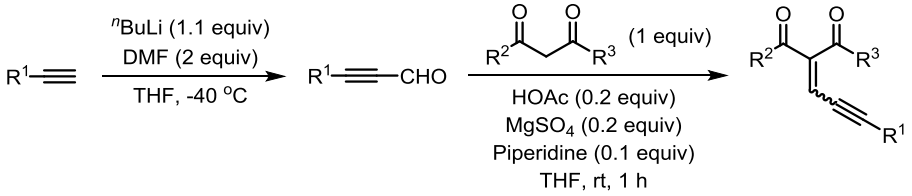
1. General

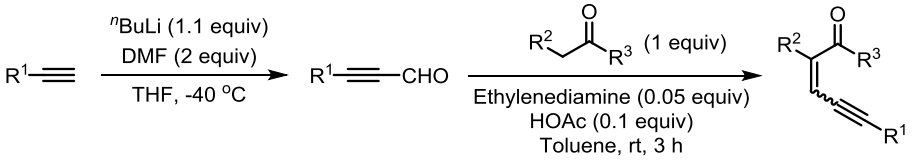
All the palladium-catalyzed reactions were performed under nitrogen atmosphere in a flame-dried reaction tube. For chromatography, 200–300 mesh silica gel (Qingdao, China) was employed. HPLC grade methanol was used as the solvent without further purification. ^{1}H NMR spectra were recorded on Bruker ARX 400 (400 MHz) or Bruker ARX 500 (500 MHz); ^{13}C NMR spectra were recorded on Bruker ARX 400 (101 MHz) or Bruker ARX 500 (126 MHz). The data for NMR spectra were reported as follows: chemical shifts (δ) were reported in ppm using tetramethylsilane as internal standard when using $CDCl_3$ as solvent, and coupling constants (J) were in Hertz (Hz). The resonances for carbon atoms directly attached to boron were not observed due to quadrupolar relaxation. IR spectra were recorded on Nicolet 5MX–S infrared spectrometer and were reported in terms of frequency of absorption (cm^{-1}). HRMS were obtained on Bruker APEX IV FTMS. Conjugated enynones were synthesized according to the previous report.[1,2] Other starting materials were purchased from commercial source and used without further purification. (PE: petroleum ether; EA: ethyl acetate).

2. General Procedure for Preparation of Conjugated Enynones



Conjugated enynones **1a-1l**, **1o-1s** were prepared according to our previous report.[1] The alkyne (10 mmol) was dissolved in dry THF (15 mL) and the solution was cooled to −40 ℃ under nitrogen, *n*-butyllithium (1.6 M in hexanes, 6.8 mL, 11 mmol) was added dropwise over *ca.* 2 minutes while maintaining the temperature between −35 and −40 ℃. After completion of the addition, anhydrous DMF (1.55 mL, 20 mmol) was added in one portion and the cold bath was removed. The reaction mixture was allowed to warm to room temperature and aged for 30 minutes. The THF solution was poured into a vigorously stirred biphasic solution prepared from aqueous solution of KH_2PO_4 (50 mL, 30 mmol) and Et_2O (30 mL) cooled over ice to about 5 ℃. Layers were separated and the organic extract was washed with water (2 × 30 mL). Combined aqueous layers were back extracted with Et_2O (30 mL). Combined organic layers were dried over Na_2SO_4 and filtered. Then solvent was removed in vacuo to leave a crude acetylenicaldehyde.

The crude product was then dissolved in THF (8 mL), and 1,3-dicarbonyl compounds (10 mmol) were added into the solution. Then HOAc (2 mmol), $MgSO_4$ (2 mmol) and piperidine (1 mmol) were added to the reaction mixture. The mixture was stirred at room temperature for about 1 hour. When the reaction was completed as monitored by TLC, filtration through celite and removal of the solvent by rotary evaporation gave the crude product. The conjugated enynones were purified by chromatography on silica gel with the appropriate mixture of PE and EA (PE = petroleum ether, EA = ethyl acetate). The unsymmetric 1,3-dicarbonyl compounds would afford conjugated enynones as a mixture of *E*- and *Z*-isomer. The conjugated enynones need to be kept in refrigerator below 0 ℃.



Conjugated enynones **1m-1n** were prepared according to the literature report.[2] The alkyne (10 mmol) was dissolved in dry THF (15 mL) and the solution was cooled to −40 ℃ under nitrogen, *n*-butyllithium (1.6 M in hexanes, 6.8 mL, 11 mmol) was added dropwise over *ca.* 2 minutes while maintaining the temperature between −35 and −40 ℃. After completion of the addition, anhydrous DMF (1.55 mL, 20 mmol) was added in one portion and the cold bath was removed. The reaction mixture was allowed to warm to room temperature and aged for 30 minutes. The THF solution was poured into a vigorously stirred biphasic solution prepared from aqueous solution of KH_2PO_4 (50 mL, 30 mmol) and Et_2O (30 mL) cooled over ice to about 5 ℃. Layers were separated and the organic extract was washed with water (2 × 30 mL). Combined aqueous layers were back extracted with Et_2O (30 mL). Combined organic layers were dried over Na_2SO_4 and filtered. Then solvent was removed in vacuo to leave a crude acetylenicaldehyde.

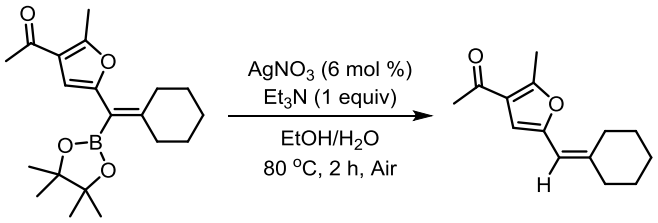
The crude product was then dissolved in toluene (15 mL), and carbonyl compounds (10 mmol) were added into the solution. Then ethylenediamine (0.5 mmol) and HOAc (1 mmol) were added to the reaction mixture. The mixture was stirred at room temperature for about 3 hours. When the reaction was completed as monitored by TLC, filtration through celite and removal of the solvent by rotary evaporation gave the crude product. The conjugated enynones were purified by chromatography on silica gel with the appropriate mixture of PE and EA (PE = petroleum ether, EA = ethyl acetate).

3. General Procedure for Pd-Catalyzed Oxidative Borylation Reaction of Conjugated Enynones

$Pd(OAc)_2$ (0.9 mg, 0.004 mmol, 2 mol %), PPh_3 (2.6 mg, 0.01 mmol, 5 mol %), 2,6-dimethyl-1,4-benzoquinone (54.4 mg, 0.4 mmol, 2 equiv), diboron compound (**2**, 0.4 mmol, 2 equiv) were added to a flame-dried 10 mL Schleck reaction tube. The reaction tube was degassed three times with nitrogen, then MeOH (HPLC grade, 4 mL) was added using a syringe. $^{i}Pr_2NEt$ (51.6 mg, 0.4 mmol, 2 equiv), conjugated enynone (**1**, 0.2 mmol) were added by syringe successively. The reaction tube was stirred at 40 °C for 10 h, then cooled to room temperature. The mixture was filtered through a short plug of silica gel and washed with EtOAc as the eluent. Solvent was then removed in vacuo to leave a crude mixture, which was purified by silica gel column chromatography to afford pure product alkenylboronates **3** or **4**.

4. Procedure for Transformations of Alkenylboronate 3q

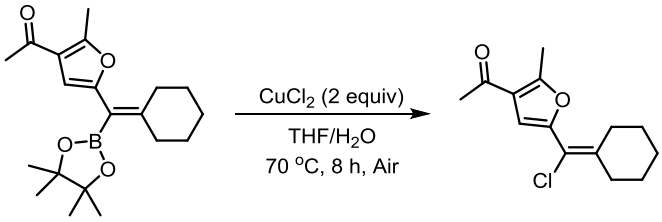
Procedure for Protodeboronation of 3q



3q **5a**, 77%

According to the literature procedure,[3] a mixture of **3q** (34.4 mg, 0.1 mmol), $AgNO_3$ (1.0 mg, 0.006 mmol, 6 mol %), NEt_3 (10.1 mg, 0.1 mmol) and $EtOH/H_2O$ (0.5 mL/0.5 mL) was stirred at 80 ℃ for 3 h under air atmosphere. After the completion of reaction, the reaction mixture was quenched with brine (5 mL) and extracted with ethyl acetate (10 mL × 3). The combined organic layer was dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The crude product was then purified by column chromatography on silica gel to afford compound **5a** (16.8 mg, 77%).

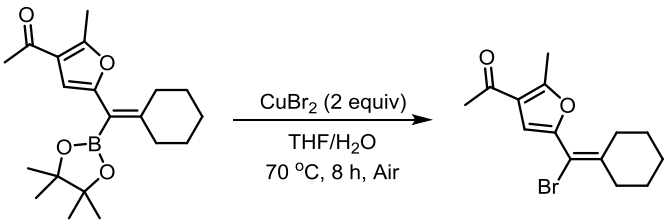
Procedure for Chlorination of 3q



3q **5b**, 82%

According to the literature procedure,[4] a mixture of **3q** (34.4 mg, 0.1 mmol), $CuCl_2$ (26.9 mg, 0.2 mmol) and THF/H_2O (0.5 mL/0.5 mL) was stirred at 70 ℃ for 8 h at air atmosphere. After the completion of reaction, the reaction mixture was quenched with brine (5 mL) and extracted with ethyl acetate (10 mL × 3). The combined organic layer was dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The crude product was then purified by column chromatography on silica gel to afford compound **5b** (20.6 mg, 82%).

Procedure for Bromination of 3q

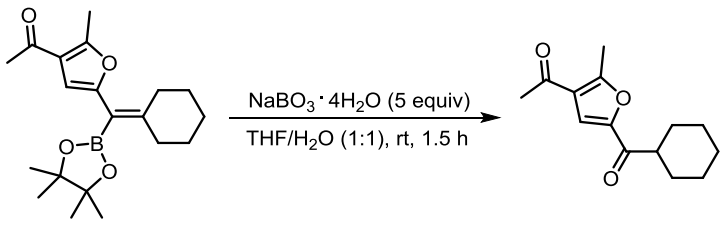


3q **5c**, 75%

According to the literature procedure,[4] a mixture of **3q** (34.4 mg, 0.1 mmol), $CuBr_2$ (44.7 mg, 0.2 mmol) and THF/H_2O (0.5 mL/0.5 mL) was stirred at 70 ℃ for 8 h at air atmosphere. After the completion of reaction, the reaction mixture was quenched with brine (5 mL) and extracted with ethyl acetate (10 mL × 3). The combined organic layer was dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The crude product was then

purified by column chromatography on silica gel to afford compound **5c** (22.0 mg, 75%).

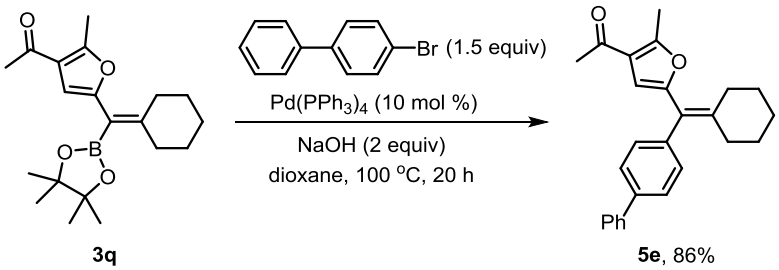
Procedure for oxidation of 3q



3q **5d**, 73%

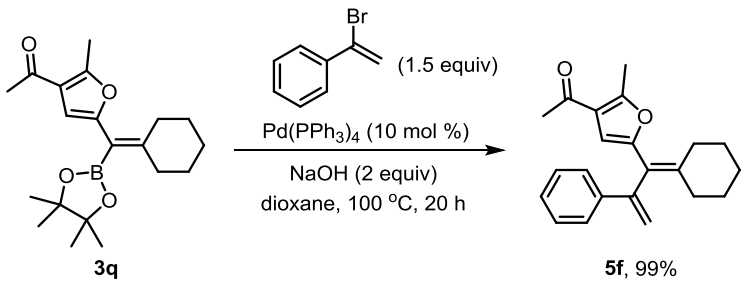
According to the literature procedure,[5] a mixture of **3q** (62.5 mg, 0.18 mmol), $NaBO_3 \cdot 4H_2O$ (138.5 mg, 0.9 mmol) and THF/H_2O (2 mL/2 mL) was stirred at room temperature for 1.5 h. After the completion of reaction, the reaction mixture was quenched with brine (5 mL) and extracted with ethyl acetate (10 mL × 3). The combined organic layer was dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The crude product was then purified by column chromatography on silica gel to afford compound **5d** (30.6 mg, 73%).

Procedure for Suzuki coupling of 3q



3q **5e**, 86%

According to the literature procedure,[6] $Pd(PPh_3)_4$ (11.6 mg, 0.01 mmol) and 4-bromo-1,1'-biphenyl (35.0 mg, 0.15 mmol) were added to a flame-dried 10 mL Schleck reaction tube. The reaction tube was degassed three times with nitrogen, and dioxane (1 mL) was added using a syringe. Then 2 M NaOH aqueous solution (100 μL, 0.2 mmol) and **3q** (34.4 mg, 0.1 mmol) were added by syringe successively. The reaction tube was stirred at 100 ℃ for 20 h, then cooled to room temperature. The mixture was then filtered through a short plug of silica gel and washed with PE/EA = 10/1 as the eluent. Solvent was then removed in vacuo to leave a crude mixture, which was purified by silica gel column chromatography to afford **5e** (32.0 mg, 86%).

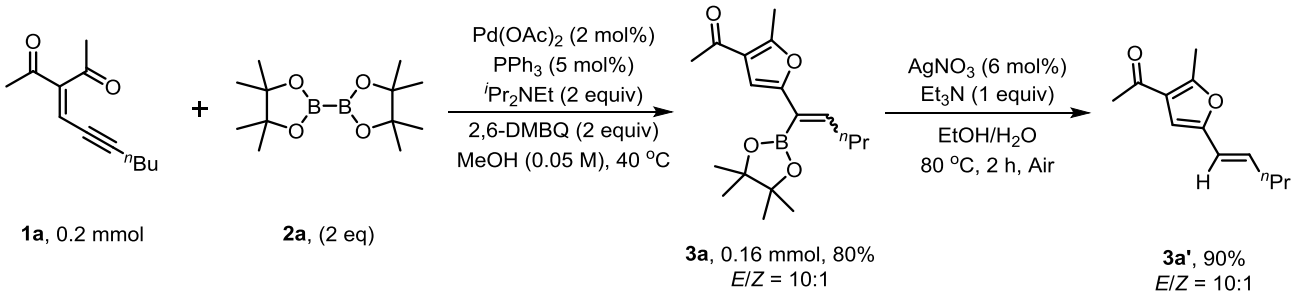


3q **5f**, 99%

According to the literature procedure,[6] $Pd(PPh_3)_4$ (11.6 mg, 0.01 mmol) was added to a flame-dried 10 mL Schleck reaction tube. The reaction tube was degassed with nitrogen, and dioxane (1 mL) was added using a syringe. Then 2 M NaOH aqueous solution (100 μL, 0.2 mmol), (1-bromovinyl)benzene (27.5 mg, 0.15 mmol) and

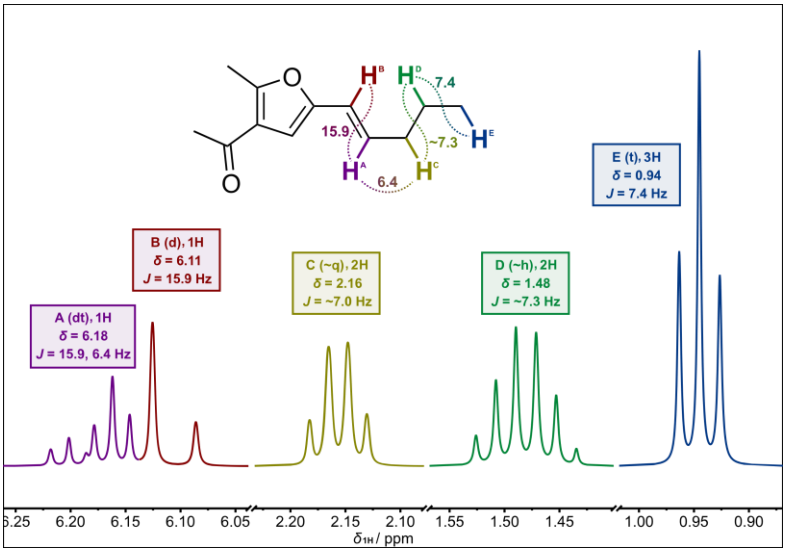
3q (34.4 mg, 0.1 mmol) were added successively. The reaction tube was stirred at 100 ℃ for 20 h, then cooled to room temperature. The mixture was then filtered through a short plug of silica gel and washed with PE/EA = 10:1 as the eluent. Solvent was then removed in vacuo, and the residue was purified by silica gel chromatography to afford **5f** (31.9 mg, 99%).

5. Double-Bond Configuration of the Products



1a, 0.2 mmol　　**2a**, (2 eq)　　**3a**, 0.16 mmol, 80%, *E*/*Z* = 10:1　　**3a'**, 90%, *E*/*Z* = 10:1

According to the general procedure for palladium-catalyzed oxidative borylation reaction, **3a** (50.7 mg, 0.16 mmol) was synthesized by **1a** (0.2 mmol) and **2a** (0.4 mmol). To identify the double-bond configuration of **3a**, subsequent protodeboronation was carried out to afford **3a′** (27.6 mg) in 90% yield according to the procedure (Supplement **4a**).

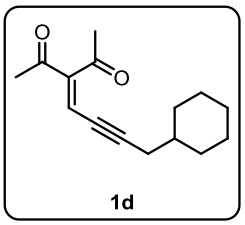


Then we analyzed the ^{1}H-NMR spectrum of **3a′**. The sp^3–sp^3 $J_{\mathbf{D-E}}$ and $J_{\mathbf{C-D}}$ on the propyl group are about 7.3 Hz, and the sp^2–sp^3 J_{A-C} between the vinyl proton **A** and the adjacent methylene **C** is 6.4 Hz, which are all typical values. The chemical shifts of **A** and **B** are close enough to cause strong second-order coupling, resulting in the "roof-like" peaks. The $J_{\mathbf{A-B}}$ value is 15.9 Hz, indicating that **3a′** is a *trans*-disubstituted alkene. Considering that the protodeboronation would resist the double-bond configuration,[3] we could judge that boronate group is *cis* to the alkyl group.

6. Characterization Data

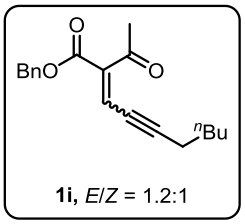
Characterization Data of Unknown Conjugated Enynones

3-(4-Cyclohexylbut-2-yn-1-ylidene)pentane-2,4-dione (1d)



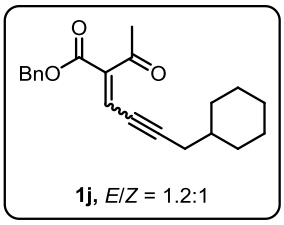
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.71 (t, J = 2.6 Hz, 1H), 2.47 (s, 3H), 2.34 (dd, J = 6.6, 2.5 Hz, 2H), 2.32 (s, 3H), 1.82 – 1.62 (m, 5H), 1.59 − 1.48 (m, 1H), 1.33 – 1.11 (m, 3H), 1.07 − 0.94 (m, 2H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 201.31, 195.75, 149.40, 123.22, 109.57, 77.59, 37.07, 32.60, 30.87, 27.90, 27.07, 25.99, 25.92; **HRMS (ESI, *m*/*z*):** calcd for $C_{15}H_{21}O_2$ $[M+H]^+$ 233.1536, found 233.1534; **IR (film):** 2926, 2853, 2209, 1716, 1690, 1665, 1588, 1449, 1420, 1376, 1248, 1164, 1155 cm^{-1}

Benzyl 2-acetyldec-2-en-4-ynoate (1i)



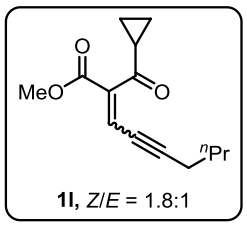
^{1}H NMR (400 MHz, $CDCl_3$) Major isomer: δ 7.46 – 7.29 (m, overlapping, 5H), 6.81 (t, J = 2.4 Hz, 1H), 5.31 (s, 2H), 2.32 (s, 3H), 2.30 (td, J = 7.2, 2.4 Hz, 2H), 1.51 – 1.42 (m, 2H), 1.41 – 1.22 (m, overlapping, 4H), 0.94 – 0.85 (t, J = 7.0 Hz, overlapping, 3H); Minor isomer: δ 7.46 – 7.29 (m, overlapping, 5H), 6.84 (t, J = 2.4 Hz, 1H), 5.23 (s, 2H), 2.44 (s, 3H), 2.41 (td, J = 7.2, 2.4 Hz, 2H), 1.59 – 1.51 (m, 2H), 1.41 – 1.22 (m, overlapping, 4H), 0.94 – 0.85 (t, J = 7.0 Hz, overlapping, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.81, 193.93, 165.39, 163.79, 141.69, 140.99, 135.26, 135.23, 128.58, 128.50, 128.36, 128.32, 128.10, 125.97, 124.60, 110.38, 109.01, 76.86, 76.55, 67.10, 67.07, 30.94, 30.91, 30.43, 27.71, 27.34, 22.05, 20.11, 20.04, 13.85; **HRMS (ESI, *m*/*z*):** calcd for $C_{19}H_{23}O_3$ $[M+H]^+$ 299.1642, found 299.1639; **IR (film):** 2954, 2926, 2874, 2852, 2212, 1716, 1671, 1594, 1455, 1363, 1248, 1202, 1161, 1059, 757, 747, 698 cm^{-1}.

Benzyl 2-acetyl-6-cyclohexylhex-2-en-4-ynoate (1j)



^{1}H NMR (400 MHz, $CDCl_3$) Major isomer: δ 7.44 – 7.30 (m, over-lapping, 5H), 6.81 (t, J = 2.4 Hz, 1H), 5.31 (s, 2H), 2.32 (s, 3H), 2.21 (dd, J = 6.4, 2.4 Hz, 2H), 1.82 – 1.60 (m, overlapping, 5H), 1.56 – 1.38 (m, overlapping, 1H), 1.30 – 1.07 (m, overlapping, 3H), 1.06 – 0.90 (m, overlapping, 2H); Minor isomer: δ 7.44 – 7.30 (m, overlapping, 5H), 6.85 (t, J = 2.4 Hz, 1H), 5.23 (s, 2H), 2.44 (s, 3H), 2.31 (dd, J = 6.4, 2.4 Hz, overlapping, 2H), 1.82 – 1.60 (m, overlapping, 5H), 1.56 – 1.38 (m, overlapping, 1H), 1.30 – 1.07 (m, overlapping, 3H), 1.06 – 0.90 (m, overlapping, 2H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.84, 193.92, 165.45, 163.81, 141.61, 141.04, 135.25, 128.59, 128.52, 128.38, 128.33, 128.11, 125.93, 124.66, 109.46, 108.15, 77.74, 77.42, 67.11, 67.07, 37.04, 32.59, 30.47, 27.91, 27.87, 27.31, 26.03, 25.96; **HRMS (ESI, *m*/*z*):** calcd for $C_{21}H_{25}O_3$ $[M+H]^+$ 325.1798, found 325.1797; **IR (film):** 2924, 2849, 2215, 1719, 1674, 1600, 1450, 1363, 1247, 1207, 1161, 1066 cm^{-1}.

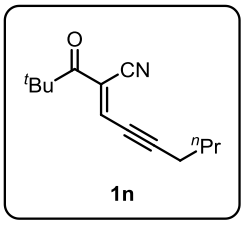
Methyl 2-(cyclopropanecarbonyl)non-2-en-4-ynoate (1l)



1l, *Z*/*E* = 1.8:1

^{1}H NMR (400 MHz, $CDCl_3$) Major isomer: δ 6.86 (t, J = 2.4 Hz, 1H), 3.80 (s, 3H), 2.41 (td, J = 7.0, 2.4 Hz, 2H), 2.31 – 2.19 (m, overlapping, 1H), 1.60 – 1.49 (m, overlapping, 2H), 1.47 – 1.35 (m, overlapping, 2H), 1.26 – 1.20 (m, 2H), 1.05 – 0.97 (m, overlapping, 2H), 0.92 (t, J = 7.2 Hz, overlapping, 3H); Minor isomer: δ 6.82 (t, J = 2.4 Hz, 1H), 3.88 (s, 3H), 2.46 (td, J = 7.0, 2.4 Hz, 2H), 2.31 – 2.19 (m, overlapping, 1H), 1.60 – 1.49 (m, overlapping, 2H), 1.47 – 1.35 (m, overlapping, 2H), 1.19 – 1.14 (m, 2H), 1.05 – 0.97 (m, overlapping, 2H), 0.93 (t, J = 7.2 Hz, overlapping, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 201.69, 196.45, 166.15, 164.55, 142.00, 141.57, 124.77, 124.20, 109.22, 107.96, 76.94, 76.57, 52.37, 52.17, 30.16, 30.12, 21.97, 21.81, 21.76, 19.84, 19.71, 18.33, 13.42, 12.35, 12.30; **HRMS (ESI, *m*/*z*):** calcd for $C_{14}H_{19}O_3$ $[M+H]^+$ 235.1329, found 235.1327; **IR (film):** 2961, 2933, 2865, 2211, 1723, 1691, 1597, 1436, 1389, 1258, 1170, 1137, 1039, 1008 cm^{-1}.

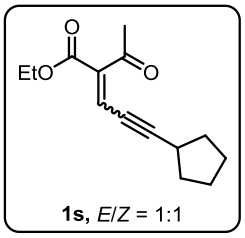
(*E*)-2-Pivaloylnon-2-en-4-ynenitrile (1n)



1n

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (t, J = 2.4 Hz, 1H), 2.56 (td, J = 7.0, 2.4 Hz, 2H), 1.68 – 1.58 (m, 2H), 1.54 – 1.42 (m, 2H), 1.35 (s, 9H), 0.95 (t, J = 7.3 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 196.43, 139.83, 118.51, 116.52, 115.37, 77.92, 44.01, 29.96, 26.00, 21.87, 20.18, 13.43; **HRMS (ESI, *m*/*z*):** calcd for $C_{14}H_{20}NO$ $[M+H]^+$ 218.1539, found 218.1537; **IR (film):** 2962, 2874, 2206, 1694, 1556, 1479, 1368, 1275, 1143, 962 cm^{-1}.

Ethyl 2-acetyl-5-cyclopentylpent-2-en-4-ynoate (1s)

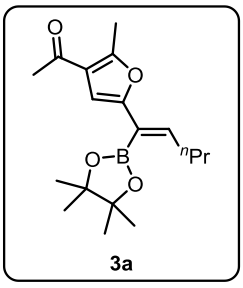


1s, *E*/*Z* = 1:1

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.82 (d, J = 2.4 Hz, 1H), 6.80 (d, J = 2.4 Hz, 1H), 4.34 (q, J = 7.2 Hz, 2H), 4.25 (q, J = 7.2 Hz, 2H), 2.90 – 2.79 (m, 2H), 2.45 (s, 3H), 2.35 (s, 3H), 2.02 – 1.90 (m, 4H), 1.77 – 1.56 (m, 12H), 1.36 (t, J = 7.2 Hz, 3H), 1.30 (t, J = 7.2 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.09, 193.97, 165.60, 163.97, 141.81, 141.30, 125.50, 124.08, 113.86, 112.57, 76.44, 76.16, 61.41, 61.35, 33.43, 33.38, 31.33, 31.21, 30.37, 27.24, 25.01, 24.97, 14.09, 14.02; **HRMS (ESI, *m*/*z*):** calcd for $C_{14}H_{19}O_3$ $[M+H]^+$ 235.1329, found 235.1328; **IR (film):** 2968, 2207, 1719, 1671, 1598, 1373, 1251, 1212, 1064 cm^{-1}.

Characterization Data of the Products

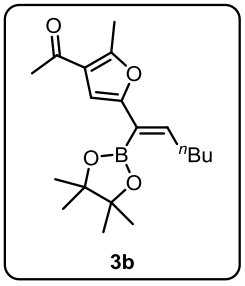
(*E*)-1-(2-Methyl-5-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pent-1-en-1-yl)furan-3-yl)ethan-1-one (3a)



3a

Yield: 80% (50.9 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.80 (t, J = 7.9 Hz, 1H), 6.57 (s, 1H), 2.57 (s, 3H), 2.45 (q, J = 7.4 Hz, 2H), 2.39 (s, 3H), 1.49 (h, J = 7.3 Hz, 2H), 1.35 (s, 12H), 0.94 (t, J = 7.4 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 194.44, 156.94, 153.00, 144.98, 122.79, 106.87, 83.53, 33.25, 29.01, 24.76, 22.94, 14.35, 13.74; **^{11}B NMR (160 MHz, $CDCl_3$)** δ 29.88; **HRMS (ESI, *m*/*z*):** calcd for $C_{18}H_{28}BO_4$ $[M+H]^+$ 319.2075, found 319.2071; **IR (film):** 2979, 2960, 2929, 2870, 1677, 1582, 1543, 1416, 1391, 1315, 1265, 1230, 1144, 1010, 951, 854 cm^{-1}.

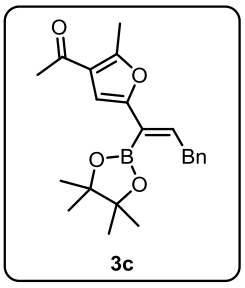
(*E*)-1-(2-Methyl-5-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hex-1-en-1-yl)furan-3-yl)ethan-1-one (3b)



3b

Yield: 80% (53.1 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.80 (t, J = 7.9 Hz, 1H), 6.57 (s, 1H), 2.57 (s, 3H), 2.47 (q, J = 7.5 Hz, 2H), 2.39 (s, 3H), 1.51 – 1.37 (m, 4H), 1.35 (s, 12H), 0.92 (t, J = 7.1 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 194.42, 156.91, 153.01, 145.30, 122.79, 106.83, 83.51, 31.88, 30.94, 29.00, 24.76, 22.26, 14.34, 13.86; **HRMS (ESI, *m*/*z*):** calcd for $C_{19}H_{30}BO_4$ $[M+H]^+$ 333.2232, found 333.2229; **IR (film):** 2976, 2961, 2927, 2865, 1677, 1581, 1414, 1263, 1142, 1015, 950, 855 cm^{-1}.

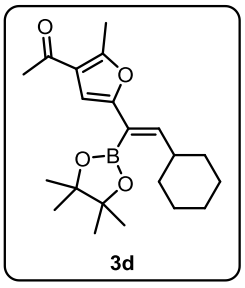
(*E*)-1-(2-Methyl-5-(3-phenyl-1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl)furan-3-yl)ethan-1-one (3c)



3c

Yield: 37% (27.3 mg); yellow solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.33 – 7.24 (m, 4H), 7.23 – 7.18 (m, 1H), 6.90 (t, J = 8.0 Hz, 1H), 6.66 (s, 1H), 3.84 (d, J = 8.0 Hz, 2H), 2.53 (s, 3H), 2.39 (s, 3H), 1.37 (s, 12H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 194.42, 157.19, 152.68, 142.90, 140.43, 128.73, 128.48, 126.08, 122.87, 107.74, 83.78, 37.58, 29.04, 24.84, 14.36; **HRMS (ESI, *m*/*z*):** calcd for $C_{22}H_{28}BO_4$ $[M+H]^+$ 367.2075, found 367.2071; **IR (film):** 2995, 2981, 1677, 1582, 1415, 1263, 1145, 754 cm^{-1}.

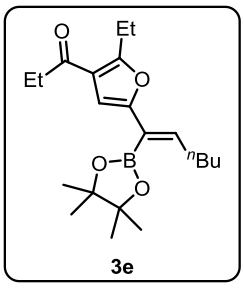
(*E*)-1-(5-(2-Cyclohexyl-1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)vinyl)-2-methylfuran-3-yl)ethan-1-one (3d)



3d

Yield: 80% (57.3 mg); yellow solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.60 (d, J = 9.7 Hz, 1H), 6.54 (s, 1H), 2.74 – 2.64 (m, 1H), 2.57 (s, 3H), 2.38 (s, 3H), 1.78 – 1.64 (m, 5H), 1.35 (s, 12H), 1.30 – 1.10 (m, 5H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 194.41, 156.95, 152.95, 149.94, 122.77, 106.93, 83.52, 39.98, 33.40, 29.00, 25.87, 25.78, 24.71, 14.35; **HRMS (ESI, *m*/*z*):** calcd for $C_{21}H_{32}BO_4$ $[M+H]^+$ 359.2388, found 359.2387; **IR (film):** 2979, 2927, 2847, 1678, 1583, 1416, 1391, 1269, 1229, 1143, 855 cm^{-1}.

