

## Supplementary Information

### Visible light-induced gem-difluoroallylation of [1.1.1]propellane to access gem-difluoroallylic bicyclo[1.1.1]pentanes

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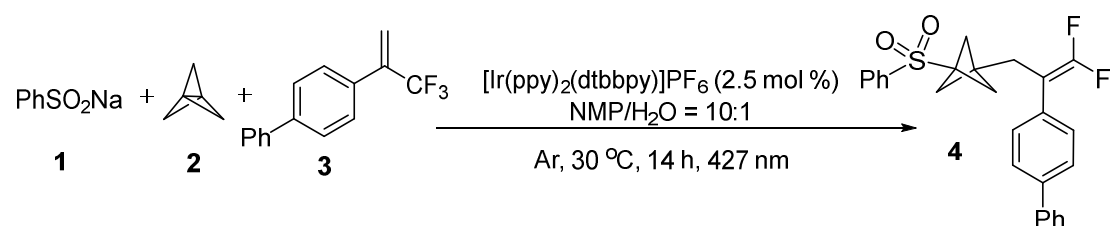
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## 1. General information and chemicals

**General:** The progress of the reactions was monitored by TLC with silica gel plates, and the visualization was carried out under UV light (254 nm). Melting points were determined using a Büchi B-540 capillary melting point apparatus. NMR spectra were recorded using Bruker Avance III 400 MHz or Bruker Avance III 600 MHz spectrometers. Chemical shifts of  $^1\text{H}$  NMR were reported relative to the solvent signal ( $\text{CDCl}_3$ :  $\delta = 7.26$  ppm). Chemical shifts of  $^{13}\text{C}$  NMR were reported relative to the solvent signal ( $\text{CDCl}_3$ :  $\delta = 77.00$  ppm). HRMS spectra were recorded on an electrospray ionization quadrupole time-of-flight (ESI-Q-TOF) mass spectrometer. Column chromatography was performed on silica gel (300 - 400 mesh). For blue light irradiation, a Kessil PR160L-blue LED lamp (30 W High Luminous DEX 2100 LED,  $\lambda_{\text{max}} = 427$  nm) was placed 9 cm away from the reaction vials.

**Chemicals:** Compounds (**1c-1p**)<sup>[1]</sup>, (**3q**, **3r**, **3s**, **3u**, **3w**, **3x**, **3z**, **3aa**, **3ab**, and **3ad**)<sup>[2]</sup>, (**3t**, **3v**, and **3y**)<sup>[3]</sup>, **3ac**<sup>[4]</sup>, and **3af**<sup>[5]</sup> were prepared using reported procedures. [1.1.1]propellane was prepared according to a literature procedure<sup>[6]</sup> (typically concentrations are 0.45 – 0.80 M with this protocol). Other compounds were purchased from Energy Chemical or Bidepharm and used without further purification.

## 2. General method

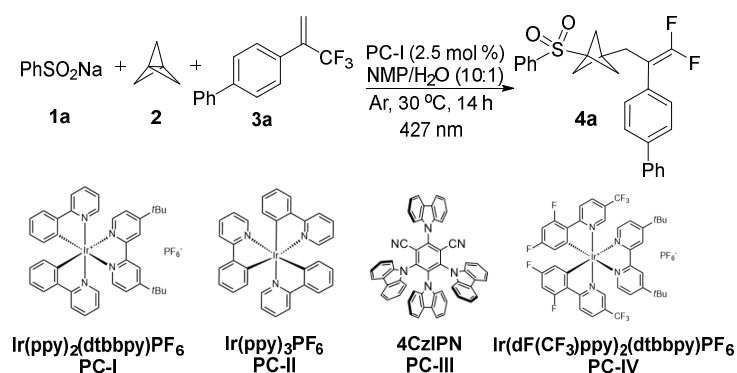


An oven-dried 10 mL Schlenk tube equipped with a magnetic stir bar was charged with sodium sulfonates **1** (32.8 mg, 0.2 mmol, 1.0 equiv.),  $[\text{Ir}(\text{ppy})_2(\text{dtbbpy})]\text{PF}_6$  (4.6 mg, 0.005 mmol, 0.025 equiv.), **3** (99.3 mg, 0.4 mmol, 2.0 equiv.), NMP:  $\text{H}_2\text{O} = 10:1$  (1.1 mL), and [1.1.1]propellane **2** (0.24 mmol, 1.2 equiv.) sequentially under argon atmosphere. The resulting solution was irradiated by a 40 W Kessil lamp (427 nm, blue light, fourth gear) with stirring at a distance of 9 cm (with cooling by a fan) at 30 °C for about 14 h. The reaction was diluted with  $\text{H}_2\text{O}$  (10 mL) and then

extracted with EtOAc (15 mL × 4). The combined organic layers were washed with brine (15 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated. The residue was purified by flash chromatography on silica gel with petroleum ether/ethyl acetate to afford the desired product **4**.

### 3. Optimization studies

**Table S1.** Optimization of reaction conditions<sup>a</sup>

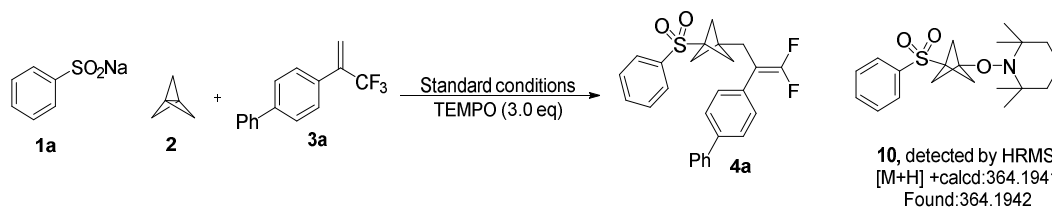


Entry	Change from standard conditions	Yield (%) <sup>b</sup>
1	None	<b>90 (85)<sup>c</sup></b>
2	DCM instead of NMP	49
3	DMF instead of NMP	74
4	EtOH instead of NMP	31
5	PC-II instead of PC-I	trace
6	PC-III instead of PC-I	64
7	PC-IV instead of PC-I	53
8	395 nm instead of 427 nm	13
9	w/o light	trace
10	w/o photocatalyst	trace

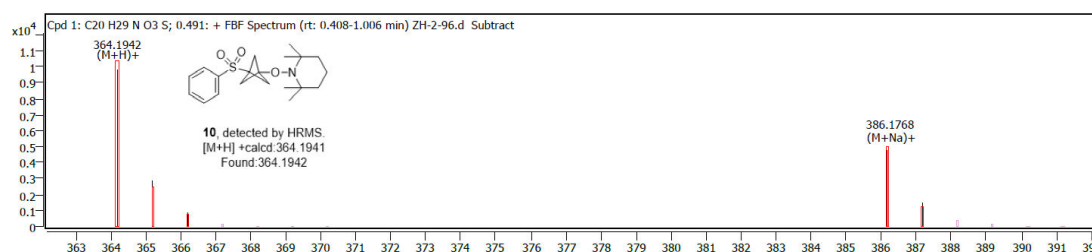
<sup>a</sup> Reaction conditions: **1a** (0.1 mmol), **2** (0.12 mmol), **3a** (0.2 mmol), and photocatalyst (2.5 mol %) in solvent = H<sub>2</sub>O = 10:1 (0.55 mL) under blue LEDs irradiation (427 nm, 30 W) at 30 °C for 14 h under argon atmosphere. <sup>b</sup> AY = assay yields, determined by <sup>1</sup>H NMR analysis of the crude reaction mixture using 1,3,5-trimethoxybenzene as an internal standard. <sup>c</sup> Isolated yields in parentheses. DCM = dichloromethane, NMP = *N*-methylpyrrolidone, DMF = *N,N*-dimethylformamide, EtOH = ethanol.

## 4. Control experiments

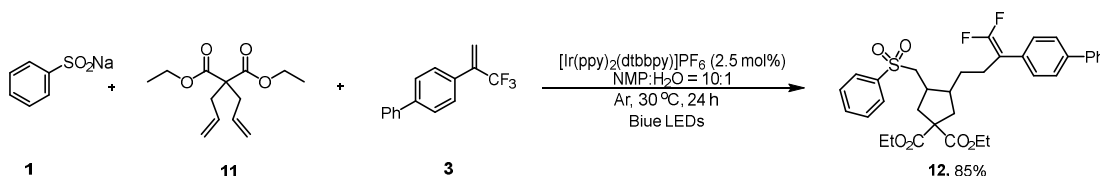
### a) Radical inhibiting experiment



An oven-dried 10 mL Schlenk tube equipped with a magnetic stir bar was charged with sodium sulfonates **1** (32.8 mg, 0.2 mmol, 1.0 equiv.), [Ir(ppy)<sub>2</sub>(dtbbpy)]PF<sub>6</sub> (4.6 mg, 0.005 mmol, 0.025 equiv.), TEMPO (93.7 mg, 0.6 mmol, 3.0 equiv.), 4-(3,3,3-trifluoroprop-1-en-2-yl)-1,1'-biphenyl **3** (99.3 mg, 0.4 mmol, 2.0 equiv.), NMP: H<sub>2</sub>O = 10:1 (1.1 mL), and [1.1.1]propellane **2** (0.24 mmol, 1.2 equiv.) sequentially under argon atmosphere. The resulting solution was irradiated by a 40 W Kessil lamp (427 nm, blue light, fourth gear) with stirring at a distance of 9 cm (with cooling by a fan) at 30 °C for about 14 h. Only trace amount of product was detected and the corresponding adducts **10** was detected by the HRMS. HRMS (ESI) m/z: calcd for C<sub>20</sub>H<sub>30</sub>NO<sub>3</sub>S [M+H]<sup>+</sup> 364.1941, found: 364.1942.



### b) Radical clock cyclization experiment



An oven-dried 10 mL Schlenk tube equipped with a magnetic stir bar was charged with sodium sulfonates **1** (32.8 mg, 0.2 mmol, 1.0 equiv.), [Ir(ppy)<sub>2</sub>(dtbbpy)]PF<sub>6</sub> (4.6 mg, 0.005 mmol, 0.025 equiv.), 4-(3,3,3-trifluoroprop-1-en-2-yl)-1,1'-biphenyl **3** (99.3 mg, 0.4 mmol, 2.0 equiv.) and diethyl 2,2-diallylmalonate **11** (57.6 mg, 0.24 mmol, 1.2 equiv.), NMP: H<sub>2</sub>O = 10:1 (1.1 mL) under argon atmosphere. The resulting solution was irradiated by a 40 W Kessil lamp (427 nm, blue light,

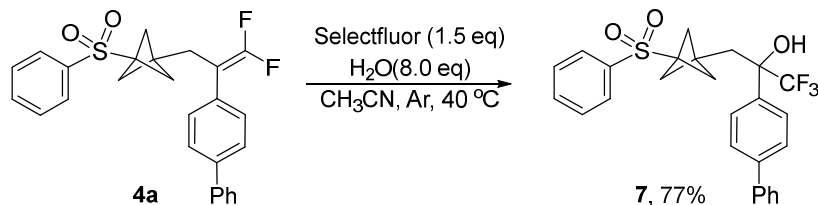
fourth gear) with stirring at a distance of 9 cm (with cooling by a fan) at 30 °C for about 14 h. The reaction was diluted with H<sub>2</sub>O (10 mL) and then extracted with EtOAc (15 mL × 4). The combined organic layers were washed with brine (15 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated. The residue was purified by flash chromatography on silica gel with petroleum ether/ethyl acetate to afford the desired product **12** as a colorless oil (108.0 mg, 85%); <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.90 – 7.83 (m, 2H), 7.65 – 7.56 (m, 5H), 7.54 – 7.40 (m, 4H), 7.40 – 7.30 (m, 3H), 4.24 – 4.11 (m, 4H), 3.15 – 2.90 (m, 2H), 2.71 – 2.20 (m, 6H), 2.19 – 2.06 (m, 1H), 2.04 – 1.95 (m, 1H), 1.34 – 1.13 (m, 8H). <sup>13</sup>C NMR (100 MHz, Chloroform-*d*) δ 172.41, 172.04, 153.48 (dd, *J* = 291.4, 287.2 Hz), 140.27, 140.02, 139.54, 133.63, 131.98 (t, *J* = 3.6 Hz), 129.21, 128.72, 128.38 (t, *J* = 3.2 Hz), 127.80, 127.36, 127.08, 126.86, 91.56 (dd, *J* = 21.4, 13.0 Hz), 61.62, 61.54, 58.28, 55.51, 42.01, 38.13, 37.96, 36.20, 27.22, 25.74, 13.89, 13.87. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -90.41 (d, *J* = 42.2 Hz, 1F), -90.69 (d, *J* = 42.2 Hz, 1F). HRMS (ESI) *m/z*: calcd for C<sub>34</sub>H<sub>37</sub>F<sub>2</sub>O<sub>6</sub>S [M+H]<sup>+</sup> 611.2273, found: 611.2277.

## 5. Gram-scale synthesis and derivatization of **4a**

### a) Gram-scale synthesis of **4a**

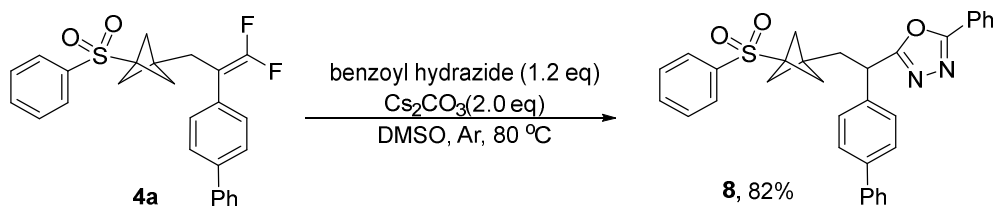
An oven-dried 250 mL two-necked flask equipped with a magnetic stir bar was charged with sodium sulfonates **1** (820.8 mg, 5.0 mmol, 1.0 equiv.), [Ir(ppy)<sub>2</sub>(dtbbpy)]PF<sub>6</sub> (100 mg, 0.125 mmol, 0.025 equiv.), 4-(3,3,3-trifluoroprop-1-en-2-yl)-1,1'-biphenyl **3** (2482.5 mg, 10.0 mmol, 2.0 equiv.), NMP: H<sub>2</sub>O = 10:1 (27.5 mL), and [1.1.1]propellane **2** (6.0 mmol, 1.2 equiv.) under argon atmosphere. The resulting solution was irradiated by a 40 W Kessil lamp (427 nm, blue light, fourth gear) with stirring at a distance of 9 cm (with cooling by a fan) at 30 °C for about 14 h. The reaction was diluted with H<sub>2</sub>O (100 mL) and then extracted with EtOAc (50 mL × 4). The combined organic layers were washed with brine (100 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated. The residue was purified by flash chromatography on silica gel with petroleum ether/ethyl acetate to afford the desired product **4a** as a white solid (1.7899, 82%).

**b) Synthesis of 2-([1,1'-biphenyl]-4-yl)-1,1,1-trifluoro-3-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)propan-2-ol 7<sup>[7]</sup>**



In an oven-dried 10 mL Schlenk tube with a magnetic stir bar was charged with **4a** (87.2 mg, 0.2 mmol, 1.0 equiv.), Selectfluor (106.3 mg, 0.3 mmol, 1.5 equiv.), and H<sub>2</sub>O (28.8 mg, 1.6 mmol, 8.0 equiv.), and anhydrous CH<sub>3</sub>CN (0.8 mL). After being stirred at 40 °C for 4 h, the reaction was quenched with 10 mL of water at room temperature. The resulting mixture was extracted with ethyl acetate (10 mL × 3), and the combined organic layers were washed with brine (10 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated. The residue was purified by flash chromatography on silica gel with petroleum ether/ethyl acetate = 3:1 (v/v) to afford the desired product **5** as a white solid (73.6 mg, 77%); M.p.: 154 - 157 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.76 – 7.70 (m, 2H), 7.65 – 7.57 (m, 5H), 7.55 – 7.43 (m, 6H), 7.43 – 7.35 (m, 1H), 2.64 (s, 1H), 2.53 (d, *J* = 15.0 Hz, 1H), 2.24 (d, *J* = 15.0 Hz, 1H), 1.83 – 1.76 (m, 3H), 1.69 – 1.64 (m, 3H). <sup>13</sup>C NMR (100 MHz, Chloroform-*d*) δ 141.57, 139.99, 136.62, 133.98, 133.57, 129.02, 128.85, 128.45, 127.71, 127.10, 126.97, 126.68, 124.97 (q, *J* = 285.2 Hz), 76.43, 52.23, 52.17, 35.38, 35.27. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -81.24. HRMS (ESI) *m/z*: calcd for C<sub>26</sub>H<sub>24</sub>F<sub>3</sub>O<sub>3</sub>S [M+H]<sup>+</sup> 473.1393, found: 473.1405.

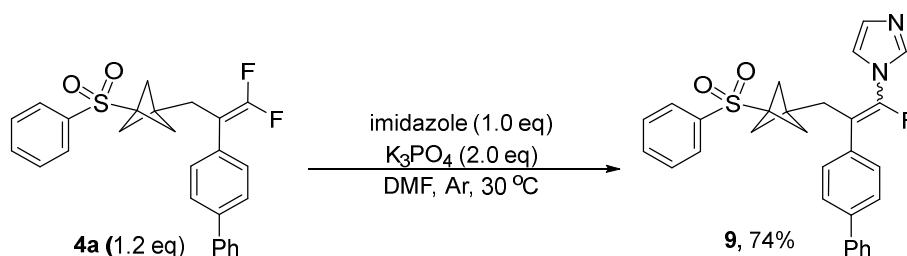
**c) Synthesis of 2-(1-([1,1'-biphenyl]-4-yl)-2-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)ethyl)-5-phenyl-1,3,4-oxadiazole 8<sup>[8]</sup>**



In an oven-dried 10 mL Schlenk tube with a magnetic stir bar was charged with **4a** (87.2 mg,

0.2 mmol, 1.0 equiv.), benzoyl hydrazide (32.7 mg, 0.24 mmol, 1.2 equiv.) and Cs<sub>2</sub>CO<sub>3</sub> (130.3 mg, 0.4 mmol, 2.0 equiv.), and anhydrous DMSO (1 mL) was stirred at 80 °C for 6 h. When the reaction finished, the resulting mixture was cooled to room temperature, 10 mL of water was added to quench the reaction, the resulting mixture was extracted with ethyl acetate (10 mL × 3). The combined organic layers were washed with brine (10 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated. The residue was purified by flash chromatography on silica gel with petroleum ether/ethyl acetate = 2:1 (v/v) to afford the desired product **6** as a colorless oil (87.1 mg, 82%); <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 8.07 – 7.94 (m, 2H), 7.83 – 7.75 (m, 2H), 7.65 – 7.58 (m, 1H), 7.59 – 7.30 (m, 14H), 4.28 (t, *J* = 7.8 Hz, 1H), 2.67 (dd, *J* = 14.7, 8.3 Hz, 1H), 2.40 (dd, *J* = 14.6, 7.3 Hz, 1H), 1.86 (s, 6H). <sup>13</sup>C NMR (100 MHz, Chloroform-*d*) δ 167.13, 164.98, 140.95, 140.17, 136.91, 136.69, 133.60, 131.75, 129.05, 129.98, 128.78, 128.51, 128.07, 127.73, 127.52, 126.98, 126.81, 123.60, 51.47, 50.92, 40.61, 38.06, 34.90. HRMS (ESI) *m/z*: calcd for C<sub>33</sub>H<sub>29</sub>F<sub>2</sub>O<sub>3</sub>S [M+H]<sup>+</sup> 533.1893, found: 533.1902.

**d) Synthesis of 1-(2-([1,1'-biphenyl]-4-yl)-1-fluoro-3-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)prop-1-en-1-yl)-1H-imidazole **9**<sup>9l</sup>**

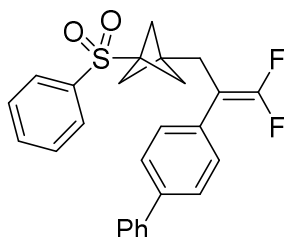


In an oven-dried 10 mL Schlenk tube with a magnetic stir bar was charged with **4a** (87.2 mg, 0.24 mmol, 1.2 equiv.), imidazole (13.6 mg, 0.2 mmol, 1.0 equiv.), K<sub>3</sub>PO<sub>4</sub> (84.9 mg, 0.4 mmol, 2.0 equiv.), and anhydrous DMF (1 mL) was heated at 30 °C constant stirring for 12 h. When the reaction finished, 10 mL of water was added to quench the reaction, and the resulting mixture was extracted with ethyl acetate (10 mL × 3). The combined organic layers were washed with brine (10 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated. The residue was purified by flash chromatography on silica gel with petroleum ether/ethyl acetate = 2:1 (v/v) to afford the desired product **7** as a white solid (84.3 mg, 74%); M.p.: 179-182 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)

$\delta$  7.81 – 7.75 (m, 2H), 7.63 – 7.56 (m, 1H), 7.54 – 7.47 (m, 4H), 7.46 – 7.36 (m, 4H), 7.36 – 7.28 (m, 2H), 7.02 – 6.95 (m, 3H), 6.88 (s, 1H), 2.90 – 2.85 (m, 2H), 1.86 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  143.31 (d,  $J = 260.7$  Hz), 140.84, 139.61, 137.13, 136.64, 133.53, 133.37 (d,  $J = 4.4$  Hz), 129.67, 128.97, 128.71, 128.39, 128.03 (d,  $J = 3.0$  Hz), 127.60, 127.45, 126.78, 118.56, 110.85 (d,  $J = 24.3$  Hz), 51.49, 51.03, 37.78 (d,  $J = 2.6$  Hz), 31.91.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -92.08. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{29}\text{H}_{26}\text{FN}_2\text{O}_2\text{S}$   $[\text{M}+\text{H}]^+$  485.1694, found: 485.1696.

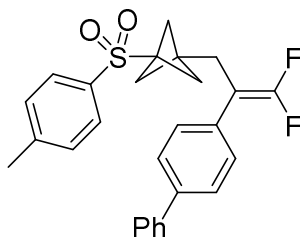
## 6. Characterization of products

### 1-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)-3-(phenylsulfonyl)bicyclo[1.1.1]pentane (**4a**)



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4a** was isolated as a colorless oil (78.6 mg, 85%); M.p.: 138-140 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.80 (d,  $J = 7.6$  Hz, 2H), 7.65 – 7.41 (m, 9H), 7.39 – 7.29 (m, 3H), 2.68 (s, 2H), 1.86 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.33 (dd,  $J = 293.4, 288.0$  Hz), 140.19, 140.12, 136.68, 133.56, 131.92 (t,  $J = 4.0$  Hz), 129.01, 128.78, 128.46, 128.11 (t,  $J = 3.6$  Hz), 127.48, 127.12, 126.87, 89.04 (dd,  $J = 21.8, 13.6$  Hz), 51.41, 50.89, 38.22 – 38.10 (m), 29.35.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -89.06 (d,  $J = 38.8$  Hz, 1F), -89.79 (d,  $J = 38.8$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{26}\text{H}_{23}\text{F}_2\text{O}_2\text{S}$   $[\text{M}+\text{H}]^+$  437.1381, found: 437.1386.

### 1-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)-3-tosylbicyclo[1.1.1]pentane (**4b**)

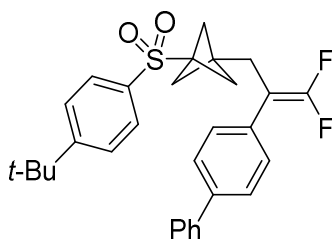


Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4b** was isolated as a colorless oil (61.8 mg, 83 %);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.68 (d,  $J = 6.8$  Hz, 2H), 7.62 – 7.54 (m, 4H), 7.49 – 7.41 (m, 2H), 7.40 – 7.28 (m, 5H), 2.67 (s, 2H), 2.43 (s, 3H), 1.84 (s, 6H).  $^{13}\text{C}$  NMR



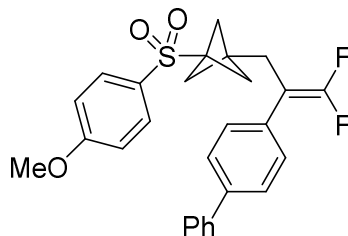
(100 MHz, Chloroform-*d*)  $\delta$  154.38 (dd,  $J = 293.6, 288.0$  Hz), 144.51, 140.22, 140.18, 133.79, 131.99 (t,  $J = 4.0$  Hz), 129.68, 128.81, 128.52, 128.14 (t,  $J = 3.6$  Hz), 127.51, 127.14, 126.91, 89.10 (dd,  $J = 21.8, 13.4$  Hz), 51.48, 50.90, 38.15 – 38.04 (m), 29.42, 21.57.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -89.06 (d,  $J = 38.9$  Hz, 1F), -89.81 (d,  $J = 38.9$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{27}\text{H}_{24}\text{F}_2\text{NaO}_2\text{S}$   $[\text{M}+\text{Na}]^+$  473.1357, found: 473.1361.

**1-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)-3-((4-*tert*-butyl)phenyl)sulfonyl)bicyclo[1.1.1]pentane (4c)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4c** was isolated as a colorless oil (70.8 mg, 66 %);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.72 (d,  $J = 8.0$  Hz, 2H), 7.62 – 7.55 (m, 4H), 7.54 – 7.50 (m, 2H), 7.48 – 7.42 (m, 2H), 7.40 – 7.30 (m, 3H), 2.68 (s, 2H), 1.87 (s, 6H), 1.34 (s, 9H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  157.39, 154.34 (dd,  $J = 293.2, 288.0$  Hz), 140.18, 140.14, 133.70, 131.98 (t,  $J = 4.0$  Hz), 128.78, 128.33, 128.13 (t,  $J = 3.6$  Hz), 127.48, 127.11, 126.88, 126.00, 89.10 (dd,  $J = 21.8, 13.6$  Hz), 51.45, 50.87, 38.16 – 38.05 (m), 35.14, 30.96, 29.40.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -89.07 (d,  $J = 38.8$  Hz, 1F), -89.83 (d,  $J = 38.8$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{30}\text{H}_{31}\text{F}_2\text{O}_2\text{S}$   $[\text{M}+\text{H}]^+$  493.2007, found: 493.2016.

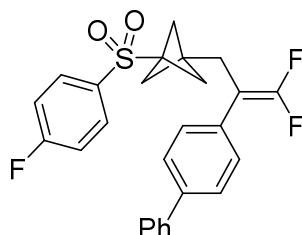
**1-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)-3-((4-methoxyphenyl)sulfonyl) bicyclo[1.1.1]pentane (4d)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4d** was isolated as a colorless oil (60.7 mg, 80 %);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.72 (d,  $J = 8.0$  Hz, 2H), 7.62 – 7.54 (m, 4H), 7.49 – 7.41 (m, 2H), 7.39 – 7.29 (m, 3H), 6.97 (d,  $J = 8.0$  Hz, 2H), 3.85 (s, 3H), 2.67 (s, 2H), 1.84 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  163.61, 154.36 (dd,  $J = 293.4, 288.0$  Hz), 140.18,

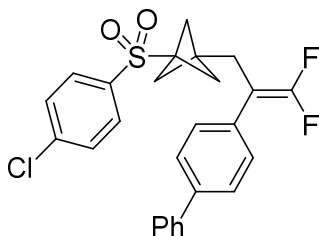
140.16, 131.98 (t,  $J = 4.0$  Hz), 130.59, 128.79, 128.25, 128.13 (t,  $J = 3.6$  Hz), 127.49, 127.12, 126.89, 114.24, 89.10 (dd,  $J = 22.0, 13.4$  Hz), 55.55, 51.60, 50.84, 38.09 – 37.94 (m), 29.39.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -89.06 (d,  $J = 38.8$  Hz, 1F), -89.82 (d,  $J = 38.8$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{27}\text{H}_{25}\text{F}_2\text{O}_3\text{S}$   $[\text{M}+\text{H}]^+$  467.1487, found: 467.1483.

**1-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)-3-((4-fluorophenyl)sulfonyl)bicyclo[1.1.1]pentane (4e)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4e** was isolated as a white solid (59.3 mg, 65 %); M.p.: 130-133 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.86 – 7.75 (m, 2H), 7.63 – 7.55 (m, 4H), 7.49 – 7.42 (m, 2H), 7.39 -7.29 (m, 3H), 7.24 – 7.16 (m, 2H), 2.69 (s, 2H), 1.86 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  165.75 (d,  $J = 256.1$ ), 154.37 (dd,  $J = 293.6, 288.0$  Hz), 140.24, 140.11, 132.81 (d,  $J = 3.0$  Hz), 131.89 (t,  $J = 4.0$  Hz), 131.30 (d,  $J = 9.6$  Hz), 128.81, 128.12 (t,  $J = 3.6$  Hz), 127.52, 127.14, 126.88, 116.39 (d,  $J = 22.4$  Hz), 89.02 (dd,  $J = 21.8, 13.6$  Hz), 51.50, 50.91, 38.30 – 38.17 (m), 29.33.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -89.01 (d,  $J = 39.5$  Hz, 1F), -89.71 (d,  $J = 38.7$  Hz, 1F), -103.62. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{26}\text{H}_{22}\text{F}_3\text{O}_2\text{S}$   $[\text{M}+\text{H}]^+$  455.1287, found: 455.1292.

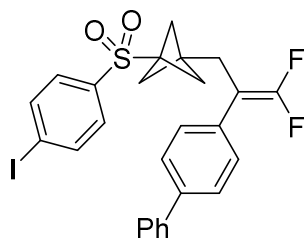
**1-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)-3-((4-chlorophenyl) sulfonyl)bicyclo[1.1.1]pentane (4f)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4f** was isolated as a colorless oil (61.8 mg, 67 %);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.73 (d,  $J = 8.2$  Hz, 2H), 7.62 – 7.55 (m, 4H), 7.52 – 7.42 (m, 4H), 7.40 – 7.30 (m, 3H), 2.69 (s, 2H), 1.86 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.37 (dd,  $J = 293.6, 288.2$  Hz), 140.34, 140.25, 140.11, 135.27, 131.87 (t,  $J =$

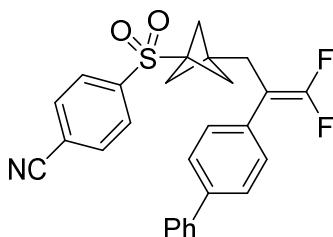
4.0 Hz), 129.96, 129.41, 128.81, 128.11 (t,  $J = 3.6$  Hz), 127.52, 127.15, 126.89, 89.00 (dd,  $J = 21.8$ , 13.6 Hz), 51.43, 50.95, 38.36 – 38.23 (m), 29.33.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -89.09 (d,  $J = 38.8$  Hz, 1F), -89.85 (d,  $J = 38.8$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{26}\text{H}_{22}\text{ClF}_2\text{O}_2\text{S}$   $[\text{M}+\text{H}]^+$  471.0992, found: 471.0994.

**1-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)-3-((4-iodophenyl)sulfonyl)bicyclo[1.1.1]pentane (4g)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4g** was isolated as a colorless oil (77.5 mg, 68 %);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.88 (d,  $J = 8.0$  Hz, 2H), 7.64 – 7.55 (m, 4H), 7.54 – 7.43 (m, 4H), 7.42 – 7.28 (m, 3H), 2.68 (s, 2H), 1.85 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.36 (dd,  $J = 293.6$ , 288.0 Hz), 140.23, 140.10, 138.34, 136.44, 131.86 (t,  $J = 4.0$  Hz), 129.84, 128.80, 128.11 (t,  $J = 3.6$  Hz), 127.51, 127.15, 126.88, 101.60, 88.99 (dd,  $J = 21.8$ , 13.6 Hz), 51.37, 50.94, 38.35 – 38.24 (m), 29.33.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -88.97 (d,  $J = 38.8$  Hz, 1 F), -89.73 (d,  $J = 38.8$  Hz, 1 F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{26}\text{H}_{22}\text{F}_2\text{IO}_2\text{S}$   $[\text{M}+\text{H}]^+$  563.0348, found: 563.0363.

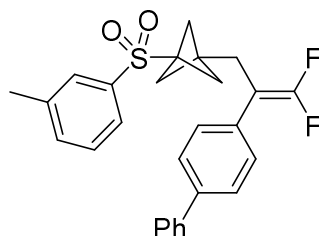
**4-((3-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)bicyclo[1.1.1]pentan-1-yl)sulfonyl)benzotrile (4h)**



Eluent in chromatography: petroleum ether/ethyl acetate 5:1 to 3:1, **4h** was isolated as a colorless oil (62.2 mg, 67 %);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.91 (d,  $J = 8.0$  Hz, 2H), 7.82 (d,  $J = 8.0$  Hz, 2H), 7.61 – 7.55 (m, 4H), 7.48 – 7.42 (m, 2H), 7.40 – 7.30 (m, 3H), 2.69 (s, 2H), 1.87 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.33 (dd,  $J = 293.8$ , 288.2 Hz), 141.01, 140.22, 139.97, 132.78, 131.72 (t,  $J = 4.0$  Hz), 129.17, 128.80, 128.06 (t,  $J = 3.6$  Hz), 127.54, 127.12, 126.81, 117.34,

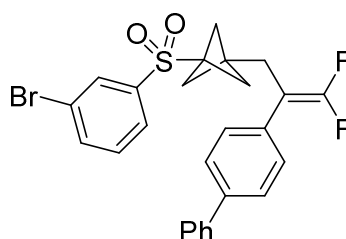
117.04, 88.86 (dd,  $J = 21.8, 13.8$  Hz), 51.29, 51.03, 38.61 – 38.44 (m), 29.21.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -88.88 (d,  $J = 38.6$  Hz, 1F), -89.51 (d,  $J = 38.6$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{27}\text{H}_{22}\text{F}_2\text{NO}_2\text{S}$   $[\text{M}+\text{H}]^+$  462.1334, found: 462.1324.

**1-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)-3-(*m*-tolylsulfonyl)bicyclo[1.1.1]pentane (4i)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4i** was isolated as a colorless oil (62.9 mg, 79 %);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.64 – 7.53 (m, 6H), 7.52 – 7.36 (m, 5H), 7.32 (d,  $J = 8.0$  Hz, 2H), 2.68 (s, 2H), 2.42 (s, 3H), 1.86 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.39 (dd,  $J = 293.4, 288.0$  Hz), 140.24, 140.18, 139.27, 136.63, 134.37, 131.99 (t,  $J = 4.0$  Hz), 128.89, 128.81, 128.74, 128.15 (t,  $J = 3.6$  Hz), 127.52, 127.16, 126.92, 125.68, 89.09 (dd,  $J = 21.8, 13.6$  Hz), 51.42, 50.95, 38.19 – 33.08 (m), 29.42, 21.27.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -89.03 (d,  $J = 39.2$  Hz, 1F), -89.78 (d,  $J = 38.8$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{27}\text{H}_{24}\text{F}_2\text{NaO}_2\text{S}$   $[\text{M}+\text{Na}]^+$  473.1357, found: 473.1364.

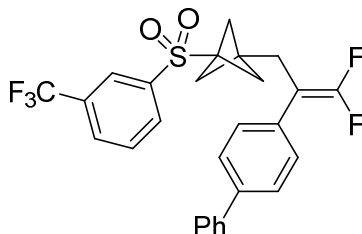
**1-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)-3-((4-bromophenyl)sulfonyl) bicyclo[1.1.1]pentane (4j)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4j** was isolated as a colorless oil (66.5 mg, 64 %);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.95 (s, 1H), 7.79 – 7.68 (m, 2H), 7.62 – 7.54 (m, 4H), 7.50 – 7.30 (m, 6H), 2.69 (s, 2H), 1.88 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.40 (dd,  $J = 293.6, 288.2$  Hz), 140.31, 140.14, 138.77, 136.69, 131.88 (t,  $J = 4.0$  Hz), 131.31, 130.59, 128.82, 128.13 (t,  $J = 3.6$  Hz), 127.53, 127.20, 127.11, 126.93, 123.13, 89.01 (dd,  $J = 21.8, 13.8$  Hz), 51.47, 51.08, 38.44 – 38.32 (m), 29.38.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -88.94 (d,

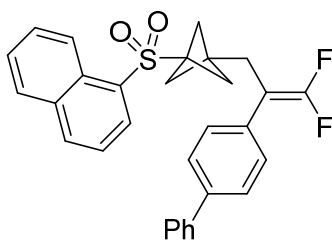
$J = 38.2$  Hz, 1F),  $-89.64$  (d,  $J = 38.8$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $C_{26}H_{22}BrF_2O_2S$   $[M+H]^+$  515.0486, found: 515.0486.

**1-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)-3-((3-(trifluoromethyl)phenyl)sulfonyl)bicyclo[1.1.1]pentane (4k)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4k** was isolated as a colorless oil (70.6 mg, 70 %);  $^1H$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  8.09 (s, 1H), 8.00 (d,  $J = 7.8$  Hz, 1H), 7.90 (d,  $J = 7.8$  Hz, 1H), 7.72 – 7.65 (m, 1H), 7.63 – 7.55 (m, 4H), 7.50 – 7.42 (m, 2H), 7.40 – 7.30 (m, 3H), 2.75 – 2.65 (m, 2H), 1.89 (s, 1H).  $^{13}C$  NMR (100 MHz, Chloroform- $d$ )  $\delta$  154.38 (dd,  $J = 293.6, 288.1$  Hz), 140.32, 140.11, 138.16, 132.35 – 131.28 (m, 3C), 130.29 (q,  $J = 3.3$  Hz), 129.91, 128.81, 128.12 (t,  $J = 3.5$  Hz), 127.54, 127.18, 126.90, 125.52 (q,  $J = 3.8$  Hz), 123.05 (d,  $J = 273.0$  Hz), 88.96 (dd,  $J = 21.9, 13.8$  Hz), 51.41, 51.03, 38.54 – 38.40 (m), 29.31.  $^{19}F$  NMR (376 MHz, )  $\delta$   $-62.78$  (3F),  $-88.96$  (d,  $J = 38.9$  Hz, 1F),  $-89.65$  (d,  $J = 38.1$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $C_{27}H_{21}F_5NaO_2S$   $[M+Na]^+$  527.1075, found: 527.1098.

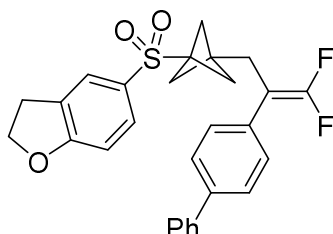
**1-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)-3-(naphthalen-1-ylsulfonyl)bicyclo[1.1.1]pentane (4m)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4m** was isolated as a white solid (57.5 mg, 59%); M.p.: 149-151 °C;  $^1H$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  8.73 (d,  $J = 8.5$  Hz, 1H), 8.26 – 8.19 (m, 1H), 8.10 (d,  $J = 8.2$  Hz, 1H), 7.95 – 7.90 (m, 1H), 7.66 – 7.50 (m, 7H), 7.50 – 7.43 (m, 2H), 7.39 – 7.34 (m, 1H), 7.31 – 7.25 (m, 2H), 2.68 – 2.55 (m, 2H), 1.87 (s, 6H).  $^{13}C$  NMR (100 MHz, Chloroform- $d$ )  $\delta$  154.29 (dd,  $J = 293.5, 288.0$  Hz), 140.17, 140.14, 135.04, 133.96, 132.67, 131.90 (t,  $J = 4.0$  Hz), 131.09, 129.70, 128.77, 128.75, 128.09 (t,  $J = 3.7$  Hz), 128.03, 127.47,

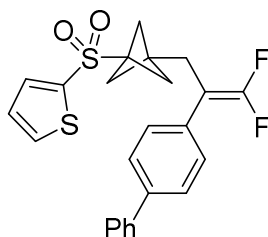
127.09, 126.88, 126.87, 125.04, 124.38, 89.04 (dd,  $J = 21.9, 13.6$  Hz), 52.34, 51.44, 37.34 – 37.21 (m), 29.25.  $^{19}\text{F}$  NMR (376 MHz, Chloroform- $d$ )  $\delta$  -89.04 (d,  $J = 39.2$  Hz, 1F), -89.79 (d,  $J = 38.8$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{30}\text{H}_{25}\text{F}_2\text{O}_2\text{S}$   $[\text{M}+\text{H}]^+$  487.1538, found: 487.1535.

**5-((3-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)bicyclo[1.1.1]pentan-1-yl)sulfonyl)-2,3-dihydrobenzofuran (4n)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4n** was isolated as a white solid (73.6 mg, 74 %); M.p.: 140-143 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  7.62 – 7.53 (m, 6H), 7.48 – 7.41 (m, 2H), 7.38 – 7.31 (m, 3H), 6.83 (d,  $J = 8.2$  Hz, 1H), 4.66 (t,  $J = 8.9$  Hz, 2H), 3.23 (t,  $J = 8.9$  Hz, 2H), 2.68 (s, 2H), 1.85 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform- $d$ )  $\delta$  164.56, 154.32 (dd,  $J = 293.6, 288.0$  Hz), 140.09, 131.96 (t,  $J = 4.0$  Hz), 130.12, 128.76, 128.34, 128.13, 128.10, 128.07, 127.46, 127.06, 126.83, 125.52, 109.52, 89.09 (dd,  $J = 21.8, 13.4$  Hz), 72.26, 51.56, 50.80, 38.03 – 37.78 (m), 29.36, 28.83.  $^{19}\text{F}$  NMR (376 MHz, Chloroform- $d$ )  $\delta$  -89.19 (d,  $J = 39.0$  Hz, 1F), -89.96 (d,  $J = 39.4$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{28}\text{H}_{25}\text{F}_2\text{O}_3\text{S}$   $[\text{M}+\text{H}]^+$  479.1487, found: 479.1494.

