Supporting Information

Nickel-Catalyzed Reductive Cross-Coupling of Monofluoroalkyl Triflates with Aryl Halides for Monofluoroalkylated Arenes

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Supporting Information

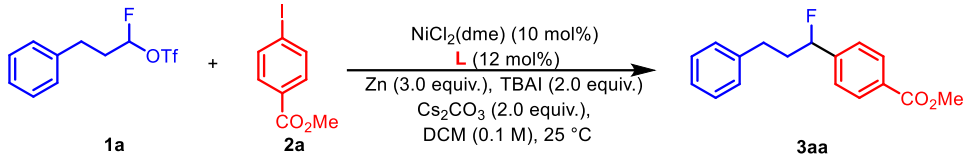
contents

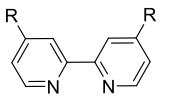
1. General Information

All reactions dealing with air- or moisture-sensitive compounds were performed in the argon-filled glovebox. Commercial reagents were used without further purification. Flash column chromatography was performed using 200–300 or 300–400 mesh silica gel. Thin layer chromatography (TLC) was performed on glass plates coated with silica gel 60 with F254 indicator. Proton nuclear magnetic resonance (^{1}H NMR) and (^{19}F NMR) spectra were recorded on a Bruker 400 MHz spectrometer. Chemical shifts (δ) are reported in ppm from the resonance of tetramethyl silane as the internal standard ($CDCl_3$: δ = 7.26 ppm for ^{1}H, TMS: δ = 0 ppm for ^{1}H, δ = 77.16 ppm for ^{13}C). ^{13}C NMR spectra were recorded on 100 MHz or 150 MHz with complete proton decoupling spectrophotometers. Data are represented as follows: chemical shift, multiplicity (*br* = broad, *s* = singlet, *d* = doublet, *t* = triplet, *q* = quartet, *m* = multiplet), coupling constants (*J*) are given in Hertz (Hz). High resolution mass spectra (HRMS) were measured with Bruker micr OTOF II ESI-TOF using a positive electrospray ionization (ESI^{+}).

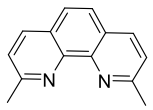
2. Optimization Studies

Table S1. The Effect of the Ligands on the Reaction[a].

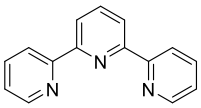




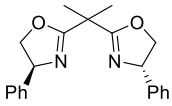
L1, R = H, trace
L2, R = ^{t}Bu, trace
^{t}Bu, 41%[b]
L3, R = OMe, trace
L4, R = Me, trace



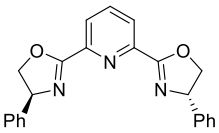
L5, trace



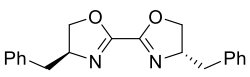
L6, trace



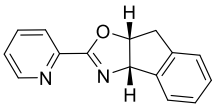
L7, trace



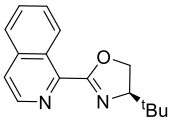
L8, < 5%



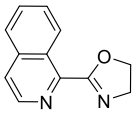
L9, 40%



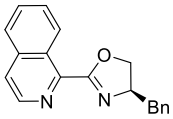
L10, 44%



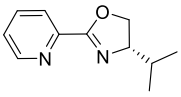
L11, 18%



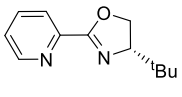
L12, 21%



L13, 35%



L14, 21%

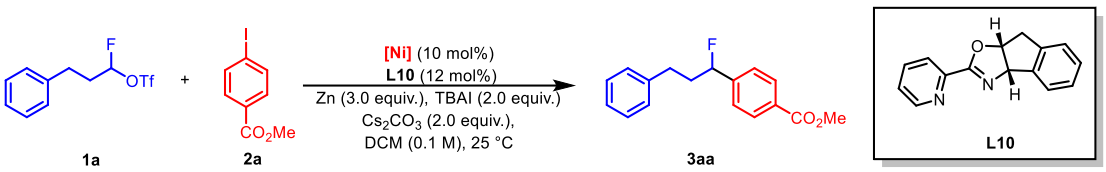


L15, 23%

[a]Reaction condition: **1a** (0.2 mmol), **2a** (2.0 equiv.), $NiCl_2$(dme) (10 mol%), **L** (12 mol%), Cs_2CO_3 (2.0 equiv.), TBAI (2.0 equiv.), Zn (3.0 equiv.), DCM (0.1 M), operating in the glovebox, 25 °C, 48 h. Yield of isolated product.

[b] THF (0.1M), 40 °C, 48 h.

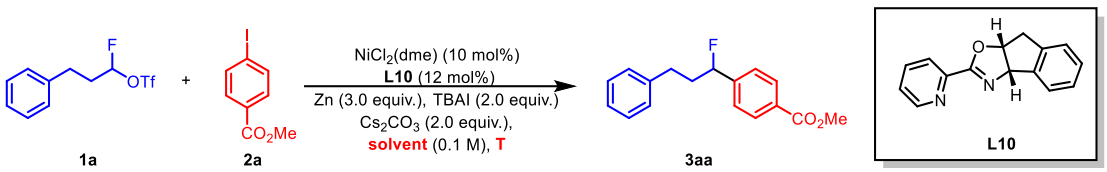
Table S2. The Effect of the Nickel Catalysts on the Reaction[a].



Entry	[Ni]	Yield[b](**3aa**) %
1	$NiCl_2(dme)$	44
2	$NiBr_2(dme)$	37
3	$Ni(OTf)_2$	24
4	$Ni(acac)_2$	trace
5	$Ni(ClO_4)_2$	8
6	$Ni(OAc)_2{\cdot}4H_2O$	30
7	$NiBr_2$	trace

[a] Reaction condition: **1a** (0.2 mmol), **2a** (2.0 equiv.), [Ni] (10 mol%), **L10** (12 mol%), Cs_2CO_3 (2.0 equiv.), TBAI (2.0 equiv.), Zn (3.0 equiv.), DCM (0.1 M), operating in the glovebox, 25 °C, 48 h. [b] Yield of isolated product.

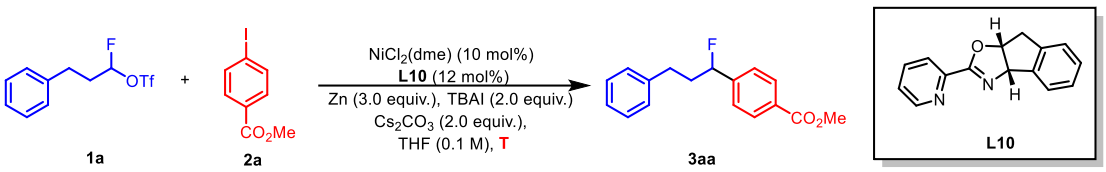
Table S3. The Effects of the Solvents and Temperature on the Reaction[a]



Entry	Solvent	T/°C	Yield[b](**3aa**) %
1	DCM	25	44
2	NMP	25	<5
3	MeCN	25	26
4	DCM	40	56
5	THF	40	96
6	1,4-dioxide	40	57
7	Et_2O	40	35

[a] Reaction condition: **1a** (0.2 mmol), **2a** (2.0 equiv.), $NiCl_2$(dme) (10 mol%), **L10** (12 mol%), Cs_2CO_3 (2.0 equiv.), TBAI (2.0 equiv.), Zn (3.0 equiv.), solvent (0.1 M), operating in the glovebox, T °C, 48 h. [b] Yield of isolated product.

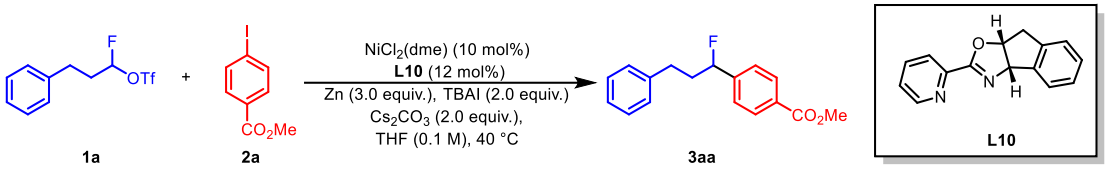
Table S4. The Effect of the Temperature on the Reaction[a].



Entry	Solvent	T/°C	Yield[b](**3aa**) %
1	THF	25	68
2	THF	40	96
3	THF	60	90
4	THF	80	93

[a] Reaction condition: **1a** (0.2 mmol), **2a** (2.0 equiv.), $NiCl_2$(dme) (10 mol%), **L10** (12 mol%), Cs_2CO_3 (2.0 equiv.), TBAI (2.0 equiv.), Zn (3.0 equiv.), THF (0.1 M), operating in the glovebox, T °C, 48 h. [b] Yield of isolated product.

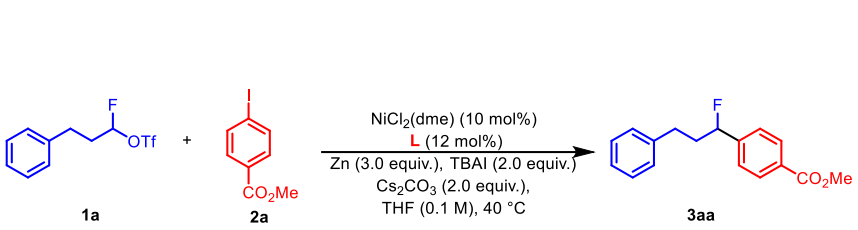
Table S5. Control Experiments[a].

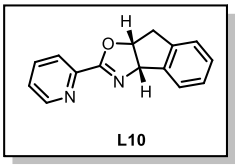


Entry	Conditions	Yield[b](**3aa**) %
1	none	96
2	without [Ni]	N.D.
3	without **L10**	23
4	without TBAI	N.D.
5	without Cs_2CO_3	71
6	without Zn	N.D.

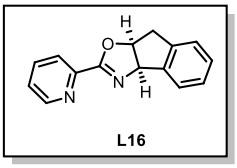
[a] Reaction condition: **1a** (0.2 mmol), **2a** (2.0 equiv.), $NiCl_2$(dme) (10 mol%), **L10** (12 mol%), Cs_2CO_3 (2.0 equiv.), TBAI (2.0 equiv.), Zn (3.0 equiv.), THF (0.1 M), operating in the glovebox, 40 °C, 48 h. [b] Yield of isolated product.

Table S6. The Effect of the **L16** and (±)-**L10** on the Reaction[a].





L10



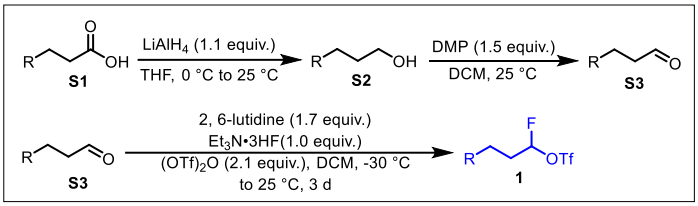
L16

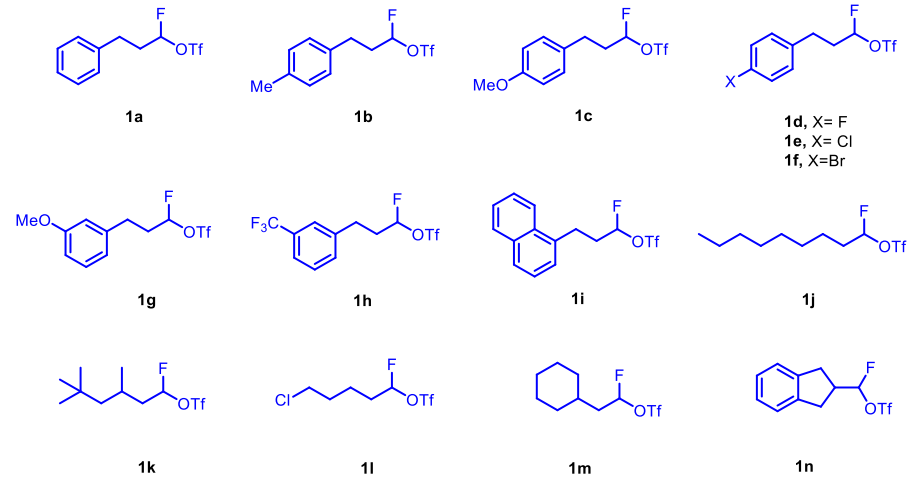
Entry	**L**	Yield[b](**3aa**) %
1	**L10**	96
2	**L16**	91
3	**L10/L16**(1:1)	85

[a] Reaction condition: **1a** (0.2 mmol), **2a** (2.0 equiv.), $NiCl_2$(dme) (10 mol%), **L** (12 mol%), Cs_2CO_3 (2.0 equiv.), TBAI (2.0 equiv.), Zn (3.0 equiv.), THF (0.1 M), operating in the glovebox, 40 °C, 48 h. [b] Yield of isolated product.

3. Preparation of the Substrates.

Procedure A: General procedure for the preparation of monofluoroalkyl triflates

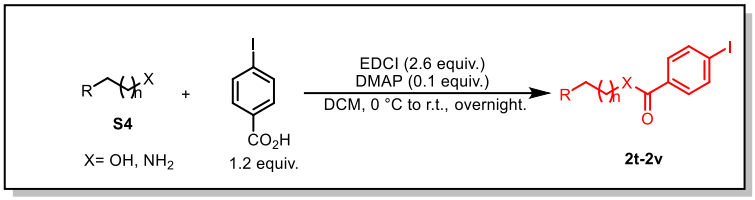


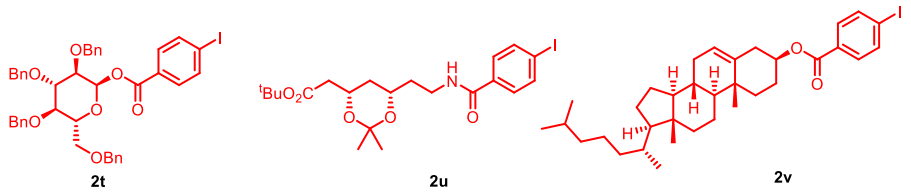


All of monofluoroalkyl triflates (**1a-1n**) were prepared through the reported procedures,[1] and the characterization of the compounds **1a, 1c-1g, 1i**, **1j**, and **1l-1m** were reported in the literature. **1b**, **1h**, **1k** and **1n** were synthesized according to the previous report.[1]

Under nitrogen atmosphere, in a 250 mL Schlenck bottle equipped with a magnetic stirring. Triflic anhydride (21 mmol, 2.1 equiv.), corresponding aldehyde (20 mmol, 1.0 equiv.), 2.6-lutidine (17 mmol, 1.7 equiv.) and 50 mL DCM was added at -30 ℃. The reaction mixture was stirred under N_2 for 2 days at -30 ℃. Et_3N•3HF (10 mmol, 1.0 equiv.) in 10 mL of DCM was added dropwise to the mixture of bistriflate at -30 ℃. The mixture was allowed to reach room temperature and stirred for overnight. The reaction was slowly quenched by the addition of saturated $NaHCO_3$ (50 mL) at 0 ℃ and the organic layer was washed with 1N HCl (50 mL) and saturated $NaHCO_3$ (50 mL). The organic phase was dried with Na_2SO_4, filtered, and concentrated for flash column chromatography to give the 1-fluoroalkyl triflates.

Procedure B: General procedure for the preparation of aryl halides.



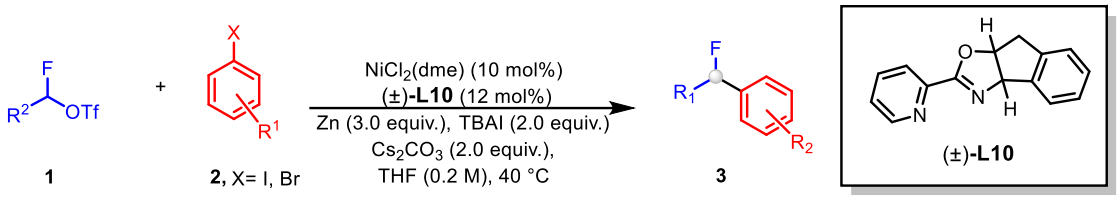


Aryl halides **2t-2v** were prepared according to the reported procedures,[2] other aryl halides were commercially available.

An oven round-bottom flask equipped with a magnetic stirring. 4-Iodobenzoic acid (1.2 equiv.), corresponding alcohol or alkylamine (1.0 equiv.), DMAP (0.1 equiv.), and 1-(3-Dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDCI, 2.6 equiv.) and DCM (20 mL) were added. The reaction mixture was stirred at room temperature for 12 h. Then the reaction was filtrated with a short silica pad and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 5:1 to 40:1) to afford the corresponding aryl halides.

4. Experimental Procedure.

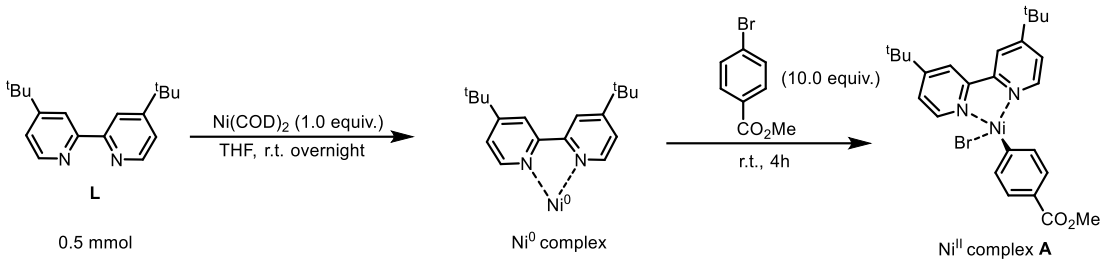
Procedure C: General procedure for the preparation of benzyl monofluoroalkanes



Inside a nitrogen-filled glovebox, a dry 4 mL gram bottle containing a stirring bar was added sequentially substrates **1** (0.4 mmol, 1.0 equiv.), aryl halides **2** (0.8 mmol, 2.0 equiv.), $NiCl_2$(dme) (8.8 mg, 10 mol%), (±)-**L10** (11.4 mg, 12 mol%), TBAI (295.4 mg, 0.8 mmol, 2.0 equiv.), and Cs_2CO_3 (260 mg, 0.8 mmol, 2.0 equiv.), Zn (78 mg, 1.2 mmol, 3.0 equiv.) in 2 mL of anhydrous THF. Then the reaction was taken out of the glovebox and stirred reaction at 40 ℃ for 48 hours. After the reaction was finished according to TLC, then the reaction was filtrated with a short silica pad and concentrated in vacuo. The mixture was purified by flash chromatography on silica gel, eluting with petroleum ether/ethyl acetate to afford the corresponding products.

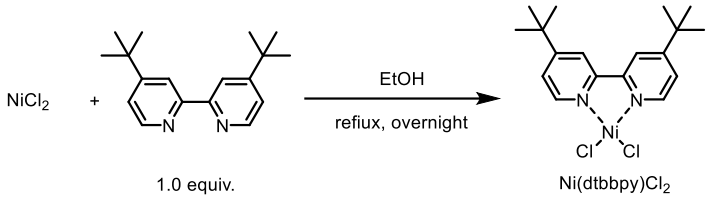
5. Control Experiments.

5.1 Procedure D: Synthesis of Ar-Ni(L)Br (Ni^{II} complex A).



The Ni^{II} complex **A** was prepared according to the literature procedure.[3] Inside a nitrogen-filled glovebox, a flame-dried round-bottomed flask was charged with $Ni(COD)_2$ (138 mg, 0.5 mmol, 1.0 equiv.), dry THF (4 mL) and 4,4'-di-tert-butyl-2,2'-bipyridine (134 mg, 0.5 mmol, 1.0 equiv.). After stirring at room temperature overnight, methyl 4-bromobenzoate (1.07 g, 5 mmol, 10.0 equiv.) was added and stirred for additional 4 h. Dry hexane (30 mL) was added slowly to the deep red colored mixture and filtered. The resulting precipitate was washed with hexane (4 x 10 mL) and dried under vacuum to afford Ni^{II} complex **A** as an orange solid (250 mg, 92% yield). The Ni^{II} complex **A** was used without further purification. The Ni^{II} complex **A** was stored in a nitrogen-filled glovebox at -20 ºC.

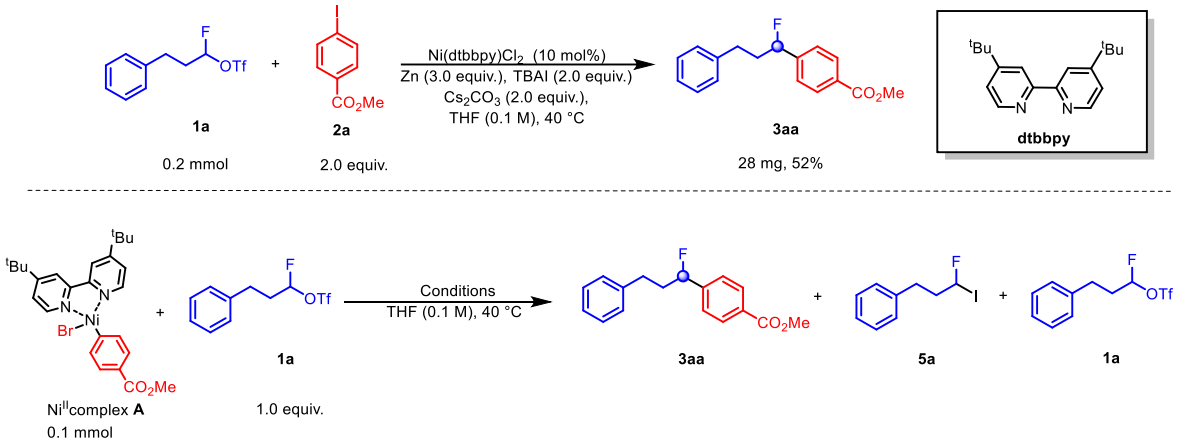
5.2 Procedure E: Synthesis of $NiCl_2$(4,4'-di*t*Bu-bpy).



1.0 equiv. Ni(dtbbpy)Cl_2

The **$NiCl_2$(4,4'-di*t*Bu-bpy)** was prepared according to the literature procedure.[4] An oven round-bottom flask equipped with a magnetic stirring. A solution of $NiCl_2$ (0.5 g, 3.85 mmol) in anhydrous ethanol (50 mL) was added to a stirred solution of 4,4'-ditBu-bpy (1.03 g, 3.85 mmol) in anhydrous ethanol (25 mL). The reaction mixture was refluxed with stirring at 85 ºC. Overnight the yellow solution slowly became green. The solution was filtrated and the filtrate was evaporated in vacuo to give a crude product (1.27 g, 83% yield). The product was recrystallized from anhydrous methanol.

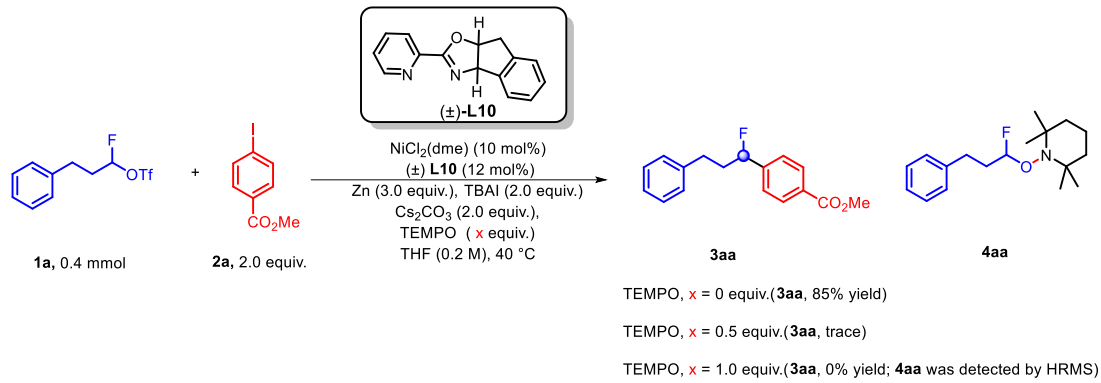
5.3 Stoichiometric reaction of Ni^{II} complex A with 1a.



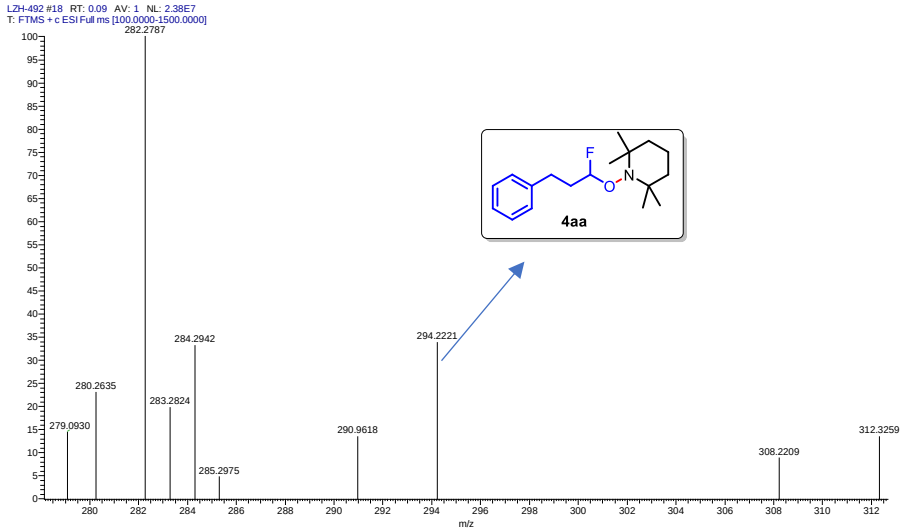
Entry	Zn	TBAI	Cs_2CO_3	**3aa**/%	**5a**/%	**1a**
1	3.0 equiv.	2.0 equiv.	2.0 equiv.	59	12	0
2	0	2.0 equiv.	2.0 equiv.	66	trace	0
3	3.0 equiv.	0	2.0 equiv.	26	0	trace
4	0	0	2.0 equiv.	7	0	trace
5	3.0 equiv.	2.0 equiv.	0	51	34	0

Inside a nitrogen-filled glovebox, a dry 4 mL gram bottle containing a stirring bar was charged with Ni^{II} complex **A** (54.2 mg, 0.1 mmol), TBAI (2.0 equiv.), and Cs_2CO_3 (2.0 equiv.), Zn (3.0 equiv.). Dissolved the substrate **1a** (28.6 mg, 0.1 mmol, 1.0 equiv.) in anhydrous THF (1 mL), and then added to the system through a dropper. Then the reaction was taken out of the glovebox and stirred at 40 ℃ for 48 hours. After the reaction was finished according to TLC, then the reaction was filtrated with a short silica pad and concentrated in vacuo. The mixture was purified by flash chromatography on silica gel, eluting with petroleum ether/ethyl acetate to afford the product **3aa** and by-product **5a**.

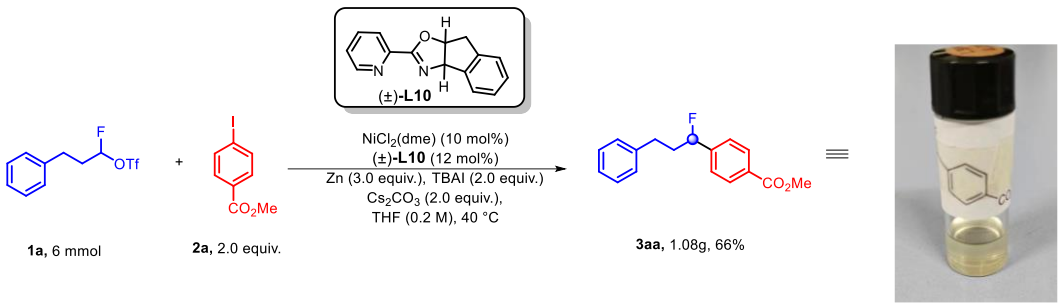
5.4 Reaction in presence of radical scavengers.



In a nitrogen-filled glovebox, a dry 4 mL gram bottle containing a stirring bar was charged with substrate **1a** (0.4 mmol, 1.0 equiv.), aryl halide **2a** (0.8 mmol, 2.0 equiv.), $NiCl_2$(dme) (8.8 mg, 10 mol%), (±)-**L10** (11.4 mg, 12 mol%), TBAI (295.4 mg, 0.8 mmol, 2.0 equiv.), and Cs_2CO_3 (260 mg, 0.8 mmol, 2.0 equiv.), Zn (78 mg, 1.2 mmol, 3.0 equiv.) in 2 mL of anhydrous THF. Then, the indicated amount of radical scavengers TEMPO (x equiv.) was added. Then the reaction was taken out of the glovebox and stirred at 40 ℃ for 48 hours. After the reaction was finished according to TLC, then the reaction was filtrated with a short silica pad and concentrated in vacuo. The mixture was purified by flash chromatography on silica gel, eluting with petroleum ether/ethyl acetate to afford the corresponding products **3aa**, **4aa**, and **4aa** was detected by HRMS. HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{18}H_{29}FNO$ = 294.2228, found: 294.2221.



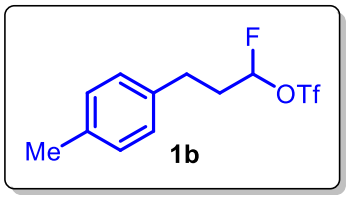
6. Gram-scale experiment



In a nitrogen-filled glovebox, a dry 50 mL sealed tube containing a stirring bar was charged with substrate **1a** (1.71g, 6.0 mmol, 1.0 equiv.), aryl halide **2a** (3.14 g, 12 mmol, 2.0 equiv.), $NiCl_2$(dme) (131 mg, 10 mol%), (±)-**L10** (170 mg, 12 mol%), TBAI (4.43 g, 12 mmol, 2.0 equiv.), and Cs_2CO_3 (3.9 g, 12 mmol, 2.0 equiv.), Zn (1.17 g, 18 mmol, 3.0 equiv.) in 30 mL of anhydrous THF. Then the reaction was taken out of the glovebox and stirred at 40 ℃ for 48 hours. After the reaction was finished according to TLC, then the reaction was filtrated with a short silica pad and concentrated in vacuo. The mixture was purified by flash chromatography on silica gel, eluting with petroleum ether/ethyl acetate to afford the corresponding products **3aa** (1.08 g, 66% yield).

7. Characterization Date of Substrates 1 and Products.

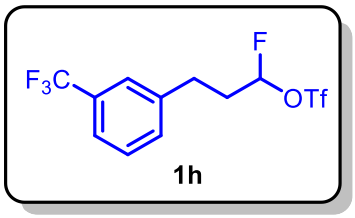
1-fluoro-3-(p-tolyl) propyl trifluoromethanesulfonate



The general **procedure A** was followed using 3-(*p*-tolyl) propanal (1.90 g, 12.8 mmol) purification by column chromatography on silica gel, yielding **1b** (0.95 g, 25%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (d, *J* = 7.8 Hz, 2H), 7.09 (d, *J* = 7.8 Hz, 2H), 6.12 (dt, *J* = 54.4, 5.1 Hz, 1H), 2.78 (t, *J* = 7.9 Hz, 2H), 2.35 (s, 3H), 2.32–2.24 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.5, 135.6, 129.7, 128.2, 118.4 (q, *J* = 319.4 Hz), 112.0 (d, *J* = 245.9 Hz), 35.4 (d, *J* = 20.0 Hz), 28.3 (d, *J* = 5.4 Hz), 21.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ -74.94, -120.24; HRMS (ESI) *m/z*: $[M+K]^+$ calcd. for $C_{11}H_{12}F_4O_3SK$ = 339.0075, found: 339.0082.

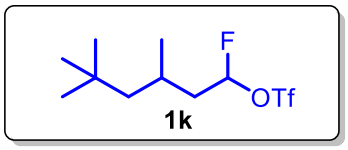
1-fluoro-3-(3-(trifluoromethyl)phenyl) propyl trifluoromethanesulfonate



The general **procedure A** was followed using 3-(3-(trifluoromethyl) phenyl) propanal (3.20 g, 15.8 mmol) purification by column chromatography on silica gel, yielding **1h** (0.95 g, 37%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54–7.39 (m, 4H), 6.17 (dt, *J* = 54.2, 4.9 Hz, 1H), 2.90 (t, *J* = 8.0 Hz, 2H), 2.39–2.29 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.8, 131.8, 131.8(4)–130.9 (q, *J* = 32 Hz), 129.5, 128.2–120.1 (q, *J* = 270.0 Hz), 125.1 (q, *J* = 3.9 Hz), 123.9 (q, *J* = 3.9 Hz), 118.4 (q, *J* = 319.6 Hz), 111.5 (d, *J* = 245.7 Hz), 35.1 (d, *J* = 20.4 Hz), 28.4 (d, *J* = 5.4 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.77, -74.98, -120.43; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{11}H_{10}F_7O_3S$ = 355.0233, found: 355.0243.

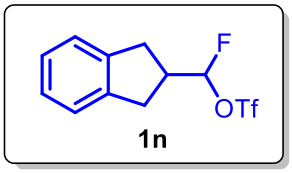
1-fluoro-3,5,5-trimethylhexyl trifluoromethanesulfonate



The general **procedure A** was followed using 3,5,5-trimethylhexanal (1.40 g, 10.0 mmol) purification by column chromatography on silica gel, yielding **1k** (1.56 g, 53%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.25–6.08 (m, 1H) (major + minor), 2.09–1.92 (m, 1H), 1.88–1.68 (m, 2H), 1.27 (dd, *J* = 4.1, 1.8 Hz, 1H) (minor); 1.23 (dd, *J* = 4.1, 1.8 Hz, 1H) (major), 1.19 (d, *J* = 6.0 Hz, 1H) (major); 1.16 (d, *J* = 6.0 Hz, 1H) (minor), 1.03 (dd, *J* = 6.6, 3.6 Hz, 3H) (major + minor), 0.92 (s, 9H) (major); 0.91 (s, 9H) (minor); ^{13}C NMR (101 MHz, $CDCl_3$) δ 123.2-113.6 (q, *J* = 319.4 Hz) , 112.3 (dd, *J* = 245.7, 16.4 Hz) (major + minor), 51.0 (d, *J* = 10.7 Hz) (major + minor), 42.7 (dd, *J* = 18.8, 9.7 Hz) (major + minor), 31.2 (d, *J* = 2.6 Hz) (major + minor), 29.9 (d, *J* = 6.4 Hz) (major + minor), 24.7 (d, *J* = 4.2 Hz) (major + minor), 22.4 (d, *J* = 13.9 Hz) (major + minor); ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.12, -75.18, -117.95, -119.46; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{10}H_{19}F_4O_3S$ =295.0986, found: 295.0992.

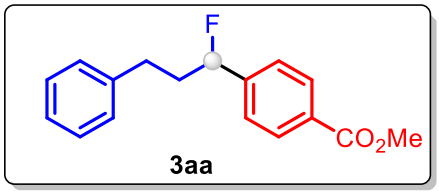
(2,3-dihydro-1*H*-inden-2-yl) fluoromethyl trifluoromethanesulfonate



The general **procedure A** was followed using 2,3-dihydro-1***H***-indene-2-carbaldehyde (1.90 g, 13.0 mmol) purification by column chromatography on silica gel, yielding **1n** (0.85 g, 22%) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24–7.17 (m, 4H), 6.12 (dd, *J* = 54.6, 5.6 Hz, 1H), 3.22–3.13 (m, 2H), 3.10–2.91 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.5 (d, *J* = 14.0 Hz), 127.2, 124.7, 118.4 (q, *J* = 319.4 Hz), 113.8 (d, *J* = 247.3 Hz), 42.5 (d, *J* = 19.5 Hz), 33.4 (d, *J* = 5.6 Hz), 33.2 (d, *J* = 3.2 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -74.73, -122.16 ; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{11}H_{11}F_4O_3S$ = 299.0360, found: 299.0360.

Methyl 4-(1-fluoro-3-phenylpropyl) benzoate

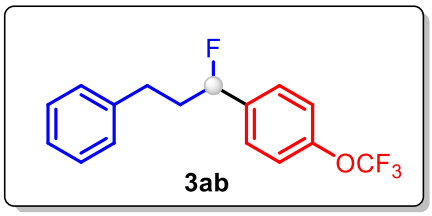


3aa

The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3aa** (92.0 mg, 85%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.2 Hz, 2H), 7.39 (d, *J* = 8.2 Hz, 2H), 7.30 (t, *J* = 7.6 Hz, 2H), 7.25–7.14 (m, 3H), 5.50 (ddd, *J* = 47.8, 8.7, 4.1 Hz, 1H), 3.93 (s, 3H), 2.88–2.69 (m, 2H), 2.38–2.01 (m, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.27. The analytical data are in accordance to those reported in the literature[5].

1-(1-fluoro-3-phenylpropyl)-4-(trifluoromethoxy)benzene

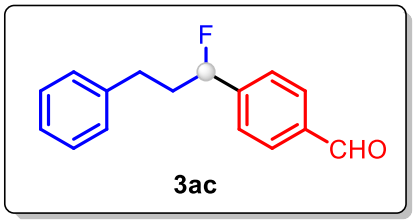


3ab

The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and 1-iodo-4-(trifluoromethoxy)benzene (**2b**) (230.4 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ab** (56.0 mg, 47%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36–7.28 (m, 4H), 7.23–7.18 (m, 5H), 5.43 (ddd, *J* = 47.6, 8.7, 4.3 Hz, 1H), 2.86–2.71 (m, 2H), 2.34–2.01 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.2, 140.9, 139.1, 138.9, 128.6 (d, *J* = 9.1 Hz), 127.2 (d, *J* = 6.8 Hz), 120.7 (q, *J* = 257.4 Hz) 124.4 , 121.1 , 92.9 (d, *J* = 171.9 Hz) , 39.0 (d, *J* = 23.6 Hz) , 31.4 (d, *J* = 4.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -57.64 , -176.33; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{16}H_{14}F_4ONa$ =321.0873, found: 321.0885.

4-(1-fluoro-3-phenylpropyl) benzaldehyde

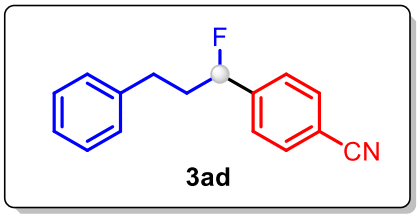


3ac

The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and 4-iodobenzaldehyde (**2c**) (185.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ac** (58.0 mg, 60%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.02 (s, 1H), 7.90 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 2H), 7.34–7.30 (m, 2H), 7.28–7.18 (m, 3H), 5.53 (ddd, *J* = 47.8, 8.6, 4.0 Hz, 1H), 2.88–2.75 (m, 2H), 2.35–2.06 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.8, 147.0 (d, *J* = 19.7 Hz), 140.8, 136.2, 130.02 , 128.60 (d, *J* = 11.8 Hz), 126.31 , 125.90 (d, *J* = 7.5 Hz), 92.85 (d, *J* = 173.5 Hz), 39.0 (d, *J* = 23.1 Hz), 31.20 (d, *J* = 3.9 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -181.07; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{16}H_{15}FONa$ = 265.0999, found: 365.1004.

4-(1-fluoro-3-phenylpropyl) benzonitrile

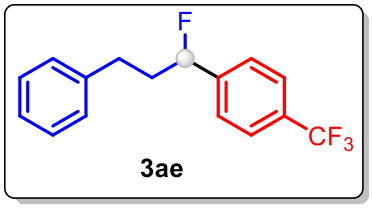


3ad

The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and 4-bromobenzonitrile (**2d**) (145.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ad** (68.0 mg, 71%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.1 Hz, 2H), 7.43 (d, *J* = 8.1 Hz, 2H), 7.32 (t, *J* = 7.4 Hz, 2H), 7.26–7.19 (m, 3H), 5.50 (ddd, *J* = 47.7, 8.7, 4.0 Hz, 1H), 2.87–2.75 (m, 2H), 2.33–2.04 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 145.6, 145.4, 140.6, 132.4, 128.6 (d, *J* = 16.3 Hz), 126.4, 126.0 (d, *J* = 7.7 Hz), 118.6, 112.0, 92.4 (d, *J* = 174.3 Hz), 38.9 (d, *J* = 23.1 Hz), 31.1 (d, *J* = 3.8 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -181.79; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{16}H_{15}FN$ = 240.1183, found: 240.1186.

1-(1-fluoro-3-phenylpropyl)-4-(trifluoromethyl)benzene

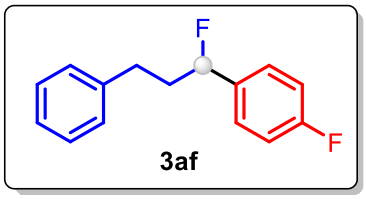


3ae

The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and 1-iodo-4-(trifluoromethyl)benzene (**2e**) (217.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ae** (57.0 mg, 51%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, J = 8.0 Hz, 2H), 7.42 (d, J = 8.0 Hz, 2H), 7.32–7.28 (m, 2H), 7.22–7.18 (m, 3H), 5.48 (ddd, J = 47.8, 8.7, 4.1 Hz, 1H), 2.85–2.72 (m, 2H), 2.33–2.03 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.4, 144.2, 140.8, 130.5 (q, J = 33.2 Hz), 128.6 (d, J = 11.1 Hz), 126.4, 125.8 (d, J = 7.5 Hz), 125.6 (q, J = 3.6 Hz), 122.8, 92.8 (d, J = 173.2 Hz), 39.1 (d, J = 23.3 Hz), 31.2 (d, J = 4.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.54 , -180.22; HRMS (ESI) *m/z*: $[M+K]^+$ calcd. for $C_{16}H_{14}F_4K$ = 321.0663, found: 321.0659.

1-fluoro-4-(1-fluoro-3-phenylpropyl) benzene

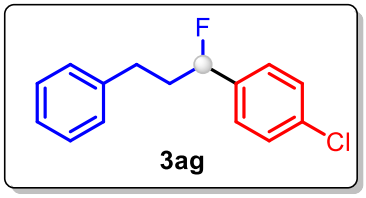


3af

The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and 1-fluoro-4-iodobenzene (**2f**) (177.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3af** (40.0 mg, 43%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31–7.27 (m, 4H), 7.23–7.18 (m, 3H), 7.07–7.03 (m, 2H), 5.40 (ddd, J = 47.8, 8.5, 4.3 Hz, 1H), 2.83–2.69 (m, 2H), 2.35–2.00 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.0, 161.5, 141.1, 136.1 (d, J = 20.0 Hz), 128.6 (d, J = 7.0 Hz), 127.6 (t, J = 7.4 Hz), 126.3, 115.6 (d, J = 21.5 Hz), 93.2 (d, J = 170.9 Hz), 38.9 (d, J = 24.0 Hz), 31.4 (d, J = 4.2 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -113.62 , -173.74; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{15}H_{15}F_2$= 233.1136, found: 233.1138.

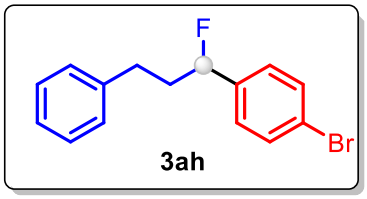
1-chloro-4-(1-fluoro-3-phenylpropyl) benzene



The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and 1-chloro-4-iodobenzene (**2g**) (190.7 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ag** (65.0 mg, 66%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34–7.18 (m, 9H), 5.39 (ddd, J = 47.6, 8.4, 4.1 Hz, 1H), 2.83–2.68 (m, 2H), 2.33–1.99 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.0, 138.9, 138.7, 134.2 (d, J = 2.5 Hz), 128.8, 128.6 (d, J = 8.3 Hz), 127.1 (d, J = 6.8 Hz), 126.3 , 93.0 (d, J = 171.9 Hz), 38.9 (d, J = 23.7 Hz), 31.3 (d, J = 4.2 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -176.43; HRMS (ESI) *m/z*: $[M+K]^+$ calcd. for $C_{15}H_{14}ClFK$ = 287.0400, found: 287.0396.

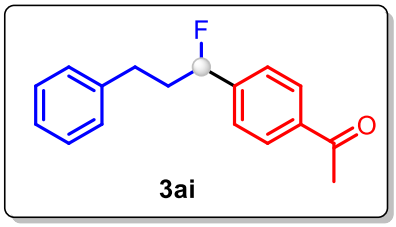
1-bromo-4-(1-fluoro-3-phenylpropyl) benzene



The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and 1-bromo-4-iodobenzene (**2h**) (226.3 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ah** (83.0 mg, 71%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, J = 8.3 Hz, 2H), 7.31–7.27 (m, 2H), 7.22–7.17 (m, 5H), 5.37 (ddd, J = 47.7, 8.6, 4.4 Hz, 1H), 2.82–2.68 (m, 2H), 2.32–1.98 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.9, 139.3 (d, J = 20.2 Hz), 137.7, 131.7, 128.6 (d, J = 8.4 Hz), 127.4 (d, J = 6.8 Hz), 126.3, 122.3 (d, J = 2.5 Hz), 93.0 (d, J = 171.8 Hz), 38.9 (d, J = 23.4 Hz), 31.3 (d, J = 4.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -176.93; HRMS (ESI) *m/z*: $[M+K]^+$ calcd. for $C_{15}H_{14}BrFK$ = 330.9894, found: 330.9883.

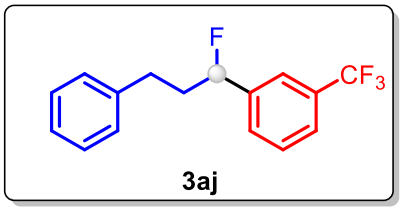
1-(4-(1-fluoro-3-phenylpropyl) phenyl) ethan-1-one



The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (57.2 mg, 0.2 mmol) and 1-(4-iodophenyl)ethan-1-one (**2i**) (98.4 mg, 0.4 mmol) purification by column chromatography on silica gel, yielding **3ai** (23.0 mg, 45%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.7 Hz, 2H), 7.42 (d, *J* = 8.1 Hz, 2H), 7.33–7.29 (m, 2H), 7.23–7.19 (m, 3H), 5.50 (ddd, *J* = 47.8, 8.6, 4.1 Hz, 1H), 2.86–2.73 (m, 2H), 2.62 (s, 3H), 2.34–2.05 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.8, 145.6 , 145.4, 140.8, 137.0, 128.7 (d, *J* = 2.0 Hz), 128.6, 126.3, 125.6 (d, *J* = 7.4 Hz), 93.0 (d, *J* = 173.0 Hz), 39.0 (d, *J* = 23.2 Hz), 31.2 (d, *J* = 4.1 Hz), 26.8; ^{9}F NMR (376 MHz, $CDCl_3$) δ -180.45; HRMS (ESI) *m/z*: $[M+K]^+$ calcd. for $C_{17}H_{17}FOK$ = 295.0895, found: 295.0876.

