

## Supporting Information

### Selectfluor®-Enabled Photochemical Selective C(sp<sup>3</sup>)-H (Sulfonyl)amidation

Yuehua Chen,<sup>a</sup> Boxuan Yang,<sup>a</sup> Qian-Yu Li,<sup>a</sup> Yu-Mei Lin,<sup>a</sup> and Lei Gong\*<sup>a,b</sup>

<sup>a</sup> Key Laboratory of Chemical Biology of Fujian Province, College of Chemistry and Chemical Engineering, Xiamen University, Xiamen 361005, China.

<sup>b</sup> Innovation Laboratory for Sciences and Technologies of Energy Materials of Fujian Province (IKKEM), Xiamen 361005, China.

E-mail: gongl@xmu.edu.cn

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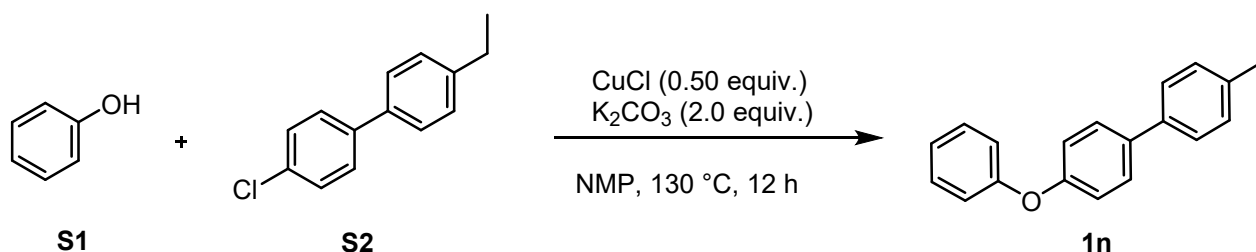
## 1. General Information

Solvents were distilled under argon from calcium hydride ( $\text{CH}_2\text{Cl}_2$ ) and  $\text{ClCH}_2\text{CH}_2\text{Cl}$  were purchased from SCR<sup>®</sup> and used without additional purification. Solvents for flash column chromatography (*n*-pentane, petroleum ether, EtOAc,  $\text{CH}_2\text{Cl}_2$ ) were purchased from Titan and used without additional purification. All other reagents were purchased from commercial suppliers (TCI, Sigma-Aldrich, Alfa Aesar, Macklin, Bide Pharmatech, Adamas-beta<sup>®</sup>, J&K Scientific, etc.) and used without further purification. Photochemical reactions were performed in 10 mL Schlenk tubes under an atmosphere of argon and upon irradiation with a 50 W LEDs ( $\lambda_{\text{max}} = 395 \text{ nm}$  or  $\lambda_{\text{max}} = 365 \text{ nm}$ ; commercial supplier: Hong Chang Lighting Co. Ltd., website: <http://hongchang-led.taobao.com>). Flash column chromatography was performed with silica gel (300–400 mesh, pH = 6.7–7.0). <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on a Bruker AM (500 MHz) or Bruker AM (600 MHz) spectrometer at ambient temperature. NMR standards were used as follows:  $\text{CDCl}_3 = 7.26 \text{ ppm}$  (<sup>1</sup>H NMR), 77.0 ppm (<sup>13</sup>C NMR). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), and integration. IR spectra were recorded on a Nicolet Avatar 330 FT-IR spectrophotometer. High-resolution mass spectra were recorded on an Agilent 1290-G6545XT QTOF instrument using ESI technique.

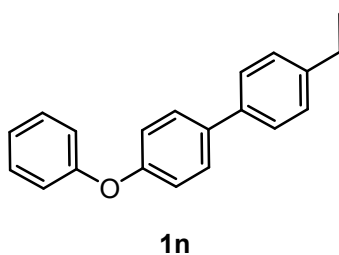
## 2. Synthesis of the Substrates

Ethylbenzene derivatives **1l**, **1m** and **1zc** were prepared by published procedures.<sup>1</sup> **1a–1k**, **1o–1zb**, **1ze** and **1zg–1zi** were purchased from TCI, Sigma-Aldrich, Alfa, Macklin, Bide Pharmatech, Adamas-beta®, J&K, etc.) and used without further purification.

### Preparation of substrates **1n**, **1zd**, **1zf** and **1zj**.



### 4-ethyl-4'-phenoxy-1,1'-biphenyl (**1n**)

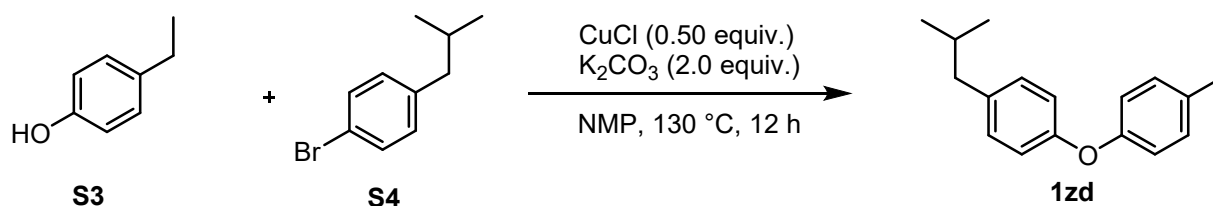


To a solution of phenol (**S1**, 470.1 mg, 5.00 mmol) in dry *N*-methylpyrrolidone (NMP, 30 mL) was added copper(I) chloride (247.5 mg, 2.50 mmol), potassium carbonate (1382.0 mg, 10.0 mmol) and 4-chloro-4'-ethyl-1,1'-biphenyl (**S2**, 1191.9 mg, 5.50 mmol) at room temperature. The reaction mixture was stirred at 130 °C under argon for 12 h, then quenched with H<sub>2</sub>O (20 mL) and extracted with CH<sub>2</sub>Cl<sub>2</sub> (30 mL x 3). The combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The residue was subjected to silica gel chromatography [petroleum ether (PE): ethyl acetate (EA) = 20:1] to afford the pure **1n** as a white solid (891.7 mg, 3.30 mmol, 65% yield).

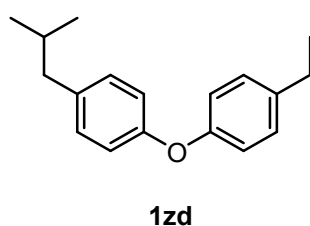
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.57 – 7.52 (m, 2H), 7.52 – 7.48 (m, 2H), 7.39 – 7.33 (m, 2H), 7.29 – 7.26 (m, 2H), 7.15 – 7.10 (m, 1H), 7.09 – 7.04 (m, 4H), 2.70 (q, *J* = 7.6 Hz, 2H), 1.29 (t, *J* = 7.6 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 157.3, 156.6, 143.2, 138.0, 136.3, 129.8, 128.3, 128.2, 126.8, 123.3, 119.0, 119.0, 28.5, 15.6.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3726, 3628, 3602, 3055, 2965, 2930, 1897, 1734, 1685, 1654, 1589, 1523, 1489, 1455, 1400, 1265, 1240, 1167, 1138, 1111, 1071, 1023, 1005, 898, 871, 825, 775, 746, 692.



### 1-ethyl-4-(4-isobutylphenoxy)benzene (**1zd**)

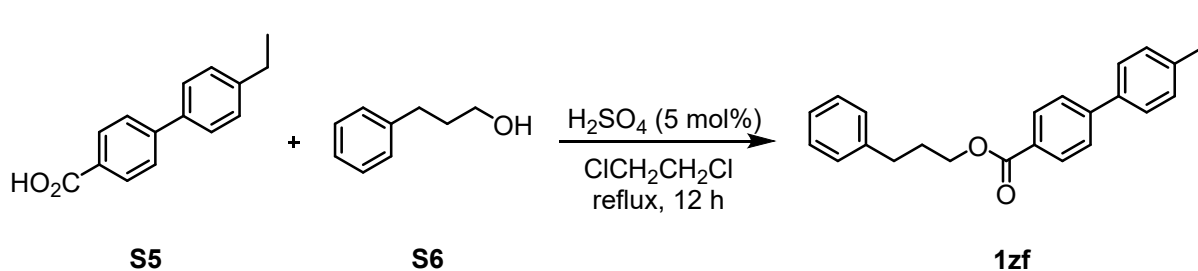


To a solution of 4-ethylphenol (**S3**, 610.8 mg, 5.00 mmol) in dry *N*-methylpyrrolidone (NMP, 30 mL) was added copper(I) chloride (247.5 mg, 2.50 mmol), potassium carbonate (1382.1 mg, 10.0 mmol) and 1-bromo-4-isobutylbenzene (**S4**, 1172.1 mg, 5.50 mmol) at room temperature. The reaction mixture was stirred at 130 °C under argon for 12 h, then quenched with H<sub>2</sub>O (30 mL) and extracted with CH<sub>2</sub>Cl<sub>2</sub> (30 mL x 3). The combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The residue was subjected to silica gel chromatography (PE: EA = 20:1) to afford the pure **1zd** as a yellow oil (674.1 mg, 2.70 mmol, 53% yield).

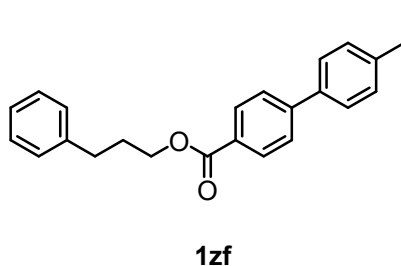
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.18 – 7.13 (m, 2H), 7.12 – 7.06 (m, 2H), 6.92 (t,  $J$  = 8.2 Hz, 4H), 2.64 (q,  $J$  = 7.6 Hz, 2H), 2.45 (d,  $J$  = 7.2 Hz, 2H), 1.86 (m,  $J$  = 13.5, 6.8 Hz, 1H), 1.24 (t,  $J$  = 7.6 Hz, 3H), 0.92 (d,  $J$  = 6.7 Hz, 6H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  155.5, 155.4, 138.9, 136.3, 130.2, 128.9, 118.7, 118.4, 44.7, 30.3, 28.1, 22.3, 15.7.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3727, 2960, 1735, 1685, 1654, 1602, 1560, 1501, 1466, 1239, 1168, 874, 833.



### 3-phenylpropyl 4'-ethyl-[1,1'-biphenyl]-4-carboxylate (**1zf**)



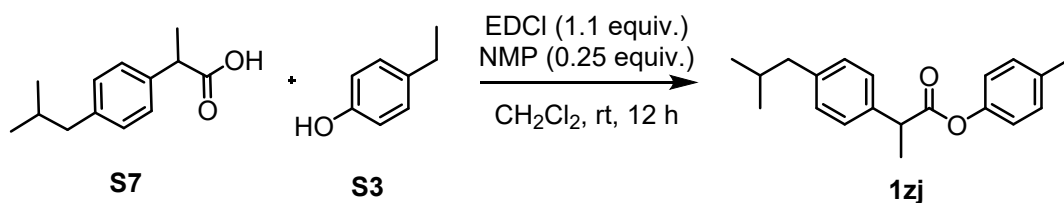
To a solution of 4'-ethyl-(1,1'-biphenyl)-4-carboxylic acid (**S5**, 1131.4 mg, 5.00 mmol) and 3-phenylpropan-1-ol (**S6**, 1021.5 mg, 7.50 mmol) in  $\text{ClCH}_2\text{CH}_2\text{Cl}$  (30 mL) were added concentrated sulfuric acid (1.0 mL, 18.4 M). The reaction mixture was stirred at 85 °C and neutralize with aqueous  $\text{Na}_2\text{CO}_3$ . The whole mixture was then extracted with  $\text{CH}_2\text{Cl}_2$  (30 mL x 3) and washed with water.  $\text{CH}_2\text{Cl}_2$  layer was dried over  $\text{Na}_2\text{SO}_4$  and evaporated under reduced pressure. The residue was subjected to silica gel chromatography (PE: EA = 20:1) to afford the pure **1zf** as a yellow oil (1636.1 mg, 4.80 mmol, 95% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.13 – 8.01 (m, 2H), 7.65 – 7.60 (m, 2H), 7.55 – 7.51 (m, 2H), 7.31 – 7.25 (m, 4H), 7.23 – 7.16 (m, 3H), 4.34 (t,  $J = 6.5$  Hz, 2H), 2.78 (t,  $J = 7.6$  Hz, 2H), 2.68 (q,  $J = 7.6$  Hz, 2H), 2.15 – 2.05 (m, 2H), 1.26 (t,  $J = 7.6$  Hz, 3H).

