

## Supporting Information

For

### Remote Azidation of C(sp<sup>3</sup>)-H Bonds to Synthesize $\delta$ -Azido Sulfonamides *via* Iron-catalyzed Radical Relay

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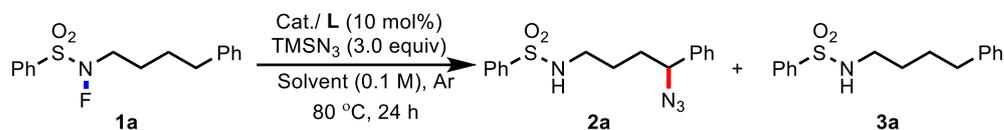
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## **I. General Information:**

NMR spectra were recorded on Bruker-400 and Bruker-500 (400 MHz for  $^1\text{H}$  (Bruker-400); 500 MHz for  $^1\text{H}$  (Bruker-500), 126 MHz for  $^{13}\text{C}$  (Bruker-500), and 376 MHz for  $^{19}\text{F}$  (Bruker-400)) instruments internally referenced to  $\text{SiMe}_4$  signal. High resolution mass spectra were recorded on P-SIMS-Gly of Bruker Daltonics Inc. using ESI-TOF (electrospray ionization-time of flight) or Micromass GCT using EI (electron impact). Catalyst,  $\text{TMSN}_3$ , and solvent were purchased from J&K etc. and used as received.

## II. Optimization of conditions

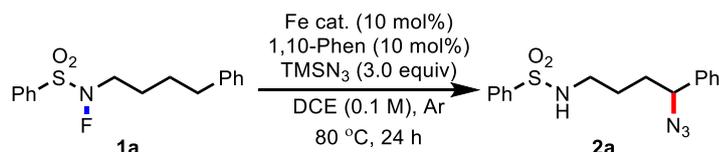
### Catalyst screening



Entry	Cat.	2a <sup>[b]</sup>	3a <sup>[b]</sup>
1	$\text{Cu}(\text{MeCN})_4\text{PF}_6$	75%	21%
2	$\text{CuCN}$	78%	15%
3	$\text{CuSCN}$	84%	20%
4	$\text{CuI}$	N.D.	~50%
5	$\text{Cu}(\text{OAc})_2$	77%	14%
6	$\text{Cu}(\text{OTf})_2$	trace	trace

[a] Reaction conditions: **1a** (0.1 mmol, 1.0 equiv), Cu cat. (10 mol%), 1,10-Phen (12 mol%),  $\text{TMSN}_3$  (3.0 equiv), DCE (0.1 M), Ar, 80 °C, 24 h.

[b] Yields detected by crude  $^1\text{H}$  NMR with  $\text{CH}_2\text{Br}_2$  as internal standard.

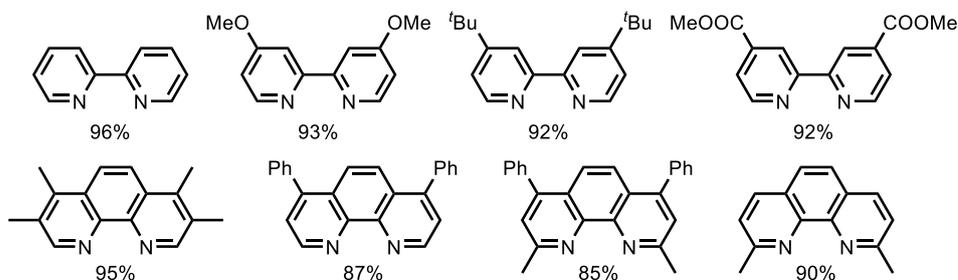
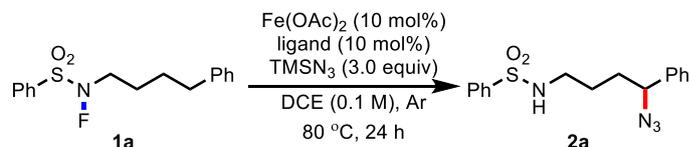


Entry	Fe cat.	2a <sup>[b]</sup>
1	$\text{FeF}_2$	57%
2	$\text{FeCl}_2$	38%
3	$\text{Fe}(\text{OTf})_2$	85%
4	$\text{Fe}(\text{acac})_3$	91%

[a] Reaction conditions: **1a** (0.1 mmol, 1.0 equiv), Fe cat. (10 mol%), 1,10-Phen (10 mol%),  $\text{TMSN}_3$  (3.0 equiv), DCE (0.1 M), Ar, 80 °C, 24 h.

[b] Yields detected by crude  $^1\text{H}$  NMR with  $\text{CH}_2\text{Br}_2$  as internal standard.

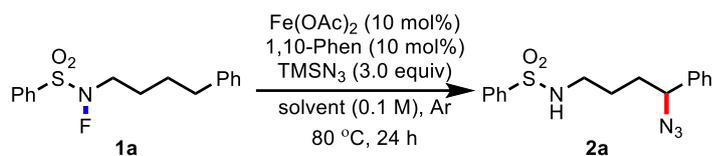
### Ligand screening



[a] Reaction conditions: **1a** (0.1 mmol, 1.0 equiv),  $\text{Fe}(\text{OAc})_2$  (10 mol%), ligand (10 mol%),  $\text{TMSN}_3$  (3.0 equiv), DCE (0.1 M), Ar, 80 °C, 24 h.

[b] Yields detected by crude  $^1\text{H}$  NMR with  $\text{CH}_2\text{Br}_2$  as internal standard.

## Solvent screening



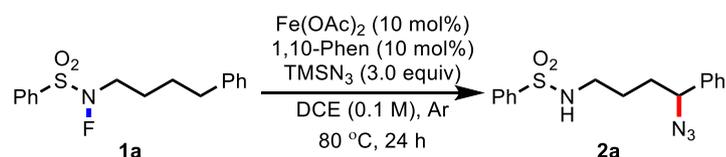
Entry	solvent	2a <sup>[b]</sup>
1	MeCN	89%
2	DCE	99% (94%) <sup>[c]</sup>
3	1,4-dioxane	81%
4	PhCl	87%
5	$\text{PhCF}_3$	85%

[a] Reaction conditions: **1a** (0.1 mmol, 1.0 equiv),  $\text{Fe}(\text{OAc})_2$  (10 mol%), 1,10-Phen (10 mol%),  $\text{TMSN}_3$  (3.0 equiv), solvent (0.1 M), Ar, 80 °C, 24 h.

[b] Yields detected by crude  $^1\text{H}$  NMR with  $\text{CH}_2\text{Br}_2$  as internal standard.

[c] Isolated yields.

## Other Variables



Entry	deviation from standard condition	2a <sup>[b]</sup>
1	$\text{TMSN}_3$ (2.0 equiv)	81%
2	60 °C	93%
3	w/o Fe/L	0%
4	w/o L	56%
5	under air	29%

[a] Reaction conditions: **1a** (0.1 mmol, 1.0 equiv),  $\text{Fe}(\text{OAc})_2$  (10 mol%), 1,10-Phen (10 mol%),  $\text{TMSN}_3$  (3.0 equiv), DCE (0.1 M), Ar, 80 °C, 24 h.

[b] Yields detected by crude  $^1\text{H}$  NMR with  $\text{CH}_2\text{Br}_2$  as internal standard.

### III. Experimental procedures and data

#### Synthesis of Products

##### General Procedure A – Iron-catalyzed Remote Azidation

Fe(OAc)<sub>2</sub> (1.7 mg, 0.01 mmol), 1,10-phenanthroline (1.8mg, 0.01mmol) were combined in a 25 mL oven-dried sealed tube. The vessel was evacuated and backfilled with N<sub>2</sub> (repeated for 3 times), after that, substrate **1** (0.1 mmol), TMSN<sub>3</sub> (0.3 mmol, 3.0 equiv) and DCE (1.0 mL) were then added via syringe under N<sub>2</sub>. The tube was sealed with a Teflon lined cap and moved into a preheated oil bath at 80 °C for 24 h. The reaction mixture was then cooled to room temperature, diluted with EtOAc (10 mL) and filtered through a pad of celite. The filtrate was concentrated, and the residue was then purified by flash column chromatography to give **2a-2x**.

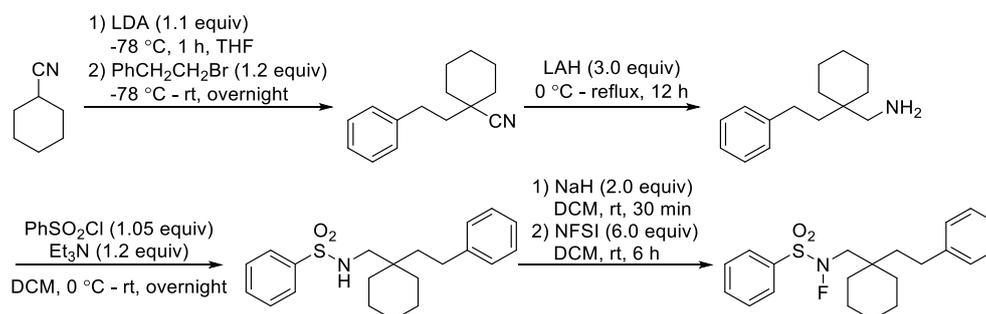
##### General Procedure B – Derivatization-Click Reaction<sup>2</sup>

CuI (2 equiv) was added in a 25 mL oven-dried sealed tube. The vessel was evacuated and backfilled with N<sub>2</sub> (repeated for 3 times), after that, product **2a** (0.1 mmol) dissolved in CH<sub>3</sub>CN (0.05 M), DIPEA (3 equiv), phenylacetylene (1.1 equiv) were then added via syringe under N<sub>2</sub>. The tube was sealed with a Teflon lined cap and moved into a preheated oil bath at RT for 2h. The reaction mixture was then cooled to room temperature, diluted with EtOAc (10 mL) and filtered through a pad of celite. The filtrate was concentrated, and the residue was then purified by flash column chromatography to give **4**.

#### Synthesis of Starting Materials

All known starting materials were synthesized through the method reported in Ref. S1

##### General Procedure C (for the synthesis of **1g**, **1h**)



Step 1 : To solution of cyclohexanecarbonitrile (1.0 equiv) in THF (0.25 M) was added LDA (1.1 equiv) dropwise under -78 °C and the mixture was allowed to stir for 1 h at room temperature. The solution was cooled to -78 °C again and added with (2-bromoethyl)benzene (1.2 equiv). Then, the mixture was warmed to room temperature and stirred overnight. Until completion, the reaction was quenched with saturated NH<sub>4</sub>Cl (aq). The reaction mixture was then cooled to room

temperature, extracted with DCM three times. The combined organic layer was washed with brine, dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated. The residue was then purified by column chromatography to afford the product.

Step 2 : To a mixture of LAH (3.0 equiv) dispersed in THF (0.25 M) was added the solution the product (1.0 equiv) obtained from step 1 under  $0\text{ }^\circ\text{C}$  and the mixture then transferred to oil bath and refluxed overnight. Until completion, the mixture was quenched with water and 10% NaOH (aq) in sequence and the slurry was added with anhydrous  $\text{Na}_2\text{SO}_4$  and filtered over a pad of celite. The filtrate was collected and concentrated under vacuum to afford the crude product which can be used directly without further purification.

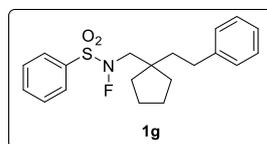
Step 3 : To a solution of amine (1.0 equiv) obtained in step 2 and  $\text{Et}_3\text{N}$  (1.2 equiv) in DCM (0.2 M) was slowly added with benzenesulfonyl chloride (1.05 equiv) under  $0\text{ }^\circ\text{C}$ . The mixture was allowed to warm to room temperature and stirred overnight. . Until completion, the reaction was diluted with DCM and the organic layer was washed with aqueous HCl (1N) and brine in sequence; the organic layer was dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated. The residue was then purified by column chromatography to afford the product.

Step 4 : To a stirred suspension of NaH (6 mmol, 60 wt% in mineral oil) in anhydrous  $\text{CH}_2\text{Cl}_2$  (24 mL) in a 100 mL round-bottomed flask was slowly added a solution of sulfonamide obtained in step 3 (3 mmol) in anhydrous  $\text{CH}_2\text{Cl}_2$  (6 mL) at room temperature under an  $\text{N}_2$  atmosphere. After the mixture was stirred for 30 min, N-fluorobenzenesulfonimide (NFSI, 5.67 g, 18 mmol) was added in one portion and allowed to stir for another 6 h. Until completion, the reaction was quenched by the addition of water. The mixture was extracted with DCM ( $3 \times 30\text{ mL}$ ) and the organic layers were combined, washed with brine, and dried over anhydrous  $\text{Na}_2\text{SO}_4$ . The crude mixture was filtered through celite and concentrated. The resulting residue was purified by column chromatography on silica gel with a gradient eluent of petroleum ether and ethyl acetate.

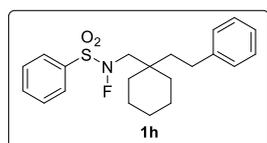
## Analytical data for compounds

### 1. Substrates:

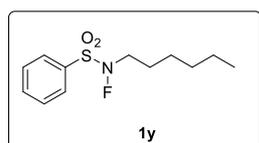
Most substrates data were reported in previous work<sup>1</sup>. New substrates are shown as follow:



N-fluoro-N-((1-phenethylcyclopentyl)methyl)benzenesulfonamide was prepared following general procedure C and was purified by column chromatography with petroleum ether and ethyl acetate (PE/EA = 19:1) to afford the product **1g** (68% yield) as light yellow oil. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 7.6 Hz, 2H), 7.75 (t, *J* = 7.5 Hz, 1H), 7.63 (t, *J* = 7.8 Hz, 2H), 7.27 (d, *J* = 7.8 Hz, 2H), 7.17 (d, *J* = 6.8 Hz, 3H), 3.30 (s, 1H), 3.19 (s, 1H), 2.64 – 2.45 (m, 2H), 1.82 – 1.74 (m, 2H), 1.71 – 1.51 (m, 8H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 142.83, 134.96, 132.79, 129.94, 129.45, 128.49, 128.49, 125.83, 59.44 (d, *J* = 10.6 Hz), 46.12, 39.99, 36.32, 31.09, 24.46. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -40.78 (t, *J* = 44.0 Hz). HRMS (ESI) (*m/z*): [M+Na]<sup>+</sup> calcd. for C<sub>20</sub>H<sub>24</sub>NO<sub>2</sub>SFNa: 384.1404, found: 384.1414.

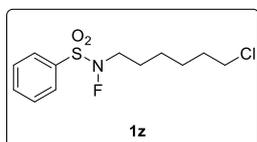


N-fluoro-N-((1-phenethylcyclohexyl)methyl)benzenesulfonamide was prepared following general procedure C and was purified by column chromatography with petroleum ether and ethyl acetate (PE/EA = 19:1) to afford the product **1h** (64% yield) as light yellow oil. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.98 (d, *J* = 7.7 Hz, 2H), 7.75 (t, *J* = 7.5 Hz, 1H), 7.63 (t, *J* = 7.8 Hz, 2H), 7.26 (d, *J* = 7.0 Hz, 2H), 7.18 (d, *J* = 7.5 Hz, 3H), 3.30 (s, 1H), 3.19 (s, 1H), 2.61 – 2.37 (m, 2H), 1.86 – 1.66 (m, 2H), 1.50 – 1.36 (m, 10H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 143.03, 134.95, 132.88, 129.92, 129.45, 128.55, 128.49, 125.81, 59.49 (d, *J* = 9.9 Hz), 38.22, 36.77, 34.13, 29.39, 26.08, 21.43. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -36.19 (t, *J* = 42.6 Hz). HRMS (ESI) (*m/z*): [M+Na]<sup>+</sup> calcd. for C<sub>21</sub>H<sub>26</sub>NO<sub>2</sub>SFNa: 398.1560, found: 398.1569.

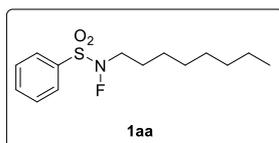


N-fluoro-N-hexylbenzenesulfonamide was prepared following general procedure C and was purified by column chromatography with petroleum ether and ethyl acetate (PE/EA = 19:1) to afford the product **1y** (68% yield) as light yellow oil. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 7.6 Hz, 2H), 7.75 (t, *J* = 7.5 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 2H), 3.27 (t, *J* = 7.0 Hz, 1H), 3.17 (t, *J* = 6.9 Hz, 1H), 1.71 (p, *J* = 7.2 Hz, 2H), 1.48 – 1.28 (m, 6H), 0.88 (t, *J* = 6.0 Hz, 3H). <sup>13</sup>C NMR (101 MHz,

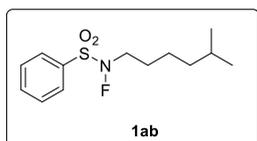
$\text{CDCl}_3$ )  $\delta$  134.99, 132.21, 130.08, 129.41, 53.82 (d,  $J = 12.7$  Hz), 31.40, 26.37, 22.58, 14.10.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -50.06 (t,  $J = 40.7$  Hz). HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd. for  $\text{C}_{12}\text{H}_{18}\text{NO}_2\text{SFNa}$ : 282.0934, found: 282.0941.



**1z** N-(6-chlorohexyl)-N-fluorobenzenesulfonamide was prepared following general procedure C and was purified by column chromatography with petroleum ether and ethyl acetate (PE/EA = 19:1) to afford the product **1z** (69% yield) as light yellow oil.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.95 (d,  $J = 7.6$  Hz, 2H), 7.75 (t,  $J = 7.5$  Hz, 1H), 7.63 (t,  $J = 7.8$  Hz, 2H), 3.53 (t,  $J = 6.6$  Hz, 2H), 3.29 (t,  $J = 6.9$  Hz, 1H), 3.19 (t,  $J = 6.9$  Hz, 1H), 1.82 – 1.70 (m, 4H), 1.54 – 1.40 (m, 4H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  135.05, 132.17, 130.08, 129.44, 53.59 (d,  $J = 12.6$  Hz), 44.99, 32.44, 26.48, 26.26, 25.98.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -49.90 (t,  $J = 40.4$  Hz). HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd. for  $\text{C}_{12}\text{H}_{17}\text{NO}_2\text{SFCINa}$ : 316.0545, found: 316.0549.

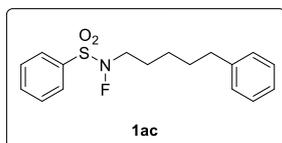


**1aa** N-fluoro-N-octylbenzenesulfonamide was prepared following general procedure C and was purified by column chromatography with petroleum ether and ethyl acetate (PE/EA = 19:1) to afford the product **1aa** (69% yield) as light yellow oil.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.95 (d,  $J = 7.4$  Hz, 2H), 7.75 (t,  $J = 7.5$  Hz, 1H), 7.62 (t,  $J = 7.8$  Hz, 2H), 3.27 (t,  $J = 7.0$  Hz, 1H), 3.17 (t,  $J = 7.0$  Hz, 1H), 1.71 (p,  $J = 7.3$  Hz, 2H), 1.44 – 1.35 (m, 2H), 1.33-1.23 (m, 8H), 0.87 (t,  $J = 6.8$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  134.98, 130.09, 129.41, 53.81 (d,  $J = 12.5$  Hz), 31.85, 29.19, 26.70, 26.42, 22.74, 14.21.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -49.99 (t,  $J = 40.6$  Hz). HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd. for  $\text{C}_{14}\text{H}_{22}\text{NO}_2\text{SFNa}$ : 310.1247, found: 310.1254.



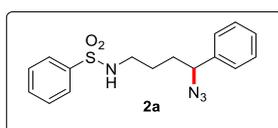
**1ab** N-fluoro-N-(5-methylhexyl)benzenesulfonamide was prepared following general procedure C and was purified by column chromatography with petroleum ether and ethyl acetate (PE/EA = 19:1) to afford the product **1ab** (66% yield) as light yellow oil.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.96 (d,  $J = 7.7$  Hz, 2H), 7.75 (t,  $J = 7.5$  Hz, 1H), 7.63 (t,  $J = 7.8$  Hz, 2H), 3.27 (t,  $J = 7.0$  Hz, 1H), 3.17 (t,  $J = 7.0$  Hz, 1H), 1.70 (p,  $J = 7.4$  Hz, 2H), 1.56 – 1.50 (m, 1H), 1.40 (q,  $J = 7.9$  Hz, 2H), 1.23 – 1.15 (m, 2H), 0.86 (d,  $J = 6.6$  Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  134.99, 132.22, 130.09, 129.42, 53.87

(d,  $J = 12.4$  Hz), 38.51, 27.96, 26.66, 24.52, 22.65.  $^{19}\text{F}$  NMR (376 MHz, Chloroform- $d$ )  $\delta$  -50.04 (t,  $J = 40.6$  Hz). HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd. for  $\text{C}_{13}\text{H}_{20}\text{NO}_2\text{SFNa}$ : 296.1091, found: 296.1094.

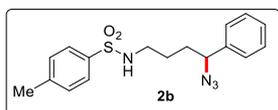


N-fluoro-N-(5-phenylpentyl)benzenesulfonamide was prepared following general procedure C and was purified by column chromatography with petroleum ether and ethyl acetate (PE/EA = 19:1) to afford the product **1ac** (66% yield) as light yellow oil.  $^1\text{H}$  NMR (500 MHz, Chloroform- $d$ )  $\delta$  7.98 – 7.91 (m, 2H), 7.74 (t,  $J = 7.5$  Hz, 1H), 7.61 (t,  $J = 7.9$  Hz, 2H), 7.29-7.27 (m, 2H), 7.21 – 7.13 (m, 3H), 3.26 (t,  $J = 7.0$  Hz, 1H), 3.18 (t,  $J = 7.0$  Hz, 1H), 2.70 – 2.56 (m, 2H), 1.75 (p,  $J = 7.3$  Hz, 2H), 1.64 (p,  $J = 7.7$  Hz, 2H), 1.44 (p,  $J = 7.7, 7.2$  Hz, 2H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  142.24, 134.87, 132.13, 129.95, 129.29, 128.39, 128.34, 125.78, 53.54 (d,  $J = 12.5$  Hz), 35.68, 30.90, 26.19.  $^{19}\text{F}$  NMR (376 MHz, Chloroform- $d$ )  $\delta$  -49.88 (t,  $J = 40.6$  Hz). HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd. for  $\text{C}_{17}\text{H}_{20}\text{NO}_2\text{SFNa}$ : 344.1091, found: 344.1102.

## 2. Products:

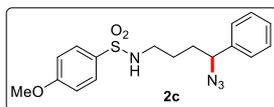


N-(4-azido-4-phenylbutyl)benzenesulfonamide was prepared following general procedure A the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2a** (92% yield) as a light yellow liquid.  $^1\text{H}$  NMR (500 MHz, Chloroform- $d$ )  $\delta$  7.88 – 7.81 (m, 2H), 7.61 – 7.55 (m, 1H), 7.53 – 7.48 (m, 2H), 7.39 – 7.30 (m, 3H), 7.25 – 7.21 (m, 2H), 4.60 (t,  $J = 6.2$  Hz, 1H), 4.37 (dd,  $J = 7.9, 6.3$  Hz, 1H), 2.97 (qd,  $J = 6.7, 3.3$  Hz, 2H), 1.85 – 1.68 (m, 2H), 1.60 – 1.53 (m, 1H), 1.50 – 1.43 (m, 1H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  139.82, 139.15, 132.73, 129.18, 128.90, 128.43, 127.01, 126.80, 65.70, 42.74, 33.13, 26.36. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{H}-\text{N}_2]^+$  calcd. for  $\text{C}_{16}\text{H}_{19}\text{N}_2\text{O}_2\text{S}$ : 303.1162, found: 303.1161.

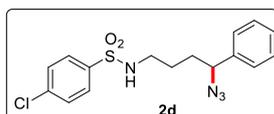


N-(4-azido-4-phenylbutyl)-4-methylbenzenesulfonamide was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2b** (95% yield) as a light yellow liquid.  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  7.78 – 7.69 (m, 2H), 7.38 – 7.26 (m, 5H), 7.25 – 7.19 (m, 2H), 4.78 (t,  $J = 6.2$  Hz, 1H), 4.35 (dd,  $J = 7.9, 6.4$  Hz, 1H), 2.93 (qd,  $J = 6.8, 1.9$  Hz, 2H), 2.42 (s, 3H), 1.81 – 1.66 (m, 2H), 1.62 – 1.40 (m, 2H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  143.43, 139.13, 136.74, 129.69, 128.78, 128.30, 127.00, 126.73, 65.63, 42.60,

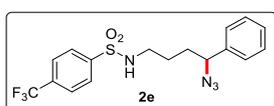
33.02, 26.22, 21.47. HRMS (ESI) ( $m/z$ ):  $[M+H-N_2]^+$  calcd. for  $C_{17}H_{21}N_2O_2S$ : 317.1318, found: 317.1331.



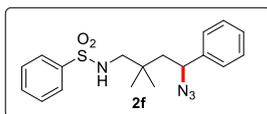
N-(4-azido-4-phenylbutyl)-4-methoxybenzenesulfonamide was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 5:1) to afford the product **2c** (85% yield) as a light yellow liquid.  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.81 – 7.74 (m, 2H), 7.39 – 7.28 (m, 3H), 7.25 – 7.19 (m, 2H), 6.99 – 6.92 (m, 2H), 4.75 (t,  $J$  = 6.2 Hz, 1H), 4.36 (dd,  $J$  = 7.8, 6.4 Hz, 1H), 3.86 (s, 3H), 2.92 (qd,  $J$  = 6.7, 1.6 Hz, 2H), 1.82 – 1.67 (m, 2H), 1.62 – 1.39 (m, 2H).  $^{13}C$  NMR (126 MHz,  $CDCl_3$ )  $\delta$  162.83, 139.12, 129.11, 128.78, 128.30, 126.73, 114.22, 65.63, 55.56, 42.57, 33.04, 26.19. HRMS (ESI) ( $m/z$ ):  $[M+H-N_2]^+$  calcd. for  $C_{17}H_{21}N_2O_3S$ : 333.1268, found: 333.1265.



N-(4-azido-4-phenylbutyl)-4-chlorobenzenesulfonamide was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2d** (84% yield) as a light yellow liquid.  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.83 – 7.71 (m, 2H), 7.51 – 7.43 (m, 2H), 7.41 – 7.29 (m, 3H), 7.25 – 7.20 (m, 2H), 4.81 (t,  $J$  = 5.2 Hz, 1H), 4.38 (dd,  $J$  = 7.8, 6.3 Hz, 1H), 2.95 (q,  $J$  = 5.7 Hz, 2H), 1.83 – 1.68 (m, 2H), 1.63 – 1.40 (m, 2H).  $^{13}C$  NMR (126 MHz,  $CDCl_3$ )  $\delta$  139.17, 139.03, 138.33, 129.42, 128.85, 128.42, 128.40, 126.71, 65.61, 42.69, 33.06, 26.27. HRMS (ESI) ( $m/z$ ):  $[M+H-N_2]^+$  calcd. for  $C_{16}H_{18}N_2O_2S$ Cl: 337.0773, found: 337.0792.

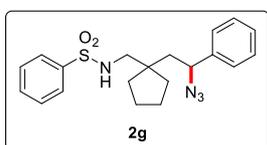


N-(4-azido-4-phenylbutyl)-4-(trifluoromethyl)benzenesulfonamide was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2e** (95% yield) as a light yellow liquid.  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.05 – 7.93 (m, 2H), 7.85 – 7.72 (m, 2H), 7.43 – 7.29 (m, 3H), 7.25 – 7.18 (m, 2H), 4.97 (t,  $J$  = 6.3 Hz, 1H), 4.38 (dd,  $J$  = 7.9, 6.2 Hz, 1H), 3.12 – 2.89 (m, 2H), 1.85 – 1.67 (m, 2H), 1.65 – 1.41 (m, 2H).  $^{13}C$  NMR (126 MHz,  $CDCl_3$ )  $\delta$  143.44, 139.00, 134.38 (q,  $J$  = 33.1 Hz), 128.86, 128.43, 127.47, 126.70, 126.31 (q,  $J$  = 3.7 Hz), 123.26 (q,  $J$  = 272.9 Hz), 65.60, 42.76, 33.04, 26.32.  $^{19}F$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -63.09. HRMS (ESI) ( $m/z$ ):  $[M+H-N_2]^+$  calcd. for  $C_{17}H_{18}N_2O_2SF_3$ : 371.1036, found: 371.1036.



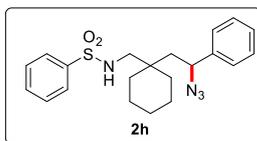
N-(4-azido-2,2-dimethyl-4-phenylbutyl)benzenesulfonamide was

prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2f** (94% yield) as a light yellow liquid.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.92 – 7.78 (m, 2H), 7.60 – 7.54 (m, 1H), 7.54 – 7.47 (m, 2H), 7.39 – 7.29 (m, 3H), 7.28 – 7.23 (m, 2H), 5.09 (t,  $J$  = 7.2 Hz, 1H), 4.47 (dd,  $J$  = 9.7, 3.3 Hz, 1H), 2.81 – 2.66 (m, 2H), 1.81 (dd,  $J$  = 14.9, 9.7 Hz, 1H), 1.51 (dd,  $J$  = 14.9, 3.4 Hz, 1H), 0.95 (s, 3H), 0.94 (s, 3H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.18, 139.82, 132.53, 129.07, 128.95, 128.39, 126.87, 126.66, 62.79, 52.30, 45.00, 33.92, 26.48, 25.23. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{H}-\text{N}_2]^+$  calcd. for  $\text{C}_{18}\text{H}_{23}\text{N}_2\text{O}_2\text{S}$ : 331.1475, found: 331.1485.



N-((1-(2-azido-2-phenylethyl)cyclopentyl)methyl)benzenesulfonamide

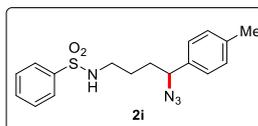
was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2g** (94% yield) as a light yellow liquid.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.92 – 7.82 (m, 2H), 7.61 – 7.56 (m, 1H), 7.55 – 7.49 (m, 2H), 7.40 – 7.29 (m, 3H), 7.28 – 7.22 (m, 2H), 5.20 – 5.04 (m, 1H), 4.44 (dd,  $J$  = 9.8, 2.9 Hz, 1H), 2.87 – 2.75 (m, 2H), 1.95 – 1.88 (m, 1H), 1.72 – 1.52 (m, 6H), 1.41 – 1.31 (m, 3H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.19, 139.74, 132.55, 129.09, 128.99, 128.42, 126.88, 126.61, 63.83, 49.07, 45.66, 43.97, 36.92, 35.81, 24.29, 24.15. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{H}-\text{N}_2]^+$  calcd. for  $\text{C}_{20}\text{H}_{25}\text{N}_2\text{O}_2\text{S}$ : 357.1632, found: 357.1614.



N-((1-(2-azido-2-phenylethyl)cyclohexyl)methyl)benzenesulfonamide was

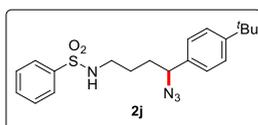
prepared following general procedure and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2h** (88% yield) as a light yellow liquid.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.91 – 7.84 (m, 2H), 7.61 – 7.49 (m, 3H), 7.41 – 7.29 (m, 3H), 7.27 (d,  $J$  = 1.7 Hz, 1H), 7.25 (q,  $J$  = 2.5, 2.0 Hz, 1H), 5.08 (dd,  $J$  = 9.3, 5.2 Hz, 1H), 4.50 (dd,  $J$  = 10.0, 2.7 Hz, 1H), 3.00 (dd,  $J$  = 12.8, 9.4 Hz, 1H), 2.75 (dd,  $J$  = 12.8, 5.2 Hz, 1H), 1.77 (dd,  $J$  = 15.3, 10.0 Hz, 1H), 1.57 (dd,  $J$  = 15.3, 2.6 Hz, 1H), 1.49 – 1.21 (m, 10H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.54, 140.01, 132.65, 129.23, 129.16, 128.55, 127.03, 126.71, 62.20, 48.80, 42.93, 36.12, 34.77, 33.68, 26.06, 21.33, 21.11. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{H}-\text{N}_2]^+$  calcd. for  $\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}_2\text{S}$ :

371.1788, found: 371.1736.



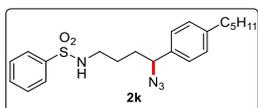
N-(4-azido-4-(p-tolyl)butyl)benzenesulfonamide was prepared following

general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2i** (85% yield) as a light yellow liquid. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.90 – 7.78 (m, 2H), 7.61 – 7.54 (m, 1H), 7.50 (ddt, *J* = 8.3, 6.8, 1.4 Hz, 2H), 7.20 – 7.07 (m, 4H), 4.79 (t, *J* = 6.2 Hz, 1H), 4.32 (dd, *J* = 7.8, 6.5 Hz, 1H), 2.95 (qd, *J* = 6.8, 1.5 Hz, 2H), 2.34 (s, 3H), 1.83 – 1.61 (m, 2H), 1.61 – 1.38 (m, 2H). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 139.76, 138.13, 136.00, 132.63, 129.47, 129.10, 126.94, 126.68, 65.44, 42.67, 32.95, 26.30, 21.09. HRMS (ESI) (*m/z*): [M+H-N<sub>2</sub>]<sup>+</sup> calcd. for C<sub>17</sub>H<sub>21</sub>N<sub>2</sub>O<sub>2</sub>S: 317.1319, found: 317.1331.



N-(4-azido-4-(4-(tert-butyl)phenyl)butyl)benzenesulfonamide was

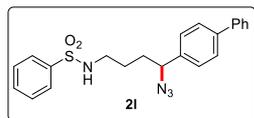
prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2j** (93% yield) as a light yellow liquid. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.90 – 7.81 (m, 2H), 7.60 – 7.54 (m, 1H), 7.53 – 7.48 (m, 2H), 7.40 – 7.33 (m, 2H), 7.18 – 7.12 (m, 2H), 4.80 (s, 1H), 4.33 (dd, *J* = 7.9, 6.3 Hz, 1H), 3.06 – 2.86 (m, 2H), 1.84 – 1.64 (m, 2H), 1.63 – 1.38 (m, 2H), 1.31 (s, 9H). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 151.25, 139.77, 136.05, 132.63, 129.10, 126.95, 126.39, 125.67, 65.38, 42.69, 34.53, 32.99, 31.24, 26.32. HRMS (ESI) (*m/z*): [M+Na]<sup>+</sup> calcd. for C<sub>20</sub>H<sub>26</sub>N<sub>4</sub>O<sub>2</sub>SNa: 409.1669, found: 409.1656.



