Supporting information

Solventless and Metal-free Regioselective Hydrofluorination of Functionalized Alkynes and Allenes: An Efficient Protocol for the Synthesis of *gem*-Difluorides

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Contents

Section 1. General experimental details

^{1}H NMR and ^{13}C NMR spectra were recorded at 400 MHz and 100 MHz respectively, using $CDCl_3$ as a solvent. The chemical shifts are reported in δ (ppm) values relative to $CHCl_3$ (δ 7.26 ppm for ^{1}H NMR), multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), h (hextet), m (multiplet) and br (broad). Coupling constants, *J*, are reported in Hertz.

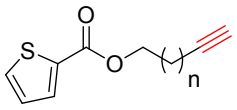
Solvents like DCM, Et_2O, Toluene, DMF were chemically dried using a commercial solvent purification system. Other solvents like DCE, dioxane, EtOAc and DMSO were dried with activated 4 Å molecular sieves overnight. Anhydrous hydrogen fluoride (HF) gas cylinder was purchased from Synquest Laboratories Inc.

All other reagents and solvents were employed without further purification. The products were purified using a CombiFlash system. TLC was developed on Merck silica gel 60 F254 aluminum sheets and $KMnO_4$ stain was used for TLC developing. $KMnO_4$ stain was prepared by mixing $KMnO_4$ (1.5 g), K_2CO_3 (10 g), and NaOH (10 wt%, 1.25 mL) in 200 mL water. All NMR solvents were purchased from Cambridge Isotope Laboratories, Inc.

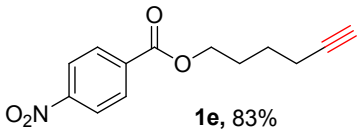
$KHSO_4$-13HF[1] and DMPU-12HF[2] were freshly prepared in our lab with the same method in the literature.

Section 2. Preparation of substrates

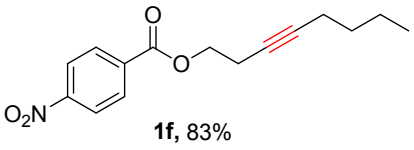
Alkyne substrates 1 (the identity of these substrates was confirmed by ^{1}H and ^{13}C NMR spectra. Substrates **1j**, [3] **1k,** [4] **1l**, [5] **1m**, [6] **1ai**, [7] **1aj**[8] were prepared using literature methods and the NMR spectra obtained were in accord with the literature. Substrate **1n** was purchased from Sigma-Aldrich).



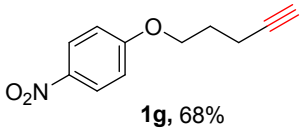
1a, n = 3, 81%
1b, n = 2, 75%
1c, n = 1, 71%
1d, n = 0, 73%



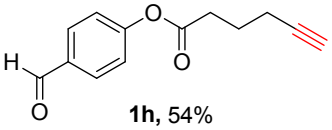
1e, 83%



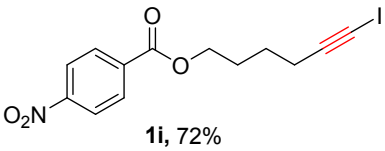
1f, 83%



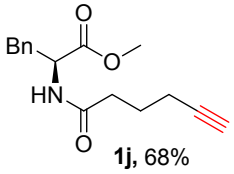
1g, 68%



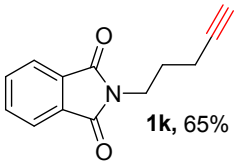
1h, 54%



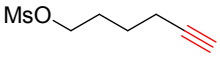
1i, 72%



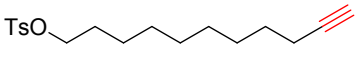
1j, 68%



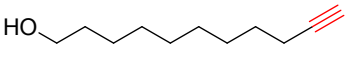
1k, 65%



1l, 83%

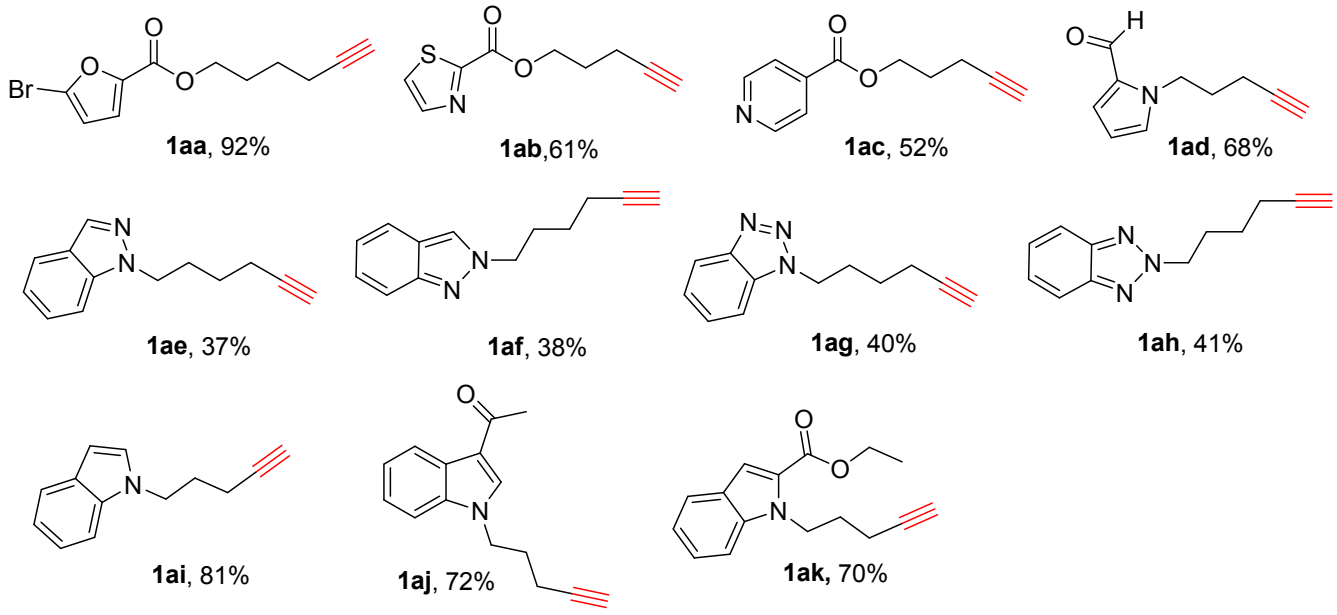


1m, 44%

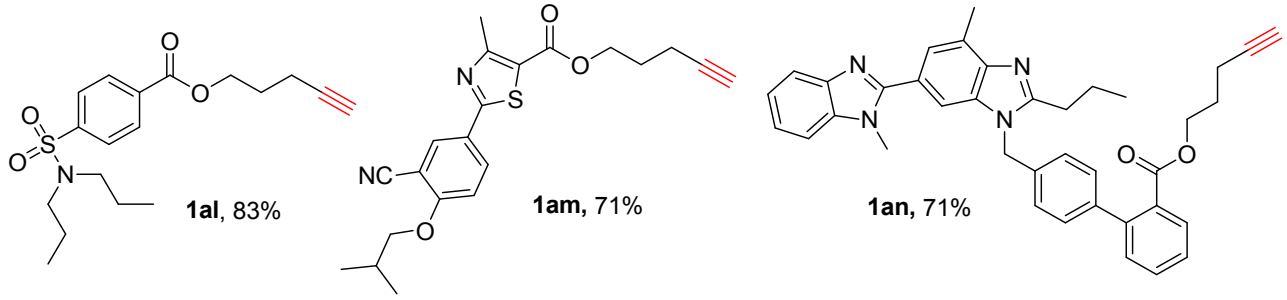


1n, 66%

Heterocyclic substrates:



Natural product and drug derivatives substrates:



Allene substrates [9]:

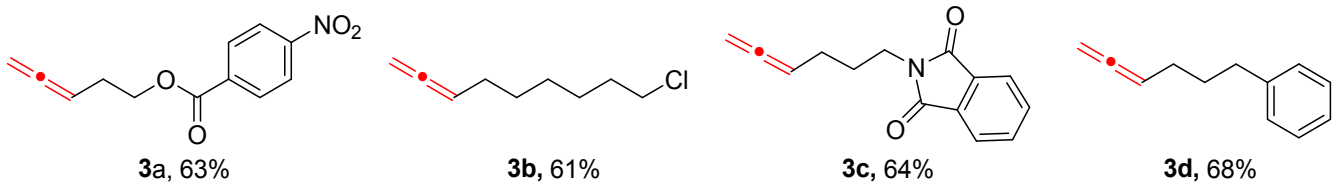
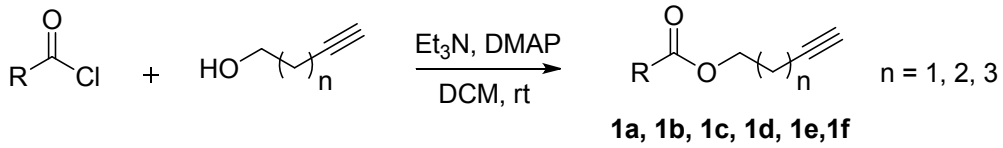


Figure S 1. Prepared alkyne and allene substrates.

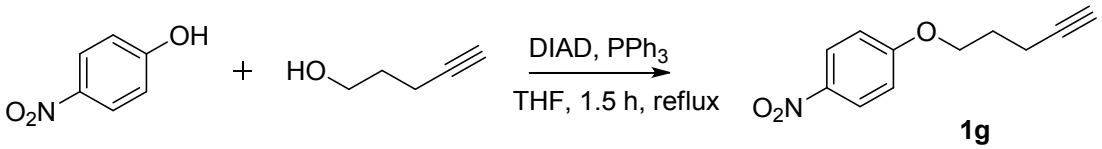
General synthetic procedure for the synthesis of esters 1a, 1b, 1c, 1d, 1e, 1f.



1a, 1b, 1c, 1d, 1e,1f

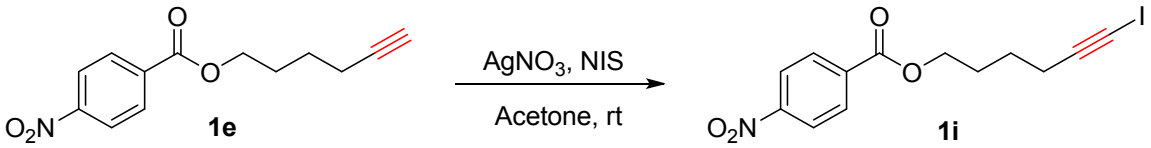
A 20-mL vial fitted with a stirring bar was charged with alcohol (2 mmol), Et_3N (2 equiv) and dry DCM (10 mL). The mixture was cooled down to 0 °C. Acyl chloride (1.2 equiv) and 5 mg DMAP were then added sequentially. The mixture was stirred overnight and then it was diluted with 50 mL DCM, washed with 1M aqueous HCl (2 x 20 mL), saturated $NaHCO_3$ (20 mL) and brine (20 mL), sequentially. The organic layer was then dried with Na_2SO_4 and concentrated. The residue was purified with column chromatography to afford the desired esters **1a** (yield: 93%), **1b** (yield: 75%), **1c** (yield: 71%,), **1d** (yield: 73%), **1e** (yield: 83%), **1f** (yield: 83%).

General synthetic procedure for the synthesis of ether 1g.



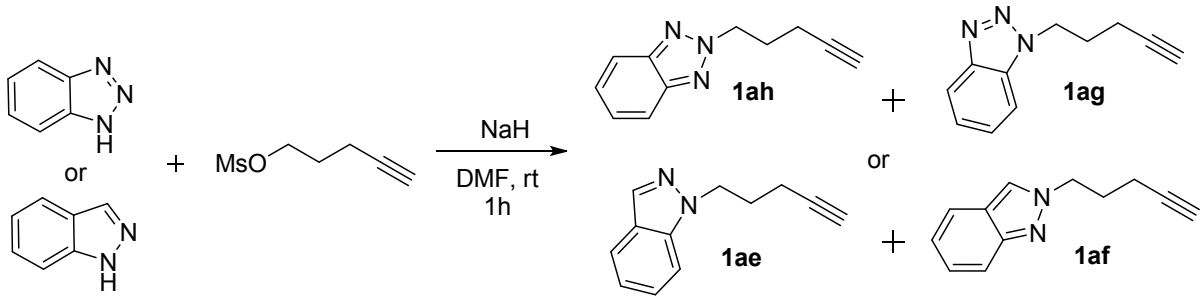
A solution of phenol (3 mmol), 3-methyl-3-buten-1-ol (3.6 mmol), triphenylphosphine (3.6 mmol), diisopropyl azodicarboxylate (3.6 mmol) in THF (27 mL) was heated for 1.5 hours at reflux. After concentrated in vacuo, the residue was purified by flash column chromatography to give the desired ethers **1g** (yield: 68%).

Synthetic procedure for the synthesis of ester 1i.



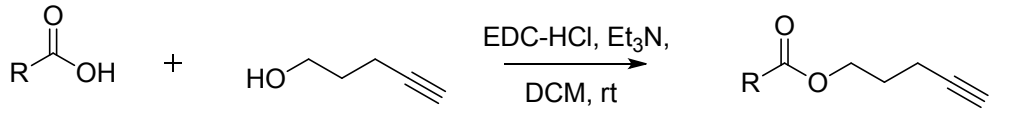
Alkyne **1e** (1.5 mmol) and silver nitrate (0.3 mmol) were stirred in acetone (10 mL) for 5 minutes and then N-iodosuccinimide (3.6 mmol) was added. The reaction was stirred at rt for 5 hours until complete consumption of the starting material according to TLC. It was then poured onto iced water. The aqueous layer was extracted with hexane (3 times), and the combined organic extracts were dried over MgSO4, filtered, and the solvent removed by evaporation under reduced pressure. The iodo-alkyne was isolated by flash column chromatography (yield: 72%).

General synthetic procedure for the synthesis of 1ae, 1af, 1ag, 1ah.



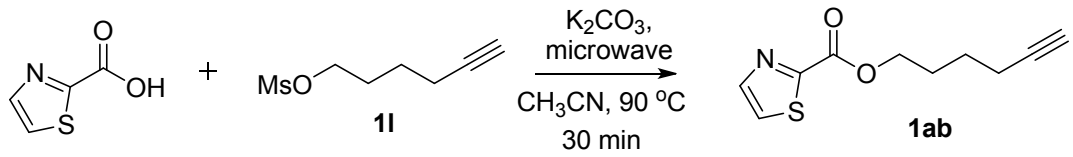
A 50-mL flask fitted with a stirring bar was charged with a solution of starting material (2 mmol) in DMF (10 mL). The mixture was cooled down to °C and NaH (1.5 equiv) was then added. The mesylate was also added and the mixture was stirred at rt for 1h. The reaction was quenched by NH_4Cl (1 M) and diluted with CH_2Cl_2 (50 mL). After being washed with 5% LiCl aqueous solution (2 x 20 mL), the aqueous layer was extracted with CH_2Cl_2 (20 mL). The combined organic extracts were washed with water (20 mL), dried and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography to afford **1ah** (yield: 41%), **1ag** (yield: 40%), **1ae** (yield: 37%), **1af** (yield: 38%).

General synthetic procedure for the preparation of esters 1h, 1aa, 1ac, 1al, 1am, 1an.



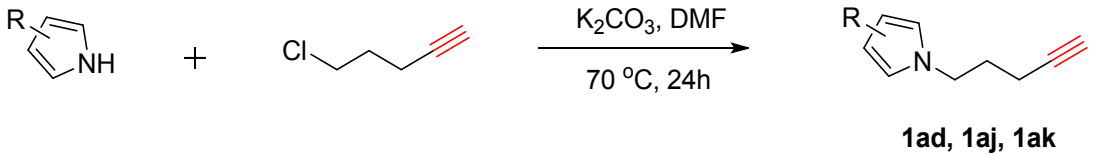
A 50-mL flask fitted with a stirring bar was charged with a solution of alcohol (2 mmol), EDCI (1.2 equiv), triethylamine (1.5 equiv), and DMAP (0.1 equiv) in dichloromethane (10 mL). Nicotinic acid (1 equiv) was then added at 0 °C, and the reaction mixture was stirred overnight at room temperature. After the reaction was complete, the resulting mixture was diluted with DCM (50 mL), washed by 1 N HCl (2 x 20 mL), 1 N aqueous $NaHCO_3$ (2 x 20 mL), and brine (1 x 20 mL). The organic layer was dried (Na_2SO_4) and evaporated in vacuo. The resulting residue was purified by column chromatography to afford the desired ester **1h** (yield: 54%), **1aa** (yield: 92%), **1ac** (yield: 52%), **1al** (yield: 83%), **1am** (yield: 71%), **1an** (yield: 71%).

General synthetic procedure for the preparation of ester 1ab.



A 10-mL microwave tube fitted with a stirring bar was charged with a solution of acid starting material (1.5 mmol), K_2CO_3 (1.5 equiv), mesylate (1 equiv) in CH_3CN (2 mL). The mixture was stirred under microwave conditions at 90 °C for 30 mins. The reaction was concentrated and purified directly by silica gel flash chromatography to afford **1ab** (yield: 61%).

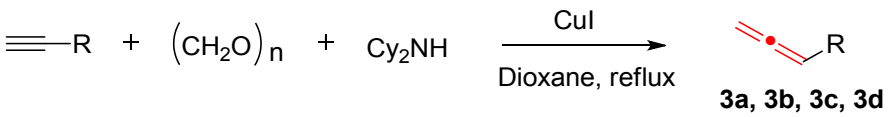
General synthetic procedure for the preparation of 1ad, 1aj, 1ak.



1ad, 1aj, 1ak

To a 10-mL flask were added 5-chloro-1-pentyne (2.4 mmol), indole or pyrrole derivatives (2.0 mmol), anhydrous potassium carbonate (2.0 mmol), and DMF (2 mL). The mixture was stirred for 24 h at 70 °C in an oil bath, cooled, diluted with water (40 mL), and extracted with CH_2Cl_2 (3*10 mL). The combined organic phase was washed with water (4*20 mL), dried over sodium sulfate, and concentrated in vacuo. The crude was purified with flash chromatography to afford **1ad** (yield: 68%), **1aj** (yield: 72%), **1ak** (yield: 70%).

General synthetic procedure for the preparation of allene 3a, 3b, 3c, 3d. [9]

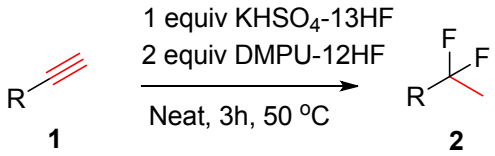


3a, 3b, 3c, 3d

A 50-mL flask fitted with a stirring bar was charged with $(CH_2O)_n$ (10 mmol), CuI (2 mmol), dioxane (20 mL), alkyne (4 mmol), and amine (7.2 mmol) sequentially. The flask was equipped with a reflux condenser under an argon atmosphere and the resulting mixture was stirred under reflux. When the reaction was complete as determined by TLC, it was cooled to rt. Water (25 mL) and ether (50 mL) were added and then the aqueous solution was separated and extracted with ether (3 × 25 mL). The organic layer was then washed with brine and dried over anhydrous Na_2SO_4. Evaporation and flash column chromatography on silica gel afforded the terminal allene **3a** (yield: 63%), **3b** (yield: 61%), **3c** (yield: 64%), **3d** (yield: 68%).

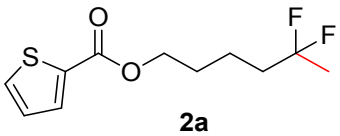
Section 3. General procedure for hydrofluorination of alkynes

3.1 Hydrofluorination of alkynes.



1 **2**

An 8-mL polytetrafluoroethylene (PTFE) vial fitted with a stirring bar was charged with alkyne starting material (0.2 mmol). DPMU-12HF (134 µL, 24 equiv based on HF) and $KHSO_4$-13HF (52 µL, 13 equiv based on HF) were then added in one portion at room temperature. The reaction was heated to 50 °C and stirred for 3 hours. The progress of the reaction was monitored by TLC (visualized by $KMnO_4$ stain). The product usually shows a slightly more polar spot than the starting material on TLC (R_f difference < 0.1 in most cases). The reaction was cooled down to 0 °C with an ice bath and calcium chloride powder was added to quench the reaction until no bubbling was observed. The crude was mixed with silica gel and concentrated. The resulting residue was purified with flash chromatography.



2a

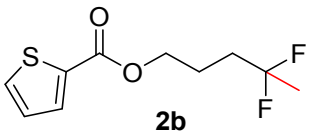
Colorless oil, isolated yield: 81%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.80 (dd, *J* = 3.7, 1.1 Hz, 1H), 7.55 (dd, *J* = 5.0, 1.1 Hz, 1H), 7.10 (dd, *J* = 5.0, 3.7 Hz, 1H), 4.31 (t, *J* = 6.4 Hz, 2H), 1.98 – 1.85 (m, 2H), 1.85 – 1.76 (m, 2H), 1.67 – 1.61 (m, 2H), 1.64 – 1.55 (t, *J* = 20 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 162.20, 133.80, 133.36, 132.32, 127.74, 124.07 (t, *J* = 237.7 Hz), 64.63, 37.48 (t, *J* = 25.6 Hz), 28.35, 23.28 (t, *J* = 28.0 Hz), 19.35 (t, *J* = 4.6 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.75 – -90.90 (m, 2F).

HRMS: (APCI$^+$) [M+H] cal. for $C_{11}H_{15}F_2O_2S$: 249.0761; found:249.0756.



2b

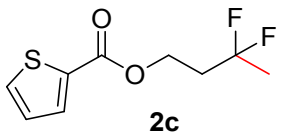
Colorless oil, isolated yield: 75%.

^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.85 – 7.79 (dd, *J* = 3.6, 1.2 Hz, 1H), 7.59 (dd, *J* = 4.8, 1.2 Hz, 1H), 7.14 (dd, *J* = 4.8, 3.6 Hz, 1H), 4.37 (t, J = 5.8 Hz, 2H), 2.10 – 1.93 (m, 4H), 1.66 (t, J = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 162.13, 133.64, 133.48, 132.45, 127.79, 123.84 (t, *J* = 238.0 Hz), 64.27, 34.61 (t, *J* = 26.0 Hz), 23.46 (t, *J* = 27.9 Hz), 22.25 (t, *J* = 4.5 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -89.72 – -93.27 (m, 2F).

HRMS: (APCI$^+$) [M+H] cal. for $C_{10}H_{13}F_2O_2S$: 235.0604; found:235.0599.



2c

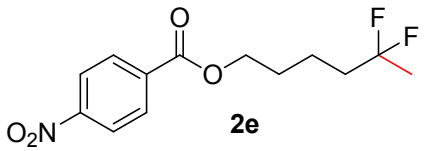
Colorless oil, isolated yield: 71%.

^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.82 (dd, *J* = 3.8, 1.2 Hz, 1H), 7.58 (dd, *J* = 5.0, 1.2 Hz, 1H), 7.12 (dd, *J* = 5.0, 3.8 Hz, 1H), 4.50 (t, *J* = 6.5 Hz, 2H), 2.36 (tt, *J* = 15.2, 6.5 Hz, 2H), 1.71 (t, J = 18.7 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 161.87, 133.68, 133.32, 132.65, 127.83, 122.90 (t, *J* = 238.1 Hz), 59.30 (t, *J* = 6.2 Hz), 37.11 (t, *J* = 26.2 Hz), 23.79 (t, *J* = 27.4 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -89.43 (qt, J = 18.7, 15.2 Hz, 2F).

HRMS: (APCI$^+$) [M+H] cal. for $C_9H_{11}F_2O_2S$: 221.0448; found:221.0442.



2e

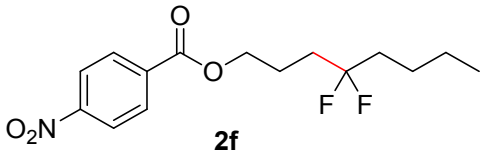
White solid, isolated yield: 83%.

^{1}H NMR (500 MHz, $CDCl_3$) δ = 8.30 (d, *J* = 8.9 Hz, 2H), 8.21 (d, *J* = 8.9 Hz, 2H), 4.40 (t, *J* = 6.5 Hz, 1H), 1.98 – 1.90 (m, 2H), 1.89 – 1.84 (m, 2H), 1.70 – 1.62 (m, 2H), 1.61 (t, J = 18.7 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 164.79, 150.65, 135.75, 130.79, 124.11 (t, *J* = 238.0 Hz), 123.68, 65.59, 37.57 (t, *J* = 25.6 Hz), 28.41, 23.51 (t, *J* = 28.0 Hz), 19.41 (t, *J* = 4.5 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.73 – -91.36 (m, 2F).

HRMS: (EI$^+$) [M] cal. for $C_{13}H_{15}F_2NO_4$: 287.0969; found:287.0966.



2f

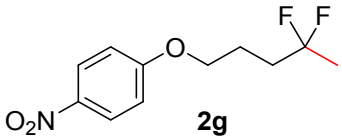
White solid, isolated yield: 81%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.22 – 8.13 (m, 2H), 8.12 – 8.03 (m, 2H), 4.30 (t, *J* = 6.1 Hz, 1H), 1.93 – 1.81(m, 4H), 1.80 – 1.67 (m, 2H), 1.38 – 1.31 (m, 2H), 1.29– 1.20 (m, 2H), 0.80 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 164.63, 150.61, 135.54, 130.72, 124.80 (t, *J* = 240.6 Hz), 123.62, 65.27, 36.38 (t, *J* = 25.1 Hz), 32.99 (t, *J* = 26.2 Hz), 29.74, 24.44 (t, *J* = 4.3 Hz), 22.50, 21.82 (t, *J* = 4.2 Hz), 13.88.

^{19}F NMR (376 MHz, $CDCl_3$) δ = -97.54 – -97.72 (m, 0.07F), -98.62 – -98.79 (m, 2F).

HRMS: (EI$^+$) [M] cal. for $C_{15}H_{19}F_2NO_4$: 315.1282; found:315.1281.



2g

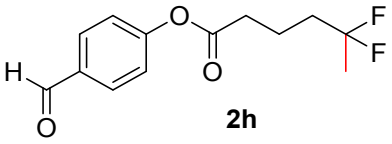
White solid, isolated yield: 46%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.99 – 7.92 (m, 2H), 6.74 – 6.65 (m, 2H), 3.87 (t, *J* = 5.7 Hz, 2H), 1.93 – 1.74 (m, 4H), 1.42 (t, *J* = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 163.79, 141.54, 125.91, 123.84 (t, *J* = 238.2 Hz), 114.35, 67.84, 34.43 (t, *J* = 25.9 Hz), 23.56 (t, *J* = 27.9 Hz), 22.42 (t, *J* = 4.3 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -91.67 – -92.00 (m, 2F).

HRMS: (EI$^+$) [M] cal. for $C_{11}H_{13}F_2NO_3$: 245.0863; found:245.0866.



2h

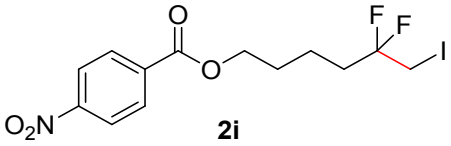
Colorless oil, isolated yield: 54%.

^{1}H NMR (500 MHz, $CDCl_3$) δ = 9.87 (s, 1H), 7.84 (d, *J* = 8.0 Hz, 2H), 7.00 (d, *J* = 8.0 Hz, 2H), 2.47 (t, *J* = 7.0 Hz, 2H), 2.02 – 1.76 (m, 4H), 1.63 (t, *J* = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 191.47, 178.41, 161.87, 132.57, 129.59, 123.86 (t, *J* = 237.8 Hz), 116.01, 36.95 (t, *J* = 25.7 Hz), 33.16, 23.29 (t, *J* = 27.8 Hz), 17.85 (t, *J* = 4.7 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.94 – -91.25 (m, 2F).

HRMS: (EI^+) [M] cal. for $C_{13}H_{14}F_2O_3$: 256.0911; found:256.0906.



2i

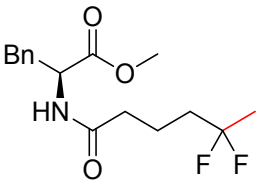
White solid, isolated yield: 72%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.41 (d, *J* = 8.7 Hz, 2H), 8.32 (d, *J* = 8.7 Hz, 2H), 4.52 (t, *J* = 6.4 Hz, 2H), 3.54 (t, *J* = 14.3 Hz, 2H), 2.42 – 2.18 (m, 2H), 2.07 – 1.92 (m, 2H), 1.89 – 1.73 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 164.50, 150.41, 135.41, 130.54, 123.43, 120.75 (t, *J* = 242.5 Hz), 65.13, 34.73 (t, *J* = 25.0 Hz), 27.96, 18.82 (t, *J* = 4.0 Hz), 3.55 (t, *J* = 31.8 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -94.76 – -94.96 (m, 2F).

HRMS: (EI^+) [M] cal. for $C_{13}H_{14}F_2INO_4$: 412.9936; found: 412.9919.



2j

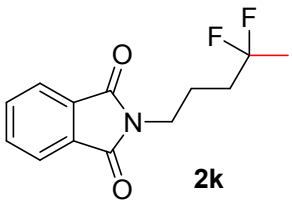
White solid, isolated yield: 68%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.30 – 7.18 (m, 3H), 7.09 – 7.03 (m, 2H), 5.92 (d, *J* = 7.7 Hz, 1H), 4.87 (dt, *J* = 7.8, 6.0 Hz, 1H), 3.71 (s, 3H), 3.13 (dd, *J* = 13.9, 5.7 Hz, 1H), 3.04 (dd, *J* = 13.9, 6.2 Hz, 1H), 2.19 (t, *J* = 6.7 Hz, 2H), 1.86 – 1.72 (m, 4H), 1.54 (t, *J* = 18.5 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 172.08, 171.65, 135.78, 129.17, 128.56, 127.13, 124.04 (t, *J* = 237.9 Hz), 52.92, 52.32, 37.86, 36.93 (t, *J* = 25.6 Hz), 35.45, 23.23 (t, *J* = 27.9 Hz), 18.67 (t, *J* = 4.7 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.49 – -90.81 (m, 2F).

HRMS: (APCI$^+$) [M+H] cal. for $C_{16}H_{22}F_2NO_3$: 314.1568; found:314.1566.



2k

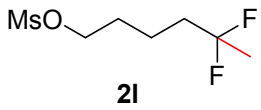
White solid, isolated yield: 65%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.78-7.76 (m, 2H), 7.66-7.64 (m, 2H), 3.66 (t, *J* = 6.6 Hz, 2H), 1.88-1.78 (m, 4H), 1.51 (t, *J* = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.20, 133.91, 131.92, 123.61 (t, *J* = 238.1 Hz), 123.18, 37.31, 35.30 (t, *J* = 26.1 Hz), 23.25 (t, *J* = 27.9 Hz), 21.99 (t, *J* = 4.3 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.89 – -91.12 (m, 2F).

HRMS: (EI$^+$) [M] cal. for $C_{13}H_{13}F_2NO_2$: 253.0914; found:253.0907.



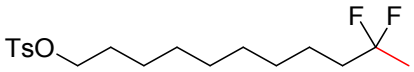
2l

Colorless oil, isolated yield: 83%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 4.26 (t, J = 6.3 Hz, 2H), 3.03 (s, 3H), 2.03 – 1.78 (m, 4H), 1.69 – 1.60 (m, 2H), 1.61 (t, *J* = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 123.91 (t, *J* = 237.8 Hz), 69.39, 37.33, 37.14(t, *J* = 25.7 Hz), 28.71, 23.30 (t, *J* = 27.9 Hz), 18.81 (t, *J* = 4.6 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -91.27 – -91.58 (m, 2F).



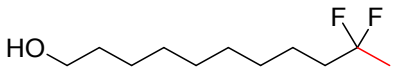
2m

White solid, isolated yield: 44%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.73 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.3 Hz, 2H), 3.96 (t, *J* = 6.5 Hz, 2H), 2.39 (s, 3H), 1.81 – 1.69 (m, 2H), 1.61 – 1.52 (m, 2H), 1.51 (t, *J* = 18.4 Hz, 3H), 1.38 (dt, *J* = 12.0, 7.6 Hz, 2H), 1.26 – 1.10 (m, 10H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 144.58, 133.20, 129.75, 127.83, 124.38 (t, *J* = 237.4 Hz), 70.61, 37.88 (t, *J* = 25.3 Hz), 29.19, 29.16, 29.14, 28.81, 28.76, 25.26, 23.18 (t, *J* = 28.2 Hz), 22.64 (t, *J* = 4.5 Hz), 21.59.

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.36 – -90.59 (m, 2F).



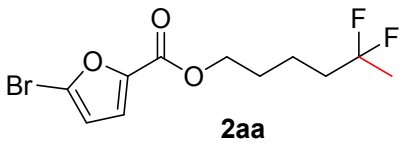
2n

White needle crystal, isolated yield: 66%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = δ 3.80 (t, *J* = 6.6 Hz, 2H), 2.07 – 1.89 (m, 2H), 1.77 – 1.70 (m, 2H), 1.73 (t, *J* = 18.4 Hz, 3H), 1.66 – 1.55 (m, 2H), 1.56 – 1.40 (m, 10H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 124.43 (t, *J* = 237.4 Hz), 63.04, 37.93 (t, *J* = 25.3 Hz), 32.75, 29.38, 29.34, 29.29, 25.68, 23.18 (t, *J* = 28.1 Hz), 22.70 (t, *J* = 4.4 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.22 – -90.55 (m, 2F).



2aa

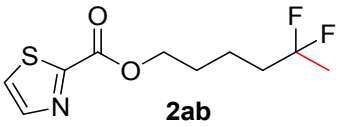
Colorless oil, isolated yield: 92%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.01 (d, *J* = 3.5 Hz, 1H), 6.35 (d, *J* = 3.5 Hz, 1H), 4.21 (t, *J* = 6.5 Hz, 2H), 1.87 – 1.73 (m, 2H), 1.73 – 1.65 (m, 2H), 1.55 – 1.46 (m, 2H), 1.49 (t, *J* = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 157.65, 146.30, 127.54, 124.07 (t, *J* = 237.7 Hz), 120.06, 113.94, 77.39, 77.07, 76.75, 64.74, 37.49 (t, *J* = 25.6 Hz), 28.34, 23.34 (t, *J* = 28.0 Hz), 19.29 (t, *J* = 4.6 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.67 – -90.90 (m, 2F).

HRMS: (APCI^{+}) [M+H] cal. for $C_{11}H_{14}BrF_2O_3$: 311.0094; found:311.0090.



2ab

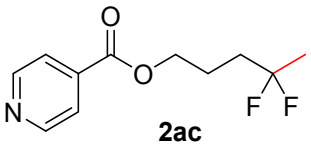
Colorless oil, isolated yield: 78%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.03 (d, *J* = 3.0 Hz, 1H), 7.64 (d, *J* = 3.1 Hz, 1H), 4.43 (t, *J* = 6.6 Hz, 2H), 1.97 – 1.83 (m, 4H), 1.68 – 1.60 (m, 2H), 1.59 (t, *J* = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 159.99, 158.27, 145.00, 125.27, 124.00 (t, *J* = 238.0 Hz), 66.03, 37.44 (t, *J* = 25.6 Hz), 28.23, 23.30 (t, *J* = 28.0 Hz), 19.26 (t, *J* = 4.6 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.78 – -91.07 (m, 2F).

HRMS: (APCI^{+}) [M+H] cal. for $C_{10}H_{14}F_2NO_2S$: 250.0713; found:250.0710.



2ac

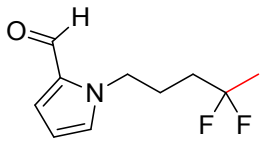
Colorless oil, isolated yield: 71%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.63 (d, *J* = 4.8 Hz, 2H), 7.68 (d, *J* = 4.8 Hz, 2H), 4.24 (t, *J* = 5.7 Hz, 2H), 1.91 – 1.78 (m, 4H), 1.48 (t, *J* = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 164.89, 150.53, 137.17, 123.58(d, *J* = 237.9 Hz), 122.69, 64.85, 34.46 (t, *J* = 26.1 Hz), 23.39 (t, *J* = 27.9 Hz), 21.96 (t, *J* = 4.4 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -91.52 – -91.82 (m, 2F).

HRMS: (APCI$^+$) [M+H] cal. for $C_{11}H_{14}F_2NO_2$: 239.0993; found:230.0989.



2ad

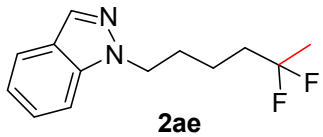
Dark red oil, isolated yield: 53%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 9.38 (s, 1H), 6.87 – 6.73 (m, 2H), 6.09 (dd, *J* = 3.8, 2.6 Hz, 1H), 4.22 (t, *J* = 7.0 Hz, 2H), 1.90 – 1.75 (m, 2H), 1.75 – 1.60 (m, 2H), 1.43 (t, *J* = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 179.43, 131.39, 131.34, 125.17, 124.02 (d, *J* = 238.1 Hz), 109.85, 48.36, 34.72 (t, *J* = 25.9 Hz), 24.77 (t, *J* = 4.2 Hz), 23.44 (t, *J* = 27.9 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.67 – -90.90 (m, 2F).

HRMS: (APCI$^+$) [M+H] cal. for $C_{10}H_{14}F_2NO$: 202.1043; found:202.1039.



2ae

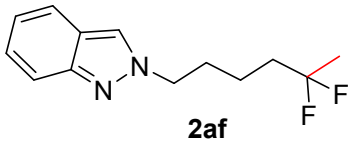
Colorless oil, isolated yield: 75%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.92 (d, *J* = 0.6 Hz, 1H), 7.66 (dt, *J* = 8.1, 0.9 Hz, 1H), 7.39 – 7.24 (m, 2H), 7.07 (ddd, J = 7.9, 6.1, 1.7 Hz, 1H), 4.37 – 4.28 (m, 2H), 1.96 – 1.86 (m, 2H), 1.85 – 1.71 (m, 2H), 1.49 – 1.39 (m, 2H), 1.46 (t, *J* = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 139.38, 132.91, 126.24, 124.05 (t, *J* = 6.4 Hz), 124.02, 121.21, 120.50, 108.87, 48.47, 37.43 (t, *J* = 25.6 Hz), 29.74, 29.45, 23.24 (t, *J* = 28.0 Hz), 20.17 (t, *J* = 4.6 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.53 – -90.88 (m, 2F).

HRMS: (APCI$^+$) [M+H] cal. for $C_{13}H_{17}F_2N_2$: 239.1360; found:239.1356.



2af

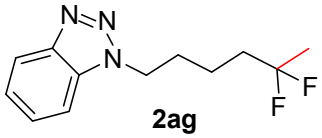
Off-white solid, isolated yield: 76%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.93 (s, 1H), 7.74 (d, *J* = 8.7 Hz, 1H), 7.68 (d, *J* = 8.4 Hz, 1H), 7.42 – 7.21 (m, 1H), 7.11 (dd, *J* = 7.8, 7.2 Hz, 1H), 4.45 (t, *J* = 7.0 Hz, 2H), 2.17 – 2.02 (m, 2H), 1.96 – 1.83 (m, 2H), 1.58 – 1.51 (m, 2H), 1.58 (t, *J* = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.80, 125.76, 123.85 (t, *J* = 237.0 Hz), 122.55, 121.63, 121.54, 119.96, 117.27, 53.27, 37.22 (t, *J* = 25.7 Hz), 30.12, 23.22 (t, *J* = 27.9 Hz), 19.82 (t, *J* = 4.5 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.96 – -91.10 (m, 2F).

HRMS: (APCI^{+}) [M+H] cal. for $C_{13}H_{17}F_2N_2$: 239.1360; found:239.1357.



2ag

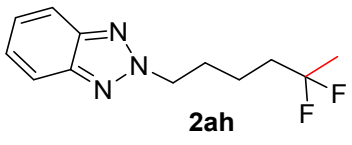
White solid, isolated yield: 80%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.10 (d, *J* = 8.4 Hz, 1H), 7.61 – 7.47 (m, 2H), 7.41 (t, *J* = 7.4 Hz, 1H), 4.69 (t, *J* = 7.0 Hz, 2H), 2.16 – 2.04 (m, 3H), 1.99 – 1.84 (m, 3H), 1.65 – 1.50 (m, 2H).1.58 (t, *J* = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 146.00, 132.89, 127.34, 123.95 (t, *J* = 237.0 Hz), 123.93, 120.09, 109.21, 47.80, 37.18 (t, *J* = 25.7 Hz), 29.23, 23.32 (t, *J* = 27.9 Hz), 20.01 (t, *J* = 4.5 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.58 – -90.93 (m, 2F).

HRMS: (APCI^{+}) [M+H] cal. for $C_{13}H_{16}F_2N_3$: 240.1312; found:240.1309.



2ah

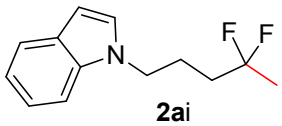
Light yellow oil, isolated yield: 82%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.96 (dd, J = 6.5, 3.0 Hz, 2H), 7.48 (dd, J = 6.5, 3.0 Hz, 2H), 4.84 (t, J = 7.0 Hz, 2H), 2.33 – 2.18 (m, 2H), 2.10 – 1.91 (m, 2H), 1.73 – 1.56 (m, 2H), 1.65 (t, *J* = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 144.39, 126.38, 124.03 (t, *J* = 237.9 Hz), 118.04, 56.21, 37.31 (t, *J* = 25.7 Hz), 29.67, 23.37 (t, *J* = 27.9 Hz), 19.95 (t, *J* = 4.4 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.60 – -90.83 (m, 2F).

HRMS: (APCI^{+}) [M+H] cal. for $C_{13}H_{16}F_2N_3$: 240.1312; found:240.1309.



2ai

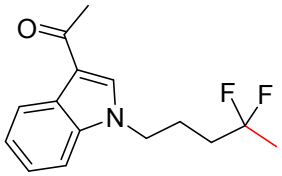
Colorless oil, isolated yield: 27%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.73 (d, *J* = 7.9 Hz, 1H), 7.43 (d, *J* = 8.2 Hz, 1H), 7.31 (t, *J* = 7.6 Hz, 1H), 7.20 – 7.18 (m, 2H), 6.60 (d, *J* = 3.1 Hz, 1H), 4.29 (t, *J* = 7.0 Hz, 2H), 2.22 – 2.08 (m, 2H), 1.98 – 1.82 (m, 2H), 1.65 (t, *J* = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 135.84, 127.53, 126.20 (t, *J* = 238.5 Hz), 121.52, 121.00, 119.33, 109.14, 101.35, 45.62, 35.06 (t, *J* = 26.1 Hz), 23.51 (t, *J* = 27.5 Hz), 23.48.

^{19}F NMR (376 MHz, $CDCl_3$) δ = -91.11 – -91.37 (m, 2F).

HRMS: (APCI^{+}) [M+H] cal. for $C_{13}H_{16}F_2N$: 224.1251; found:224.1247.



2aj

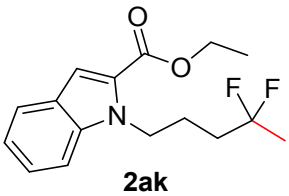
White solid, isolated yield: 60%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.50 – 8.44 (m, 1H), 7.81 (s, 1H), 7.48 – 7.35 (m, 3H), 4.30 (t, *J* = 7.2 Hz, 1H), 2.60 (s, 3H), 2.24 – 2.13 (m, 2H), 2.02 – 1.87 (m, 2H), 1.67 (t, J = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 192.96, 136.63, 134.48, 126.35, 123.66 (t, *J* = 238.4 Hz), 123.38, 122.70, 122.59, 117.24, 109.63, 46.30, 34.82 (t, *J* = 25.9 Hz), 27.64, 23.64 (t, *J* = 27.8 Hz), 23.11 (t, *J* = 3.7 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -91.18 – -91.45 (m, 2F).

HRMS: (APCI$^+$) [M+H] cal. for $C_{15}H_{18}F_2NO$: 266.1356; found:266.1354.



2ak

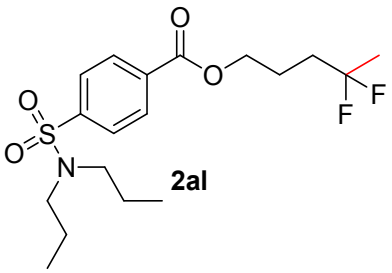
Colorless oil, isolated yield: 44%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.67 (d, *J* = 8.0 Hz, 1H), 7.39 (d, *J* = 8.4 Hz, 1H), 7.35 (d, *J* = 7.0 Hz, 1H), 7.32 (s, 1H), 7.15 (t, *J* = 7.2 Hz, 1H), 4.61 (t, *J* = 7.2 Hz, 2H), 4.37 (q, *J* = 7.1 Hz, 2H), 2.05 – 1.98 (m, 2H), 1.96 – 1.83 (m, 2H), 1.57 (t, *J* = 20.4, 18.4 Hz, 3H), 1.41 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 161.98, 138.95, 127.30, 125.96, 125.08, 123.99 (t, *J* = 237.0 Hz), 122.70, 120.62, 110.72, 110.23, 60.57, 43.96, 35.12 (t, *J* = 25.9 Hz), 23.88 (t, *J* = 3.8 Hz), 23.36 (t, *J* = 27.9 Hz), 14.34.

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.53 – -90.76 (m, 2F).

HRMS: (APCI$^+$) [M+H] cal. for $C_{16}H_{20}F_2NO_2$: 296.1462; found:296.1459.



2al

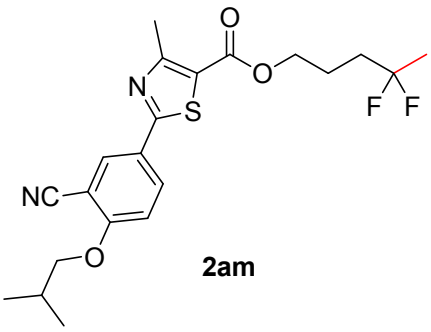
Colorless oil, isolated yield: 83%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.25 – 8.10 (m, 2H), 7.99 – 7.83 (m, 2H), 4.45 (t, *J* = 6.0 Hz, 2H), 3.17 – 3.12 (m, 4H), 2.18 – 1.93 (m, 4H), 1.69 (t, *J* = 18.4 Hz, 2H), 1.65– 1.55 (m, 4H), 0.91 (t, *J* = 7.4 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.15, 144.37, 133.40, 130.21, 127.05, 123.76 (t, *J* = 238.1 Hz), 64.80, 49.94, 34.64 (t, *J* = 26.0 Hz), 23.52 (t, *J* = 27.9 Hz), 22.17 (t, *J* = 4.4 Hz), 21.95, 11.17.

^{19}F NMR (376 MHz, $CDCl_3$) δ = -91.26 – -91.53 (m, 2F).

HRMS: (ESI^+) [M+Na] cal. for $C_{18}H_{27}F_2NO_4SNa$: 414.1527; found:414.1522.



2am

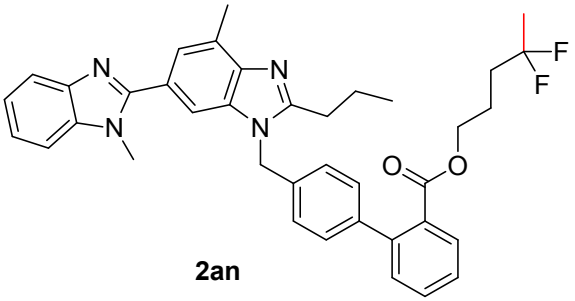
White solid, isolated yield: 71%.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.16 (d, *J* = 2.3 Hz, 1H), 8.08 (dd, *J* = 8.9, 2.3 Hz, 1H), 7.00 (d, *J* = 8.9 Hz, 1H), 4.33 (t, *J* = 6.0 Hz, 2H), 3.89 (d, *J* = 6.5 Hz, 2H), 2.75 (s, 3H), 2.20 (dt, *J* = 13.3, 6.7 Hz, 1H), 2.08 – 1.89 (m, 4H), 1.64 (t, *J* = 18.4 Hz, 3H), 1.08 (d, *J* = 6.7 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 167.29, 162.49, 161.85, 161.37, 132.52, 132.06, 125.89, 123.72 (t, *J* = 237.0 Hz), 121.44, 115.34, 112.58, 102.94, 75.67, 64.45, 34.58 (t, *J* = 26.1 Hz), 28.12, 23.47 (t, *J* = 27.9 Hz), 22.15 (t, *J* = 4.4 Hz), 19.02, 17.48.

^{19}F NMR (376 MHz, $CDCl_3$) δ = -91.26 – -91.53 (m, 2F).

HRMS: (ESI^+) [M+Na] cal. for $C_{21}H_{24}F_2N_2NaO_3S$: 445.1373; found:445.1370.



2an

Colorless syrup, isolated yield: 71%.

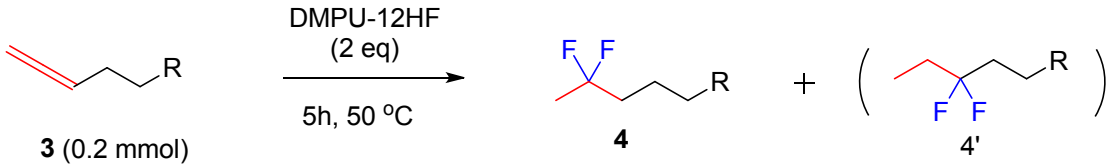
^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.78 (dd, *J* = 10.7, 5.8 Hz, 2H), 7.51 – 7.32 (m, 5H), 7.29 – 7.21 (m, 5H), 7.08 (d, J = 8.1 Hz, 2H), 5.42 (s, 2H), 4.01 (t, *J* = 5.5 Hz, 2H), 3.78 (s, 3H), 2.94 – 2.88 (m, 2H), 2.75 (s, 3H), 1.86 (dt, *J* = 13.2, 6.6 Hz, 2H), 1.56 – 1.51 (m, 4H), 1.45 (t, *J* = 18.4 Hz, 3H), 1.04 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.32, 156.47, 154.66, 143.16, 142.79, 141.61, 141.27, 136.59, 134.97, 131.37, 130.68, 130.62, 129.93, 129.45, 128.94, 127.39, 125.95, 123.84, 123.80, 123.78 (t, *J* = 237.5 Hz), 122.46, 122.28, 119.49, 109.45, 108.85, 64.12, 46.96, 34.30 (t, *J* = 25.8 Hz), 31.74, 29.75, 23.15 (t, *J* = 27.9 Hz), 21.88, 21.76 (t, *J* = 4.5 Hz), 16.85, 14.02.

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.53 – -90.85 (m, 2F).

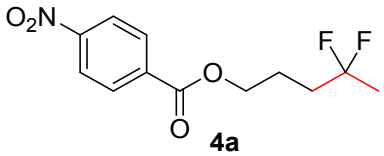
HRMS: (ESI^+) [M+H] cal. for $C_{38}H_{39}F_2N_4O_2$: 621.3041; found:621.3037.

3.2 Hydrofluorination of allenes.



3 (0.2 mmol) **4** 4'

An 8-mL polytetrafluoroethylene (PTFE) vial fitted with a stirring bar was charged alkyne starting material (0.2 mmol). DPMU-12HF (134 µL, 24 equiv based on HF) was then added in one portion at room temperature. The reaction was heated to 50 °C and stirred for 5 hours. The progress of reaction was monitored by TLC (visualized by $KMnO_4$ stain). The product usually shows a slightly more polar spot than the starting material on TLC (R_f difference < 0.1 in most cases). The reaction was cooled down to 0 °C with ice bath and calcium chloride powder was added to quench the reaction until no bubbling was observed. The crude was mixed with silica gel and concentrated. The result residue was purified with flash chromatography.



4a

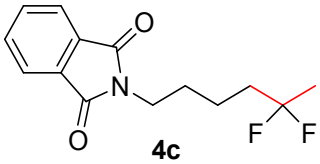
Yellow solid, isolated yield: 66% (**4a** : **4a'** = 66 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.28 (d, J = 8.9 Hz, 2H), 8.19 (d, J = 8.8 Hz, 2H), 4.41 (t, J = 5.7 Hz, 2H), 2.05 – 1.92 (m, 4H), 1.63 (t, J = 18.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 164.14, 150.14, 135.07, 130.26, 123.34 (t, *J* = 238.0 Hz), 123.13, 64.68, 34.13 (t, *J* = 26.1 Hz), 23.04 (t, *J* = 27.8 Hz), 21.68 (t, *J* = 4.4 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -91.26 – -91.73 (m, 2F), -99.80 – -99.97 (m, 0.03F).

HRMS: (EI^+) [M] cal. for $C_{12}H_{13}F_2NO_4$: 273.0813; found: 273.0808.



4c

White solid, isolated yield: 61% (**4c** : **4c'** = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.86 – 7.80 (m, 2H), 7.73 – 7.67 (m, 2H), 3.75 – 3.65 (m, 2H), 1.97 – 1.79 (m, 2H), 1.78 – 1.66 (m, 2H), 1.62 – 1.47 (m, 4H), 0.98 (t, J = 7.5 Hz, 0.45H).

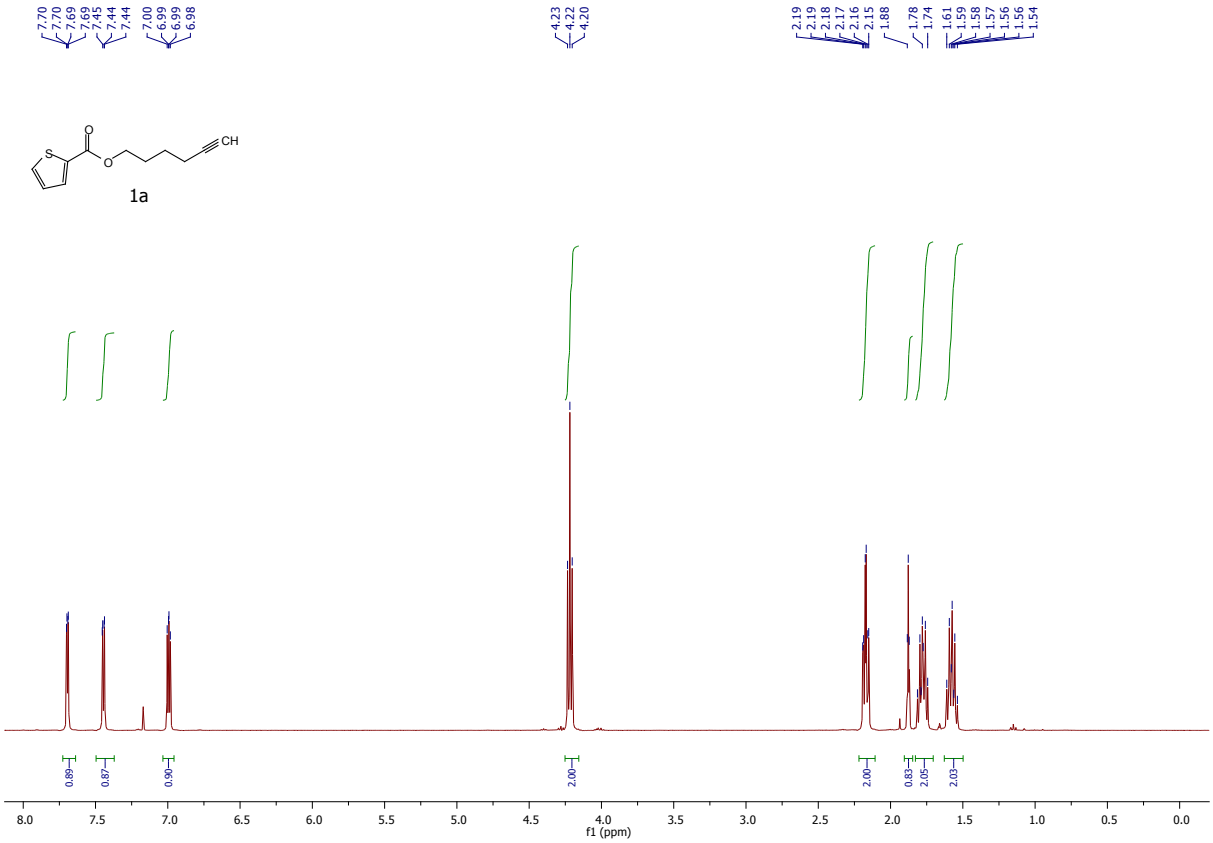
^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.34, 133.91, 132.05, 124.02 (t, *J* = 237.0 Hz), 123.20, 37.52, 37.36 (t, *J* = 26.2 Hz), 34.84, 33.39 (t, *J* = 26.2 Hz), 32.45, 29.60 (t, *J* = 26.2 Hz), 28.21, 23.51, 23.23, 22.96, 23.23 (t, J = 27.9 Hz), 21.69 (t, *J* = 4.0 Hz), 20.00 (t, *J* = 4.6 Hz, 1H).