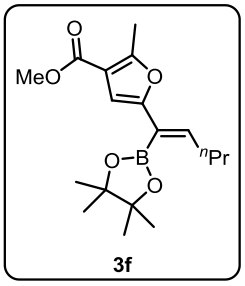
(*E*)-1-(2-Ethyl-5-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hex-1-en-1-yl)furan-3-yl)propan-1-one (3e)



3e

Yield: 81% (58.3 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.80 (t, J = 7.9 Hz, 1H), 6.58 (s, 1H), 3.00 (q, J = 7.6 Hz, 2H), 2.73 (q, J = 7.3 Hz, 2H), 2.47 (q, J = 7.5 Hz, 2H), 1.51 – 1.37 (m, 4H), 1.35 (s, 12H), 1.24 (t, J = 7.7 Hz, 3H), 1.15 (t, J = 7.3 Hz, 3H), 0.92 (t, J = 7.2 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 197.34, 161.82, 152.91, 145.11, 121.26, 106.40, 83.52, 34.17, 31.94, 31.02, 24.79, 22.31, 21.66, 13.88, 12.13, 7.78; **HRMS (ESI, *m*/*z*):** calcd for $C_{21}H_{34}BO_4$ $[M+H]^+$ 361.2545, found 361.2541; **IR (film):** 2976, 2930, 1679, 1579, 1416, 1373, 1215, 1143, 924, 859 cm^{-1}.

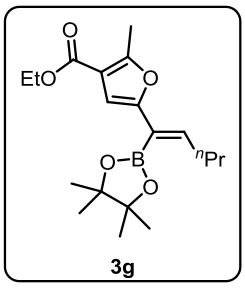
Methyl (*E*)-2-methyl-5-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pent-1-en-1-yl)furan-3-carboxylate (3f)



3f

Yield: 66% (44.2 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.78 (t, J = 7.9 Hz, 1H), 6.58 (s, 1H), 3.81 (s, 3H), 2.56 (s, 3H), 2.44 (q, J = 7.6 Hz, 2H), 1.48 (h, J = 7.3 Hz, 2H), 1.34 (s, 12H), 0.94 (t, J = 7.3 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 164.85, 157.78, 153.14, 144.58, 114.53, 107.08, 83.53, 51.12, 33.27, 24.77, 22.99, 13.79, 13.76; **HRMS (ESI, *m*/*z*):** calcd for $C_{18}H_{28}BO_5$ $[M+H]^+$ 335.2024, found 335.2025; **IR (film):** 2972, 2955, 1719, 1602, 1439, 1416, 1372, 1271, 1230, 1144, 1089, 778 cm^{-1}.

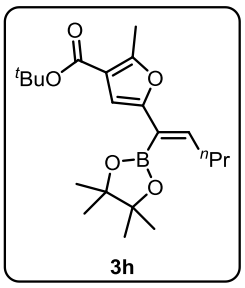
Ethyl (*E*)-2-methyl-5-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pent-1-en-1-yl)furan-3-carboxylate (3g)



3g

Yield: 55% (38.3 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.77 (t, J = 7.9 Hz, 1H), 6.59 (s, 1H), 4.27 (q, J = 7.2 Hz, 2H), 2.55 (s, 3H), 2.43 (q, J = 7.5 Hz, 2H), 1.48 (h, J = 7.3 Hz, 2H), 1.36 – 1.31 (m, 15H), 0.94 (t, J = 7.4 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 164.40, 157.52, 153.07, 144.38, 114.86, 107.16, 83.51, 59.86, 33.28, 24.75, 22.98, 14.33, 13.81, 13.76; **HRMS (ESI, *m*/*z*):** calcd for $C_{19}H_{30}BO_5$ $[M+H]^+$ 349.2181, found 349.2182; **IR (film):** 2979, 2930, 2871, 1716, 1600, 1416, 1372, 1270, 1228, 1211, 1144, 1085, 855, 778 cm^{-1}.

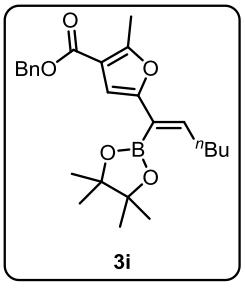
***t*-Butyl (*E*)-2-methyl-5-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pent-1-en-1-yl)furan-3-carboxylate (3h)**



3h

Yield: 61% (45.9 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.74 (t, J = 7.9 Hz, 1H), 6.55 (s, 1H), 2.52 (s, 3H), 2.42 (q, J = 7.5 Hz, 2H), 1.54 (s, 9H), 1.47 (h, J = 7.4 Hz, 2H), 1.33 (s, 12H), 0.93 (t, J = 7.4 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.83, 156.81, 152.81, 143.93, 116.32, 107.63, 83.53, 80.16, 33.32, 28.30, 24.78, 23.02, 13.88, 13.79; **HRMS (ESI, *m*/*z*):** calcd for $C_{21}H_{34}BO_5$ $[M+H]^+$ 377.2494, found 377.2490; **IR (film):** 2979, 1712, 1603, 1417, 1371, 1288, 1231, 1172, 1145, 1089 cm^{-1}.

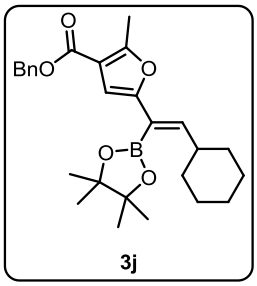
Benzyl (*E*)-2-methyl-5-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hex-1-en-1-yl)furan-3-carboxylate (3i)



3i

Yield: 81% (68.6 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.42 – 7.30 (m, 5H), 6.77 (t, J = 7.9 Hz, 1H), 6.64 (s, 1H), 5.28 (s, 2H), 2.55 (s, 3H), 2.45 (q, J = 7.5 Hz, 2H), 1.46 – 1.34 (m, 4H), 1.32 (s, 12H), 0.91 (t, J = 7.2 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 164.12, 157.86, 153.21, 144.85, 136.45, 128.44, 127.91, 127.77, 114.55, 107.18, 83.51, 65.56, 31.90, 30.97, 24.75, 22.28, 13.91, 13.88; **HRMS (ESI, *m*/*z*):** calcd for $C_{25}H_{34}BO_5$ $[M+H]^+$ 425.2494, found 425.2490; **IR (film):** 2978, 2958, 2926, 1717, 1600, 1415, 1267, 1216, 1143, 1085, 1076, 851, 775, 698 cm^{-1}.

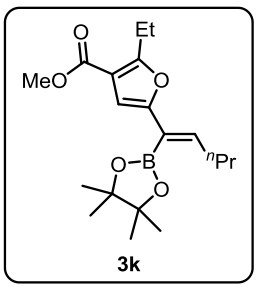
Benzyl (*E*)-5-(2-cyclohexyl-1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)vinyl)-2-methylfuran-3-carboxylate (3j)



3j

Yield: 79% (71.1 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.42 – 7.31 (m, 5H), 6.60 (s, 1H), 6.58 (d, J = 9.7 Hz, 1H), 5.27 (s, 2H), 2.72 – 2.61 (m, 1H), 2.55 (s, 3H), 1.78 – 1.62 (m, 5H), 1.33 (s, 12H), 1.26 – 1.08 (m, 5H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 164.13, 157.92, 153.17, 149.50, 136.47, 128.46, 127.93, 127.77, 114.56, 107.29, 83.54, 65.58, 40.03, 33.45, 25.91, 25.82, 24.73, 13.94; **HRMS (ESI, *m*/*z*):** calcd for $C_{27}H_{36}BO_5$ $[M+H]^+$ 451.2650, found 451.2644; **IR (film):** 2973, 2927, 2846, 1717, 1600, 1414, 1269, 1225, 1207, 1142, 1084, 1074, 775 cm^{-1}.

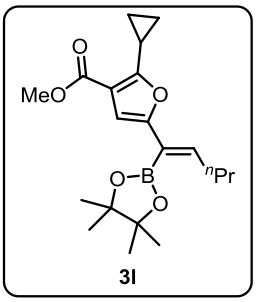
Methyl (*E*)-2-ethyl-5-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pent-1-en-1-yl)furan-3-carboxylate (3k)



3k

Yield: 70% (48.7 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.78 (t, J = 7.9 Hz, 1H), 6.58 (s, 1H), 3.80 (s, 3H), 2.98 (q, J = 7.6 Hz, 2H), 2.44 (q, J = 7.6 Hz, 2H), 1.48 (q, J = 7.4 Hz, 2H), 1.34 (s, 12H), 1.24 (t, J = 7.5 Hz, 3H), 0.94 (t, J = 7.4 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 164.77, 162.71, 153.06, 144.57, 113.57, 107.04, 83.53, 51.11, 33.31, 24.78, 23.02, 21.21, 13.80, 12.42; **HRMS (ESI, *m*/*z*):** calcd for $C_{19}H_{30}BO_5$ $[M+H]^+$ 349.2181, found 349.2181; **IR (film):** 2979, 1719, 1597, 1438, 1417, 1312, 1248, 1215, 1145, 1094, 1044 cm^{-1}.

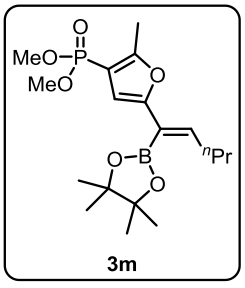
Methyl (*E*)-2-cyclopropyl-5-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pent-1-en-1-yl)furan-3-carboxylate (3l)



3l

Yield: 51% (36.7 mg); yellow oil; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 6.61 (t, J = 7.9 Hz, 1H), 6.56 (s, 1H), 3.82 (s, 3H), 2.79 – 2.71 (m, 1H), 2.40 (q, J = 7.6 Hz, 2H), 1.46 (h, J = 7.3 Hz, 2H), 1.33 (s, 12H), 1.07 – 1.00 (m, 4H), 0.92 (t, J = 7.4 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 165.11, 161.81, 151.89, 144.07, 114.05, 107.26, 83.52, 51.10, 33.29, 24.77, 23.04, 13.78, 9.30, 8.81; **HRMS (ESI, *m*/*z*):** calcd for $C_{20}H_{30}BO_5$ $[M+H]^+$ 361.2181, found 361.2184; **IR (film):** 2972, 2958, 1717, 1594, 1441, 1230, 1145, 1071 cm^{-1}.

Dimethyl (*E*)-(2-methyl-5-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pent-1-en-1-yl)furan-3-yl) phosphonate (3m)

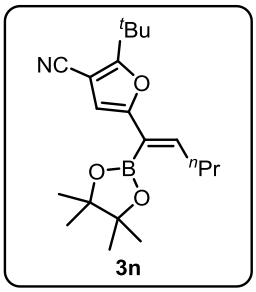


3m

Yield: 99% (76.0 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.80 (t, J = 7.9 Hz, 1H), 6.41 (d, J = 3.3 Hz, 1H), 3.74 (s, 3H), 3.71 (s, 3H), 2.50 (d, J = 2.1 Hz, 3H), 2.44 (q, J = 7.5 Hz, 2H), 1.48 (h, J = 7.4 Hz, 2H), 1.33 (s, 12H), 0.94 (t, J = 7.4 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 159.17 (d, J = 26.7 Hz), 154.38 (d, J = 15.3 Hz), 145.15, 108.52 (d, J = 12.1 Hz), 106.52 (d, J = 215.3 Hz), 83.57, 52.32, 52.26, 33.29, 24.79, 22.98, 13.79, 13.50; **HRMS (ESI, *m*/*z*):** calcd for $C_{18}H_{31}BO_6P$ $[M+H]^+$ 385.1946, found 385.1952; **IR (film):**

2975, 1585, 1417, 1346, 1242, 1144, 1056, 1030, 832, 785 cm^{-1}.

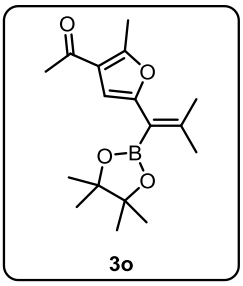
(*E*)-2-(*tert*-butyl)-5-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pent-1-en-1-yl)furan-3-carbonitrile (3n)



3n

Yield: 45% (30.9 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.81 (t, J = 7.9 Hz, 1H), 6.48 (s, 1H), 2.46 (q, J = 7.6 Hz, 2H), 1.51 – 1.44 (m, 2H), 1.42 (s, 9H), 1.33 (s, 12H), 0.94 (t, J = 7.4 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 169.24, 153.55, 146.77, 115.50, 108.52, 91.62, 83.66, 34.72, 33.23, 28.88, 24.79, 22.92, 13.77; **^{11}B NMR (160 MHz, $CDCl_3$)** δ 29.77; **HRMS (ESI, *m*/*z*):** calcd for $C_{20}H_{31}BNO_3$ $[M+H]^+$ 344.2392, found 344.2387; **IR (film):** 2983, 2232, 1417, 1372, 1317, 1249, 1144, 980, 854 cm^{-1}.

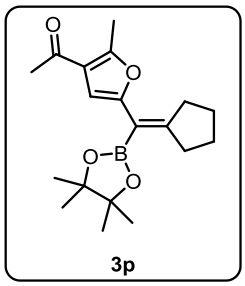
1-(2-Methyl-5-(2-methyl-1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl)furan-3-yl)ethan-1-one (3o)



3o

Yield: 89% (54.1 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.34 (s, 1H), 2.55 (s, 3H), 2.39 (s, 3H), 2.06 (s, 3H), 1.97 (s, 3H), 1.33 (s, 12H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 194.35, 156.33, 152.92, 149.75, 122.43, 107.85, 83.62, 29.04, 25.56, 24.60, 23.37, 14.35; **HRMS (ESI, *m*/*z*):** calcd for $C_{17}H_{26}BO_4$ $[M+H]^+$ 305.1919, found 305.1917; **IR (film):** 2983, 2232, 1417, 1372, 1317, 1249, 1144, 980, 854 cm^{-1}.

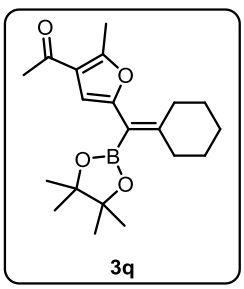
1-(5-(Cyclopentylidene(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)-2-methylfuran-3-yl)ethan-1-one (3p)



3p

Yield: 82% (54.3 mg); yellow solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.45 (s, 1H), 2.66 – 2.58 (m, 4H), 2.56 (s, 3H), 2.39 (s, 3H), 1.75 – 1.67 (m, 4H), 1.34 (s, 12H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 194.52, 161.89, 156.11, 153.58, 122.49, 107.39, 83.42, 35.14, 34.83, 29.05, 26.38, 26.28, 24.72, 14.45; **HRMS (ESI, *m*/*z*):** calcd for $C_{19}H_{28}BO_4$ $[M+H]^+$ 331.2075, found 331.2075; **IR (film):** 2975, 2952, 1677, 1581, 1398, 1368, 1306, 1237, 1144, 1011, 961, 858 cm^{-1}.

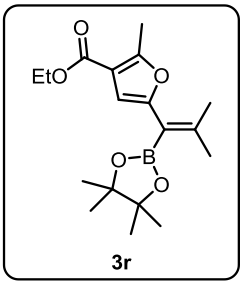
1-(5-(Cyclohexylidene(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)-2-methylfuran-3-yl)ethan-1-one (3q)



3q

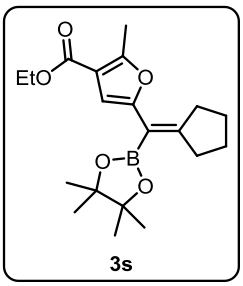
Yield: 98% (67.6 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.31 (s, 1H), 2.55 (s, 3H), 2.48 – 2.42 (m, 4H), 2.38 (s, 3H), 1.70 – 1.58 (m, 6H), 1.31 (s, 12H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 194.30, 158.29, 156.52, 152.46, 122.36, 107.67, 83.55, 35.71, 32.62, 29.02, 28.59, 27.98, 26.34, 24.53, 14.34; **HRMS (ESI, *m*/*z*):** calcd for $C_{20}H_{30}BO_4$ $[M+H]^+$ 345.2232, found 345.2231; **IR (film):** 2975, 2926, 2852, 1677, 1582, 1390, 1356, 1329, 1304, 1232, 1145, 1009, 952, 854 cm^{-1}.

Ethyl 2-methyl-5-(2-methyl-1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-en-1-yl)furan-3-carboxylate (3r)



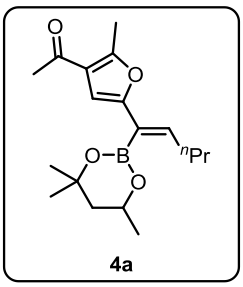
Yield: 67% (44.8 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.37 (s, 1H), 4.27 (q, J = 7.1 Hz, 2H), 2.53 (s, 3H), 2.04 (s, 3H), 1.96 (s, 3H), 1.34 – 1.31 (m, 15H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 164.45, 156.96, 152.99, 149.20, 114.42, 108.13, 83.61, 59.87, 25.61, 24.63, 23.34, 14.37, 13.77; **HRMS (ESI, *m*/*z*):** calcd for $C_{18}H_{28}BO_5$ $[M+H]^+$ 335.2024, found 335.2026; **IR (film):** 2979, 2933, 1715, 1602, 1372, 1346, 1306, 1233, 1145, 1092, 856, 778 cm^{-1}.

Ethyl 5-(cyclopentylidene(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)-2-methylfuran-3-carboxylate (3s)



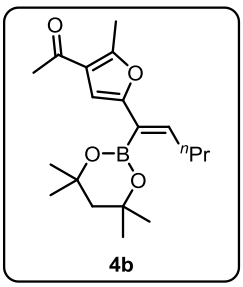
Yield: 63% (45.3 mg); yellow solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.46 (s, 1H), 4.27 (q, J = 7.1 Hz, 2H), 2.64 – 2.56 (m, 4H), 2.54 (s, 3H), 1.75 – 1.66 (m, 4H), 1.35 – 1.28 (m, 15H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 164.57, 161.19, 156.74, 153.64, 114.51, 107.66, 83.43, 59.86, 35.09, 34.75, 26.44, 26.31, 24.74, 14.38, 13.87; **HRMS (ESI, *m*/*z*):** calcd for $C_{20}H_{30}BO_5$ $[M+H]^+$ 361.2181, found 361.2180; **IR (film):** 2974, 2958, 2936, 1715, 1604, 1370, 1306, 1234, 1145, 1084 cm^{-1}.

(*E*)-1-(2-Methyl-5-(1-(4,4,6-trimethyl-1,3,2-dioxaborinan-2-yl)pent-1-en-1-yl)furan-3-yl)ethan-1-one (4a)



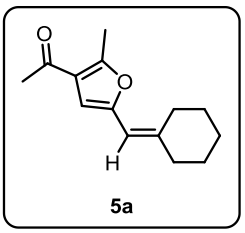
Yield: 70% (44.8 mg); colorless oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.63 (t, J = 7.9 Hz, 1H), 6.48 (s, 1H), 4.35 (dqd, J = 12.3, 6.2, 3.0 Hz, 1H), 2.56 (s, 3H), 2.39 – 2.32 (m, 5H), 1.89 (dd, J = 14.0, 3.0 Hz, 1H), 1.61 (dd, J = 14.0, 11.8 Hz, 1H), 1.48 (h, J = 7.3 Hz, 2H), 1.39 (s, 3H), 1.37 (s, 3H), 1.33 (d, J = 6.2 Hz, 3H), 0.94 (t, J = 7.3 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 194.51, 156.82, 153.59, 141.33, 122.73, 106.52, 71.54, 65.30, 45.93, 33.43, 31.14, 28.98, 28.12, 23.10, 23.03, 14.39, 13.88; **HRMS (ESI, *m*/*z*):** calcd for $C_{18}H_{28}BO_4$ $[M+H]^+$ 319.2075, found 319.2073; **IR (film):** 2975, 2958, 2868, 1678, 1583, 1396, 1304, 1230 cm^{-1}.

(*E*)-1-(2-Methyl-5-(1-(4,4,6,6-tetramethyl-1,3,2-dioxaborinan-2-yl)pent-1-en-1-yl)furan-3-yl)ethan-1-one (4b)



Yield: 76% (50.3 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.62 (t, J = 7.9 Hz, 1H), 6.47 (s, 1H), 2.56 (s, 3H), 2.39 – 2.31 (m, 5H), 1.94 (s, 2H), 1.48 (h, J = 7.4 Hz, 2H), 1.44 (s, 12H), 0.94 (t, J = 7.4 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 194.46, 156.80, 153.68, 140.70, 122.69, 106.40, 71.39, 48.83, 33.45, 31.74, 28.94, 23.06, 14.36, 13.88; **HRMS (ESI, *m*/*z*):** calcd for $C_{19}H_{30}BO_4$ $[M+H]^+$ 333.2232, found 333.2235; **IR (film):** 2974, 2927, 1677, 1583, 1405, 1386, 1369, 1228, 1205, 773 cm^{-1}.

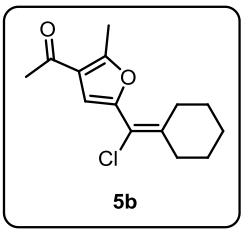
1-(5-(Cyclohexylidenemethyl)-2-methylfuran-3-yl)ethan-1-one (5a)



5a

Yield: 77% (16.8 mg); colorless oil; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 6.34 (s, 1H), 5.88 (s, 1H), 2.58 (s, 3H), 2.55 (t, J = 5.3 Hz, 2H), 2.39 (s, 3H), 2.23 (t, J = 5.5 Hz, 2H), 1.65 – 1.58 (m, 6H); **^{13}C NMR (126 MHz, $CDCl_3$)** δ 194.2, 156.5, 150.9, 144.8, 122.5, 110.2, 107.5, 37.6, 30.2, 29.1, 28.5, 27.5, 26.3, 14.4; **HRMS (ESI, *m*/*z*):** calcd for $C_{14}H_{19}O_2$ $[M+H]^+$ 219.1380, found 219.1378; **IR (film):** 2927, 2852, 1678, 1585, 1236, 958 cm^{-1}.

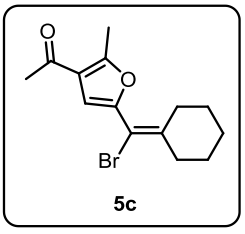
1-(5-(Chloro(cyclohexylidene)methyl)-2-methylfuran-3-yl)ethan-1-one (5b)



5b

Yield: 82% (20.6 mg); colorless oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.58 (s, 1H), 2.61 (s, 3H), 2.55 – 2.50 (m, 2H), 2.48 – 2.43 (m, 2H), 2.41 (s, 3H), 1.69 – 1.56 (m, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 193.88, 157.88, 148.02, 142.85, 122.20, 112.20, 110.65, 32.21, 32.10, 29.10, 27.73, 27.13, 26.14, 14.52; **HRMS (ESI, *m*/*z*):** calcd for $C_{14}H_{18}ClO_2$ $[M+H]^+$ 253.0990, found 253.0987; **IR (film):** 2939, 2856, 1682, 1582, 1452, 1396, 1231, 956 cm^{-1}.

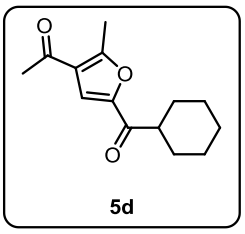
1-(5-(Bromo(cyclohexylidene)methyl)-2-methylfuran-3-yl)ethan-1-one (5c)



5c

Yield: 75% (22.0 mg); colorless oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.54 (s, 1H), 2.61 (s, 3H), 2.57 – 2.51 (m, 2H), 2.43 – 2.38 (m, 5H), 1.70 – 1.54 (m, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 193.86, 157.89, 148.76, 146.74, 122.25, 110.58, 102.35, 35.41, 33.01, 29.10, 27.78, 27.18, 26.08, 14.56; **HRMS (ESI, *m*/*z*):** calcd for $C_{14}H_{18}BrO_2$ $[M+H]^+$ 297.0485, found 297.0482; **IR (film):** 2933, 2853, 1681, 1581, 1448, 1408, 1230, 1003, 955, 815, 795 cm^{-1}.

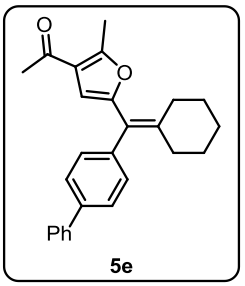
1-(5-(Cyclohexanecarbonyl)-2-methylfuran-3-yl)ethan-1-one (5d)



5d

Yield: 73% (30.6 mg); colorless oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.38 (s, 1H), 3.01 (tt, J = 11.7, 3.2 Hz, 1H), 2.69 (s, 3H), 2.47 (s, 3H), 1.91 – 1.80 (m, 4H), 1.57 – 1.24 (m, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 193.36, 192.50, 162.21, 149.54, 123.09, 117.14, 46.18, 29.02, 28.91, 25.69, 25.64, 14.86; **HRMS (ESI, *m*/*z*):** calcd for $C_{14}H_{19}O_3$ $[M+H]^+$ 235.1329, found 235.1327; **IR (film):** 2934, 2855, 1675, 1577, 1528, 1450, 1244, 1212, 1155, 1003, 953 cm^{-1}.

1-(5-([1,1'-biphenyl]-4-yl(cyclohexylidene)methyl)-2-methylfuran-3-yl)ethan-1-one (5e)

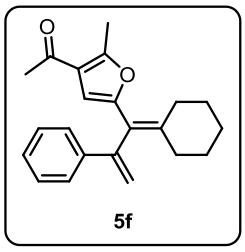


5e

Yield: 86% (32.0 mg); yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.65 – 7.59 (m, 2H), 7.58 – 7.53 (m, 2H), 7.47 – 7.41 (m, 2H), 7.37 – 7.30 (m, 1H), 7.24 – 7.19 (m, 2H), 6.18 (s, 1H), 2.64 – 2.58 (m, 2H), 2.56 (s, 3H), 2.35 (s, 3H), 2.23 – 2.15 (m, 2H), 1.73 – 1.54 (m, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 194.33, 157.15, 152.85, 144.04, 140.77, 139.49, 139.33, 130.20, 128.74, 127.21, 126.97, 126.78, 123.24, 122.31, 109.74, 32.89, 32.53, 29.14, 28.60, 28.46, 26.62, 14.56; **HRMS (ESI, *m*/*z*):** calcd for $C_{26}H_{27}O_2$ $[M+H]^+$ 371.2006, found 371.2000; **IR (film):** 2928, 2921, 2851, 1677, 1582, 1484, 1444, 1392, 1233, 1006, 957, 764, 733

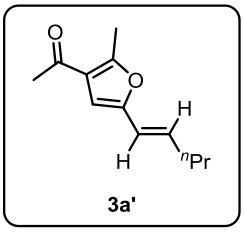
cm^{-1}.

1-(5-(1-Cyclohexylidene-2-phenylallyl)-2-methylfuran-3-yl)ethan-1-one (5f)



Yield: 99% (31.9 mg); orange oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.46 – 7.41 (m, 2H), 7.31 – 7.20 (m, 3H), 6.27 (s, 1H), 5.75 (d, *J* = 1.5 Hz, 1H), 5.18 (d, *J* = 1.5 Hz, 1H), 2.62 (t, *J* = 6.0 Hz, 2H), 2.53 (s, 3H), 2.30 (s, 3H), 2.26 (t, *J* = 6.0 Hz, 2H), 1.74 – 1.66 (m, 2H), 1.65 – 1.58 (m, 2H), 1.57 – 1.49 (m, 2H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 194.28, 156.97, 151.85, 146.28, 143.76, 139.46, 128.30, 127.58, 126.11, 122.69, 122.36, 115.32, 109.42, 33.11, 31.91, 29.05, 28.56, 28.40, 26.60, 14.56; **HRMS (ESI, *m*/*z*):** calcd for $C_{22}H_{25}O_2$ $[M+H]^+$ 321.1849, found 321.1846; **IR (film):** 2927, 2853, 1678, 1582, 1492, 1446, 1396, 1232, 953, 905, 808, 780, 703 cm^{-1}.

(*E*)-1-(2-methyl-5-(pent-1-en-1-yl)furan-3-yl)ethan-1-one (3a')



Yield: 90% (27.6 mg); colorless oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.31 (s, 1H), 6.18 (dt, *J* = 15.9, 6.4 Hz, 1H), 6.11 (d, *J* = 15.9 Hz, 1H), 2.58 (s, 3H), 2.38 (s, 3H), 2.16 (q, *J* = 7.0 Hz, 2H), 1.48 (h, *J* = 7.3 Hz, 2H), 0.94 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 194.17, 157.33, 150.97, 130.91, 122.61, 117.74, 105.92, 34.81, 29.07, 22.28, 14.38, 13.65; **HRMS (ESI, *m*/*z*):** calcd for $C_{12}H_{17}O_2$ $[M+H]^+$ 193.1223, found 193.1222; **IR (film):** 2961, 2930, 2871, 1674, 1585, 1399, 1238, 950 cm^{-1}.

