Supplementary Information (SI) for Chemical Communications.
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Supporting Information

for

Halogen-Atom Transfer of Fluoroiodomethane for Photoredox-Catalyzed Fluoromethylation

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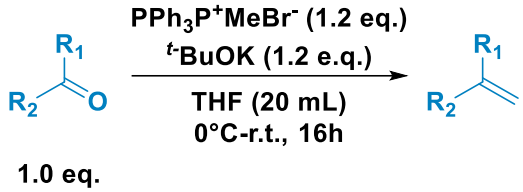
I. General Information

Anhydrous Dimethylacetamide (DMA), Acetonitrile (MeCN), Tetrahydrofuran (THF), *N*,*N*-dimethylformamide (DMF), Triethyl orthoformate (TEM), Methanol (MeOH) and 1,2-Dichloroethane (DCE) were purchased from J&K Scientific. All reagents were purchased at the highest commercial quality and used without further purification unless otherwise stated. Yields refer to chromatographically and spectroscopically (^{1}HNMR) homogeneous material, unless otherwise stated. Reactions were monitored by thin layer chromatography (TLC), GC/MS, GC/FID, or LC/MS. TLC was performed on pre-coated silica gel plated, using short-wave UV light as the visualizing agent, and phosphomolybdic acid, *p*-anisaldehyde, or $KMnO_4$ and heat as developing agents. Column chromatography was performed using silica gel 60 (300-400 mesh). ^{1}H NMR, ^{13}C NMR, and ^{19}F NMR were measured on a Bruker AVANCE III-400, 500, 600 spectrometer. Chemical shifts are reported in ppm (δ) relative to internal tetramethylsilane (TMS, δ 0.0 ppm) or with the solvent reference relative to TMS employed as the internal standard. Data are reported as follows: chemical shift (multiplicity [singlet (s), doublet (d), triplet (t), quartet (q), broad (br) and multiplet (m)], coupling constants [Hz], integration). Melting points are uncorrected. HRMS(ESI) was recorded on a Bruker miccOTOF-Q111, and HRMS(EI) was recorded on a Waters Micromass GCT Premier. The blue LEDs light was purchased from Kessil A360WE (90W MAX).

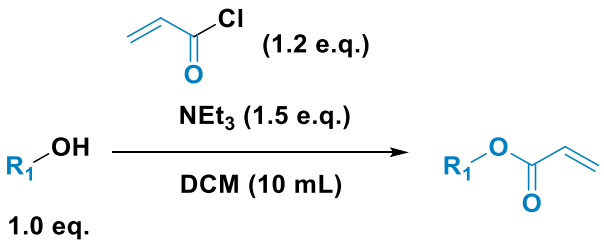
II. General procedures for hydrofluoromethylation reactions Substrates Preparation

Method A



Known olefins were synthesized according to literature procedures.[1] Methyltriphenylphosphonium bromide (6 mmol, 1.2 e.q.) was suspended in anhydrous tetrahydrofuran (THF) (15 mL) at 0 °C. Potassium tert-butoxide (673 mg, 6 mmol, 1.2 e.q.) was then added to the suspension, resulting in a bright yellow coloration. The reaction mixture was stirred at 0 °C for 30 minutes. Subsequently, the ketone substrate (5 mmol, 1.0 e.q.) was introduced, and the mixture was maintained at 0 °C for 16 hours to ensure the reaction reached completion. The reaction was quenched with water (10 mL) and extracted with ethyl acetate (3 × 6 mL). The combined organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure to afford the crude alkene product. The crude material was then purified by silica gel column chromatography to yield the desired alkene product.

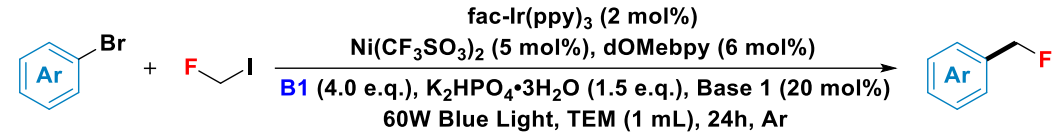
Method B



Known olefins were synthesized according to literature procedures.[2]The alcohol or phenol derivative (5.0 mmol) was dissolved in dry CH_2Cl_2 (10 mL) and combined with Et_3N (7.5 mmol). The reaction mixture was then cooled to 0°C using an ice-water bath. Acryloyl chloride (6.0 mmol) was added dropwise to the solution. The mixture was allowed to stir at room temperature overnight. Following the reaction, the solvent was evaporated under reduced pressure, and the residue was purified by silica gel column chromatography to yield the desired product.

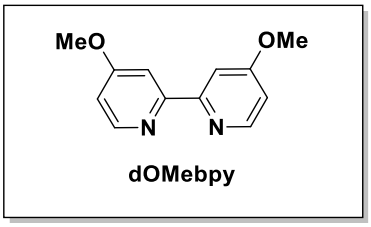
Experimental procedures

General procedure A

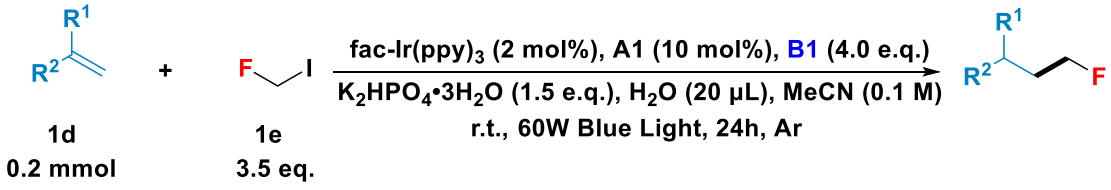


1c
0.2 mmol

1e
2.3 eq.

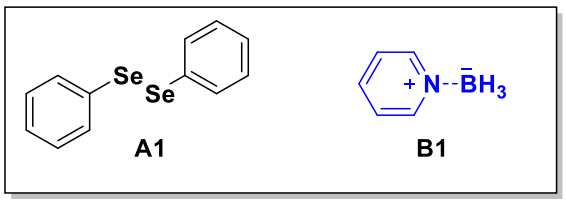


In the glove box, fac-Ir(ppy)$_3$ (2.6 mg, 2 mol%), Ni(CF$_3$SO$_3$)$_2$ (3.6 mg, 5 mol%), dOMebpy (2.6 mg, 6 mol%), and K$_2$HPO$_4$·3H$_2$O (68.5 mg, 1.5 equiv.) were added to a 10 mL vial equipped with a stir bar, along with aryl bromo-substituents (0.2 mmol, 1.0 equiv.; if the alkene is liquid, it is added after the solvent). TEM (1.0 mL) was then introduced to achieve a reaction concentration of 0.2 M with respect to the alkene. CH$_2$FI (30 μL, 2.3 equiv.), Py-BH$_3$ (80 μL, 4.0 equiv.), and 2-isopropylpyridine (20 mol%) were added at room temperature. The mixture was stirred and irradiated with a 60 W 456 nm LED lamp for 24 hours. The reaction mixture was subsequently concentrated under reduced pressure and purified by column chromatography on silica gel to yield the desired hydrofluoromethylated product.



1d
0.2 mmol

1e
3.5 eq.



General procedure B

In the glovebox, to an 10 mL vial equipped stir bar was added fac-Ir(ppy)$_3$ (2.6 mg, 2 mol%), PhSeSePh (6.2 mg, 10 mol%), K$_2$HPO$_4$-3H$_2$O (68.5 mg, 1.5 e.q.), alkenes (0.2

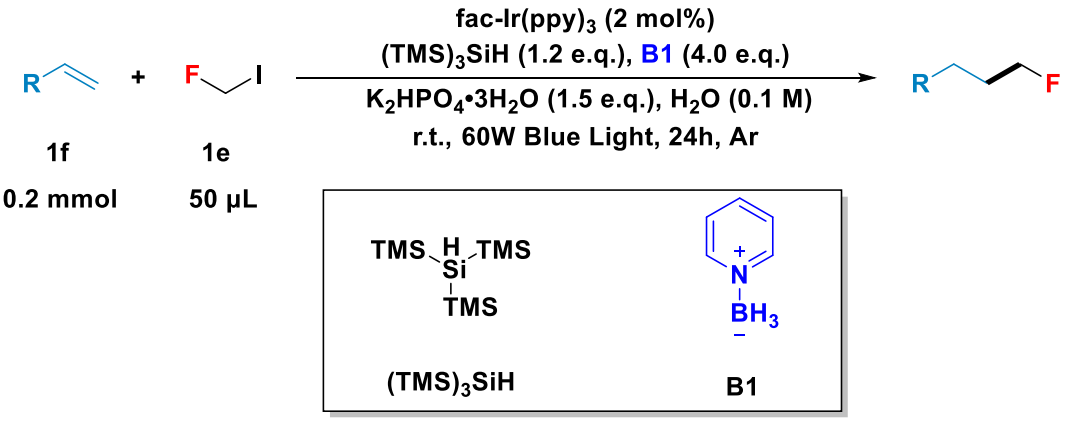
mmol, 1 e.q.; if alkenes were liquid, they were added after adding solvent), followed by MeCN (2.0 mL) for a reaction concentration of 0.1 M relative to alkenes. Then add CH_2FI (50 μL, 3.5 e.q.), Py-BH_3 (80 μL, 4.0 e.q.) and H_2O (20 μL) at room temperature. The resulting mixture was stirred and irradiated with 60 W 456 nm LED lamp for 24 hours. After that, the reaction mixture was concentrated under reduced pressure, purified by column chromatography over silica gel to obtain the desired hydrofluoromethylation products.

Reaction setup:



Fig. 1 0.2 mmol scale reactions **Fig. 2, 3** Reaction devices

General procedure C



In the glovebox, to an 10 mL vial equipped stir bar was added fac-Ir(ppy)$_3$ (2.6 mg, 2 mol%), K_2HPO_4-$3H_2O$ (68.5 mg, 1.5equiv.), alkenes (0.2 mmol, 1 equiv.; if alkenes were liquid, they were added after adding solvent), followed by H_2O (2.0 mL) for a reaction concentration of 0.1 M relative to alkenes. Then add CH_2FI (50 μL, 3.5 equiv.), Py-BH_3 (80 μL, 4.0 equiv.) and TMS_3SiH (80 μL, 1.5 equiv.) at room temperature.

The resulting mixture was stirred and irradiated with 60 W 456 nm LED lamp for 24 hours. After that, the reaction mixture was concentrated under reduced pressure, purified by column chromatography over silica gel to obtain the desired hydrofluoromethylation products.

General procedure D

In the glovebox, to an 10 mL vial equipped stir bar was added fac-Ir(ppy)$_3$ (130 mg, 2 mol%), PhSeSePh (310 mg, 10 mol%), K_2HPO_4-3H_2O (3.42 g, 1.5 e.q.), alkenes (10 mmol, 1 e.q.), followed by MeCN (100 mL) for a reaction concentration of 0.1 M relative to alkenes. Then add CH_2FI (2.5 mL, 3.5 e.q.), Py-BH_3 (4.0 mL, 4.0 e.q.) and H_2O (1.0 mL) at room temperature. The resulting mixture was stirred and irradiated with 60 W 456 nm LED lamp for 7 days. After that, the reaction mixture was concentrated under reduced pressure, purified by column chromatography over silica gel to obtain the desired hydrofluoromethylation products.

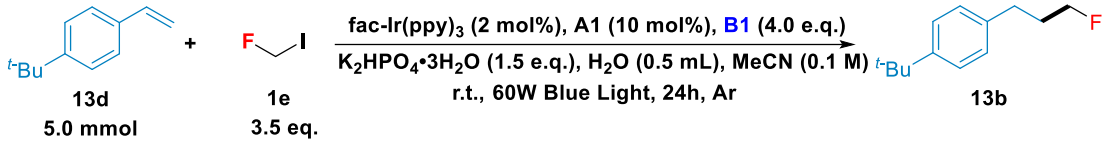
Fig. 4 Gram scale reactions

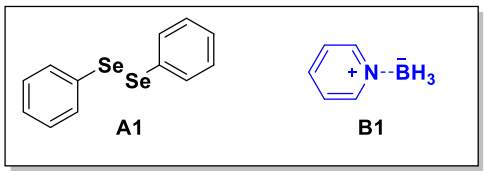
General procedure E

In the glovebox, to an 10 mL vial equipped stir bar was added fac-Ir(ppy)3 (2.6 mg, 2 mol%), PhSeSePh (6.2 mg, 10 mol%), K_2HPO_4-3H_2O (68.5 mg, 1.5 e.q.), alkenes (0.2 mmol, 1.0 e.q.), followed by MeCN (2.0 mL) for a reaction concentration of 0.1 M relative to alkenes. Then add ethyl fluoroiodoacetate (or ethyl iododifluoroacetate) (3.5 equiv.), Py-BH_3 (80μL, 4.0 e.q.) and H_2O (20 μL) at room temperature. The resulting mixture was stirred and irradiated with 60 W 456 nm LED lamp for 24 hours. After that, the reaction mixture was concentrated under reduced pressure, purified by column chromatography over silica gel to obtain the desired the products.

Procedure for flow reaction

General procedure F



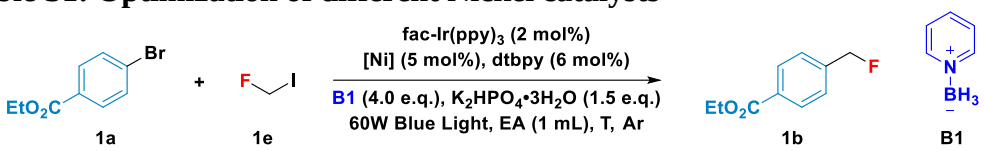


In the glovebox, to an 10 mL vial equipped stir bar was added fac-Ir(ppy)$_3$ (65 mg, 2 mol%), PhSeSePh (155 mg, 10 mol%), K_2HPO_4-3H_2O (1.71 g, 1.5 e.q.), alkenes (5 mmol, 1 e.q.), followed by MeCN (50 mL) for a reaction concentration of 0.1 M relative to alkenes. Then add CH_2FI (1.25 mL, 3.5 e.q.), Py-BH_3 (2.0 mL, 4.0 e.q.) and H_2O (0.5 mL) at room temperature. Thoroughly mix the solution. Subsequently, transfer it into a 60 mL disposable syringe, which is then attached to a syringe pump. The flow apparatus was initially purged with degassed acetonitrile to eliminate any residual air.The reaction mixture is pumped from the syringe through the pump into the flow reactor. High-purity Teflon (FEP) tubing (O.D. = 1.6 mm, I.D. = 1.0 mm, length = 14 m, volume = 11 mL) was coiled around a vessel with an outer diameter of 8.5 cm. The flow system was configured to achieve a residence time (T_R) of 18 h and a flow rate of 10 μL/min. The product mixture was collected after being stirred and irradiated with 60 W 456 nm LED lamp for 3.5 days. After that, the reaction mixture was concentrated under reduced pressure, purified by column chromatography over silica gel to obtain the desired hydrofluoromethylation product 13b (448 mg) in 46% yield.

III. Evaluation of reaction conditions

Optimization of conditions for cross coupling of fluoroiodomethane and aryl bromine substitutes

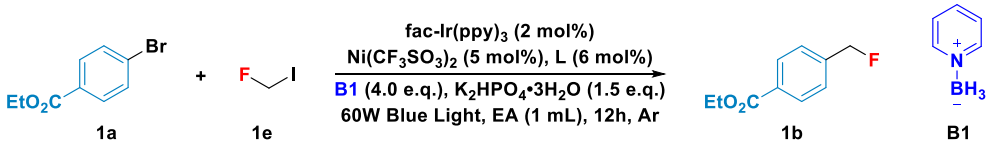
Table S1: Optimization of different Nickel catalysts [a]

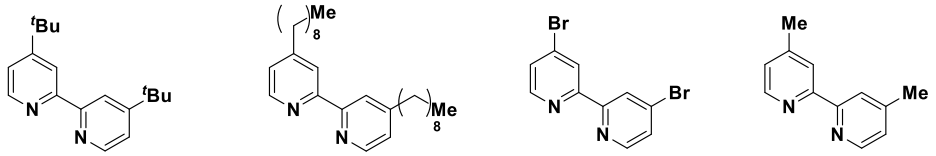


entry	[Ni]	T (h)	Yield (%)[b]
1	$NiBr_2$•Diglyme	24	4
2	$Ni(acac)_2$	24	4
3	NiI_2	24	8
4	NiF_2	24	2
5	$NiCl_2$•glyme	24	n.d.
6	$Ni(CF_3SO_3)_2$	24	11
7	$Ni(PPh_3)_2Cl_2$	24	n.d.
8	$Ni(CF_3SO_3)_2$	12	3

[a] All reactions were carried out with **1a** (0.2 mmol, 1.0 e.q.), **1e** (0.46 mmol, 2.3 e.q.), fac-Ir(ppy)$_3$ (0.004 mmol, 2 mol%), Nickel catalyst (5 mol%), dtbpy (6 mol%), B1 (4.0 e.q.) and Additive (1.5 e.q.) in EA (1.0 mL) at 25 °C under Ar at 60W Blue Light for T h. [b] GC yield. (undecane as internal standard).

Table S2: Optimization of different ligands [a]



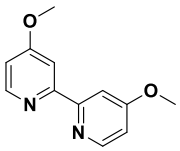


L1, 3 %

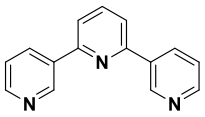
L2, 14 %

L3, 3 %

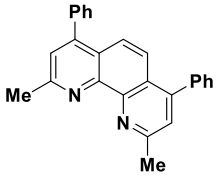
L4, 3 %



L5, 25 %



L6, n.d.



L7, n.d.

[a] All reactions were carried out with **1a** (0.2 mmol, 1.0 e.q.), **1e** (0.46 mmol, 2.3 e.q.), fac-Ir(ppy)$_3$ (0.004 mmol, 2 mol%), Ni(CF$_3$SO$_3$)$_2$ (5 mol%), ligands (6 mol%), B1 (4.0 e.q.) and Additive (1.5 e.q.) in EA (1.0 mL) at 25 °C under Ar at 60W Blue Light for 12 h. [b] GC yield. (undecane as internal standard).

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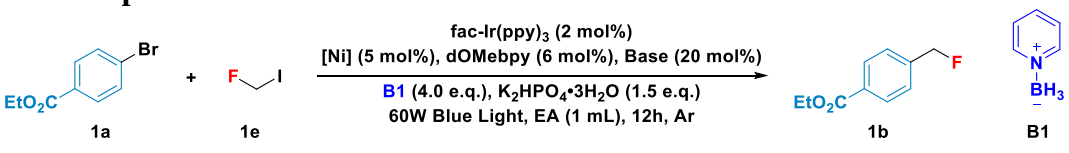
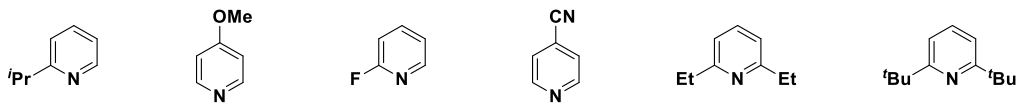
Table S3: Optimization of different Bases [a]





Base 1
32 %

Base 2
5 %

Base 3
18 %

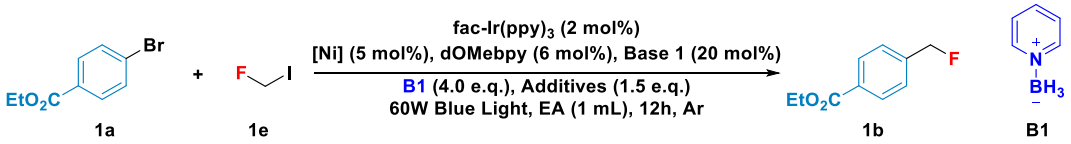
Base 4
19 %

Base 5
20 %

Base 6
15 %

[a] All reactions were carried out with **1a** (0.2 mmol, 1.0 e.q.), **1e** (0.46 mmol, 2.3 e.q.), fac-Ir(ppy)$_3$ (0.004 mmol, 2 mol%), Ni(CF$_3$SO$_3$)$_2$ (5 mol%), dOMebpy (6 mol%), B1 (4.0 e.q.), Base (20 mol%) and Additive (1.5 e.q.) in EA (1.0 mL) at 25 °C under Ar at 60W Blue Light for 12 h. [b] GC yield. (undecane as internal standard).

Table S4: Optimization of different Additives [a]

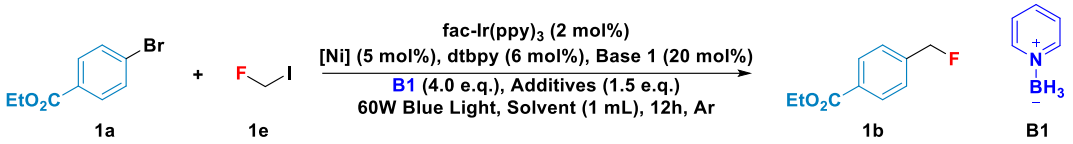


1a (0.2 mmol) + 1e (2.3 eq.) → 1b + B1

entry	Additives	Yield (%)[b]
1	K_2HPO_4	20
2	KH_2PO_4	n.d.
3	K_3PO_4	n.d.
4	$K_3PO_4•H_2O$	n.d.
5	Na_2HPO_4	n.d.
6	Na_3PO_4	n.d.
7	Li_3PO_4	n.d.

[a] All reactions were carried out with **1a** (0.2 mmol, 1.0 eq.), **1e** (0.46 mmol, 2.3 eq.), fac-Ir(ppy)$_3$ (0.004 mmol, 2 mol%), Ni(CF_3SO_3)$_2$ (5 mol%), dOMebpy (6 mol%), B1 (4.0 eq.), Base 1 (20 mol%) and Additive (1.5 eq.) in EA (1.0 mL) at 25 °C under Ar at 60W Blue Light for 12 h. [b] GC yield. (undecane as internal standard).

Table S5: Optimization of different Solvents [a]

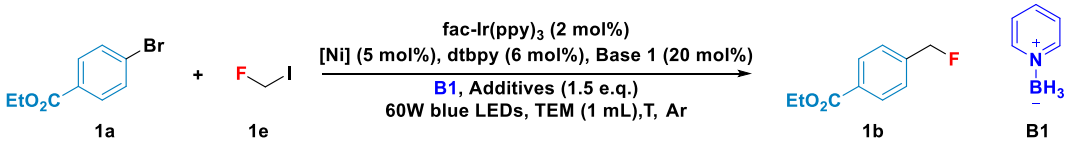


1a (0.2 mmol) 1e (2.3 eq.) 1b B1

entry	Solvent	Yield (%)[b]
1	DMC	3
2	TEM	68
3	MeCN	n.d.
4	DMA	2
5	DMF	n.d.
6	THF	22
7	1,4-Dioxane	24
8	DCM	4
9	Isopropyl acetate	21
10	DME	11
11	dDME	24
12	Me-THF	17
13	MeOH	3
14	p-Xylene	23
15	DCE	2

[a] All reactions were carried out with **1a** (0.2 mmol, 1.0 e.q.), **1e** (0.46 mmol, 2.3 e.q.), fac-Ir(ppy)$_3$ (0.004 mmol, 2 mol%), Ni(CF$_3$SO$_3$)$_2$ (5 mol%), dOMebpy (6 mol%), B1 (4.0 e.q.), Base 1 (20 mol%) and Additive (1.5 e.q.) in Solvent (1.0 mL) at 25 °C under Ar at 60W Blue Light for 12 h. [b] GC yield. (undecane as internal standard).

Table S6: Optimization of Chemical equivalent andTime [a]

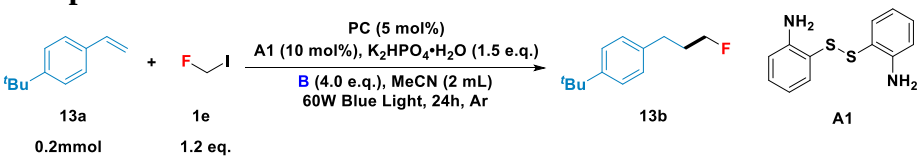


entry	1aa (e.q.)	B1 (e.q.)	T (h)	Yield (%)[b]
1	1.5	4.0	12	9
2	3.5	4.0	12	25
3	6.0	4.0	12	8
4	7.5	4.0	12	2
5	2.3	1.0	12	6
6	2.3	1.5	12	7
7	2.3	2.0	12	16
8	2.3	2.5	12	15
9	2.3	3.0	12	21
10	2.3	3.5	12	23
11	2.3	5.0	12	42
12	2.3	4.0	24	74
13	1.5	2.5	24	22
14	3.5	6.5	24	75
15	6.0	10.5	24	39
16	7.5	13.0	24	66
17	2.3	4.0	36	78
18	2.3	4.0	48	79
19	2.3	4.0	60	79

[a] All reactions were carried out with **1a** (0.2 mmol, 1.0 eq.), **1e**, fac-Ir(ppy)$_3$ (0.004 mmol, 2 mol%), Ni(CF$_3$SO$_3$)$_2$ (5 mol%), dOMebpy (6 mol%), B1, Base 1 (20 mol%) and Additive (1.5 e.q.) in TEM (1.0 mL) at 25 °C under Ar at 60W Blue Light for T. [b] GC yield. (undecane as internal standard).

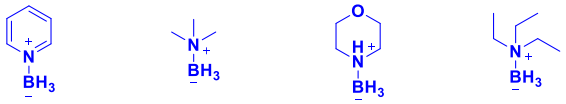
Optimization of conditions for monofluoromethyl radical addition to olefins

Table S7: Optimization of different PC and Borane [a]



13a (0.2mmol) + 1e (1.2 eq.) → 13b; A1

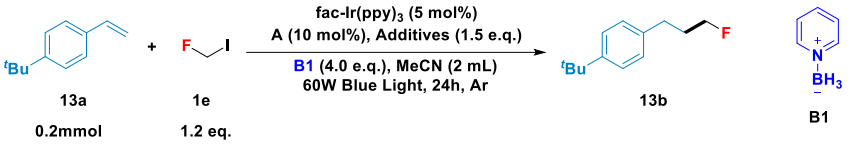
	PC	B	Yield (%)[b]
1	Ir[dF(CF3)ppy]2(dtbbpy)PF6	B1	18
2	Ir[dF(CF3)ppy]2(dCF3bpy)PF6	B1	18
3	fac-Ir(ppy)3	B1	32
4	4CzIPN	B1	18
5	4DPAIPN	B1	14
6	fac-Ir(ppy)3	B2	9
7	fac-Ir(ppy)3	B3	9
8	fac-Ir(ppy)3	B4	16



B1 B2 B3 B4

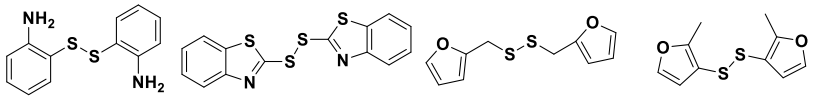
[a] All reactions were carried out with **13a** (0.2 mmol, 1.0 e.q.), **1e** (0.24 mmol, 1.2 e.q.), PC (0.01 mmol, 5 mol%), A1 (10 mol%), Additive (1.5 e.q.) and B (4.0 e.q.) in MeCN (2.0 mL) at 25 °C under Ar at 60W Blue Light for 24h.
[b] GC yield. (undecane as internal standard).

Table S8: Optimization of different A and Additives [a]

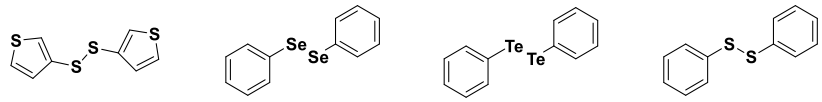


13a 0.2mmol | 1e 1.2 eq. | 13b | B1

	A	Additives	Yield (%)[b]
1	A1	$K_2HPO_4•3H_2O$	32
2	A2	$K_2HPO_4•3H_2O$	25
3	A3	$K_2HPO_4•3H_2O$	32
4	A4	$K_2HPO_4•3H_2O$	25
5	A5	$K_2HPO_4•3H_2O$	53
6	A6	$K_2HPO_4•3H_2O$	57
7	A7	$K_2HPO_4•3H_2O$	12
8	A8	$K_2HPO_4•3H_2O$	28
9	A6	K_2CO_3	14
10	A6	KH_2PO_4	18
11	A6	CF_3COOH	12
12	A6	/	22
13	A6	$NaHCO_3$	20
14	A6	Hydroquinone	16
15	A6	KHSO4	15
16	A6	HOBT	15
17	A6	NH_2OH-HCl	20
18	A6	NH_4Cl	8



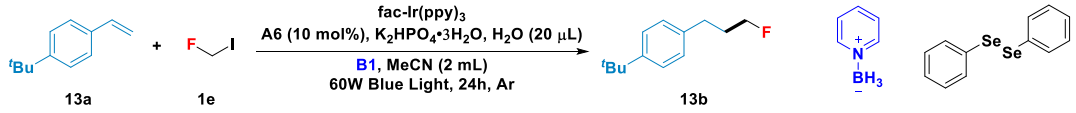
A1 A2 A3 A4



A5 A6 A7 A8

[a] All reactions were carried out with **13a** (0.2 mmol, 1.0 e.q.), **1e** (0.24 mmol, 1.2 e.q.), PC (0.01 mmol, 5 mol%), A (10 mol%), Additive (1.5 e.q.) and B1 (4.0 e.q.) in MeCN (2.0 mL) at 25 °C under Ar at 60W Blue Light for 24h.
[b] GC yield. (undecane as internal standard).

Table S9: Optimization of Chemical equivalent [a]



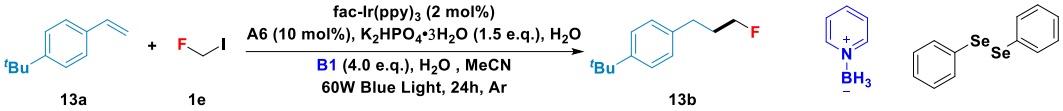
13a 1e 13b B1 A6

0.2mmol

	PC (mol%)	$K_2HPO_4•H_2O$ (e.q.)	B1 (e.q.)	1e (e.q.)	Yield (%)[b]
1	2	1.5	4.0	2.0	55
2	2	0.5	4.0	2.0	23
3	2	1.0	4.0	2.0	30
4	2	2.0	4.0	2.0	51
5	2	1.5	1.5	2.0	31
6	2	1.5	2.5	2.0	41
7	2	1.5	5	2.0	55
8	2	1.5	4.0	3.5	61
9	2	1.5	4.0	8.0	62

[a] All reactions were carried out with **13a** (0.2 mmol, 1.0 e.q.), **1e**, PC, A (10 mol%), Additive (1.5 e.q.) and B1 in MeCN (2.0 mL) at 25 °C under Ar at 60W Blue Light for 24h. [b] GC yield. (undecane as internal standard).

Table S10: Optimization of Chemical equivalent [a]



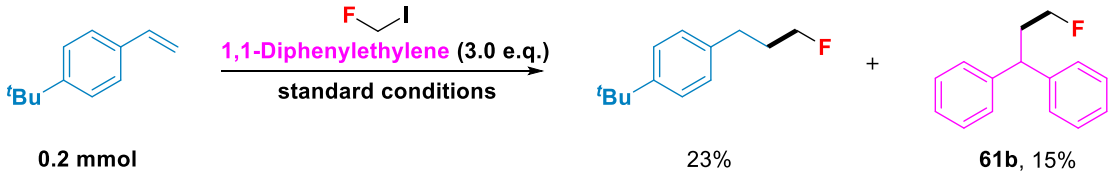
13a 0.2mmol | 1e 3.5 eq. | 13b | B1 | A6

	H_2O (μL)	Solvent (mL)	Yield (%)[b]
1	5	2.0	61
2	10	2.0	56
3	15	2.0	51
4	20	2.0	84
5	25	2.0	46
6	30	2.0	48
7	35	2.0	44
8	40	2.0	48
9	45	2.0	48
10	50	2.0	51
11	20	0.5	35
12	20	1.0	38
13	20	1.5	65
14	20	2.5	68

[a] All reactions were carried out with **13a** (0.2 mmol, 1.0 e.q.), **1e** (0.70 mmol,3.5 e.q.), PC (0.01 mmol, 5 mol%), A6 (10 mol%), Additive (1.5 e.q.), H_2O and B1 (4.0 e.q.) in MeCN at 25 °C under Ar at 60W Blue Light for T. [b] GC yield. (undecane as internal standard).

Ⅳ. Mechanistic investigation

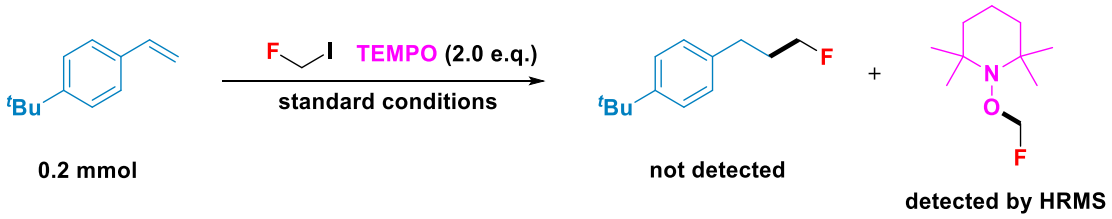
a) Radical trapping experiments



0.2 mmol　　23%　　**61b**, 15%

When adding 3.0 equivalent 1,1-Diphenylethylene into the reaction mixture, the desired addition product yield is only 23%, whereas the CFH_2-1,1-Diphenylethylene adduct was detected by GC-MS and the 19F NMR spectrum with 15% yield, indicating the formation of CFH_2 radical during the reaction process.

b) Radical suppression test



0.2 mmol　　not detected　　detected by HRMS

When adding 2.0 equivalent TEMP into the reaction mixture, the desired addition product was not detected, whereas the CFH_2-TEMP adduct was detected by HRMS, indicating the formation of CFH_2 radical during the reaction process.

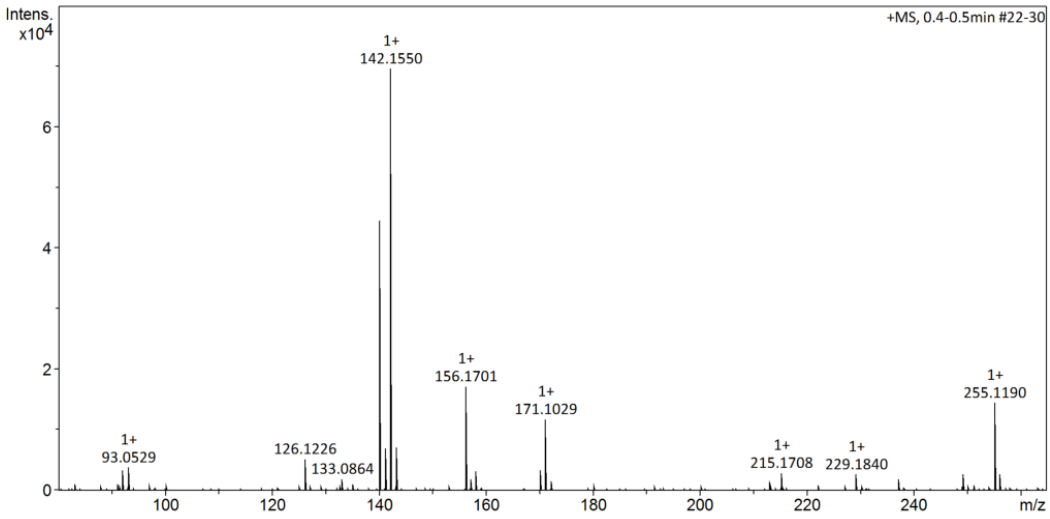
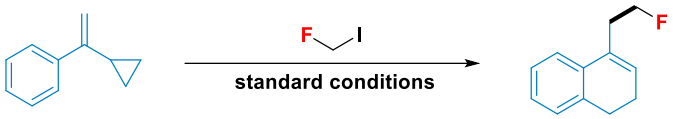


Figure 5. GC-MS spectrum of CFH_2-TEMPO.

c) Radical clock reaction

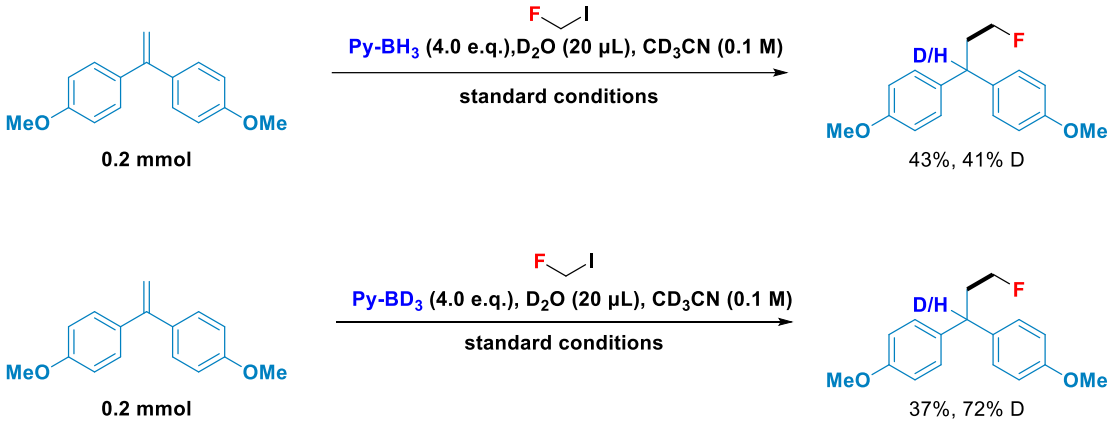


62b, 34%

Free radical clock experiments using cyclopropylstyrene facilitated the isolation of the ring-opening product in 34% yield under standard conditions.

Following the general procedure B, the ring-opening product 62b (12.0 mg, colorless oil) was obtained in 34% yield. **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.15 (d, J = 8.7 Hz, 4H), 6.82 (d, J = 8.7 Hz, 4H), 4.20 (t, J = 7.4 Hz, 1H), 3.88 (q, J = 7.2 Hz, 2H), 3.76 (s, 6H), 2.88 (td, J = 15.4, 7.4 Hz, 2H), 1.19 (t, J = 7.2 Hz, 3H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 163.9 (t, J = 32.5 Hz), 158.4, 135.5, 128.7, 114.0, 62.8, 55.3, 43.3 (t, J = 4.9 Hz), 40.7 (t, J = 23.1 Hz), 29.8, 13.8. **^{19}F NMR (376 MHz, Chloroform-d):** δ -103.4. **HRMS (ESI)** m/z calcd for $C_{12}H_{13}F^{+}$ $(M+Na)^{+}$ 199.0899, found m/z 199.0898.

d) Deuterium labelling study



Several parallel experiments utilizing Py-BH_3, Py-BD_3, heavy water, and deuterated acetonitrile were performed. Notably, a combination of deuterated aminoborane, heavy water, and deuterated acetonitrile resulted in a higher yield of 37% and an increased deuterium content (72% D). The measured kinetic isotope effect (KIE) value of 2.0 suggests that the generation of boron radicals is likely involved in the rate-determining step. Interestingly, even in the absence of deuterated aminoborane, the presence of heavy water and deuterated acetonitrile still resulted in 43% deuterium incorporation in the final product, indicating that proton exchange may occur during the reaction.

Following the general procedure B, the reaction was performed using D_2O and CD_3CN

instead or using Py-BD_3, D_2O and CD_3CN.

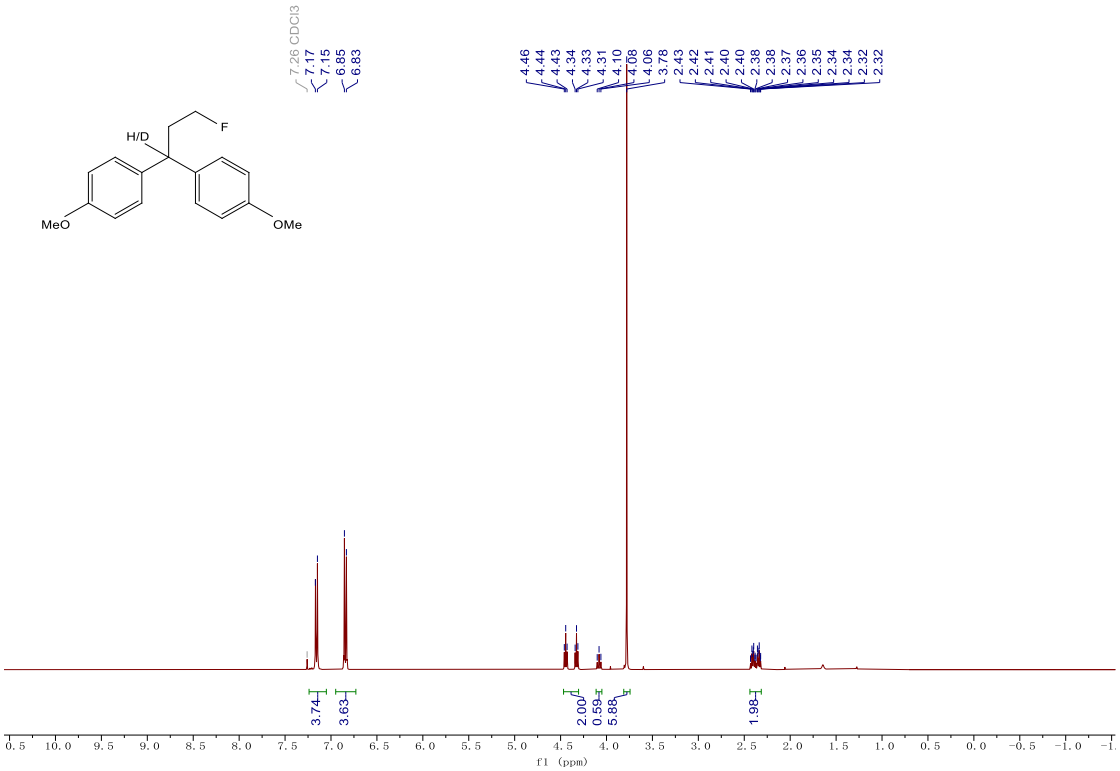


Figure 6. ^1H NMR spectrum of the reaction yield using D_2O and CD_3CN

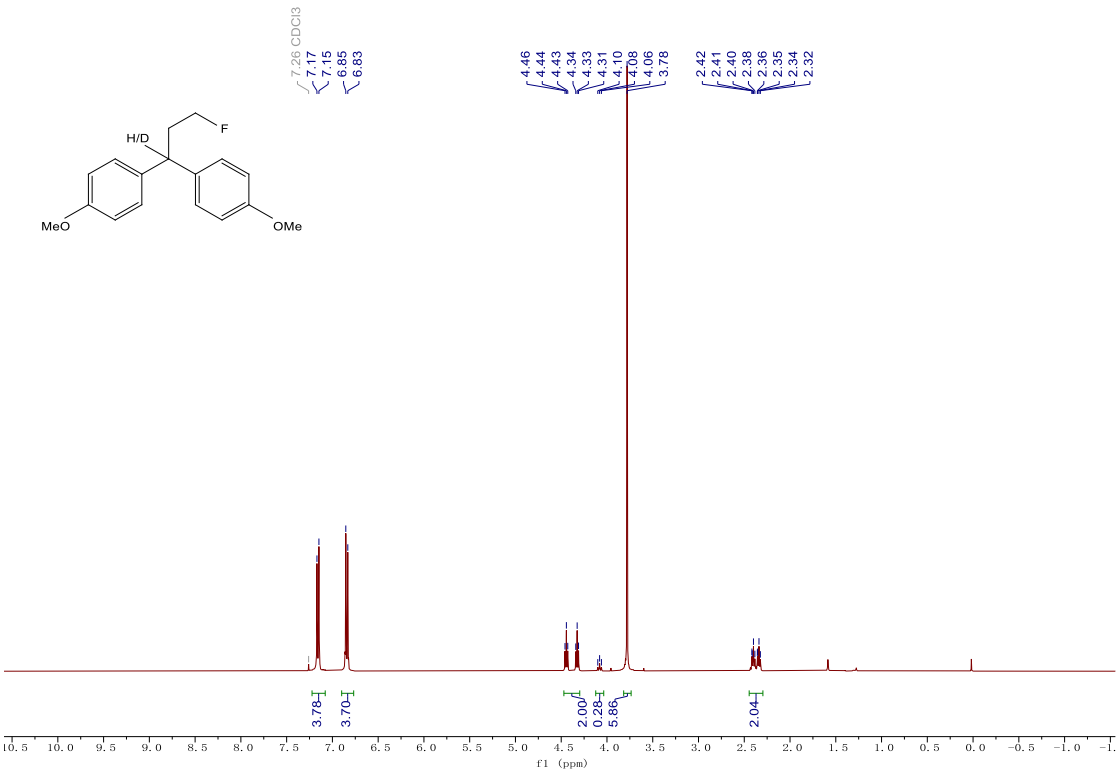


Figure 7. ^1H NMR spectrum of the reaction yield using Py-BD_3, D_2O and CD_3CN

e) Stern-Volmer fluorescence quenching studies

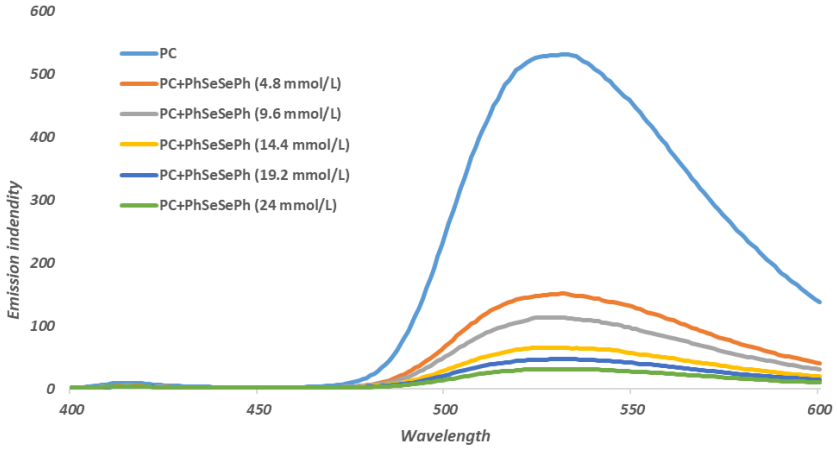


Figure 8. Fluorescence quenching study of fac-Ir(ppy)$_3$ with different equivalents of PhSeSePh

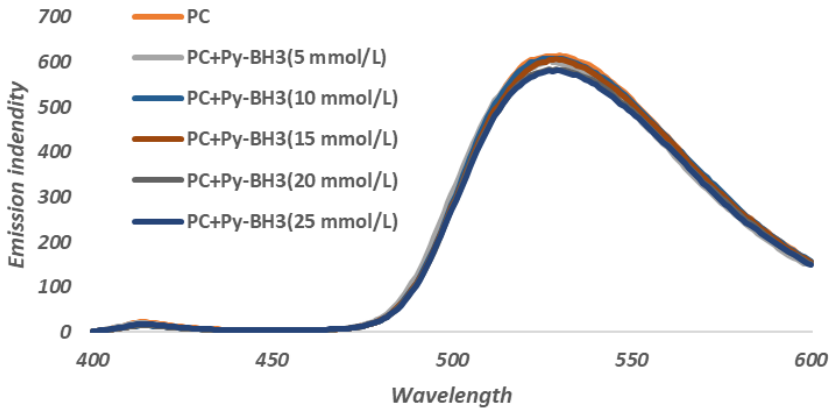


Figure 9. Fluorescence quenching study of fac-Ir(ppy)$_3$ with different equivalents of Py-BH$_3$

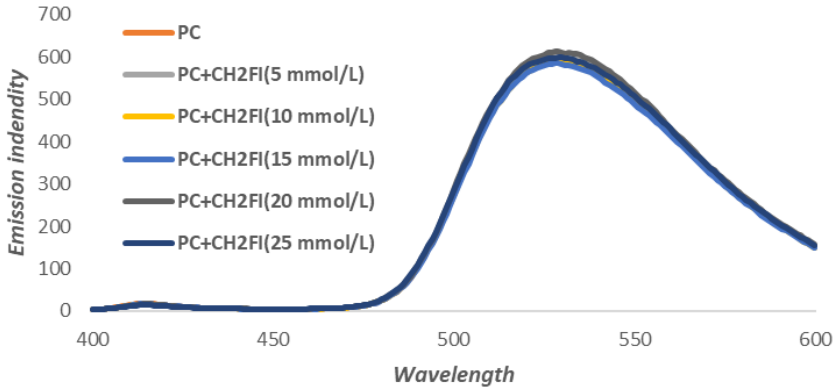


Figure 10. Fluorescence quenching study of fac-Ir(ppy)$_3$ with different equivalents of CH_2FI

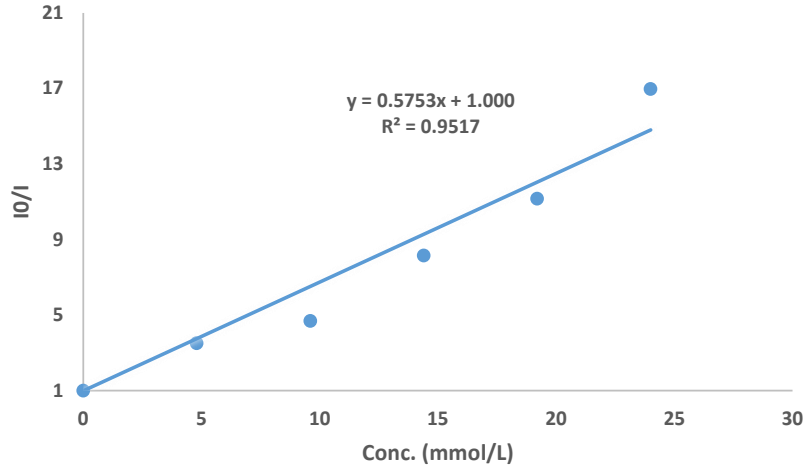
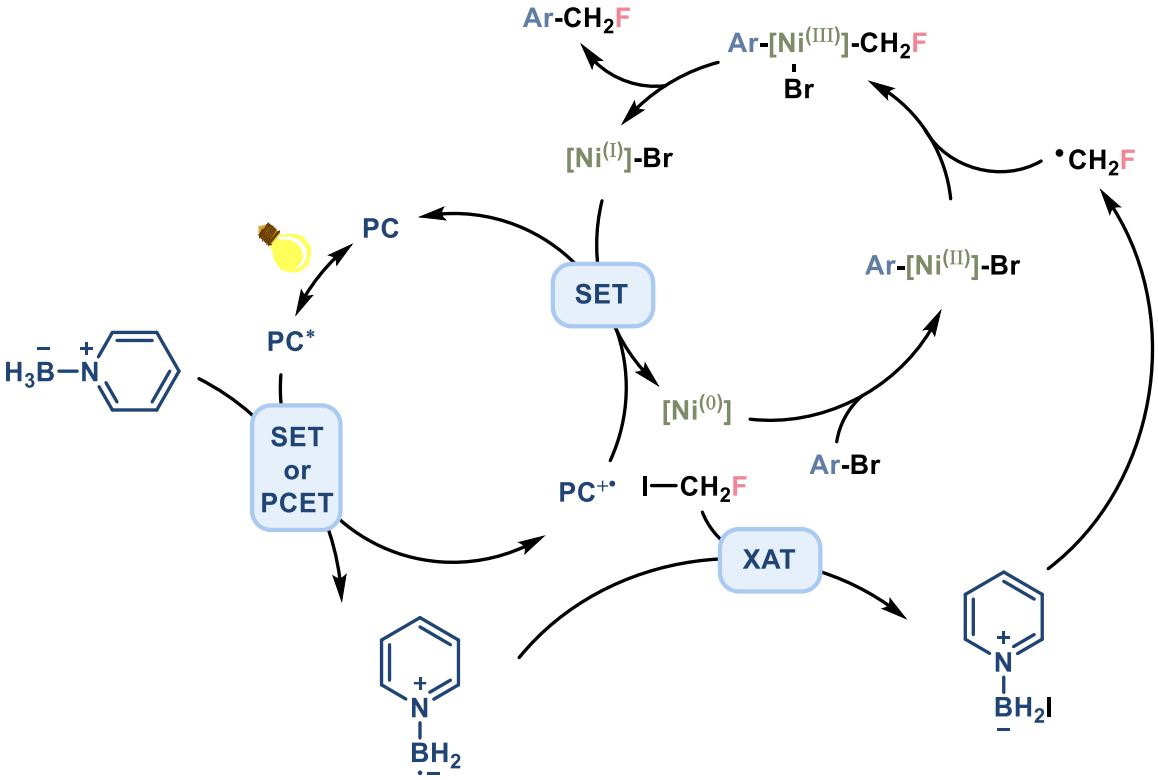


Figure 11. Combined quenching data

Stern-Volmer quenching experiments involving PhSeSePh demonstrated that this compound efficiently quenches the excited photocatalyst, indicating its participation in the oxidation quenching catalytic cycle.

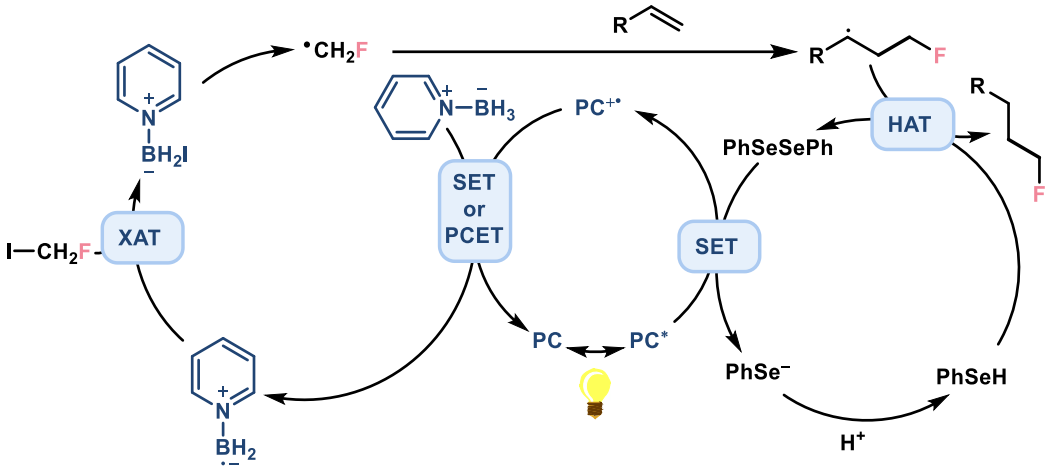
f) Possible mechanism

Proposed mechanisms for aryl bromo substituents



Mechanistically, the process can be described by the standard [Ni (0) - (II) - (III) - (I)] catalytic cycle. In this cycle, SET between the [Ni (I)] species and the oxidized photocatalyst, fac-Ir(ppy)$_3^+$, generates the [Ni (II)] intermediate and restores the ground-state fac-Ir(ppy)$_3$. Although both the [Ni (I)] and fac-Ir(ppy)$_3$ intermediates are highly electrophilic, they are expected to follow distinct mechanistic pathways. Specifically, oxidative addition of [Ni (0)] to aryl bromides forms the Ar-Ni (II) intermediate. Concurrently, upon photoexcitation, fac-Ir(ppy)$_3$ undergoes SET with Py-BH3,[3] The resulting boron-centered radical engages in an XAT reaction with fluoroiodomethane, producing a key CH_2F radical. Given that these species capture [Ni (II)] intermediates at diffusion-controlled rates, the subsequent step involves the formation of the alkyl-Ni (III)-Ar species. This intermediate undergoes elimination via reduction, leading to the final cross-coupling product, while regenerating the catalytically active [Ni (I)] species and completing the catalytic cycle. It is crucial that Py-BH_3 does not react with the [Ni (I)/(II)] catalyst, as such an interaction could lead to the formation of undesirable [Ni (I)/(II)]-H species, which would disrupt the catalytic process.

Proposed mechanisms for olefins



Based on extensive experimental data and a thorough review of previous literature,[4] we propose a preliminary mechanistic pathway. Upon blue light irradiation, diselenides undergo homolytic cleavage, leading to the generation of aryl selenium radicals. These radicals are subsequently reduced by the excited state of fac-Ir(ppy)$_3$, resulting in the formation of selenium anions and the oxidized form of the photocatalyst (PC^+). The oxidized photocatalyst [fac-Ir(ppy)$_3^+$], facilitates the oxidation of Py-BH_3 via two possible pathways: either through deprotonation following SET or via a PCET process, generating aminoboron radicals. The nucleophilic boron radical then undergoes XAT with CFH_2I, yielding a transient CH_2F radical. This CH_2F radical proceeds to undergo an intermolecular radical addition with an olefinic substrate, followed by a subsequent HAT from the radical adduct to selenophenol. This sequence of reactions culminates in the formation of the radical addition product and the regeneration of the selenophenol substrate, thereby completing the catalytic cycle.

Ⅴ. References

1 N. Sellet, M. Sebbat, M. Elhabiri, M. Cormier and J.-P. Goddard, *Chem. Commun*. 2022, **58**, 13759-13762.

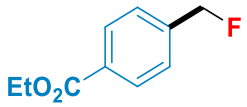
2 J. Zhang, S. Zhang and H. Zou, *Org. Lett*. 2021, **23**, 3466-3471.

3 Z. Zhang, M. J. Tilby and D. Leonori, *Nat. Synth.* 2024, **3**, 1221-1230.

4 Z.-Q. Zhang, Y.-Q. Sang, C.-Q. Wang, P. Dai, X.-S. Xue, J. L. Piper, Z.-H. Peng, J.-A. Ma, F.-G. Zhang and J. Wu, *J. Am. Chem. Soc.*, 2022, **144**, 14288-14296.

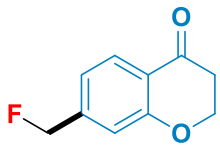
Ⅵ. Characterization Data

ethyl 4-(fluoromethyl)benzoate (1b)



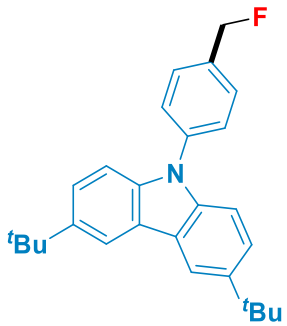
Following the general procedure condition **A**. Colorless oil. (25.5 mg, 70%)。R_f= 0.31 (Eluent: PE/EA 20:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.07 (d, *J* = 7.3 Hz, 2H), 7.42 (d, *J* = 7.8 Hz, 2H), 5.44 (d, *J* = 47.2 Hz, 2H), 4.39 (q, *J* = 7.1 Hz, 2H), 1.40 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 166.3, 141.2 (d, *J* = 17.2 Hz), 130.8 (d, *J* = 2.3 Hz), 130.0, 126.7 (d, *J* = 6.4 Hz), 83.9 (d, *J* = 168.4 Hz), 61.2, 14.4. **^{19}F NMR (376 MHz, Chloroform-d):** δ -212.7. **HRMS (ESI)** m/z calcd for $C_{10}H_{11}FO_2^+$ $(M+Na)^+$ 205.0641, found m/z 205.0641.

7-(fluoromethyl)chroman-4-one (2b)



Following the general procedure condition **A**. Colorless oil. (12.6 mg, 35%)。R_f= 0.15 (Eluent: PE/EA 20:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.91 (dd, *J* = 8.5, 1.2 Hz, 1H), 6.98 (t, *J* = 3.9 Hz, 2H), 5.38 (d, *J* = 47.0 Hz, 2H), 4.57 – 4.53 (m, 2H), 2.86 – 2.78 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 191.5, 162.1, 144.9 (d, *J* = 17.3 Hz), 127.8, 121.2, 119.2 (d, *J* = 6.5 Hz), 115.7 (d, *J* = 7.8 Hz), 83.4 (d, *J* = 170.0 Hz), 67.3, 37.8. **^{19}F NMR (376 MHz, Chloroform-d):** δ -215.5. **HRMS (ESI)** m/z calcd for $C_{10}H_9FO_2^+$ $(M+Na)^+$ 203.0485, found m/z 203.0485.

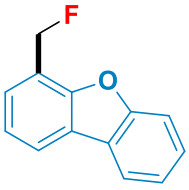
3,6-di-tert-butyl-9-(4-(fluoromethyl)phenyl)-9H-carbazole (3b)



Following the general procedure condition **A**. Colorless oil. (18.6 mg, 24%)。R_f= 0.68

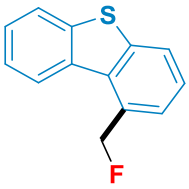
(Eluent: PE/EA 20:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.16 (d, *J* = 1.9 Hz, 2H), 7.61 (s, 4H), 7.48 (dd, *J* = 8.7, 2.0 Hz, 2H), 7.36 (d, *J* = 8.6 Hz, 2H), 5.50 (d, *J* = 47.8 Hz, 2H), 1.48 (s, 18H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 143.2, 139.2, 138.8 (d, *J* = 3.1 Hz), 129.9, 129.2 (d, *J* = 5.8 Hz), 126.9, 123.8, 123.6, 116.4, 109.3, 84.3 (d, *J* = 166.7 Hz), 34.9, 32.2. **^{19}F NMR (376 MHz, Chloroform-d):** δ -206.4. **HRMS (ESI)** m/z calcd for $C_{27}H_{30}FN^+$ $(M+Na)^+$ 410.2260, found m/z 410.2261.

4-(fluoromethyl)dibenzo[b,d]furan (4b)



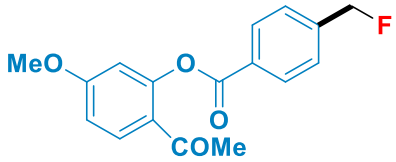
Following the general procedure condition **A**. Colorless oil. (17.2 mg, 43%)。 R_f= 0.50 (Eluent: PE/EA 50:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.97 (d, *J* = 7.6 Hz, 2H), 7.62 (d, *J* = 8.2 Hz, 1H), 7.56 – 7.45 (m, 2H), 7.37 (tdd, *J* = 7.5, 3.3, 1.0 Hz, 2H), 5.80 (d, *J* = 47.8 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 156.3, 127.6, 127.1 (d, *J* = 6.0 Hz), 124.7, 124.1, 123.1, 122.9 (d, *J* = 1.9 Hz), 121.6 (d, *J* = 3.3 Hz), 120.9, 120.4, 120.2, 112.0, 79.8 (d, *J* = 166.0 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -210.8. **HRMS (ESI)** m/z calcd for $C_{13}H_9FO^+$ $(M+Na)^+$ 223.0535, found m/z 223.0536.

1-(fluoromethyl)dibenzo[b,d]thiophene (5b)



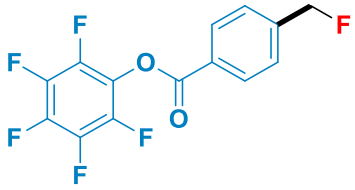
Following the general procedure condition **A**. Colorless oil. (35.1 mg, 81%)。 R_f= 0.60 (Eluent: PE/EA 50:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.34 – 8.20 (m, 1H), 7.99 – 7.82 (m, 2H), 7.60 – 7.40 (m, 4H), 5.96 (d, *J* = 47.8 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 140.5 (d, *J* = 1.8 Hz), 139.9, 134.9, 133.7 (d, *J* = 3.0 Hz), 131.9 (d, *J* = 16.4 Hz), 126.7, 126.2, 125.8 (d, *J* = 9.5 Hz), 125.1 (d, *J* = 5.6 Hz), 124.9, 123.9 (d, *J* = 3.2 Hz), 122.9, 83.7 (d, *J* = 168.2 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -206.0. **HRMS (ESI)** m/z calcd for $C_{13}H_9FS^+$ $(M+Na)^+$ 239.0307, found m/z 239.0306.

2-acetyl-5-methoxyphenyl 4-(fluoromethyl)benzoate (6b)



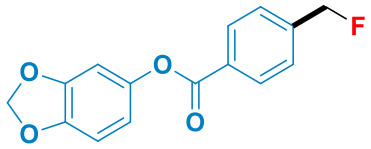
Following the general procedure condition **A**. Colorless oil. (16.9 mg, 28%)。R_f= 0.25 (Eluent: PE/EA 5:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.24 (d, *J* = 7.8 Hz, 2H), 7.89 (d, *J* = 8.8 Hz, 1H), 7.51 (d, *J* = 8.0 Hz, 2H), 6.88 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.73 (d, *J* = 2.5 Hz, 1H), 5.49 (d, *J* = 47.1 Hz, 2H), 3.87 (s, 3H), 2.49 (s, 3H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 195.7, 164.9, 163.9, 151.7, 142.2 (d, *J* = 17.4 Hz), 132.6, 130.8, 129.6 (d, *J* = 2.2 Hz), 126.9 (d, *J* = 6.8 Hz), 123.5, 112.1, 109.4, 83.7 (d, *J* = 169.1 Hz), 55.9, 29.5. **^{19}F NMR (376 MHz, Chloroform-d):** δ -213.8. **HRMS (ESI)** m/z calcd for $C_{17}H_{15}FO_4^+$ $(M+Na)^+$ 325.0582, found m/z 325.0582.

perfluorophenyl 4-(fluoromethyl)benzoate (7b)



Following the general procedure condition **A**. Colorless oil. (26.5 mg, 41%)。R_f= 0.20 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.23 (d, *J* = 7.4 Hz, 2H), 7.54 (d, *J* = 7.6 Hz, 2H), 5.51 (d, *J* = 46.9 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 162.2, 143.4 (d, *J* = 17.3 Hz), 142.7, 139.2, 136.7, 131.0, 130.7, 126.9 (d, *J* = 2.2 Hz), 126.7 (d, *J* = 6.9 Hz), 83.3 (d, *J* = 170.0 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -152.4 – -152.6 (m), -157.7 – -157.9 (m), -162.3 (tt, *J* = 22.3, 5.5 Hz), -215.2. **HRMS (ESI)** m/z calcd for $C_{14}H_6F_6O_2^+$ $(M+Na)^+$ 343.0170, found m/z 343.0172.

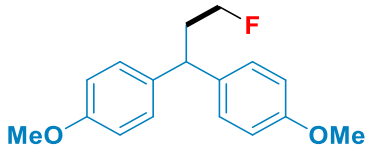
benzo[d][1,3]dioxol-5-yl 4-(fluoromethyl)benzoate (8b)



Following the general procedure condition **A**. Colorless oil. (33.9 mg, 62%)。R_f= 0.35 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.20 (d, *J* = 7.2 Hz, 2H), 7.50 (d, *J* = 7.7 Hz, 2H), 6.83 (d, *J* = 8.4 Hz, 1H), 6.74 (d, *J* = 2.3 Hz, 1H), 6.66 (dd, *J*

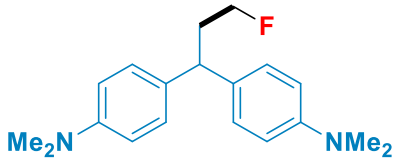
= 8.3, 2.3 Hz, 1H), 6.01 (s, 2H), 5.49 (d, *J* = 47.1 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 165.2, 148.2, 145.5 (d, *J* = 34.1 Hz), 142.1 (d, *J* = 17.3 Hz), 130.6, 129.7 (d, *J* = 2.3 Hz), 128.7, 126.8 (d, *J* = 6.6 Hz), 114.2, 108.2, 104.0, 101.9, 83.7 (d, *J* = 169.0 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -213.6. **HRMS (ESI)** m/z calcd for $C_{15}H_{11}FO_4^+$ $(M+Na)^+$ 297.0539, found m/z 297.0540.

4,4'-(3-fluoropropane-1,1-diyl)bis(methoxybenzene) (9b)



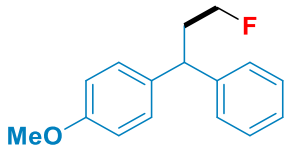
Following the general procedure condition **B**. Colorless oil. (46.2 mg, 84%)。 ***R*$_f$** = 0.40 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-*d*):** δ 7.17 (d, *J* = 8.6 Hz, 4H), 6.86 (d, *J* = 8.7 Hz, 4H), 4.40 (dt, *J* = 47.1, 6.0 Hz, 2H), 4.10 (t, *J* = 8.0 Hz, 1H), 3.79 (s, 6H), 2.39 (ddt, *J* = 24.1, 8.0, 6.0 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-*d*):**δ 158.2, 136.5, 128.8, 114.0, 82.3 (d, J = 164.4 Hz), 55.3, 44.8 (d, J = 5.2 Hz), 36.5 (d, J = 19.6 Hz). **^{19}F NMR (376 MHz, Chloroform-*d*):** δ -220.7. **HRMS (ESI)** m/z calcd for $C_{17}H_{19}FO_2^+$ $(M+Na)^+$ 297.1267, found m/z 297.1269.

4,4'-(3-fluoropropane-1,1-diyl)bis(N,N-dimethylaniline) (10b)



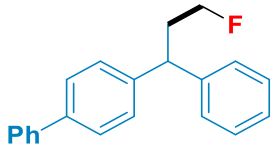
Following the general procedure condition **B**. Colorless oil. (3.6 mg, 6%)。 ***R*$_f$** = 0.30 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.11 (d, *J* = 8.7 Hz, 4H), 6.72 (d, *J* = 8.3 Hz, 4H), 4.38 (dt, *J* = 47.1, 6.1 Hz, 2H), 3.98 (t, *J* = 8.0 Hz, 1H), 2.91 (s, 12H), 2.41 – 2.29 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 128.6, 113.4, 82.7 (d, *J* = 163.8 Hz), 41.2, 36.5 (d, *J* = 19.7 Hz), 29.8. **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.4. **HRMS (ESI)** m/z calcd for $C_{19}H_{25}FN_2^+$ $(M+H)^+$ 301.2080, found m/z 301.2081.

1-(3-fluoro-1-phenylpropyl)-4-methoxybenzene (11b)



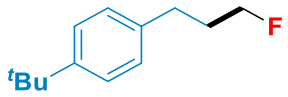
Following the general procedure condition **B**. Colorless oil. (28.0 mg, 57%)。R_f= 0.50 (Eluent: PE/EA 50:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.28 – 7.09 (m, 7H), 6.79 (d, *J* = 8.7 Hz, 2H), 4.33 (dt, *J* = 47.1, 6.0 Hz, 2H), 4.07 (t, *J* = 8.0 Hz, 1H), 3.72 (s, 3H), 2.41 – 2.30 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 158.2, 144.4, 136.1, 128.9, 128.7, 127.9, 126.5, 114.1, 82.2 (d, *J* = 164.7 Hz), 55.3, 45.7 (d, *J* = 5.4 Hz), 36.3 (d, *J* = 19.6 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.8. **HRMS (ESI)** m/z calcd for $C_{16}H_{17}FO+$ $(M+Na)^+$ 267.1161, found m/z 267.1164.

4-(3-fluoro-1-phenylpropyl)-1,1'-biphenyl (12b)



Following the general procedure condition **B**. Colorless oil. (31.5 mg, 54%)。R_f= 0.25 (pentane). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.57 – 7.48 (m, 4H), 7.39 (dd, *J* = 8.4, 6.9 Hz, 2H), 7.35 – 7.23 (m, 7H), 7.22 – 7.16 (m, 1H), 4.40 (dt, *J* = 47.0, 6.0 Hz, 2H), 4.20 (t, *J* = 7.9 Hz, 1H), 2.45 (ddt, *J* = 24.1, 8.0, 6.0 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 143.9, 143.1, 140.9, 139.5, 128.9, 128.8, 128.4, 128.0, 127.4, 127.3, 127.1, 126.7, 82.2 (d, *J* = 164.8 Hz), 46.2 (d, *J* = 5.0 Hz), 36.2 (d, *J* = 19.8 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.8. **HRMS (ESI)** m/z calcd for $C_{21}H_{19}F+$ $(M+Na)^+$ 313.1369, found m/z 313.1367.

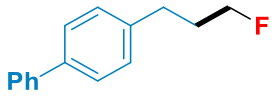
1-(tert-butyl)-4-(3-fluoropropyl)benzene (13b)



Following the general procedure condition **B**. Colorless oil. (30.6 mg, 79%)。R_f= 0.25 (pentane). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.34 (d, *J* = 8.3 Hz, 2H), 7.16 (d, *J* = 8.3 Hz, 2H), 4.48 (dt, *J* = 47.3, 6.0 Hz, 2H), 2.80 – 2.67 (m, 2H), 2.11 – 1.93 (m, 2H), 1.33 (s, 9H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 149.0, 138.2, 128.3, 125.5, 83.4 (d, *J* = 164.6 Hz), 34.5, 32.1 (d, *J* = 19.7 Hz), 31.5, 30.9 (d, *J* = 5.7 Hz). **^{19}F NMR (376**

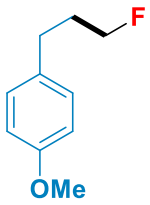
MHz, Chloroform-d): δ -219.8. **HRMS (ESI)** m/z calcd for $C_{13}H_{19}F^{+}$ $(M+Na)^{+}$ 217.1369, found m/z 217.1370.

4-(3-fluoropropyl)-1,1'-biphenyl (14b)



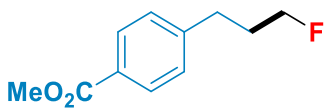
Following the general procedure condition **B**. Colorless oil. (38.6 mg, 90%)。 R_f= 0.25 (pentane). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.66 – 7.58 (m, 2H), 7.58 – 7.53 (m, 2H), 7.45 (dd, *J* = 8.5, 6.8 Hz, 2H), 7.39 – 7.33 (m, 1H), 7.32 – 7.27 (m, 2H), 4.51 (dt, *J* = 47.2, 5.9 Hz, 2H), 2.82 (dd, *J* = 8.7, 6.7 Hz, 2H), 2.17 – 1.98 (m, 2H).**^{13}C NMR (101 MHz, Chloroform-d):** δ 141.1, 140.4, 139.2, 129.0, 128.9, 127.3, 127.2, 127.1, 83.3 (d, *J* = 164.8 Hz), 32.1 (d, *J* = 19.7 Hz), 31.1 (d, *J* = 5.4 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.0. **HRMS (ESI)** m/z calcd for $C_{15}H_{15}F^{+}$ $(M+Na)^{+}$ 237.1056, found m/z 237.1058.

1-(3-fluoropropyl)-4-methoxybenzene (15b)



Following the general procedure condition **B**. Colorless oil. (15.8 mg, 47%)。 R_f= 0.50 (Eluent: PE/EA 50:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.12 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 4.45 (dt, *J* = 47.2, 6.0 Hz, 2H), 3.79 (s, 3H), 2.73 – 2.65 (m, 2H), 2.05 – 1.90 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 158.1, 133.3, 129.5, 114.0, 83.3 (d, *J* = 164.6 Hz), 55.4, 32.4 (d, *J* = 19.6 Hz), 30.5 (d, *J* = 5.4 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.1. **HRMS (ESI)** m/z calcd for $C_{10}H_{13}FO^{+}$ $(M+Na)^{+}$ 191.0848, found m/z 191.0849.

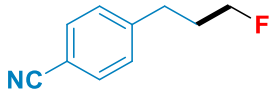
methyl 4-(3-fluoropropyl)benzoate (16b)



Following the general procedure condition **B**. Colorless oil. (25.1 mg, 64%)。 R_f= 0.40

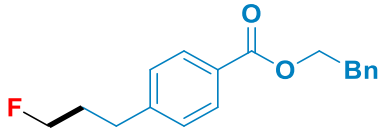
(Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.97 (d, *J* = 8.3 Hz, 2H), 7.27 (d, *J* = 8.3 Hz, 2H), 4.45 (dt, *J* = 47.2, 5.9 Hz, 2H), 3.90 (s, 3H), 2.84 – 2.77 (m, 2H), 2.10 – 1.94 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 167.2, 146.7, 130.0, 128.6, 128.2, 83.0 (d, *J* = 165.3 Hz), 52.1, 31.8 (d, *J* = 19.8 Hz), 31.5 (d, *J* = 5.3 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.2. **HRMS (ESI)** m/z calcd for $C_{11}H_{13}FO_2^+$ $(M+H)^+$ 197.0978, found m/z 197.0979.

4-(3-fluoropropyl)benzonitrile (17b)



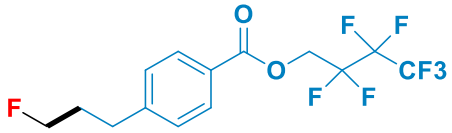
Following the general procedure condition **B**. Colorless oil. (21.2 mg, 65%)。***R*$_f$** = 0.15 (Eluent: PE/EA 50:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.58 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 8.2 Hz, 2H), 4.45 (dt, *J* = 47.1, 5.8 Hz, 2H), 2.84 – 2.79 (m, 2H), 2.07 – 1.94 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 146.9, 132.4, 129.4, 119.1, 110.1, 82.7 (d, *J* = 165.7 Hz), 31.7, 31.6 (d, *J* = 15.9 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.4. **HRMS (ESI)** m/z calcd for $C_{10}H_{10}FN^+$ $(M+Na)^+$ 186.0695, found m/z 186.0698.

phenethyl 4-(3-fluoropropyl)benzoate (18b)



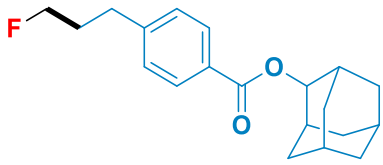
Following the general procedure condition **B**. Colorless oil. (40.1 mg, 70%)。***R*$_f$** = 0.40 (Eluent: PE/EA 20:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.93 (d, *J* = 8.3 Hz, 2H), 7.33 – 7.20 (m, 7H), 4.52 – 4.47 (m, 3H), 4.37 (t, *J* = 5.8 Hz, 1H), 3.06 (t, *J* = 7.0 Hz, 2H), 2.81 – 2.76 (m, 2H), 2.07 – 1.92 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 166.5, 146.7, 138.1, 129.9, 129.1, 128.6, 128.3, 126.7, 65.5, 35.4, 31.8 (d, *J* = 19.9 Hz), 31.5 (d, *J* = 5.2 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.1. **HRMS (ESI)** m/z calcd for $C_{18}H_{19}FO_2^+$ $(M+Na)^+$ 309.1267, found m/z 309.1266

2,2,3,3,4,4,4-heptafluorobutyl 4-(3-fluoropropyl)benzoate (19b)



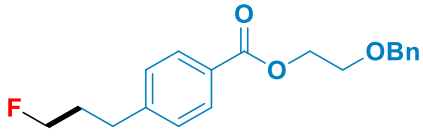
Following the general procedure condition **B**. Colorless oil. (27 mg, 37%)。R_f= 0.40 (Eluent: PE/EA 20:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.00 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 4.80 (tt, *J* = 13.3, 1.4 Hz, 2H), 4.46 (dt, *J* = 47.1, 5.8 Hz, 2H), 2.91 – 2.78 (m, 2H), 2.13 – 1.95 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 165.0, 148.0, 130.4, 129.0, 126.4, 114.3, 82.9 (d, *J* = 165.6 Hz), 60.2, 59.9, 59.6, 31.8 (d, *J* = 19.2 Hz), 31.6. **^{19}F NMR (376 MHz, Chloroform-d):** δ -80.9 (t, *J* = 9.9 Hz), -120.3 (q, *J* = 11.3 Hz), -127.3 – -127.8 (m), -220.3. **HRMS (ESI)** m/z calcd for $C_{14}H_{12}F_8O_2^+$ $(M+Na)^+$ 387.0608, found m/z 387.0609.