^{19}F NMR (376 MHz, $CDCl_3$) δ = -90.58 – -90.89 (m, 2F), -100.39 – -100.60 (m, 0.36F).

HRMS: (EI^+) [M] cal. for $C_{14}H_{15}F_2NO_2$: 267.1071; found: 267.1067.

Section 4. Spectral data of starting materials and products

4.1 Starting materials NMR spectra.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7.69
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7.44
7.44
7.00
6.99
6.99
6.98
4.23
4.22
4.20
2.19
2.19
2.18
2.17
2.16
2.15
1.88
1.78
1.74
1.61
1.59
1.58
1.57
1.56
1.56
1.54
O
S
O
CH
1a
0.89
0.87
0.90
2.00
2.00
0.83
2.05
2.03
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.5
3.0
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1.0
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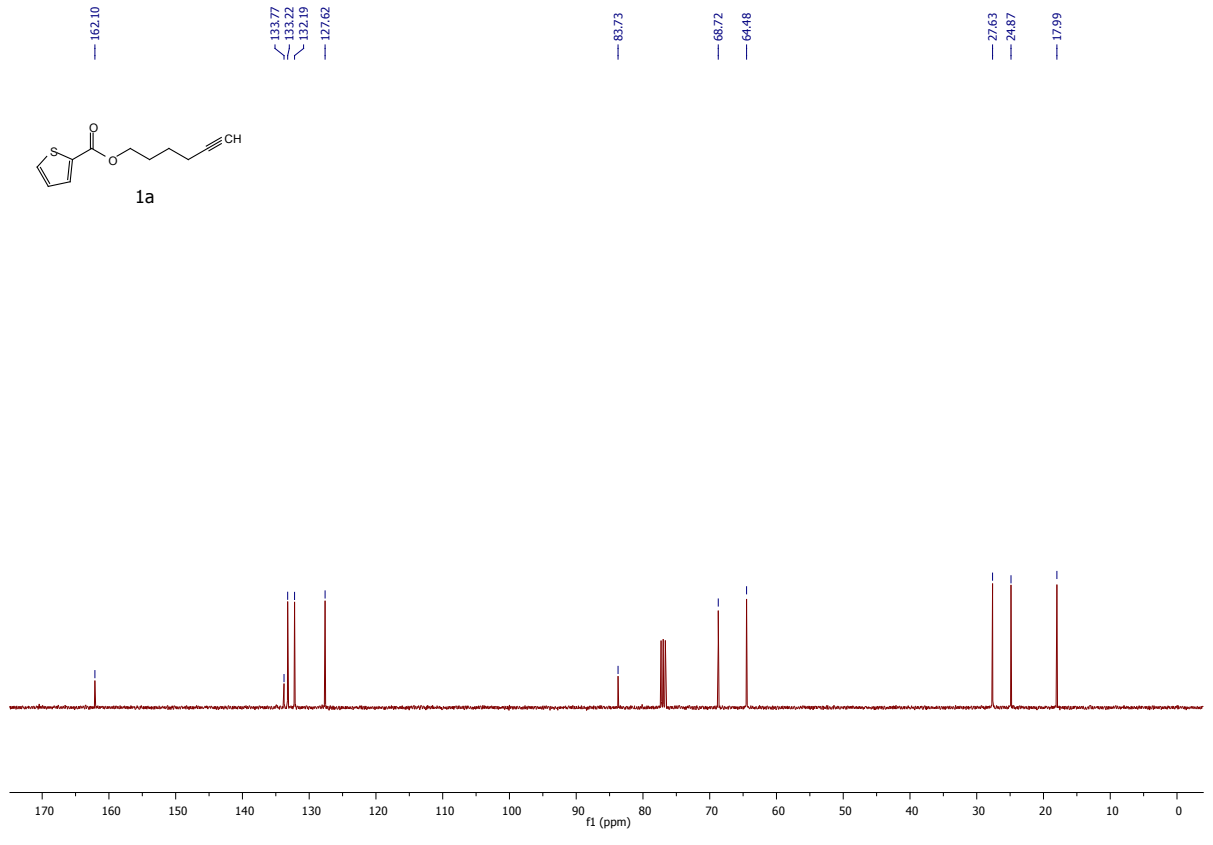
0.0
f1 (ppm)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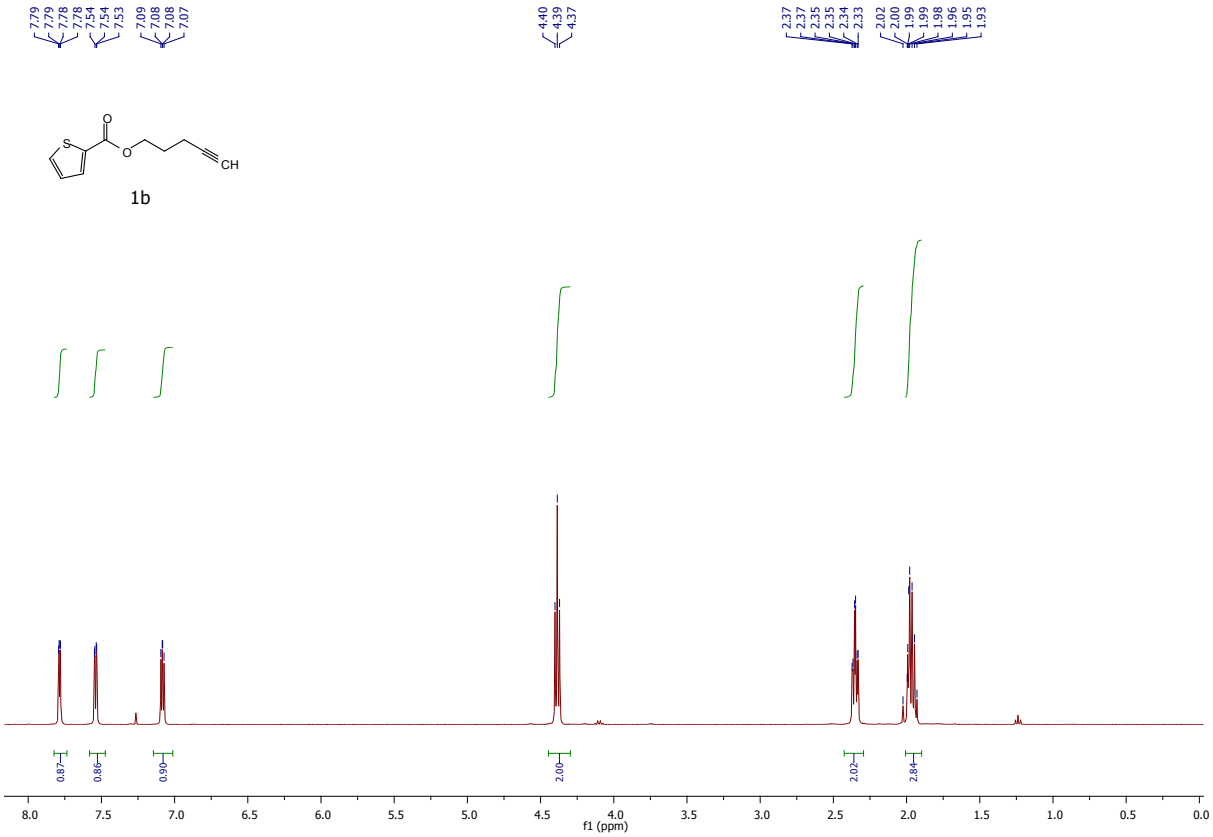
133.22
132.19
127.62
83.73
68.72
64.48
27.63
24.87
17.99
O
S
O
CH
1a
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.09
7.08
7.08
7.07
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4.39
4.37
2.37
2.37
2.35
2.35
2.34
2.33
2.02
2.00
1.99
1.99
1.98
1.96
1.95
1.93
O
S
O
CH
1b
0.87
0.86
0.90
2.00
2.02
2.84
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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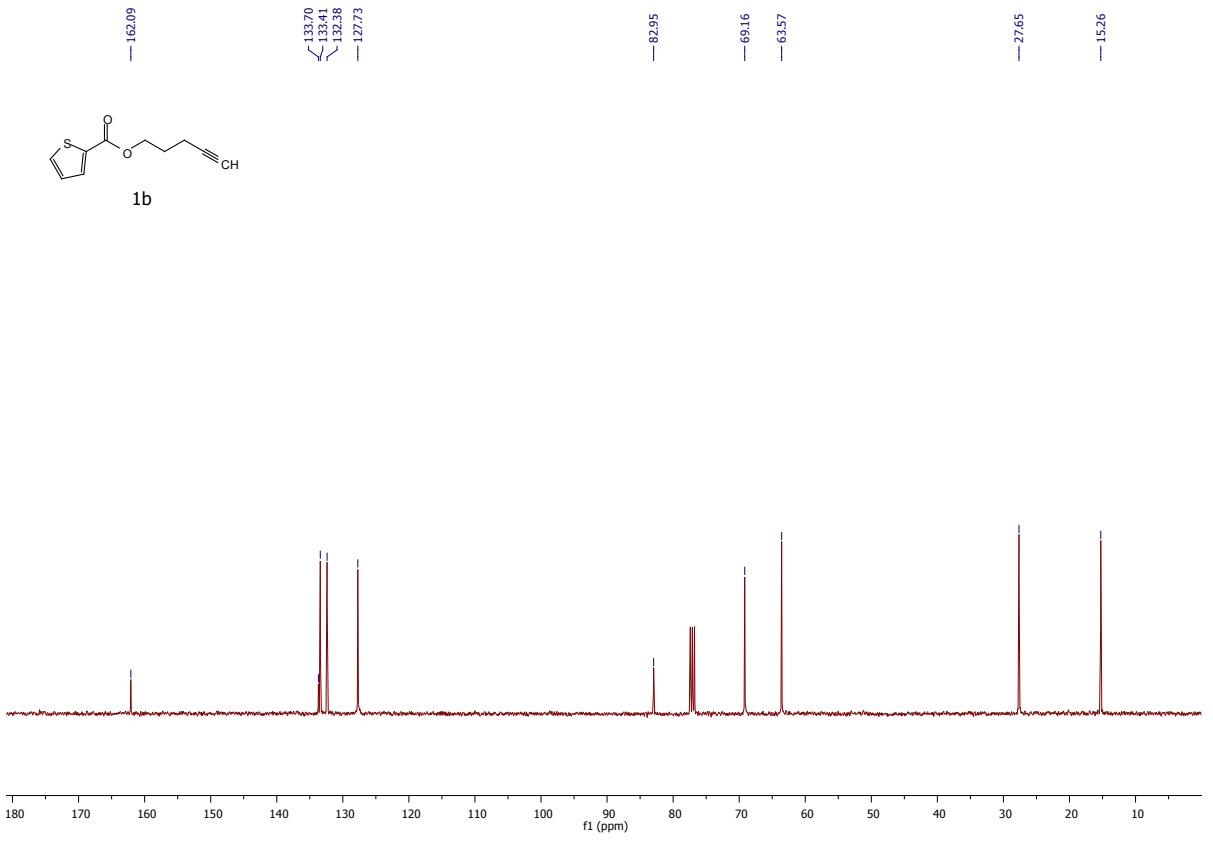
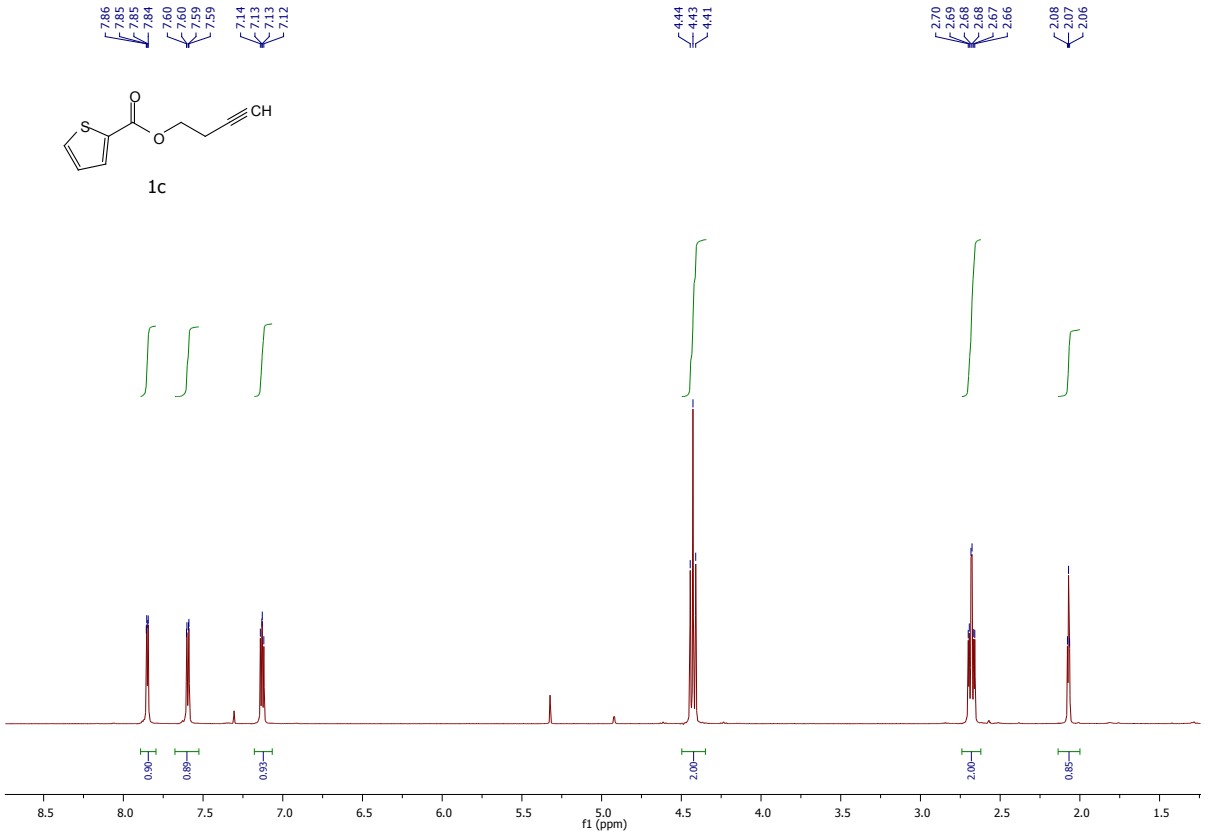
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


162.09
133.70
133.41
132.38
127.73
82.95
69.16
63.57
27.65
15.26
O
S
O
CH
1b
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.85
7.84
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7.59
7.59
7.14
7.13
7.13
7.12
4.44
4.43
4.41
2.70
2.69
2.68
2.68
2.67
2.66
2.08
2.07
2.06
O
S
O
CH
1c
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0.89
0.93
2.00
2.00
0.85
8.5
8.0
7.5
7.0
6.5
6.0
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5.0
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4.0
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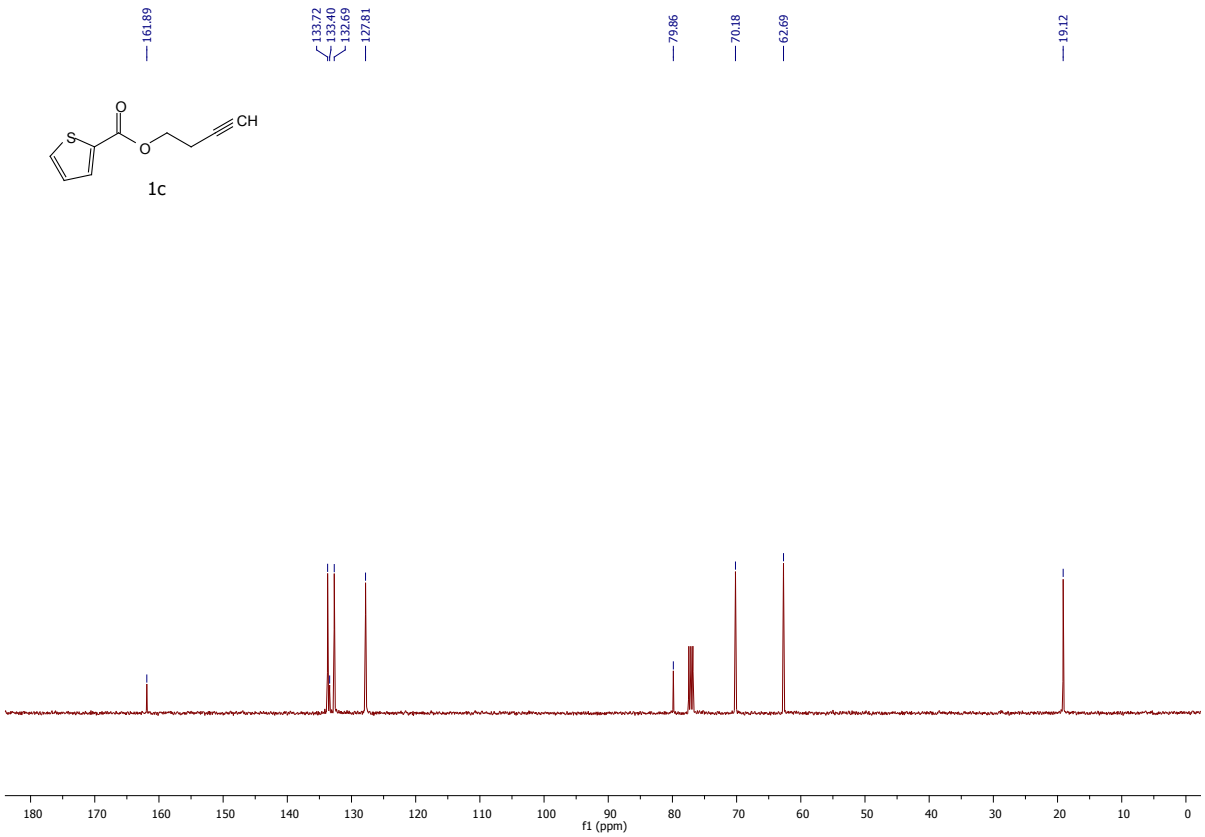
3.0
2.5
2.0
1.5
f1 (ppm)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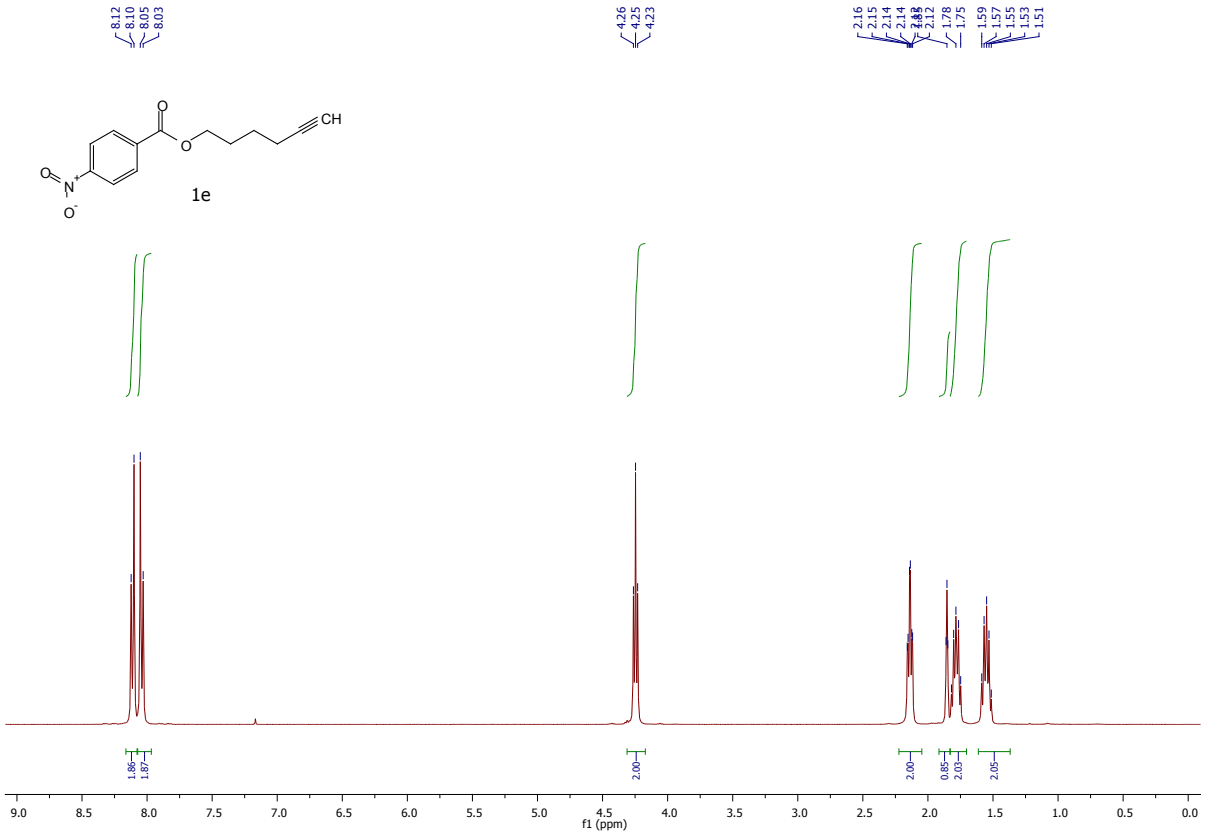
133.72
133.40
132.69
127.81
79.86
70.18
62.69
19.12
O
S
O
CH
1c
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.05
8.03
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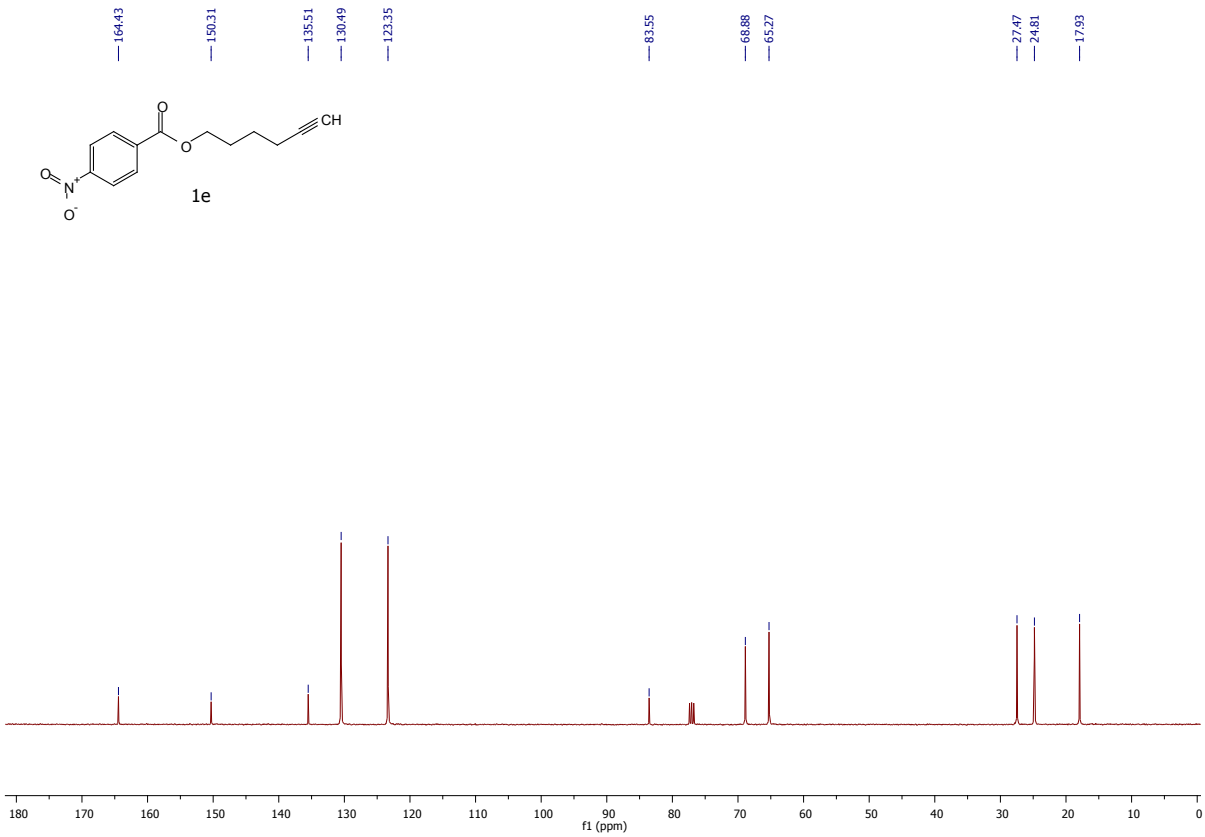
4.25
4.23
2.16
2.15
2.14
2.12
1.78
1.75
1.59
1.57
1.55
1.53
1.51
CH
1e
1.86
1.87
2.00
2.00
0.85
2.03
2.05
f1 (ppm)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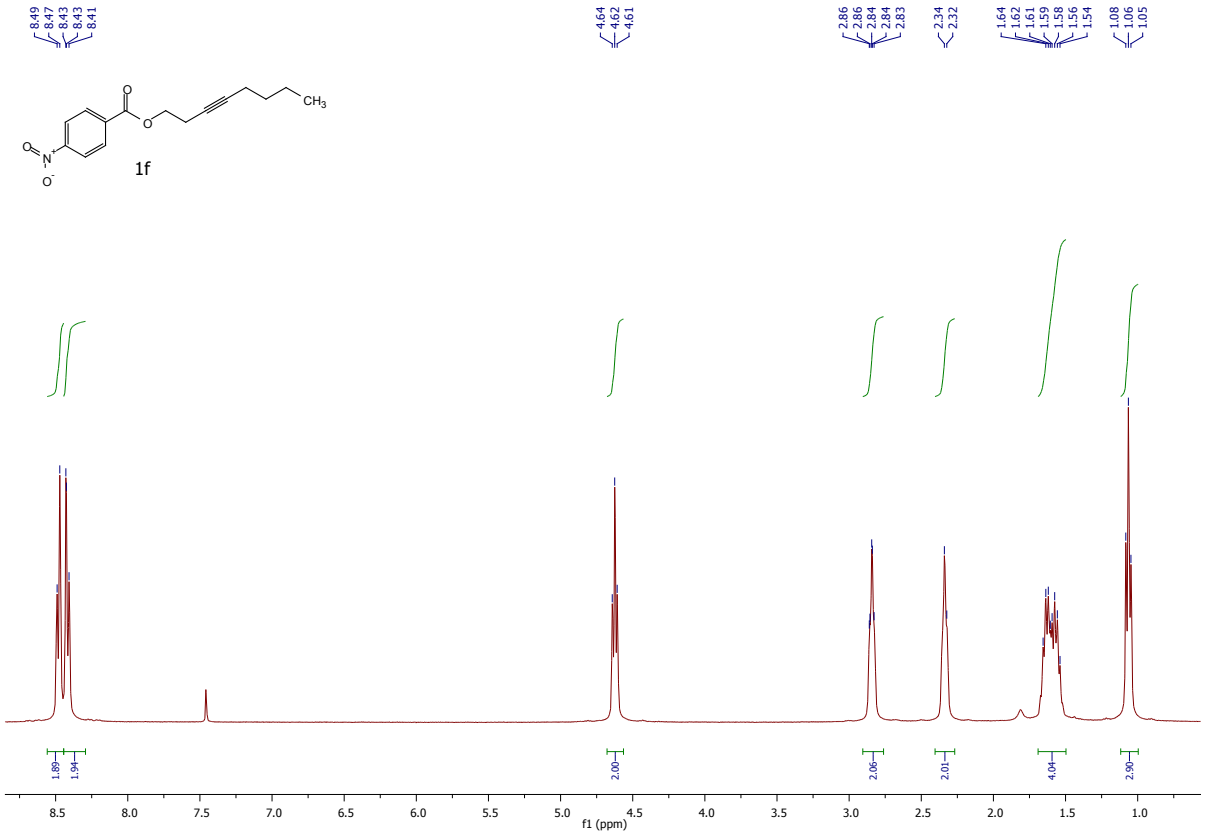
27.47
24.81
17.93
CH
1e
f1 (ppm)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8.43
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4.62
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2.86
2.84
2.84
2.83
2.34
2.32
1.64
1.62
1.61
1.59
1.58
1.56
1.54
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1f
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2.00
2.06
2.01
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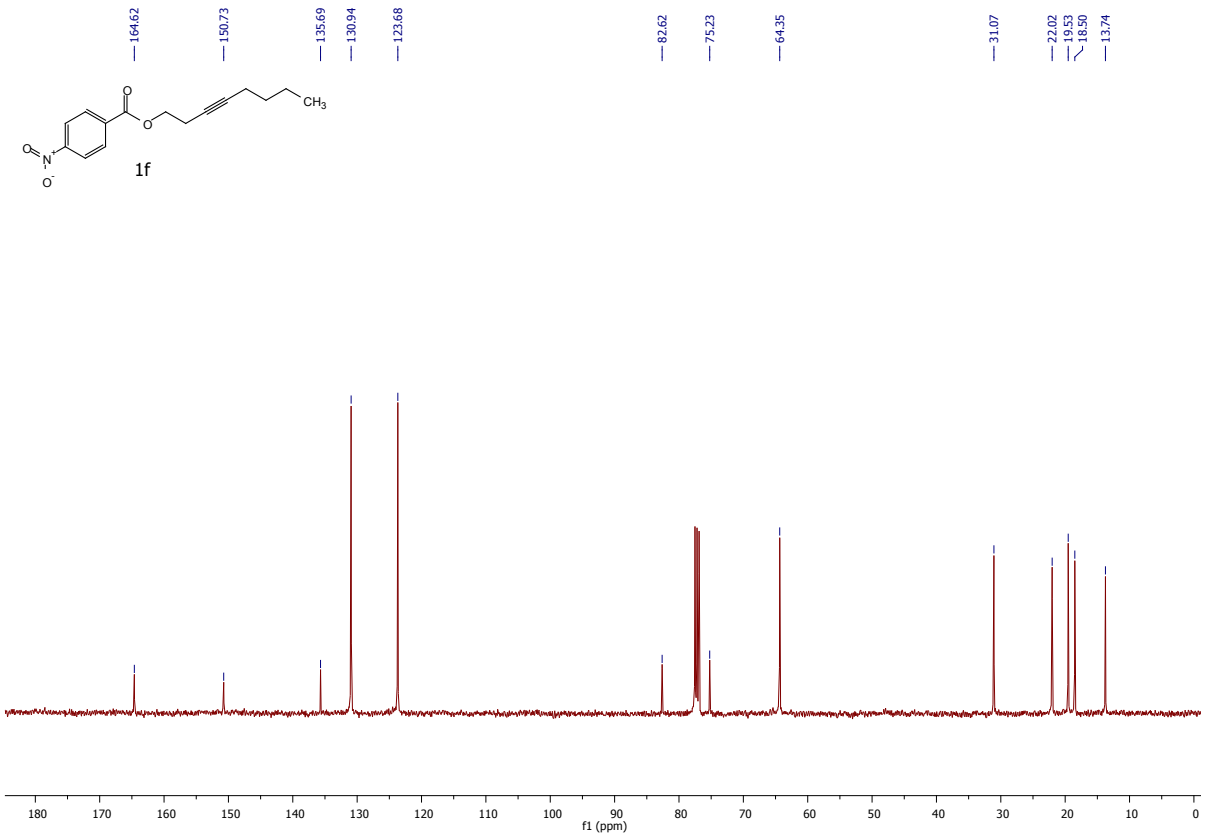
f1 (ppm)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19.53
18.50
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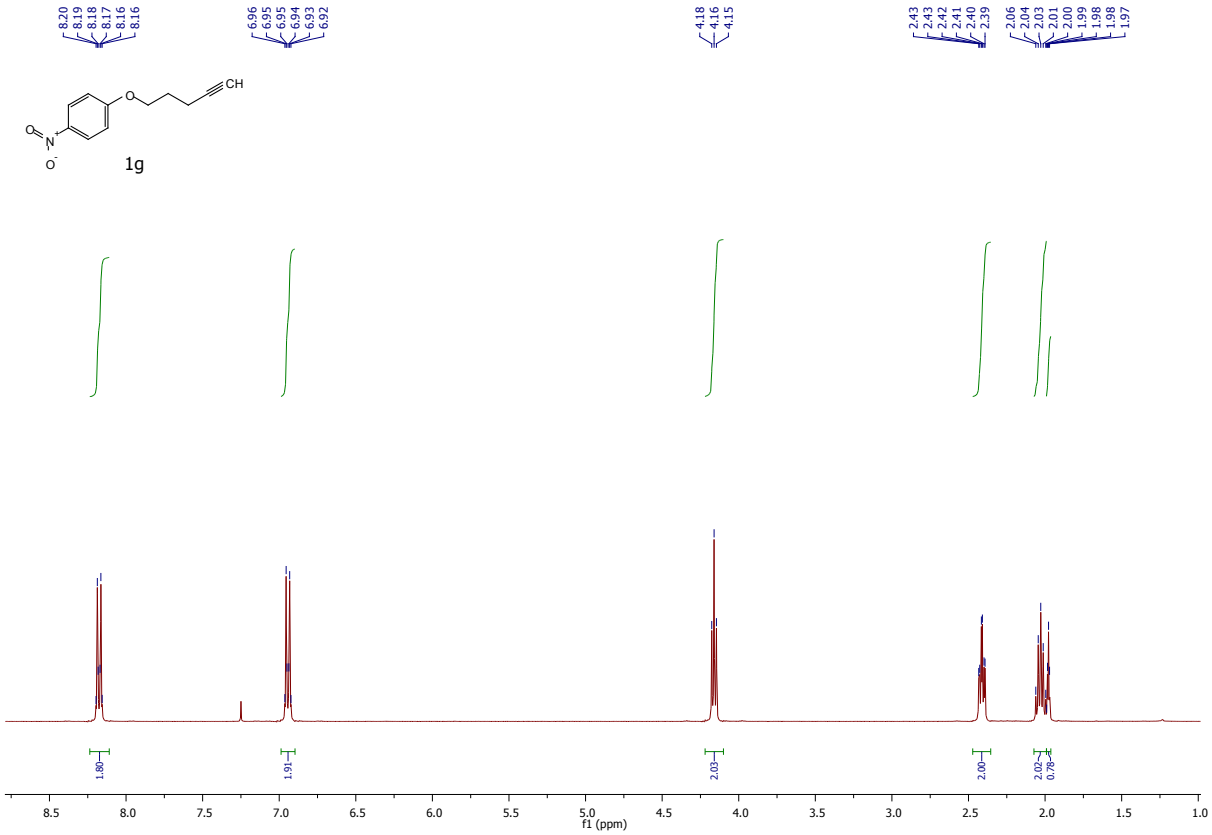
1f
f1 (ppm)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8.17
8.16
8.16
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6.95
6.95
6.94
6.93
6.92
4.18
4.16
4.15
2.43
2.43
2.42
2.41
2.40
2.39
2.06
2.04
2.03
2.01
2.00
1.99
1.98
1.98
1.97
CH
O
N
O
O
1g
1.80
1.91
2.03
2.00
2.03
0.78
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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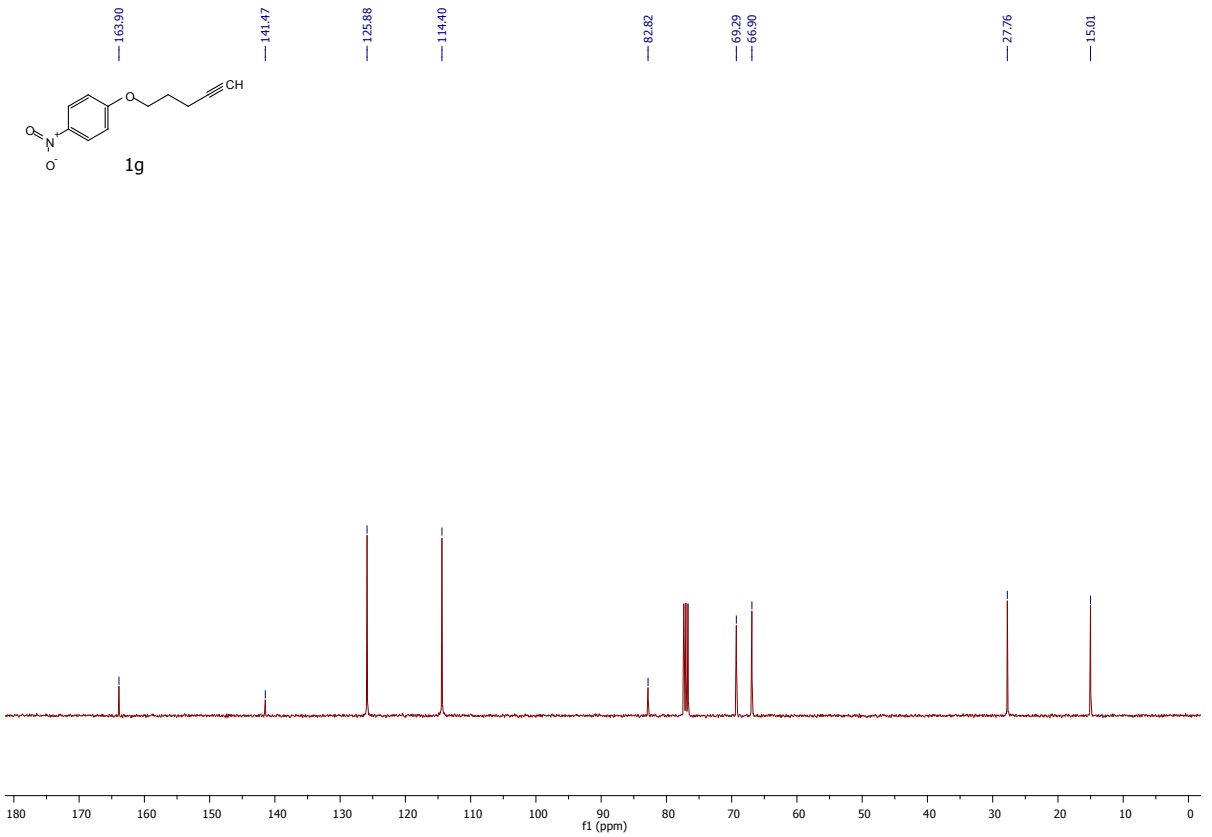
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)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66.90
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15.01
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O
N
O
O
1g
180
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120
110
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90
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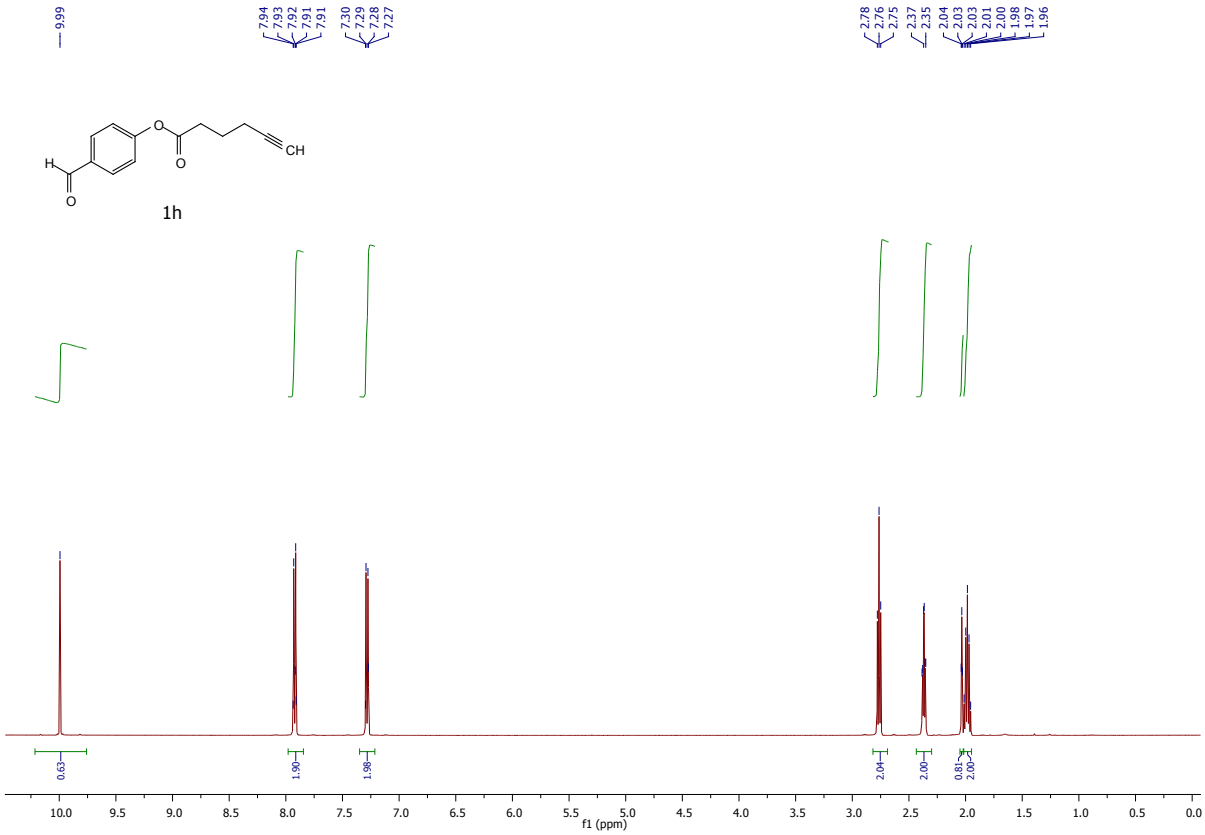
50
40
30
20
10
0
f1 (ppm)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7.91
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7.29
7.28
7.27
2.78
2.76
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2.04
2.03
2.03
2.01
2.00
1.98
1.97
1.96
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H
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CH
1h
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1.90
1.98
2.04
2.00
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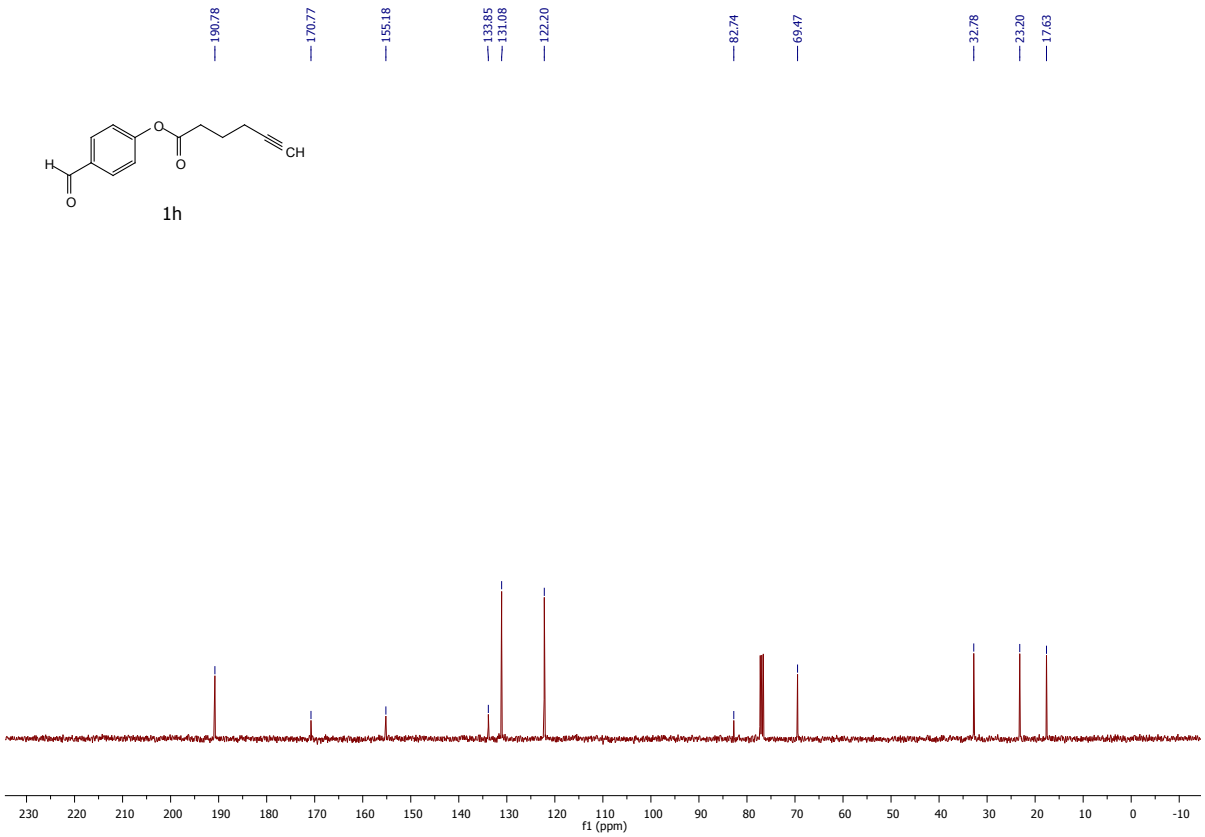
2.00
f1 (ppm)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23.20
17.63
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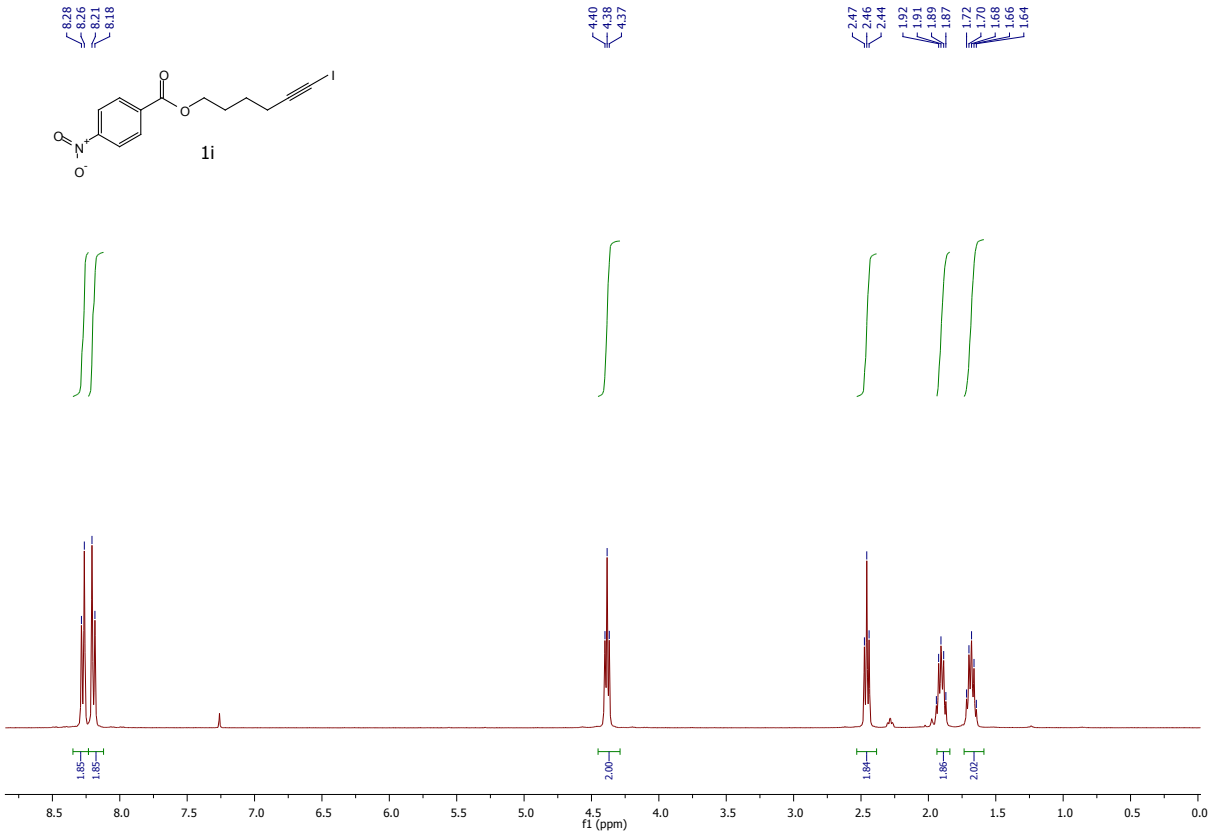
O
H
O
CH
1h
f1 (ppm)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1.91
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2.00
1.84
1.86
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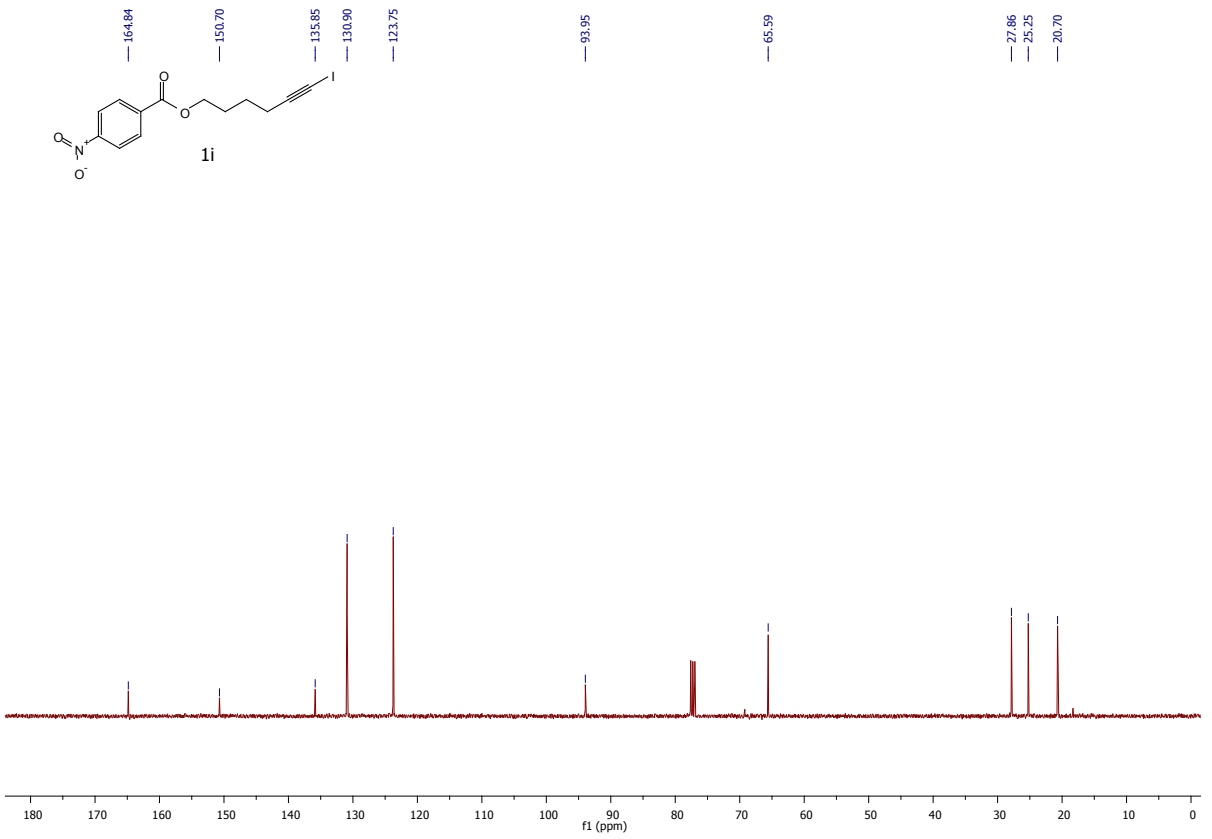
f1 (ppm)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93.95
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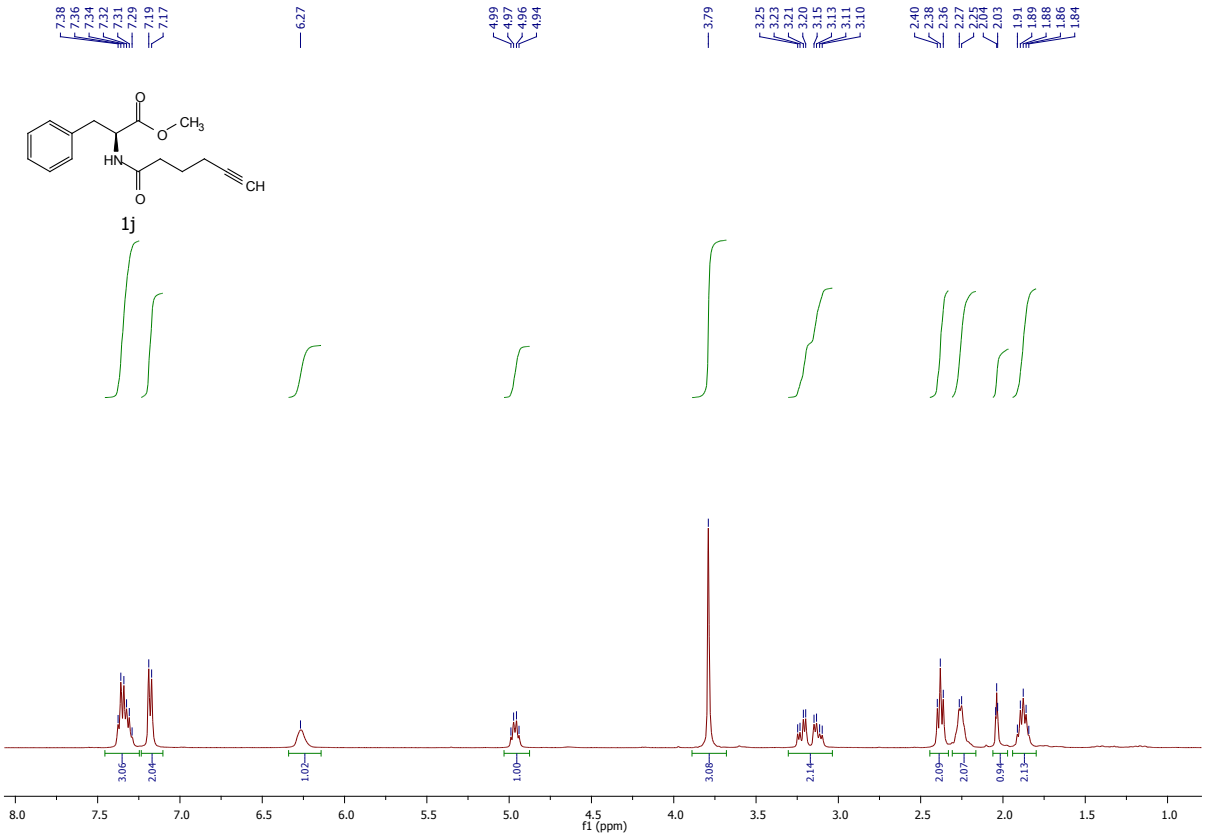
27.86
25.25
20.70
1i
f1 (ppm)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7.29
7.19
7.17
6.27
4.99
4.97
4.96
4.94
3.79
3.25
3.23
3.21
3.20
3.15
3.13
3.11
3.10
2.40
2.38
2.36
2.27
2.25
2.04
2.03
1.91
1.89
1.88
1.86
1.84
O
O
CH3
HN
O
CH
1j
3.06
2.04
1.02
1.00
3.08
2.14
2.09
2.07
0.94
2.13
8.0
7.5
7.0
6.5
6.0
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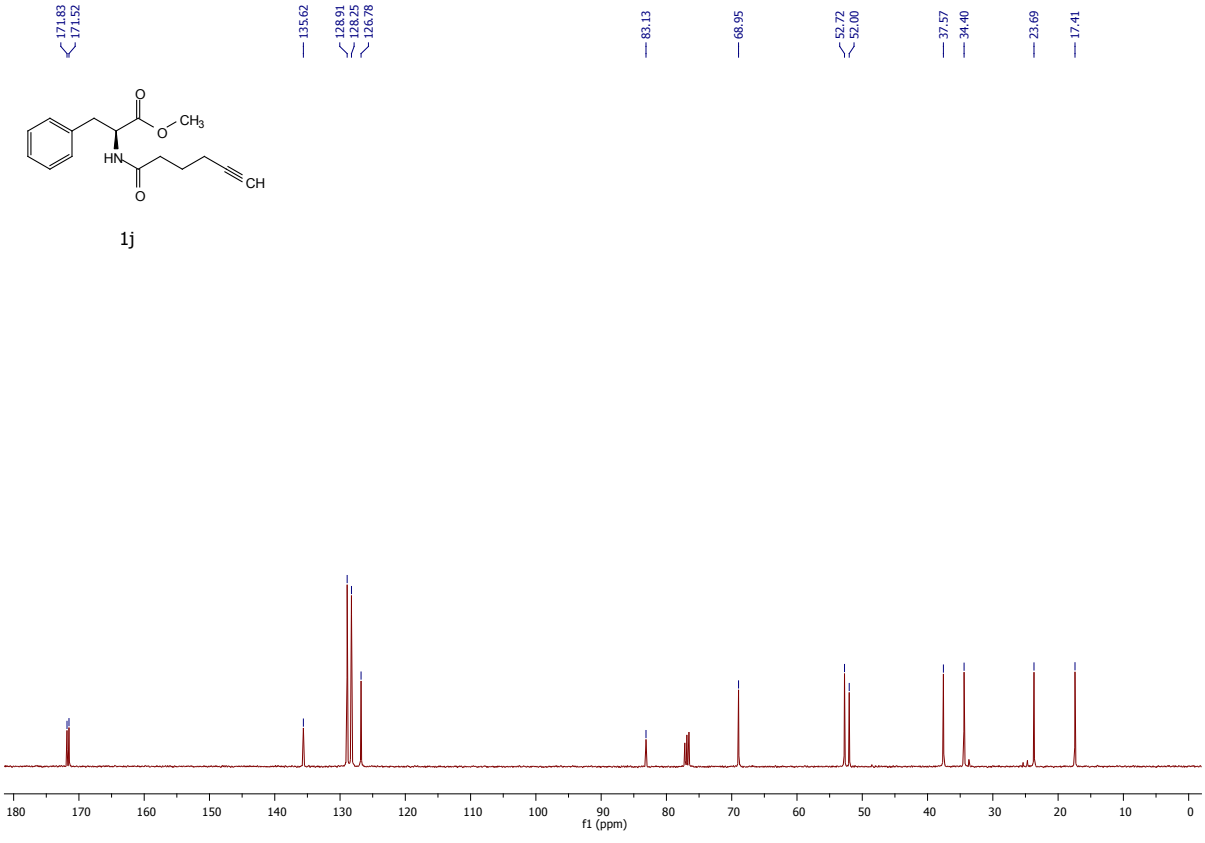
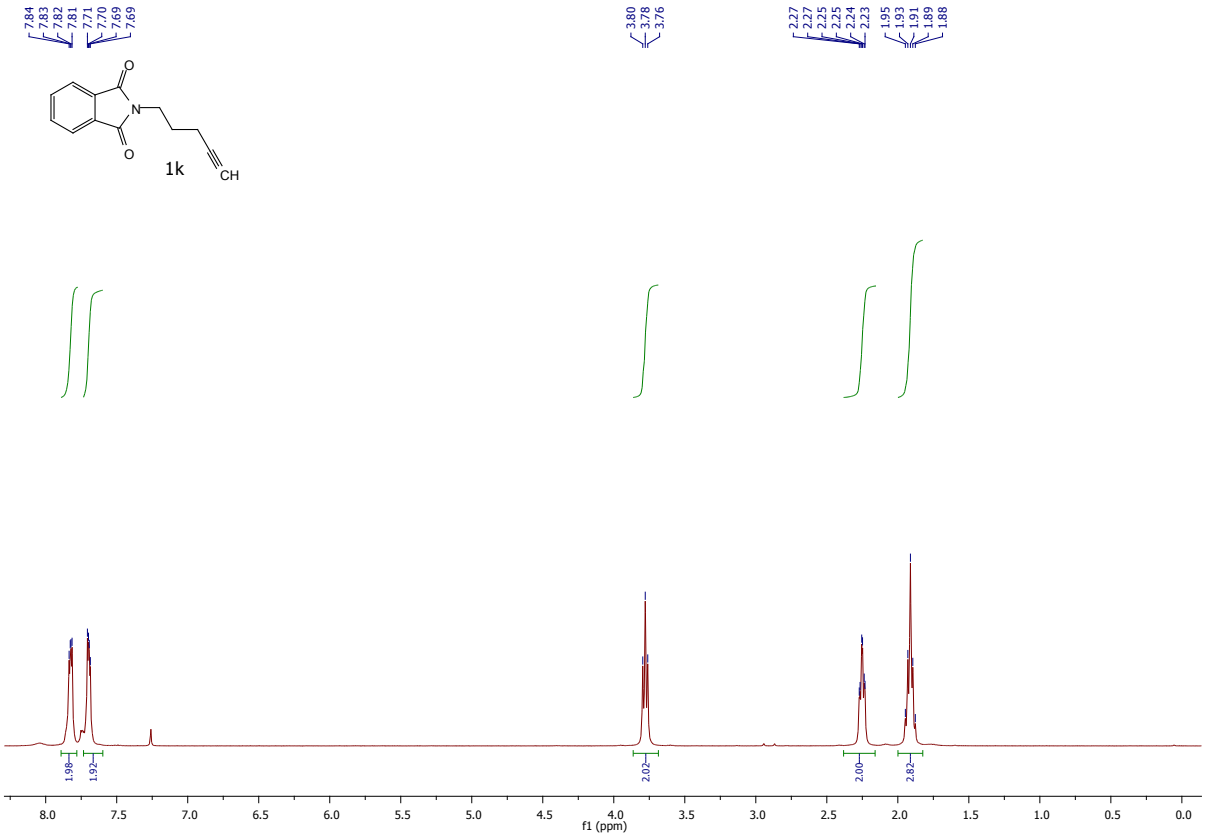
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)


