Electronic Supplementary Information for:

Copper-mediated pentafluoroethylation of organoboronates and terminal alkynes with $TMSCF_3$

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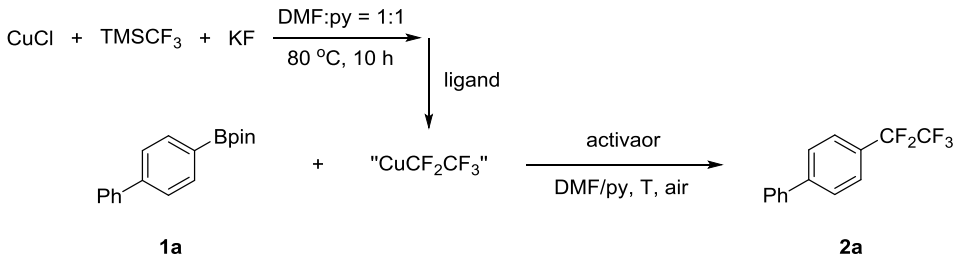
1. General Information

Unless otherwise mentioned, all solvents and reagents were purchased from commercial sources and used as received. *N*,*N*-dimethylformamide (DMF), tetrahydrofuran (THF) and toluene were dried by passing through a solvent purification system. Dry pyridine (Py), hexamethylphosphoramide (HMPA), dimethyl sulfoxide (DMSO), 1,3-dimethyl-3,4,5,6-tetra-hydro-2(1*H*)-pyrimidinone (DMPU) and 1-methyl-2-pyrrolidinone (NMP) were obtained through distillation over CaH_2. CuCl was purified according to: W. L. F. Armarego, D. D. Perrin, *Purification of Laboratory Chemicals*, 5th ed.; Butterworth Heinemann: Oxford, **1997**. All the melting points were uncorrected. ^{1}H NMR spectra were recorded at 400 or 500 MHz. ^{19}F NMR spectra were recorded at 376 MHz. ^{13}C NMR spectra were recorded at 100 or 126 MHz. ^{1}H NMR chemical shifts were determined relative to internal $(CH_3)_4Si$ (TMS) at δ 0.00 ppm or to the signal of the residual protonated solvent: DMSO-d_6 at δ 2.50 ppm, $CDCl_3$ at δ 7.26 ppm. ^{19}F NMR chemical shifts were determined relative to internal or external $CFCl_3$ at δ 0.00 ppm or $PhCF_3$ at δ -63.00 ppm. ^{13}C NMR chemical shifts were determined relative to the signal of the solvent: $CDCl_3$ at δ 77.16 ppm, DMSO-d_6 at δ 39.50 ppm. Data for ^{1}H, ^{13}C, ^{19}F NMR were recorded as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, q = quartet, dd = doublet of doublets, dt = doublet of triplets, td = triplet of doublets, qt = quartet of triplets, tq = triplet of quartets, br = broad). High-resolution mass data were recorded on a high-resolution mass spectrometer in the EI mode, FI positive ion mode or ESI positive ion mode.

2. Optimization of Reaction Conditions

2.1 General Procedures for Optimization of Pentafluoroethylation of Boronates



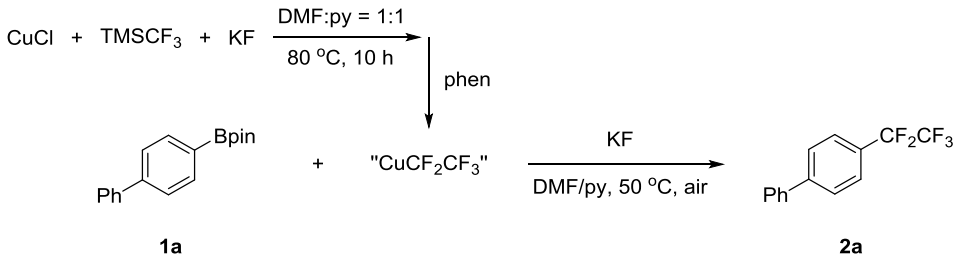
1a **2a**

Typical Procedures:

Preparation of "$CuCF_2CF_3$": To an oven-dried sealed tube were added CuCl (2.250 mmol, 222.9 mg) and KF (1.500 mmol, 87.2 mg) in glove box. Then in fume hood, DMF (3.0 mL), $TMSCF_3$ (1.500 mmol, 220 μL) and pyridine (3.0 mL) were successively added under N_2 atmosphere. After stirred at room temperature for 5 minutes, the reaction mixture was immersed into 80 ^oC oil bath and stirred for 10 h. Then the reaction mixture was cooled to room temperature and filtrated in the glove box. The concentration of "$CuCF_2CF_3$" in the filtrate was obtained by quantitative ^{19}F NMR analysis of a 1.0 mL aliquot of this solution using $PhCF_3$ as the internal standard. The NMR result indicated that "$CuCF_2CF_3$" was in 0.08 M concentration.

Pentafluoroethylation of boronates: Adding the ligand into the rest of "$CuCF_2CF_3$" (2.0 mL) solution without $PhCF_3$. Then the reaction mixture was stirred at room temperature for 1 h. Under N_2 atmosphere, to a Schlenk flask was added **1a** (0.067 mmol, 18.8 mg), activaor and the "$CuCF_2CF_3$" solution prepared above. After that, the reaction mixture was stirred at the temperature required for 3 h in the open air. When the reaction was completed, the internal standard was added. After that, the reaction mixture was quenched with 3.0 M HCl solution or water, extracted with ether. Then, the organic layer was collected and analyzed by ^{19}F NMR.

Table S1. The influence of equivalence of KF and phen[a]

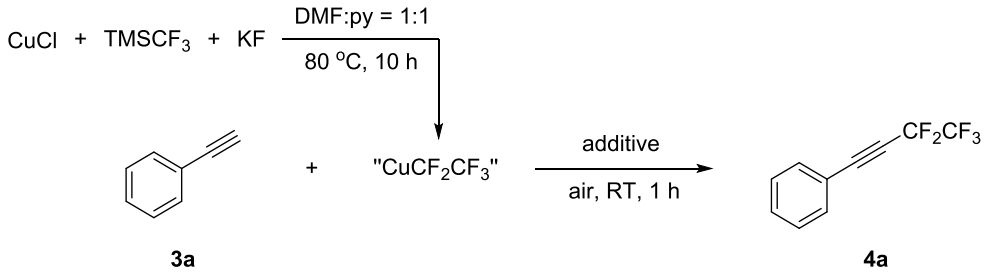


1a **2a**

Entry	Equiv (phen)	Yield (%)[b]
1[c]	11.2	65
2	11.2	72
3	10.0	70
4	9.0	69
5	8.0	56
6	7.0	55
7	6.0	57
8	5.0	55
9	4.0	49
10	3.0	46

[a] Reaction conditions: $CuCF_2CF_3$ was prepared from $TMSCF_3$ (1.500 mmol), KF (1.500 mmol), CuCl (2.250 mmol), DMF (3.0 mL) and pyridine (3.0 mL) at 80 °C for 10 h, and used after filtration. Prepared $CuCF_2CF_3$ (2.4 equiv), ligand (0.750 mmol, 11.2 equiv), rt, 1 h. Then **1a** (0.067 mmol, 1.0 equiv), 50 °C, 3 h, air. [b] The yields were determined by ^{19}F NMR spectroscopy using $PhCF_3$ as the internal standard. [c] KF (0.200 mmol, 3.0 equiv) was used.

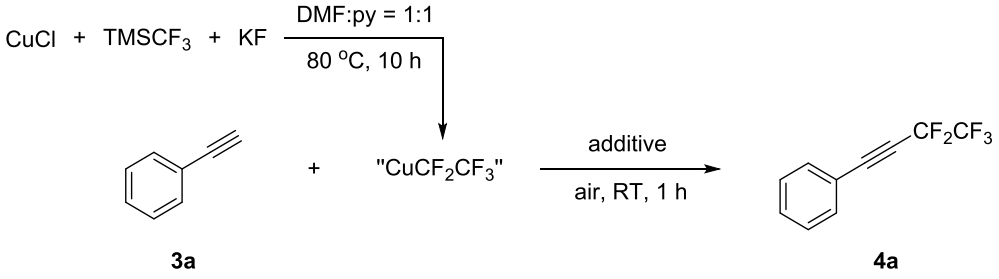
2.2 General Procedures for Optimization of Pentafluoroethylaion of Acetylenes



Typical Procedures:

To an oven-dried sealed tube were added CuCl (0.750 mmol, 74.3 mg) and KF (0.500 mmol, 29.1 mg) in glove box. Then in fume hood, DMF (1.0 mL), $TMSCF_3$ (0.500 mmol, 73 μL) and pyridine (1.0 mL) were successively added under N_2 atmosphere. After stirred at room temperature for 5 minutes, the reaction mixture was immersed into 80 ^oC oil bath and stirred for 10 h. Then the reaction mixture was cooled to room temperature. Then **3a** was dissolved in the DMF (0.5 mL) and added to the sealed tube containing "$CuCF_2CF_3$" solution by the injector pump over 10 min in the open air. After that, the reaction mixture was stirred at room temperature for 1 h. When the reaction was completed, the internal standard was added. After that, the reaction mixture was quenched with 3.0 M HCl solution or water, extracted with ether. Then, the organic layer was collected and analyzed by ^{19}F NMR.

Table S2. Survey of reaction conditions of pentafluoroethylation of acetylenes[a]



3a **4a**

Entry	**3a** (mmol)	Additive (mmol)	Yield (%)
1	0.13	-	78
2[b]	0.13	-	65
3	0.13	$CuOAc_2$ (0.13)	80
4	0.10	-	74
5	0.15	-	78
6	0.15	TMEDA (0.75)	6
7[c]	0.15	-	87
8[c][d]	0.45	-	86

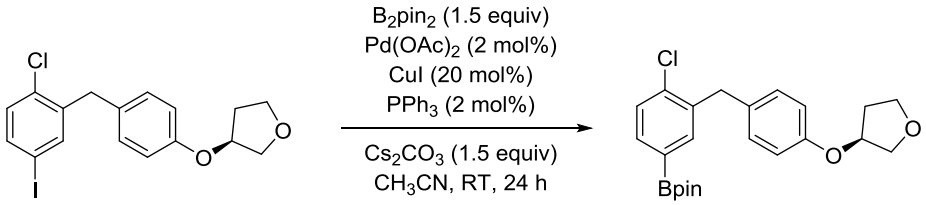
[a] Reaction conditions: CuCl (0.75 mmol), KF (0.50 mmol), $TMSCF_3$ (0.50 mmol), DMF (1.0 mL) and pyridine (1.0 mL). **3a** was dissolved in the DMF (0.5 mL) and added by the injector pump over 10 min. The yields were determined by ^{19}F NMR spectroscopy using $PhOCF_3$ as the internal standard. [b] **3a** was added into the reaction directly. [c] The solution of $CuCF_2CF_3$ stirred in air for 5 min before the **3a** was added. [d] 0.45 mmol-scale reaction was performed: **3a** (0.45 mmol, 1.0 equiv), CuCl (2.25 mmol, 5.0 equiv), $TMSCF_3$ (1.50 mmol, 3.3 equiv), KF (1.50 mmol, 3.3 equiv), air, RT, 1 h. The yield of **4a** in the scale of 0.45 mmol was determined by ^{19}F NMR spectroscopy using $PhOCF_3$ as the internal standard.

3. Syntheses of Boronates and Alkynes

3.1 Syntheses of Arylboronates

a.

(*R*)-2-(4-chloro-3-(4-((tetrahydrofuran-3-yl)oxy)benzyl)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1h)



1h

(*R*)-2-(4-chloro-3-(4-((tetrahydrofuran-3-yl)oxy)benzyl)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane was prepared according to reference[1].

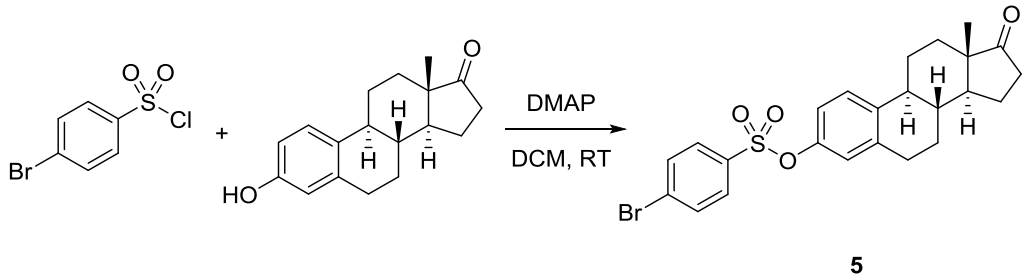
Under N_2 atmosphere, into an oven-dried sealed tube were added (3*S*)-3-[4-[(2-Chloro-5-iodophenyl)methyl]phenoxy]tetrahydrofuran (6.00 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.12 mmol, 0.02 equiv), B_2pin_2 (9.00 mmol, 1.5 equiv), CuI (1.20, 0.20 equiv), PPh_3 (0.12 mmol, 0.02 equiv), Cs_2CO_3 (9.00 mmol, 1.5 equiv) and dry CH_3CN (20 mL), then the reaction mixture was stirred at room temperature for 24 h. After the reaction was completed, the reaction mixture was added water (20 mL), and extracted with EA (3 × 20 mL). The organic layers were combined, washed with brine, dried over Na_2SO_4 and concentrated under vacuum. The crude product was purified by fast column chromatography to afford the desired product **1h** as a white solid in 68% yield (1.70 g).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.69 (s, 1H), 7.60 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.36 (d, *J* = 7.9 Hz, 1H), 7.10 (d, *J* = 8.6 Hz, 2H), 6.76 (d, *J* = 8.6 Hz, 2H), 4.89 – 4.86 (m, 1H), 4.05 (s, 2H), 4.02 – 3.93 (m, 3H), 3.88 (td, *J* = 8.0, 4.8 Hz, 1H), 2.22 – 2.11 (m, 2H), 1.33 (s, 12H).

All the characterization data are consistent with the previous report[2].

b.

(8*R*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[a]phenanthren-3-yl 4-bromobenzenesulfonate (5)



5

(8*R*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[a]phenanthren-3-yl 4-bromobenzenesulfonate was prepared according to the

reference[3].

To a Schlenk tube was added 4-bromobenzenesulfonyl chloride (6.6 mmol, 1.686 g, 1.1 equiv), then the tube was evacuated/backfilled with N_2 for three times and estrone (6.0 mmol, 1.784 g, 1.0 equiv), DMAP (2.199 g, 3.0 equiv) and dry DCM (20 mL) were added. After that, the reaction was stirred at room temperature for 12 h. After the reaction was completed, the reaction was quenched with 3 M HCl and extracted with DCM (3 × 20 mL). The organic layer was combined, dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was purified by column chromatography to obtain the desired product as a white solid **5** in 89% yield (2.60 g).

White solid; Mp: 160-162 ^oC; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 – 7.56 (m, 4H), 7.19 (d, J = 8.6 Hz, 1H), 6.79 (d, J = 2.4 Hz, 1H), 6.68 (dd, J = 8.6, 2.5 Hz, 1H), 2.85 (dd, J = 8.8, 4.1 Hz, 2H), 2.51 (dd, J = 18.9, 8.6 Hz, 1H), 2.41 – 1.91 (m, 6H), 1.76 – 1.35 (m, 6H), 0.91 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.42, 139.19, 138.74, 134.84, 132.56, 130.03, 129.54, 126.74, 122.40, 119.18, 77.48, 77.16, 76.84, 50.52, 47.98, 44.20, 37.93, 35.92, 31.61, 29.42, 26.28, 25.78, 21.67, 13.93.

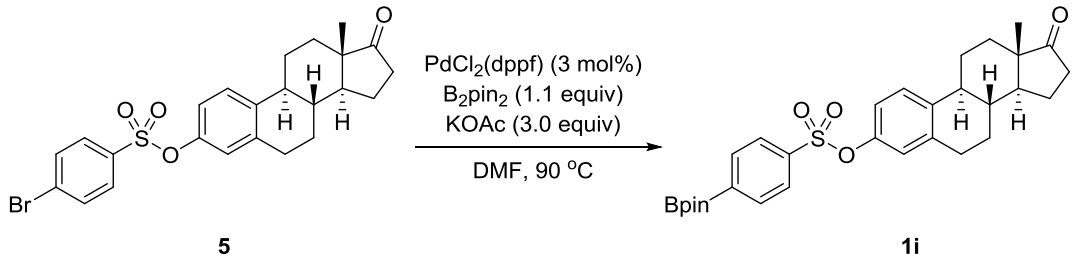
MS (**ESI**, *m/z*): 511 ($M+Na^+$);

HRMS (**ESI**): Calcd for: $[C_{14}H_{25}BrSO_4Na]^+$, 511.0549, found: 511.0553.

IR (film): 3090, 3060, 2931, 2860, 1736, 1575, 1490, 1471, 1437, 1453, 1391, 1376, 1262, 1207, 930, 918 cm^{-1}.

c.

(8*R*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[a]phenanthren-3-yl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzenesulfonate (1i)

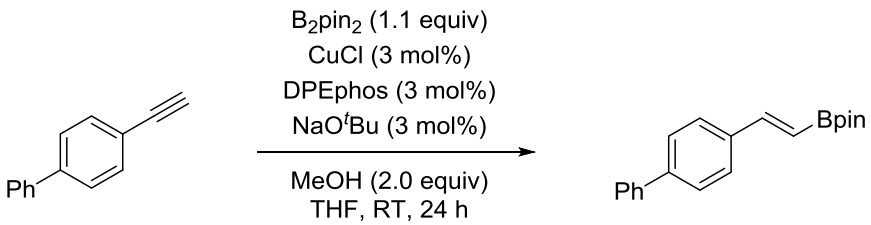


(8*R*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[a]phenanthren-3-yl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzenesulfonate was prepared according to reference[4].

Under N_2 atmosphere, into an oven-dried sealed tube were added **5** (978.8 mg, 1.00 equiv), $PdCl_2$(dppf) (43.9 mg, 0.03 equiv), B_2pin_2 (558.7mg, 1.10 equiv), KOAc (588.8 mg, 3.00 equiv) and dry DMF (10 mL), then the reaction mixture was stirred at 90 ^oC for 12 h. After the reaction was completed, the reaction mixture was added water (20 mL), and extracted with DCM (3 × 20 mL). The organic layers were combined, washed with water (3 × 30 mL), dried over Na_2SO_4 and concentrated under vacuum. The crude product was purified by fast column chromatography to afford the desired product **1i** as a white solid in 40% yield (430 mg).

White solid; Mp: 90-92 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.94 (d, J = 8.2 Hz, 2H), 7.84 (d, J = 8.2 Hz, 2H), 7.15 (d, J = 8.6 Hz, 1H), 6.80 (d, J = 2.3 Hz, 1H), 6.64 (dd, J = 8.6, 2.5 Hz, 1H), 2.84 (dd, J = 8.7, 3.8 Hz, 2H), 2.51 (dd, J = 18.8, 8.6 Hz, 1H), 2.42 – 1.88 (m, 6H), 1.75 – 1.40 (m, 6H), 1.37 (s, 12H), 0.91 (s, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 220.74, 147.61, 138.96, 138.60, 138.01, 135.33, 127.52, 126.63, 122.50, 119.26, 84.74, 50.54, 48.02, 44.21, 37.95, 35.95, 31.63, 29.42, 26.32, 25.79, 25.03, 24.99, 21.70, 13.95.
MS (**ESI**, *m/z*): 558 ($M+Na^+$);
HRMS (**ESI**): Calcd for: $[C_{30}H_{37}{}^{10}BSO_6Na]^+$, 558.2332, found: 558.2337.
IR (film): 2976, 2931, 2865, 1737, 1601, 1492, 1392, 1373, 1359, 1271, 1207, 1189, 1142, 930, 918, 855 cm^{-1}.

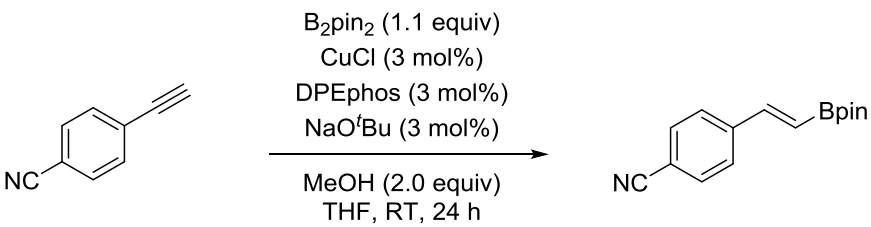
d. (*E*)-2-(2-([1,1'-biphenyl]-4-yl)vinyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1y)



1y

(*E*)-2-(2-([1,1'-biphenyl]-4-yl)vinyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane was prepared according to reference[5].
Under N_2 atmosphere, into an oven-dried Schlenk flask were added CuCl (24 mg, 0.03 equiv), NaO^{t}Bu (48 mg, 0.03 equiv), DPEphos (128 mg, 0.03 equiv) and dry THF (8 mL). The reaction mixture was stirred at room temperature for 30 min. After that, B_2pin_2 (2.23 g, 1.10 equiv) and THF (4 mL) were added. Then the mixture was stirred for another 10 min. The 4-biphenylacetylene (1.43 g, 1.00 equiv) was added, followed by MeOH (0.6 mL). The reaction mixture was stirred at romm temperature for 24 h. When the reaction was completed, the mixture was filtered through a pad of Celite and concerntrated. The crude product was purified by fast column chromatography to afford the desired product **1y** as a yellow solid in 55% yield (1.34 g).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 – 7.53 (m, 6H), 7.44 (t, J = 8.5 Hz, 3H), 7.34 (t, J = 7.3 Hz, 1H), 6.21 (d, J = 18.3 Hz, 1H), 1.33 (s, 12H).
All the characterization data are consistent with the previous report[6].

e. (*E*)-4-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)vinyl)benzonitrile (1aa)



1aa

(*E*)-4-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)vinyl)benzonitrile was prepared according to reference[5].

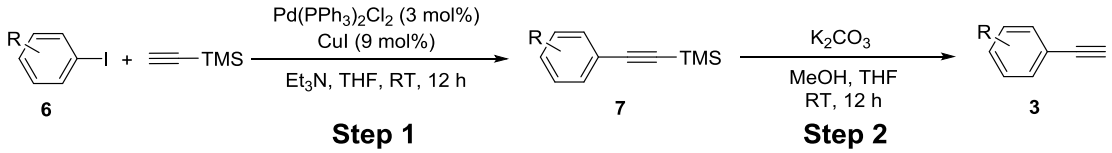
Under N_2 atmosphere, into an oven-dried Schlenk flask were added CuCl (30 mg, 0.03 equiv), NaO^{t}Bu (60 mg, 0.03 equiv), DPEphos (160 mg, 0.03 equiv) and dry THF (10 mL). The reaction mixture was stirred at room temperature for 30 min. After that, B_2pin_2 (2.79 g, 1.10 equiv) and THF (5 mL) were added. Then the mixture was stirred for another 10 min. The 4-cyanophenylacetylene (1.27 g, 1.00 equiv) was added, followed by MeOH (0.8 mL). The reaction mixture was stirred at romm temperature for 24 h. When the reaction was completed, the mixture was filtered through a pad of Celite and concerntrated. The crude product was purified by fast column chromatography to afford the desired product **1aa** as a white solid in 56% yield (1.42 g).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, J = 8.3 Hz, 2H), 7.55 (d, J = 8.1 Hz, 2H), 7.37 (d, J = 18.4 Hz, 1H), 6.28 (d, J = 18.5 Hz, 1H), 1.32 (s, 12H).

All the characterization data are consistent with the previous report[5].

3.2 Syntheses of Alkynes

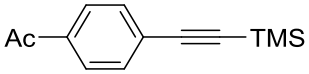
General Procedure A:



Step 1:

To a Schlenk tube were added CuI (172 mg, 0.9 mmol, 0.09 equiv), $Pd(PPh_3)_2Cl_2$ (210 mg, 0.3 mmol, 0.03 equiv) and **6** (10 mmol, 1.0 equiv), then THF (20 mL) was added under nitrogen atmosphere. After that, triethylamine (10 mL) and ethynyltrimethylsilane (1.05 g, 1.5 mL, 10.8 mmol, 1.1 equiv) were added successively. The reaction mixture was stirred at room temperature for 12 h. The resulting mixture was filtered under reduced pressure. After the solvent was removed under vacuum, the crude product was purified by column chromatography on silica gel to afford the product **7**.

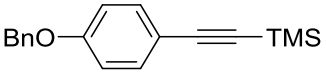
1-(4-((Trimethylsilyl)ethynyl)phenyl)ethan-1-one (7b)[7]



Performed on 10 mmol scale, eluted with petroleum ether/ethyl acetate = 50/1 to give **7b** (1.77g, 82%) as a pale-yellow liquid.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.87 (d, J = 8.0 Hz, 2H), 7.52 (d, J = 8.0 Hz, 2H), 2.58 (s, 3H), 0.25 (s, 9H).

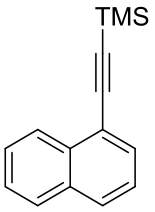
((4-(Benzyloxy)phenyl)ethynyl)trimethylsilane (7h)[7]



Performed on 10 mmol scale, eluted with petroleum ether to give **7h** (2.55 g, 91%) as a pale-yellow solid.

^{1}H NMR ($CDCl_3$, 400MHz): δ 7.41–7.29 (m, 7H), 6.87 (d, J = 8.0 Hz, 2H), 5.05 (s, 2H), 0.22 (s, 9H).

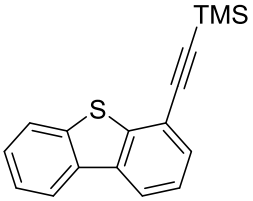
Trimethyl(naphthalen-1-ylethynyl)silane (7q)[8]



Performed on 7 mmol scale, eluted with petroleum ether to give **7q** (720 mg, 46%) as a yellow solid.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.32 (d, J = 8.0 Hz, 1H), 7.82 (t, J = 8.0 Hz, 2H), 7.69 (d, J = 8.0 Hz, 1H), 7.57 (t, J = 6.8 Hz, 1H), 7.50 (t, J = 6.8 Hz, 1H), 7.39 (t, J = 7.6 Hz, 1H), 0.32 (s, 9H).

(Dibenzo[*b*,*d*]thiophen-4-ylethynyl)trimethylsilane (7r)



Performed on 10 mmol scale, eluted with petroleum ether to give **7r** (2.73 g, 98%) as a yellow solid.

Mp: 84–86 ^{o}C. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.24–8.07 (m, 2H), 7.93–7.84 (m, 1H), 7.58 (d, J = 7.2 Hz, 1H), 7.55–7.38 (m, 3H), 0.35 (s, 9H);

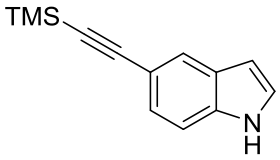
^{13}C NMR ($CDCl_3$, 100 MHz) δ 143.2, 139.7, 135.8, 135.5, 129.9, 127.1, 124.6, 124.5, 123.0, 121.9, 121.7, 118.0, 102.5, 100.4, 0.17;

MS (**EI**, *m/z*, %): 280 (M^+, 53.5), 265 (100);

HRMS (**EI**): Calcd. For $C_{17}H_{16}SSi$: 280.0736; Found: 280.0753;

IR (film): 3065, 2959, 2897, 1576, 1479, 1440, 1384, 1321, 1249, 1111, 902, 844, 795, 749, 727, 637 cm^{-1}.

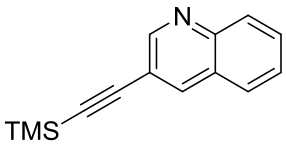
5-((Trimethylsilyl)ethynyl)-1H-indole (7s)[10]



Performed on 10 mmol scale, eluted with petroleum ether/ethyl acetate = 25/1 to give **7s** (1.83 g, 86%) as a yellow oil.

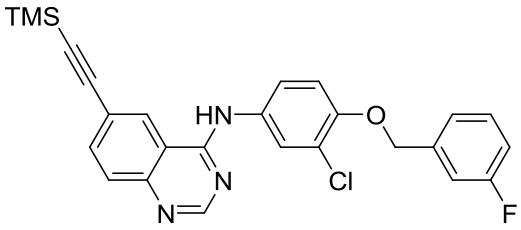
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.18 (s, 1H), 7.80 (s, 1H), 7.29 (s, 2H), 7.19 (t, J = 2.4 Hz, 1H), 6.51–6.50 (m, 1H), 0.25 (s, 9H).

3-((Trimethylsilyl)ethynyl)quinoline (7u)[9]



Performed on 10 mmol scale, eluted with petroleum ether/ethyl acetate = 25/1 to give **7u** (2.20 g, 98%) as a yellow liquid.
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.89 (d, J = 2.0 Hz, 1H), 8.22 (s, 1H), 8.05 (d, J = 9.2 Hz, 1H), 7.79–7.61 (m, 2H), 7.59–7.46 (m, 1H), 0.28 (s, 9H).

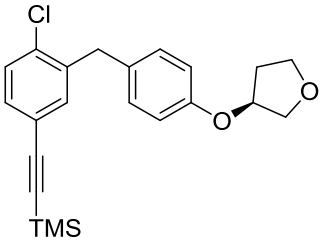
***N*-(3-chloro-4-((3-fluorobenzyl)oxy)phenyl)-6-((trimethylsilyl)ethynyl)quinazolin-4-amine (7aa)**



Performed on 7 mmol scale, eluted from petroleum ether/ethyl acetate = 10/1 to ethyl acetate to give **7aa** (2.85 g, 86%) as a yellow solid.
Yellow solid. Mp: 156–158 ^{o}C. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.73 (s, 1H), 7.99 (s, 1H), 7.87–7.79 (m, 3H), 7.51 (dd, J = 8.8, 2.4 Hz, 1H), 7.40–7.32 (m, 2H), 7.23 (t, J = 7.6 Hz, 2H), 7.06–6.96 (m, 2H), 5.17 (s, 2H), 0.29 (s, 9H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –113.1 (m, 1F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 163.1 (d, J = 244.7 Hz), 157.2, 155.7, 151.4, 149.6, 139.1 (d, J = 7.4 Hz), 136.0, 132.0, 130.3 (d, J = 8.1 Hz), 129.0, 125.0, 124.4, 123.8, 122.6 (d, J = 2.9 Hz), 121.9, 121.7, 115.1 (d, J = 21.0 Hz), 114.8, 114.5, 114.1 (d, J = 22.1 Hz), 103.9, 96.8, 70.6, 0.01;
MS (**ESI**, m/z): 476.1 ($M+H^+$);
HRMS (**ESI**): Calcd. For $C_{28}H_{26}NO_2ClFSi$: 476.1369 ($M+H^+$); Found: 476.1353;
IR (film): 2976, 2929, 2177, 1618, 1593, 1568, 1528, 1499, 1418, 1251, 1067, 909, 843, 551 cm^{-1}.

(*S*)-((4-chloro-3-(4-((tetrahydrofuran-3-yl)oxy)benzyl)phenyl)ethynyl)trimethylsilane (7ab)

Performed on 15 mmol scale, eluted with petroleum ether/ethyl acetate = 25/1 to give **7ab** (5.71 g, 99%) as a yellow solid.



Yellow solid. Mp: 54–56 °C. $[\alpha]_D$ = 9.80 ($CHCl_3$, c= 0.7300 w/v%). **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.34–7.24 (m, 3H), 7.10 (d, J = 8.4 Hz, 2H), 6.80 (d, J = 8.4 Hz, 2H), 4.92–4.85 (m, 1H), 4.05–3.95 (m, 5H), 3.93–3.86 (m, 1H), 2.24–2.09 (m, 2H), 0.26 (s, 9H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 155.8, 139.0, 134.4, 134.1, 131.2, 131.0, 129.9, 129.4, 121.8, 115.3, 103.9, 95.0, 76.7, 73.0, 67.1, 38.0, 32.9, 0.17;

MS (**EI**, *m/z*, %): 384 (M^+, 47.47), 299 (100.00);

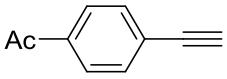
HRMS (**EI**): Calcd. For $C_{22}H_{25}O_2ClSi$: 384.1307; Found: 384.1308;

IR (film): 2957, 2864, 2153, 1611, 1577, 1508, 1472, 1434, 1389, 1242, 1176, 1116, 1083, 956, 845, 760, 678 cm^{-1}.

Step 2:

To a Schlenk tube was added K_2CO_3 (3.5 equiv) and **7** (1.0 equiv), then methanol and THF were added under nitrogen atmosphere. The reaction mixture was stirred at room temperature for 12 h. The resulting mixture was filtered under reduced pressure and then extracted with ethyl acetate (30 mL × 2). The organic layer was washed with water (20 mL × 2) and brine (20 mL × 2), dried with sodium sulfate anhydrous and filtered under reduced pressure. The solvent was removed under reduced pressure to afford the product **3**.

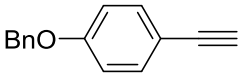
1-(4-Ethynylphenyl)ethan-1-one (3b)[7]



Performed on 5.9 mmol scale. 12 mL of THF and 12 mL of methanol was used as solvents. **3b** (751 mg，88%) was obtained as a pale-yellow solid.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.89 (d, J = 8.4 Hz, 2H), 7.55 (d, J = 8.4 Hz, 2H), 3.23 (s, 1H), 2.59 (s, 3H).

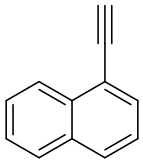
1-(Benzyloxy)-4-ethynylbenzene (3h)[7]



Performed on 5.0 mmol scale. 10 mL of THF and 10 mL of methanol was used as solvents. **3h** (1.0 g, 97%) was obtained as a pale-yellow solid.

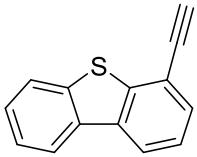
^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.44–7.30 (m, 7H), 6.90 (d, J = 8.8 Hz, 2H), 5.06 (s, 2H), 2.97 (s, 1H).

1-Ethynylnaphthalene (3q)[11]



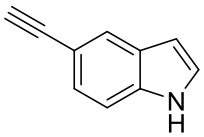
Performed on 2.5 mmol scale. 8 mL of THF and 8 mL of methanol was used as solvents. **3q** (300 mg, 79%) was obtained as a yellow liquid.
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.36 (d, J = 8.0 Hz, 1H), 7.86 (d, J = 8.0 Hz, 2H), 7.73 (d, J = 7.2 Hz, 1H), 7.58 (t, J = 8.0 Hz, 1H), 7.52 (t, J = 8.0 Hz, 1H), 7.42 (t, J = 8.0 Hz, 1H), 3.46 (s, 1H).

4-Ethynyldibenzo[*b*,*d*]thiophene (3r)



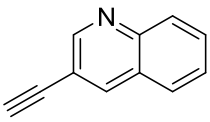
Performed on 10 mmol scale. 15 mL of THF and 15 mL of methanol was used as solvents. **3r** (1.61g, 78%) was obtained as a white solid.
Mp: 65–67 ^{o}C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.19 (d, J = 4.0 Hz, 2H), 7.95–7.93 (m, 1H), 7.67 (d, J = 7.4 Hz, 1H), 7.56–7.46 (m, 3H), 3.57 (s, 1H);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 143.0, 139.5, 135.6 (2 carbon), 130.4, 127.1, 124.6, 124.4, 123.0, 122.0, 121.9, 116.8, 82.5, 81.3;
MS (**EI**, *m/z*, %): 208 (M^+, 100), 163 (20.86);
HRMS (**EI**): Calcd. For $C_{14}H_8S$: 208.0341; Found: 208.0345;
IR (film): 3290, 3064, 1576, 1479, 1440, 1386, 1306, 1321, 1251, 1161, 1110, 1047, 1018, 796, 749, 713, 661, 648, 594 cm^{-1}.

5-Ethynyl-1*H*-indole (3s)[12]



Performed on 8.6 mmol scale. 15 mL of THF and 15 mL of methanol was used as solvents. **3s** (1.07g, 88%) was obtained as a black solid.
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.27 (br, 1H), 7.89 (s, 1H), 7.38 (s, 2H), 7.28–7.26 (m, 1H), 6.60–6.59 (m, 1H), 3.06 (s, 1H).

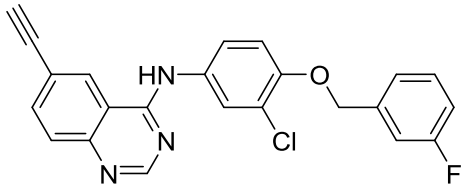
3-Ethynylquinoline (3u)[9]



Performed on 10 mmol scale. 15 mL of THF and 15 mL of methanol was used as solvents. **3u** (1.33g, 87%) was obtained as a yellow solid.
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.93 (d, J = 2.0 Hz, 1H), 8.27 (d, J = 2.0 Hz, 1H),

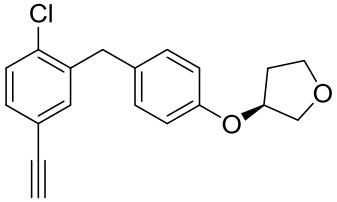
8.07 (d, *J* = 8.4 Hz, 1H), 7.77–7.70 (m, 2H), 7.57–7.53 (m, 1H), 3.26 (s, 1H).

***N*-(3-chloro-4-((3-fluorobenzyl)oxy)phenyl)-6-ethynylquinazolin-4-amine (3aa)**



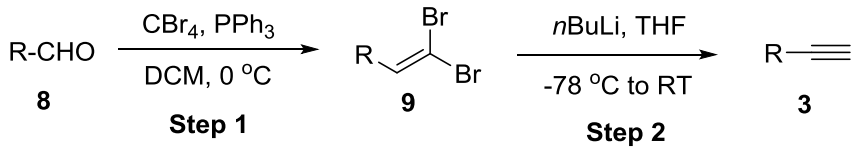
Performed on 4.84 mmol scale. 10 mL of THF and 10 mL of methanol was used as solvents. **3aa** (1.7 g, 87%) was obtained as a yellow solid.
Mp: 218–220 ^{o}C. **^{1}H NMR** (DMSO-d_6, 400 MHz) δ 9.84 (s, 1H), 8.72 (s, 1H), 8.59 (s, 1H), 8.04 (d, *J* = 2.0 Hz, 1H), 7.83 (d, *J* = 8.8 Hz, 1H), 7.73 (t, *J* = 9.2 Hz, 2H), 7.50–7.40 (m, 1H), 7.34–7.26 (m, 2H), 7.23 (d, *J* = 8.8 Hz, 1H), 7.15 (t, *J* = 8.0 Hz, 1H), 5.22 (s, 2H), 4.38 (s, 1H);
^{19}F NMR (DMSO-d_6, 376 MHz) δ –113.1 (m, 1F);
^{13}C NMR (DMSO-d_6, 100 MHz) δ 162.2 (d, *J* = 242.6 Hz), 157.0, 155.3, 149.8, 149.4, 139.6 (d, *J* = 7.3 Hz), 135.3, 133.0, 130.5 (d, *J* = 8.2 Hz), 128.3, 126.9, 123.9, 123.3 (d, *J* = 2.9 Hz), 122.1, 121.1, 119.4, 114.9, 114.7 (d, *J* = 20.7 Hz), 114.2, 114.0 (d, *J* = 21.8 Hz), 83.1, 81.9, 69.4;
MS (**ESI**, m/z): 404.0 ($M+H^+$);
HRMS (**ESI**): Calcd. For $C_{23}H_{16}N_3OClF$: 404.0960 ($M+H^+$); Found: 404.0963;
IR (film): 3284, 2995, 1770, 1759, 1592, 1550, 1527, 1499, 1419, 1374, 1357, 1247, 1145, 1061, 942, 890, 843, 797, 776, 661, 542 cm^{-1}.

(*S*)-3-(4-(2-chloro-5-ethynylbenzyl)phenoxy)tetrahydrofuran (3ab)



Performed on 15 mmol scale. 30 mL of THF and 30 mL of methanol was used as solvents. **3ab** (4.01g, 85%) was obtained as a brown oil.
$[\alpha]_D$ = 8.13 ($CHCl_3$, c= 0.5050 w/v%). **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.35–7.24 (m, 3H), 7.09 (d, *J* = 8.8 Hz, 2H), 6.80 (d, *J* = 8.4 Hz, 2H), 4.93–4.86 (m, 1H), 4.04–3.94 (m, 5H), 3.93–3.85 (m, 1H), 3.07 (s, 1H), 2.24–2.10 (m, 2H);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 156.0, 139.3, 134.8, 134.3, 131.1, 131.0, 130.0, 129.6, 120.8, 115.4, 82.7, 78.2, 77.2, 73.1, 67.1, 38.1, 33.0;
MS (**EI**, *m/z*, %): 312 (M^+, 65.73), 207 (100.00);
HRMS (**EI**): Calcd. For $C_{19}H_{17}O_2Cl$: 312.0912; Found: 312.0920;
IR (film): 3290, 2952, 2866, 1610, 1577, 1508, 1471, 1438, 1241, 1177, 1116, 1081, 1043, 905, 822 cm^{-1}.

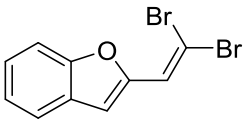
General procedure B:



Step 1:

To a solution of CBr_4 (6.64 g, 20 mmol, 2.0 equiv) in dichloromethane (40 mL) was added PPh_3 (10.5 g, 40 mmol, 4.0 equiv) in dichloromethane (30 mL) under nitrogen atmosphere at 0 °C. A solution of **8** (10 mmol, 1.0 equiv) in dichloromethane (10 mL) was added under nitrogen atmosphere. The reaction mixture was stirred for 2 h at 0 °C. The resulting mixture was extracted with diethyl ether (30 mL × 2). The organic layer was washed with water (20 mL × 2) and brine (20 mL × 2), dried with sodium sulfate anhydrous and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel to afford the product **9**.

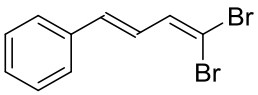
2-(2,2-Dibromovinyl)benzofuran (9a)[13]



Performed on 10 mmol scale, eluted with petroleum ether to give **9a** (2.72 g, 91%) as a yellow solid.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.59 (d, J = 7.8 Hz, 1H), 7.52 (s, 1H), 7.47–7.42 (m, 1H), 7.32 (m, 1H), 7.29 (s, 1H), 7.24–7.19 (m, 1H).

(*E*)-(4,4-Dibromobuta-1,3-dien-1-yl)benzene (9b)[13]



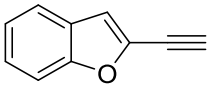
Performed on 10 mmol scale, eluted with petroleum ether to give **9b** (1.5 g, 52%) as a white solid.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.45 (dd, J = 8.4, 1.6 Hz, 2H), 7.38–7.27 (m, 3H), 7.10 (d, J = 9.6 Hz, 1H), 6.85–6.67 (m, 2H).

Step 2:

To a Schlenk tube was added **9** (1.0 equiv), then THF (30 mL) was added under nitrogen atmosphere. *n*-BuLi (2.5 M in hexane, 2.5 equiv) was added dropwise at -78 °C in 15 min. The reaction solution was stirred at -78 °C for 30 min. After the resulting mixture was slowly warmed to room temperature, it was quenched with aqueous sat. NH_4Cl and extracted with diethyl ether (50 mL × 2). The organic layer was washed with water (20 mL × 2) and brine (20 mL × 2), dried with sodium sulfate anhydrous and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel to afford the product **3**.

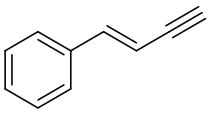
2-Ethynylbenzofuran (3t)[13]



Performed on 8.94 mmol scale, eluted with petroleum ether to give **3t** (680 mg, 54%) as a brown liquid.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.55 (d, J = 7.8 Hz, 1H), 7.45 (d, J = 8.4 Hz, 1H), 7.34 (t, J = 7.8 Hz, 1H), 7.26–7.22 (m, 1H), 7.00 (s, 1H), 3.49 (d, J = 1.6 Hz, 1H).

(*E*)-but-1-en-3-yn-1-ylbenzene (3v)[13]

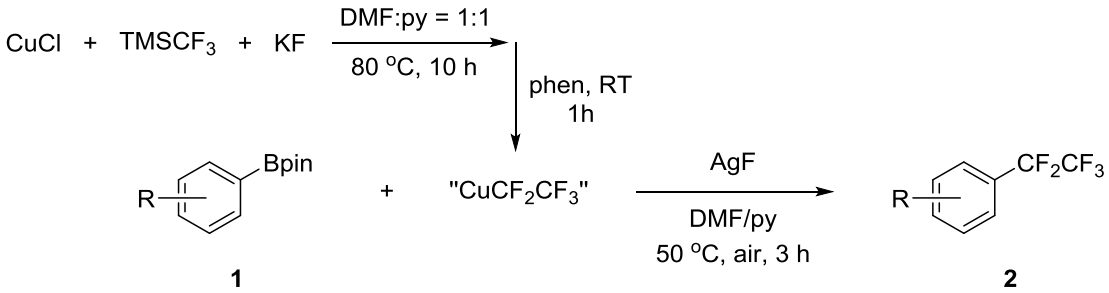


Performed on 5.2 mmol scale, eluted with petroleum ether to give **3v** (226 mg, 34%) as a brown liquid.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.39–7.26 (m, 5H), 7.03 (d, J = 16.4 Hz, 1H), 6.12 (dd, J = 16.4, 2.4 Hz, 1H), 3.04 (d, J = 2.4 Hz, 1H).

4. General Procedures for Pentafluoroethylation of Substrates

4.1 General Procedures for Pentafluoroethylation of Boronates

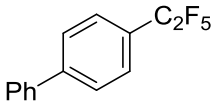


Typical Procedures:

To an oven-dried sealed tube were added CuCl (4.50 mmol, 445.8 mg, 11.2 equiv) and KF (3.0 mmol, 174.4 mg, 7.5 equiv) in glove box. Then in fume hood, DMF (6 mL), $TMSCF_3$ (3.0 mmol, 440 μL, 7.5 equiv) and pyridine (6 mL) were successively added under N_2 atmosphere. After stirred at room temperature for 5 minutes, the reaction mixture was immersed into 80 °C oil bath and stirred for 10 h. Then the reaction mixture was cooled to room temperature and filtrated in the glove box. The filtrate was added in to the sealed tube containing phen (1.60 mmol, 288.3 mg, 4.0 equiv). Then the reaction mixture was stirred at room temperature for 1 h. Under N_2 atmosphere, to a Schlenk flask was added arylboronate (0.40 mmol, 1.0 equiv), AgF (1.60 mmol, 203.0 mg, 4.0 equiv) and the "$CuCF_2CF_3$" solution prepared above. After that, the reaction mixture was stirred 50 °C for 3 h in the open air. When the reaction

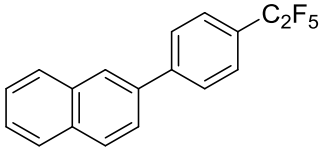
was completed, the reaction mixture was quenched with 50 mL 3.0 M HCl solution or 20 mL ammonium hydroxide, extracted with CH_2Cl_2 (30 mL×3). The combined organic layer was washed with H_2O (30 mL×3) and brine (40 mL), dried over Na_2SO_4, concentrated under vacuum. The residue was purified by column chromatography on silica gel to afford **2**. ^{19}F NMR data (using $PhCF_3$ as the internal standard) were provided for the volatile products.

4-(perfluoroethyl)-1,1'-biphenyl (2a)：



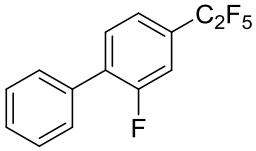
White solid (71 mg, 65%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.68 (q, J = 8.6 Hz, 4H), 7.62 – 7.56 (m, 2H), 7.51 – 7.44 (m, 2H), 7.44 – 7.37 (m, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -85.19 (s, 3F), -115.15 (s, 2F);
^{13}C NMR ($CDCl_3$, 126 MHz) δ 145.00 (d, J = 1.7 Hz), 139.82, 129.14, 128.39, 127.56, 127.44, 127.06 (t, J = 6.2 Hz), 119.31 (qt, J = 286.0, 39.6 Hz), 113.68 (tq, J = 253.7, 38.2 Hz).
All the characterization data are consistent with the previous report[3].

2-(4-(perfluoroethyl)phenyl)naphthalene (2b):



White solid (54 mg, 56%). Mp: 114-116 ^{o}C; **^{1}H NMR** ($CDCl_3$, 400 MHz,) δ 8.05 (d, J = 1.2 Hz, 1H), 7.96-7.85 (m, 3H), 7.82 (d, J = 8.6 Hz, 2H), 7.73 (d, J = 1.8 Hz, 1H), 7.70 (d, J = 8.5 Hz, 2H), 7.56-7.46 (m, 2H);
^{19}F NMR ($CDCl_3$, 376 MHz,) δ -85.18 (s, 3F), -115.17 (s, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 144.94, 137.10, 133.70, 133.15, 128.93, 128.47, 127.85, 127.81, 127.64 (t, J = 23.9 Hz), 127.13 (t, J = 6.2 Hz), 126.75, 126.66, 126.55, 125.31, 119.34 (qt, J = 286.2, 39.5 Hz), 113.72 (tq, J = 252.0, 38.1 Hz);
MS (**EI**, *m/z*): 322 (M^+);
HRMS (**EI**): Calcd for: $C_{18}H_{11}F_5^+$, 322.0781, found: 322.0788.
IR (film): 3069, 1438, 1410, 1341, 1275, 1202, 1144, 1112, 1094, 975, 833, 815, 754, 707 cm^{-1}.

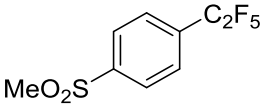
2-fluoro-4-(perfluoroethyl)-1,1'-biphenyl (2c):



Colorless liquid (98 mg, 84%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.54 (ddd, J = 4.6, 3.9, 2.4 Hz, 3H), 7.48-7.36 (m, 5H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -85.11 (s, 3F), -115.09 (s, 2F), -116.04 (t, J = 8.9 Hz,

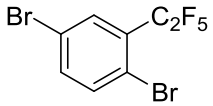
1F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.59 (d, J = 250.3 Hz), 134.46, 133.13 (d, J = 13.6 Hz), 131.50 (d, J = 3.8 Hz), 129.56 (td, J = 24.8, 8.0 Hz), 129.17 (d, J = 2.9 Hz), 128.83, 128.74, 122.60 (dd, J = 10.4, 5.8 Hz), 119.16 (qt, J = 284.2, 39.1 Hz), 114.99 (dt, J = 26.7, 6.5 Hz), 112.93 (tqd, J = 252.8, 38.4, 1.7 Hz).
MS (**FI**, *m/z*): 290 (M^+);
HRMS (**FI**): Calcd for: $C_{14}H_8F_6^+$, 290.0525, found: 290.0531.
IR (film): 3077, 3043, 2917, 2852, 1585, 1519, 1485, 1418, 1337, 1303, 1209, 1194, 1127, 1093, 1009, 869, 767, 747 cm^{-1}.

1-(methylsulfonyl)-4-(perfluoroethyl)benzene (2d):



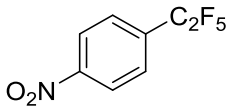
Light yellow solid (84 mg, 77%). Mp: 100-102 ^oC; **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.13 (d, J = 8.3 Hz, 2H), 7.85 (d, J = 8.4 Hz, 2H), 3.12 (s, 3H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -84.95 (s, 3F), -115.85 (s, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 144.30, 134.05 (t, J = 24.2 Hz), 128.08, 127.94 (t, J = 6.0 Hz), 118.84 (qt, J = 286.1, 38.4 Hz), 112.80 (tq, J = 255.1, 38.7 Hz), 44.38;
MS (**EI**, *m/z*): 274 (M^+);
HRMS (**EI**): Calcd for: $C_9H_7F_5O_2S^+$, 274.0087, found: 274.0089.
All the characterization data are consistent with the previous report[14].

1,4-dibromo-2-(perfluoroethyl)benzene (2e):



Yellow liquid (97 mg, 69%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.74 (d, J = 2.3 Hz, 1H), 7.59 (d, J = 8.5 Hz, 1H), 7.50 (dd, J = 8.5, 2.3 Hz, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -83.50 (s, 3F), -111.48 (s, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 137.29, 136.28, 133.05 (t, J = 9.1 Hz), 129.95 (t, J = 23.1 Hz), 121.59, 119.58 (t, J = 2.8 Hz), 119.04 (qt, J = 287.4, 38.2 Hz), 112.55 (tq, J = 257.9, 39.7 Hz)
MS (**EI**, *m/z*): 352 (M^+);
HRMS (**EI**): Calcd for: $C_8H_3Br_2F_5^+$, 351.8522, found: 351.8518.
IR (film): 2921, 2852, 1463, 1382, 1351, 1286, 1207, 1159, 1132, 1097, 1036, 979, 820, 734 cm^{-1}.

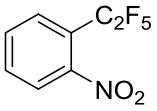
1-nitro-4-(perfluoroethyl)benzene (2f):



Yellow liquid (67 mg, 70%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.39 (d, J = 8.8 Hz, 2H), 7.84 (d, J = 8.8 Hz, 2H);

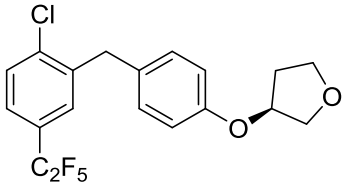
^{19}F NMR ($CDCl_3$, 376 MHz) δ -84.94 (s, 3F), -115.80 (s, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 150.38, 134.74 (t, J = 24.3 Hz), 128.18 (t, J = 6.1 Hz), 124.12, 118.84 (qt, J = 286.2, 38.4 Hz), 112.79 (tq, J = 253.8, 38.9 Hz).
MS (**EI**, *m/z*): 241 (M^+);
HRMS (**EI**): Calcd for: $C_8H_4F_5NO_2^+$, 241.0162, found: 241.0169.
All the characterization data are consistent with the previous report[3].

1-nitro-2-(perfluoroethyl)benzene (2g)



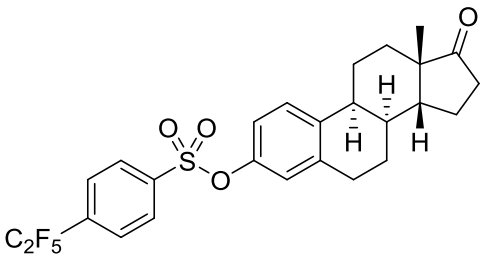
Yellow oil (64 mg, 66%); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 – 7.72 (m, 3H), 7.67 (d, J = 6.8 Hz, 1H).
^{19}F NMR (376 MHz, $CDCl_3$) δ -83.50 (s, 3F), -110.02 (s, 2F).
^{13}C NMR (126 MHz, $CDCl_3$) δ 149.58, 133.59, 131.68, 129.62 (t, J = 7.3 Hz), 124.50, 121.07 (t, J = 24.6 Hz), 118.72 (qt, J = 287.1, 37.9 Hz), 112.61 (tq, J = 257.1, 40.0 Hz).
All the characterization data are consistent with the previous report[3].

(*R*)-3-(4-(2-chloro-5-(perfluoroethyl)benzyl)phenoxy)tetrahydrofuran (2h)



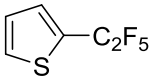
Yellow oil (122 mg, 75%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.50 (d, J = 8.2 Hz, 1H), 7.39 (d, J = 8.9 Hz, 2H), 7.08 (d, J = 8.1 Hz, 2H), 6.81 (d, J = 8.1 Hz, 2H), 4.89 (s, 1H), 4.08 (s, 2H), 4.01-3.95 (m, 3H), 3.92-3.87 (m, 1H), 2.20-2.15 (m, 2H).
^{19}F NMR ($CDCl_3$, 376 MHz) δ -84.81 (s, 3F), -114.90 (s, 2F).
^{13}C NMR ($CDCl_3$, 126 MHz) δ 156.22, 140.17, 138.41, 130.66, 130.17, 130.02, 128.85 (t, J = 6.4 Hz), 127.43 (t, J = 24.3 Hz), 125.68 (t, J = 6.4 Hz), 119.01 (qt, J = 285.9, 39.3 Hz), 115.59, 113.14 (tq, J = 254.5, 38.5 Hz), 77.37, 73.16, 67.25, 38.43, 33.06.
All the characterization data are consistent with the previous report[3].

(8*S*,9*S*,13*S*,14*R*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[a]phenanthren-3-yl-4-(perfluoroethyl)benzenesulfonate (2i):



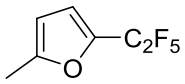
Yellow solid (80 mg, 76%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.02 (d, J = 8.2 Hz, 2H), 7.80 (d, J = 8.2 Hz, 2H), 7.21 (d, J = 8.7 Hz, 1H), 6.77 – 6.65 (m, 2H), 2.82 (s, 2H), 2.51 (dd, J = 19.0, 8.6 Hz, 1H), 2.40 – 1.87 (m, 6H), 1.72 – 1.37 (m, 6H), 0.91 (s, 3H).
^{19}F NMR ($CDCl_3$, 376 MHz) δ -84.96 (s, 3F), -116.01 (s, 2F).
^{13}C NMR ($CDCl_3$, 100 MHz) δ 220.77, 147.25, 139.55, 139.40, 138.84, 134.27 (t, J = 24.3 Hz), 129.04, 127.60 (t, J = 6.2 Hz), 126.85, 122.30, 119.14, 118.79 (qt, J = 286.1, 38.5 Hz), 112.73 (tq, J = 255.1, 38.6 Hz), 50.46, 47.97, 44.17, 37.88, 35.91, 31.56, 29.37, 26.22, 25.74, 21.65, 13.89.
All the characterization data are consistent with the previous report[3].

2-(perfluoroethyl)thiophene (2j):



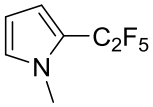
Yield: 56%;
^{19}F NMR (376 MHz) δ -85.67 (s, 3F), -104.60 (s, 2F).
The yield was determined by ^{19}F NMR using $PhCF_3$ as the internal standard.

2-methyl-5-(perfluoroethyl)furan (2k):



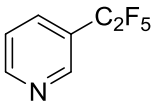
Yield: 58%;
^{19}F NMR (376 MHz) δ -84.98 (s, 3F), -113.65 (s, 2F).
The yield was determined by ^{19}F NMR using $PhCF_3$ as the internal standard.

1-methyl-2-(perfluoroethyl)-1*H*-pyrrole (2l):



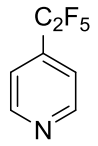
Yield: 76%;
^{19}F NMR (376 MHz) δ -84.04 (s, 3F), -106.22 (s, 2F).
The yield was determined by ^{19}F NMR using $PhCF_3$ as the internal standard.

3-(perfluoroethyl)pyridine (2m):



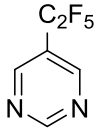
Yield: 95%;
^{19}F NMR (376 MHz) δ -85.86 (s, 3F), -116.02 (s, 2F).
The yield was determined by ^{19}F NMR using $PhCF_3$ as the internal standard.

4-(perfluoroethyl)pyridine (2n):



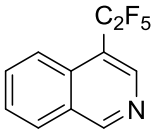
Yield: 85%;
^{19}F NMR (376 MHz) δ -85.49 (s, 3F), -117.56 (s, 2F).
The yield was determined by ^{19}F NMR using $PhCF_3$ as the internal standard.

5-(perfluoroethyl)pyrimidine (2o):



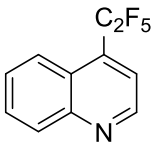
Yield: 89%;
^{19}F NMR (376 MHz) δ -85.91 (s, 3F), -116.81 (s, 2F).
The yield was determined by ^{19}F NMR using $PhCF_3$ as the internal standard.

4-(perfluoroethyl)isoquinoline (2p):



Yellow liquid (76 mg, 77%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 9.40 (s, 1H), 8.82 (s, 1H), 8.19 (d, J = 8.0 Hz, 1H), 8.07 (d, J = 7.9 Hz, 1H), 7.87-7.79 (m, 1H), 7.71 (t, J = 7.2 Hz, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -84.21 (s, 3F), -110.40 (s, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 157.35, 143.72 (t, J = 10.0 Hz), 132.47, 132.32, 128.85, 128.58, 128.19, 124.23-123.67 (m), 119.46 (qt, J = 286.7, 38.5 Hz), 118.72 (t, J = 21.8 Hz), 114.81 (tq, J = 255.8, 39.6 Hz).
MS (**FI**, *m/z*): 247 (M^+);
HRMS (**FI**): Calcd for: $C_{11}H_6F_5N^+$, 247.0415, found: 247.0418.
IR (film): 3060, 3017, 2965, 2917, 2865, 1623, 1586, 1570, 1508, 1372, 1329, 1298, 1264, 1047, 942, 908 cm^{-1}.

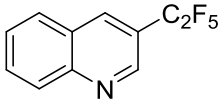
4-(perfluoroethyl)quinoline (2q):



Yellow liquid (60 mg, 61%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 9.06 (d, J = 4.2 Hz, 1H), 8.24 (d, J = 8.6 Hz, 2H), 8.20 (d, J = 8.7 Hz, 1H), 7.86-7.78 (m, 2H), 7.74-7.63 (m, 2H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -83.75 (s, 3F), -111.08 (s, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.41, 149.28, 133.19 (t, J = 22.8 Hz), 130.84,

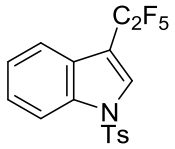
130.24, 128.44, 124.62, 124.05, 120.63 (t, *J* = 8.4 Hz), 119.26 (qt, *J* = 285.3, 37.6 Hz), 114.25 (tq, *J* = 256.09, 39.7 Hz);
MS (**EI**, *m/z*): 247 (M^+);
HRMS (**EI**): Calcd for: $C_{11}H_6F_5N^+$, 247.0420, found: 247.0425.
All the characterization data are consistent with the previous report[15].

3-(perfluoroethyl)quinoline (2r):



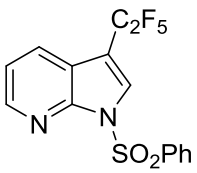
Yellow liquid (85 mg, 86%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 9.13 – 9.03 (m, 1H), 8.44 (s, 1H), 8.21 (d, *J* = 8.5 Hz, 1H), 7.93 (d, *J* = 8.2 Hz, 1H), 7.87 (t, *J* = 7.7 Hz, 1H), 7.67 (t, *J* = 7.5 Hz, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -85.14 (s, 3F), -115.20 (s, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.55, 146.63 (t, *J* = 5.35 Hz), 135.79 (t, *J* = 6.61 Hz), 132.12, 129.76, 128.74, 128.18, 126.47, 121.98 (t, *J* = 24.19 Hz), 119.12 (qt, *J* = 286.0, 38.90 Hz), 113.17 (tq, *J* = 254.85, 39.06 Hz);
MS (**EI**, *m/z*): 247 (M^+);
HRMS (**EI**): Calcd for: $C_{11}H_6F_5N^+$, 247.0420, found: 247.0432.
All the characterization data are consistent with the previous report[16].

3-(perfluoroethyl)-1-tosyl-1*H*-indole (2s):



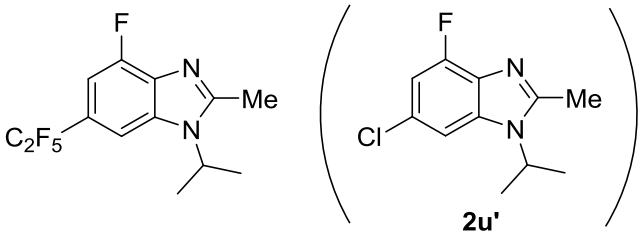
Light yellow solid (100 mg, 86%). Mp: 102-104 ^{o}C; **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.93-7.83 (m, 2H), 7.71 (d, *J* = 8.2 Hz, 2H), 7.54 (d, *J* = 7.9 Hz, 1H), 7.28 (t, *J* = 7.8 Hz, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.13 (d, *J* = 8.1 Hz, 2H), 2.21 (s, 3H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -85.19 (s, 3F), -111.24 (s, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 146.12, 134.87, 134.61, 130.38, 127.62 (t, *J* = 8.5 Hz), 127.26, 126.41, 125.94, 124.42, 120.90, 119.27 (qt, *J* = 285.8, 39.0 Hz), 113.76, 112.96 (tq, *J* = 249.85, 40.10 Hz) 110.91 (t, *J* = 28.0 Hz), 21.69.
MS (**FI**, *m/z*): 389 (M^+);
HRMS (**FI**): Calcd for: $C_{17}H_{12}F_5NO_2S^+$, 389.0503, found: 389.0508.
IR (film): 3112, 2927, 1596, 1566, 1493, 1382, 1333, 1253, 1211, 1191, 1177, 1141, 1090, 1022, 990, 751, 666 cm^{-1}.

3-(perfluoroethyl)-1-(phenylsulfonyl)-1*H*-pyrrolo[2,3-*b*]pyridine (2t):



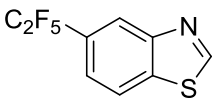
Yellow solid (101 mg, 67%). Mp: 117-119 °C; **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.52 (d, J = 4.6 Hz, 1H), 8.28 (d, J = 7.5 Hz, 2H), 8.11 (s, 1H), 7.97 (d, J = 7.8 Hz, 1H), 7.65 (t, J = 7.5 Hz, 1H), 7.55 (t, J = 7.3 Hz, 2H), 7.32-7.27 (m, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -85.33 (s, 3F), -111.57 (s, 2F)
^{13}C NMR ($CDCl_3$, 100 MHz) δ 146.69, 146.51, 137.55, 134.93, 129.53, 129.43, 128.71, 127.37 (t, J = 8.0 Hz), 120.07, 119.14 (qt, J = 283.9, 38.9 Hz), 119.10, 112.61 (tq, J = 249.5, 40.2 Hz), 107.81 (t, J = 28.7 Hz).
MS (**EI**, *m/z*): 376 (M^+);
HRMS (**EI**): Calcd for: $C_{15}H_9F_5N_2O_2S^+$, 376.0305, found: 376.0301.
IR (film): 3145, 3069, 2923, 2917, 2853, 1678, 1597, 1558, 1480, 1449, 1401, 1386, 1336, 1247, 1194, 1179, 1126, 989, 919 cm^{-1}.

4-fluoro-1-isopropyl-2-methyl-6-(perfluoroethyl)-1*H*-benzo[*d*]imidazole (2u):



Yellow liquid (106 mg, 85%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.45 (s, 1H), 7.06 (dd, J = 10.1, 5.0 Hz, 1H), 4.66 (dtd, J = 13.9, 7.0, 3.6 Hz, 1H), 2.61 (d, J = 4.9 Hz, 3H), 1.59 (dd, J = 7.0, 4.9 Hz, 6H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -85.09 (d, J = 10.9 Hz, 3F), -113.08 (d, J = 6.7 Hz, 2F), -127.25 (d, J = 9.8 Hz, 1F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 154.06, 153.20 (d, J = 253.9 Hz), 136.30 (d, J = 9.4 Hz), 134.21 (d, J = 16.6 Hz), 122.27 (td, J = 24.6, 7.1 Hz), 119.14 (qt, J = 285.9, 40.0 Hz), 113.49 (tqd, J = 252.9, 38.2, 1.7 Hz), 106.06 (dd, J = 10.4, 6.9 Hz), 105.64 (dt, J = 21.2, 6.2 Hz), 48.79, 21.31, 15.01.
MS (**EI**, *m/z*): 310 (M^+);
HRMS (**EI**): Calcd for: $C_{13}H_{12}F_6N_2^+$, 310.0905, found: 310.0909.
IR (film): 2992, 2942, 1636, 1591, 1517, 1492, 1452, 1441, 1373, 1324, 1257, 1209, 1189, 1095, 896, 885, 861 cm^{-1}.
The ratio of the pentafluorinated product and the cholorinated byproduct was 11:1.

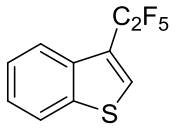
5-(perfluoroethyl)benzo[*d*]thiazole (2v):



Yellow liquid (61 mg, 60%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 9.13 (s, 1H), 8.42 (s, 1H), 8.10 (d, J = 8.5 Hz, 2H), 7.66 (d, J = 8.5 Hz, 2H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -85.07 (s, 3F), -114.26 (s, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 156.16, 153.11, 137.63, 127.29 (t, J = 24.4 Hz), 123.09 (t, J = 5.8 Hz), 122.73, 122.53 (t, J = 6.8 Hz), 119.24 (qt, J = 286.1, 39.4 Hz), 113.57 (tq, J = 252.8, 38.3 Hz).

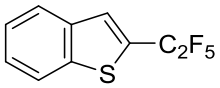
MS (**FI**, *m/z*): 253 (M^+);
HRMS (**FI**): Calcd for: $C_9H_4F_5NS^+$, 252.9979, found: 252.9983.
IR (film): 3078, 2916, 2847, 1608, 1472, 1448, 1415, 1321, 1301, 1204, 1176, 1134, 1088, 1063, 998, 828 cm^{-1}.

3-(perfluoroethyl)benzo[*b*]thiophene (2w)



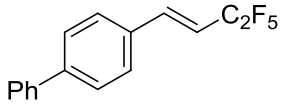
Colorless liquid (84 mg, 83%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.00 (d, J = 7.9 Hz, 1H), 7.92-7.90 (m, 2H), 7.51 – 7.42 (m, 2H).
^{19}F NMR ($CDCl_3$, 376 MHz) δ -84.69 (s, 3F), -110.68 (s, 2F).
^{13}C NMR ($CDCl_3$, 100 MHz) δ 140.52, 135.31, 130.92 (t, J = 8.1 Hz), 125.48, 125.47, 124.26 (t, J = 26.1 Hz), 123.40 (t, J = 1.0 Hz), 122.92, 119.37 (qt, J = 286.4, 39.1 Hz), 113.07 (tq, J = 253.0, 39.7 Hz).
All the characterization data are consistent with the previous report[17].

2-(Perfluoroethyl)benzo[*b*]thiophene (2x):



Yellow liquid (79 mg, 78%); **^{1}H NMR** ($CDCl_3$, 400 MHz): δ 7.97 (d, J = 7.8 Hz, 1H), 7.89 (s, 1H), 7.87 (d, J = 1.4 Hz, 1H), 7.44 (pd, J = 7.1, 1.3 Hz, 2H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -84.68 (s, 3F), -110.69 (s, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 140.52, 135.32, 130.93 (t, J = 7.7 Hz), 125.49, 125.47, 124.26 (t, J = 26.2 Hz), 123.41 (m), 122.92, 119.36 (qt, J = 286.22, 39.07 Hz), δ 113.06 (tq, J = 252.92, 39.66 Hz).
MS (**EI**, *m/z*): 252 (M^+);
HRMS (**EI**): Calcd for: $C_{10}H_5F_5S^+$, 252.0032, found: 252.0028.
IR (film): 3112, 2926, 2852, 1559, 1524, 1463, 1428, 1369, 1331, 1201, 1174, 1153, 1128, 1069, 1044, 920, 842, 757 cm^{-1}.

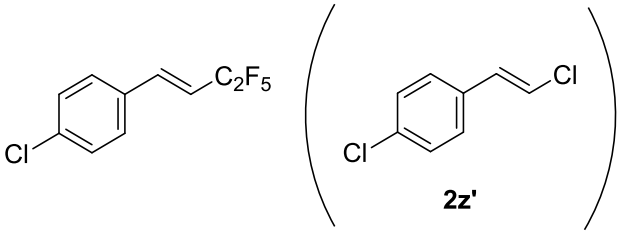
(*E*)-4-(3,3,4,4,4-pentafluorobut-1-en-1-yl)-1,1'-biphenyl (2y):



White solid (80 mg, 67%). Mp: 107-109 ^{o}C; **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.61-7.55 (m, 4H), 7.49 (d, J = 7.9 Hz, 2H), 7.43 (t, J = 7.5 Hz, 2H), 7.35 (t, J = 6.8 Hz, 1H), 7.19 (d, J = 16.3 Hz, 1H), 6.19 (dd, J = 27.9, 12.0 Hz, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -85.53 (s, 3F), -115.23 (d, J = 12.0 Hz, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 143.13, 140.22, 139.35 (t, J = 9.1 Hz), 132.59, 129.07, 128.25, 128.04, 127.73, 127.20, 119.25 (qt, J = 285.6, 38.6 Hz), 114.05 (t, J = 23.1 Hz), 112.99 (tq, J = 248.9, 38.5 Hz);

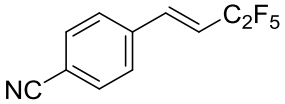
MS (**EI**, *m/z*): 298 (M^+);
HRMS (**EI**): Calcd for: $C_{16}H_{11}F_5^+$, 298.0781, found: 298.0789.
All the characterization data are consistent with the previous report[16].

(*E*)-1-chloro-4-(3,3,4,4,4-pentafluorobut-1-en-1-yl)benzene (2z):



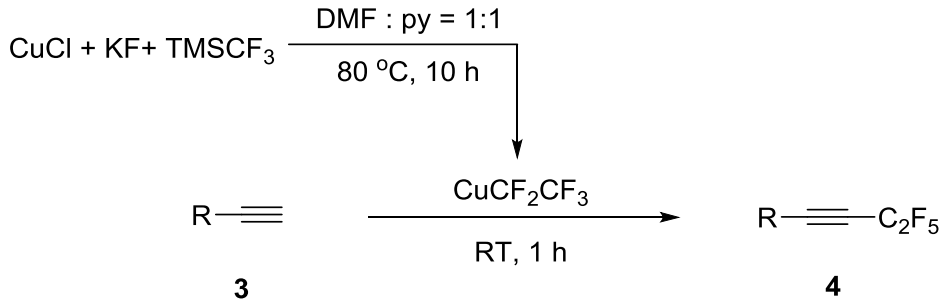
Yellow liquid (72 mg, 70%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.40 (d, J = 8.6 Hz, 2H), 7.37 (d, J = 8.8 Hz, 2H), 7.13 (dd, J = 16.2, 2.0 Hz, 1H), 6.15 (dt, J = 16.2, 11.8 Hz, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -85.55 (d, J = 2.2 Hz, 3F), -114.38 - -117.42 (m, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 138.55 (t, J = 9.3 Hz), 136.31, 132.13, 129.38, 128.97, 119.12 (qt, J = 285.6, 38.4 Hz), 114.86 (t, J = 23.2 Hz), 112.76 (tq, J = 249.2, 38.7 Hz).
MS (**FI**, *m/z*): 256 (M^+);
HRMS (**FI**): Calcd for: $C_{10}H_6F_5Cl^+$, 256.0073, found: 256.0071.
IR (film): 2927, 2857, 1661, 1595, 1493, 1408, 1344, 1324, 1304, 1196, 1099, 1036, 1014, 972, 809, 792 cm^{-1}.

(*E*)-4-(3,3,4,4,4-pentafluorobut-1-en-1-yl)benzonitrile (2aa):



Yellow liquid (91 mg, 92%); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.68 (d, J = 8.4 Hz, 2H), 7.57 (d, J = 8.2 Hz, 2H), 7.20 (d, J = 16.2 Hz, 1H), 6.30 (dt, J = 16.2, 11.6 Hz, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ -85.37 (s, 3F), -115.90 (d, J = 11.6 Hz, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 138.00 (t, J = 9.2 Hz), 137.77, 132.82, 128.24, 118.29, 118.94 (qt, J = 283.8, 37.9 Hz), 117.89 (t, J = 23.3 Hz), 113.71, 112.40 (tq, J = 249.6, 38.8 Hz);
MS (**EI**, *m/z*): 247 (M^+);
HRMS (**EI**): Calcd for: $C_{11}H_6F_5N^+$, 247.0420, found: 247.0429.
IR (film): 3066, 2928, 2857, 2230, 1662, 1607, 1563, 1504, 1413, 1345, 1329, 1303, 1197, 1104, 1037, 975, 955, 820, 686 cm^{-1}.

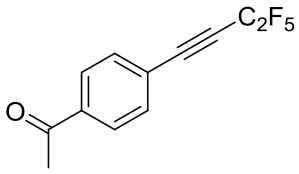
4.2 General Procedures for Pentafluoroethylation of Acetylenes



Typical Procedures:

To an oven-dried sealed tube were added CuCl (2.25 mmol, 222.9 mg, 5.0 equiv) and KF (1.5 mmol, 87.2 mg, 3.3 equiv) in glove box. Then in fume hood, DMF (3 mL), $TMSCF_3$ (1.5 mmol, 220 μL, 3.3 equiv) and pyridine (3 mL) were successively added under N_2 atmosphere. After stirred at room temperature for 5 minutes, the reaction mixture was immersed into 80 °C oil bath and stirred for 10 hours. Then the reaction mixture was cooled to room temperature and stirred under air for 5 minutes. The mixture turned black, and alkyne **3** (0.45 mmol, 1.0 equiv) dissolved in DMF (1.5 mL) was added slowly in 15 minutes using a syringe pump. The reaction mixture was stirred at room temperature under air for additional 1 hour. After the reaction was completed, the reaction mixture was quenched with 50 mL 3.0 M HCl solution or 20 mL ammonium hydroxide, extracted with CH_2Cl_2 (30 mL×3). The combined organic layer was washed with H_2O (30 mL×2) and brine (40 mL), dried over Na_2SO_4, concentrated under vacuum. The residue was purified by column chromatography on silica gel to afford **4**.

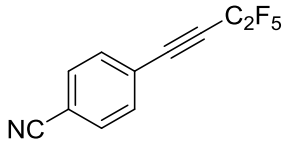
1-(4-(Perfluorobut-1-yn-1-yl)phenyl)ethan-1-one (4b)



Quenched with 3.0 M HCl, eluted with pentane/dichloromethane to give **4b** (71 mg, 60% yield).
Yellow solid. Mp: 35–37 °C. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.98 (d, J = 8.4 Hz, 2H), 7.67 (d, J = 8.4 Hz, 2H), 2.62 (s, 3H);

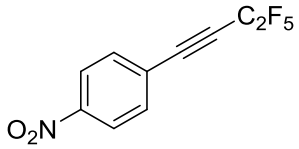
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.6 (t, J = 4.1 Hz, 3F), –102.2 (q, J = 4.1 Hz, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 197.0, 138.7, 132.9 (t, J = 2.5 Hz), 128.5, 123.0 (t, J = 3.1 Hz), 118.0 (qt, J = 280.0, 34.2 Hz), 105.6 (tq, J = 244.0, 42.0 Hz), 90.1 (t, J = 6.3 Hz), 77.0 (t, J = 36.4 Hz), 26.8;
MS (**EI**, *m/z*, %): 262 (M^+, 21.25), 247 (100.00);
HRMS (**EI**): Calcd. For $C_{12}H_7OF_5$: 262.0412; Found: 262.0415
IR (film): 2928, 2247, 1693, 1605, 1562, 1431, 1361, 1283, 1262, 1218, 1184, 1152, 1117, 1039, 959, 832, 758, 695, 646, 591 cm^{-1}.

4-(Perfluorobut-1-yn-1-yl)benzonitrile (4c)



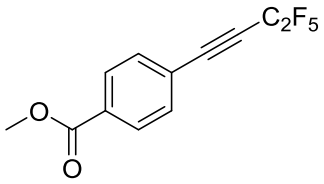
Quenched with 3.0 M HCl, eluted with pentane/dichloromethane to give **4c** (59 mg, 53% yield).
Yellow solid. Mp: 35–38 ^oC. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.72 (d, J = 8.4 Hz, 2H), 7.69 (d, J = 8.4 Hz, 2H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.6 (t, J = 4.1 Hz, 3F), –102.6 (q, J = 4.5 Hz, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 133.2 (t, J = 2.3 Hz), 132.4, 123.2 (t, J = 3.0 Hz), 117.9 (qt, J = 283.9, 36.6 Hz), 117.7, 114.9, 105.4 (tq, J = 244.5, 42.0 Hz), 88.9 (t, J = 6.3 Hz), 77.9 (t, J = 36.6 Hz);
MS (**EI**, *m/z*, %): 245 (M^+, 30.21), 176 (100.00);
HRMS (**EI**): Calcd. For $C_{11}H_4NF_5$: 245.0258; Found: 245.0267;
IR (film): 2925, 2251, 1276, 1260, 1232, 1154, 1040, 836, 764, 750 cm^{-1}.

1-Nitro-4-(perfluorobut-1-yn-1-yl)benzene (4d)



Quenched with 3.0 M HCl, eluted with pentane/ethyl acetate to give **4d** (62 mg, 52% yield).
Yellow solid. Mp: 63–66 ^oC. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.29 (d, J = 8.8 Hz, 2H), 7.77 (d, J = 8.4 Hz, 2H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.5 (t, J = 4.5 Hz, 3F), –102.7 (q, J = 4.1 Hz, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.1, 133.7 (t, J = 2.4 Hz), 125.0 (t, J = 2.9 Hz), 124.0, 117.9 (qt, J = 283.9, 36.6 Hz), 105.4 (tq, J = 244.8, 42.2 Hz), 88.6 (t, J = 6.1 Hz), 78.4 (t, J = 36.7 Hz);
MS (**EI**, *m/z*, %): 265 (M^+, 64.46), 169 (100.00);
HRMS (**EI**): Calcd. For $C_{10}H_4NO_2F_5$: 265.0157; Found: 265.0157;
IR (film): 3113, 2955, 2925, 2855, 2249, 1603, 1536, 1491, 1405, 1374, 1352, 1284, 116, 1154, 1118, 1040 913, 858, 818, 748, 686 cm^{-1}.

Methyl 4-(perfluorobut-1-yn-1-yl)benzoate (4e)



Quenched with 3.0 M HCl, eluted with pentane/dichloromethane to give **4e** (90 mg, 72% yield).

Yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.07 (d, J = 8.0 Hz, 2H), 7.65 (d, J = 8.0 Hz, 2H), 3.95(s, 3H);

^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.2 (t, J = 4.1 Hz, 3F), –101.7 (q, J = 4.1 Hz, 2F);

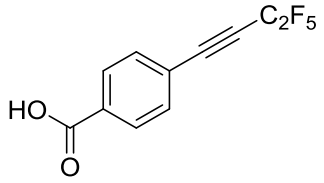
^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.0, 132.6 (t, J = 2.2 Hz), 132.4, 129.8, 122.9 (t, J = 2.8 Hz), 118.0 (qt, J = 283.8, 36.9 Hz), 105.6 (tq, J = 244.0, 42.1 Hz), 90.2 (t, J = 6.2 Hz), 76.7 (t, J = 36.3 Hz), 52.6;

MS (**EI**, *m/z*, %): 278 (M^+, 32.35), 247 (100.00);

HRMS (**EI**): Calcd. For $C_{12}H_7O_2F_5$: 278.0361; Found: 278.0358;

IR (film): 2968, 2247, 1731, 1613, 1438, 1406, 1345, 1282, 1219, 1179, 1152, 1116, 1039, 1019, 918, 859, 809, 768, 747, 693, 674 cm^{-1}.

4-(Perfluorobut-1-yn-1-yl)benzoic acid (4f)



Quenched with 3.0 M HCl, eluted with petroleum ether/ethyl acetate to give **4f** (53 mg, 45% yield).

White solid. Mp: 192–194 ^{o}C. **^{1}H NMR** (DMSO-d_6, 400 MHz) δ 8.02 (d, J = 8.4 Hz, 2H), 7.81 (d, J = 8.0 Hz, 2H);

^{19}F NMR (DMSO-d_6, 376 MHz) δ –84.8 (t, J = 4.9 Hz, 3F), –100.6 (q, J = 4.5 Hz , 2F);

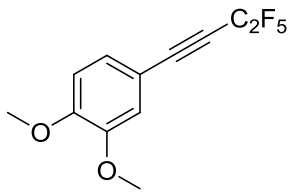
^{13}C NMR (DMSO-d_6, 100 MHz) δ 166.3, 133.7, 132.9 (t, J = 2.3 Hz), 129.8, 120.7 (t, J = 2.9 Hz), 117.5 (qt, J = 283.9, 37.3 Hz), 105.2 (tq, J = 242.8, 41.8 Hz), 91.6 (t, J = 6.3 Hz), 74.7 (t, J = 36.1 Hz);

MS (**EI**, *m/z*, %): 264 (M^+, 49.83), 195 (100.00);

HRMS (**EI**): Calcd. For $C_{11}H_5O_2F_5$: 264.0204; Found: 264.0205;

IR (film): 3000, 2840, 2671, 2563, 2252, 1684, 1612, 1564, 1428, 1317, 1285, 1213, 1182, 1148, 1110, 1040, 927, 864, 770, 695 cm^{-1}.

1,2-Dimethoxy-4-(perfluorobut-1-yn-1-yl)benzene (4g)



Quenched with 3.0 M HCl, eluted with petroleum ether/ethyl acetate to give **4g** (103 mg, 82% yield).

Colorless oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.20 (d, J = 8.0 Hz, 1H), 7.01 (s, 1H), 6.85 (d, J = 8.4 Hz, 1H), 3.92 (s, 3H), 3.90 (s, 3H);

^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.7 (t, J = 4.5 Hz, 3F), –101.0 (q, J = 4.5 Hz, 2F);

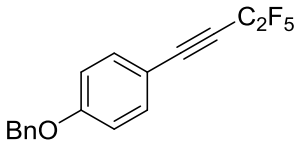
^{13}C NMR ($CDCl_3$, 100 MHz) δ 151.8, 149.0, 126.6 (t, J = 2.7 Hz), 118.1 (qt, J = 283.7, 37.6 Hz), 114.6, 111.1, 110.3, 105.8 (tq, J = 242.6, 41.9 Hz), 92.1 (t, J = 6.1 Hz), 73.4 (t, J = 36.2 Hz), 55.9, 55.8;

MS (**EI**, *m/z*, %): 280 (M^+,100.00), 247 (73.9);

HRMS (**EI**): Calcd. For $C_{12}H_9O_2F_5$: 280.0517; Found: 280.0522

IR (film): 3008, 2941, 2842, 2238, 1599, 1580, 1516, 1466, 1444, 1414, 1348, 1302, 1253, 1216, 1181, 1133, 1044, 935, 855, 809, 764, 717, 653, 619 cm^{-1}.

1-(Benzyloxy)-4-(perfluorobut-1-yn-1-yl)benzene (4h)



Quenched with 3.0 M HCl, eluted with petroleum ether to give **4h** (122 mg, 83% yield).

White solid. Mp: 36–37 ^{o}C. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.51 (d, J = 8.4 Hz, 2H), 7.45–7.31 (m, 5H), 6.97 (d, J = 8.4 Hz, 2H), 5.10 (s, 2H);

^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.7 (t, J = 4.5 Hz, 3F), –102.2 (q, J = 4.5 Hz, 2F);

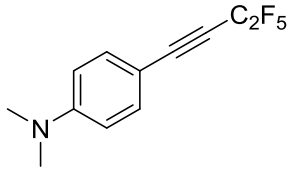
^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.0, 136.2, 134.4 (t, J = 2.4 Hz), 128.9, 128.4, 127.6, 118.2 (qt, J = 283.7, 37.6 Hz), 115.4, 110.7 (t, J = 3.0 Hz), 105.9 (tq, J = 242.6, 41.8 Hz), 92.1 (t, J = 6.1 Hz), 73.9 (t, J = 36.1 Hz), 70.3;

MS (**EI**, *m/z*, %): 326 (M^+, 5.8), 91 (100.00);

HRMS (**EI**): Calcd. For $C_{17}H_{11}OF_5$: 326.0725; Found: 326.0728.

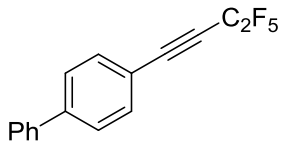
IR (film): 2933, 2239, 1605, 1510, 1452, 1285, 1252, 1215, 1146,1109, 1038, 832, 731, 696 cm^{-1}.

***N,N*-dimethyl-4-(perfluorobut-1-yn-1-yl)aniline (4i)**



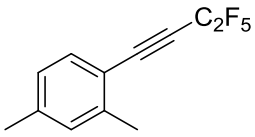
Quenched with ammonium hydroxide, eluted with petroleum ether/ethyl acetate to give **4i** (78 mg, 66% yield).
Yellow solid. Mp: 75–77 °C. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.41 (d, J = 8.8 Hz, 2H), 6.62 (d, J = 8.8 Hz, 2H), 3.02 (s, 6H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.7 (t, J = 4.5 Hz, 3F), –100.0 (q, J = 4.9 Hz , 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 151.8, 134.0 (t, J = 2.5 Hz), 118.4 (qt, J = 283.7, 38.0 Hz), 111.6, 106.3 (tq, J = 241.7, 41.6 Hz), 104.4 (t, J = 3.0 Hz), 94.2 (t, J = 6.4 Hz), 73.2 (t, J = 36.1 Hz), 40.0;
MS (**EI**, *m/z*, %): 263 (M^+, 100.00), 194 (78.57);
HRMS (**EI**): Calcd. For $C_{12}H_{10}NF_5$: 263.0728; Found: 263.0739;
IR (film): 2922, 2827, 2229, 1609, 1528, 1447, 1373, 1302, 1210, 1139, 1100, 1066, 1036, 816, 804, 703, 534 cm^{-1}.

4-(Perfluorobut-1-yn-1-yl)-1,1'-biphenyl (4j)



Quenched with 3.0 M HCl, eluted with petroleum ether to give **4j** (104 mg, 78% yield).
White solid. Mp: 59–61°C. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.68–7.58 (m, 6H), 7.48 (t, J = 7.2 Hz, 2H), 7.40 (t, J = 7.2 Hz, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.7 (t, J = 4.1 Hz, 3F), –101.5 (q, J = 4.1 Hz, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 144.0, 139.8, 133.1 (t, J = 2.7 Hz), 129.2, 128.4, 127.4, 127.3, 118.2 (qt, J = 283.8, 37.3 Hz), 117.3 (t, J = 3.1 Hz), 105.9 (tq, J = 243.1, 41.9 Hz), 91.6 (t, J = 6.2 Hz), 75.2 (t, J = 36.1 Hz);
MS (**EI**, *m/z*, %): 296 (M^+, 78.71), 227 (100.00);
HRMS (**EI**): Calcd. For $C_{16}H_{19}F_5$: 296.0619; Found: 296.0630.
IR (film): 3089, 3041, 2243, 1486, 1447, 1405, 1344, 1292, 1214, 1151, 1120, 1042, 1020, 841, 764, 720, 710, 692, 558 cm^{-1}.