(1R,3S,5r,7r)-adamantan-2-yl 4-(3-fluoropropyl)benzoate (20b)



Following the general procedure condition **B**. Colorless oil. (49.4 mg, 78%)。R_f= 0.40 (Eluent: PE/EA 20:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.02 (d, *J* = 8.2 Hz, 2H), 7.28 (d, *J* = 8.2 Hz, 2H), 5.18 (s, 1H), 4.46 (dt, *J* = 47.2, 5.9 Hz, 2H), 2.85 – 2.78 (m, 2H), 2.19 – 2.12 (m, 4H), 2.08 – 1.97 (m, 2H), 1.92 – 1.83 (m, 6H), 1.78 (d, *J* = 2.8 Hz, 2H), 1.63 (d, *J* = 11.9 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 165.9, 146.5, 130.0, 129.2, 128.6, 83.0 (d, *J* = 165.3 Hz), 77.5, 37.5, 36.5, 32.2, 31.9 (d, *J* = 19.8 Hz), 31.6 (d, *J* = 5.1 Hz), 27.5, 27.2. **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.2. **HRMS (ESI)** m/z calcd for $C_{20}H_{25}FO_2^+$ $(M+Na)^+$ 339.1737, found m/z 339.1739.

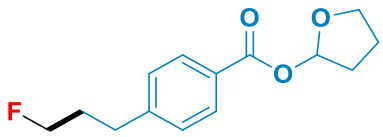
2-(benzyloxy)ethyl 4-(3-fluoropropyl)benzoate (21b)



Following the general procedure condition **B**. Colorless oil. (31.6 mg, 50%)。R_f= 0.20 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.00 (d, *J* = 8.3 Hz, 2H), 7.44 – 7.15 (m, 7H), 4.61 (s, 2H), 4.52 – 4.48 (m, 3H), 4.39 (t, *J* = 5.9 Hz, 1H), 3.82 –

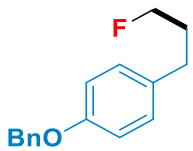
3.79 (m, 2H), 2.81 (d, J = 11.7 Hz, 2H), 2.08 – 1.95 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 166.5, 146.7, 138.0, 130.0, 128.5, 128.5, 128.1, 127.8, 127.7, 82.9 (d, J = 165.3 Hz), 73.2, 68.0, 64.1, 31.7 (d, J = 20.0 Hz), 31.4 (d, J = 5.1 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.2. **HRMS (ESI)** m/z calcd for $C_{19}H_{21}FO_3^+$ $(M+Na)^+$ 339.1373, found m/z 339.1375.

tetrahydrofuran-2-yl 4-(3-fluoropropyl)benzoate (22b)



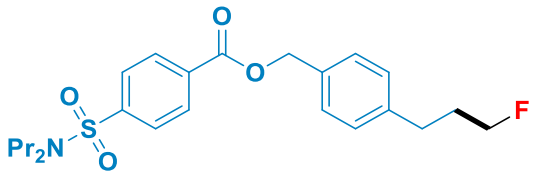
Following the general procedure condition **B**. Colorless oil. (29.8 mg, 59%)。***R**$_f$* = 0.13 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.00 (d, J = 8.3 Hz, 2H), 7.30 (d, J = 8.0 Hz, 2H), 5.57 (td, J = 4.5, 2.3 Hz, 1H), 4.48 (dt, J = 47.2, 5.9 Hz, 2H), 4.08 – 3.92 (m, 4H), 2.84 (d, J = 11.7 Hz, 2H), 2.31 (dtd, J = 16.8, 8.4, 6.3 Hz, 1H), 2.22 – 2.15 (m, 1H), 2.12 – 1.98 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 166.3, 147.0, 130.0, 128.6, 128.1, 82.9 (d, J = 165.4 Hz), 75.4, 73.3, 67.2, 33.1, 31.8 (d, J = 19.8 Hz), 31.5 (d, J = 5.3 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.2. **HRMS (ESI)** m/z calcd for $C_{14}H_{17}FO_3^+$ $(M+Na)^+$ 275.1060, found m/z 275.1061.

1-(benzyloxy)-4-(3-fluoropropyl)benzene (23b)



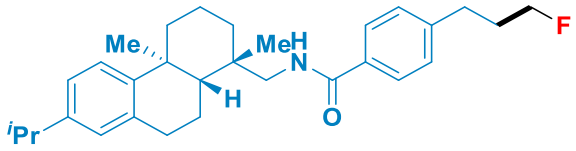
Following the general procedure condition **B**. Colorless oil. (33.7 mg, 69%)。***R**$_f$* = 0.40 (Eluent: PE/EA 50:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.54 – 7.45 (m, 2H), 7.45 – 7.38 (m, 2H), 7.38 – 7.32 (m, 1H), 7.15 (d, J = 8.6 Hz, 2H), 6.95 (d, J = 8.6 Hz, 2H), 5.07 (s, 2H), 4.48 (dt, J = 47.2, 5.9 Hz, 2H), 2.75 – 2.69 (m, 2H), 2.08 – 1.94 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 157.3, 137.3, 133.6, 129.5, 128.7, 128.0, 127.6, 115.0, 83.2 (d, J = 164.7 Hz), 70.2, 32.3 (d, J = 19.7 Hz), 30.5 (d, J = 5.5 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.0. **HRMS (ESI)** m/z calcd for $C_{16}H_{17}FO^+$ $(M+Na)^+$ 267.1161, found m/z 267.1160.

4-(3-fluoropropyl)benzyl 4-(N,N-dipropylsulfamoyl)benzoate (24b)



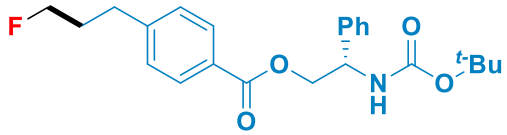
Following the general procedure condition **B**. Colorless oil. (16.6 mg, 19%)。R_f= 0.30 (Eluent: PE/EA 5:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.18 (d, *J* = 8.5 Hz, 2H), 7.86 (d, *J* = 8.5 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 8.1 Hz, 2H), 5.36 (s, 2H), 4.46 (dt, *J* = 47.2, 5.9 Hz, 2H), 3.11 – 3.07 (m, 4H), 2.83 – 2.71 (m, 2H), 2.08 – 1.95 (m, 2H), 1.57 – 1.51 (m, 4H), 0.86 (t, *J* = 7.4 Hz, 6H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 165.3, 144.5, 141.8, 133.6, 133.4, 130.5, 129.0, 128.9, 127.1, 83.1 (d, *J* = 165.0 Hz), 67.3, 50.0, 32.1 (d, *J* = 19.8 Hz), 31.2 (d, *J* = 5.2 Hz), 22.0, 11.3. **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.1. **HRMS (ESI)** m/z calcd for $C_{23}H_{30}FNO_4S^+$ $(M+Na)^+$ 458.1778, found m/z 458.1780.

4-(3-fluoropropyl)-N-(((1S,4aS,10aR)-7-isopropyl-1,4a-dimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthren-1-yl)methyl)benzamide (25b)



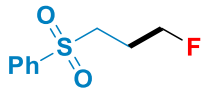
Following the general procedure condition **B**. Colorless oil. (62.1 mg, 69%)。R_f= 0.35 (Eluent: PE/EA 5:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.68 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.1 Hz, 1H), 7.01 (d, *J* = 8.7 Hz, 1H), 6.90 (s, 1H), 6.18 (t, *J* = 6.5 Hz, 1H), 4.44 (dt, *J* = 47.1, 5.8 Hz, 2H), 3.39 (ddd, *J* = 48.2, 13.7, 6.4 Hz, 2H), 2.97 – 2.77 (m, 5H), 2.31 (dt, *J* = 13.1, 3.8 Hz, 1H), 2.06 – 1.95 (m, 3H), 1.82 – 1.68 (m, 3H), 1.51 (ddt, *J* = 9.9, 6.9, 2.8 Hz, 2H), 1.38 (qd, *J* = 8.9, 4.4 Hz, 2H), 1.25 – 1.22 (m, 9H), 1.02 (s, 3H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 167.6, 147.1, 145.7, 144.9, 134.9, 132.8, 128.8, 127.2, 127.1, 124.3, 124.0, 82.9 (d, *J* = 165.3 Hz), 50.3, 45.8, 38.4, 37.8, 37.7, 36.5, 33.5, 31.9 (d, *J* = 19.9 Hz), 31.3 (d, *J* = 5.1 Hz), 30.6, 25.6, 24.1, 19.2, 18.9, 18.8. **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.2. **HRMS (ESI)** m/z calcd for $C_{30}H_{40}FNO^+$ $(M+Na)^+$ 472.2992, found m/z 472.2990.

(S)-2-((tert-butoxycarbonyl)amino)-2-phenylethyl-4-(3-fluoropropyl)benzoate (26b)



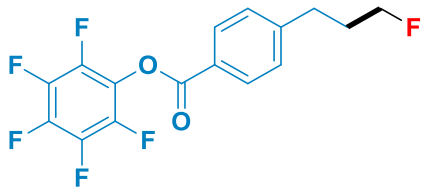
Following the general procedure condition **B**. Colorless oil. (44.2 mg, 55%)。R_f= 0.20 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.94 (d, *J* = 8.3 Hz, 2H), 7.39 – 7.35 (m, 4H), 7.33 – 7.28 (m, 1H), 7.25 (d, *J* = 8.1 Hz, 2H), 5.24 (d, *J* = 8.2 Hz, 1H), 5.15 (s, 1H), 4.56 – 4.47 (m, 3H), 4.38 (t, *J* = 5.9 Hz, 1H), 2.86 – 2.75 (m, 2H), 2.11 – 1.92 (m, 2H), 1.39 (s, 9H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 166.5, 155.3, 147.0, 130.1, 128.9, 128.7, 128.0, 127.8, 126.7, 82.9 (d, *J* = 165.4 Hz), 67.2, 53.9, 31.8 (d, *J* = 19.9 Hz), 31.5 (d, *J* = 5.3 Hz), 28.4. **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.2. **HRMS (ESI)** m/z calcd for $C_{23}H_{28}FNO_4^+$ $(M+Na)^+$ 424.1900, found m/z 424.1900.

((3-fluoropropyl)sulfonyl)benzene (27b)



Following the general procedure condition **B**. Colorless oil. (29.5 mg, 73%)。R_f= 0.25 (Eluent: PE/EA 20:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.13 – 7.73 (m, 2H), 7.71 – 7.62 (m, 1H), 7.62 – 7.48 (m, 2H), 4.50 (dt, *J* = 46.9, 5.7 Hz, 2H), 3.24 – 3.20 (m, 2H), 2.19 – 2.06 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 139.0, 134.0, 129.5, 128.1, 81.6 (d, *J* = 167.7 Hz), 52.6 (d, *J* = 4.2 Hz), 24.2 (d, *J* = 20.8 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.5. **HRMS (ESI)** m/z calcd for $C_9H_{11}FO_2S^+$ $(M+Na)^+$ 225.0362, found m/z 225.0363.

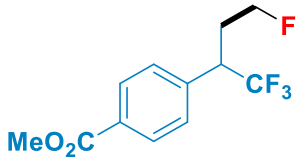
perfluorophenyl 4-(3-fluoropropyl)benzoate (28b)



Following the general procedure condition **B**. Colorless oil. (35.5 mg, 51%)。R_f= 0.45 (Eluent: PE/EA 20:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.14 (d, *J* = 8.3 Hz, 2H),

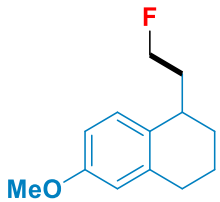
7.39 (d, J = 8.3 Hz, 2H), 4.48 (dt, J = 47.1, 5.8 Hz, 2H), 2.90 – 2.85 (m, 2H), 2.13 – 1.99 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 162.5, 148.9, 142.7, 140.8, 140.1, 139.2, 136.7, 131.0, 129.1, 124.8, 82.7 (d, J = 165.7 Hz), 31.7 (d, J = 6.4 Hz), 31.6 (d, J = 8.5 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -152.4 – -152.7 (m), -158.2 (t, J = 21.9 Hz), -162.3 – -162.7 (m), -220.4. **HRMS (ESI)** m/z calcd for $C_{16}H_{10}F_6O_2^+$ $(M+Na)^+$ 371.0483, found m/z 371.0485.

methyl 4-(1,1,1,4-tetrafluorobutan-2-yl)benzoate (29b)



Following the general procedure condition **B**. Colorless oil. (46.5 mg, 88%)。***R$_f$***= 0.20 (Eluent: PE/EA 20:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.05 (d, J = 8.4 Hz, 2H), 7.39 (d, J = 8.1 Hz, 2H), 4.48 (ddt, J = 46.2, 9.4, 4.7 Hz, 1H), 4.13 (dtd, J = 47.4, 9.6, 3.7 Hz, 1H), 3.92 (s, 3H), 3.67 – 3.55 (m, 1H), 2.52 (dddt, J = 20.9, 14.6, 9.6, 4.6 Hz, 1H), 2.13 (dddt, J = 33.8, 14.9, 11.1, 3.9 Hz, 1H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 166.6, 138.7, 130.6, 130.2, 129.3, 128.0, 125.2, 80.1 (d, J = 167.1 Hz), 52.3, 46.1 (qd, J = 27.4, 4.4 Hz), 30.1 (d, J = 20.5 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -69.5, -222.6. **HRMS (ESI)** m/z calcd for $C_{12}H_{12}F_4O_2^+$ $(M+Na)^+$ 287.0671, found m/z 287.0672.

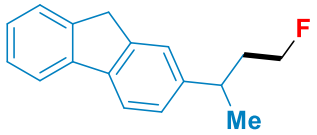
1-(2-fluoroethyl)-6-methoxy-1,2,3,4-tetrahydronaphthalene (30b)



Following the general procedure condition **B**. Colorless oil. (4.2 mg, 10%)。***R$_f$***= 0.50 (Eluent: PE/EA 50:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.09 (d, J = 8.5 Hz, 1H), 6.77 – 6.69 (m, 1H), 6.63 (d, J = 2.8 Hz, 1H), 4.65 – 4.49 (m, 2H), 3.79 (s, 3H), 3.03 – 2.92 (m, 1H), 2.78 – 2.73 (m, 2H), 2.16 – 2.05 (m, 1H), 1.96 – 1.82 (m, 3H), 1.77 – 1.69 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 157.6, 138.4, 132.3, 129.7, 113.8, 112.1, 82.6 (d, J = 164.3 Hz), 55.3, 37.5 (d, J = 19.1 Hz), 33.2 (d, J = 4.9 Hz), 30.0, 28.0, 19.6. **^{19}F NMR (376 MHz, Chloroform-d):** δ -218.9. **HRMS (ESI)** m/z

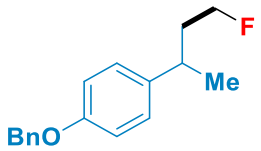
calcd for $C_{13}H_{17}FO^+$ $(M+Na)^+$ 231.1161, found m/z 231.1160.

2-(4-fluorobutan-2-yl)-9H-fluorene (31b)



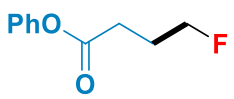
Following the general procedure condition **B**. Colorless oil. (25.9 mg, 54%)。R_f= 0.23 (pentane). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.76 (dd, *J* = 15.7, 7.7 Hz, 2H), 7.55 (d, *J* = 7.4 Hz, 1H), 7.48 – 7.19 (m, 4H), 4.55 – 4.28 (m, 2H), 3.90 (s, 2H), 3.09 – 2.96 (m, 1H), 2.15 – 1.92 (m, 2H), 1.38 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 145.1, 143.8, 143.3, 141.8, 140.2, 126.8, 126.5, 125.8, 125.1, 123.8, 120.0, 119.8, 82.6 (d, *J* = 164.0 Hz), 39.0 (d, *J* = 19.3 Hz), 37.0, 36.0 (d, *J* = 5.1 Hz), 22.5. **^{19}F NMR (376 MHz, Chloroform-d):** δ -219.8. **HRMS (ESI)** m/z calcd for $C_{17}H_{17}F^+$ $(M+Na)^+$ 263.1212, found m/z 263.1215.

1-butoxy-4-(4-fluorobutan-2-yl)benzene (32b)



Following the general procedure condition **B**. Colorless oil. (14.4 mg, 32%)。R_f= 0.40 (Eluent: PE/EA 50:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.10 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 4.47 – 4.20 (m, 2H), 3.94 (t, *J* = 6.5 Hz, 2H), 2.95 – 2.80 (m, 1H), 2.05 – 1.81 (m, 2H), 1.79 – 1.72 (m, 2H), 1.52 – 1.46 (m, 2H), 1.28 – 1.25 (m, 3H), 0.97 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 138.1, 127.9, 114.6, 82.6 (d, *J* = 163.7 Hz), 67.8, 39.0 (d, *J* = 19.2 Hz), 35.0 (d, *J* = 5.1 Hz), 31.5, 22.4, 19.4, 14.0. **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.0. **HRMS (ESI)** m/z calcd for $C_{14}H_{21}FO^+$ (M+H)+ 247.1474, found m/z 247.1476.

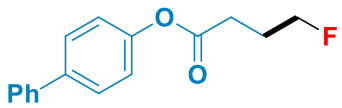
phenyl 4-fluorobutanoate (33b)



Following the general procedure condition **B**. Colorless oil. (13.1 mg, 36%)。R_f= 0.32 (Eluent: PE/EA 20:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.41 – 7.30 (m, 2H),

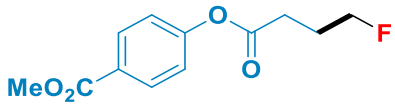
7.20 (td, J = 7.6, 1.4 Hz, 1H), 7.14 – 6.96 (m, 2H), 4.54 (dt, J = 47.1, 5.7 Hz, 2H), 2.70 (t, J = 7.3 Hz, 2H), 2.11 (dtt, J = 26.2, 7.1, 5.7 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.6, 150.7, 129.6, 126.0, 121.6, 82.9 (d, J = 165.6 Hz), 30.3 (d, J = 4.9 Hz), 25.9 (d, J = 20.1 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.8. **HRMS (ESI)** m/z calcd for $C_{10}H_{11}FO_2^+$ $(M+Na)^+$ 205.0641, found m/z 205.0639.

[1,1'-biphenyl]-4-yl 4-fluorobutanoate (34b)



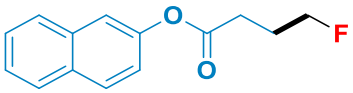
Following the general procedure condition **B**. Colorless oil. (30.5 mg, 59%)。***R**$_f$* = 0.32 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.64 – 7.56 (m, 4H), 7.51 – 7.42 (m, 2H), 7.41 – 7.31 (m, 1H), 7.25 – 7.11 (m, 2H), 4.60 (dt, J = 47.1, 5.8 Hz, 2H), 2.77 (t, J = 7.3 Hz, 2H), 2.18 (dtt, J = 26.2, 7.3, 5.7 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.6, 150.1, 140.4, 139.2, 128.9, 128.3, 127.5, 127.2, 121.9, 82.9 (d, J = 165.5 Hz), 30.3 (d, J = 5.0 Hz), 25.9 (d, J = 20.2 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.7. **HRMS (ESI)** m/z calcd for $C_{16}H_{15}FO_2^+$ $(M+Na)^+$ 281.0954, found m/z 281.0953.

methyl 4-((4-fluorobutanoyl)oxy)benzoate (35b)



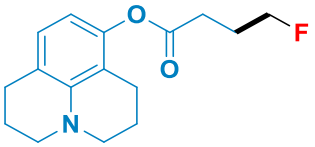
Following the general procedure condition **B**. Colorless oil. (31.9 mg, 66%)。***R**$_f$* = 0.36 (Eluent: PE/EA 5:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.06 (d, J = 8.7 Hz, 2H), 7.16 (d, J = 8.7 Hz, 2H), 4.56 (dt, J = 47.1, 5.7 Hz, 2H), 3.90 (s, 3H), 2.74 (t, J = 7.3 Hz, 2H), 2.13 (dtt, J = 26.2, 7.2, 5.7 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.0, 166.4, 154.3, 131.3, 127.9, 121.6, 82.8 (d, J = 165.8 Hz), 52.3, 30.3 (d, J = 4.9 Hz), 25.8 (d, J = 20.3 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.9. **HRMS (ESI)** m/z calcd for $C_{12}H_{13}FO_4^+$ $(M+Na)^+$ 263.0696, found m/z 263.0697.

naphthalen-2-yl 4-fluorobutanoate (36b)



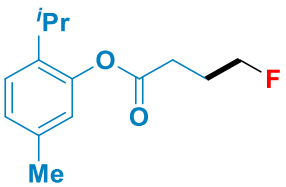
Following the general procedure condition **B**. Colorless oil. (26.2 mg, 35%)。R_f= 0.45 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.90 – 7.79 (m, 3H), 7.57 (d, *J* = 2.3 Hz, 1H), 7.54 – 7.42 (m, 2H), 7.24 (dd, *J* = 8.8, 2.3 Hz, 1H), 4.61 (dt, *J* = 47.1, 5.7 Hz, 2H), 2.80 (t, *J* = 7.3 Hz, 2H), 2.19 (dtt, *J* = 26.2, 7.3, 5.7 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.7, 148.3, 133.8, 131.5, 129.5, 127.8, 127.7, 126.6, 125.8, 121.1, 118.5, 82.9 (d, *J* = 165.5 Hz), 30.2 (d, *J* = 4.8 Hz), 25.8 (d, *J* = 20.3 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.7. **HRMS (ESI)** m/z calcd for $C_{14}H_{13}FO_2^+$ $(M+Na)^+$ 255.0798, found m/z 255.0799.

2,3,6,7-tetrahydro-1H,5H-pyrido[3,2,1-ij]quinolin-8-yl 4-fluorobutanoate (37b)



Following the general procedure condition **B**. Colorless oil. (34.4 mg, 62%)。R_f= 0.52 (Eluent: PE/EA 5:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 6.78 (d, *J* = 8.1 Hz, 1H), 6.24 (d, *J* = 8.1 Hz, 1H), 4.57 (dt, *J* = 47.1, 5.8 Hz, 2H), 3.12 (td, *J* = 6.8, 5.5 Hz, 4H), 2.73 (dt, *J* = 12.3, 6.9 Hz, 4H), 2.55 (t, *J* = 6.6 Hz, 2H), 2.15 (dtt, *J* = 26.2, 7.4, 5.8 Hz, 2H), 1.99 – 1.93 (m, 4H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.4, 147.3, 144.0, 127.0, 119.3, 113.5, 108.7, 83.0 (d, *J* = 165.3 Hz), 50.0, 49.5, 30.0 (d, *J* = 5.1 Hz), 27.6, 26.0 (d, *J* = 20.4 Hz), 22.0, 21.6, 21.4. **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.7. **HRMS (ESI)** m/z calcd for $C_{16}H_{20}FNO_2^+$ $(M+Na)^+$ 300.1376, found m/z 300.1377.

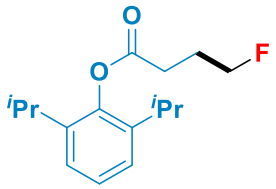
2-isopropyl-5-methylphenyl 4-fluorobutanoate (38b)



Following the general procedure condition **B**. Colorless oil. (27.6 mg, 58%)。R_f= 0.32 (Eluent: PE/EA 20:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.21 (d, *J* = 7.9 Hz, 1H), 7.04 (d, *J* = 7.5 Hz, 1H), 6.82 (s, 1H), 4.58 (dt, *J* = 47.2, 5.8 Hz, 2H), 2.97 (hept, *J* =

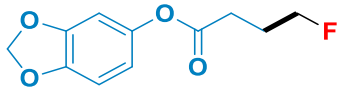
6.9 Hz, 1H), 2.76 (t, *J* = 7.3 Hz, 2H), 2.33 (s, 3H), 2.17 (dtt, *J* = 26.2, 7.4, 5.8 Hz, 2H), 1.20 (d, *J* = 6.9 Hz, 7H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.8, 147.9, 137.1, 136.7, 127.3, 126.6, 122.8, 82.9 (d, *J* = 165.5 Hz), 30.1 (d, *J* = 5.0 Hz), 27.2, 25.9 (d, *J* = 20.1 Hz), 23.1, 20.9. **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.8. **HRMS (ESI)** m/z calcd for $C_{14}H_{19}FO_2^+$ $(M+Na)^+$ 261.1267, found m/z 261.1268.

2,6-diisopropylphenyl 4-fluorobutanoate (39b)



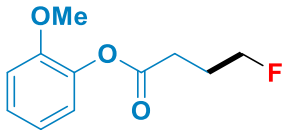
Following the general procedure condition **B**. Colorless oil. (33.0 mg, 62%)。***R*$_f$** = 0.52 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.35 – 7.22 (m, 3H), 4.66 (dt, *J* = 47.1, 5.7 Hz, 2H), 2.98 (hept, *J* = 6.9 Hz, 2H), 2.89 (t, *J* = 7.4 Hz, 2H), 2.27 (dtt, *J* = 26.2, 7.4, 5.7 Hz, 2H), 1.29 (d, *J* = 6.9 Hz, 13H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.6, 145.6, 140.4, 126.7, 124.1, 82.9 (d, *J* = 165.5 Hz), 29.8 (d, *J* = 4.9 Hz), 27.7, 25.9 (d, *J* = 20.3 Hz), 23.3 (d, *J* = 66.2 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -221.1. **HRMS (ESI)** m/z calcd for $C_{16}H_{23}FO_2^+$ $(M+Na)^+$ 289.1580, found m/z 289.1581.

benzo[d][1,3]dioxol-5-yl 4-fluorobutanoate (40b)



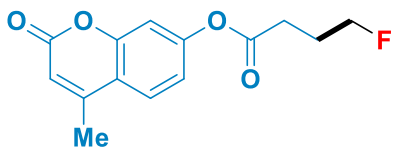
Following the general procedure condition **B**. Colorless oil. (20.8 mg, 46%)。***R*$_f$** = 0.38 (Eluent: PE/EA 5:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 6.77 (d, *J* = 8.4 Hz, 1H), 6.60 (d, *J* = 2.3 Hz, 1H), 6.52 (dd, *J* = 8.4, 2.3 Hz, 1H), 5.97 (s, 2H), 4.55 (dt, *J* = 47.2, 5.7 Hz, 2H), 2.69 (t, *J* = 7.3 Hz, 2H), 2.12 (dtt, *J* = 26.2, 7.3, 5.7 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.9, 148.1, 145.5, 145.0, 114.0, 108.1, 103.8, 101.8, 82.9 (d, *J* = 165.7 Hz), 30.2 (d, *J* = 4.9 Hz), 25.9 (d, *J* = 20.3 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.8. **HRMS (ESI)** m/z calcd for $C_{11}H_{11}FO_4^+$ $(M+Na)^+$ 249.0539, found m/z 249.0538.

2-methoxyphenyl 4-fluorobutanoate (41b)



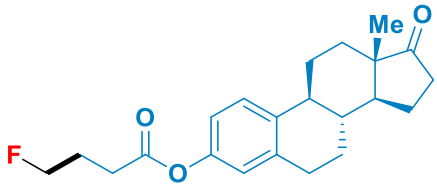
Following the general procedure condition **B**. Colorless oil. (26.3 mg, 62%)。R_f= 0.25 (Eluent: PE/EA 10:1). **^{1}H NMR (500 MHz, Chloroform-d):** δ 7.21 (td, *J* = 7.9, 1.7 Hz, 1H), 7.04 (dd, *J* = 7.8, 1.7 Hz, 1H), 7.02 – 6.90 (m, 2H), 4.58 (dt, *J* = 47.2, 5.9 Hz, 2H), 3.82 (s, 3H), 2.75 (t, *J* = 7.3 Hz, 2H), 2.16 (dtt, *J* = 26.0, 7.3, 5.8 Hz, 2H). **^{13}C NMR (126 MHz, Chloroform-d):** δ 171.1, 151.2, 139.8, 127.0, 122.8, 120.9, 112.5, 82.9 (d, *J* = 165.3 Hz), 55.9, 29.8 (d, *J* = 5.3 Hz), 26.0 (d, *J* = 20.3 Hz). **^{19}F NMR (471 MHz, Chloroform-d):** δ -220.7. **HRMS (ESI)** m/z calcd for $C_{11}H_{13}FO_3^+$ $(M+Na)^+$ 235.0747, found m/z 235.0748.

4-methyl-2-oxo-2H-chromen-7-yl 4-fluorobutanoate (42b)



Following the general procedure condition **B**. Colorless oil. (43.3 mg, 82%)。R_f= 0.36 (Eluent: PE/EA 1:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.60 (d, *J* = 8.7 Hz, 1H), 7.11 (d, *J* = 2.2 Hz, 1H), 7.07 (dd, *J* = 8.6, 2.3 Hz, 1H), 6.27 (d, *J* = 1.3 Hz, 1H), 4.57 (dt, *J* = 47.1, 5.7 Hz, 2H), 2.77 (t, *J* = 7.2 Hz, 2H), 2.43 (d, *J* = 1.3 Hz, 3H), 2.23 – 2.07 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.0, 160.6, 154.3, 153.1, 152.1, 125.5, 118.2, 118.0, 114.7, 110.6, 82.8 (d, *J* = 165.8 Hz), 30.3 (d, *J* = 4.6 Hz), 25.8 (d, *J* = 20.3 Hz), 18.8. **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.7. **HRMS (ESI)** m/z calcd for $C_{14}H_{13}FO_4^+$ $(M+Na)^+$ 287.0696, found m/z 287.0697.

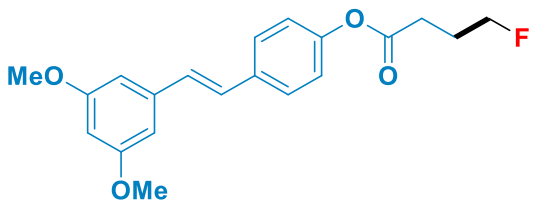
(8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl 4-fluorobutanoate (43b)



Following the general procedure condition **B**. Colorless oil. (36.6 mg, 51%)。R_f= 0.26

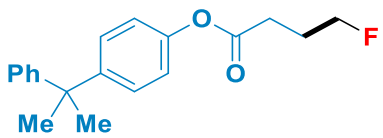
(Eluent: PE/EA 3:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.28 (d, *J* = 8.0 Hz, 1H), 6.85 (dd, *J* = 8.4, 2.6 Hz, 1H), 6.81 (d, *J* = 2.5 Hz, 1H), 4.56 (dt, *J* = 47.1, 5.8 Hz, 2H), 2.90 (dd, *J* = 8.5, 3.8 Hz, 2H), 2.70 (t, *J* = 7.3 Hz, 2H), 2.50 (dd, *J* = 18.8, 8.6 Hz, 1H), 2.45 – 2.35 (m, 1H), 2.28 (td, *J* = 10.9, 3.9 Hz, 1H), 2.20 – 1.94 (m, 7H), 1.64 – 1.41 (m, 7H), 0.90 (s, 3H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 220.8, 171.8, 148.5, 138.1, 137.5, 126.5, 121.6, 118.7, 82.9 (d, *J* = 165.5 Hz), 50.5, 48.0, 44.2, 38.1, 35.9, 31.6, 30.2 (d, *J* = 5.0 Hz), 29.5, 26.4, 25.9 (d, *J* = 20.1 Hz), 25.8, 21.6, 13.9. **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.7. **HRMS (ESI)** m/z calcd for $C_{22}H_{27}FO_3^+$ $(M+Na)^+$ 381.1842, found m/z 381.1843.

(E)-4-(3,5-dimethoxystyryl)phenyl 4-fluorobutanoate (44b)



Following the general procedure condition **B**. Colorless oil. (44.1 mg, 64%)。***R*$_f$** = 0.40 (Eluent: PE/EA 5:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.51 (d, *J* = 8.6 Hz, 2H), 7.10 – 6.96 (m, 4H), 6.66 (d, *J* = 2.2 Hz, 2H), 6.41 (t, *J* = 2.3 Hz, 1H), 4.58 (dt, *J* = 47.1, 5.7 Hz, 2H), 3.83 (s, 6H), 2.74 (t, *J* = 7.3 Hz, 2H), 2.15 (dtt, *J* = 26.2, 7.3, 5.7 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.6, 161.1, 150.2, 139.3, 135.1, 129.1, 128.3, 127.6, 121.9, 104.7, 100.2, 82.9 (d, *J* = 165.6 Hz), 55.5, 30.3 (d, *J* = 4.7 Hz), 25.9 (d, *J* = 20.3 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.8. **HRMS (ESI)** m/z calcd for $C_{20}H_{21}FO_4^+$ $(M+Na)^+$ 367.1322, found m/z 367.1321.

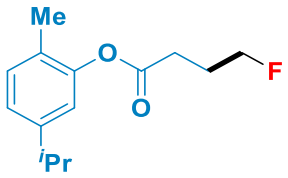
4-(2-phenylpropan-2-yl)phenyl 4-fluorobutanoate (45b)



Following the general procedure condition **B**. Colorless oil. (19.8mg, 33%)。***R*$_f$** = 0.50 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.31 – 7.17 (m, 7H), 7.03 – 6.97 (m, 2H), 4.58 (dt, *J* = 47.1, 5.8 Hz, 2H), 2.73 (t, *J* = 7.3 Hz, 2H), 2.15 (dtt, *J* = 26.2, 7.3, 5.7 Hz, 2H), 1.70 (s, 6H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.7, 150.4, 148.5, 148.4, 128.2, 128.0, 126.9, 125.9, 120.9, 82.9 (d, *J* = 165.5 Hz), 42.8,

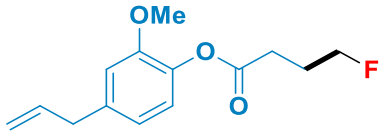
30.9, 30.3 (d, J = 4.9 Hz), 25.9 (d, J = 20.2 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.7. **HRMS (ESI)** m/z calcd for $C_{19}H_{21}FO_2^+$ $(M+Na)^+$ 323.1424, found m/z 323.1425.

5-isopropyl-2-methylphenyl 4-fluorobutanoate (46b)



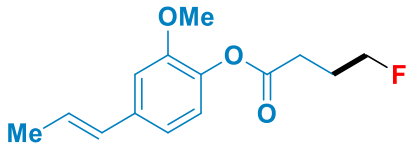
Following the general procedure condition **B**. Colorless oil. (35.7 mg, 75%)。***R$_f$*** = 0.50 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.16 (d, J = 7.8 Hz, 1H), 7.03 (dd, J = 7.8, 1.8 Hz, 1H), 6.88 (d, J = 1.8 Hz, 1H), 4.59 (dt, J = 47.2, 5.8 Hz, 2H), 2.89 (hept, J = 6.9 Hz, 1H), 2.77 (t, J = 7.3 Hz, 2H), 2.24 – 2.10 (m, 5H), 1.25 (d, J = 6.9 Hz, 7H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.3, 149.3, 148.2, 131.0, 127.2, 124.3, 119.8, 82.9 (d, J = 165.5 Hz), 33.7, 30.0 (d, J = 5.0 Hz), 25.9 (d, J = 20.3 Hz), 24.0, 15.9. **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.8. **HRMS (ESI)** m/z calcd for $C_{14}H_{19}FO_2^+$ $(M+Na)^+$ 261.1267, found m/z 261.1269.

4-allyl-2-methoxyphenyl 4-fluorobutanoate (47b)



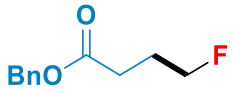
Following the general procedure condition **B**. Colorless oil. (32.8 mg, 65%)。***R$_f$*** = 0.50 (Eluent: PE/EA 5:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 6.95 (d, J = 7.9 Hz, 1H), 6.82 – 6.74 (m, 2H), 5.97 (ddt, J = 16.8, 10.2, 6.7 Hz, 1H), 5.16 – 5.04 (m, 2H), 4.58 (dt, J = 47.1, 5.8 Hz, 2H), 3.81 (s, 3H), 3.38 (d, J = 6.7 Hz, 2H), 2.74 (t, J = 7.3 Hz, 2H), 2.24 – 2.07 (m, 2H).**^{13}C NMR (101 MHz, Chloroform-d):** δ 171.2, 150.9, 139.1, 138.0, 137.1, 122.5, 120.8, 116.2, 112.8, 82.9 (d, J = 165.2 Hz), 55.9, 40.2, 29.8 (d, J = 5.4 Hz), 26.0 (d, J = 20.4 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.7. **HRMS (ESI)** m/z calcd for $C_{14}H_{17}FO_3^+$ $(M+Na)^+$ 275.1060, found m/z 275.1061.

(E)-2-methoxy-4-(prop-1-en-1-yl)phenyl 4-fluorobutanoate (48b)



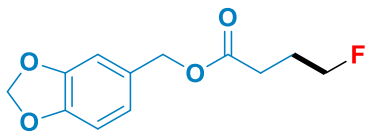
Following the general procedure condition **B**. Colorless oil. (25.2 mg, 50%)。R_f= 0.50 (Eluent: PE/EA 5:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 6.99 – 6.86 (m, 3H), 6.37 (dq, *J* = 15.7, 1.7 Hz, 1H), 6.19 (dq, *J* = 15.7, 6.6 Hz, 1H), 4.57 (dt, *J* = 47.1, 5.9 Hz, 2H), 3.83 (s, 3H), 2.74 (t, *J* = 7.3 Hz, 2H), 2.15 (dtt, *J* = 26.1, 7.3, 5.8 Hz, 2H), 1.89 (ddd, *J* = 8.3, 7.0, 1.7 Hz, 3H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.2, 151.0, 138.6, 137.2, 130.6, 126.3, 122.7, 118.5, 109.7, 82.9 (d, *J* = 165.3 Hz), 55.9, 29.8 (d, *J* = 5.1 Hz), 26.0 (d, *J* = 20.4 Hz), 18.5. **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.7. **HRMS (ESI)** m/z calcd for $C_{14}H_{17}FO_3^+$ $(M+Na)^+$ 275.1060, found m/z 275.1062.

benzyl 4-fluorobutanoate (49b)



Following the general procedure condition **B**. Colorless oil. (18.8 mg, 48%)。R_f= 0.30 (Eluent: PE/EA 20:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.65 – 7.21 (m, 5H), 5.14 (s, 2H), 4.49 (dt, *J* = 47.2, 5.8 Hz, 2H), 2.52 (t, *J* = 7.3 Hz, 2H), 2.05 (dtt, *J* = 26.2, 7.3, 5.8 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 172.8, 136.0, 128.7, 128.4, 128.3, 83.0 (d, *J* = 165.3 Hz), 66.5, 30.1 (d, *J* = 5.0 Hz), 25.9 (d, *J* = 20.4 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.6. **HRMS (ESI)** m/z calcd for $C_{11}H_{13}FO_2^+$ $(M+Na)^+$ 219.0798, found m/z 219.0799.

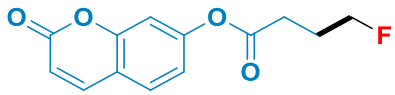
benzo[d][1,3]dioxol-5-ylmethyl 4-fluorobutanoate (50b)



Following the general procedure condition **B**. Colorless oil. (23.5 mg, 49%)。R_f= 0.30 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 6.96 – 6.64 (m, 3H), 5.96 (s, 2H), 5.02 (s, 2H), 4.47 (dt, *J* = 47.1, 5.8 Hz, 2H), 2.49 (t, *J* = 7.3 Hz, 2H), 2.02 (dtt, *J* = 26.2, 7.3, 5.7 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 172.8, 147.9, 147.8, 129.7, 122.4, 109.1, 108.4, 101.3, 83.0 (d, *J* = 165.5 Hz), 66.5, 30.1 (d, *J* = 5.0

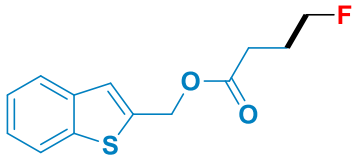
Hz), 25.8 (d, J = 20.2 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.6. **HRMS (ESI)** m/z calcd for $C_{12}H_{13}FO_4^+$ $(M+Na)^+$ 263.0696, found m/z 263.0696.

2-oxo-2H-chromen-7-yl 4-fluorobutanoate (51b)



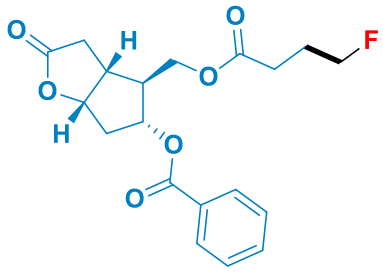
Following the general procedure condition **B**. Colorless oil. (31.0 mg, 62%)。R_f= 0.25 (Eluent: PE/EA 5:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.69 (d, J = 9.5 Hz, 1H), 7.48 (d, J = 8.4 Hz, 1H), 7.11 (d, J = 2.3 Hz, 1H), 7.04 (dd, J = 8.4, 2.2 Hz, 1H), 6.38 (d, J = 9.6 Hz, 1H), 4.57 (dt, J = 47.1, 5.7 Hz, 2H), 2.76 (t, J = 7.2 Hz, 2H), 2.15 (dtt, J = 26.6, 7.3, 5.6 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 170.9, 160.5, 154.8, 153.2, 143.0, 128.7, 118.5, 116.8, 116.2, 110.5, 82.8 (d, J = 165.8 Hz), 30.3 (d, J = 4.9 Hz), 25.7 (d, J = 20.2 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.9. **HRMS (ESI)** m/z calcd for $C_{13}H_{11}FO_4^+$ $(M+Na)^+$ 273.0539, found m/z 273.0540.

benzo[b]thiophen-2-ylmethyl 4-fluorobutanoate (52b)



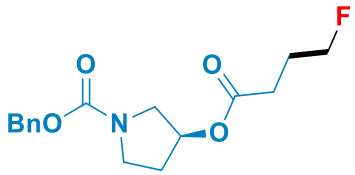
Following the general procedure condition **B**. Colorless oil. (21.2 mg, 42%)。R_f= 0.38 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.86 – 7.71 (m, 2H), 7.41 – 7.28 (m, 3H), 5.38 (d, J = 0.8 Hz, 2H), 4.49 (dt, J = 47.1, 5.8 Hz, 2H), 2.54 (t, J = 7.3 Hz, 2H), 2.05 (dtt, J = 26.2, 7.4, 5.8 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 172.6, 140.5, 139.3, 138.8, 124.9, 124.6, 124.5, 123.9, 122.5, 82.9 (d, J = 165.4 Hz), 61.5, 30.0 (d, J = 5.2 Hz), 25.8 (d, J = 20.3 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.7. **HRMS (ESI)** m/z calcd for $C_{13}H_{13}FO_2S^+$ $(M+Na)^+$ 275.0518, found m/z 275.0519.

(3aS,4R,5R,6aS)-4-((4-fluorobutanoyl)oxy)-2-oxohexahydro-2H-cyclopenta[b]furan-5-yl benzoate (53b)



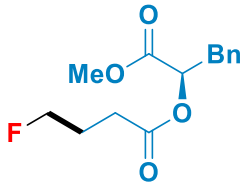
Following the general procedure condition **B**. Colorless oil. (38.5 mg, 55%)。R_f= 0.25 (Eluent: PE/EA 2:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.97 (d, *J* = 7.1 Hz, 2H), 7.55 (t, *J* = 7.5 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 2H), 5.34 (dt, *J* = 6.2, 3.7 Hz, 1H), 5.07 (td, *J* = 6.5, 1.7 Hz, 1H), 4.46 (dt, *J* = 47.1, 5.7 Hz, 2H), 4.18 – 4.08 (m, 2H), 2.92 (dd, *J* = 18.1, 10.3 Hz, 1H), 2.83 – 2.73 (m, 1H), 2.56 (dd, *J* = 18.1, 2.3 Hz, 2H), 2.53 – 2.43 (m, 5H), 2.40 – 2.30 (m, 1H), 2.00 (dtt, *J* = 26.2, 7.2, 5.7 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 176.3, 172.7, 166.0, 133.5, 129.7, 129.5, 128.6, 84.2, 82.9 (d, *J* = 165.5 Hz), 77.3, 64.1, 51.7, 40.6, 38.2, 35.8, 30.0 (d, *J* = 4.8 Hz), 25.7 (d, *J* = 20.2 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.8. **HRMS (ESI)** m/z calcd for $C_{19}H_{21}FO_6^+$ $(M+Na)^+$ 387.1220, found m/z 387.1243.

benzyl (S)-3-((4-fluorobutanoyl)oxy)pyrrolidine-1-carboxylate (54b)



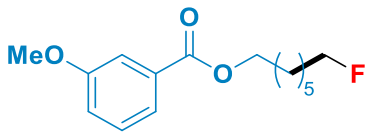
Following the general procedure condition **B**. Colorless oil. (35.9 mg, 58%)。R_f= 0.20 (Eluent: PE/EA 2:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.47 – 7.27 (m, 5H), 5.31 (s, 1H), 5.14 (s, 2H), 4.47 (dt, *J* = 47.1, 5.8 Hz, 2H), 3.64 – 3.46 (m, 4H), 2.45 (t, *J* = 7.3 Hz, 2H), 2.09 – 1.93 (m, 4H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 172.5, 154.9, 136.9, 128.6, 128.1, 128.0, 82.9 (d, *J* = 165.6 Hz), 73.5 (d, *J* = 81.4 Hz), 67.0, 51.9 (d, *J* = 36.1 Hz), 44.2 (d, *J* = 34.9 Hz), 31.2 (d, *J* = 81.0 Hz), 30.2 (d, *J* = 4.8 Hz), 25.8 (d, *J* = 20.3 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.6. **HRMS (ESI)** m/z calcd for $C_{16}H_{20}FNO_4^+$ $(M+Na)^+$ 332.1274, found m/z 332.1273.

(R)-1-methoxy-1-oxo-3-phenylpropan-2-yl 4-fluorobutanoate (55b)



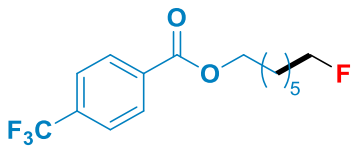
Following the general procedure condition **B**. Colorless oil. (31.7 mg, 59%)。R_f= 0.35 (Eluent: PE/EA 5:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.34 – 7.21 (m, 5H), 5.26 (dd, *J* = 8.9, 4.3 Hz, 1H), 4.42 (dt, *J* = 47.1, 5.8 Hz, 2H), 3.74 (s, 3H), 3.26 – 3.05 (m, 2H), 2.61 – 2.41 (m, 2H), 1.98 (dtt, *J* = 26.1, 7.3, 5.8 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 172.3, 170.1, 136.0, 129.4, 128.6, 127.2, 82.7 (d, *J* = 165.3 Hz), 73.1 (d, *J* = 11.6 Hz), 52.4 (d, *J* = 11.8 Hz), 37.4, 29.7 (d, *J* = 5.2 Hz), 25.7 (d, *J* = 20.2 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.8. **HRMS (ESI)** m/z calcd for $C_{14}H_{17}FO_4^+$ $(M+Na)^+$ 291.1009, found m/z 291.1010.

7-fluoroheptyl 3-methoxybenzoate (56b)



Following the general procedure condition **C**. Colorless oil. (39.7 mg, 74%)。R_f= 0.40 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.63 (dt, *J* = 7.6, 1.3 Hz, 1H), 7.56 (dd, *J* = 2.8, 1.5 Hz, 1H), 7.34 (t, *J* = 7.9 Hz, 1H), 7.10 (ddd, *J* = 8.3, 2.7, 1.0 Hz, 1H), 4.44 (dt, *J* = 47.4, 6.1 Hz, 2H), 4.31 (t, *J* = 6.7 Hz, 2H), 3.85 (s, 3H), 1.81 – 1.63 (m, 5H), 1.47 – 1.41 (m, 5H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 166.7, 159.7, 131.9, 129.5, 122.1, 119.4, 114.2, 84.2 (d, *J* = 164.0 Hz), 65.2, 55.6, 30.5 (d, *J* = 19.4 Hz), 29.0, 28.7, 26.1, 25.2 (d, *J* = 5.4 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -218.2. **HRMS (ESI)** m/z calcd for $C_{15}H_{21}FO_3^+$ $(M+Na)^+$ 291.1373, found m/z 291.1374.

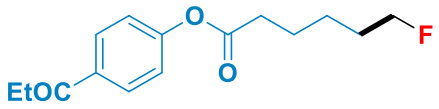
7-fluoroheptyl 4-(trifluoromethyl)benzoate (57b)



Following the general procedure condition **C**. Colorless oil. (24.5 mg, 40%)。R_f= 0.65

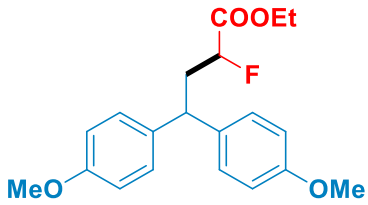
(Eluent: PE/EA 20:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.15 (d, *J* = 8.0 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 2H), 4.44 (dt, *J* = 36.6 Hz, *J* = 6.1 Hz, 2H), 4.35 (t, *J* = 3.1 Hz, 2H), 1.81 – 1.65 (m, 4H), 1.48 – 1.41 (m, 6H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 165.6, 134.5 (q, *J* = 32.6 Hz), 133.8, 130.1, 125.5 (q, *J* = 4.0 Hz), 123.8 (q, *J* = 272.9 Hz), 84.2 (d, *J* = 164.4 Hz), 65.7, 30.5 (d, *J* = 19.7 Hz), 29.0, 28.7, 26.1, 25.3 (d, *J* = 5.5 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -63.1, -218.3. **HRMS (ESI)** m/z calcd for $C_{15}H_{18}F_4O_2^+$ $(M+Na)^+$ 329.1141, found m/z 329.1142.

4-propionylphenyl 6-fluorohexanoate (58b)



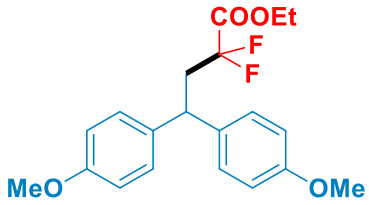
Following the general procedure condition **C**. Colorless oil. (26.6 mg, 50%)。 R_f= 0.50 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 8.06 (d, *J* = 8.8 Hz, 2H), 7.15 (d, *J* = 8.7 Hz, 2H), 4.46 (dt, *J* = 47.3, 6.0 Hz, 2H), 3.90 (s, 3H), 2.59 (t, *J* = 7.5 Hz, 2H), 1.82 – 1.70 (m, 4H), 1.57 – 1.50 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 171.5, 166.4, 154.4, 131.2, 127.8, 121.7, 83.8 (d, *J* = 164.8 Hz), 52.3, 34.3, 30.1 (d, *J* = 19.9 Hz), 24.9 (d, *J* = 5.2 Hz), 24.5. **^{19}F NMR (376 MHz, Chloroform-d):** δ -218.6. **HRMS (ESI)** m/z calcd for $C_{15}H_{19}FO_3^+$ $(M+Na)^+$ 289.1216, found m/z 289.1218.

ethyl 2-fluoro-4,4-bis(4-methoxyphenyl)butanoate (59b)



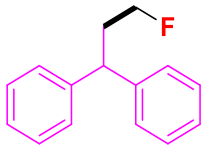
Following the general procedure condition **E**. Colorless oil. (46.4 mg, 67%)。 R_f= 0.20 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.18 (dd, *J* = 11.3, 8.6 Hz, 4H), 6.85 (dd, *J* = 15.0, 8.7 Hz, 4H), 4.72 (ddd, *J* = 49.1, 9.3, 3.9 Hz, 1H), 4.25 – 4.12 (m, 3H), 3.77 (d, *J* = 5.8 Hz, 6H), 2.66 – 2.46 (m, 2H), 1.29 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 170.2 (d, *J* = 23.2 Hz), 158.3 (d, *J* = 20.4 Hz), 135.6 (d, *J* = 143.6 Hz), 128.8 (d, *J* = 51.1 Hz), 114.1 (d, *J* = 27.5 Hz), 87.3 (d, *J* = 184.4 Hz), 61.6, 55.3, 44.6 (d, *J* = 2.8 Hz), 38.7 (d, *J* = 20.9 Hz), 14.2. **^{19}F NMR (376 MHz, Chloroform-d):** δ -193.6. **HRMS (ESI)** m/z calcd for $C_{20}H_{23}FO_4^+$ $(M+Na)^+$ 369.1478, found m/z 369.1479.

ethyl 2,2-difluoro-4,4-bis(4-methoxyphenyl)butanoate (60b)



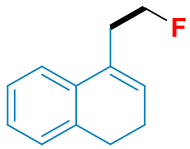
Following the general procedure condition **E**. Colorless oil. (68.5 mg, 94%)。R_f= 0.20 (Eluent: PE/EA 10:1). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.15 (d, J = 8.7 Hz, 4H), 6.82 (d, J = 8.7 Hz, 4H), 4.20 (t, J = 7.4 Hz, 1H), 3.88 (q, J = 7.2 Hz, 2H), 3.76 (s, 6H), 2.88 (td, J = 15.4, 7.4 Hz, 2H), 1.19 (t, J = 7.2 Hz, 3H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 163.9 (t, J = 32.5 Hz), 158.4, 135.5, 128.7, 114.0, 62.8, 55.3, 43.3 (t, J = 4.9 Hz), 40.7 (t, J = 23.1 Hz), 29.8, 13.8. **^{19}F NMR (376 MHz, Chloroform-d):** δ -103.4. **HRMS (ESI)** m/z calcd for $C_{20}H_{22}F_2O_4^+$ $(M+Na)^+$ 387.1384, found m/z 387.1385.

(3-fluoropropane-1,1-diyl)dibenzene (61b)



Following the general procedure condition **B**. Colorless oil. (68.5 mg, 44%)。R_f= 0.55 (Eluent: PE). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.26 – 7.05 (m, 8H), 7.04 – 6.88 (m, 2H), 4.21 (dt, J = 47.1, 6.0 Hz, 2H), 3.99 (t, J = 7.9 Hz, 1H), 2.26 (ddt, J = 24.0, 8.0, 6.0 Hz, 2H). **^{13}C NMR (101 MHz, Chloroform-d):** δ 144.0, 128.7, 128.0, 126.6, 82.2 (d, J = 164.6 Hz), 46.6 (d, J = 5.2 Hz), 36.2 (d, J = 19.8 Hz). **^{19}F NMR (376 MHz, Chloroform-d):** δ -220.8. **HRMS (ESI)** m/z calcd for $C_{15}H_{15}F^+$ $(M+Na)^+$ 237.1056, found m/z 237.1057.

4-(2-fluoroethyl)-1,2-dihydronaphthalene (62b)

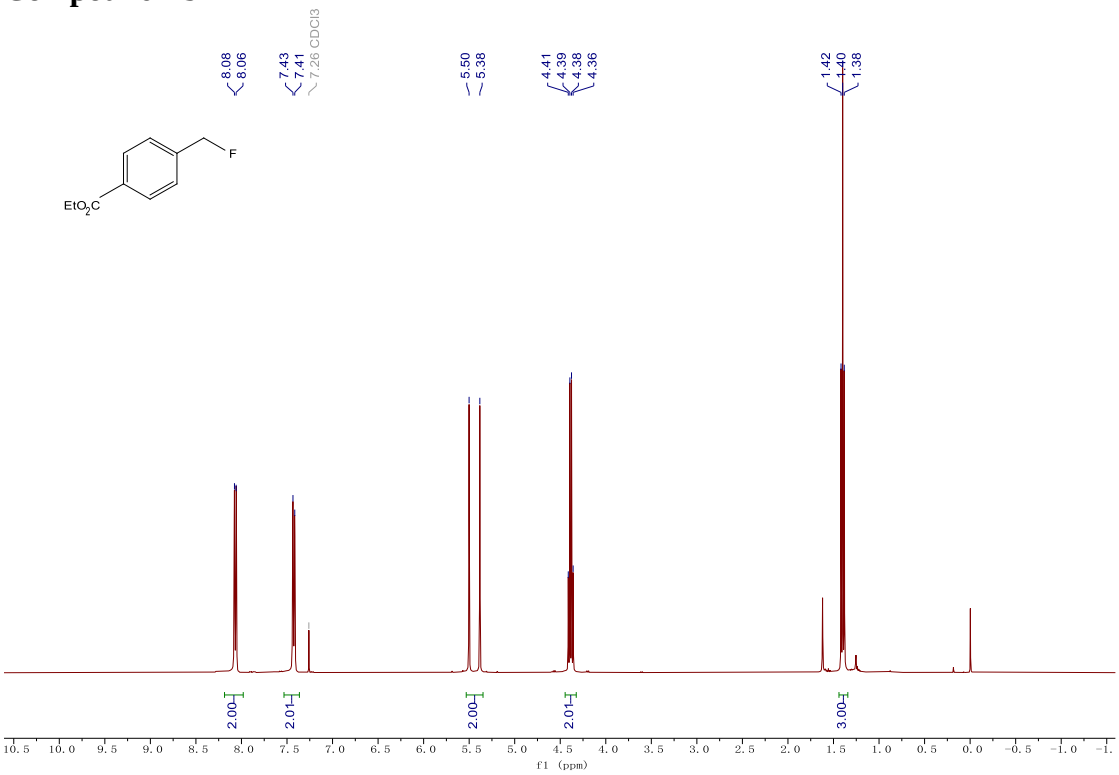


Following the general procedure condition **B**. Colorless oil. (12,0 mg, 34%)。R_f= 0.55 (Eluent: PE). **^{1}H NMR (400 MHz, Chloroform-d):** δ 7.15 (d, J = 8.7 Hz, 4H), 6.82 (d, J = 8.7 Hz, 4H), 4.20 (t, J = 7.4 Hz, 1H), 3.88 (q, J = 7.2 Hz, 2H), 3.76 (s, 6H), 2.88 (td, J = 15.4, 7.4 Hz, 2H), 1.19 (t, J = 7.2 Hz, 3H). **^{13}C NMR (101 MHz, Chloroform-**

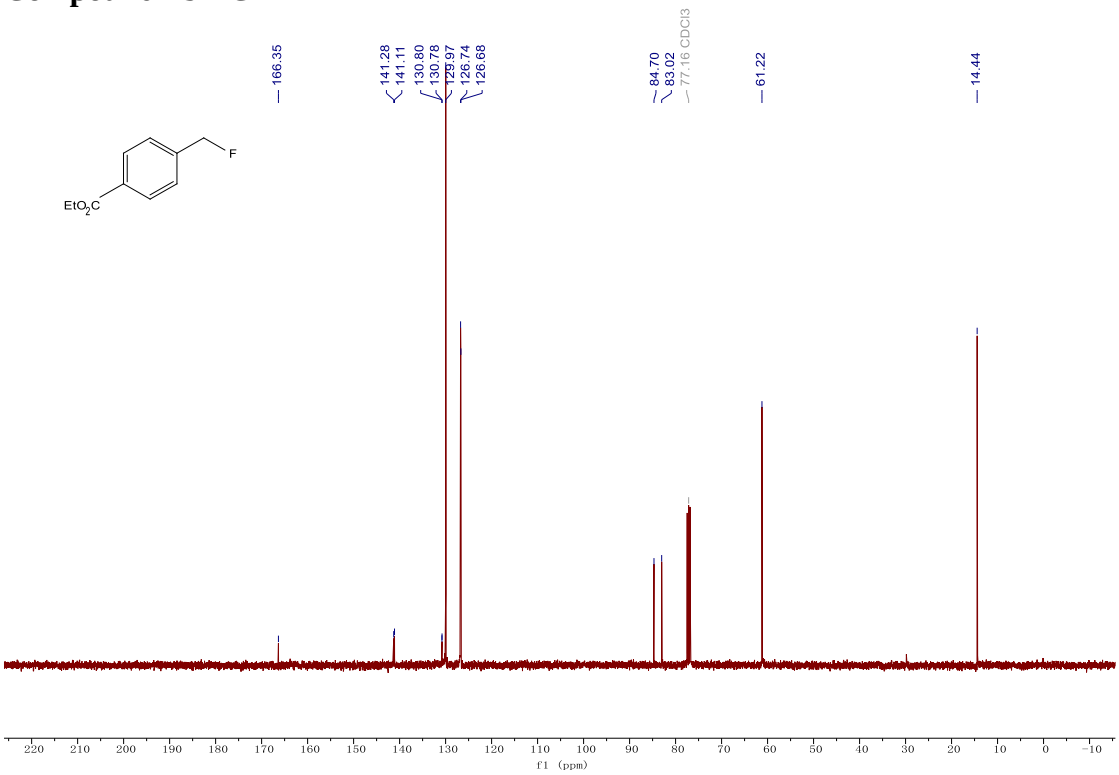
d): δ 163.9 (t, *J* = 32.5 Hz), 158.4, 135.5, 128.7, 114.0, 62.8, 55.3, 43.3 (t, *J* = 4.9 Hz), 40.7 (t, *J* = 23.1 Hz), 29.8, 13.8. **^{19}F NMR (376 MHz, Chloroform-d):** δ -103.4. **HRMS (ESI)** m/z calcd for $C_{12}H_{13}F^{+}$ $(M+Na)^{+}$ 199.0899, found m/z 199.0898.