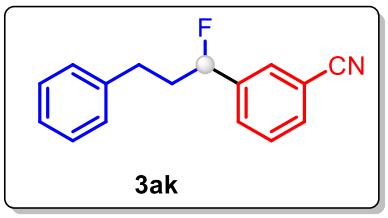
1-(1-fluoro-3-phenylpropyl)-3-(trifluoromethyl)benzene



The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and 1-iodo-3-(trifluoromethyl)benzene (**2j**) (218.0 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3aj** (55.0 mg, 49%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58–7.55 (m, 2H), 7.51–7.45 (m, 2H), 7.32–7.28 (m, 2H), 7.22–7.19 (m, 3H), 5.47 (ddd, *J* = 47.7, 8.8, 4.1 Hz, 1H), 2.86–2.73 (m, 2H), 2.35 –2.03 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.4 (d, *J* = 20.4 Hz), 140.8, 131.0 (q, *J* = 32.4 Hz), 129.2, 128.8 (d, *J* = 6.9 Hz), 128.6 (d, *J* = 10.6 Hz), 126.4, 125.4, 125.2, 122.8, 122.4 (dd, *J* = 7.5, 3.8 Hz), 92.8 (d, *J* = 172.9 Hz), 39.0 (d, *J* = 23.4 Hz), 31.3 (d, *J* = 4.0 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.63 , -178.86; HRMS (ESI) *m/z*: $[M+K]^+$ calcd. for $C_{16}H_{14}F_4K$ = 321.0663, found: 321.0687.

3-(1-fluoro-3-phenylpropyl) benzonitrile

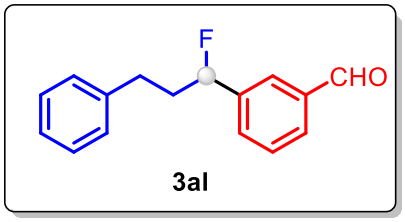


3ak

The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (57.2 mg, 0.2 mmol) and 3-iodobenzonitrile (**2k**) (91.6 mg, 0.4 mmol) purification by column chromatography on silica gel, yielding **3ak** (40.0 mg, 84%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 5.4 Hz, 2H), 7.54 (d, *J* = 7.9 Hz, 1H), 7.49–7.45 (m, 1H), 7.32–7.28 (m, 2H), 7.24–7.18 (m, 3H), 5.45 (ddd, *J* = 47.5, 8.7, 4.0 Hz, 1H), 2.85–2.72 (m, 2H), 2.32–2.02 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.0, 141.8, 140.6, 131.9, 129.8 (d, *J* = 7.0 Hz), 129.5, 129.0 (d, *J* = 7.9 Hz), 128.6 (d, *J* = 16.9 Hz), 126.4, 118.6, 112.8 , 92.3 (d, *J* = 173.9 Hz), 38.9 (d, *J* = 23.2 Hz), 31.2 (d, *J* = 4.0 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.22; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{16}H_{15}FN$ = 240.1183, found: 240.1186.

3-(1-fluoro-3-phenylpropyl) benzaldehyde

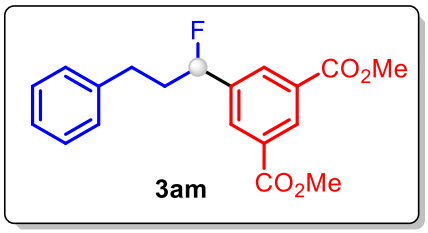


3al

The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and 3-iodobenzaldehyde (**2l**) (185.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3al** (63.0 mg, 65%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.04 (s, 1H), 7.85 (d, *J* = 5.6 Hz, 2H), 7.63–7.54 (m, 2H), 7.34–7.30 (m, 2H), 7.22–7.19 (m, 3H), 5.53 (ddd, *J* = 47.7, 8.7, 4.1 Hz, 1H), 2.89–2.75 (m, 2H), 2.39–2.08 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.0, 141.7, 141.5, 140.8, 136.6, 131.4 (d, *J* = 7.0 Hz), 129.8, 129.4, 128.6 (d, *J* = 10.4 Hz), 126.5 (d, *J* = 7.2 Hz), 126.3, 92.8 (d, *J* = 172.7 Hz), 39.0 (d, *J* = 23.3 Hz), 31.2 (d, *J* = 4.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -178.67; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{16}H_{16}FO$ =243.1185, found: 243.1173.

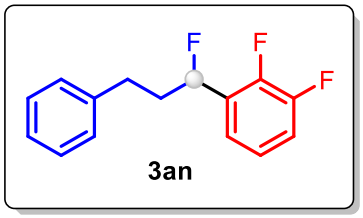
Dimethyl 5-(1-fluoro-3-phenylpropyl) isophthalate



The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and dimethyl 5-iodoisophthalate (**2m**) (256.0 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3am** (61.0 mg, 46%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (s, 1H), 8.19 (s, 2H), 7.32–7.28 (m, 2H), 7.21 (dd, *J* = 7.5, 5.5 Hz, 3H), 5.53 (ddd, *J* = 47.6, 8.8, 4.0 Hz, 1H), 3.95 (s, 6H), 2.87–2.74 (m, 2H), 2.37–2.06 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.9, 141.4 (d, *J* = 20.7 Hz), 140.6, 133.8, 131.0, 130.8 (d, *J* = 7.1 Hz), 130.5, 128.6 (d, *J* = 9.7 Hz), 126.3, 92.5 (d, *J* = 173.5 Hz), 52.6, 38.9 (d, *J* = 23.3 Hz), 31.2 (d, *J* = 3.9 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -179.21; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{19}H_{20}FO_4$ = 331.1340, found: 331.1343.

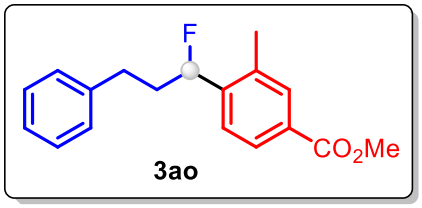
1,2-difluoro-3-(1-fluoro-3-phenylpropyl) benzene



The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and 1,2-difluoro-3-iodobenzene (**2n**) (192.0 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3an** (56.0 mg, 56%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33–7.27 (m, 2H), 7.24–7.20 (m, 4H), 7.18–7.10 (m, 2H), 5.77 (ddd, *J* = 47.0, 8.5, 4.2 Hz, 1H), 2.92–2.73 (m, 2H), 2.39–2.09 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.6 (d, *J* = 12.8 Hz), 149.2 (d, *J* = 12.3 Hz), 140.8, 130.1 (dd, *J* = 21.9, 10.1 Hz), 128.6 (d, *J* = 11.4 Hz), 126.3, 124.5 (d, *J* = 4.4 Hz), 124.5 (d, *J* = 4.7 Hz), 121.7, 117.1 (d, *J* = 17.0 Hz), 88.8–87.0 (dt, *J* = 173.0 Hz, *J* = 3.0 Hz), 37.9 (d, *J* = 23.3 Hz), 31.2 (d, *J* = 3.8 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -138.33, -144.00 , -183.18; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{15}H_{13}F_3Na$ = 273.0862, found: 273.0836.

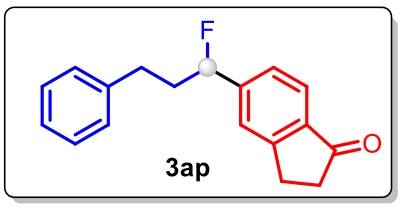
Methyl 4-(1-fluoro-3-phenylpropyl)-3-methylbenzoate



The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and methyl 4-iodo-3-methylbenzoate (**2o**) (220.8 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ao** (50.0 mg, 44%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.2 Hz, 1H), 7.82 (s, 1H), 7.50 (d, *J* = 8.2 Hz, 1H), 7.33–7.29 (m, 2H), 7.23–7.20 (m, 3H), 5.64 (ddd, *J* = 47.5, 9.0, 3.2 Hz, 1H), 3.91 (s, 3H), 2.93–2.77 (m, 2H), 2.30–1.97 (m, 5H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.0, 143.6 (d, *J* = 18.8 Hz), 140.9, 134.3 (d, *J* = 5.2 Hz), 131.7, 129.7, 128.6 (d, *J* = 4.6 Hz), 127.5, 126.3, 125.3, 125.2, 90.6 (d, *J* = 171.3 Hz), 52.3, 37.9 (d, *J* = 23.9 Hz), 31.6 (d, *J* = 3.2 Hz), 18.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -182.51; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{18}H_{20}FO_2$= 287.1442, found: 287.1434.

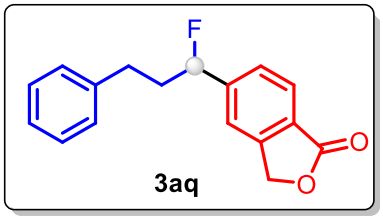
5-(1-fluoro-3-phenylpropyl)-2,3-dihydro-1H-inden-1-one



The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and 5-bromo-2,3-dihydro-1*H*-inden-1-one (**2p**) (168.8 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ap** (80.0 mg, 75%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.9 Hz, 1H), 7.45 (s, 1H), 7.32–7.28 (m, 3H), 7.23–7.19 (m, 3H), 5.51 (ddd, *J* = 47.9, 8.8, 3.8 Hz, 1H), 3.15 (t, *J* = 5.8 Hz, 2H), 2.87–2.76 (m, 2H), 2.73–2.70 (m, 2H), 2.35–2.05 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.6, 155.6, 147.5, 147.3, 140.8, 137.0, 128.6 (d, *J* = 10.7 Hz), 126.3, 124.6 (d, *J* = 7.0 Hz), 124.0, 123.4 (d, *J* = 7.9 Hz), 93.2 (d, *J* = 173.5 Hz), 39.2 (d, *J* = 23.2 Hz), 36.5, 31.2 (d, *J* = 3.9 Hz), 25.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.27; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{18}H_{17}FONa$ = 291.1156, found: 291.1152.

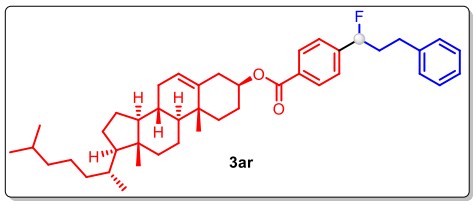
5-(1-fluoro-3-phenylpropyl) isobenzofuran-1(3*H*)-one



The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (114.4 mg, 0.4 mmol) and 5-bromoisobenzofuran-1(3*H*)-one (**2q**) (170.4 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3aq** (92.0 mg, 77%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.8 Hz, 1H), 7.55–7.43 (m, 2H), 7.32–7.28 (m, 2H), 7.23–7.19 (m, 3H), 5.56 (ddd, *J* = 47.7, 8.7, 4.0 Hz, 1H), 5.31 (s, 2H), 2.87–2.75 (m, 2H), 2.34–2.06 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.6, 147.2, 147.0 (d, *J* =3.1 Hz), 140.5, 134.0, 128.6 (d, *J* = 15.2 Hz), 126.2 (d, *J* = 9.2 Hz), 125.8, 125.6 (d, *J* = 15.4 Hz), 122.2, 118.9 (d, *J* = 8.4 Hz), 92.8 (d, *J* = 174.3 Hz), 69.6, 39.2 (d, *J* = 23.1 Hz), 31.1 (d, *J* = 3.8 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.70; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{17}H_{16}FO_2$ = 271.1129, found: 271.1128.

(3*S*,8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[a]phenanthren-3-yl 4-(1-fluoro-3-phenylpropyl) benzoate

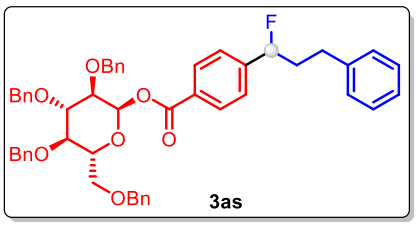


The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (57.2 mg, 0.2 mmol) and (3*S*,8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[a]phenanthren-3-yl 4-iodobenzoate (**2r**) (253.0 mg, 0.4 mmol) purification by column chromatography on silica gel, yielding **3ar** (66.0 mg, 52%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.1 Hz, 2H), 7.38 (d, *J* = 8.1 Hz, 2H), 7.32–7.28 (m, 2H), 7.23–7.19 (m, 3H), 5.57–5.42 (m, 2H), 4.91–4.83 (m, 1H), 2.84–2.72 (m, 2H), 2.47 (d, *J* = 7.6 Hz, 2H), 2.34–2.08 (m, 2H), 2.05–1.98 (m, 3H), 1.92 (dt, *J* = 13.5, 3.5 Hz, 1H), 1.87–1.69 (m, 2H), 1.63–1.45 (m, 6H), 1.39–1.25 (m, 6H), 1.22–1.09 (m, 5H), 1.07 (s, 3H), 1.06–0.96 (m, 3H), 0.93 (d, *J* = 6.5 Hz, 3H), 0.87 (dd, *J* = 6.6, 1.9

Hz, 6H), 0.69 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 145.1 (d, *J* = 19.5 Hz), 141.0, 139.7, 130.8, 129.9, 128.6 (d, *J* = 8.0 Hz), 126.3, 125.3 (d, *J* = 7.3 Hz), 123.0, 93.1 (d, *J* = 172.8 Hz), 74.8, 56.8, 56.3, 50.2, 42.5, 39.9, 39.7, 39.2, 38.9, 38.3, 37.2, 36.8, 36.3, 36.0, 32.1 (d, *J* = 6.6 Hz), 31.3 (d, *J* = 3.7 Hz), 28.4, 28.2, 28.0, 24.4, 24.0, 23.0, 22.7, 21.2, 19.5, 18.9, 12.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.02; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{43}H_{59}FO_2Na$ = 649.4391, found: 649.4397.

(2*R*,3*R*,4*S*,5*R*,6*R*)-3,4,5-tris(benzyloxy)-6-((benzyloxy)methyl) tetrahydro-2*H*-pyran-2-yl 4-(1-fluoro-3-phenylpropyl) benzoate

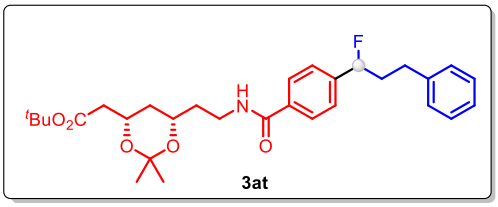


3as

The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (57.2 mg, 0.2 mmol) and (2*R*,3*R*,4*S*,5*R*,6*R*)-3,4,5-tris(benzyloxy)-6-((benzyloxy)methyl)tetrahydro-2*H*-pyran-2-yl 4-iodobenzoate (**2s**) (308.2 mg, 0.4 mmol) purification by column chromatography on silica gel, yielding **3as** (39.0 mg, 25%) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.0 Hz, 2H), 7.41 (d, *J* = 8.0 Hz, 2H), 7.36–7.27 (m, 15H), 7.24–7.21 (m, 8H), 7.17 (dd, *J* = 7.2, 2.5 Hz, 2H), 5.93–5.88 (m, 1H), 5.52 (ddd, *J* = 47.8, 8.6, 4.1 Hz, 1H), 4.95–4.76 (m, 5H), 4.65–4.48 (m, 3H), 3.87–3.74 (m, 5H), 3.68 (dt, *J* = 9.8, 2.7 Hz, 1H), 2.86–2.75 (m, 2H), 2.37–2.06 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.6, 146.1 (d, *J* = 2.4 Hz), 145.9 (d, *J* = 2.5 Hz), 140.9, 138.5, 138.1, 138.0, 137.9, 130.4, 129.2, 128.7, 128.6, 128.6, 128.5, 128.1(4), 128.1, 128.0(3), 128.0, 127.9, 127.8, 126.3, 125.4(5), 125.4, 94.8, 93.0 (d, *J* = 173.3 Hz), 85.0, 81.0, 77.3, 75.8, 75.7, 75.2, 73.7, 68.2, 39.0 (d, *J* = 25.1 Hz), 31.2 (d, *J*= 3.2 Hz), 29.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.75; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{50}H_{49}FO_7Na$ = 803.3355, found: 803.3347.

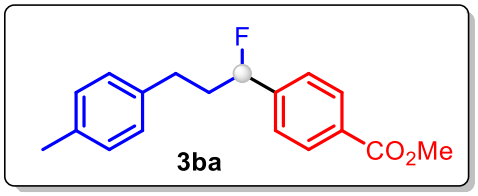
Tert-butyl 2-((4*S*,6*S*)-6-(2-(4-(1-fluoro-3-phenylpropyl) benzamido) ethyl)-2,2-dimethyl-1,3-dioxan-4-yl) acetate



The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (57.2 mg, 0.2 mmol) and *tert*-butyl 2-((4*S*,6*S*)-6-(2-(4-iodobenzamido)ethyl)-2,2-dimethyl-1,3-dioxan-4-yl)acetate (**2t**) (220.8 mg, 0.4 mmol) purification by column chromatography on silica gel, yielding **3at** (43.0 mg, 42%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.32–7.27(m, 2H), 7.22–7.19 (m, 3H), 5.47 (ddd, *J* = 47.8, 8.7, 4.1 Hz, 1H), 4.32–4.26 (m, 1H), 4.11 (t, *J* = 10.0 Hz, 1H), 3.79–3.71 (m, 1H), 3.46–3.39 (m, 1H), 2.84–2.71 (m, 2H), 2.44 (dd, *J* = 15.2, 7.0 Hz, 1H), 2.32 (dd, *J* = 15.4, 6.2 Hz, 1H), 2.26–2.08 (m, 2H), 1.85–1.83 (m, 1H), 1.79–1.66 (m, 1H), 1.58 (d, *J* = 13.0 Hz, 1H), 1.48 (s, 3H), 1.45 (s, 9H), 1.42 (s, 3H), 1.38–1.26 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 166.6, 143.6 (d, *J* = 19.8 Hz), 140.9, 134.7, 128.6 (d, *J* = 7.4 Hz), 127.1, 126.2, 125.6 (d, *J* = 7.2 Hz), 99.0, 93.1 (d, *J* = 172.3 Hz), 80.8, 69.9, 66.3, 42.6, 39.0 (d, *J* = 23.3 Hz), 38.1, 36.1, 34.8, 31.3 (d, *J* = 4.1 Hz), 30.4, 28.2, 20.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ -179.09; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{30}H_{41}FNO_5$ = 514.2963, found: 514.2964.

Methyl 4-(1-fluoro-3-(p-tolyl) propyl) benzoate

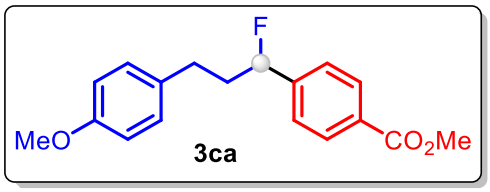


The general **procedure C** was followed using monofluoroalkyl triflate (**1b**) (120.0 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ba** (77.0 mg, 68%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.1 Hz, 2H), 7.41 (d, *J* = 8.1 Hz, 2H), 7.15–7.11 (m, 4H), 5.51 (ddd, *J* = 47.8, 8.6, 4.1 Hz, 1H), 3.94 (s, 3H), 2.84–2.71 (m, 2H), 2.35 (s, 3H), 2.35– 2.04 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 145.4 (d, *J* = 19.8 Hz), 137.8, 135.7, 130.0, 129.8, 129.3, 128.4, 125.3 (d, *J* = 7.3 Hz), 93.0 (d, *J* =

172.7 Hz), 52.2, 39.1 (d, J = 23.2 Hz), 30.7 (d, J = 4.1 Hz), 21.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.12; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{18}H_{20}FO_2$ = 287.1442, found: 287.1442.

Methyl 4-(1-fluoro-3-(4-methoxyphenyl) propyl) benzoate

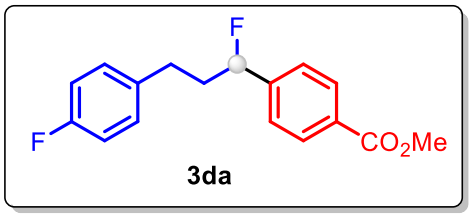


3ca

The general **procedure C** was followed using monooroalkyl triflate (**1c**) (126.5 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ca** (72.0 mg, 60%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, J = 7.7 Hz, 2H), 7.39 (d, J = 8.1 Hz, 2H), 7.12 (d, J = 8.6 Hz, 2H), 6.87–6.83 (m, 2H), 5.48 (ddd, J = 47.8, 8.6, 4.1 Hz, 1H), 3.92 (s, 3H), 3.79 (s, 3H), 2.79–2.67 (m, 2H), 2.31–2.00 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 158.1, 145.4 (d, J = 19.9 Hz), 132.9, 130.0, 129.9, 129.5, 125.3 (d, J = 7.2 Hz), 114.0, 93.0 (d, J = 172.7 Hz), 55.3, 52.3 39.2 (d, J = 23.1 Hz), 30.3 (d, J = 4.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.25; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{18}H_{20}FO_3$ = 303.1391, found: 303.1399.

Methyl 4-(1-fluoro-3-(4-fluorophenyl) propyl) benzoate



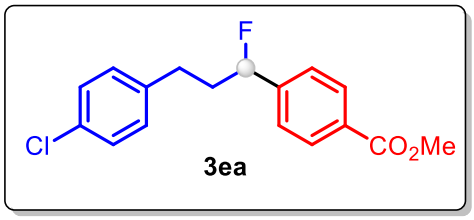
3da

The general **procedure C** was followed using monofluoroalkyl triflate (**1d**) (120.0 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3da** (64.0 mg, 55%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, J = 8.1 Hz, 2H), 7.39 (d, J = 8.1 Hz, 2H), 7.17–7.12 (m, 2H), 7.01–6.95 (m, 2H), 5.48 (ddd, J = 47.8, 8.6, 4.1 Hz, 1H), 3.92 (s, 3H), 2.82–2.70 (m, 2H), 2.31–2.01 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 162.7, 160.3, 145.2 (d, J = 19.7 Hz), 136.5 (d, J = 3.2 Hz), 130.1, 129.9 (t, J = 3.9 Hz), 125.3 (d, J = 7.3 Hz), 115.4 (d, J = 21.2 Hz), 92.9 (d, J = 173.0 Hz), 52.3, 39.1 (d, J = 23.3 Hz), 30.4 (d, J = 4.0 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -117.11, -180.50; HRMS (ESI)

m/z: $[M+H]^+$ calcd. for $C_{17}H_{17}F_2O_2$ = 291.1191, found: 291.1199.

Methyl 4-(3-(4-chlorophenyl)-1-fluoropropyl) benzoate

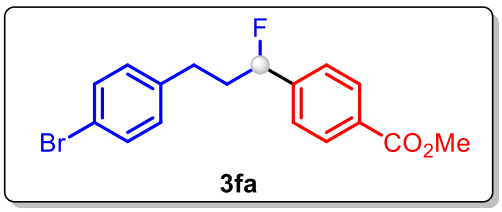


3ea

The general **procedure C** was followed using monofluoroalkyl triflate (**1e**) (128.0 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ea** (71.0 mg, 58%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.1 Hz, 2H), 7.37 (d, *J* = 8.1 Hz, 2H), 7.27 –7.23 (m, 2H), 7.11 (d, *J* = 8.4 Hz, 2H), 5.46 (ddd, *J* = 47.8, 8.6, 4.0 Hz, 1H), 3.91 (s, 3H), 2.80–2.68 (m, 2H), 2.29–1.99 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.7, 145.1 (d, *J* = 19.8 Hz), 139.3, 132.0, 130.1, 129.9, 129.8,128.7, 125.3 (d, *J* = 7.4 Hz), 92.8 (d, *J* = 173.2 Hz), 52.3, 38.8 (d, *J* = 23.3 Hz), 30.6 (d, *J* = 4.0 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.51; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. $C_{17}H_{17}ClFO_2$ = 307.0896, found: 307.0867.

Methyl 4-(3-(4-bromophenyl)-1-fluoropropyl) benzoate

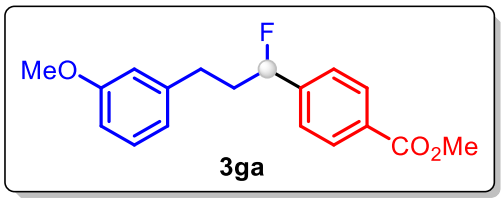


3fa

The general **procedure C** was followed using monofluoroalkyl triflate (**1f**) (146.0 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3fa** (70.0 mg, 50%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.0 Hz, 2H), 7.43–7.37 (m, 4H), 7.07 (d, *J* = 8.4 Hz, 2H), 5.47 (ddd, *J* = 47.8, 8.6, 4.0 Hz, 1H), 3.92 (s, 3H), 2.80–2.68 (m, 2H), 2.30–2.00 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 145.1 (d, *J* = 19.7 Hz), 139.8, 131.7, 130.3, 130.2 (d, *J* = 1.6 Hz), 129.9, 125.3 (d, *J* = 7.3 Hz), 120.0, 92.8 (d, *J* = 173.2 Hz), 52.3, 38.8 (d, *J* = 23.3 Hz), 30.6 (d, *J* = 4.0 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.52; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{17}H_{17}BrFO_2$ = 373.0210, found: 373.0240.

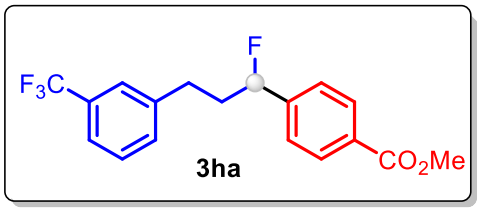
Methyl 4-(1-fluoro-3-(3-methoxyphenyl) propyl) benzoate



The general **procedure C** was followed using monofluoroalkyl triflate (**1g**) (126.5 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ga** (87.0 mg, 73%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.1 Hz, 2H), 7.38 (d, *J* = 8.1 Hz, 2H), 7.24–7.19 (m, 1H), 6.80–6.74 (m, 3H), 5.48 (ddd, *J* = 47.8, 8.6, 4.1 Hz, 1H), 3.91 (s, 3H), 3.78 (s, 3H), 2.81–2.69 (m, 2H), 2.32–2.03 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 159.8, 145.3 (d, *J* = 19.9 Hz), 142.5, 130.0, 129.9, 129.6, 125.3 (d, *J* = 7.3 Hz), 120.9, 114.3, 111.5, 93.0 (d, *J* = 172.9 Hz), 55.2, 52.2, 38.8 (d, *J* = 23.3 Hz), 31.2 (d, *J* = 3.9 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.20; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{18}H_{19}FO_3Na$ = 325.1210, found: 325.1240.

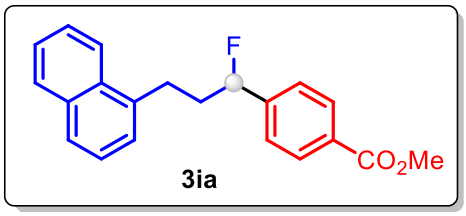
Methyl 4-(1-fluoro-3-(3-(trifluoromethyl)phenyl) propyl) benzoate



The general **procedure C** was followed using monofluoroalkyl triflate (**1h**) (141.7 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ha** (80.0 mg, 60%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.0 Hz, 2H), 7.51–7.35 (m, 6H), 5.51 (ddd, *J* = 47.8, 8.5, 4.1 Hz, 1H), 3.92 (s, 3H), 2.92–2.79 (m, 2H), 2.35–2.07 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 144.9 (d, *J* = 19.7 Hz), 141.8, 132.0, 131.0 (q, *J* = 31.8 Hz), 130.2, 130.0, 129.1, 125.6, 125.3 (dd, *J* = 8.5, 5.6 Hz), 123.2 (q, *J* = 3.8 Hz), 122.9, 92.9 (d, *J* = 173.5 Hz), 52.3, 38.7 (d, *J* = 23.5 Hz), 31.1 (d, *J* = 4.0 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.56, -180.53; HRMS (ESI) *m/z*: $[M+K]^+$ calcd. for $C_{18}H_{16}F_4O_2K$ = 309.0718, found: 309.0723.

Methyl 4-(1-fluoro-3-(naphthalen-1-yl) propyl) benzoate

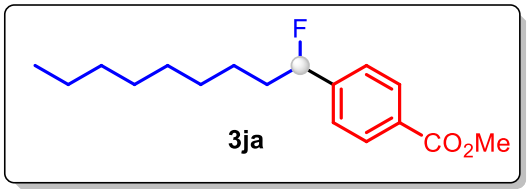


3ia

The general **procedure C** was followed using monofluoroalkyl triflate (**1i**) (134.5 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ia** (51.0 mg, 40%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.1 Hz, 2H), 7.99 (d, *J* = 7.7 Hz, 1H), 7.88 (dd, *J* = 7.4, 2.0 Hz, 1H), 7.75 (d, *J* = 8.7 Hz, 1H), 7.55–7.47 (m, 2H), 7.43–7.35 (m, 4H), 5.61 (ddd, *J* = 47.9, 8.5, 3.9 Hz, 1H), 3.93 (s, 3H), 3.36–3.17 (m, 2H), 2.47–2.19 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 145.2 (d, *J* = 19.9 Hz), 137.8, 137.0, 134.1, 131.8, 131.1, 130.1, 129.9, 129.0, 127.2, 126.2 (d, *J* = 20.8 Hz), 125.7 (d, *J* = 3.0 Hz), 125.4 (d, *J* = 7.4 Hz), 123.6, 93.3 (d, *J* = 173.1 Hz), 52.3, 38.3 (d, *J* = 23.2 Hz), 28.3 (d, *J* = 3.8 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.37; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{21}H_{19}FO_2Na$ = 345.1261, found: 345.1245.

Methyl 4-(1-fluorononyl) benzoate

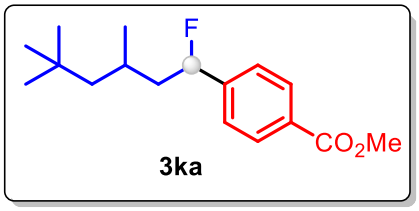


3ja

The general **procedure C** was followed using monofluoroalkyl triflate (**1j**) (117.7 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ja** (52.0 mg, 46%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.0 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 2H), 5.48 (ddd, *J* = 47.8, 8.0, 4.7 Hz, 1H), 3.92 (s, 3H), 1.99–1.72 (m, 2H), 1.49–1.25 (m, 12H), 0.87 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.9, 145.8 (d, *J* = 19.9 Hz), 129.9, 129.8, 125.4 (d, *J* = 7.3 Hz), 94.1 (d, *J* = 172.1 Hz), 52.3, 37.4 (d, *J* = 23.1 Hz), 32.0, 29.5, 29.4, 29.3, 25.0 (d, *J* = 4.1 Hz), 22.8, 14.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ -178.35; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{17}H_{26}FO_2$ = 281.1911, found: 281.1907.

Methyl 4-(1-fluoro-3,5,5-trimethylhexyl) benzoate

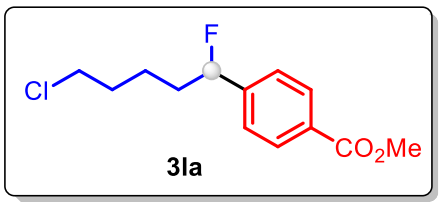


3ka

The general **procedure C** was followed using monofluoroalkyl triflate (**1k**) (117.7 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ka** (59.0 mg, 53%, *dr* = 1.5:1) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.2 Hz, 2H), 7.38 (d, *J* = 8.2 Hz, 2H), 5.53 (dddd, *J* = 48.3, 16.7, 9.5, 3.5 Hz, 1H) (major + minor), 3.92 (s, 3H), 2.04–1.79 (m, 1H), 1.74–1.59 (m, 1H), 1.57–1.32 (m, 1H), 1.17 (dtd, *J* = 29.8, 15.6, 14.8, 5.3 Hz, 2H), 1.03 (dd, *J* = 16.0, 6.5 Hz, 3H), 0.89 (d, *J* = 10.8 Hz, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.9, 146.3 (d, *J* = 19.7 Hz) (major), 146.0 (d, *J* = 19.7 Hz) (minor), 129.9, 125.4 (d, *J* = 7.1 Hz) (minor), 125.2 (d, *J* = 7.3 Hz) (major), 92.8 (d, *J* = 171.6 Hz) (minor), 92.4 (d, *J* = 171.9 Hz) (major), 52.3 (major), 51.8 (minor), 50.8, 47.5 (d, *J* = 22.7 Hz) (major), 47.2 (d, *J* = 22.4 Hz) (minor), 31.4 (major), 31.2 (minor), 30.1 (d, *J* = 6.2 Hz), 26.1 (d, *J* = 3.3 Hz) (minor), 25.8 (d, *J* = 2.1 Hz) (major), 23.6 (minor), 22.1 (major); ^{19}F NMR (376 MHz, $CDCl_3$) δ -177.04, -180.48; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{17}H_{25}FO_2Na$ = 303.1731, found: 303.1718.

Methyl 4-(5-chloro-1-fluoropentyl) benzoate



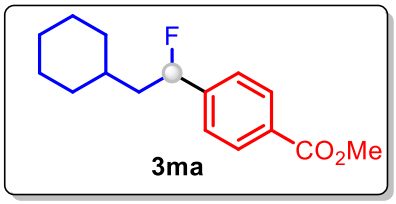
3la

The general **procedure C** was followed using monofluoroalkyl triflate (**1l**) (111.3 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3la** (54.0 mg, 53%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.0 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 2H), 5.49 (ddd, *J* = 47.8, 8.1, 4.6 Hz, 1H), 3.92 (s, 3H), 3.53 (t, *J* = 6.6 Hz, 2H), 2.03–1.86 (m, 2H), 1.85–1.78 (m, 2H), 1.69–1.49 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 145.3 (d, *J* = 20.1 Hz), 130.1, 129.9, 125.3 (d, *J* = 7.4 Hz), 93.7 (d, *J* = 173.0 Hz), 52.3, 44.7, 36.6 (d, *J* = 23.2 Hz), 32.3, 22.5 (d, *J* = 4.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -

178.98; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{13}H_{17}ClFO_2$ = 259.0896, found: 259.0890.

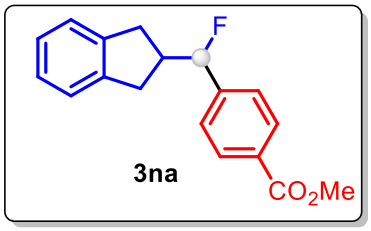
Methyl 4-(2-cyclohexyl-1-fluoroethyl) benzoate



The general **procedure C** was followed using monofluoroalkyl triflate (**1m**) (111.3 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3ma** (77.0 mg, 69%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 8.1 Hz, 2H), 7.38 (d, *J* = 8.1 Hz, 2H), 5.58 (ddd, *J* = 48.2, 9.5, 3.7 Hz, 1H), 3.91 (s, 3H), 1.93–1.81 (m, 2H), 1.74–1.62 (m, 4H), 1.60–1.46 (m, 2H), 1.31–1.10 (m, 3H), 1.04–0.91 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 146.2 (d, *J* = 19.7 Hz), 129.8, 125.3 (d, *J* = 7.1 Hz), 92.2 (d, *J* = 171.7 Hz), 52.2, 45.3 (d, *J* = 22.7 Hz), 34.1 (d, *J* = 2.9 Hz), 34.0, 32.8, 26.5, 26.2 (d, *J* = 16.2 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -177.89; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{16}H_{21}FO_2Na$ = 287.1481, found: 287.1415.

Methyl 4-((2,3-dihydro-1*H*-inden-2-yl) fluoromethyl) benzoate

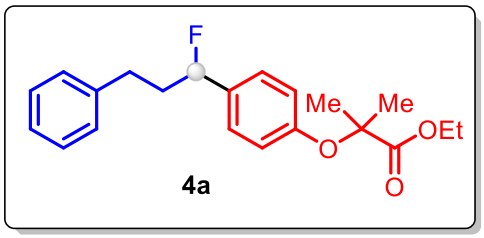


The general **procedure C** was followed using monofluoroalkyl triflate (**1n**) (111.3 mg, 0.4 mmol) and methyl 4-iodobenzoate (**2a**) (209.6 mg, 0.8 mmol) purification by column chromatography on silica gel, yielding **3na** (60.0 mg, 56%) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, *J* = 7.6 Hz, 2H), 7.46 (d, *J* = 7.6 Hz, 2H), 7.24 –7.22(m, 1H), 7.19–7.14 (m, 3H), 5.45 (dd, *J* = 47.4, 7.4 Hz, 1H), 3.95 (s, 3H), 3.09 (d, *J* = 6.7 Hz, 2H), 2.99 (td, *J* = 15.7, 7.3 Hz, 1H), 2.85–2.73 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 144.4 (d, *J* = 20.1 Hz), 142.1 (d, *J* = 44.1 Hz), 130.3, 129.9, 126.6 (d, *J* = 12.1 Hz), 126.1 (d, *J* = 6.7 Hz), 124.6 (d, *J* = 26.0 Hz), 96.3 (d, *J* = 174.4 Hz), 52.3, 45.8 (d, *J* = 22.8 Hz), 35.2 (d, *J* = 2.9 Hz), 35.1 (d, *J* = 7.2 Hz); ^{19}F NMR

(376 MHz, $CDCl_3$) δ -177.49; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{18}H_{17}FO_2Na$ = 307.1105, found: 307.1124.

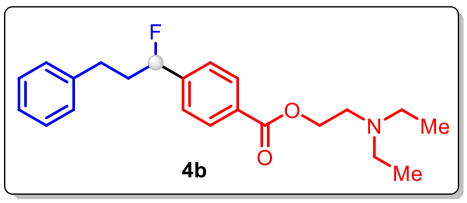
Ethyl 2-(4-(1-fluoro-3-phenylpropyl) phenoxy)-2-methylpropanoate



The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (57.2 mg, 0.2 mmol) and ethyl 2-(4-iodophenoxy)-2-methylpropanoate (**2u**) (133.6 mg, 0.4 mmol) purification by column chromatography on silica gel, yielding **4a** (27.5 mg, 40%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31–7.26 (m, 2H), 7.22–7.18 (m, 5H), 6.83 (d, *J* = 8.5 Hz, 2H), 5.36 (ddd, *J* = 47.8, 8.5, 4.5 Hz, 1H), 4.23 (q, *J* = 7.1 Hz, 2H), 2.83–2.67 (m, 2H), 2.37–2.23 (m, 1H), 2.16–2.00 (m, 1H), 1.60 (s, 6H), 1.24 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.3, 155.7, 141.3, 133.7, 133.5, 128.6, 127.0 (d, *J* = 6.1 Hz), 126.2, 118.9, 93.6 (d, *J* = 169.6 Hz), 79.2, 61.6, 38.8, 38.6, 31.6 (d, *J* = 4.4 Hz), 25.5, 14.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ -171.50; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{21}H_{25}FO_3Na$ = 367.1680, found: 367.1673.

2-(diethylamino) ethyl 4-(1-fluoro-3-phenylpropyl) benzoate

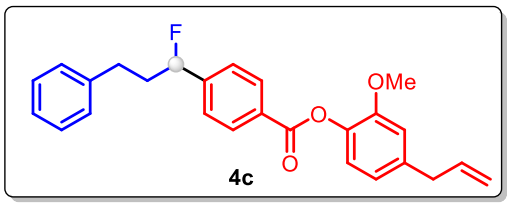


The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (57.2 mg, 0.2 mmol) and 2-(diethylamino)ethyl 4-iodobenzoate (**2v**) (138.8 mg, 0.4 mmol) purification by column chromatography on silica gel, yielding **4b** (32.8 mg, 46%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.32–7.28 (m, 2H), 7.22–7.19 (m, 3H), 5.49 (ddd, *J* = 47.9, 8.6, 4.2 Hz, 1H), 4.40 (t, *J* = 6.2 Hz, 2H), 2.86 (t, *J* = 6.2 Hz, 2H), 2.81–2.72 (m, 2H), 2.64 (q, *J* = 7.1 Hz, 4H), 2.32–2.06 (m, 2H), 1.07 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.3, 145.4, 145.2, 140.9, 130.2, 129.9, 128.6 (d, *J* = 8.5 Hz), 126.3, 125.4 (d, *J* = 7.3 Hz), 93.0 (d,

J = 172.9 Hz), 63.6, 51.1, 48.0, 39.0 (d, *J* = 23.3 Hz), 31.2 (d, *J* = 3.9 Hz), 12.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.20; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{22}H_{29}FNO_2$ = 358.2117, found: 358.2113.

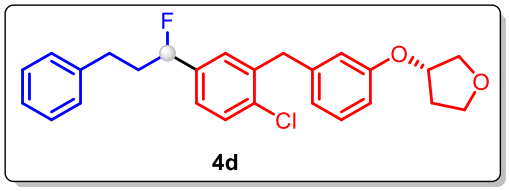
4-allyl-2-methoxyphenyl 4-(1-fluoro-3-phenylpropyl) benzoate



The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (57.2 mg, 0.2 mmol) and 4-allyl-2-methoxyphenyl 4-iodobenzoate (**2w**) (157.7 mg, 0.4 mmol) purification by column chromatography on silica gel, yielding **4c** (51.0 mg, 64%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 8.0 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 2H), 7.32–7.28 (m, 2H), 7.21 (dd, *J* = 7.3, 4.1 Hz, 3H), 7.06 (d, *J* = 7.9 Hz, 1H), 6.83–6.80 (m, 2H), 5.98 (ddt, *J* = 16.8, 10.0, 6.7 Hz, 1H), 5.52 (ddd, *J* = 47.8, 8.5, 4.1 Hz, 1H), 5.14–5.08 (m, 2H), 3.79 (s, 3H), 3.40 (d, *J* = 6.7 Hz, 2H), 2.86–2.73 (m, 2H), 2.35–2.06 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.6, 151.1, 145.9 (d, *J* = 19.9 Hz), 140.9, 139.2, 138.2, 137.2, 130.6, 129.4, 128.6 (d, *J* = 9.1 Hz), 126.3, 125.4 (d, *J* = 7.3 Hz), 122.7, 120.8, 116.3, 112.9, 93.0 (d, *J* = 173.0 Hz), 56.0, 40.2, 39.0 (d, *J* = 23.2 Hz), 31.2 (d, *J* = 4.0 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.44; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{26}H_{26}FO_3$ = 405.1860, found: 405.1855.

(3*S*)-3-(3-(2-chloro-5-(1-fluoro-3-phenylpropyl)benzyl)phenoxy)tetrahydrofuran

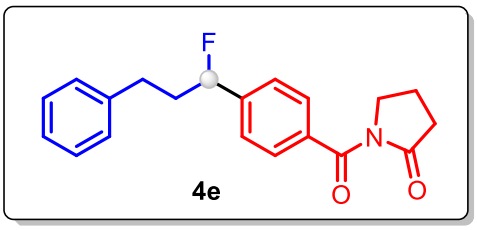


The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (57.2 mg, 0.2 mmol) and (***S***)-3-(3-(2-chloro-5-iodobenzyl)phenoxy)tetrahydrofuran (**2x**) (165.8 mg, 0.4 mmol) purification by column chromatography on silica gel, yielding **4d** (64.0 mg, 76%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.1 Hz, 1H), 7.29–7.26 (m, 2H), 7.22–7.19 (m, 1H), 7.17–7.13 (m, 3H), 7.11–7.06 (m, 3H), 6.78 (d, *J* = 8.6 Hz, 2H) 5.34 (ddd, *J*

= 47.7, 8.5, 4.4 Hz, 1H), 4.87 (dq, *J* = 8.2, 2.7 Hz, 1H), 4.03 (s, 2H), 3.99–3.93 (m, 3H), 3.87 (td, *J* = 8.0, 4.7 Hz, 1H), 2.79–2.65 (m, 2H), 2.32–1.96 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.0, 141.0, 139.6, 139.2, 139.0 (d, *J* = 20.0 Hz), 136.7, 134.1, 131.6, 130.1, 130.0, 129.8, 128.6 (d, *J* = 7.0 Hz), 128.3 (d, *J* = 6.7 Hz), 126.2, 124.9 (d, *J* = 6.8 Hz), 115.5, 93.0 (d, *J* = 171.7 Hz), 77.3, 73.2, 67.3, 38.8 (d, *J* = 23.8 Hz), 38.4, 33.1, 31.3 (d, *J* = 4.4 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -175.88; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{26}H_{26}ClFO_2Na$ = 447.1498, found: 447.1490.

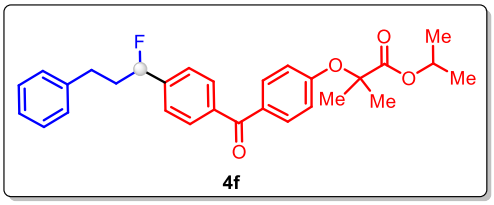
1-(4-(1-fluoro-3-phenylpropyl)benzoyl)pyrrolidin-2-one



4e

The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (57.2 mg, 0.2 mmol) and 1-(4-iodobenzoyl)pyrrolidin-2-one (**2y**) (126.0 mg, 0.4 mmol) purification by column chromatography on silica gel, yielding **4e** (33.2 mg, 51%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.0 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.32–7.28 (m, 2H), 7.21–7.19 (m, 3H), 5.48 (ddd, *J* = 47.8, 8.7, 4.0 Hz, 1H), 3.95 (t, *J* = 7.1 Hz, 2H), 2.86–2.73 (m, 2H), 2.61 (t, *J* = 8.0 Hz, 2H), 2.34–2.06 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.7, 170.4, 144.3, 144.1, 141.0, 134.2, 129.4, 128.6 (d, *J* = 4.1 Hz), 126.2, 124.8 (d, *J* = 7.3 Hz), 93.1 (d, *J* = 172.8 Hz), 46.7, 38.9 (d, *J* = 23.1 Hz), 33.4, 31.3 (d, *J* = 4.0 Hz), 17.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -180.10; HRMS (ESI) *m/z*: $[M+K]^+$ calcd. for $C_{20}H_{20}FNO_2K$ = 364.1110, found: 364.1108.