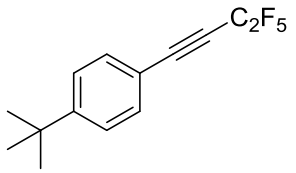
2,4-Dimethyl-1-(perfluorobut-1-yn-1-yl)benzene (4k)



Quenched with 3.0 M HCl, eluted with pentane to give **4k** (79 mg, 71% yield).
Colorless oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.41 (d, J = 8.0 Hz, 1H), 7.07 (s, 1H), 7.02 (d, J = 8.0 Hz, 1H), 2.41 (s, 3H), 2.35 (s, 3H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.8 (t, J = 4.1 Hz, 3F), –101.2 (q, J = 4.1 Hz, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 142.0 (t, J = 2.1 Hz), 141.8, 132.9 (t, J = 2.5 Hz), 130.9, 126.9, 118.3 (qt, J = 283.7, 37.5 Hz), 115.5 (t, J = 2.8 Hz), 106.0 (tq, J = 242.6,

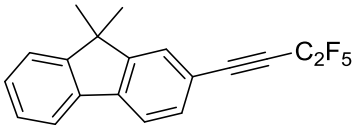
41.8 Hz), 91.4 (t, *J* = 5.2 Hz), 77. 9 (t, *J* = 36.2 Hz), 21.7, 20.2;
MS (**EI**, *m/z*, %): 248 (M^+, 100.00), 179 (87.39);
HRMS (**EI**): Calcd. For $C_{12}H_9F_5$: 248.0619; Found: 248.0625;
IR (film): 2927, 2239, 1612, 1497, 1452, 1345, 1291, 1222, 1068, 1113, 1037, 933, 817, 802, 735, 715, 657, 565 cm^{-1}.

1-(*tert*-Butyl)-4-(perfluorobut-1-yn-1-yl)benzene (4l)



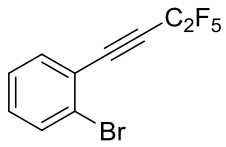
Quenched with 3.0 M HCl, eluted with pentane to give **4l** (96 mg, 77% yield).
Colorless oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.50 (d, *J* = 8.4 Hz, 2H), 7.42 (d, *J* = 8.4 Hz, 2H), 1.32 (s, 9H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.8 (t, *J* = 4.9 Hz, 3F), –101.3 (q, *J* = 4.1 Hz, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 155.0, 132.5 (t, *J* = 2.3 Hz), 125.9, 118.2 (qt, *J* = 283.7, 37.4 Hz), 115.7 (t, *J* = 2.9 Hz), 105.9 (tq, *J* = 242.8, 41.8 Hz), 92.0 (t, *J* = 6.1 Hz), 74.3 (t, *J* = 35.9 Hz), 35.2, 31.2;
MS (**EI**, *m/z*, %): 276 (M^+, 19.32), 261 (100.00);
HRMS (**EI**): Calcd. For $C_{14}H_{13}F_5$: 276.0932; Found: 276.0944;
IR (film): 2968, 2873, 2244, 1605, 1509, 1367, 1346, 1290, 1268, 1221, 1154, 1107, 1039, 913, 847, 836, 747, 685, 564 cm^{-1}.

9,9-Dimethyl-2-(perfluorobut-1-yn-1-yl)-9H-fluorene (4m)



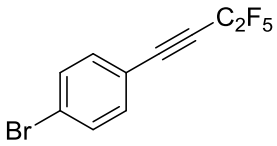
Quenched with 3.0 M HCl, eluted with petroleum ether to give **4m** (120 mg, 79% yield).
Colorless oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.78–7.71 (m, 2H), 7.63(s, 1H), 7.56 (d, *J* = 8.0 Hz, 1H), 7.50–7.44 (m, 1H), 7.42–7.35 (m, 2H), 1.50 (s, 6H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.6 (t, *J* = 4.5 Hz, 3F), –101.1 (q, *J* = 4.5 Hz , 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 154.3, 154.0, 142.4, 137.9, 132.0 (t, *J* = 2.4 Hz), 128.8, 127.5, 127.0 (t, *J* = 2.5 Hz), 123.0, 120.9, 120.3, 118.3 (qt, *J* = 283.7, 37.4 Hz), 116.7 (t, *J* = 3.0 Hz), 106.0 (tq, *J* = 242.9, 41.9 Hz), 92.7 (t, *J* = 6.4 Hz), 74.6 (t, *J* = 36.1 Hz), 47.2, 27.0;
MS (**EI**, *m/z*, %): 336 (M^+, 71.04), 321 (100.00);
HRMS (**EI**): Calcd. For $C_{19}H_{13}F_5$: 336.0932; Found: 336.0932;
IR (film): 2965, 2926, 2877, 2241, 1609, 1470, 1462, 1448, 1346, 1303, 1291, 1216, 1150, 1111, 1077, 1039, 1005, 913, 884, 833, 781, 758, 736, 721, 708, 567 cm^{-1}.

1-Bromo-2-(perfluorobut-1-yn-1-yl)benzene (4n)



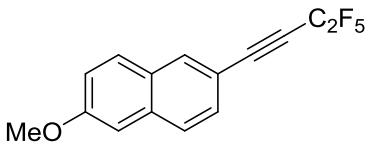
Quenched with 3.0 M HCl, eluted with pentane to give **4n** (97 mg, 72% yield).
Colorless oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.69–7.62 (m, 1H), 7.61–7.54 (m, 1H), 7.39–7.30 (m, 2H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.5 (t, J = 4.1 Hz, 3F), –102.3 (q, J = 4.1 Hz, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 134.5 (t, J = 2.3 Hz), 133.0, 132.3, 127.4, 126.4 (t, J = 2.6 Hz), 121.3 (t, J = 3.0 Hz), 118.1 (qt, J = 284.0, 36.8 Hz), 105.7 (tq, J = 243.7, 42.1 Hz), 89.6 (t, J = 6.2 Hz), 78.5 (t, J = 36.5Hz);
MS (**EI**, *m/z*, %): 298 (M^+, 43), 229 (100.00);
HRMS (**EI**): Calcd. For $C_{10}H_4F_5Br$: 297.9411; Found: 297.9421;
IR (film): 2972, 2933, 2873, 2249, 1471, 1346, 1295, 1251, 1228, 1157, 1118, 1054, 1035, 913, 836, 748 cm^{-1}.

1-Bromo-4-(perfluorobut-1-yn-1-yl)benzene (4o)



Quenched with 3.0 M HCl, eluted with pentane to give **4o** (87 mg, 65% yield).
Colorless oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.56 (d, J = 8.4 Hz, 2H), 7.44 (d, J = 8.0 Hz, 2H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.7 (t, J = 4.1 Hz, 3F), –101.1 (q, J = 4.5 Hz , 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 134.0 (t, J = 2.5 Hz), 132.3, 126.1, 118.0 (qt, J = 283.7, 37.0 Hz), 117.6 (t, J = 2.9 Hz), 105.7 (tq, J = 243.6, 42.2 Hz), 90.3 (t, J = 6.3 Hz), 75.7 (t, J = 36.3 Hz);
MS (**EI**, *m/z*, %): 298 (M^+, 58.09), 229 (100.00);
HRMS (**EI**): Calcd. For $C_{10}H_4F_5Br$: 297.9411; Found: 297.9418;
IR (film): 2926, 2855, 2248, 1589, 1489, 1395, 1344, 1286, 1220, 1150, 1118, 1072, 1039, 1013, 910, 825, 749, 681 cm^{-1}.

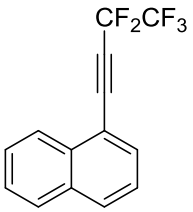
2-Methoxy-6-(perfluorobut-1-yn-1-yl)naphthalene (4p)



Quenched with 3.0 M HCl, eluted with petroleum ether to give **4p** (106 mg, 78% yield).
Red solid. Mp: 51–53 ^{o}C. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.05 (s, 1H), 7.74 (d, J = 3.6

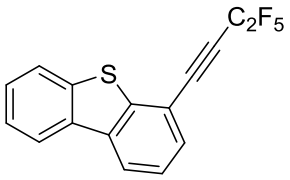
Hz, 1H), 7.72 (d, *J* = 3.2 Hz, 1H), 7.51 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.21 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.13 (d, *J* = 2.8 Hz, 1H), 3.94 (s, 3H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.7 (t, *J* = 4.5 Hz, 3F), –101.1 (q, *J* = 4.5 Hz , 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.6, 135.7, 133.7 (t, *J* = 2.7 Hz), 129.8, 128.4 (t, *J* = 2.2 Hz), 128.1, 127.4, 120.3, 118.3 (qt, *J* = 283.8, 37.5 Hz), 113.2 (t, *J* = 3.1 Hz), 106.0, 105.9 (tq, *J* = 242.8, 41.9 Hz), 92.5 (t, *J* = 6.4 Hz), 79.2 (t, *J* = 36.1 Hz), 55.5;
MS (**EI**, *m/z*, %): 300 (M^+, 100.00), 231 (65.18);
HRMS (**EI**): Calcd. For $C_{15}H_9OF_5$: 300.0568; Found: 300.0580;
IR (film): 3004, 2970, 2844, 2237, 1630, 1604, 1500, 1484, 1415, 1393, 1336, 1288, 1263, 1220, 1166, 1142, 1109, 1037, 968, 894, 847, 801, 744, 693, 472 cm^{-1}.

1-(Perfluorobut-1-yn-1-yl)naphthalene (4q)



Quenched with 3.0 M HCl, eluted with petroleum ether to give **4q** (92 mg, 76% yield).
Colorless oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.19 (d, *J* = 8.4 Hz, 1H), 7.99 (d, *J* = 8.4 Hz, 1H), 7.91 (d, *J* = 8.0 Hz, 1H), 7.84 (d, *J* = 6.8 Hz, 1H), 7.66 (t, *J* = 8.0 Hz, 1H), 7.59 (t, *J* = 8.0 Hz, 1H), 7.49 (t, *J* = 8.0 Hz, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.6 (t, *J* = 4.5 Hz, 3F), –101.4 (q, *J* = 4.5 Hz, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 133.3 (t, *J* = 1.4 Hz), 133.1, 132.8 (t, *J* = 2.7 Hz), 131.9, 128.8, 128.1, 127.2, 125.3, 125.1, 118.3 (qt, *J* = 283.7, 37.2 Hz), 116.1 (t, *J* = 2.9 Hz), 106.0 (tq, *J* = 243.2, 41.9 Hz), 90.3 (t, *J* = 6.6 Hz), 79.2 (t, *J* = 36.3 Hz);
MS (**EI**, *m/z*, %): 270 (M^+, 76.9), 201 (100.00);
HRMS (**EI**): Calcd. For $C_{14}H_7F_5$: 270.0462; Found: 270.0466;
IR (film): 3062, 2238, 1585, 1512, 1400, 1350, 1292, 1218, 1192, 1165, 1155, 1112, 1054, 1022, 1006, 912, 877, 804, 771, 715, 659 cm^{-1}.

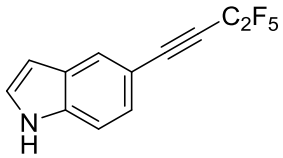
4-(Perfluorobut-1-yn-1-yl)dibenzo[*b,d*]thiophene (4r)



Quenched with 3.0 M HCl, eluted with petroleum ether to give **4r** (105 mg, 72% yield).
Yellow solid. Mp: 44–46 ^{o}C. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.13 (d, *J* = 8.0 Hz, 1H), 8.07 (d, *J* = 8.4 Hz, 1H), 7.84 (d, *J* = 7.2 Hz, 1H), 7.63 (d, *J* = 7.6 Hz, 1H), 7.54–7.44

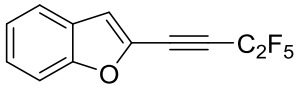
(m, 2H), 7.41 (t, *J* = 7.6 Hz, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.5 (t, *J* = 4.1 Hz, 3F), –101.9 (q, *J* = 4.1 Hz , 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 143.6 (t, *J* = 2.3 Hz), 139.3, 136.1, 135.1, 131.0 (t, *J* = 2.5 Hz), 127.6, 124.9, 124.5, 124.0, 123.0, 122.0, 118.2 (qt, *J* = 284.0, 37.0 Hz), 113.0 (t, *J* = 3.0 Hz), 105.9 (tq, *J* = 243.7, 42.1 Hz), 89.1 (t, *J* = 6.4 Hz), 79.3 (t, *J* = 36.4 Hz);
MS (**EI**, *m/z*, %): 326 (M^+, 66.65), 40 (100.00);
HRMS (**EI**): Calcd. For $C_{16}H_7F_5S$: 326.0183; Found: 326.0193;
IR (film): 3074, 2246, 1482, 1388, 1323, 1286, 1218, 1188, 1163, 1114, 1093, 1058, 1033, 1019, 888, 796, 749, 682 cm^{-1}.

5-(Perfluorobut-1-yn-1-yl)-1*H*-indole (4s)



Quenched with 3.0 M HCl, eluted with petroleum ether/ethyl acetate to give **4s** (79 mg, 68% yield).
Yellow solid. Mp: 55–57 °C. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.28 (br, 1H), 7.94 (s, 1H), 7.42–7.34 (m, 2H), 7.28 (t, *J* = 2.8 Hz, 1H), 6.61 (t, *J* = 2.4 Hz, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.7 (t, *J* = 4.5 Hz, 3F), –100.6 (q, *J* = 4.5 Hz , 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 136.9, 127.8, 126.6 (t, *J* = 2.6 Hz), 126.99, 125.96 (t, *J* = 2.2 Hz), 118.3 (qt, *J* = 283.7, 37.7 Hz), 111.6, 109.5 (t, *J* = 2.8 Hz), 106.1 (tq, *J* = 242.1, 41.6 Hz), 103.4, 94.1 (t, *J* = 6.3 Hz), 72.8 (t, *J* = 36.1 Hz);
MS (**EI**, *m/z*, %): 259 (M^+, 72.76), 190 (100.00);
HRMS (**EI**): Calcd. For $C_{12}H_6NF_5$: 259.0415; Found: 259.0422;
IR (film): 3489, 3432, 2237, 1618, 1472, 1415, 1346, 1299, 1259, 1216, 1194, 1138, 1116, 1034, 935, 890, 807, 764, 733, 664, 599 cm^{-1}.

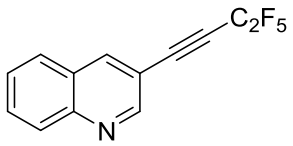
2-(Perfluorobut-1-yn-1-yl)benzofuran (4t)



Quenched with 3.0 M HCl, eluted with petroleum ether to give **4t** (70 mg, 60% yield).
Red oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.63 (d, *J* = 8.0 Hz, 1H), 7.51 (dd, *J* = 8.4, 0.8 Hz, 1H), 7.45 (td, *J* = 7.2, 1.2 Hz, 1H), 7.34–7.31 (m, 1H), 7.30–7.28 (m, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.4 (t, *J* = 4.1 Hz, 3F), –102.8 (q, *J* = 4.1 Hz , 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 155.8, 134.6 (t, *J* = 3.2 Hz), 127.6, 126.6, 124.2, 122.2, 118.0 (qt, *J* = 284.0, 36.6 Hz), 116.9 (t, *J* = 2.3 Hz), 111.9, 105.7 (tq, *J* = 244.9, 42.2 Hz), 81.9 (t, *J* = 6.3 Hz), 80.3 (t, *J* = 36.7 Hz);
MS (**EI**, *m/z*, %): 260 (M^+, 97.31), 191 (100.00);
HRMS (**EI**): Calcd. For $C_{12}H_5OF_5$: 260.0255; Found: 260.0263;

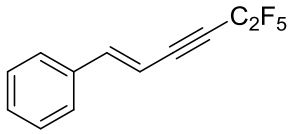
IR (film): 3074, 2244, 1448, 1290, 1214, 1149, 1123, 1041, 949, 825, 749, 741, 699 cm^{-1}.

3-(Perfluorobut-1-yn-1-yl)quinoline (4u)



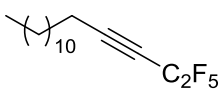
Quenched with ammonium hydroxide, eluted with petroleum ether/ethyl acetate to give **4u** (57 mg, 48% yield).
White solid. Mp: 99–100 ^{o}C. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.95 (s, 1H), 8.40 (s, 1H), 8.12 (d, J = 8.4 Hz, 1H), 7.80 (t, J = 7.6 Hz, 2H), 7.62 (t, J = 7.6 Hz, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.5 (t, J = 4.5 Hz, 3F), –102.0 (q, J = 4.1 Hz , 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 151.2 (t, J = 2.0 Hz), 148.1, 141.1 (t, J = 2.6 Hz), 131.9, 129.8, 128.1, 128.0, 126.6, 118.1 (qt, J = 284.0, 36.9 Hz), 112.7 (t, J = 3.0 Hz), 105.6 (tq, J = 244.2, 42.1 Hz), 88.8 (t, J = 6.2 Hz), 77.5 (t, J = 36.5 Hz);
MS (**EI**, *m/z*, %): 271 (M^+, 76.01), 202 (100.00);
HRMS (**EI**): Calcd. For $C_{13}H_6NF_5$: 271.0415; Found: 271.0413;
IR (film): 3048, 2239, 1621, 1569, 1491, 1356, 1333, 1287, 1209, 1150, 1113, 1039, 976, 914, 787, 753, 715, 499 cm^{-1}.

(*E*)-(5,5,6,6,6-pentafluorohex-1-en-3-yn-1-yl)benzene (4v)



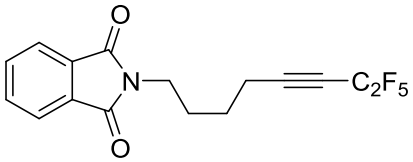
Quenched with 3.0 M HCl, eluted with pentane to give **4v** (83 mg, 75% yield).
Yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.45–7.36 (m, 5H), 7.25 (d, J = 16.4 Hz, 1H), 6.18 (td, J = 16.4, 4.4 Hz, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.7 (t, J = 4.5 Hz, 3F), –101.2 (m, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 147.9 (t, J = 3.6 Hz), 134.9, 130.4, 129.1, 127.1, 118.2 (qt, J = 283.7, 37.5 Hz), 105.8 (tq, J = 242.5, 41.9 Hz), 103.6 (t, J = 3.6 Hz), 91.1 (t, J = 6.6 Hz), 76.0 (t, J = 36.0 Hz);
MS (**EI**, *m/z*, %): 246 (M^+, 33.54), 177 (100.00);
HRMS (**EI**): Calcd. For $C_{12}H_7F_5$: 246.0462; Found: 246.0475;
IR (film): 3074, 3035, 2227, 1610, 1556, 1493, 1449, 1331, 1277, 1215, 1114, 1083, 1010, 981, 748, 698, 689 cm^{-1}.

1,1,1,2,2-Pentafluorohexadec-3-yne (4w)



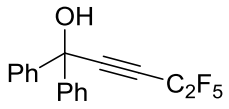
Quenched with 3.0 M HCl, eluted with pentane to give **4w** (110 mg, 79% yield).
Colorless oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 2.39–2.29 (m, 2H), 1.63–1.55 (m, 2H), 1.44–1.35 (m, 2H), 1.34–1.20 (m, 16H), 0.88 (t, J = 6.8 Hz, 3H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –86.1 (t, J = 4.1 Hz, 3F), –100.9 (m, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 118.2 (qt, J = 283.3, 37.4 Hz), 105.2 (tq, J = 241.9, 41.8 Hz), 94.6 (t, J = 6.0 Hz), 67.5 (t, J = 35.8 Hz), 32.2, 29.9 (2 carbon), 29.8, 29.64, 29.61, 29.2, 28.9, 27.5 (t, J = 2.1 Hz), 22.9, 18.6 (t, J = 2.7 Hz), 14.2;
MS (**EI**, *m/z*, %): 57 (100), 55 (58.96);
HRMS (**EI**): Calcd. For $C_{16}H_{25}F_5$: 312.1871; Found: 312.1877;
IR (film): 2928, 2857, 2256, 1467, 1350, 1321, 1253, 1214, 1113, 1074, 942, 756, 662 cm^{-1}.

2-(7,7,8,8,8-Pentafluorooct-5-yn-1-yl)isoindoline-1,3-dione (4x)



Quenched with 3.0 M HCl, eluted with petroleum ether/ethyl acetate to give **4x** (133 mg, 86% yield).
White solid. Mp: 84–85 ^oC. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.89–7.82 (m, 2H), 7.77–7.68 (m, 2H), 3.72 (t, J = 6.8 Hz, 2H), 2.48–2.37 (m, 2H), 1.88–1.75 (m, 2H), 1.71–1.60 (m, 2H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –86.0 (t, J = 4.1 Hz, 3F), –101.1 (m, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 168.4, 134.0, 132.1, 123.3, 117.9 (qt, J = 283.7, 37.6 Hz), 105.0 (tq, J = 242.1, 41.7 Hz), 93.6 (t, J = 6.0 Hz), 67.7 (t, J = 35.9 Hz), 37.1, 27.7, 24.6 (t, J = 2.2 Hz), 18.0 (t, J =2.6 Hz);
MS (**EI**, *m/z*, %): 345 (M^+, 11.52), 160 (100.00);
HRMS (**EI**): Calcd. For $C_{16}H_{12}O_2F_5$: 345.0783; Found: 345.0778.
IR (film): 2933, 2868, 2259, 1778, 1703, 1616, 1467, 1403, 1366, 1331, 1255, 1207, 1102, 1069, 1038, 1026, 1000, 915, 745, 719, 530 cm^{-1}.

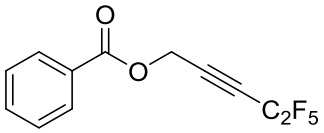
4,4,5,5,5-Pentafluoro-1,1-diphenylpent-2-yn-1-ol (4y)



Quenched with 3.0 M HCl, eluted with petroleum ether/ethyl acetate to give **4y** (73 mg, 50% yield).
Yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.53 (d, J = 7.6 Hz, 4H), 7.41–7.30 (m, 6H), 2.92 (br, 1H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.6 (t, J = 4.1 Hz, 3F), –102.8 (q, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 142.6, 128.8, 128.7, 126.0, 118.0 (qt, J = 283.8, 36.8

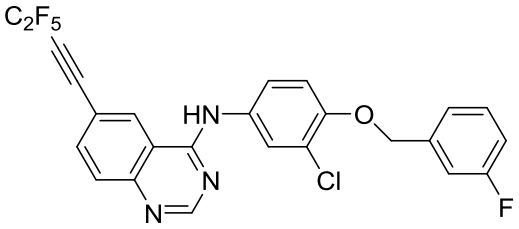
Hz), 105.3 (tq, *J* = 244.5, 42.1 Hz), 94.5 (t, *J* = 6.3 Hz), 74.6, 73.1 (t, *J* = 36.6 Hz);
MS (**EI**, *m/z*, %): 326 (M^+, 13.28), 207 (100.00);
HRMS (**EI**): Calcd. For $C_{17}H_{11}OF_5$: 326.0725; Found: 326.0734;
IR (film): 3536, 3442, 3045, 2253, 1601, 1492, 1451, 1336, 1215, 1120, 1070, 1029, 983, 890, 764, 714, 697 cm^{-1}.

4,4,5,5,5-Pentafluoropent-2-yn-1-yl benzoate (4z)



Quenched with 3.0 M HCl, eluted with pentane/dichloromethane to give **4z** (88 mg, 71% yield).
Yellow oil. **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 8.11–8.04 (m, 2H), 7.62 (tt, *J* = 6.8, 1.2 Hz, 1H), 7.52–7.45 (m, 2H), 5.06 (t, *J* = 4.4 Hz, 2H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.8 (t, *J* = 3.8 Hz, 3F), –103.1 (m, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.5, 133.9, 130.1, 128.8, 128.7, 117.8 (qt, *J* = 283.7, 36.5 Hz), 104.8 (tq, *J* = 244.4, 42.2 Hz), 86.4 (t, *J* = 6.3 Hz), 72.5 (t, *J* = 36.2 Hz), 51.4;
MS (**EI**, *m/z*, %): 278 (M^+, 19.21), 105 (100.00);
HRMS (**EI**): Calcd. For $C_{12}H_7O_2F_5$: 278.0361; Found: 278.0372;
IR (film): 3078, 2931, 2853, 2266, 1735, 1603, 1453, 1372, 1342, 1317, 1269, 1247, 1216, 1178, 1122, 1095, 1077, 1028, 984, 937, 758, 710, 662 cm^{-1}.

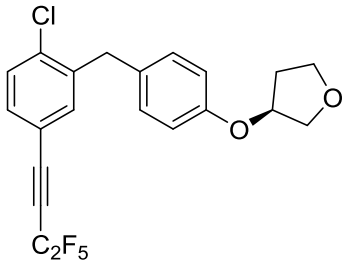
***N*-(3-chloro-4-((3-fluorobenzyl)oxy)phenyl)-6-(perfluorobut-1-yn-1-yl)quinazolin-4-amine (4aa)**



Quenched with ammonium hydroxide, eluted with petroleum ether/ethyl acetate to give **4aa** (167 mg, 71% yield).
Yellow solid. Mp: 140–141 ^{o}C. **^{1}H NMR** (DMSO-d_6, 400 MHz) δ 9.82 (s, 1H), 8.80 (s, 1H), 8.56 (s, 1H), 7.98 (d, *J* = 2.4 Hz, 1H), 7.85 (d, *J* = 8.8 Hz, 1H), 7.68 (d, *J* = 8.8 Hz, 2H), 7.41 (q, *J* = 8.0 Hz, 1H), 7.32–7.22 (m, 2H), 7.19–7.07 (m, 2H), 5.17 (s, 2H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.5 (t, *J* = 4.5 Hz, 3F), –101.9 (q, *J* = 4.1 Hz, 2F), –113.0 (m, 1F);
^{13}C NMR (DMSO-d_6, 100 MHz) δ 162.2 (d, *J* = 242.3 Hz), 157.0, 156.4, 150.9,

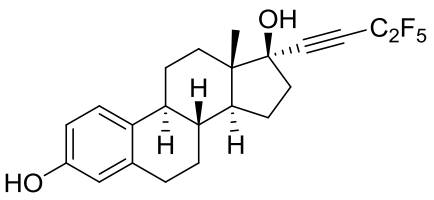
149.9, 139.6 (d, *J* = 7.5 Hz), 134.7, 132.7, 130.4 (d, *J* = 8.2 Hz), 129.2, 128.7, 123.9, 123.1, 121.9, 121.1, 117.6 (qt, *J* = 283.9, 37.4 Hz), 114.8, 114.6 (d, *J* = 20.8 Hz), 114.0 (2 carbon), 113.8 (d, *J* = 6.6 Hz), 105.3 (tq, *J* = 242.6, 41.7 Hz), 92.2 (t, *J* = 6.3 Hz), 73.5 (t, *J* = 36.0 Hz), 69.4;
MS (**ESI**, *m/z*): 521.9 ($M+H^+$);
HRMS (**ESI**): Calcd. For $C_{25}H_{15}ON_3ClF_6$: 522.0802 ($M+H^+$); Found: 522.0801;
IR (film): 3300, 3080, 2924, 2250, 1588, 1568, 1530, 1499, 1421, 1361, 1279, 1216, 1150, 1121, 1041, 918, 843, 782, 728 cm^{-1}.

(*S*)-3-(4-(2-chloro-5-(perfluorobut-1-yn-1-yl)benzyl)phenoxy)tetrahydrofuran (4ab)



Quenched with ammonium hydroxide, eluted with petroleum ether/ethyl acetate to give **4ab** (127 mg, 67% yield).
Yellow oil. $[\alpha]_D$ = 4.35 ($CHCl_3$, c = 0.9050 w/v%). **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.44–7.30 (m, 3H), 7.09 (d, *J* = 8.4 Hz, 2H), 6.81 (d, *J* = 8.4 Hz, 2H), 4.94–4.86 (m, 1H), 4.06–3.85 (m, 6H), 2.26–2.09 (m, 2H);
^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.6 (t, *J* = 4.1 Hz, 3F), –101.7 (q, *J* = 4.5 Hz, 2F);
^{13}C NMR ($CDCl_3$, 100 MHz) δ 156.3, 140.2, 137.6, 134.7 (t, *J* =2.4 Hz), 131.6 (t, *J* =2.5 Hz), 130.7, 130.2, 130.1, 118.0 (qt, *J* = 283.9, 37.2 Hz), 117.2 (t, *J* = 3.0 Hz), 115.7, 105.6 (tq, *J* = 243.5, 41.9 Hz), 90.5 (t, *J* = 6.3 Hz), 77.5, 75.3 (t, *J* = 36.2 Hz), 73.2, 67.3, 38.2, 33.1;
MS (**EI**, *m/z*, %): 430 (M^+, 62.57), 71 (100.00);
HRMS (**EI**): Calcd. For $C_{21}H_{16}O_2F_5Cl$: 430.0753; Found: 430.0761;
IR (film): 2952, 2870, 2244, 1614, 1582, 1509, 1299, 1217, 1186, 1143, 1114, 1050, 1038, 823, 677 cm^{-1}.

(8*R*,9*S*,13*S*,14*S*,17*S*)-13-methyl-17-(perfluorobut-1-yn-1-yl)-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[*a*]phenanthrene-3,17-diol (4ac)



Quenched with 3.0 M HCl, eluted with petroleum ether/ethyl acetate to give **4ac** (125 mg, 67% yield).

White solid. Mp: 77–80 °C. $[\alpha]_D$ = –9.82 ($CHCl_3$, c = 0.66 w/v%). **^{1}H NMR** (DMSO-d_6, 400 MHz) δ 9.01 (s, 1H), 7.03 (d, J = 8.4 Hz, 1H), 6.52 (dd, J = 8.4, 2.0 Hz, 1H), 6.44 (d, J = 2.0 Hz, 1H), 6.10 (s, 1H), 2.90–2.60 (m, 2H), 2.29 (d, J = 10.8 Hz, 1H), 2.25–2.13 (m, 1H), 2.05–1.90 (m, 2H), 1.84–1.54 (m, 4H), 1.52–1.13 (m, 5H), 0.79 (s, 3H);

^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.8 (t, J = 4.1 Hz, 3F), –102.2 (m, 2F);

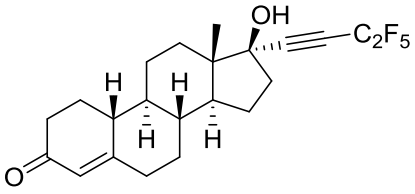
^{13}C NMR (DMSO-d_6, 100 MHz) δ 155.1, 137.1, 129.8, 126.1, 117.6 (qt, J = 283.8, 37.7 Hz), 115.0, 112.8, 105.0 (tq, J = 241.5, 41.5 Hz), 99.9 (t, J = 6.7 Hz), 78.5 (m, 2 carbon), 69.5 (t, J = 35.7 Hz), 49.8, 47.5, 43.5, 38.3, 32.7, 29.1, 27.1, 26.0, 22.5, 12.5;

MS (**ESI**, m/z): 415.1 ($M+H^+$);

HRMS (**ESI**): Calcd. For $C_{22}H_{24}O_2F_5$ ($M+H^+$): 415.1691; Found: 415.1691;

IR (film): 3379, 2933, 2872, 2246, 1610, 1582, 1499, 1448, 1342, 1286, 1209, 1116, 1070, 1049, 872, 675 cm^{-1}.

(8*R*,9*S*,10*R*,13*S*,14*S*,17*S*)-17-hydroxy-13-methyl-17-(perfluorobut-1-yn-1-yl)-1,2,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-3*H*-cyclopenta[*a*]phenanthren-3-one (4ad)



Quenched with 3.0 M HCl, eluted with petroleum ether/ethyl acetate to give **4ad** (159 mg, 76% yield).

White solid. Mp: 153–154 °C. $[\alpha]_D$ = 2.36 ($CHCl_3$, c = 0.93 w/v%). **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 5.83 (s, 1H), 3.48 (s, 1H), 2.52–2.19 (m, 6H), 2.14–1.98 (m, 2H), 1.91 (dd, J = 13.6, 2.4 Hz, 1H), 1.86–1.78 (m, 1H), 1.72 (d, J = 12.0 Hz, 2H), 1.61–1.48 (m, 2H), 1.45–1.20 (m, 4H), 1.15–1.00 (m, 1H), 0.92 (s, 3H), 0.88–0.75 (m, 1H);

^{19}F NMR ($CDCl_3$, 376 MHz) δ –85.8 (t, J = 4.1 Hz, 3F), –102.2 (m, 2F);

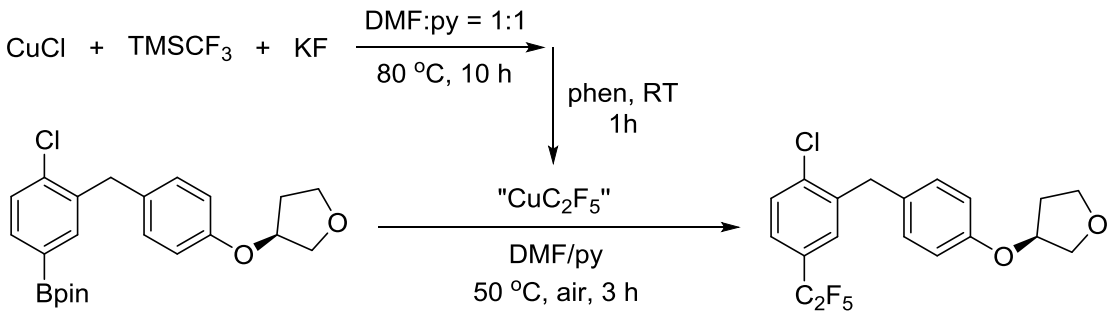
^{13}C NMR ($CDCl_3$, 100 MHz) δ 200.4, 166.8, 124.7, 117.9 (qt, J = 283.8, 37.1 Hz), 105.2 (tq, J = 243.3, 41.9 Hz), 96.5 (t, J = 6.5 Hz), 79.6, 72.1 (t, J = 36.3 Hz), 49.8, 49.2, 47.7, 42.6, 41.0, 38.6, 36.5, 35.4, 32.6, 30.7, 26.6, 26.2, 23.1, 12.7;

MS (**ESI**, m/z): 417.1 ($M+H^+$);

HRMS (**ESI**): Calcd. For $C_{22}H_{26}O_2F_5$ ($M+H^+$): 417.1847; Found: 417.1847;

IR (film): 3389, 2935, 2870, 2244, 1661, 1620, 1454, 1337, 1255, 1207, 1111, 1073, 1056, 1037, 970, 890, 671 cm^{-1}.

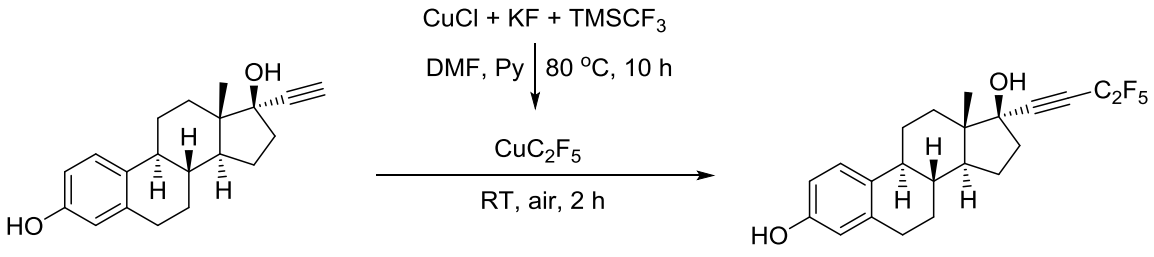
5. Procedures for Scale-up Syntheses



1h
2.0 mmol, 0.83 g

2h
70%, 570 mg

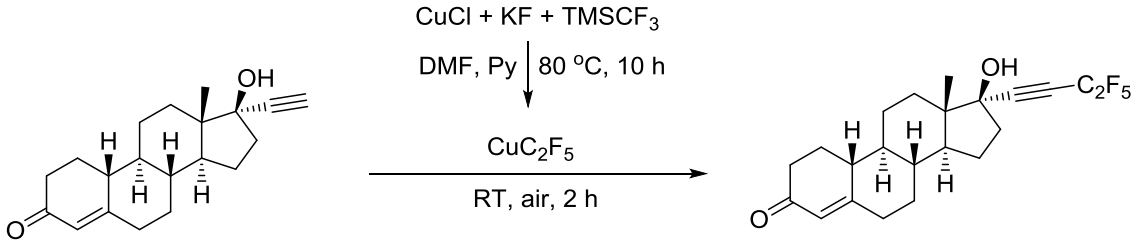
To an oven-dried sealed tube were added CuCl (22.5 mmol, 2.23 g, 11.2 equiv) and KF (15.0 mmol, 0.87 g, 7.5 equiv) in glove box. Then in fume hood, DMF (30 mL), $TMSCF_3$ (15.0 mmol, 2.2 mL, 7.5 equiv) and pyridine (30 mL) were successively added under N_2 atmosphere. After stirred at room temperature for 5 minutes, the reaction mixture was immersed into 80 °C oil bath and stirred for 10 h. Then the reaction mixture was cooled to room temperature and filtrated in the glove box. The filtrate was added in to the sealed tube containing phen (8.0 mmol, 1.44 g, 4.0 equiv). Then the reaction mixture was stirred at room temperature for 1 h. Under N_2 atmosphere, to a Schlenk flask was added **1h** (2.0 mmol, 0.83 g, 1.0 equiv), AgF (8.0 mmol, 1.01 g, 4.0 equiv) and the "$CuCF_2CF_3$" solution prepared above. After that, the reaction mixture was stirred 50 °C under air for 3 h. When the reaction was completed, the reaction mixture was quenched with 50 mL 3.0 M HCl solution or 20 mL ammonium hydroxide, extracted with CH_2Cl_2 (30 mL×3). The combined organic layer was washed with H_2O (30 mL×2) and brine (40 mL), dried over Na_2SO_4, concentrated under vacuum. The residue was purified by column chromatography on silica gel to afford **2h** as yellow oil in 70% yield (570 mg).



3aa
4.5 mmol, 1.33 g

4aa
60%, 1.12 g

To a 100 mL oven-dried sealed tube were added CuCl (22.5 mmol, 2.23 g, 5.0 equiv) and KF (15 mmol, 873 mg, 3.3 equiv) in glove box. Then in fume hood, DMF (30 mL), $TMSCF_3$ (15 mmol, 2.2 mL, 3.3 equiv) and pyridine (30 mL) were successively added under N_2 atmosphere. After being stirred at room temperature for 5 minutes, the reaction mixture was immersed into 80 ^oC oil bath and stirred for 10 hours. Then the reaction mixture was cooled to room temperature and bubled with air until the mixture turned black. After that, **3aa** (4.5 mmol, 1.33 g, 1.0 equiv) dissolved in DMF (15 mL) was added slowly to the mixture using a syringe pump for about 15 minutes. The reaction mixture was further stirred under air at room temperature for 2 hours (air was bubbled into the mixture all the time since the mixture was first exposed to air). After the reaction was completed, the reaction mixture was quenched with 200 mL 3.0 M HCl, extracted with CH_2Cl_2 (80 mL×3). The combined organic layer was washed with H_2O (100 mL×2) and brine (100 mL), dried over $MgSO_4$, then concentrated under vacuum. The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate as eluent to afford **4aa** as a yellow solid in 60% yield (1.12 g).



3ab

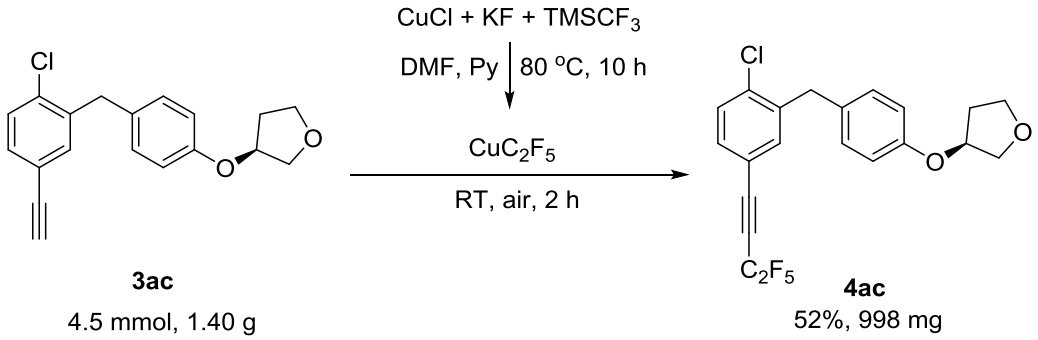
4.5 mmol, 1.34 g

4ab

60%, 1.10 g

To a 100 mL oven-dried sealed tube were added CuCl (22.5 mmol, 2.23 g, 5.0 equiv) and KF (15 mmol, 872 mg, 3.3 equiv) in glove box. Then in fume hood, DMF (30 mL), $TMSCF_3$ (15 mmol, 2.2 mL, 3.3 equiv) and pyridine (30 mL) were successively added under N_2 atmosphere. After being stirred at room temperature for 5 minutes, the reaction mixture was immersed into 80 ^oC oil bath and stirred for 10 hours. Then the reaction mixture was cooled to room temperature and bubled with air until the

mixture turned black. After that, **3ab** (4.5 mmol, 1.34 g, 1.0 equiv) dissolved in DMF (15 mL) was added slowly to the mixture using a syringe pump for about 15 minutes. The reaction mixture was further stirred under air at room temperature for 2 hours (air was bubbled into the mixture all the time since the mixture was first exposed to air).After the reaction was completed, the reaction mixture was quenched with 200 mL 3.0 M HCl, extracted with CH_2Cl_2 (80 mL×3). The combined organic layer was washed with H_2O (100 mL×2) and brine (100 mL), dried over $MgSO_4$, then concentrated under vacuum. The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate as eluent to afford **4ab** as a yellow solid in 60% yield (1.10 g).



3ac
4.5 mmol, 1.40 g

4ac
52%, 998 mg

To a 100 mL oven-dried sealed tube were added CuCl (22.5 mmol, 2.23 g, 5.0 equiv) and KF (15 mmol, 872 mg, 3.3 equiv) in glove box. Then in fume hood, DMF (30 mL), $TMSCF_3$ (15 mmol, 2.2 mL, 3.3 equiv) and pyridine (30 mL) were successively added under N_2 atmosphere. After being stirred at room temperature for 5 minutes, the reaction mixture was immersed into 80 °C oil bath and stirred for 10 hours. Then the reaction mixture was cooled to room temperature and bubled with air until the mixture turned black. After that, **3ac** (4.5 mmol, 1.34 g, 1.0 equiv) dissolved in DMF (15 mL) was added slowly to the mixture using a syringe pump for about 15 minutes. The reaction mixture was further stirred under air at room temperature for 2 h (air was bubbled into the mixture all the time since the mixture was first exposed to air). After the reaction was completed, the reaction mixture was quenched with 200 mL 3.0 M HCl, extracted with CH_2Cl_2 (80 mL×3). The combined organic layer was washed with H_2O (100 mL×2) and brine (100 mL), dried over $MgSO_4$, then concentrated

under vacuum. The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate as eluent to afford **4ac** as a yellow solid in 52% yield (998 mg).

6. References

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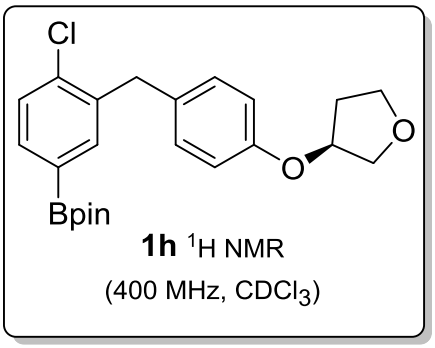
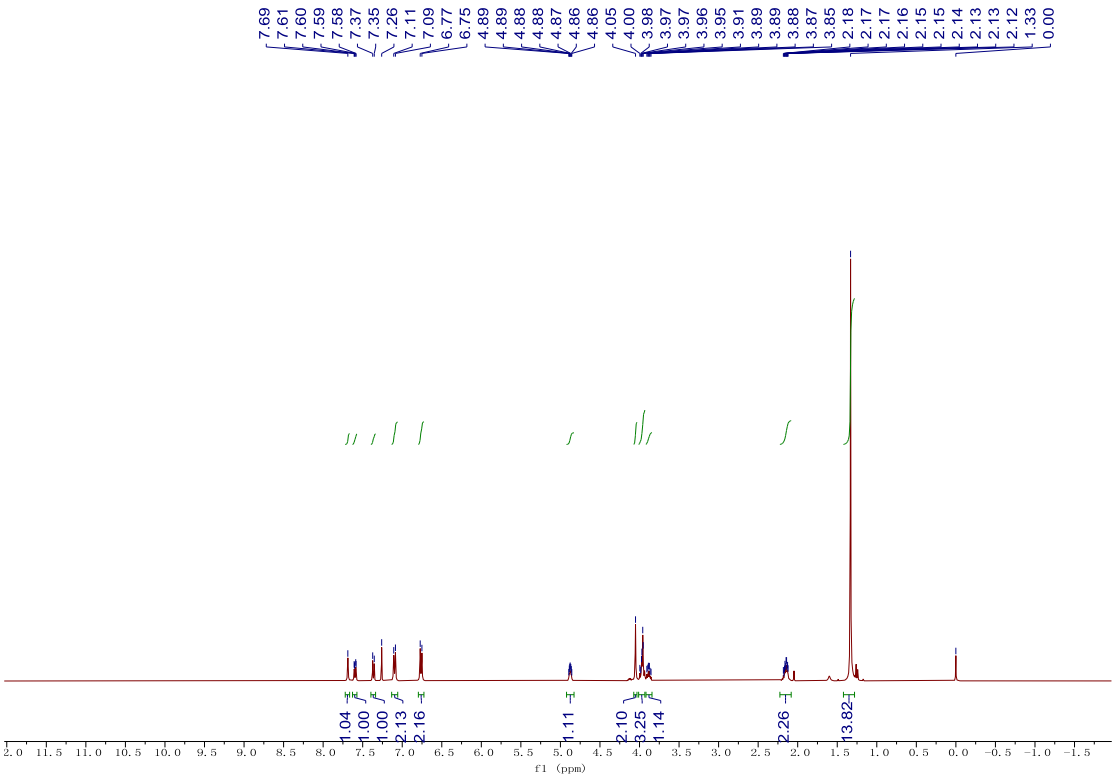
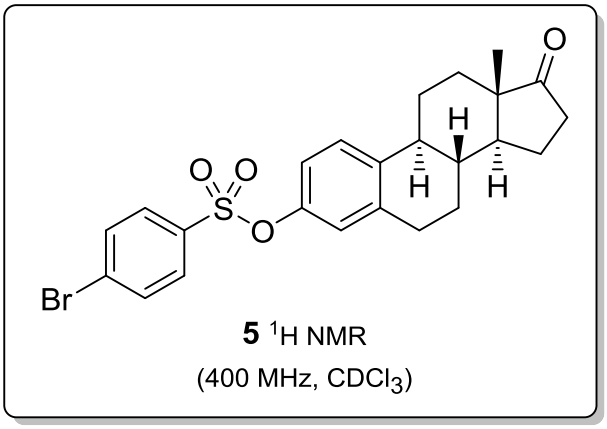
7. ^{1}H, ^{19}F, ^{13}C NMR Spectra of New Compounds

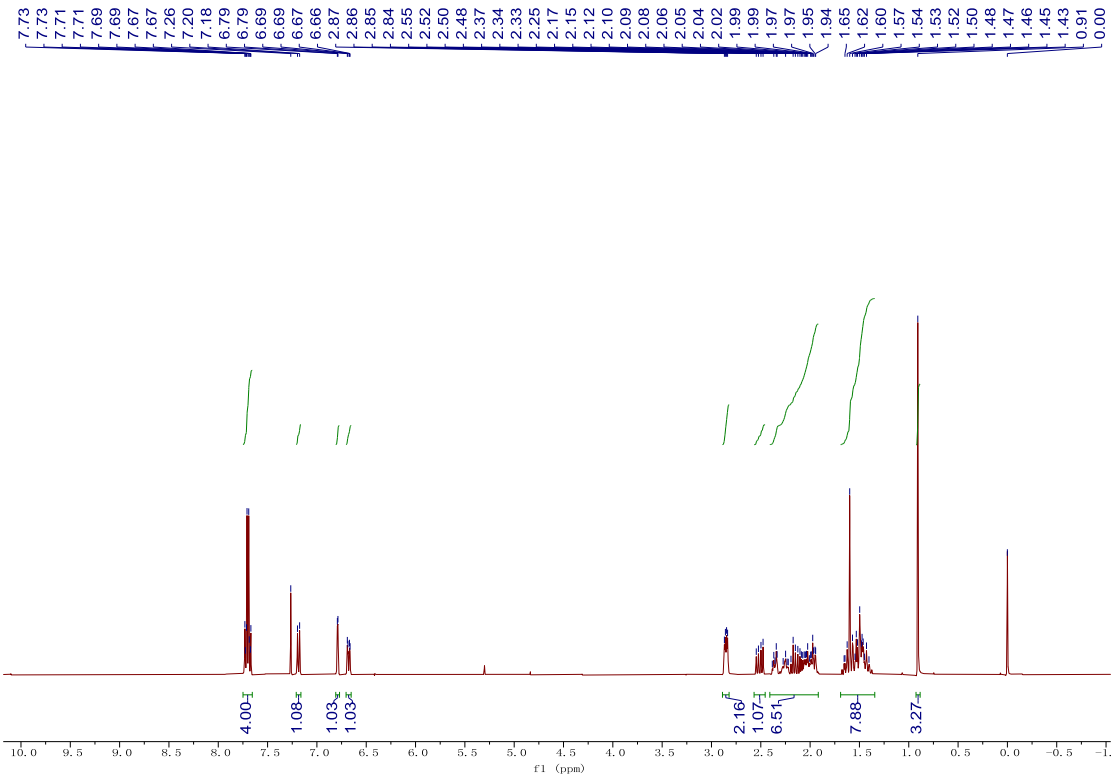


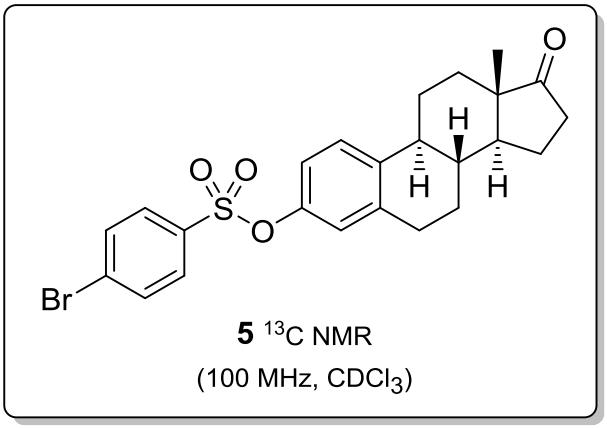




5 ^{1}H NMR

(400 MHz, $CDCl_3$)

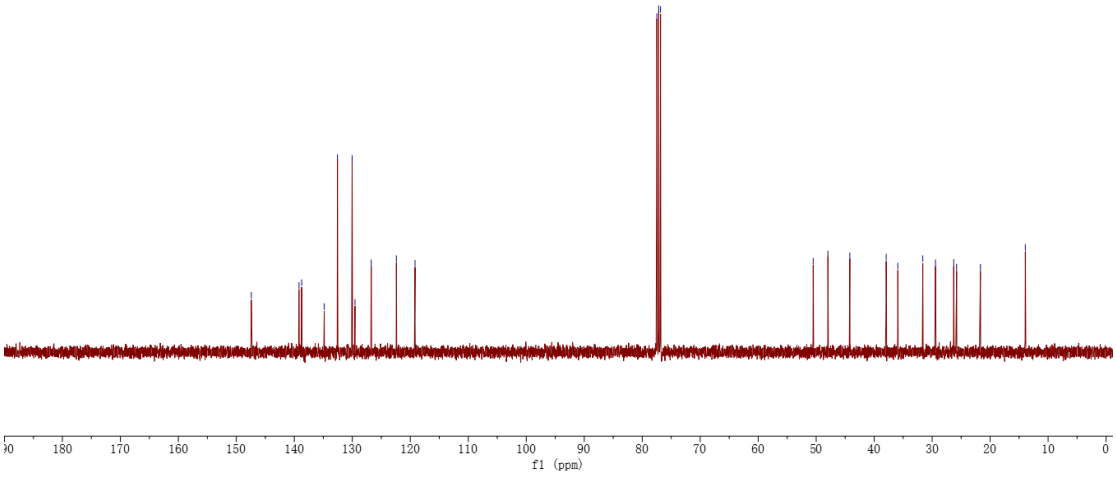


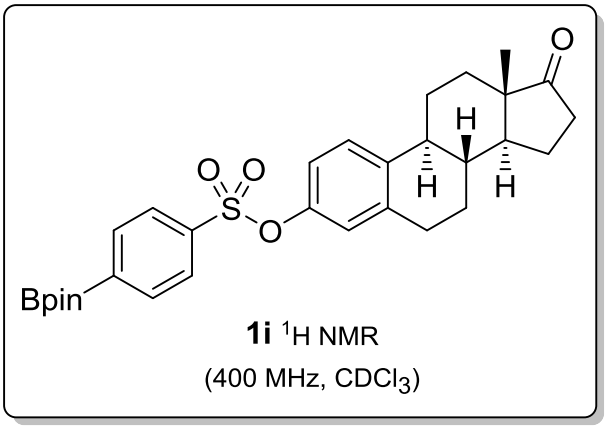


5 ^{13}C NMR

(100 MHz, $CDCl_3$)

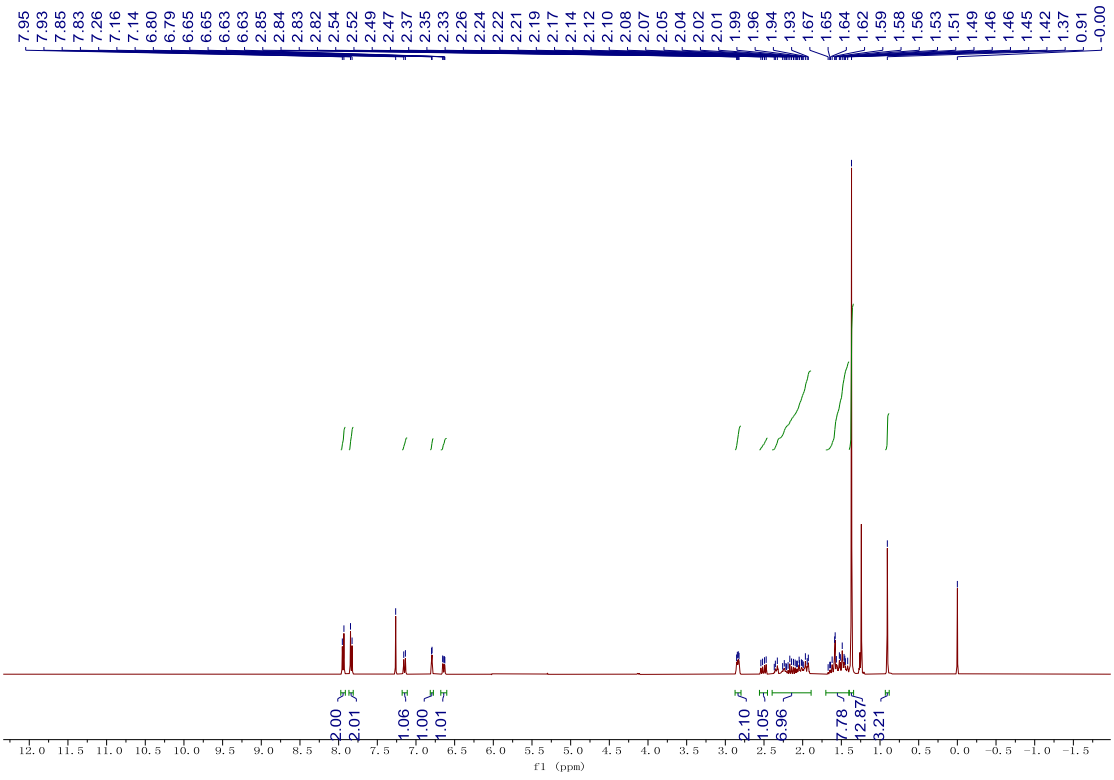
147.42
139.19
138.74
134.84
132.56
130.03
129.54
126.74
122.40
119.18
77.48
77.16
76.84
50.52
47.98
44.20
37.93
35.92
31.61
29.42
26.28
25.78
21.67
13.93

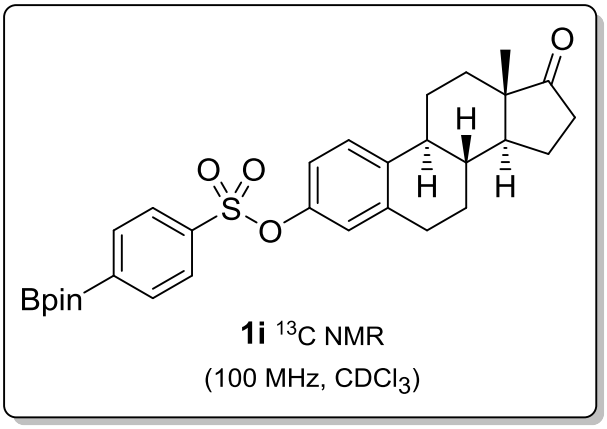




1i ^{1}H NMR

(400 MHz, $CDCl_3$)

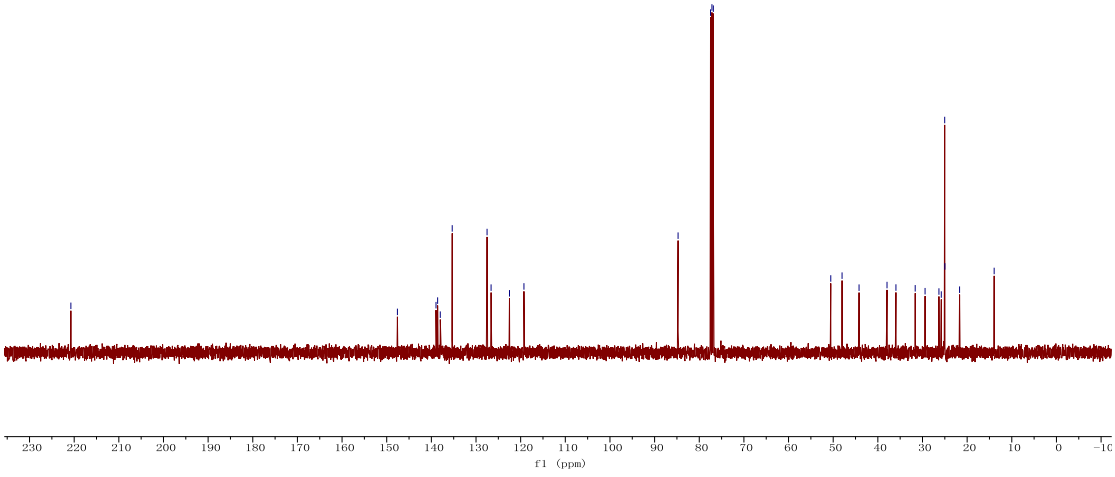


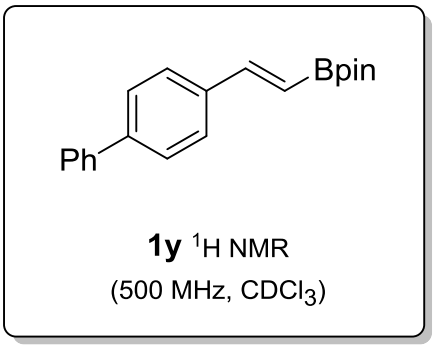


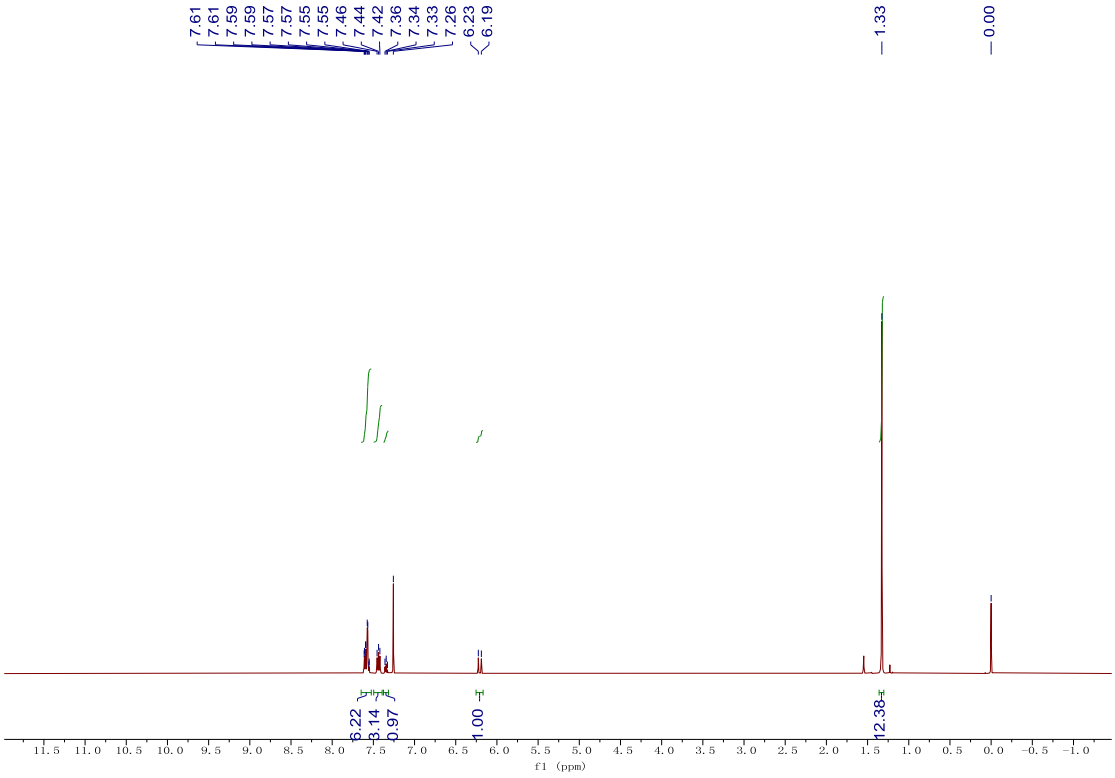
1i ^{13}C NMR

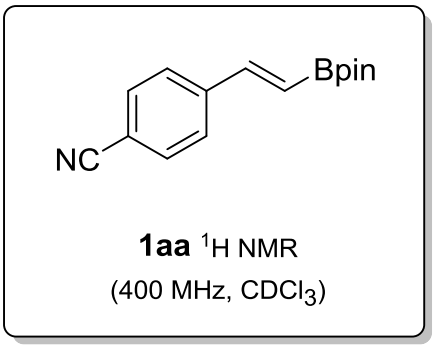
(100 MHz, $CDCl_3$)

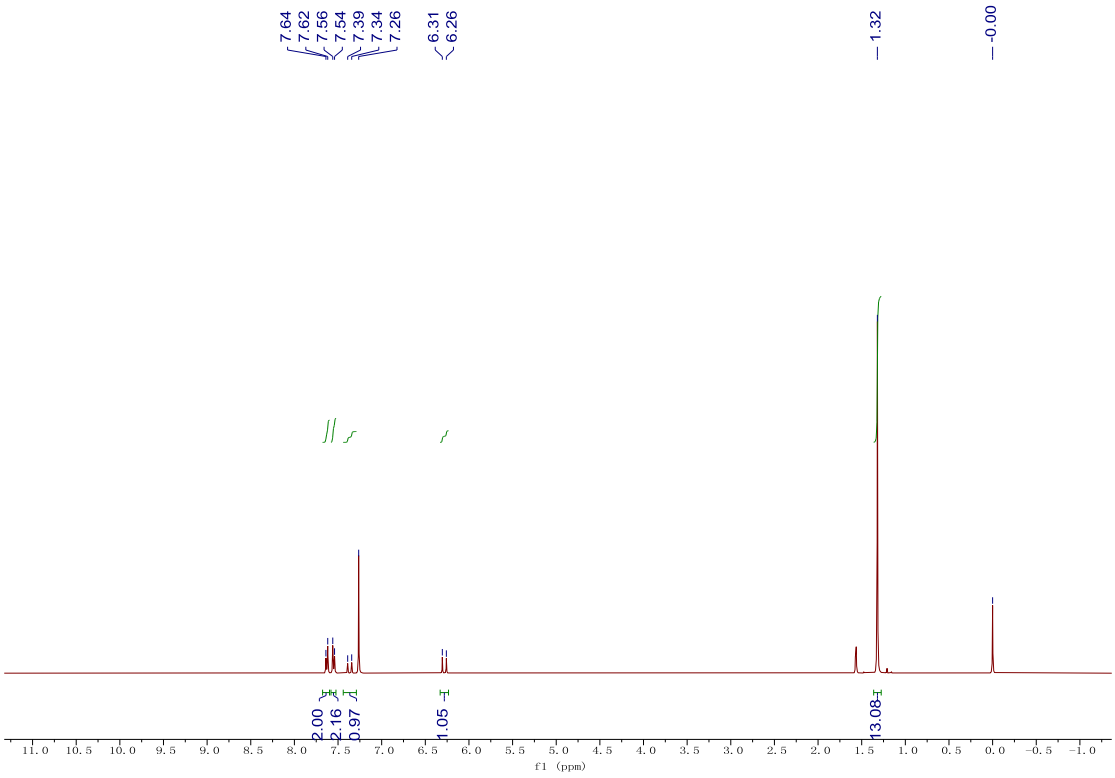
220.74
147.61
138.96
138.60
138.01
135.33
127.52
126.63
122.50
119.26
84.74
77.48
77.16
76.84
50.54
48.02
44.21
37.95
35.95
31.63
29.42
26.32
25.79
25.03
24.99
21.70
13.95

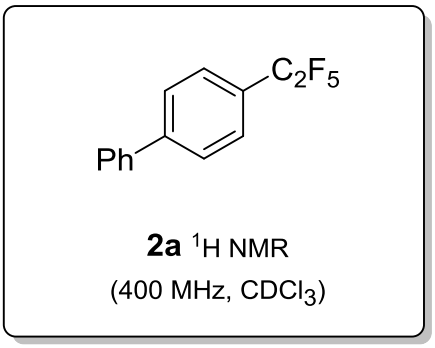



Bpin
Ph
1y 1H NMR
(500 MHz, CDCl3)

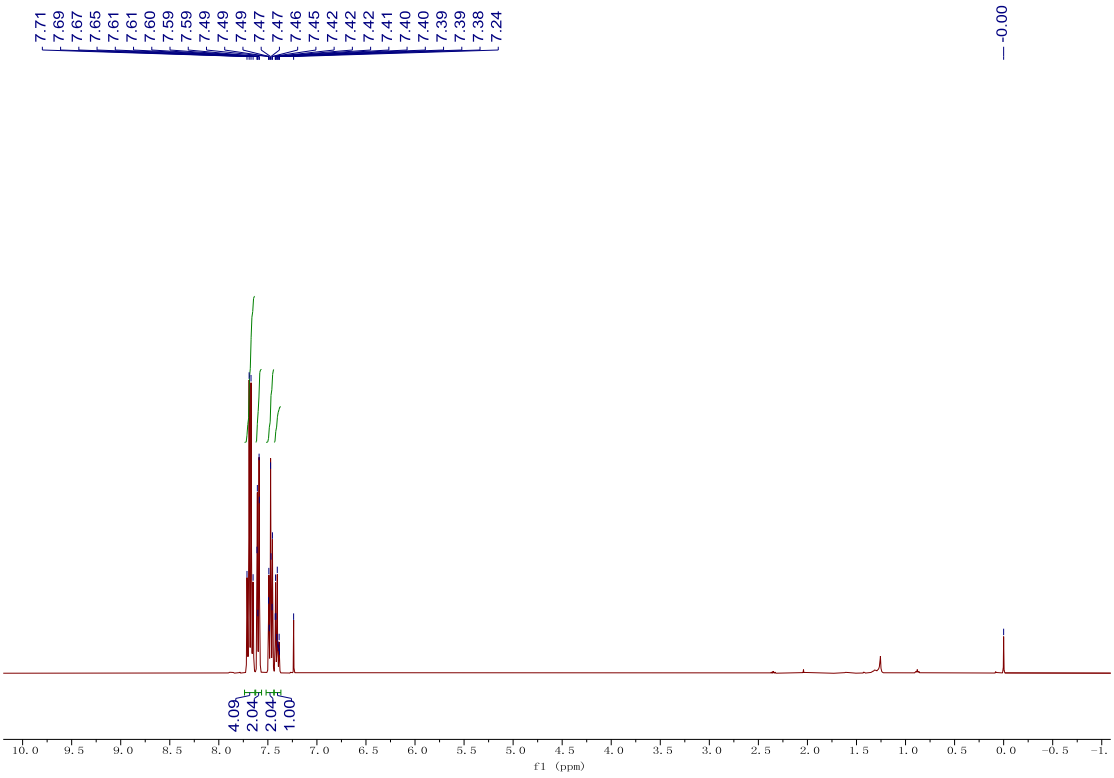

7.61
7.61
7.59
7.59
7.57
7.57
7.55
7.55
7.46
7.44
7.42
7.36
7.34
7.33
7.26
6.23
6.19
1.33
0.00
6.22
3.14
0.97
1.00
12.38
f1 (ppm)

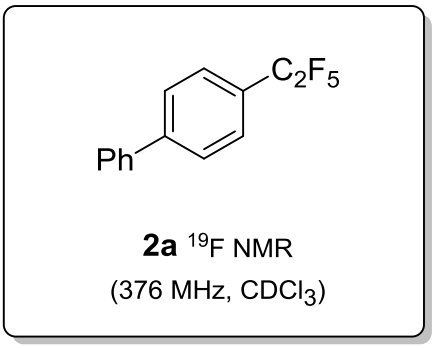

Bpin
NC
1aa 1H NMR
(400 MHz, CDCl3)


7.64
7.62
7.56
7.54
7.39
7.34
7.26
6.31
6.26
1.32
0.00
2.00
2.16
0.97
1.05
13.08
f1 (ppm)



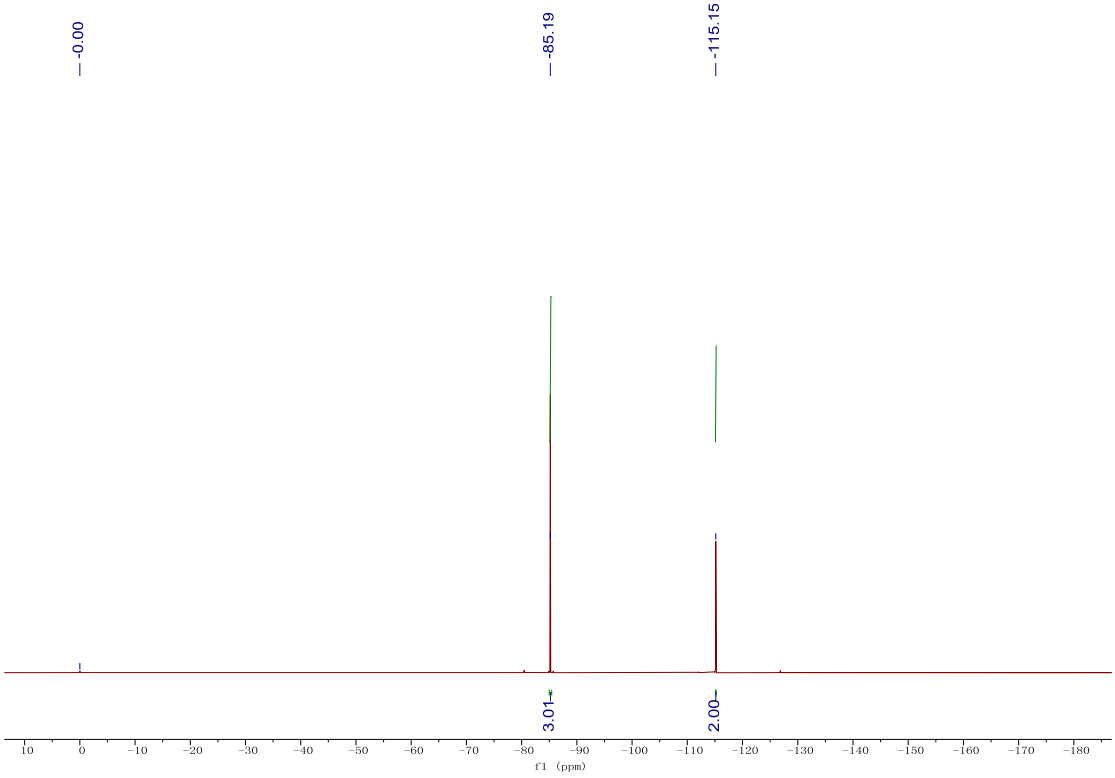
2a ^{1}H NMR
(400 MHz, $CDCl_3$)

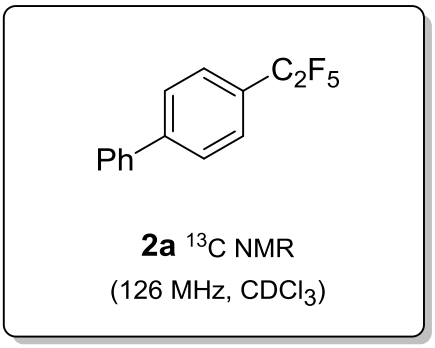




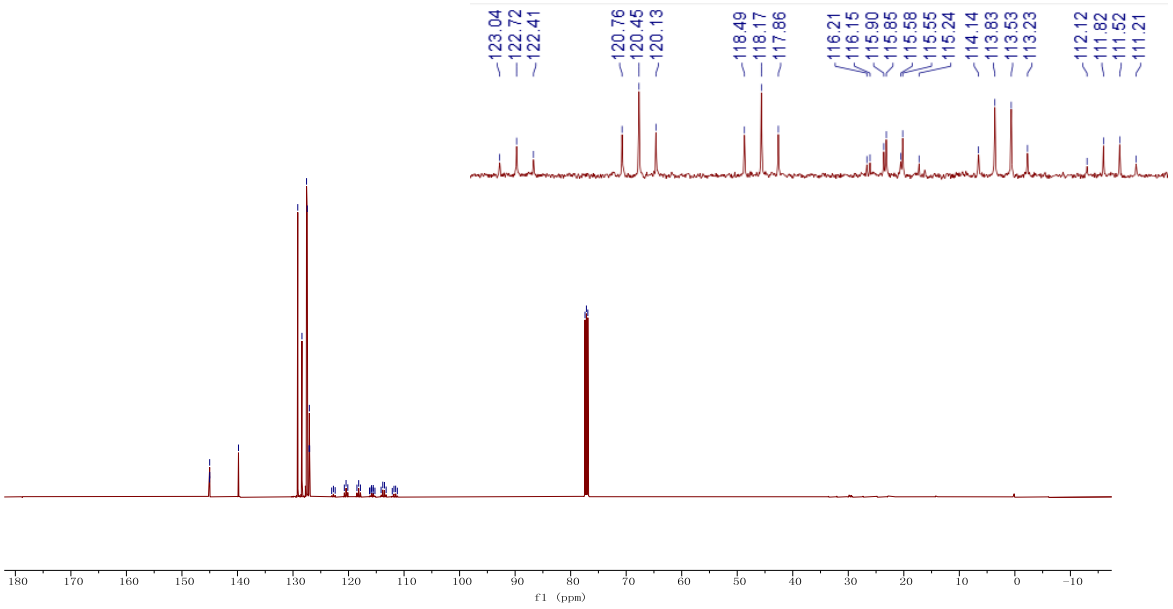
2a ^{19}F NMR

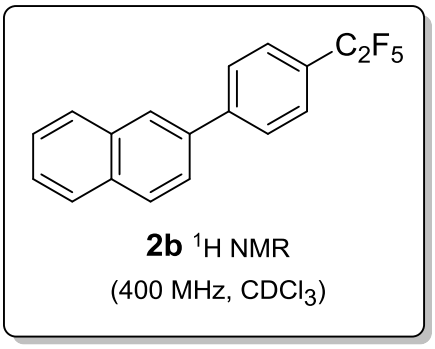
(376 MHz, $CDCl_3$)



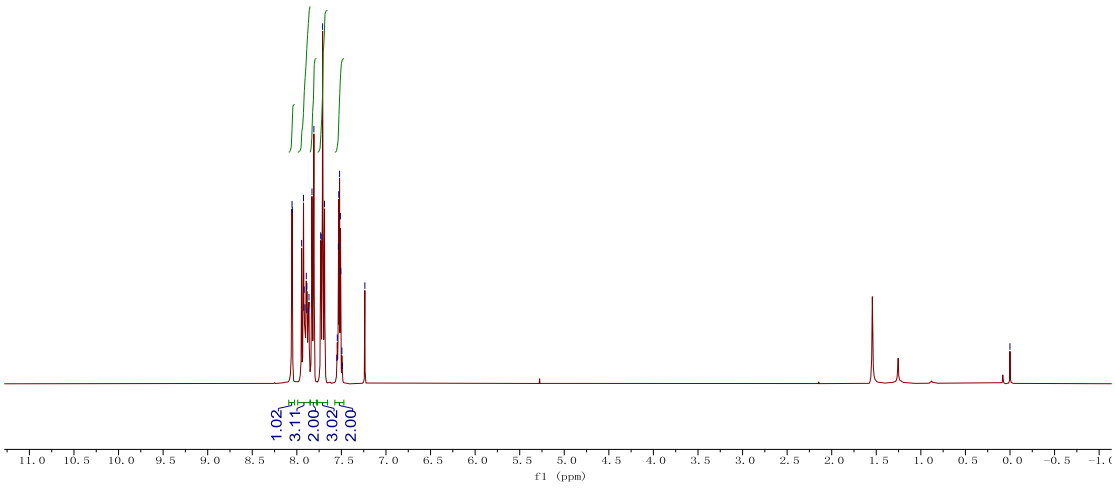

C2F5
Ph
2a 13C NMR
(126 MHz, CDCl3)

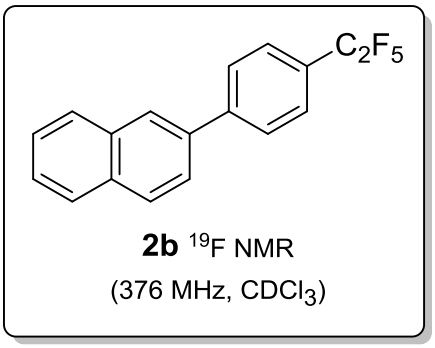

145.03
145.01
145.00
139.82
129.14
128.39
127.56
127.44
127.11
127.06
127.01
123.04
122.72
122.41
120.76
120.45
120.13
118.49
118.17
117.86
116.21
116.15
115.90
115.85
115.58
115.55
115.24
114.14
113.83
113.53
113.23
112.12
111.82
111.52
111.21
77.41
77.16
76.90


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


C_2F_5
2b ^{1}H NMR
(400 MHz, $CDCl_3$)

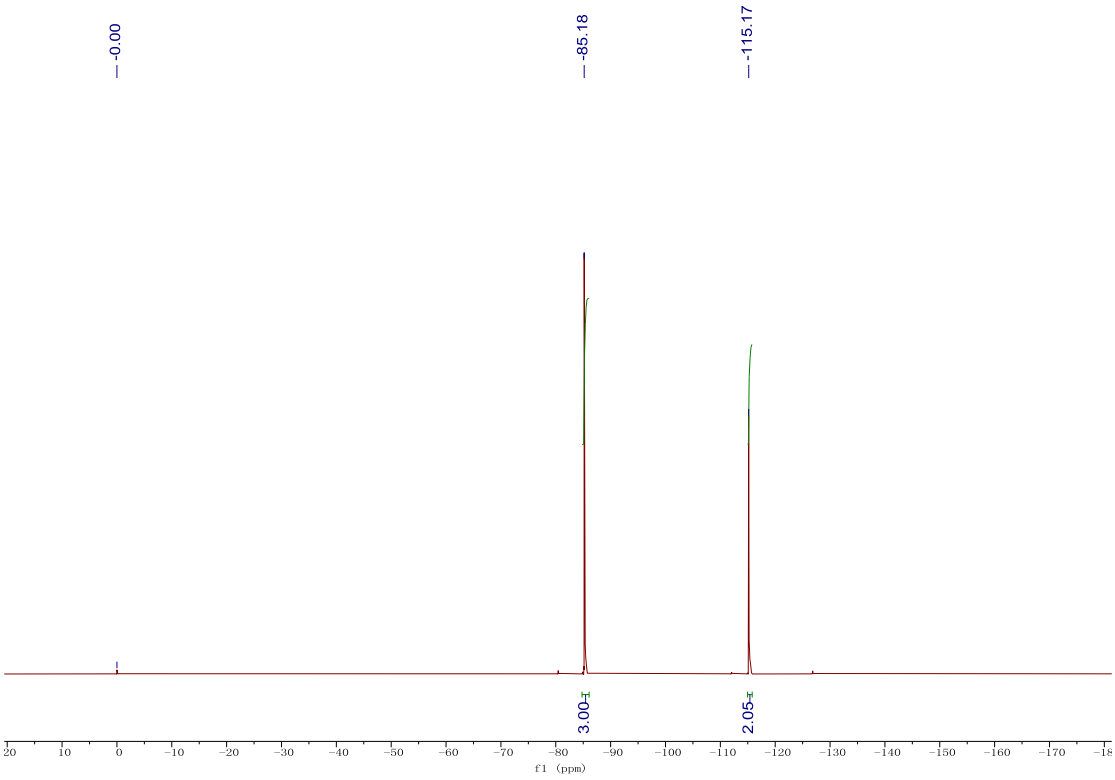

8.05
8.05
7.95
7.92
7.92
7.91
7.89
7.89
7.87
7.86
7.83
7.81
7.73
7.73
7.71
7.69
7.55
7.55
7.53
7.53
7.52
7.51
7.51
7.49
7.49
7.24
0.00

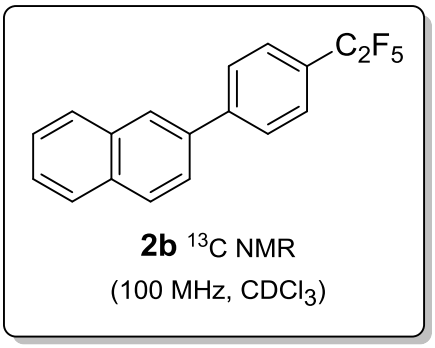

1.02
3.11
2.00
3.02
2.00
f1 (ppm)

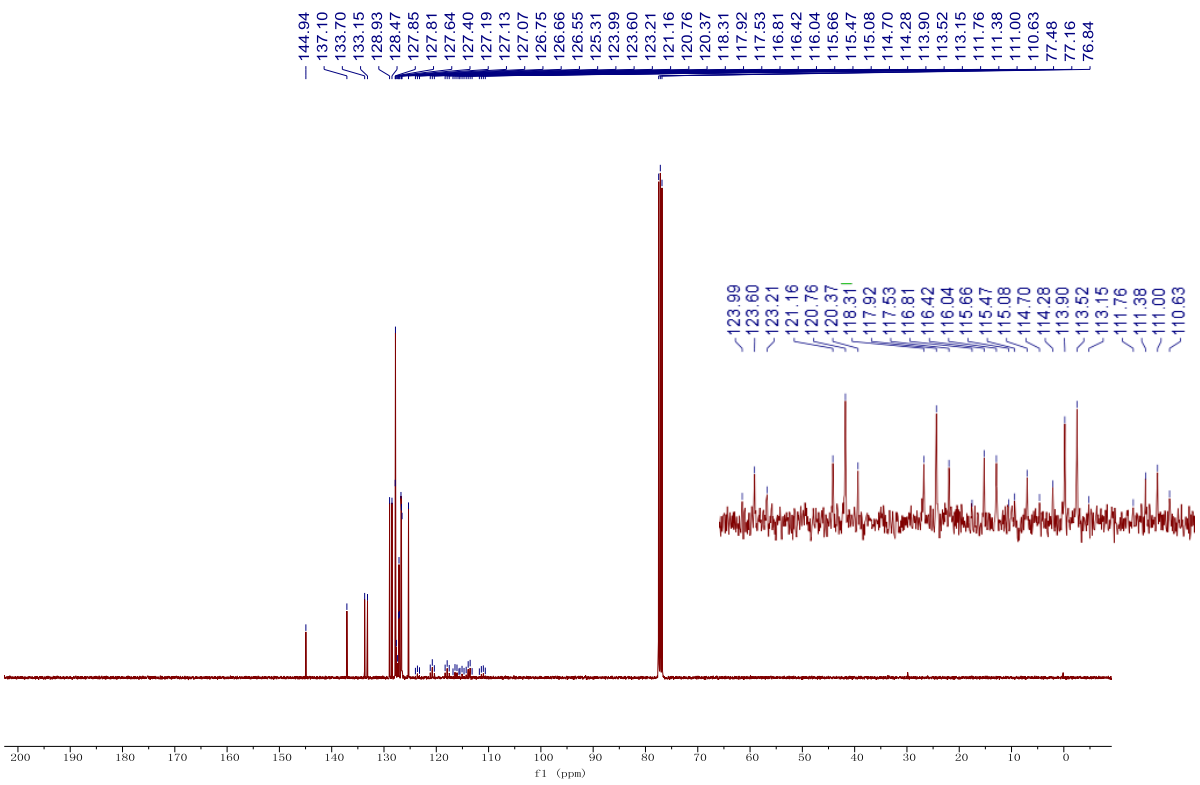


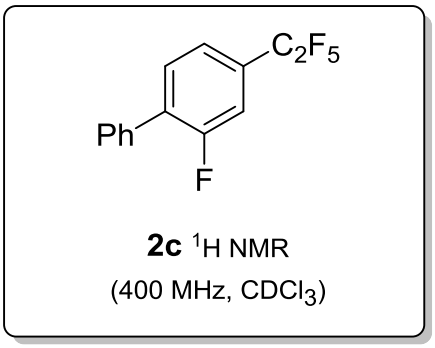
2b ^{19}F NMR

(376 MHz, $CDCl_3$)

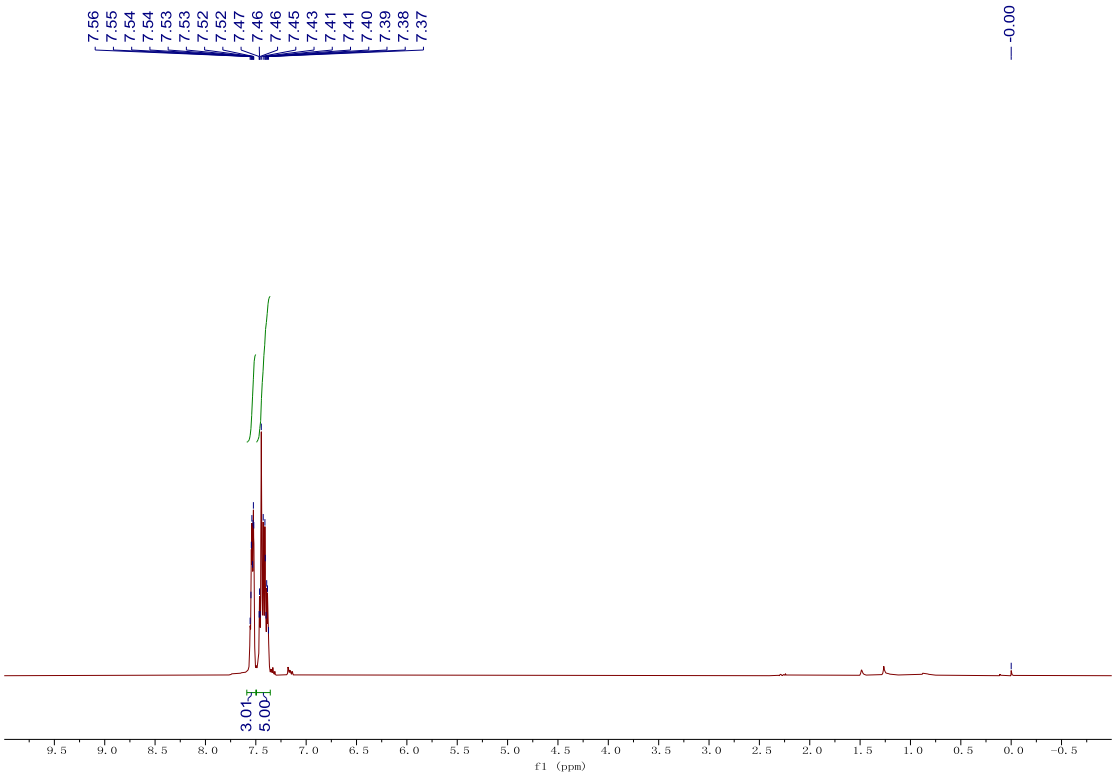


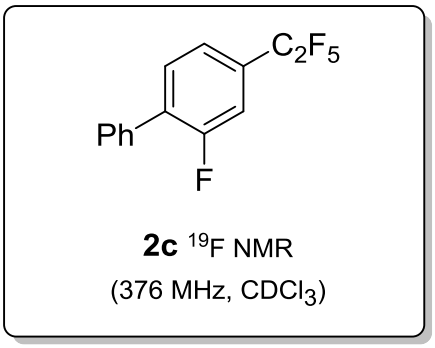

C_2F_5
2b ^{13}C NMR
(100 MHz, $CDCl_3$)


144.94
137.10
133.70
133.15
128.93
128.47
127.85
127.81
127.64
127.40
127.19
127.13
127.07
126.75
126.66
126.55
125.31
123.99
123.60
123.21
121.16
120.76
120.37
118.31
117.92
117.53
116.81
116.42
116.04
115.66
115.47
115.08
114.70
114.28
113.90
113.52
113.15
111.76
111.38
111.00
110.63
77.48
77.16
76.84
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



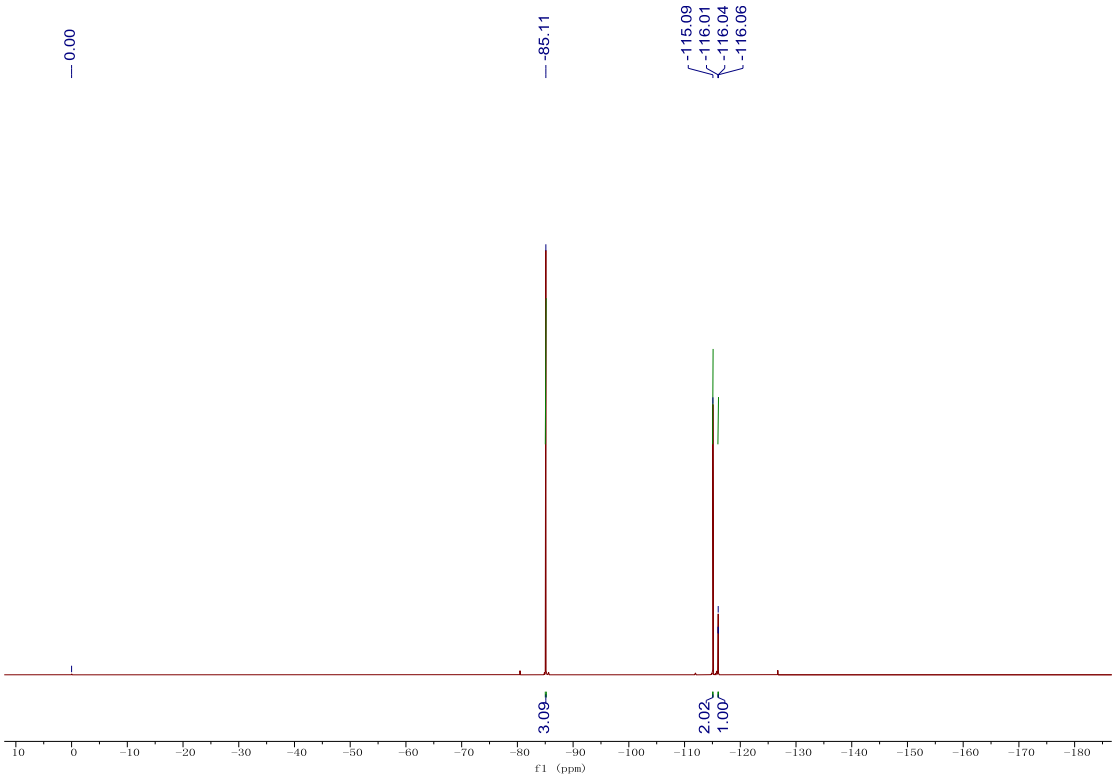
2c ^{1}H NMR
(400 MHz, $CDCl_3$)

