Supporting Information for

Photo-induced Decarboxylative Coupling Reaction between Aliphatic *N*-hydroxyphthalimide Esters and Terminal 2-Trifluoromethylalkenes

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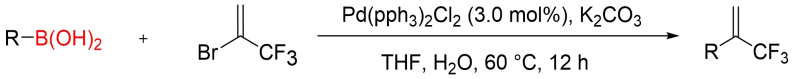
1. General information

^{1}H NMR and ^{13}C NMR spectra were recorded on an Agilent MR400 spectrometer. ^{19}F NMR was recorded on an Agilent MR400 spectrometer. Chemical shifts (δ) are reported in ppm and coupling constants (J) are in Hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. High-resolution mass spectra were recorded on a Thermo Scientific Q Exactive HF with Fourier-transform (orbitrap) mass spectrometer and Agilent Technologies 7250 GCQTOF in laboratory of mass spectrometry analysis at Shanghai Institute of Organic Chemistry and Guizhou Natural Products Research Center. Melting points were taken on a SGW X-4 Melting Point Apparatus.

Materials: All reagents were either used directly as received from commercial sources or prepared according to the descriptions in the literature. They were all weighed and handled in air at room temperature.

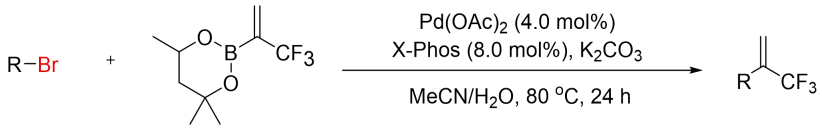
2. General procedure for the synthesis of trifluoromethyl alkenes

Method A[1]**:**



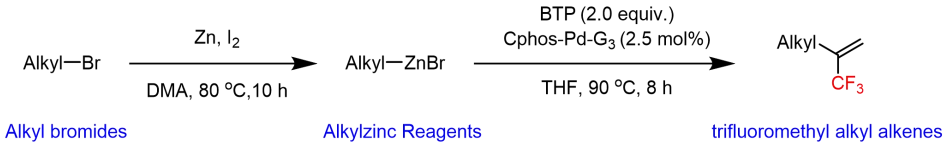
To a 25 mL Schlenk tube was added aryl boric acid (3.0 mmol, 1.0 equiv.), potassium carbonate (12.0 mmol, 4.0 equiv.), bis(triphenylphosphine)palladium(II) chloride (0.09 mmol, 3 mol%). Then removed air under vacuum and charged with Ar (3 times). The tube was added THF (9.0 mL), 2-bromo-3,3,3- trifluoropropene (4.5 mmol, 1.5 equiv.), degassed deionized H_2O (6.0 mL) subsequently and sealed tightly. The reaction was heated to 60 ºC for 12 hours. The mixture was cooled to room temperature, extracted with ethyl acetate and remove solvent under vacuum. The residue was purified through column chromatography utilizing silica gel, with a mixture of ethyl acetate and petroleum ether serving as the eluent. The trifluoromethyl alkenes employed in the synthesis of **3a-3c**, **3e**, **3f**, **3h**, **3i**, **3k** were prepared in accordance with method A.

Method B[2]**:**



To a 25 mL Schlenk tube was added aryl brmide (1.0 mmol, 1.0 equiv.), X-Phos (0.08 mmol, 8 mol%), potassium carbonate (414.6mg, 3.0 mmol, 3.0 equiv.), Palladium (II) Acetate (0.04 mmol, 4 mol%). The reaction mixture was evacuated and backfilled with Ar (3 times). The tube was added 4,4,6-trimethyl-2-(3,3,3-trifluoroprop-1-en-2-yl)-1,3,2-dioxaborinane (1.5 equiv.), MeCN (3.0 mL), degassed deionized H_2O (1.5 mL) subsequently and sealed tightly. The tube was screw-capped, and the reaction mixture was stirred for 48 h at 80 °C. The mixture was cooled to room temperature, extracted with ethyl acetate and remove solvent under vacuum. The residue was purified through column chromatography utilizing silica gel, with a mixture of ethyl acetate and petroleum ether serving as the eluent. The trifluoromethyl alkenes employed in the synthesis of **3d**, **3g**, **3j**, **3h**, **3l-3n** were prepared in accordance with method B.

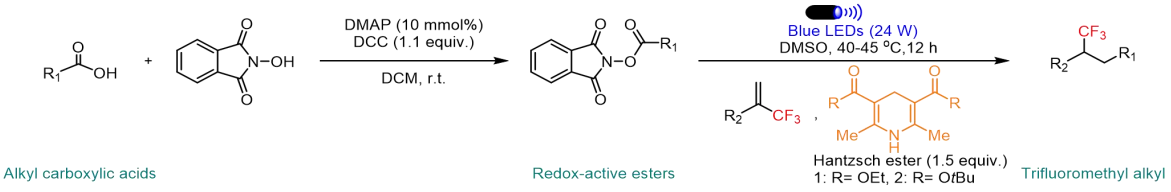
Method C[3]**:**



step 1: A 25 mL of Schlenk tube was charged with zinc powder (295.0 mg, 4.5 mmol) and heated to 80 °C under vacuum for 30 min. After the tube was back-filled with argon and cooled to room temperature, iodine (38.0 mg, 0.15 mmol) and DMA (3.0 mL) were added. The resulting mixture was stirred until the brown color disappeared, then alkylbromide (3.0 mmol) was added. The reaction mixture was heated to 80 °C. After stirring for 10 h at 80 °C, the mixture was cooled to room temperature. The gray solution was filtered and the filtrate was stored under argon in a Schlenk tube, the solution of the alkylzinc reagent was titrated with I_2 according to Knochel's method.

General Procedure for the Palladium Catalyzed Cross-Coupling of Alkylzinc Reagents With 2-Bromo-3, 3, 3-trifluoropropane (**step 2**): To a 25 mL of Schlenk tube was added Cphos-Pd-G_3 (4.0 mg, 2.5 mol%). The tube was evacuated and backfilled with argon for three times, then alkylzinc reagent (0.3 mmol, 1.0 equiv), 2-bromo-3, 3, 3-trifluoropropane (0.6 mmol, 2.0 equiv) and THF (2.0 mL) were added. The Schlenk tube was screw capped and put into a preheated oil bath (90 °C). After stirring for 8 h, the reaction mixture was cooled to room temperature. The reaction mixture was diluted with EtOAc (50.0 mL) and filtered with a pad of cellite. The filtrate was washed with water (15.0 mL). The organic layer was dried over Na_2SO_4, filtered and concentrated. The residue was purified with silica gel chromatography to give the pure product. The trifluoromethyl alkenes employed in the synthesis of **3o-3r** were prepared in accordance with method B.

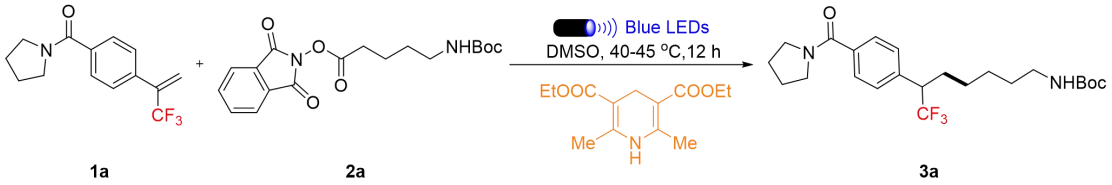
3. General procedure for the decarboxylative coupling reaction between aliphatic *N*-hydroxyphthalimide esters and terminal 2-trifluoromethylalkenes



Redox-active esters were prepared according to our previous reported procedures (**step 1**)[4]. (**step 2**): A 25 mL oven-dried Schlenk tube equipped with a magnetic stirrer bar was charged with the redox active esters (0.4 mmol, 2.0 equiv), Hantzsch ester (0.3 mmol, 1.5 equiv). The tube was evacuated and backfilled with argon for three times, followed by the addition of dry DMSO (2.0 mL) and trifluoromethyl alkenes (0.2 mmol, 1.0 equiv). The tube was screw capped and heated to 40 °C under irradiation of 24 W blue LEDs. After stirred for 12 h, the reaction mixture was diluted with EtOAc (150.0 mL) washed with water and brine, dried over anhydrous Na_2SO_4,

filtered and concentrated under vacuum. The product was purified through silica gel chromatography, resulting in the corresponding pure product.

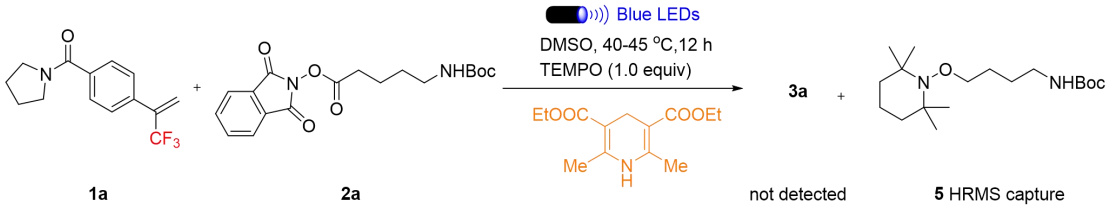
4. Detailed procedure for the gram scale synthesis



A 100.0 mL oven-dried Schlenk tube equipped with a magnetic stirrer bar was charged with **2a** (7.5 mmol, 1.5 equiv), Hantzsch ester (7.5 mmol, 1.5 equiv). The tube was evacuated and backfilled with argon for three times, followed by the addition of dry DMSO (50.0 mL) and **1a** (5.0 mmol, 1.0 equiv). The tube was screw capped and heated to 40-45 ℃ (heat was produced by LEDs) under irradiation of blue LEDs. After stirring for 12 h, the reaction mixture was diluted with ethyl acetate. The organic layer was washed with brine, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The product **3a** (2.1272 g, 96% yield) was purified with silica gel chromatography to give corresponding pure product.

5. Mechanism studies

5.1 Addition of radical and SET inhibitors



A 25.0 mL oven-dried Schlenk tube equipped with a magnetic stirrer bar was charged with the **2a** (0.3 mmol, 1.5 equiv), Hantzsch ester (0.3 mmol, 1.5 equiv), TEMPO (0.2 mmol, 1.0 equiv). The tube was evacuated and backfilled with argon for three times, followed by the addition of dry DMSO (0.5 mL) and **1a** (0.1 mmol, 1.0 equiv). The tube was screw capped and heated to 40-45 °C (heat was produced by LED) under irradiation of blue LEDs. After stirring for 12 h, the reaction mixture was cooled to room temperature, the reaction solution was diluted with 2.0 mL ethyl acetate,

pretreatment and monitored by TLC and sent to HRMS. The results indicated that the reaction was totally suppressed by the addition of a radical scavenger TEMPO, which suggests that the involvement of radical intermediates is likely during the reaction.

5.2 High resolution ESI-MS and MS/MS experiments for detecting TEMPO complex

High resolution ESI-MS and MS/MS spectra were recorded on a Q Exactive HF Orbitrap mass spectrometer (Thermo Fisher Scientific Inc.) equipped with ESI ion source. The ESI conditions were: spray voltage 3500 V; capillary temperature, 275 °C; sheath gas flow rate 35 arb. units. Data acquisition and analysis were done with the Thermo Xcalibur (version 4.2.47) software package.

The elemental composition analysis of the ion at *m/z* 328.2729 by HRMS (Figure S1) supported the proposed structure of TEMPO complex.

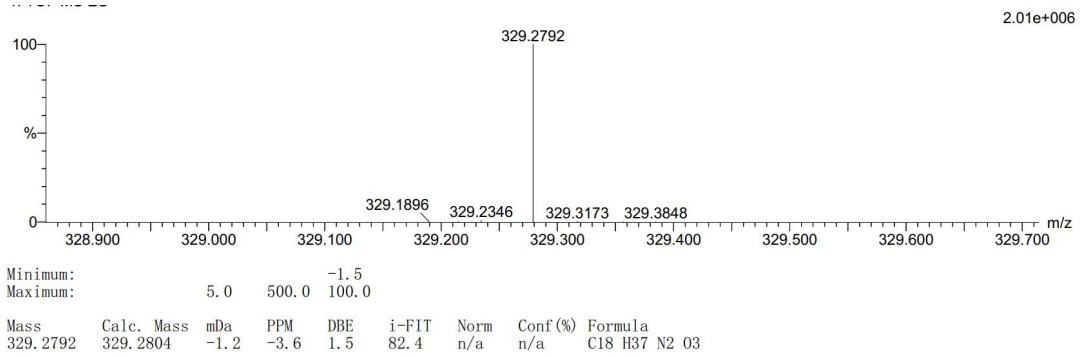
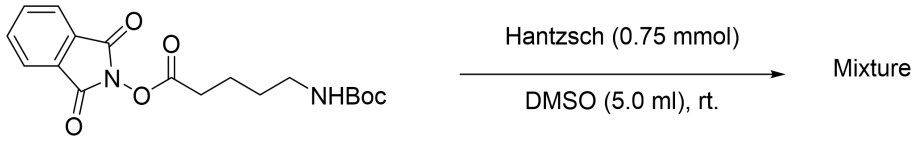


Figure S1. High resolution ESI-MS spectrum of TEMPO complex calculated for $C_{18}H_{37}N_2O_3$ ($[M+H]^+$): 329.2804; Found: 329.2792.

5.3 UV-vis experiments



2a (0.75 mmol)

UV-vis absorption spectra of the reaction mixture in DMSO were measured to provide information on formation of EDA complex.

Solution 1: **2a** (271.8 mg, 0.75 mmol) was added in DMSO (5.0 mL). The mixture was stirred for 30 minutes under natural light conditions and then filtered.

Solution 2: HE (190.0 mg, 0.75 mmol) was added in DMSO (5.0 mL). The mixture

was stirred for 30 minutes under natural light conditions and then filtered.

Solution 3: **2a** (271.8 mg, 0.75 mmol) and HE (190.0 mg, 0.75 mmol) were added in DMSO (5.0 mL). The mixture was stirred for 30 minutes under natural light conditions and then filtered.

Performed on UV visible spectrophotometer, recorded in 1 cm path quartz cuvettes using T6 Xinyue visible spectrophotometer (PERSEETM).

A / λ/nm	**2a**+DMSO	HE+DMSO	**2a** + HE+DMSO
330	2.028	2.235	2.787
340	2.062	2.305	2.593
350	1.420	2.502	2.591
360	0.912	2.386	2.731
370	0.857	2.122	2.585
380	0.397	2.271	2.694
390	0.169	2.221	2.794
400	0.071	2.169	2.453
410	0.044	2.028	2.432
420	0.036	1.933	2.274
430	0.028	1.986	2.345
440	0.022	1.262	1.988
450	0.018	0.256	1.129
460	0.017	0.015	0.675
470	0.016	0.001	0.447
480	0.015	0.001	0.273
490	0.014	0.001	0.170
500	0.012	0.001	0.102
510	0.011	0.001	0.059
520	0.009	0.001	0.035
530	0.008	0.000	0.021
540	0.007	0.000	0.012
550	0.007	0.000	0.008
560	0.006	0.000	0.005

570	0.006	0.000	0.004
580	0.005	0.000	0.003
590	0.005	0.000	0.002
600	0.005	0.000	0.000

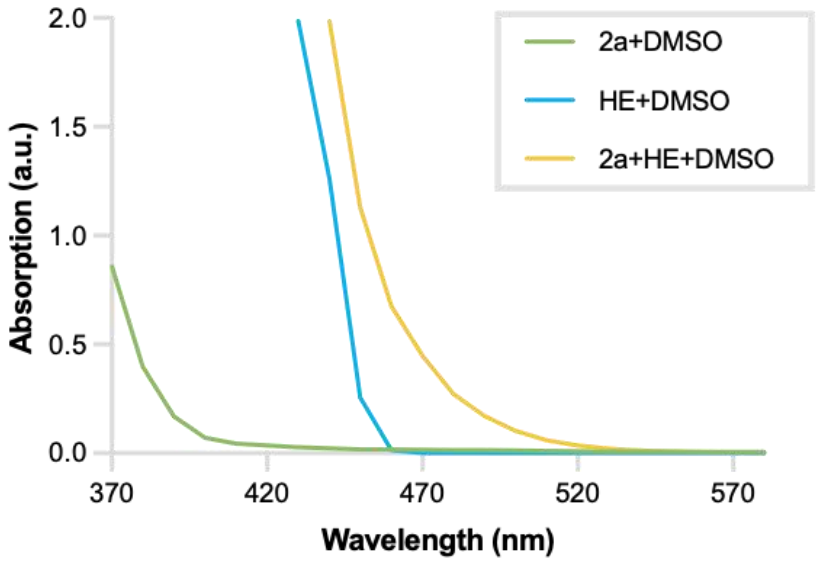


Figure S2. Optical absorption spectra studies.

6. Materials and methods of tumor cell growth inhibition test

6.1 Cell lines and culture condition

SK-Hep-1 cells (catalog number CL-0212, Wuhan, China) and A549 cells (catalog number STCC10201, Wuhan, China) were purchased from Wuhan Proxel Life Science and Technology Co., Ltd. and Wuhan Sewell Biotechnology Co., Ltd., respectively, and the complete medium consisted of MEM and Ham's F-12K basal medium(Servicebio Technology Co., Ltd., catalog numbers G4556-500 and G4560-500, Wuhan, China), 1% penicillin and streptomycin(Cellcook Biotech Co., Ltd., catalog numbers CM1005-005, Guangzhou, China), and 10% FBS (Procell Life Science&Technology Co., Ltd., catalog numbers 164210-50, Wuhan, China), respectively. The incubation environment was 37 °C and 5 per cent CO_2.

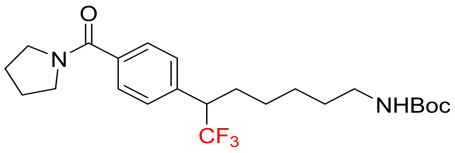
6.2 Tumour cell growth inhibition assay

In vitro experiments were performed using SK-Hep-1 and A549 cells during the

logarithmic growth phase. SK-Hep-1 and A549 cells were seeded into 96-well plates at a density of 5 × 10^3 cells per well. A range of concentrations of compounds (0, 2, 4, 8, 16, 32, and 64 μM) were used to assess proliferative effects on SK-Hep-1 and A549 cells. After 48 h of incubation, add 100 μl of CCK-8-containing solution (medium to CCK-8 in a 9:1 ratio) (APExBIO, catalog number K1018, USA) and incubate at 37 °C for 1.5 h. The corresponding absorbance was detected at a wavelength of 450 nm. Absorbance values were converted to cell viability (%) by Microsoft Excel and visualized by GraphPad Prism 10 software.

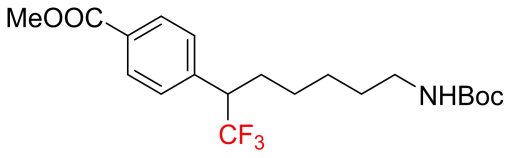
7. Characterization data for the products

tert-butyl (7,7,7-trifluoro-6-(4-(pyrrolidine-1-carbonyl)phenyl)heptyl)carbamate (3a)



Obtained in 98% yield as a colorless oily liquid (86.7 mg, eluent: PE/EA = 2:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.48 (d, J = 8.0 Hz, 2H), 7.27 (d, J = 8.0 Hz, 2H), 4.53 (s,1H), 3.61 (t, J = 6.4 Hz, 2H), 3.41 (t, J = 6.4 Hz, 2H), 3.24 – 3.15 (m, 1H), 3.03 – 2.98 (m, 2H), 1.89 (dt, J = 35.2, 6.8 Hz, 6H), 1.39 (s, 9H), 1.36 – 1.32 (m, 2H), 1.28 – 1.20 (m, 2H), 1.16 – 1.12 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.3, 156.1, 137.1, 136.6, 129.0, 127.6, 126.8 (q, J = 280.8 Hz), 79.1, 49.9 (q, J = 26.7 Hz), 49.7, 46.3, 40.5, 29.9, 28.6, 28.5, 26.4, 24.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.60 – -69.73 (m, 3F). **HRMS (ESI)**: calculated for $C_{23}H_{34}O_3N_2F_3$ ($[M+H]^+$): 443.2516; Found: 443.2521.

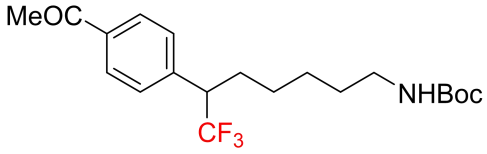
methyl 4-(7-((_tert_-butoxycarbonyl)amino)-1,1,1-trifluoroheptan-2-yl)benzoate (3b)



Obtained in 84% yield as a colorless oily liquid (67.8 mg, eluent: PE/EA = 30:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.02 (d, J = 8.0 Hz, 2H), 7.35 (d, J = 8.0 Hz, 2H), 4.48 (s, 1H), 3.91 (s, 3H), 3.32 – 3.22 (m, 1H), 3.06 – 3.01 (m, 2H), 2.05 – 1.97 (m, 1H), 1.91– 1.81 (m, 1H), 1.42 (s, 9H), 1.39 – 1.35 (m, 2H), 1.33 – 1.22 (m, 2H), 1.21 – 1.11(m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.8, 156.1, 140.0 (d, J = 2.0 Hz), 130.2, 130.0 (d, J = 4.6 Hz), 129.2, (d, J = 13.0 Hz),

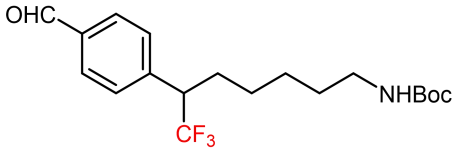
126.7 (q, J = 281.3 Hz), 79.2, 52.3 (d, J = 16.7 Hz), 50.1 (dd, J = 26.0, 4.5 Hz), 40.4, 29.9, 28.7, 28.5, 28.4, 26.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -70.14 (d, J = 8.6 Hz, 3F). **HRMS (ESI)**: calculated for $C_{20}H_{28}O_4NF_3Na$ ($[M+Na]^+$): 426.1863; Found: 426.1862.

***tert*-butyl (6-(4-acetylphenyl)-7,7,7-trifluoroheptyl)carbamate (3c)**



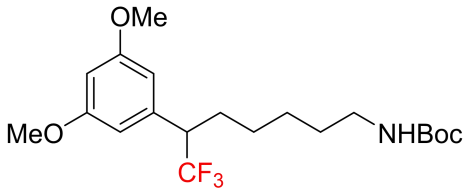
Obtained in 76% yield as a colorless oily liquid (58.9 mg, eluent: PE/EA = 5:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.92 (dd, J = 8.0, 2.0 Hz, 2H), 7.35 (d, J = 7.6 Hz, 2H), 4.55 (s, 1H), 3.32 – 3.22 (m, 1H), 3.04 – 2.99 (m, 2H), 2.58 (d, J = 1.6 Hz, 3H), 2.02 – 1.95 (m, 1H), 1.90 – 1.80 (m, 1H), 1.40 (s, 9H), 1.36 – 1.32 (m, 2H), 1.31 – 1.21 (m, 2H), 1.20 – 1.08 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.7, 156.1, 140.2 (d, J = 2.1 Hz), 137.0, 129.3 (d, J = 13.7 Hz), 128.8 (d, J = 1.9 Hz), 126.6 (q, J = 281.1 Hz), 79.2, 50.1 (dd, J = 26.1, 5.5 Hz), 40.4, 29.8, 28.5, 28.4 (d, J = 8.9 Hz), 26.8, 26.6, 26.4. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.50 (d, J = 9.0 Hz, 3F). **HRMS (ESI)**: calculated for $C_{20}H_{28}O_3NF_3Na$ ($[M+Na]^+$): 410.1913; Found: 410.1917.

***tert*-butyl (7,7,7-trifluoro-6-(4-formylphenyl)heptyl)carbamate (3d)**



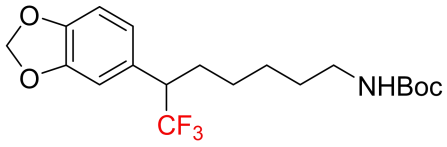
Obtained in 84% yield as a colorless oily liquid (62.7 mg, eluent: PE/EA = 5:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.01 (s, 1H), 7.87 (d, J = 8.4 Hz, 2H), 7.45 (d, J = 8.0 Hz, 2H), 4.52 (s, 1H), 3.36 – 3.25 (m, 1H), 3.06 – 3.01 (m, 2H), 2.06 – 1.98 (m, 1H), 1.92 – 1.82 (m, 1H), 1.41 (s, 9H), 1.39 – 1.34 (m, 2H), 1.32 – 1.23 (m, 2H), 1.20 – 1.07 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 191.9 (d, J = 3.3 Hz), 156.1, 141.7 (d, J = 2.2 Hz), 136.3, 130.1, 129.8 (d, J = 10.4 Hz), 126.6 (q, J = 281.6 Hz), 79.2, 50.3 (q, J = 27.1 Hz), 40.4, 29.9, 28.7, 28.5, 28.4, 26.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.42 (d, J = 8.3 Hz, 3F). **HRMS (ESI)**: calculated for $C_{19}H_{26}O_3NF_3Na$ ($[M+Na]^+$): 396.1757; Found: 396.1761.

***tert*-butyl (6-(3,5-dimethoxyphenyl)-7,7,7-trifluoroheptyl)carbamate (3e)**



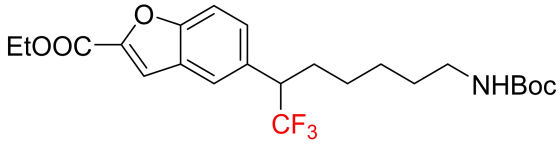
Obtained in 54% yield as a colorless oily liquid (43.7 mg, eluent: PE/EA = 10:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.41 (s, 3H), 4.49 (s, 1H), 3.79 (s, 6H), 3.16 – 3.09 (m, 1H), 3.08 – 3.02 (m, 2H), 1.98 – 1.89 (m, 1H), 1.85 – 1.75 (m, 1H), 1.42 (s, 9H), 1.41 – 1.35 (m, 2H), 1.34 – 1.25 (m, 2H), 1.23 – 1.16 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 160.9, 156.1, 137.2 (d, J = 2.1 Hz), 127.0 (q, J = 281.2 Hz), 107.3 (d, J = 10.7 Hz), 99.6 (d, J = 10.6 Hz), 79.2, 55.4 (q, J = 13.7 Hz), 50.3 (dd, J = 26.6, 8.5 Hz), 40.5, 29.9, 28.8, 28.5 (d, J = 7.3 Hz), 26.6, 26.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.62 (s, 3F). **HRMS (ESI)**: calculated for $C_{20}H_{30}O_4NF_3Na$ ($[M+Na]^+$): 428.2019; Found: 428.2018.

***tert*-butyl (6-(benzo[d][1,3]dioxol-5-yl)-7,7,7-trifluoroheptyl)carbamate (3f)**



Obtained in 34% yield as a colorless oily liquid (26.5 mg, eluent: PE/EA = 20:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.78 – 6.76 (m, 2H), 6.70 (d, J = 9.2 Hz, 1H), 5.97 (d, J = 0.8 Hz, 2H), 4.47 (s, 1H), 3.16 – 3.08 (m, 1H), 3.07 – 3.03 (m, 2H), 1.98 – 1.89 (m, 1H), 1.82 – 1.72 (m, 1H), 1.43 (s, 9H), 1.41 – 1.35 (m, 2H), 1.34 – 1.25 (m, 2H), 1.23 – 1.14 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.0, 148.0, 147.5, 128.4, 127.0 (q, J = 280.9 Hz), 122.9, 108.7 (d, J = 6.7 Hz), 108.4 (d, J = 3.8 Hz), 101.3 (t, J = 19.2 Hz), 79.1, 49.7 (dd, J = 25.8, 10.1 Hz), 40.4, 29.9, 28.8, 28.6, 28.4 (d, J = 9.1 Hz), 26.4. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -70.20 (d, J = 6.4 Hz, 3F). **HRMS (ESI)**: calculated for $C_{19}H_{26}O_4NF_3Na$ ($[M+Na]^+$): 412.1706; Found: 412.1710.

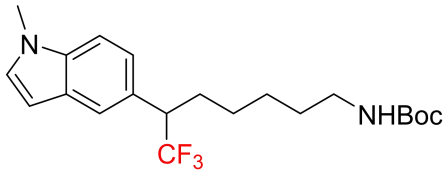
ethyl 5-(7-((*tert*-butoxycarbonyl)amino)-1,1,1-trifluoroheptan-2-yl)benzofuran-2-carboxylate (3g)



Obtained in 68% yield as a colorless oily liquid (62.4 mg, eluent: PE/EA = 10:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ

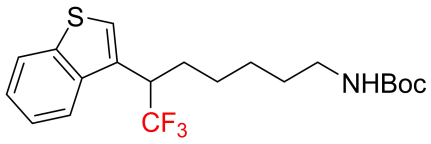
7.57 (d, J = 9.2 Hz, 2H), 7.51 (s, 1H), 7.35 (d, J = 8.8 Hz, 1H), 4.44 (q, J = 7.2 Hz, 3H), 3.36 – 3.26 (m, 1H), 3.04 – 3.00 (m, 2H), 2.07 – 1.99 (m, 1H), 1.94 – 1.84 (m, 1H), 1.41 (s, 9H), 1.39 – 1.35 (m, 2H), 1.33 – 1.28 (m, 2H), 1.24 (s, 3H), 1.20 – 1.13 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.6, 156.1, 155.4, 146.5, 130.6 (d, J = 2.3 Hz), 128.4 (d, J =7.7 Hz),, 127.4, 127.0 (q, J = 281.4 Hz), 123.4 (d, J = 6.8 Hz), 113.8 (d, J = 9.3 Hz), 112.7 – 112.6 (m), 79.2, 61.8, 50.0 (q, J = 26.7 Hz), 40.5, 29.9 (d, J = 8.2 Hz), 28.9, 28.5 (d, J = 5.1 Hz), 26.54, 26.50, 14.4 (d, J = 5.3 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.95 (d, J = 9.4 Hz, 3F). **RMS (ESI)**: calculated for $C_{23}H_{30}O_5NF_3Na$ ([M+Na]$^+$): 480.1968; Found: 480.1969.

***tert*-butyl (7,7,7-trifluoro-6-(1-methyl-1H-indol-5-yl)heptyl)carbamate (3h)**



Obtained in 83% yield as a brown oily liquid (65.9 mg, eluent: PE/EA = 5:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54 (s, 1H), 7.31 (d, J = 8.8 Hz, 1H), 7.14 (d, J = 8.4 Hz, 1H), 7.07 (d, J = 2.8 Hz, 1H), 6.49 – 6.48 (m, 1H), 4.50 (s, 1H), 3.78 (s, 3H), 3.37– 3.25 (m, 1H), 3.03 (s, 2H), 2.08 – 1.90 (m, 2H), 1.44 (s, 9H), 1.40 – 1.35 (m, 2H), 1.33 – 1.26 (m, 2H), 1.24 – 1.16 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.1, 136.5, 129.6, 128.7, 127.5 (q, J = 281.3 Hz), 125.6, 122.3, 121.6, 109.4, 101.1, 79.2, 50.2 (q, J = 26.3 Hz), 40.6, 33.0, 29.9, 29.0, 28.5, 26.7, 26.6. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.91 (d, J = 9.4 Hz, 3F). **HRMS (ESI)**: calculated for $C_{21}H_{30}O_2N_2F_3$ ([M+H]$^+$): 399.2254; Found: 399.2257.

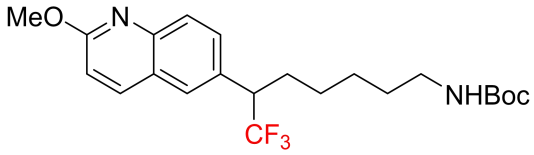
***tert*-butyl (6-(benzo[b]thiophen-3-yl)-7,7,7-trifluoroheptyl)carbamate (3i)**



Obtained in 31% yield as a colorless oily liquid (24.9 mg, eluent: PE/EA = 30:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82 – 7.80 (m, 1H), 7.75 (dd, J = 6.8, 2.0 Hz, 1H), 7.38 – 7.31(m, 2H), 7.24 (s, 1H), 4.50 (s, 1H), 3.65 – 3.54 (m, 1H), 3.06 (q, J = 6.6 Hz, 2H), 2.08 – 2.01 (m, 1H), 1.93 – 1.83 (m, 1H), 1.43 (s, 9H), 1.41 – 1.36 (m, 2H), 1.34 –1.26 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.1, 139.7,

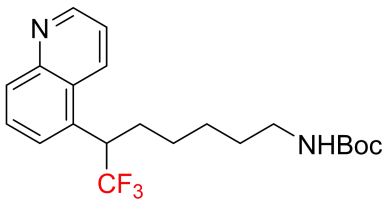
139.3, 137.8, 126.2 (q, J = 281.2 Hz), 124.7, 124.6, 123.6, 122.4, 79.2, 46.4 (q, J = 28.3 Hz), 40.5, 29.9, 29.7 (d, J = 2.0 Hz), 28.5, 26.5, 26.4. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -71.18 – -71.22 (m, 3F). **HRMS (ESI)**: calculated for $C_{20}H_{26}O_2NF_3NaS$ ($[M+Na]^+$): 424.1529; Found: 424.1535.

***tert*-butyl (7,7,7-trifluoro-6-(2-methoxyquinolin-6-yl)heptyl)carbamate (3j)**



Obtained in 55% yield as a colorless oily liquid (46.8 mg, eluent: PE/EA = 15:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.96 (d, J = 8.8 Hz, 1H), 7.85 (d, J = 8.4 Hz, 1H), 7.60 (s, 1H), 7.53 (d, J = 8.4 Hz, 1H), 6.92 (d, J = 9.2 Hz, 1H), 4.46 (s, 1H), 4.07 (s, 3H), 3.40 – 3.29 (m, 1H), 3.03 (q, J = 6.1 Hz, 2H), 2.09 – 1.90 (m, 2H), 1.42 (s, 9H), 1.38 – 1.35 (m, 2H), 1.34 – 1.28 (m, 2H), 1.23 – 1.16 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.9, 156.1, 146.3, 138.8, 130.5 (d, J = 2.0 Hz), 130.0, 128.2 (d, J = 4.9 Hz), 127.8, 127.1 (q, J = 281.4 Hz), 125.0, 113.7 (d, J = 6.0 Hz), 79.2, 53.7 (d, J = 10.3 Hz), 49.9 (q, J = 26.7 Hz), 40.5, 29.9, 28.7, 28.5 (d, J = 4.0 Hz), 26.6, 26.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.76 (d, J = 9.4 Hz, 3F).**HRMS (ESI)**: calculated for $C_{22}H_{30}O_3N_2F_3$ ($[M+H]^+$): 427.2203; Found: 427.2207.

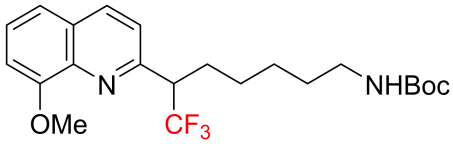
***tert*-butyl (7,7,7-trifluoro-6-(quinolin-5-yl)heptyl)carbamate (3k)**



Obtained in 68% yield as a yellow solid (54.0 mg, eluent: PE/EA = 5:1), mp: 77 – 79 °C. **^{1}H NMR** (101 MHz, $CDCl_3$) δ 8.95 (s, 1H), 8.39 (d, J = 8.8 Hz, 1H), 8.14 (d, J = 8.4 Hz, 1H), 7.76 (t, J = 8.0 Hz, 1H), 7.65 (d, J = 7.2 Hz, 1H), 7.49 (dd, J = 8.8, 4.0 Hz, 1H), 4.48 (s, 1H), 4.20 – 4.08 (m, 1H), 3.00 (q, J = 6.8 Hz, 2H), 2.23 – 2.15 (m, 1H), 2.11 – 2.01 (m, 1H), 1.40 (s, 9H), 1.36 – 1.24 (m, 4H), 1.21 – 1.12 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.1, 150.2, 148.5, 131.4, 131,2, 130.1, 129.2, 128.1, 127.0 (d, J = 281.3 Hz), 126.0, 121.6, 79.2, 42.7 (d, J = 27.6 Hz), 40.4, 29.8, 29.3, 28.5, 26.6, 26.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.23

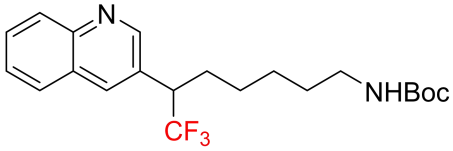
(s, 3F). **HRMS (ESI)**: calculated for $C_{21}H_{28}O_2N_2F_3$ ($[M+H]^+$): 397.2097; Found: 397.2099.

***tert*-butyl (7,7,7-trifluoro-6-(8-methoxyquinolin-2-yl)heptyl)carbamate (3l)**



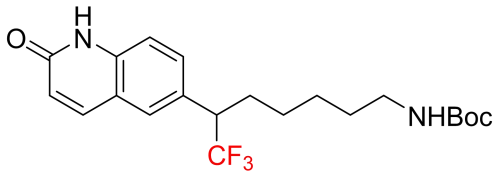
Obtained in 98% yield as a colorless oily liquid (83.6 mg, eluent: PE/EA = 6:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8,14 (d, J = 8.8 Hz, 1H), 7.49 – 7.44 (m, 2H), 7.38 (d, J = 8.0 Hz, 1H), 7.07 (d, J = 8.0 Hz, 1H), 4.53 (s, 1H), 4.07 (s, 3H), 3.99 – 3.88 (m, 1H), 3.00 (s, *2*H), 2.12 – 1.95 (m, 2H), 1.40 (s, 9H), 1.37 – 1.09 (m, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.1, 155.5, 154.6 (d, J = 2.1 Hz), 139.7, 137.2, 128.8, 127.1, 127.0 (q, J = 281.5 Hz), 120.1, 119.5, 108.6, 79.1, 56.4, 53.2 (q, J = 26.2 Hz), 40.5, 29.8, 28.8, 28.5, 26.6, 26.4. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -68.04 (d, J = 7.5 Hz, 3F). **HRMS (ESI)**: calculated for $C_{22}H_{30}O_3N_2F_3$ ($[M+H]^+$): 427.2203; Found: 427.2205.