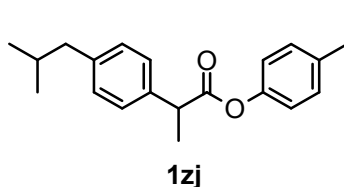
$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  166.6, 145.6, 144.5, 141.2, 137.4, 130.1, 128.9, 128.5, 128.5, 128.5, 127.2, 126.8, 126.1, 64.4, 32.4, 30.4, 28.6, 15.5.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3436, 1640, 1496, 1273, 1112.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{24}\text{H}_{24}\text{NaO}_2^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 367.1669, found: 367.1681.



#### 4-ethylphenyl 2-(4-isobutylphenyl)propanoate (**1zj**)



To a solution of 2-(4-isobutylphenyl)propanoic acid (**S7**, 2062.9 mg, 10.0 mmol) and 4-ethylphenol (**S3**, 1221.7 mg, 10.0 mmol) in  $\text{CH}_2\text{Cl}_2$  (20 mL) were added 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDCI, 2108.7 mg, 11.0 mmol) and 4-dimethylaminopyridine (DMAP, 305.4 mg, 2.50 mmol). The reaction mixture was stirred at room temperature for 12 h, then concentrated under reduced pressure. The residue was subjected to silica gel chromatography (PE: EA = 10:1) to afford the pure **1zj** as a yellow oil (2700.8 mg, 8.70 mmol, 87% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.31 (d,  $J = 8.3$ , 2.6 Hz, 2H), 7.18 – 7.13 (m, 4H), 6.92 (d,  $J = 9.0$ , 2.7 Hz, 2H), 3.94 (q,  $J = 7.2$ , 2.6 Hz, 1H), 2.63 (q,  $J = 7.6$  Hz, 2H), 2.49 (d,  $J = 7.2$ , 2.7 Hz, 2H), 1.92 – 1.82 (m, 1H), 1.61 (d,  $J = 7.1$ , 2.6 Hz, 3H), 1.22 (t,  $J = 7.6$ , 2.4 Hz, 3H), 0.93 (d,  $J = 6.7$ , 2.6 Hz, 6H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  173.4, 148.8, 141.6, 140.8, 137.4, 129.5, 128.6, 127.2, 121.1, 45.3, 45.1, 30.1, 28.3, 22.4, 18.6, 15.6.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3726, 3491, 3026, 2962, 2932, 2869, 1897, 1756, 1654, 1605, 1560, 1508, 1456, 1420, 1377, 1366, 1332, 1200, 1166, 1140, 1073, 1043, 1019, 992, 939, 895, 846.

### 3. Set-up of the Photochemical Reactions



**Figure S1.** Set-up of the photochemical reactions (Left:  $\lambda_{\text{max}} = 395 \text{ nm}$ , Right:  $\lambda_{\text{max}} = 365 \text{ nm}$ ).



## 4. Photochemical C(sp<sup>3</sup>)-H Sulfonylamidation Reaction

### 4.1 Optimization of Conditions for the C(sp<sup>3</sup>)-H Sulfonylamidation Reaction

**General procedure.** A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), *p*-methylbenzenesulfonamide (**2a**, 68.5 mg, 0.40 mmol), Selectfluor<sup>®</sup> (1.0 – 3.0 equiv.) and the indicated solvent (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395 \text{ nm}$ ) with a direct current DC fan cooling. The reaction was stirred at 40 °C for 48 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE: EA = 5:1) to afford the pure product (**3**) as a white solid.

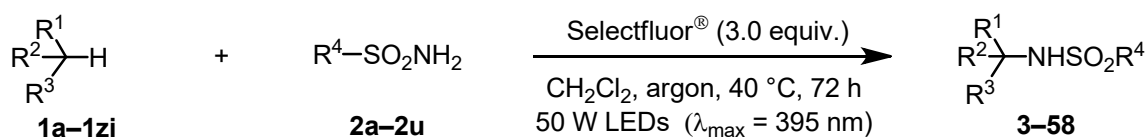
**Table S1.** Optimization for the Photochemical C(sp<sup>3</sup>)-H Sulfonylamidation Reaction <sup>a</sup>

entry	Selectfluor <sup>®</sup> (equiv.)	additive	solvent	time (h)	yield (%) <sup>b</sup>
1	3.0	none	ClCH <sub>2</sub> CH <sub>2</sub> Cl	48	66
2	3.0	none	CH <sub>2</sub> Cl <sub>2</sub>	48	74
3	3.0	none	CHCl <sub>3</sub>	48	52
4	3.0	none	acetone	48	n.a.
5	3.0	none	PhCl	48	74
6	3.0	none	HFIP	48	n.a.
7	3.0	none	THF	48	n.a.
8	3.0	none	MeCN	48	n.a.
9	3.0	none	ethyl acetate	48	15
10	3.0	none	DMF	48	n.a.
11	3.0	none	DMSO	48	n.a.
12	1.0	MnO <sub>2</sub> (0.5 equiv.)	CH <sub>2</sub> Cl <sub>2</sub>	48	n.a.
13	1.0	(NH <sub>4</sub> ) <sub>2</sub> S <sub>2</sub> O <sub>8</sub> (0.5 equiv.)	CH <sub>2</sub> Cl <sub>2</sub>	48	52
14	1.0	<i>m</i> -CPBA (0.5 equiv.)	CH <sub>2</sub> Cl <sub>2</sub>	48	63
15	1.0	Pyridine <i>N</i> -oxide (0.5 equiv.)	CH <sub>2</sub> Cl <sub>2</sub>	48	11
16	1.0	Trimethylamine <i>N</i> -oxide (0.5 equiv.)	CH <sub>2</sub> Cl <sub>2</sub>	48	20
17	1.0	CuO (0.5 equiv.)	CH <sub>2</sub> Cl <sub>2</sub>	48	29
18	1.0	CuCl <sub>2</sub> (0.5 equiv.)	CH <sub>2</sub> Cl <sub>2</sub>	48	43

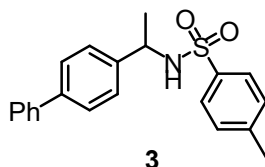
19	1.0	FeCl <sub>3</sub> (0.5 equiv.)	CH <sub>2</sub> Cl <sub>2</sub>	48	30
20	1.0	CoCl <sub>2</sub> (0.5 equiv.)	CH <sub>2</sub> Cl <sub>2</sub>	48	55
21 <sup>c</sup>	3.0	none	CH <sub>2</sub> Cl <sub>2</sub>	48	72
22 <sup>d</sup>	3.0	none	CH <sub>2</sub> Cl <sub>2</sub>	48	44
23 <sup>e</sup>	3.0	none	CH <sub>2</sub> Cl <sub>2</sub>	48	81
24 <sup>f</sup>	3.0	none	CH <sub>2</sub> Cl <sub>2</sub>	48	n.a.
25 <sup>g</sup>	3.0	none	CH <sub>2</sub> Cl <sub>2</sub>	48	n.a.
26 <sup>h</sup>	3.0	none	CH <sub>2</sub> Cl <sub>2</sub>	48	trace
27	3.0	TEMPO (4.0 equiv.)	CH <sub>2</sub> Cl <sub>2</sub>	48	n.a.
28 <sup>i</sup>	3.0	none	CH <sub>2</sub> Cl <sub>2</sub>	48	76

<sup>a</sup> Reaction conditions: **1a** (0.20 mmol, 1.0 equiv.), **2a** (0.40 mmol, 2.0 equiv.), CH<sub>2</sub>Cl<sub>2</sub> (1.0 mL), 40 °C, 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm), under argon. <sup>b</sup> Isolated yield. <sup>c</sup> Reaction under air. <sup>d</sup> Irradiation with a 50 W LEDs ( $\lambda_{\text{max}} = 427$  nm). <sup>e</sup> Irradiation with a 50 W LEDs ( $\lambda_{\text{max}} = 365$  nm). <sup>f</sup> Reaction in the dark. <sup>g</sup> Reaction in the dark and in 60 °C. <sup>h</sup> Reaction in the dark and in 120 °C. <sup>i</sup> **1a** (0.30 mmol), **2a** (0.20 mmol), CH<sub>2</sub>Cl<sub>2</sub> (1.0 mL), 40 °C, 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm), under argon. n.a. = not applicable. HFIP = 1,1,1,3,3,3-hexafluoro-2-propanol. THF = tetrahydrofuran. DMF = N,N-dimethylformamide. DMSO = dimethyl sulfoxide.

## 4.2 Substrate Scope of the C(sp<sup>3</sup>)-H Sulfonylamidation Reaction



### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-4-methylbenzenesulfonamide (**3**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *p*-toluenesulfonamide (**2a**, 34.2 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan

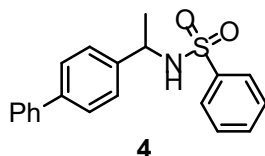
cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **3** as a white solid (56.9 mg, 0.162 mmol, 81% yield).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.76 – 7.66 (m, 2H), 7.62 – 7.57 (m, 2H), 7.55 – 7.46 (m, 4H), 7.46 – 7.38 (m, 1H), 7.26 – 7.24 (m, 4H), 5.12 – 4.95 (m, 1H), 4.65 – 4.57 (m, 1H), 2.43 (s, 3H), 1.55 (d, *J* = 6.9 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 143.1, 141.0, 140.6, 140.5, 137.7, 129.4, 128.8, 127.4, 127.2, 127.2, 127.0, 126.6, 53.4, 23.5, 21.4.

Spectral data for this compound was previously reported and matched with the current data.<sup>[2]</sup>

#### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)benzenesulfonamide (**4**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}}$  = 395 nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **4** as a white solid (63.4 mg, 0.188 mmol, 94% yield).

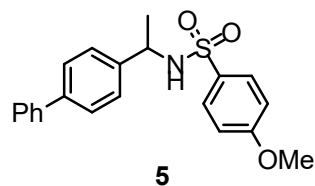
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.76 – 7.72 (m, 2H), 7.53 – 7.49 (m, 2H), 7.49 – 7.45 (m, 1H), 7.45 – 7.41 (m, 2H), 7.41 – 7.32 (m, 5H), 7.15 (d, *J* = 1.9 Hz, 2H), 5.15 (d, *J* = 7.1 Hz, 1H), 4.60 – 4.52 (m, 1H), 1.47 (d, *J* = 10.0 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 140.9, 140.7, 140.6, 140.5, 132.3, 128.8, 128.8, 127.4, 127.2, 127.1, 127.0, 126.6, 53.5, 23.5.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3292, 2844, 2118, 1652, 1448, 1407, 1157, 1016.