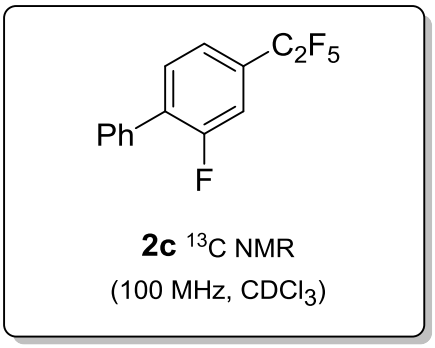




2c ^{19}F NMR

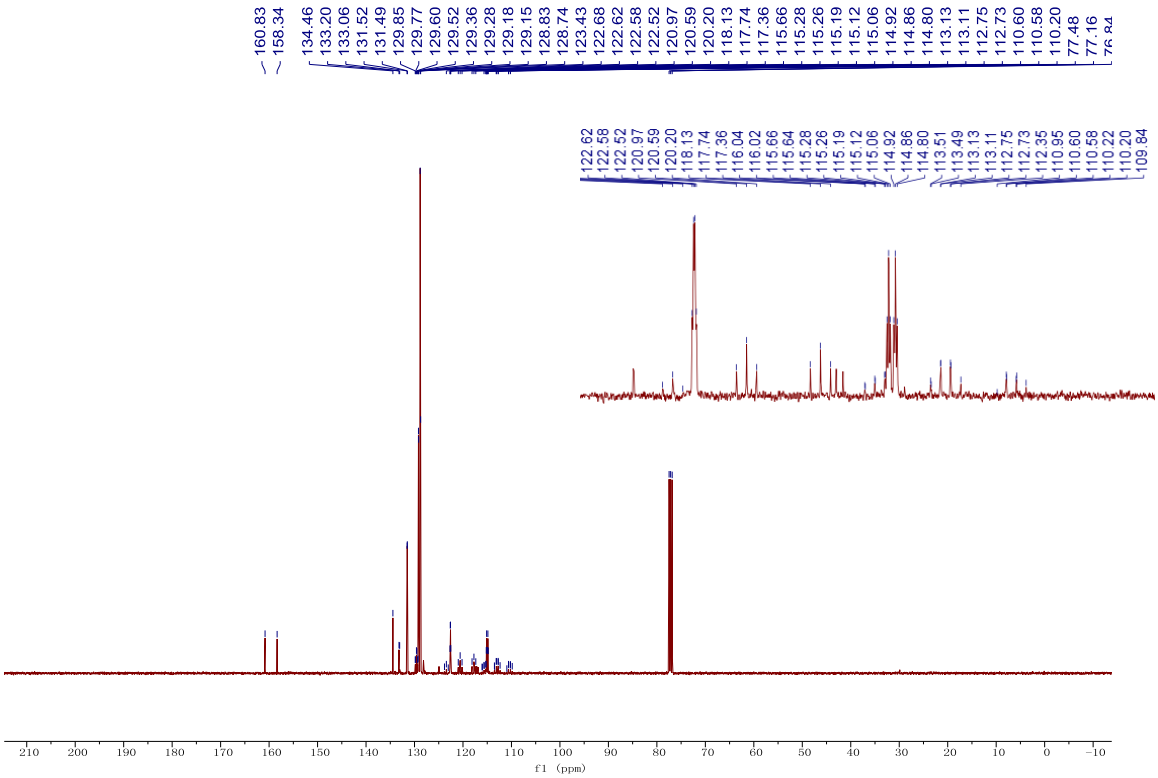
(376 MHz, $CDCl_3$)

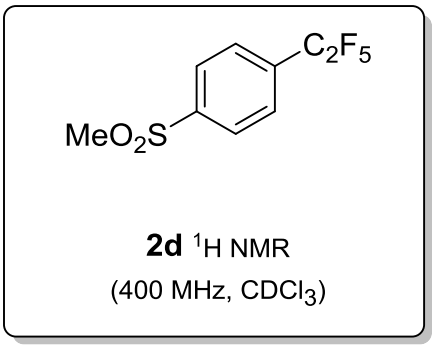


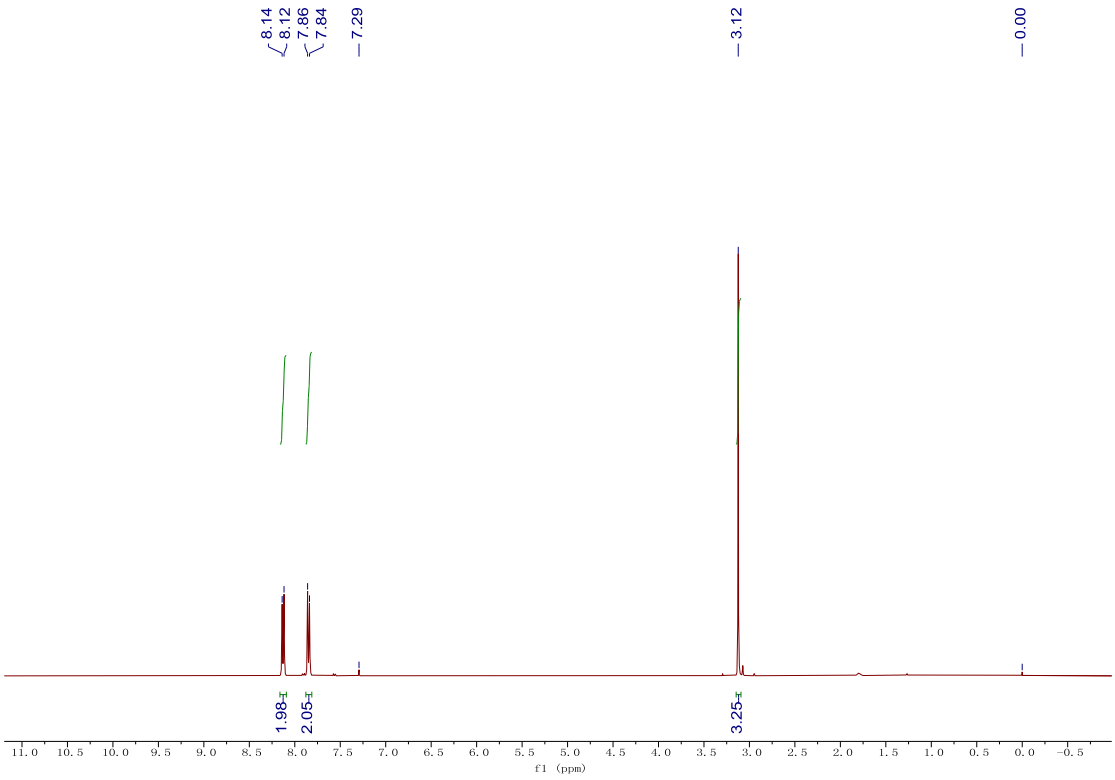


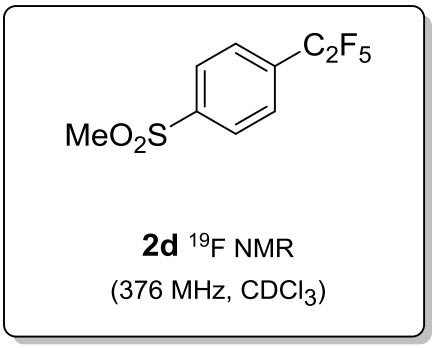
2c ^{13}C NMR

(100 MHz, $CDCl_3$)



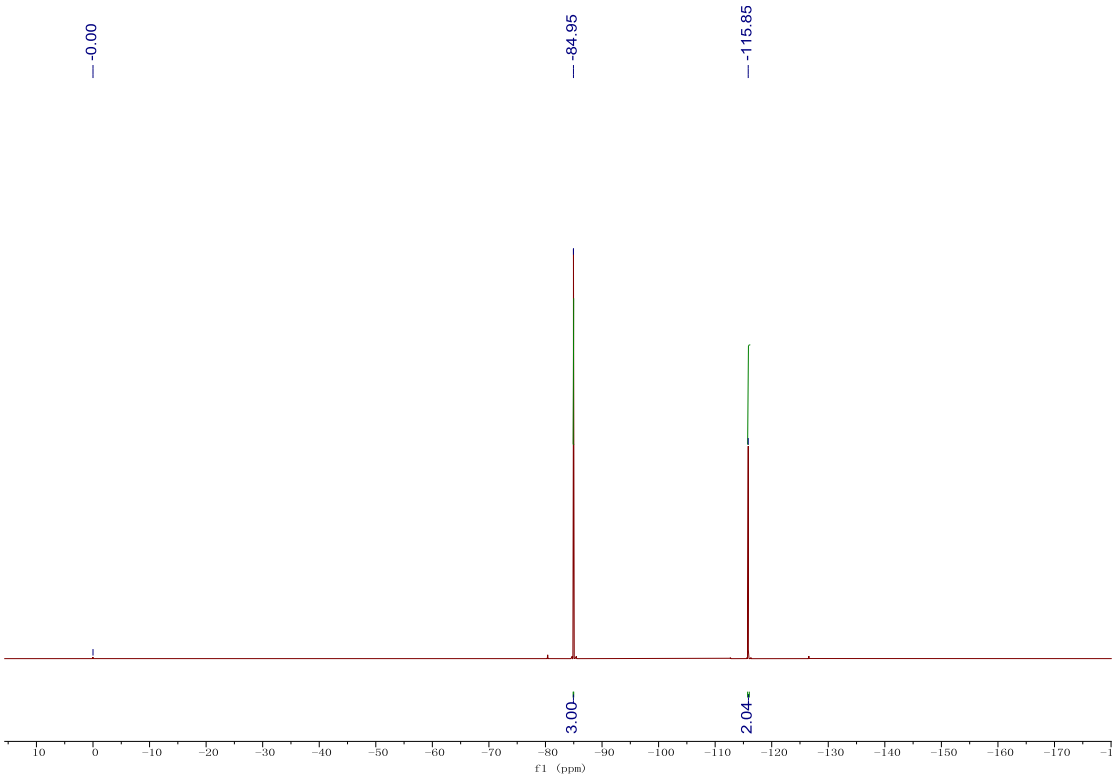

C2F5
MeO2S
2d 1H NMR
(400 MHz, CDCl3)

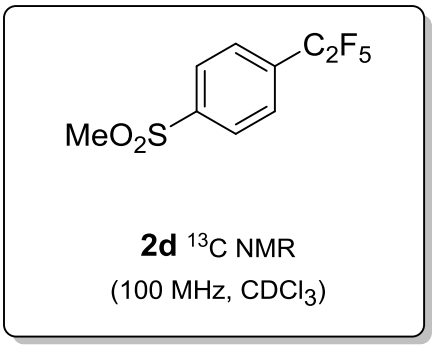

8.14
8.12
7.86
7.84
7.29
3.12
0.00
1.98
2.05
3.25
f1 (ppm)

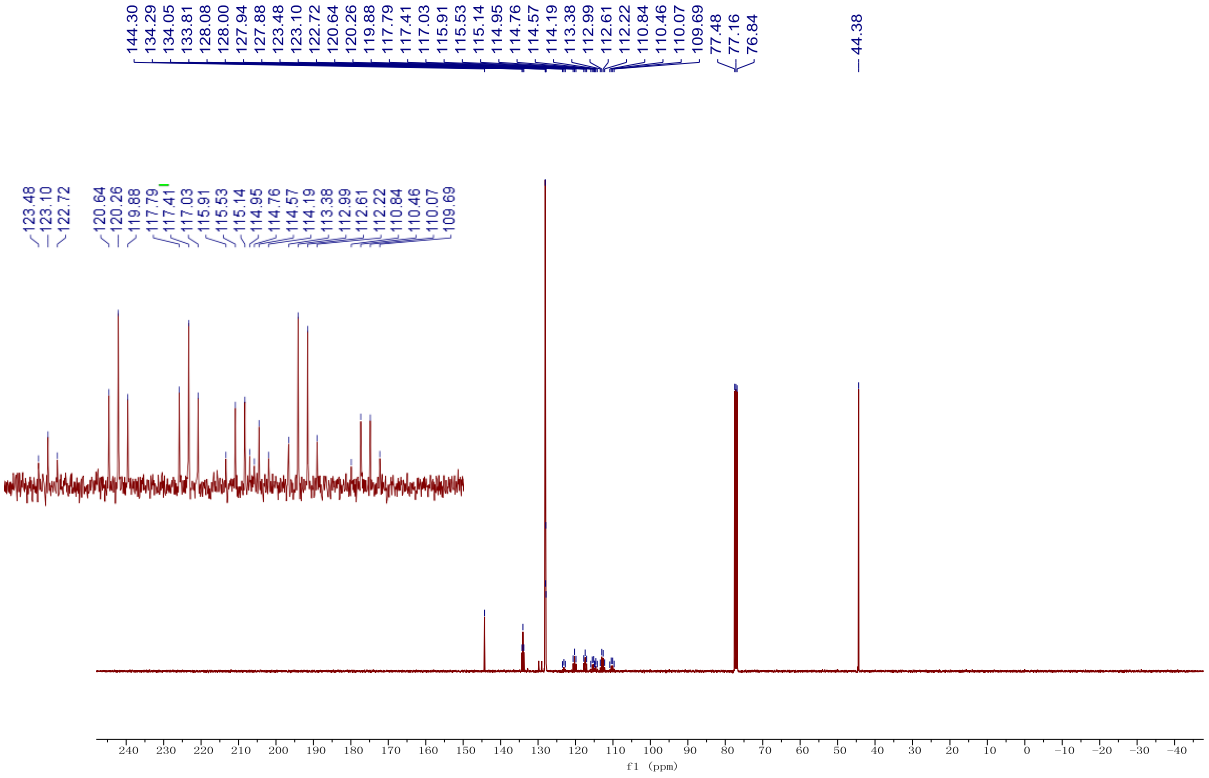


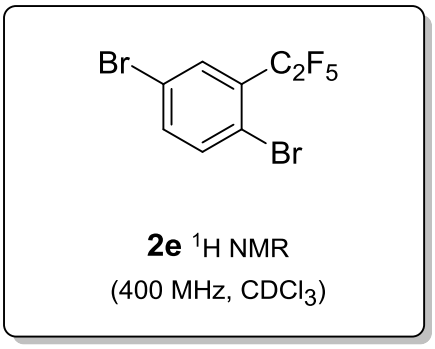
2d ^{19}F NMR

(376 MHz, $CDCl_3$)

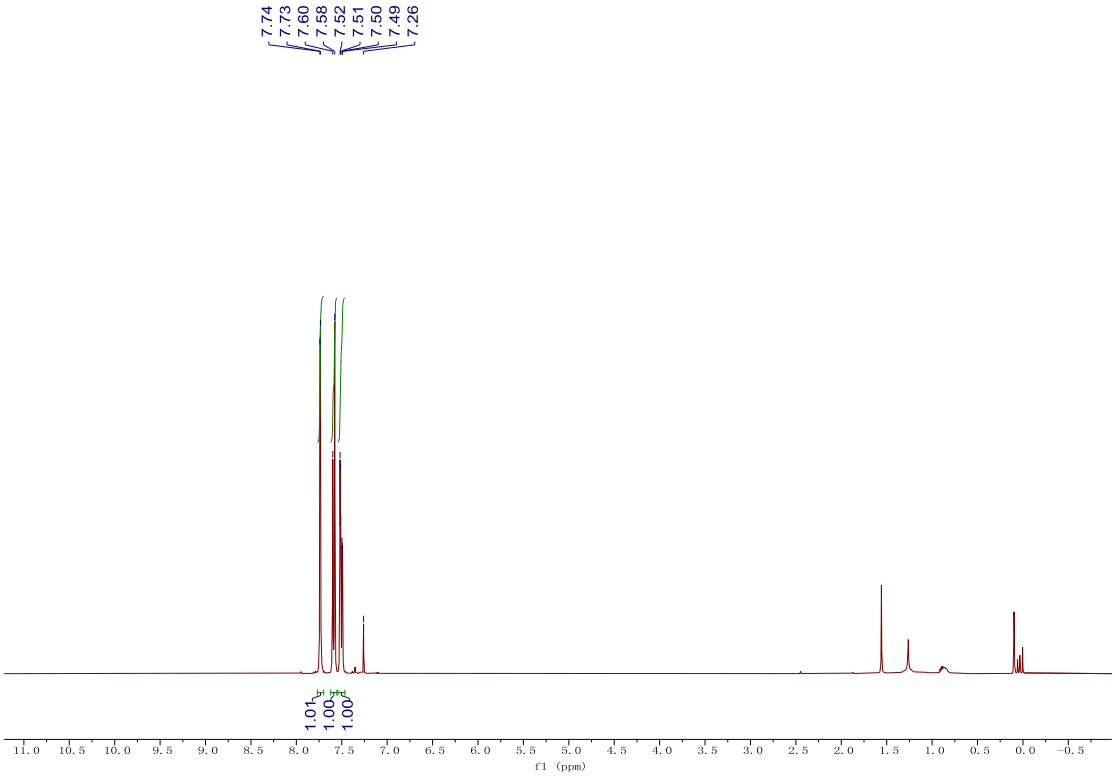


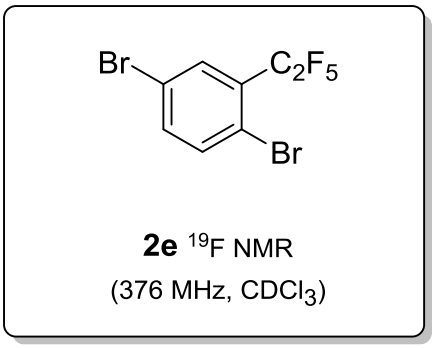

C_2F_5
MeO_2S
2d ^{13}C NMR
(100 MHz, $CDCl_3$)


144.30
134.29
134.05
133.81
128.08
128.00
127.94
127.88
123.48
123.10
122.72
120.64
120.26
119.88
117.79
117.41
117.03
115.91
115.53
115.14
114.95
114.76
114.57
114.19
113.38
112.99
112.61
112.22
110.84
110.46
110.07
109.69
77.48
77.16
76.84
44.38
f1 (ppm)



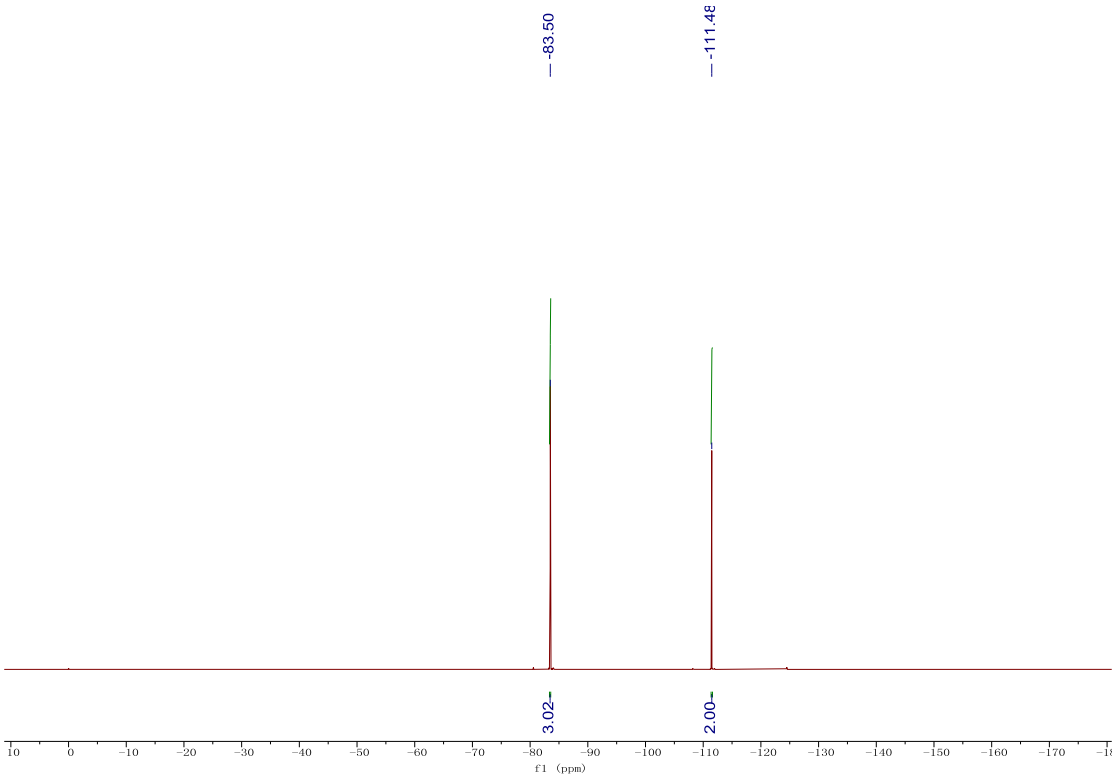
2e ^{1}H NMR
(400 MHz, $CDCl_3$)

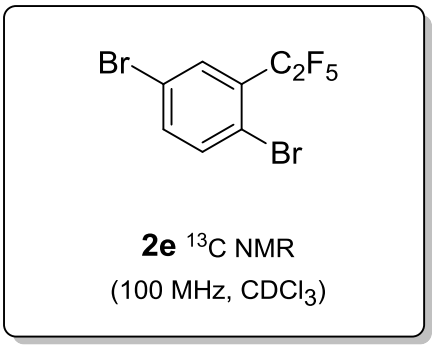




2e ^{19}F NMR

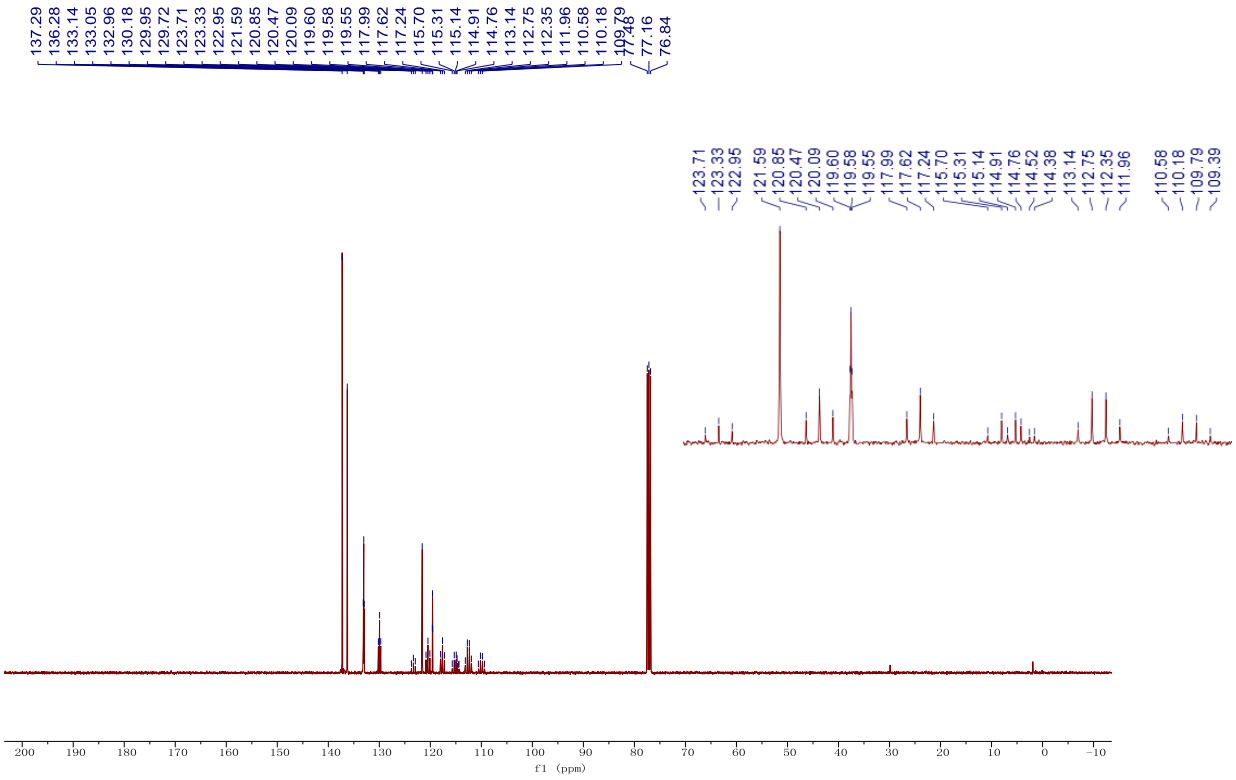
(376 MHz, $CDCl_3$)

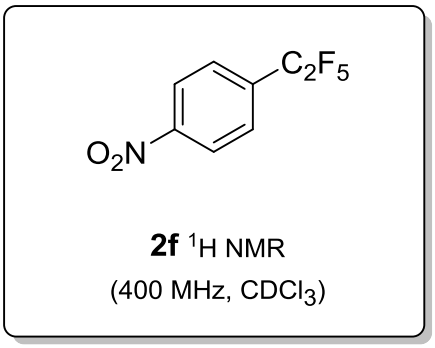


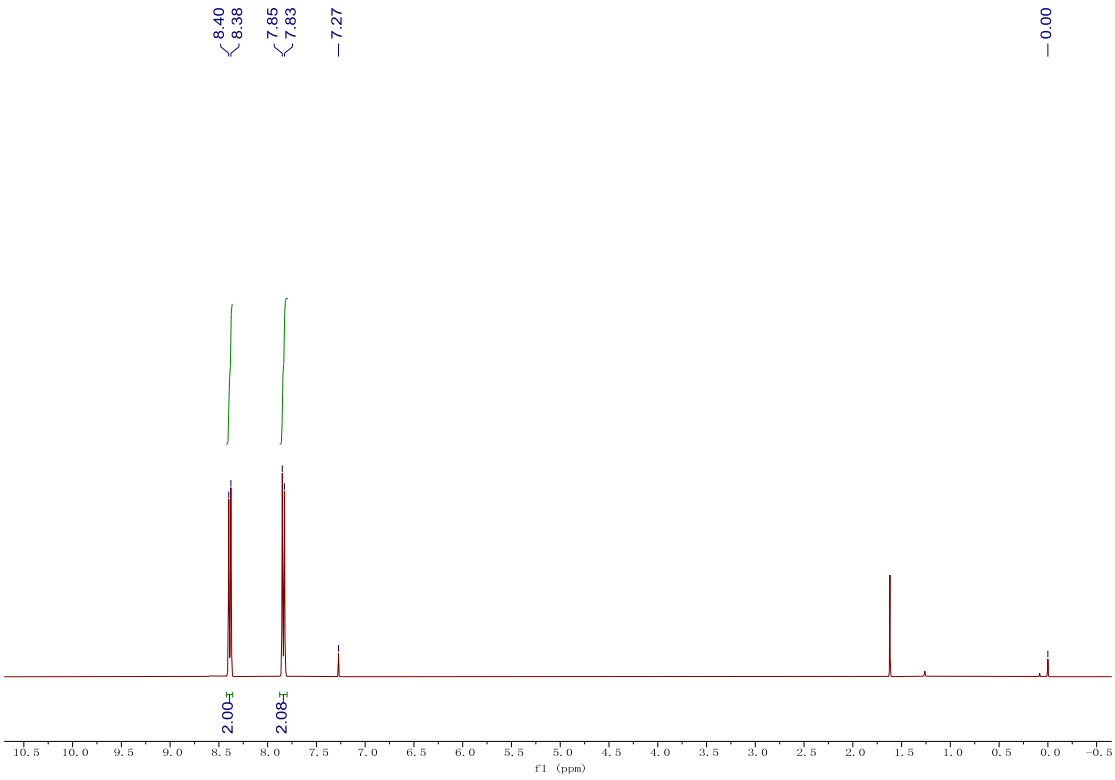


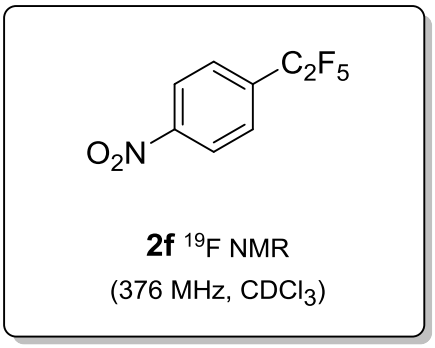
2e ^{13}C NMR

(100 MHz, $CDCl_3$)



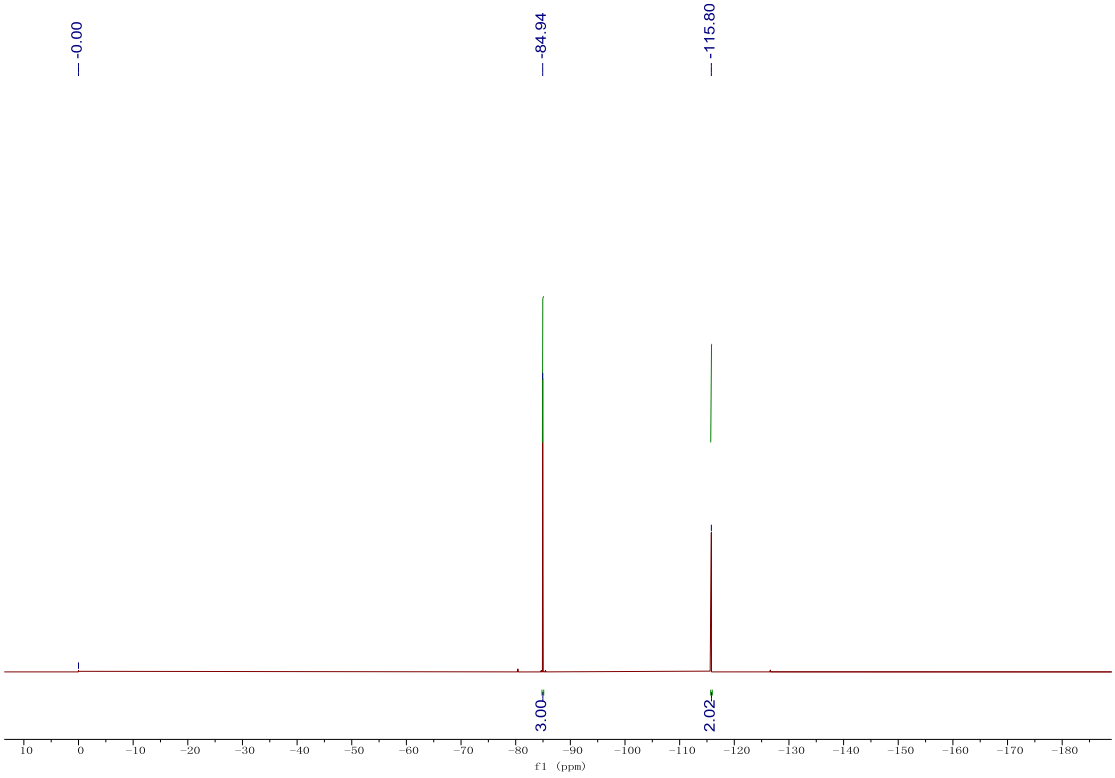

C_2F_5
O_2N
2f ^{1}H NMR
(400 MHz, $CDCl_3$)

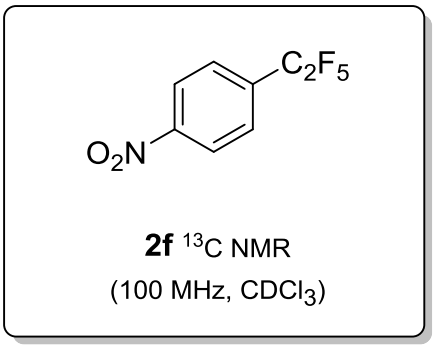

8.40
8.38
7.85
7.83
7.27
0.00
2.00
2.08
f1 (ppm)



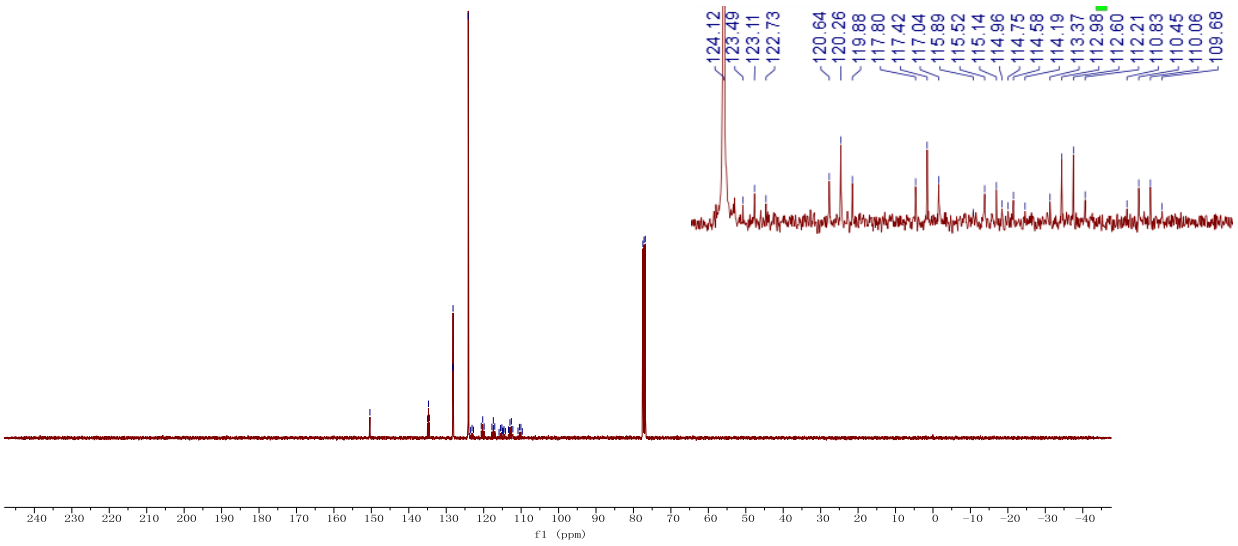
2f ^{19}F NMR

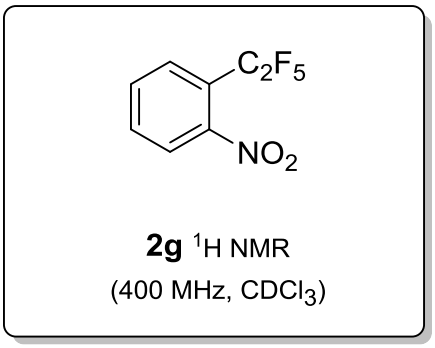
(376 MHz, $CDCl_3$)



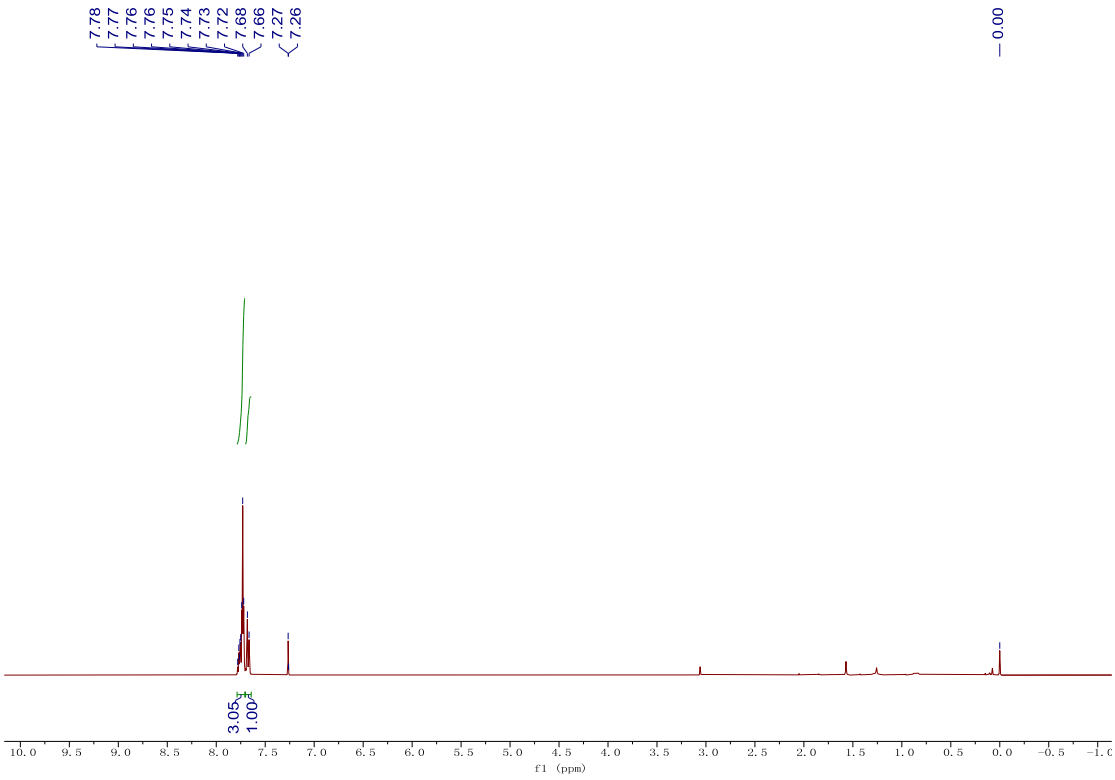

C_2F_5
O_2N
2f ^{13}C NMR
(100 MHz, $CDCl_3$)

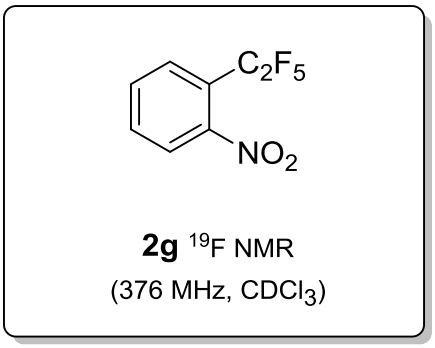

150.38
134.98
134.74
134.50
128.25
128.18
128.12
124.12
123.49
123.11
122.73
120.64
120.26
119.88
117.80
117.42
117.04
115.89
115.52
115.14
114.96
114.75
114.58
114.19
113.37
112.98
112.60
112.21
110.83
110.45
110.06
109.68
77.48
77.16
76.84


124.12
123.49
123.11
122.73
120.64
120.26
119.88
117.80
117.42
117.04
115.89
115.52
115.14
114.96
114.75
114.58
114.19
113.37
112.98
112.60
112.21
110.83
110.45
110.06
109.68
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40
f1 (ppm)

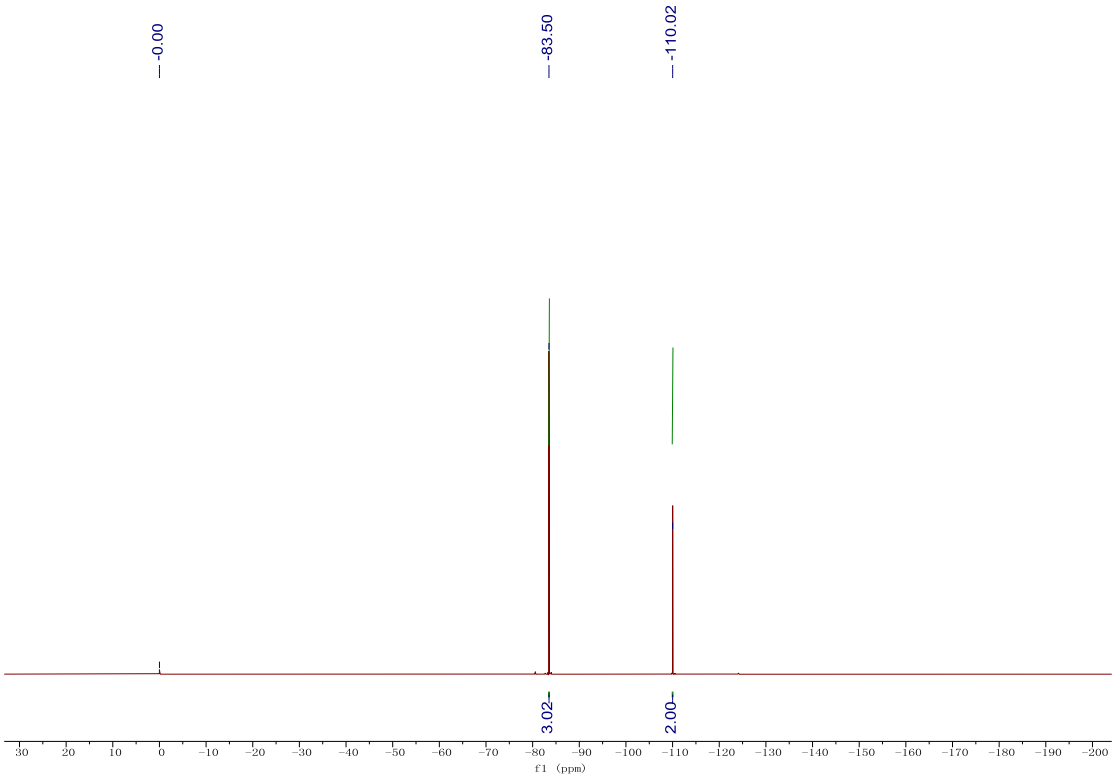


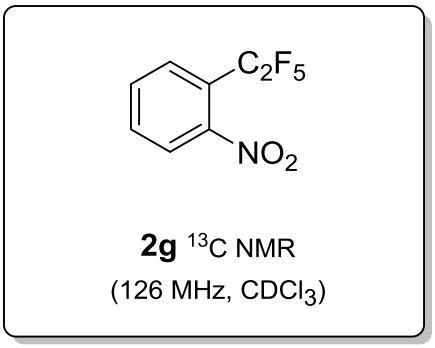
2g ^{1}H NMR
(400 MHz, $CDCl_3$)

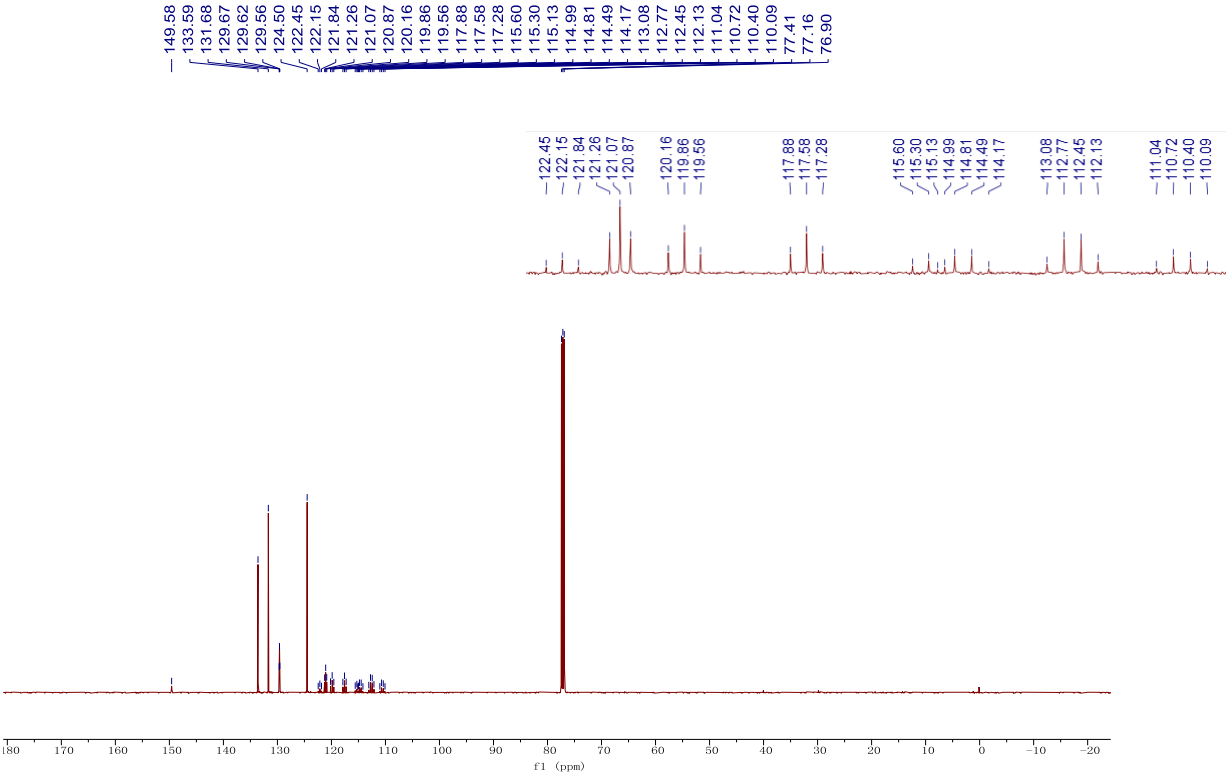




2g ^{19}F NMR
(376 MHz, $CDCl_3$)




C_2F_5
NO_2
2g ^{13}C NMR
(126 MHz, $CDCl_3$)


149.58
133.59
131.68
129.67
129.62
129.56
124.50
122.45
122.15
121.84
121.26
121.07
120.87
120.16
119.86
119.56
117.88
117.58
117.28
115.60
115.30
115.13
114.99
114.81
114.49
114.17
113.08
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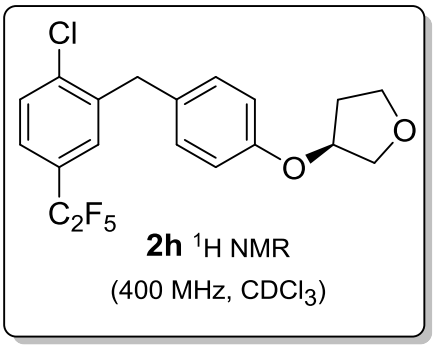
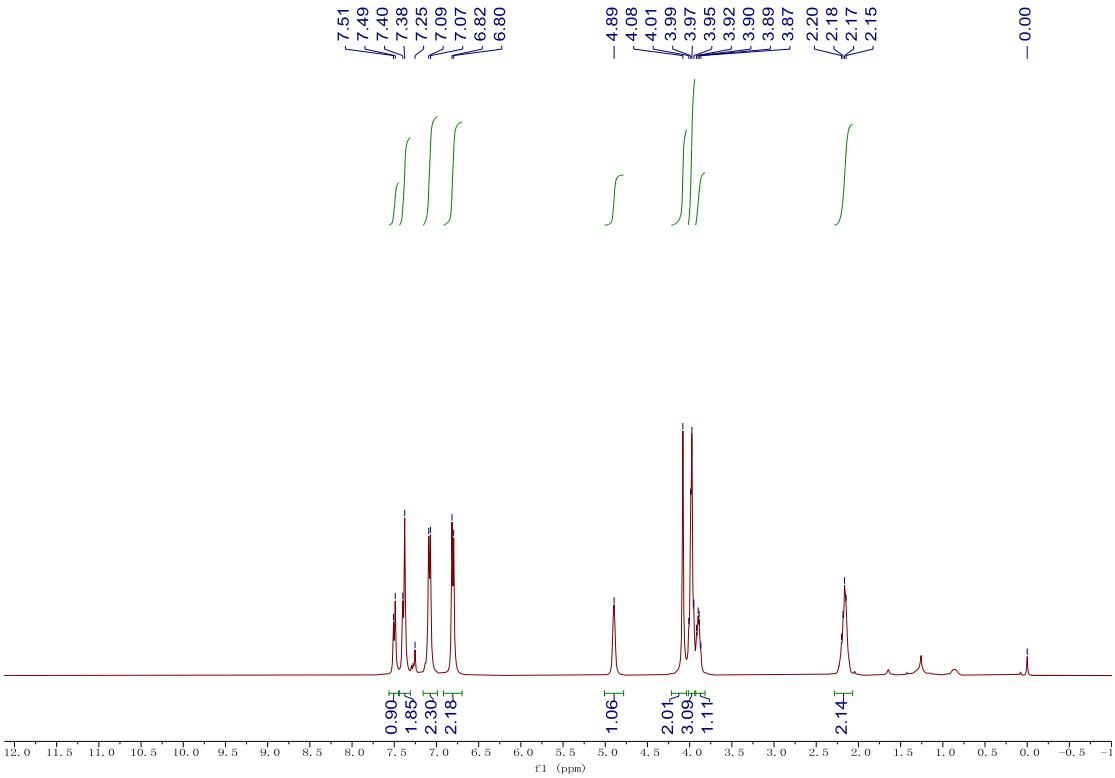
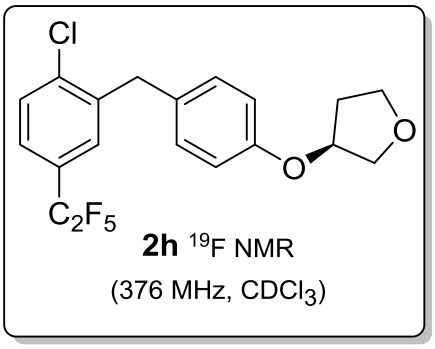
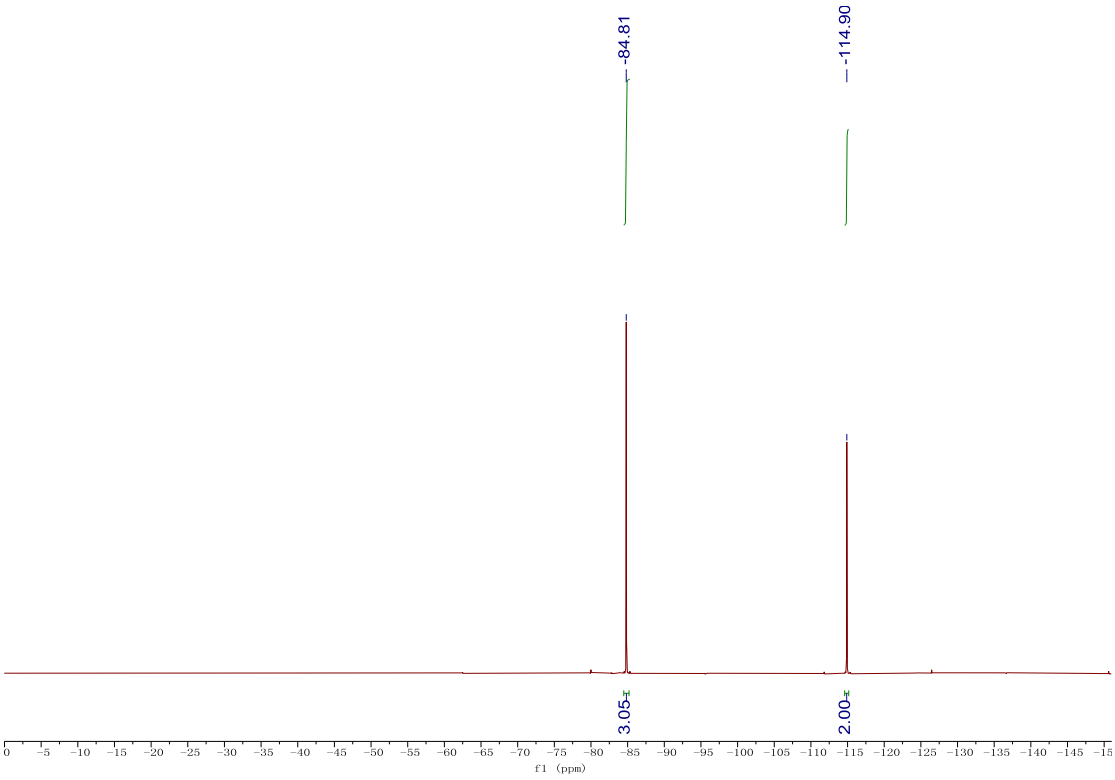
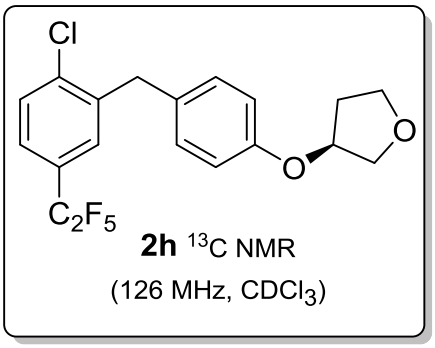
112.13
111.04
110.72
110.40
110.09
77.41
77.16
76.90
f1 (ppm)

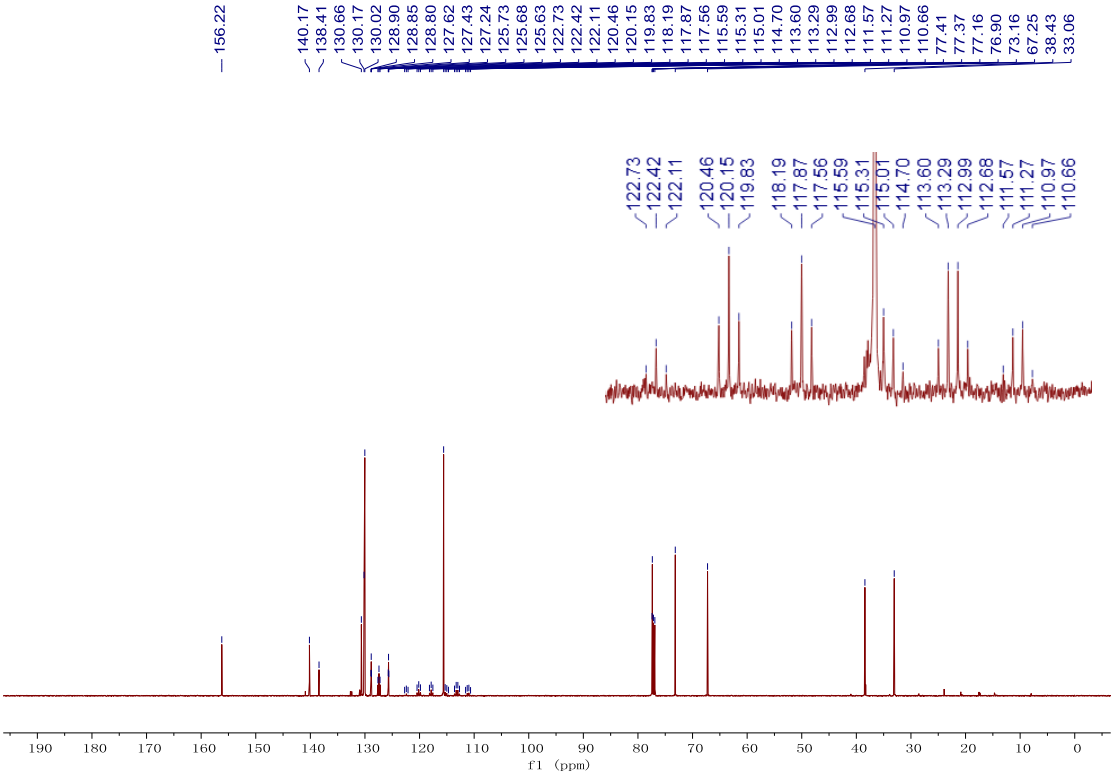

Cl
O
O
C_2F_5
2h ^{1}H NMR
(400 MHz, $CDCl_3$)

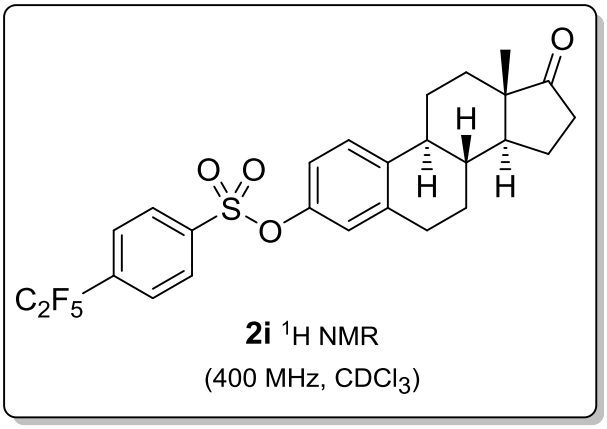

7.51
7.49
7.40
7.38
7.25
7.09
7.07
6.82
6.80
4.89
4.08
4.01
3.99
3.97
3.95
3.92
3.90
3.89
3.87
2.20
2.18
2.17
2.15
0.00
0.90
1.85
2.30
2.18
1.06
2.01
3.09
1.11
2.14
f1 (ppm)

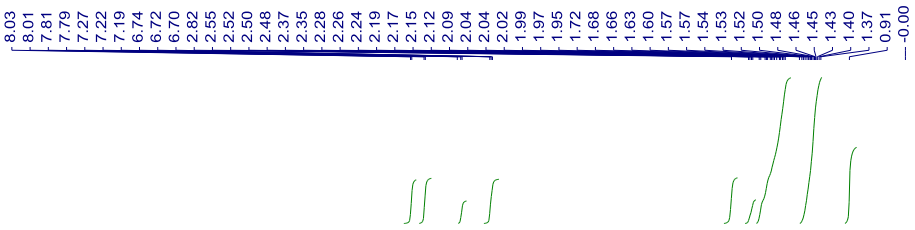

Cl
O
O
C_2F_5
2h ^{19}F NMR
(376 MHz, $CDCl_3$)

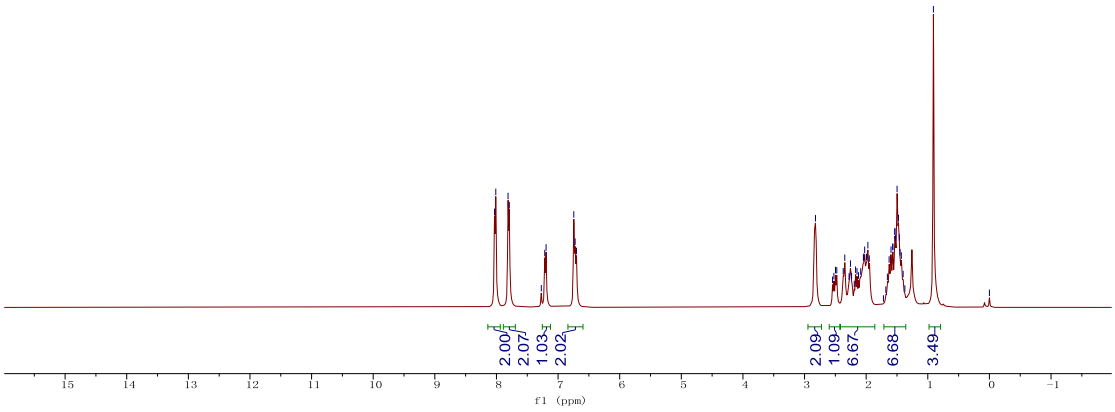

-84.81
-114.90
3.05
2.00
f1 (ppm)

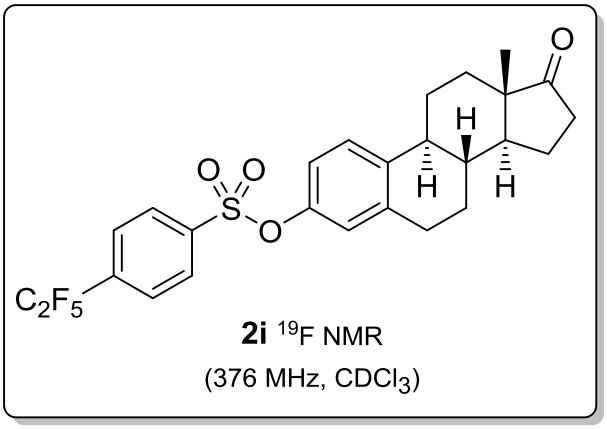

Cl
O
O
C_2F_5
2h ^{13}C NMR
(126 MHz, $CDCl_3$)


156.22
140.17
138.41
130.66
130.17
130.02
128.90
128.85
128.80
127.62
127.43
127.24
125.73
125.68
125.63
122.73
122.42
122.11
120.46
120.15
119.83
118.19
117.87
117.56
115.59
115.31
115.01
114.70
113.60
113.29
112.99
112.68
111.57
111.27
110.97
110.66
77.41
77.37
77.16
76.90
73.16
67.25
38.43
33.06
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
H
H
H
O
O
S
O
C2F5
2i 1H NMR
(400 MHz, CDCl3)

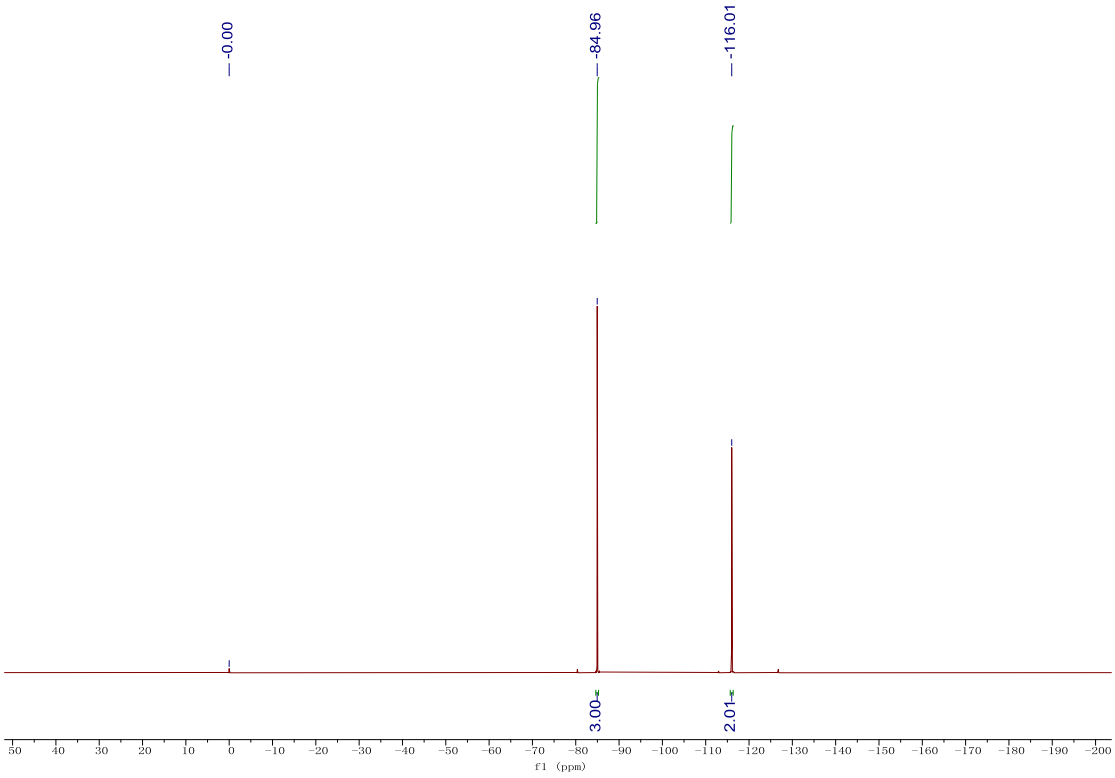

8.03
8.01
7.81
7.79
7.27
7.22
7.19
6.74
6.72
6.70
2.82
2.55
2.52
2.50
2.48
2.37
2.35
2.28
2.26
2.24
2.19
2.17
2.15
2.12
2.09
2.04
2.04
2.02
1.99
1.97
1.95
1.72
1.68
1.66
1.63
1.60
1.57
1.57
1.54
1.53
1.52
1.50
1.48
1.46
1.45
1.43
1.40
1.37
0.91
-0.00

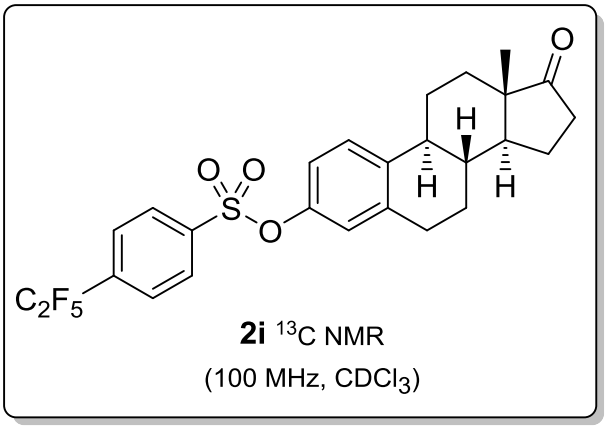

2.00
2.07
1.03
2.02
2.09
1.09
6.67
6.68
3.49
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)



2i ^{19}F NMR

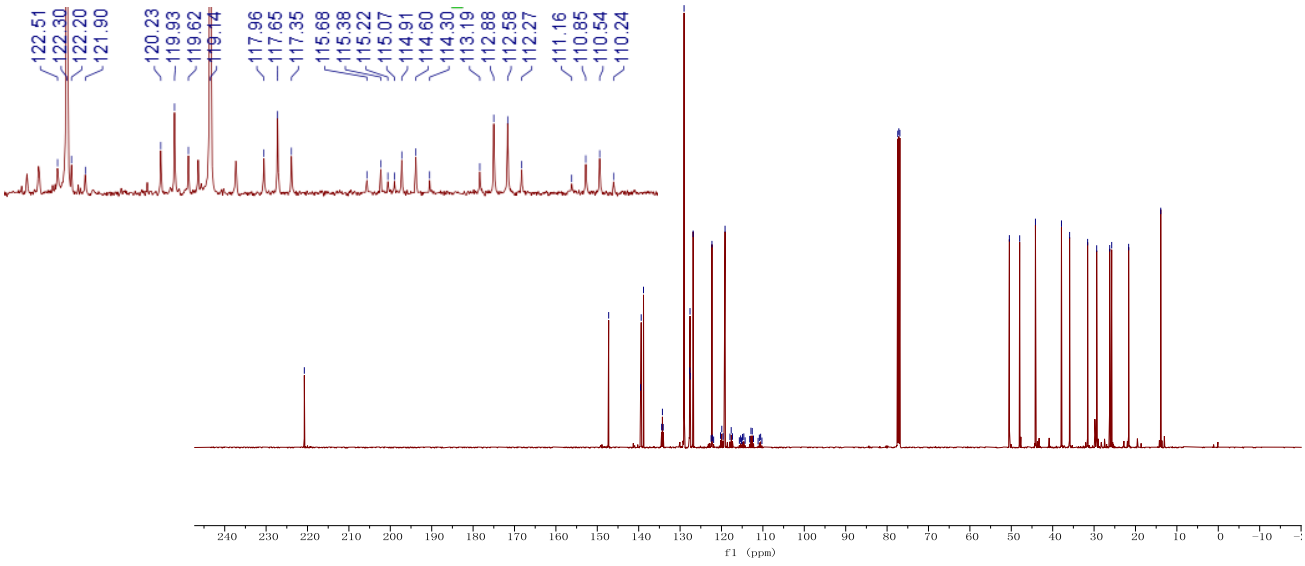
(376 MHz, $CDCl_3$)

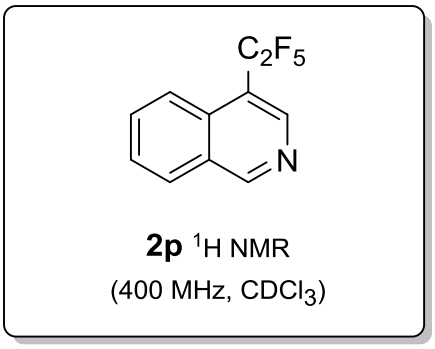


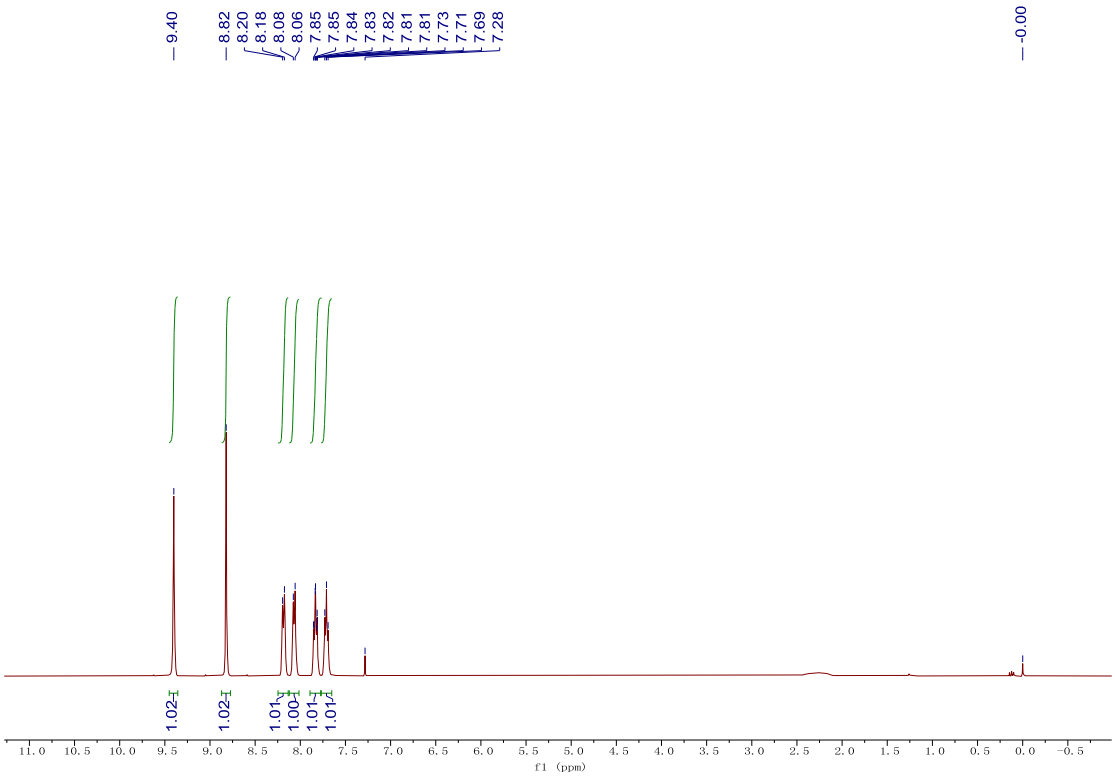


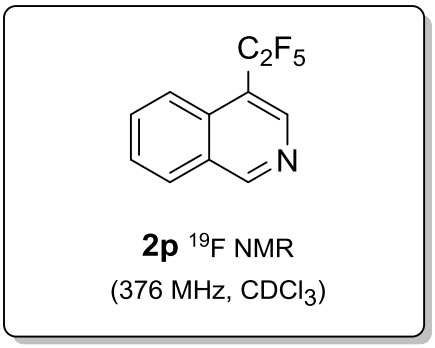
2i ^{13}C NMR

(100 MHz, $CDCl_3$)



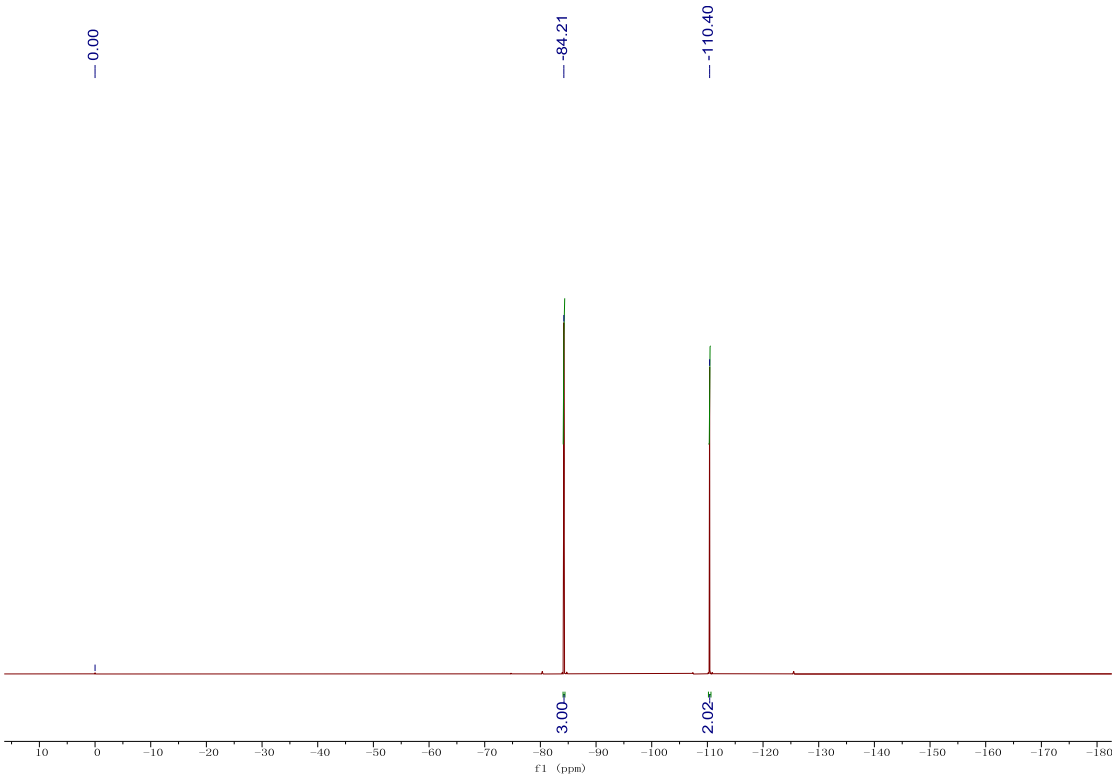

C_2F_5
N
2p ^{1}H NMR
(400 MHz, $CDCl_3$)

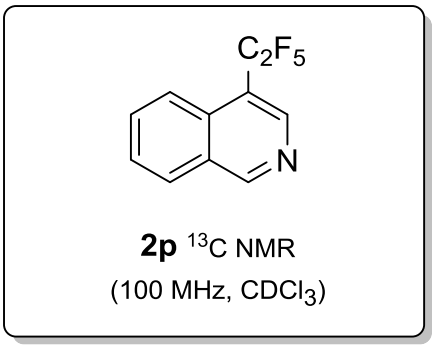

9.40
8.82
8.20
8.18
8.08
8.06
7.85
7.85
7.84
7.83
7.82
7.81
7.81
7.73
7.71
7.69
7.28
-0.00
1.02
1.02
1.01
1.00
1.01
1.01
f1 (ppm)

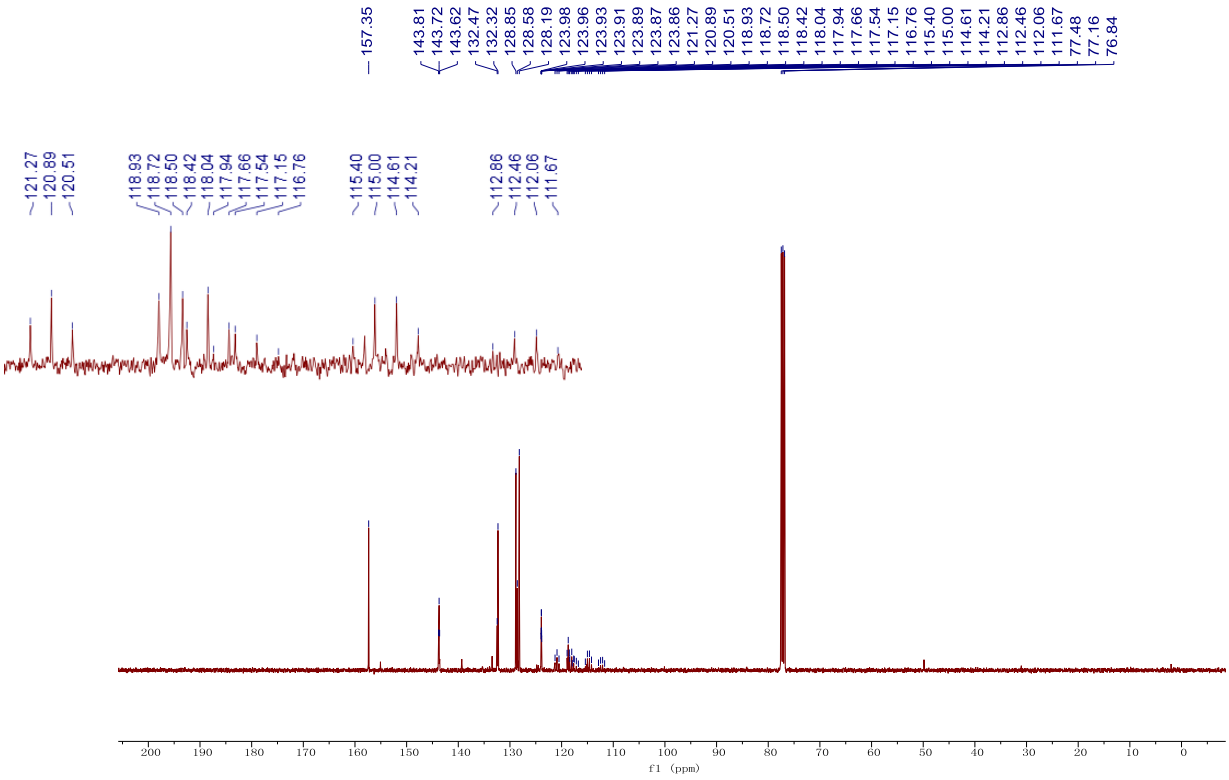


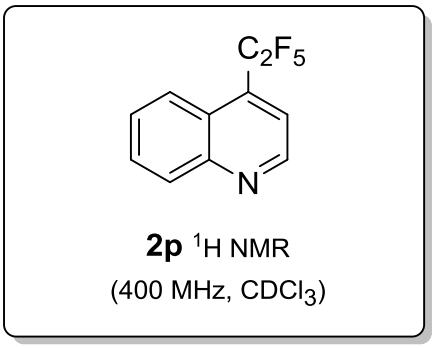
2p ^{19}F NMR

(376 MHz, $CDCl_3$)



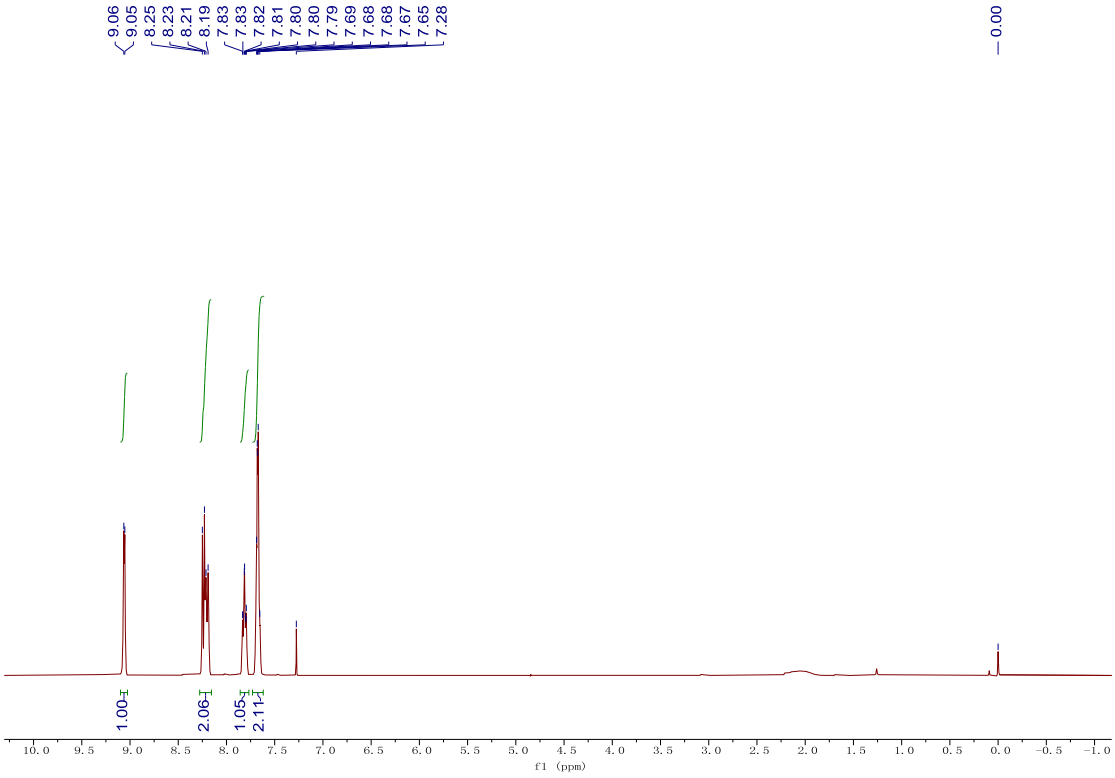

C2F5
N
2p 13C NMR
(100 MHz, CDCl3)

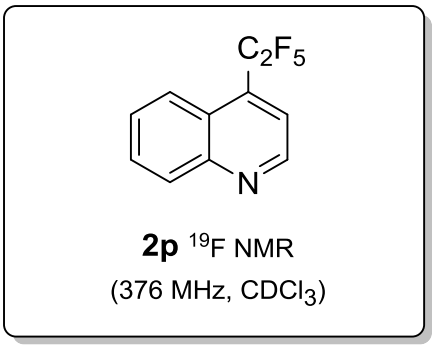

157.35
143.81
143.72
143.62
132.47
132.32
128.85
128.58
128.19
123.98
123.96
123.93
123.91
123.89
123.87
123.86
121.27
120.89
120.51
118.93
118.72
118.50
118.42
118.04
117.94
117.66
117.54
117.15
116.76
115.40
115.00
114.61
114.21
112.86
112.46
112.06
111.67
77.48
77.16
76.84
f1 (ppm)



2p ^{1}H NMR

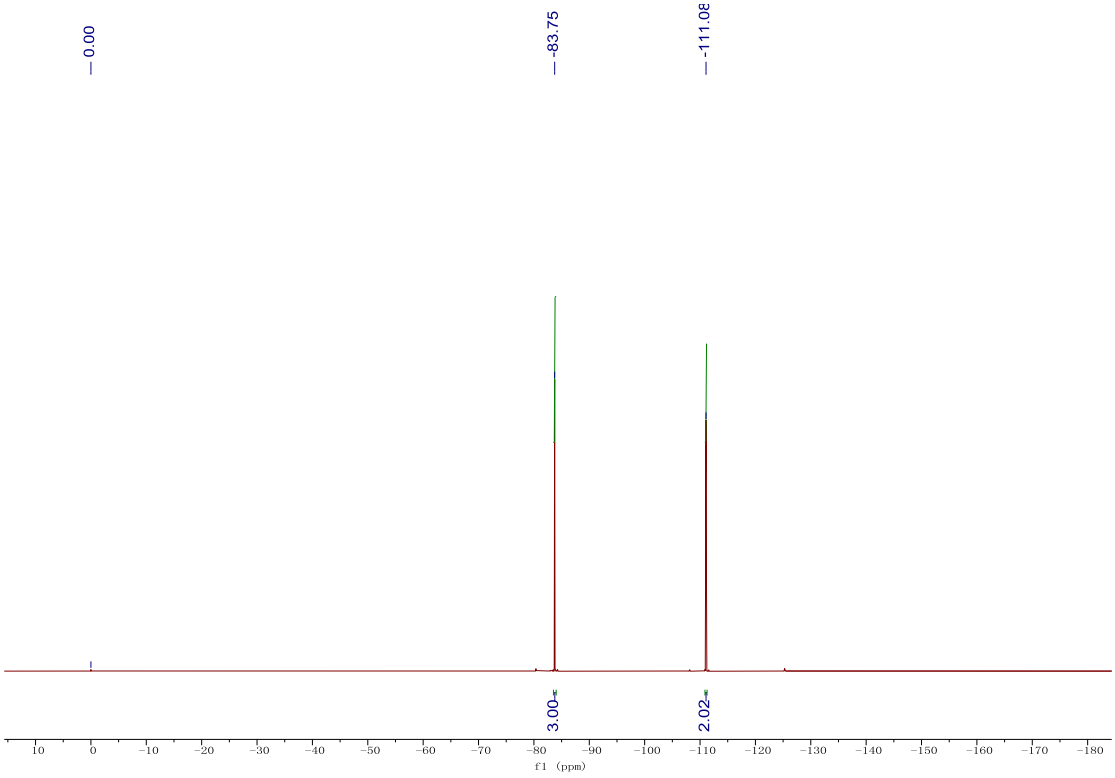
(400 MHz, $CDCl_3$)

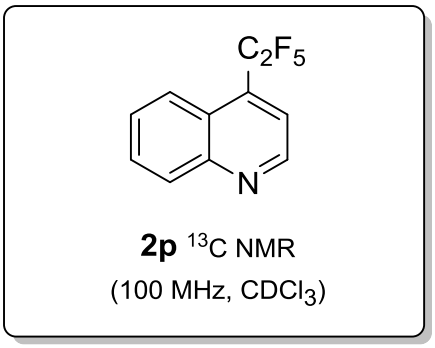


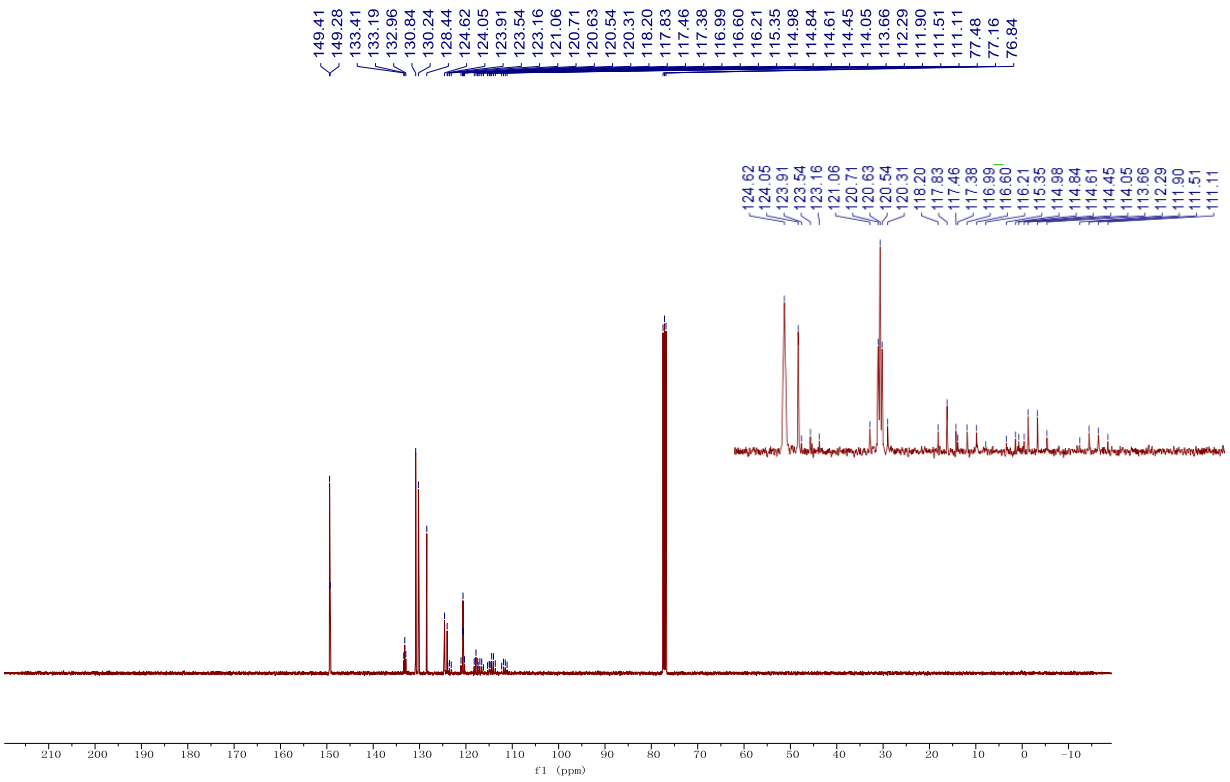


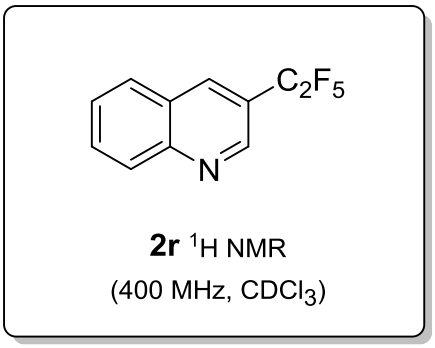
2p ^{19}F NMR

(376 MHz, $CDCl_3$)

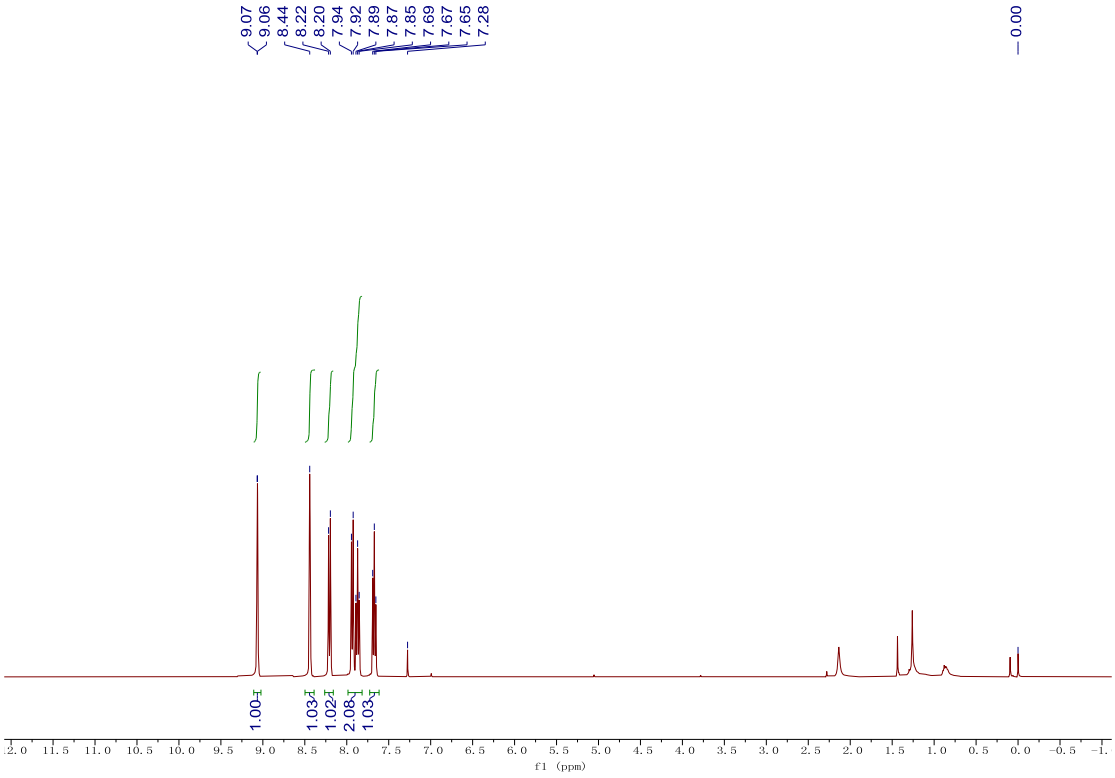


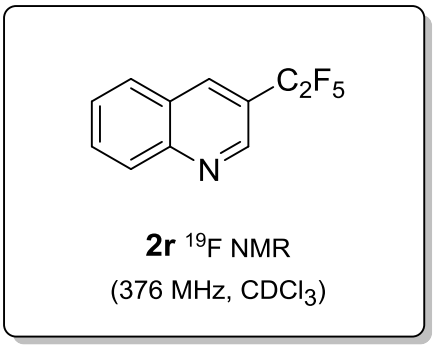

C_2F_5
N
2p ^{13}C NMR
(100 MHz, $CDCl_3$)