***tert*-butyl (7,7,7-trifluoro-6-(quinolin-3-yl)heptyl)carbamate (3m)**



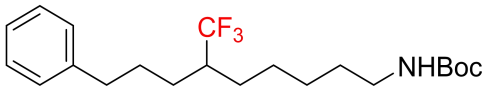
Obtained in 98% yield as a white solid (77.6 mg, eluent: PE/EA = 5:1), mp: 67 – 69°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.80 (d, J = 2.4 Hz, 1H), 8.13 – 8.08 (m, 2H), 7.82 (d, J = 8.0 Hz, 1H), 7.72 (ddd, J = 8.4, 6.8, 1.2 Hz, 1H), 7.56 (t, J = 7.4 Hz, 1H), 4.59 (s, 1H), 3.48 – 3.38 (m, 1H), 3.02 (q, J = 6.1 Hz, 2H), 2.15 – 2.06 (m, 1H), 2.03 – 1.93 (m, 1H), 1.39 (s, 9H), 1.37 – 1.14 (m, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.1, 151.0, 147.9, 136.1, 130.1, 129.2, 127.9, 127.86, 127.79, 127.3, 126.7 (q, J = 281.0 Hz), 79.1, 48.0 (q, J = 27.3 Hz), 40.4, 29.8, 28.5, 28.4, 26.5, 26.4. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.23 (s, 3F). **HRMS (ESI)**: calculated for $C_{21}H_{28}O_2N_2F_3$ ($[M+H]^+$): 397.2097; Found: 397.2098.

***tert*-butyl (7,7,7-trifluoro-6-(2-oxo-1,2-dihydroquinolin-6-yl)heptyl)carbamate (3n)**



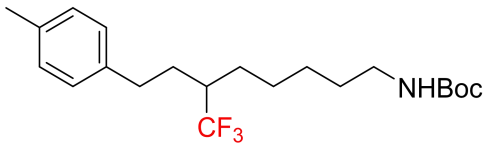
Obtained in 51% yield as a colorless oily liquid (42.2 mg, eluent: PE/EA = 2:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 12.74 (s, 1H), 7.81 (d, J = 9.2 Hz, 1H), 7.47 – 7.40 (m, 3H), 6.74 (d, J = 9.6 Hz, 1H), 4.52 (s, 1H), 3.32 – 3.21 (m, 1H), 3.03 (t, J = 7.0 Hz, 2H), 2.06 – 1.98 (m, 1H), 1.92 – 1.82 (m, 1H), 1.41 (s, 9H), 1.39 – 1.35 (m, 2H), 1.32 – 1.25 (m, 2H), 1.23 – 1.14 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 164.8, 156.1, 141.0, 138.4, 131.2, 129.3, 128.3, 126.9 (q, J = 280.3 Hz), 122.0, 120.1, 116.8, 79.2, 49.6 (q, J = 26.8 Hz), 40.5, 29.9, 28.7, 28.5, 26.52, 26.48. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.95 (s, 3F). **HRMS (ESI)**: calculated for $C_{21}H_{27}O_3N_2F_3$ Na ($[M+Na]^+$): 435.1866; Found: 435.1870.

***tert*-butyl (9-phenyl-6-(trifluoromethyl)nonyl)carbamate (3o)**



Obtained in 57% yield as a colorless oily liquid (44.0 mg, eluent: PE/EA = 50:1).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.29 (t, J = 7.4 Hz, 2H), 7.19 (t, J = 8.4 Hz, 3H), 4.56 (s, 1H) , 3.10 (q, J = 6.5 Hz, 2H), 2.62 (t, J = 7.4 Hz, 2H), 2.09 – 1.97 (m, 1H), 1.74 – 1.66 (m, 2H), 1.65 – 1.54 (m, 2H), 1.51 – 1.41 (m, 13H), 1.39 – 1.27 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.1, 141.9, 128.7 (q, J = 281.4 Hz), 128.5, 128.4, 126.0, 79.2, 42.5 (q, J = 24.9 Hz), 40.6, 36.0, 30.0, 28.7, 28.5, 27.8 (d, J = 2.5 Hz), 27.5 (d, J = 2.5 Hz), 27.0, 26.6. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.99 (d, J = 9.8 Hz, 3F). **HRMS (ESI)**: calculated for $C_{21}H_{32}O_2NF_3Na$ ($[M+Na]^+$): 410.2277; Found: 410.2283.

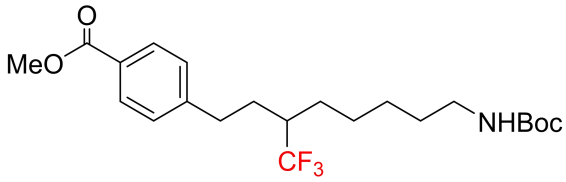
***tert*-butyl (8-(p-tolyl)-6-(trifluoromethyl)octyl)carbamate (3p)**



Obtained in 46% yield as a colorless oily liquid (35.6 mg, eluent: PE/EA = 80:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.09 (t, J = 8.4 Hz, 4H), 4.52(s, 1H), 3.10 (q, J = 6.4 Hz, 2H), 2.65 (t, J = 8.0 Hz, 2H), 2.32 (s, 3H), 2.09 – 1.99 (m, 1H), 1.96 – 1.87 (m, 1H), 1.75 – 1.68 (m, 1H), 1.66 – 1.57 (m, 1H), 1.50 –

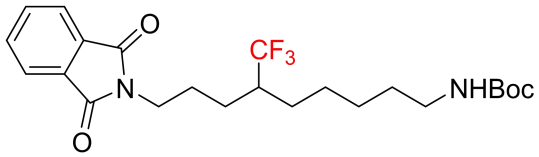
1.41 (m, 12H), 1.40 – 1.34 (m, 2H), 1.31 – 1.28 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.1, 138.3, 135.7, 128.84 (d, J = 95.3 Hz), 128.76 (d, J = 281.2 Hz), 72.9, 41.9 (d, J = 24.9 Hz), 40.6, 32.7, 30.0, 29.8 (d, J = 1.8 Hz), 28.5, 27.9 (d, J = 2.2 Hz), 27.0, 26.5, 21.1. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.86 (d, J = 7.5 Hz, 3F). **HRMS (ESI)**: calculated for $C_{21}H_{32}O_2NF_3Na$ ([M+Na]$^+$): 410.2277; Found: 410.2283.

methyl 4-(8-((*tert*-butoxycarbonyl)amino)-3-(trifluoromethyl)octyl)benzoate (3q)



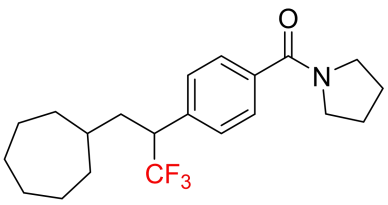
Obtained in 62% yield as a colorless oily liquid (53.5 mg, eluent: PE/EA = 20:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93 (d, J = 8.0 Hz, 2H), 7.21 (d, J = 8.0 Hz, 2H), 4.66 (s, 1H), 3,86 (s, 3H), 3.08 (q, J = 6.9 Hz, 2H), 2.71 (t, J = 8.0 Hz, 2H), 2.06 – 1.96 (m, 1H), 1.94 – 1.85 (m, 1H), 1.75 – 1.66 (m, 1H), 1.63 – 1.55 (m, 1H), 1.47 – 1.40 (m, 12H), 1.36 – 1.30 (m, 2H), 1.28 – 1.25 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.0, 156.1, 146.7, 129.9, 128.5 (q, J = 281.9 Hz), 128.4,128.2, 79.0, 52.0, 41.9 (q, J = 25.1 Hz), 40.4, 33.0, 29.9, 29.3, 28.4, 27.8 (d, J = 2.5 Hz), 26.8, 26.4. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.85 (s, 3F). **HRMS (ESI)**: calculated for $C_{22}H_{32}O_4NF_3Na$ ([M+Na]$^+$): 454.2176; Found: 454.2181.

***tert*-butyl (9-(1,3-dioxoisoindolin-2-yl)-6-(trifluoromethyl)nonyl)carbamate (3r)**



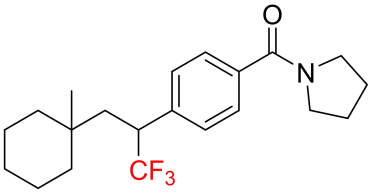
Obtained in 40% yield as a colorless oily liquid (36.5 mg, eluent: PE/EA = 8:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.86 – 7.84 (m, 2H), 7.73 – 7.71 (m, 2H), 4.55 (s, 1H), 3.68 (t, J = 7.2 Hz, 2H), 3.09 (q, J = 6.7 Hz, 2H), 2.07 – 2.03 (m, 1H), 1.80 – 1.73 (m, 2H), 1.68 – 1.56 (m, 3H), 1.52 – 1.45 (m, 3H), 1.43 (s, 9H), 1.37 – 1.28 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 168.5, 156.1, 134.1, 132.1, 128.5 (q, J = 281.4 Hz), 123.4, 79.1, 42.2 (q, J = 25.0 Hz), 40.5, 37.8, 29.9, 28.5, 27.8, 26.9, 26.6, 25.9, 25.2. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.97 (d, J = 9.4 Hz, 3F). **HRMS (ESI)**: calculated for $C_{23}H_{31}O_4N_2F_3Na$([M+Na]$^+$): 479.2128; Found: 479.2130.

(4-(3-cycloheptyl-1,1,1-trifluoropropan-2-yl)phenyl)(pyrrolidin-1-yl)methanone (4a)



Obtained in 89% yield as a white solid (65.4 mg, eluent: PE/EA = 8:1), mp: 48 – 50 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.49 (d, J = 8.0 Hz, 2H), 7.28 (d, J = 7.6 Hz, 2H), 3.62 (s, 2H), 3.42 (s, 2H), 3.37 – 3.27 (m, 1H), 1.93 (d, J = 4.8 Hz, 2H), 1.84 (d, J = 4.4 Hz, 2H), 1.80 (t, J = 6.8 Hz, 2H), 1.66 – 1.60 (m, 1H), 1.59 – 1.50 (m, 3H), 1.48 – 1.37 (m, 4H), 1.32 – 1.07 (m, 5H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.3, 136.9, 136.8, 129.2 (d, J = 1.8 Hz), 129.0, 127.6, 127.4 (d, J = 3.6 Hz), 127.1 (q, J = 281.2 Hz), 49.7, 48.1 – 47.3 (m), 46.4 – 46.2 (m), 36.3 (q, J = 12.8 Hz), 35.7, 35.2, 32.4 (d, J = 10.6 Hz), 28.6, 28.4, 26.5, 26.1, 25.8, 24.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.64 (d, J = 9.4 Hz, 3F). **HRMS (ESI)**: calculated for $C_{21}H_{29}ONF_3$ ($[M+H]^+$): 368.2196; Found: 368.2195.

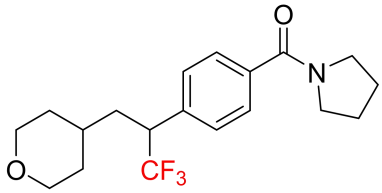
pyrrolidin-1-yl(4-(1,1,1-trifluoro-3-(1-methylcyclohexyl)propan-2-yl)phenyl)methanone (4b)



Obtained in 85% yield as a colorless oily liquid (62.4 mg, eluent: PE/EA = 8:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.47 (d, J = 8.0 Hz, 2H), 7.34 (d, J = 8.0 Hz, 2H), 3.61 (t, J = 7.0 Hz, 2H), 3.39 (t, J = 6.6 Hz, 2H), 3.36 – 3.29 (m, 1H), 1.96 – 1.90 (m, 4H), 1.87 – 1.80 (m, 2H), 1.45 – 1.32 (m, 3H), 1.24 – 1.21 (m, 5H), 1.05 – 0.94 (m, 2H), 0.70 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.3, 138.9, 136.8, 129.4, 127.4, 127.2 (q, J = 281.0 Hz), 49.7, 46.3, 45.9 (q, J = 26.4 Hz), 40.7, 38.0 (d, J = 10.0 Hz), 33.2, 26.4, 26.2, 25.0, 24.5, 21.8 (d, J = 9.0 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -70.07 (d, J = 9.8 Hz, 3F). **HRMS (ESI)**: calculated for $C_{21}H_{29}ONF_3$ ($[M+H]^+$): 368.2196; Found: 368.2194.

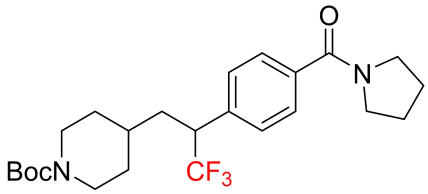
pyrrolidin-1-yl(4-(1,1,1-trifluoro-3-(tetrahydro-2H-pyran-4-yl)propan-2-

yl)phenyl)methanone (4c)



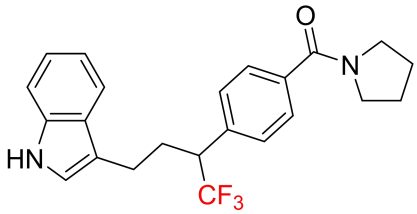
Obtained in 73% yield as a light blue oily liquid (51.9 mg, eluent: PE/EA = 1:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.50 (d, J = 8.4 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 3.85 (t, J = 10.8 Hz, 2H), 3.62 (t, J = 7.0 Hz, 2H), 3.41 (t, J = 6.6 Hz, 2H), 3.39 – 3.31 (m, 1H), 3.24 – 3.13 (m, 2H), 1.97 – 1.76 (m, 6H), 1.54 (d, J = 9.2 Hz, 1H), 1.41 (d, J = 12.0 Hz, 1H), 1.34 – 1.17 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.2, 137.3, 136.3, 129.0, 127.7, 126.9 (q, J = 281.1 Hz), 67.6 (d, J = 17.4 Hz), 49.7, 46.7 (q, J = 26.8 Hz), 46.3, 35.5, 33.6, 31.6 (d, J = 31.5 Hz), 26.5, 24.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.77 (d, J = 9.0 Hz, 3F). **HRMS (ESI)**: calculated for $C_{19}H_{25}O_2NF_3$ ($[M+H]^+$): 356.1832; Found: 356.1827.

***tert*-butyl 4-(3,3,3-trifluoro-2-(4-(pyrrolidine-1-carbonyl)phenyl)propyl)piperidine-1-carboxylate (4d)**



Obtained in 93% yield as a colorless oily liquid (84.6 mg, eluent: PE/EA = 2:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.50 (d, J = 7.6 Hz, 2H), 7.29 (d, J = 7.6 Hz, 2H), 3.99 (t, J = 13.2 Hz, 2H), 3.62 (t, J = 6.8 Hz, 2H), 3.41 (t, J = 6.8 Hz, 2H), 3.38 – 3.30 (m, 1H), 2.55 – 2.42 (m, 2H), 1.96 – 1.84 (m, 5H), 1.80 – 1.73 (m, 1H), 1.61 (d, J = 12.4 Hz, 1H), 1.46 (d, J = 12.0 Hz, 1H), 1.40 (s, 9H), 1.17 – 0.98 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.2, 154.8, 137.3, 136.3, 129.0, 127.7, 126.8 (q, J = 281.0 Hz), 79.4, 49.7, 47.0 (q, J = 26.9 Hz), 46.3, 35.2, 32.9, 32.5, 30.8, 28.5, 26.5, 24.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.81 (d, J = 9.0 Hz, 3F). **HRMS (ESI)**: calculated for $C_{24}H_{34}O_3N_2F_3$ ($[M+H]^+$): 455.2516; Found: 455.2519.

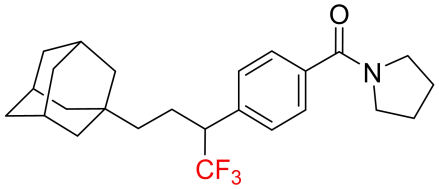
pyrrolidin-1-yl(4-(1,1,1-trifluoro-4-(1H-indol-3-yl)butan-2-yl)phenyl)methanone (4e)



Obtained in 86% yield as a Light brown solid (68.9 mg, eluent: PE/EA = 1:1), mp: 163 – 165°C. **^{1}H**

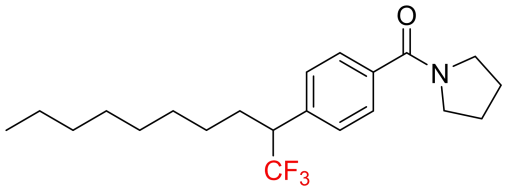
NMR (400 MHz, $CDCl_3$) δ 8.73 (s, 1H), 7.57 (d, *J* = 8.0 Hz, 2H), 7.47 (d, *J* = 8.0 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.18 (t, *J* = 7.6 Hz, 1H), 7.09 (t, *J* = 7.6 Hz, 1H), 6.80 (s, 1H), 3.70 (t, *J* = 7.0 Hz, 2H), 3.46 (t, *J* = 6.6 Hz, 2H), 3.39 – 3.28 (m, 1H), 2.76 – 2.69 (m, 1H), 2.59 – 2.43 (m, 2H), 2.34 – 2.23 (m, 1H), 2.01 – 1.85 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) *δ* 169.4, 136.9, 136.6, 136.5, 129.3, 127.5, 127.1, 126.9 (q, *J* = 281.0 Hz), 121.9, 121.8, 119.1, 118.6, 114.0, 111.4, 49.7, 49.1 (q, *J* = 26.7 Hz), 46.4, 28.8, 26.4, 24.4, 22.0. **^{19}F NMR** (376 MHz, $CDCl_3$) *δ* -69.29 (d, *J* = 9.0 Hz, 3F). **HRMS (ESI)**: calculated for $C_{23}H_{24}ON_2F_3$ ($[M+H]^+$): 401.1835; Found: 401.1839.

(4-(4-((3r,5r,7r)-adamantan-1-yl)-1,1,1-trifluorobutan-2-yl)phenyl)(pyrrolidin-1-yl)methanone (4f)



Obtained in 72% yield as a colorless oily liquid (60.7 mg, eluent: PE/EA = 15:1). **^{1}H NMR** (400 MHz, $CDCl_3$) *δ* 7.50 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 3.54 (d, *J* = 71.6 Hz, 4H), 3.18 – 3.07 (m, 1H), 2.03 – 1.74 (m, 9H), 1.618 (d, *J* = 49.2 Hz, 2H), 1.617 (d, *J* = 25.6 Hz, 4H), 1.38 (t, *J* = 14.2 Hz, 6H), 0.93 (td, *J* = 11.6, 4.8 Hz, 1H), 0.80 (td, *J* = 12.8, 4.4 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) *δ* 169.4, 137.0, 136.8, 129.1 (d, *J* = 24.4 Hz), 127.6 (d, *J* = 15.5 Hz), 126.9 (q, *J* = 281.1 Hz), 51.0 – 50.6 (m), 46.4, 42.2 (t, *J* = 14.3 Hz), 41.4, 37.2 (t, *J* = 16.3 Hz), 32.1, 28.7 (d, *J* = 12.2 Hz), 25.5 (d, *J* = 191.5 Hz), 21.7 (t, *J* = 11.1 Hz). **^{19}F NMR** (376 MHz, $CDCl_3$) *δ* -69.69 (d, *J* = 9.0 Hz, 3F). **HRMS (ESI)**: calculated for $C_{25}H_{33}ONF_3$ ($[M+H]^+$): 420.2509; Found: 420.2516.

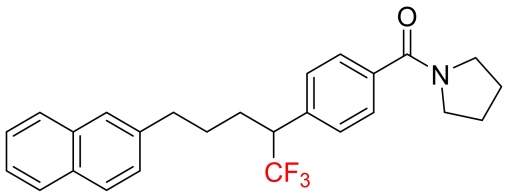
pyrrolidin-1-yl(4-(1,1,1-trifluorodecan-2-yl)phenyl)methanone (4g)



Obtained in 73% yield as a colorless oily liquid (53.6 mg, eluent: PE/EA = 7:1). **^{1}H NMR** (400 MHz, $CDCl_3$) *δ* 7.50 (d, *J* = 7.6 Hz, 2H), 7.29 (d, *J* = 7.6 Hz, 2H), 3.63 (t, *J* = 7.0 Hz, 2H), 3.42 (t, *J* = 7.0 Hz, 2H), 3.27 – 3.16 (m, 1H), 2.01 – 1.91 (m, 3H), 1.89 – 1.78 (m, 3H), 1.26 – 1.23 (m, 4H),

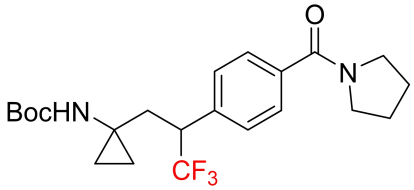
1.19 (s, 6H), 1.14 – 1.08 (m, 2H), 0.84 (t, J = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.4, 137.0, 136.9, 129.1, 127.5, 126.9 (q, J = 281.1 Hz), 50.0 (q, J = 26.6 Hz), 49.7, 46.3, 31.9, 29.3, 29.2 (d, J = 1.8 Hz), 28.6 (d, J = 1.1 Hz), 26.7, 26.5, 24.5, 22.7,14.2. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.68 (d, J = 9.4 Hz, 3F). **HRMS (ESI)**: calculated for $C_{21}H_{31}ONF_3$ ($[M+H]^+$): 370.2352; Found: 370.2349.

pyrrolidin-1-yl(4-(1,1,1-trifluoro-5-(naphthalen-2-yl)pentan-2-yl)phenyl)methanone (4h)



Obtained in 96% yield as a colorless oily liquid (81.1 mg, eluent: PE/EA = 6:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (m, 3H), 7.53 (d, J = 5.6 Hz, 2H), 7.50 (s, 1H), 7.47 – 7.40 (m, 2H), 7.29 (d, J = 8.0 Hz, 2H), 7.23 (d, J = 8.4 Hz, 1H), 3.65 (s, 2H), 3.41 (s, 2H), 3.34 – 3.23 (m, 1H), 2.75 (m, 2H), 2.13 – 2.04 (m, 1H), 1.98 – 1.85 (m, 5H), 1.66 – 1.52 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.2, 139.0, 137.1, 136.5, 133.6, 132.1, 129.0, 128.0, 127.65, 127.57, 127.4, 127.1, 126.8 (q, J = 281.2 Hz), 126.5, 126.0, 125.3, 49.9 (q, J = 26.7 Hz), 49.6, 46.3, 35.6, 28.3, 28.2, 26.4, 24.5.**^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.50 (d, J = 9.0 Hz, 3F). **HRMS (ESI)**: calculated for $C_{26}H_{27}ONF_3$ ($[M+H]^+$): 426.2039; Found: 426.2043.

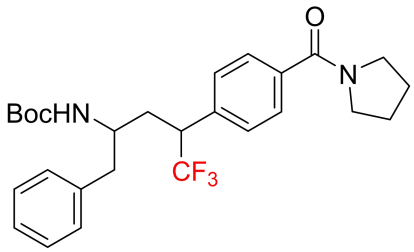
***tert*-butyl (1-(3,3,3-trifluoro-2-(4-(pyrrolidine-1-carbonyl)phenyl)propyl)cyclopropyl)carbamate (4i)**



Obtained in 87% yield as a white solid (74.2 mg, eluent: PE/EA = 1:1), mp: 106 – 108 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.46 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 7.6 Hz, 2H), 4.73 (s, 1H), 3.58 (t, J = 7.0 Hz, 2H), 3.55 – 3.49 (m, 1H), 3.37 (d, J = 6.6 Hz, 2H), 2.26 (d, J = 14.8 Hz, 1H), 2.09 – 2.03 (m, 1H), 1.94 – 1.87 (m, 2H), 1.84 – 1.78 (m, 2H), 1.33 (s, 9H), 0.68 – 0.27 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ

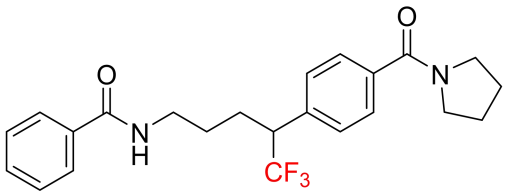
169.1, 155.3, 137.5, 137.0, 128.9, 127.6, 126.7 (q, J = 280.9 Hz), 79.4, 49.6, 46.2, 36.1, 31.5, 28.3, 26.4, 24.4, 15.0, 13.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.21 (d, J = 7.5 Hz, 3F). **HRMS (ESI)**: calculated for $C_{22}H_{30}O_3N_2F_3$ ($[M+H]^+$): 427.2203; Found: 427.2204.

***tert*-butyl((*2S*)-5,5,5-trifluoro-1-phenyl-4-(4-(pyrrolidine-1-carbonyl)phenyl)pentan-2-yl)carbamate (4j)**



Obtained in 82% yield as a white solid (80.4 mg, eluent: PE/EA = 5:1), mp: 129 – 131 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.49 (d, J = 7.6 Hz, 2H), 7.27 (q, J = 7.5 Hz, 4H), 7.22 – 7.16 (m, 1H), 7.07 (d, J = 7.2 Hz, 2H), 4.26 (d, J = 8.8 Hz, 1H), 3.86 – 3.81 (m, 1H), 3.63 (t, J =7.0 Hz, 2H), 3.42 (t, J = 5.8 Hz, 3H), 2.74 (s, 2H), 2.26 – 2.19 (m, 1H), 2.05 – 1.82 (m, 5H), 1.33 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.1, 155.1, 137.3, 137.2, 136.9, 129.4, 129.0, 128.6, 127.7, 126.7 (q, J = 281.0 Hz), 126.69, 79.4, 50.4, 49.7, 46.3, 40.7, 34.0, 28.4, 26.5, 24.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.40 (d, J = 8.6 Hz, 3F). **HRMS (ESI)**: calculated for $C_{27}H_{34}O_3N_2F_3$ ($[M+H]^+$): 491.2516; Found: 491.2522.

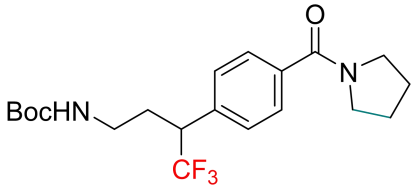
***N*-(5,5,5-trifluoro-4-(4-(pyrrolidine-1-carbonyl)phenyl)pentyl)benzamide (4k)**



Obtained in 53% yield as a colorless oily liquid (44.3 mg, eluent: PE/EA = 1:3). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 (d, J = 7.6 Hz, 2H), 7.46 – 7.34 (m, 5H), 7.24 (d, J = 7.6 Hz, 2H), 6.87 (s, 1H), 3.59 (t, J = 7.0 Hz, 2H), 3.39 (t, J =6.6 Hz, 2H), 3.35 – 3.21 (m, 3H), 2.08 – 1.79 (m, 6H), 1.50 – 1.20 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.2, 167.7, 137.1, 136.3, 134.5, 131.4, 129.0, 128.5, 127.5, 127.1, 126.7 (q, J = 280.9 Hz), 49.7, 49.6 (q, J = 26.9 Hz), 46.3,

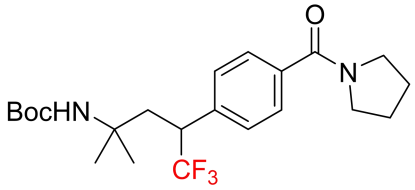
39.3, 26.9, 26.4, 26.1, 24.5. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.58 (d, J = 9.0 Hz, 3F). **HRMS (ESI)**: calculated for $C_{23}H_{26}O_2N_2F_3$ ($[M+H]^+$): 419.1941; Found: 419.1946.

***tert*-butyl (4,4,4-trifluoro-3-(4-(pyrrolidine-1-carbonyl)phenyl)butyl)carbamate (4l)**



Obtained in 81% yield as a white solid (64.8 mg, eluent: PE/EA = 1:1), mp: 121 – 123 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.47 (d, J = 8.0 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 4.78 (s, 1H), 3.60 (t, J = 7.2 Hz, 2H), 3.39 (t, J =6.6 Hz, 2H), 3.33 – 3.28 (m, 1H), 3.07 – 2.86 (m, 2H), 2.24 – 2.16 (m, 1H), 2.04 – 1.80 (m, 5H), 1.38 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.1, 155.9, 137.2, 135.9, 129.0, 127.6, 126.6 (q, J = 280.9 Hz), 79.3, 49.6, 47.5 (q, J = 27.8 Hz), 46.2, 37.9, 29.3, 28.3, 26.4, 24.4. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.52 (d, J = 9.4 Hz, 3F). **HRMS (ESI)**: calculated for $C_{20}H_{28}O_3N_2F_3$ ($[M+H]^+$): 401.2047; Found: 401.2049.

***tert*-butyl(5,5,5-trifluoro-2-methyl-4-(4-(pyrrolidine-1-carbonyl)phenyl)pentan-2-yl)carbamate (4m)**



Obtained in 79% yield as a white solid (67.7 mg, eluent: PE/EA = 3:1), mp: 73 – 75 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.46 (d, J = 8.0 Hz, 2H), 7.32 (d, J = 7.6 Hz, 2H), 4.30 (s, 1H), 3.59 (t, J = 7.0 Hz, 2H), 3.41 – 3.31 (m, 3H), 2.47 (d, J =14.4 Hz, 1H), 1.94 – 1.88 (m, 2H), 1.85 – 1.78 (m, 2H), 1.32 (s, 9H), 1.20 (s, 3H), 0.99 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.1, 154.0, 138.0, 136.9, 129.3, 127.6, 126.9 (q, J = 281.5 Hz), 79.0, 52.1, 49.6, 46.5 (q, J = 26.8 Hz), 46.2, 38.0, 28.3, 28.0 (d, J = 20.7 Hz), 26.4, 24.4. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -69.87 (s, 3F). **HRMS (ESI)**: calculated for $C_{22}H_{31}O_3N_2F_3Na$ ($[M+Na]^+$): 451.2179; Found: 451.2183.

8. Reference

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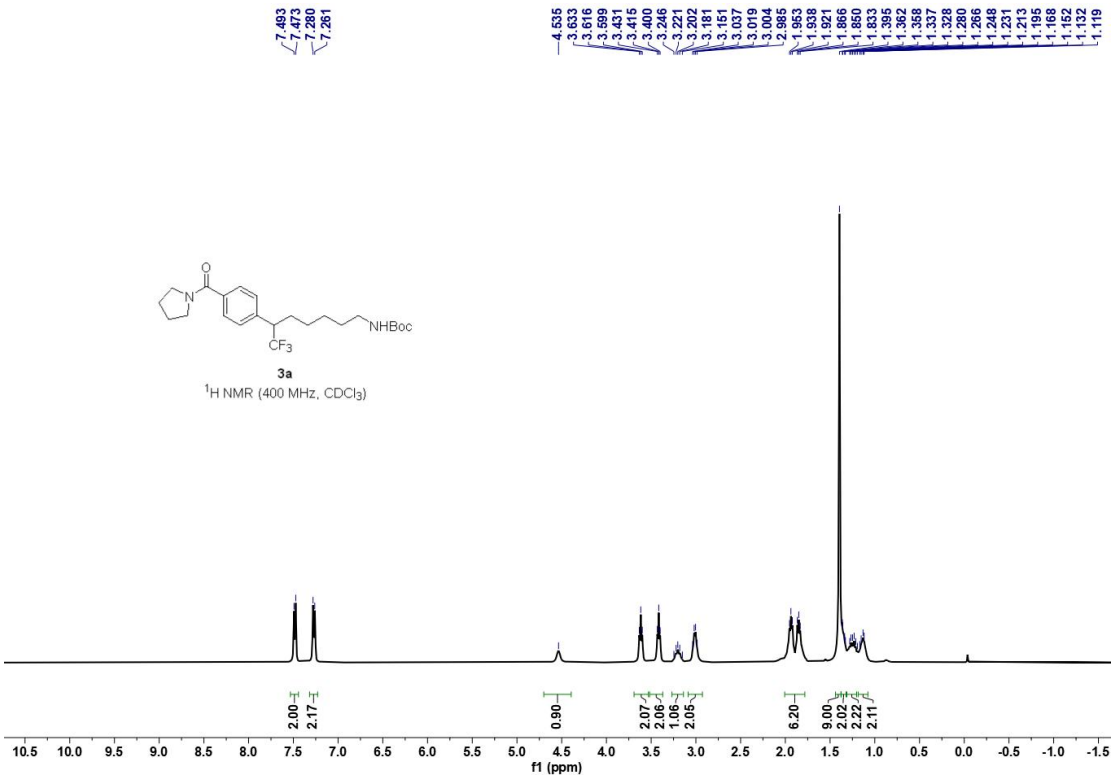
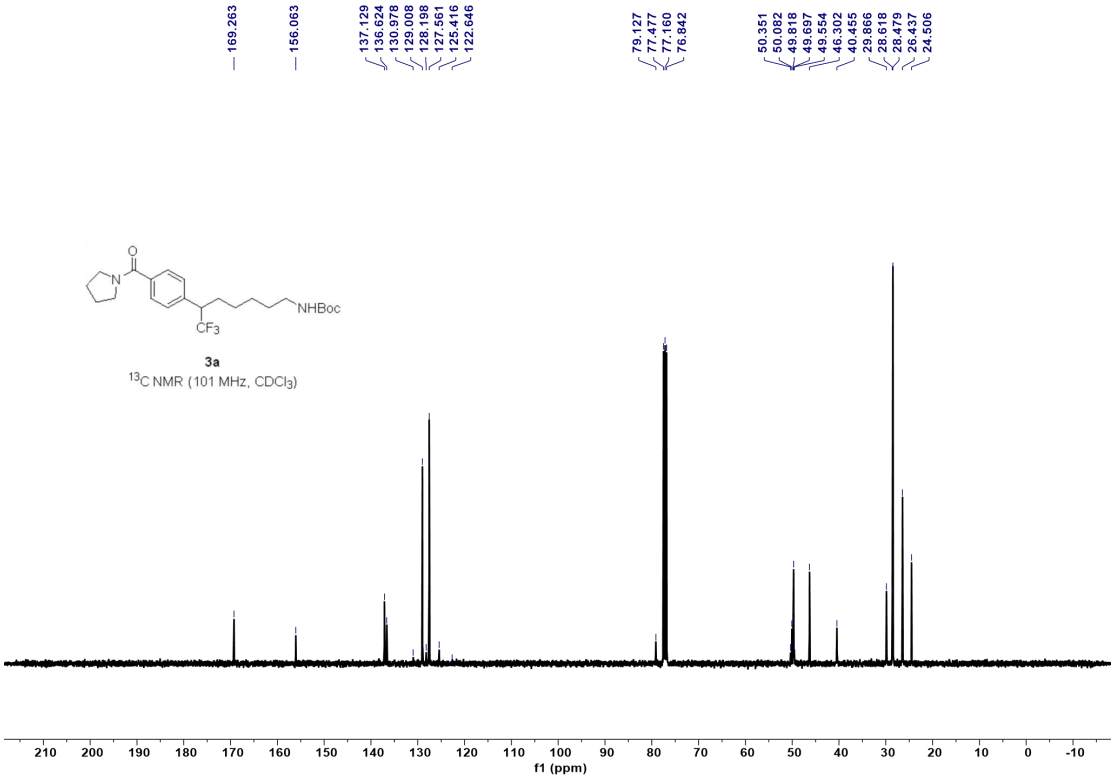
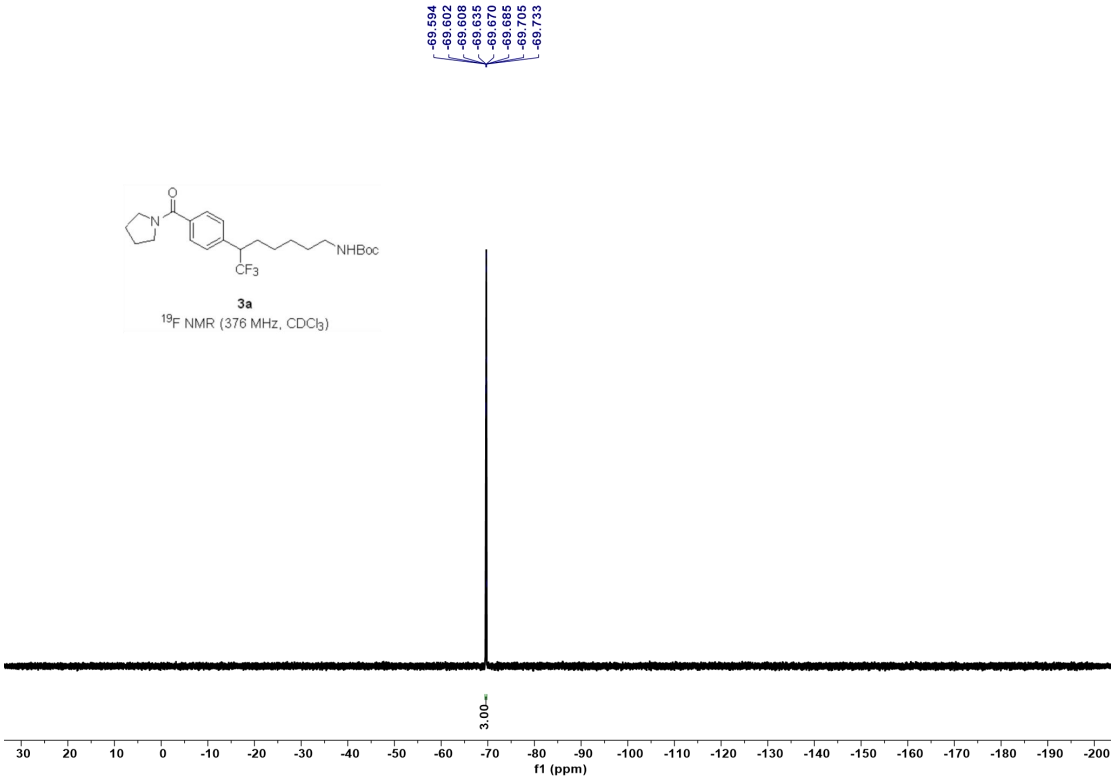
[3] Y.-G. Lou, A.-J. Wang, L. Zhao, L.-F. He, X.-F. Li, C.-Y. He, X. Zhang. Palladium-catalyzed cross-coupling of unactivated alkylzinc reagents with 2-bromo-3,3,3-trifluoropropene and its application in the synthesis of fluorinated amino acids. *Chem. Commun*, **2019**, *55*, 3705-3708.