N-(4-azido-4-(4-pentylphenyl)butyl)benzenesulfonamide was prepared

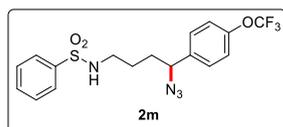
following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2k** (89% yield) as a light yellow liquid. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.90 – 7.81 (m, 2H), 7.61 – 7.53 (m, 1H), 7.53 – 7.46 (m, 2H), 7.14 (q, *J* = 8.3 Hz, 4H), 4.85 (t, *J* = 6.2 Hz, 1H), 4.32 (dd, *J* = 7.9, 6.4 Hz, 1H), 3.07 – 2.85 (m, 2H), 2.67 – 2.50 (m, 2H), 1.84 – 1.51 (m, 5H), 1.50 – 1.40 (m, 1H), 1.35 – 1.25 (m, 4H), 0.89 (t, *J* = 6.9 Hz, 3H). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 143.31, 139.92, 136.39, 132.78, 129.25, 128.93, 127.10, 126.79, 65.64, 42.83, 35.70, 33.13, 31.62, 31.15, 26.46, 22.63, 14.14. HRMS (ESI)

(*m/z*): [M+H-N<sub>2</sub>]<sup>+</sup> calcd. for C<sub>21</sub>H<sub>29</sub>N<sub>2</sub>O<sub>2</sub>S: 373.1945, found: 373.1950.



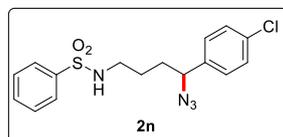
N-(4-([1,1'-biphenyl]-4-yl)-4-azidobutyl)benzenesulfonamide was

prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 5:1) to afford the product **2l** (89% yield) as a light yellow liquid. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.90 – 7.81 (m, 2H), 7.60 – 7.52 (m, 5H), 7.51 – 7.41 (m, 4H), 7.39 – 7.32 (m, 1H), 7.32 – 7.26 (m, 2H), 4.89 (t, *J* = 6.1 Hz, 1H), 4.40 (dd, *J* = 7.7, 6.5 Hz, 1H), 2.96 (qd, *J* = 6.7, 1.9 Hz, 2H), 1.88 – 1.70 (m, 2H), 1.65 – 1.41 (m, 2H). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 141.34, 140.51, 139.90, 138.24, 132.80, 129.26, 128.95, 127.64, 127.34, 127.17, 127.09, 65.51, 42.81, 33.18, 26.41. HRMS (ESI) (*m/z*): [M+H-N<sub>2</sub>]<sup>+</sup> calcd. for C<sub>22</sub>H<sub>23</sub>N<sub>2</sub>O<sub>2</sub>S: 379.1475, found: 379.1466.



N-(4-azido-4-(4-(trifluoromethoxy)phenyl)butyl)benzenesulfonamide

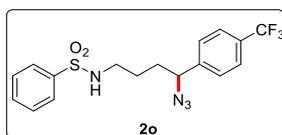
was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2m** (87% yield) as a light yellow liquid. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.89 – 7.81 (m, 2H), 7.61 – 7.55 (m, 1H), 7.54 – 7.47 (m, 2H), 7.30 – 7.25 (m, 2H), 7.23 – 7.16 (m, 2H), 4.89 (t, *J* = 6.2 Hz, 1H), 4.41 (dd, *J* = 7.9, 6.2 Hz, 1H), 2.97 (qd, *J* = 6.7, 2.8 Hz, 2H), 1.83 – 1.65 (m, 2H), 1.60 – 1.42 (m, 2H). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 148.94, 139.73, 137.99, 132.71, 129.14, 128.18, 126.93, 121.24, 120.37 (q, *J* = 257.79 Hz), 64.82, 42.56, 33.18, 26.16. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -57.83. HRMS (ESI) (*m/z*): [M+H-N<sub>2</sub>]<sup>+</sup> calcd. for C<sub>17</sub>H<sub>18</sub>N<sub>2</sub>O<sub>3</sub>SF<sub>3</sub>: 387.0985, found: 387.0986.



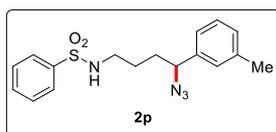
N-(4-azido-4-(4-chlorophenyl)butyl)benzenesulfonamide was prepared

following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2n** (91% yield) as a light yellow liquid. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.91 – 7.80 (m, 2H), 7.61 – 7.53 (m, 1H), 7.54 – 7.47 (m, 2H), 7.34 – 7.29 (m, 2H), 7.20 – 7.13 (m, 2H), 5.02 (t, *J* = 6.2 Hz, 1H), 4.36 (dd, *J* = 7.8, 6.3 Hz, 1H), 2.94 (qd, *J* = 6.7, 2.4 Hz, 2H), 1.84 – 1.59 (m, 2H), 1.62 – 1.37 (m, 2H). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 139.68, 137.69, 134.04, 132.68, 129.12, 128.98, 128.10, 126.89, 64.87,

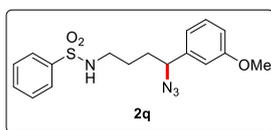
42.53, 33.00, 26.10. HRMS (ESI) ( $m/z$ ):  $[M+H-N_2]^+$  calcd. for  $C_{16}H_{18}N_2O_2S$ : 337.0773, found: 337.0754.



N-(4-azido-4-(4-(trifluoromethyl)phenyl)butyl)benzenesulfonamide was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2o** (81% yield) as a light yellow liquid.  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.89 – 7.80 (m, 2H), 7.65 – 7.54 (m, 3H), 7.54 – 7.47 (m, 2H), 7.36 (d,  $J$  = 8.2 Hz, 2H), 4.93 (t,  $J$  = 6.0 Hz, 1H), 4.47 (dd,  $J$  = 7.7, 6.3 Hz, 1H), 2.97 (qd,  $J$  = 6.6, 3.1 Hz, 2H), 1.84 – 1.70 (m, 2H), 1.63 – 1.40 (m, 2H).  $^{13}C$  NMR (126 MHz,  $CDCl_3$ )  $\delta$  143.31, 139.71, 132.73, 130.45 (q,  $J$  = 32.6 Hz), 129.15, 127.08, 126.91, 125.82 (q,  $J$  = 3.7 Hz), 123.86 (q,  $J$  = 272.1 Hz), 64.99, 42.52, 33.15, 26.07.  $^{19}F$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -62.59. HRMS (ESI) ( $m/z$ ):  $[M+H-N_2]^+$  calcd. for  $C_{17}H_{18}N_2O_2SF_3$ : 371.1036, found: 371.1036.

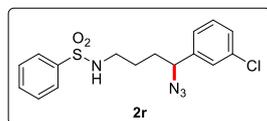


N-(4-azido-4-(*m*-tolyl)butyl)benzenesulfonamide was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2p** (95% yield) as a light yellow liquid.  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.91 – 7.76 (m, 2H), 7.62 – 7.44 (m, 3H), 7.23 (t,  $J$  = 7.6 Hz, 1H), 7.12 (d,  $J$  = 7.6 Hz, 1H), 7.07 – 6.98 (m, 2H), 4.85 (t,  $J$  = 6.2 Hz, 1H), 4.31 (dd,  $J$  = 7.9, 6.4 Hz, 1H), 2.95 (qd,  $J$  = 6.8, 1.5 Hz, 2H), 2.35 (s, 3H), 1.79 – 1.66 (m, 2H), 1.64 – 1.35 (m, 2H).  $^{13}C$  NMR (126 MHz,  $CDCl_3$ )  $\delta$  139.75, 139.03, 138.51, 132.63, 129.10, 128.66, 127.39, 126.93, 123.78, 65.68, 42.67, 33.02, 26.29, 21.40. HRMS (ESI) ( $m/z$ ):  $[M+H-N_2]^+$  calcd. for  $C_{17}H_{21}N_2O_2S$ : 317.1319, found: 317.1331.



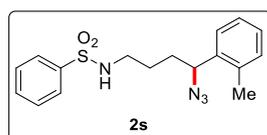
N-(4-azido-4-(3-methoxyphenyl)butyl)benzenesulfonamide was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 5:1) to afford the product **2q** (98% yield) as a light yellow liquid.  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.90 – 7.79 (m, 2H), 7.61 – 7.54 (m, 1H), 7.53 – 7.46 (m, 2H), 7.30 – 7.22 (m, 1H), 6.88 – 6.76 (m, 3H), 4.84 (t,  $J$  = 6.2 Hz, 1H), 4.33 (dd,  $J$  = 7.6, 6.5 Hz, 1H), 3.80 (s, 3H), 2.95 (qd,  $J$  = 6.8, 1.7 Hz, 2H), 1.81 – 1.63 (m, 2H), 1.62 – 1.38

(m, 2H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  160.01, 140.86, 139.89, 132.80, 130.00, 129.26, 127.08, 119.16, 113.78, 112.54, 65.72, 55.38, 42.80, 33.17, 26.39. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{H}-\text{N}_2]^+$  calcd. for  $\text{C}_{17}\text{H}_{21}\text{N}_2\text{O}_3\text{S}$ : 333.1268, found: 333.1302.



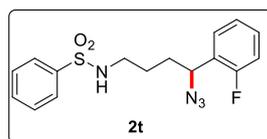
N-(4-azido-4-(3-chlorophenyl)butyl)benzenesulfonamide was prepared

following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2r** (91% yield) as a light yellow liquid.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.90 – 7.82 (m, 2H), 7.62 – 7.55 (m, 1H), 7.51 (t,  $J$  = 7.5 Hz, 2H), 7.29 (dd,  $J$  = 3.8, 1.4 Hz, 2H), 7.22 (s, 1H), 7.17 – 7.08 (m, 1H), 4.91 (t,  $J$  = 6.2 Hz, 1H), 4.35 (dd,  $J$  = 7.6, 6.4 Hz, 1H), 2.96 (qd,  $J$  = 6.6, 2.2 Hz, 2H), 1.77 – 1.65 (m, 2H), 1.62 – 1.38 (m, 2H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  141.50, 139.86, 134.84, 132.87, 130.28, 129.29, 128.64, 127.08, 127.00, 125.07, 65.16, 42.72, 33.24, 26.28. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{H}-\text{N}_2]^+$  calcd. for  $\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_2\text{S}$ : 337.0773, found: 337.0792.



N-(4-azido-4-(o-tolyl)butyl)benzenesulfonamide was prepared following

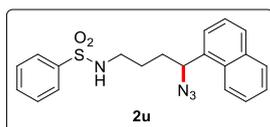
general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2s** (89% yield) as a light yellow liquid.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.90 – 7.80 (m, 2H), 7.62 – 7.53 (m, 1H), 7.53 – 7.46 (m, 2H), 7.23 (t,  $J$  = 7.6 Hz, 1H), 7.12 (d,  $J$  = 7.6 Hz, 1H), 7.05 – 6.99 (m, 2H), 4.85 (t,  $J$  = 6.2 Hz, 1H), 4.31 (dd,  $J$  = 7.9, 6.4 Hz, 1H), 2.95 (qd,  $J$  = 6.8, 1.5 Hz, 2H), 2.35 (s, 3H), 1.80 – 1.65 (m, 2H), 1.60 – 1.40 (m, 2H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  139.75, 139.03, 138.51, 132.63, 129.10, 128.66, 127.39, 126.93, 123.78, 65.68, 42.67, 33.02, 26.29, 21.40. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{H}-\text{N}_2]^+$  calcd. for  $\text{C}_{17}\text{H}_{21}\text{N}_2\text{O}_2\text{S}$ : 317.1319, found: 317.1331.



N-(4-azido-4-(2-fluorophenyl)butyl)benzenesulfonamide was prepared

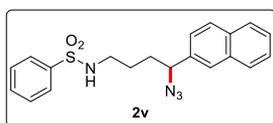
following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2t** (92% yield) as a light yellow liquid.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.90 – 7.82 (m, 2H), 7.62 – 7.55 (m, 1H), 7.51 (t,  $J$  = 7.5 Hz, 2H), 7.29 (dd,  $J$  = 3.8, 1.4 Hz, 2H), 7.22 (s, 1H), 7.17 – 7.08 (m, 1H),

4.91 (t,  $J = 6.2$  Hz, 1H), 4.35 (dd,  $J = 7.6, 6.4$  Hz, 1H), 2.96 (qd,  $J = 6.6, 2.2$  Hz, 2H), 1.75 – 1.65 (m, 2H), 1.62 – 1.38 (m, 2H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  160.01 (d,  $J = 246.8$  Hz), 139.81, 132.73, 129.87 (d,  $J = 8.3$  Hz), 129.18, 127.86 (d,  $J = 3.8$  Hz), 127.01, 126.37 (d,  $J = 13.6$  Hz), 124.68 (d,  $J = 3.6$  Hz), 115.75 (d,  $J = 22.0$  Hz), 58.83 (d,  $J = 2.2$  Hz), 42.71, 32.14, 26.26.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -118.59. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{H}-\text{N}_2]^+$  calcd. for  $\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_2\text{SF}$ : 321.1068, found: 321.1074.



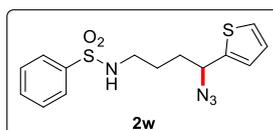
N-(4-azido-4-(naphthalen-1-yl)butyl)benzenesulfonamide was prepared

following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2u** (87% yield) as a light yellow liquid.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.01 (d,  $J = 8.2$  Hz, 1H), 7.91 – 7.76 (m, 4H), 7.60 – 7.38 (m, 7H), 5.11 (t,  $J = 6.9$  Hz, 1H), 5.00 (t,  $J = 6.1$  Hz, 1H), 3.11 – 2.84 (m, 2H), 1.91 (q,  $J = 7.4$  Hz, 2H), 1.65 – 1.45 (m, 2H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  139.84, 134.74, 134.11, 132.76, 130.65, 129.22, 129.20, 129.04, 127.06, 126.70, 126.02, 125.38, 124.50, 122.98, 62.52, 42.86, 32.43, 26.69. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{H}-\text{N}_2]^+$  calcd. for  $\text{C}_{20}\text{H}_{21}\text{N}_2\text{O}_2\text{S}$ : 353.1319, found: 353.1346.



N-(4-azido-4-(naphthalen-2-yl)butyl)benzenesulfonamide was prepared

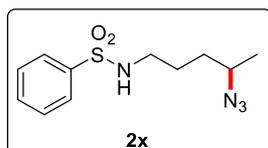
following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2v** (92% yield) as a light yellow liquid.  $^1\text{H}$  NMR (500 MHz, Chloroform-*d*)  $\delta$  7.88 – 7.77 (m, 5H), 7.65 (s, 1H), 7.53 – 7.44 (m, 3H), 7.41 (t,  $J = 7.6$  Hz, 2H), 7.32 (dd,  $J = 8.5, 1.5$  Hz, 1H), 5.03 (t,  $J = 6.1$  Hz, 1H), 4.50 (t,  $J = 7.1$  Hz, 1H), 3.03 – 2.86 (m, 2H), 1.87 – 1.72 (m, 2H), 1.60 – 1.37 (m, 2H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  139.87, 136.59, 133.29, 133.22, 132.76, 129.22, 129.03, 128.12, 127.84, 127.04, 126.61, 126.48, 126.24, 124.28, 65.97, 42.79, 33.07, 26.39. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{H}-\text{N}_2]^+$  calcd. for  $\text{C}_{20}\text{H}_{21}\text{N}_2\text{O}_2\text{S}$ : 353.1319, found: 353.1346.



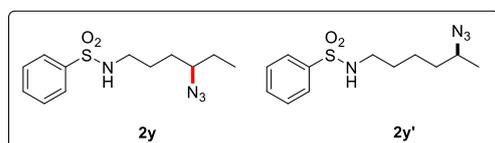
N-(4-azido-4-(thiophen-2-yl)butyl)benzenesulfonamide was prepared

following general procedure A and the reaction mixture was purified by flash column

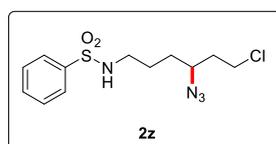
chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2w** (80% yield) as a light yellow liquid.  $^1\text{H}$  NMR (500 MHz, Chloroform-*d*)  $\delta$  7.86 (d,  $J$  = 7.4 Hz, 2H), 7.57 (t,  $J$  = 7.3 Hz, 1H), 7.50 (t,  $J$  = 7.6 Hz, 2H), 7.30 – 7.23 (m, 1H), 6.96 (d,  $J$  = 4.3 Hz, 2H), 5.13 (t,  $J$  = 6.0 Hz, 1H), 4.60 (t,  $J$  = 7.2 Hz, 1H), 2.96 (q,  $J$  = 6.6 Hz, 2H), 1.87 – 1.76 (m, 2H), 1.65 – 1.45 (m, 2H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  142.11, 139.86, 132.84, 129.29, 127.11, 126.93, 125.87, 125.74, 60.93, 42.69, 33.60, 26.48. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{H}-\text{N}_2]^+$  calcd. for  $\text{C}_{14}\text{H}_{17}\text{N}_2\text{O}_2\text{S}_2$ : 309.0726, found: 309.0753.



N-(4-azidopentyl)benzenesulfonamide was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2x** (57% yield) as a light yellow liquid.  $^1\text{H}$  NMR (500 MHz, Chloroform-*d*)  $\delta$  7.94 – 7.85 (m, 2H), 7.63 – 7.56 (m, 1H), 7.56 – 7.50 (m, 2H), 5.08 (t,  $J$  = 5.9 Hz, 1H), 3.38 (h,  $J$  = 6.5 Hz, 1H), 2.97 (p,  $J$  = 6.4 Hz, 2H), 1.60 – 1.42 (m, 4H), 1.20 (d,  $J$  = 6.5 Hz, 3H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  139.87, 132.75, 129.20, 127.04, 57.36, 42.86, 33.03, 26.20, 19.38. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd. for  $\text{C}_{11}\text{H}_{16}\text{N}_4\text{O}_2\text{SNa}$ : 291.0886, found: 291.0911.

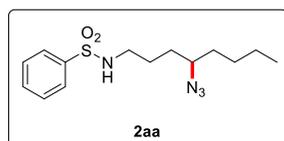


N-(4-azidoethyl)benzenesulfonamide was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2y** and **2y'** (74% yield) in the ratio higher than 10:1 as a light yellow liquid.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.88 (d,  $J$  = 7.4 Hz, 2H), 7.79 – 7.42 (m, 3H), 5.01 (t,  $J$  = 6.0 Hz, 0.89H, **2y**), 4.96 (t,  $J$  = 6.0 Hz, 0.08H, **2y'**) 3.24 – 3.05 (m, 0.08H, **2y'**), 3.18 – 3.08 (m, 0.92H, **2y**), 2.97 (q,  $J$  = 6.5 Hz, 2H), 1.71 – 1.34 (m, 6H), 1.21 (d,  $J$  = 6.5 Hz, 0.24H, **2y'**), 0.94 (t,  $J$  = 7.4 Hz, 2.76H, **2y**).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  139.92, 132.81, 129.28, 127.11, 63.95, 57.80, 42.98, 35.65, 30.89, 29.78, 29.38, 27.42, 26.30, 23.11, 19.45, 10.50. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd. for  $\text{C}_{12}\text{H}_{18}\text{N}_4\text{O}_2\text{SNa}$ : 305.1043, found: 305.1048.

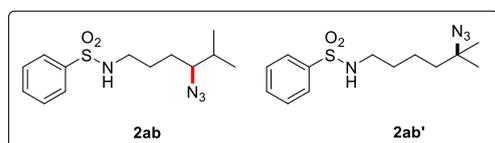


N-(4-azido-6-chlorohexyl)benzenesulfonamide was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with

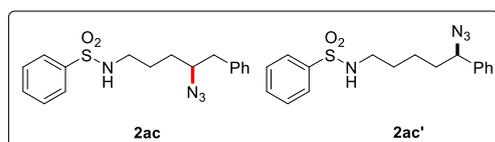
petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the single product **2z** (50% yield) as a light yellow liquid.  $^1\text{H}$  NMR (500 MHz, Chloroform-*d*)  $\delta$  7.97 – 7.84 (m, 2H), 7.63 – 7.57 (m, 1H), 7.57 – 7.51 (m, 2H), 4.83 (t,  $J$  = 6.1 Hz, 1H), 3.66 – 3.58 (m, 2H), 3.52 (p,  $J$  = 6.9 Hz, 1H), 3.01 (q,  $J$  = 6.3 Hz, 2H), 1.86 (q,  $J$  = 6.3 Hz, 2H), 1.75 – 1.47 (m, 5H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  139.97, 132.92, 129.35, 127.14, 59.49, 42.92, 41.42, 37.15, 31.39, 26.28. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd. for  $\text{C}_{12}\text{H}_{17}\text{N}_4\text{O}_2\text{SClNa}$ : 339.0653, found: 339.0657.



**2aa** N-(4-azido)benzenesulfonamide was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2aa** in the regioselectivity higher than 10:1 (70% yield) as a light yellow liquid.  $^1\text{H}$  NMR (500 MHz, Chloroform-*d*)  $\delta$  7.88 (d,  $J$  = 8.1 Hz, 2H), 7.62 – 7.56 (m, 1H), 7.53 (t,  $J$  = 7.7 Hz, 2H), 4.89 (t,  $J$  = 6.0 Hz, 1H), 3.23 – 3.12 (m, 1H), 2.97 (p,  $J$  = 7.9, 7.2 Hz, 2H), 1.76 – 1.28 (m, 10H), 0.90 (t,  $J$  = 6.7 Hz, 3H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.01, 132.81, 129.29, 127.13, 62.67, 62.55, 43.13, 43.03, 36.54, 34.12, 33.91, 31.36, 29.52, 28.23, 26.35, 23.20, 22.57, 19.40, 14.04, 13.95. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd. for  $\text{C}_{14}\text{H}_{22}\text{N}_4\text{O}_2\text{SNa}$ : 333.1356, found: 333.1361.

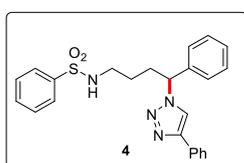


**2ab** and **2ab'** N-(4-azido-5-methylhexyl)benzenesulfonamide was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2ab** and **2ab'** (63% yield) in the ratio of 2:1 as a light yellow liquid.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.89 (d,  $J$  = 7.5 Hz, 2H), 7.71 – 7.48 (m, 3H), 5.00 (t,  $J$  = 6.0 Hz, 0.64H, **2ab**), 4.94 (t,  $J$  = 6.0 Hz, 0.33H, **2ab'**), 3.04 – 2.99 (m, 0.63H, **2ab**), 3.00 – 2.93 (m, 2H), 1.85 – 1.25 (m, 6H), 1.20 (s, 2H, **2ab'**), 0.91 (t,  $J$  = 6.5 Hz, 4H, **2ab**).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  139.97, 139.93, 132.81, 132.76, 129.28, 129.25, 127.12, 68.82, 61.53, 43.12, 43.03, 40.93, 32.64, 29.84, 28.52, 26.76, 25.96, 21.33, 19.34, 18.10. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd. for  $\text{C}_{13}\text{H}_{20}\text{N}_4\text{O}_2\text{SNa}$ : 319.1199, found: 319.1203.



**2ac** and **2ac'** N-(4-azido-5-phenylpentyl)benzenesulfonamide and N-(5-azido-5-phenylpentyl)benzenesulfonamide were afforded following general procedure A and

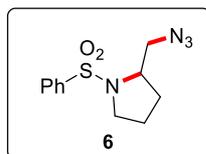
the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **2ac** and **2ac'** (75% yield) in the ratio of 1:1.1 as a light yellow liquid.  $^1\text{H}$  NMR (500 MHz, Chloroform-*d*)  $\delta$  7.90 – 7.82 (m, 2H), 7.61 – 7.55 (m, 1H), 7.54 – 7.49 (m, 2H), 7.39 – 7.34 (m, 1H), 7.34 – 7.23 (m, 4H), 7.19 – 7.14 (m, 1H), 4.778-4.58 (m, 1H), 4.34 (dd,  $J$  = 7.82, 6.50 Hz, 0.526H, **2ac'**), 3.52-3.41 (m, 0.474H, **2ac**), 2.94 (dq,  $J$  = 20.1, 6.6 Hz, 2H), 2.78 (h,  $J$  = 7.5 Hz, 0.955H, **2ac**), 1.79 – 1.21 (m, 5.052H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.03, 140.01, 139.56, 137.47, 132.84, 132.79, 129.37, 129.30, 129.27, 128.96, 128.75, 128.45, 127.14, 126.99, 126.94, 66.22, 63.71, 43.06, 42.94, 41.04, 35.72, 31.01, 29.37, 26.43, 23.32. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd. for  $\text{C}_{17}\text{H}_{20}\text{N}_4\text{O}_2\text{SNa}$ : 367.1199, found: 367.1205



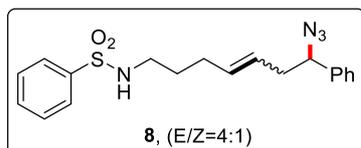
N-(4-phenyl-4-(4-phenyl-1H-1,2,3-triazol-1-yl)butyl)benzenesulfonamide

was prepared following procedure B according to Ref. S2 and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 3:1) to afford the product **4** (74% yield) as white solid.  $^1\text{H}$  NMR (500 MHz,  $\text{DMSO-}d_6$ )  $\delta$  8.75 (s, 1H), 7.85 (d,  $J$  = 7.6 Hz, 2H), 7.76 (d,  $J$  = 7.4 Hz, 2H), 7.68 – 7.53 (m, 4H), 7.47 – 7.32 (m, 8H), 5.45 (dd,  $J$  = 9.2, 6.8 Hz, 1H), 2.81 (q,  $J$  = 6.5 Hz, 2H), 2.43 – 2.35 (m, 1H), 2.28 – 2.17 (m, 1H), 1.37 – 1.25 (m, 2H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{DMSO}$ )  $\delta$  146.94, 140.96, 140.16, 132.78, 131.16, 129.65, 129.36, 129.25, 128.67, 128.37, 127.26, 126.84, 125.58, 120.74, 64.21, 42.41, 32.13, 26.56. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{H}]^+$  calcd. for  $\text{C}_{24}\text{H}_{25}\text{N}_4\text{O}_2\text{S}$ : 433.1693, found: 433.1699. Melting point: 176 °C – 177 °C

## Mechanistic Studies



2-(azidomethyl)-1-(phenylsulfonyl)pyrrolidine was prepared following general procedure A and the reaction mixture was purified by column chromatography with petroleum ether and ethyl acetate (PE/EA = 15:1) to afford the product **6** (37% yield) as a light yellow liquid.  $^1\text{H}$  NMR (500 MHz, Chloroform-*d*)  $\delta$  7.85 (d,  $J$  = 7.5 Hz, 2H), 7.63 (t,  $J$  = 7.3 Hz, 1H), 7.55 (t,  $J$  = 7.6 Hz, 2H), 3.73 (tt,  $J$  = 7.3, 3.6 Hz, 1H), 3.60 – 3.46 (m, 3H), 3.20 – 3.15 (m, 1H), 1.90 – 1.80 (m, 2H), 1.71 – 1.65 (m, 1H), 1.60 – 1.53 (m, 1H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  137.11, 133.09, 129.34, 127.66, 59.07, 55.32, 49.62, 29.44, 24.19. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{Na}]^+$  calcd. for  $\text{C}_{11}\text{H}_{14}\text{N}_4\text{O}_2\text{SNa}$ : 287.0730, found: 287.0728.

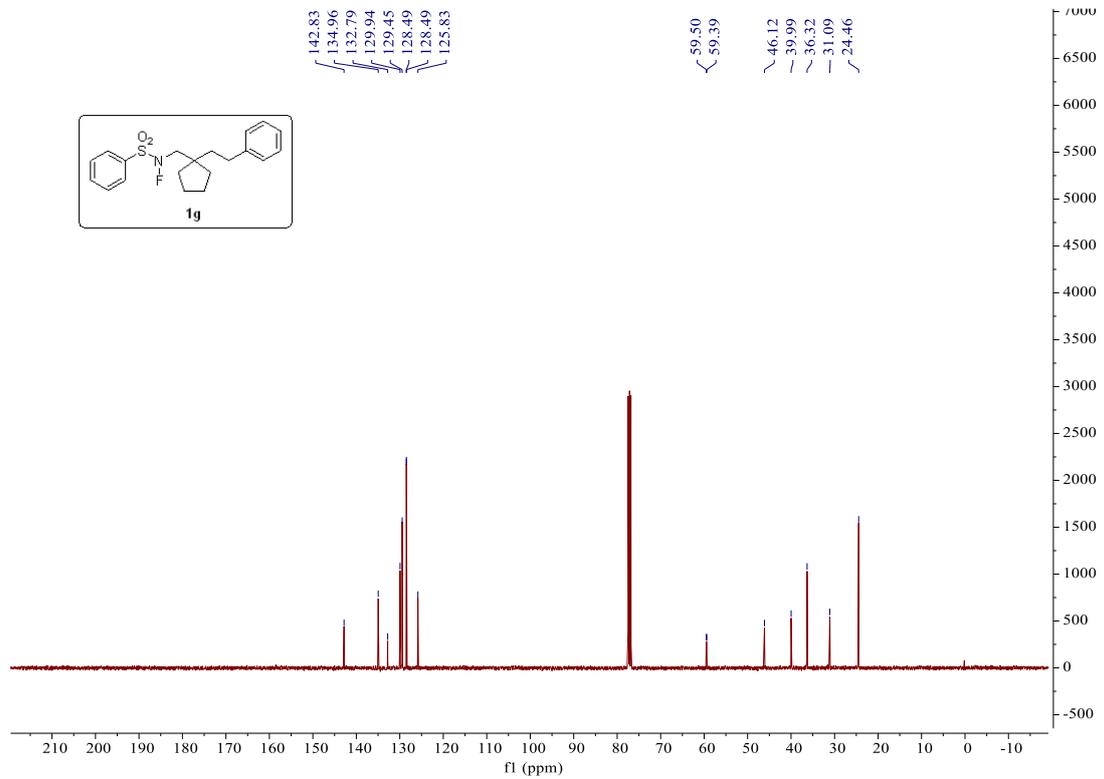
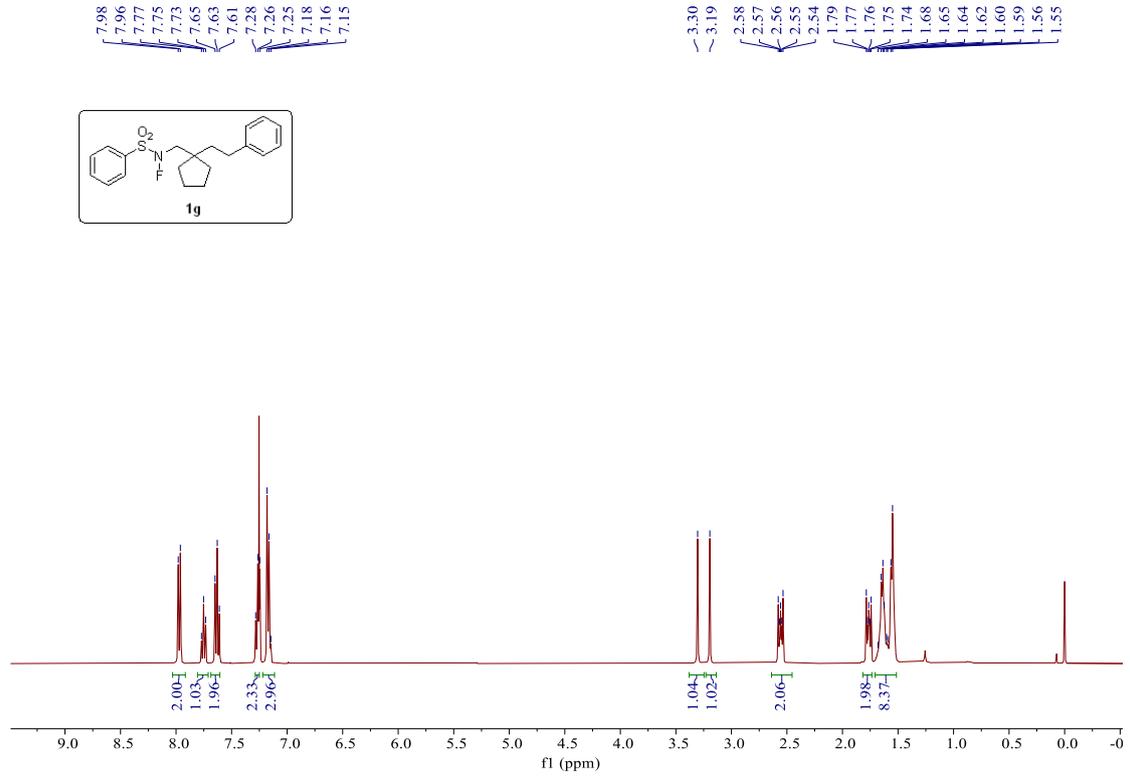