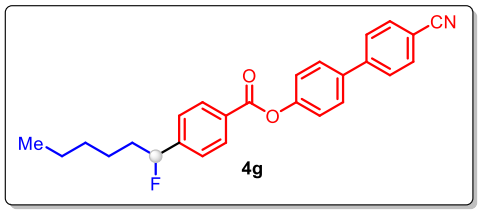
Isopropyl-2-(4-(4-(1-fluoro-3-phenylpropyl)benzoyl)phenoxy)-2-methylpropanoate



The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (57.2 mg, 0.2 mmol) and isopropyl 2-(4-(4-iodobenzoyl)phenoxy)-2-methylpropanoate (**2z**) (181.0 mg, 0.4 mmol) purification by column chromatography on silica gel, yielding **4f** (76.3 mg, 83%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.8 Hz, 4H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.32–7.25 (m, 2H), 7.22–7.19 (m, 3H), 6.89–6.85 (m, 2H), 5.51 (ddd, *J* = 47.8, 8.5, 4.1 Hz, 1H), 5.14–5.04 (m, 1H), 2.87–2.74 (m, 2H), 2.37–2.09 (m, 2H), 1.66 (s, 6H), 1.20 (d, *J* = 6.3 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.1, 173.2, 159.7, 144.4, 144.2, 140.9, 138.0, 132.1, 130.5, 130.0, 128.6 (d, *J* = 7.7 Hz), 126.2, 125.2 (d, *J* = 7.3 Hz), 117.2, 93.1 (d, *J* = 172.7 Hz), 79.4, 69.4, 39.0 (d, *J* = 23.2 Hz), 31.2 (d, *J* = 4.1 Hz), 25.4, 21.6; ^{19}F NMR (376 MHz, $CDCl_3$) δ -179.45; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{29}H_{31}FO_4Na$ = 485.2009, found: 485.2003.

4'-cyano-[1,1'-biphenyl]-4-yl 4-(1-fluorohexyl)benzoate

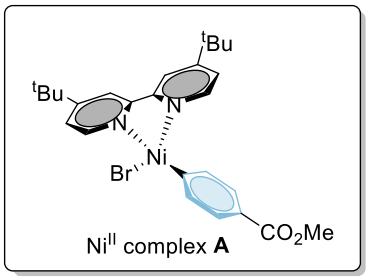


The general **procedure C** was followed using monofluoroalkyl triflate (**1a**) (57.2 mg, 0.2 mmol) and 4'-cyano-[1,1'-biphenyl]-4-yl 4-iodobenzoate (**2aa**) (170.0 mg, 0.4 mmol) purification by column chromatography on silica gel, yielding **4g** (57.2 mg, 72%) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, *J* = 8.0 Hz, 2H), 7.74 (d, *J* = 6.8 Hz, 2H), 7.69 (d, *J* = 6.8 Hz, 2H), 7.65 (d, *J* = 7.0 Hz, 2H), 7.48 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 6.8 Hz, 2H), 5.54 (ddd, *J* = 47.9, 8.2, 4.9 Hz, 1H), 2.04–1.77 (m, 2H), 1.53–1.30 (m, 6H), 0.90 (t, *J* = 6.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.9, 151.5, 146.9 (d, *J* = 19.9 Hz), 144.9, 137.1, 132.8, 130.5, 129.0, 128.6, 127.8, 125.6 (d, *J* = 7.5 Hz), 122.6, 119.0,

111.2, 94.0 (d, J = 172.8 Hz), 37.4 (d, J = 23.0 Hz), 31.6, 24.7 (d, J = 4.1 Hz), 22.6, 14.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ -178.97; HRMS (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{26}H_{25}FNO_2$ = 402.1864, found: 402.1863.

Ar-Ni(L)Br (Ni^{II} complex A)



The general **procedure D** was followed using $Ni(COD)_2$ (138 mg, 0.5 mmol, 1.0 equiv.), 4,4'-di-tert-butyl-2,2'-bipyridine (134 mg, 0.5 mmol, 1.0 equiv.) and methyl 4-bromobenzoate (1.07 g, 5 mmol, 10 equiv.) The product was used without further purification, yielding Ni^{II} complex **A** (250mg, 92%) as an orange solid.

^{1}H NMR (400 MHz, CD_2Cl_2) δ 9.25 (s, 1H), 7.89 (d, J = 12.3 Hz, 2H), 7.78 (br, 2H), 7.61–7.55 (m, 3H), 7.13 (m, 2H), 3.89 (s, 3H), 1.45 (s, 9H), 1.38 (s, 9H).

The analytical data are in accordance to those reported in the literature[6].

8. References

[1] Wu, B.-B.; Xu, J.; Gao, Q.; Bian, K.-J.; Liu, G.-K.; Wang, X.-S. *Angew. Chem. Int. Ed.* **2022**, *61*, e202208938.

[2] Lv, P.; Wang, A.; Xie, X.; Chen, Y.; Liu, Y. *Org. Lett*. **2023**, *25*, 3319−3324.

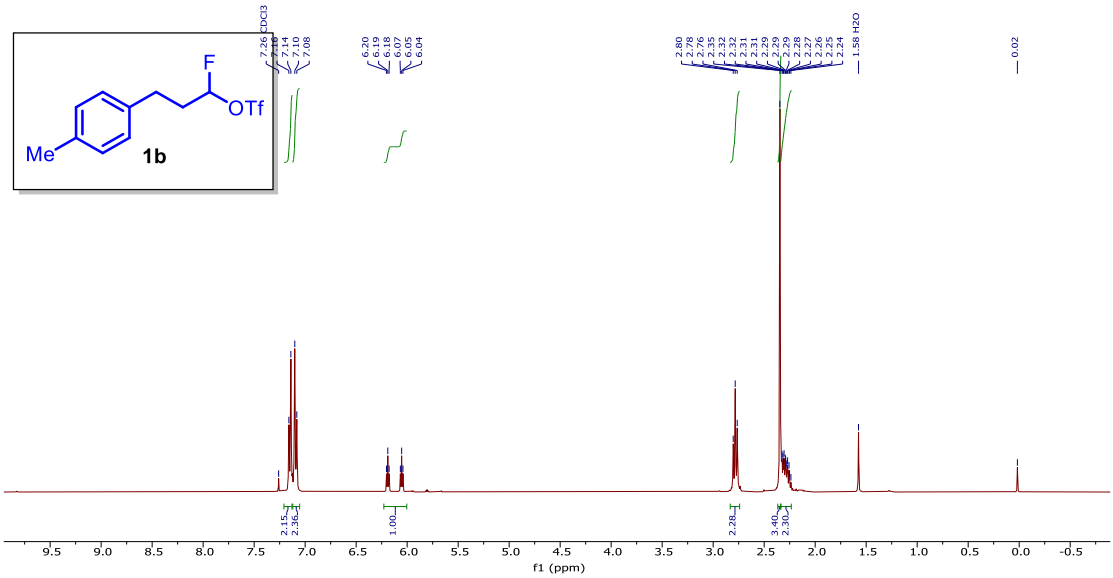
[3] a) Biswas, S.; Weix, D. J. *J. Am. Chem. Soc.* **2013**, *135*, 16192–16197. b) Sun, D.; Ma, G.; Zhao, X.; Lei, C.; Gong, H. *Chem. Sci.* **2021**, *12*, 5253–5258. c)Wang, K.; Ding, Z.; Zhou, Z.; Kong, W. *J. Am. Chem. Soc.* **2018**, *140*, 12364–12368.

[4] Xiao, Y.-L.; Min, Q.-Q.; Xu, C.; Wang, R.-W.; Zhang, X. *Angew. Chem. Int. Ed.* **2016**, *55*, 5837−5841.

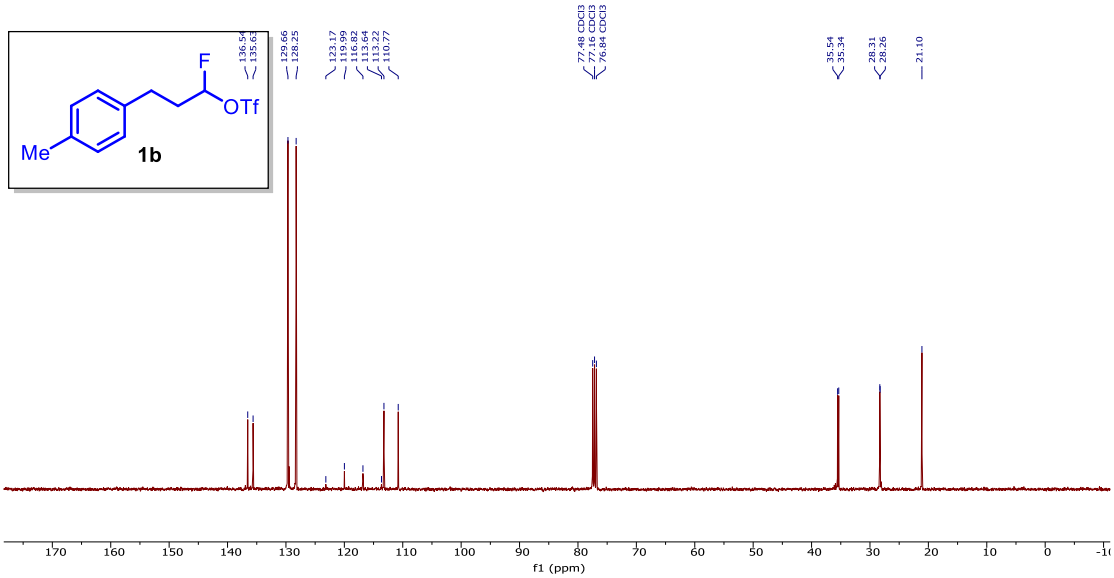
[5] Wang, H.; Liu, C.-F.; Song, Z.; Yuan, M.; Ho, Y. A.; Gutierrez, O.; Koh, M. J. *ACS Catal.* **2020**, *10*, 4451−4459.

[6] Yang, T.; Wei, Y.; Koh, M. J. *ACS Catal.* **2021**, *11*, 6519–6525.

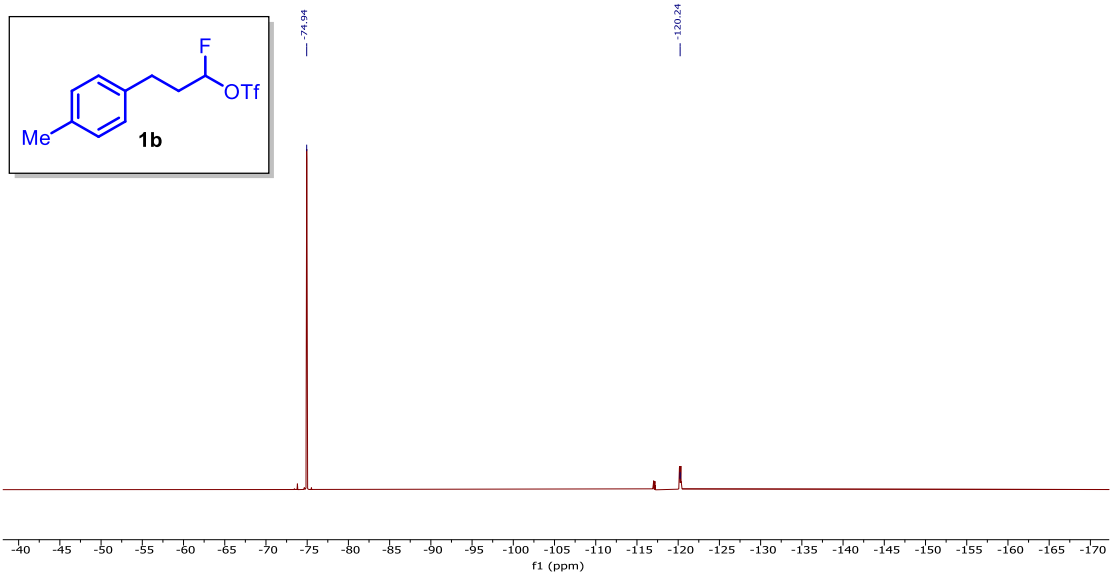
9. NMR Spectra (^{1}H NMR, ^{13}C NMR, ^{19}F NMR)



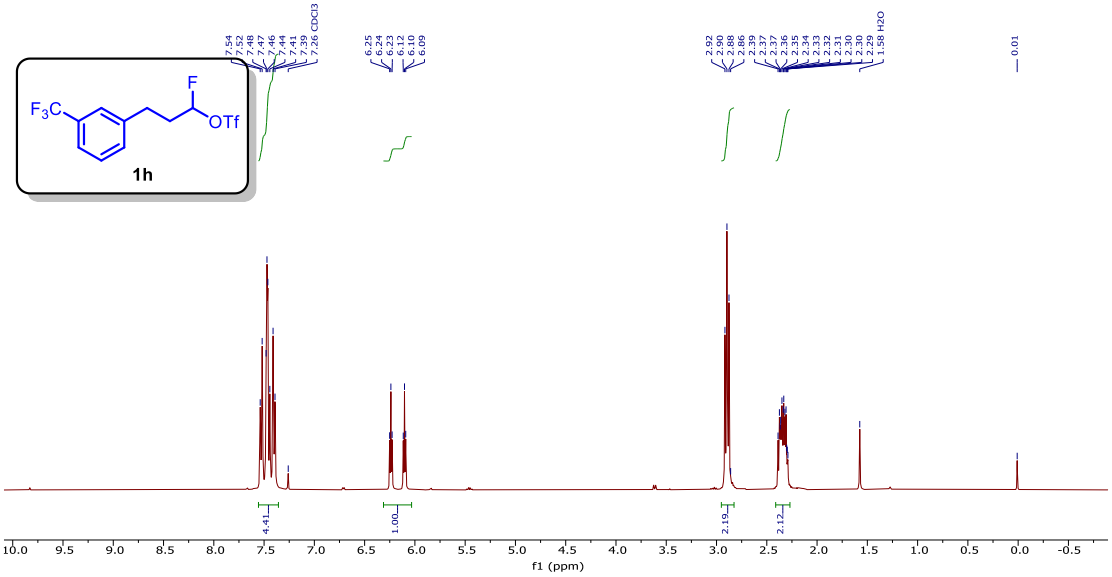
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **1b**



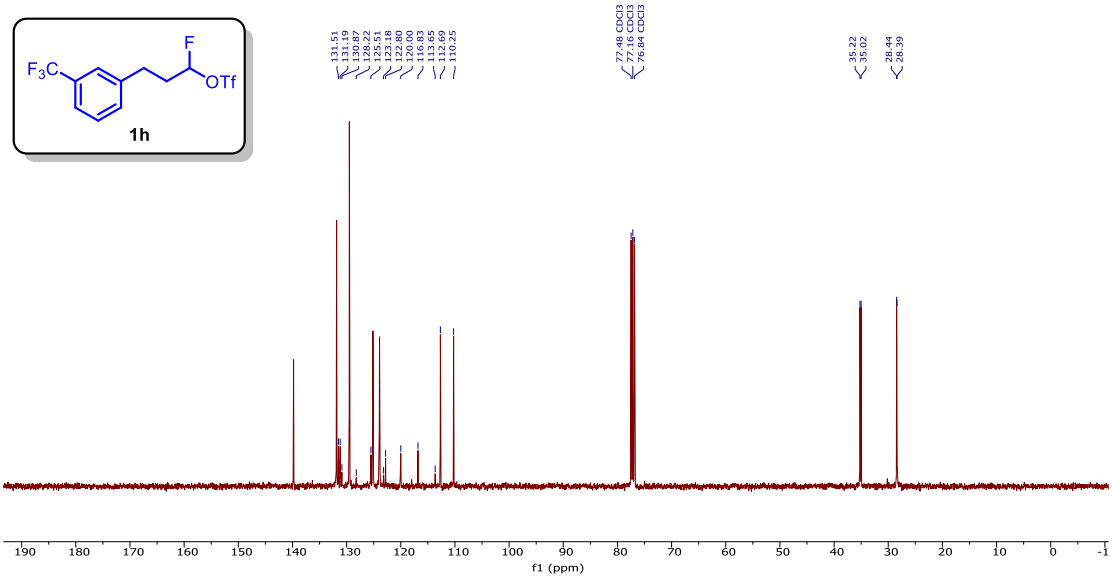
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **1b**



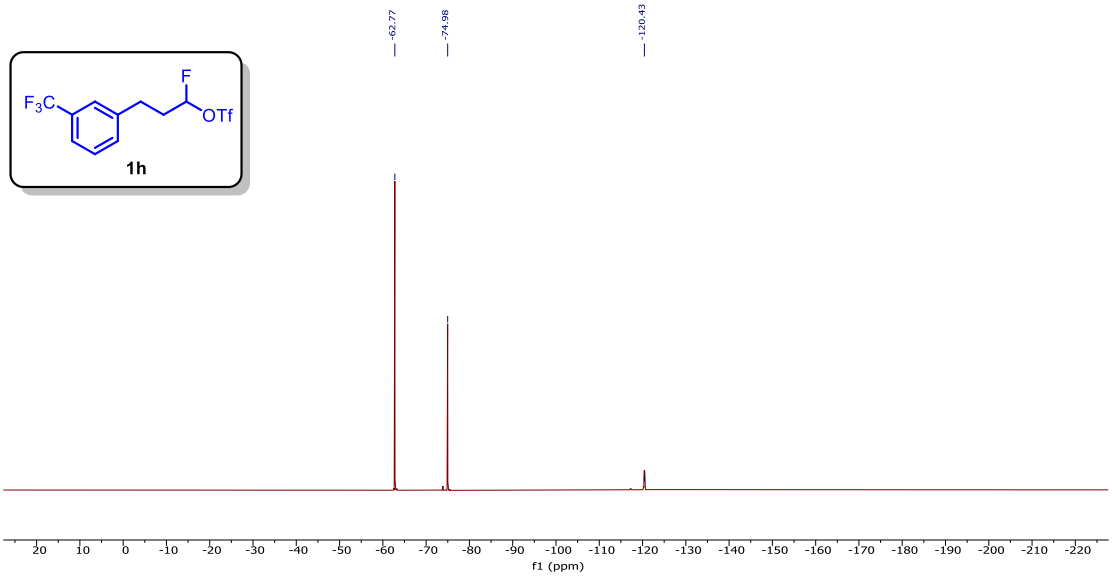
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **1b**



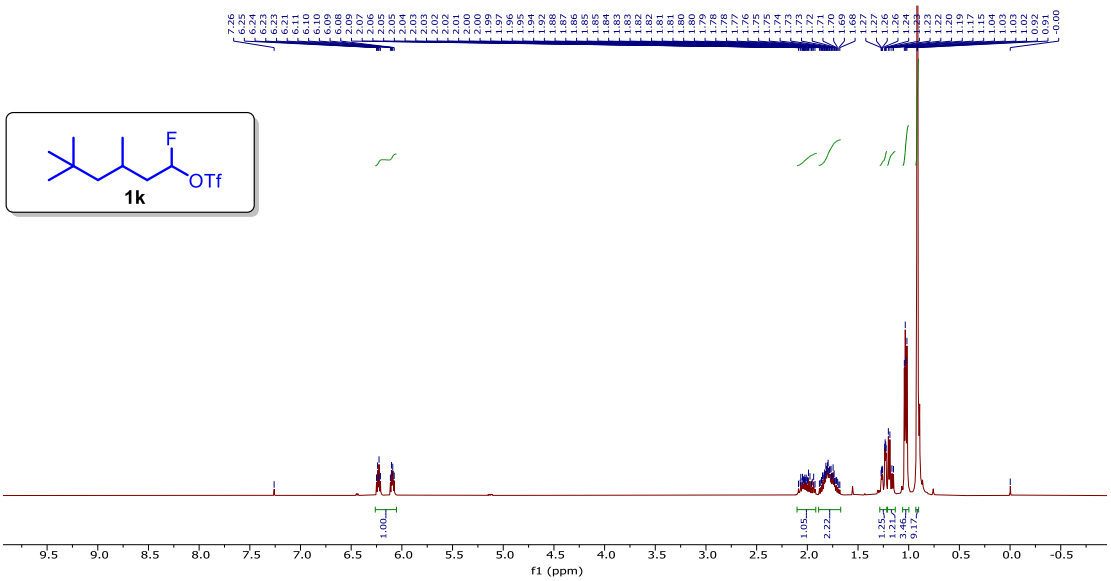
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **1h**



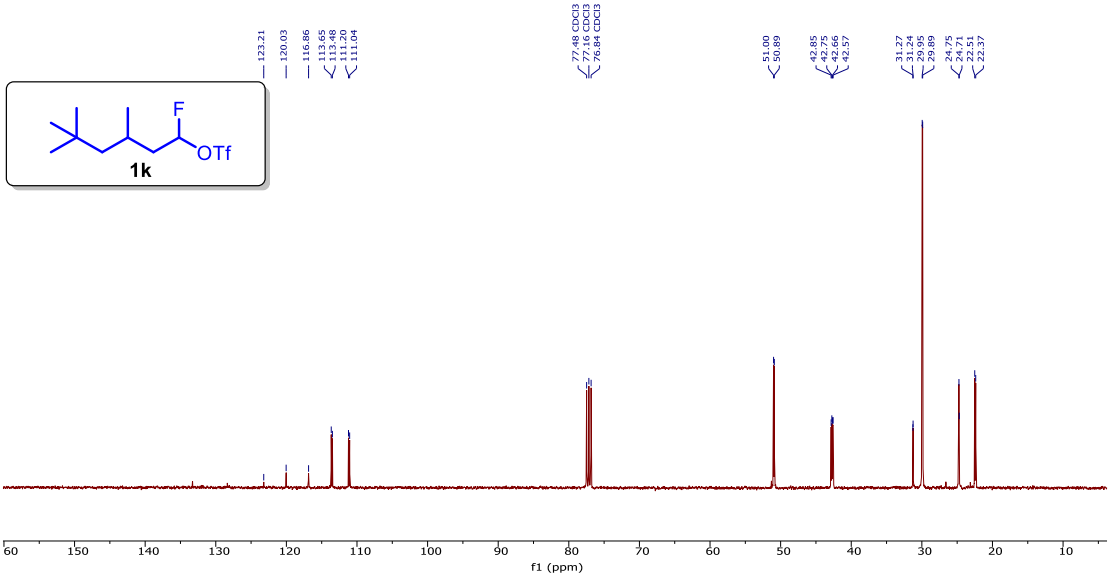
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **1h**



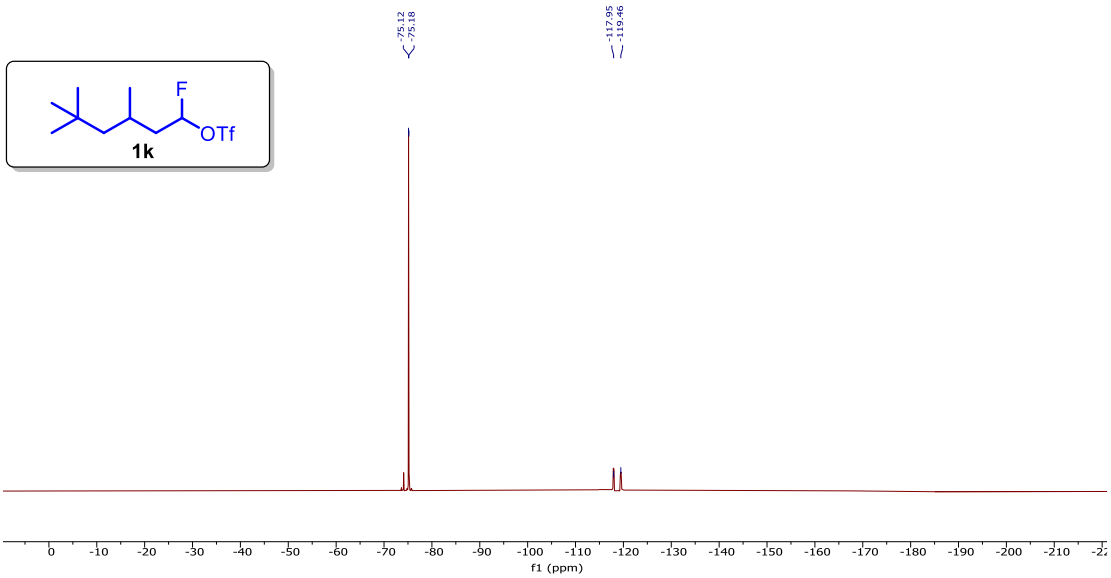
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **1h**



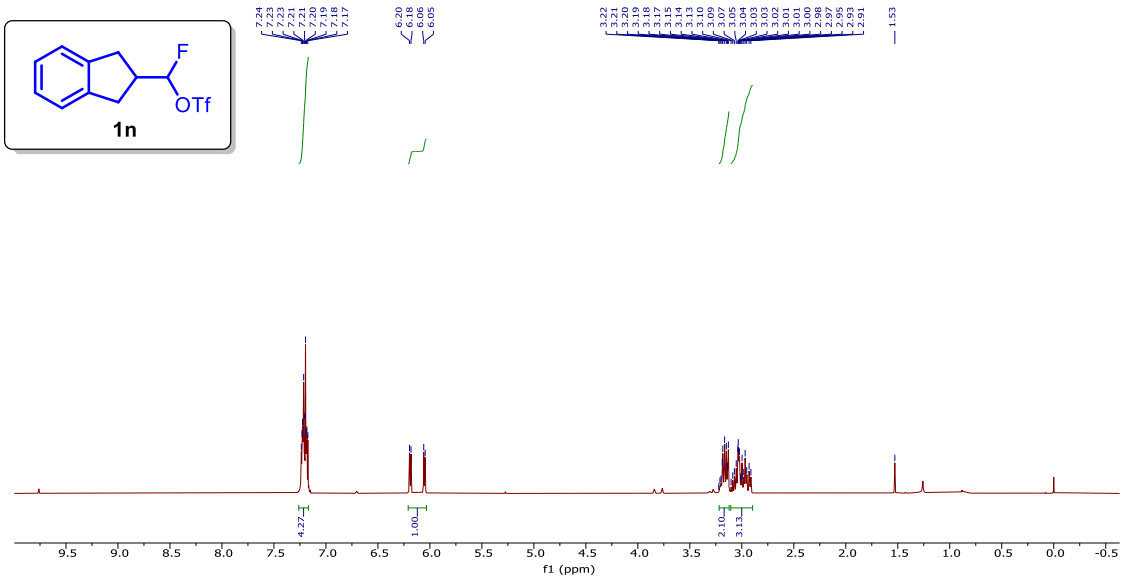
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **1k**



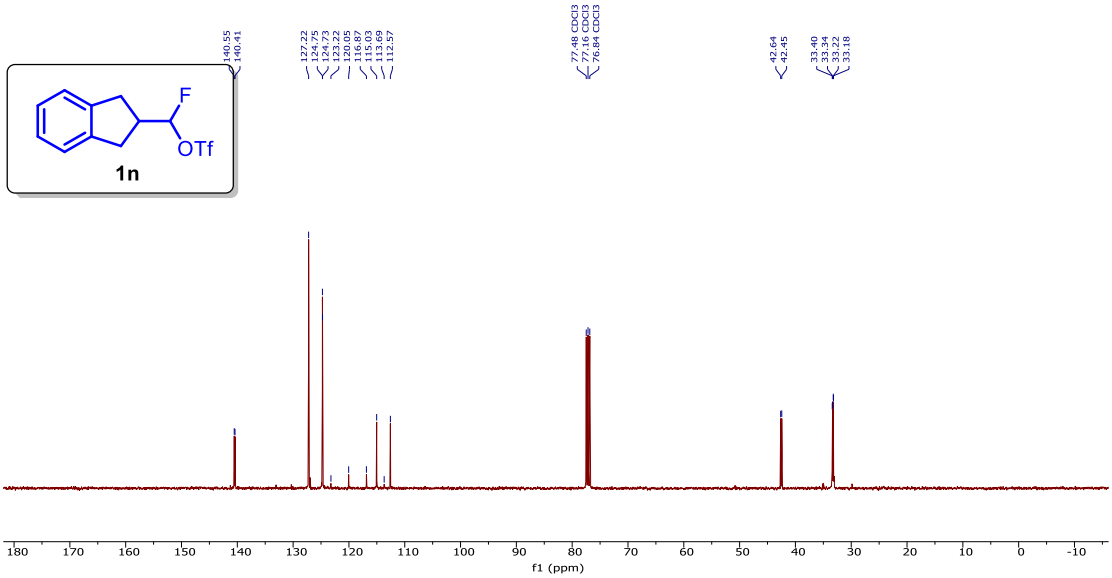
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **1k**



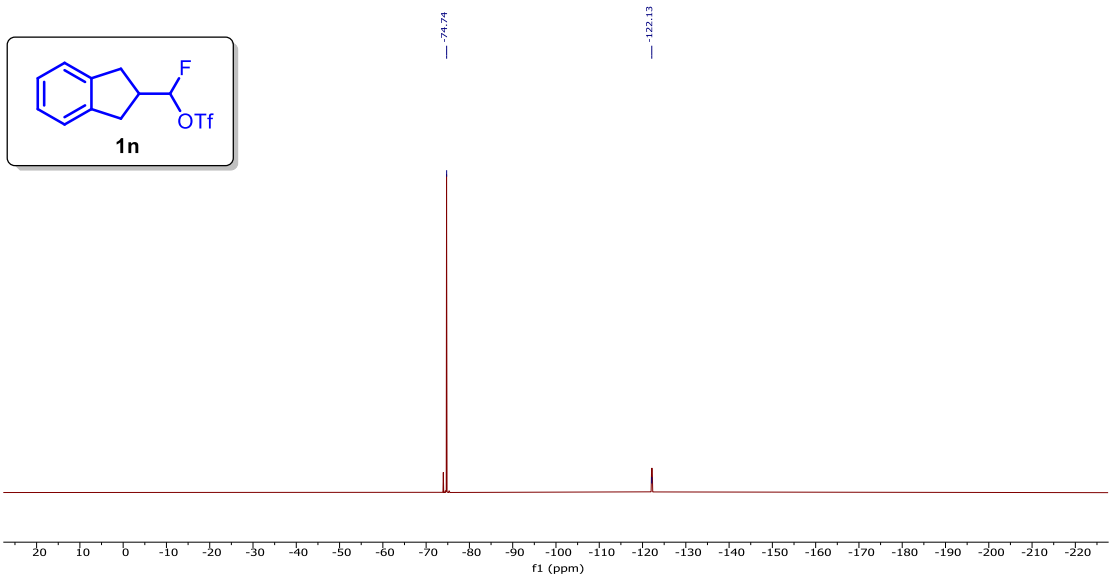
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **1k**



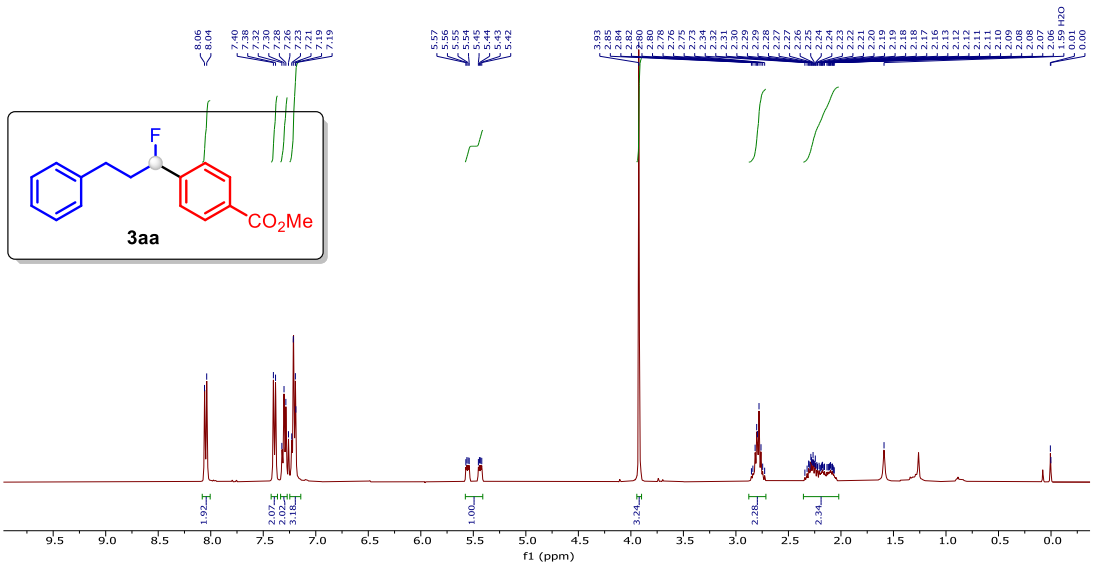
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **1n**



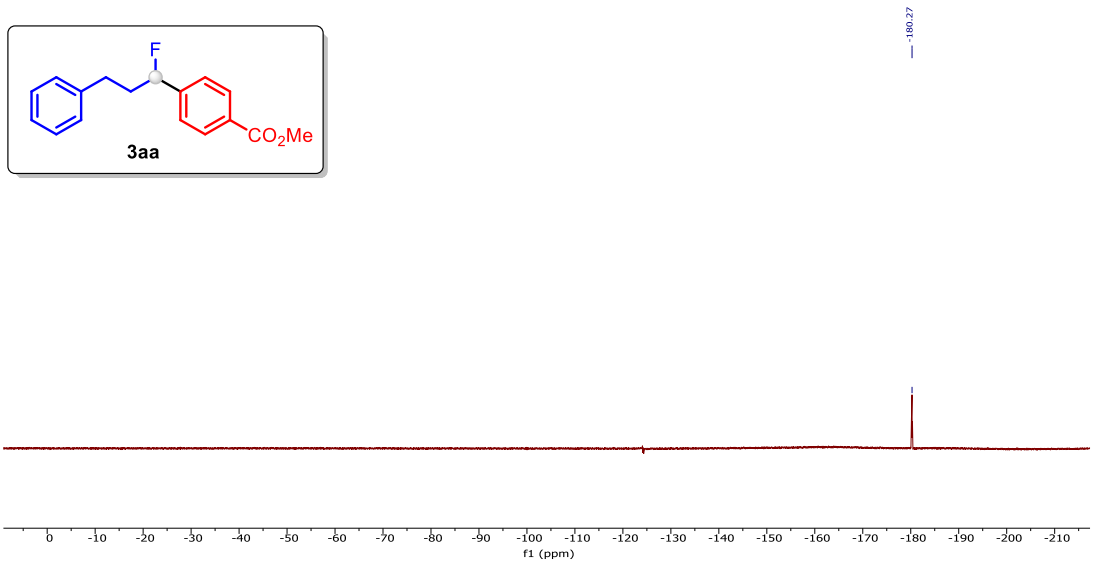
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **1n**



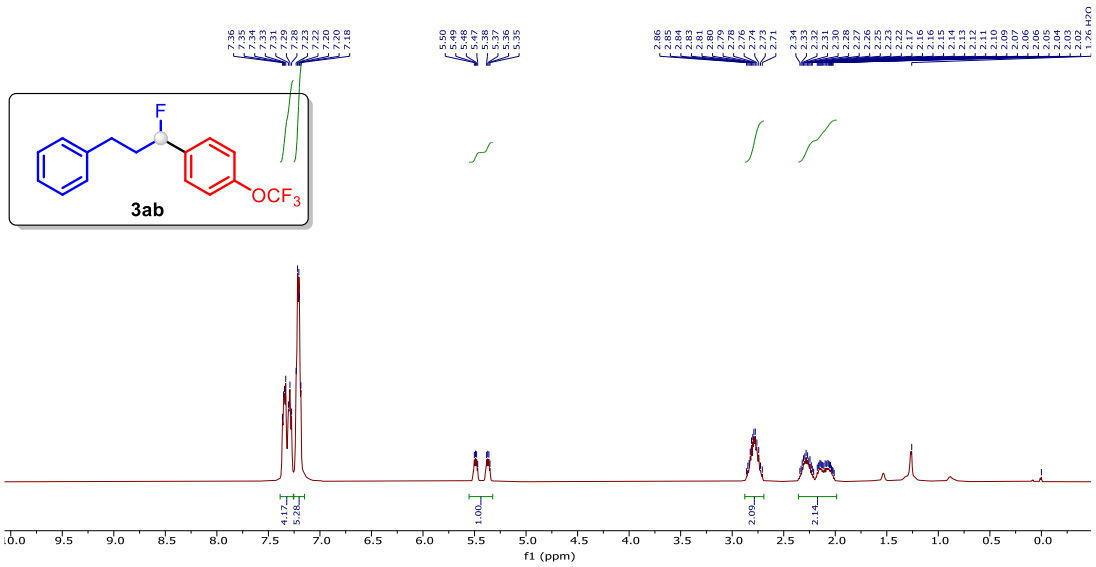
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **1n**



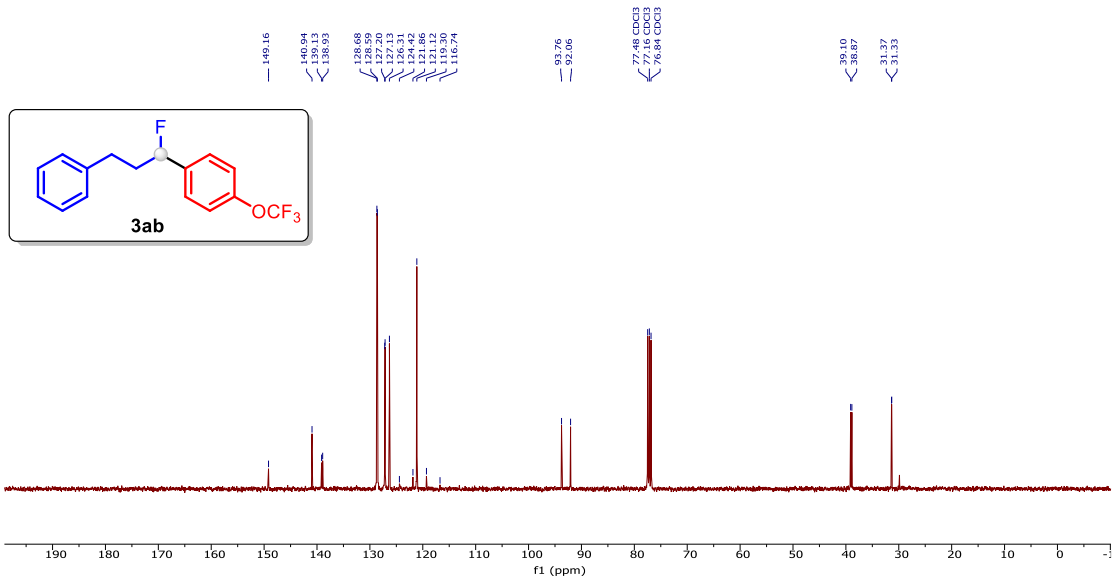
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3aa**



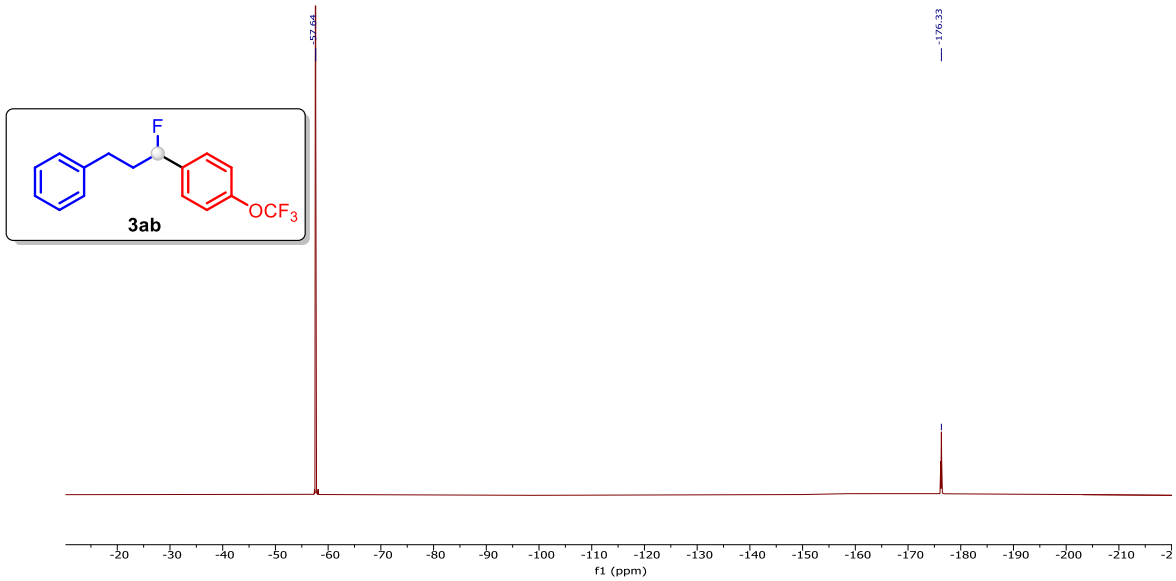
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3aa**



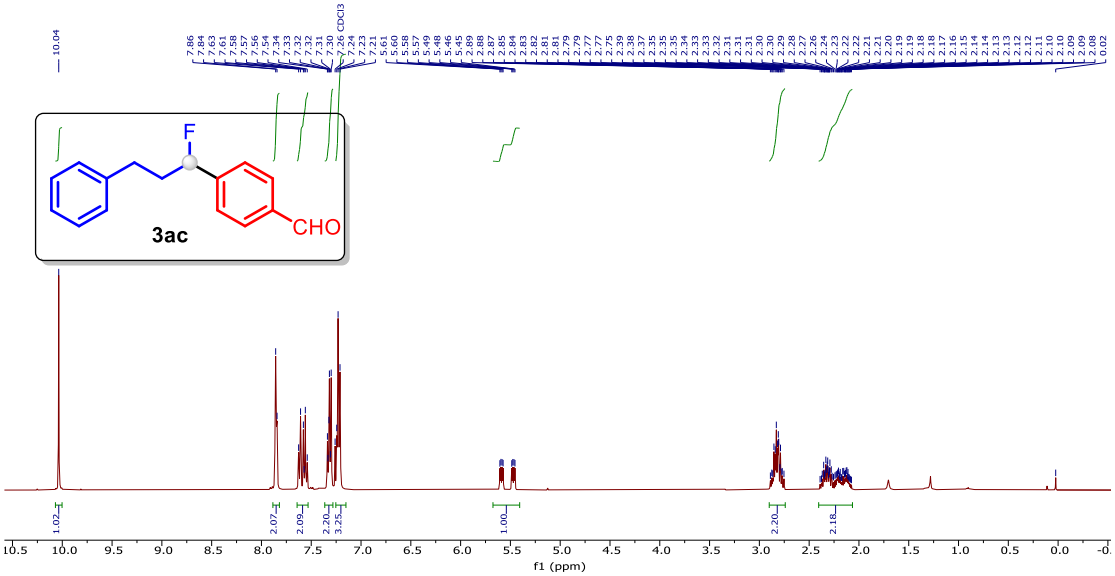
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ab**



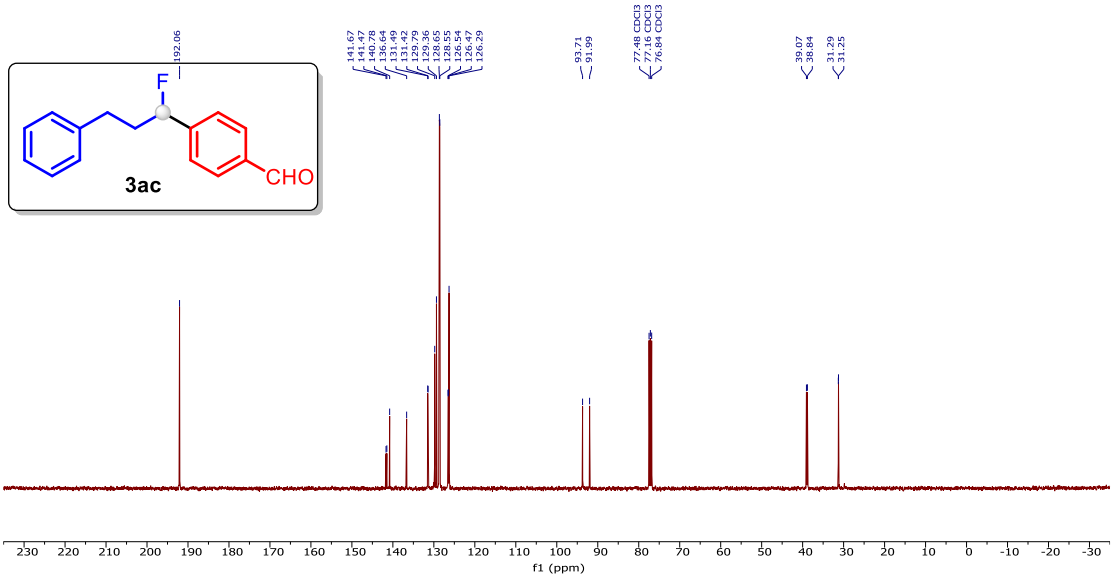
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ab**