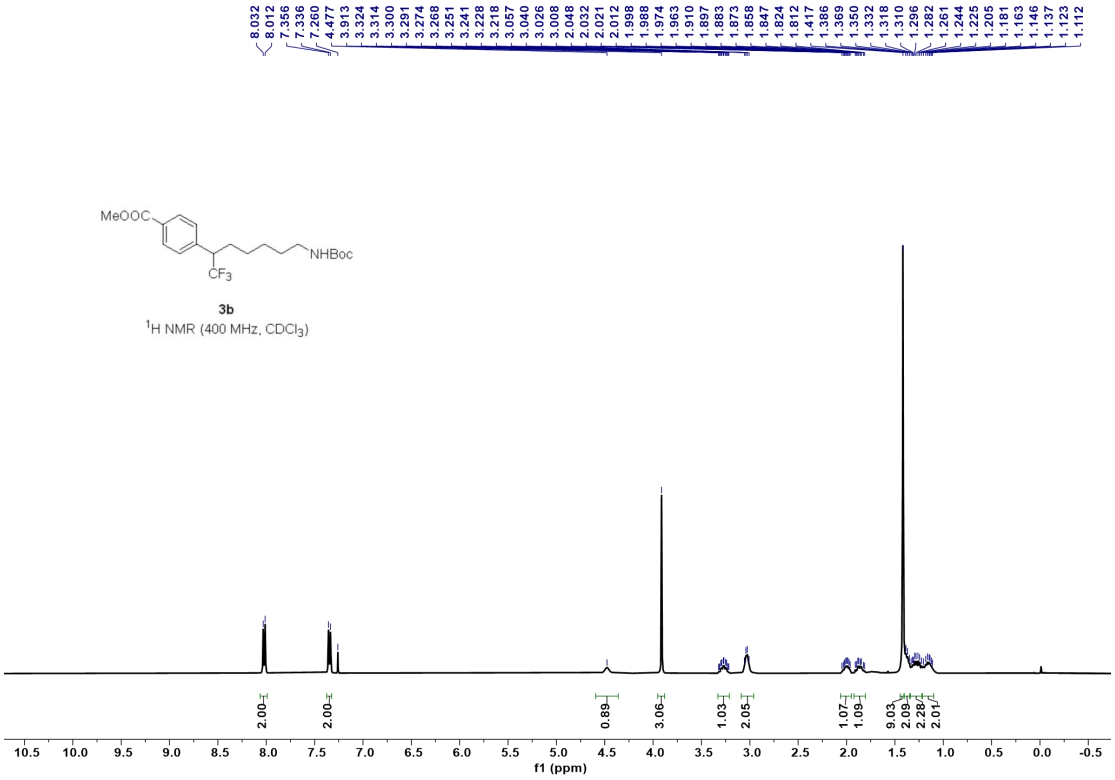
[4] a) K. Okada, K. Okamoto, M. Oda. A new and practical method of decarboxylation: photosensitized decarboxylation of N-acyloxyphthalimides via electron-transfer mechanism. *J. Am. Chem. Soc*. **1988**, *110*, 8736-8738. b) K. Okada, K. Okamoto, N. Morita, K. Okubo, M. Oda. Photosensitized decarboxylative Michael addition through N-(acyloxy)phthalimides via an electron-transfer mechanism. *J. Am. Chem. Soc*. **1991**, *113*, 9401-9402. c) T. Qin, J. Cornella, C. Li, L. R. Malins, J. T. Edwards, S. Kawamura, B. D. Maxwell, M. D. Eastgate, P. S. Baran. A general alkyl-alkyl cross-coupling enabled by redox-active esters and alkylzinc reagents. *Science*. **2016**, *352*, 801-805.

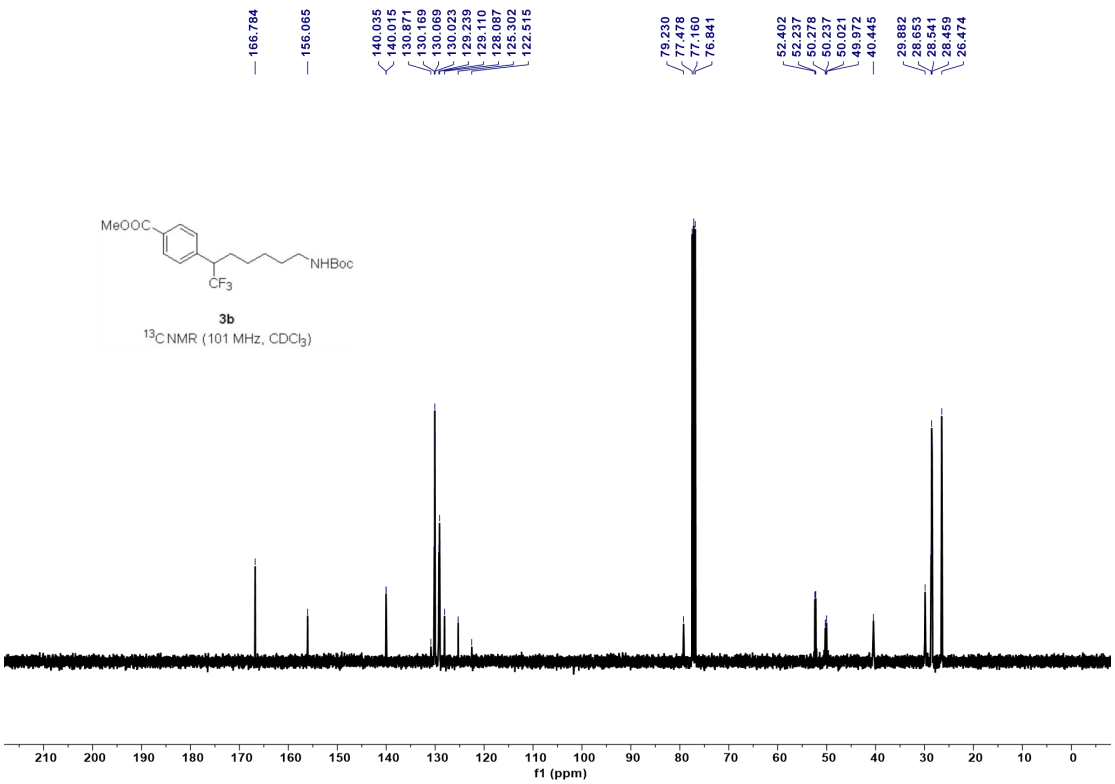
9. Copies of ^{1}H NMR, ^{19}F NMR and ^{13}C NMR charts of the Products

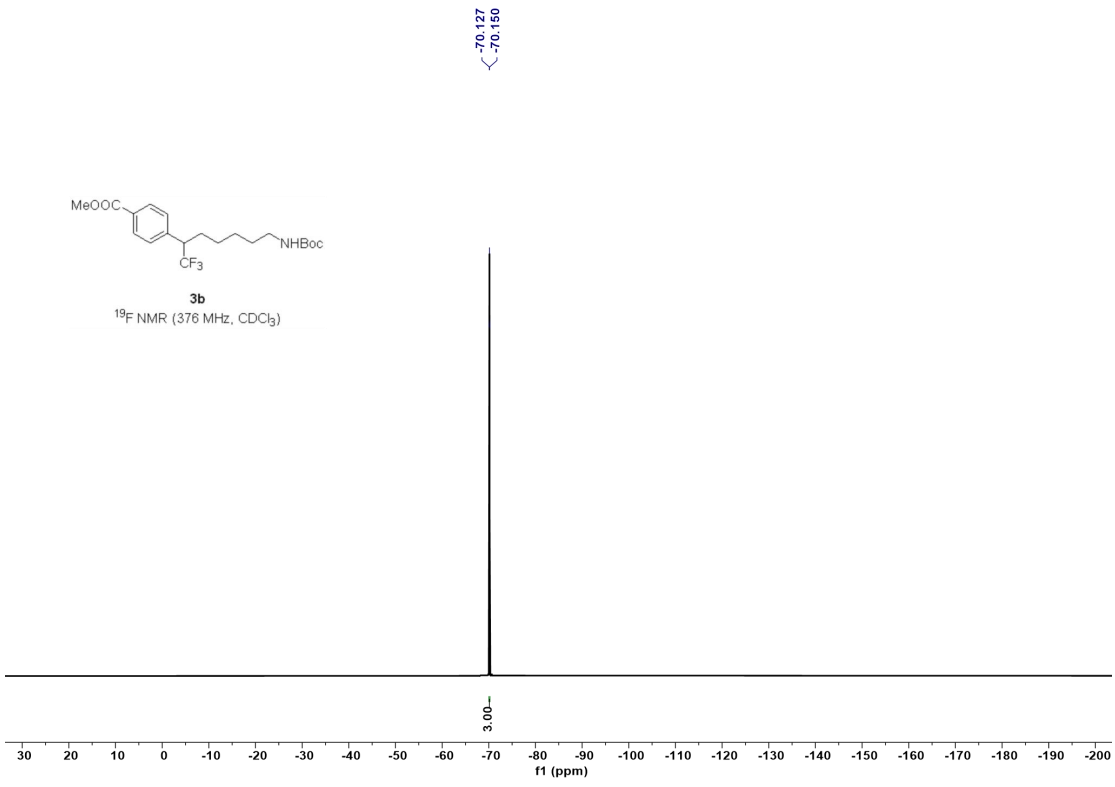

7.493
7.473
7.280
7.261
4.535
3.633
3.616
3.599
3.431
3.415
3.400
3.246
3.221
3.202
3.181
3.151
3.037
3.019
3.004
2.985
1.953
1.938
1.921
1.866
1.850
1.833
1.395
1.362
1.358
1.337
1.328
1.280
1.266
1.248
1.231
1.213
1.195
1.168
1.152
1.132
1.119
NHBoc
CF3
3a
1H NMR (400 MHz, CDCl3)
2.00
2.17
0.90
2.07
2.06
1.06
2.05
6.20
9.00
2.02
2.22
2.11
f1 (ppm)

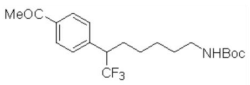

169.263
156.063
137.129
136.624
130.978
129.008
128.198
127.561
125.416
122.646
79.127
77.477
77.160
76.842
50.351
50.082
49.818
49.697
49.554
46.302
40.455
29.866
28.618
28.479
26.437
24.506
NHBoc
CF3
3a
13C NMR (101 MHz, CDCl3)
f1 (ppm)


-69.594
-69.602
-69.608
-69.635
-69.670
-69.685
-69.705
-69.733
NHBoc
CF3
3a
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)


8.032
8.012
7.356
7.336
7.260
4.477
3.913
MeOOC
NHBoc
CF3
3b
1H NMR (400 MHz, CDCl3)
2.00
2.00
0.89
3.06
1.03
2.05
1.07
1.09
9.03
2.09
2.28
2.01
f1 (ppm)

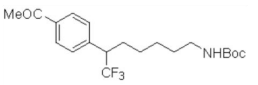

166.784
156.065
140.035
140.015
130.871
130.169
130.069
130.023
129.239
129.110
128.087
125.302
122.515
79.230
77.478
77.160
76.841
52.402
52.237
50.278
50.237
50.021
49.972
40.445
29.882
28.653
28.541
28.459
26.474
MeOOC
NHBoc
CF3
3b
13C NMR (101 MHz, CDCl3)
f1 (ppm)


-70.127
-70.150
MeOOC
NHBoc
CF3
3b
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)



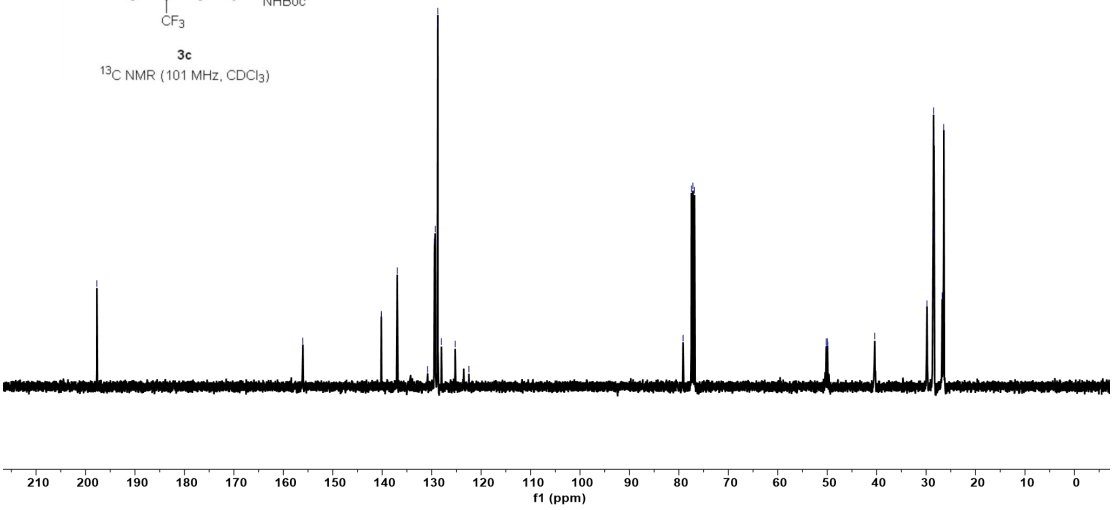
3c

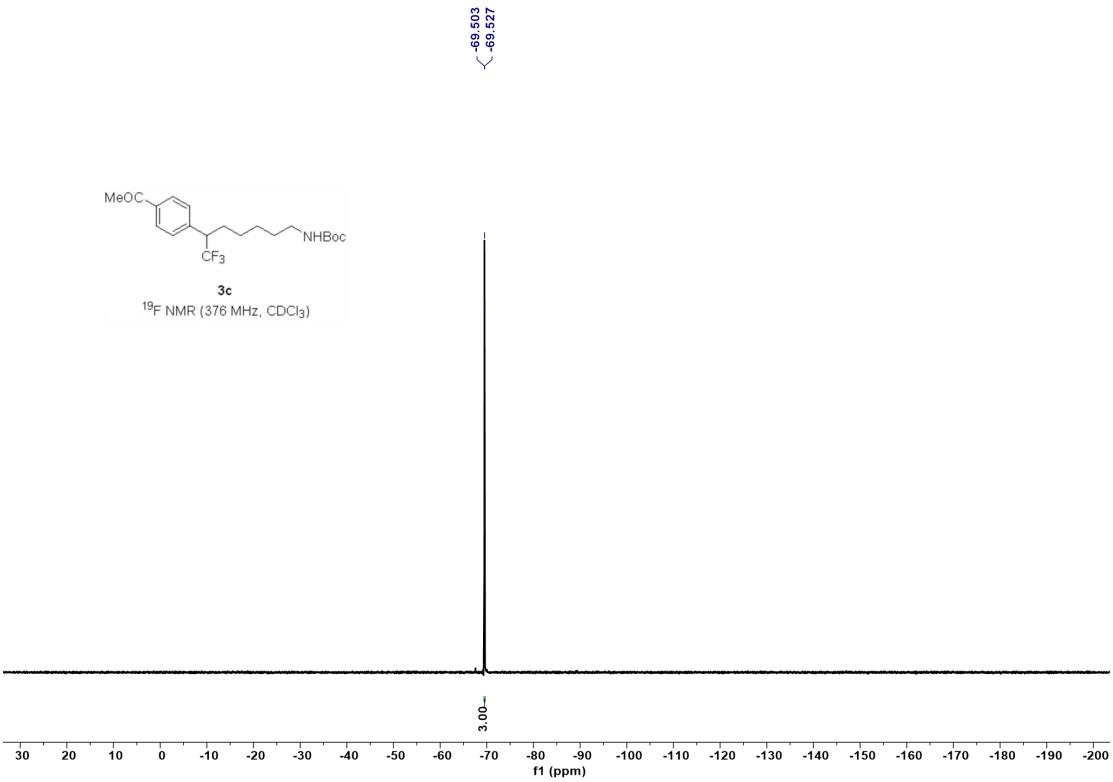
^{1}H NMR (400 MHz, $CDCl_3$)



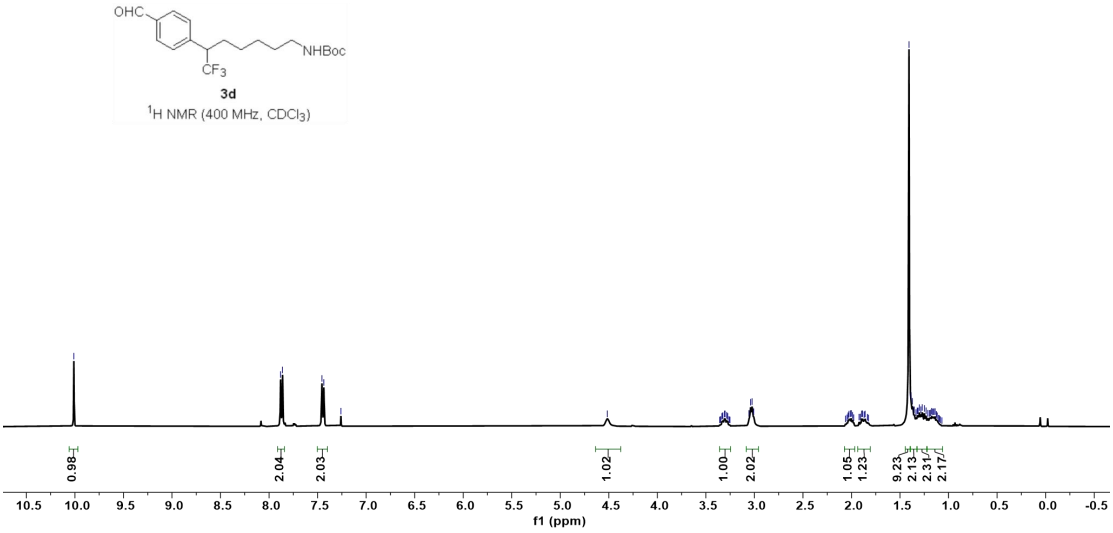
3c

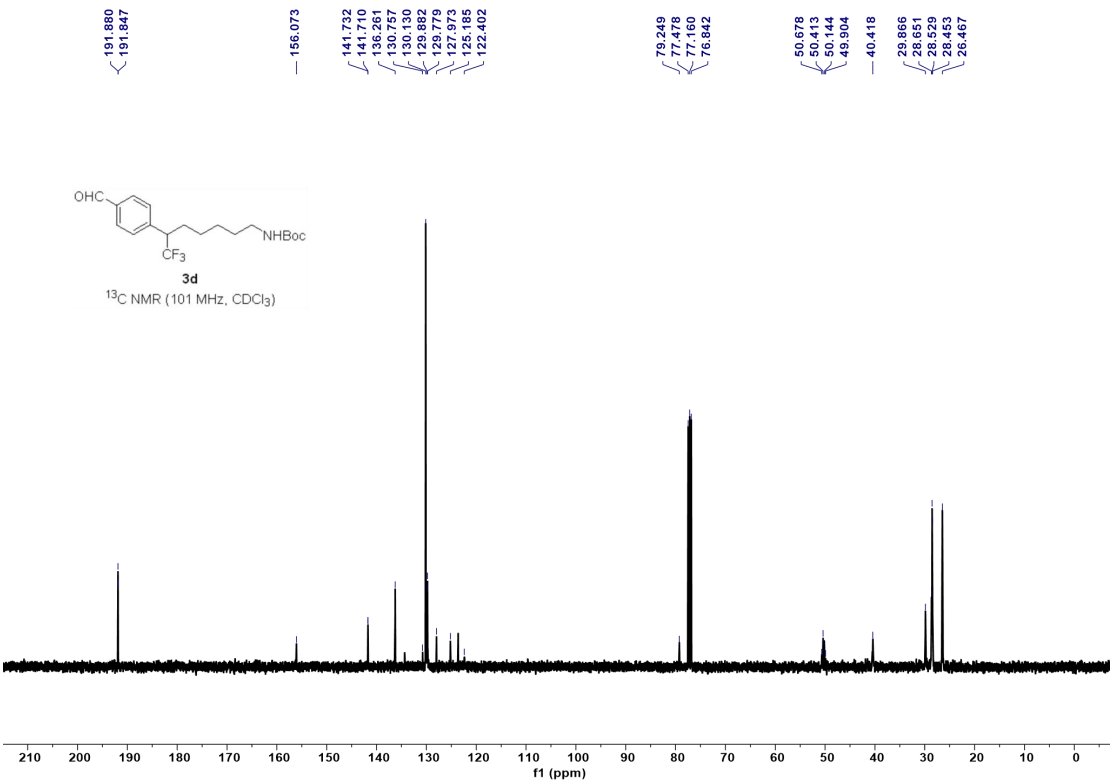
^{13}C NMR (101 MHz, $CDCl_3$)

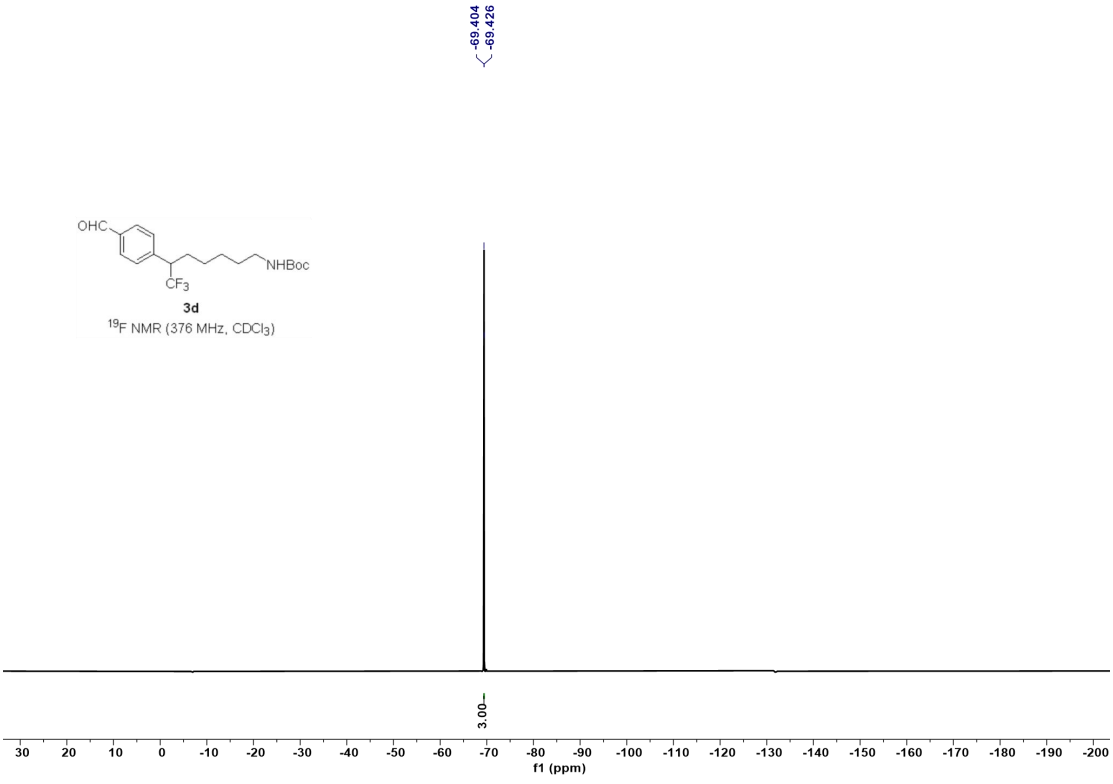


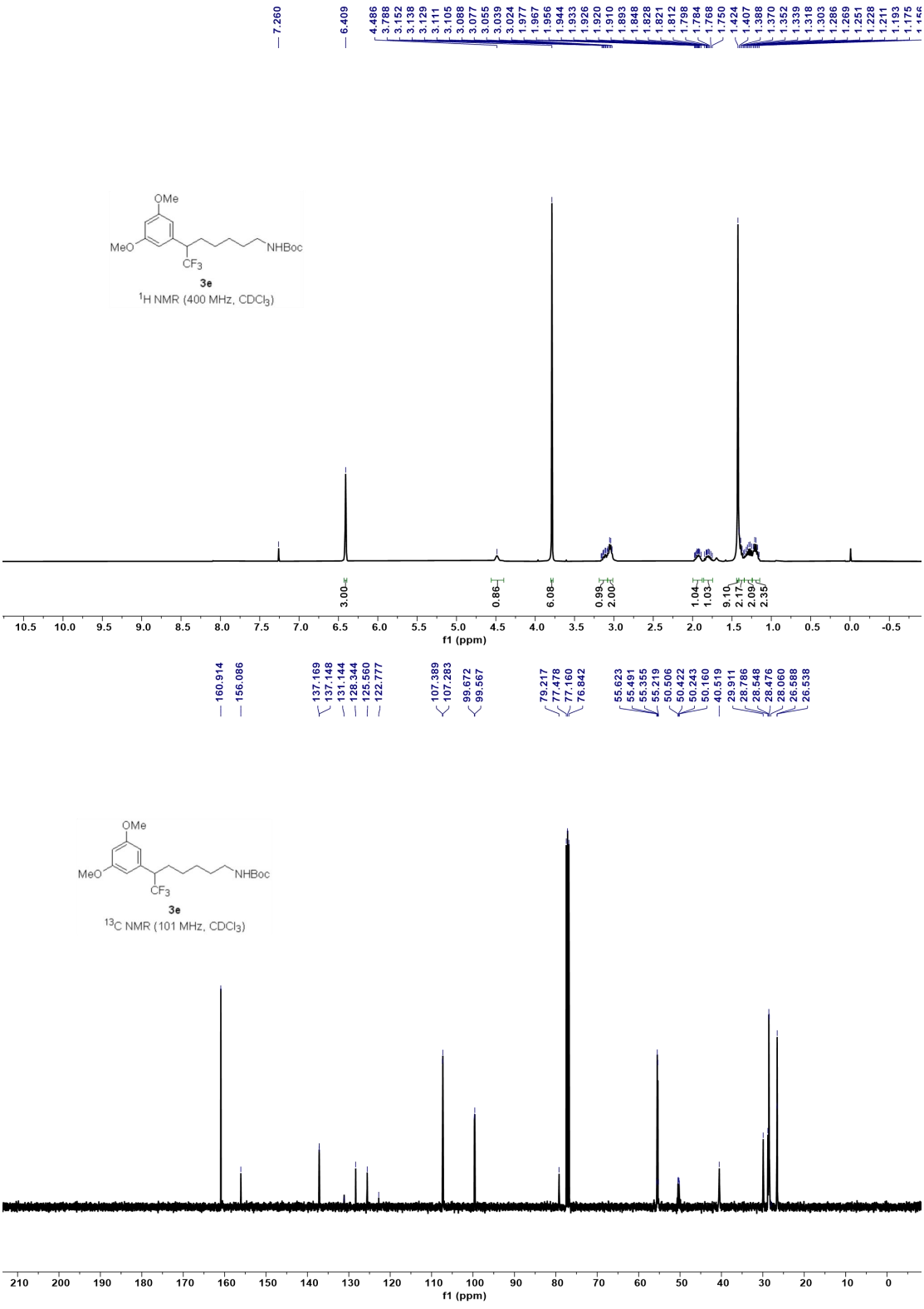

-69.503
-69.527
MeOC
NHBoc
CF3
3c
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)

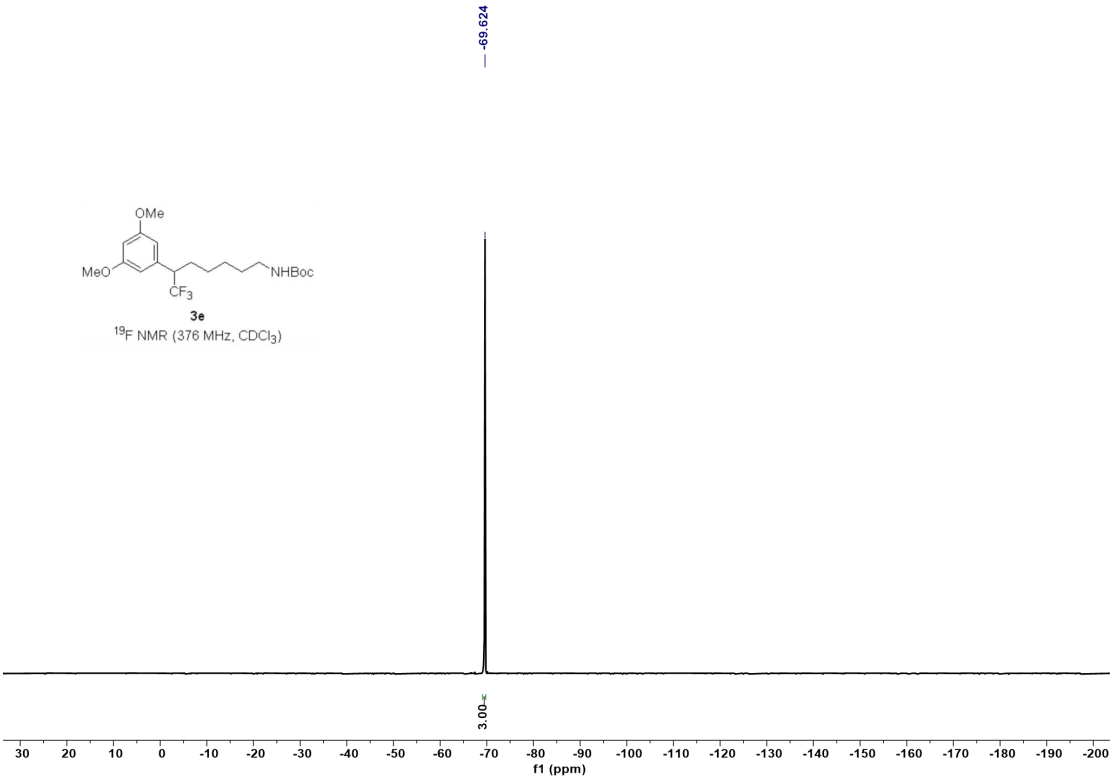

10.011
7.882
7.861
7.456
7.436
7.260
4.515


OHC
NHBoc
CF3
3d
1H NMR (400 MHz, CDCl3)
0.98
2.04
2.03
1.02
1.00
2.02
1.05
1.23
9.23
2.13
2.31
2.17
f1 (ppm)


191.880
191.847
156.073
141.732
141.710
136.261
130.757
130.130
129.882
129.779
127.973
125.185
122.402
79.249
77.478
77.160
76.842
50.678
50.413
50.144
49.904
40.418
29.866
28.651
28.529
28.453
26.467
OHC
NHBoc
CF3
3d
13C NMR (101 MHz, CDCl3)
f1 (ppm)

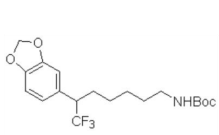

-69.404
-69.426
OHC
NHBoc
CF3
3d
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)


OMe
MeO
NHBoc
CF3
3e
1H NMR (400 MHz, CDCl3)
7.260
6.409
4.486
3.788
3.152
3.138
3.129
3.111
3.105
3.088
3.077
3.055
3.039
3.024
1.977
1.967
1.956
1.944
1.933
1.926
1.920
1.910
1.893
1.848
1.828
1.821
1.812
1.798
1.784
1.768
1.750
1.424
1.407
1.388
1.370
1.352
1.339
1.318
1.303
1.286
1.269
1.251
1.228
1.211
1.193
1.175
3.00
0.86
6.08
0.99
2.00
1.04
1.03
9.10
2.17
2.09
2.35
f1 (ppm)
160.914
156.086
137.169
137.148
131.144
128.344
125.560
122.777
107.389
107.283
99.672
99.567
79.217
77.478
77.160
76.842
55.623
55.491
55.355
55.219
50.506
50.422
50.243
50.160
40.519
29.911
28.786
28.548
28.476
28.060
26.588
26.538
OMe
MeO
NHBoc
CF3
3e
13C NMR (101 MHz, CDCl3)
f1 (ppm)



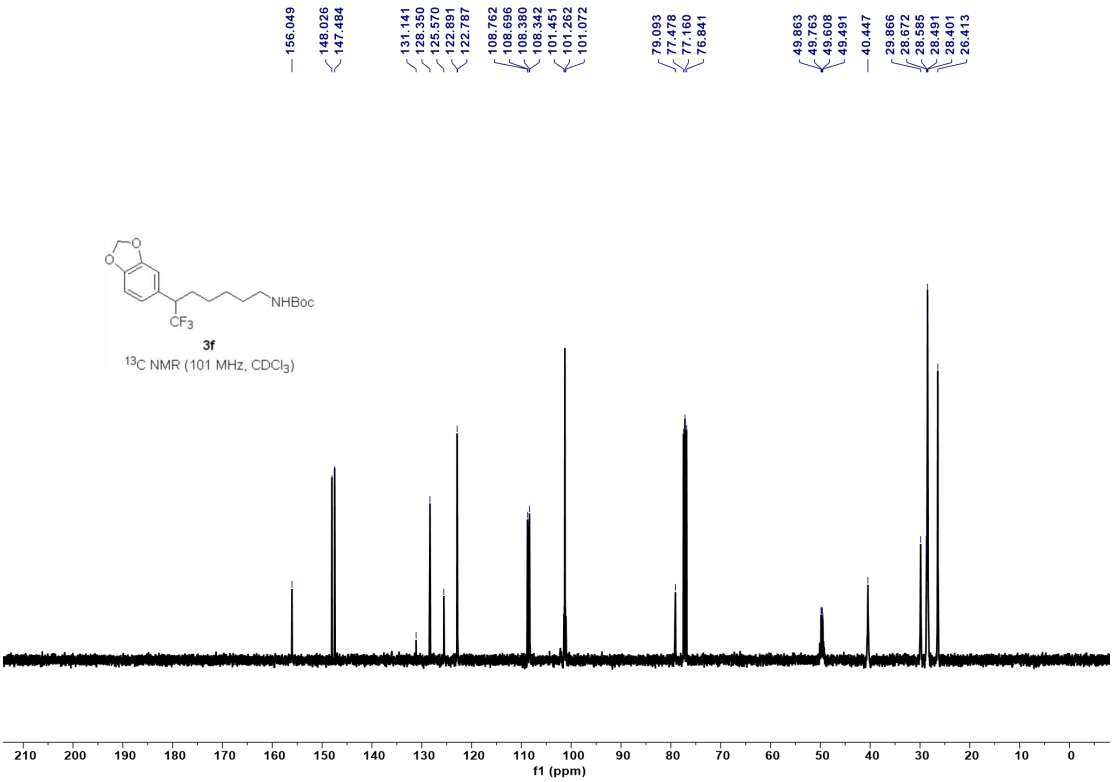
3e

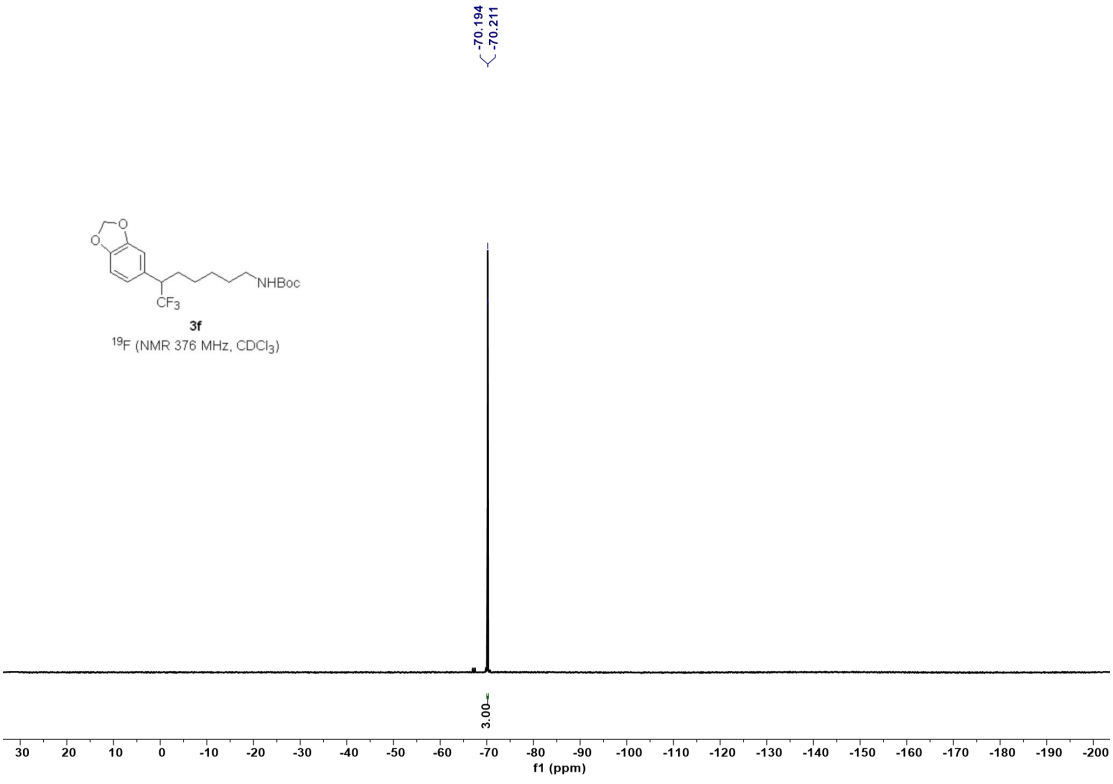
^{19}F NMR (376 MHz, $CDCl_3$)