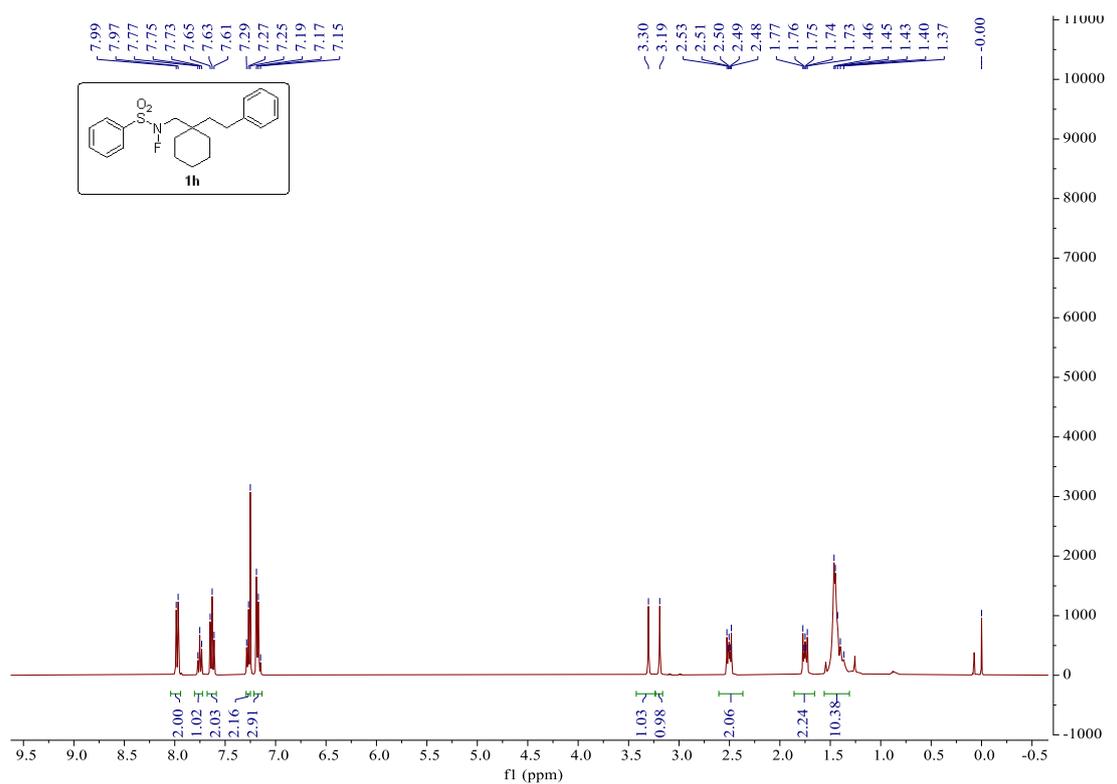
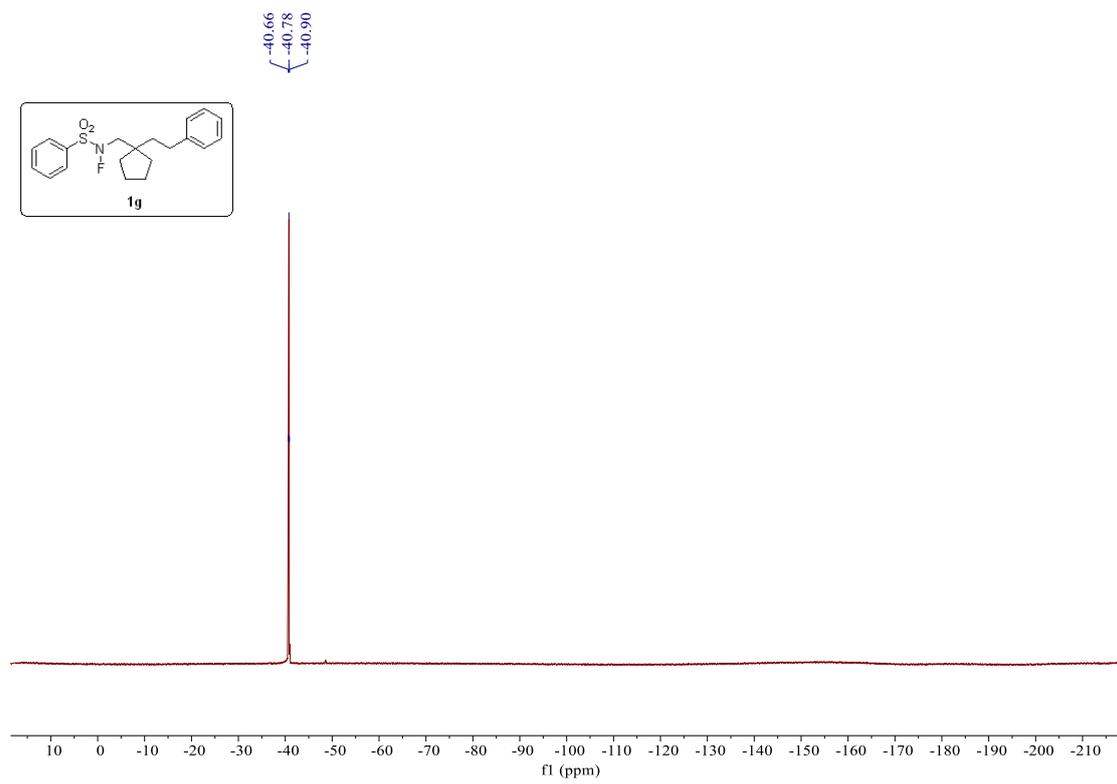
N-(7-azido-7-phenylhept-4-en-1-yl)benzenesulfonamide was prepared following general procedure A and the reaction mixture was purified by flash column chromatography with petroleum ether and ethyl acetate (PE/EA = 7:1) to afford the product **8** (82% yield, E/Z=4:1) as a light yellow liquid.  $^1\text{H}$  NMR (500 MHz, Chloroform-*d*)  $\delta$  7.91 – 7.83 (m, 2H), 7.58 (t,  $J$  = 7.3 Hz, 1H), 7.52 (t,  $J$  = 7.5 Hz, 2H), 7.39 – 7.33 (m, 2H), 7.34 – 7.25 (m, 3H), 5.44 – 5.27 (m, 2H), 4.71 – 4.62 (m, 1H), 4.48 – 4.40 (m, 1H), 2.91 (q,  $J$  = 6.6 Hz, 2H), 2.56 – 2.40 (m, 2H), 1.98 (q,  $J$  = 6.9 Hz, 2H), 1.55 – 1.37 (m, 2H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.14, 139.39, 132.85, 132.73, 129.23, 128.88, 128.85, 128.41, 128.35, 127.13, 127.11, 127.01, 126.99, 126.54, 66.13, 42.67, 39.59, 29.59, 29.12. HRMS (ESI) ( $m/z$ ):  $[\text{M}+\text{H}-\text{N}_2]^+$  calcd. for  $\text{C}_{19}\text{H}_{23}\text{N}_2\text{O}_2\text{S}$ : 343.1475, found: 343.1490.

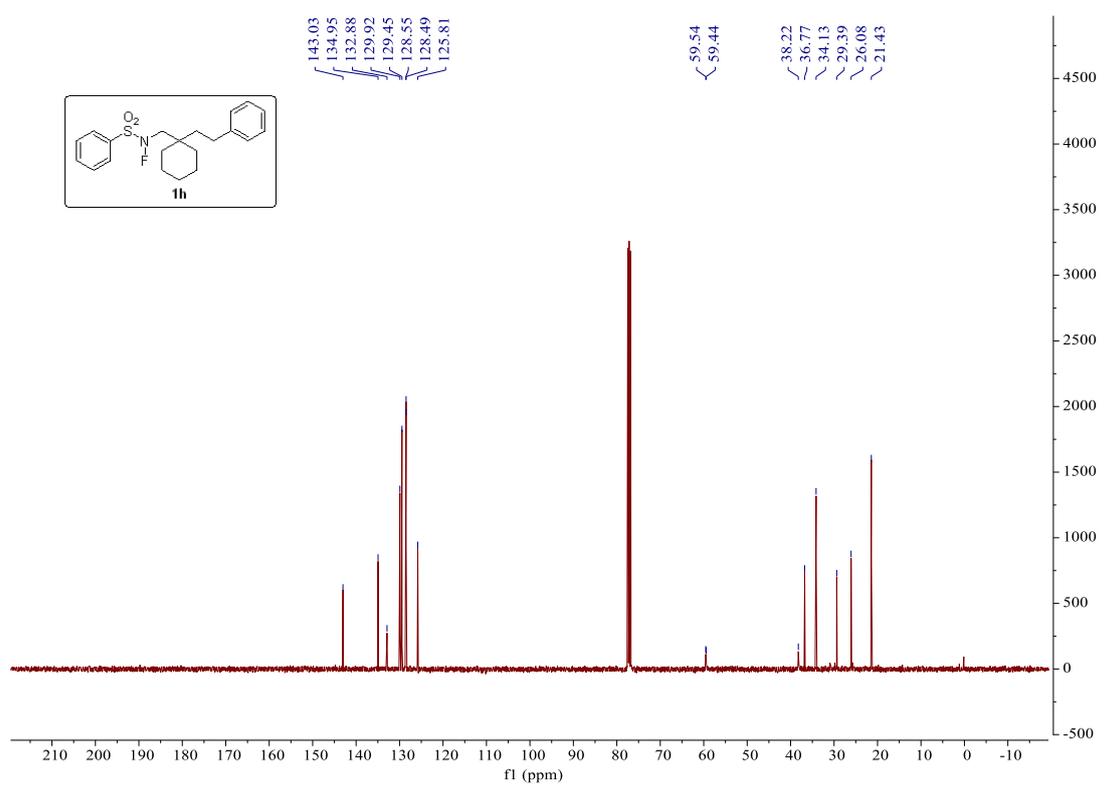
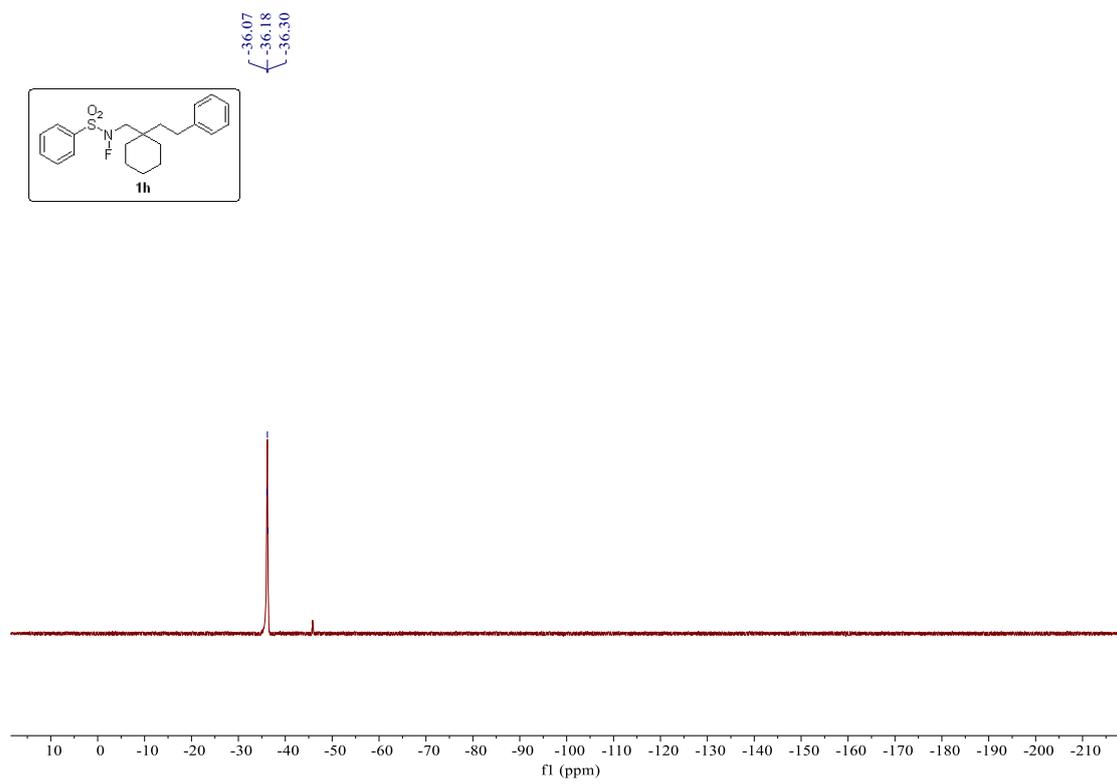
#### **IV. References:**

1. Wang, C.-Y.; Qin, Z.-Y.; Huang, Y.-L.; Jin, R.-X.; Lan, Q.; Wang, X.-S. *iScience* 2019, **21**, 490.
2. Lu, M.-Z.; Wang, C.-Q.; Loh, T.-P. *Org. Lett.* 2015, **17**, 6110

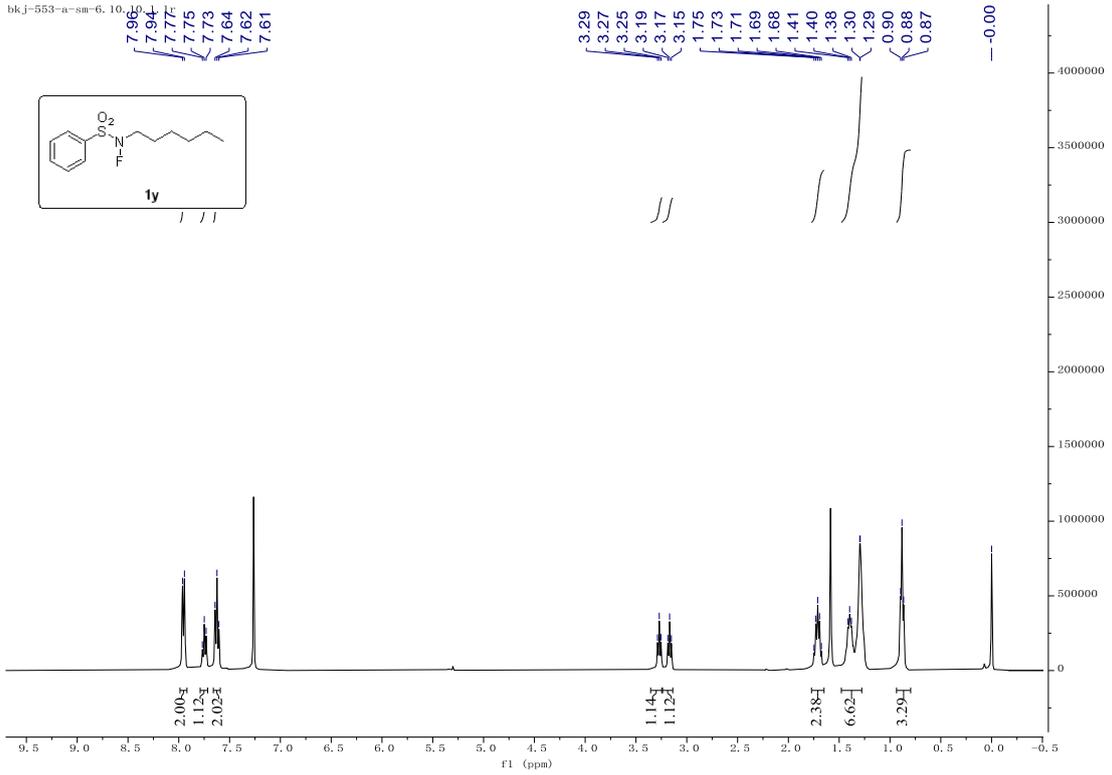
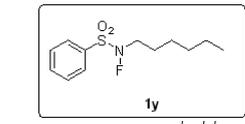
# $^1\text{H}$ , $^{19}\text{F}$ , and $^{13}\text{C}$ NMR Spectra



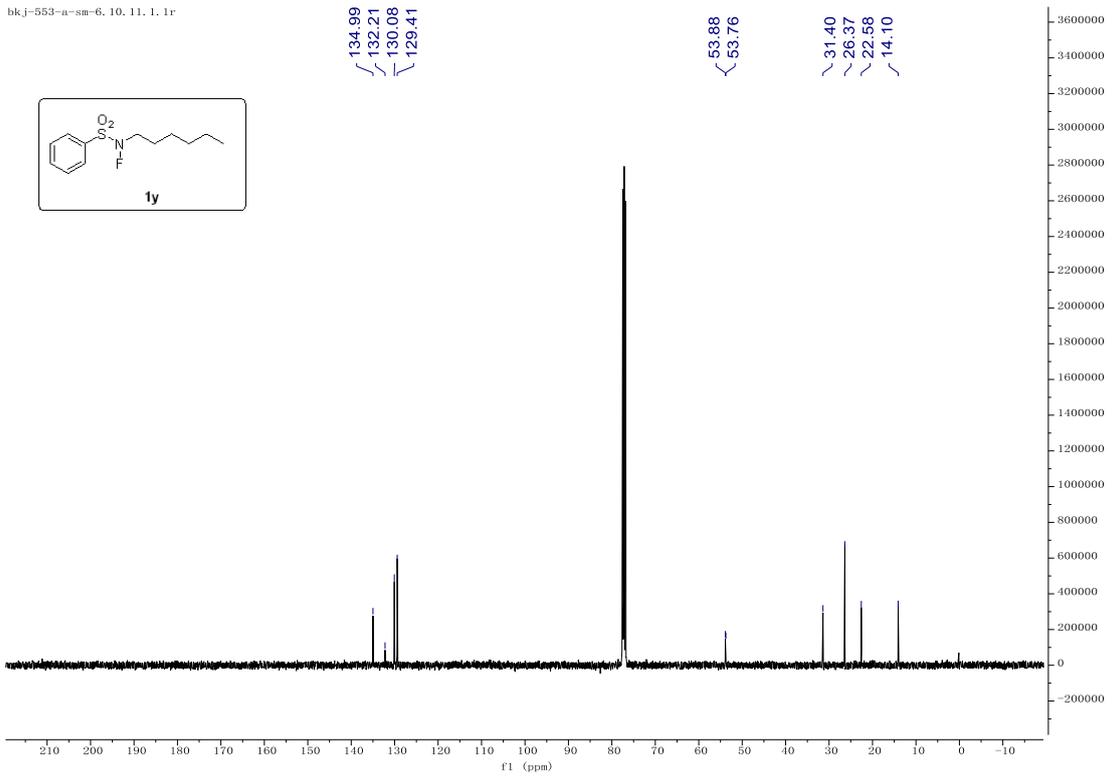
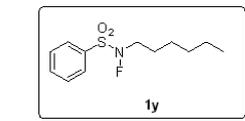




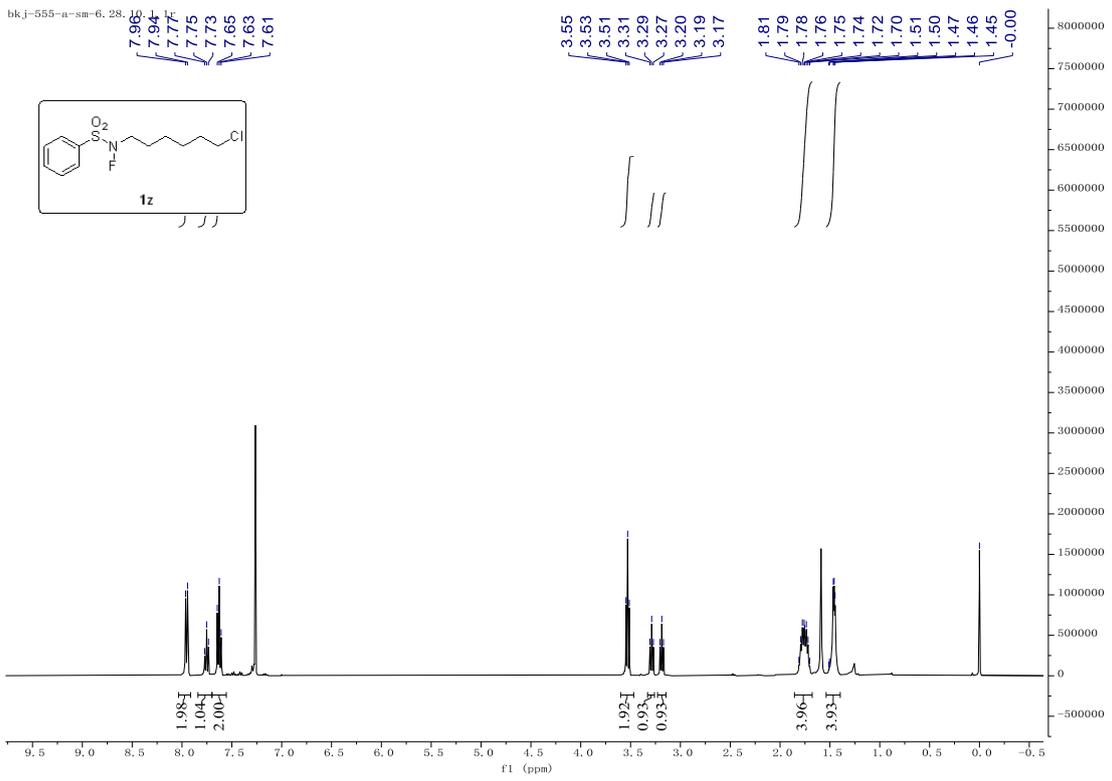
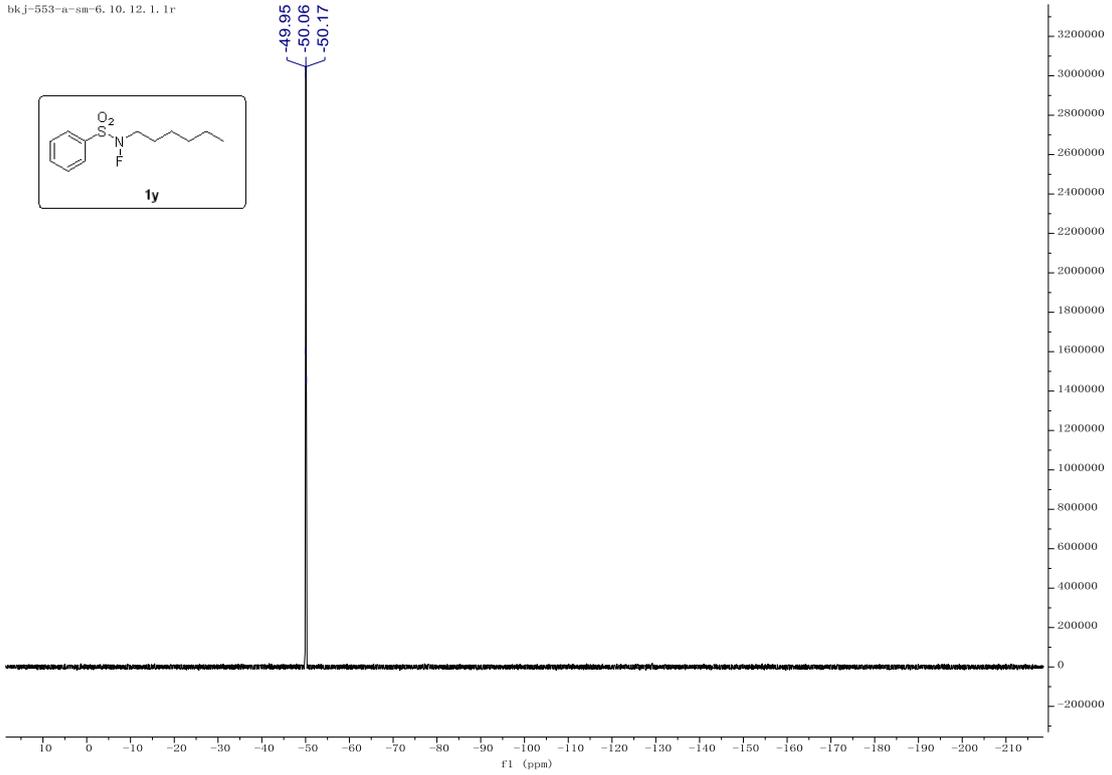
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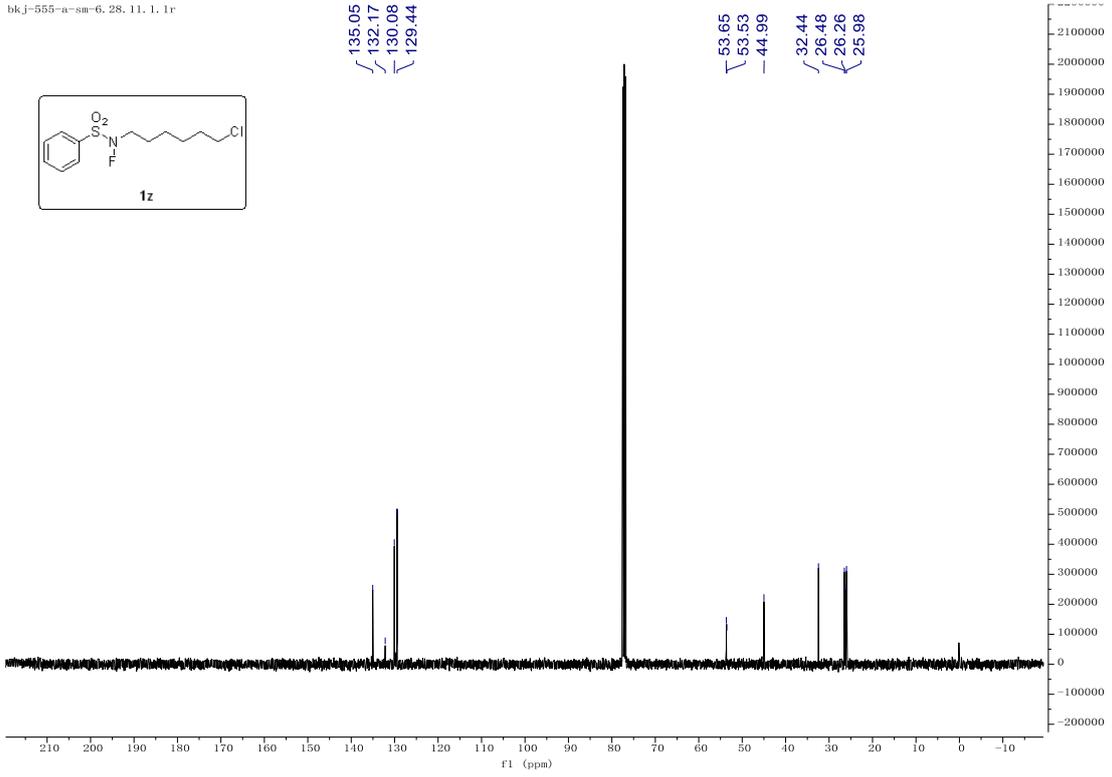
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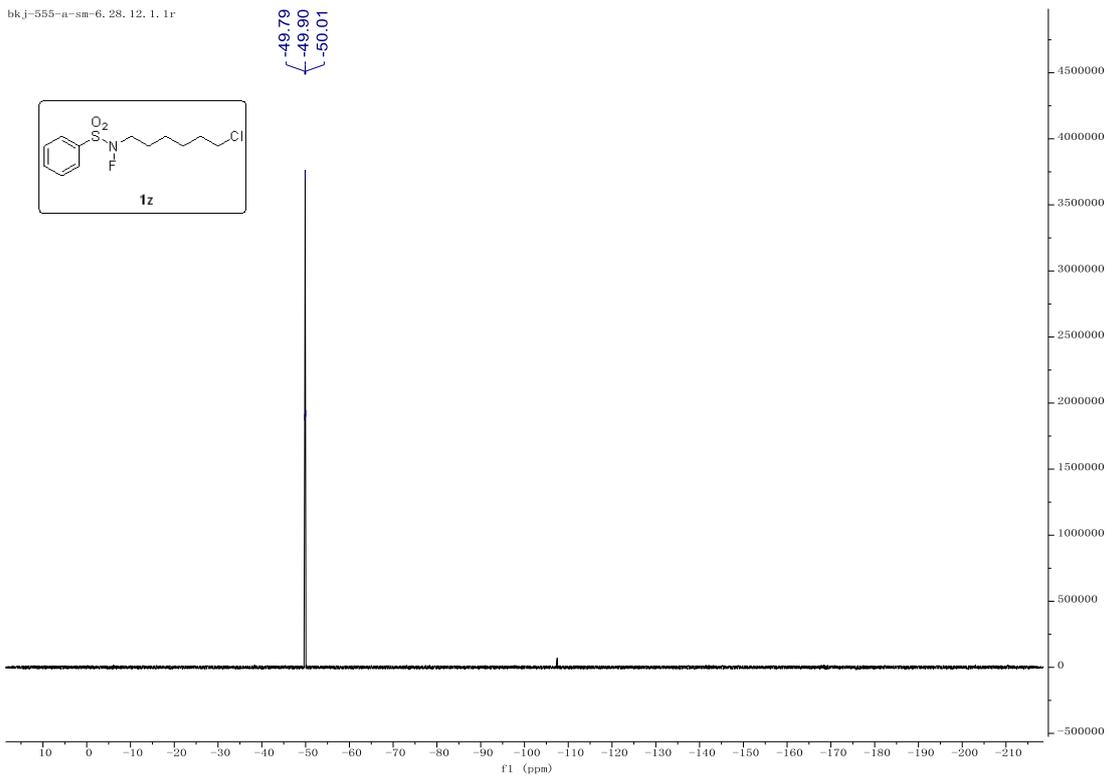
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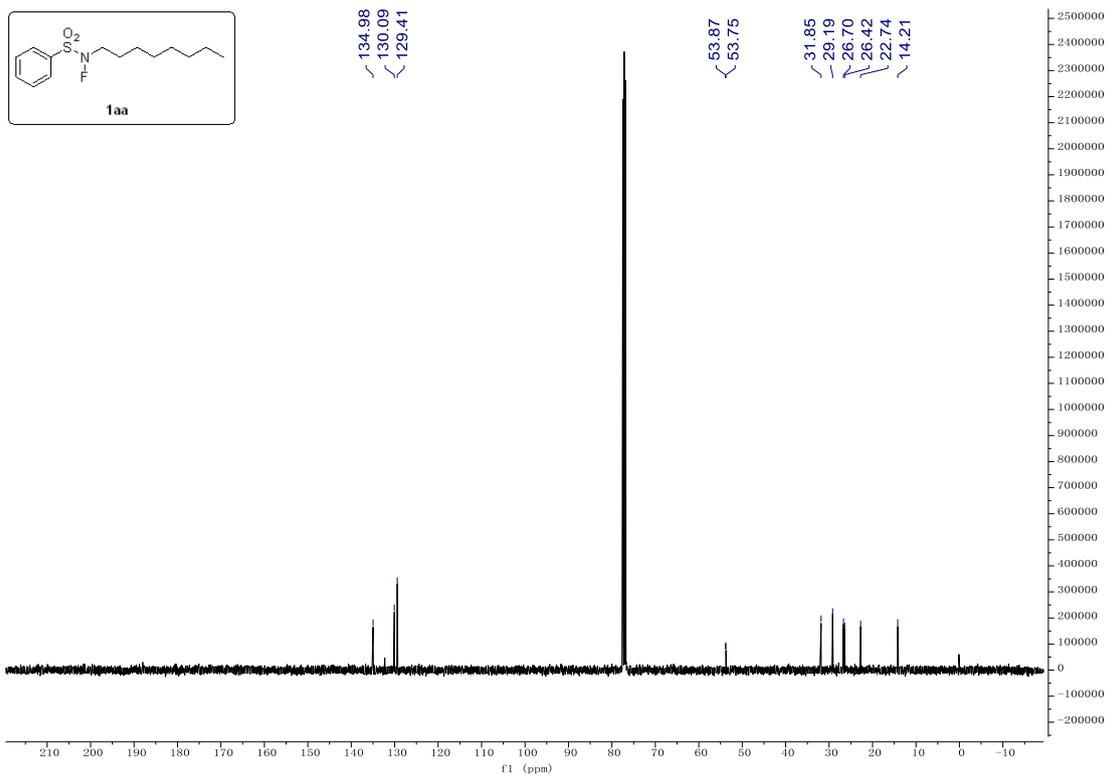
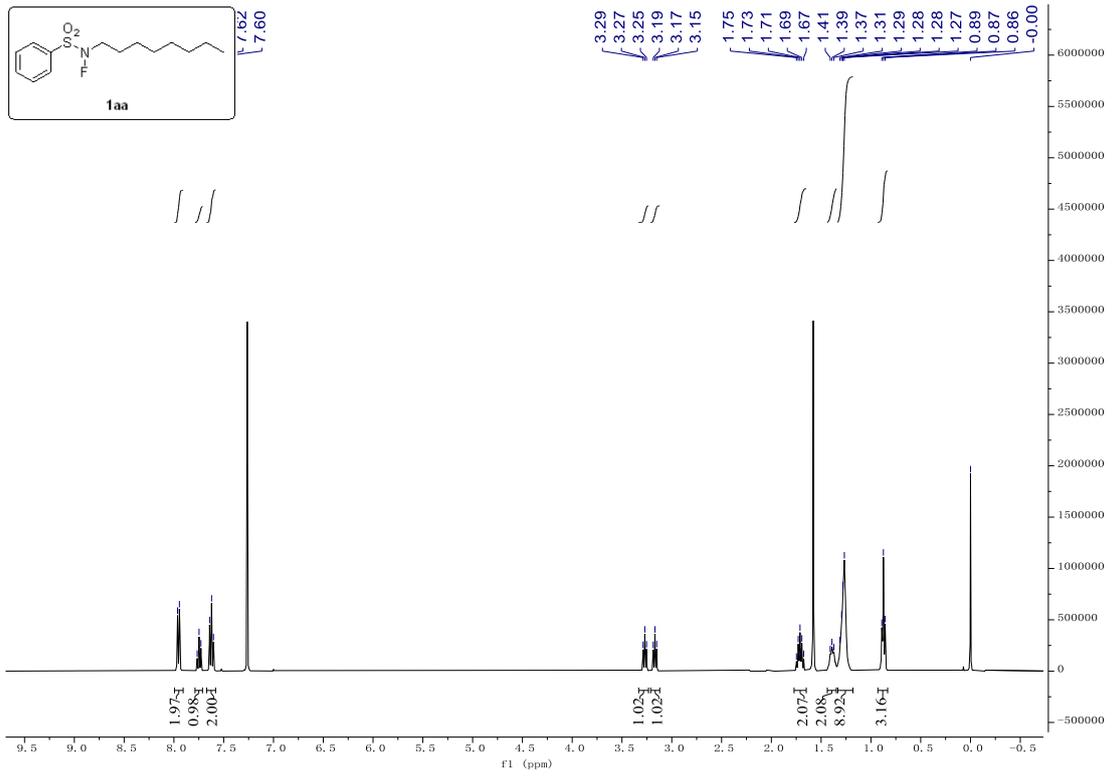