171.83
171.52
135.62
128.91
128.25
126.78
83.13
68.95
52.72
52.00
37.57
34.40
23.69
17.41
O
O
CH3
HN
O
CH
1j
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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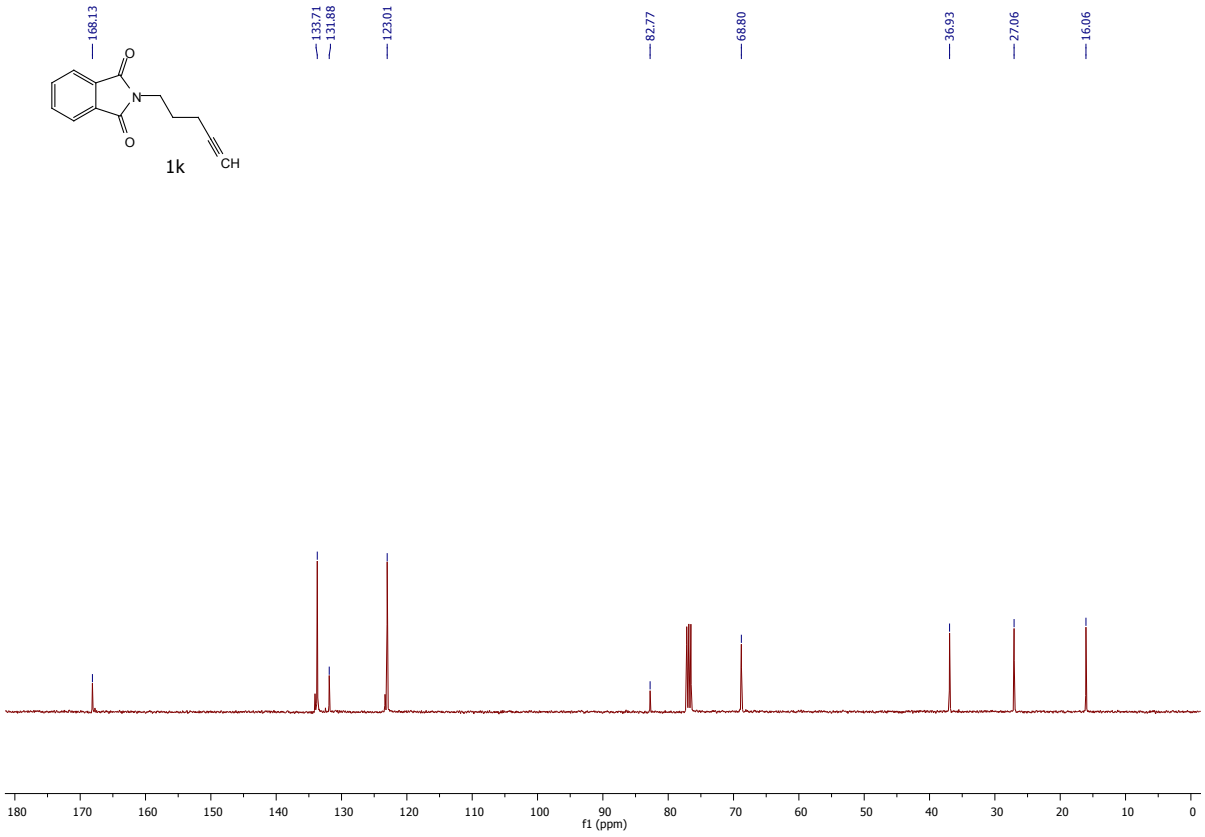
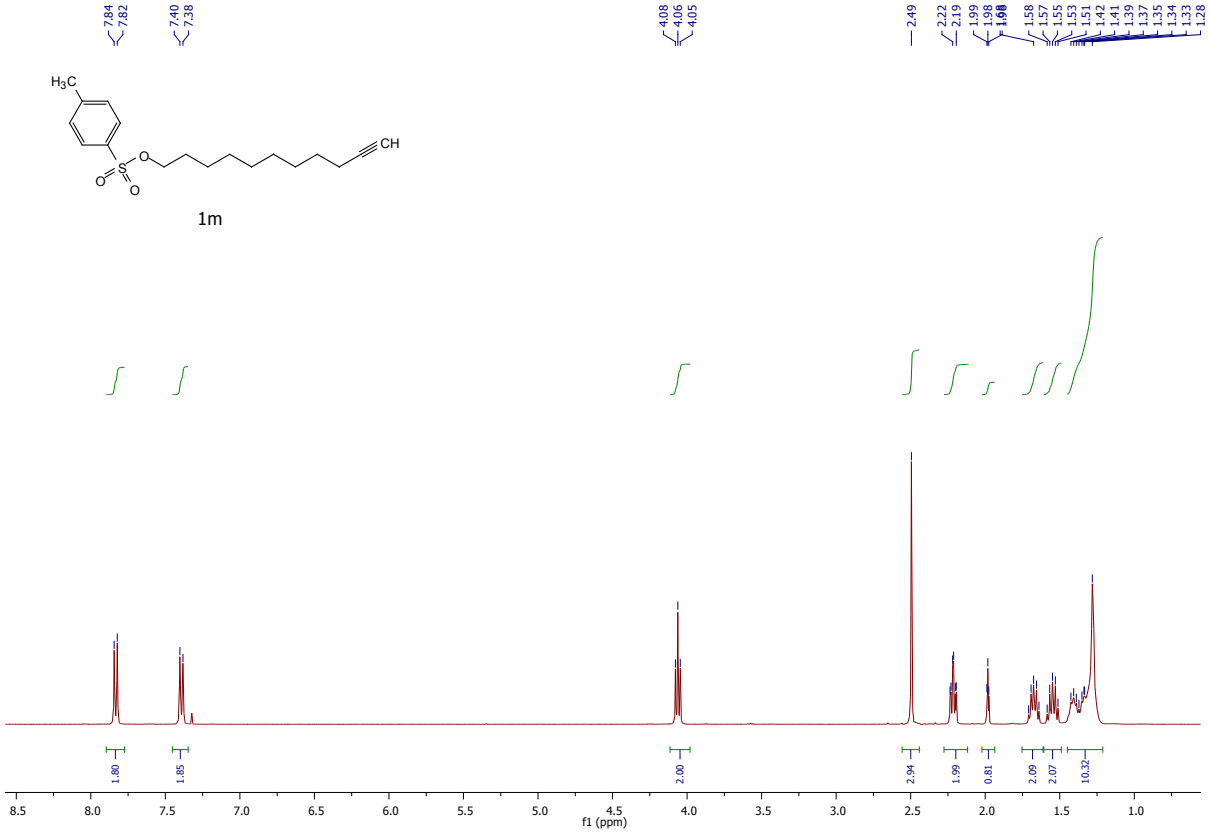
7.83
7.82
7.81
7.71
7.70
7.69
7.69
3.80
3.78
3.76
2.27
2.27
2.25
2.25
2.24
2.23
1.95
1.93
1.91
1.89
1.88
O
N
O
1k
CH
1.98
1.92
2.02
2.00
2.82
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


168.13
133.71
131.88
123.01
82.77
68.80
36.93
27.06
16.06
O
N
O
1k
CH
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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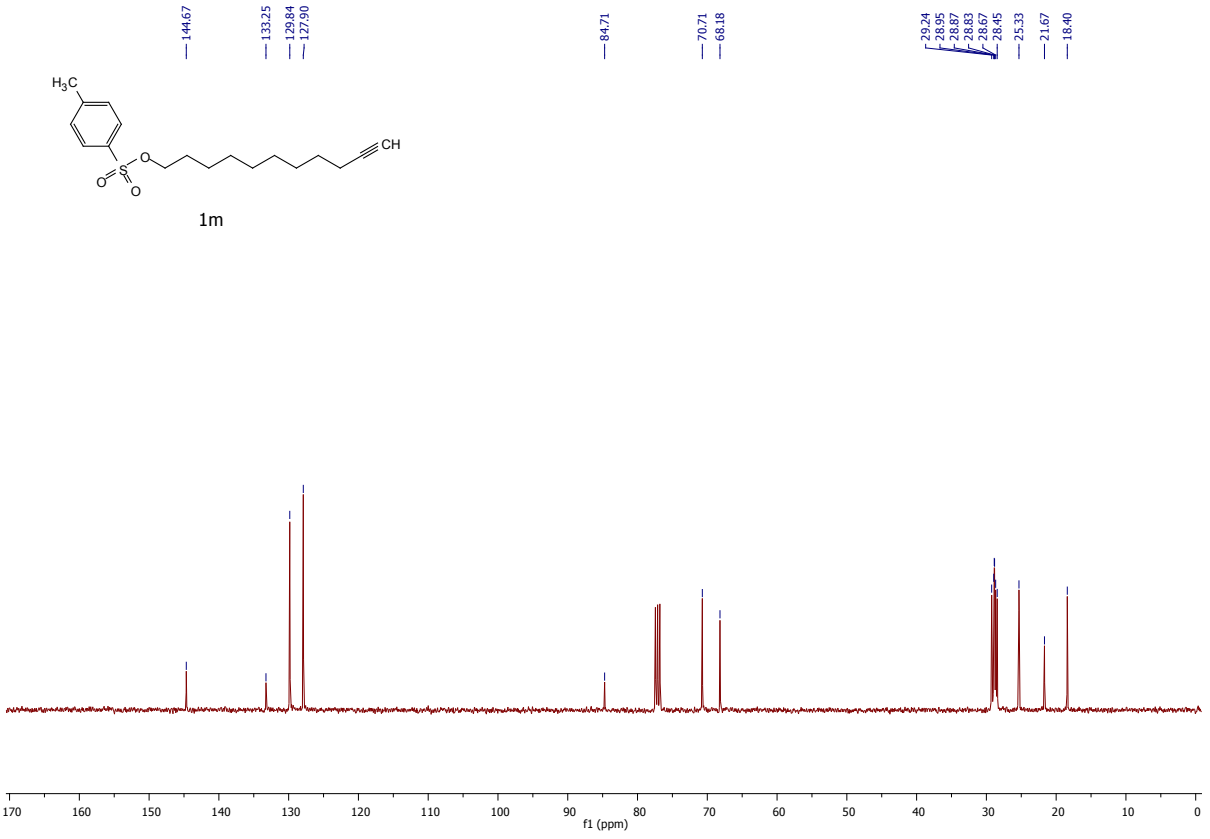
7.82
7.40
7.38
4.08
4.06
4.05
2.49
2.22
2.19
1.99
1.98
1.58
1.57
1.55
1.53
1.51
1.42
1.41
1.39
1.37
1.35
1.34
1.33
1.28
H3C
CH
O
S
O
O
1m
1.80
1.85
2.00
2.94
1.99
0.81
2.09
2.07
10.32
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)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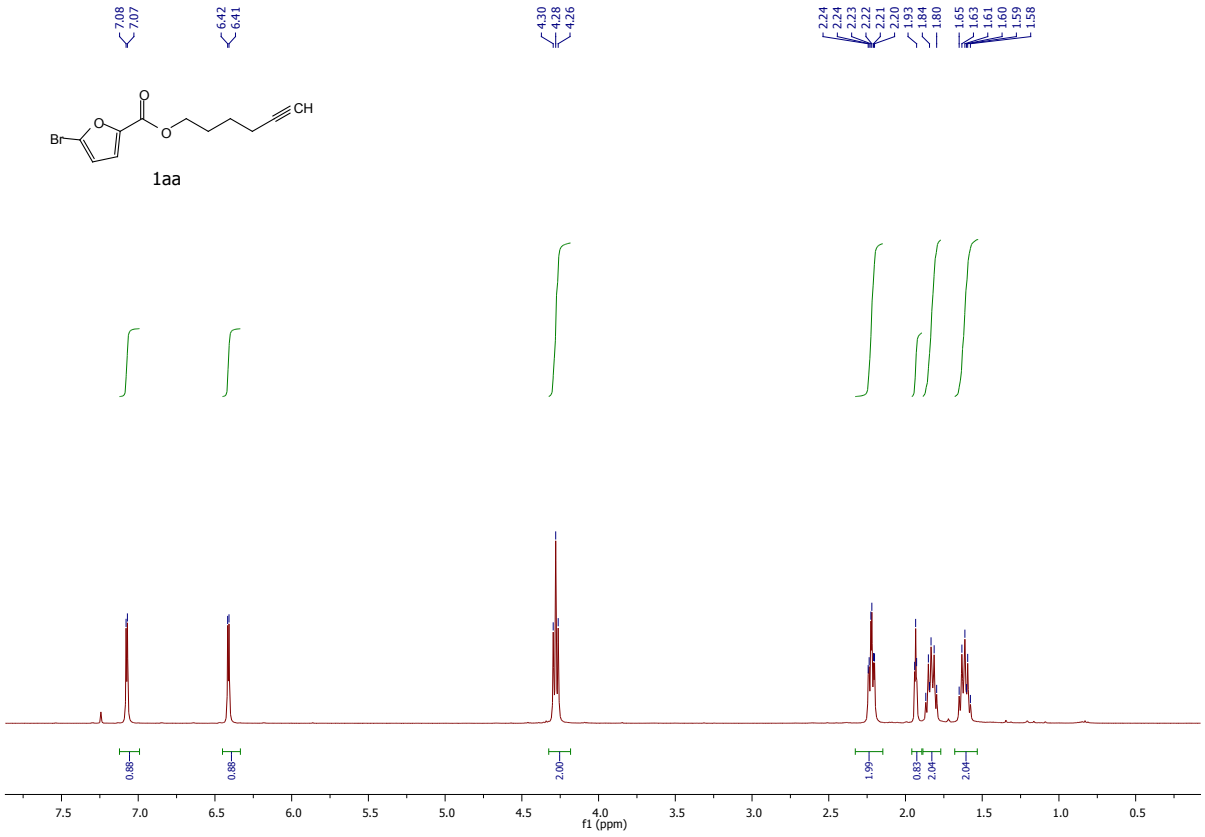
133.25
129.84
127.90
84.71
70.71
68.18
29.24
28.95
28.87
28.83
28.67
28.45
25.33
21.67
18.40
H3C
CH
O
S
O
O
1m
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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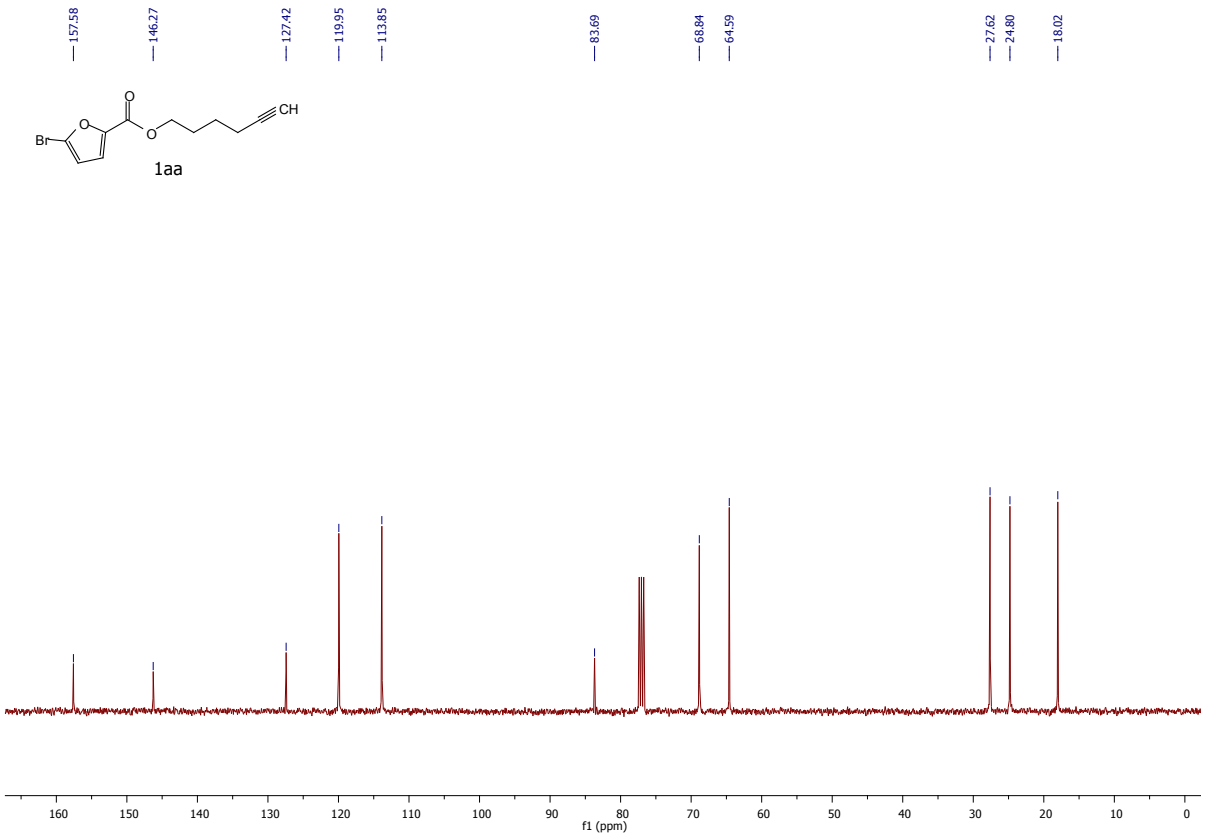
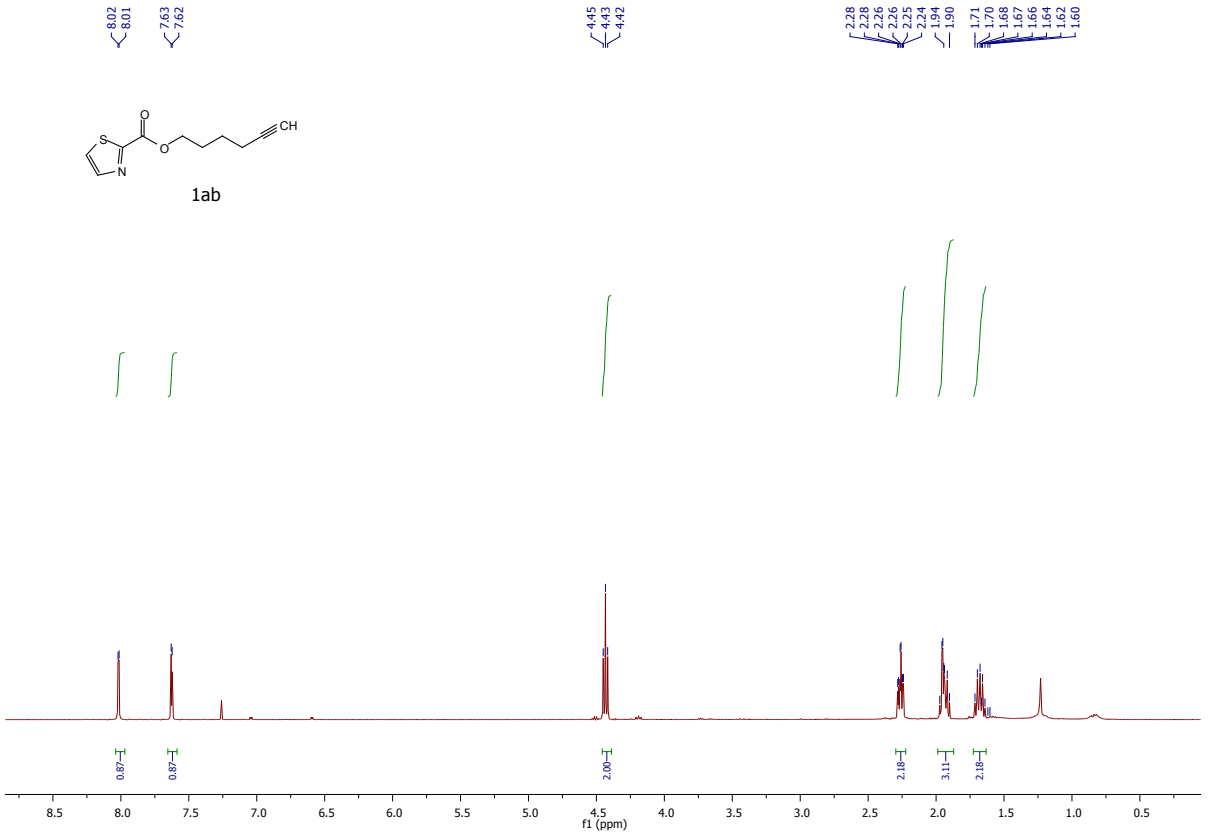
7.07
6.42
6.41
4.30
4.28
4.26
2.24
2.24
2.23
2.22
2.21
2.20
1.93
1.84
1.80
1.65
1.63
1.61
1.60
1.59
1.58
O
O
Br
CH
1aa
0.88
0.88
2.00
1.99
0.83
2.04
2.04
f1 (ppm)


157.58
146.27
127.42
119.95
113.85
83.69
68.84
64.59
27.62
24.80
18.02
O
O
Br
CH
1aa
f1 (ppm)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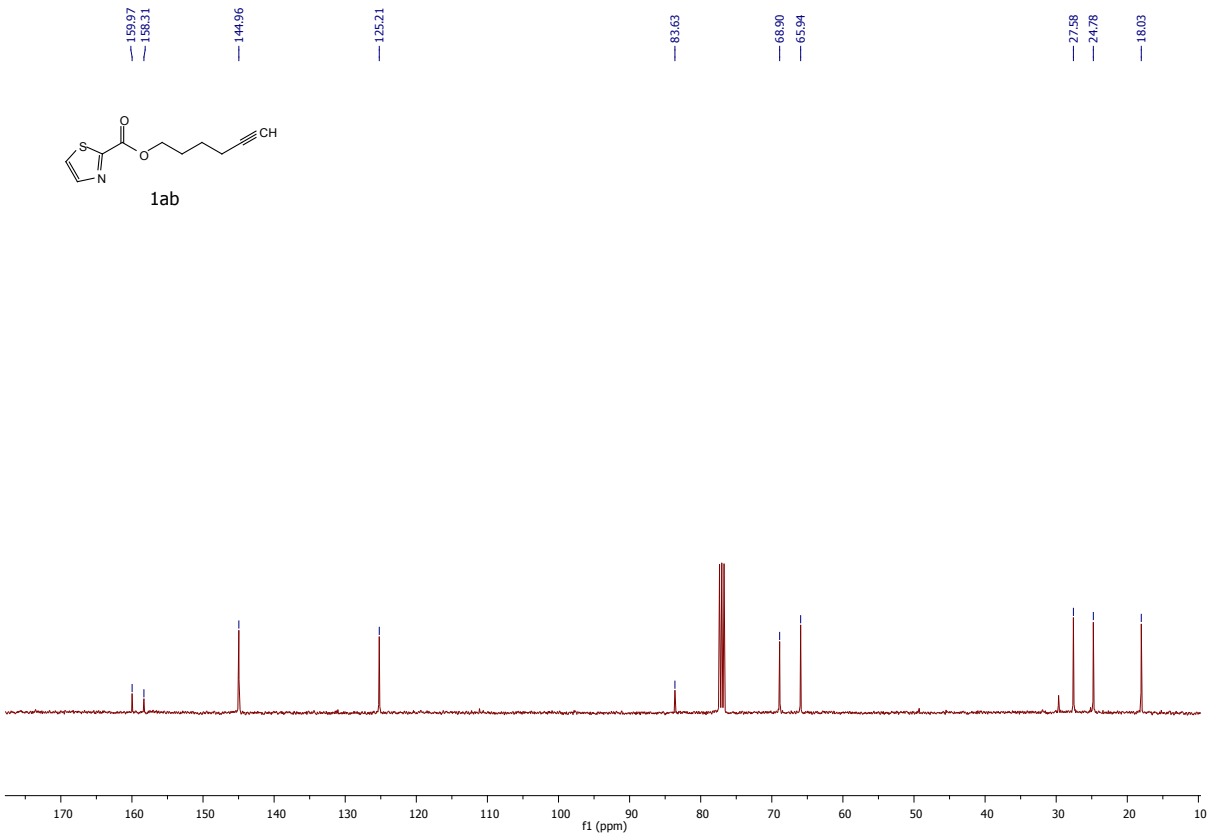
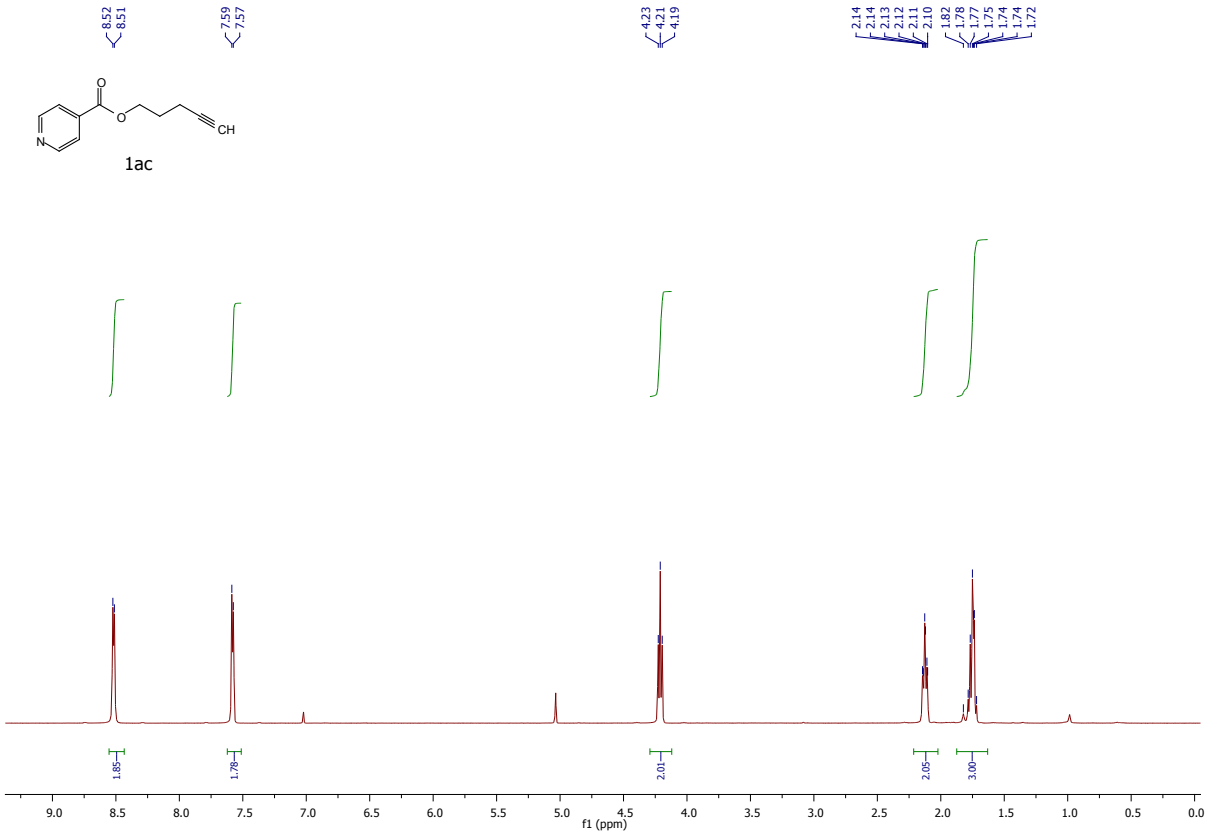
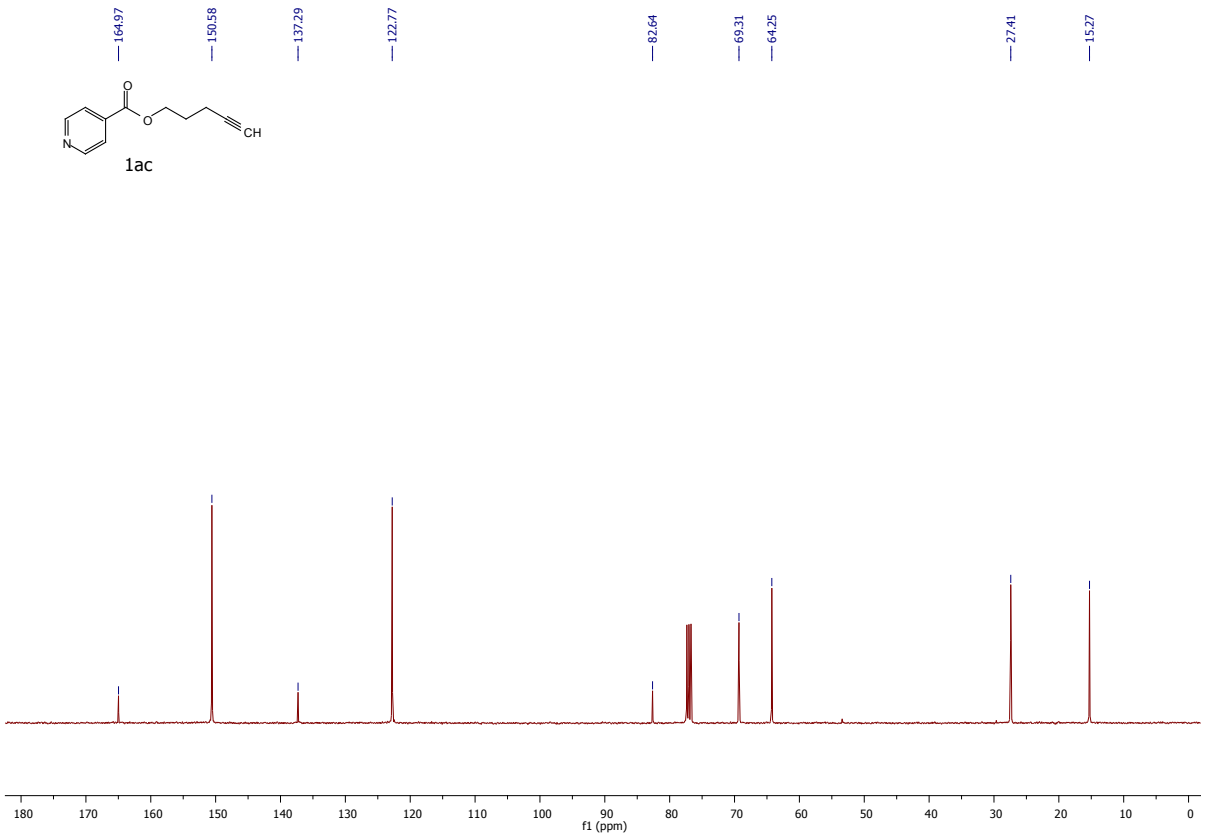
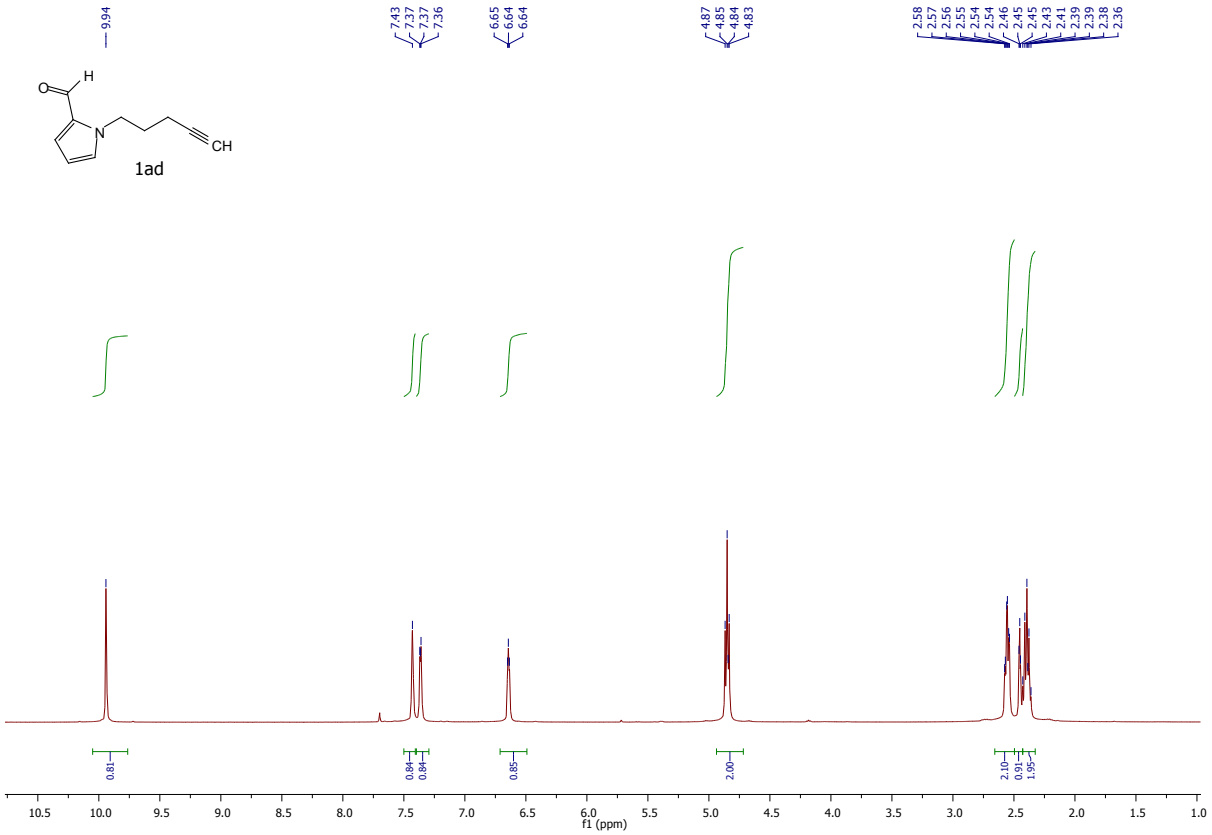
8.01
7.63
7.62
4.45
4.43
4.42
2.28
2.28
2.26
2.26
2.25
2.24
1.94
1.90
1.71
1.70
1.68
1.67
1.66
1.64
1.62
1.60
O
S
N
O
CH
1ab
0.87
0.87
2.00
2.18
3.11
2.18
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


159.97
158.31
144.96
125.21
83.63
68.90
65.94
27.58
24.78
18.03
O
S
N
O
CH
1ab
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


8.52
8.51
7.59
7.57
4.23
4.21
4.19
2.14
2.14
2.13
2.12
2.11
2.10
1.82
1.78
1.77
1.75
1.74
1.74
1.72
O
O
N
CH
1ac
1.85
1.78
2.01
2.05
3.00
f1 (ppm)


164.97
150.58
137.29
122.77
82.64
69.31
64.25
27.41
15.27
O
O
N
CH
1ac
f1 (ppm)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7.43
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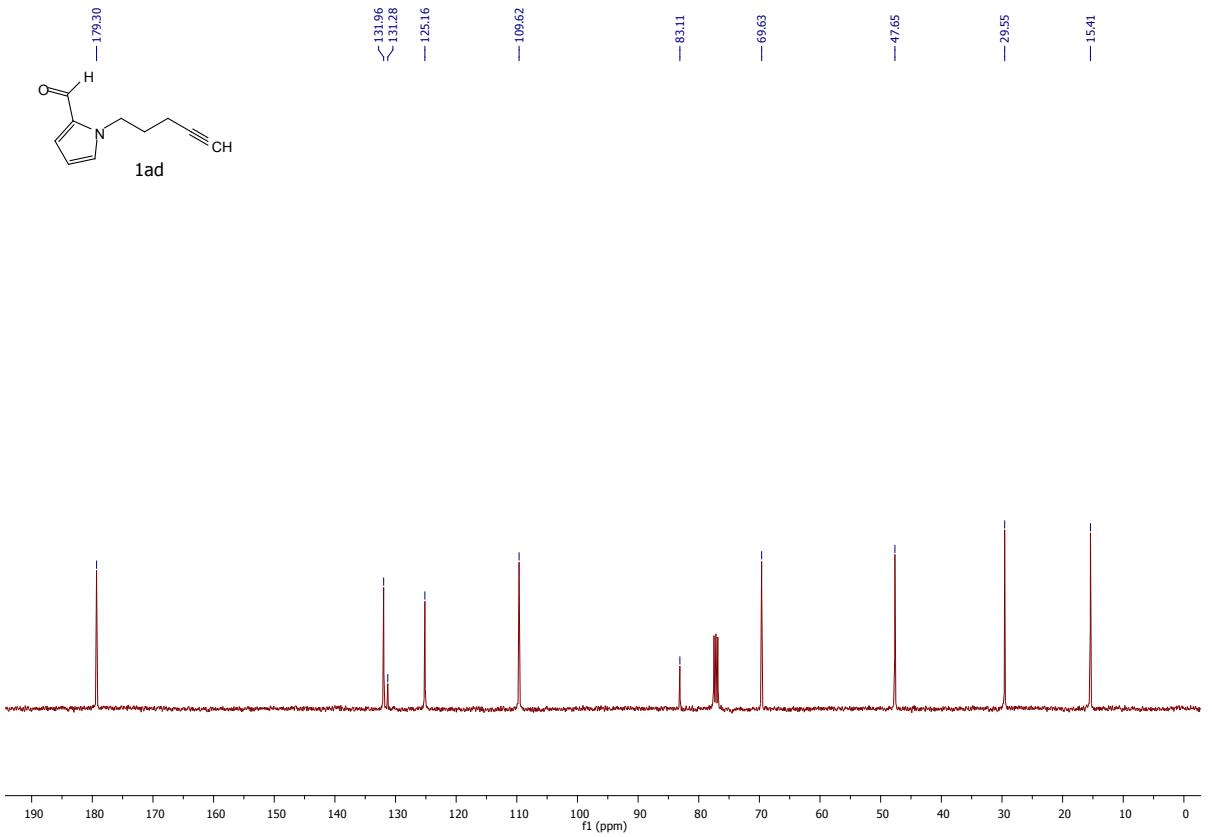
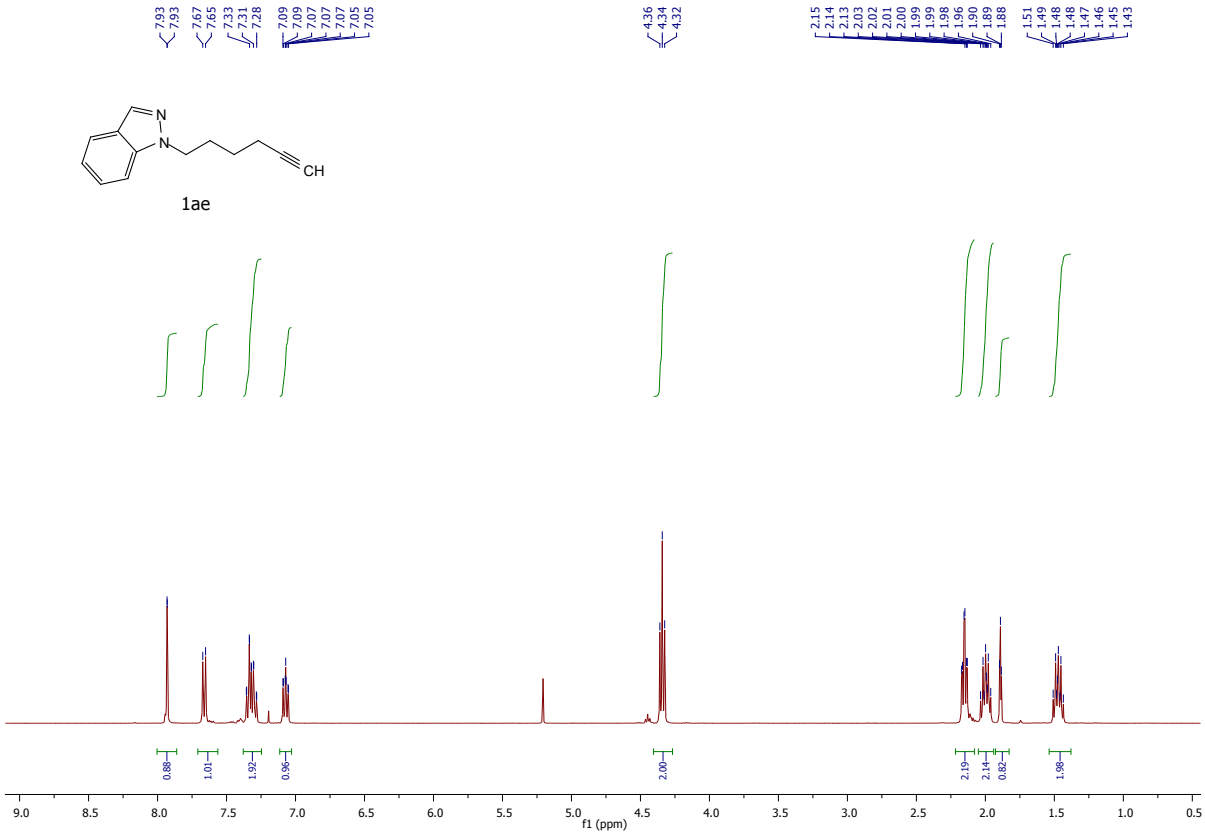
7.36
6.65
6.64
6.64
4.87
4.85
4.84
4.83
2.58
2.57
2.56
2.55
2.54
2.54
2.46
2.45
2.45
2.43
2.41
2.39
2.39
2.38
2.36
O
H
N
CH
1ad
0.81
0.84
0.84
0.85
2.00
2.10
0.91
1.95
f1 (ppm)


179.30
131.96
131.28
125.16
109.62
83.11
69.63
47.65
29.55
15.41
O
H
N
CH
1ad
f1 (ppm)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7.93
7.67
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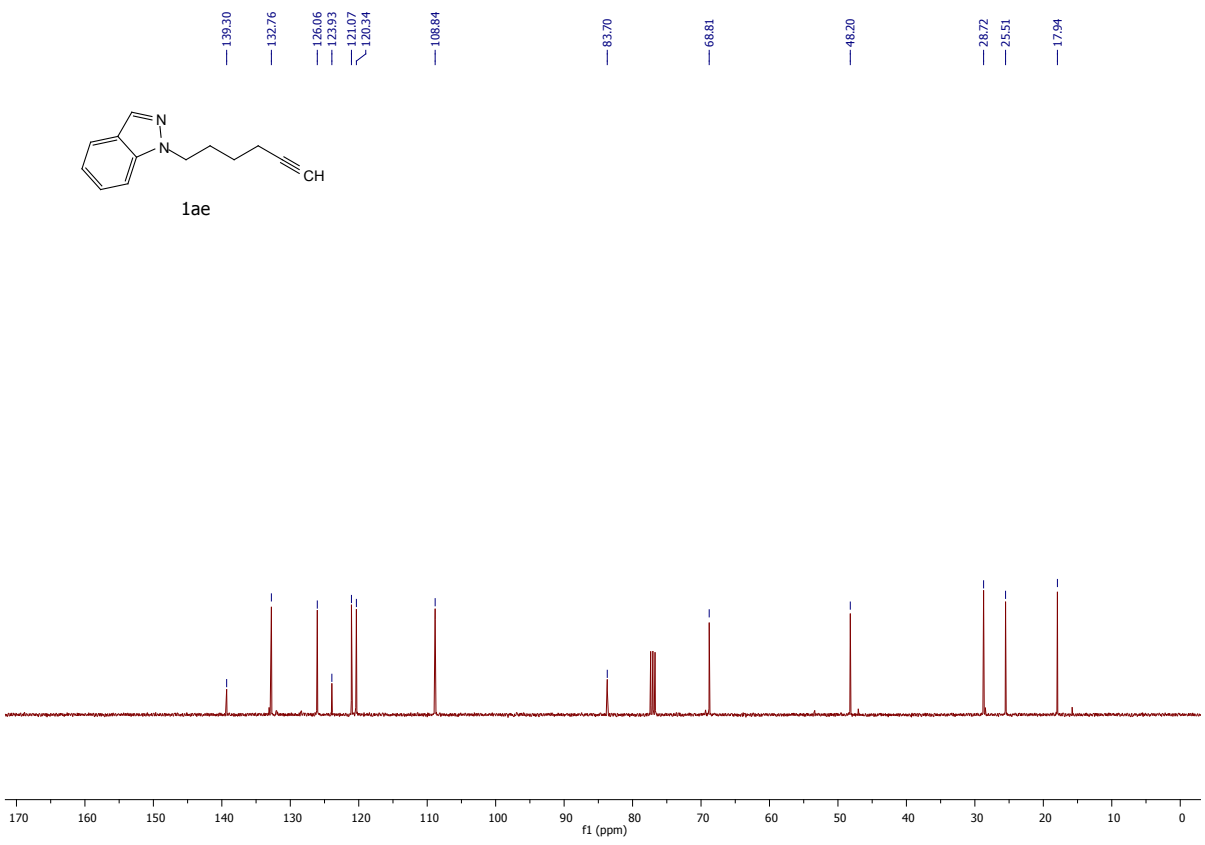
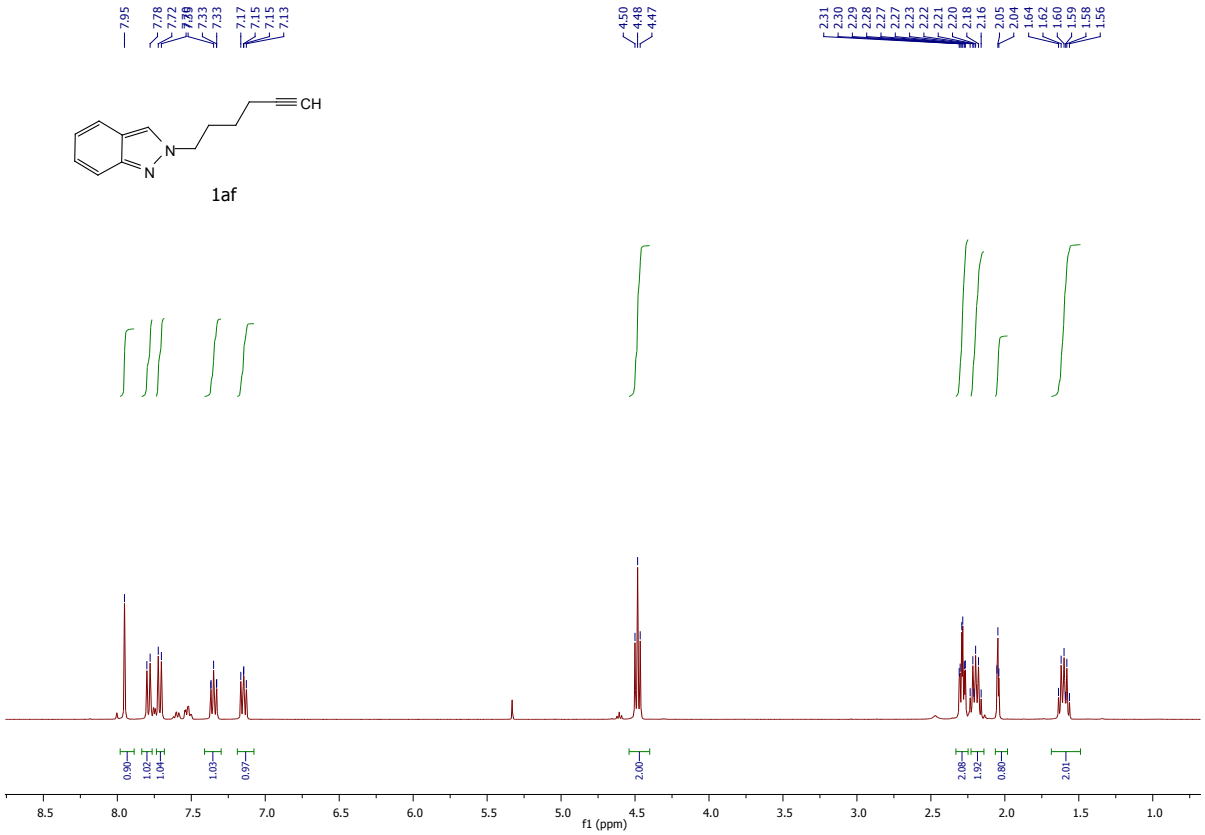
7.33
7.31
7.28
7.09
7.09
7.07
7.07
7.07
7.05
7.05
4.36
4.34
4.32
2.15
2.14
2.13
2.03
2.02
2.01
2.00
1.99
1.99
1.98
1.96
1.90
1.89
1.88
1.51
1.49
1.48
1.48
1.47
1.46
1.45
1.43
N
N
CH
1ae
0.88
1.01
1.92
0.96
2.00
2.19
2.14
0.82
1.98
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