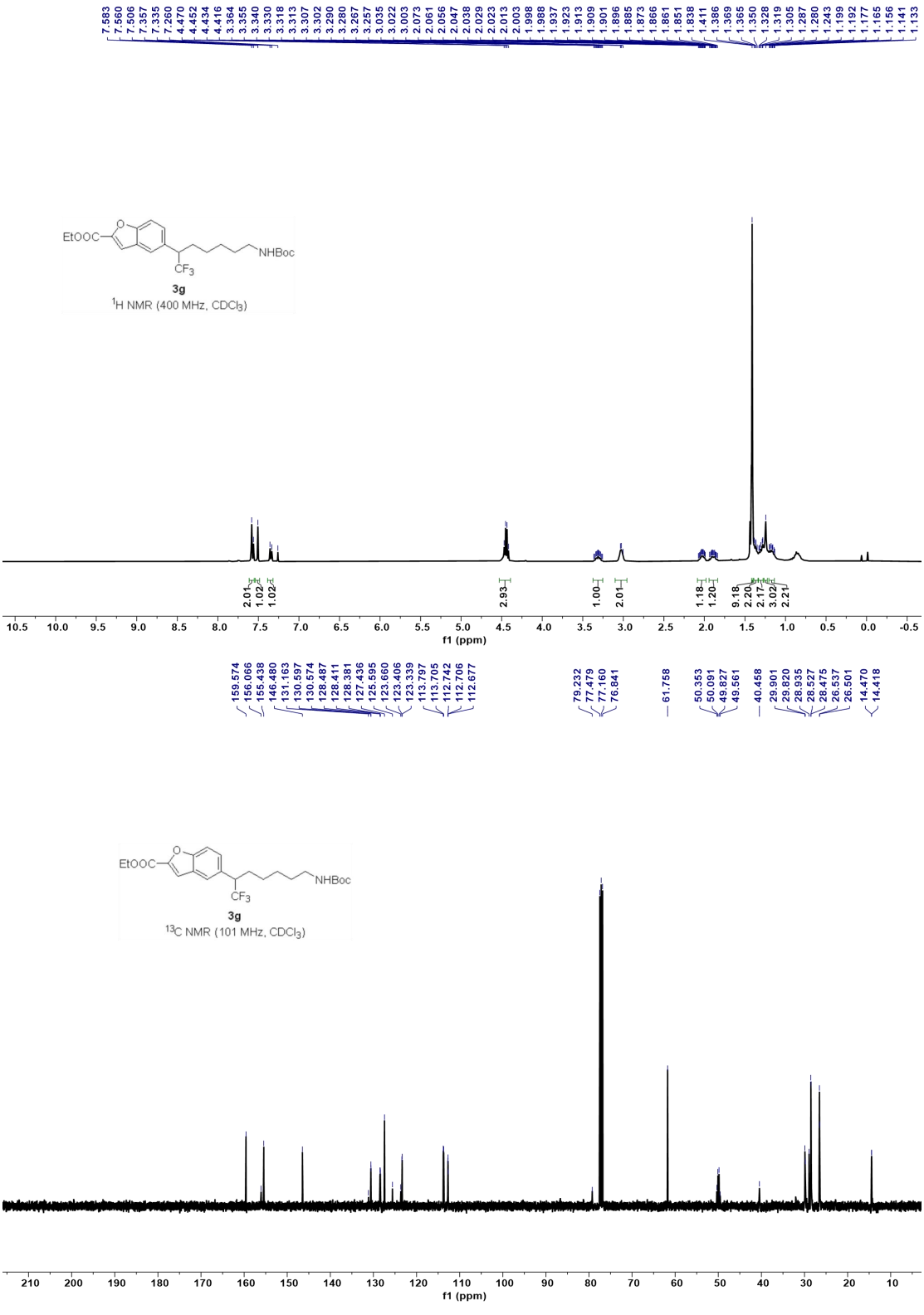


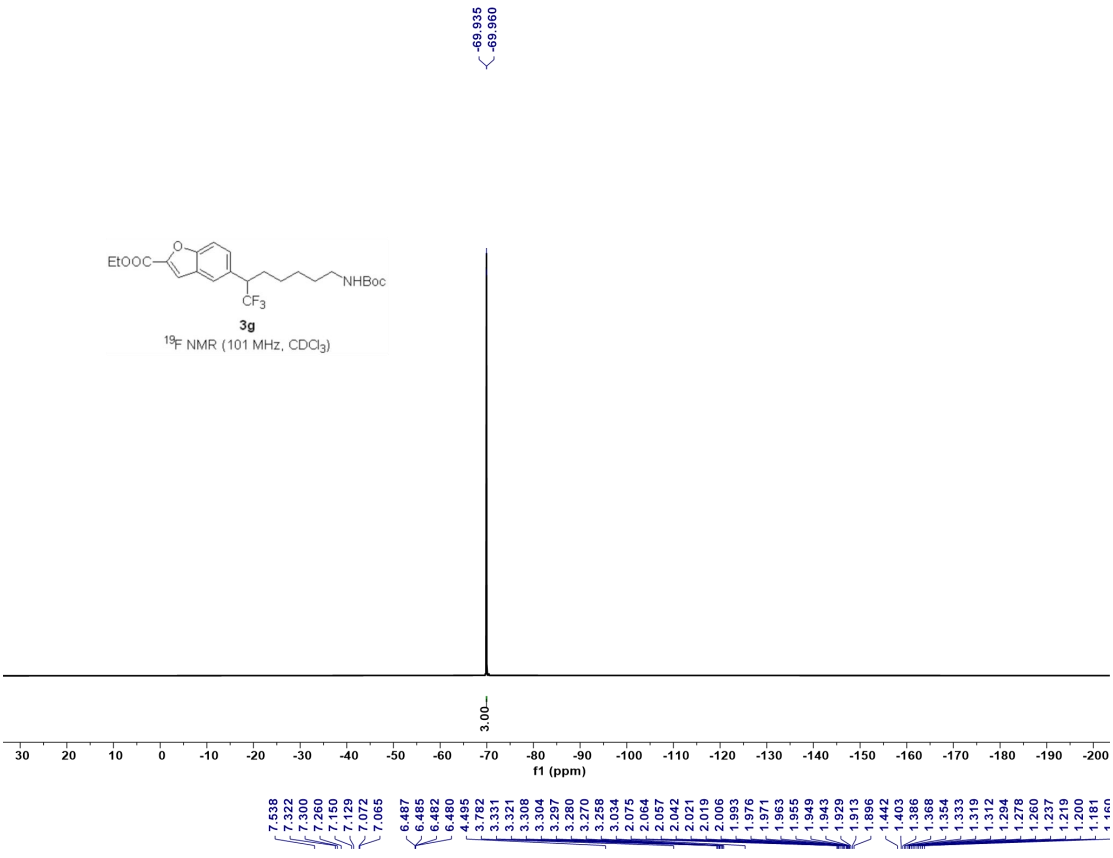
3f

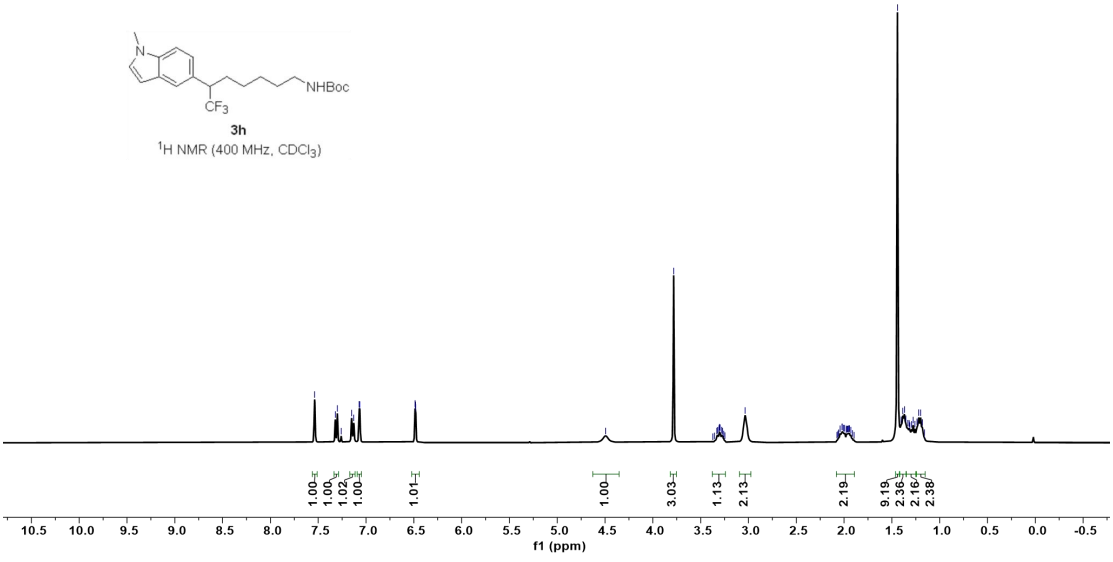
^{1}H NMR (400 MHz, $CDCl_3$)

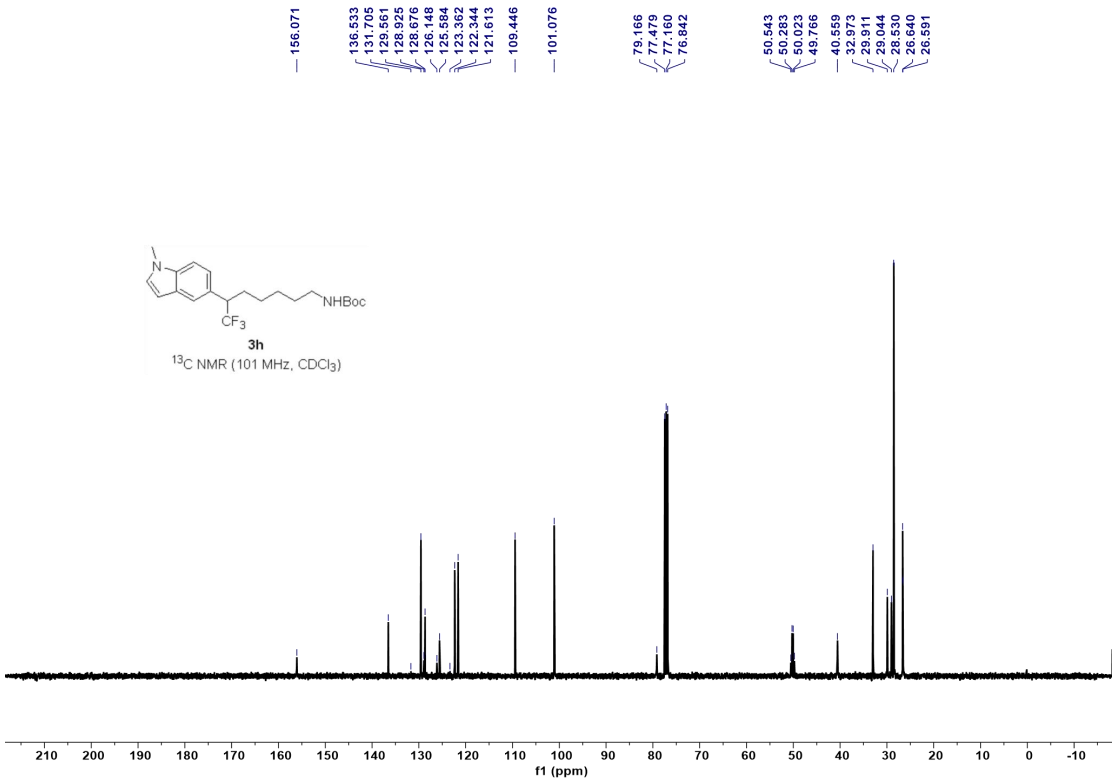

156.049
148.026
147.484
131.141
128.350
125.570
122.891
122.787
108.762
108.696
108.380
108.342
101.451
101.262
101.072
79.093
77.478
77.160
76.841
49.863
49.763
49.608
49.491
40.447
29.866
28.672
28.585
28.491
28.401
26.413
NHBoc
CF3
3f
13C NMR (101 MHz, CDCl3)
f1 (ppm)

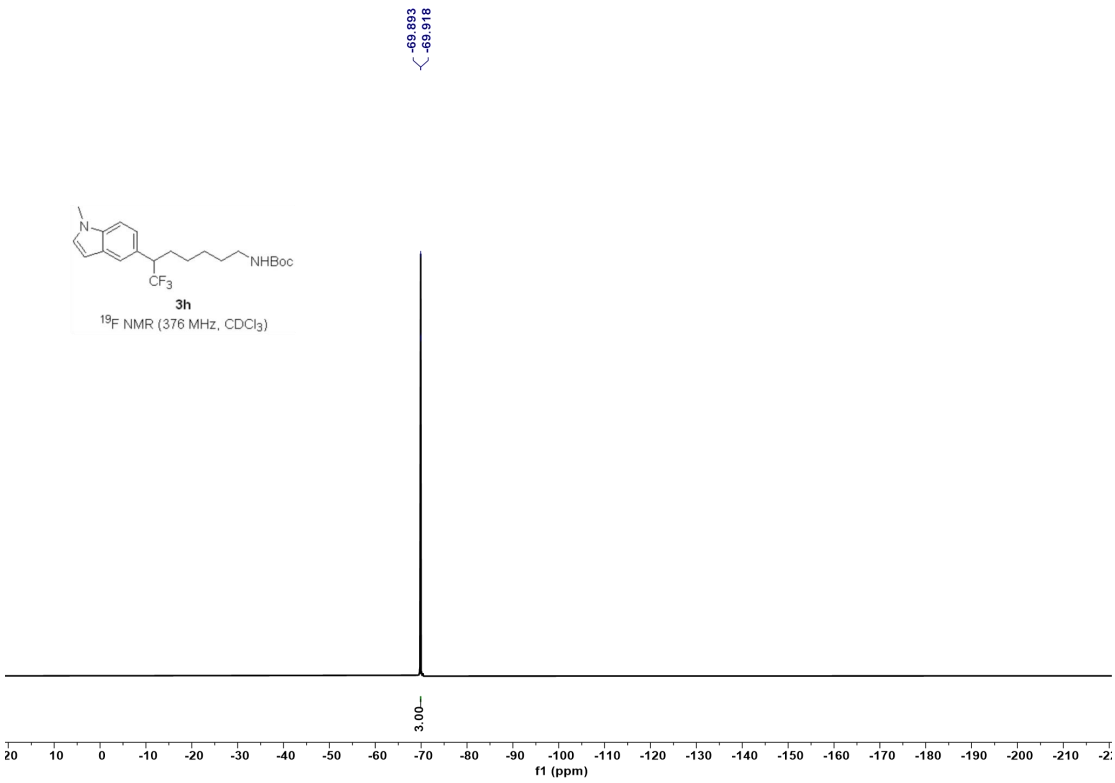

-70.194
-70.211
NHBoc
CF3
3f
19F (NMR 376 MHz, CDCl3)
3.00
f1 (ppm)

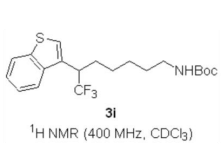

7.583 7.560 7.506 7.357 7.335 7.260 4.470 4.452 4.434 4.416 3.364 3.355 3.340 3.330 3.318 3.313 3.307 3.302 3.290 3.280 3.267 3.257 3.035 3.022 3.003 2.073 2.061 2.056 2.047 2.038 2.029 2.023 2.013 2.003 1.998 1.988 1.937 1.923 1.913 1.909 1.901 1.896 1.885 1.873 1.866 1.861 1.851 1.838 1.411 1.386 1.369 1.365 1.350 1.328 1.319 1.305 1.287 1.280 1.243 1.199 1.192 1.177 1.165 1.156 1.141 1.133
EtOOC
NHBoc
CF3
3g
1H NMR (400 MHz, CDCl3)
2.01 1.02 1.02 2.93 1.00 2.01 1.18 1.20 9.18 2.20 2.17 3.02 2.21
f1 (ppm)
159.574 156.066 155.438 146.480 131.163 130.597 130.574 128.487 128.411 128.381 127.436 125.595 123.660 123.406 123.339 113.797 113.705 112.742 112.706 112.677 79.232 77.479 77.160 76.841 61.758 50.353 50.091 49.827 49.561 40.458 29.901 29.820 28.935 28.527 28.475 26.537 26.501 14.470 14.418
EtOOC
NHBoc
CF3
3g
13C NMR (101 MHz, CDCl3)
f1 (ppm)


-69.935
-69.960
EtOOC
O
NHBoc
CF3
3g
19F NMR (101 MHz, CDCl3)
3.00
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)
7.538 7.322 7.300 7.260 7.150 7.129 7.072 7.065 6.487 6.485 6.482 6.480 4.495 3.782 3.331 3.321 3.308 3.304 3.297 3.280 3.270 3.258 3.034 2.075 2.064 2.057 2.042 2.021 2.019 2.006 1.993 1.976 1.971 1.963 1.955 1.949 1.943 1.929 1.913 1.896 1.442 1.403 1.386 1.368 1.354 1.333 1.319 1.312 1.294 1.278 1.260 1.237 1.219 1.200 1.181


N
NHBoc
CF3
3h
1H NMR (400 MHz, CDCl3)
1.00 1.00 1.02 1.00 1.01 1.00 3.03 1.13 2.13 2.19 9.19 2.36 2.16 2.38
10.5 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 -0.5
f1 (ppm)

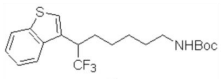

156.071
136.533
131.705
129.561
128.925
128.676
126.148
125.584
123.362
122.344
121.613
109.446
101.076
79.166
77.479
77.160
76.842
50.543
50.283
50.023
49.766
40.559
32.973
29.911
29.044
28.530
26.640
26.591
NHBoc
CF3
3h
13C NMR (101 MHz, CDCl3)
f1 (ppm)


-69.893
-69.918
NHBoc
CF3
3h
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)



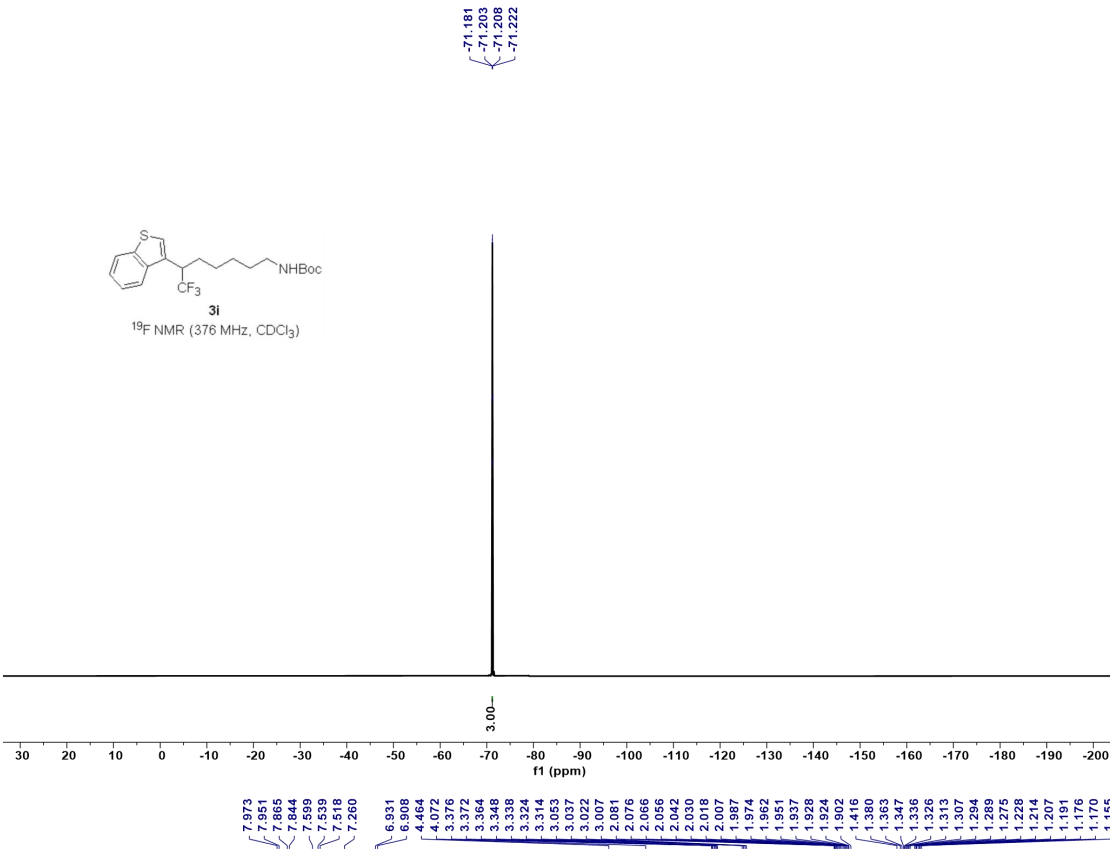
3i

^{1}H NMR (400 MHz, $CDCl_3$)



3i

^{13}C NMR (101 MHz, $CDCl_3$)


-71.181
-71.203
-71.208
-71.222
S
NHBoc
CF3
3i
19F NMR (376 MHz, CDCl3)
3.00
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)
7.973
7.951
7.865
7.844
7.599
7.539
7.518
7.260
6.931
6.908
4.464
4.072
3.376
3.372
3.364
3.348
3.338
3.324
3.314
3.053
3.037
3.022
3.007
2.081
2.076
2.066
2.056
2.042
2.030
2.018
2.007
1.987
1.974
1.962
1.951
1.937
1.928
1.924
1.902
1.416
1.380
1.363
1.347
1.336
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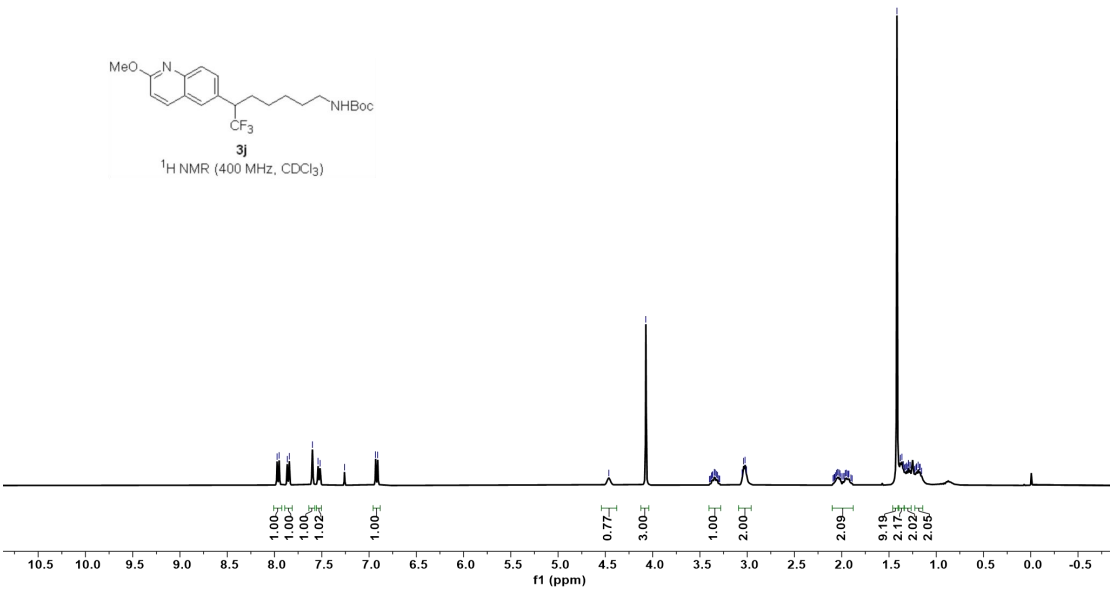
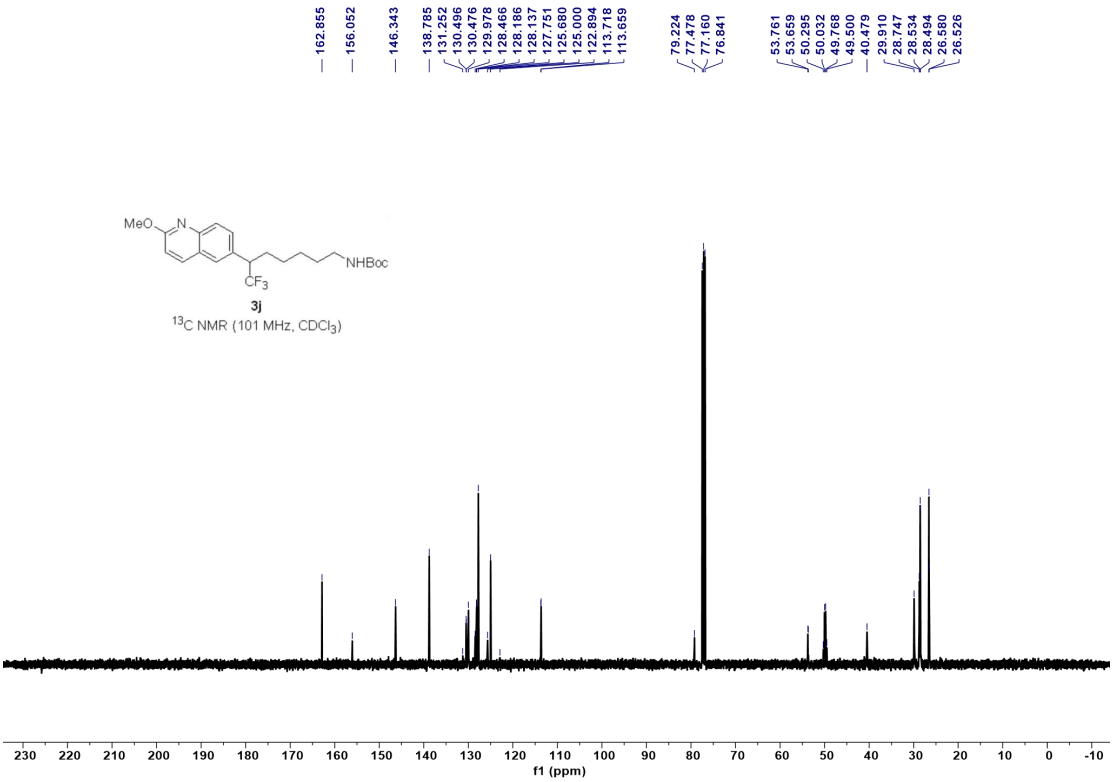
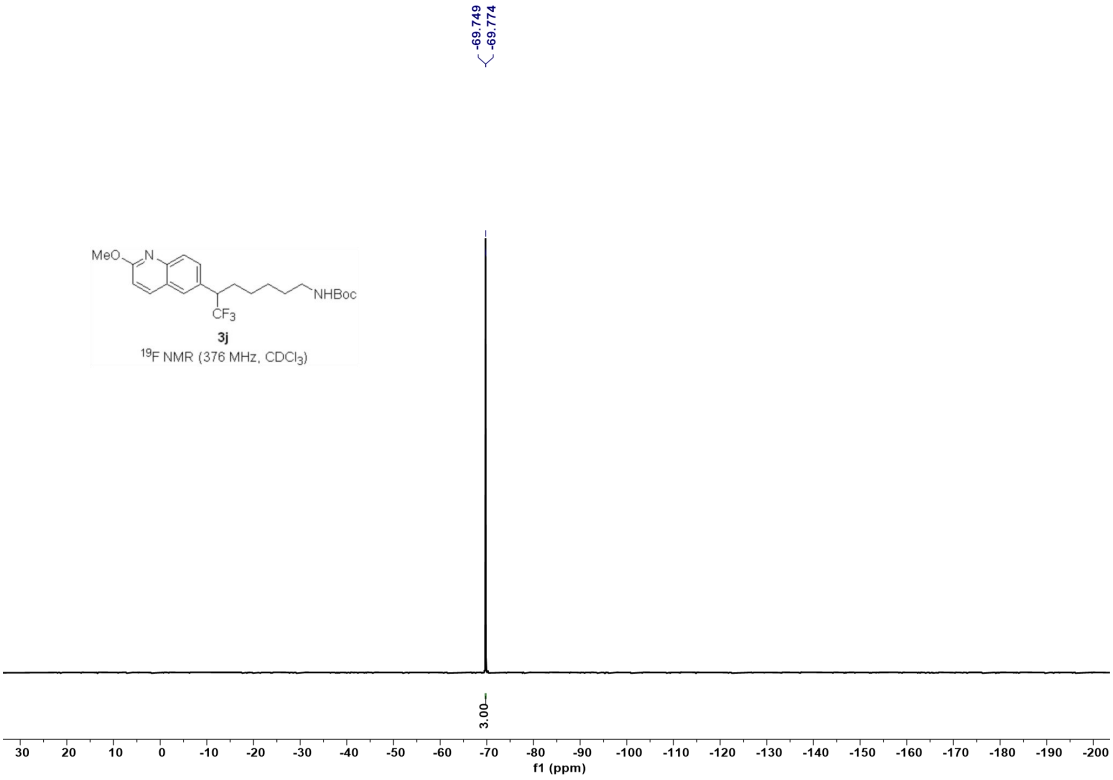
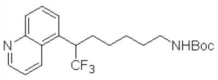
1.228
1.214
1.207
1.191
1.176
1.170


MeO
N
NHBoc
CF3
3j
1H NMR (400 MHz, CDCl3)
1.00
1.00
1.00
1.02
1.00
0.77
3.00
1.00
2.00
2.09
9.19
2.17
2.02
2.05
10.5 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 -0.5
f1 (ppm)

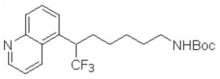

162.855
156.052
146.343
138.785
131.252
130.496
130.476
129.978
128.466
128.186
128.137
127.751
125.680
125.000
122.894
113.718
113.659
79.224
77.478
77.160
76.841
53.761
53.659
50.295
50.032
49.768
49.500
40.479
29.910
28.747
28.534
28.494
26.580
26.526
MeO
N
NHBoc
CF3
3j
13C NMR (101 MHz, CDCl3)
f1 (ppm)


-69.749
-69.774
MeO
N
NHBoc
CF3
3j
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)



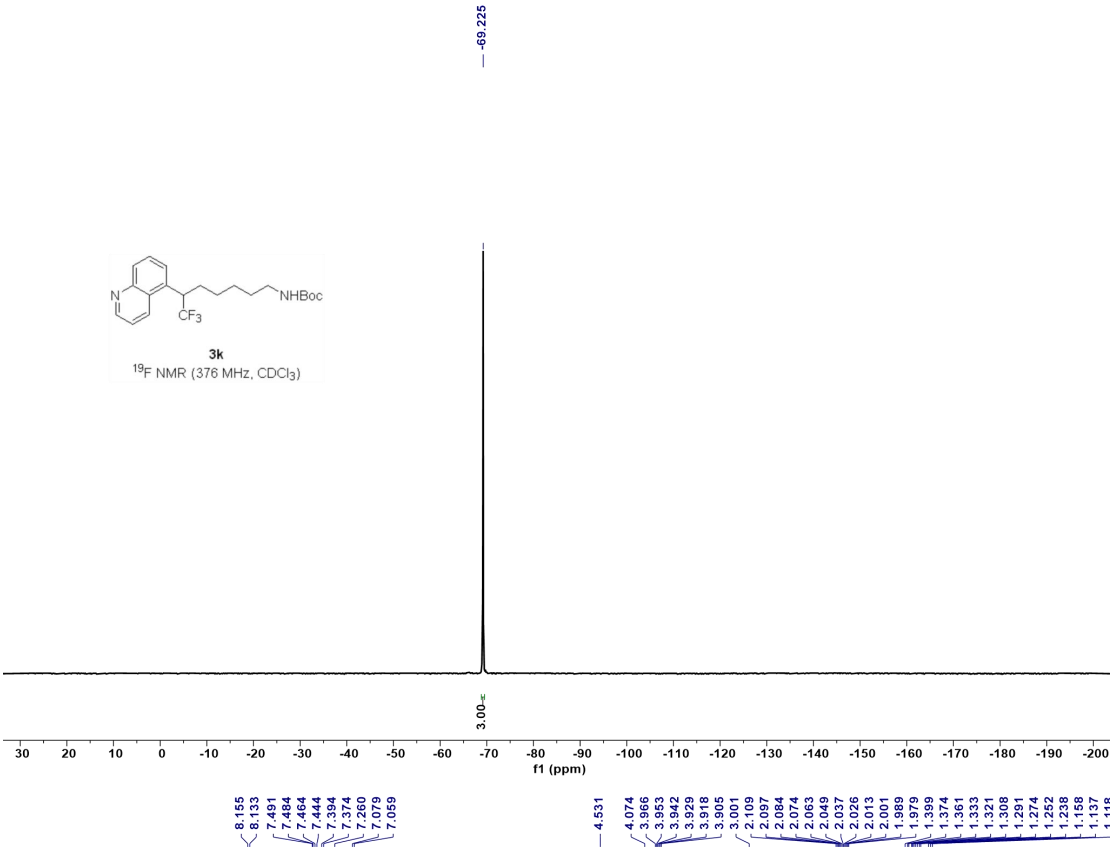
3k

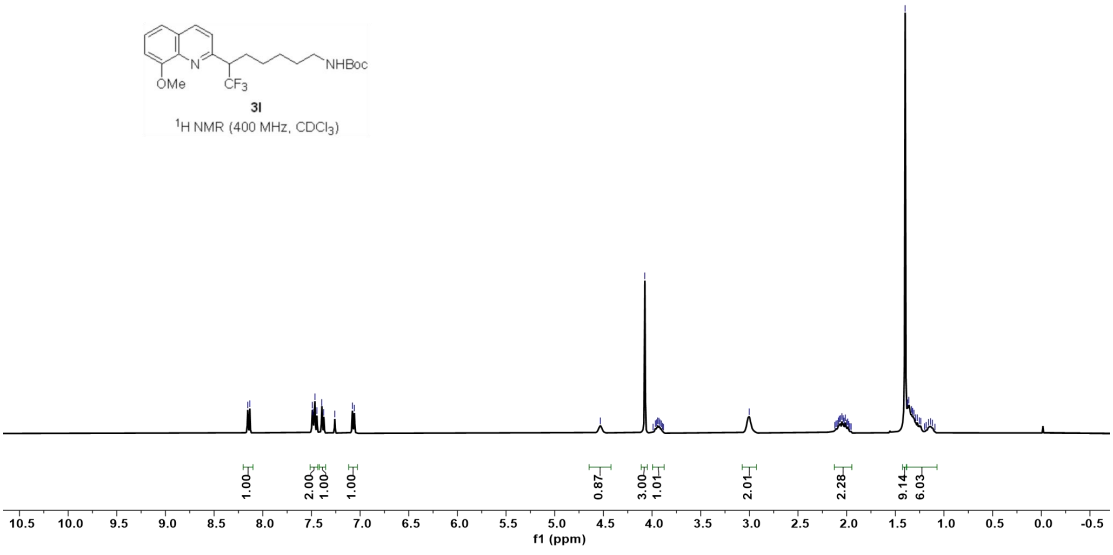
^{1}H NMR (400 MHz, $CDCl_3$)

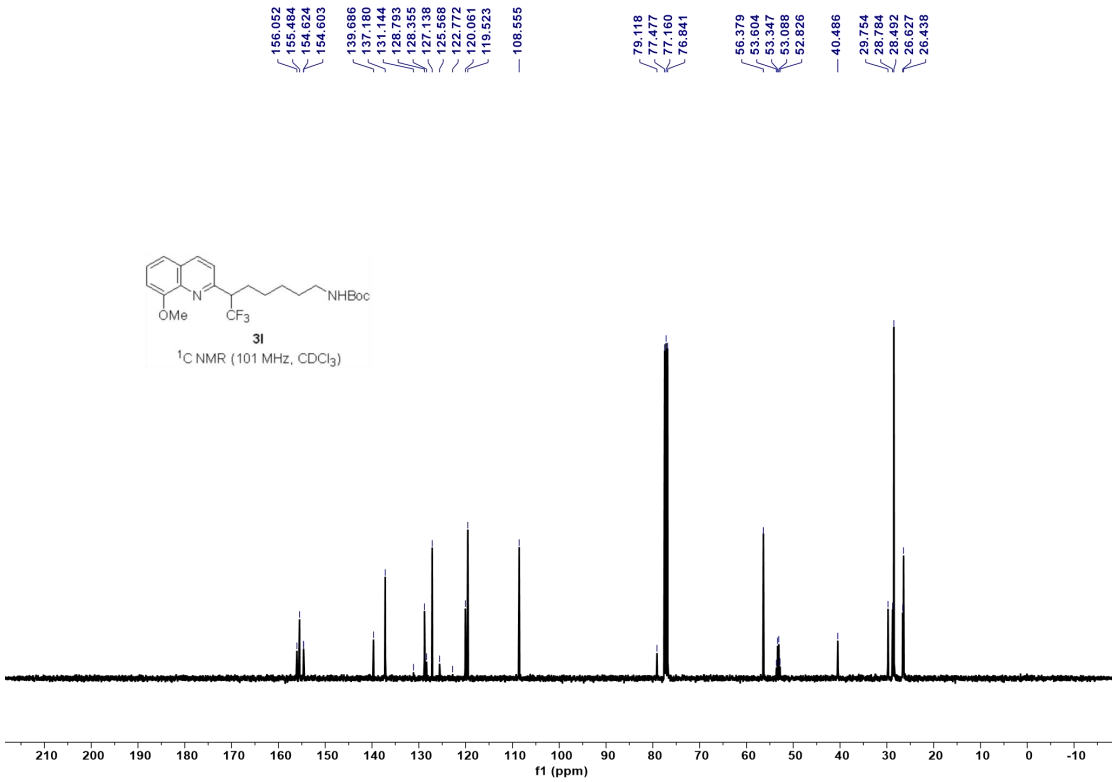


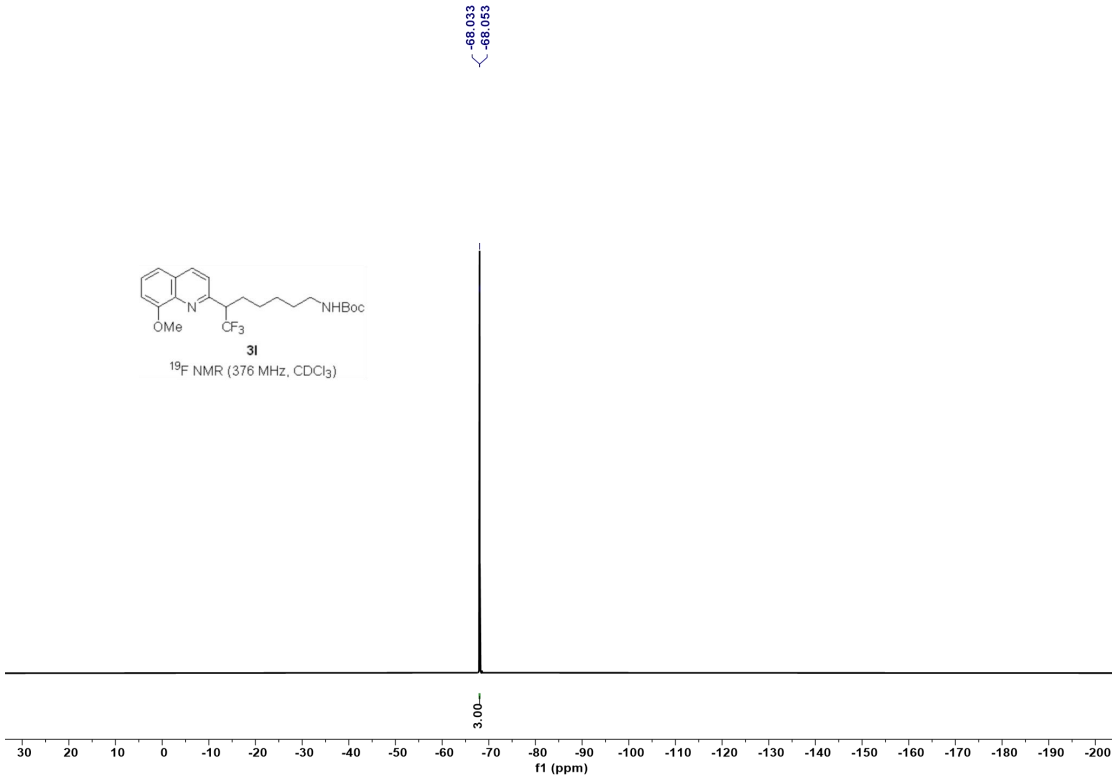
3k

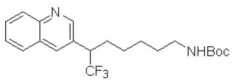
^{13}C NMR (101 MHz, $CDCl_3$)