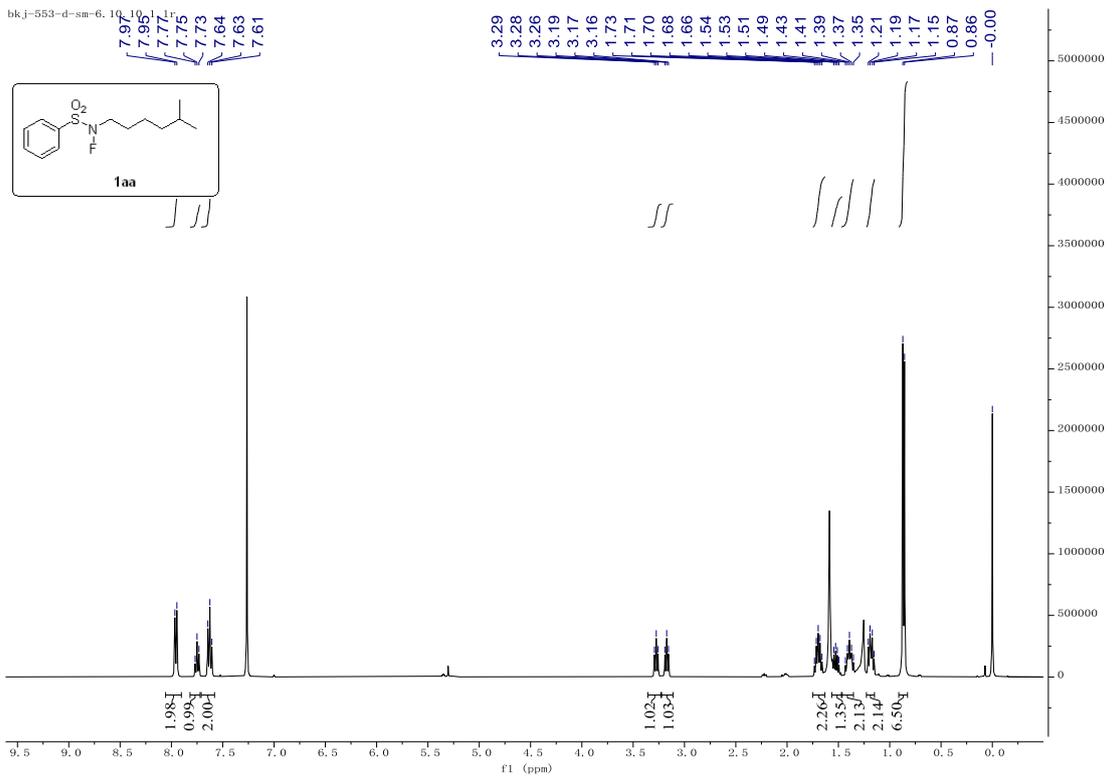
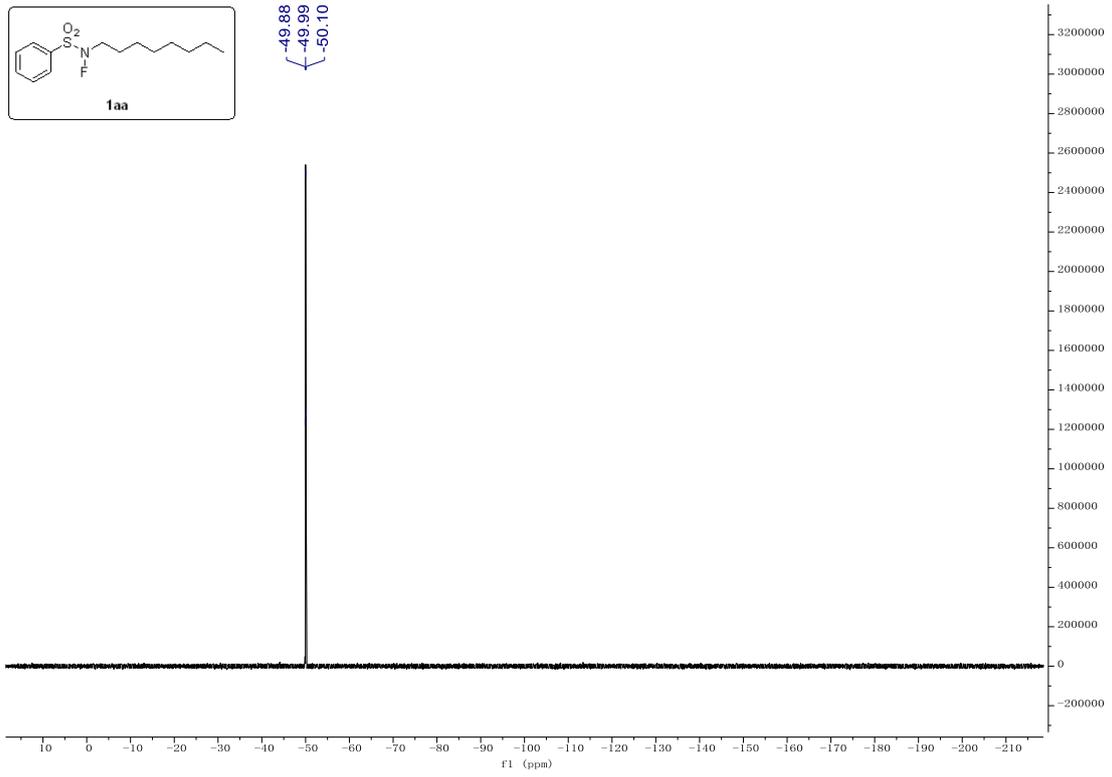
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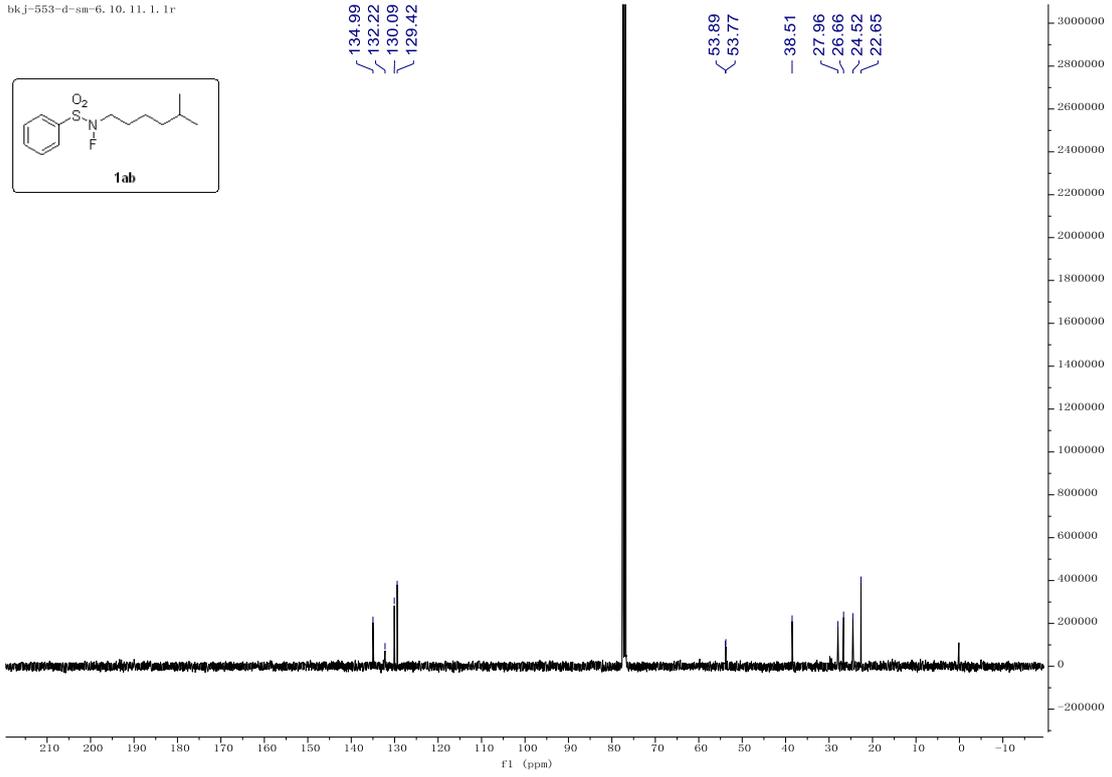
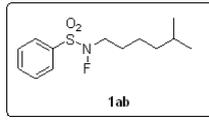
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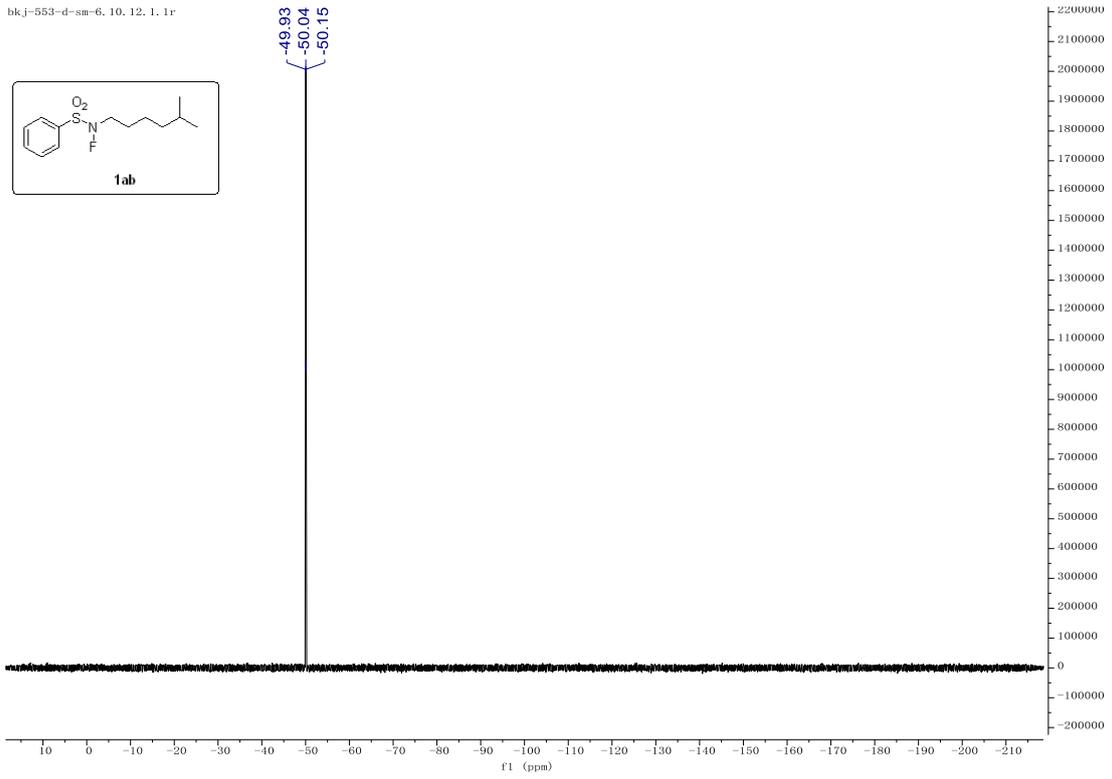
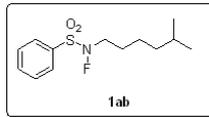


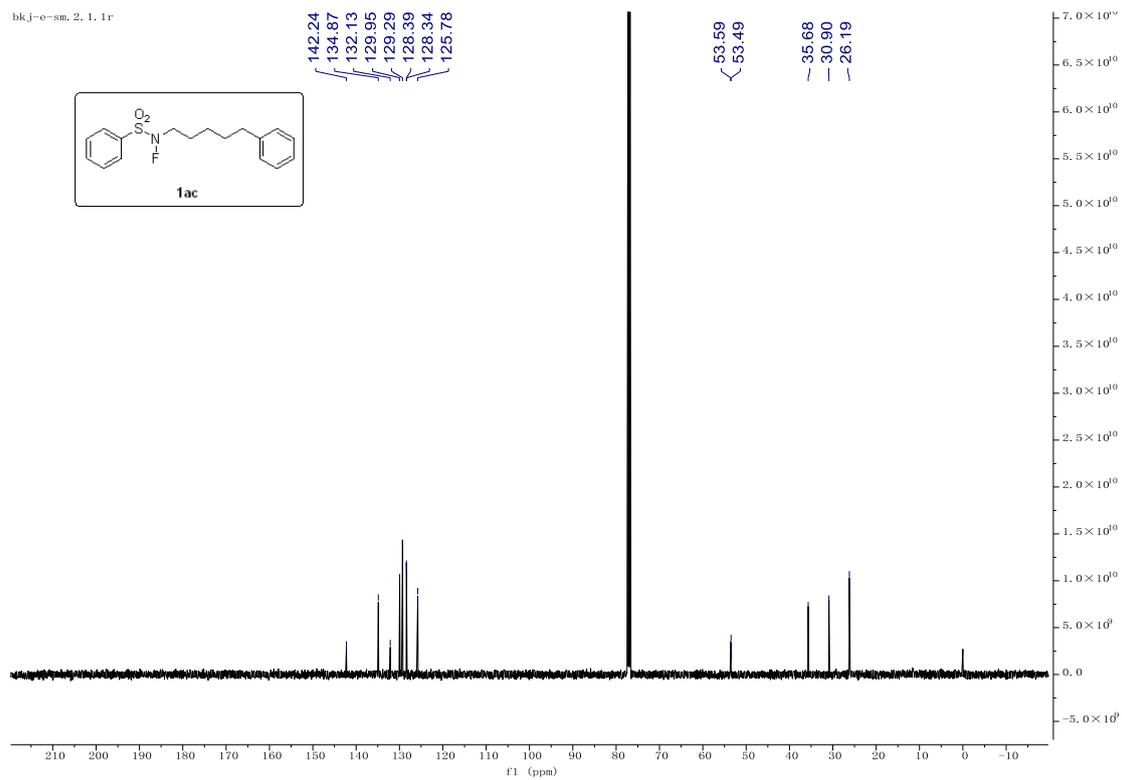
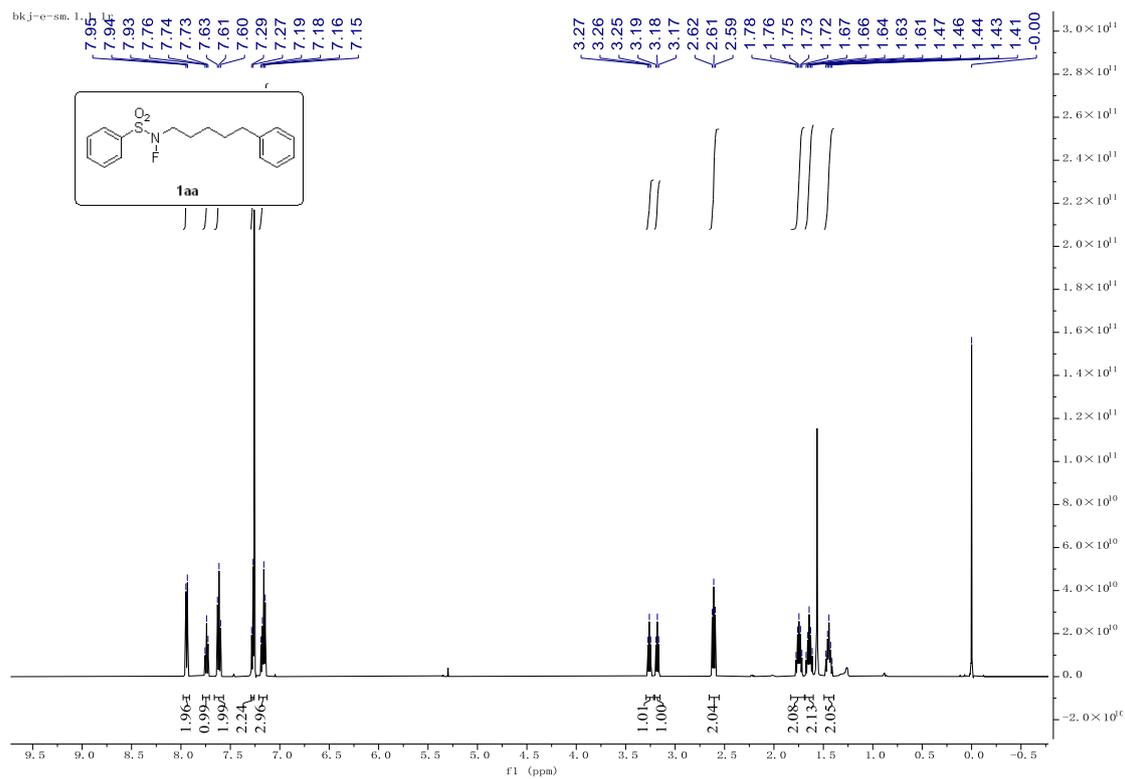


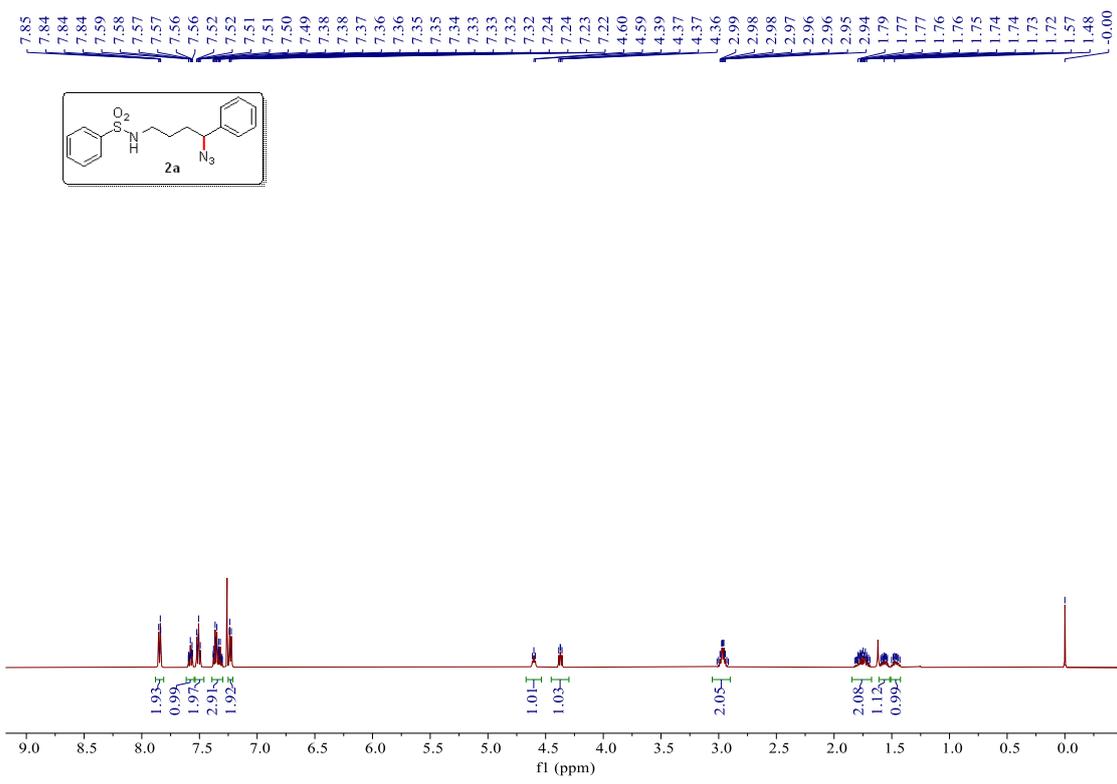
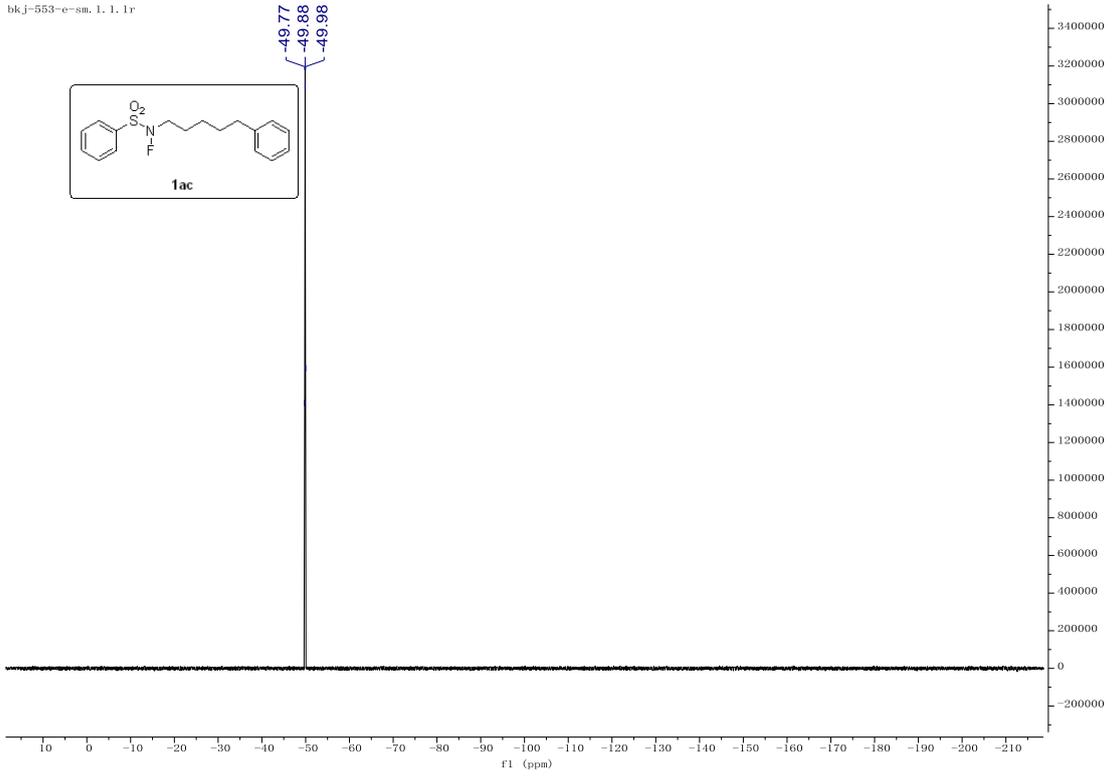
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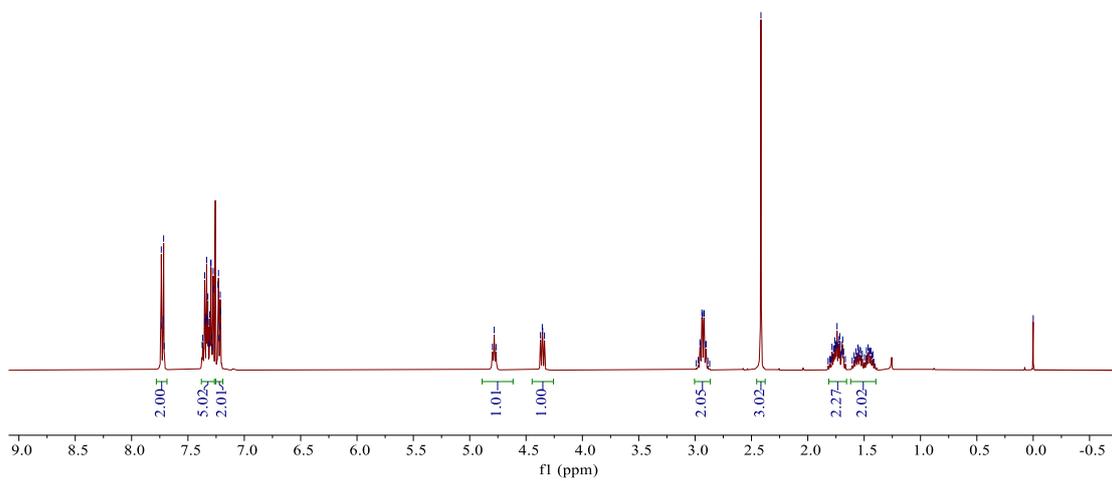
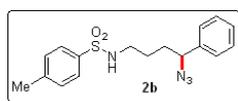
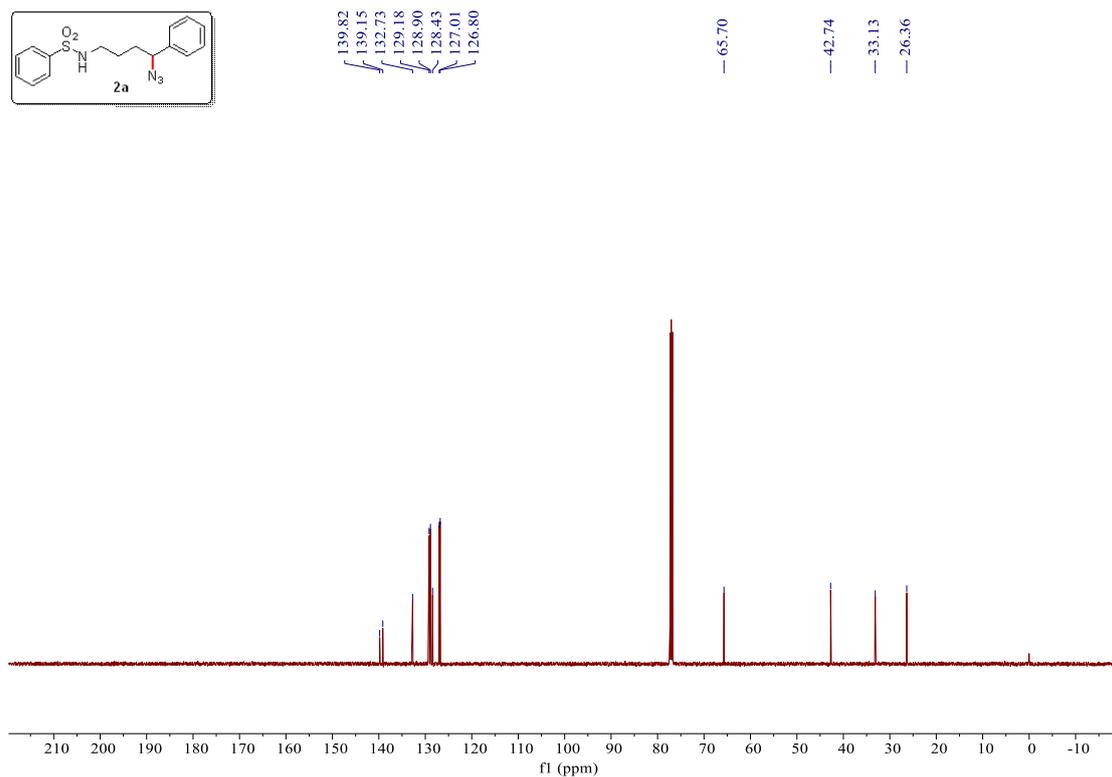
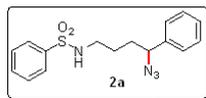


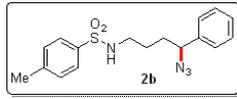
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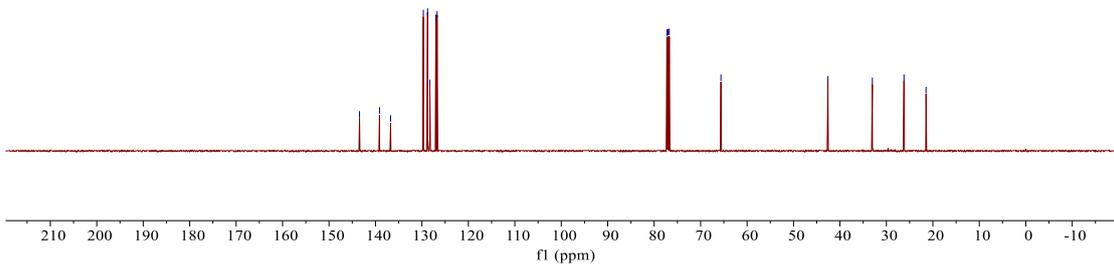