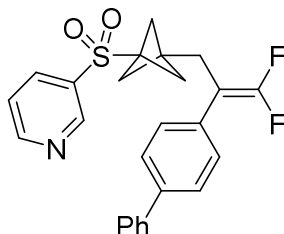
**2-((3-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)bicyclo[1.1.1]pentan-1-yl)sulfonyl)thiophene (4o)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4o** was isolated as a white solid (68.7 mg, 75 %); M.p.: 123-126 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  7.73 – 7.67 (dd,  $J = 4.9, 1.3$  Hz, 1H), 7.63 – 7.55 (m, 5H), 7.49 – 7.42 (m, 2H), 7.39 – 7.31 (m, 3H), 7.16 – 7.11 (m, 1H), 2.75 – 2.64 (m, 2H), 1.91 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform- $d$ )  $\delta$  154.35 (dd,  $J = 293.4, 288.0$  Hz), 140.22, 140.12, 137.59, 134.33, 134.19, 131.91 (t,  $J = 4.0$  Hz), 128.78, 128.12 (t,  $J = 3.6$

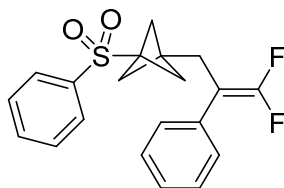
Hz), 127.90, 127.49, 127.14, 126.89, 89.05 (dd,  $J = 21.8, 13.6$  Hz), 52.18, 51.01, 37.57 – 37.46 (m), 29.28.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -89.06 (d,  $J = 38.8$  Hz, 1F), -89.78 (d,  $J = 38.8$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{24}\text{H}_{21}\text{F}_2\text{O}_2\text{S}_2$   $[\text{M}+\text{H}]^+$  443.0946, found: 443.0950.

**3-((3-(2-([1,1'-biphenyl]-4-yl)-3,3-difluoroallyl)bicyclo[1.1.1]pentan-1-yl)sulfonyl)pyridine (4p)**



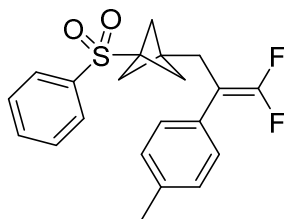
Eluent in chromatography: petroleum ether/ethyl acetate 3:1 to 2:1, **4p** was isolated as a white solid (60.1 mg, 70 %);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  9.00 (s, 1H), 8.87 – 8.81 (m, 1H), 8.12 – 8.05 (m, 1H), 7.62 – 7.53 (m, 4H), 7.50 – 7.42 (m, 3H), 7.39 – 7.29 (m, 3H), 2.69 (s, 2H), 1.88 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.37 (dd,  $J = 293.6, 288.2$  Hz), 154.09, 149.40, 140.30, 140.08, 136.25, 133.40, 131.77 (t,  $J = 4.0$  Hz), 128.79, 128.09 (t,  $J = 3.6$  Hz), 127.52, 127.18, 126.89, 123.72, 88.92 (dd,  $J = 21.8, 13.8$  Hz), 51.69, 51.00, 38.59 – 38.46 (m), 29.28.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -89.00 (d,  $J = 38.6$  Hz, 1F), -89.66 (d,  $J = 38.2$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{25}\text{H}_{22}\text{F}_2\text{NO}_2\text{S}$   $[\text{M}+\text{H}]^+$  438.1334, found: 438.1338.

**1-(3,3-difluoro-2-phenylallyl)-3-(phenylsulfonyl)bicyclo[1.1.1]pentane (4q)**



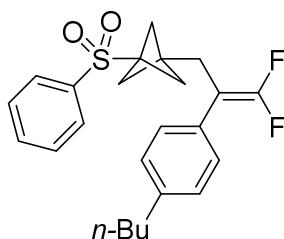
Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4q** was isolated as a yellow liquid (44.2 mg, 64%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.79 (d,  $J = 7.6$  Hz, 2H), 7.66 - 7.59 (m, 1H), 7.55 – 7.48 (m, 2H), 7.36 – 7.28 (m, 2H), 7.27 – 7.20 (m, 3H), 2.64 (s, 2H), 1.81 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.28 (dd,  $J = 293.0, 287.8$  Hz), 136.78, 133.57, 133.08 (t,  $J = 4.0$  Hz), 129.03, 128.56, 128.51, 127.83 (t,  $J = 3.6$  Hz), 127.53, 89.35 (dd,  $J = 21.8, 14.0$  Hz), 51.45, 50.91, 38.23 – 38.12 (m), 29.52.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -89.75 (d,  $J = 39.6$  Hz, 1F), -90.50 (d,  $J = 39.6$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{20}\text{H}_{19}\text{F}_2\text{O}_2\text{S}$   $[\text{M}+\text{H}]^+$  361.1068, found: 361.1086.

**1-(3,3-difluoro-2-(p-tolyl)allyl)-3-(phenylsulfonyl)bicyclo[1.1.1]pentane (4r)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4r** was isolated as a colorless oil (58 mg, 80%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.79 (d,  $J = 7.7$  Hz, 2H), 7.67 – 7.59 (m, 1H), 7.56 – 7.48 (m, 2H), 7.12 (s, 4H), 2.61 (s, 2H), 2.34 (s, 3H), 1.81 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.13 (dd,  $J = 292.3, 287.2$  Hz), 137.27, 136.75, 133.51, 129.93 (t,  $J = 3.9$  Hz), 129.20, 128.97, 128.44, 127.61 (t,  $J = 3.4$  Hz), 89.12 (dd,  $J = 21.4, 14.0$  Hz), 51.39, 50.86, 38.20 – 38.06 (m), 29.40, 21.02.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -90.46 (d,  $J = 40.9$  Hz, 1F), -91.04 (d,  $J = 40.9$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{21}\text{H}_{21}\text{F}_2\text{O}_2\text{S}$   $[\text{M}+\text{H}]^+$  375.1225, found: 375.1224.

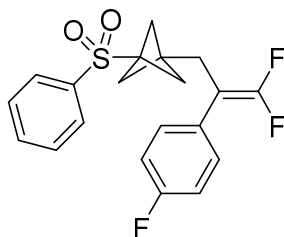
**1-(2-(4-butylphenyl)-3,3-difluoroallyl)-3-(phenylsulfonyl)bicyclo[1.1.1]pentane (4s)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4s** was isolated as a colorless oil (68.4 mg, 82%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.79 (d,  $J = 7.6$  Hz, 2H), 7.65 – 7.60 (m, 1H), 7.54 – 7.49 (m, 2H), 7.13 (s, 4H), 2.63 – 2.55 (m, 4H), 1.81 (s, 6H), 1.61 – 1.57 (m, 2H), 1.37 – 1.31 (m, 2H), 0.93 (t,  $J = 7.4$  Hz, 3H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.21 (dd,  $J = 292.6, 287.2$  Hz), 142.33, 136.82, 133.54, 130.17 (t,  $J = 3.8$  Hz), 129.01, 128.57, 128.51, 127.62 (t,  $J = 3.4$  Hz), 89.16 (dd,  $J = 21.4, 13.8$  Hz), 51.44, 50.92, 38.27 – 38.16 (m), 35.21, 33.40, 29.48, 22.30, 13.92.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -90.19 (d,  $J = 40.6$  Hz, 1F), -90.81 (d,  $J = 40.6$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{24}\text{H}_{27}\text{F}_2\text{O}_2\text{S}$   $[\text{M}+\text{H}]^+$  417.1694, found: 417.1713.

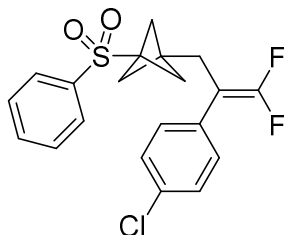


**1-(3,3-difluoro-2-(4-fluorophenyl)allyl)-3-(phenylsulfonyl)bicyclo[1.1.1]pentane (4t)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4t** was isolated as a colorless oil (59.9 mg, 78%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.81 – 7.75 (m, 2H), 7.64 – 7.59 (m, 1H), 7.54 – 7.49 (m, 2H), 7.22 – 7.17 (m, 2H), 7.04 – 6.98 (m, 2H), 2.62 – 2.57 (m, 2H), 1.79 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  161.85 (d,  $J = 247.7$  Hz), 154.24 (dd,  $J = 292.6, 287.6$  Hz), 136.78, 133.56, 129.70 – 129.40 (m, 2C), 129.02, 128.46, 115.56 (d,  $J = 21.6$  Hz), 88.60 (dd,  $J = 22.4, 14.0$  Hz), 51.45, 50.88, 38.08 – 37.95 (m), 29.60.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -89.82 (d,  $J = 39.6$  Hz, 1F), -90.59 (d,  $J = 40.4$  Hz, 1F), -113.87. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{20}\text{H}_{17}\text{F}_3\text{NaO}_2\text{S}$   $[\text{M}+\text{Na}]^+$  401.0794, found: 401.0805.

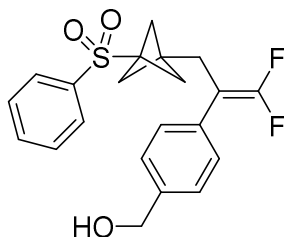
**1-(2-(4-chlorophenyl)-3,3-difluoroallyl)-3-(phenylsulfonyl)bicyclo[1.1.1]pentane (4u)**



Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4u** was isolated as a colorless oil (38.7 mg, 51%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.79 (d,  $J = 7.8$  Hz, 2H), 7.66 – 7.61 (m, 1H), 7.57 – 7.51 (m, 2H), 7.30 (d,  $J = 8.2$  Hz, 2H), 7.17 (d,  $J = 8.0$  Hz, 2H), 2.61 (s, 2H), 1.81 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.26 (dd,  $J = 293.4, 288.4$  Hz), 136.66, 133.60, 133.32, 131.51 (t,  $J = 4.0$  Hz), 129.08 (t,  $J = 3.0$  Hz), 129.04, 128.77, 128.47, 88.60 (dd,  $J = 22.4, 13.6$  Hz), 51.40, 50.85, 38.08 – 37.95 (m), 29.36.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -89.59 (d,  $J = 38.0$  Hz, 1F), -90.31 (d,  $J = 38.0$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{20}\text{H}_{17}\text{ClF}_2\text{NaO}_2\text{S}$   $[\text{M}+\text{Na}]^+$  417.0498, found: 417.0508.

**(4-(1,1-difluoro-3-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)prop-1-en-2-yl)phenyl)**

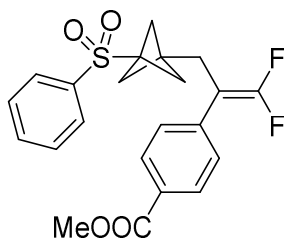
**methanol (4v)**



Eluent in chromatography: petroleum ether/ethyl acetate 5:1 to 3:1, **4v** was isolated as a colorless oil (40.9 mg, 52%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.78 (d,  $J$  = 7.2 Hz, 2H), 7.64 – 7.59 (m, 1H), 7.54 – 7.49 (m, 2H), 7.32 (d,  $J$  = 7.8 Hz, 2H), 7.22 (d,  $J$  = 7.0 Hz, 2H), 4.68 (s, 2H), 2.64 – 2.60 (m, 2H), 2.01 (br, 1H), 1.80 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.33 (dd,  $J$  = 293.0, 287.8 Hz), 140.31, 136.78, 133.57, 132.26 (t,  $J$  = 3.0 Hz), 129.02, 128.47, 127.93 (t,  $J$  = 3.4 Hz), 127.04, 89.14 (dd,  $J$  = 21.8, 14.0 Hz), 64.64, 51.45, 50.94, 38.20 – 38.05 (m), 29.45.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -89.51 (d,  $J$  = 38.1 Hz, 1 F), -90.14 (d,  $J$  = 38.2 Hz, 1 F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{21}\text{H}_{21}\text{F}_2\text{O}_3\text{S}$   $[\text{M}+\text{H}]^+$  391.1174, found: 391.1163.

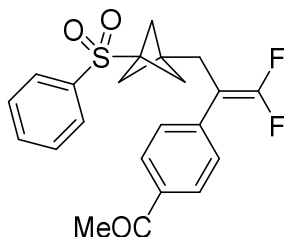
**Methyl 4-(1,1-difluoro-3-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)prop-1-en-2-yl)**

**benzoate (4w)**



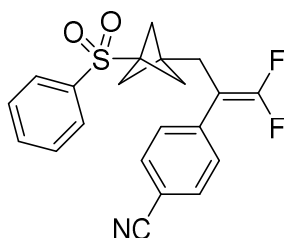
Eluent in chromatography: petroleum ether/ethyl acetate 5:1 to 3:1, **4w** was isolated as a colorless solid (60.2 mg, 72%); M.p.: 100-102 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.00 – 7.96 (m, 2H), 7.79 – 7.72 (m, 2H), 7.63 – 7.58 (m, 1H), 7.53 – 7.48 (m, 2H), 7.33 – 7.29 (m, 2H), 3.90 (s, 3H), 2.68 – 2.62 (m, 2H), 1.79 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  166.44, 154.60 (dd,  $J$  = 295.2, 289.4 Hz), 137.88 (t,  $J$  = 4.2 Hz), 136.81, 133.58, 129.78, 129.23, 129.04, 128.49, 127.73 (t,  $J$  = 3.6 Hz), 89.15 (dd,  $J$  = 22.4, 13.2 Hz), 52.11, 51.43, 50.92, 38.11 – 37.98 (m), 29.28.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -87.24 (d,  $J$  = 34.8 Hz, 1F), -88.00 (d,  $J$  = 34.8 Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{22}\text{H}_{21}\text{F}_2\text{O}_4\text{S}$   $[\text{M}+\text{H}]^+$  419.1123, found: 419.1134.

**1-(4-(1,1-difluoro-3-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)prop-1-en-2-yl)phenyl)ethan-1-one (4x)**



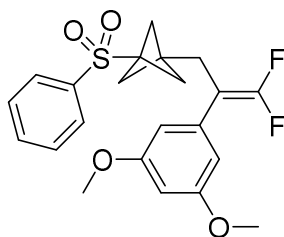
Eluent in chromatography: petroleum ether/ethyl acetate 5:1 to 3:1, **4x** was isolated as a colorless oil (56.8 mg, 74%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.91 – 7.87 (m, 2H), 7.78 – 7.72 (m, 2H), 7.63 – 7.58 (m, 1H), 7.53 – 7.48 (m, 2H), 7.35 – 7.30 (m, 2H), 2.67 – 2.63 (m, 2H), 2.58 (s, 3H), 1.79 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  197.33, 154.61 (dd,  $J$  = 295.4, 289.4 Hz), 138.06 (t,  $J$  = 4.2 Hz), 136.65, 135.98, 133.64, 129.08, 128.57, 128.51, 127.92 (t,  $J$  = 3.6 Hz), 89.12 (dd,  $J$  = 22.6, 13.0 Hz), 51.42, 50.90, 38.18 – 38.01 (m), 29.22, 26.57.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -86.97 (d,  $J$  = 33.7 Hz, 1F), -87.82 (d,  $J$  = 33.7 Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{22}\text{H}_{21}\text{F}_2\text{O}_3\text{S}$   $[\text{M}+\text{H}]^+$  403.1174, found: 403.1186.

**4-(1,1-difluoro-3-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)prop-1-en-2-yl)benzotrile (4y)**



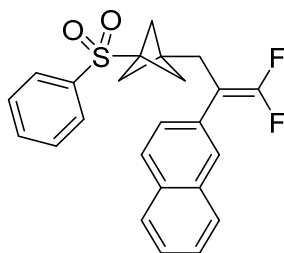
Eluent in chromatography: petroleum ether/ethyl acetate 5:1 to 3:1, **4y** was isolated as a colorless oil (41.7 mg, 54%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.80 – 7.76 (m, 2H), 7.65 – 7.59 (m, 3H), 7.56 – 7.50 (m, 2H), 7.39 – 7.33 (m, 2H), 2.71 – 2.60 (m, 2H), 1.81 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.77 (dd,  $J$  = 295.2 Hz, 288.4 Hz), 138.12 (t,  $J$  = 4.4 Hz), 136.59, 133.71, 132.36, 129.12, 128.52, 128.39 (t,  $J$  = 4.2 Hz), 118.32, 111.28, 88.83 (dd,  $J$  = 23.2, 12.6 Hz), 51.41, 50.88, 38.05 – 37.90 (m), 29.16.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -85.69 (d,  $J$  = 31.3 Hz, 1F), -86.83 (d,  $J$  = 31.3 Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{21}\text{H}_{18}\text{F}_2\text{NO}_2\text{S}$   $[\text{M}+\text{H}]^+$  386.1021, found: 386.1035.

**1-(2-(3,5-dimethoxyphenyl)-3,3-difluoroallyl)-3-(phenylsulfonyl)bicyclo[1.1.1]pentane (4z)**



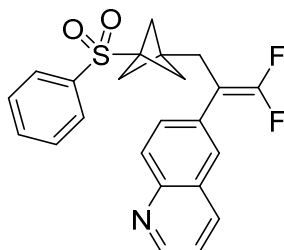
Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4z** was isolated as a colorless solid (65.6 mg, 80%); M.p.: 100-102 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.79 (d, *J* = 7.5 Hz, 2H), 7.65 – 7.60 (m, 1H), 7.55 – 7.49 (m, 2H), 6.37 (s, 3H), 3.77 (s, 6H), 2.59 (s, 2H), 1.83 (s, 6H). <sup>13</sup>C NMR (100 MHz, Chloroform-*d*) δ 160.74, 154.36 (dd, *J* = 293.0, 288.0 Hz), 136.82, 135.02 (t, *J* = 3.2 Hz), 133.58, 129.05, 128.54, 106.33 (t, *J* = 3.6 Hz), 99.13, 89.43 (dd, *J* = 21.2, 14.4 Hz), 55.32, 51.45, 50.96, 38.23 – 38.12 (m), 29.54. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -88.90 (d, *J* = 38.8 Hz, 1F), -89.06 (d, *J* = 38.8 Hz, 1F). HRMS (ESI) *m/z*: calcd for C<sub>22</sub>H<sub>23</sub>F<sub>2</sub>O<sub>4</sub>S [M+H]<sup>+</sup> 421.1280, found: 421.1292.

**1-(3,3-difluoro-2-(naphthalen-2-yl)allyl)-3-(phenylsulfonyl)bicyclo[1.1.1]pentane (4aa)**



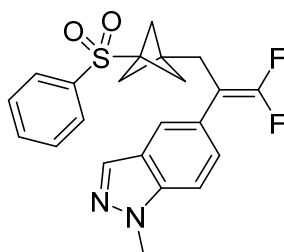
Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4aa** was isolated as a colorless solid (50.8 mg, 61%); M.p.: 132-134 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.89 – 7.80 (m, 2H), 7.79 - 7.71 (m, 3H), 7.64 – 7.57 (m, 1H), 7.54 – 7.46 (m, 4H), 7.46 – 7.40 (m, 1H), 7.33 – 7.28 (m, 1H), 2.74 (s, 2H), 1.79 (s, 6H). <sup>13</sup>C NMR (100 MHz, Chloroform-*d*) δ 154.01 (t, *J* = 289.4 Hz), 136.72, 133.82, 133.52, 130.98, 130.77 – 130.60 (m), 128.98, 128.77, 128.68, 128.48, 127.46 – 127.32 (m), 126.46, 126.04, 125.15, 124.54, 87.51 (dd, *J* = 22.2, 18.2 Hz), 51.62, 50.98, 38.27 – 38.12 (m), 31.42. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -86.95 (d, *J* = 38.8 Hz, 1F), -90.97 (d, *J* = 38.8 Hz, 1F). HRMS (ESI) *m/z*: calcd for C<sub>24</sub>H<sub>21</sub>F<sub>2</sub>O<sub>2</sub>S [M+H]<sup>+</sup> 411.1225, found: 411.1227.

**6-(1,1-difluoro-3-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)prop-1-en-2-yl)quinoline (4ab)**



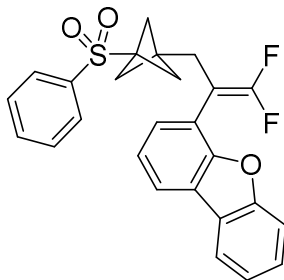
Eluent in chromatography: petroleum ether/ethyl acetate 3:1 to 2:1, **4ab** was isolated as a colorless oil (25.2 mg, 62%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.81 (s, 1H), 8.09 (d,  $J = 8.4$  Hz 1H), 8.03 – 8.00 (m, 1H), 7.80 – 7.71 (m, 4H), 7.64 – 7.56 (m, 2H), 7.53 – 7.48 (m, 2H), 2.80 – 2.75 (m, 2H), 1.85 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  174.23, 154.92 (dd,  $J = 294.6, 290.2$  Hz), 149.20 – 149.00 (m), 146.44, 136.54, 134.93 (t,  $J = 3.8$  Hz), 133.67, 130.21, 129.08, 128.69, 128.48, 127.74, 127.48, 126.44 (t,  $J = 3.6$  Hz), 86.96 (dd,  $J = 23.8, 13.8$  Hz), 51.48, 50.91, 38.08 – 37.92 (m), 29.28.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -86.84 (d,  $J = 34.2$  Hz, 1F), -88.31 (d,  $J = 34.2$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{23}\text{H}_{20}\text{F}_2\text{NO}_2\text{S}$  [ $\text{M}+\text{H}$ ] $^+$  412.1177, found: 412.1193.

**5-(1,1-difluoro-3-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)prop-1-en-2-yl)-1-methyl-1H-indazole (4ac)**



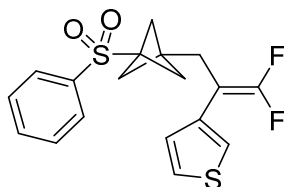
Eluent in chromatography: petroleum ether/ethyl acetate 3:1 to 2:1, **4ac** was isolated as a colorless oil (42.0 mg, 43%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.94 (s, 1H), 7.75 (d,  $J = 7.8$  Hz, 2H), 7.62 – 7.55 (m, 2H), 7.52 – 7.46 (m, 2H), 7.37 – 7.32 (m, 1H), 7.28 – 7.23 (m, 1H), 4.06 (s, 3H), 2.68 (s, 2H), 1.78 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  154.25 (dd,  $J = 291.2, 287.4$  Hz), 139.00, 136.72, 133.54, 132.67, 129.00, 128.46, 126.58 (t,  $J = 3.2$  Hz), 125.43 (t,  $J = 3.6$  Hz), 123.93, 120.45 (t,  $J = 3.2$  Hz), 109.20, 89.39 (dd,  $J = 21.8, 14.4$  Hz), 51.47, 50.90, 38.20 – 38.09 (m), 35.52, 30.06.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -90.71 (d,  $J = 39.8$  Hz, 1F), -91.47 (d,  $J = 39.8$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{22}\text{H}_{21}\text{F}_2\text{N}_2\text{O}_2\text{S}$  [ $\text{M}+\text{H}$ ] $^+$  415.1286, found: 415.1305.

**4-(1,1-difluoro-3-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)prop-1-en-2-yl)dibenzo[b,d]furan (4ad)**



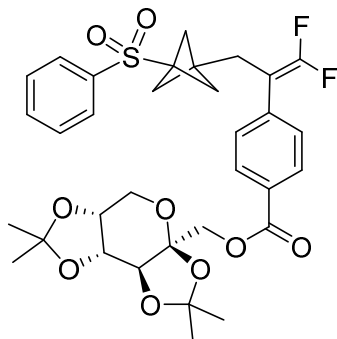
Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4ae** was isolated as a colorless oil (74.3 mg, 85%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.95 – 7.88 (m, 1H), 7.86 (dd,  $J = 7.4$ , 1.6 Hz, 1H), 7.73 – 7.66 (m, 2H), 7.55 – 7.48 (m, 2H), 7.47 – 7.37 (m, 3H), 7.36 – 7.24 (m, 2H), 7.25 – 7.22 (m, 1H), 2.83 – 2.79 (m, 2H), 1.73 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  155.85, 154.19 (dd,  $J = 292.4$ , 289.0 Hz), 153.38, 136.74, 133.46, 128.92, 128.44, 127.45, 127.38 (t,  $J = 2.6$  Hz), 124.68, 123.86, 122.99, 122.89, 120.74, 120.40, 117.71–117.59 (m), 111.73, 85.31 (dd,  $J = 24.8$ , 16.4 Hz), 51.44, 50.83, 38.29 – 38.18 (m), 29.45.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -86.92 (d,  $J = 34.9$  Hz, 1F), -90.06 (d,  $J = 34.9$  Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{26}\text{H}_{21}\text{F}_2\text{O}_3\text{S}$  [M+H] $^+$  451.1174, found: 451.1175.

**3-(1,1-difluoro-3-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)prop-1-en-2-yl)thiophene (4ae)**



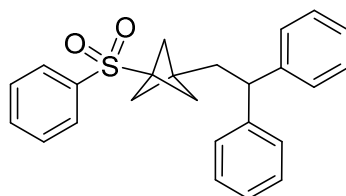
Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **4ae** was isolated as a colorless oil (54 mg, 74%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.86 – 7.77 (m, 2H), 7.65 – 7.60 (m, 1H), 7.55 – 7.50 (m, 2H), 7.32 – 7.27 (m, 1H), 7.11 – 7.05 (m, 2H), 2.62 – 2.59 (m, 2H), 1.86 (s, 6H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  154.59 (dd,  $J = 295.0$ , 287.2 Hz), 136.77, 133.61, 133.18 (t,  $J = 4.5$  Hz), 129.07, 128.53, 126.50 (dd,  $J = 6.6$ , 2.4 Hz), 125.93, 121.78 (t,  $J = 5.5$  Hz), 85.50 (dd,  $J = 24.1$ , 13.2 Hz), 51.27, 50.90, 38.16 – 38.04 (m), 29.13.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -86.42 (d,  $J = 38.1$  Hz), -90.70 (d,  $J = 38.1$  Hz). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{18}\text{H}_{17}\text{F}_2\text{O}_2\text{S}_2$  [M+H] $^+$  367.0633, found: 367.0646.

**((3aS,5aR,8aR,8bS)-2,2,7,7-tetramethyltetrahydro-3aH-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-3a-yl)methyl 4-(1,1-difluoro-3-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)prop-1-en-2-yl)benzoate (4af)**



Eluent in chromatography: petroleum ether/ethyl acetate 5:1 to 3:1, **4af** was isolated as a colorless oil (68.3 mg, 56%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.02 (d,  $J$  = 8.5 Hz, 2H), 7.82 – 7.73 (m, 2H), 7.64 – 7.59 (m, 1H), 7.51 (t,  $J$  = 7.6 Hz, 2H), 7.34 – 7.27 (m, 2H), 4.69 – 4.60 (m, 2H), 4.44 (d,  $J$  = 2.6 Hz, 1H), 4.33 (d,  $J$  = 11.8 Hz, 1H), 4.26 (dd,  $J$  = 7.8, 1.7 Hz, 1H), 3.95 (dd,  $J$  = 13.0, 1.9 Hz, 1H), 3.79 (d,  $J$  = 13.0 Hz, 1H), 2.76 – 2.54 (m, 2H), 1.79 (s, 6H), 1.55 (s, 3H), 1.45 (s, 3H), 1.37 (s, 3H), 1.34 (s, 3H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform-*d*)  $\delta$  165.33, 154.57 (dd,  $J$  = 295.5, 289.6 Hz), 138.07 (t,  $J$  = 4.3 Hz), 136.63, 133.62, 129.95, 129.06, 128.88, 128.48, 127.71 (t,  $J$  = 3.7 Hz), 109.09, 108.79, 101.54, 89.07 (dd,  $J$  = 22.5, 13.1 Hz), 70.70, 70.52, 70.01, 65.49, 61.29, 51.37, 50.84, 38.04 (dd,  $J$  = 4.0, 2.2 Hz), 29.20, 26.47, 25.81, 25.46, 23.95.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -87.05 (d,  $J$  = 34.0 Hz, 1F), -87.83 (d,  $J$  = 33.8 Hz, 1F). HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{33}\text{H}_{36}\text{F}_2\text{NaO}_9\text{S}$  [ $\text{M}+\text{Na}$ ] $^+$  669.1940, found: 669.1953.

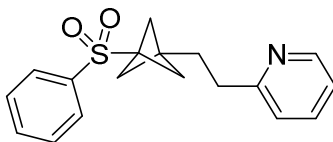
**1-(2,2-diphenylethyl)-3-(phenylsulfonyl)bicyclo[1.1.1]pentane (6a)**



Following the general procedure for the preparation of product **4** (eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1), **6a** was isolated as a colorless solid (27.1 mg, 69%);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.81 – 7.74 (m, 2H), 7.64 – 7.58 (m, 1H), 7.64 – 7.48 (m, 2H), 7.27 – 7.21 (m, 4H), 7.21 – 7.12 (m, 6H), 3.88 (t,  $J$  = 7.8 Hz, 1H), 2.31 (d,  $J$  = 7.8 Hz, 2H), 1.73 (s,

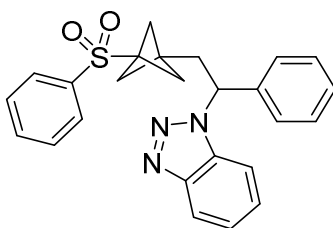
6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform- $d$ )  $\delta$  144.03, 137.03, 133.42, 128.95, 128.57, 128.51, 127.47, 126.45, 51.63, 51.06, 49.15, 38.80, 36.42. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{25}\text{H}_{24}\text{NaO}_2\text{S}$   $[\text{M}+\text{Na}]^+$  411.1389, found: 411.1391.

**2-(2-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)ethyl)pyridine (6b)**



Following the general procedure for the preparation of product **4** (eluent in chromatography: petroleum ether/ethyl acetate 3:1 to 2:1), **6b** was isolated as a colorless oil (33.9 mg, 36%).  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  8.50 – 8.45 (m, 1H), 7.87 – 7.78 (m, 2H), 7.66 – 7.60 (m, 1H), 7.59 – 7.50 (m, 3H), 7.14 – 7.04 (m, 2H), 2.75 – 2.66 (m, 2H), 2.00 – 1.93 (m, 2H), 1.88 (s, 6H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform- $d$ )  $\delta$  160.63, 149.15, 136.87, 136.39, 133.47, 128.96, 128.48, 122.62, 121.21, 51.32, 50.45, 39.29, 34.72, 30.39. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{18}\text{H}_{19}\text{NNaO}_2\text{S}$   $[\text{M}+\text{Na}]^+$  336.1029, found: 336.1030.

**1-(1-phenyl-2-(3-(phenylsulfonyl)bicyclo[1.1.1]pentan-1-yl)ethyl)-1H-benzo[d][1, 2,3]triazole (6c)**



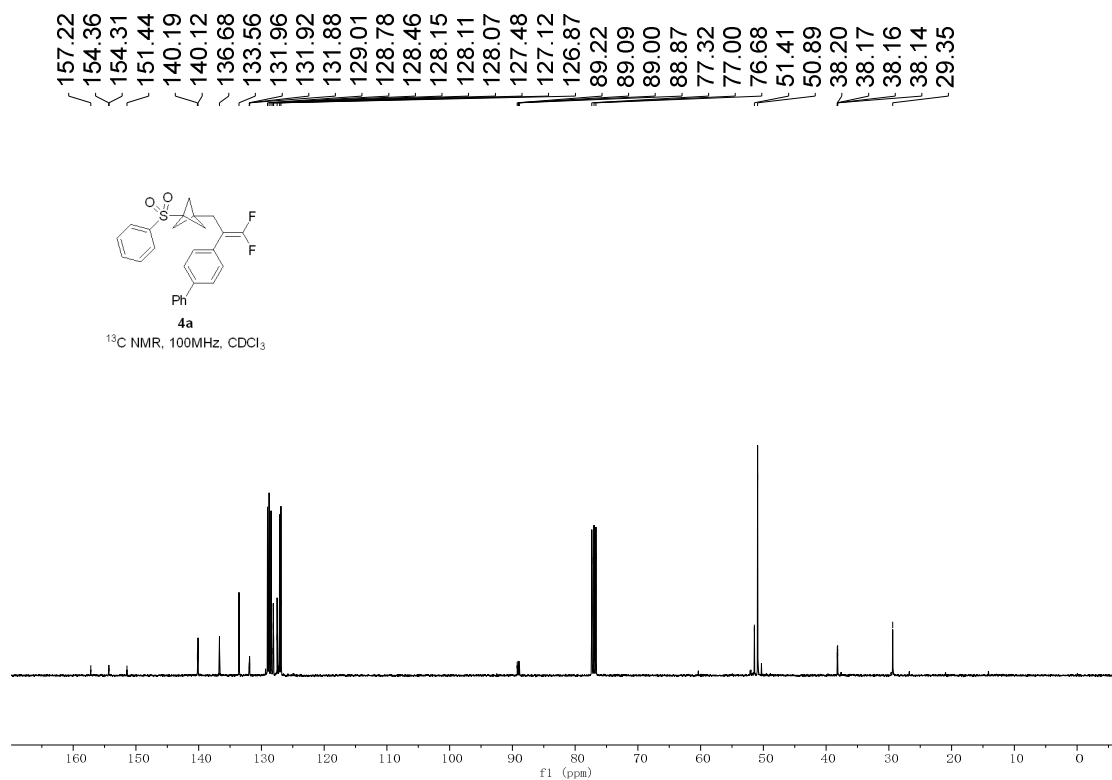
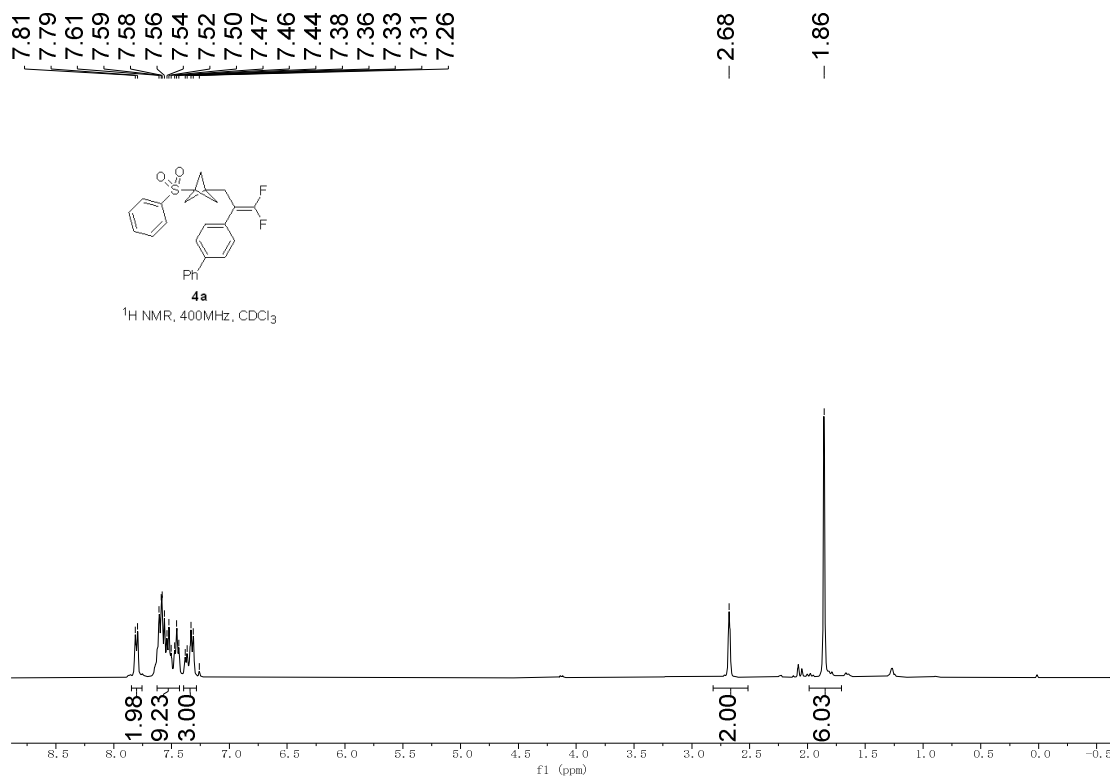
Following the general procedure for the preparation of product **4** (eluent in chromatography: petroleum ether/ethyl acetate 5:1 to 3:1), **6c** was isolated as a colorless oil (64.7 mg, 75%).  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  8.02 (d,  $J = 8.2$  Hz, 1H), 7.72 (d,  $J = 7.2$  Hz, 2H), 7.63 – 7.57 (m, 1H), 7.48 (t,  $J = 7.7$  Hz, 2H), 7.42 – 7.36 (m, 2H), 7.35 – 7.21 (m, 6H), 5.72 (dd,  $J = 10.1, 5.2$  Hz, 1H), 3.20 (dd,  $J = 14.9, 10.1$  Hz, 1H), 2.60 (dd,  $J = 14.9, 5.2$  Hz, 1H), 1.79 (dd,  $J = 9.4, 2.0$  Hz, 3H), 1.64 (dd,  $J = 9.3, 2.0$  Hz, 3H).  $^{13}\text{C}$  NMR (100 MHz, Chloroform- $d$ )  $\delta$  146.10, 138.46, 136.48, 133.59, 132.43, 129.00, 128.97, 128.56, 128.40, 127.51, 126.50, 124.14, 120.06, 109.28, 61.21, 51.26, 50.81, 37.32, 35.60. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{25}\text{H}_{23}\text{N}_3\text{NaO}_2\text{S}$   $[\text{M}+\text{Na}]^+$  452.1403, found: 452.1401.