HRMS (ESI-TOF, *m/z*) calcd for C<sub>20</sub>H<sub>19</sub>NNaO<sub>2</sub>S<sup>+</sup> (*M*+Na)<sup>+</sup>: 360.1029, found: 360.1024.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-4-methoxybenzenesulfonamide (**5**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *p*-methoxybenzenesulfonamide (**2c**, 37.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **5** as a white solid (58.1 mg, 0.158 mmol, 79% yield).

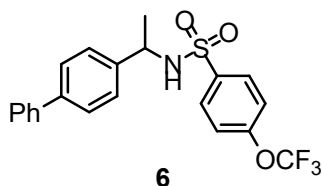
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.66 – 7.62 (m, 2H), 7.54 – 7.49 (m, 2H), 7.46 – 7.37 (m, 4H), 7.37 – 7.31 (m, 1H), 7.20 – 7.13 (m, 2H), 6.85 – 6.77 (m, 2H), 4.79 – 4.69 (m, 1H), 4.56 – 4.48 (m, 1H), 3.77 (s, 3H), 1.48 (d,  $J = 6.8$  Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  162.7, 140.9, 140.6, 140.5, 132.2, 129.2, 128.8, 127.4, 127.2, 127.0, 126.7, 113.9, 55.5, 53.4, 23.5.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3696, 3680, 3371, 2949, 2843, 1654, 1560, 1454, 1331, 1054, 1033, 1018, 429.

Spectral data for this compound was previously reported and matched with the current data.<sup>[2]</sup>

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-4-(trifluoromethoxy)benzenesulfonamide (**6**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *p*-(trifluoromethoxy)benzenesulfonamide (**2d**, 48.2 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA

= 5:1) to afford **6** as a white solid (71.6 mg, 0.170 mmol, 85% yield).

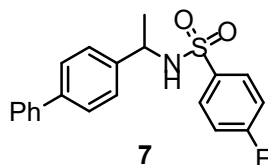
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.76 – 7.72 (m, 2H), 7.52 – 7.47 (m, 2H), 7.45 – 7.41 (m, 2H), 7.41 – 7.37 (m, 2H), 7.37 – 7.33 (m, 1H), 7.17 – 7.12 (m, 4H), 5.42 (d,  $J = 7.1$  Hz, 1H), 4.62 – 4.55 (m, 1H), 1.49 (d,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  151.8, 140.8, 140.4, 140.4, 139.1, 129.2, 128.8, 127.5, 127.2, 127.0, 126.6, 120.6, 120.2 (q,  $J = 2.1$  Hz), 53.7, 23.6.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3439, 1654, 1489, 1427, 1328, 1298, 1259 1216, 1161, 1091, 1016, 963, 808, 766, 665.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{21}\text{H}_{18}\text{F}_3\text{KNO}_3\text{S}^+$  ( $\text{M}+\text{K}$ ) $^+$ : 460.0591, found: 460.0588.

#### ***N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-4-fluorobenzenesulfonamide (7)**



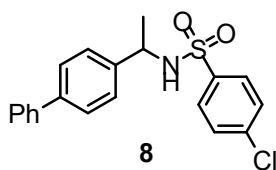
A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *p*-fluorobenzenesulfonamide (**2e**, 35.0 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **7** as a white solid (67.5 mg, 0.190 mmol, 95% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.72 – 7.67 (m, 2H), 7.53 – 7.48 (m, 2H), 7.46 – 7.38 (m, 4H), 7.38 – 7.30 (m, 1H), 7.16 – 7.11 (m, 2H), 7.05 – 6.98 (m, 2H), 4.89 (d,  $J = 6.8$  Hz, 1H), 4.60 – 4.53 (m, 1H), 1.49 (d,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.8, 140.5(d,  $J = 5.0$  Hz), 129.8, 129.8, 129.7, 128.8, 127.5, 127.3, 127.0, 126.6, 116.0, 115.8, 53.6, 23.6.

Spectral data for this compound was previously reported and matched with the current data.<sup>[2]</sup>

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-4-chlorobenzenesulfonamide (**8**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *p*-chlorobenzenesulfonamide (**2f**, 38.3 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **8** as a white solid (65.5 mg, 0.176 mmol, 88% yield).

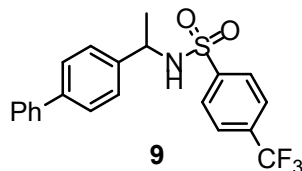
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.63 – 7.59 (m, 2H), 7.54 – 7.49 (m, 2H), 7.43 (dd,  $J = 8.5, 6.8$  Hz, 2H), 7.41 – 7.38 (m, 2H), 7.38 – 7.33 (m, 1H), 7.32 – 7.28 (m, 2H), 7.15 – 7.11 (m, 2H), 5.07 (d,  $J = 7.0$  Hz, 1H), 4.60 – 4.53 (m, 1H), 1.49 (d,  $J = 6.9$  Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  140.8, 140.5, 140.4, 139.3, 138.8, 129.0, 128.8, 128.5, 127.5, 127.3, 127.1, 126.6, 53.7, 23.5.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3394, 2949, 2843, 1654, 1560, 1413, 1053, 1033, 1017.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>20</sub>H<sub>18</sub>ClNNaO<sub>2</sub>S<sup>+</sup> ( $M+\text{Na}$ )<sup>+</sup>: 394.0639, found: 394.0647.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-4-(trifluoromethyl)benzenesulfonamide (**9**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *p*-(trifluoromethyl)benzenesulfonamide (**2g**, 45.0 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for for 72 h, then concentrated to dryness under reduced

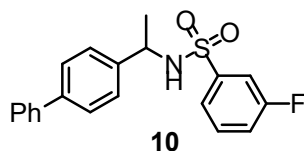
pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **9** as a white solid (77.8 mg, 0.192 mmol, 96% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.82 – 7.77 (m, 2H), 7.73 (d,  $J = 8.1$  Hz, 2H), 7.65 (d,  $J = 8.2$  Hz, 2H), 7.56 – 7.51 (m, 1H), 7.48 – 7.39 (m, 4H), 7.28 – 7.23 (m, 2H), 5.03 (d,  $J = 6.9$  Hz, 1H), 4.65 – 4.58 (m, 1H), 1.52 (d,  $J = 6.8$  Hz, 3H).

$^{13}\text{C}$  NMR (151 MHz,  $\text{CDCl}_3$ )  $\delta$  144.2, 140.8, 140.2, 140.1, 133.9 (q,  $J = 32.5$  Hz), 128.8, 127.5, 127.5, 127.2, 126.9, 126.6, 125.8 (q,  $J = 3.8$  Hz), 123.2 (q,  $J = 273.3$  Hz), 53.8, 23.5.

Spectral data for this compound was previously reported and matched with the current data.<sup>[2]</sup>

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-3-fluorobenzenesulfonamide (**10**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *m*-fluorobenzenesulfonamide (**2h**, 35.0 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **10** as a white solid (67.5 mg, 0.190 mmol, 95% yield).

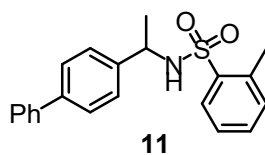
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.49 – 7.52 (m, 3H), 7.47 – 7.33 (m, 6H), 7.33 – 7.28 (m, 1H), 7.19 – 7.10 (m, 3H), 5.44 (d,  $J = 7.3$  Hz, 1H), 4.64 – 4.56 (m, 1H), 1.50 (d,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  163.2, 161.2, 140.7, 140.6, 140.5, 130.6 (d,  $J = 8.8$  Hz), 128.8, 127.4, 127.3, 127.0, 126.6, 122.8 (d,  $J = 3.8$  Hz), 119.4 (d,  $J = 21.4$  Hz), 114.5 (d,  $J = 25.2$  Hz), 53.8, 23.5.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3285, 1735, 1654, 1597, 1477, 1437, 1328, 1304, 1271, 1225, 1156, 1085, 1017, 964, 839, 789, 766, 734, 695, 679, 584.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{20}\text{H}_{18}\text{FNNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ )<sup>+</sup>: 378.0934, found: 378.0940.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-2-methylbenzenesulfonamide (**11**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *o*-methylbenzenesulfonamide (**2i**, 34.2 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **11** as a white solid (44.9 mg, 0.128 mmol, 64% yield).

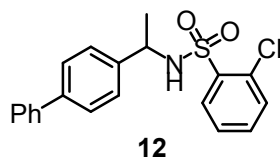
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.85 (7.84 – 7.86, 1H), 7.53 – 7.48 (m, 2H), 7.46 – 7.40 (m, 2H), 7.40 – 7.30 (m, 4H), 7.19 – 7.16 (m, 2H), 7.16 – 7.10 (m, 2H), 5.02 (d,  $J = 6.8$  Hz, 1H), 4.54 – 4.47 (m, 1H), 2.57 (s, 3H), 1.49 (d,  $J = 6.9$  Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  140.7, 140.6, 140.5, 138.4, 136.9, 132.5, 132.3, 129.6, 128.8, 127.4, 127.2, 127.0, 126.6, 126.0, 53.4, 23.3, 20.3.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3727, 3583, 3284, 1735, 1685, 1654, 1560, 1321, 1158, 1065, 763.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>21</sub>H<sub>21</sub>NNaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 374.1185, found: 374.1179.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-2-chlorobenzenesulfonamide (**12**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *o*-chlorobenzenesulfonamide (**2j**, 38.3 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **12** as a white solid (66.9 mg, 0.180 mmol, 90% yield).



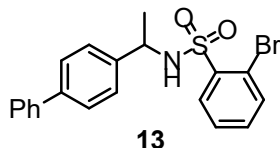
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.88 (dd,  $J = 7.8, 1.5$  Hz, 1H), 7.51 – 7.40 (m, 4H), 7.39 – 7.28 (m, 5H), 7.24 – 7.21 (m, 1H), 7.17 – 7.08 (m, 2H), 5.44 (d,  $J = 7.7$  Hz, 1H), 4.58 – 4.51 (m, 1H), 1.52 (d,  $J = 7.0$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.6, 140.6, 140.1, 138.0, 133.2, 131.3, 131.2, 131.1, 128.8, 127.4, 127.1, 127.0, 126.9, 126.5, 54.0, 22.9.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3301, 3060, 3029, 2977, 2931, 1654, 1617, 1600, 1578, 1487, 1454, 1432, 1377, 1330, 1255, 1166, 1127, 1101, 1077, 1045, 1008, 961, 867, 839, 796, 765, 750, 735, 699, 665, 613, 585, 563, 422.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{20}\text{H}_{18}\text{ClNNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 394.0639, found: 394.0646.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-2-bromobenzenesulfonamide (**13**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *o*-bromobenzenesulfonamide (**2k**, 47.2 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **13** as a white solid (61.6 mg, 0.148 mmol, 74% yield).

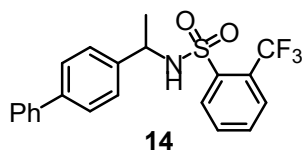
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.86 – 7.81 (m, 1H), 7.48 – 7.46 (m, 1H), 7.41 – 7.37 (m, 2H), 7.36 – 7.31 (m, 2H), 7.28 – 7.24 (m, 3H), 7.22 – 7.14 (m, 2H), 7.10 – 7.05 (m, 2H), 5.41 (d,  $J = 7.6$  Hz, 1H), 4.49 – 4.40 (m, 1H), 1.43 (d,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.6, 140.6, 140.0, 139.6, 134.7, 133.1, 131.4, 128.8, 127.5, 127.4, 127.1, 127.0, 126.6, 119.8, 54.1, 22.9.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3314, 1735, 1654, 1560, 1420, 1331, 1165, 763, 735.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{20}\text{H}_{18}\text{BrNNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 438.0134, found: 438.0149.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-2-(trifluoromethyl)benzenesulfonamide (**14**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *o*-(trifluoromethyl)benzenesulfonamide (**2l**, 45.0 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **14** as a white solid (60.0 mg, 0.148 mmol, 74% yield).