7. References

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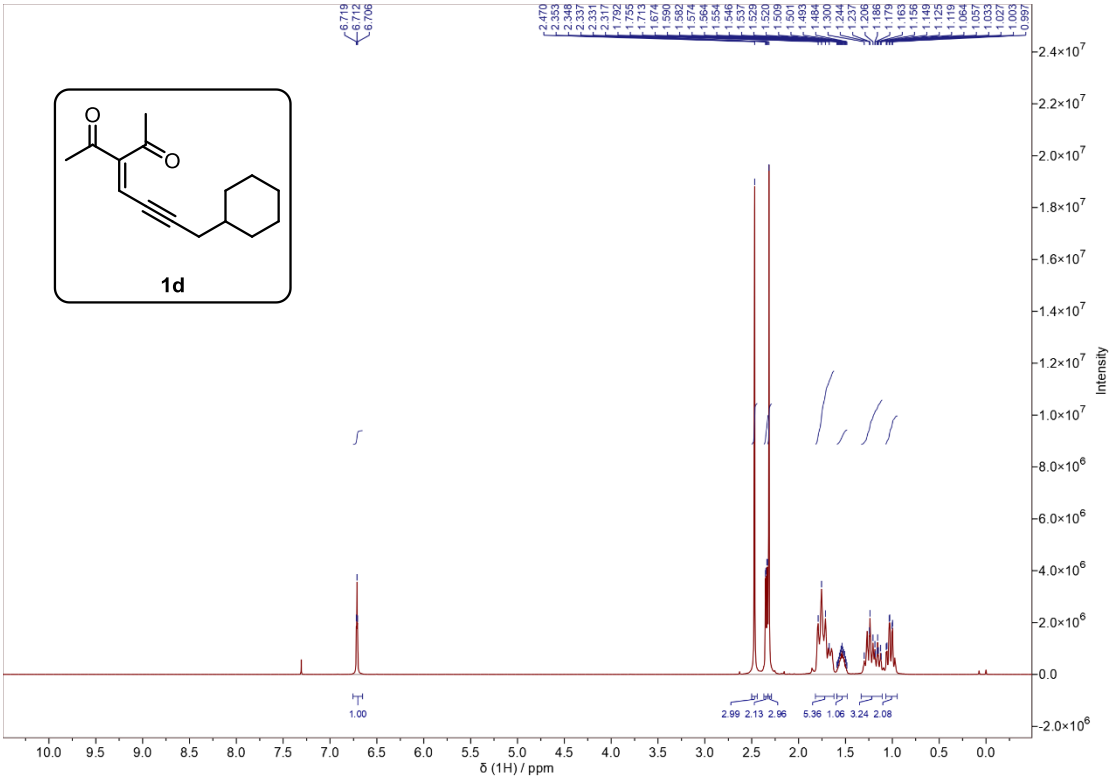
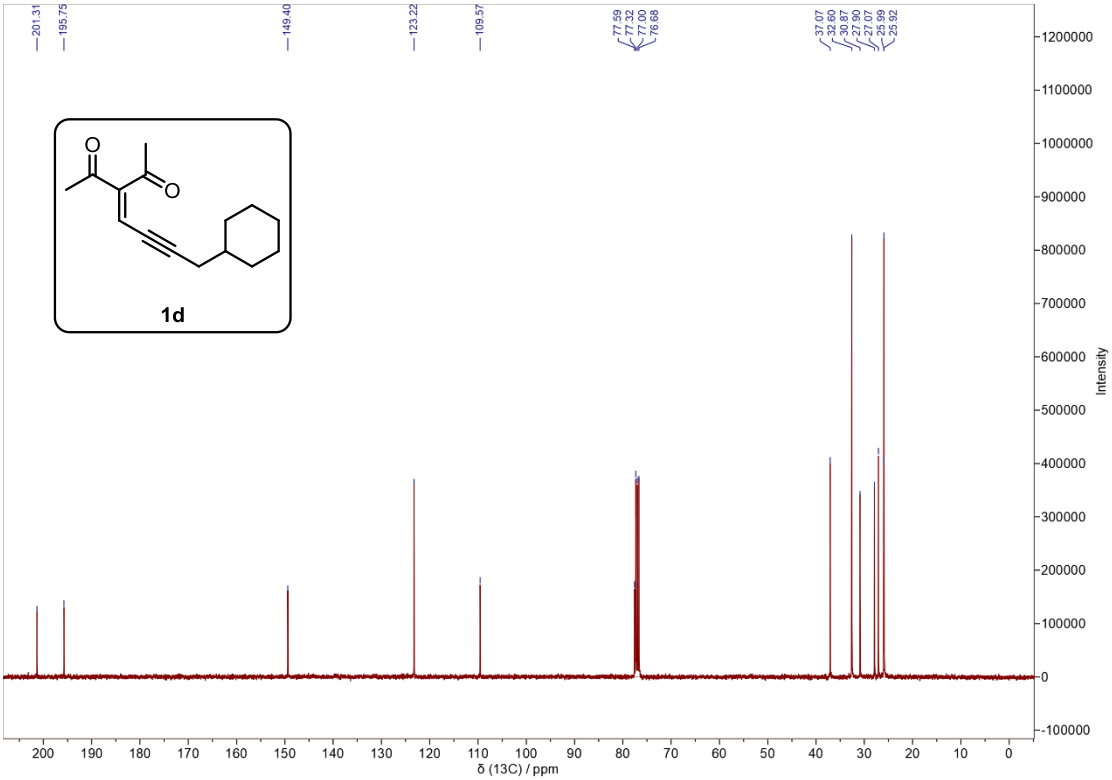
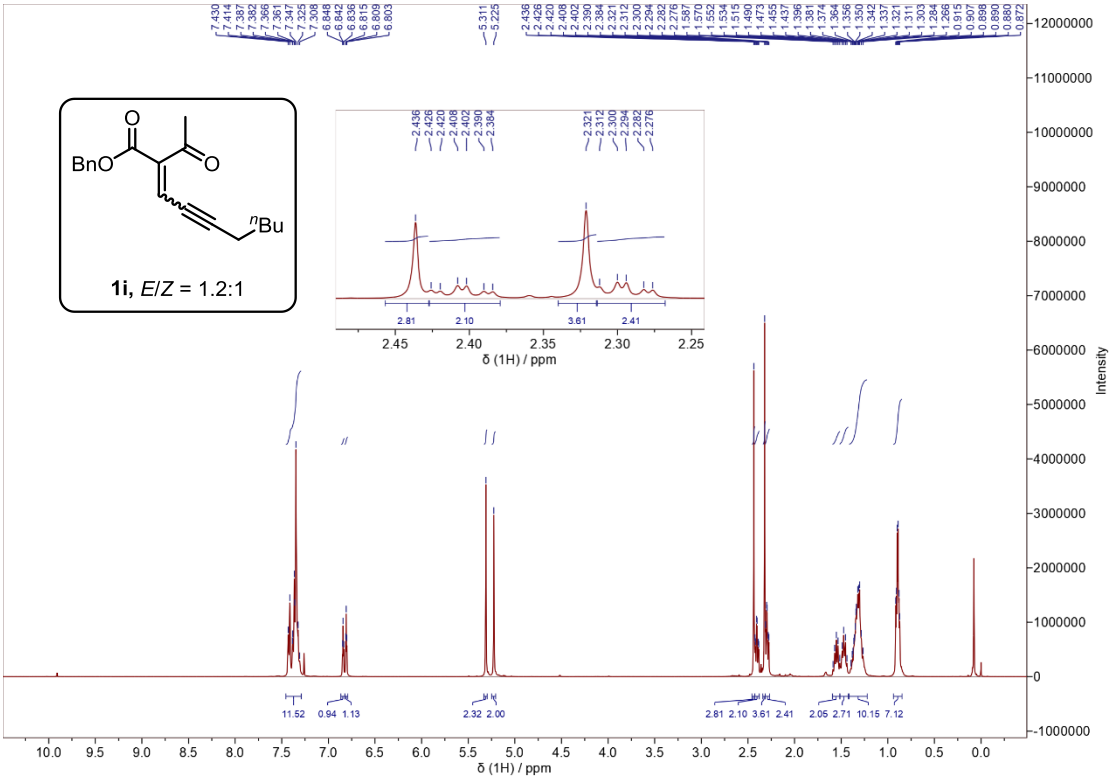
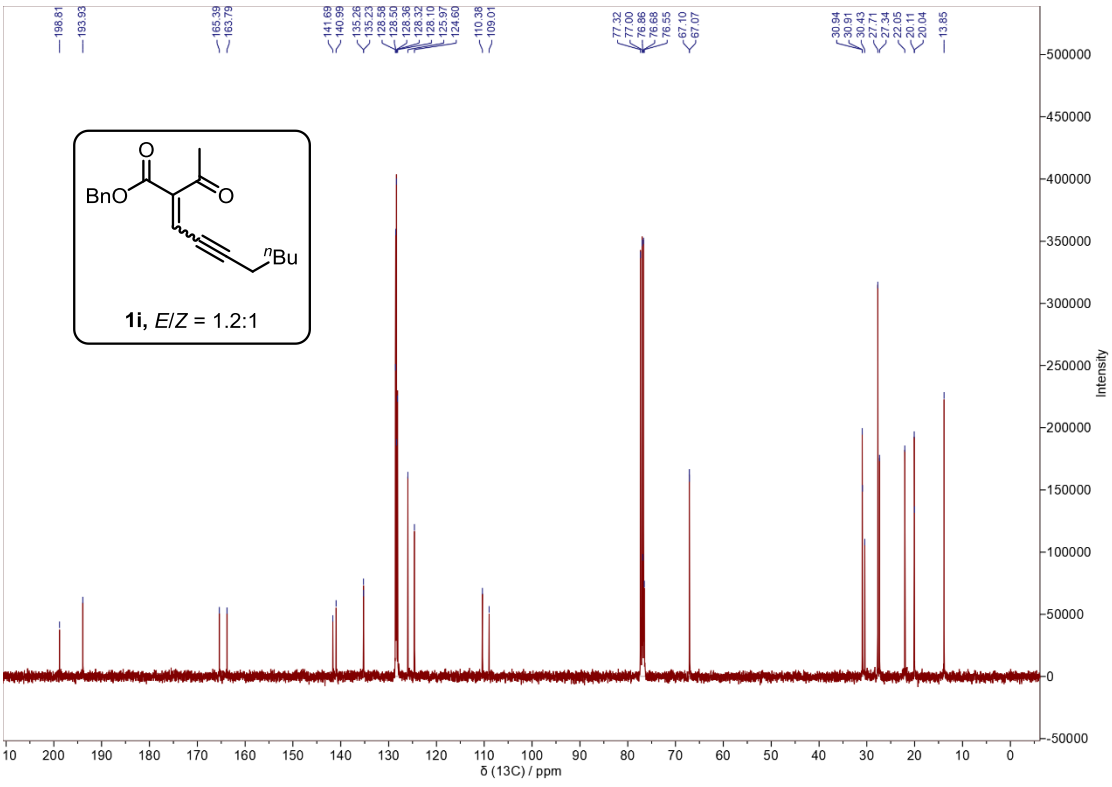
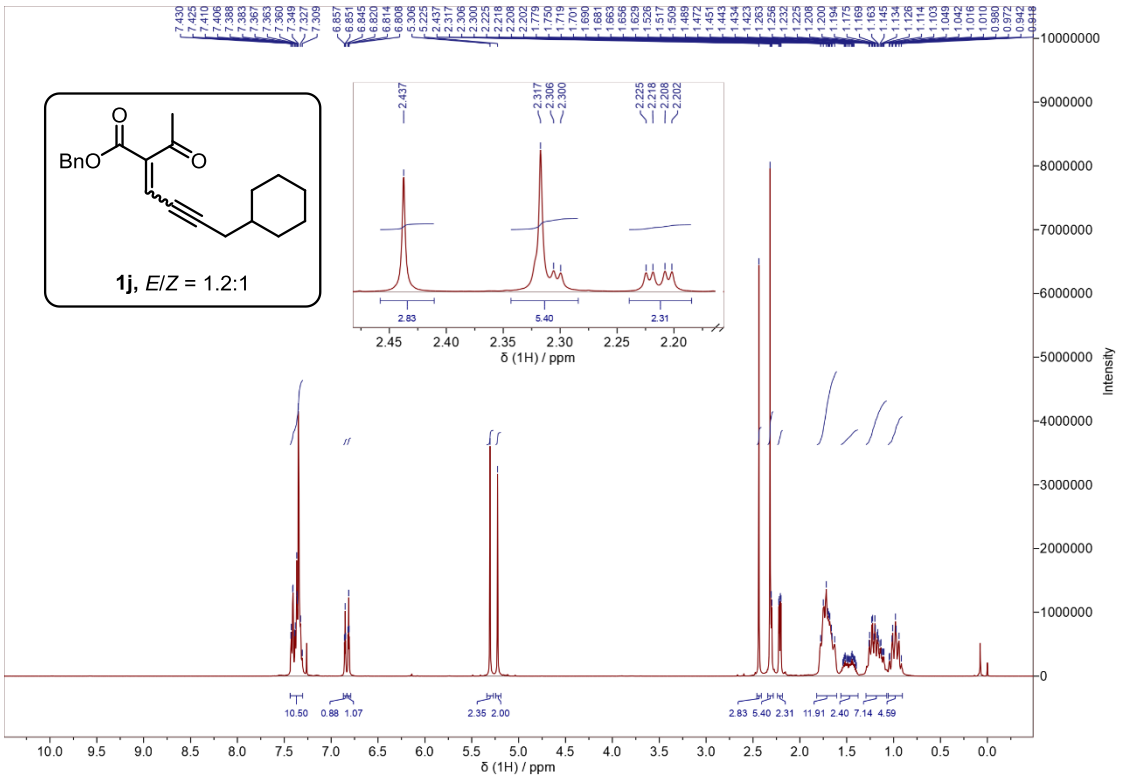
8. ^{1}H and ^{13}C NMR spectra

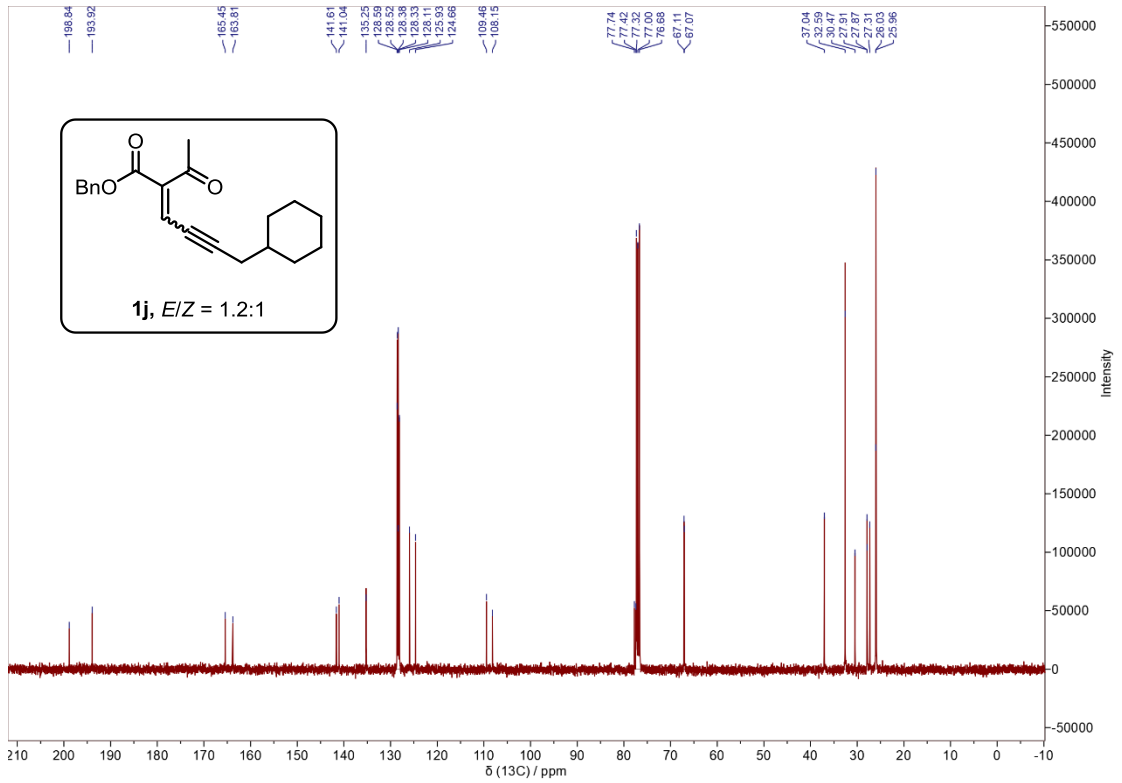





BnO
O
O
nBu
1i, E/Z = 1.2:1
δ (1H) / ppm
Intensity


BnO
O
O
nBu
1i, E/Z = 1.2:1
δ (13C) / ppm
Intensity


BnO
O
O
1j, E/Z = 1.2:1
δ (1H) / ppm
Intensity


BnO
O
O
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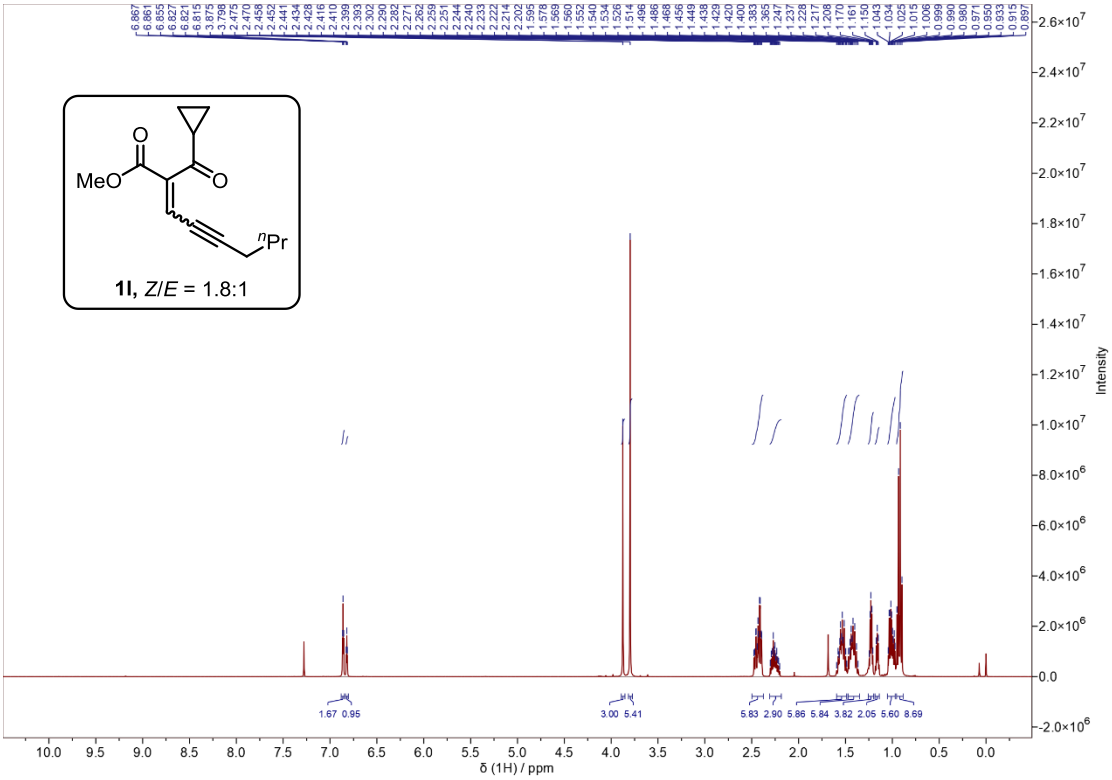
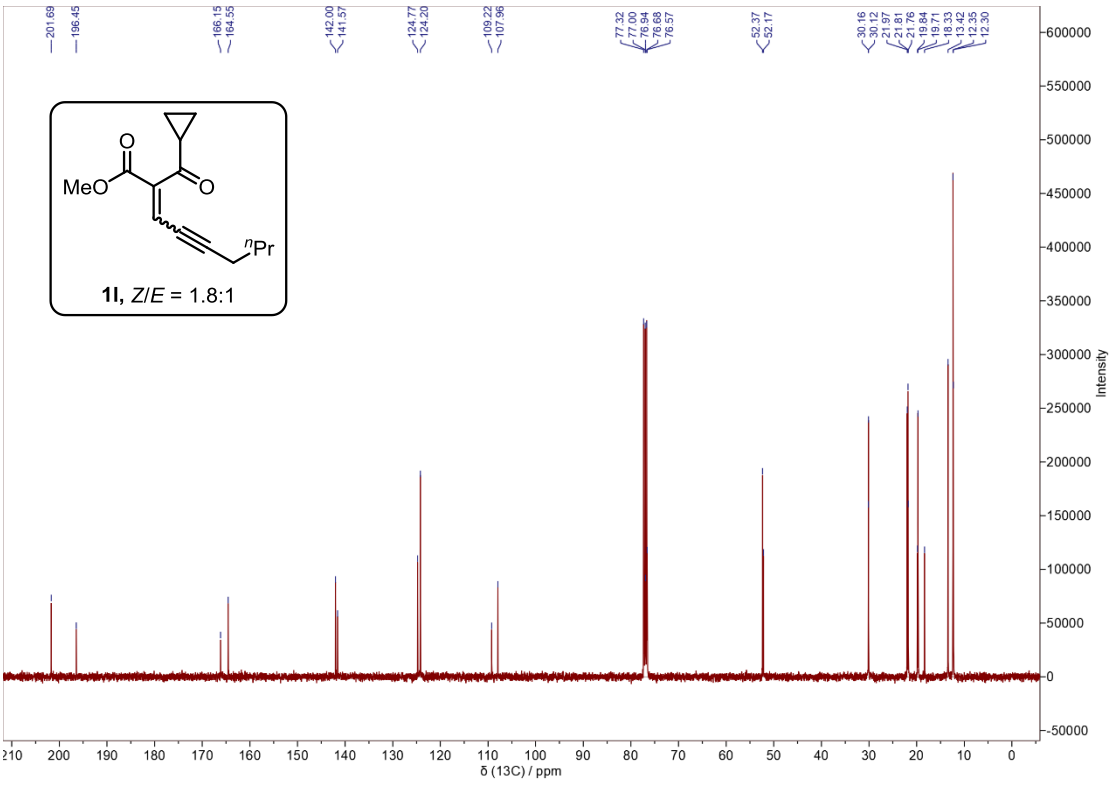
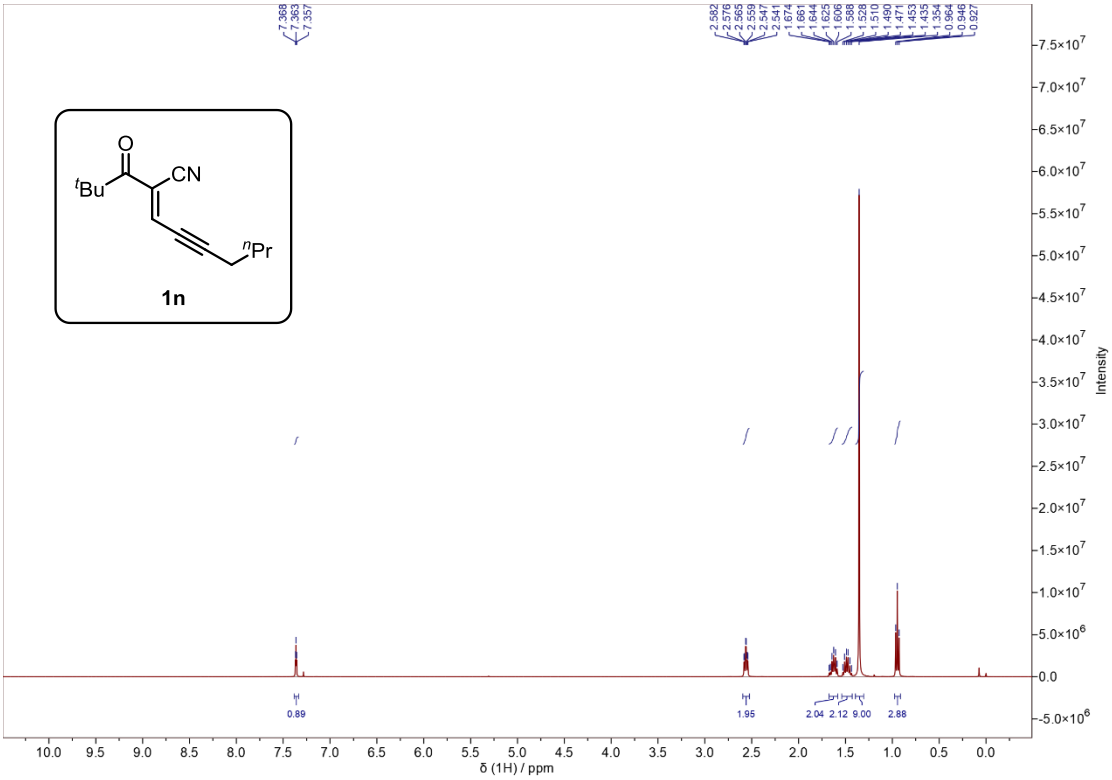
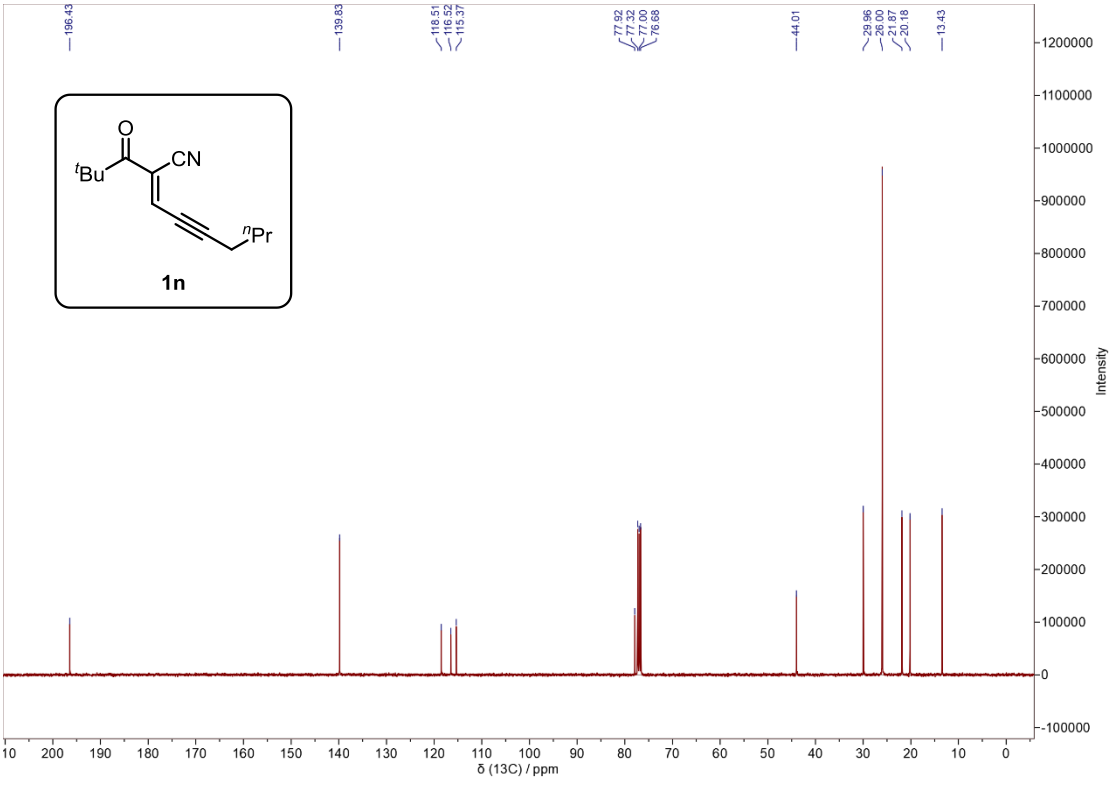
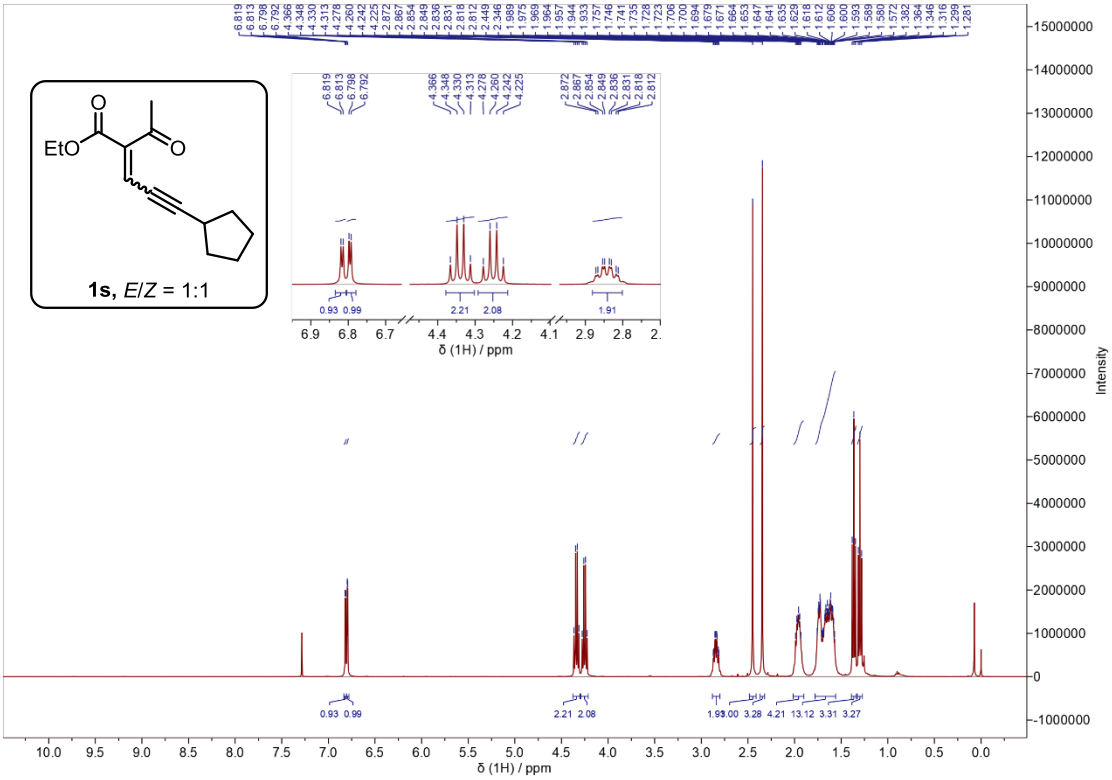
δ (13C) / ppm
Intensity