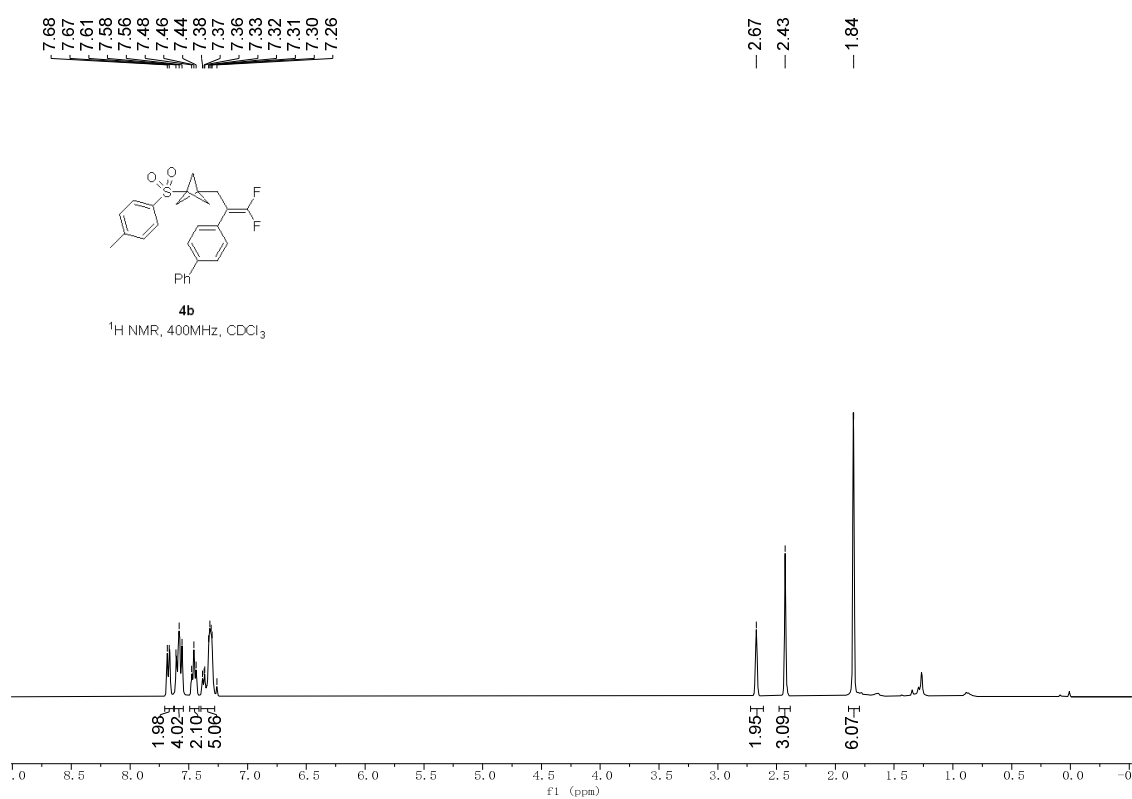
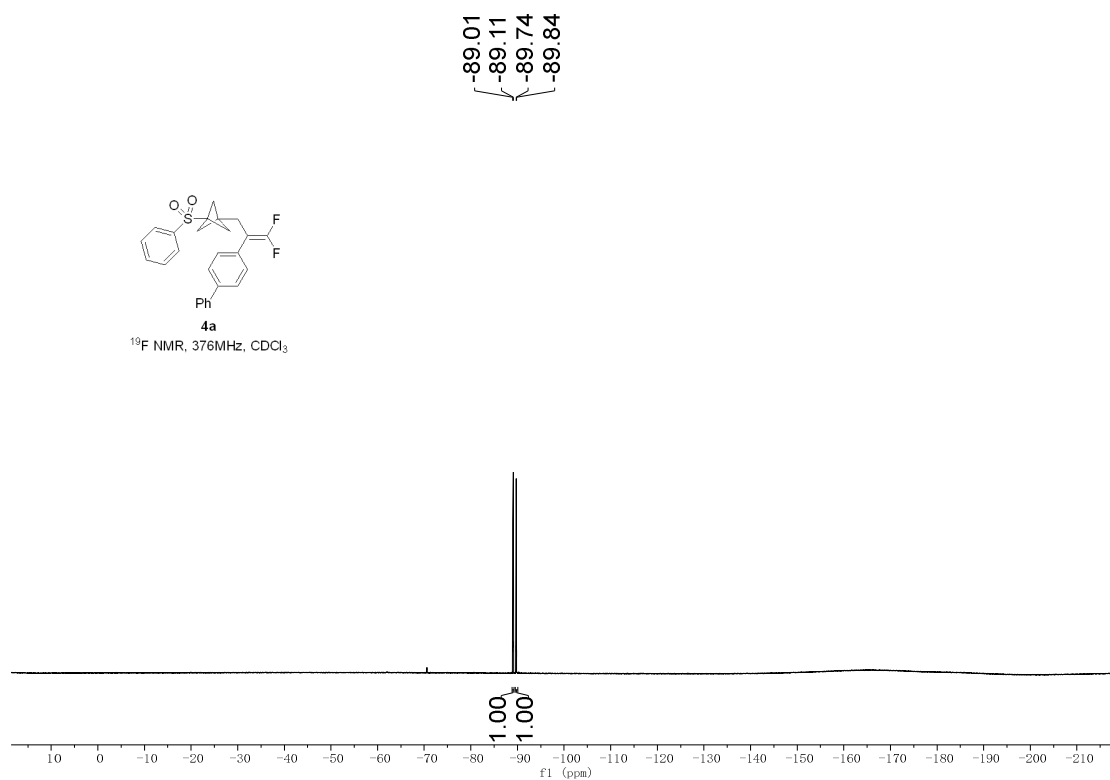


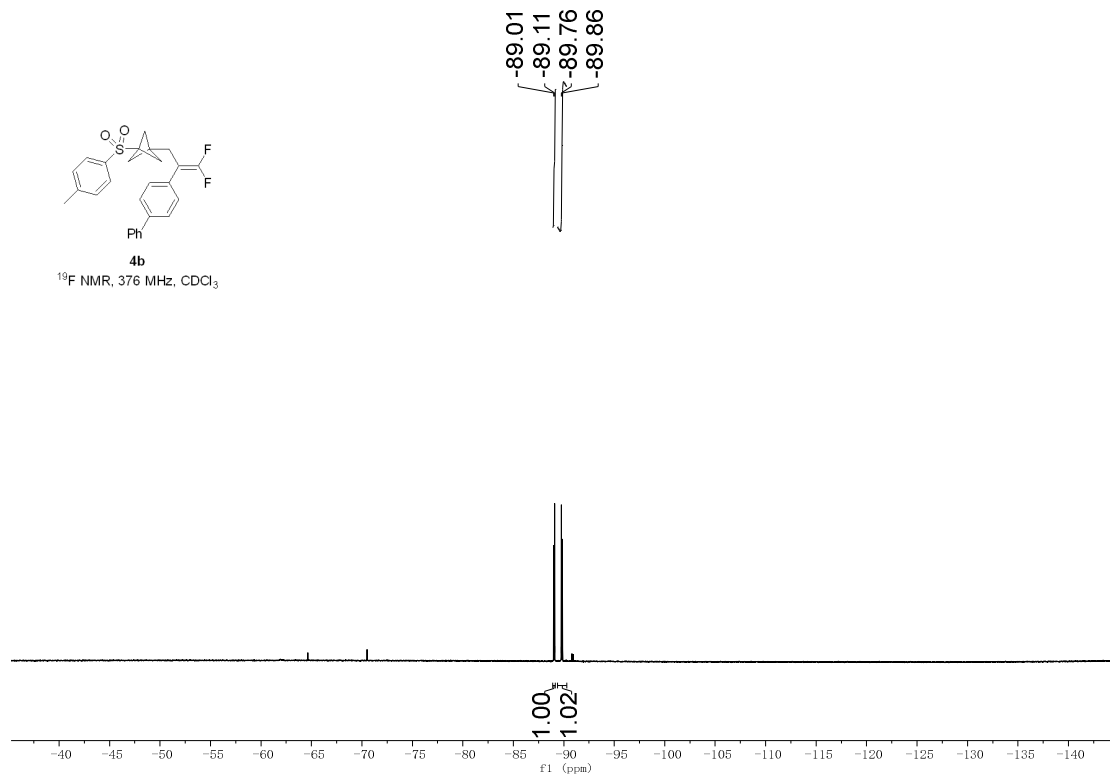
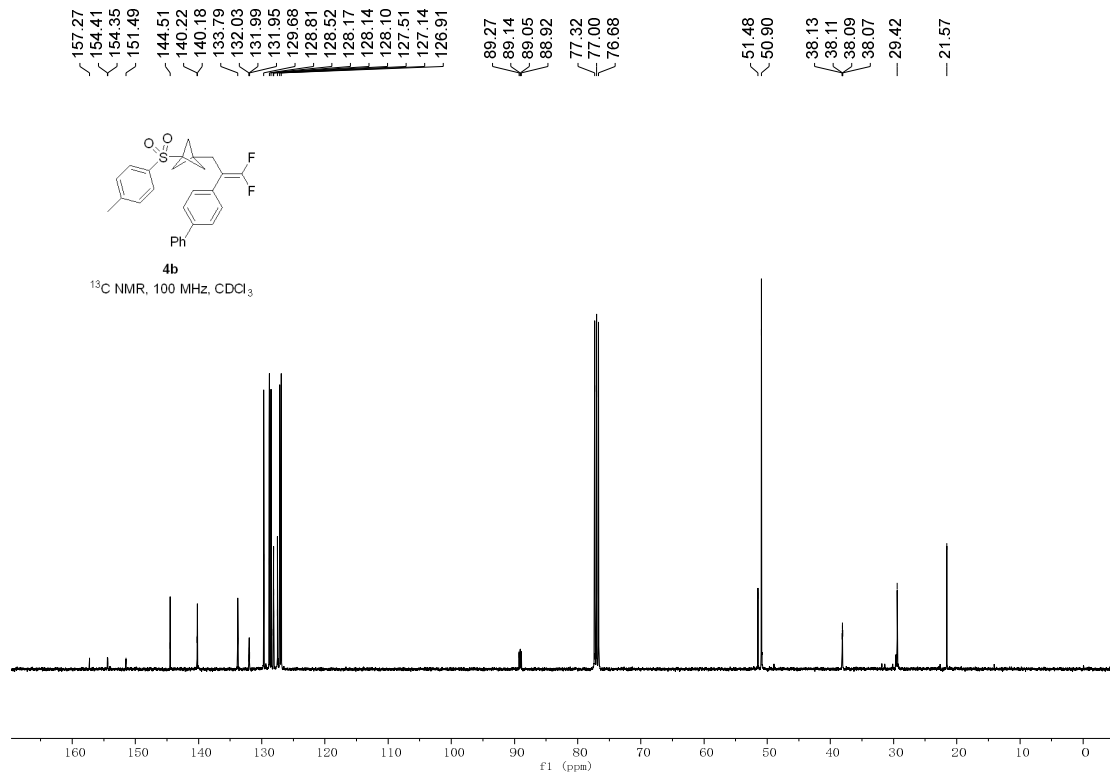
## 7. References

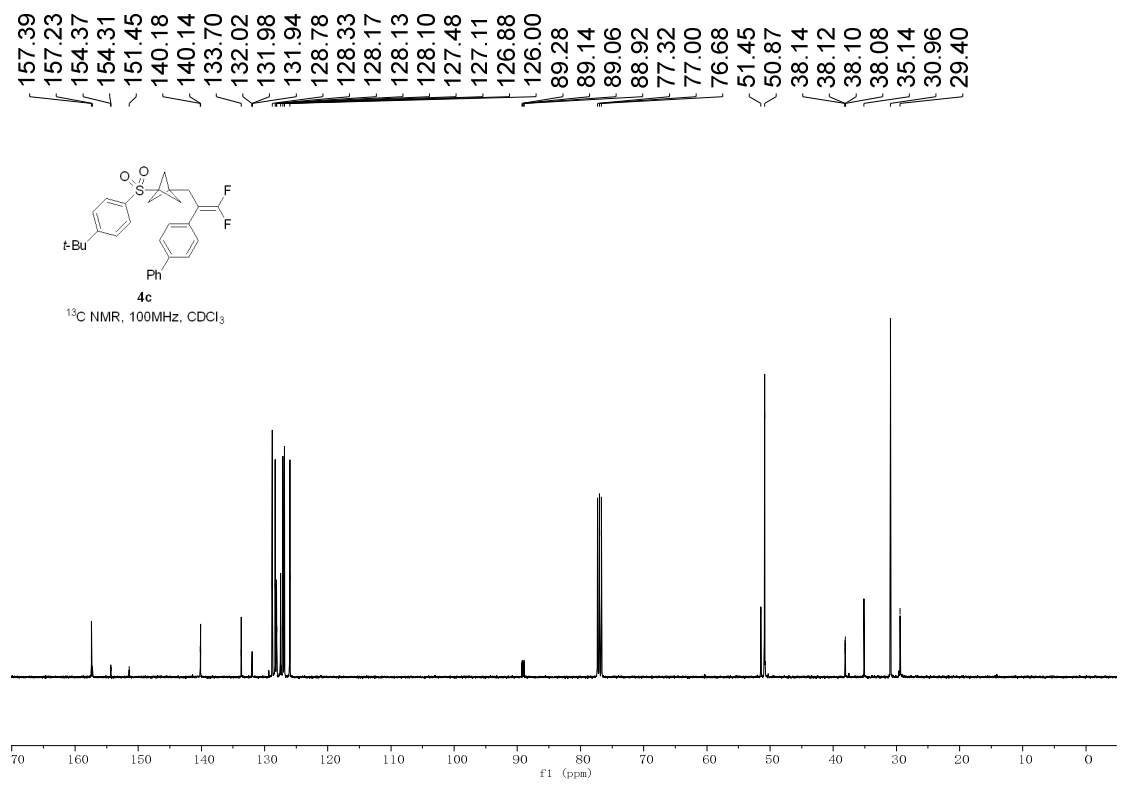
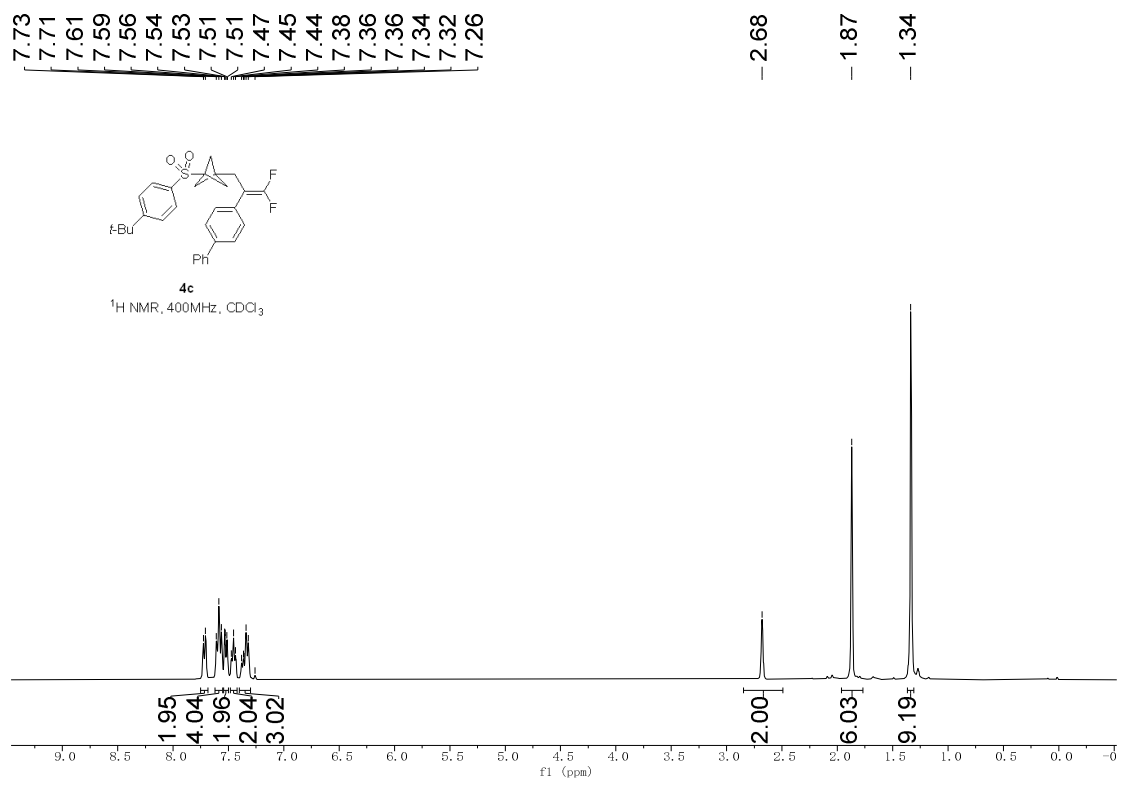
- [1] (a) Y.-X. Chen, Z.-J. Wang, J.-A. Xiao, K. Chen, H.-Y. Xiang and H. Yang, *Org. Lett.*, 2021, **23**, 6558-6562; (b) S. Xie, Y. Li, P. Liu and P. Sun, *Org. Lett.*, 2020, **22**, 8774-8779.
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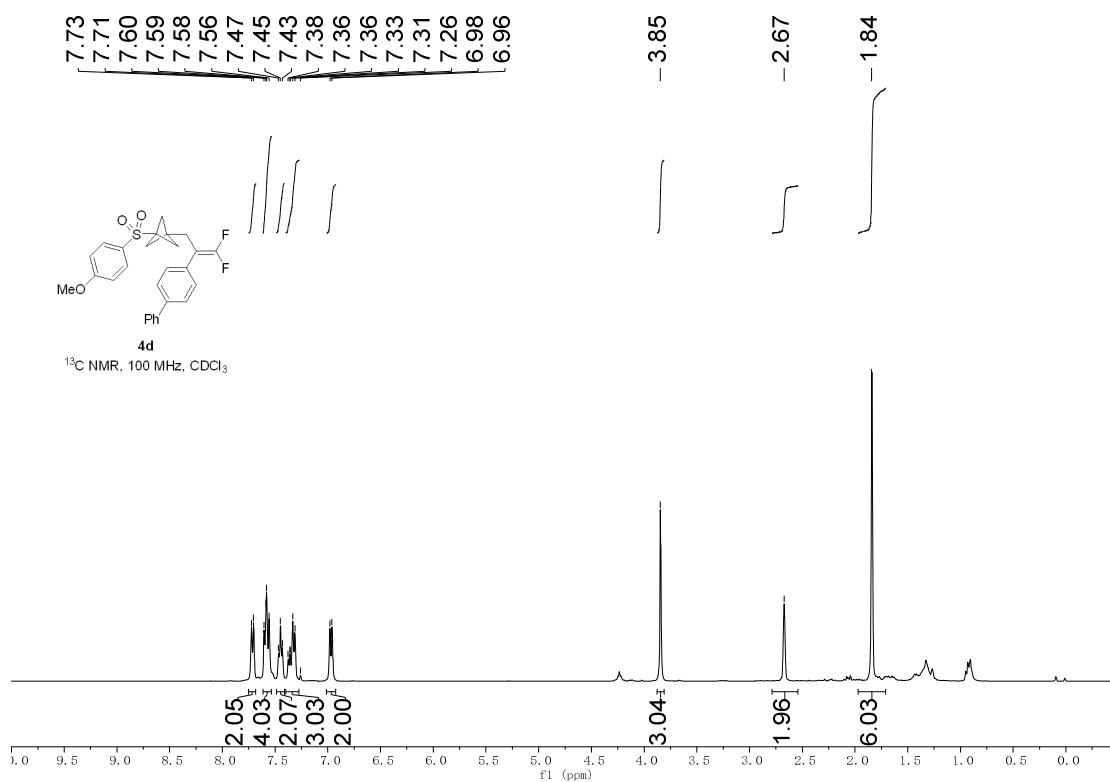
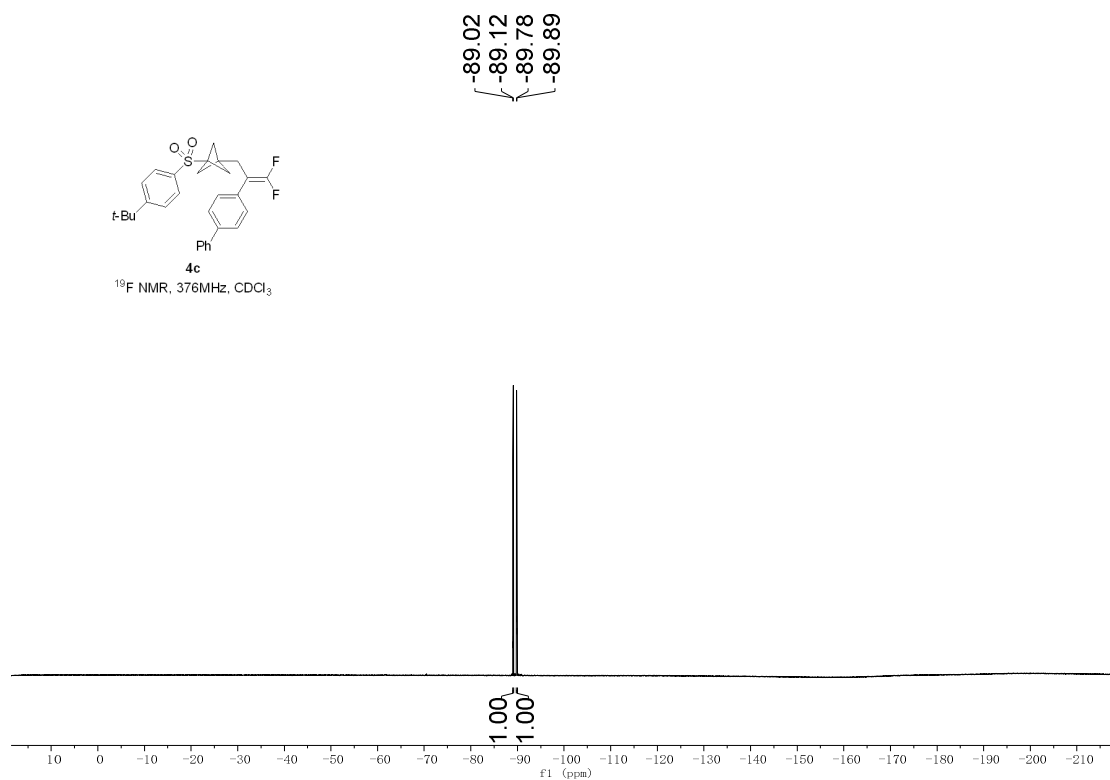
## 8. Copies of $^1\text{H}$ , $^{13}\text{C}$ , and $^{19}\text{F}$ NMR spectra of all products



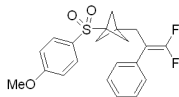






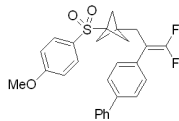
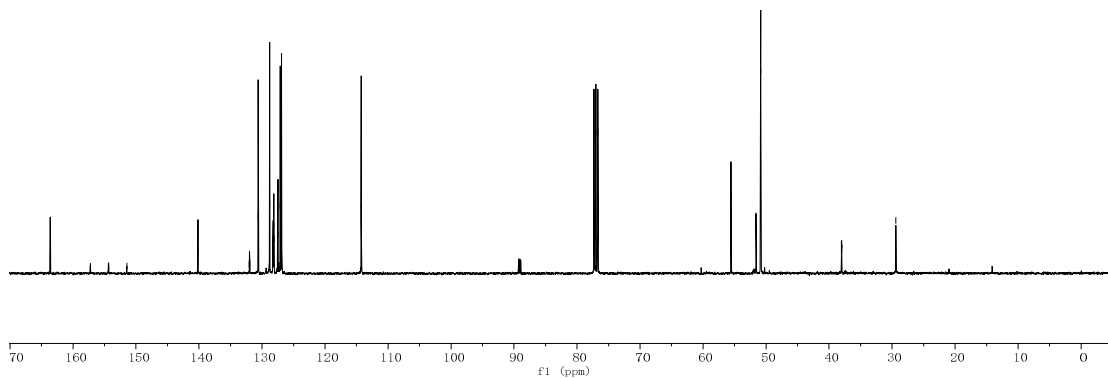


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 131.94  
 130.59  
 128.79  
 128.25  
 128.17  
 128.13  
 128.09  
 127.49  
 127.12  
 126.89  
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 89.28  
 89.15  
 89.06  
 88.93  
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 29.39



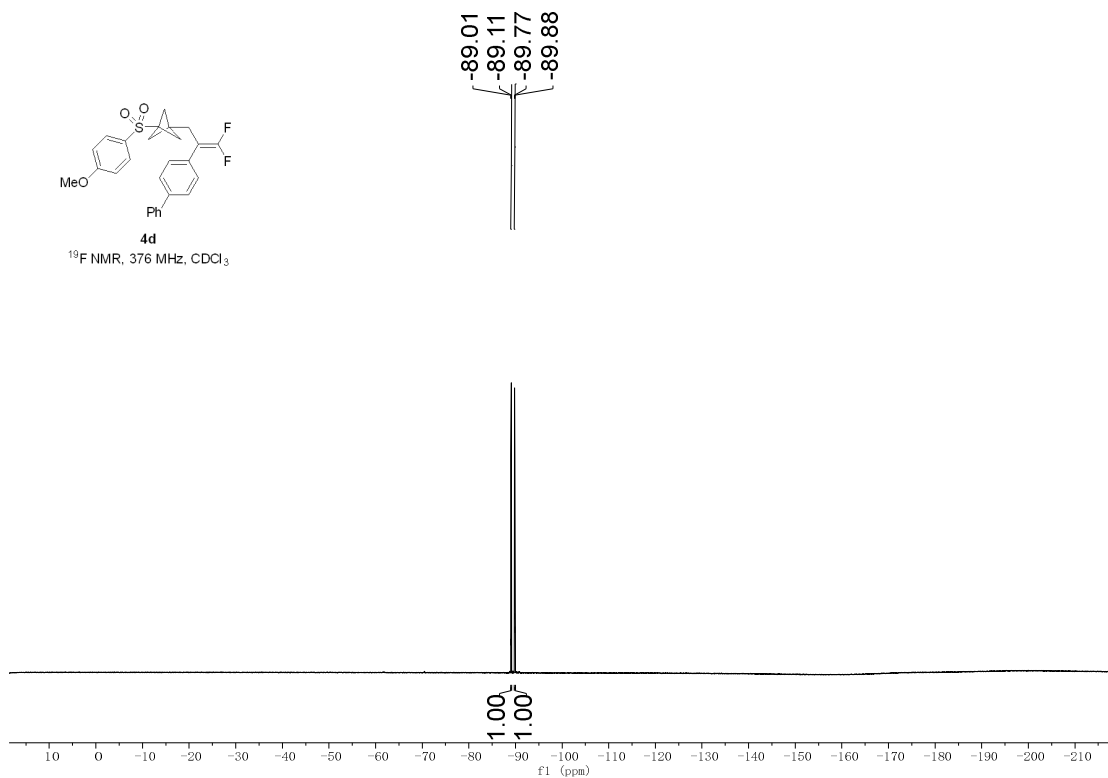
4d

<sup>13</sup>C NMR, 100 MHz, CDCl<sub>3</sub>



4d

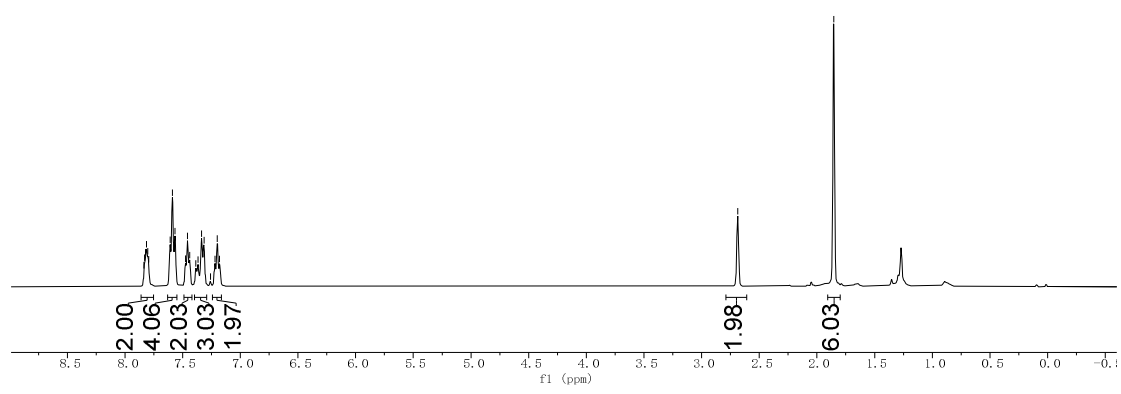
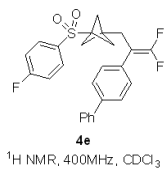
<sup>19</sup>F NMR, 376 MHz, CDCl<sub>3</sub>



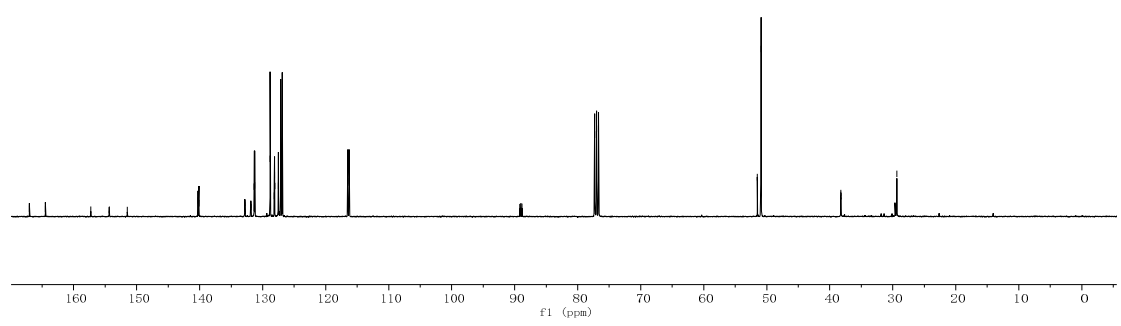
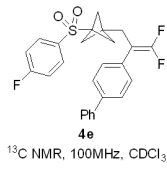
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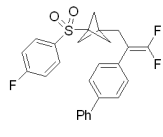
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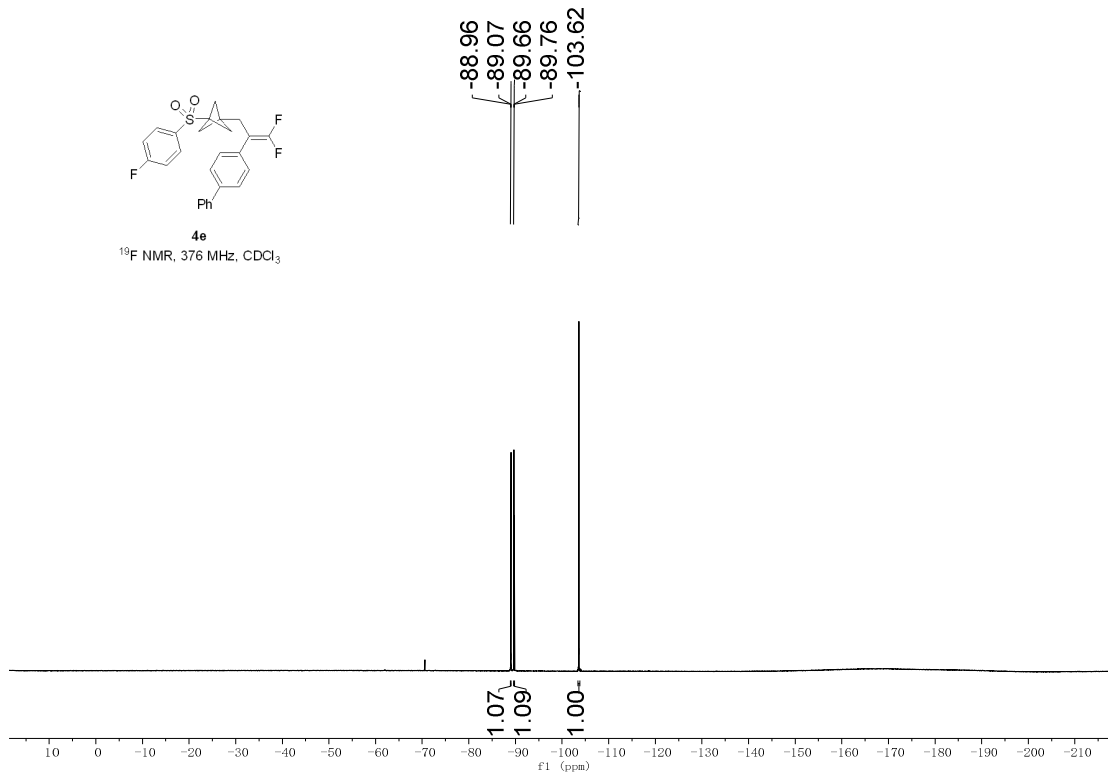
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88.84  
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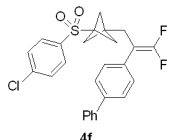
<sup>19</sup>F NMR, 376 MHz, CDCl<sub>3</sub>



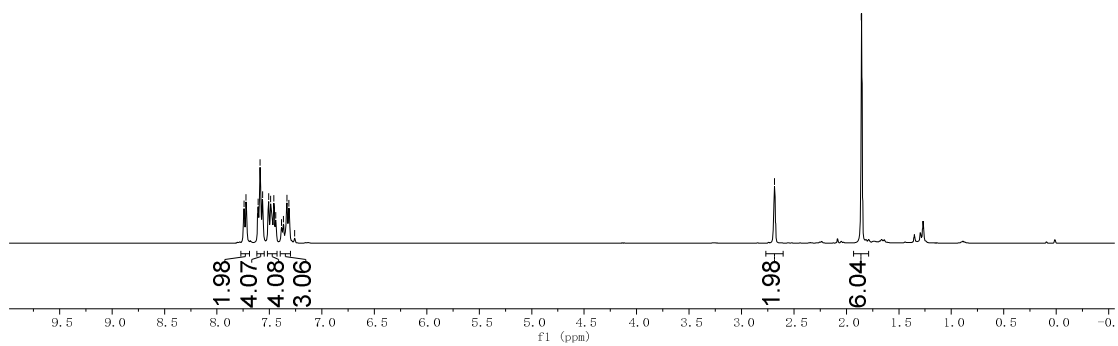
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7.33  
7.31  
7.26

- 2.69

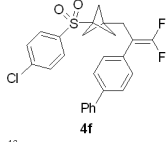
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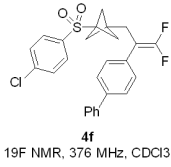
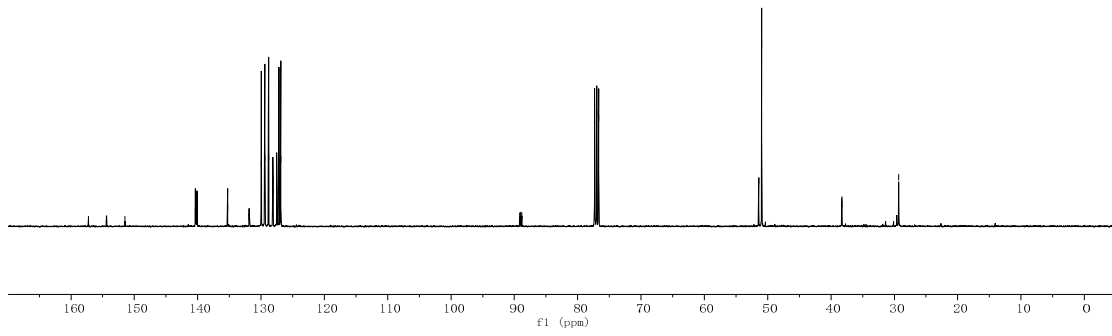
<sup>1</sup>H NMR, 400MHz, CDCl<sub>3</sub>



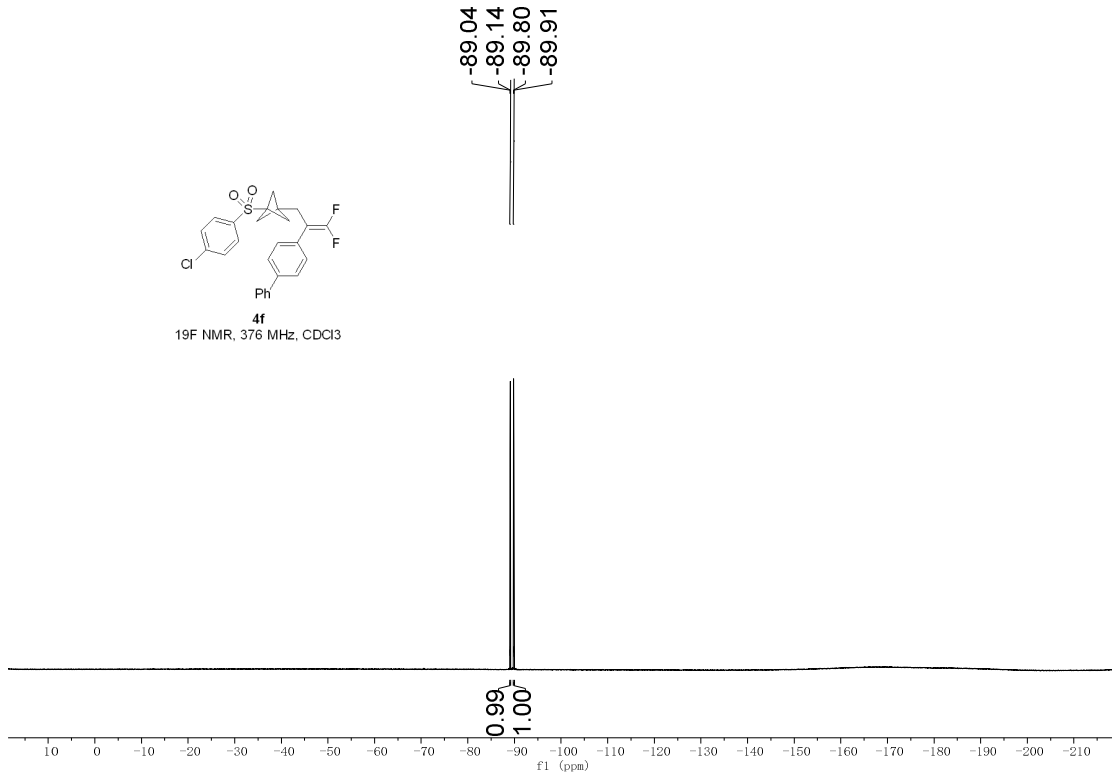
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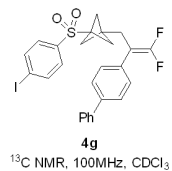
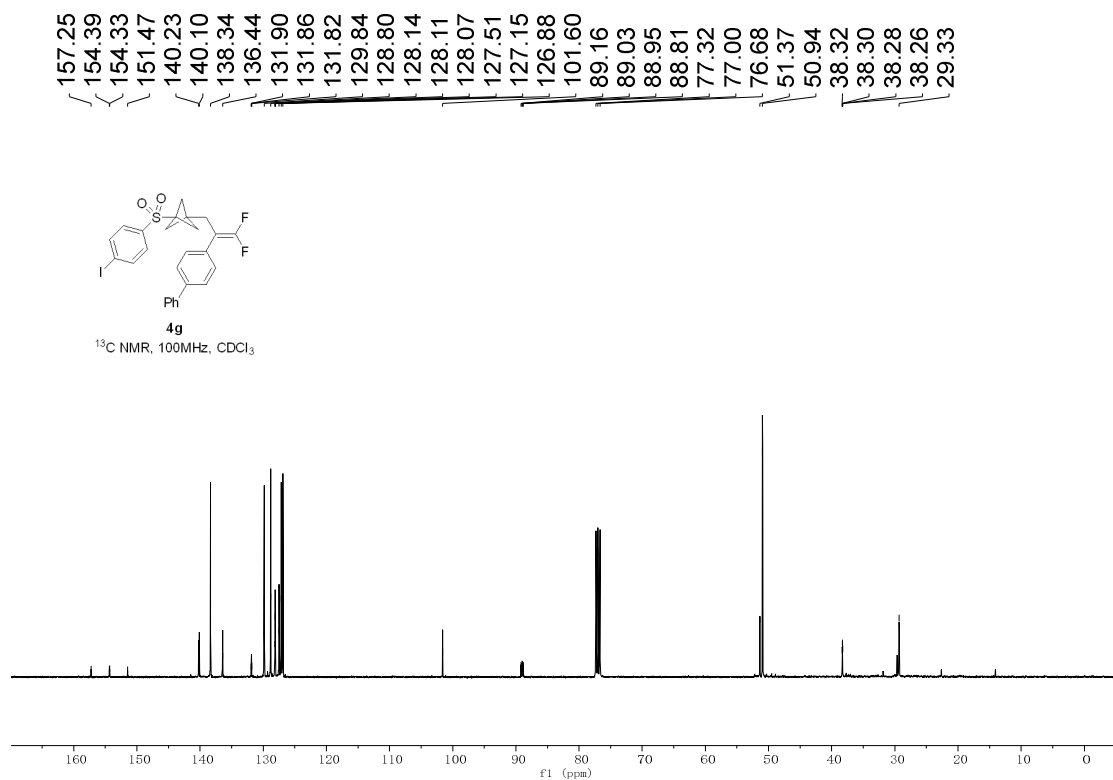
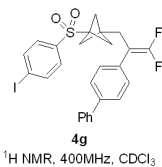
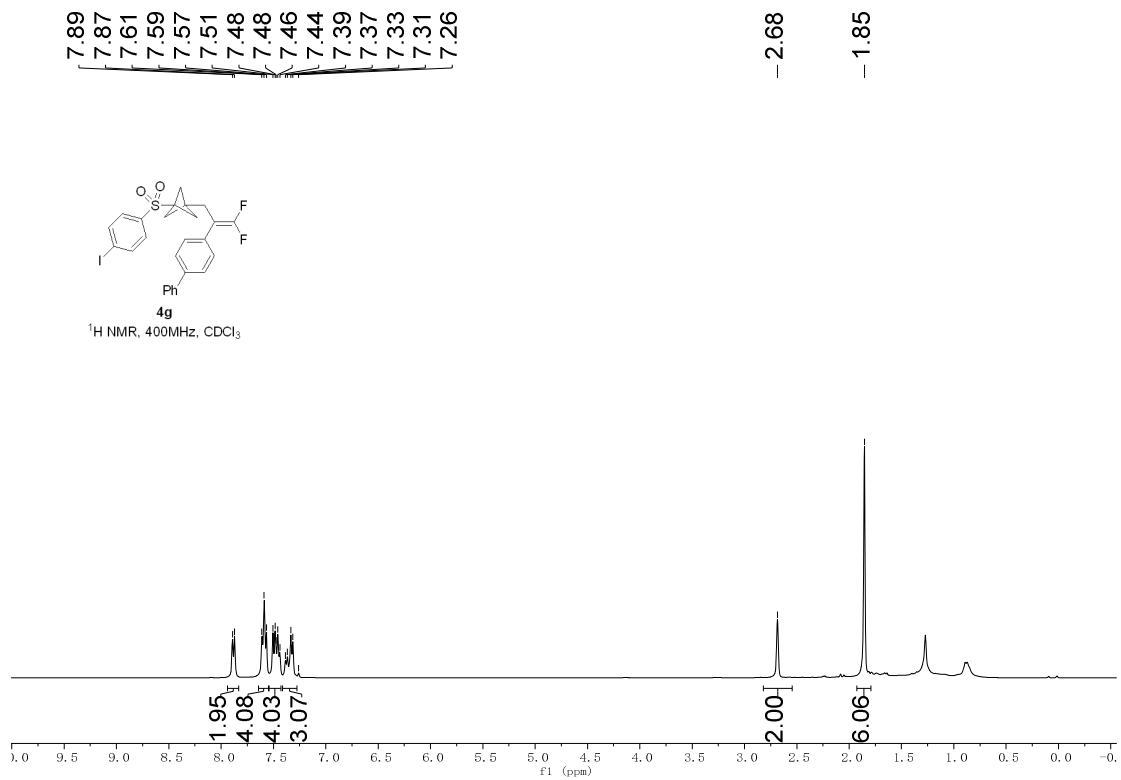


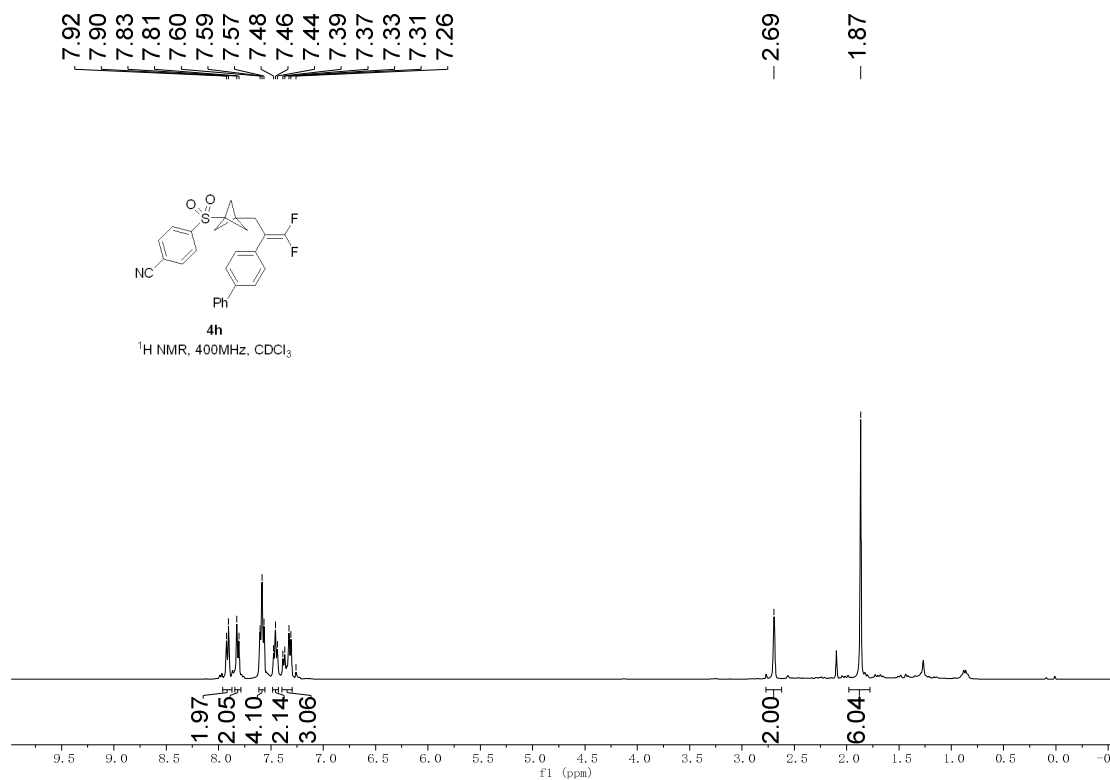
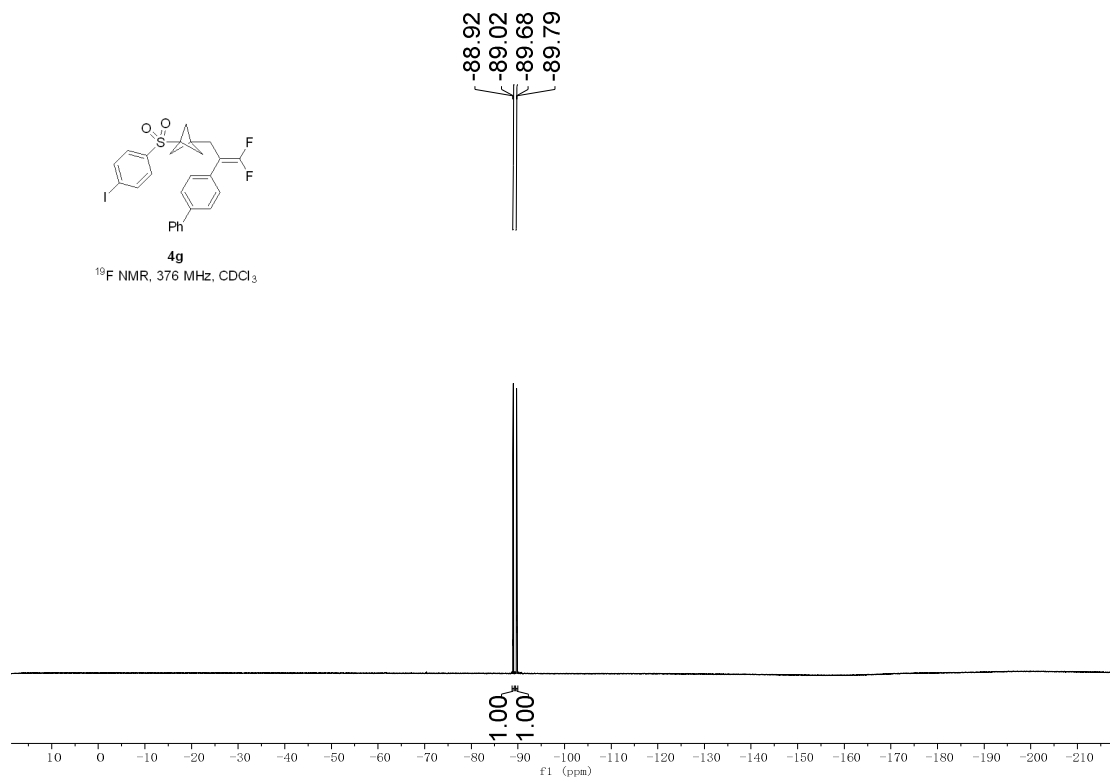
<sup>13</sup>C NMR, 101MHz, CDCl<sub>3</sub>