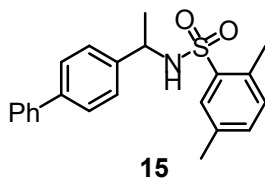
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.93 (d,  $J = 7.9$  Hz, 1H), 7.75 (d,  $J = 7.8$  Hz, 1H), 7.53 (t,  $J = 7.6$  Hz, 1H), 7.50 – 7.46 (m, 2H), 7.46 – 7.39 (m, 3H), 7.38 – 7.31 (m, 3H), 7.15 – 7.10 (m, 2H), 5.12 (d,  $J = 7.5$  Hz, 1H), 4.68 – 4.60 (m, 1H), 1.49 (d,  $J = 6.9$  Hz, 3H).

<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  140.5, 140.4, 140.3, 139.2, 132.1, 132.0, 131.6, 128.8, 128.0 (q,  $J = 3.5$  Hz), 127.4, 127.1, 126.9, 126.7 (q,  $J = 32.7$  Hz), 126.5, 123.0 (q,  $J = 273.8$  Hz), 54.0, 23.3.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3727, 3629, 3317, 3031, 2979, 1735, 1685, 1654, 1597, 1560, 1487, 1438, 1420, 1346, 1308, 1269, 1167, 1117, 1095, 1077, 1037, 965, 839, 767, 734, 713, 699, 646, 613, 586.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>21</sub>H<sub>18</sub>F<sub>3</sub>NNaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 428.0903, found: 428.0905.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-2,5-dimethylbenzenesulfonamide (**15**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), 2,5-dimethylbenzenesulfonamide (**2m**, 37.0 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced

pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **15** as a white solid (42.4 mg, 0.116 mmol, 58% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 (d,  $J$  = 1.8 Hz, 1H), 7.52 – 7.47 (m, 2H), 7.45 – 7.42 (m, 2H), 7.39 – 7.32 (m, 3H), 7.17 – 7.11 (m, 3H), 7.07 (d,  $J$  = 7.7 Hz, 1H), 4.97 (d,  $J$  = 6.6 Hz, 1H), 4.55 – 4.48 (m, 1H), 2.53 (s, 3H), 2.22 (s, 3H), 1.49 (d,  $J$  = 6.9 Hz, 3H).

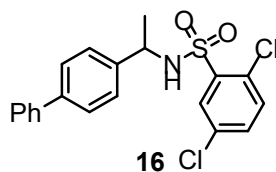
$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.7, 140.6, 140.4, 138.0, 135.8, 133.6, 133.1, 132.2, 130.1, 128.8, 127.4, 127.1, 127.0, 126.6, 53.4, 23.3, 20.7, 19.8.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3451, 2117, 1654, 1485, 1420, 1315, 1154, 1101, 1065, 1016, 959, 765, 733, 696.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{22}\text{H}_{23}\text{NNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 388.1342, found: 388.1346.

*Note:* Side products such as self-coupling products of 2,5-dimethylbenzenesulfonamide were detected by HRMS.

#### ***N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-2,5-dichlorobenzenesulfonamide (16)**



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), 2,5-dichlorobenzenesulfonamide (**2n**, 45.2 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}}$  = 395 nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **16** as a white solid (78.8 mg, 0.194 mmol, 97% yield).

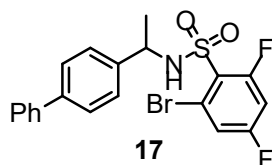
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.69 (d,  $J$  = 2.4 Hz, 1H), 7.42 – 7.38 (m, 2H), 7.38 – 7.33 (m, 2H), 7.29 – 7.23 (m, 3H), 7.18 – 7.15 (m, 1H), 7.13 (s, 1H), 7.09 – 7.03 (m, 2H), 5.31 (d,  $J$  = 7.8 Hz, 1H), 4.55 – 4.47 (m, 1H), 1.47 (d,  $J$  = 7.0 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.9, 140.4, 139.4, 139.3, 133.1, 132.8, 132.2, 131.0, 129.4, 128.8, 127.5, 127.1, 127.0, 126.6, 54.2, 22.8.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3436, 1654, 1560, 1450, 1331, 1167, 429.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>20</sub>H<sub>17</sub>Cl<sub>2</sub>NNaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 428.0249, found: 428.0251.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-2-bromo-4,6-difluorobenzenesulfonamide (**17**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), 2-bromo-4,6-difluorobenzenesulfonamide (**2o**, 54.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **17** as a white solid (72.4 mg, 0.160 mmol, 80% yield).

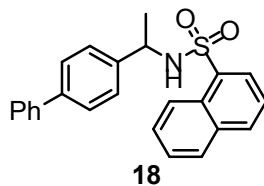
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.49 – 7.40 (m, 4H), 7.39 – 7.32 (m, 3H), 7.23 – 7.17 (m, 2H), 7.11 (dt,  $J = 7.8, 2.1$  Hz, 1H), 6.65 (m, 1H), 5.60 (d,  $J = 8.2$  Hz, 1H), 4.75 – 4.60 (m, 1H), 1.58 (d,  $J = 7.0$  Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  163.5 (dd,  $J = 260.8, 12.6$  Hz), 160.8 (dd,  $J = 263.3, 12.6$  Hz), 141.1, 140.4, 139.4, 128.8, 127.5, 127.2, 127.0, 126.5, 125.7 (dd,  $J = 12.6, 5.0$  Hz), 122.9 (dd,  $J = 11.3, 1.3$  Hz), 118.8 (dd,  $J = 25.2, 3.8$  Hz), 105.1 (dd,  $J = 27.7, 25.2$  Hz), 54.3, 22.7.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3454, 2110, 1640, 1485, 1420, 1350, 1175, 1127, 1072.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>20</sub>H<sub>16</sub>BrF<sub>2</sub>NNaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 473.9945, found: 473.9946.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)naphthalene-1-sulfonamide (**18**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), naphthalene-1-sulfonamide (**2p**, 41.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and

dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **18** as a white solid (45.0 mg, 0.116 mmol, 58% yield).

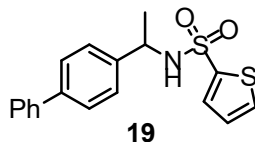
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.59 (d,  $J = 8.6$  Hz, 1H), 8.09 (d,  $J = 7.3$  Hz, 1H), 7.91 (d,  $J = 8.2$  Hz, 1H), 7.87 – 7.79 (m, 1H), 7.66 – 7.62 (m, 1H), 7.57 – 7.54 (m, 1H), 7.45 – 7.38 (m, 4H), 7.37 – 7.29 (m, 2H), 7.13 – 7.08 (m, 2H), 6.91 – 6.86 (m, 2H), 5.07 (d,  $J = 7.0$  Hz, 1H), 4.55 – 4.48 (m, 1H), 1.41 (d,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.6, 140.2, 135.1, 134.1, 133.9, 129.9, 129.0, 128.7, 128.2, 128.1, 127.3, 127.0, 126.8, 126.6, 126.3, 124.3, 124.0, 53.7, 23.4.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 1735, 1654, 1484, 1320, 1160, 1135, 802, 767, 733, 697, 677.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{24}\text{H}_{21}\text{NNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 410.1185, found: 410.1196.

#### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)thiophene-2-sulfonamide (**19**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), thiophene-2-sulfonamide (**2q**, 32.6 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **19** as a white solid (27.5 mg, 0.080 mmol, 40% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.55 – 7.51 (m, 2H), 7.49 (dd,  $J = 5.0, 1.4$  Hz, 1H), 7.48 – 7.40 (m, 5H), 7.38 – 7.31 (m, 1H), 7.25 – 7.18 (m, 2H), 6.94 (dd,  $J = 5.0, 3.7$  Hz, 1H), 5.04 (d,  $J = 7.1$  Hz, 1H), 4.65 – 4.58 (m, 1H), 1.52 (d,  $J = 6.9$  Hz, 3H).

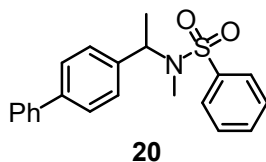
$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  141.8, 140.8, 140.6, 140.6, 132.3, 131.7, 128.8, 127.4, 127.3, 127.1,

127.0, 126.5, 53.8, 23.4.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3428, 1651, 1324, 1156, 1055 1033, 1015.

HRMS (ESI-TOF, m/z) calcd for C<sub>18</sub>H<sub>17</sub>NNaO<sub>2</sub>S<sub>2</sub><sup>+</sup> (M+Na)<sup>+</sup>: 366.0951, found: 366.0953.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-*N*-methylbenzenesulfonamide (**20**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *N*-methylbenzenesulfonamide (**2r**, 34.2 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **20** as a white solid (54.8 mg, 0.156 mmol, 78% yield).

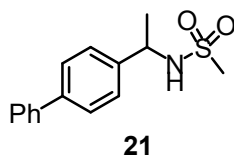
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.91 – 7.86 (m, 2H), 7.62 – 7.51 (m, 7H), 7.46 – 7.41 (m, 2H), 7.38 – 7.31 (m, 3H), 5.34 (q,  $J = 7.0$  Hz, 1H), 2.64 (s, 3H), 1.34 (d,  $J = 7.0$  Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  140.6, 140.5, 140.3, 138.8, 132.4, 129.1, 128.8, 127.7, 127.4, 127.1, 127.1, 127.1, 54.7, 28.6, 15.5.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3727, 3582, 1735, 1685, 1654, 1560, 1332, 1148, 417.

HRMS (ESI-TOF, m/z) calcd for C<sub>21</sub>H<sub>21</sub>NNaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 374.1185, found: 374.1180.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)methanesulfonamide (**21**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), methanesulfonamide (**2s**, 19.0 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan

cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **21** as a white solid (40.2 mg, 0.146 mmol, 73% yield).

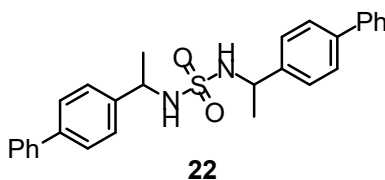
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.66 – 7.57 (m, 4H), 7.49 – 7.40 (m, 4H), 7.40 – 7.30 (m, 1H), 5.07 (d, *J* = 7.0 Hz, 1H), 4.75 – 4.67 (m, 1H), 2.69 (s, 3H), 1.59 (d, *J* = 6.9 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 141.5, 140.9, 140.4, 128.9, 127.6, 127.5, 127.0, 126.7, 53.5, 41.9, 24.0.

IR (film): ν (cm<sup>-1</sup>) 3428, 2973, 1654, 1315, 1054, 1033, 1016, 428.

HRMS (ESI-TOF, *m/z*) calcd for C<sub>15</sub>H<sub>17</sub>NNaO<sub>2</sub>S<sup>+</sup> (*M*+*Na*)<sup>+</sup>: for 298.0876, found: 298.0872.