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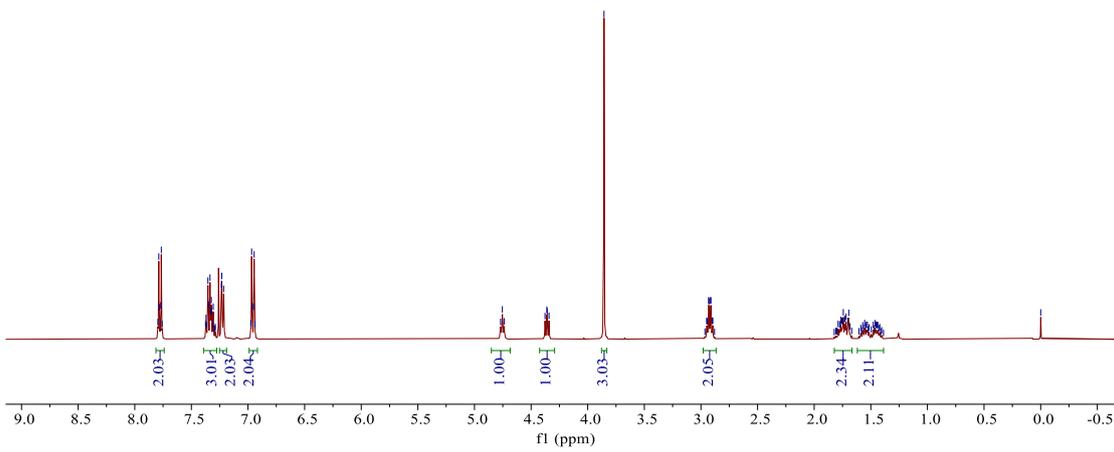
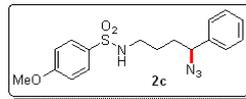
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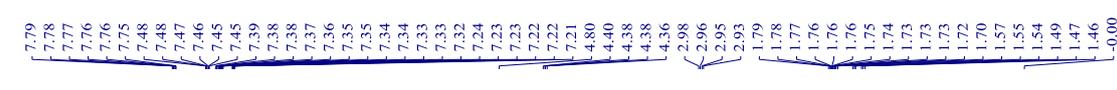
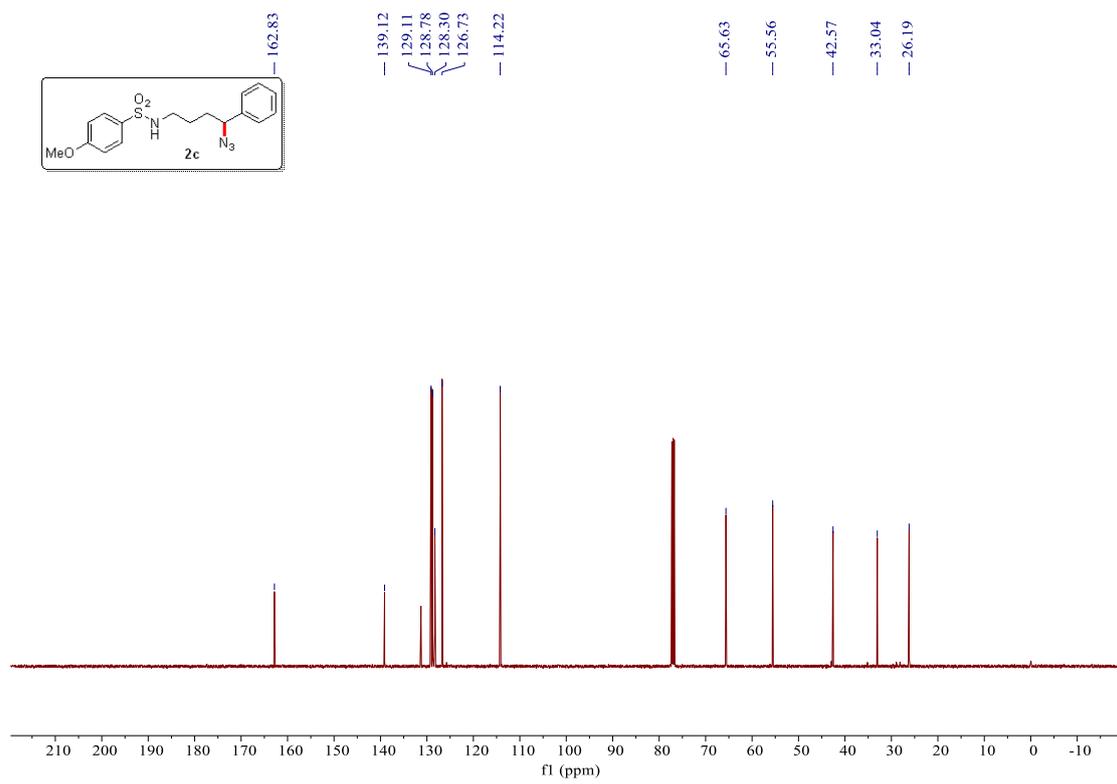
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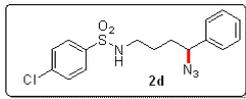
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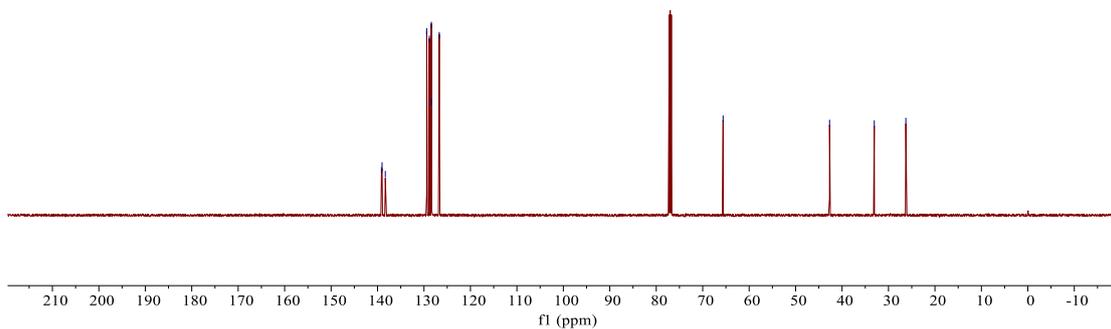
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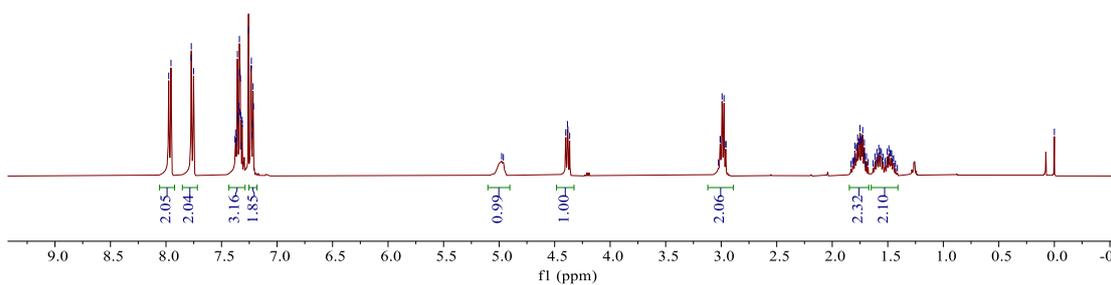
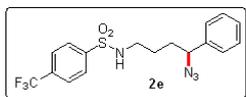
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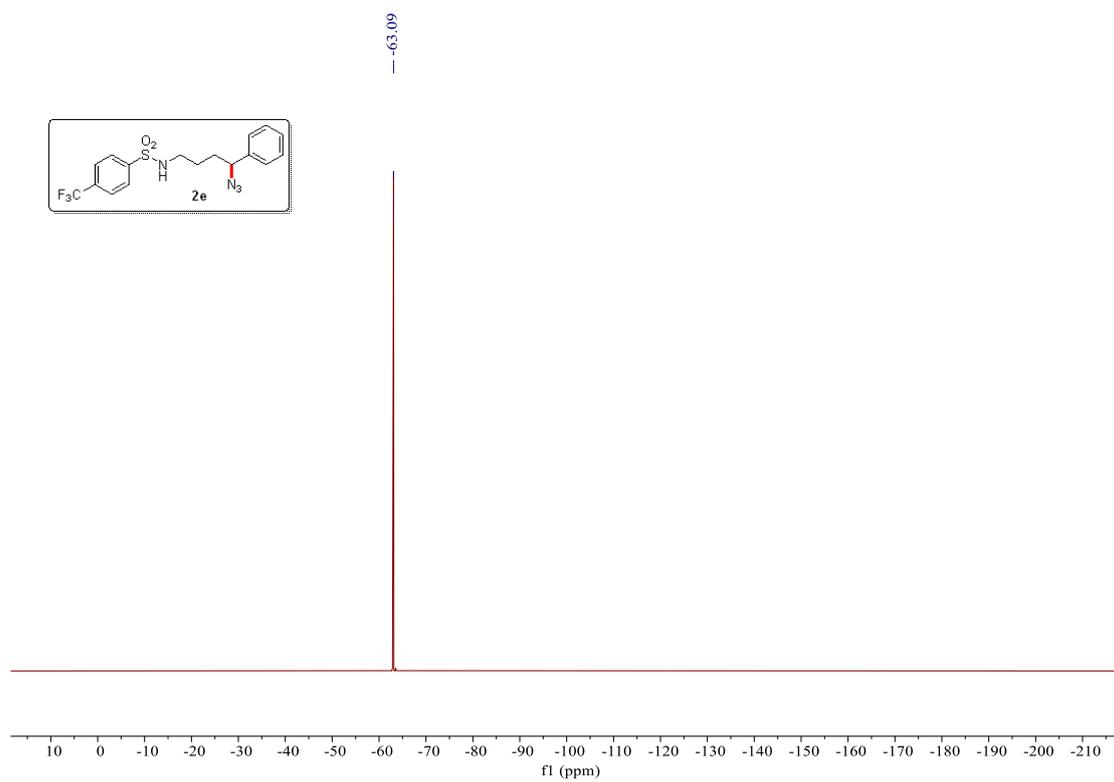
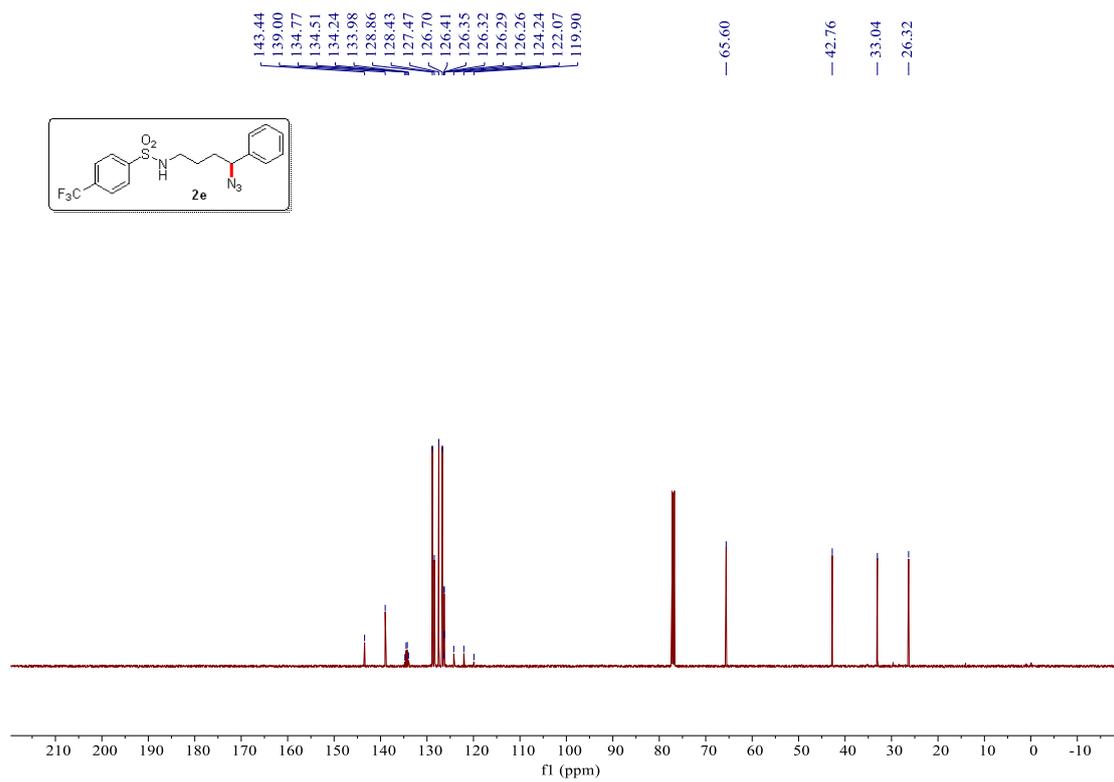
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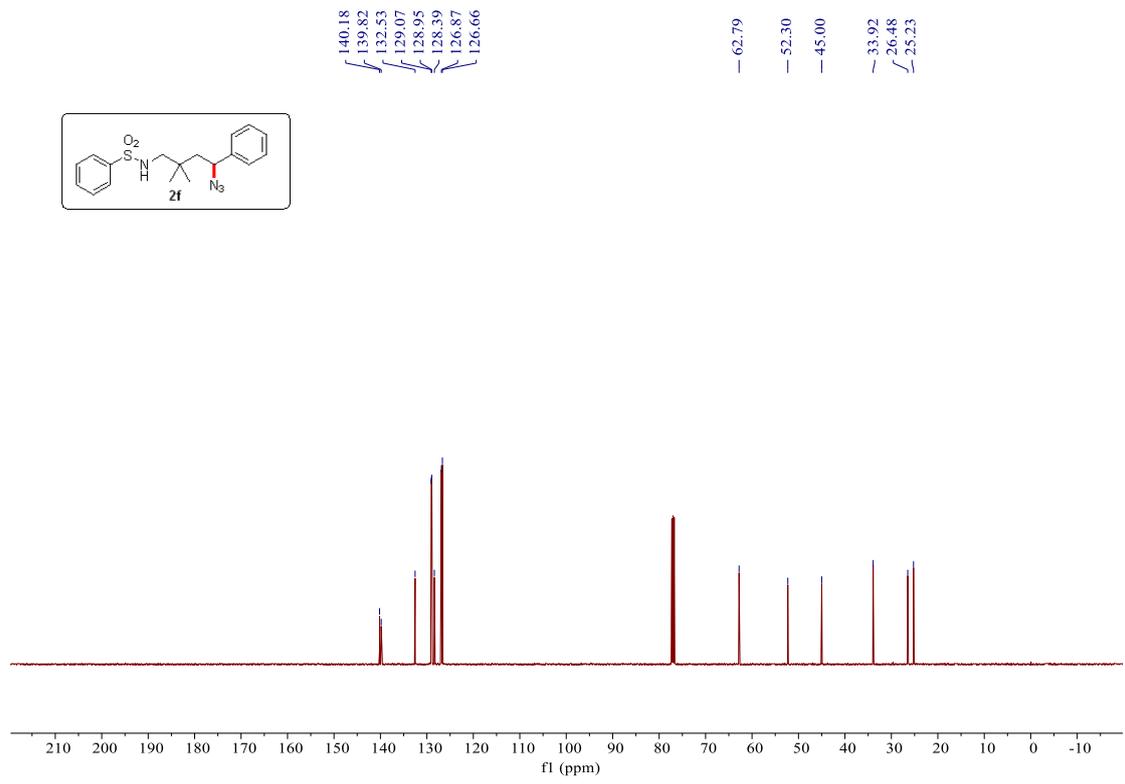
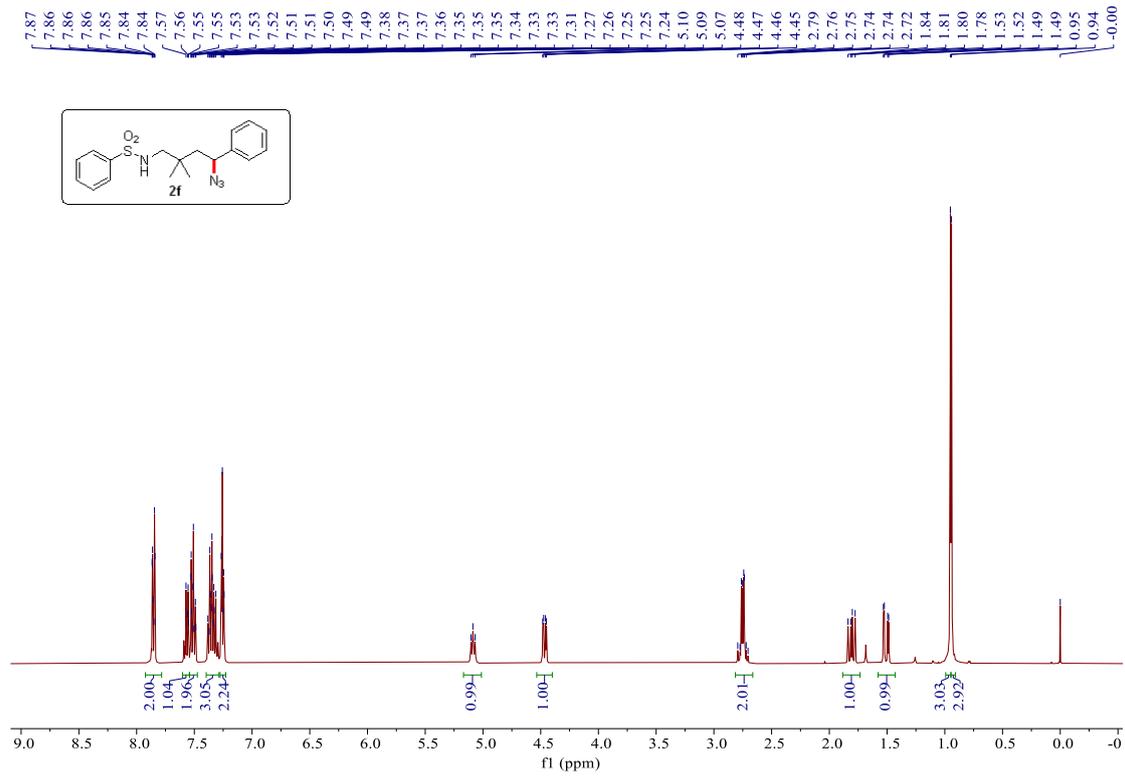
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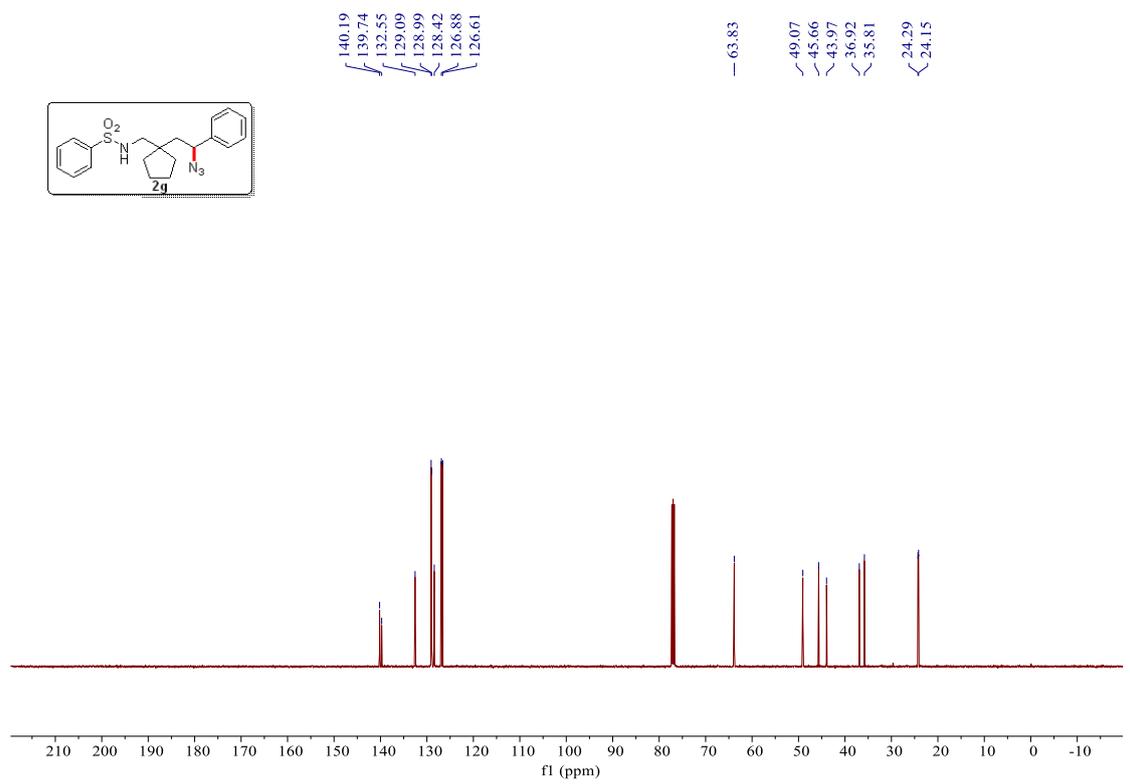
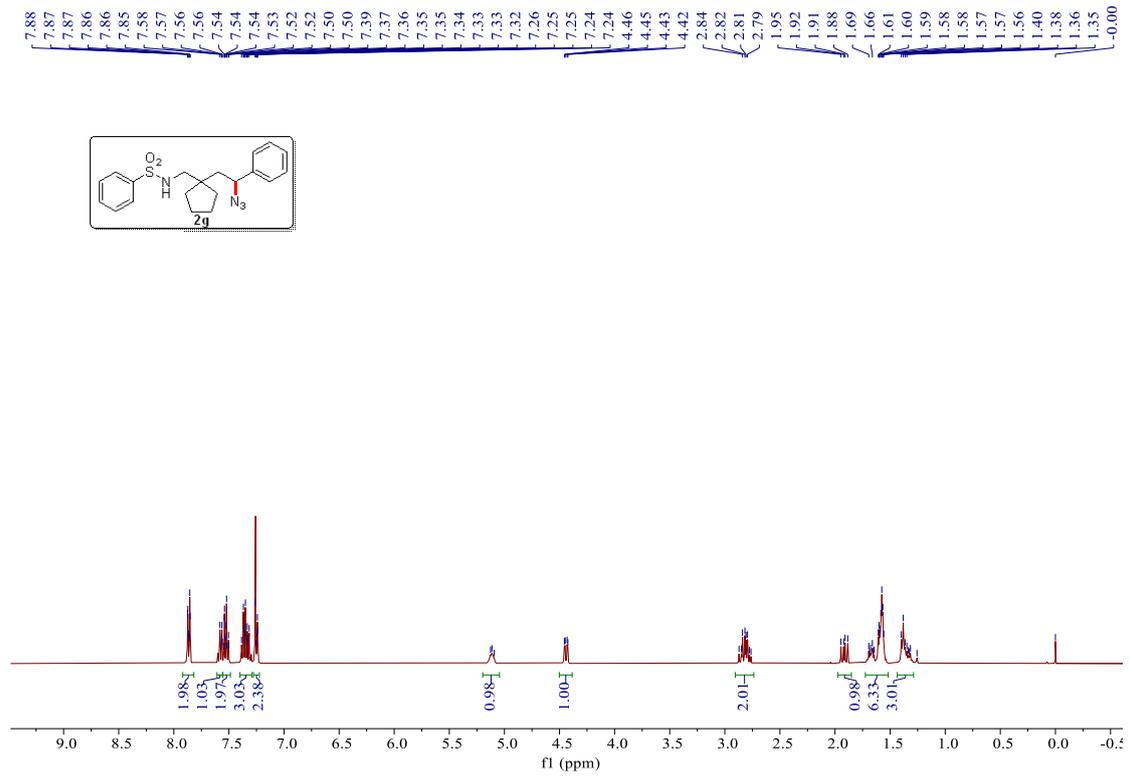


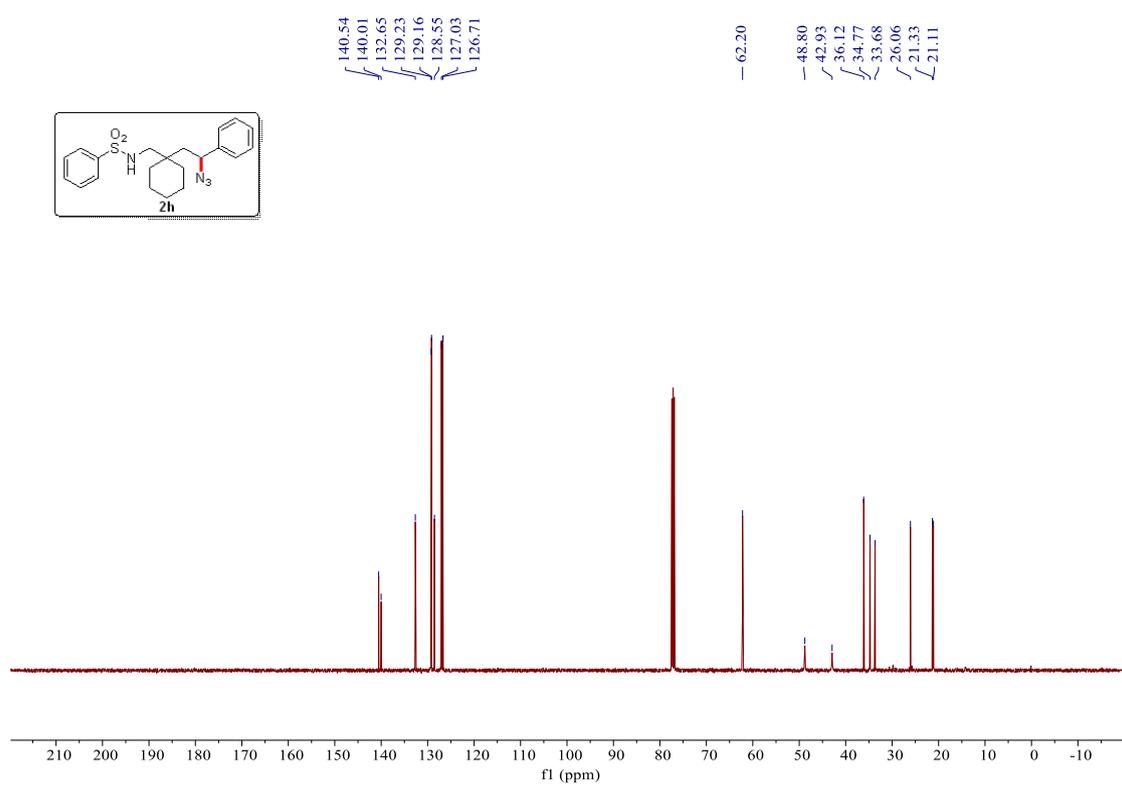
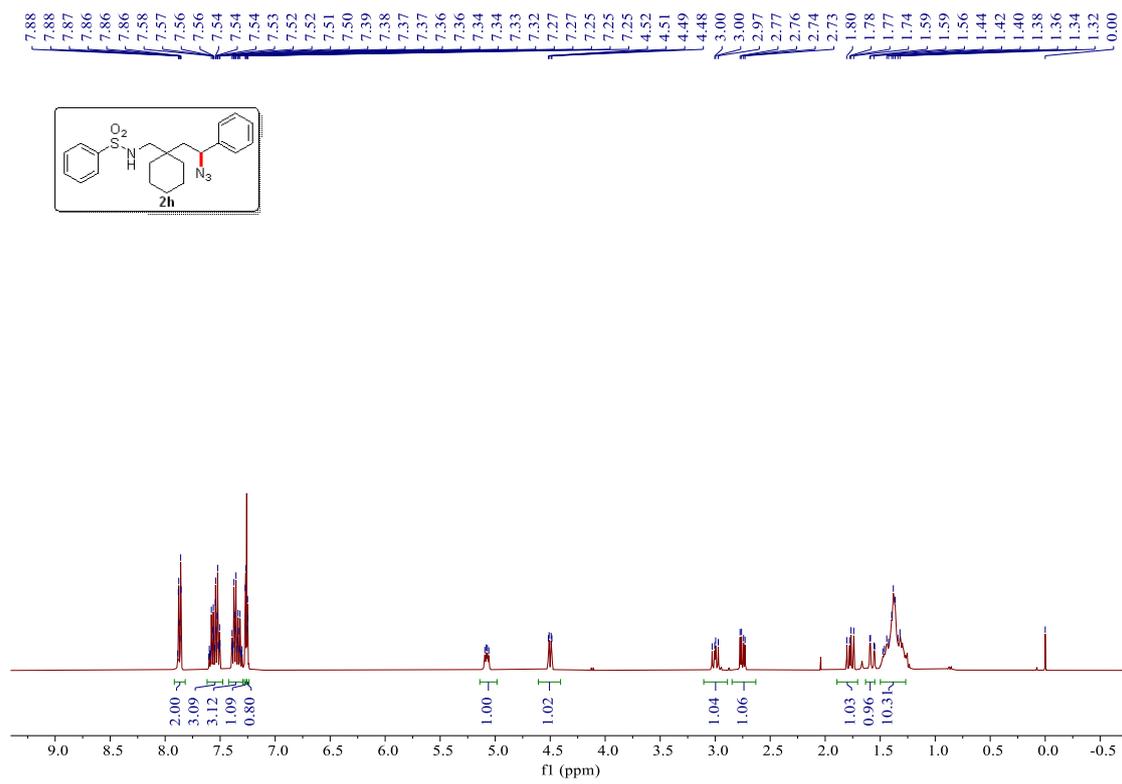
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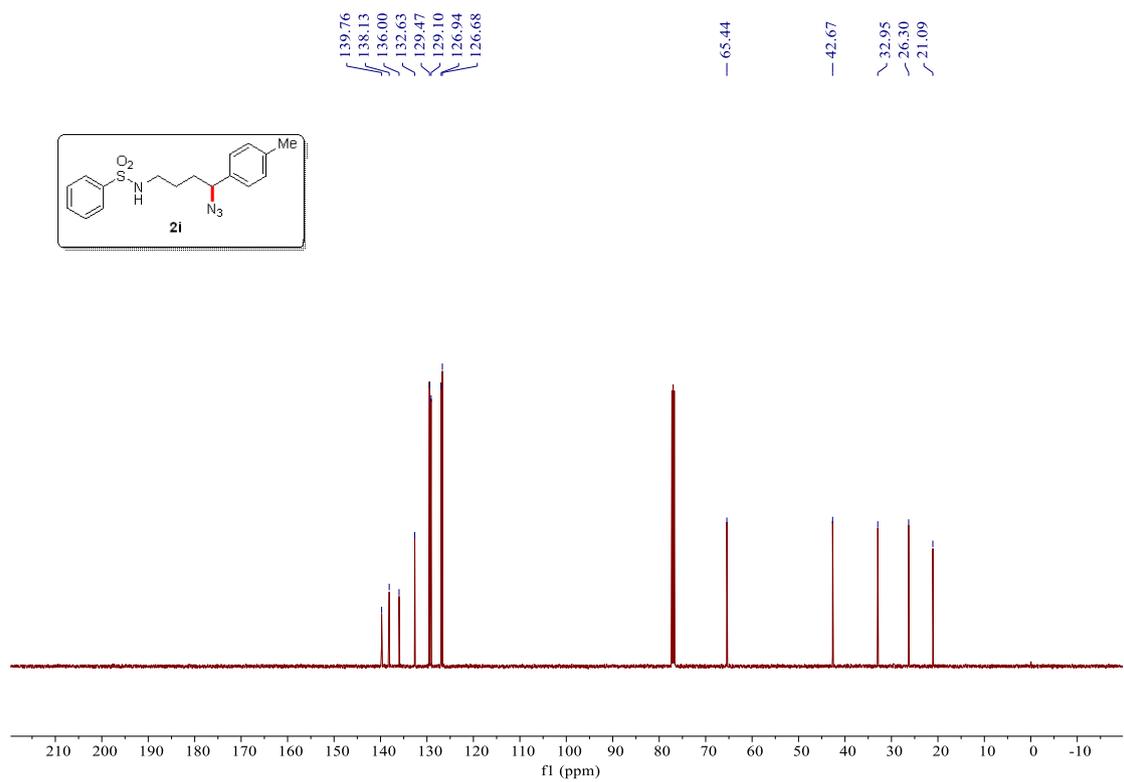
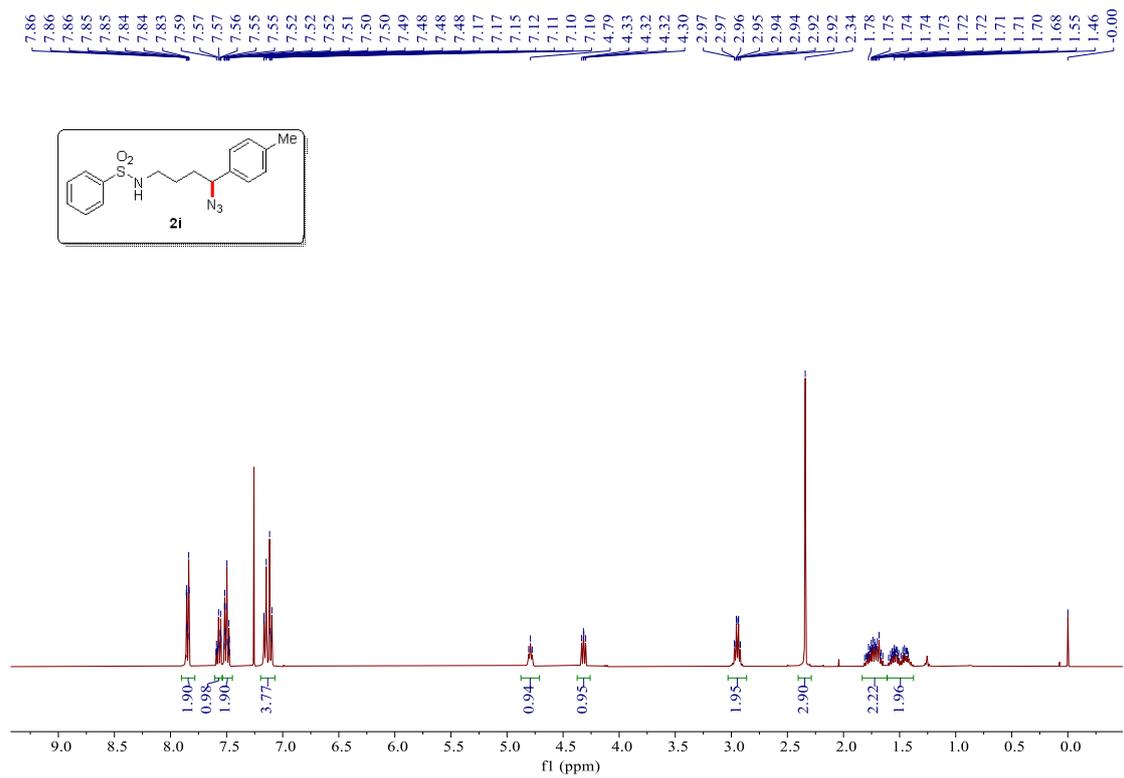