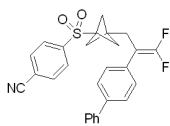
<sup>19</sup>F NMR, 376 MHz, CDCl<sub>3</sub>





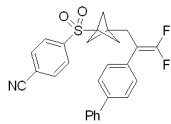
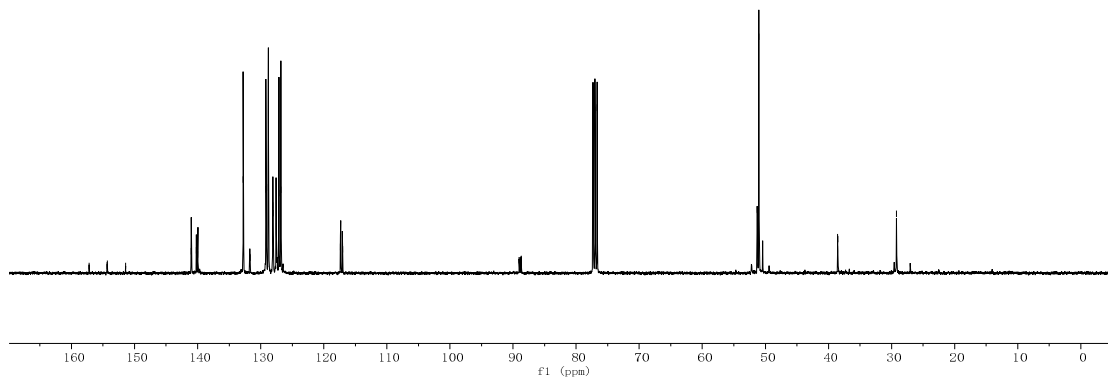


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 89.04  
 88.90  
 88.82  
 88.68  
 77.32  
 77.00  
 76.68  
 51.29  
 51.03  
 38.57  
 38.55  
 38.54  
 38.51  
 29.21



4h

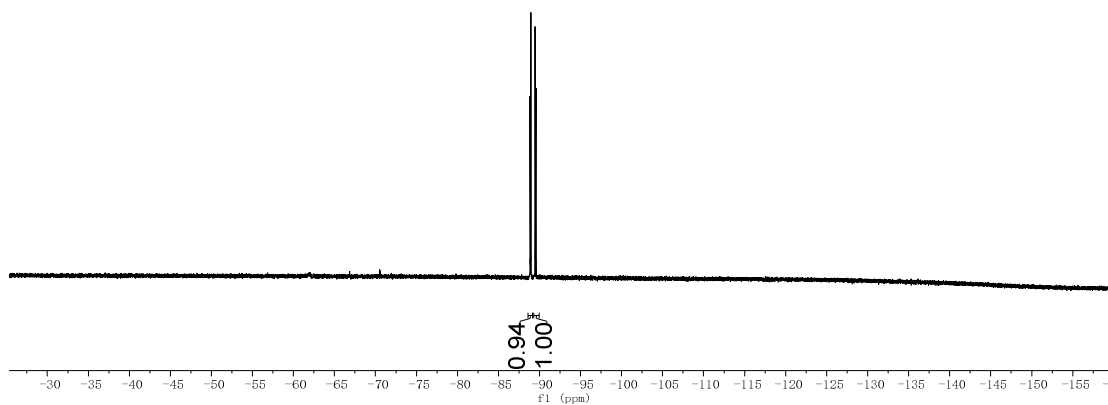
<sup>13</sup>C NMR, 100MHz, CDCl<sub>3</sub>

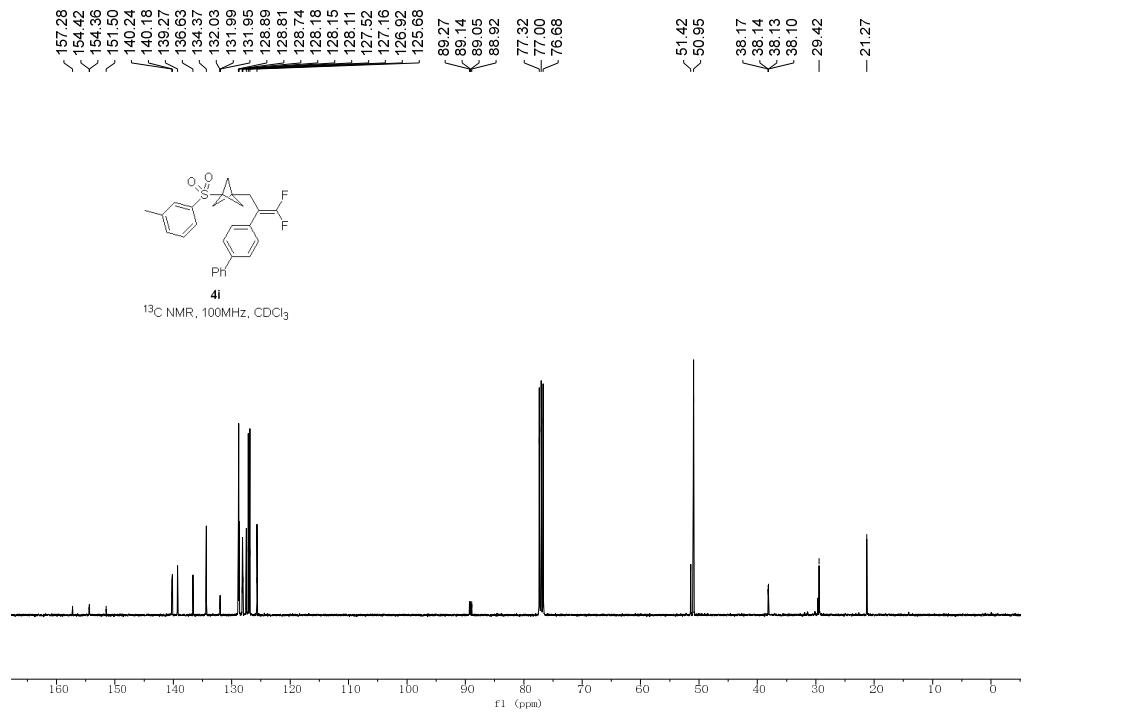
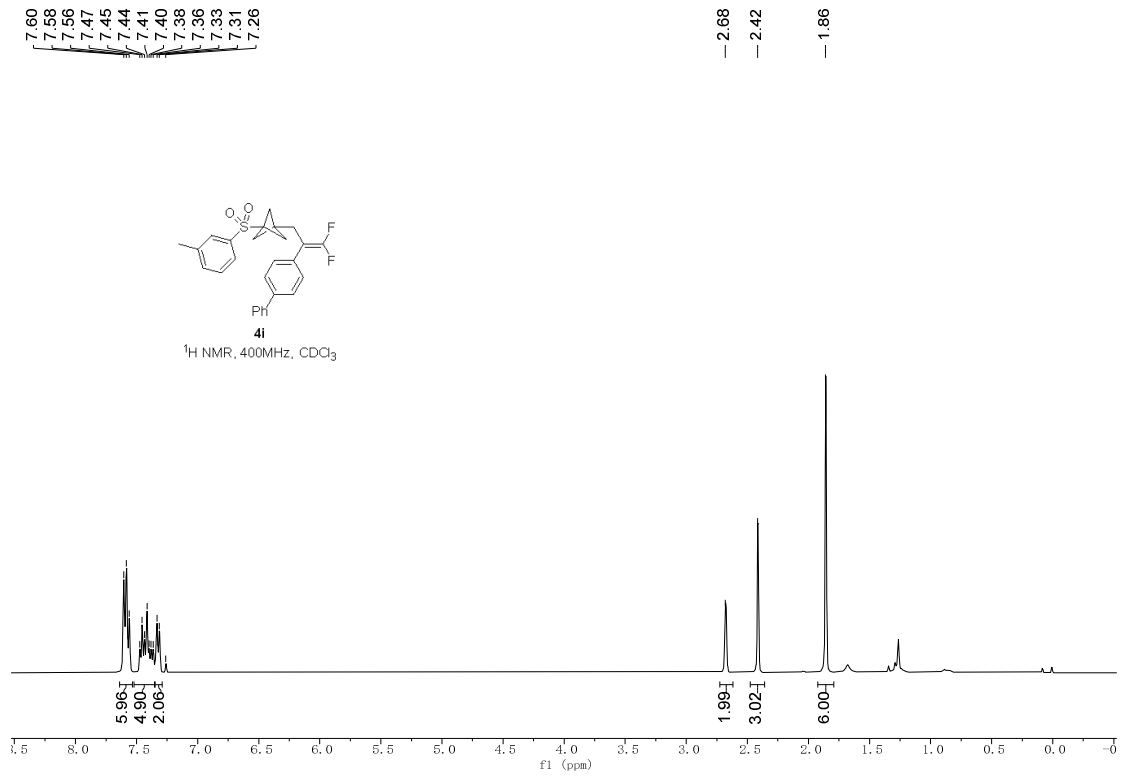


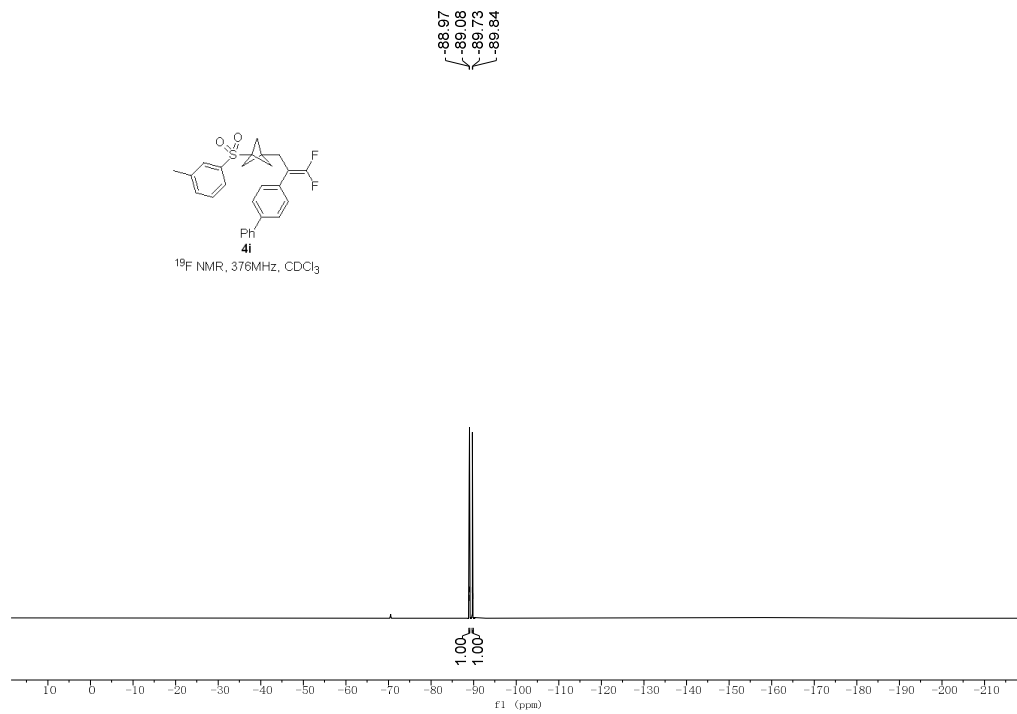
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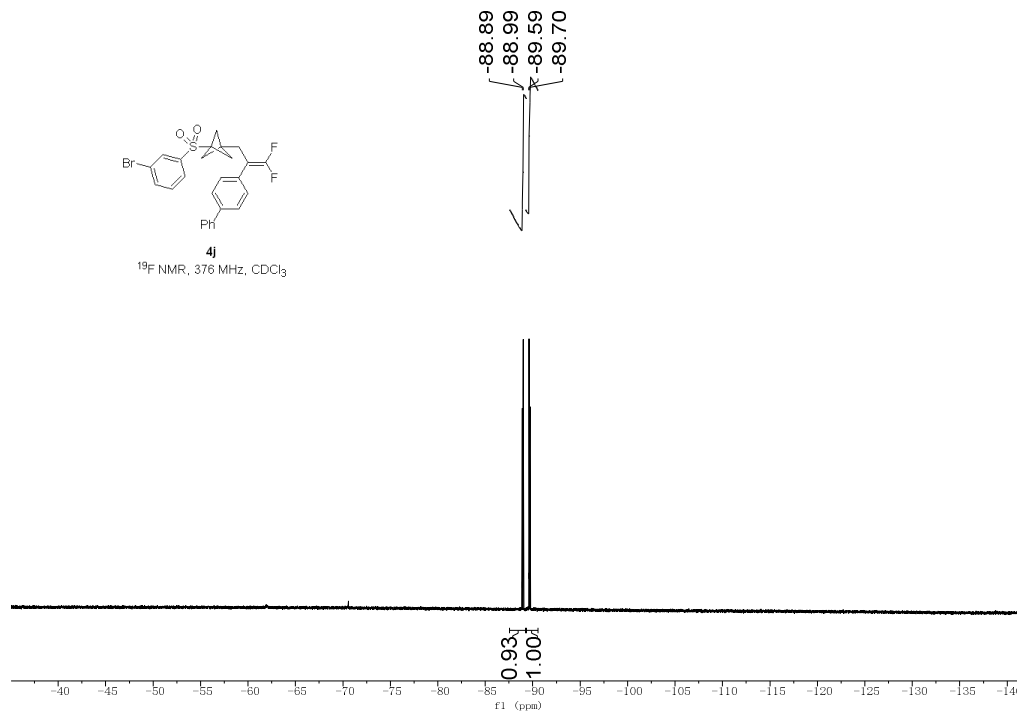
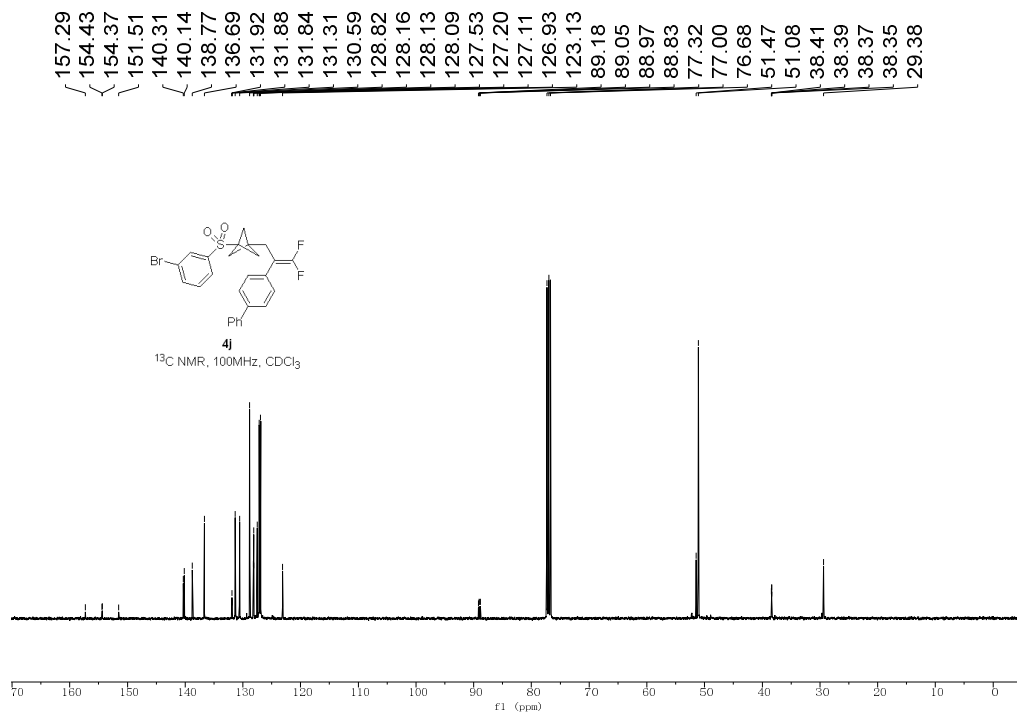
<sup>19</sup>F NMR, 376 MHz, CDCl<sub>3</sub>

-88.83  
 -88.93  
 -89.46  
 -89.56

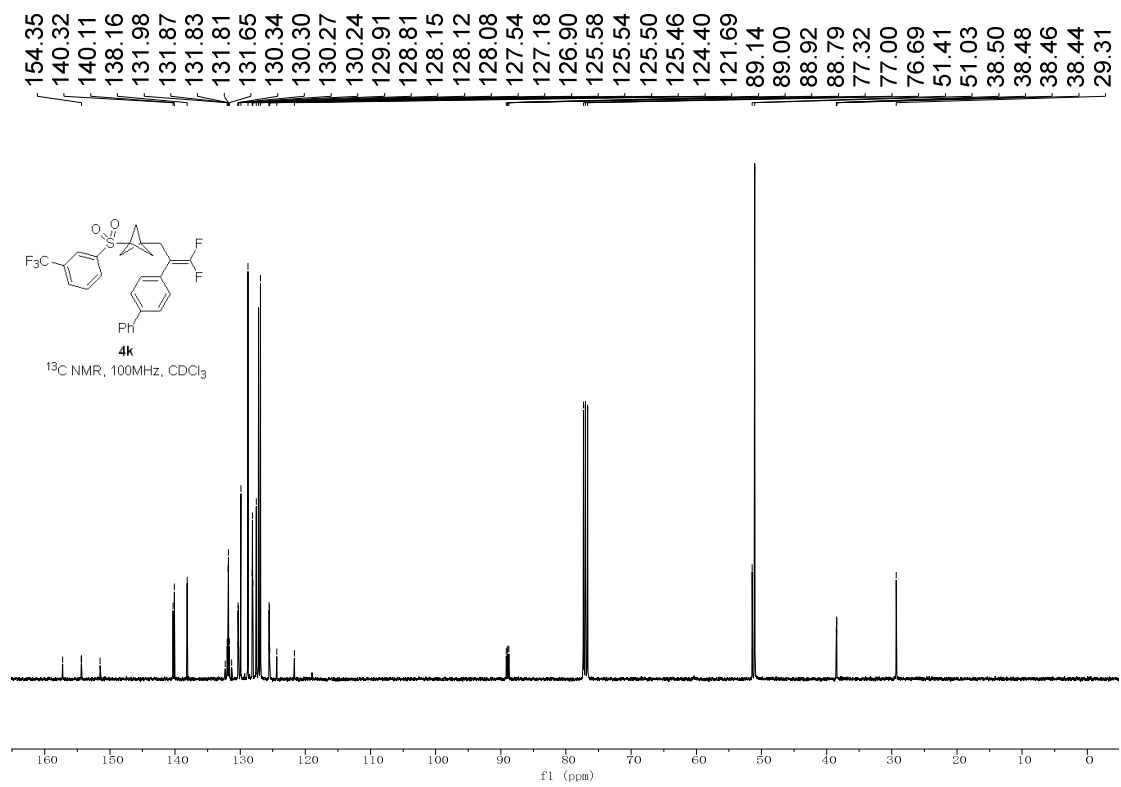
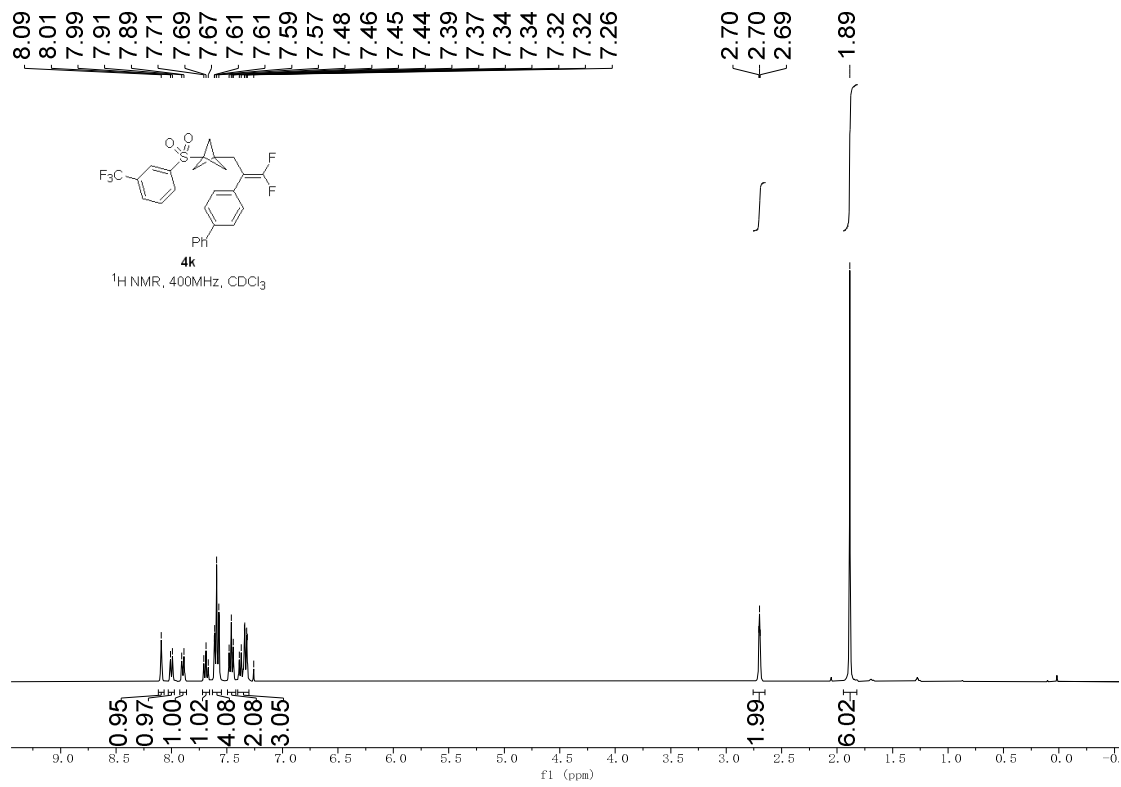


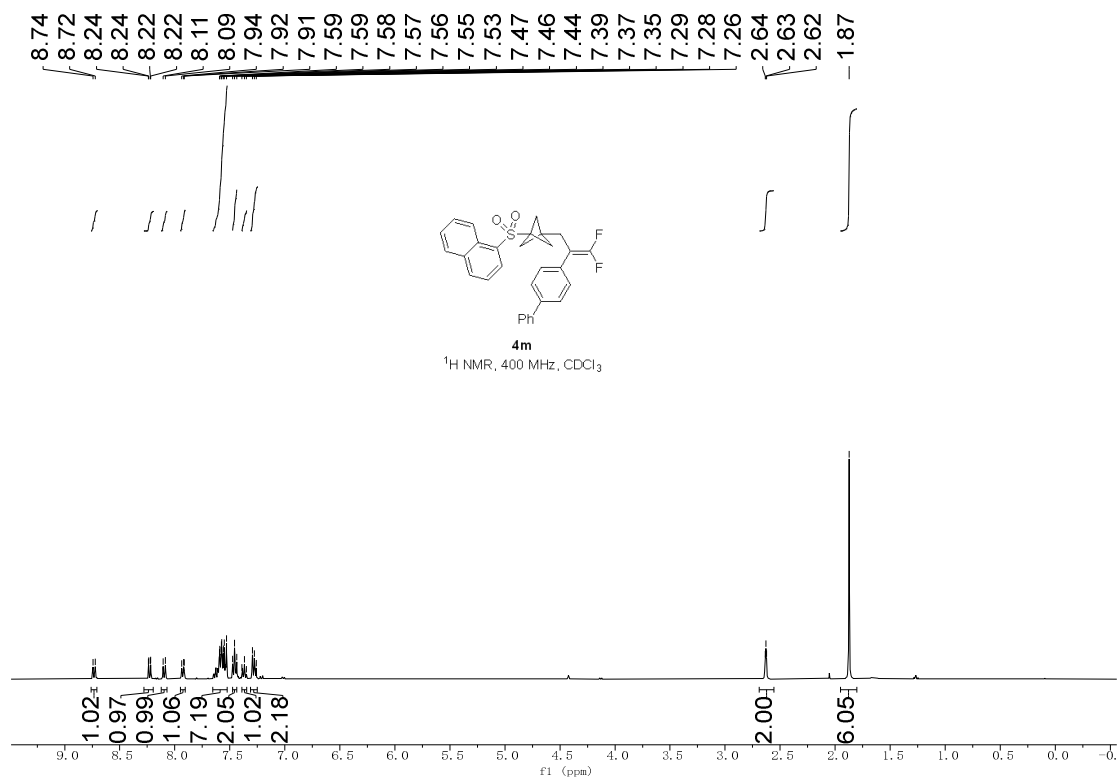
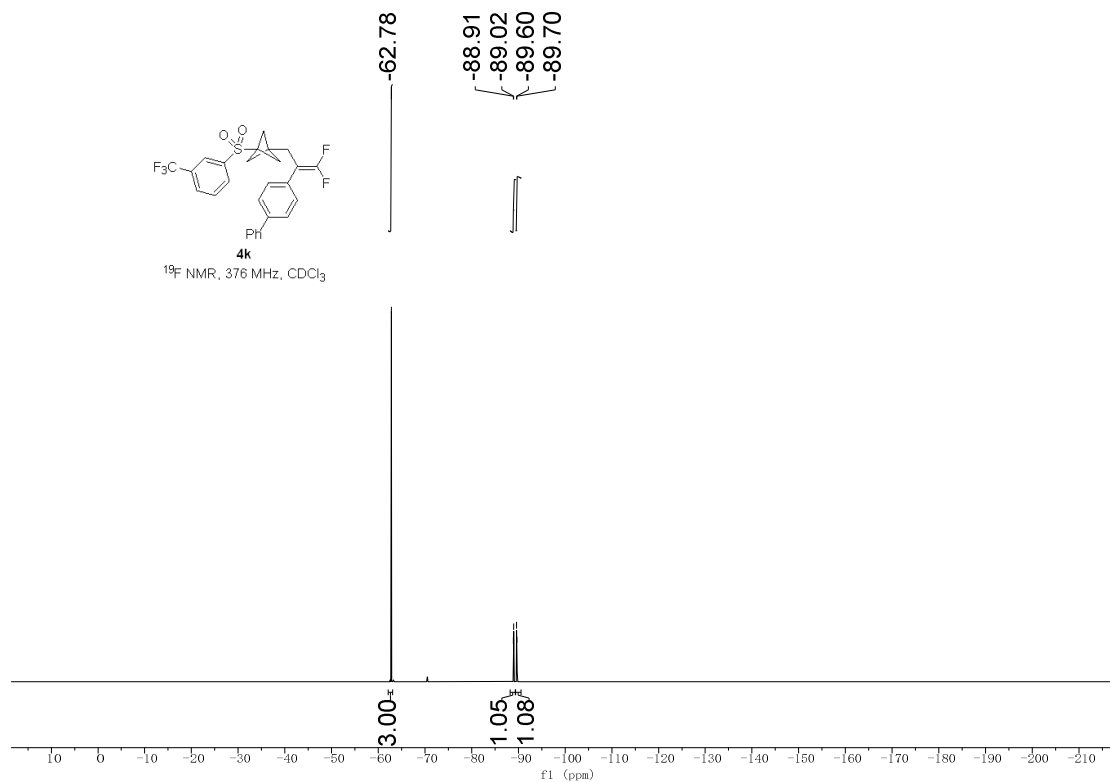




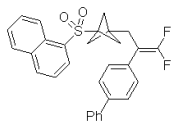






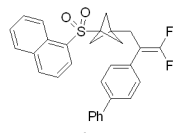
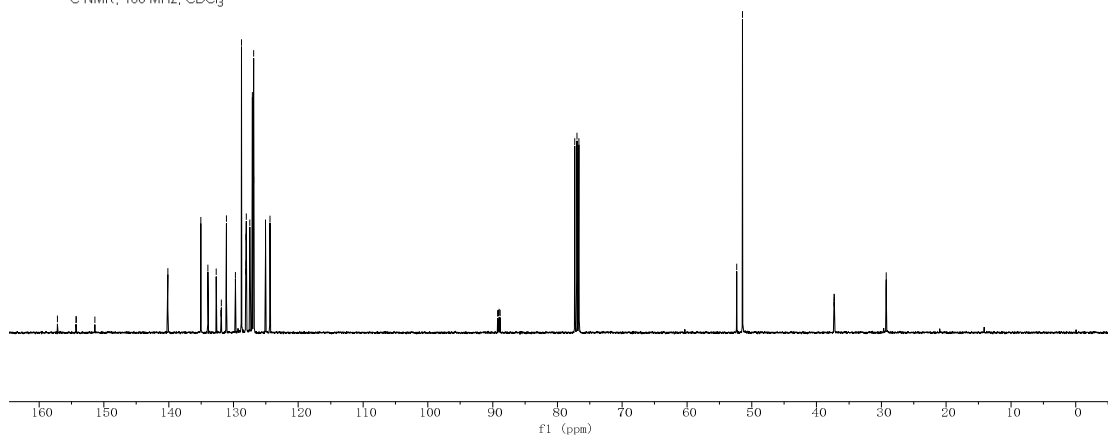


157.18  
 154.32  
 154.26  
 151.40  
 140.17  
 140.14  
 135.04  
 133.96  
 132.67  
 131.94  
 131.90  
 131.86  
 131.09  
 129.70  
 128.77  
 128.75  
 128.13  
 128.09  
 128.05  
 128.03  
 127.47  
 127.09  
 126.88  
 126.87  
 125.04  
 124.38  
 89.22  
 89.08  
 89.00  
 88.86  
 77.32  
 77.00  
 76.68  
 52.34  
 51.44  
 37.31  
 37.29  
 37.27  
 37.25  
 29.25



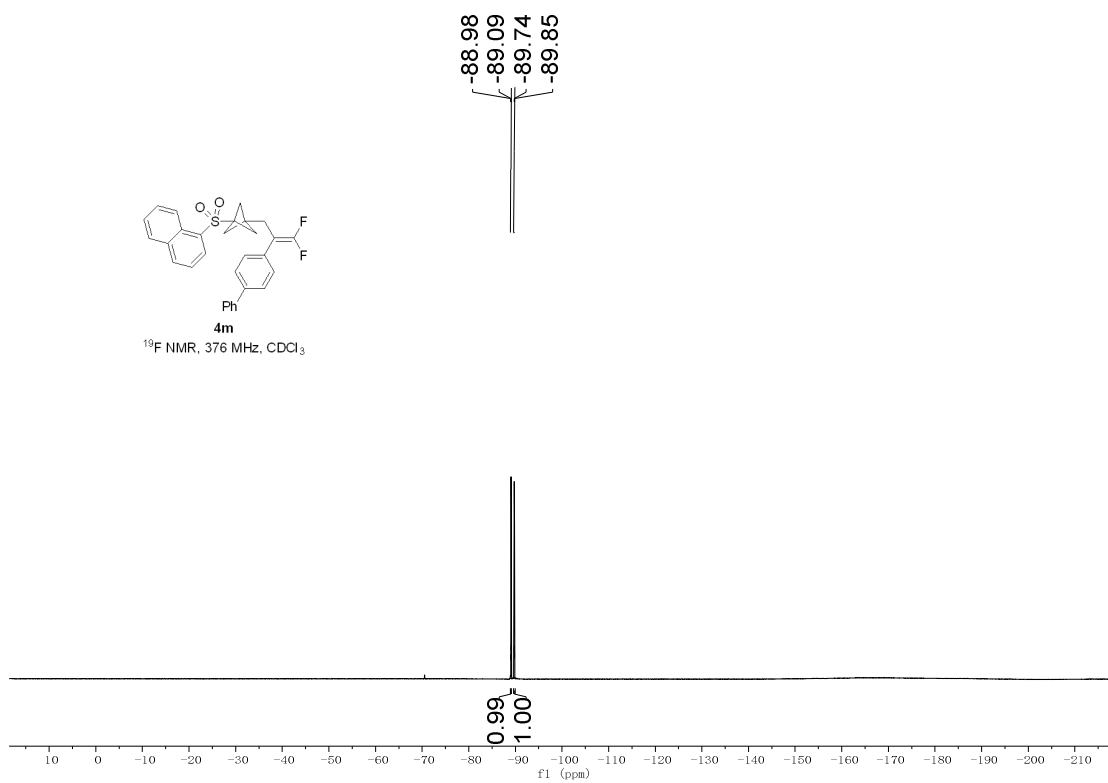
4m

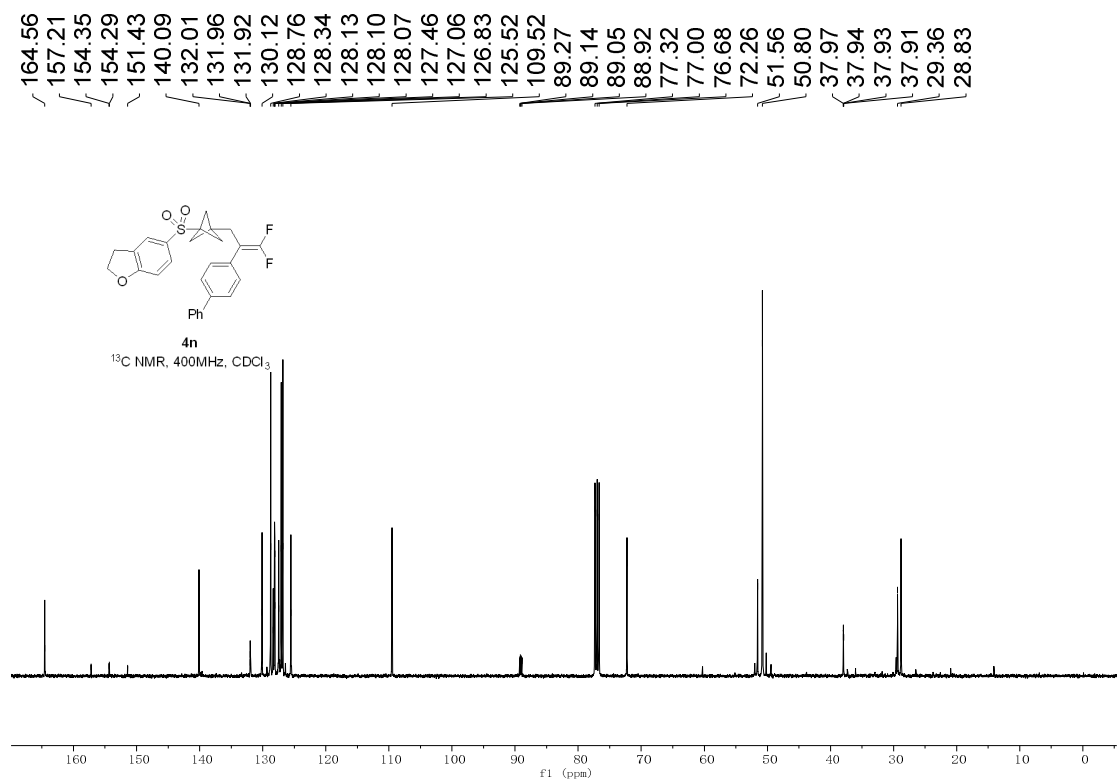
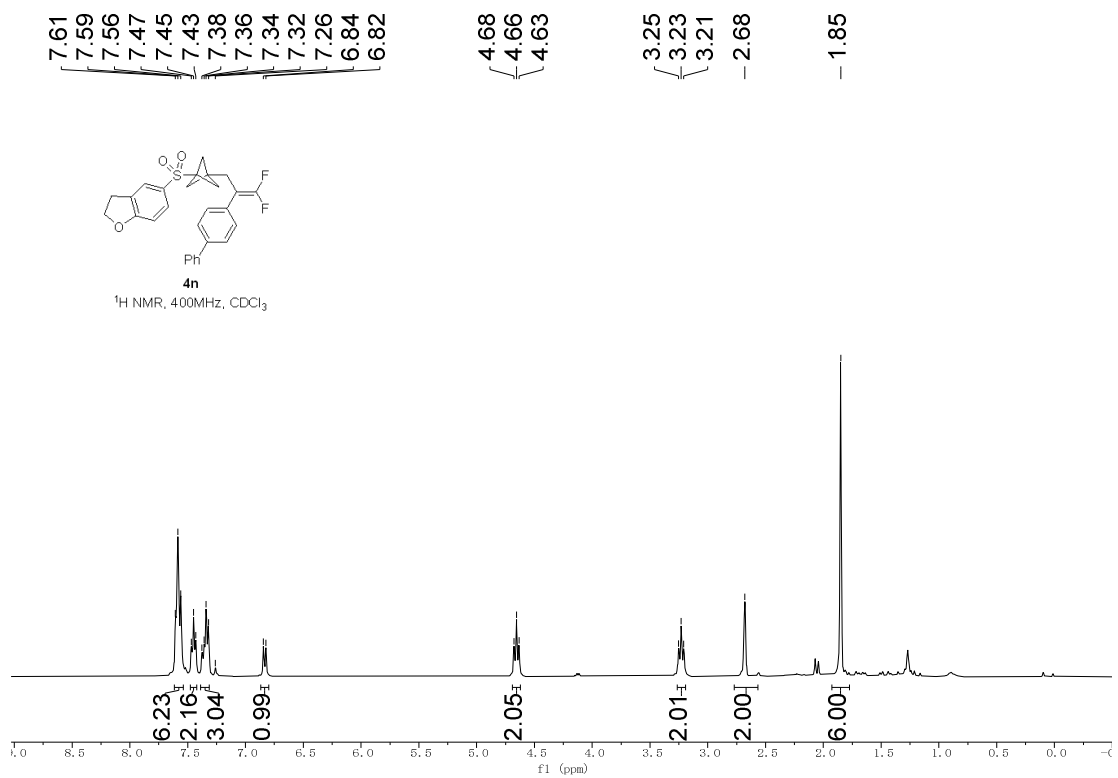
<sup>13</sup>C NMR, 100 MHz, CDCl<sub>3</sub>

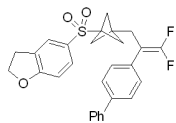


4m

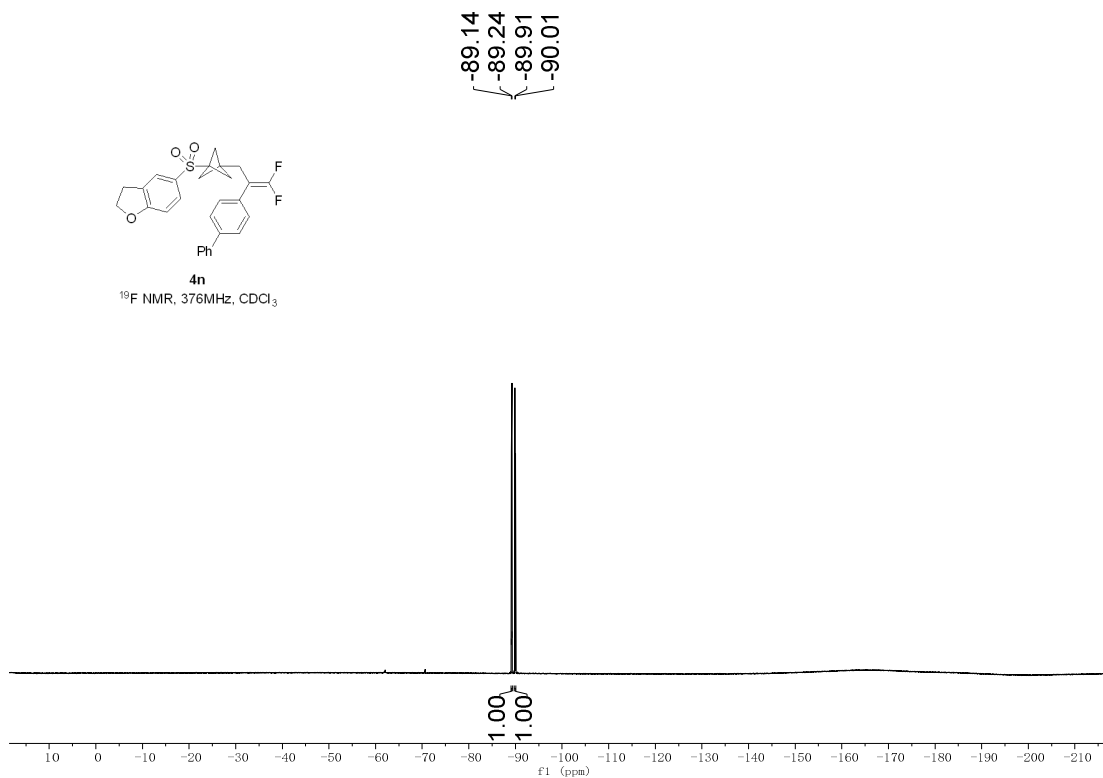
<sup>19</sup>F NMR, 376 MHz, CDCl<sub>3</sub>