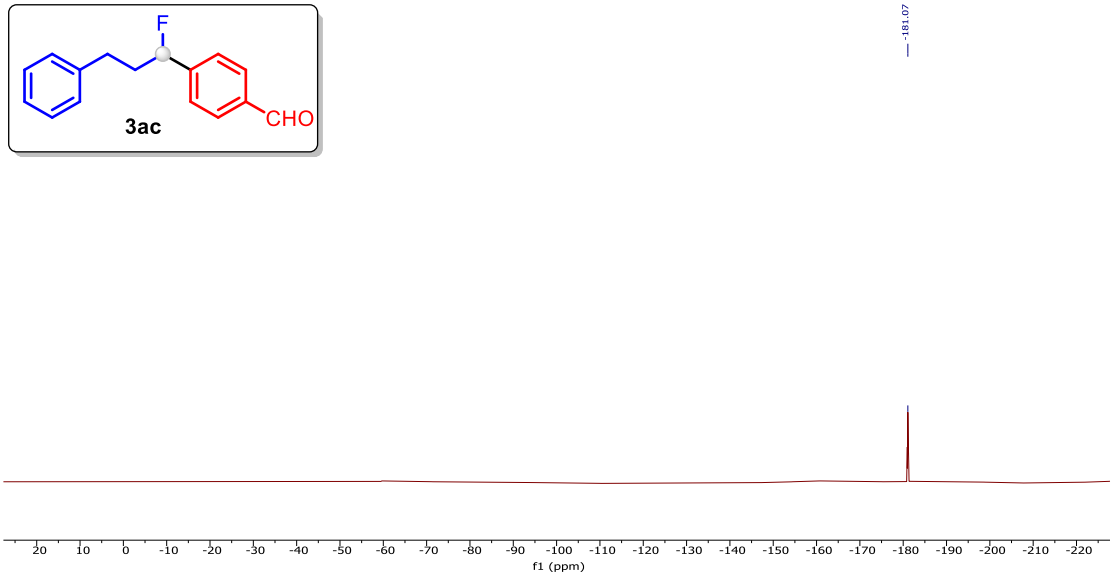
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ab**



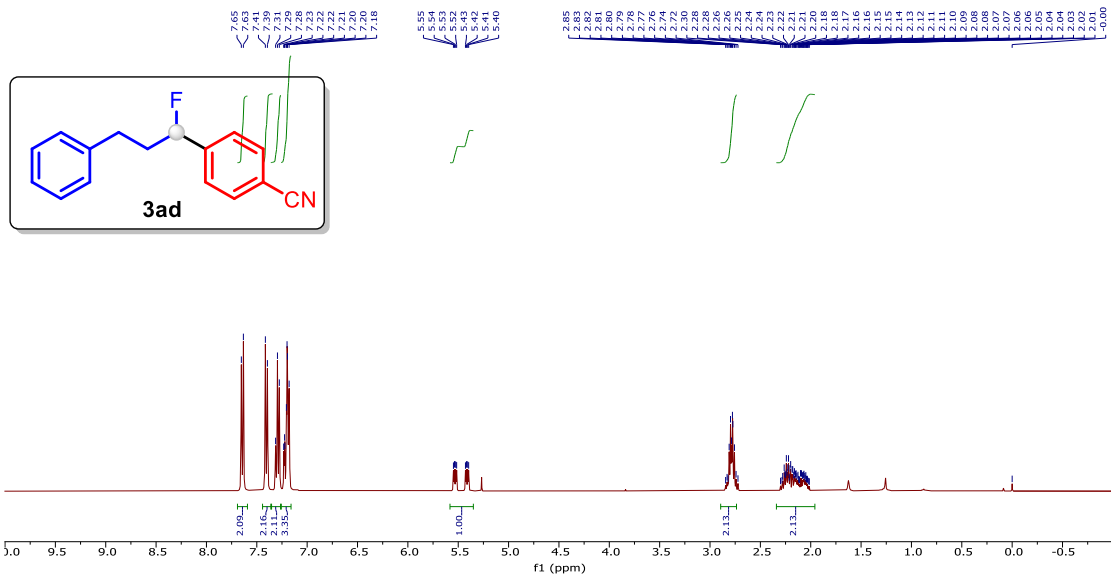
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ac**



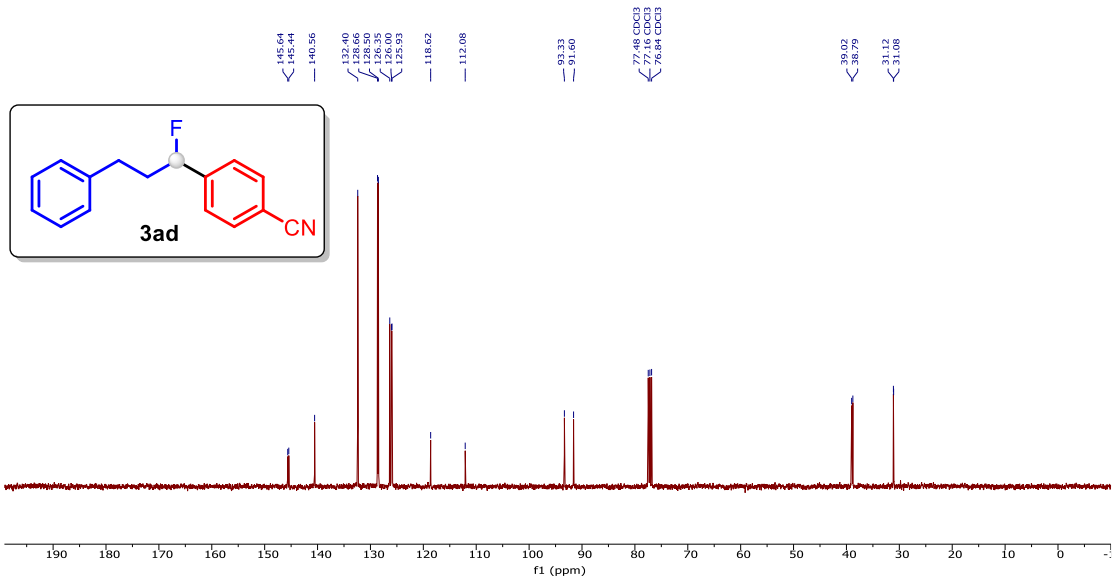
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ac**



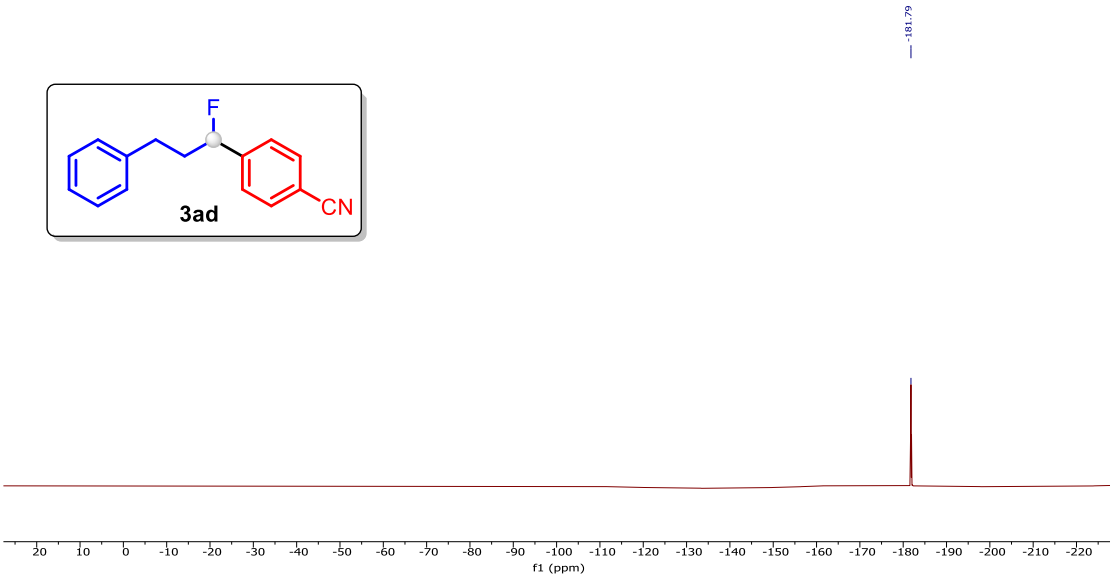
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ac**



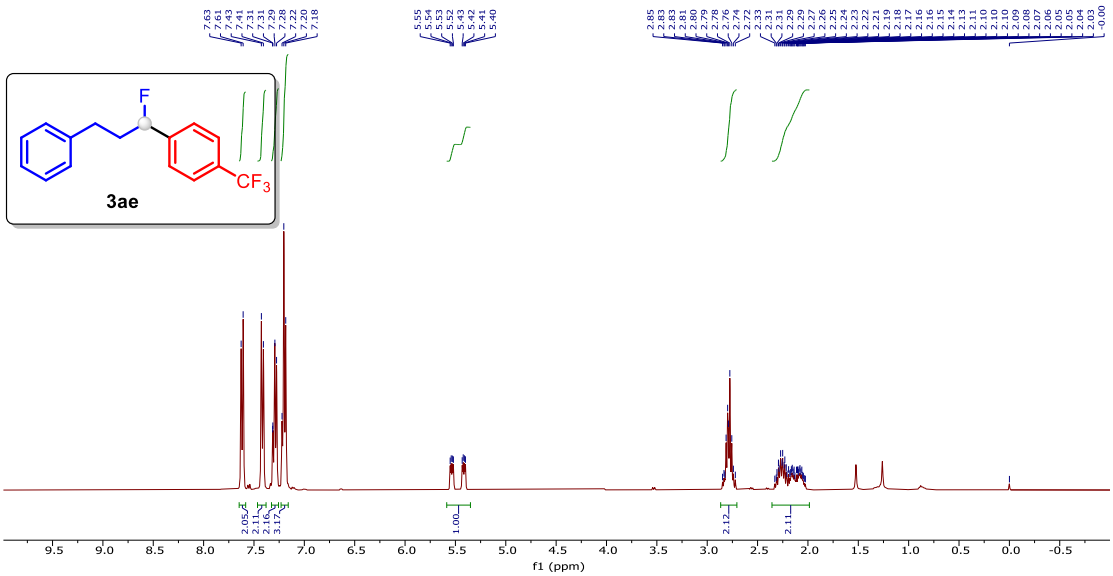
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ad**



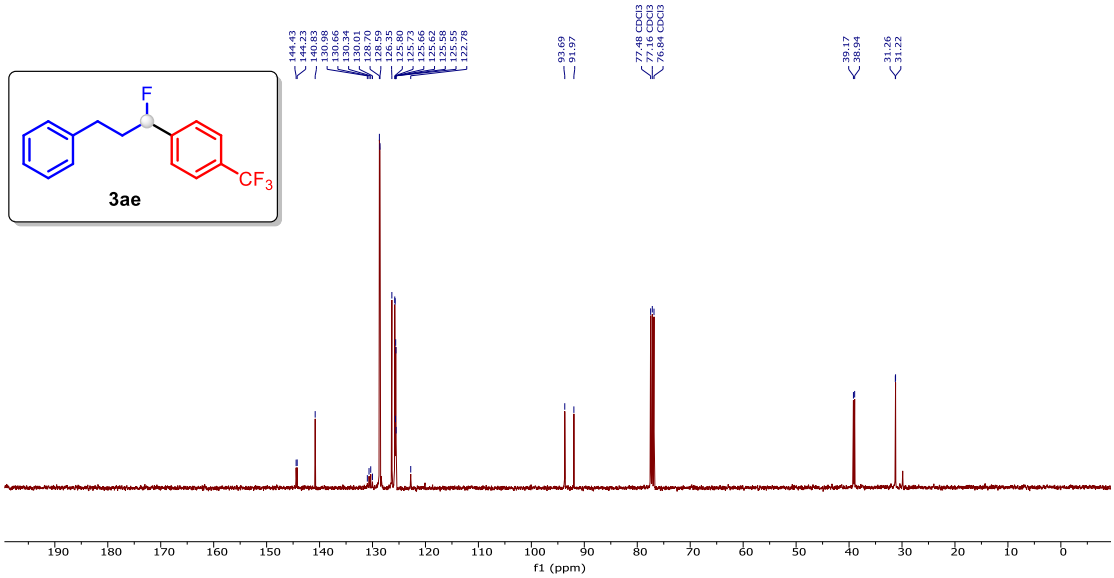
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ad**



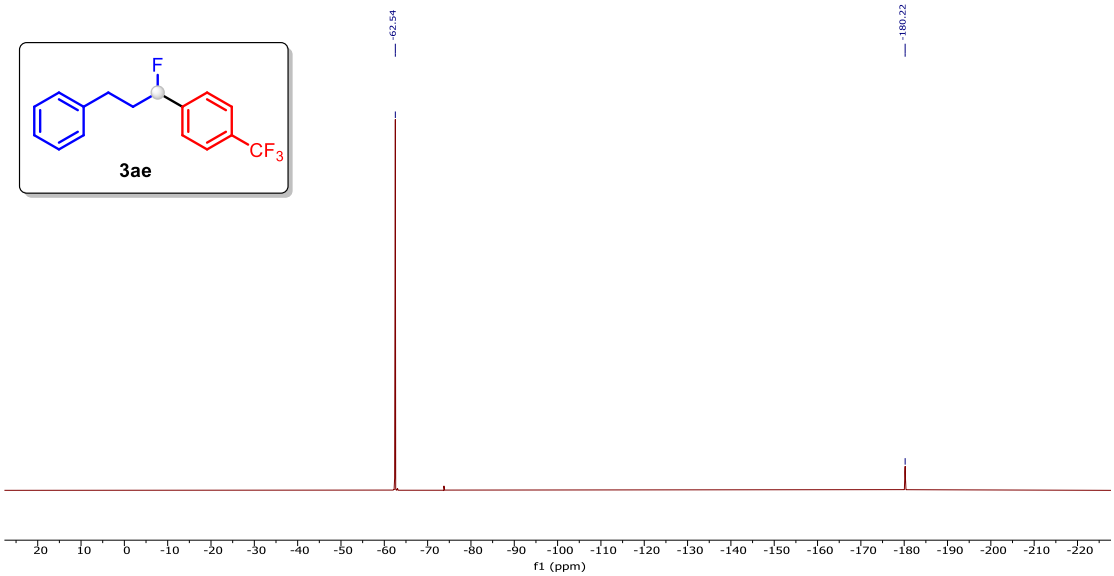
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ad**



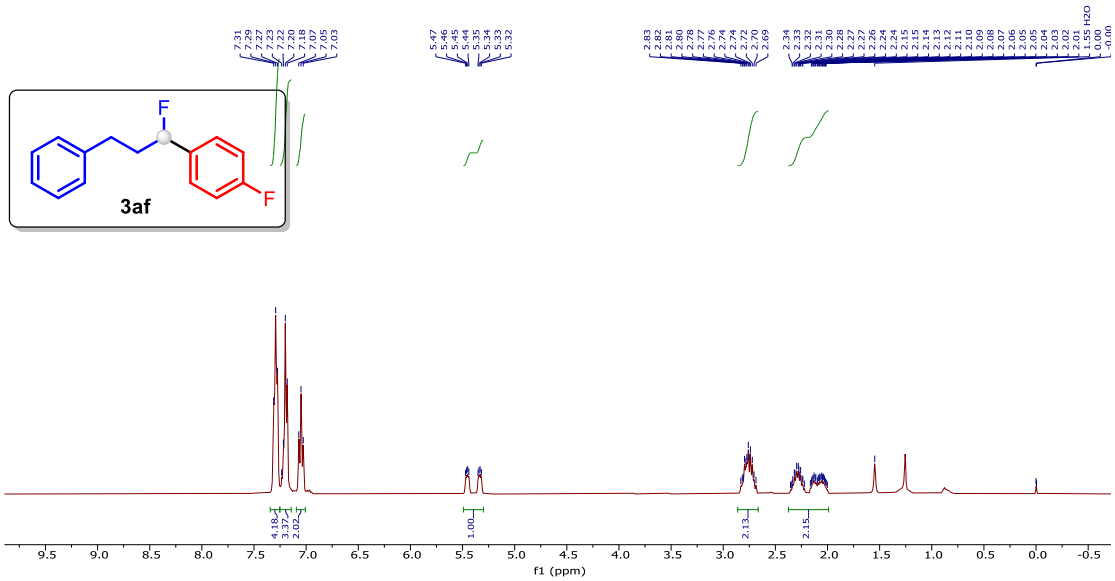
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ae**



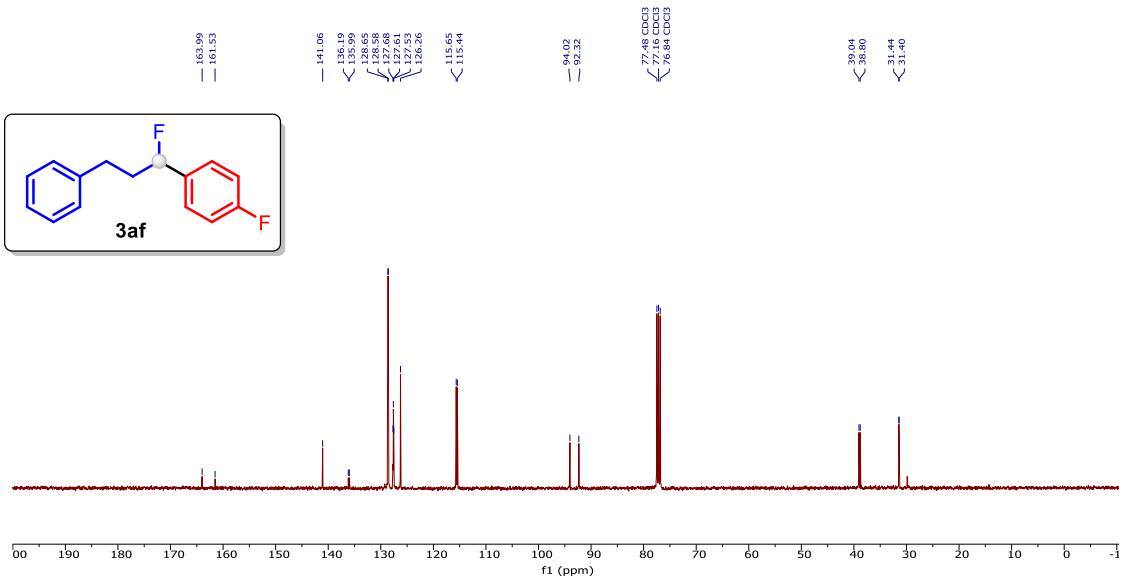
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ae**



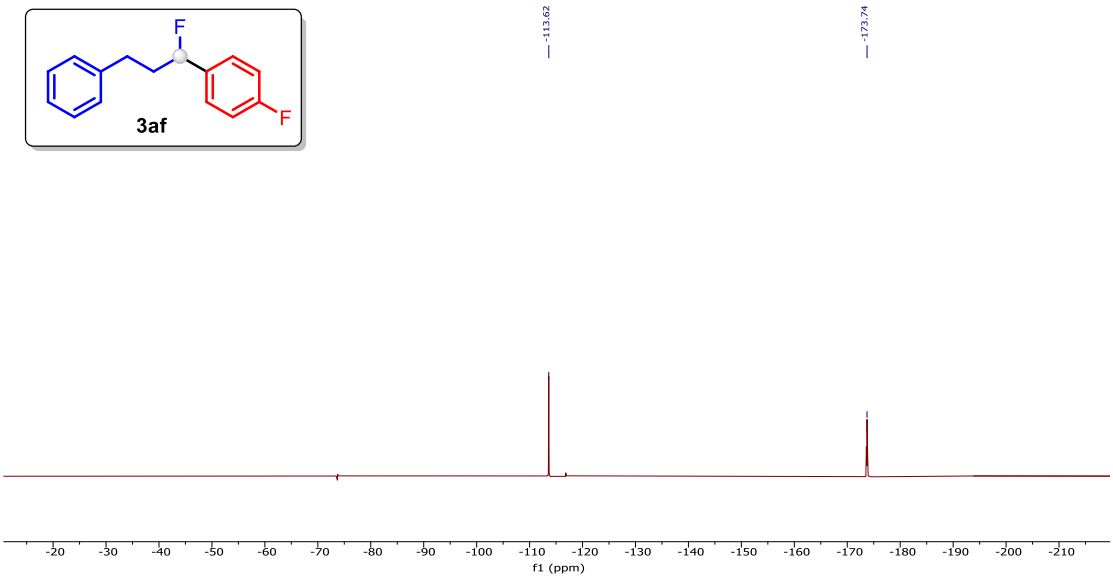
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ae**



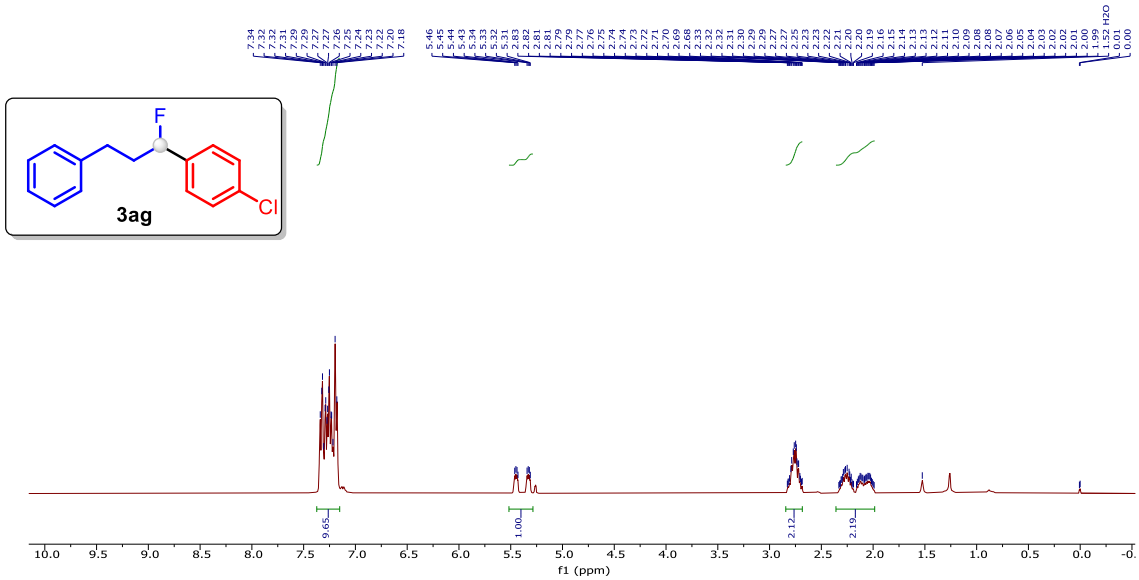
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3af**



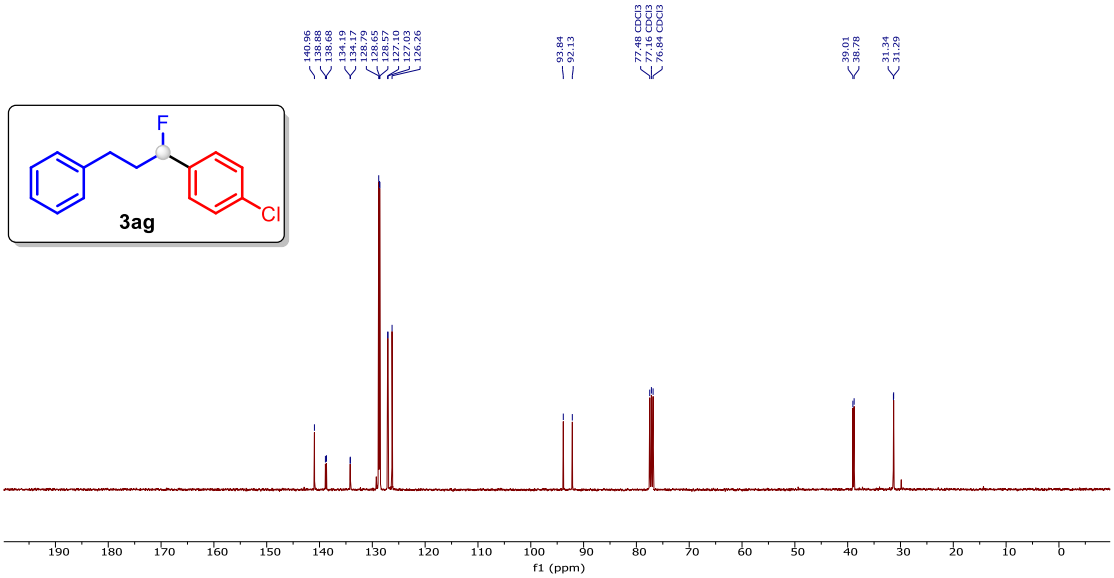
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3af**



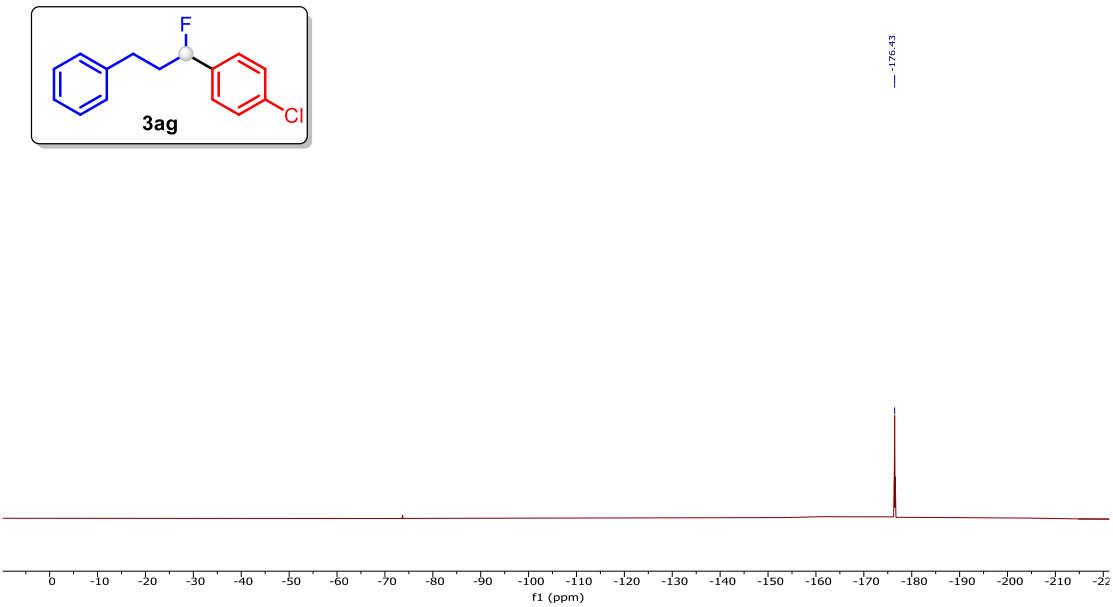
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3af**



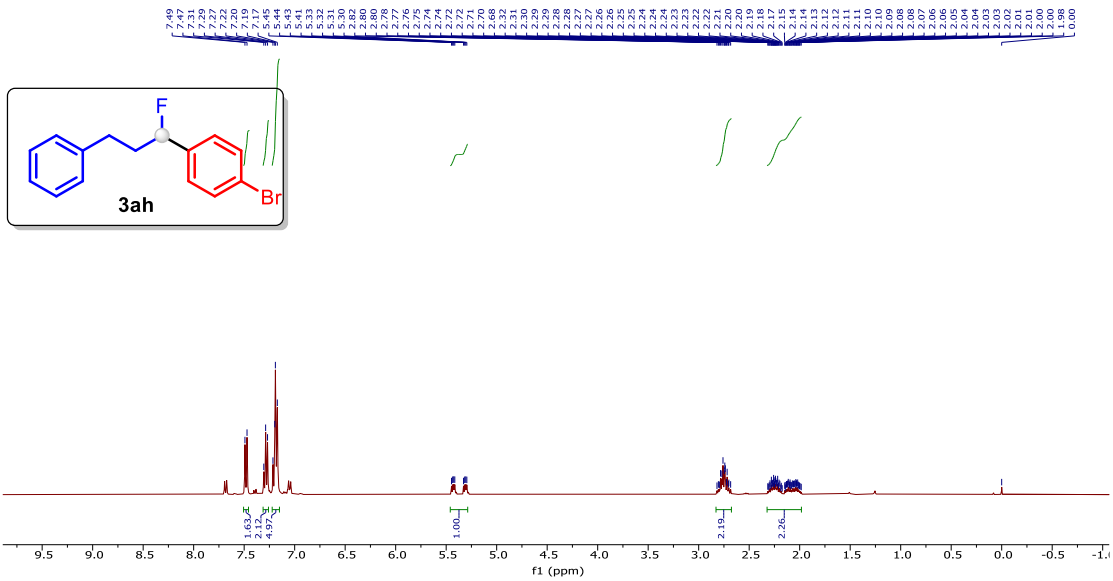
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ag**



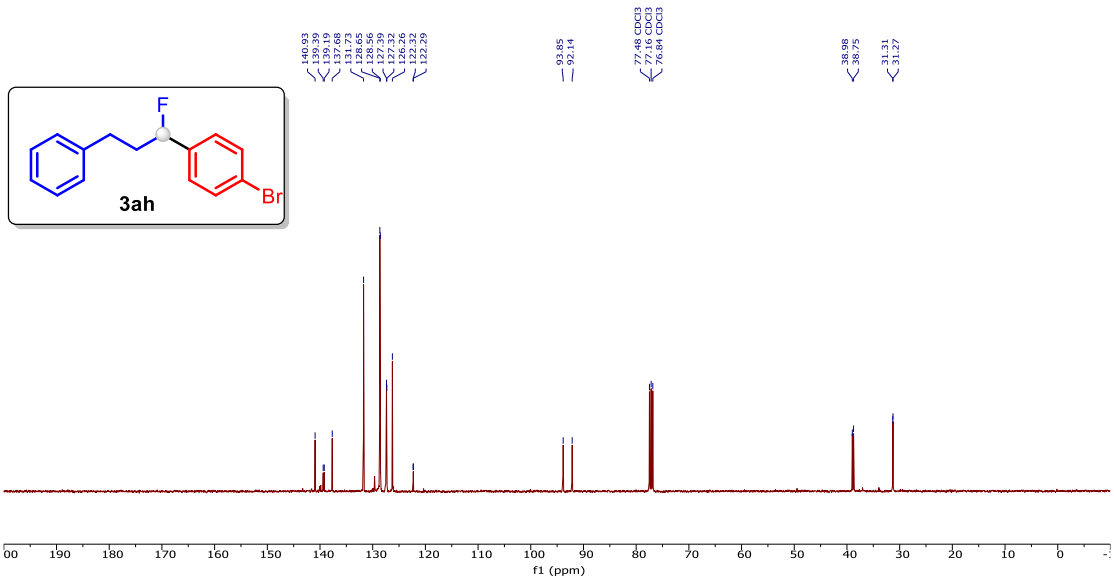
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ag**



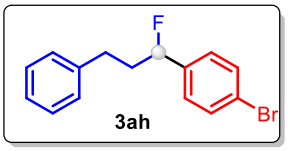
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ag**

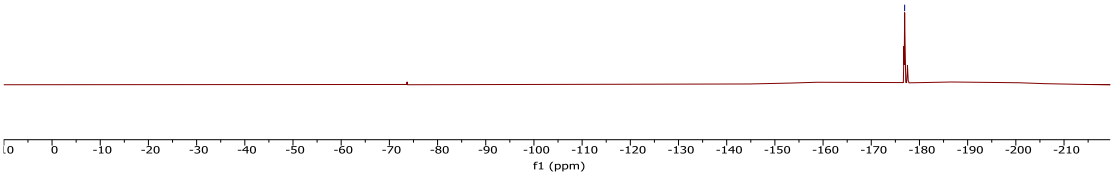


^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ah**

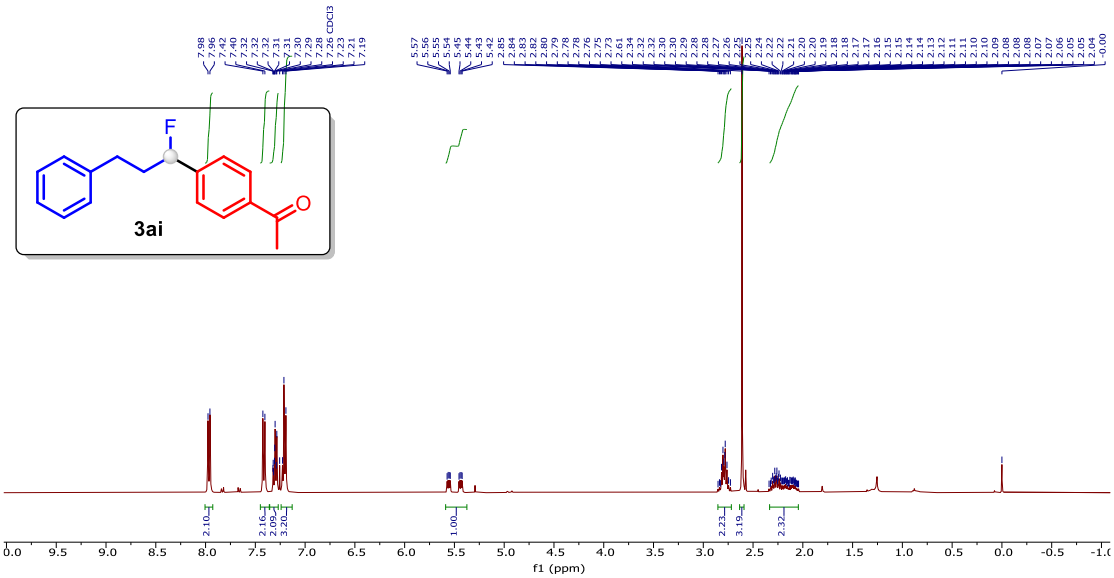


^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ah**

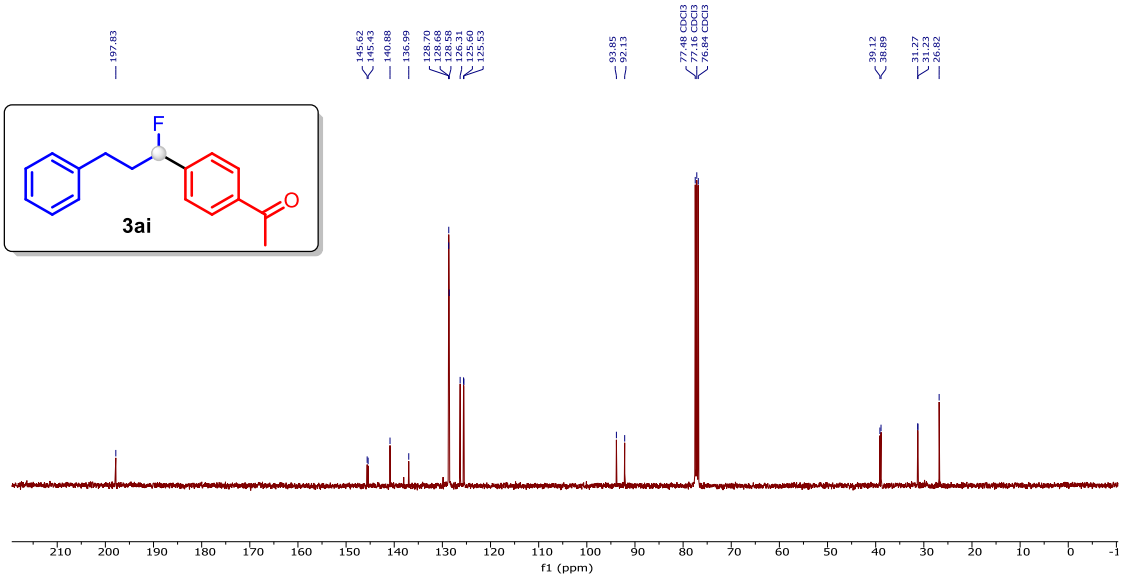




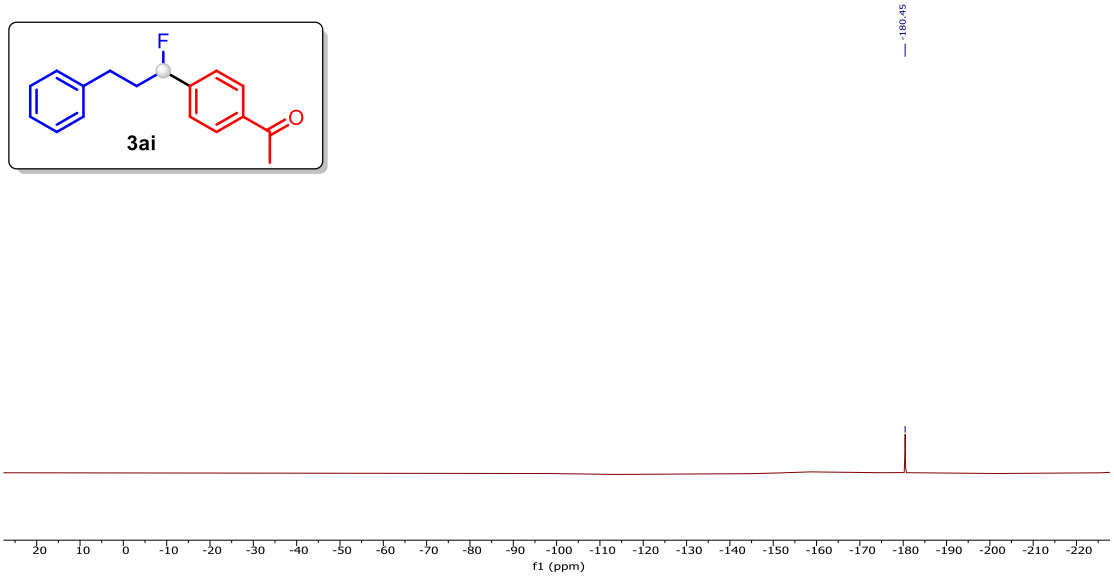
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ah**



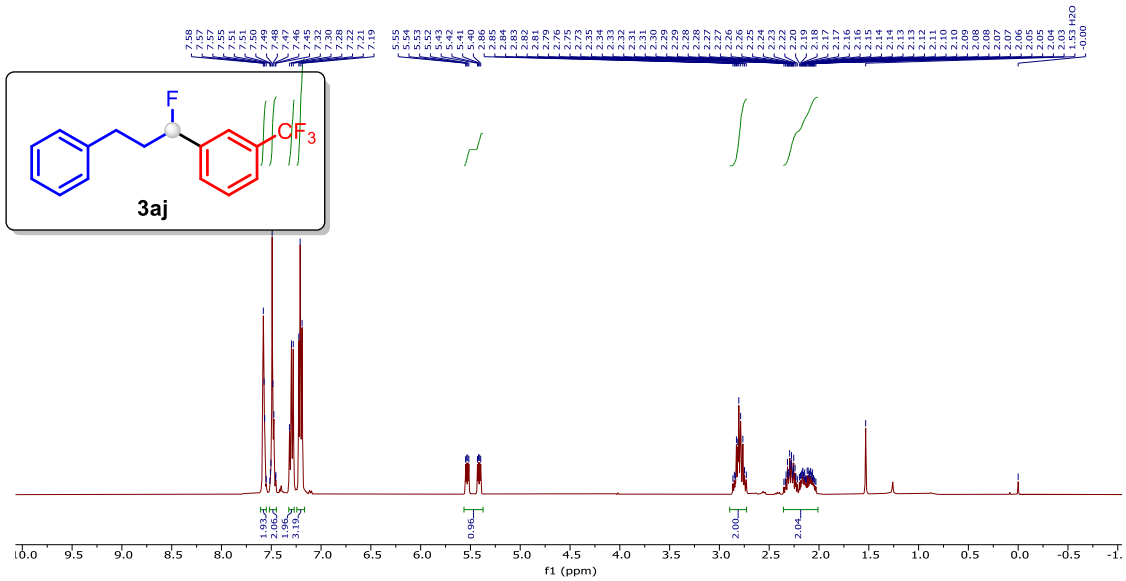
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ai**



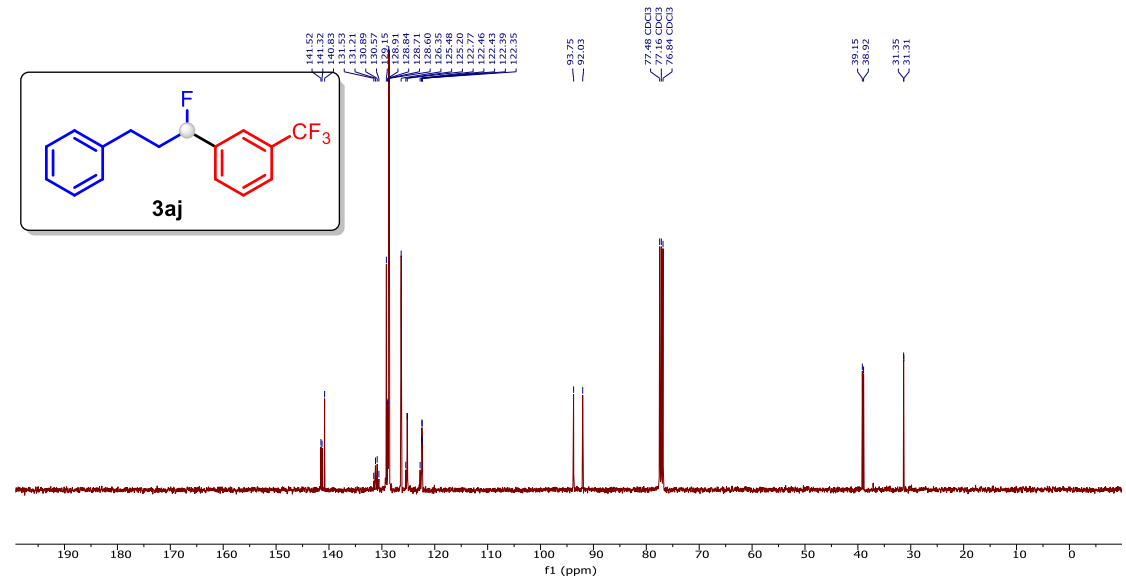
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ai**



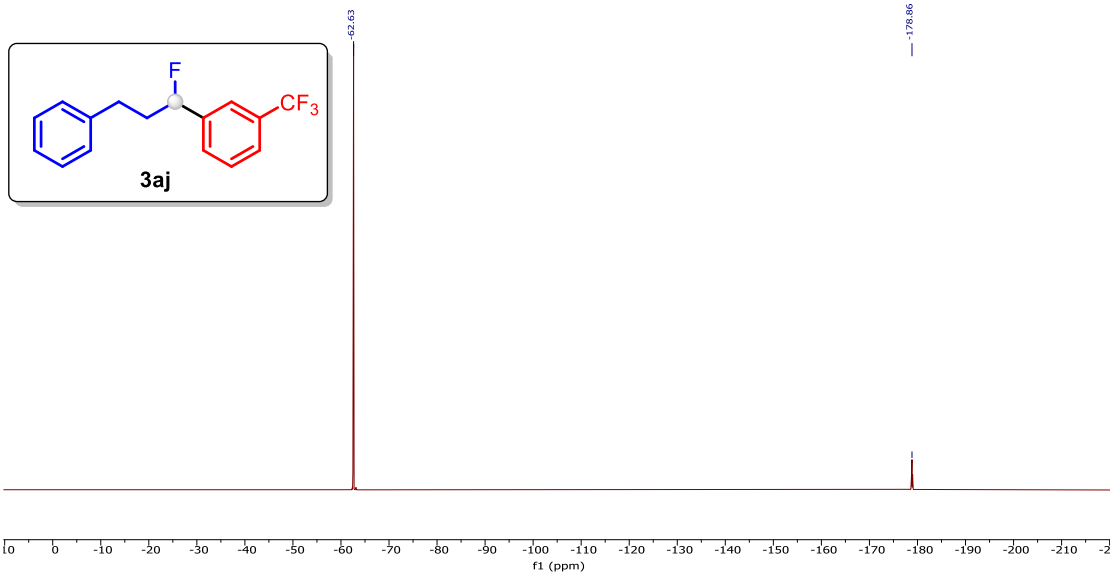
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ai**



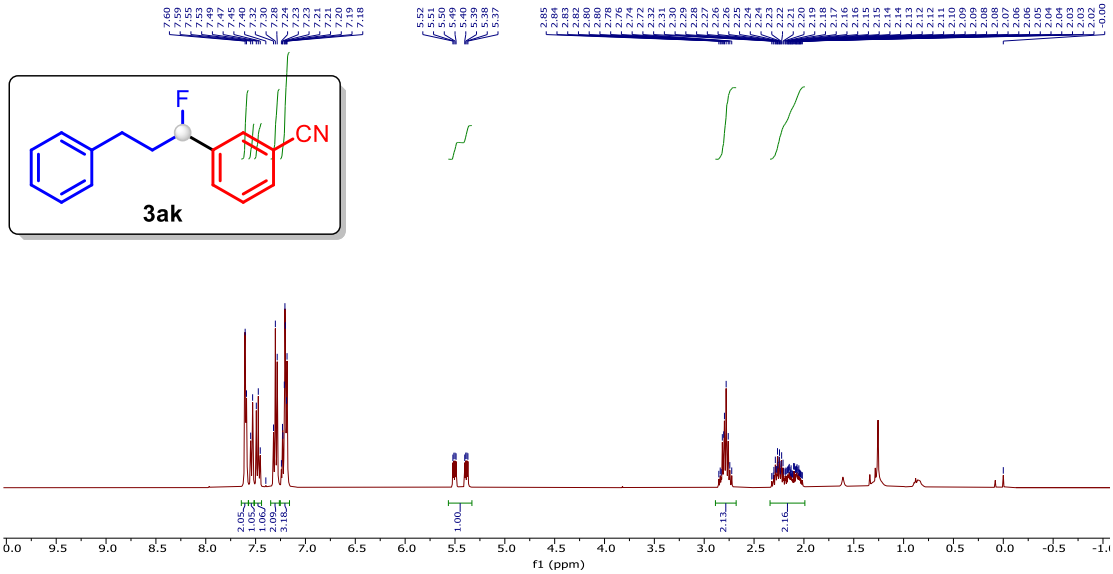
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3aj**