#### 4,4-(sulfonylbis(propane-1,2-diyl))*di*-1,1'-biphenyl (**22**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 91.1 mg, 0.50 mmol), sulfonyl diamide (**2t**, 48.0 mg, 0.50 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **22** as a white solid (56.6 mg, 0.124 mmol, 62% yield, 3.3:1 *d.r.*), regional selectivity was determined by crude <sup>1</sup>H NMR analysis.

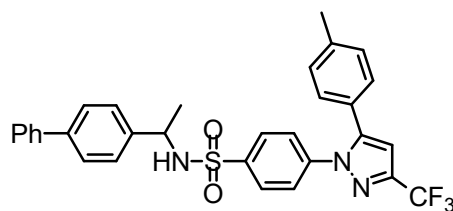
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.57 – 7.51 (m, 6H), 7.47 – 7.38 (m, 5H), 7.38 – 7.31 (m, 6H), 7.22 – 7.18 (m, 1 H), 4.62 – 4.38 (m, 4H), 1.53 – 1.31 (m, 6H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 142.0, 140.6, 140.6, 128.8, 127.4, 127.4, 127.0, 126.7, 53.5, 23.5.

IR (film): ν (cm<sup>-1</sup>) 3278, 3029, 2973, 2928, 1912, 1804, 1752, 1735, 1664, 1600, 1561, 1519, 1487, 1450, 1427, 1376, 1318, 1263, 1211, 1155, 1126, 1103, 1078, 1022, 1008, 977, 895, 838, 799, 765, 734, 697, 620, 569.

HRMS (ESI-TOF, *m/z*) calcd for C<sub>28</sub>H<sub>28</sub>N<sub>2</sub>NaO<sub>2</sub>S<sup>+</sup> (*M*+*Na*)<sup>+</sup>: 479.1764, found: 479.1766.

***N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-4-(5-(*p*-tolyl)-3-(trifluoromethyl)-1*H*-pyrazol-1-yl)benzenesulfonamide (**23**)**



**23**

A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), Celecoxib (**2u**, 76.3 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **23** as a white solid (94.4 mg, 0.168 mmol, 84% yield).

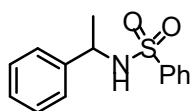
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.69 – 7.64 (m, 2H), 7.48 – 7.43 (m, 2H), 7.41 – 7.38 (m, 4H), 7.35 – 7.32 (m, 1H), 7.32 – 7.28 (m, 2H), 7.16 – 7.09 (m, 4H), 7.06 – 6.99 (m, 2H), 6.72 (s, 1H), 5.23 (d,  $J = 7.0$  Hz, 1H), 4.60 – 4.53 (m, 1H), 2.34 (s, 3H), 1.48 (d,  $J = 6.9$  Hz, 3H).

<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  145.0, 143.9 (q,  $J = 37.9$  Hz), 142.0, 140.5 (d,  $J = 18.0$  Hz), 140.4 (d,  $J = 24.0$  Hz), 140.3, 140.1, 139.6, 129.6, 128.7, 128.6, 127.9, 127.3, 127.1, 126.8, 126.6 (d,  $J = 18.0$  Hz), 125.6, 125.0, 121.1 (q,  $J = 268.8$  Hz), 106.2, 53.6, 23.3, 21.2.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3462, 1654, 1470, 1407, 1371, 1327, 1271, 1236, 1160 1132, 1093, 975, 838, 765, 425.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>31</sub>H<sub>26</sub>F<sub>3</sub>N<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 584.1590, found: 584.1592.

***N*-(1-phenylethyl)benzenesulfonamide (**24**)**



**24**

A dried 10 mL Schlenk tube was charged with ethylbenzene (**1b**, 63.7 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and



dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **24** as a pale yellow oil (34.0 mg, 0.130 mmol, 65% yield).

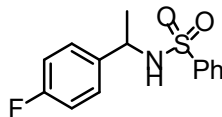
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.77 – 7.69 (m, 2H), 7.53 – 7.43 (m, 1H), 7.39 – 7.36 (m, 2H), 7.20 – 7.15 (m, 3H), 7.12 – 7.03 (m, 2H), 4.96 (d,  $J = 7.1$  Hz, 1H), 4.53 – 4.47 (m, 1H), 1.44 (d,  $J = 6.8$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  141.8, 140.7, 132.3, 128.8, 128.6, 127.6, 127.0, 126.1, 53.7, 23.6.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3278, 3064, 3031, 2976, 2929, 1735, 1654, 1603, 1560, 1495, 1481, 1447, 1427, 1377, 1324, 1208, 1162, 1121, 1092, 1020, 1000, 961, 868, 785, 754, 721, 700, 689, 613, 581, 545.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{14}\text{H}_{15}\text{NNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 284.0716, found: 284.0735.

#### ***N*-(1-(4-fluorophenyl)ethyl)benzenesulfonamide (25)**



**25**

A dried 10 mL Schlenk tube was charged with 1-ethyl-4-fluorobenzene (**1c**, 74.5 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **25** as a pale yellow oil (46.9 mg, 0.168 mmol, 84% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.16 – 8.09 (m, 2H), 7.96 – 7.86 (m, 1H), 7.85 – 7.74 (m, 2H), 7.54 – 7.42 (m, 2H), 7.32 – 7.19 (m, 2H), 5.73 (d,  $J = 6.7$  Hz, 1H), 4.94 – 4.87 (m, 1H), 1.82 (d,  $J = 6.9$  Hz, 3H).

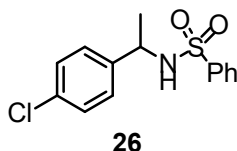
$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  162.5 (d,  $J = 119.7$  Hz), 140.6, 137.7 (d,  $J = 3.8$  Hz), 132.4, 128.8,

127.8 (d,  $J = 8.8$  Hz), 127.0, 115.3 (d,  $J = 18.9$  Hz), 53.1, 23.6.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3277, 2977, 1735, 1654, 1605, 1510, 1448, 1379, 1323, 1225, 1161, 1120, 1090, 1023, 961, 868, 836, 755, 743, 719, 689, 598, 426.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{14}\text{H}_{14}\text{FNNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 302.0621, found: 302.0625.

#### ***N*-(1-(4-chlorophenyl)ethyl)benzenesulfonamide (26)**



A dried 10 mL Schlenk tube was charged with 1-chloro-4-ethylbenzene (**1d**, 84.4 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **26** as a pale yellow oil (36.7 mg, 0.124 mmol, 62% yield).

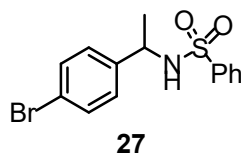
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.73 – 7.66 (m, 2H), 7.55 – 7.45 (m, 1H), 7.42 – 7.33 (m, 2H), 7.17 – 7.11 (m, 2H), 7.06 – 6.99 (m, 2H), 5.01 (d,  $J = 6.8$  Hz, 1H), 4.52 – 4.44 (m, 1H), 1.40 (d,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.5, 140.4, 133.3, 132.5, 128.9, 128.6, 127.6, 127.0, 53.1, 23.5.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3451, 1654, 1492 1447, 1323, 1160, 1090, 1014, 961, 827, 754, 722, 689, 422.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{14}\text{H}_{14}\text{ClNNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 318.0326, found: 318.0327.

#### ***N*-(1-(4-bromophenyl)ethyl)benzenesulfonamide (27)**



A dried 10 mL Schlenk tube was charged with 1-bromo-4-ethylbenzene (**1e**, 111.0 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk

tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **27** as a pale yellow oil (57.2 mg, 0.168 mmol, 84% yield).

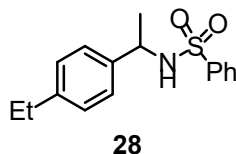
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.71 – 7.68 (m, 2H), 7.55 – 7.46 (m, 1H), 7.42 – 7.33 (m, 2H), 7.31 – 7.26 (m, 2H), 6.99 – 6.92 (m, 2H), 5.18 (d,  $J = 10.0 \text{ Hz}$ , 1H), 4.48 – 4.42 (m, 1H), 1.39 (d,  $J = 6.9 \text{ Hz}$ , 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.9, 140.5, 132.5, 131.6, 128.9, 127.9, 127.0, 121.4, 53.1, 23.4.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3277, 3066, 2976, 1735, 1685, 1654, 1592, 1560, 1489, 1447, 1378, 1324, 1161, 1124, 1090, 1011, 961, 868, 823, 779, 754, 722, 689, 645, 588.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{14}\text{H}_{14}\text{BrNNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 361.9821, found: 361.9824.

#### ***N*-(1-(4-ethylphenyl)ethyl)benzenesulfonamide (28)**



A dried 10 mL Schlenk tube was charged with 1,4-diethylbenzene (**1f**, 80.5 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **28** as a pale yellow oil (40.5 mg, 0.140 mmol, 70% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75 – 7.69 (m, 2H), 7.52 – 7.45 (m, 1H), 7.42 – 7.32 (m, 2H), 7.04 – 6.94 (m, 4H), 4.70 (d,  $J = 6.9 \text{ Hz}$ , 1H), 4.50 – 4.43 (m, 1H), 2.57 (q,  $J = 7.6 \text{ Hz}$ , 2H), 1.43 (d,  $J = 6.9 \text{ Hz}$ , 3H), 1.18 (t,  $J = 7.6 \text{ Hz}$ , 3H).

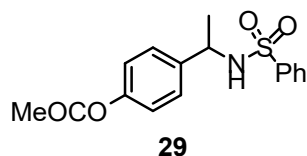
$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  143.6, 140.7, 139.1, 132.3, 128.8, 128.0, 127.1, 126.1, 53.5, 28.4, 23.5, 15.6.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3278, 2966, 2930, 2872, 1654, 1617, 1514, 1447, 1376, 1324, 1162, 1127, 1092,

1019, 963, 868, 831, 754, 722, 689, 599.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $C_{16}H_{19}NNaO_2S^+$  ( $M+Na$ ) $^+$ : 312.1029, found: 312.1033.

#### 4-(1-(phenylsulfonamido)ethyl)phenyl acetate (**29**)



A dried 10 mL Schlenk tube was charged with 4-ethylphenyl acetate (**1g**, 98.5 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **29** as a pale yellow oil (45.4 mg, 0.142 mmol, 71% yield).

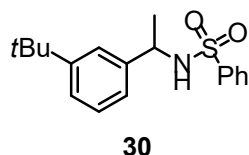
$^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  7.74 – 7.66 (m, 2H), 7.53 – 7.43 (m, 1H), 7.43 – 7.32 (m, 2H), 7.16 – 7.04 (m, 2H), 6.92 – 6.84 (m, 2H), 5.15 (d,  $J = 7.2$  Hz, 1H), 4.54 – 4.46 (m, 1H), 2.27 (s, 3H), 1.40 (d,  $J = 6.9$  Hz, 3H).

$^{13}C$  NMR (126 MHz,  $CDCl_3$ )  $\delta$  169.3, 149.9, 140.6, 139.4, 132.4, 128.9, 127.2, 127.0, 121.6, 53.2, 23.5, 21.1.

IR (film):  $\nu$  ( $cm^{-1}$ ) 3281, 3066, 2977, 2932, 1970, 1902, 1758, 1665, 1606, 1508, 1481, 1448, 1431, 1371, 1324, 1198, 1161, 1122, 1090, 1018, 962, 913, 811, 755, 720, 690, 656, 594, 569, 546.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $C_{16}H_{21}N_2O_4S^+$  ( $M+NH_4$ ) $^+$ : 337.1217, found: 337.1218.