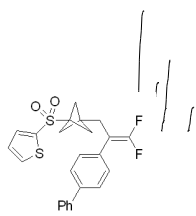




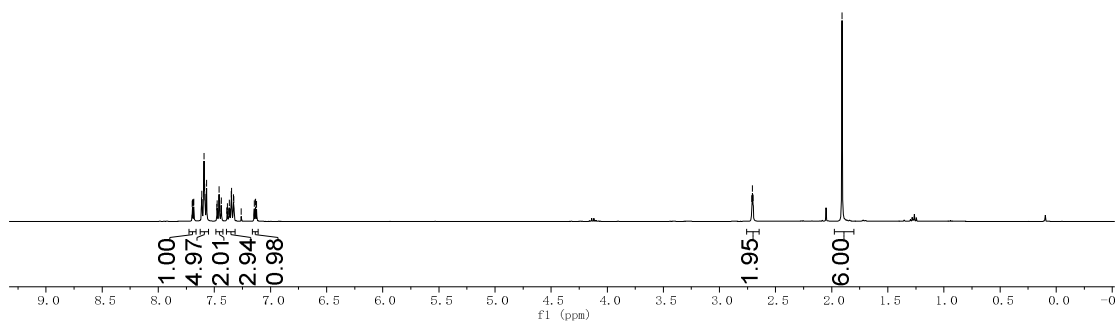
**4n**  
<sup>19</sup>F NMR, 376MHz, CDCl<sub>3</sub>



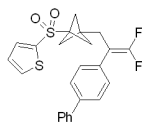
7.70  
7.69  
7.69  
7.68  
7.62  
7.61  
7.60  
7.59  
7.59  
7.58  
7.58  
7.57  
7.48  
7.48  
7.47  
7.46  
7.45  
7.44  
7.44  
7.39  
7.38  
7.38  
7.37  
7.37  
7.36  
7.36  
7.35  
7.35  
7.33  
7.33  
7.26  
7.15  
7.14  
7.13  
7.12  
2.71  
2.71  
2.70  
1.91



**4o**  
<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>

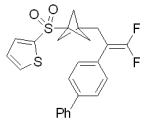
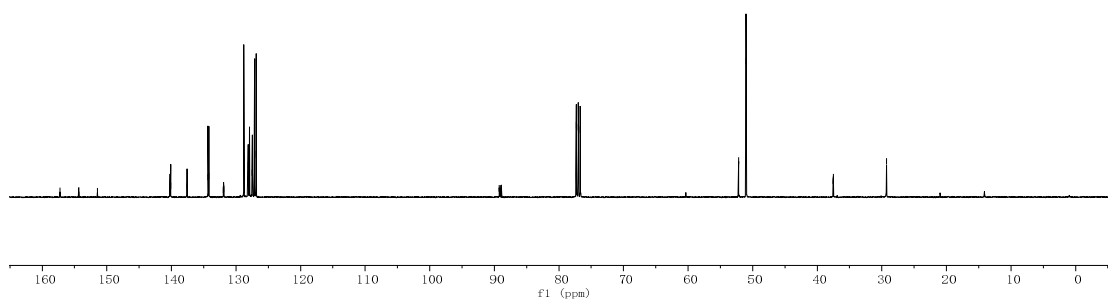


157.24  
 154.38  
 154.33  
 151.46  
 140.22  
 140.12  
 137.59  
 134.33  
 134.19  
 131.95  
 131.91  
 131.87  
 128.78  
 128.16  
 128.12  
 128.09  
 127.90  
 127.49  
 127.14  
 126.89  
 89.23  
 89.09  
 89.01  
 88.88  
 77.32  
 77.00  
 76.68  
 52.18  
 51.01  
 37.54  
 37.52  
 37.50  
 37.48  
 29.28



4o

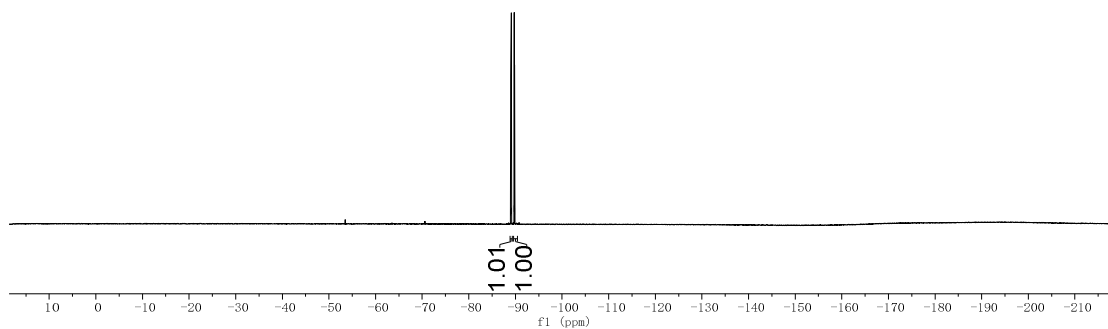
<sup>13</sup>C NMR, 100 MHz, CDCl<sub>3</sub>



4o

<sup>19</sup>F NMR, 376 MHz, CDCl<sub>3</sub>

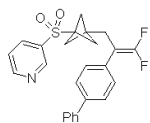
-89.01  
 -89.11  
 -89.73  
 -89.83



9.00  
8.85  
8.84  
8.09  
8.07  
8.07  
7.60  
7.58  
7.56  
7.49  
7.48  
7.47  
7.46  
7.45  
7.43  
7.37  
7.36  
7.32  
7.30  
7.26

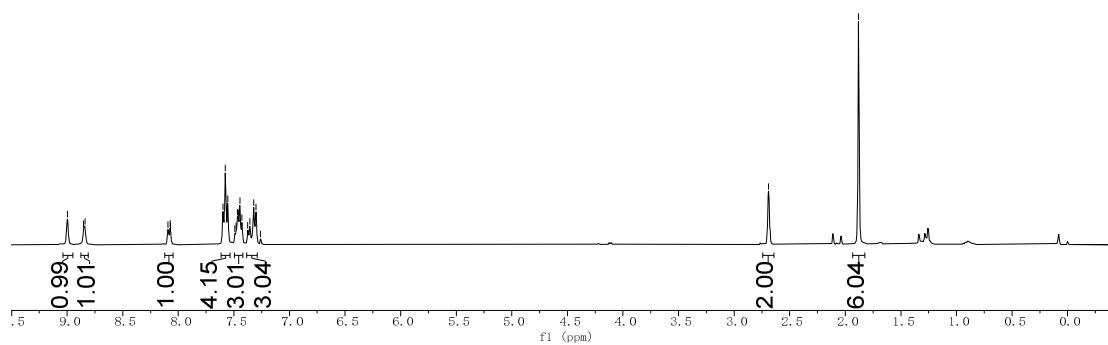
-2.69

-1.88

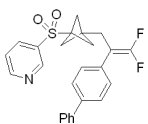


4p

<sup>1</sup>H NMR, 400MHz, CDCl<sub>3</sub>

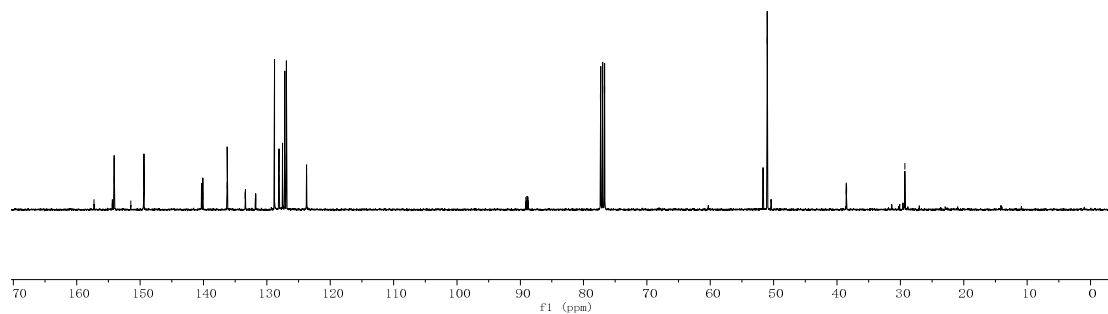


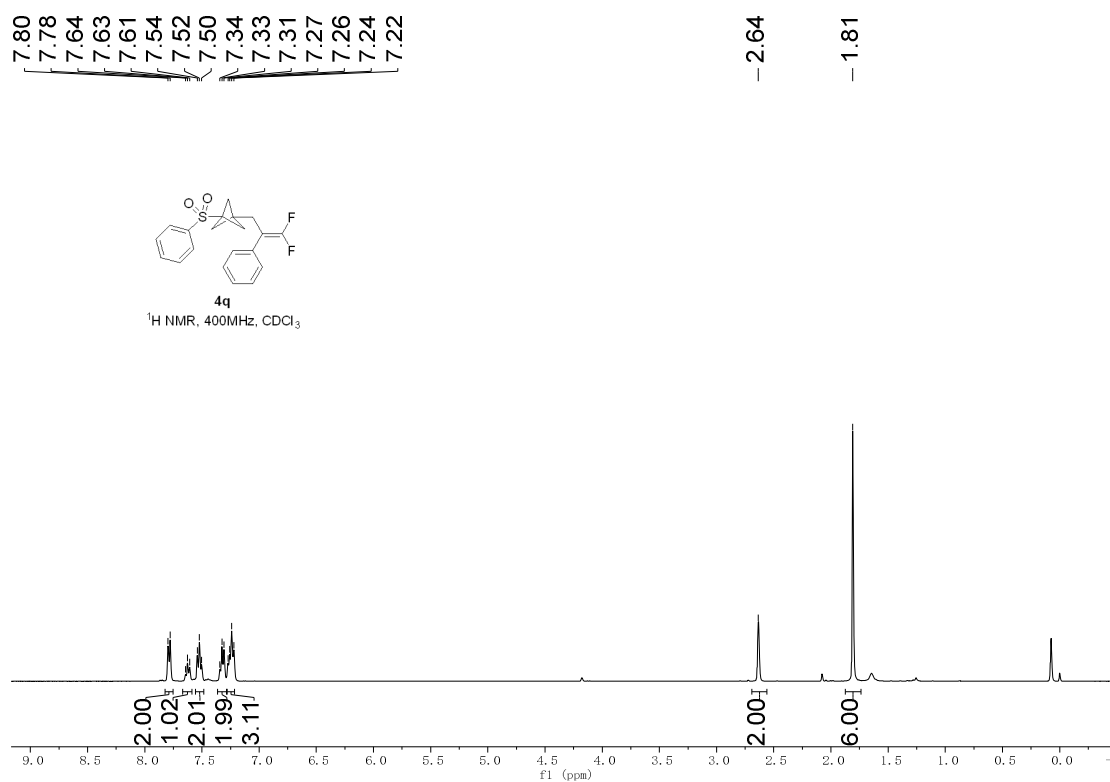
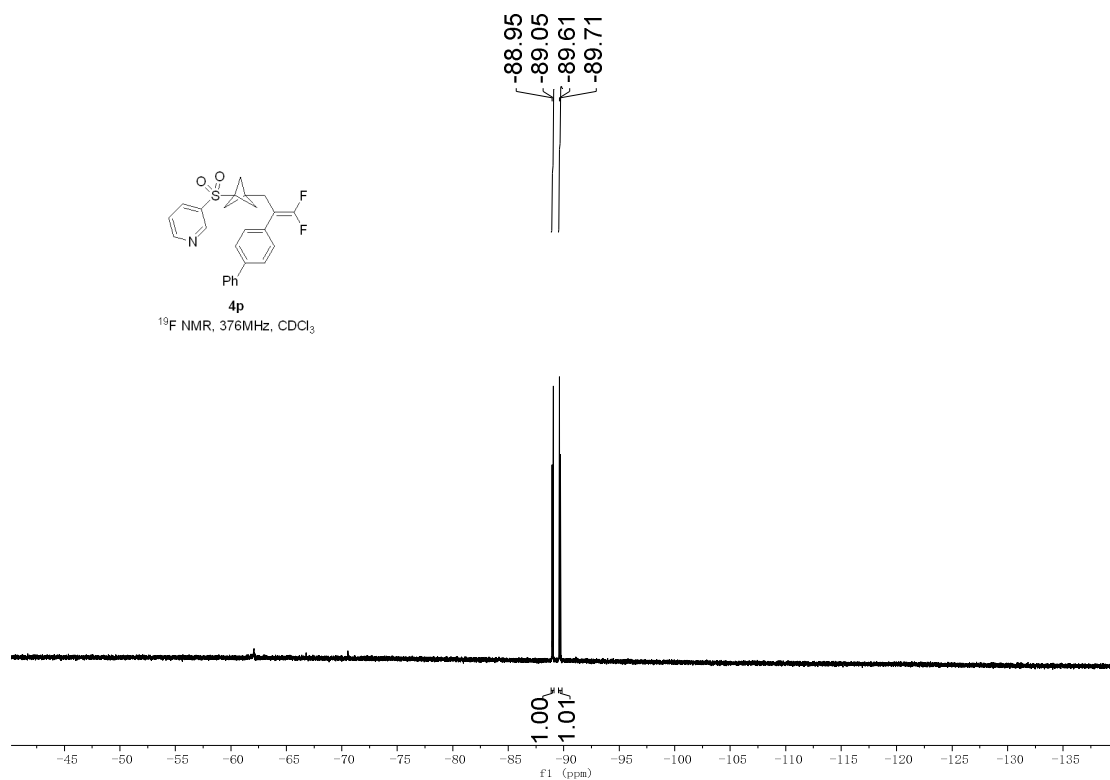
157.26  
154.39  
154.34  
154.09  
151.47  
149.40  
140.30  
140.08  
136.25  
133.40  
131.81  
131.77  
131.73  
128.79  
128.12  
128.09  
128.05  
127.52  
127.18  
126.89  
123.72  
89.10  
88.96  
88.88  
88.75  
77.32  
77.00  
76.68  
51.69  
51.00  
38.56  
38.53  
38.52  
38.50  
29.28



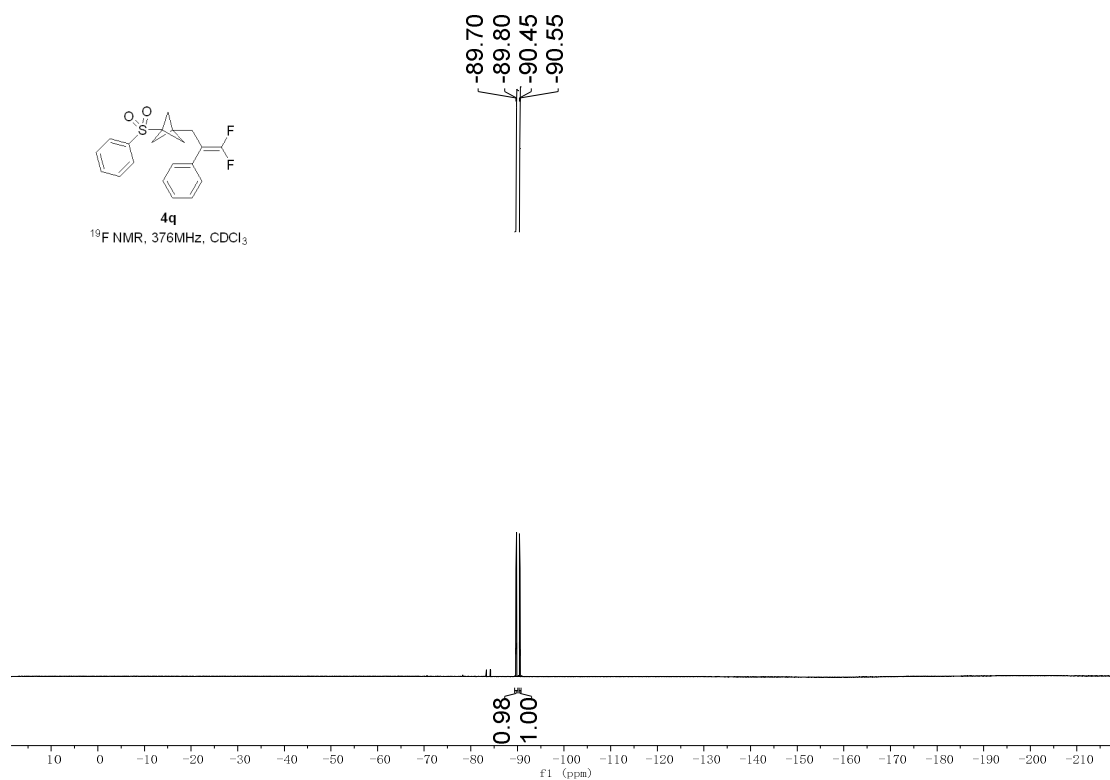
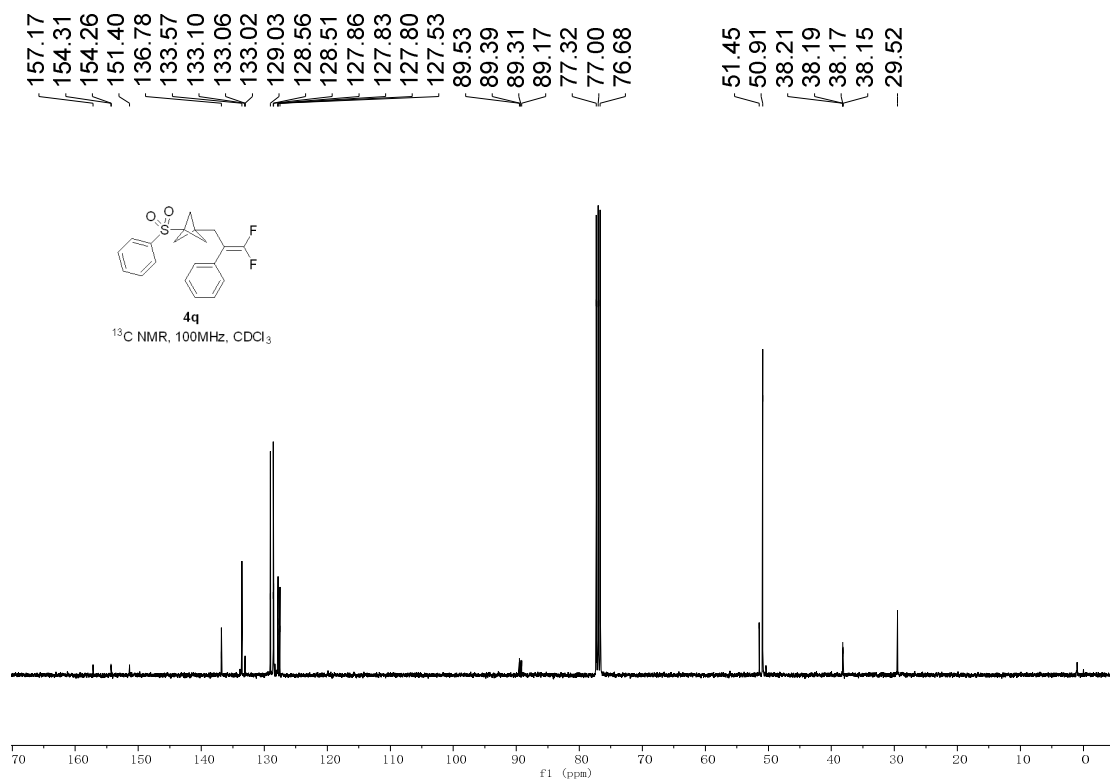
4p

<sup>13</sup>C NMR, 100MHz, CDCl<sub>3</sub>



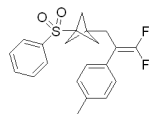






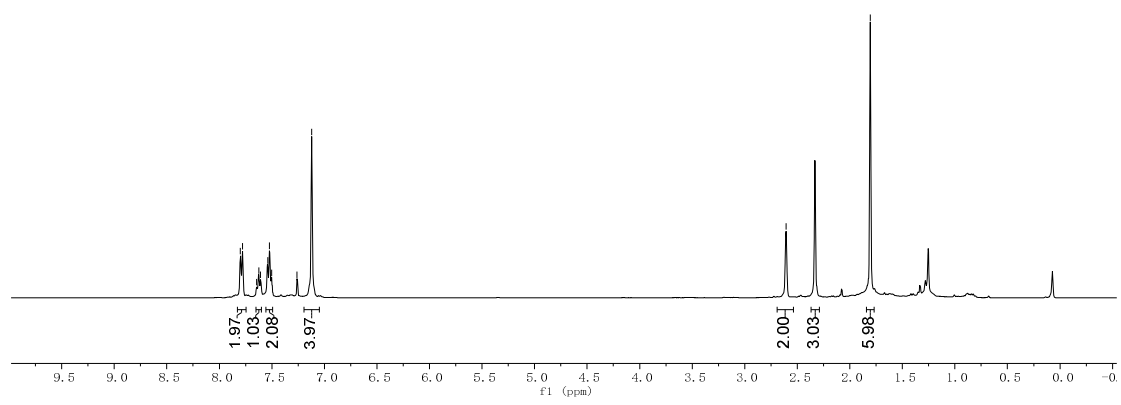
7.80  
7.78  
7.64  
7.62  
7.61  
7.54  
7.52  
7.50  
7.26  
7.12

-2.61  
-2.34  
-1.81



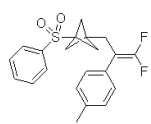
4r

<sup>1</sup>H NMR, 400MHz, CDCl<sub>3</sub>



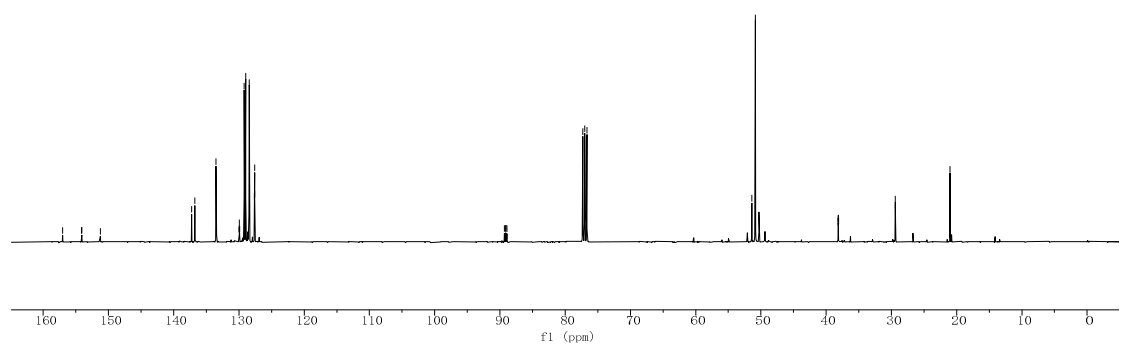
157.01  
154.15  
154.10  
137.27  
136.75  
133.51  
129.97  
129.93  
129.89  
129.20  
128.97  
128.44  
127.65  
127.61  
127.58  
89.30  
89.16  
89.08  
88.94  
77.32  
77.00  
76.68

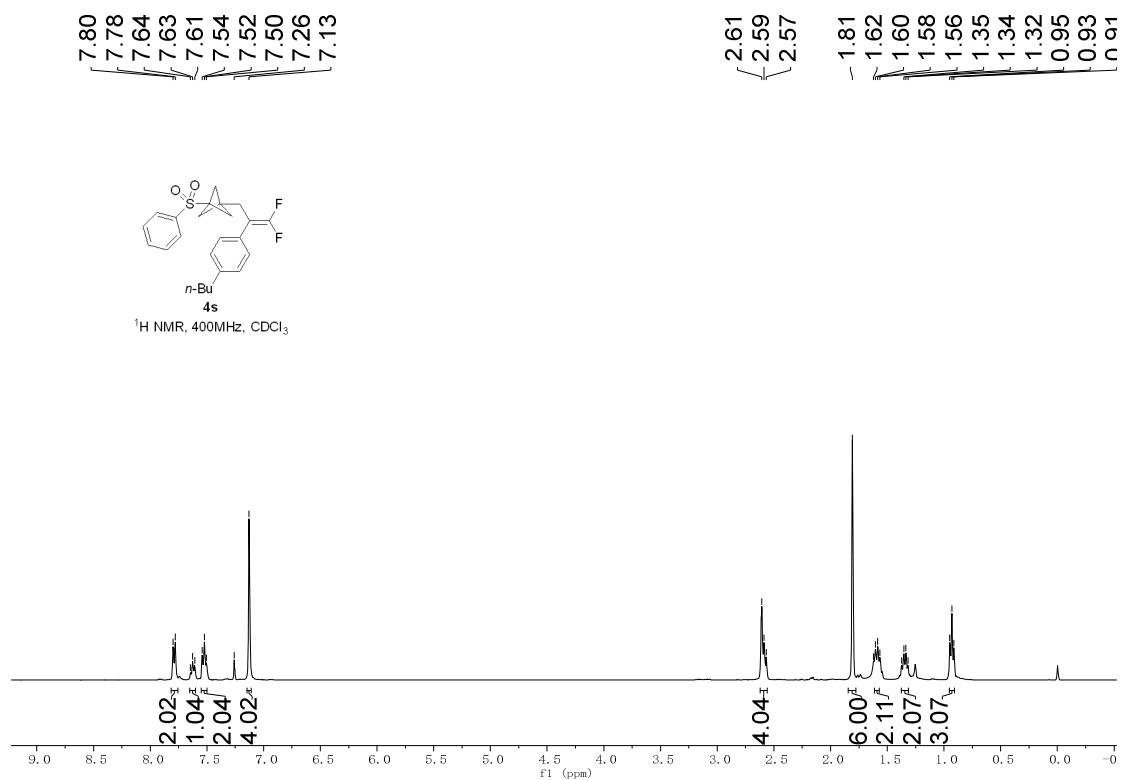
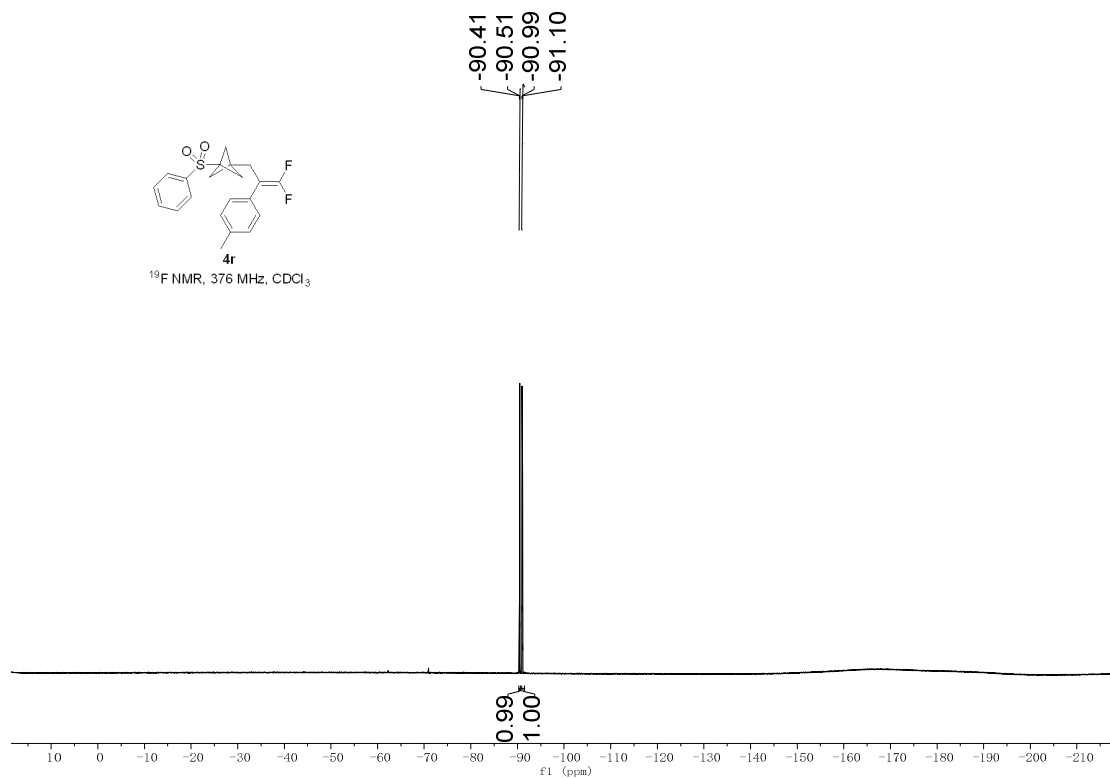
51.39  
50.86  
38.17  
38.15  
38.13  
38.11  
29.40  
21.02

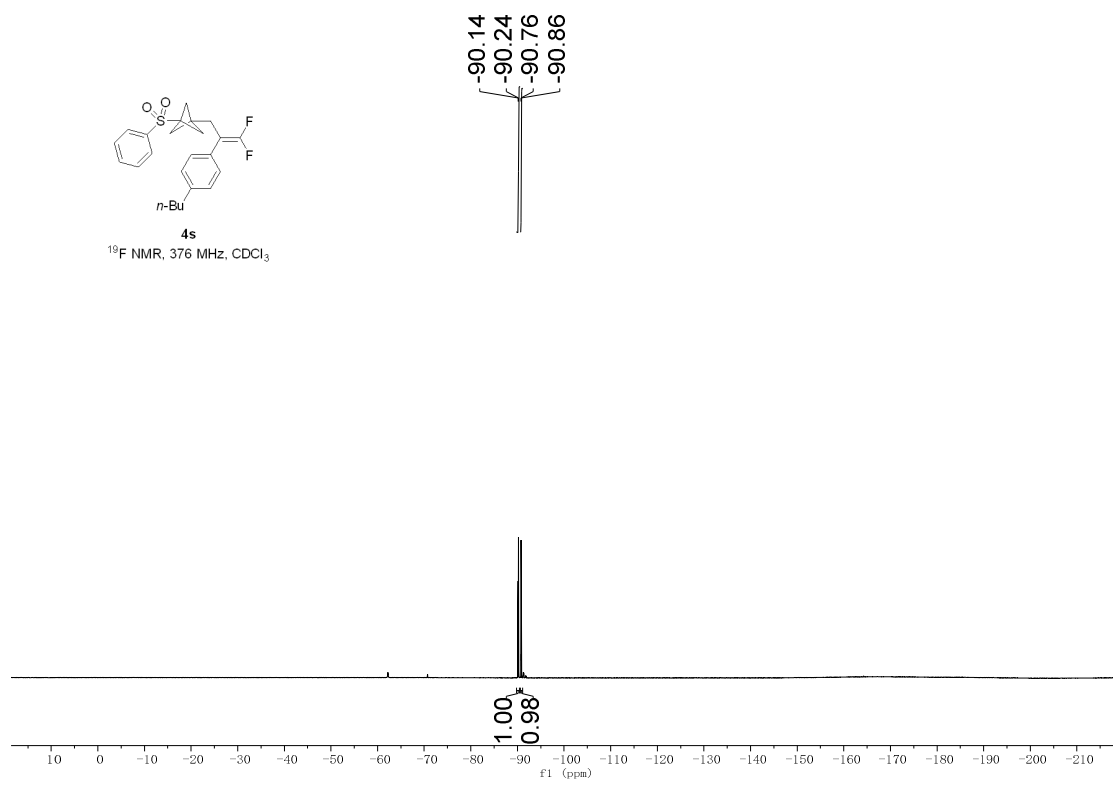
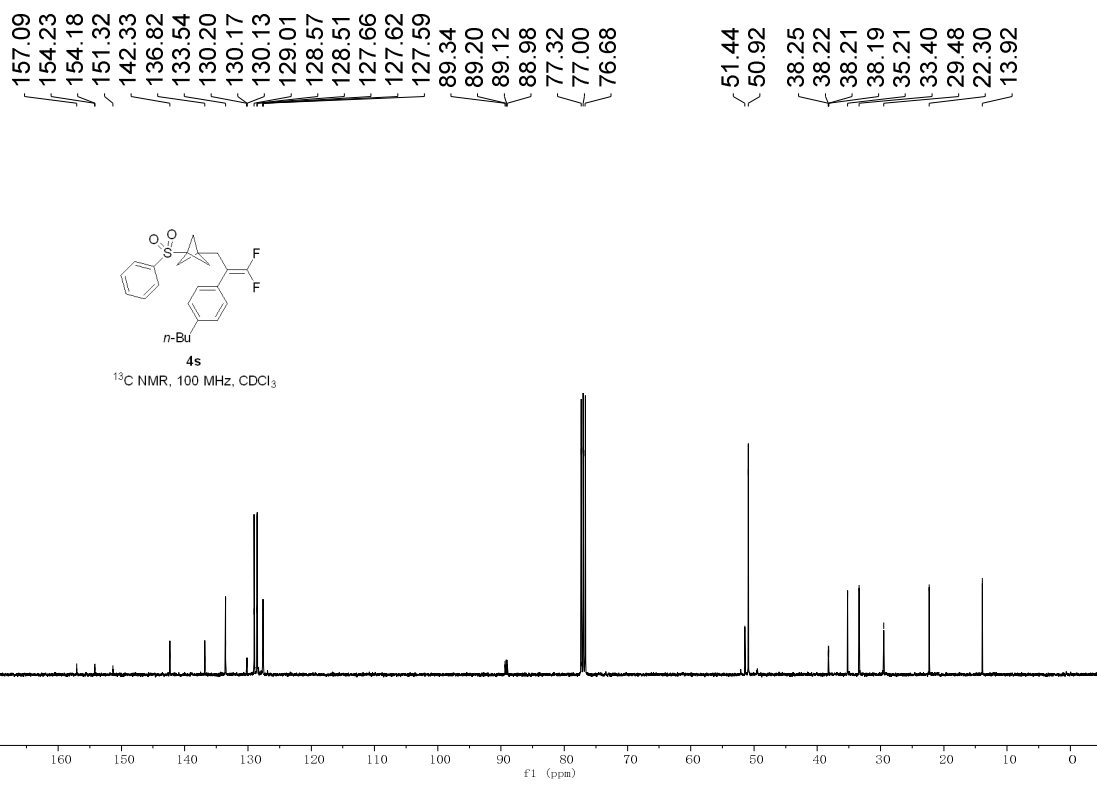


4r

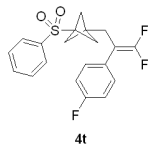
<sup>1</sup>H NMR, 100 MHz, CDCl<sub>3</sub>



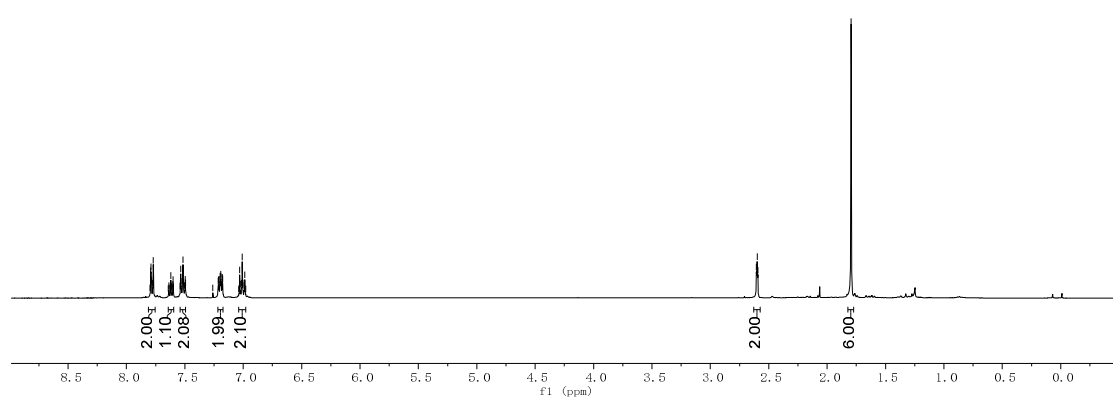




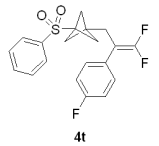
7.80  
7.79  
7.79  
7.78  
7.77  
7.77  
7.64  
7.64  
7.63  
7.62  
7.62  
7.61  
7.60  
7.60  
7.60  
7.54  
7.54  
7.53  
7.52  
7.52  
7.51  
7.50  
7.50  
7.49  
7.26  
7.21  
7.21  
7.20  
7.20  
7.19  
7.19  
7.18  
7.18  
7.18  
7.04  
7.03  
7.02  
7.01  
7.01  
7.00  
6.99  
6.99  
6.98  
2.61  
2.60  
2.59  
1.79



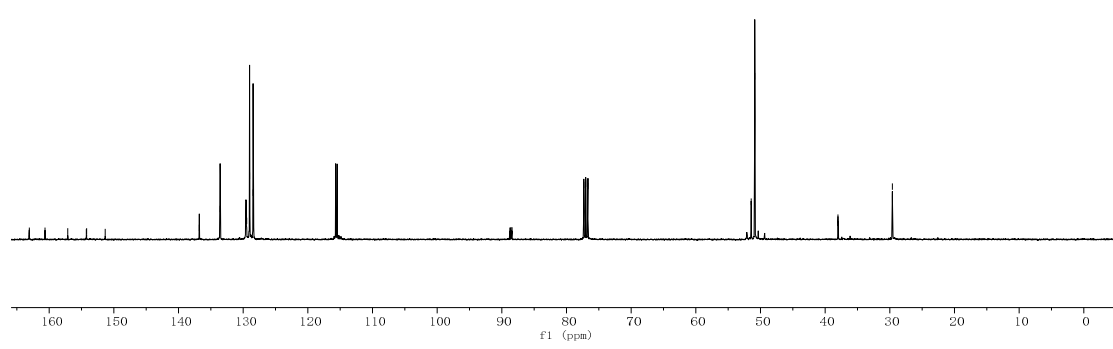
<sup>1</sup>H NMR, 400MHz, CDCl<sub>3</sub>

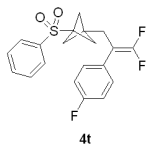


163.08  
160.62  
157.12  
154.27  
154.22  
151.35  
136.78  
133.56  
129.62  
129.58  
129.54  
129.50  
129.47  
128.92  
128.46  
115.67  
115.45  
88.78  
88.64  
88.56  
88.42  
77.32  
77.00  
76.68  
51.45  
50.88  
38.05  
38.03  
38.01  
37.99  
29.60

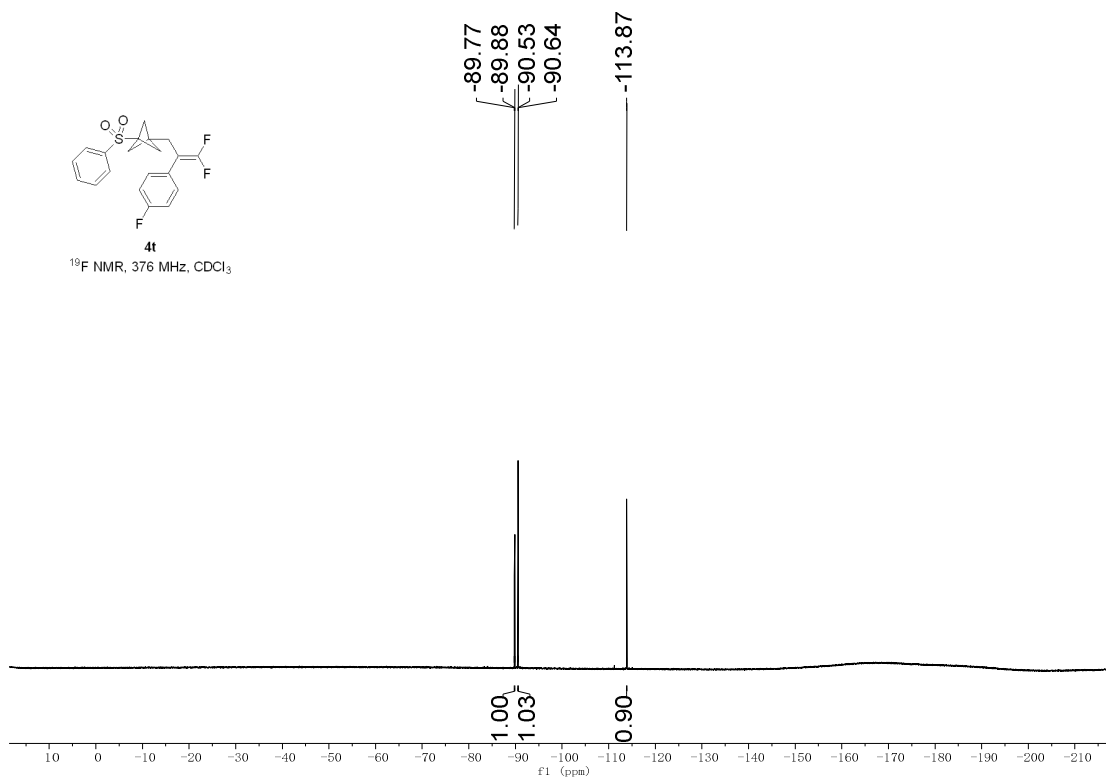


<sup>13</sup>C NMR, 100MHz, CDCl<sub>3</sub>





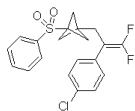
<sup>19</sup>F NMR, 376 MHz, CDCl<sub>3</sub>