Ⅶ. NMR Spectra

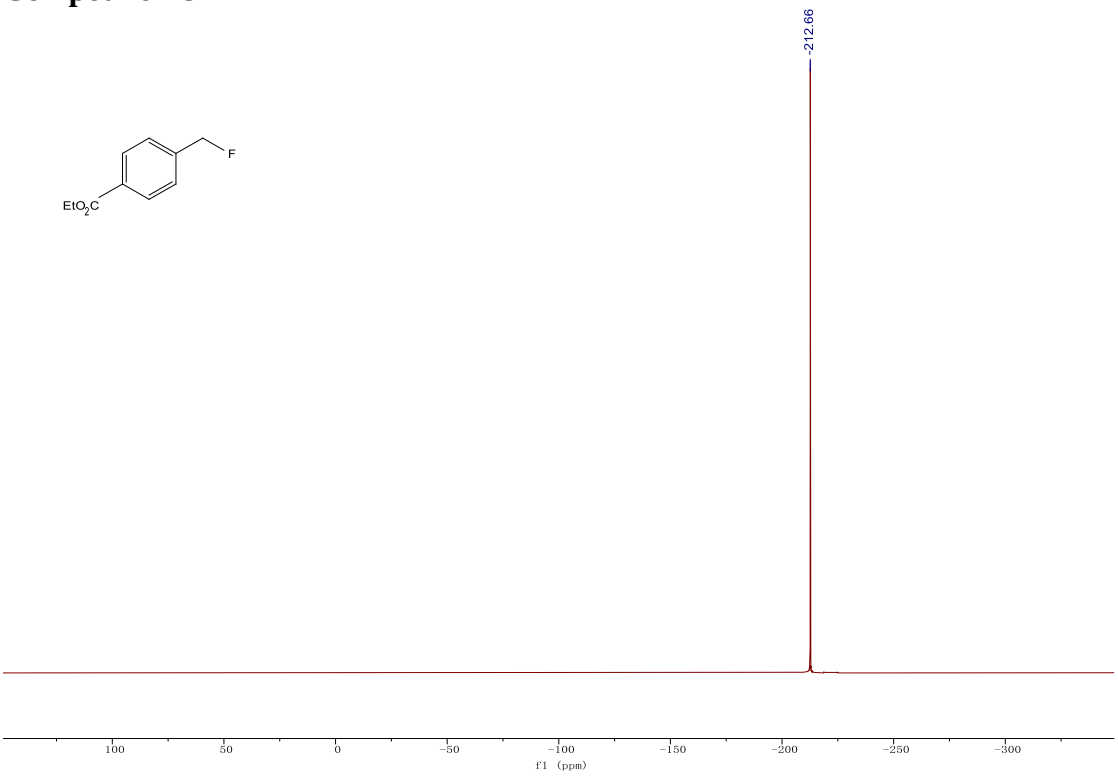
Compound 1b ^{1}H NMR



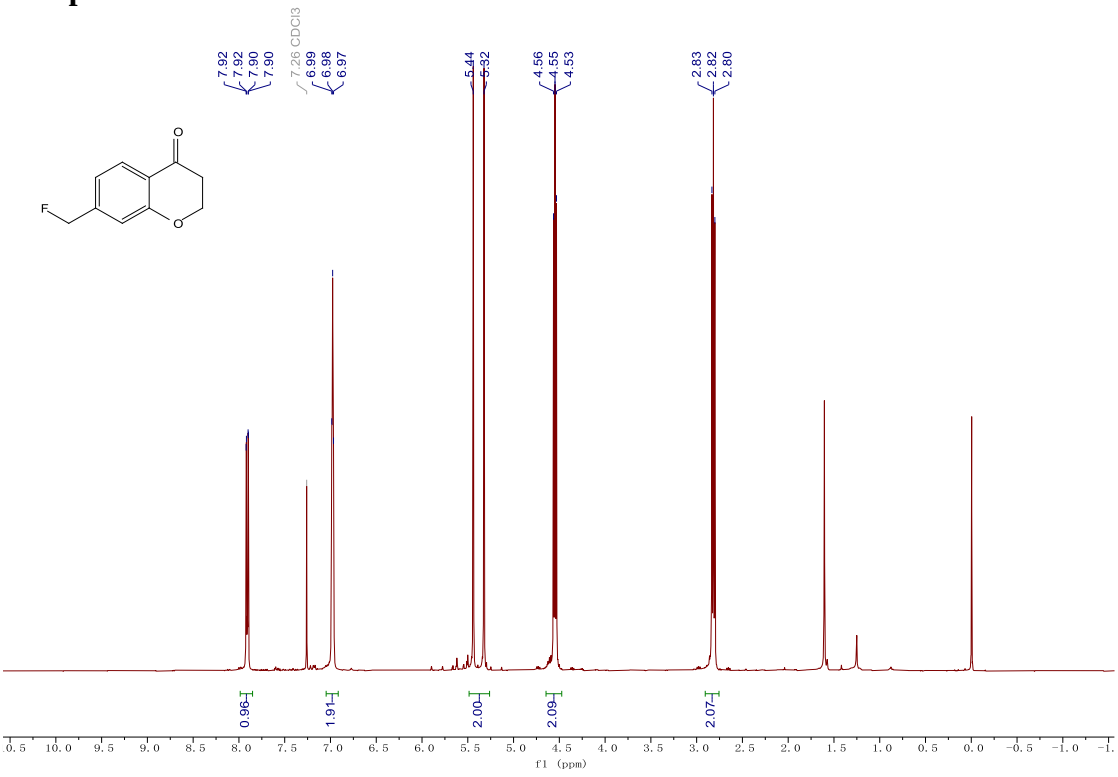
Compound 1b ^{13}C NMR



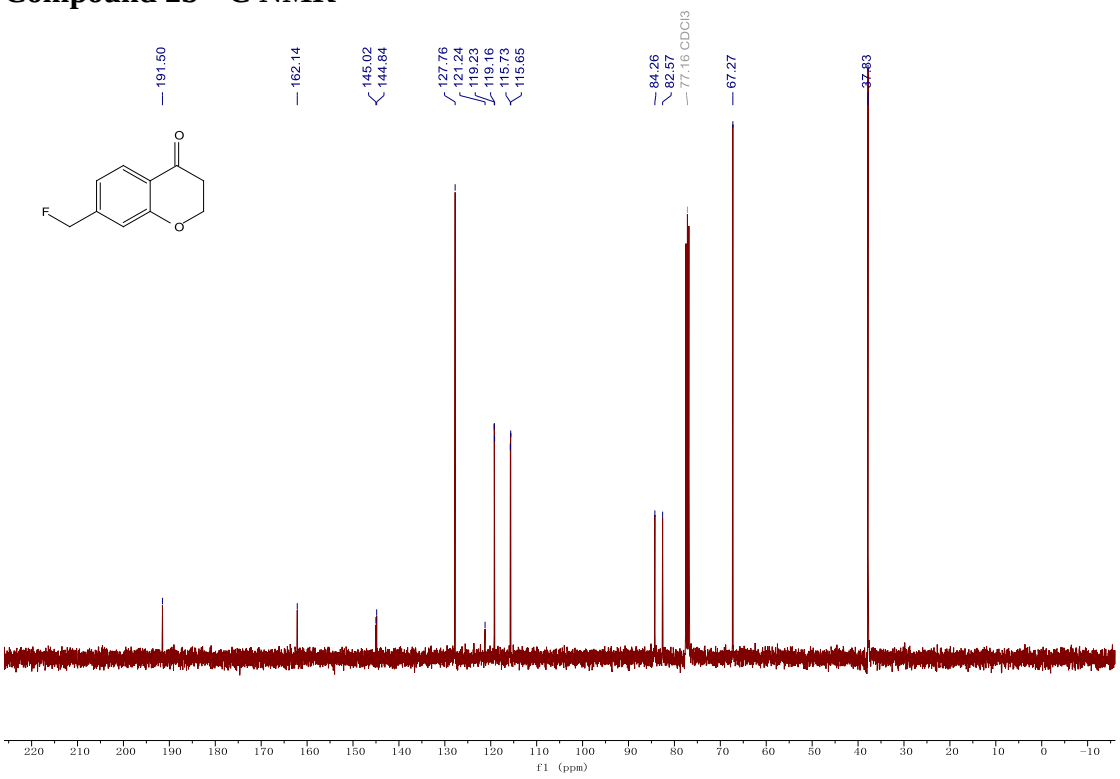
Compound 1b ^{19}F NMR



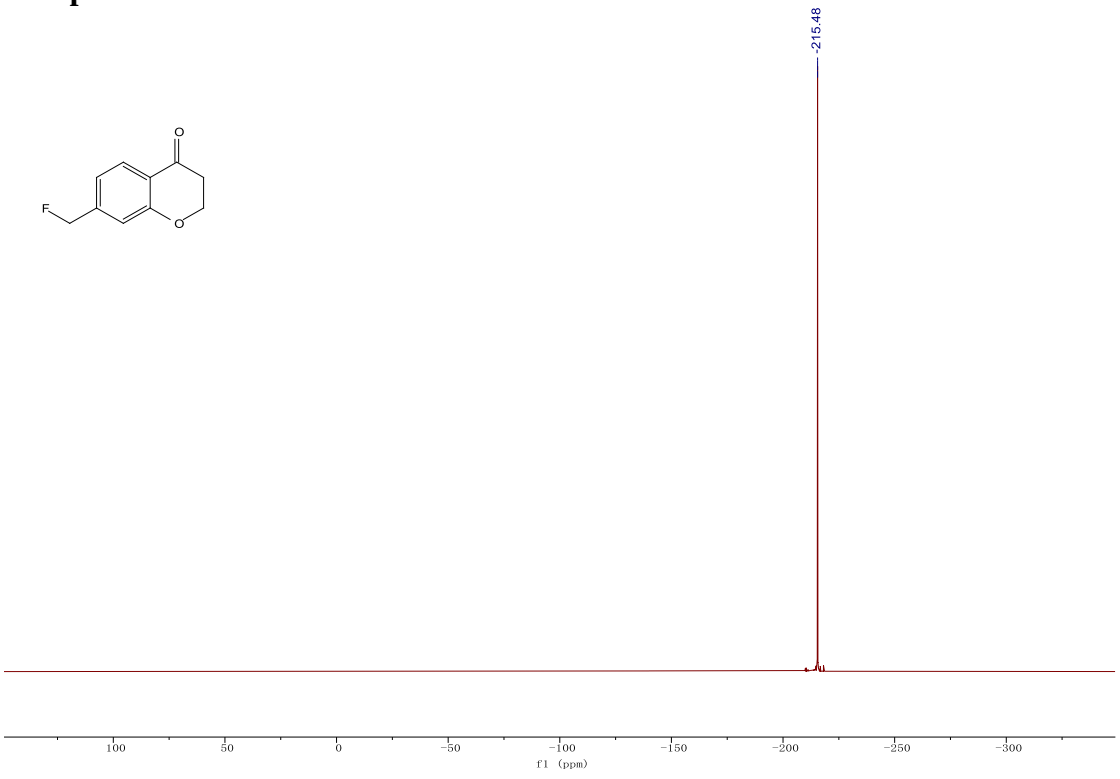
Compound 2b ^{1}H NMR



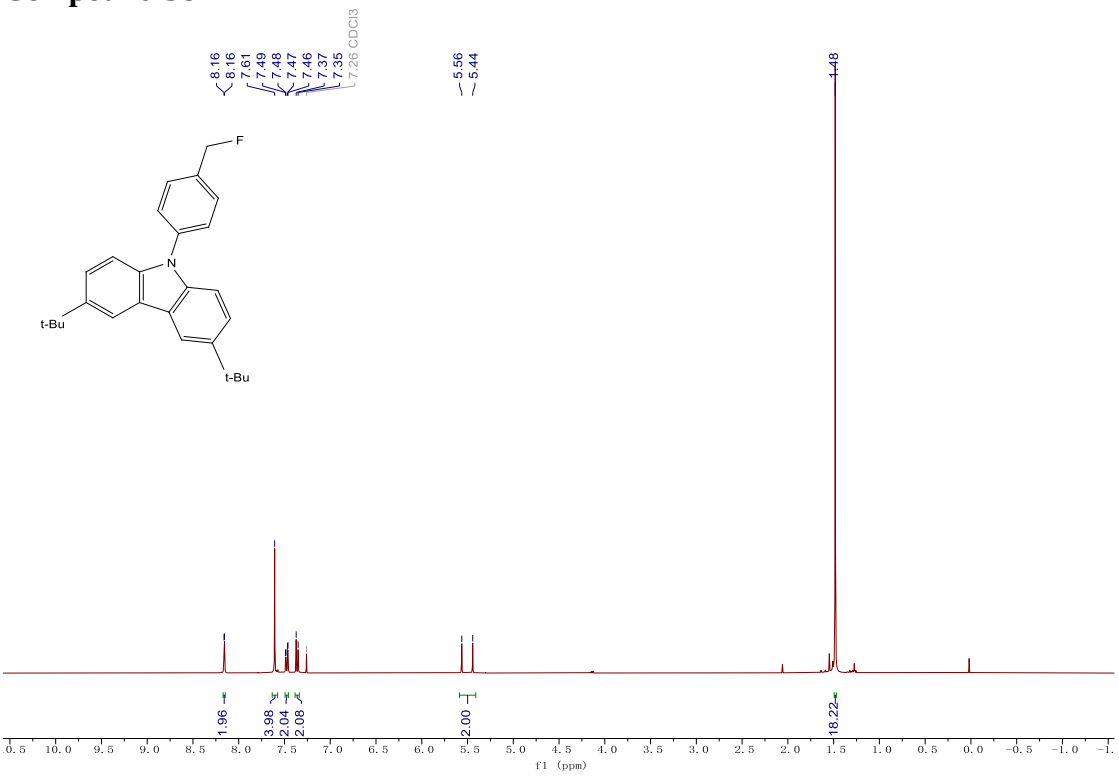
Compound 2b ^{13}C NMR



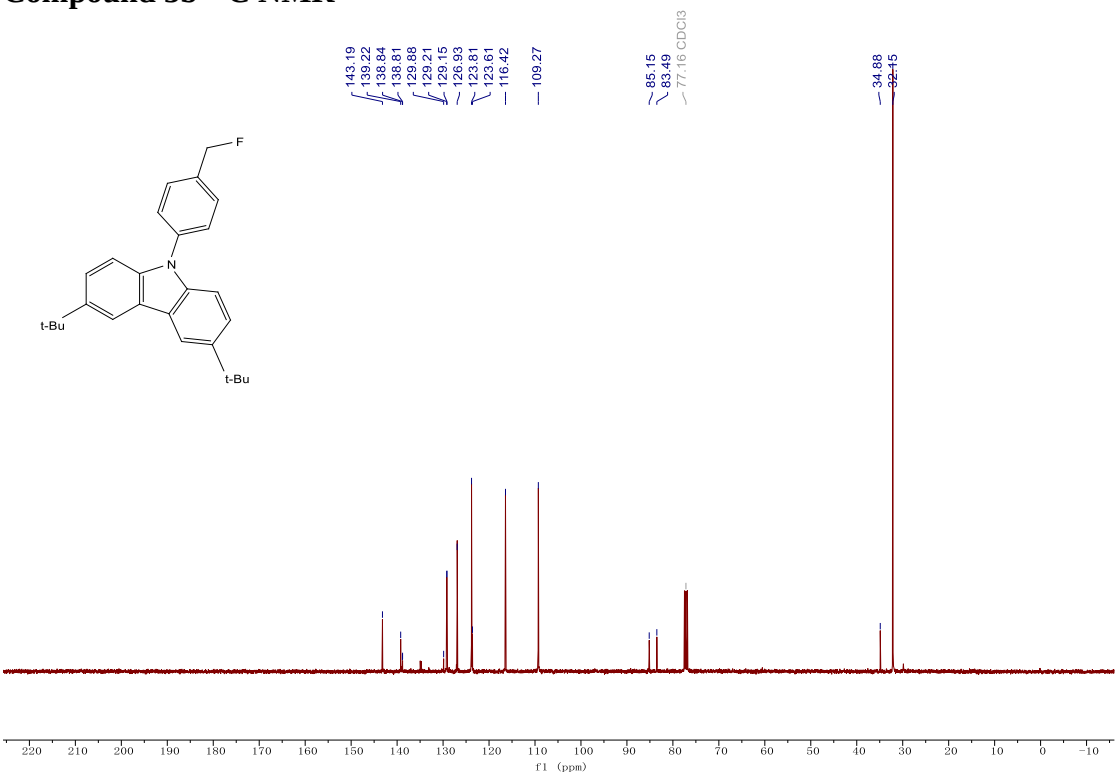
Compound 2b ^{19}F NMR



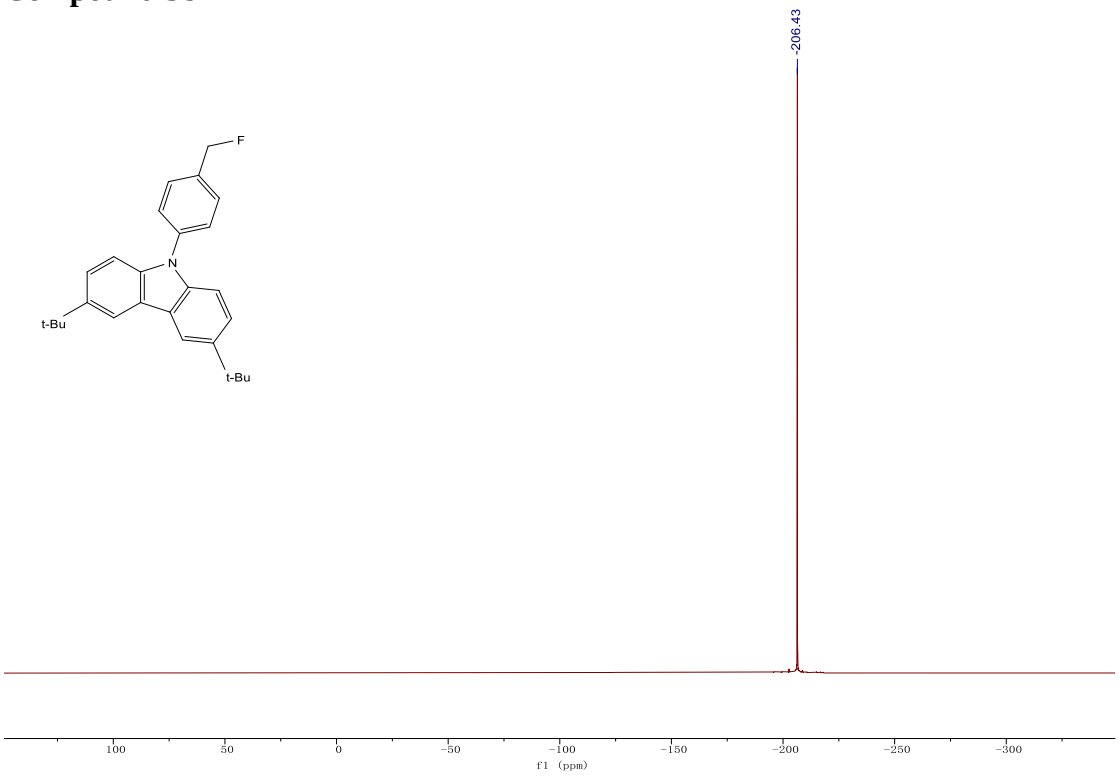
Compound 3b ^{1}H NMR



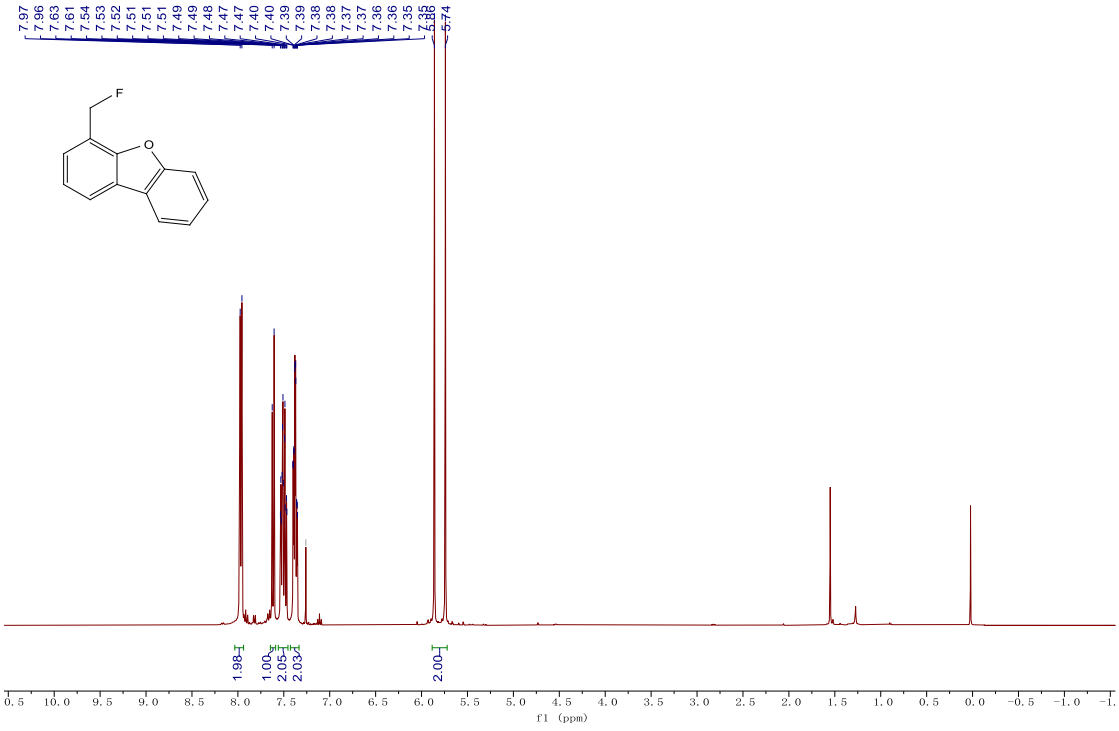
Compound 3b ^{13}C NMR



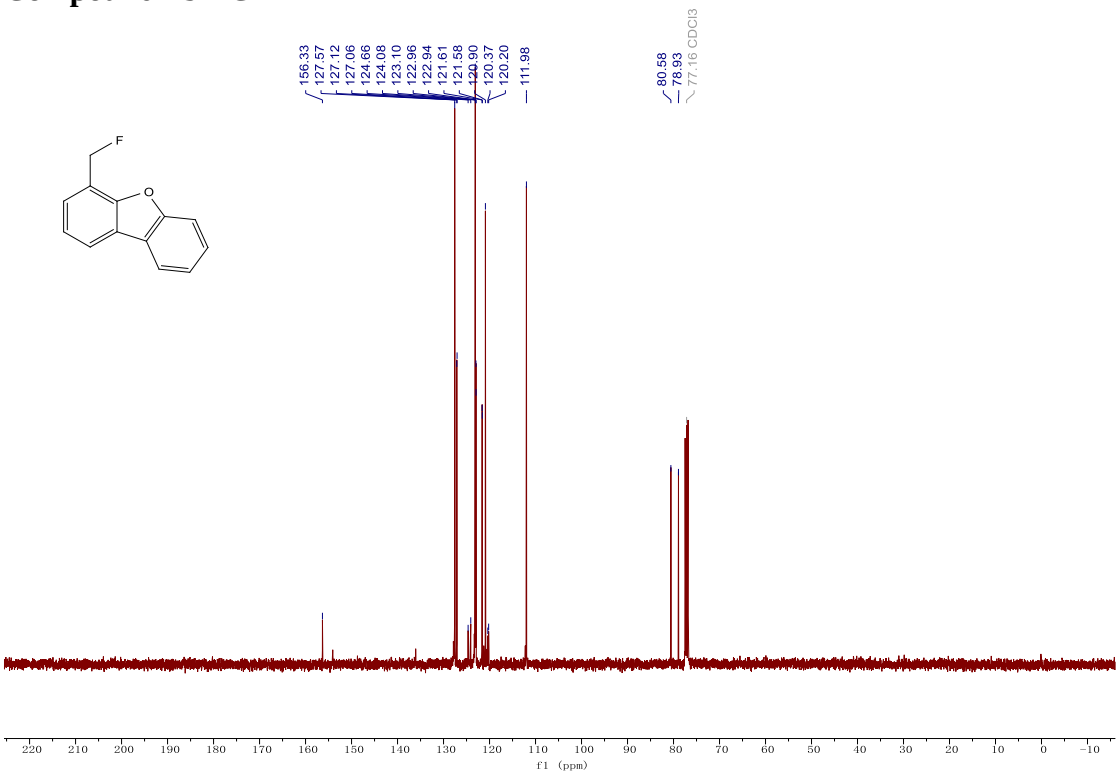
Compound 3b ^{19}F NMR



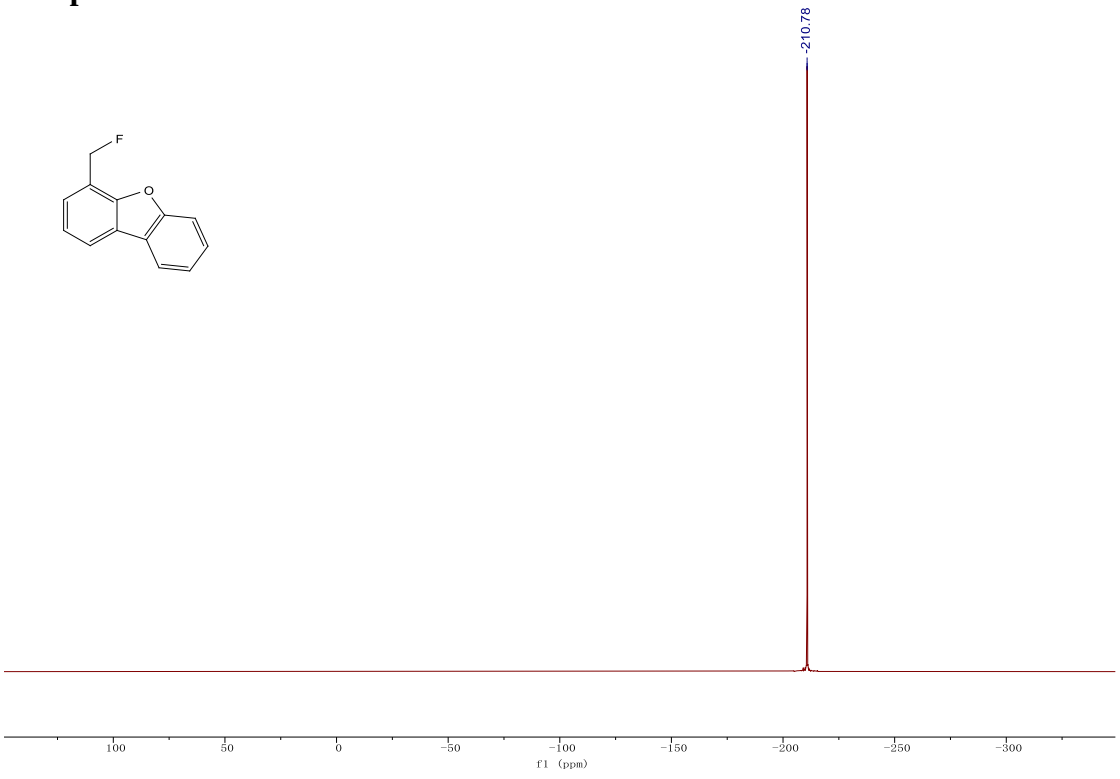
Compound 4b ^{1}H NMR



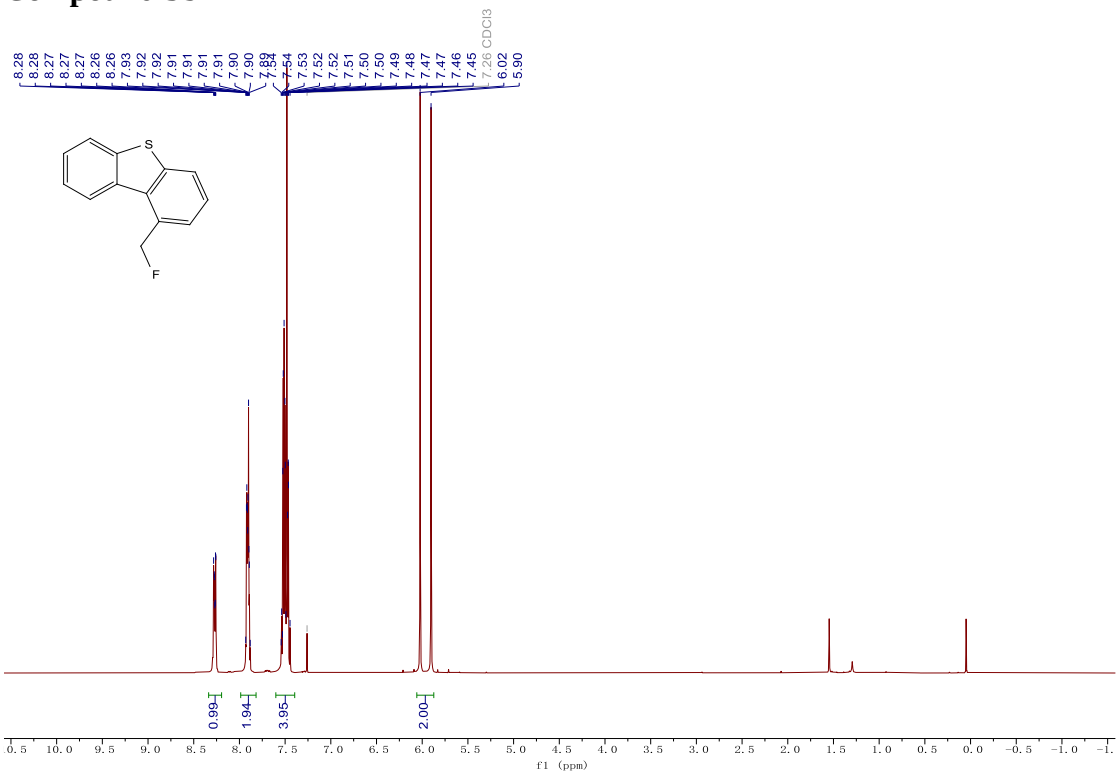
Compound 4b ^{13}C NMR



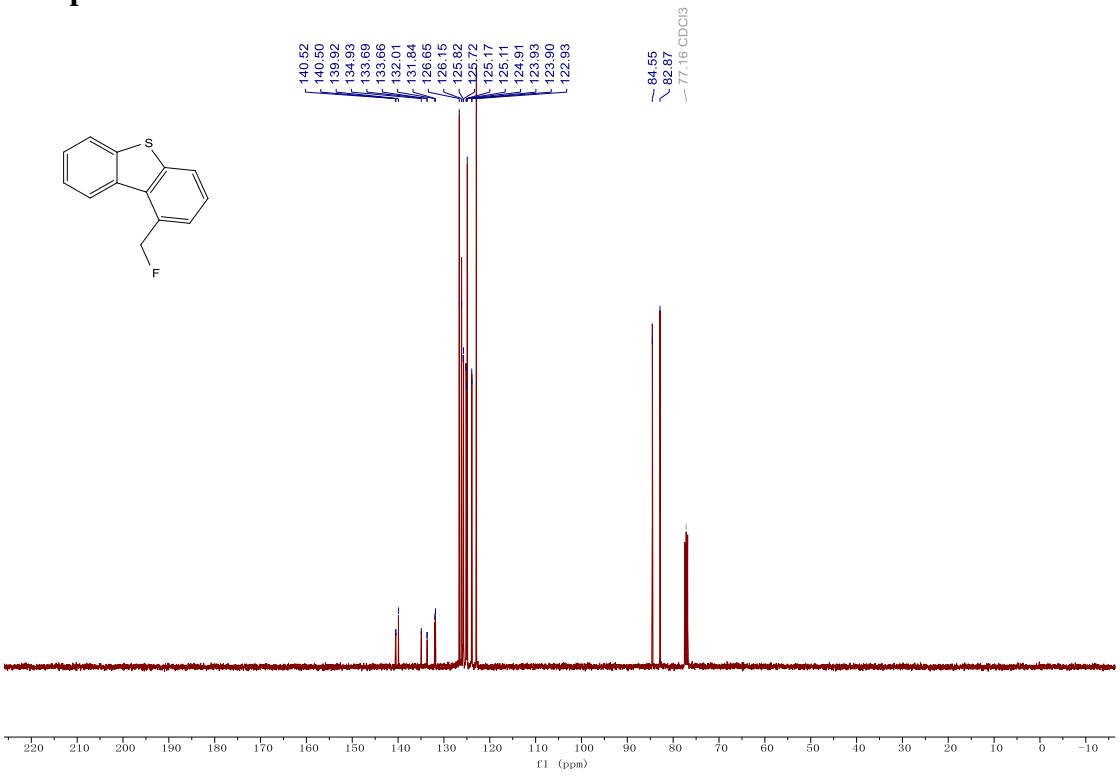
Compound 4b ^{19}F NMR



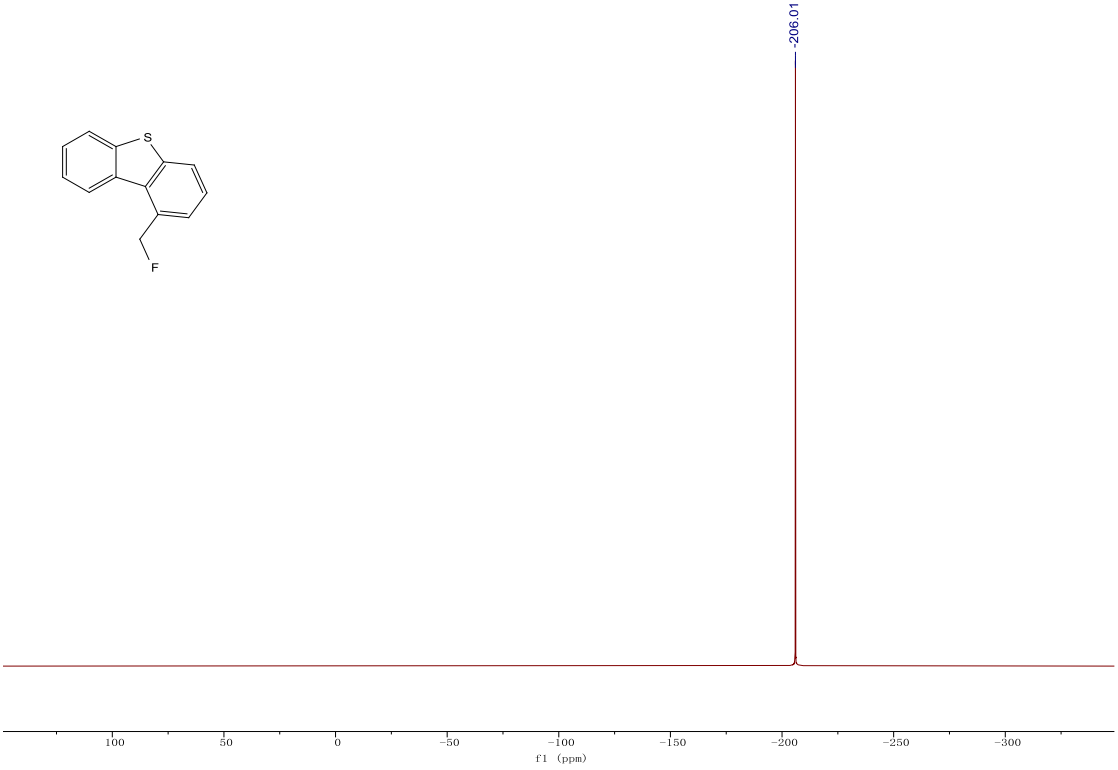
Compound 5b ^{1}H NMR



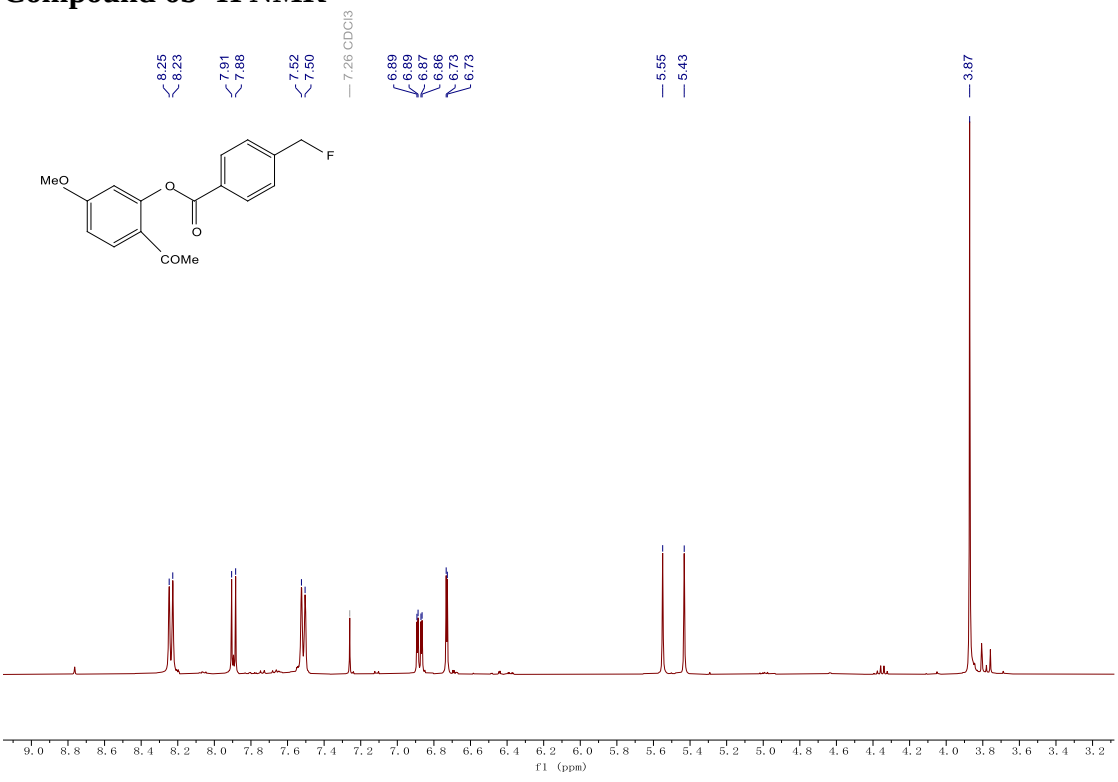
Compound 5b ^{13}C NMR



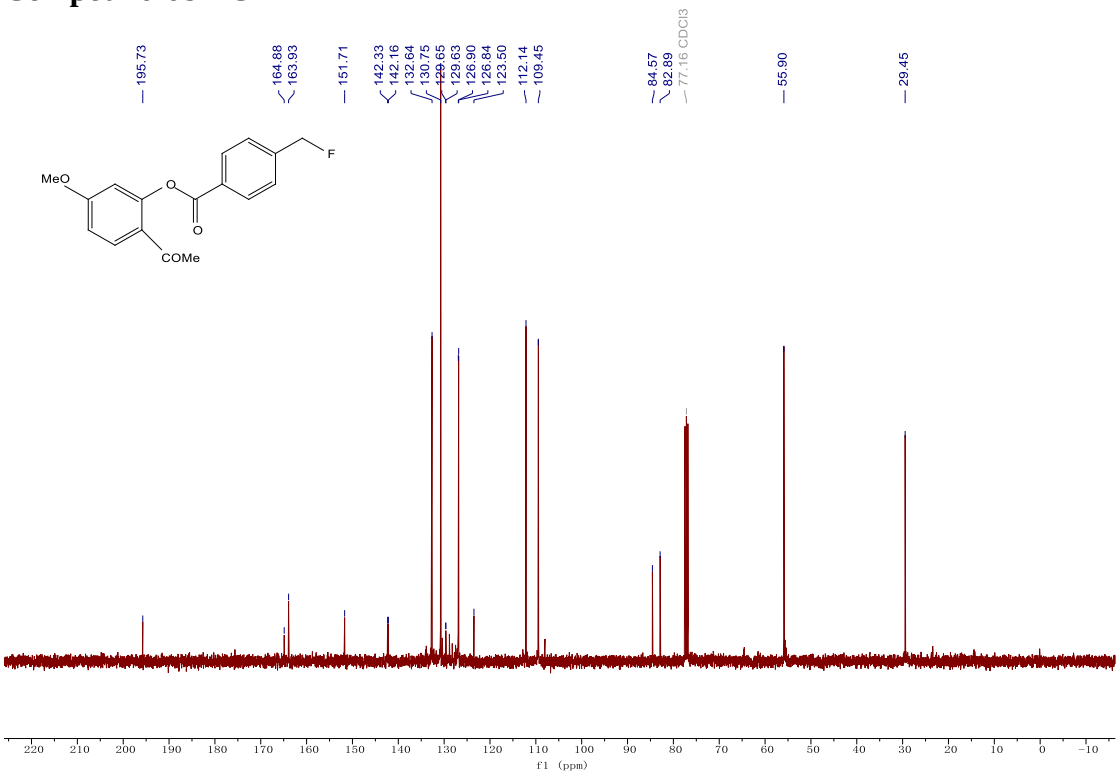
Compound 5b ^{19}F NMR



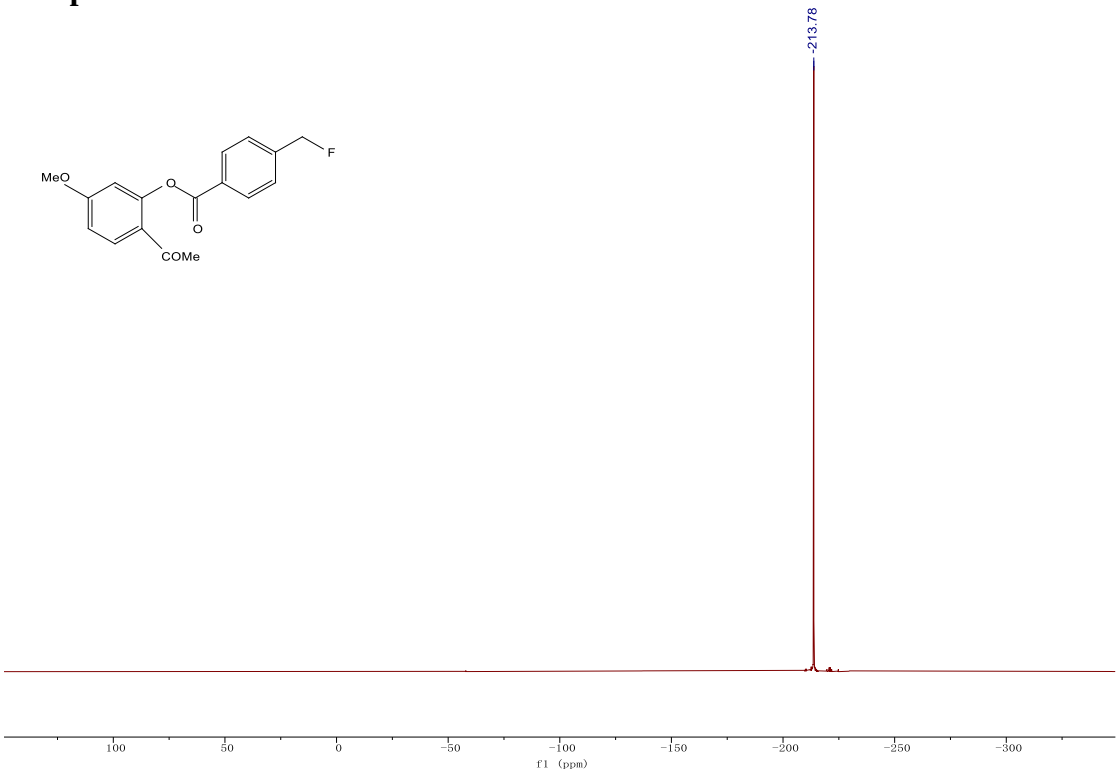
Compound 6b ^{1}H NMR



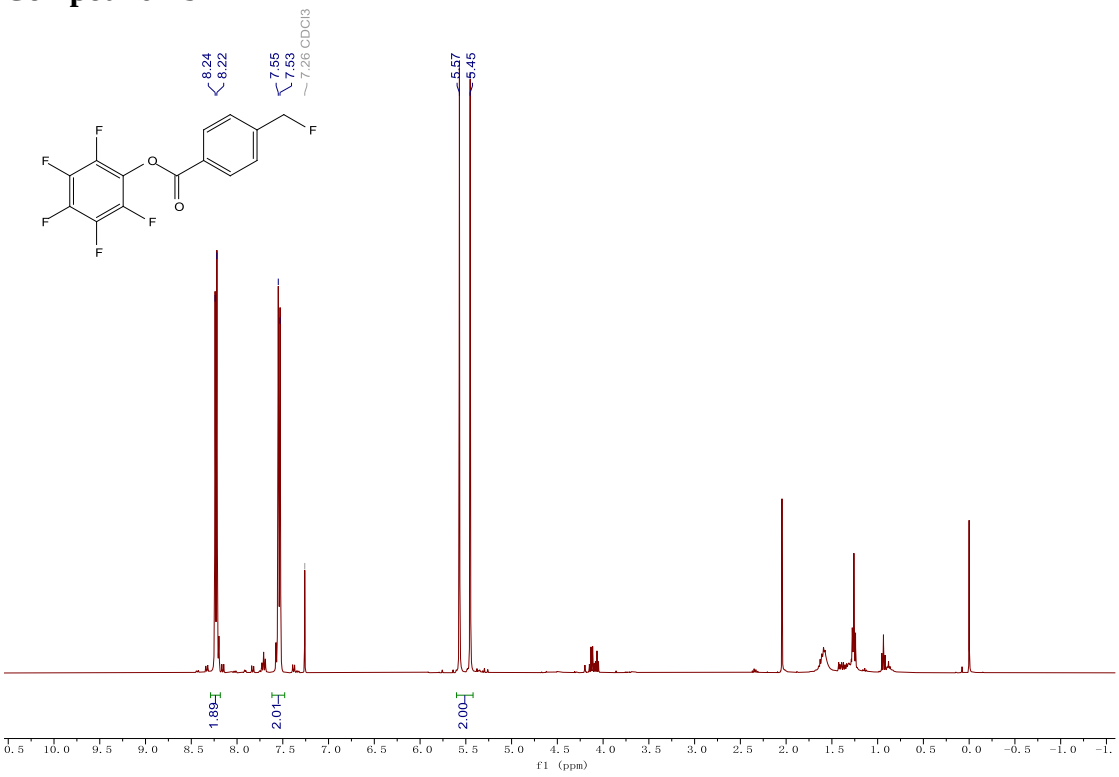
Compound 6b ^{13}C NMR



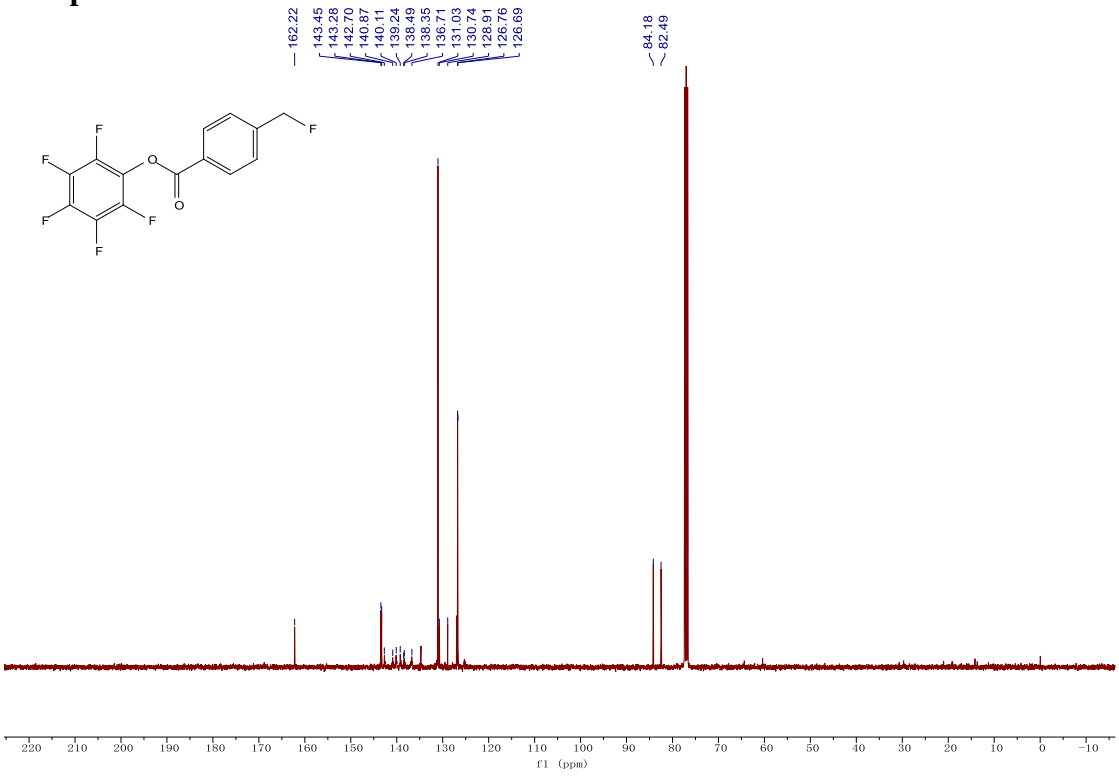
Compound 6b ^{19}F NMR



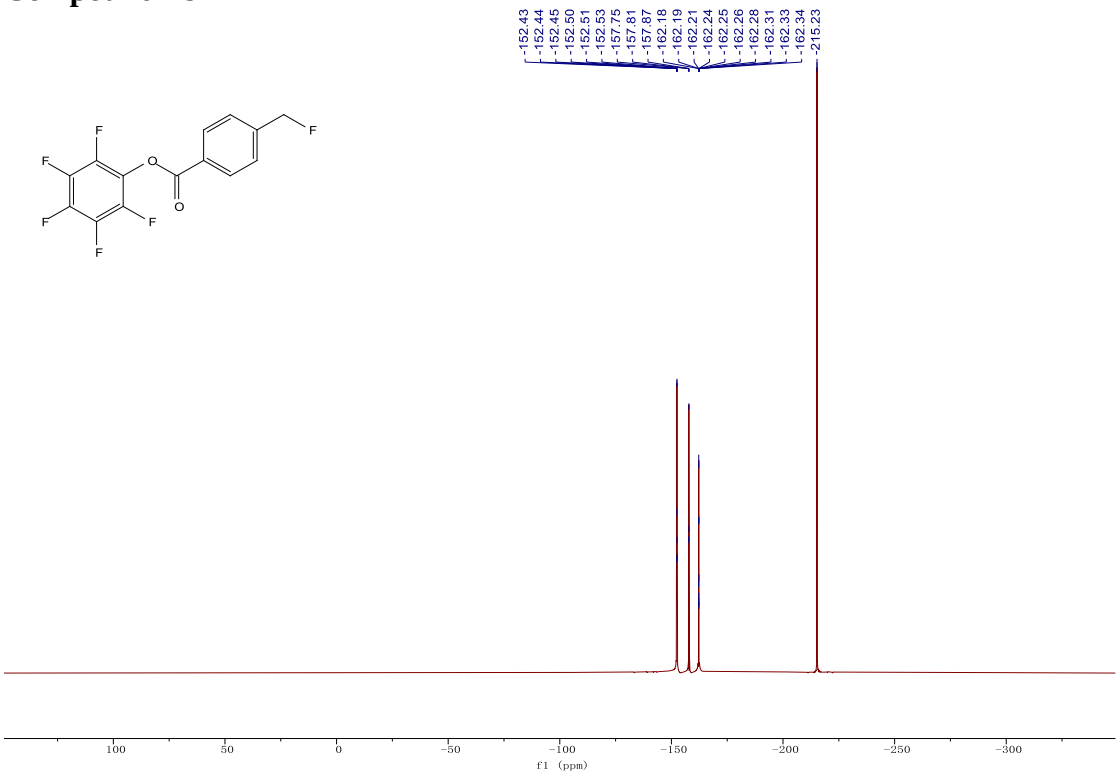
Compound 7b ^{1}H NMR



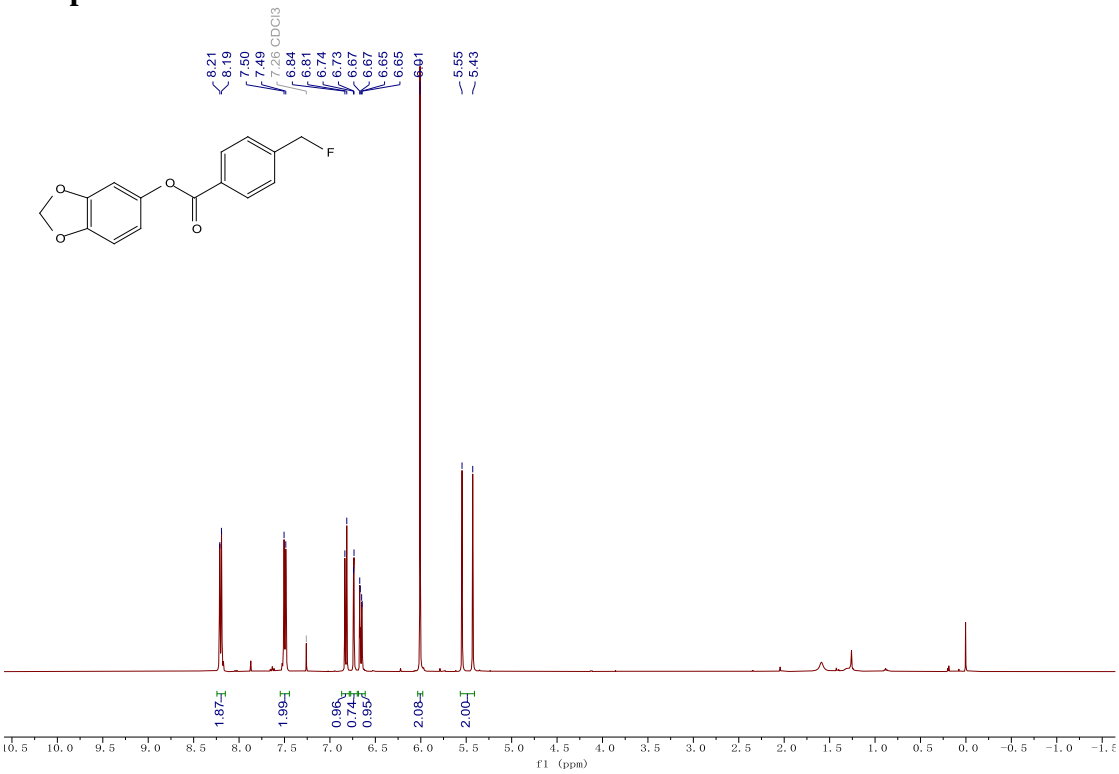
Compound 7b ^{13}C NMR



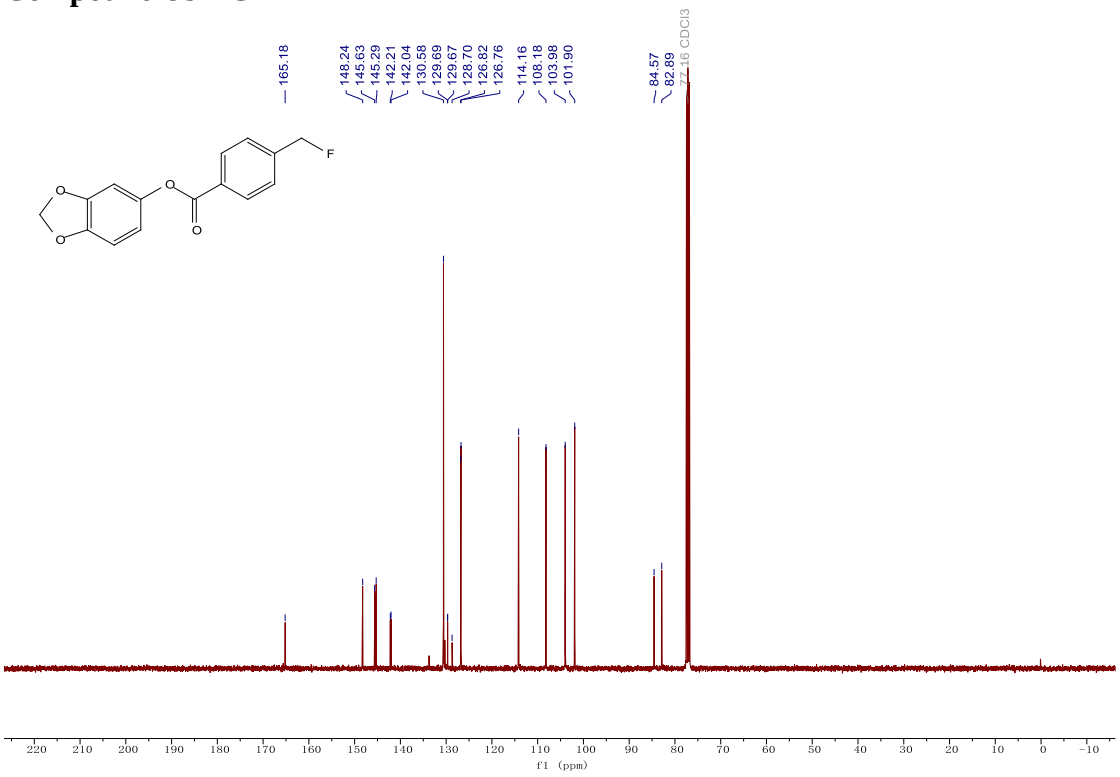
Compound 7b ^{19}F NMR



Compound 8b ^{1}H NMR



Compound 8b ^{13}C NMR



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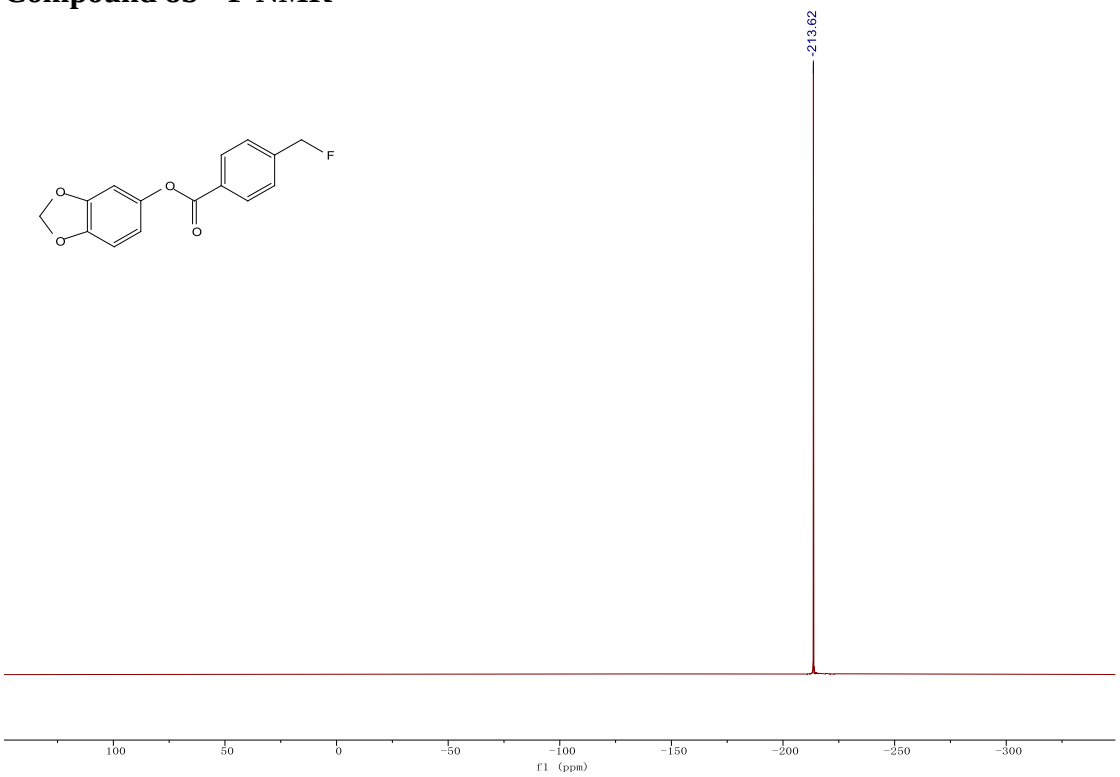
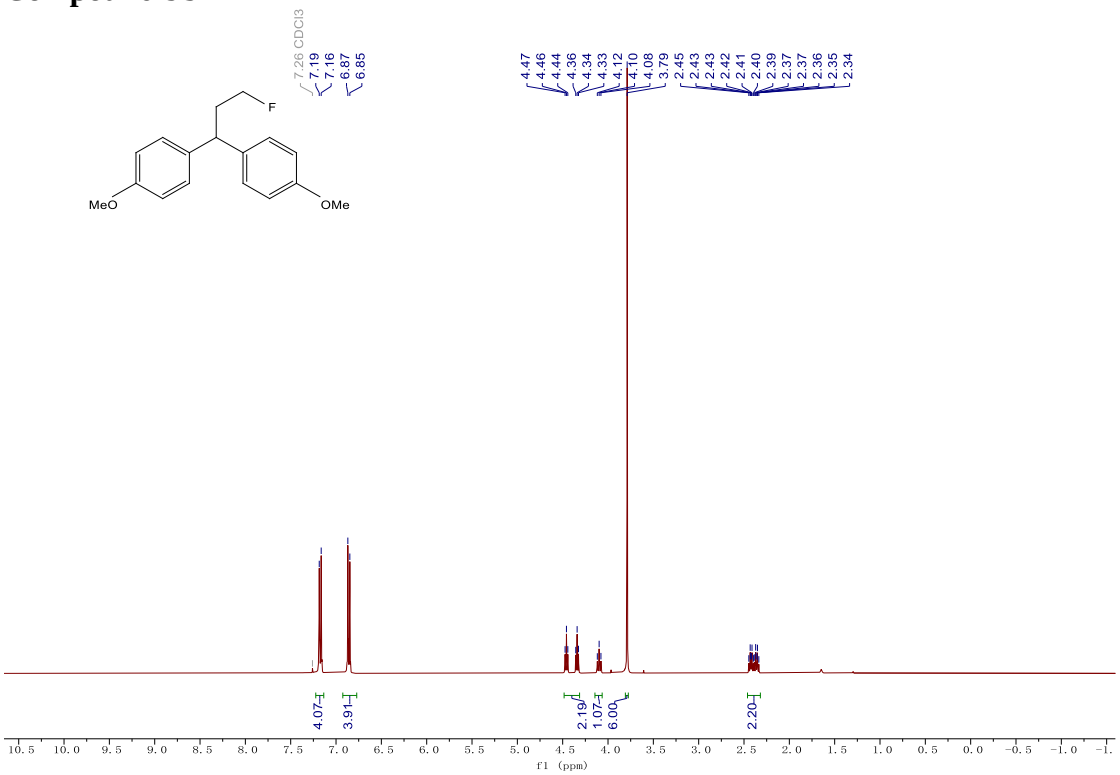
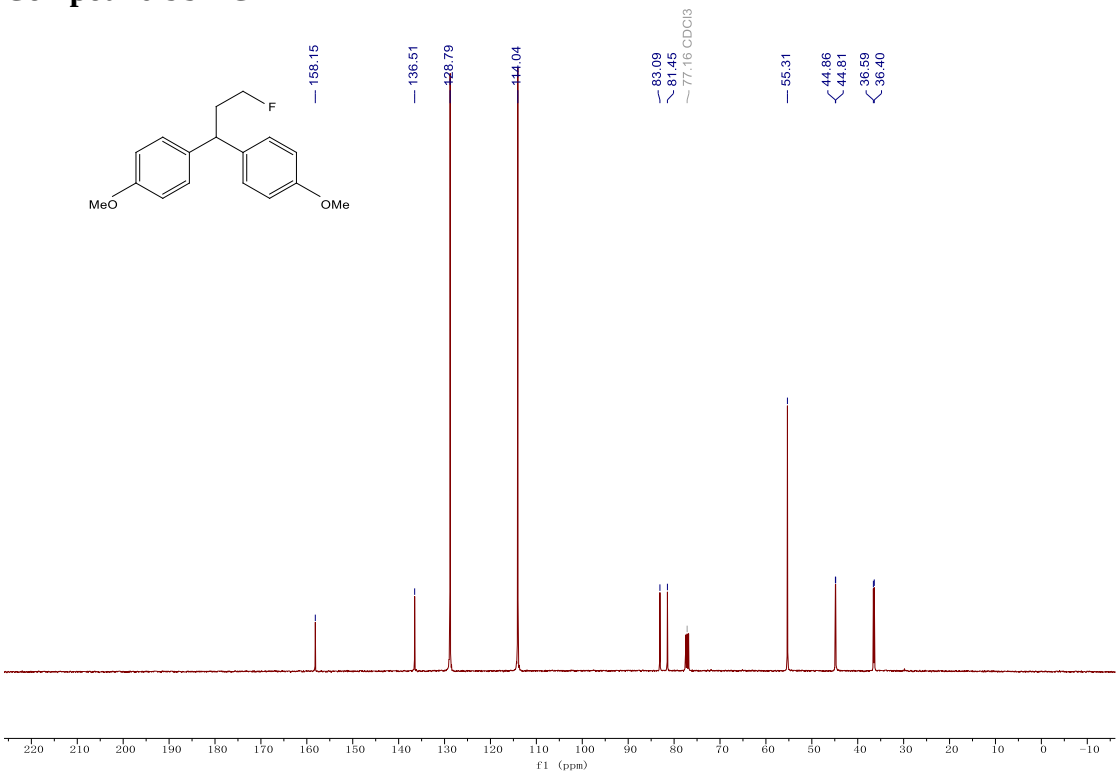


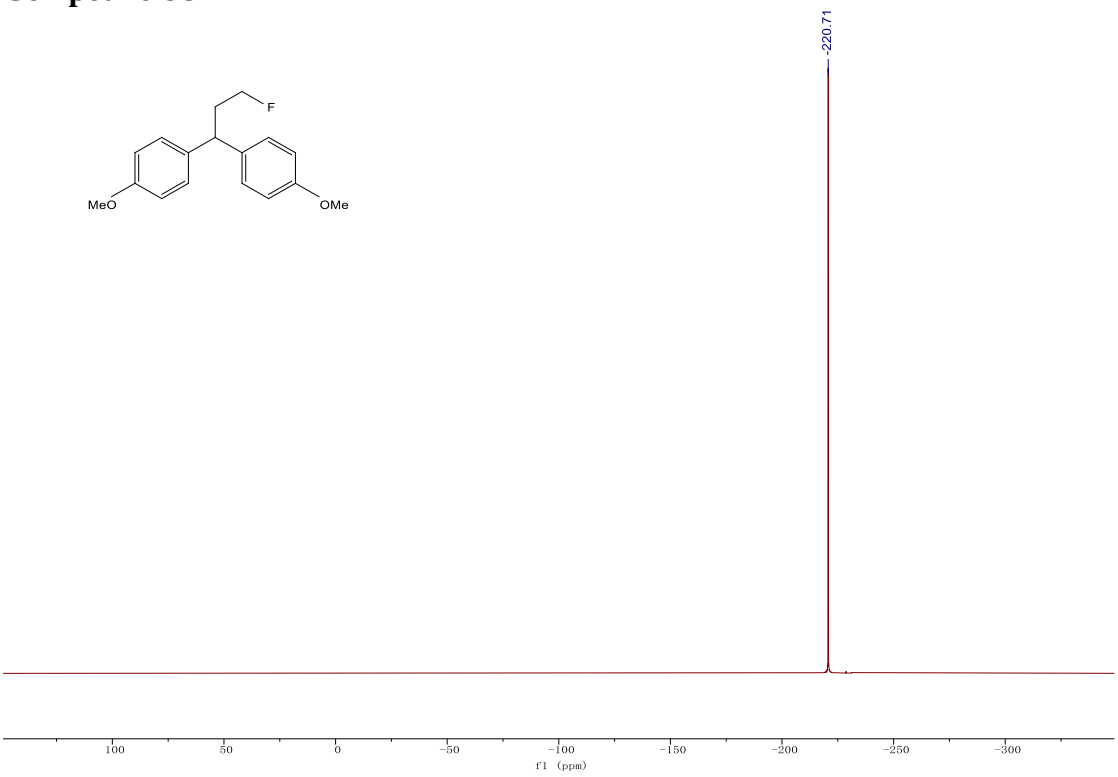
Compound 9b ^{1}H NMR



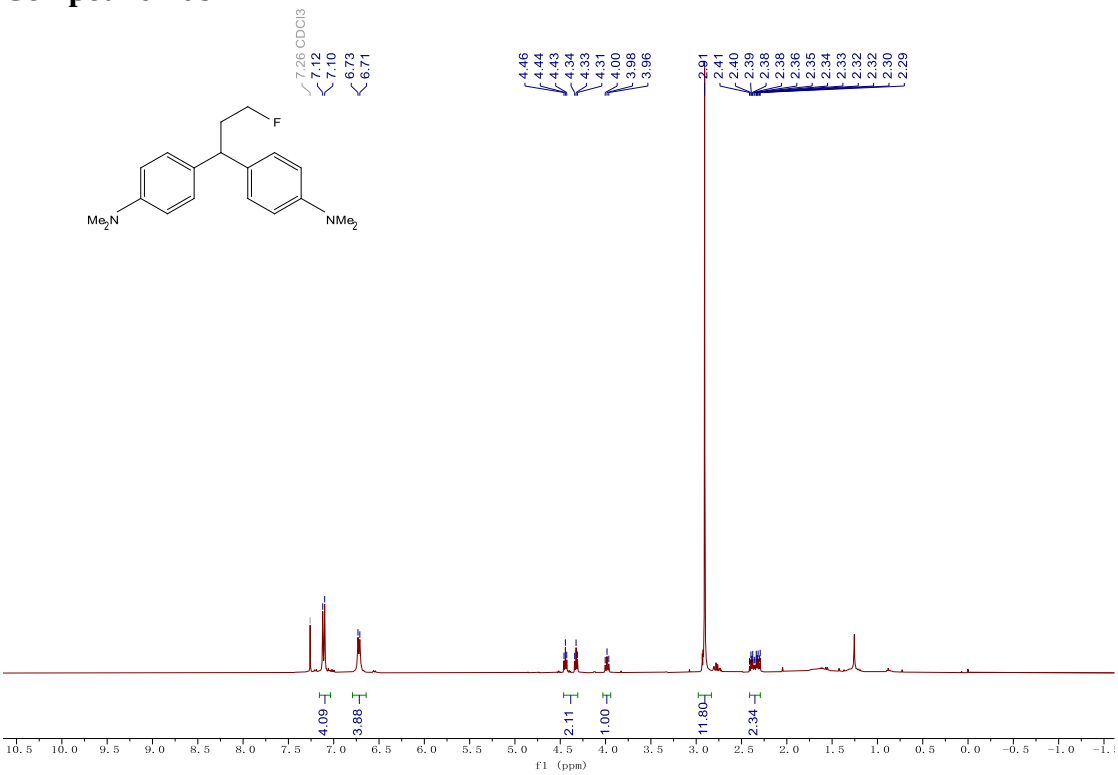
Compound 9b ^{13}C NMR



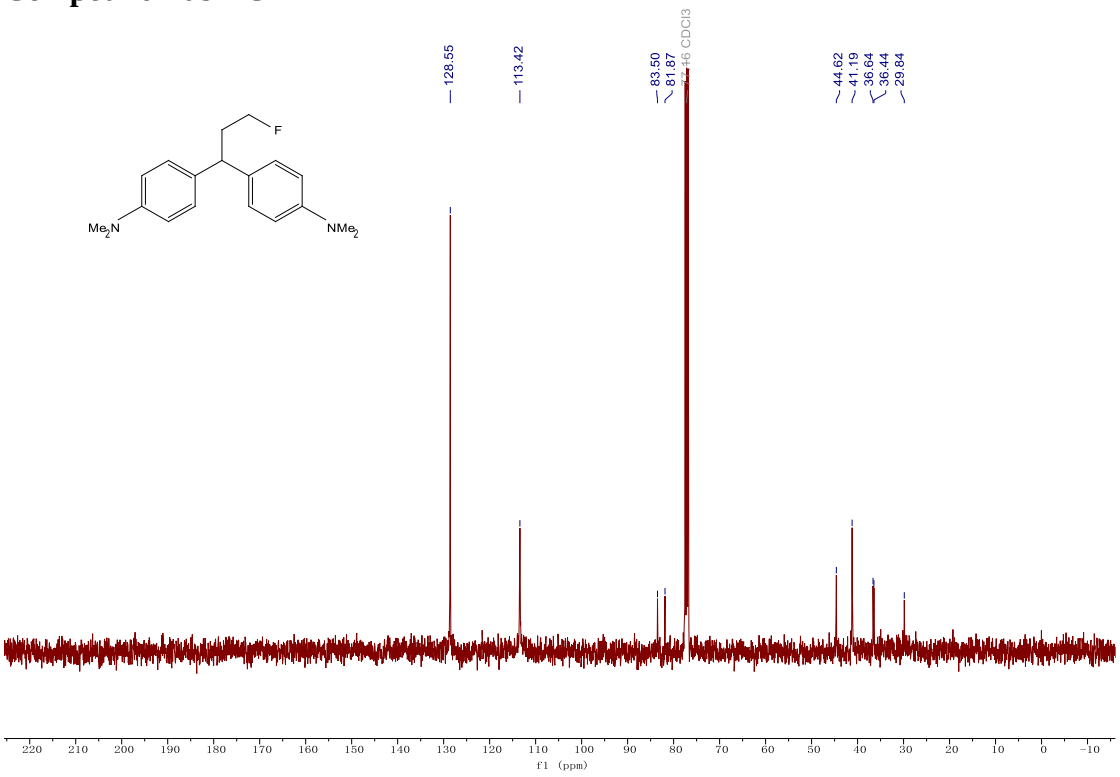
Compound 9b ^{19}F NMR



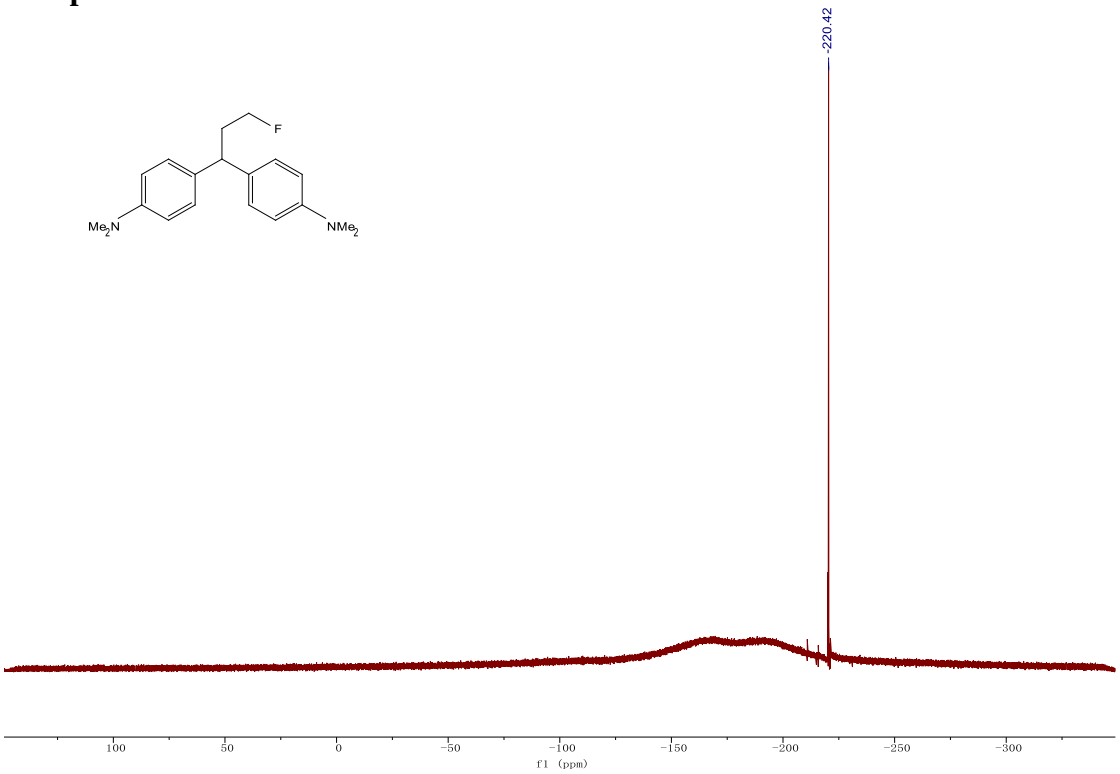
Compound 10b ^{1}H NMR



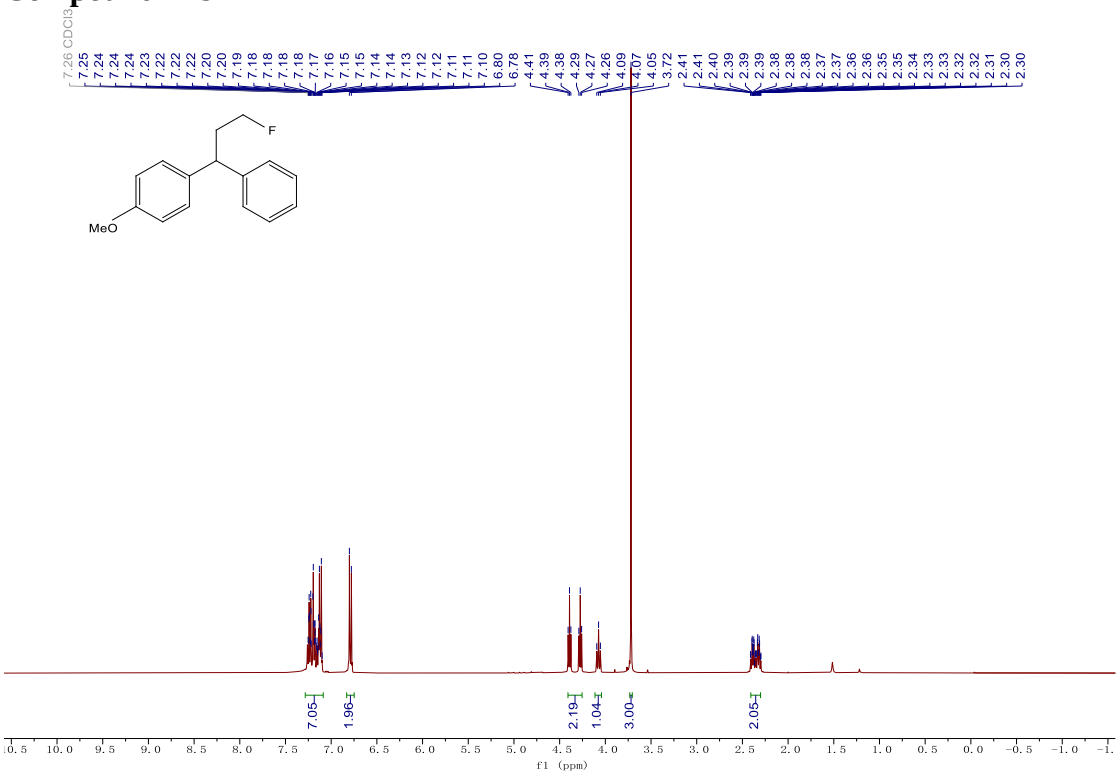
Compound 10b ^{13}C NMR



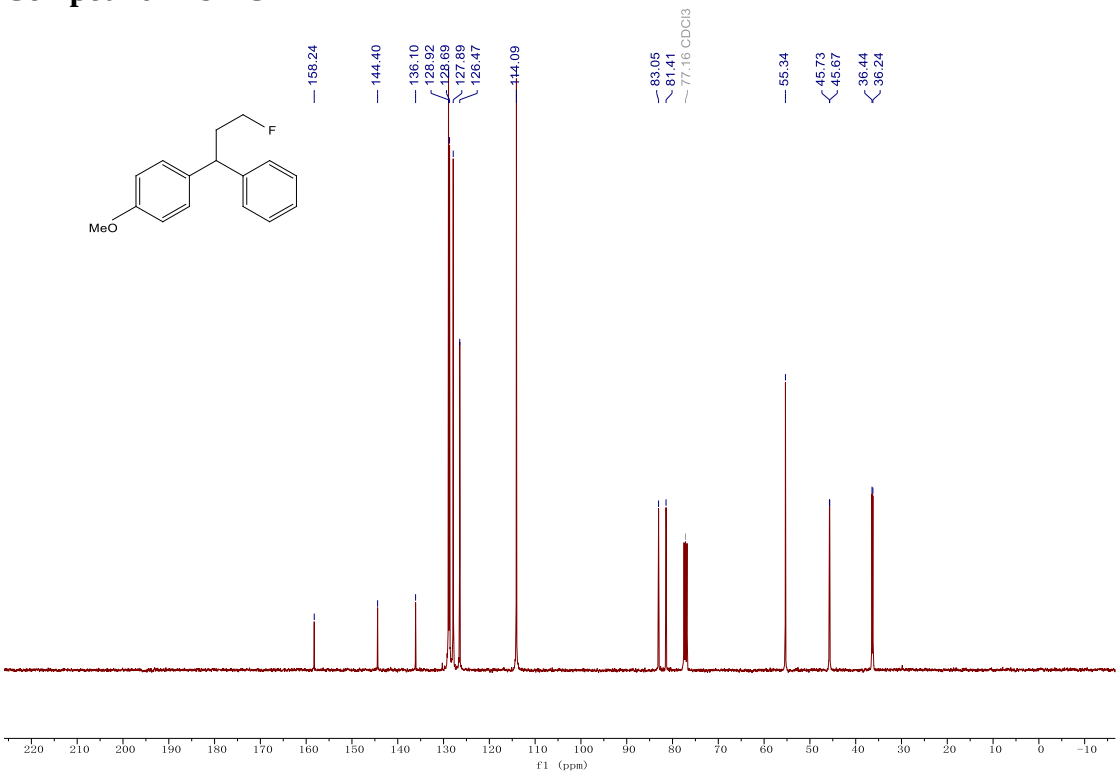
Compound 10b ^{19}F NMR



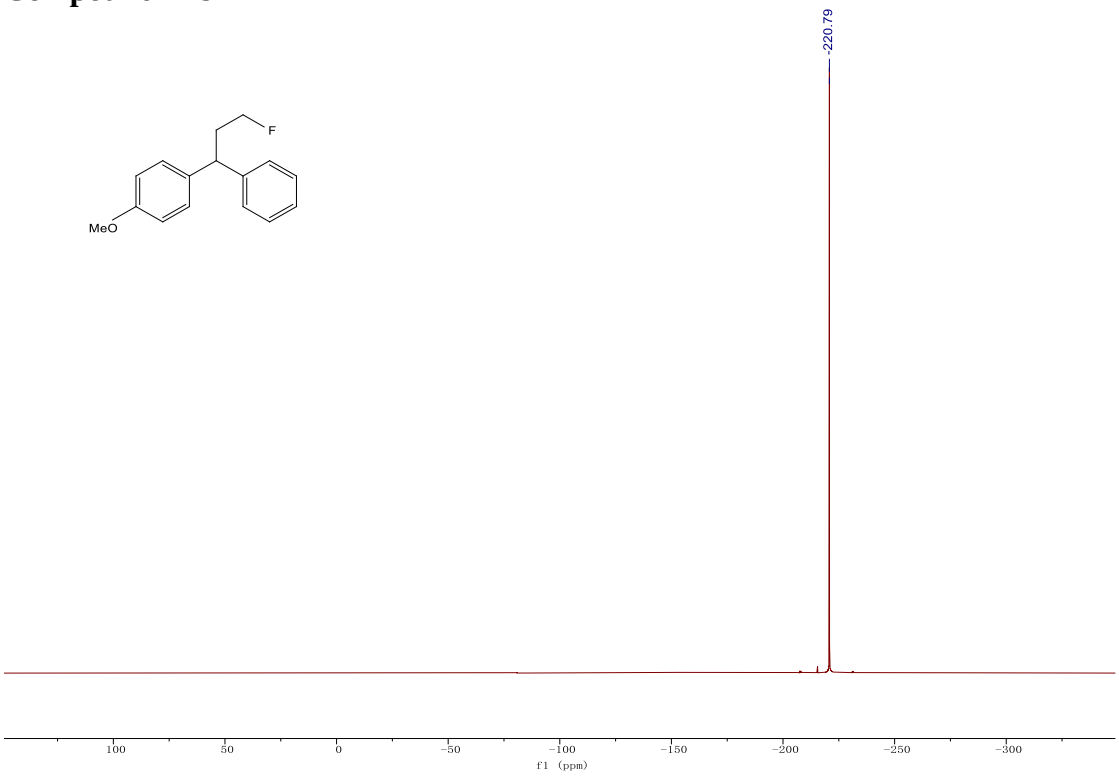
Compound 11b ^{1}H NMR



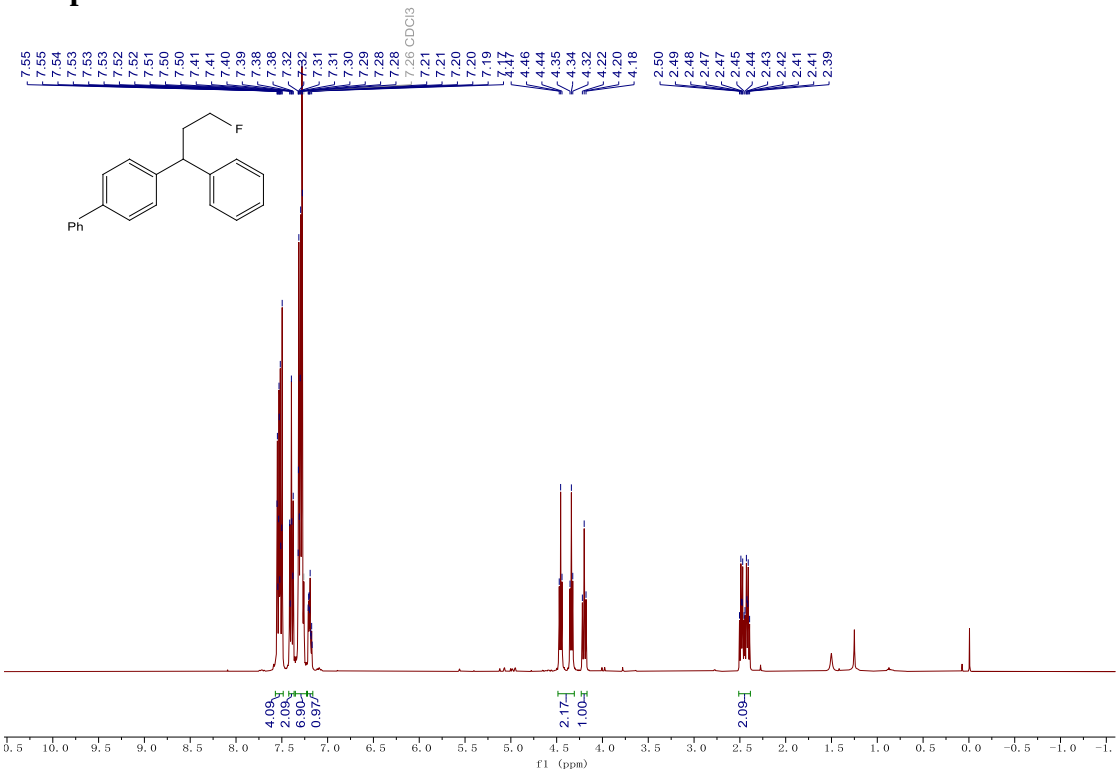
Compound 11b ^{13}C NMR



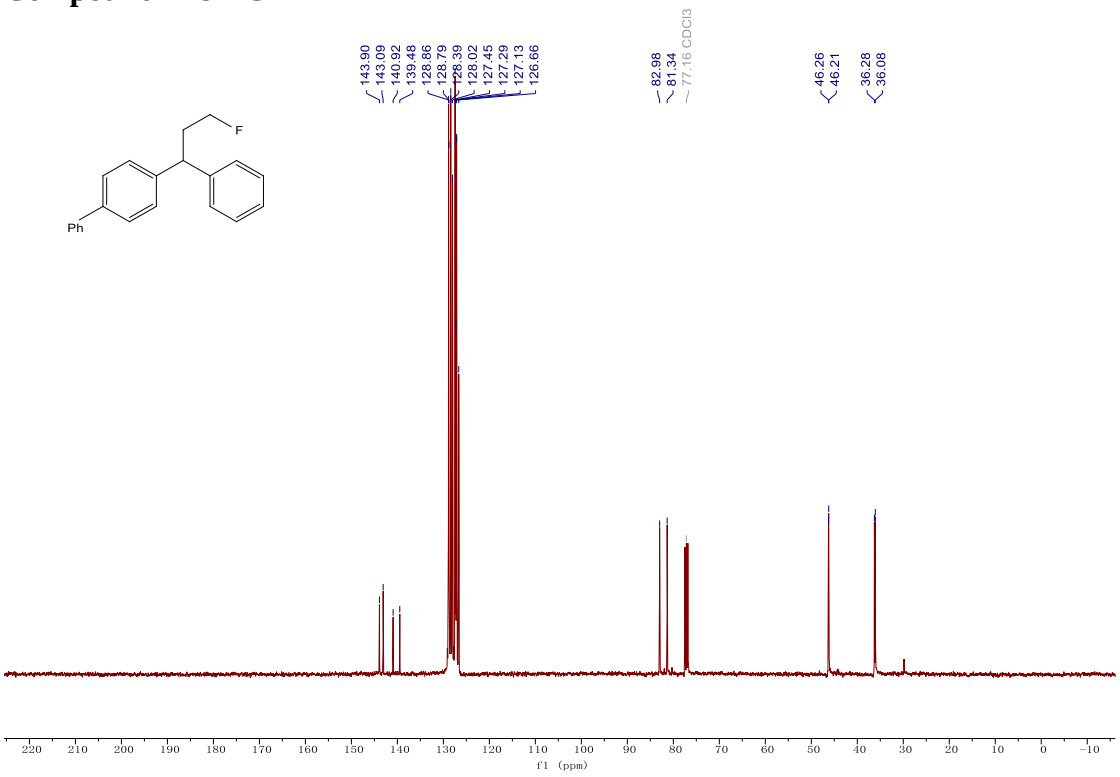
Compound 11b ^{19}F NMR



Compound 12b ^{1}H NMR



Compound 12b ^{13}C NMR



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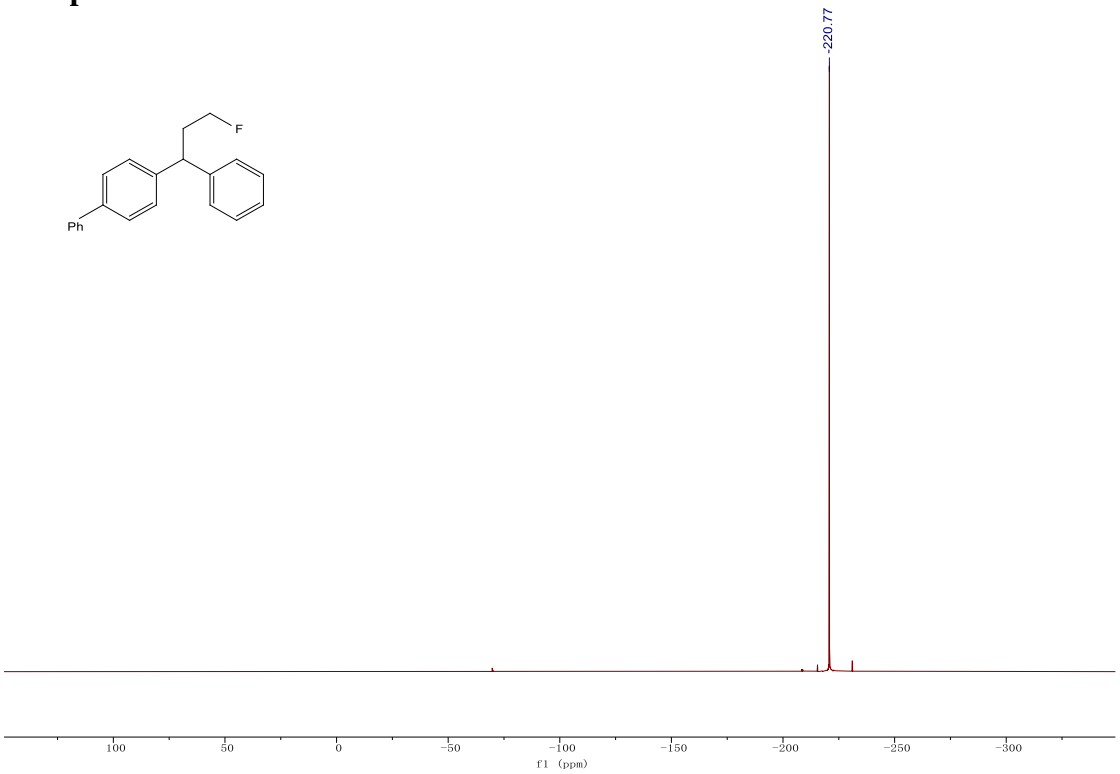
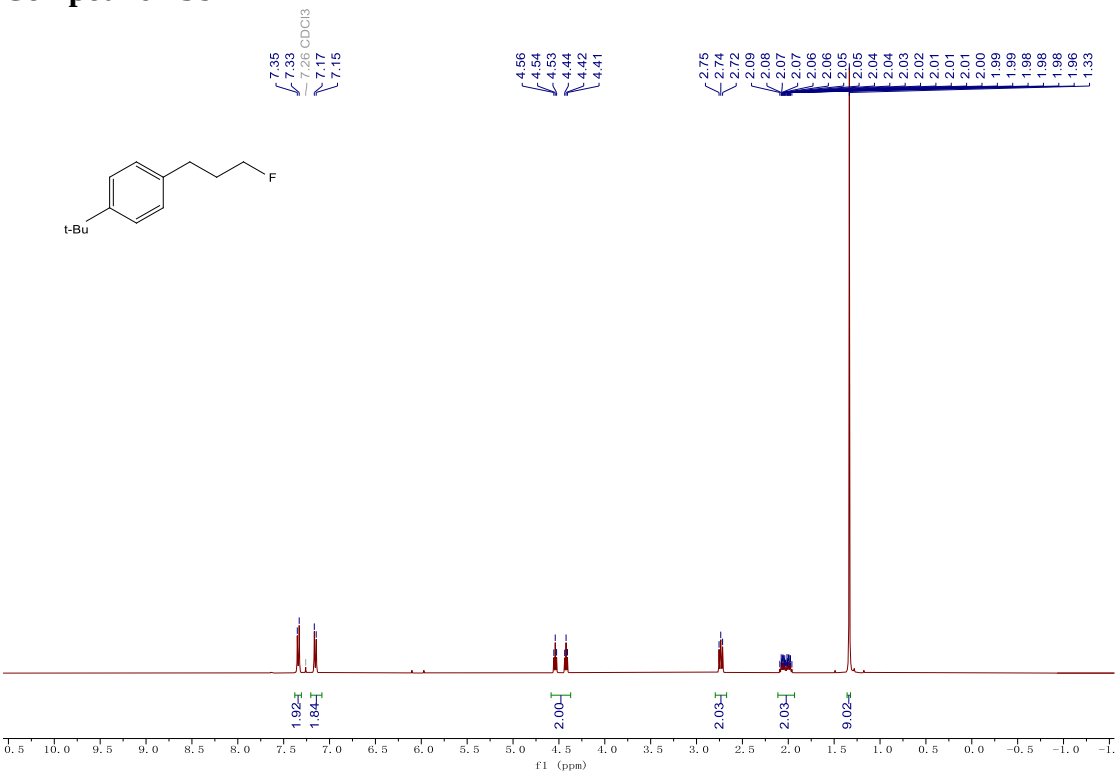
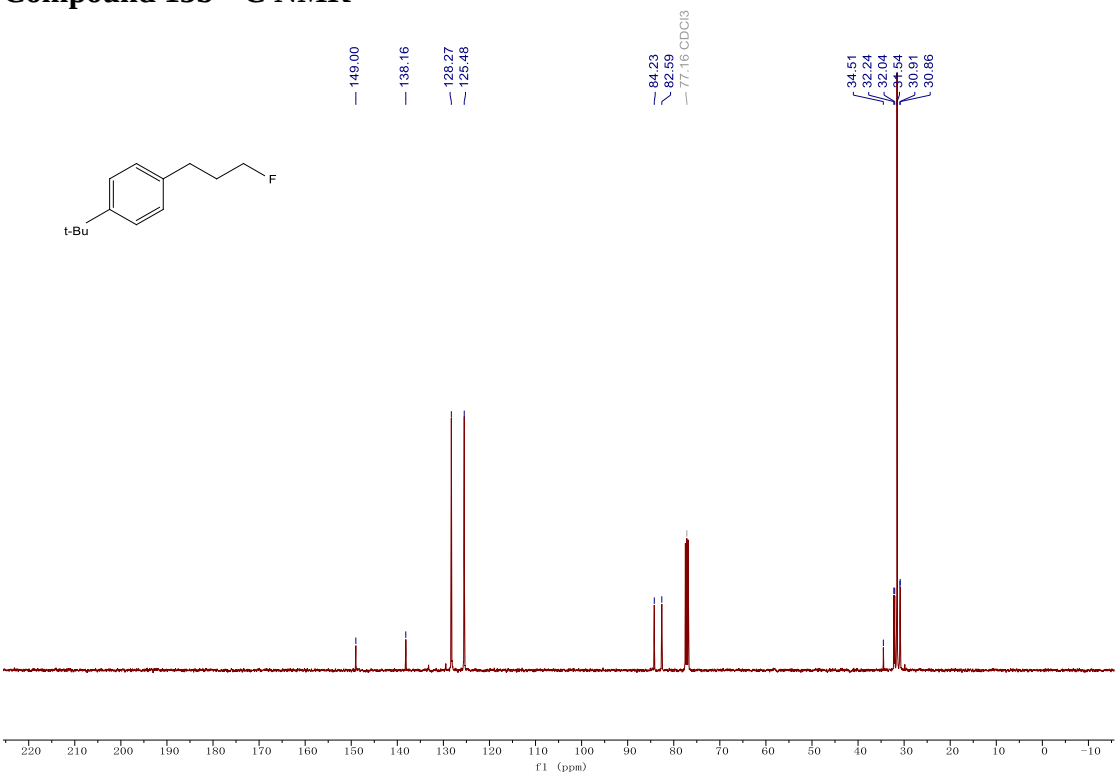