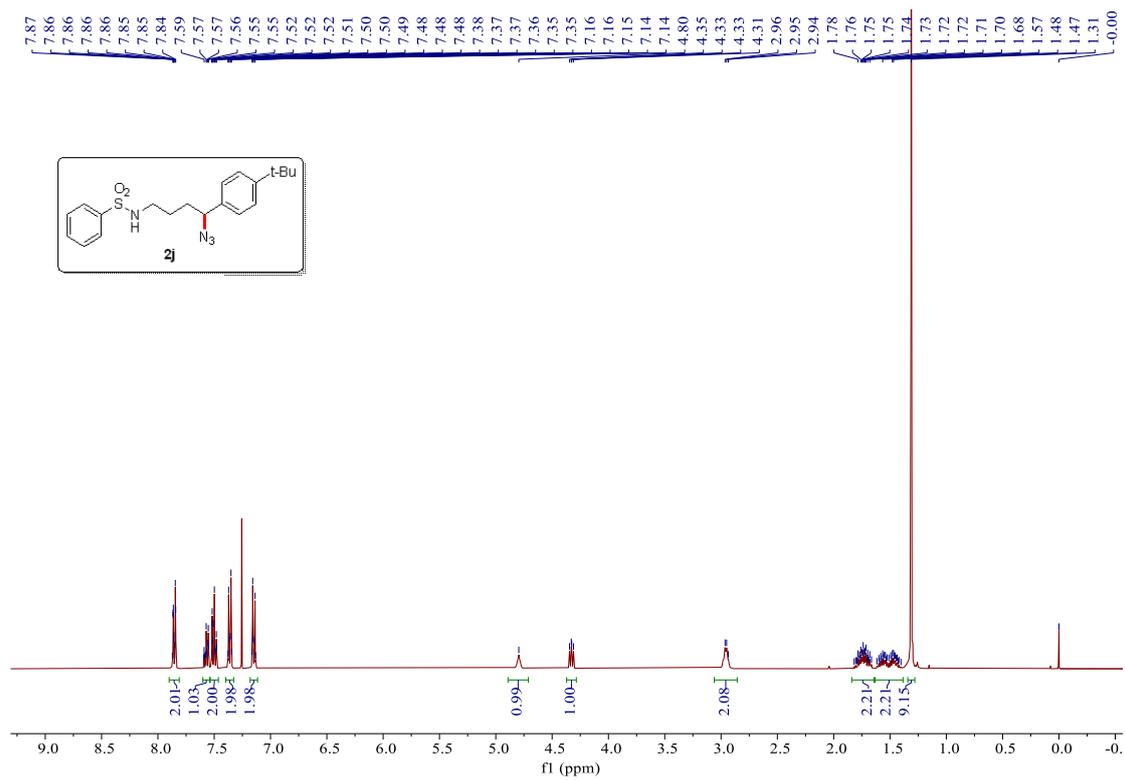




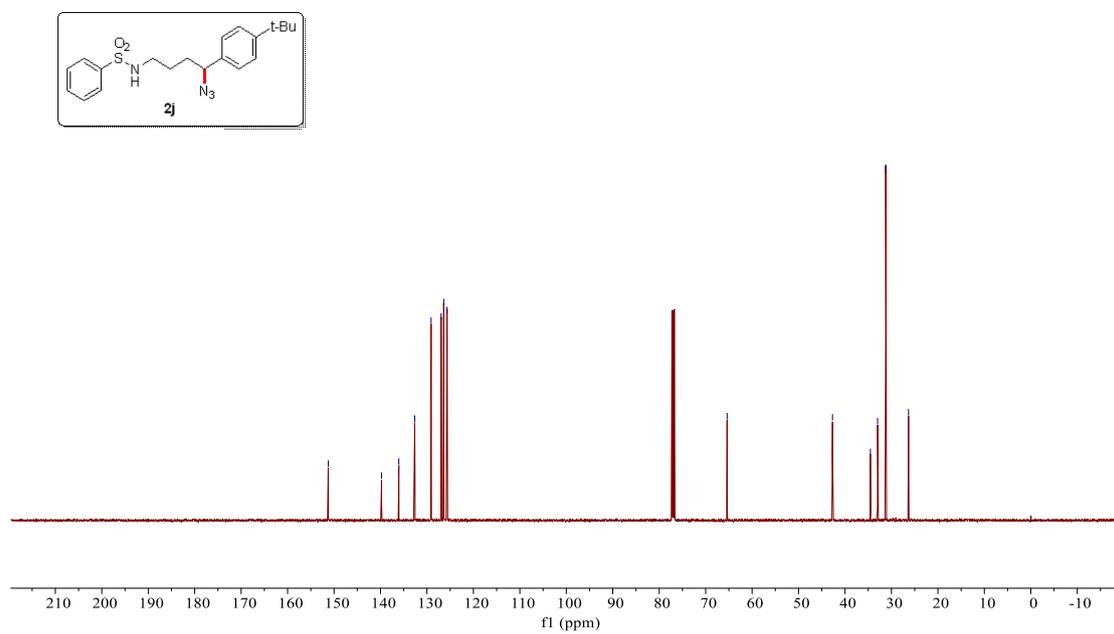


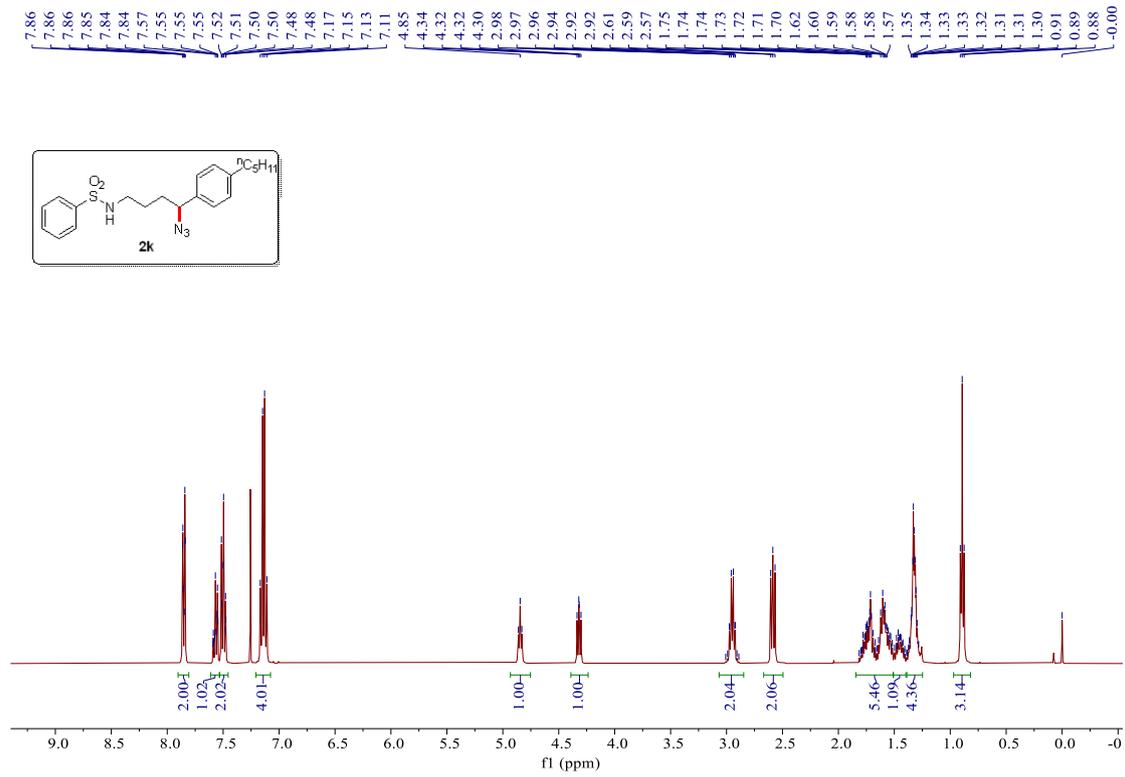




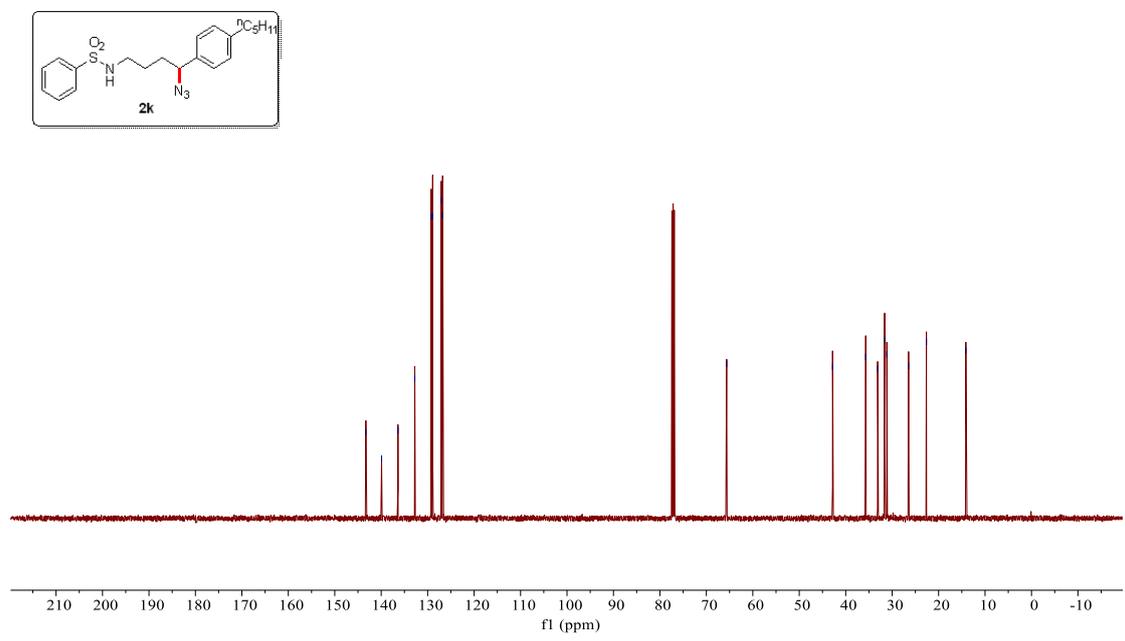


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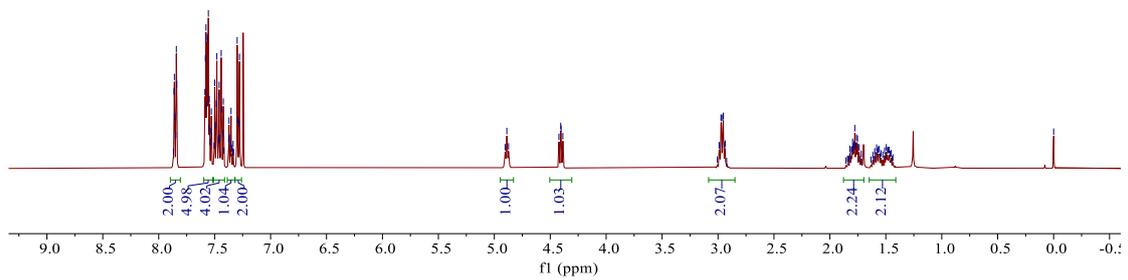
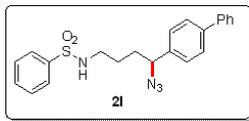




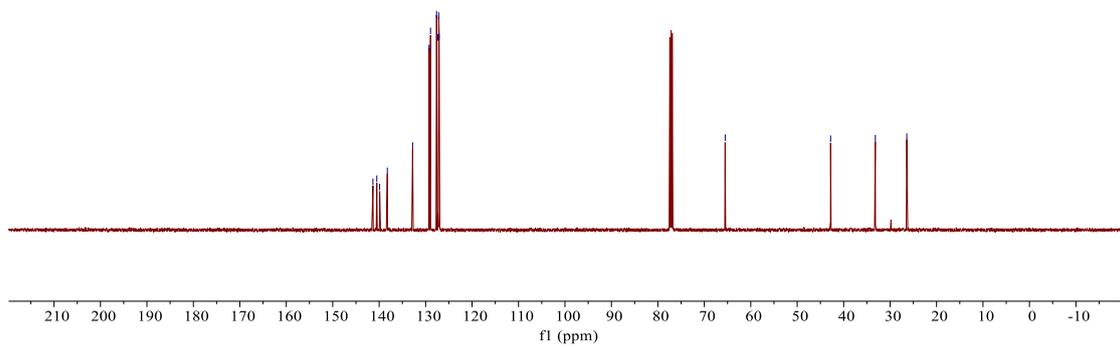
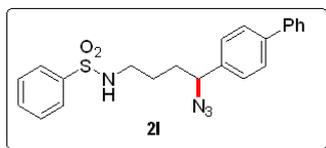
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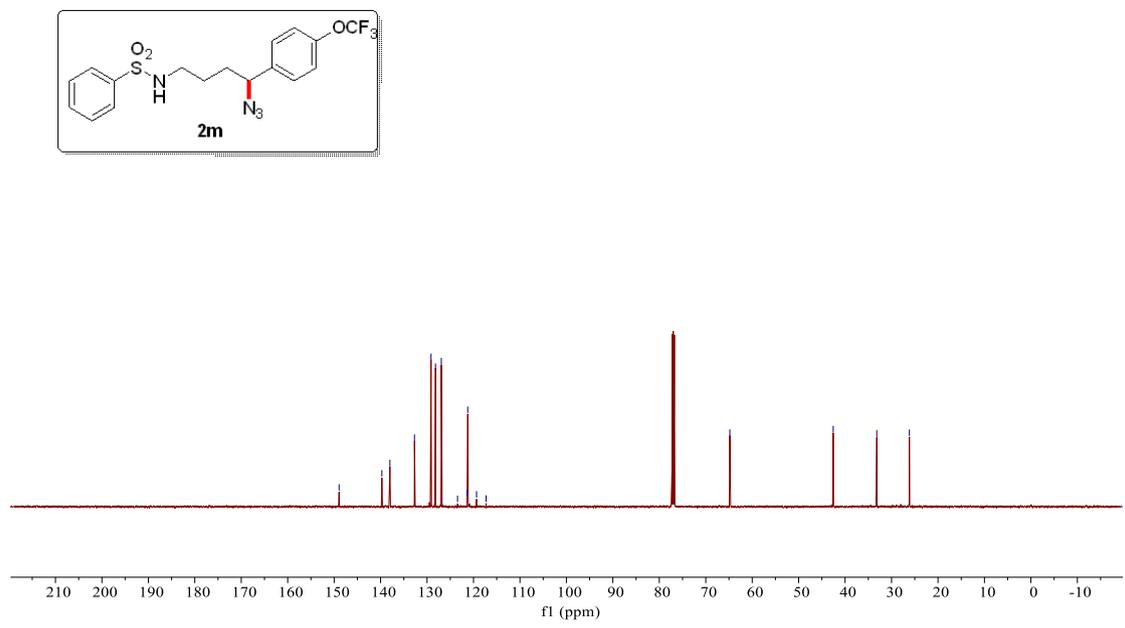
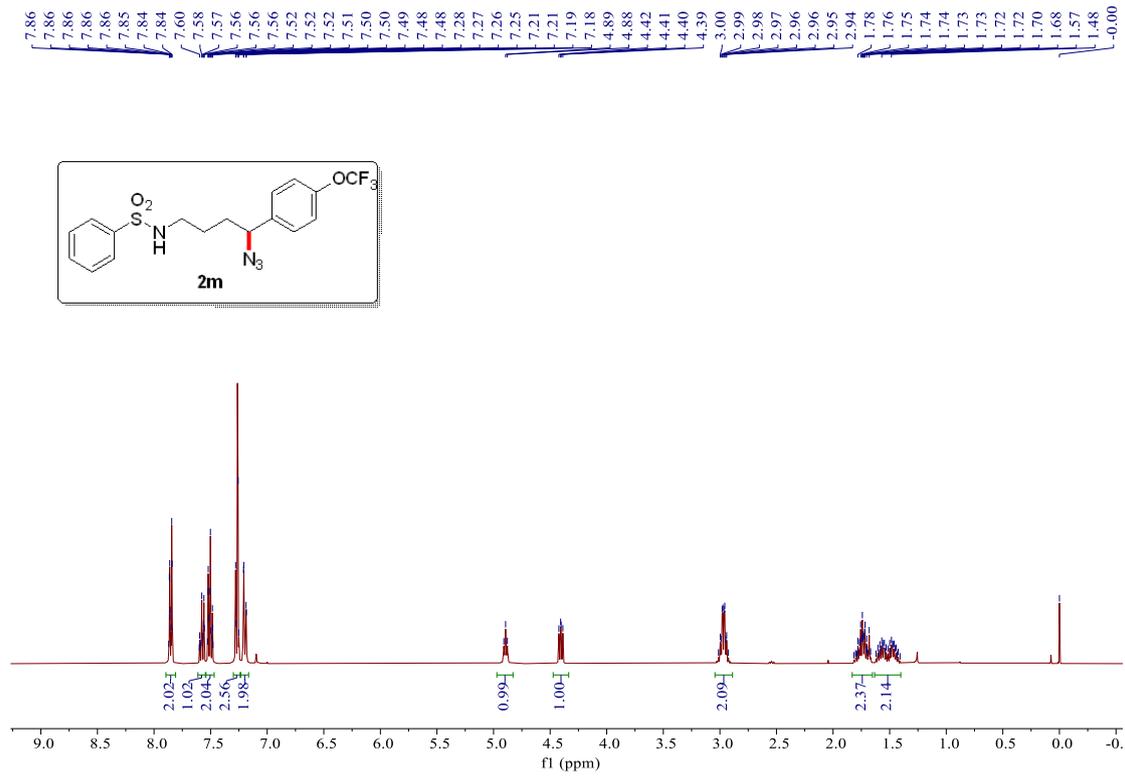


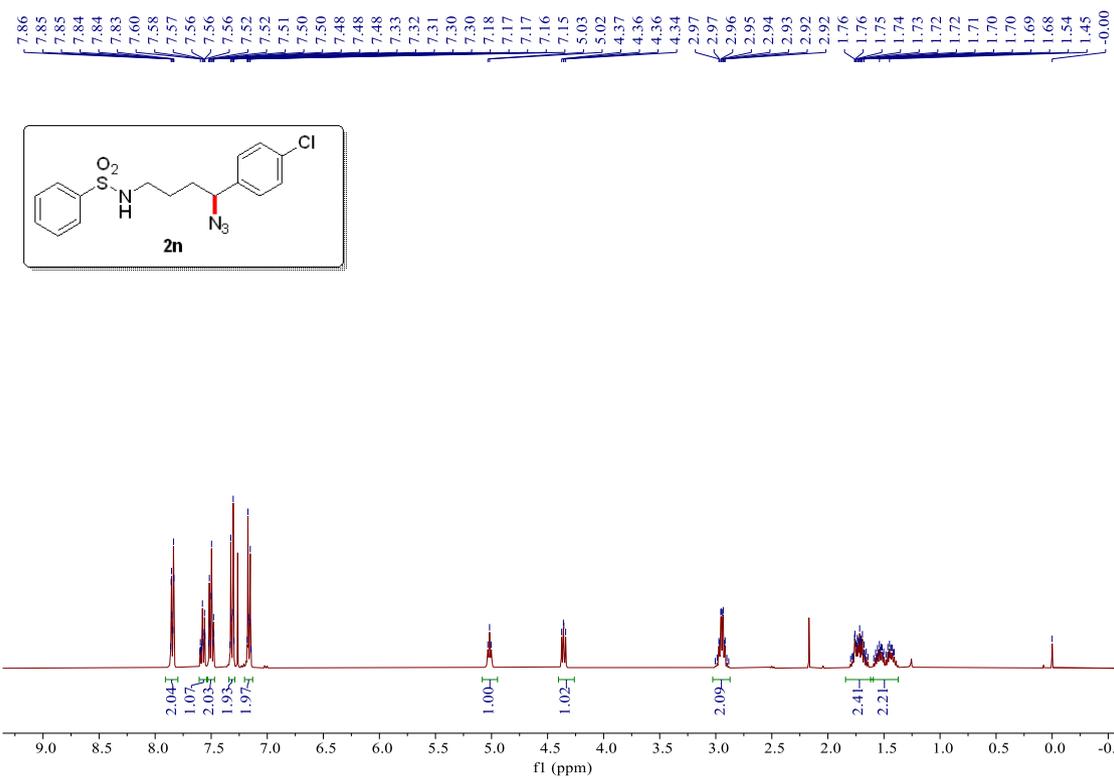
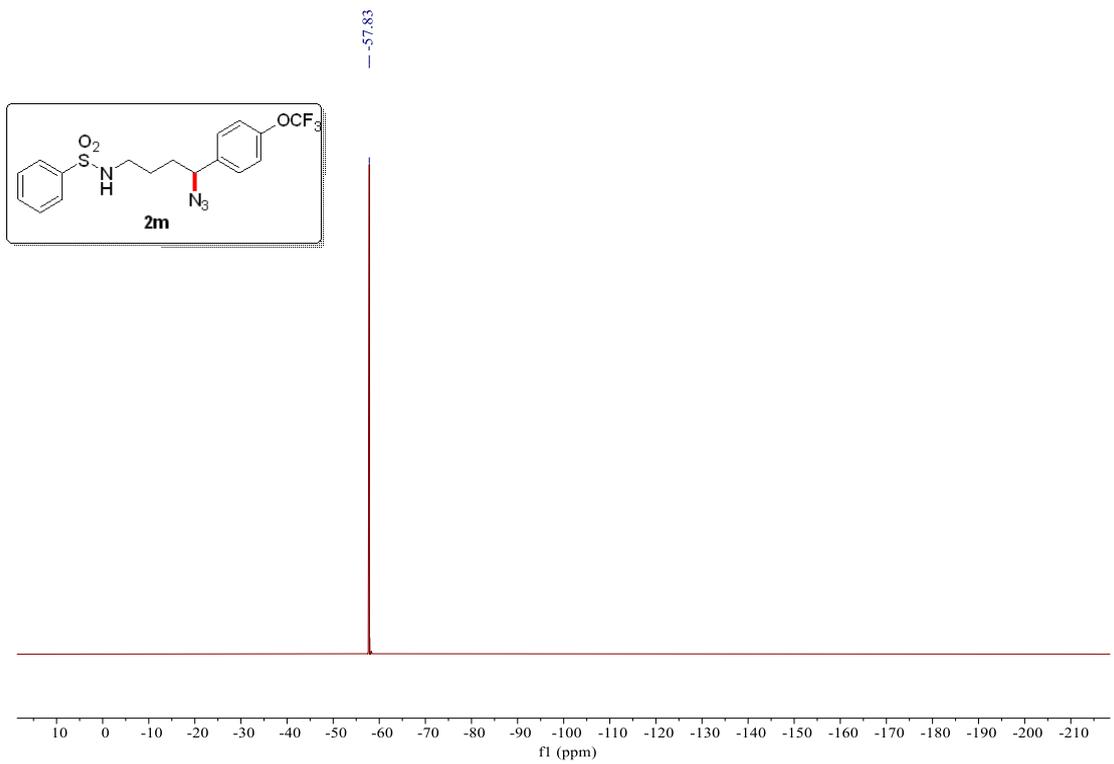
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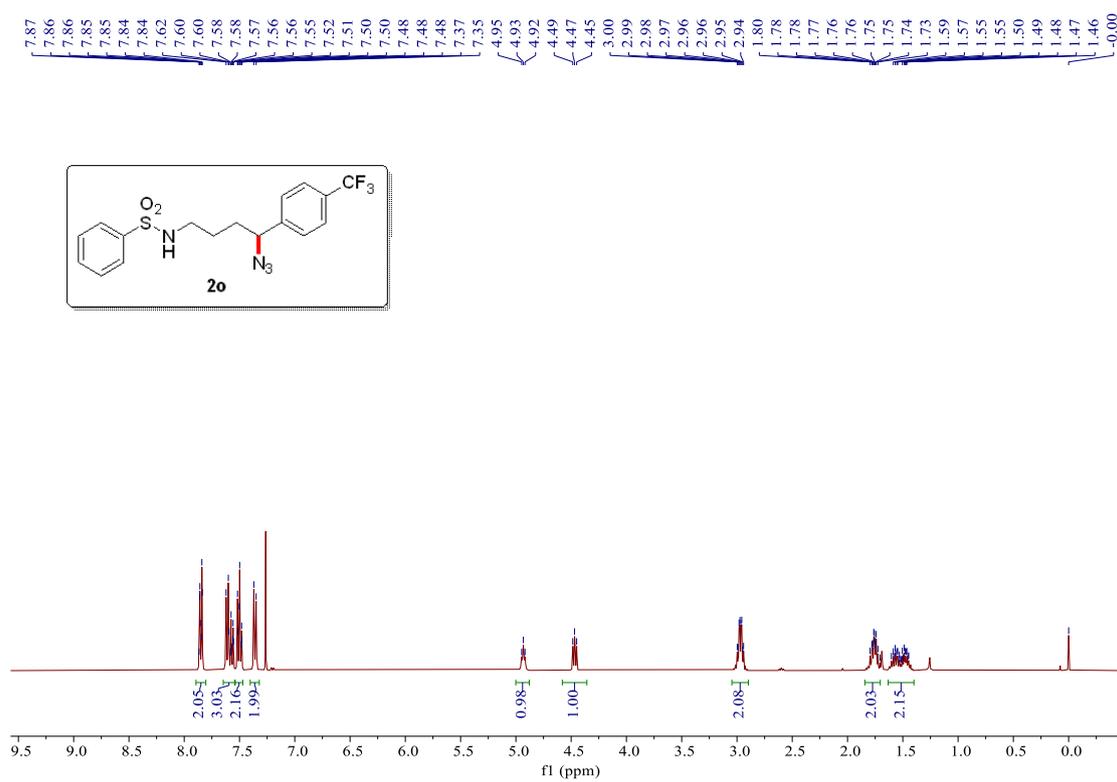
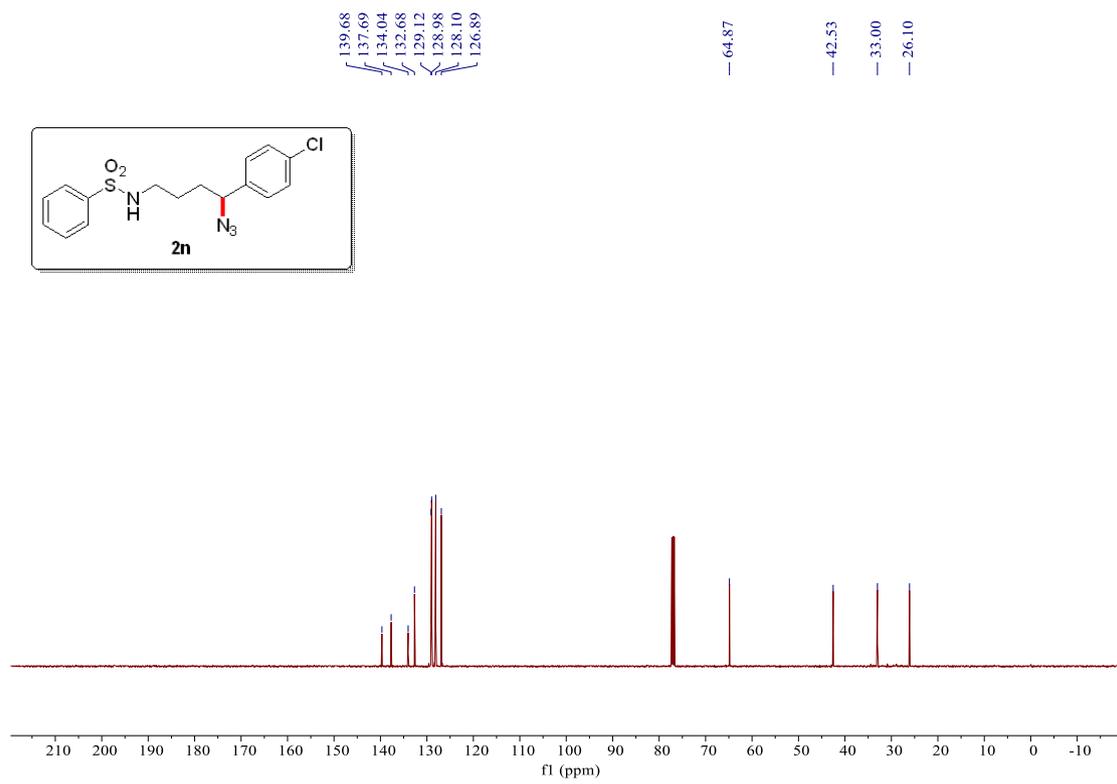


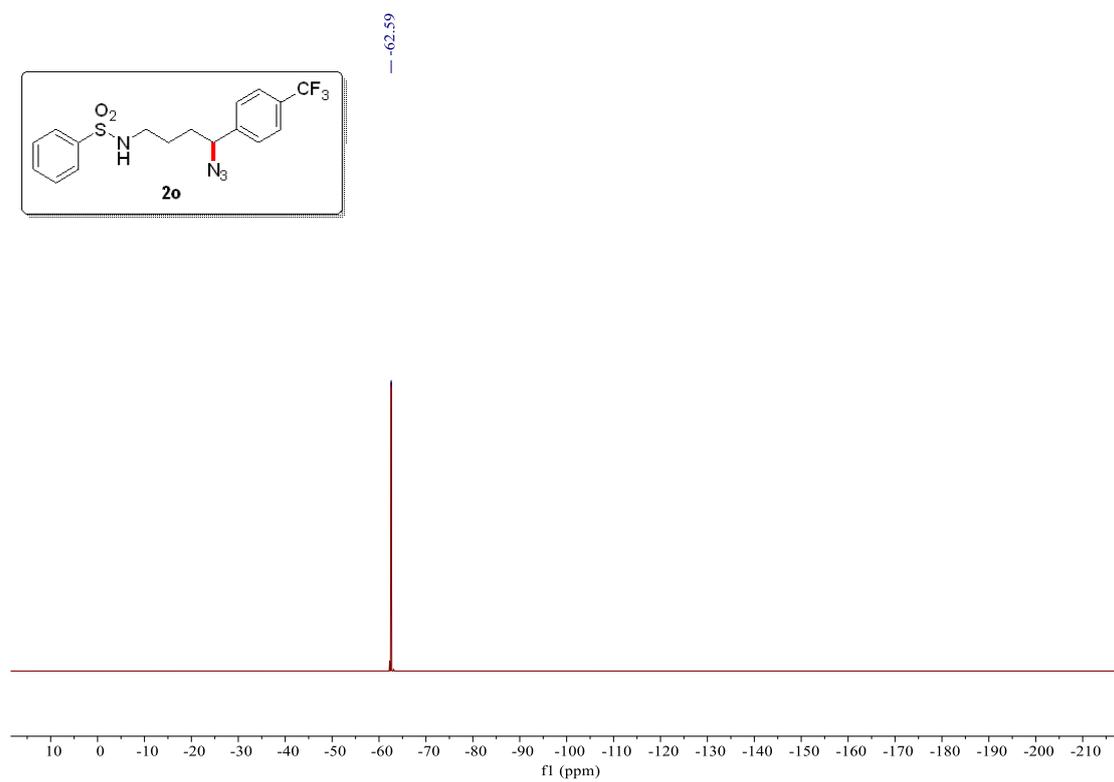
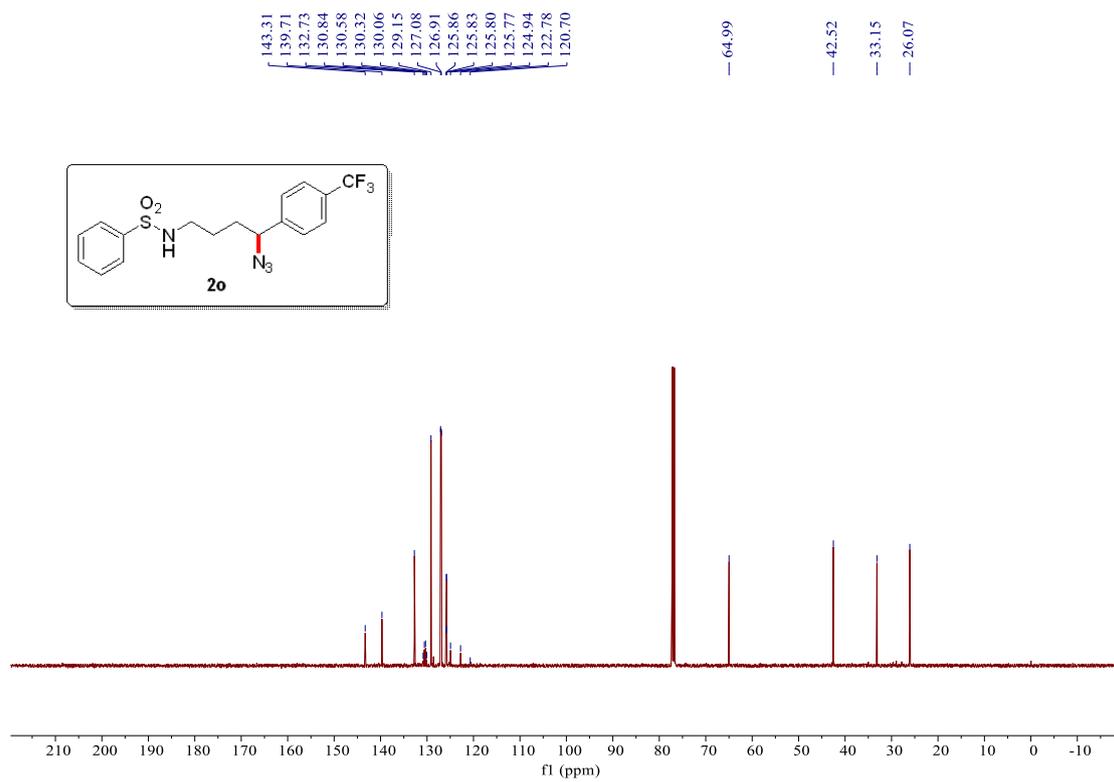
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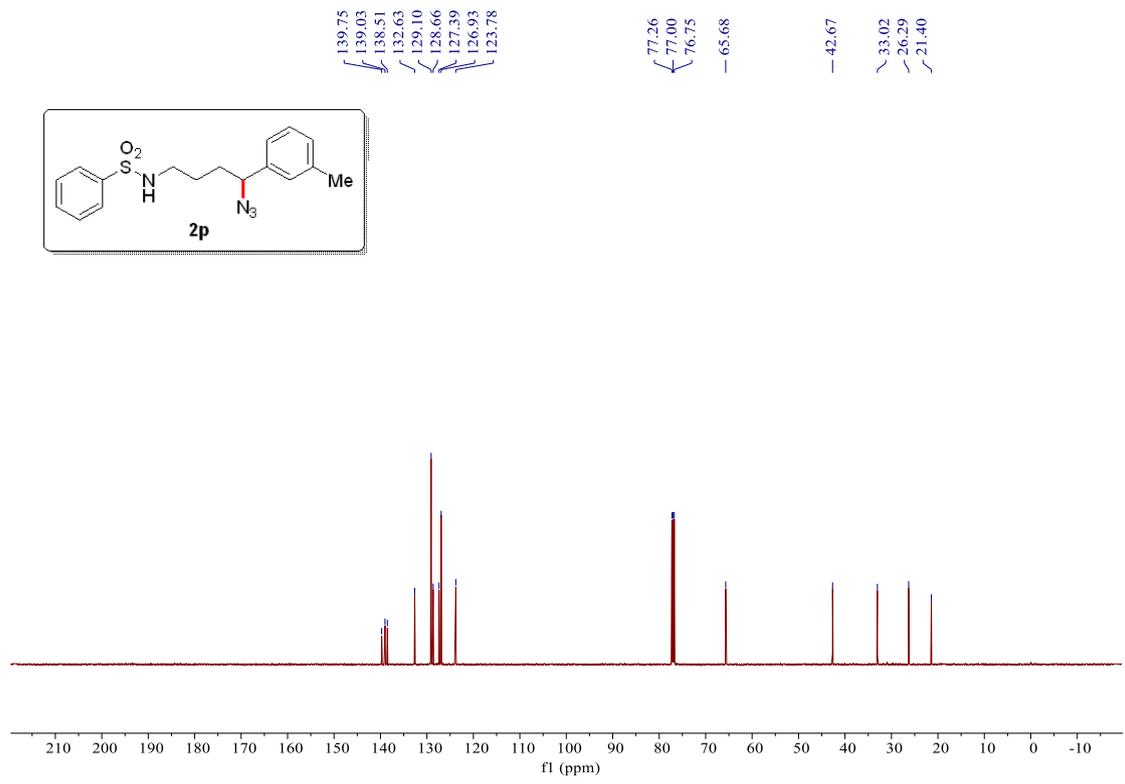
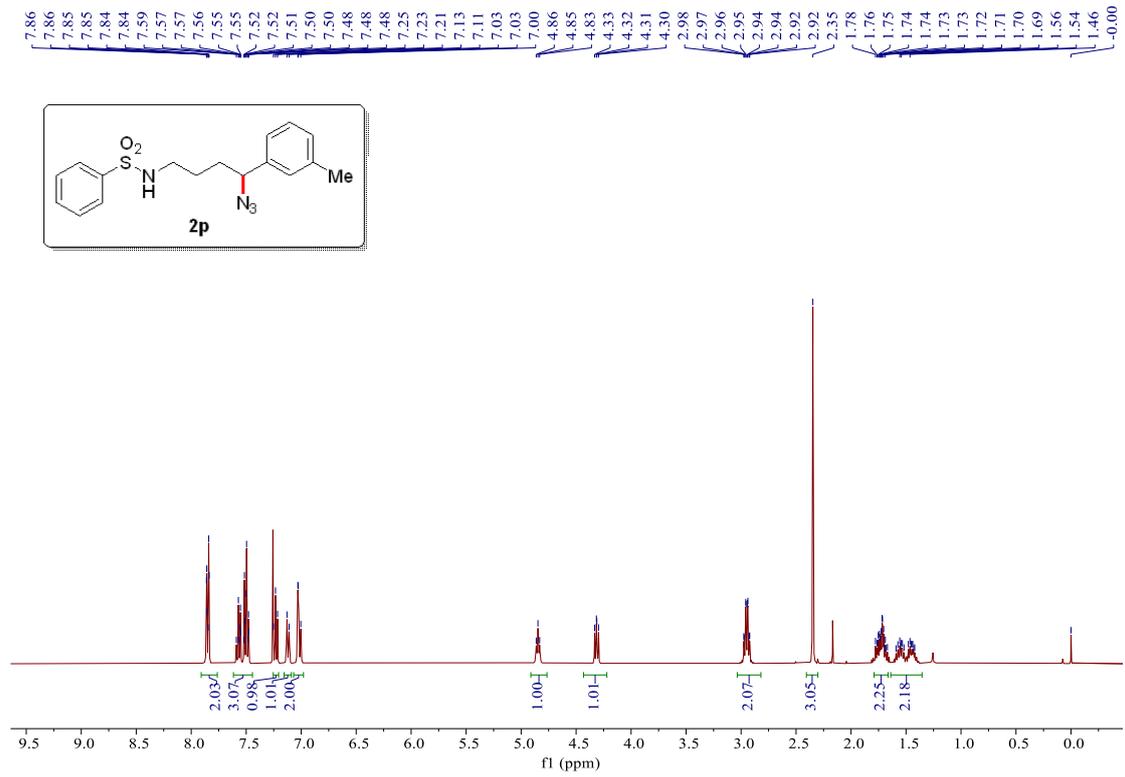