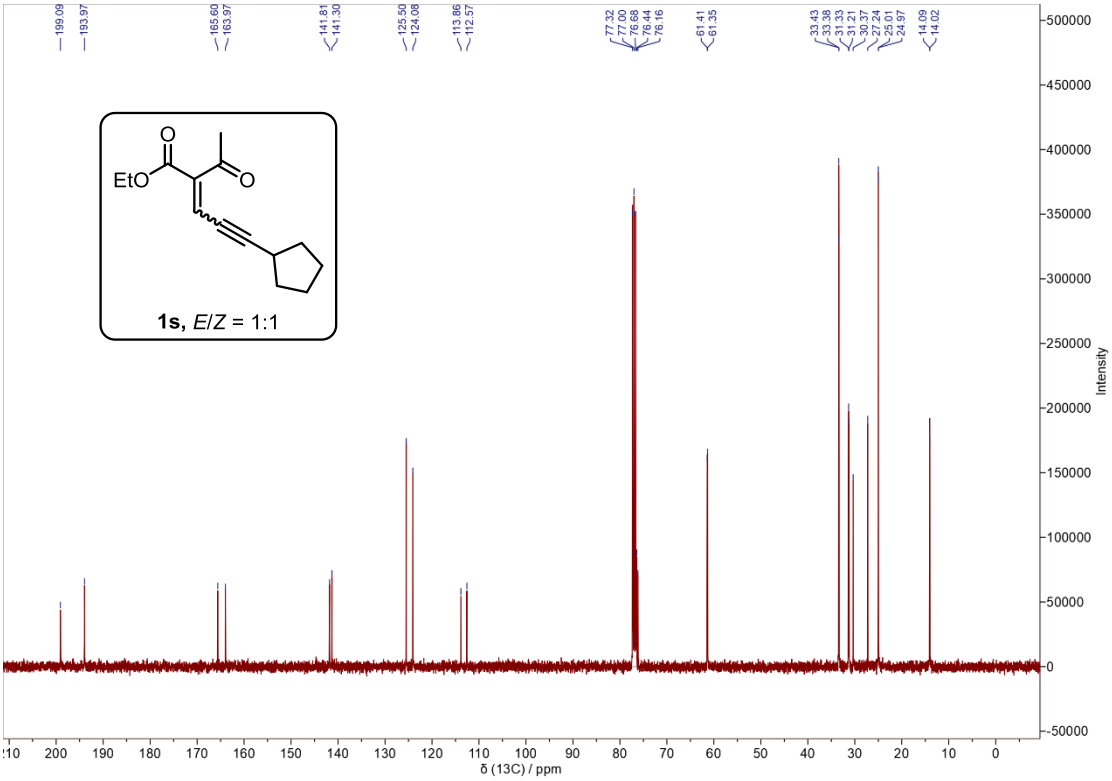

MeO
O
O
nPr
1l, Z/E = 1.8:1
δ (1H) / ppm
Intensity

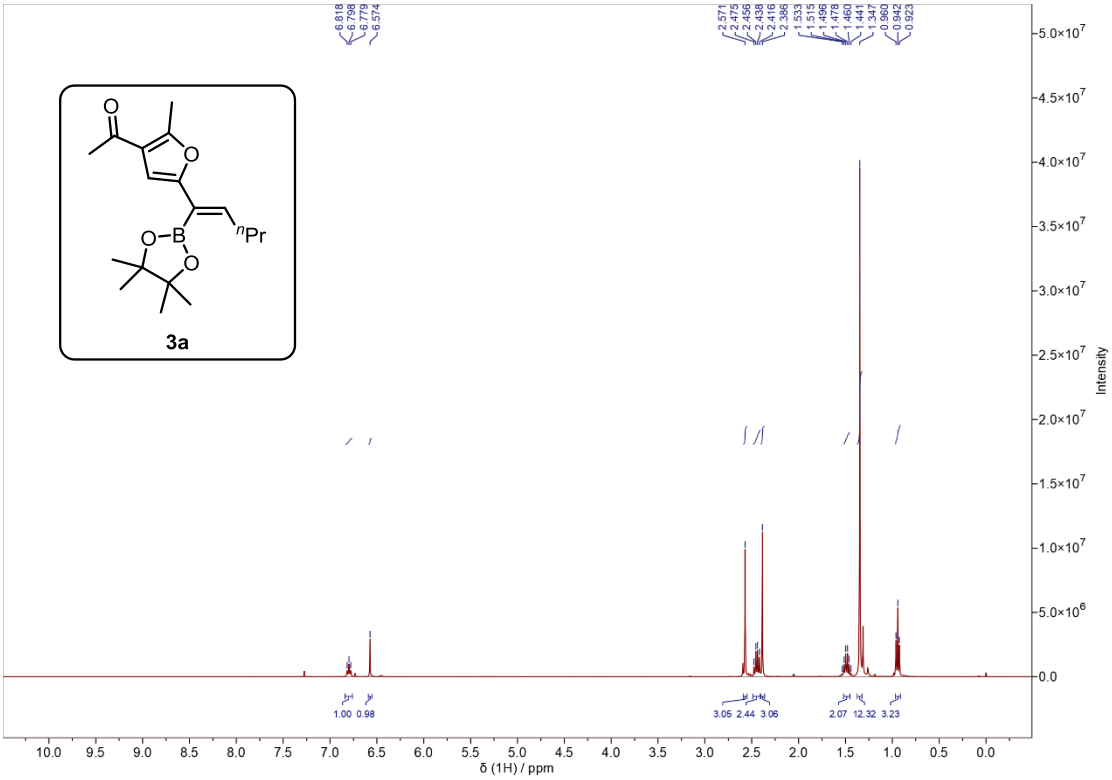

MeO
O
O
nPr
1l, Z/E = 1.8:1
δ (13C) / ppm
Intensity

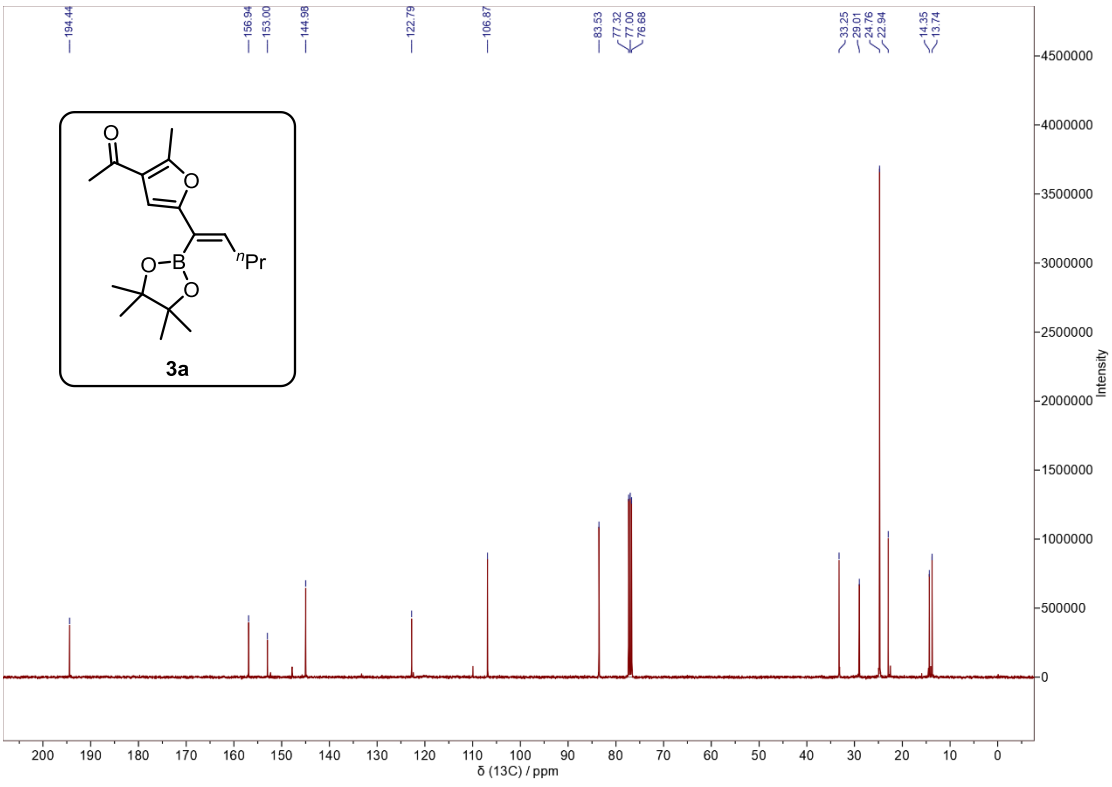

1n
δ (1H) / ppm
Intensity

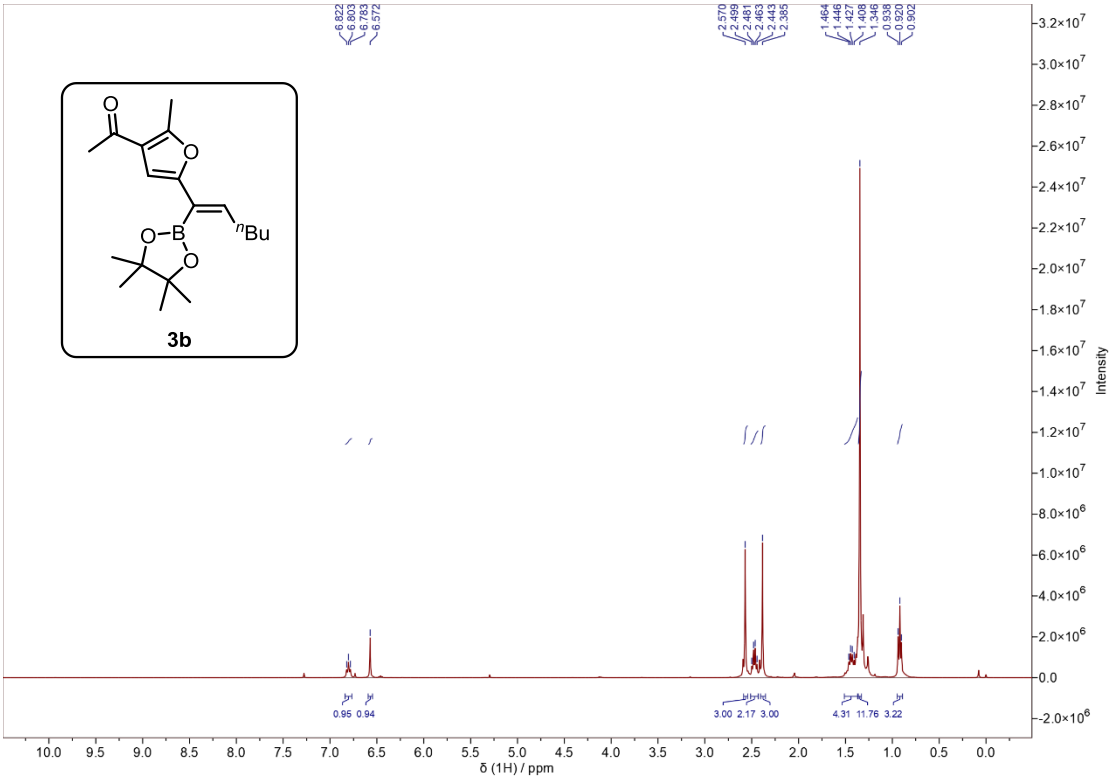

1n
δ (13C) / ppm
Intensity

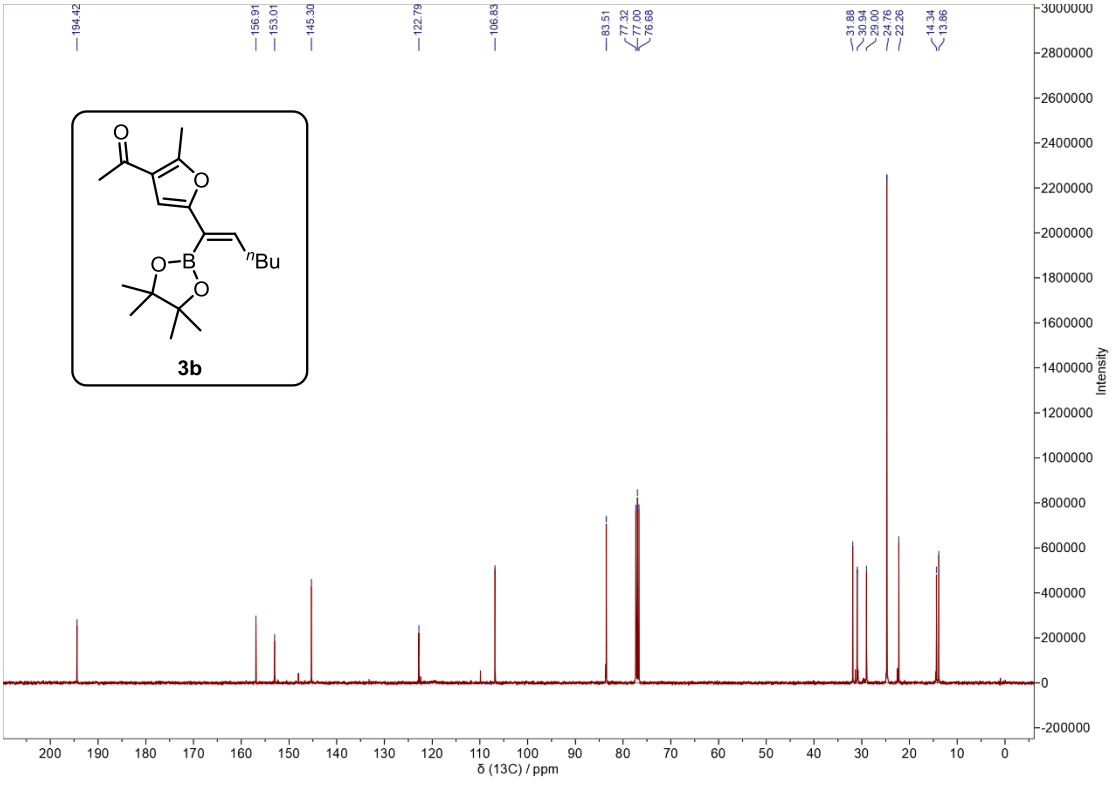

EtO
O
O
1s, E/Z = 1:1
δ (1H) / ppm
Intensity

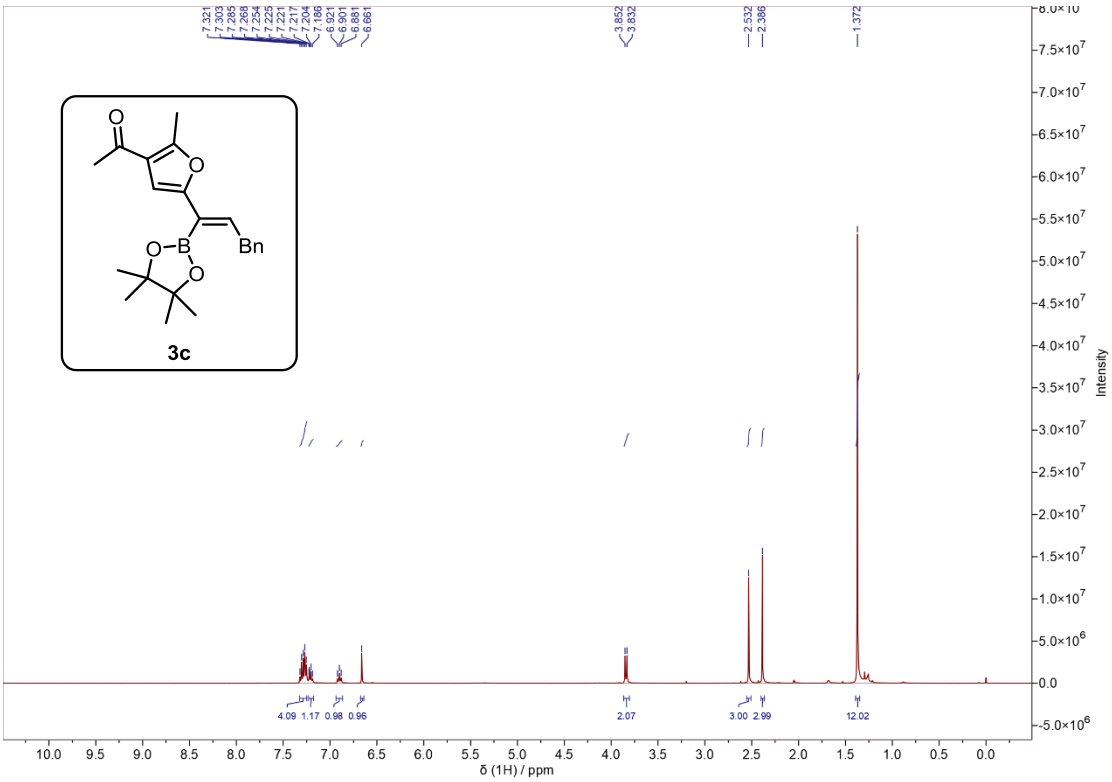

EtO
O
O
1s, E/Z = 1:1
δ (13C) / ppm
Intensity

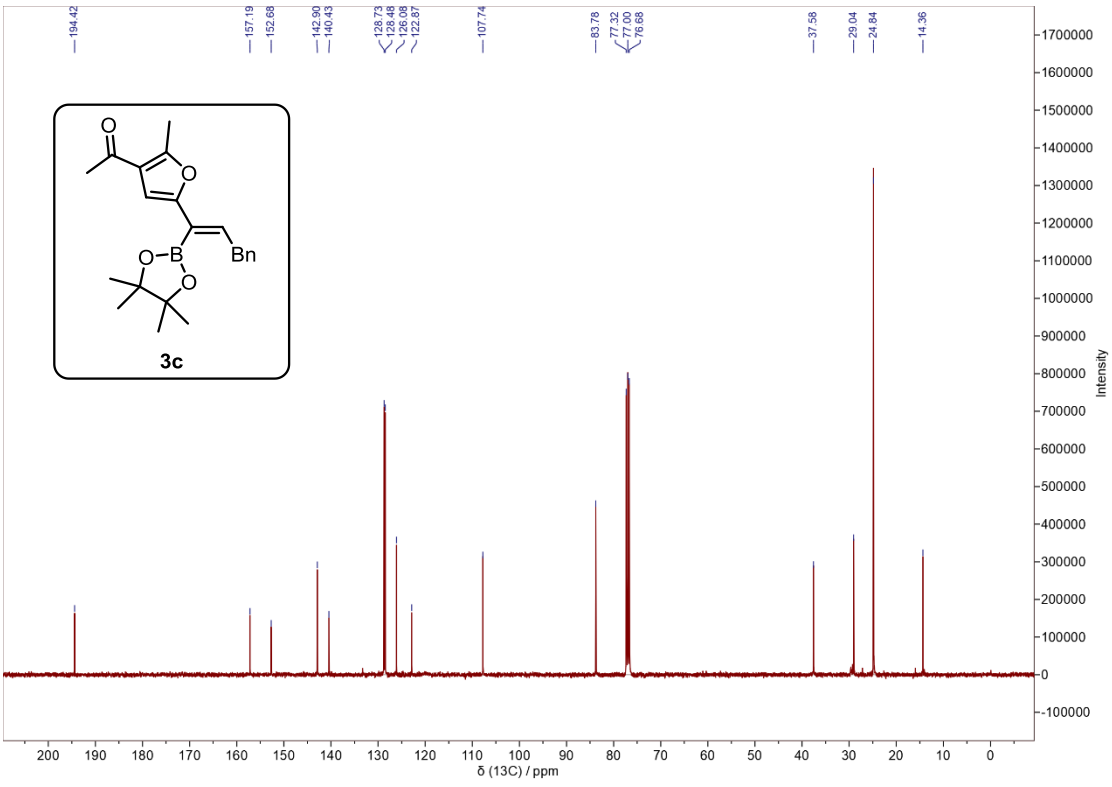

6.818
6.798
6.779
6.574
2.571
2.475
2.456
2.438
2.416
2.396
1.533
1.515
1.496
1.478
1.460
1.441
1.347
0.960
0.942
0.923
O
O
O–B
O
nPr
3a
Intensity
δ (1H) / ppm
1.00 0.98
3.05 2.44 3.06
2.07 12.32 3.23

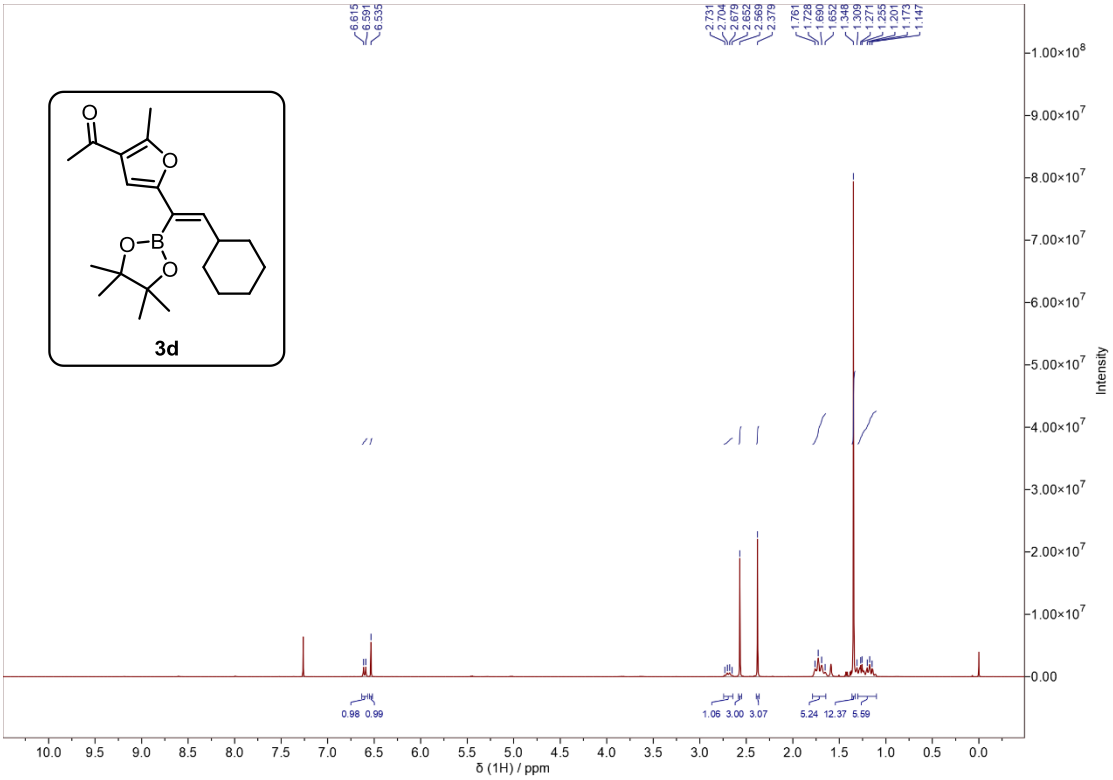

194.44
156.94
153.00
144.98
122.79
106.87
83.53
77.32
77.00
76.68
33.25
29.01
24.76
22.94
14.35
13.74
O
O
O–B
O
nPr
3a
Intensity
δ (13C) / ppm

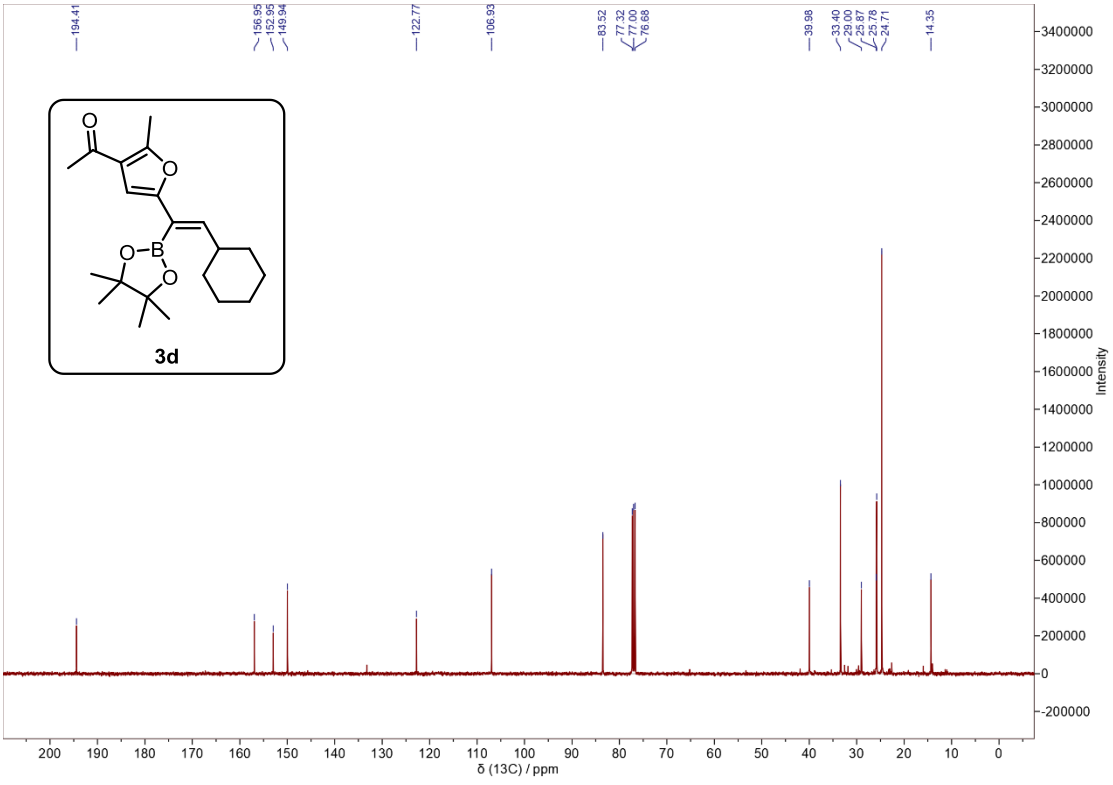

6.822
6.803
6.793
6.572
2.570
2.499
2.481
2.463
2.443
2.365
1.464
1.446
1.427
1.408
1.346
0.938
0.920
0.902
3b
Intensity
0.95 0.94
3.00 2.17 3.00
4.31 11.76 3.22
δ (1H) / ppm

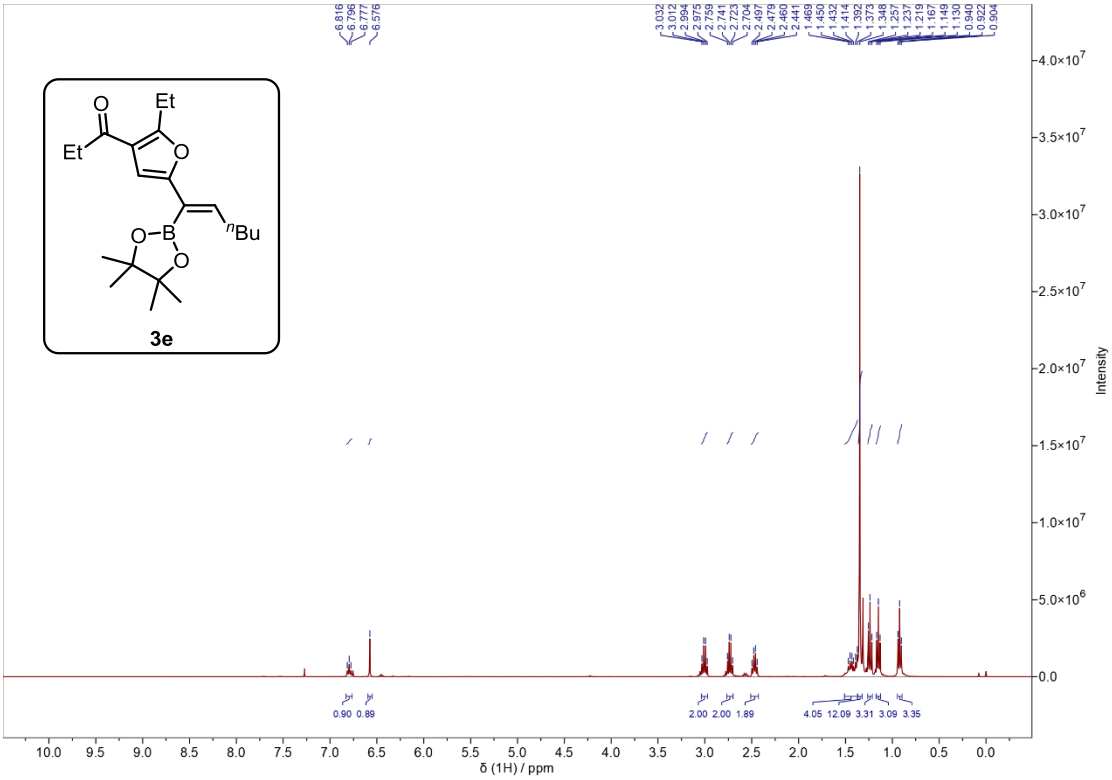

194.42
156.91
153.01
145.30
122.79
106.83
83.51
77.32
77.00
76.68
31.88
30.94
29.00
24.76
22.26
14.34
13.86
3b
Intensity
δ (13C) / ppm

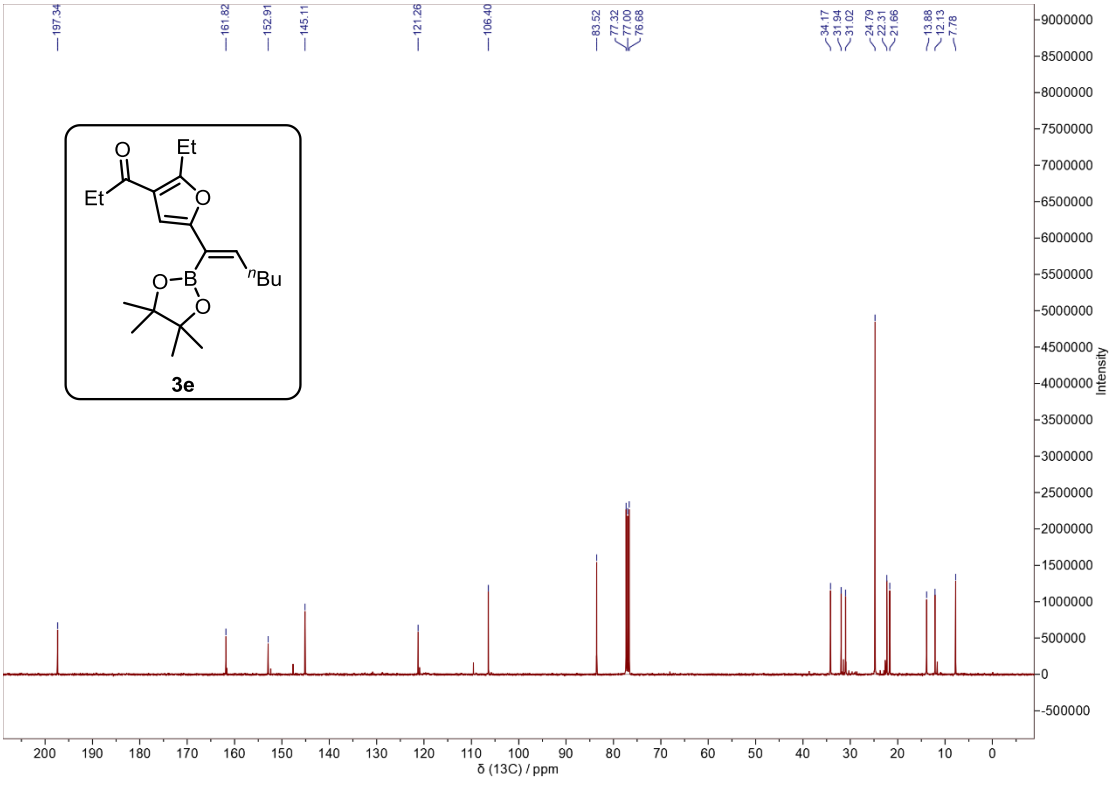

7.321
7.303
7.285
7.268
7.254
7.225
7.221
7.217
7.204
7.186
6.921
6.901
6.881
6.661
3.852
3.832
2.532
2.386
1.372
O
O
O-B
O
Bn
3c
Intensity
4.09
1.17
0.98
0.96
2.07
3.00
2.99
12.02
δ (1H) / ppm

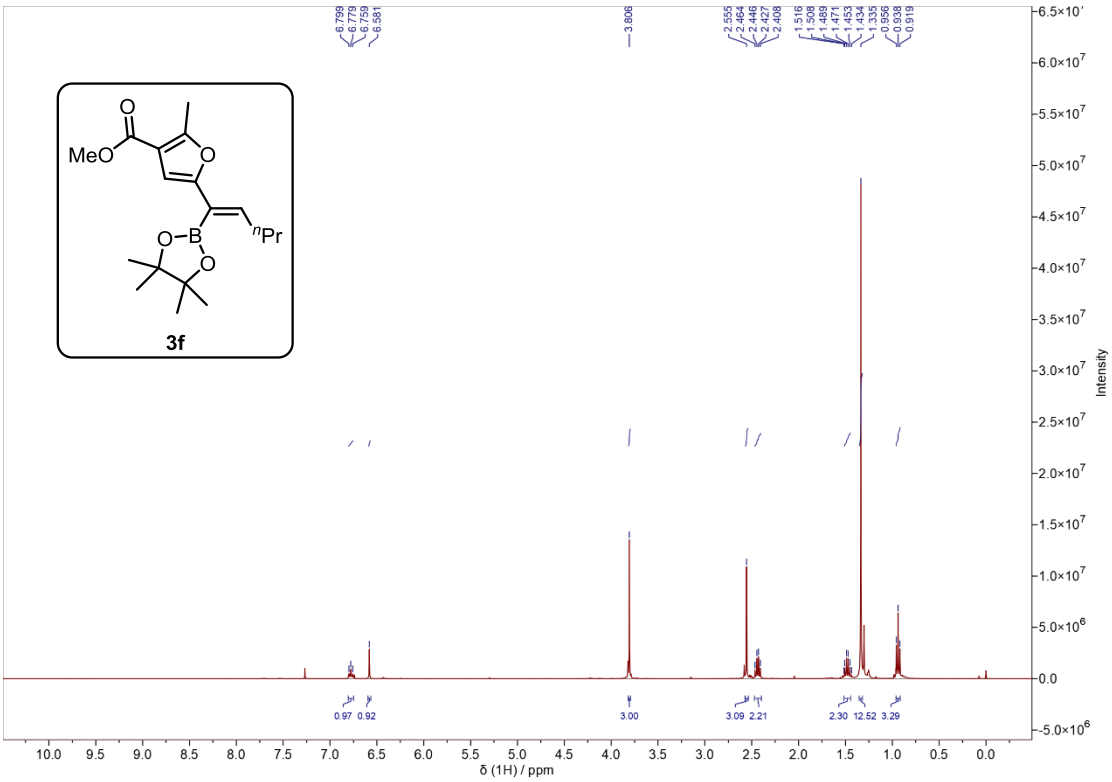

194.42
157.19
152.68
142.90
140.43
128.73
128.48
126.08
122.87
107.74
83.78
77.32
77.00
76.68
37.58
29.04
24.84
14.36
O
O
O-B
O
Bn
3c
Intensity
δ (13C) / ppm