#### *N*-(1-(3-(*tert*-butyl)phenyl)ethyl)benzenesulfonamide (**30**)



A dried 10 mL Schlenk tube was charged with 1-(*tert*-butyl)-3-ethylbenzene (**1h**, 97.4 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk

tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **30** as a pale yellow oil (38.1 mg, 0.120 mmol, 60% yield).

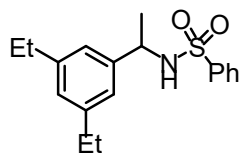
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.74 – 7.69 (m, 2H), 7.48 – 7.43 (m, 1H), 7.38 – 7.32 (m, 2H), 7.19 (m, 1H), 7.11 (t,  $J = 7.7 \text{ Hz}$ , 1H), 7.05 (t,  $J = 1.9 \text{ Hz}$ , 1H), 6.90 (t,  $J = 7.4$ , 1H), 5.02 (d,  $J = 7.2 \text{ Hz}$ , 1H), 4.54 – 4.46 (m, 1H), 1.46 (d,  $J = 6.9 \text{ Hz}$ , 3H), 1.21 (s, 9H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  151.5, 141.4, 140.7, 132.3, 128.8, 128.3, 127.0, 124.6, 123.2, 123.1, 54.1, 34.6, 31.3, 23.7.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3726, 3277, 2963, 1735, 1654, 1560, 1447, 1323, 1160, 1092.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{18}\text{H}_{23}\text{NNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 340.1342, found: 340.1344.

#### *N*-(1-(3,5-diethylphenyl)ethyl)benzenesulfonamide (**31**)



**31**

A dried 10 mL Schlenk tube was charged with 1,3,5-triethylbenzene (**1i**, 97.4 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **31** as a pale yellow oil (33.0 mg, 0.104 mmol, 52% yield).

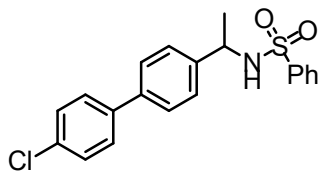
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.78 – 7.65 (m, 2H), 7.50 – 7.42 (m, 1H), 7.41 – 7.31 (m, 2H), 6.83 (d,  $J = 1.7 \text{ Hz}$ , 1H), 6.68 (d,  $J = 1.6 \text{ Hz}$ , 2H), 4.87 (d,  $J = 7.1 \text{ Hz}$ , 1H), 4.49 – 4.41 (m, 1H), 2.49 (q,  $J = 7.6 \text{ Hz}$ , 4H), 1.45 (d,  $J = 6.9 \text{ Hz}$ , 3H), 1.14 (t,  $J = 7.6 \text{ Hz}$ , 6H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  144.6, 141.7, 140.8, 132.2, 128.7, 127.0, 126.7, 123.0, 53.9, 28.7, 23.5, 15.5.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3727, 3278, 2966, 1735, 1685, 1654, 1604, 1560, 1447, 1324, 1159, 1093, 870, 753, 721, 688, 429.

HRMS (ESI-TOF, m/z) calcd for C<sub>18</sub>H<sub>23</sub>NNaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 340.1342, found: 340.1351.

### *N*-(1-(4'-chloro-[1,1'-biphenyl]-4-yl)ethyl)benzenesulfonamide (**32**)



**32**

A dried 10 mL Schlenk tube was charged with 4-chloro-4'-ethyl-1,1'-biphenyl (**1j**, 65.0 mg, 0.30 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}}$  = 395 nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **32** as a white solid (38.7 mg, 0.104 mmol, 52% yield).

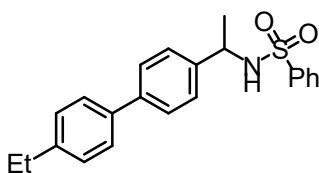
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.77 – 7.69 (m, 1H), 7.50 – 7.45 (m, 1H), 7.45 – 7.32 (m, 4H), 7.20 – 7.10 (m, 1H), 5.15 (d, *J* = 7.1 Hz, 1H), 4.58 – 4.51 (m, 1H), 1.46 (d, *J* = 6.9 Hz, 2H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  141.3, 140.7, 139.2, 139.1, 133.5, 132.3, 129.0, 128.8, 128.2, 127.1, 126.7, 53.4, 23.5.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3726, 3278, 3063, 1735, 1654, 1598, 1560, 1486, 1448, 1324, 1162, 1091, 1028, 1005, 961, 818, 754, 721, 689, 589, 557.

HRMS (ESI-TOF, m/z) calcd for C<sub>20</sub>H<sub>18</sub>ClNNaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 394.0639, found: 394.0639.

### *N*-(1-(4'-ethyl-[1,1'-biphenyl]-4-yl)ethyl)benzenesulfonamide (**33**)



**33**

A dried 10 mL Schlenk tube was charged with 4,4'-diethyl-1,1'-biphenyl (**1k**, 63.1 mg, 0.30 mmol),

benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **33** as a white solid (62.9 mg, 0.172 mmol, 86% yield).

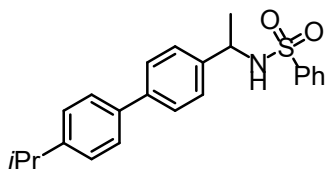
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.68 – 7.62 (m, 2H), 7.41 – 7.31 (m, 3H), 7.30 – 7.22 (m, 4H), 7.21 – 7.14 (m, 2H), 7.08 – 7.02 (m, 2H), 5.25 (d,  $J = 7.2$  Hz, 1H), 4.52 – 4.44 (m, 1H), 2.60 (q,  $J = 7.6$  Hz, 2H), 1.37 (d,  $J = 6.9$  Hz, 3H), 1.19 (t,  $J = 7.6$  Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  143.5, 140.7, 140.6, 140.4, 138.0, 132.3, 128.8, 128.3, 127.1, 127.1, 127.0, 126.6, 53.5, 28.5, 23.5, 15.6.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3449, 2966, 1735, 1654, 1560, 1497, 1446, 1322, 1158, 1090, 1033, 960, 820, 753, 720, 688, 425.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>22</sub>H<sub>23</sub>NNaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 388.1342, found: 388.1344.

#### *N*-(1-(4'-isopropyl-[1,1'-biphenyl]-4-yl)ethyl)benzenesulfonamide (**34**)



**34**

A dried 10 mL Schlenk tube was charged with 4-ethyl-4'-isopropyl-1,1'-biphenyl (**11**, 67.3 mg, 0.30 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **34** as a white solid (41.7 mg, 0.110 mmol, 55% yield).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.76 – 7.72 (m, 2H), 7.46 (dd,  $J = 16.4, 7.8$  Hz, 3H), 7.39 – 7.34 (m, 4H), 7.32 – 7.27 (m, 2H), 7.14 (d,  $J = 8.1$  Hz, 2H), 5.16 (d,  $J = 7.1$  Hz, 1H), 4.59 – 4.52 (m, 1H), 3.02

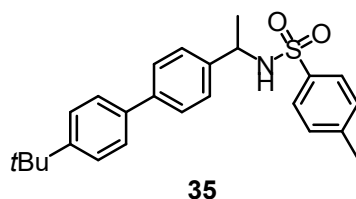
– 2.91 (m, 1H), 1.48 (d,  $J = 6.9$  Hz, 3H), 1.30 (d,  $J = 6.8$  Hz, 6H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  148.2, 140.7, 140.5, 140.4, 138.1, 132.3, 128.8, 127.1, 127.1, 126.9, 126.9, 126.6, 53.5, 33.8, 24.0, 23.5.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3277, 2961, 1735, 1685, 1654, 1617, 1560, 1497, 1447, 1324, 1161, 1091, 1019, 962, 821, 753, 721, 689, 433.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{23}\text{H}_{25}\text{NNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 402.1498, found: 402.1501.

### *N*-(1-(4'-(*tert*-butyl)-[1,1'-biphenyl]-4-yl)ethyl)-4-methylbenzenesulfonamide (**35**)



A dried 10 mL Schlenk tube was charged with 4-(*tert*-butyl)-4'-ethyl-1,1'-biphenyl (**1m**, 71.5 mg, 0.30 mmol), *p*-methylbenzenesulfonamide (**2a**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **35** as a white solid (62.0 mg, 0.152 mmol, 76% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.66 – 7.59 (m, 2H), 7.46 (s, 4H), 7.42 – 7.37 (m, 2H), 7.20 – 7.13 (m, 4H), 5.20 (d,  $J = 7.1$  Hz, 1H), 4.56 – 4.48 (m, 1H), 2.36 (s, 3H), 1.46 (d,  $J = 6.9$  Hz, 3H), 1.37 (s, 9H).

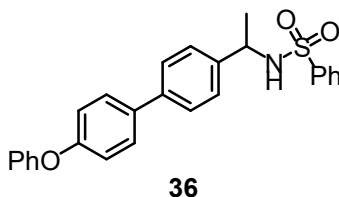
$^{13}\text{C}$  NMR (151 MHz,  $\text{CDCl}_3$ )  $\delta$  150.4, 143.1, 140.7, 140.3, 137.8, 137.7, 129.4, 127.2, 127.1, 126.7, 126.6, 125.7, 53.4, 34.6, 31.4, 23.5, 21.5.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3662, 3514, 3275, 3029, 2964, 2869, 2305, 1912, 1794, 1655, 1599, 1497, 1460, 1430, 1377, 1363, 1324, 1267, 1210, 1185, 1160, 1091, 1020, 1004, 961, 868, 852, 822, 738, 706, 671, 576, 552.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{25}\text{H}_{29}\text{NNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 430.1811, found: 430.1813.



### *N*-(1-(4'-phenoxy-[1,1'-biphenyl]-4-yl)ethyl)benzenesulfonamide (**36**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-4'-phenoxy-1,1'-biphenyl (**1n**, 82.3 mg, 0.30 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **36** as a white solid (53.3 mg, 0.124 mmol, 62% yield).

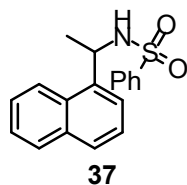
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.79 – 7.70 (m, 2H), 7.54 – 7.43 (m, 3H), 7.42 – 7.32 (m, 6H), 7.18 – 7.10 (m, 3H), 7.10 – 7.03 (m, 4H), 4.98 (m, 1H), 4.59 – 4.52 (m, 1H), 1.47 (d,  $J = 6.9$  Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  157.0, 140.7, 140.6, 140.6, 139.8, 135.6, 132.3, 129.8, 128.8, 128.3, 127.1, 127.0, 126.6, 123.5, 119.1, 119.0, 53.5, 23.5.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3421, 2950, 2843, 2137, 1654, 1454, 1413, 1239, 1108, 1032, 1019, 429.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>26</sub>H<sub>23</sub>NNaO<sub>3</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 452.1291, found: 452.1300.

### *N*-(1-(naphthalen-1-yl)ethyl)benzenesulfonamide (**37**)



A dried 10 mL Schlenk tube was charged with 1-ethylnaphthalene (**1o**, 93.7 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **37** as a brown oil (54.2 mg, 0.174 mmol, 87% yield).

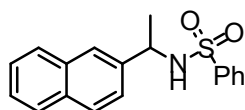
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.85 – 7.80 (m, 1H), 7.73 – 7.68 (m, 1H), 7.62 – 7.55 (m, 3H), 7.38 – 7.34 (m, 2H), 7.32 – 7.24 (m, 2H), 7.22 – 7.13 (m, 3H), 5.32 – 5.20 (m, 2H), 1.50 (d,  $J$  = 6.6 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.5, 137.5, 133.8, 132.3, 130.2, 128.9, 128.7, 128.2, 127.0, 126.4, 125.7, 125.2, 123.4, 122.6, 49.9, 23.3.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3726, 3281, 1735, 1654, 1617, 1560, 1447, 1326, 1159, 1093, 800, 778, 725, 688.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{18}\text{H}_{17}\text{NNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 334.0872, found: 334.0880.