Compound 13b ^{1}H NMR



Compound 13b ^{13}C NMR



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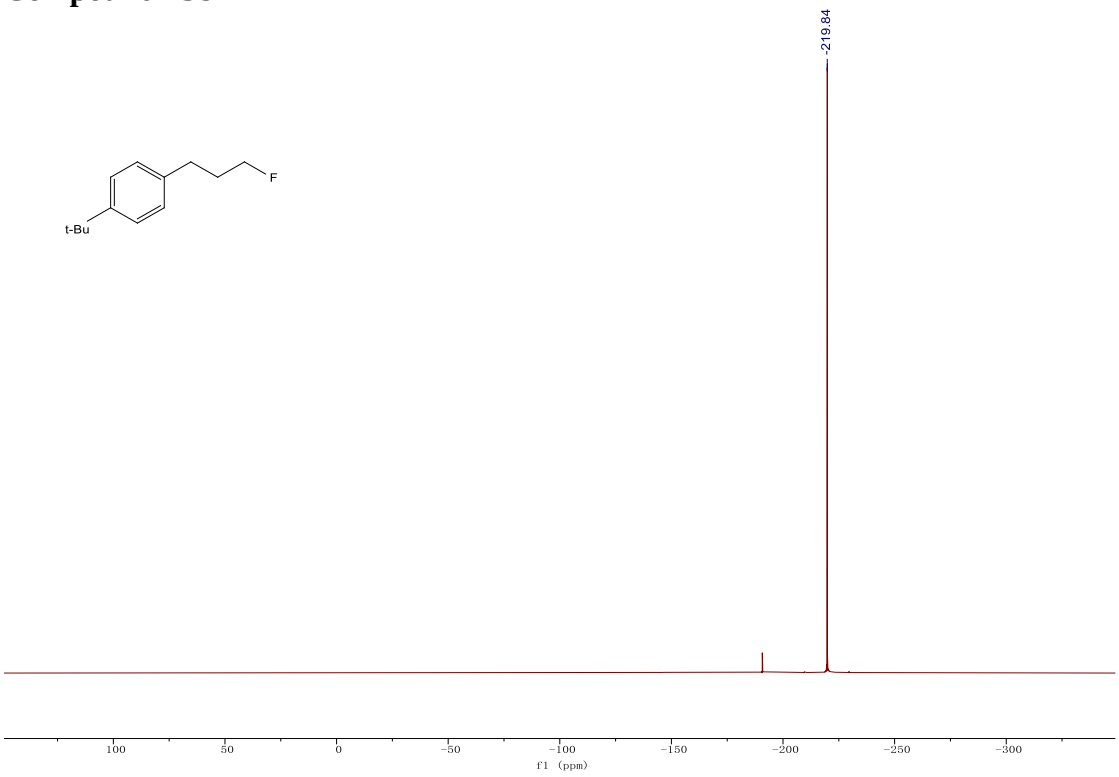
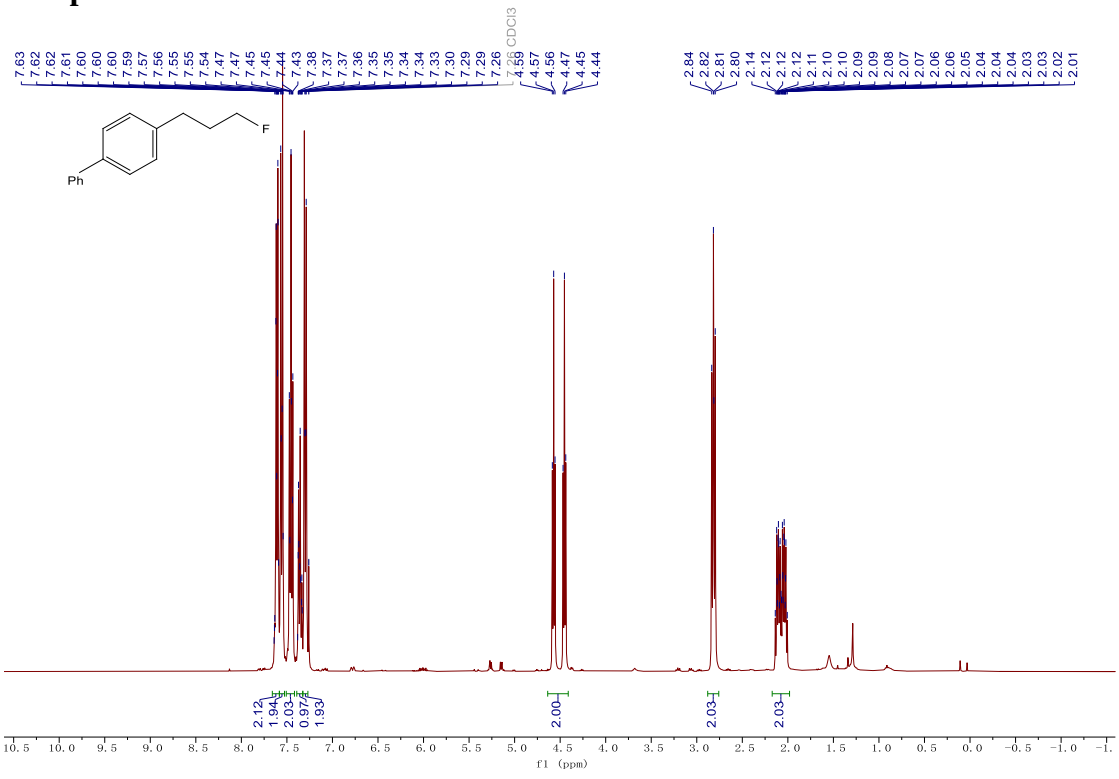
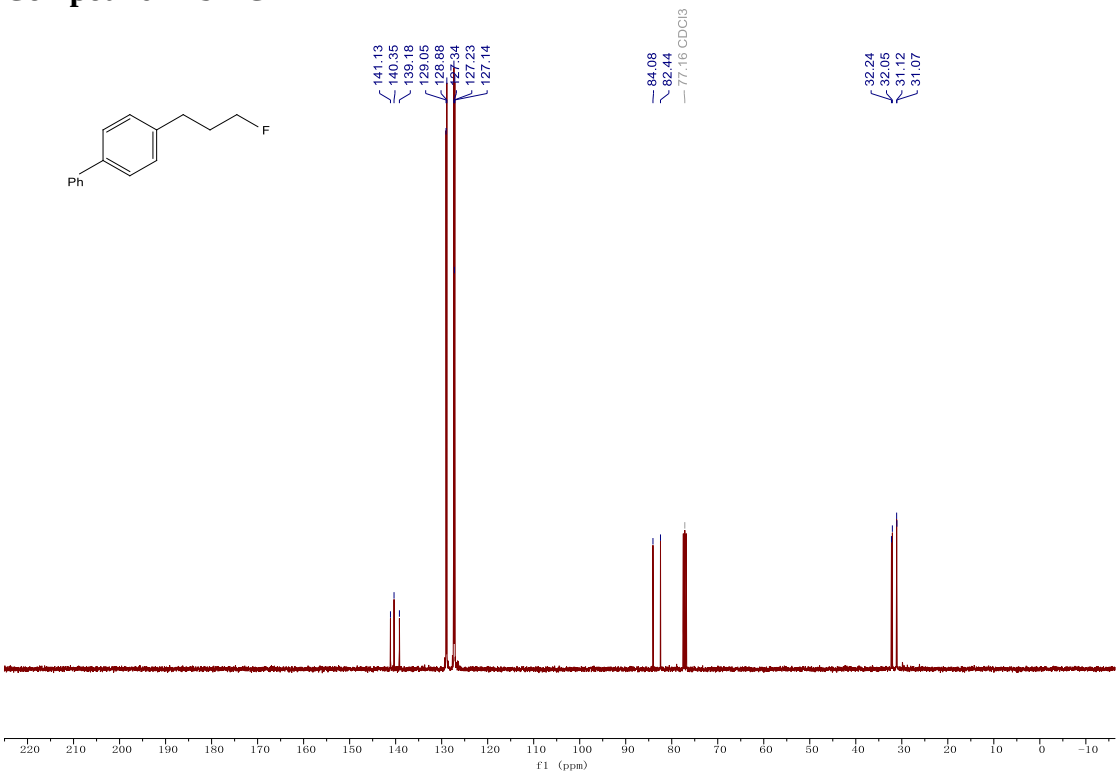


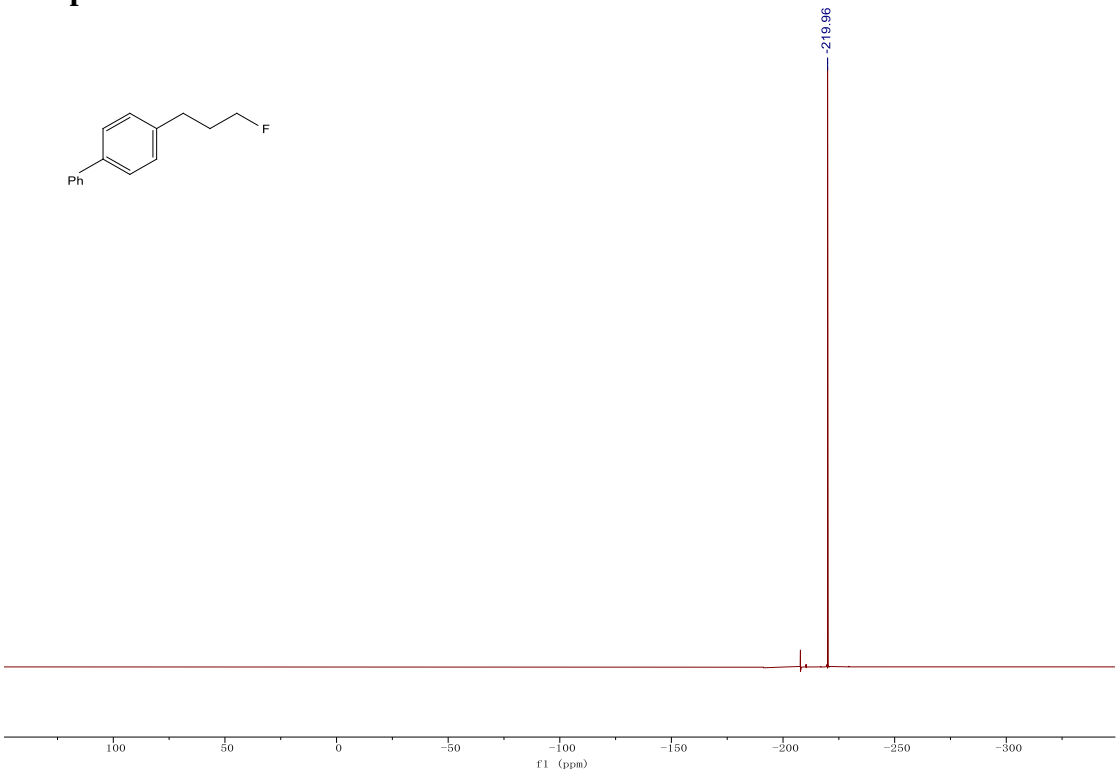
Compound 14b ^{1}H NMR



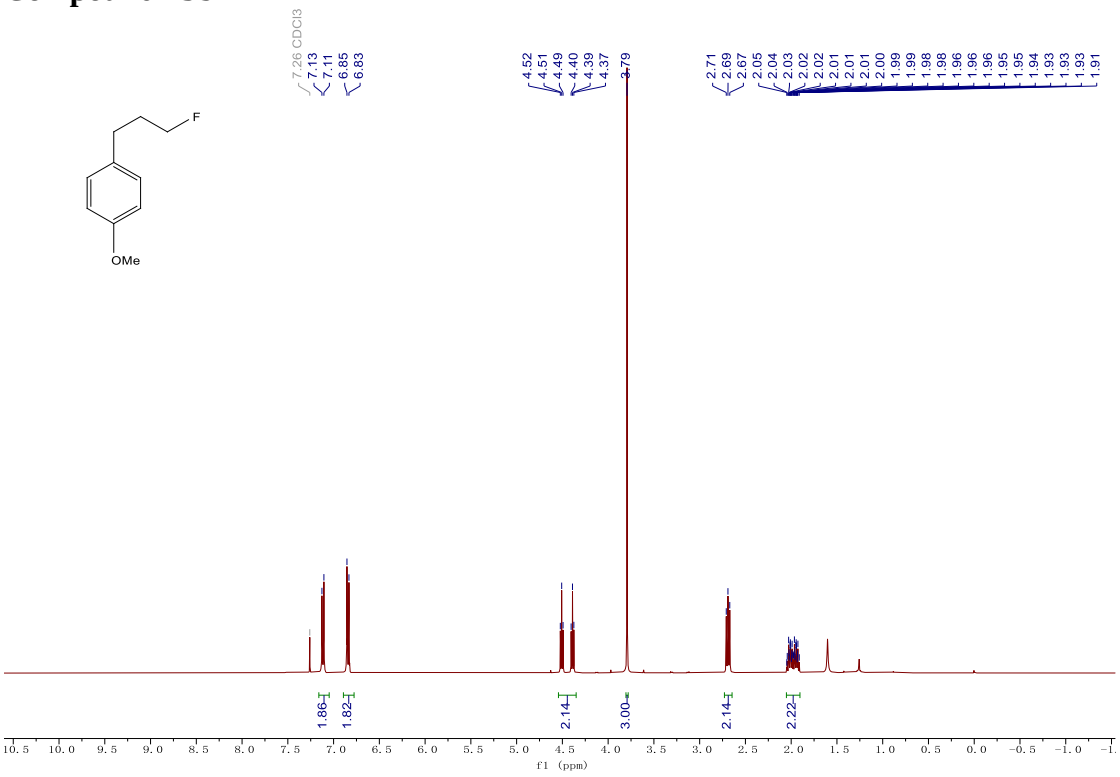
Compound 14b ^{13}C NMR



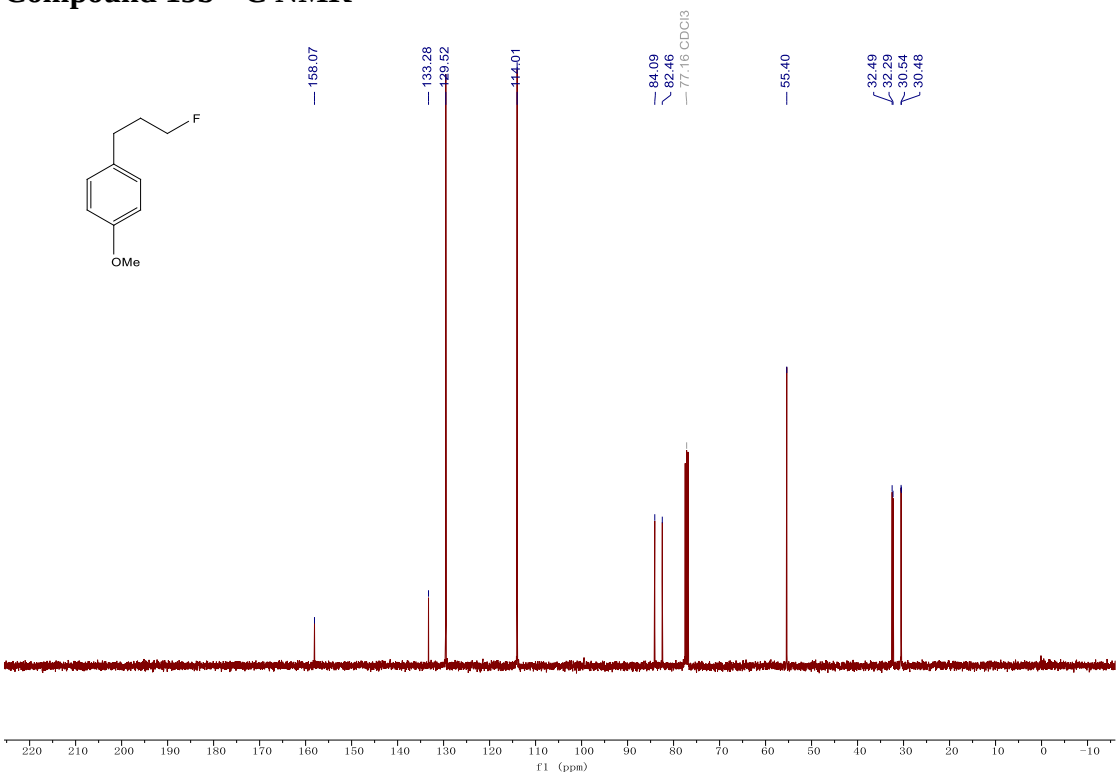
Compound 14b ^{19}F NMR



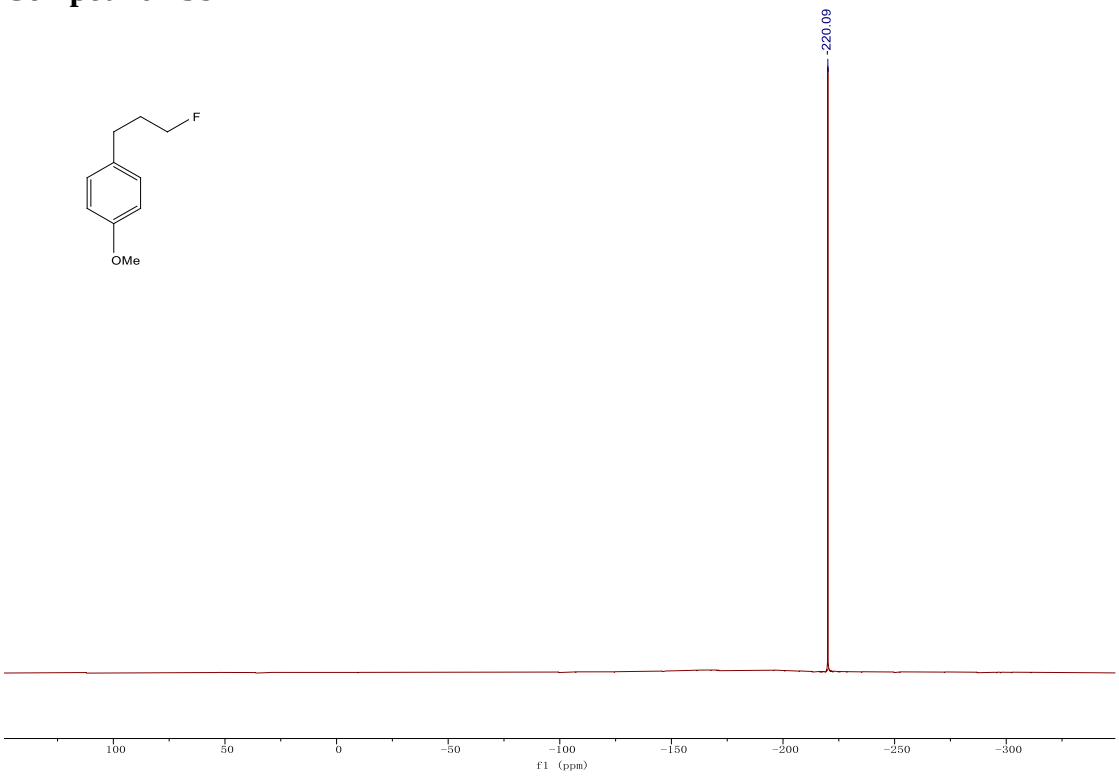
Compound 15b ^{1}H NMR



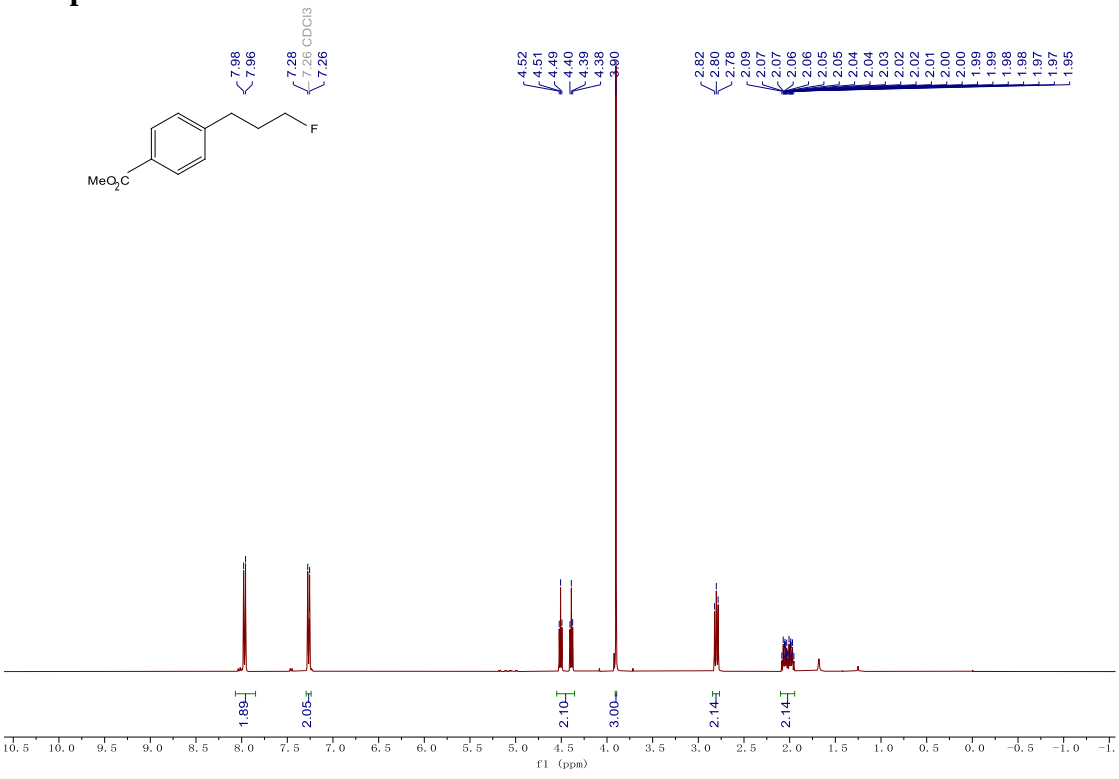
Compound 15b ^{13}C NMR



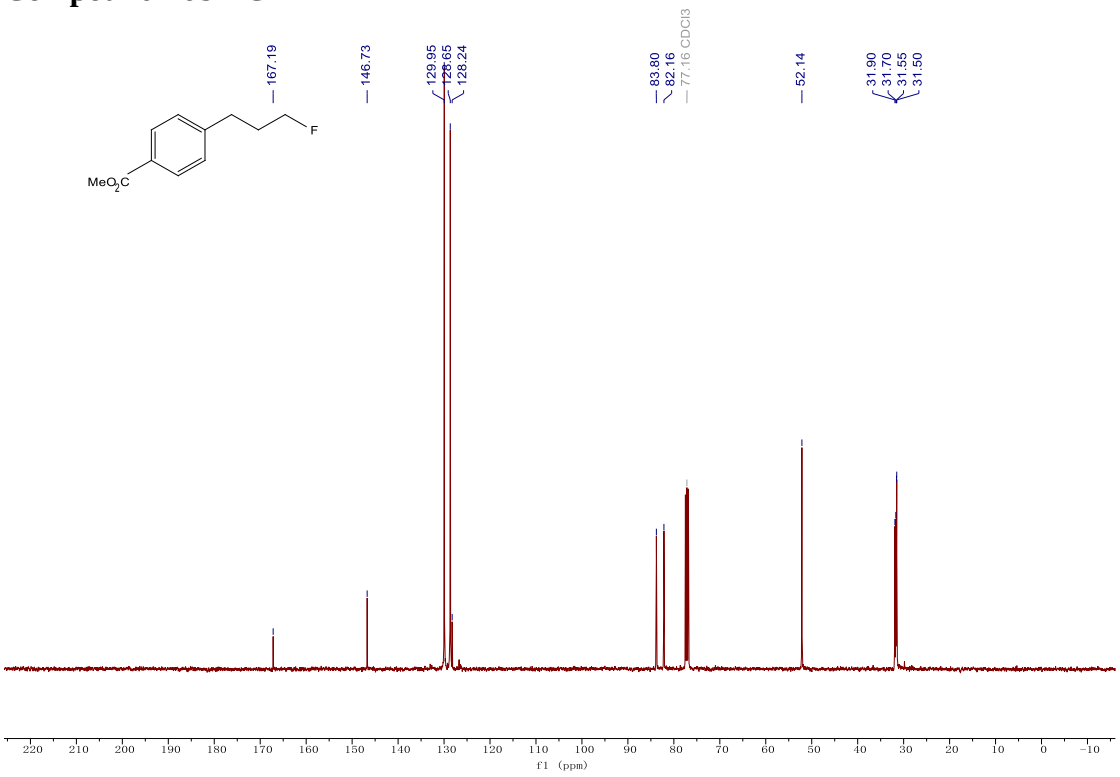
Compound 15b ^{19}F NMR



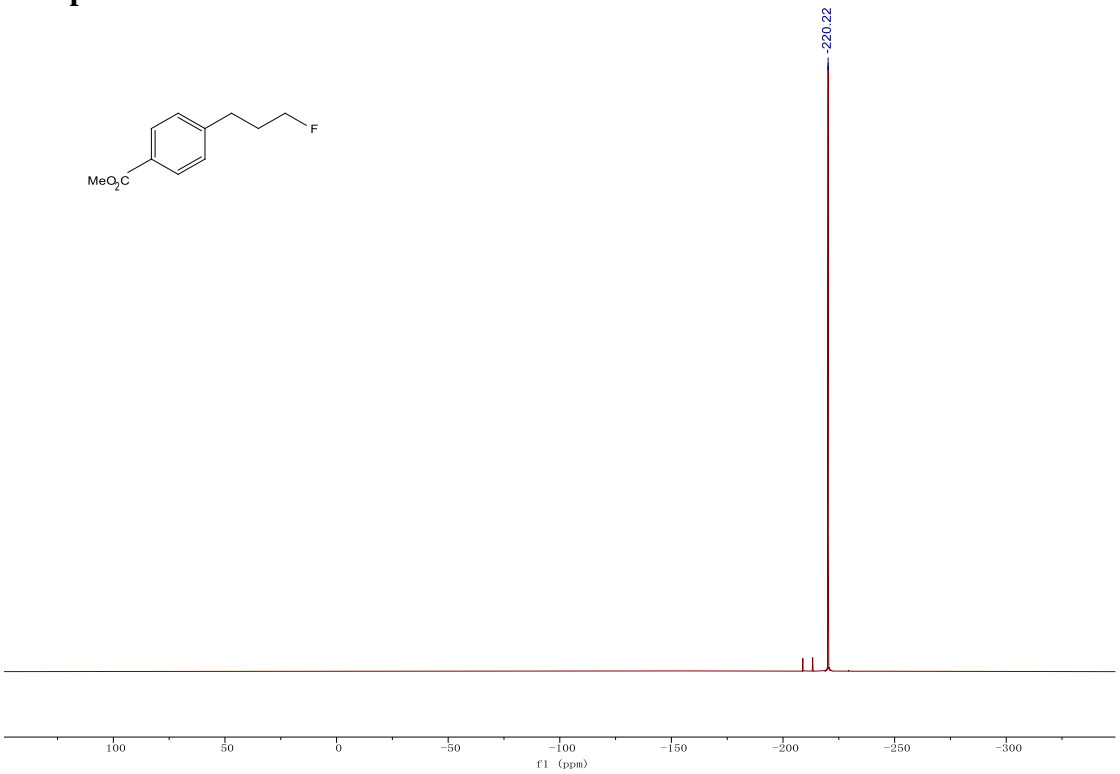
Compound 16b ^{1}H NMR



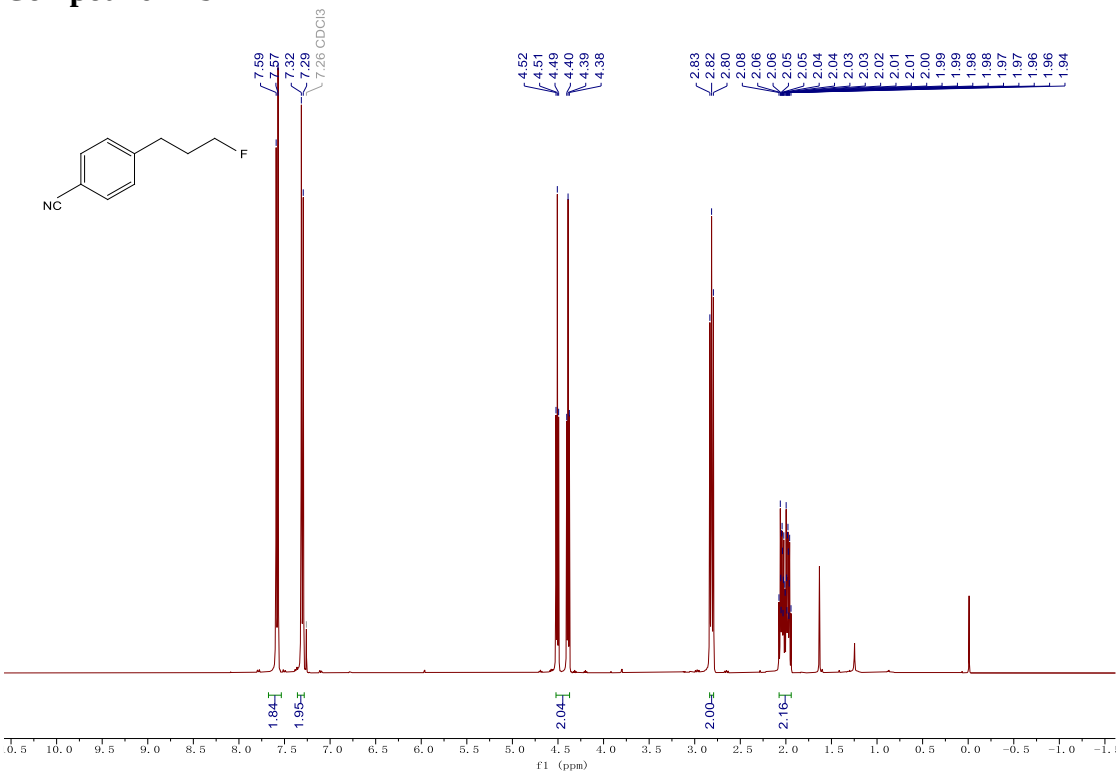
Compound 16b ^{13}C NMR



Compound 16b ^{19}F NMR



Compound 17b ^{1}H NMR



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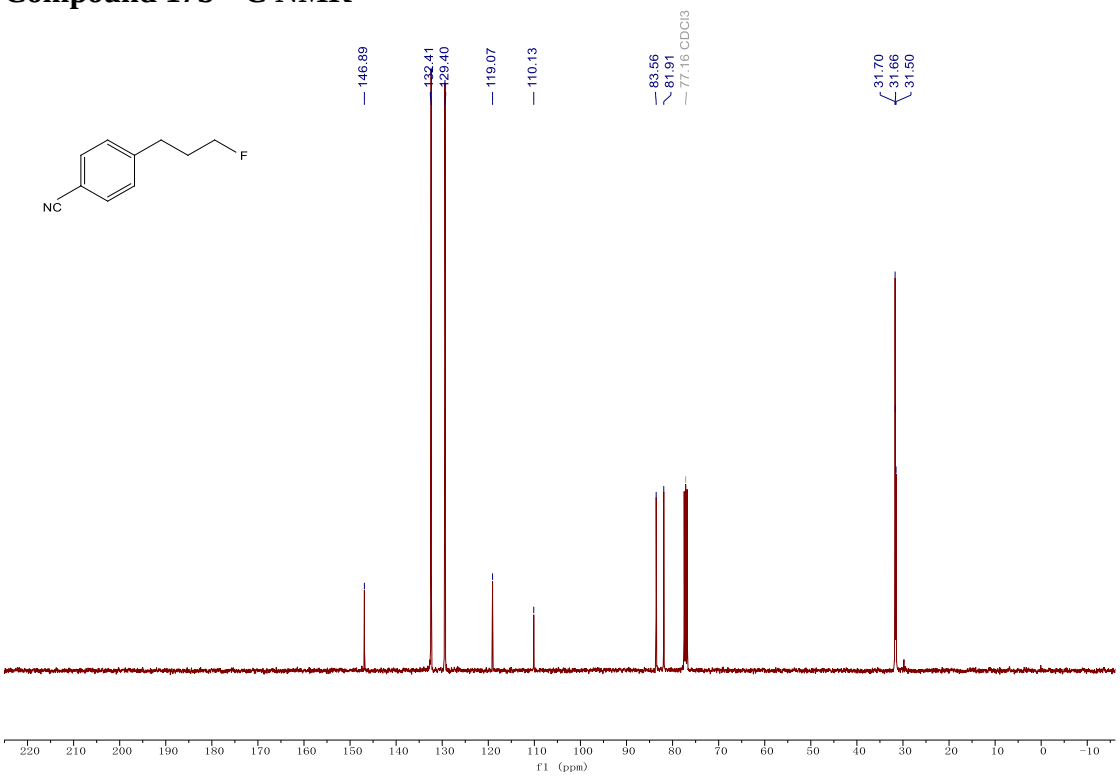

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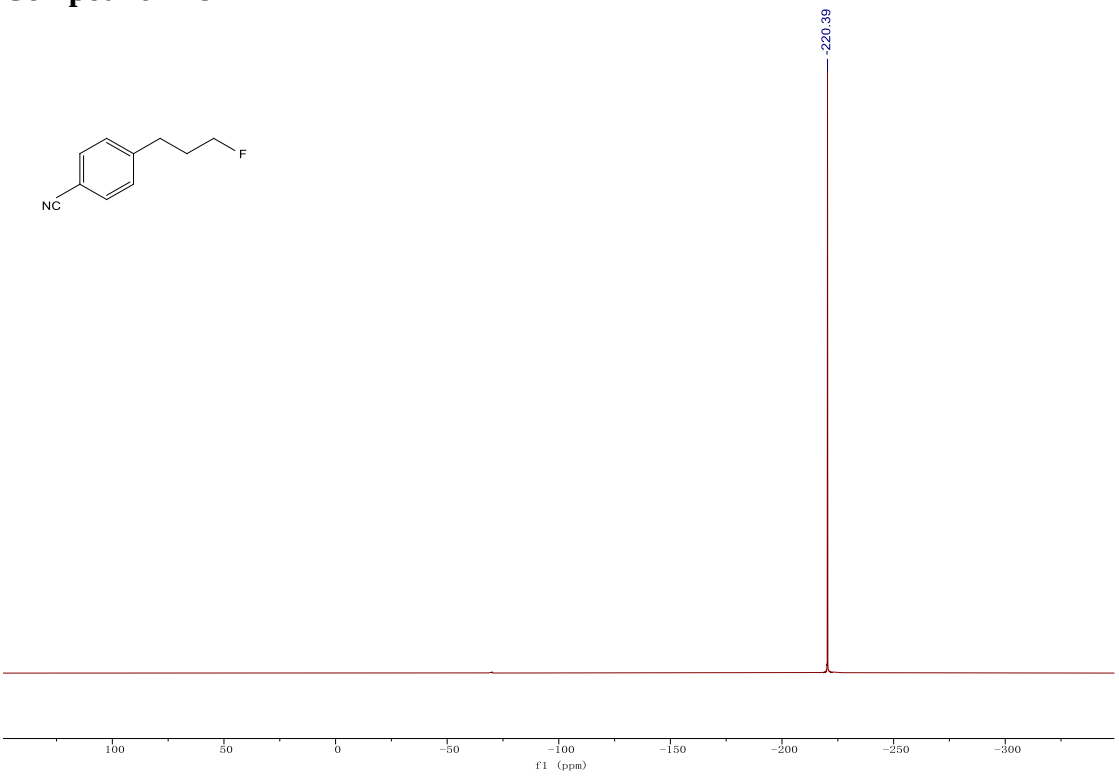

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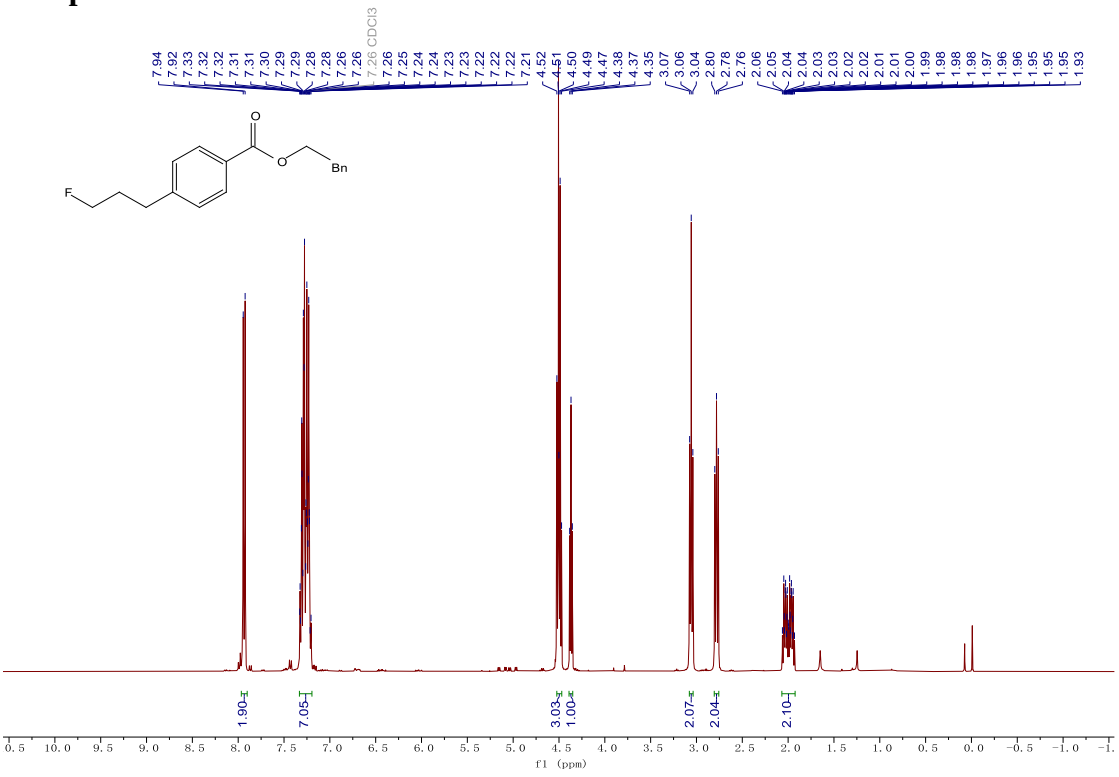
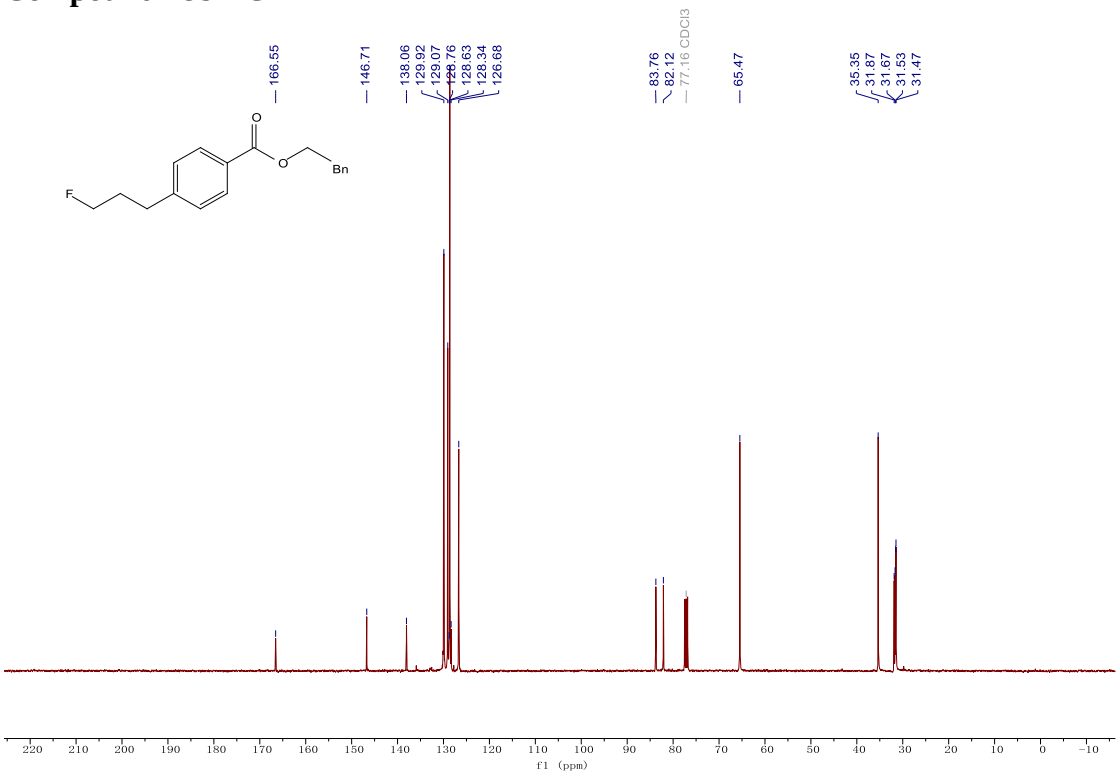


Compound 18b ^{13}C NMR



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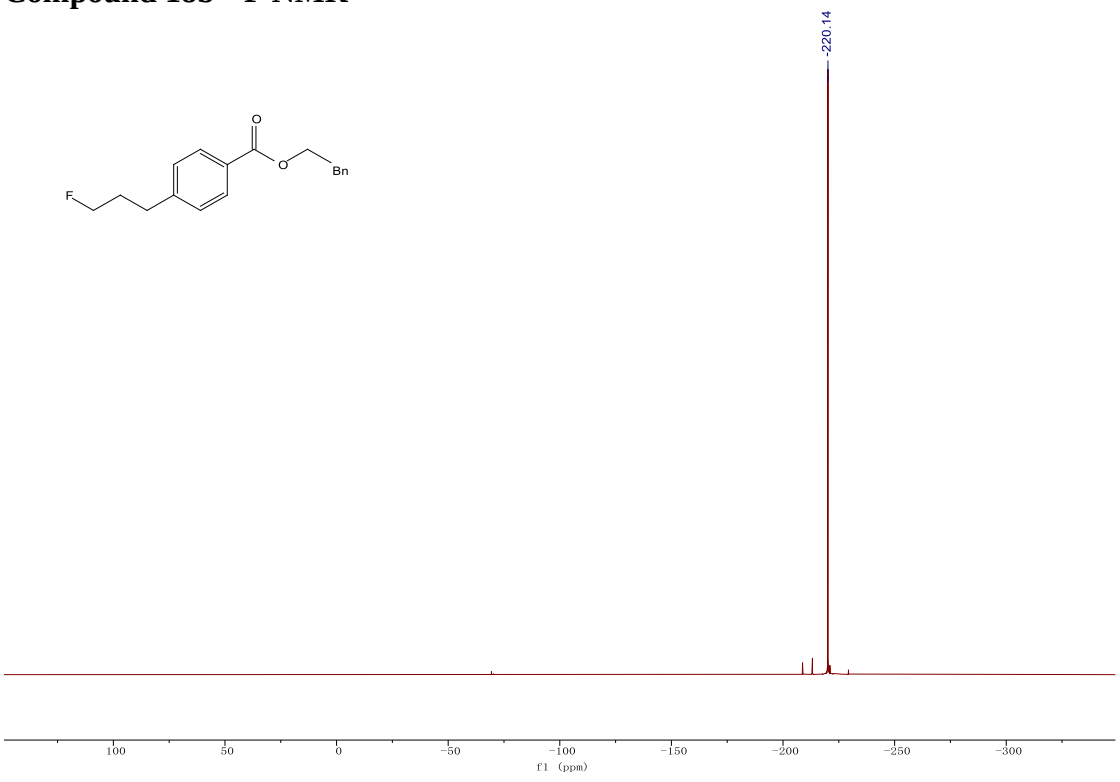
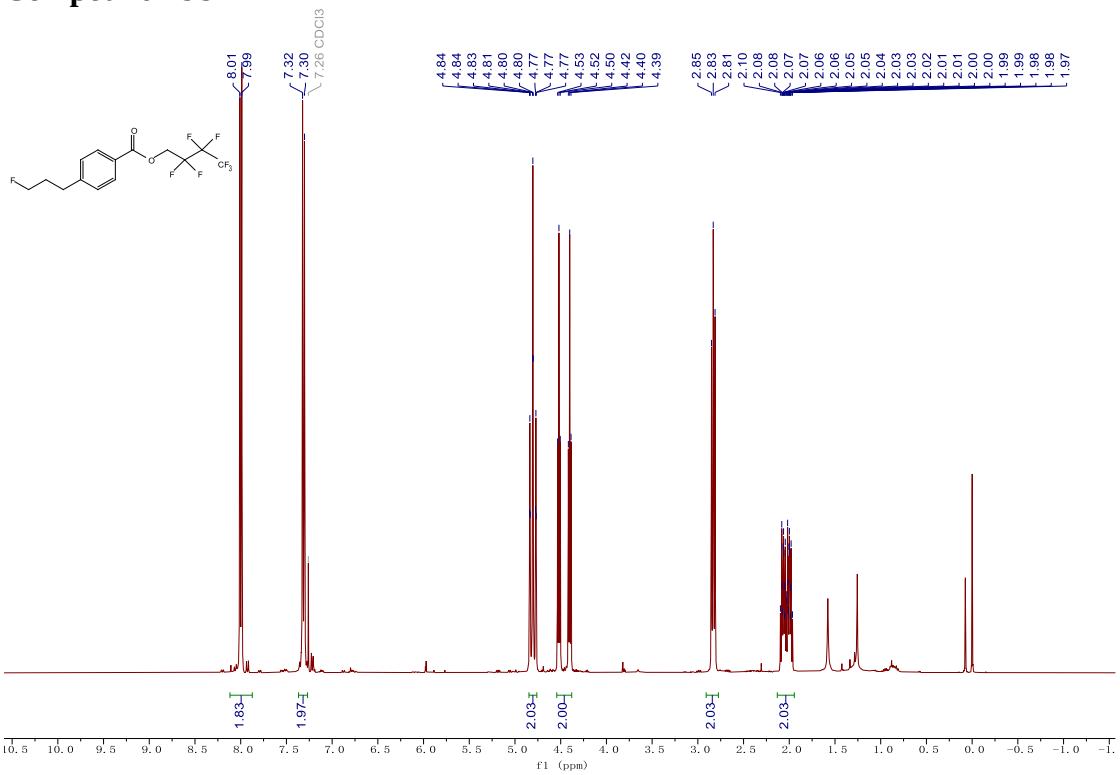
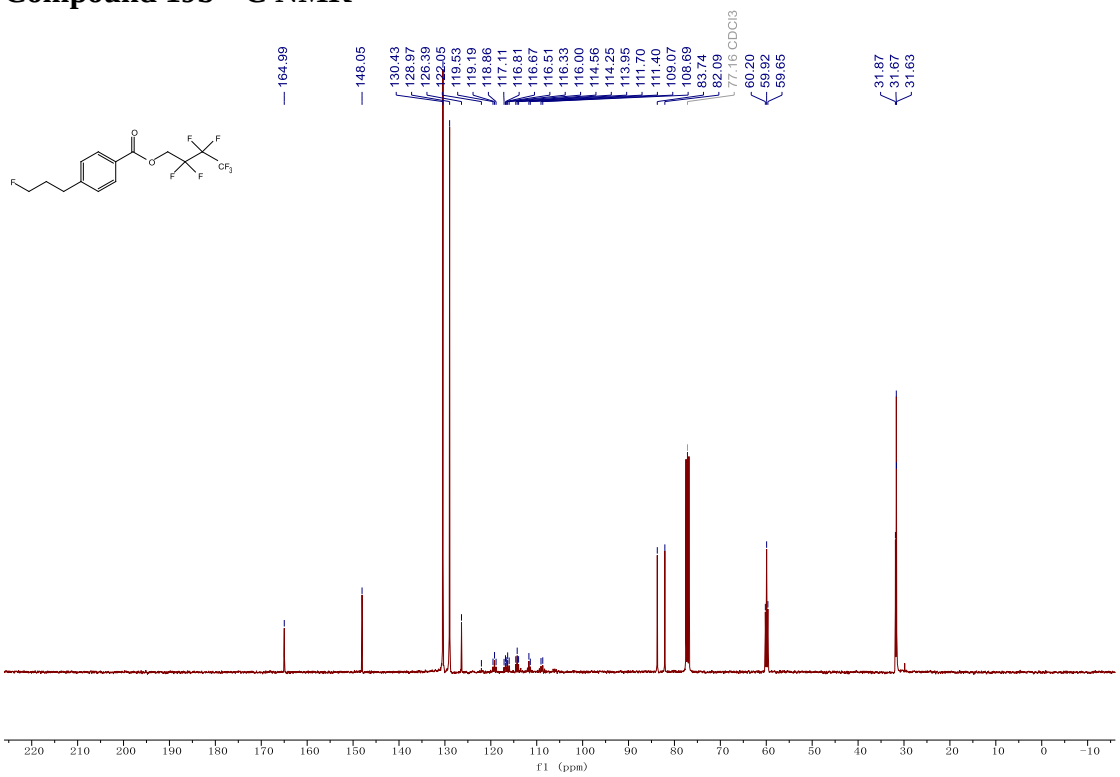