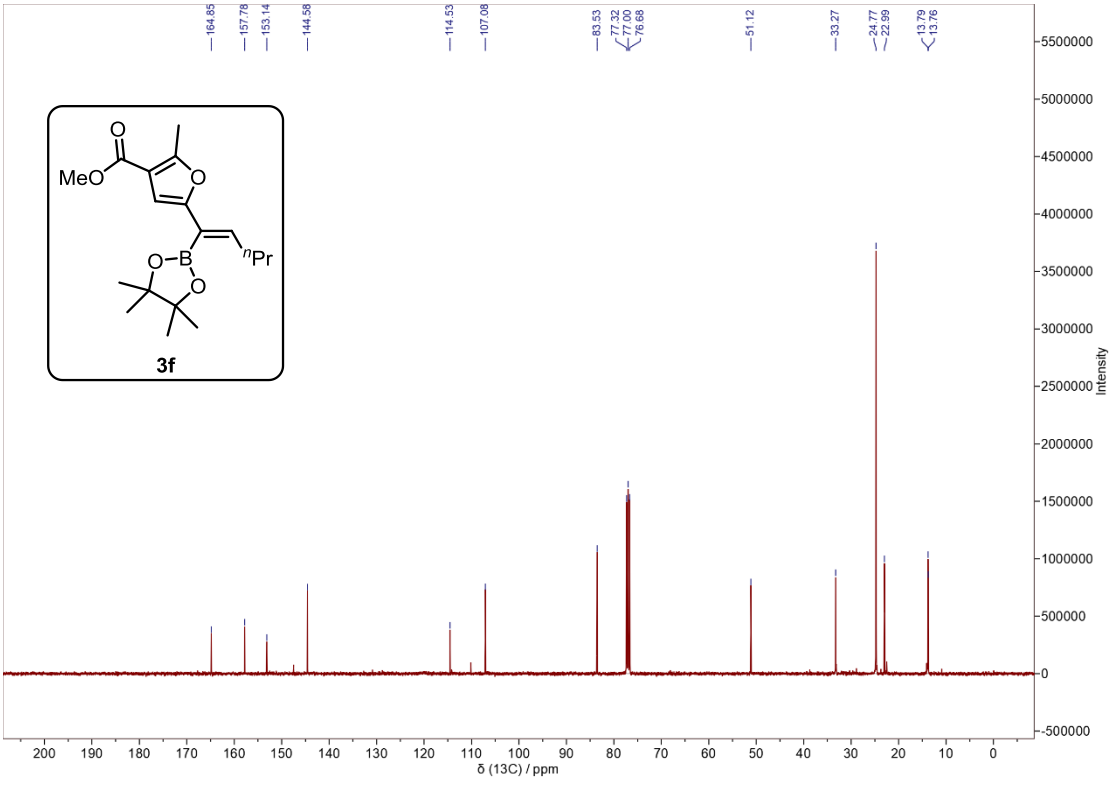

3d
δ (1H) / ppm
Intensity

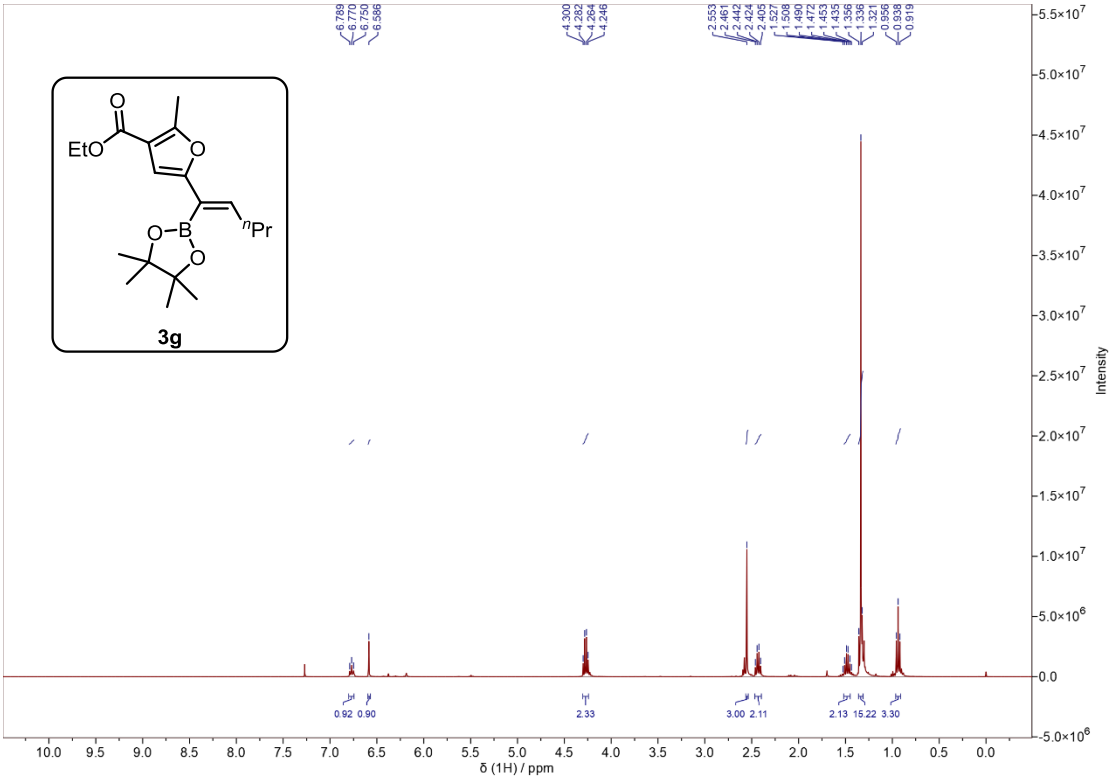

3d
δ (13C) / ppm
Intensity

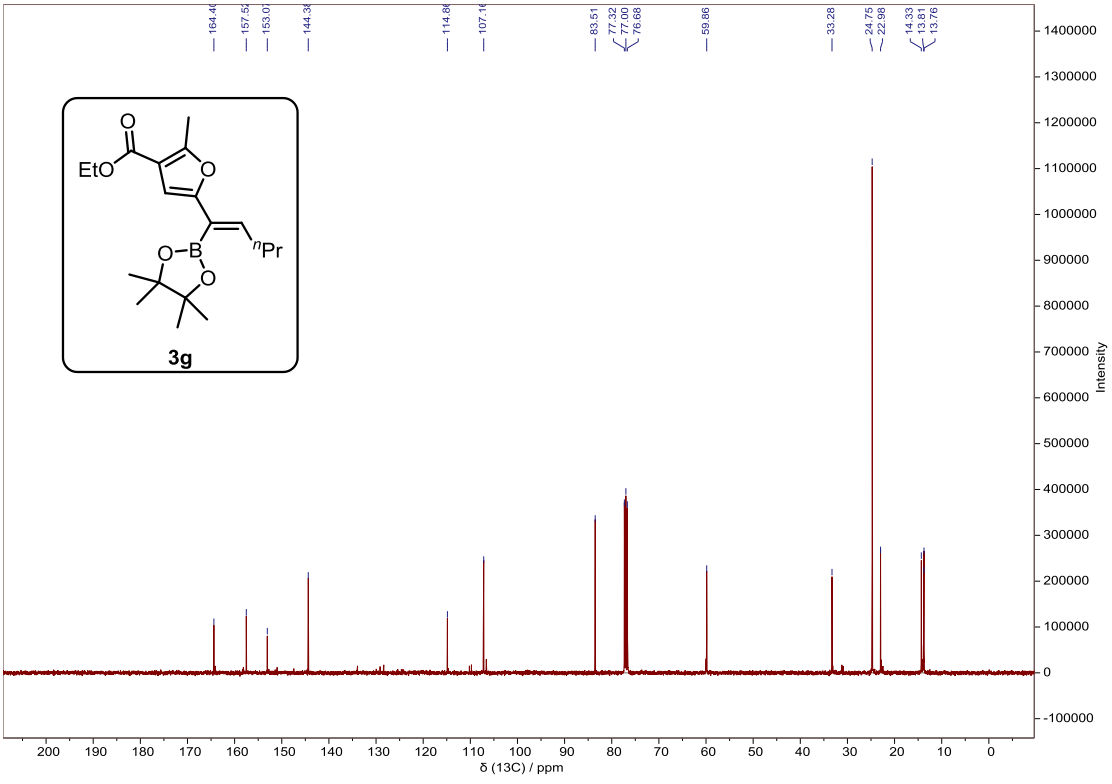

3e
δ (1H) / ppm
Intensity

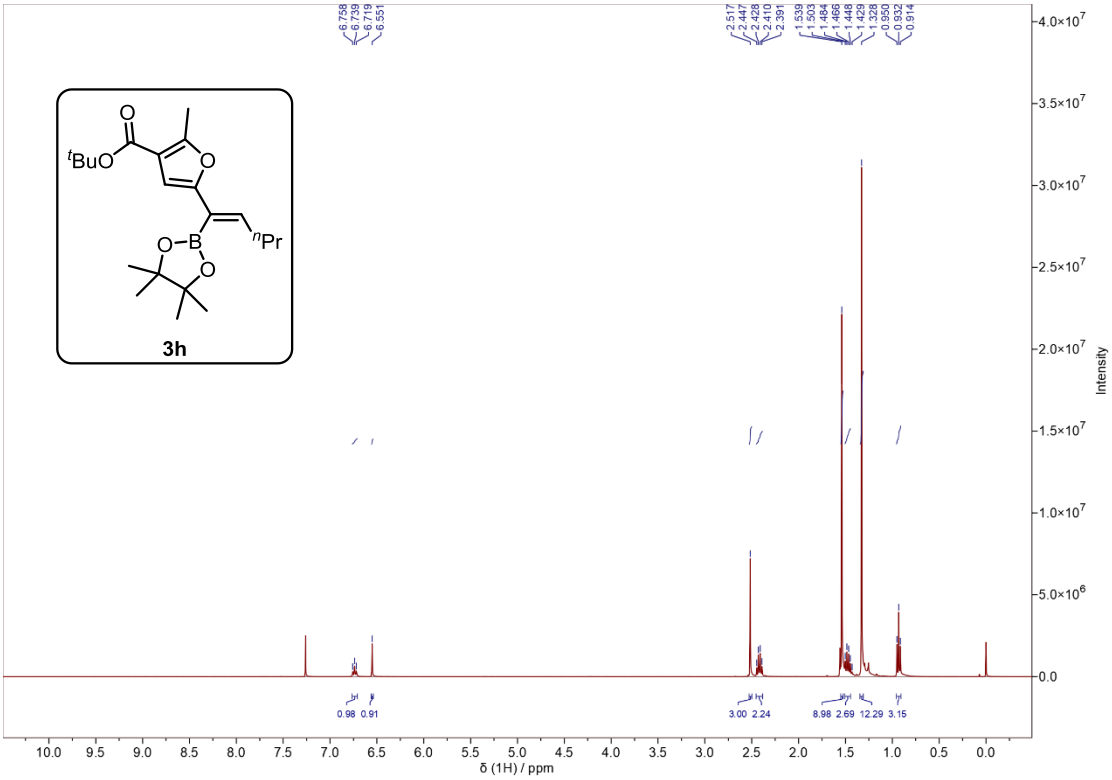

3e
δ (13C) / ppm
Intensity

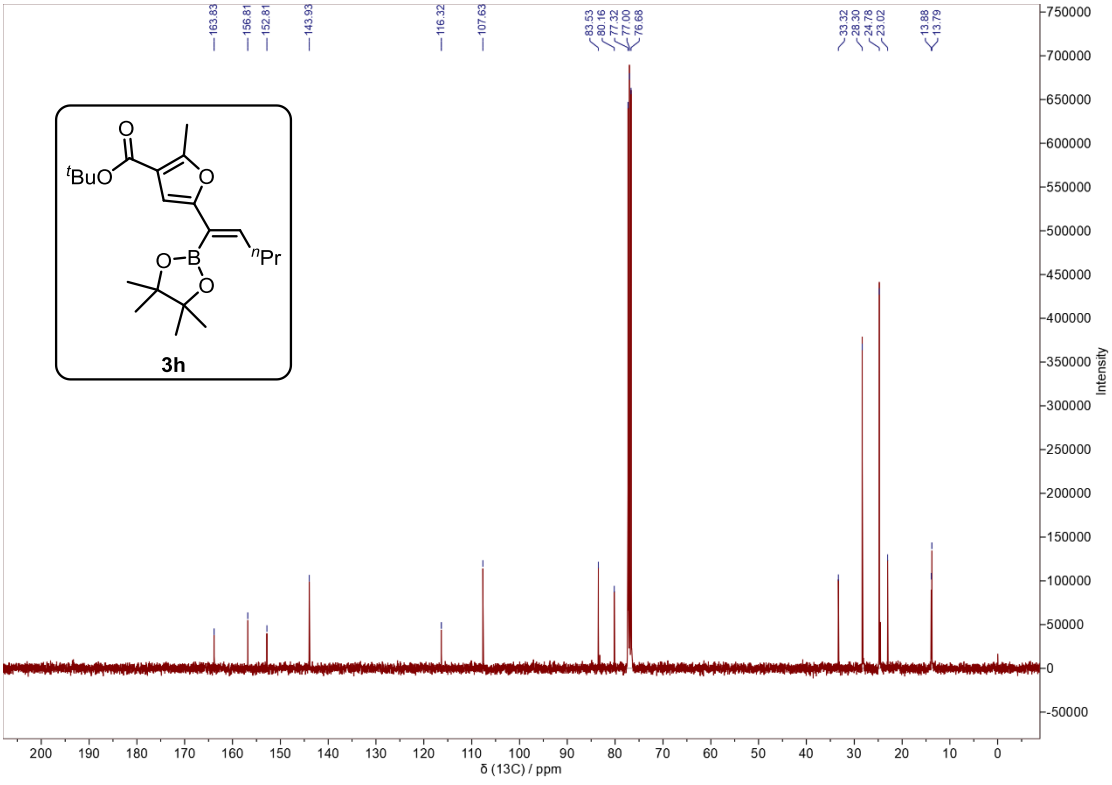

6.799
6.779
6.759
6.581
3.806
2.555
2.464
2.446
2.427
2.408
1.516
1.508
1.489
1.471
1.453
1.434
1.335
0.956
0.938
0.919
MeO
O
O
O-B
O
nPr
3f
Intensity
0.97 0.92
3.00
3.09 2.21
2.30 12.52 3.29
δ (1H) / ppm

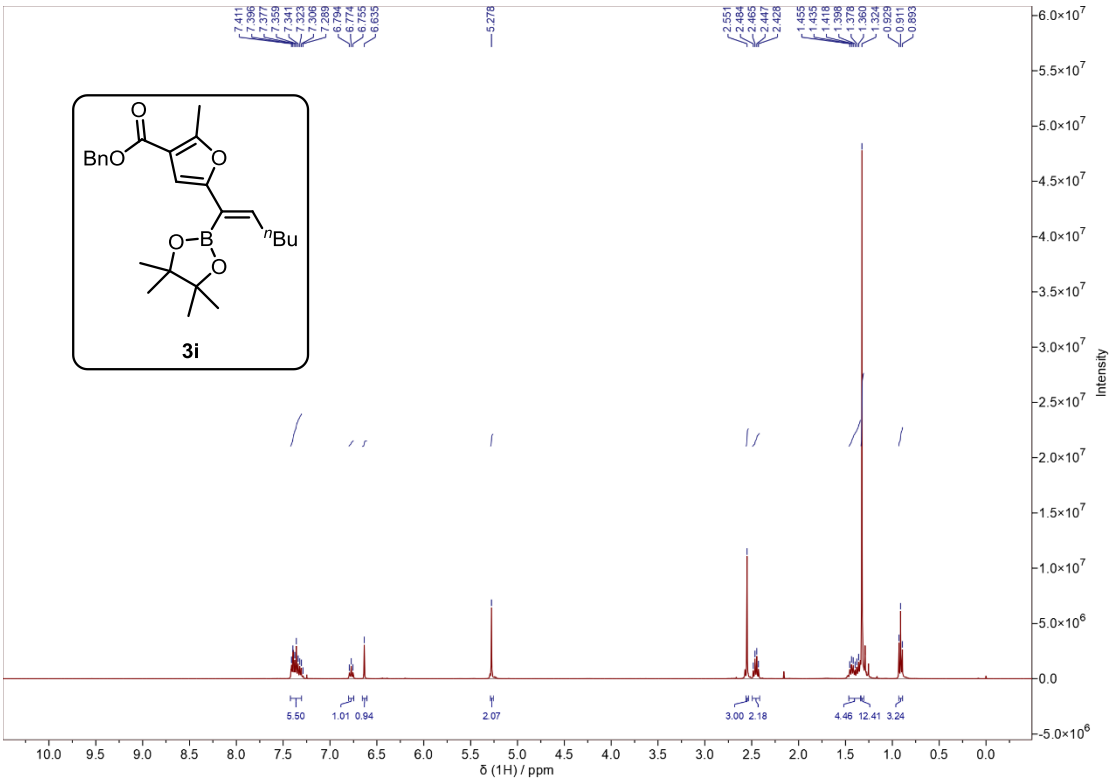

164.85
157.78
153.14
144.58
114.53
107.08
83.53
77.32
77.00
76.68
51.12
33.27
24.77
22.99
13.79
13.76
MeO
O
O
O-B
O
nPr
3f
Intensity
δ (13C) / ppm

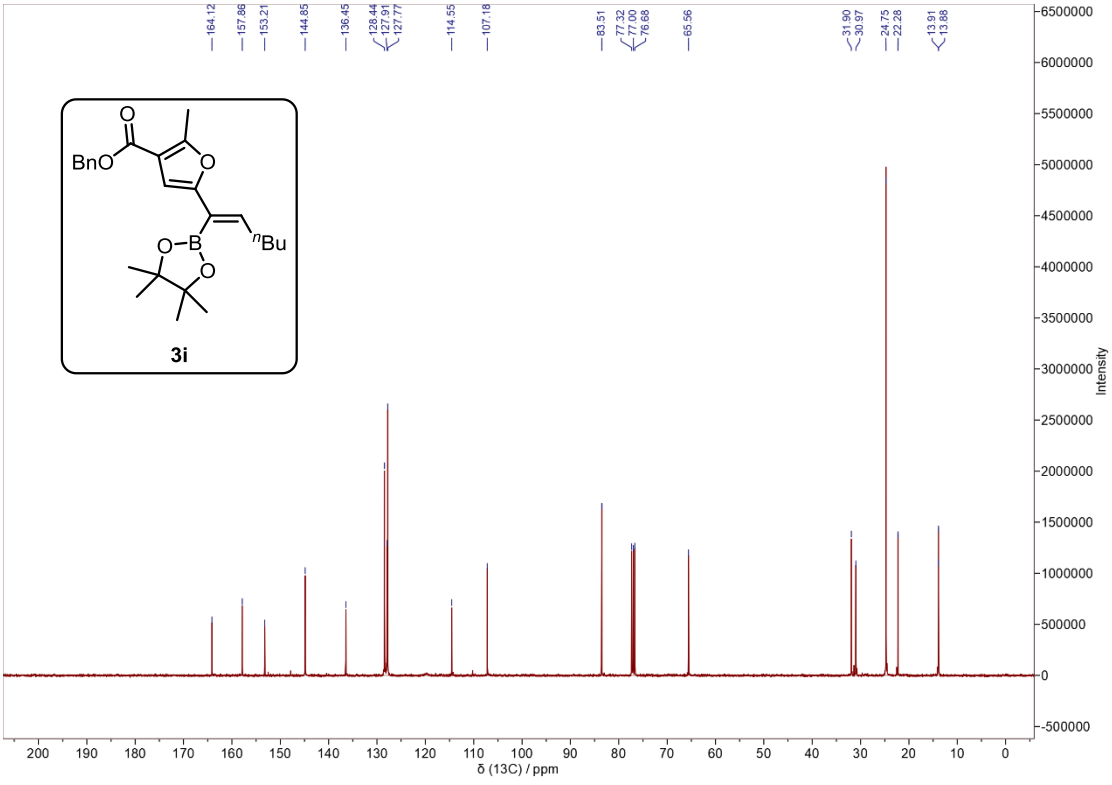

3g
δ (1H) / ppm
Intensity

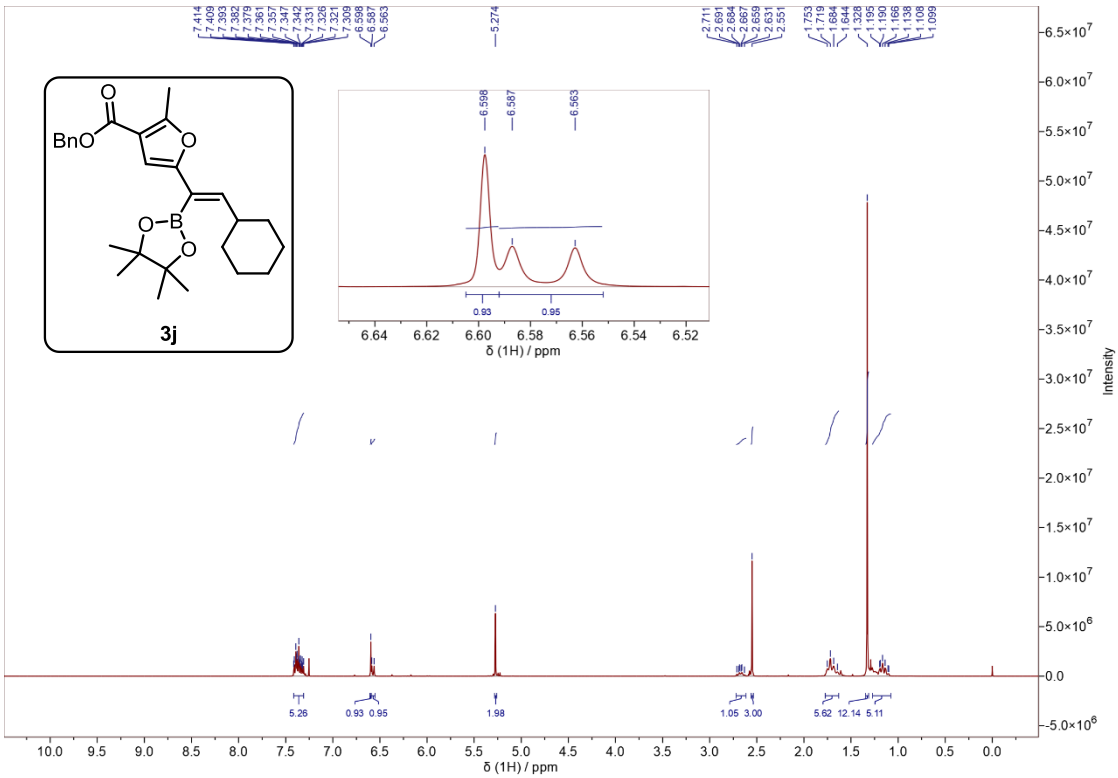

3g
δ (13C) / ppm
Intensity

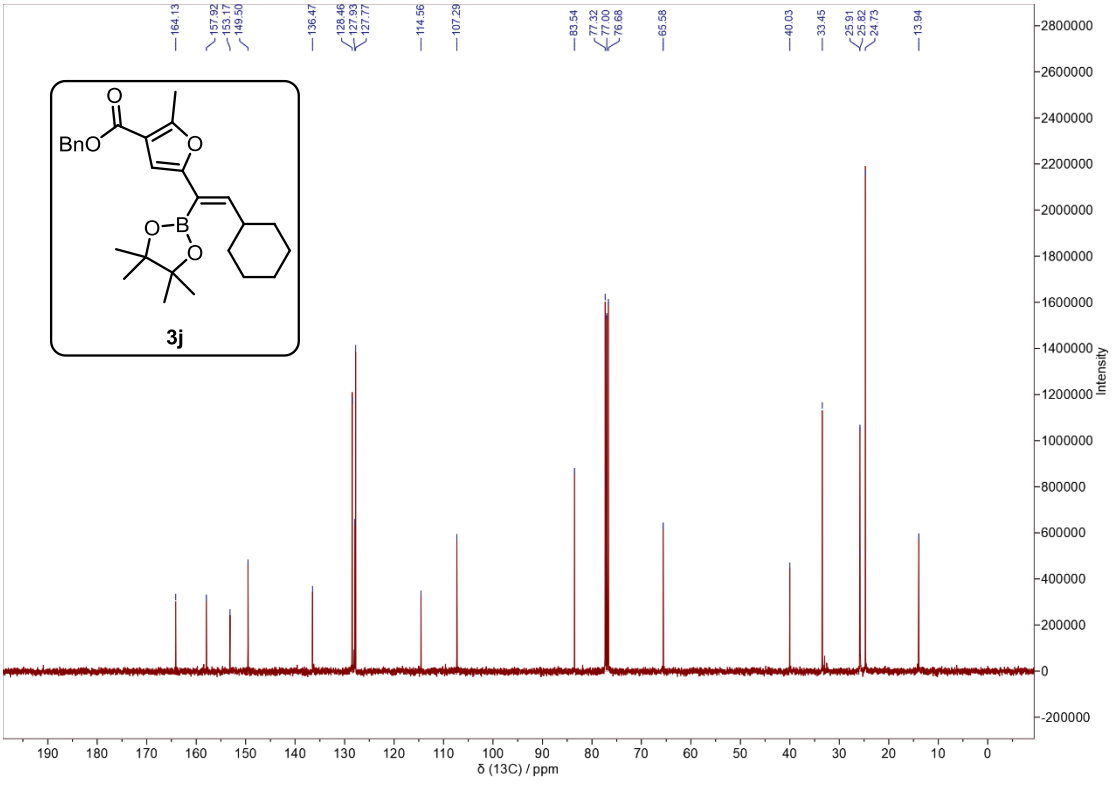

3h
δ (1H) / ppm
Intensity

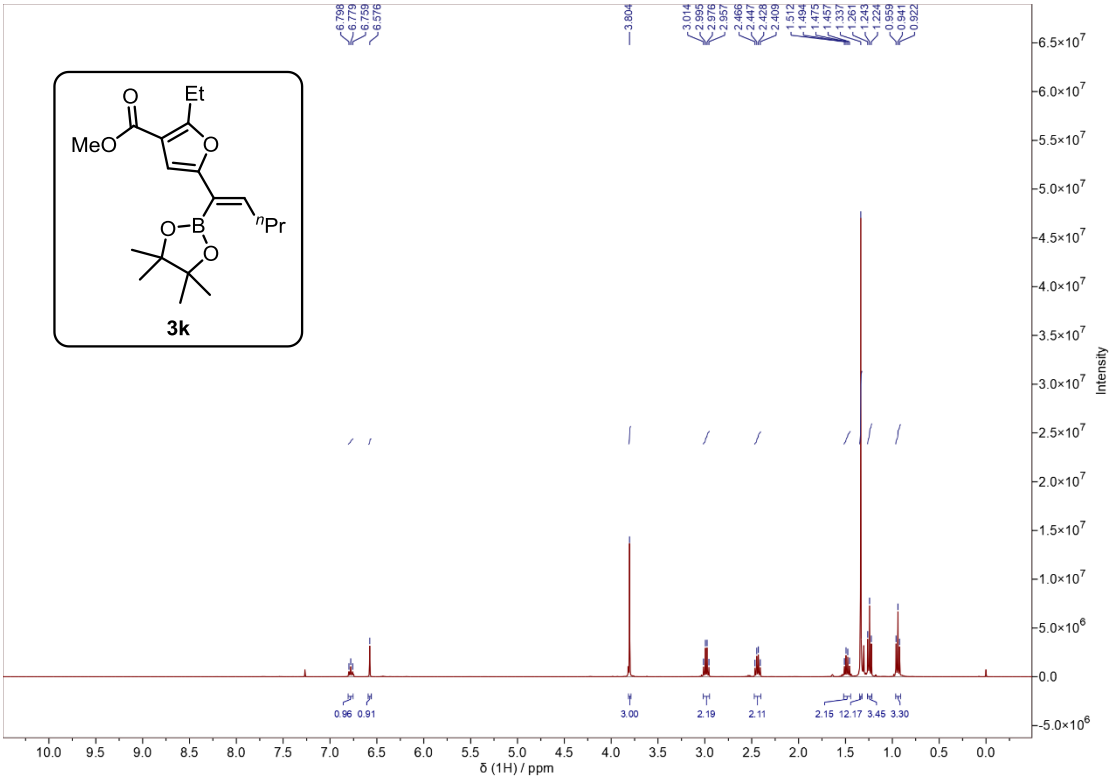

3h
δ (13C) / ppm
Intensity

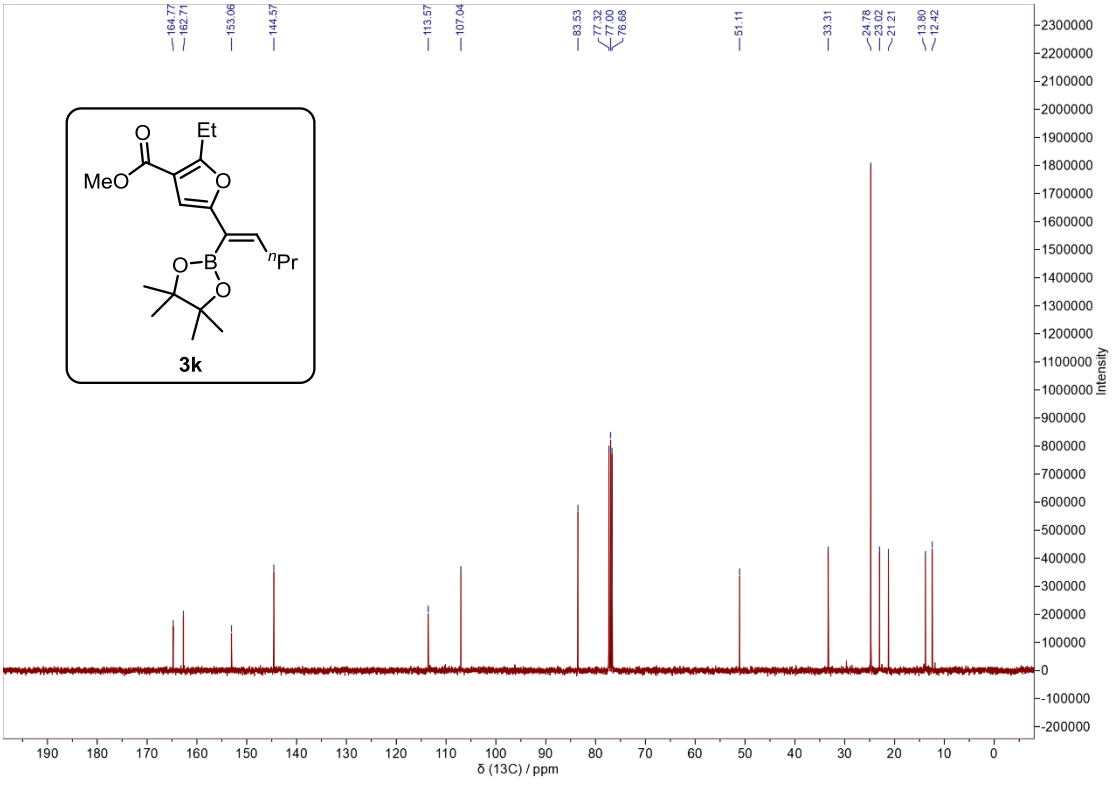

7.411
7.396
7.377
7.359
7.341
7.323
7.306
7.289
6.794
6.774
6.754
6.635
5.278
2.551
2.494
2.465
2.447
2.428
1.455
1.435
1.418
1.398
1.378
1.360
1.324
0.929
0.911
0.893
BnO
O
O
O
O
B
nBu
3i
Intensity
6.0×10^7
5.5×10^7
5.0×10^7
4.5×10^7
4.0×10^7
3.5×10^7
3.0×10^7
2.5×10^7
2.0×10^7
1.5×10^7
1.0×10^7
5.0×10^6
0.0
-5.0×10^6
5.50
1.01
0.94
2.07
3.00
2.18
4.46
12.41
3.24
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ (1H) / ppm

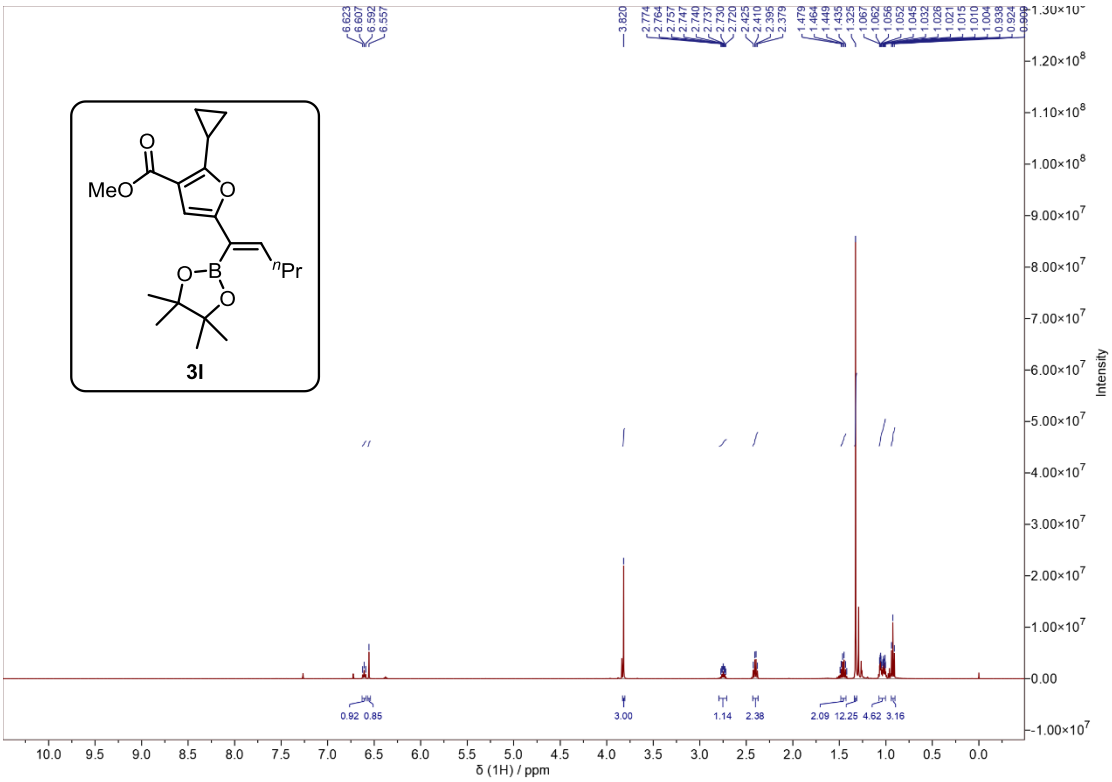

164.12
157.86
153.21
144.85
136.45
128.44
127.91
127.77
114.55
107.18
83.51
77.32
77.00
76.68
65.56
31.90
30.97
24.75
22.28
13.91
13.88
BnO
O
O
O
O
B
nBu
3i
Intensity
6500000
6000000
5500000
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
-500000
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (13C) / ppm