-69.225
NHBoc
CF3
3k
19F NMR (376 MHz, CDCl3)
3.00
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)
8.155 8.133 7.491 7.484 7.464 7.444 7.394 7.374 7.260 7.079 7.059
4.531 4.074 3.966 3.953 3.942 3.929 3.918 3.905 3.001 2.109 2.097 2.084 2.074 2.063 2.049 2.037 2.026 2.013 2.001 1.989 1.979 1.399 1.374 1.361 1.333 1.321 1.308 1.291 1.274 1.252 1.238 1.158 1.137


N
NHBoc
OMe
CF3
3l
1H NMR (400 MHz, CDCl3)
1.00 2.00 1.00 1.00 0.87 3.00 1.01 2.01 2.28 9.14 6.03
10.5 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 -0.5
f1 (ppm)

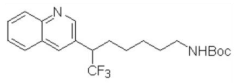

156.052
155.484
154.624
154.603
139.686
137.180
131.144
128.793
128.355
127.138
125.568
122.772
120.061
119.523
108.555
79.118
77.477
77.160
76.841
56.379
53.604
53.347
53.088
52.826
40.486
29.754
28.784
28.492
26.627
26.438
NHBoc
OMe
CF3
3l
1C NMR (101 MHz, CDCl3)
f1 (ppm)


-68.033
-68.053
NHBoc
OMe
CF3
3l
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)



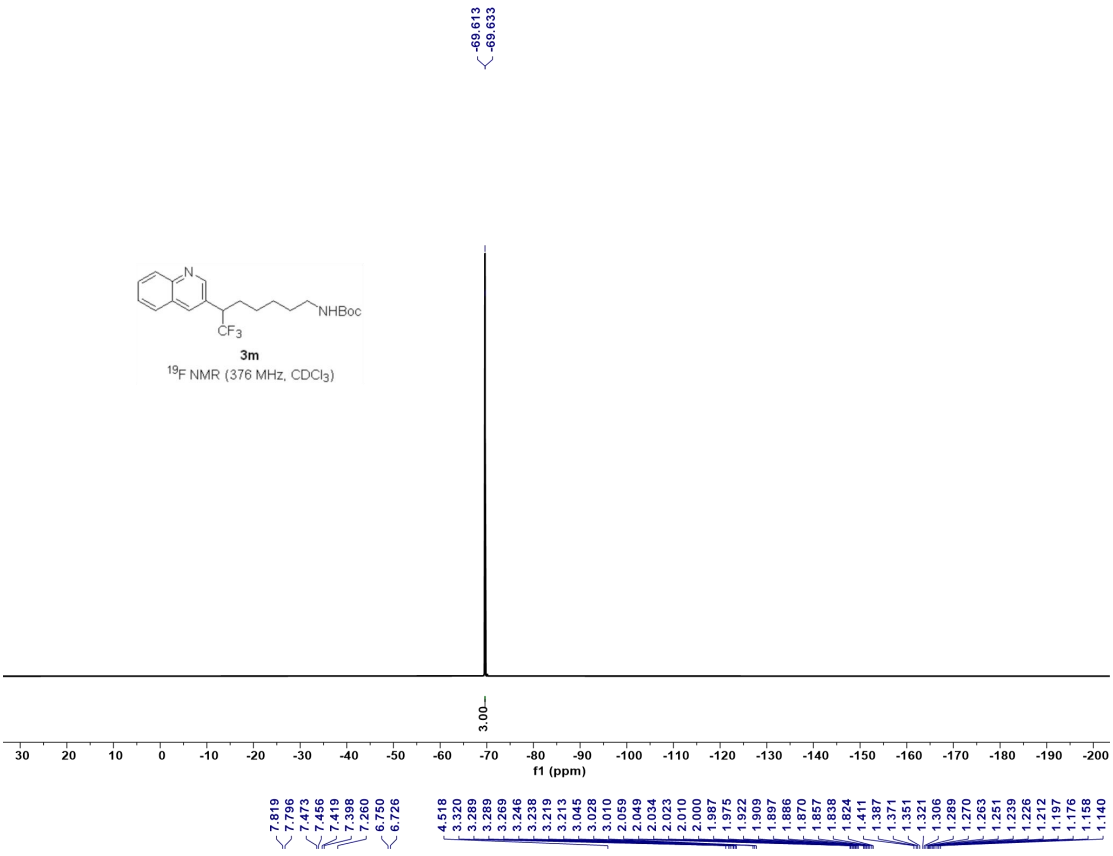
3m

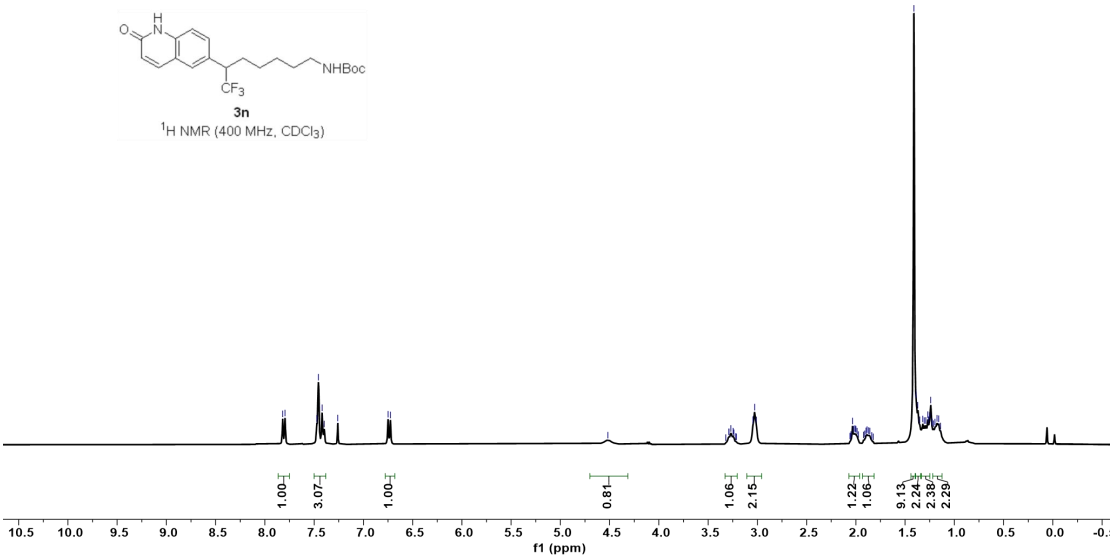
^{1}H NMR (400 MHz, $CDCl_3$)

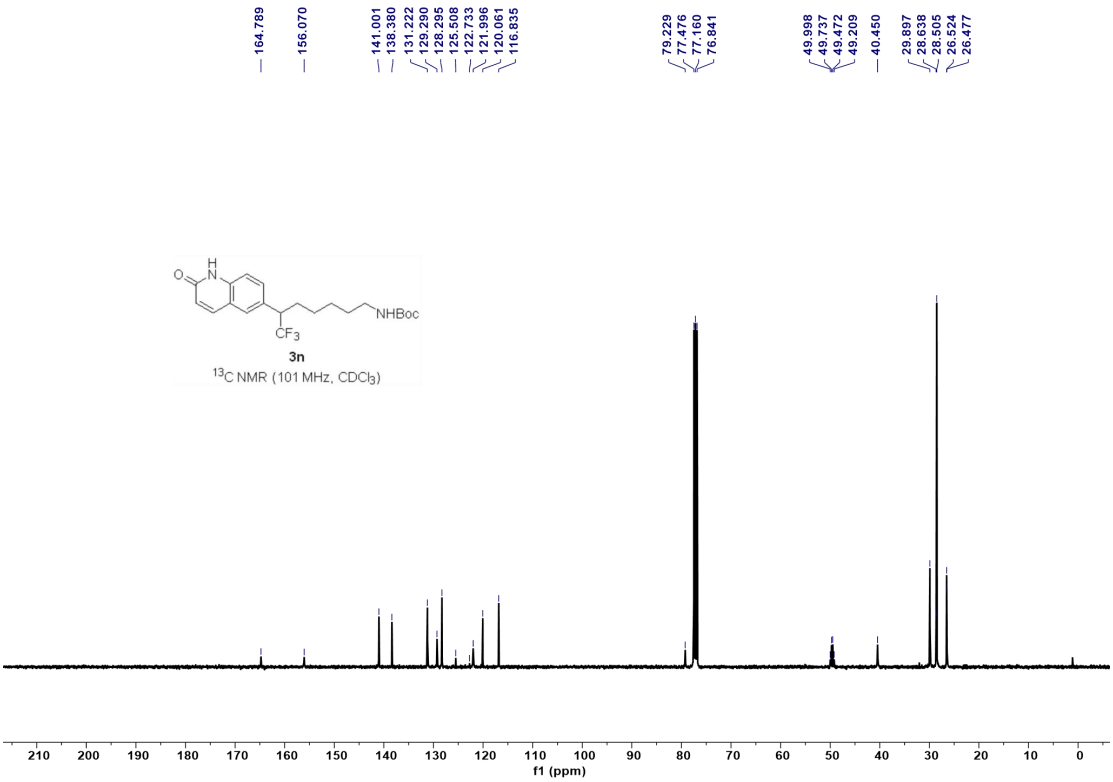


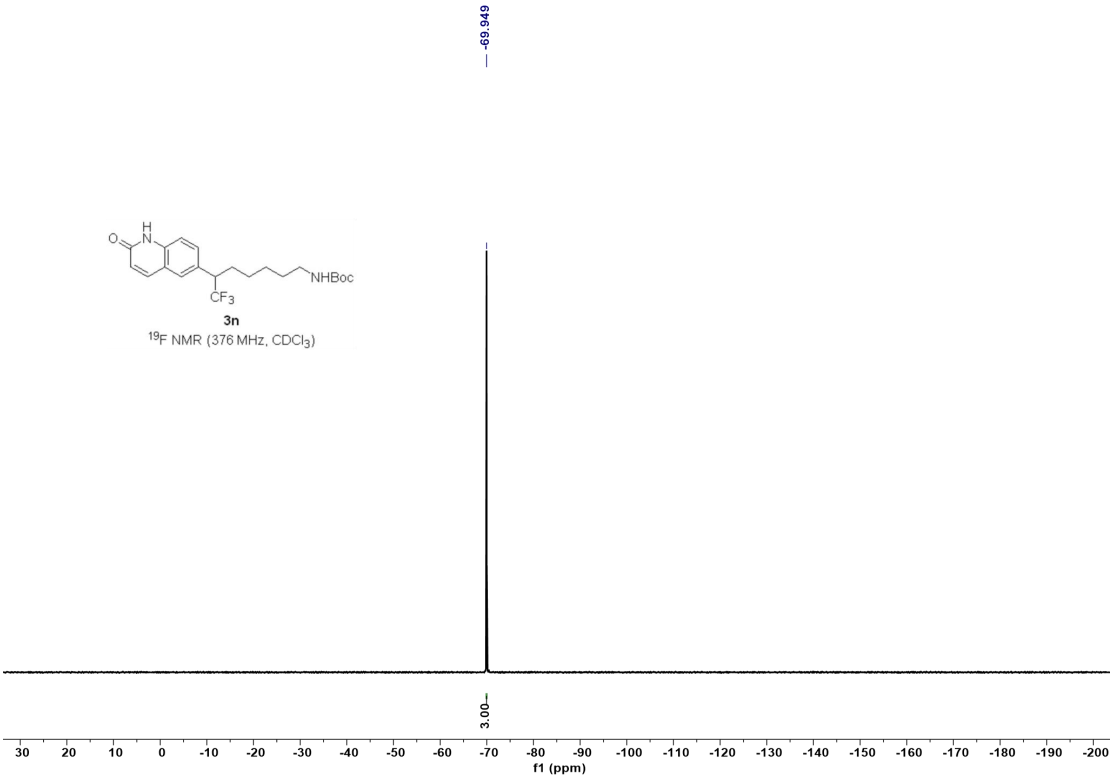
3m

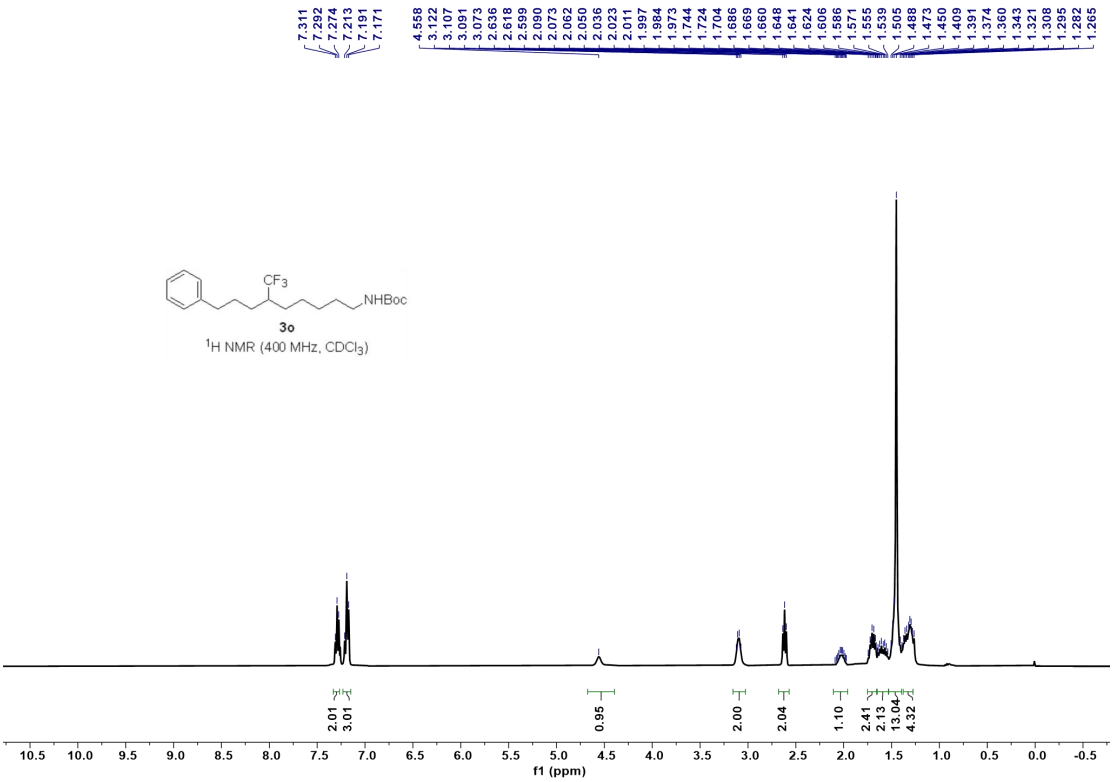
^{13}C NMR (101 MHz, $CDCl_3$)

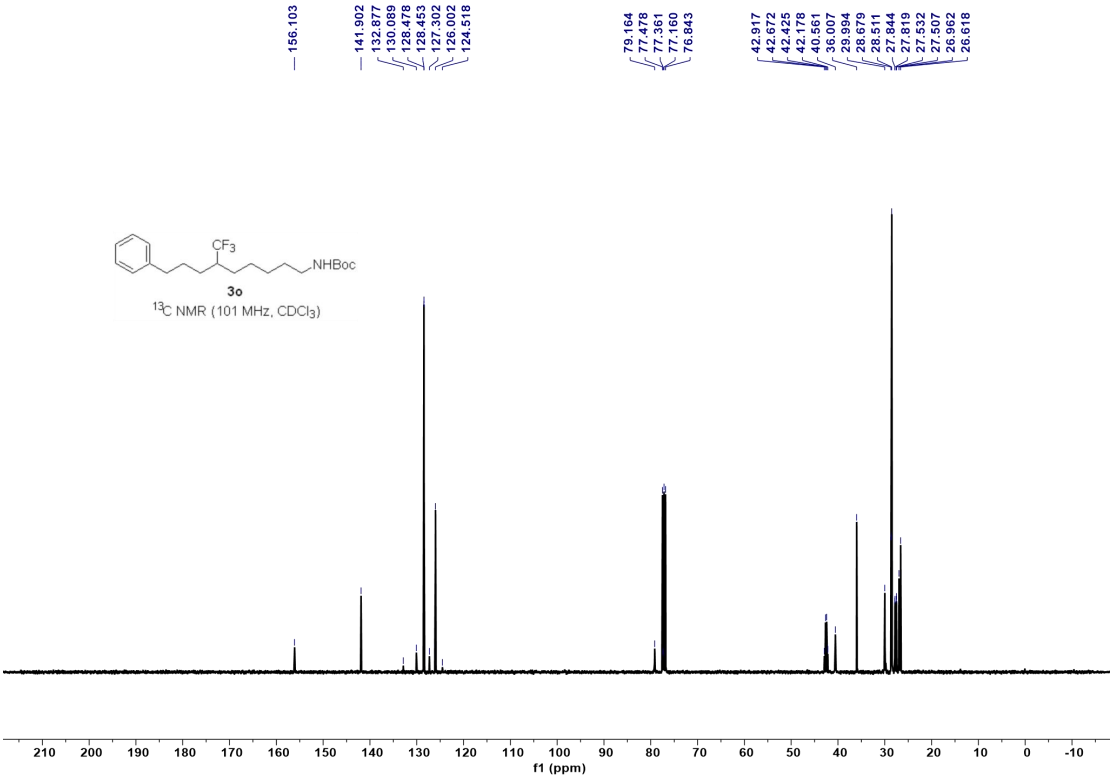

-69.613
-69.633
NHBoc
CF3
3m
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)

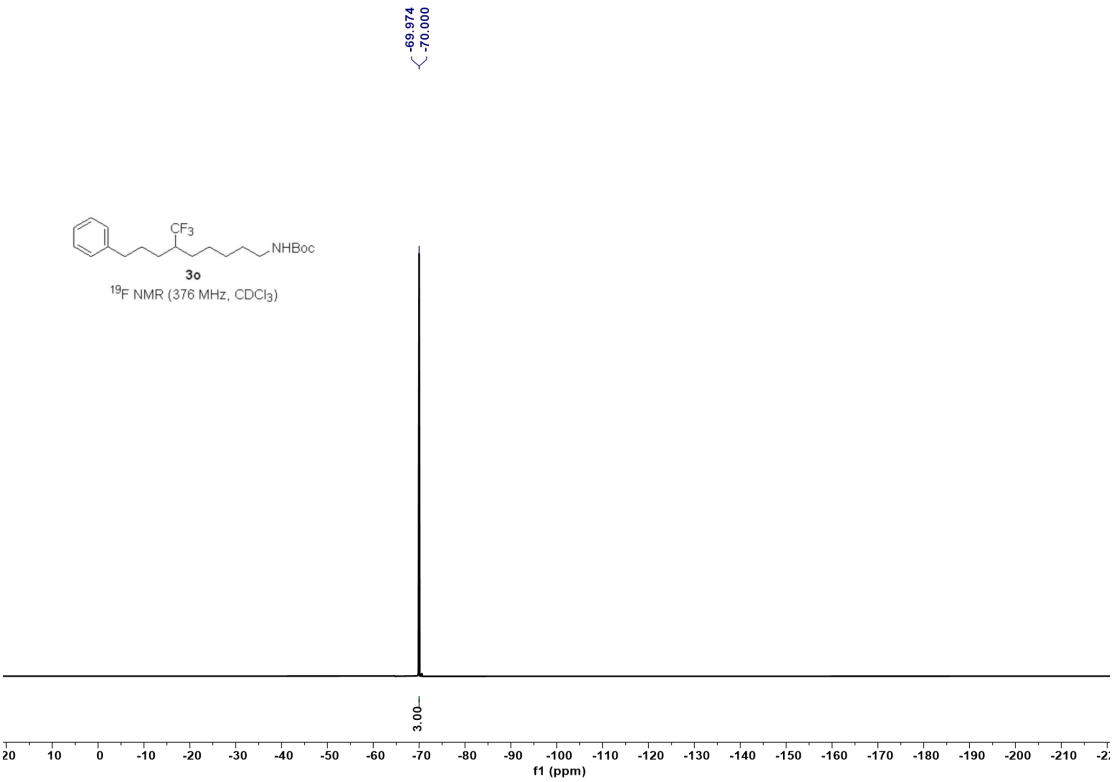

NHBoc
CF3
3n
1H NMR (400 MHz, CDCl3)
1.00
3.07
1.00
0.81
1.06
2.15
1.22
1.06
9.13
2.24
2.38
2.29
f1 (ppm)

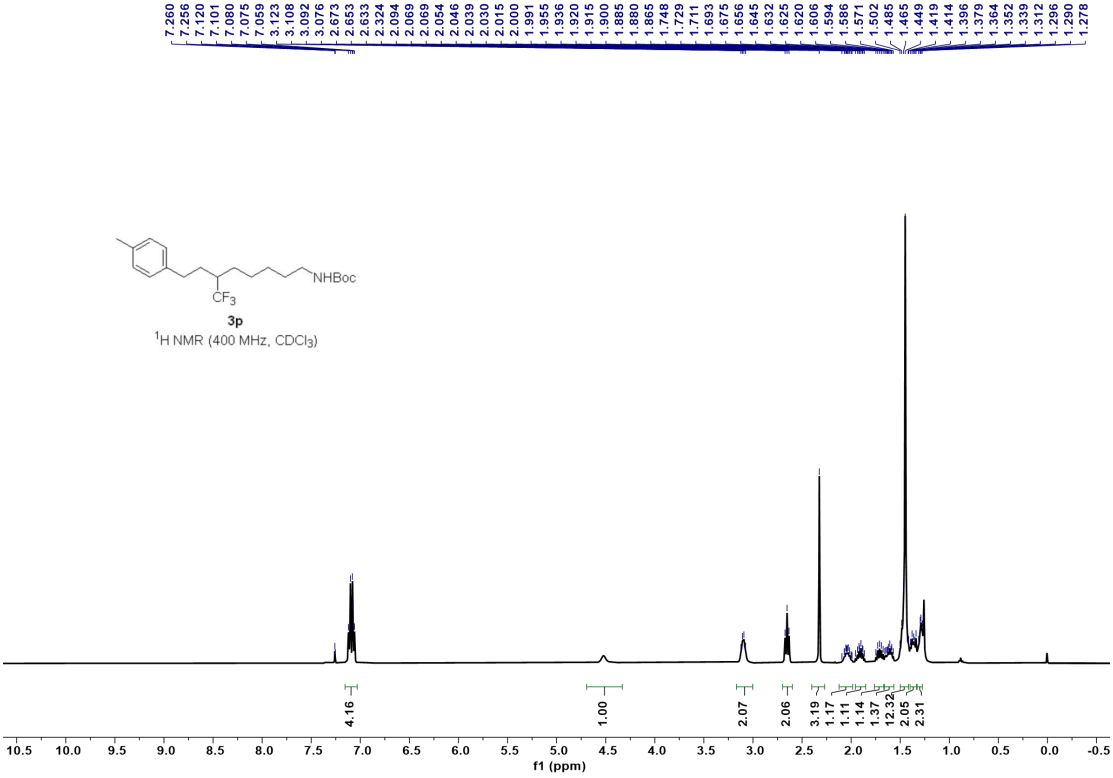

164.789
156.070
141.001
138.380
131.222
129.290
128.295
125.508
122.733
121.996
120.061
116.835
79.229
77.476
77.160
76.841
49.998
49.737
49.472
49.209
40.450
29.897
28.638
28.505
26.524
26.477
NHBoc
CF3
3n
13C NMR (101 MHz, CDCl3)
f1 (ppm)

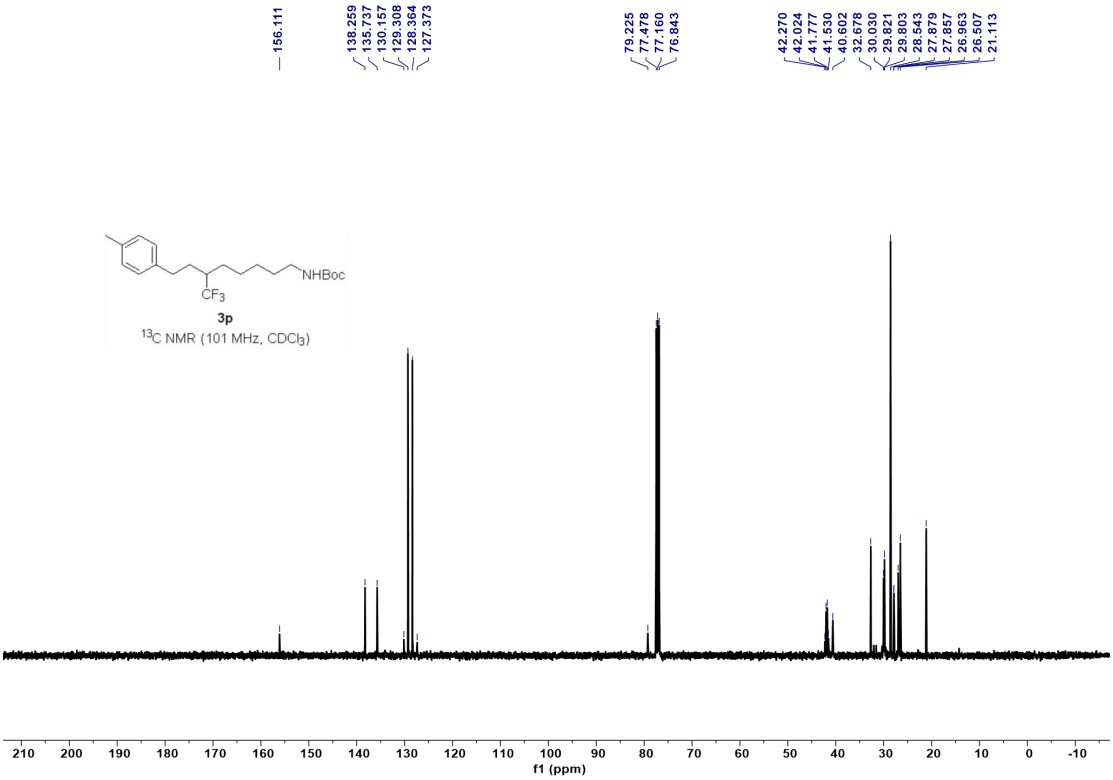

-69.949
NHBoc
CF3
3n
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)

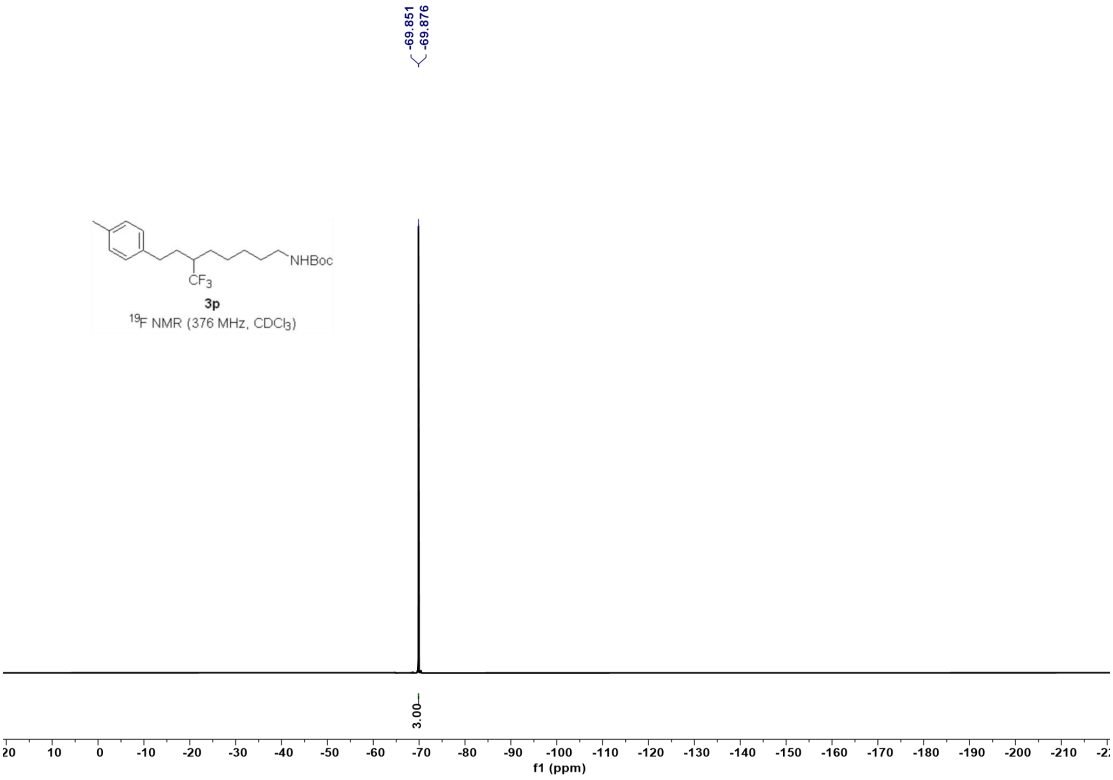

7.311
7.292
7.274
7.213
7.191
7.171
4.558
3.122
3.107
3.091
3.073
2.636
2.618
2.599
2.090
2.073
2.062
2.050
2.036
2.023
2.011
1.997
1.984
1.973
1.744
1.724
1.704
1.686
1.669
1.660
1.648
1.641
1.624
1.606
1.586
1.571
1.555
1.539
1.505
1.488
1.473
1.450
1.409
1.391
1.374
1.360
1.343
1.321
1.308
1.295
1.282
1.265
CF3
NHBoc
3o
1H NMR (400 MHz, CDCl3)
2.01
3.01
0.95
2.00
2.04
1.10
2.41
2.13
13.04
4.32
f1 (ppm)

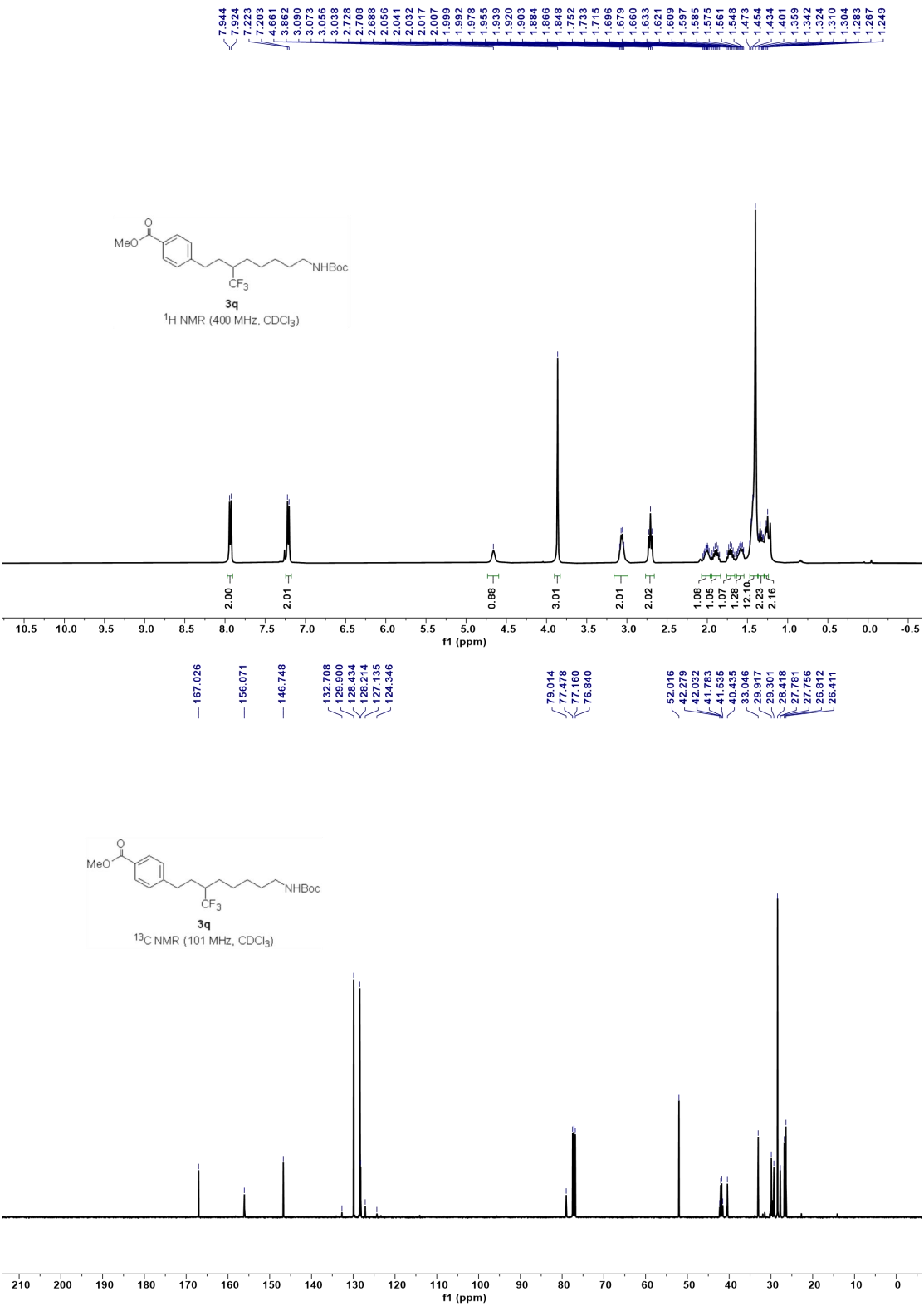

156.103
141.902
132.877
130.089
128.478
128.453
127.302
126.002
124.518
79.164
77.478
77.361
77.160
76.843
42.917
42.672
42.425
42.178
40.561
36.007
29.994
28.679
28.511
27.844
27.819
27.532
27.507
26.962
26.618
CF3
NHBoc
3o
13C NMR (101 MHz, CDCl3)
f1 (ppm)

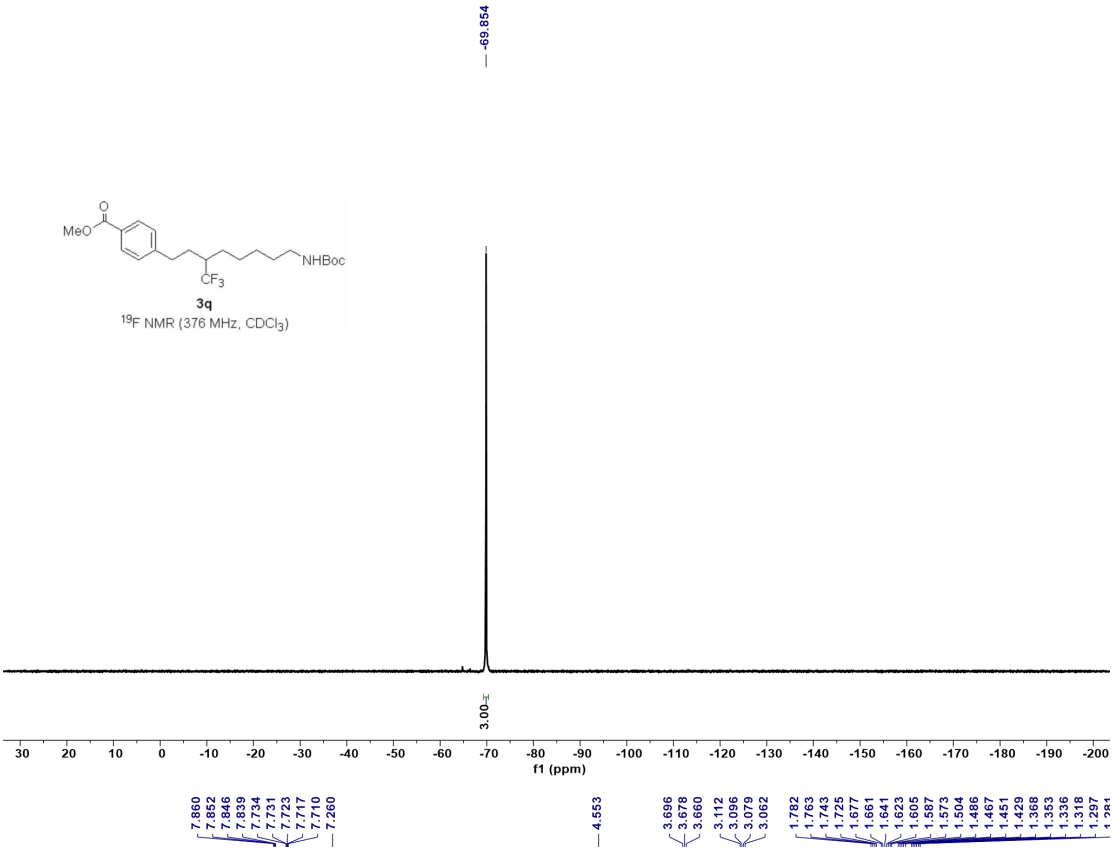

-69.974
-70.000
CF3
NHBoc
3o
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)