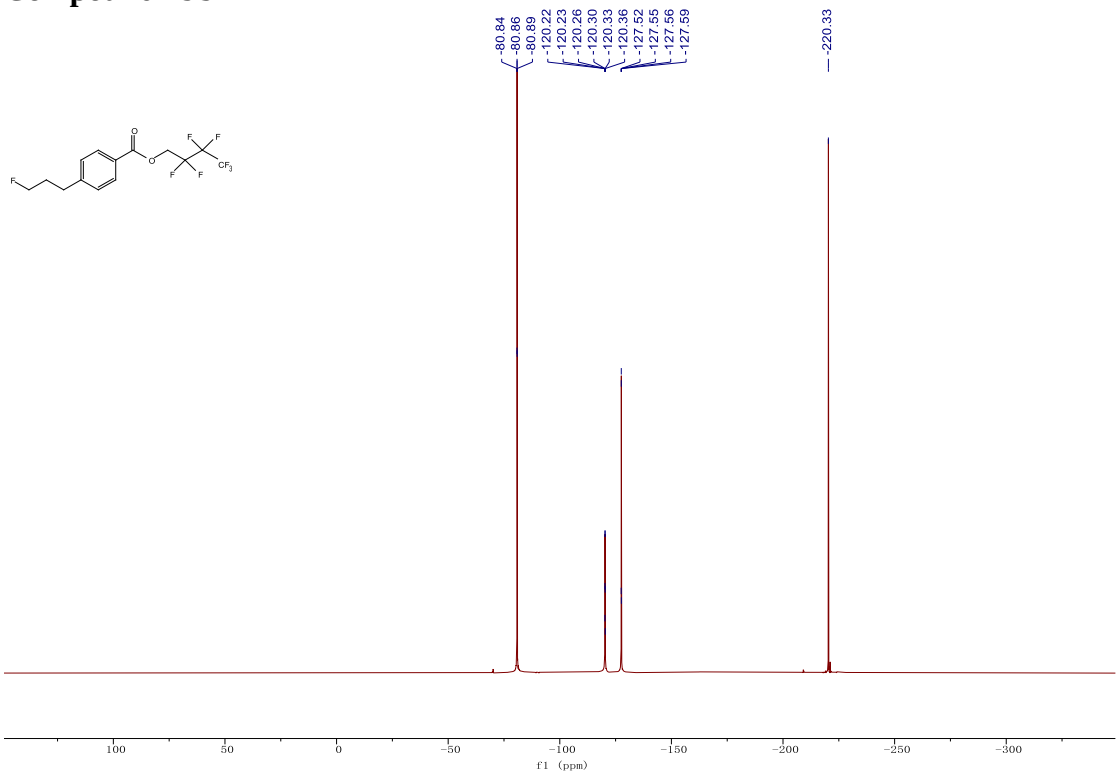
Compound 19b ^{1}H NMR



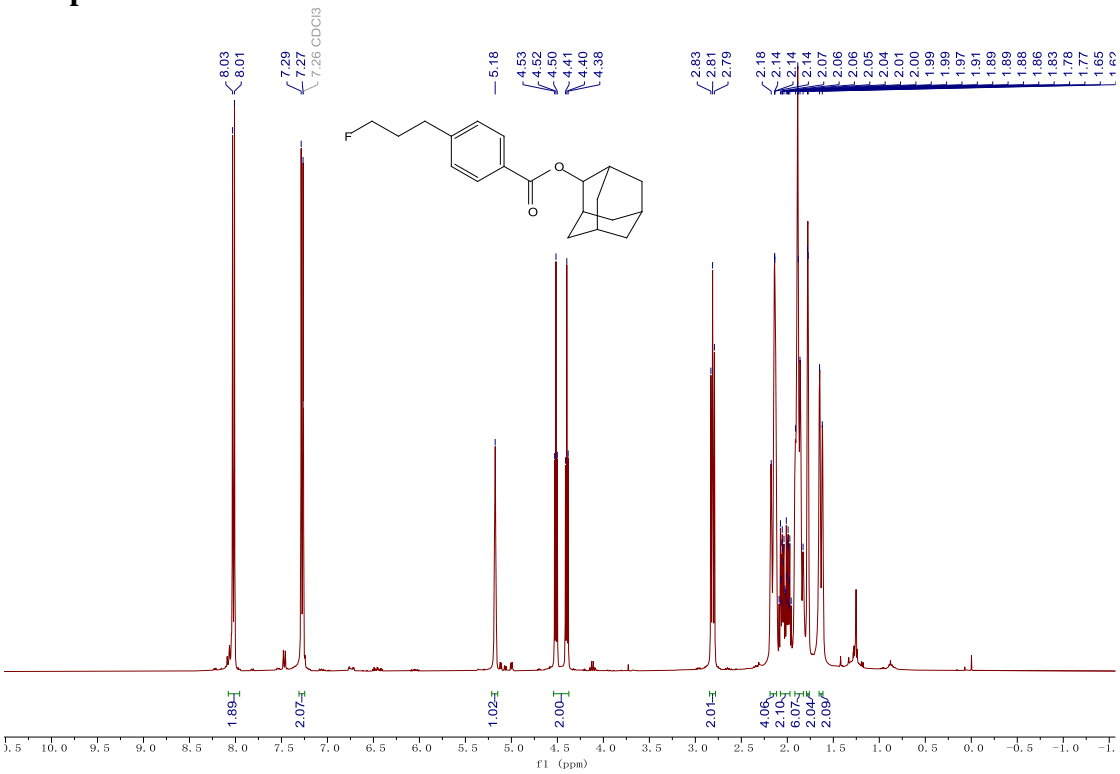
Compound 19b ^{13}C NMR



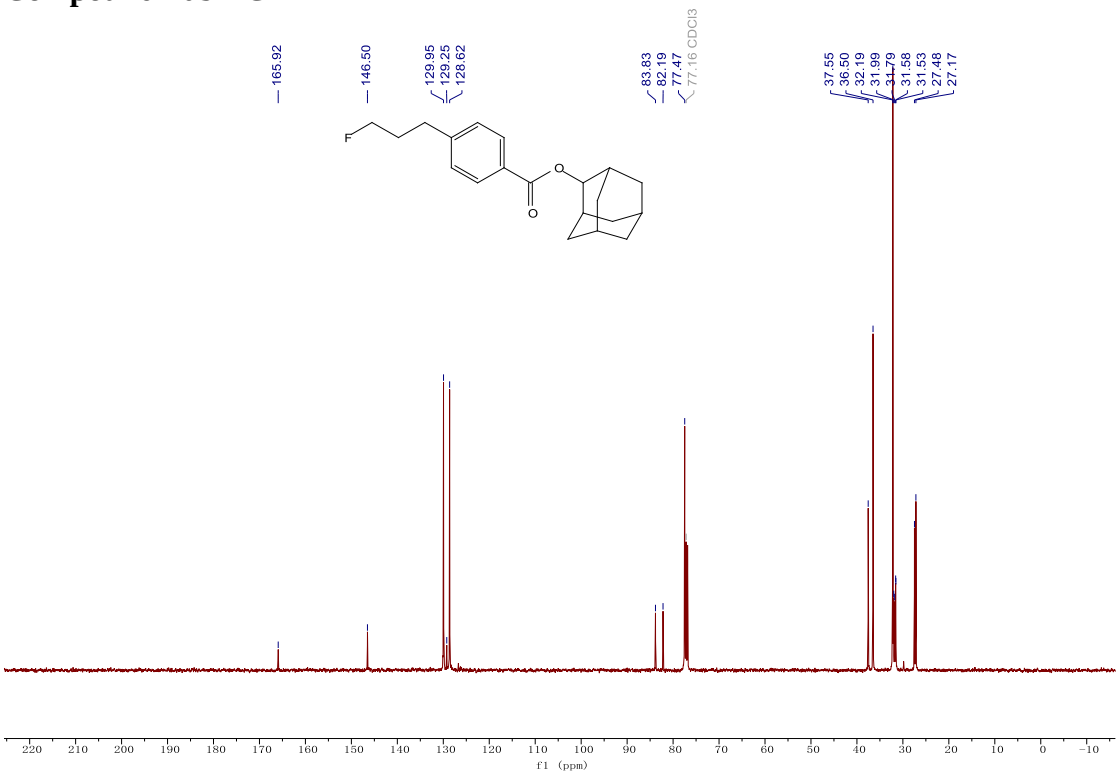
Compound 19b ^{19}F NMR



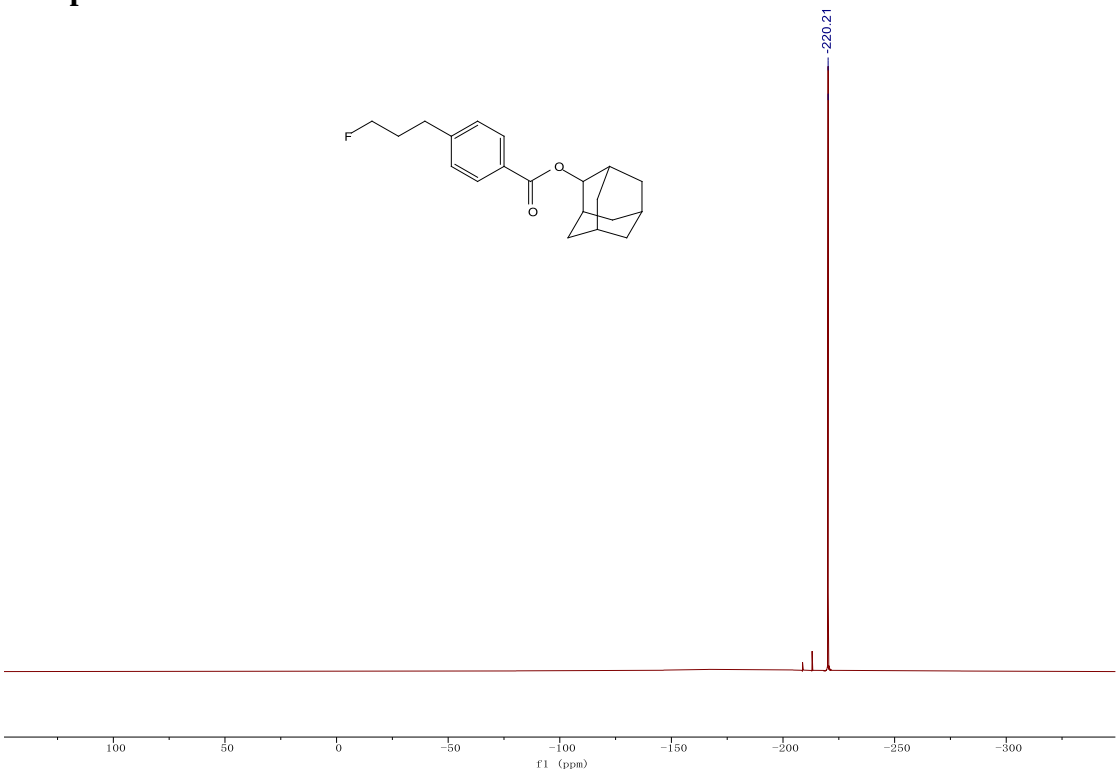
Compound 20b ^{1}H NMR



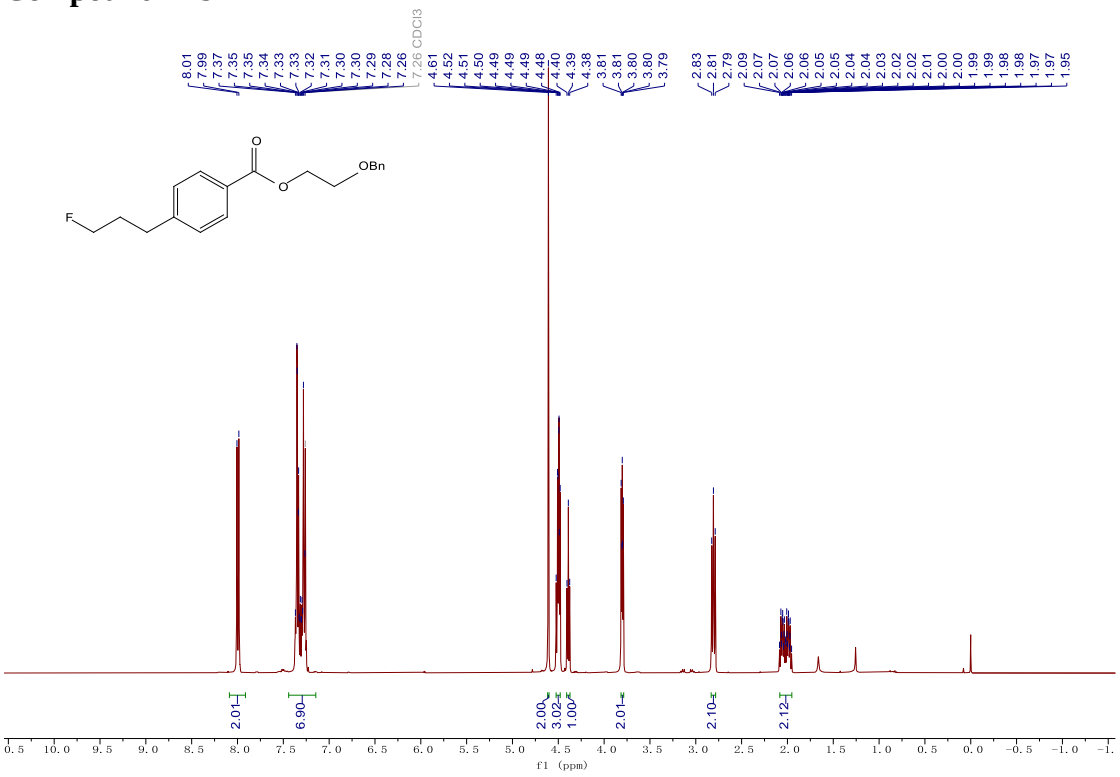
Compound 20b ^{13}C NMR



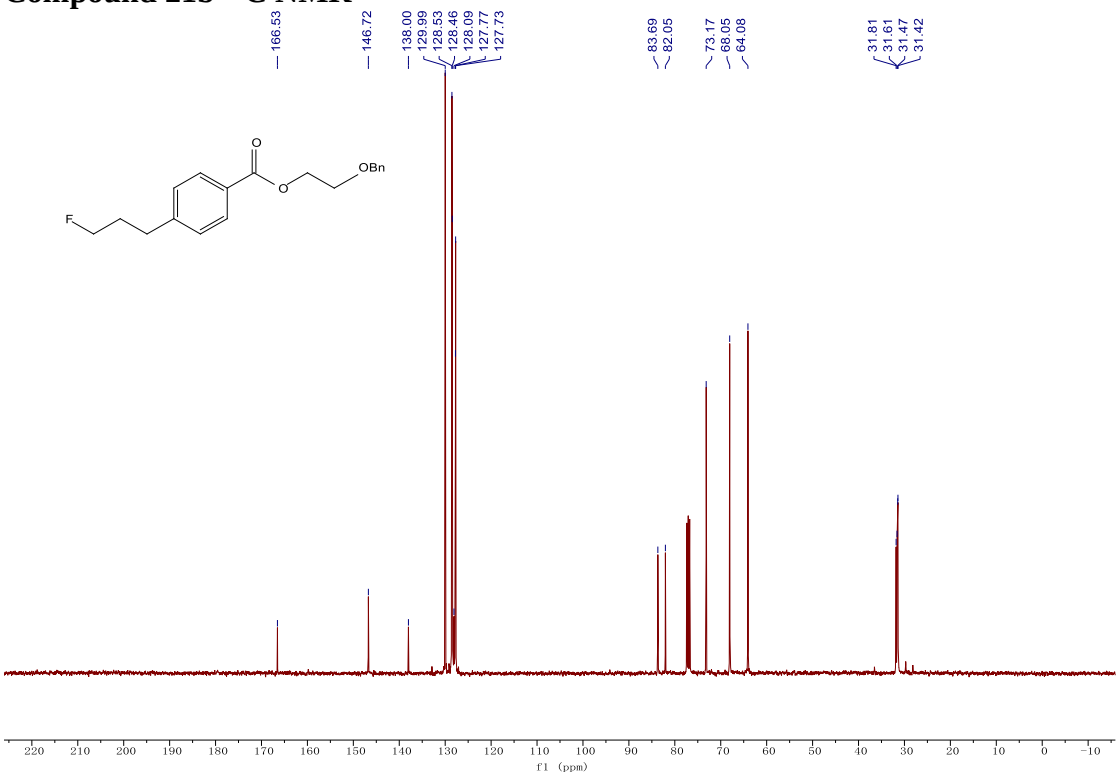
Compound 20b ^{19}F NMR



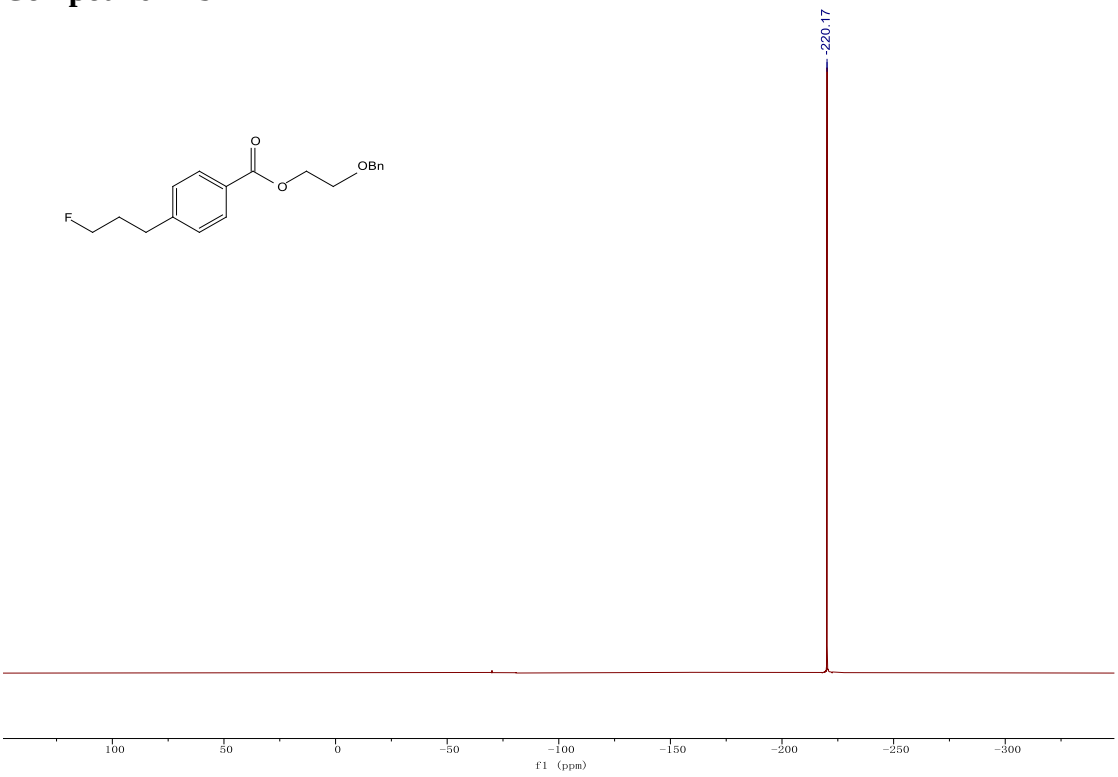
Compound 21b ^{1}H NMR



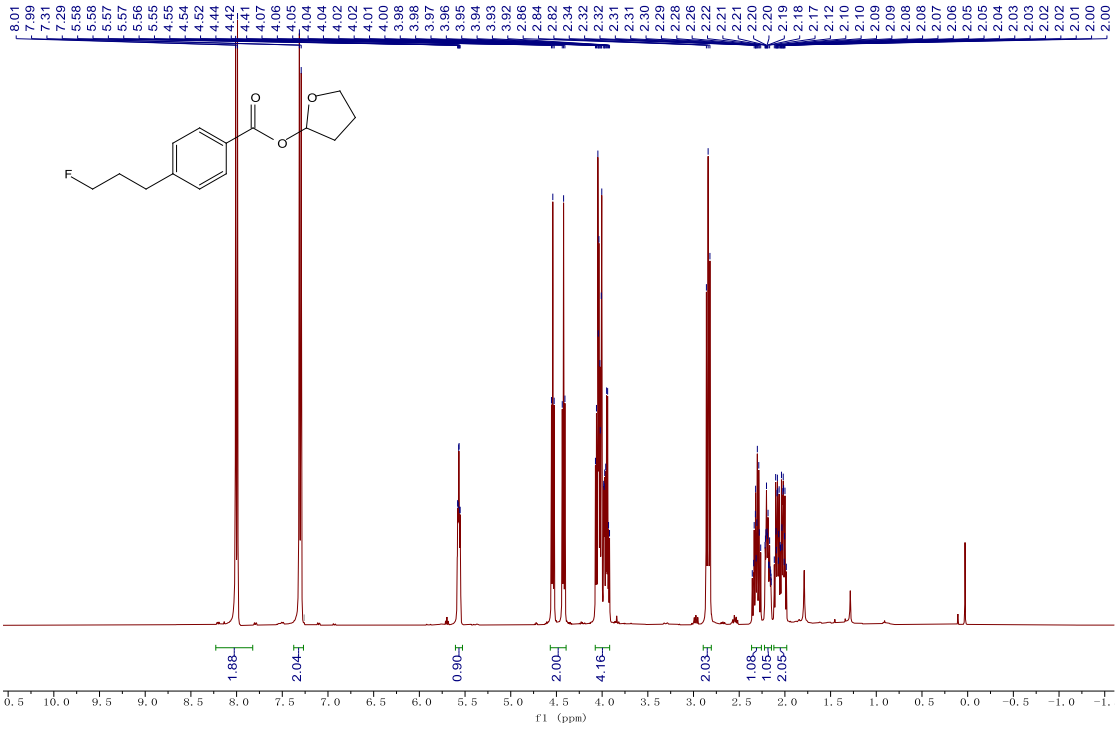
Compound 21b ^{13}C NMR



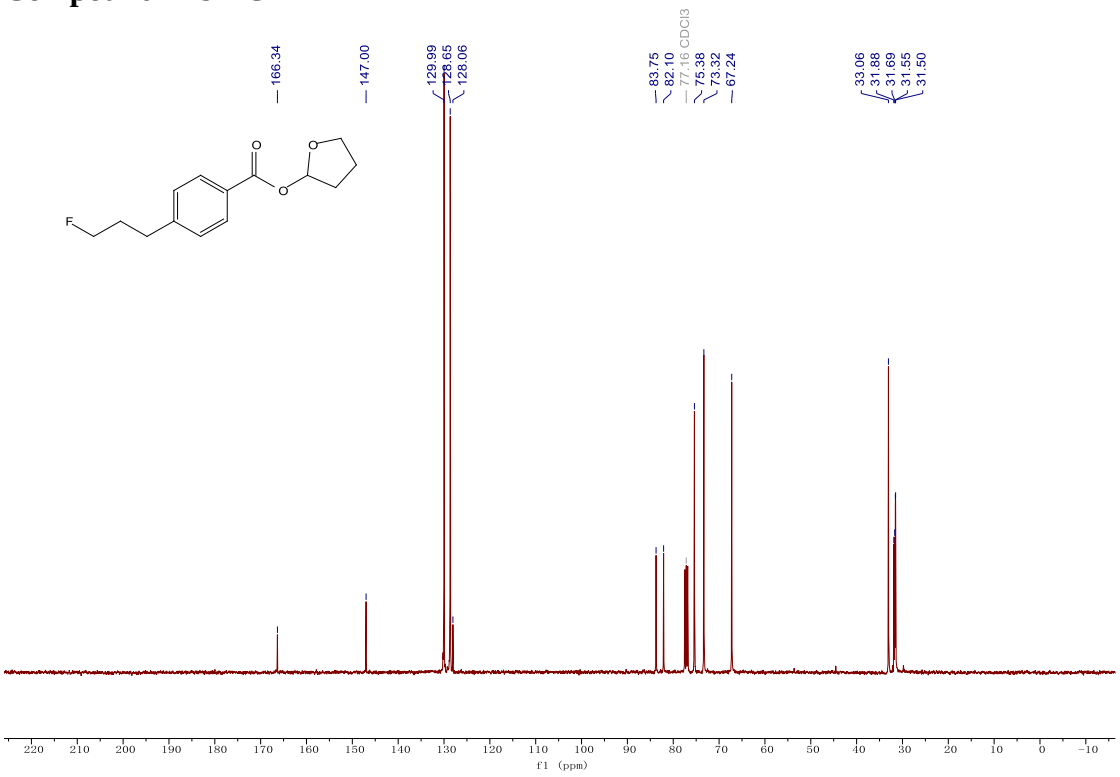
Compound 21b ^{19}F NMR



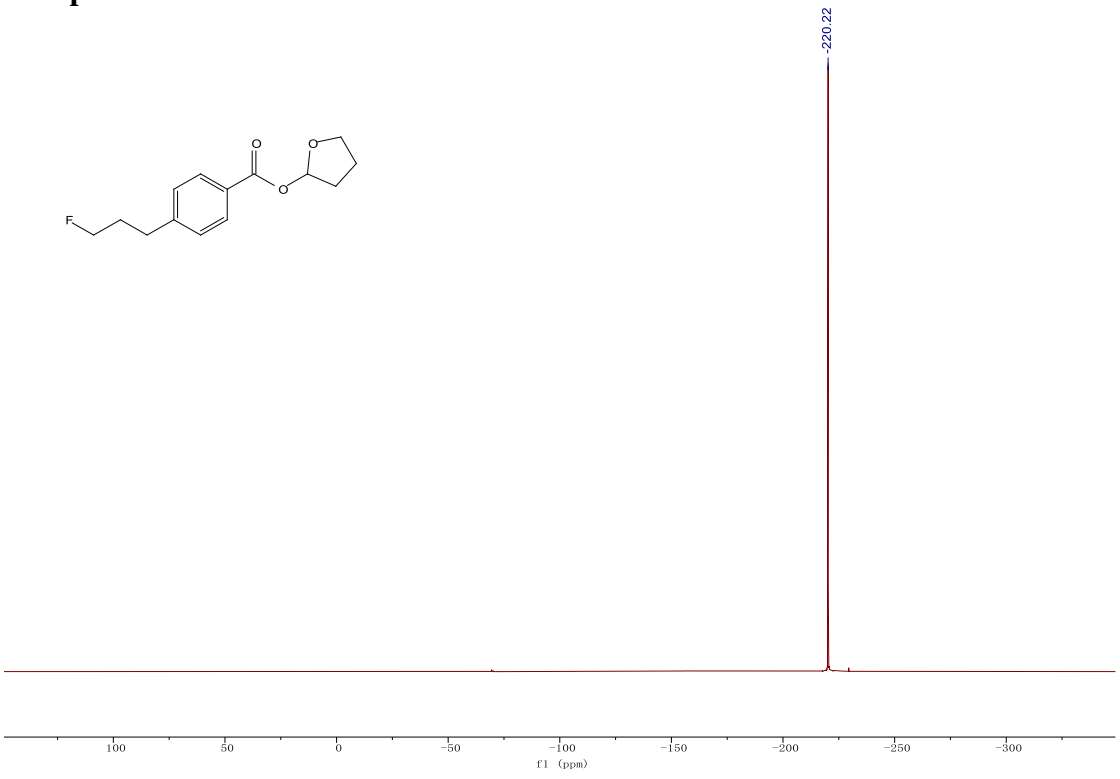
Compound 22b ^{1}H NMR



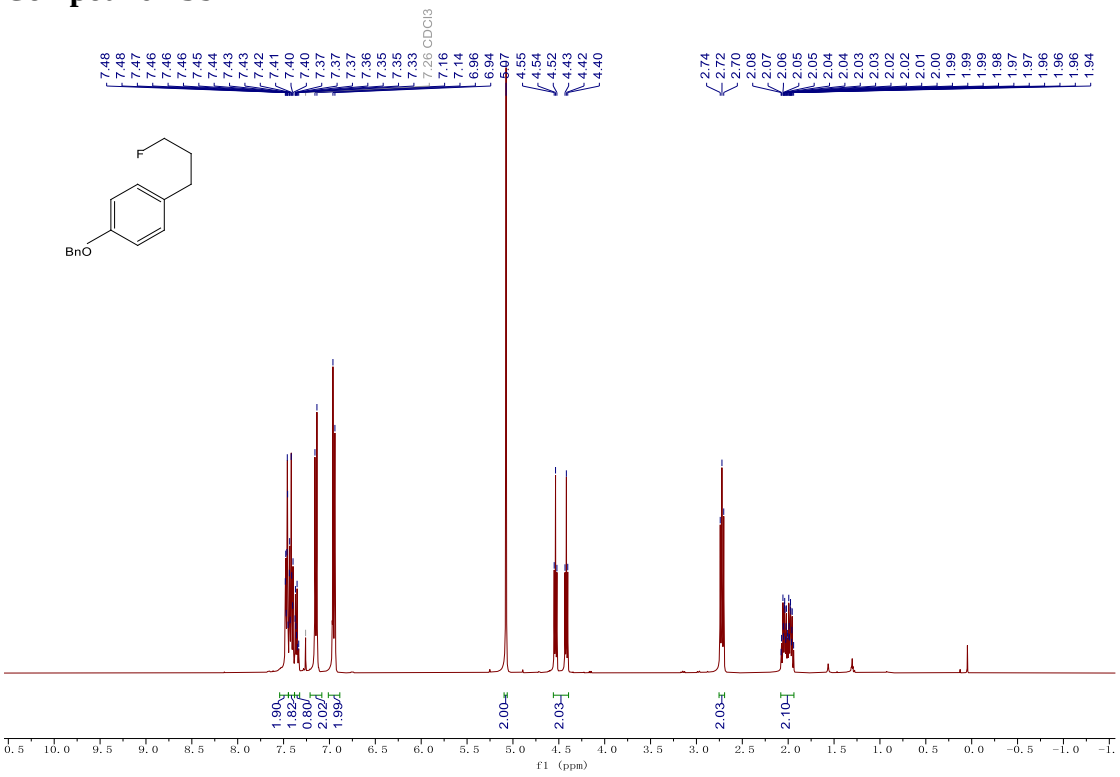
Compound 22b ^{13}C NMR



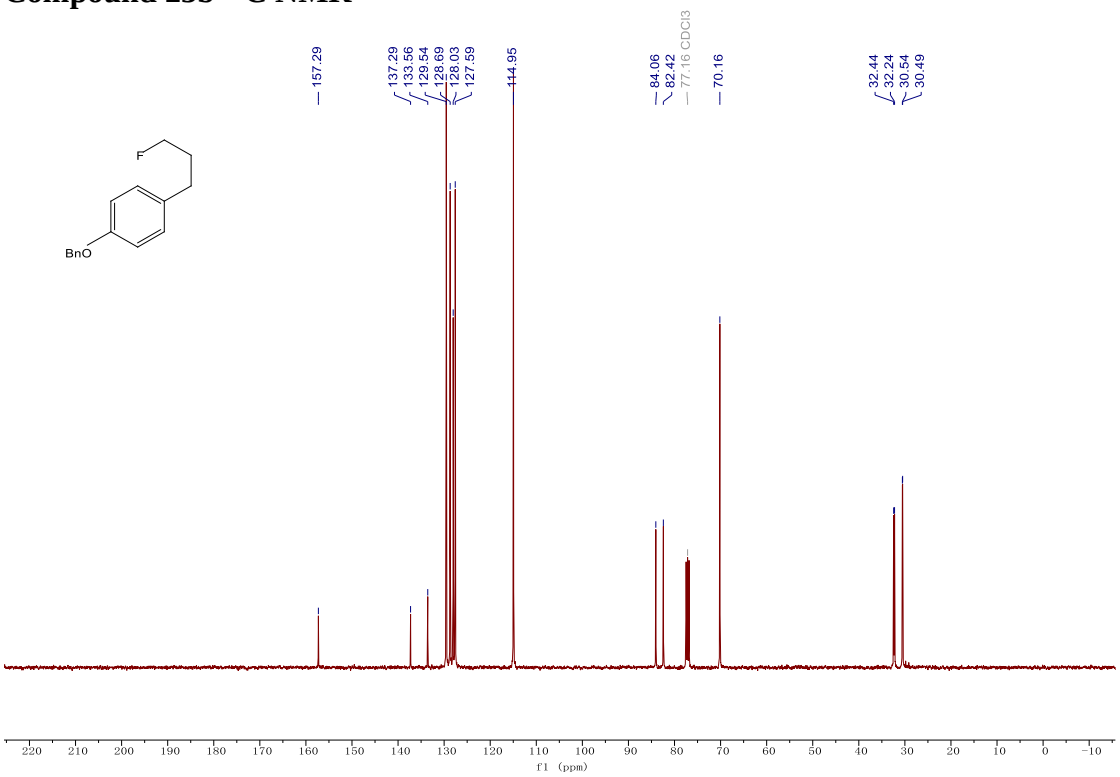
Compound 22b ^{19}F NMR



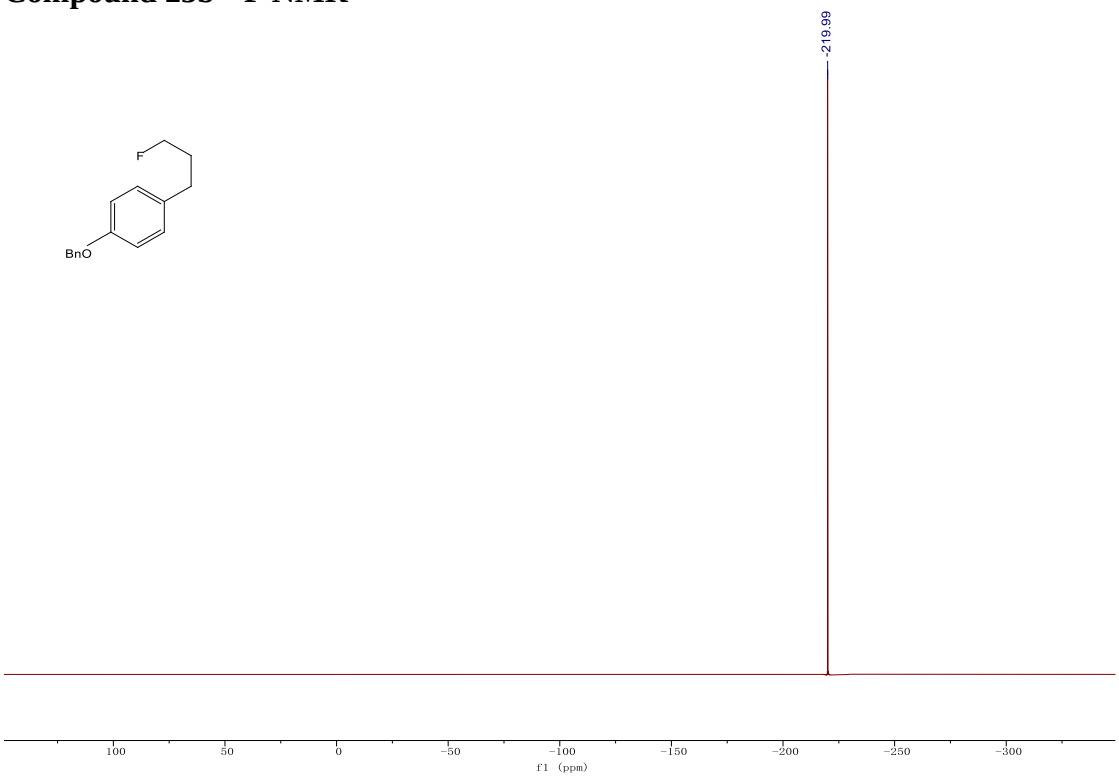
Compound 23b ^{1}H NMR



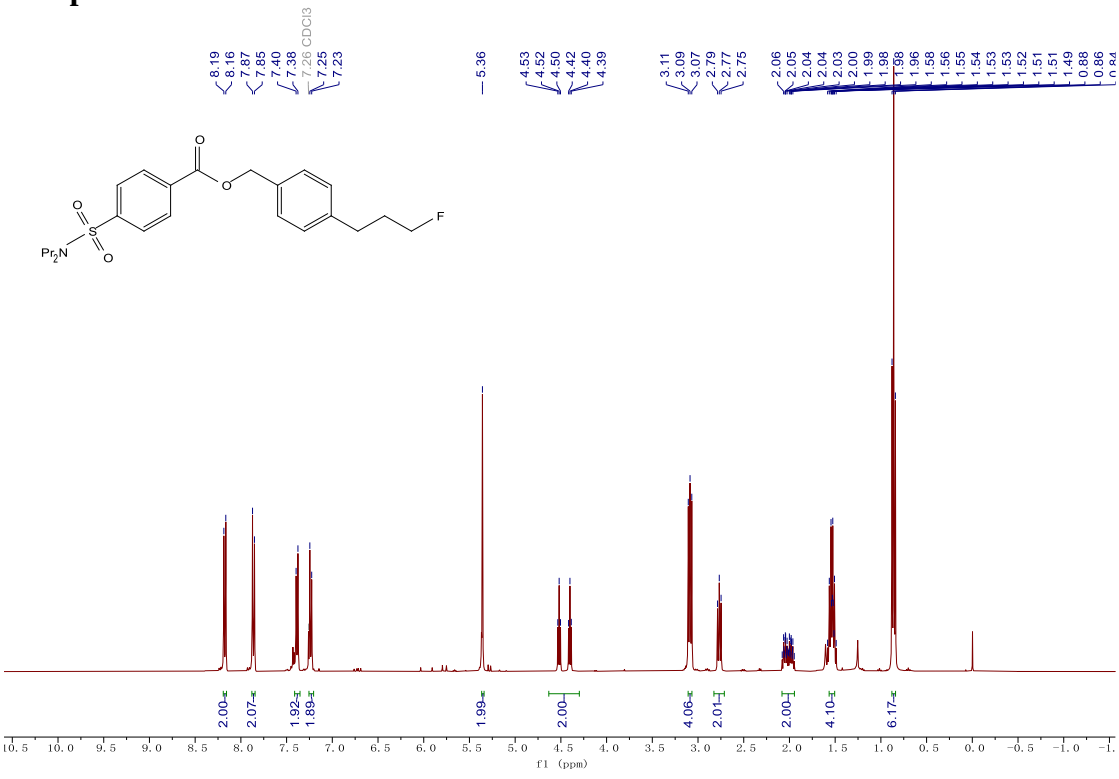
Compound 23b ^{13}C NMR



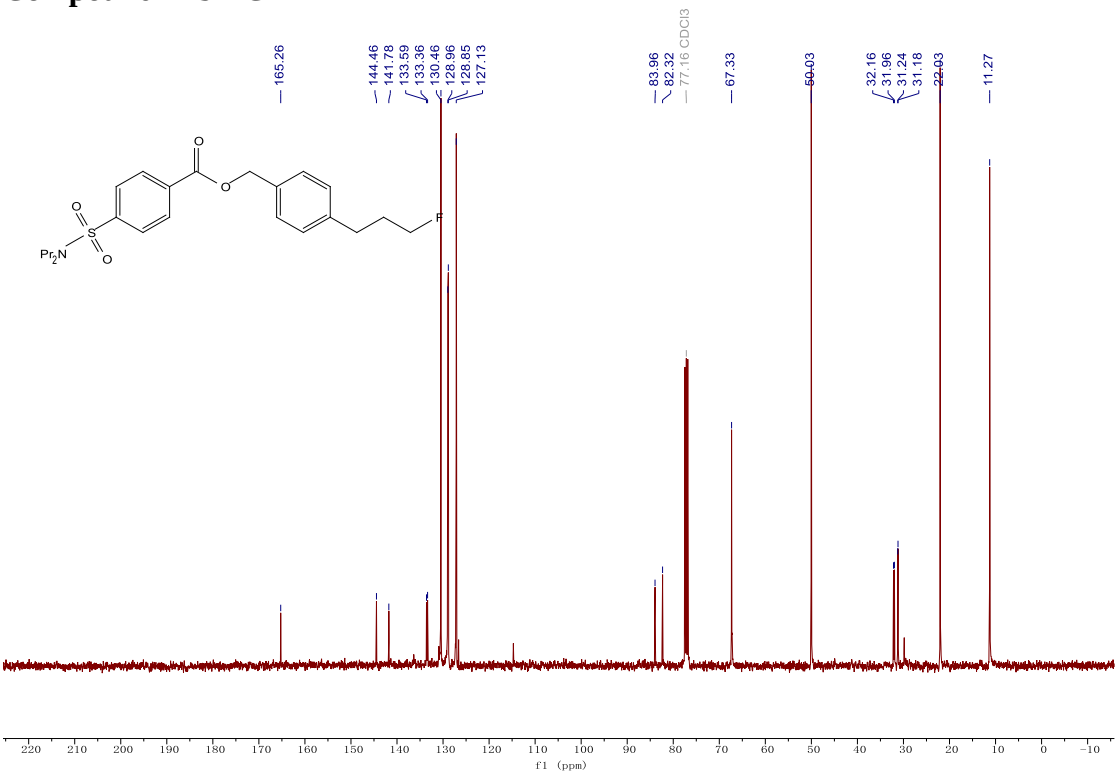
Compound 23b ^{19}F NMR



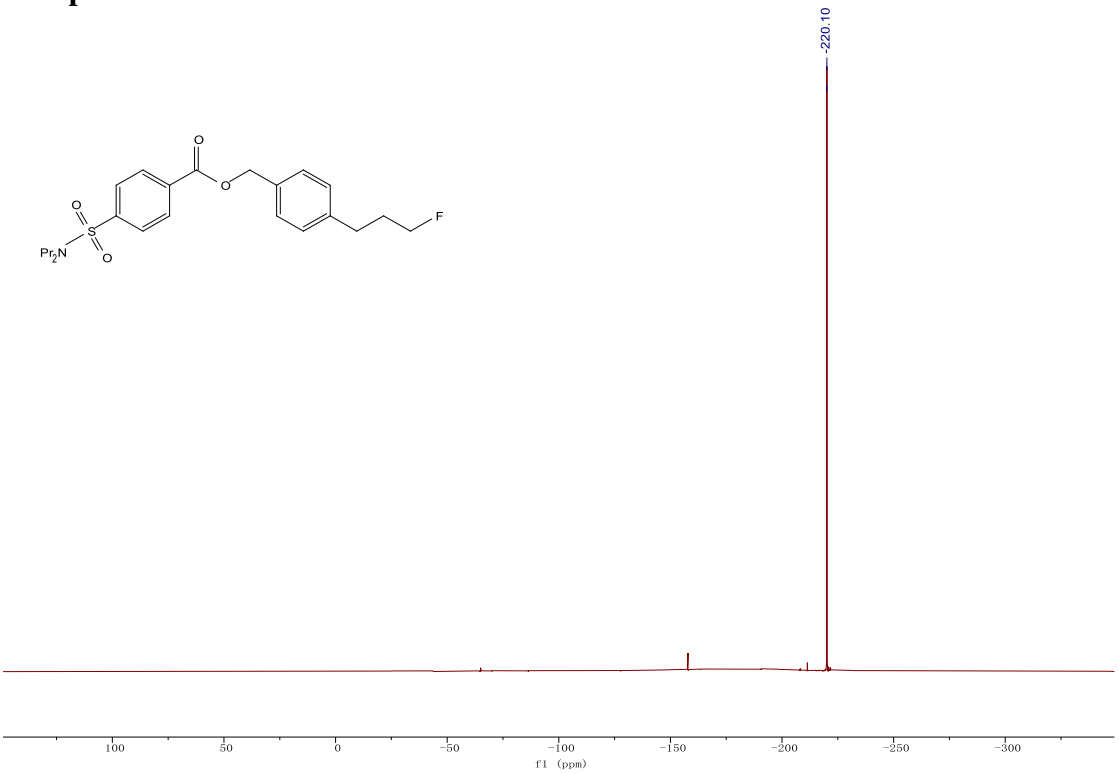
Compound 24b ^{1}H NMR



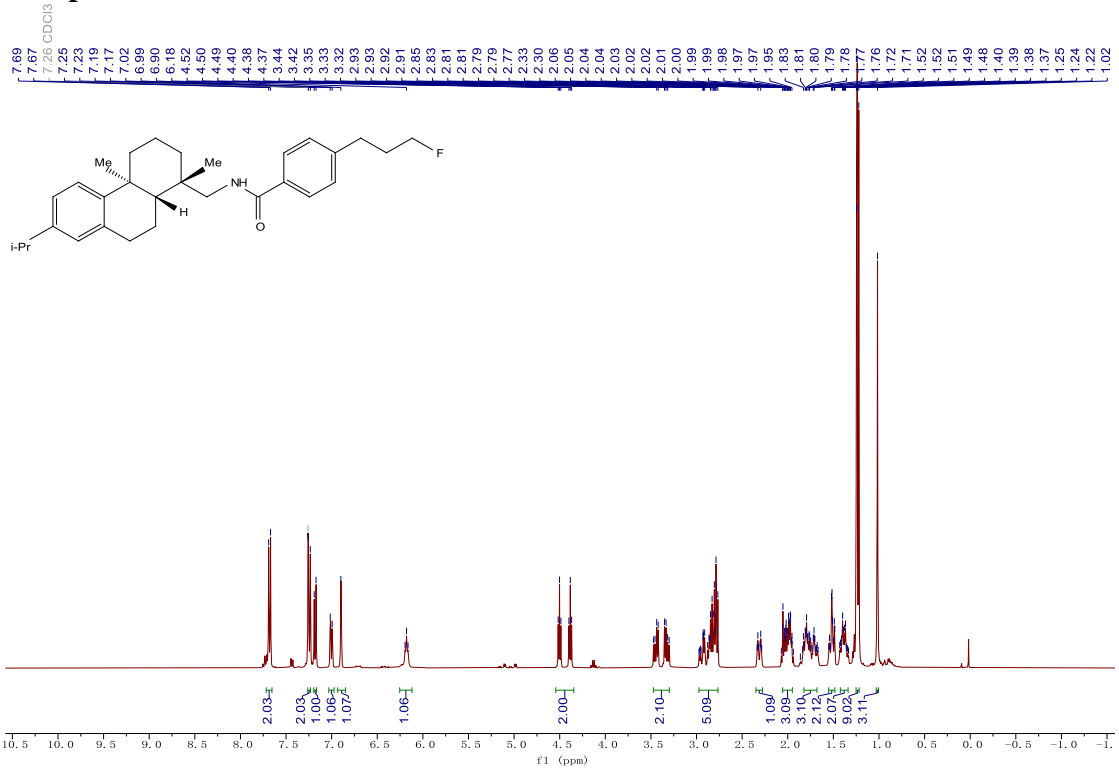
Compound 24b ^{13}C NMR



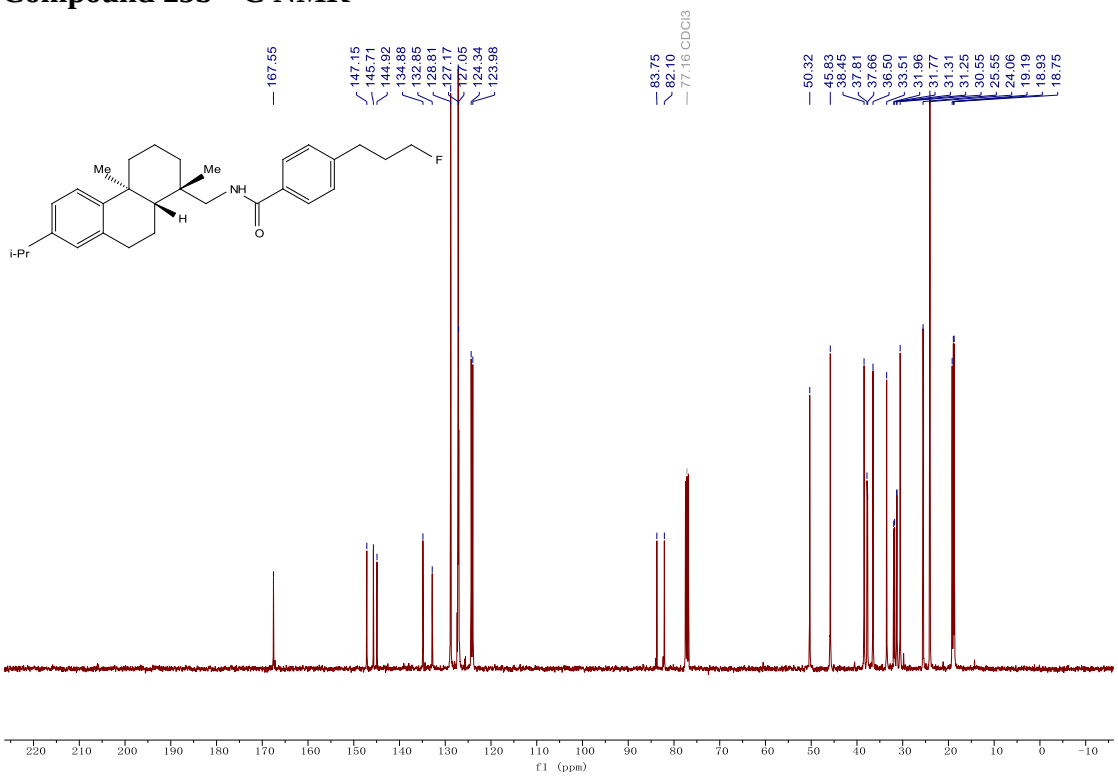
Compound 24b ^{19}F NMR



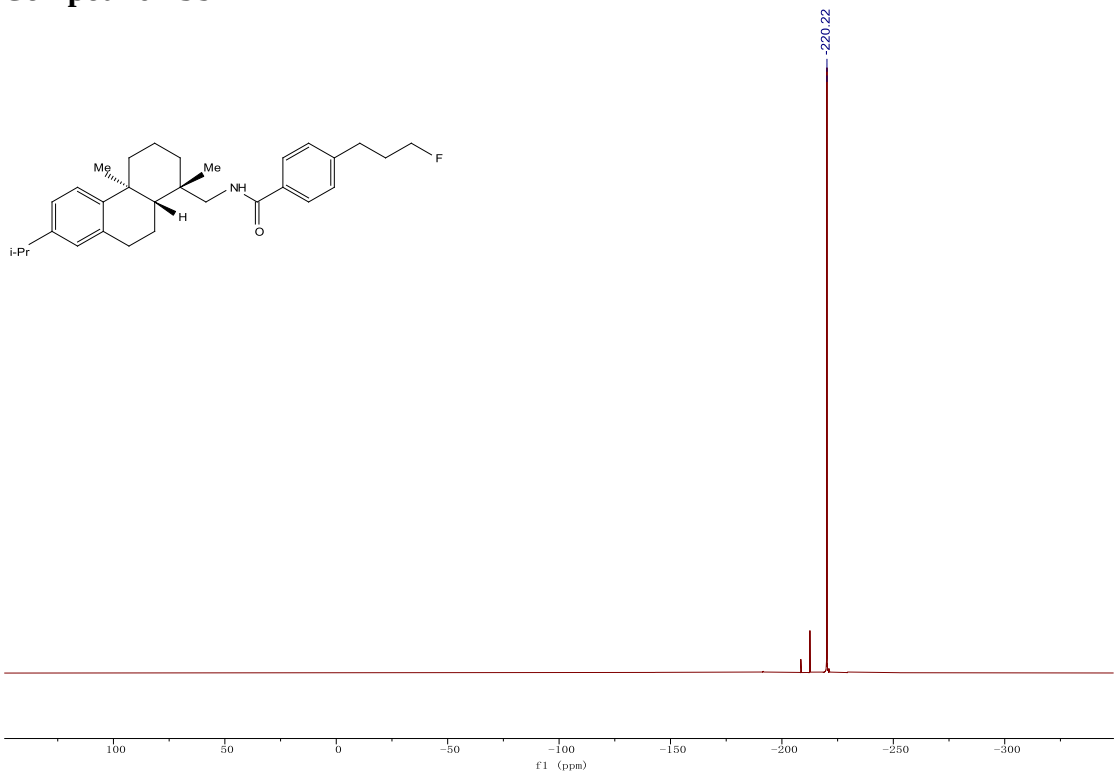
Compound 25b ^{1}H NMR



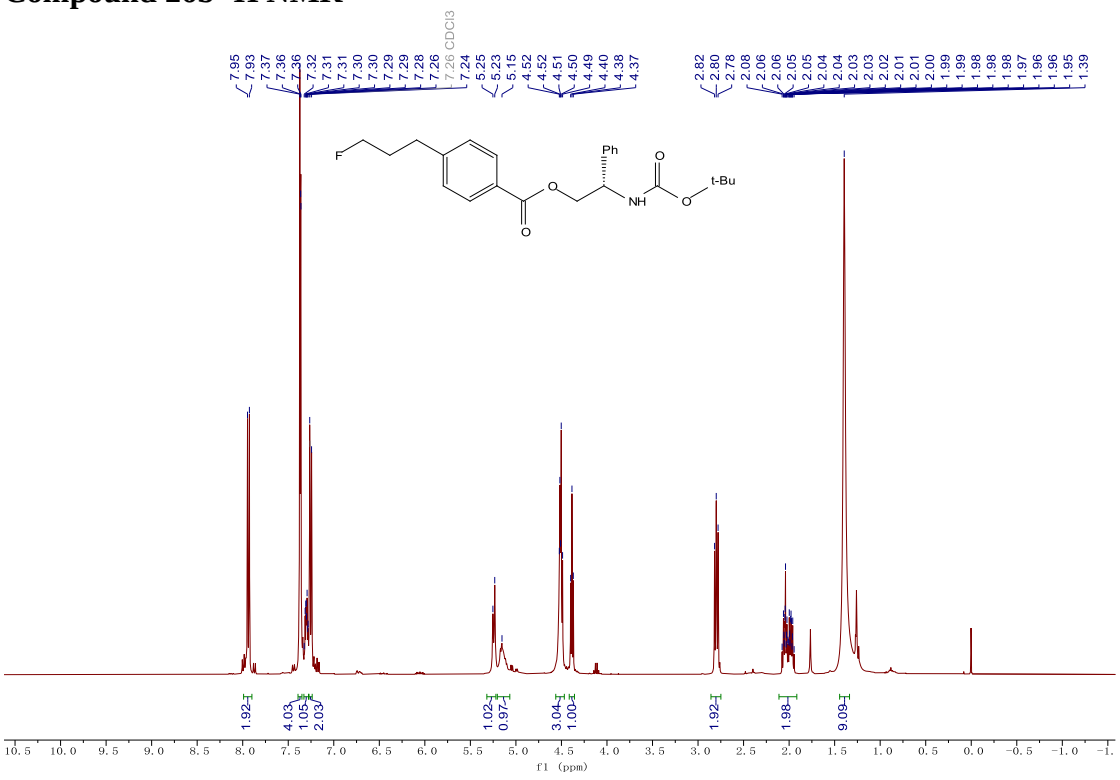
Compound 25b ^{13}C NMR



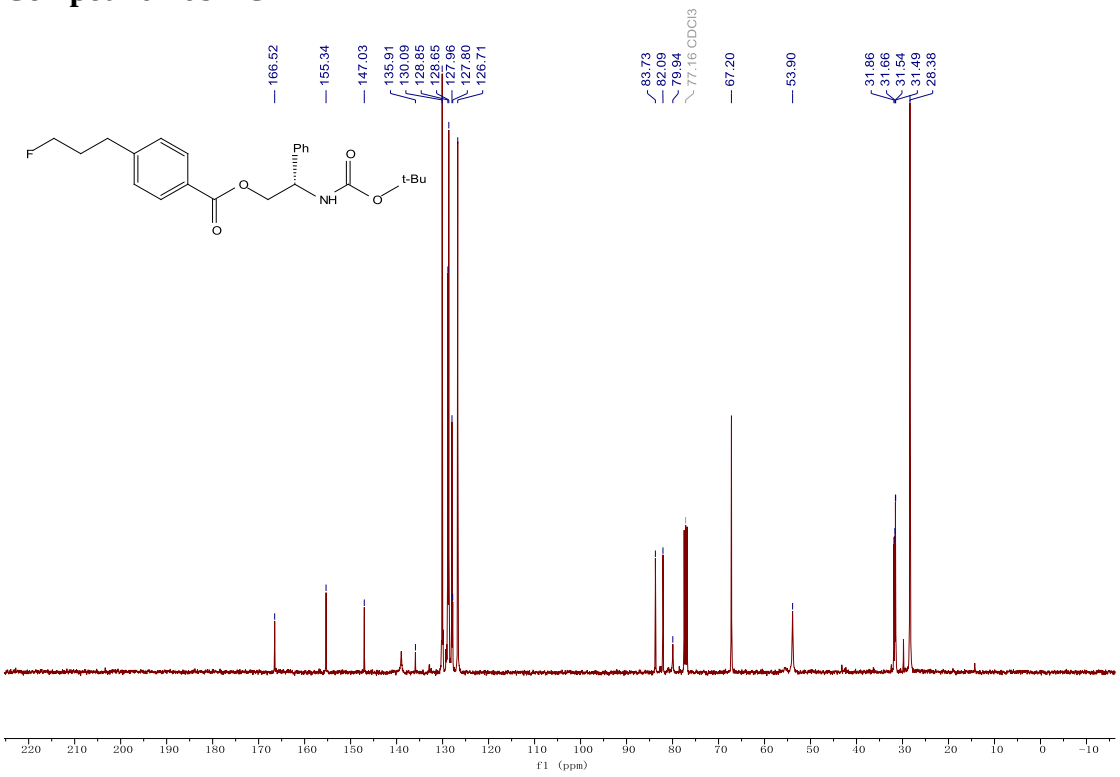
Compound 25b ^{19}F NMR



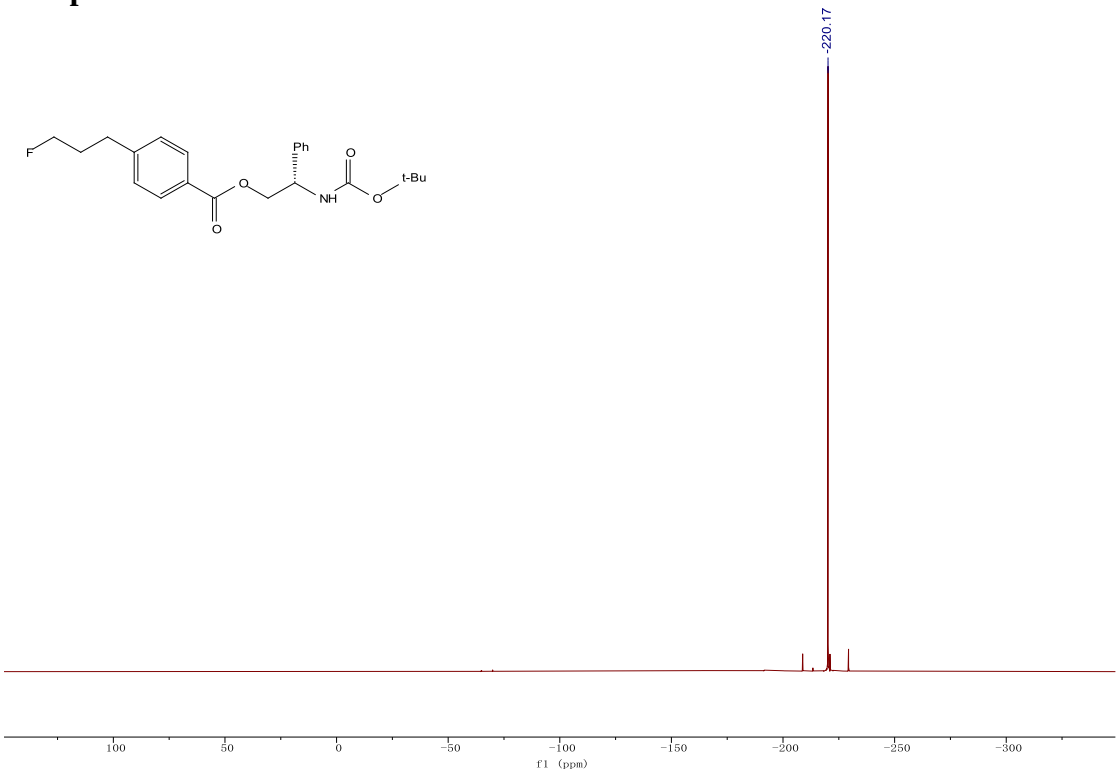
Compound 26b ^{1}H NMR



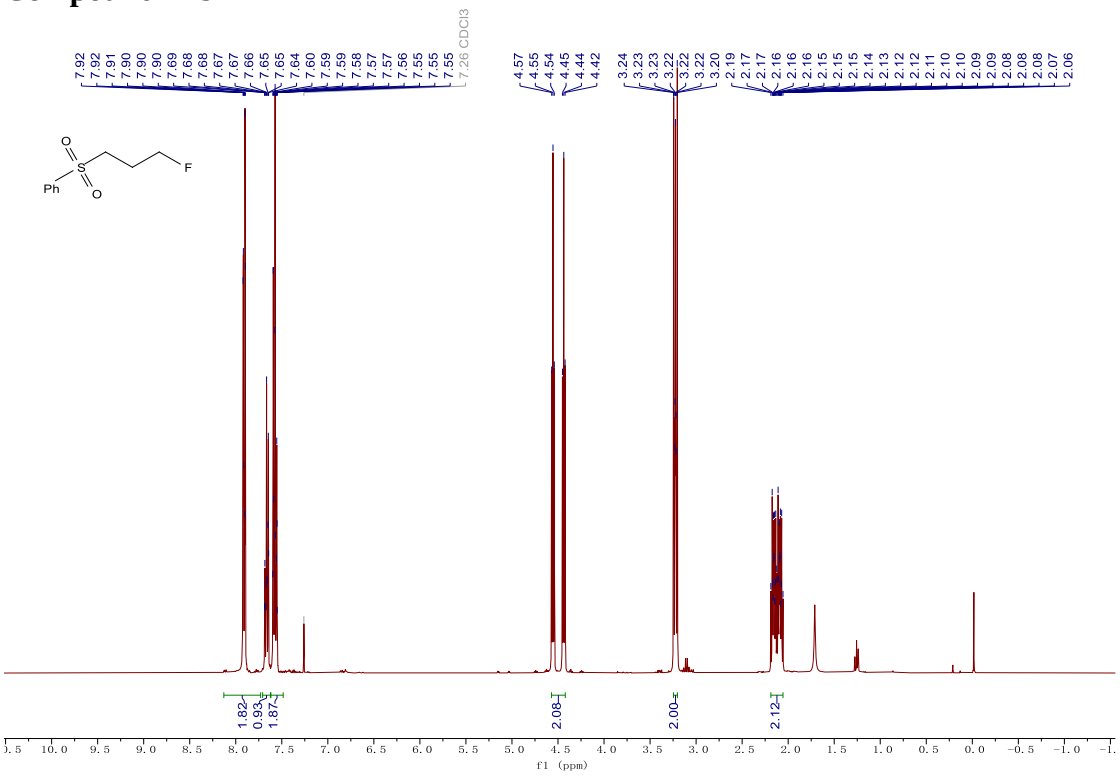
Compound 26b ^{13}C NMR



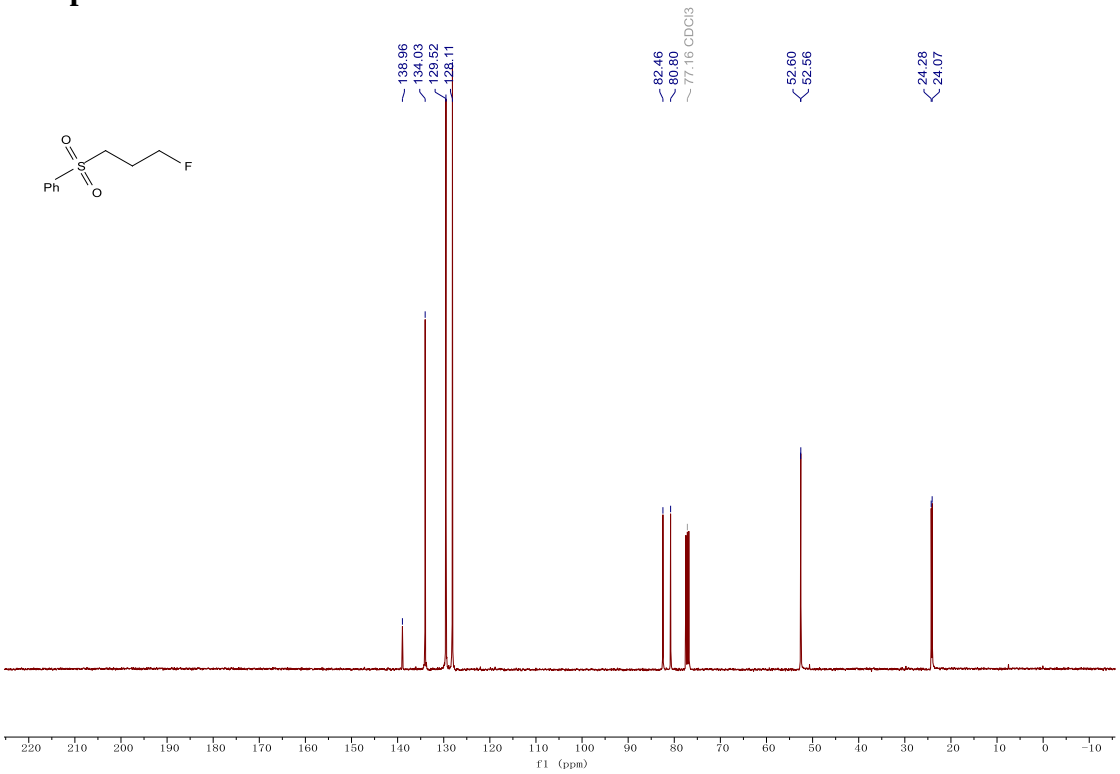
Compound 26b ^{19}F NMR



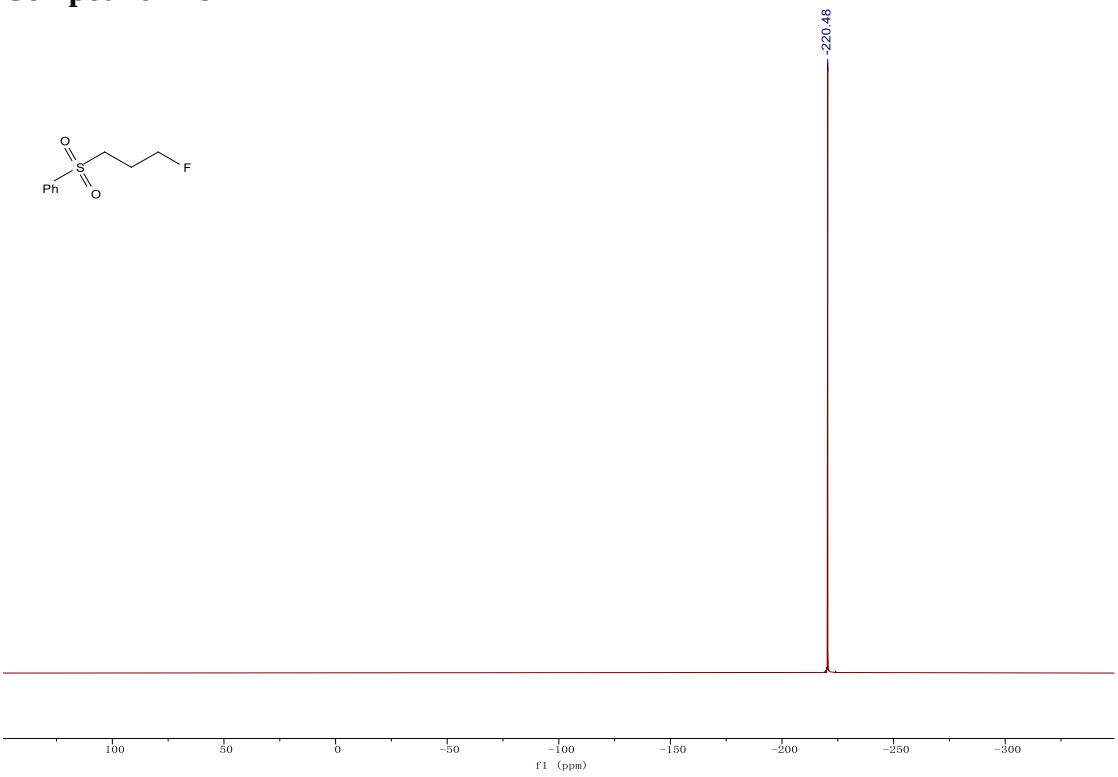
Compound 27b ^{1}H NMR



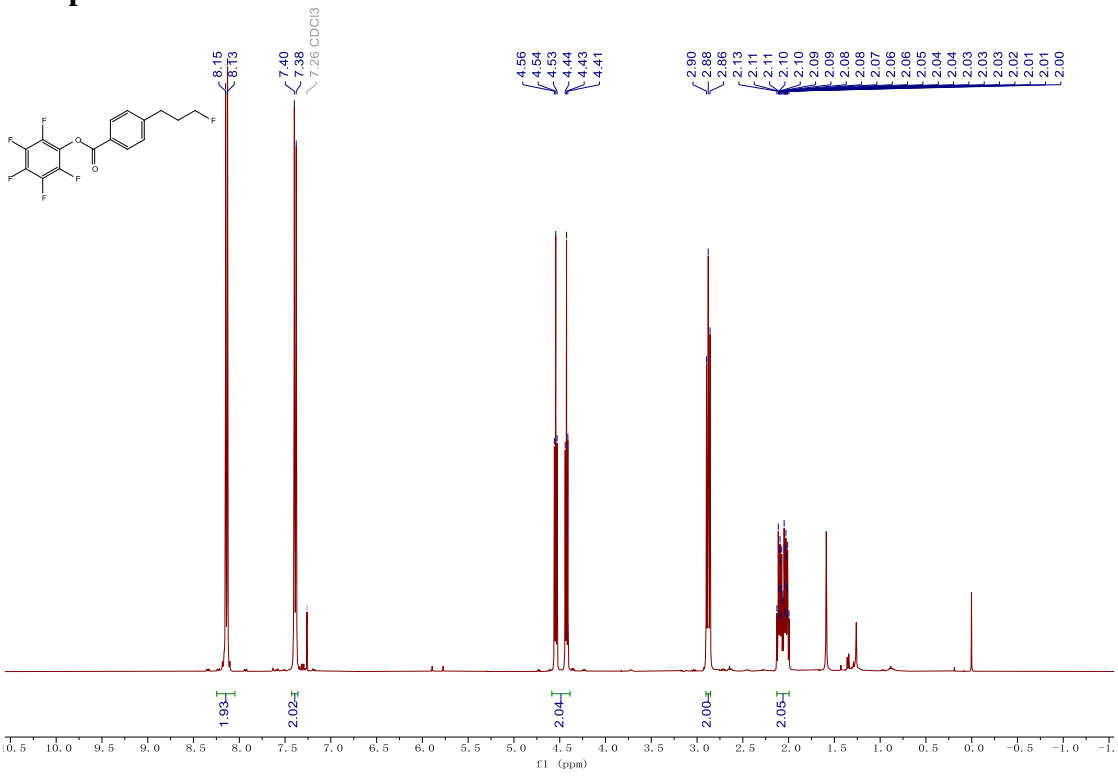
Compound 27b ^{13}C NMR



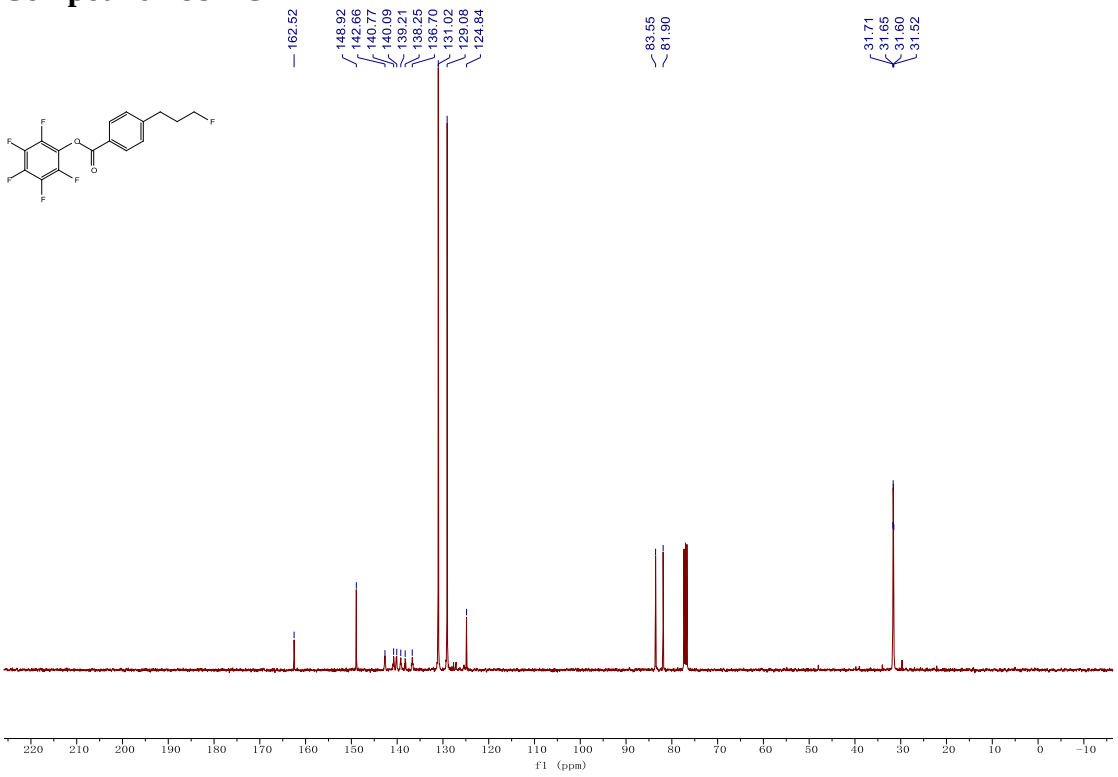
Compound 27b ^{19}F NMR



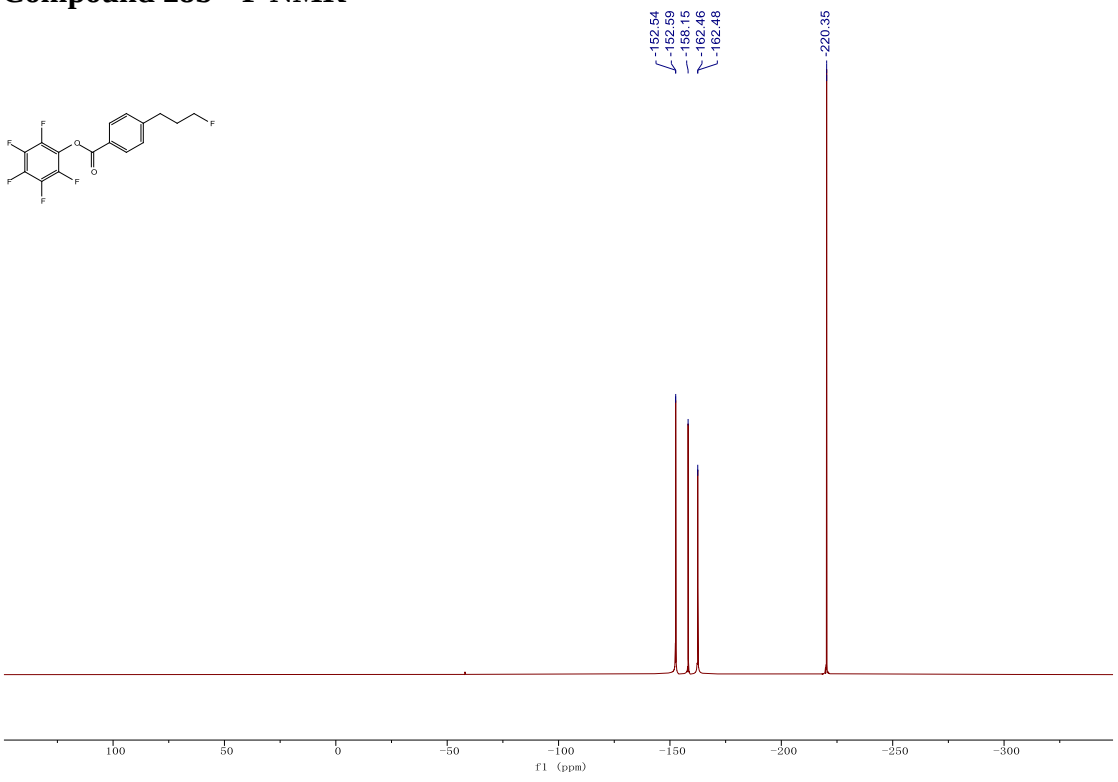
Compound 28b ^{1}H NMR



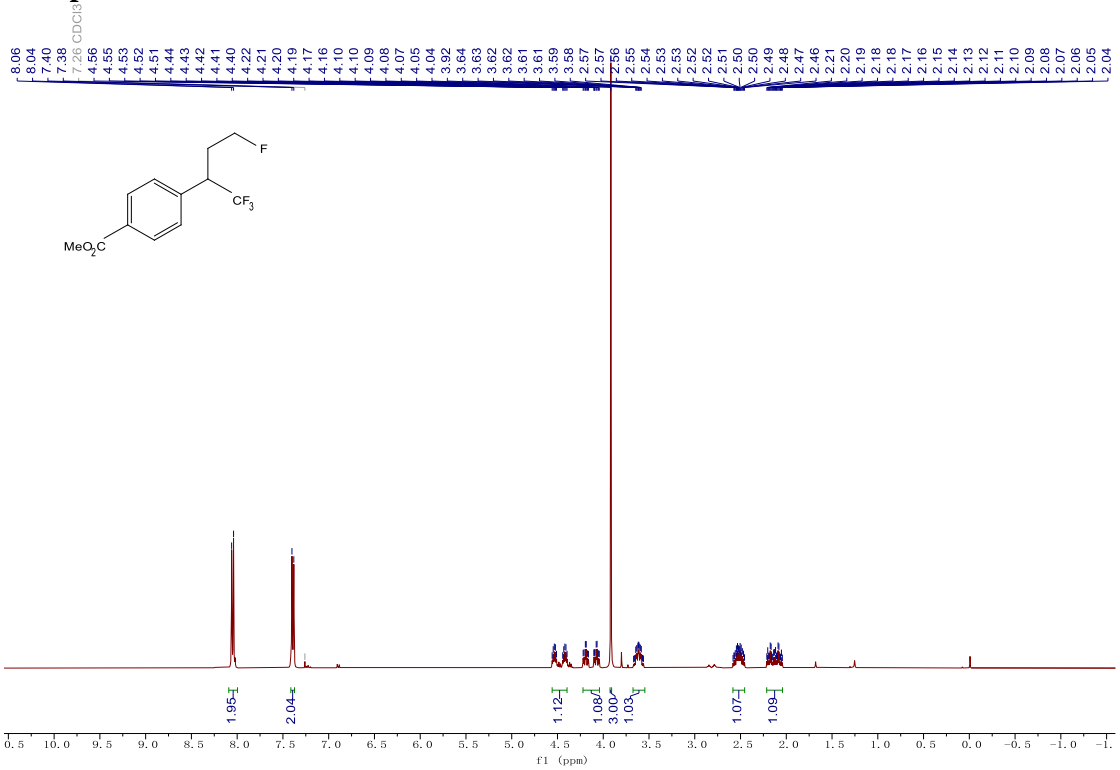
Compound 28b ^{13}C NMR



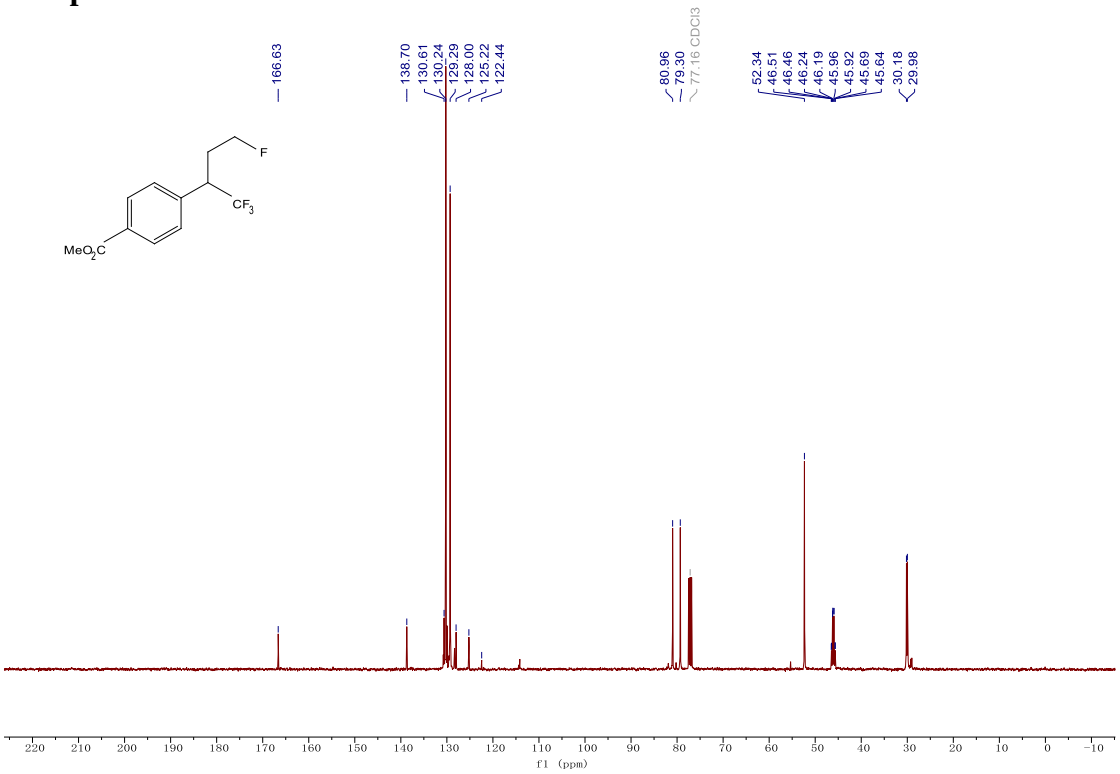
Compound 28b ^{19}F NMR



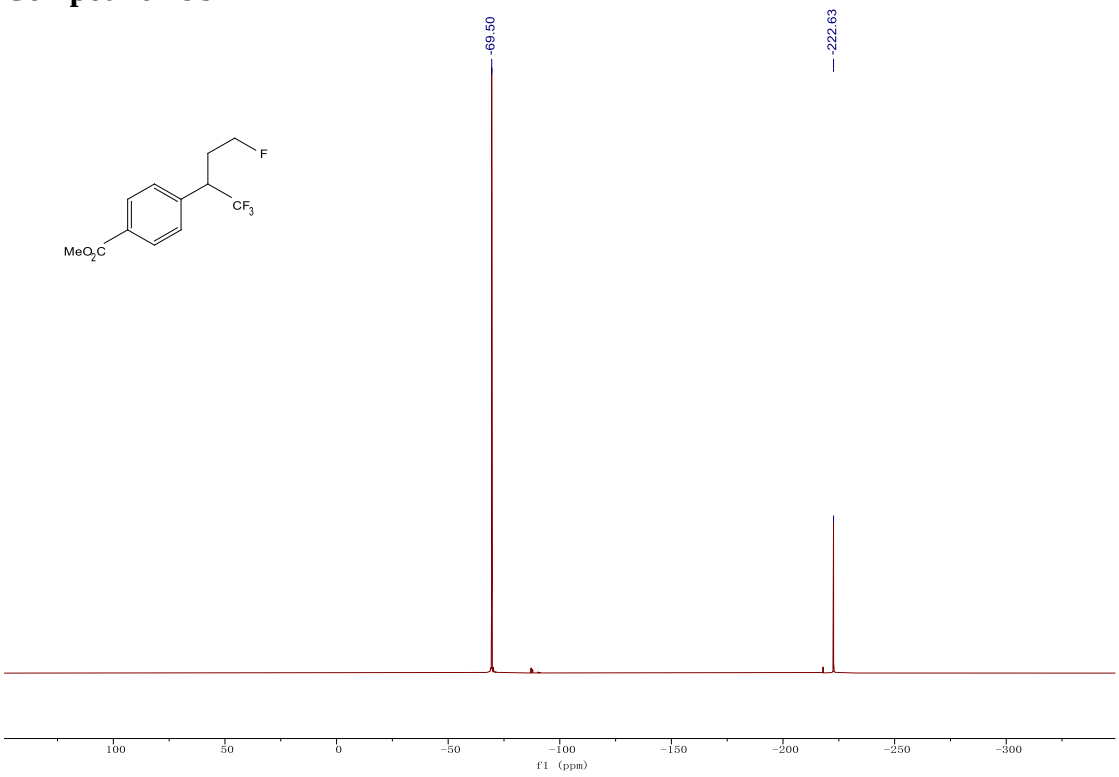
Compound 29b ^{1}H NMR



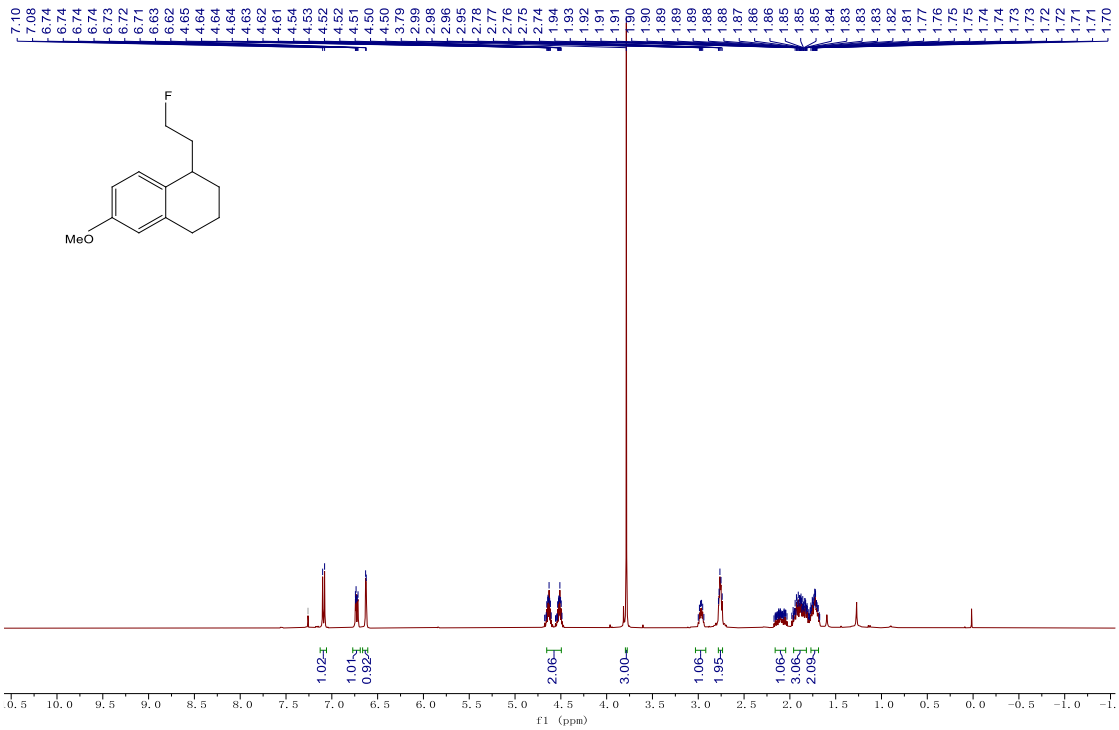
Compound 29b ^{13}C NMR