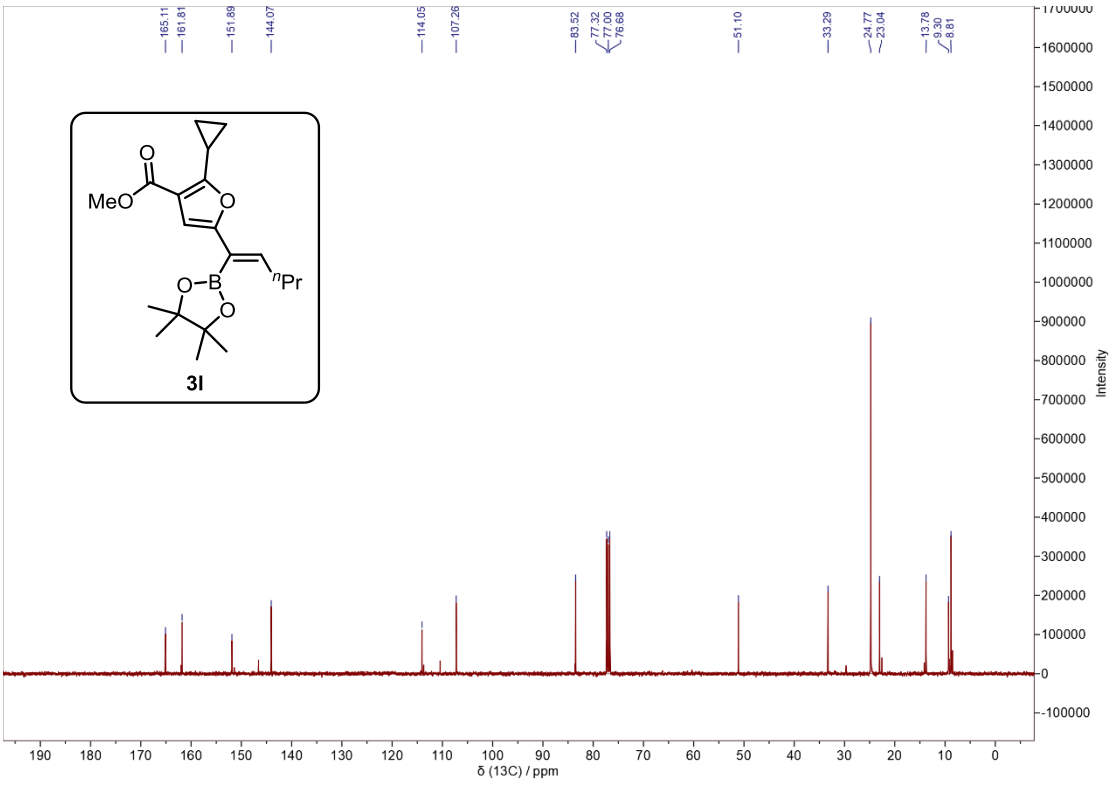

3j
δ (1H) / ppm
Intensity

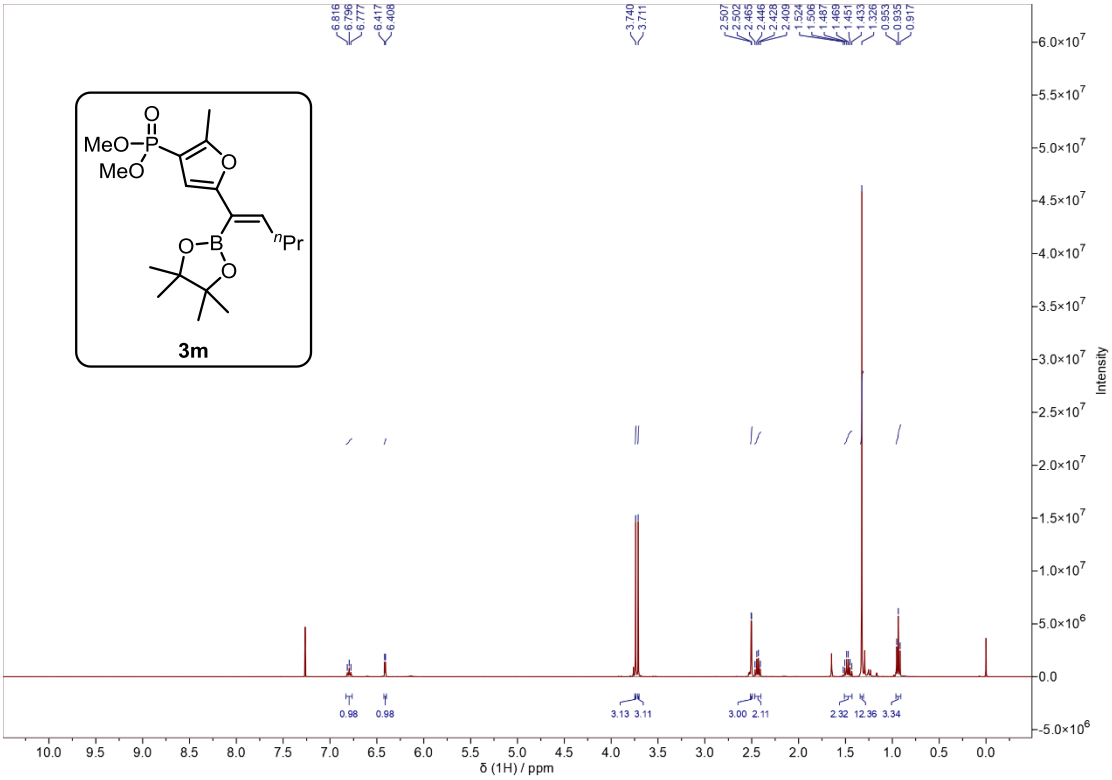

3j
δ (13C) / ppm
Intensity

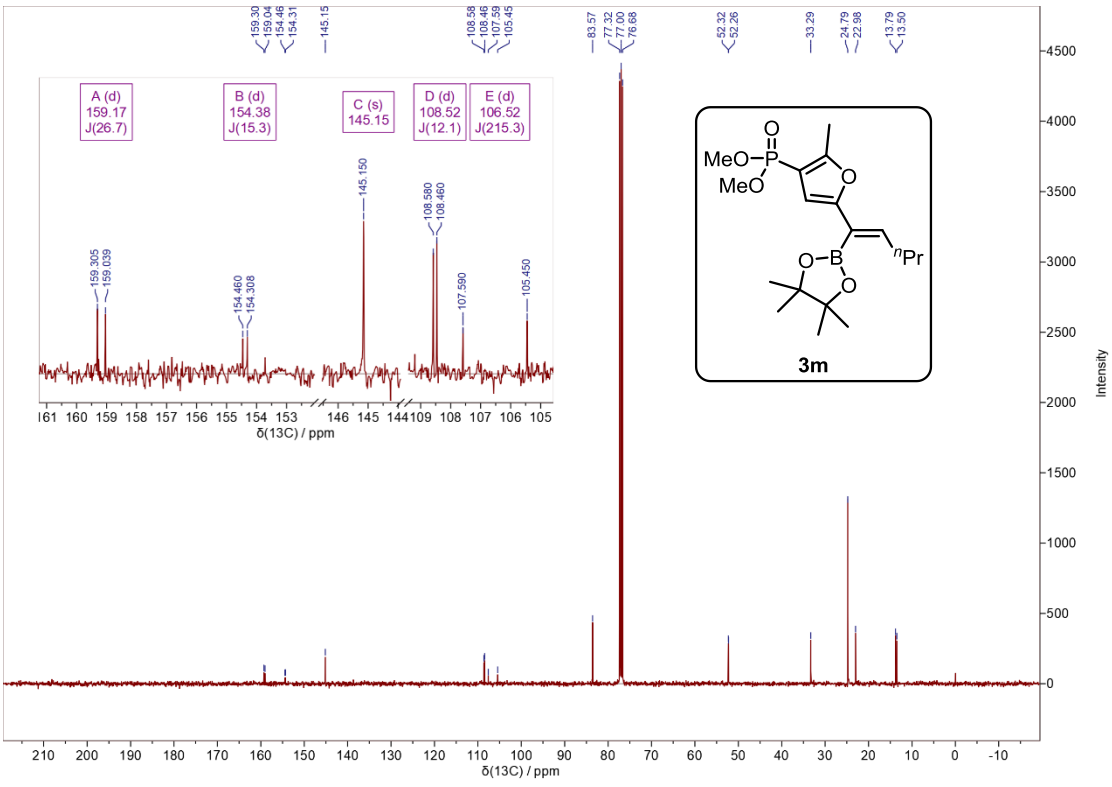

6.798
6.779
6.759
6.576
3.804
3.014
2.995
2.978
2.957
2.466
2.447
2.428
2.409
1.512
1.494
1.475
1.457
1.337
1.261
1.243
1.224
0.959
0.941
0.922
Et
O
MeO
O
O-B
O
nPr
3k
Intensity
0.96 0.91
3.00
2.19
2.11
2.15 12.17 3.45 3.30
δ (1H) / ppm

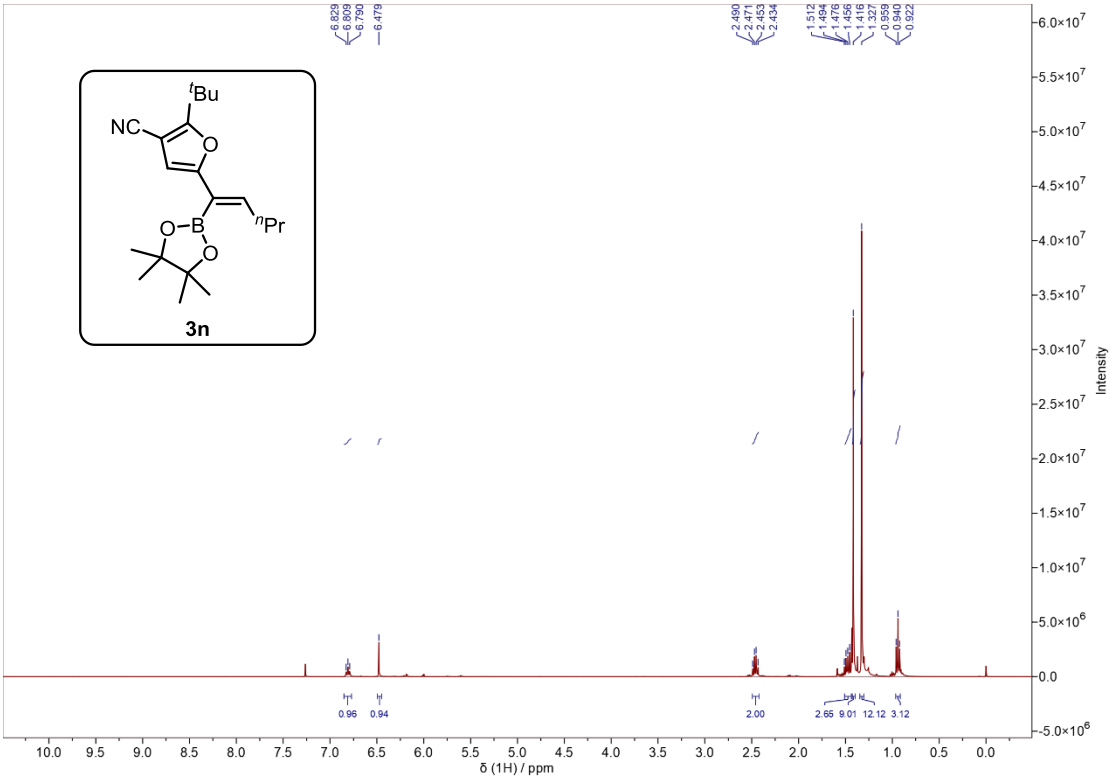

164.77
162.71
153.06
144.57
113.57
107.04
83.53
77.32
77.00
76.68
51.11
33.31
24.78
23.02
21.21
13.80
12.42
Et
O
MeO
O
O-B
O
nPr
3k
Intensity
δ (13C) / ppm

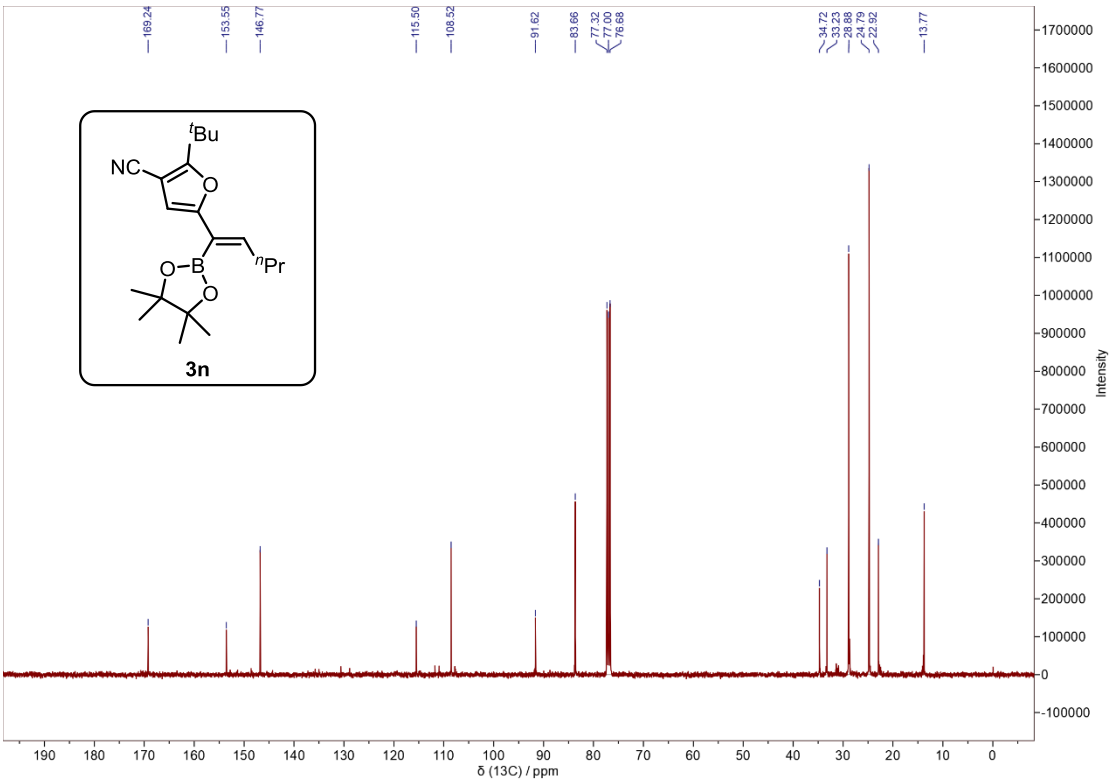

6.623
6.607
6.592
6.557
3.820
2.774
2.764
2.757
2.747
2.740
2.737
2.730
2.720
2.425
2.410
2.395
2.379
1.479
1.464
1.448
1.435
1.325
1.067
1.062
1.056
1.052
1.045
1.032
1.026
1.021
1.015
1.010
1.004
0.938
0.924
MeO
O
O
O-B
O
nPr
3l
Intensity
0.92 0.85
3.00
1.14
2.38
2.09 12.25 4.62 3.16
δ (1H) / ppm

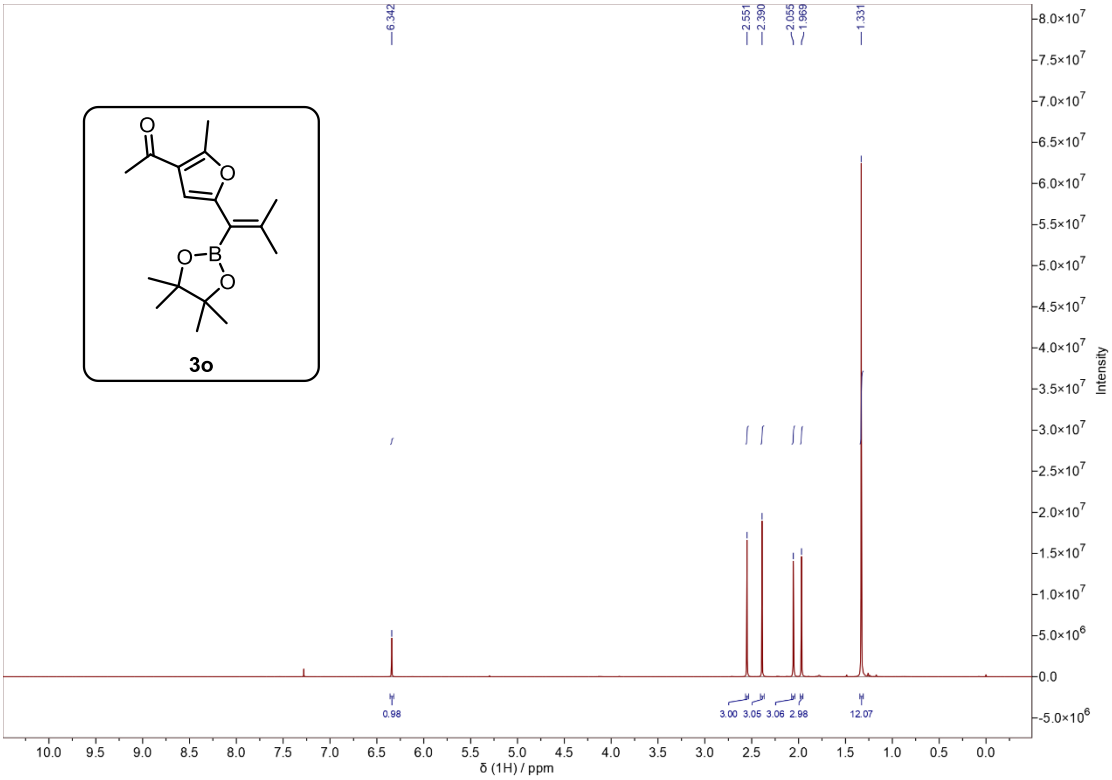

165.11
161.81
151.89
144.07
114.05
107.26
83.52
77.32
77.00
76.68
51.10
33.29
24.77
23.04
13.78
8.30
8.81
MeO
O
O
O-B
O
nPr
3l
Intensity
δ (13C) / ppm

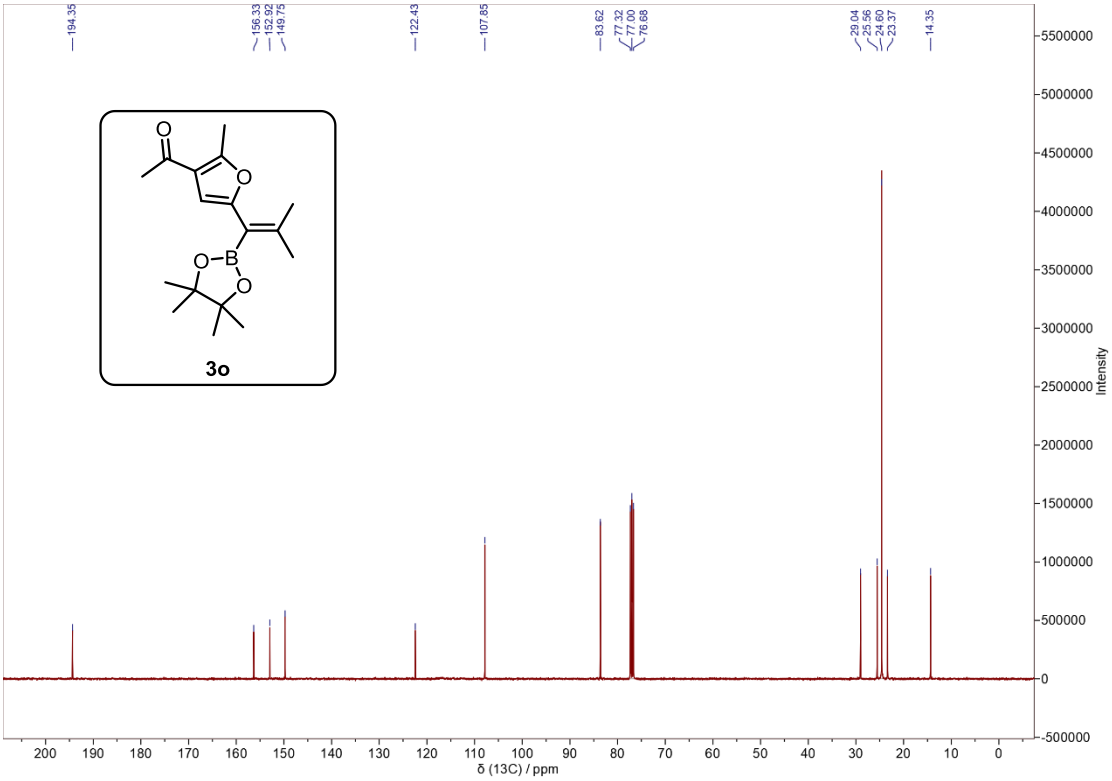

6.816
6.796
6.777
6.417
6.408
3.740
3.711
2.507
2.502
2.465
2.446
2.428
2.409
1.524
1.505
1.497
1.469
1.451
1.433
1.326
0.953
0.935
0.917
MeO
MeO
O
O
O
B
O
nPr
3m
Intensity
0.98
0.98
3.13
3.11
3.00
2.11
2.32
12.36
3.34
δ (1H) / ppm

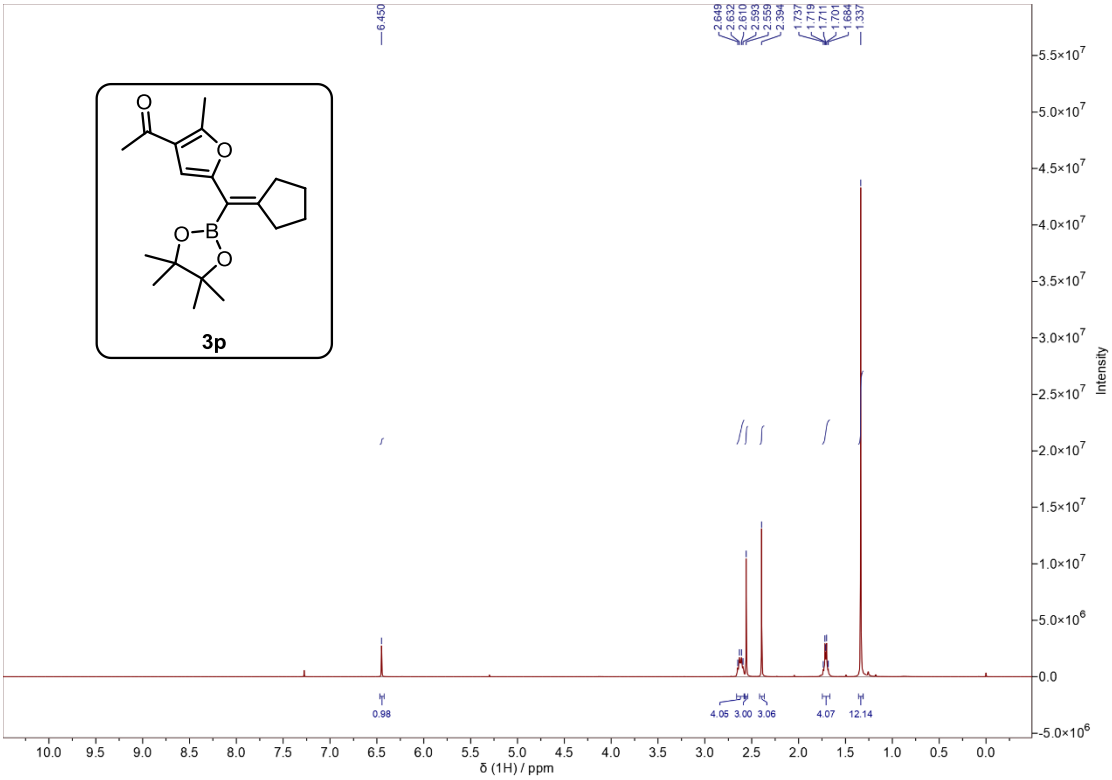

159.30
159.04
154.46
154.31
145.15
108.58
108.46
107.59
105.45
83.57
77.32
77.00
76.68
52.32
52.26
33.29
24.79
22.98
13.79
13.50
A (d)
159.17
J(26.7)
B (d)
154.38
J(15.3)
C (s)
145.15
D (d)
108.52
J(12.1)
E (d)
106.52
J(215.3)
159.305
159.039
154.460
154.308
145.150
108.580
108.460
107.590
105.450
δ(13C) / ppm
MeO
MeO
O
O
O
B
O
nPr
3m
Intensity
δ(13C) / ppm

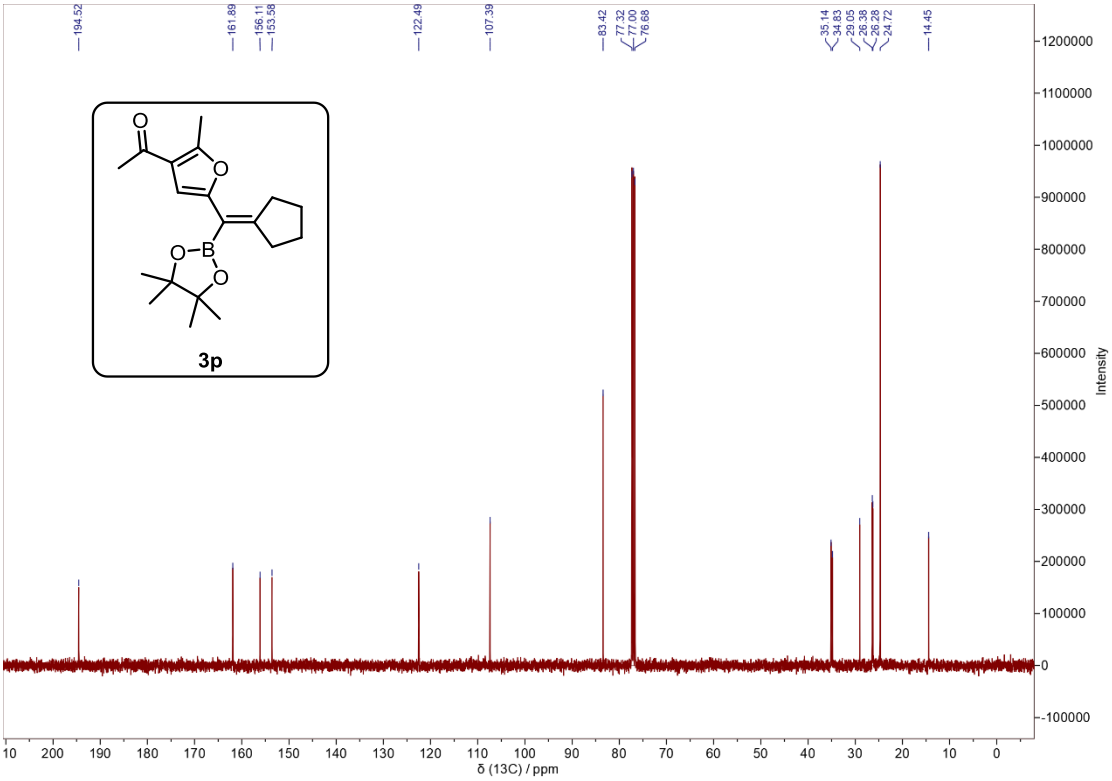

6.829
6.809
6.790
6.479
2.490
2.471
2.453
2.434
1.512
1.494
1.476
1.456
1.416
1.327
0.959
0.940
0.922
tBu
NC
O
nPr
O-B
O
3n
Intensity
0.96
0.94
2.00
2.65
9.01
12.12
3.12
δ (1H) / ppm

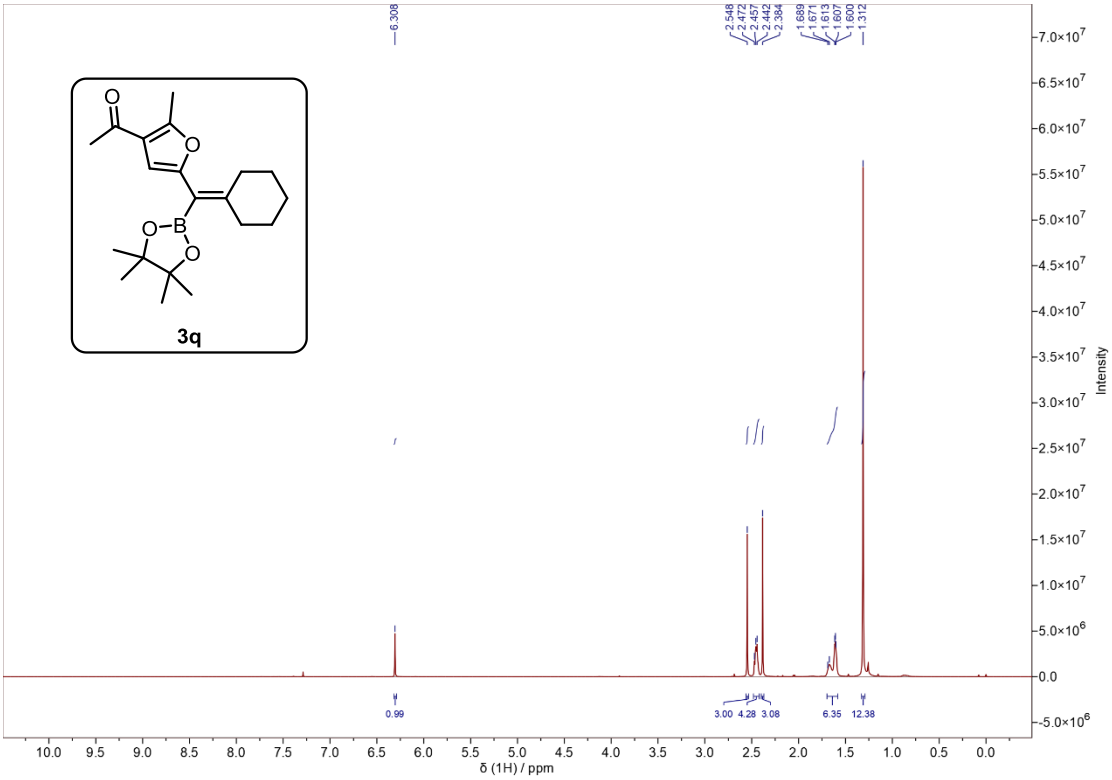

169.24
153.55
146.77
115.50
108.52
91.62
83.66
77.32
77.00
76.68
34.72
33.23
28.88
24.79
22.92
13.77
tBu
NC
O
nPr
O-B
O
3n
Intensity
δ (13C) / ppm

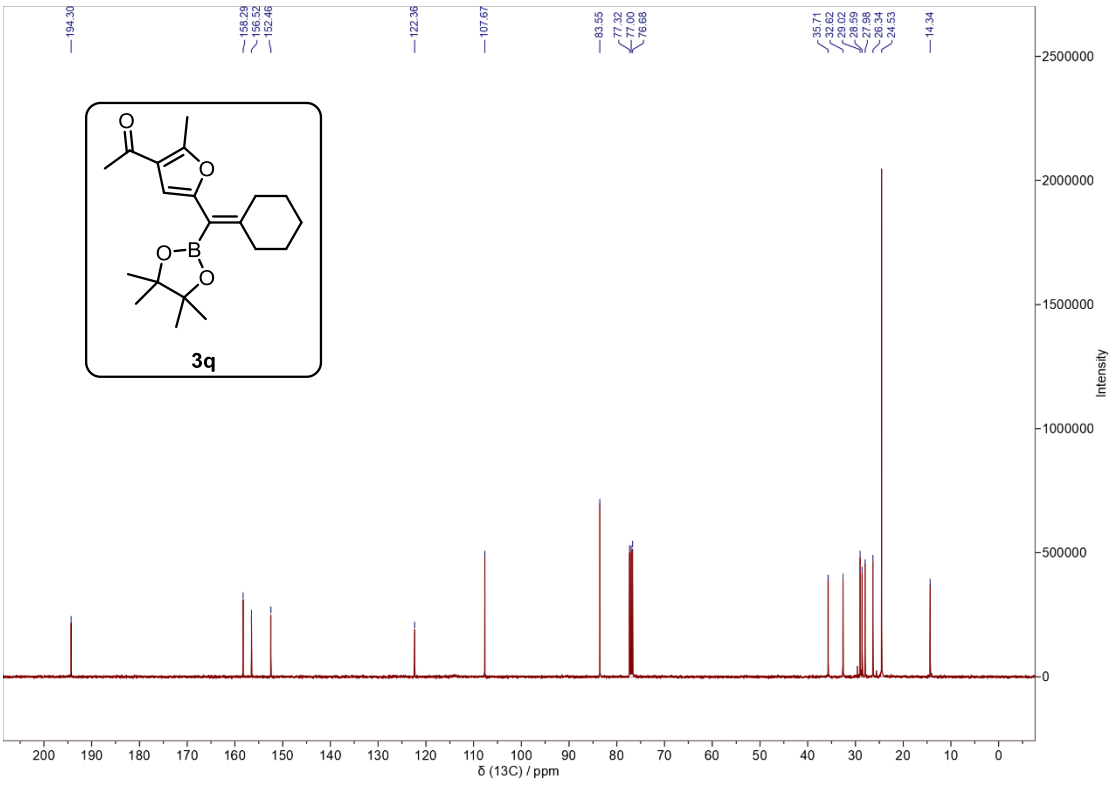

6.342
2.551
2.390
2.055
1.969
1.331
3o
0.98
3.00
3.05
3.06
2.98
12.07
δ (1H) / ppm
Intensity