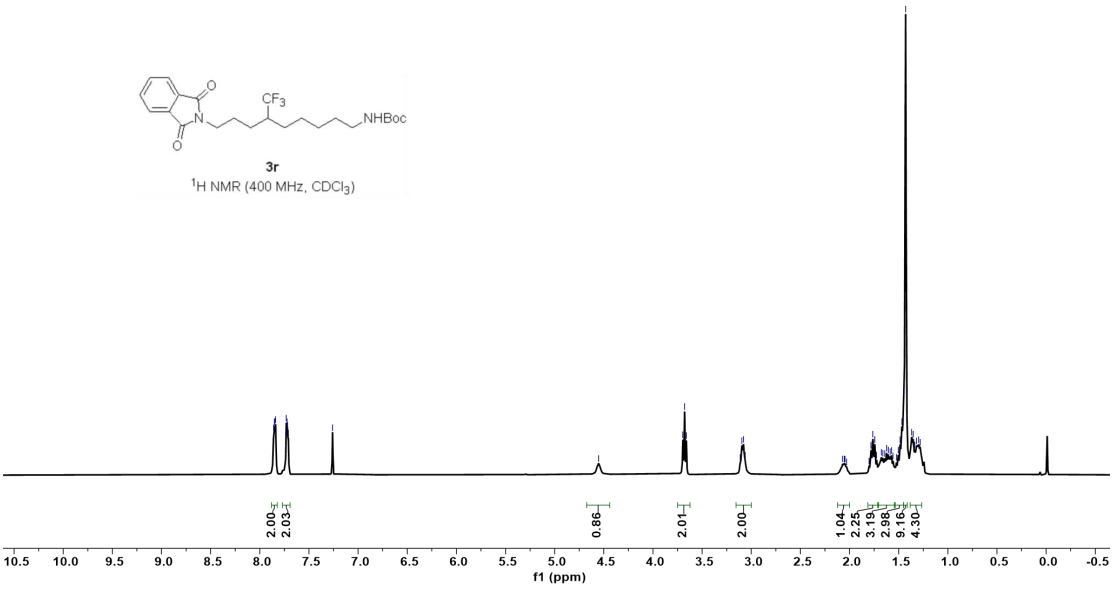

NHBoc
CF3
3p
1H NMR (400 MHz, CDCl3)
4.16
1.00
2.07
2.06
3.19
1.17
1.11
1.14
1.37
12.32
2.05
2.31
f1 (ppm)

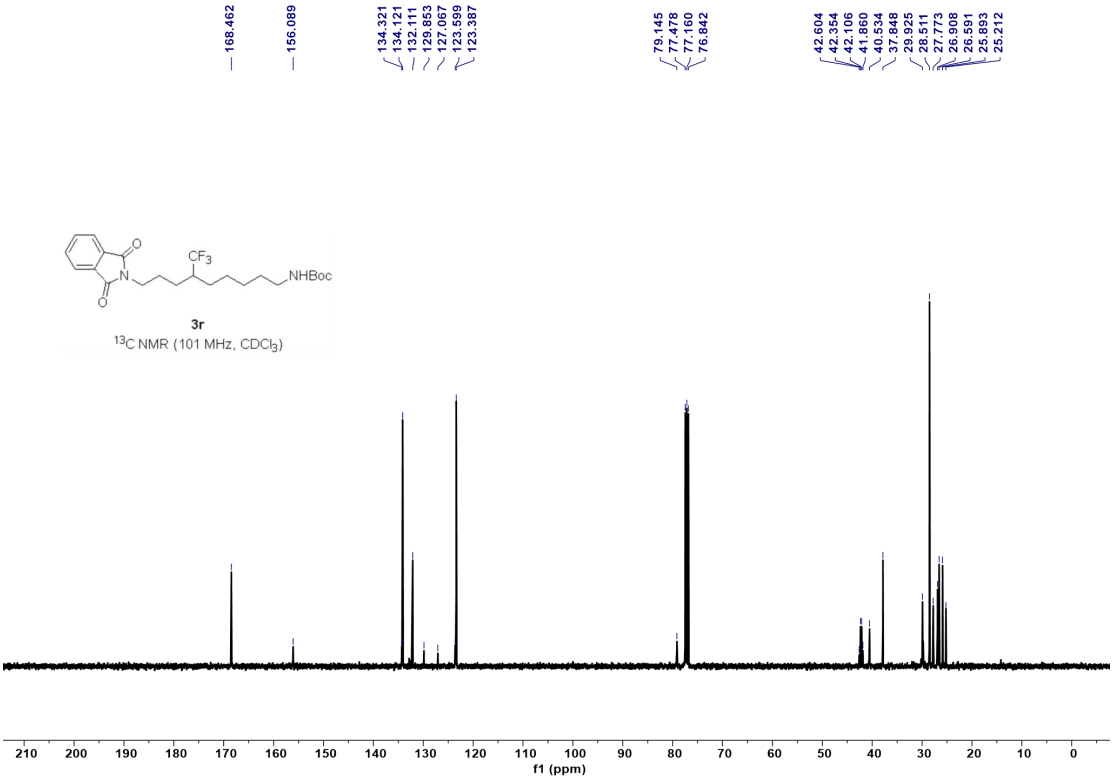

156.111
138.259
135.737
130.157
129.308
128.364
127.373
79.225
77.478
77.160
76.843
42.270
42.024
41.777
41.530
40.602
32.678
30.030
29.821
29.803
28.543
27.879
27.857
26.963
26.507
21.113
NHBoc
CF3
3p
13C NMR (101 MHz, CDCl3)
f1 (ppm)

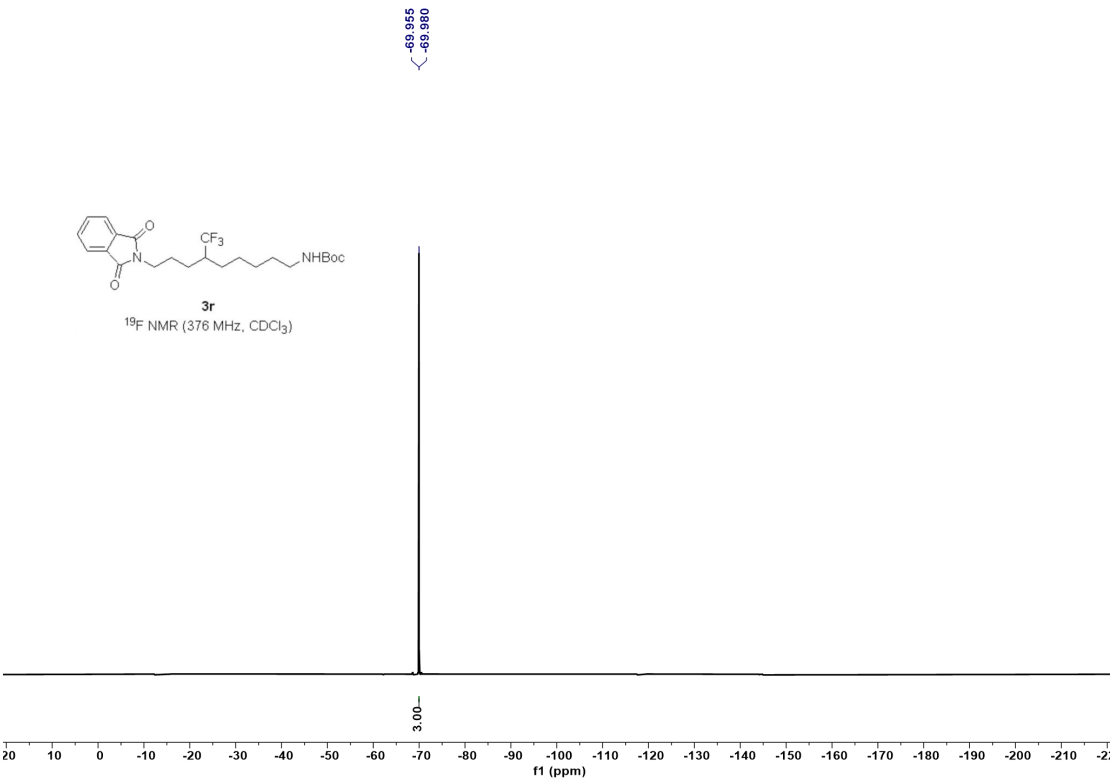

-69.851
-69.876
NHBoc
CF3
3p
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)

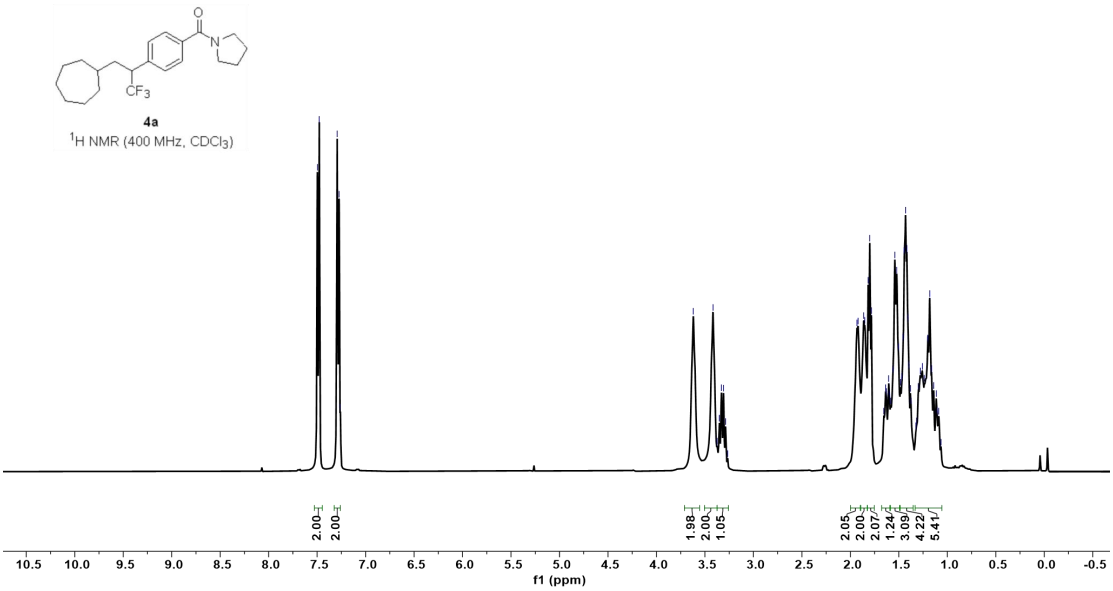

7.944
7.924
7.223
7.203
4.661
3.862
3.090
3.073
3.056
3.038
2.728
2.708
2.688
2.056
2.041
2.032
2.017
2.007
1.999
1.992
1.978
1.955
1.939
1.920
1.903
1.884
1.866
1.848
1.752
1.733
1.715
1.696
1.679
1.660
1.633
1.621
1.609
1.597
1.585
1.575
1.561
1.548
1.473
1.454
1.434
1.401
1.359
1.342
1.324
1.310
1.304
1.283
1.267
1.249
MeO
O
NHBoc
CF3
3q
1H NMR (400 MHz, CDCl3)
2.00
2.01
0.88
3.01
2.01
2.02
1.08
1.05
1.07
1.28
12.10
2.23
2.16
10.5
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
-0.5
f1 (ppm)
167.026
156.071
146.748
132.708
129.900
128.434
128.214
127.135
124.346
79.014
77.478
77.160
76.840
52.016
42.279
42.032
41.783
41.535
40.435
33.046
29.917
29.301
28.418
27.781
27.756
26.812
26.411
MeO
O
NHBoc
CF3
3q
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

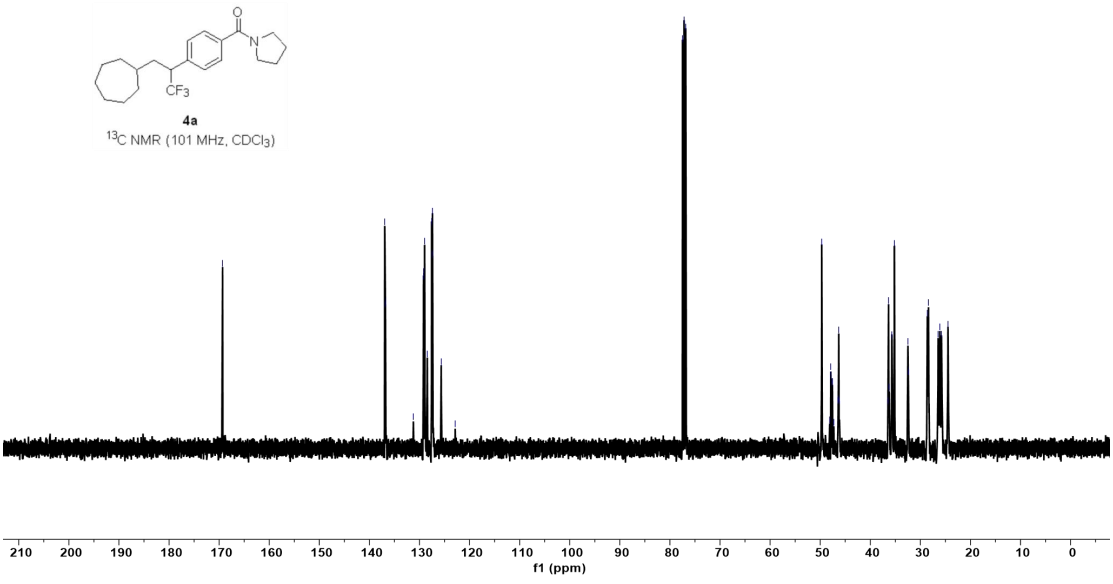

-69.854
MeO
O
NHBoc
CF3
3q
19F NMR (376 MHz, CDCl3)
3.00
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)
7.860
7.852
7.846
7.839
7.734
7.731
7.723
7.717
7.710
7.260
4.553
3.696
3.678
3.660
3.112
3.096
3.079
3.062
1.782
1.763
1.743
1.725
1.677
1.661
1.641
1.623
1.605
1.587
1.573
1.504
1.486
1.467
1.451
1.429
1.368
1.353
1.336
1.318
1.297

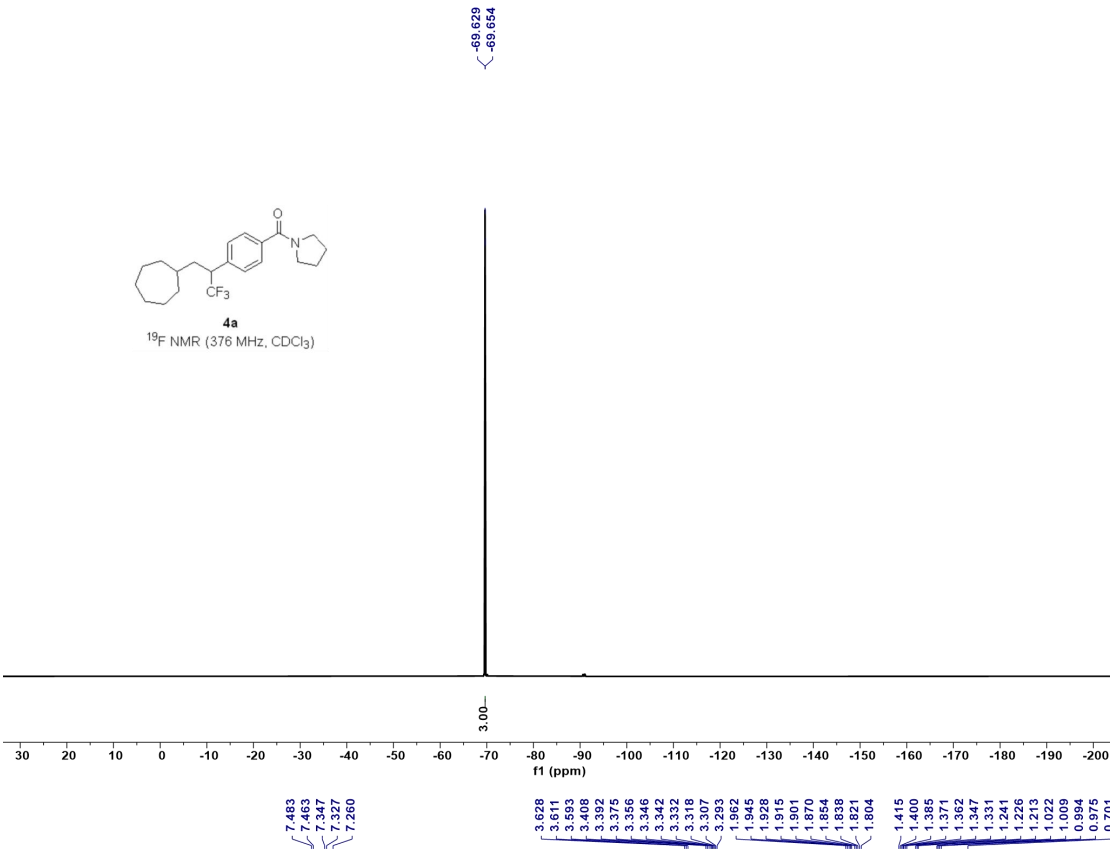

O
CF3
N
NHBoc
O
3r
1H NMR (400 MHz, CDCl3)
2.00
2.03
0.86
2.01
2.00
1.04
2.25
3.19
2.98
9.16
4.30
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

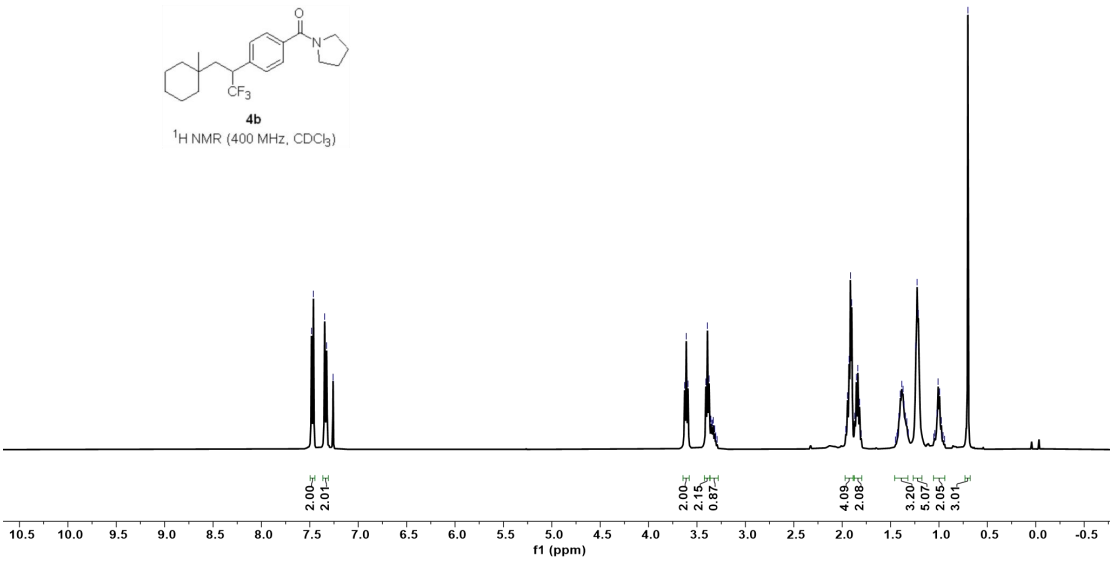

168.462
156.089
134.321
134.121
132.111
129.853
127.067
123.599
123.387
79.145
77.478
77.160
76.842
42.604
42.354
42.106
41.860
40.534
37.848
29.925
28.511
27.773
26.908
26.591
25.893
25.212
CF3
NHBoc
3r
13C NMR (101 MHz, CDCl3)
f1 (ppm)

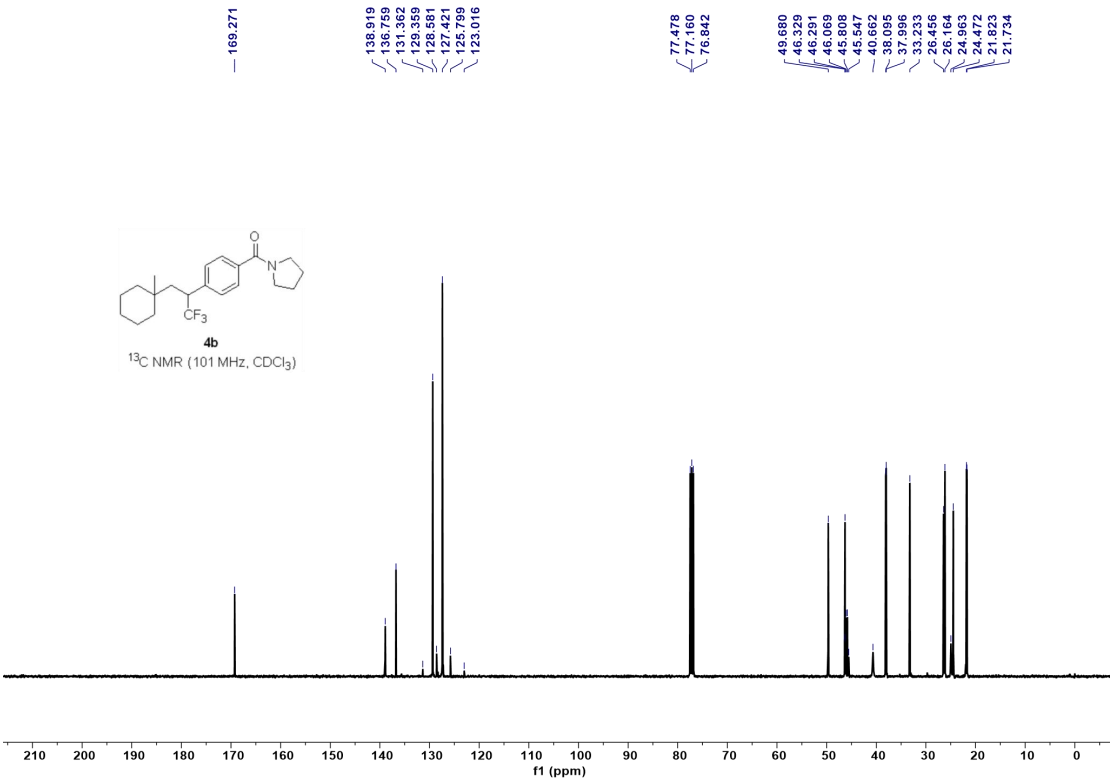

-69.955
-69.980
CF3
NHBoc
3r
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)

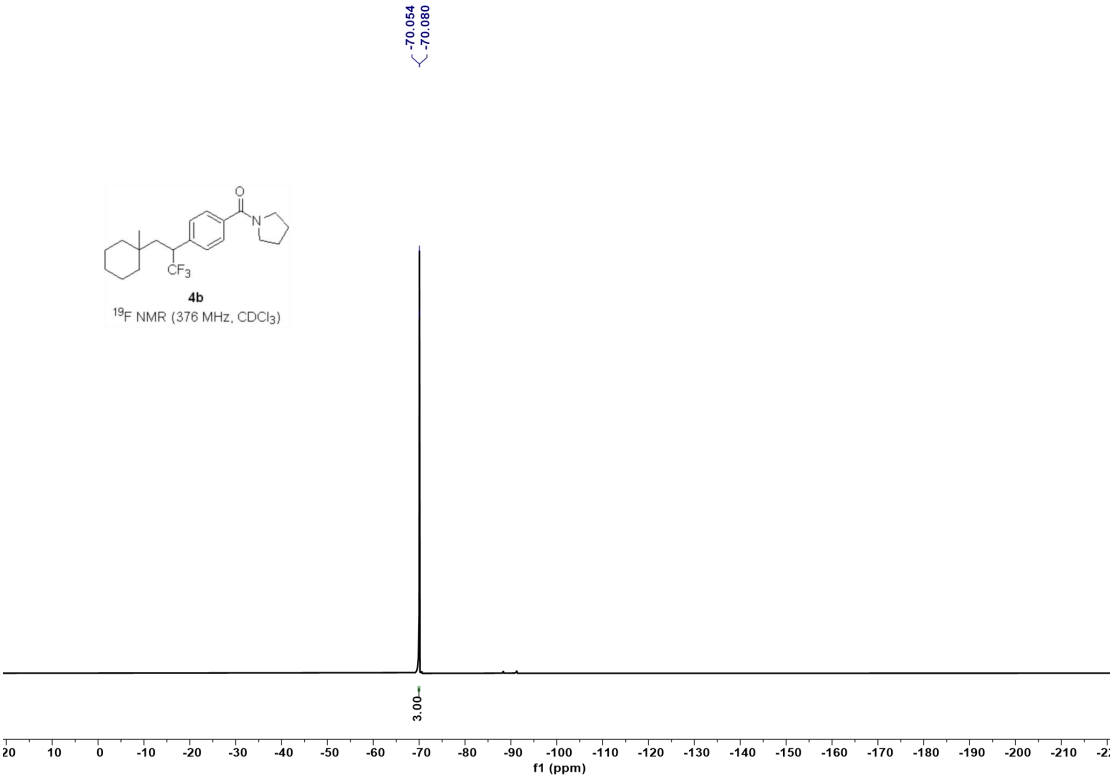

4a
1H NMR (400 MHz, CDCl3)
f1 (ppm)

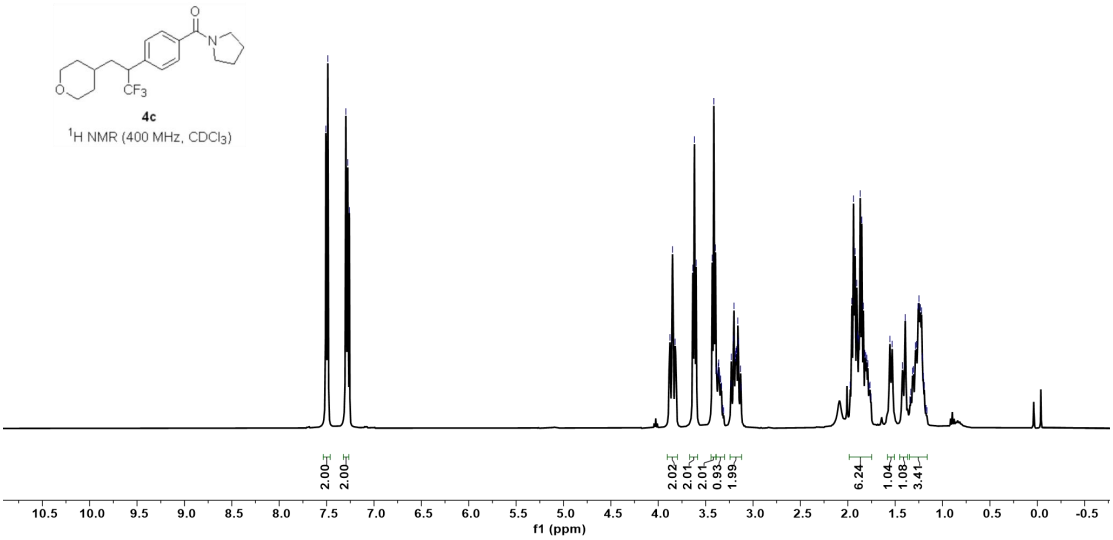

4a
13C NMR (101 MHz, CDCl3)
f1 (ppm)

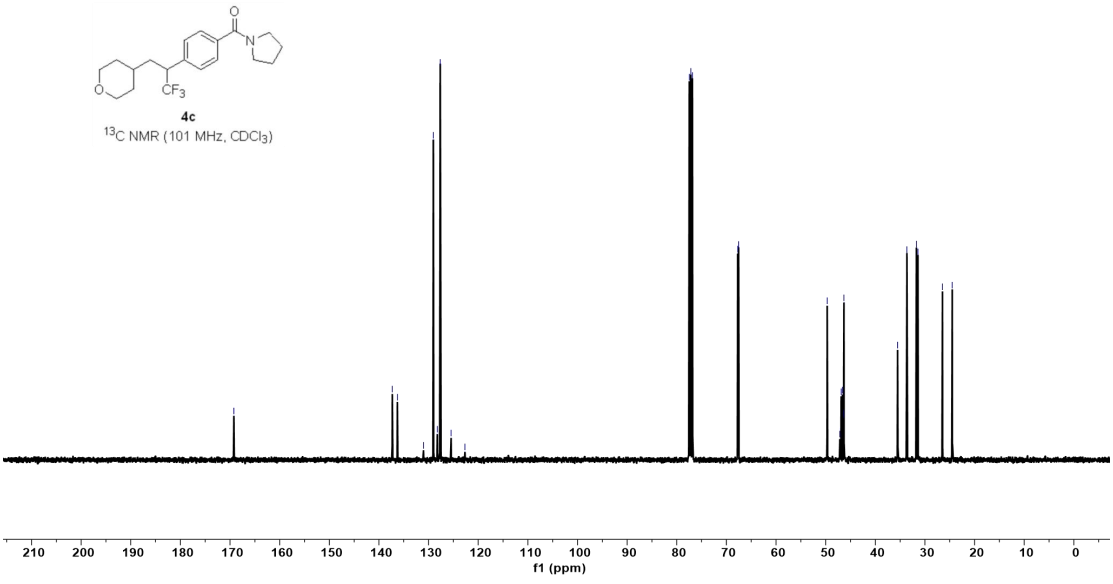

-69.629
-69.654
O
N
CF3
4a
19F NMR (376 MHz, CDCl3)
3.00
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)
7.483
7.463
7.347
7.327
7.260
3.628
3.611
3.593
3.408
3.392
3.375
3.356
3.346
3.342
3.332
3.318
3.307
3.293
1.962
1.945
1.928
1.915
1.901
1.870
1.854
1.838
1.821
1.804
1.415
1.400
1.385
1.371
1.362
1.347
1.331
1.241
1.226
1.213
1.022
1.009
0.994
0.975


O
N
CF3
4b
1H NMR (400 MHz, CDCl3)
2.00
2.01
2.00
2.15
0.87
4.09
2.08
3.20
5.07
2.05
3.01
10.5 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 -0.5
f1 (ppm)


169.271
138.919
136.759
131.362
129.359
128.581
127.421
125.799
123.016
77.478
77.160
76.842
49.680
46.329
46.291
46.069
45.808
45.547
40.662
38.095
37.996
33.233
26.456
26.164
24.963
24.472
21.823
21.734
O
N
CF3
4b
13C NMR (101 MHz, CDCl3)
f1 (ppm)

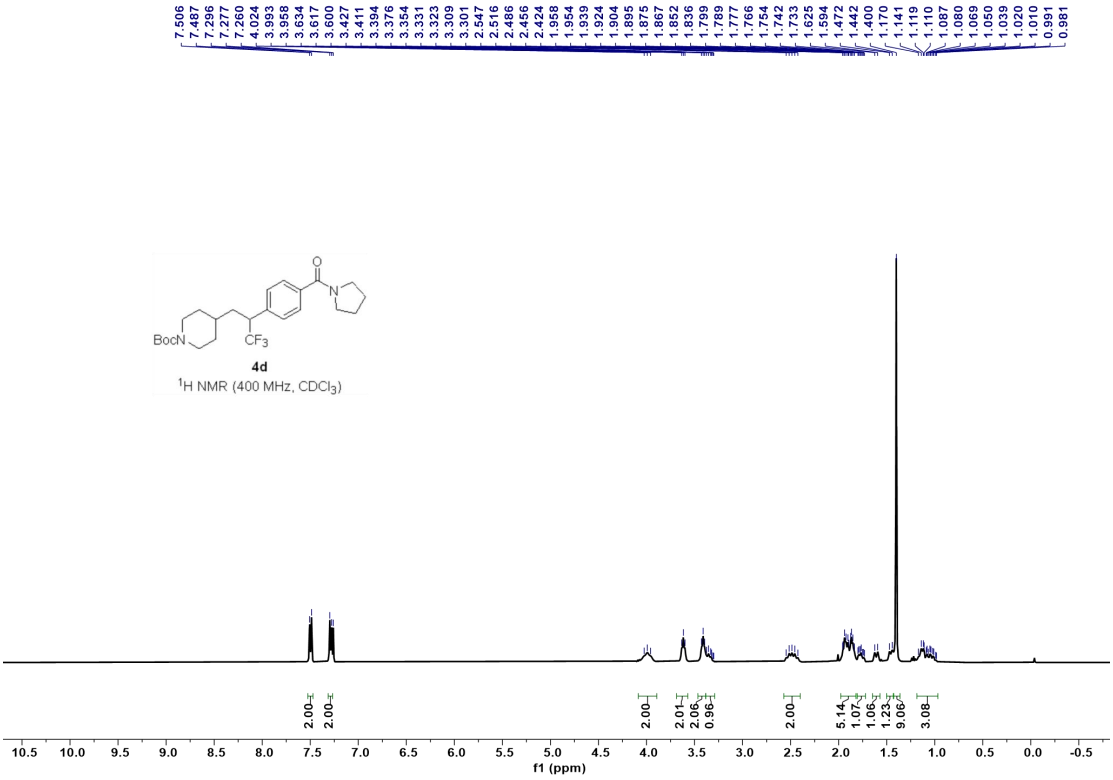

-70.054
-70.080
O
N
CF3
4b
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)


4c
1H NMR (400 MHz, CDCl3)
f1 (ppm)


4c
13C NMR (101 MHz, CDCl3)
f1 (ppm)

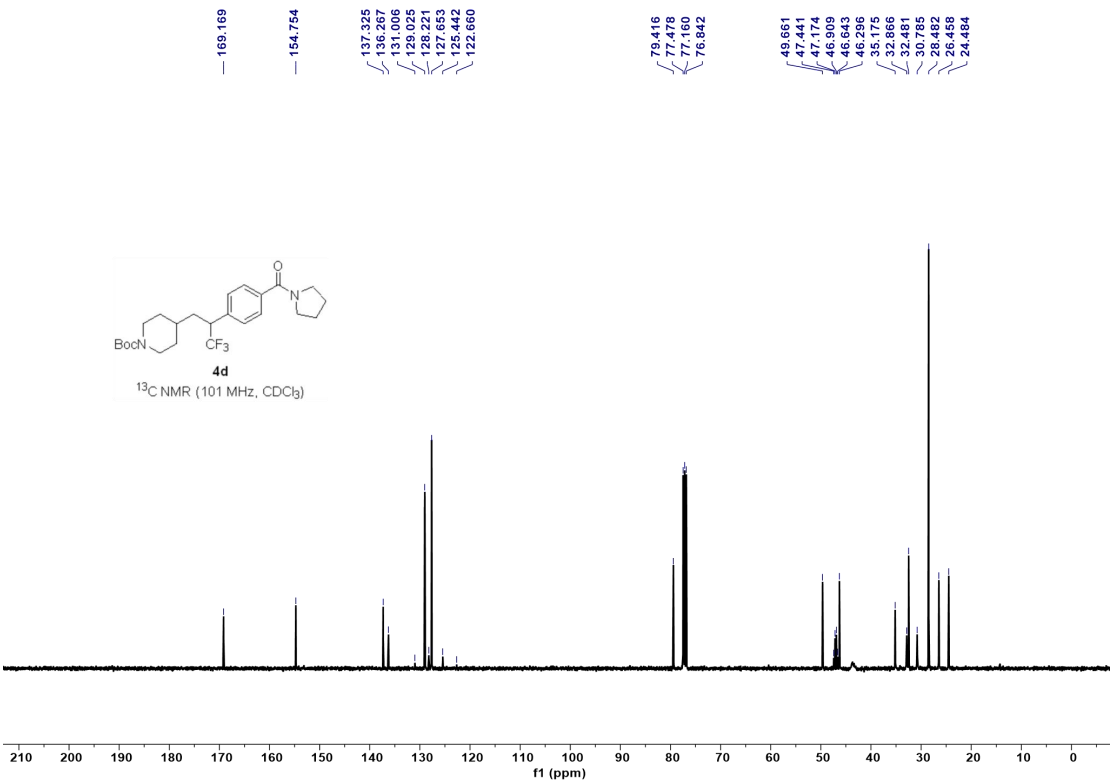
4c

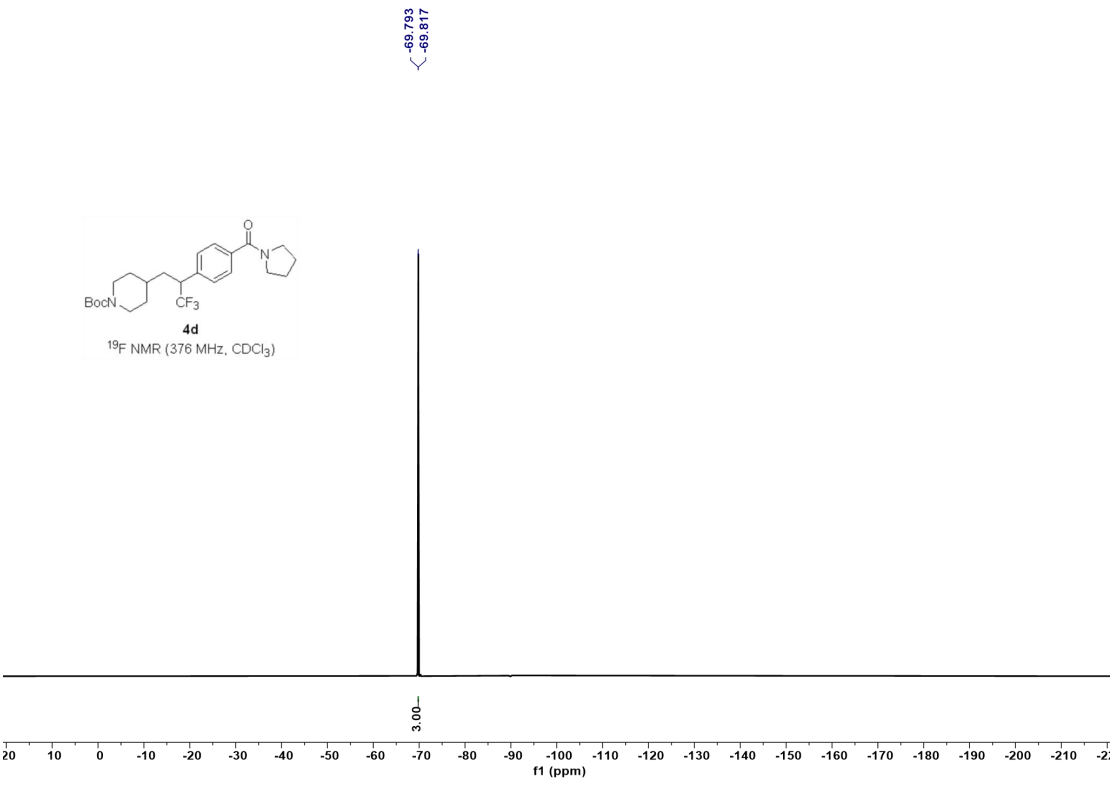
^{19}F NMR (376 MHz, $CDCl_3$)