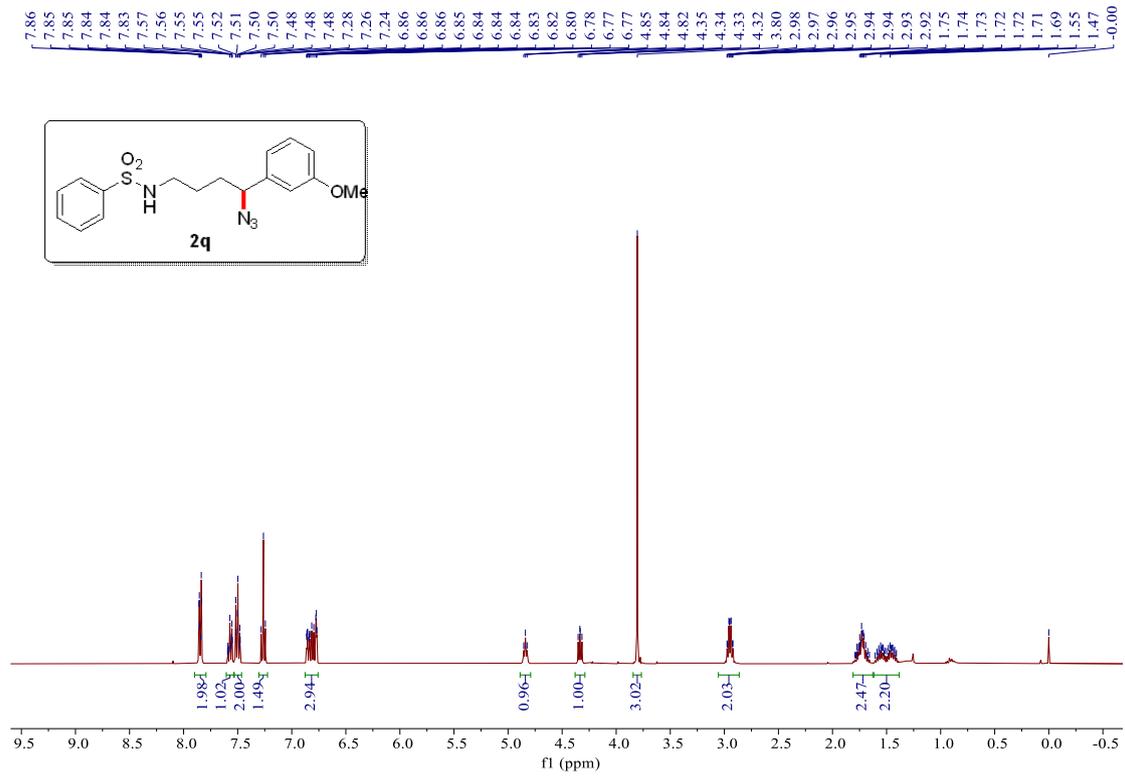




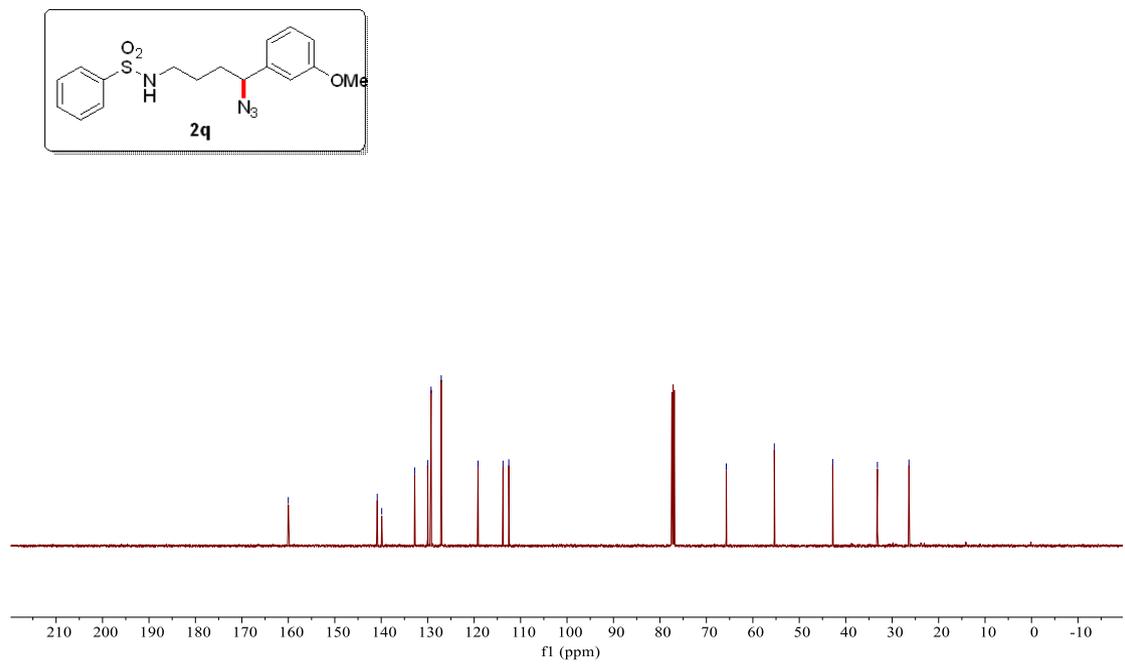


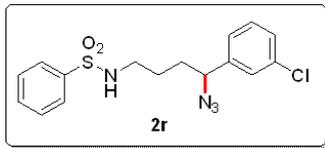
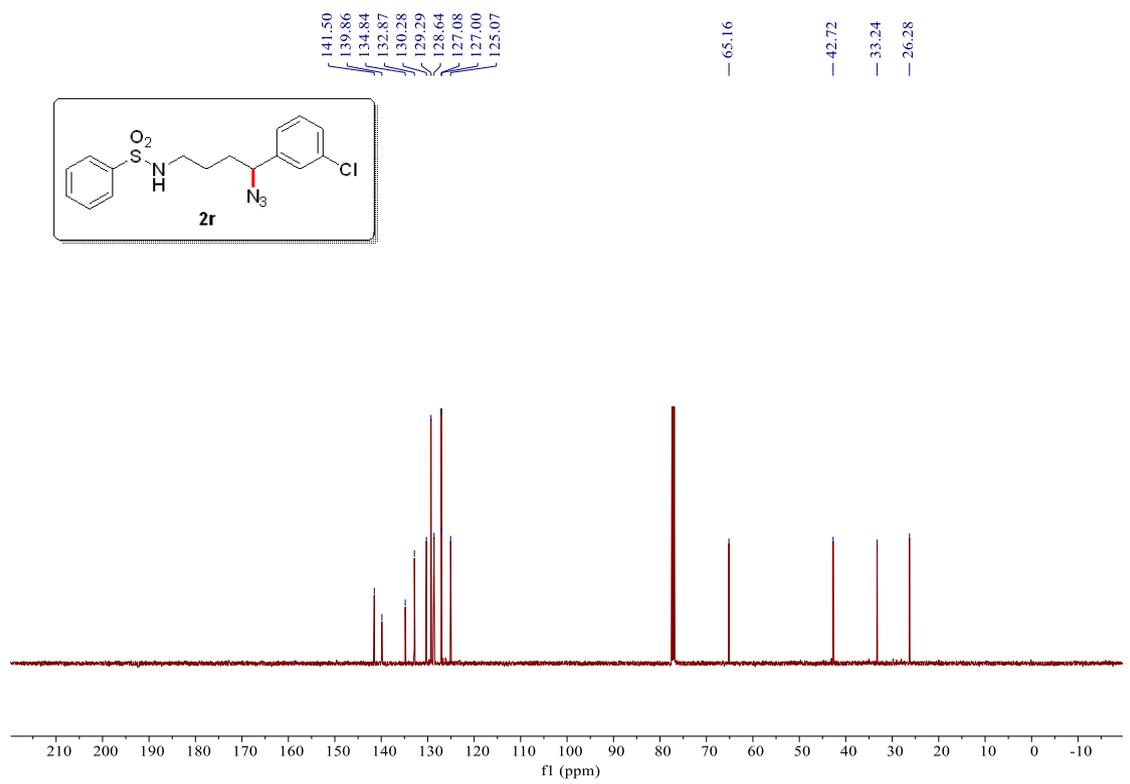
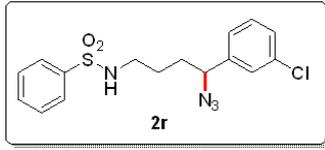
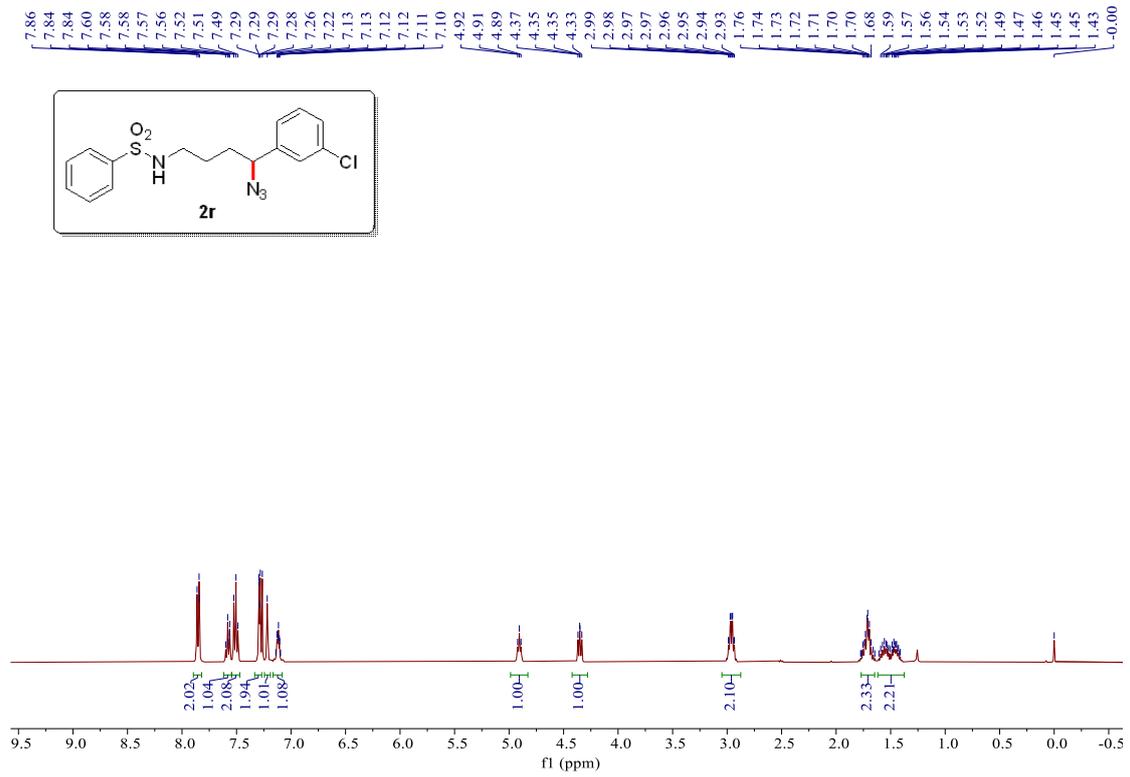


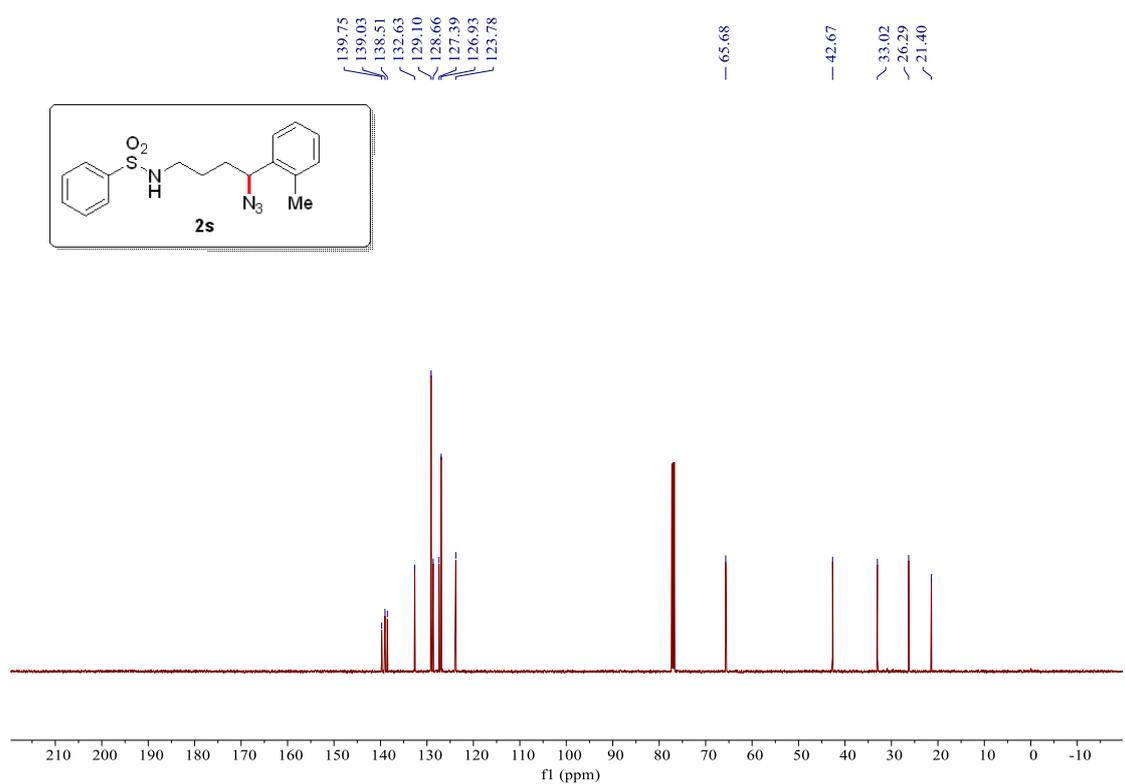
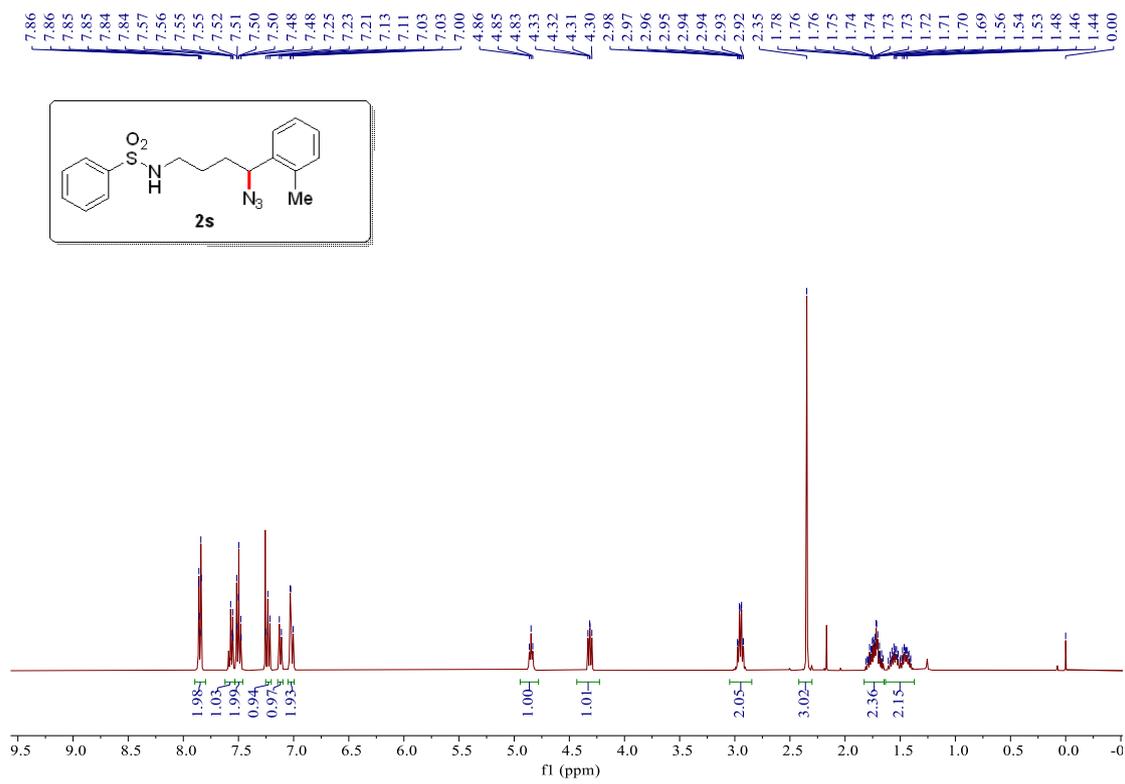




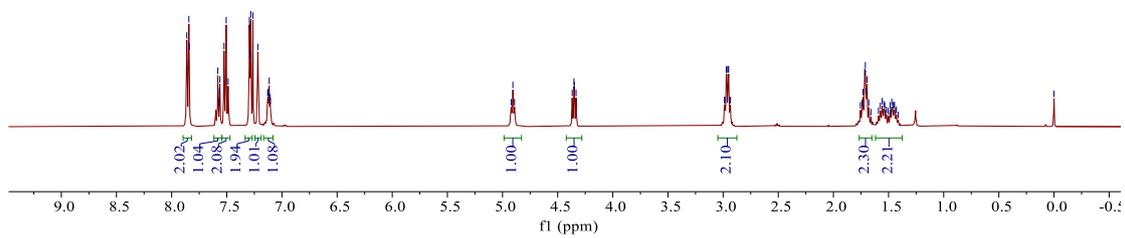
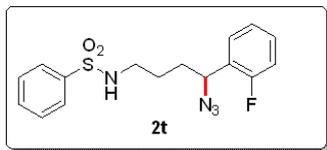
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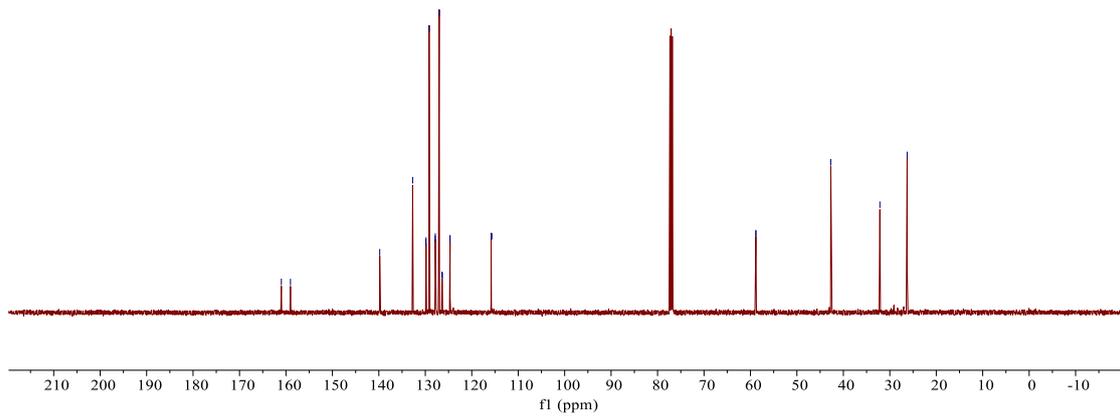
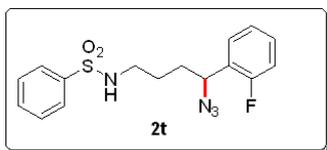


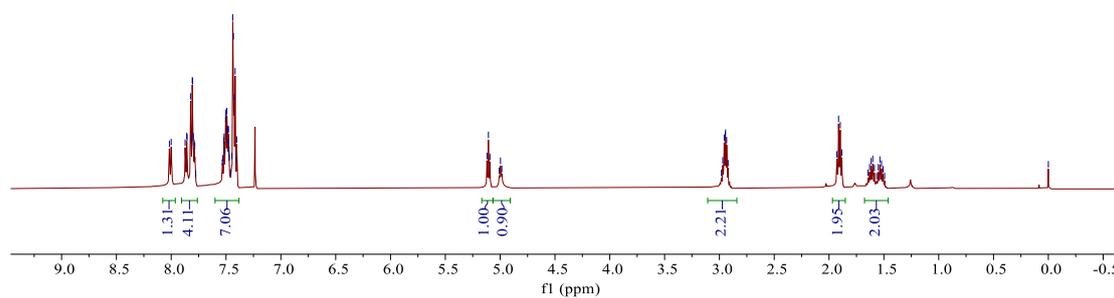
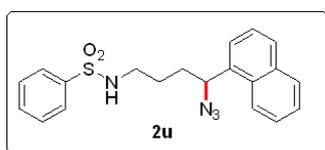
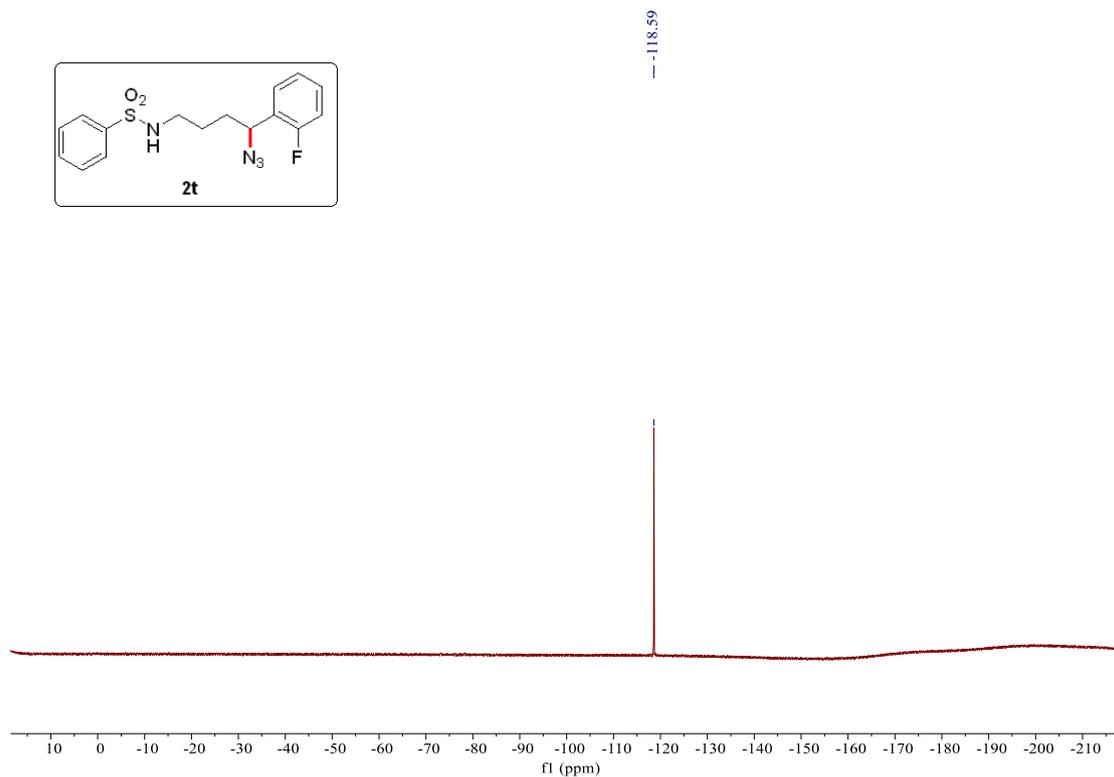
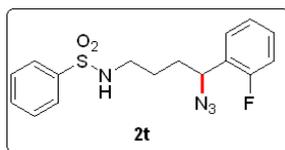


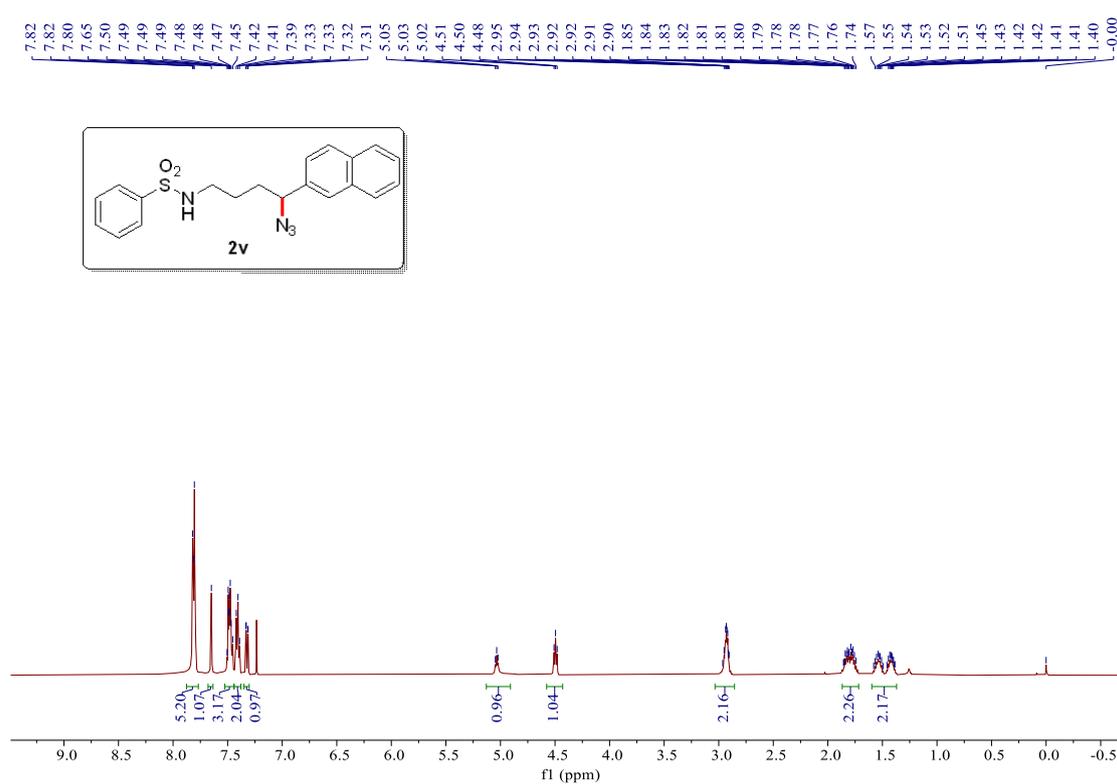
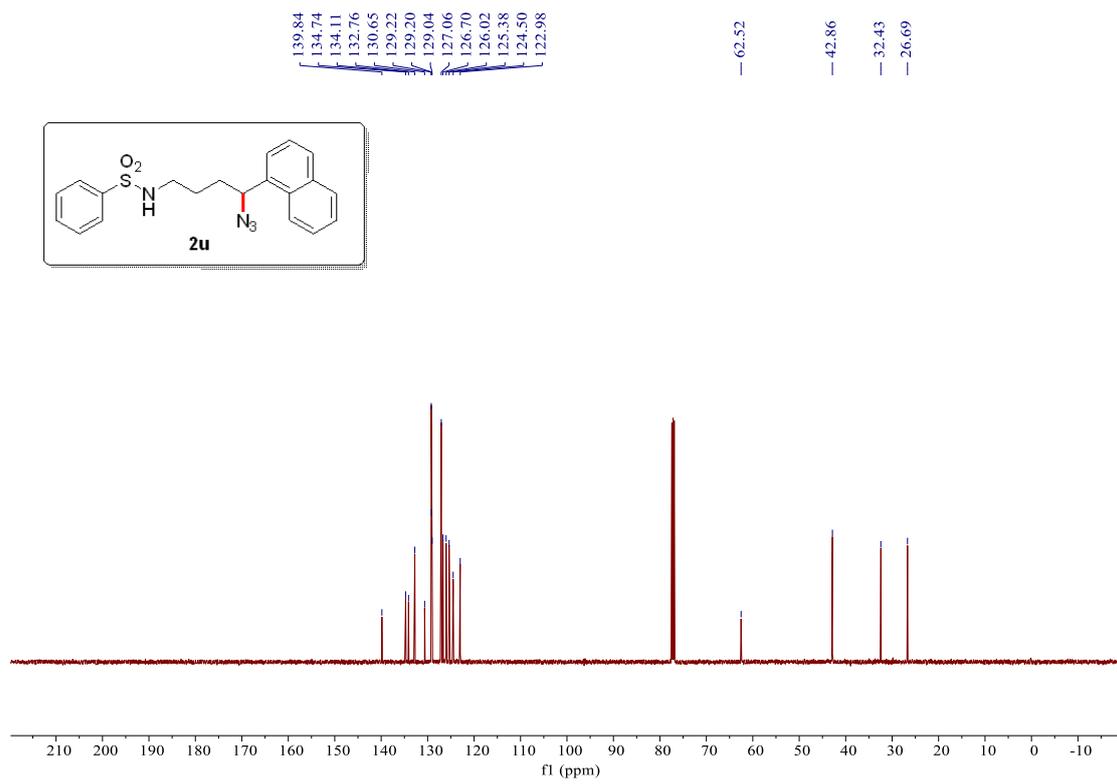
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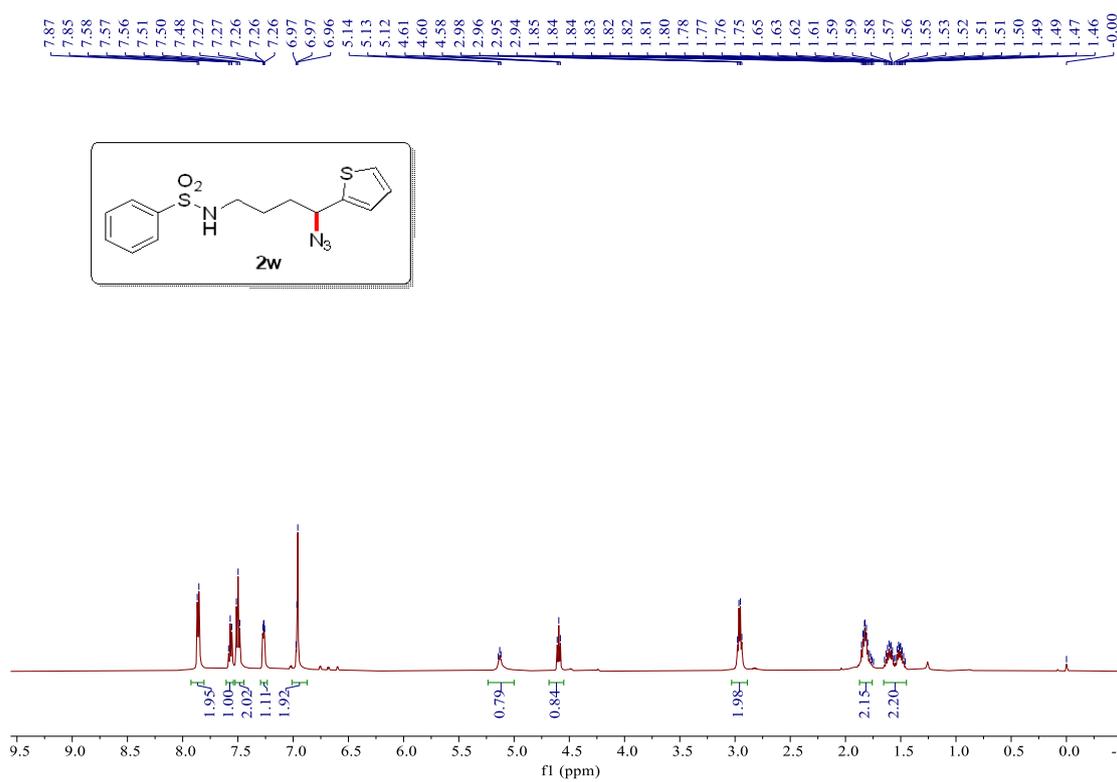
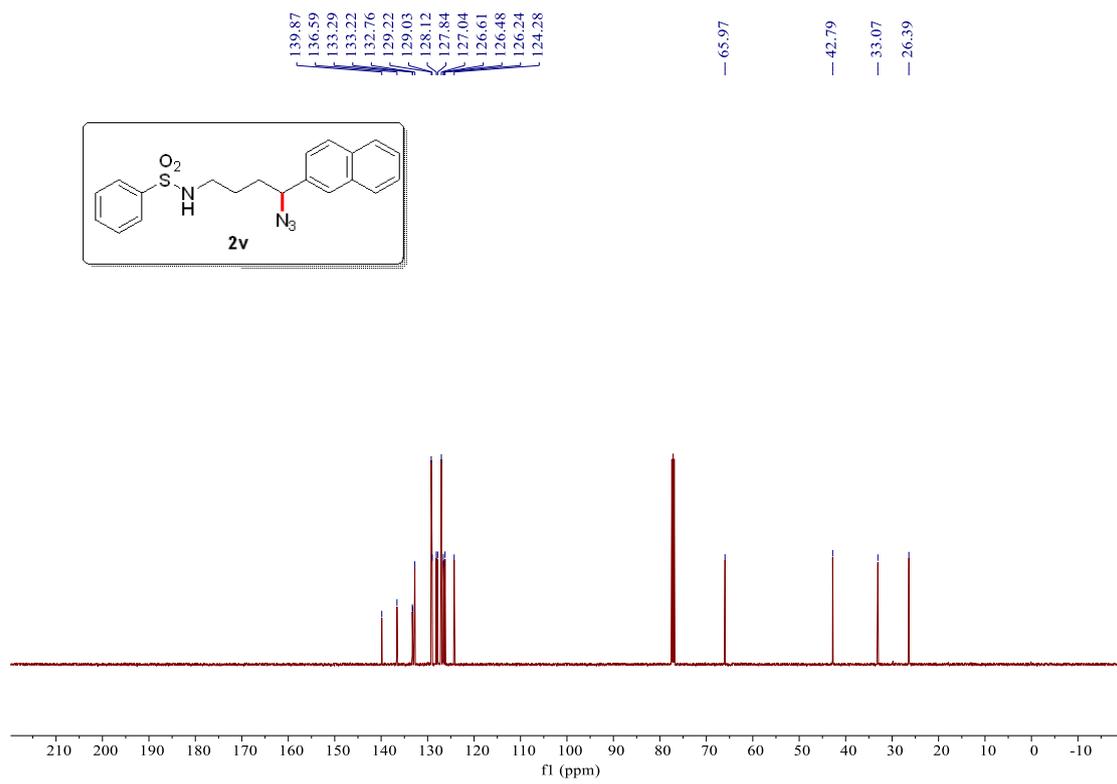