139.30
132.76
126.06
123.93
121.07
120.34
108.84
83.70
68.81
48.20
28.72
25.51
17.94
N
N
CH
1ae
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.78
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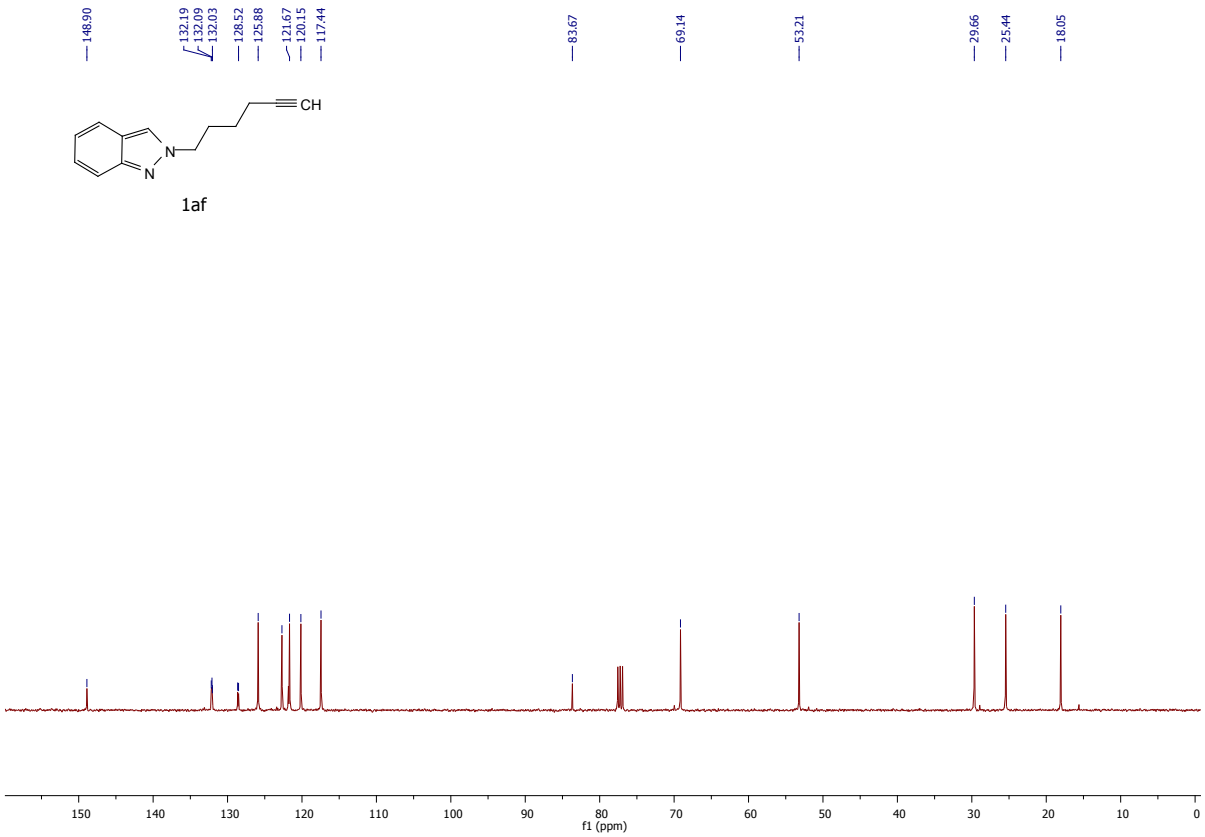
7.17
7.15
7.13
4.50
4.48
4.47
2.31
2.30
2.29
2.28
2.27
2.23
2.22
2.21
2.20
2.18
2.16
2.05
2.04
1.64
1.62
1.60
1.59
1.58
1.56
CH
N
N
1af
0.90
1.02
1.04
1.03
0.97
2.00
2.08
1.92
0.80
2.01
f1 (ppm)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132.03
128.52
125.88
121.67
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117.44
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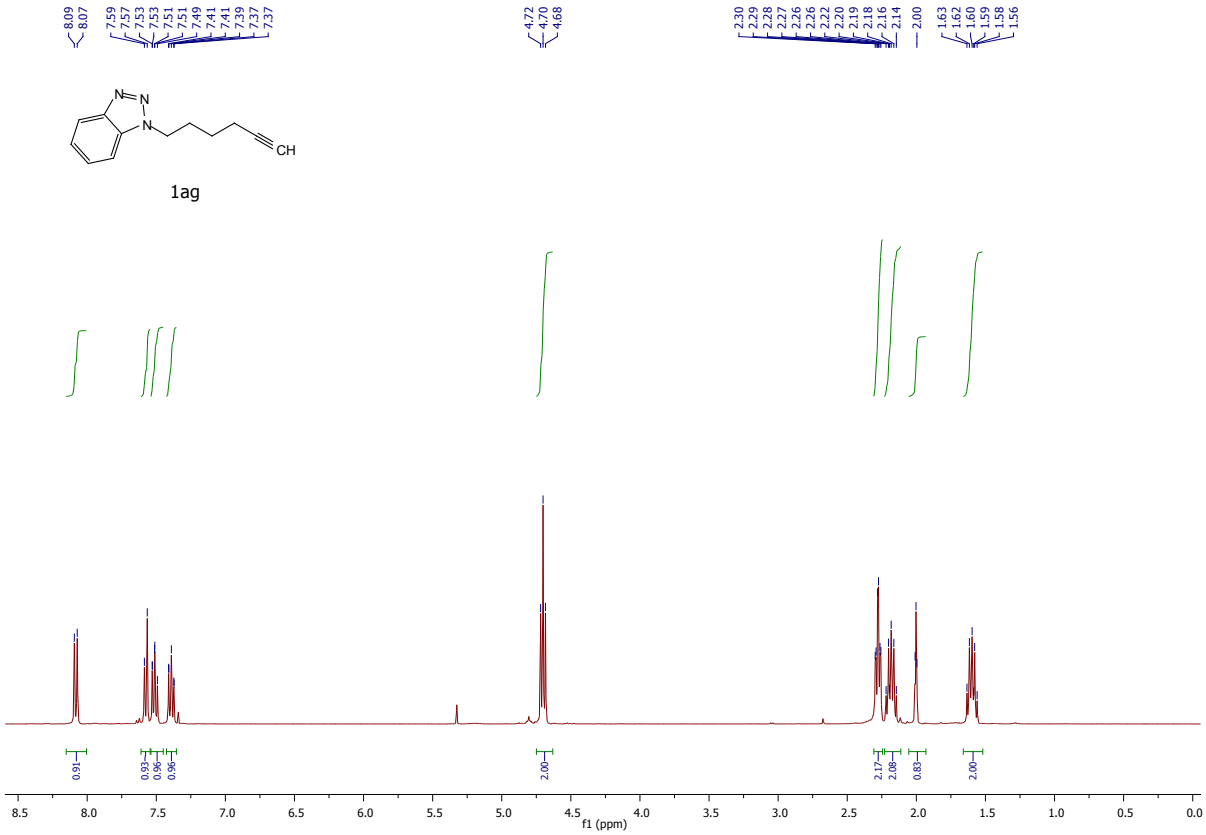
69.14
53.21
29.66
25.44
18.05
CH
N
N
1af
f1 (ppm)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8.07
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7.53
7.53
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7.51
7.49
7.41
7.41
7.39
7.37
7.37
4.72
4.70
4.68
2.30
2.29
2.28
2.27
2.26
2.26
2.22
2.20
2.19
2.18
2.16
2.14
2.00
1.63
1.62
1.60
1.59
1.58
1.56
N
N
N
CH
1ag
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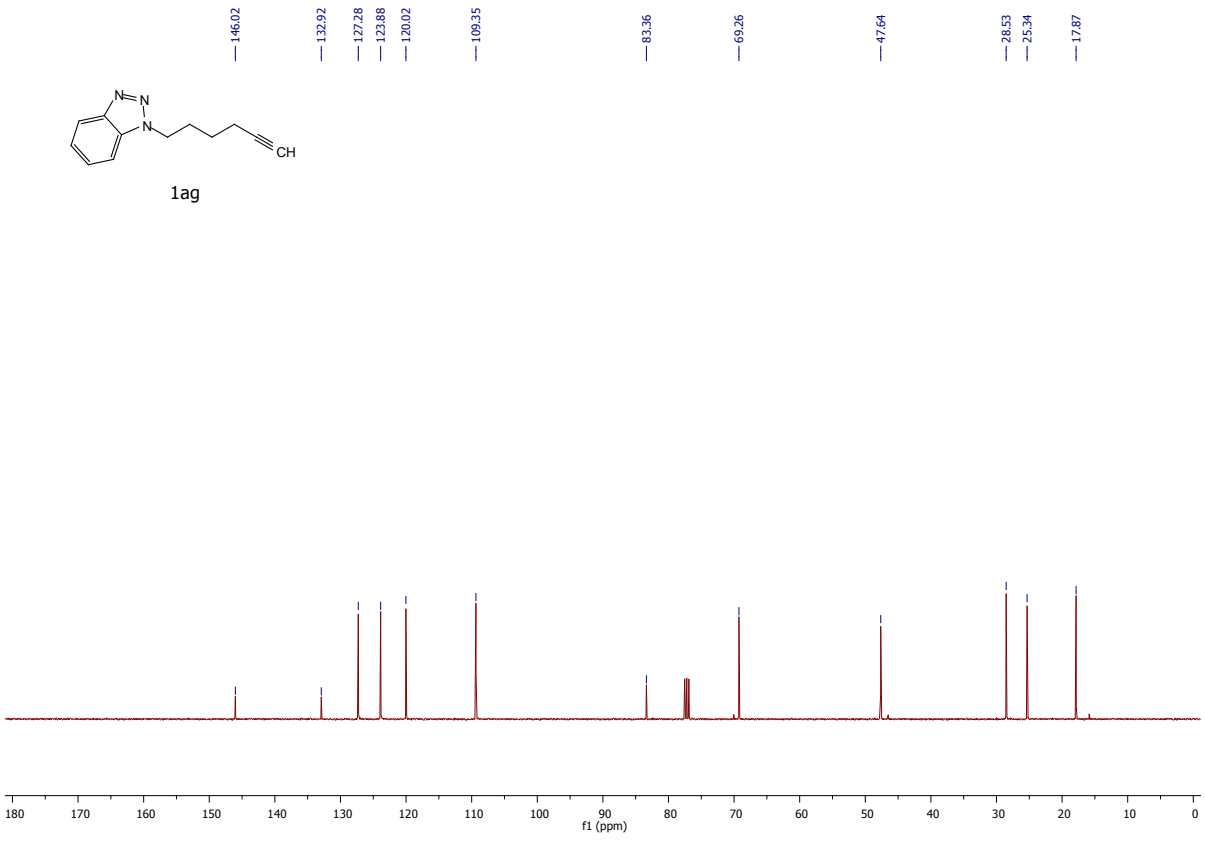
0.93
0.98
0.98
2.00
2.17
2.08
0.83
2.00
f1 (ppm)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123.88
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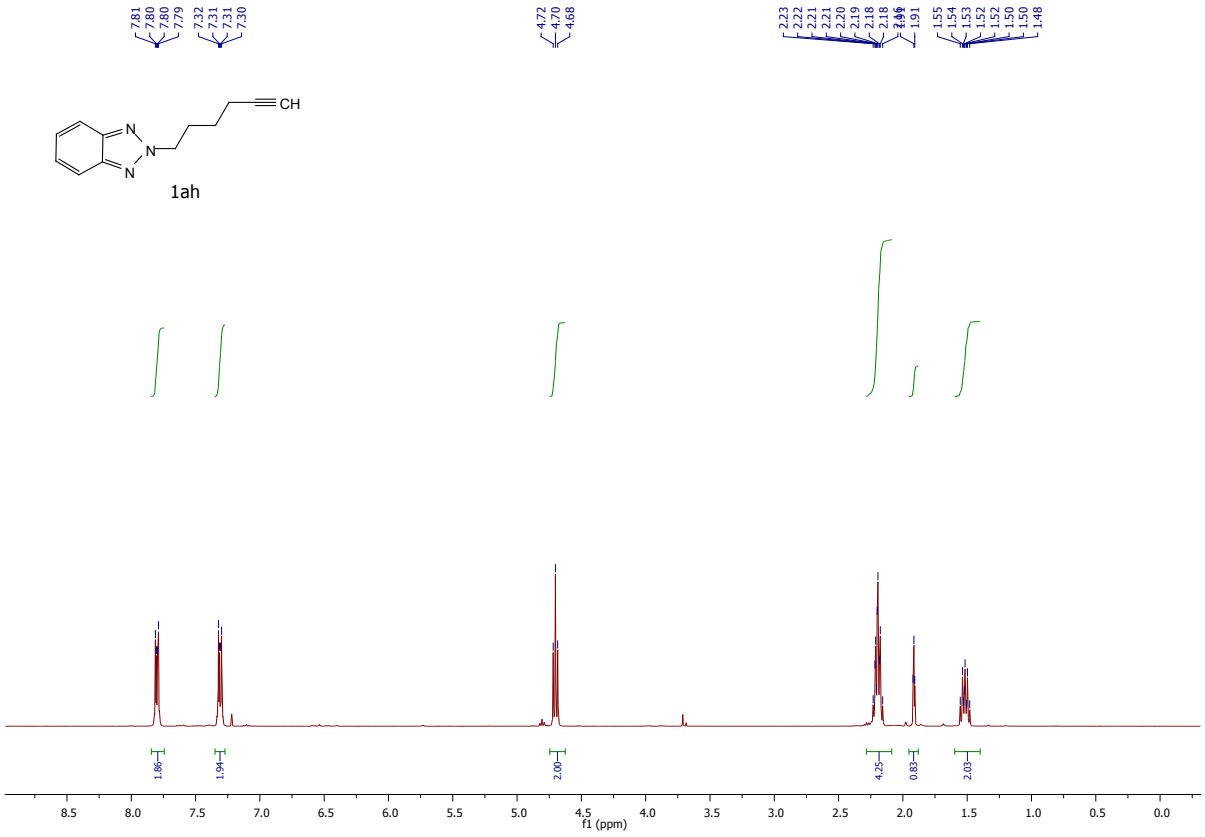
69.26
47.64
28.53
25.34
17.87
N
N
N
CH
1ag
f1 (ppm)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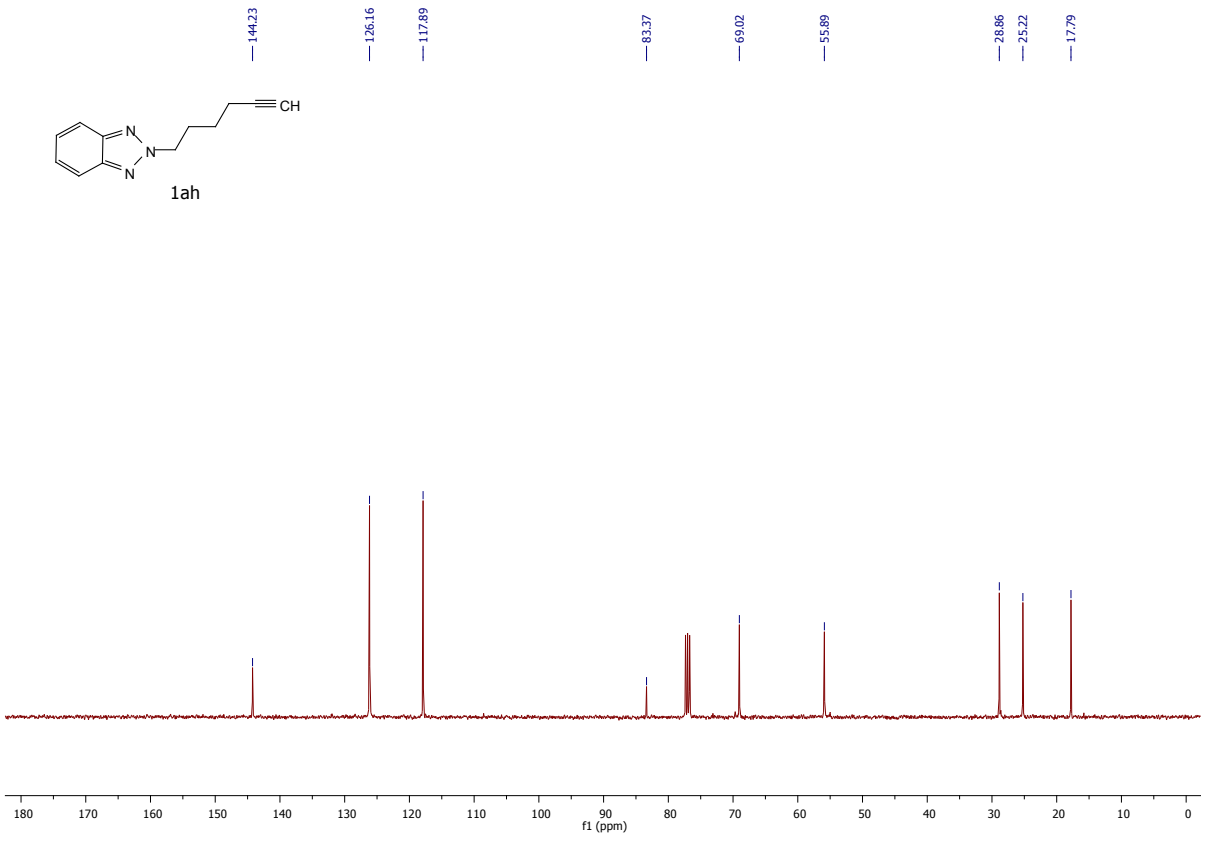
7.31
7.31
7.30
4.72
4.70
4.68
2.23
2.22
2.21
2.21
2.20
2.19
2.18
2.18
1.91
1.55
1.54
1.53
1.52
1.52
1.50
1.50
1.48
CH
N
N
N
1ah
1.86
1.94
2.00
4.25
0.83
2.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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69.02
55.89
28.86
25.22
17.79
CH
N
N
N
1ah
180
170
160
150
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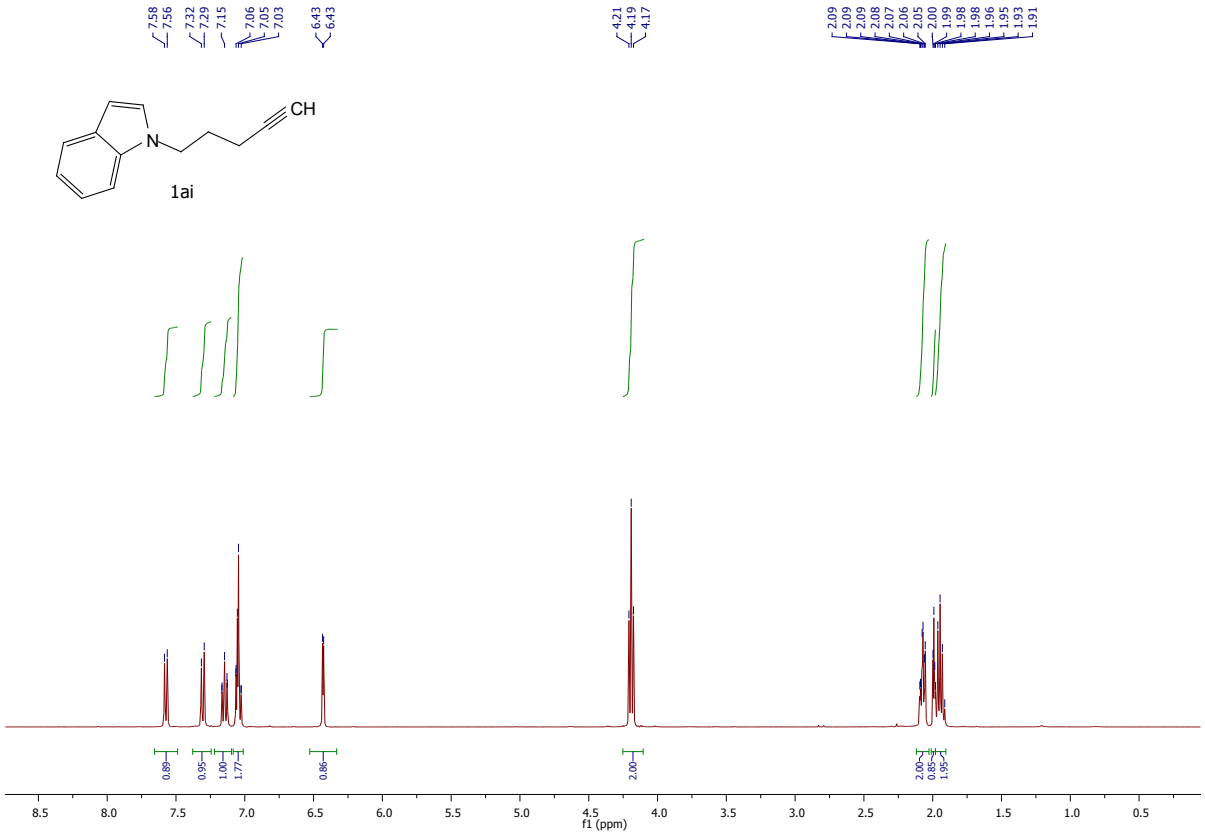
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.56
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7.29
7.15
7.06
7.05
7.03
6.43
6.43
4.21
4.19
4.17
2.09
2.09
2.09
2.08
2.07
2.06
2.05
2.00
1.99
1.98
1.98
1.96
1.95
1.93
1.91
CH
N
1ai
0.89
0.95
1.00
1.77
0.86
2.00
2.00
0.85
1.95
8.5
8.0
7.5
7.0
6.5
6.0
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5.0
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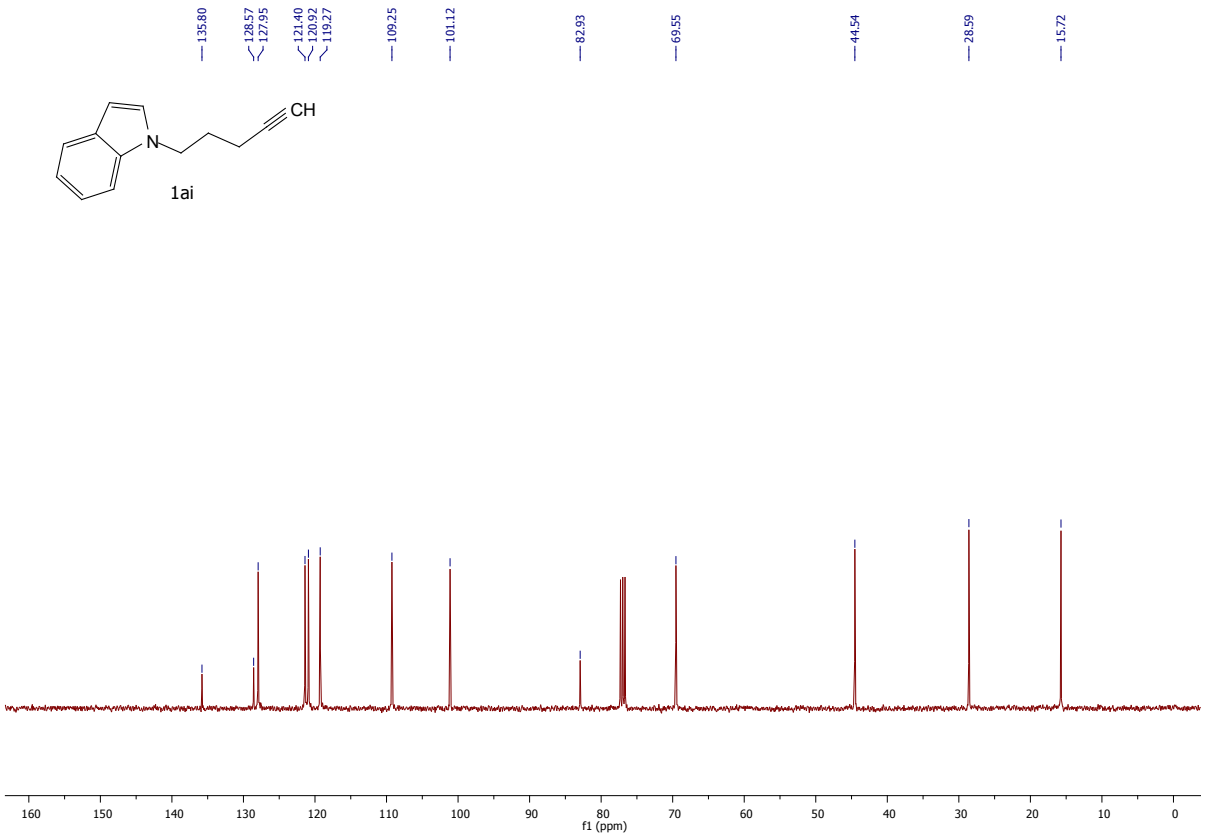
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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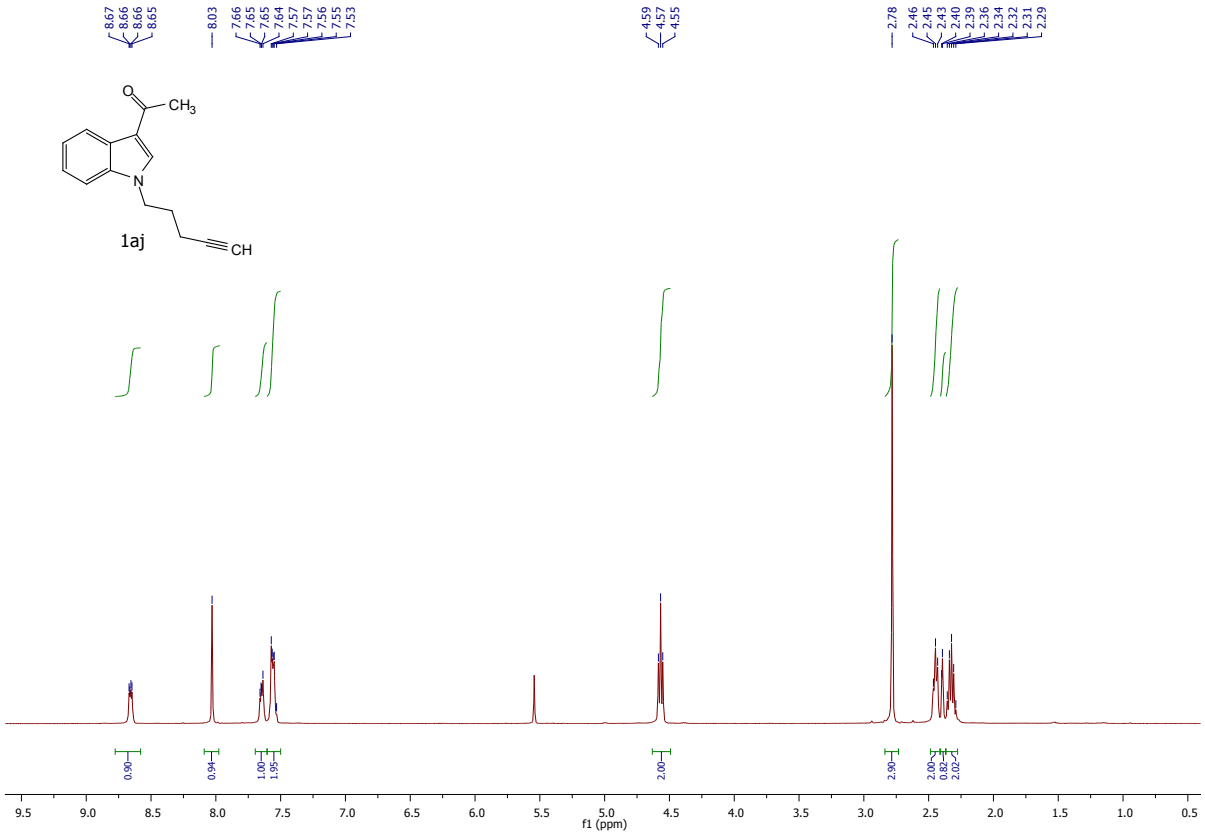
119.27
109.25
101.12
82.93
69.55
44.54
28.59
15.72
CH
N
1ai
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.65
7.65
7.64
7.57
7.57
7.56
7.55
7.53
4.59
4.57
4.55
2.78
2.46
2.45
2.43
2.40
2.39
2.36
2.34
2.32
2.31
2.29
O
CH3
N
CH
1aj
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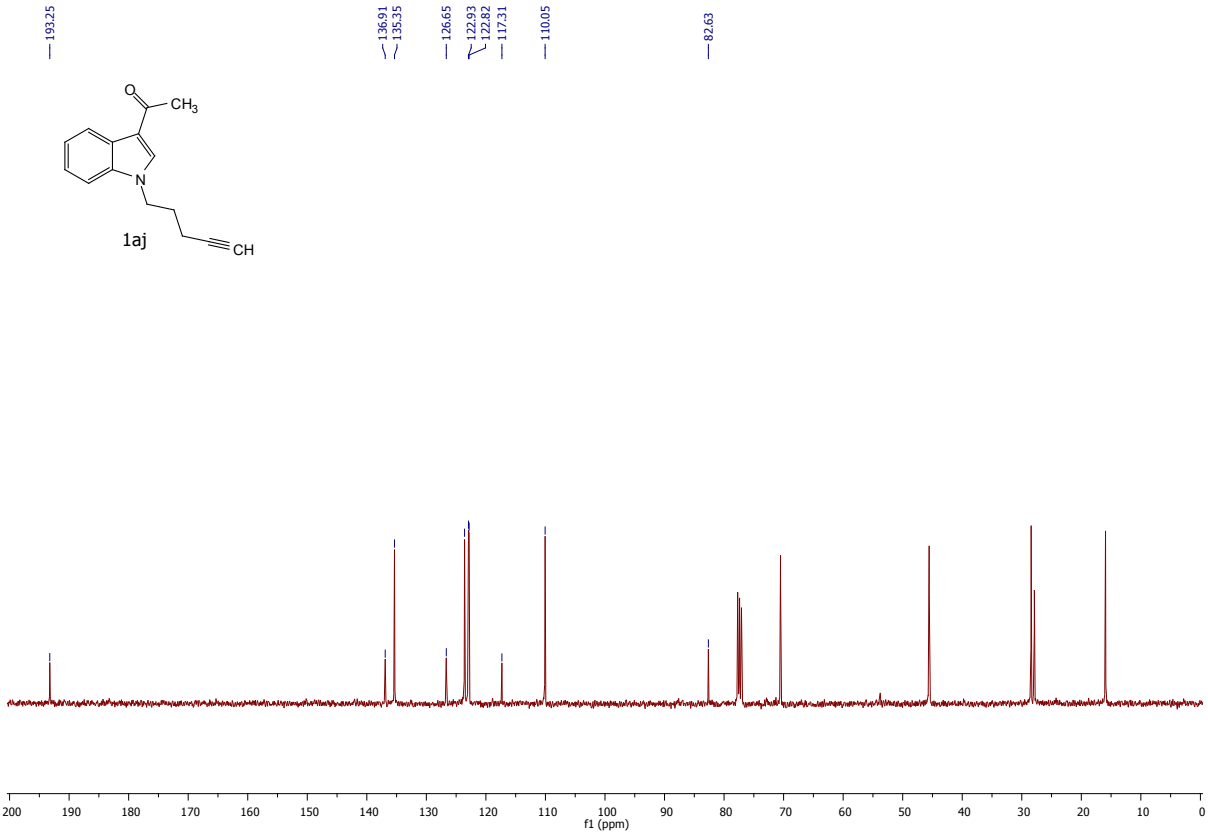
0.94
1.00
1.95
2.00
2.90
2.00
0.82
2.02
f1 (ppm)


193.25
136.91
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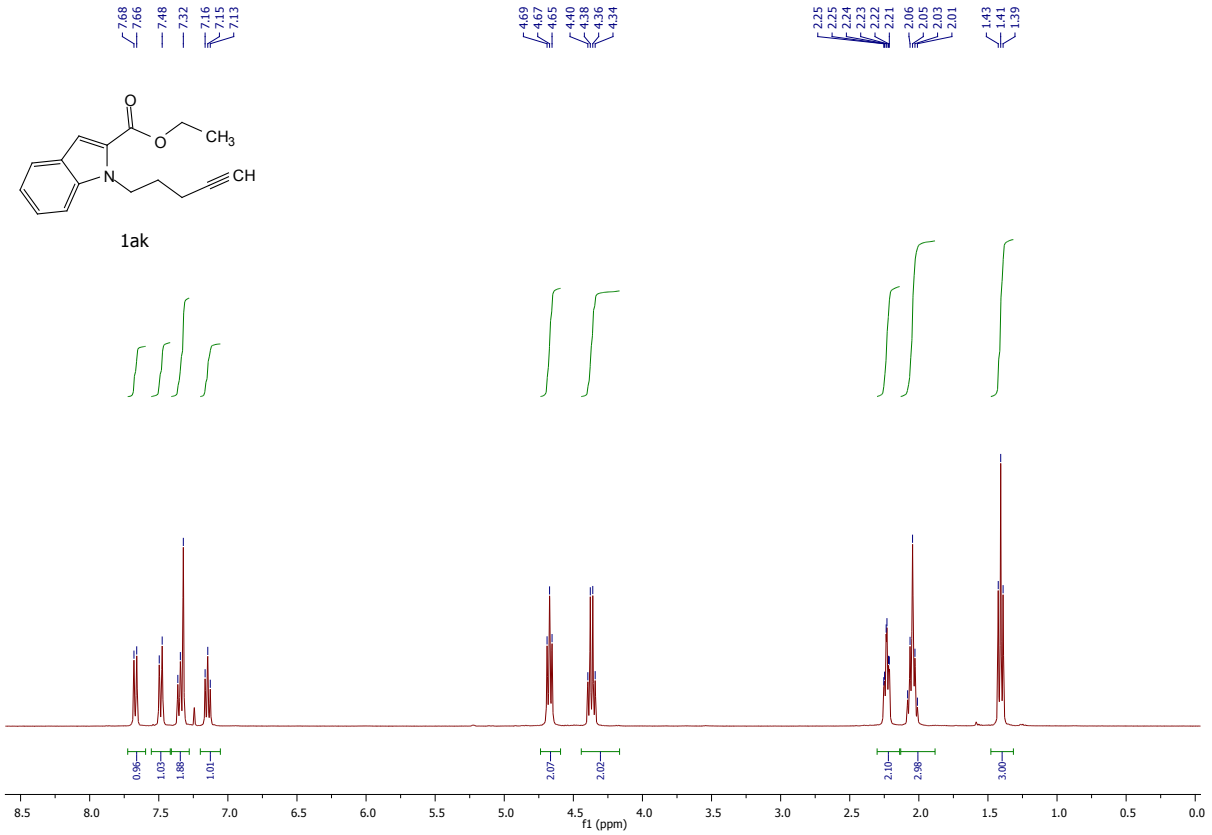
122.93
122.82
117.31
110.05
82.63
O
CH3
N
CH
1aj
f1 (ppm)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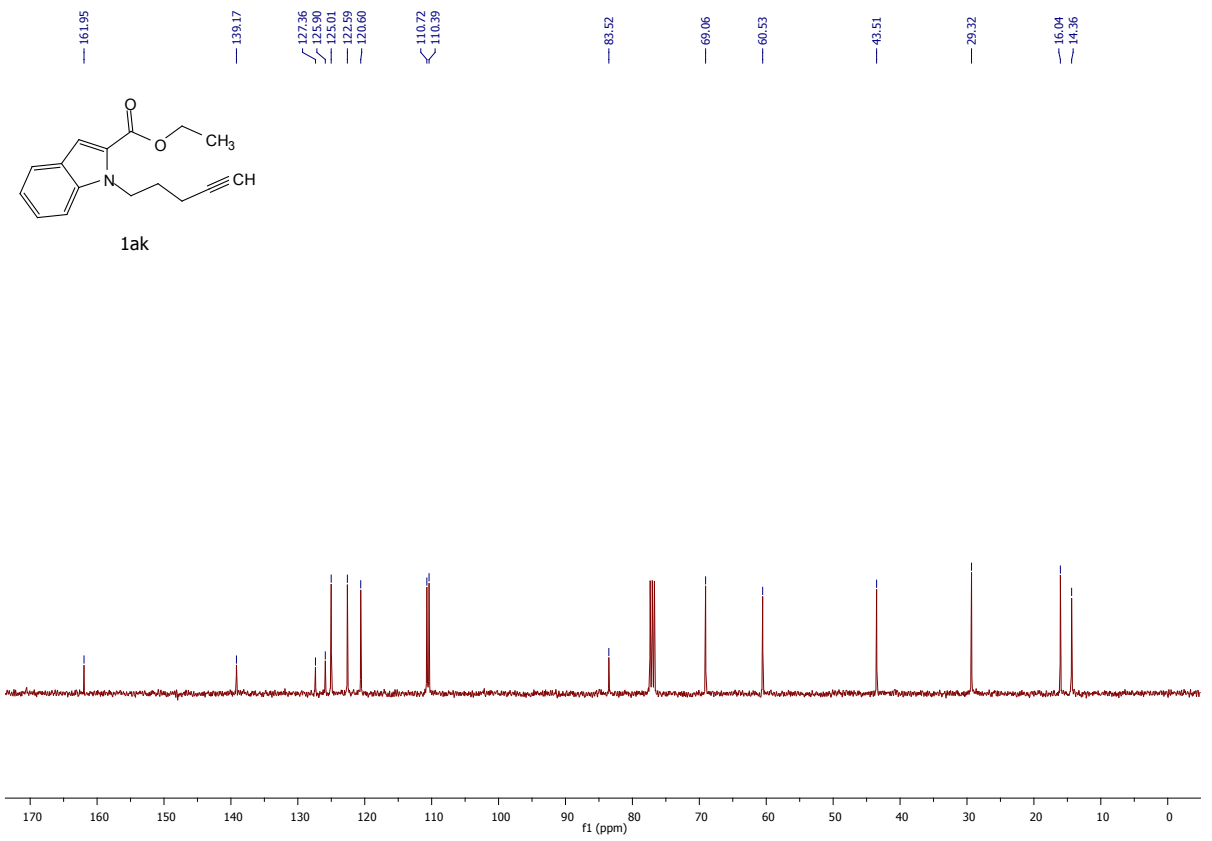
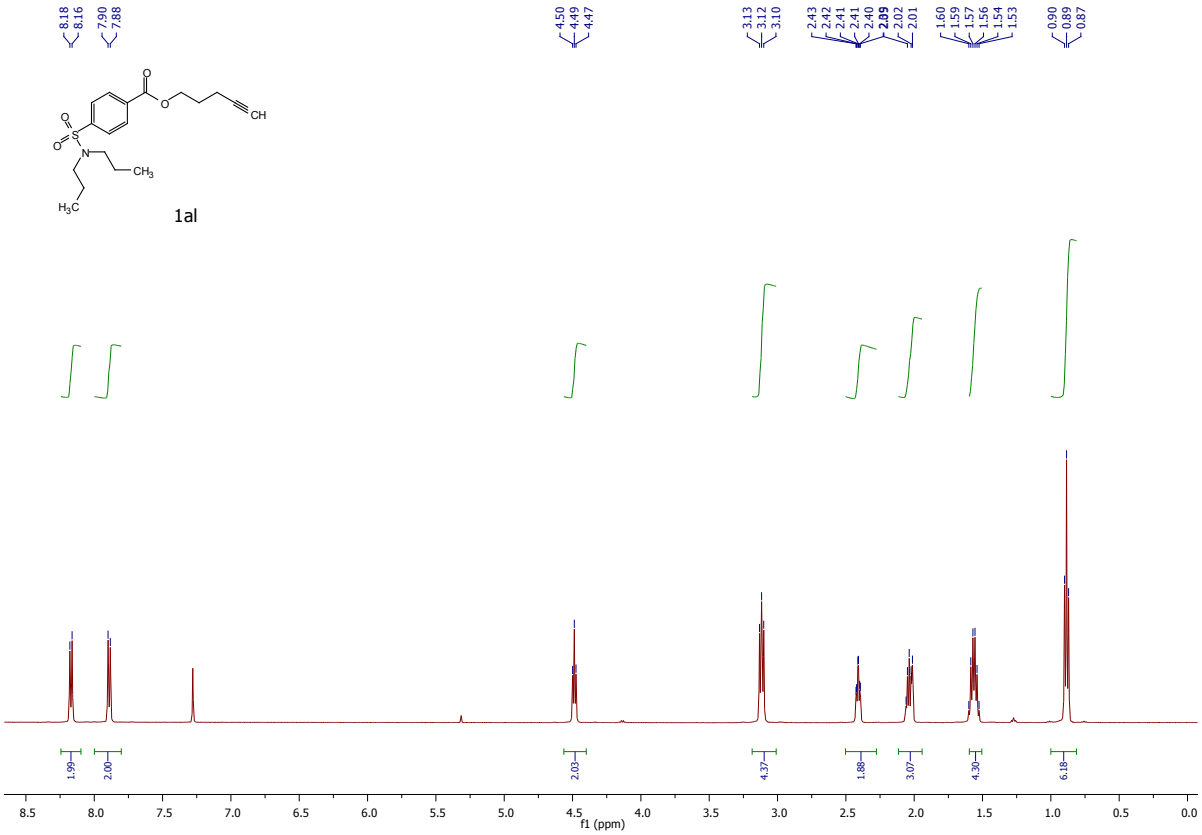
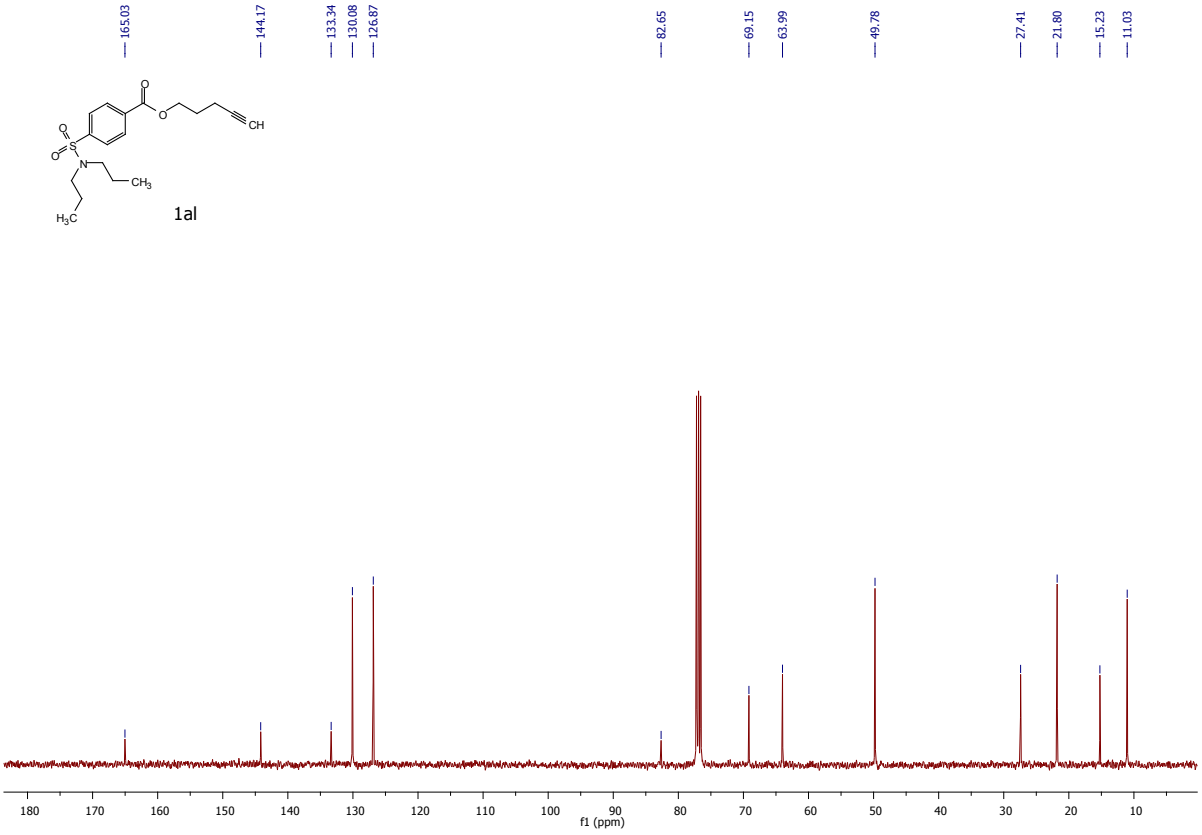
7.15
7.13
4.69
4.67
4.65
4.40
4.38
4.36
4.34
2.25
2.25
2.24
2.23
2.22
2.21
2.06
2.05
2.03
2.01
1.43
1.41
1.39
O
O
CH3
N
CH
0.96
1.03
1.88
1.01
2.07
2.02
2.10
2.98
3.00
f1 (ppm)

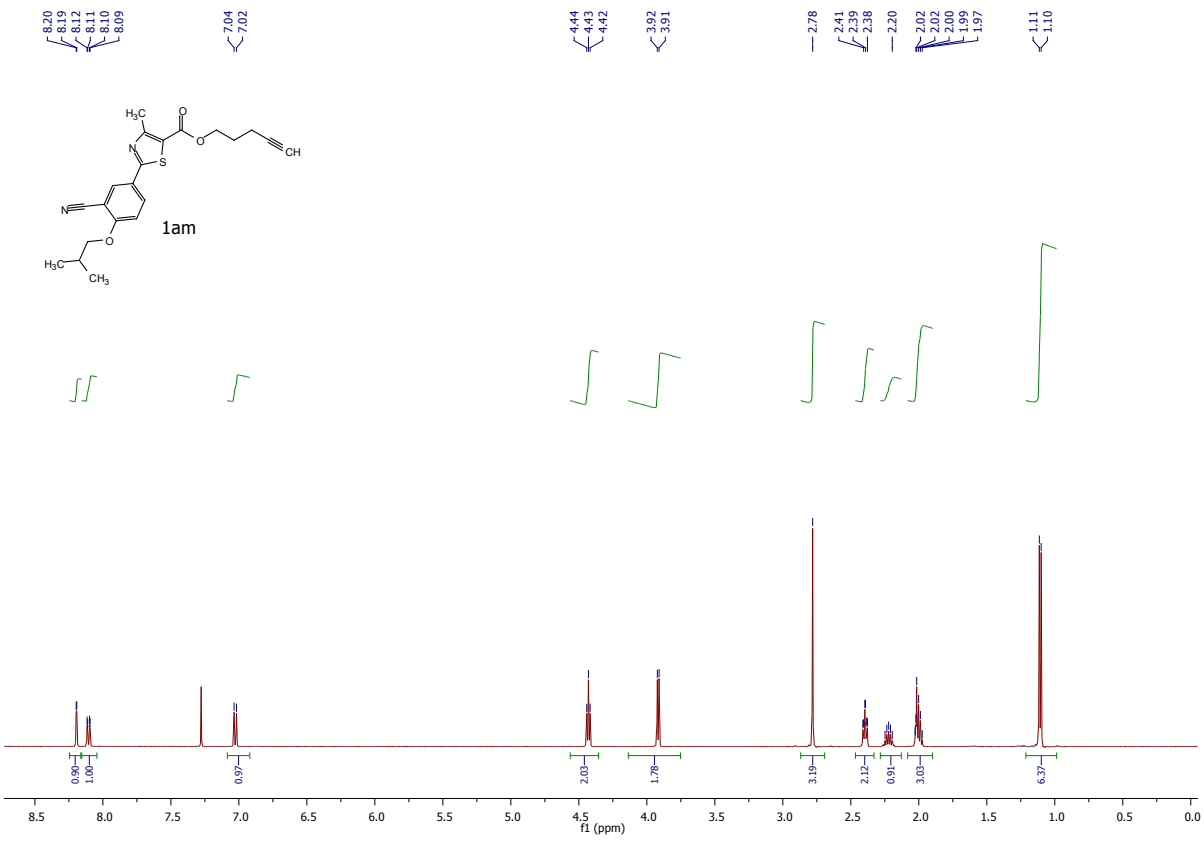
1ak

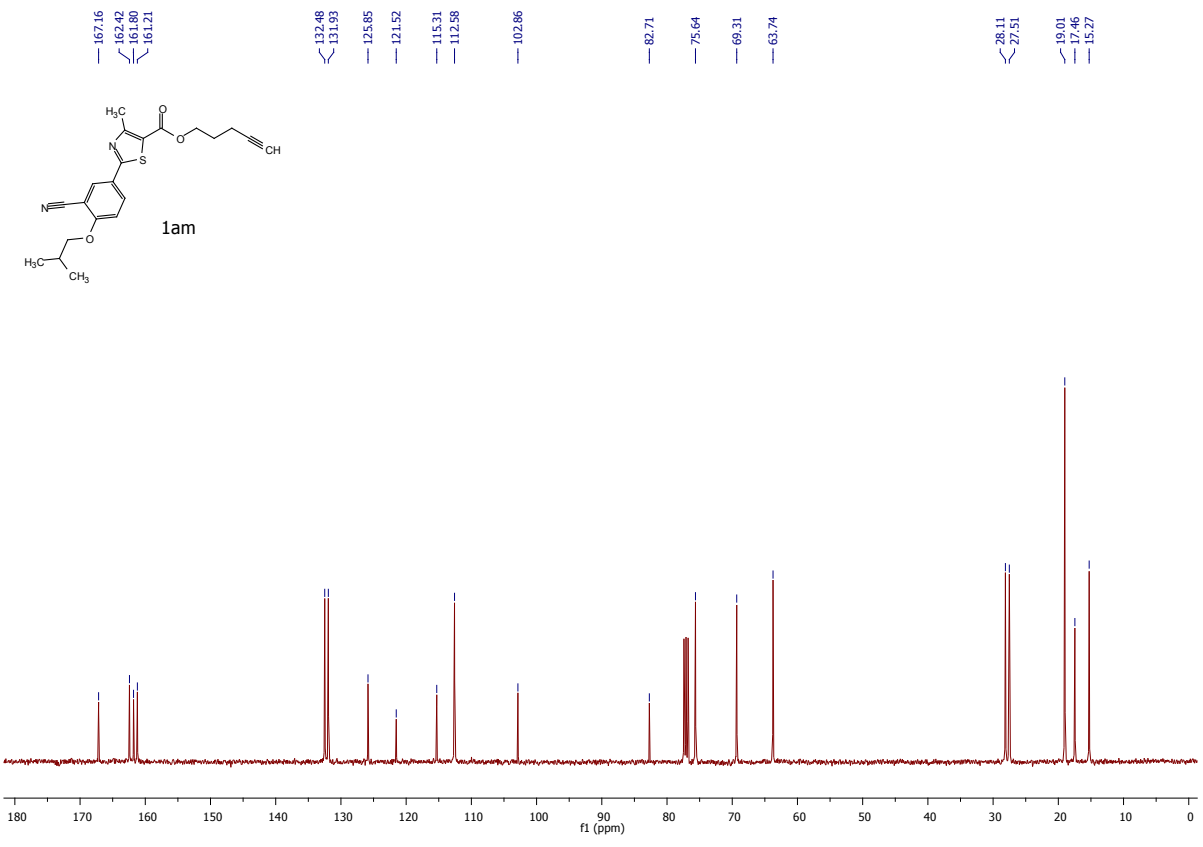

161.95
139.17
127.36
125.90
125.01
122.59
120.60
110.72
110.39
83.52
69.06
60.53
43.51
29.32
16.04
14.36
O
O
CH3
N
CH
f1 (ppm)

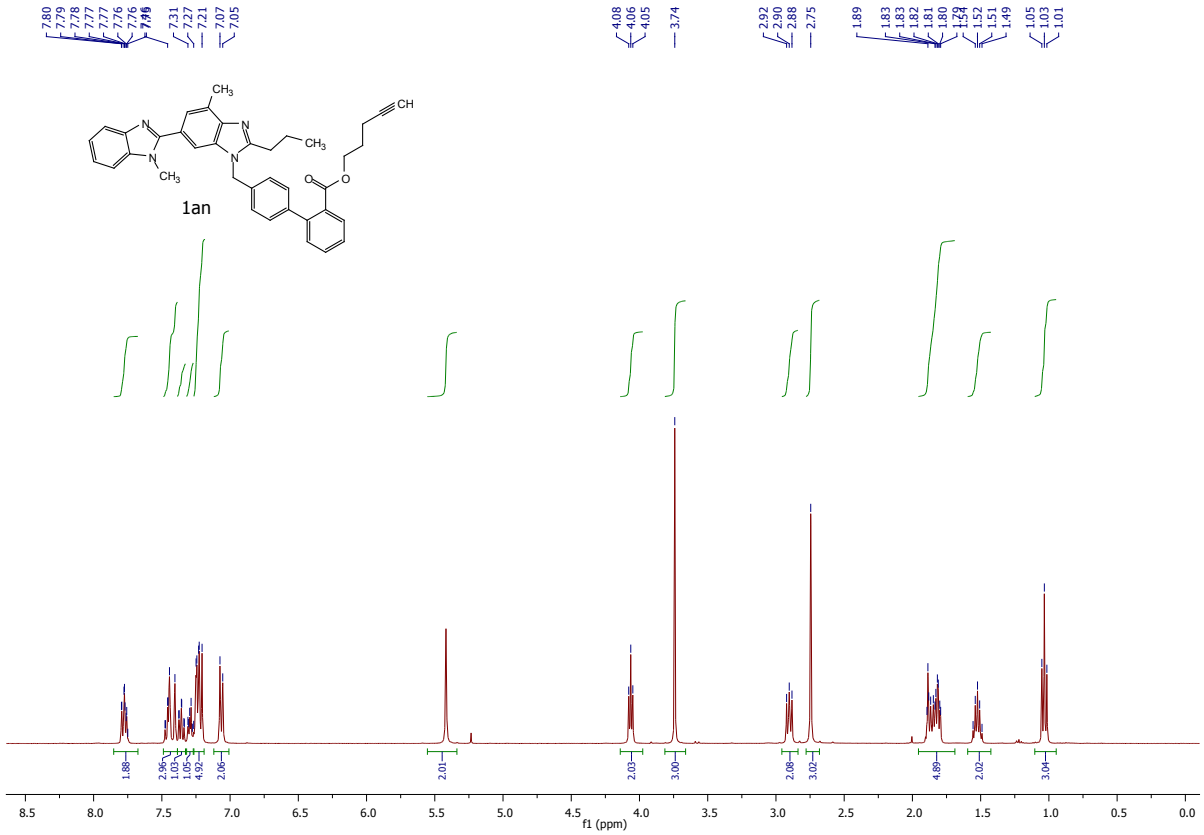
1ak

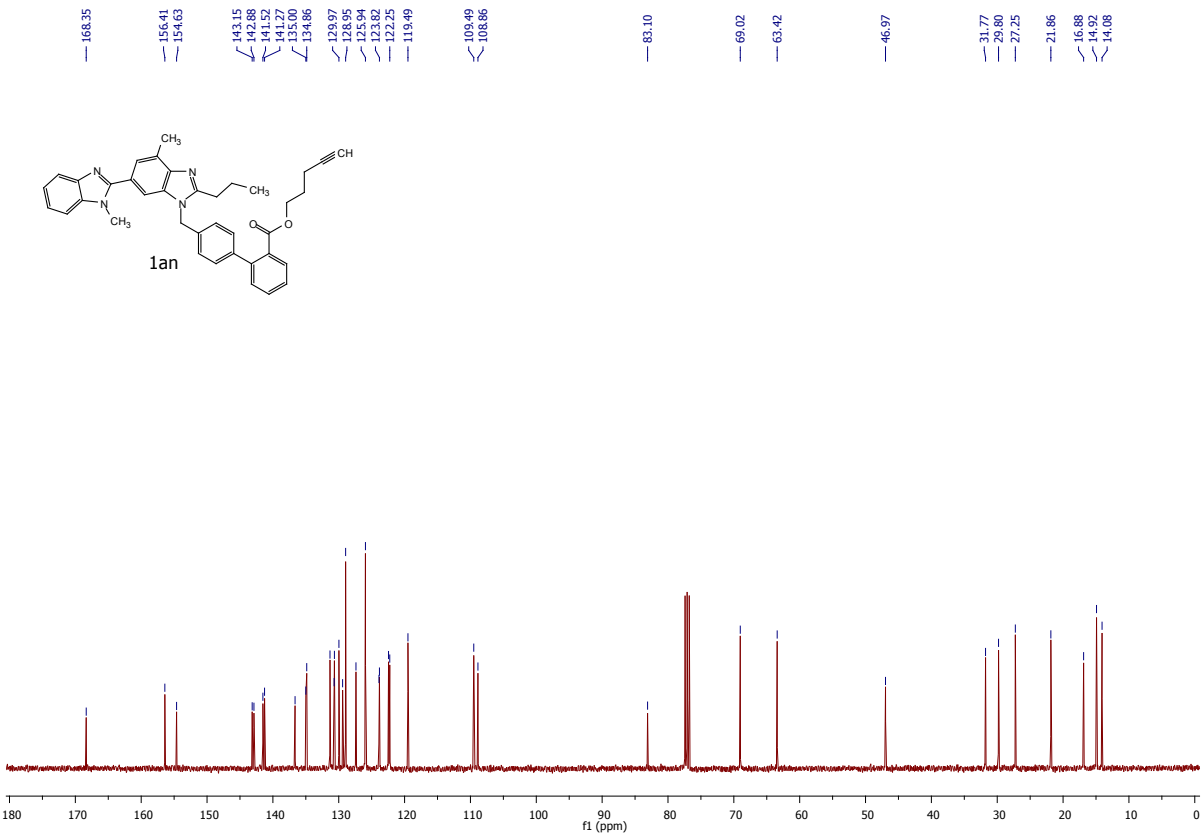

8.18
8.16
7.90
7.88
4.50
4.49
4.47
3.13
3.12
3.10
2.43
2.42
2.41
2.41
2.40
2.05
2.02
2.01
1.60
1.59
1.57
1.56
1.54
1.53
0.90
0.89
0.87
1al
1.99
2.00
2.03
4.37
1.88
3.07
4.30
6.18
f1 (ppm)

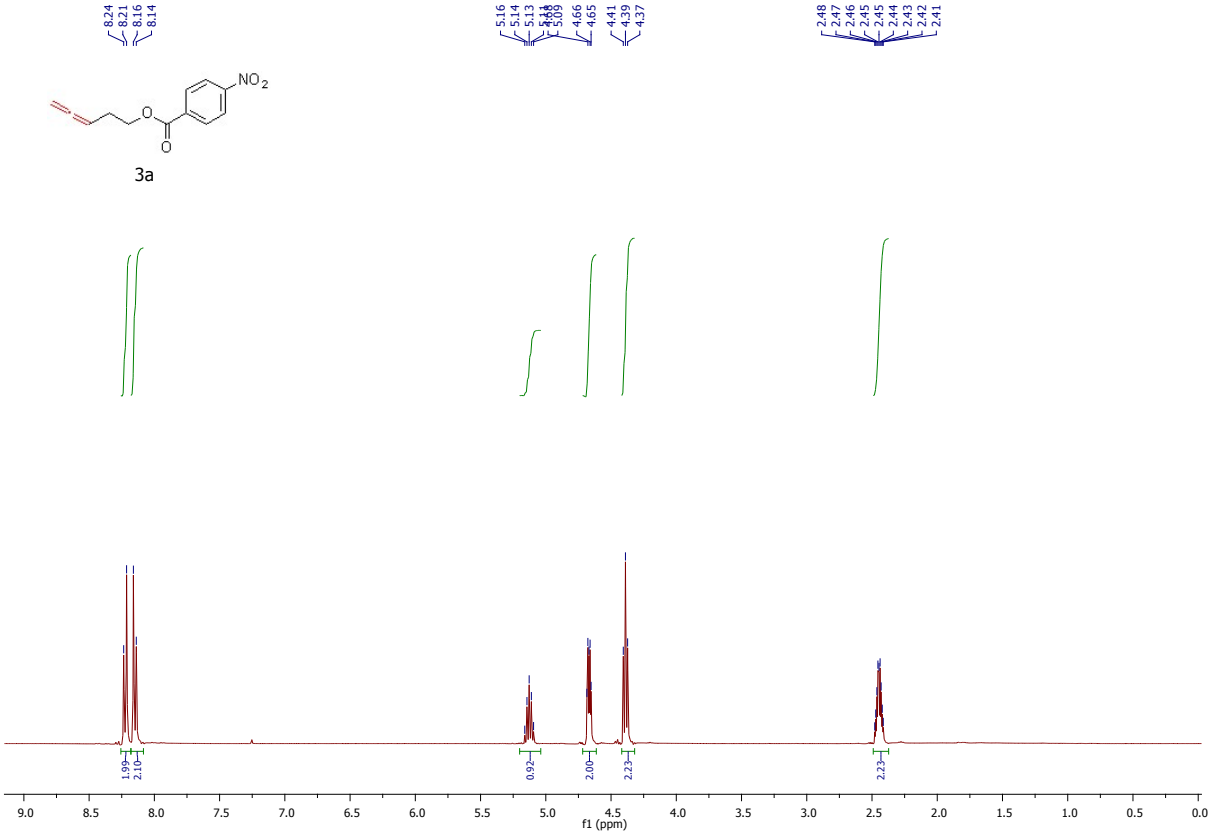

165.03
144.17
133.34
130.08
126.87
82.65
69.15
63.99
49.78
27.41
21.80
15.23
11.03
1al
f1 (ppm)