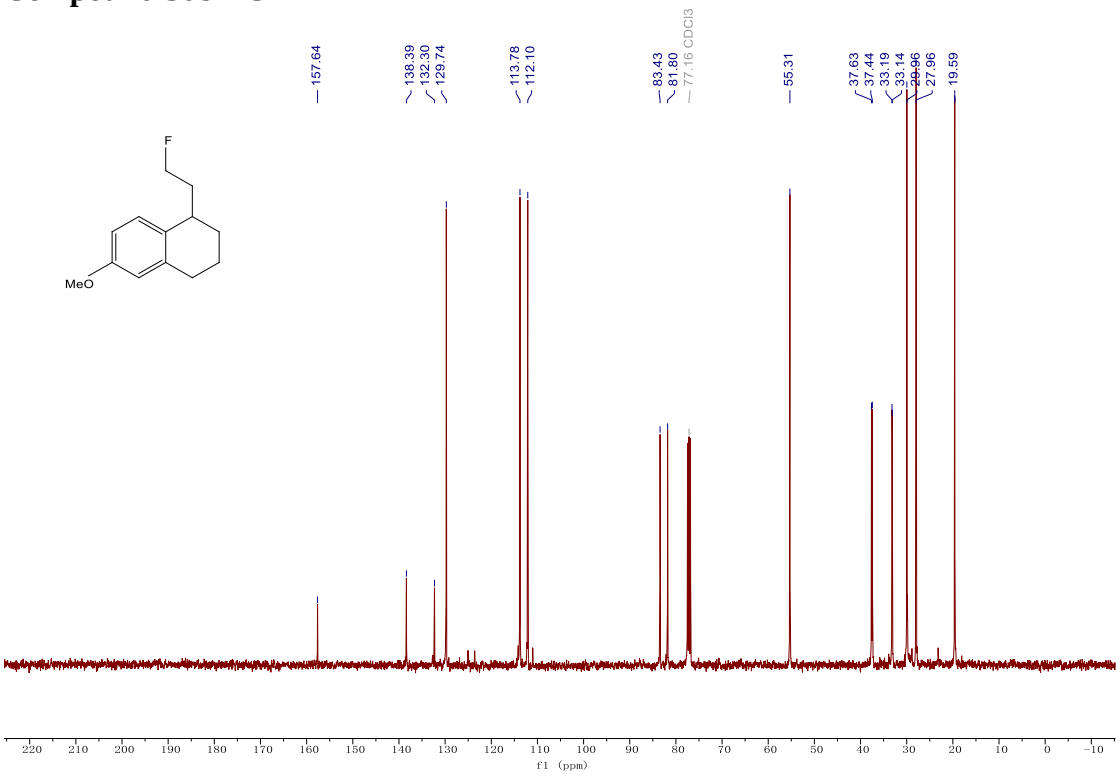
Compound 29b ^{19}F NMR



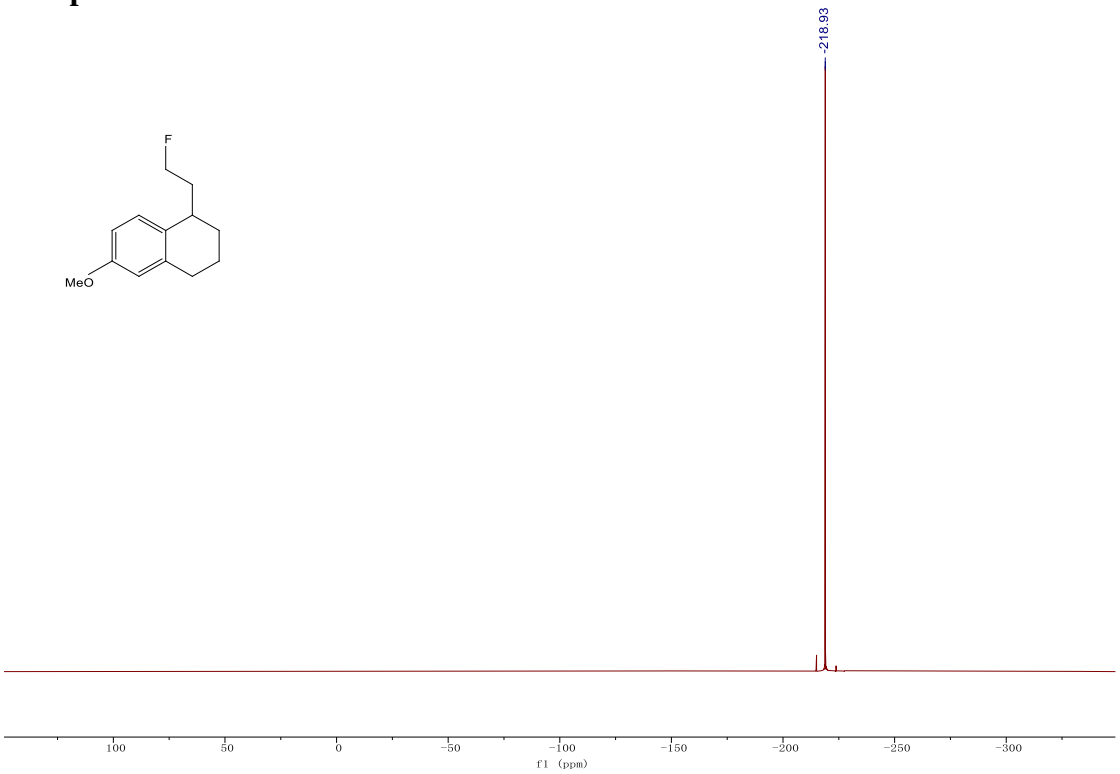
Compound 30b ^{1}H NMR



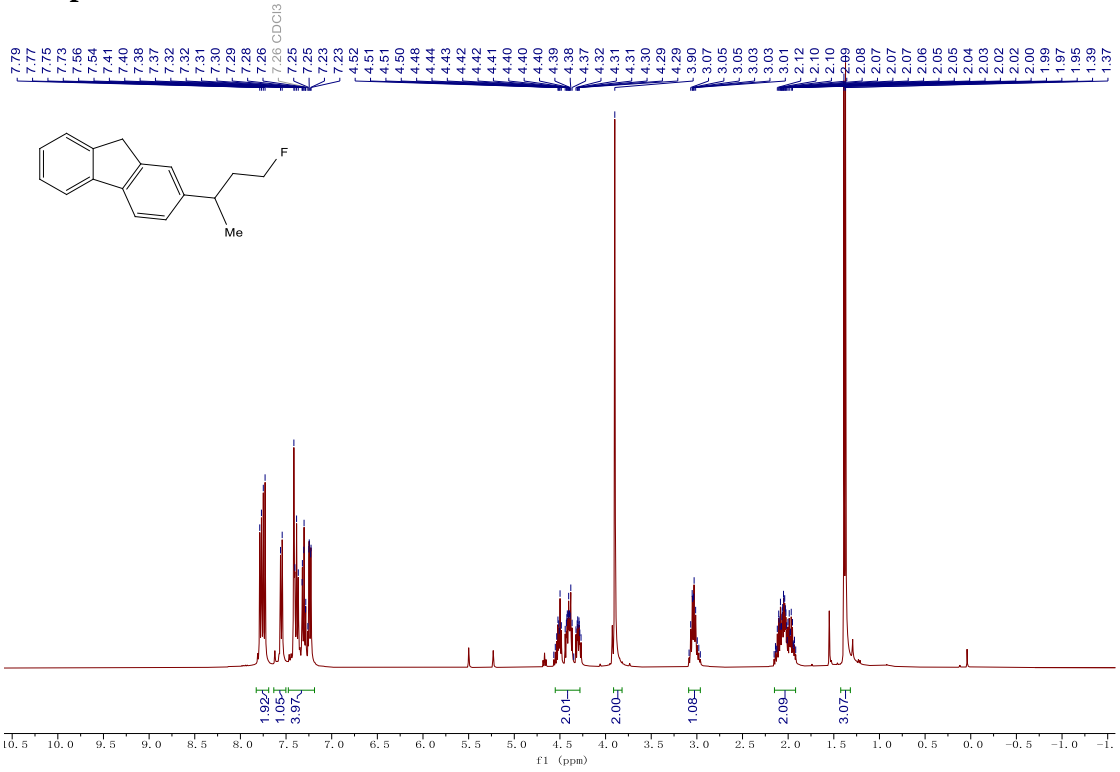
Compound 30b ^{13}C NMR



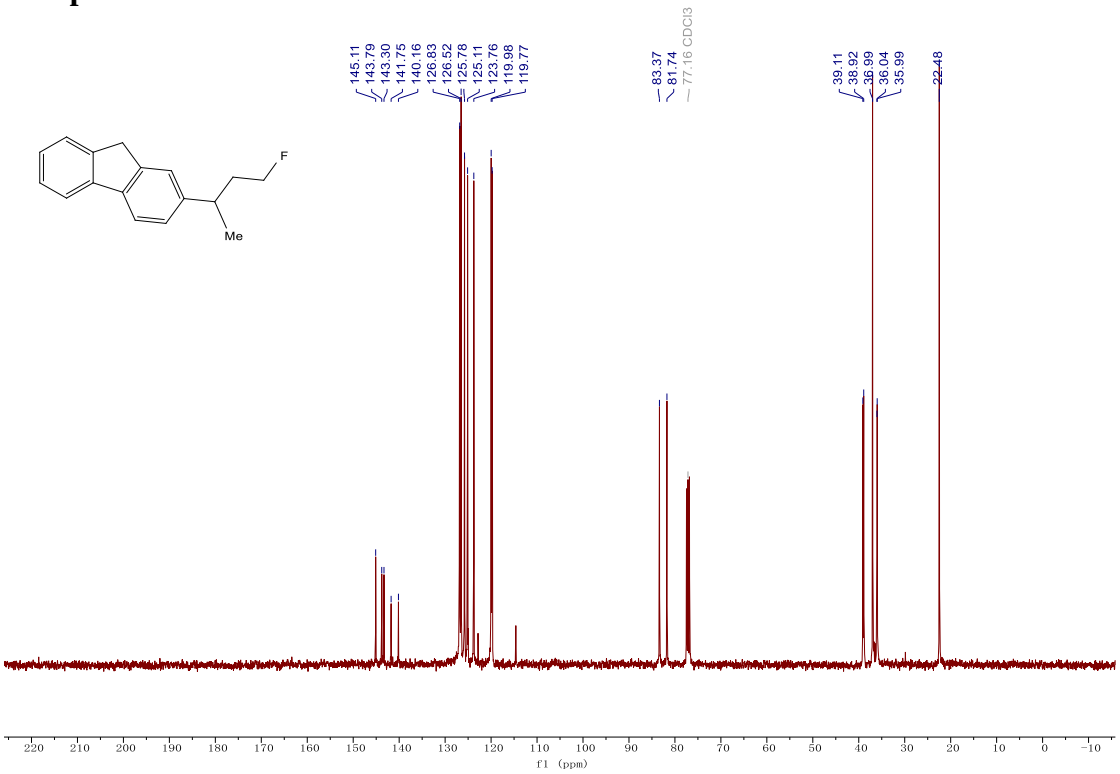
Compound 30b ^{19}F NMR



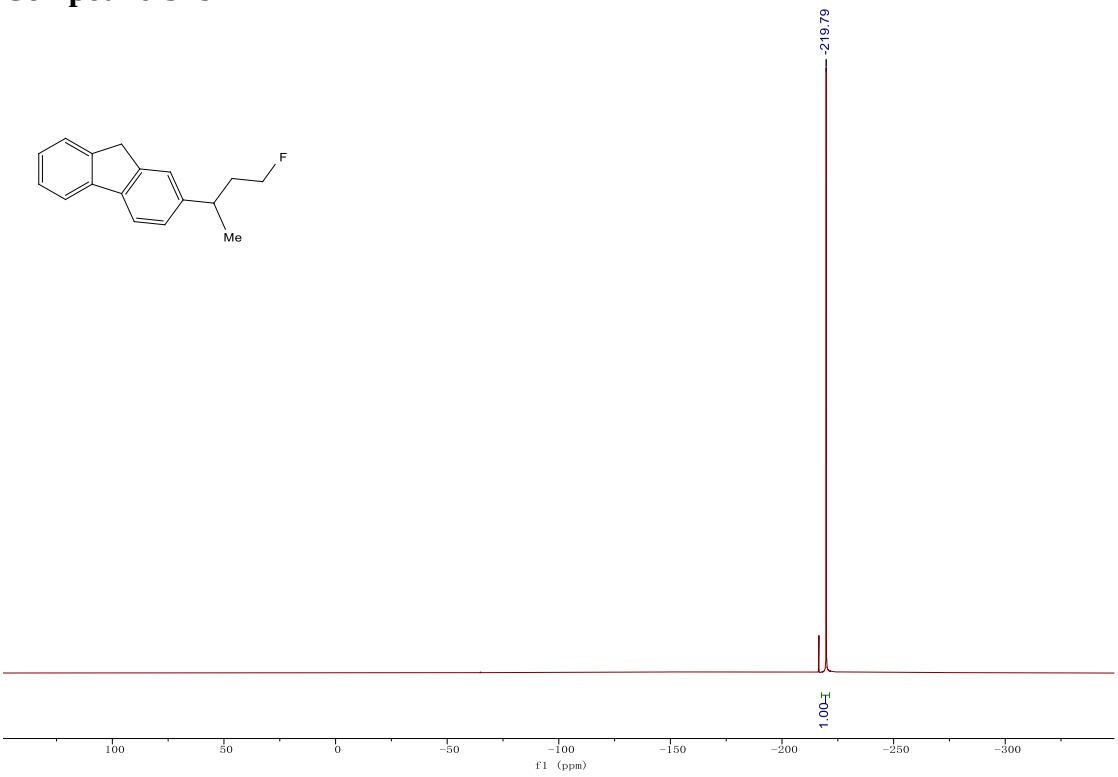
Compound 31b ^{1}H NMR



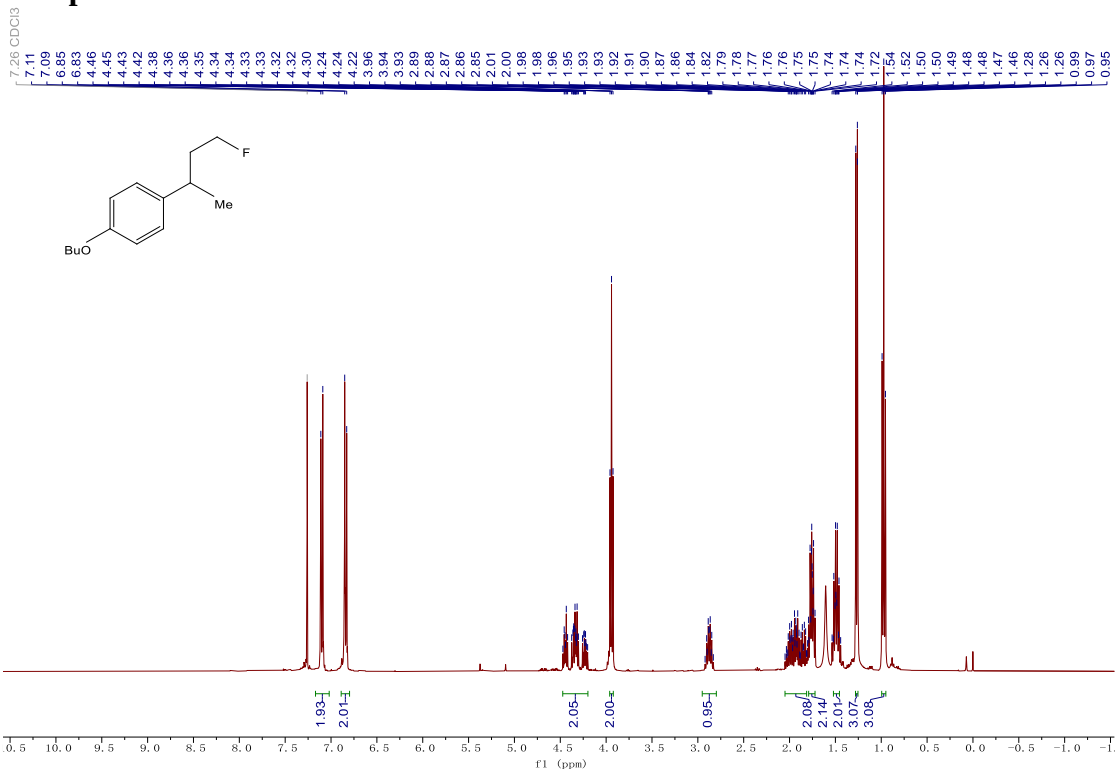
Compound 31b ^{13}C NMR



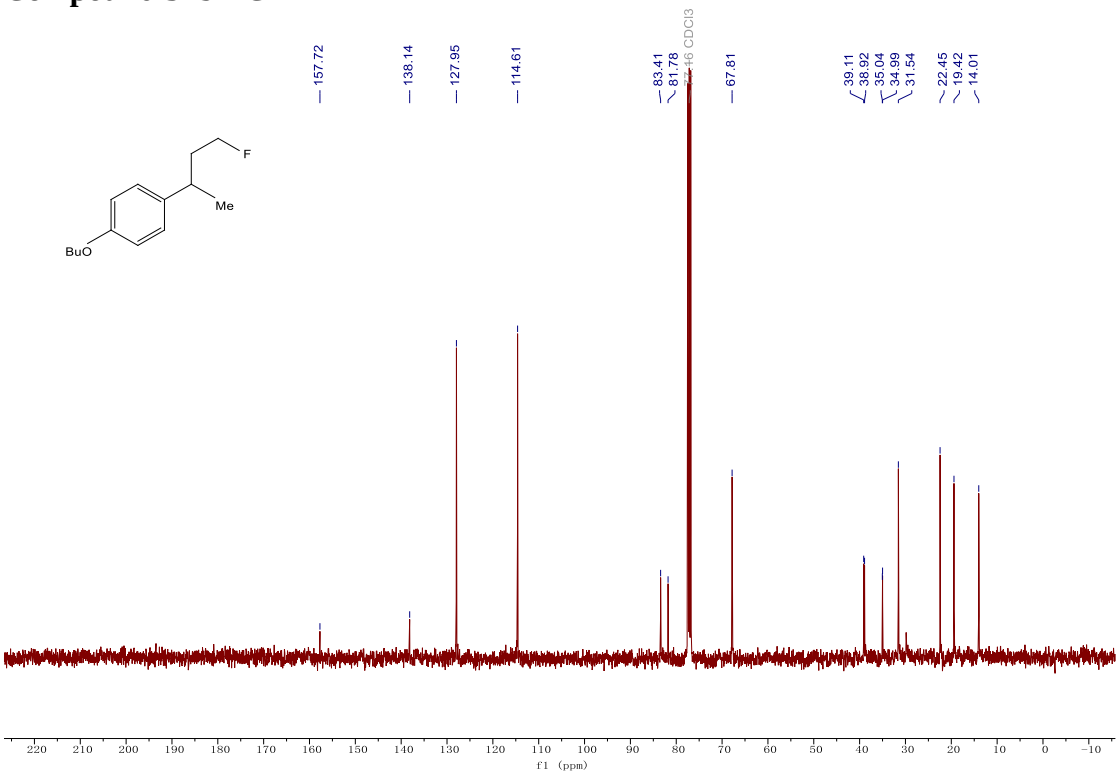
Compound 31b ^{19}F NMR



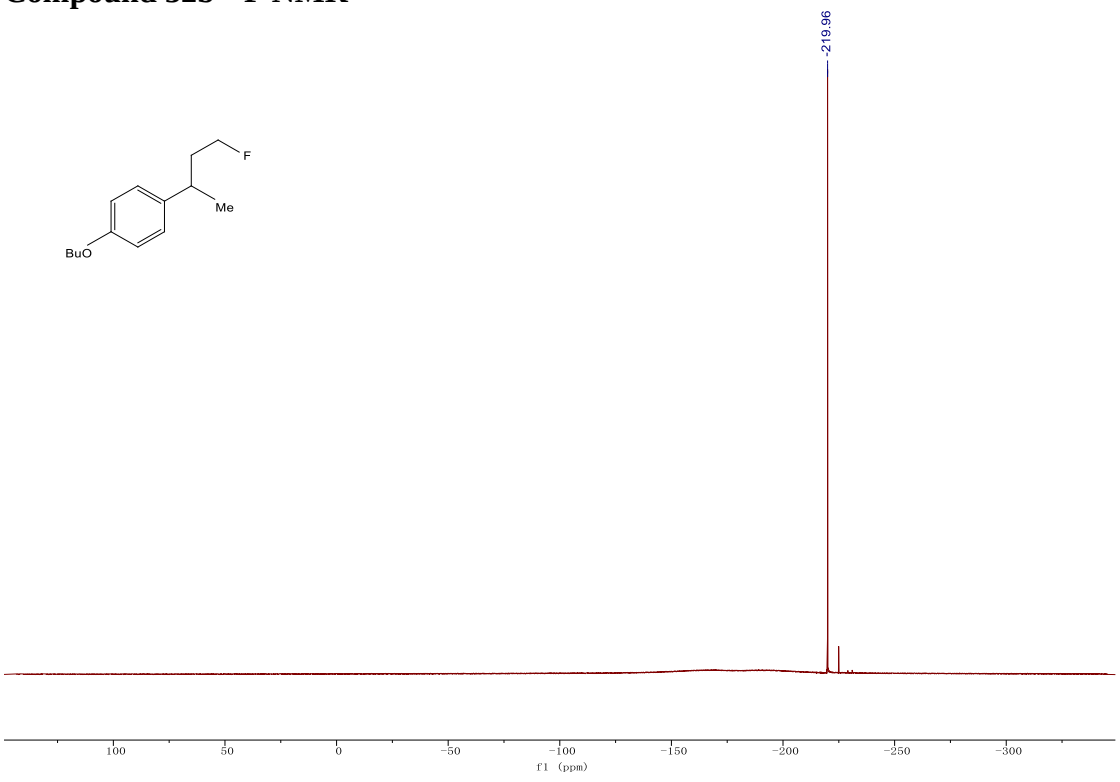
Compound 32b ^{1}H NMR



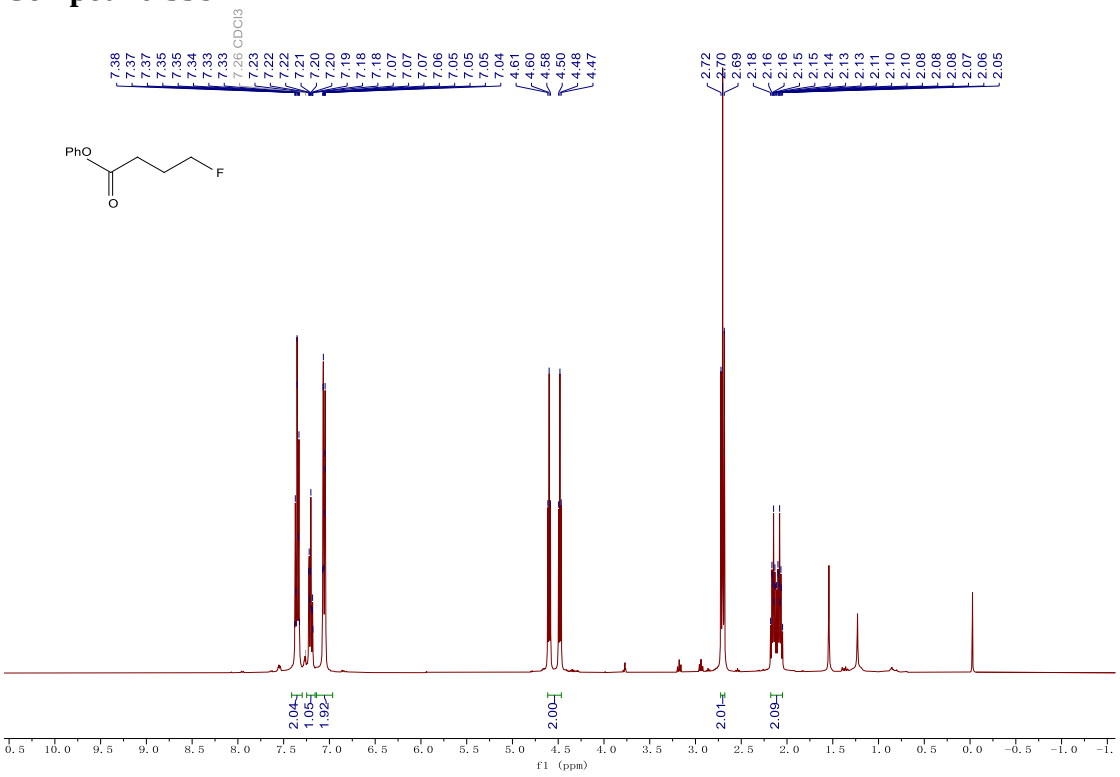
Compound 32b ^{13}C NMR



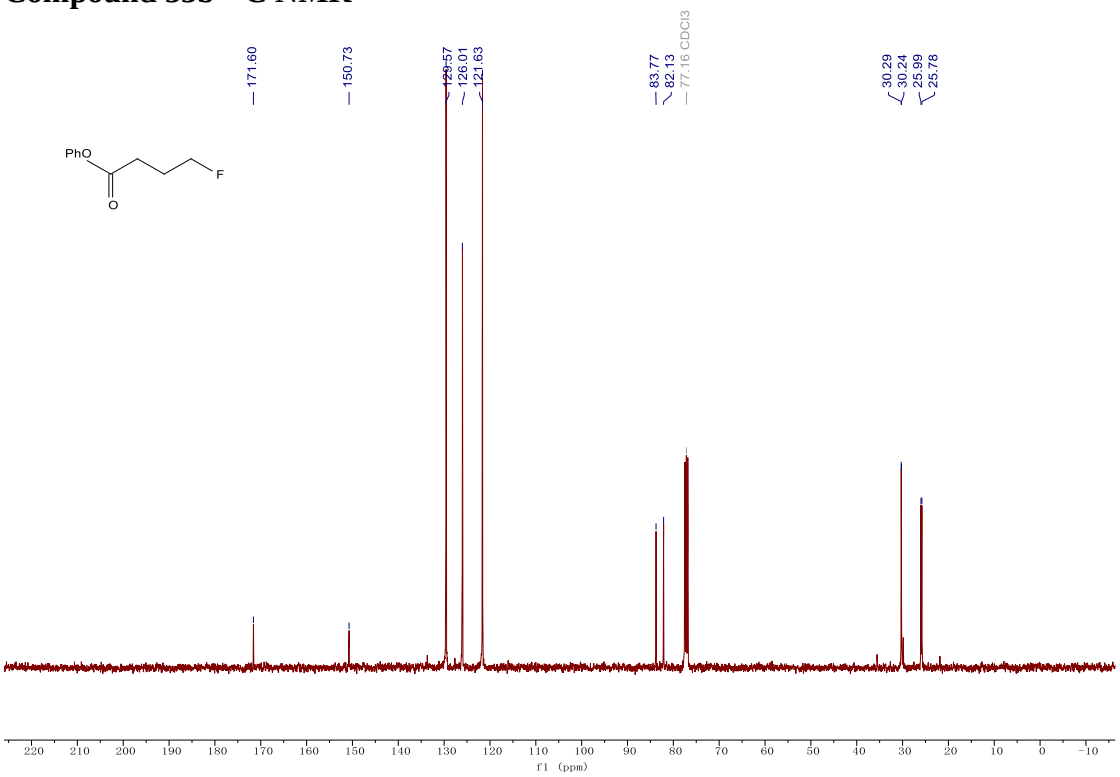
Compound 32b ^{19}F NMR



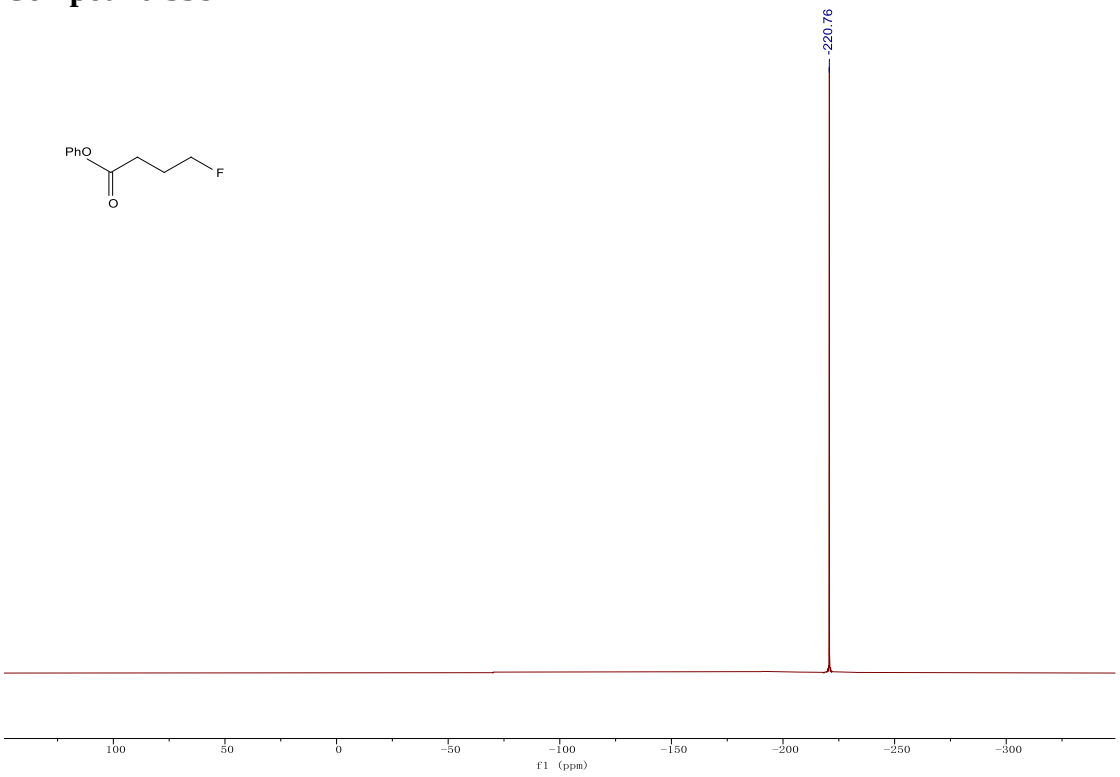
Compound 33b ^{1}H NMR



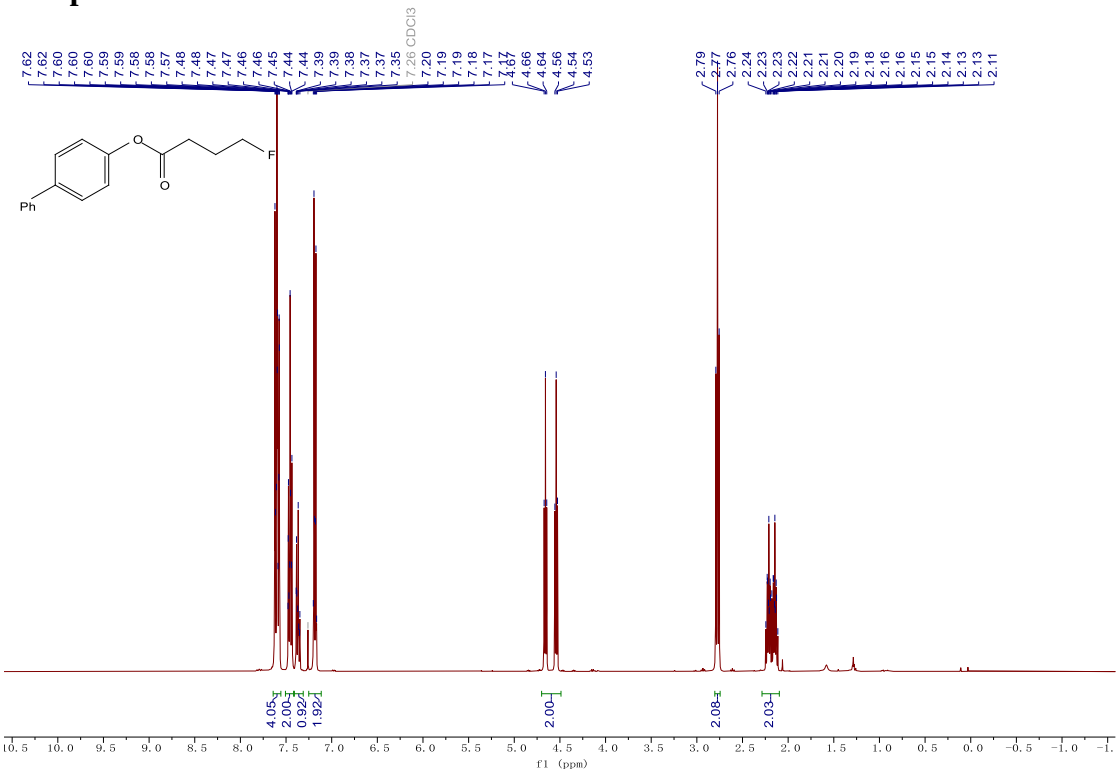
Compound 33b ^{13}C NMR



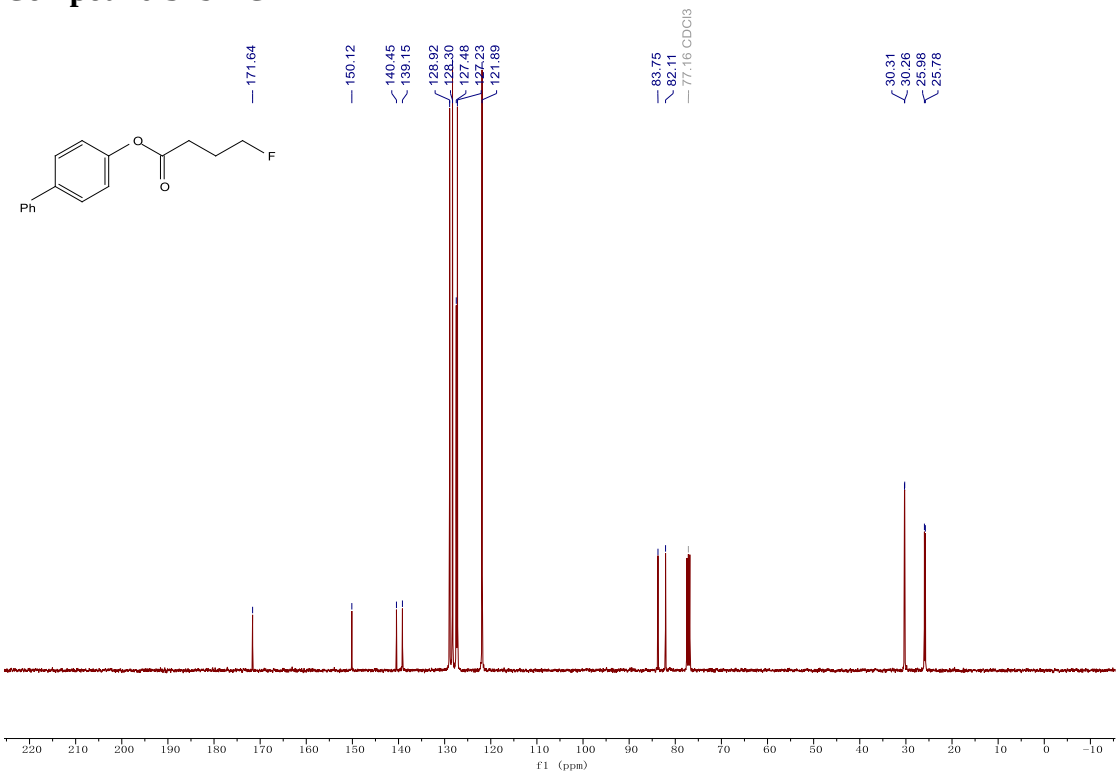
Compound 33b ^{19}F NMR



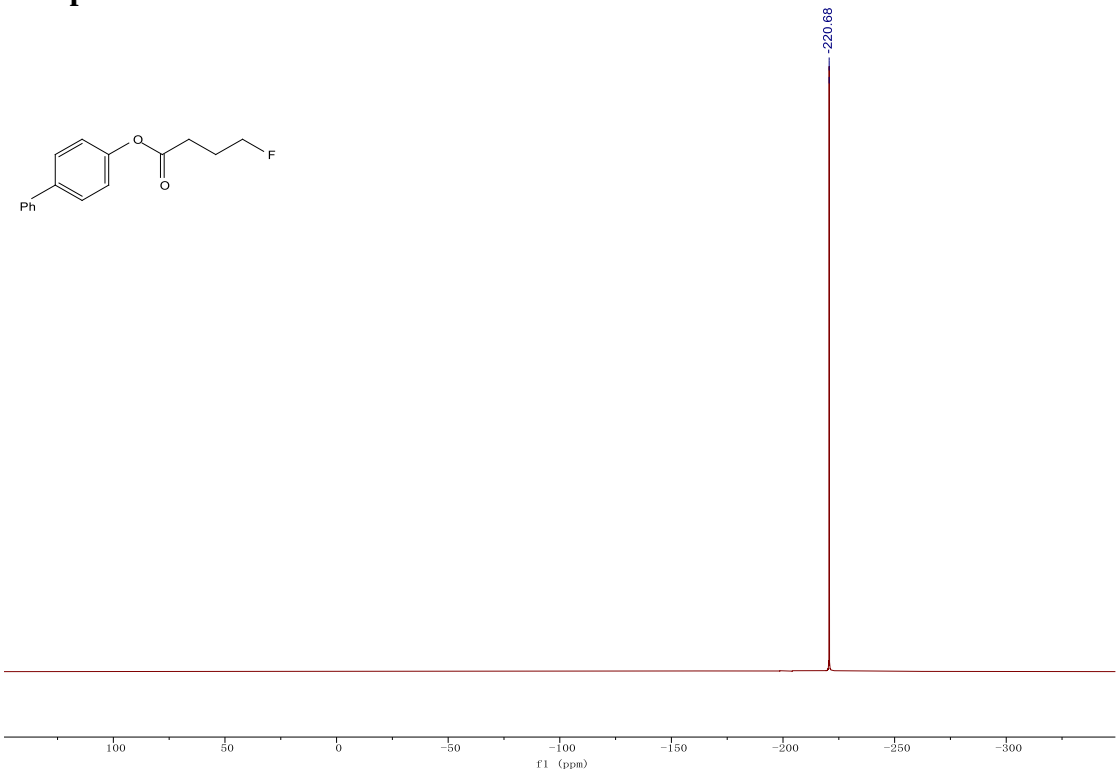
Compound 34b ^{1}H NMR



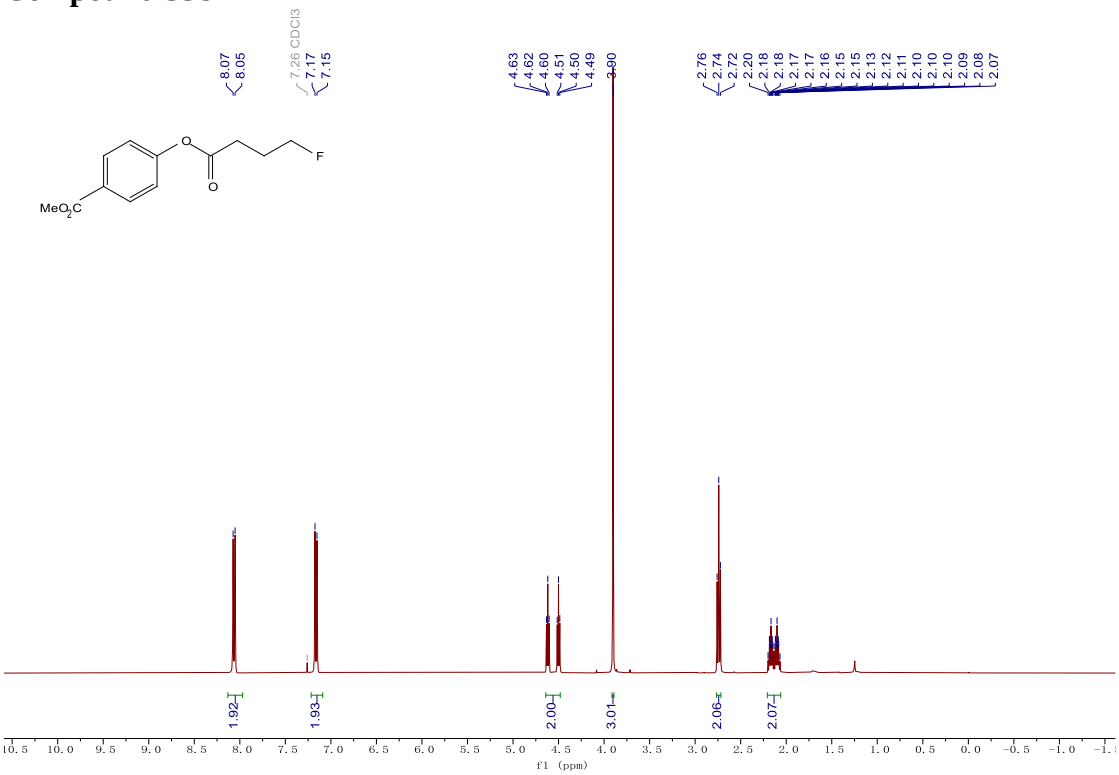
Compound 34b ^{13}C NMR



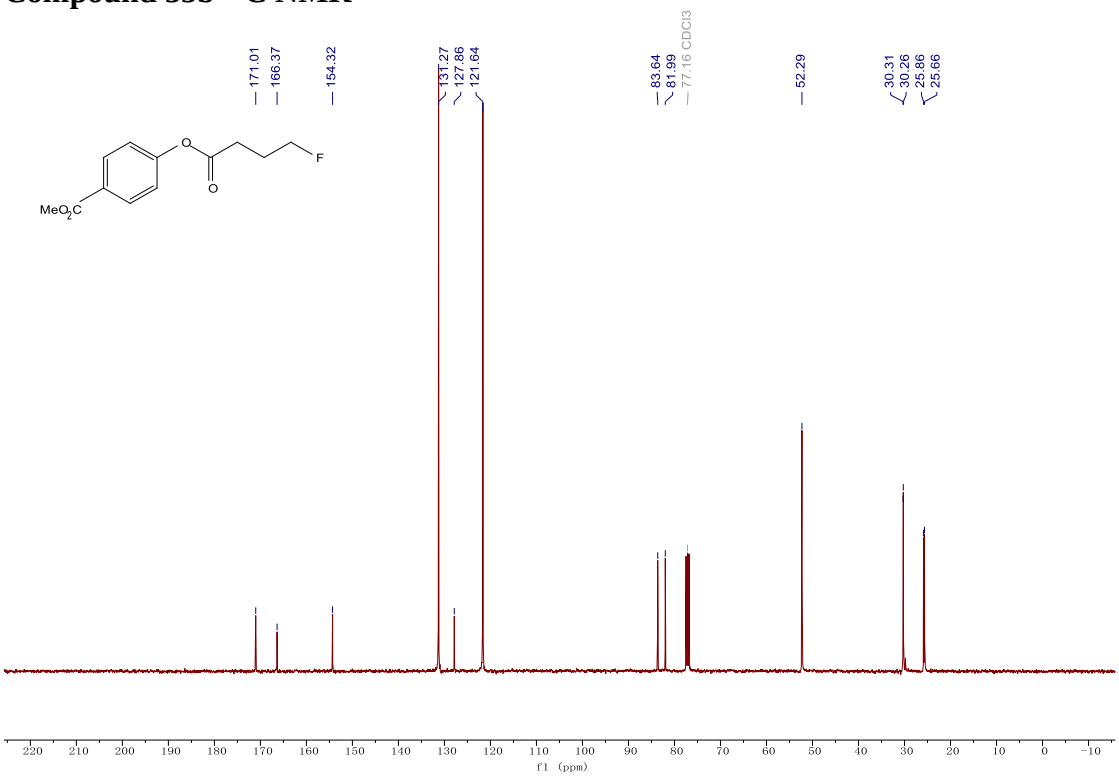
Compound 34b ^{19}F NMR



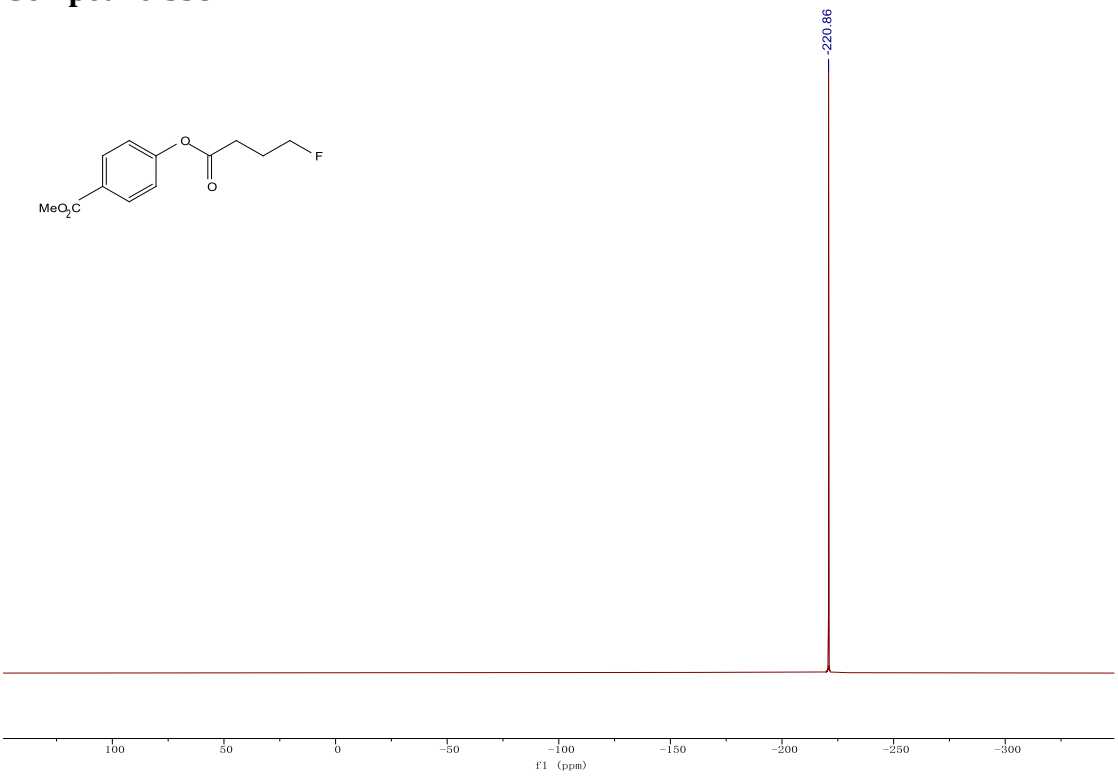
Compound 35b ^{1}H NMR



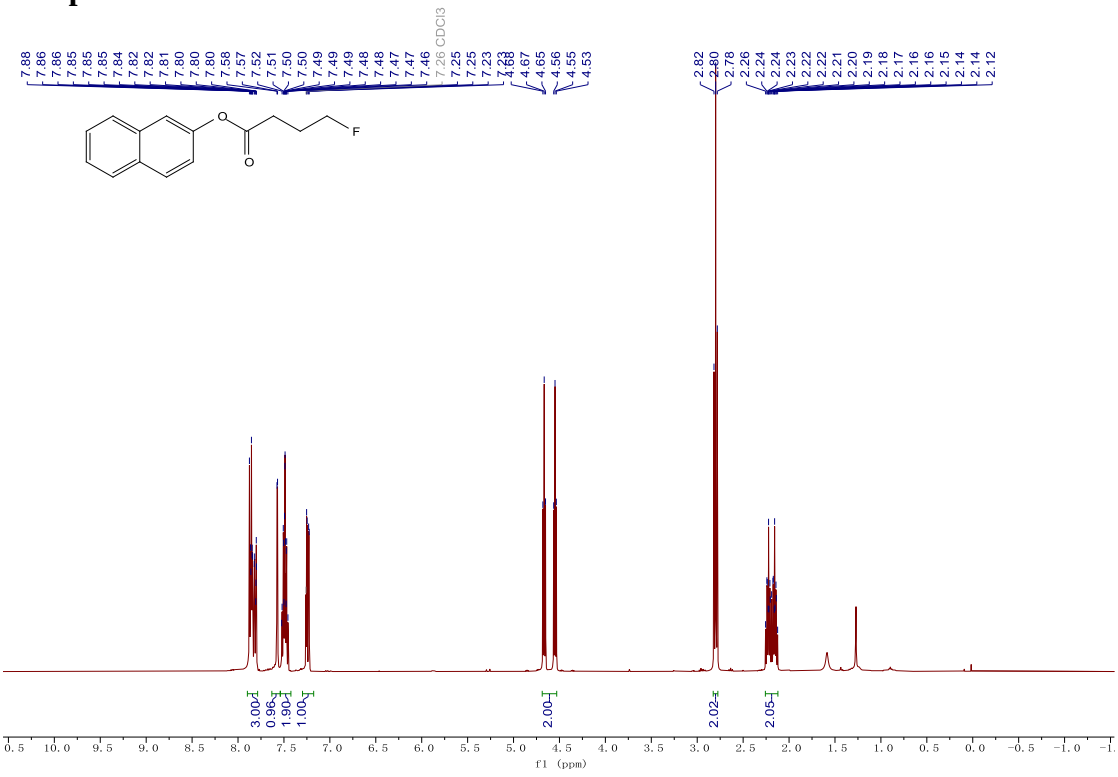
Compound 35b ^{13}C NMR



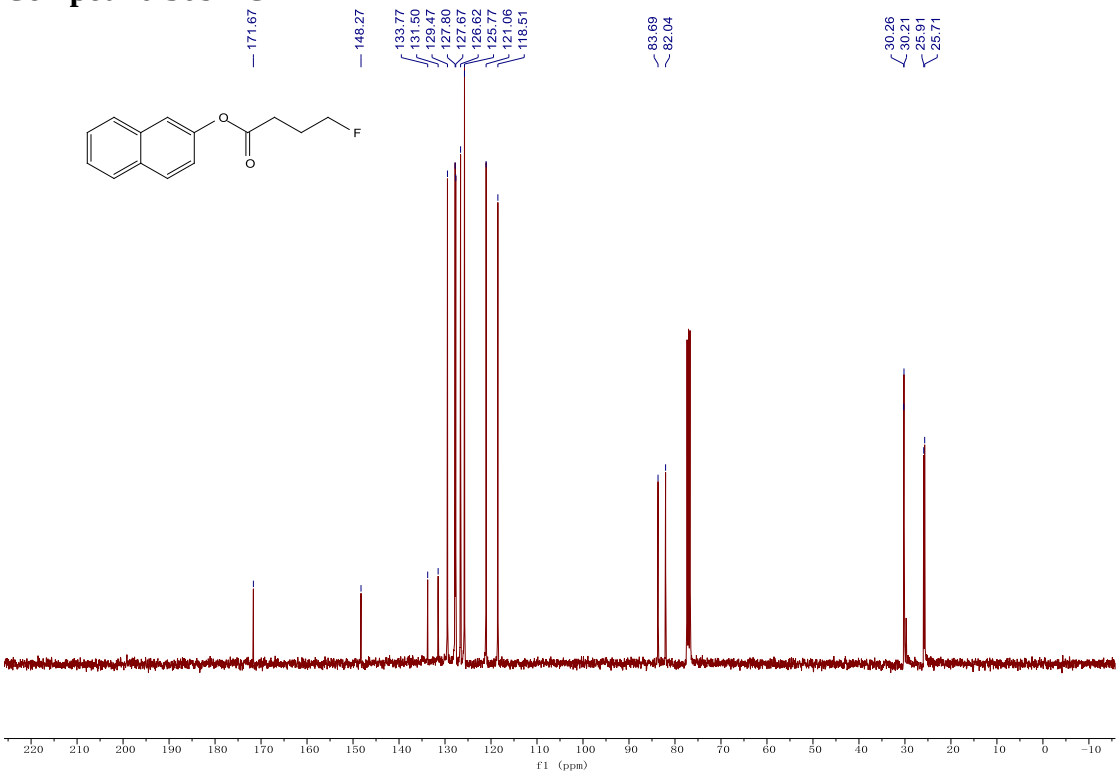
Compound 35b ^{19}F NMR



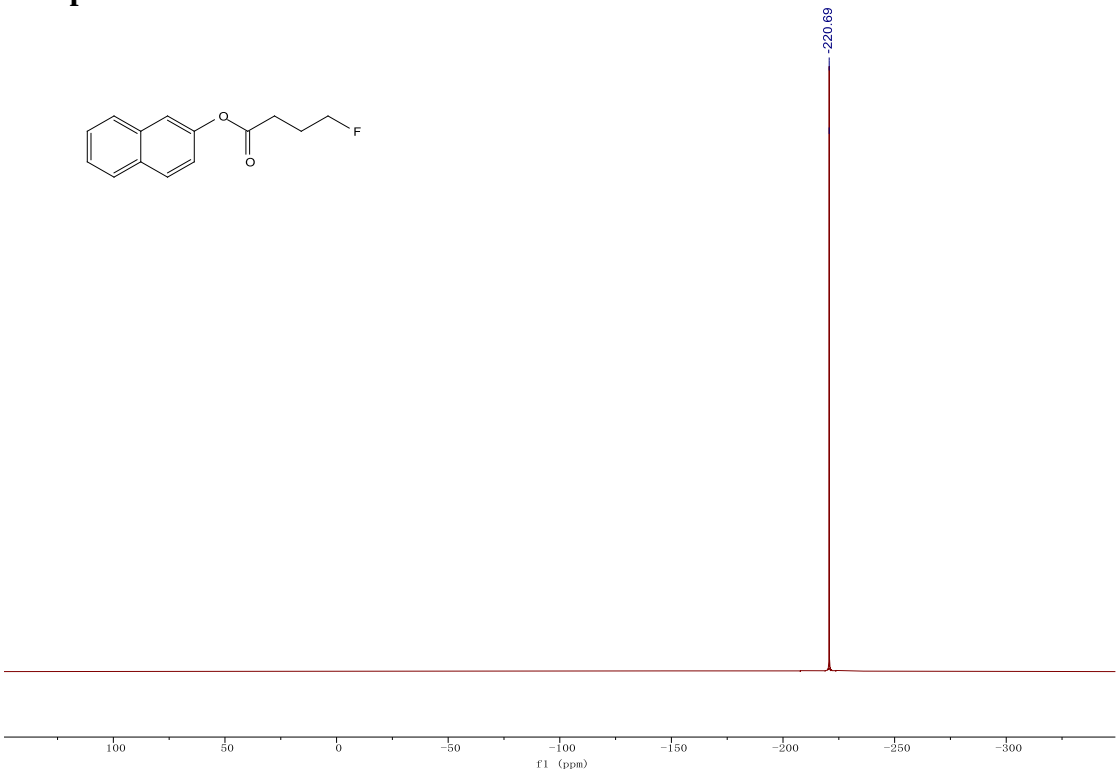
Compound 36b ^{1}H NMR



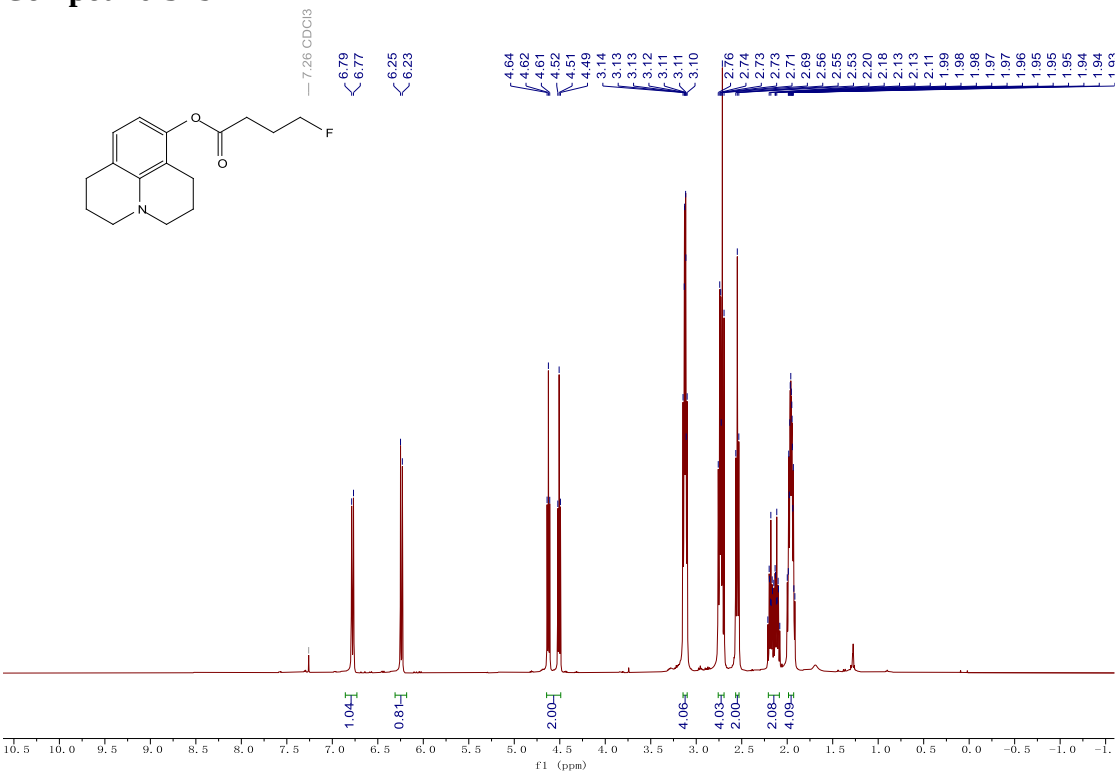
Compound 36b ^{13}C NMR



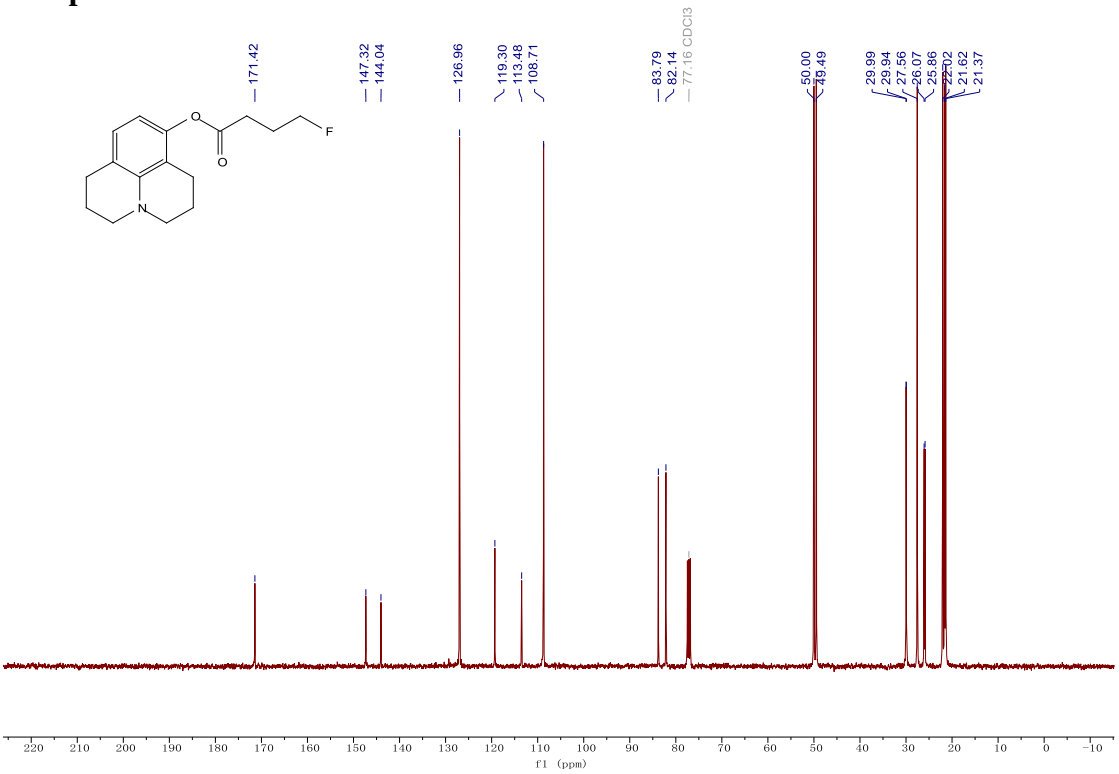
Compound 36b ^{19}F NMR



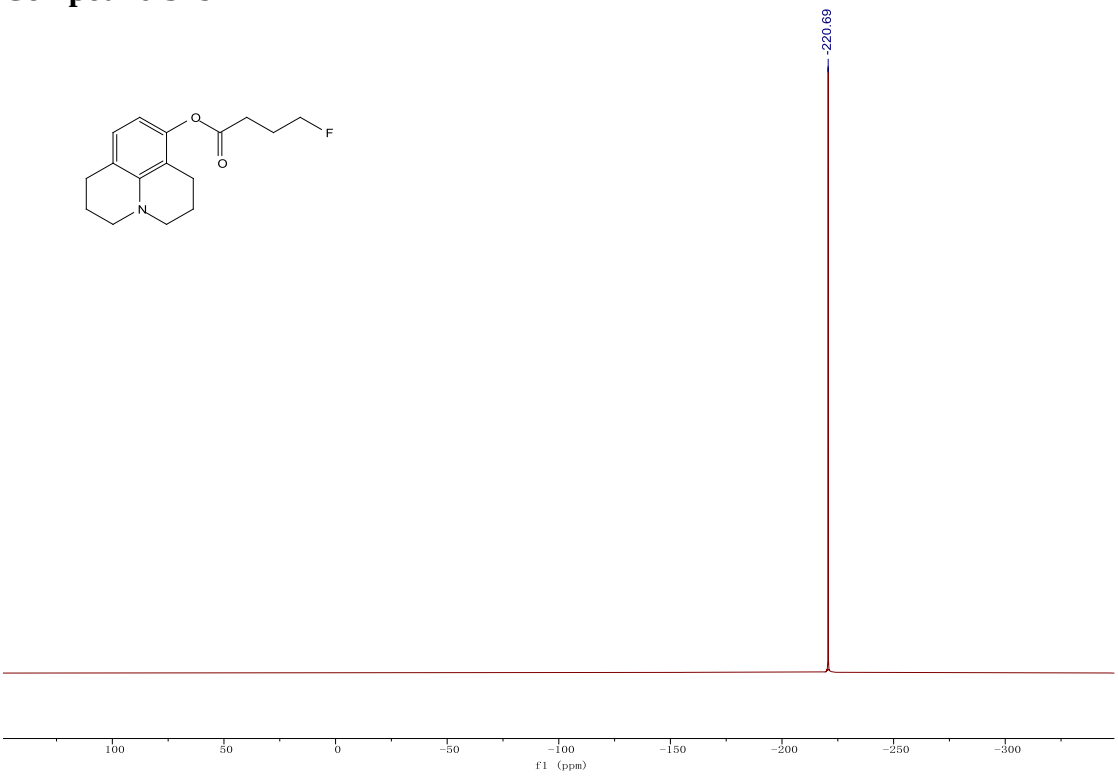
Compound 37b ^{1}H NMR



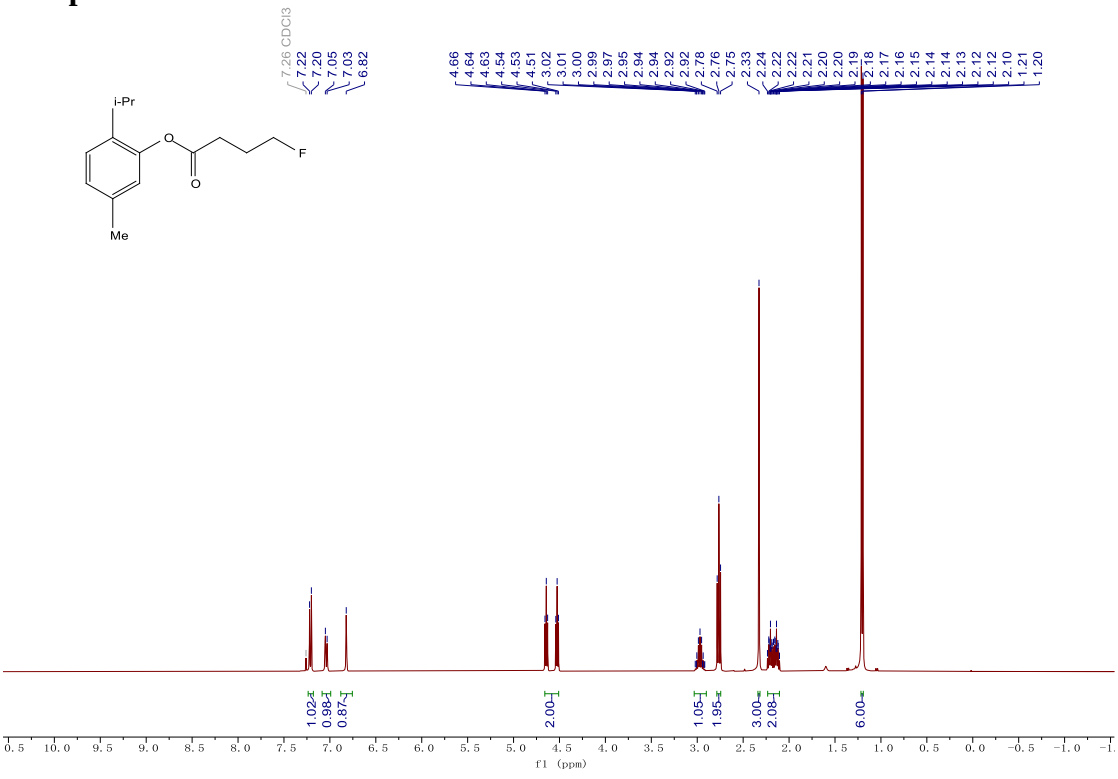
Compound 37b ^{13}C NMR



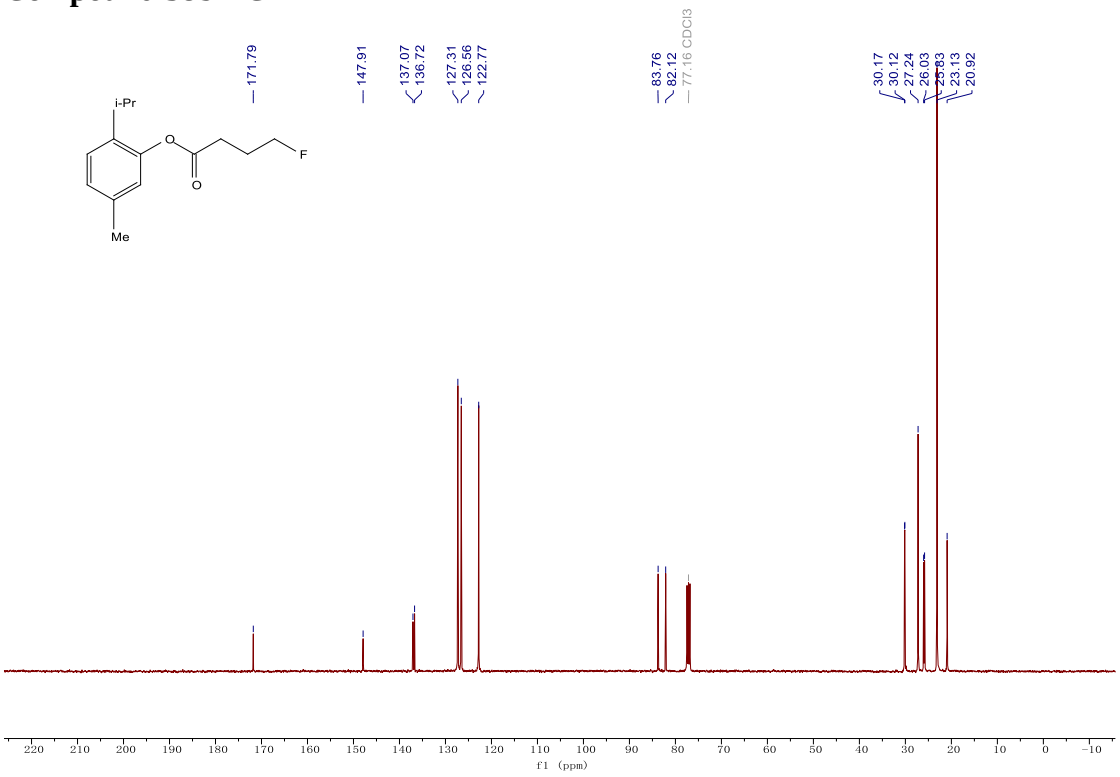
Compound 37b ^{19}F NMR



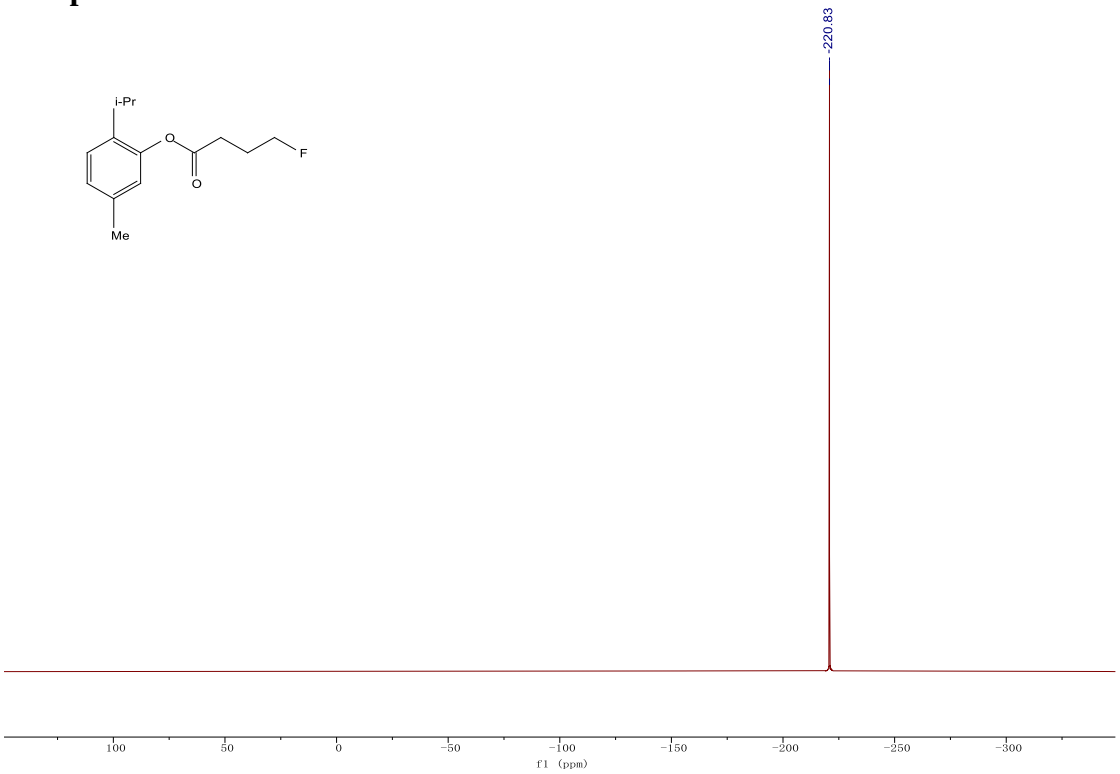
Compound 38b ^{1}H NMR



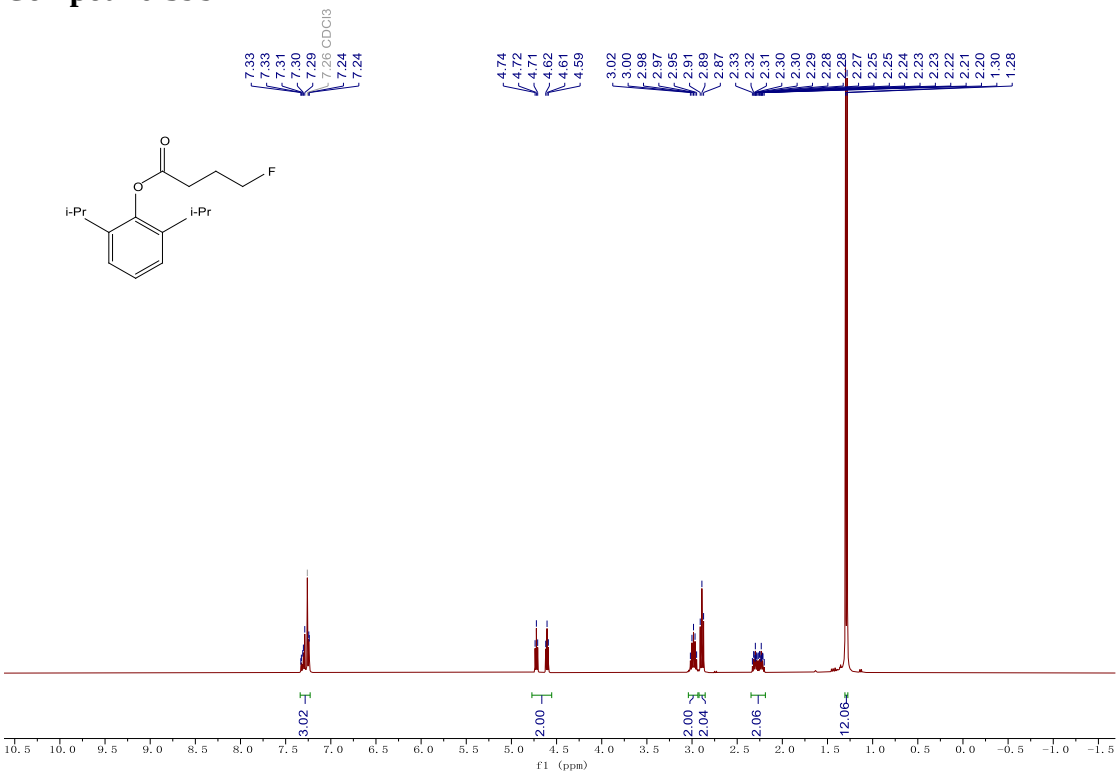
Compound 38b ^{13}C NMR



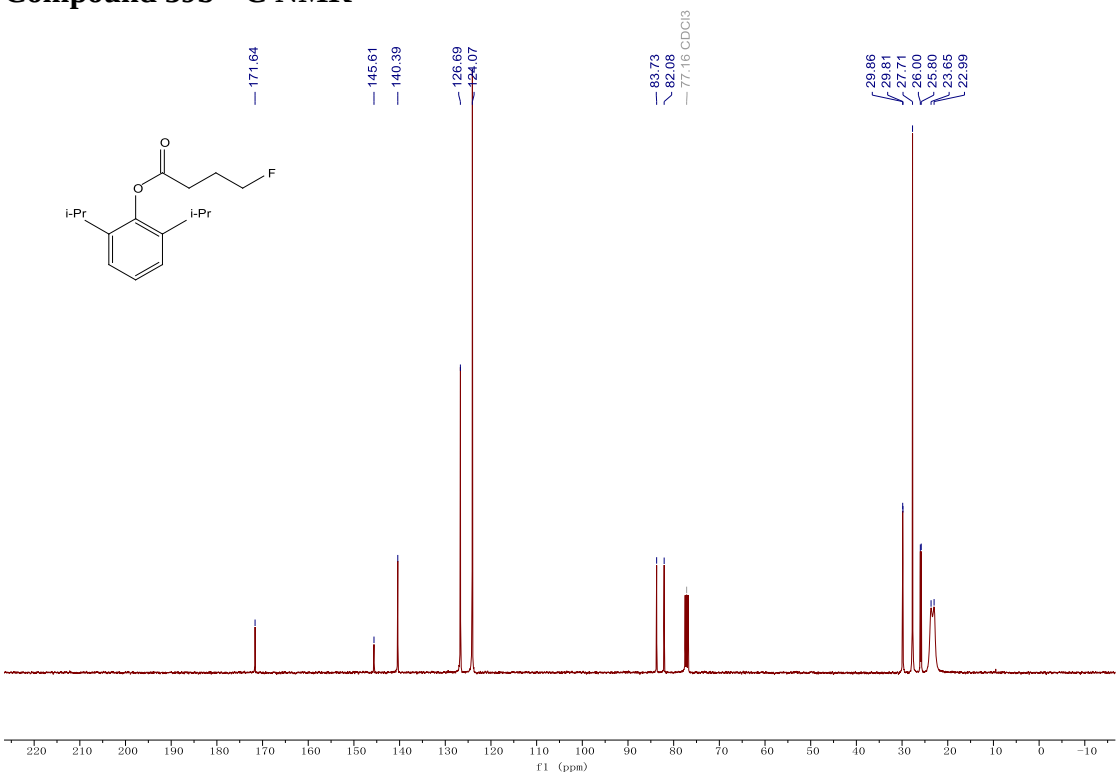
Compound 38b ^{19}F NMR



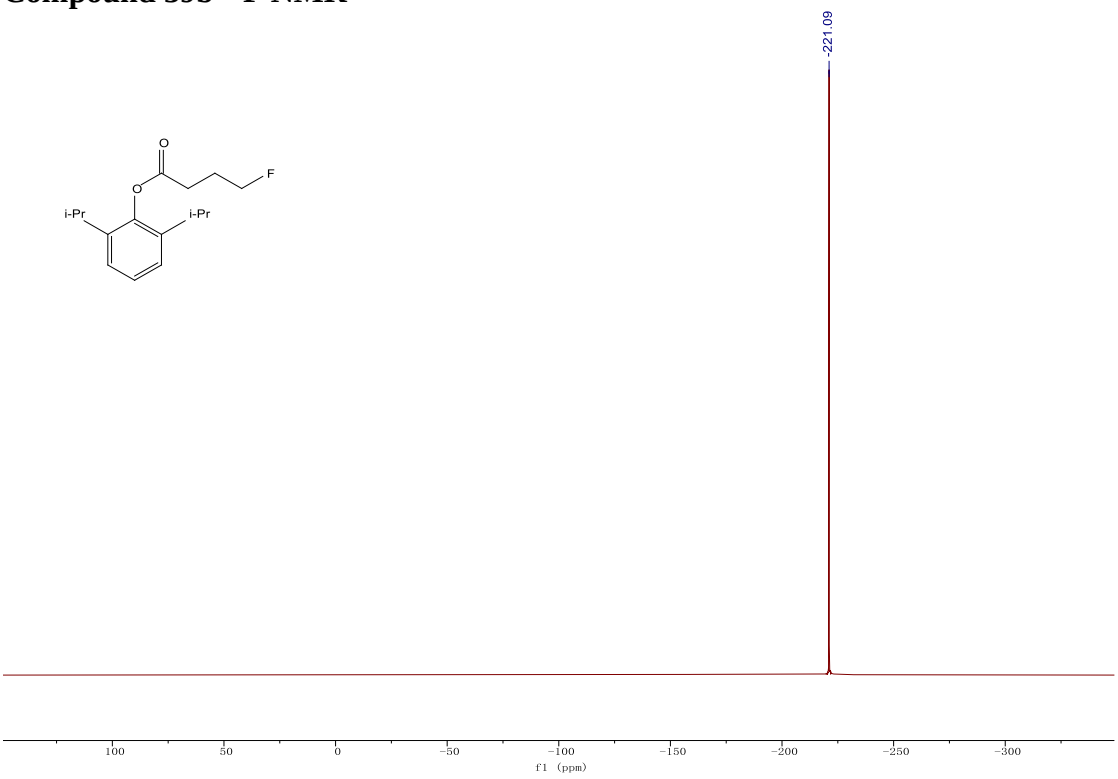
Compound 39b ^{1}H NMR



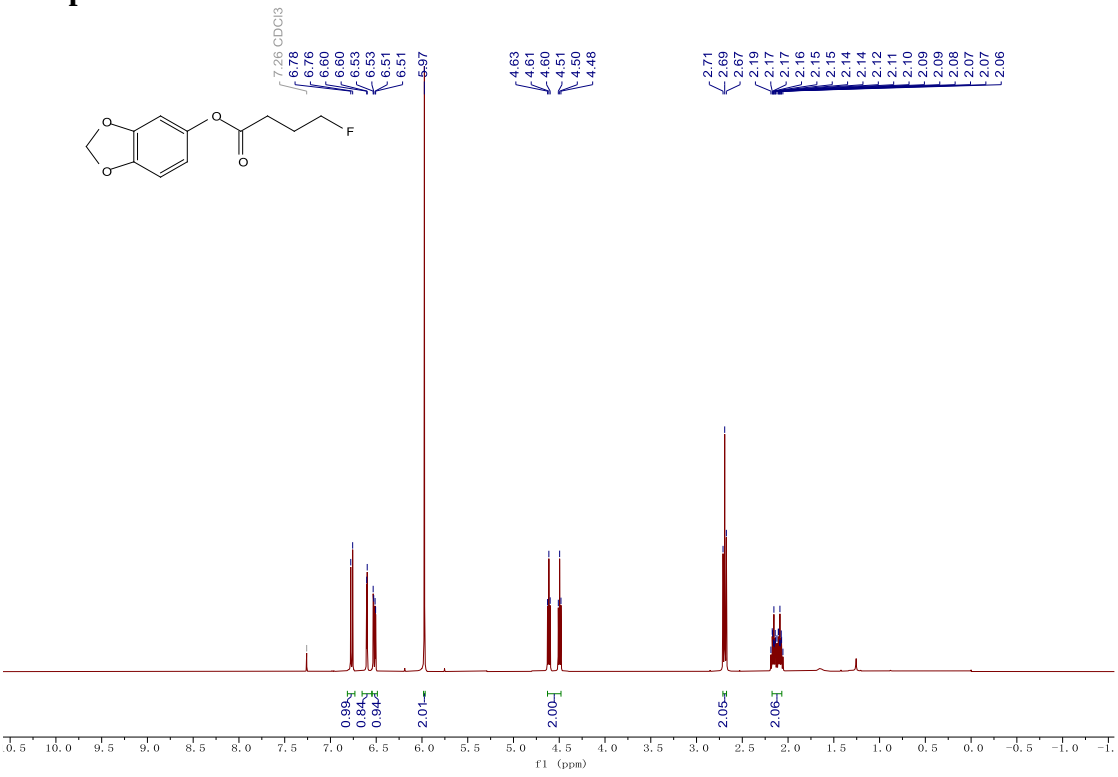
Compound 39b ^{13}C NMR



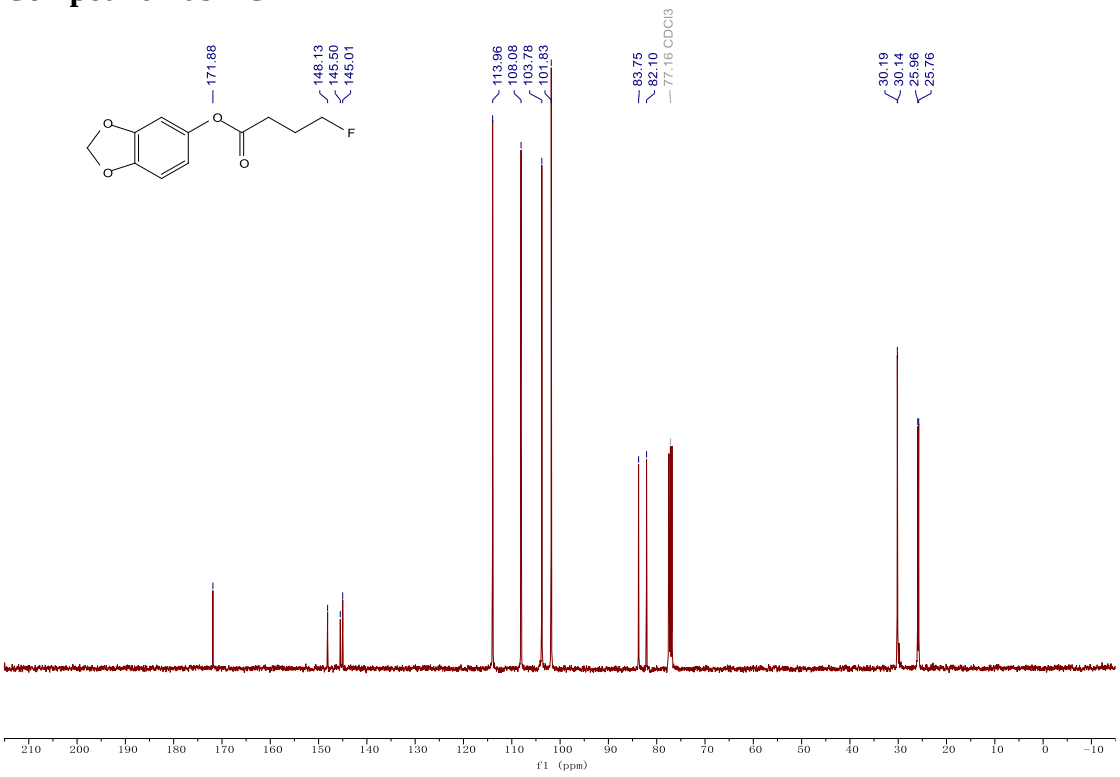
Compound 39b ^{19}F NMR



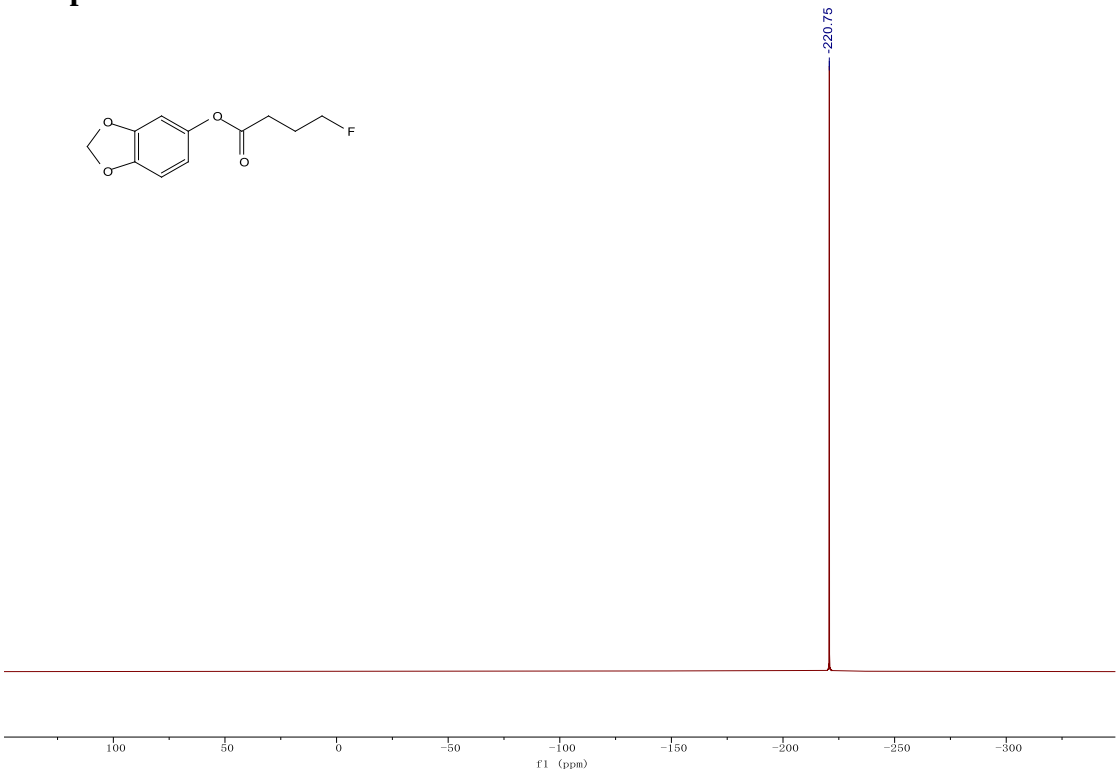
Compound 40b ^{1}H NMR



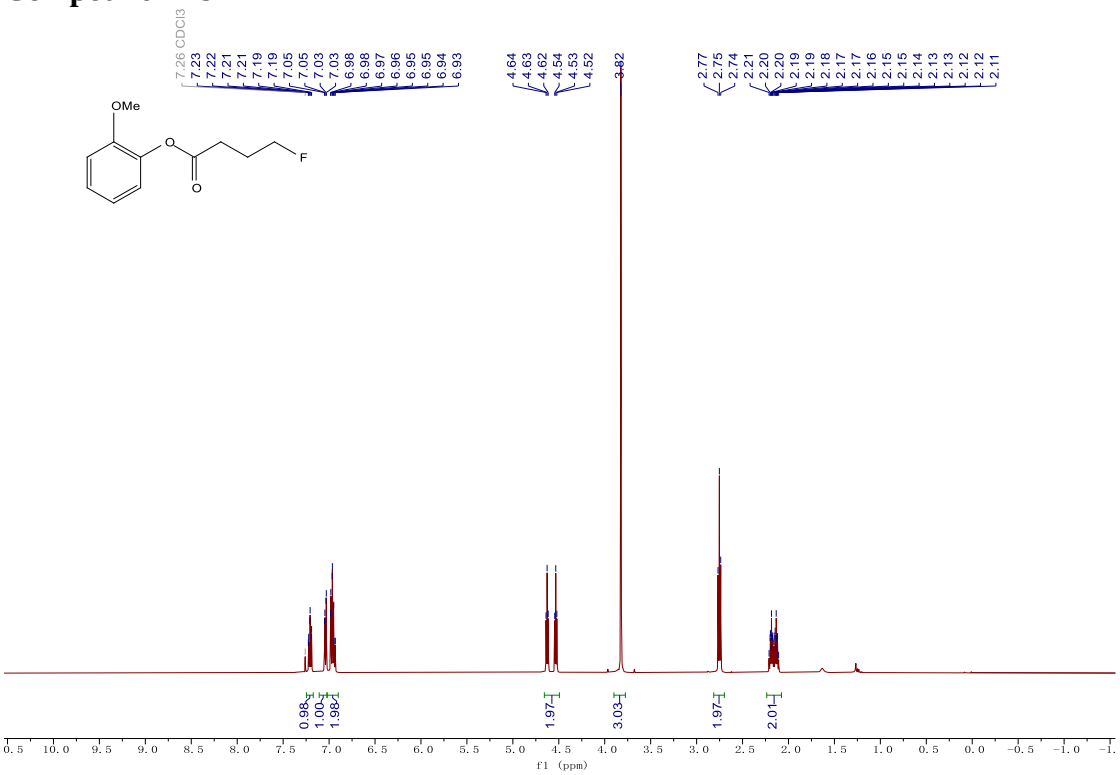