149.41
149.28
133.41
133.19
132.96
130.84
130.24
128.44
124.62
124.05
123.91
123.54
123.16
121.06
120.71
120.63
120.54
120.31
118.20
117.83
117.46
117.38
116.99
116.60
116.21
115.35
114.98
114.84
114.61
114.45
114.05
113.66
112.29
111.90
111.51
111.11
77.48
77.16
76.84
f1 (ppm)



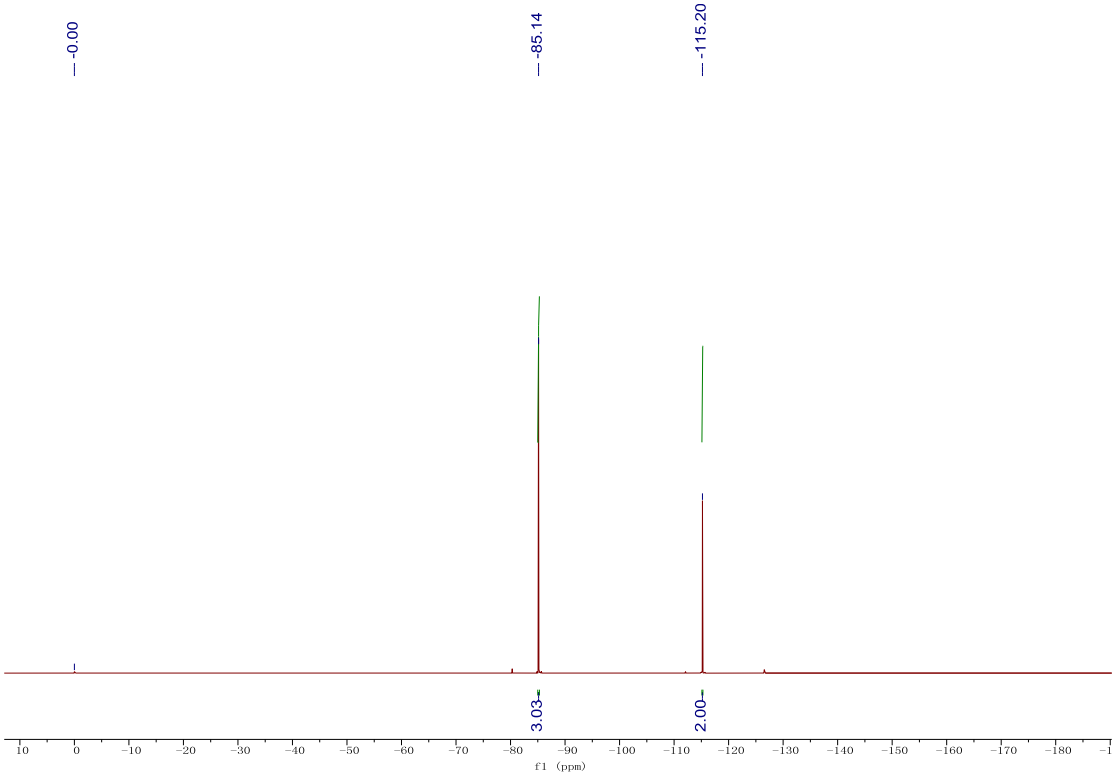
2r ^{1}H NMR
(400 MHz, $CDCl_3$)

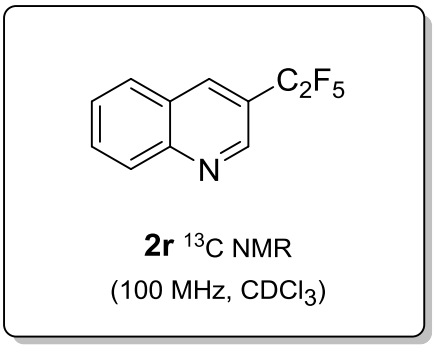




2r ^{19}F NMR

(376 MHz, $CDCl_3$)





2r ^{13}C NMR

(100 MHz, $CDCl_3$)

149.55
146.68
146.63
146.57
135.86
135.79
135.73
132.12
129.76
128.74
128.18
126.47
123.76
123.00
122.22
121.98
121.74
120.92
120.54
120.15
118.08
117.70
117.31
116.29
115.90
115.51
115.24
115.13
114.85
114.46
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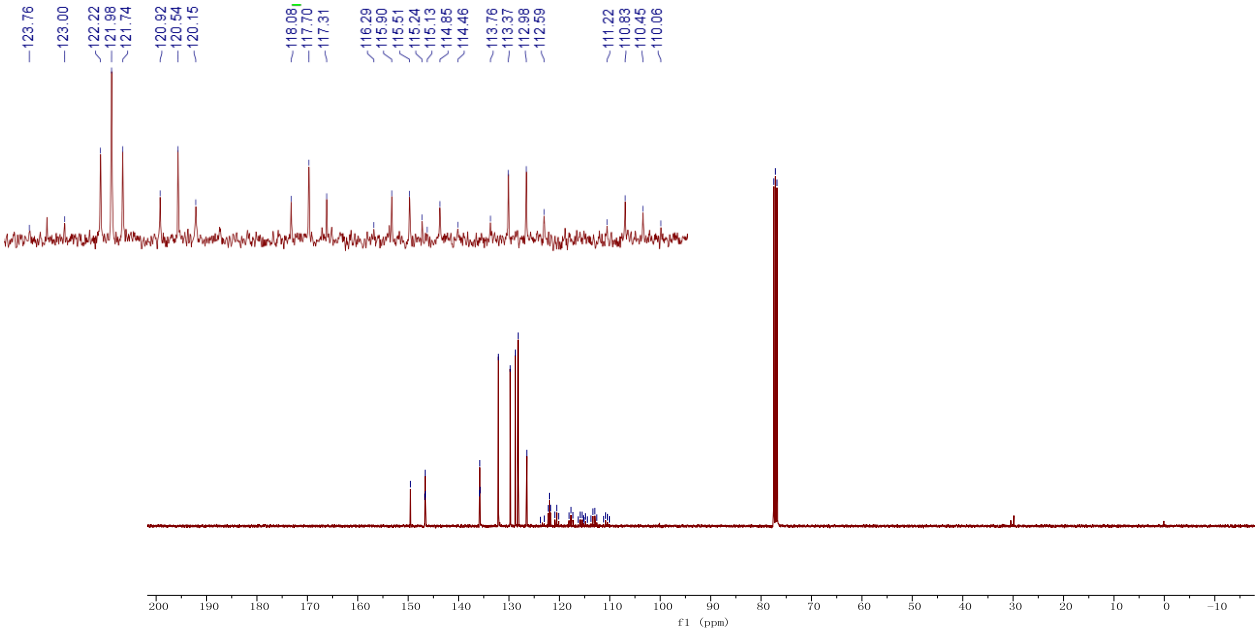
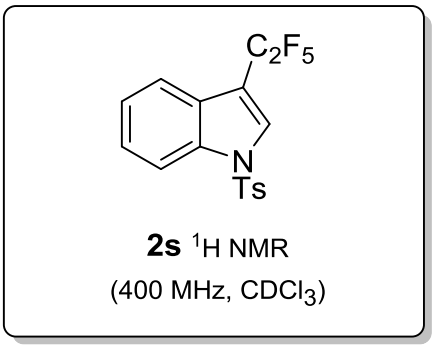
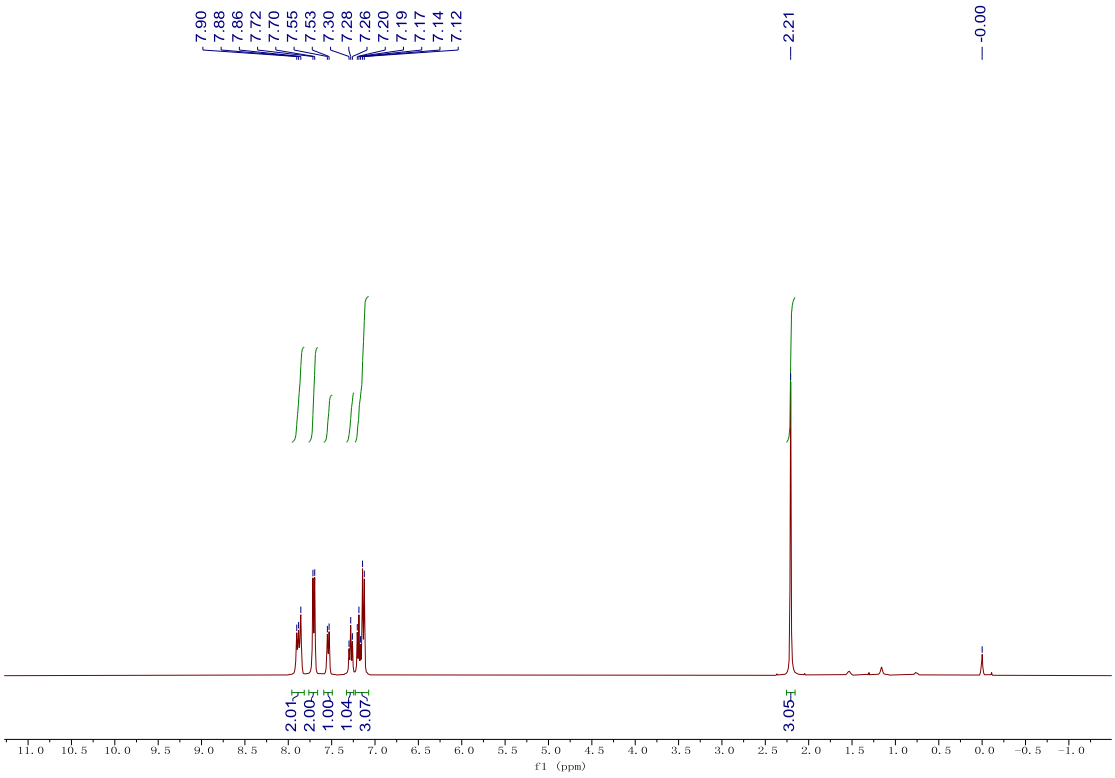
112.98
112.59
111.22
110.83
110.45
110.06
77.48
77.16
76.84

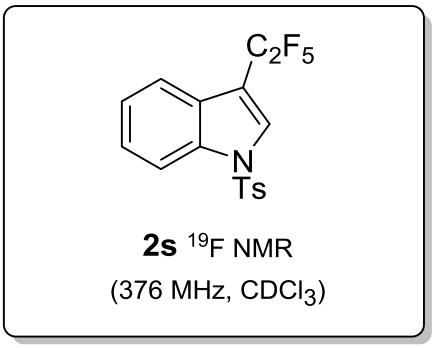




2s ^{1}H NMR

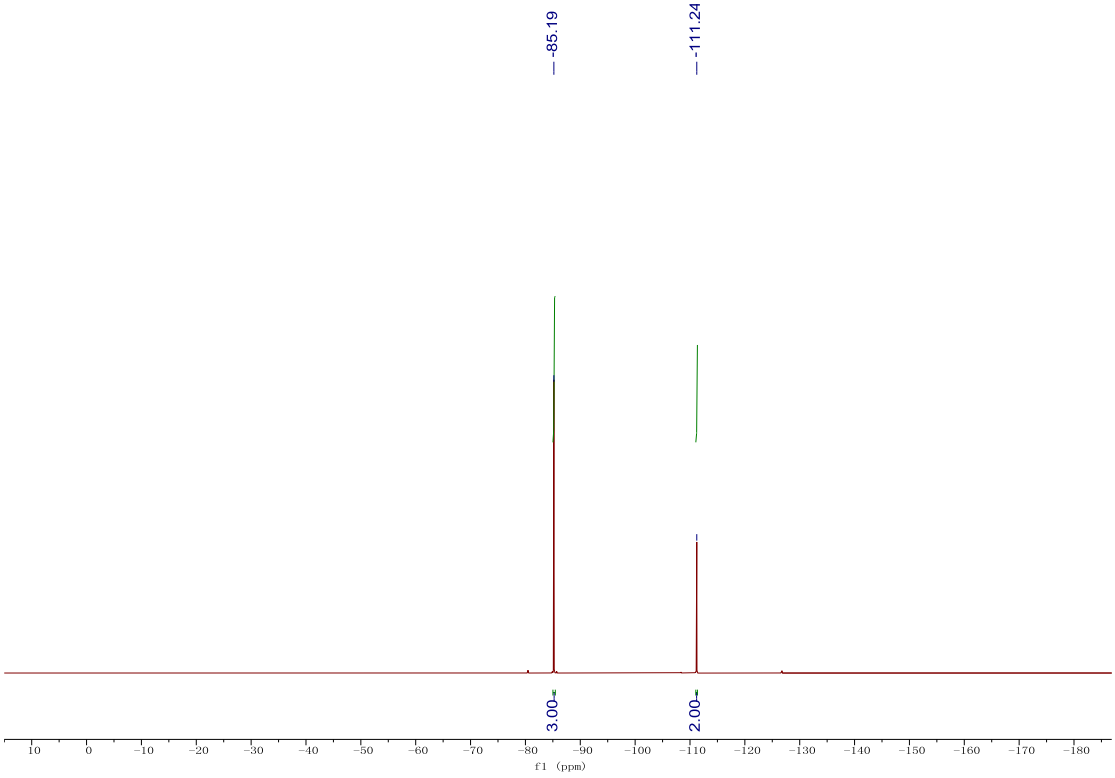
(400 MHz, $CDCl_3$)

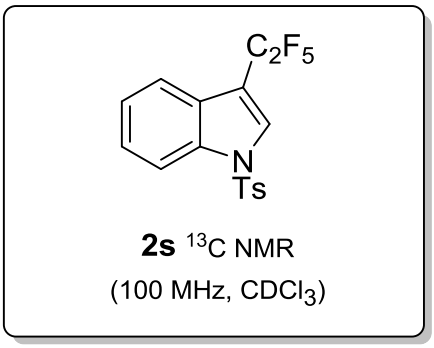




2s ^{19}F NMR

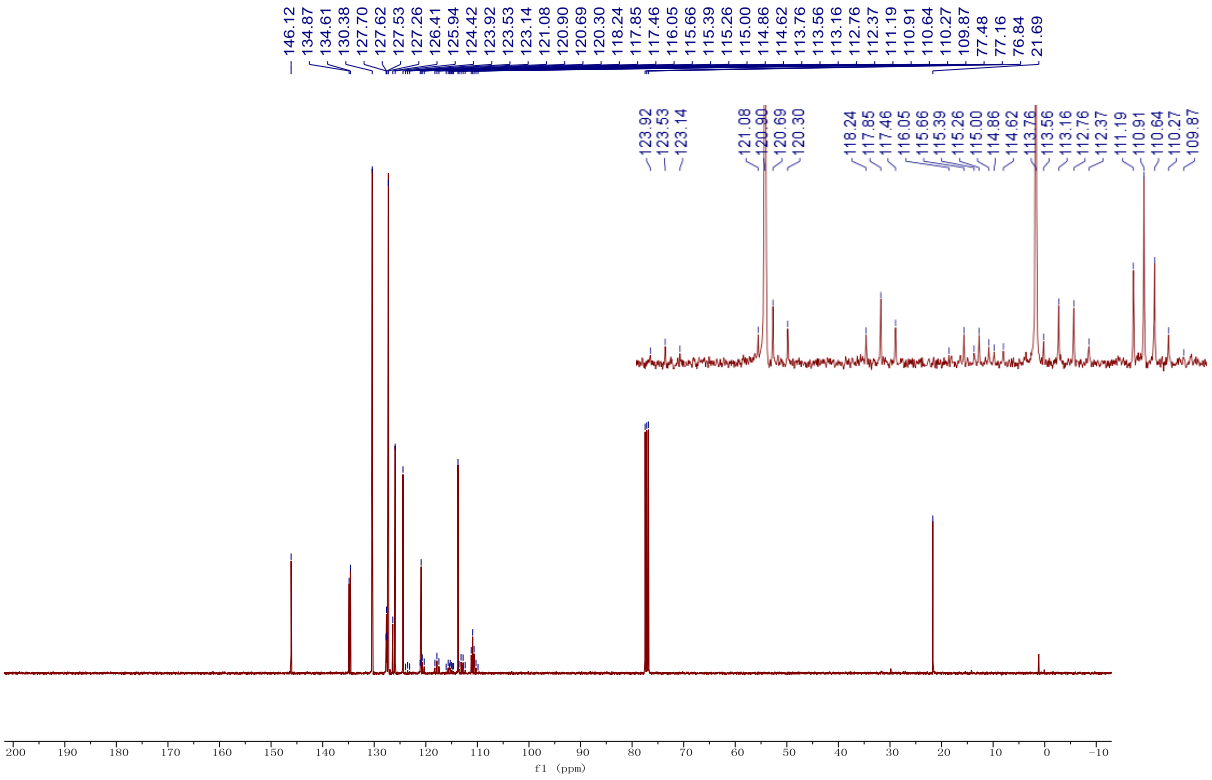
(376 MHz, $CDCl_3$)

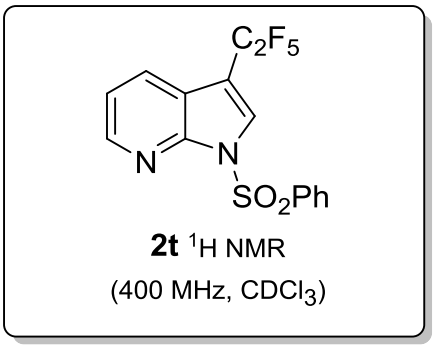




2s ^{13}C NMR

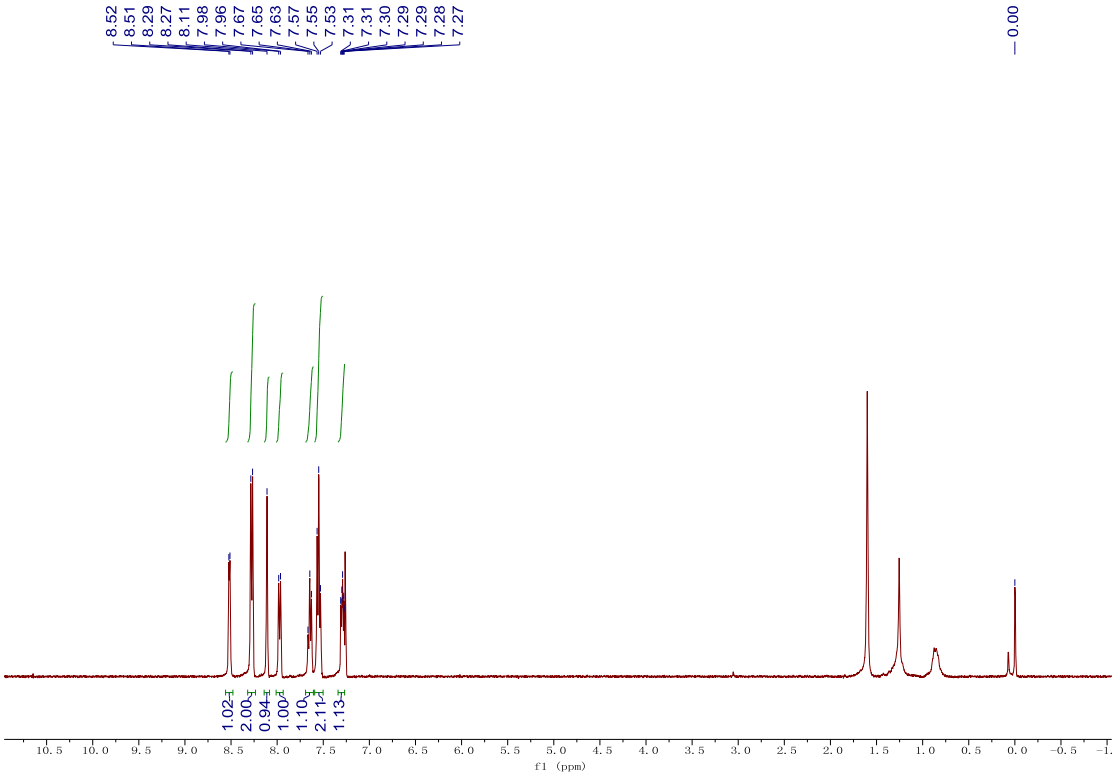
(100 MHz, $CDCl_3$)

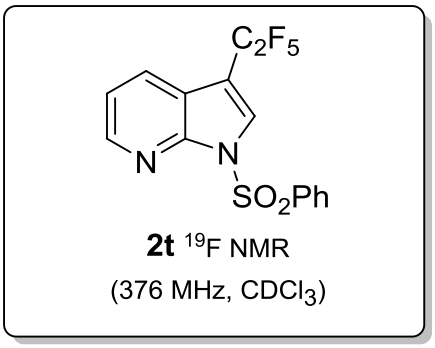




2t ^{1}H NMR

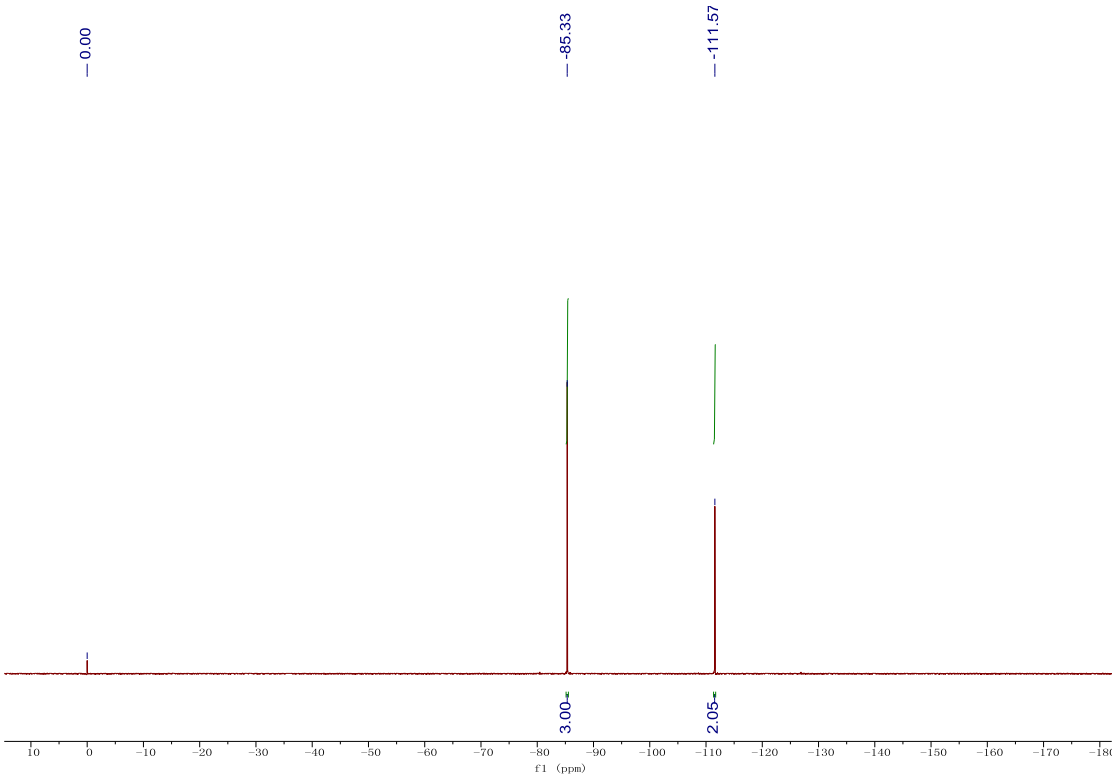
(400 MHz, $CDCl_3$)

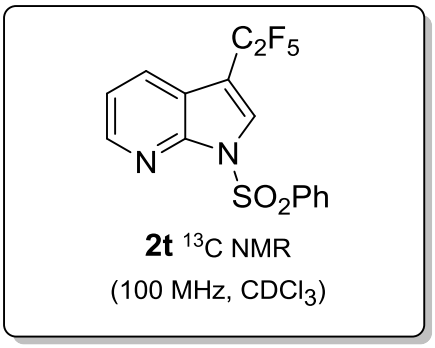


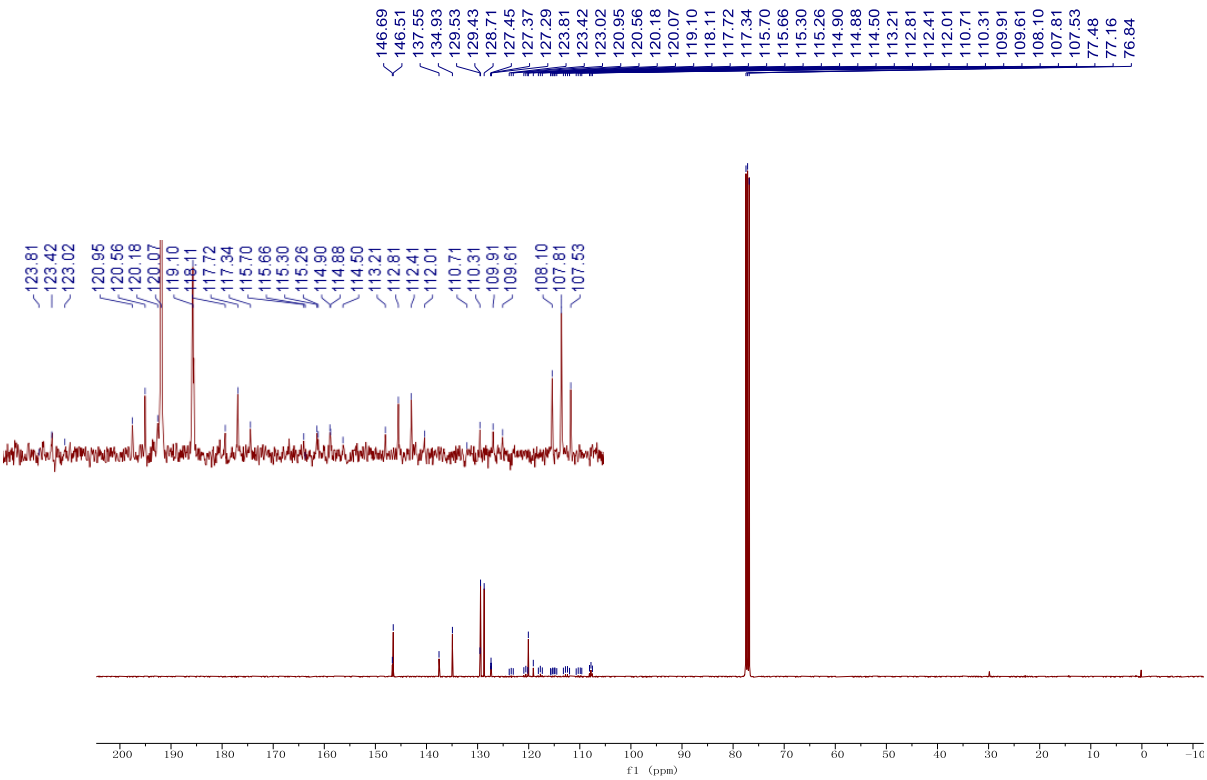


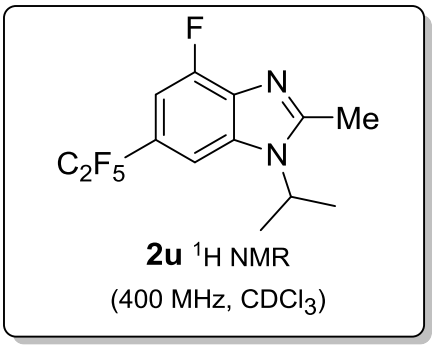
2t ^{19}F NMR

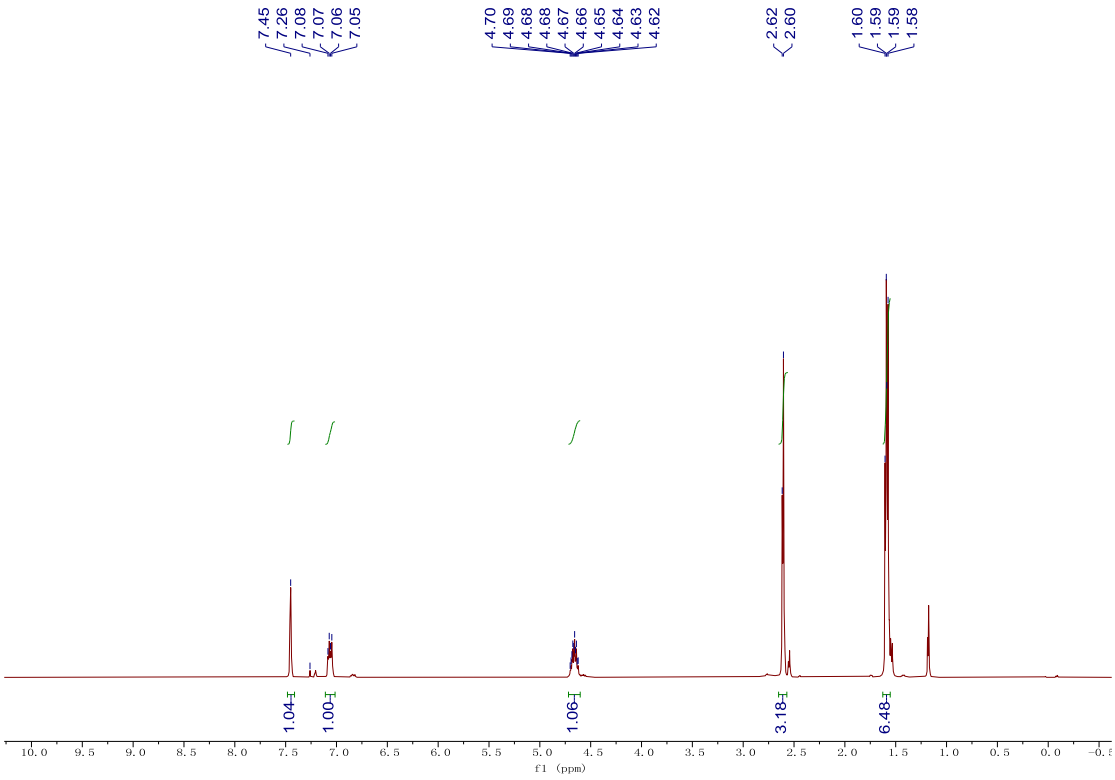
(376 MHz, $CDCl_3$)

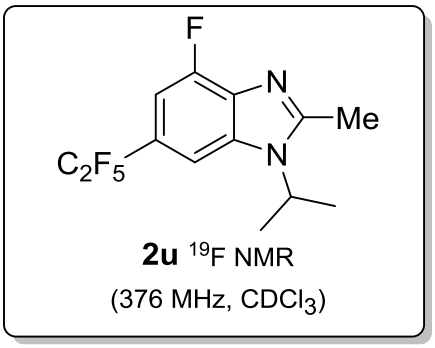



C2F5
N
N
SO2Ph
2t 13C NMR
(100 MHz, CDCl3)


146.69
146.51
137.55
134.93
129.53
129.43
128.71
127.45
127.37
127.29
123.81
123.42
123.02
120.95
120.56
120.18
120.07
119.10
118.11
117.72
117.34
115.70
115.66
115.30
115.26
114.90
114.88
114.50
113.21
112.81
112.41
112.01
110.71
110.31
109.91
109.61
108.10
107.81
107.53
77.48
77.16
76.84
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

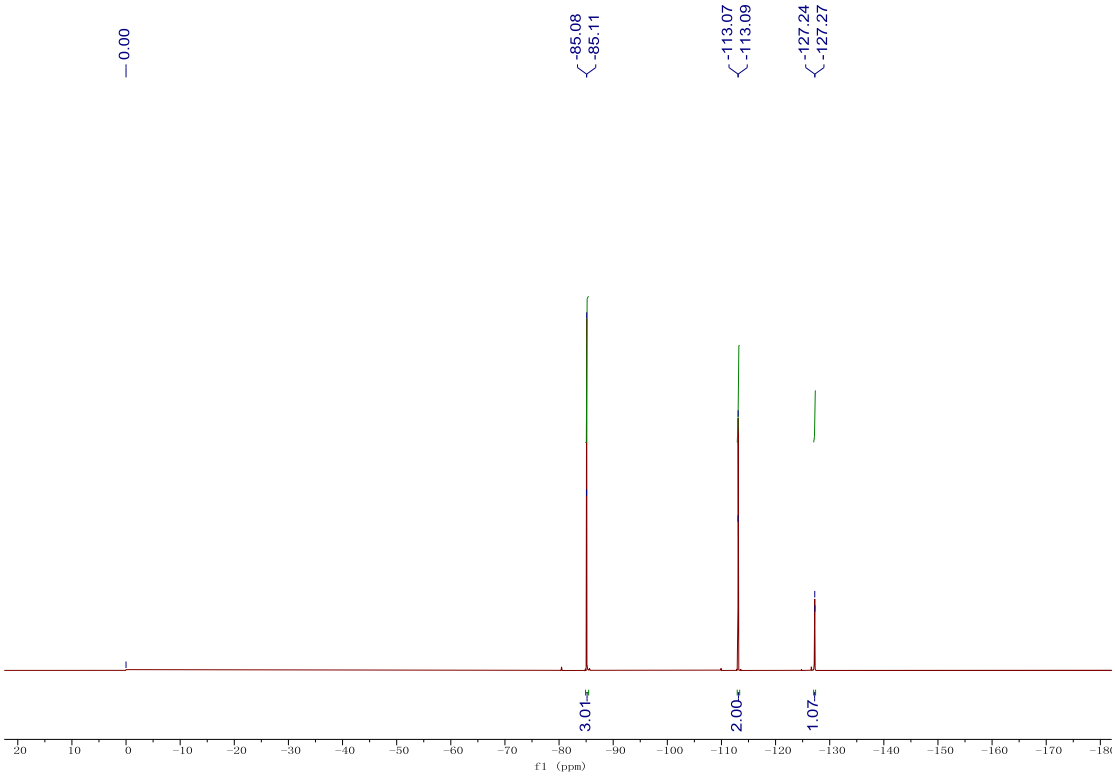

F
N
Me
N
C_2F_5
2u ^{1}H NMR
(400 MHz, $CDCl_3$)

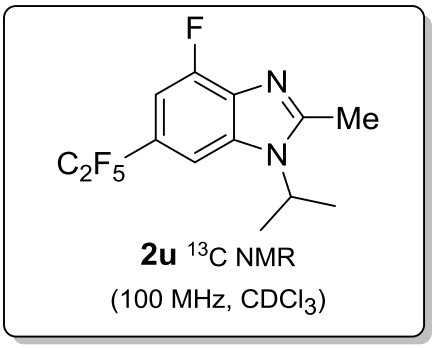

7.45
7.26
7.08
7.07
7.06
7.05
4.70
4.69
4.68
4.68
4.67
4.66
4.65
4.64
4.63
4.62
2.62
2.60
1.60
1.59
1.59
1.58
1.04
1.00
1.06
3.18
6.48
f1 (ppm)



2u ^{19}F NMR

(376 MHz, $CDCl_3$)

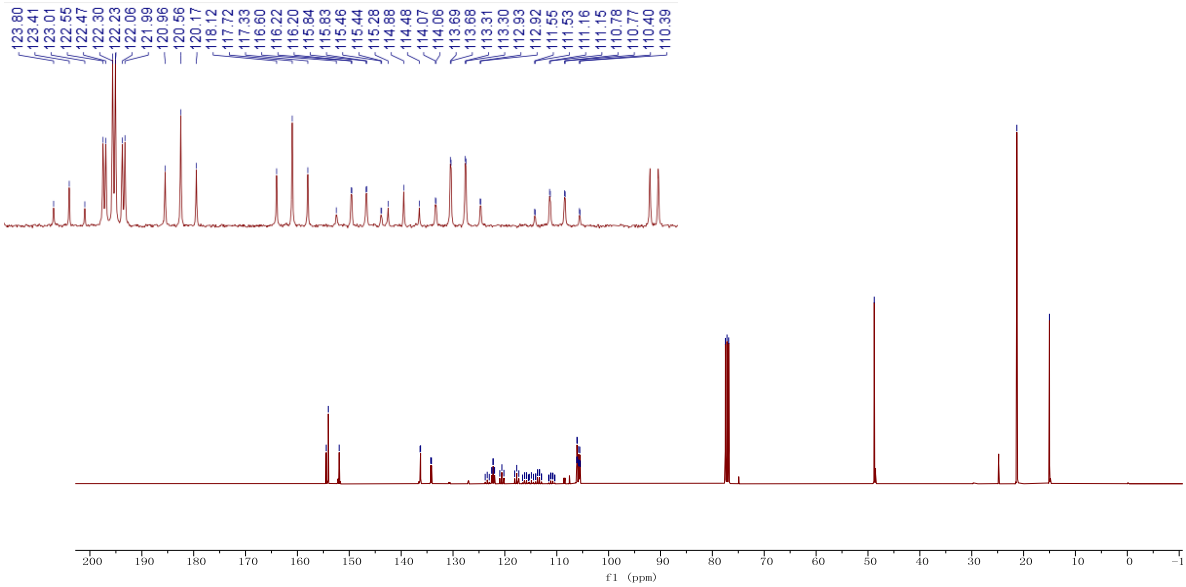


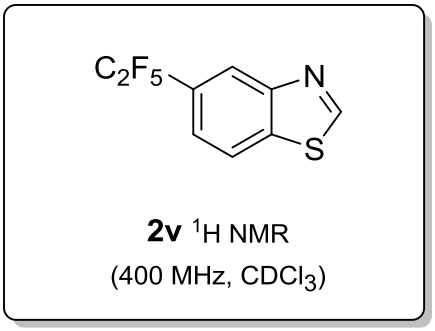


2u ^{13}C NMR

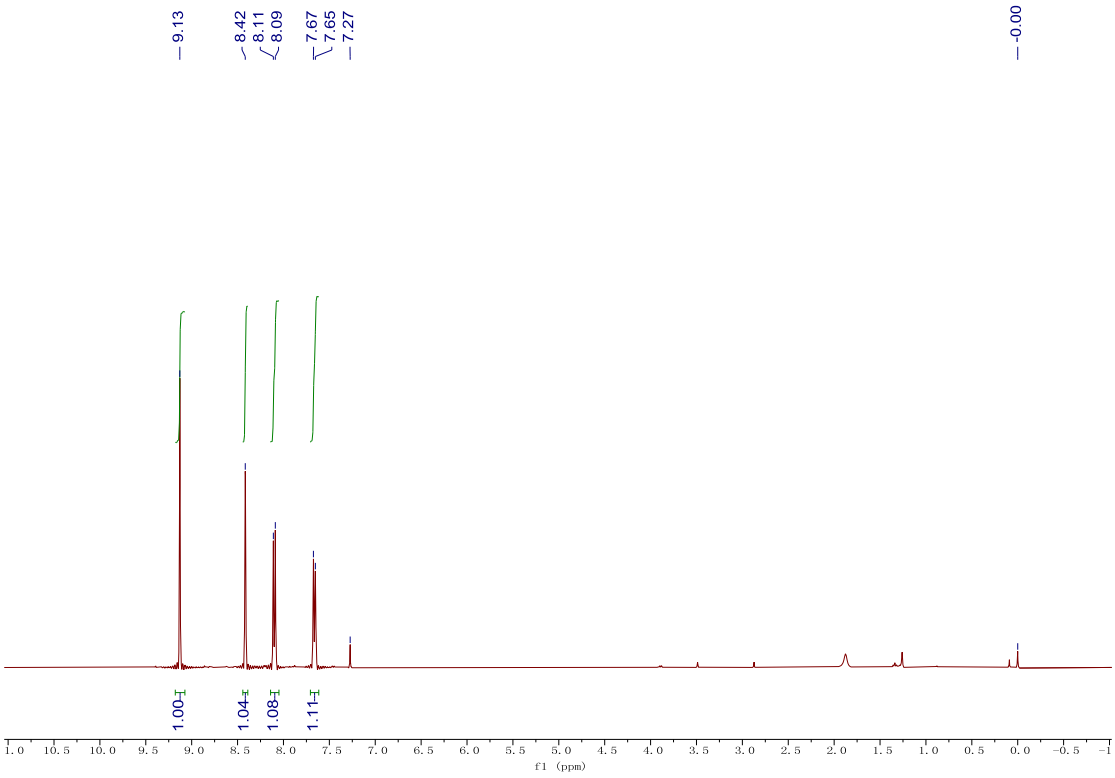
(100 MHz, $CDCl_3$)

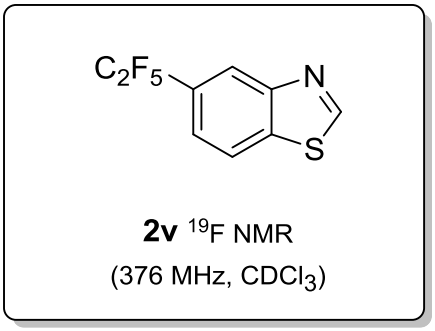
154.47
154.06
151.94
136.35
136.25
134.29
134.13
123.80
123.41
123.01
122.55
122.47
122.30
122.23
122.06
121.99
120.96
120.56
120.17
118.12
117.72
117.33
116.60
116.22
116.20
115.84
115.83
115.46
115.44
115.28
114.88
114.48
114.07
114.06
113.69
113.68
113.31
113.30
112.93
112.92
111.55
111.53
111.16
111.15
110.78
110.77
110.40
110.39





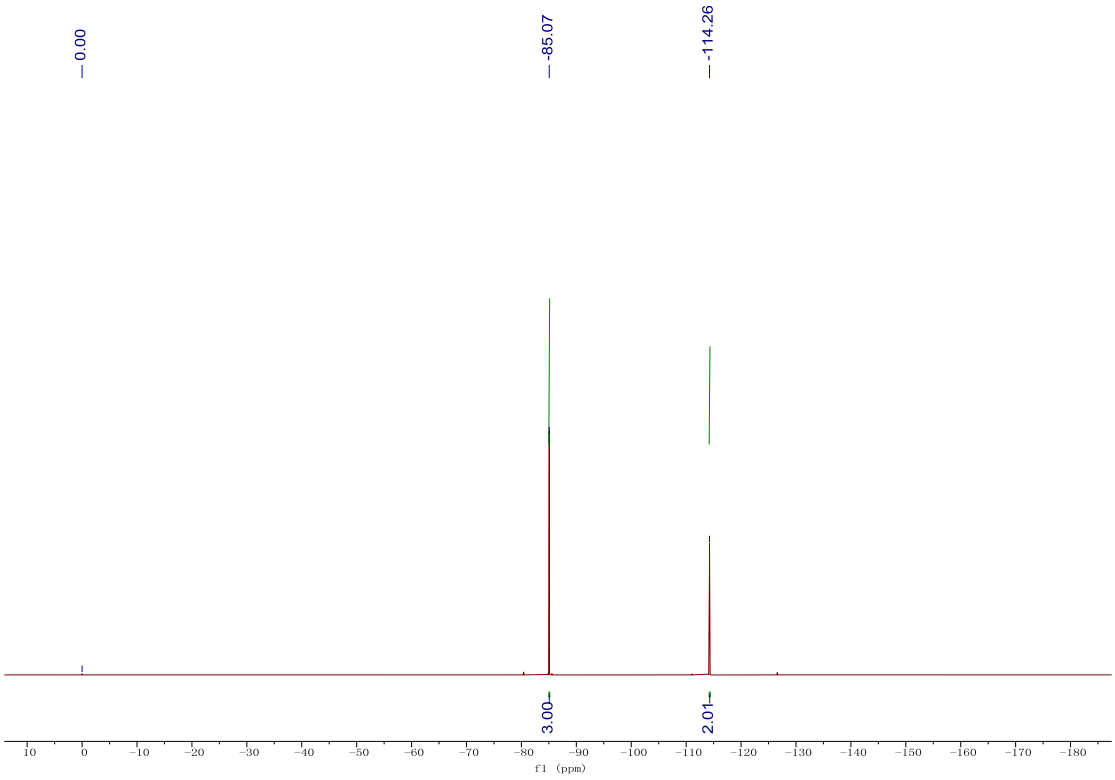
2v ^{1}H NMR
(400 MHz, $CDCl_3$)

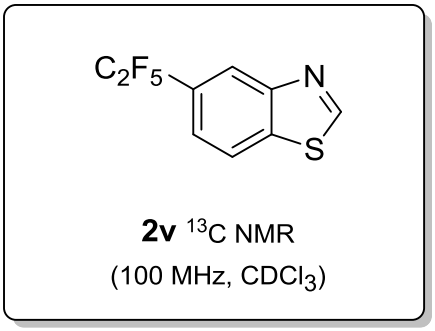


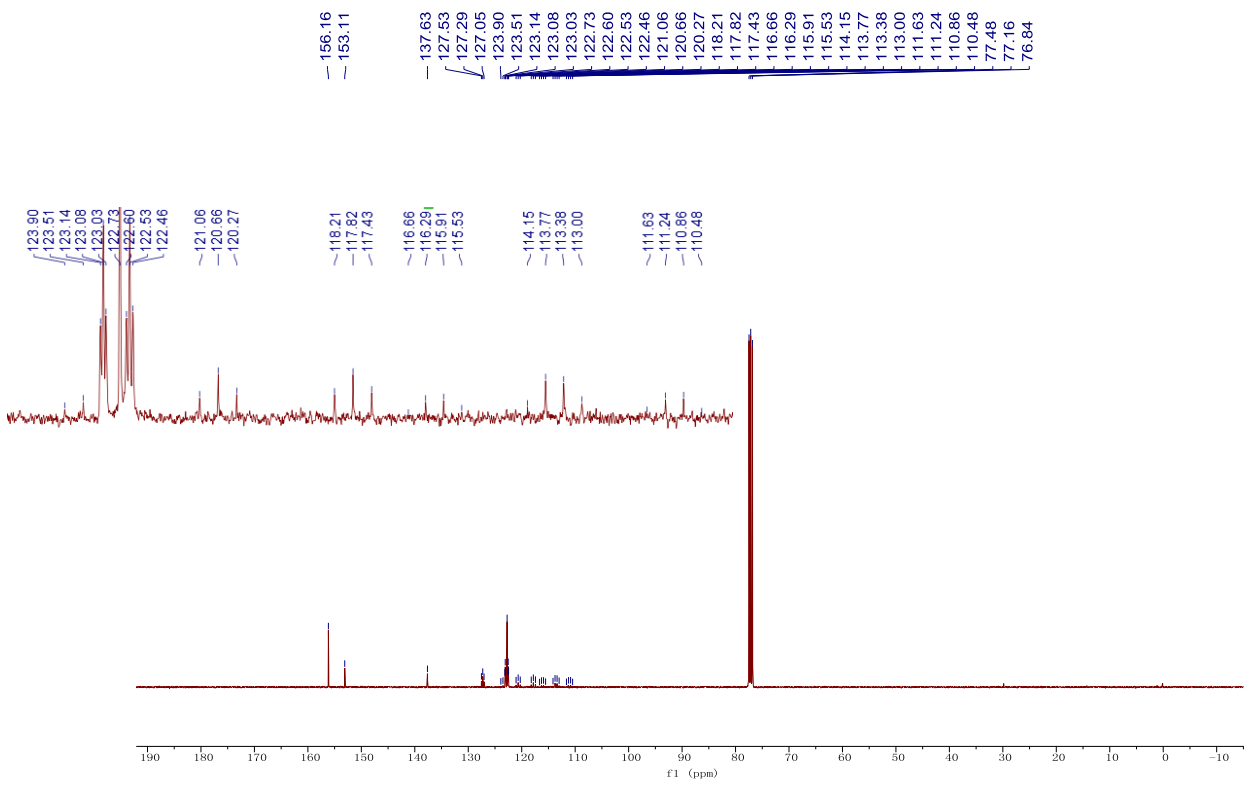


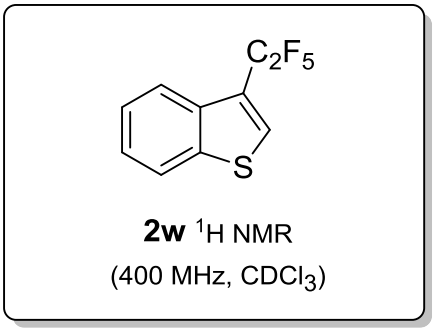
2v ^{19}F NMR

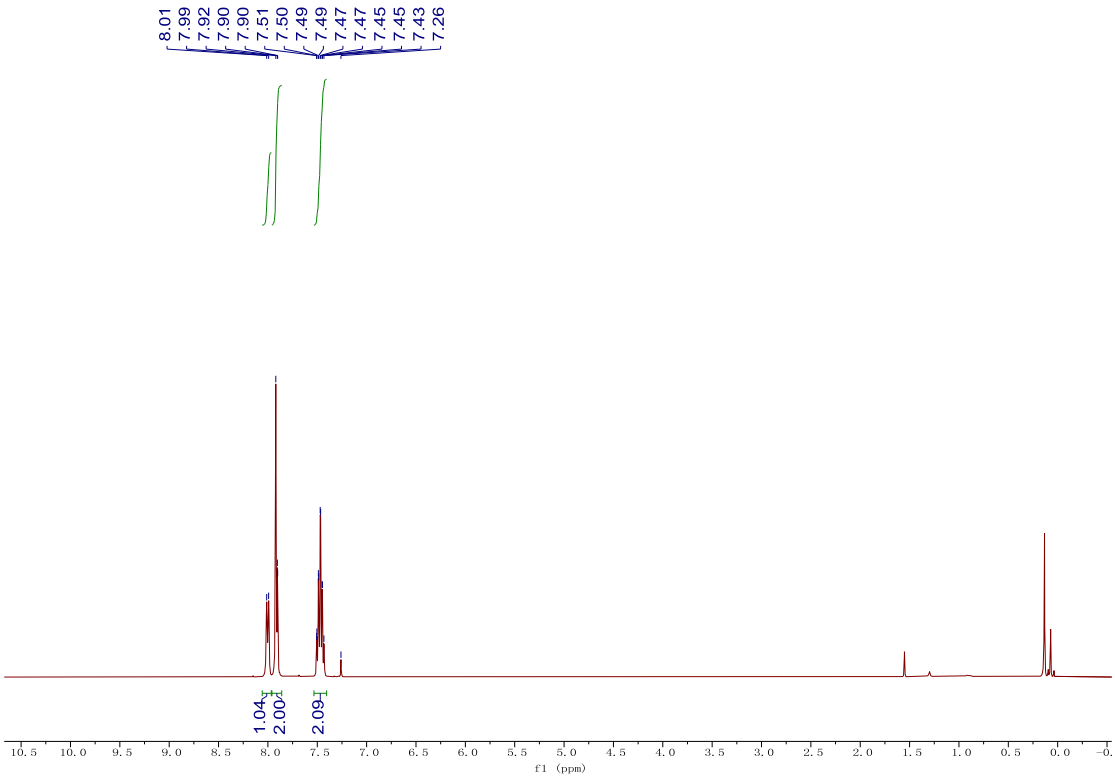
(376 MHz, $CDCl_3$)

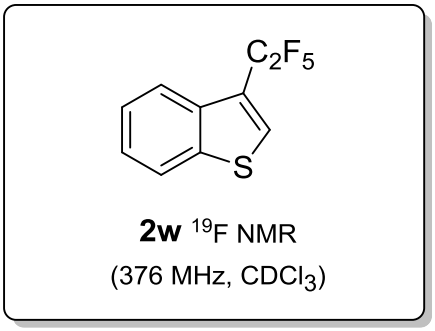



C2F5
N
S
2v 13C NMR
(100 MHz, CDCl3)


156.16
153.11
137.63
127.53
127.29
127.05
123.90
123.51
123.14
123.08
123.03
122.73
122.60
122.53
122.46
121.06
120.66
120.27
118.21
117.82
117.43
116.66
116.29
115.91
115.53
114.15
113.77
113.38
113.00
111.63
111.24
110.86
110.48
77.48
77.16
76.84
f1 (ppm)

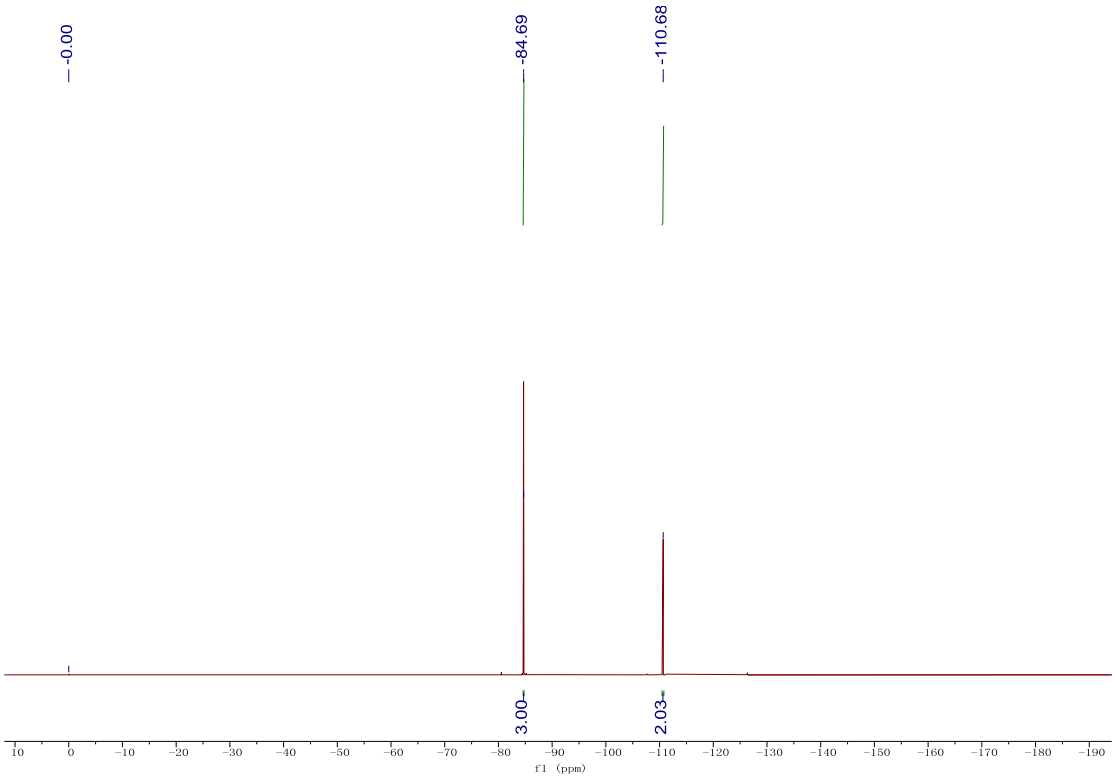

C_2F_5
S
2w ^{1}H NMR
(400 MHz, $CDCl_3$)

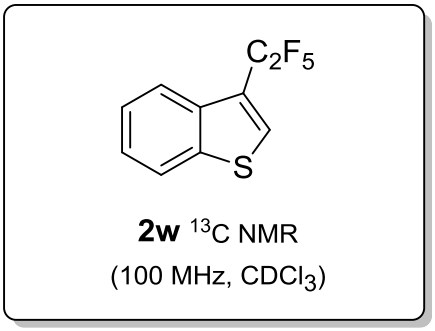

8.01
7.99
7.92
7.90
7.90
7.51
7.50
7.49
7.49
7.47
7.47
7.45
7.45
7.43
7.26
1.04
2.00
2.09
f1 (ppm)

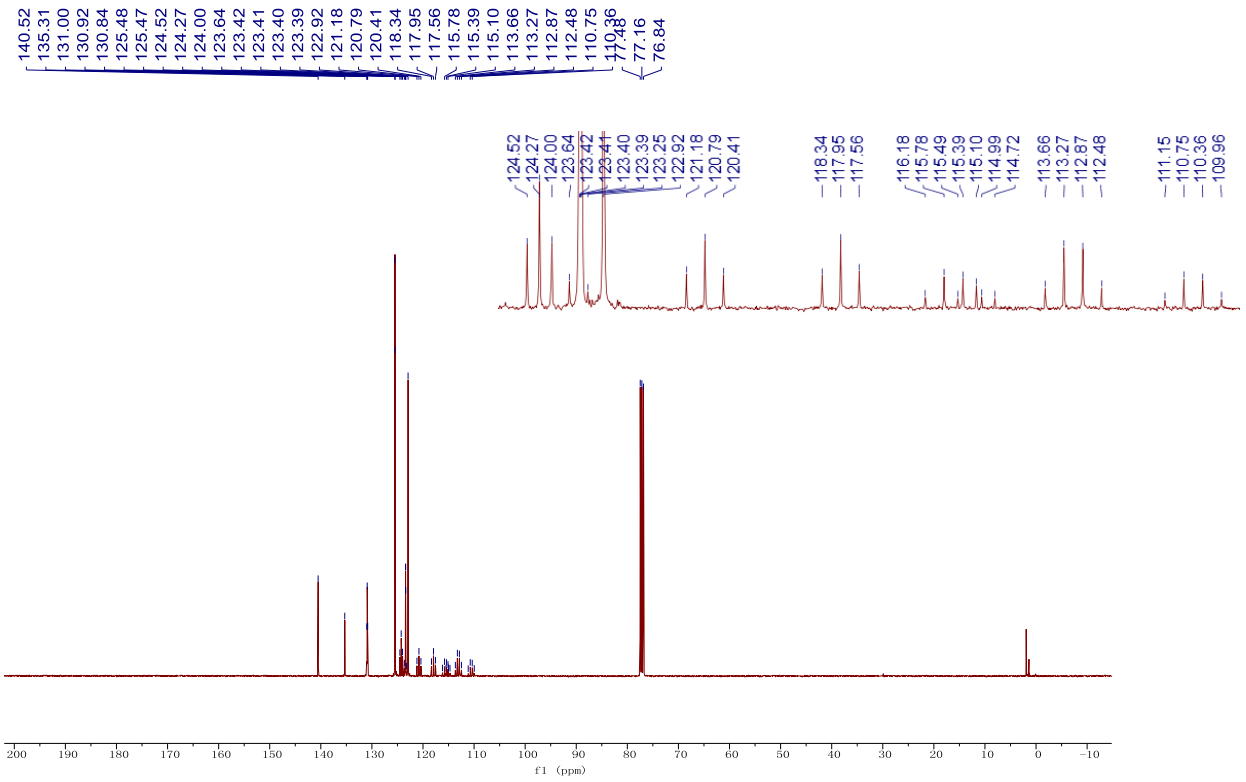


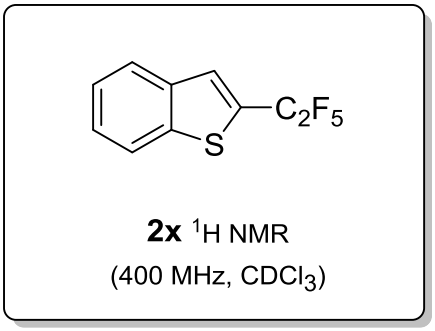
2w ^{19}F NMR

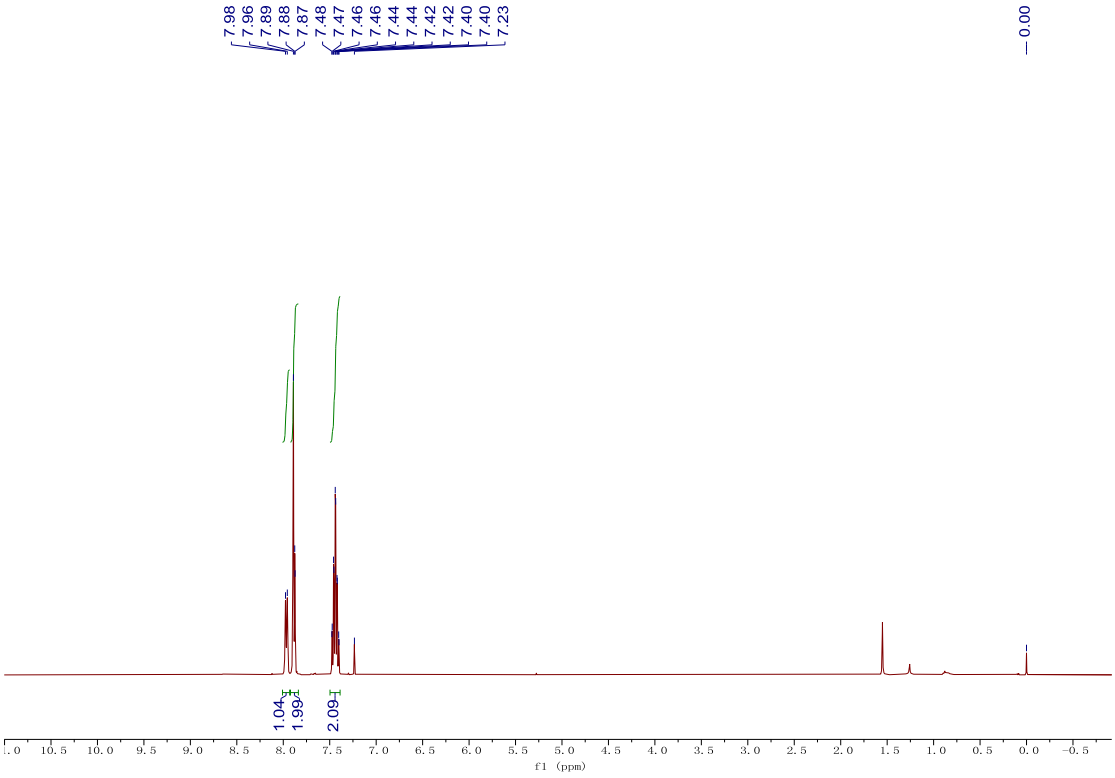
(376 MHz, $CDCl_3$)

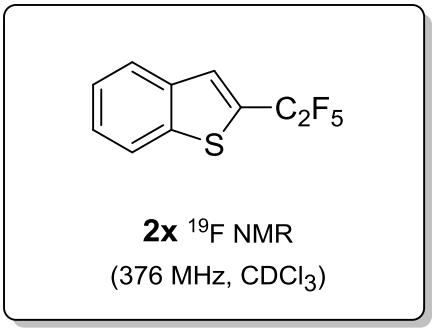



C_2F_5
S
2w ^{13}C NMR
(100 MHz, $CDCl_3$)


140.52
135.31
131.00
130.92
130.84
125.48
125.47
124.52
124.27
124.00
123.64
123.42
123.41
123.40
123.39
122.92
121.18
120.79
120.41
118.34
117.95
117.56
115.78
115.39
115.10
113.66
113.27
112.87
112.48
110.75
110.36
77.48
77.16
76.84
f1 (ppm)

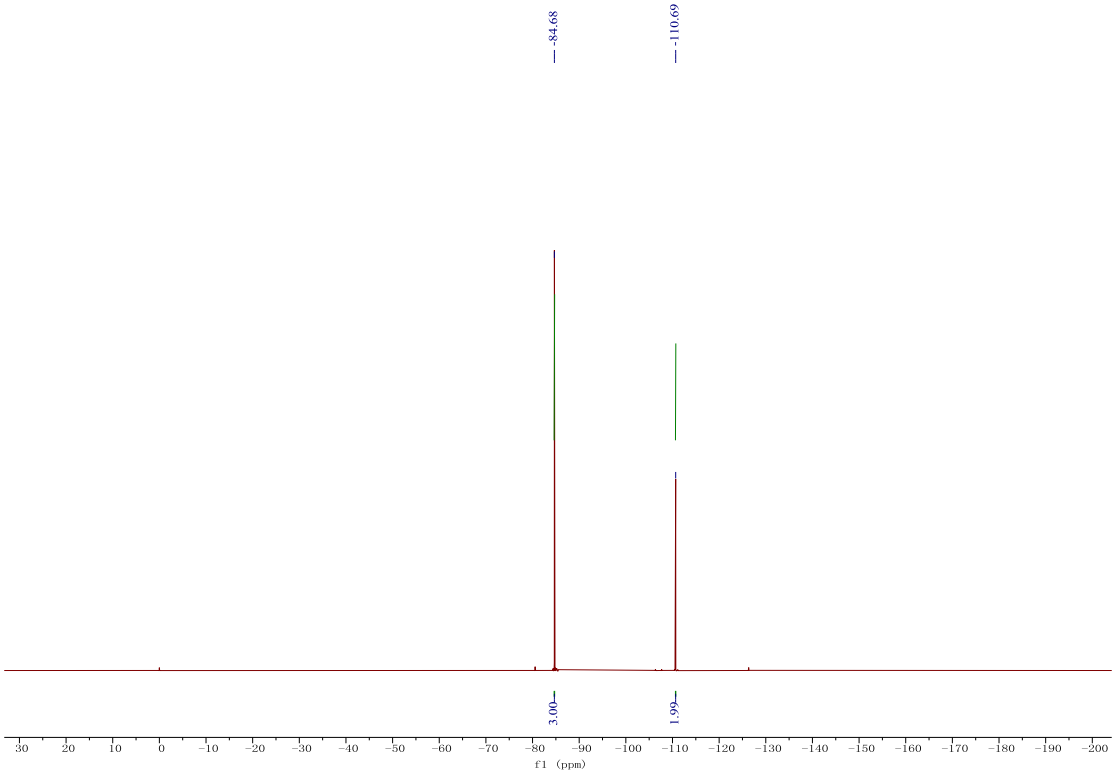

C_2F_5
S
2x ^{1}H NMR
(400 MHz, $CDCl_3$)

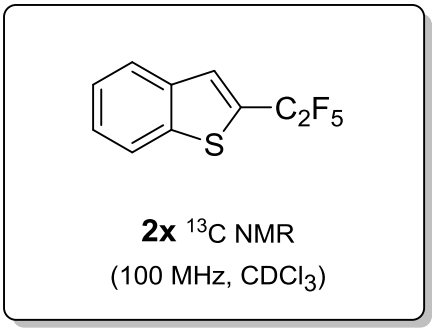

7.98
7.96
7.89
7.88
7.87
7.48
7.47
7.46
7.46
7.44
7.44
7.42
7.42
7.40
7.40
7.23
0.00
1.04
1.99
2.09
f1 (ppm)



2x ^{19}F NMR

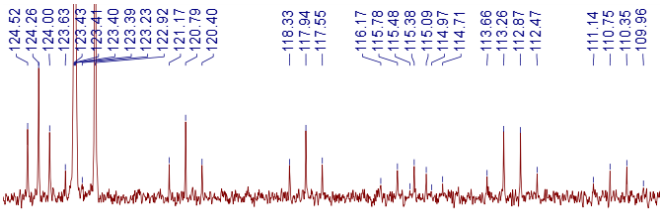
(376 MHz, $CDCl_3$)

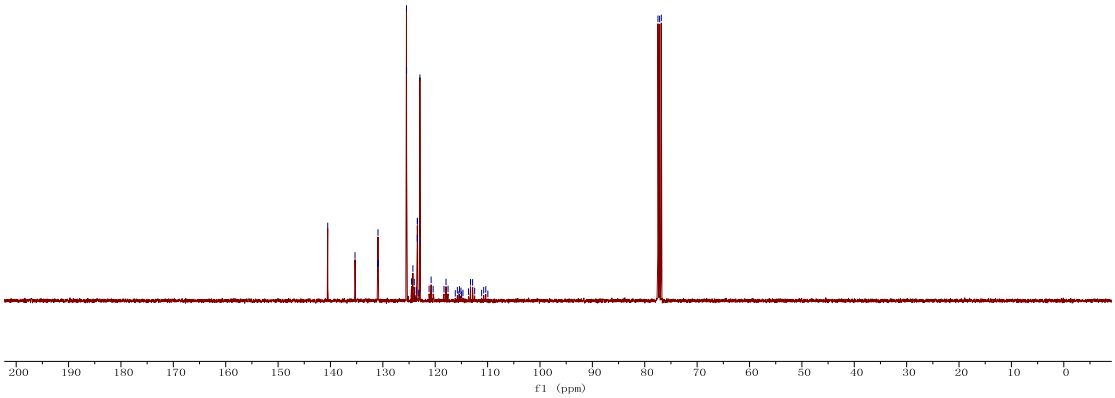


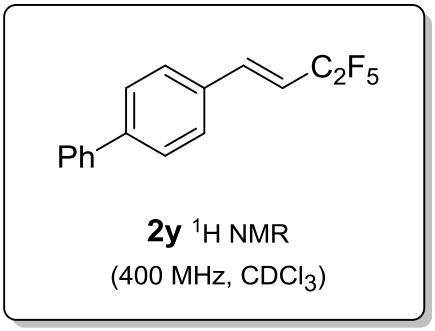


2x ^{13}C NMR

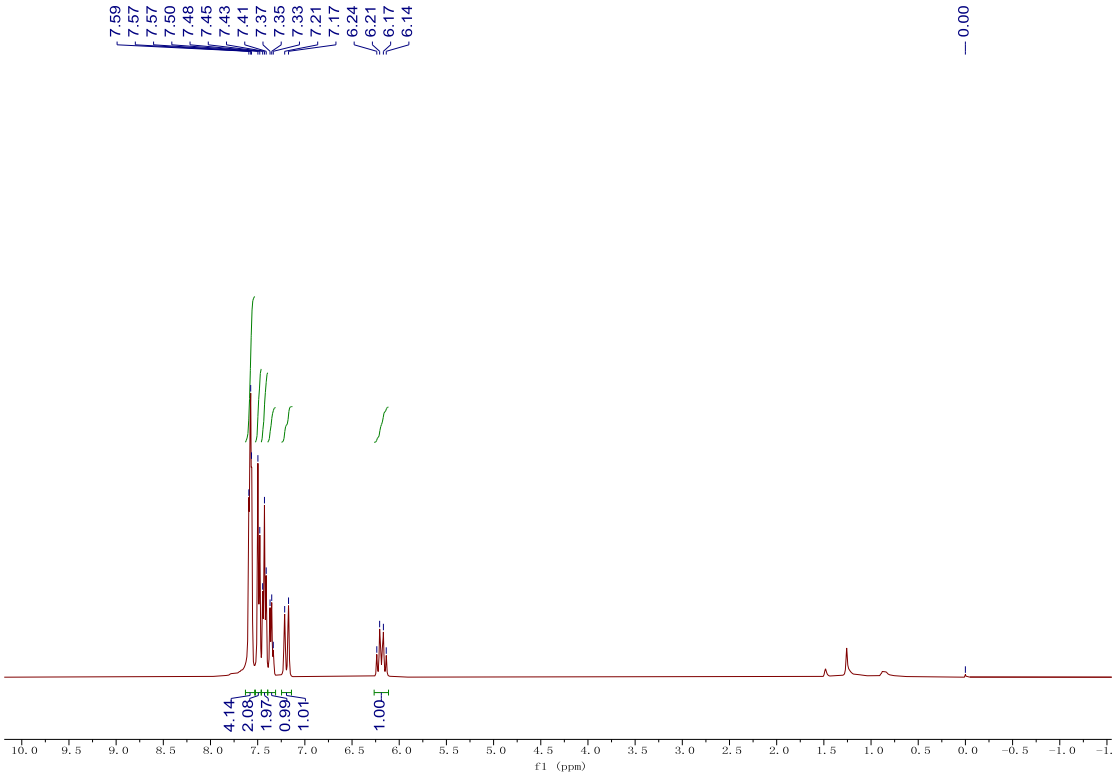
(100 MHz, $CDCl_3$)

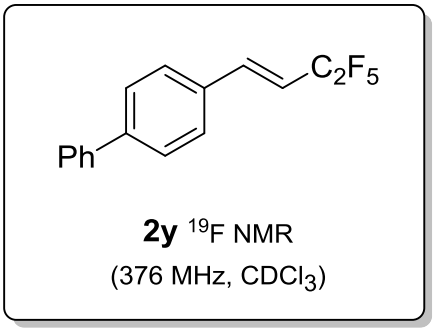






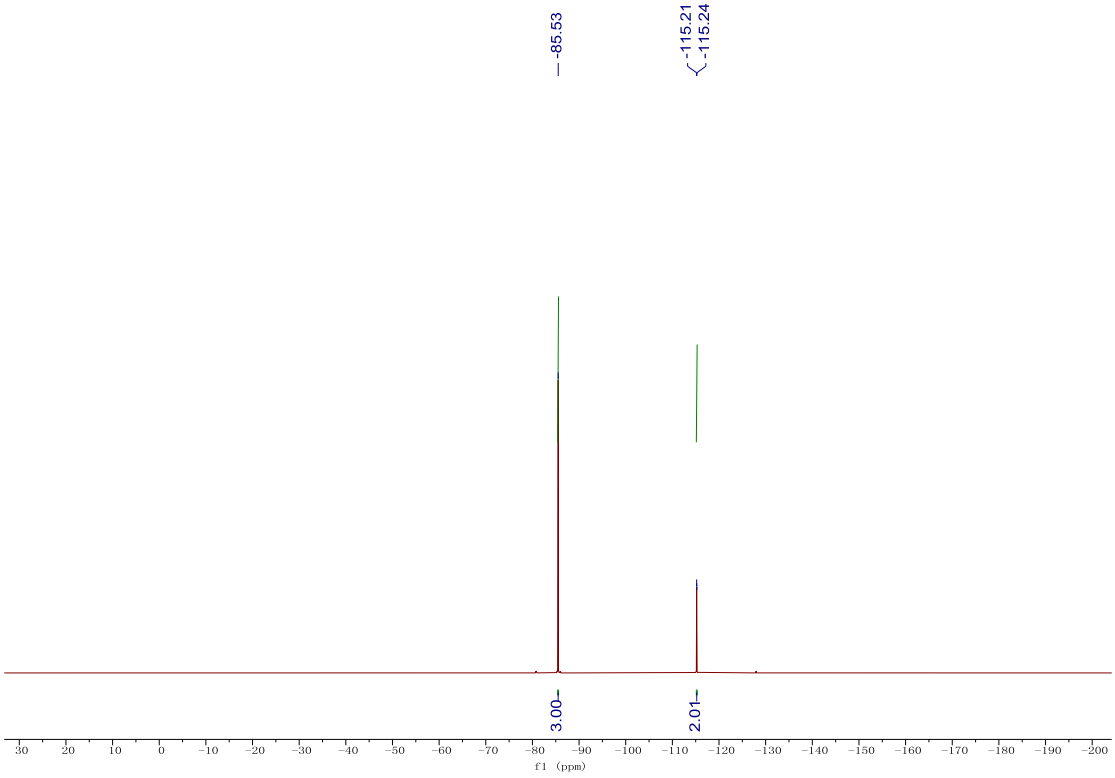
2y ^{1}H NMR
(400 MHz, $CDCl_3$)

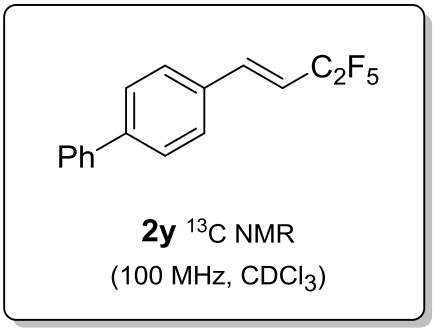




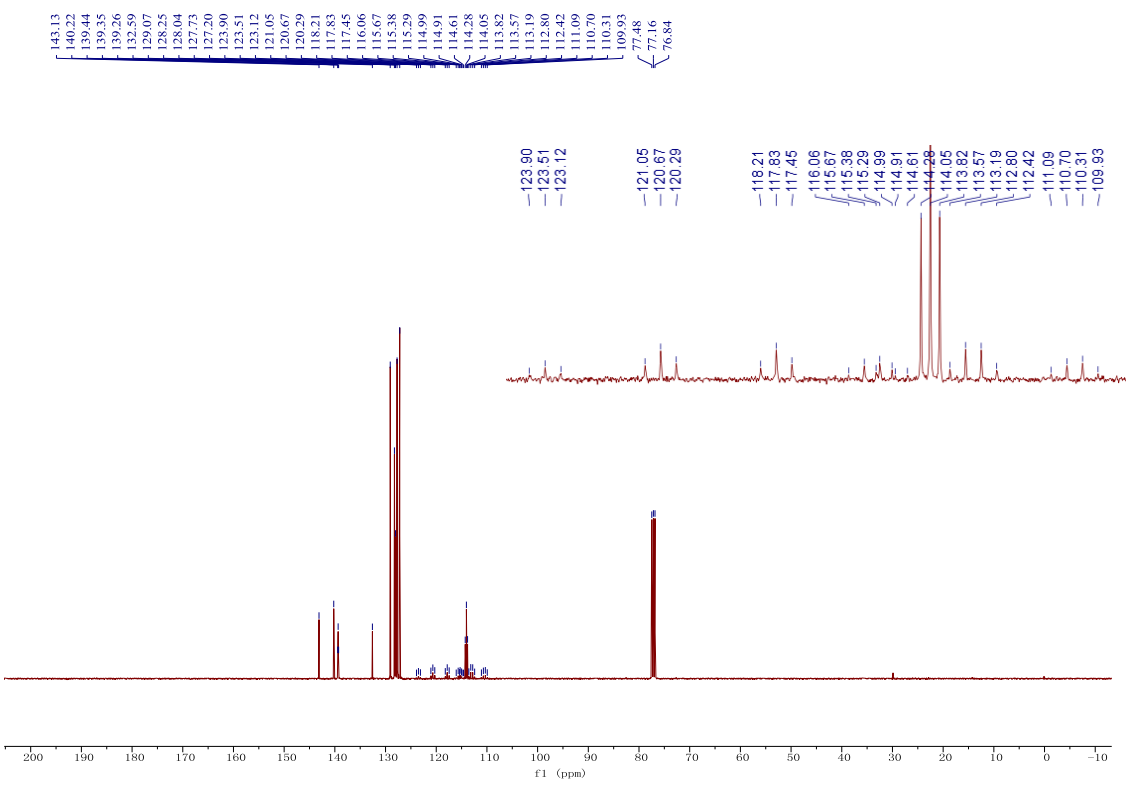
2y ^{19}F NMR

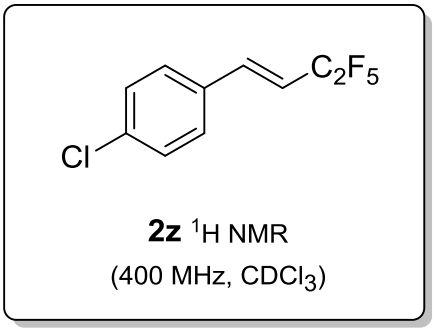
(376 MHz, $CDCl_3$)

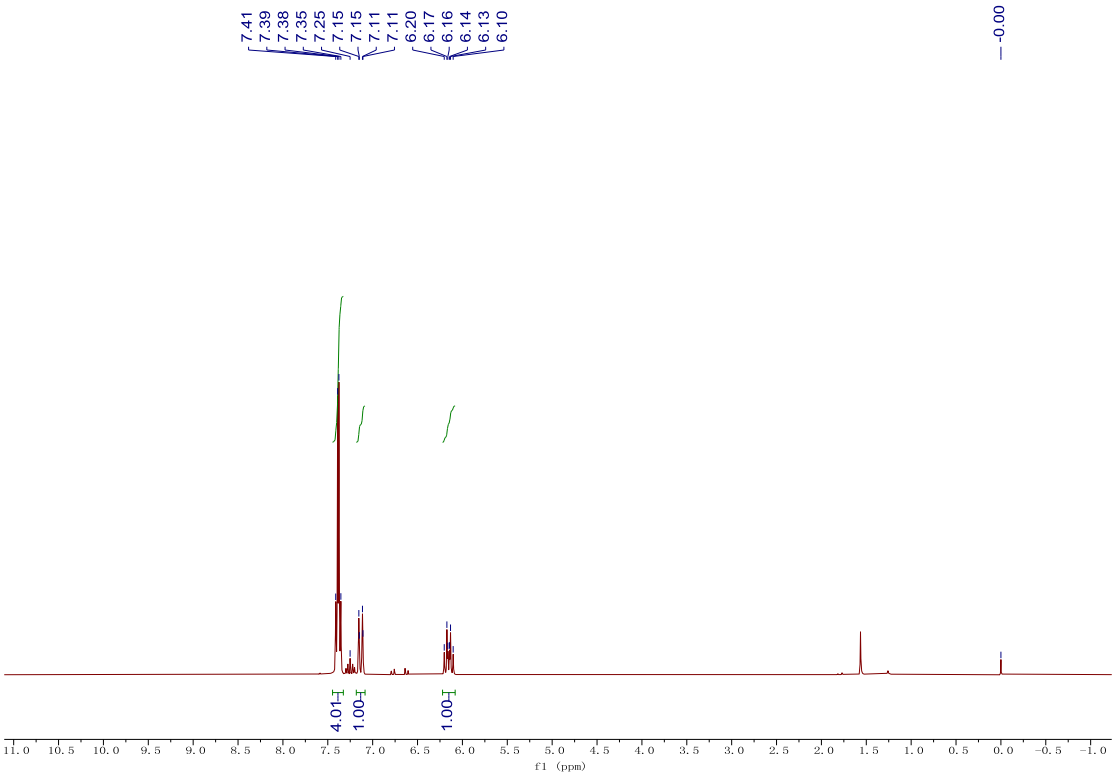


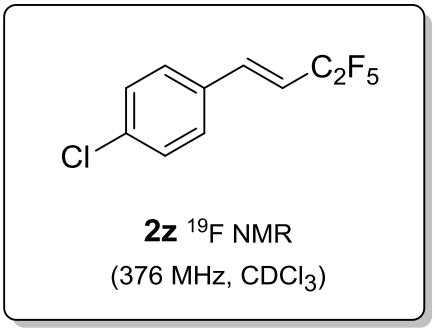


2y ^{13}C NMR
(100 MHz, $CDCl_3$)



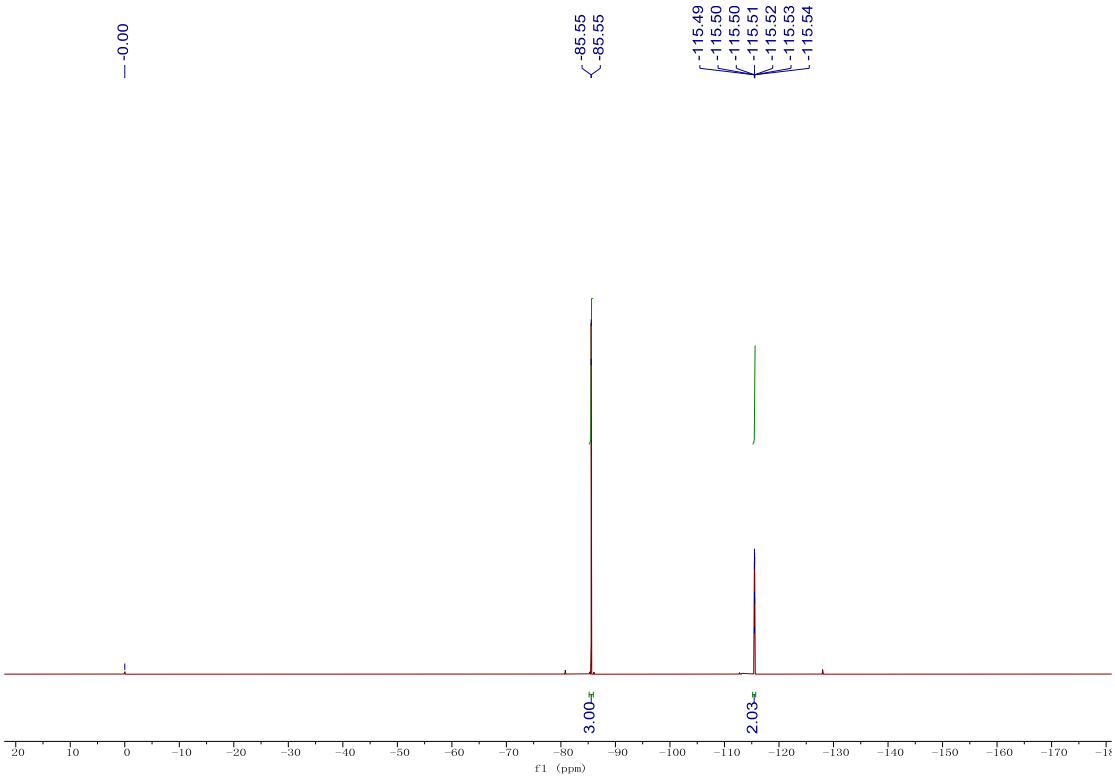

C2F5
Cl
2z 1H NMR
(400 MHz, CDCl3)

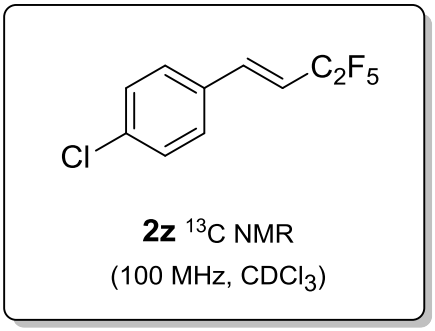

7.41
7.39
7.38
7.35
7.25
7.15
7.15
7.11
7.11
6.20
6.17
6.16
6.14
6.13
6.10
0.00
4.01
1.00
1.00
f1 (ppm)

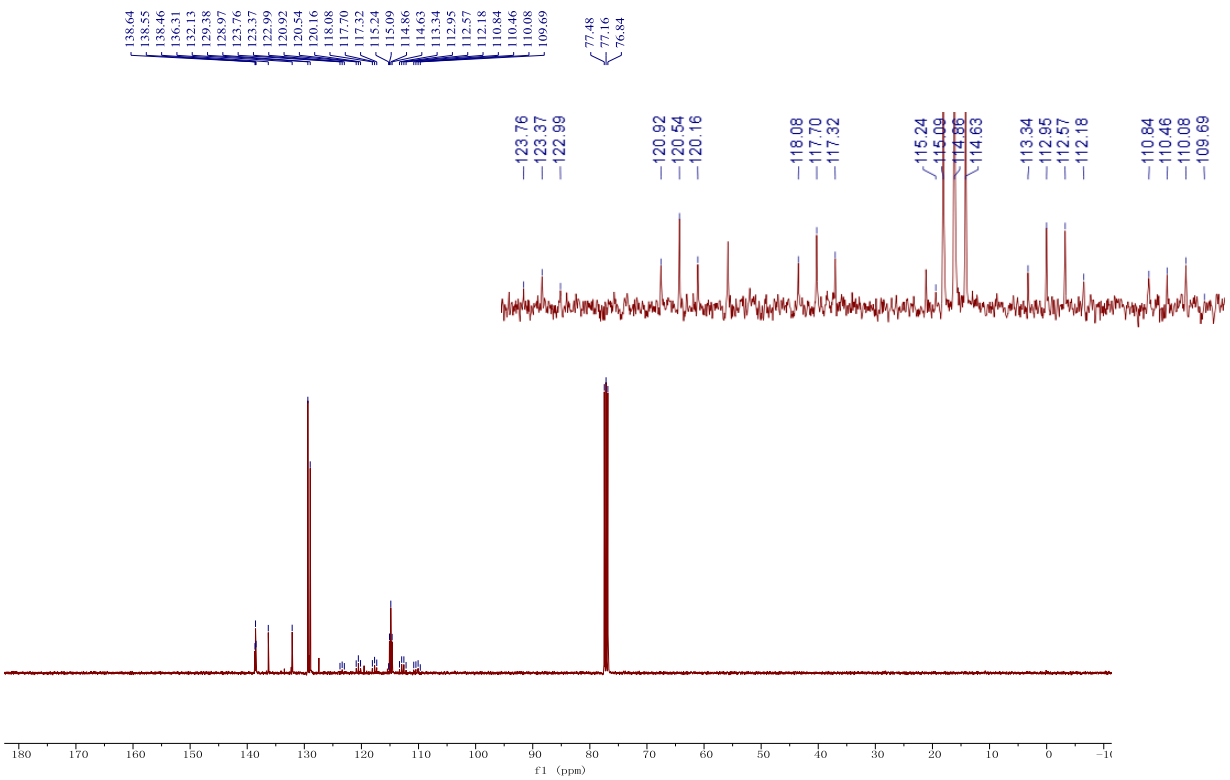


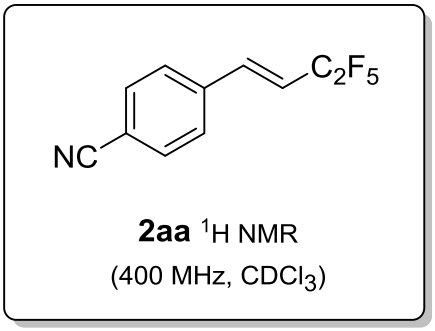
2z ^{19}F NMR

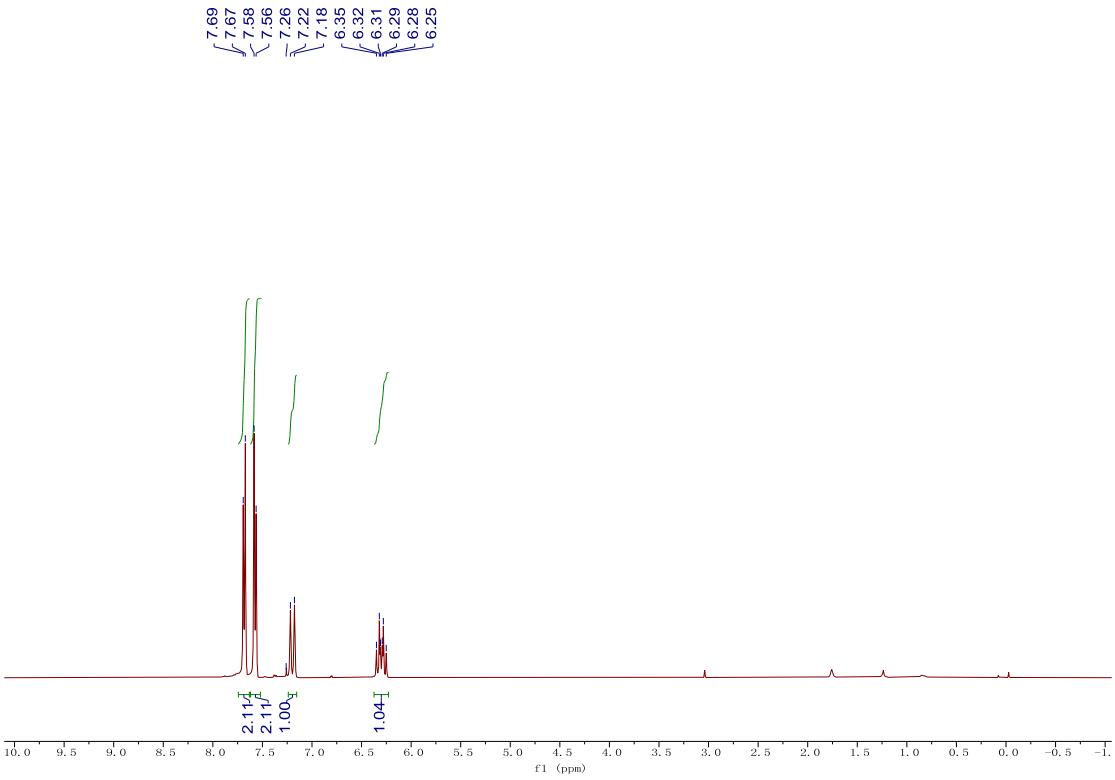
(376 MHz, $CDCl_3$)