8.20
8.19
8.12
8.11
8.10
8.09
7.04
7.02
4.44
4.43
4.42
3.92
3.91
2.78
2.41
2.39
2.38
2.20
2.02
2.02
2.00
1.99
1.97
1.11
1.10
1am
0.90
1.00
0.97
2.03
1.78
3.19
2.12
0.91
3.03
6.37
f1 (ppm)

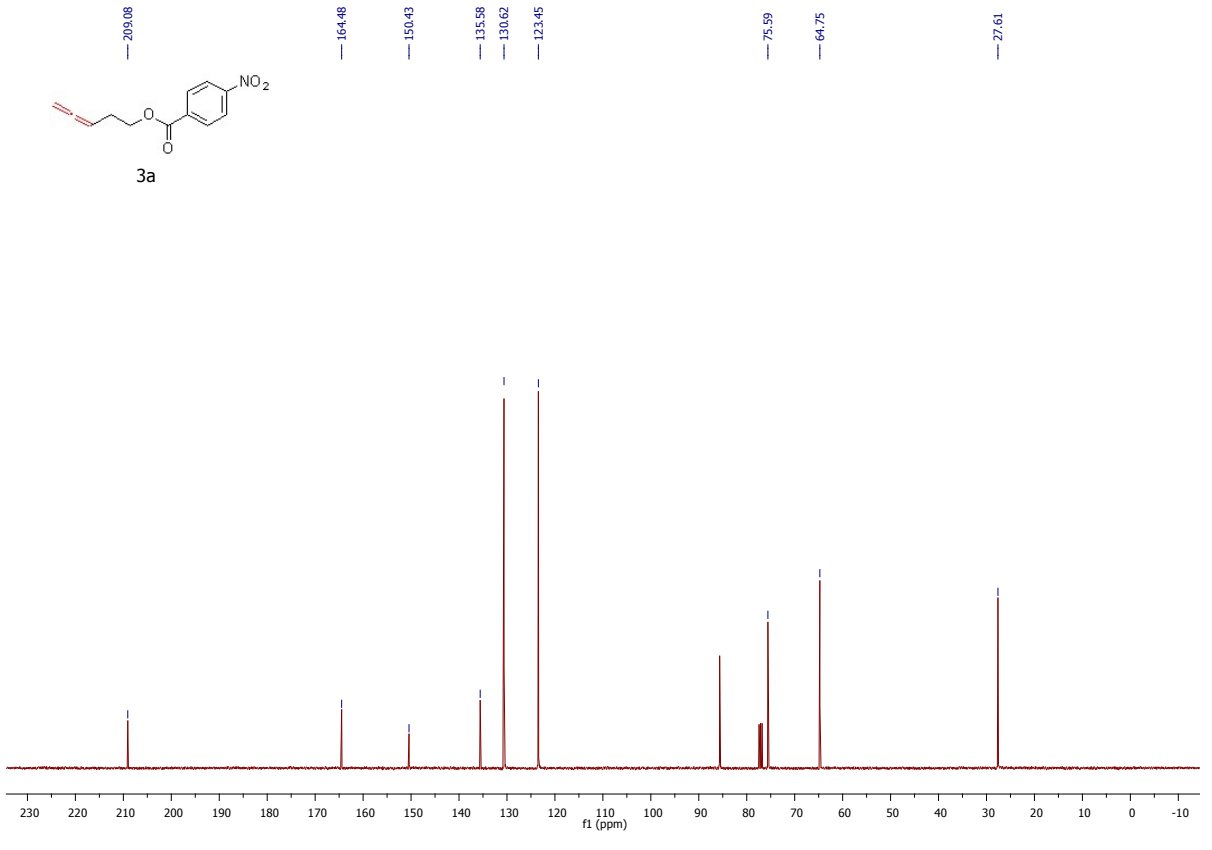

167.16
162.42
161.80
161.21
132.48
131.93
125.85
121.52
115.31
112.58
102.86
82.71
75.64
69.31
63.74
28.11
27.51
19.01
17.46
15.27
1am
f1 (ppm)


1an
f1 (ppm)

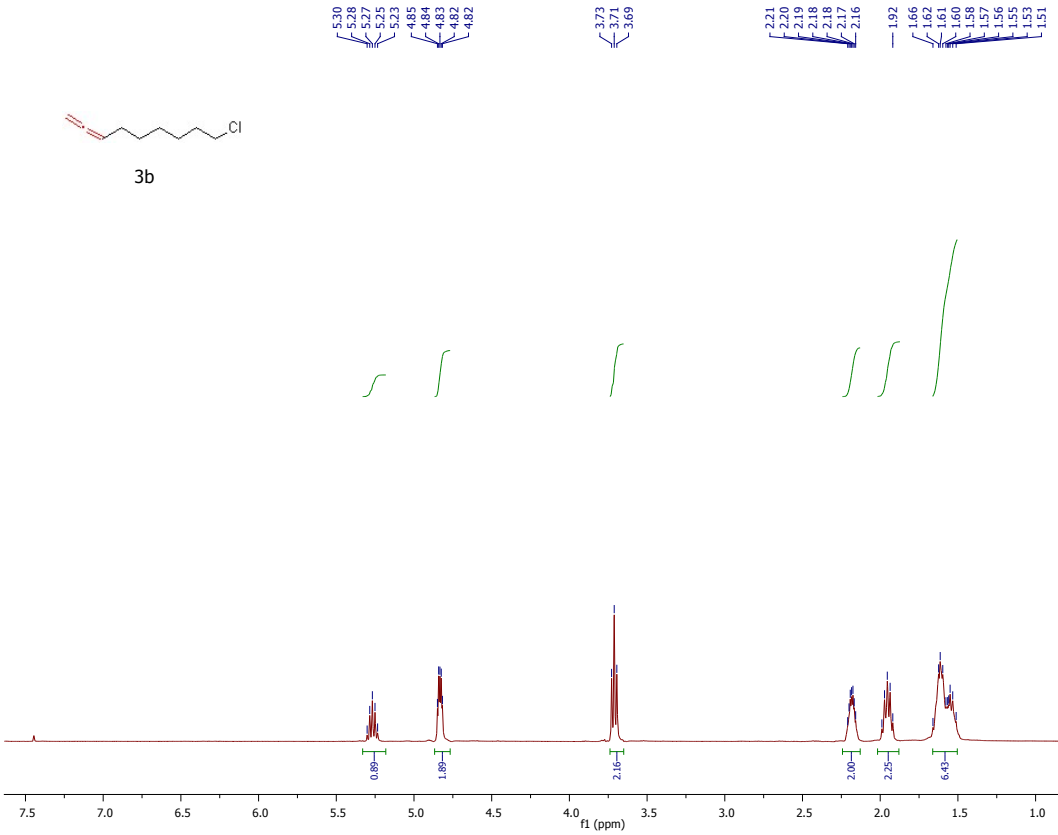

1an
f1 (ppm)

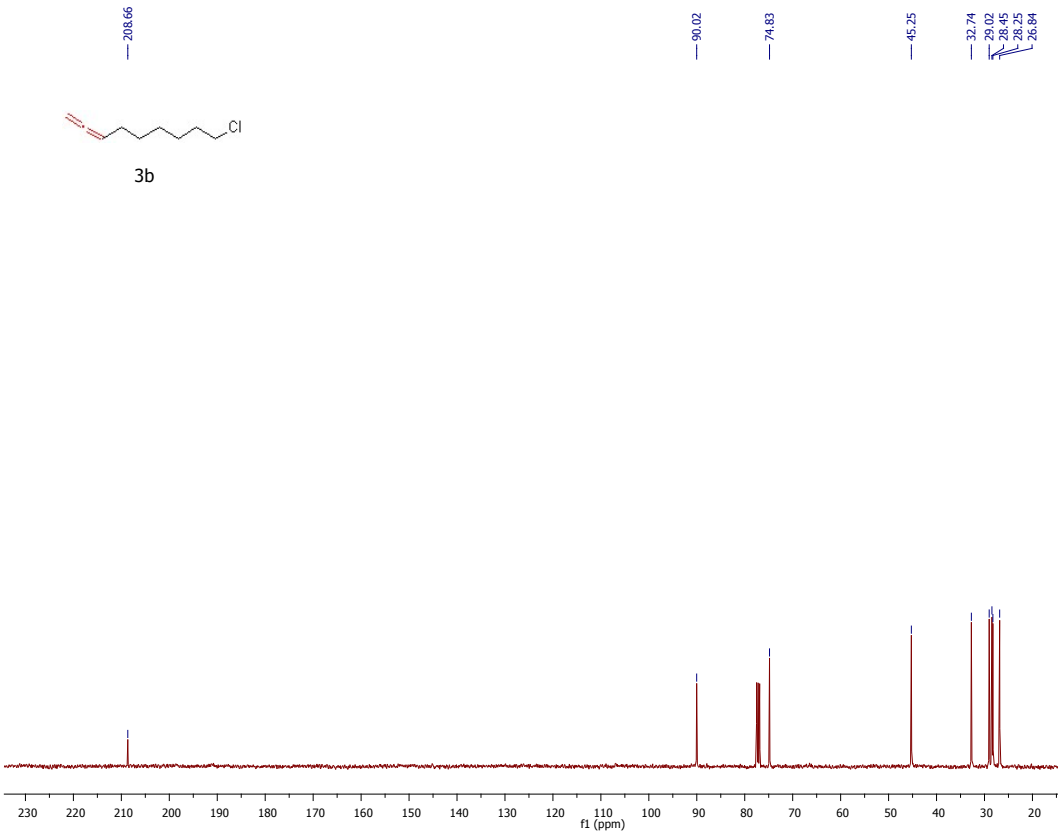

8.24
8.21
8.16
8.14
5.16
5.14
5.13
4.66
4.65
4.41
4.39
4.37
2.48
2.47
2.46
2.45
2.45
2.44
2.43
2.42
2.41
NO2
O
O
1.99
2.10
0.92
2.00
2.23
2.23
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

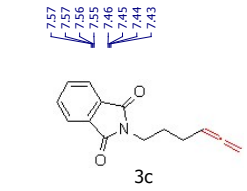
3a


209.08
164.48
150.43
135.58
130.62
123.45
75.59
64.75
27.61
NO2
O
O
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

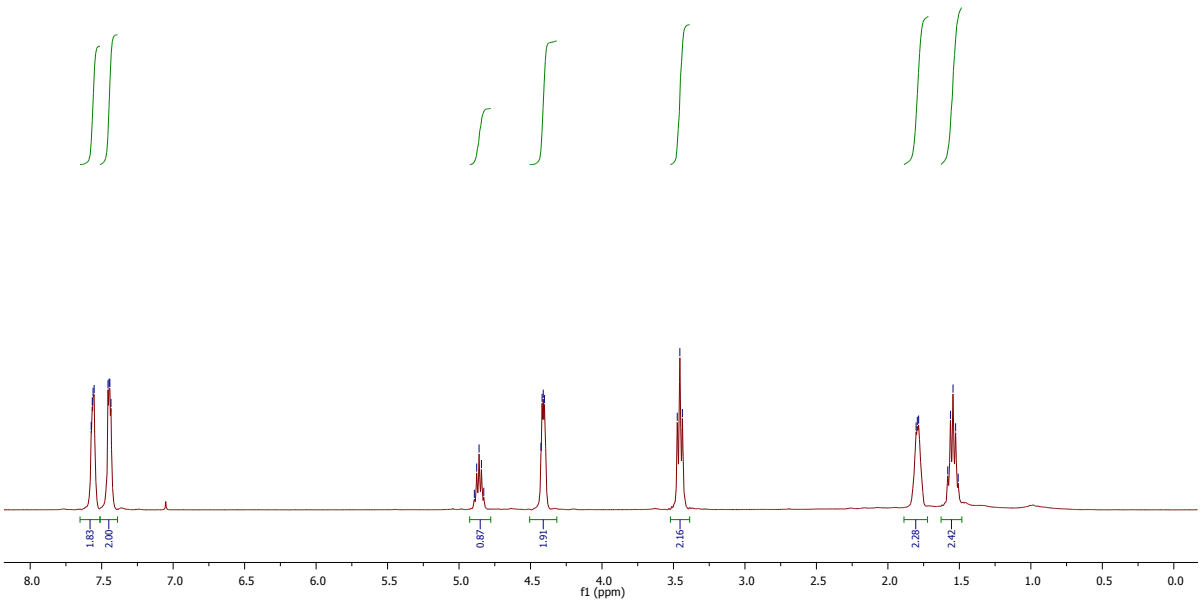
3a


5.30
5.28
5.27
5.25
5.23
4.85
4.84
4.83
4.82
4.82
3.73
3.71
3.69
2.21
2.20
2.19
2.18
2.18
2.17
2.16
1.92
1.66
1.62
1.61
1.60
1.58
1.57
1.56
1.55
1.53
1.51
Cl
3b
0.89
1.89
2.16
2.00
2.25
6.43
f1 (ppm)

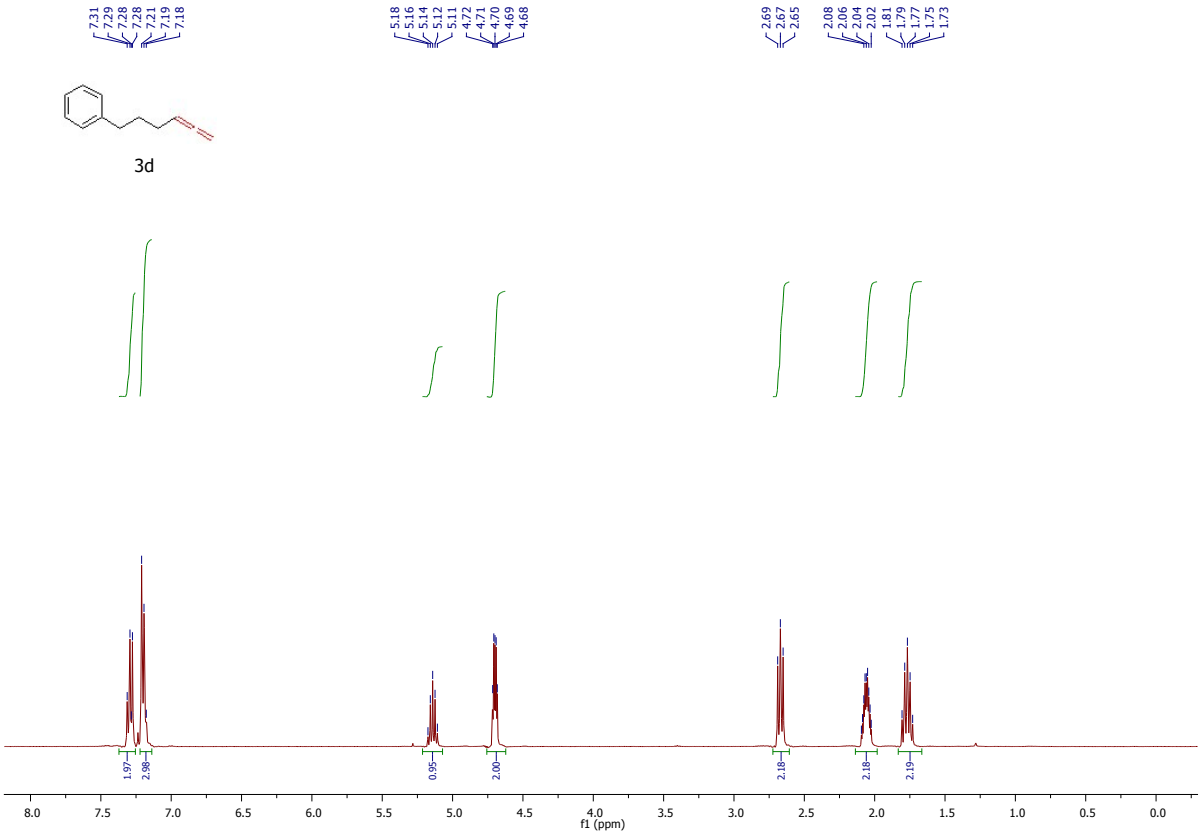

208.66
90.02
74.83
45.25
32.74
29.02
28.45
28.25
26.84
Cl
3b
f1 (ppm)

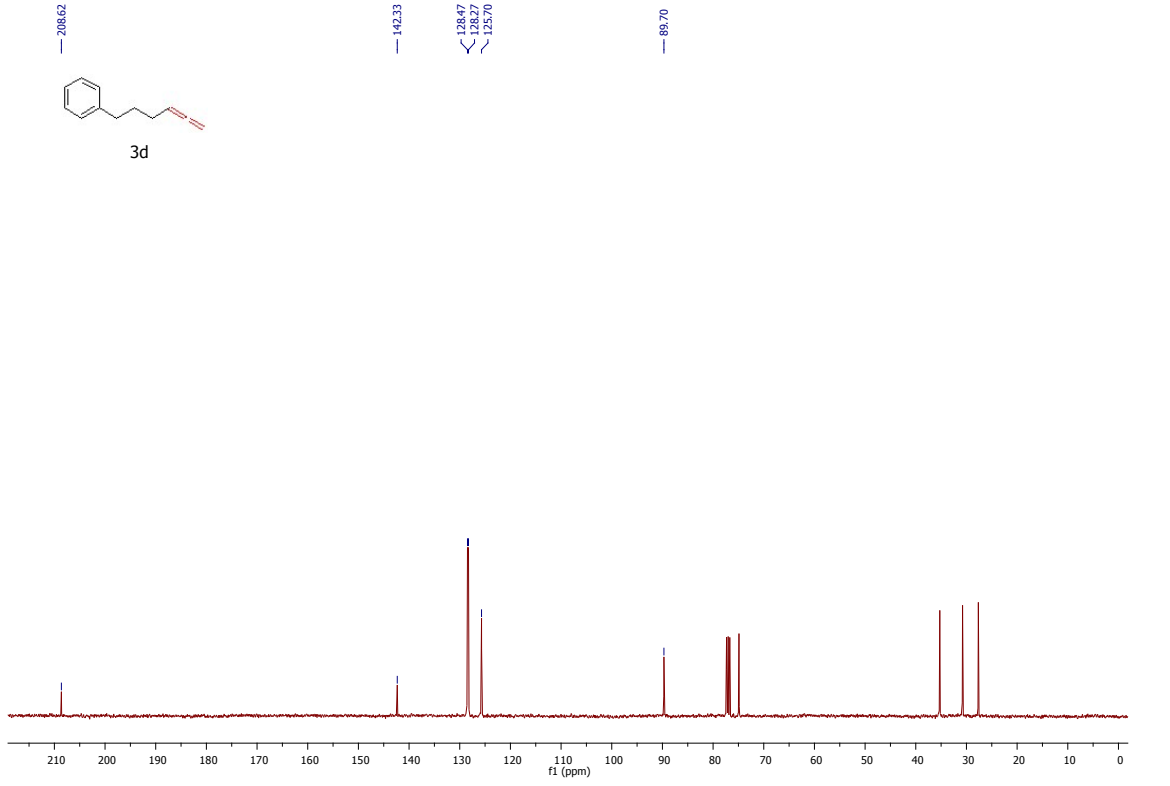


3c

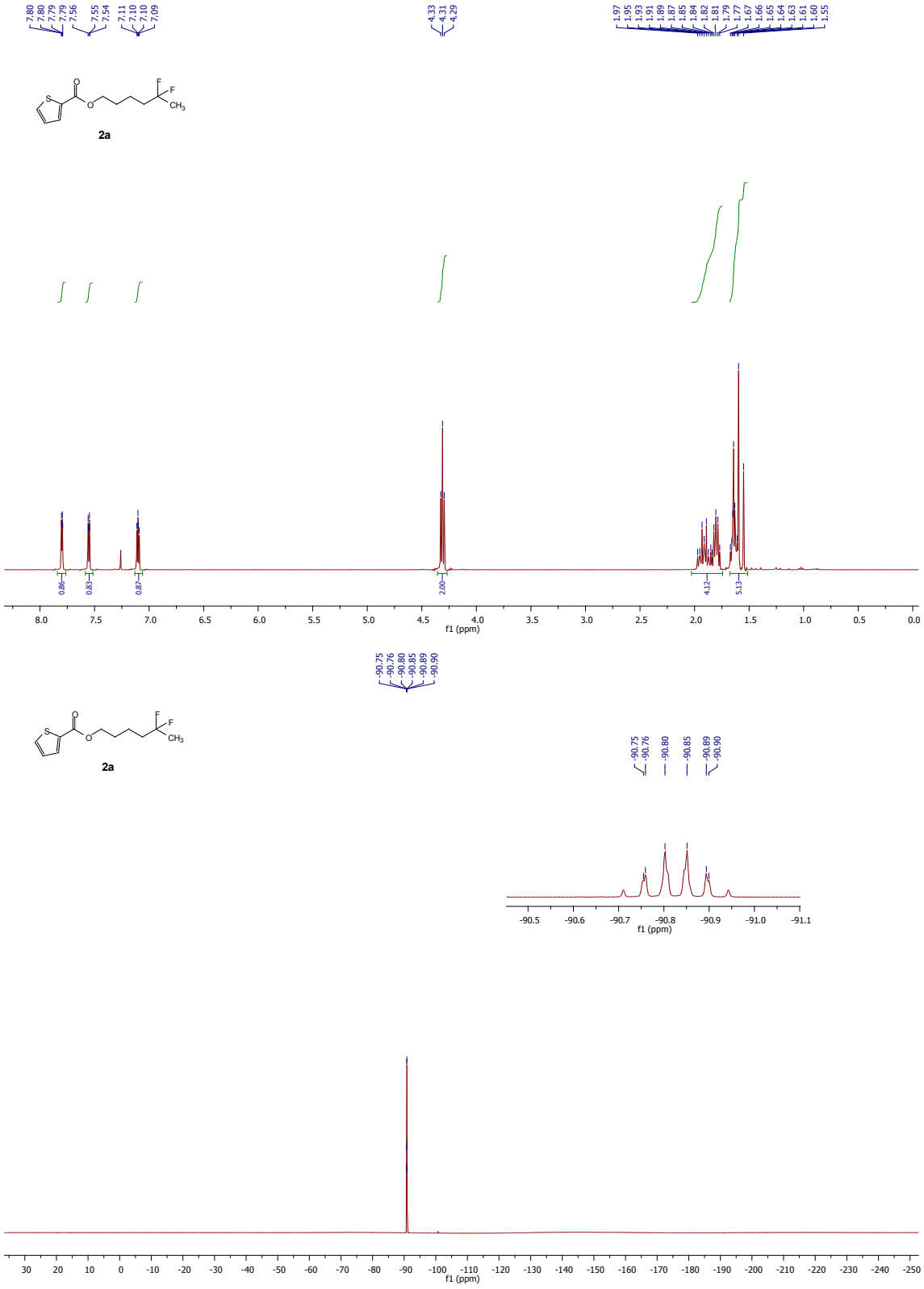


3c



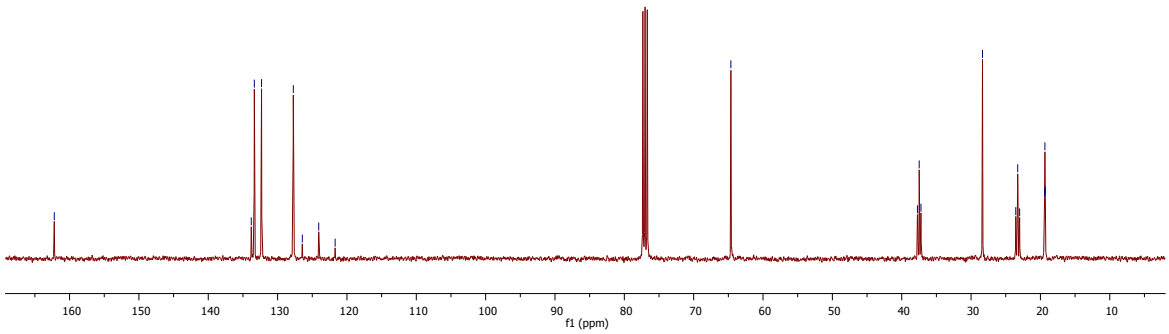


4.2 Products NMR spectra.

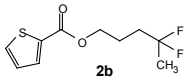

7.80
7.80
7.79
7.79
7.56
7.55
7.54
7.11
7.10
7.10
7.09
4.33
4.31
4.29
1.97
1.95
1.93
1.91
1.89
1.87
1.85
1.84
1.82
1.81
1.79
1.77
1.67
1.66
1.65
1.64
1.63
1.61
1.60
1.55
2a
0.86
0.83
0.87
2.00
4.12
5.13
f1 (ppm)
-90.75
-90.76
-90.80
-90.85
-90.89
-90.90
2a
f1 (ppm)
f1 (ppm)


162.20
133.80
133.36
132.32
127.74
126.44
124.07
121.70
64.63
37.73
37.48
37.22
28.35
23.28
23.00
19.40
19.35
19.30

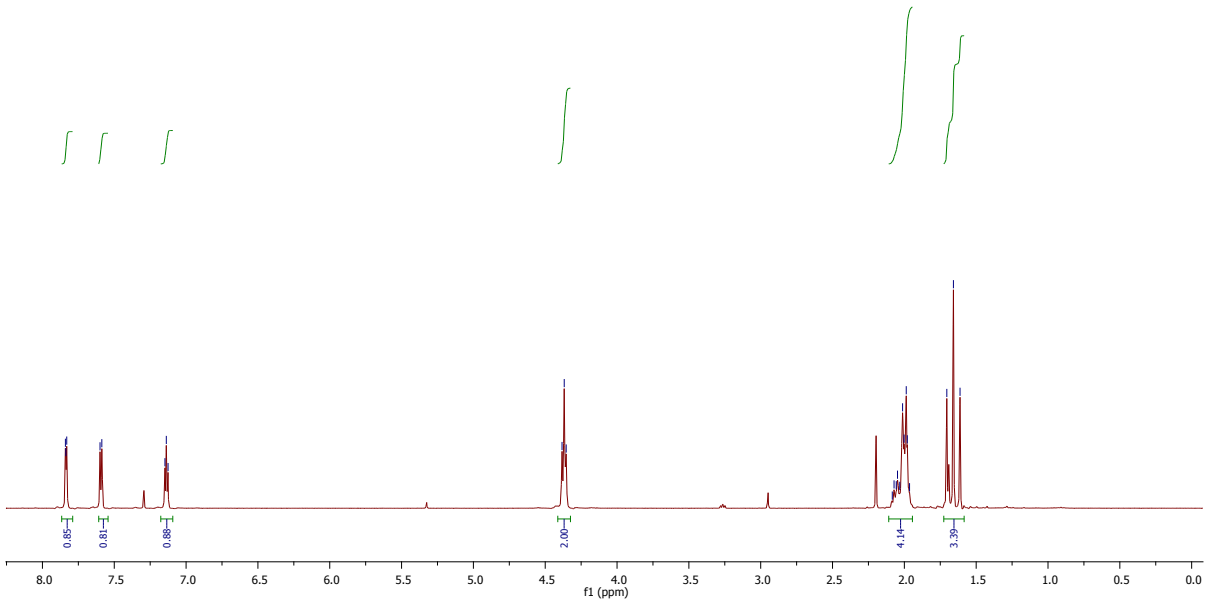
2a

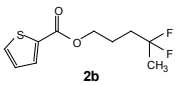

160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.840
7.837
7.831
7.599
7.587
7.147
7.138
7.126
4.383
4.368
4.354
2.084
2.072
2.056
2.048
2.032
2.012
2.002
1.987
1.980
1.964
1.704
1.658
1.612



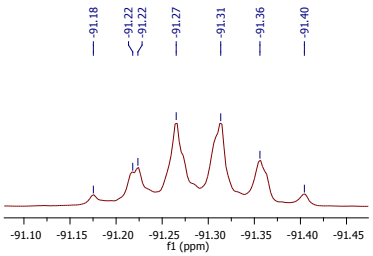
2b

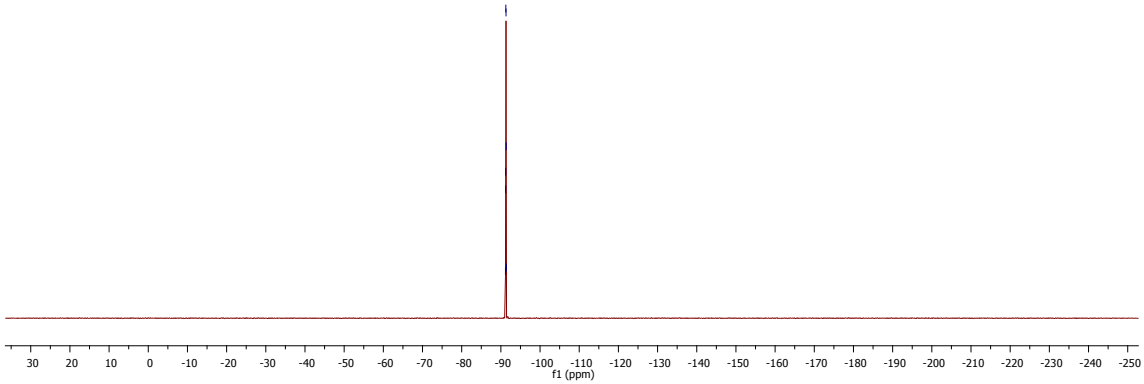

0.85
0.81
0.88
2.00
4.14
3.39
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


O
S
O
F
F
CH3

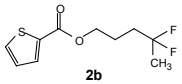
2b


-91.18
-91.22
-91.22
-91.27
-91.31
-91.36
-91.40

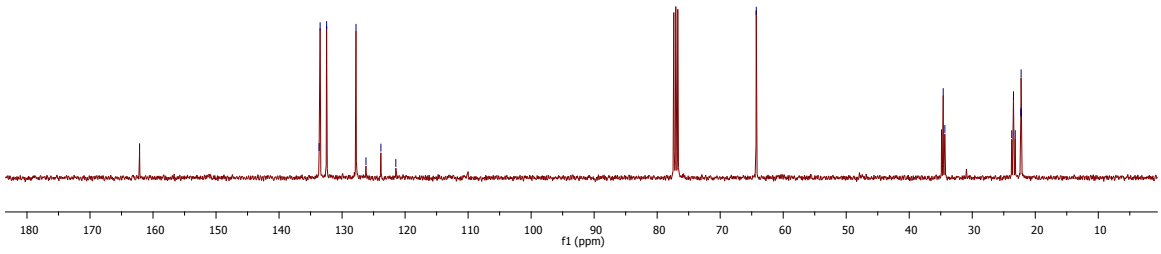

-91.18
-91.22
-91.22
-91.27
-91.31
-91.36
-91.40
-91.10 -91.15 -91.20 -91.25 -91.30 -91.35 -91.40 -91.45
f1 (ppm)

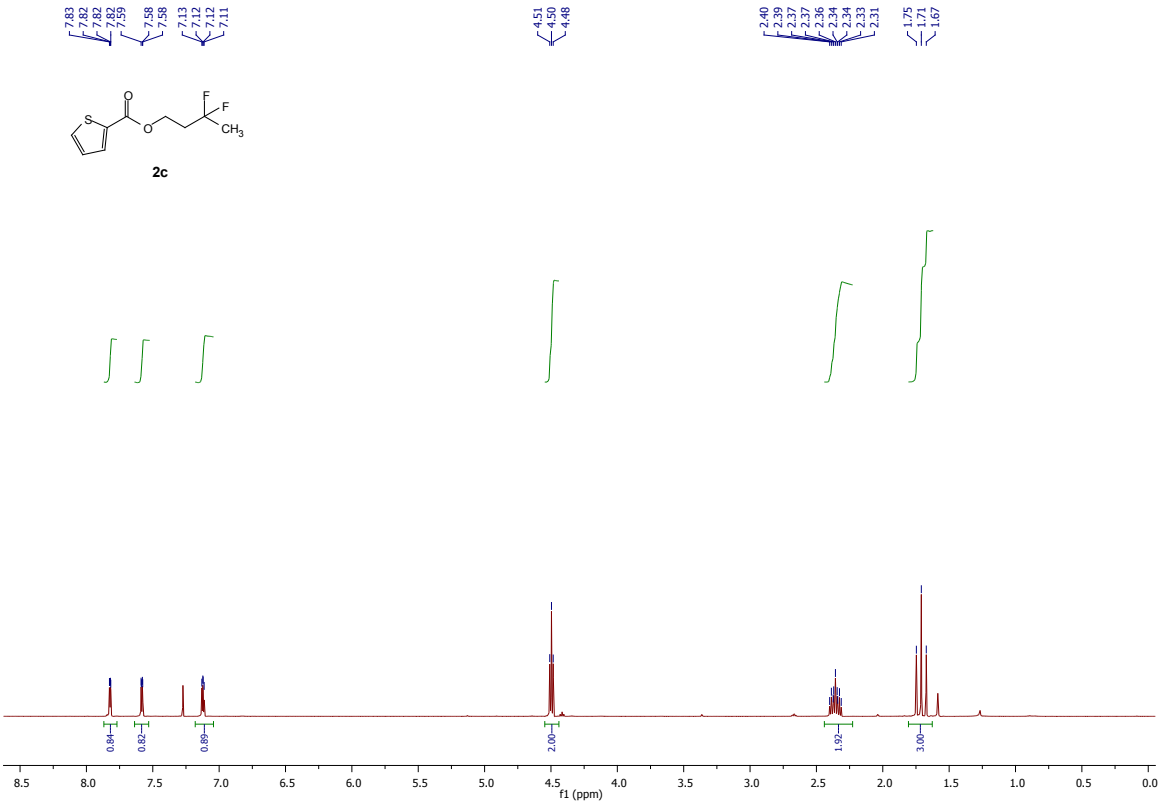

30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240 -250
f1 (ppm)

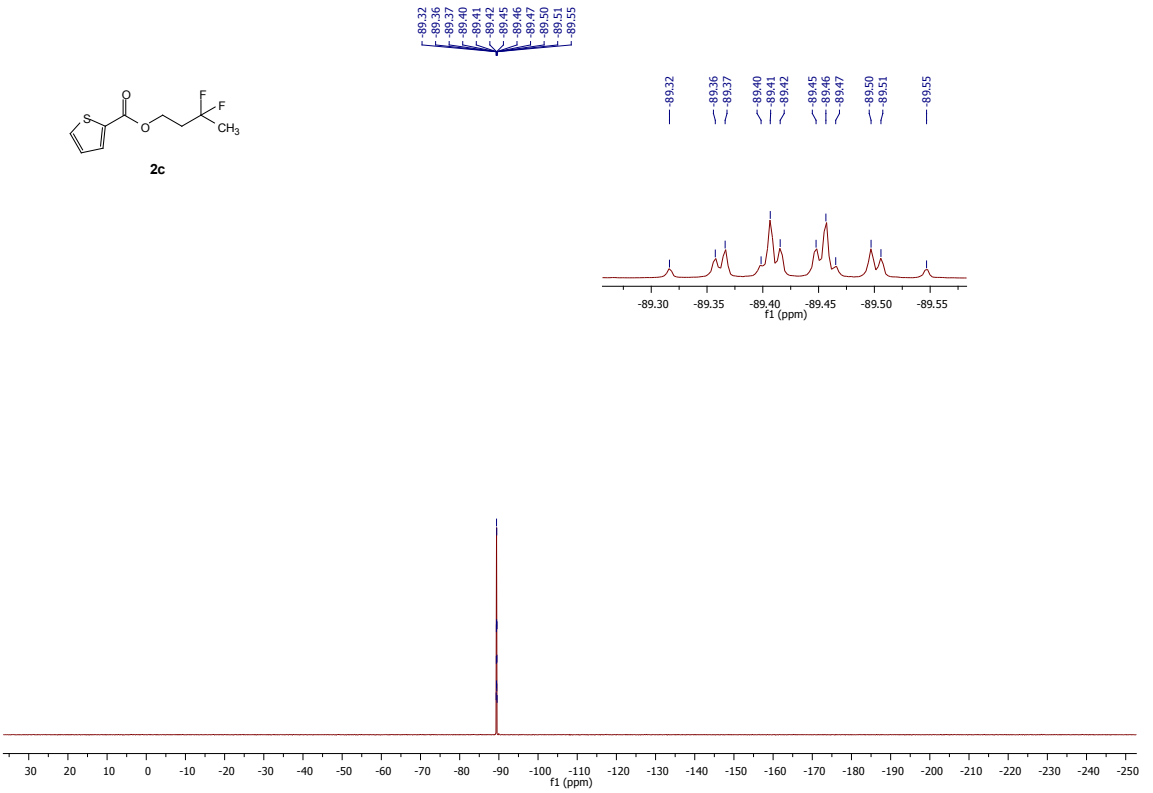

162.13
133.64
133.48
132.45
127.79
126.21
123.64
121.47
64.27
34.87
34.61
34.35
23.74
23.46
23.18
22.29
22.25
22.20

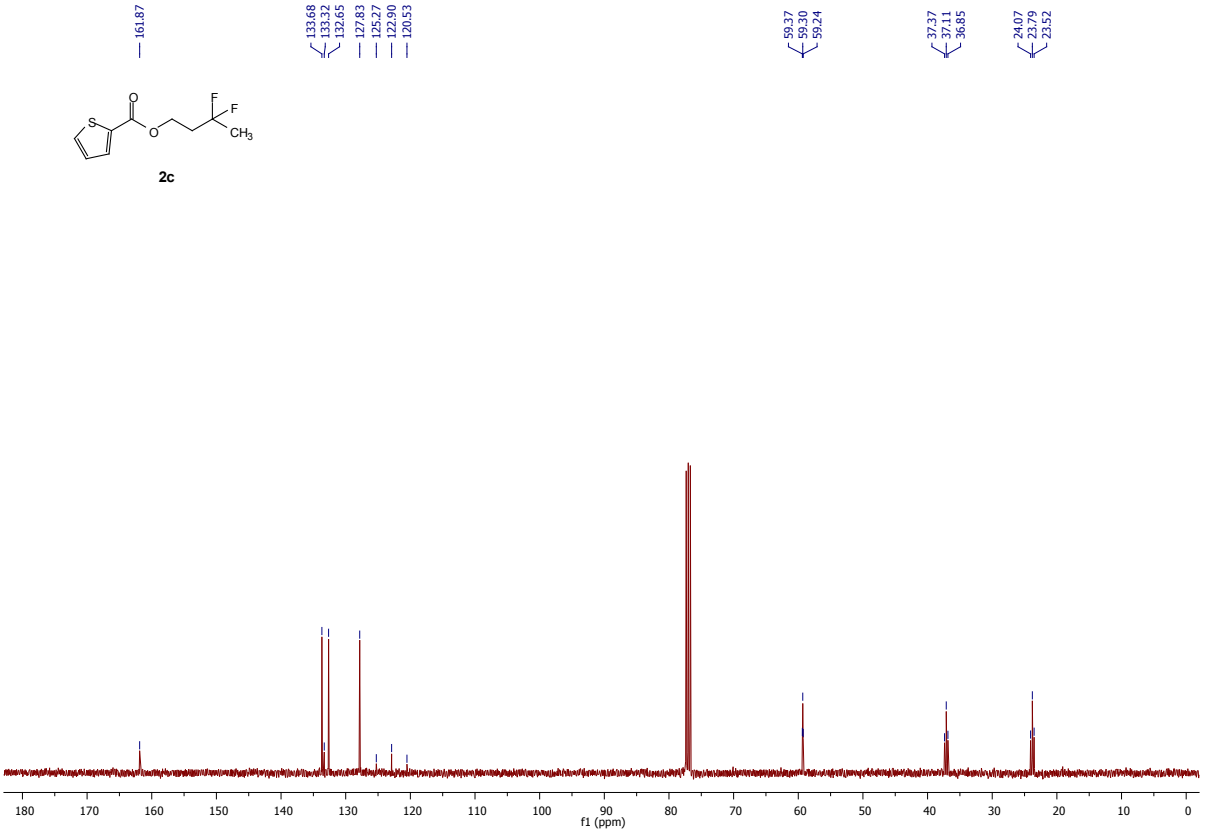

O
S
O
F
F
CH3

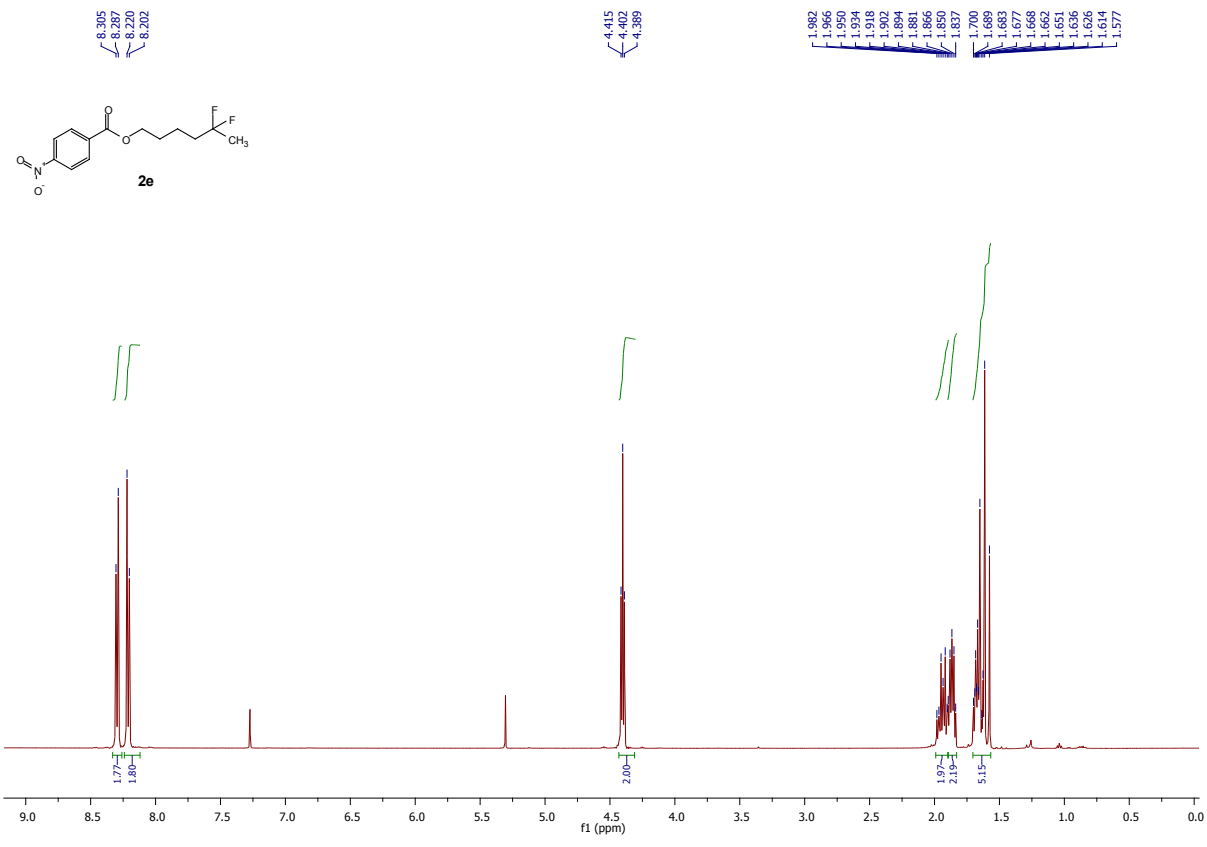
2b


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

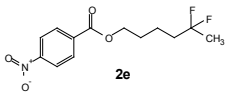

7.83
7.82
7.82
7.82
7.59
7.58
7.58
7.13
7.12
7.12
7.11
4.51
4.50
4.48
2.40
2.39
2.37
2.37
2.36
2.34
2.34
2.33
2.31
1.75
1.71
1.67
O
S
O
F
F
CH3
2c
0.84
0.82
0.89
2.00
1.92
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


-89.32
-89.36
-89.37
-89.40
-89.41
-89.42
-89.45
-89.46
-89.47
-89.50
-89.51
-89.55
O
S
O
F
F
CH3
2c
-89.30
-89.35
-89.40
-89.45
-89.50
-89.55
f1 (ppm)
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
-220
-230
-240
-250
f1 (ppm)

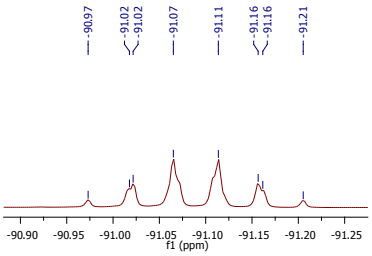

161.87
133.68
133.32
132.65
127.83
125.27
122.90
120.53
59.37
59.30
59.24
37.37
37.11
36.85
24.07
23.79
23.52
S
O
O
F
F
CH3
2c
f1 (ppm)

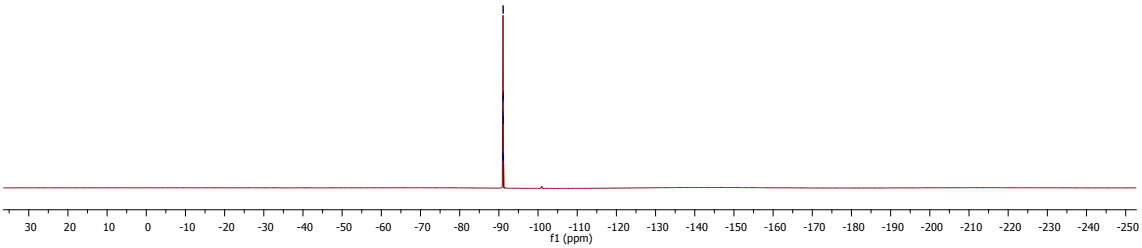

8.305
8.287
8.220
8.202
4.415
4.402
4.389
1.982
1.966
1.950
1.934
1.918
1.902
1.894
1.881
1.866
1.850
1.837
1.700
1.689
1.683
1.677
1.668
1.662
1.651
1.636
1.626
1.614
1.577
O
O
F
F
CH3
O
N
O
2e
1.77
1.80
2.00
1.97
2.19
5.15
f1 (ppm)


-90.97
-91.02
-91.02
-91.07
-91.11
-91.16
-91.16
-91.21

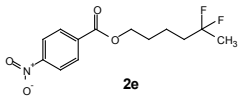

O
O
F
F
CH3
O
N
O
-

2e

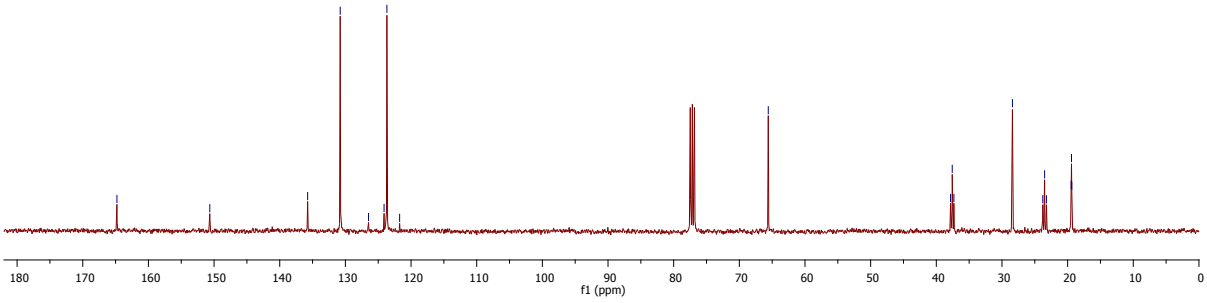

-90.97
-91.02
-91.02
-91.07
-91.11
-91.16
-91.16
-91.21
-90.90
-90.95
-91.00
-91.05
-91.10
-91.15
-91.20
-91.25
f1 (ppm)

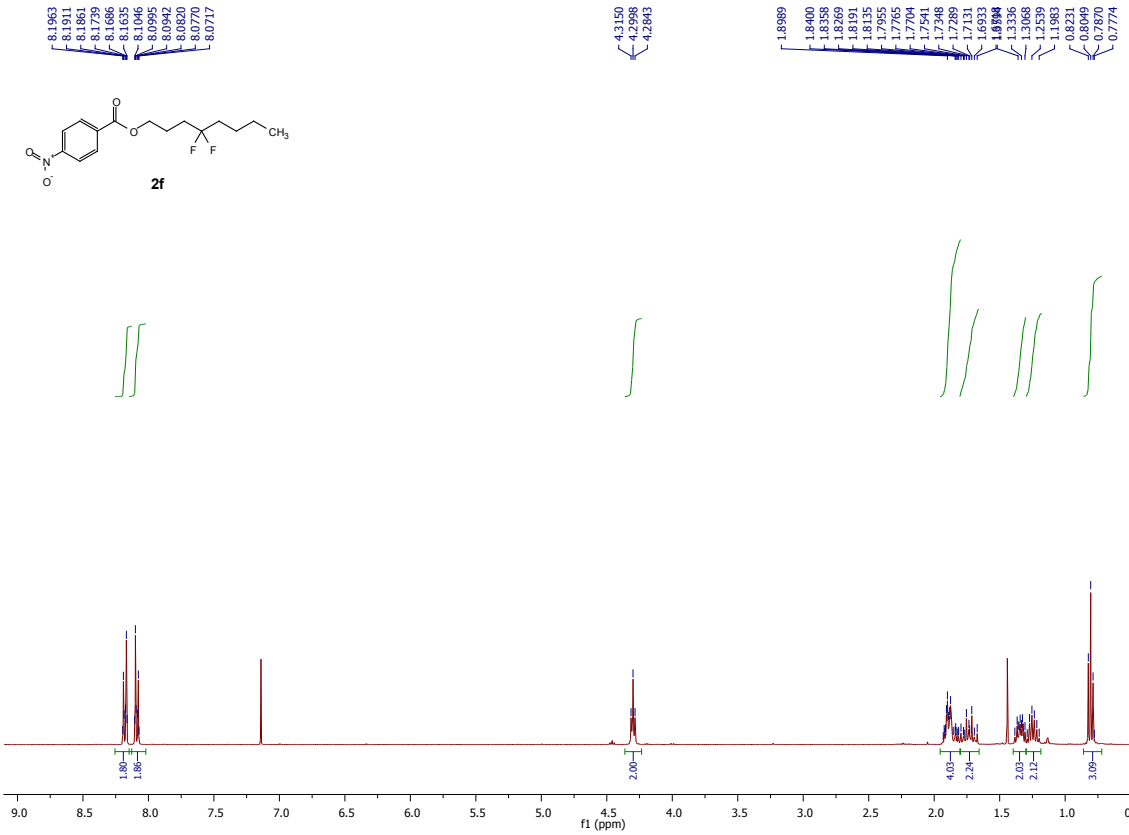

30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
-220
-230
-240
-250
f1 (ppm)

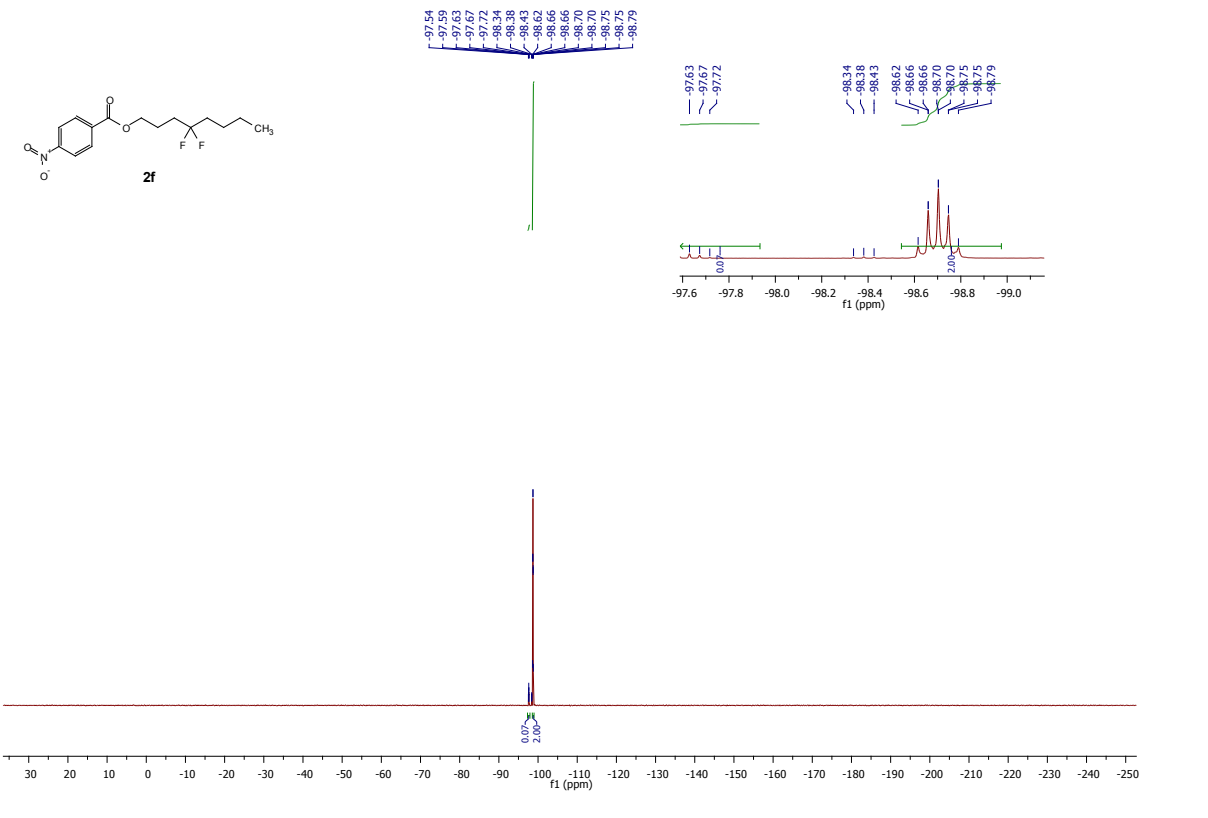

164.79
150.65
135.75
130.79
126.48
124.11
123.68
121.74
65.59
37.83
37.57
37.32
28.41
23.51
23.23
19.46
19.41
19.37

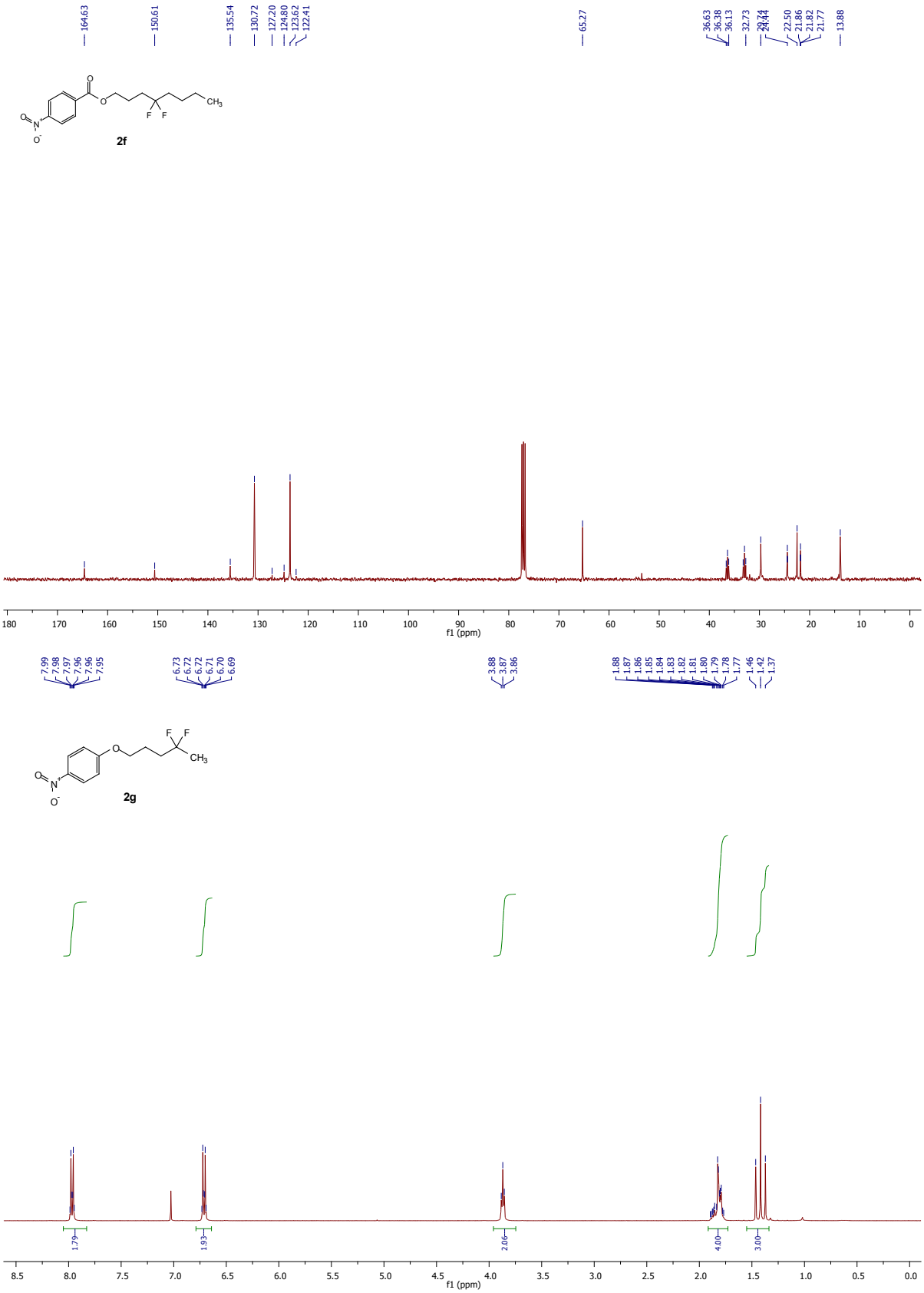

O
O
F
F
CH3
O
N
O
-

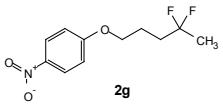
2e


180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

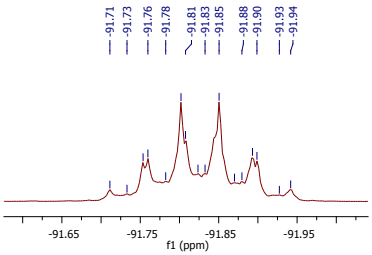

8.1963
8.1911
8.1861
8.1739
8.1686
8.1635
8.1046
8.0995
8.0942
8.0820
8.0770
8.0717
4.3150
4.2998
4.2843
1.8989
1.8400
1.8358
1.8269
1.8191
1.8135
1.7955
1.7765
1.7704
1.7541
1.7348
1.7289
1.7131
1.6933
1.3336
1.3068
1.2539
1.1983
0.8231
0.8049
0.7870
0.7774
2f
1.80
1.86
2.00
4.03
2.24
2.03
2.12
3.09
f1 (ppm)