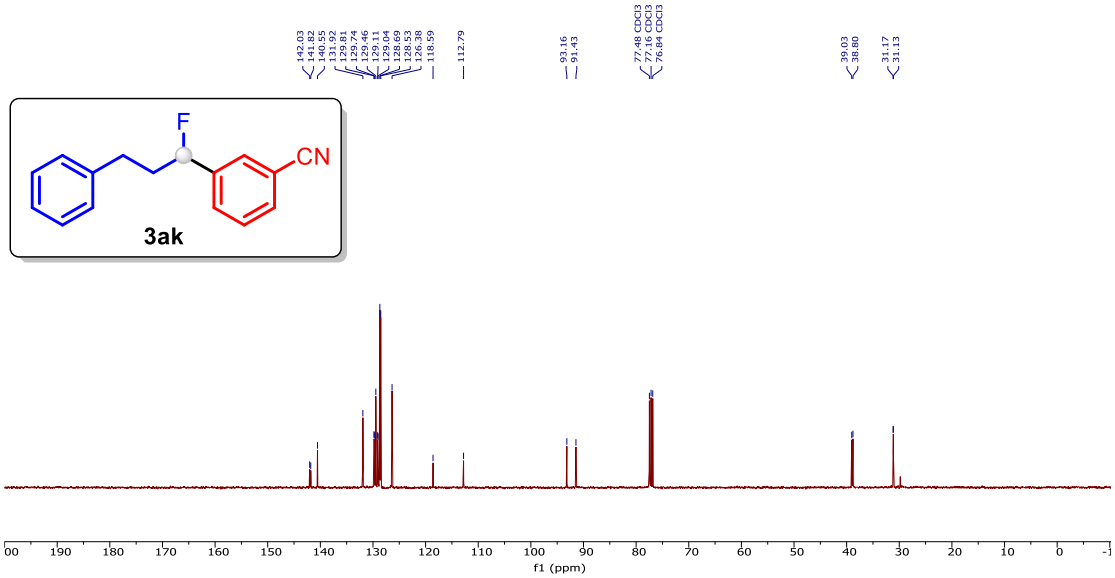
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3aj**



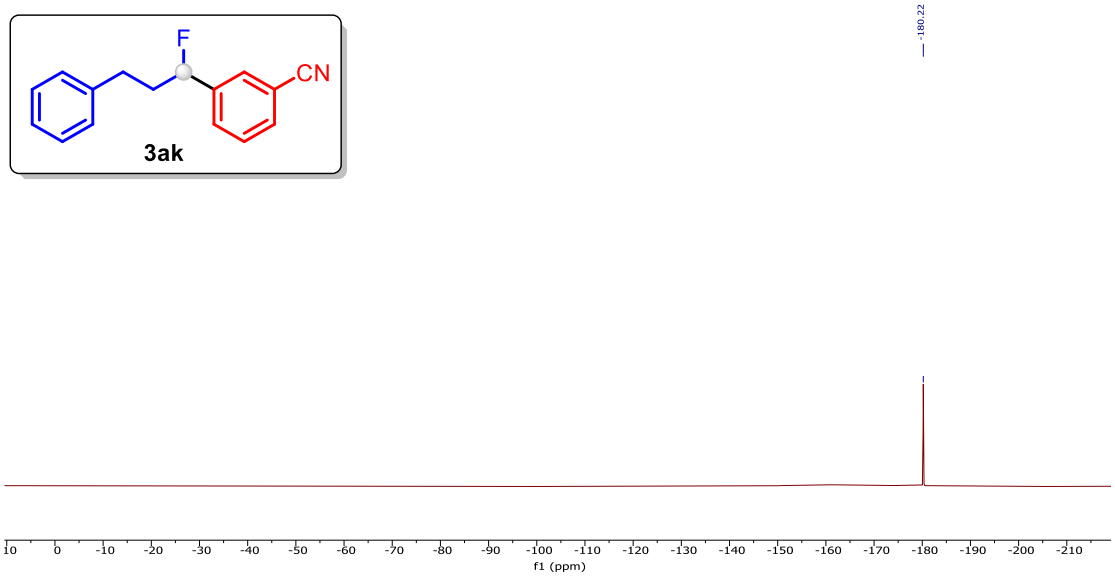
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3aj**



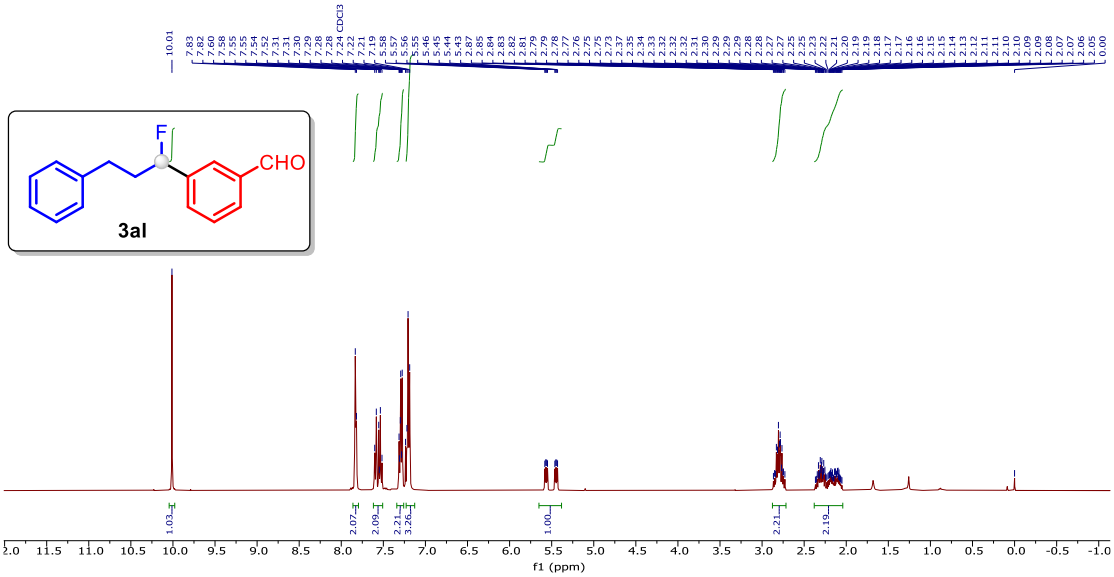
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ak**



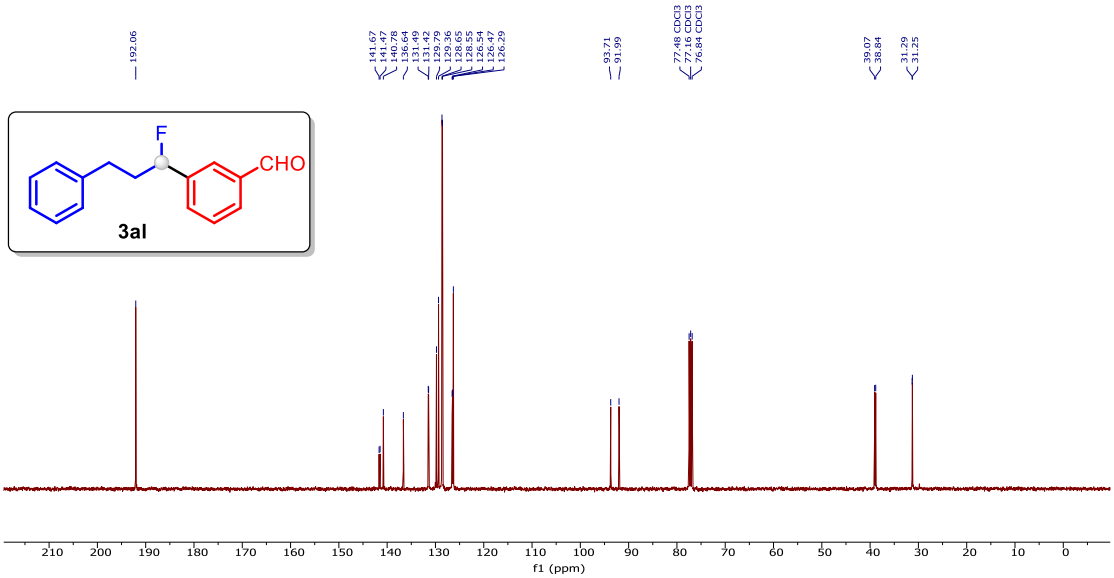
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ak**



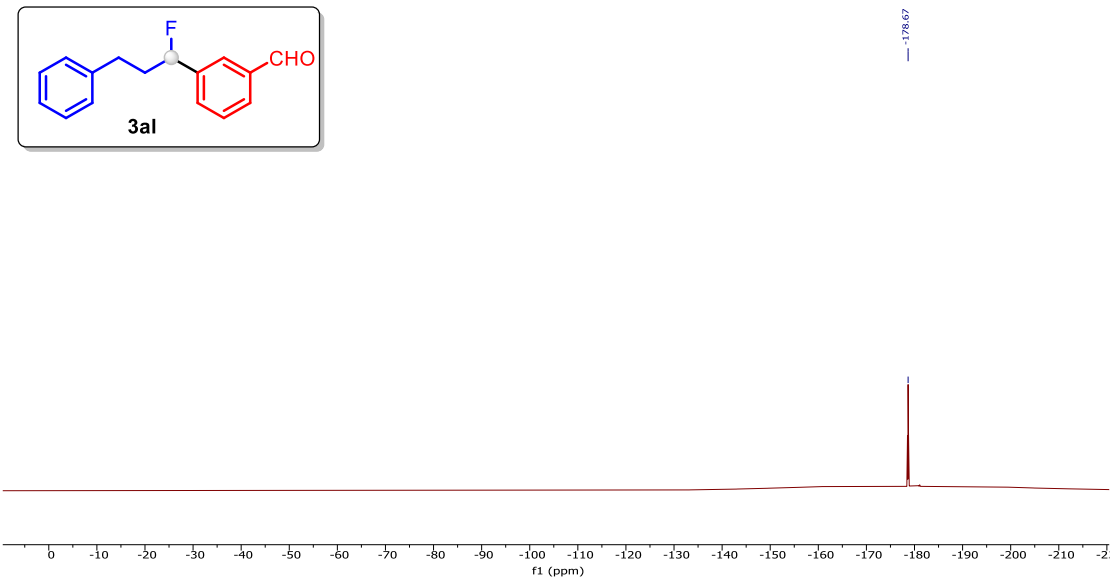
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ak**



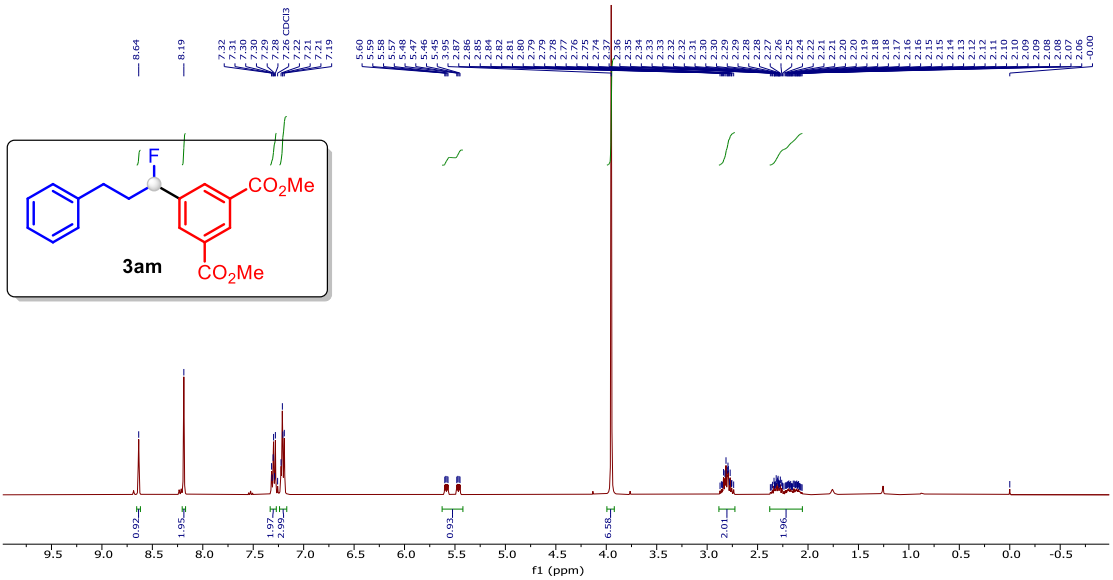
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3al**



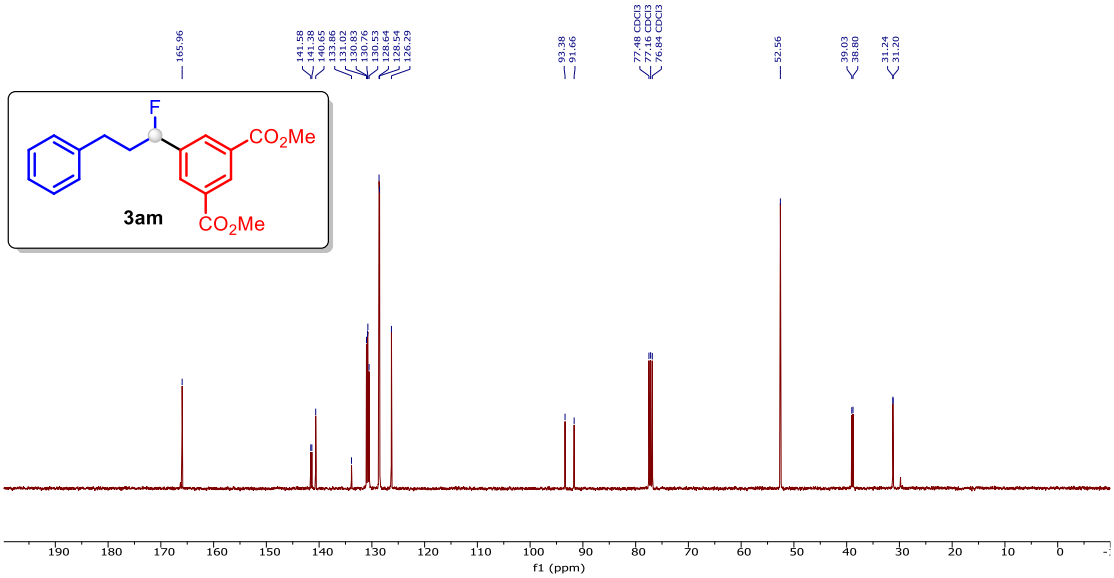
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3al**



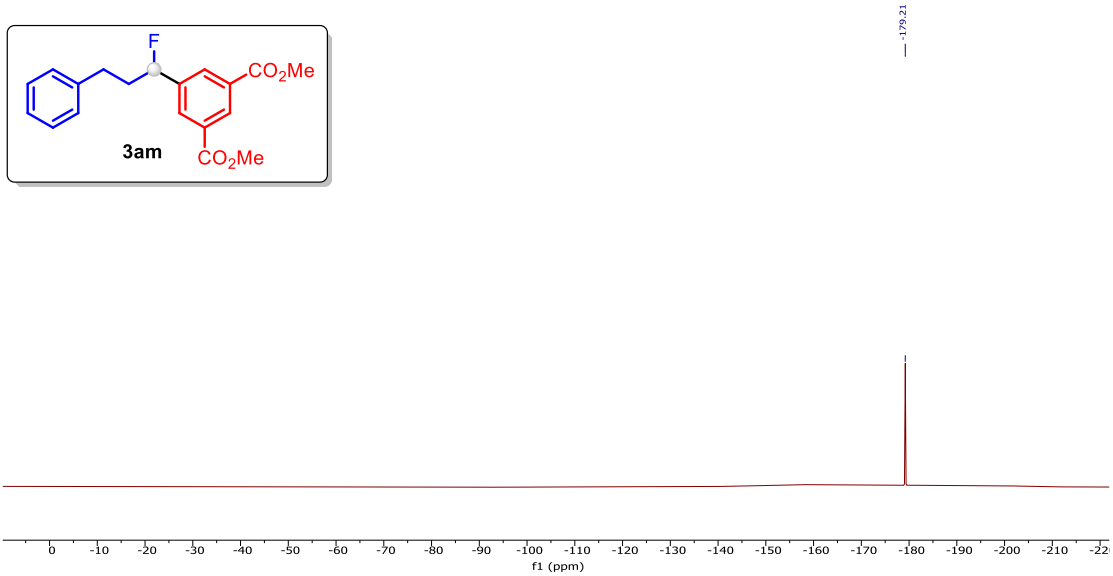
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3al**



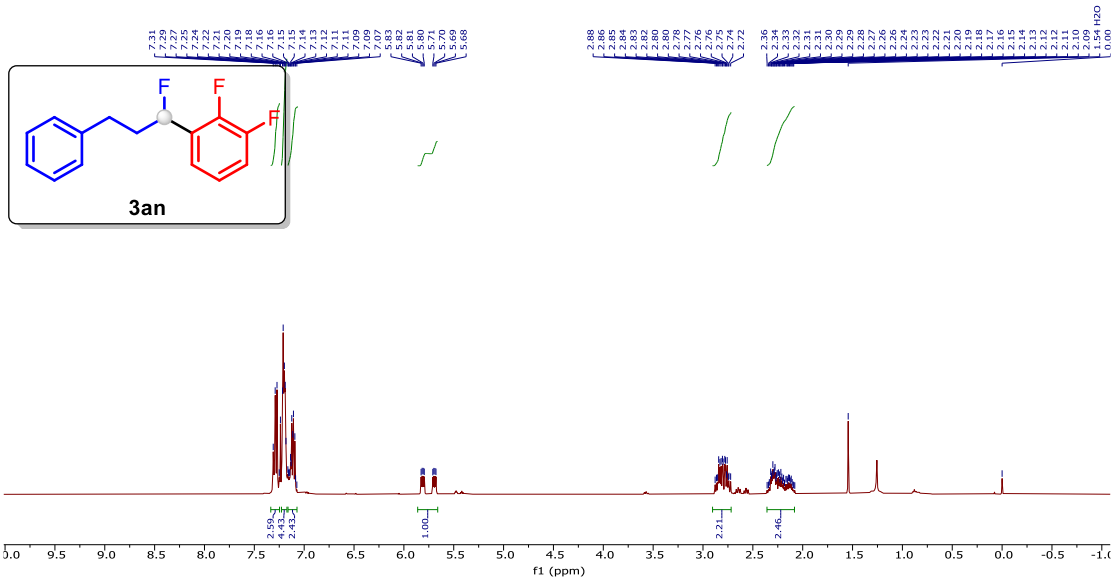
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3am**



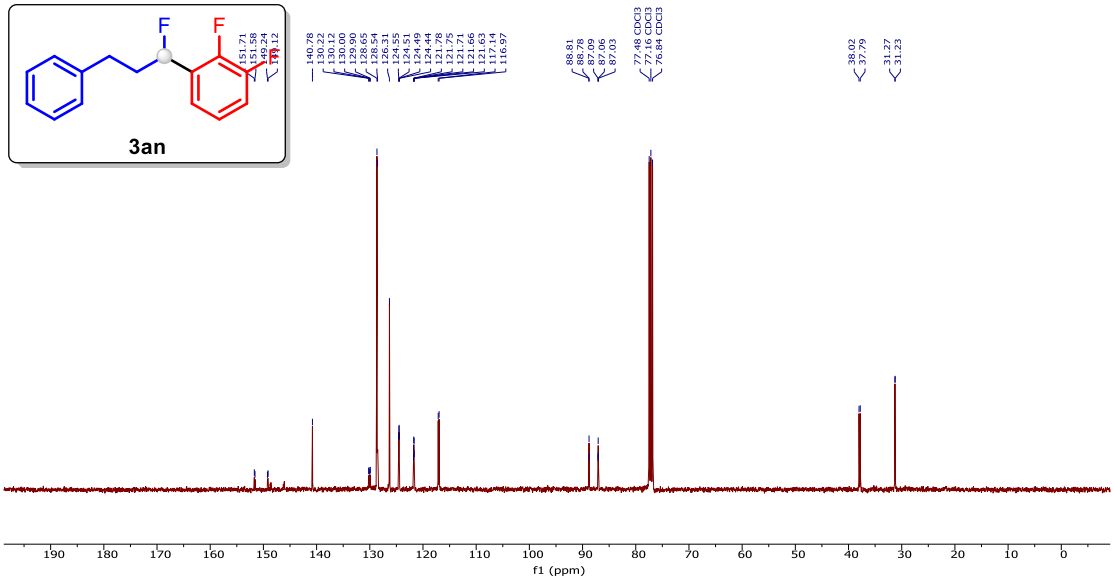
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3am**



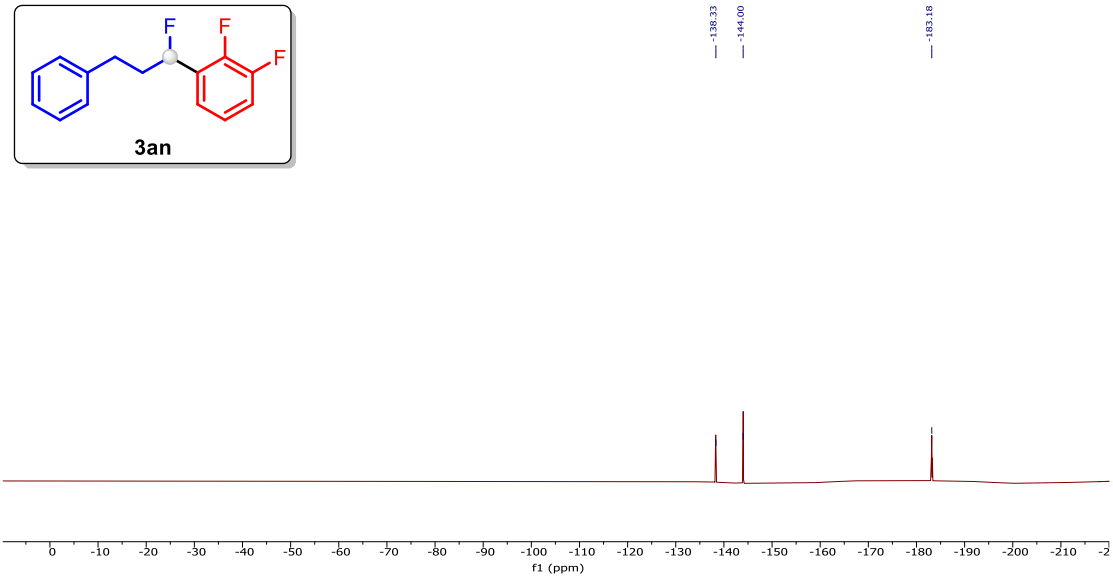
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3am**



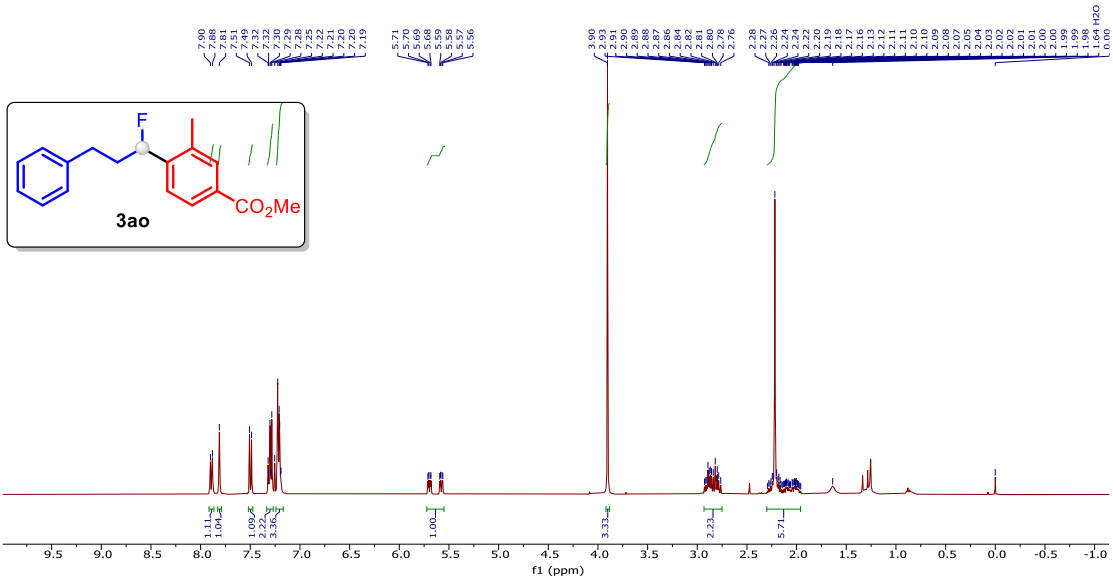
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3an**



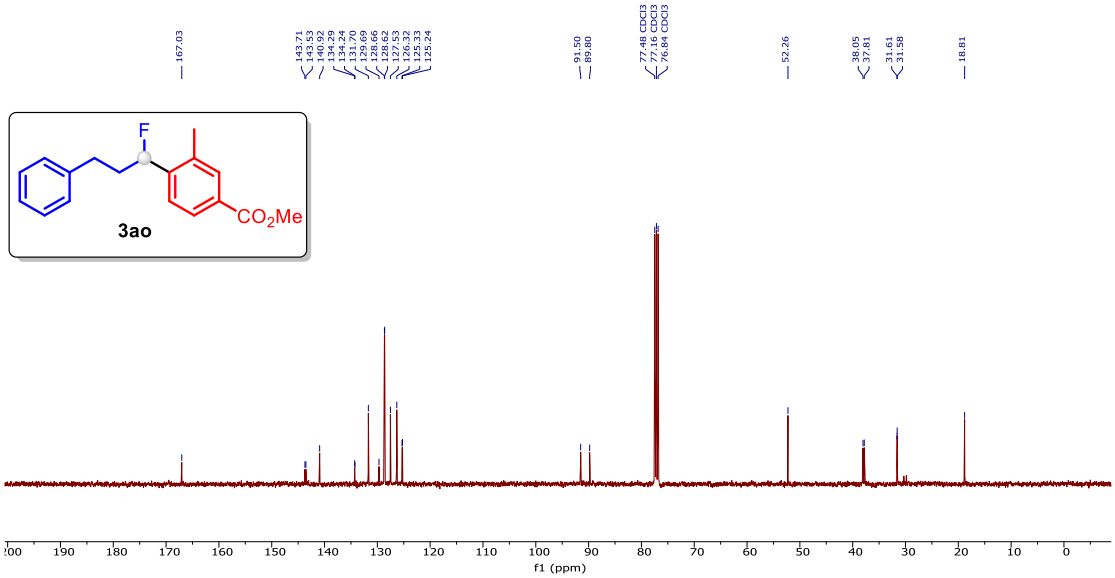
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3an**



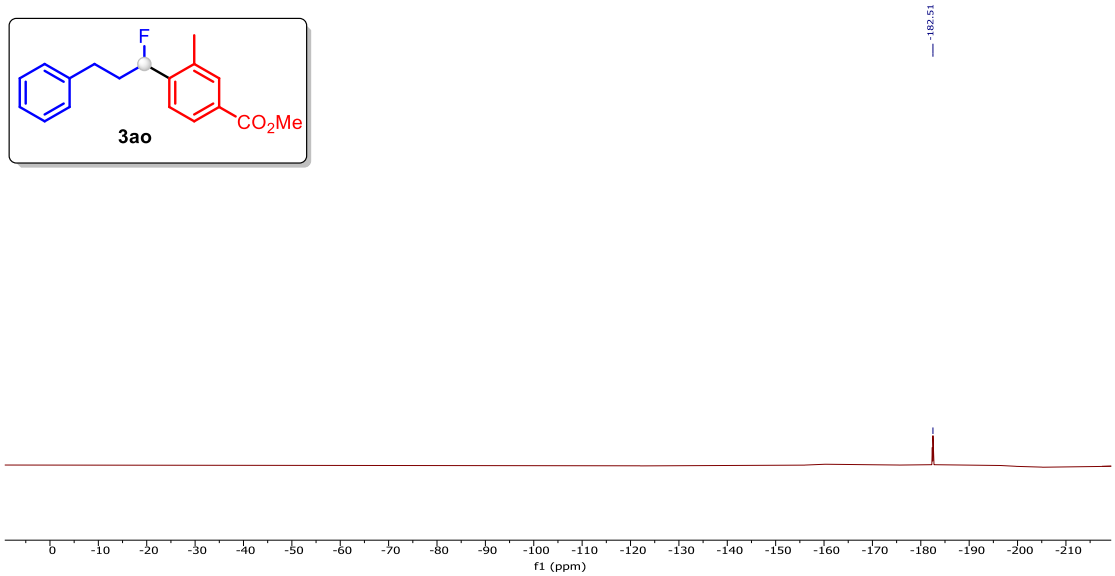
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3an**



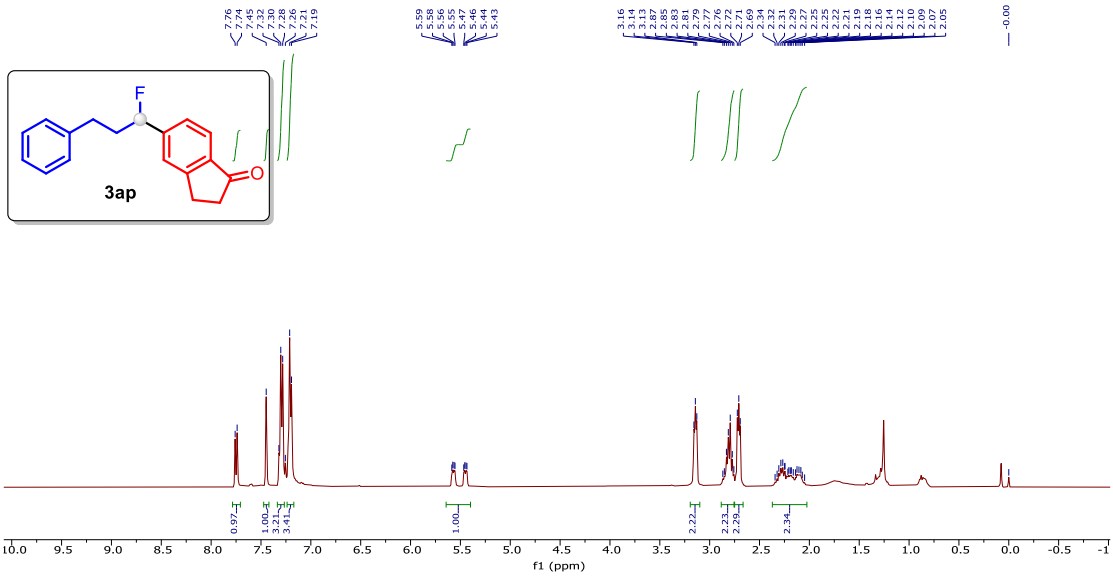
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ao**



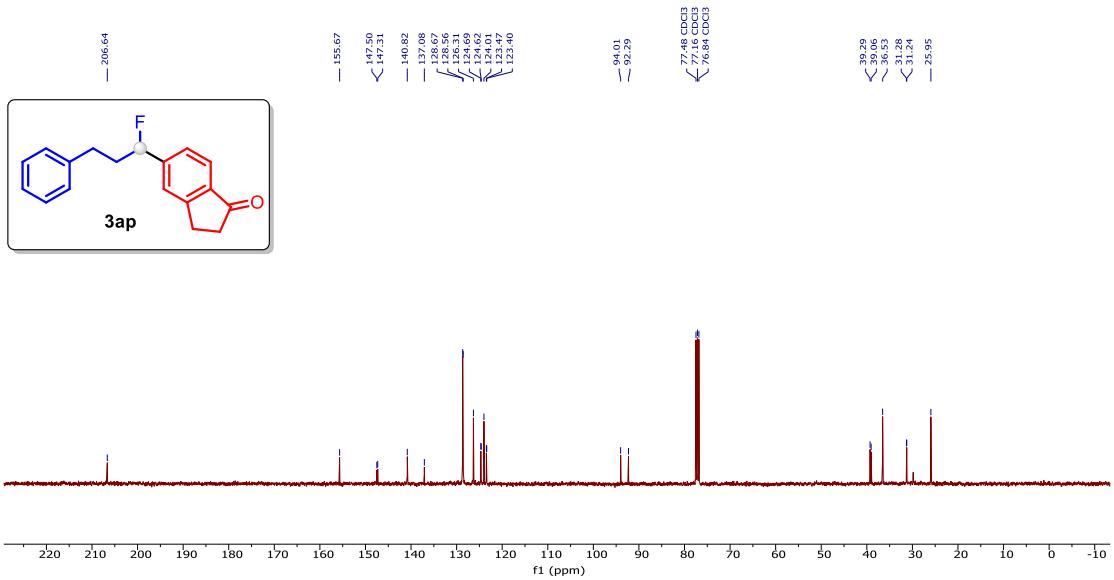
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ao**



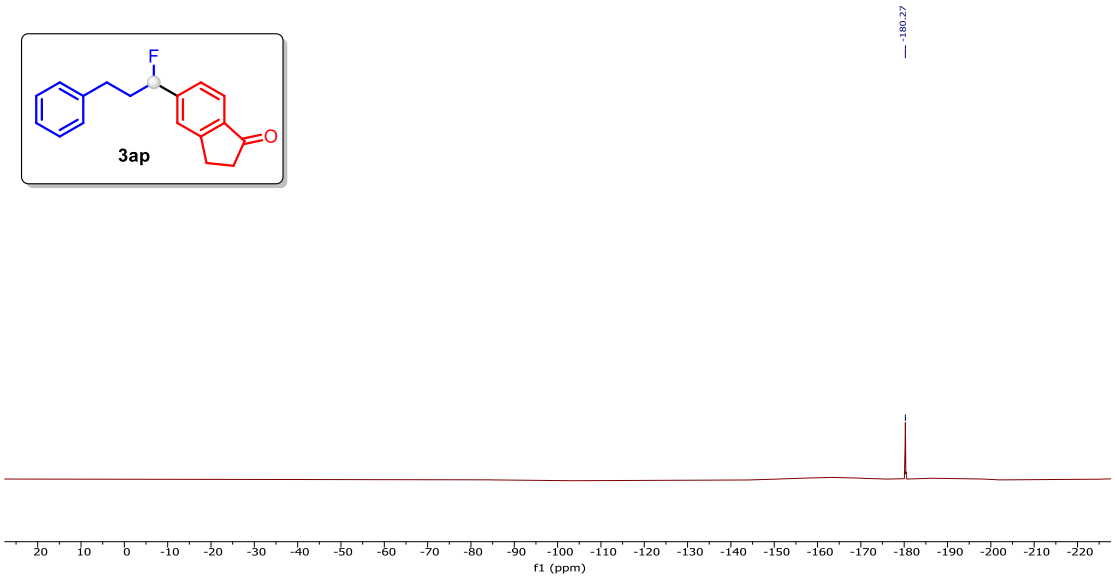
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ao**



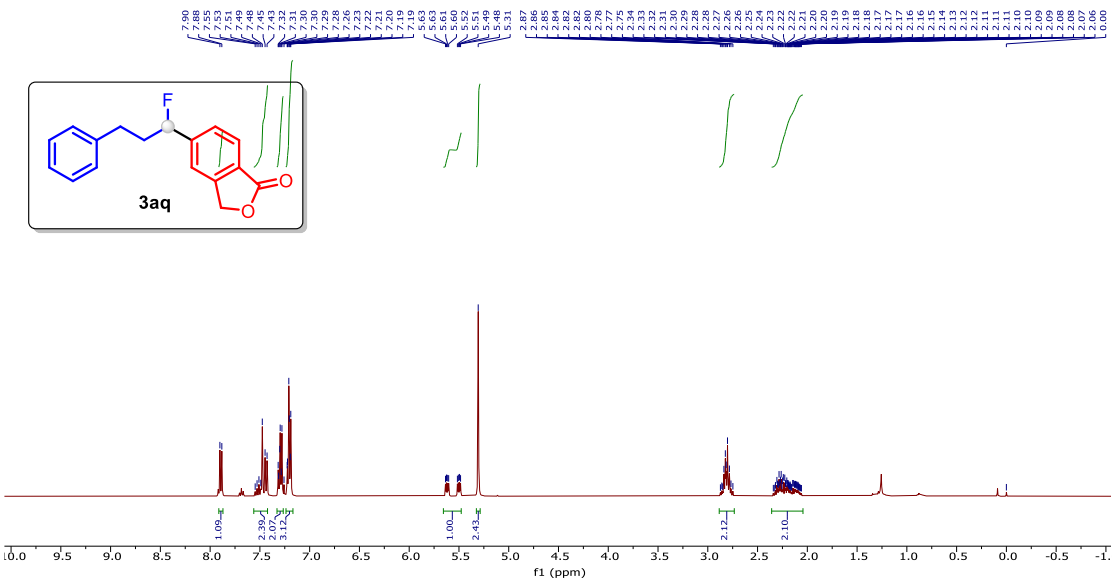
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ap**



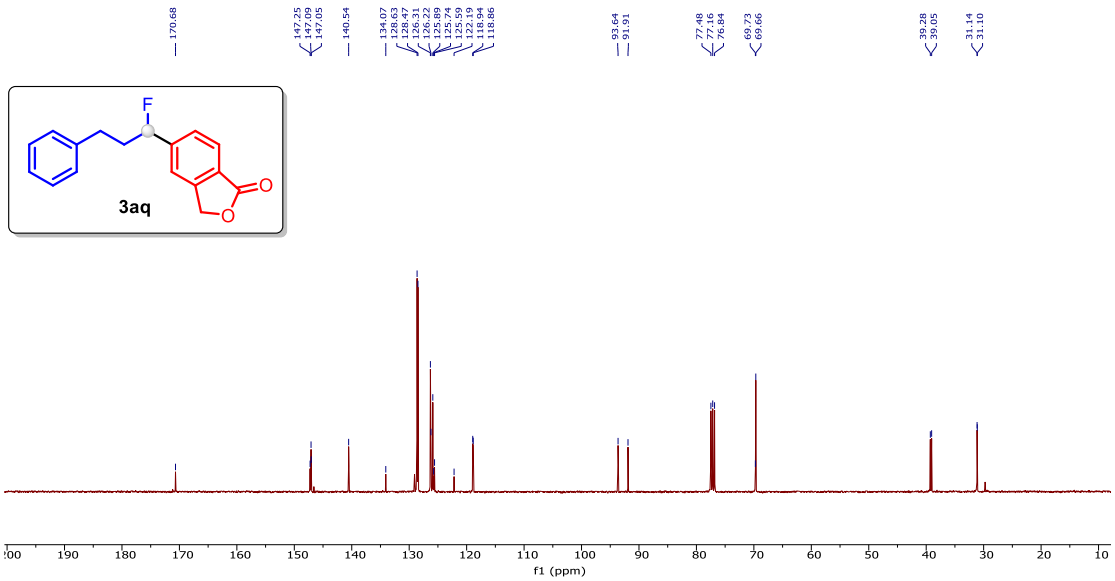
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ap**



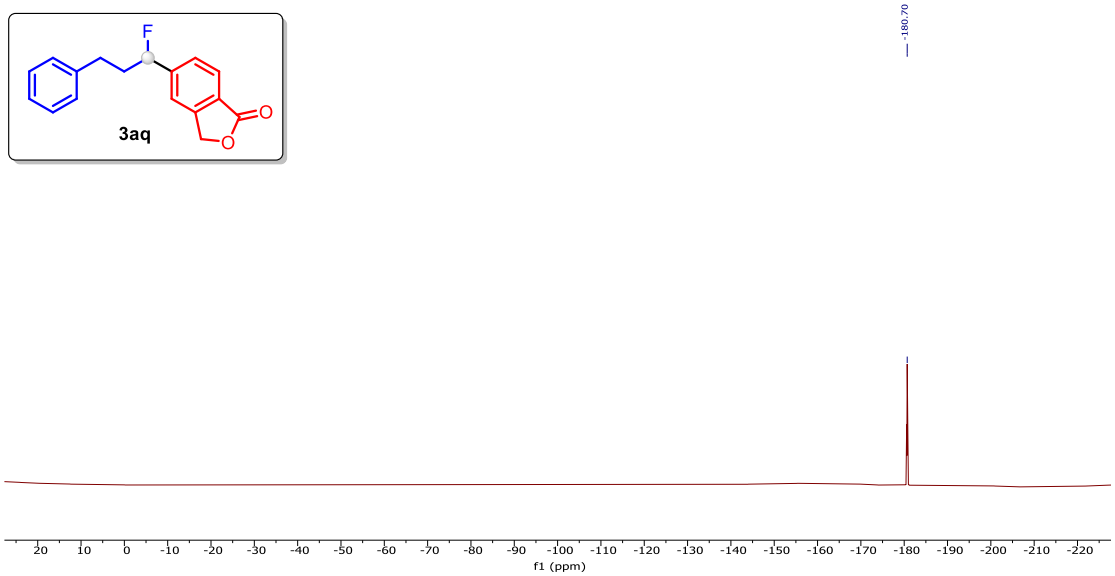
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ap**



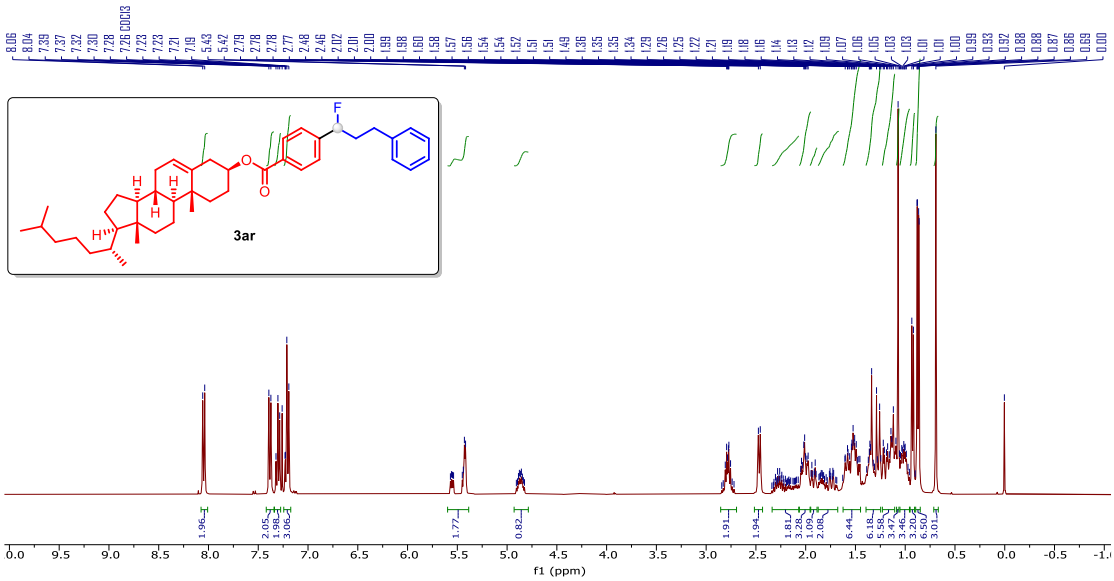
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3aq**



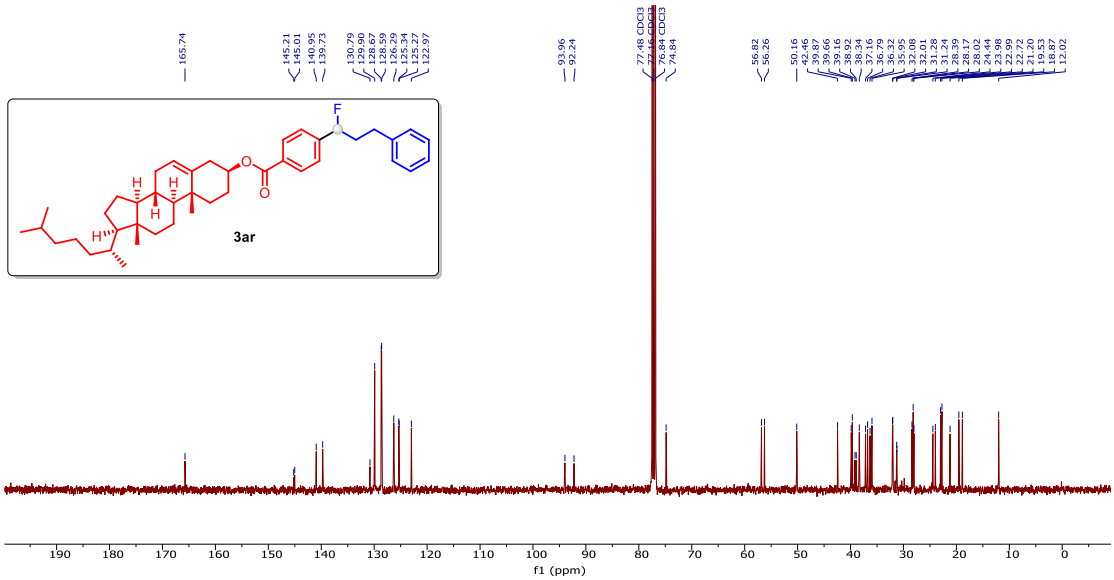
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3aq**



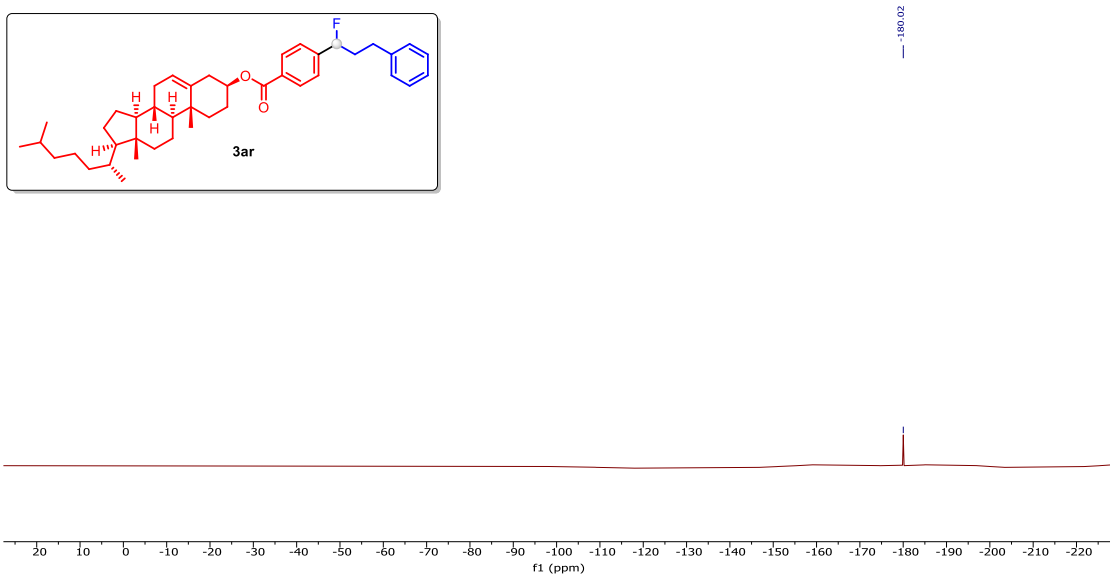
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3aq**