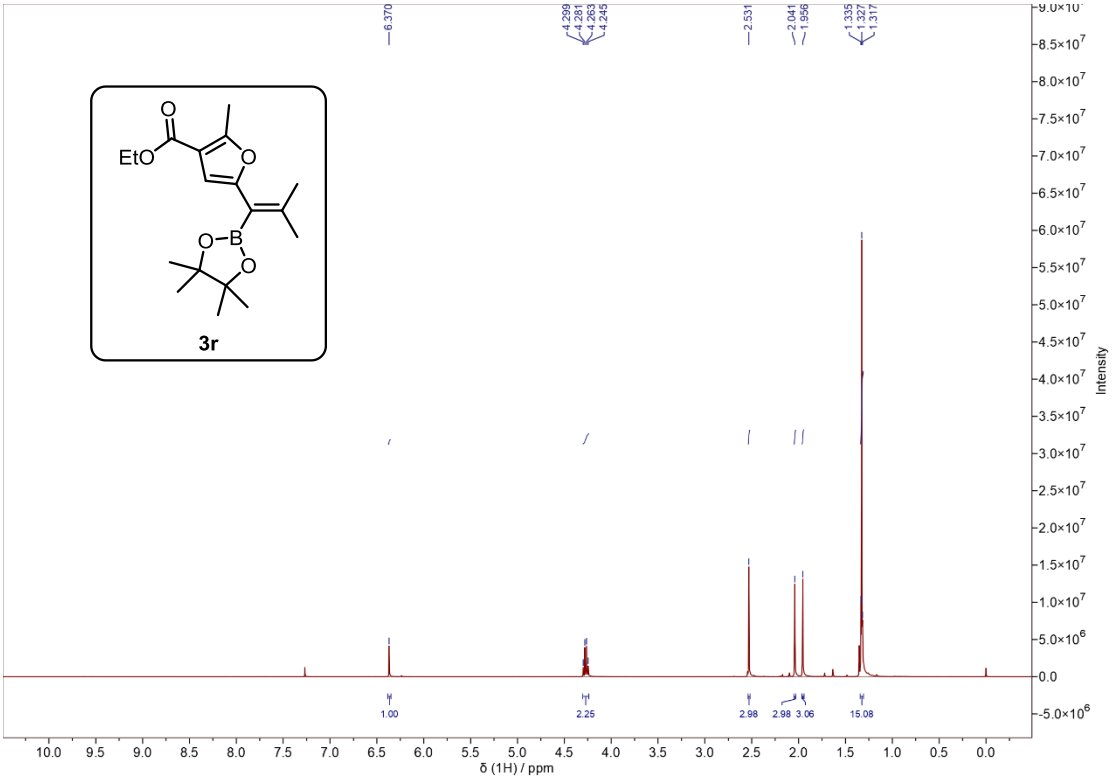

194.35
156.33
152.92
149.75
122.43
107.85
83.62
77.32
77.00
76.68
29.04
25.56
24.60
23.37
14.35
3o
δ (13C) / ppm
Intensity

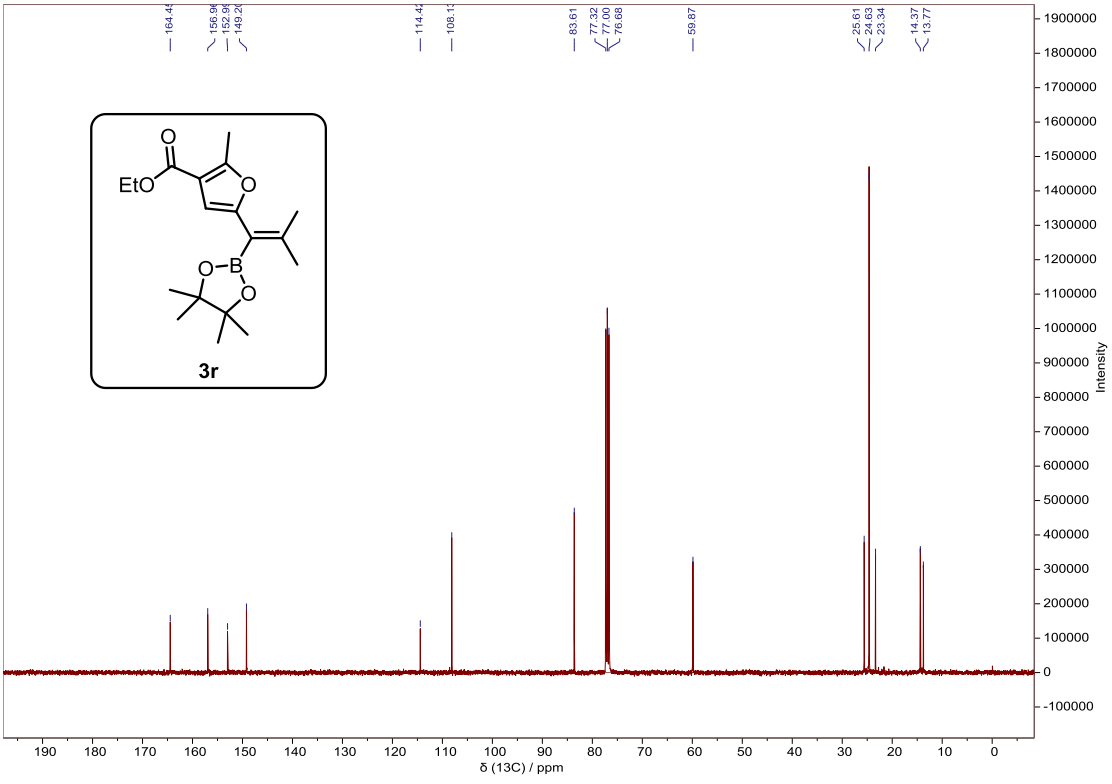

6.450
2.649
2.632
2.610
2.585
2.559
2.394
1.737
1.719
1.711
1.701
1.684
1.337
3p
0.98
4.05
3.00
3.06
4.07
12.14
δ (1H) / ppm
Intensity

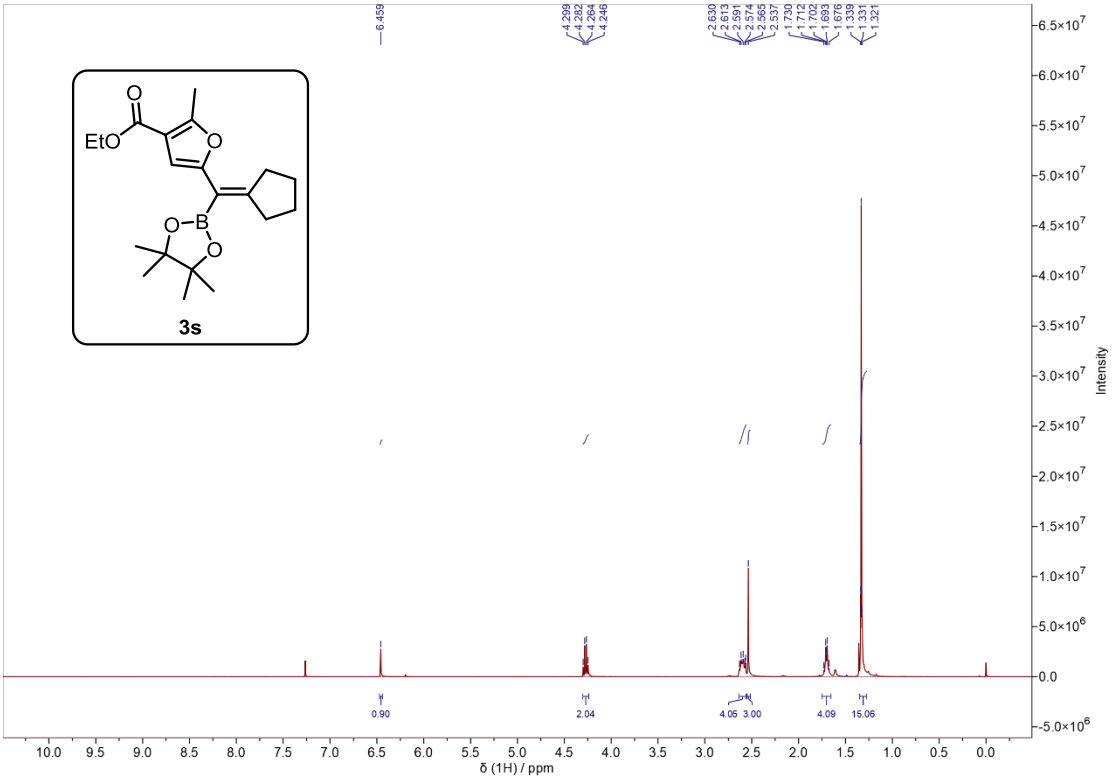

194.52
161.89
156.11
153.58
122.49
107.39
83.42
77.32
77.00
76.68
35.14
34.83
29.05
26.38
26.28
24.72
14.45
3p
δ (13C) / ppm
Intensity

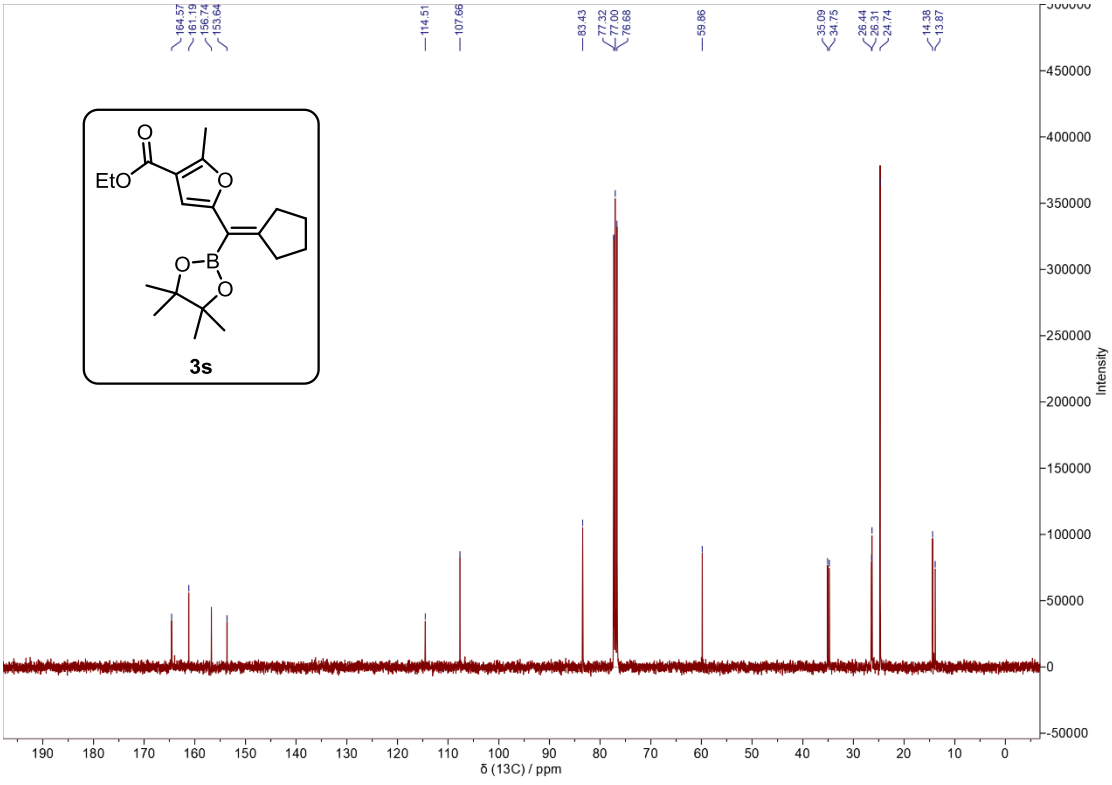

6.308
2.548
2.472
2.457
2.442
2.384
1.689
1.671
1.613
1.607
1.600
1.312
3q
0.99
3.00
4.28
3.08
6.35
12.38
Intensity
δ (1H) / ppm

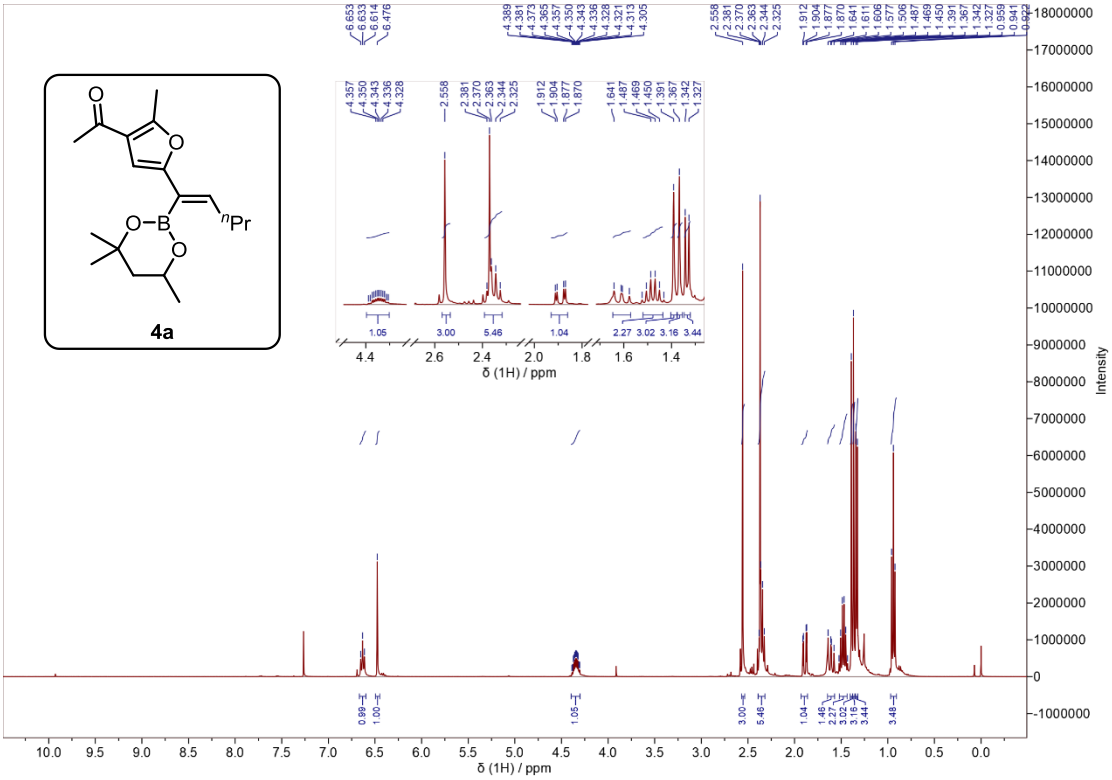

194.30
158.29
156.52
152.46
122.36
107.67
83.55
77.32
77.00
76.68
35.71
32.62
29.02
28.89
27.98
26.34
24.53
14.34
3q
Intensity
δ (13C) / ppm

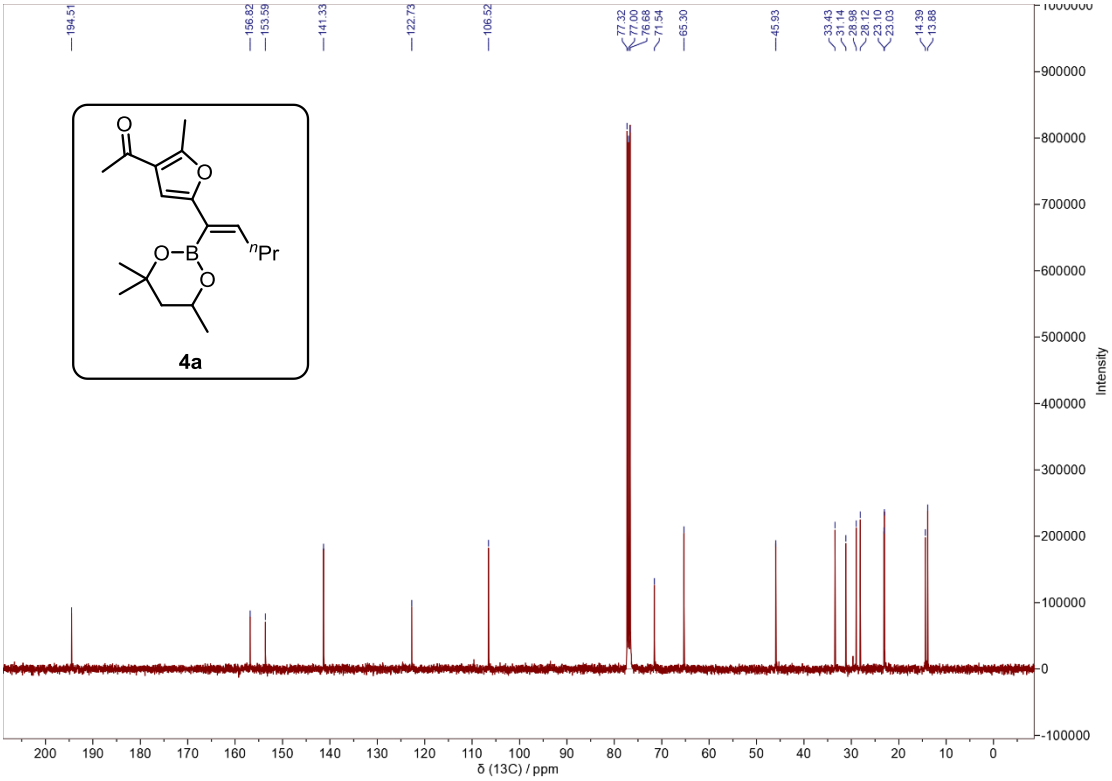

6.370
4.299
4.281
4.263
4.245
2.531
2.041
1.956
1.335
1.327
1.317
1.00
2.25
2.98
2.98
3.06
15.08
δ (1H) / ppm
Intensity
3r

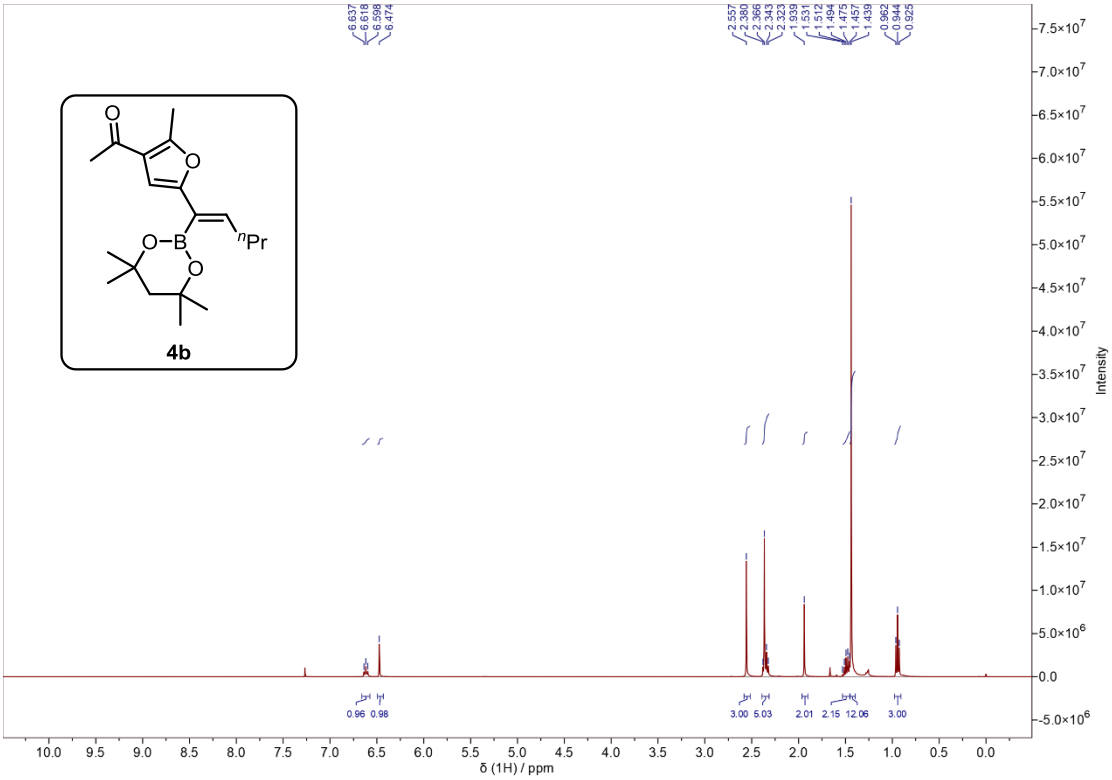

164.44
156.98
152.98
149.20
114.44
108.15
83.61
77.32
77.00
76.68
59.87
25.61
24.63
23.34
14.37
13.77
δ (13C) / ppm
Intensity
3r

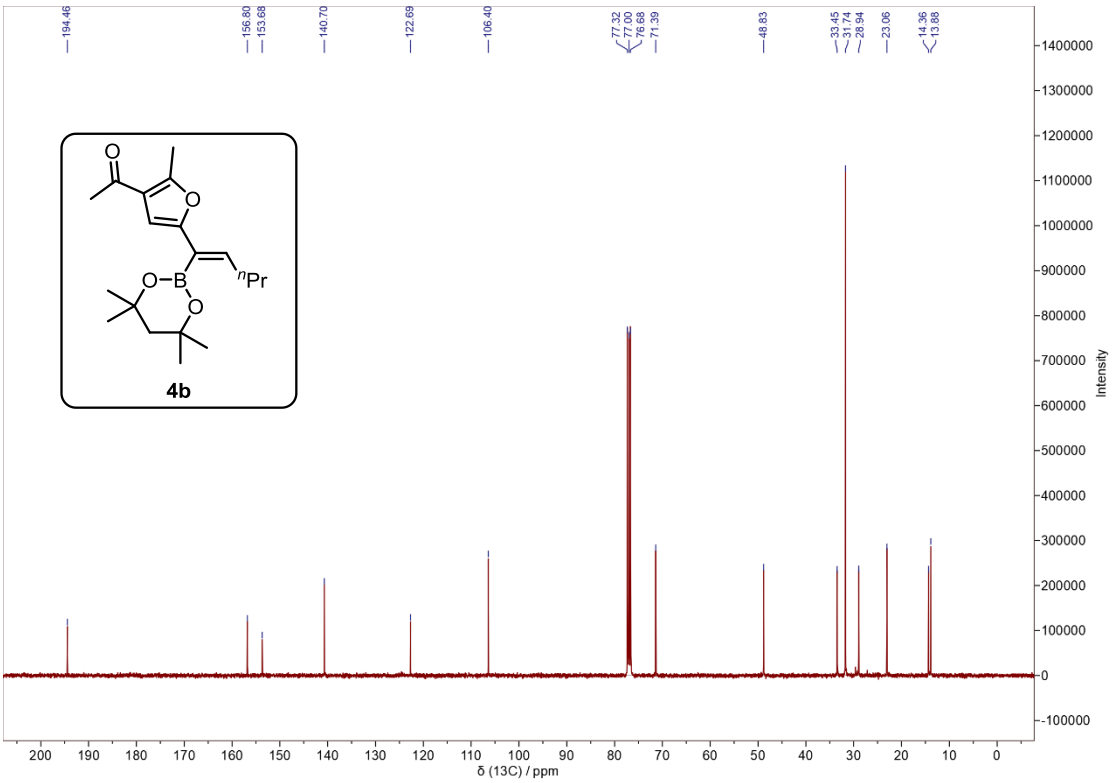

6.459
4.299
4.282
4.264
4.246
2.630
2.613
2.591
2.574
2.565
2.537
1.730
1.712
1.702
1.693
1.676
1.339
1.331
1.321
EtO
O
O
O-B
O
3s
Intensity
0.90
2.04
4.05
3.00
4.09
15.06
δ (1H) / ppm

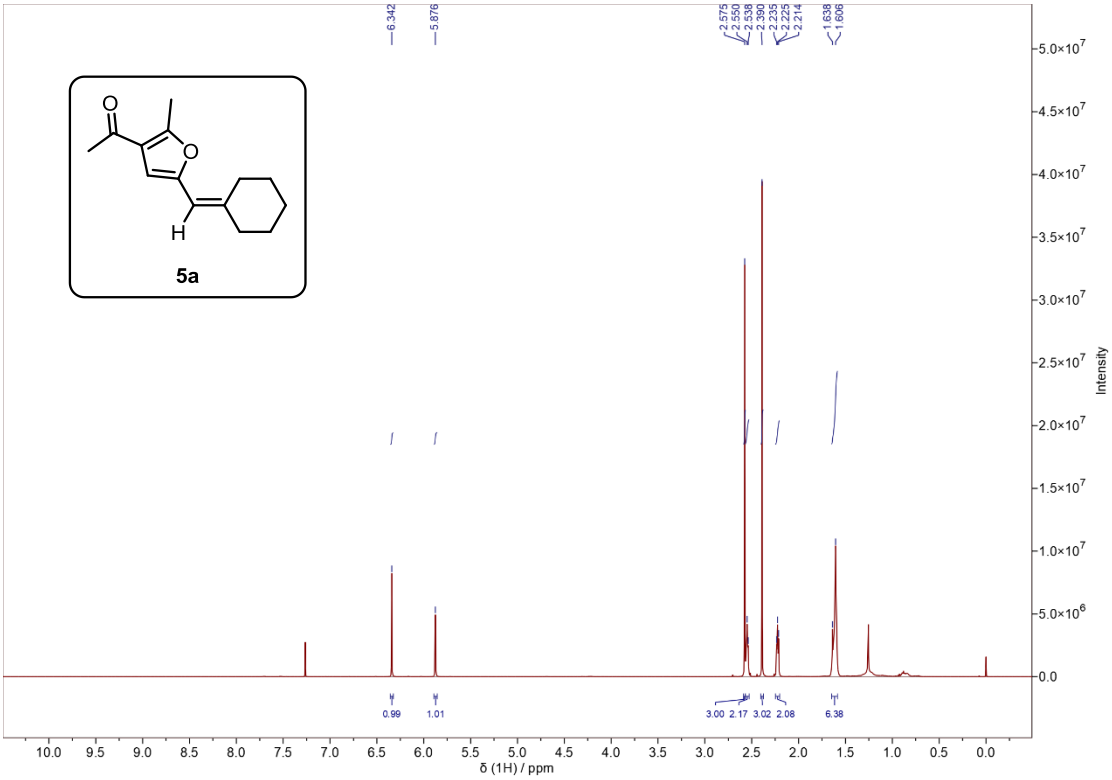

164.57
161.19
156.74
153.64
114.51
107.66
83.43
77.32
77.00
76.68
59.86
35.09
34.75
26.44
26.31
24.74
14.38
13.87
EtO
O
O
O-B
O
3s
Intensity
δ (13C) / ppm

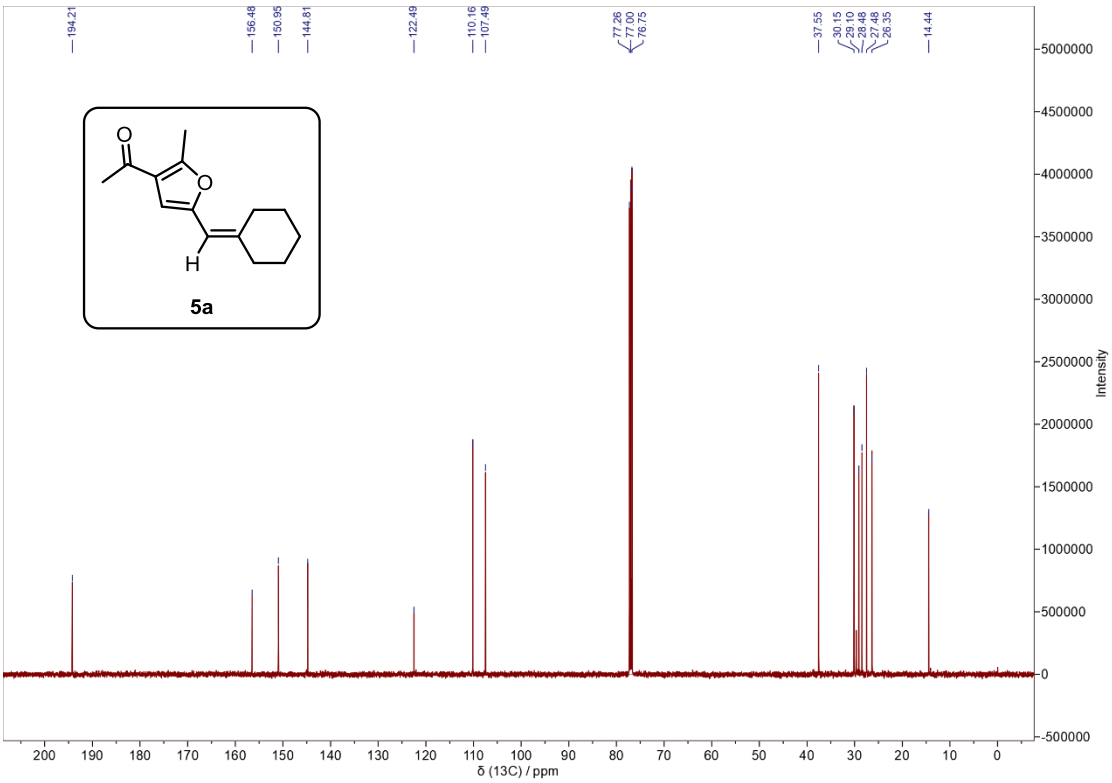

4a
δ (1H) / ppm
Intensity

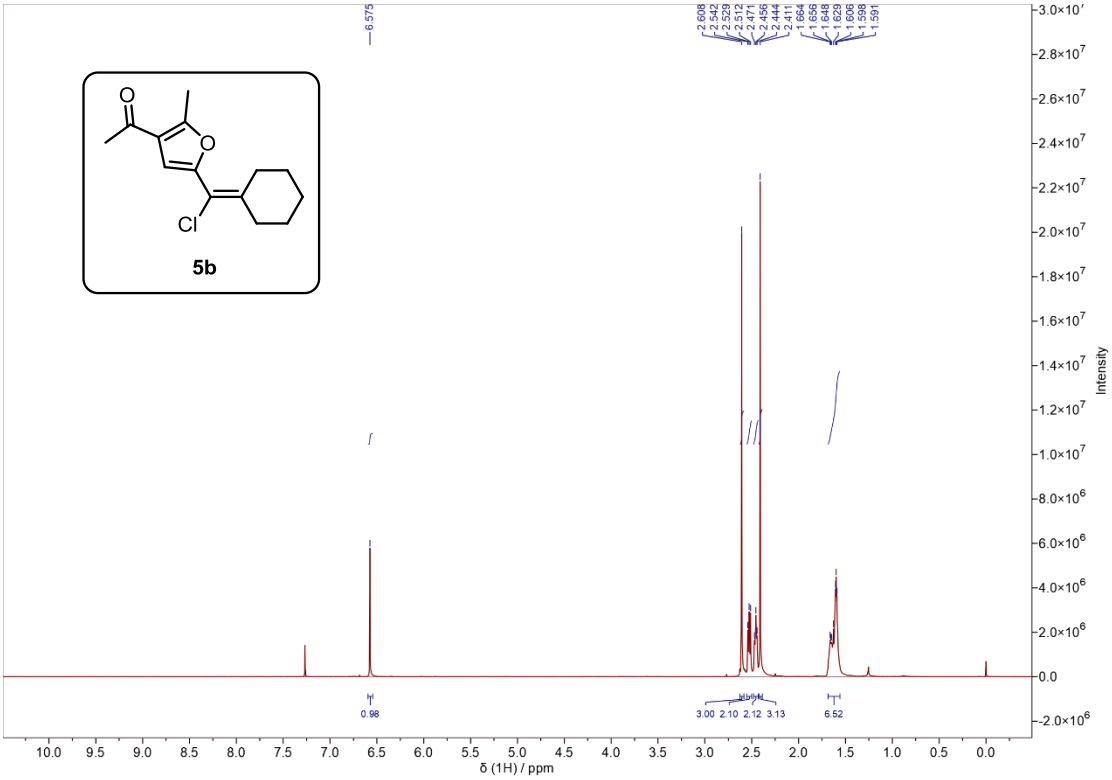

4a
δ (13C) / ppm
Intensity

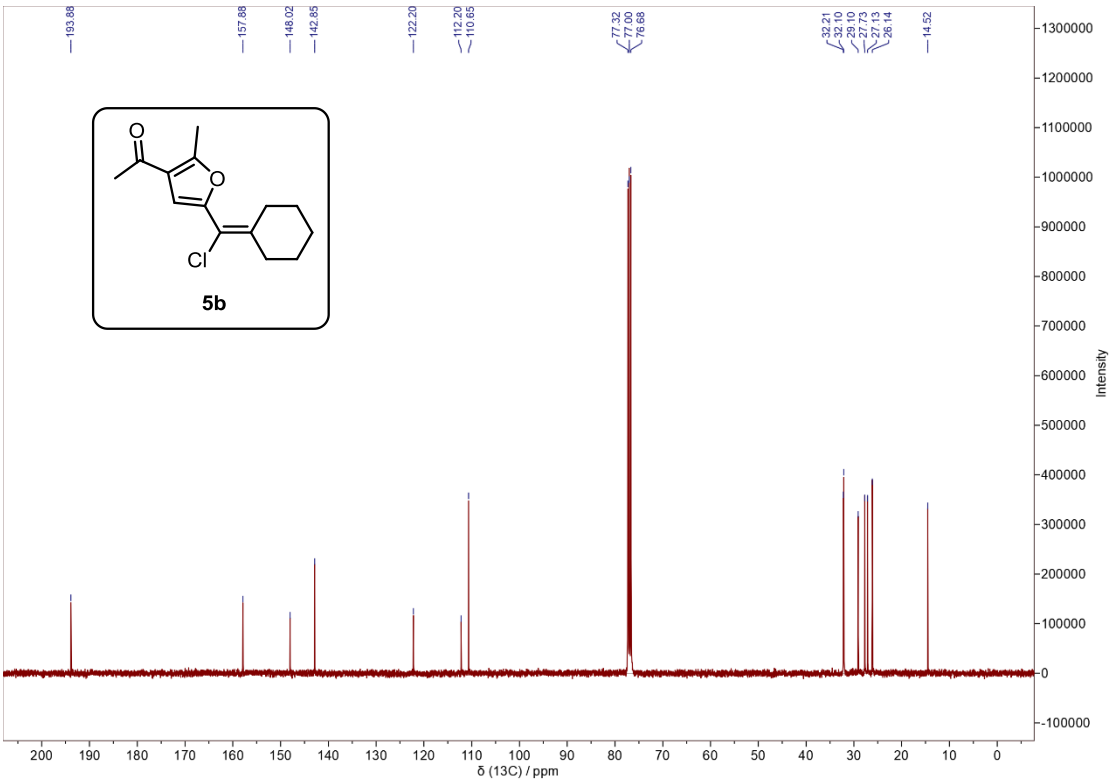

6.637
6.618
6.598
6.474
2.557
2.380
2.366
2.343
2.323
1.939
1.531
1.512
1.494
1.475
1.457
1.439
0.962
0.944
0.925
4b
Intensity
0.96 0.98
3.00 5.03 2.01 2.15 12.06 3.00
δ (1H) / ppm