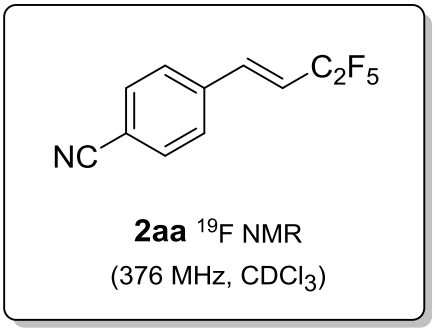



Cl
C_2F_5
2z ^{13}C NMR
(100 MHz, $CDCl_3$)


138.64
138.55
138.46
136.31
132.13
129.38
128.97
123.76
123.37
122.99
120.92
120.54
120.16
118.08
117.70
117.32
115.24
115.09
114.86
114.63
113.34
112.95
112.57
112.18
110.84
110.46
110.08
109.69
77.48
77.16
76.84
123.76
123.37
122.99
120.92
120.54
120.16
118.08
117.70
117.32
115.24
115.09
114.86
114.63
113.34
112.95
112.57
112.18
110.84
110.46
110.08
109.69
f1 (ppm)

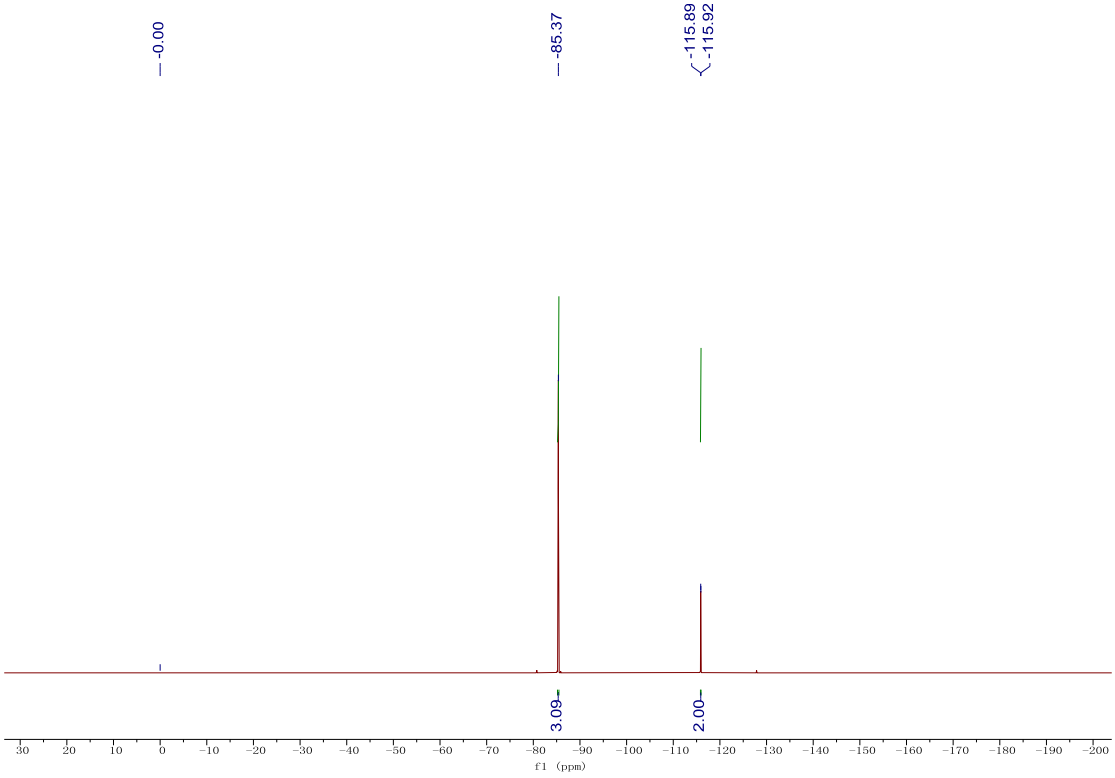

NC
C_2F_5
2aa ^{1}H NMR
(400 MHz, $CDCl_3$)

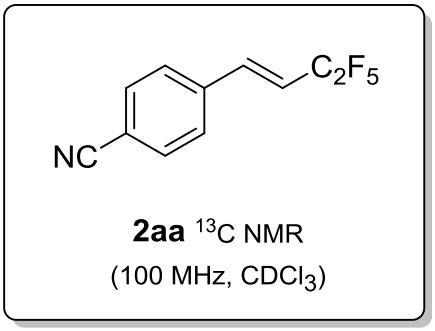

7.69
7.67
7.58
7.56
7.26
7.22
7.18
6.35
6.32
6.31
6.29
6.28
6.25
2.11
2.11
1.00
1.04
f1 (ppm)

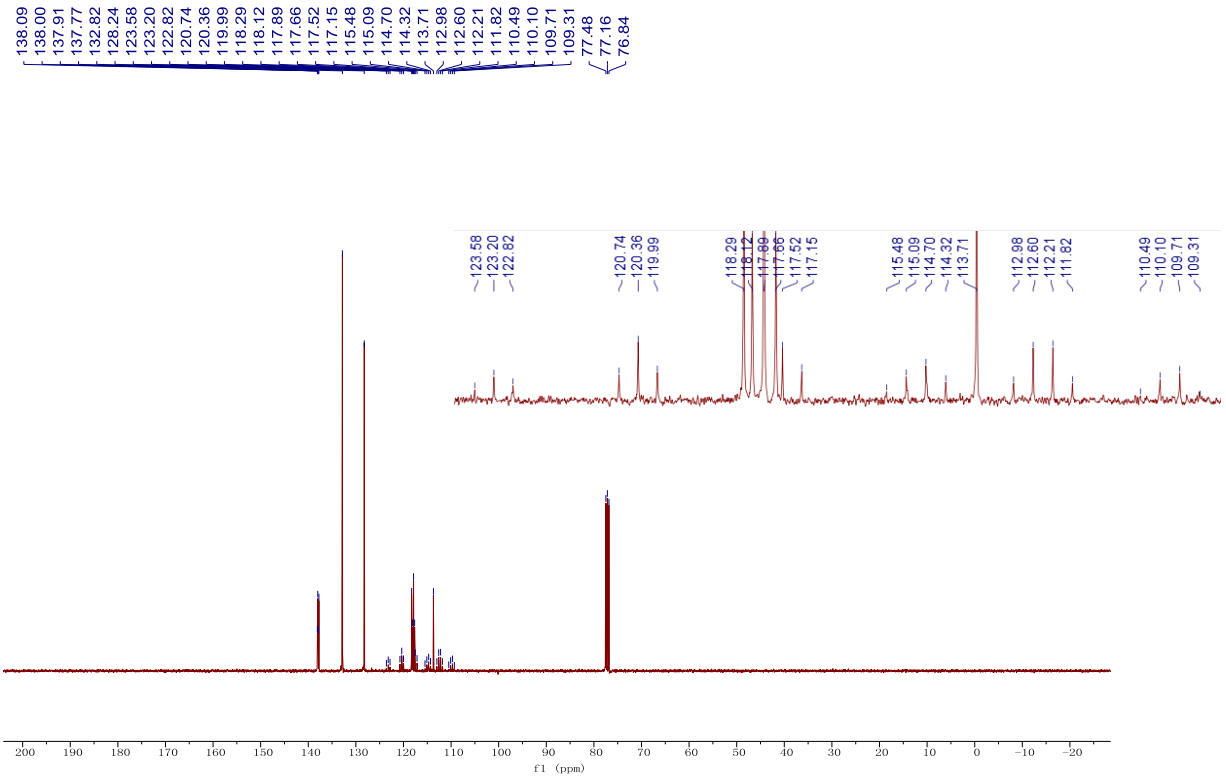


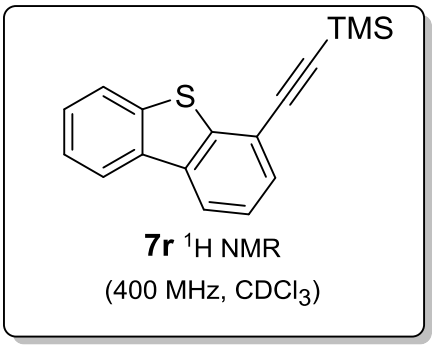
2aa ^{19}F NMR

(376 MHz, $CDCl_3$)



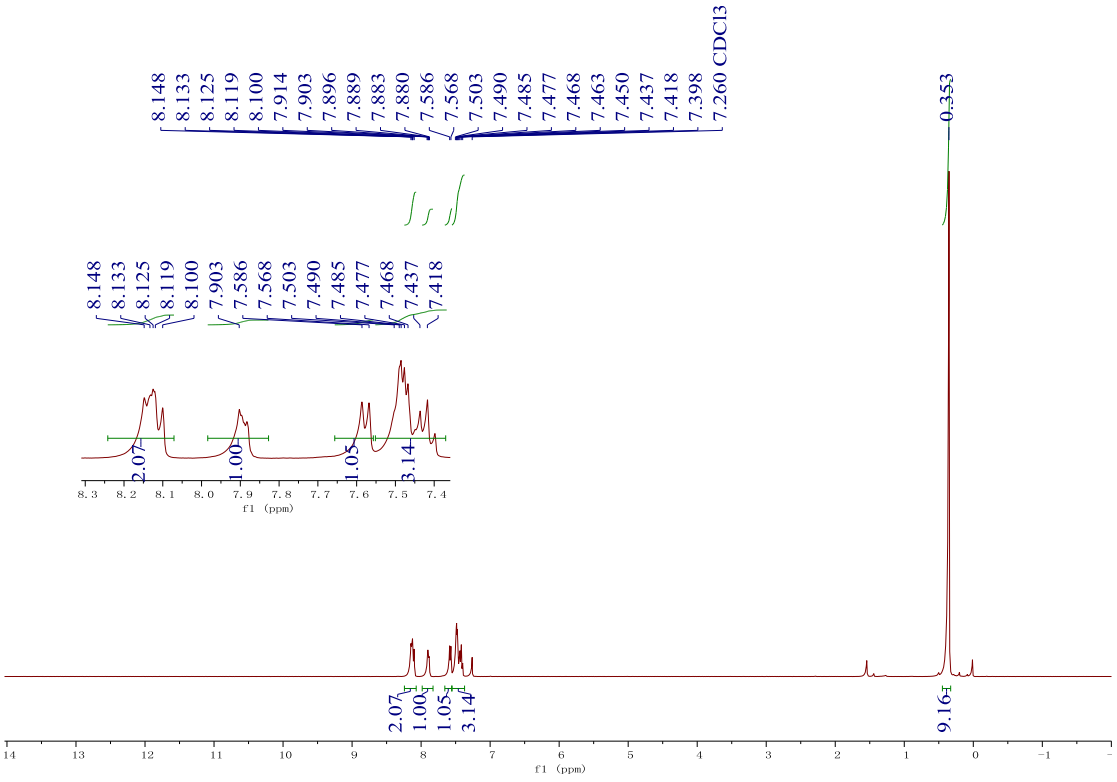

NC
C_2F_5
2aa ^{13}C NMR
(100 MHz, $CDCl_3$)

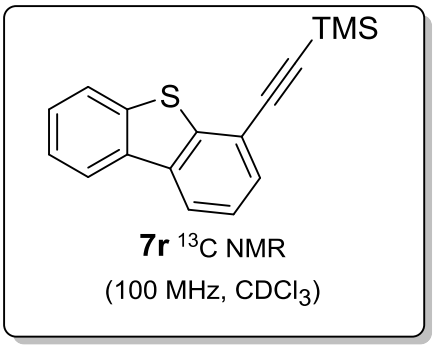

138.09
138.00
137.91
137.77
132.82
128.24
123.58
123.20
122.82
120.74
120.36
119.99
118.29
118.12
117.89
117.66
117.52
117.15
115.48
115.09
114.70
114.32
113.71
112.98
112.60
112.21
111.82
110.49
110.10
109.71
109.31
77.48
77.16
76.84
f1 (ppm)

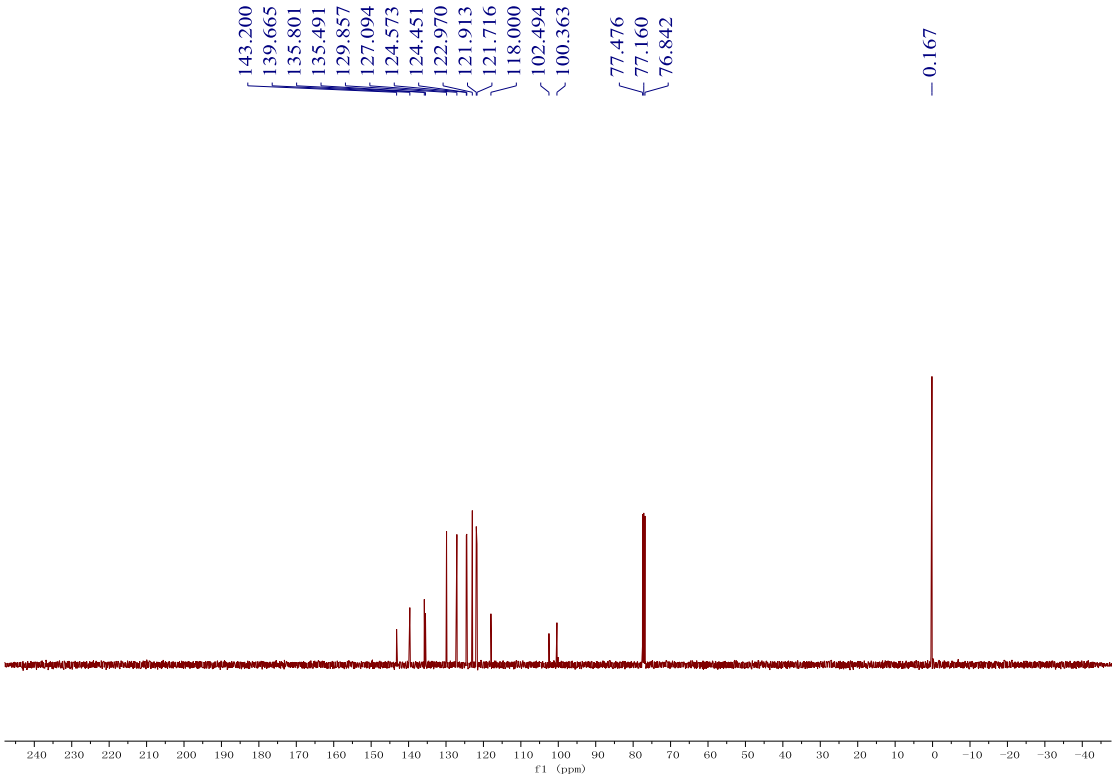


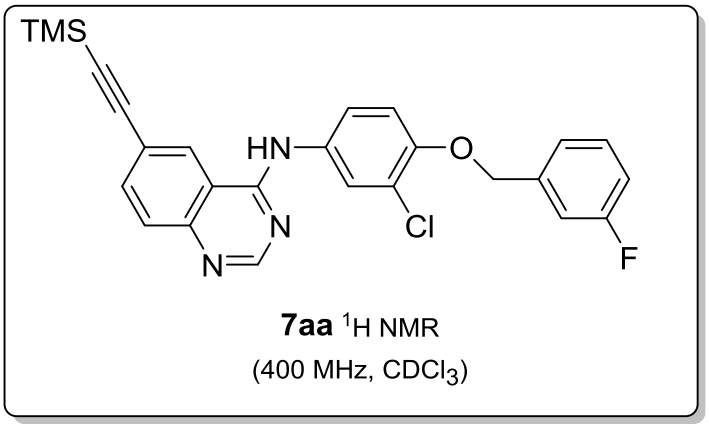
7r ^{1}H NMR

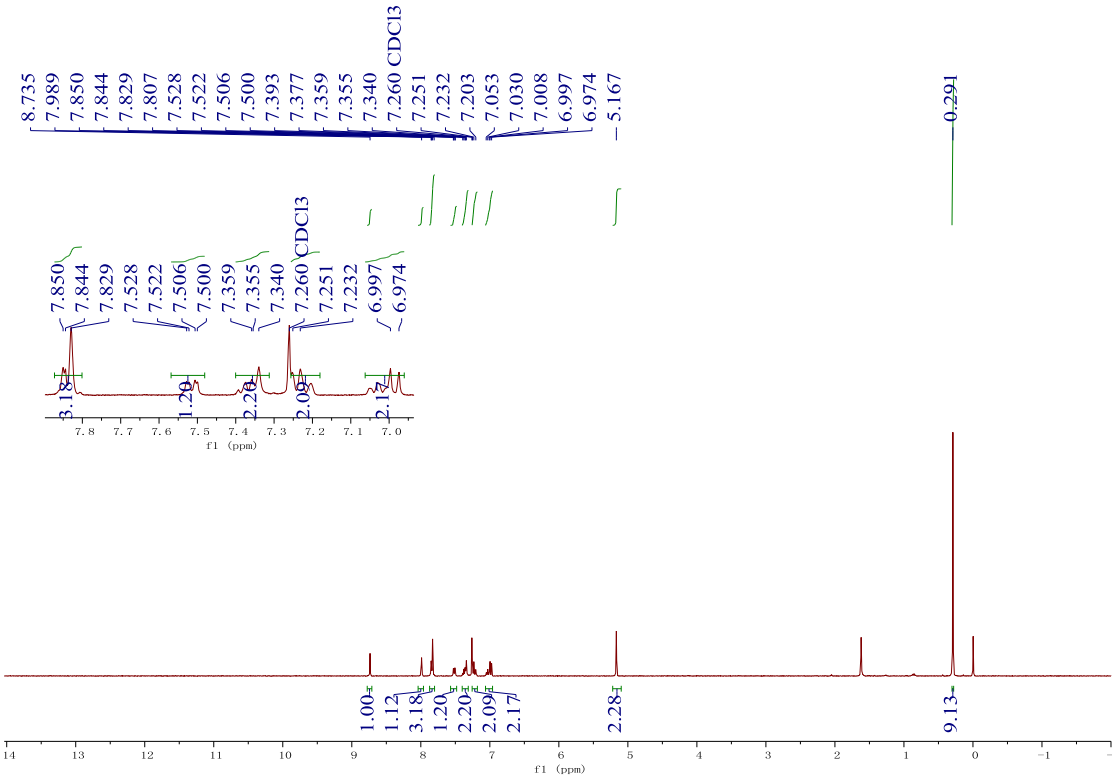
(400 MHz, $CDCl_3$)

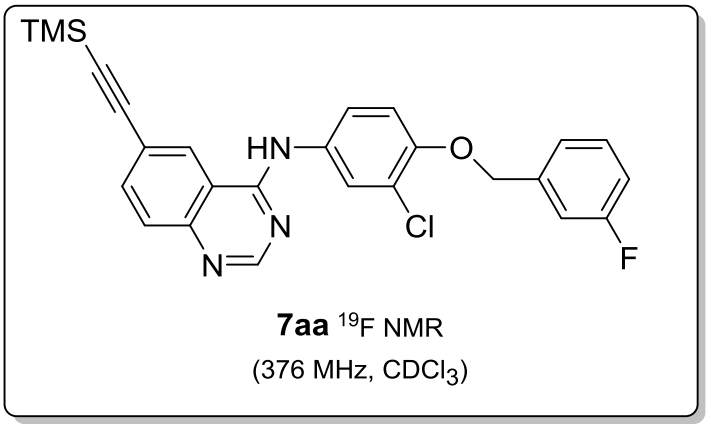



TMS
S
7r 13C NMR
(100 MHz, CDCl3)


143.200
139.665
135.801
135.491
129.857
127.094
124.573
124.451
122.970
121.913
121.716
118.000
102.494
100.363
77.476
77.160
76.842
0.167
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40
f1 (ppm)

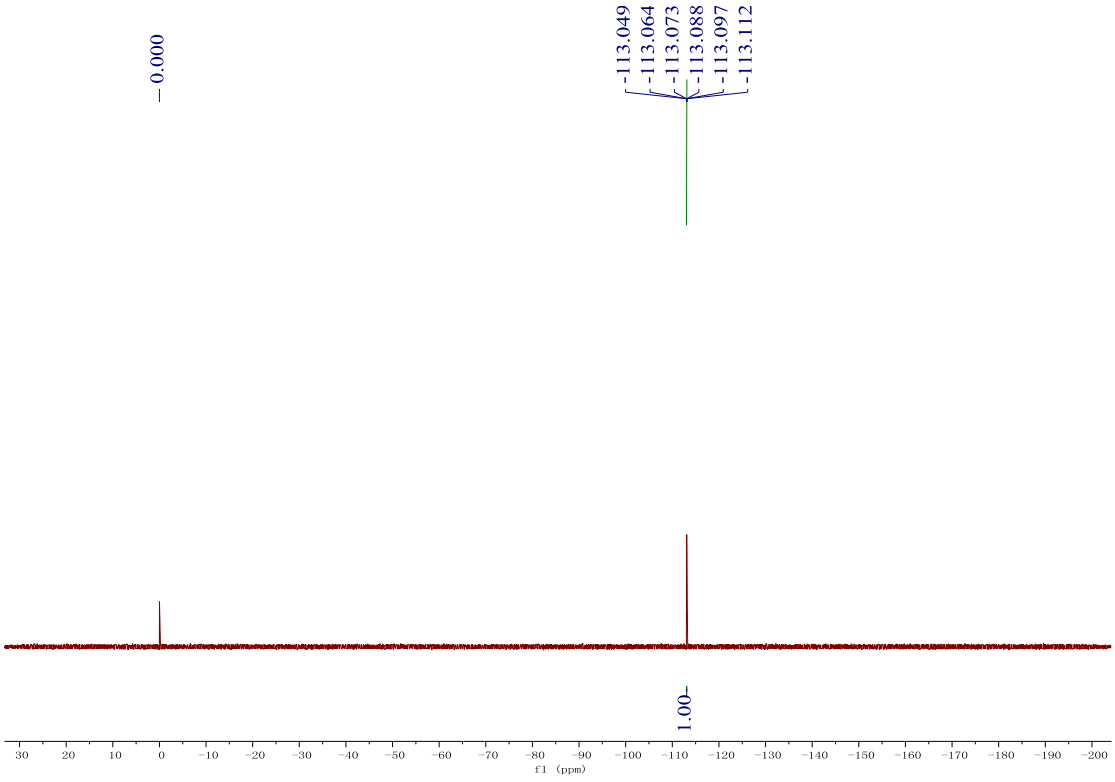

TMS
HN
O
N
Cl
N
F
7aa 1H NMR
(400 MHz, CDCl3)

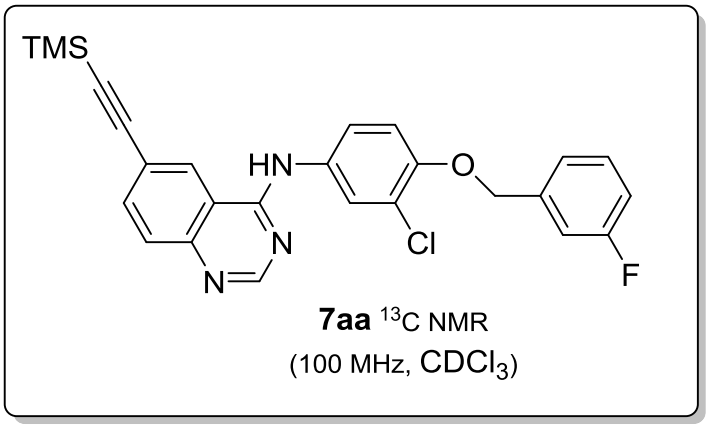

8.735
7.989
7.850
7.844
7.829
7.807
7.528
7.522
7.506
7.500
7.393
7.377
7.359
7.355
7.340
7.260 CDCl3
7.251
7.232
7.203
7.053
7.030
7.008
6.997
6.974
5.167
0.291
f1 (ppm)
1.00
1.12
3.18
1.20
2.20
2.09
2.17
2.28
9.13

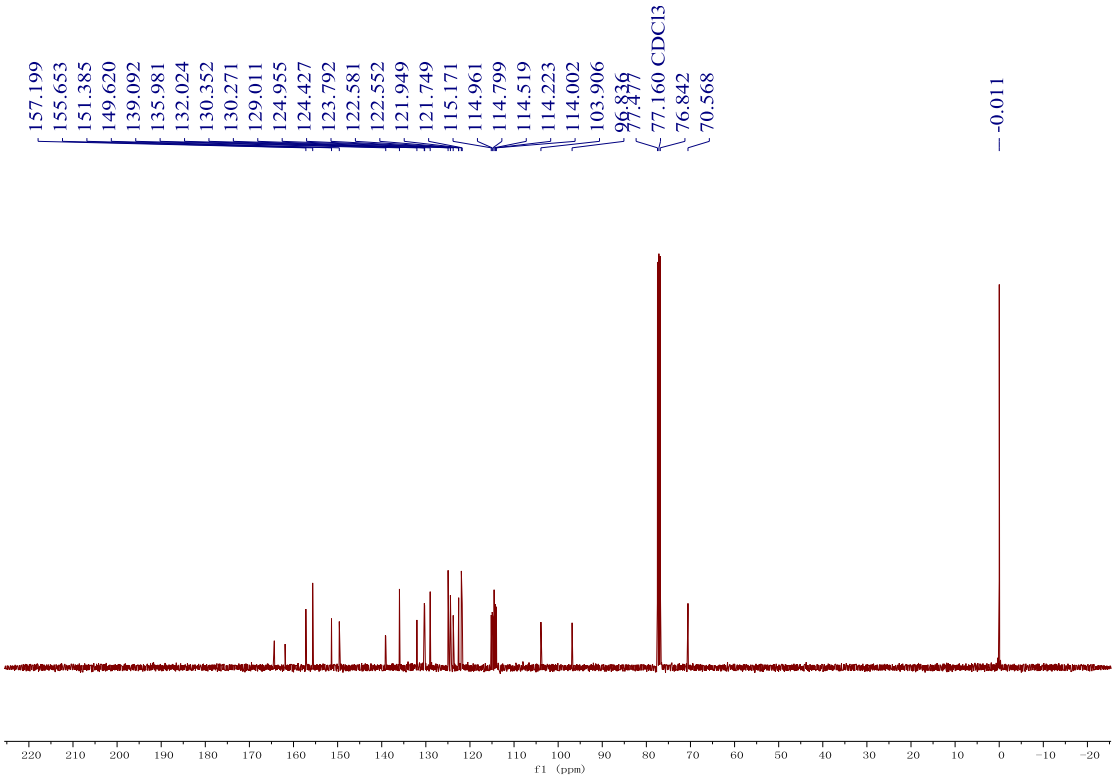


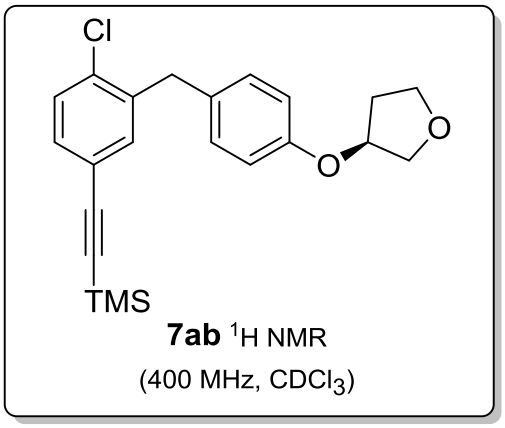
7aa ^{19}F NMR

(376 MHz, $CDCl_3$)



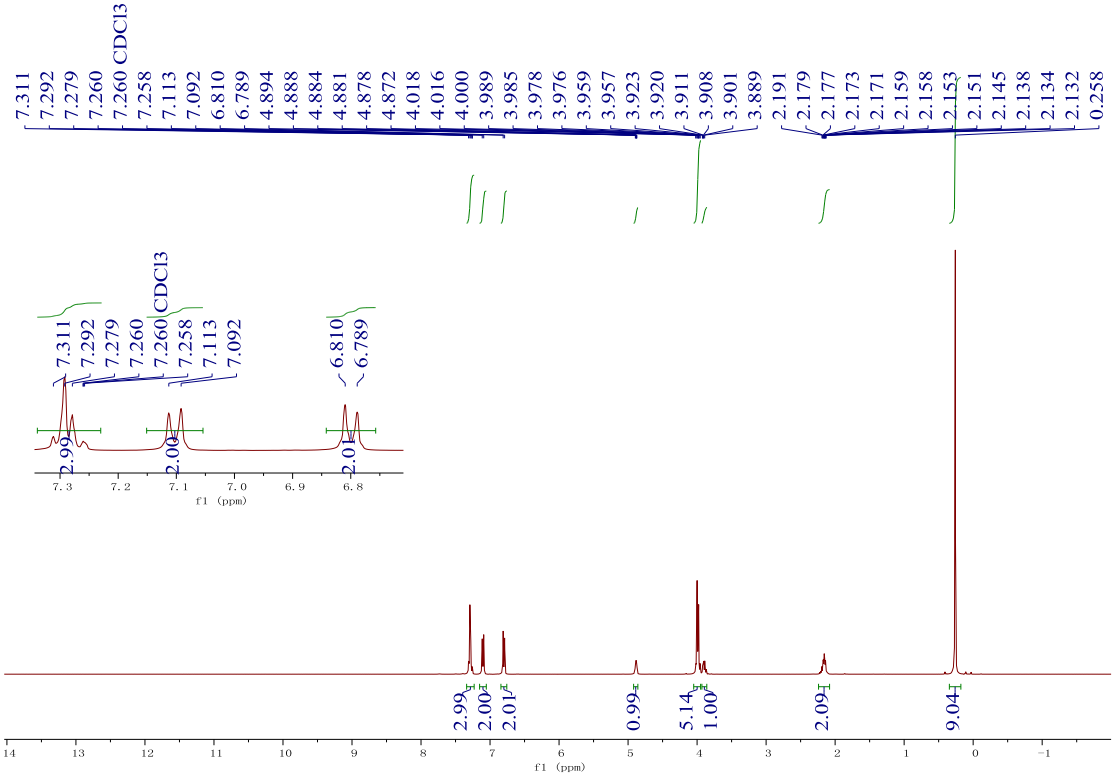

TMS
HN
O
N
N
Cl
F
7aa 13C NMR
(100 MHz, CDCl3)

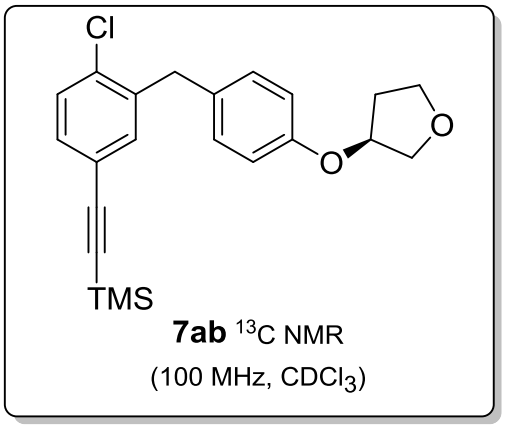

157.199
155.653
151.385
149.620
139.092
135.981
132.024
130.352
130.271
129.011
124.955
124.427
123.792
122.581
122.552
121.949
121.749
115.171
114.961
114.799
114.519
114.223
114.002
103.906
96.836
77.477
77.160 CDCl3
76.842
70.568
-0.011
f1 (ppm)

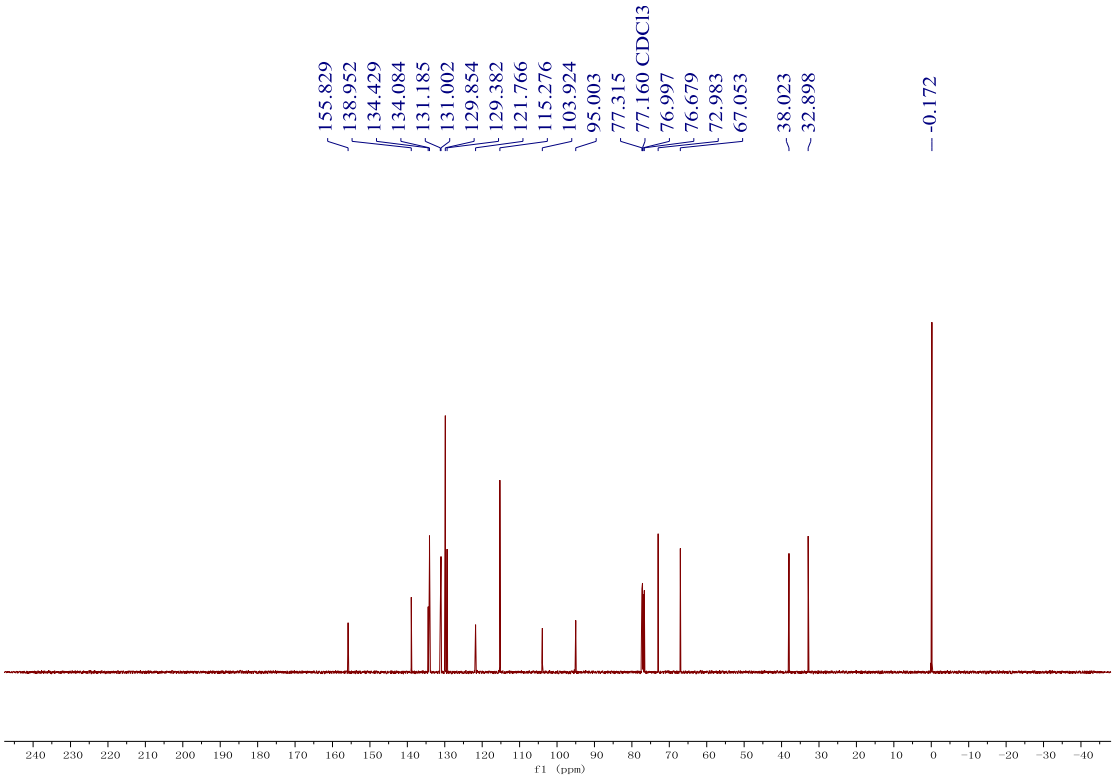


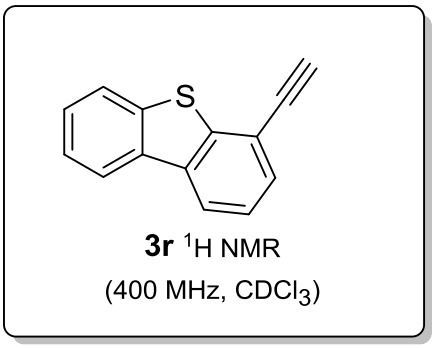
7ab ^{1}H NMR

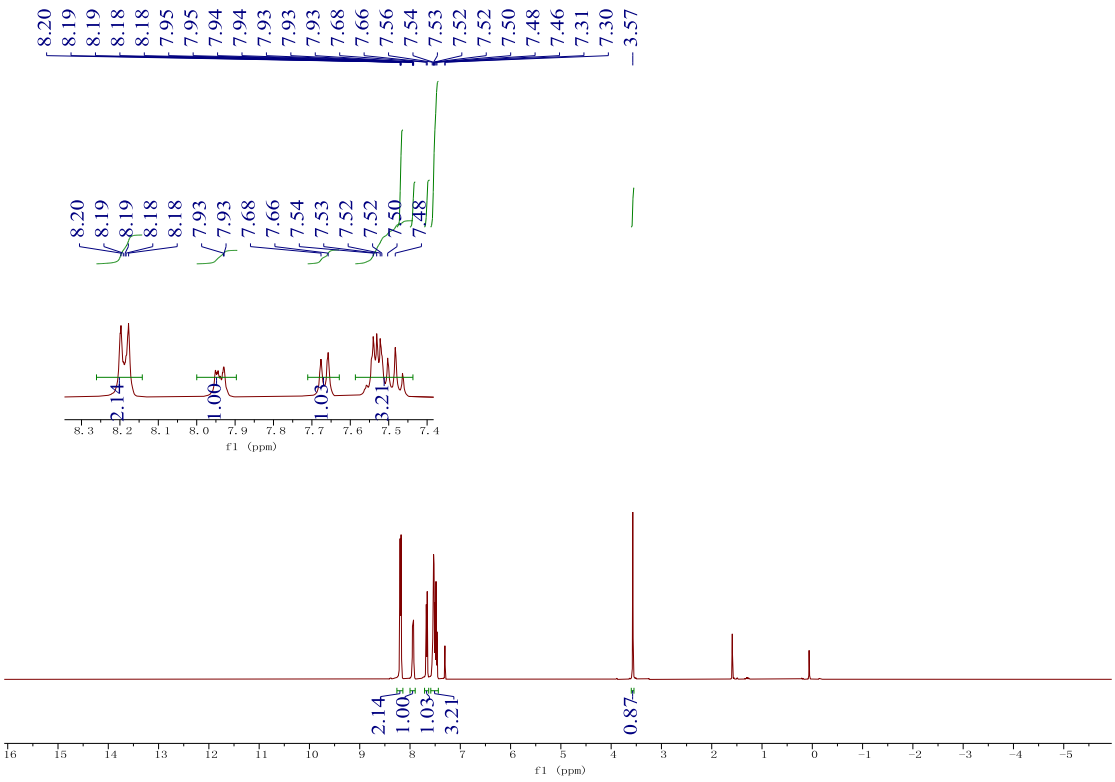
(400 MHz, $CDCl_3$)

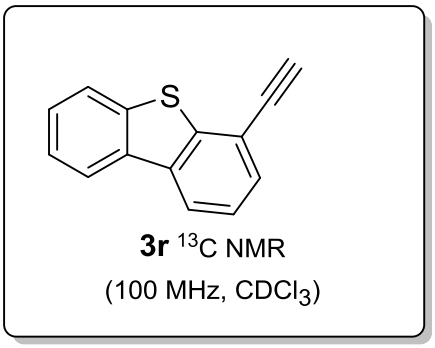


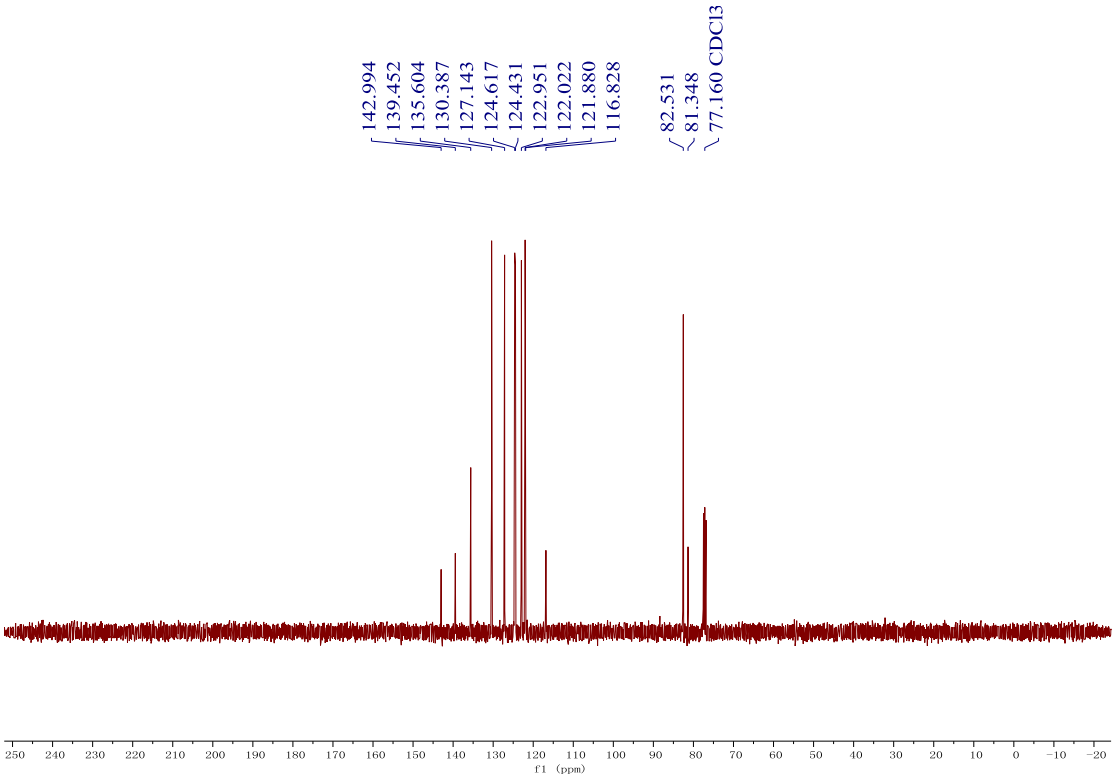

Cl
O
O
TMS
7ab ^{13}C NMR
(100 MHz, $CDCl_3$)

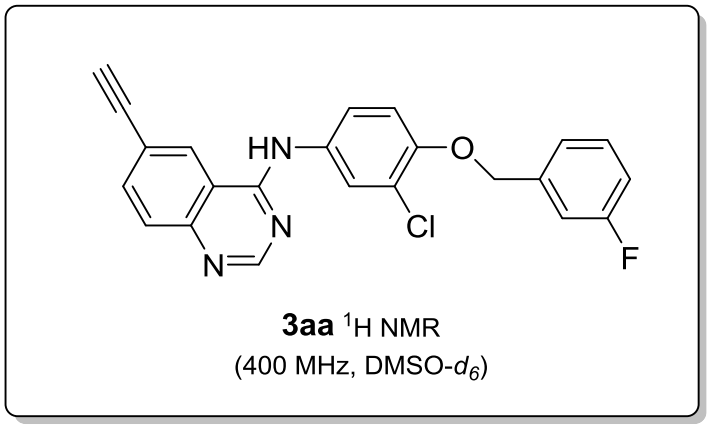

155.829
138.952
134.429
134.084
131.185
131.002
129.854
129.382
121.766
115.276
103.924
95.003
77.315
77.160 CDCl3
76.997
76.679
72.983
67.053
38.023
32.898
-0.172
f1 (ppm)

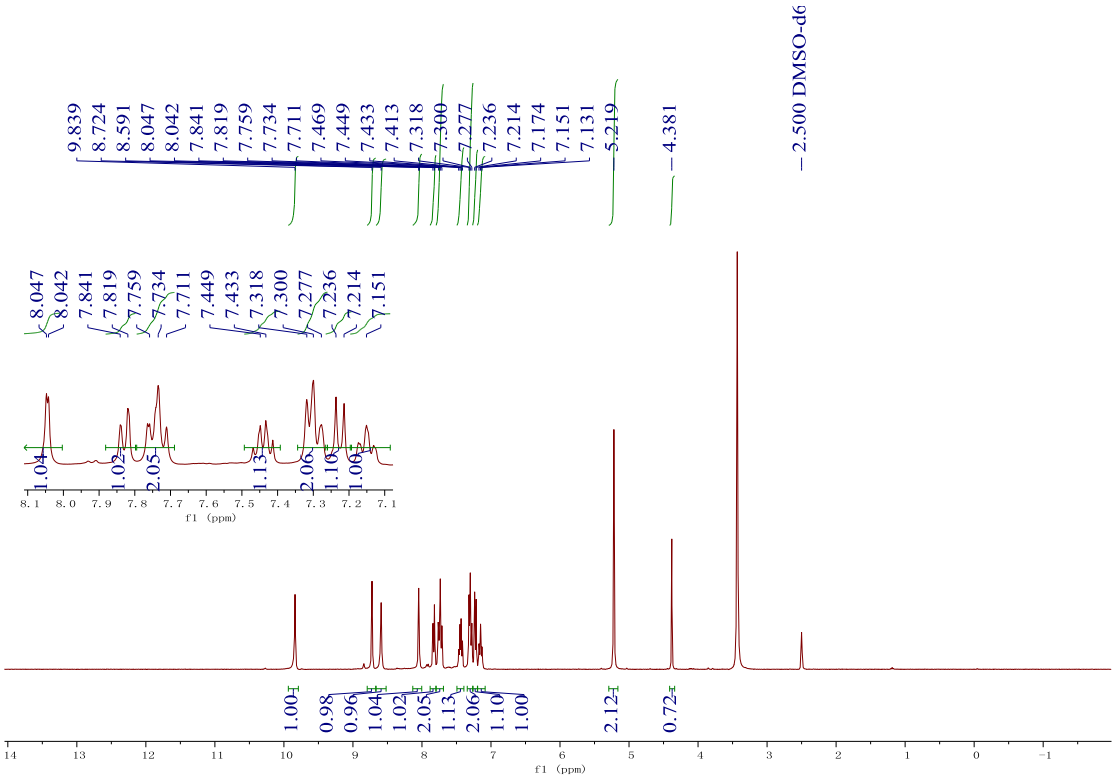

S
3r 1H NMR
(400 MHz, CDCl3)

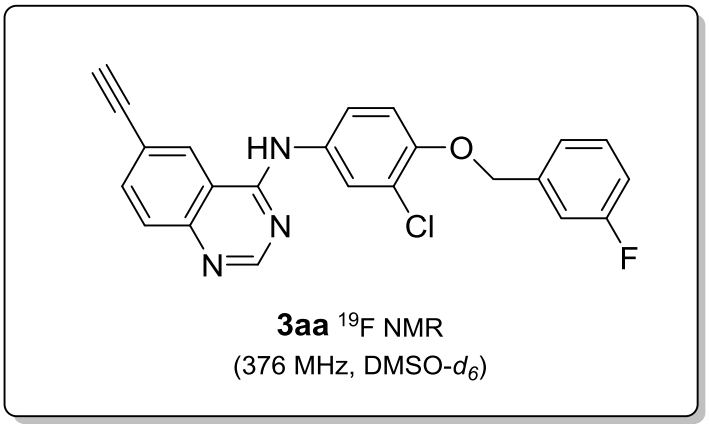

8.20
8.19
8.19
8.18
8.18
7.95
7.95
7.94
7.94
7.93
7.93
7.93
7.68
7.66
7.56
7.54
7.53
7.52
7.52
7.50
7.48
7.46
7.31
7.30
3.57
2.14
1.00
1.03
3.21
0.87
f1 (ppm)


S
3r 13C NMR
(100 MHz, CDCl3)

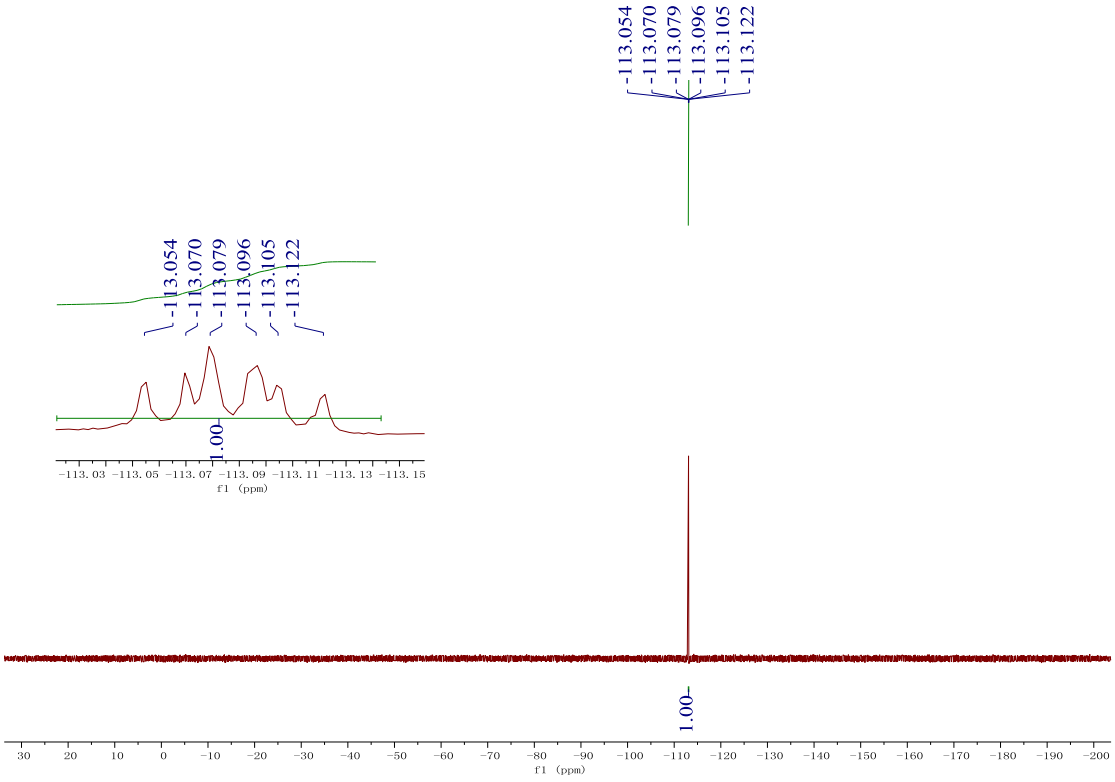

142.994
139.452
135.604
130.387
127.143
124.617
124.431
122.951
122.022
121.880
116.828
82.531
81.348
77.160 CDCl3
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20
f1 (ppm)

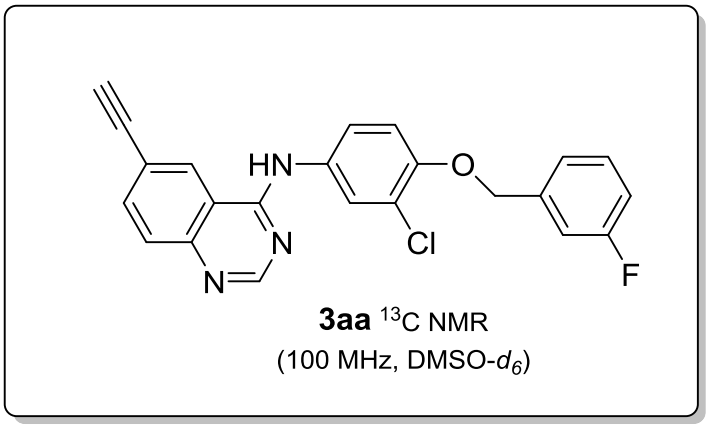

HN
O
N
N
Cl
F
3aa 1H NMR
(400 MHz, DMSO-d6)


9.839
8.724
8.591
8.047
8.042
7.841
7.819
7.759
7.734
7.711
7.469
7.449
7.433
7.413
7.318
7.300
7.277
7.236
7.214
7.174
7.151
7.131
5.219
4.381
2.500 DMSO-d6
f1 (ppm)

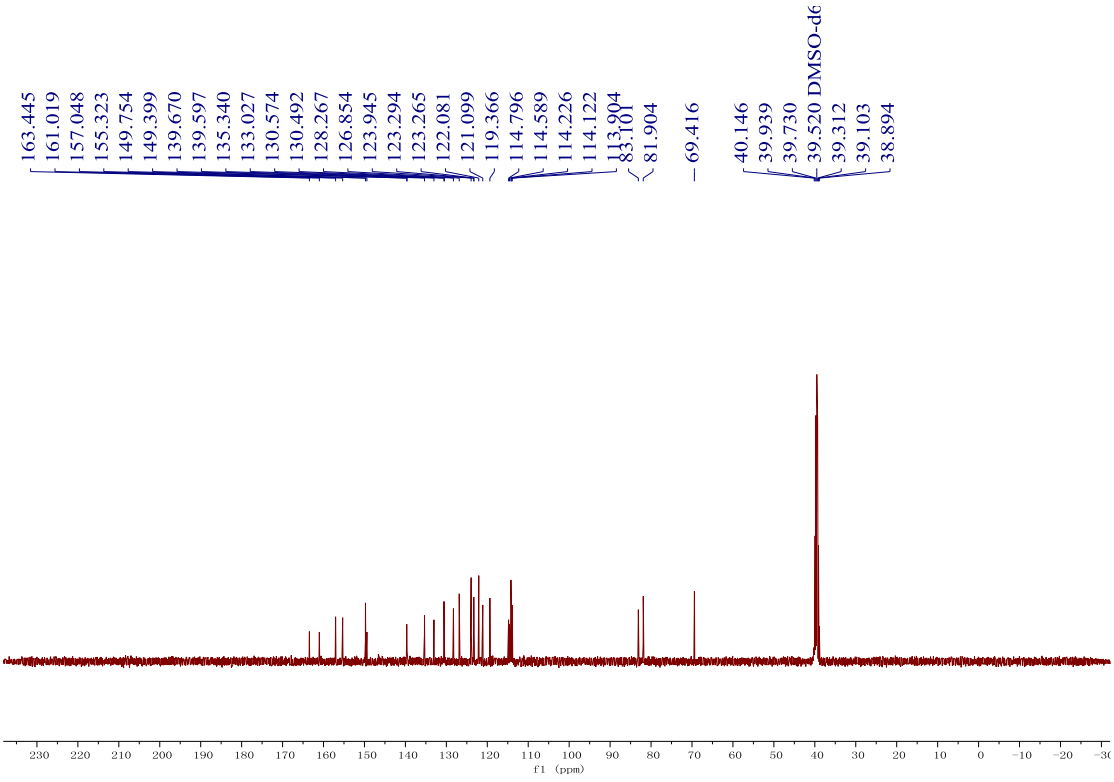


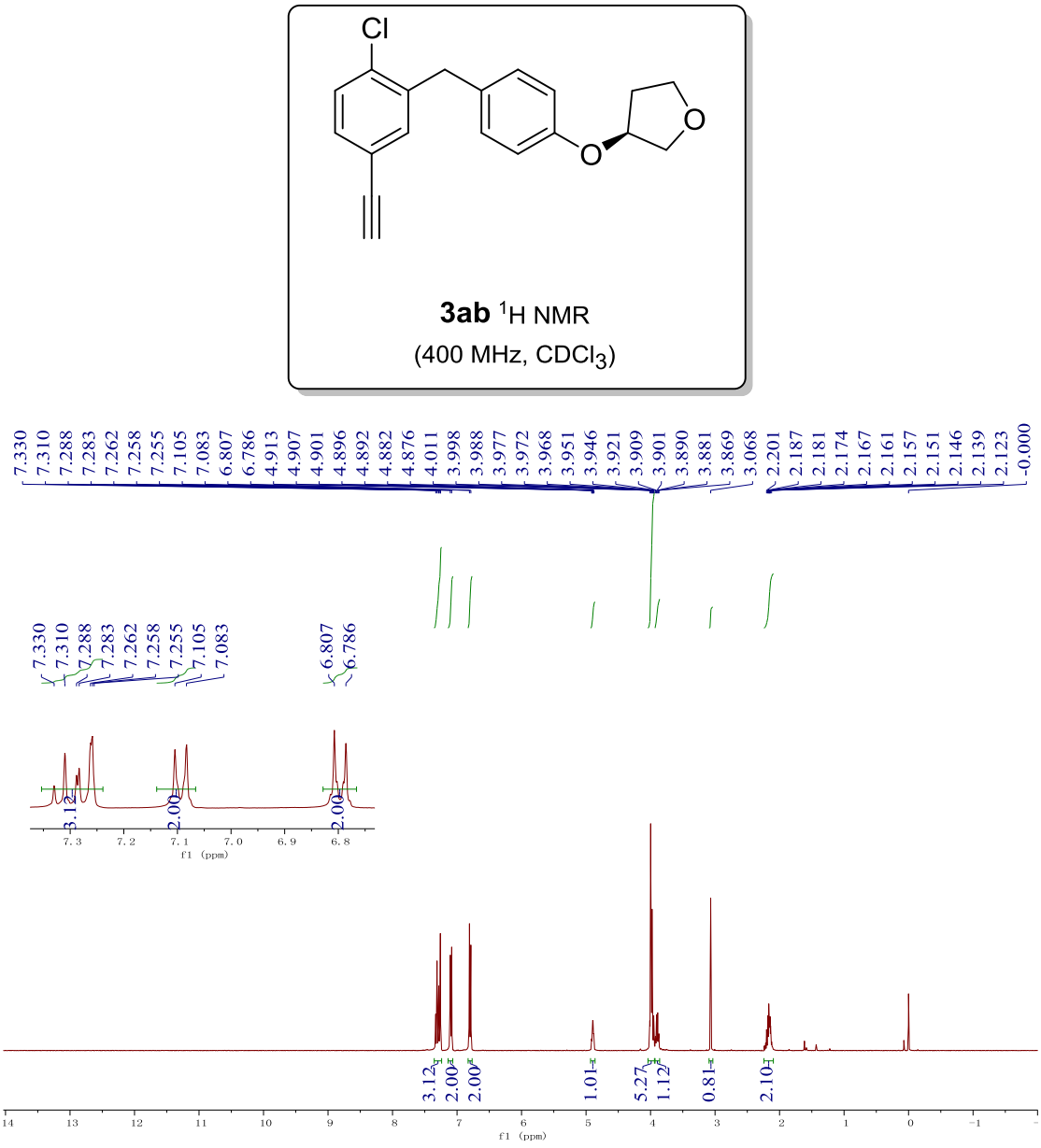
3aa ^{19}F NMR
(376 MHz, DMSO-d_6)

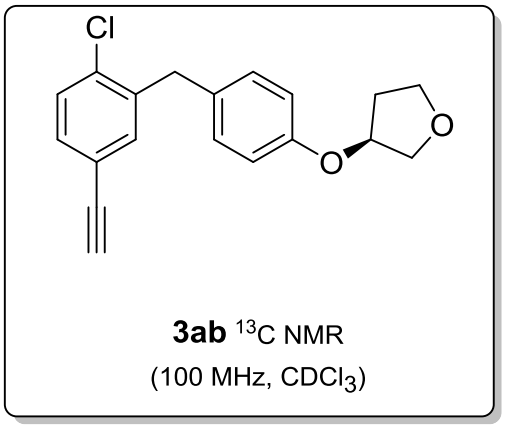




3aa ^{13}C NMR
(100 MHz, DMSO-d_6)

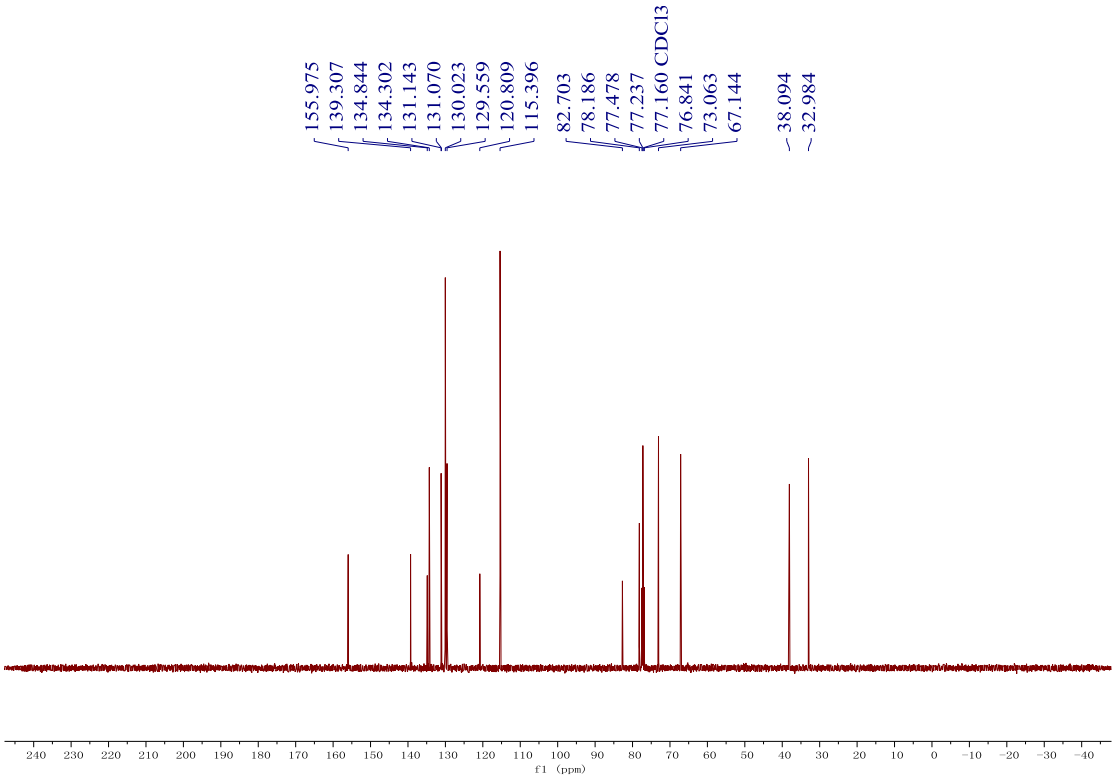


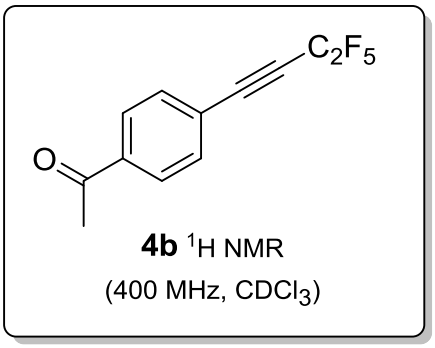

Cl
O
O
3ab 1H NMR
(400 MHz, CDCl3)
7.330
7.310
7.288
7.283
7.262
7.258
7.255
7.105
7.083
6.807
6.786
4.913
4.907
4.901
4.896
4.892
4.882
4.876
4.011
3.998
3.988
3.977
3.972
3.968
3.951
3.946
3.921
3.909
3.901
3.890
3.881
3.869
3.068
2.201
2.187
2.181
2.174
2.167
2.161
2.157
2.151
2.146
2.139
2.123
-0.000
3.12
2.00
2.00
1.01
5.27
1.12
0.81
2.10
f1 (ppm)



3ab ^{13}C NMR

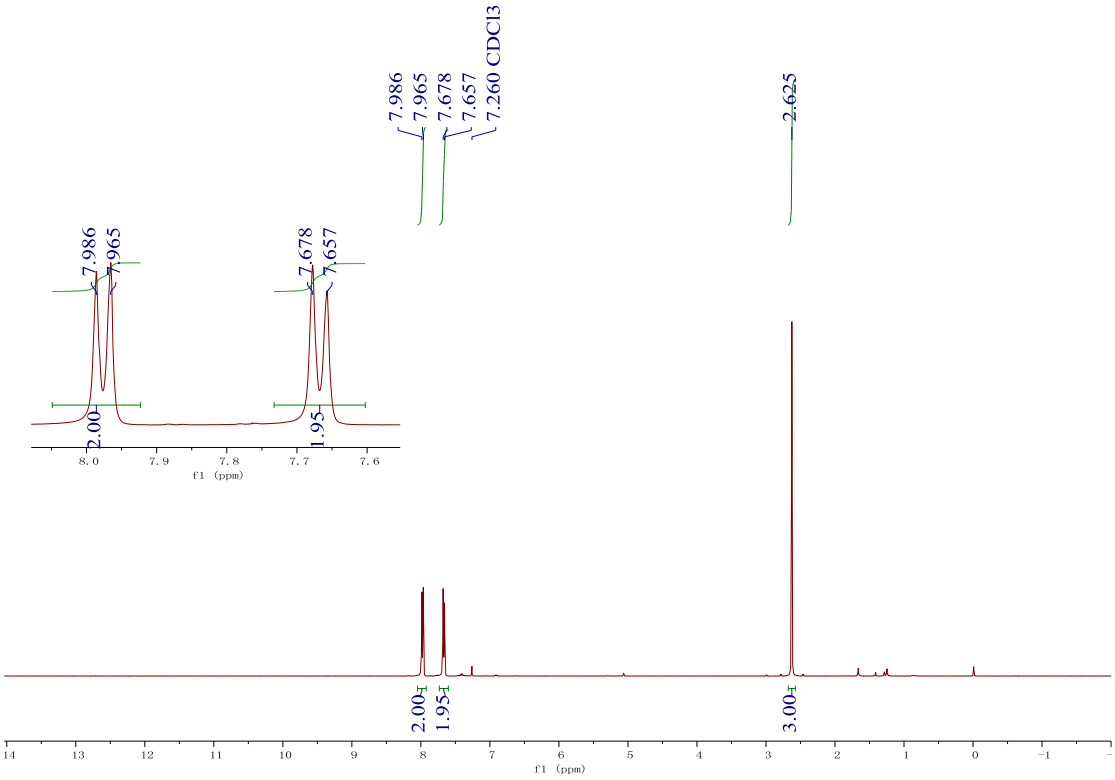
(100 MHz, $CDCl_3$)

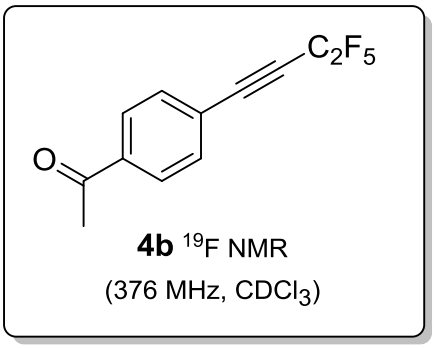




4b ^{1}H NMR

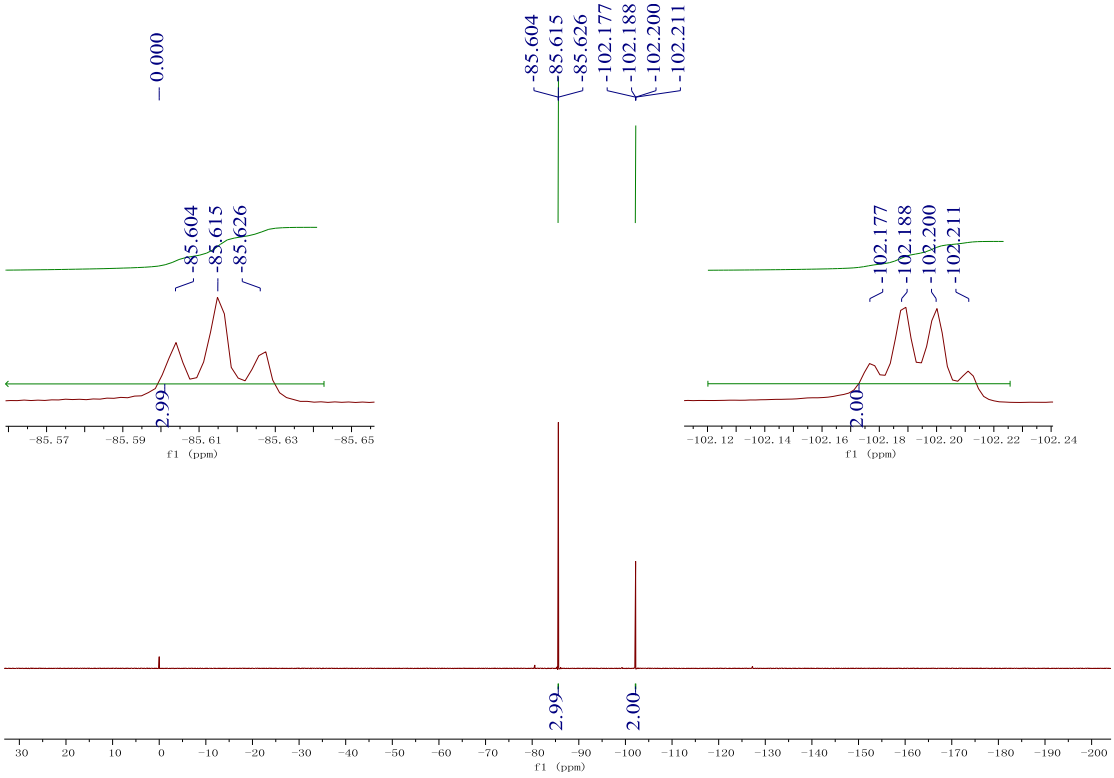
(400 MHz, $CDCl_3$)

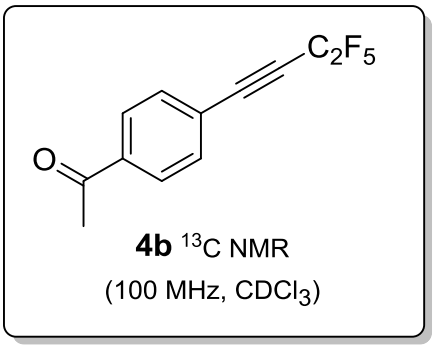


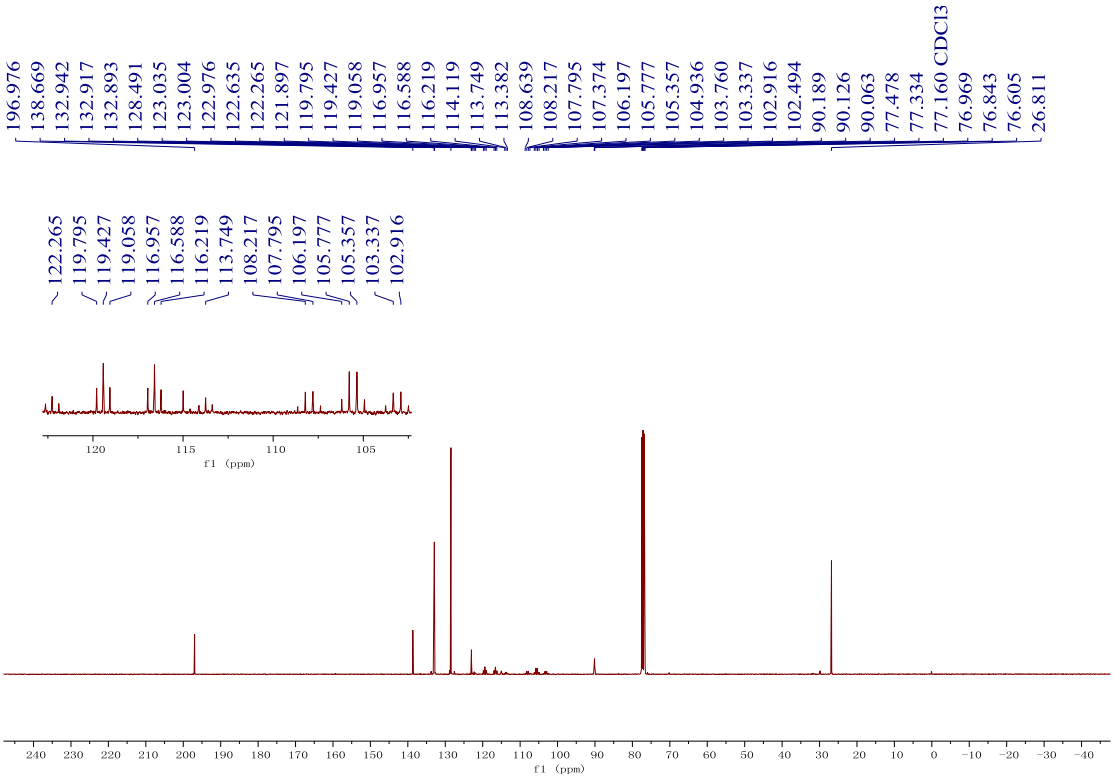


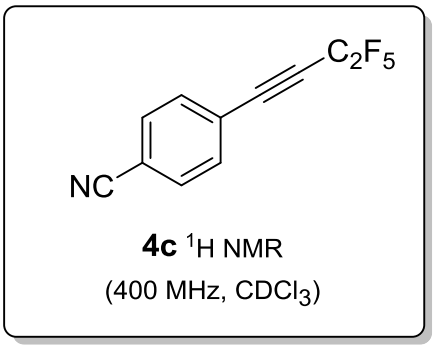
4b ^{19}F NMR

(376 MHz, $CDCl_3$)

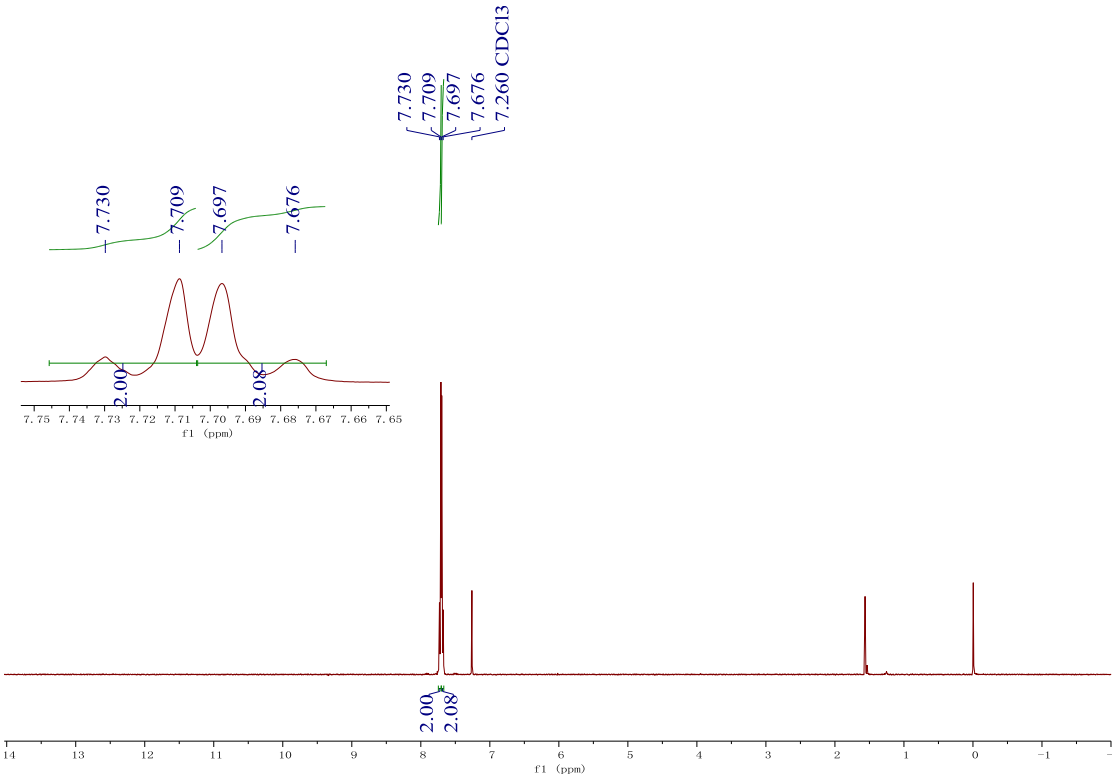


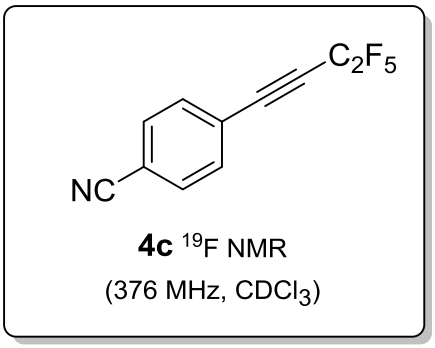

C_2F_5
O
4b ^{13}C NMR
(100 MHz, $CDCl_3$)


196.976
138.669
132.942
132.917
132.893
128.491
123.035
123.004
122.976
122.635
122.265
121.897
119.795
119.427
119.058
116.957
116.588
116.219
114.119
113.749
113.382
108.639
108.217
107.795
107.374
106.197
105.777
105.357
104.936
103.760
103.337
102.916
102.494
90.189
90.126
90.063
77.478
77.334
77.160 CDCl3
76.969
76.843
76.605
26.811
122.265
119.795
119.427
119.058
116.957
116.588
116.219
113.749
108.217
107.795
106.197
105.777
105.357
103.337
102.916
120
115
110
105
f1 (ppm)
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40
f1 (ppm)



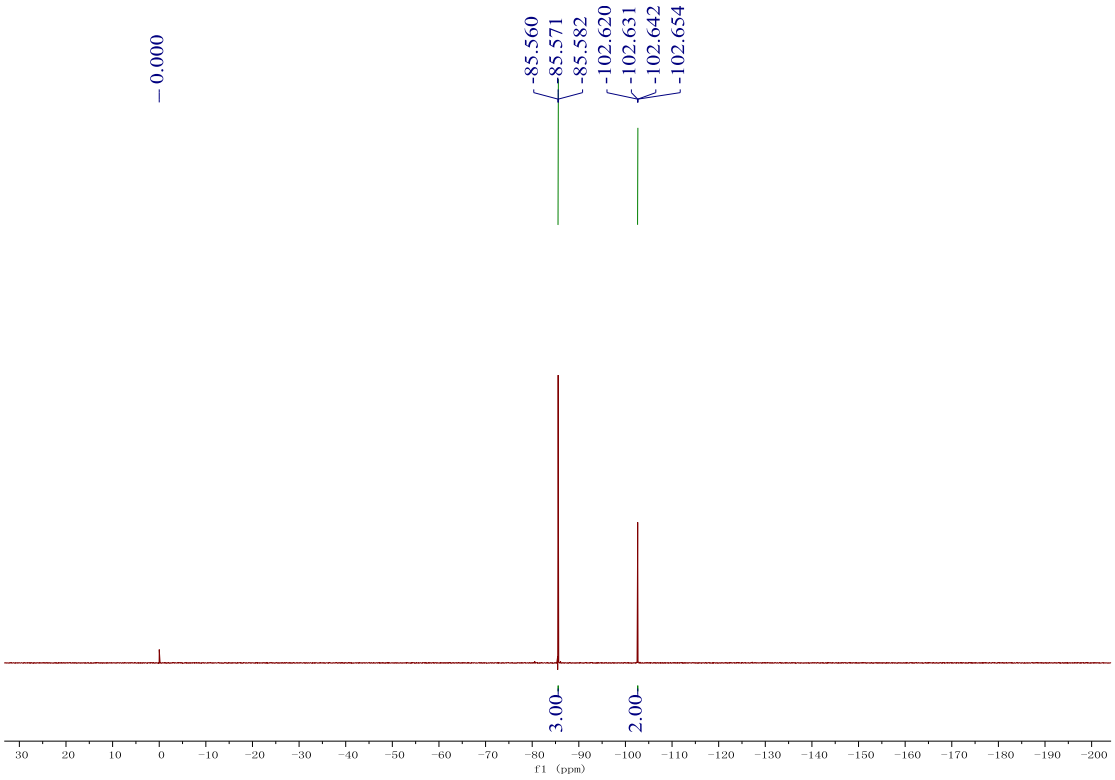
4c ^{1}H NMR
(400 MHz, $CDCl_3$)

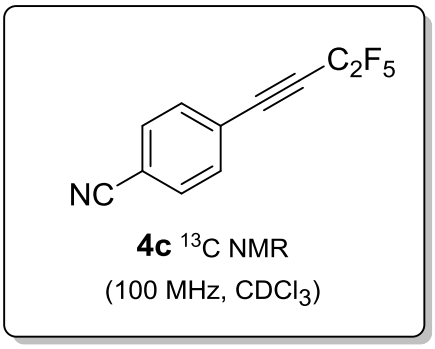




4c ^{19}F NMR

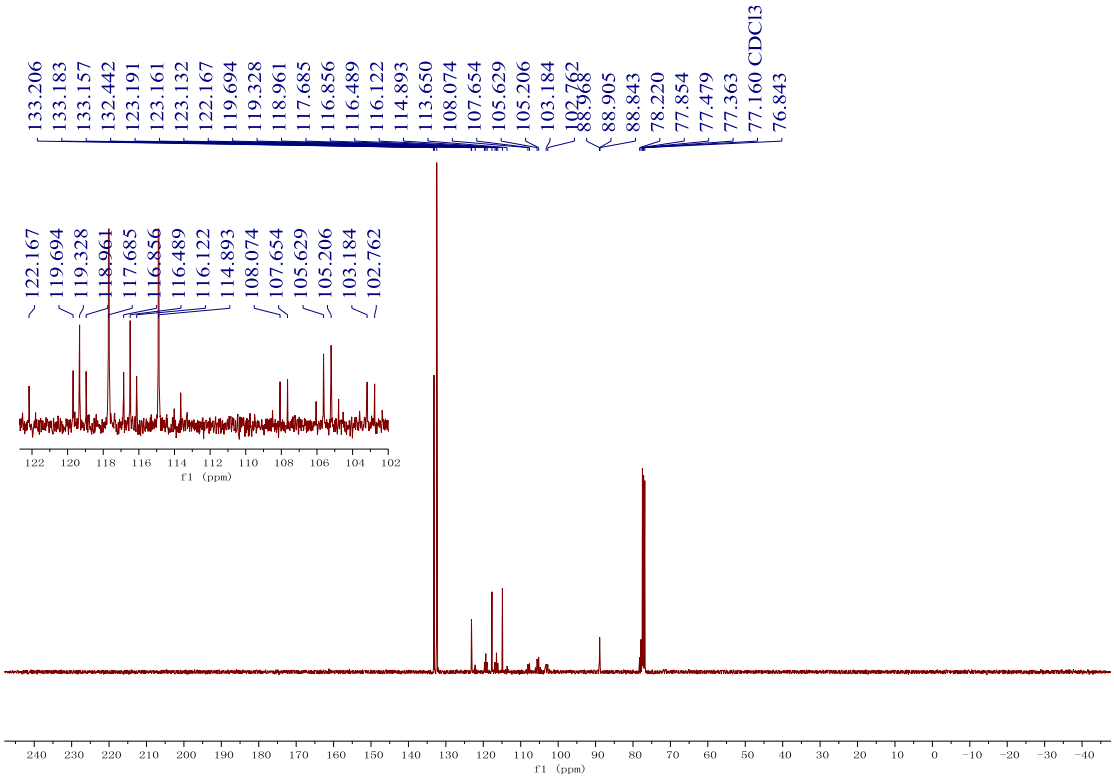
(376 MHz, $CDCl_3$)

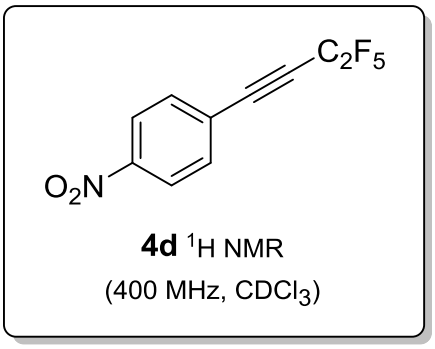




4c ^{13}C NMR

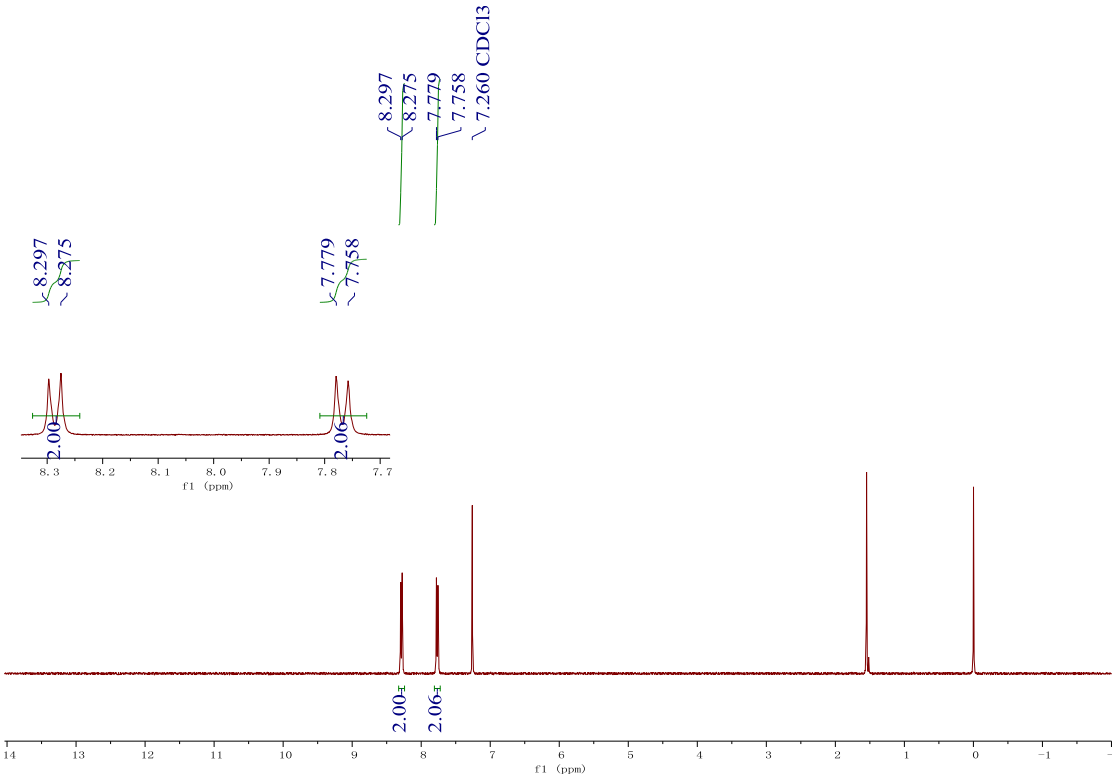
(100 MHz, $CDCl_3$)

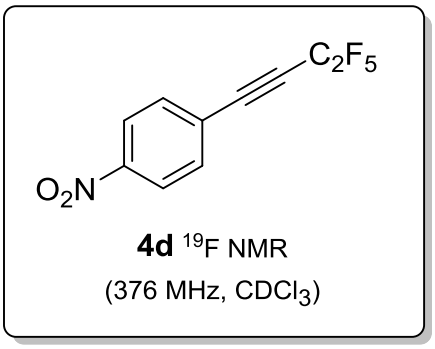


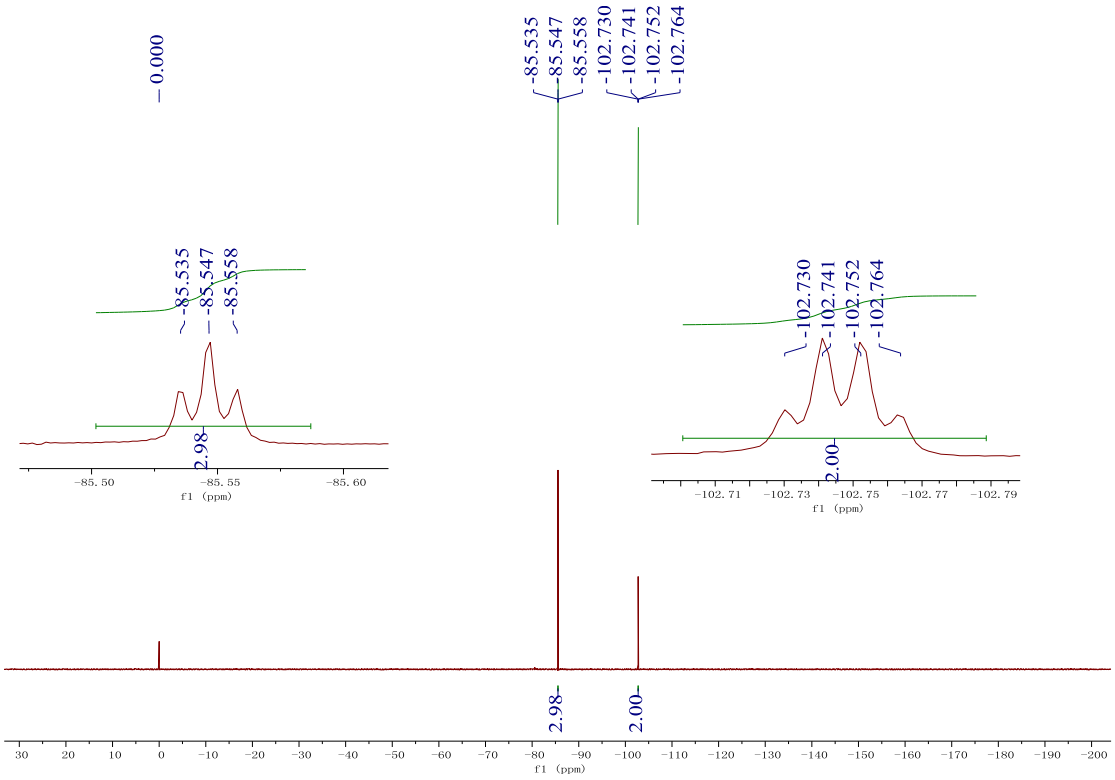


4d ^{1}H NMR

(400 MHz, $CDCl_3$)