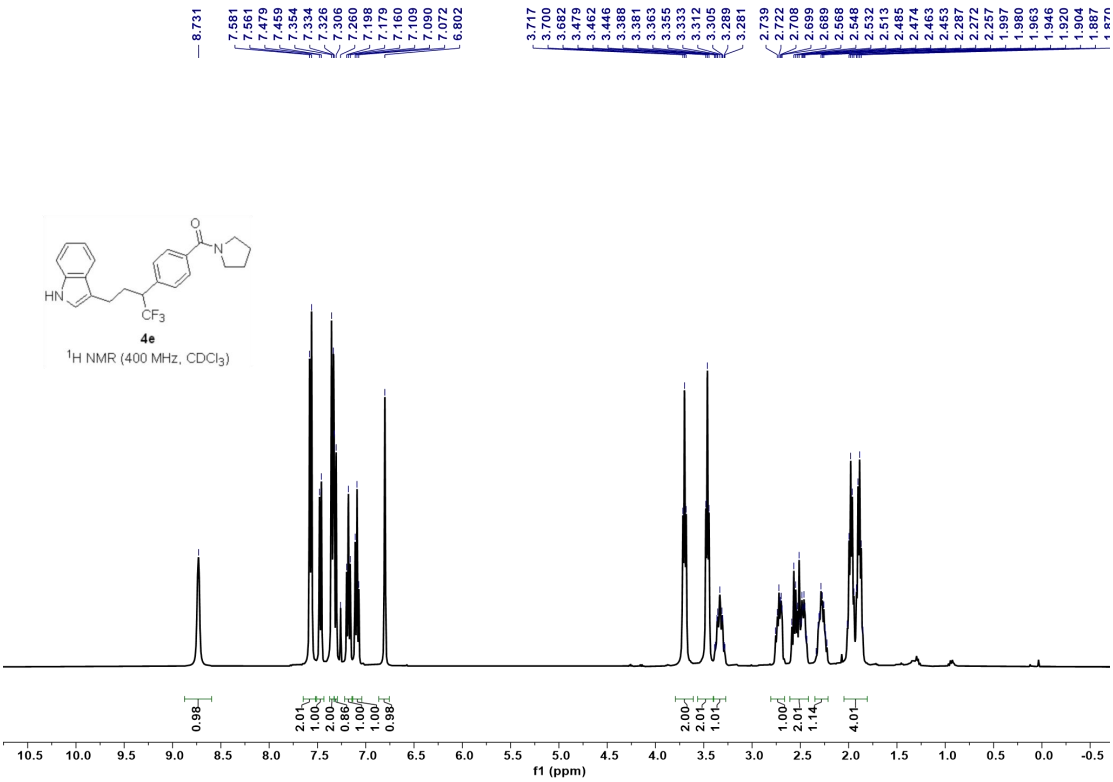


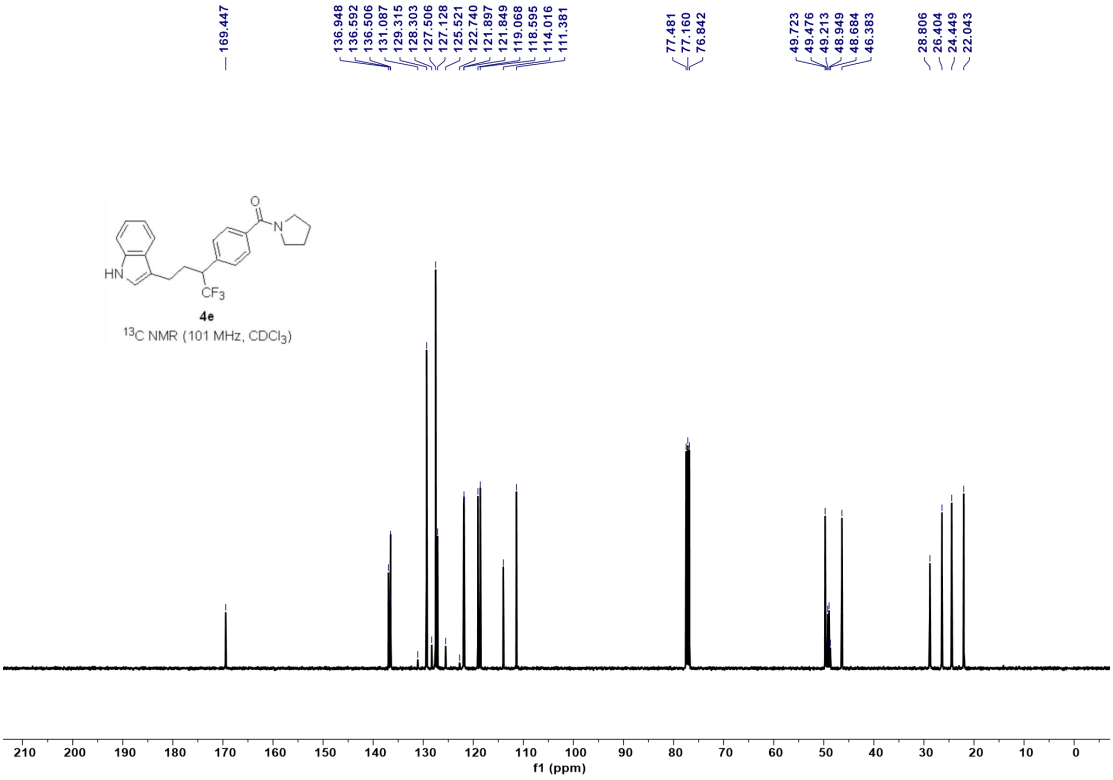
4d

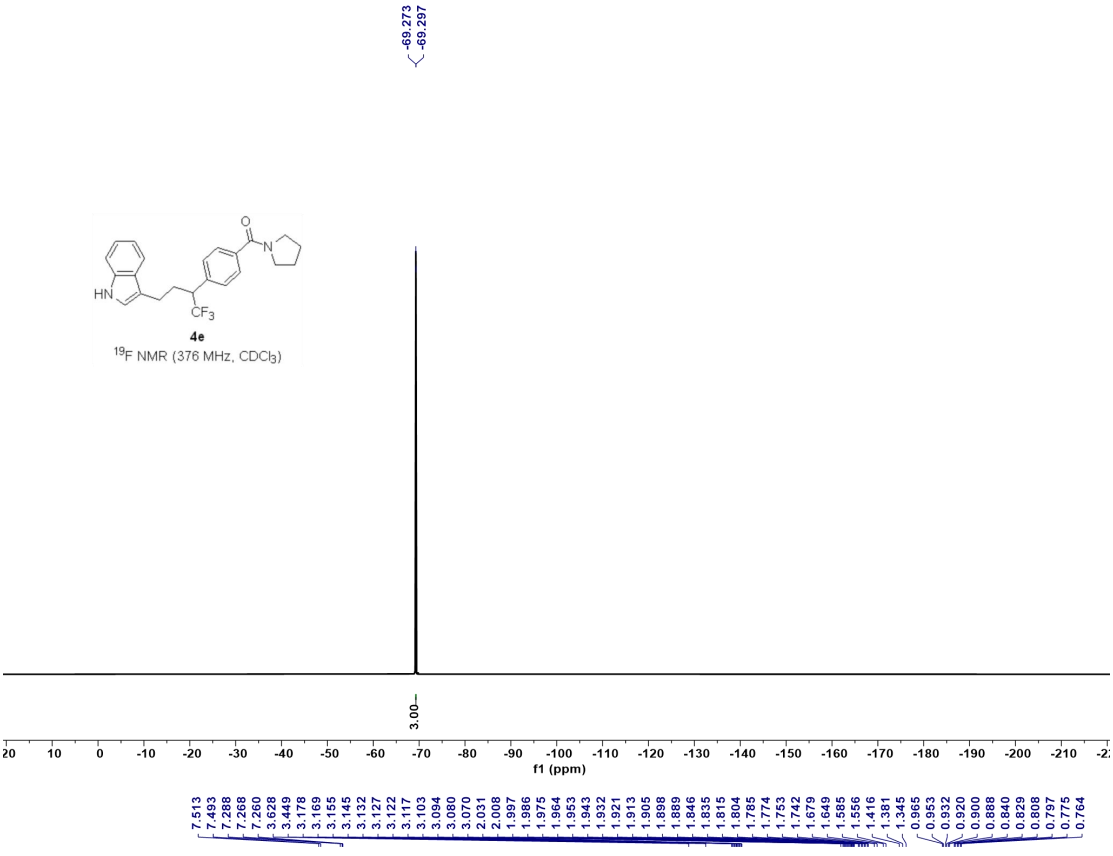
^{1}H NMR (400 MHz, $CDCl_3$)

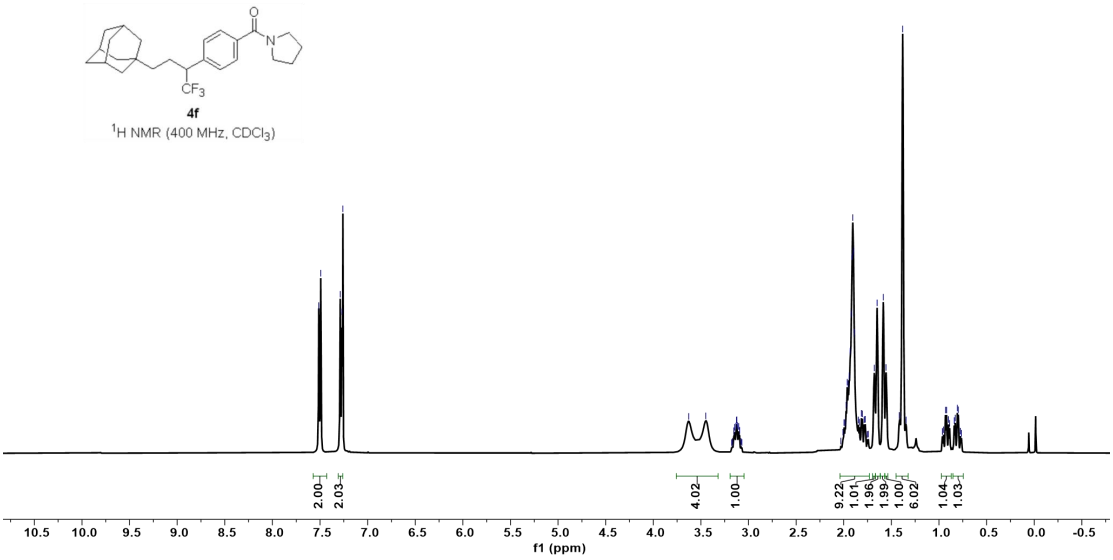

169.169
154.754
137.325
136.267
131.006
129.025
128.221
127.653
125.442
122.660
79.416
77.478
77.160
76.842
49.661
47.441
47.174
46.909
46.643
46.296
35.175
32.866
32.481
30.785
28.482
26.458
24.484
BocN
CF3
O
N
4d
13C NMR (101 MHz, CDCl3)
f1 (ppm)

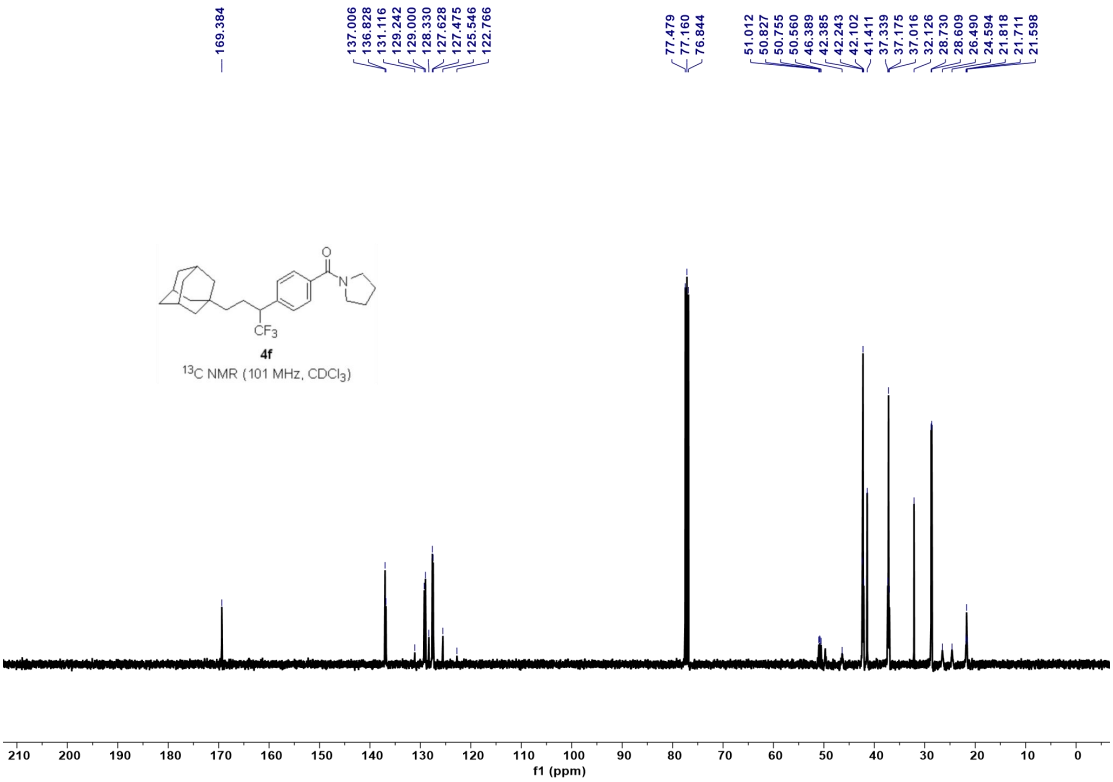

-69.793
-69.817
BocN
CF3
O
N
4d
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)

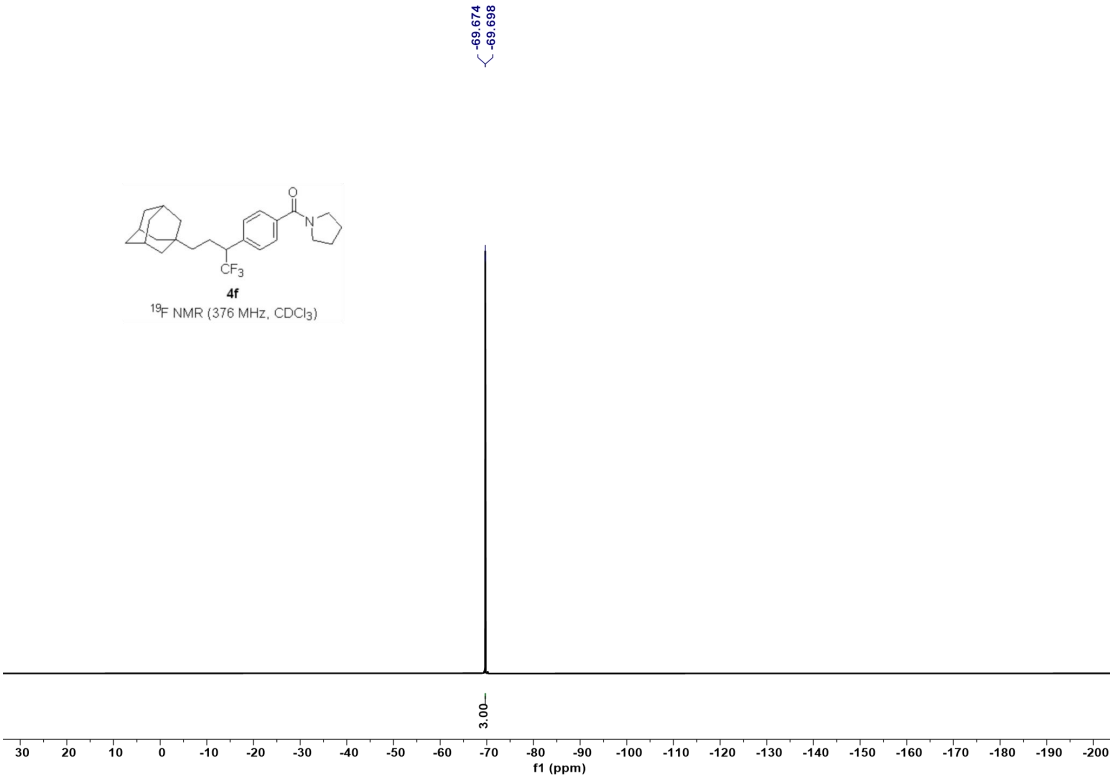

8.731
7.581
7.561
7.479
7.459
7.354
7.334
7.326
7.306
7.260
7.198
7.179
7.160
7.109
7.090
7.072
6.802
3.717
3.700
3.682
3.479
3.462
3.446
3.388
3.381
3.363
3.355
3.333
3.312
3.305
3.289
3.281
2.739
2.722
2.708
2.699
2.689
2.568
2.548
2.532
2.513
2.485
2.474
2.463
2.453
2.287
2.272
2.257
1.997
1.980
1.963
1.946
1.920
1.904
1.887
HN
O
N
CF3
4e
1H NMR (400 MHz, CDCl3)
0.98
2.01
1.00
2.00
0.86
1.00
1.00
0.98
2.00
2.01
1.01
1.00
2.01
1.14
4.01
10.5 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 -0.5
f1 (ppm)

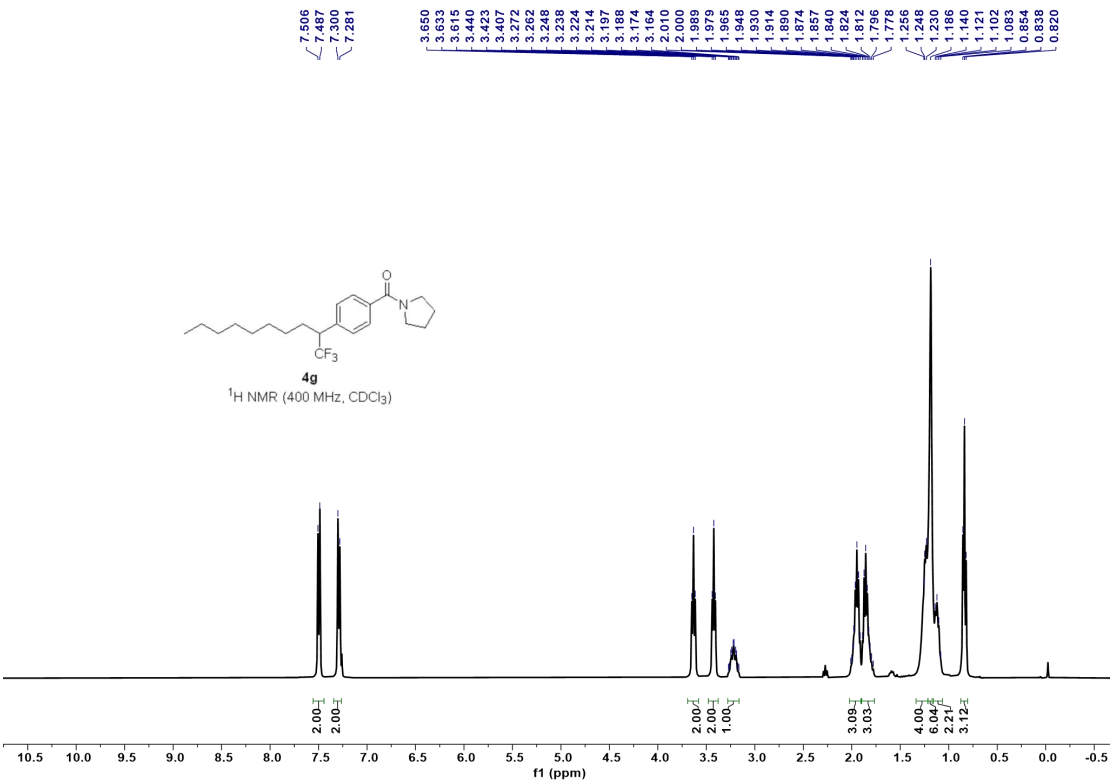

169.447
136.948
136.592
136.506
131.087
129.315
128.303
127.506
127.128
125.521
122.740
121.897
121.849
119.068
118.595
114.016
111.381
77.481
77.160
76.842
49.723
49.476
49.213
48.949
48.684
46.383
28.806
26.404
24.449
22.043
HN
O
N
CF3
4e
13C NMR (101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

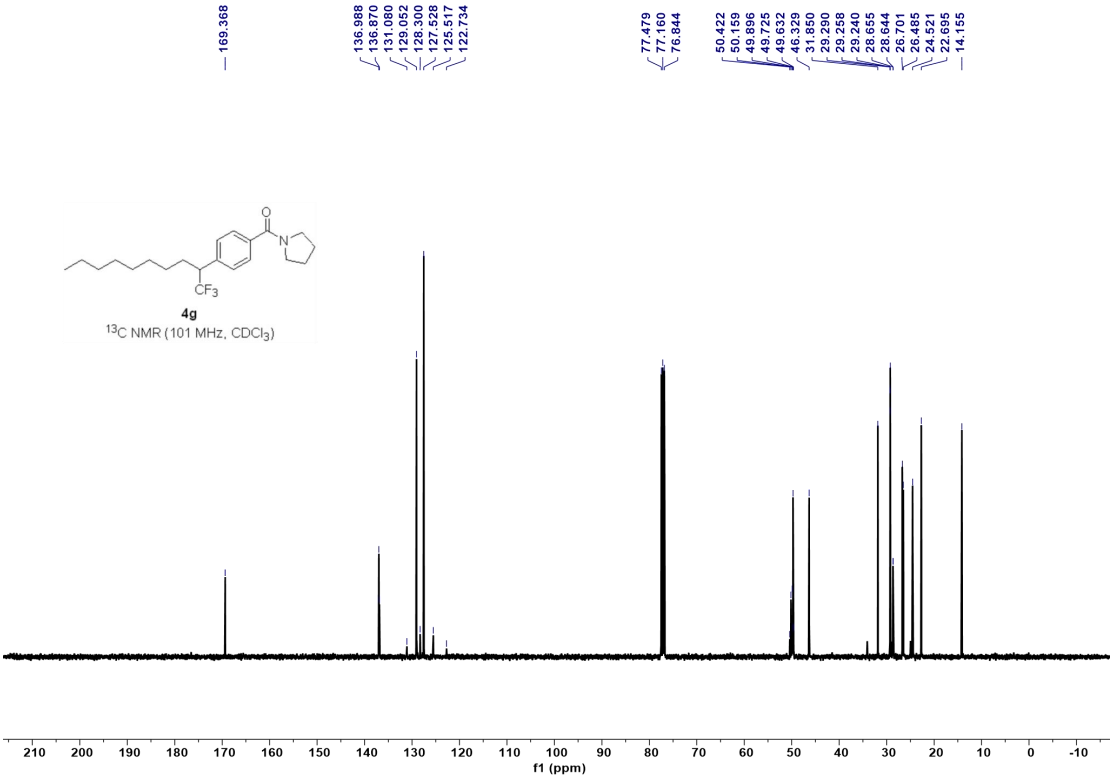

-69.273
-69.297
HN
O
N
CF3
4e
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)

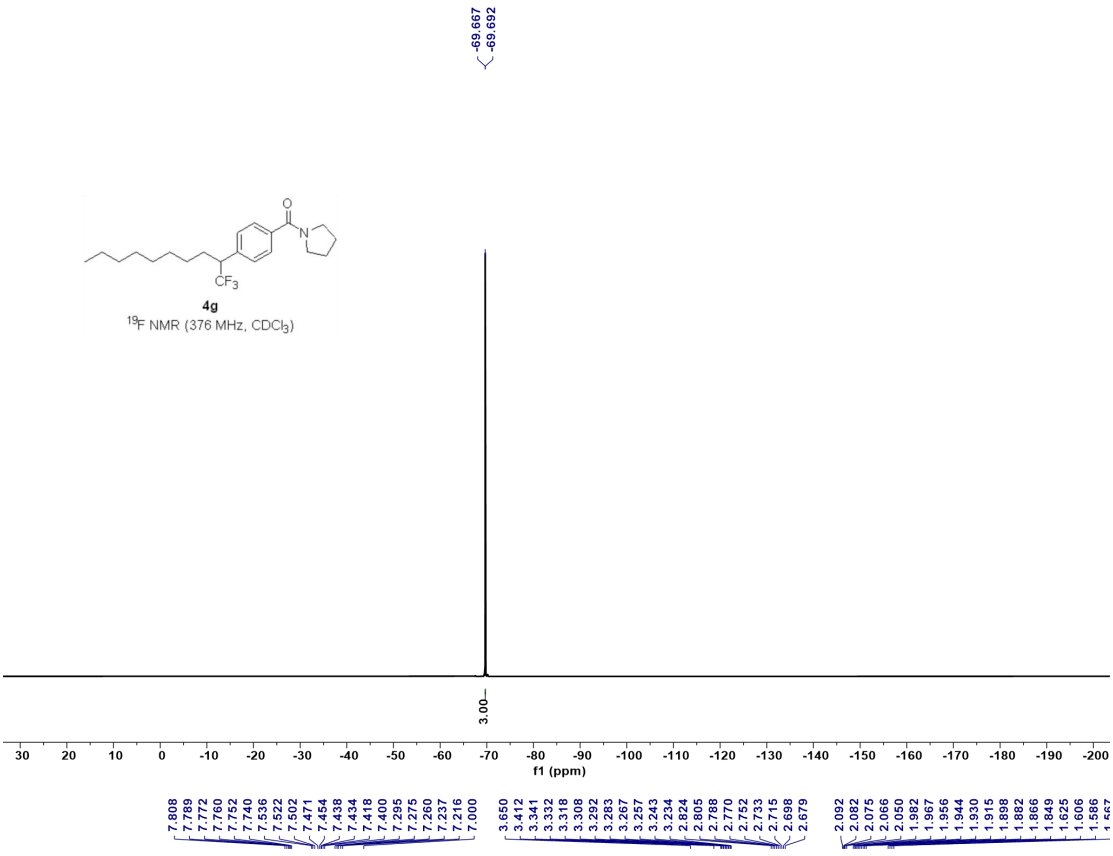

O
N
CF3
4f
1H NMR (400 MHz, CDCl3)
2.00
2.03
4.02
1.00
9.22
1.01
1.96
1.99
1.00
6.02
1.04
1.03
f1 (ppm)

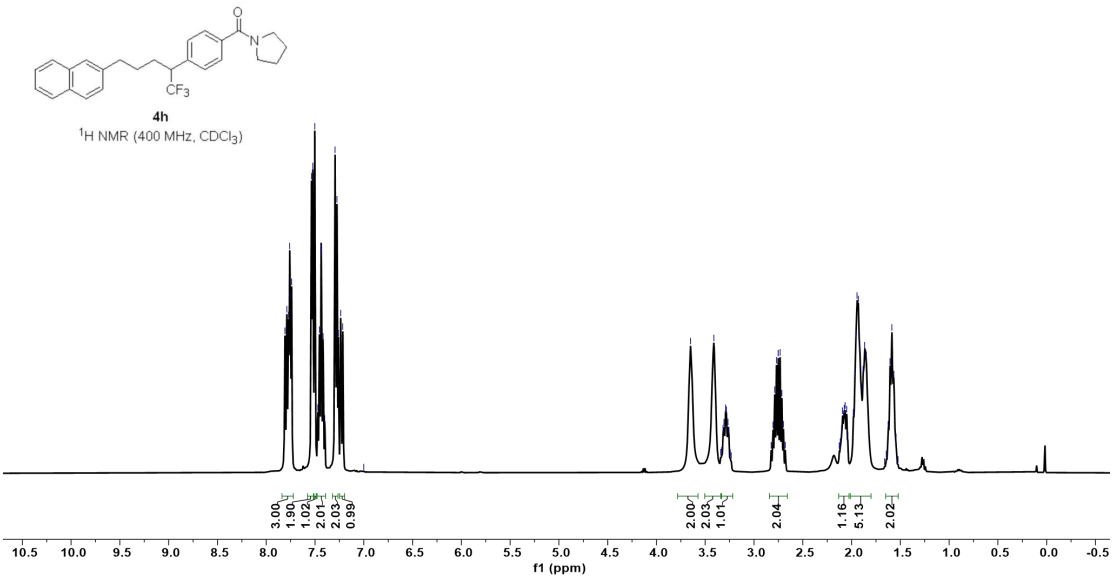

169.384
137.006
136.828
131.116
129.242
129.000
128.330
127.628
127.475
125.546
122.766
77.479
77.160
76.844
51.012
50.827
50.755
50.560
46.389
42.385
42.243
42.102
41.411
37.339
37.175
37.016
32.126
28.730
28.609
26.490
24.594
21.818
21.711
21.598
O
N
CF3
4f
13C NMR (101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

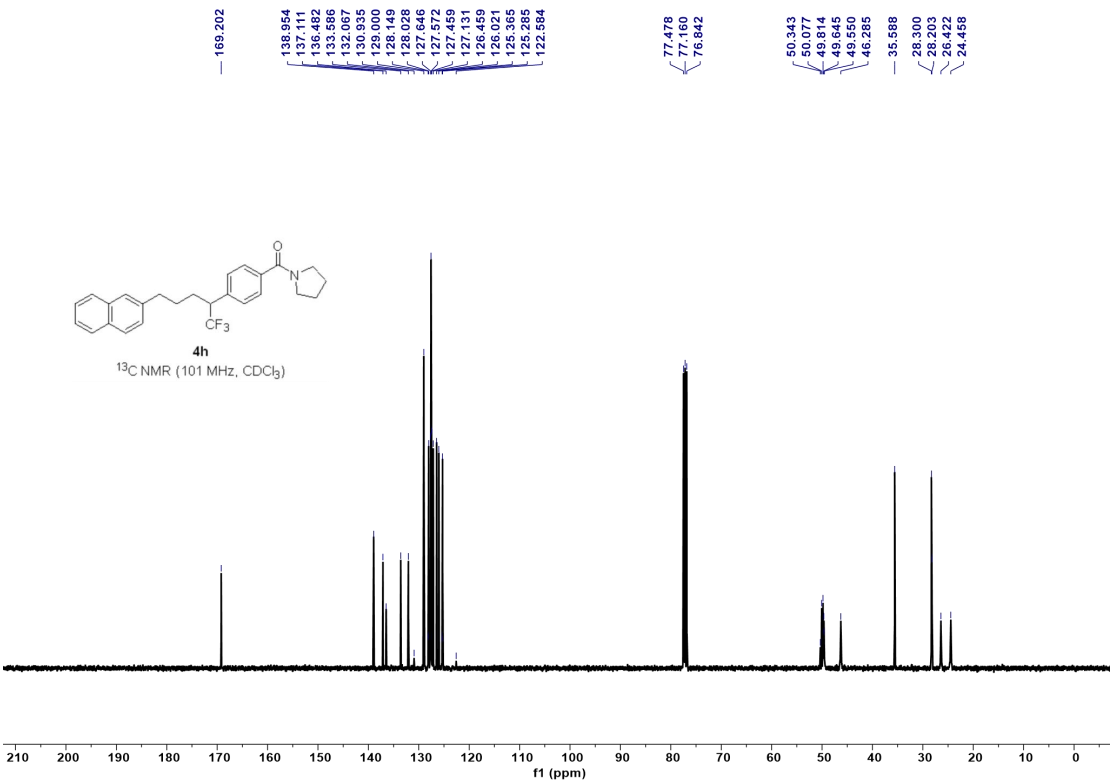

-69.674
-69.698
O
N
CF3
4f
19F NMR (376 MHz, CDCl3)
3.00
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)

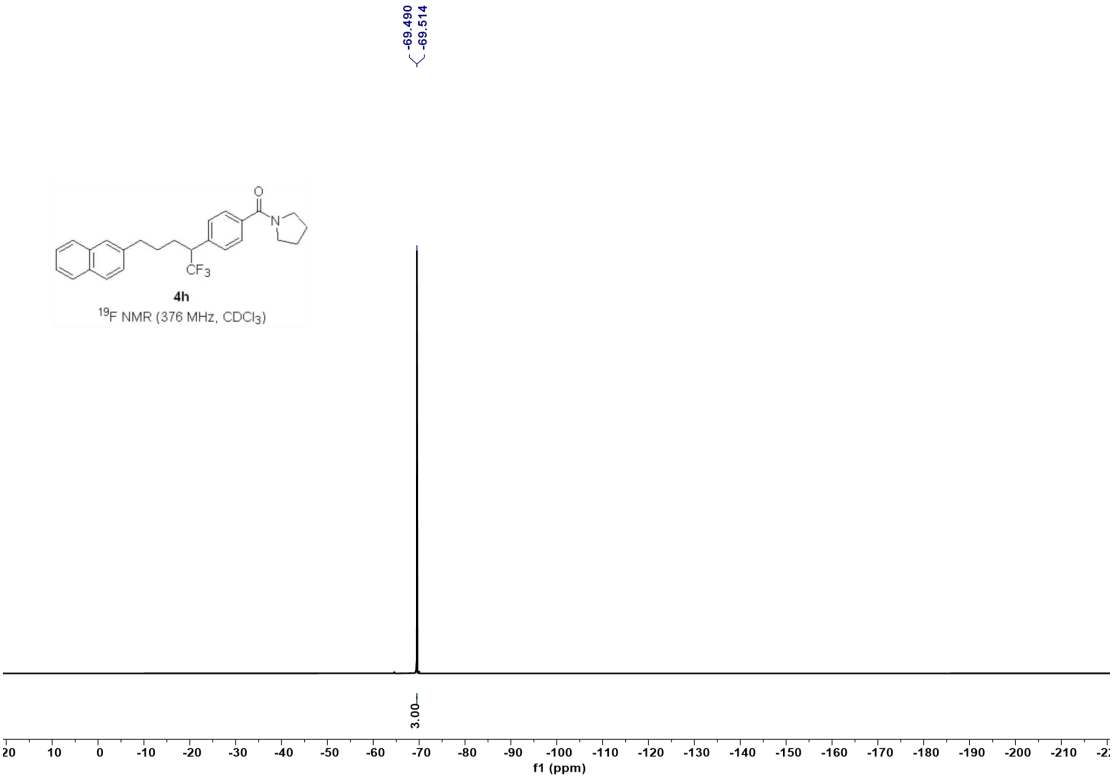

7.506
7.487
7.300
7.281
3.650
3.633
3.615
3.440
3.423
3.407
3.272
3.262
3.248
3.238
3.224
3.214
3.197
3.188
3.174
3.164
2.010
2.000
1.989
1.979
1.965
1.948
1.930
1.914
1.890
1.874
1.857
1.840
1.824
1.812
1.796
1.778
1.256
1.248
1.230
1.186
1.140
1.121
1.102
1.083
0.854
0.838
0.820
4g
1H NMR (400 MHz, CDCl3)
2.00
2.00
2.00
2.00
1.00
3.09
3.03
4.00
6.04
2.21
3.12
f1 (ppm)

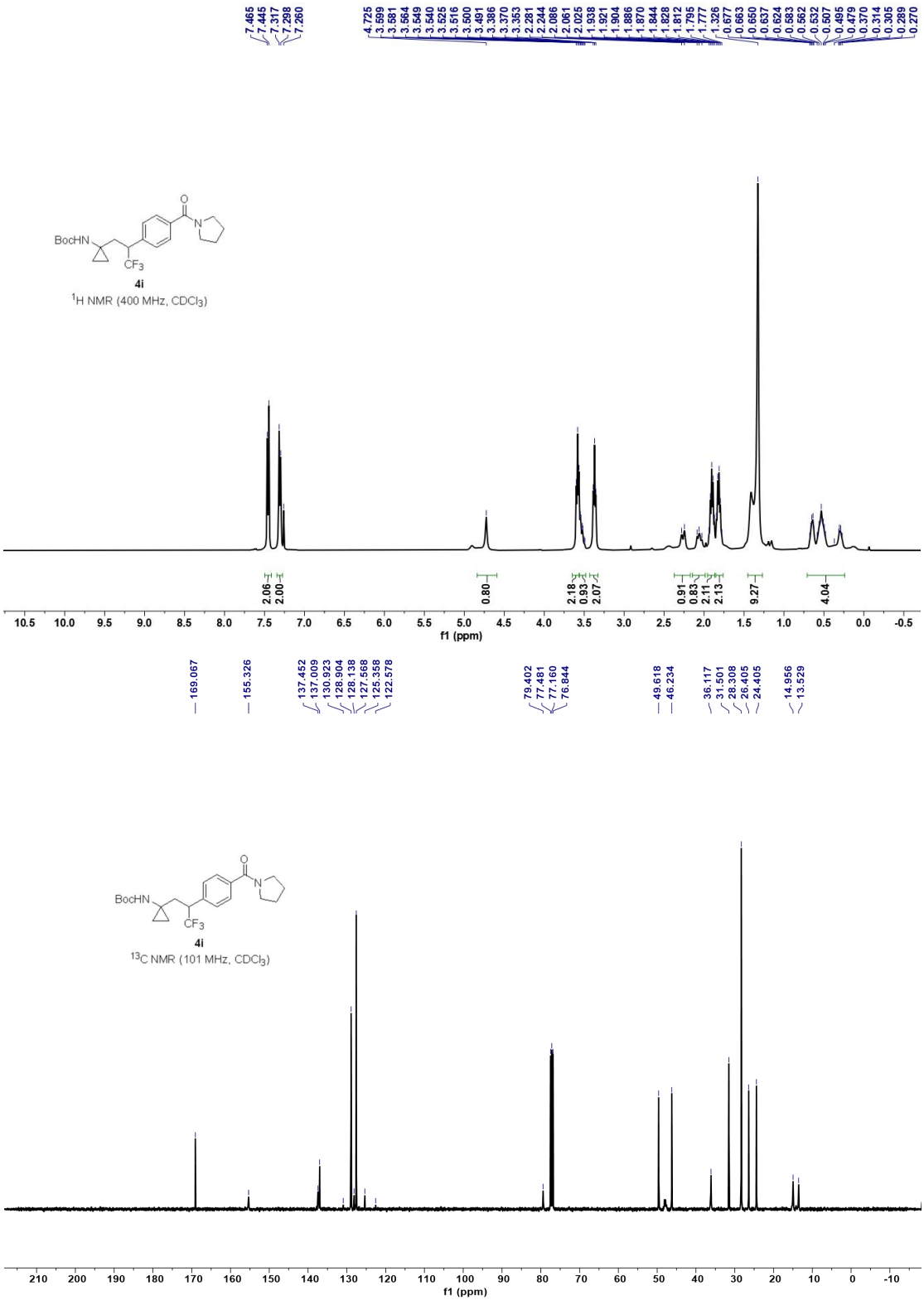

169.368
136.988
136.870
131.080
129.052
128.300
127.528
125.517
122.734
77.479
77.160
76.844
50.422
50.159
49.896
49.725
49.632
46.329
31.850
29.290
29.258
29.240
28.655
28.644
26.701
26.485
24.521
22.695
14.155
4g
13C NMR (101 MHz, CDCl3)
f1 (ppm)