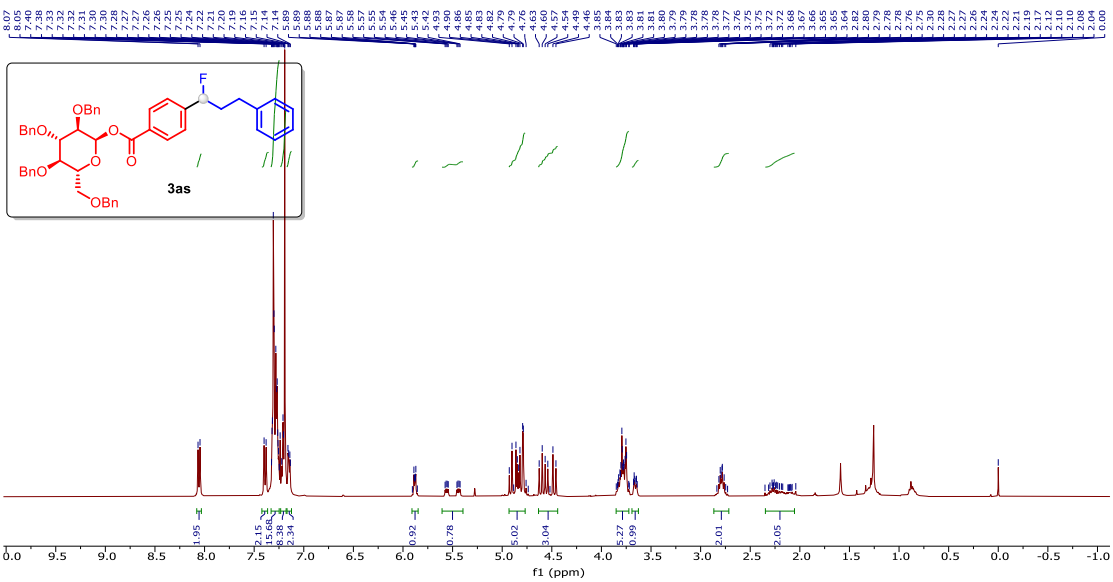
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ar**



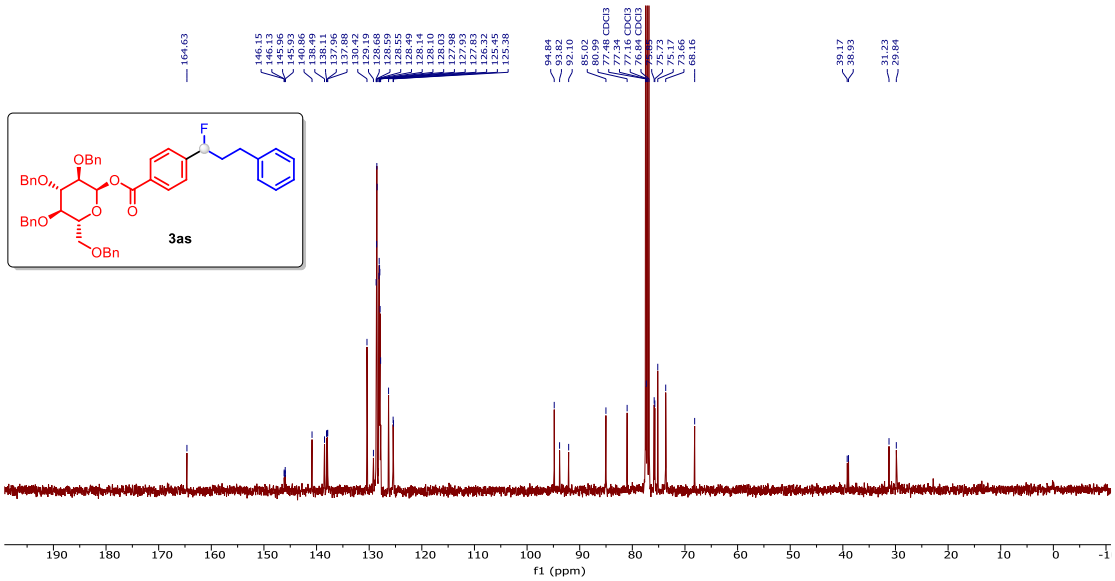
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ar**



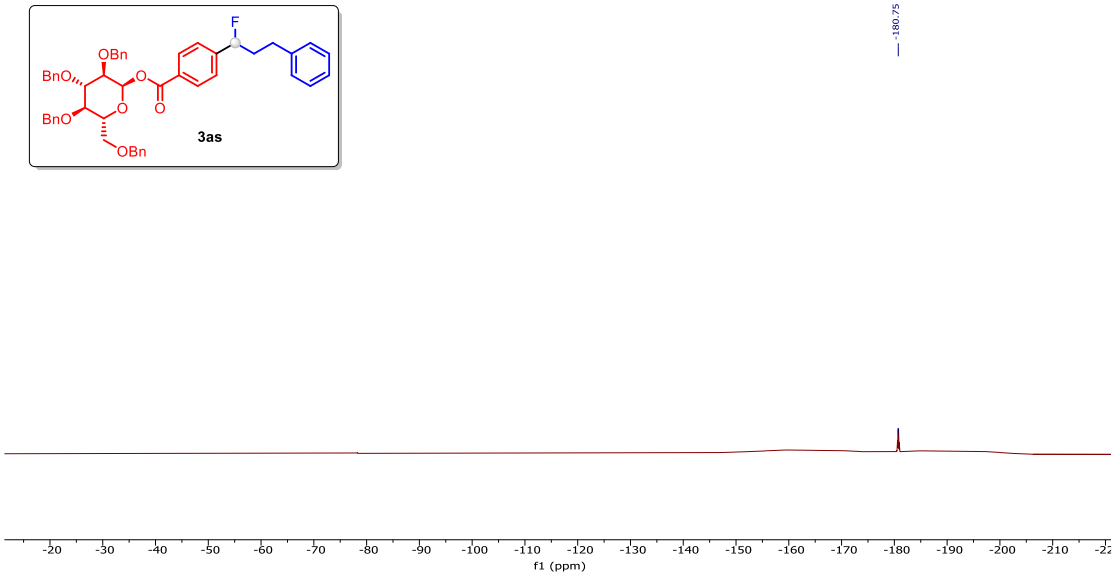
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ar**



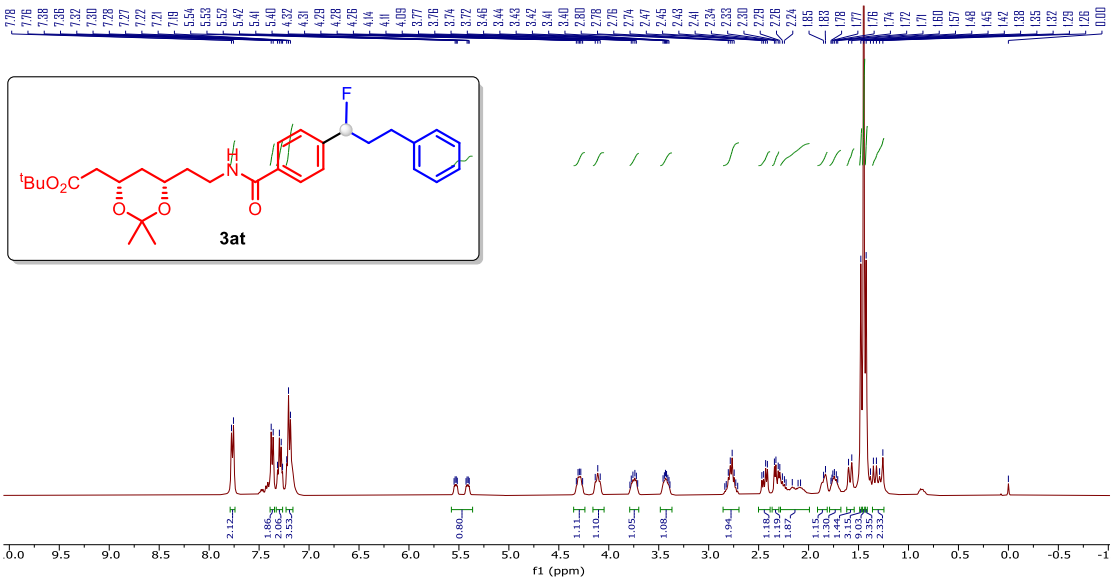
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3as**



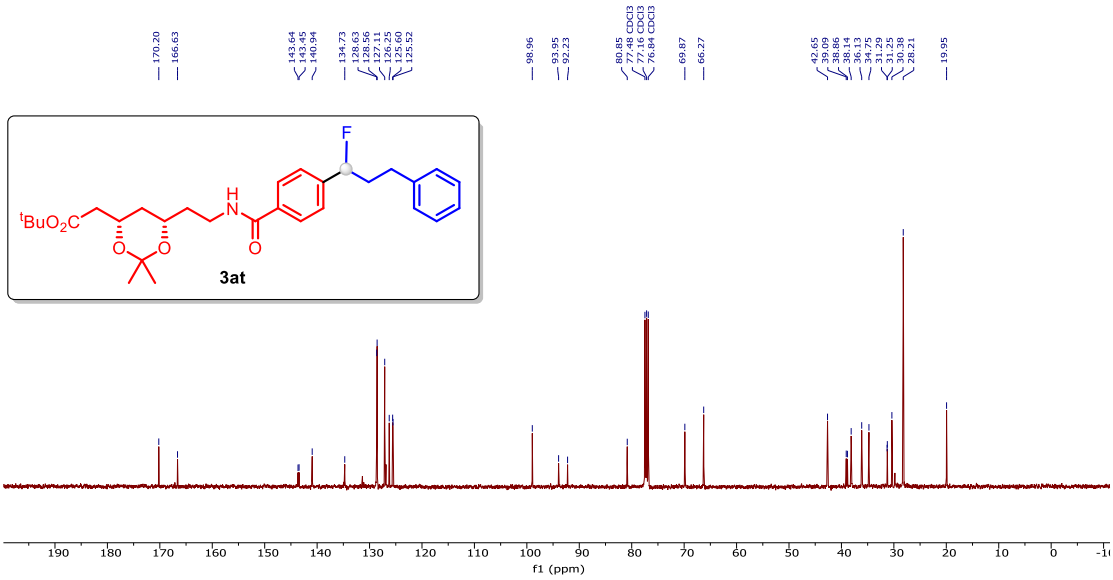
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3as**



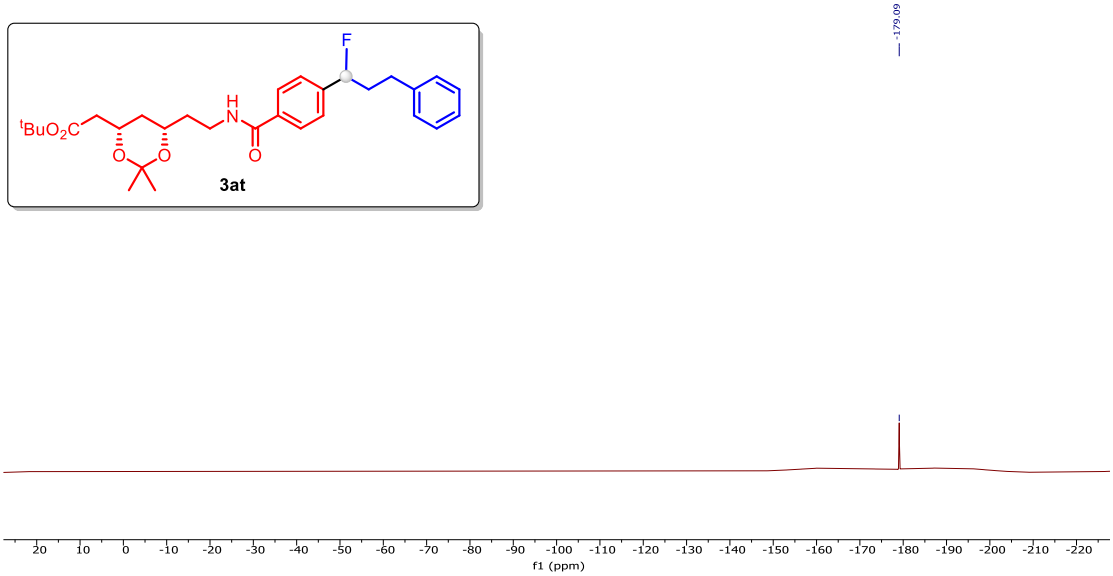
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3as**



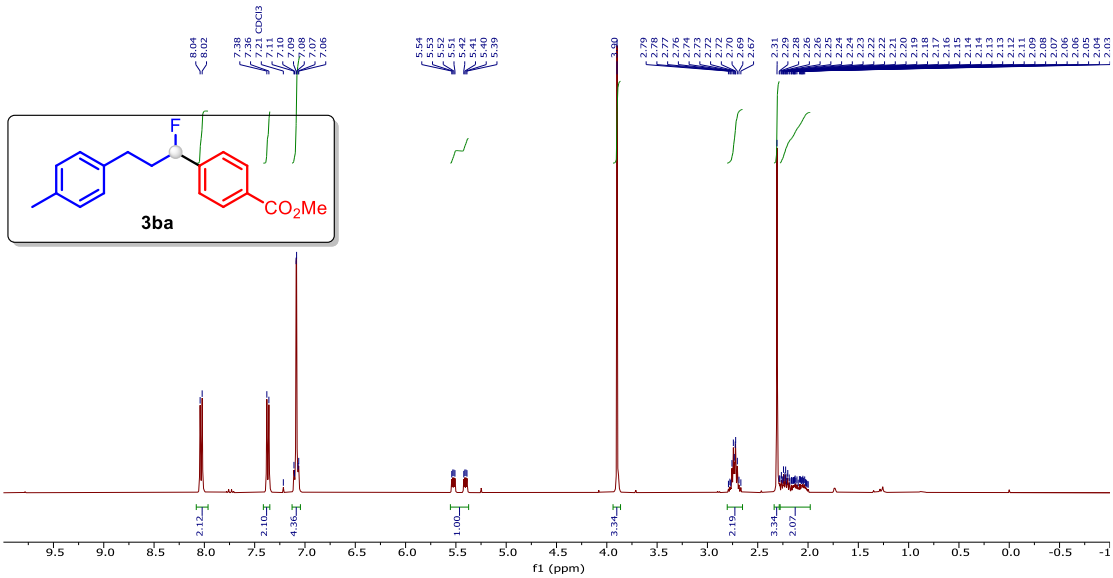
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3at**



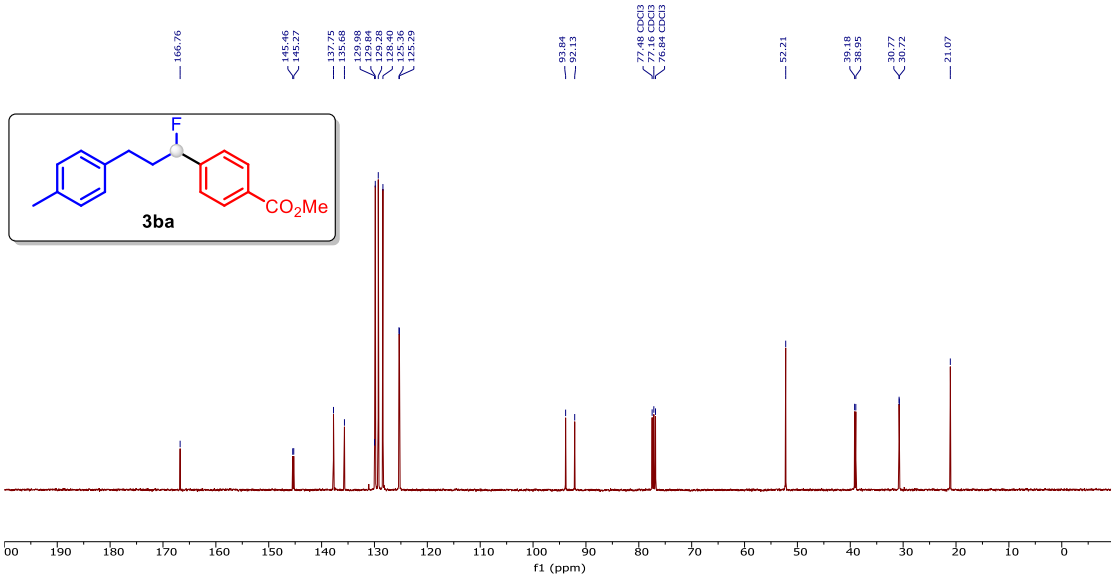
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3at**



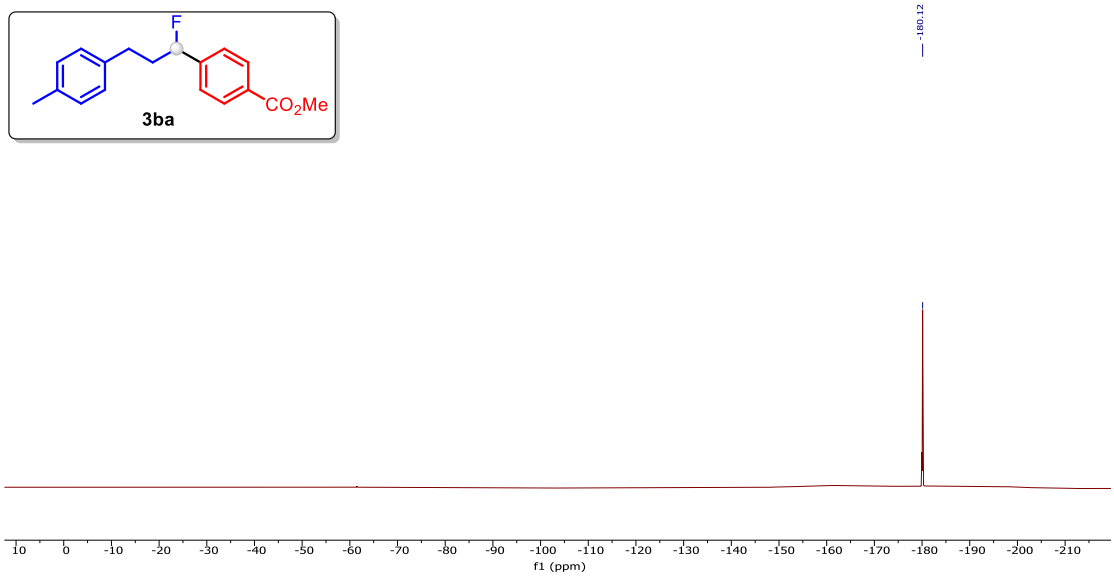
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3at**



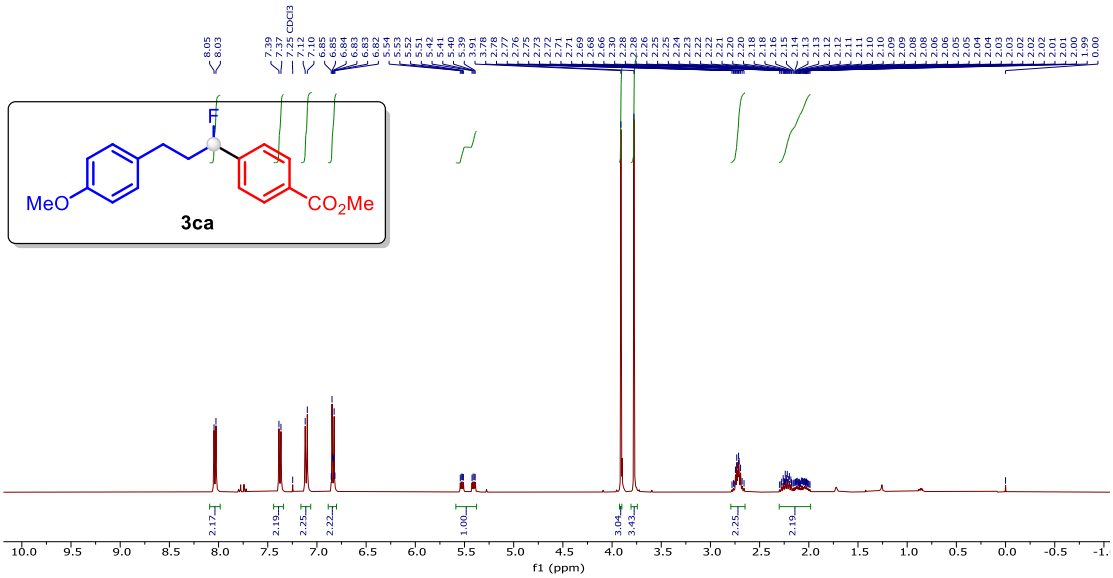
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ba**



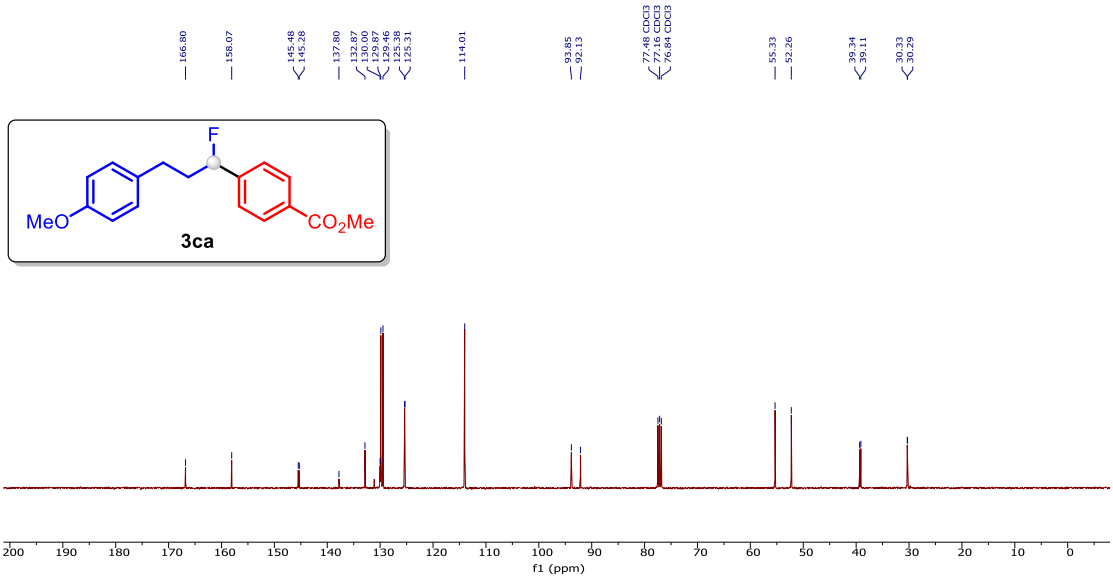
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ba**



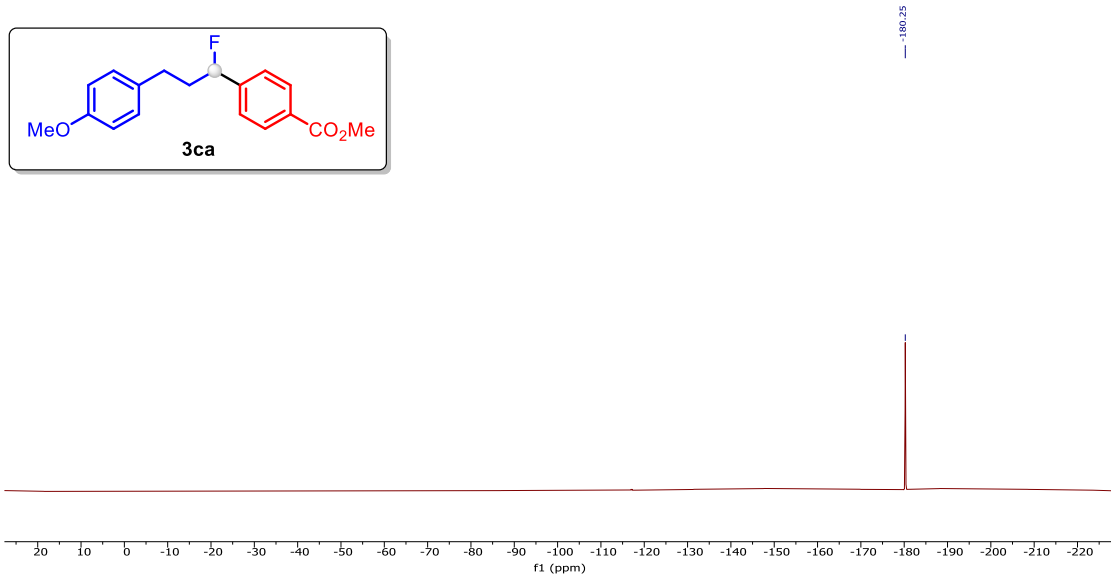
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ba**



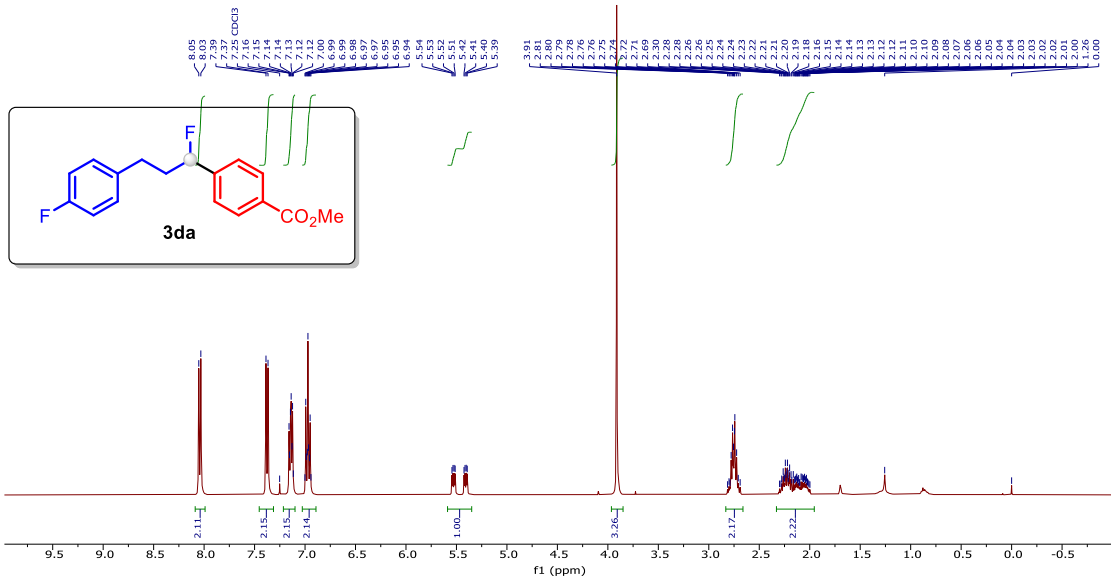
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ca**



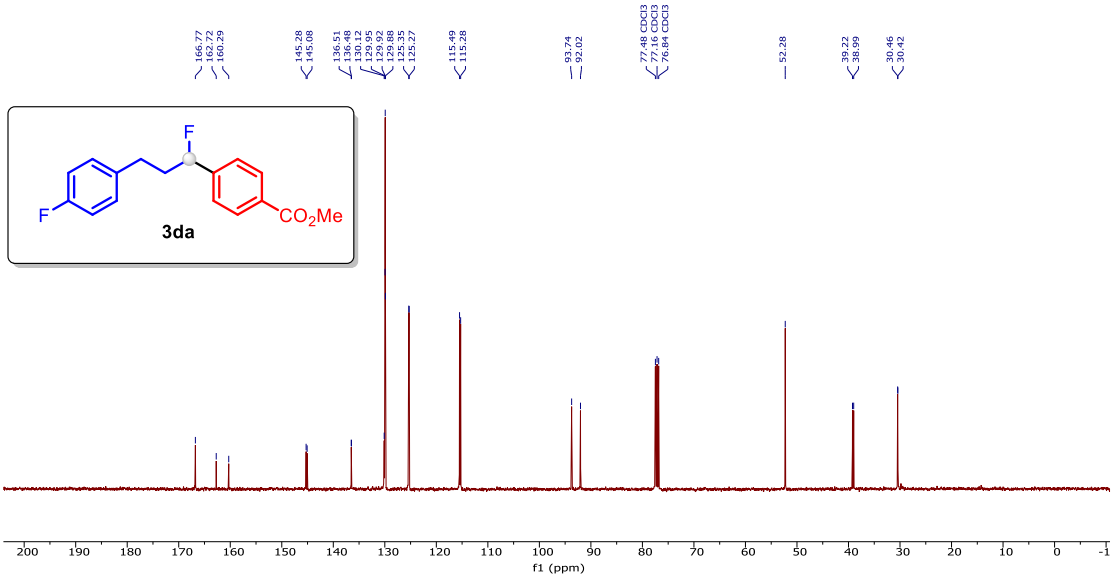
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ca**



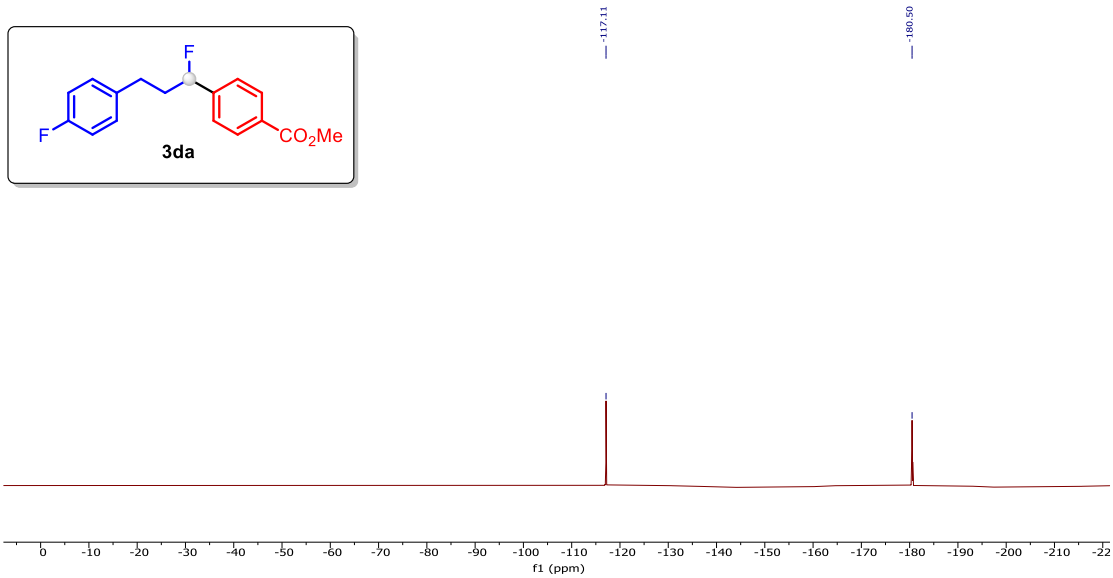
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ca**



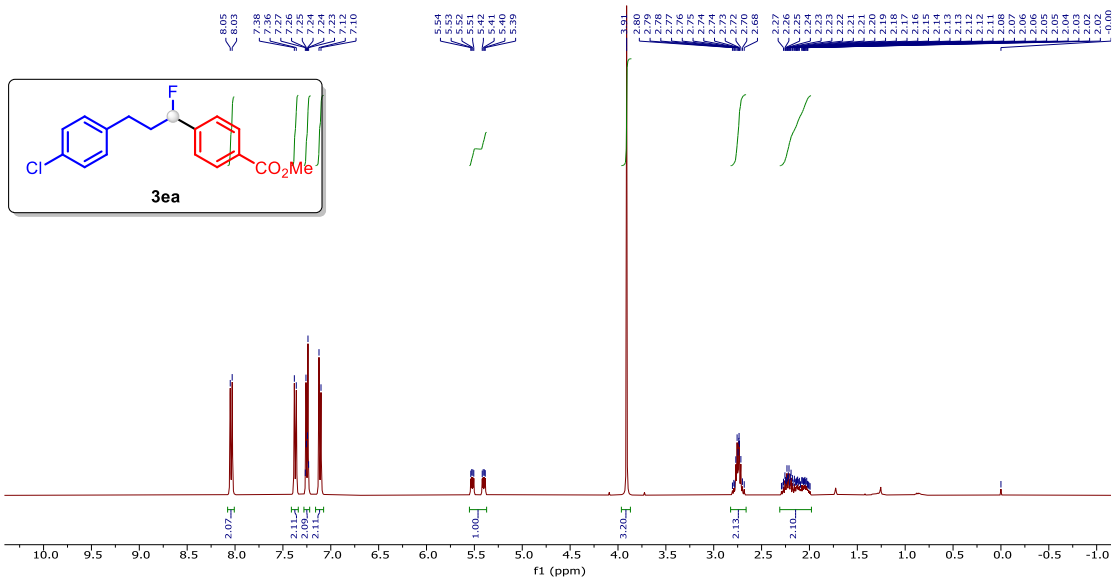
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3da**



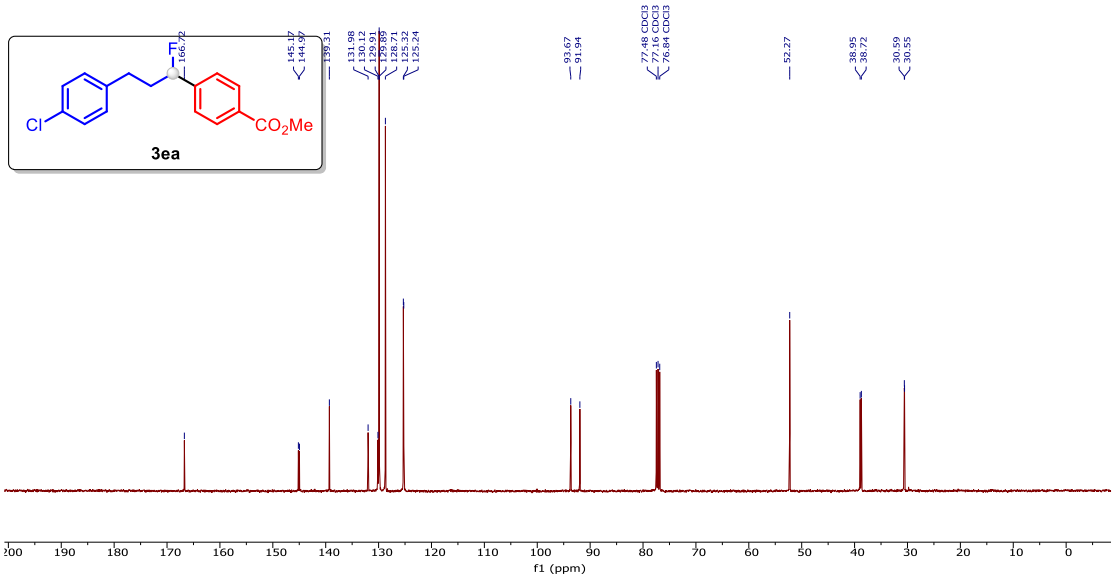
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3da**



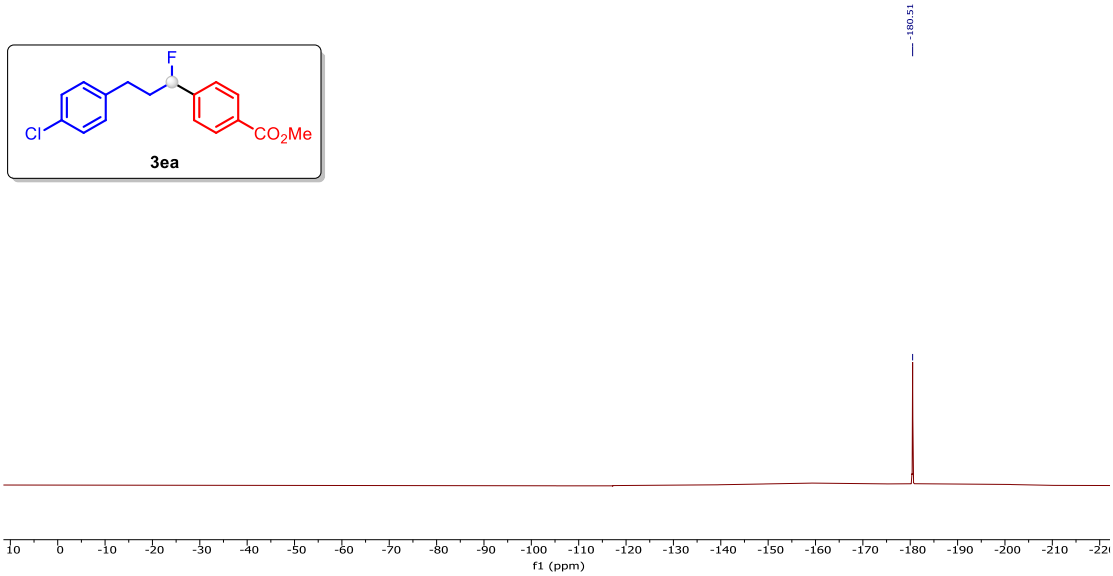
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3da**



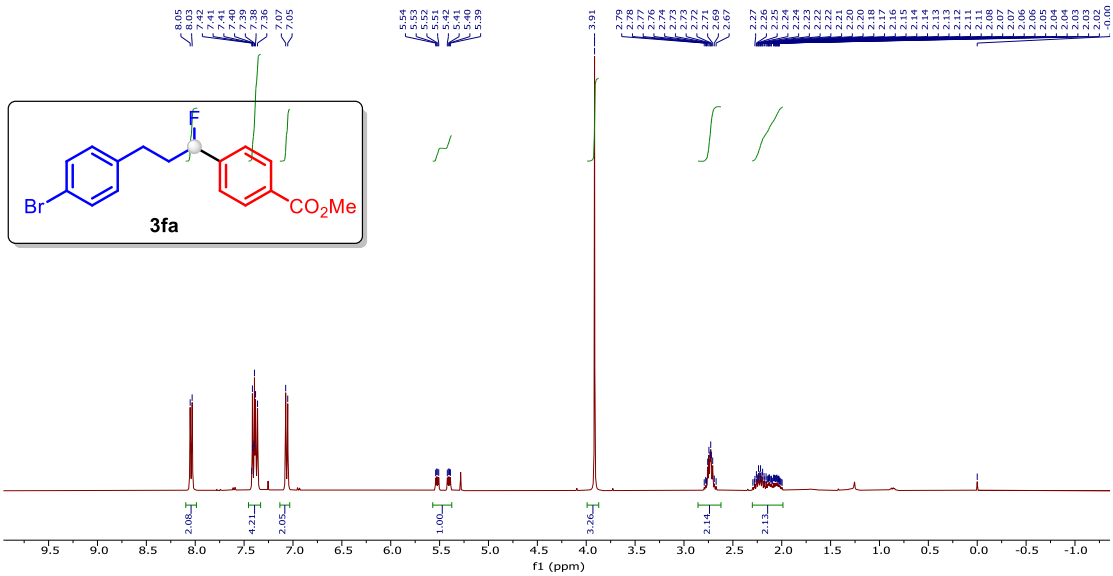
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ea**



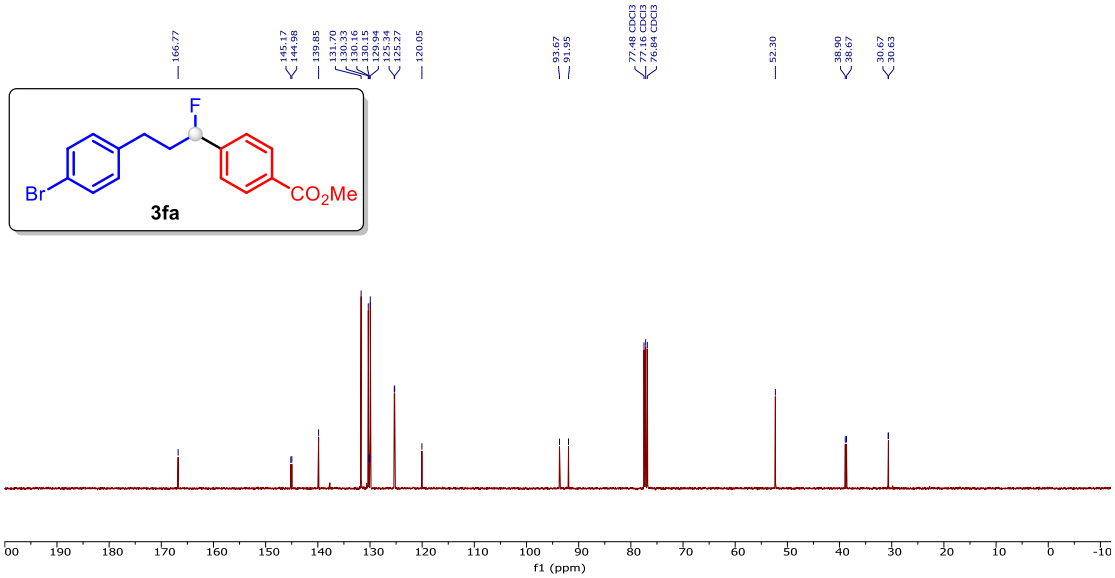
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ea**



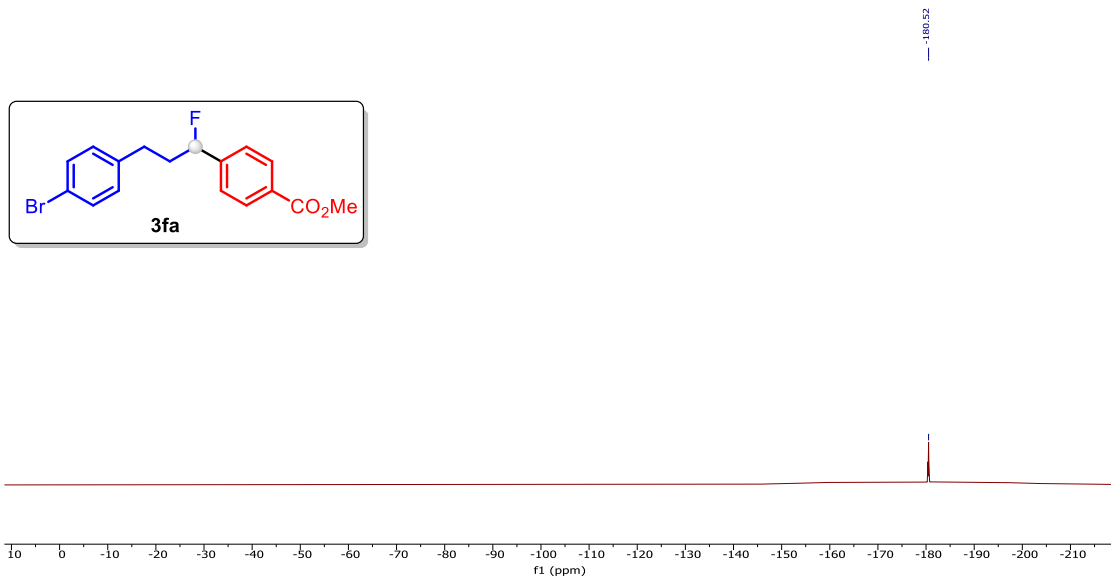
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ea**



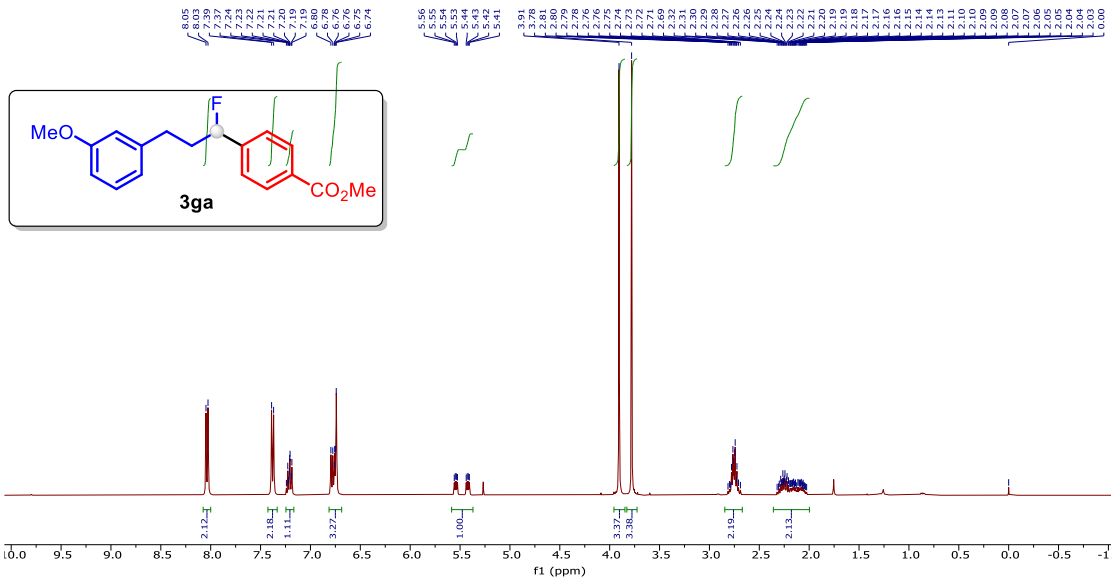
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3fa**