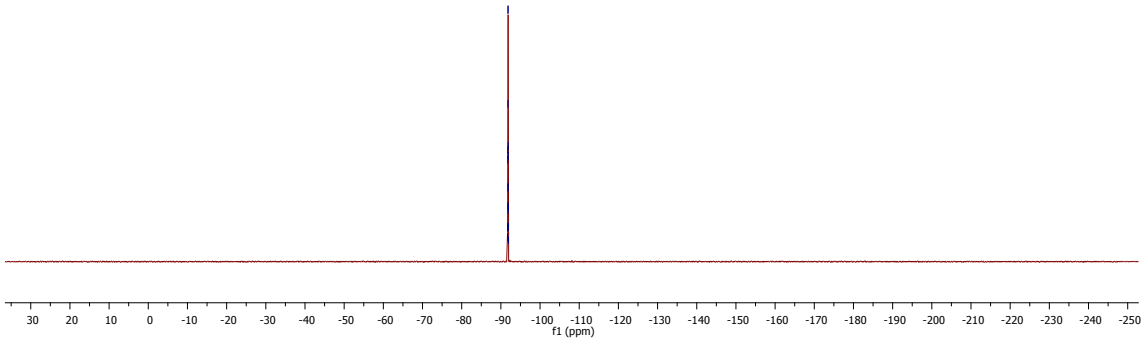

-97.54
-97.59
-97.63
-97.67
-97.72
-98.34
-98.38
-98.43
-98.62
-98.66
-98.66
-98.70
-98.70
-98.75
-98.75
-98.79
2f
0.07
2.00
f1 (ppm)


164.63
150.61
135.54
130.72
127.20
124.80
123.62
122.41
65.27
36.63
36.38
36.13
32.73
29.34
29.14
24.44
22.50
21.86
21.82
21.77
13.88
2f
f1 (ppm)
7.99
7.98
7.97
7.96
7.96
7.95
6.73
6.72
6.72
6.71
6.70
6.69
3.88
3.87
3.86
1.88
1.87
1.86
1.85
1.84
1.83
1.82
1.81
1.80
1.79
1.78
1.77
1.46
1.42
1.37
2g
1.79
1.93
2.00
4.00
3.00
f1 (ppm)

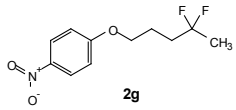

F
F
CH3
O
N+
O-
O

2g

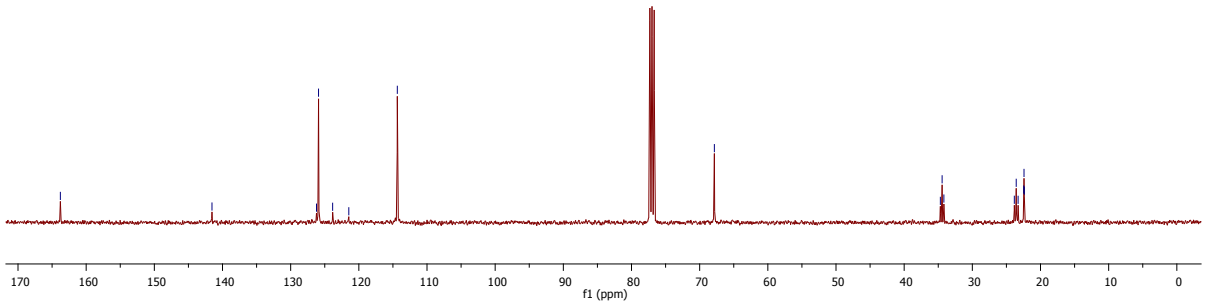

-91.71
-91.73
-91.76
-91.78
-91.81
-91.83
-91.85
-91.88
-91.90
-91.93
-91.94
-91.65
-91.75
-91.85
-91.95
f1 (ppm)


30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240 -250
f1 (ppm)


163.79
141.54
126.21
125.91
123.84
121.47
114.35
67.84
34.69
34.43
34.17
23.84
23.56
23.28
22.46
22.42
22.37

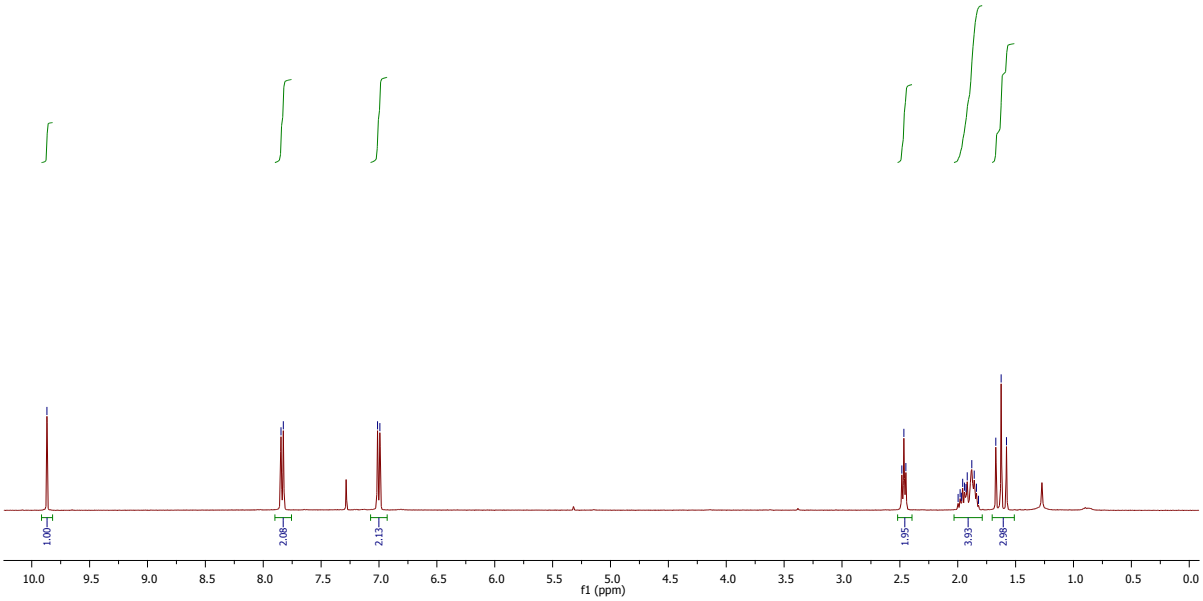

F
F
CH3
O
N+
O-
O

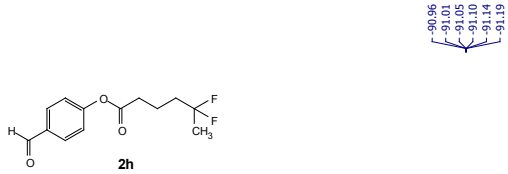
2g


170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

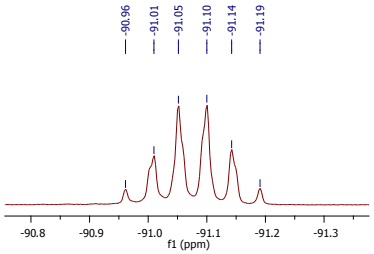

9.87
7.85
7.83
7.01
6.99
2.48
2.47
2.45
1.94
1.92
1.86
1.82
1.67
1.63
1.58
O
O
F
F
CH3
H
O

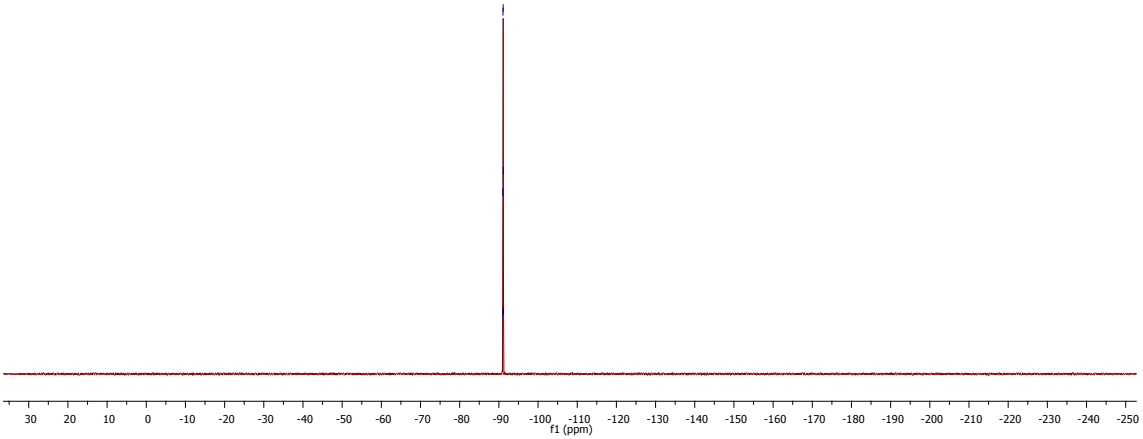
2h

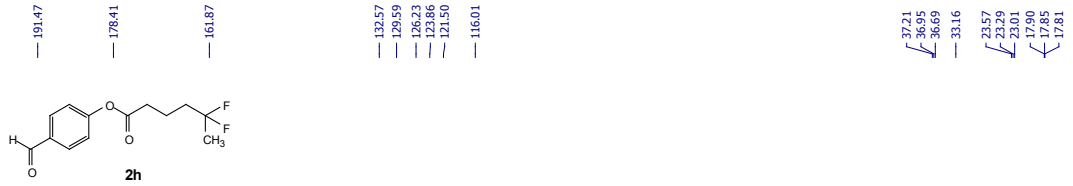

1.00
2.08
2.13
1.95
3.93
2.98
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


-90.96
-91.01
-91.05
-91.10
-91.14
-91.19
O
O
F
F
CH3
H
O

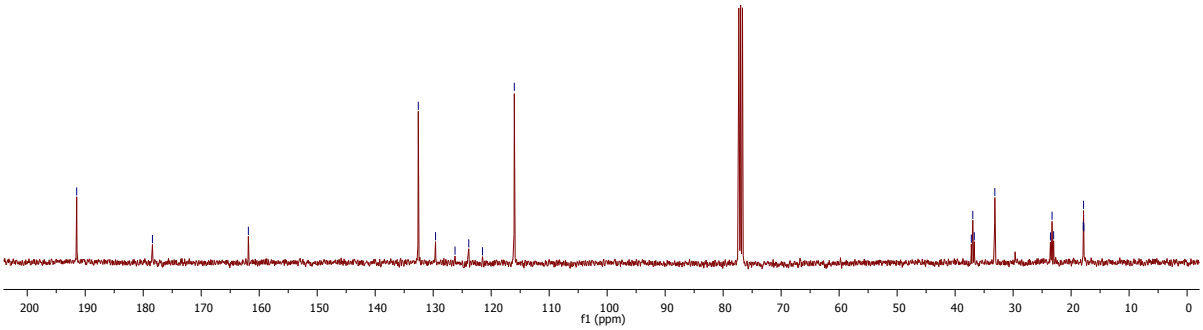
2h

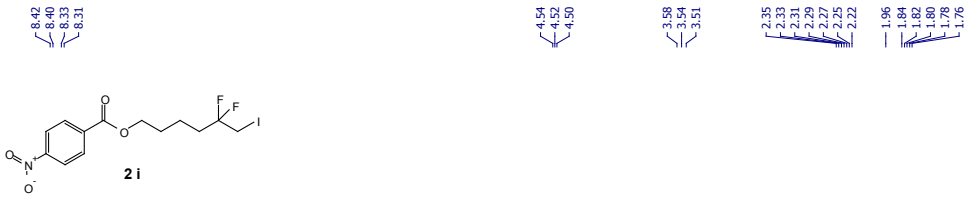

-90.96
-91.01
-91.05
-91.10
-91.14
-91.19
-90.8
-90.9
-91.0
-91.1
-91.2
-91.3
f1 (ppm)


30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
-220
-230
-240
-250
f1 (ppm)

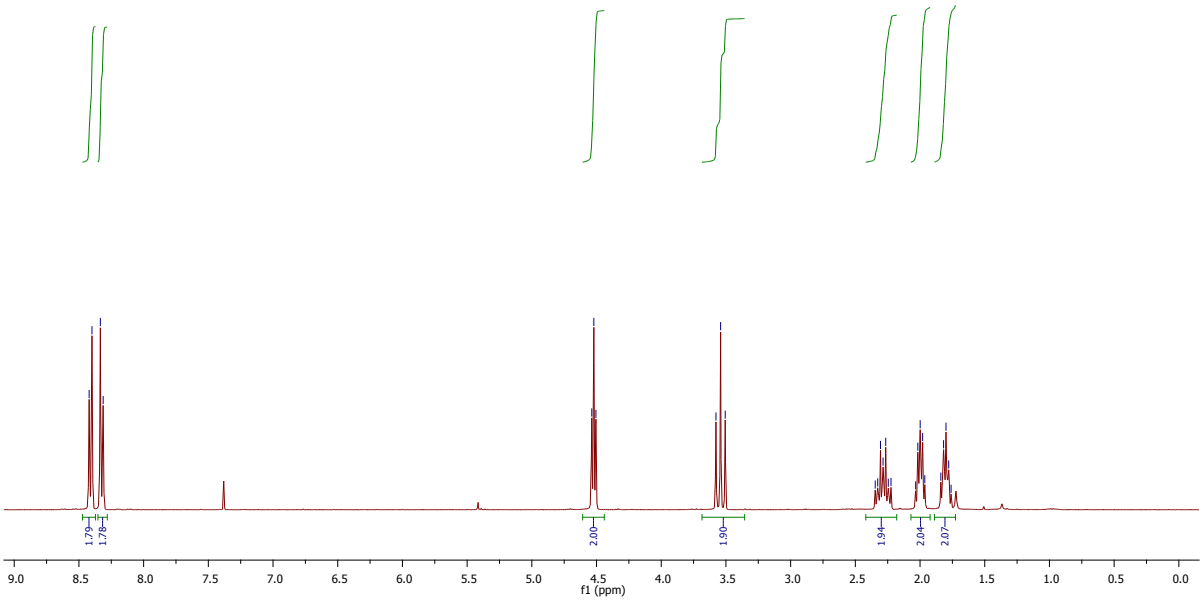

191.47
178.41
161.87
132.57
129.59
126.23
123.86
121.50
116.01
37.21
36.95
36.69
33.16
23.57
23.29
23.01
17.90
17.85
17.81
H
O
O
O
F
F
CH3

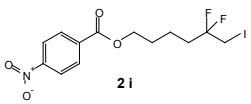
2h


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.42
8.40
8.33
8.31
4.54
4.52
4.50
3.58
3.54
3.51
2.35
2.33
2.31
2.29
2.27
2.25
2.22
1.96
1.84
1.82
1.80
1.78
1.76
O
O
F
F
I
O
N+
O-

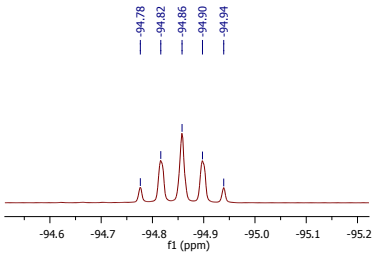
2 i

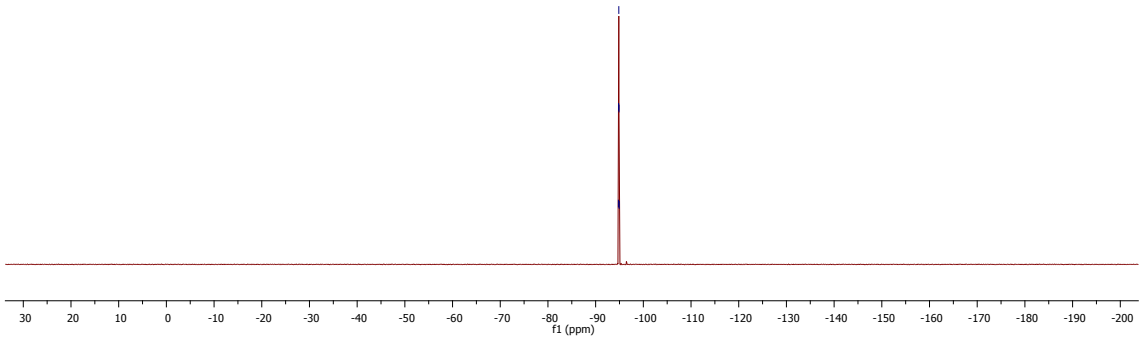

1.79
1.78
2.00
1.90
1.94
2.04
2.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)



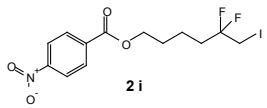
2 i


-94.78
-94.82
-94.86
-94.90
-94.94

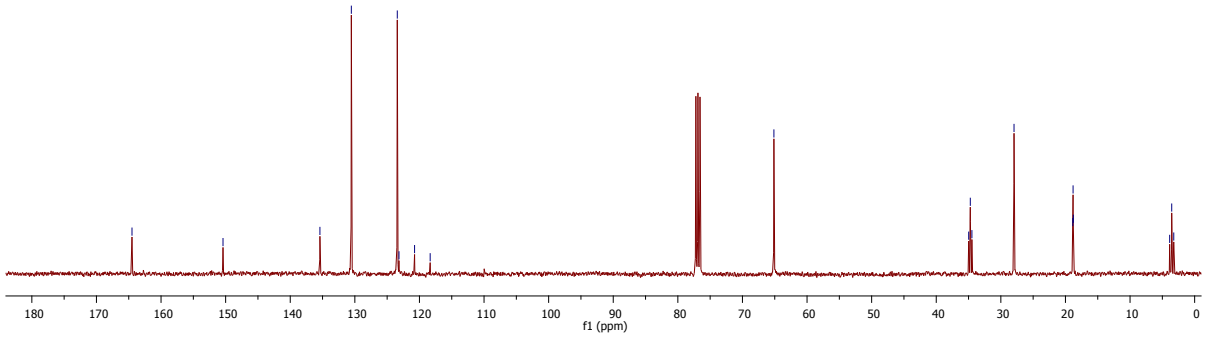

-94.78
-94.82
-94.86
-94.90
-94.94
-94.6
-94.7
-94.8
-94.9
-95.0
-95.1
-95.2
f1 (ppm)

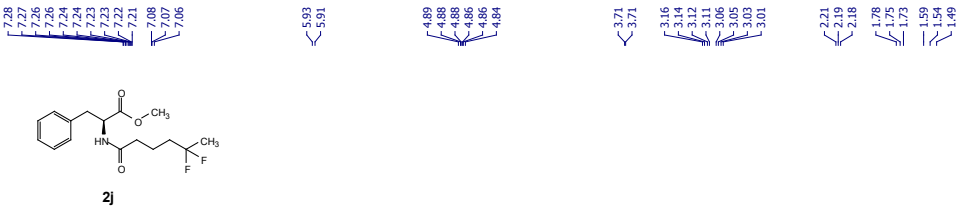

30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)


164.50
150.41
135.41
130.54
123.43
123.17
120.75
118.34
65.13
34.98
34.73
34.48
27.96
18.86
18.82
18.78
3.87
3.55
3.23



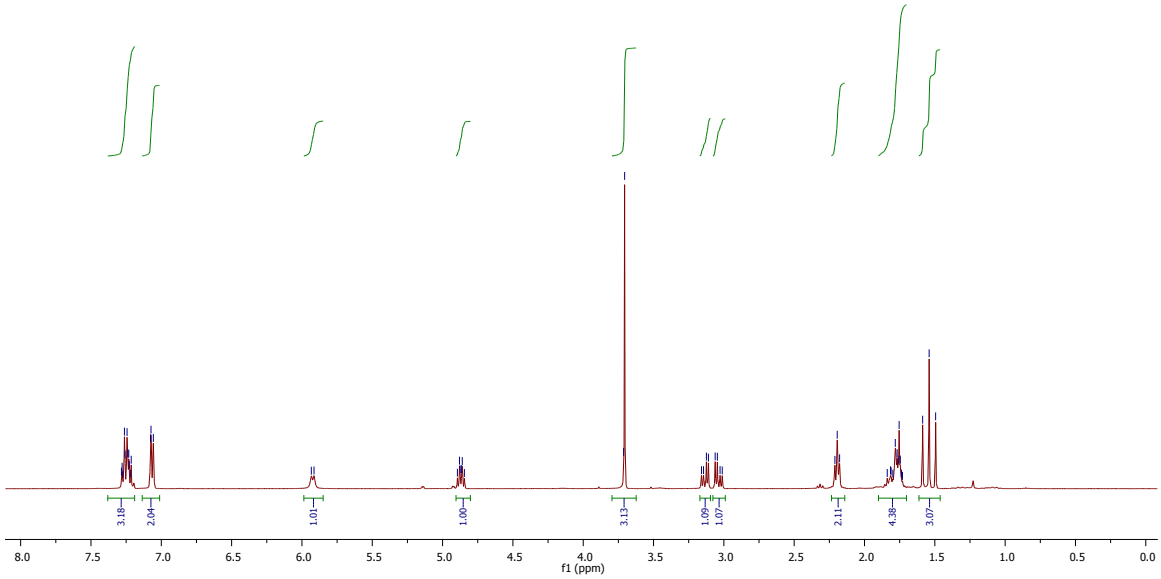
2 i


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

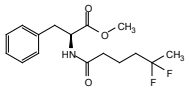

7.28
7.27
7.26
7.26
7.24
7.24
7.23
7.23
7.22
7.21
7.08
7.07
7.06
5.93
5.91
4.89
4.88
4.88
4.86
4.86
4.84
3.71
3.71
3.16
3.14
3.12
3.11
3.06
3.05
3.03
3.01
2.21
2.19
2.18
1.78
1.75
1.73
1.59
1.54
1.49

O
O
CH3
HN
O
CH3
F
F

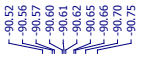
2j

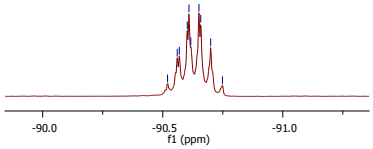

3.18
2.04
1.01
1.00
3.13
1.09
1.07
2.11
4.38
3.07
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

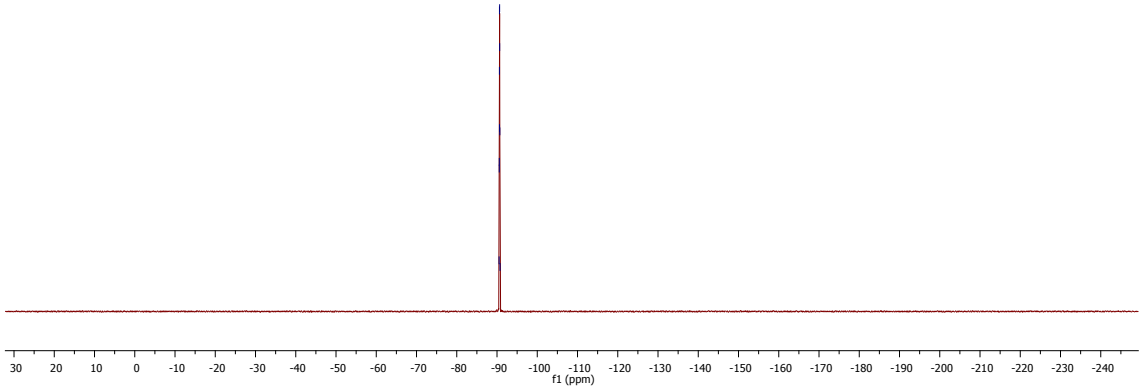

-90.52
-90.56
-90.57
-90.60
-90.61
-90.62
-90.65
-90.66
-90.70
-90.75

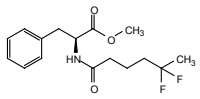

O
O
CH3
HN
O
CH3
F
F

2j

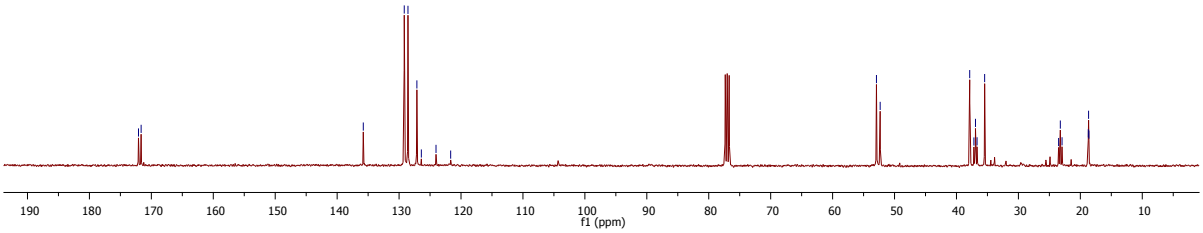

-90.52
-90.56
-90.57
-90.60
-90.61
-90.62
-90.65
-90.66
-90.70
-90.75

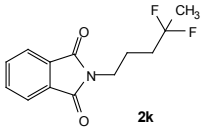

-90.0
-90.5
-91.0
f1 (ppm)


30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
-220
-230
-240
f1 (ppm)

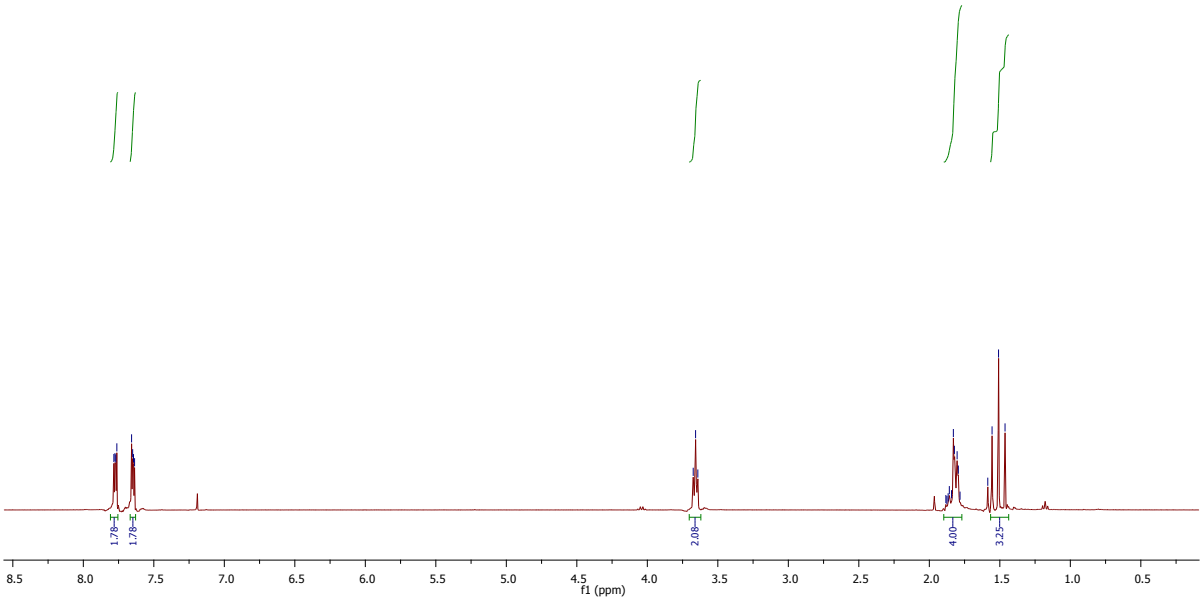


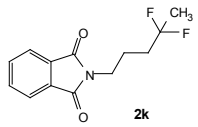
2j





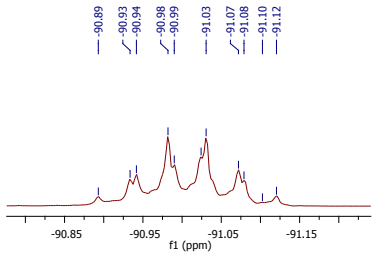
2k

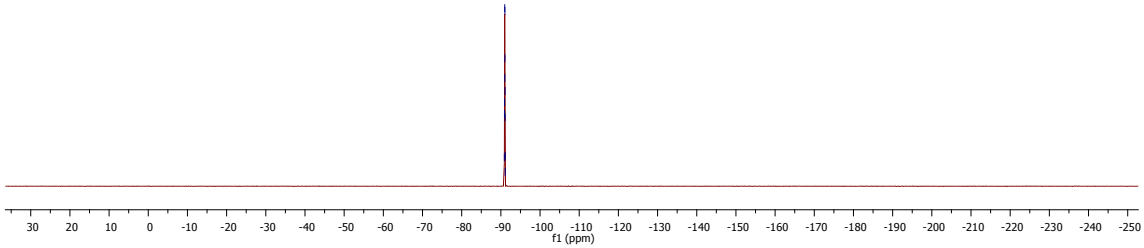



F
CH3
F
O
N
O

2k

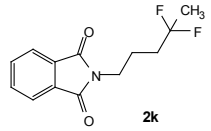

-90.89
-90.93
-90.94
-90.98
-90.99
-91.02
-91.03
-91.07
-91.08
-91.10
-91.12


-90.89
-90.93
-90.94
-90.98
-90.99
-91.03
-91.07
-91.08
-91.10
-91.12
-90.85
-90.95
-91.05
-91.15
f1 (ppm)

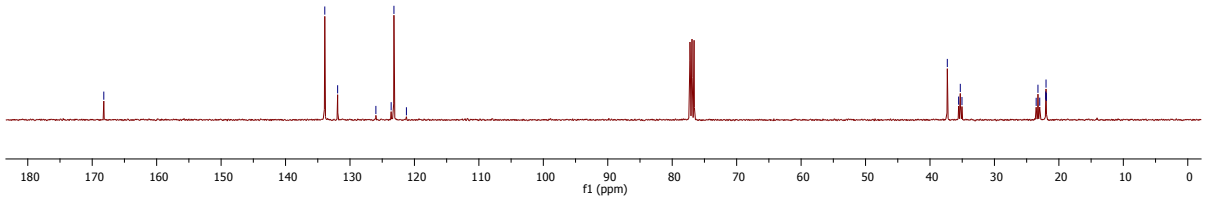

30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240 -250
f1 (ppm)

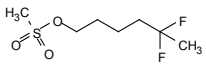

168.20
133.91
131.92
125.98
123.61
123.18
121.24


37.31
35.56
35.30
35.04
23.53
23.25
22.97
22.04
21.99
21.95


F
CH3
F
O
N
O

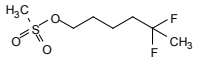
2k


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

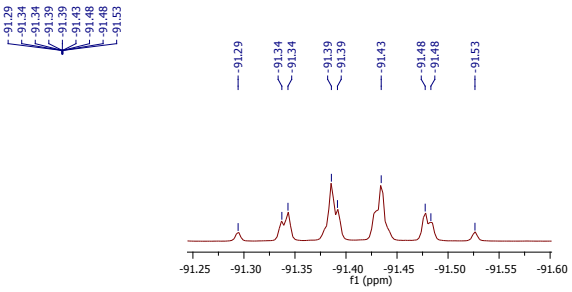


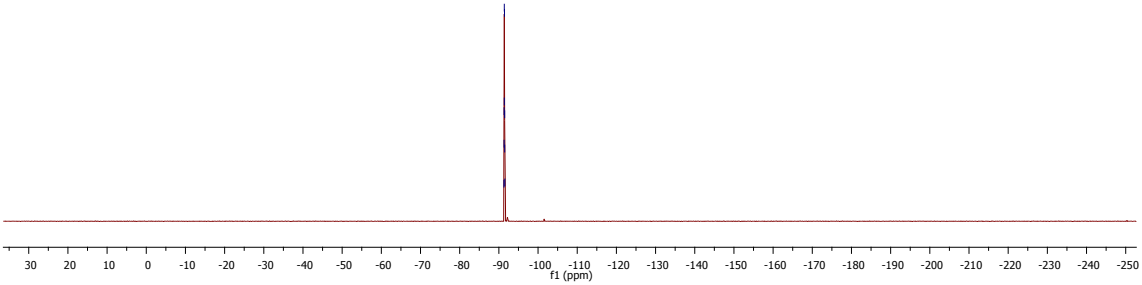
2 l

4.28
4.26
4.24
3.03
1.92
1.90
1.88
1.86
1.84
1.83
1.81
1.79
1.69
1.68
1.66
1.65
1.64
1.61
1.60
2.00
2.98
4.17
5.11
f1 (ppm)



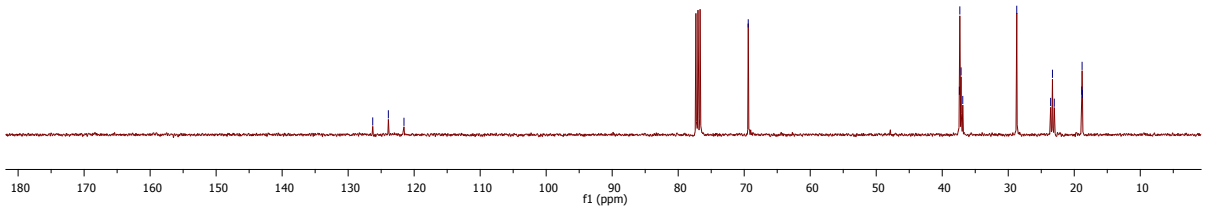
2 l


-91.29
-91.34
-91.34
-91.39
-91.39
-91.43
-91.48
-91.48
-91.53
f1 (ppm)


f1 (ppm)

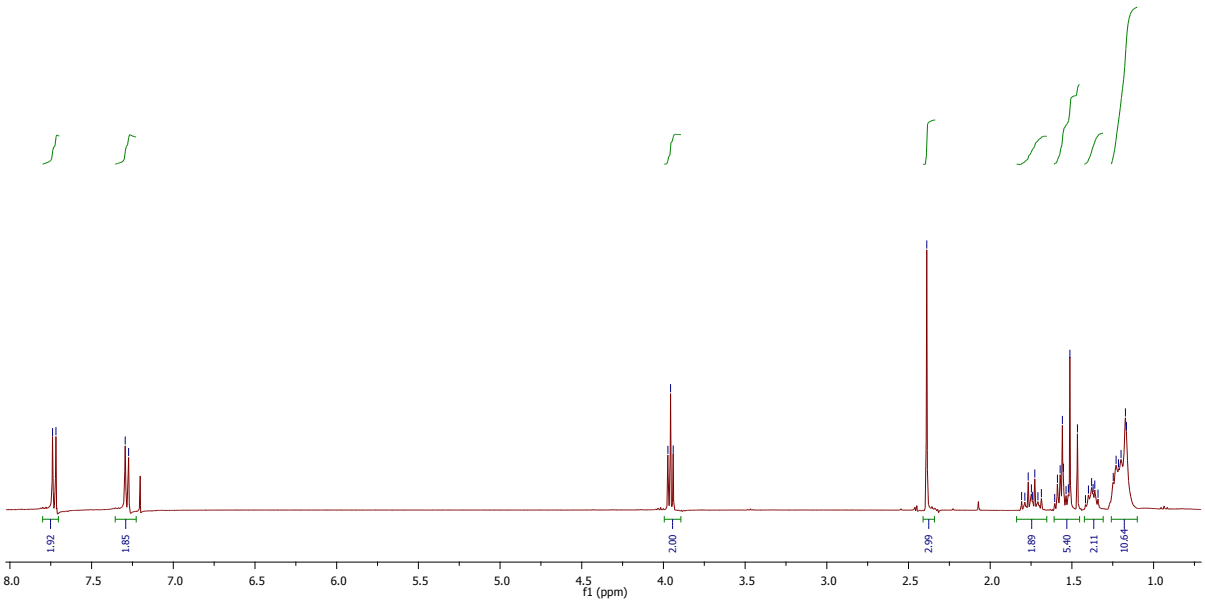

126.28
123.91
121.54
69.39
37.40
37.33
37.14
36.89
28.71
23.30
23.02
18.86
18.81
18.77

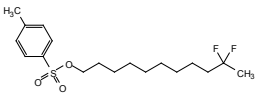
2 l


f1 (ppm)

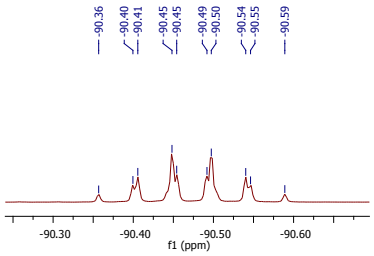

7.74
7.72
7.29
7.27
3.97
3.96
3.94
2.39
1.77
1.61
1.57
1.55
1.52
1.47
1.40
1.37
1.34
1.25
1.23
1.21
1.20
1.17
1.17

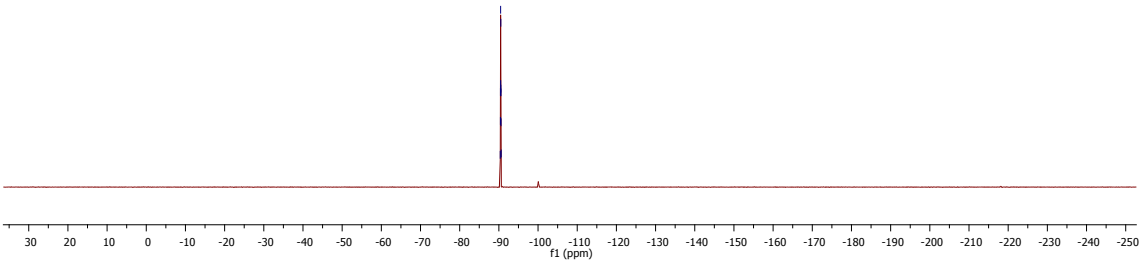
2 m

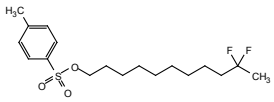

1.92
1.85
2.00
2.99
1.89
5.40
2.11
10.64
f1 (ppm)



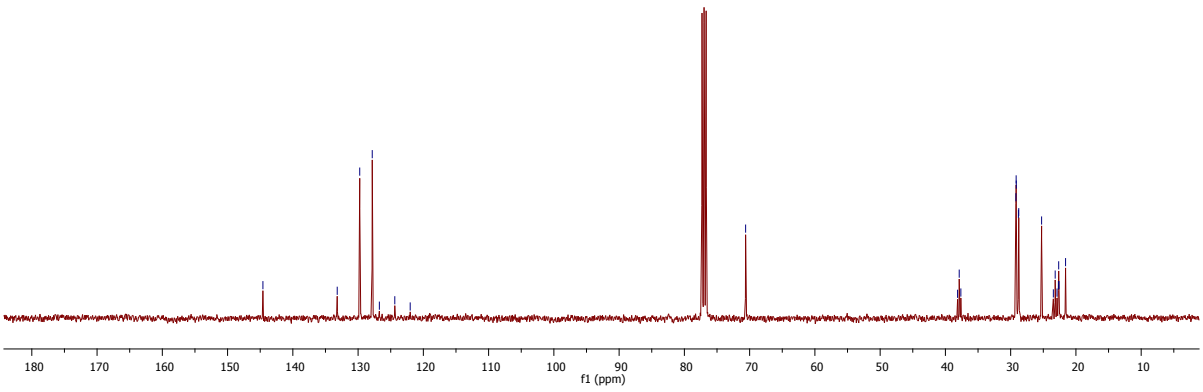
2 m

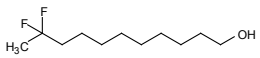




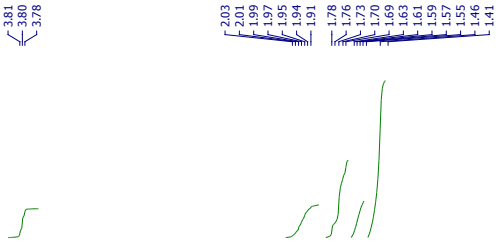


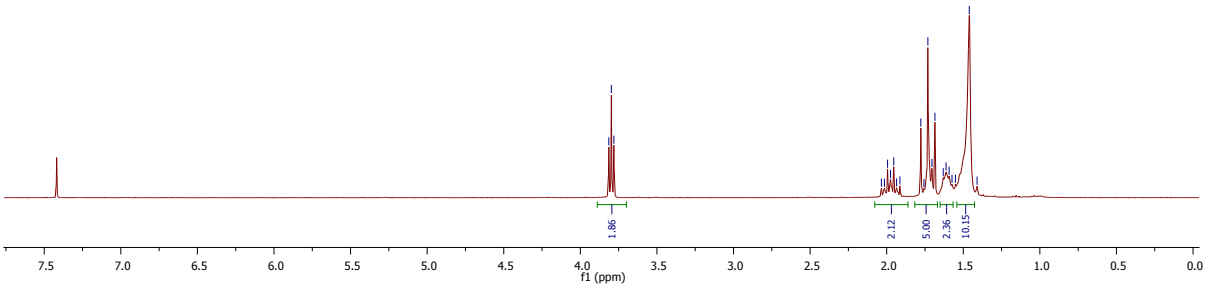
2 m

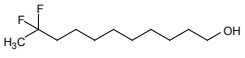




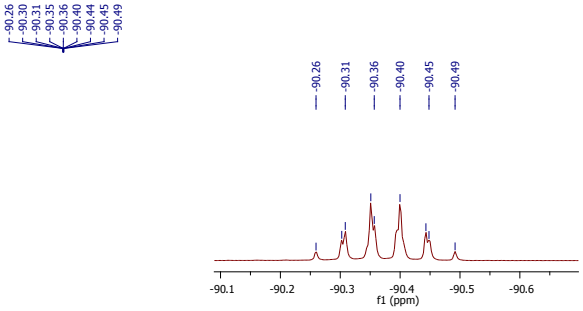
2n

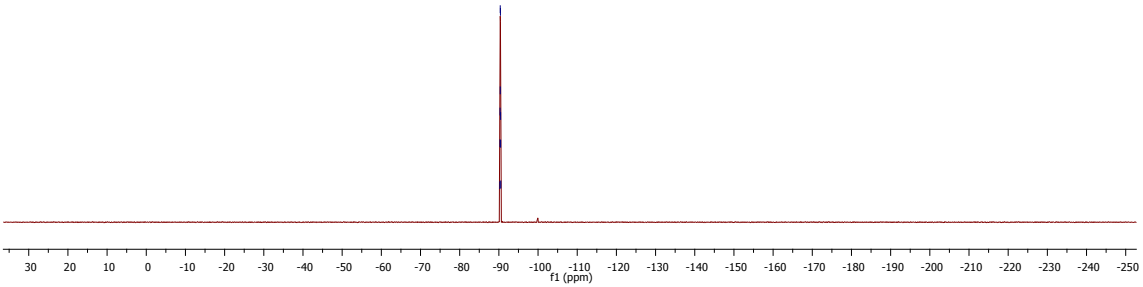

3.81
3.80
3.78
2.03
2.01
1.99
1.97
1.95
1.94
1.91
1.78
1.76
1.73
1.70
1.69
1.63
1.61
1.59
1.57
1.55
1.46
1.41


1.86
2.12
5.00
2.36
10.15
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
f1 (ppm)



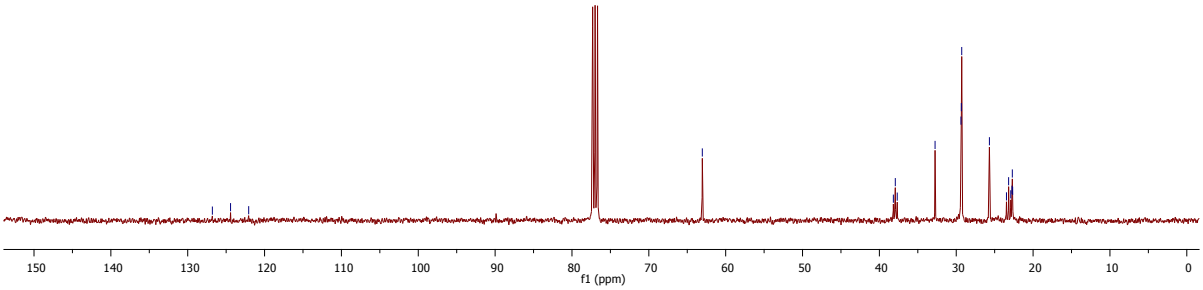
2n

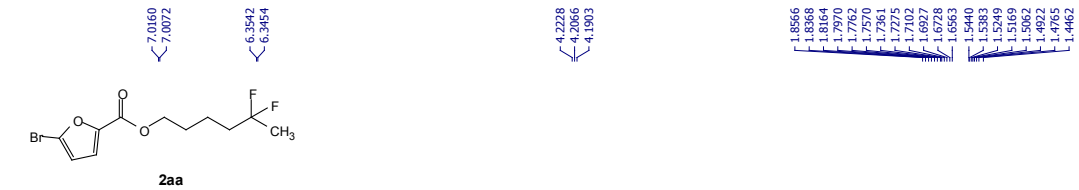

-90.26
-90.30
-90.31
-90.35
-90.36
-90.40
-90.44
-90.45
-90.49
-90.1
-90.2
-90.3
-90.4
-90.5
-90.6
f1 (ppm)


30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
-220
-230
-240
-250
f1 (ppm)

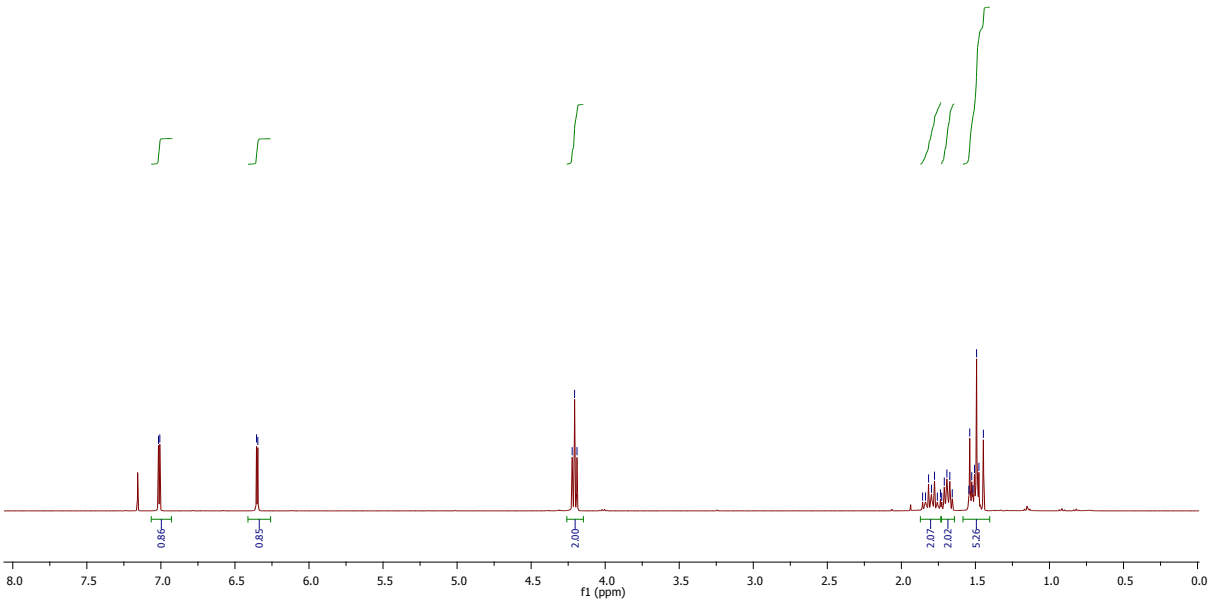

126.79
124.43
122.07
63.04
38.18
37.93
37.68
32.75
29.29
25.68
23.46
23.18
22.90
22.75
22.70
22.66
F
F
H3C
OH

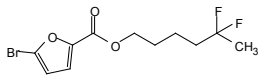
2n


150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.0160
7.0072
6.3542
6.3454
4.2228
4.2066
4.1903
1.8566
1.8368
1.8164
1.7970
1.7762
1.7570
1.7361
1.7275
1.7102
1.6927
1.6728
1.6563
1.5440
1.5383
1.5249
1.5169
1.5062
1.4922
1.4765
1.4462
O
O
O
Br
F
F
CH3

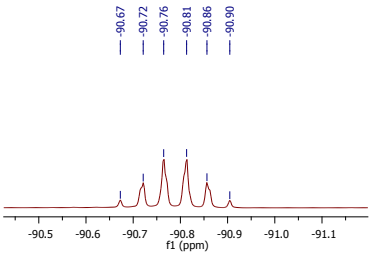
2aa

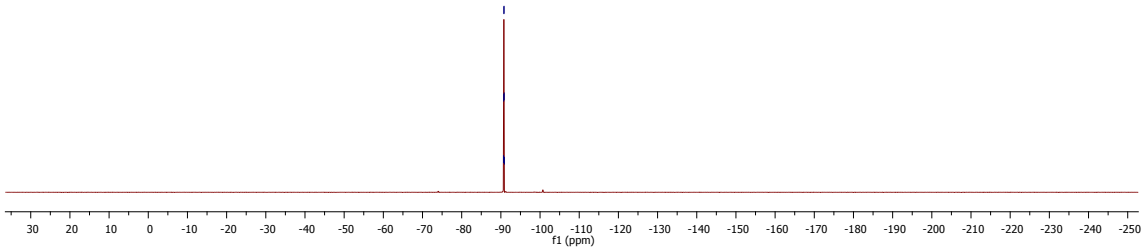

0.86
0.85
2.00
2.07
2.02
5.26
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Br
O
O
O
F
F
CH3

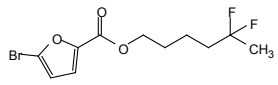
2aa


-90.67
-90.72
-90.76
-90.81
-90.86
-90.90

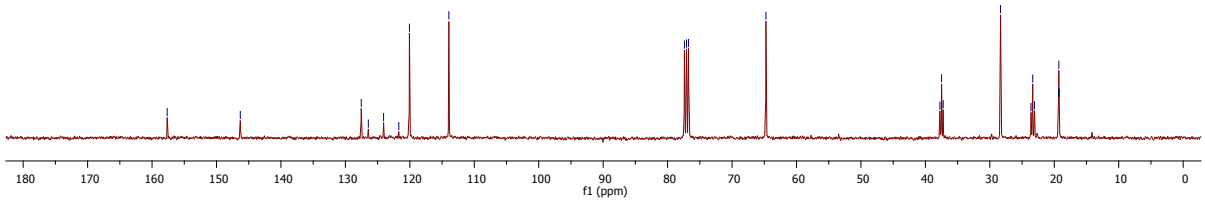

-90.67
-90.72
-90.76
-90.81
-90.86
-90.90
-90.5
-90.6
-90.7
-90.8
-90.9
-91.0
-91.1
f1 (ppm)

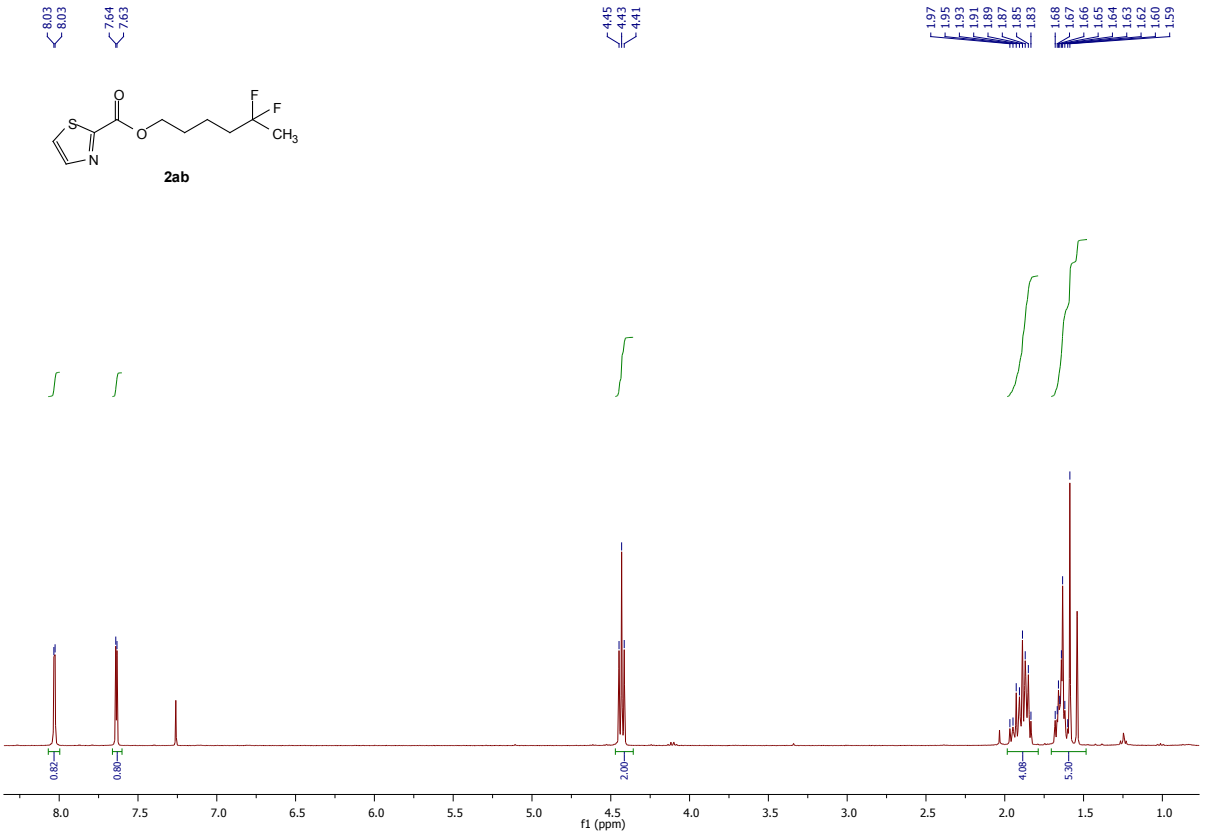

30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240 -250
f1 (ppm)