### *N*-(1-(naphthalen-2-yl)ethyl)benzenesulfonamide (**38**)



**38**

A dried 10 mL Schlenk tube was charged with 2-ethylnaphthalene (**1p**, 93.7 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}}$  = 365 nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **38** as a brown oil (46.7 mg, 0.150 mmol, 75% yield).

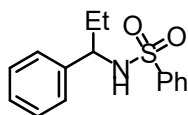
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.69 – 7.57 (m, 4H), 7.56 (d,  $J$  = 8.5 Hz, 1H), 7.43 (d,  $J$  = 1.8 Hz, 1H), 7.38 – 7.32 (m, 2H), 7.31 – 7.25 (m, 1H), 7.20 – 7.15 (m, 2H), 7.12 (dd,  $J$  = 8.5, 1.8 Hz, 1H), 5.16 (d,  $J$  = 7.1 Hz, 1H), 4.63 – 4.55 (m, 1H), 1.43 (d,  $J$  = 6.9 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.6, 139.1, 133.1, 132.7, 132.3, 128.7, 128.5, 127.8, 127.5, 127.0, 126.2, 126.0, 125.1, 124.0, 53.9, 23.4.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3277, 3057, 2976, 2930, 1735, 1685, 1654, 1601, 1560, 1508, 1447, 1426, 1379, 1327, 1161, 1130, 1018, 969, 944, 881, 858, 819, 751, 723, 689, 665, 595, 571.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{18}\text{H}_{17}\text{NNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 334.0872, found: 334.0889.

### *N*-(1-phenylpropyl)benzenesulfonamide (**39**)



**39**

A dried 10 mL Schlenk tube was charged with propylbenzene (**1q**, 72.1 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **39** as a pale yellow oil (24.8 mg, 0.090 mmol, 45% yield).

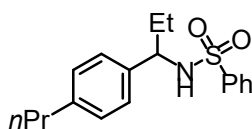
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.65 (d,  $J = 8.1 \text{ Hz}$ , 2H), 7.42 (t,  $J = 7.5 \text{ Hz}$ , 1H), 7.31 (t,  $J = 7.8 \text{ Hz}$ , 2H), 7.18 – 7.09 (m, 3H), 6.99 (dt,  $J = 6.7, 2.8 \text{ Hz}$ , 2H), 4.97 (d,  $J = 7.2 \text{ Hz}$ , 1H), 4.22 (q,  $J = 7.3 \text{ Hz}$ , 1H), 1.91 – 1.64 (m, 2H), 0.80 (t,  $J = 7.4 \text{ Hz}$ , 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  140.7, 140.5, 132.2, 128.7, 128.4, 127.4, 127.0, 126.5, 59.9, 30.6, 10.4.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3726, 3278, 2925, 1735, 1654, 1560, 1494, 1448, 1322, 1161, 1092, 753, 720, 688.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>15</sub>H<sub>17</sub>NNaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 298.0872, found: 298.0880.

### *N*-(1-(4-propylphenyl)propyl)benzenesulfonamide (**40**)



**40**

A dried 10 mL Schlenk tube was charged with 1,4-dipropylbenzene (**1r**, 97.4 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LED ( $\lambda_{\text{max}} = 365 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **40** as a pale yellow oil (39.4 mg, 0.124 mmol, 62% yield).

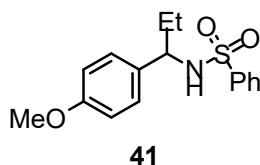
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.66 – 7.62 (m, 2H), 7.43 – 7.37 (m, 1H), 7.32 – 7.27 (m, 2H), 6.96 – 6.85 (m, 4H), 5.10 (d,  $J = 7.5$  Hz, 1H), 4.19 (q,  $J = 7.3$  Hz, 1H), 2.47 (t,  $J = 8.5$  Hz, 2H), 1.87 – 1.75 (m, 1H), 1.76 – 1.66 (m, 1H), 1.62 – 1.50 (m, 2H), 0.90 (t,  $J = 7.3$  Hz, 3H), 0.79 (t,  $J = 7.4$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  141.8, 140.8, 137.7, 132.1, 128.6, 128.5, 127.0, 126.4, 59.7, 37.6, 30.6, 24.5, 13.8, 10.5.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3853, 3790, 3727, 3629, 3602, 3278, 2962, 2931, 2872, 1735, 1685, 1654, 1617, 1560, 1513, 1448, 1322, 1161, 1093, 1049, 920, 802, 753, 720, 688, 425.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{18}\text{H}_{23}\text{NNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 340.1342, found: 340.1345.

#### ***N*-(1-(4-methoxyphenyl)propyl)benzenesulfonamide (41)**



A dried 10 mL Schlenk tube was charged with 1-methoxy-4-propylbenzene (**1s**, 90.1 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **41** as a pale yellow oil (28.7 mg, 0.094 mmol, 47% yield).

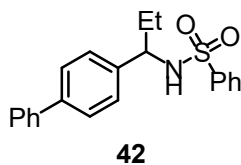
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.68 – 7.62 (m, 2H), 7.47 – 7.40 (m, 1H), 7.36 – 7.29 (m, 2H), 6.94 – 6.88 (m, 2H), 6.69 – 6.62 (m, 2H), 5.05 (d,  $J = 7.2$  Hz, 1H), 4.16 (q,  $J = 7.2$  Hz, 1H), 3.73 (s, 3H), 1.86 – 1.65 (m, 2H), 0.77 (t,  $J = 7.4$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  158.8, 140.8, 132.6, 132.1, 128.6, 127.7, 127.0, 113.8, 59.4, 55.2, 30.5, 10.5.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3279, 2964, 1735, 1685, 1654, 1612, 1560, 1513, 1448, 1321, 1250, 1160, 1092, 1030, 829, 754, 719, 689, 425.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{16}\text{H}_{19}\text{NNaO}_3\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 328.0978, found: 328.0981.

### *N*-(1-([1,1'-biphenyl]-4-yl)propyl)benzenesulfonamide (**42**)



A dried 10 mL Schlenk tube was charged with 4-propyl-1,1'-biphenyl (**1t**, 58.9 mg, 0.30 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **42** as a white solid (54.1 mg, 0.154 mmol, 77% yield).

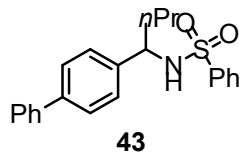
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.68 – 7.62 (m, 2H), 7.52 – 7.47 (m, 2H), 7.45 – 7.40 (m, 3H), 7.38 – 7.33 (m, 3H), 7.33 – 7.27 (m, 2H), 7.08 – 7.03 (m, 2H), 4.93 (d,  $J = 7.2 \text{ Hz}$ , 1H), 4.28 (q,  $J = 7.3 \text{ Hz}$ , 1H), 1.91 – 1.70 (m, 2H), 0.84 (t,  $J = 7.4 \text{ Hz}$ , 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  140.8, 140.6, 140.4, 139.5, 132.1, 128.8, 128.7, 127.4, 127.1, 127.0, 127.0, 127.0, 59.7, 30.5, 10.5.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3727, 3409, 2925, 1654, 1617, 1447, 1321, 1160, 1091, 765.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>21</sub>H<sub>21</sub>NNaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 374.1185, found: 374.1198.

### *N*-(1-([1,1'-biphenyl]-4-yl)butyl)benzenesulfonamide (**43**)



A dried 10 mL Schlenk tube was charged with 4-butyl-1,1'-biphenyl (**1u**, 63.1 mg, 0.30 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **43** as a white solid (49.7 mg, 0.136 mmol, 68% yield).

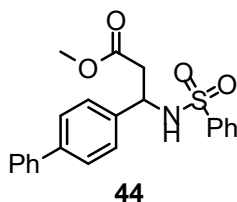
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.68 – 7.64 (m, 2H), 7.51 – 7.47 (m, 2H), 7.45 – 7.38 (m, 3H), 7.36 – 7.31 (m, 3H), 7.30 – 7.26 (m, 2H), 7.09 – 7.04 (m, 2H), 5.37 (d,  $J = 7.6$  Hz, 1H), 4.37 (q,  $J = 7.4$  Hz, 1H), 1.82 – 1.68 (m, 2H), 1.35 – 1.19 (m, 2H), 0.85 (t,  $J = 7.4$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.7, 140.7, 140.2, 139.9, 132.1, 128.8, 128.6, 127.3, 127.1, 127.0, 127.0, 127.0, 57.9, 39.6, 19.2, 13.6.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3277, 3059, 3029, 2961, 2931, 2873, 1735, 1654, 1599, 1487, 1447, 1322, 1161, 1093, 1008, 927, 838, 766, 754, 735, 720, 689, 587, 560.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{22}\text{H}_{23}\text{NNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 388.1342, found: 388.1355.

#### methyl 3-([1,1'-biphenyl]-4-yl)-3-(phenylsulfonamido)propanoate (**44**)



A dried 10 mL Schlenk tube was charged with methyl 3-([1,1'-biphenyl]-4-yl)propanoate (**1v**, 72.1 mg, 0.30 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **44** as a white solid (41.9 mg, 0.106 mmol, 53% yield).

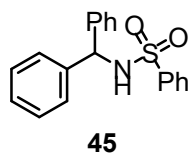
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75 – 7.70 (m, 2H), 7.52 – 7.48 (m, 2H), 7.48 – 7.44 (m, 1H), 7.44 – 7.40 (m, 2H), 7.40 – 7.31 (m, 5H), 7.19 – 7.12 (m, 2H), 5.93 (d,  $J = 7.7$  Hz, 1H), 4.83 (dt,  $J = 8.0, 6.3$  Hz, 1H), 3.58 (s, 3H), 2.97 – 2.73 (m, 2H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  171.1, 140.7, 140.4, 140.4, 138.1, 132.4, 128.9, 128.8, 127.5, 127.3, 127.1, 127.0, 126.9, 54.2, 52.0, 41.1.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3279, 3060, 3030, 2954, 1735, 1601, 1560, 1488, 1447, 1437, 1325, 1291, 1262, 1206, 1162, 1093, 1073, 1008, 964, 839, 799, 767, 755, 735, 689, 588.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{22}\text{H}_{21}\text{NNaO}_4\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 418.1083, found: 418.1087.

### *N*-benzhydrylbenzenesulfonamide (**45**)



A dried 10 mL Schlenk tube was charged with diphenylmethane (**1w**, 100.9 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **45** as a white solid (55.0 mg, 0.170 mmol, 85% yield).

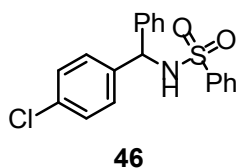
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.73 – 7.64 (m, 2H), 7.51 – 7.42 (m, 1H), 7.33 (t,  $J = 7.8$  Hz, 2H), 7.24 – 7.16 (m, 6H), 7.13 – 7.05 (m, 4H), 5.61 (d,  $J = 7.2$  Hz, 1H), 5.23 (d,  $J = 7.2$  Hz, 1H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  140.4, 140.4, 132.4, 128.8, 128.6, 127.7, 127.4, 127.1, 61.4.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3727, 3267, 2922, 1654, 1585, 1493, 1457, 1448, 1348, 1315, 1165, 1083, 1044, 1029, 938, 923, 905, 830, 754, 743, 723, 699, 686, 590.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>19</sub>H<sub>17</sub>NNaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 346.0872, found: 346.0874.