Compound 40b ^{13}C NMR



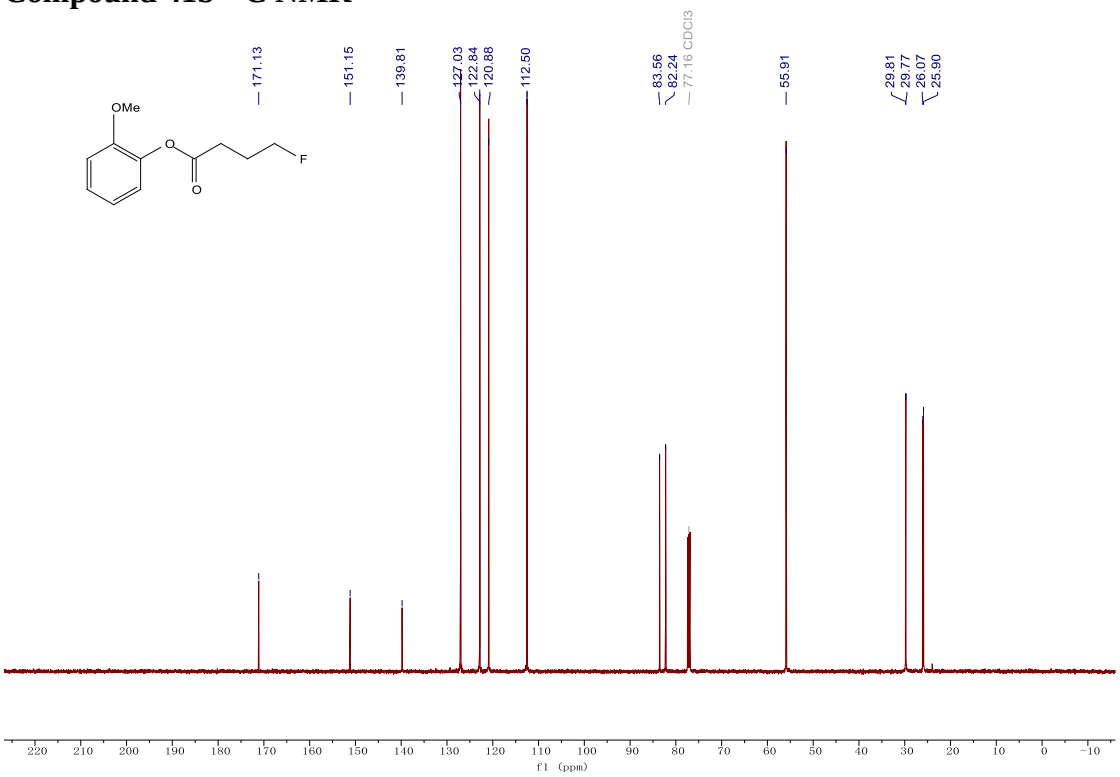
Compound 40b ^{19}F NMR



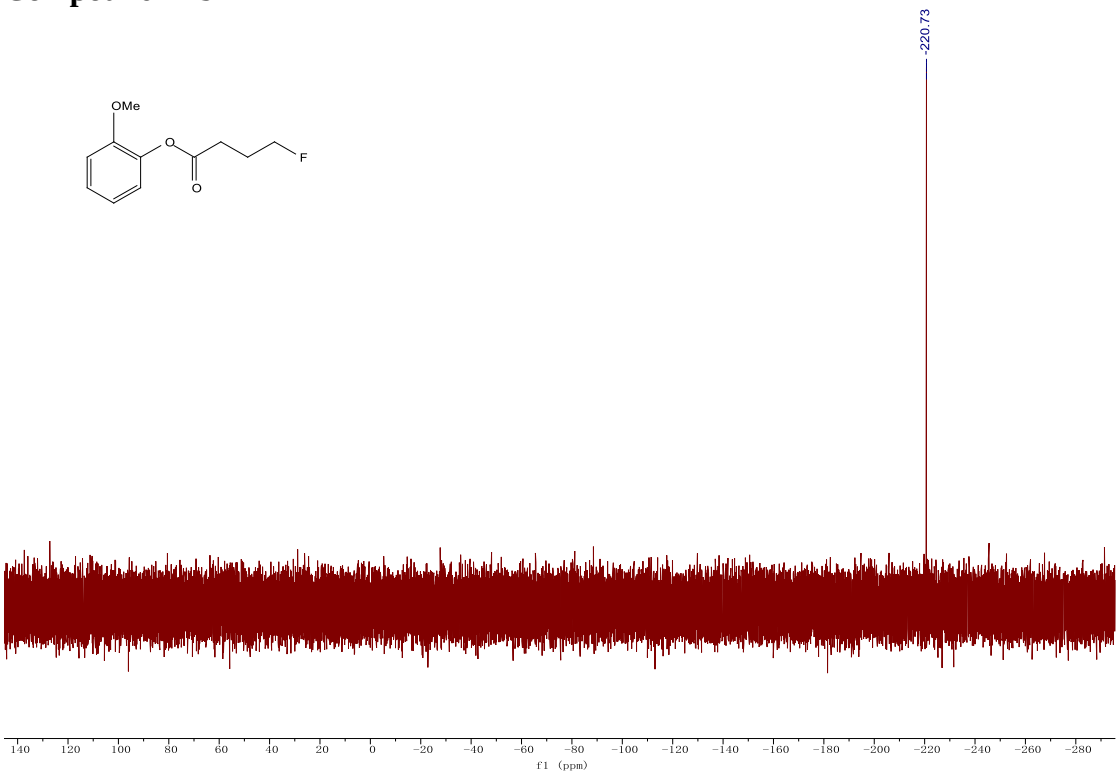
Compound 41b ^{1}H NMR



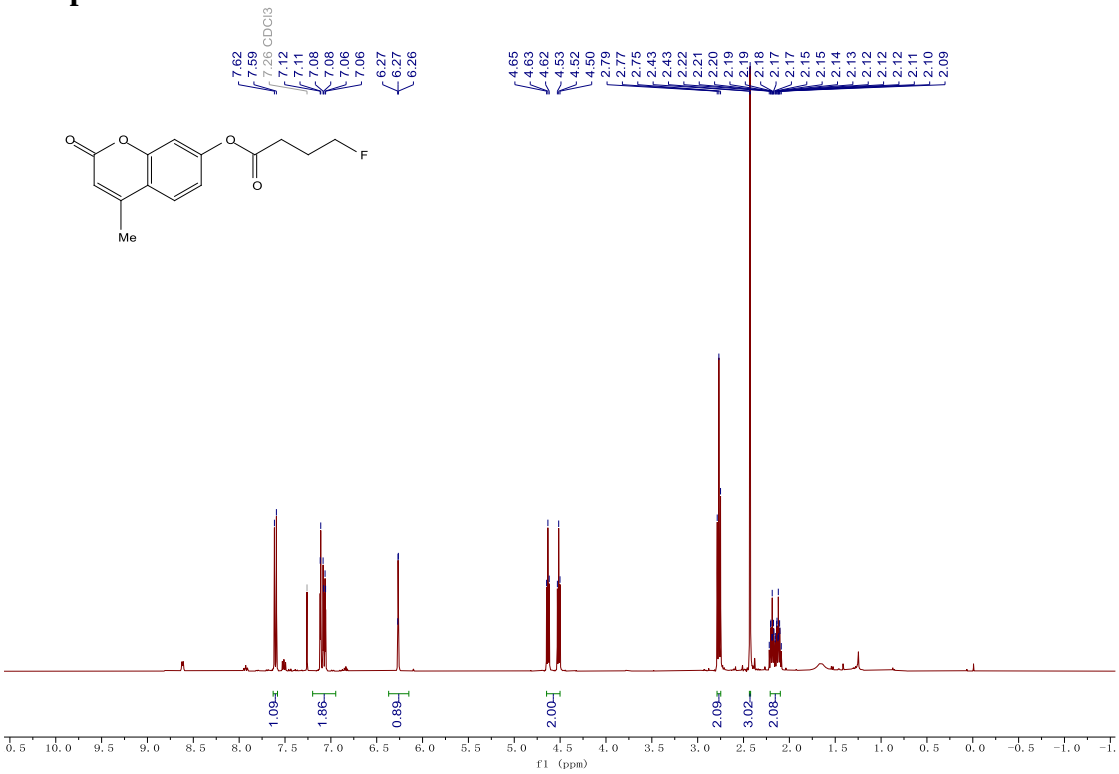
Compound 41b ^{13}C NMR



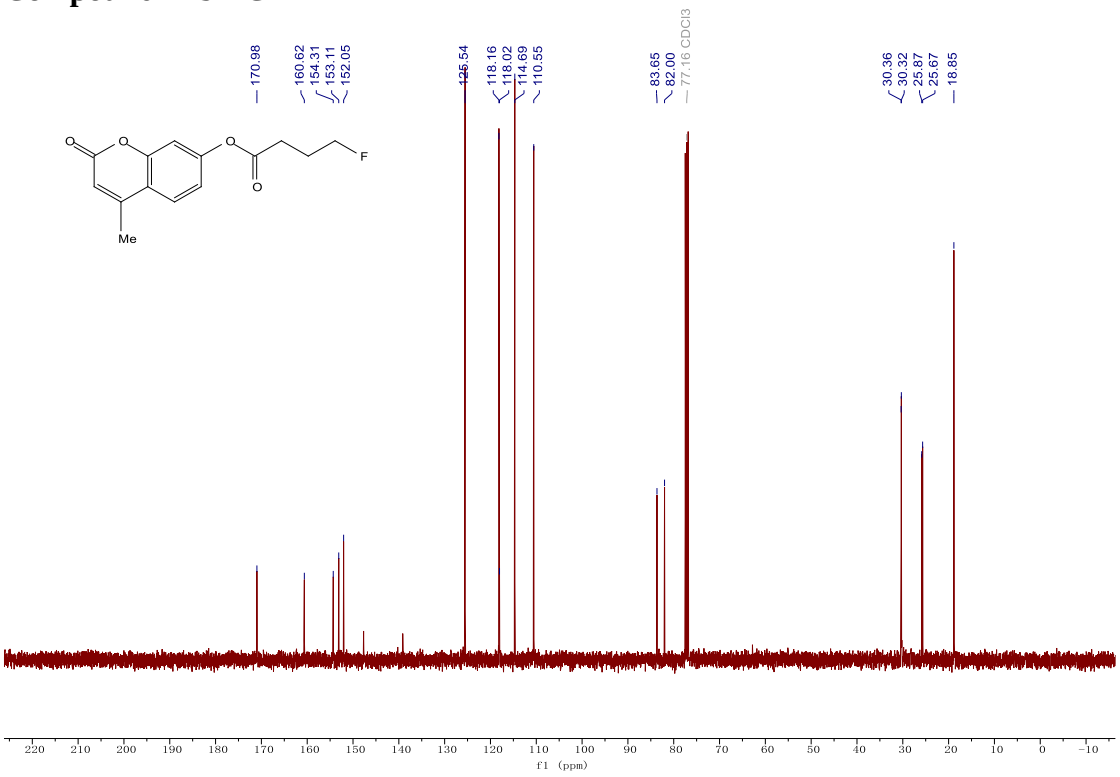
Compound 41b ^{19}F NMR



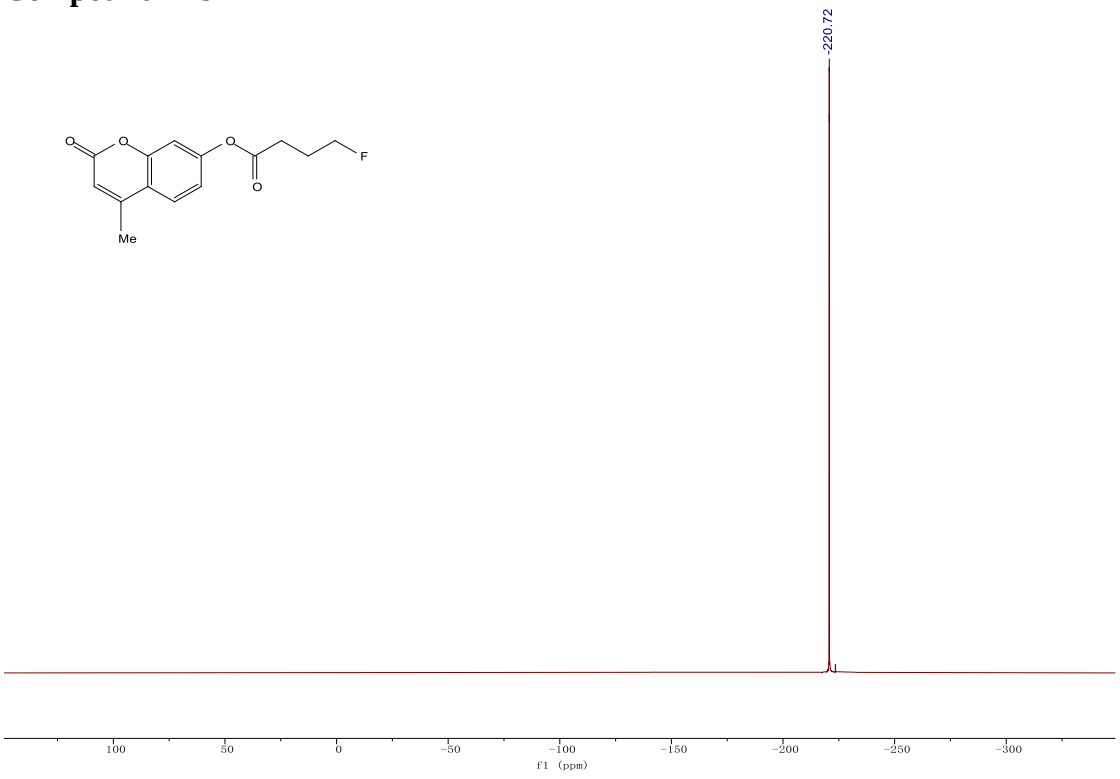
Compound 42b ^{1}H NMR



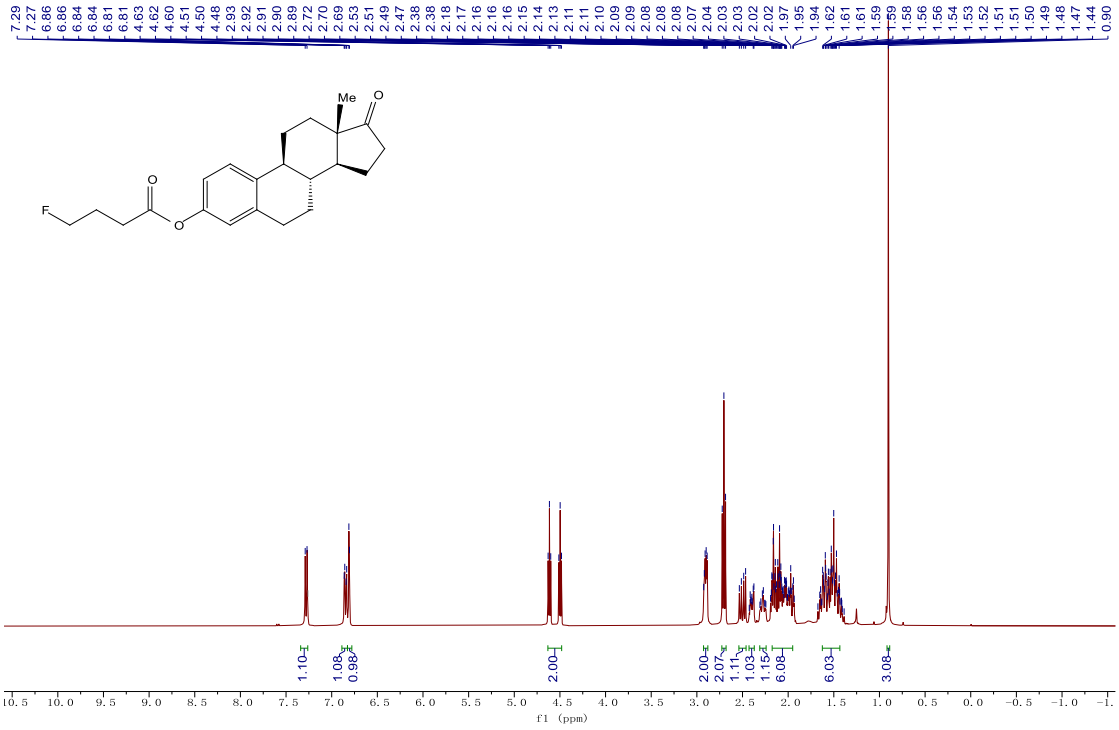
Compound 42b ^{13}C NMR



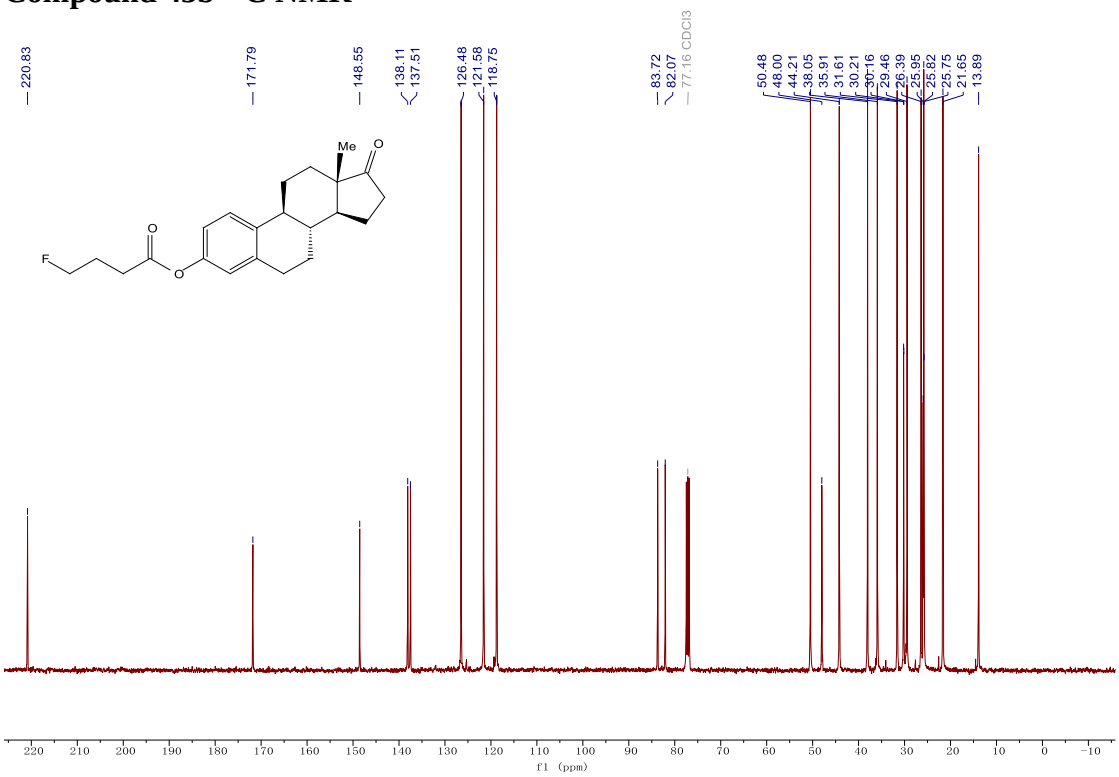
Compound 42b ^{19}F NMR



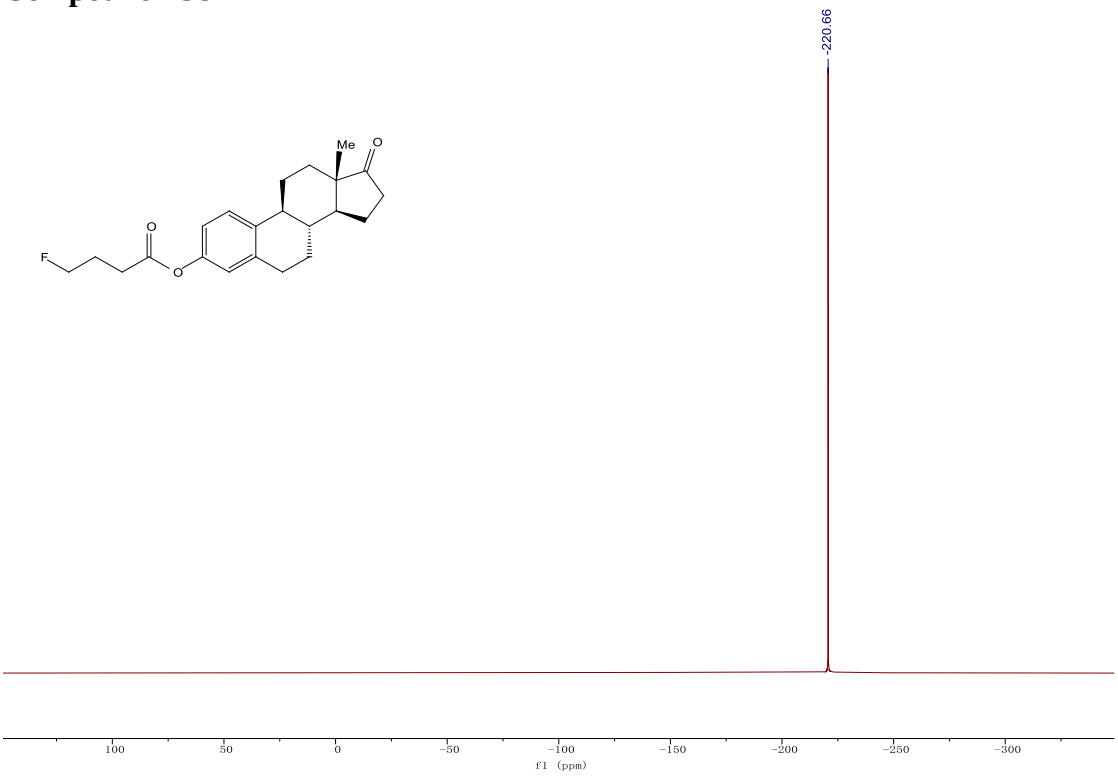
Compound 43b ^{1}H NMR



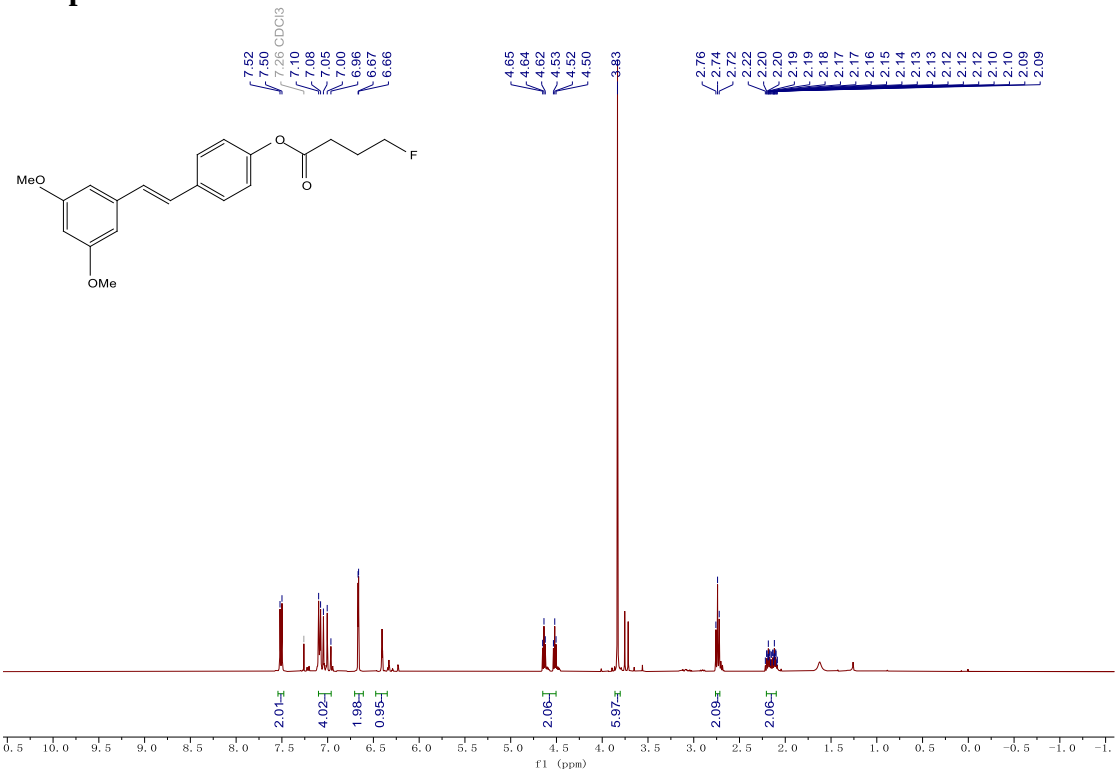
Compound 43b ^{13}C NMR



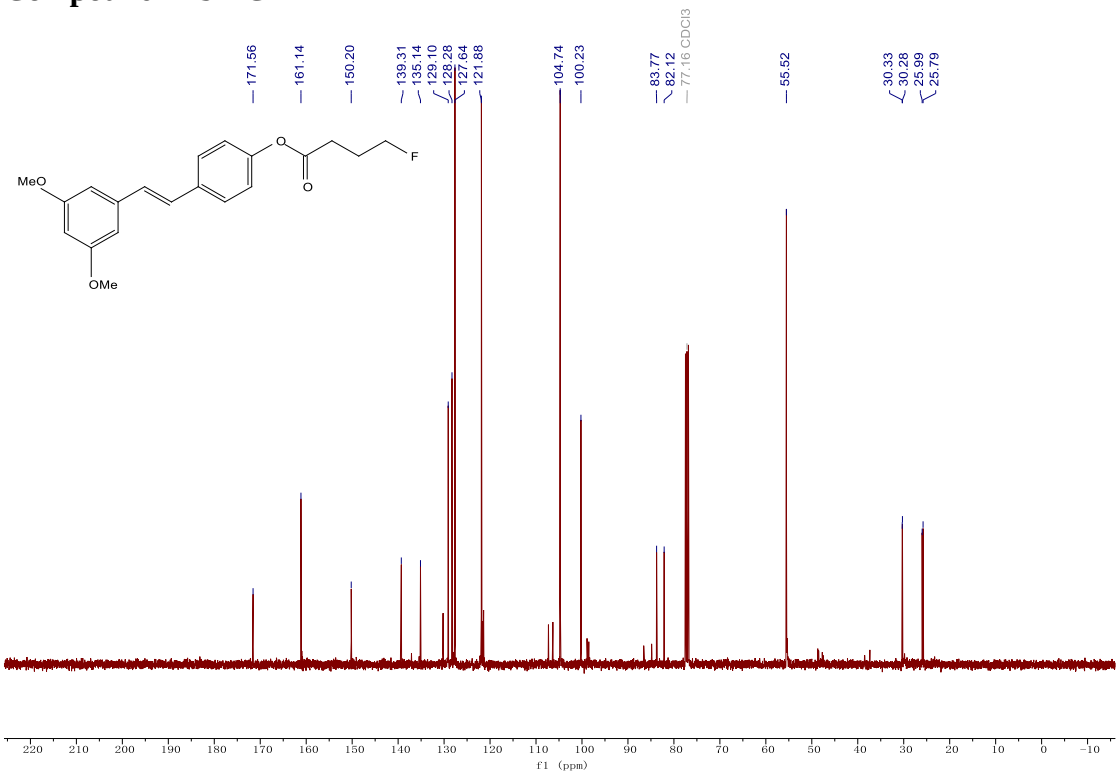
Compound 43b ^{19}F NMR



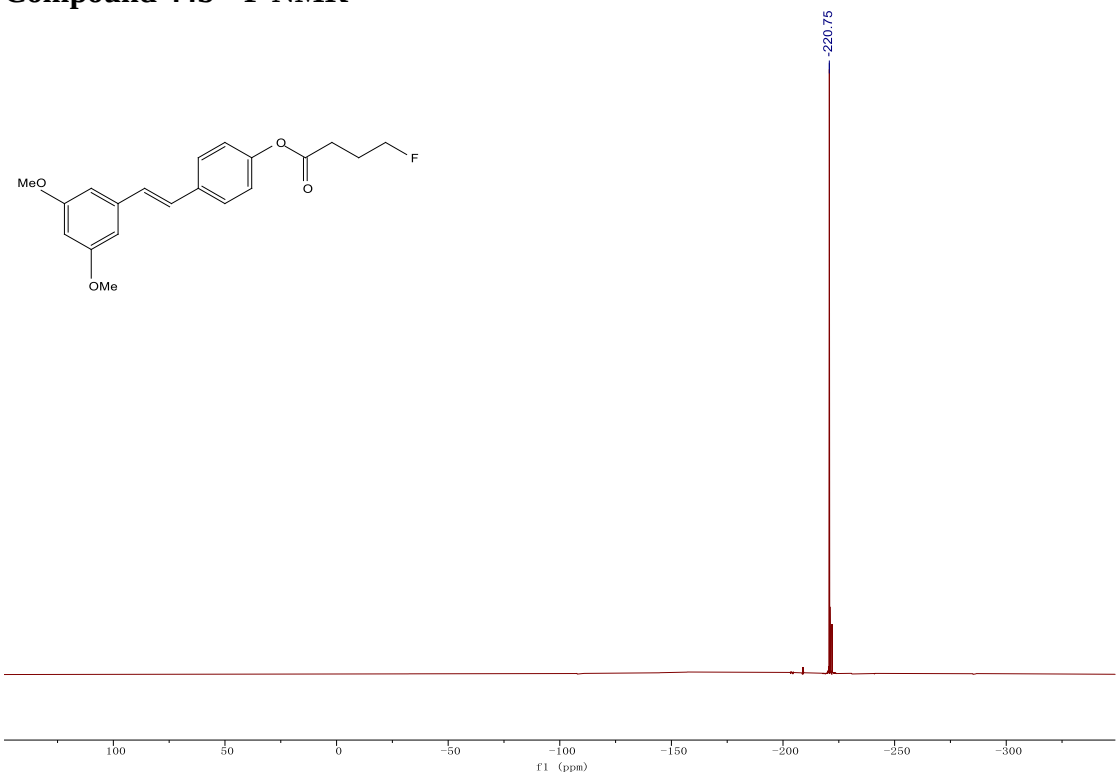
Compound 44b ^{1}H NMR



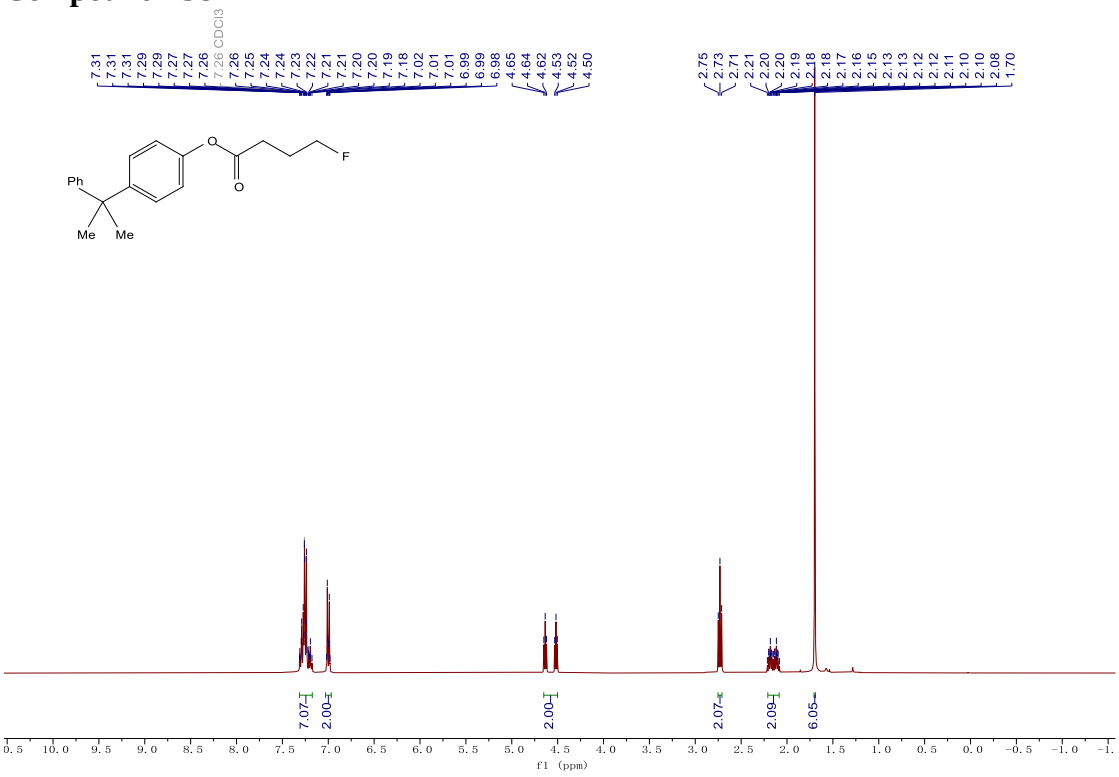
Compound 44b ^{13}C NMR



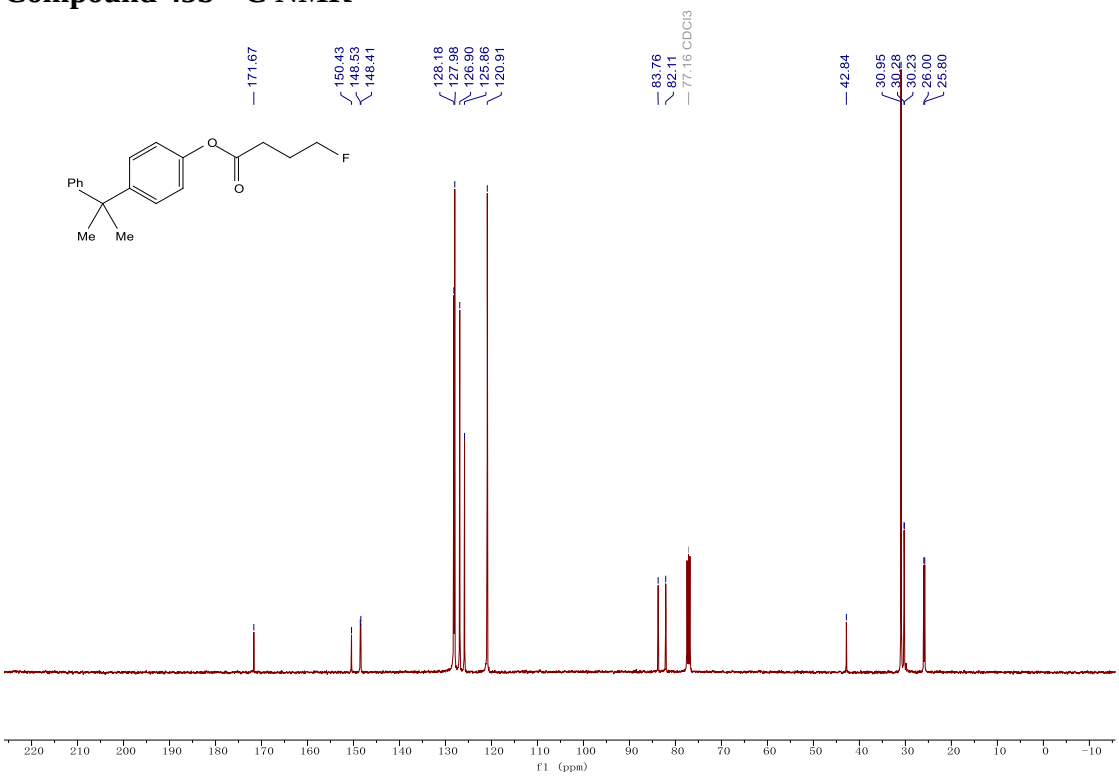
Compound 44b ^{19}F NMR



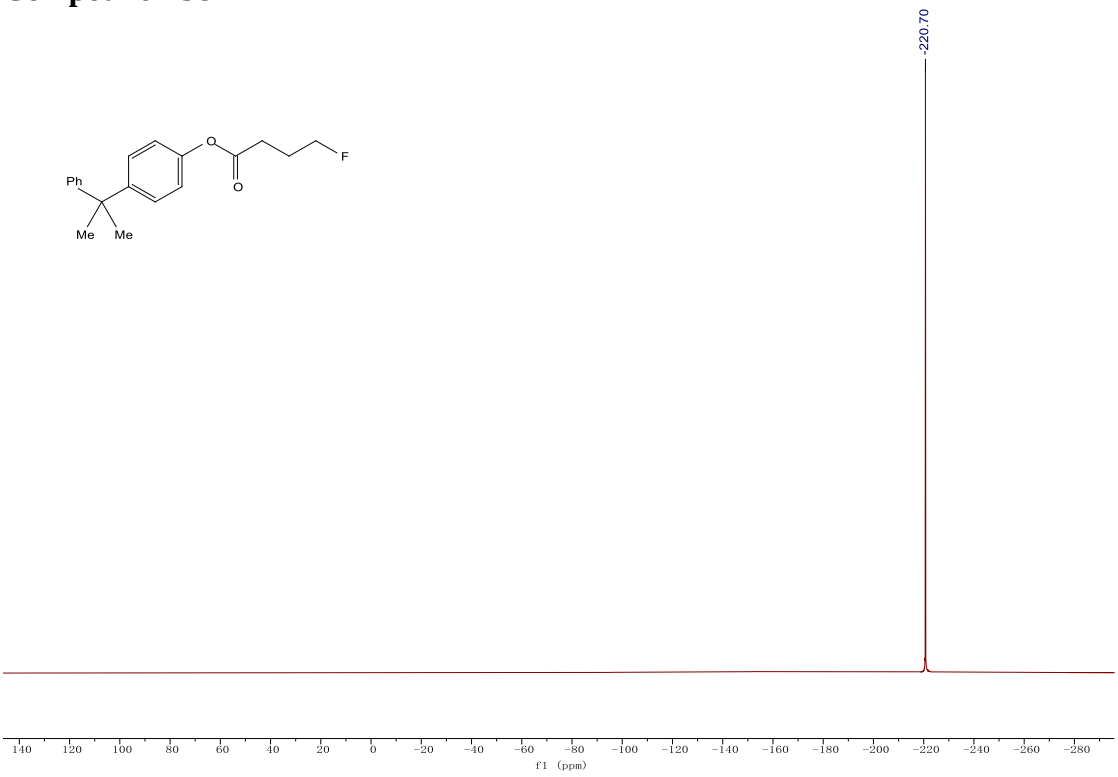
Compound 45b ^{1}H NMR



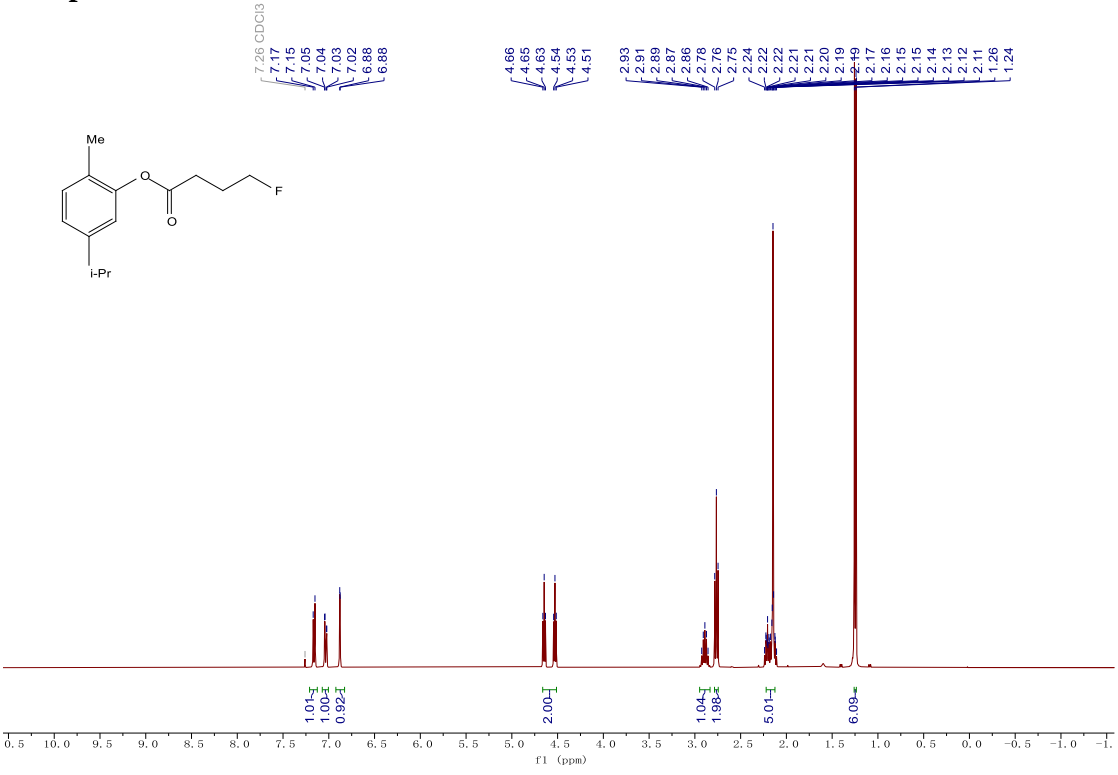
Compound 45b ^{13}C NMR



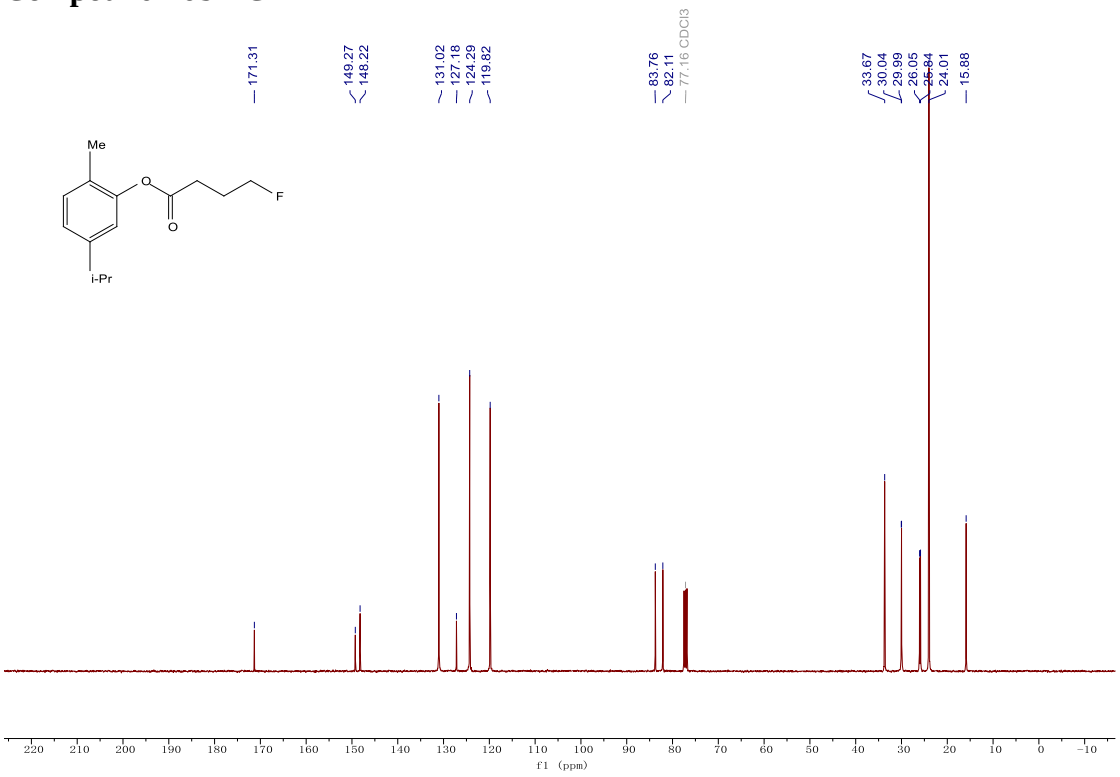
Compound 45b ^{19}F NMR



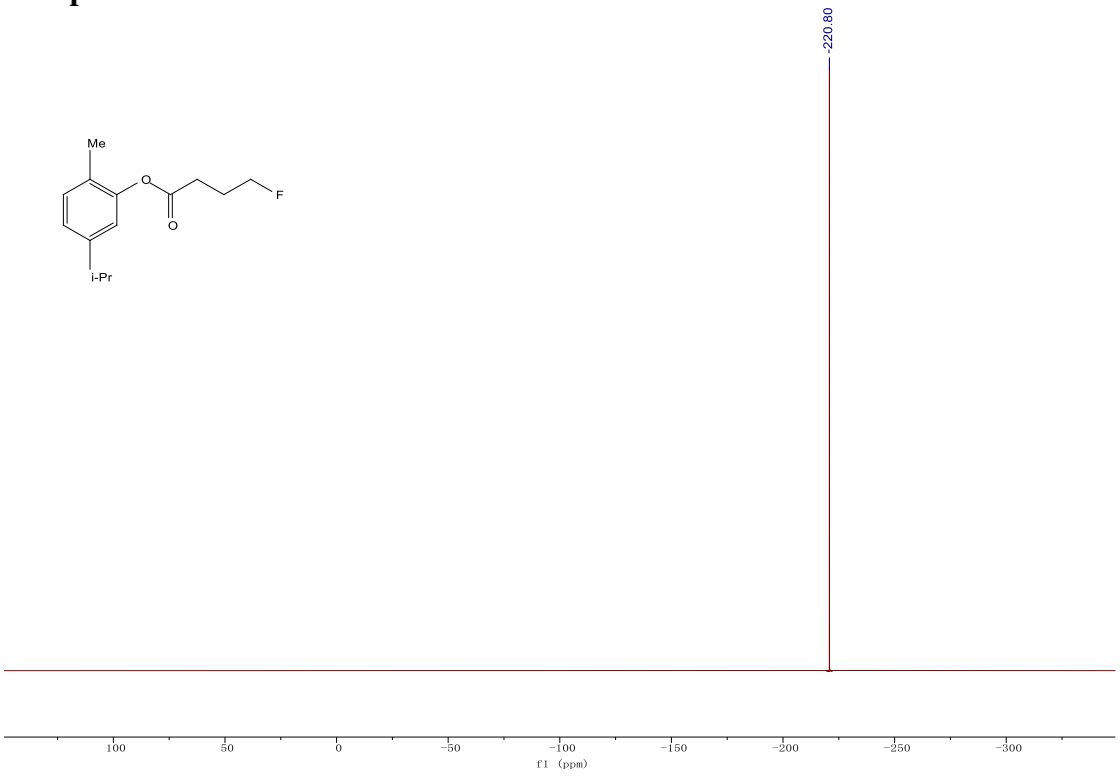
Compound 46b ^{1}H NMR



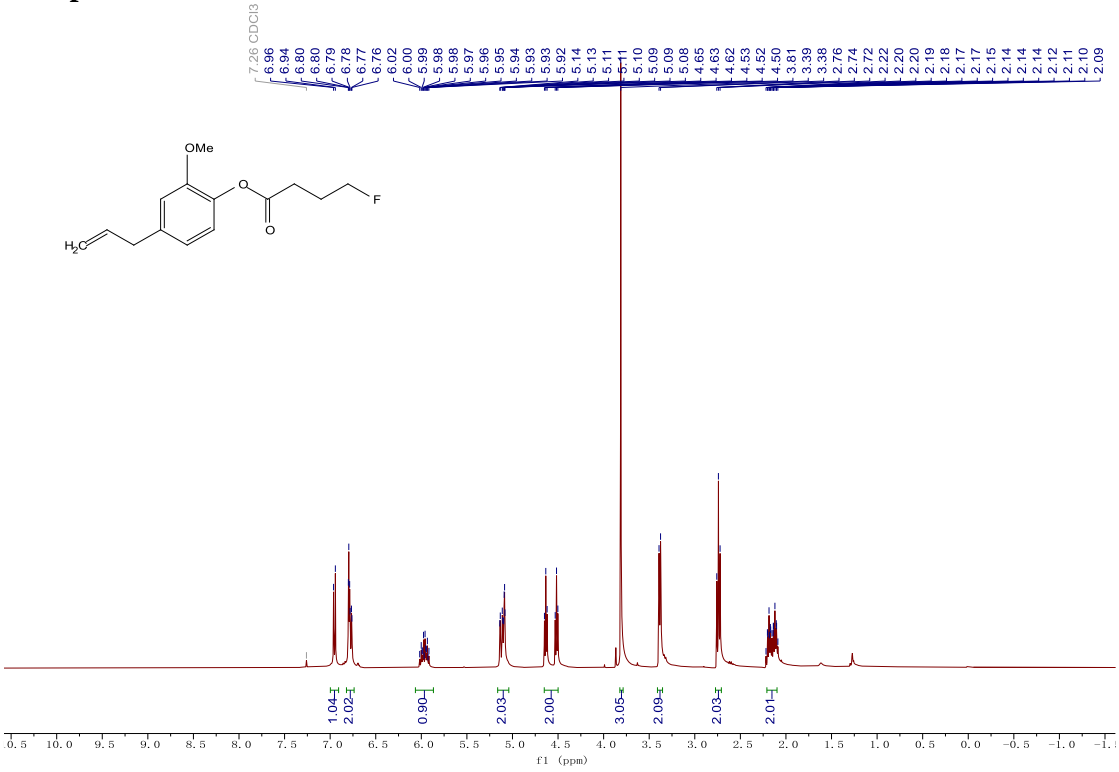
Compound 46b ^{13}C NMR



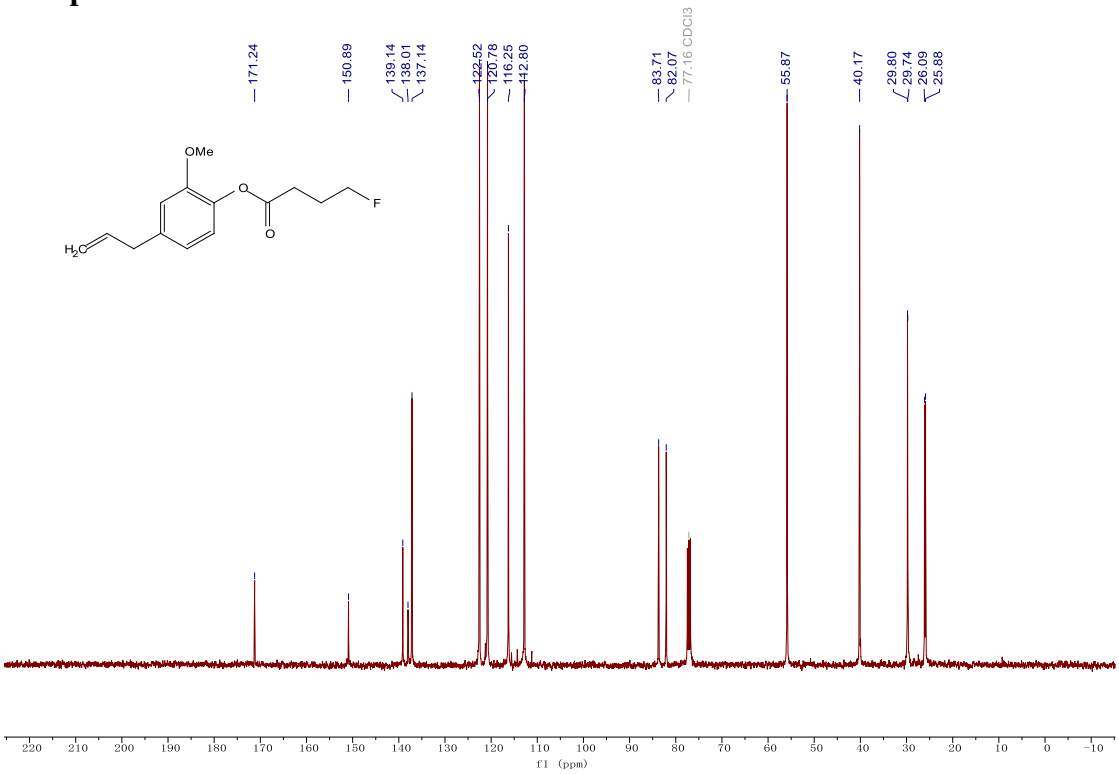
Compound 46b ^{19}F NMR



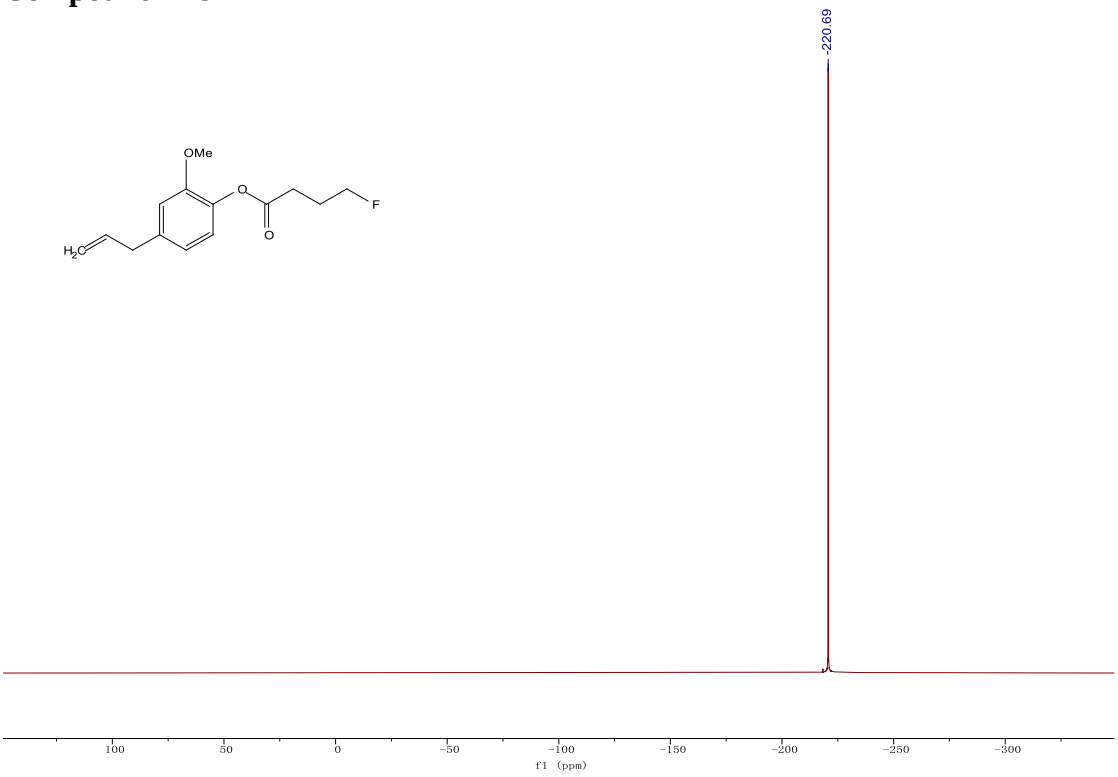
Compound 47b ^{1}H NMR



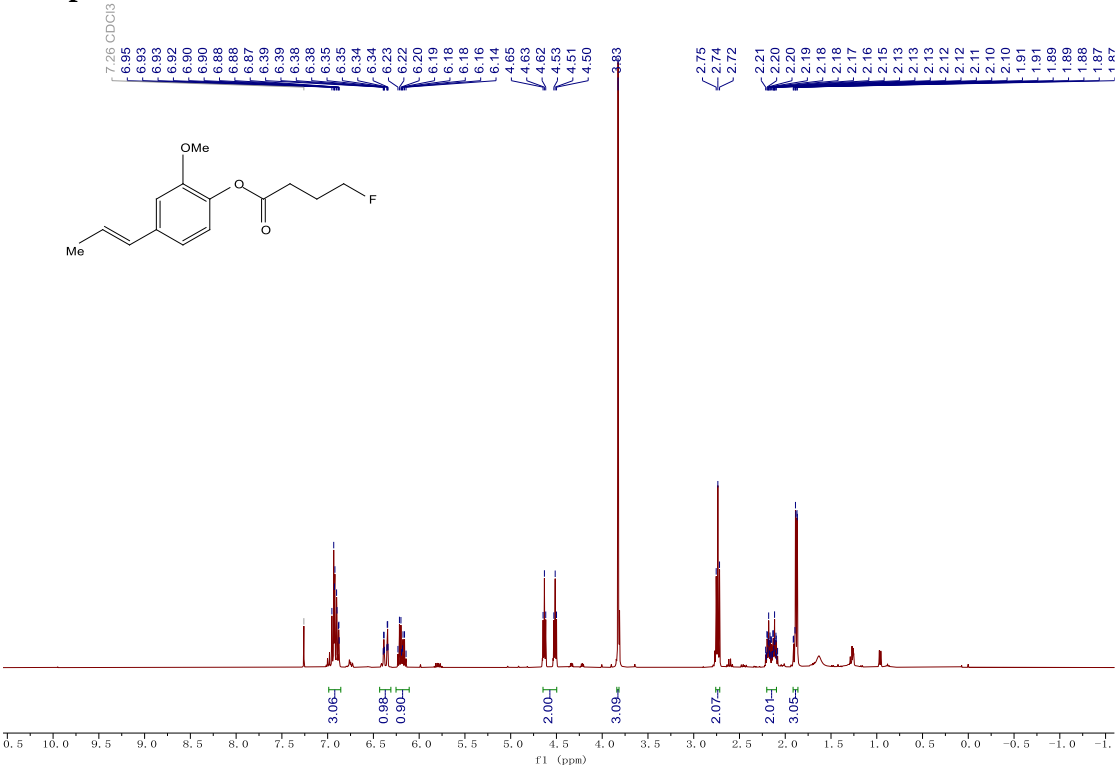
Compound 47b ^{13}C NMR



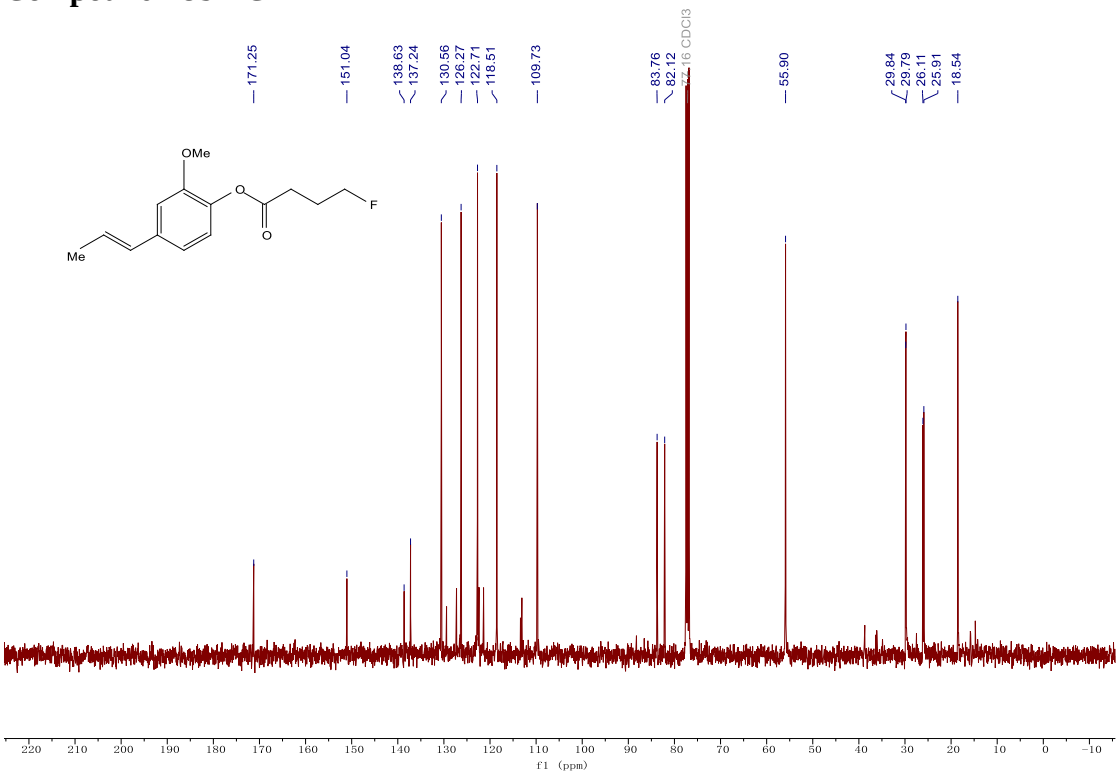
Compound 47b ^{19}F NMR



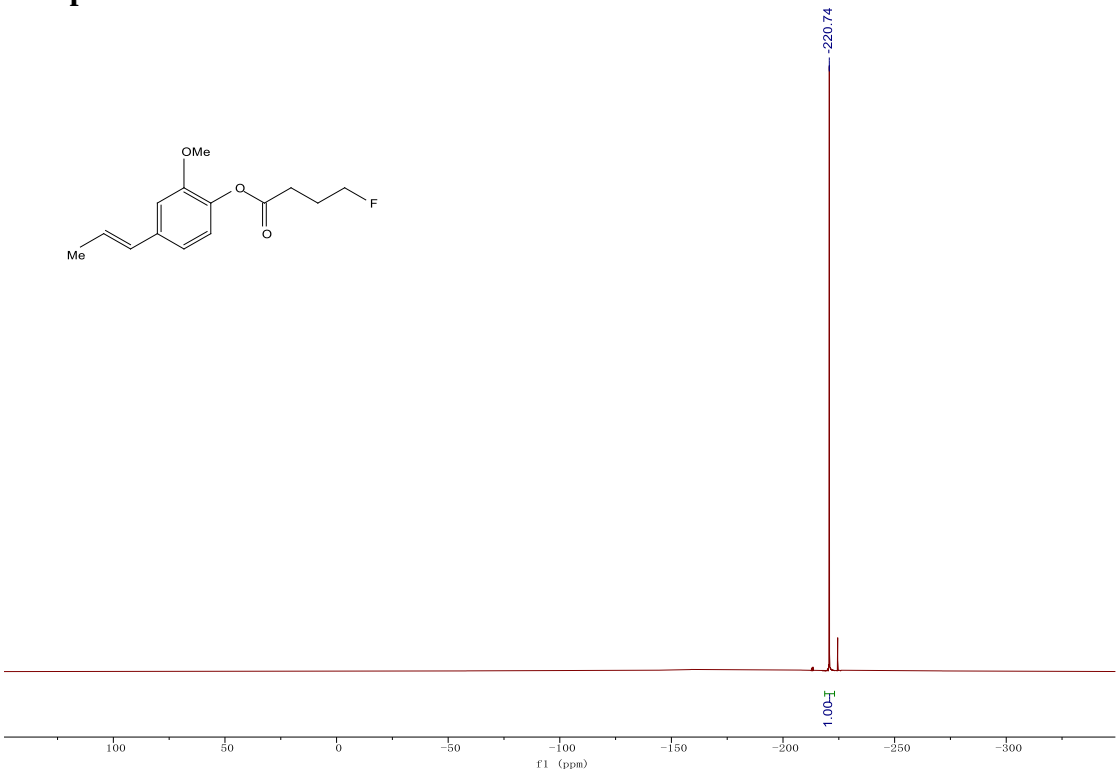
Compound 48b ^{1}H NMR



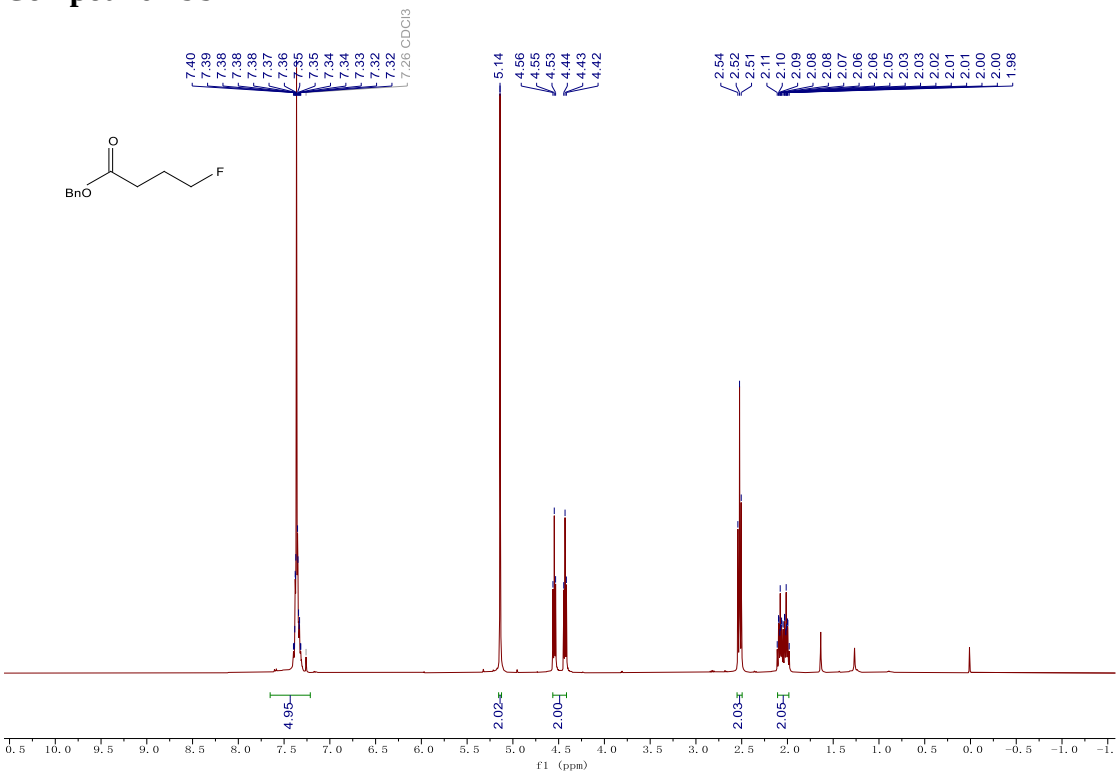
Compound 48b ^{13}C NMR



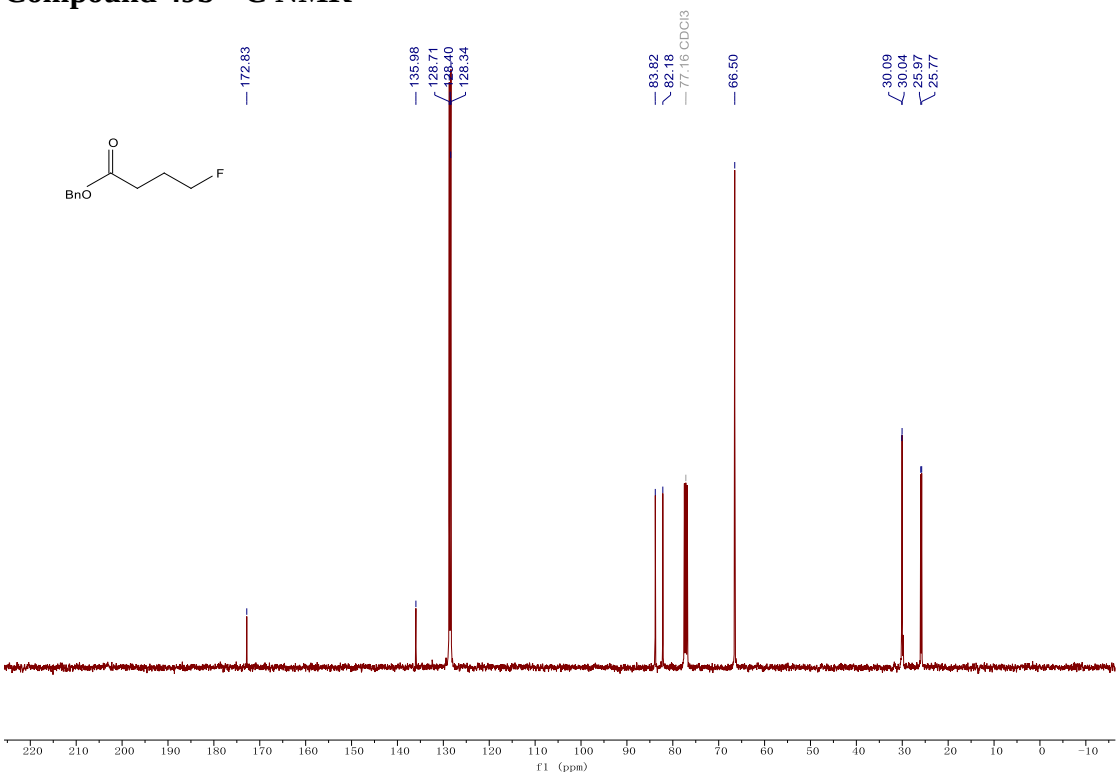
Compound 48b ^{19}F NMR



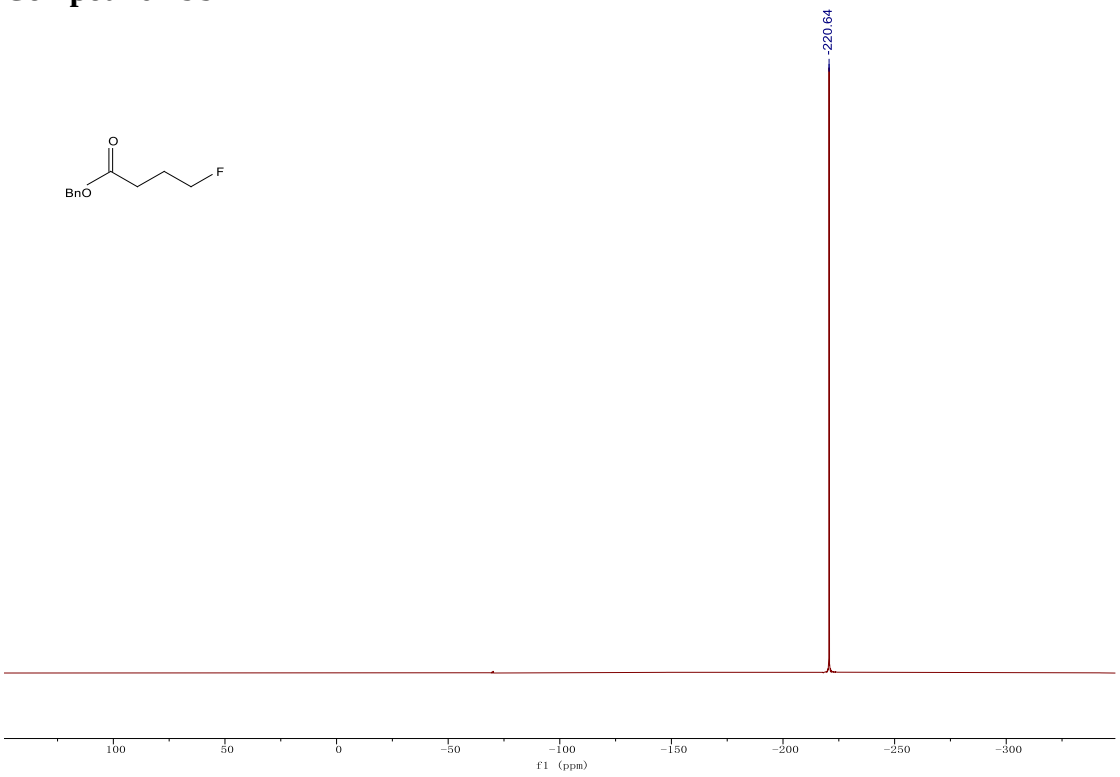
Compound 49b ^{1}H NMR



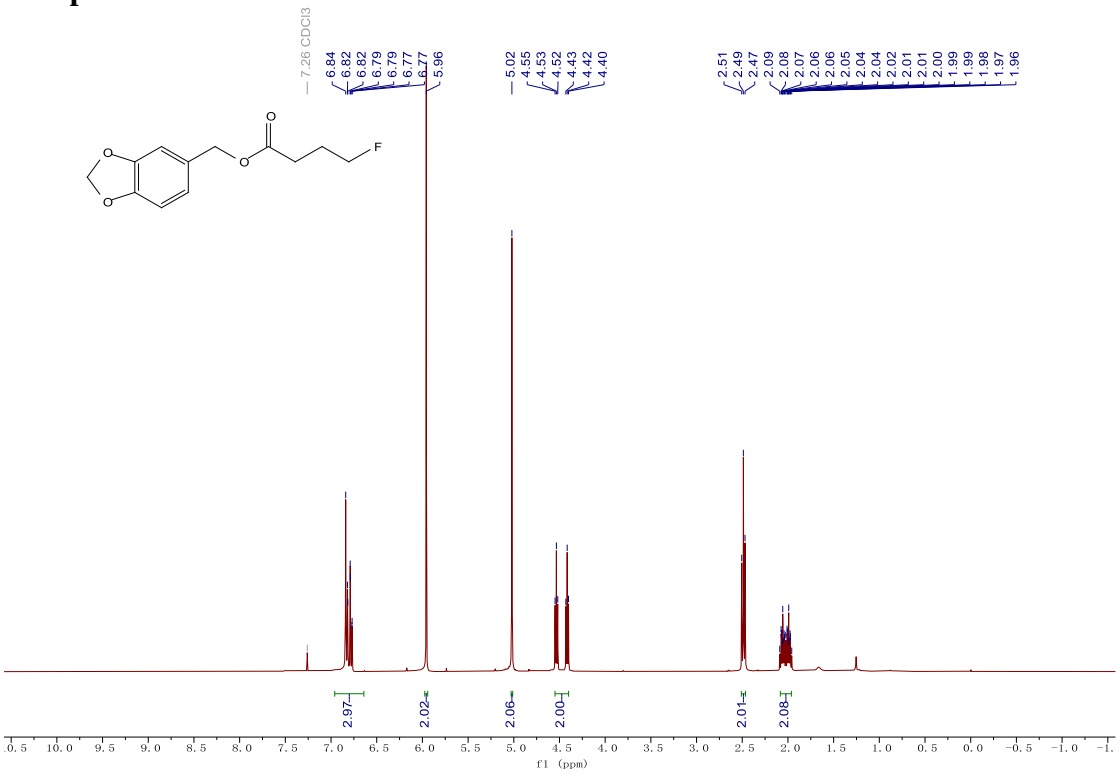
Compound 49b ^{13}C NMR



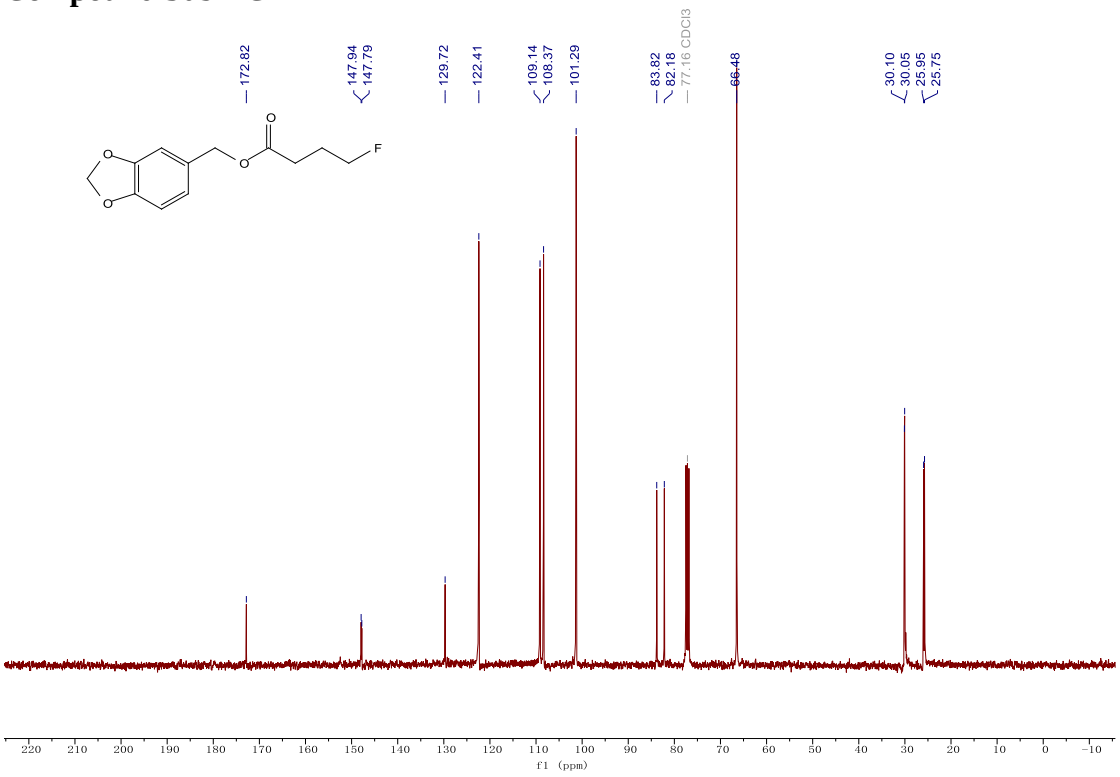
Compound 49b ^{19}F NMR



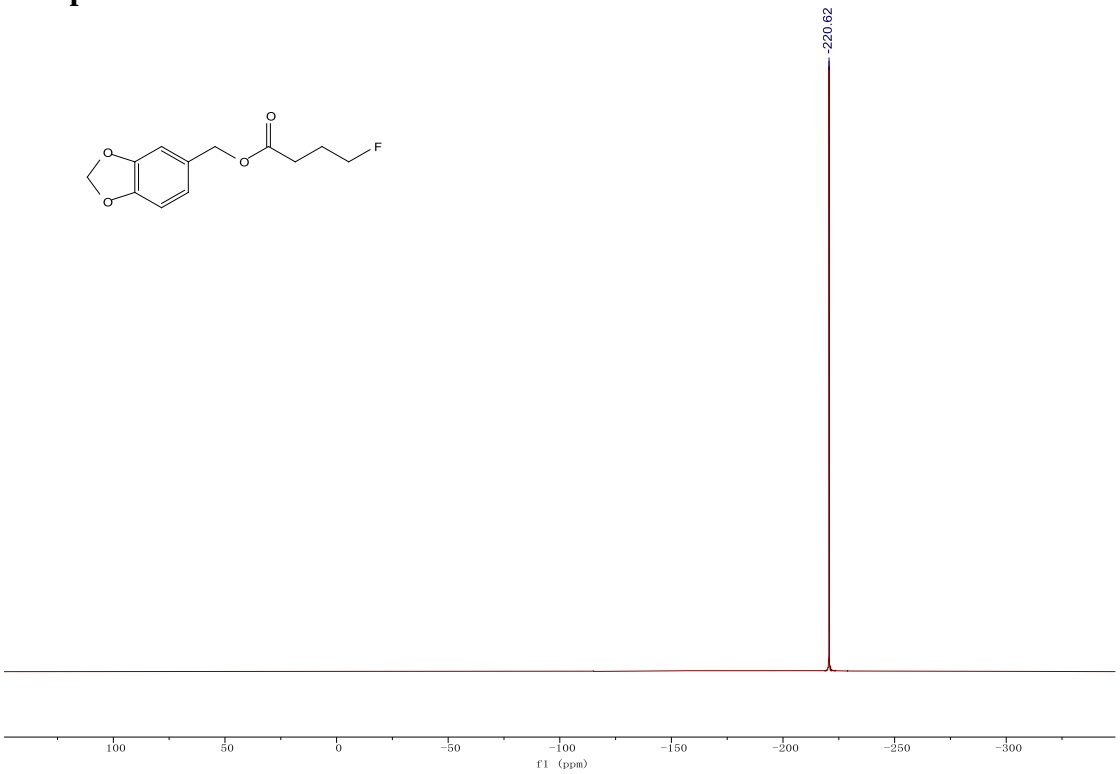
Compound 50b ^{1}H NMR



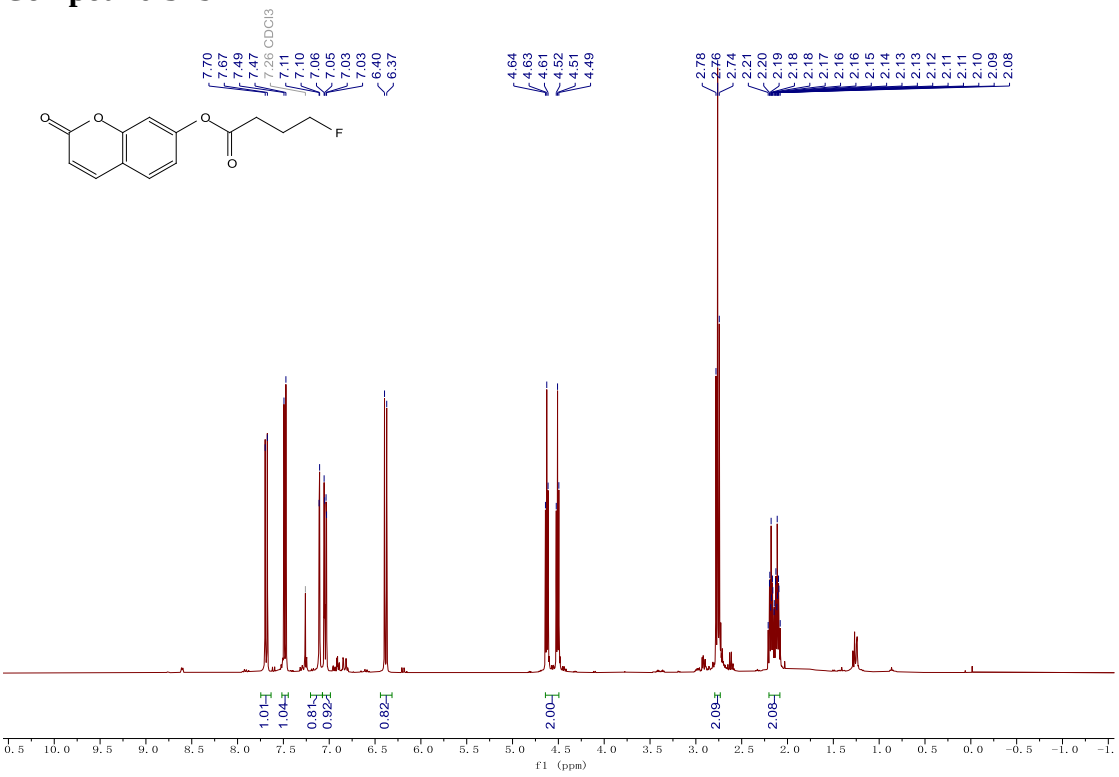
Compound 50b ^{13}C NMR



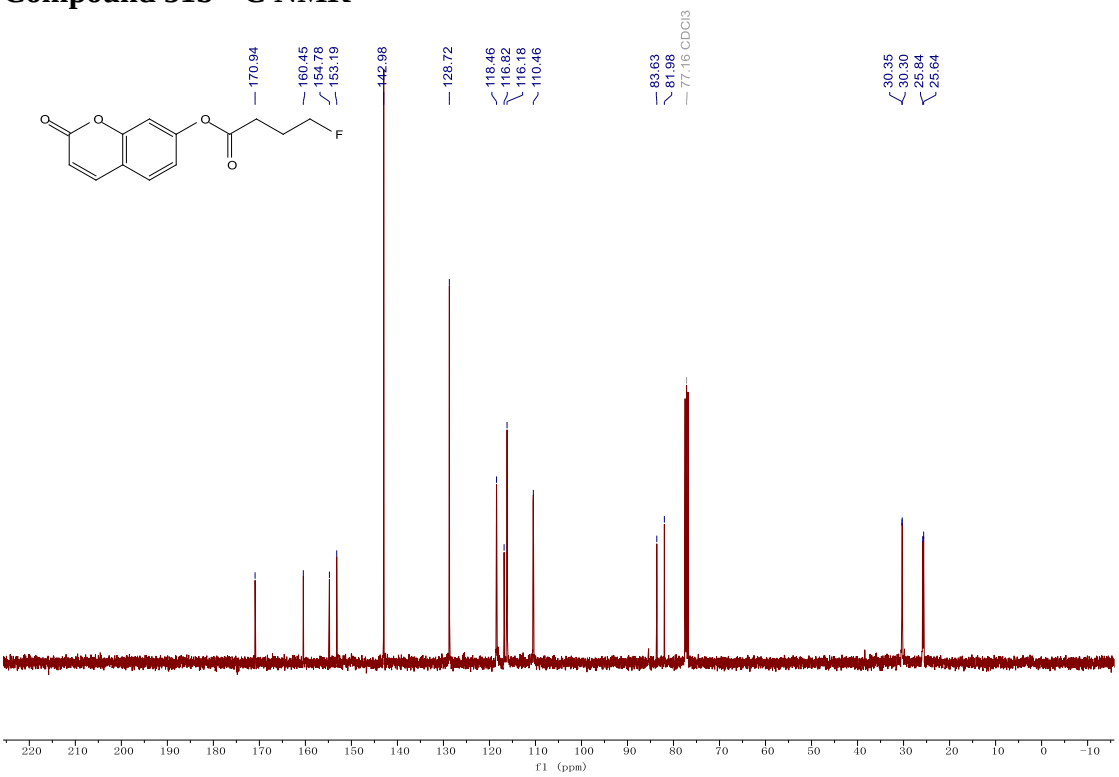
Compound 50b ^{19}F NMR



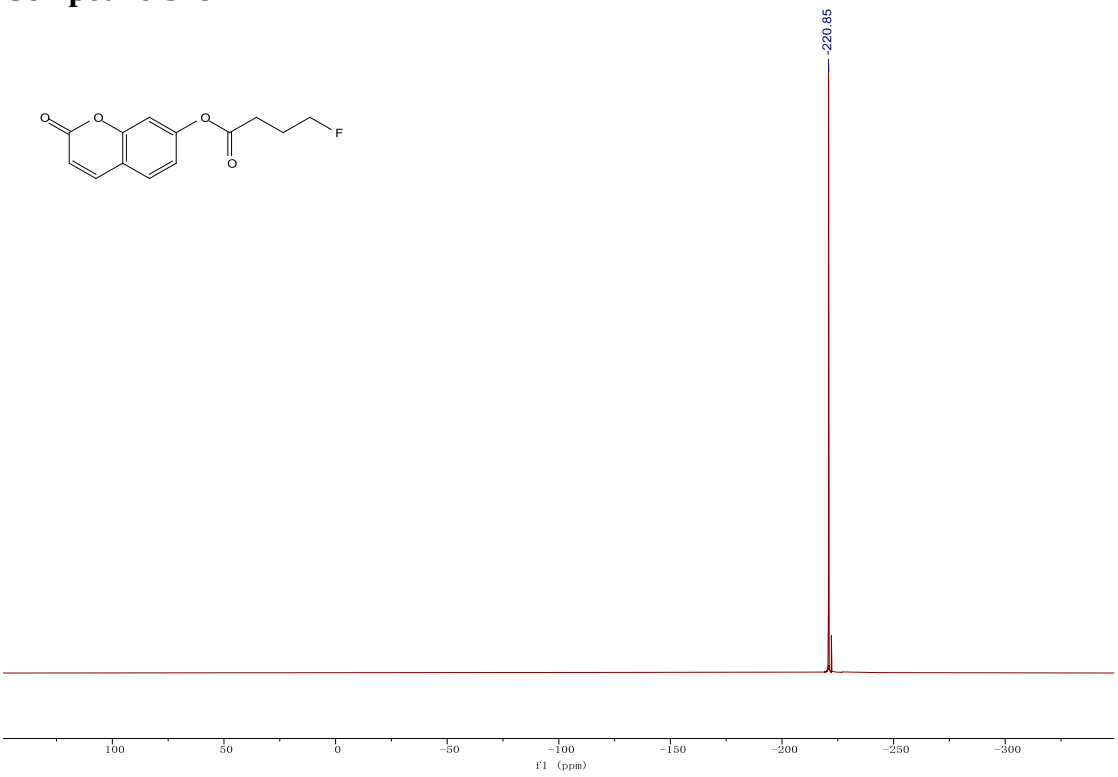
Compound 51b ^{1}H NMR