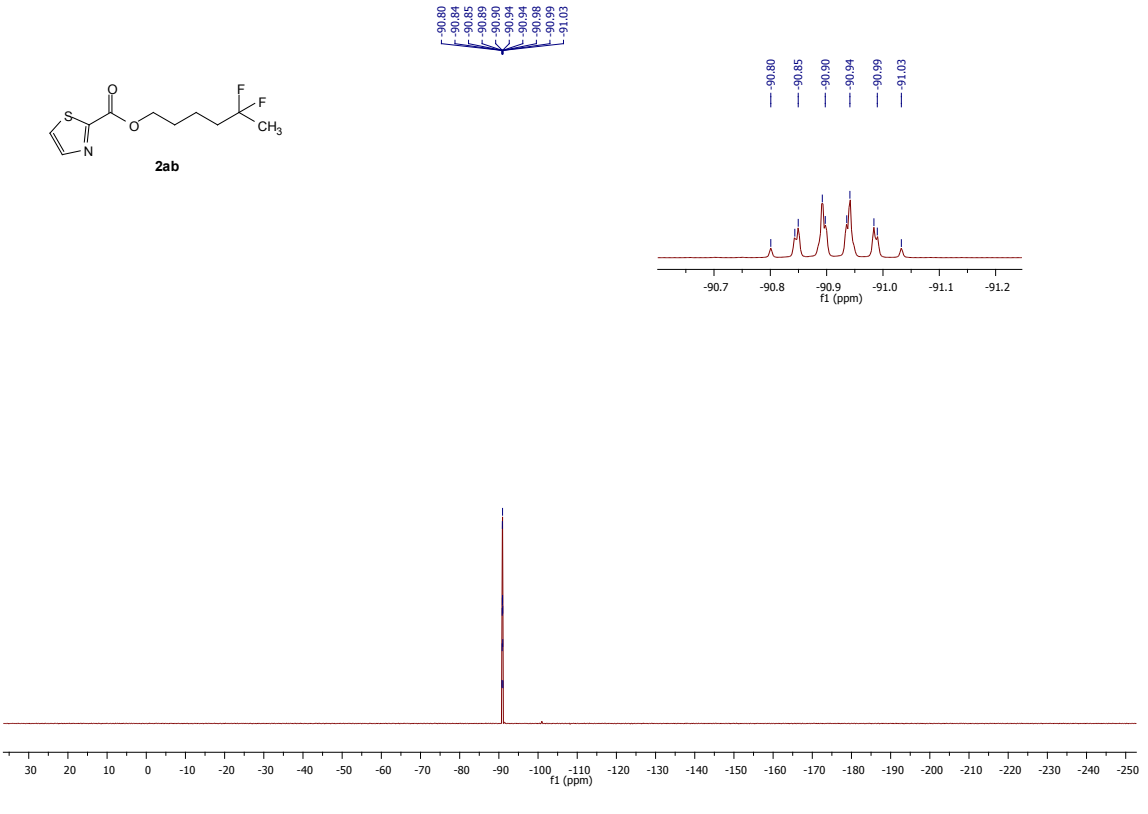

157.65
146.30
127.54
126.44
124.07
121.71
120.06
113.94
77.39
77.07
76.75
64.74
37.74
37.49
37.23
28.34
23.34
23.06
19.34
19.29
19.25

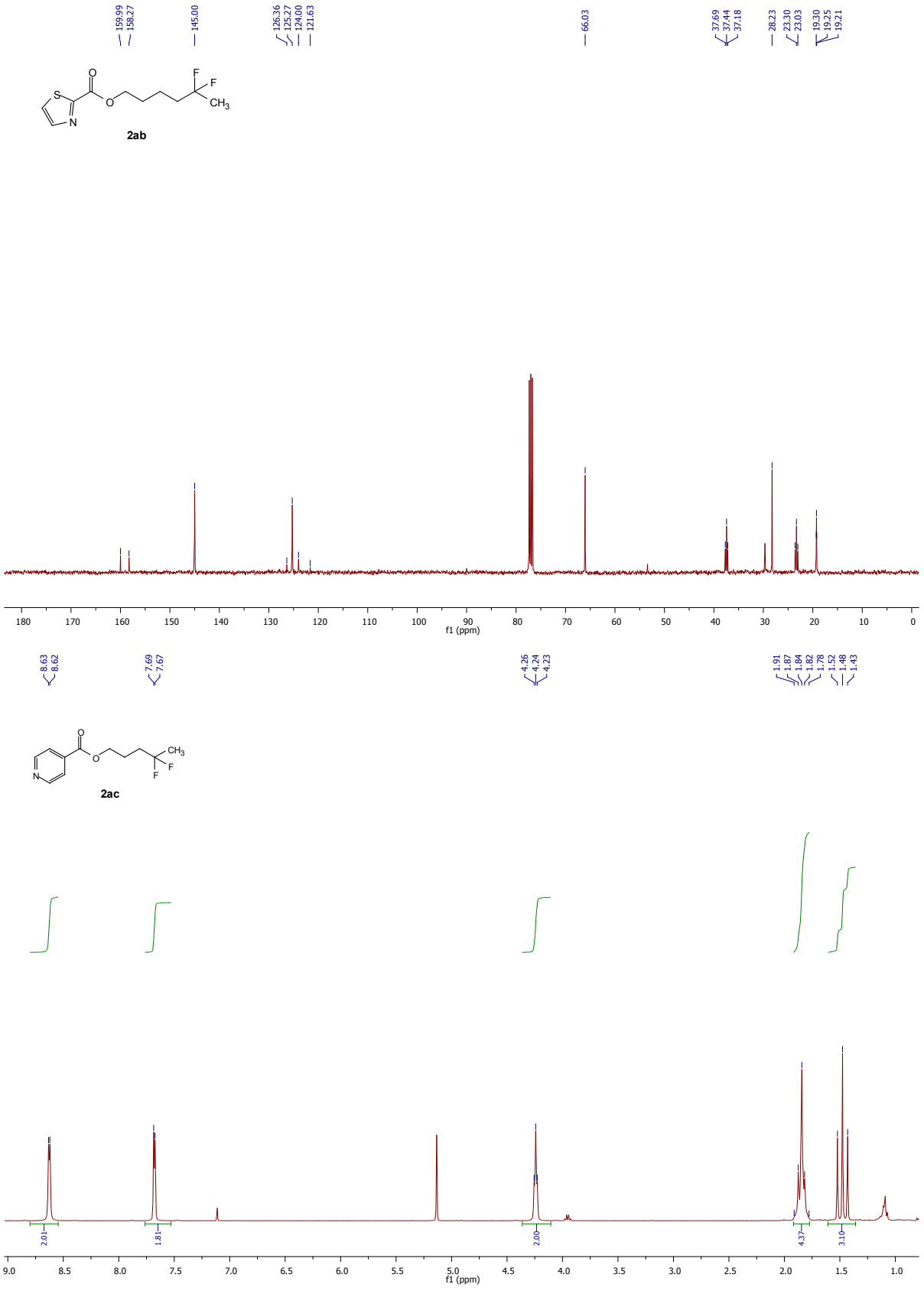

Br
O
O
O
F
F
CH3

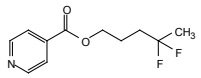
2aa


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.03
8.03
7.64
7.63
4.45
4.43
4.41
1.97
1.95
1.93
1.91
1.89
1.87
1.85
1.83
1.68
1.67
1.66
1.65
1.64
1.63
1.62
1.60
1.59
O
S
N
O
F
F
CH3
2ab
0.82
0.80
2.00
4.08
5.30
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
f1 (ppm)

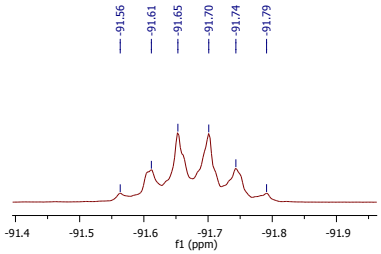

-90.80
-90.84
-90.85
-90.89
-90.90
-90.94
-90.94
-90.98
-90.99
-91.03
O
S
N
O
F
F
CH3
2ab
-90.80
-90.85
-90.90
-90.94
-90.99
-91.03
-90.7
-90.8
-90.9
-91.0
-91.1
-91.2
f1 (ppm)
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
-220
-230
-240
-250
f1 (ppm)

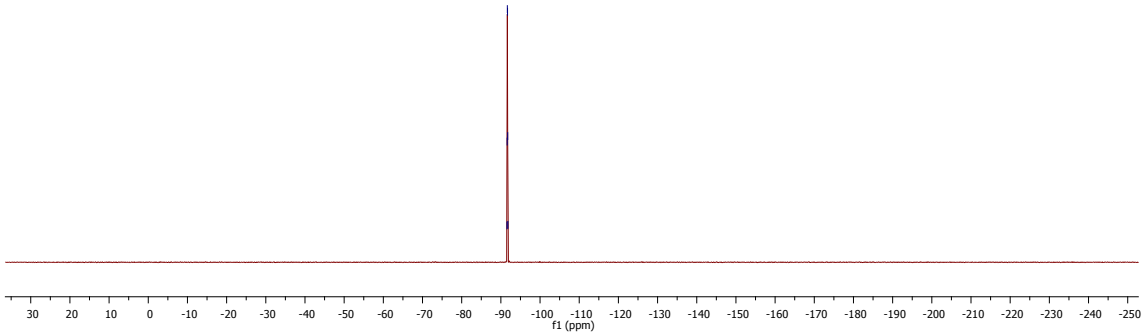

159.99
158.27
145.00
126.36
125.27
124.00
121.63
66.03
37.69
37.44
37.18
28.23
23.30
23.03
19.30
19.25
19.21
O
S
N
O
F
F
CH3
2ab
f1 (ppm)
8.63
8.62
7.69
7.67
4.26
4.24
4.23
1.91
1.87
1.84
1.82
1.78
1.52
1.48
1.43
O
N
O
CH3
F
F
2ac
2.01
1.81
2.00
4.37
3.10
f1 (ppm)



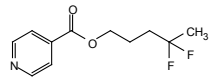
2ac


-91.56
-91.61
-91.65
-91.70
-91.74
-91.79

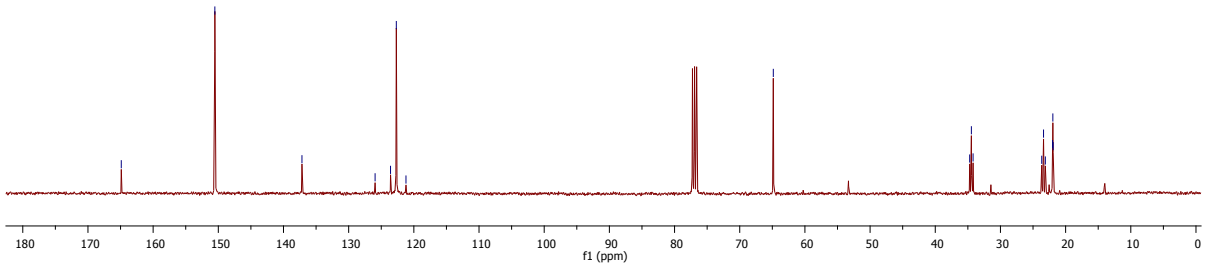

-91.56
-91.61
-91.65
-91.70
-91.74
-91.79
-91.4
-91.5
-91.6
-91.7
-91.8
-91.9
f1 (ppm)

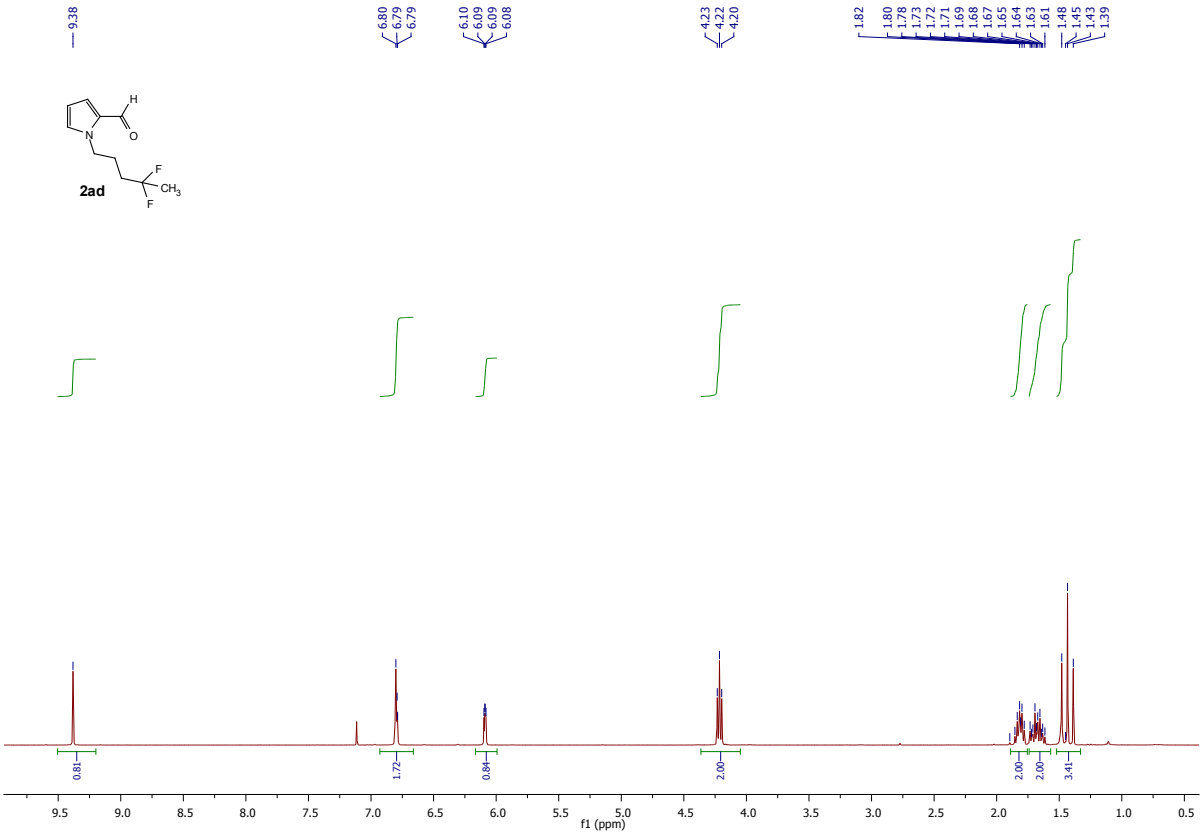

30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240 -250
f1 (ppm)

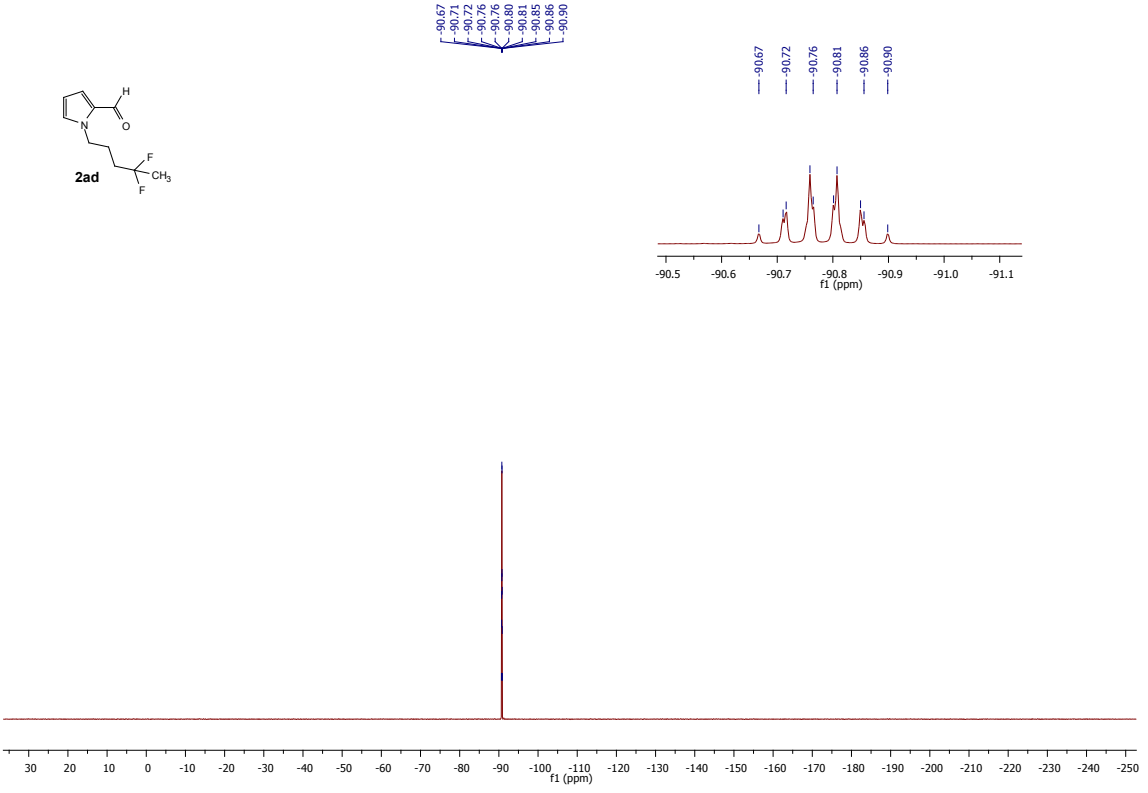

164.89
150.53
137.17
125.94
123.58
122.69
121.21
64.85
34.72
34.46
34.20
23.67
23.39
23.11
22.00
21.96
21.92



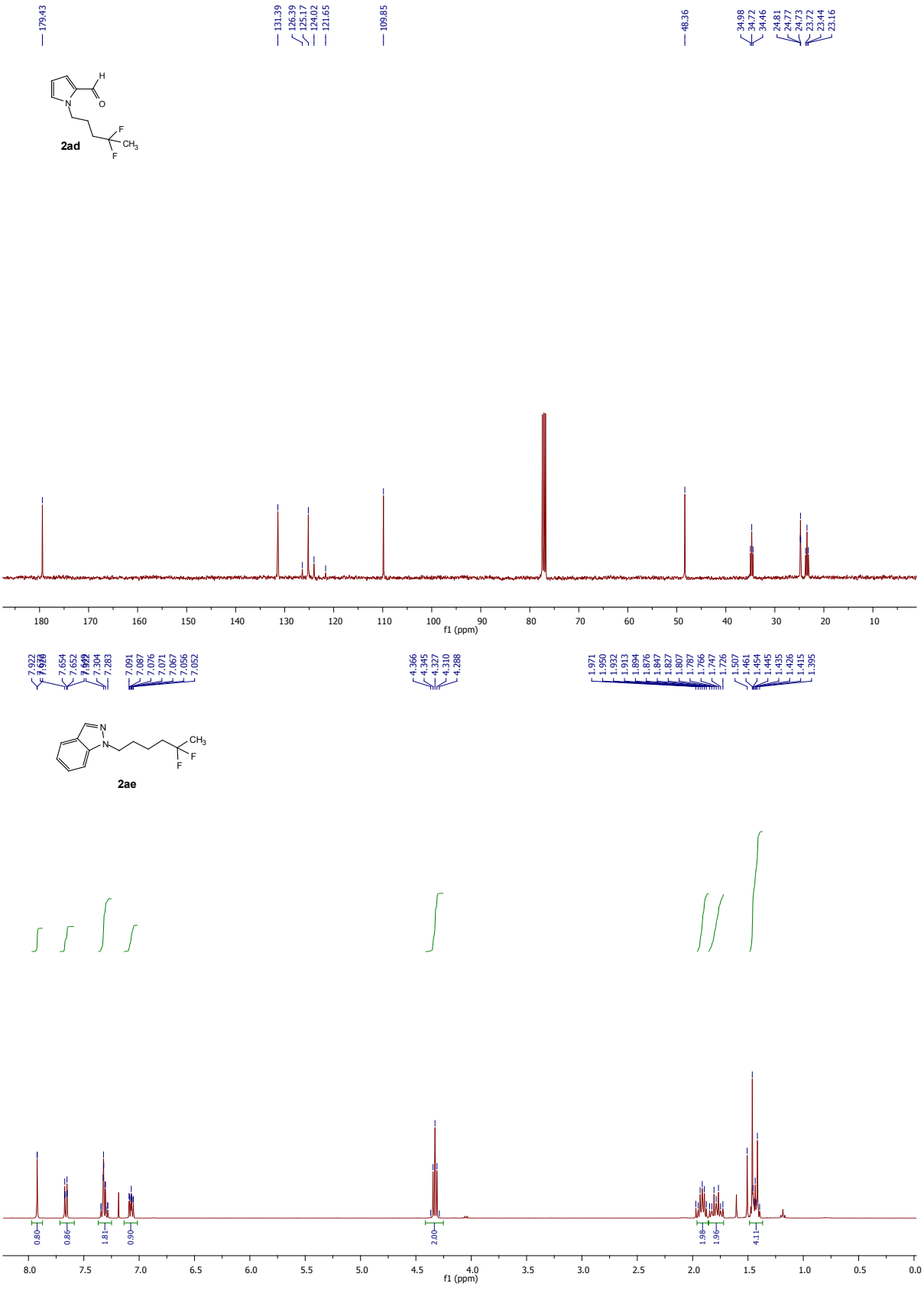
2ac

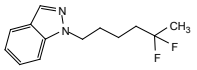

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


9.38
6.80
6.79
6.79
6.10
6.09
6.09
6.08
4.23
4.22
4.20
1.82
1.80
1.78
1.73
1.72
1.71
1.69
1.68
1.67
1.65
1.64
1.63
1.61
1.48
1.45
1.43
1.39
2ad
0.81
1.72
0.84
2.00
2.00
2.00
3.41
f1 (ppm)


-90.67
-90.71
-90.72
-90.76
-90.76
-90.80
-90.81
-90.85
-90.86
-90.90
2ad
f1 (ppm)

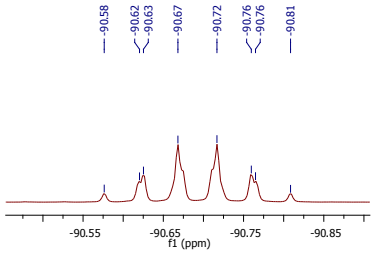
f1 (ppm)

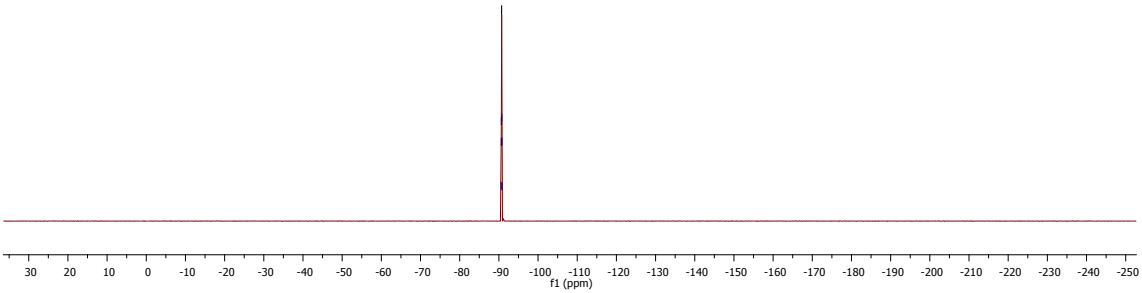

179.43
131.39
126.39
125.17
124.02
121.65
109.85
48.36
34.98
34.72
34.46
24.81
24.77
24.73
23.72
23.44
23.16
2ad
f1 (ppm)
7.922
7.654
7.652
7.304
7.283
7.091
7.087
7.076
7.071
7.067
7.056
7.052
4.366
4.345
4.327
4.310
4.288
1.971
1.950
1.932
1.913
1.894
1.876
1.847
1.827
1.807
1.787
1.766
1.747
1.726
1.507
1.461
1.454
1.445
1.435
1.426
1.415
1.395
2ae
0.80
0.86
1.81
0.90
2.00
1.98
1.96
4.11
f1 (ppm)



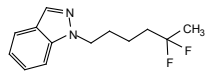
2ae


-90.58
-90.62
-90.63
-90.67
-90.72
-90.76
-90.76
-90.81

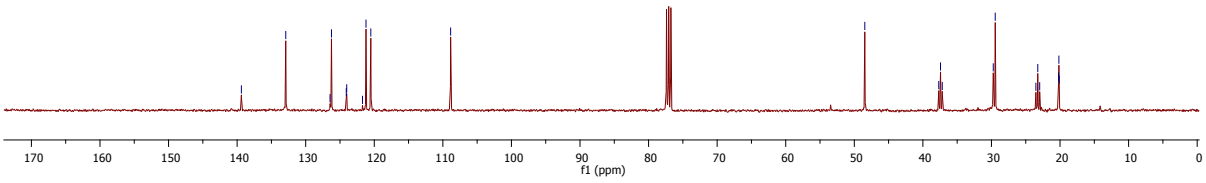

-90.58
-90.62
-90.63
-90.67
-90.72
-90.76
-90.76
-90.81
-90.55
-90.65
-90.75
-90.85
f1 (ppm)

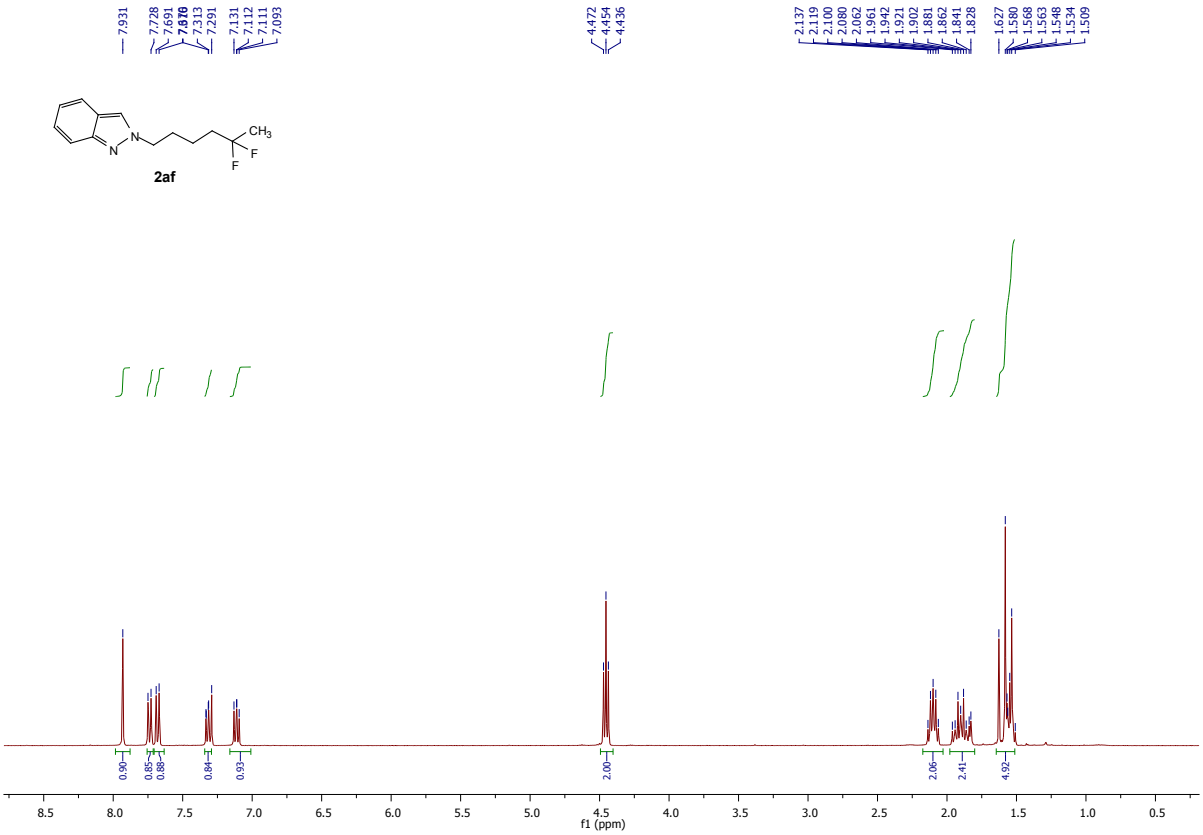

30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240 -250
f1 (ppm)

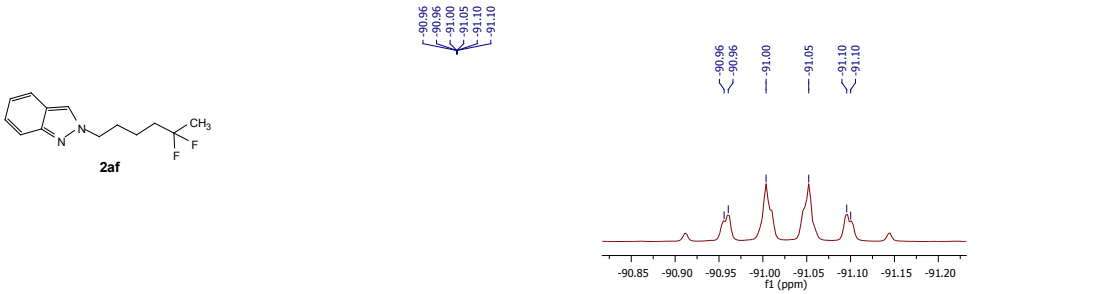

139.38
132.91
126.45
126.24
124.02
121.72
121.21
120.50
108.87
48.47
37.69
37.43
37.17
29.74
29.45
23.24
22.97
20.21
20.17
20.12

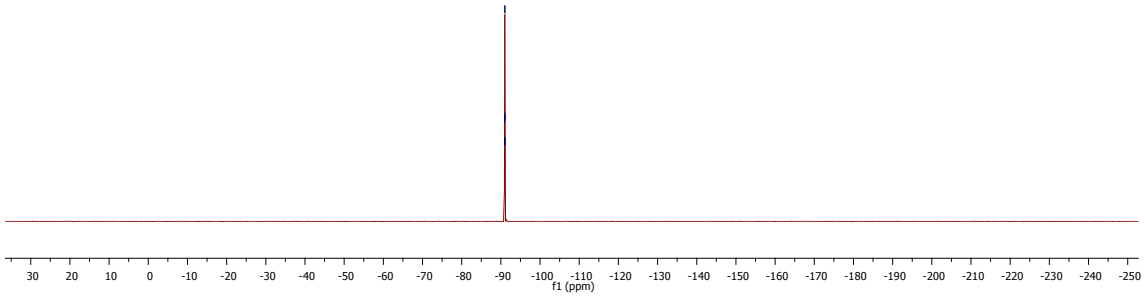


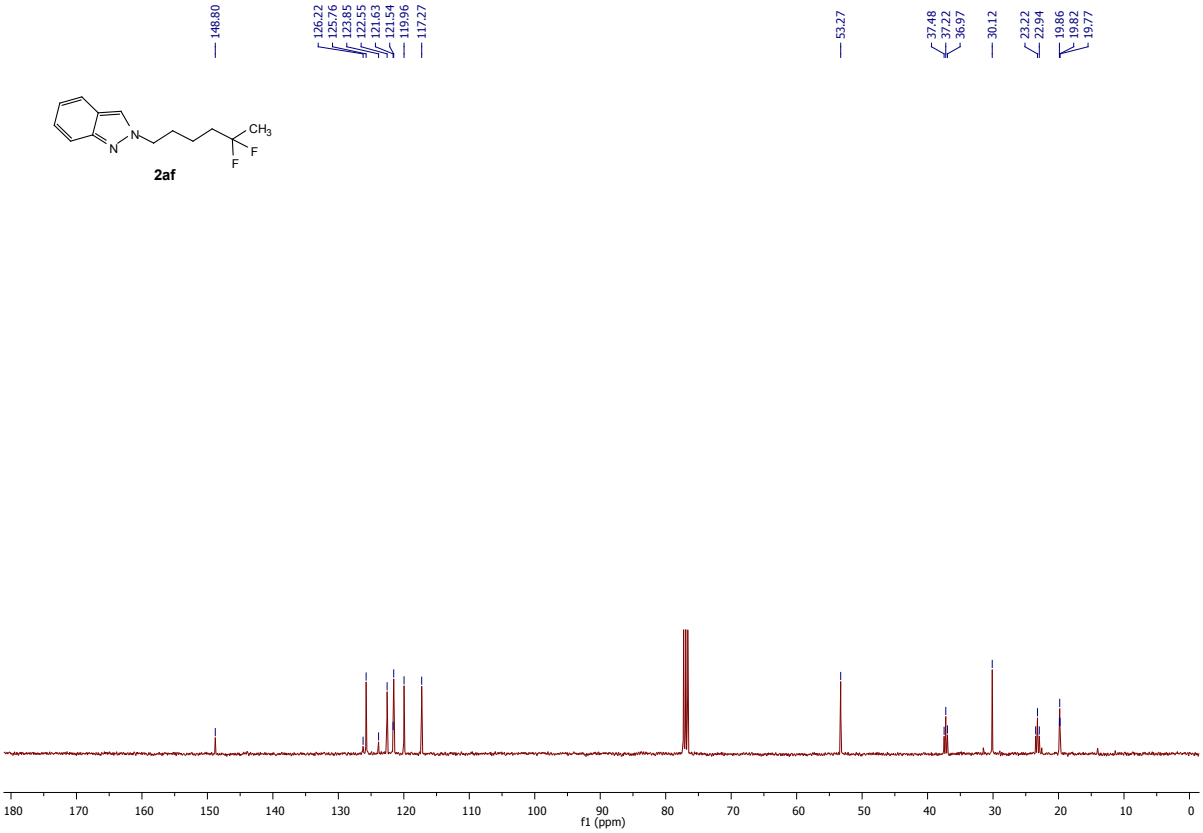
2ae

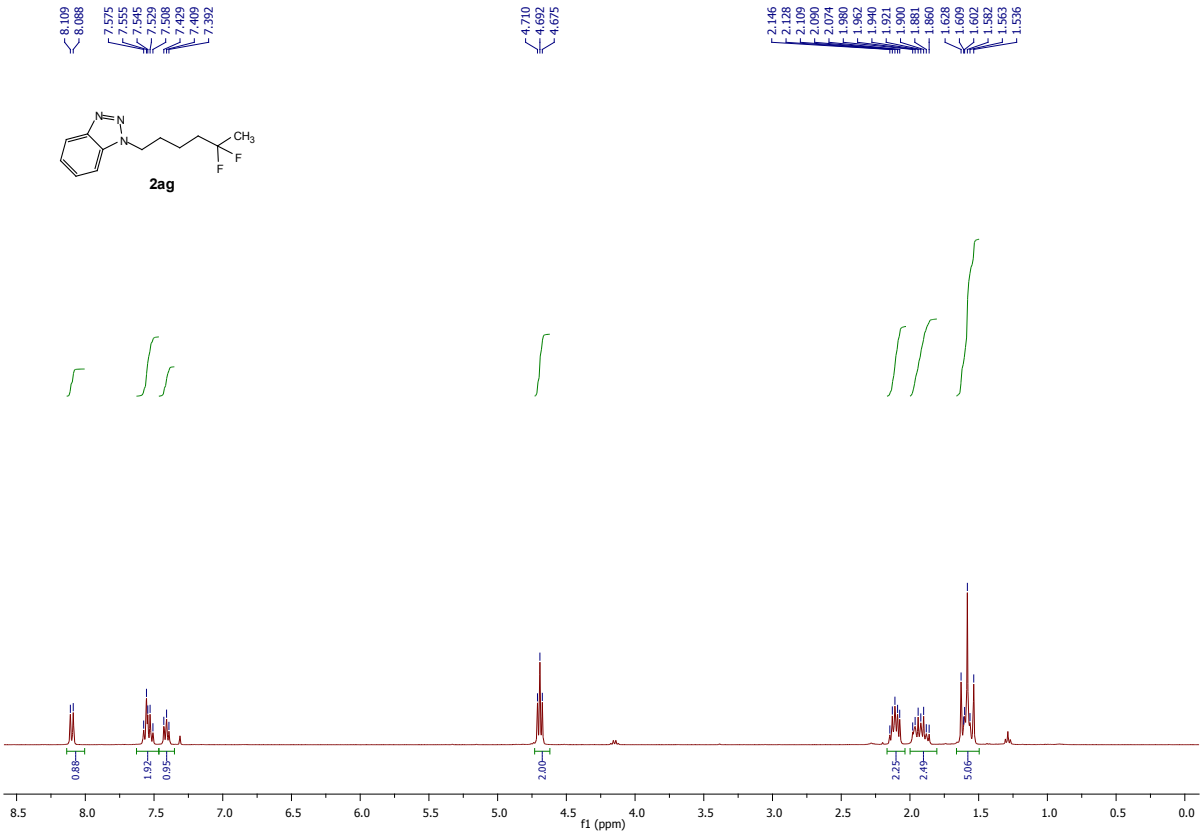

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

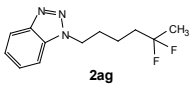

7.931
7.728
7.691
7.313
7.291
7.131
7.112
7.111
7.093
4.472
4.454
4.436
2.137
2.119
2.100
2.080
2.062
1.961
1.942
1.921
1.902
1.881
1.862
1.841
1.828
1.627
1.580
1.568
1.563
1.548
1.534
1.509
CH3
F
F
N
N
2af
0.90
0.85
0.88
0.84
0.93
2.00
2.06
2.41
4.92
f1 (ppm)


-90.96
-90.96
-91.00
-91.05
-91.10
-91.10
CH3
F
F
N
N
2af
f1 (ppm)


f1 (ppm)

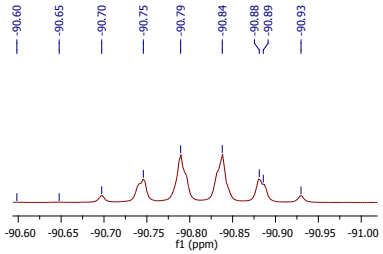

148.80
126.22
125.76
123.85
122.55
121.63
121.54
119.96
117.27
53.27
37.48
37.22
36.97
30.12
23.22
22.94
19.86
19.82
19.77
CH3
F
F
N
N
2af
f1 (ppm)

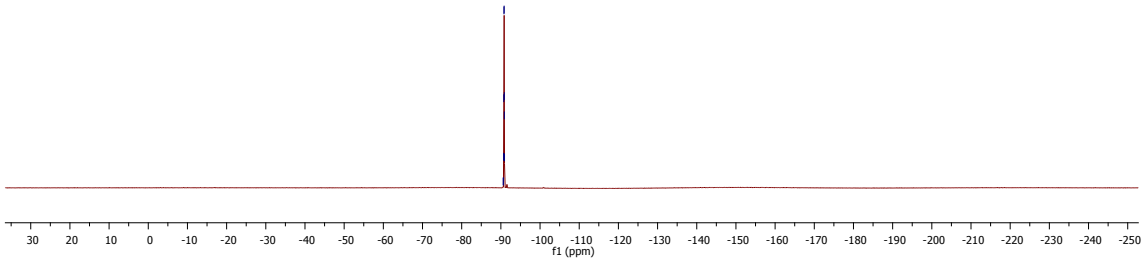

8.109
8.088
7.575
7.555
7.545
7.529
7.508
7.429
7.409
7.392
4.710
4.692
4.675
2.146
2.128
2.109
2.090
2.074
1.980
1.962
1.940
1.921
1.900
1.881
1.860
1.628
1.609
1.602
1.582
1.563
1.536
CH3
F
F
N
N
2ag
0.88
1.92
0.95
2.00
2.25
2.49
5.06
f1 (ppm)

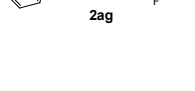

CH3
F
F

2ag

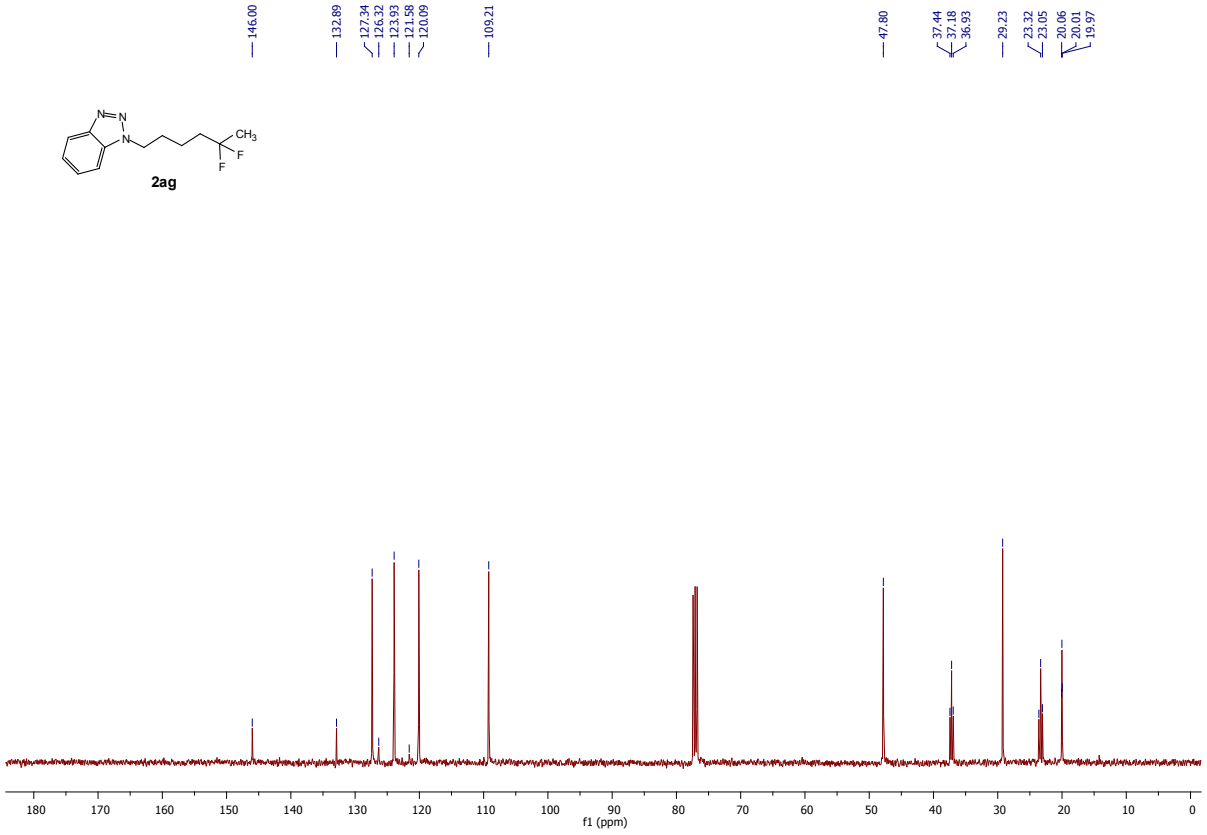

-90.60
-90.65
-90.70
-90.75
-90.79
-90.84
-90.88
-90.89
-90.93

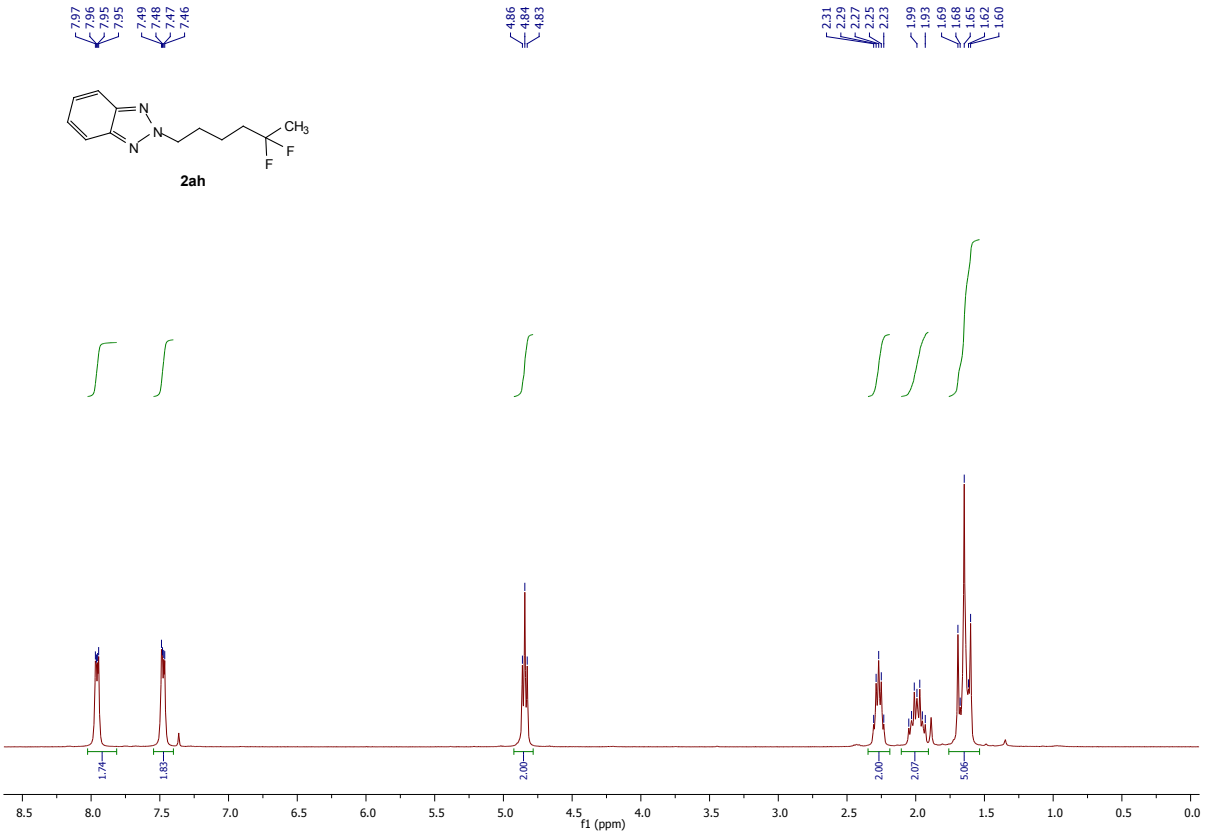

-90.60
-90.65
-90.70
-90.75
-90.79
-90.84
-90.88
-90.89
-90.93
-90.60 -90.65 -90.70 -90.75 -90.80 -90.85 -90.90 -90.95 -91.00
f1 (ppm)

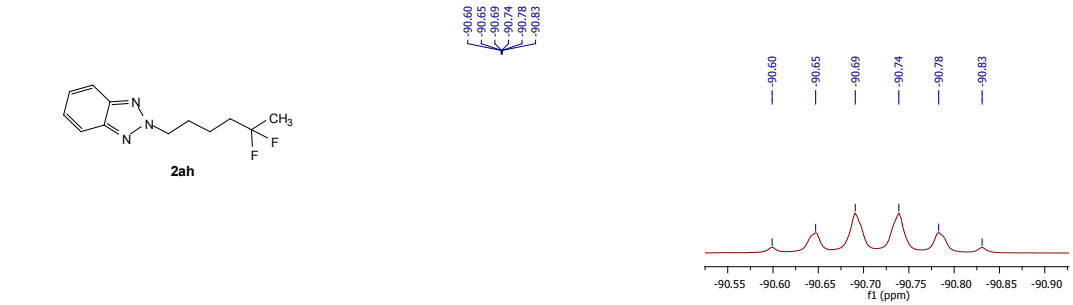

30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240 -250
f1 (ppm)

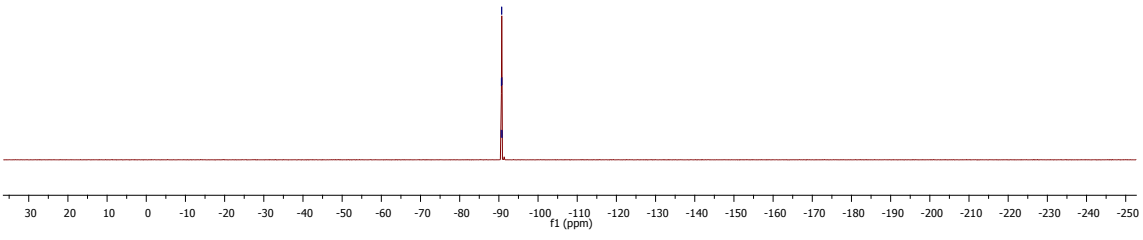

CH3
F
F

2ag


146.00
132.89
127.34
126.32
123.93
121.58
120.09
109.21
47.80
37.44
37.18
36.93
29.23
23.32
23.05
20.06
20.01
19.97
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

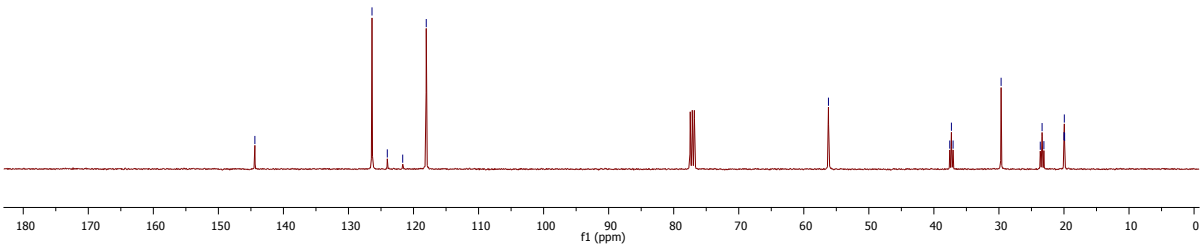

7.97
7.96
7.95
7.95
7.49
7.48
7.47
7.46
4.86
4.84
4.83
2.31
2.29
2.27
2.25
2.23
1.99
1.93
1.69
1.68
1.65
1.62
1.60
CH3
F
F
2ah
1.74
1.83
2.00
2.00
2.07
5.06
f1 (ppm)

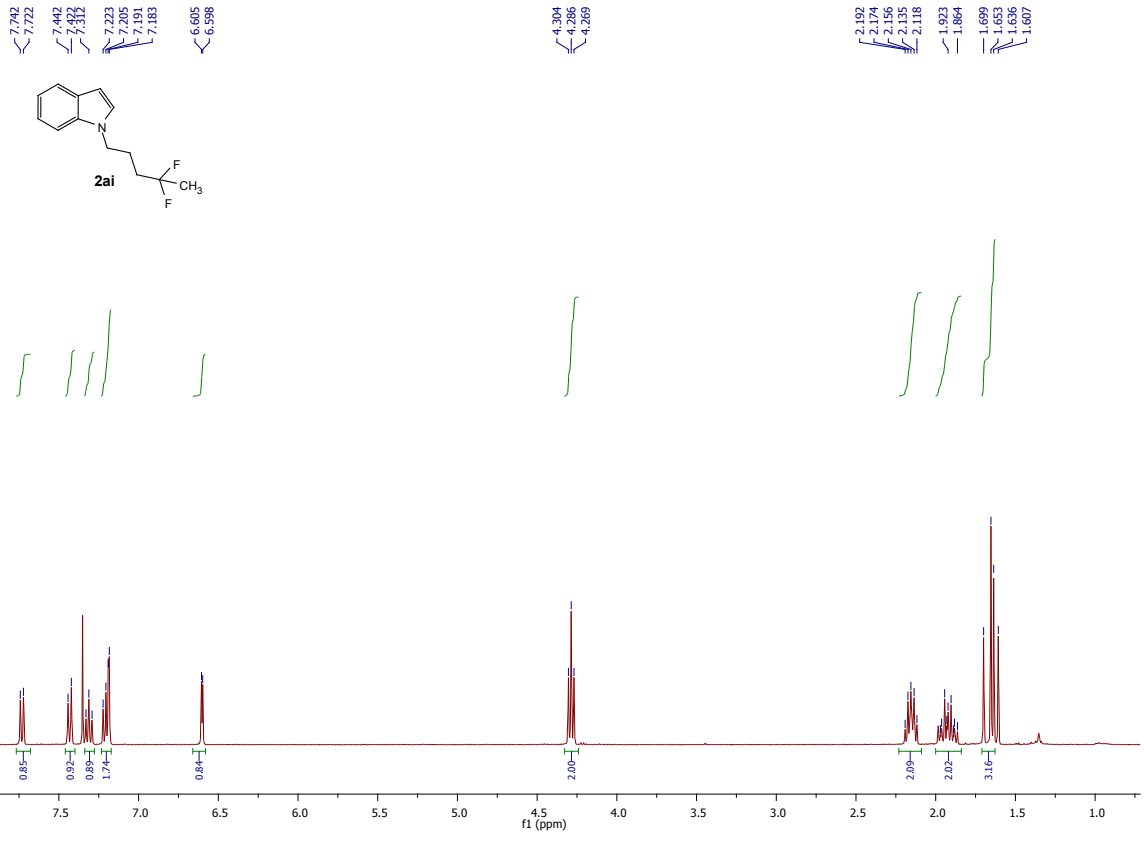

-90.60
-90.65
-90.69
-90.74
-90.78
-90.83
CH3
F
F
2ah
f1 (ppm)


f1 (ppm)

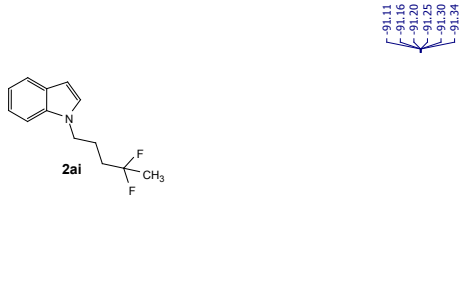

144.39
126.38
124.03
121.66
118.04
56.21
37.56
37.31
37.05
29.67
23.37
23.09
19.99
19.95
19.90
N
N
N
CH3
F
F

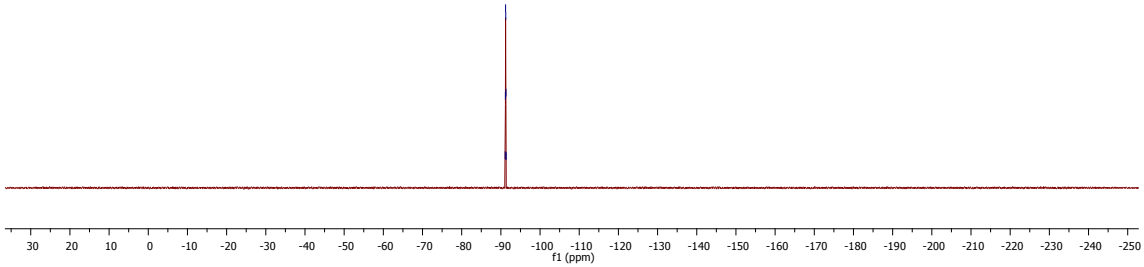
2ah

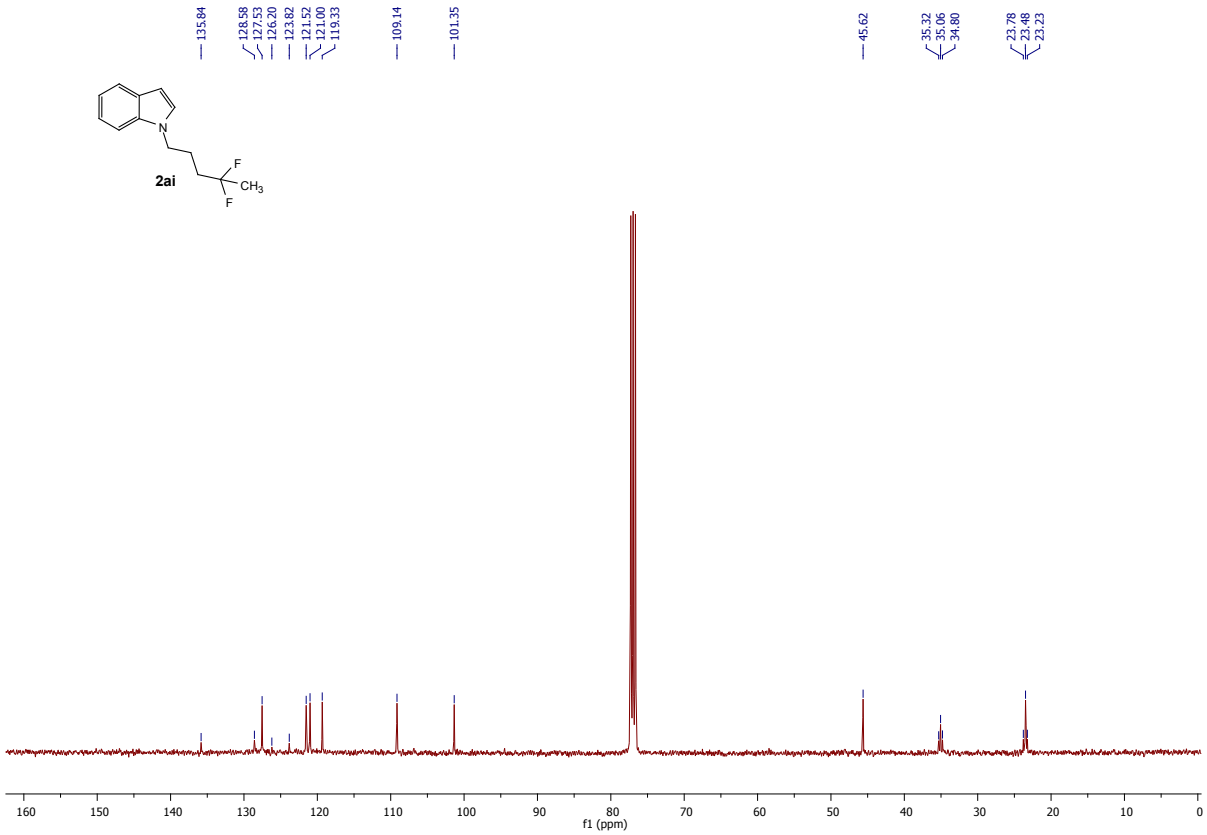

180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

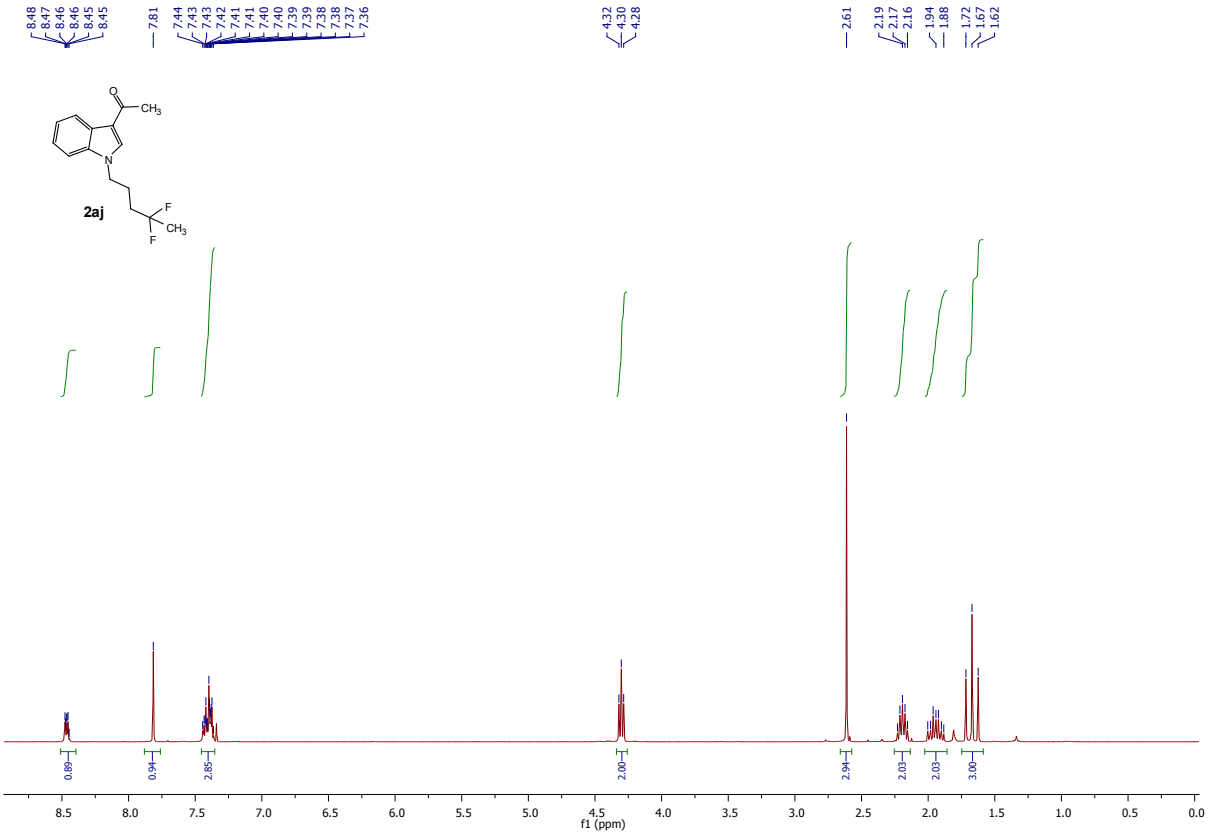

7.742
7.722
7.442
7.422
7.312
7.223
7.205
7.191
7.183
6.605
6.598
4.304
4.286
4.269
2.192
2.174
2.156
2.135
2.118
1.923
1.864
1.699
1.653
1.636
1.607
N
F
CH3
F
0.85
0.92
0.89
1.74
0.84
2.00
2.09
2.02
3.16
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)