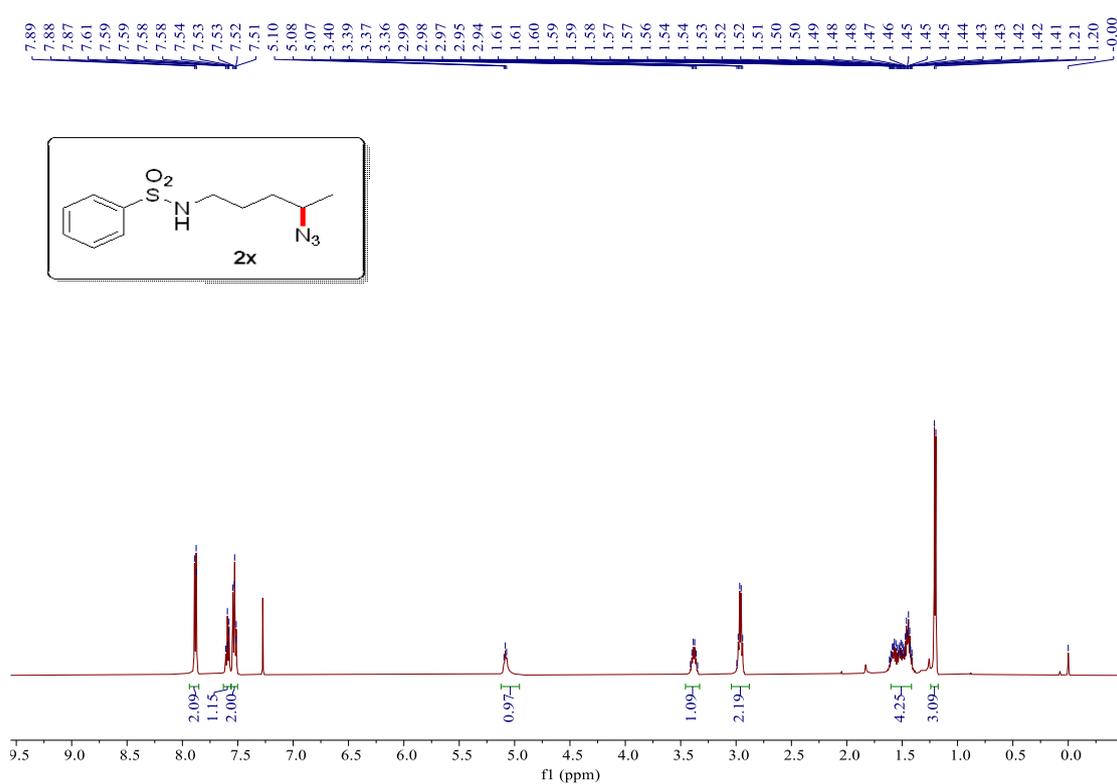
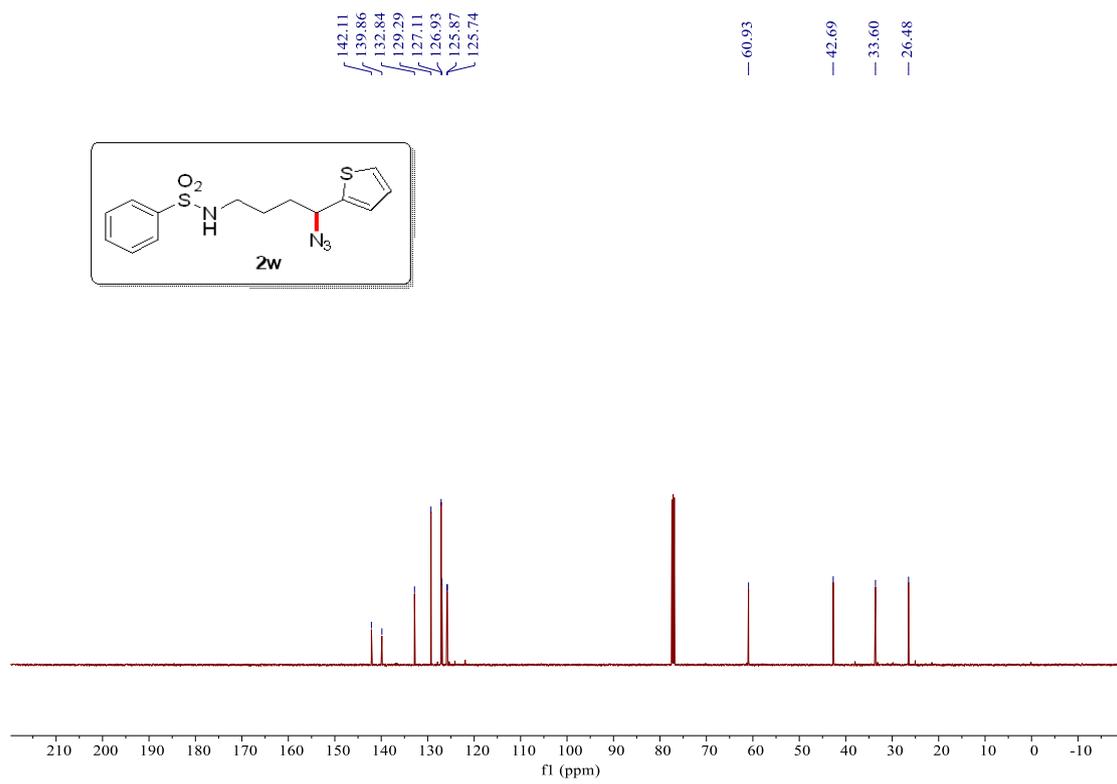
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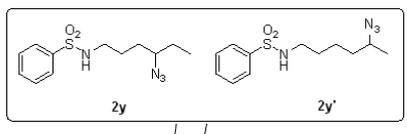
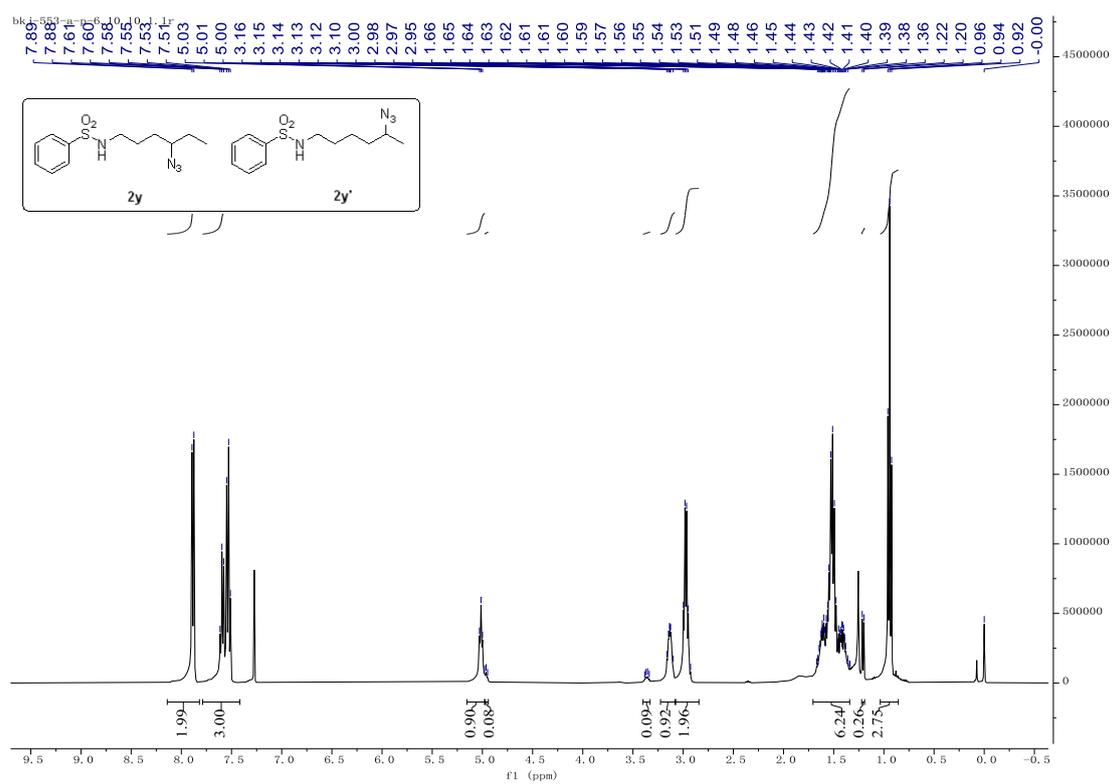
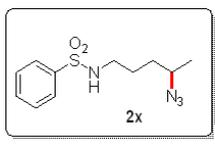
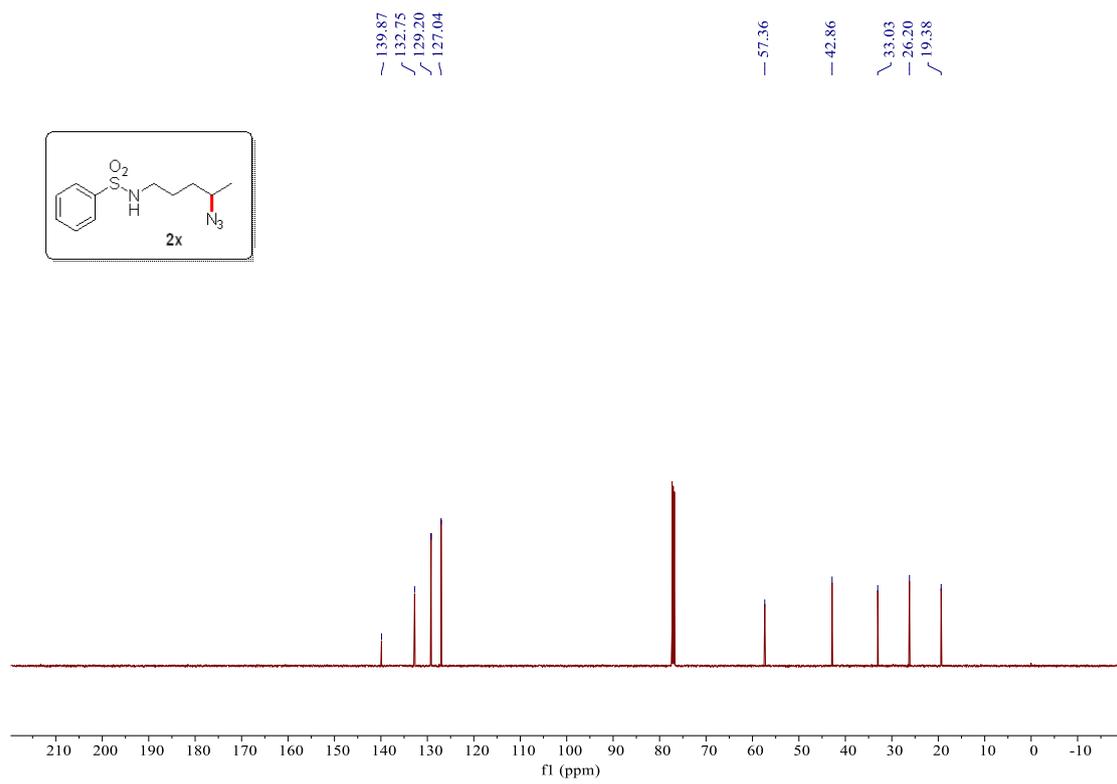




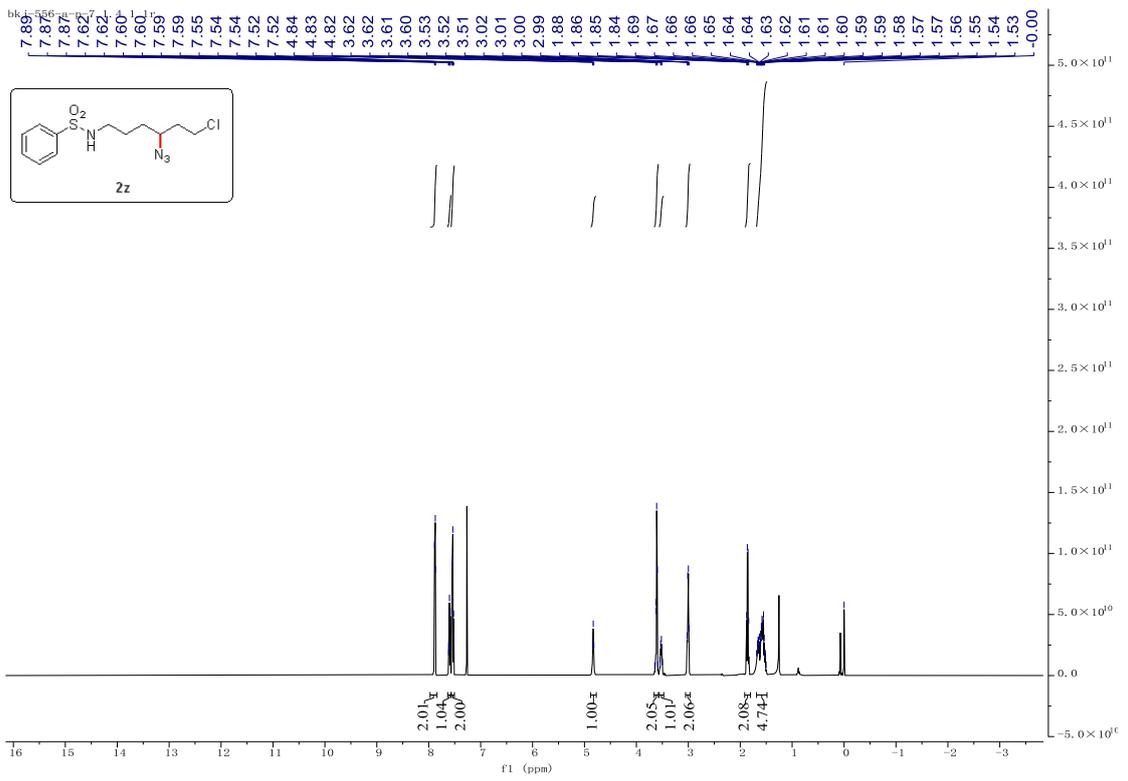
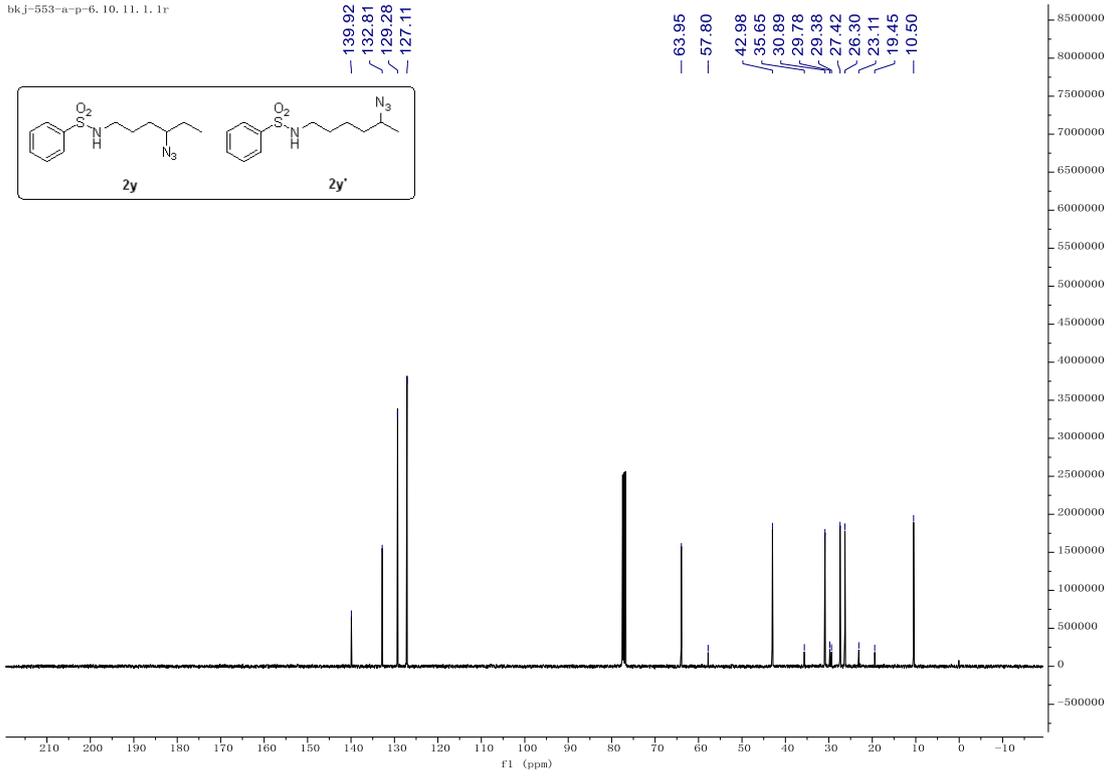


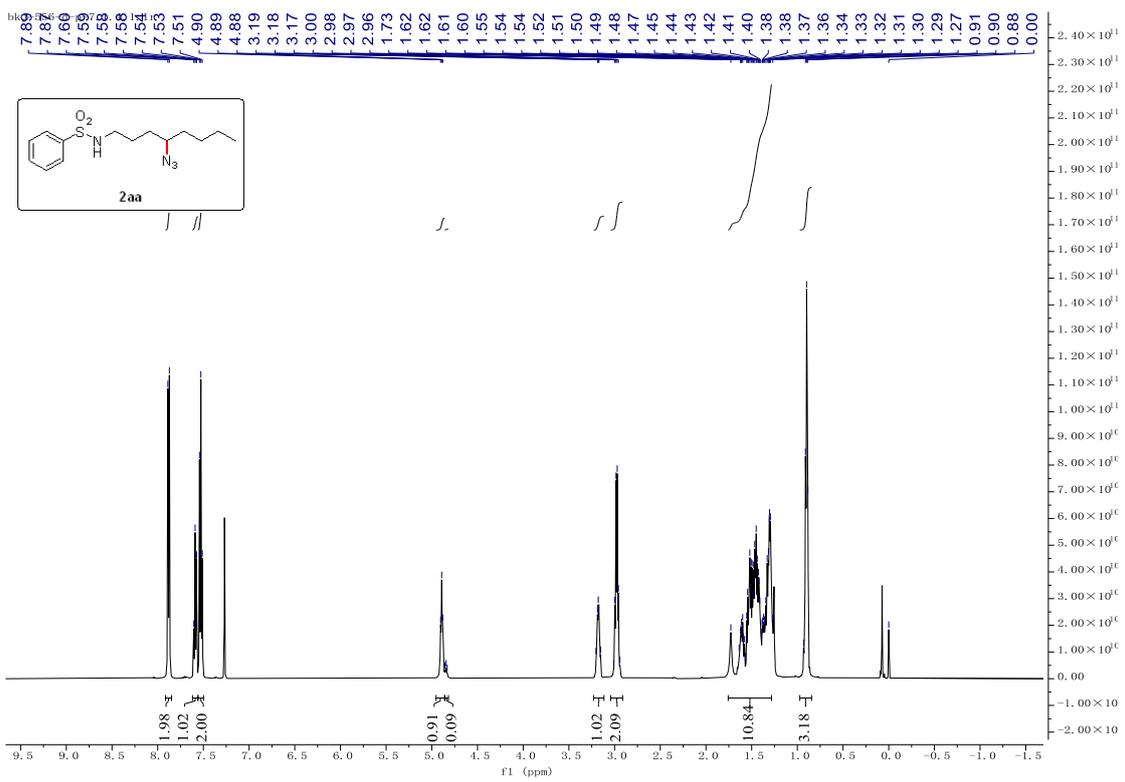
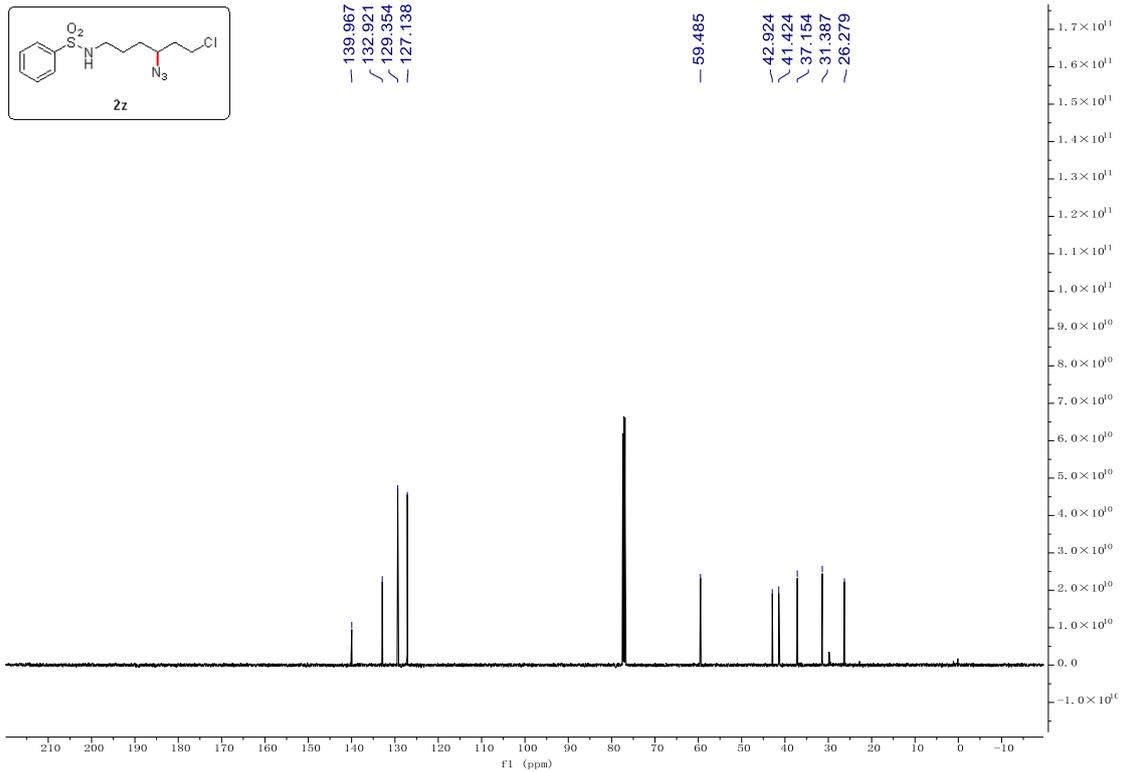




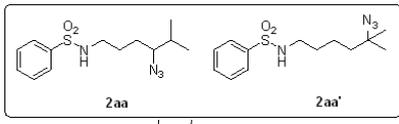
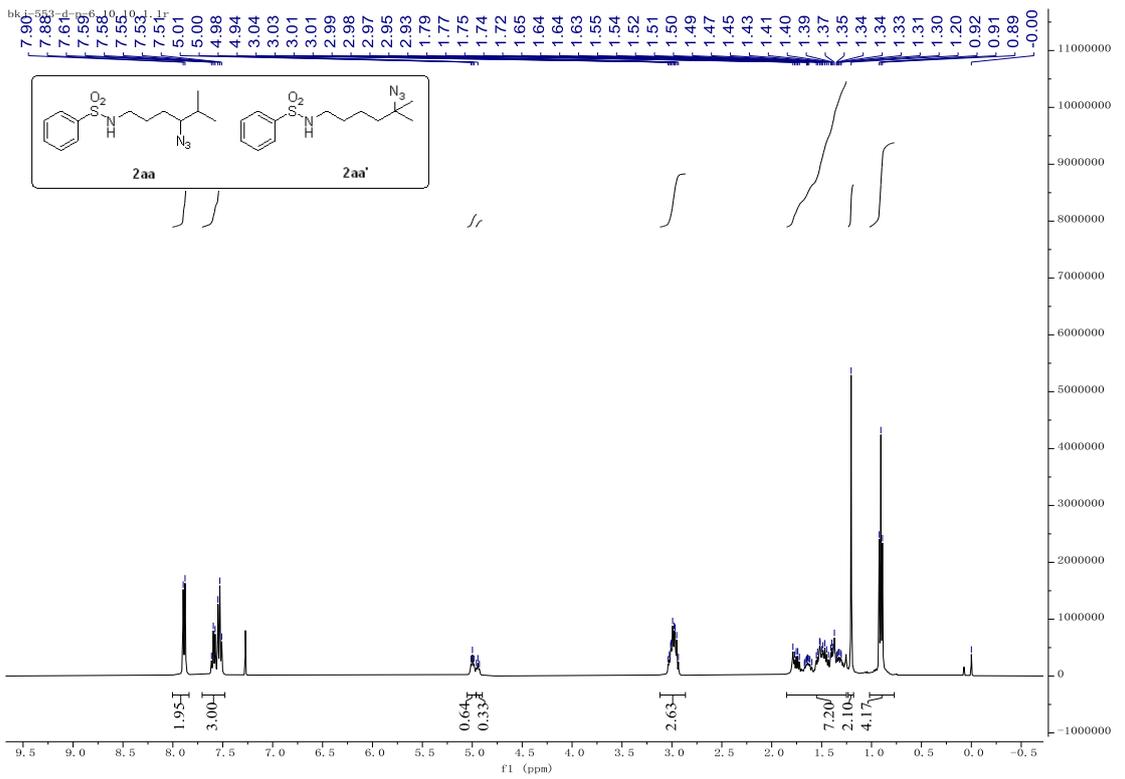
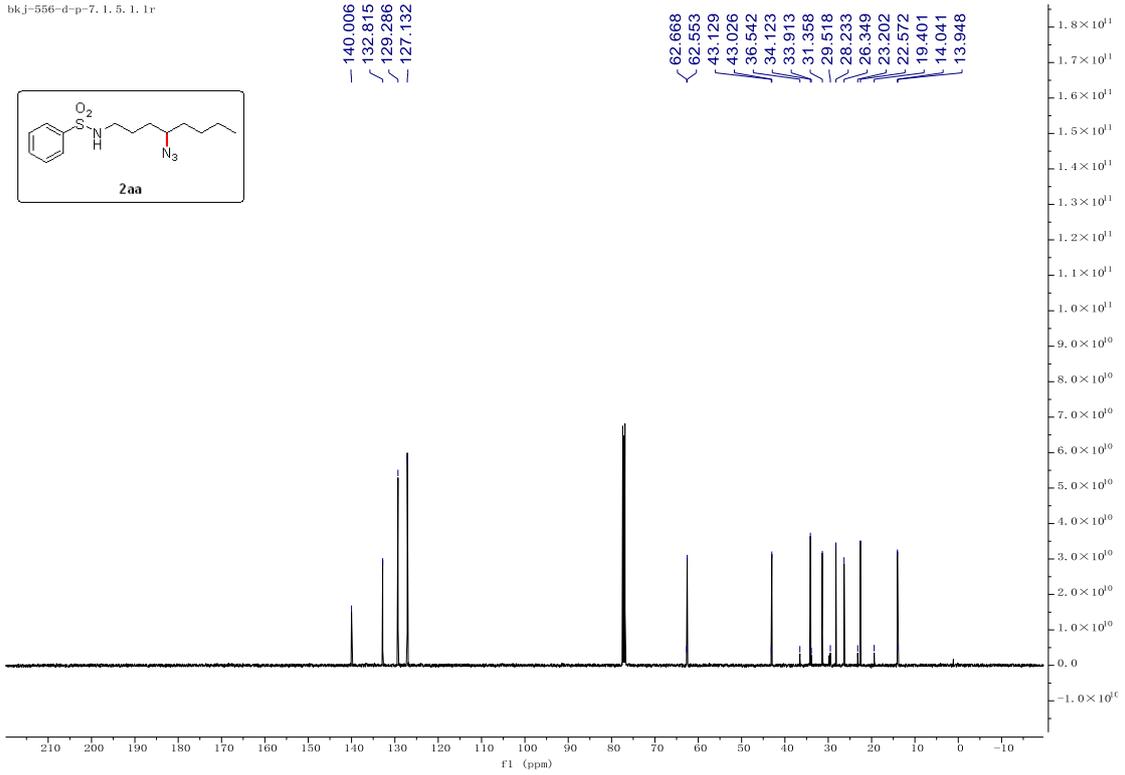
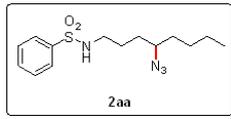


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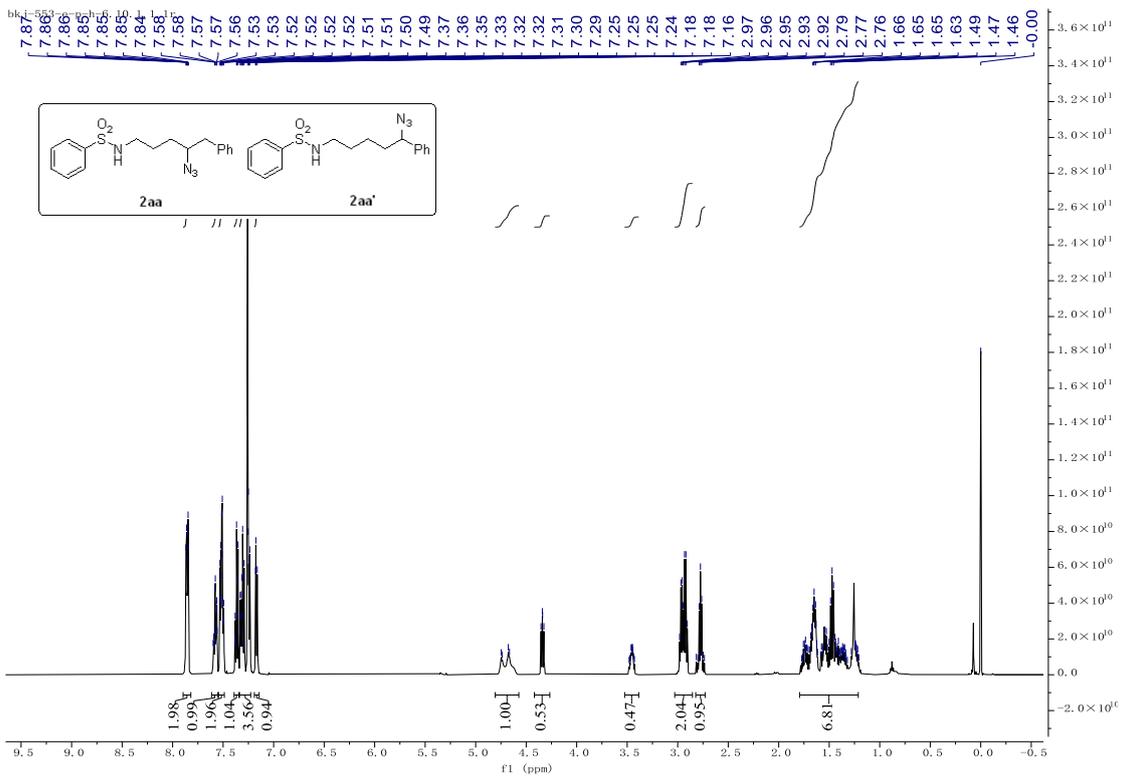
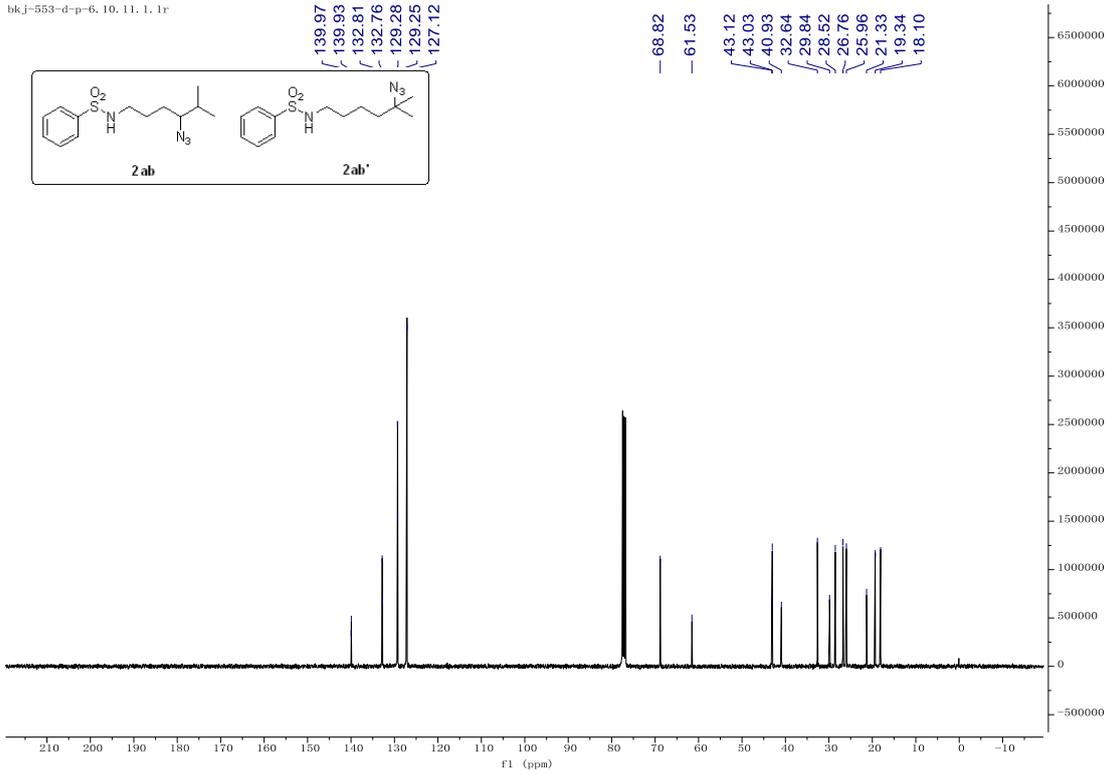




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bkj-553-d-p-6. 10. 11. 1. 1r



bkj-553-e-p-h-6, 10-C, 1, 1, 1r