C_2F_5
O_2N
4d ^{19}F NMR
(376 MHz, $CDCl_3$)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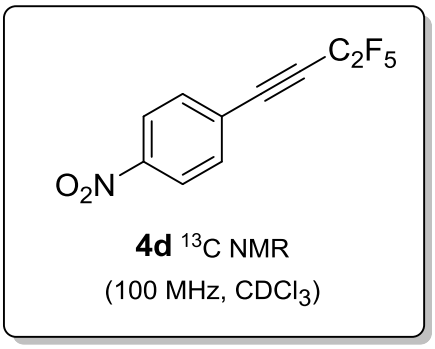
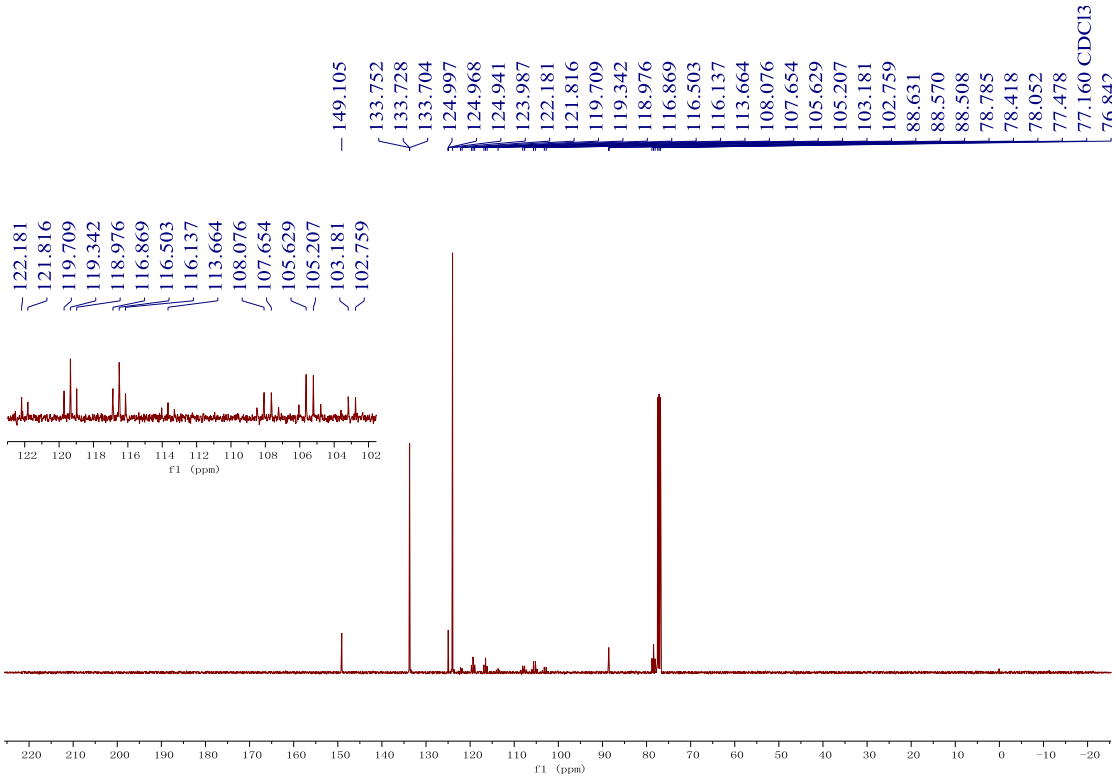
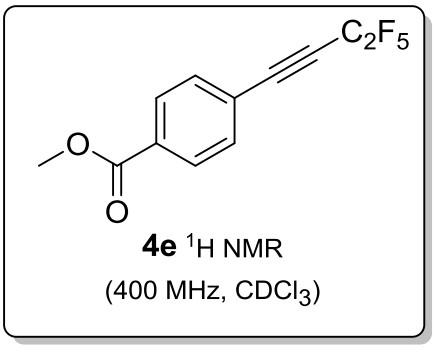
-85.558
-102.730
-102.741
-102.752
-102.764
2.98
2.00
f1 (ppm)



4d ^{13}C NMR

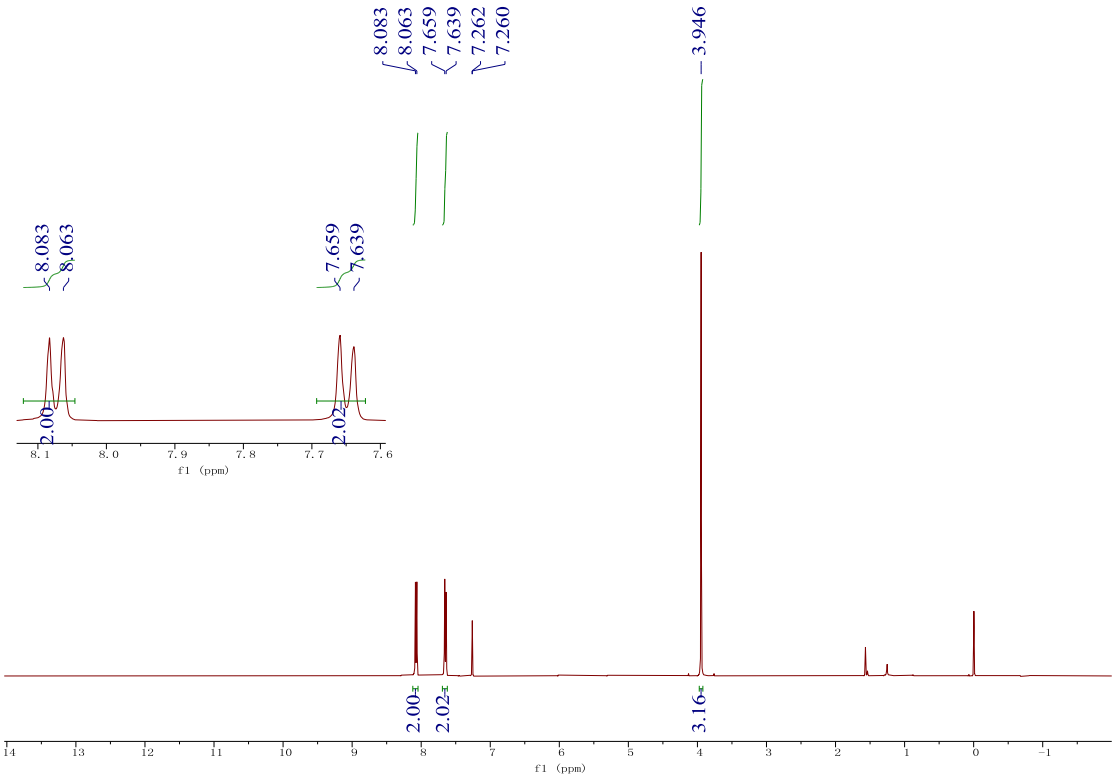
(100 MHz, $CDCl_3$)

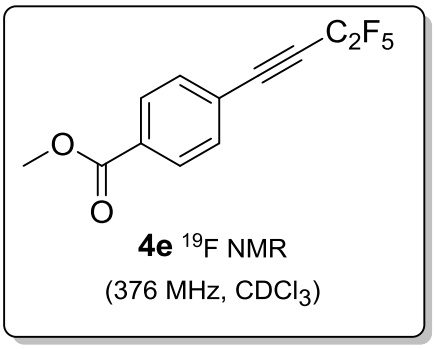




4e ^{1}H NMR

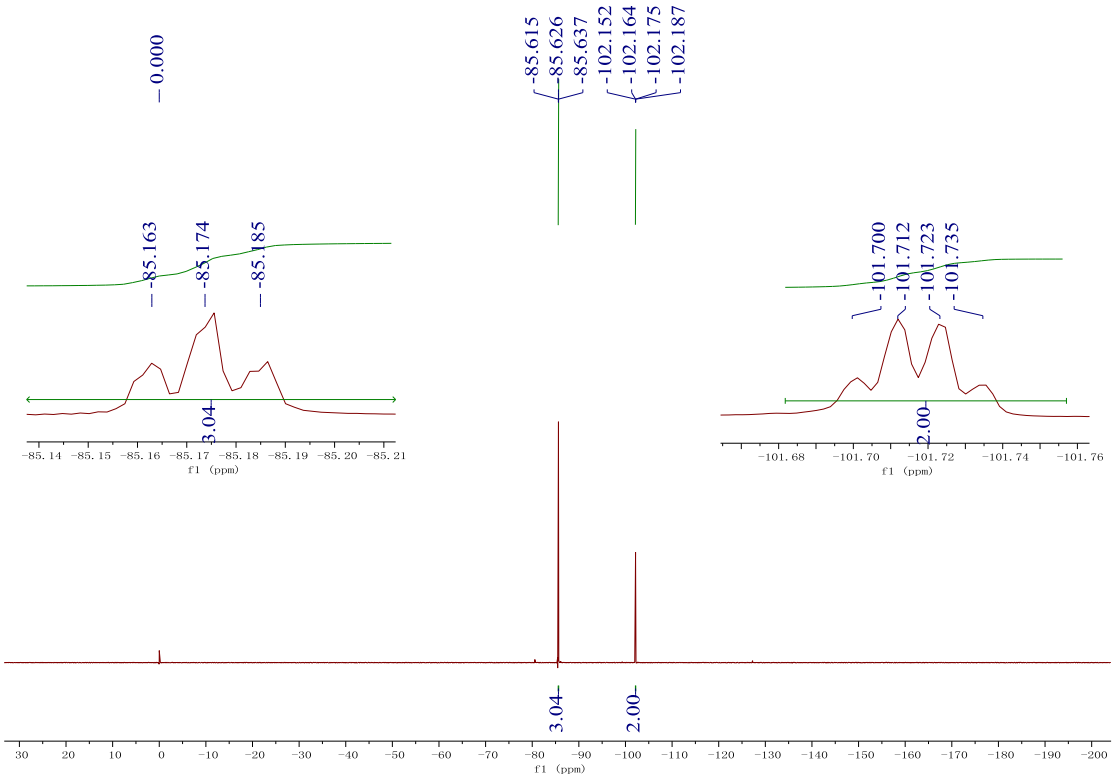
(400 MHz, $CDCl_3$)

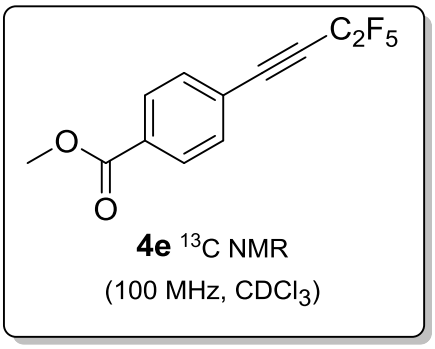




4e ^{19}F NMR

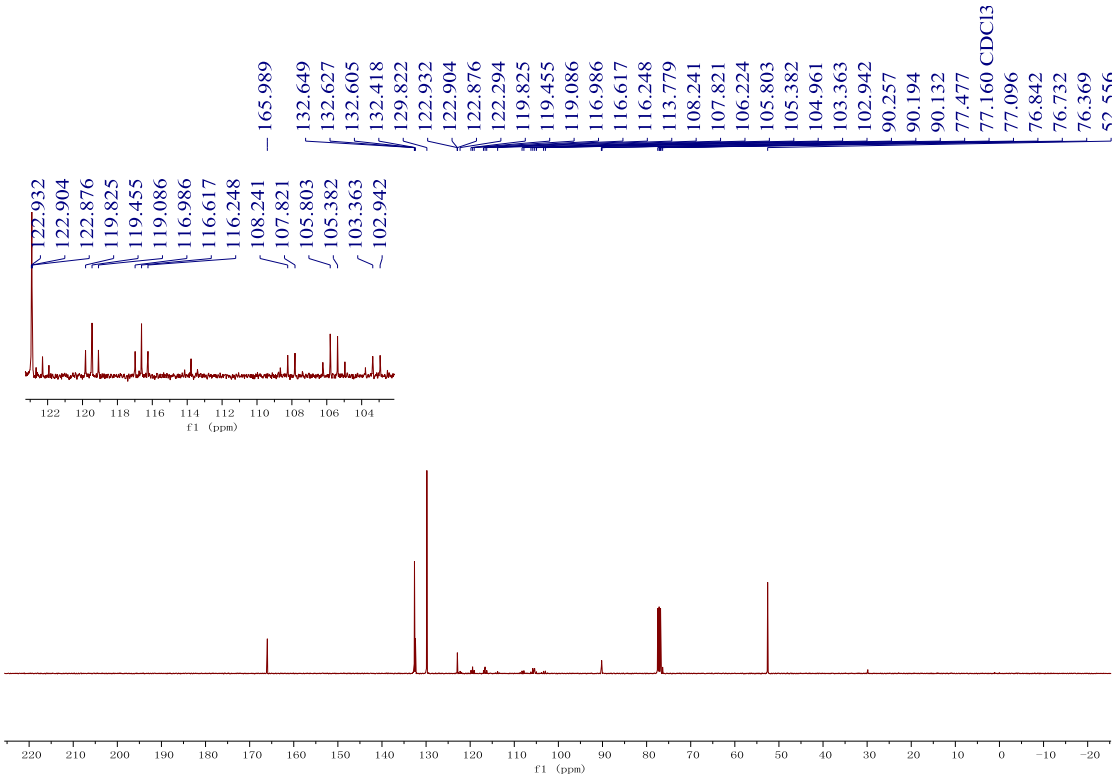
(376 MHz, $CDCl_3$)

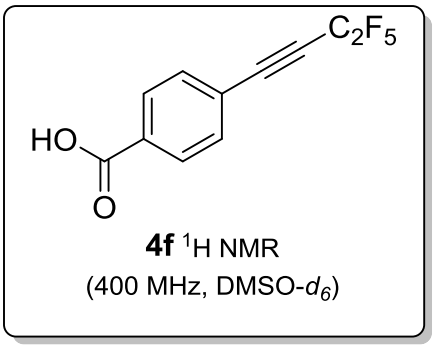




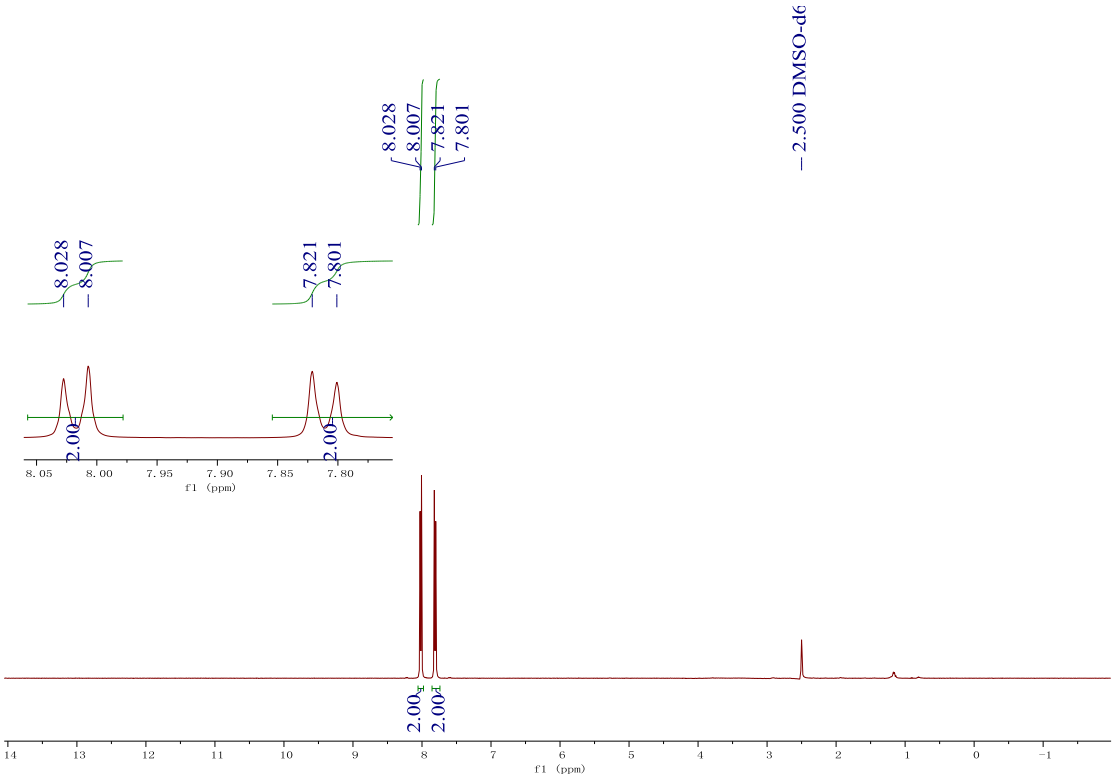
4e ^{13}C NMR

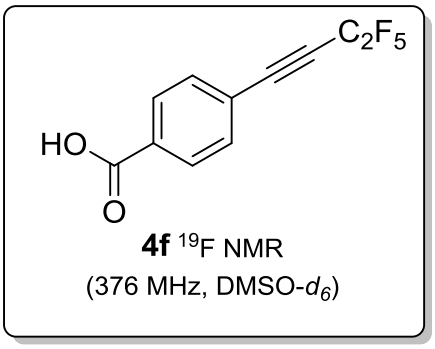
(100 MHz, $CDCl_3$)



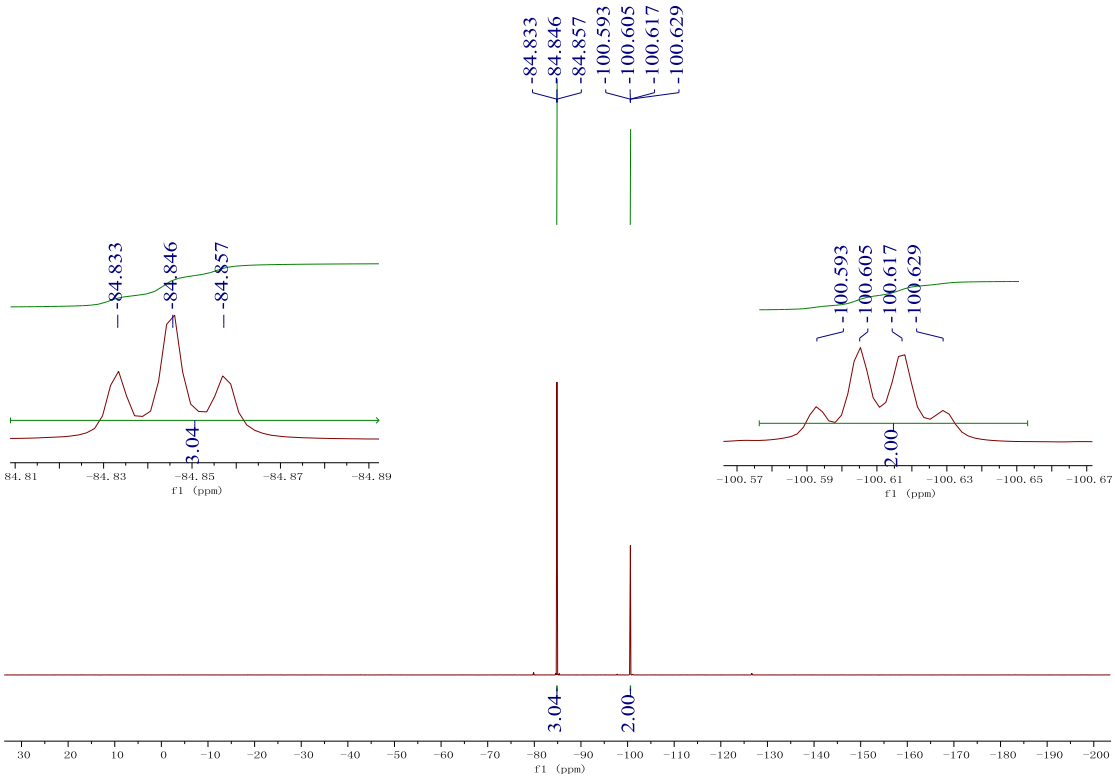


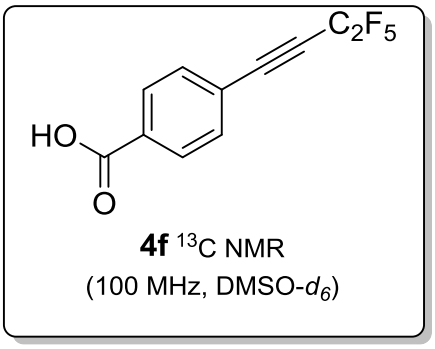
4f ^{1}H NMR
(400 MHz, DMSO-d_6)



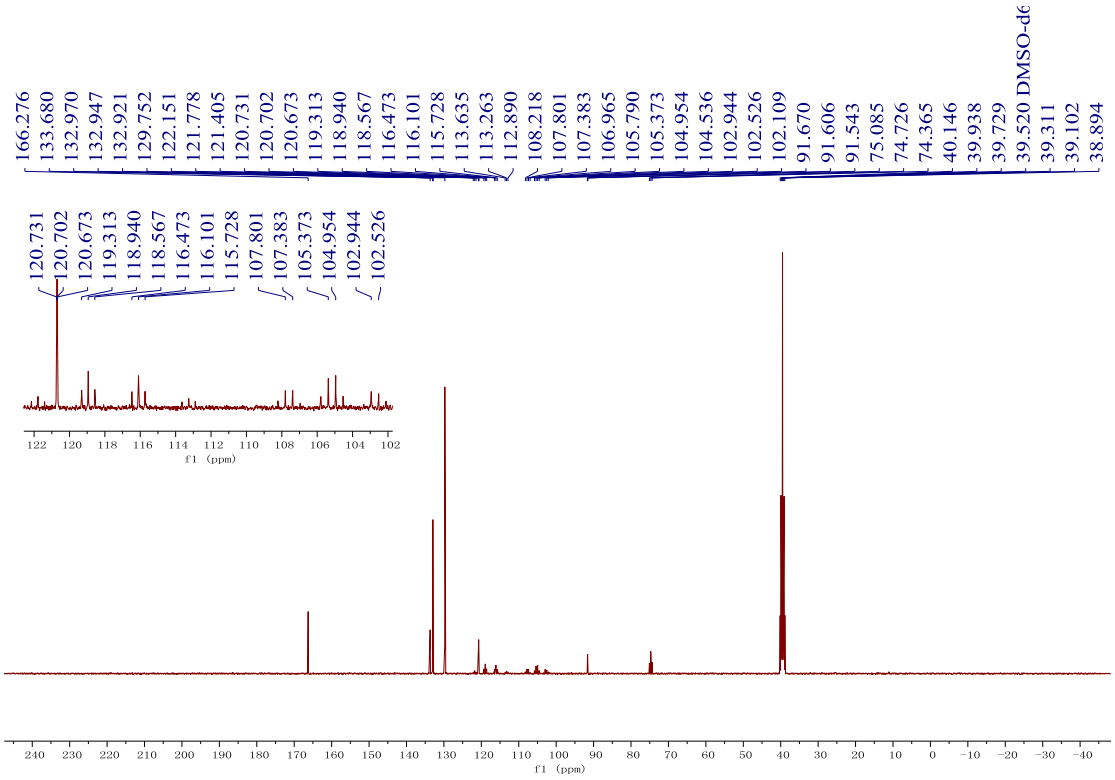


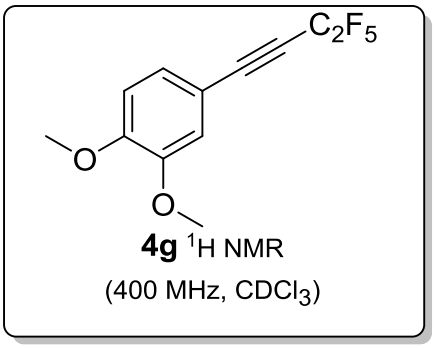
4f ^{19}F NMR
(376 MHz, DMSO-d_6)

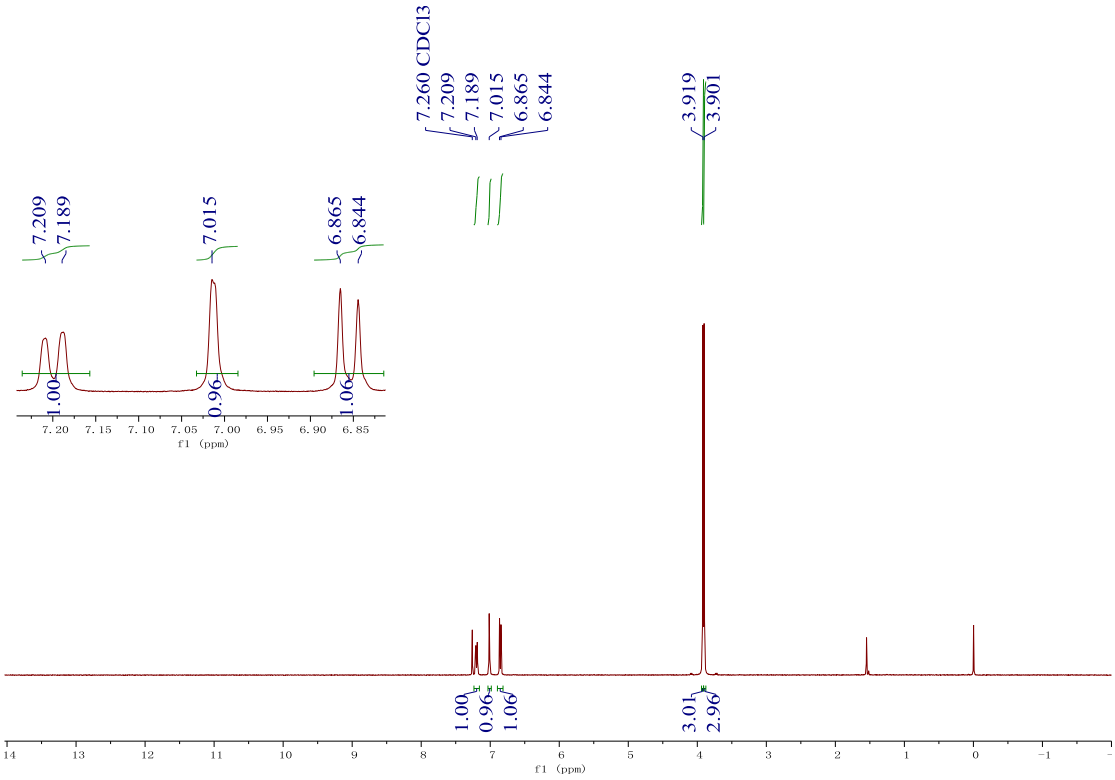


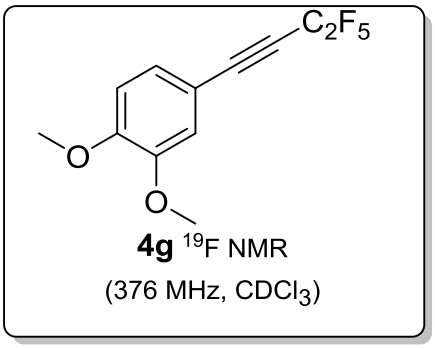


4f ^{13}C NMR
(100 MHz, DMSO-d_6)



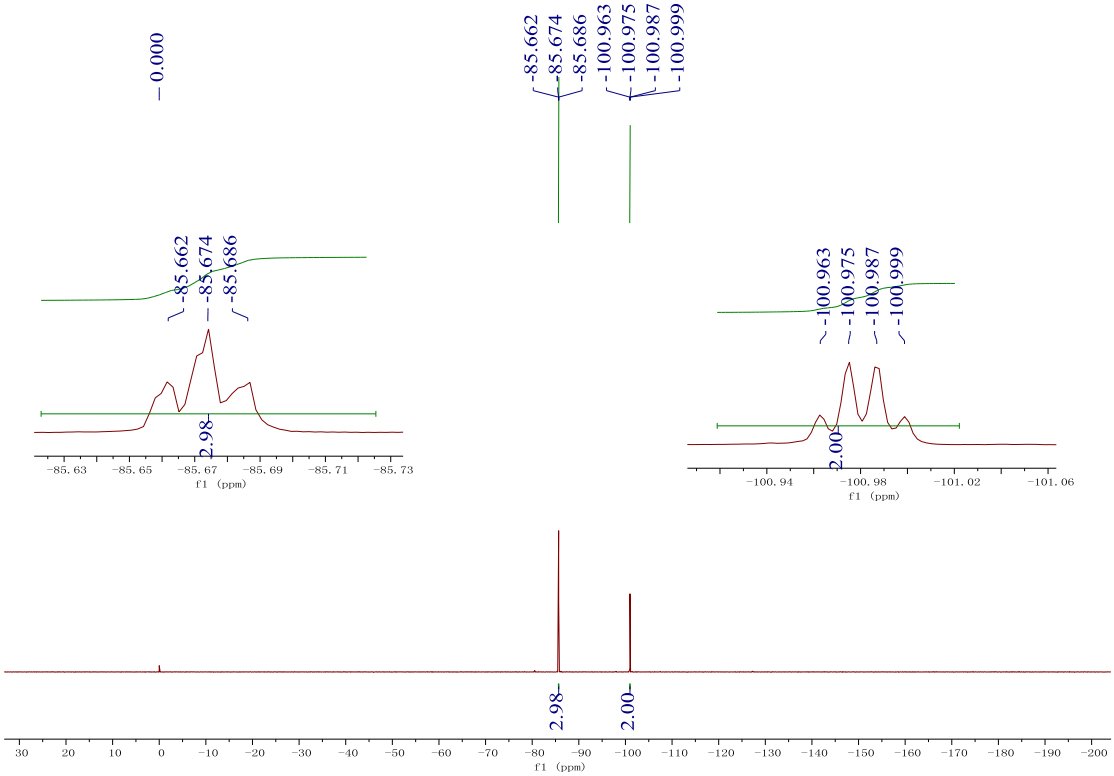

C_2F_5
O
O
4g ^{1}H NMR
(400 MHz, $CDCl_3$)

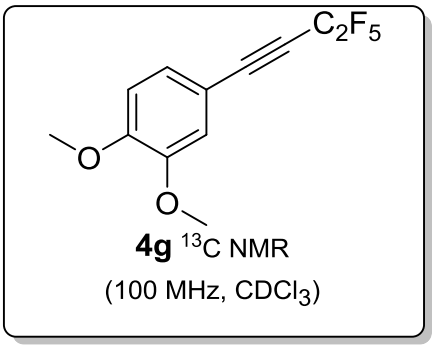

7.260 CDCl3
7.209
7.189
7.015
6.865
6.844
3.919
3.901
1.00
0.96
1.06
3.01
2.96
f1 (ppm)



4g ^{19}F NMR

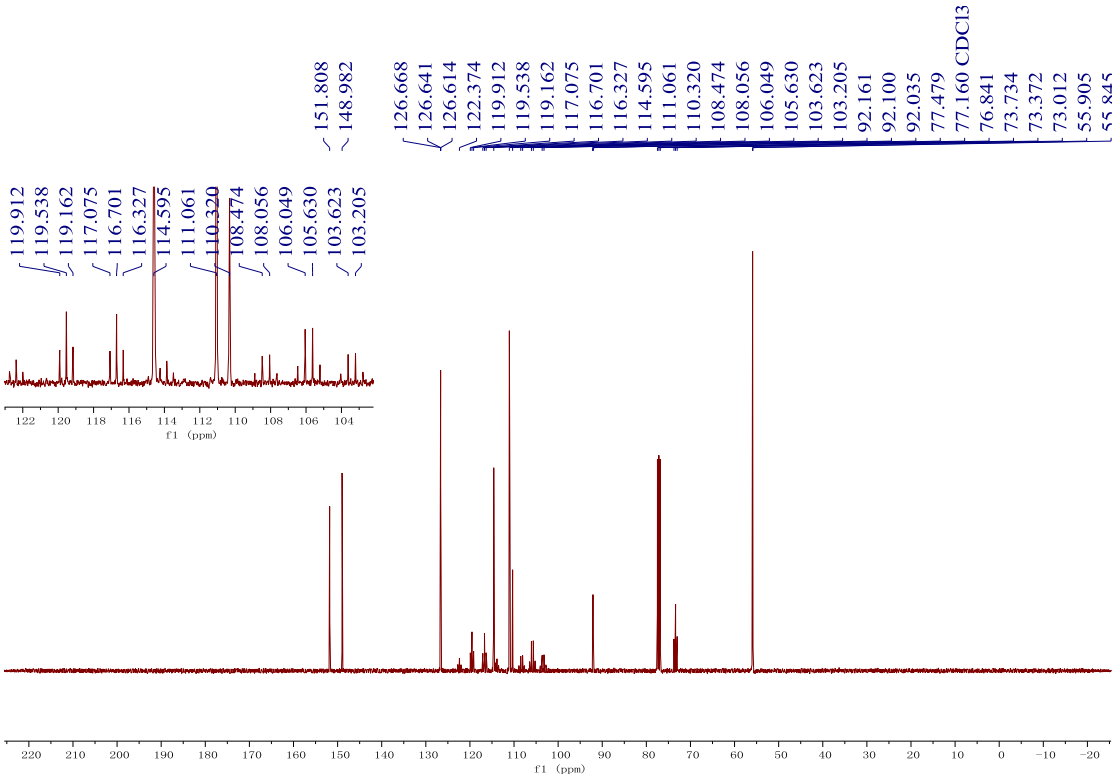
(376 MHz, $CDCl_3$)

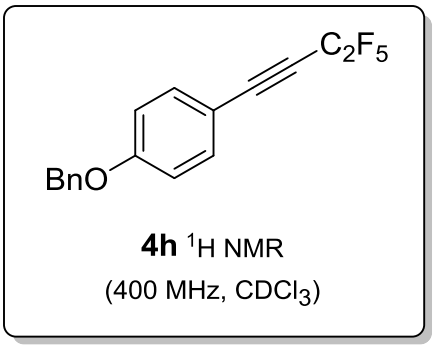




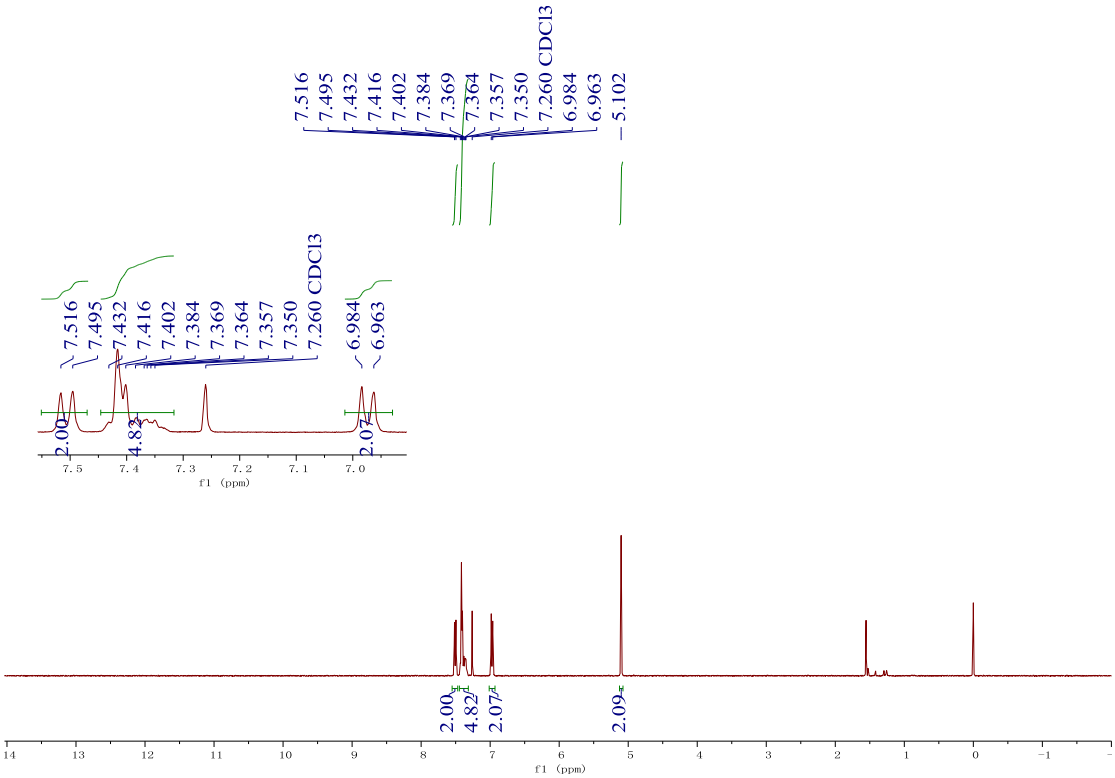
4g ^{13}C NMR

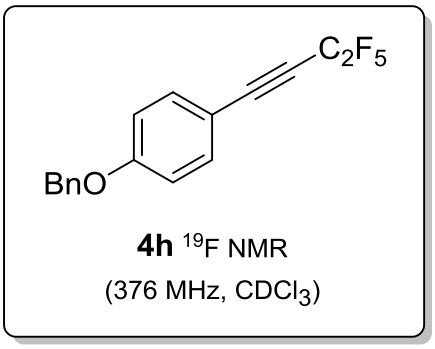
(100 MHz, $CDCl_3$)





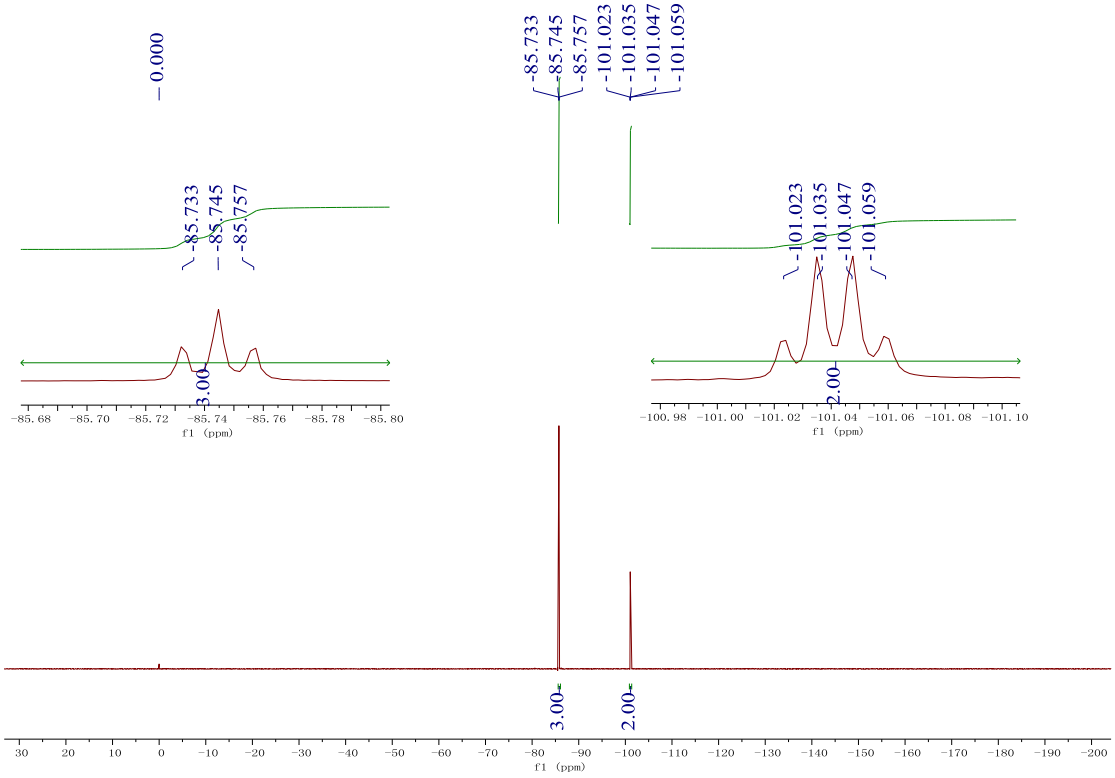
4h ^{1}H NMR
(400 MHz, $CDCl_3$)

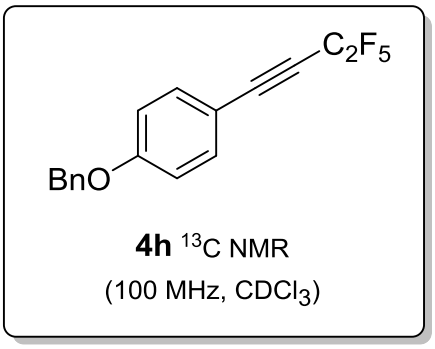




4h ^{19}F NMR

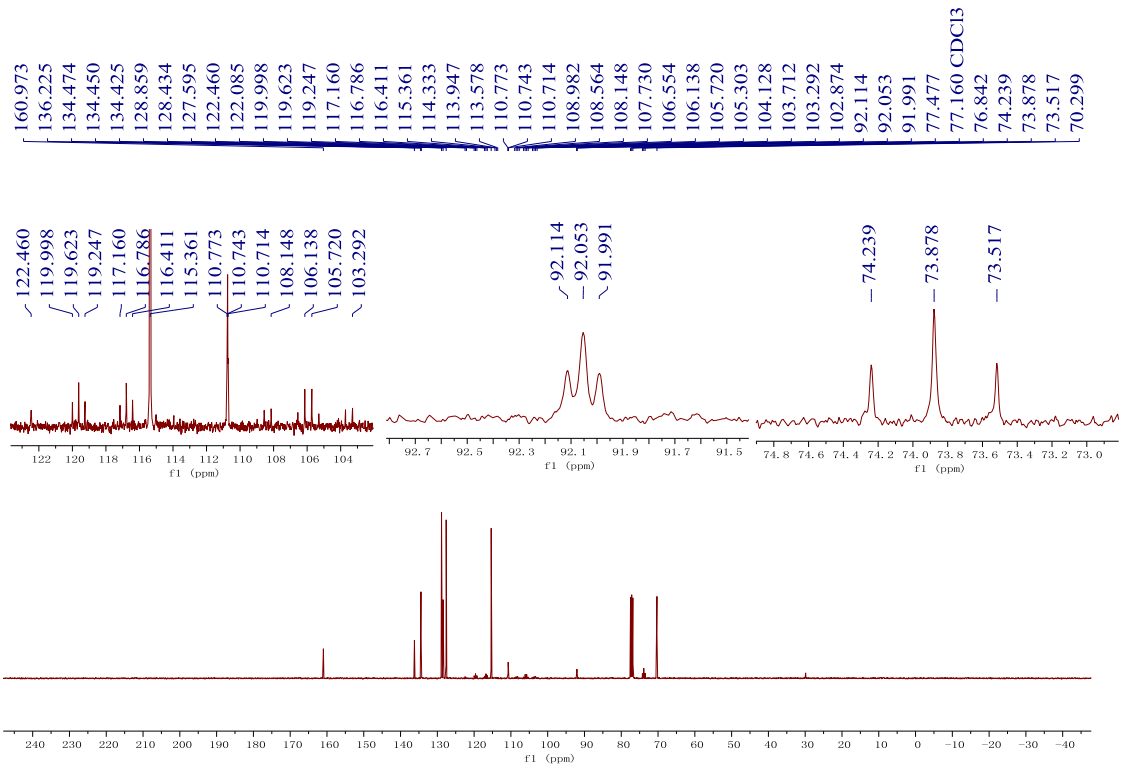
(376 MHz, $CDCl_3$)

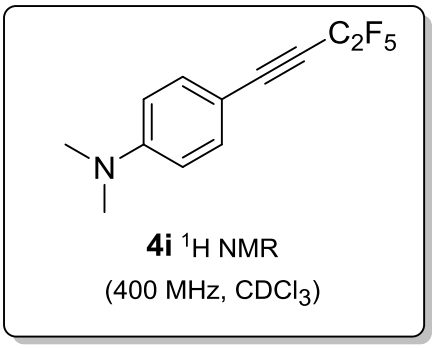




4h ^{13}C NMR

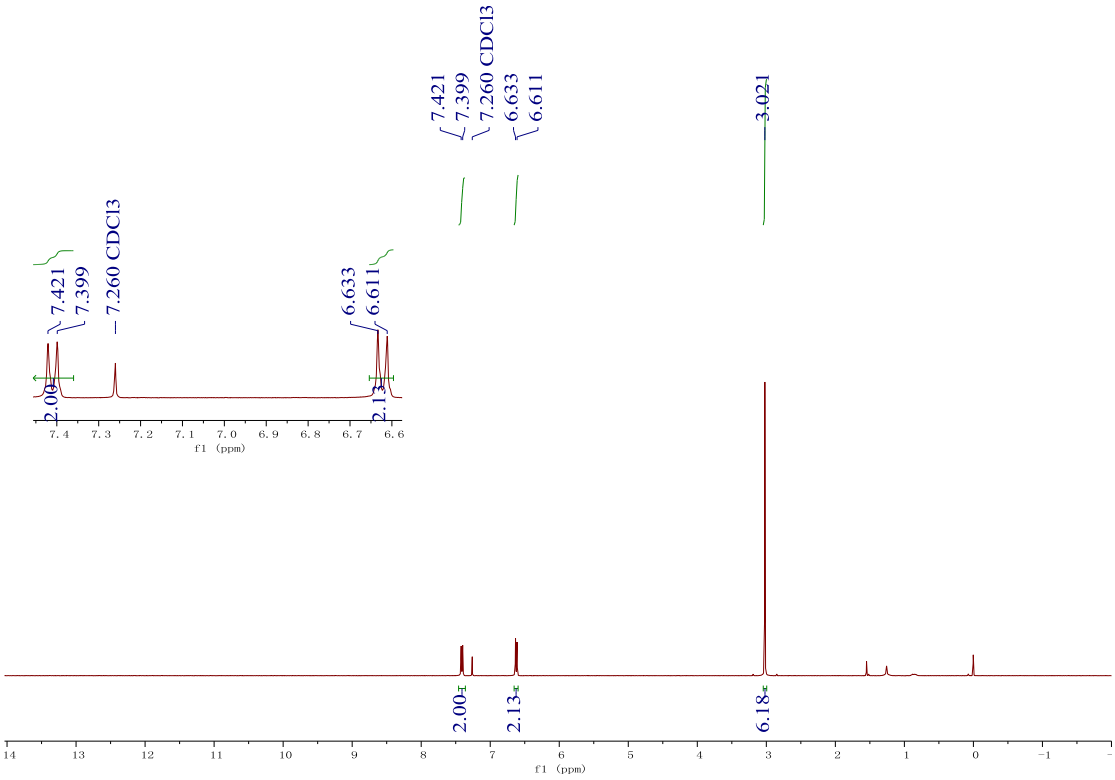
(100 MHz, $CDCl_3$)

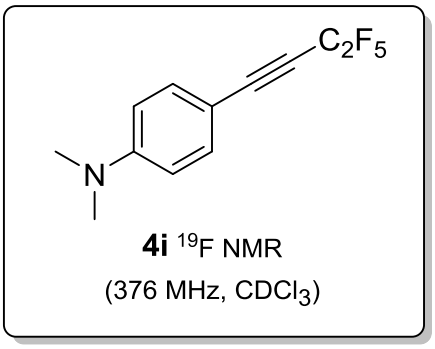




4i ^{1}H NMR

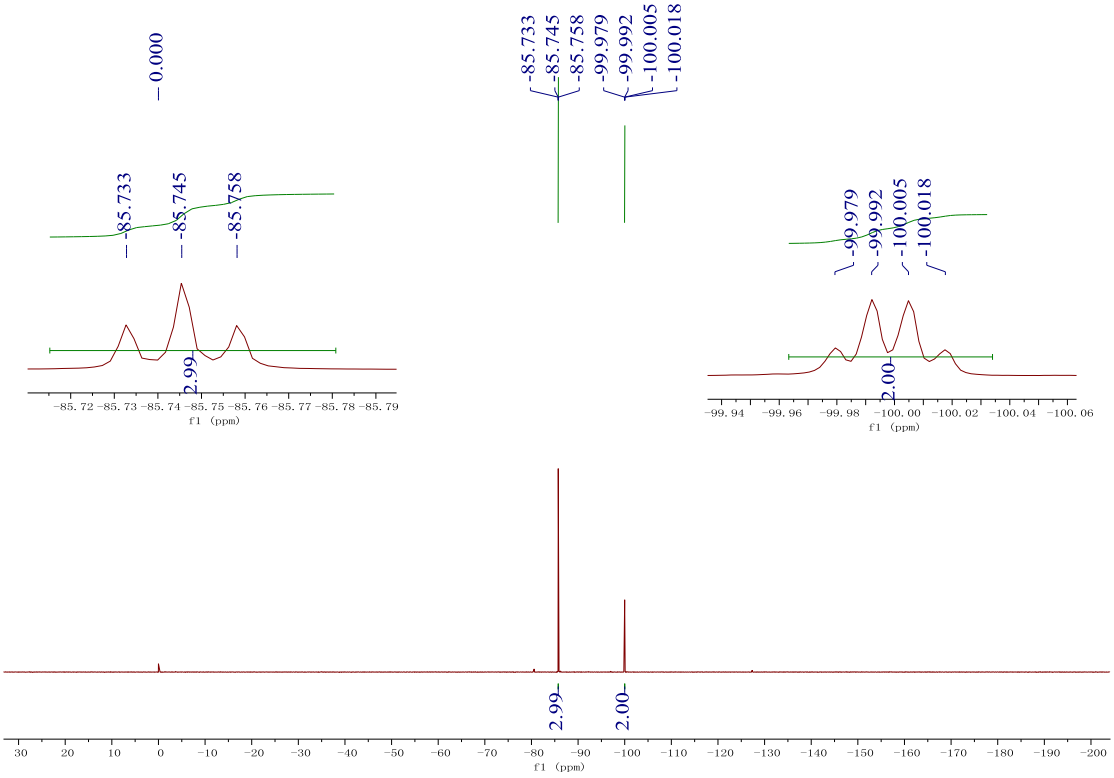
(400 MHz, $CDCl_3$)

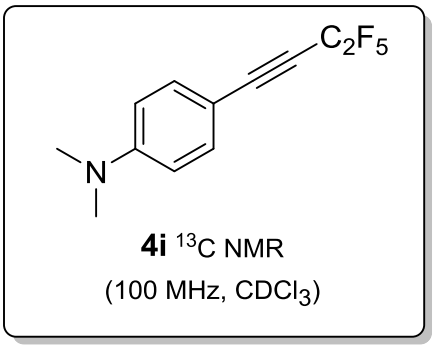




4i ^{19}F NMR

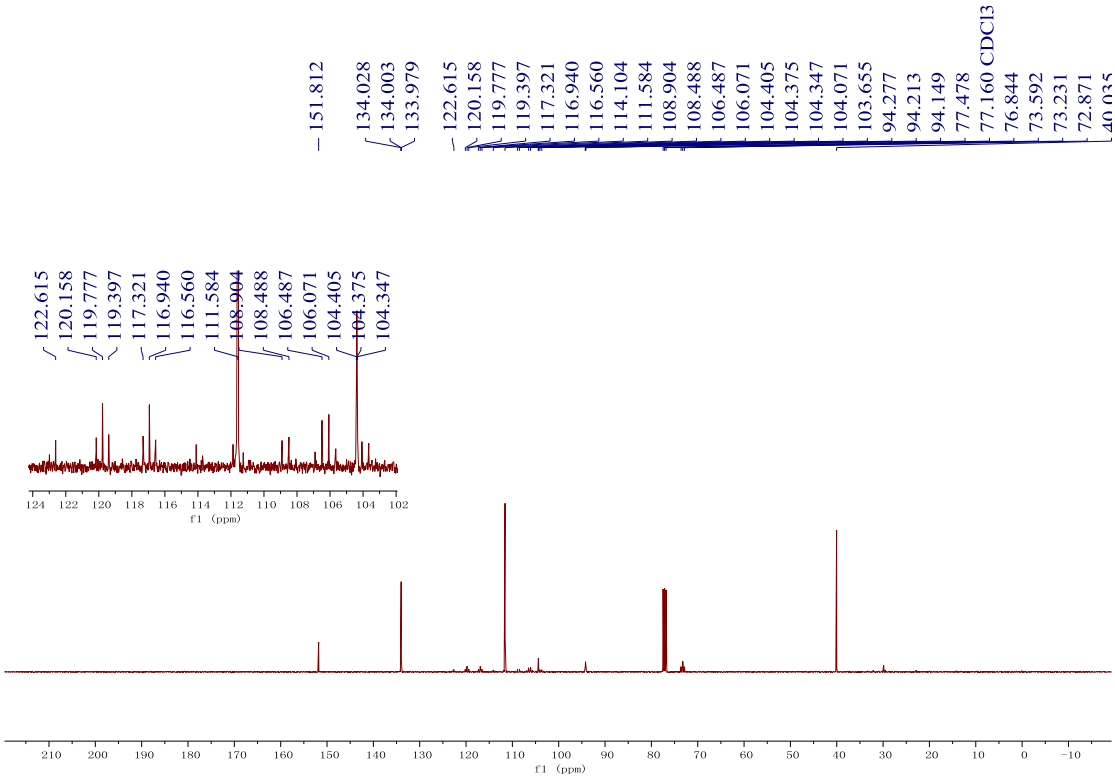
(376 MHz, $CDCl_3$)

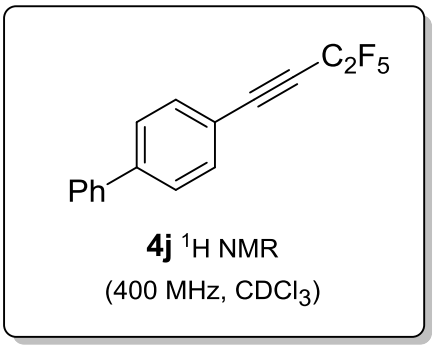




4i ^{13}C NMR

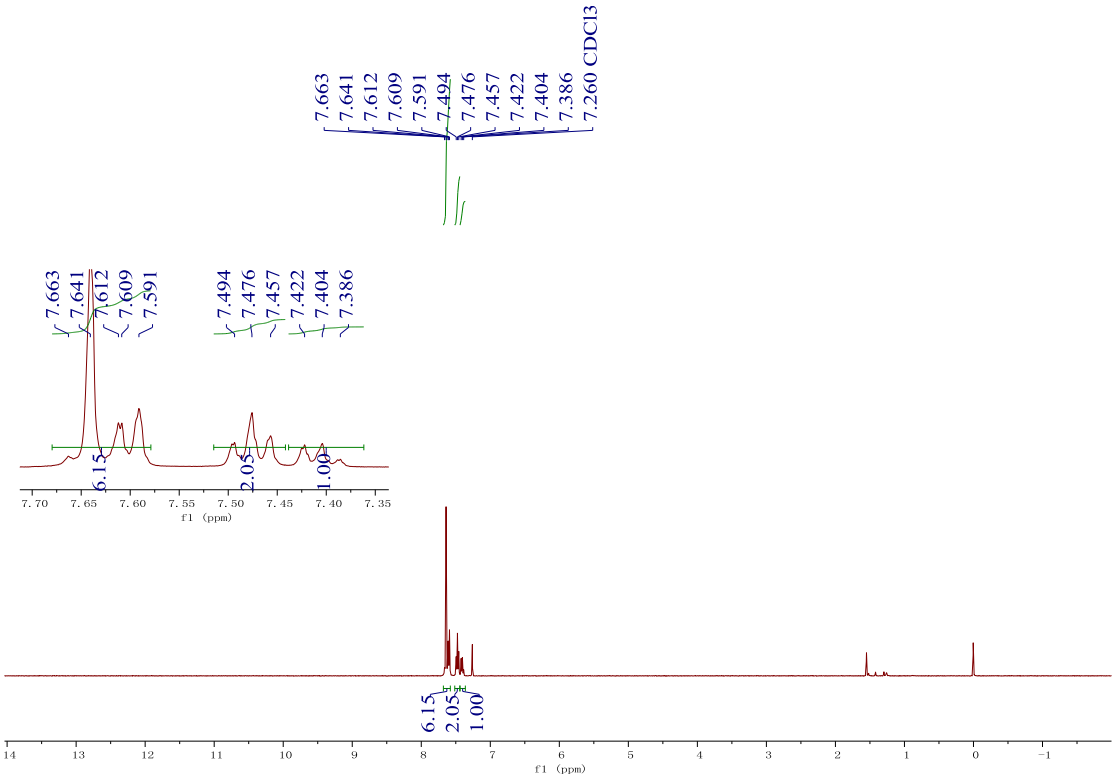
(100 MHz, $CDCl_3$)

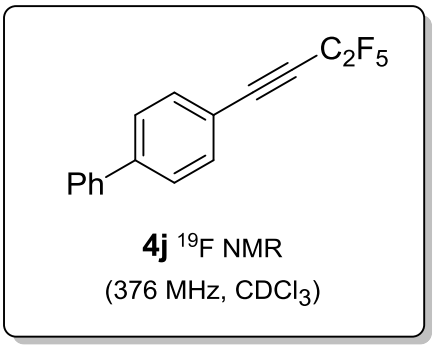




4j ^{1}H NMR

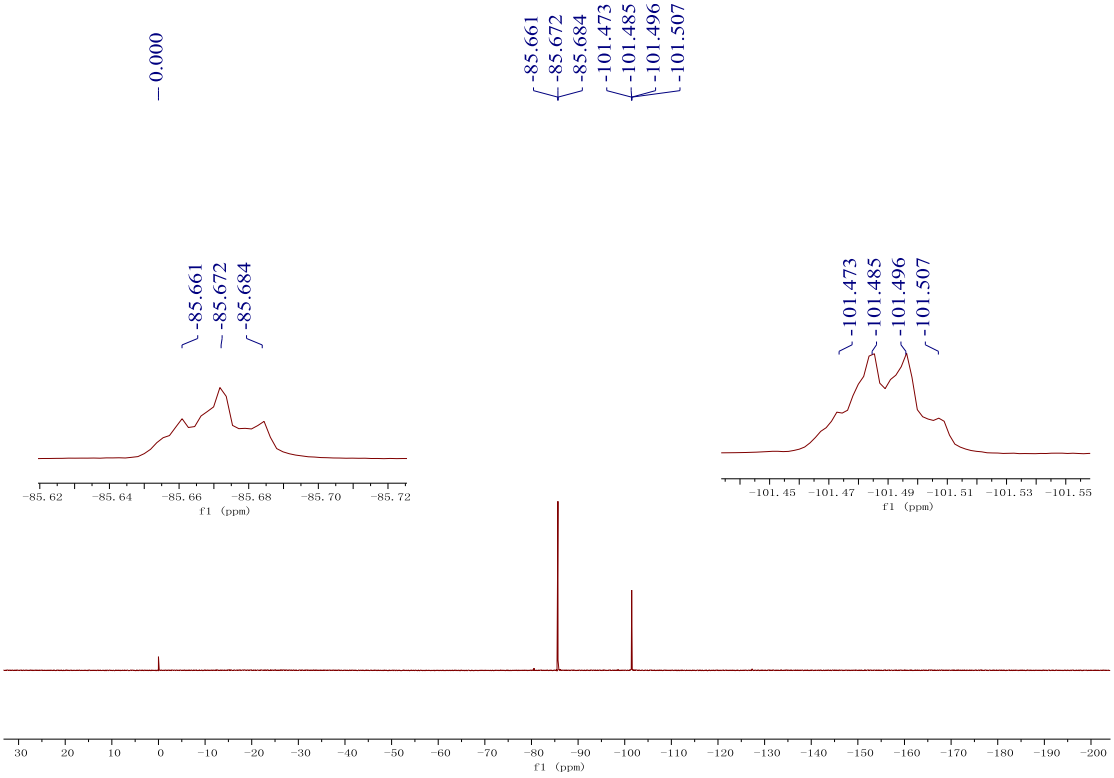
(400 MHz, $CDCl_3$)

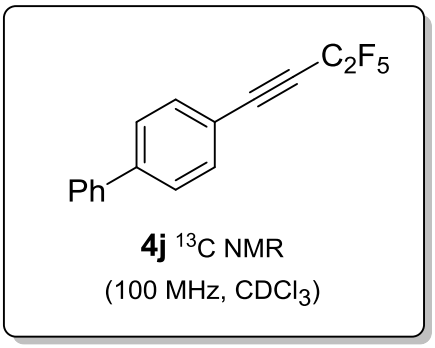




4j ^{19}F NMR

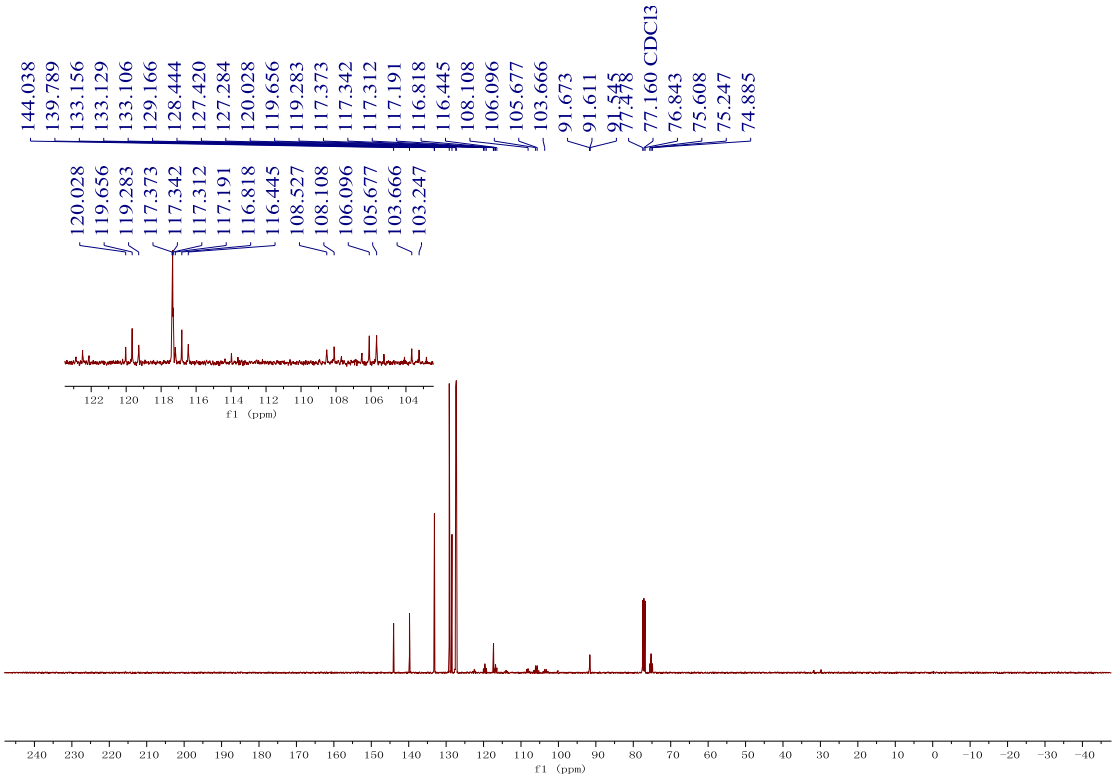
(376 MHz, $CDCl_3$)

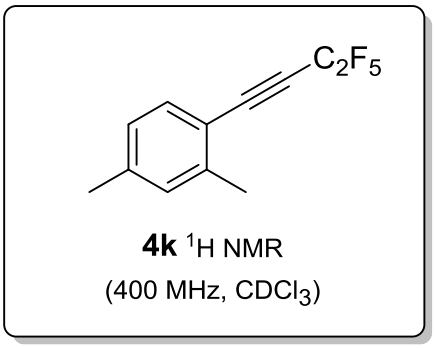




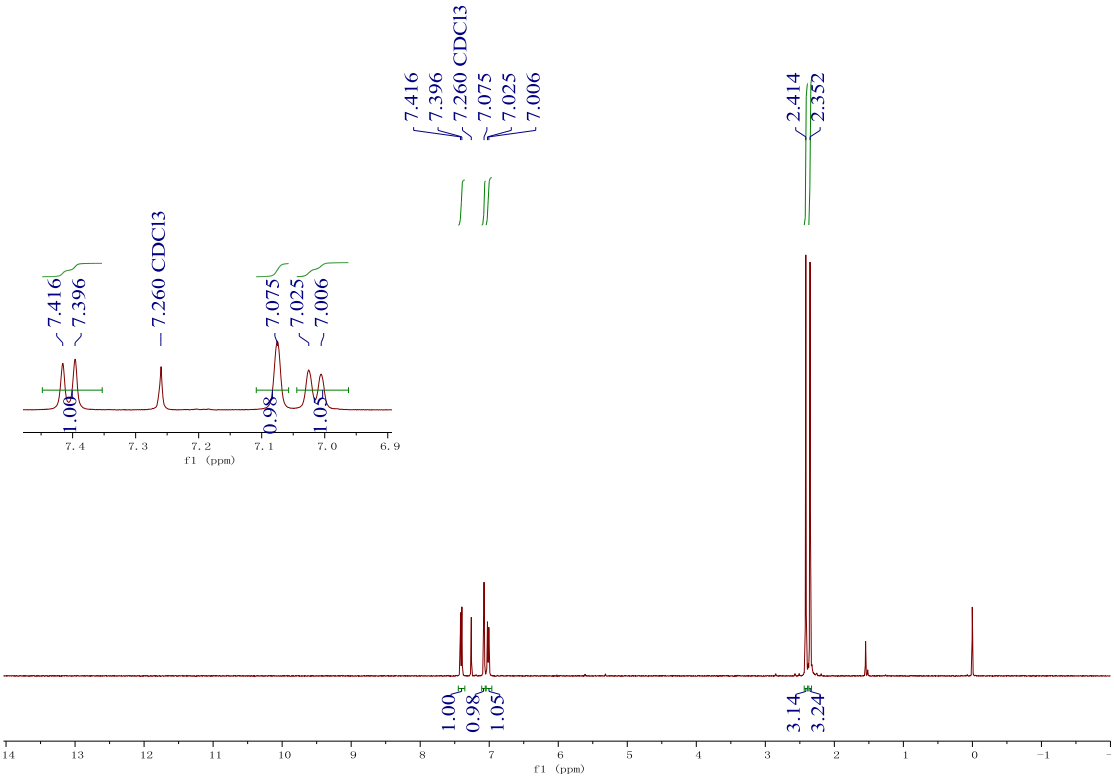
4j ^{13}C NMR

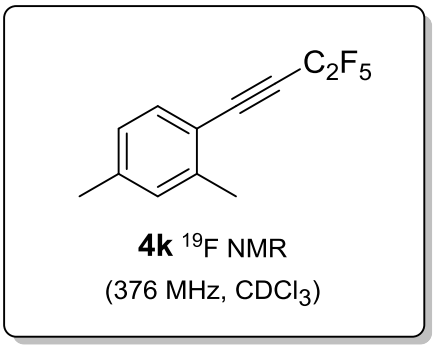
(100 MHz, $CDCl_3$)

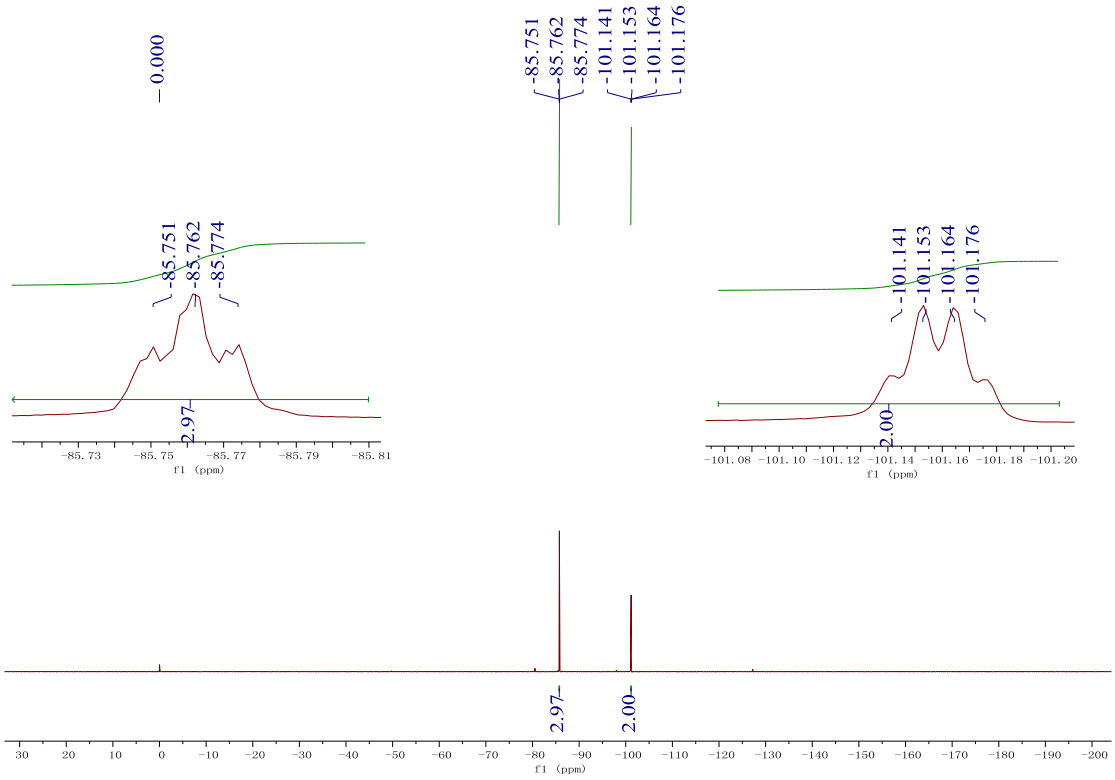




4k ^{1}H NMR
(400 MHz, $CDCl_3$)




C_2F_5
4k ^{19}F NMR
(376 MHz, $CDCl_3$)


-0.000
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-85.774
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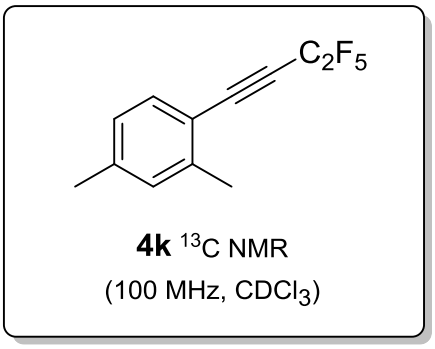
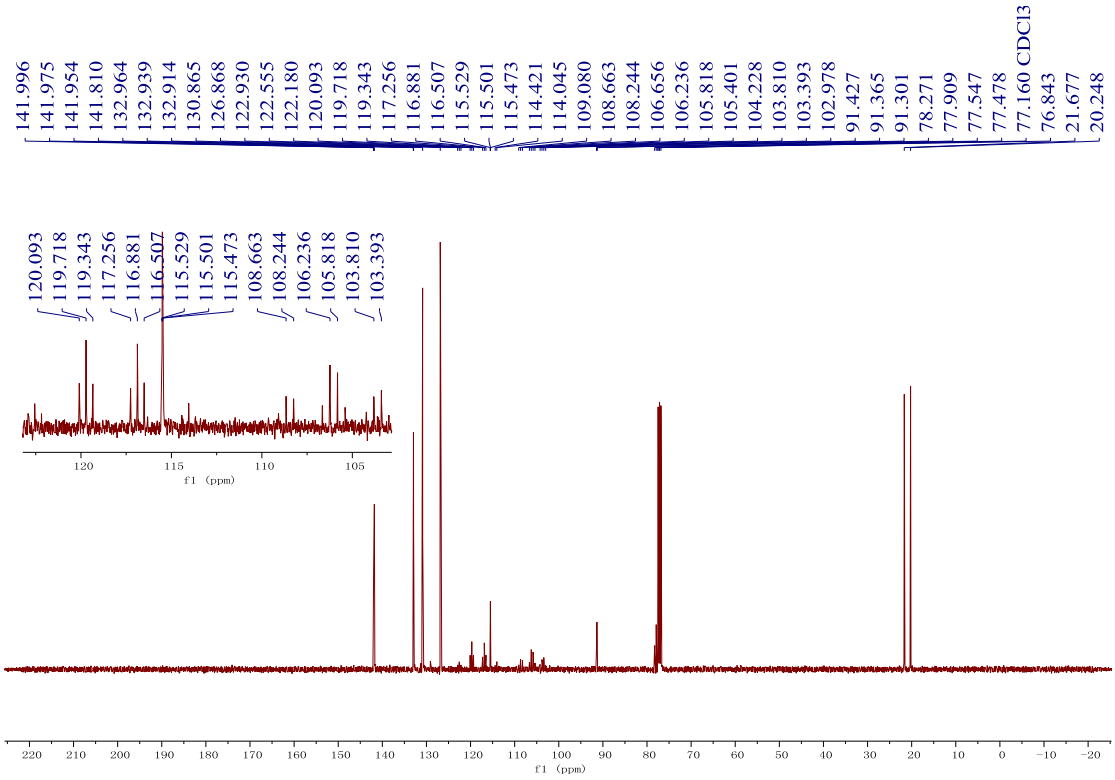
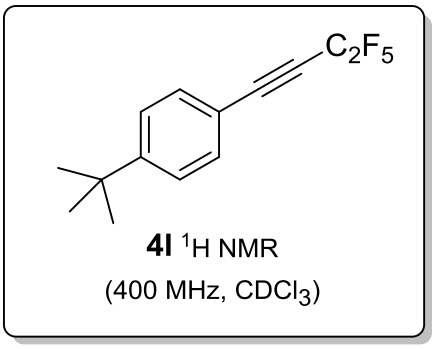
-101.153
-101.164
-101.176
2.97
2.00
f1 (ppm)

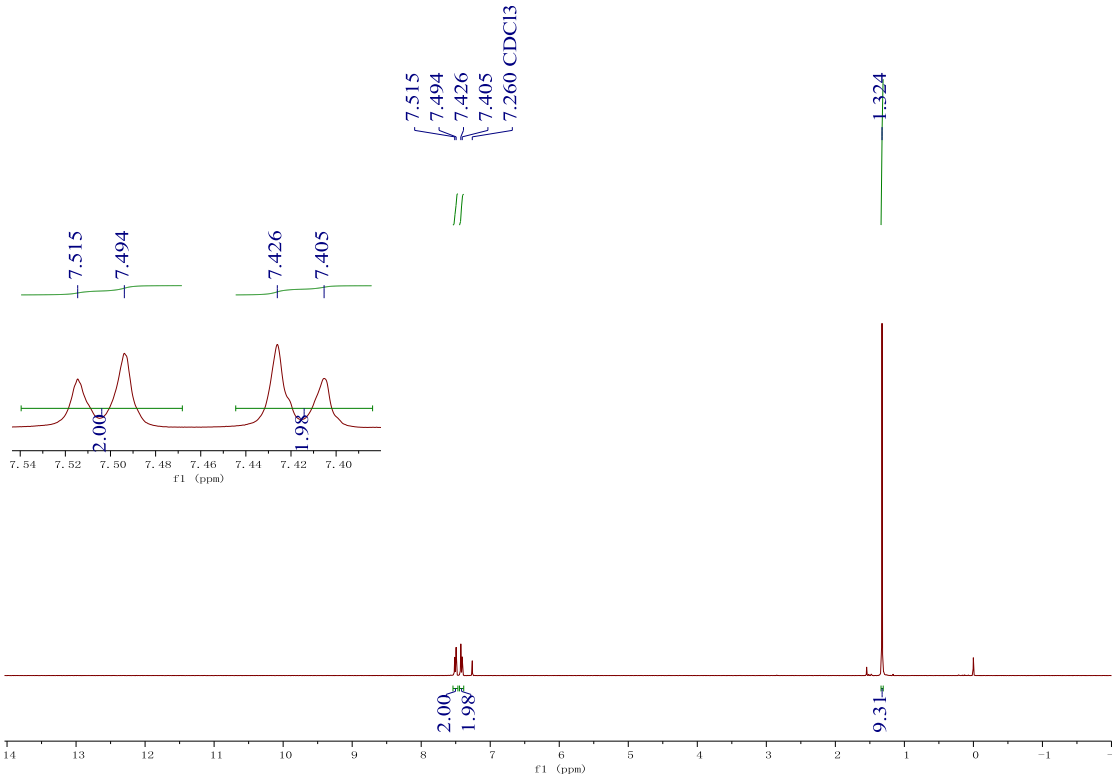


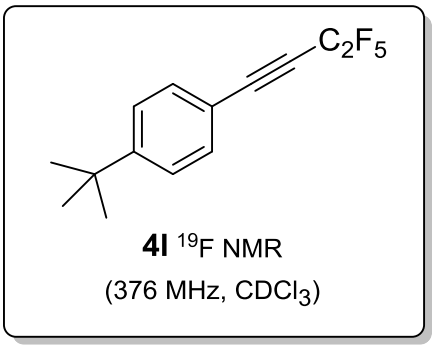
4k ^{13}C NMR

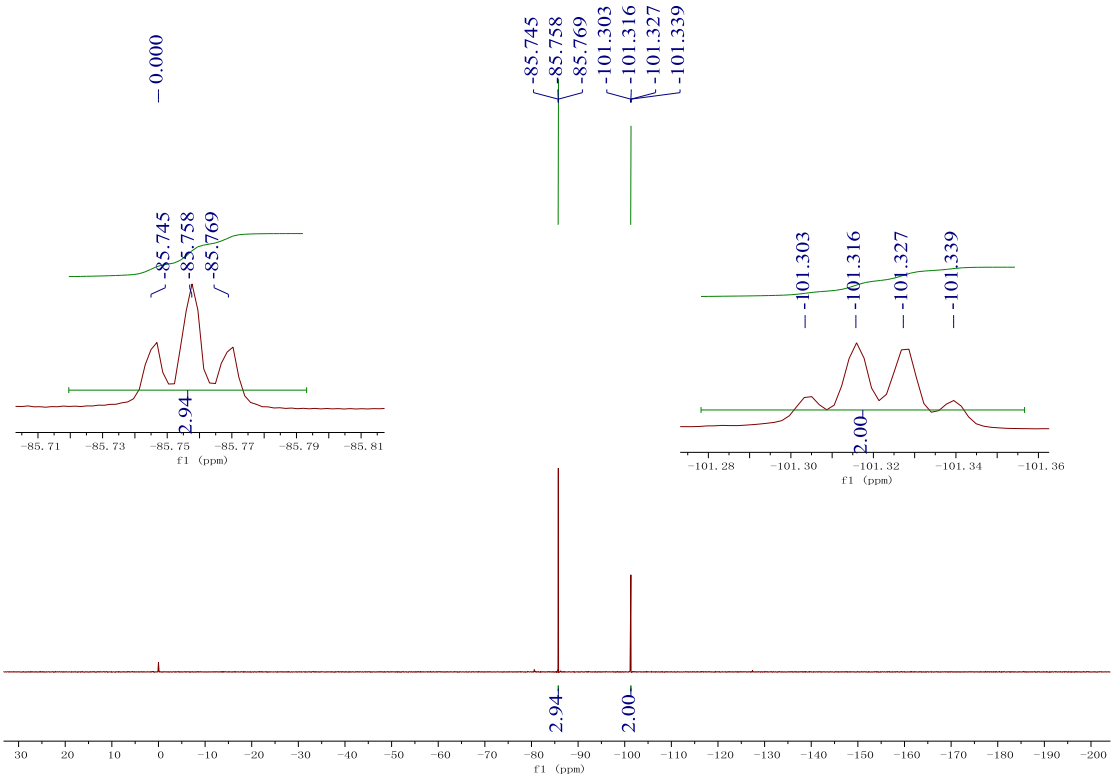
(100 MHz, $CDCl_3$)




C_2F_5
4l ^{1}H NMR
(400 MHz, $CDCl_3$)


7.515
7.494
7.426
7.405
7.260 CDCl3
1.324
2.00
1.98
9.31
f1 (ppm)


C_2F_5
4l ^{19}F NMR
(376 MHz, $CDCl_3$)


-0.000
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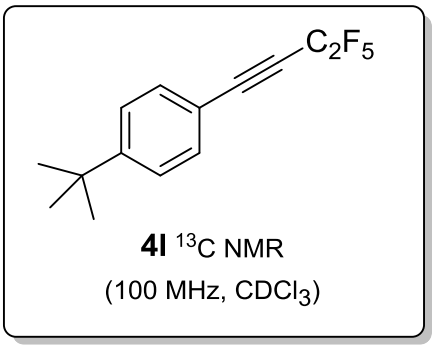
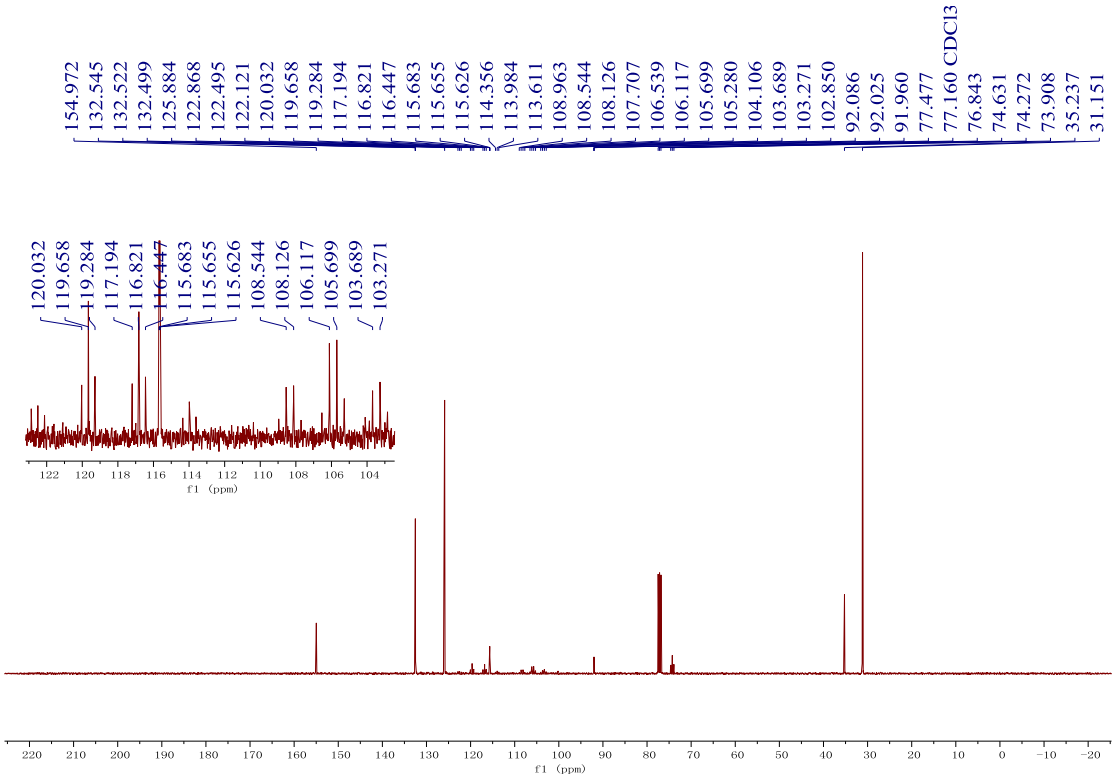
-101.339
2.94
2.00
f1 (ppm)


C_2F_5
4l ^{13}C NMR
(100 MHz, $CDCl_3$)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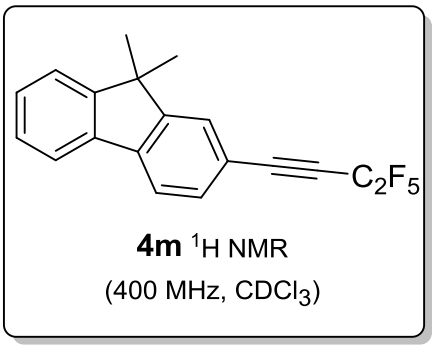
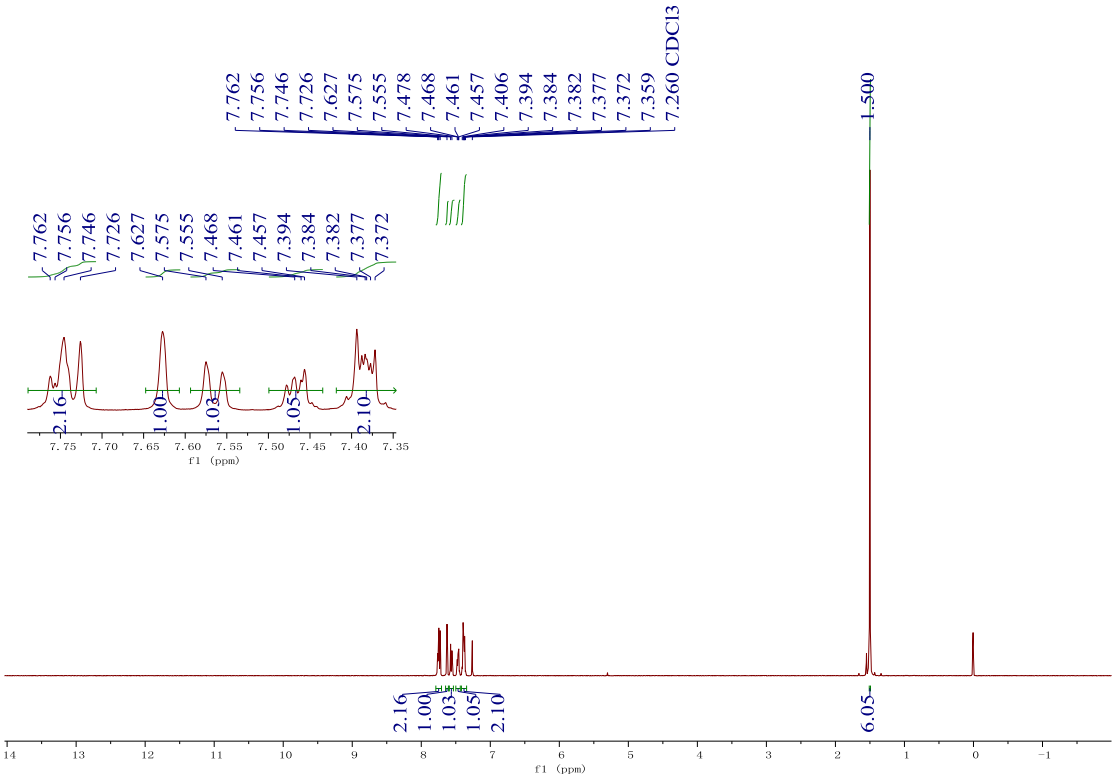
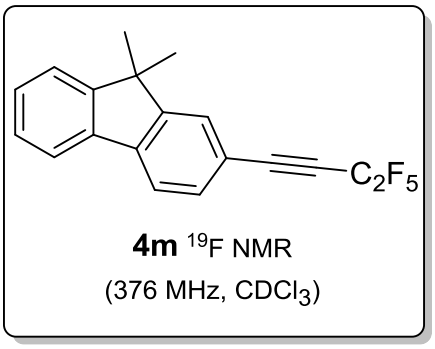
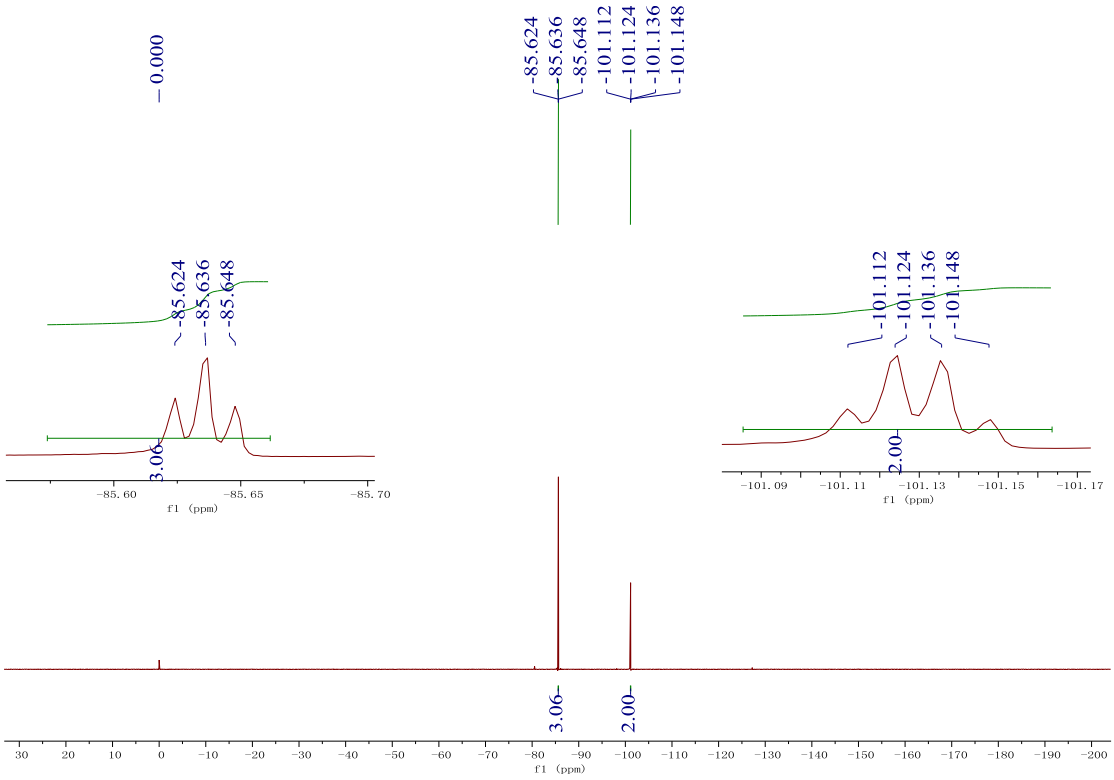
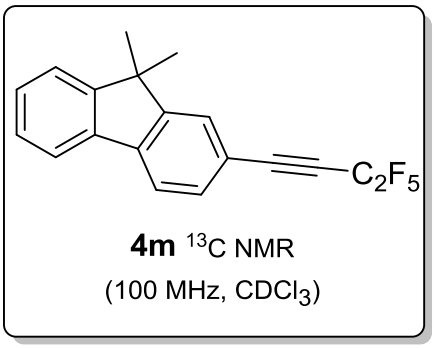
132.545
132.522
132.499
125.884
122.868
122.495
122.121
120.032
119.658
119.284
117.194
116.821
116.447
115.683
115.655
115.626
114.356
113.984
113.611
108.963
108.544
108.126
107.707
106.539
106.117
105.699
105.280
104.106
103.689
103.271
102.850
92.086
92.025
91.960
77.477
77.160 CDCl3
76.843
74.631
74.272
73.908
35.237
31.151
f1 (ppm)

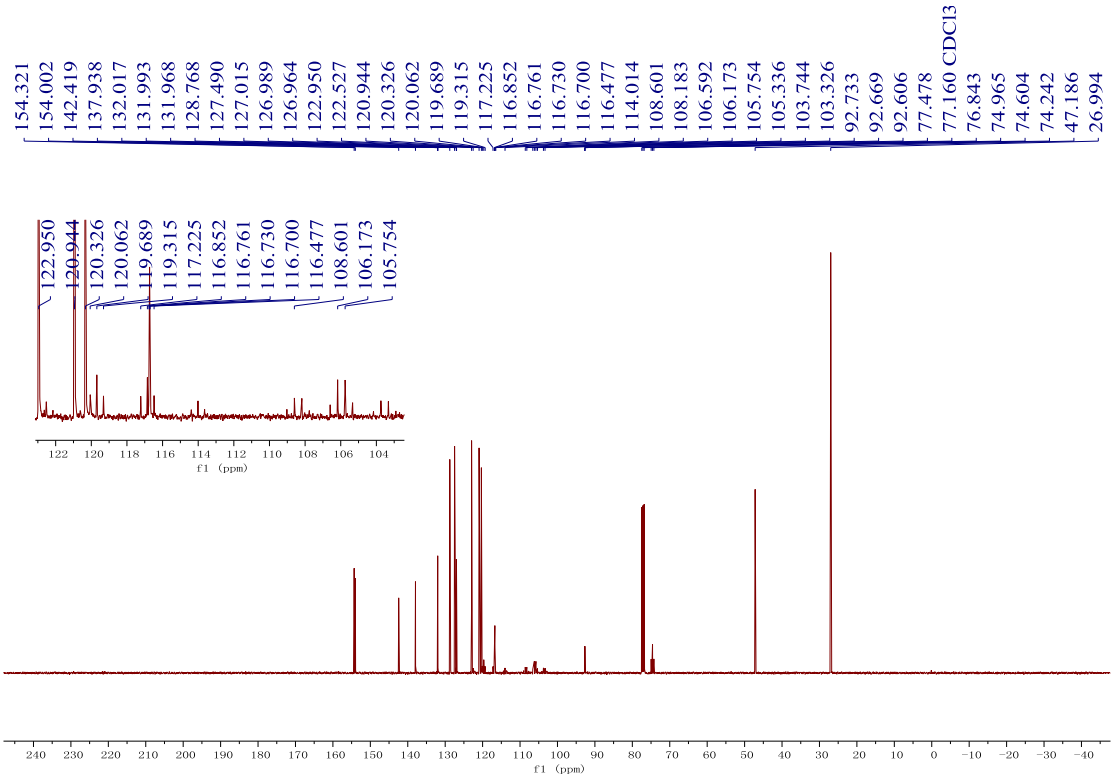

C_2F_5
4m ^{1}H NMR
(400 MHz, $CDCl_3$)