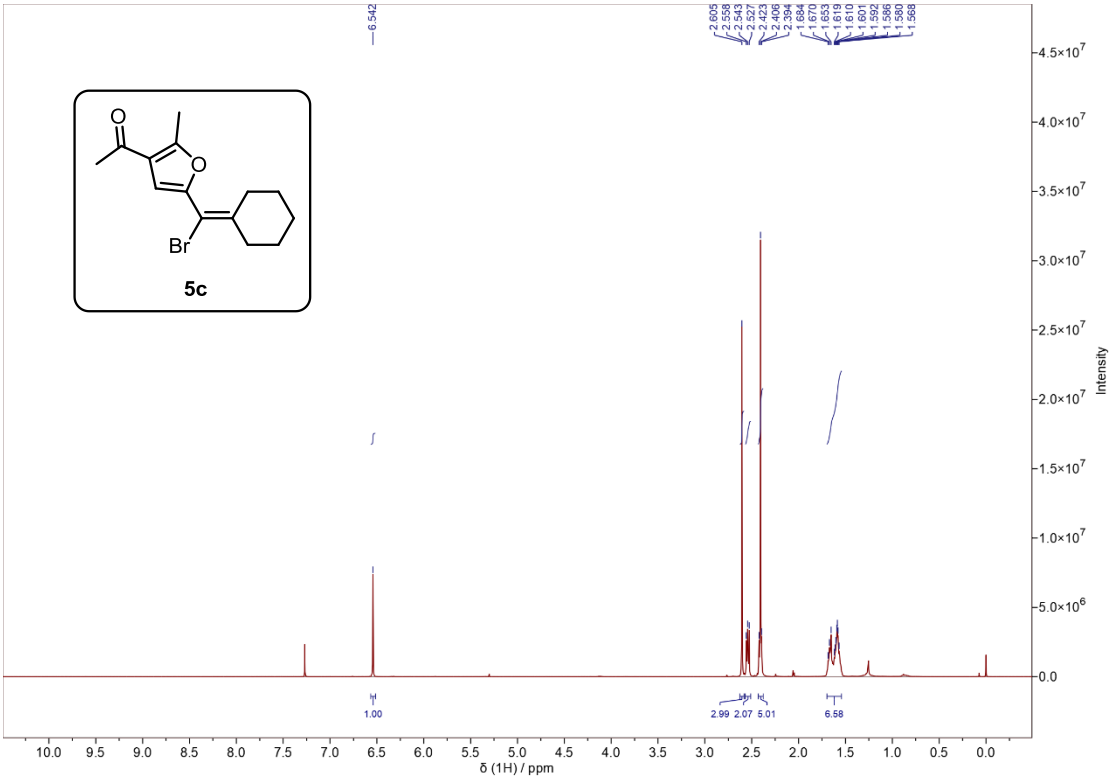

194.46
156.80
153.68
140.70
122.69
106.40
77.32
77.00
76.68
71.39
48.83
33.45
31.74
28.94
23.06
14.36
13.88
4b
Intensity
δ (13C) / ppm

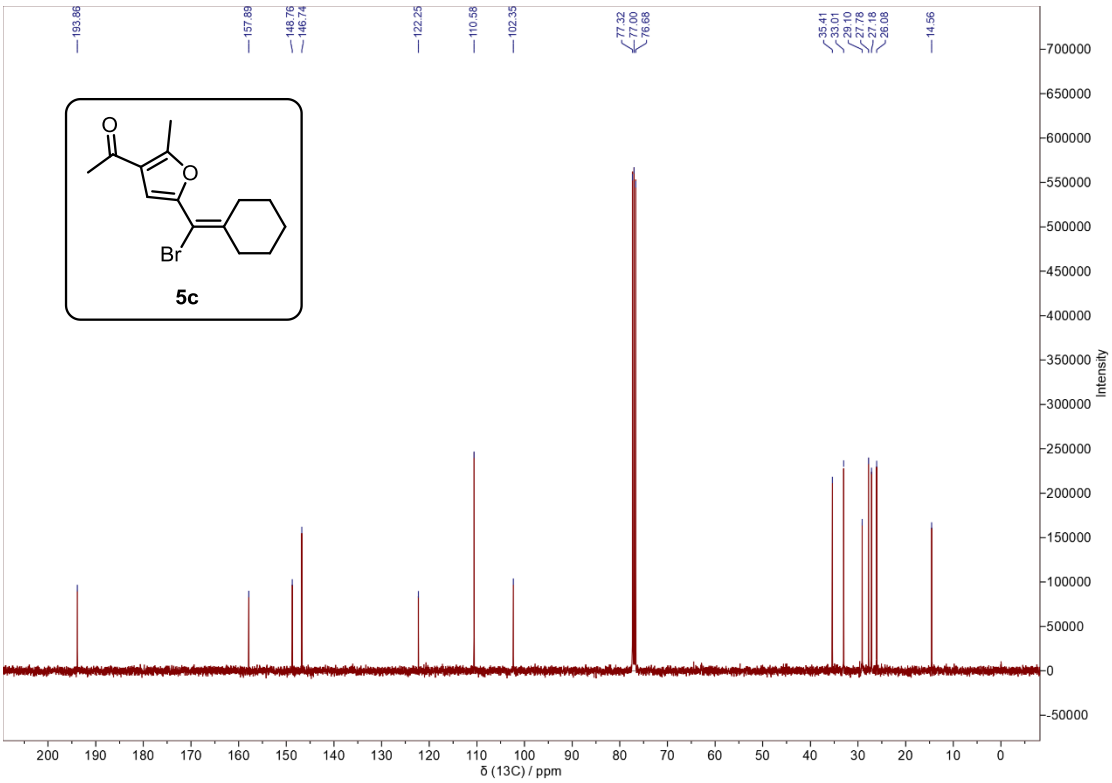

6.342
5.876
2.575
2.550
2.538
2.390
2.235
2.225
2.214
1.638
1.608
5.0×10^7
4.5×10^7
4.0×10^7
3.5×10^7
3.0×10^7
2.5×10^7
2.0×10^7
1.5×10^7
1.0×10^7
5.0×10^6
0.0
Intensity
0.99
1.01
3.00
2.17
3.02
2.08
6.38
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ (1H) / ppm
O
O
H
5a

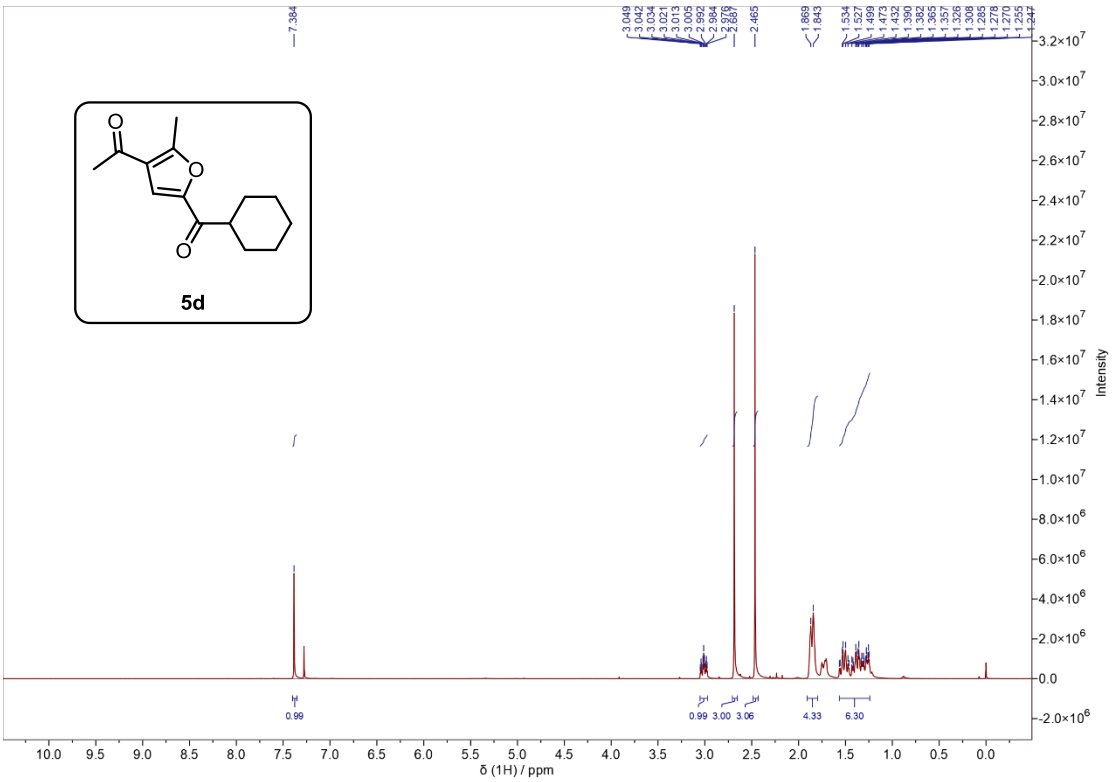

194.21
156.48
150.95
144.81
122.49
110.16
107.49
77.26
77.00
76.75
37.55
30.15
29.10
28.48
27.48
26.35
14.44
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
-500000
Intensity
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (13C) / ppm
O
O
H
5a

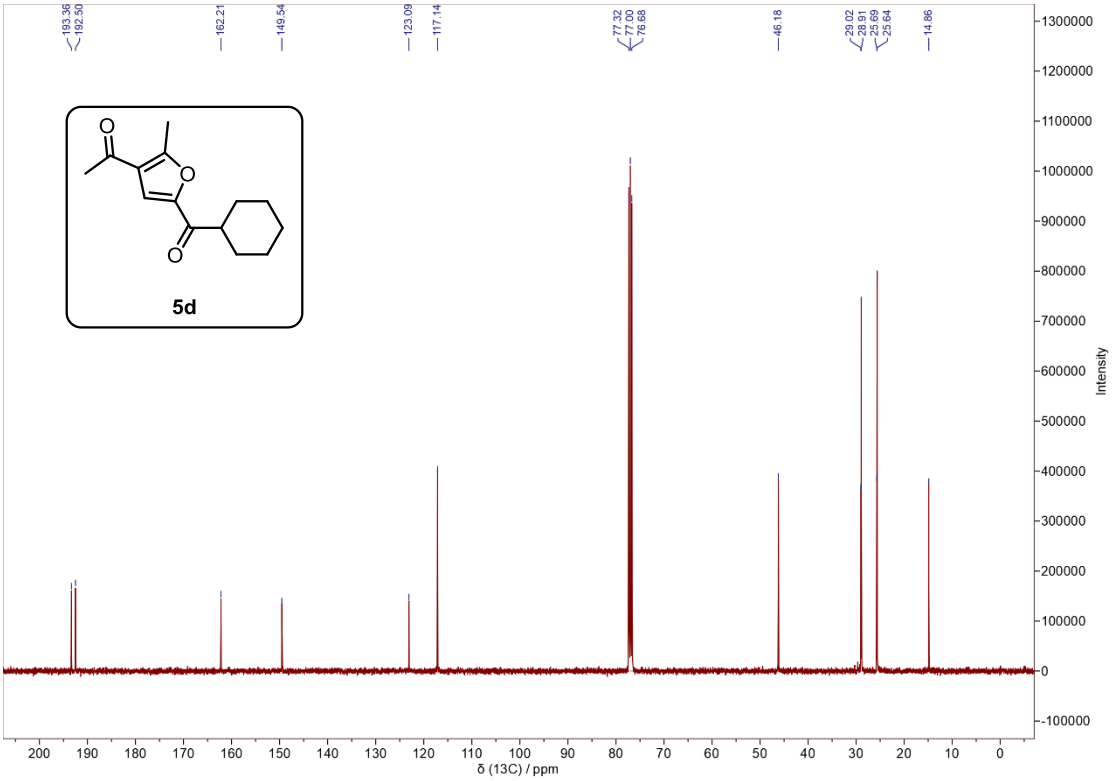

6.575
2.608
2.543
2.529
2.512
2.471
2.456
2.444
2.411
1.664
1.636
1.618
1.629
1.606
1.598
1.591
O
O
Cl
5b
0.98
3.00
2.10
2.12
3.13
6.52
δ (1H) / ppm
Intensity

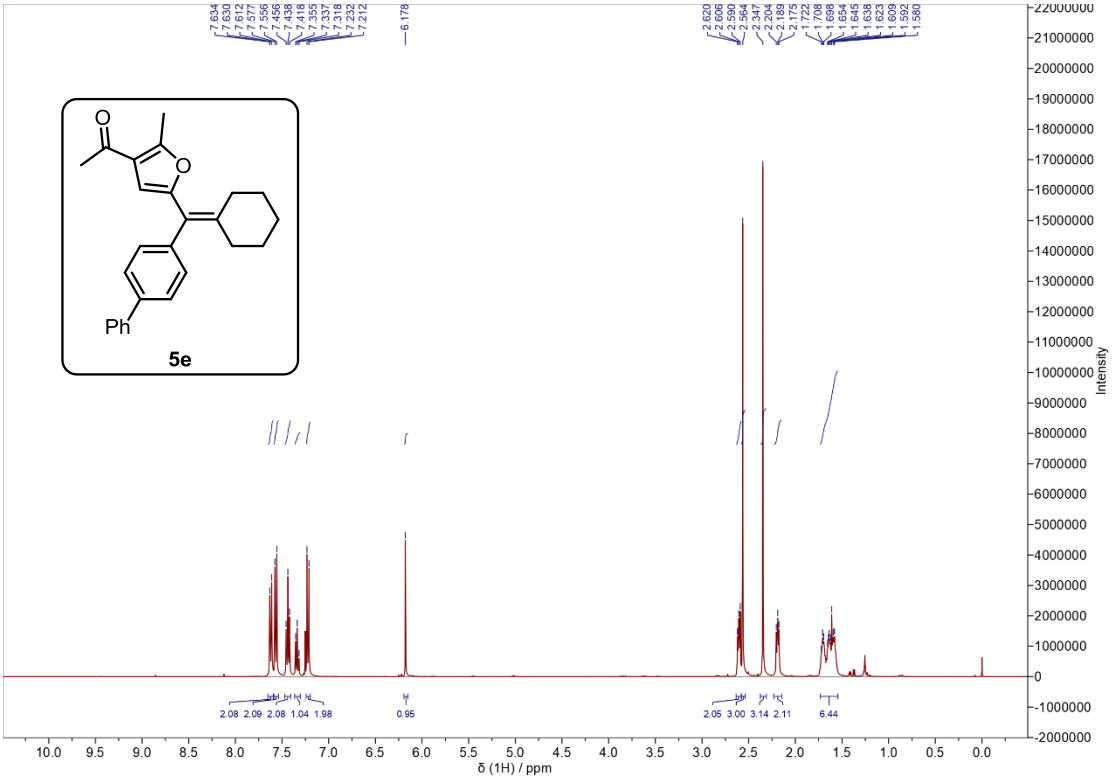

193.88
157.88
148.02
142.85
122.20
112.20
110.65
77.32
77.00
76.68
32.21
32.10
29.10
27.73
27.13
26.14
14.52
O
O
Cl
5b
δ (13C) / ppm
Intensity

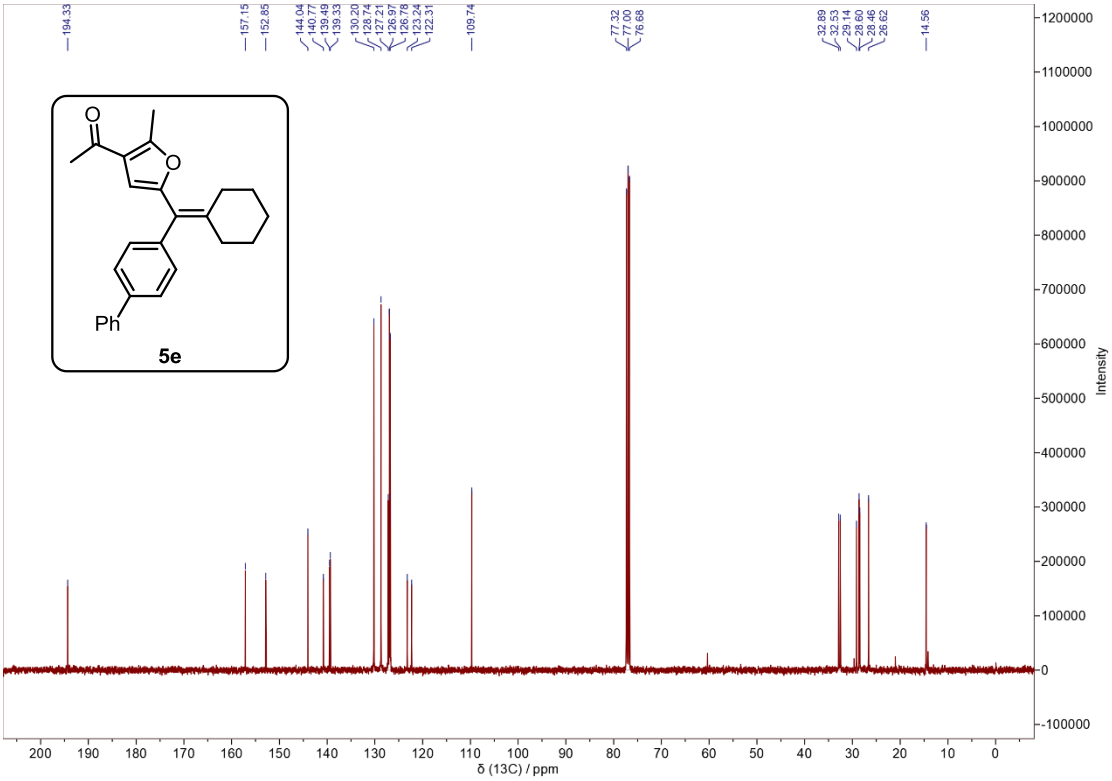

6.542
2.605
2.558
2.543
2.527
2.423
2.406
2.394
1.684
1.670
1.653
1.619
1.610
1.601
1.592
1.586
1.580
1.568
O
O
Br
5c
Intensity
1.00
2.99
2.07
5.01
6.58
δ (1H) / ppm

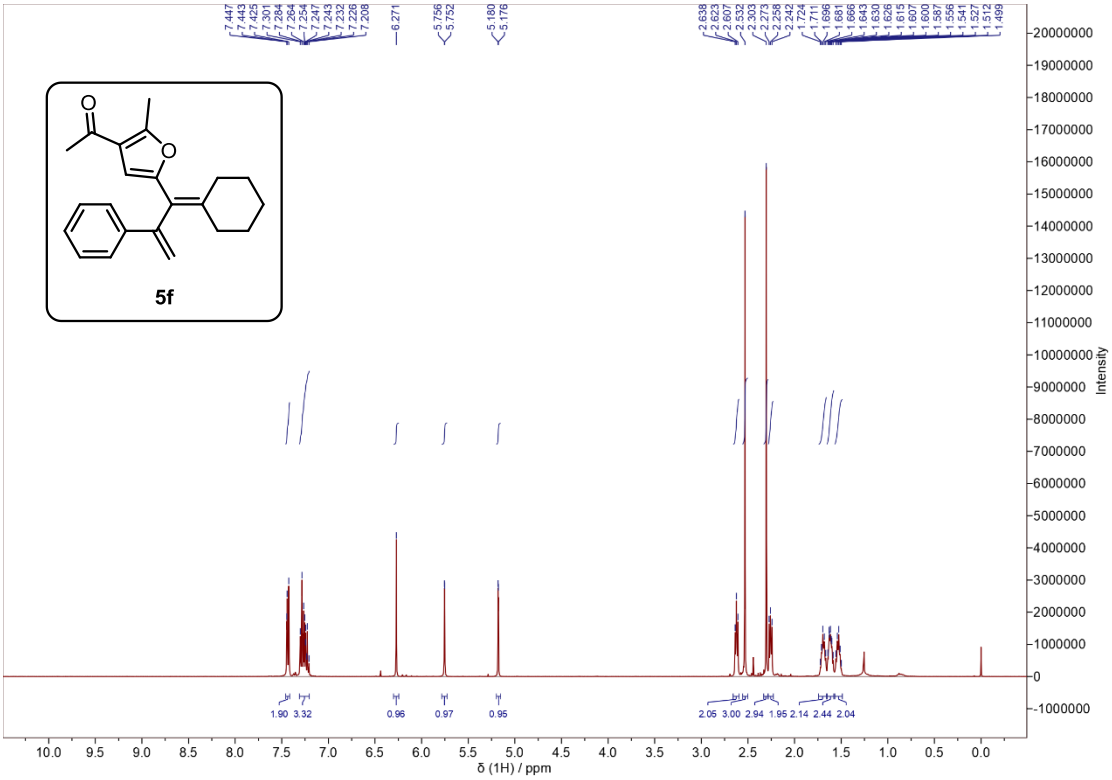

193.86
157.89
148.76
146.74
122.25
110.58
102.35
77.32
77.00
76.68
35.41
33.01
29.10
27.78
27.18
26.08
14.56
O
O
Br
5c
Intensity
δ (13C) / ppm

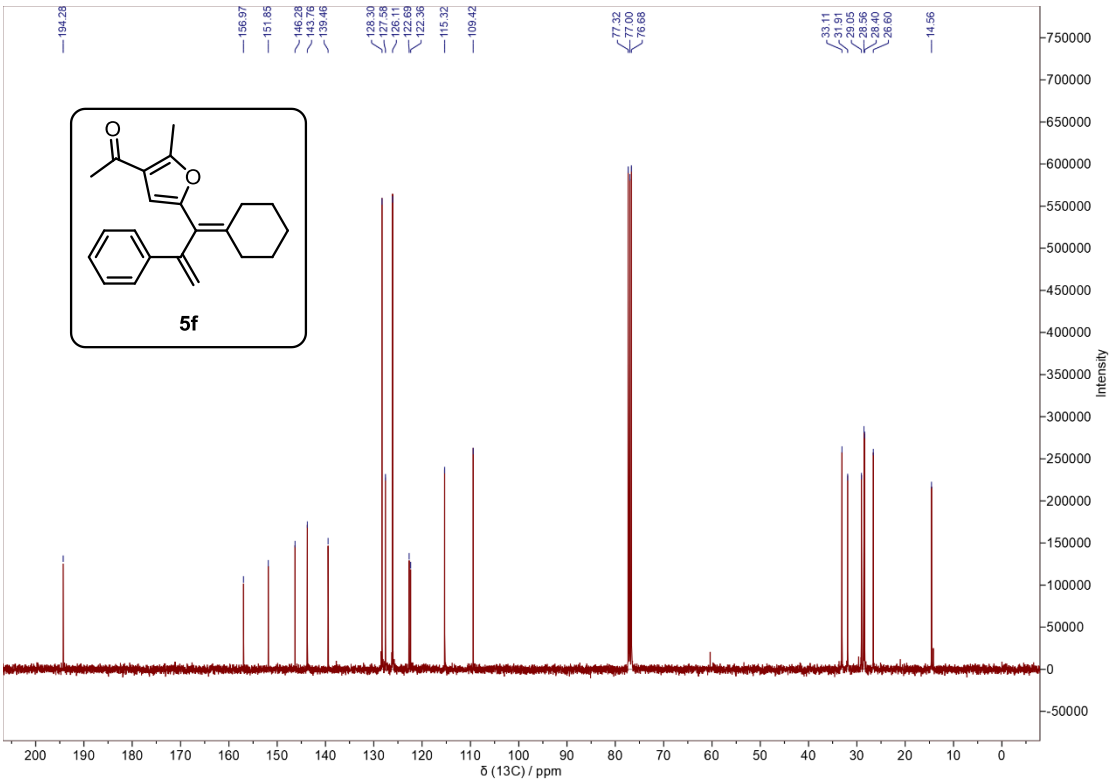

5d
δ (1H) / ppm
Intensity

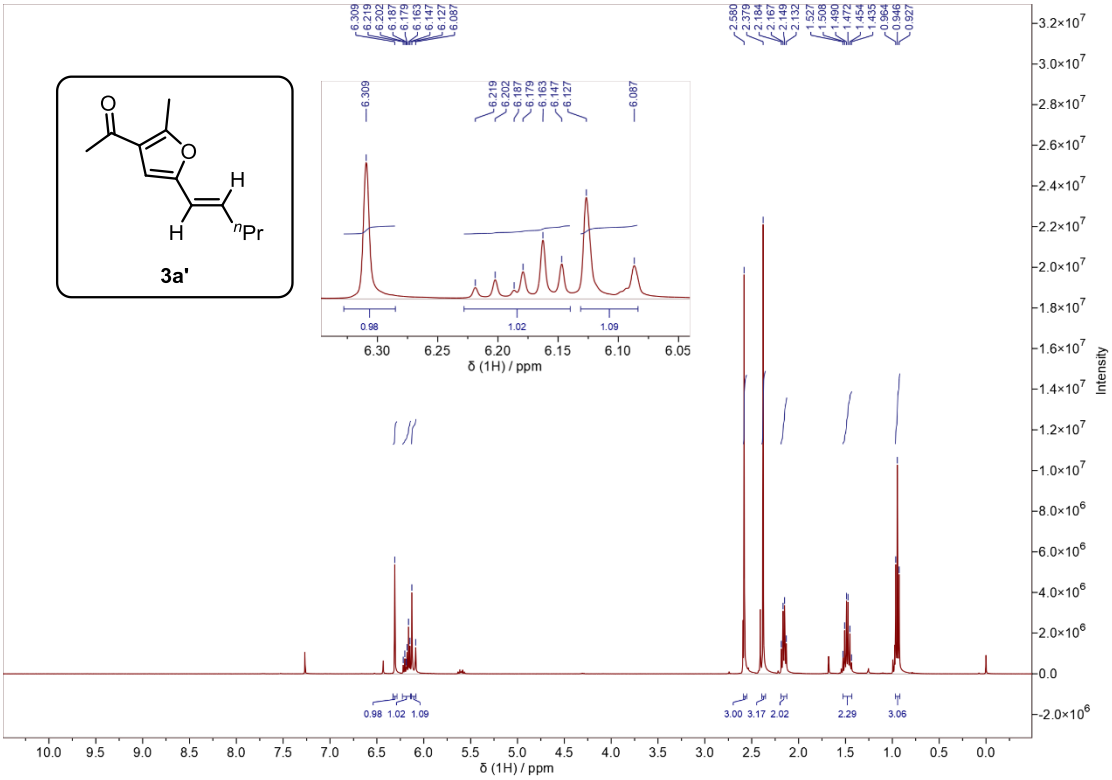

5d
δ (13C) / ppm
Intensity

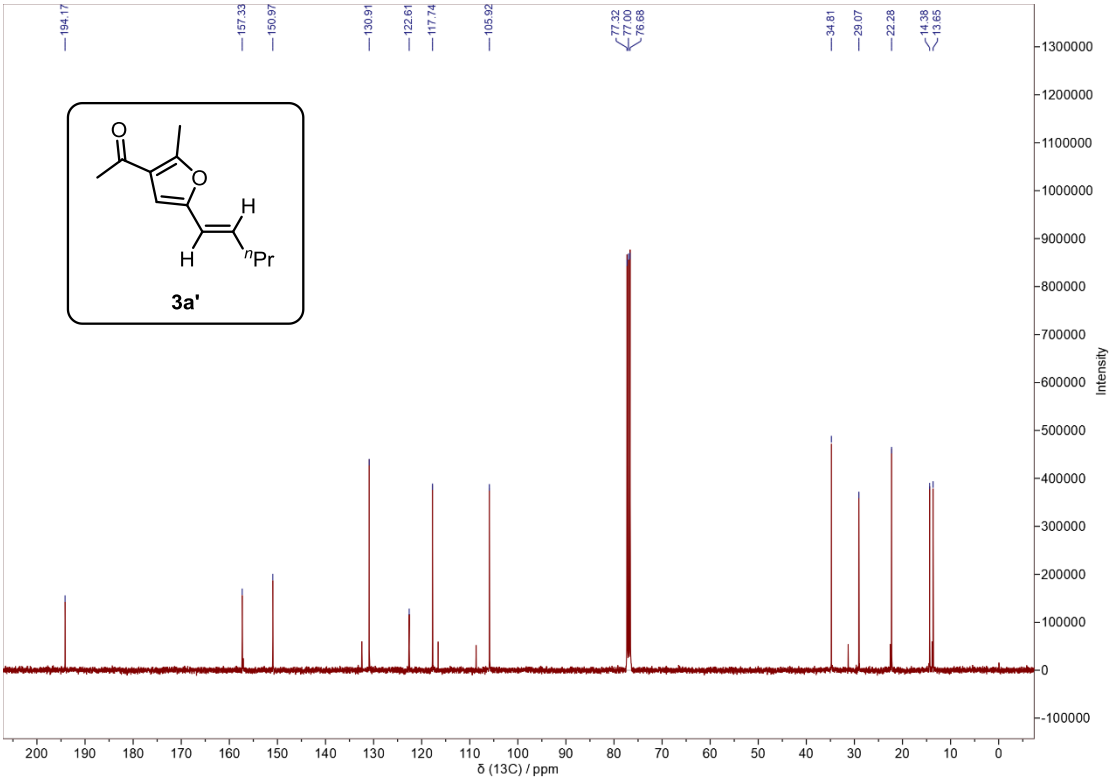

7.634
7.630
7.612
7.577
7.556
7.456
7.438
7.418
7.355
7.337
7.318
7.232
7.212
6.178
2.620
2.606
2.590
2.564
2.347
2.204
2.189
2.175
1.722
1.708
1.698
1.654
1.645
1.638
1.623
1.609
1.592
1.580
O
O
Ph
5e
2.08 2.09 2.08 1.04 1.98
0.95
2.05 3.00 3.14 2.11
6.44
Intensity
δ (1H) / ppm


194.33
157.15
152.85
144.04
140.77
139.49
139.33
130.20
128.74
127.21
126.97
126.78
123.24
122.31
109.74
77.32
77.00
76.68
32.89
32.53
29.14
28.60
28.46
26.62
14.56
O
O
Ph
5e
Intensity
δ (13C) / ppm


7.447
7.443
7.425
7.301
7.284
7.264
7.254
7.247
7.243
7.232
7.228
7.208
6.271
5.756
5.752
5.180
5.176
2.638
2.623
2.607
2.532
2.303
2.273
2.258
2.242
1.724
1.711
1.696
1.681
1.666
1.643
1.630
1.626
1.615
1.607
1.600
1.587
1.556
1.541
1.527
1.512
1.499
5f
1.90 3.32
0.96
0.97
0.95
2.05 3.00 2.94 1.95 2.14 2.44 2.04
δ (1H) / ppm
Intensity


194.28
156.97
151.85
146.28
143.76
139.46
128.30
127.58
126.11
122.69
122.36
115.32
109.42
77.32
77.00
76.68
33.11
31.91
29.05
28.56
28.40
26.60
14.56
5f
δ (13C) / ppm
Intensity


3a'
δ (1H) / ppm
Intensity


3a'
δ (13C) / ppm
Intensity