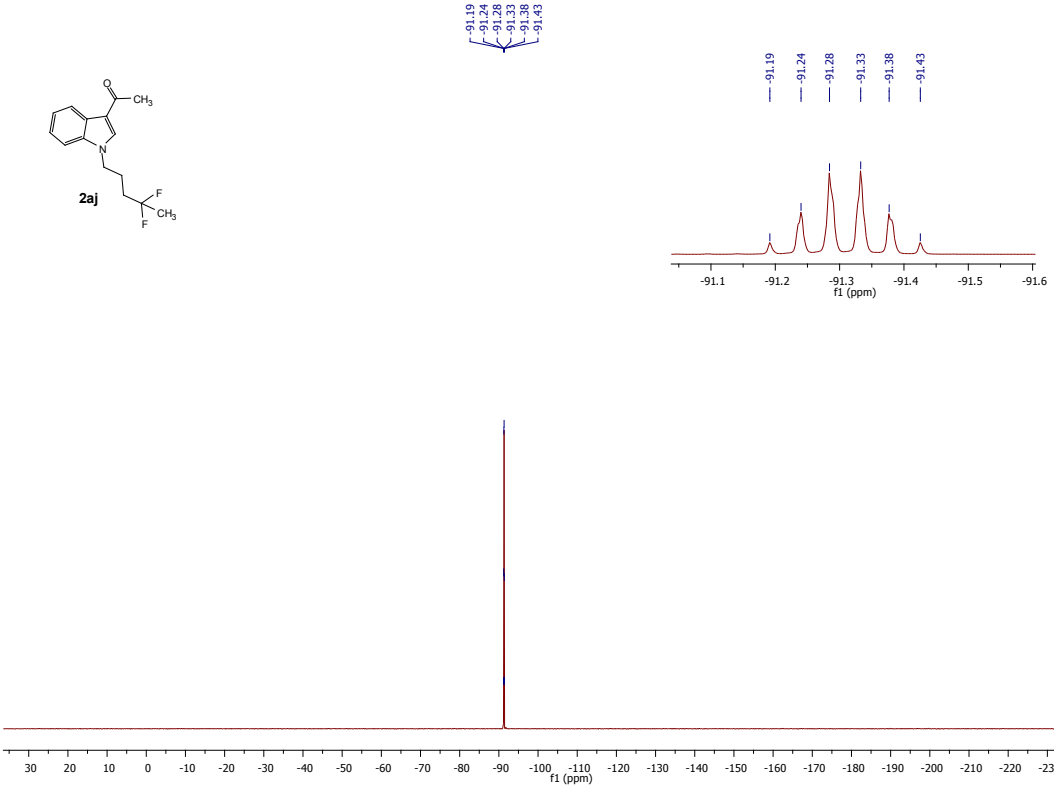
2ai

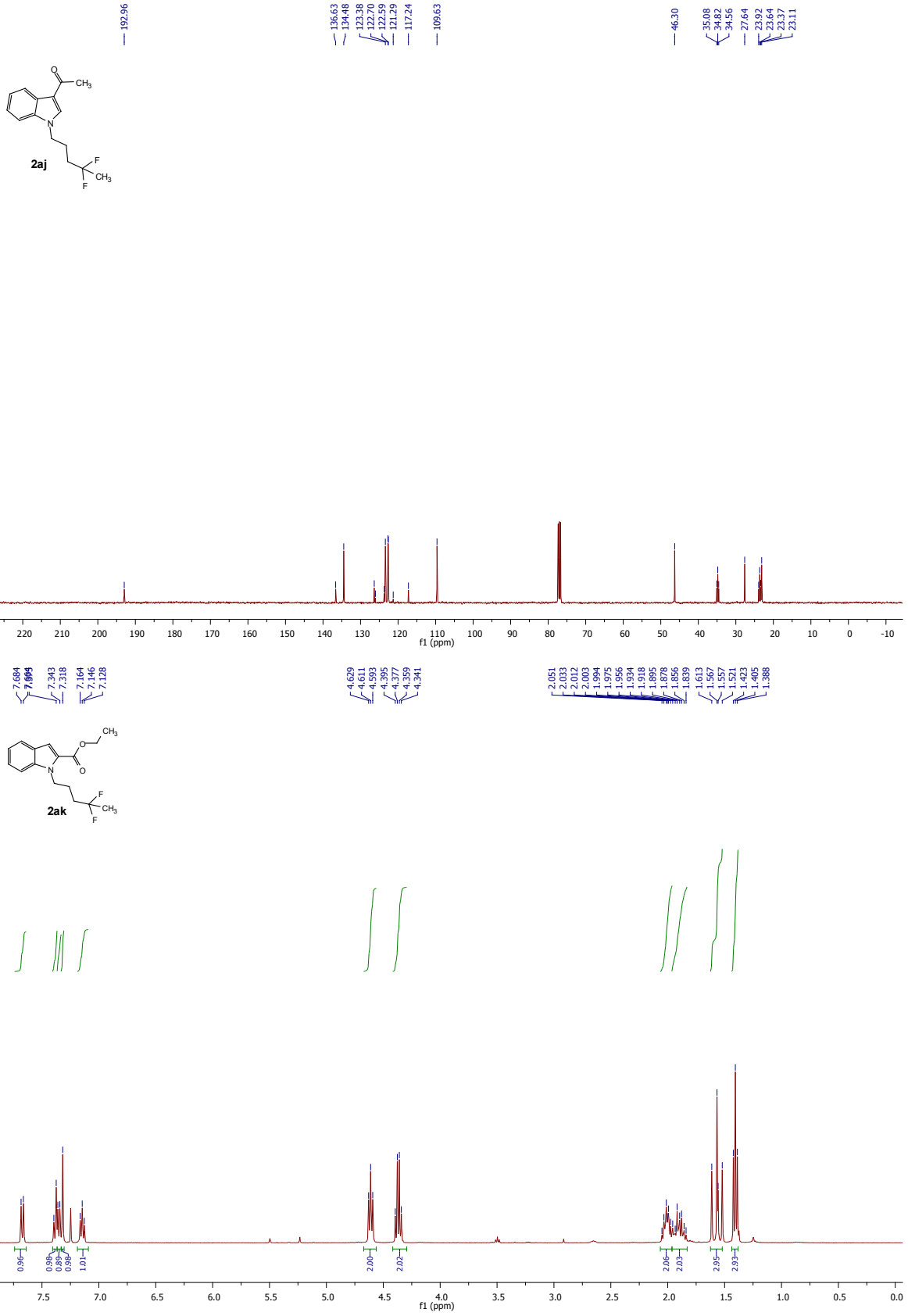

-91.11
-91.16
-91.20
-91.25
-91.30
-91.34
2ai
F
CH3
F
-90.9
-91.0
-91.1
-91.2
-91.3
-91.4
-91.5
-91.6
f1 (ppm)

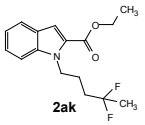

30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
-220
-230
-240
-250
f1 (ppm)


135.84
128.58
127.53
126.20
123.82
121.52
121.00
119.33
109.14
101.35
45.62
35.32
35.06
34.80
23.78
23.48
23.23
2ai
F
CH3
F
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

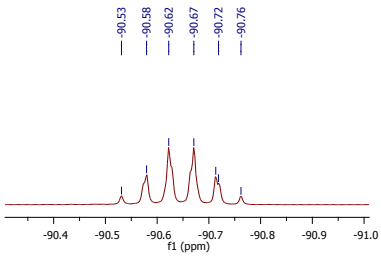

8.48
8.47
8.46
8.46
8.45
8.45
7.81
7.44
7.43
7.43
7.42
7.41
7.41
7.40
7.40
7.39
7.39
7.38
7.38
7.37
7.36
4.32
4.30
4.28
2.61
2.19
2.17
2.16
1.94
1.88
1.72
1.67
1.62
O
CH3
N
F
CH3
F
2aj
0.89
0.94
2.85
2.00
2.94
2.03
2.03
3.00
f1 (ppm)

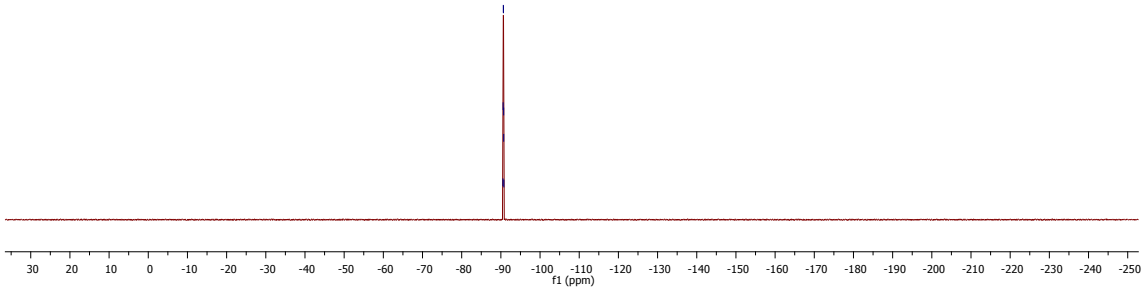

-91.19
-91.24
-91.28
-91.33
-91.38
-91.43
O
CH3
N
F
CH3
F
2aj
f1 (ppm)
f1 (ppm)


192.96
136.63
134.48
123.38
122.70
122.59
121.29
117.24
109.63
46.30
35.08
34.82
34.56
27.64
23.92
23.64
23.37
23.11
2aj
f1 (ppm)
7.684
7.343
7.318
7.164
7.146
7.128
4.629
4.611
4.593
4.395
4.377
4.359
4.341
2.051
2.033
2.012
2.003
1.994
1.975
1.956
1.934
1.918
1.895
1.878
1.856
1.839
1.613
1.567
1.557
1.521
1.423
1.405
1.388
2ak
0.96
0.98
0.89
0.98
1.01
2.00
2.02
2.06
2.03
2.95
2.93
f1 (ppm)

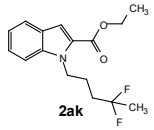

2ak

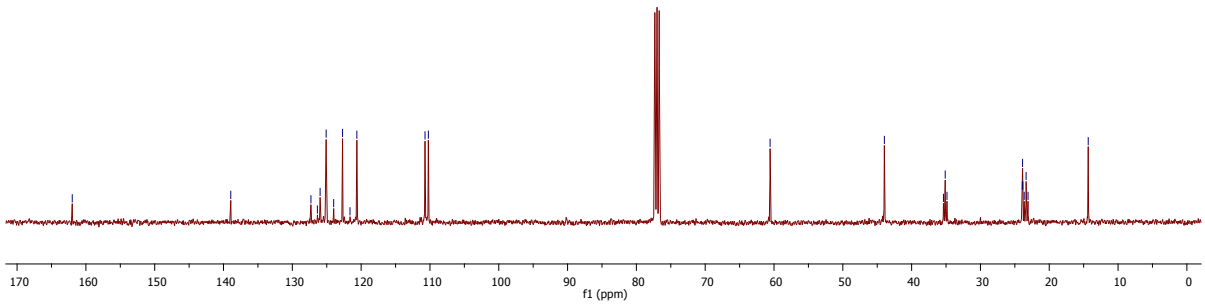

-90.53
-90.58
-90.62
-90.67
-90.71
-90.72
-90.76

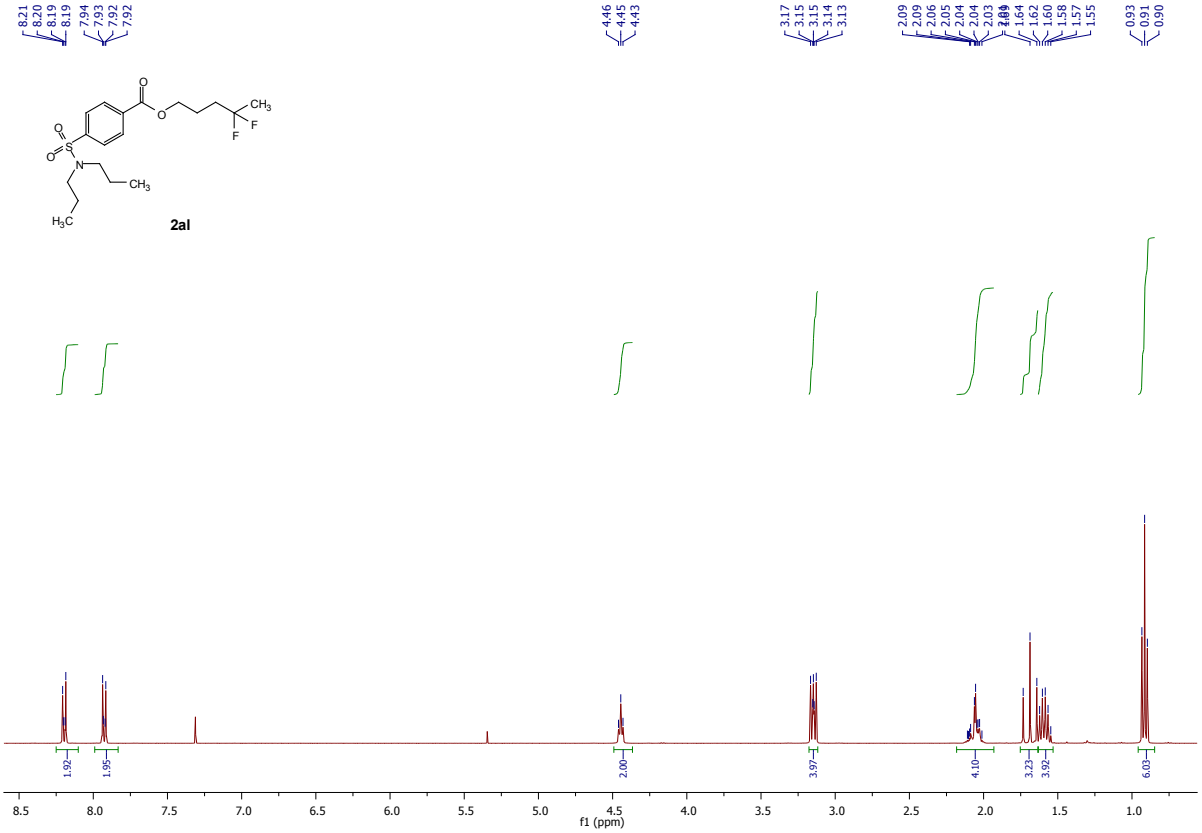

-90.53
-90.58
-90.62
-90.67
-90.72
-90.76
-90.4
-90.5
-90.6
-90.7
-90.8
-90.9
-91.0
f1 (ppm)

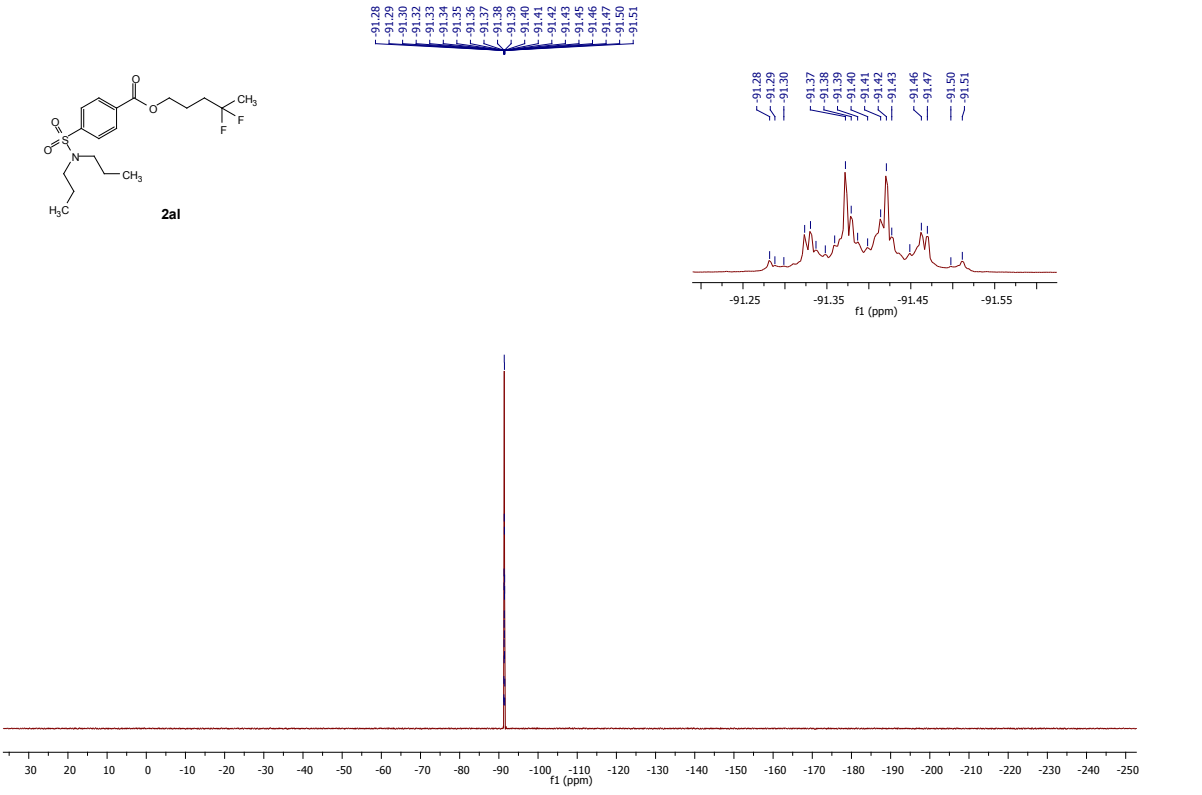

30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240 -250
f1 (ppm)


161.98
138.95
127.30
126.36
125.96
125.08
123.99
122.70
121.62
120.62
110.72
110.23
60.57
43.96
35.38
35.12
34.86
23.92
23.88
23.84
23.64
23.36
23.09
14.34


2ak


170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.21
8.20
8.19
8.19
7.94
7.93
7.92
7.92
4.46
4.45
4.43
3.17
3.15
3.15
3.14
3.13
2.09
2.09
2.06
2.05
2.04
2.04
2.03
1.64
1.62
1.60
1.58
1.57
1.55
0.93
0.91
0.90
2al
1.92
1.95
2.00
3.97
4.10
3.23
3.92
6.03
f1 (ppm)


-91.28
-91.29
-91.30
-91.32
-91.33
-91.34
-91.35
-91.36
-91.37
-91.38
-91.39
-91.40
-91.41
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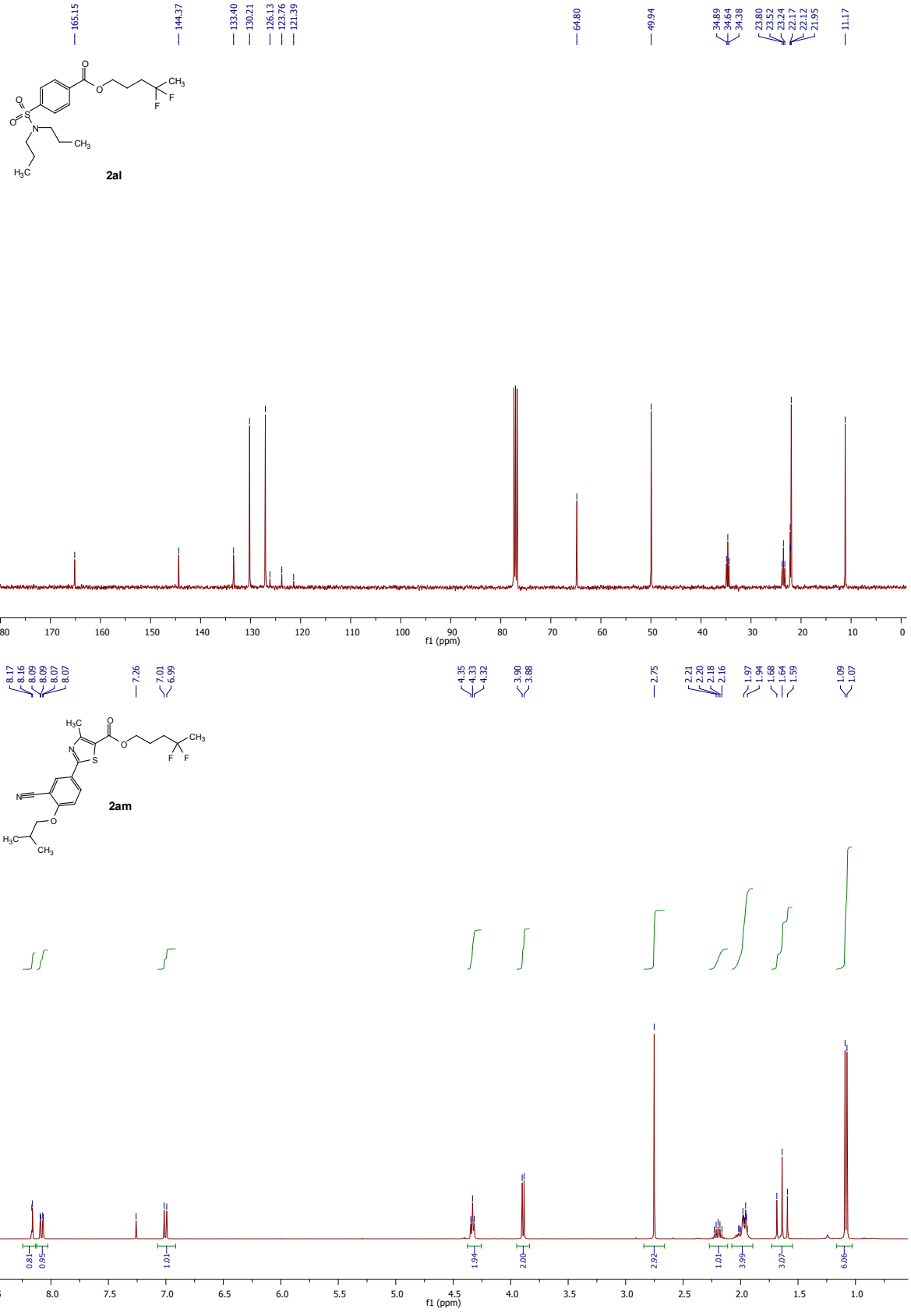
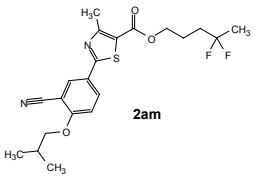
-91.45
-91.46
-91.47
-91.50
-91.51
2al
f1 (ppm)


165.15
144.37
133.40
130.21
126.13
123.76
121.39
64.80
49.94
34.89
34.64
34.38
23.80
23.52
23.24
22.17
22.12
21.95
11.17
2al
f1 (ppm)
8.17
8.16
8.09
8.09
8.09
8.07
8.07
7.26
7.01
6.99
4.35
4.33
4.32
3.90
3.88
2.75
2.21
2.20
2.18
2.16
1.97
1.94
1.68
1.64
1.59
1.09
1.07
2am
0.81
0.95
1.01
1.94
2.00
2.92
1.01
3.99
3.07
6.05
f1 (ppm)


2am


-91.28
-91.32
-91.33
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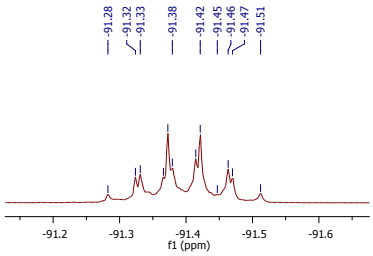
-91.38
-91.41
-91.42
-91.45
-91.46
-91.47
-91.51


-91.28
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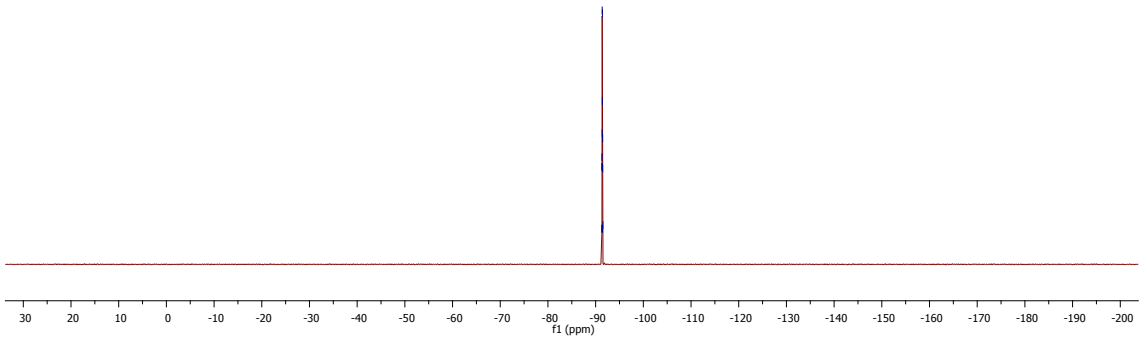
-91.33
-91.38
-91.42
-91.45
-91.46
-91.47
-91.51
-91.2
-91.3
-91.4
-91.5
-91.6
f1 (ppm)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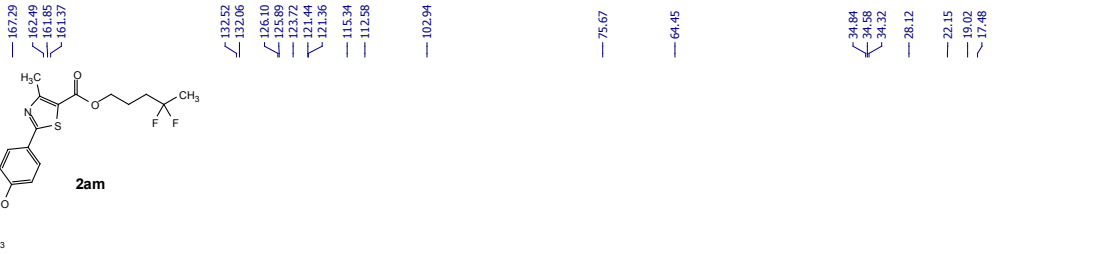
f1 (ppm)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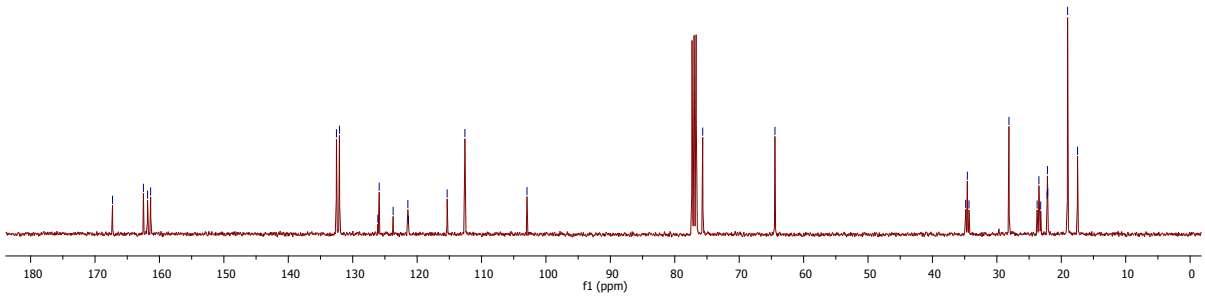
162.49
161.85
161.37
132.52
132.06
126.10
125.89
123.72
121.44
121.36
115.34
112.58
102.94
75.67
64.45
34.84
34.58
34.32
28.12
22.15
19.02
17.48

2am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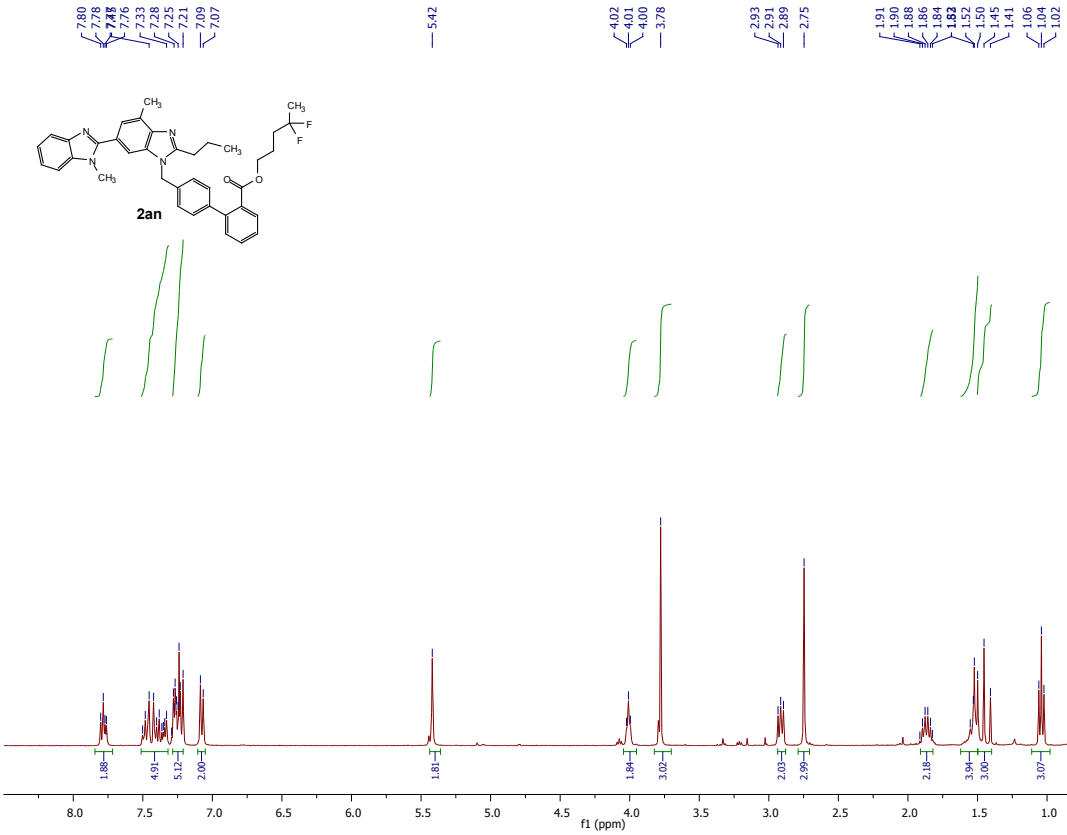
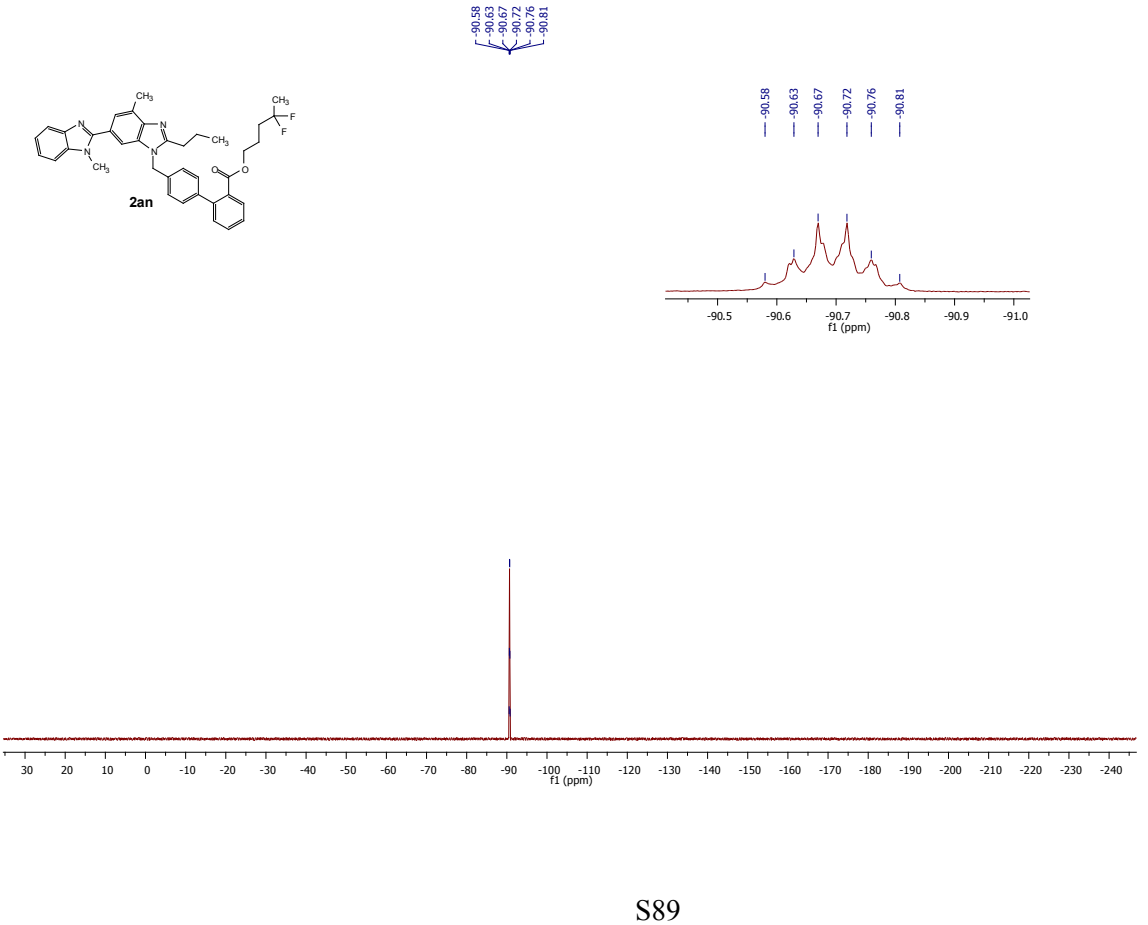
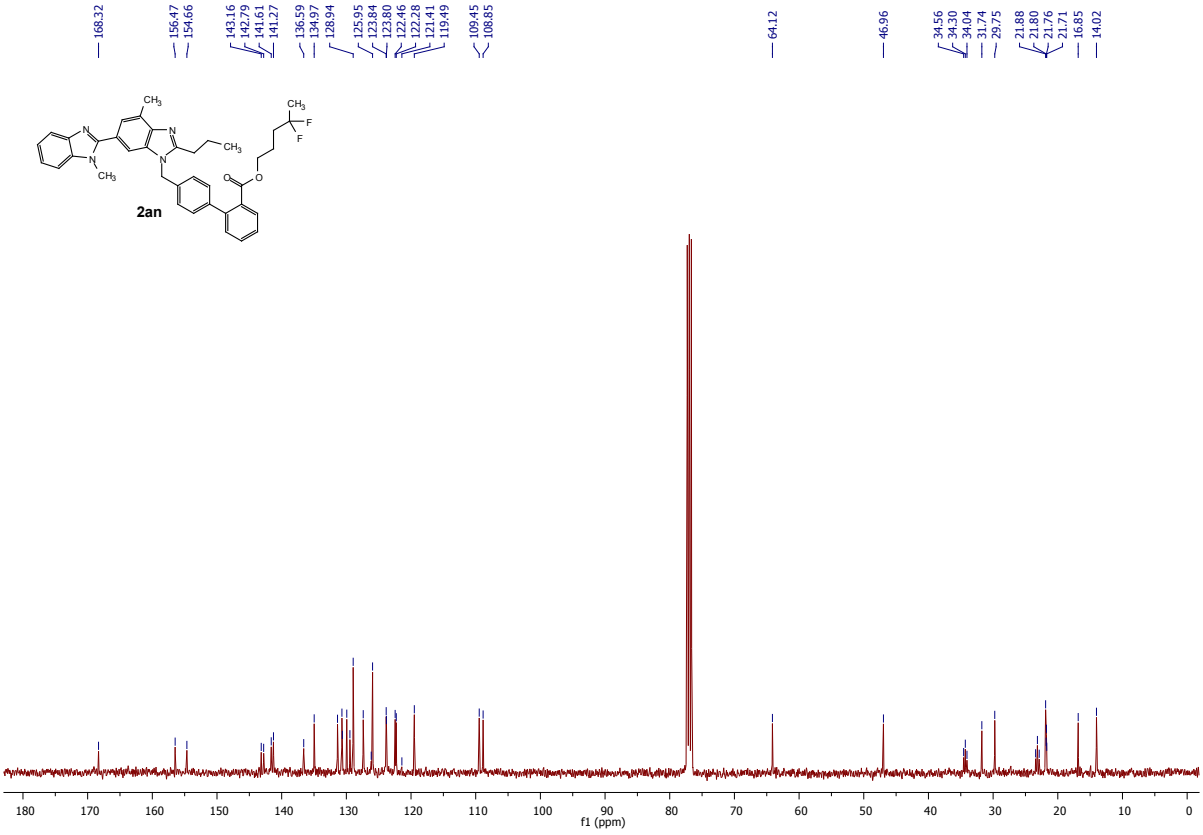
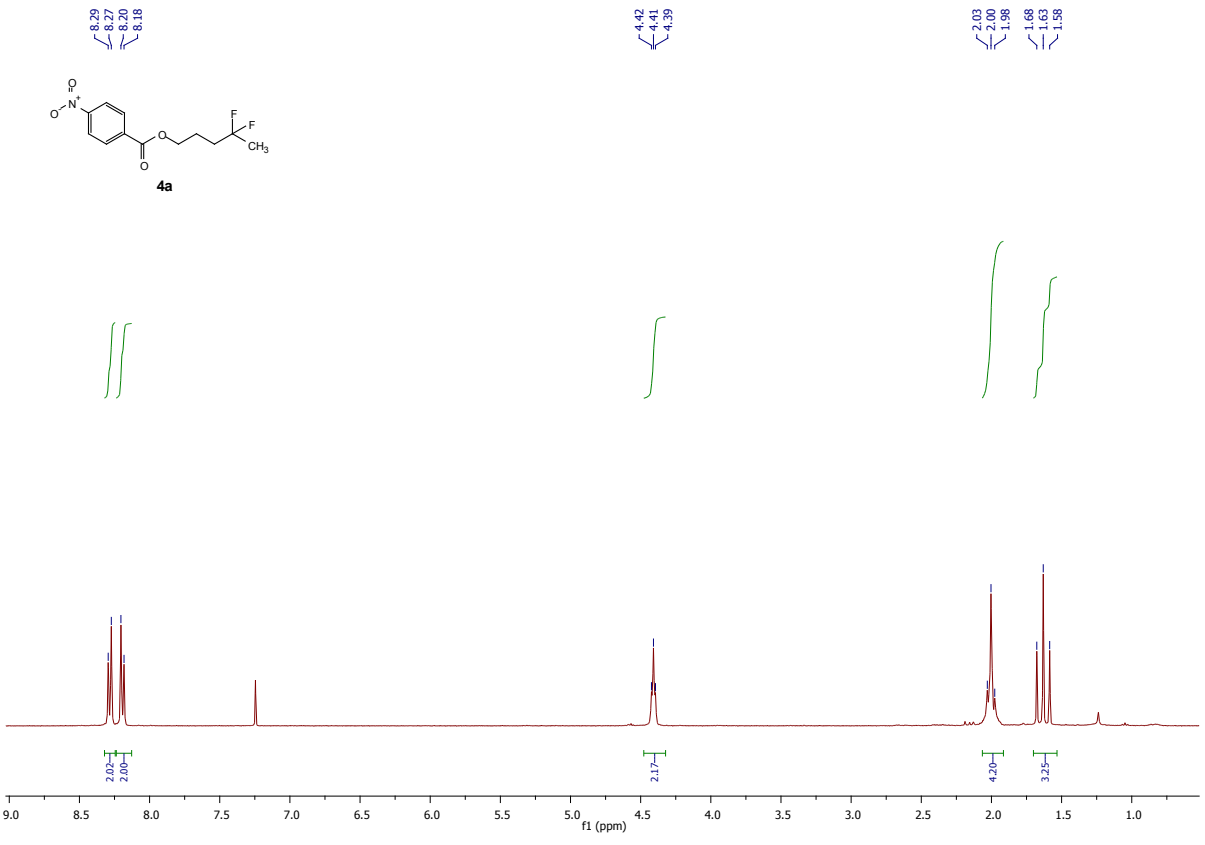
f1 (ppm)

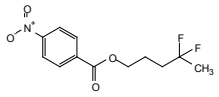

2an
f1 (ppm)


2an
f1 (ppm)
f1 (ppm)


168.32
156.47
154.66
143.16
142.79
141.61
141.27
136.59
134.97
128.94
125.95
123.84
123.80
122.46
122.28
121.41
119.49
109.45
108.85
64.12
46.96
34.56
34.30
34.04
31.74
29.75
21.88
21.80
21.76
21.71
16.85
14.02
2an
f1 (ppm)


8.29
8.27
8.20
8.18
4.42
4.41
4.39
2.03
2.00
1.98
1.68
1.63
1.58
4a
2.02
2.00
2.17
4.20
3.25
f1 (ppm)

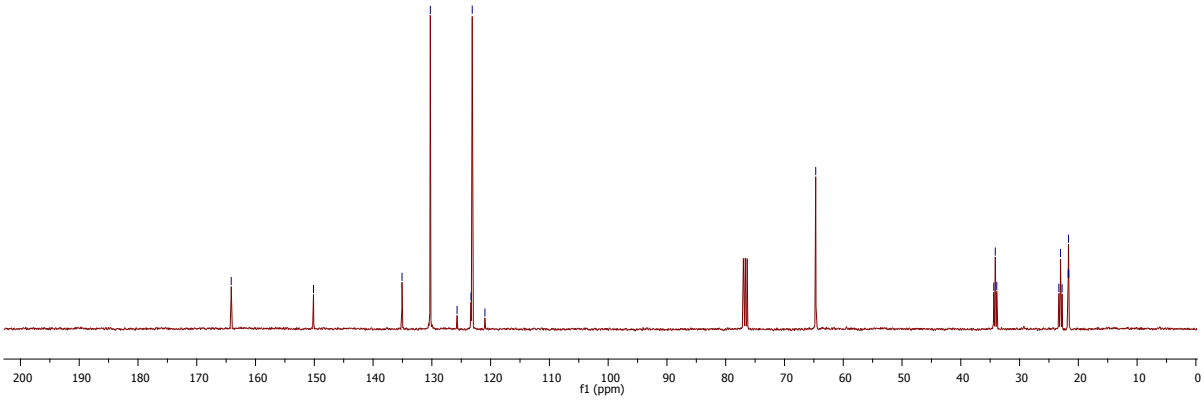
4a

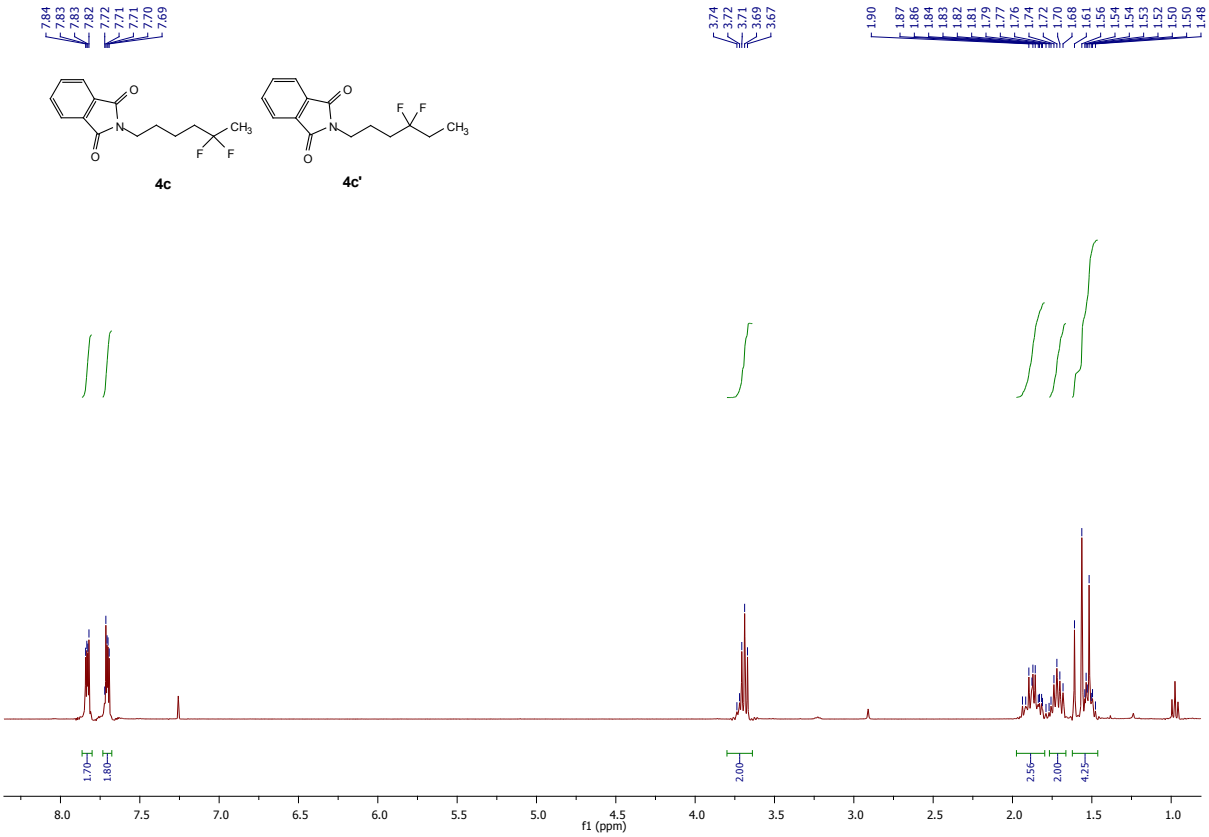


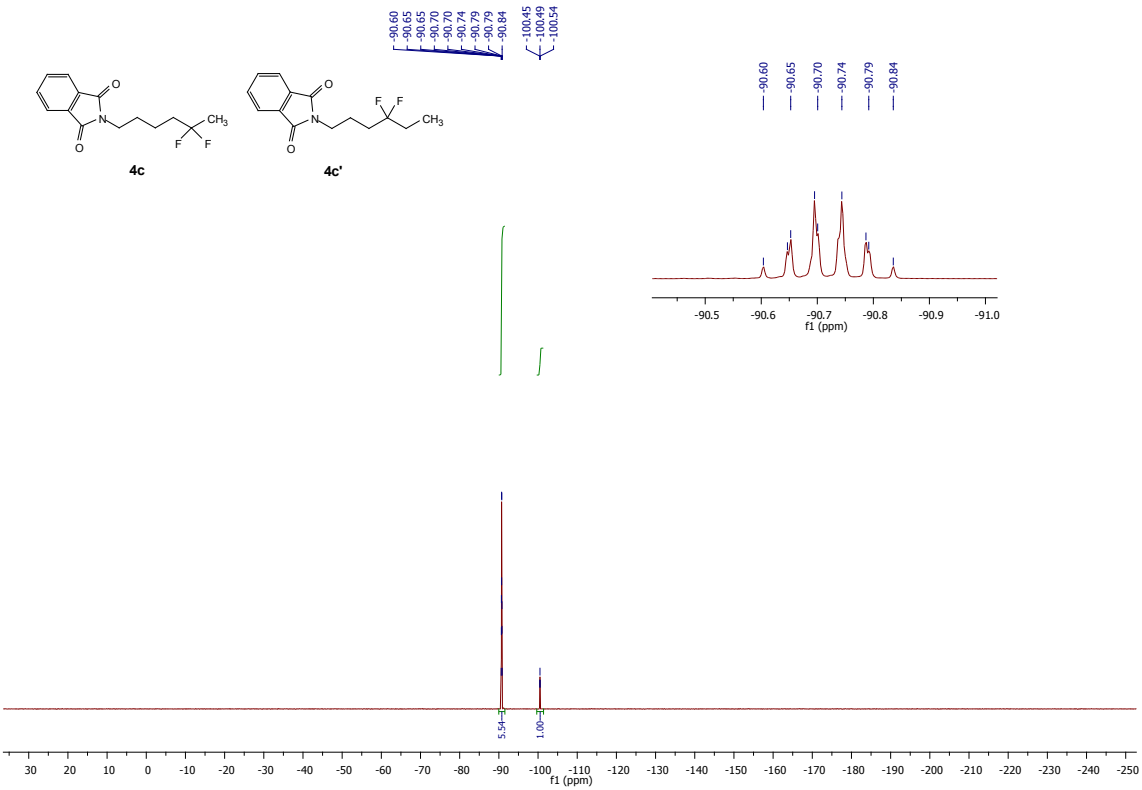
-91.42
-91.47
-91.51
-91.56
-91.60
-91.65
-99.84
-99.89
-99.93
62.29
1.00
f1 (ppm)

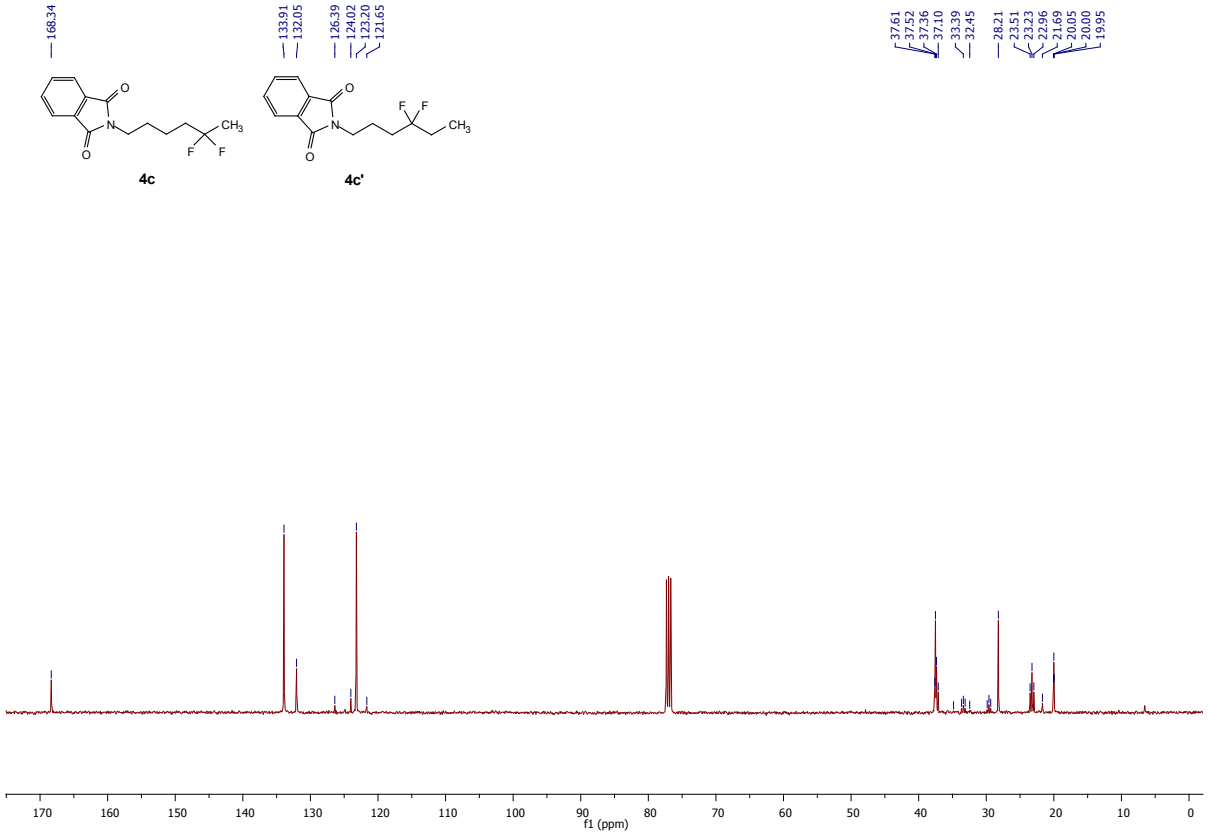

164.14
150.14
135.07
130.26
125.71
123.34
123.13
120.97
64.68
34.39
34.13
33.87
23.32
23.04
22.76
21.72
21.68
21.63

4a


f1 (ppm)


7.84
7.83
7.83
7.82
7.72
7.71
7.71
7.70
7.69
3.74
3.72
3.71
3.69
3.67
1.90
1.87
1.86
1.84
1.83
1.82
1.81
1.79
1.77
1.76
1.74
1.72
1.70
1.68
1.61
1.56
1.54
1.54
1.53
1.52
1.50
1.50
1.48
4c
4c'
1.70
1.80
2.00
2.56
2.00
4.25
f1 (ppm)


-90.60
-90.65
-90.65
-90.70
-90.70
-90.74
-90.79
-90.79
-90.84
-100.45
-100.49
-100.54
4c
4c'
-90.60
-90.65
-90.70
-90.74
-90.79
-90.84
f1 (ppm)
5.54
1.00
f1 (ppm)



Section 5. References

1. Z. Lu, X. Zeng, G. B. Hammond and B. Xu, *J. Am. Chem. Soc.*, 2017, **139**, 18202-18205.
2. O. E. Okoromoba, J. Han, G. B. Hammond and B. Xu, *J. Am. Chem. Soc.*, 2014, **136**, 14381-14384.
3. T. P. Curran, A. L. Grant, R. A. Lucht, J. C. Carter and J. Affonso, *Org. Lett.*, 2002, **4**, 2917-2920.
4. K. Semba, T. Fujihara, T. Xu, J. Terao and Y. Tsuji, *Adv. Synth. Catal.*, 2012, **354**, 1542-1550.
5. W. Hess and J. W. Burton, *Chem. Eur. J.*, 2010, **16**, 12303-12306.
6. K. Asano and S. Matsubara, *Org. Lett.*, 2009, **11**, 1757-1759.
7. Z. Wang, L. Chen, Y. Yao, Z. Liu, J.-M. Gao, X. She and H. Zheng, *Org. Lett.*, 2018, **20**, 4439-4443.
8. S. A. Snyder, D. A. Vosburg, M. G. Jarvis and J. H. Markgraf, *Tetrahedron*, 2000, **56**, 5329-5335.
9. J. Kuang and S. Ma, *J. Org. Chem.*, 2009, **74**, 1763-1765.