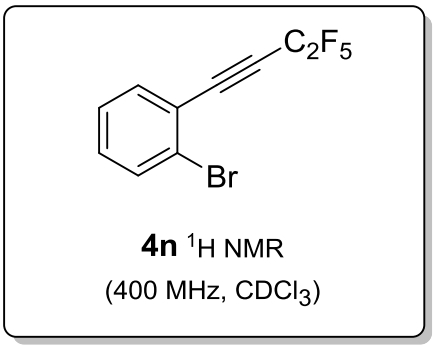

7.762
7.756
7.746
7.726
7.627
7.575
7.555
7.478
7.468
7.461
7.457
7.406
7.394
7.384
7.382
7.377
7.372
7.359
7.260 CDCl3
1.500
2.16
1.00
1.03
1.05
2.10
6.05
f1 (ppm)


C2F5
4m 19F NMR
(376 MHz, CDCl3)


0.000
-85.624
-85.636
-85.648
-101.112
-101.124
-101.136
-101.148
3.06
2.00
f1 (ppm)

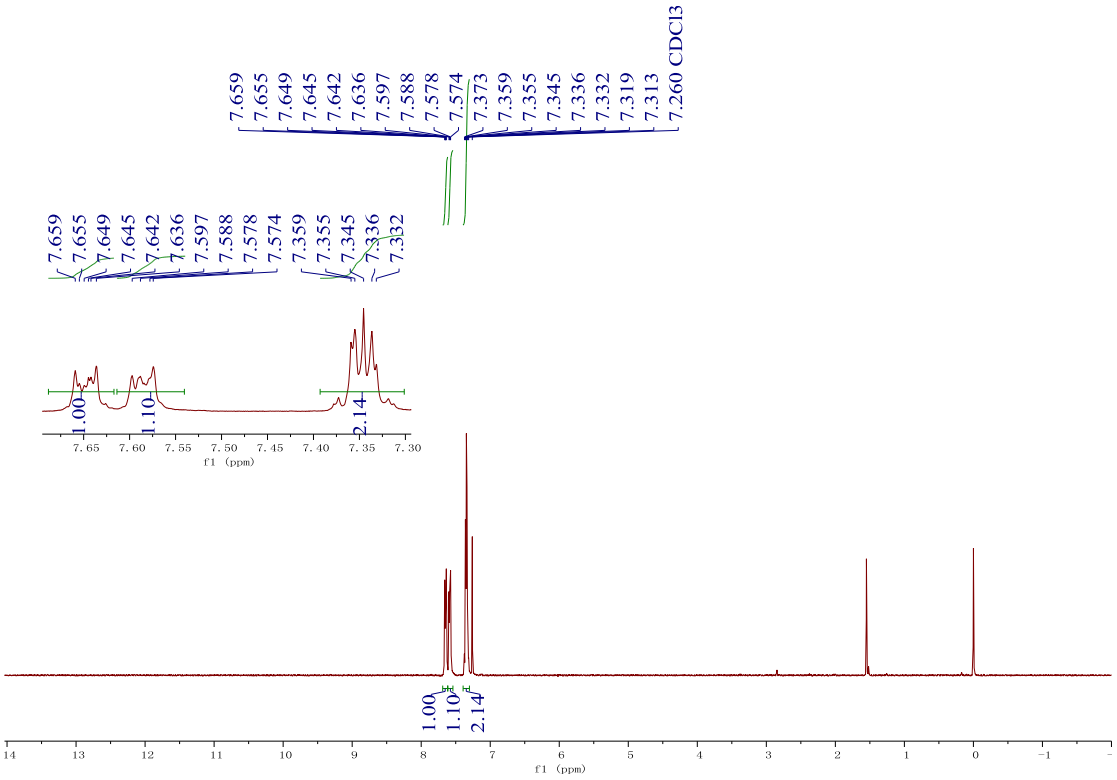

C_2F_5
4m ^{13}C NMR
(100 MHz, $CDCl_3$)

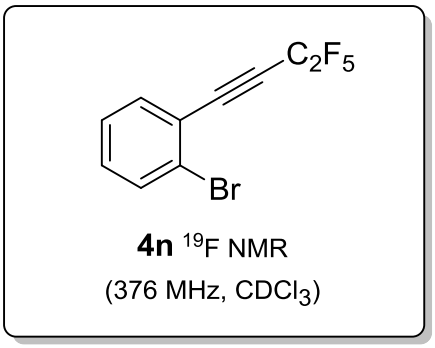

154.321
154.002
142.419
137.938
132.017
131.993
131.968
128.768
127.490
127.015
126.989
126.964
122.950
122.527
120.944
120.326
120.062
119.689
119.315
117.225
116.852
116.761
116.730
116.700
116.477
114.014
108.601
108.183
106.592
106.173
105.754
105.336
103.744
103.326
92.733
92.669
92.606
77.478
77.160 CDCl3
76.843
74.965
74.604
74.242
47.186
26.994
f1 (ppm)



4n ^{1}H NMR

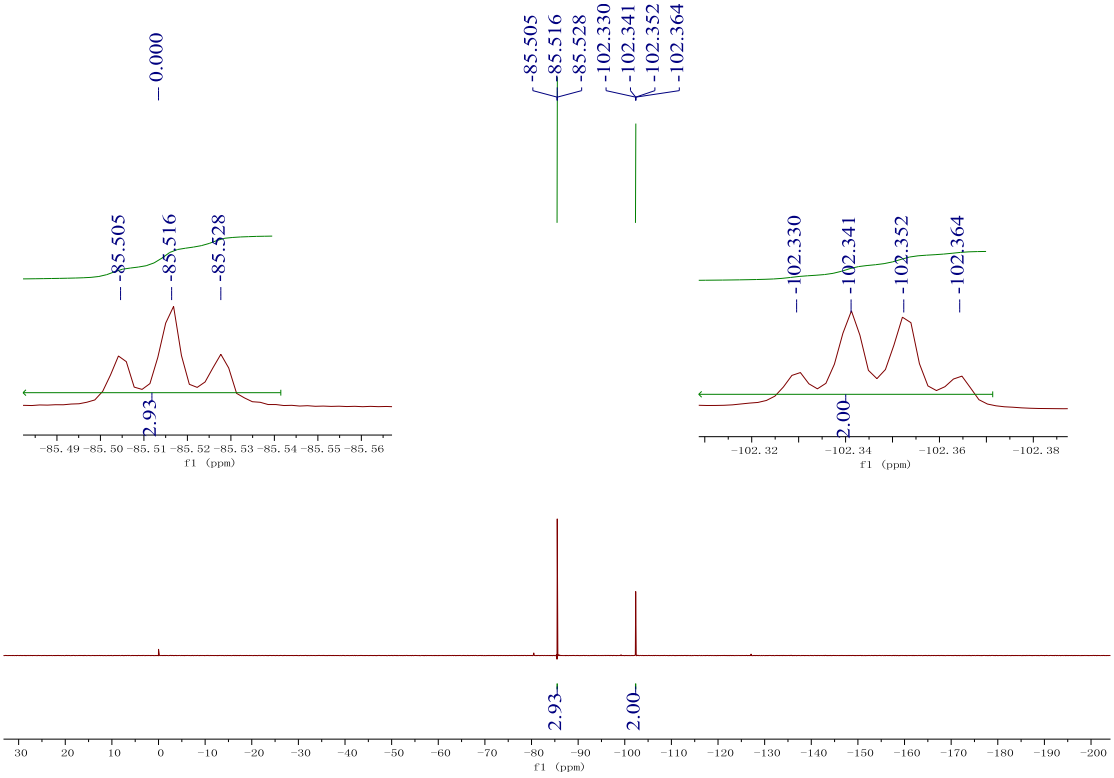
(400 MHz, $CDCl_3$)

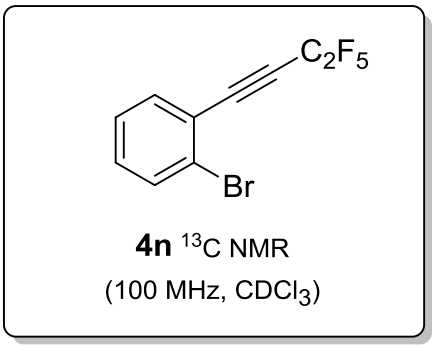




4n ^{19}F NMR

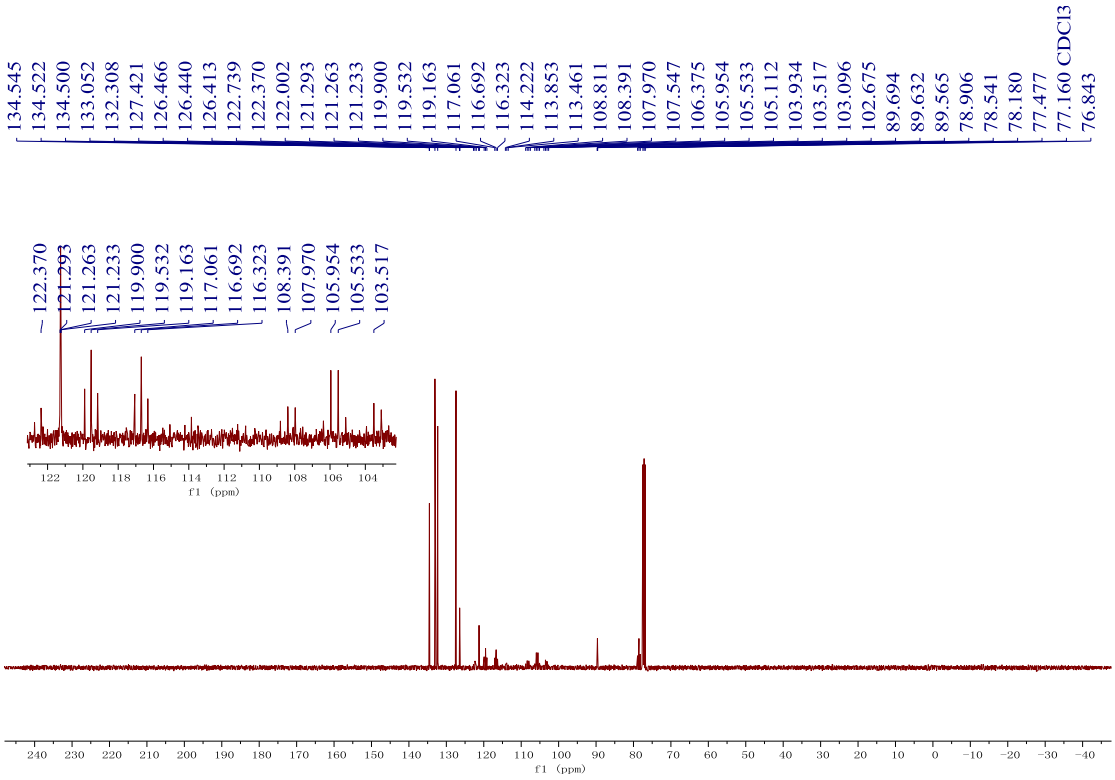
(376 MHz, $CDCl_3$)

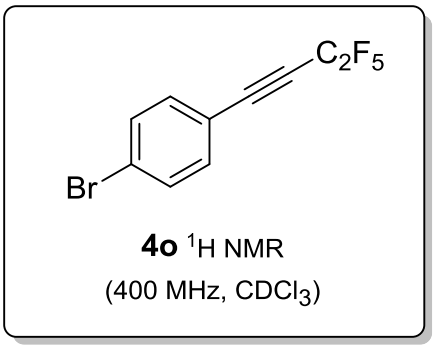


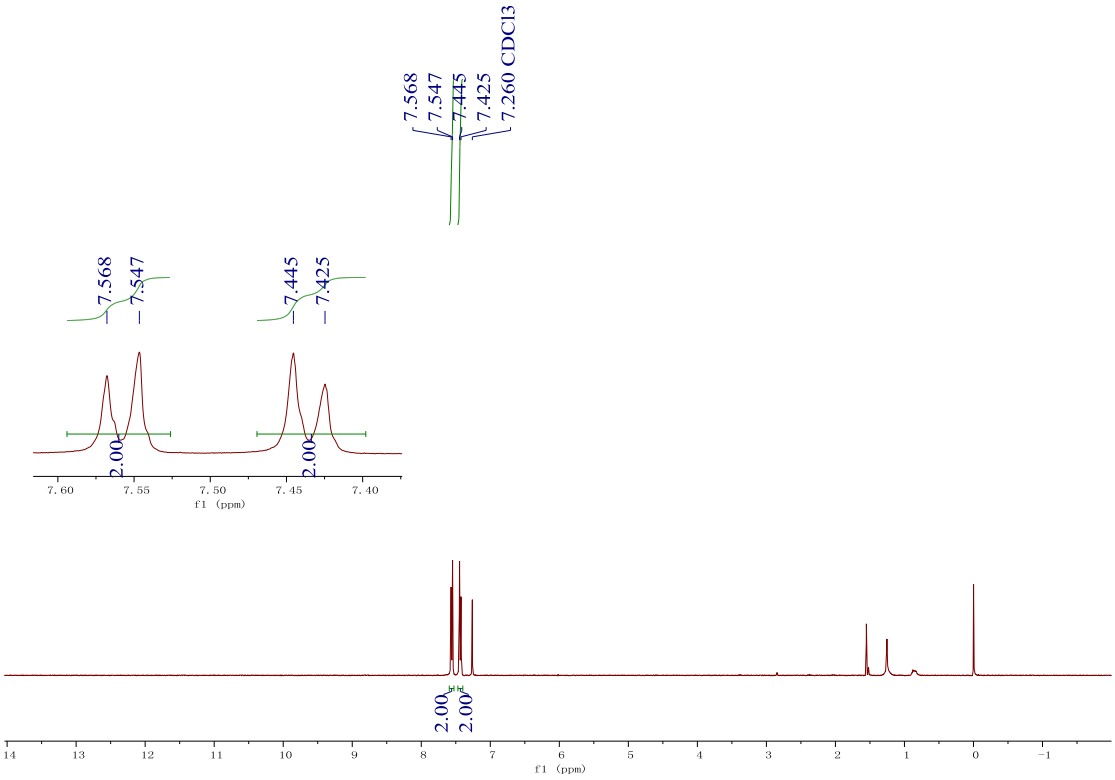


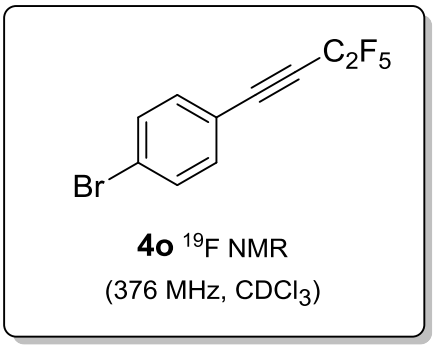
4n ^{13}C NMR

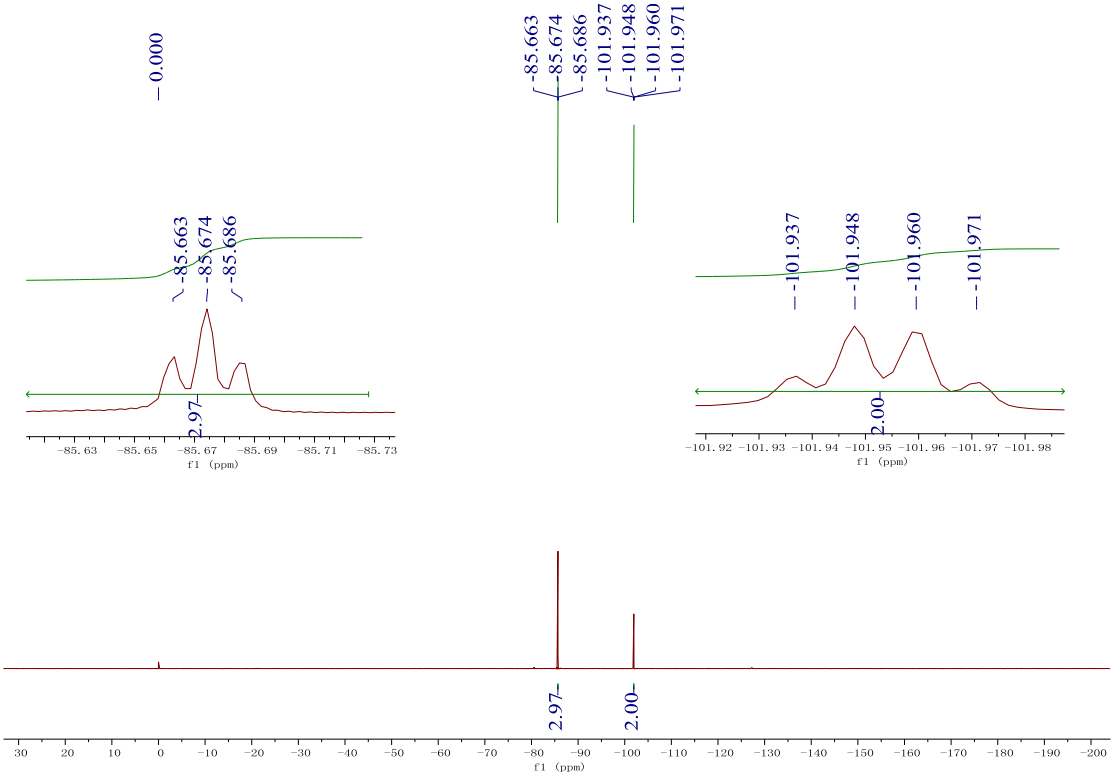
(100 MHz, $CDCl_3$)

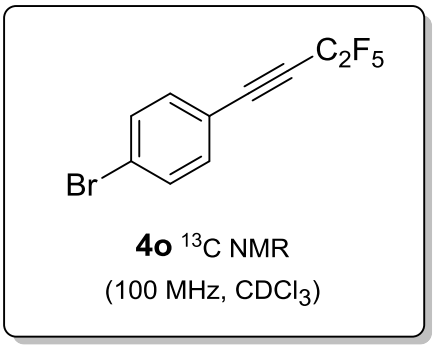



C2F5
Br
4o 1H NMR
(400 MHz, CDCl3)


7.568
7.547
7.445
7.425
7.260 CDCl3
2.00
2.00
f1 (ppm)
2.00
2.00
f1 (ppm)

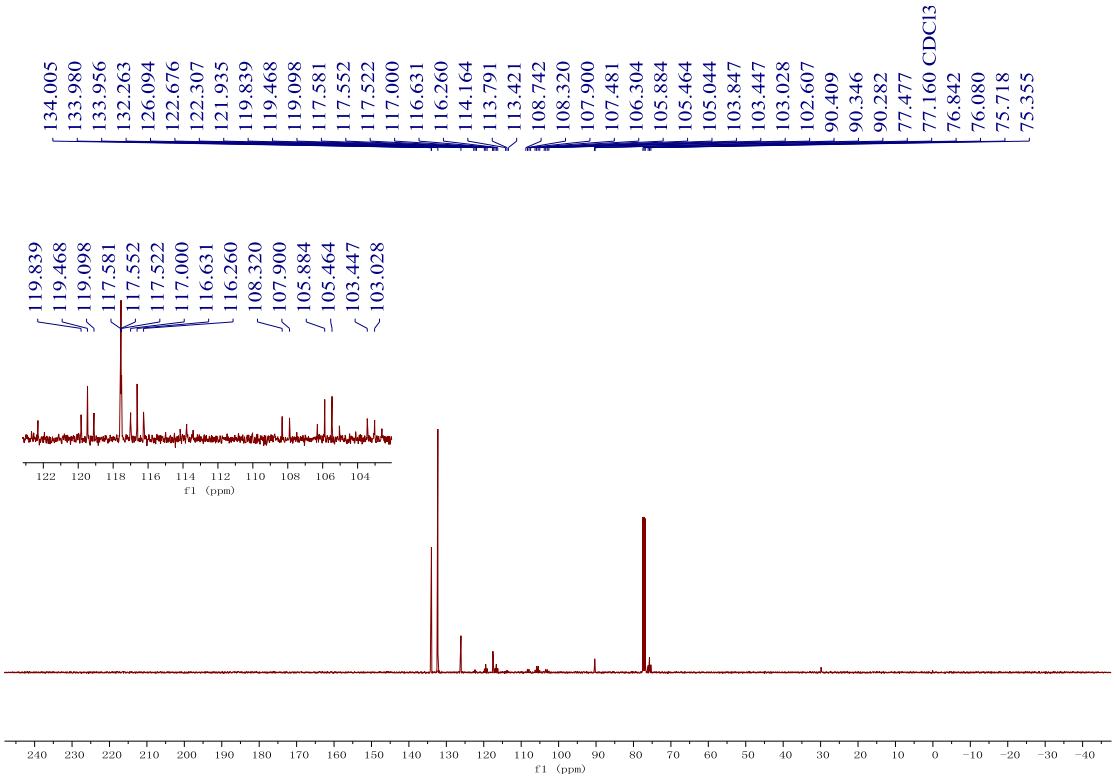

C_2F_5
Br
4o ^{19}F NMR
(376 MHz, $CDCl_3$)

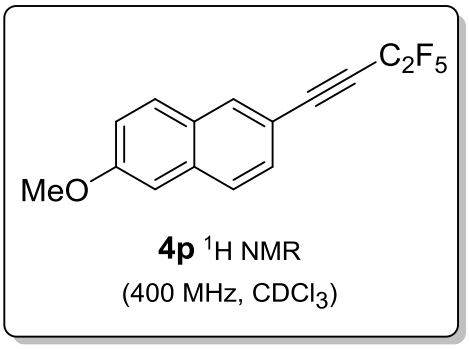

-0.000
-85.663
-85.674
-85.686
-101.937
-101.948
-101.960
-101.971
2.97
2.00
f1 (ppm)



4o ^{13}C NMR

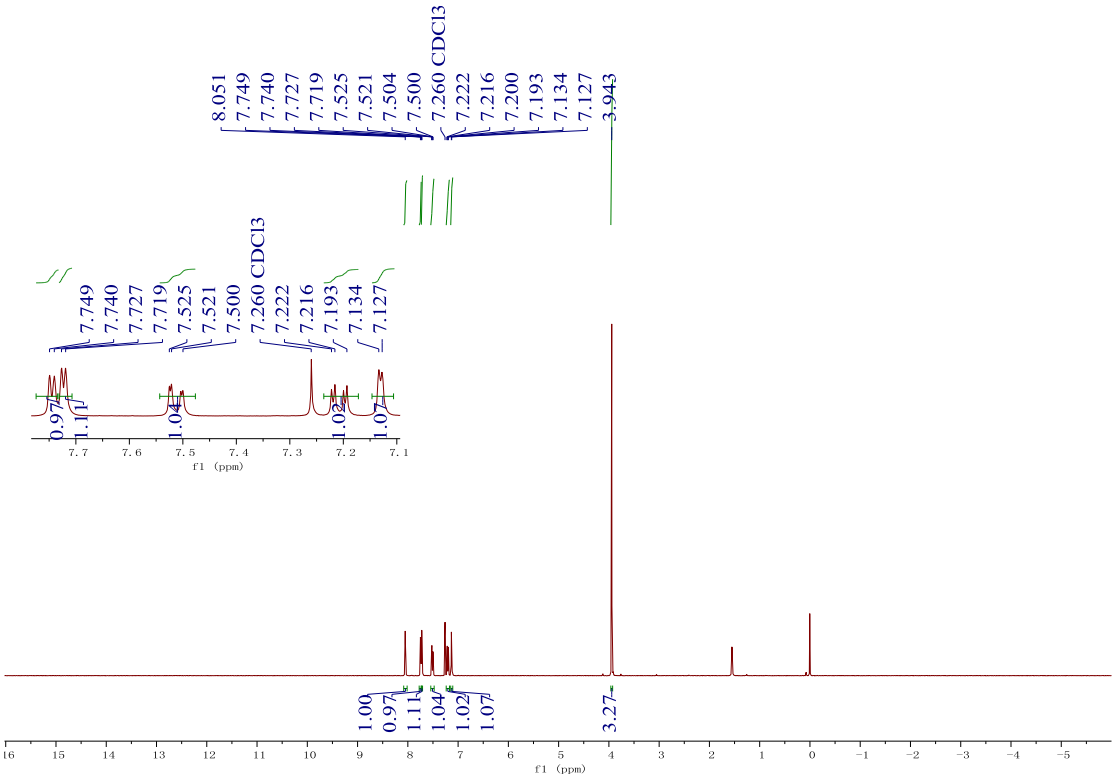
(100 MHz, $CDCl_3$)

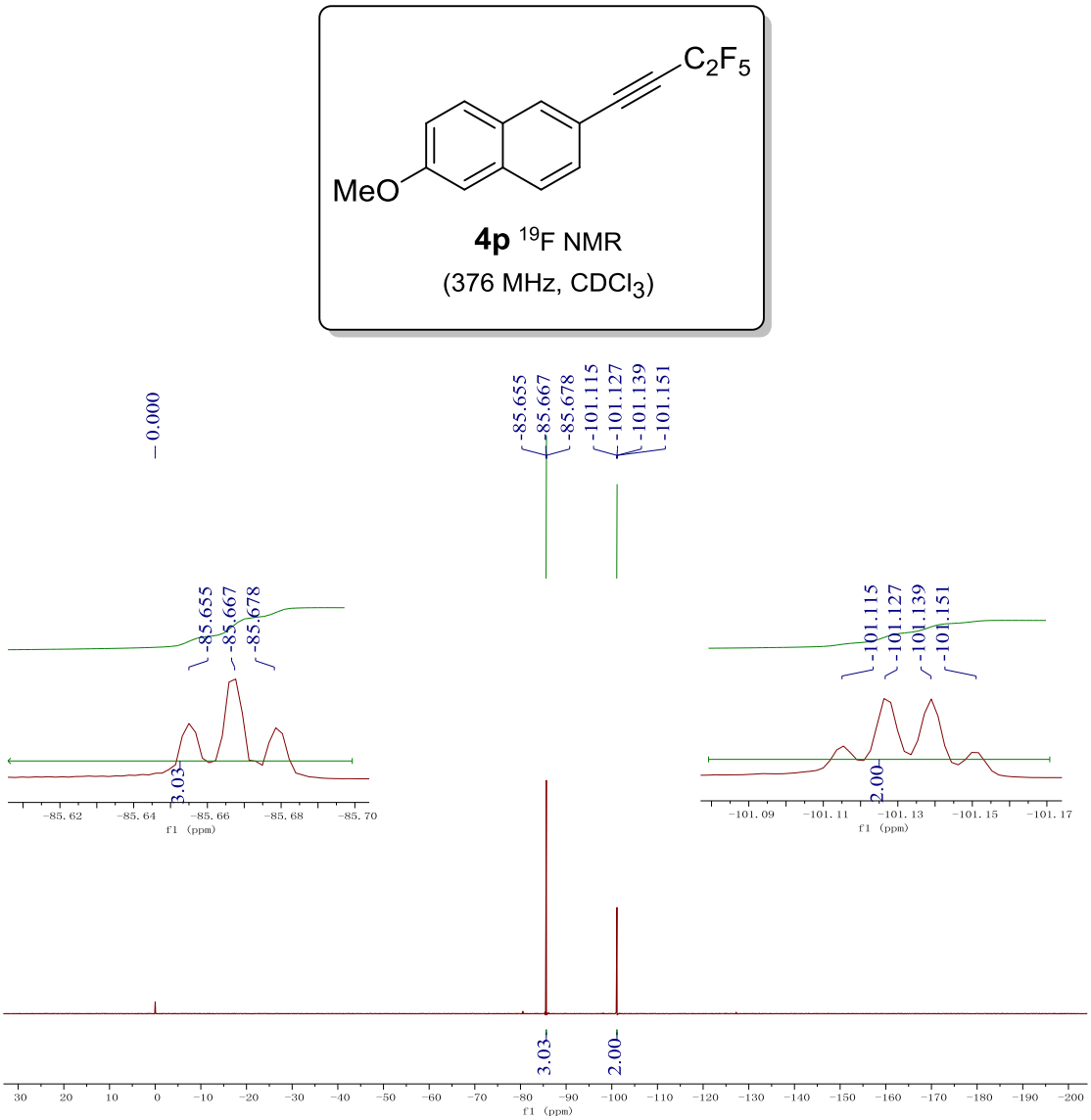


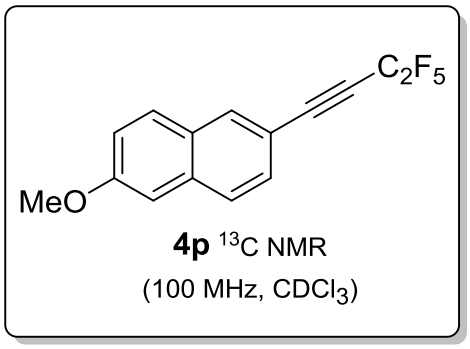


4p ^{1}H NMR

(400 MHz, $CDCl_3$)

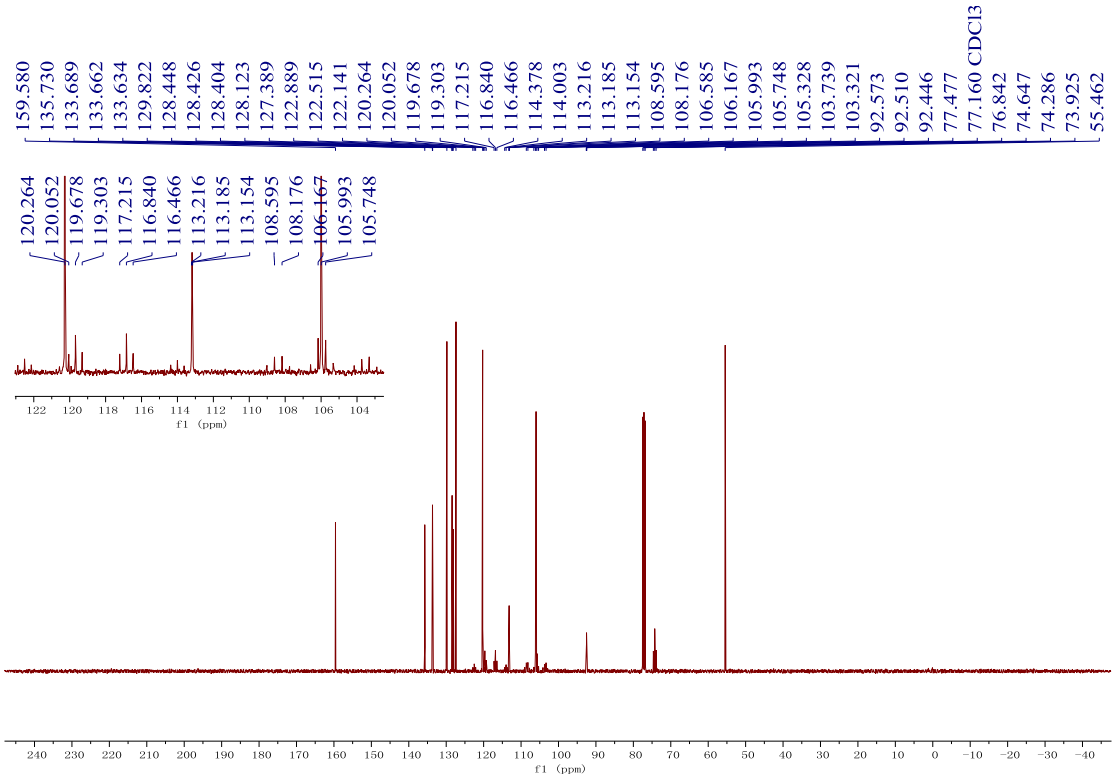


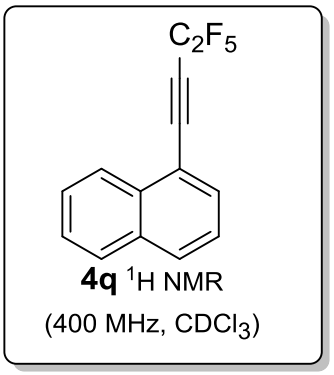

C2F5
MeO
4p 19F NMR
(376 MHz, CDCl3)
-0.000
-85.655
-85.667
-85.678
-101.115
-101.127
-101.139
-101.151
3.03
2.00
f1 (ppm)

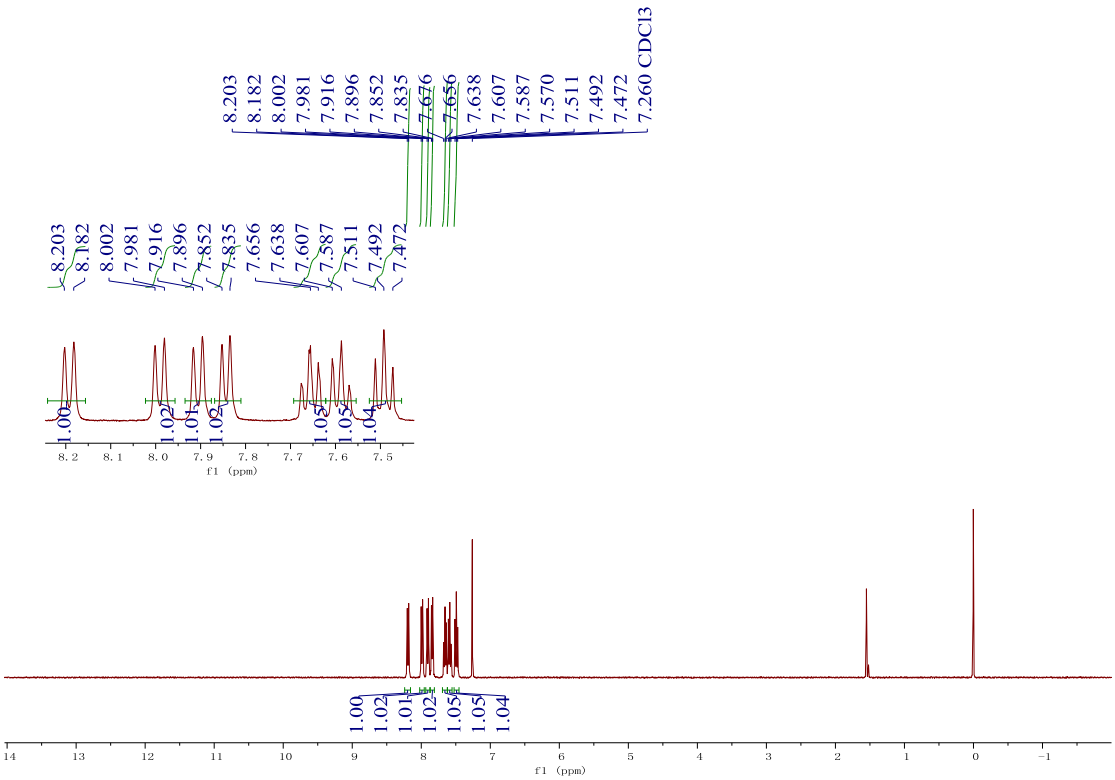


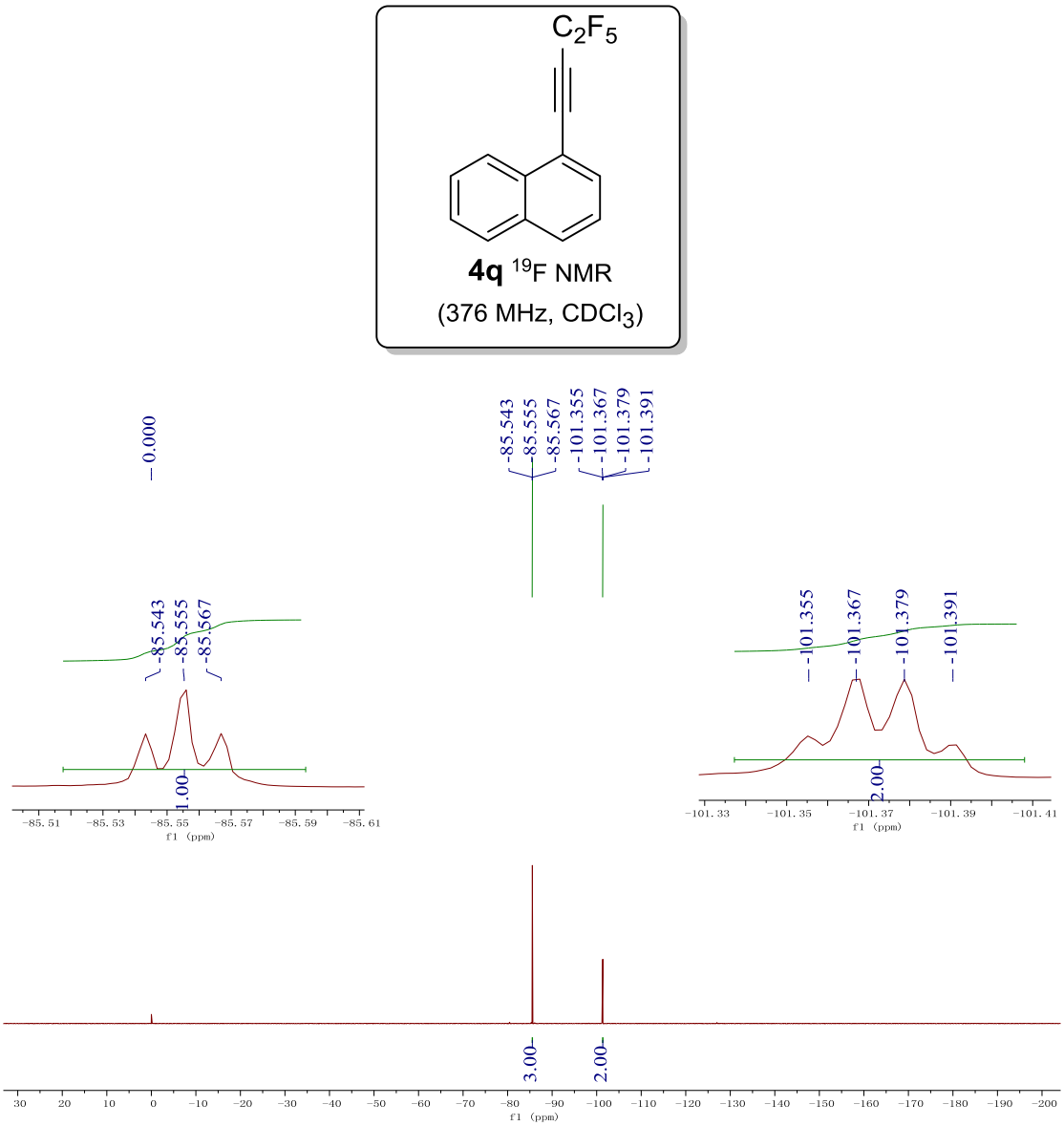
4p ^{13}C NMR

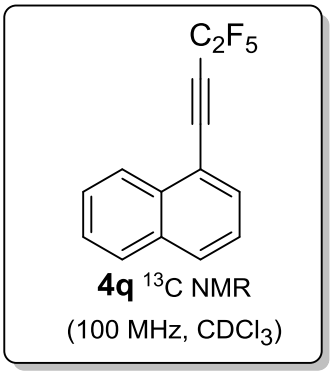
(100 MHz, $CDCl_3$)

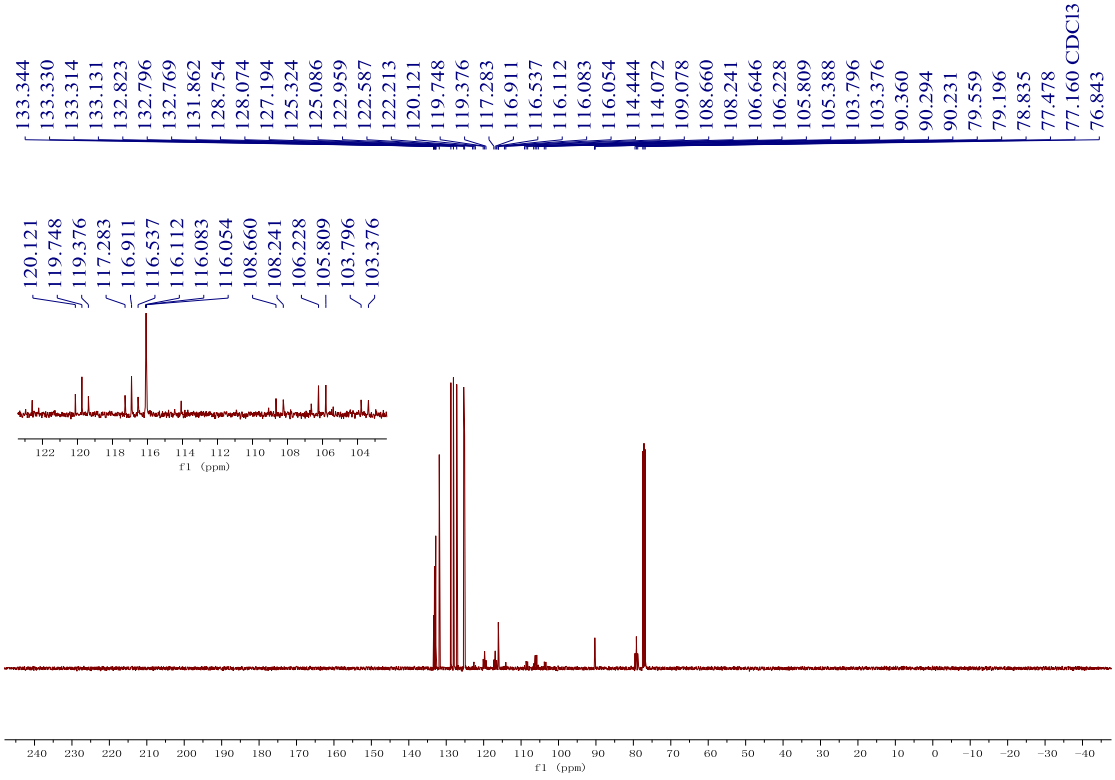


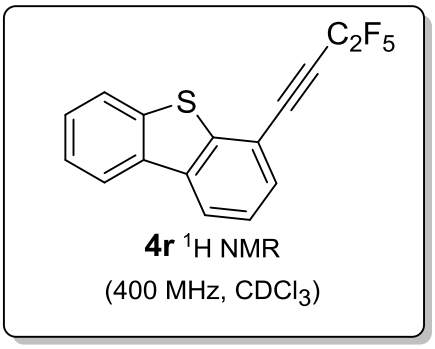

C_2F_5
4q ^{1}H NMR
(400 MHz, $CDCl_3$)

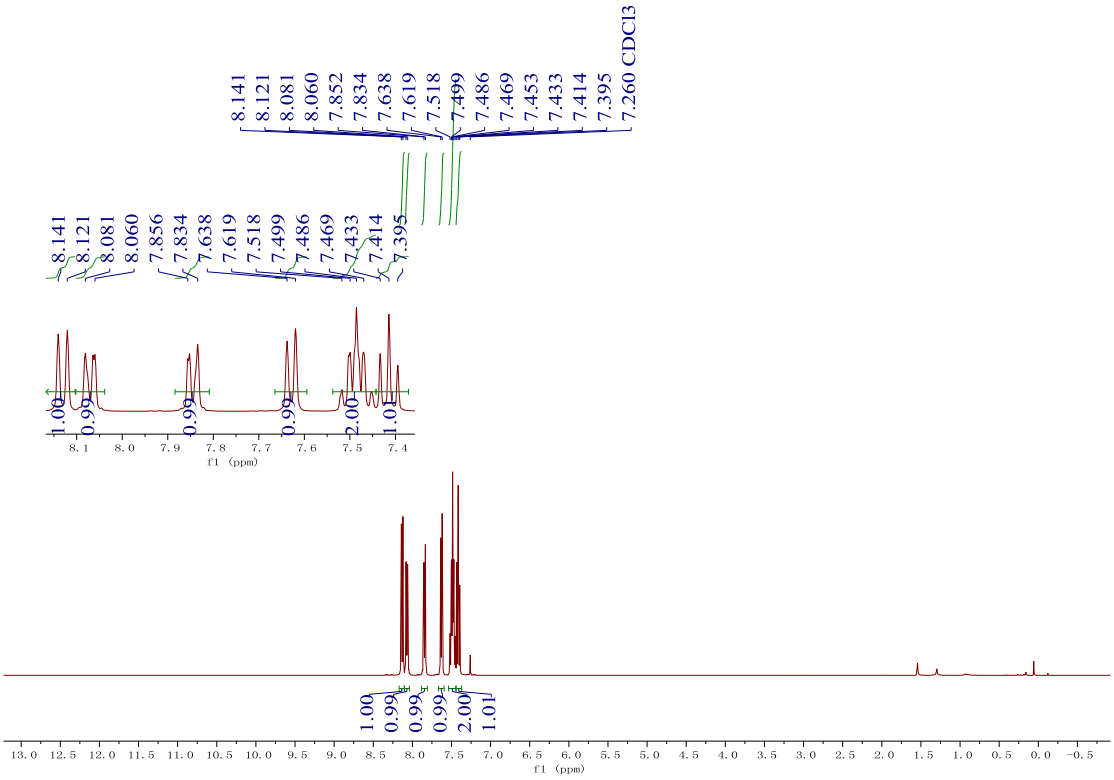

8.203
8.182
8.002
7.981
7.916
7.896
7.852
7.835
7.676
7.656
7.638
7.607
7.587
7.570
7.511
7.492
7.472
7.260 CDCl3
1.00
1.02
1.01
1.02
1.05
1.05
1.04
f1 (ppm)

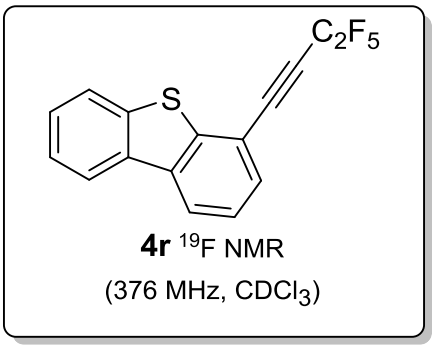

C2F5
4q 19F NMR
(376 MHz, CDCl3)
-0.000
-85.543
-85.555
-85.567
-101.355
-101.367
-101.379
-101.391
1.00
2.00
3.00
2.00
f1 (ppm)


C_2F_5
4q ^{13}C NMR
(100 MHz, $CDCl_3$)


133.344
133.330
133.314
133.131
132.823
132.796
132.769
131.862
128.754
128.074
127.194
125.324
125.086
122.959
122.587
122.213
120.121
119.748
119.376
117.283
116.911
116.537
116.112
116.083
116.054
114.444
114.072
109.078
108.660
108.241
106.646
106.228
105.809
105.388
103.796
103.376
90.360
90.294
90.231
79.559
79.196
78.835
77.478
77.160 CDCl3
76.843
120.121
119.748
119.376
117.283
116.911
116.537
116.112
116.083
116.054
108.660
108.241
106.228
105.809
103.796
103.376
122 120 118 116 114 112 110 108 106 104
f1 (ppm)
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40
f1 (ppm)

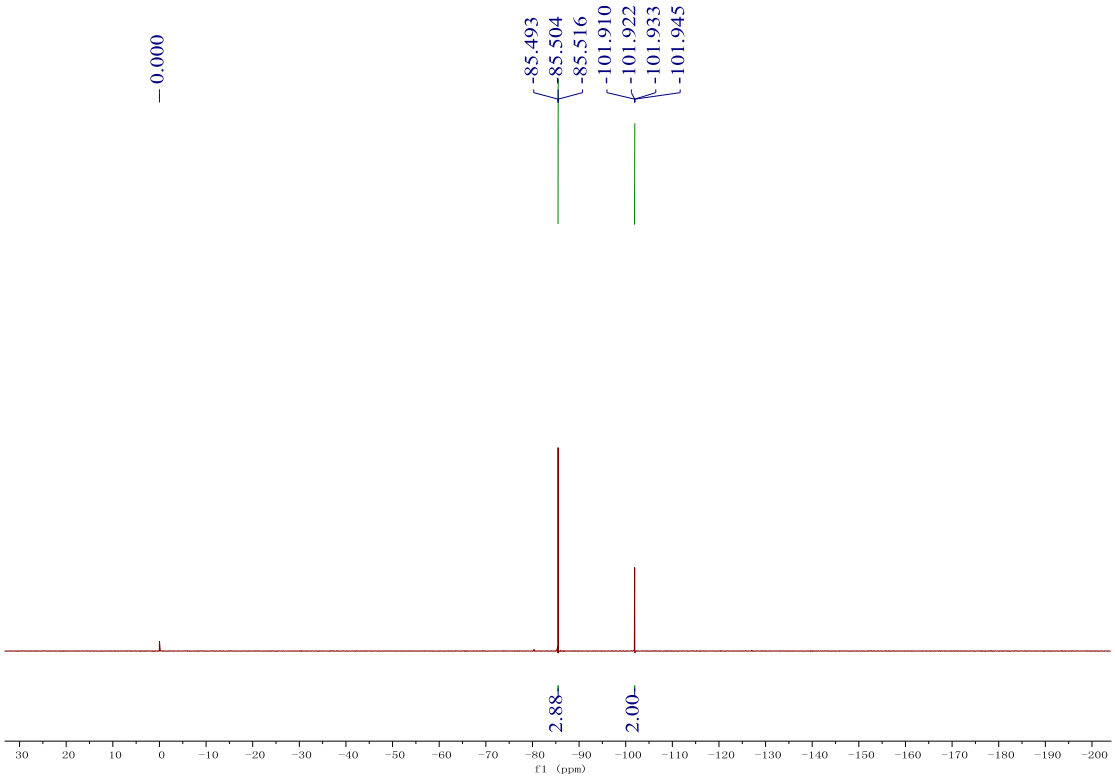

C2F5
S
4r 1H NMR
(400 MHz, CDCl3)

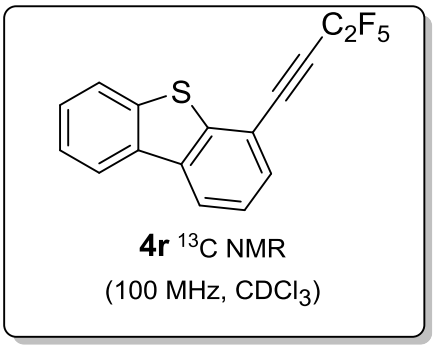

8.141
8.121
8.081
8.060
7.852
7.834
7.638
7.619
7.518
7.499
7.486
7.469
7.453
7.433
7.414
7.395
7.260 CDCl3
1.00
0.99
0.99
0.99
2.00
1.01
f1 (ppm)

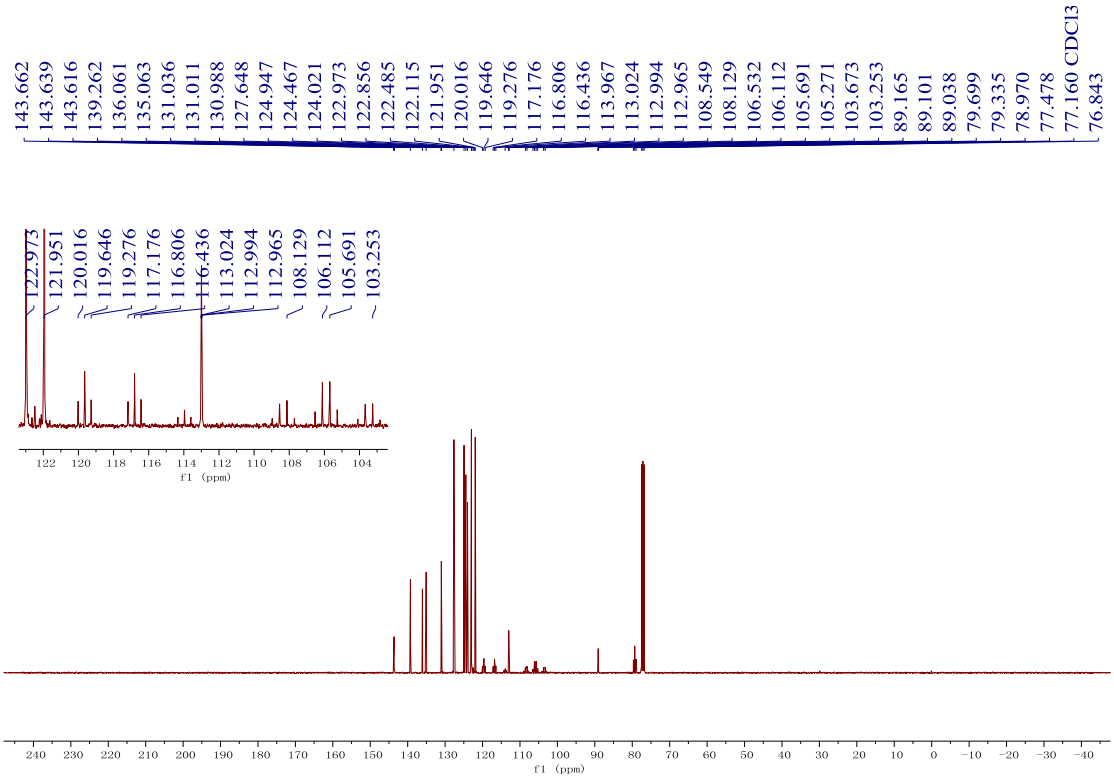


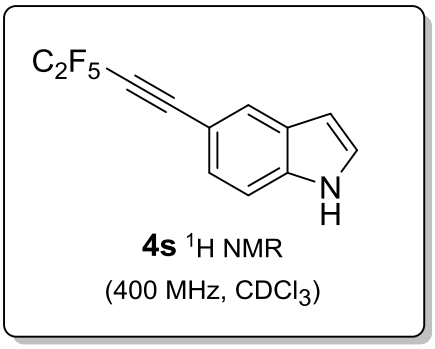
4r ^{19}F NMR

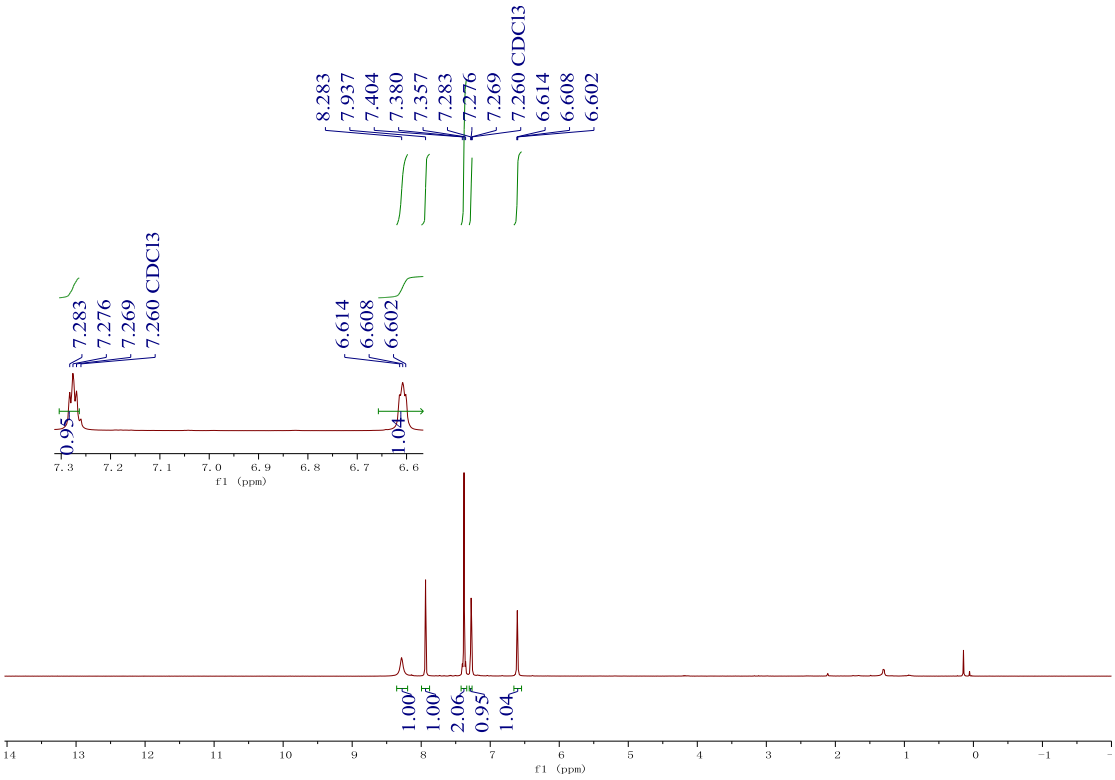
(376 MHz, $CDCl_3$)

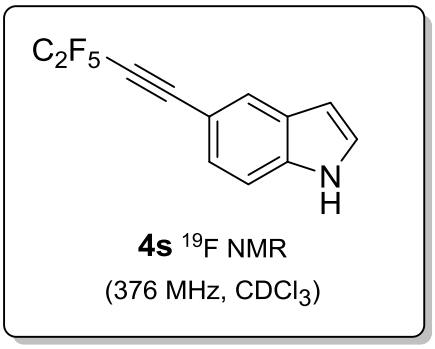



C2F5
S
4r 13C NMR
(100 MHz, CDCl3)


143.662
143.639
143.616
139.262
136.061
135.063
131.036
131.011
130.988
127.648
124.947
124.467
124.021
122.973
122.856
122.485
122.115
121.951
120.016
119.646
119.276
117.176
116.806
116.436
113.967
113.024
112.994
112.965
108.549
108.129
106.532
106.112
105.691
105.271
103.673
103.253
89.165
89.101
89.038
79.699
79.335
78.970
77.478
77.160 CDCl3
76.843
122.973
121.951
120.016
119.646
119.276
117.176
116.806
116.436
113.024
112.994
112.965
108.129
106.112
105.691
103.253
f1 (ppm)
f1 (ppm)

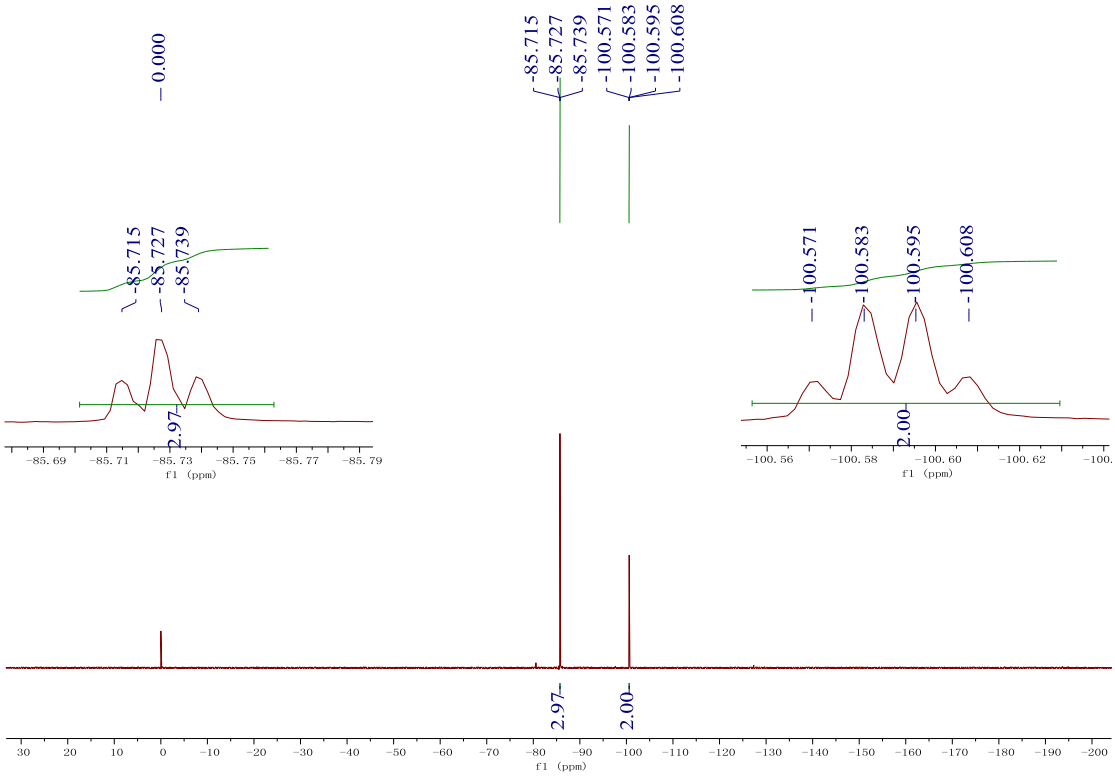

C2F5
N
H
4s 1H NMR
(400 MHz, CDCl3)

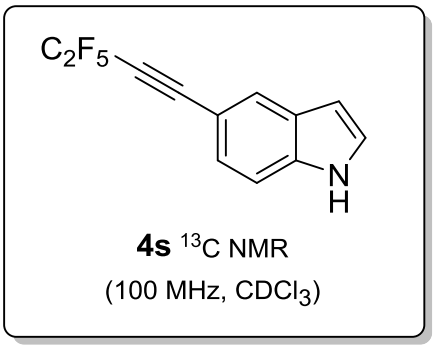

8.283
7.937
7.404
7.380
7.357
7.283
7.276
7.269
7.260 CDCl3
6.614
6.608
6.602
0.95
1.04
1.00
1.00
2.06
0.95
1.04
f1 (ppm)



4s ^{19}F NMR

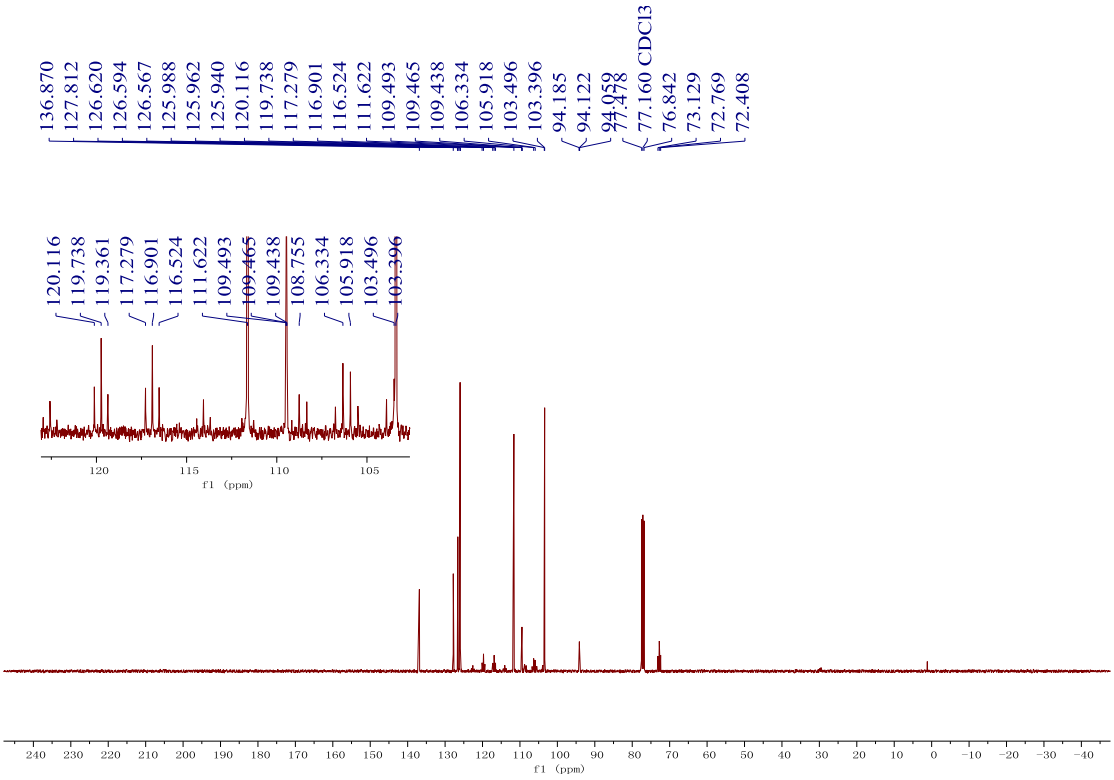
(376 MHz, $CDCl_3$)

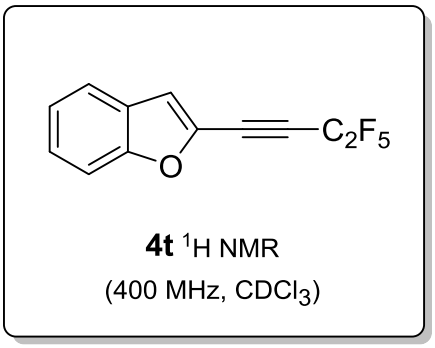


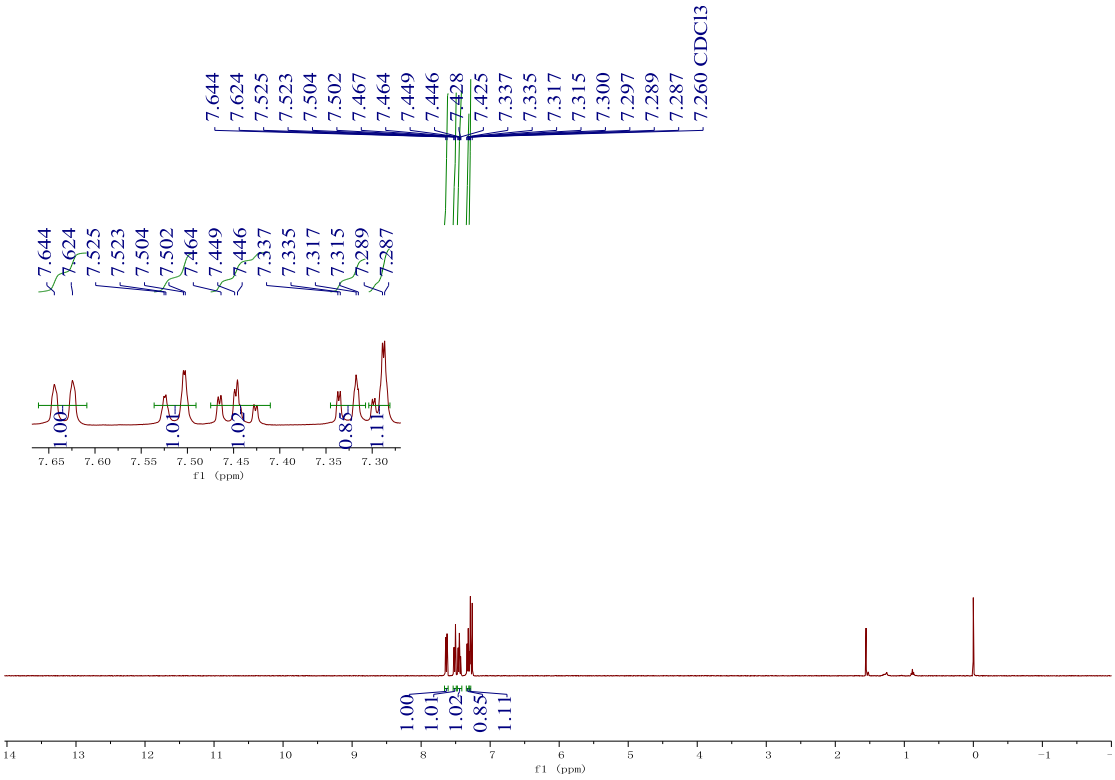


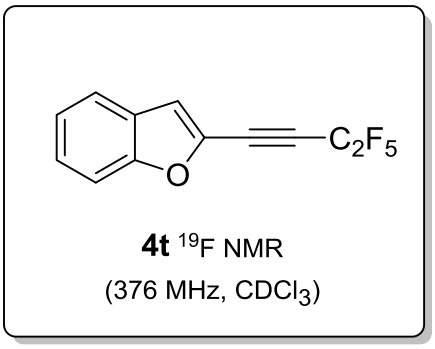
4s ^{13}C NMR

(100 MHz, $CDCl_3$)



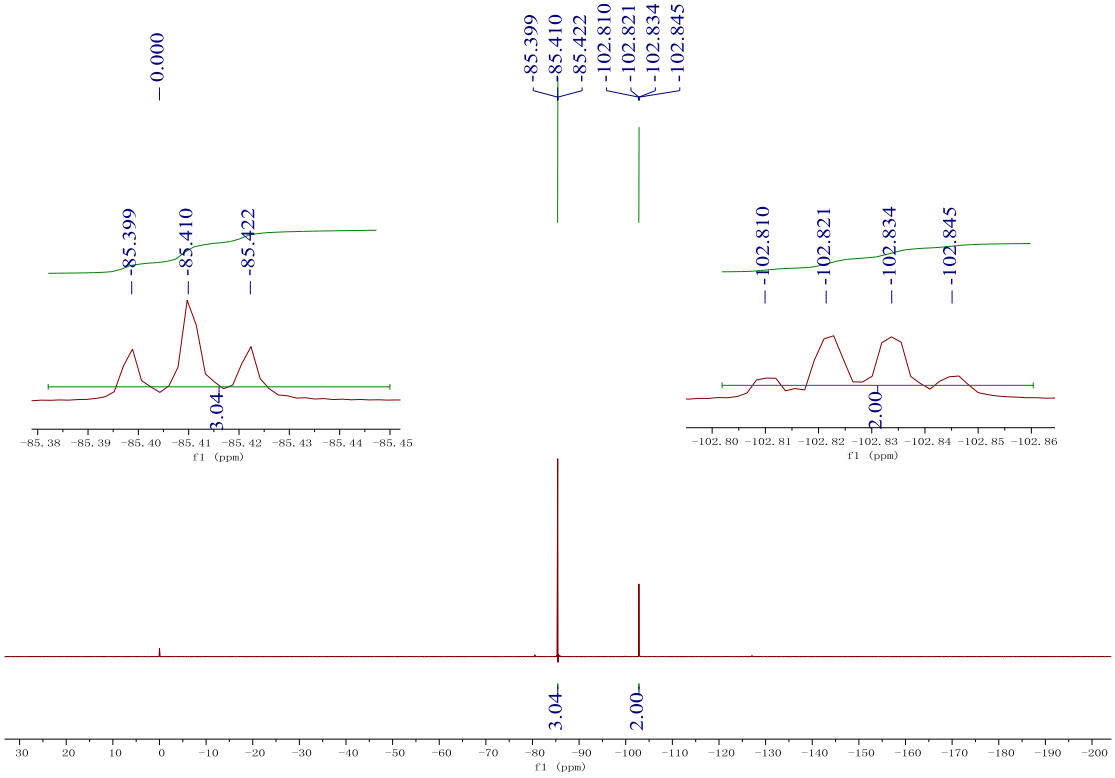

C2F5
O
4t 1H NMR
(400 MHz, CDCl3)

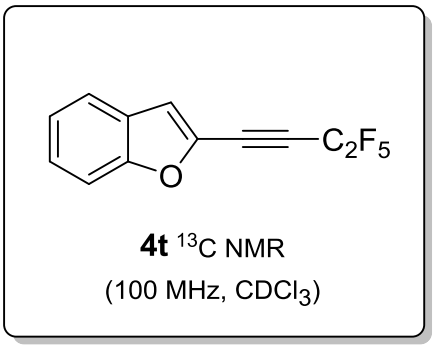

7.644
7.624
7.525
7.523
7.504
7.502
7.467
7.464
7.449
7.446
7.428
7.425
7.337
7.335
7.317
7.315
7.300
7.297
7.289
7.287
7.260 CDCl3
1.00
1.01
1.02
0.85
1.11
f1 (ppm)



4t ^{19}F NMR

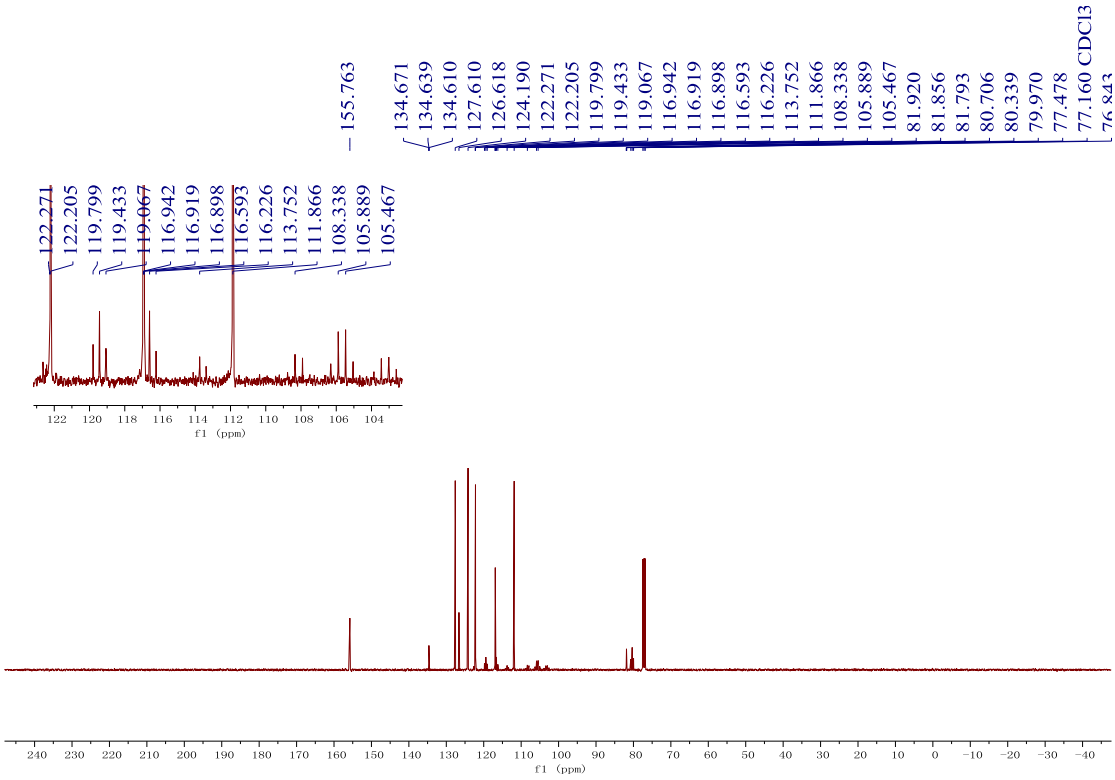
(376 MHz, $CDCl_3$)

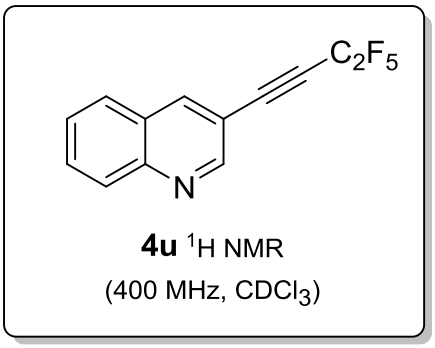




4t ^{13}C NMR

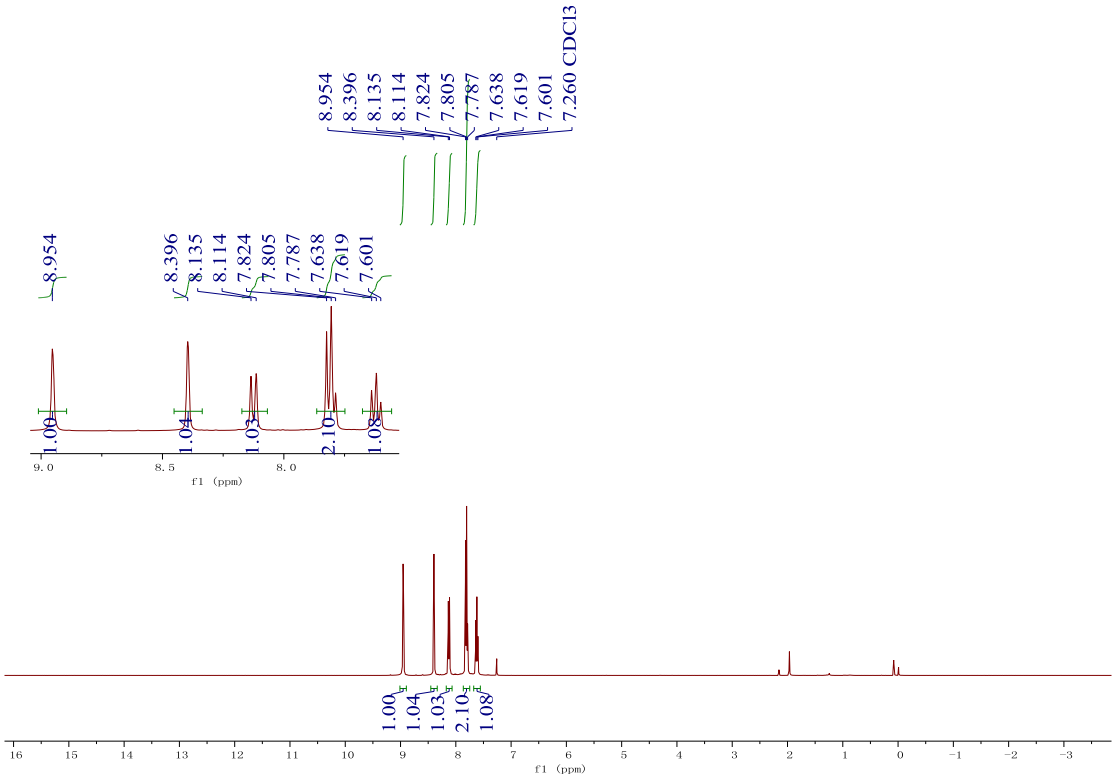
(100 MHz, $CDCl_3$)

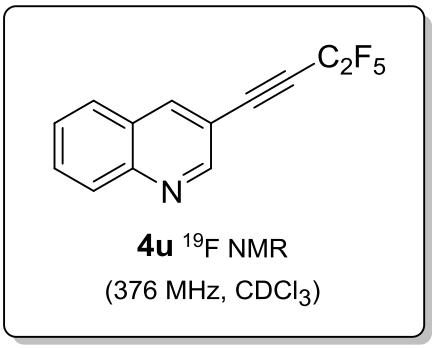




4u ^{1}H NMR

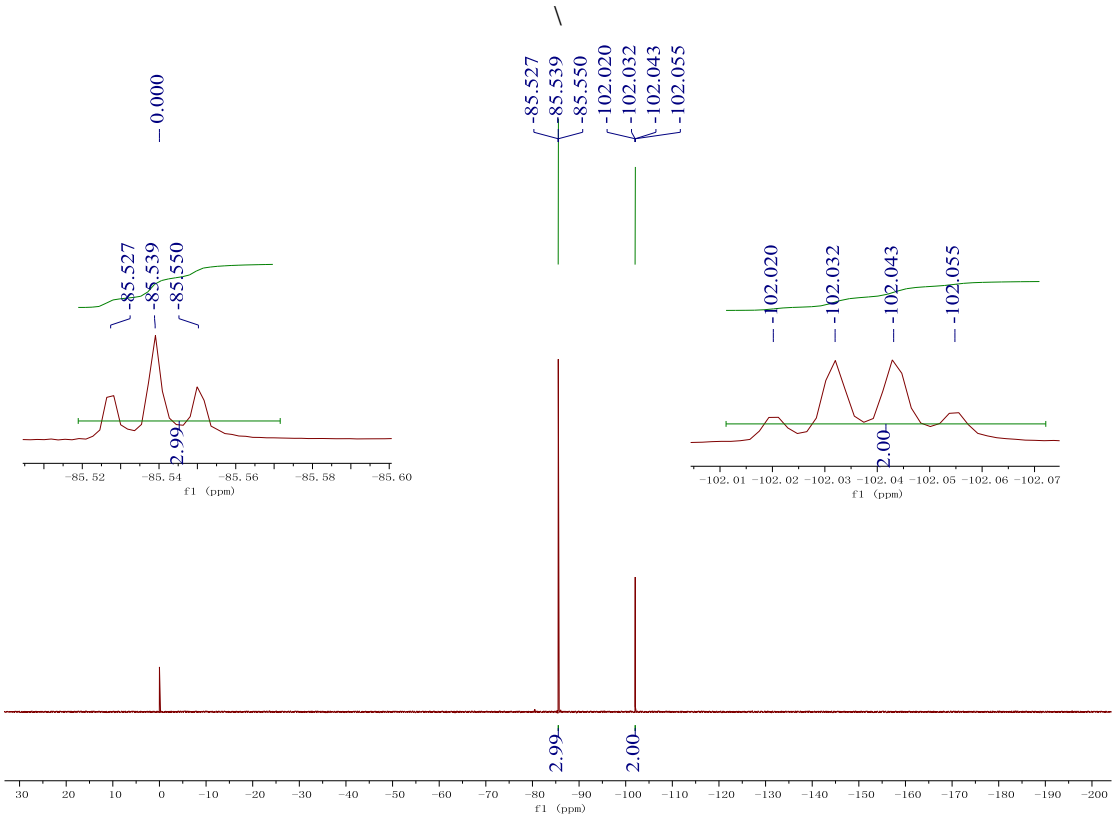
(400 MHz, $CDCl_3$)

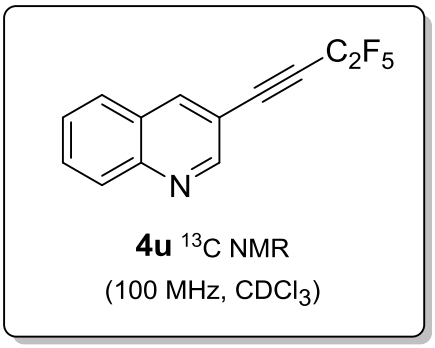




4u ^{19}F NMR

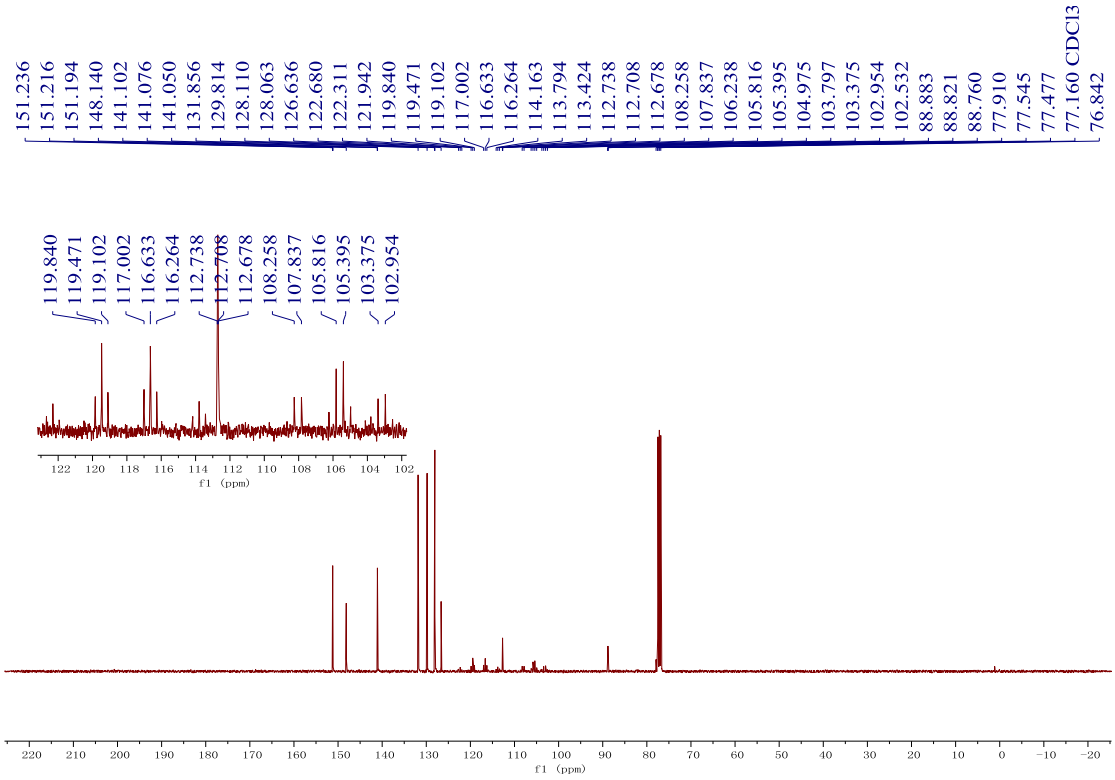
(376 MHz, $CDCl_3$)

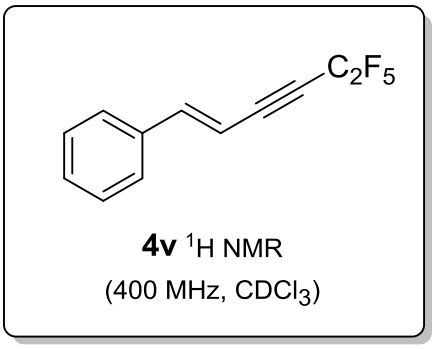


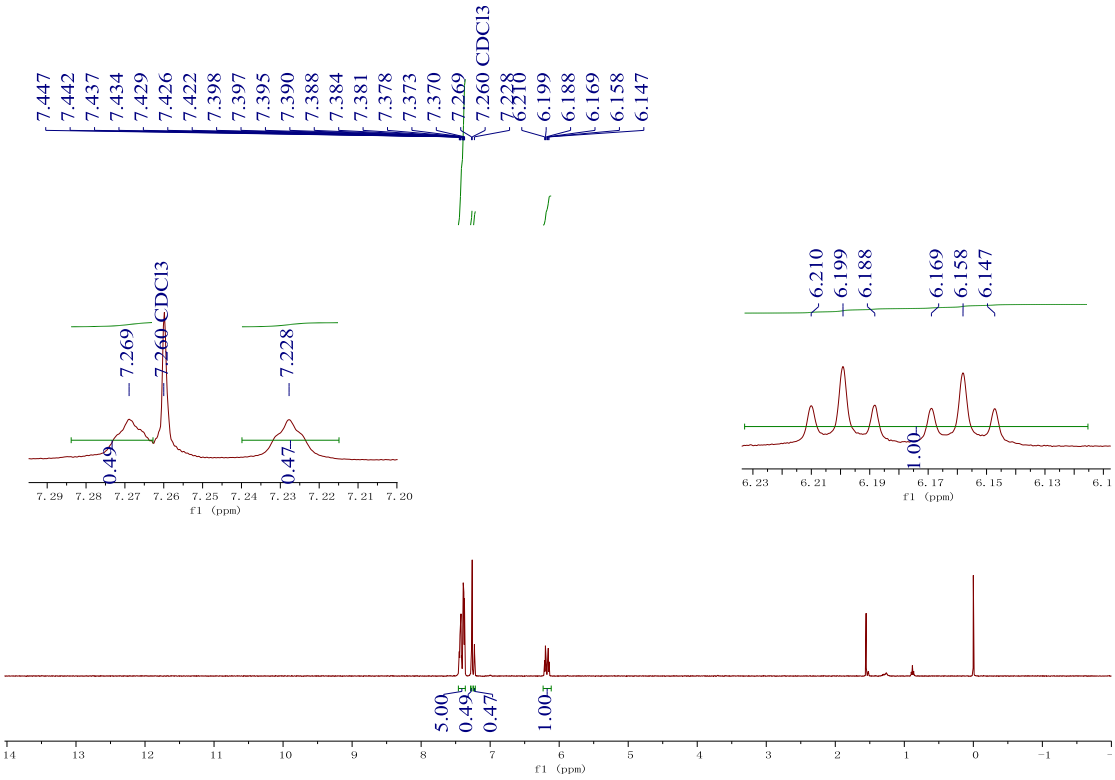


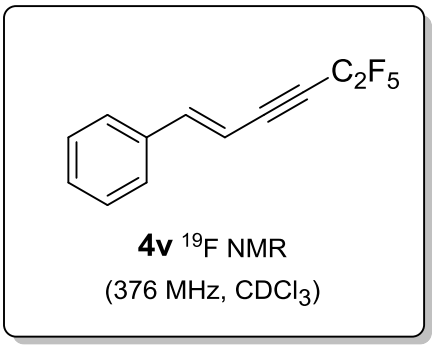
4u ^{13}C NMR

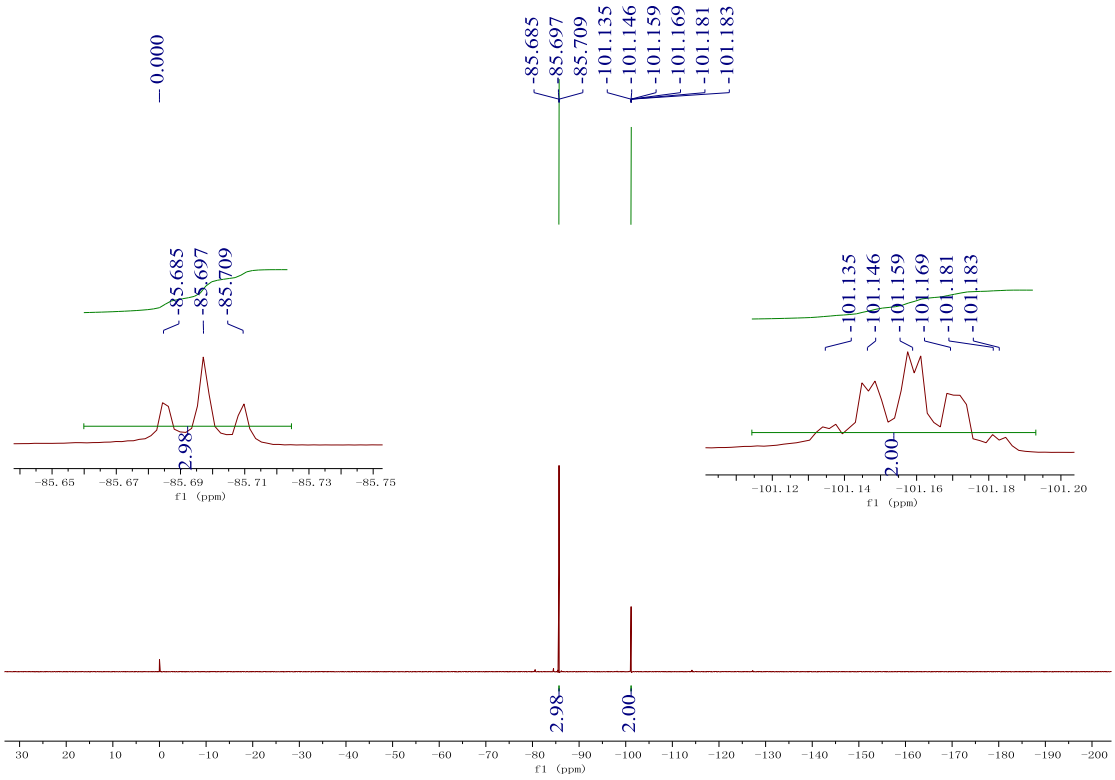
(100 MHz, $CDCl_3$)