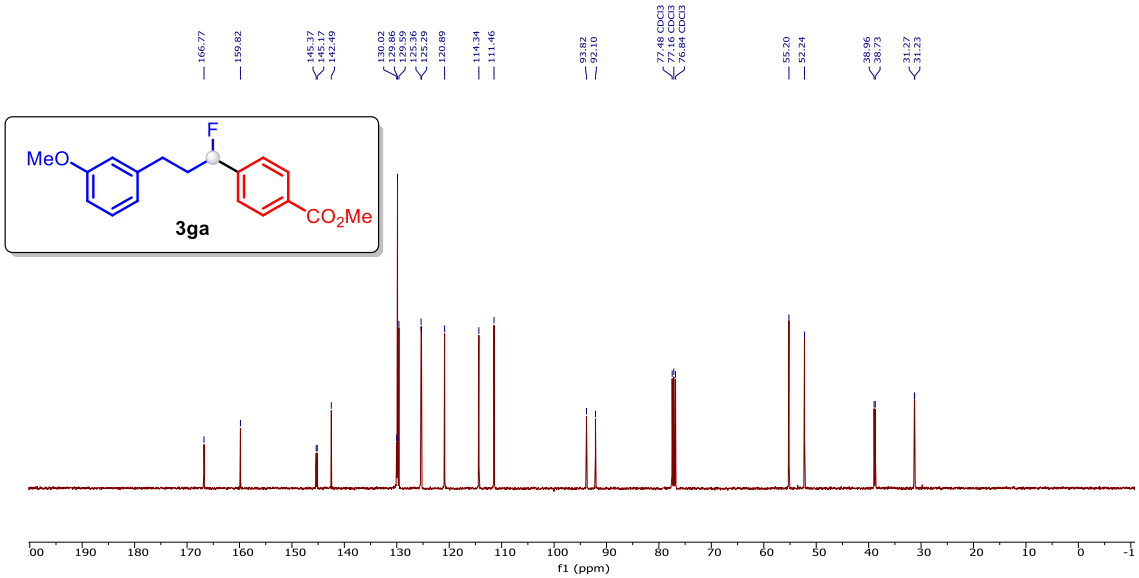
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3fa**



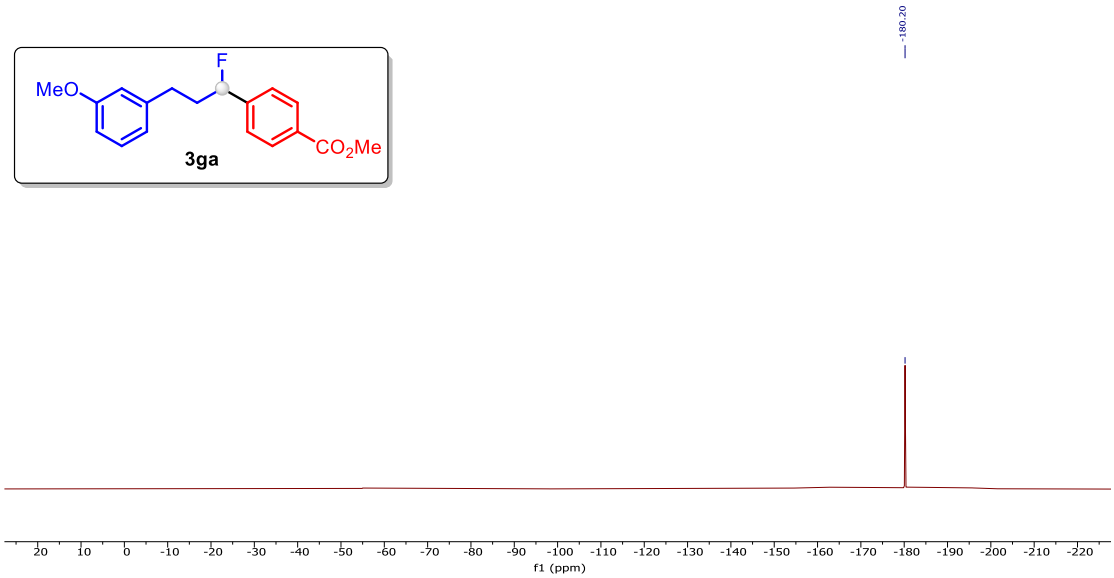
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3fa**



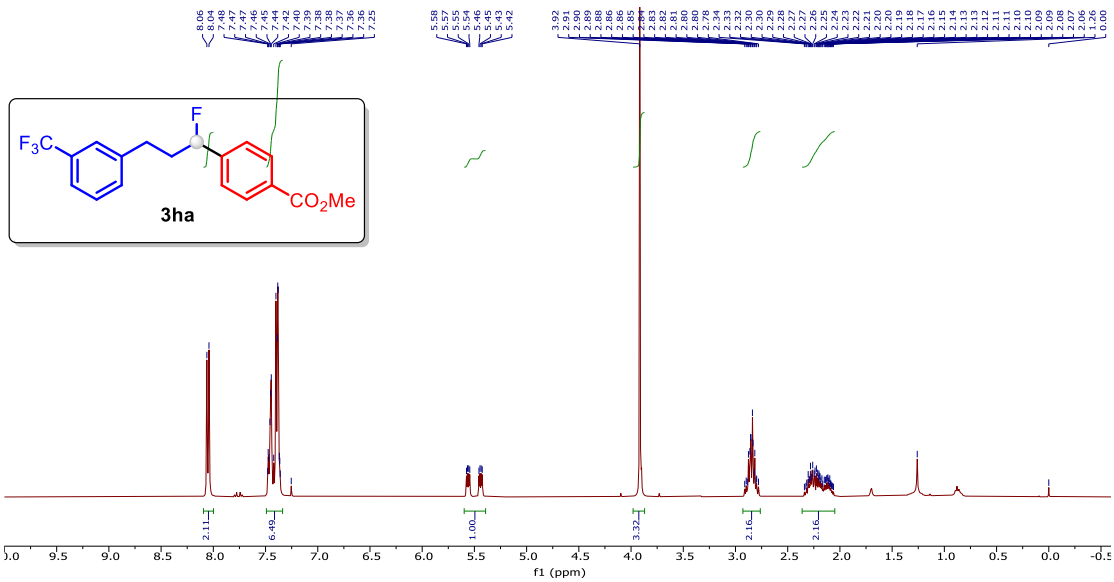
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ga**



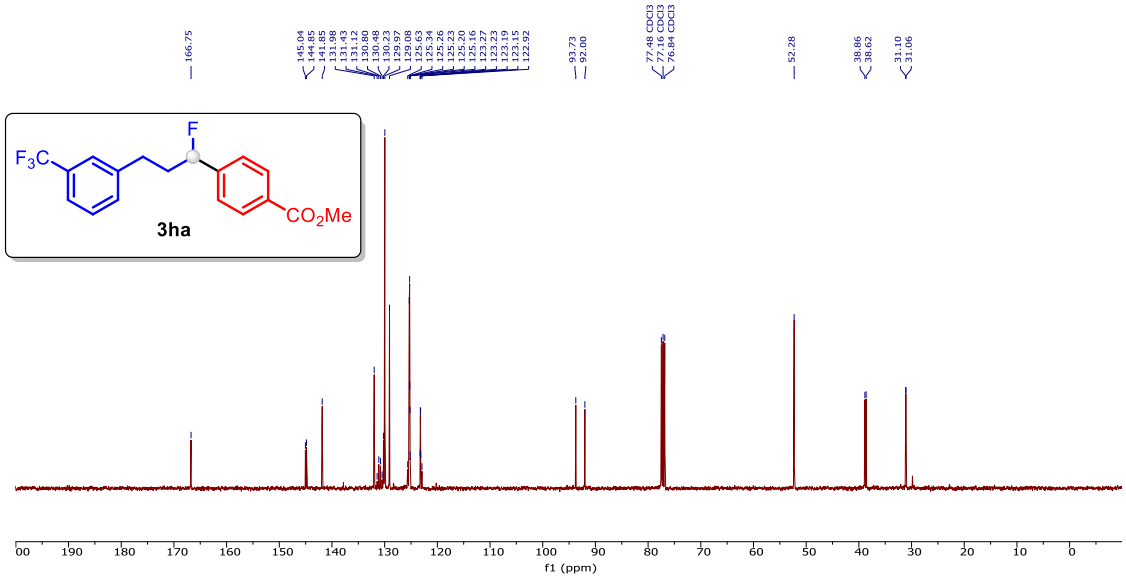
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ga**



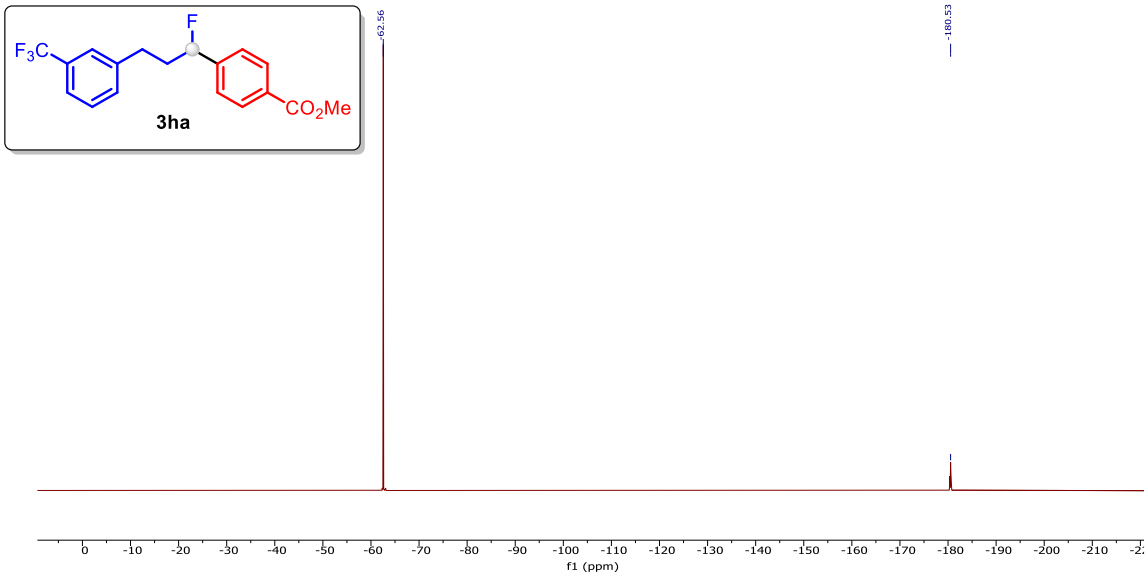
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ga**



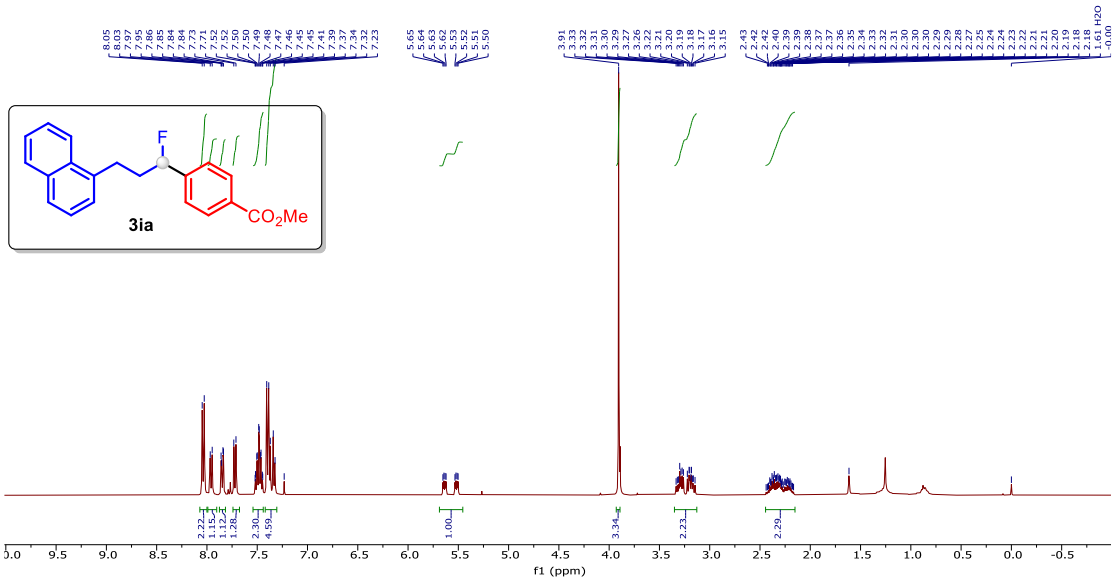
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ha**



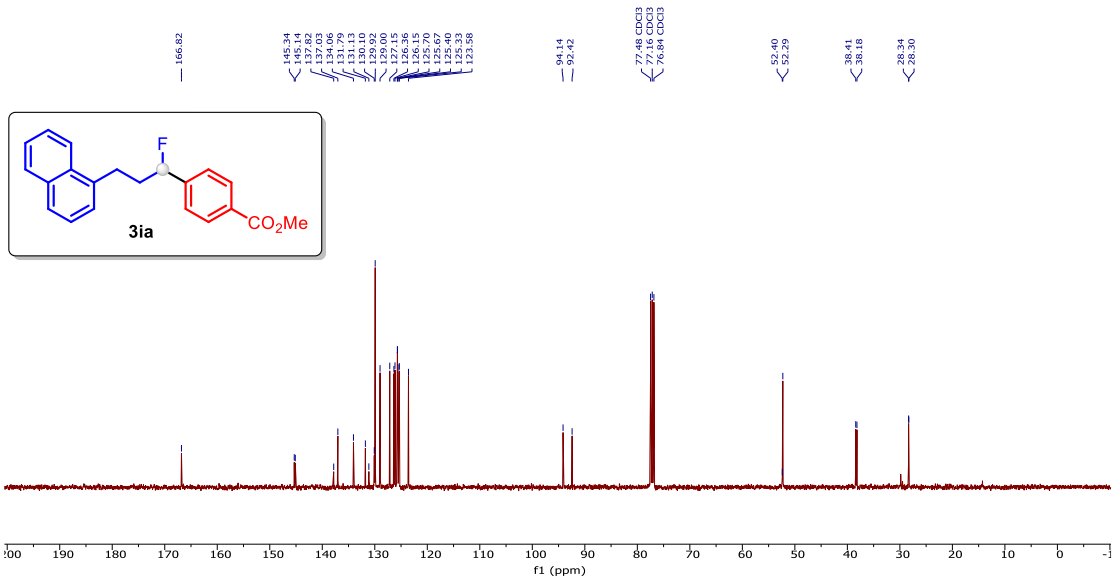
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ha**



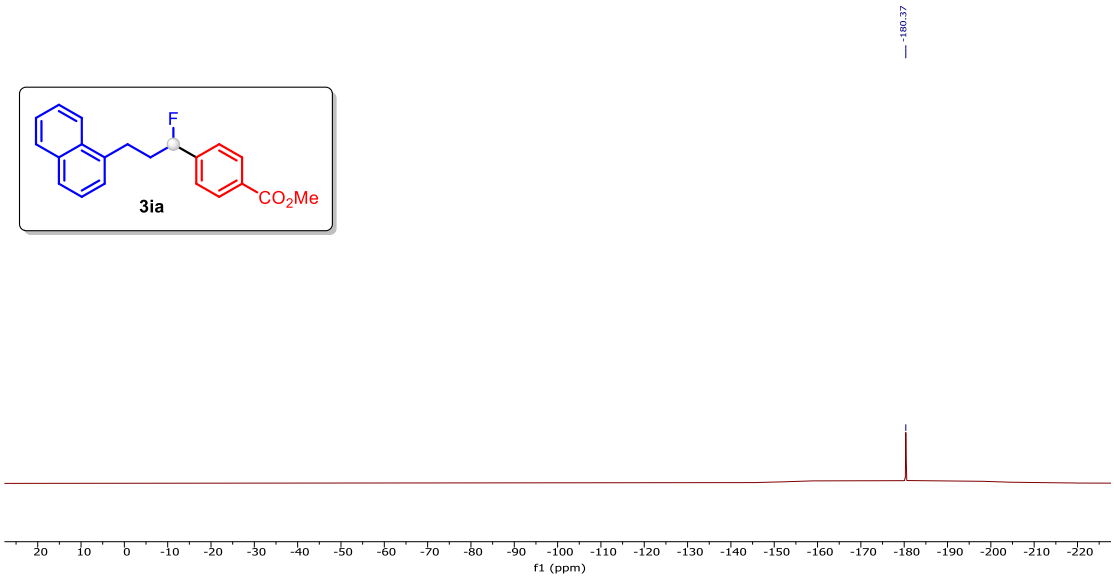
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ha**



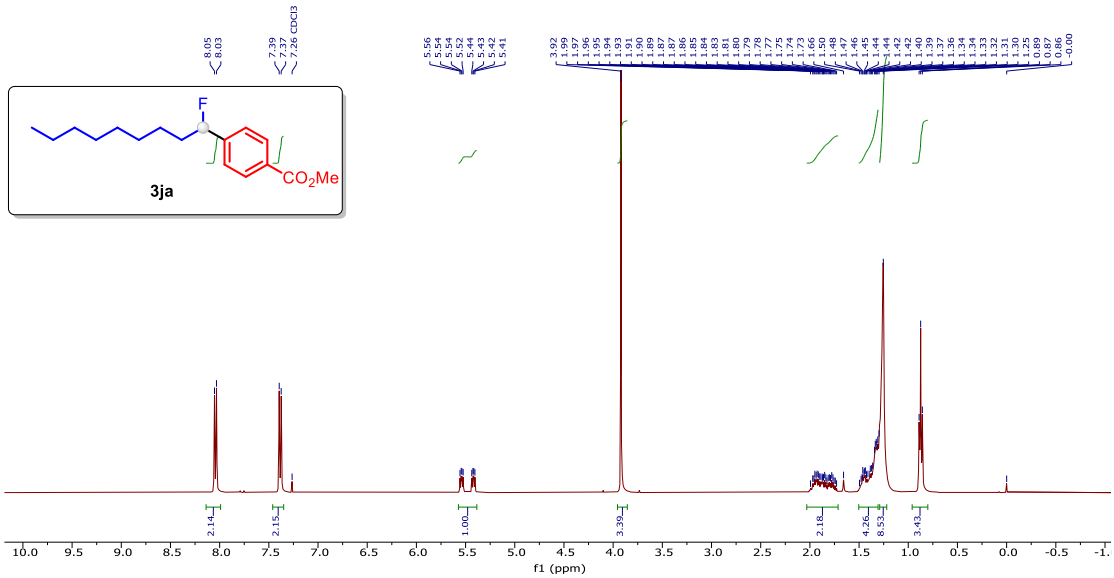
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ia**



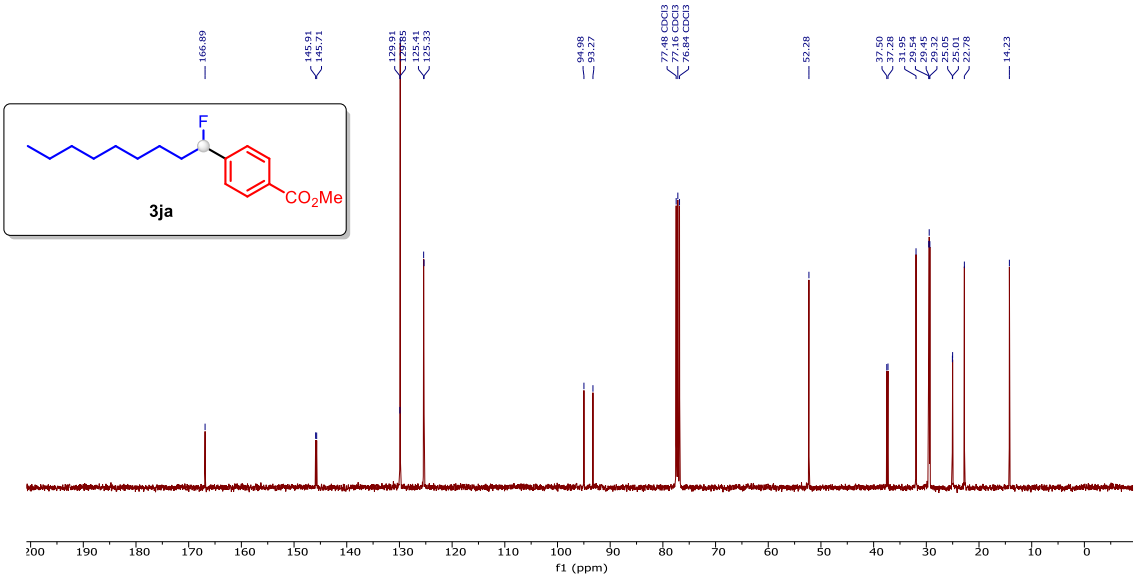
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ia**



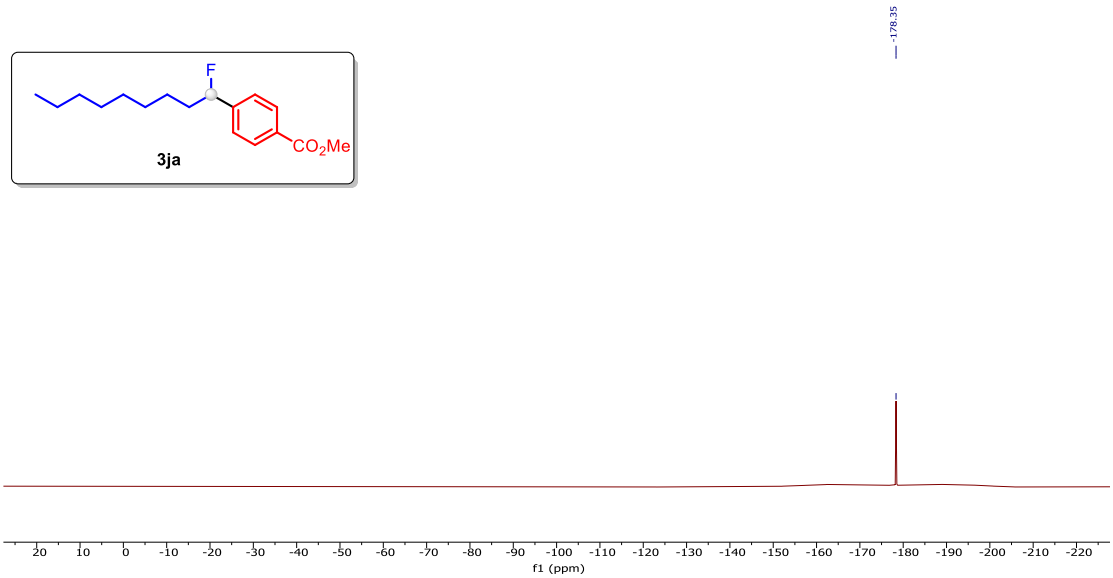
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ia**



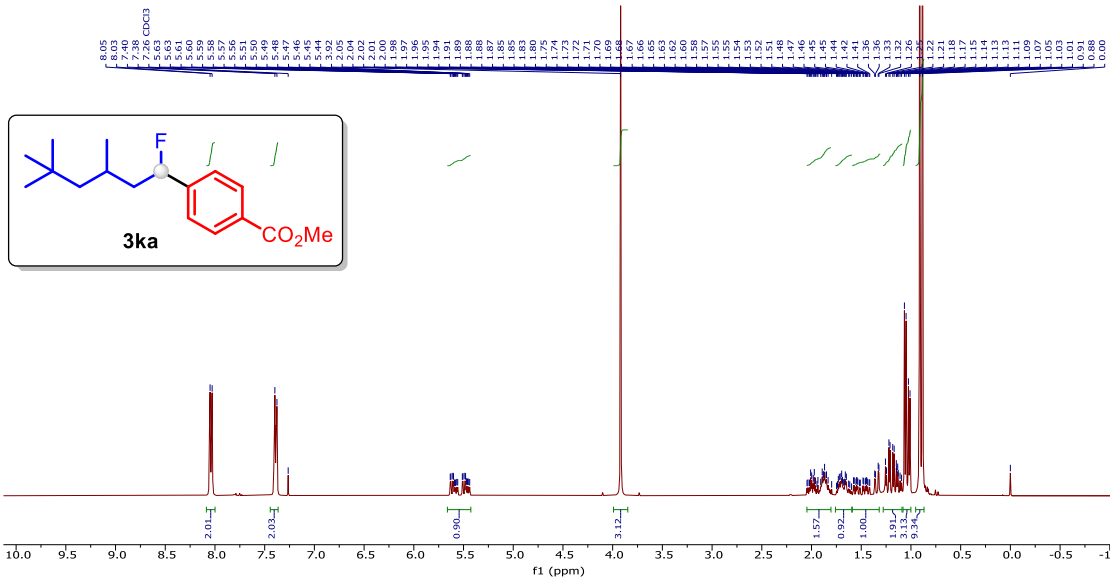
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ja**



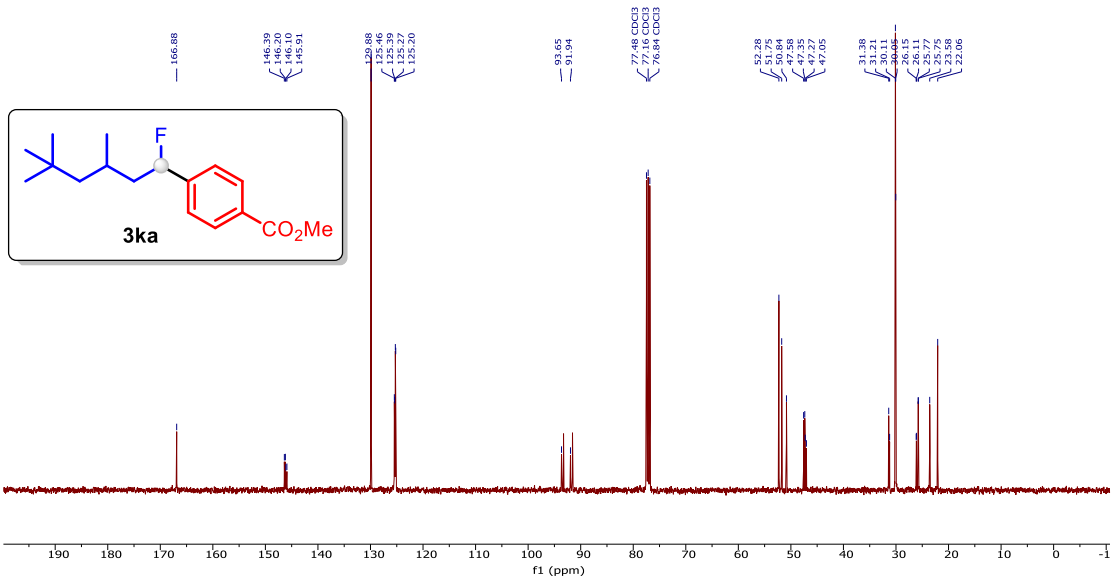
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ja**



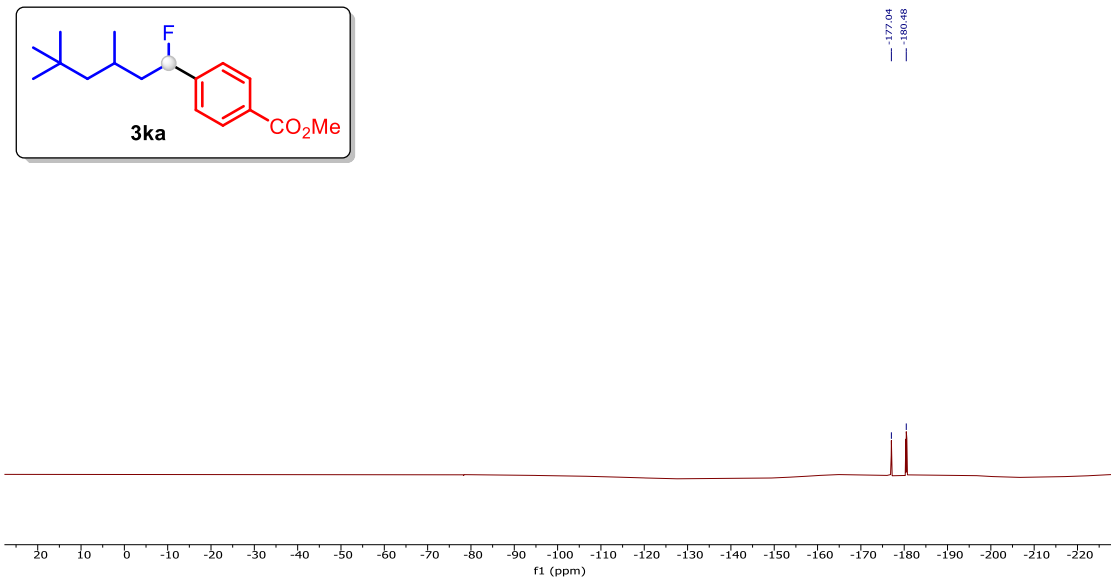
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ja**



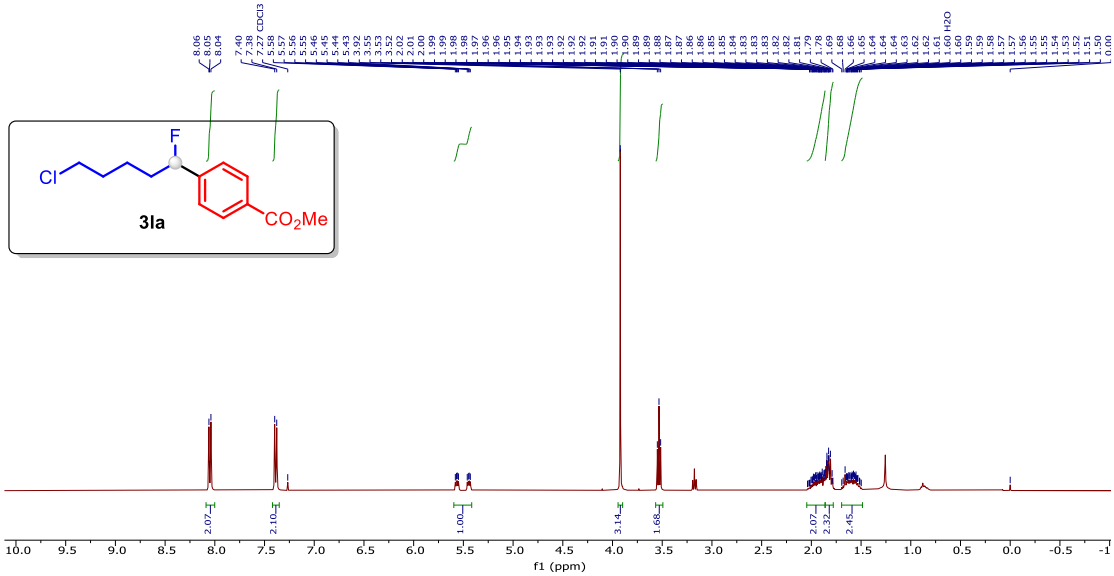
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ka**



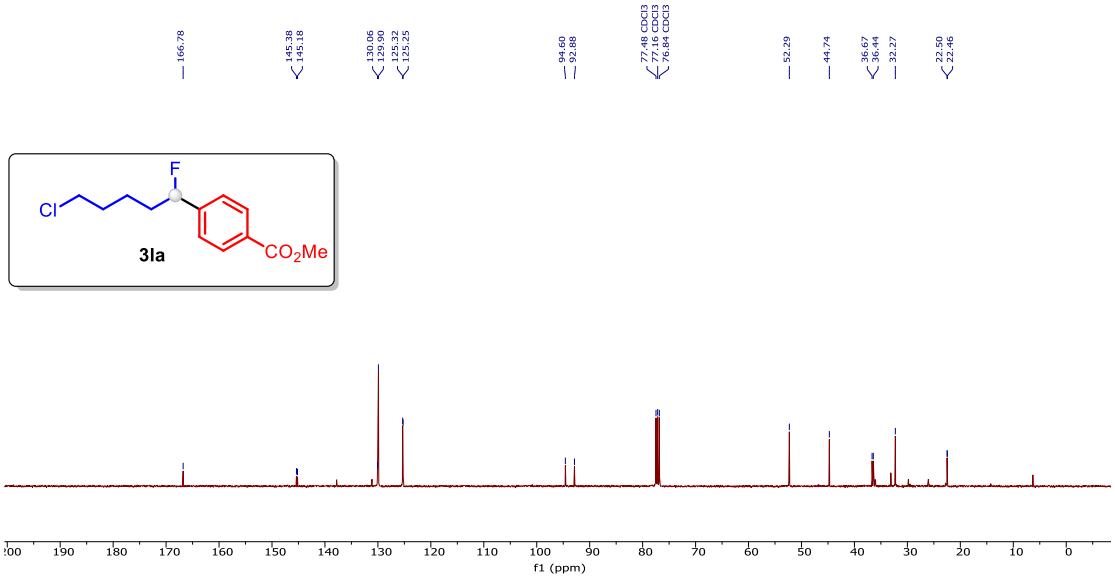
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ka**



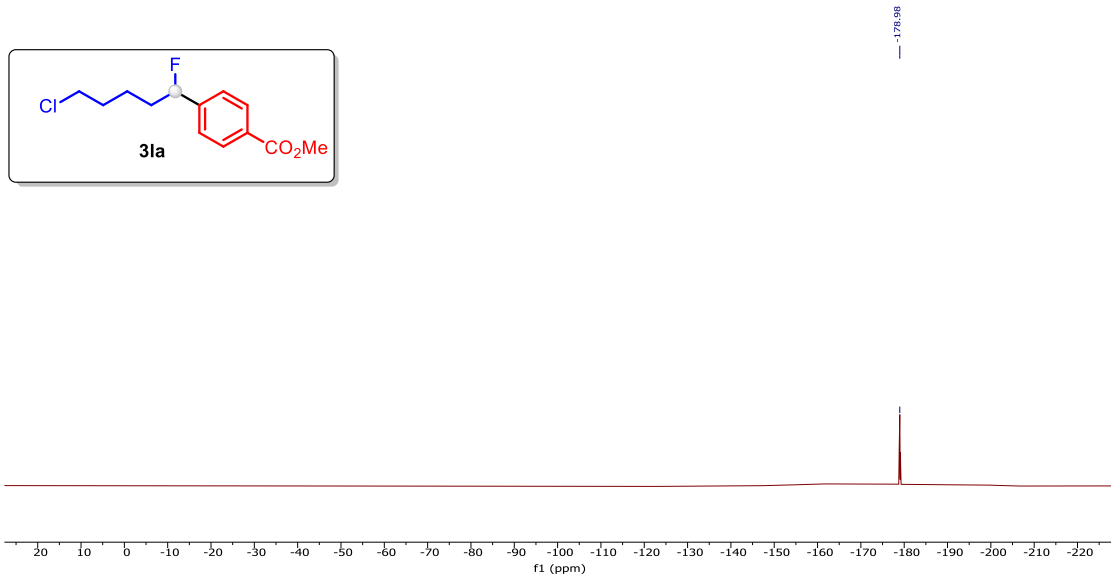
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ka**



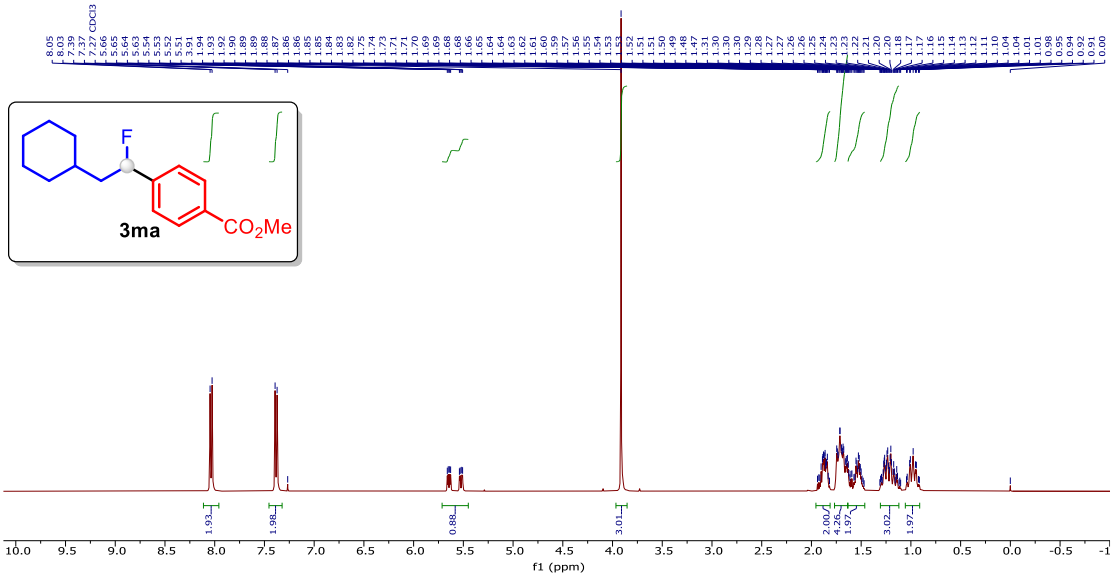
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3la**



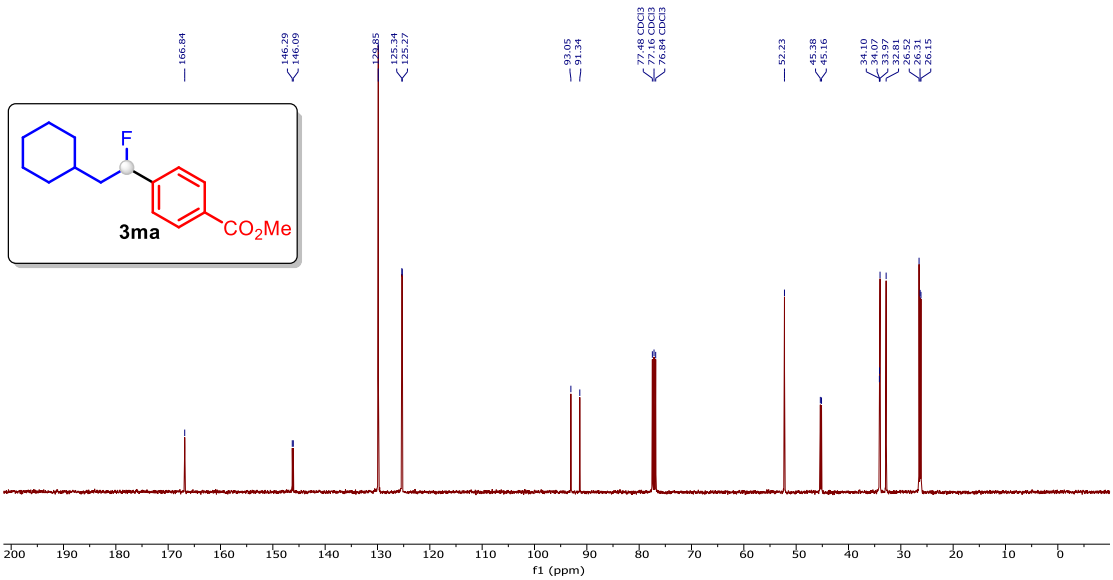
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3la**



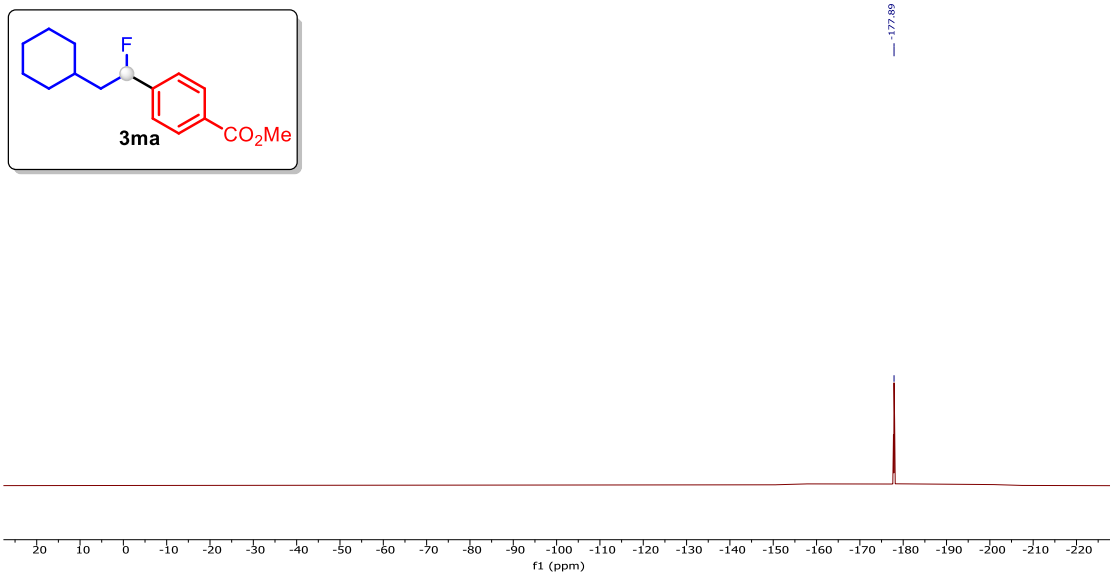
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3la**



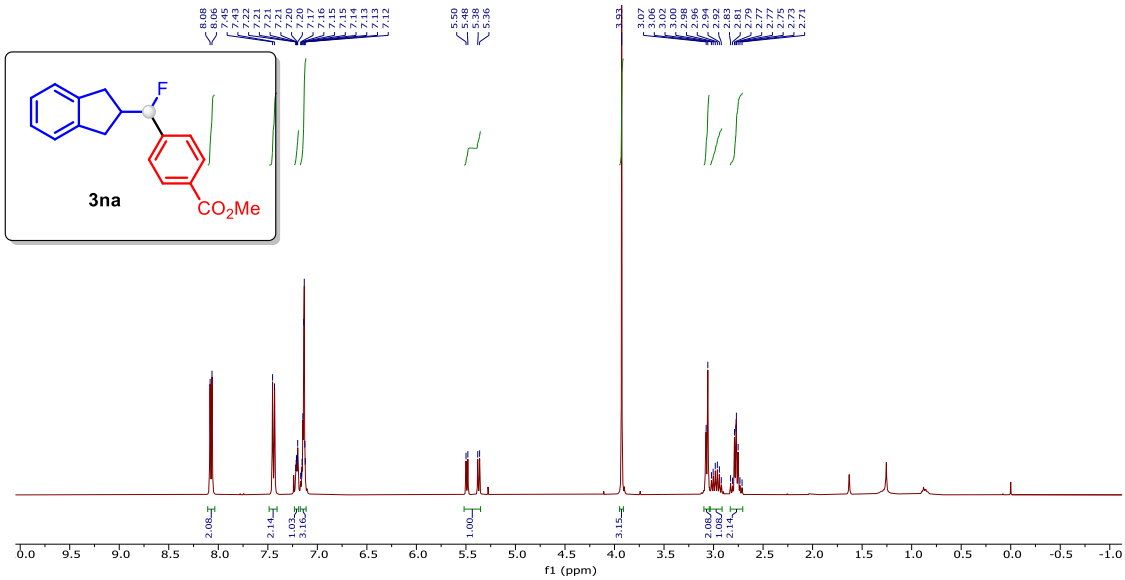
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3ma**



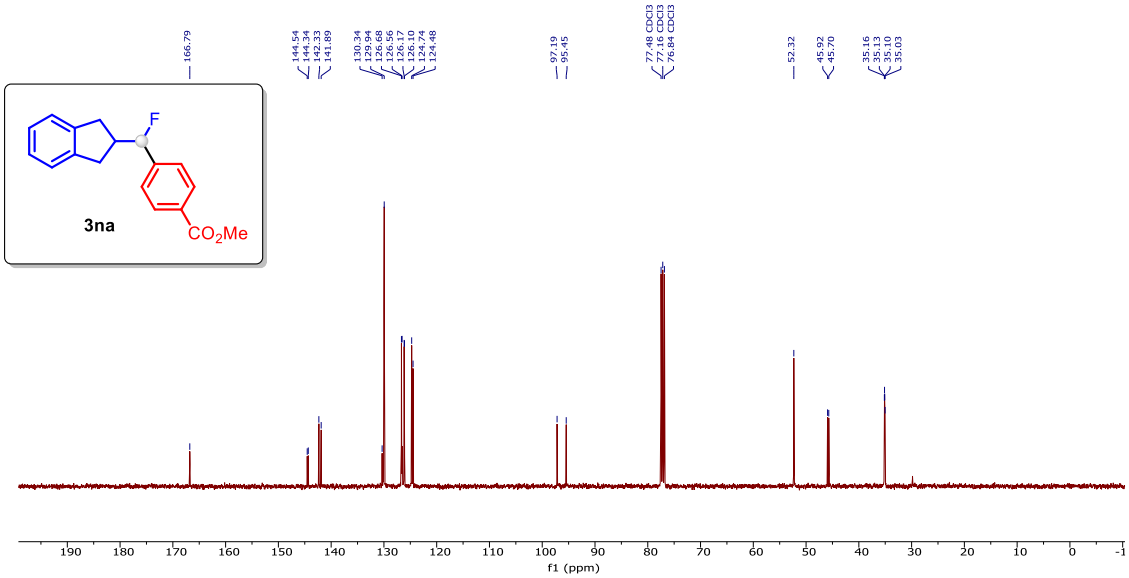
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3ma**



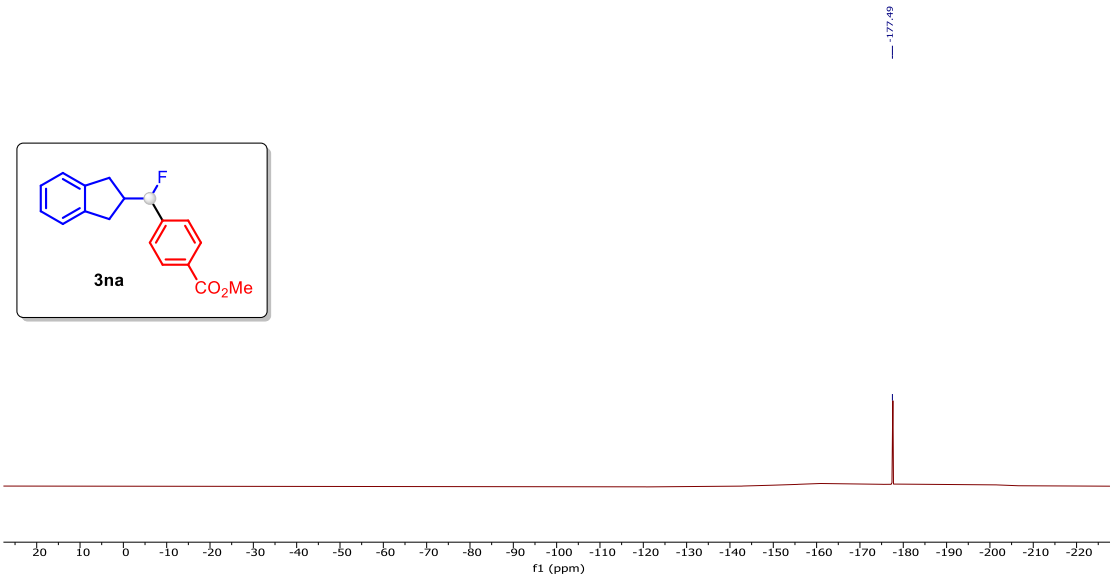
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3ma**