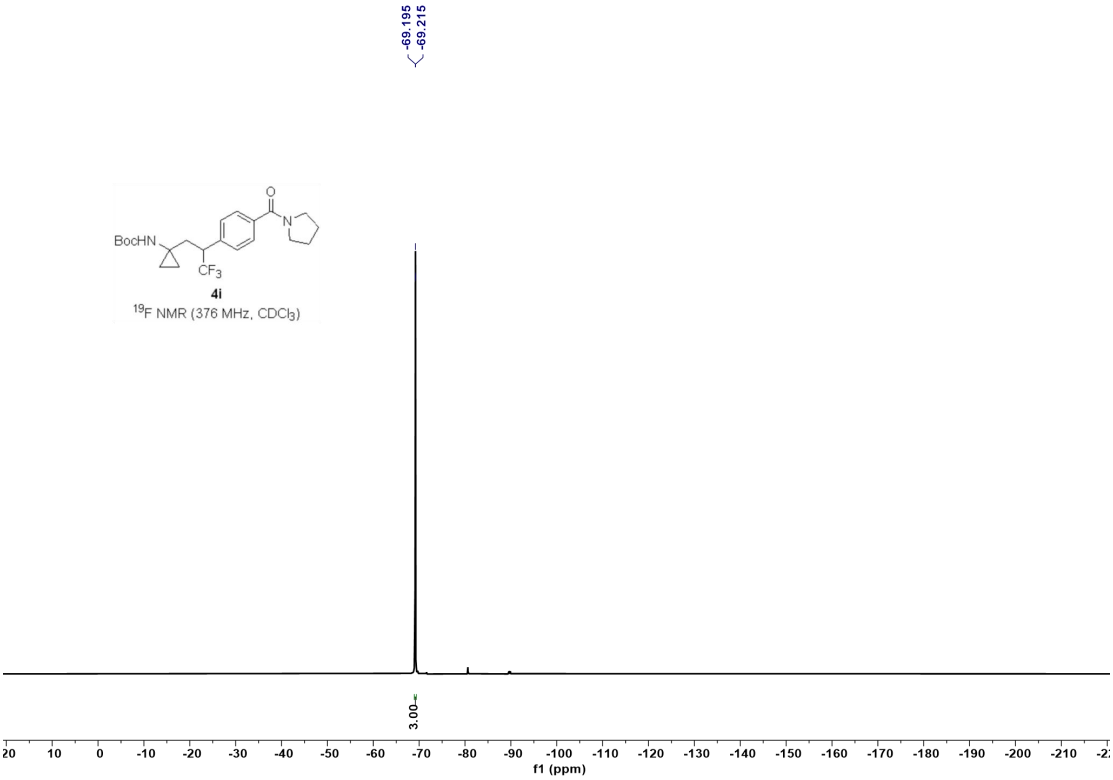

-69.667
-69.692
4g
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)


4h
1H NMR (400 MHz, CDCl3)
3.00
1.90
1.02
2.01
2.03
0.99
2.00
2.03
1.01
2.04
1.16
5.13
2.02
f1 (ppm)


169.202
138.954
137.111
136.482
133.586
132.067
130.935
129.000
128.149
128.028
127.646
127.572
127.459
127.131
126.459
126.021
125.365
125.285
122.584
77.478
77.160
76.842
50.343
50.077
49.814
49.645
49.550
46.285
35.588
28.300
28.203
26.422
24.458
O
N
CF3
4h
13C NMR (101 MHz, CDCl3)
f1 (ppm)

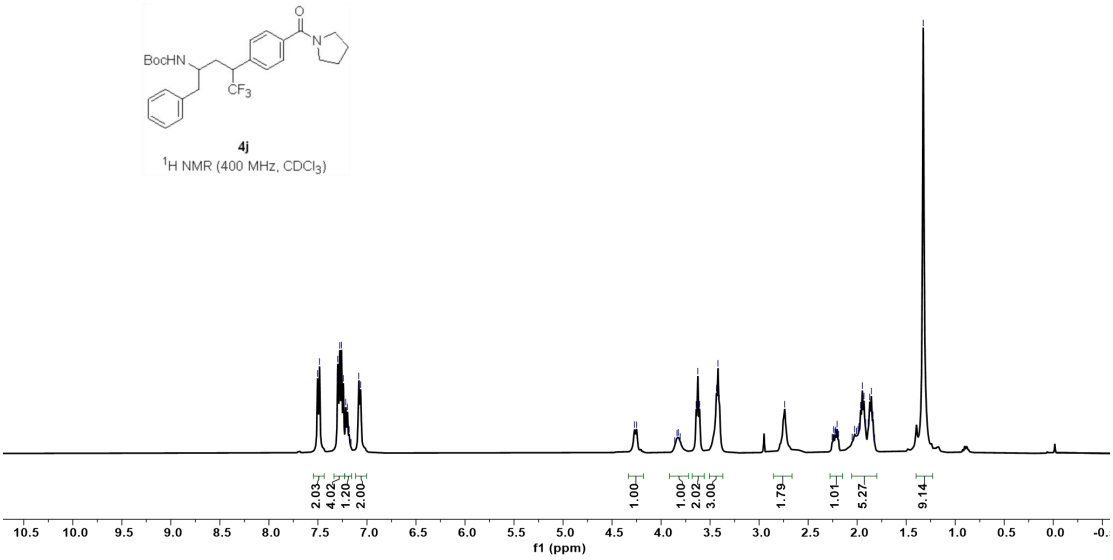

-69.490
-69.514
O
N
CF3
4h
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)


7.465
7.445
7.317
7.298
7.260
4.725
3.599
3.581
3.564
3.549
3.540
3.525
3.516
3.500
3.491
3.386
3.370
3.353
2.281
2.244
2.086
2.061
2.025
1.938
1.921
1.904
1.886
1.870
1.844
1.828
1.812
1.795
1.777
1.326
0.677
0.663
0.650
0.637
0.624
0.583
0.562
0.532
0.507
0.495
0.479
0.370
0.314
0.305
0.289
0.270
BocHN
CF3
O
N
4i
1H NMR (400 MHz, CDCl3)
2.06
2.00
0.80
2.18
0.93
2.07
0.91
0.83
2.11
2.13
9.27
4.04
10.5 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 -0.5
f1 (ppm)
169.067
155.326
137.452
137.009
130.923
128.904
128.138
127.568
125.358
122.578
79.402
77.481
77.160
76.844
49.618
46.234
36.117
31.501
28.308
26.405
24.405
14.956
13.529
BocHN
CF3
O
N
4i
13C NMR (101 MHz, CDCl3)
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)



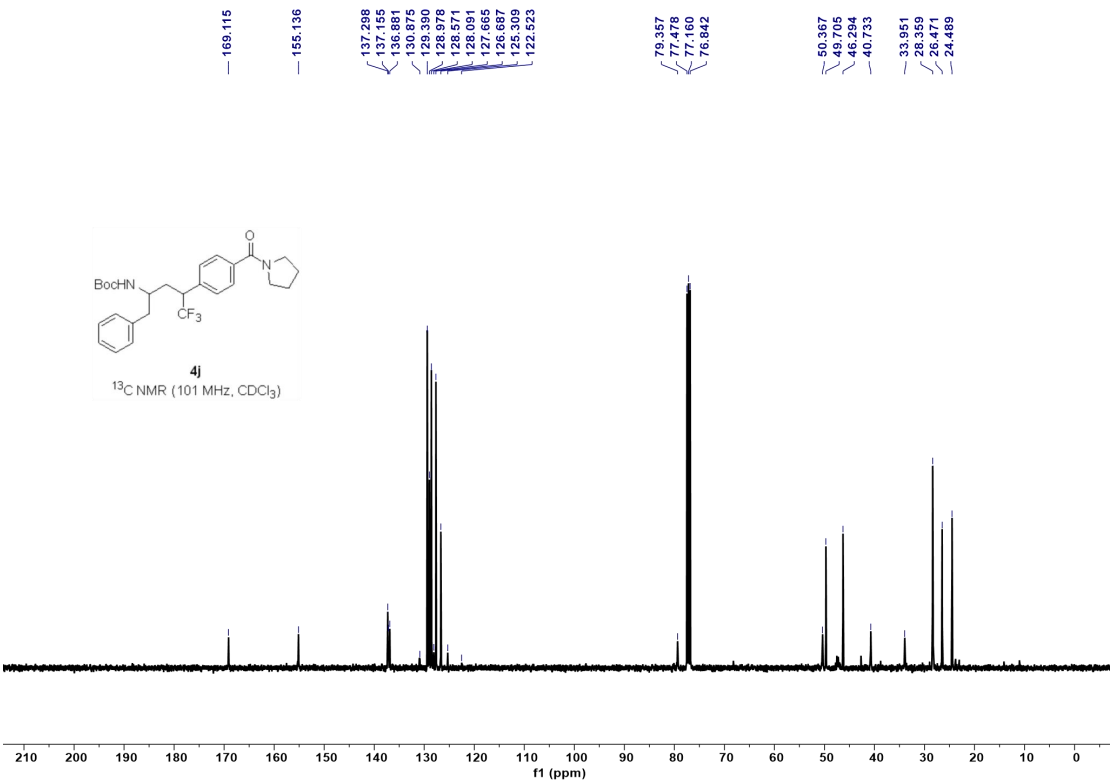
4i

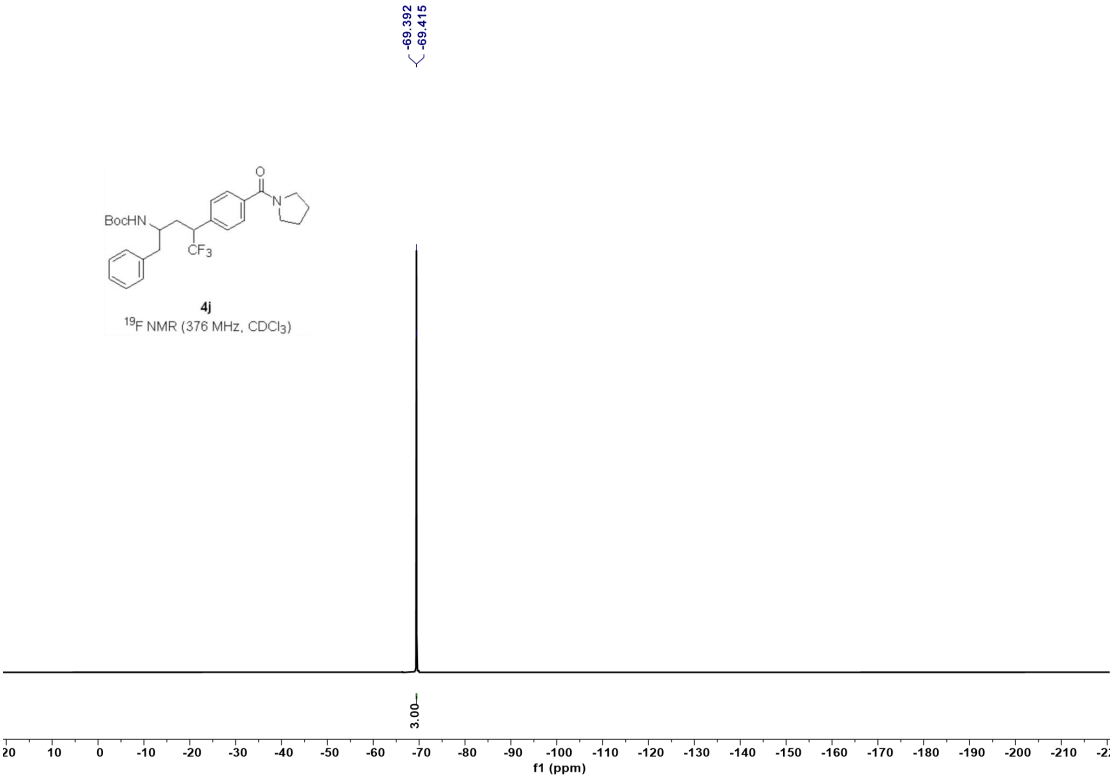
^{19}F NMR (376 MHz, $CDCl_3$)

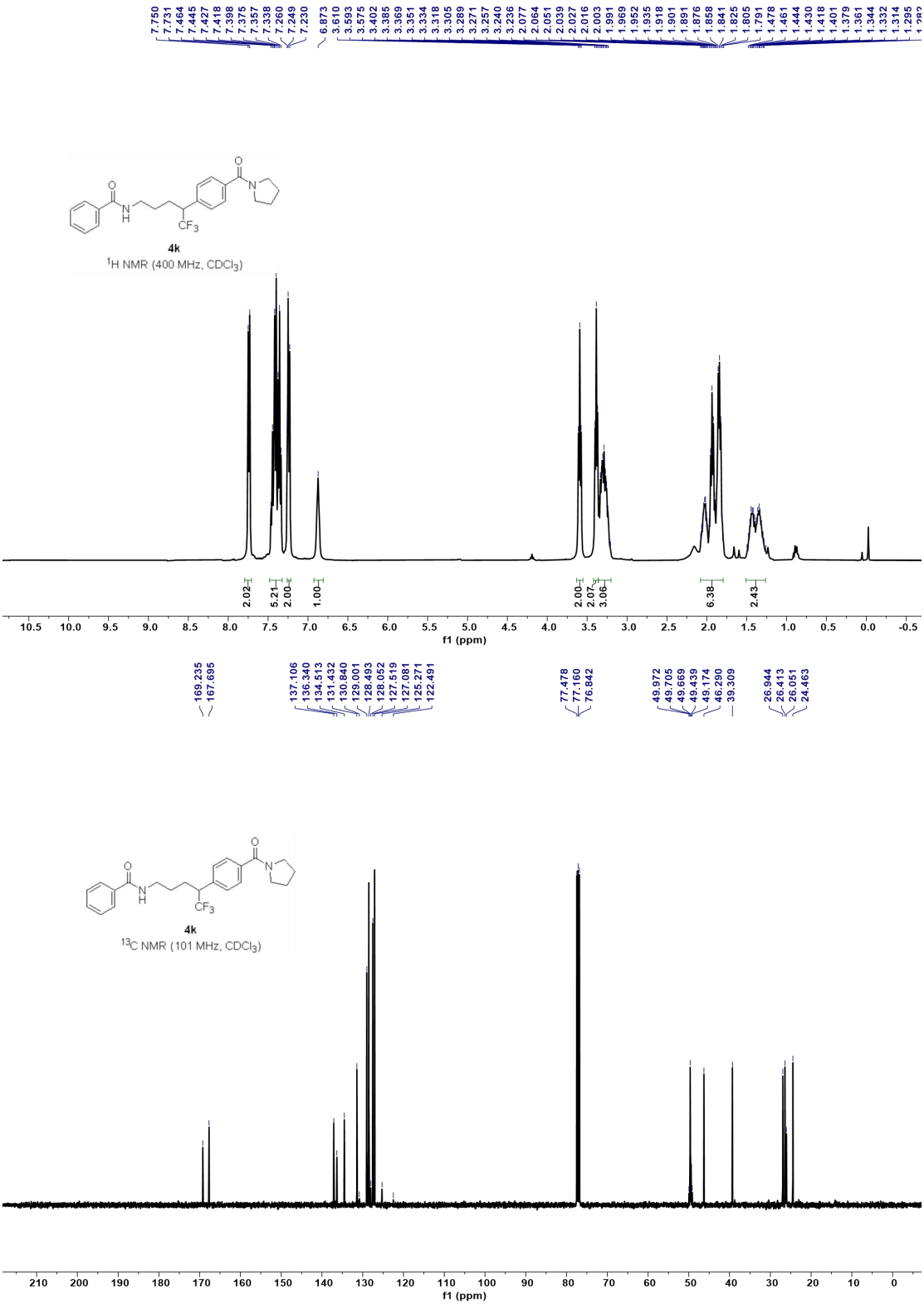


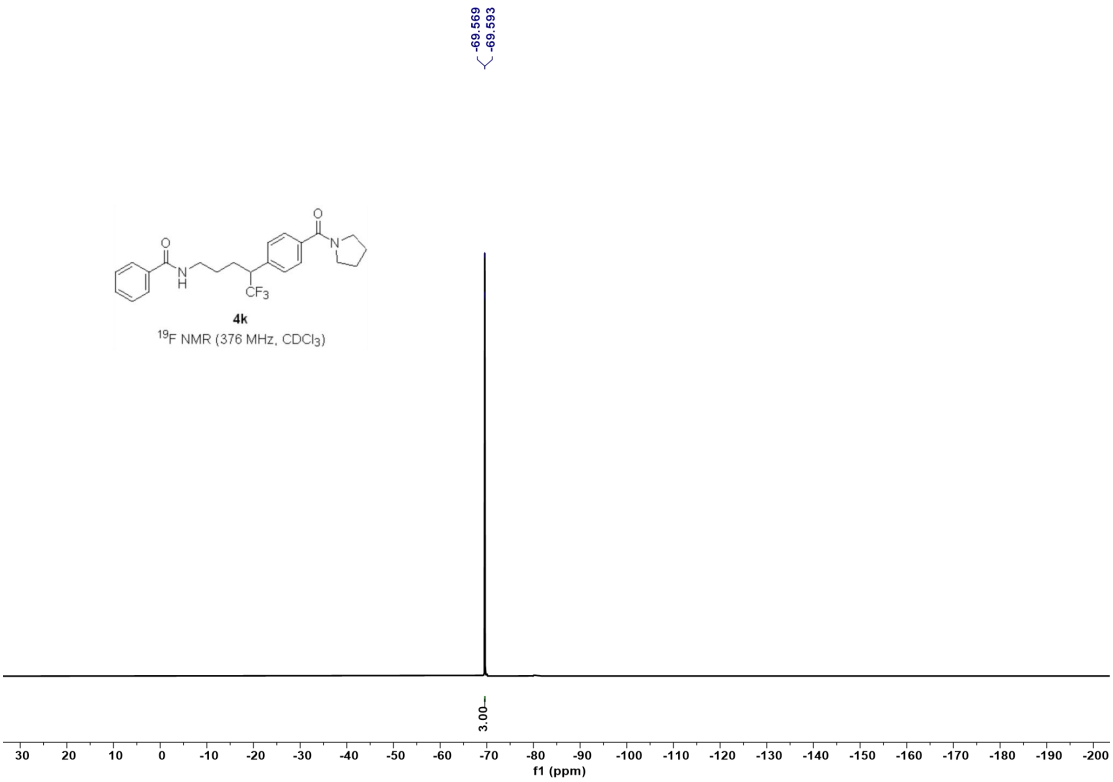
4j

^{1}H NMR (400 MHz, $CDCl_3$)


169.115
155.136
137.298
137.155
136.881
130.875
129.390
128.978
128.571
128.091
127.665
126.687
125.309
122.523
79.357
77.478
77.160
76.842
50.367
49.705
46.294
40.733
33.951
28.359
26.471
24.489
BocHN
CF3
O
N
4j
13C NMR (101 MHz, CDCl3)
f1 (ppm)

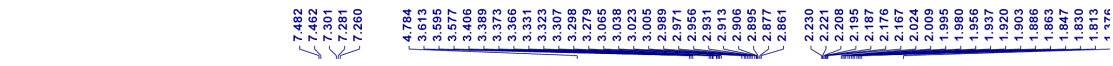

-69.392
-69.415
BocHN
CF3
O
N
4j
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)

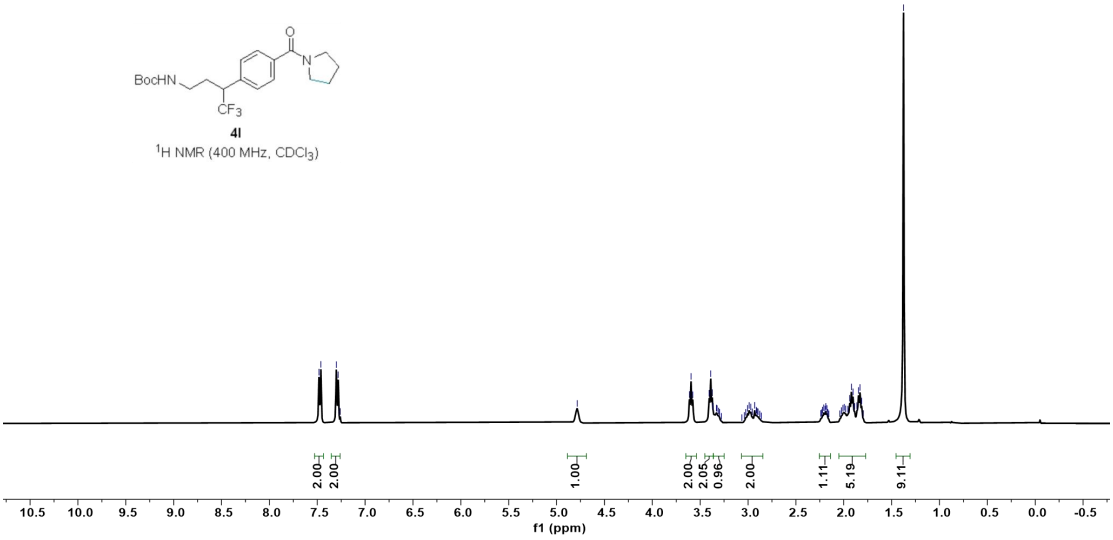

4k
1H NMR (400 MHz, CDCl3)
4k
13C NMR (101 MHz, CDCl3)
f1 (ppm)



4k

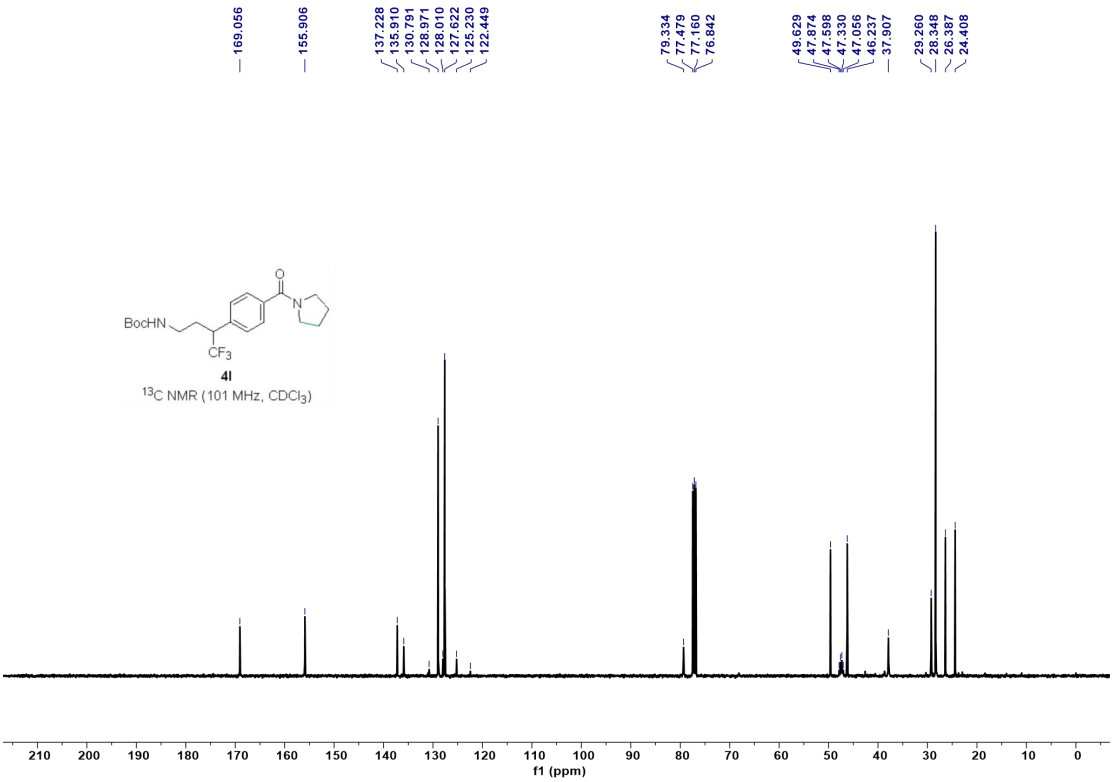
^{19}F NMR (376 MHz, $CDCl_3$)

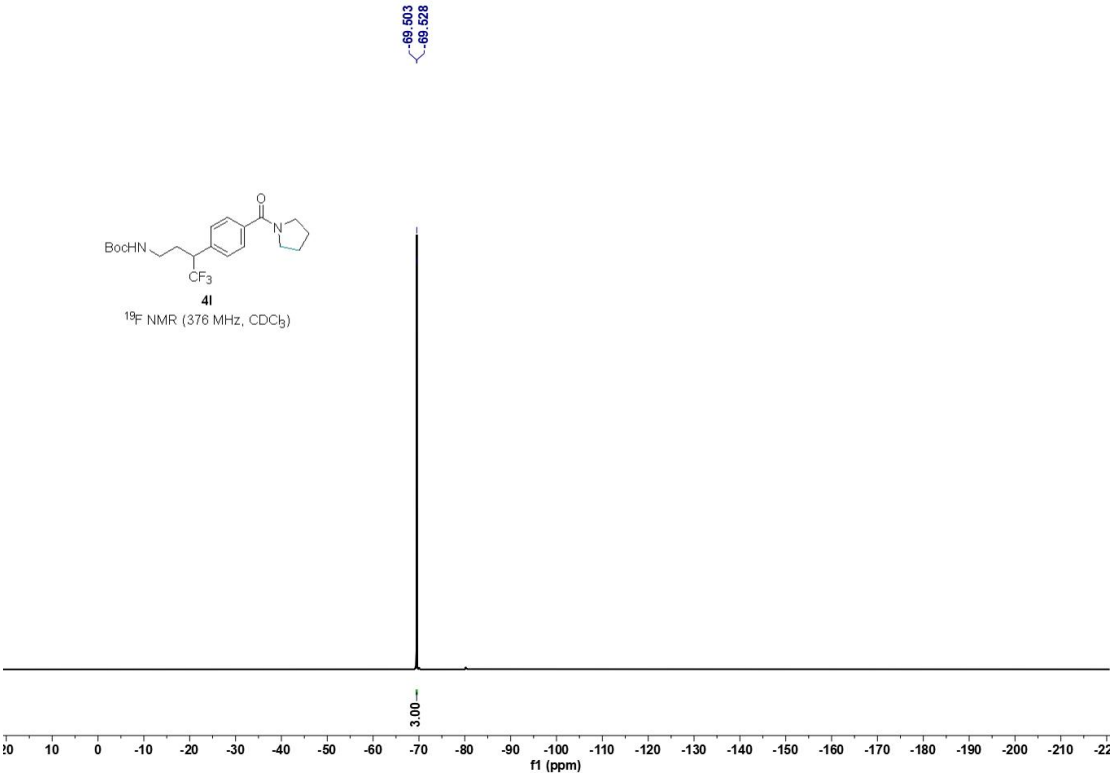


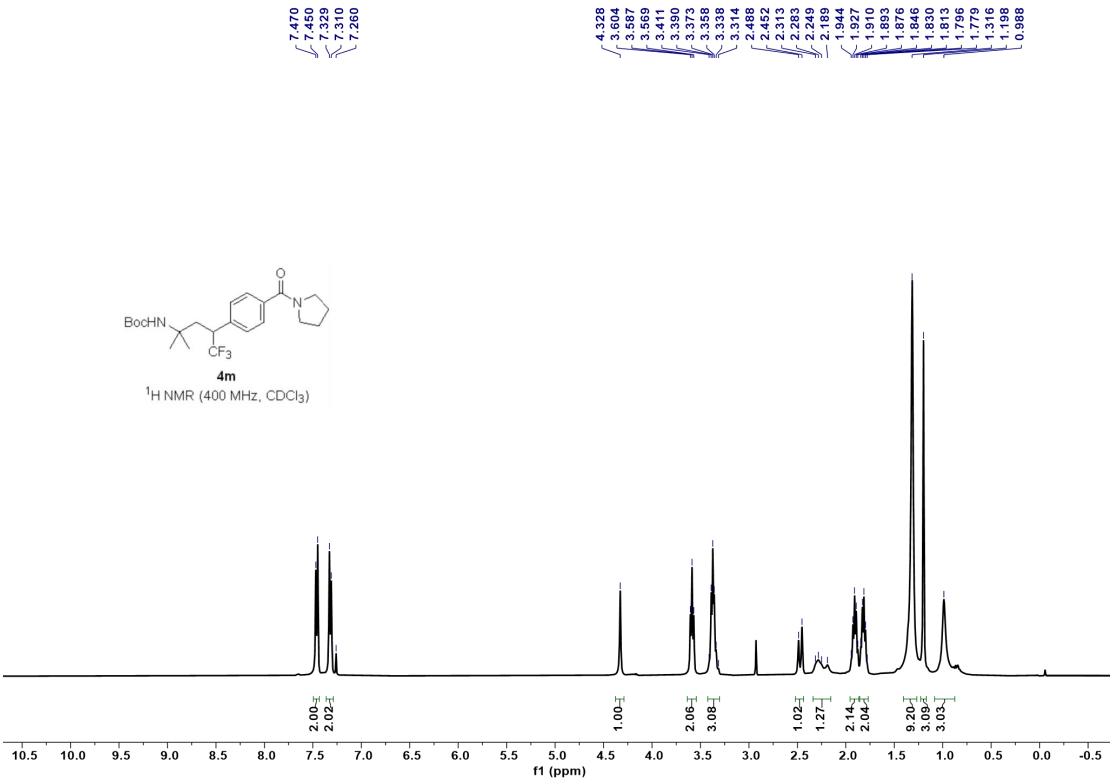


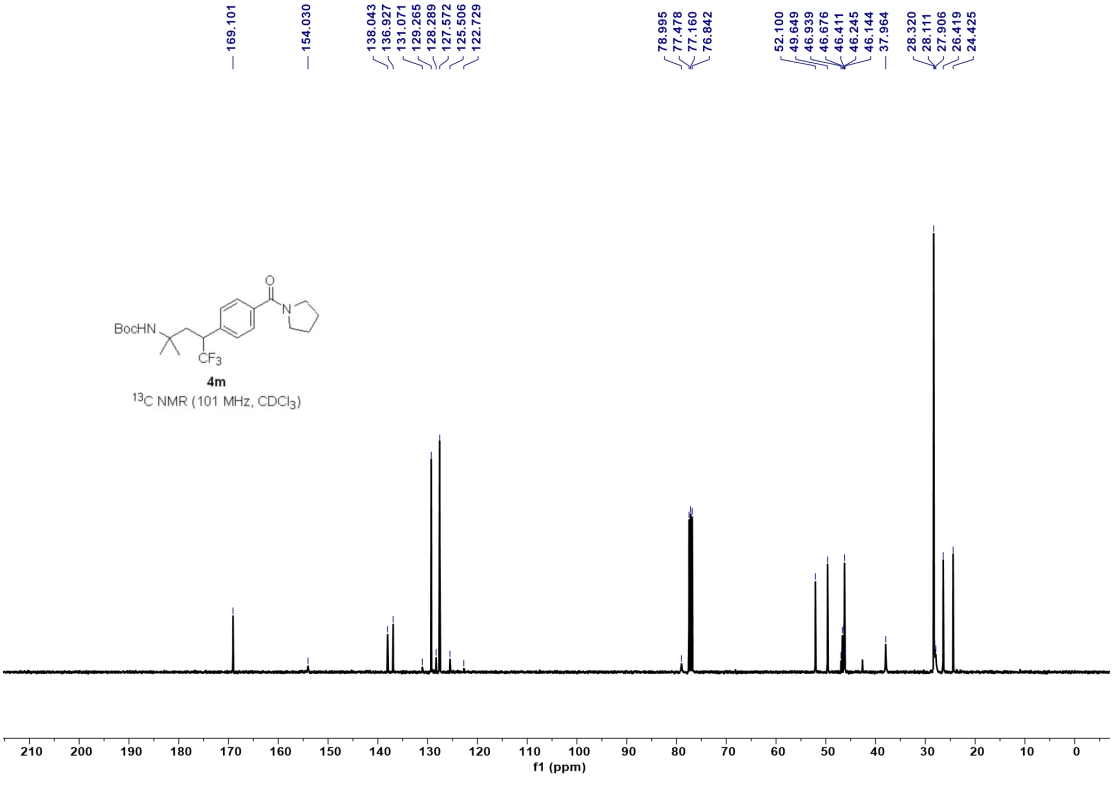
4l

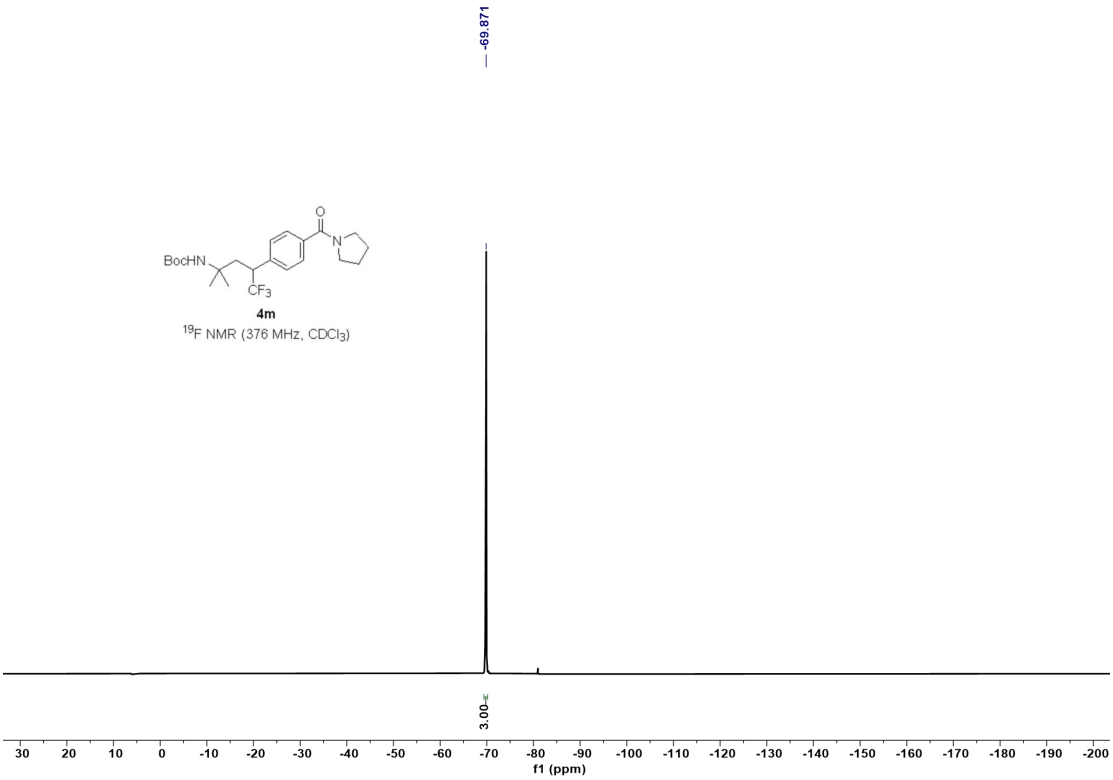
^{1}H NMR (400 MHz, $CDCl_3$)


169.056
155.906
137.228
135.910
130.791
128.971
128.010
127.622
125.230
122.449
79.334
77.479
77.160
76.842
49.629
47.874
47.598
47.330
47.056
46.237
37.907
29.260
28.348
26.387
24.408
BocHN
CF3
O
N
4l
13C NMR (101 MHz, CDCl3)
f1 (ppm)


-69.503
-69.528
BocHN
CF3
O
N
4l
19F NMR (376 MHz, CDCl3)
3.00
f1 (ppm)


7.470
7.450
7.329
7.310
7.260
4.328
3.604
3.587
3.569
3.411
3.390
3.373
3.358
3.338
3.314
2.488
2.452
2.313
2.283
2.249
2.189
1.944
1.927
1.910
1.893
1.876
1.846
1.830
1.813
1.796
1.779
1.316
1.198
0.988
BocHN
CF3
4m
1H NMR (400 MHz, CDCl3)
2.00
2.02
1.00
2.06
3.08
1.02
1.27
2.14
2.04
9.20
3.09
3.03
f1 (ppm)


169.101
154.030
138.043
136.927
131.071
129.265
128.289
127.572
125.506
122.729
78.995
77.478
77.160
76.842
52.100
49.649
46.939
46.676
46.411
46.245
46.144
37.964
28.320
28.111
27.906
26.419
24.425
BocHN
CF3
4m
13C NMR (101 MHz, CDCl3)
f1 (ppm)


-69.871
BocHN
CF3
O
N
4m
19F NMR (376 MHz, CDCl3)
3.00
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)