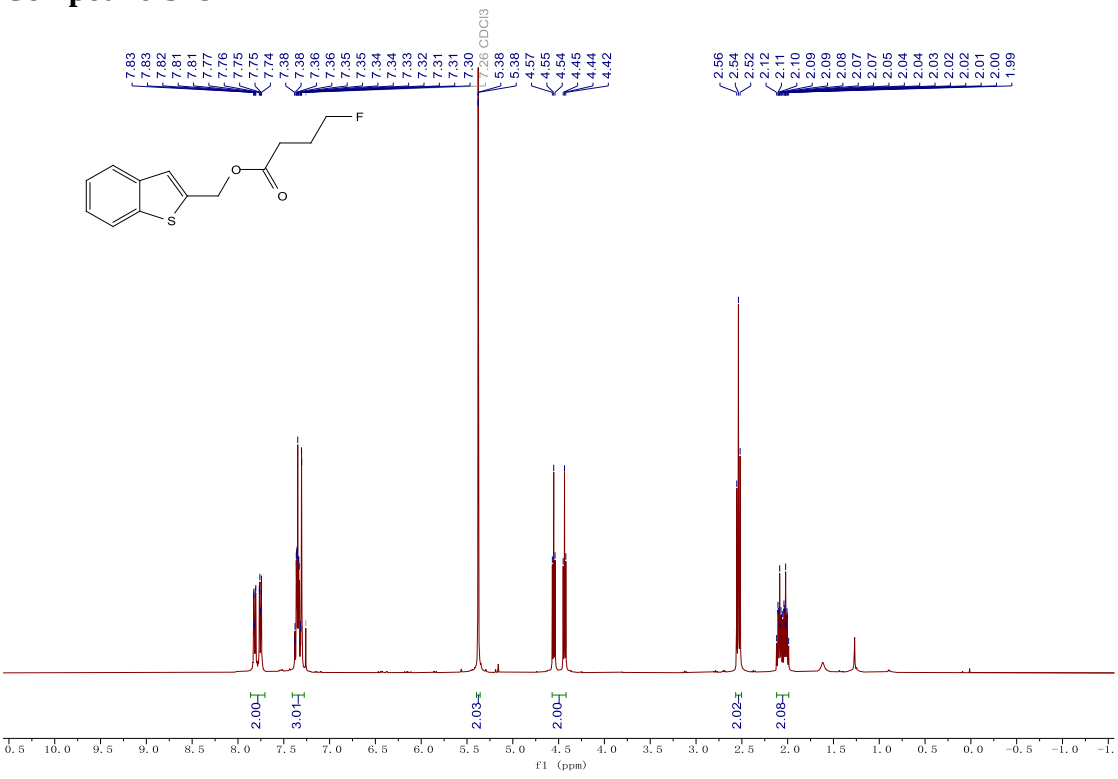
Compound 51b ^{13}C NMR



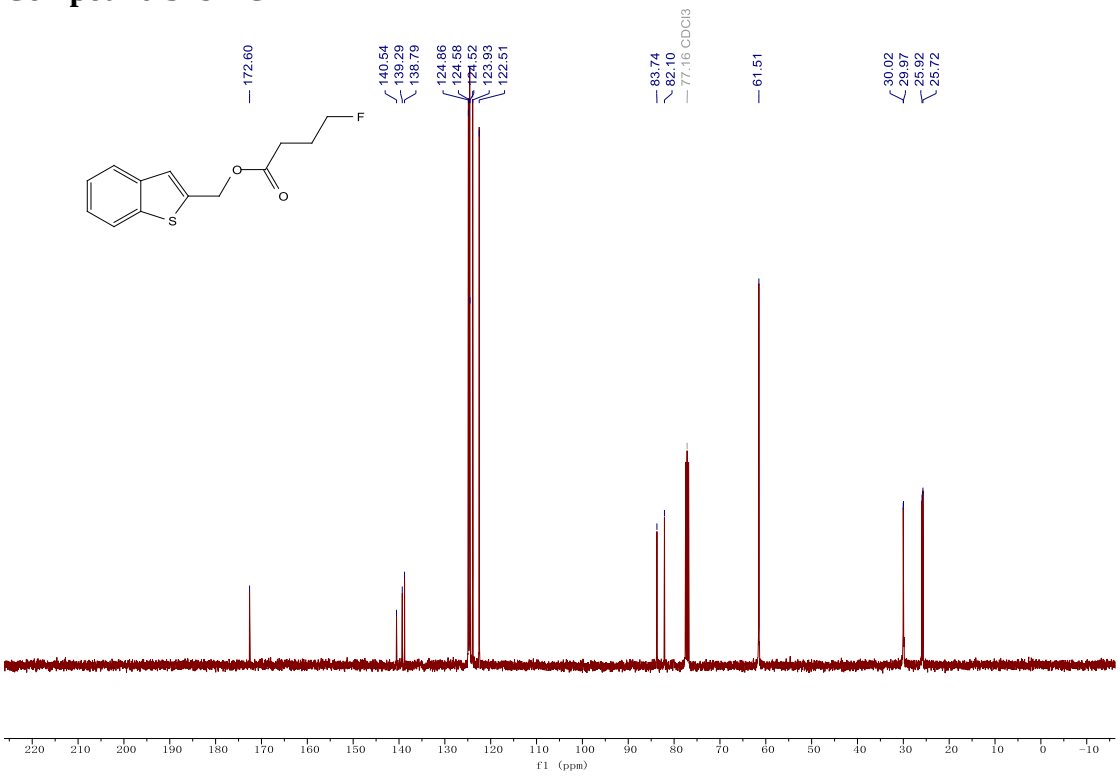
Compound 51b ^{19}F NMR



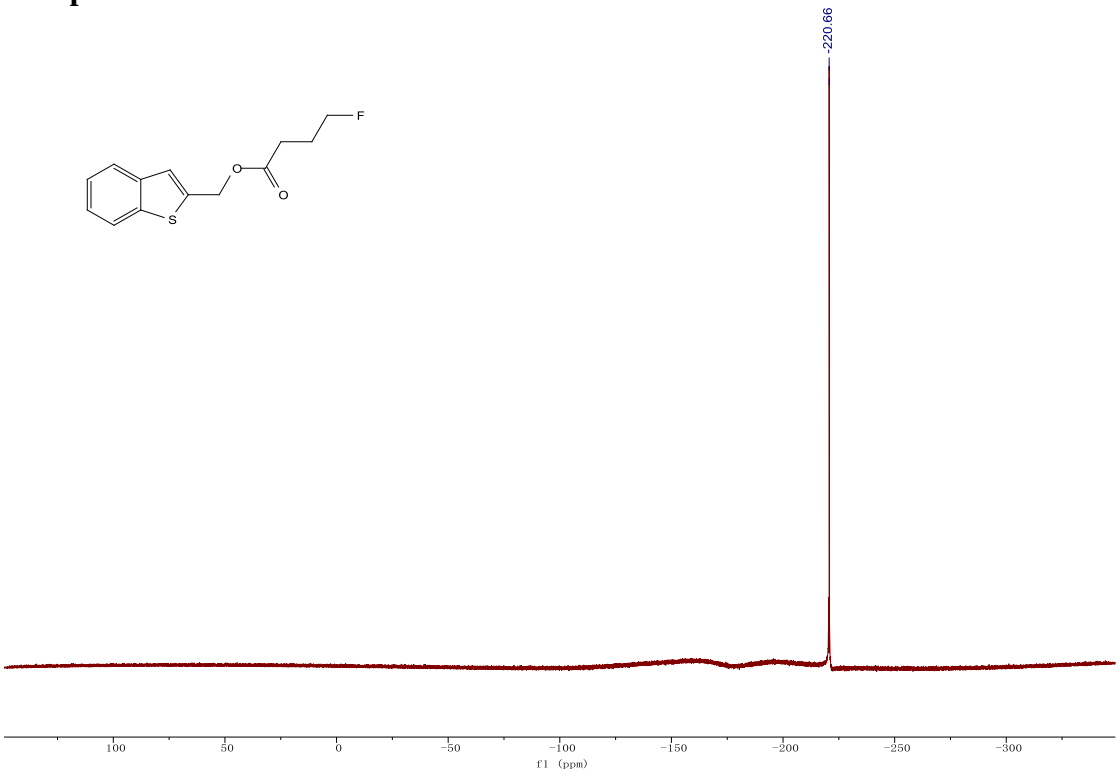
Compound 52b ^{1}H NMR



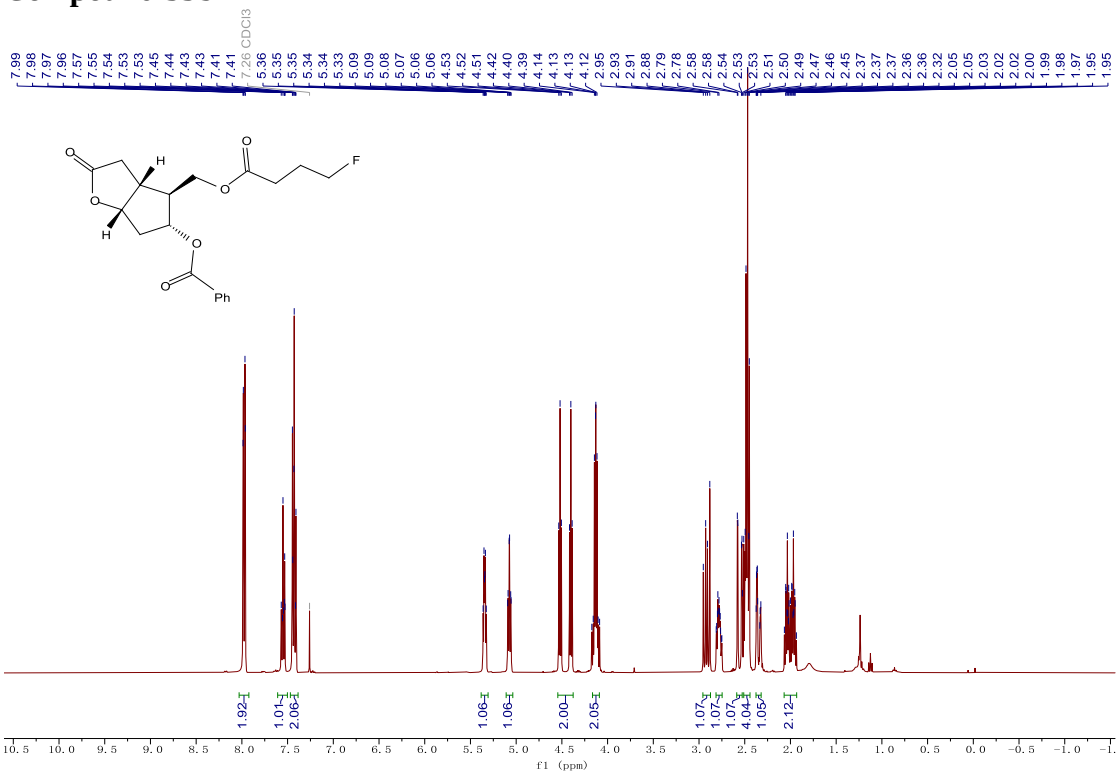
Compound 52b ^{13}C NMR



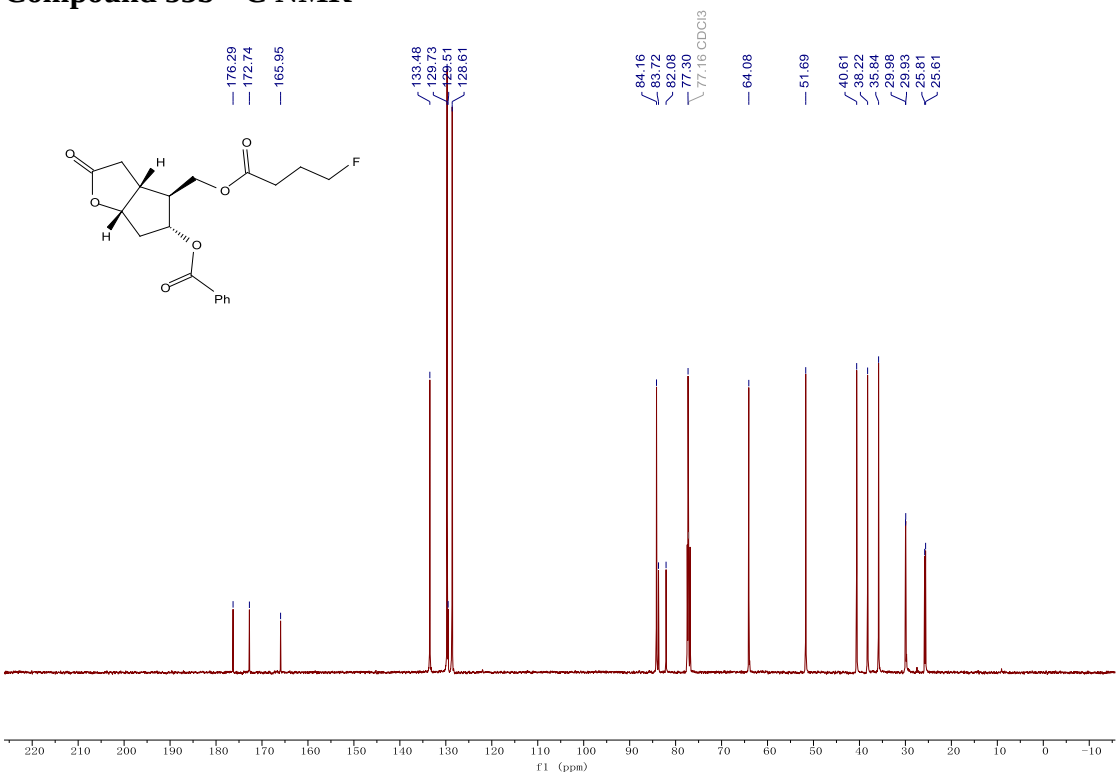
Compound 52b ^{19}F NMR



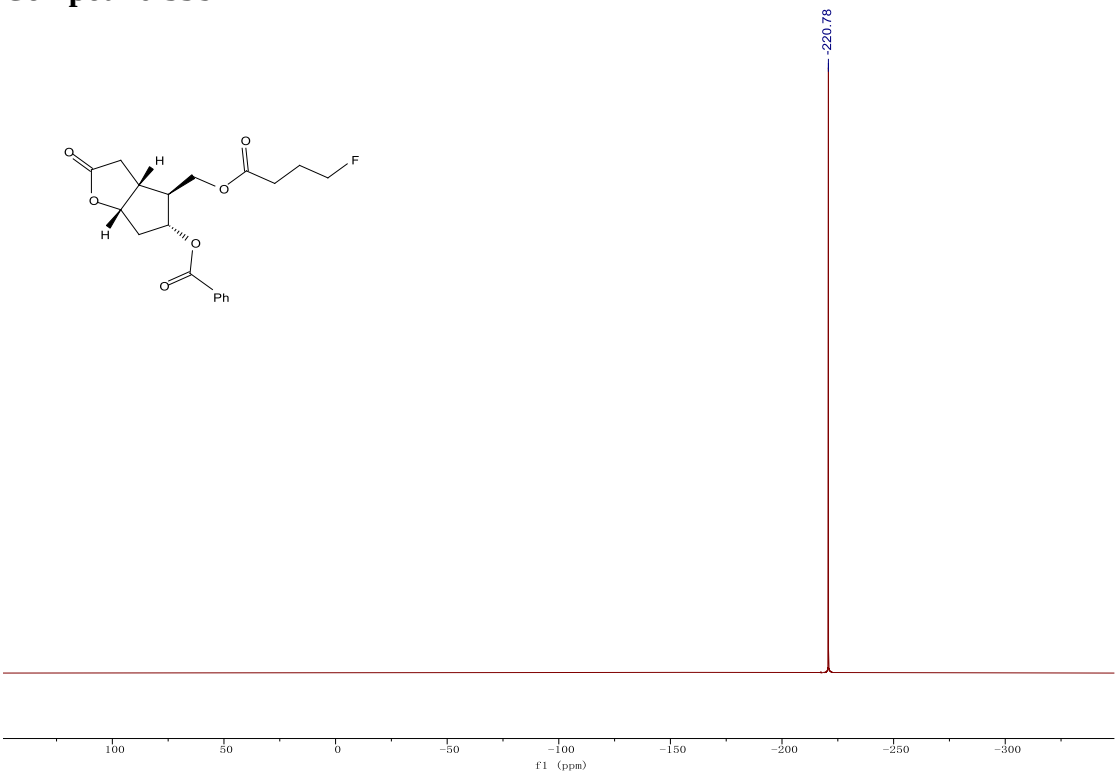
Compound 53b ^{1}H NMR



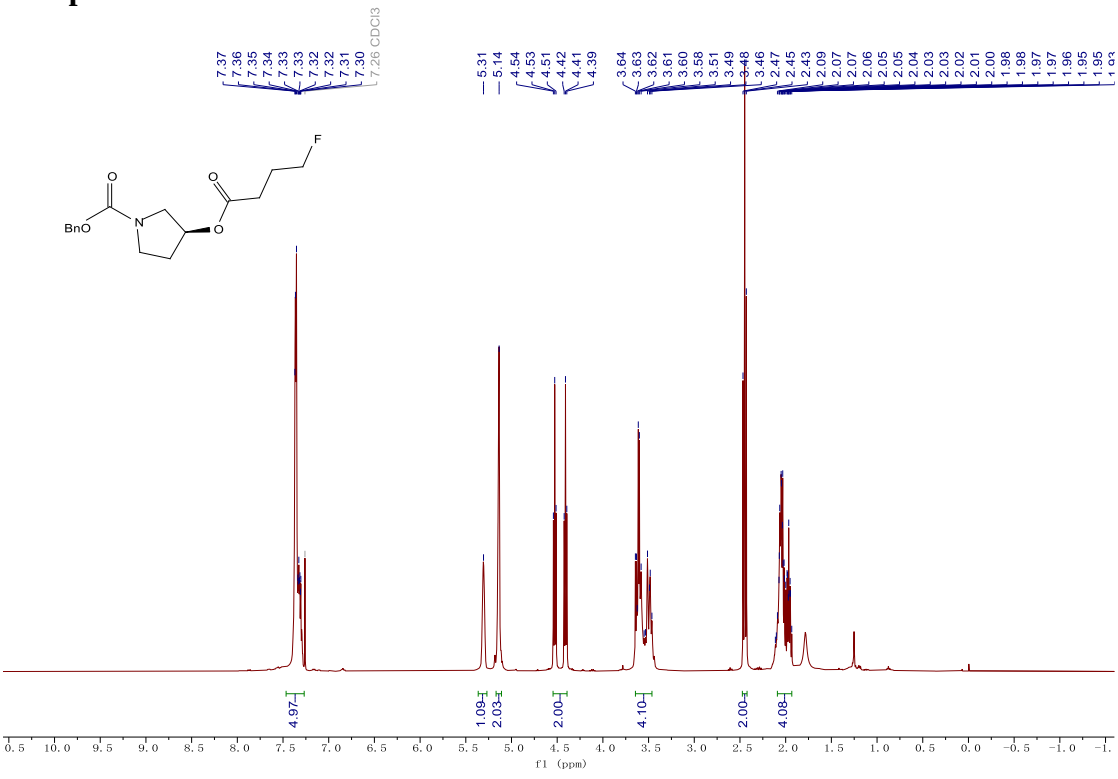
Compound 53b ^{13}C NMR



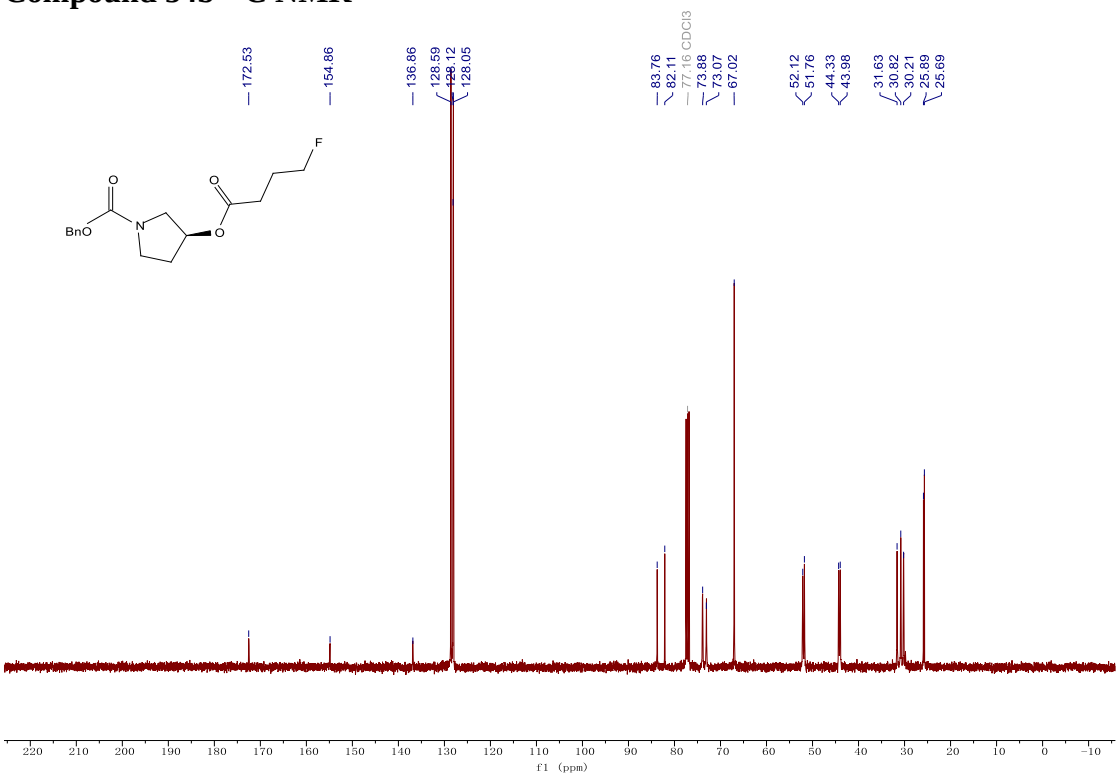
Compound 53b ^{19}F NMR



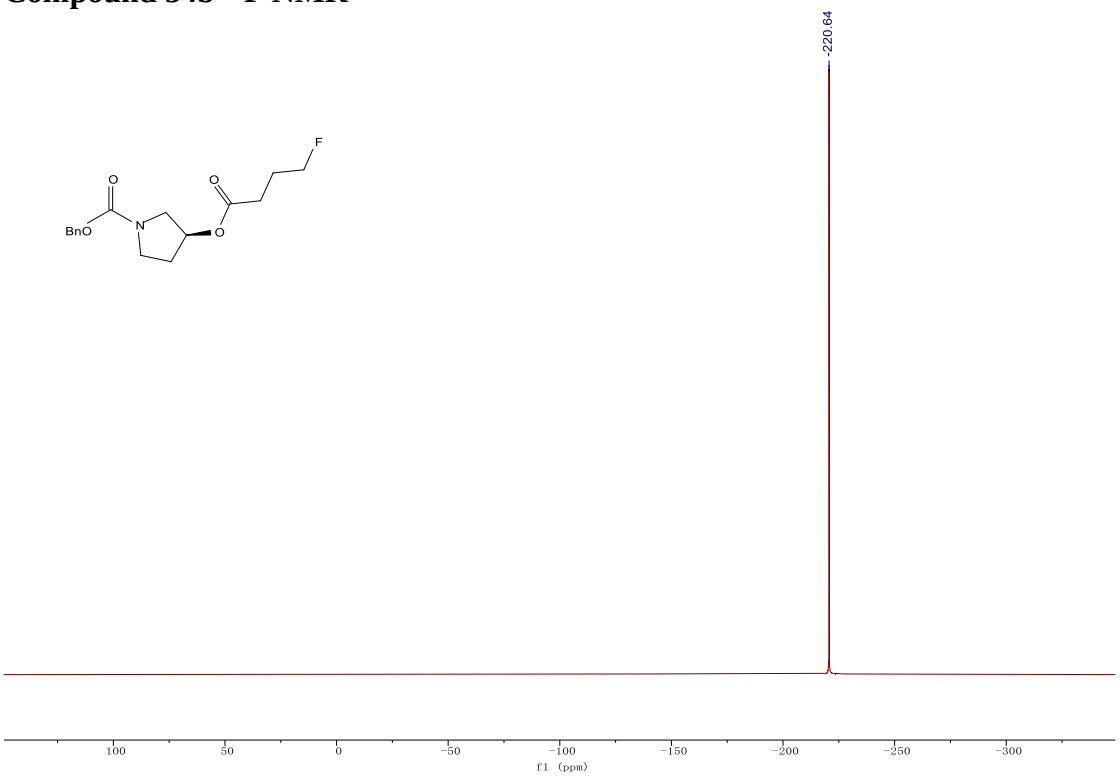
Compound 54b ^{1}H NMR



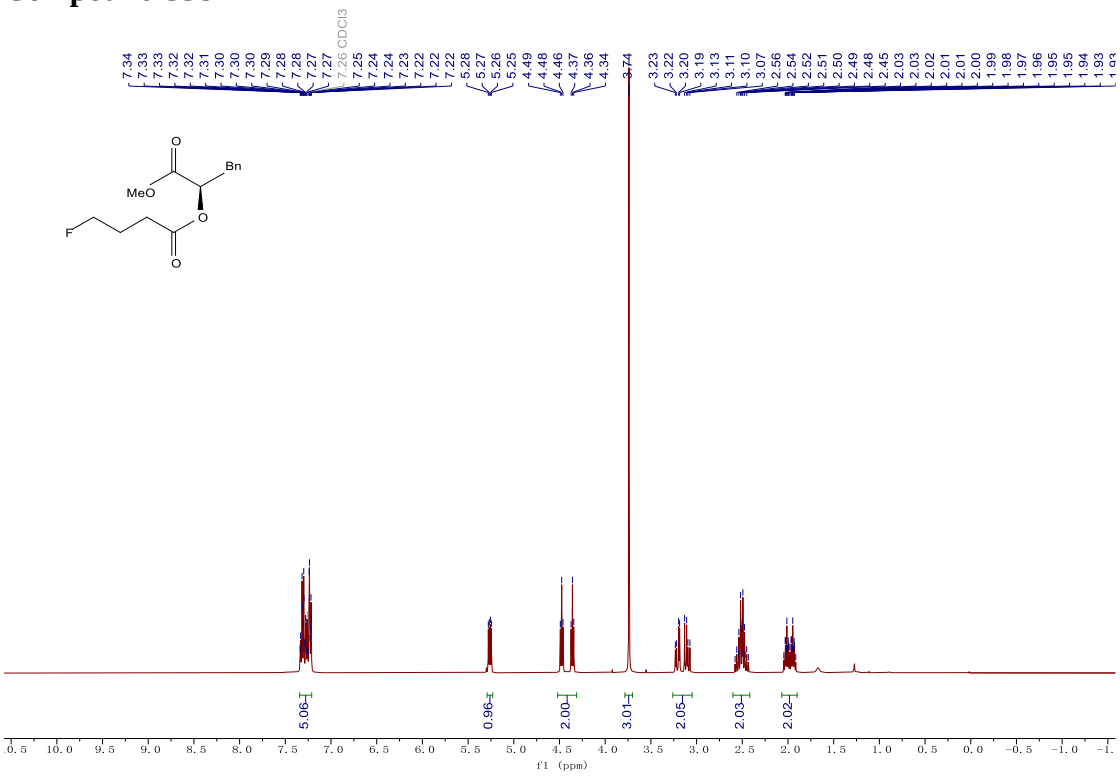
Compound 54b ^{13}C NMR



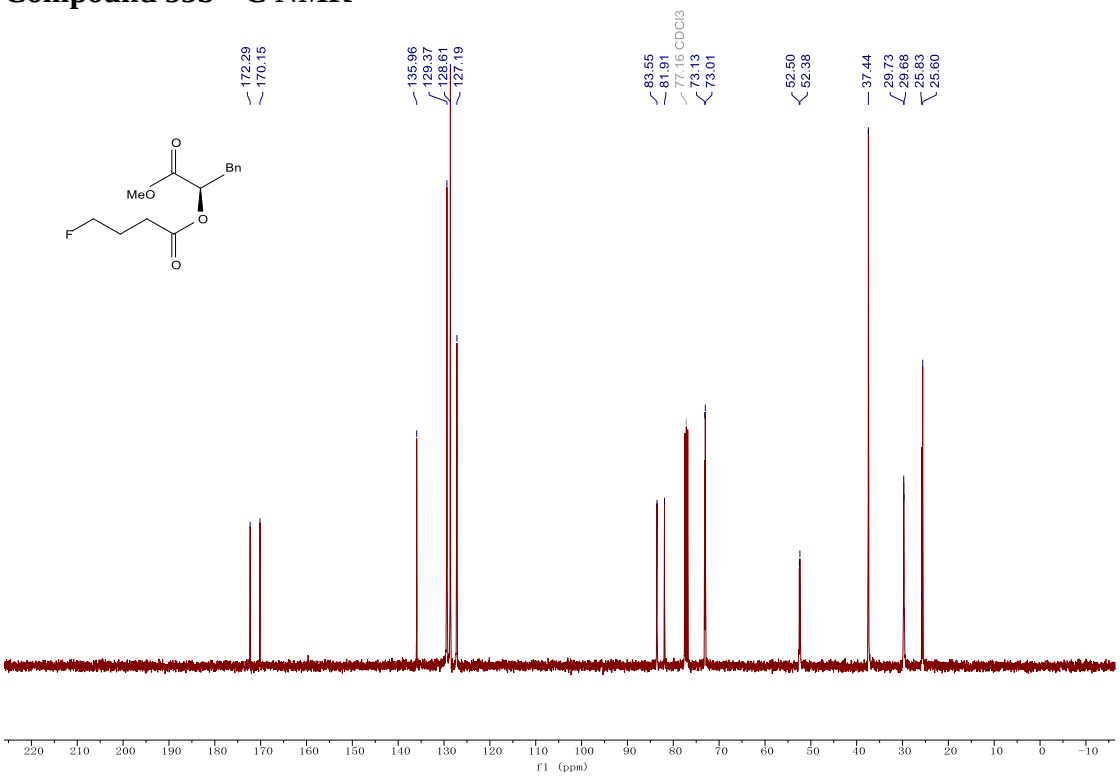
Compound 54b ^{19}F NMR



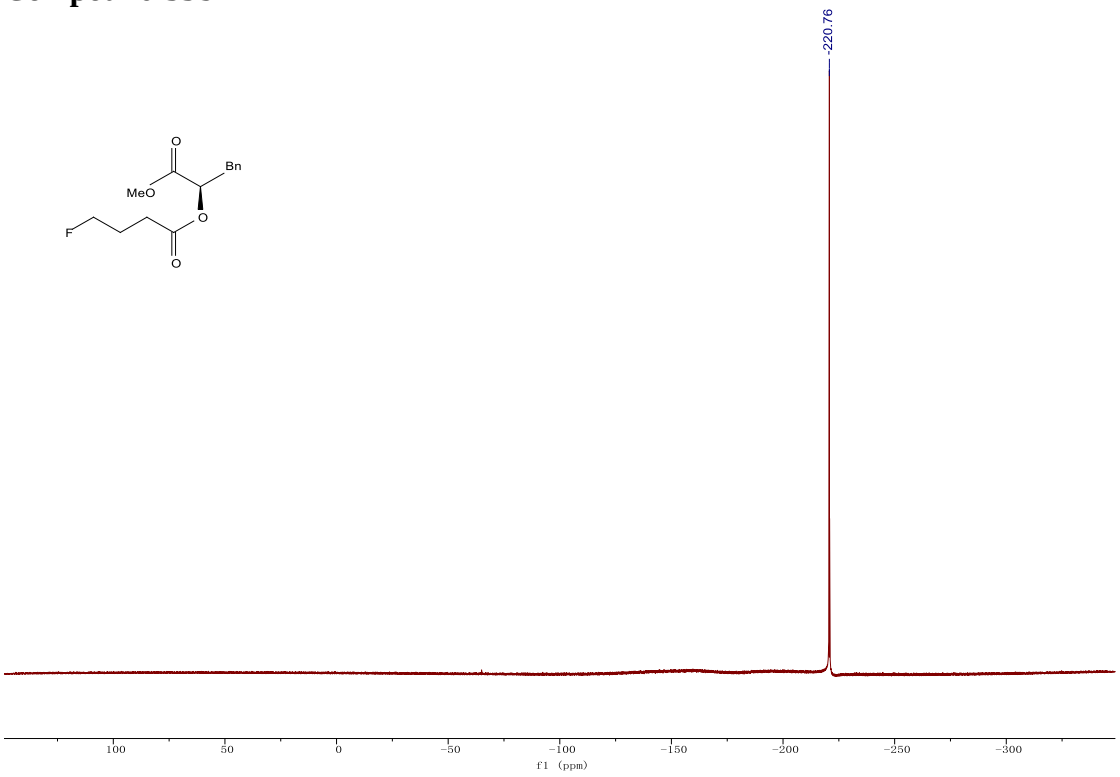
Compound 55b ^{1}H NMR



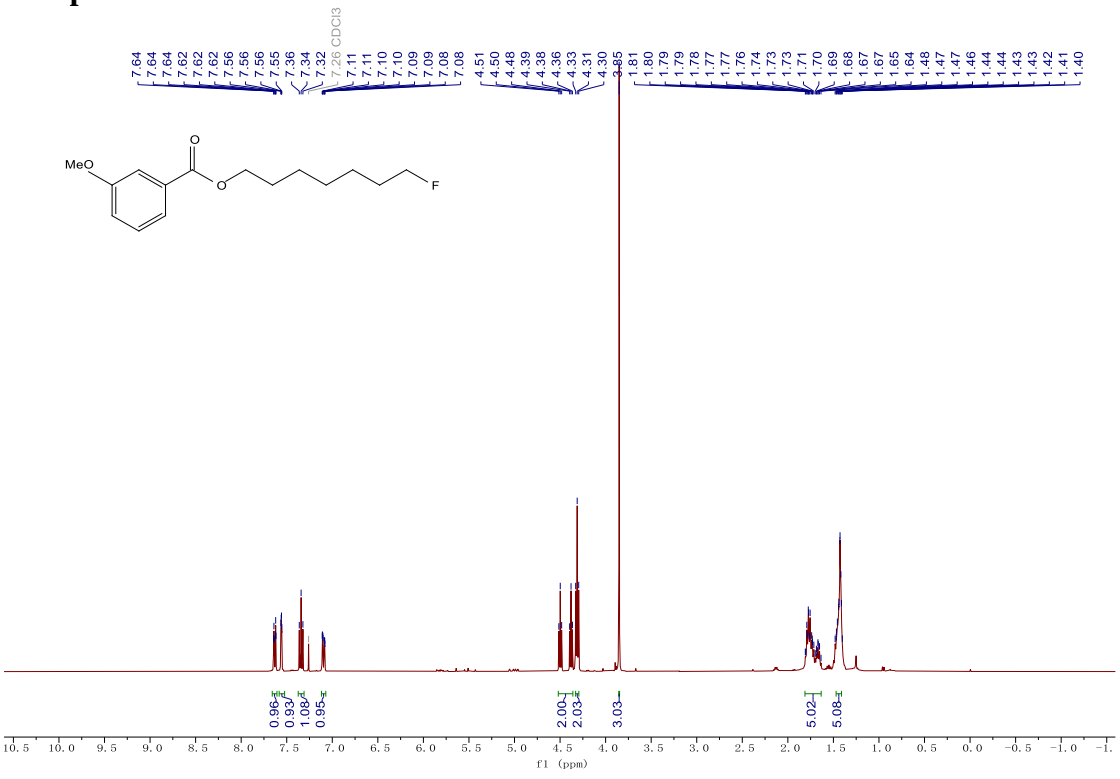
Compound 55b ^{13}C NMR



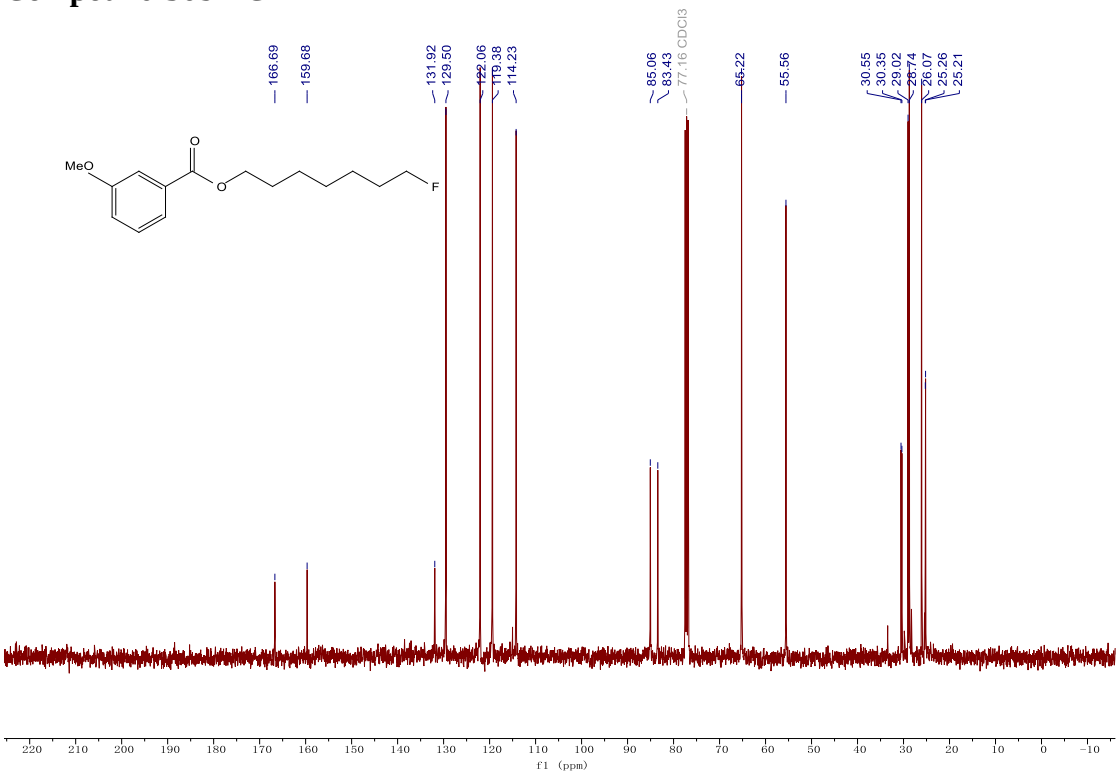
Compound 55b ^{19}F NMR



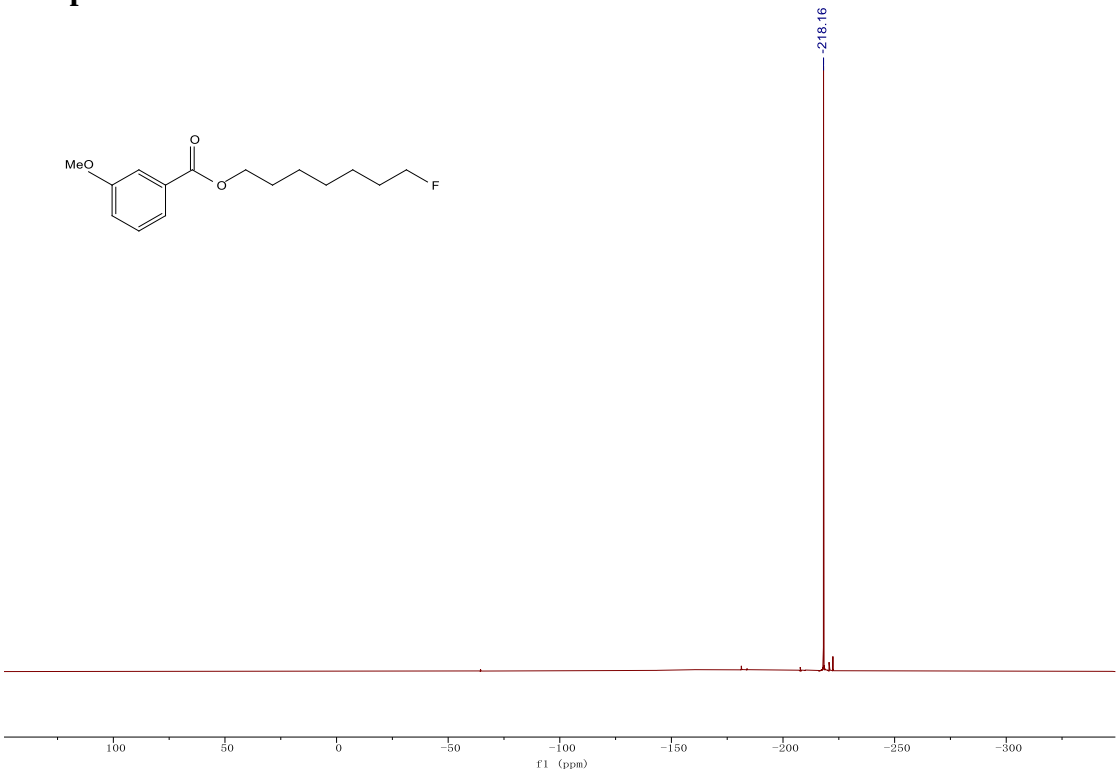
Compound 56b ^{1}H NMR



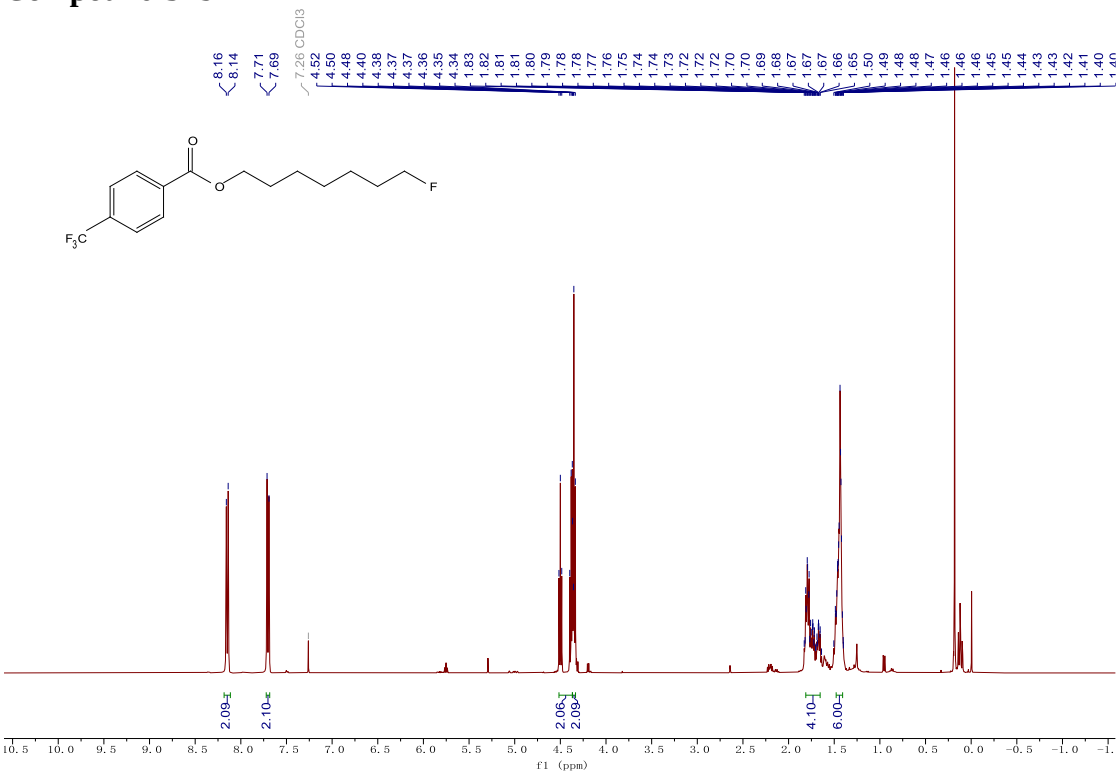
Compound 56b ^{13}C NMR



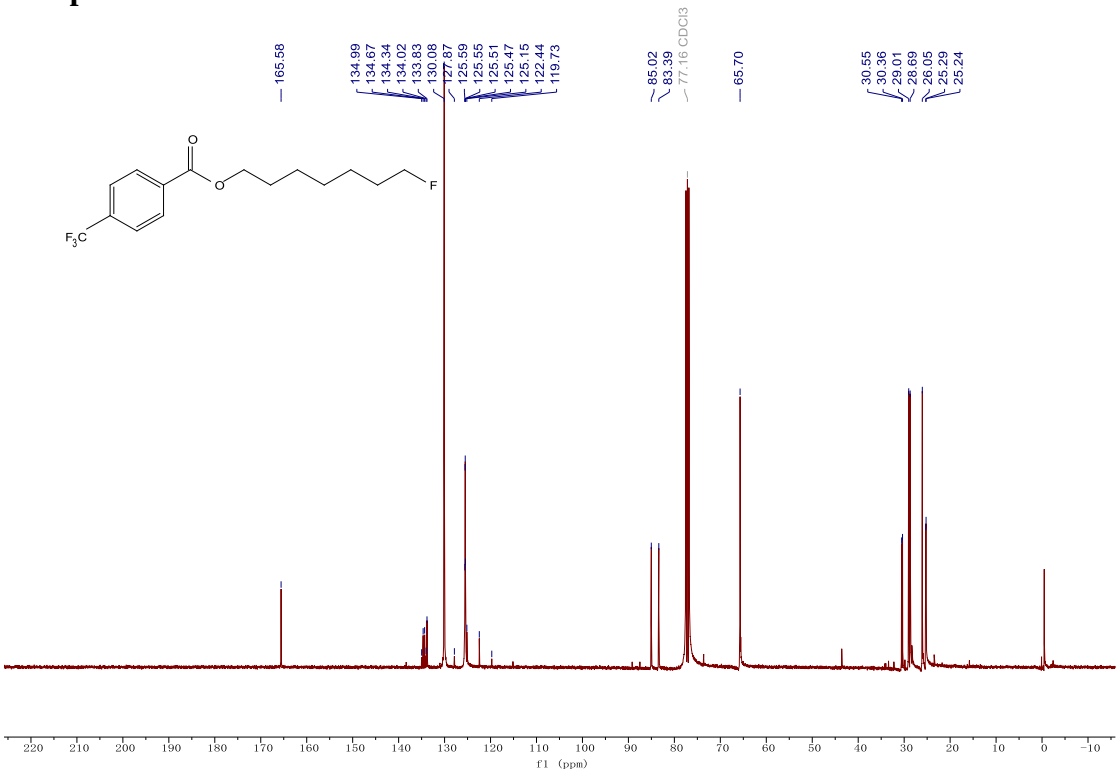
Compound 56b ^{19}F NMR



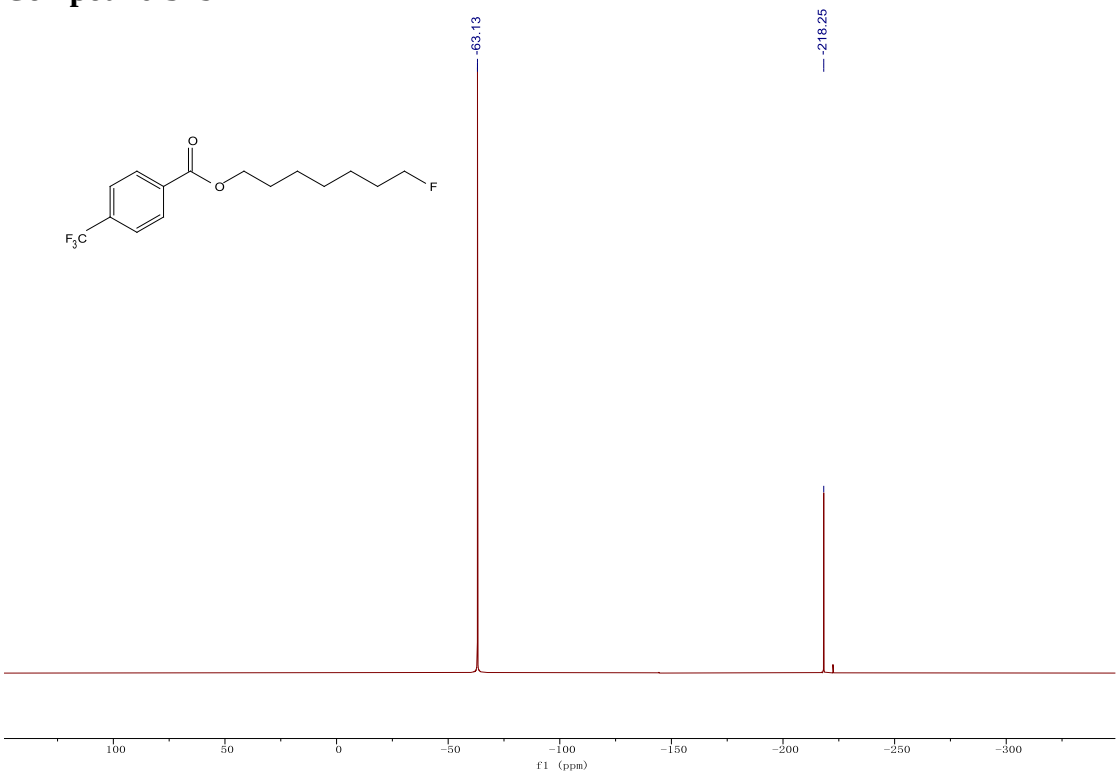
Compound 57b ^{1}H NMR



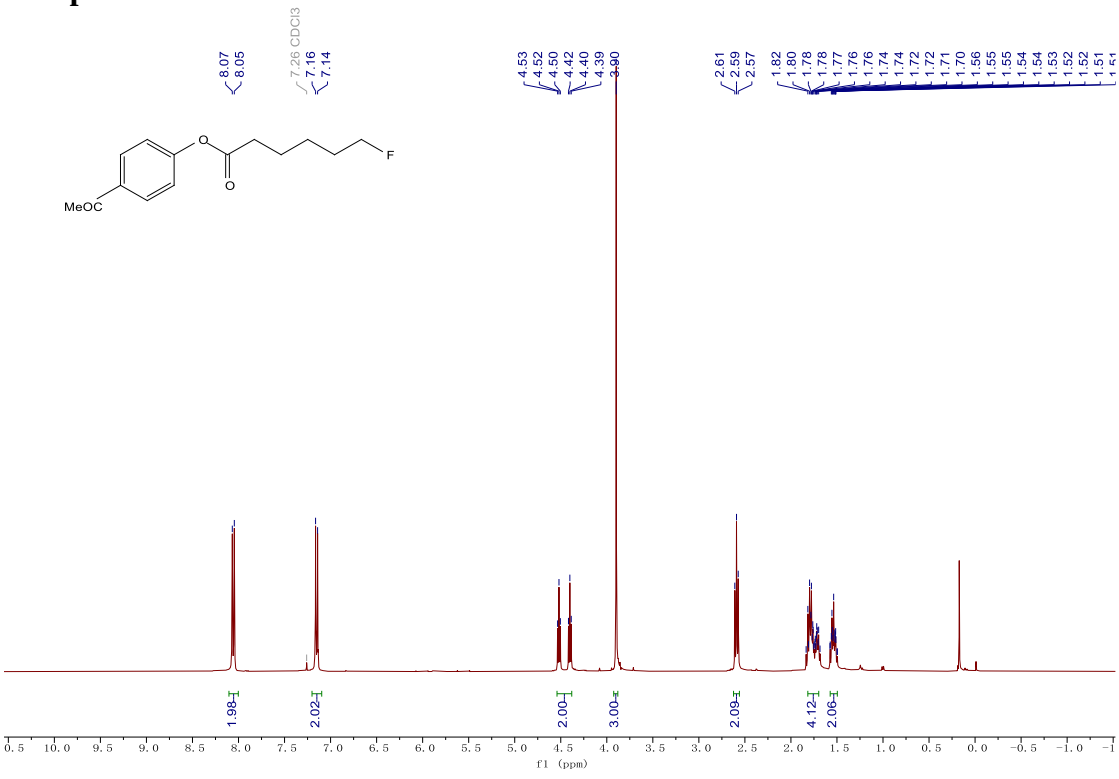
Compound 57b ^{13}C NMR



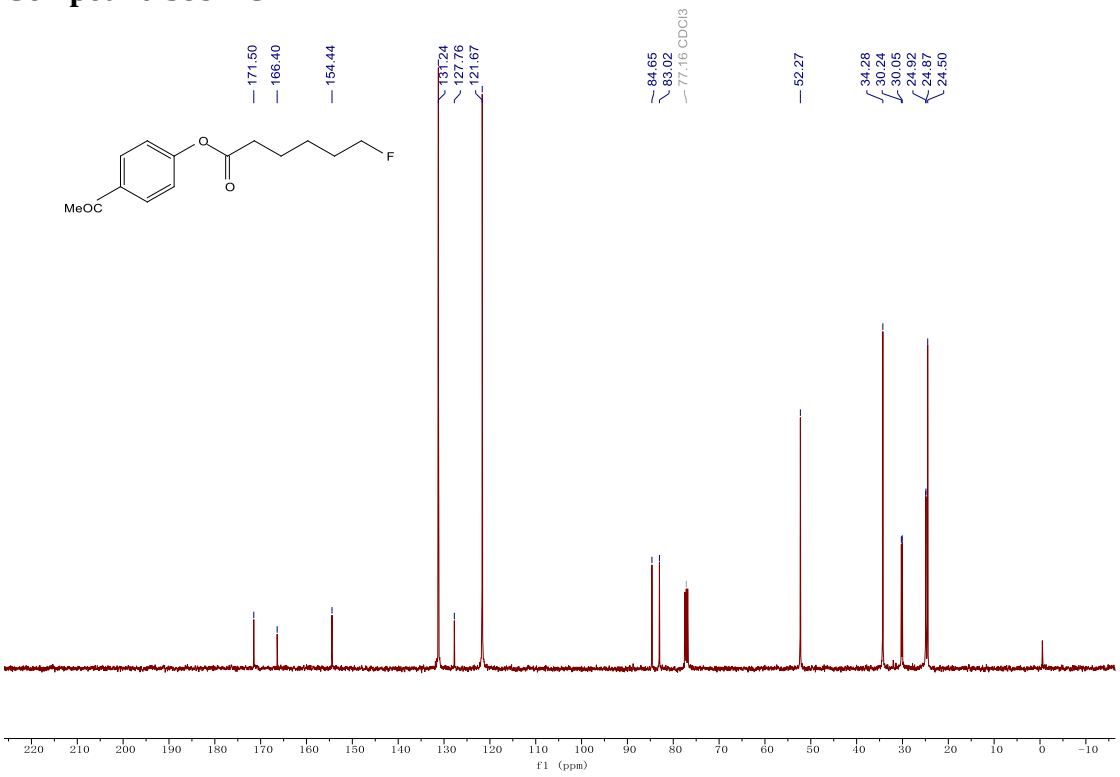
Compound 57b ^{19}F NMR



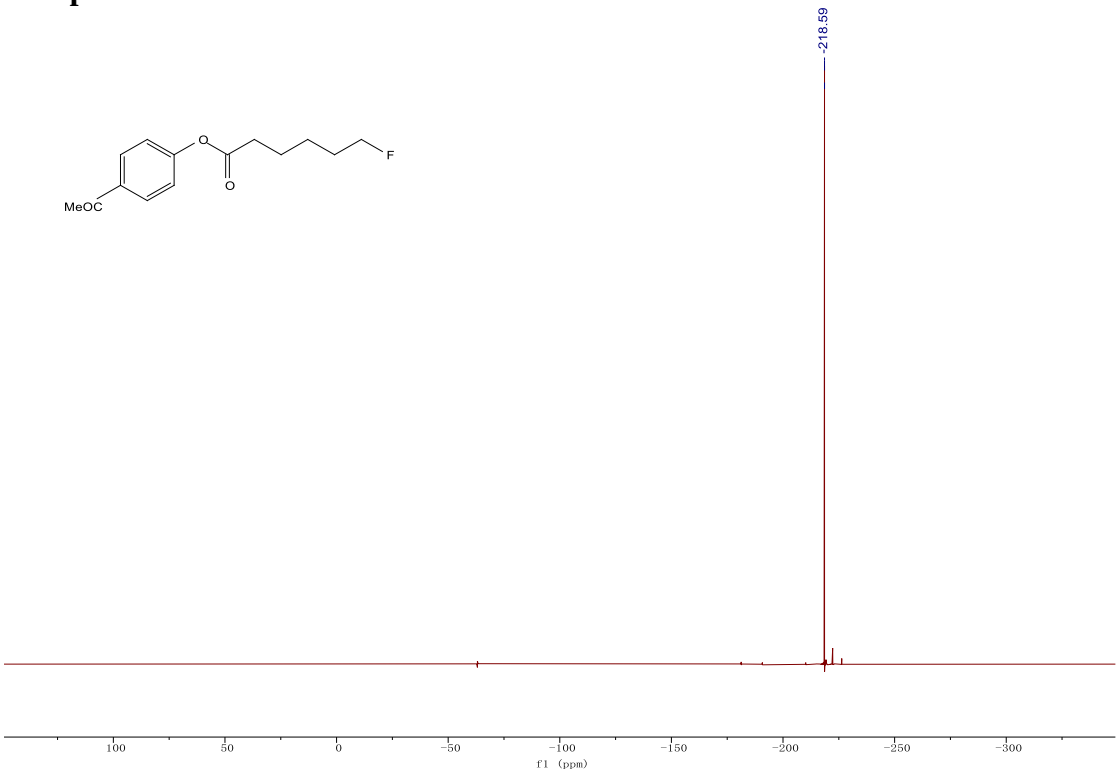
Compound 58b ^{1}H NMR



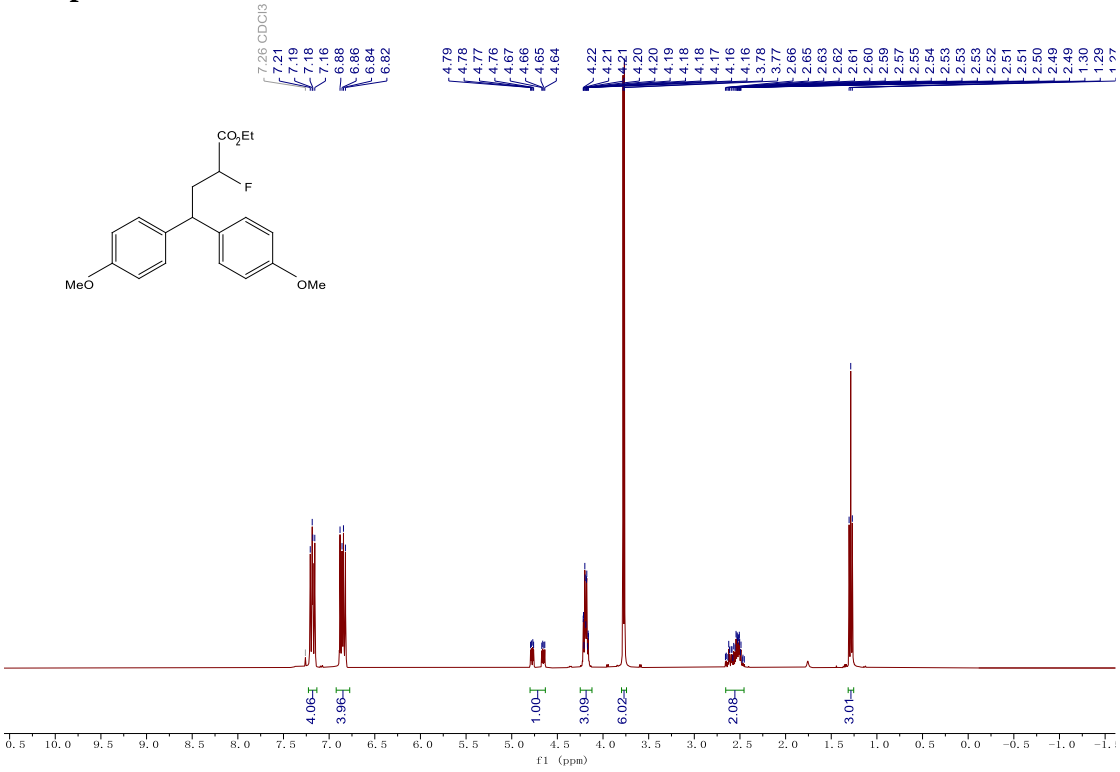
Compound 58b ^{13}C NMR



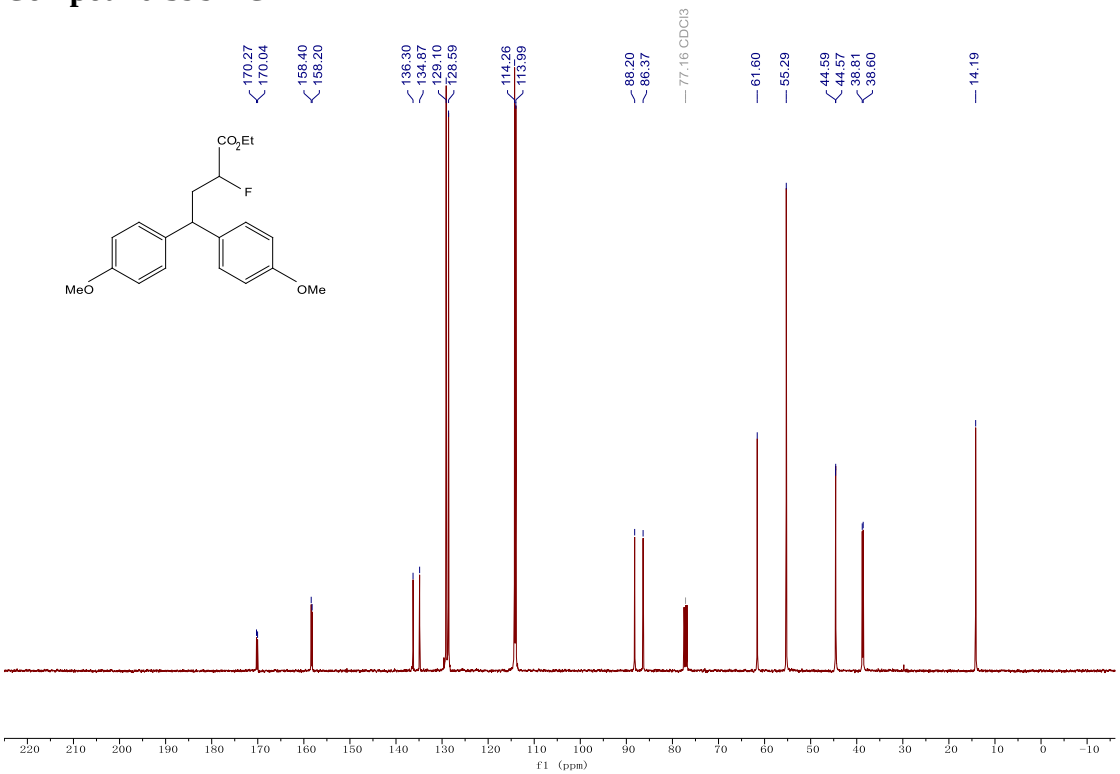
Compound 58b ^{19}F NMR



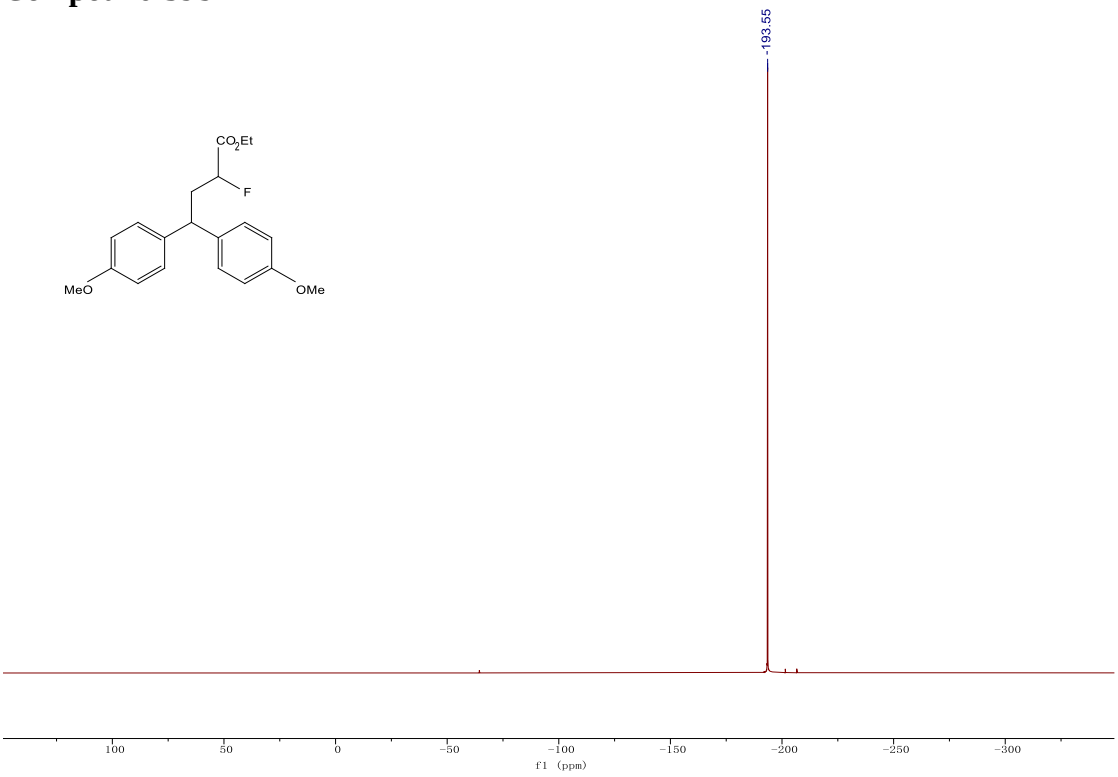
Compound 59b ^{1}H NMR



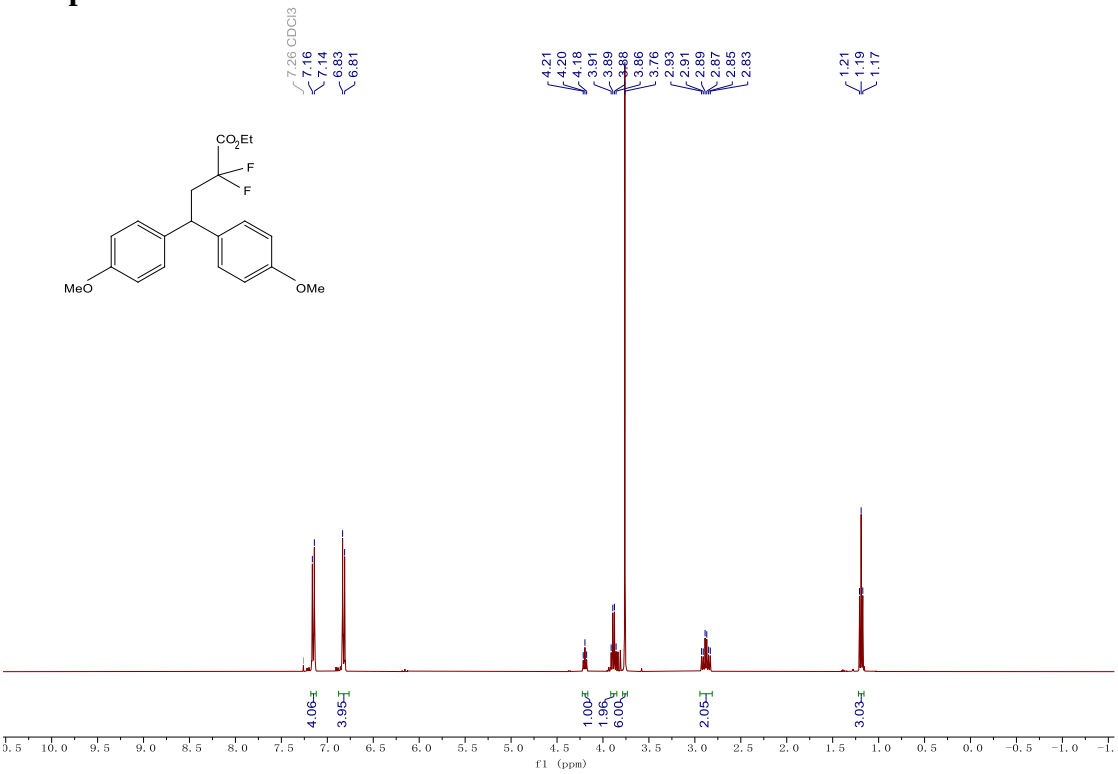
Compound 59b ^{13}C NMR



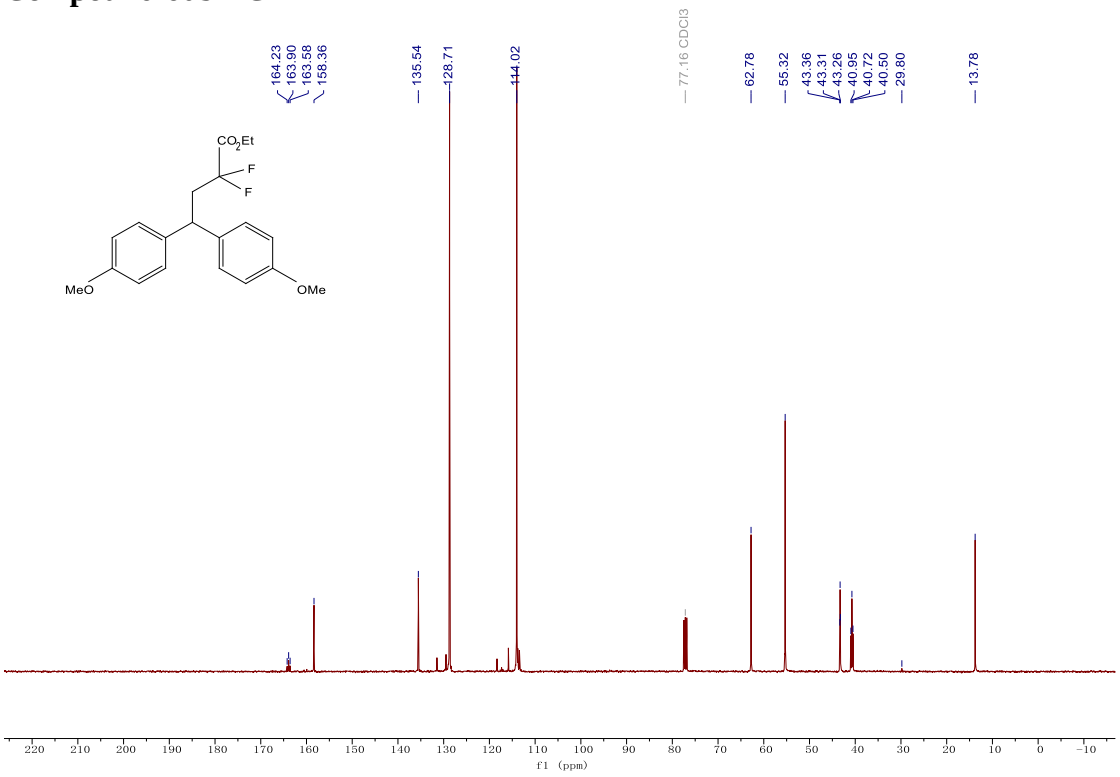
Compound 59b ^{19}F NMR



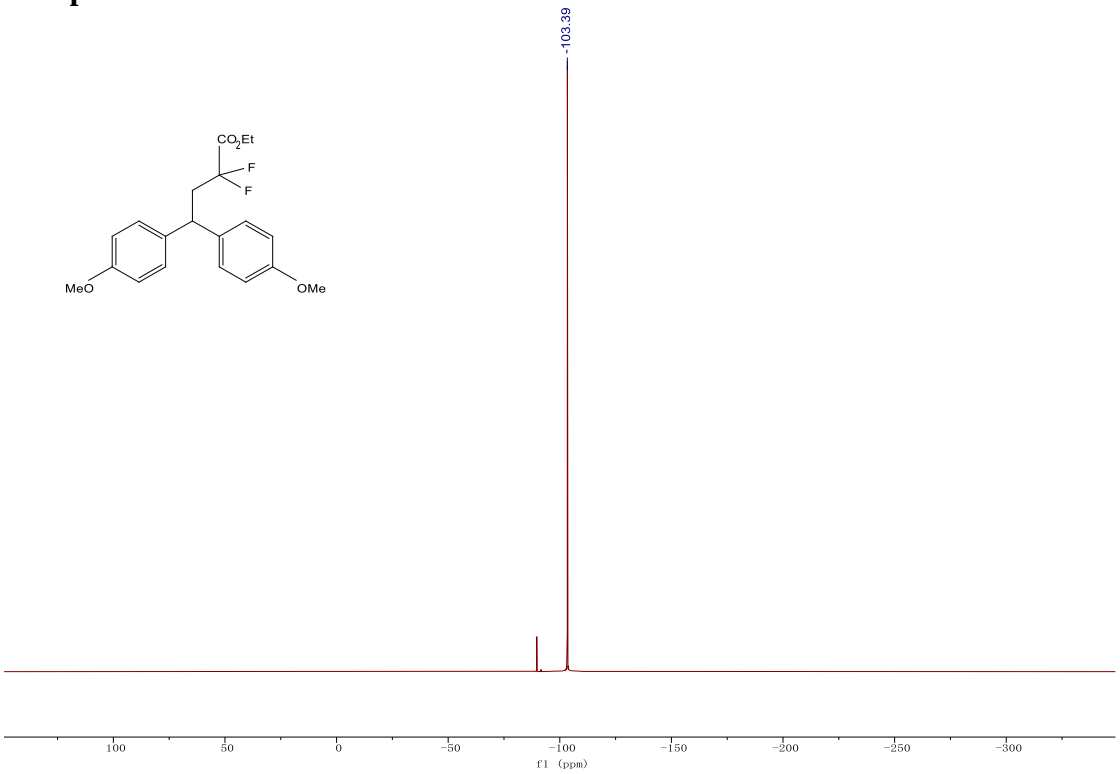
Compound 60b ^{1}H NMR



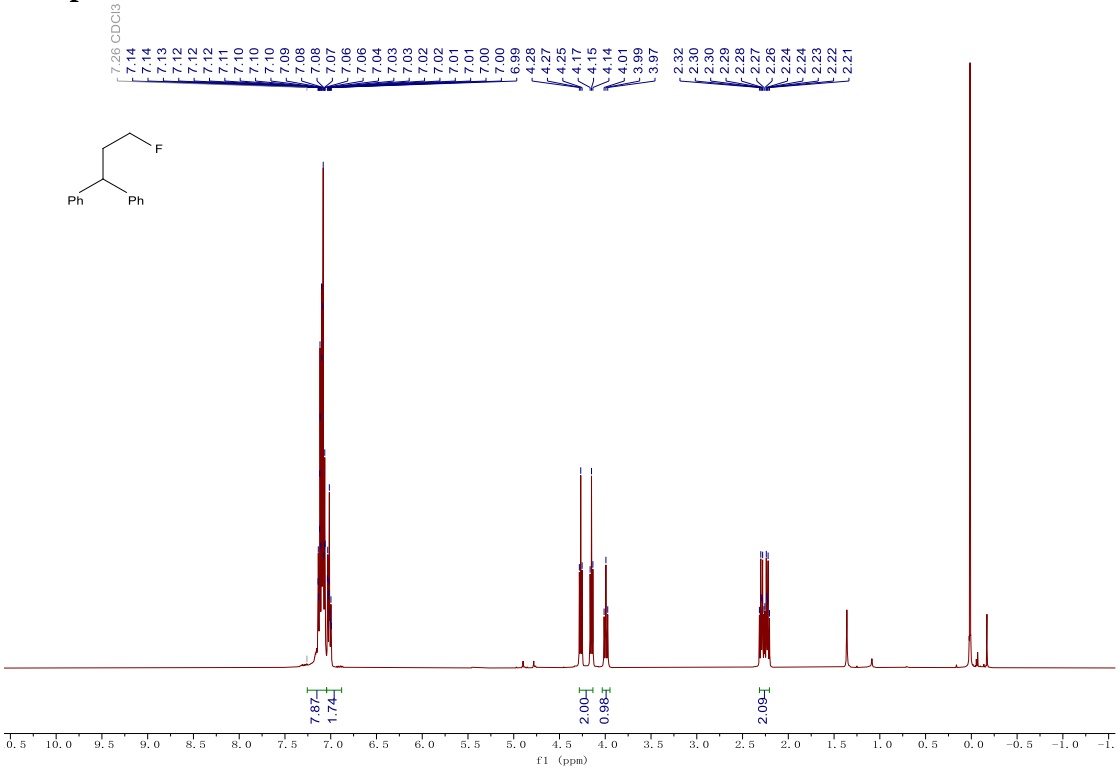
Compound 60b ^{13}C NMR



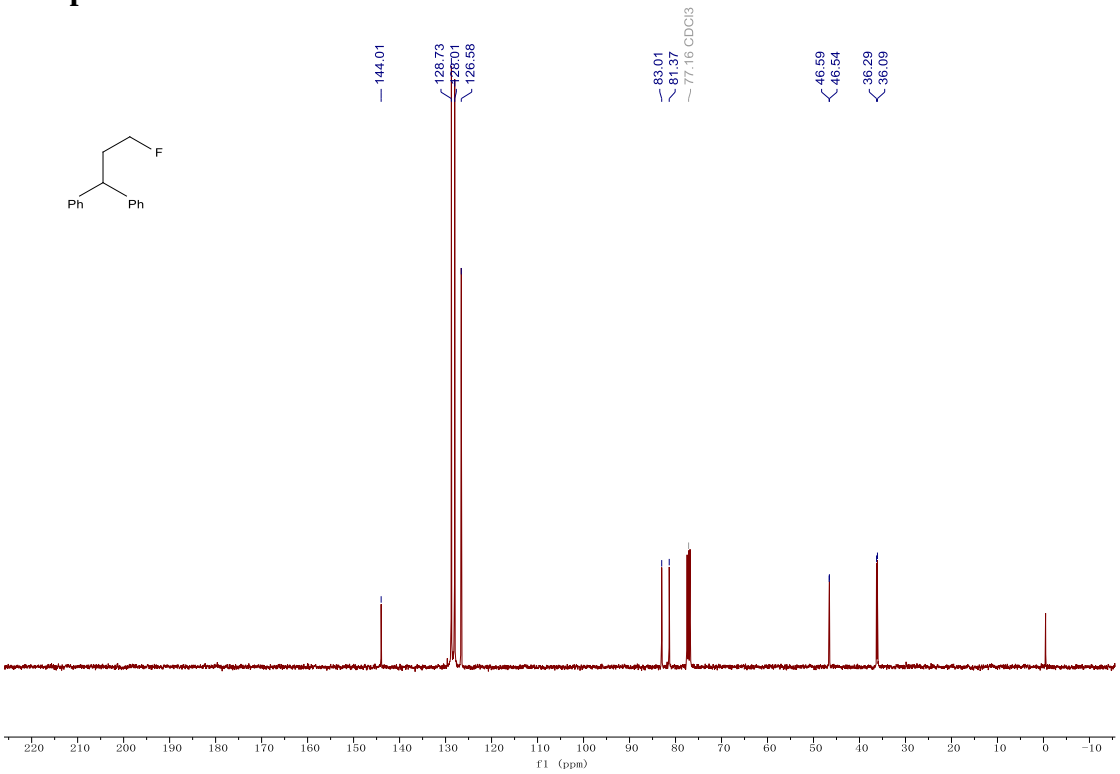
Compound 60b ^{19}F NMR



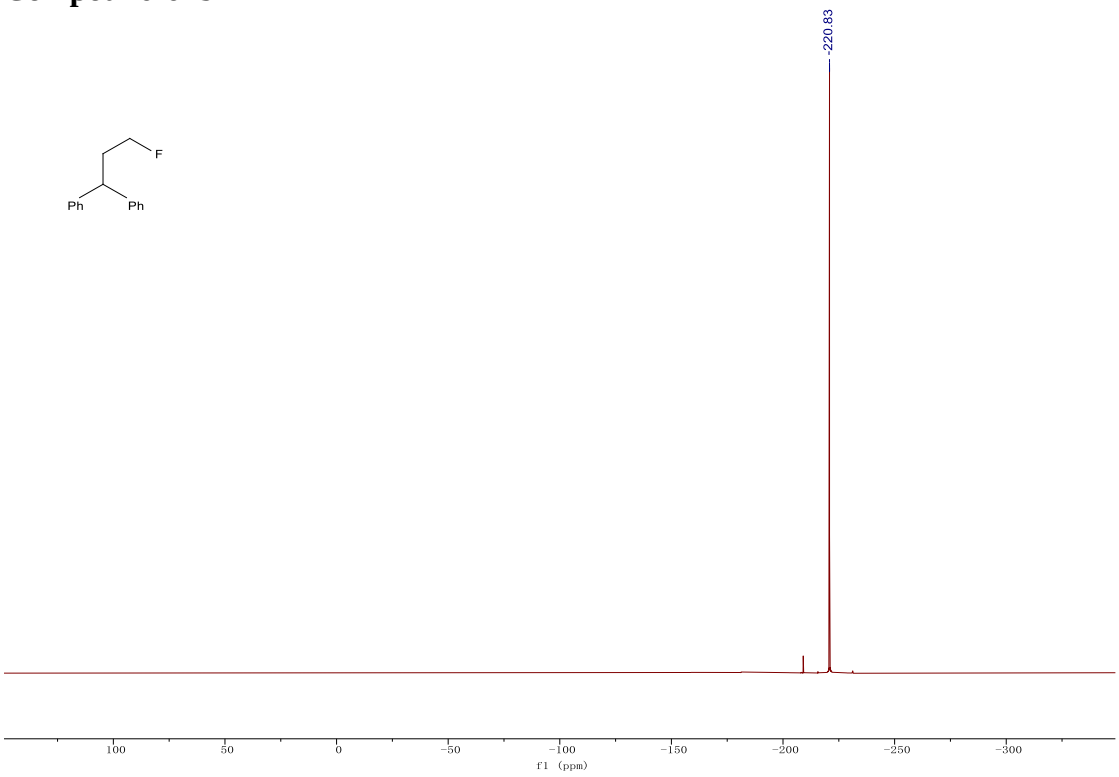
Compound 61b ^{1}H NMR



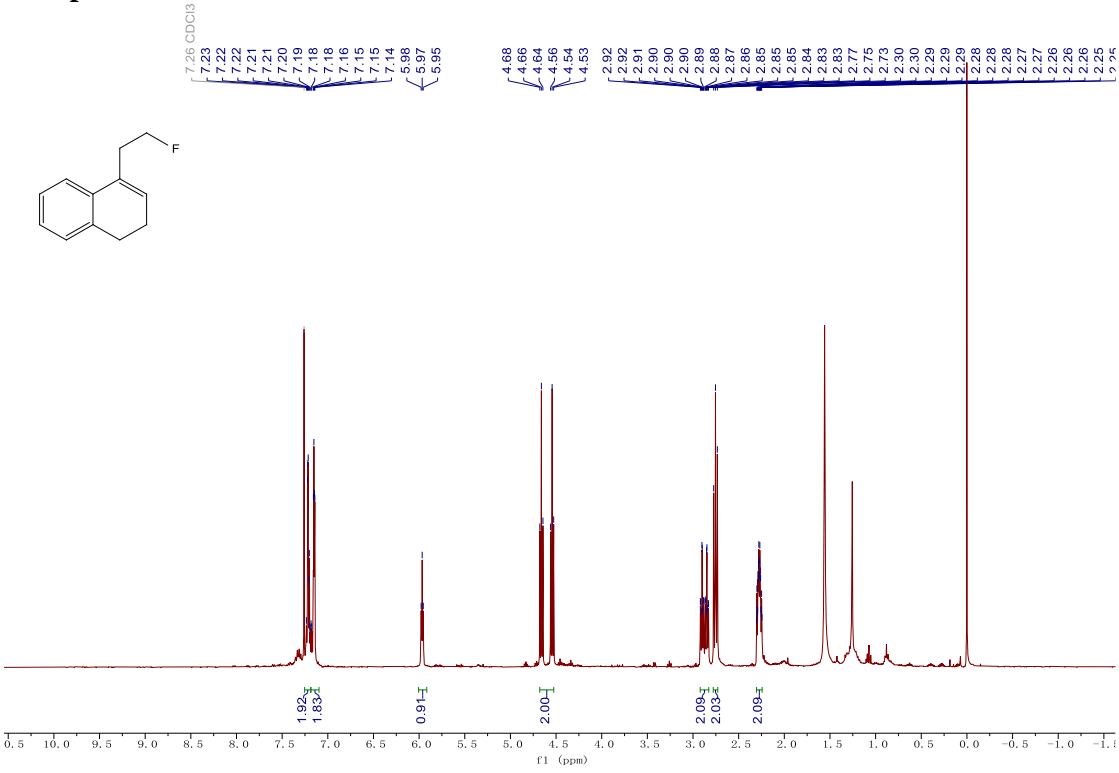
Compound 61b ^{13}C NMR



Compound 61b ^{19}F NMR



Compound 62b ^{1}H NMR



Compound 62b ^{19}F NMR