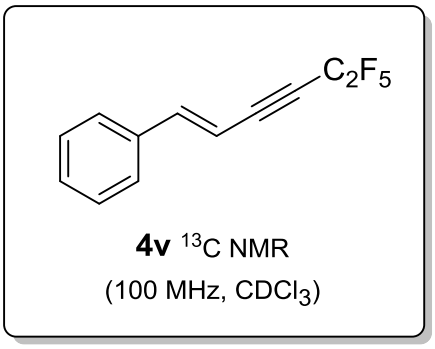


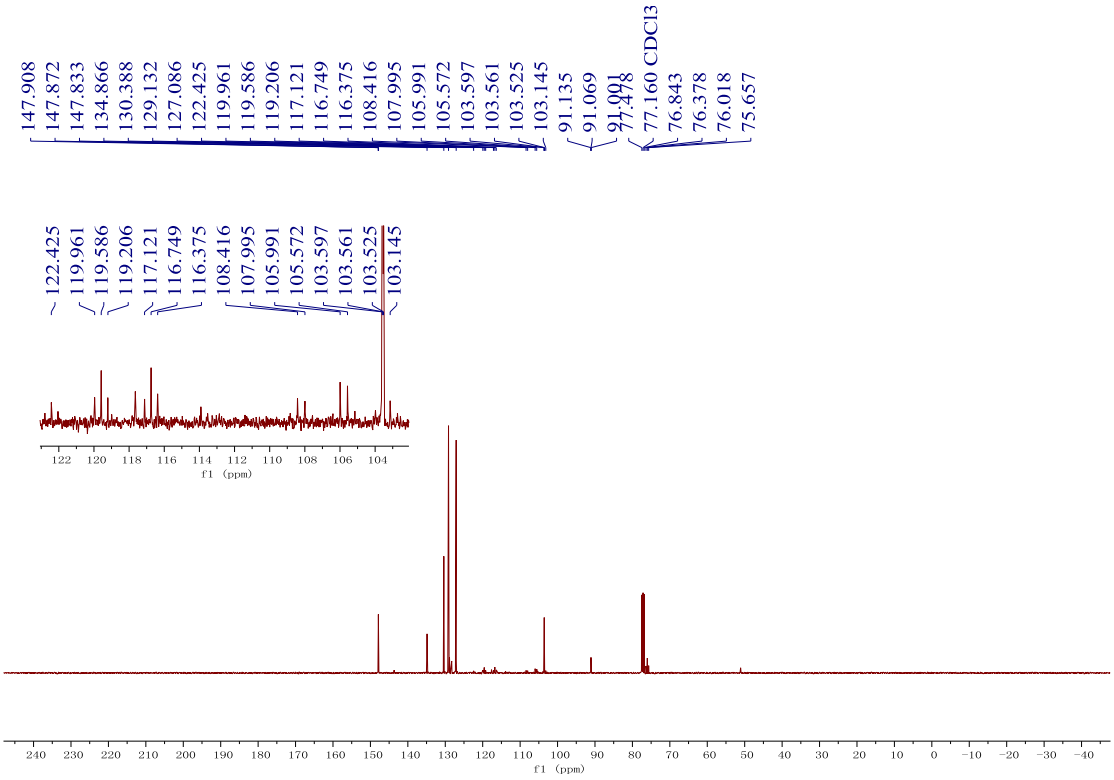

C_2F_5
4v ^{1}H NMR
(400 MHz, $CDCl_3$)

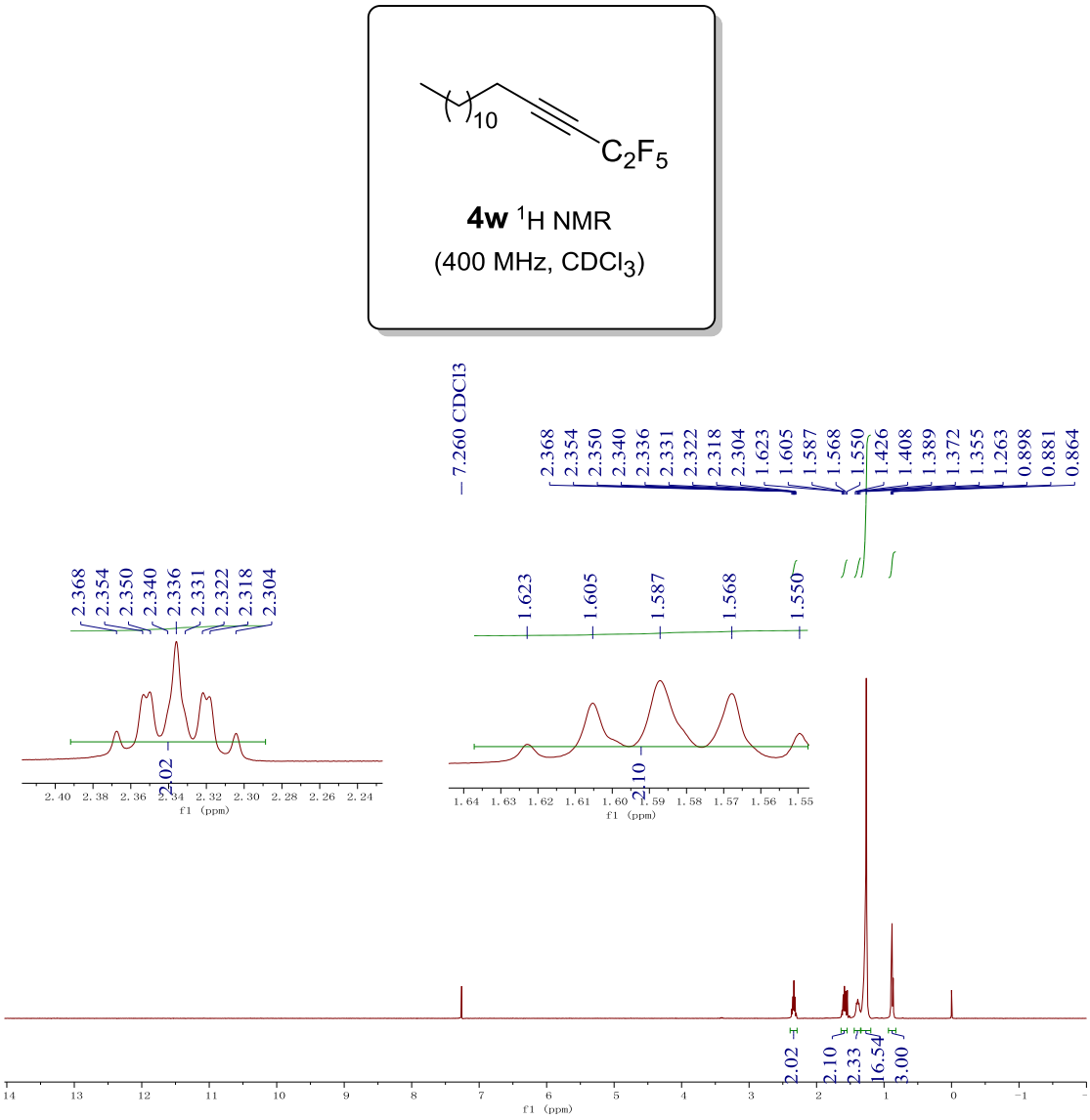

7.447
7.442
7.437
7.434
7.429
7.426
7.422
7.398
7.397
7.395
7.390
7.388
7.384
7.381
7.378
7.373
7.370
7.269
7.260 CDCl3
7.228
6.210
6.199
6.188
6.169
6.158
6.147
7.269
7.260 CDCl3
7.228
0.49
0.47
1.00
5.00
f1 (ppm)

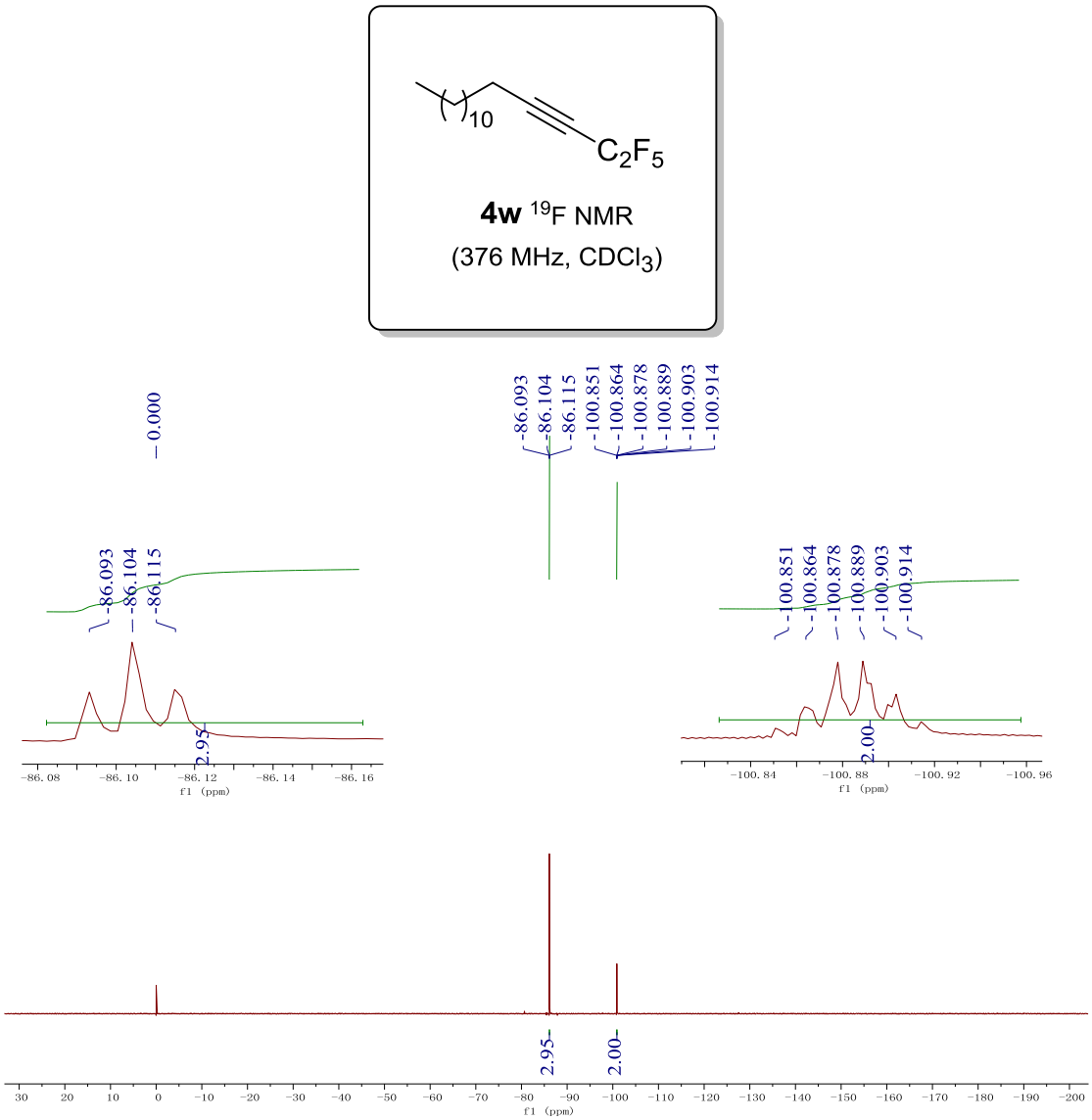

C_2F_5
4v ^{19}F NMR
(376 MHz, $CDCl_3$)

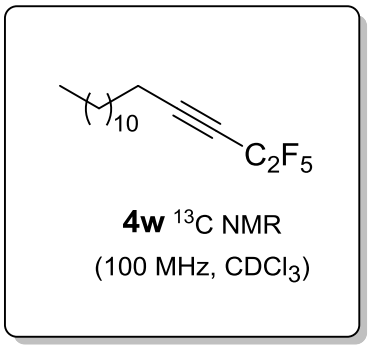

-0.000
-85.685
-85.697
-85.709
-101.135
-101.146
-101.159
-101.169
-101.181
-101.183
2.98
2.00
f1 (ppm)


C_2F_5
4v ^{13}C NMR
(100 MHz, $CDCl_3$)


147.908
147.872
147.833
134.866
130.388
129.132
127.086
122.425
119.961
119.586
119.206
117.121
116.749
116.375
108.416
107.995
105.991
105.572
103.597
103.561
103.525
103.145
91.135
91.069
77.160 CDCl3
76.843
76.378
76.018
75.657
f1 (ppm)

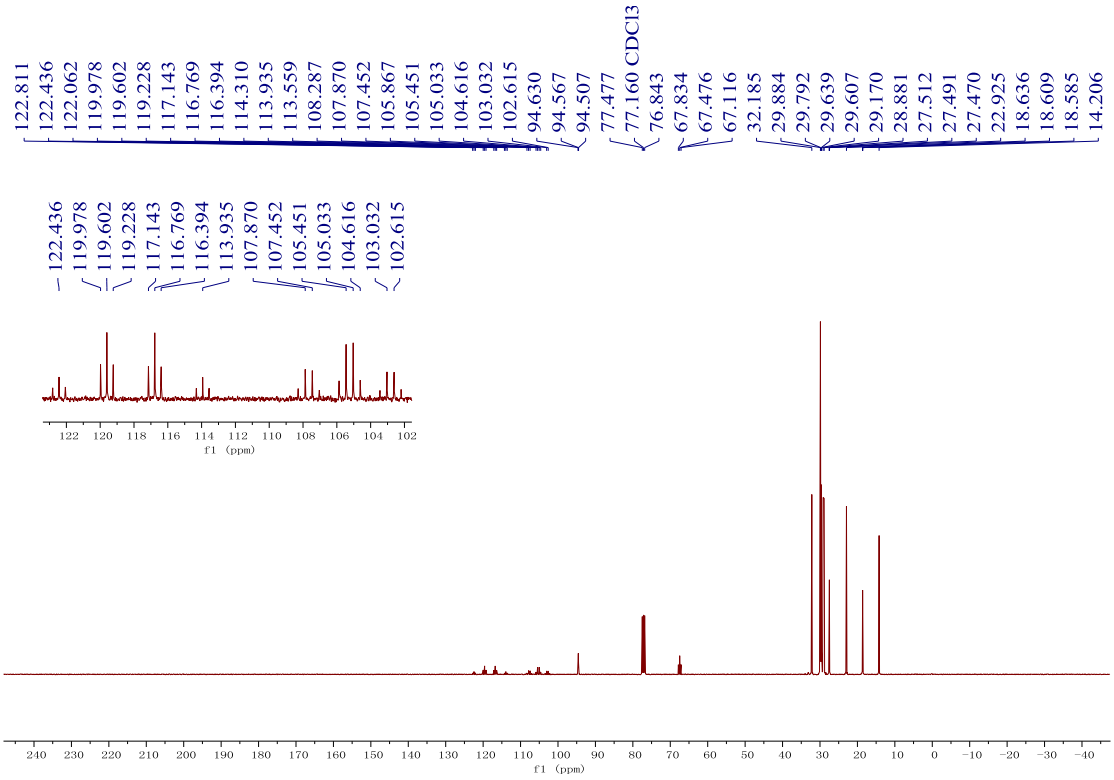

10
C2F5
4w 1H NMR
(400 MHz, CDCl3)
7.260 CDCl3
2.368
2.354
2.350
2.340
2.336
2.331
2.322
2.318
2.304
1.623
1.605
1.587
1.568
1.550
1.426
1.408
1.389
1.372
1.355
1.263
0.898
0.881
0.864
2.02
2.10
2.33
16.54
3.00
f1 (ppm)

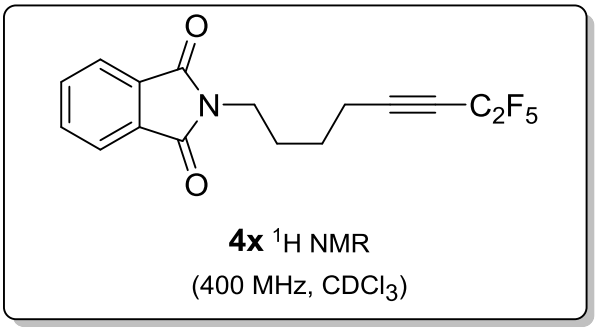

10
C2F5
4w 19F NMR
(376 MHz, CDCl3)
-0.000
-86.093
-86.104
-86.115
-100.851
-100.864
-100.878
-100.889
-100.903
-100.914
2.95
2.00
f1 (ppm)

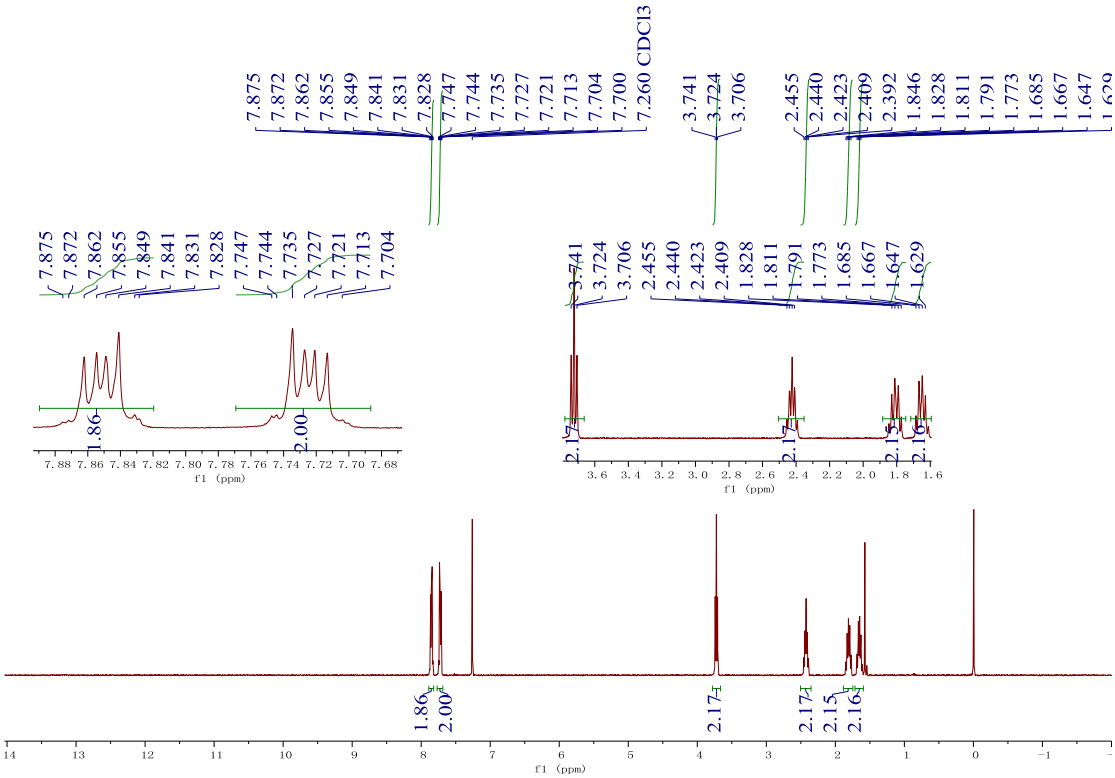


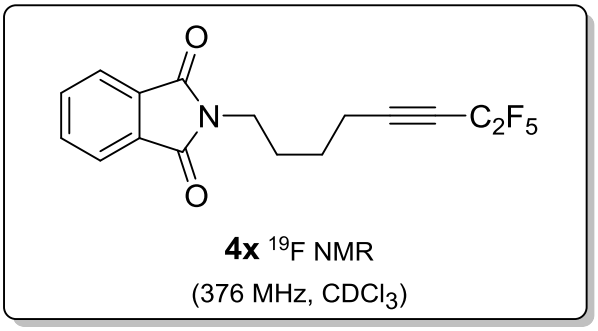
4w ^{13}C NMR

(100 MHz, $CDCl_3$)



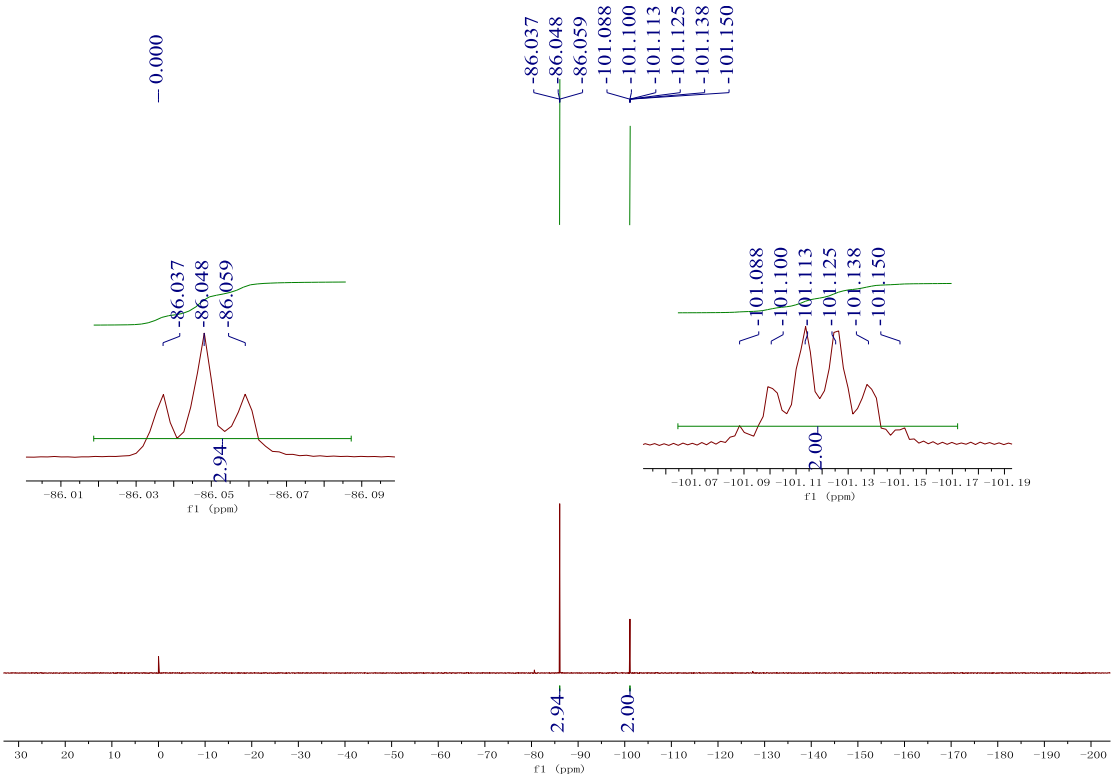

O
N
O
C_2F_5
4x ^{1}H NMR
(400 MHz, $CDCl_3$)

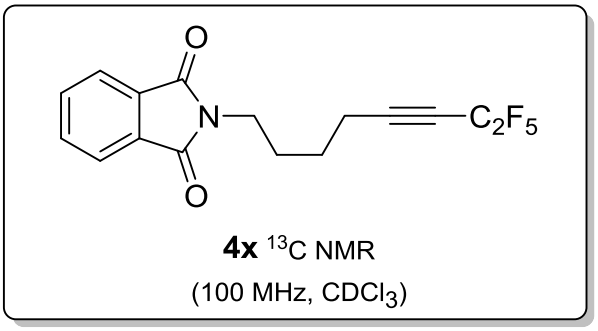

7.875
7.872
7.862
7.855
7.849
7.841
7.831
7.828
7.747
7.744
7.735
7.727
7.721
7.713
7.704
7.700
7.260 CDCl3
3.741
3.724
3.706
2.455
2.440
2.423
2.409
2.392
1.846
1.828
1.811
1.791
1.773
1.685
1.667
1.647
1.629
1.86
2.00
2.17
2.17
2.15
2.16
f1 (ppm)



4x ^{19}F NMR

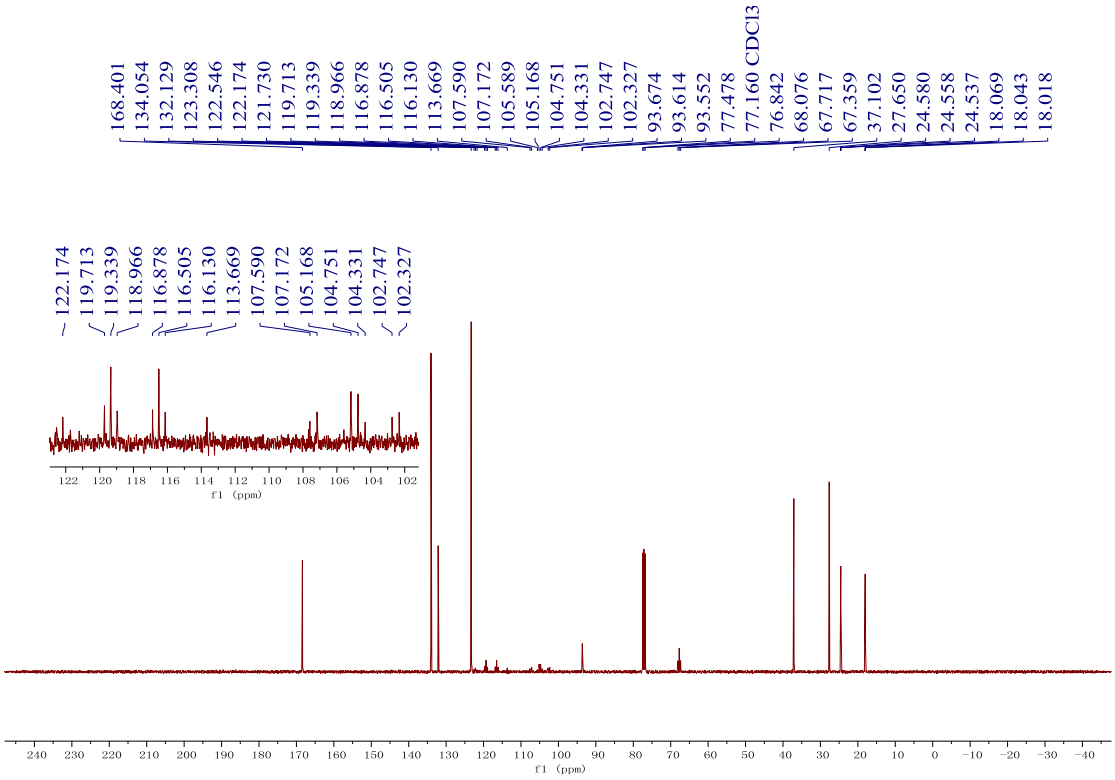
(376 MHz, $CDCl_3$)

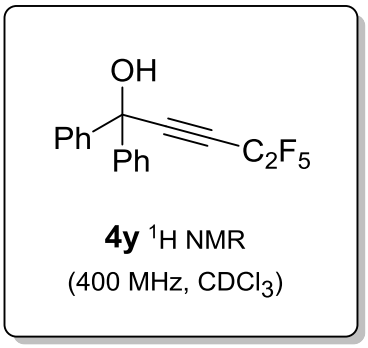


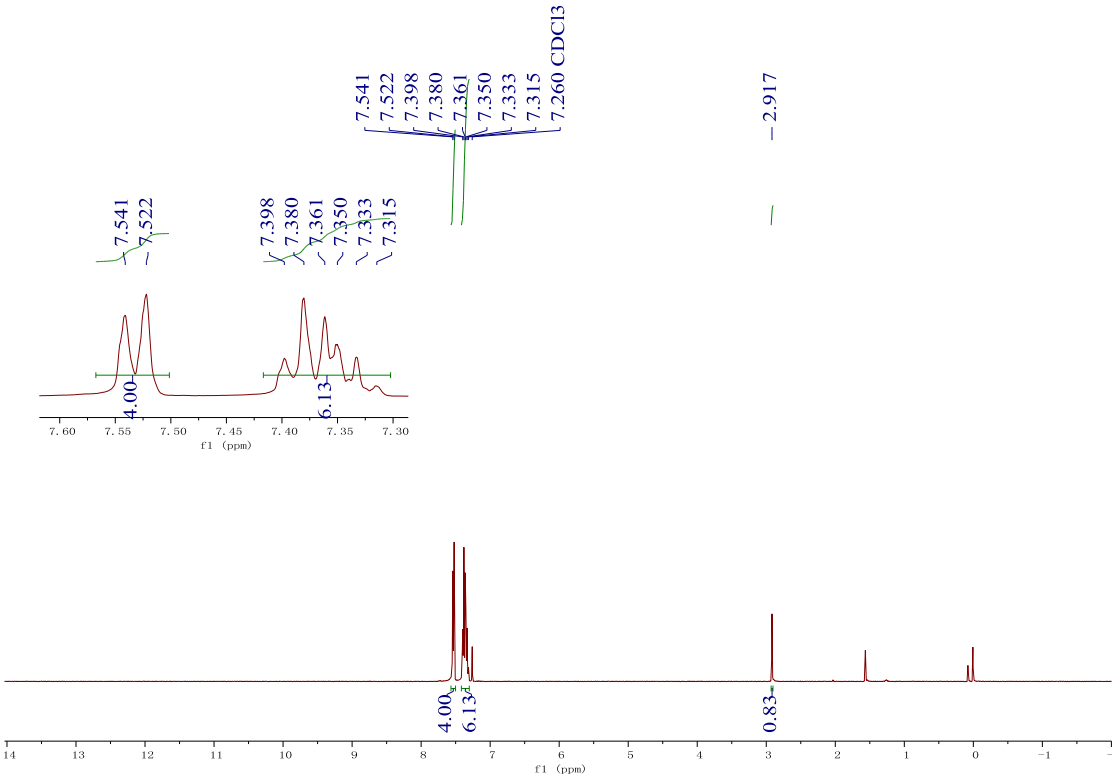


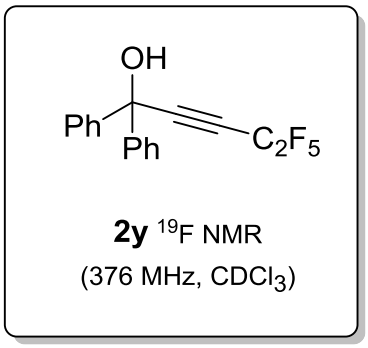
4x ^{13}C NMR

(100 MHz, $CDCl_3$)

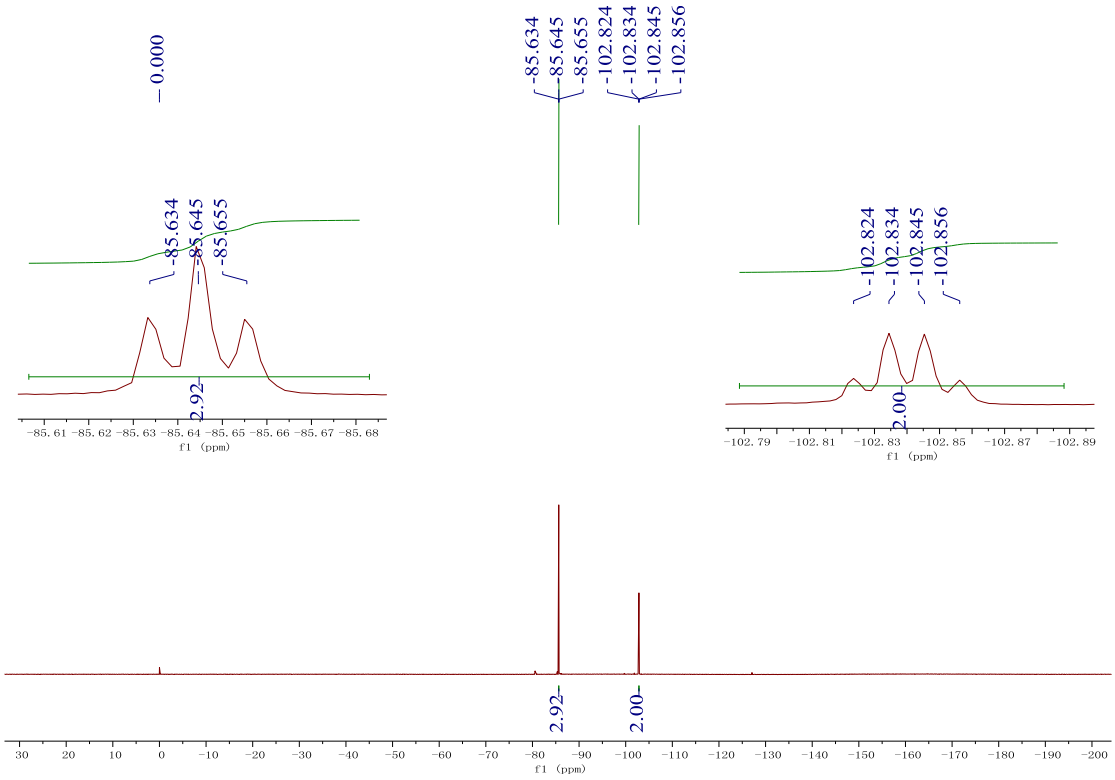


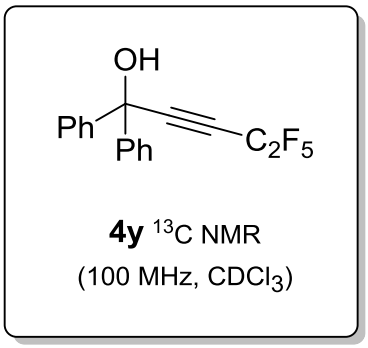

OH
Ph
Ph
C_2F_5
4y ^{1}H NMR
(400 MHz, $CDCl_3$)


7.541
7.522
7.398
7.380
7.361
7.350
7.333
7.315
7.260 CDCl3
2.917
4.00
6.13
0.83
f1 (ppm)

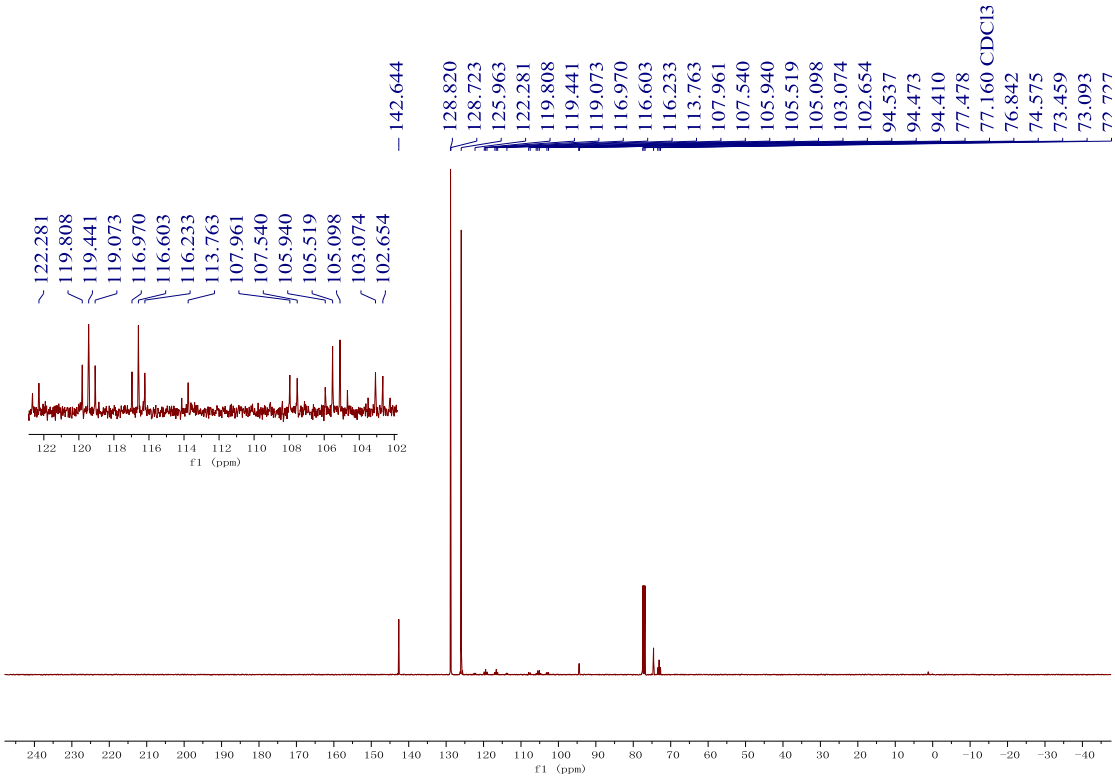


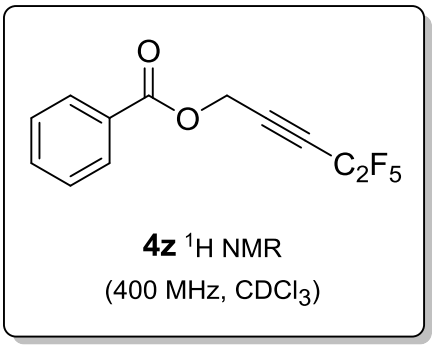
2y ^{19}F NMR
(376 MHz, $CDCl_3$)

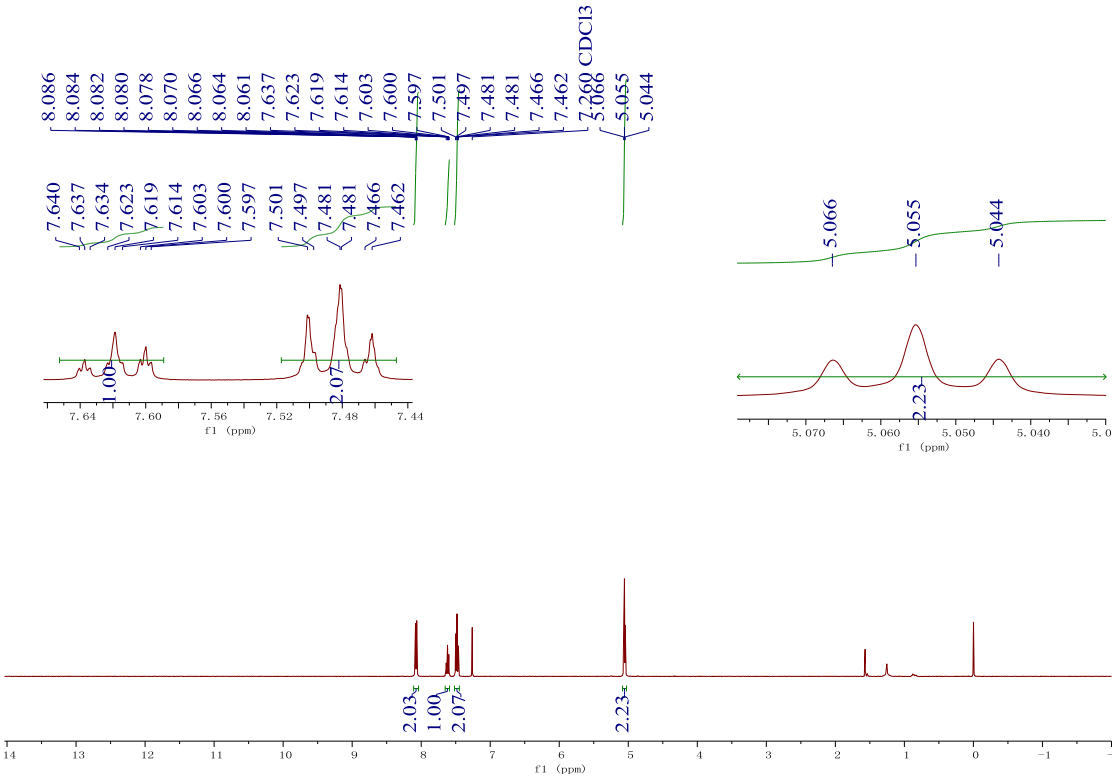


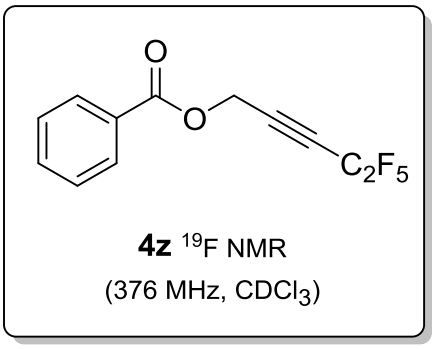


4y ^{13}C NMR
(100 MHz, $CDCl_3$)



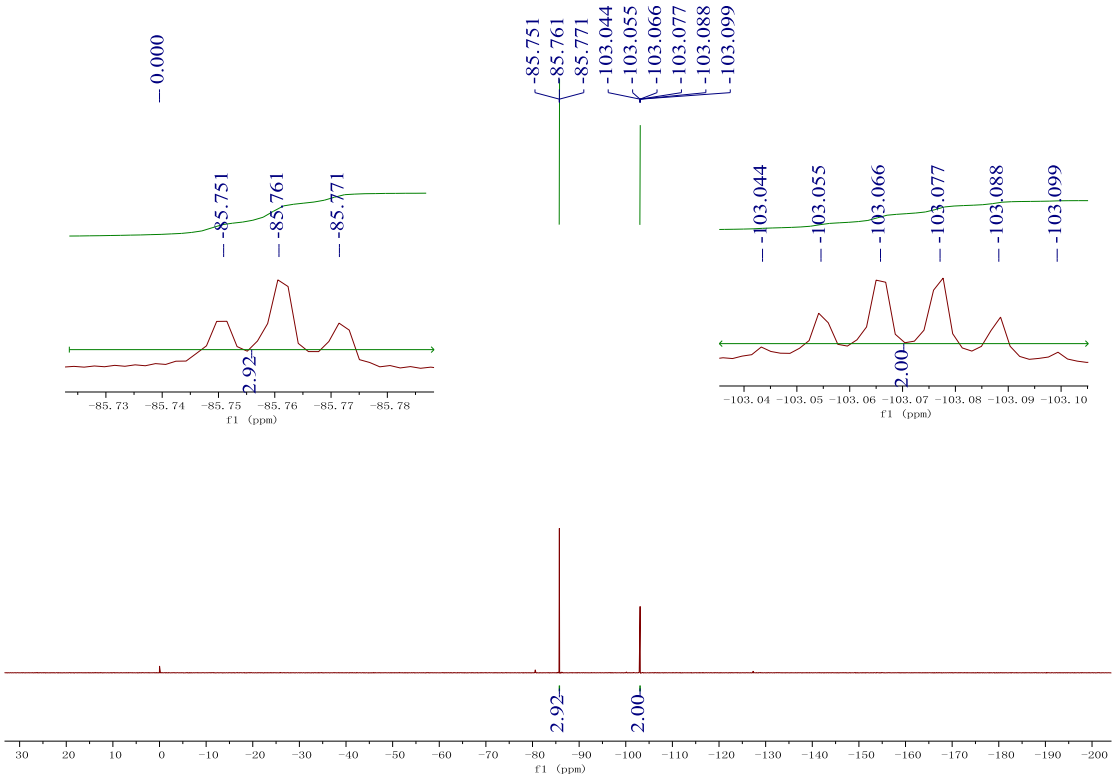

O
O
C_2F_5
4z ^{1}H NMR
(400 MHz, $CDCl_3$)

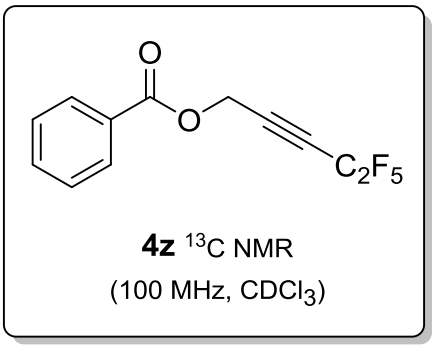

8.086
8.084
8.082
8.080
8.078
8.070
8.066
8.064
8.061
7.637
7.623
7.619
7.614
7.603
7.600
7.597
7.501
7.497
7.481
7.481
7.466
7.462
7.260 CDCl3
5.066
5.055
5.044
1.00
2.07
2.23
f1 (ppm)

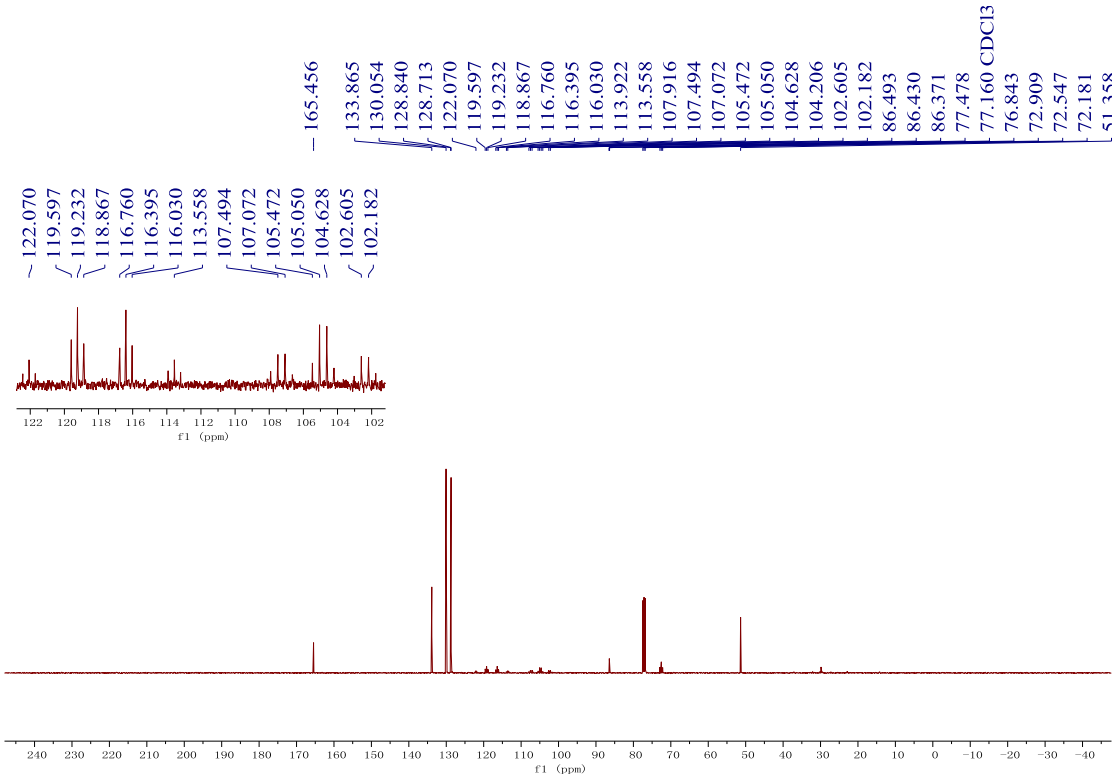


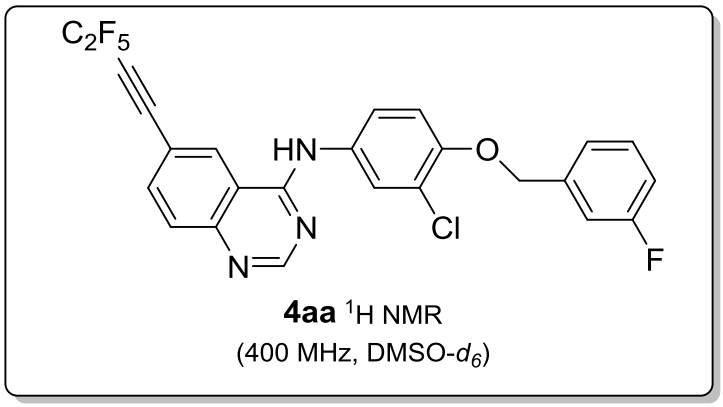
4z ^{19}F NMR

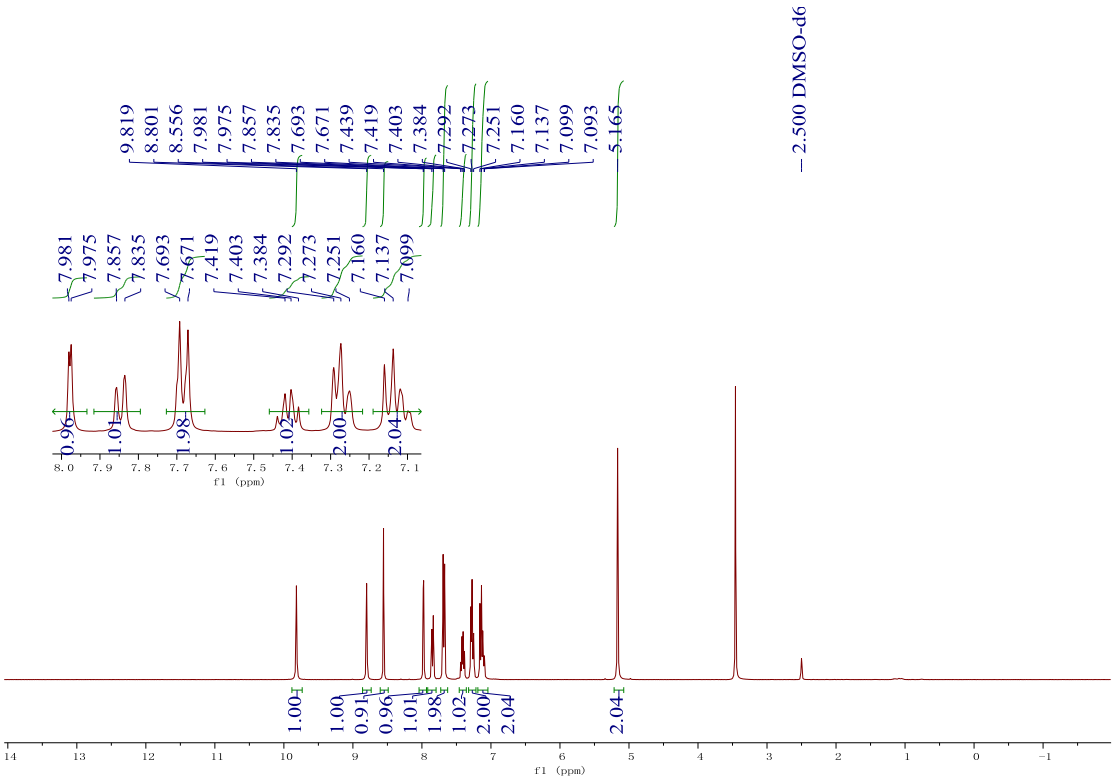
(376 MHz, $CDCl_3$)

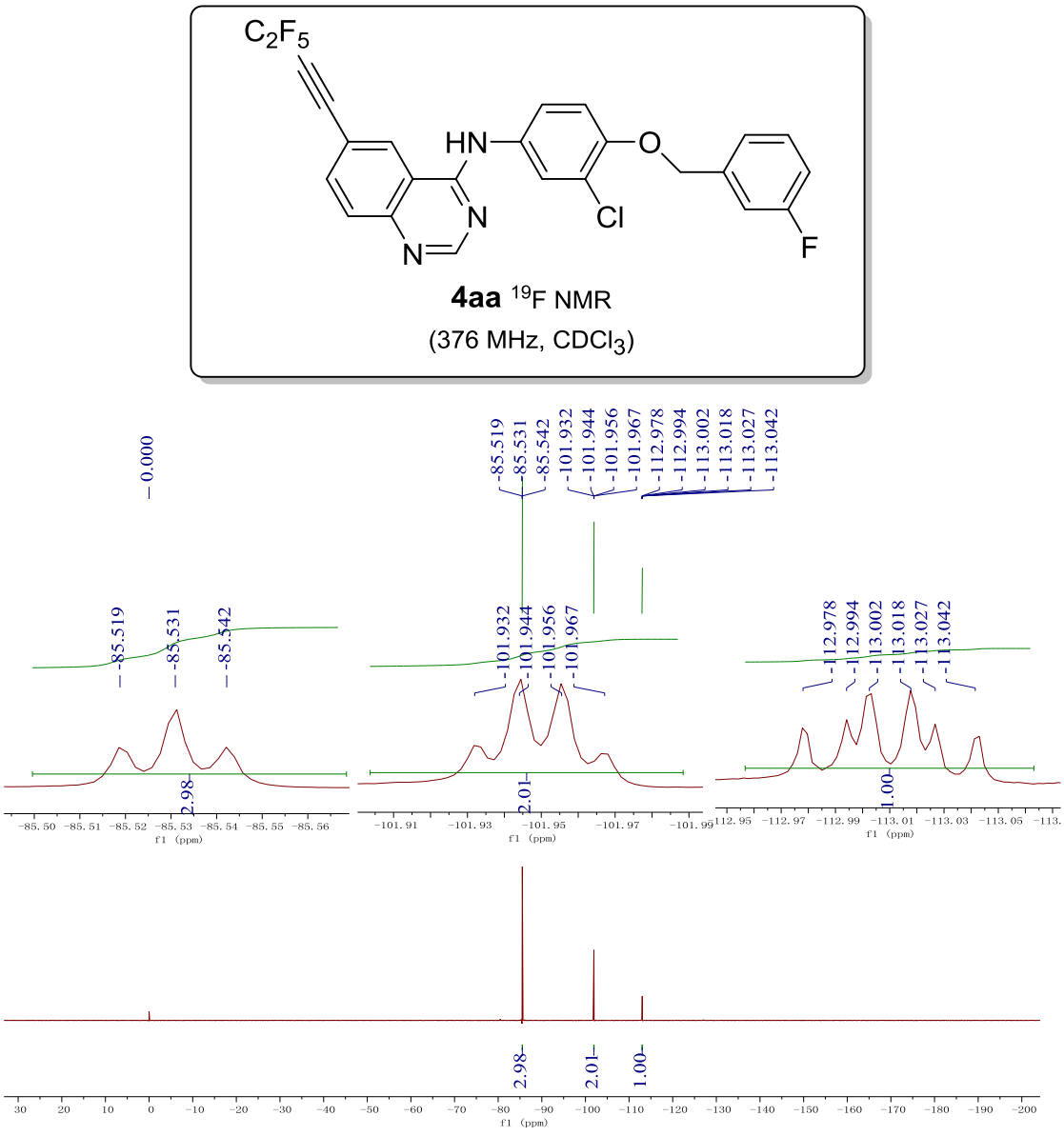


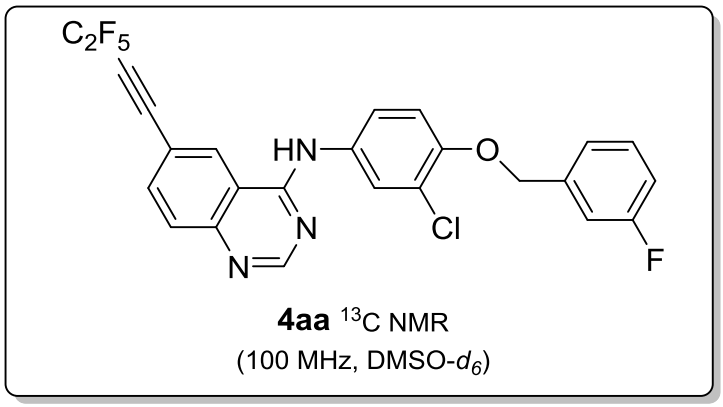

O
O
C_2F_5
4z ^{13}C NMR
(100 MHz, $CDCl_3$)


165.456
133.865
130.054
128.840
128.713
122.070
119.597
119.232
118.867
116.760
116.395
116.030
113.922
113.558
107.916
107.494
107.072
105.472
105.050
104.628
104.206
102.605
102.182
86.493
86.430
86.371
77.478
77.160 CDCl3
76.843
72.909
72.547
72.181
122.070
119.597
119.232
118.867
116.760
116.395
116.030
113.558
107.494
107.072
105.472
105.050
104.628
102.605
102.182
122 120 118 116 114 112 110 108 106 104 102
f1 (ppm)
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40
f1 (ppm)

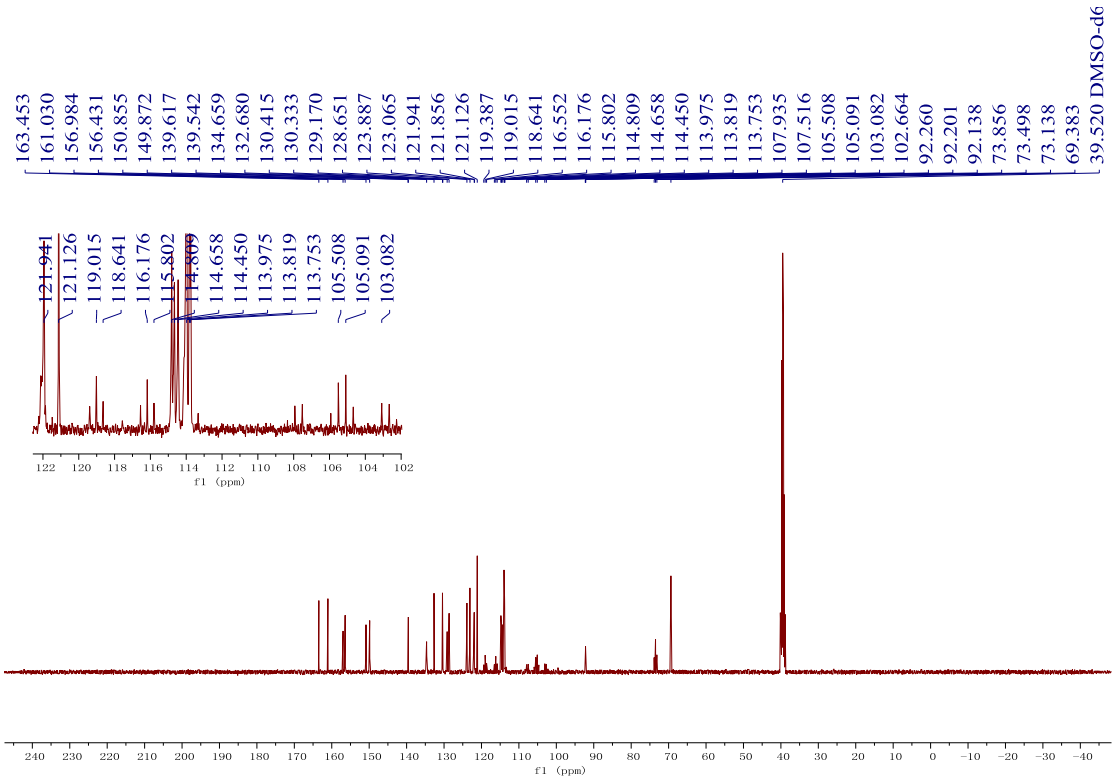

C2F5
HN
O
N
N
Cl
F
4aa 1H NMR
(400 MHz, DMSO-d6)

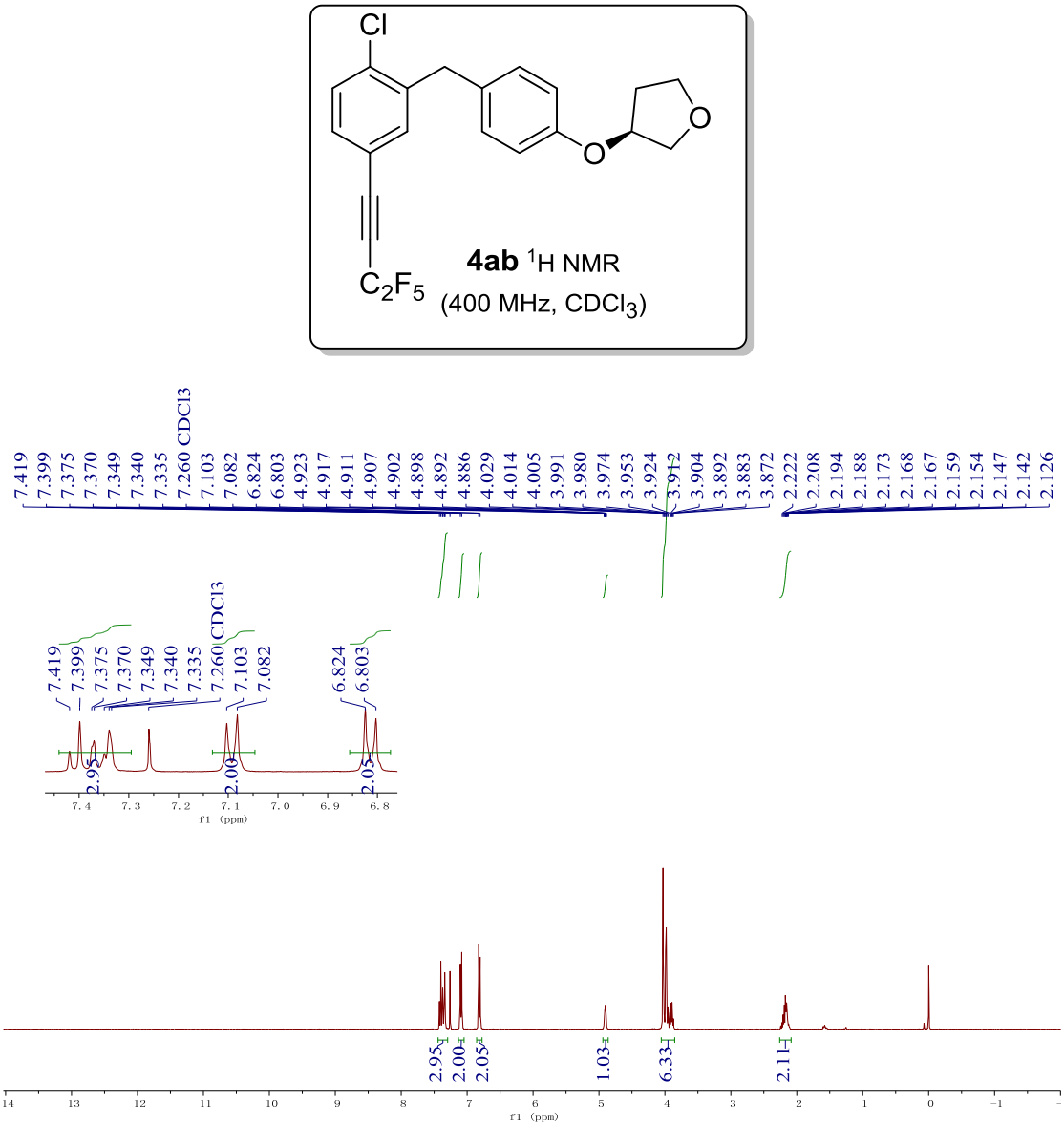

2.500 DMSO-d6
9.819
8.801
8.556
7.981
7.975
7.857
7.835
7.693
7.671
7.439
7.419
7.403
7.384
7.292
7.273
7.251
7.160
7.137
7.099
7.093
5.165
f1 (ppm)

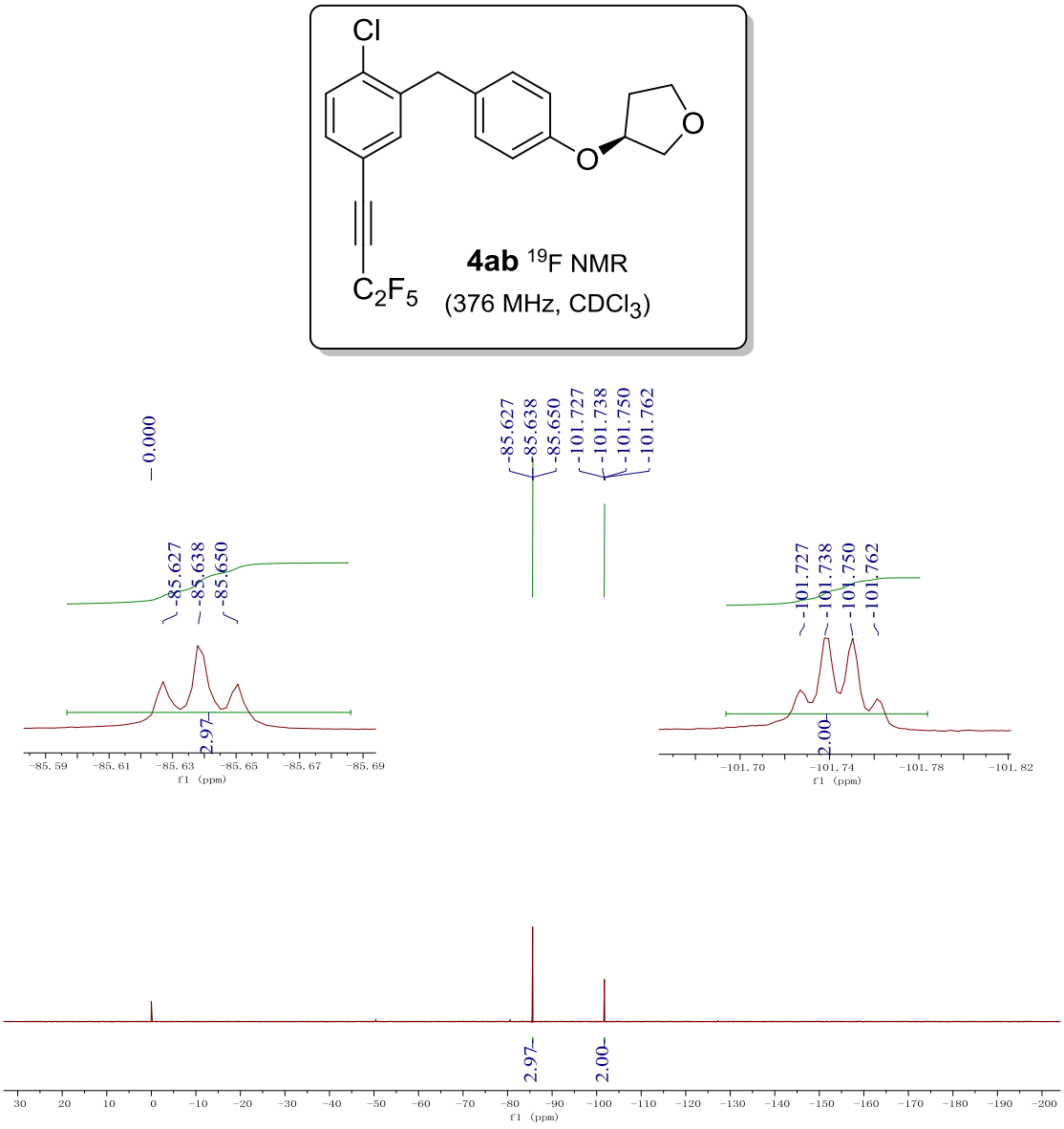

C2F5
HN
O
N
N
Cl
F
4aa 19F NMR
(376 MHz, CDCl3)
-0.000
-85.519
-85.531
-85.542
-101.932
-101.944
-101.956
-101.967
-112.978
-112.994
-113.002
-113.018
-113.027
-113.042
2.98
2.01
1.00
f1 (ppm)

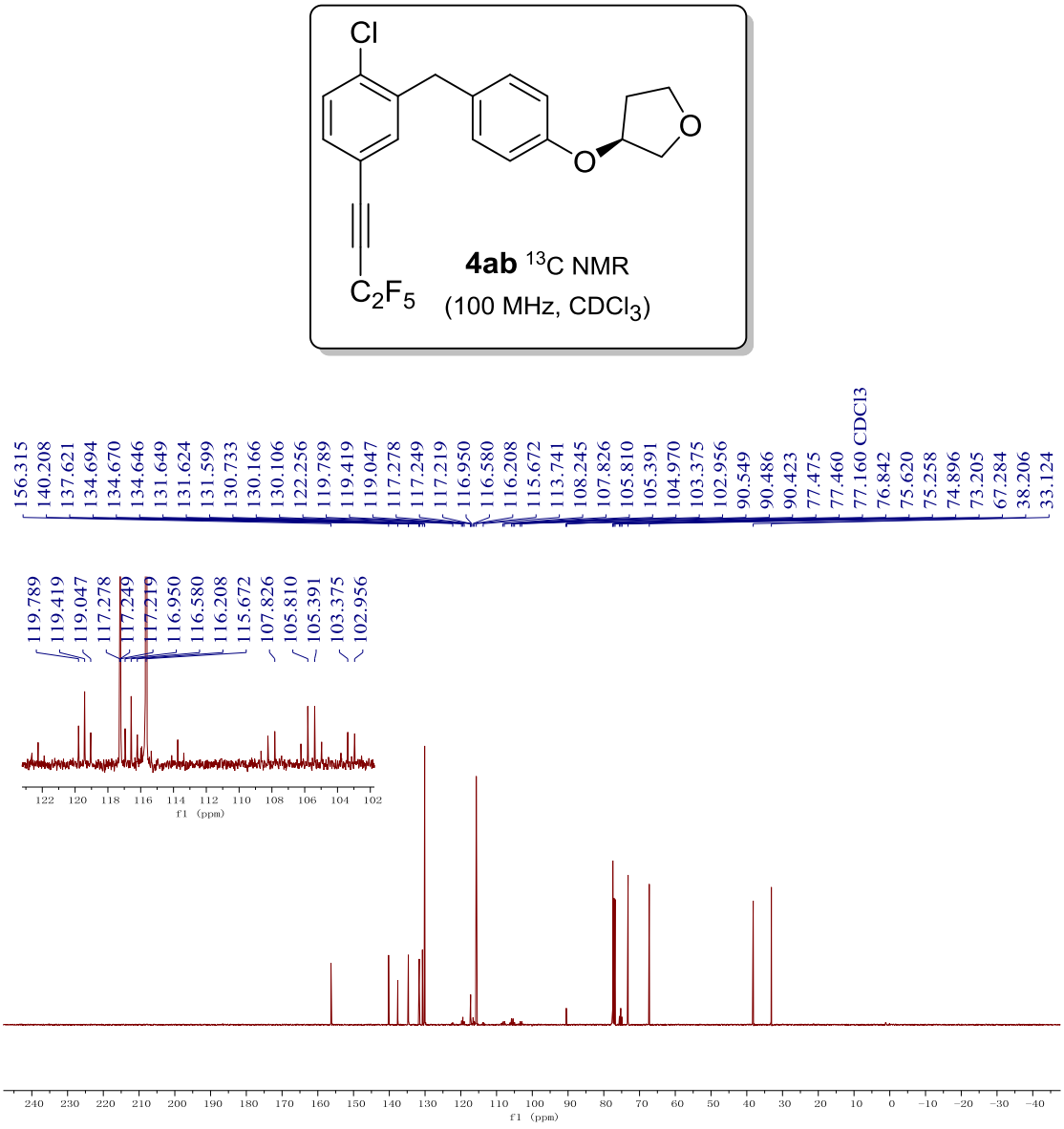


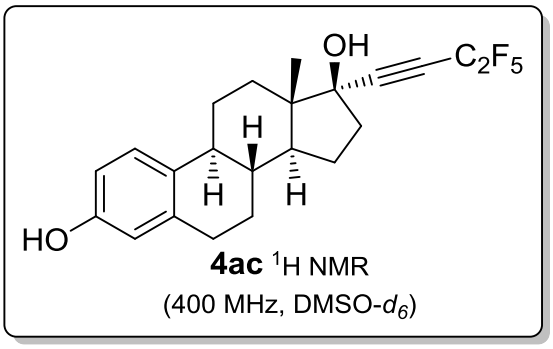
4aa ^{13}C NMR
(100 MHz, DMSO-d_6)



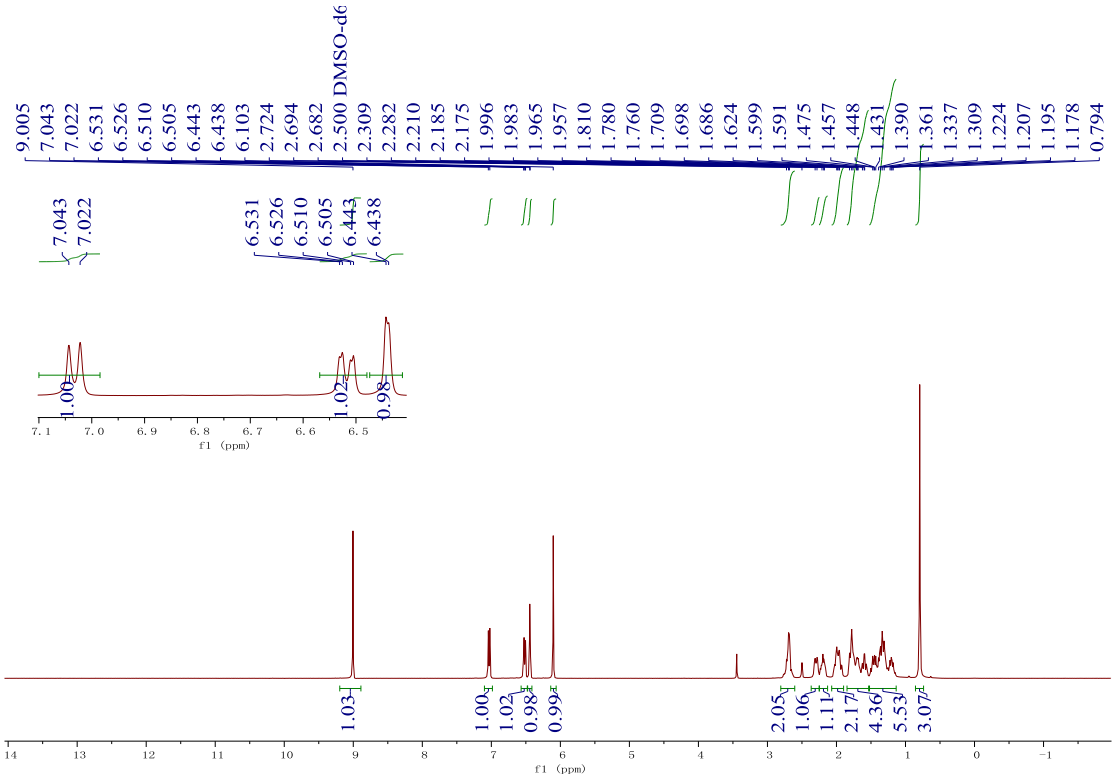

Cl
O
O
C_2F_5
4ab ^{1}H NMR
(400 MHz, $CDCl_3$)
7.419
7.399
7.375
7.370
7.349
7.340
7.335
7.260 CDCl3
7.103
7.082
6.824
6.803
4.923
4.917
4.911
4.907
4.902
4.898
4.892
4.886
4.029
4.014
4.005
3.991
3.980
3.974
3.953
3.924
3.912
3.904
3.892
3.883
3.872
2.222
2.208
2.194
2.188
2.173
2.168
2.167
2.159
2.154
2.147
2.142
2.126
2.95
2.00
2.05
1.03
6.33
2.11
f1 (ppm)

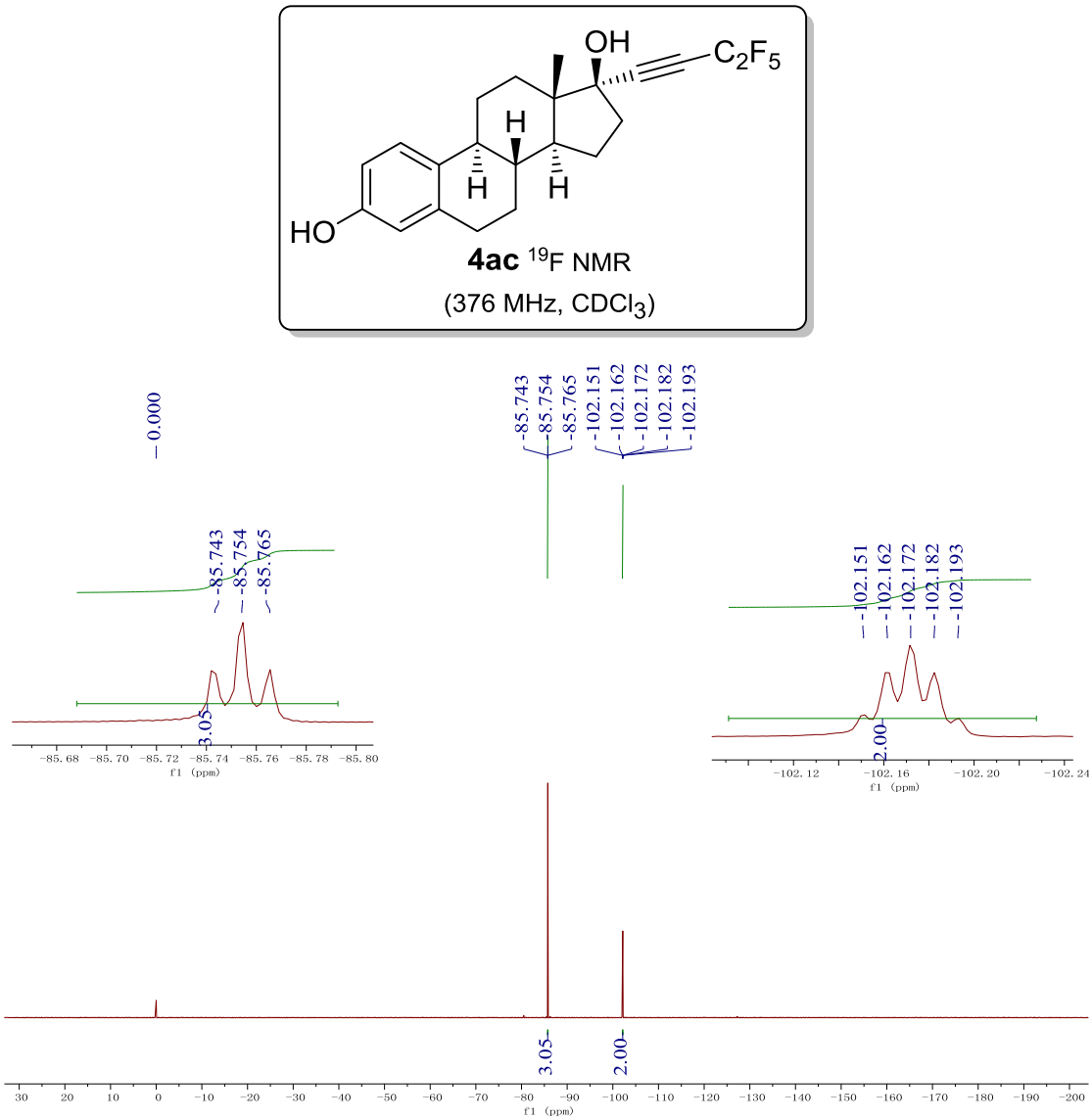

Cl
O
O
C_2F_5
4ab ^{19}F NMR
(376 MHz, $CDCl_3$)
-0.000
-85.627
-85.638
-85.650
-101.727
-101.738
-101.750
-101.762
2.97
2.00
f1 (ppm)

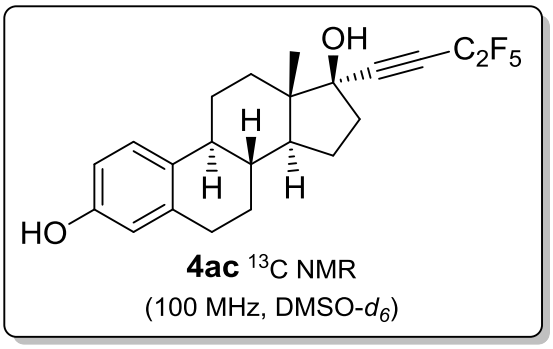

Cl
O
O
C_2F_5
4ab ^{13}C NMR
(100 MHz, $CDCl_3$)
156.315
140.208
137.621
134.694
134.670
134.646
131.649
131.624
131.599
130.733
130.166
130.106
122.256
119.789
119.419
119.047
117.278
117.249
117.219
116.950
116.580
116.208
115.672
113.741
108.245
107.826
105.810
105.391
104.970
103.375
102.956
90.549
90.486
90.423
77.475
77.460
77.160 CDCl3
76.842
75.620
75.258
74.896
73.205
67.284
38.206
33.124
f1 (ppm)



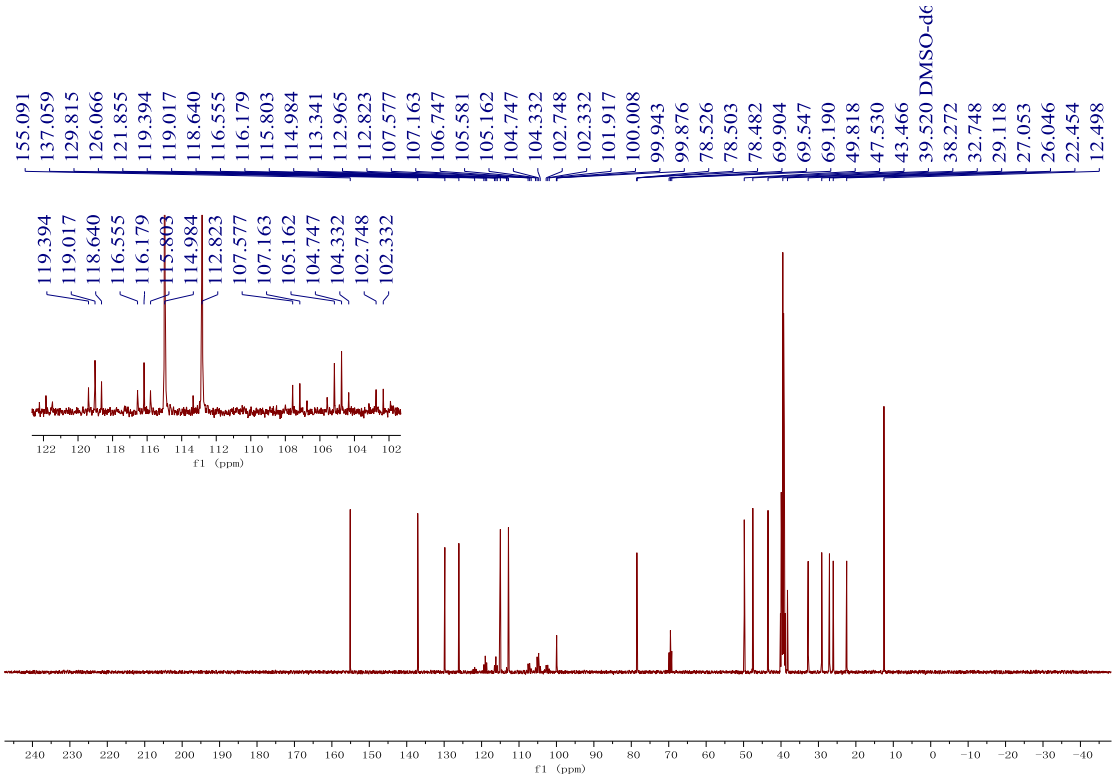
4ac ^{1}H NMR
(400 MHz, DMSO-d_6)

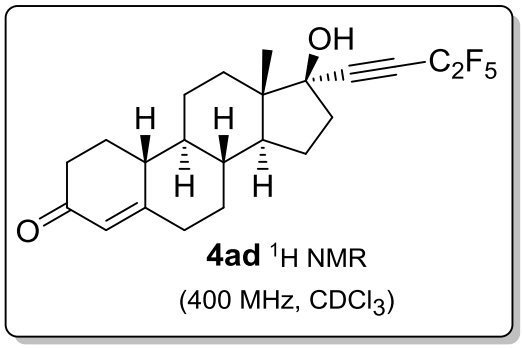



OH
C_2F_5
H
H
H
HO
4ac ^{19}F NMR
(376 MHz, $CDCl_3$)
-0.000
-85.743
-85.754
-85.765
-102.151
-102.162
-102.172
-102.182
-102.193
3.05
2.00
f1 (ppm)



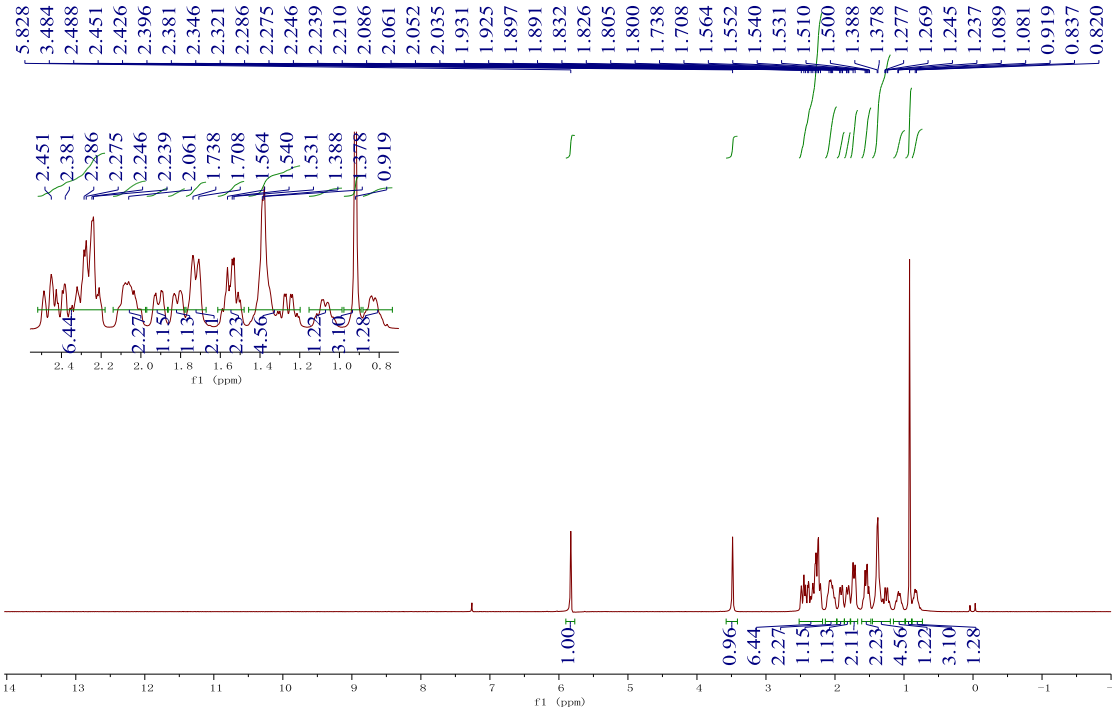
4ac ^{13}C NMR
(100 MHz, DMSO-d_6)

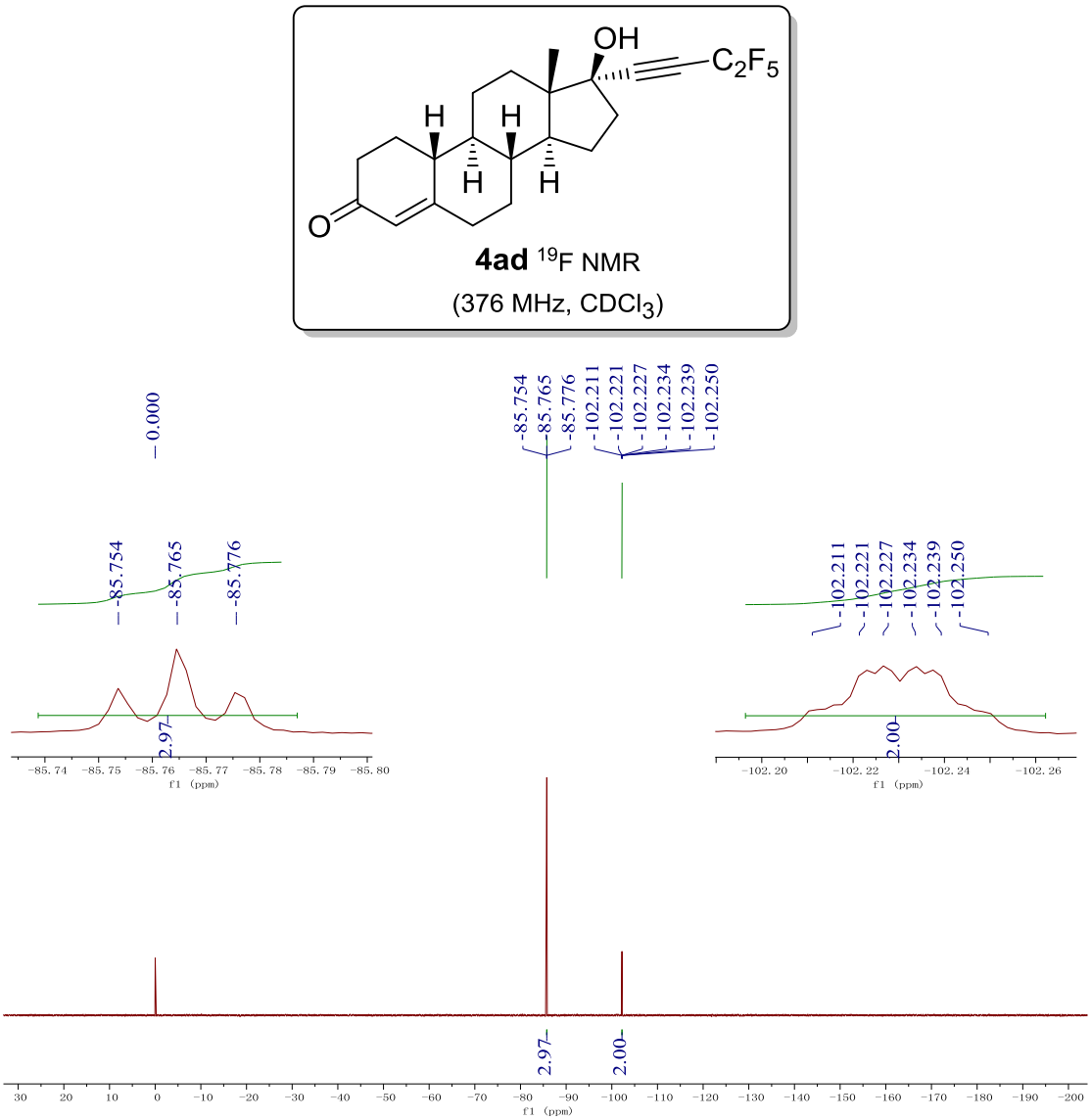


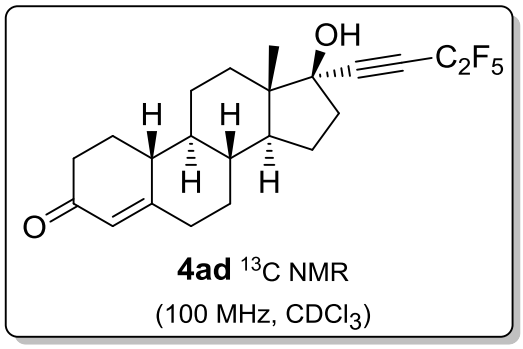


4ad ^{1}H NMR

(400 MHz, $CDCl_3$)




OH
C_2F_5
H
H
H
H
O
4ad ^{19}F NMR
(376 MHz, $CDCl_3$)
-0.000
-85.754
-85.765
-85.776
-102.211
-102.221
-102.227
-102.234
-102.239
-102.250
2.97
2.00
f1 (ppm)



4ad ^{13}C NMR

(100 MHz, $CDCl_3$)