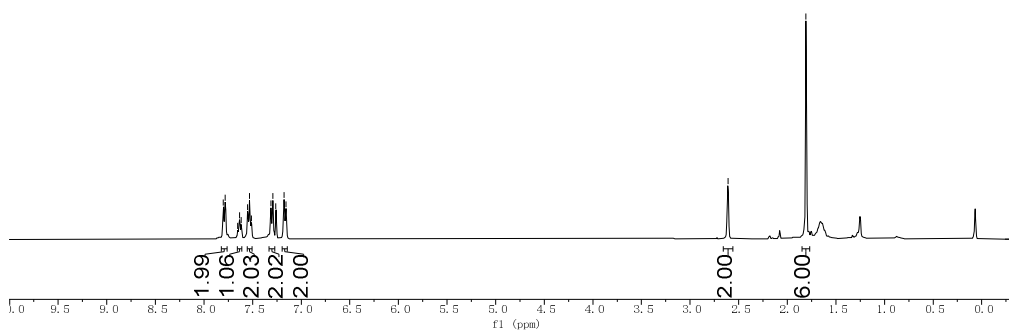
7.80  
7.78  
7.65  
7.63  
7.62  
7.55  
7.53  
7.51  
7.31  
7.29  
7.26  
7.18  
7.16

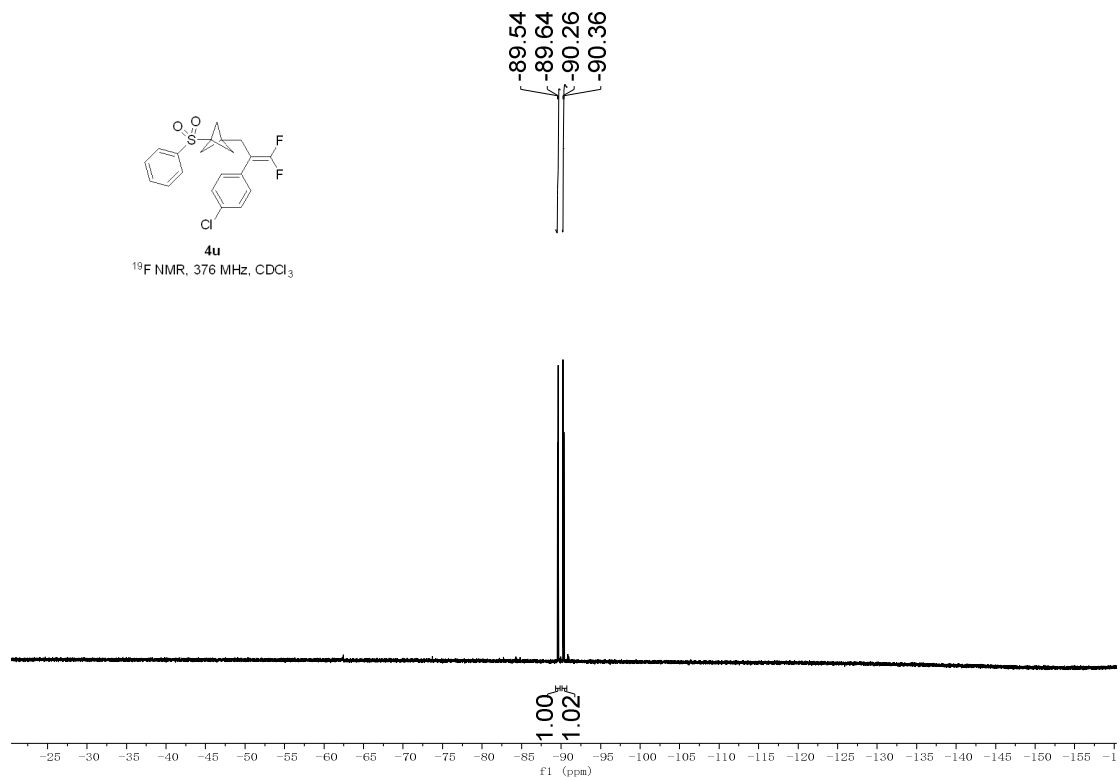
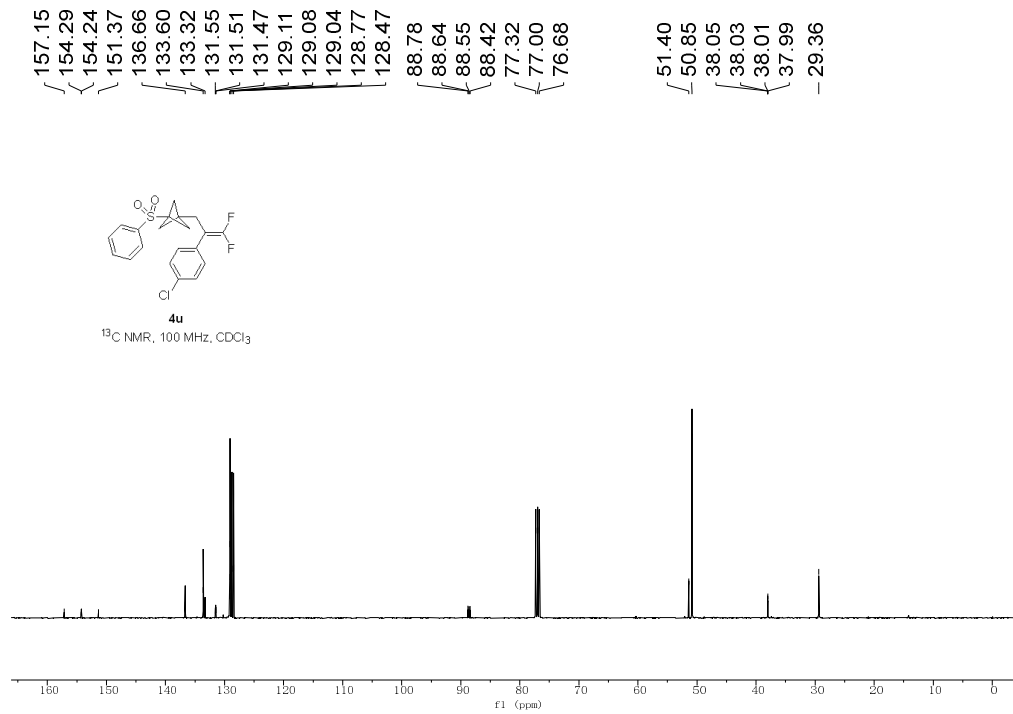
- 2.61

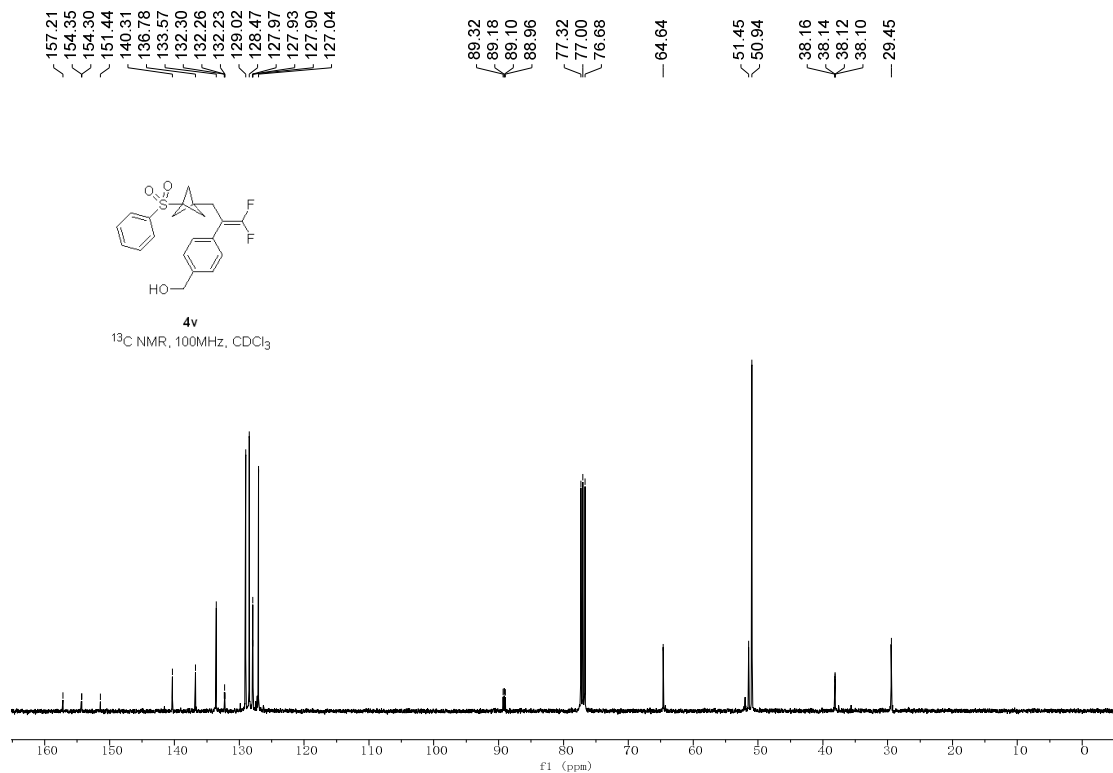
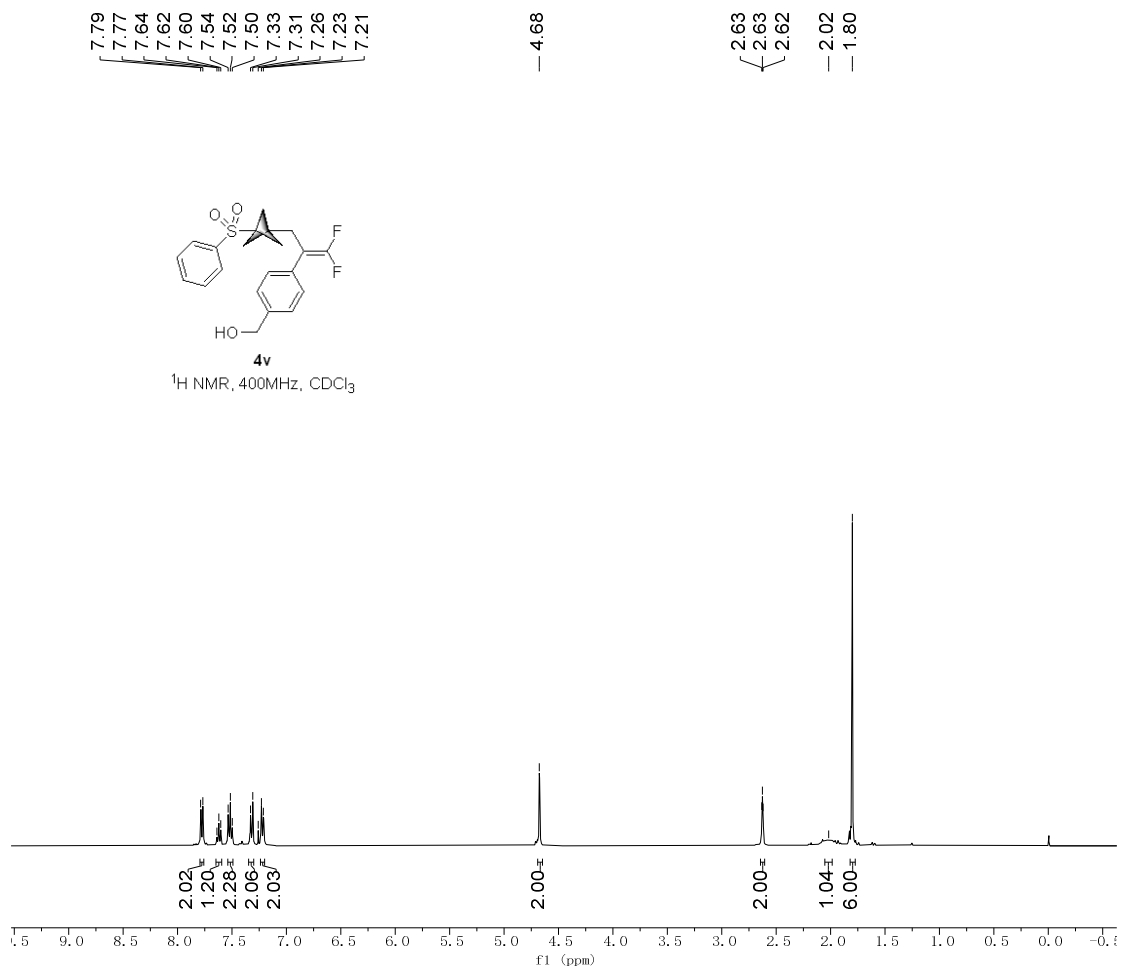
- 1.81



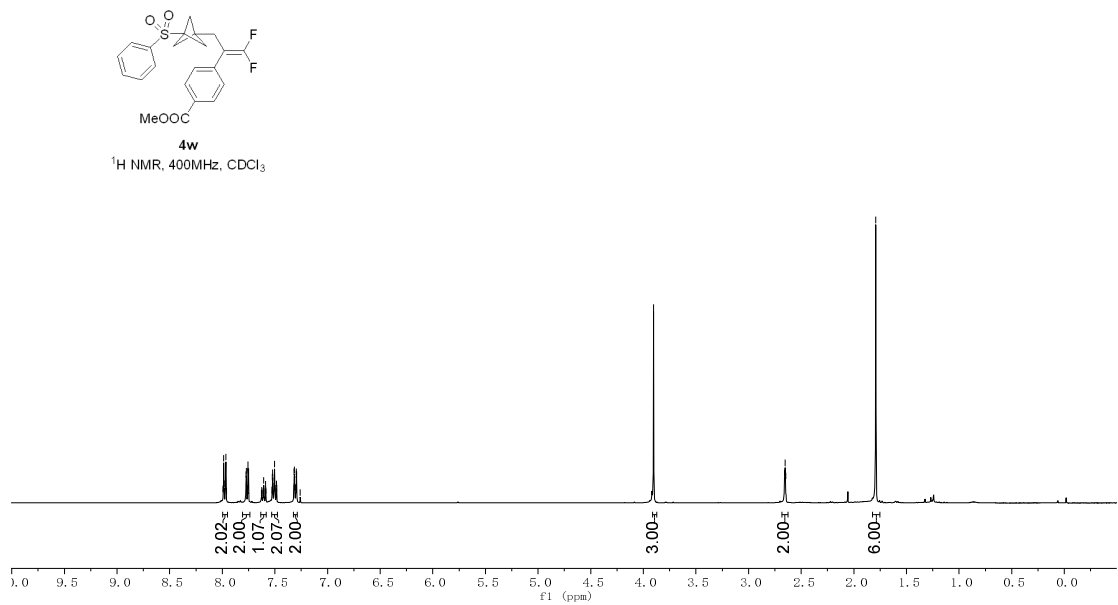
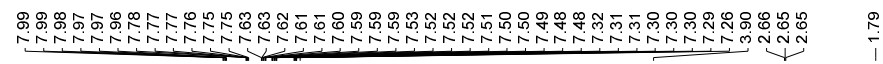
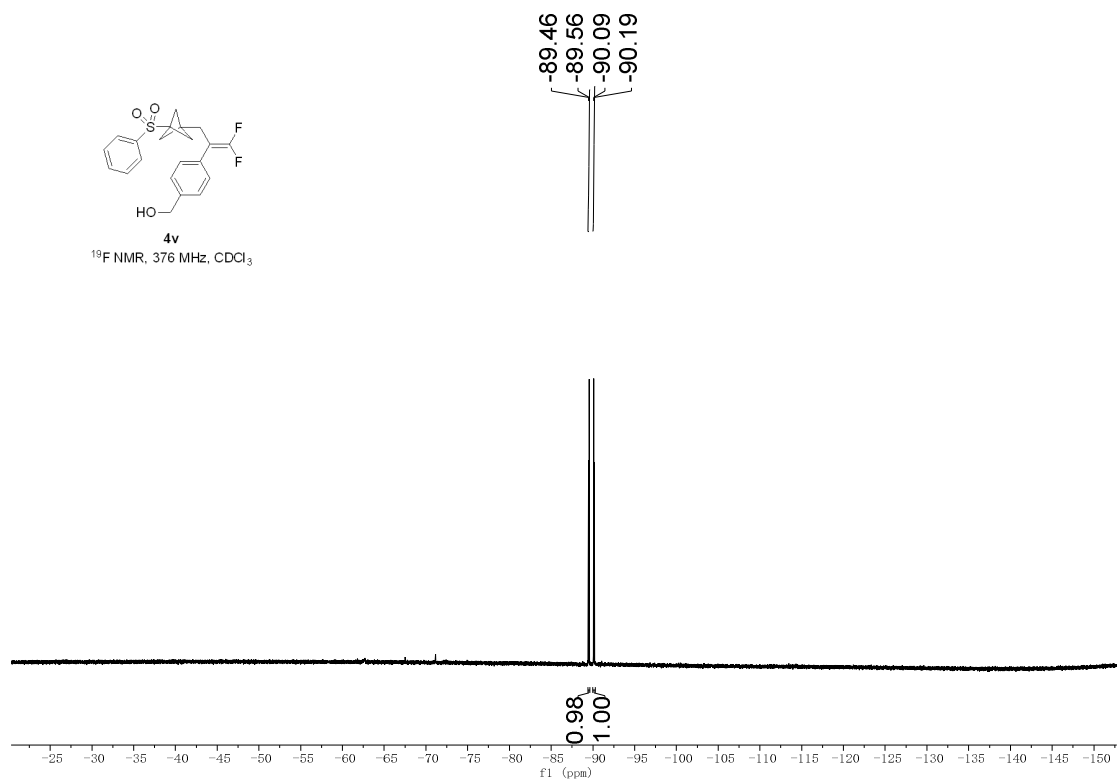
<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>

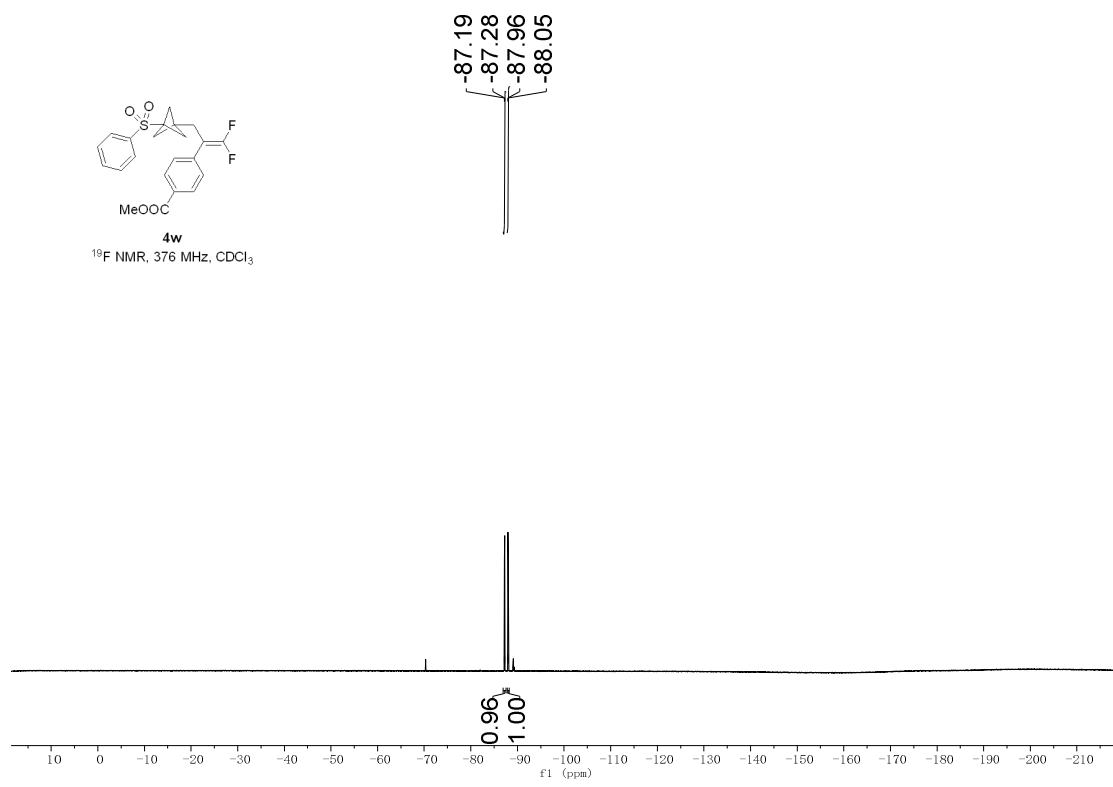
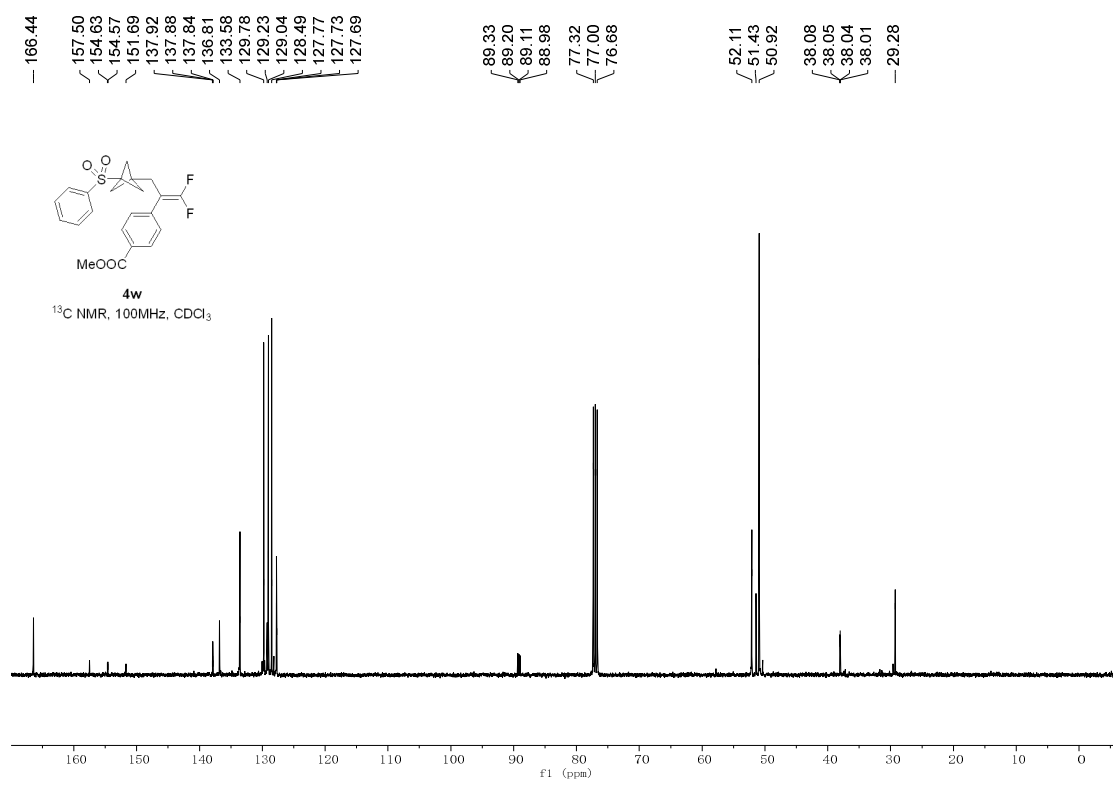


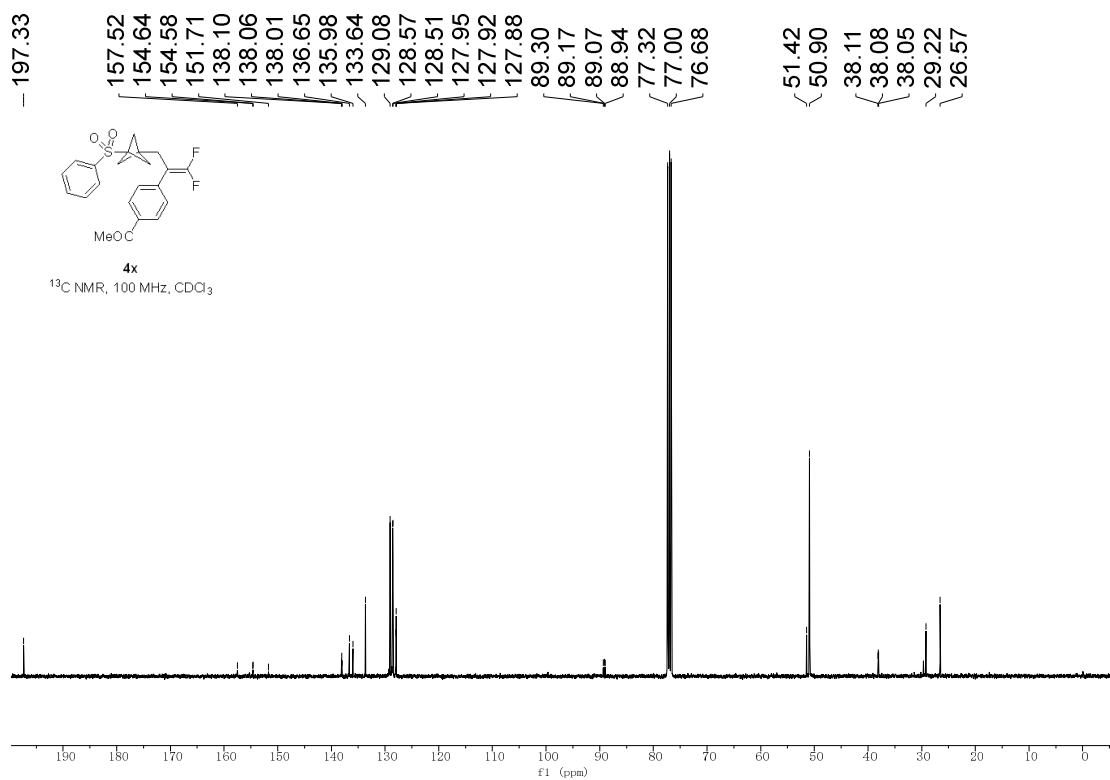
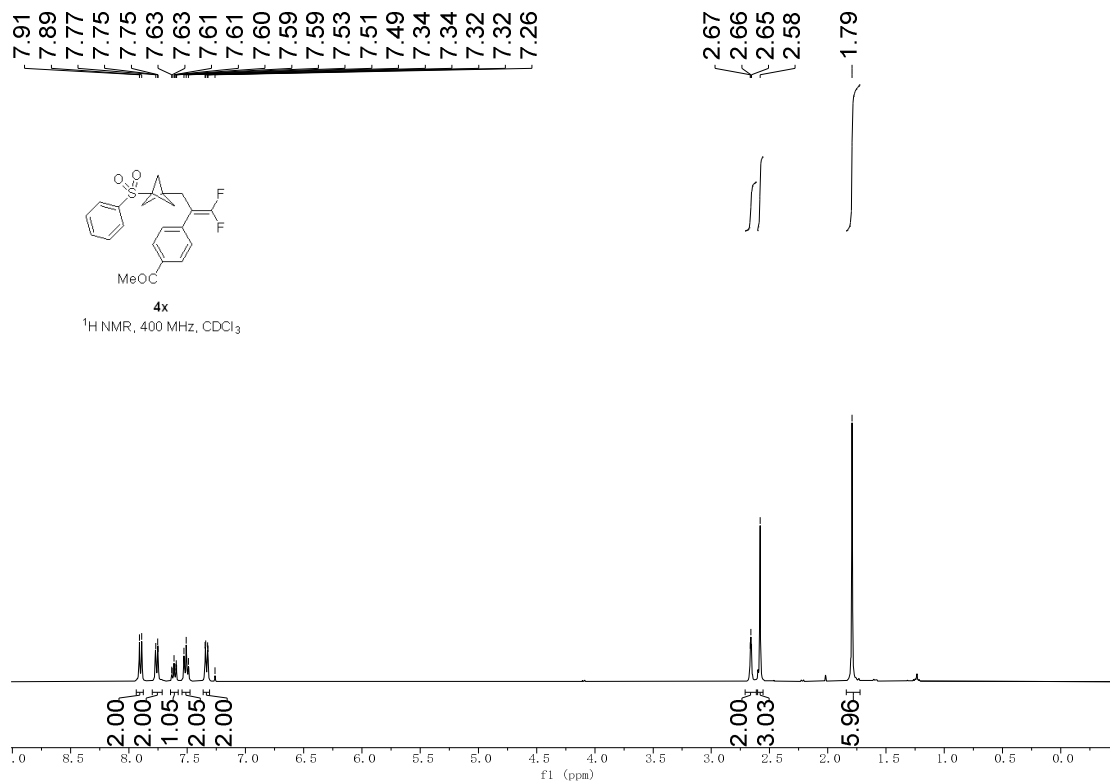




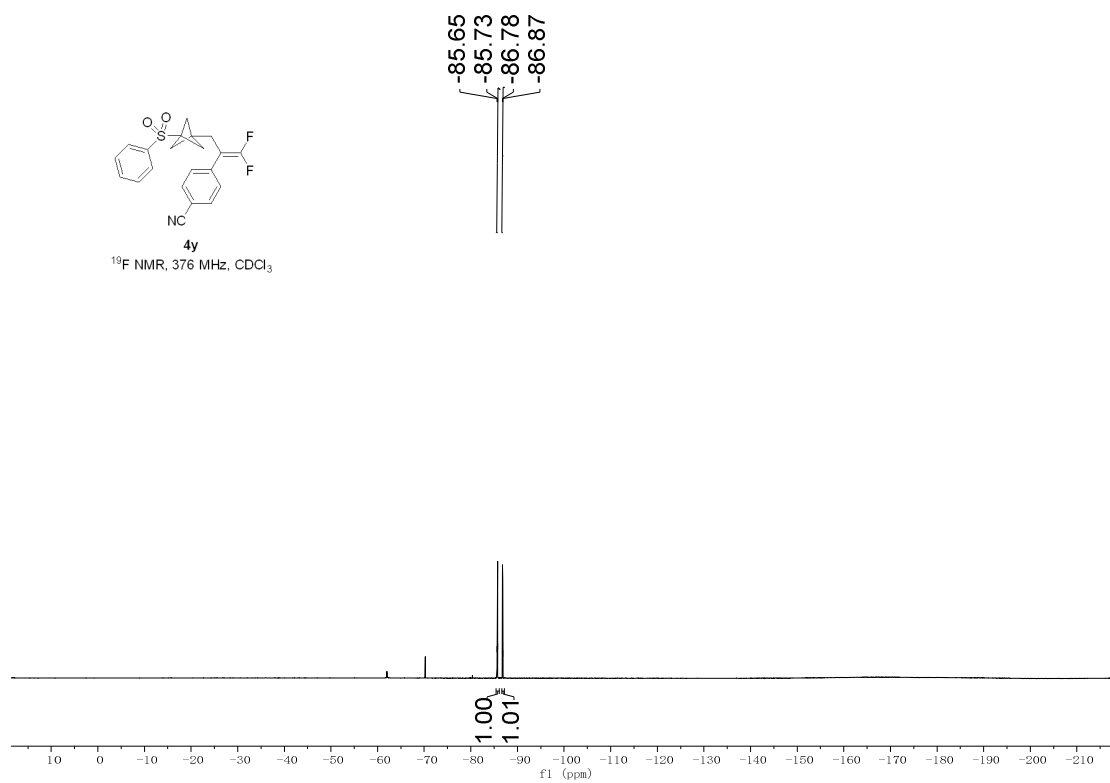
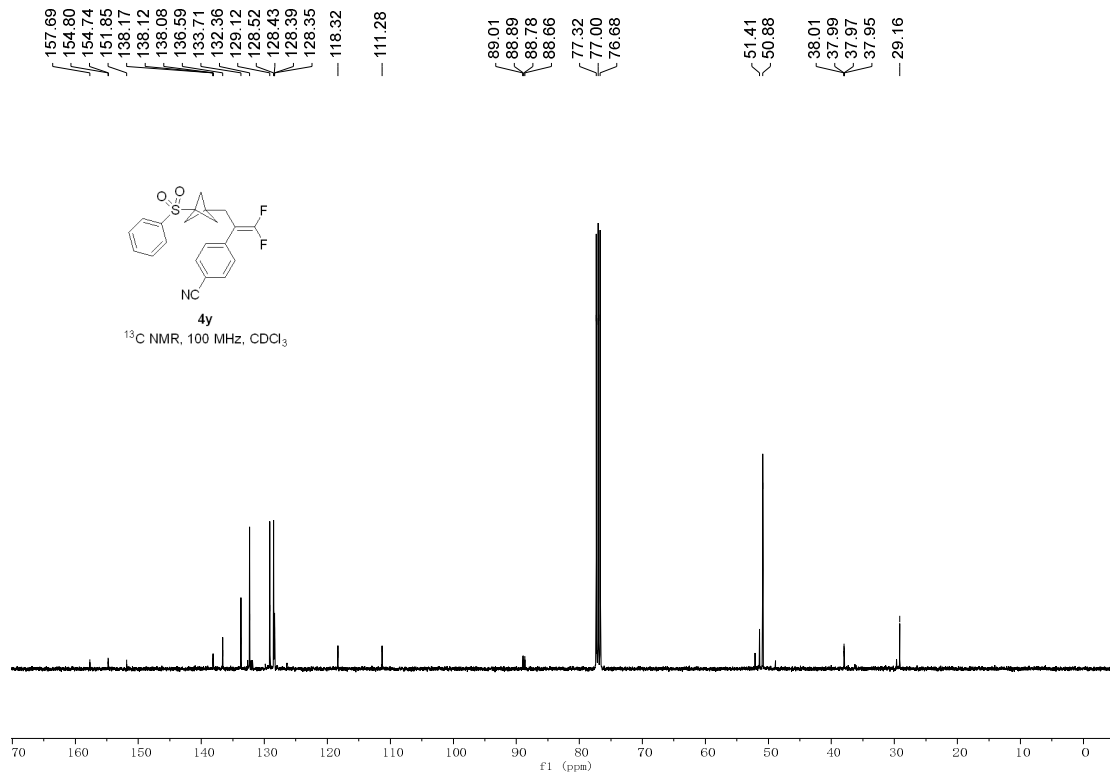


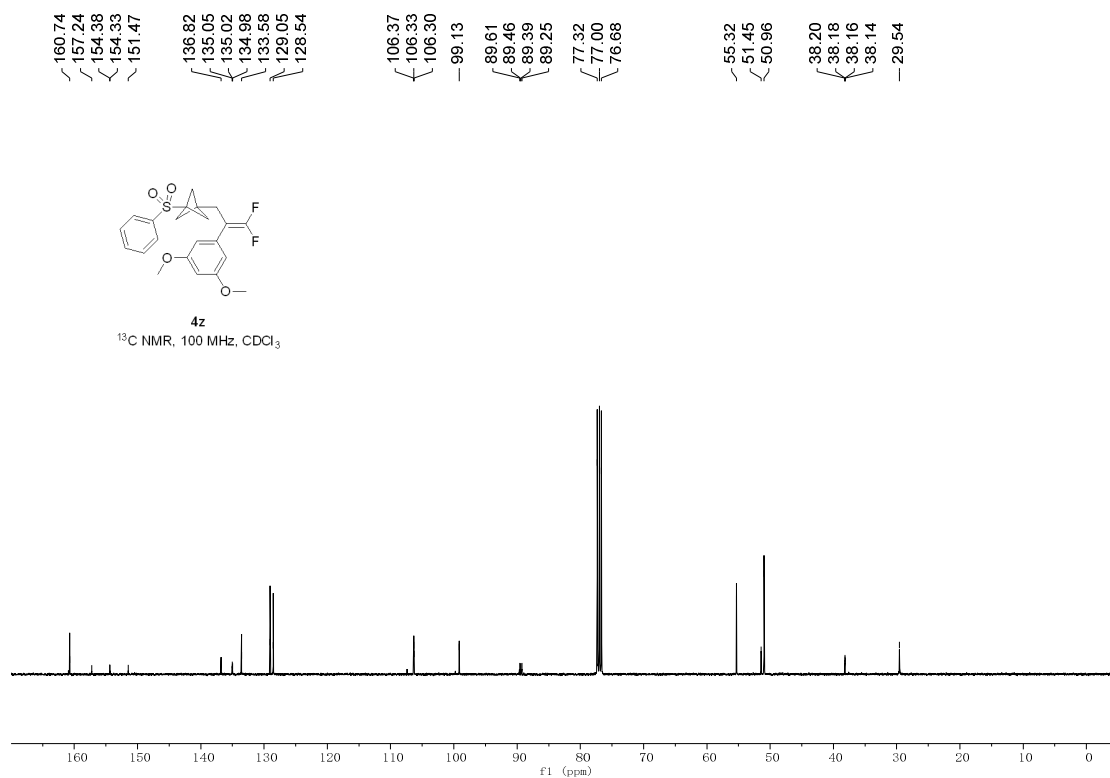
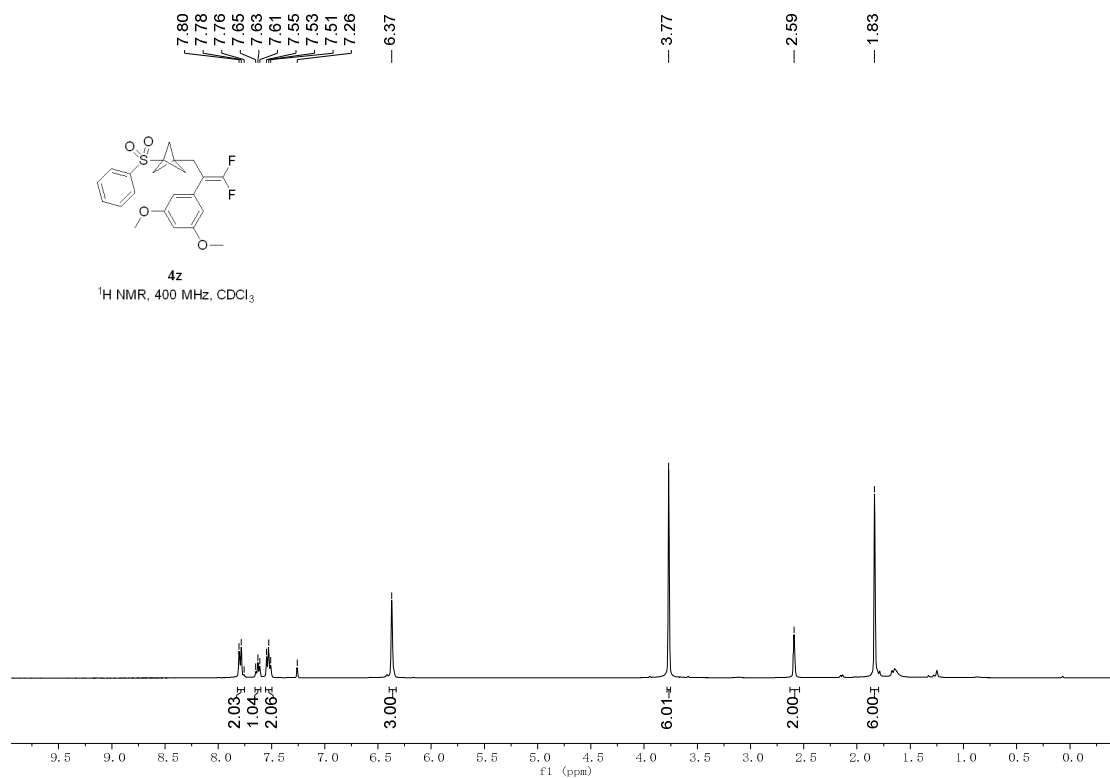


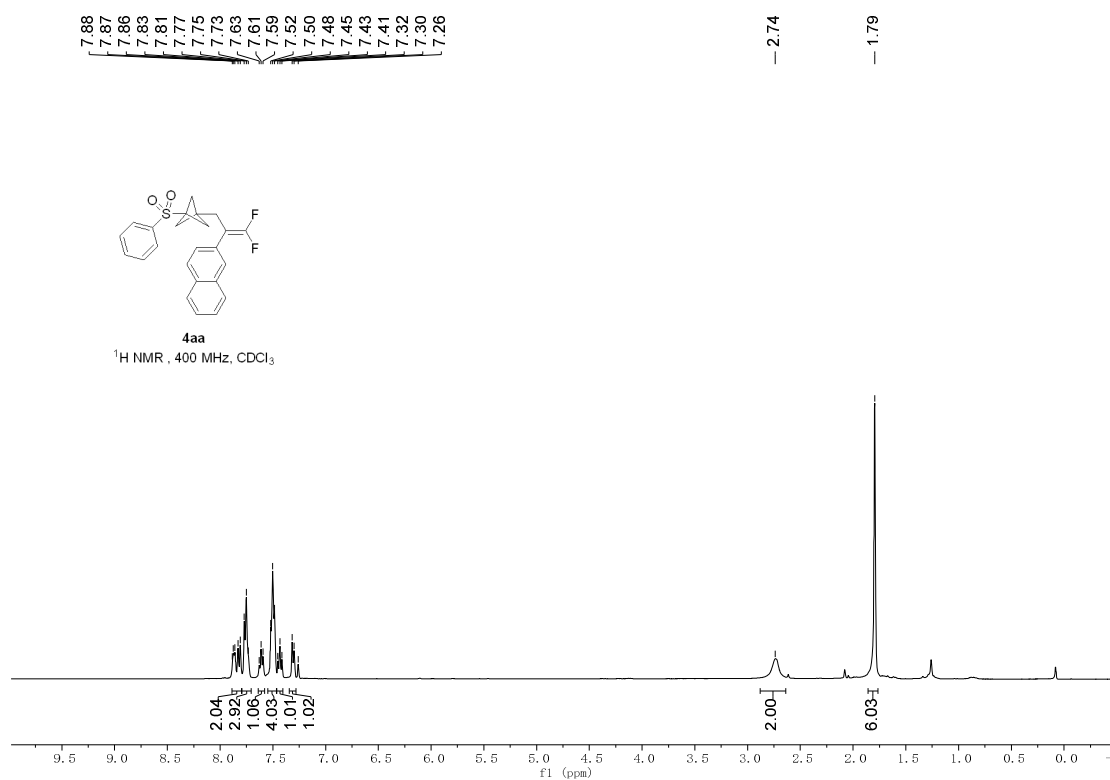
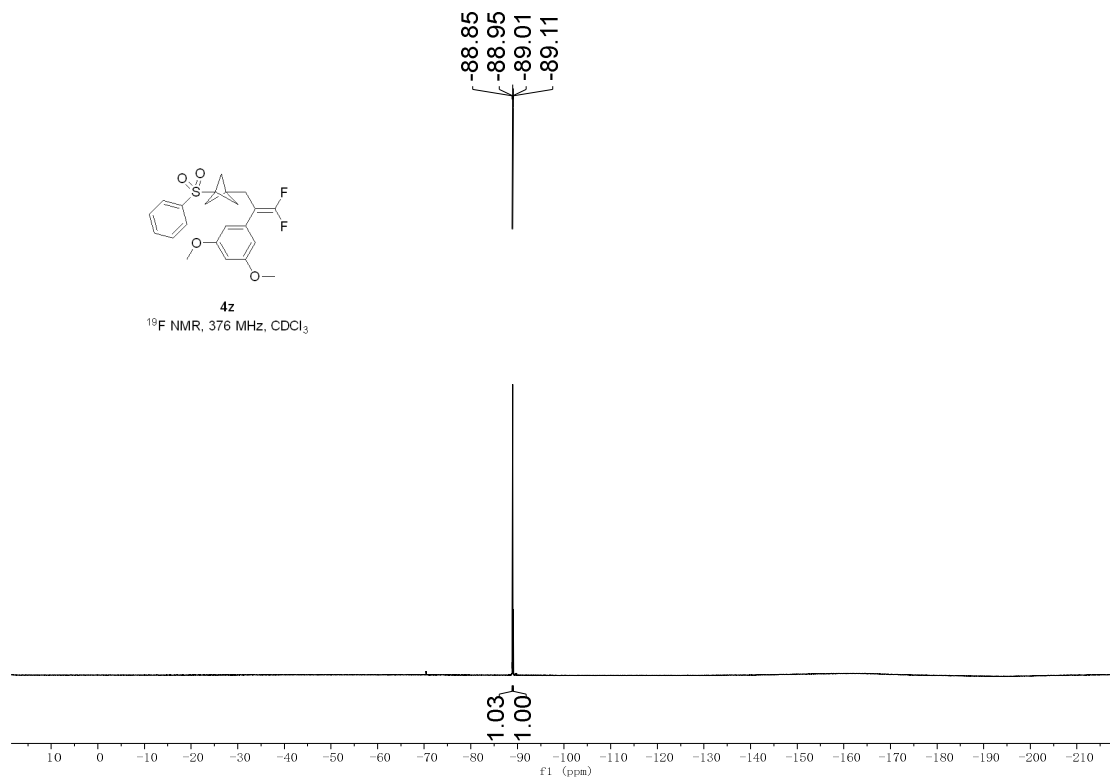