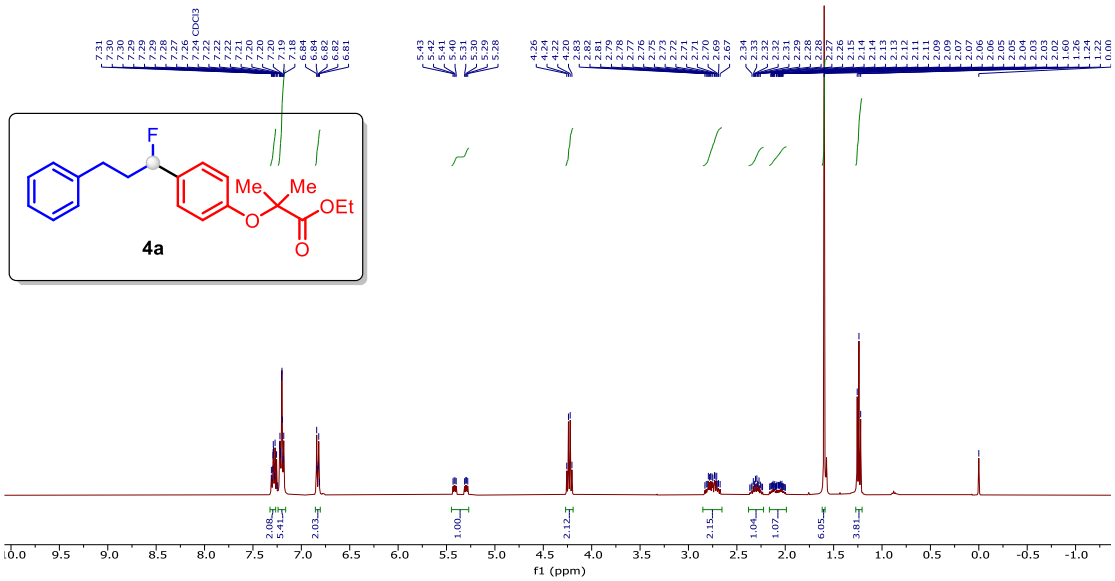
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **3na**



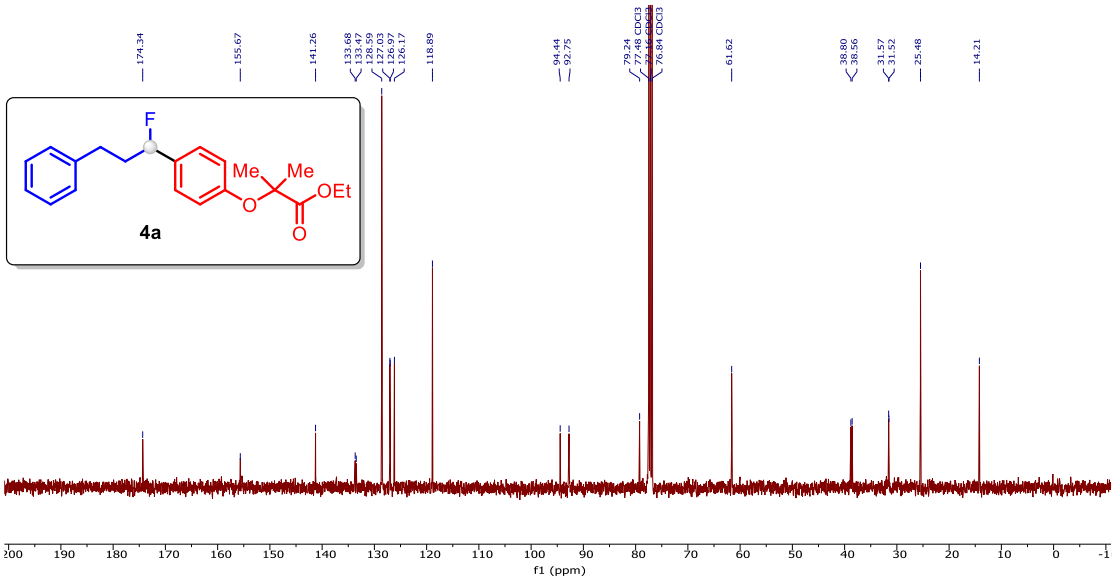
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **3na**



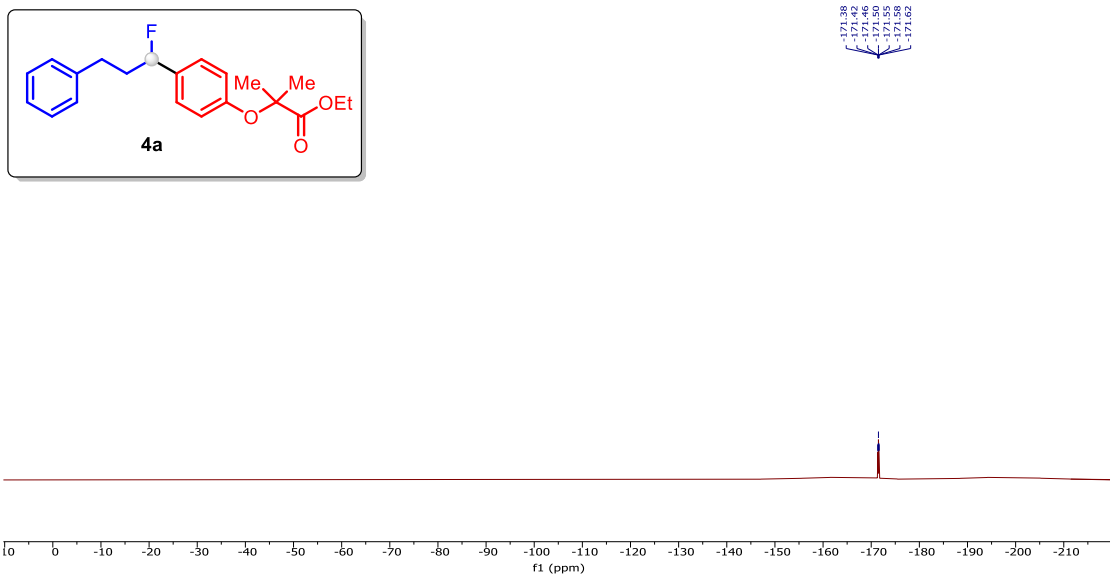
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **3na**



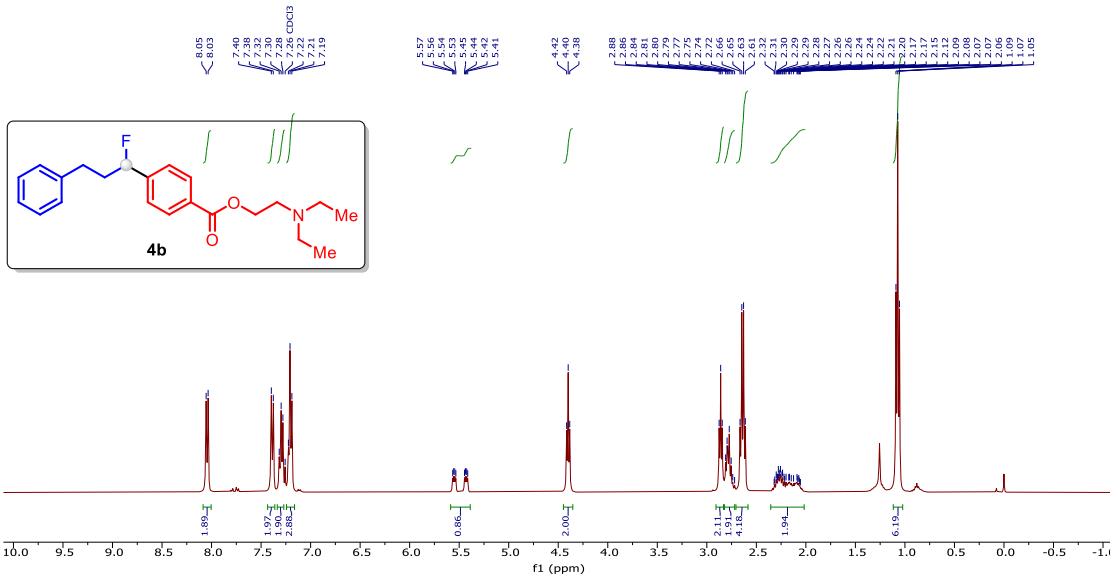
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **4a**



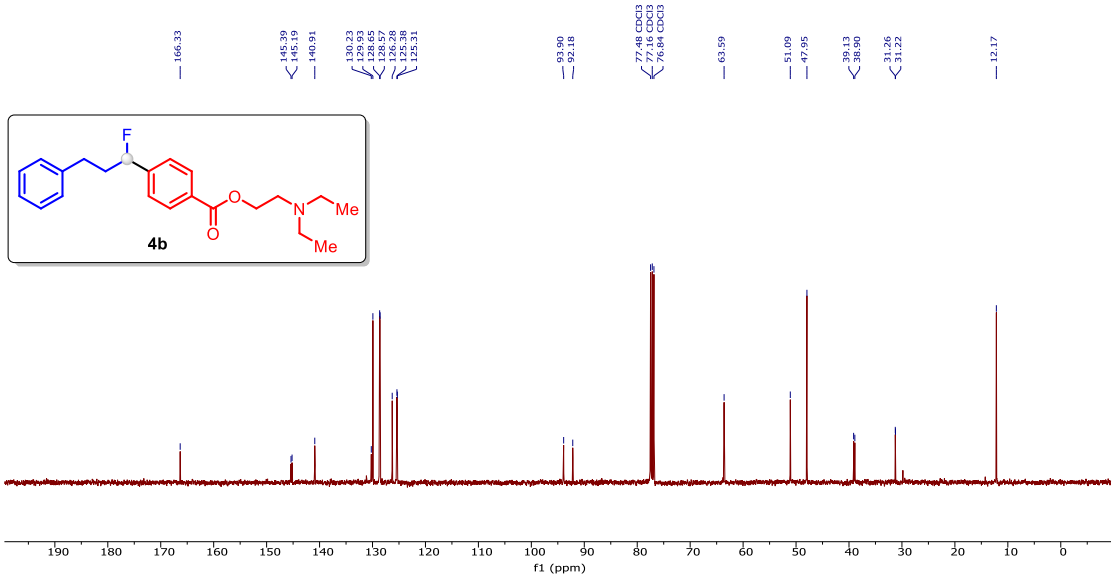
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **4a**



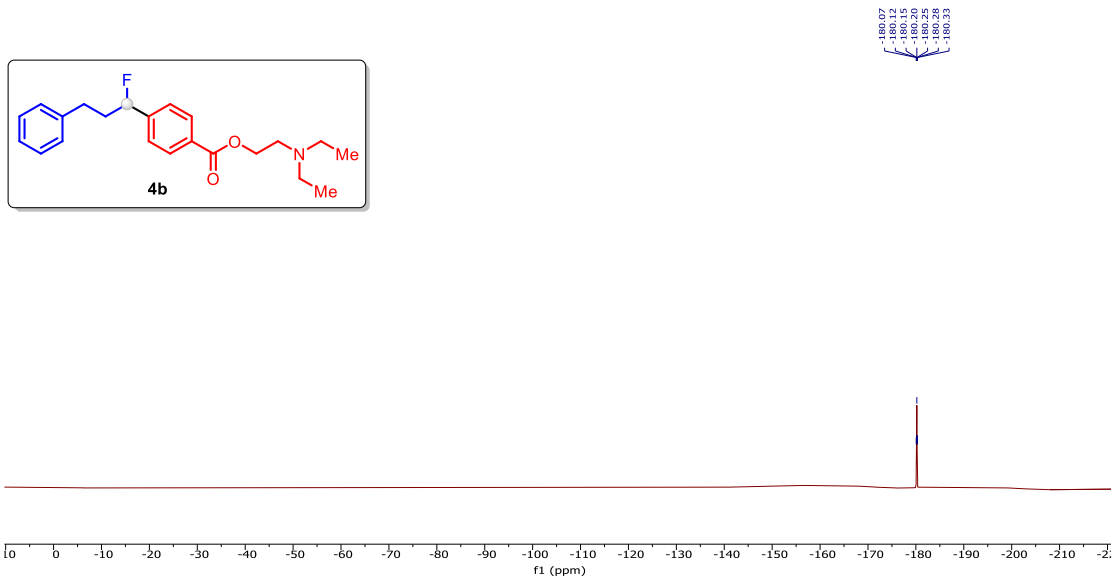
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **4a**



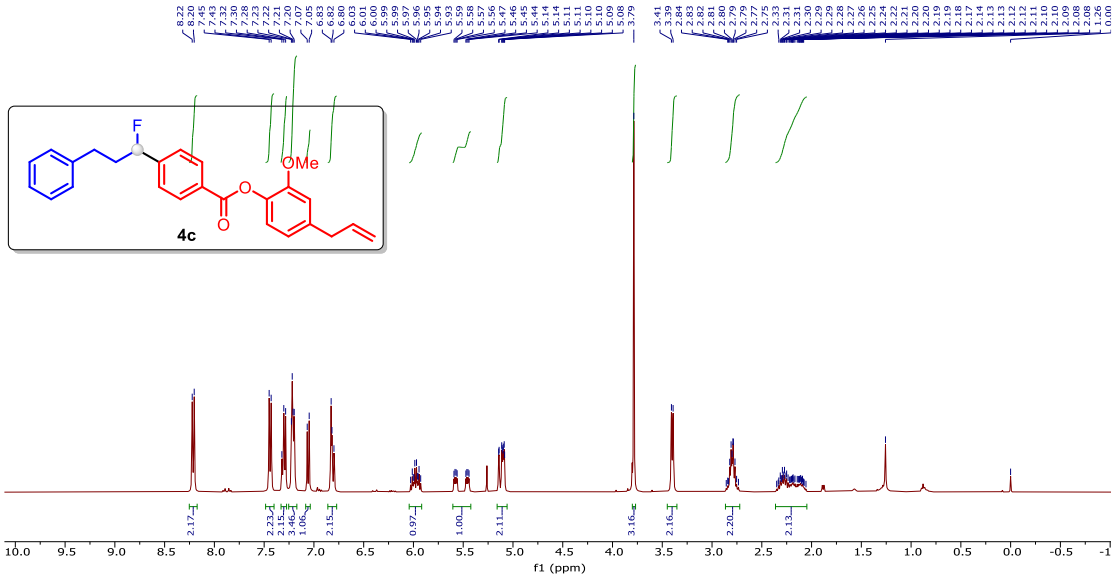
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **4b**



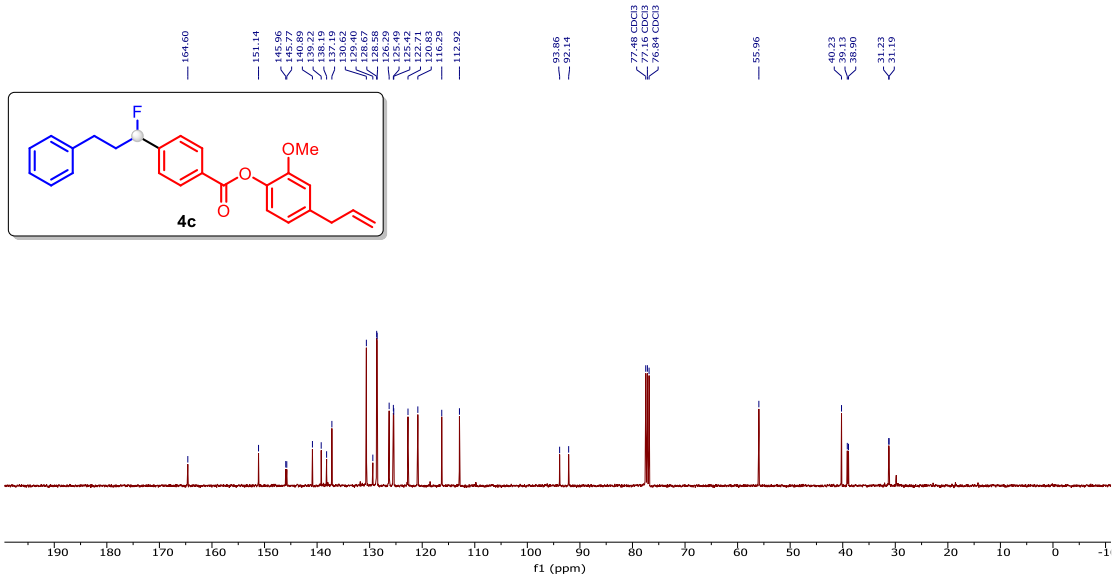
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **4b**



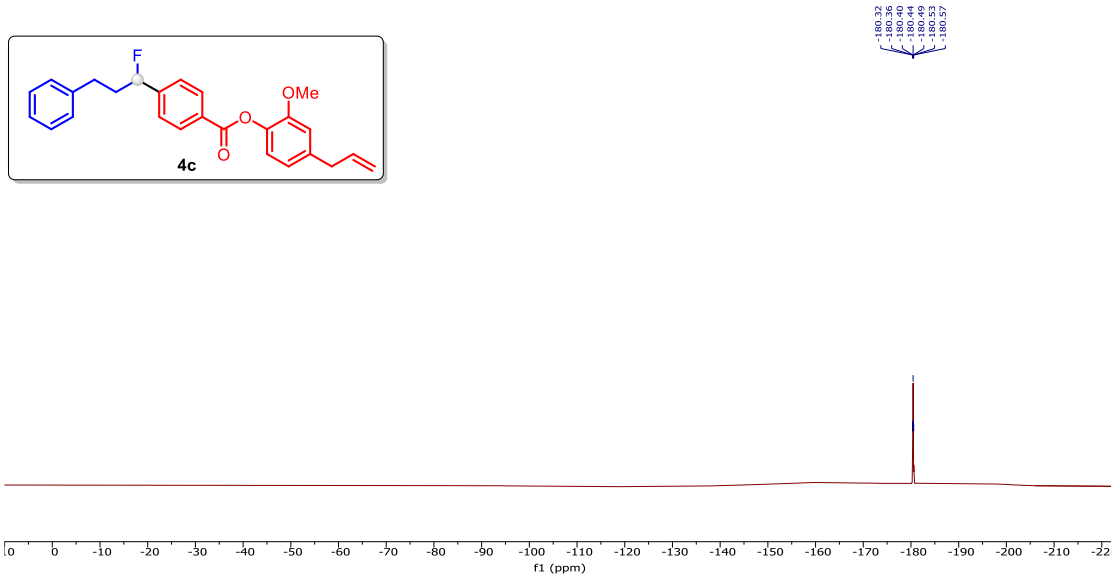
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **4b**



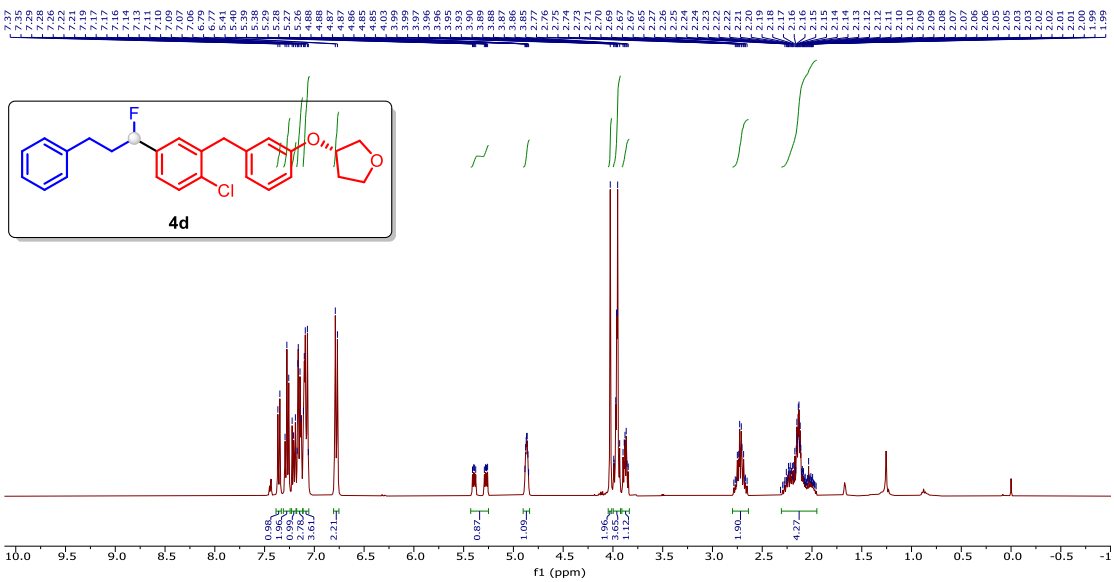
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **4c**



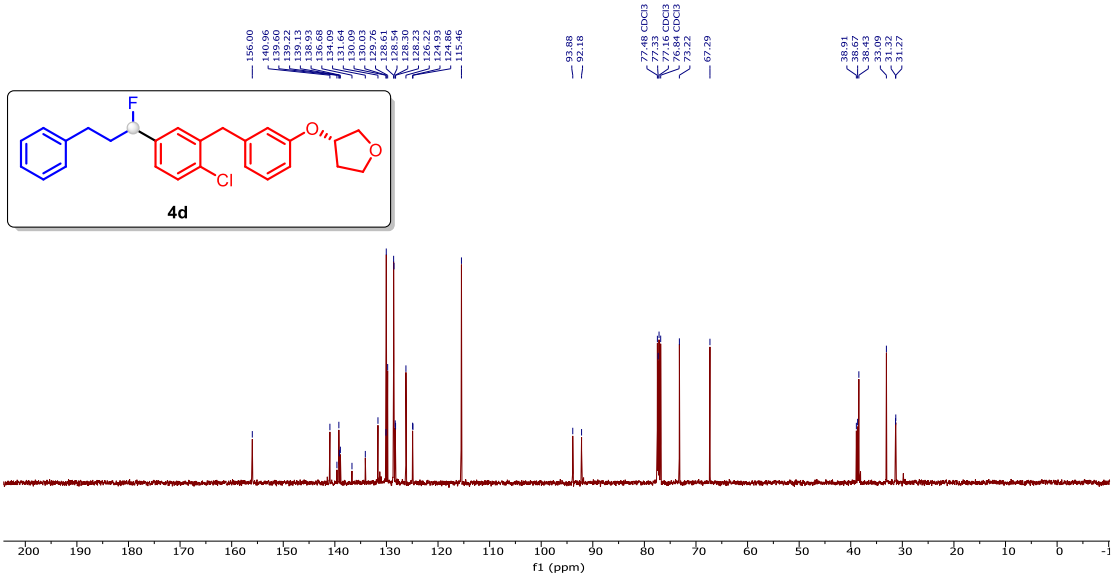
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **4c**



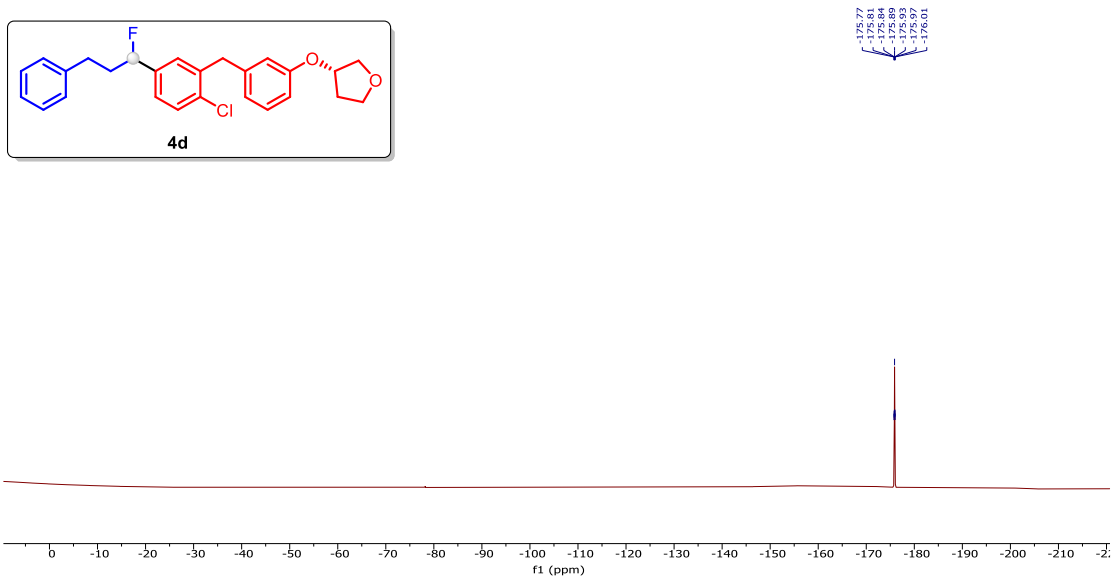
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **4c**



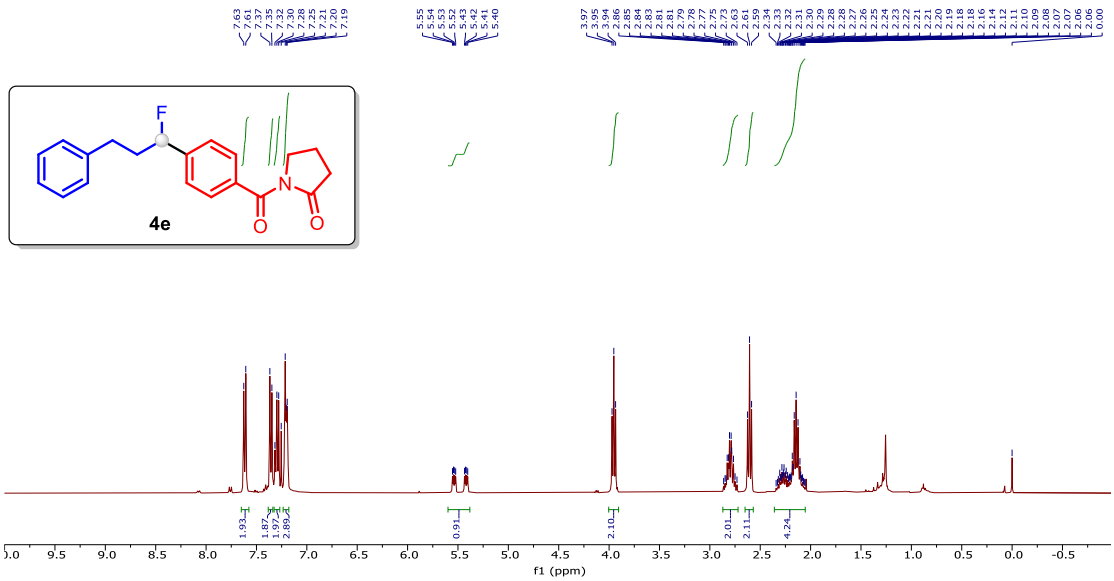
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **4d**



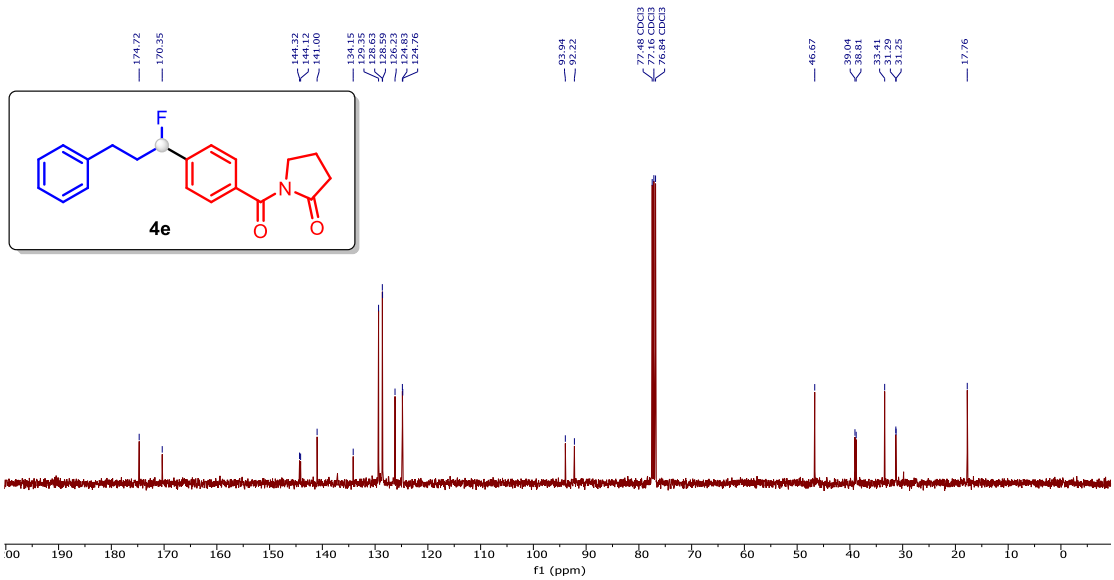
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **4d**



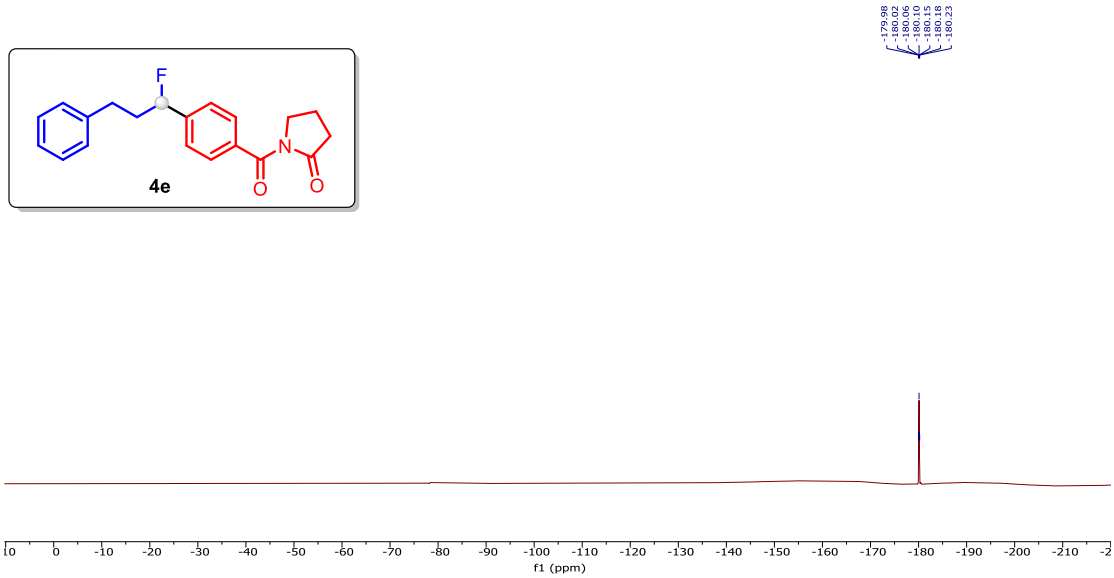
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **4d**



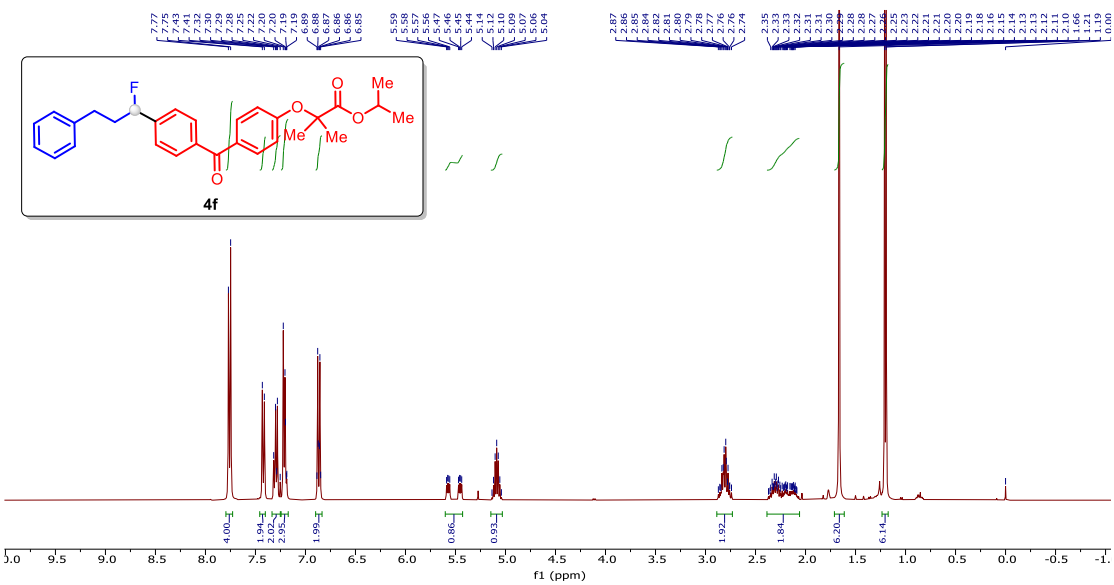
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **4e**



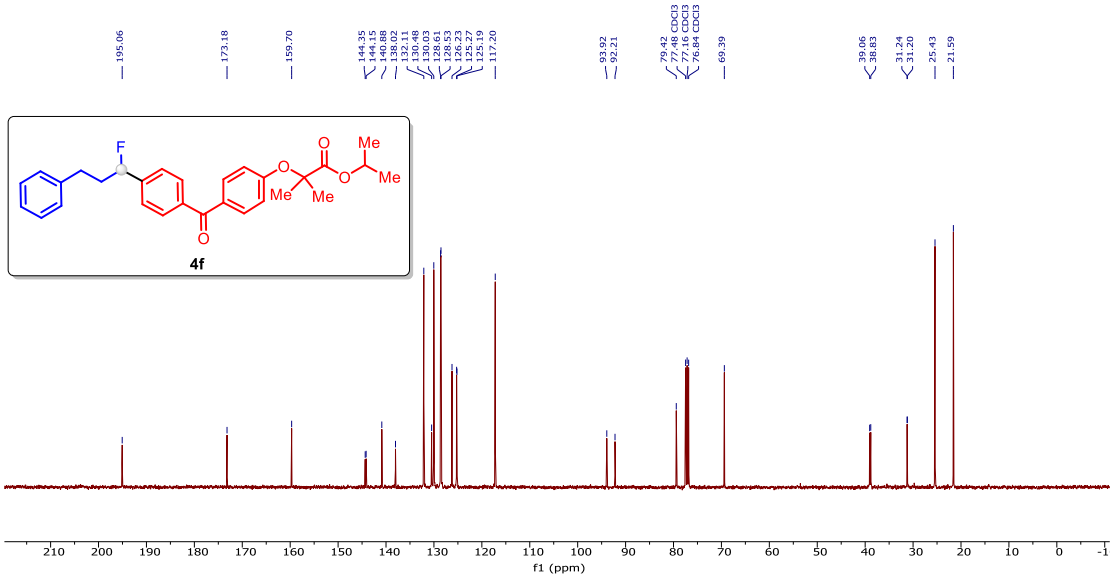
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **4e**



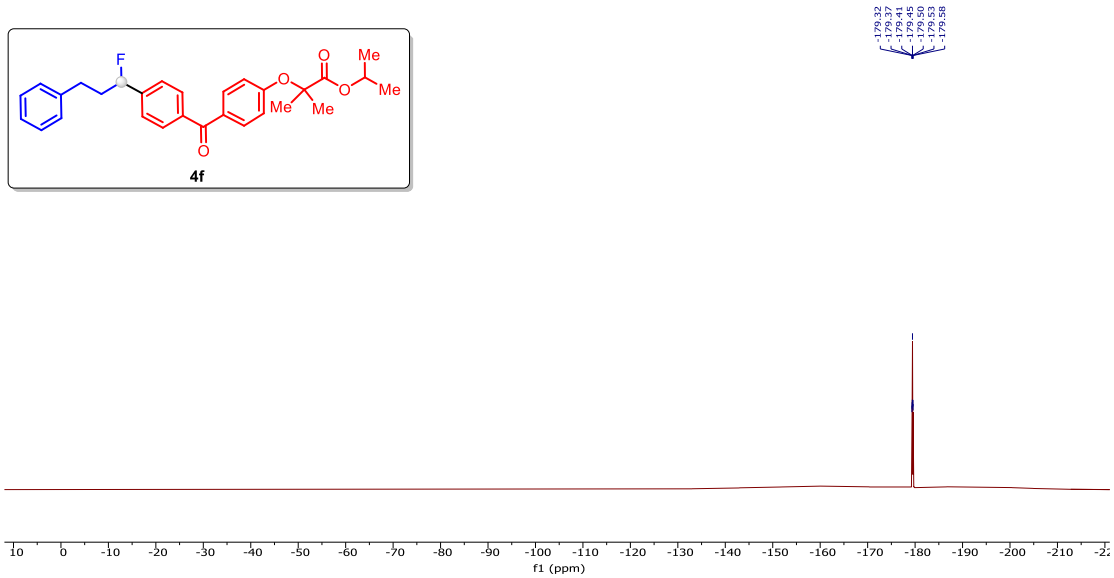
^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **4e**



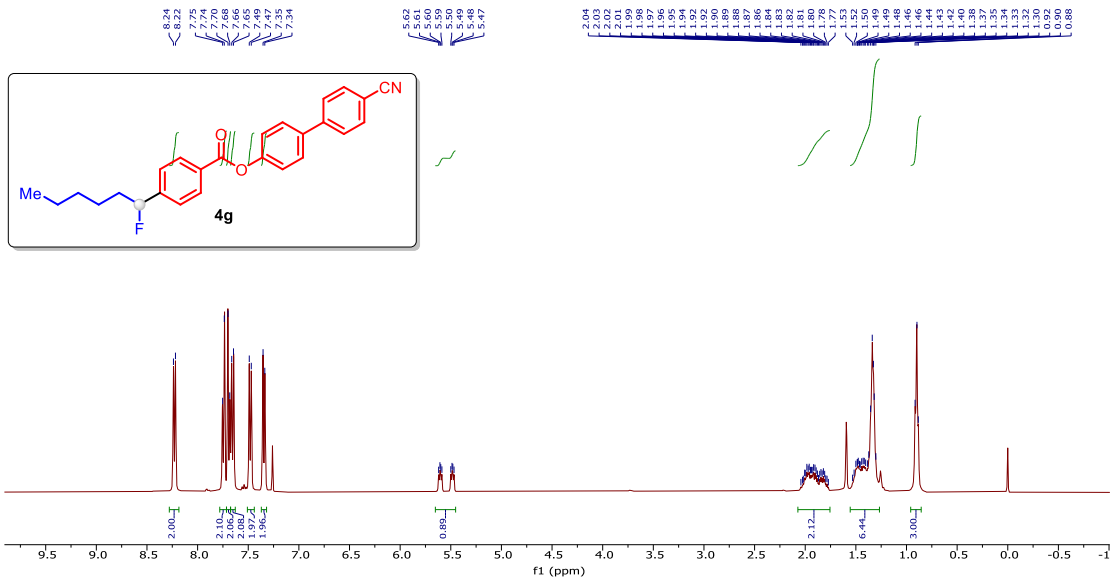
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **4f**



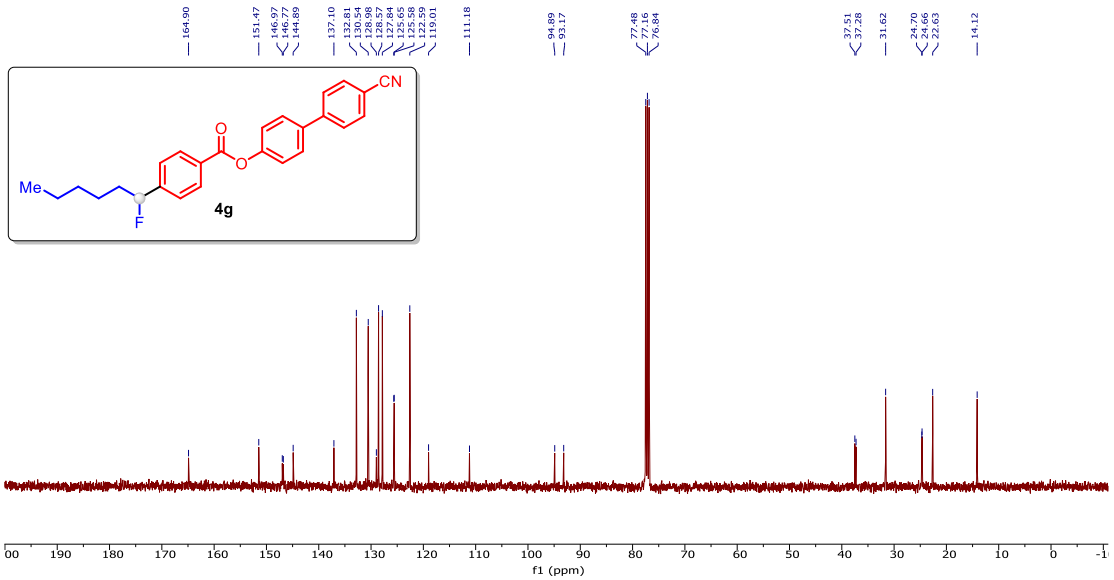
^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **4f**



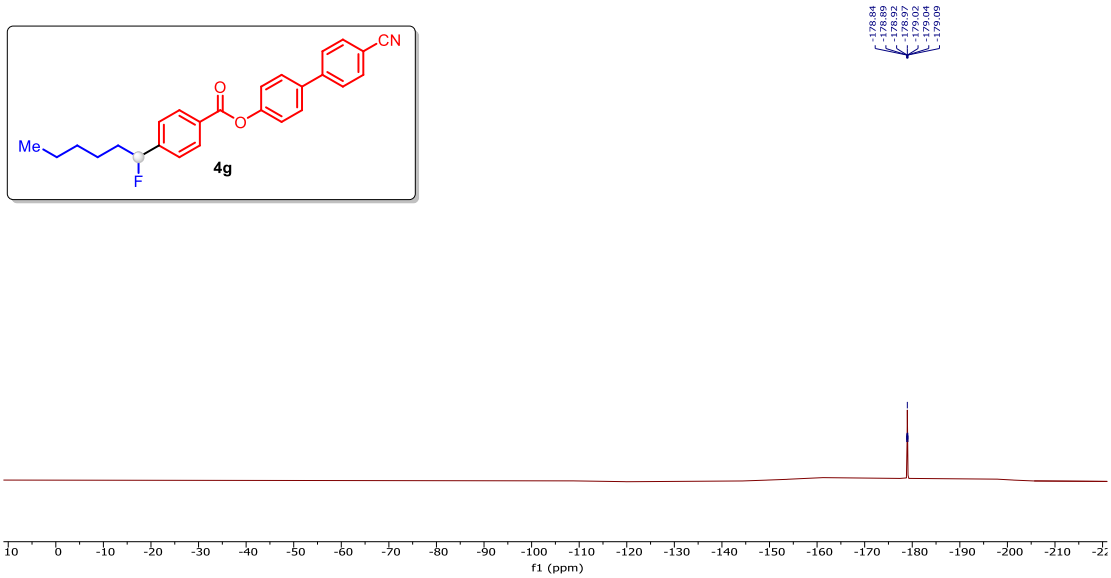
^{19}C NMR spectrum (376 MHz, $CDCl_3$) of **4f**



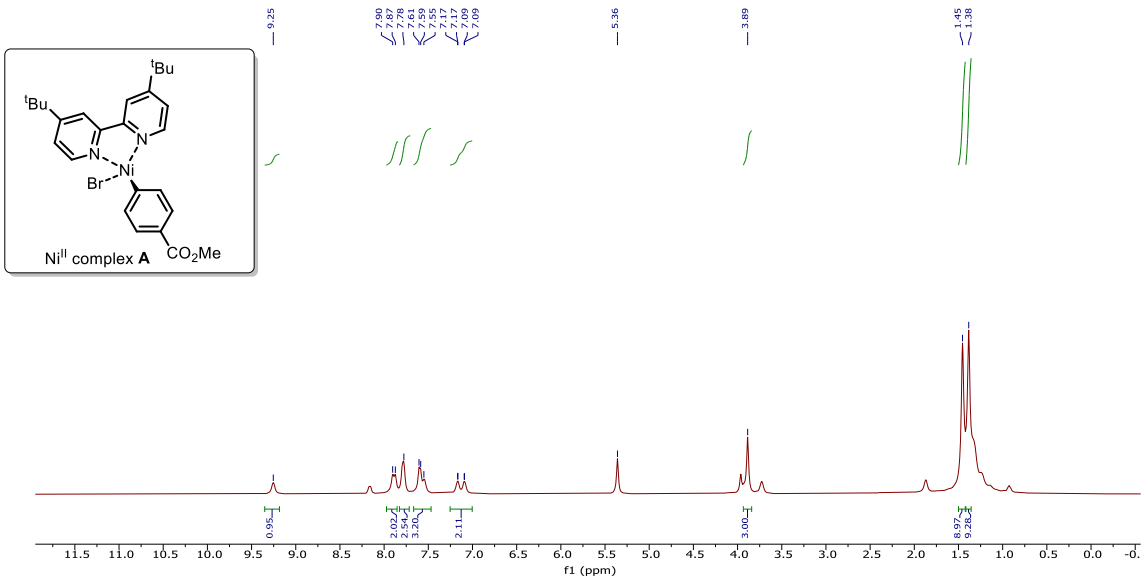
^{1}H NMR spectrum (400 MHz, $CDCl_3$) of **4g**



^{13}C NMR spectrum (101 MHz, $CDCl_3$) of **4g**



^{19}F NMR spectrum (376 MHz, $CDCl_3$) of **4g**



^{1}H NMR spectrum (400 MHz, CD_2Cl_2) of Ni^{II} complex **A**