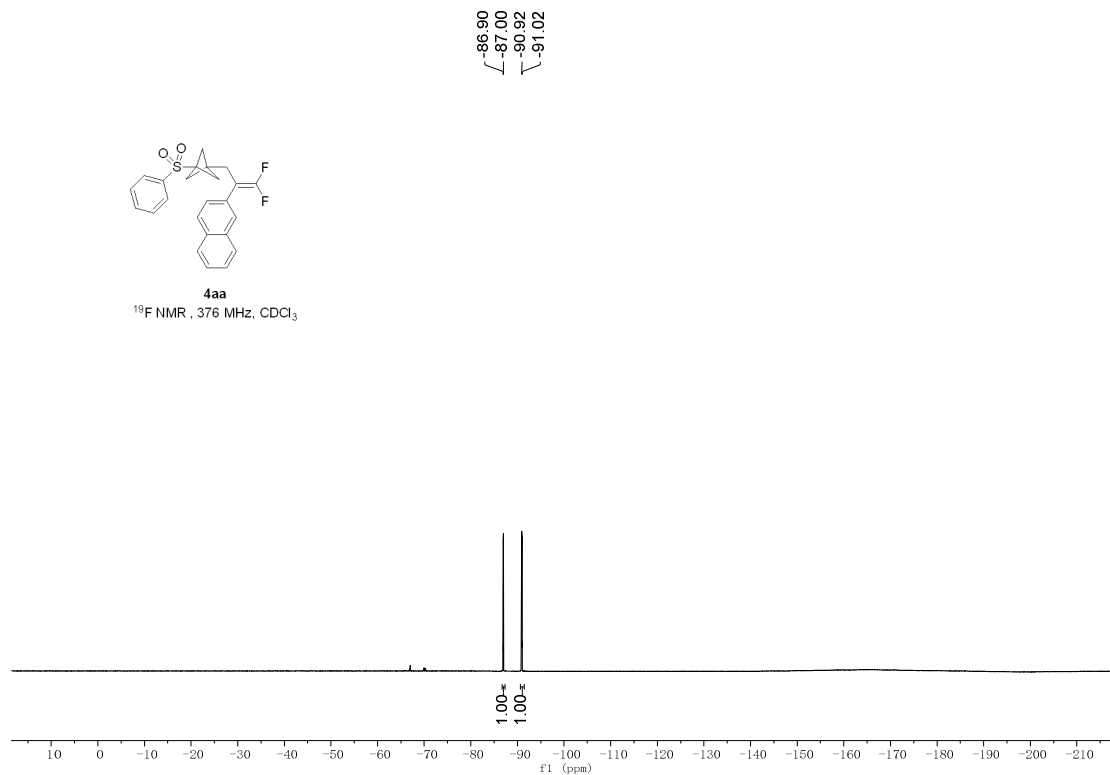
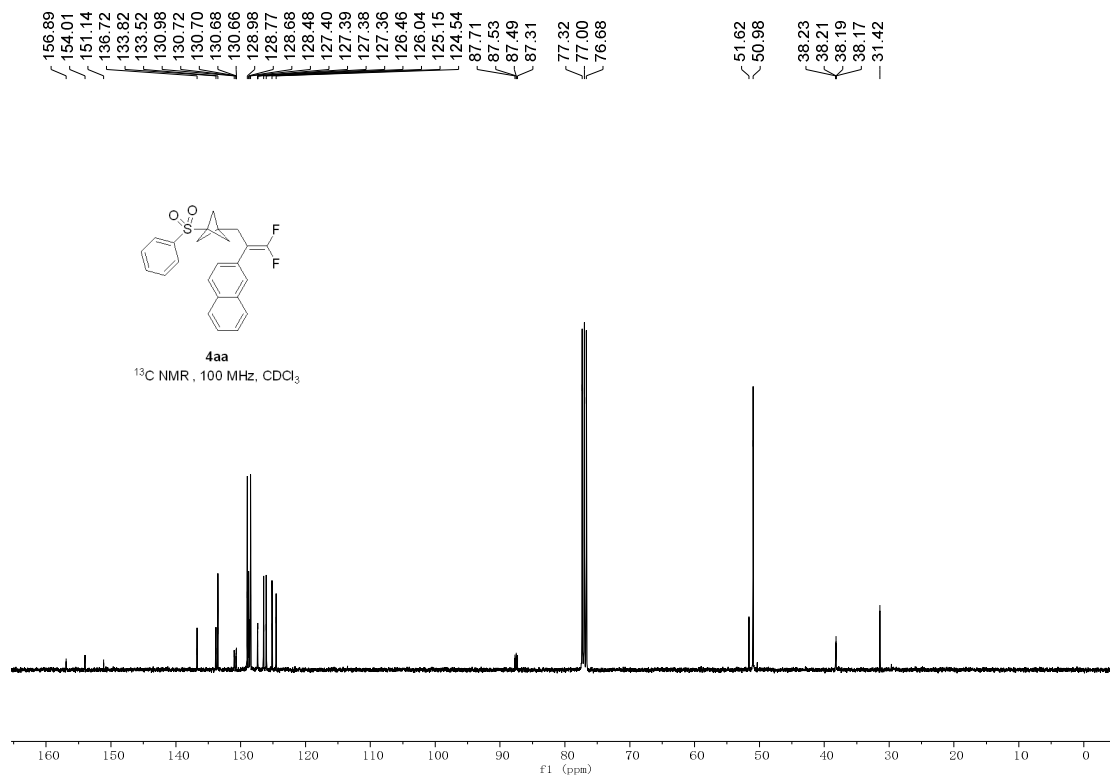




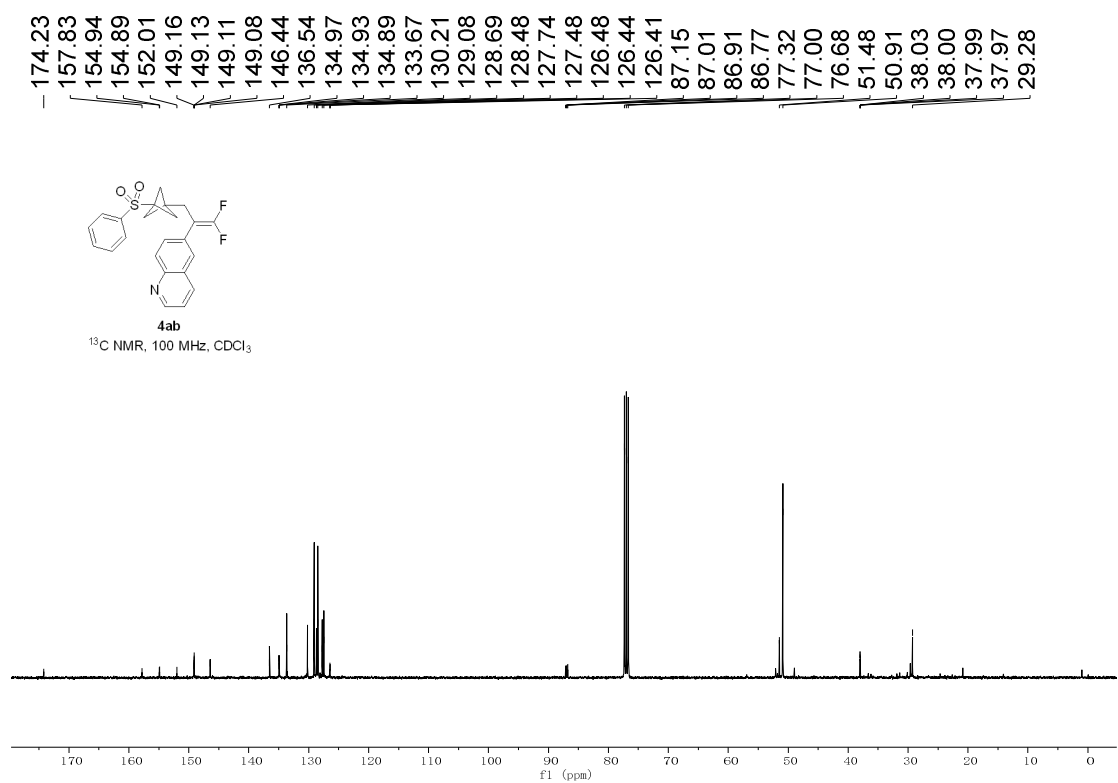
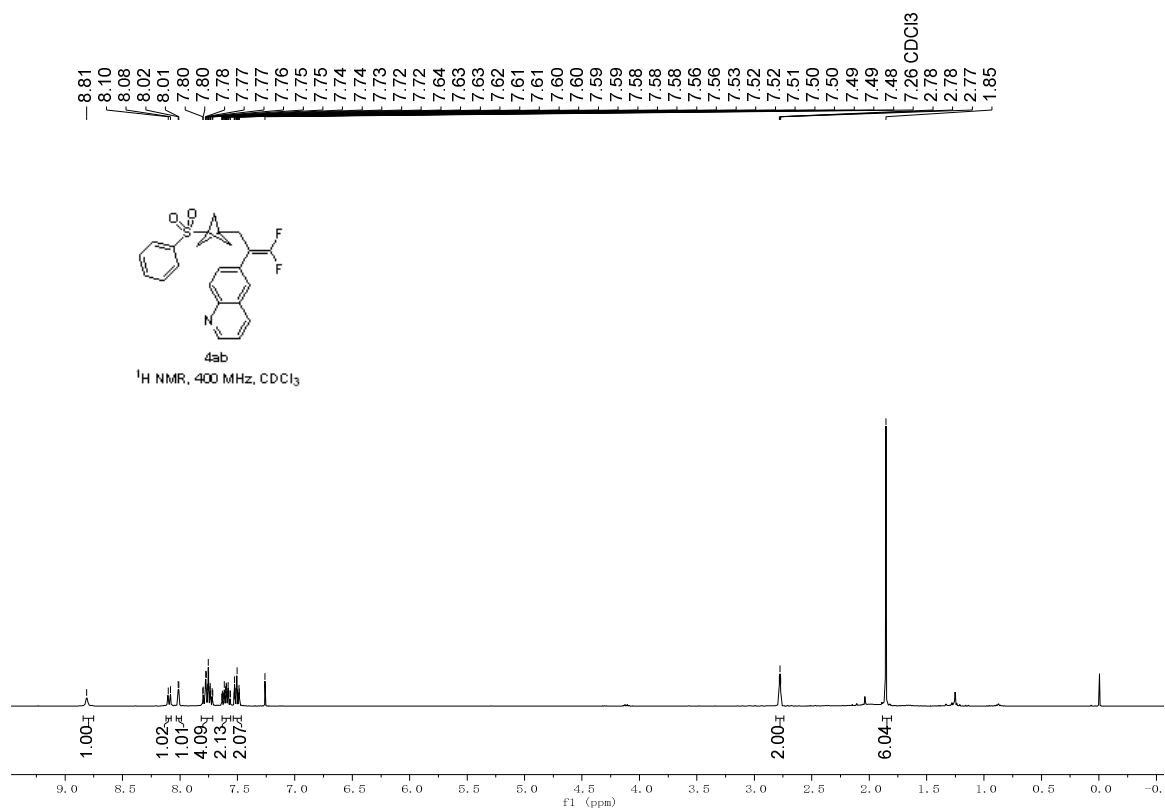


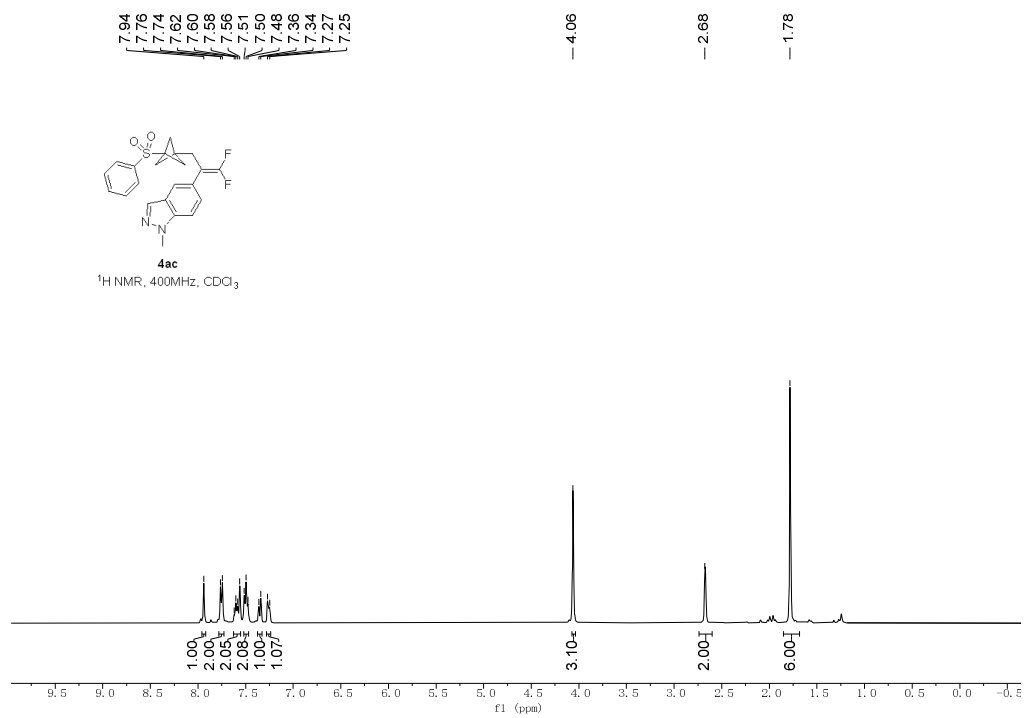
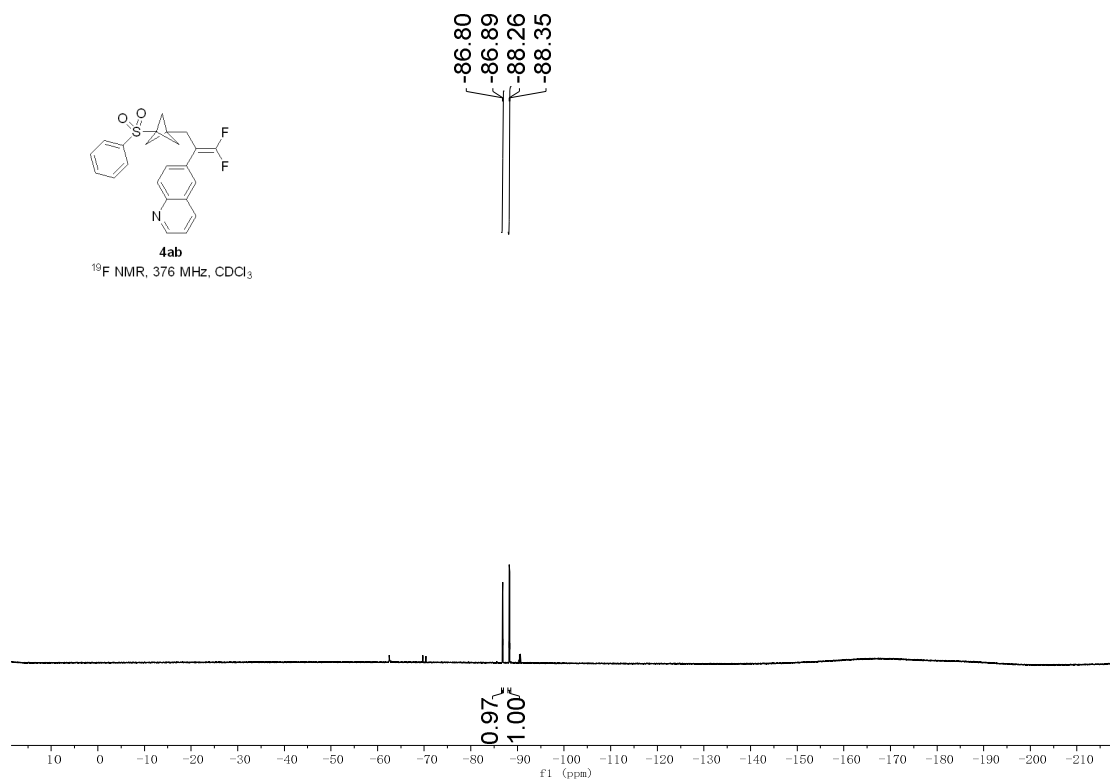




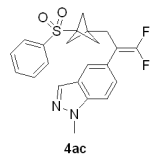




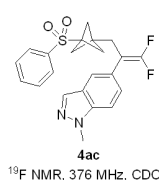
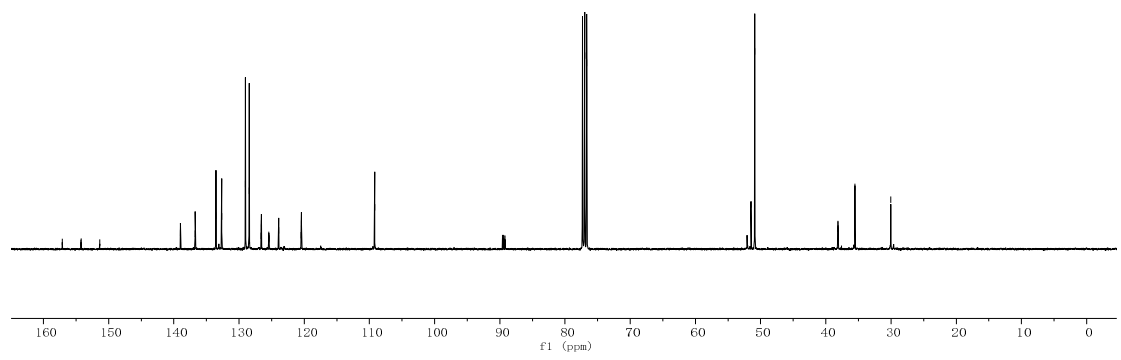




157.12  
 154.27  
 154.23  
 151.37  
 139.00  
 136.72  
 133.54  
 132.67  
 129.00  
 128.46  
 126.62  
 126.58  
 126.55  
 125.48  
 125.43  
 125.39  
 123.93  
 120.48  
 120.45  
 120.41  
 109.20  
 89.58  
 89.43  
 89.36  
 89.21  
 77.32  
 77.00  
 76.68  
 51.47  
 50.90  
 38.17  
 38.15  
 38.14  
 38.11  
 35.52  
 30.06

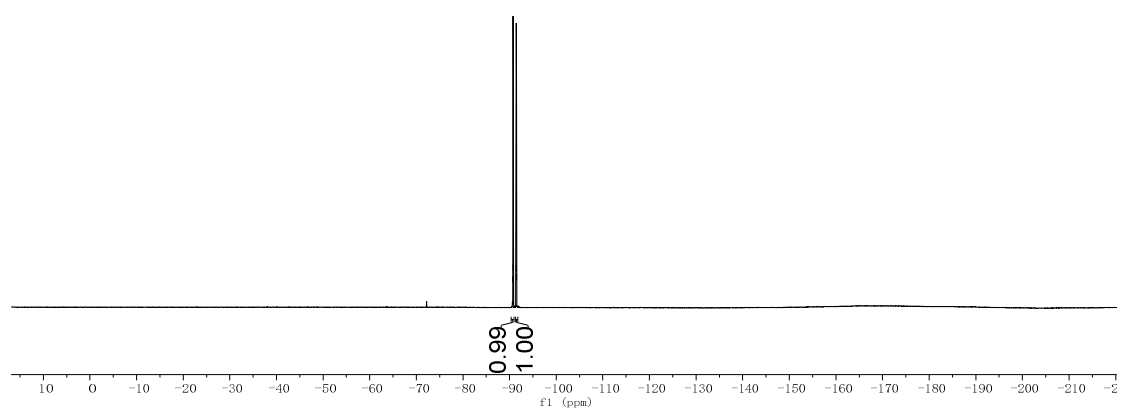


<sup>13</sup>C NMR, 100MHz, CDCl<sub>3</sub>



<sup>19</sup>F NMR, 376 MHz, CDCl<sub>3</sub>

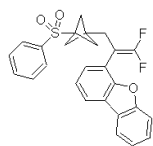
-90.66  
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 -91.42  
 -91.52



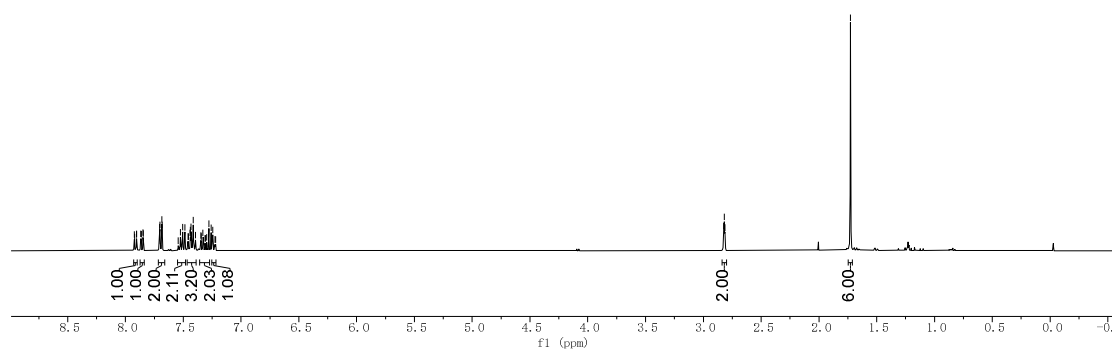
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7.92  
7.90  
7.87  
7.86  
7.85  
7.85  
7.71  
7.70  
7.69  
7.68  
7.54  
7.52  
7.51  
7.49  
7.46  
7.46  
7.44  
7.44  
7.43  
7.41  
7.39  
7.35  
7.35  
7.33  
7.31  
7.31  
7.30  
7.28  
7.26  
7.25  
7.23  
7.22

2.82  
2.81

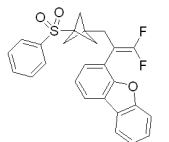
-1.73



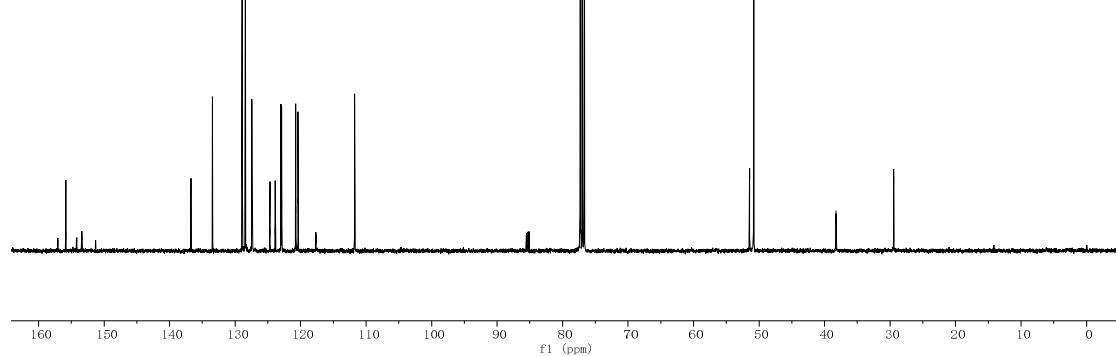
**4ad**  
<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>

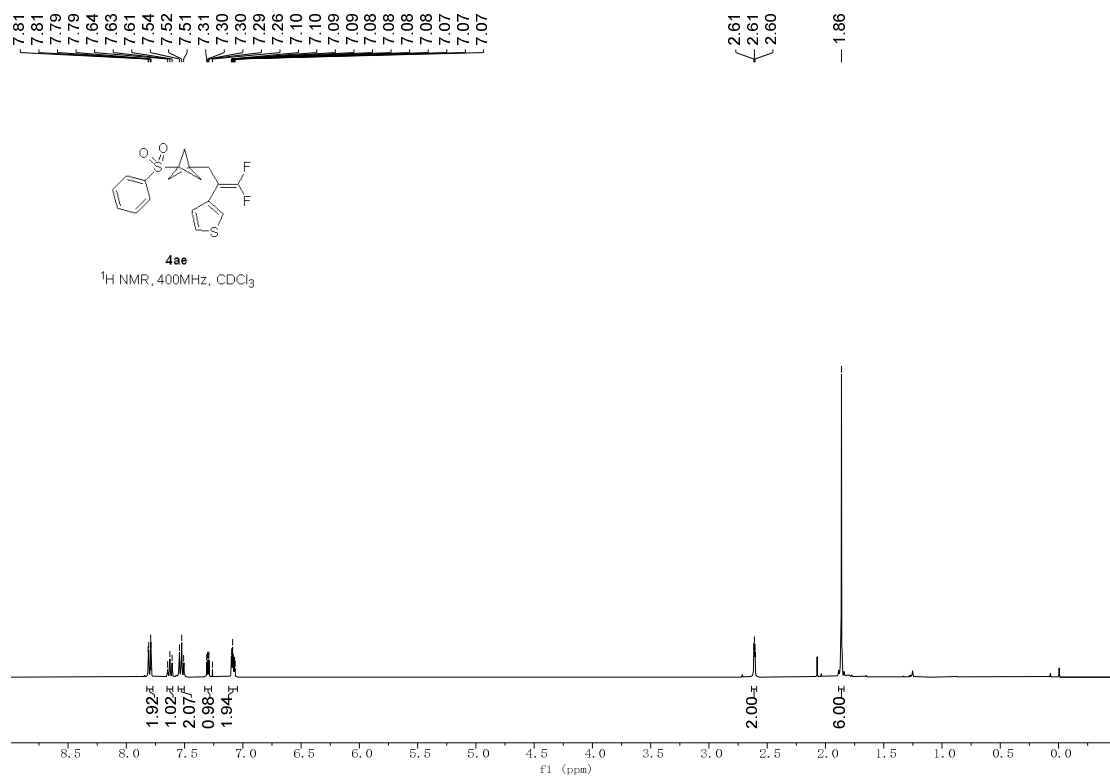
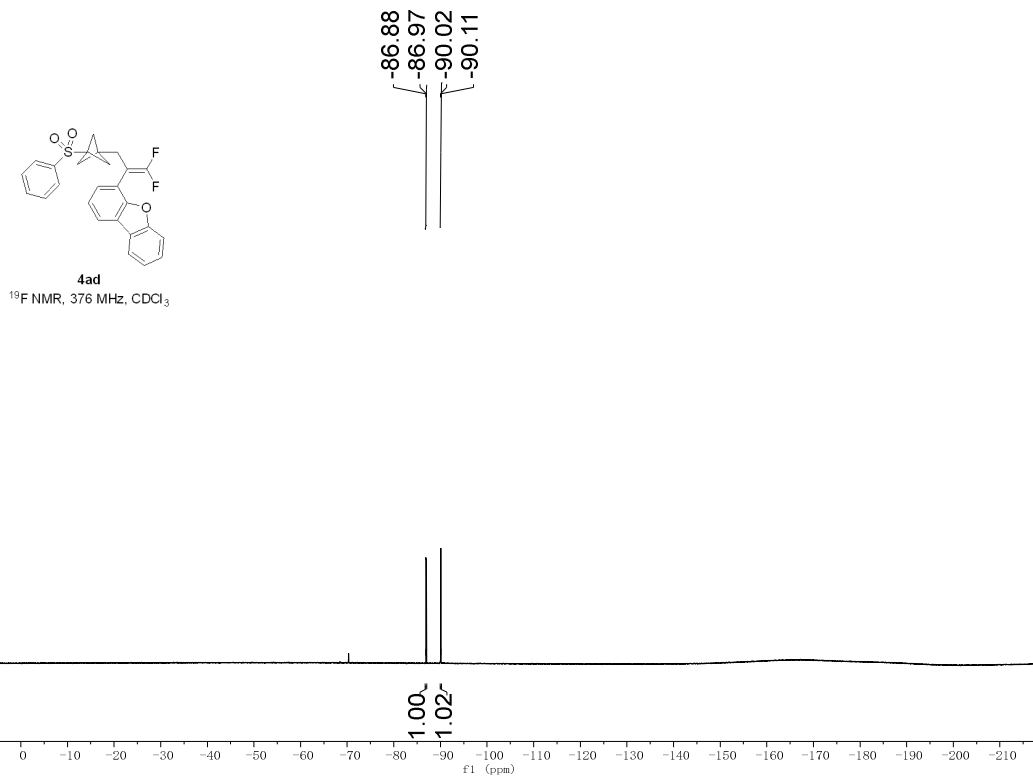


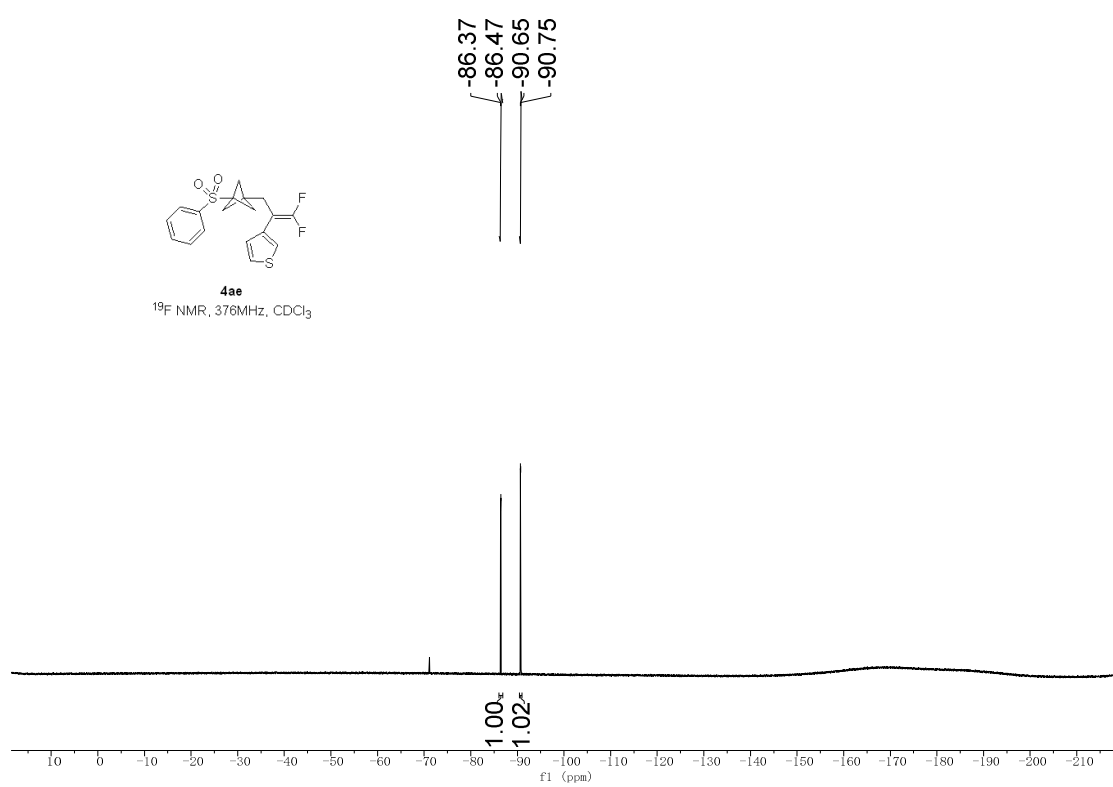
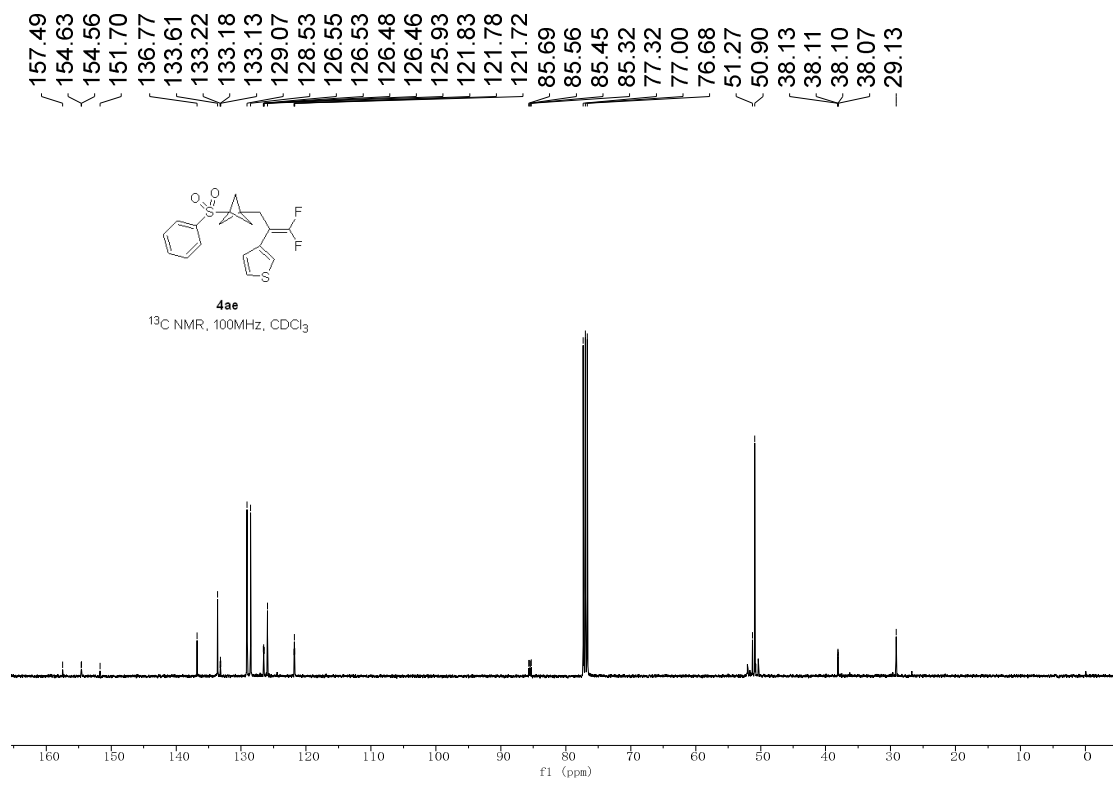
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154.21  
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153.38  
151.30  
136.74  
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128.92  
128.44  
127.45  
127.41  
127.38  
127.36  
124.68  
123.86  
122.99  
122.89  
120.74  
120.40  
117.69  
117.66  
117.64  
117.61  
111.73  
85.52  
85.36  
85.27  
85.11  
77.32  
77.00  
76.68  
51.44  
50.83  
38.27  
38.25  
38.23  
38.21  
29.45

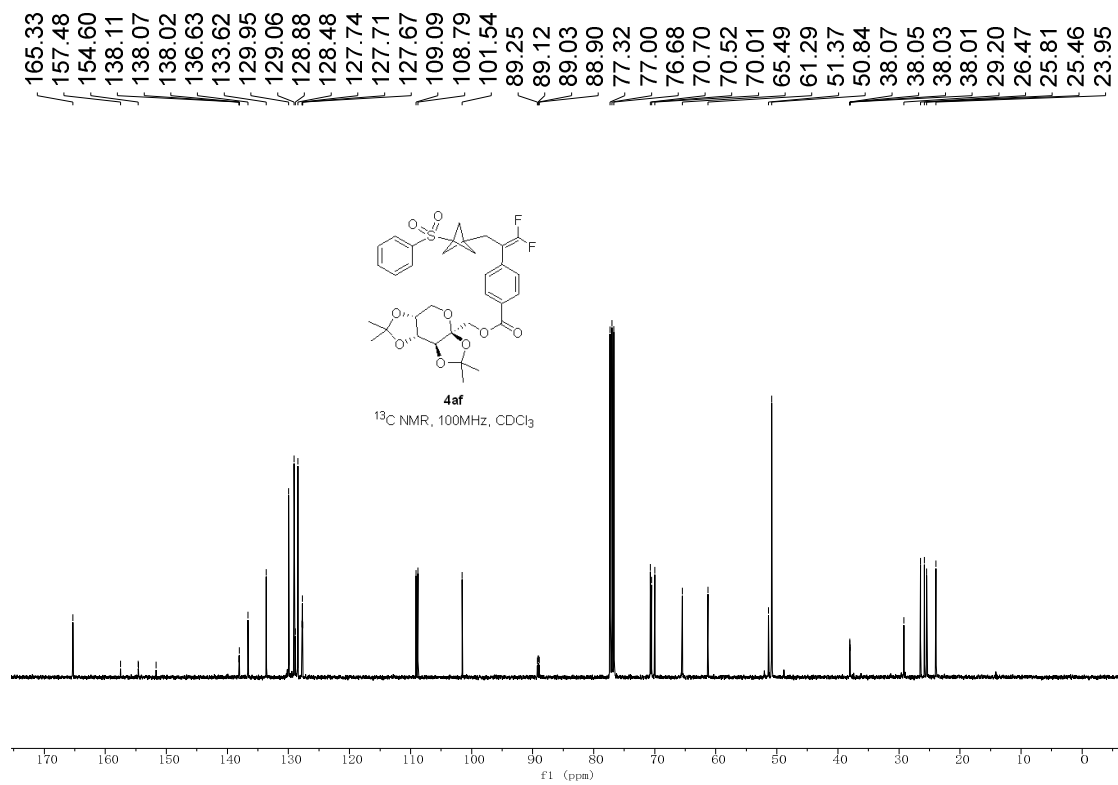
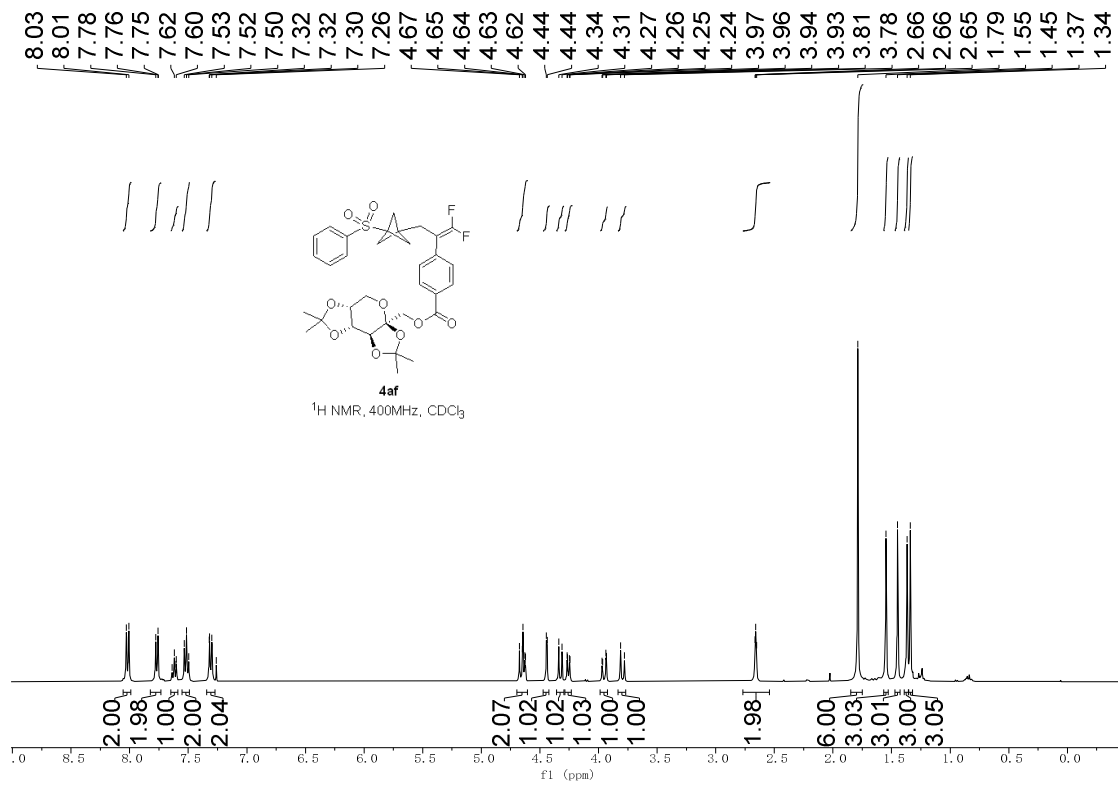


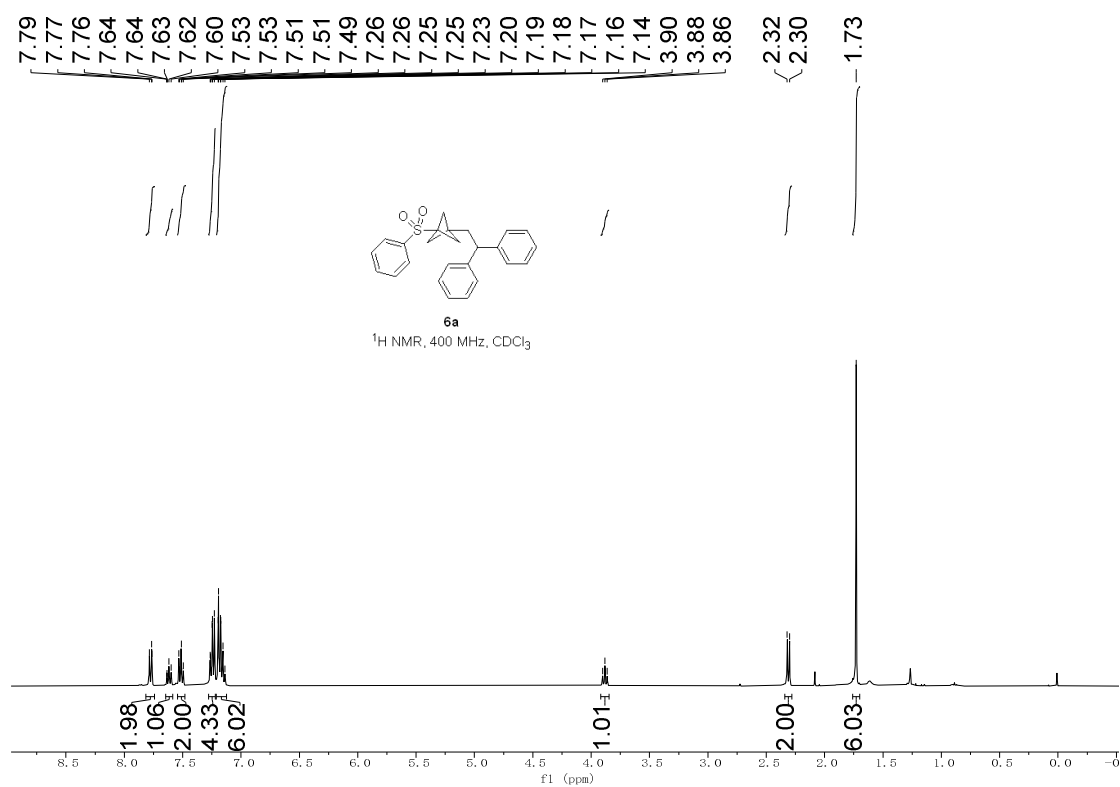
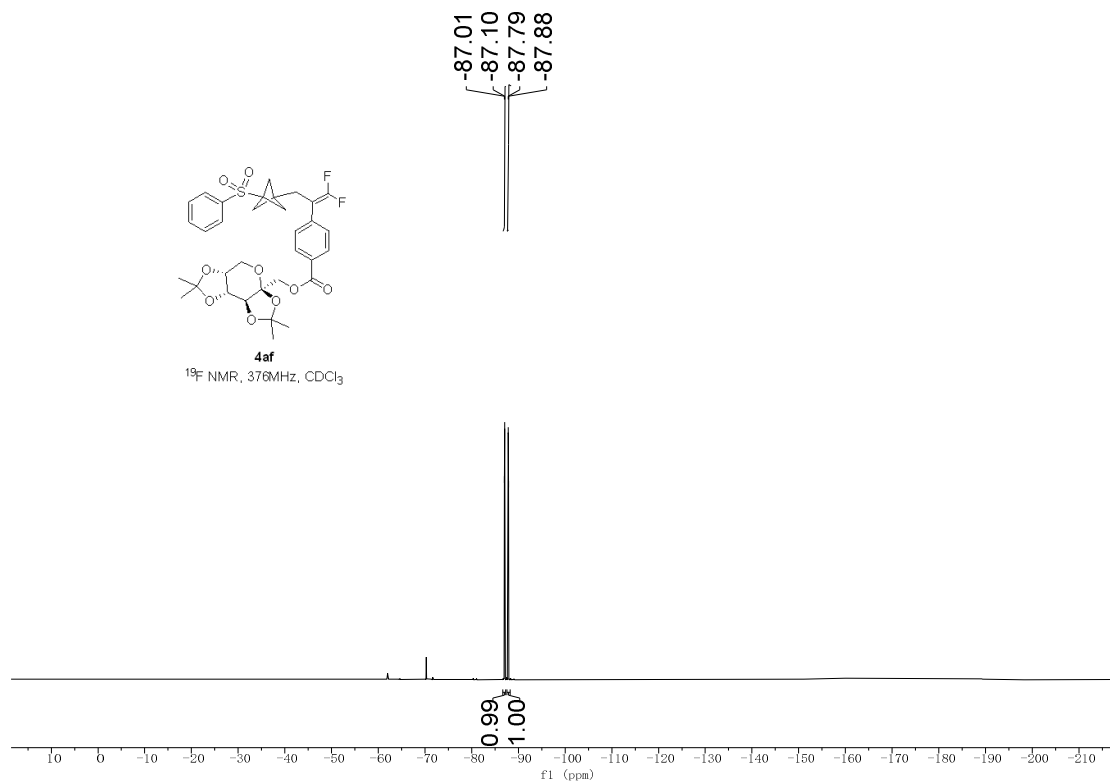
**4ad**  
<sup>13</sup>C NMR, 100MHz, CDCl<sub>3</sub>



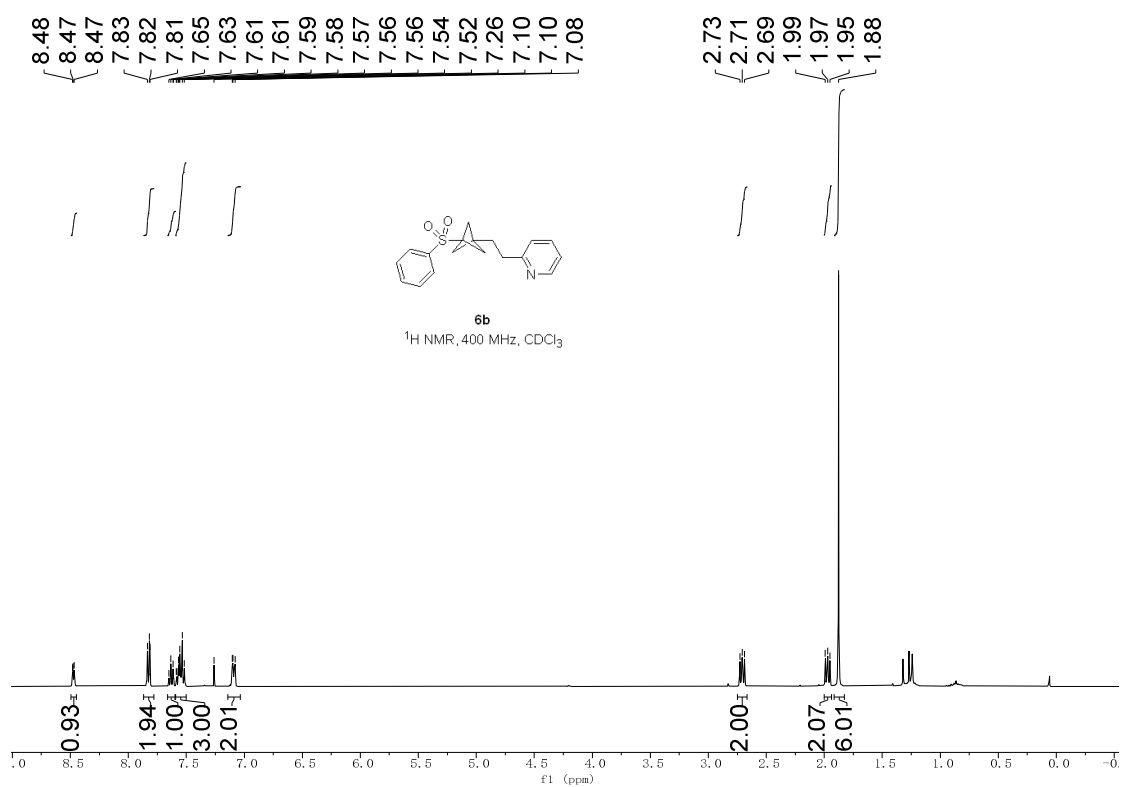
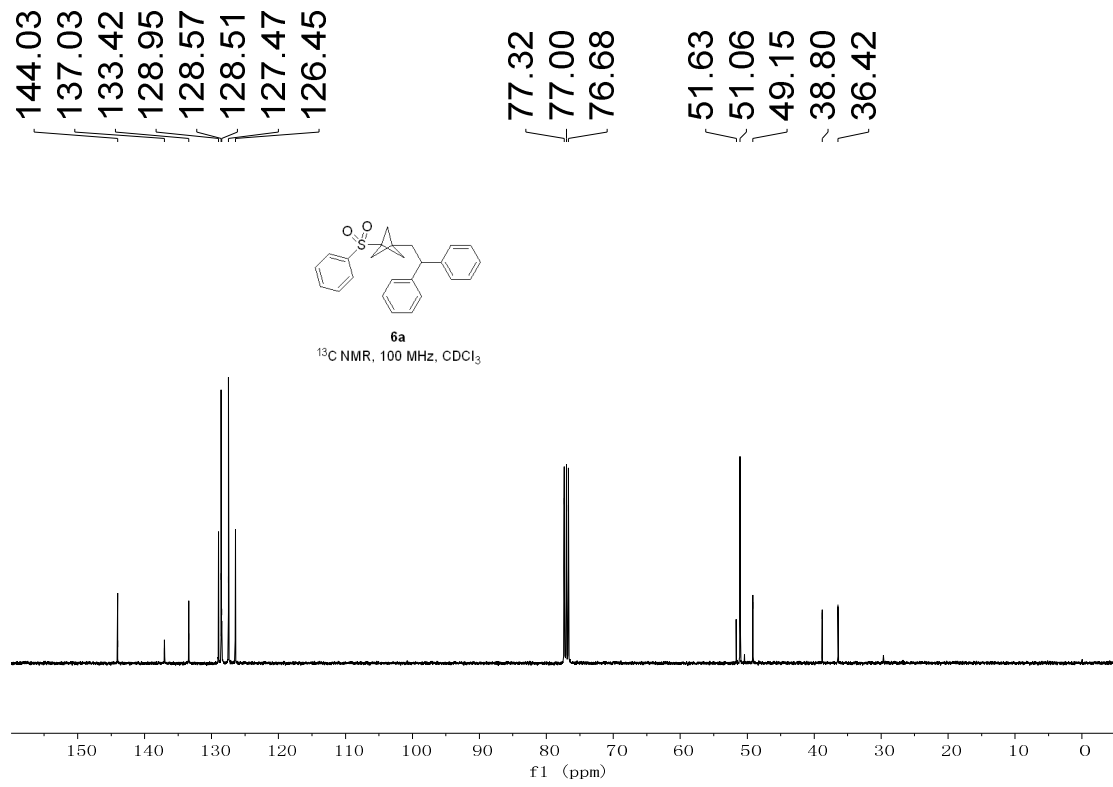


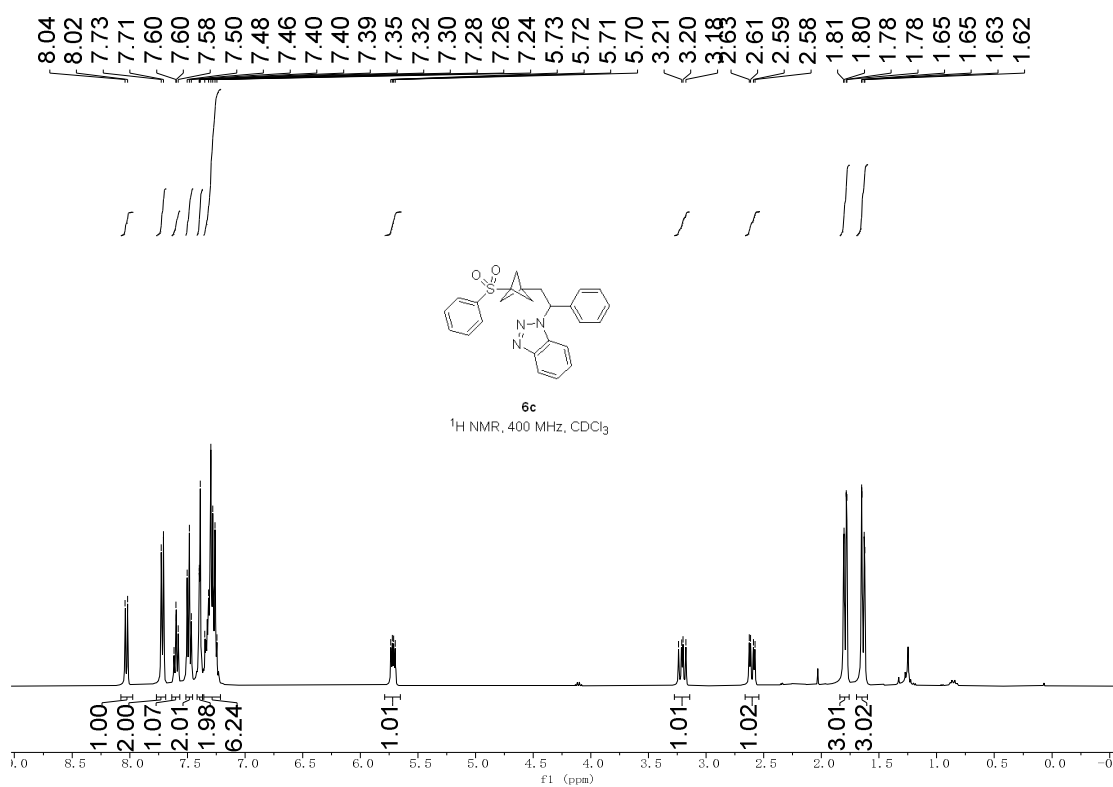
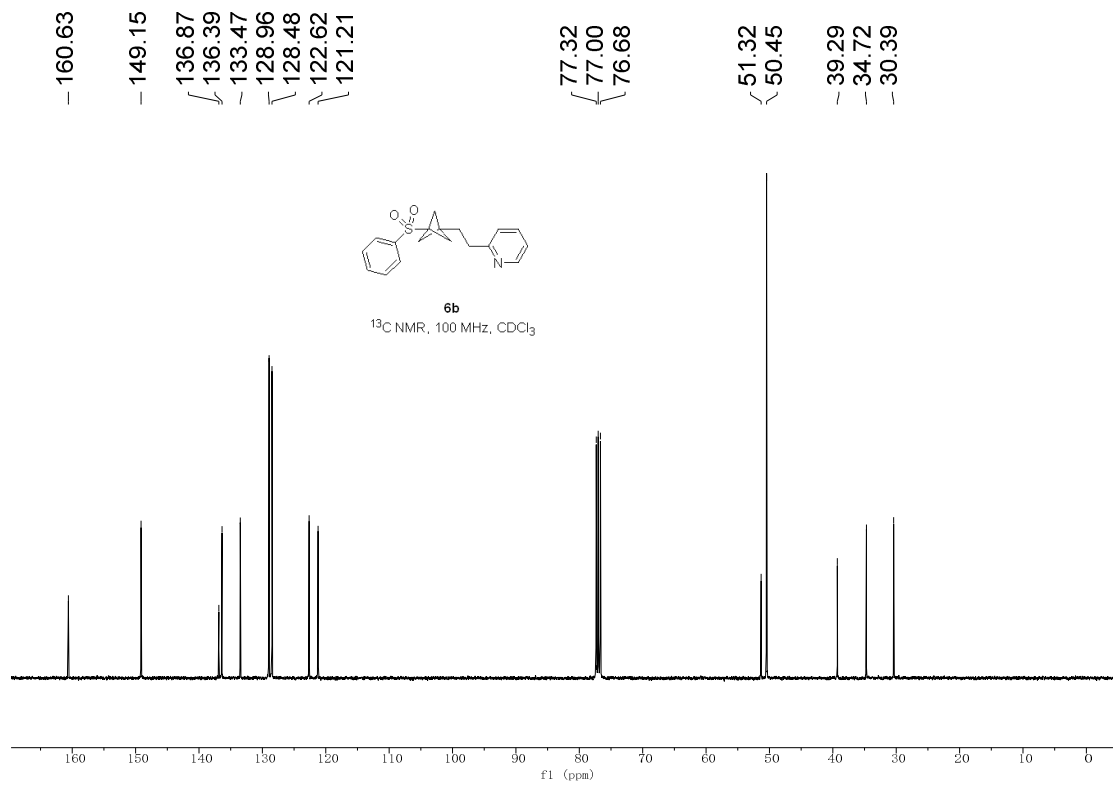




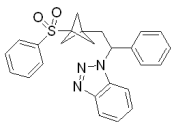




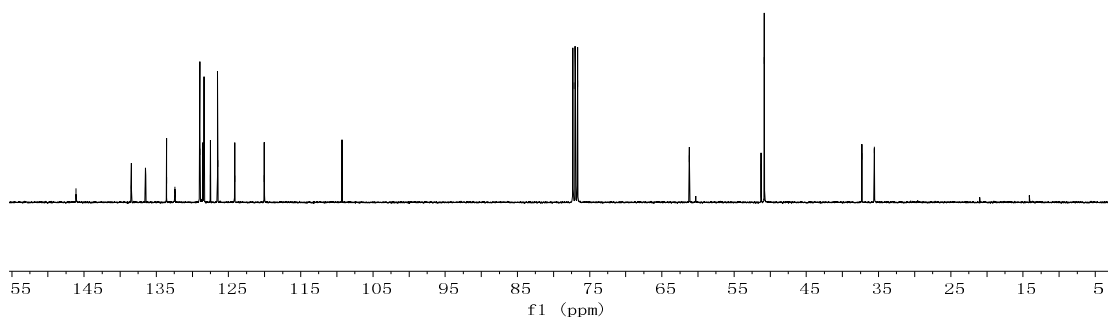




146.10  
138.46  
136.48  
133.59  
132.43  
129.00  
128.97  
128.56  
128.40  
127.51  
126.50  
124.14  
120.06  
109.28  
77.32  
77.00  
76.68  
61.21  
51.26  
50.81  
37.32  
35.60

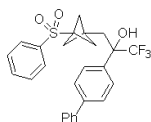
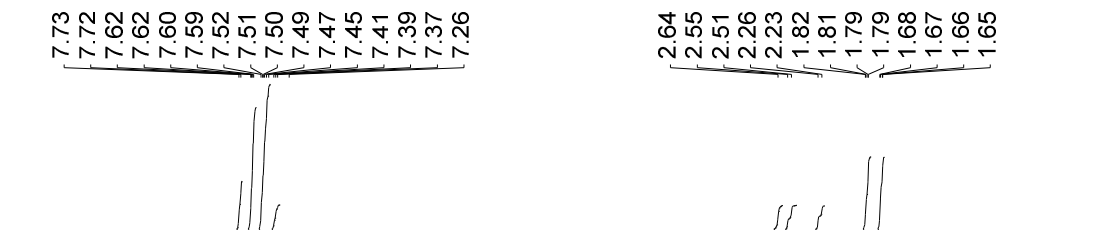


6c  
<sup>13</sup>C NMR, 100 MHz, CDCl<sub>3</sub>

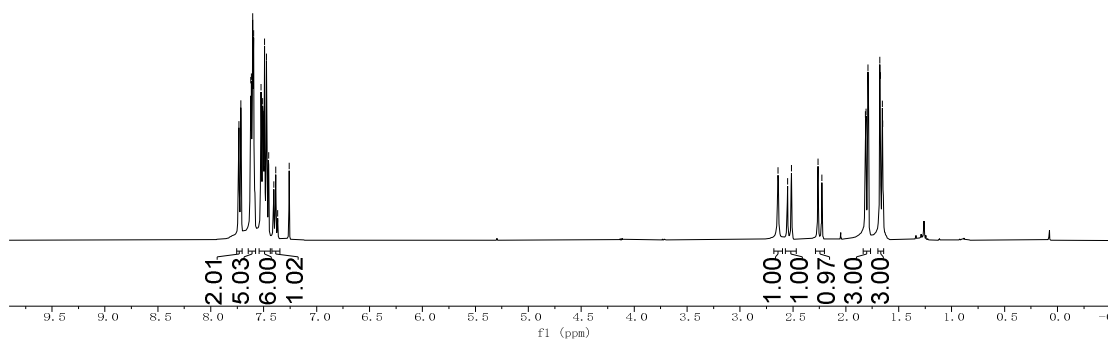


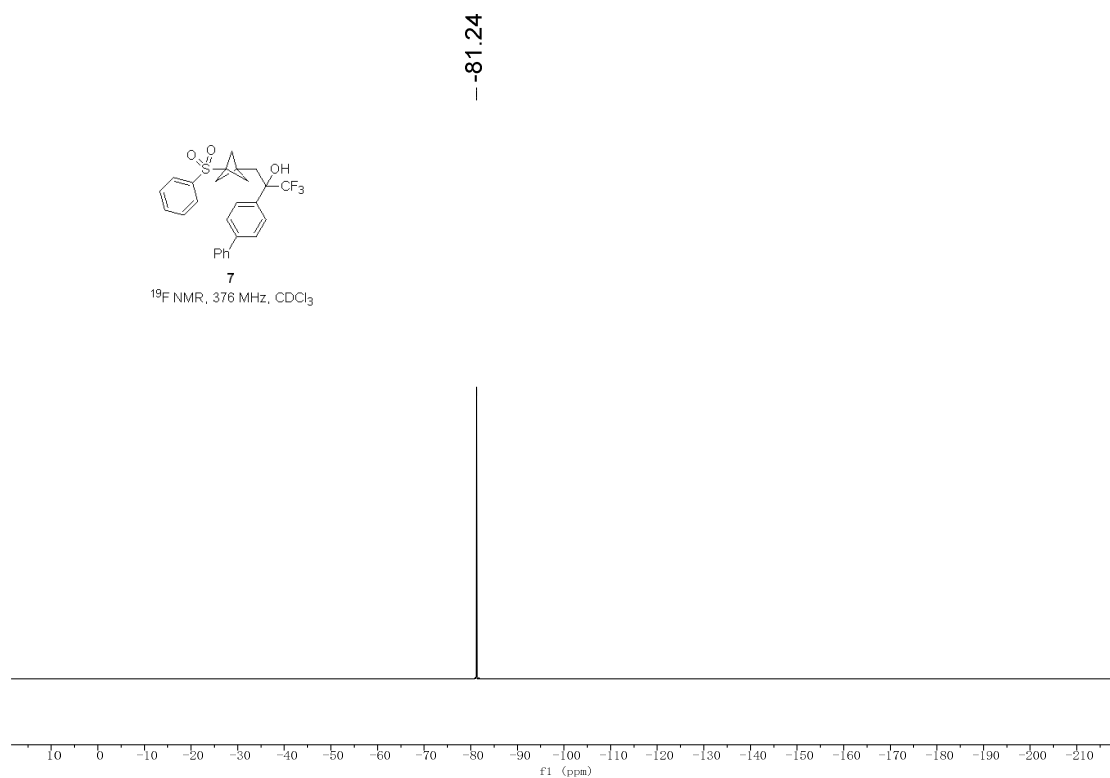
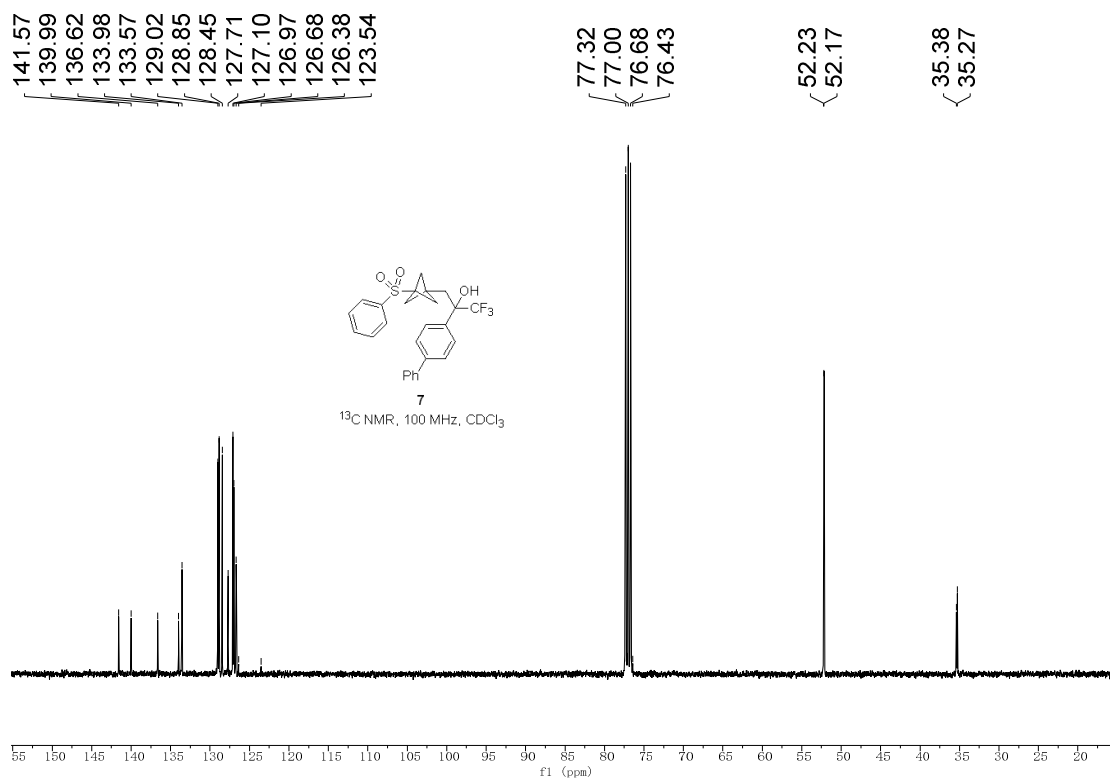
7.73  
7.72  
7.62  
7.62  
7.60  
7.59  
7.52  
7.51  
7.50  
7.49  
7.47  
7.45  
7.41  
7.39  
7.37  
7.26

2.64  
2.55  
2.51  
2.26  
2.23  
1.82  
1.81  
1.79  
1.68  
1.67  
1.66  
1.65

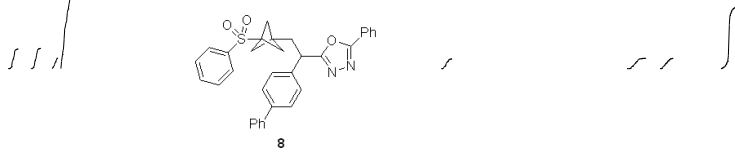


7  
<sup>1</sup>H NMR, 400MHz, CDCl<sub>3</sub>

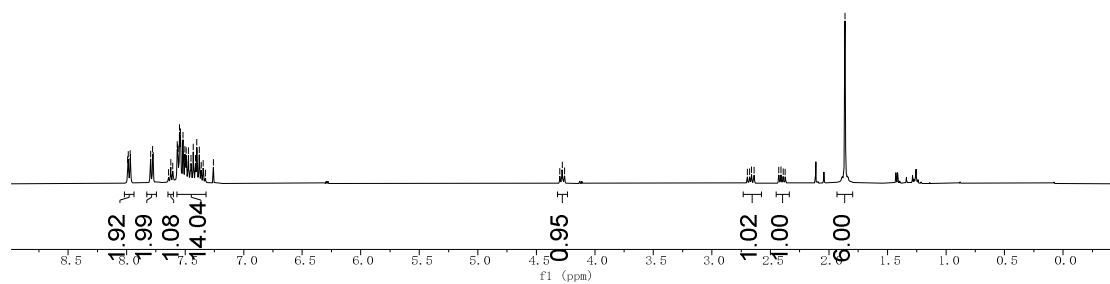




7.99  
7.99  
7.97  
7.97  
7.80  
7.78  
7.77  
7.64  
7.62  
7.61  
7.57  
7.56  
7.56  
7.55  
7.54  
7.52  
7.50  
7.49  
7.47  
7.45  
7.43  
7.41  
7.40  
7.38  
7.36  
7.35  
7.33  
7.26  
4.30  
4.28  
4.26  
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2.68  
2.66  
2.64  
2.43  
2.41  
2.39  
2.37  
1.86



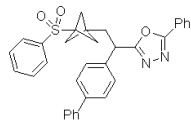
<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>



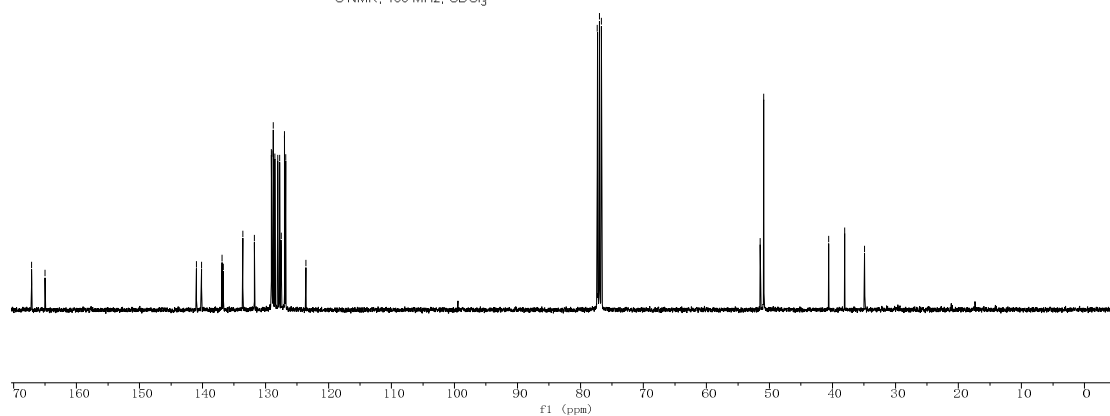
167.13  
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140.95  
140.17  
136.91  
136.69  
133.60  
131.75  
129.05  
128.98  
128.78  
128.51  
128.03  
127.73  
127.52  
126.98  
126.81  
123.60

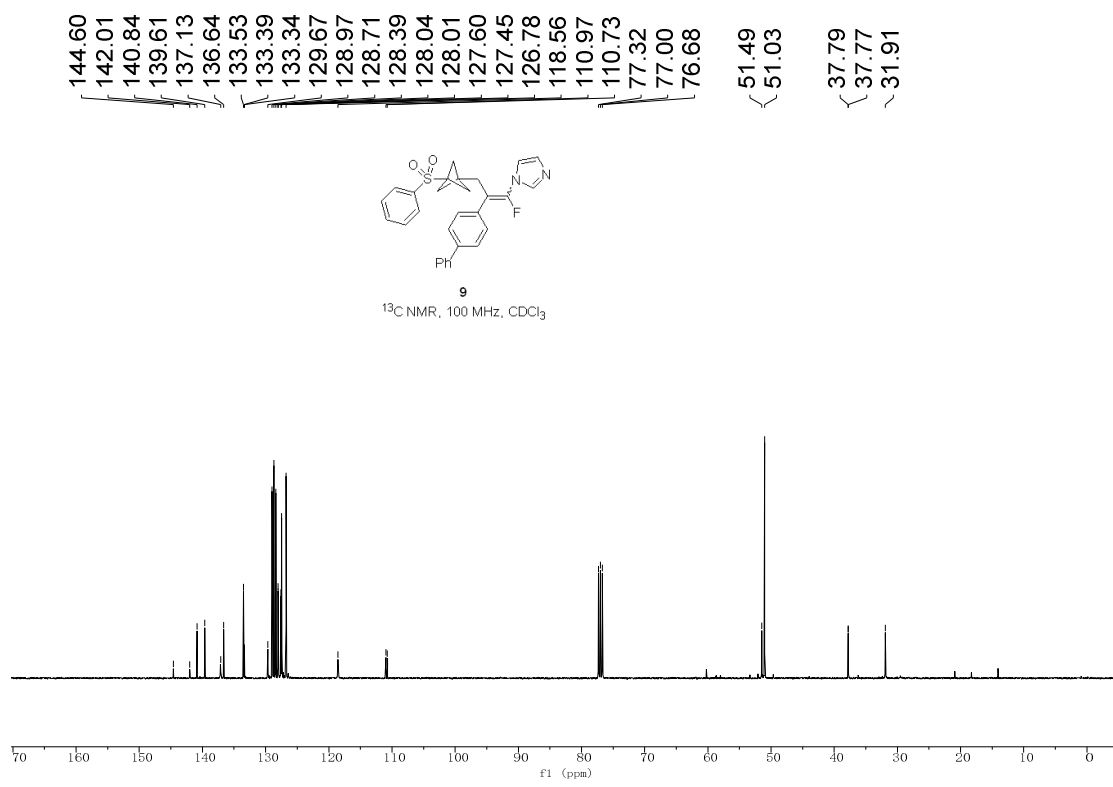
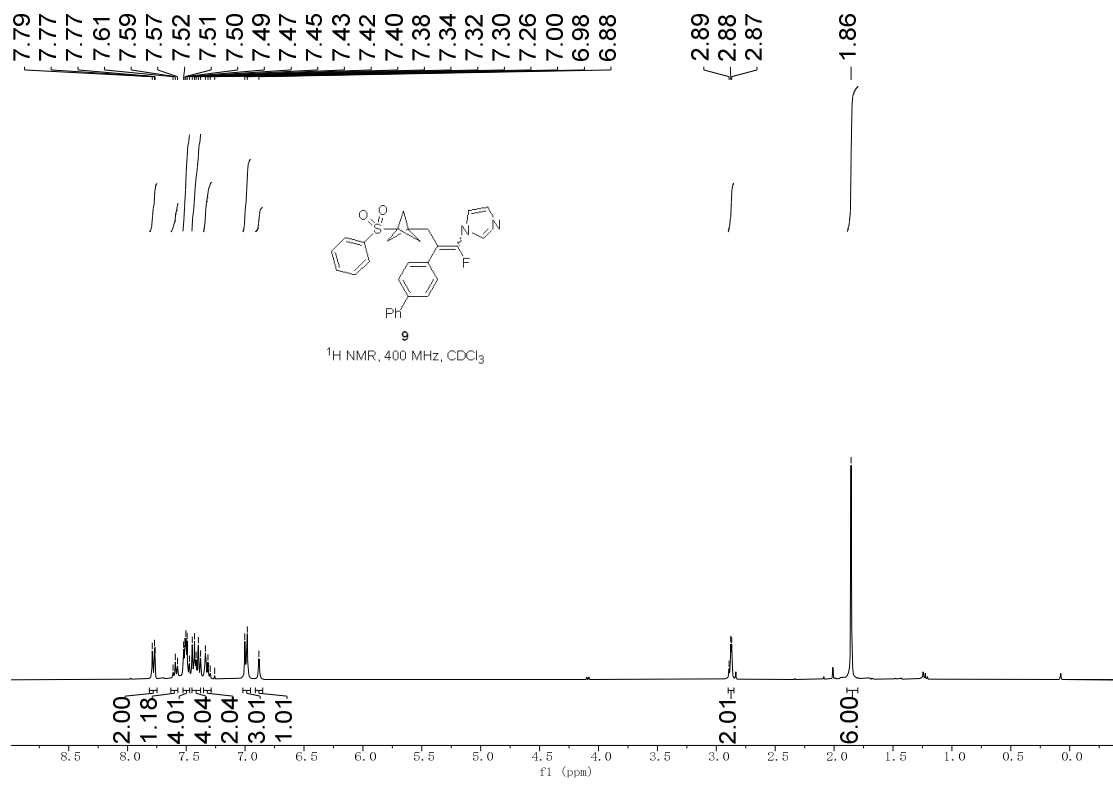
77.32  
77.00  
76.68

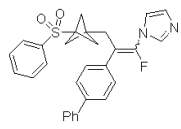
51.47  
50.92  
40.61  
38.06  
34.90



<sup>13</sup>C NMR, 100 MHz, CDCl<sub>3</sub>

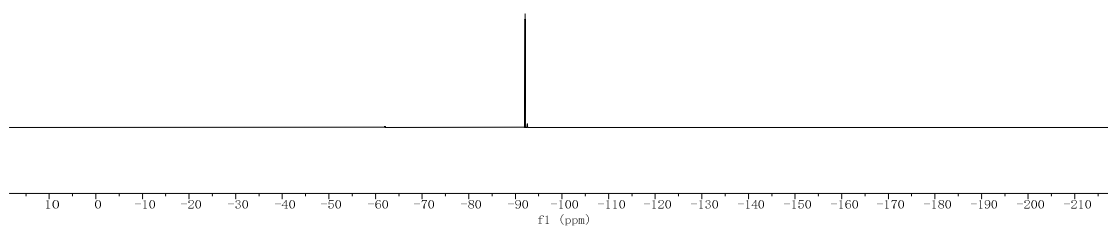




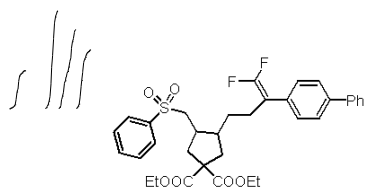


9  
<sup>19</sup>F NMR, 376 MHz, CDCl<sub>3</sub>

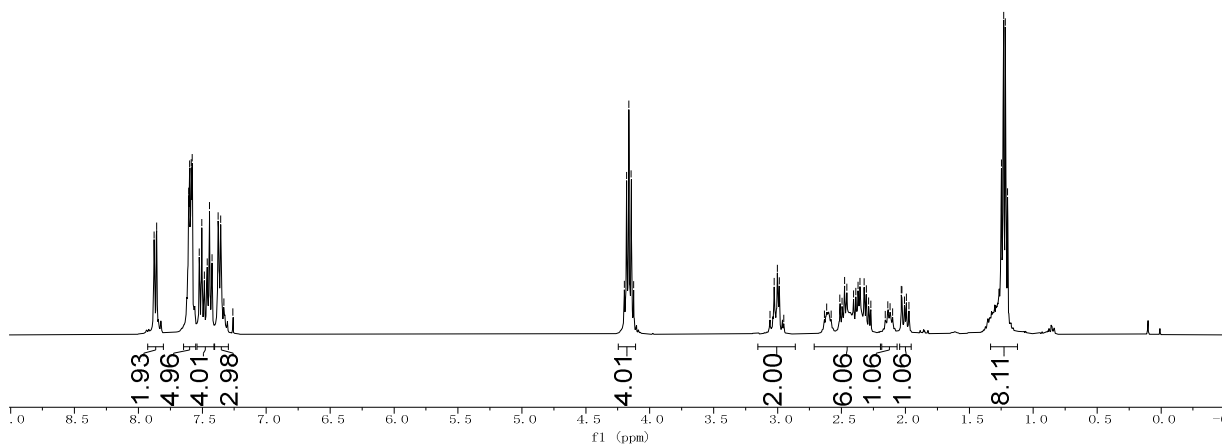
--92.08



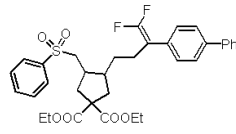
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 7.52  
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 7.48  
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 7.44  
 7.42  
 7.38  
 7.36  
 4.20  
 4.18  
 4.16  
 4.14  
 4.13  
 3.03  
 3.00  
 2.99  
 2.51  
 2.49  
 2.48  
 2.46  
 2.40  
 2.39  
 2.37  
 2.35  
 2.32  
 2.31  
 2.29  
 2.03  
 2.03  
 2.01  
 1.99  
 1.25  
 1.23  
 1.22  
 1.20



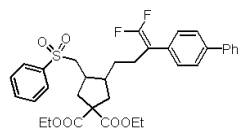
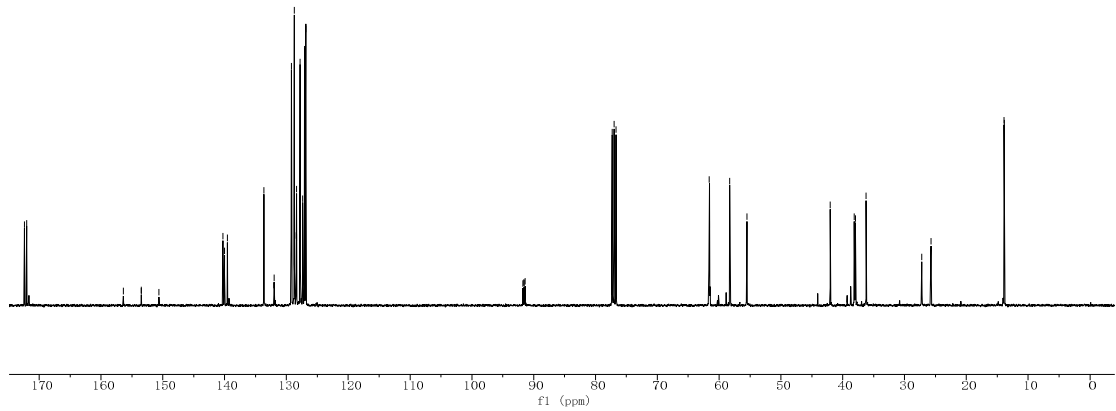
12  
<sup>1</sup>H NMR, 400MHz, CDCl<sub>3</sub>



172.41  
172.04  
156.36  
153.50  
153.46  
150.61  
140.27  
140.02  
139.54  
133.63  
132.02  
131.98  
131.95  
129.21  
128.72  
128.41  
128.38  
128.34  
127.80  
127.36  
127.08  
126.86  
91.73  
91.60  
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91.39  
77.32  
77.00  
76.68  
61.62  
61.54  
58.28  
55.51  
42.01  
38.13  
37.96  
36.20  
27.22  
25.74  
13.89



<sup>13</sup>C NMR, 100MHz, CDCl<sub>3</sub>



<sup>19</sup>F NMR, 376MHz, CDCl<sub>3</sub>