### *N*-((4-chlorophenyl)(phenyl)methyl)benzenesulfonamide (**46**)



A dried 10 mL Schlenk tube was charged with 1-benzyl-4-chlorobenzene (**1x**, 121.6 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **46** as a white solid (46.5 mg, 0.130 mmol, 65% yield).

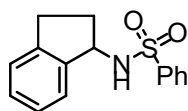
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70 – 7.64 (m, 2H), 7.51 – 7.44 (m, 1H), 7.34 (t,  $J = 7.9$  Hz, 2H), 7.22 – 7.18 (m, 3H), 7.18 – 7.13 (m, 2H), 7.09 – 7.00 (m, 4H), 5.59 – 5.53 (m, 2H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.2, 139.9, 139.0, 133.5, 132.5, 128.8, 128.8, 128.7, 128.7, 127.9, 127.3, 127.1, 60.8.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3279, 2109, 1654, 1491, 1448, 1324, 1161, 1091, 1054, 1014, 932, 843, 799, 753, 724, 688, 589, 557, 422.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{16}\text{H}_{21}\text{NNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 380.0482, found: 380.0492.

### *N*-(2,3-dihydro-1*H*-inden-1-yl)benzenesulfonamide (**47**)



**47**

A dried 10 mL Schlenk tube was charged with 2,3-dihydro-1*H*-indene (**1y**, 70.9 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **47** as a white solid (42.6 mg, 0.156 mmol, 78% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.99 – 7.93 (m, 2H), 7.64 – 7.58 (m, 1H), 7.54 (t,  $J = 8.4$ , 2H), 7.23 – 7.16 (m, 2H), 7.16 – 7.10 (m, 1H), 7.06 (d,  $J = 7.5$  Hz, 1H), 4.90 (d,  $J = 9.0$  Hz, 1H), 4.92 – 4.81 (m, 1H), 2.94 – 2.86 (m, 1H), 2.78 – 2.70 (m, 1H), 2.36 – 2.28 (m, 1H), 1.80 – 1.71 (m, 1H).

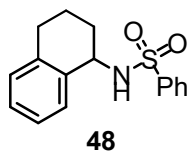
$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  142.8, 141.9, 141.3, 132.7, 129.2, 128.3, 127.1, 126.9, 124.8, 124.1, 58.8, 34.7, 30.0.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3726, 3277, 3068, 2930, 2852, 1735, 1685, 1654, 1605, 1560, 1478, 1447, 1328, 1214, 1162, 1094, 1073, 1024, 984, 911, 877, 753, 722, 690, 666, 605, 585, 553, 431, 422.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{15}\text{H}_{15}\text{NNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 296.0716, found: 296.0719.



### *N*-(1,2,3,4-tetrahydronaphthalen-1-yl)benzenesulfonamide (**48**)



A dried 10 mL Schlenk tube was charged with 1,2,3,4-tetrahydronaphthalene (**1z**, 79.3 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **48** as a white solid (35.6 mg, 0.124 mmol, 62% yield).

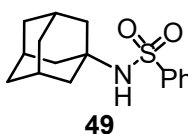
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.95 (m, 2H), 7.65 – 7.59 (m, 1H), 7.56 (t,  $J = 2.5$  Hz, 2H), 7.13 (t,  $J = 7.3, 1.4$  Hz, 1H), 7.08 – 7.01 (m, 2H), 6.92 – 6.86 (m, 1H), 4.67 (d,  $J = 7.8$  Hz, 1H), 4.51 – 4.46 (m, 1H), 2.80 – 2.61 (m, 2H), 1.91 – 1.78 (m, 3H), 1.78 – 1.68 (m, 1H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  141.2, 137.6, 135.5, 132.7, 129.3, 129.2, 128.7, 127.7, 127.1, 126.3, 52.0, 30.8, 28.9, 19.1.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3438, 1637, 1560, 1444, 1323, 1151, 1074.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>16</sub>H<sub>17</sub>NNaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 310.0872, found: 310.0874.

### *N*-((3s,5s,7s)-adamantan-1-yl)benzenesulfonamide (**49**)



A dried 10 mL Schlenk tube was charged with adamantane (**1za**, 109.0 mg, 0.80 mmol), benzenesulfonamide (**2b**, 62.9 mg, 0.40 mmol), Selectfluor<sup>®</sup> (425.1 mg, 1.20 mmol) and dichloromethane (2.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365$  nm) with a DC fan cooling. The reaction was stirred at 70 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **49** as a pale yellow oil (31.5 mg, 0.108 mmol, 27% yield).

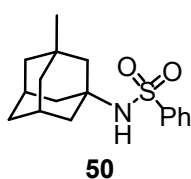
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.93 – 7.89 (m, 2H), 7.55 – 7.51 (m, 1H), 7.50 – 7.45 (m, 2H), 4.67 (s, 1H), 2.02 – 1.97 (m, 3H), 1.79 (d,  $J$  = 3.0 Hz, 6H), 1.61 – 1.53 (m, 6H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  144.0, 132.1, 128.8, 126.9, 55.2, 43.1, 35.8, 29.5.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3267, 2909, 2851, 1735, 1654, 1617, 1560, 1447, 1360, 1346, 1311, 1260, 1157, 1117, 1088, 994, 969, 873, 814, 756, 728, 690, 588, 560, 523, 503, 487, 469, 426.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{32}\text{H}_{49}\text{NNaO}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 314.1185, found: 314.1189.

### *N*-((1*s*,3*s*,5*R*,7*S*)-3-methyladamantan-1-yl)benzenesulfonamide (**50**)



A dried 10 mL Schlenk tube was charged with 1-methyladamantane (**1zb**, 120.2 mg, 0.80 mmol), benzenesulfonamide (**2b**, 62.9 mg, 0.40 mmol), Selectfluor<sup>®</sup> (425.1 mg, 1.20 mmol) and dichloromethane (2.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}}$  = 365 nm) with a DC fan cooling. The reaction was stirred at 70 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **50** as a pale yellow oil (37.9 mg, 0.124 mmol, 31% yield).

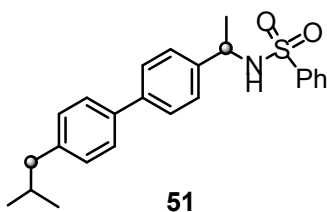
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92 – 7.88 (m, 2H), 7.56 – 7.52 (m, 1H), 7.51 – 7.45 (m, 2H), 4.54 (s, 1H), 2.06 – 2.01 (m, 2H), 1.74 (d,  $J$  = 11.4 Hz, 2H), 1.65 (d,  $J$  = 11.7 Hz, 2H), 1.51 (s, 2H), 1.50 – 1.45 (m, 2H), 1.35 – 1.28 (m, 4H), 0.77 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  144.0, 132.0, 128.8, 126.8, 56.0, 50.0, 42.9, 42.2, 35.0, 32.1, 30.2, 29.8.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3726, 3266, 2905, 2849, 1735, 1654, 1560, 1447, 1317, 1163, 1149, 1095, 1053, 979, 756, 718, 690, 587.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{17}\text{H}_{23}\text{NNaO}_2\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 328.1342, found: 328.1349.

### *N*-(1-(4'-isobutyl-[1,1'-biphenyl]-4-yl)ethyl)benzenesulfonamide (**51**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-4'-isobutyl-1,1'-biphenyl (**1zc**, 143.0 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max}$  = 365 nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **51-1** as a white solid (66.9 mg, 0.170 mmol, 85% yield, 10:1 *r.r.*), site-selectivity was determined by crude <sup>1</sup>H NMR analysis.

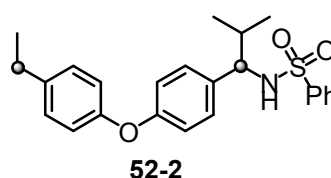
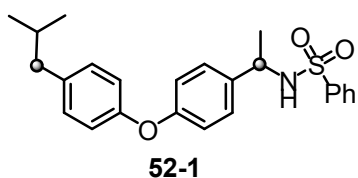
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.75 – 7.71 (m, 1H), 7.49 – 7.45 (m, 1H), 7.43 – 7.40 (m, 1H), 7.40 – 7.34 (m, 2H), 7.22 – 7.18 (m, 1H), 7.15 – 7.11 (m, 1H), 4.94 (d, *J* = 6.9 Hz, 1H), 4.59 – 4.51 (m, 1H), 2.51 (d, *J* = 7.1 Hz, 1H), 1.94 – 1.85 (m, 1H), 1.48 (d, *J* = 6.8 Hz, 2H), 0.94 (d, *J* = 6.6 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  141.0, 140.7, 140.5, 140.4, 132.3, 129.6, 128.8, 127.1, 126.7, 126.5, 53.5, 45.1, 30.2, 23.5, 22.4.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3727, 3409, 2956, 2875, 1735, 1685, 1654, 1617, 1560, 1513, 1465, 1414, 1383, 1366, 1237, 1165, 1073, 1004, 842, 800, 741, 688.

HRMS (ESI-TOF, *m/z*) calcd for C<sub>24</sub>H<sub>27</sub>NNaO<sub>2</sub>S<sup>+</sup> (*M*+Na)<sup>+</sup>: 416.1655, found: 416.1654.

### *N*-(1-(4-(4-isobutylphenoxy)phenyl)ethyl)benzenesulfonamide (**52**)



A dried 10 mL Schlenk tube was charged with 1-ethyl-4-(4-isobutylphenoxy)benzene (**1zd**, 152.6 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk

tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **52-1** and **52-2** as a pale yellow oil (total yield 0.120 mmol, 60%, **52-1**: **52-2** = 4.5:1 *r.r.*), site-selectivity was determined by isolated yield.

**52-1**:  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.76 – 7.72 (m, 2H), 7.54 – 7.48 (m, 1H), 7.41 (t,  $J = 7.7$  Hz, 2H), 7.12 – 7.07 (m, 2H), 7.05 – 6.99 (m, 2H), 6.88 – 6.83 (m, 2H), 6.81 – 6.76 (m, 2H), 4.77 (d,  $J = 6.8$  Hz, 1H), 4.53 – 4.46 (m, 1H), 2.46 (d,  $J = 7.2$  Hz, 2H), 1.90 – 1.80 (m, 1H), 1.43 (d,  $J = 6.8$  Hz, 3H), 0.91 (d,  $J = 6.6$  Hz, 6H).

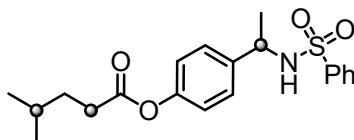
$^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  157.1, 154.8, 140.8, 136.9, 136.3, 132.3, 130.3, 128.8, 127.5, 127.1, 118.8, 118.4, 53.2, 44.7, 30.3, 23.5, 22.3.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3490, 3277, 3061, 3032, 2956, 2926, 2868, 2404, 1892, 1773, 1665, 1602, 1502, 1448, 1383, 1366, 1324, 1240, 1205, 1162, 1122, 1091, 1016, 963, 874, 836, 800, 754, 720, 689, 595, 545.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{24}\text{H}_{27}\text{NNaO}_3\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 432.1604, found: 432.1611.

**52-2**:  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 – 7.52 (m, 2H), 7.38 – 7.31 (m, 1H), 7.23 (t,  $J = 7.9$  Hz, 2H), 7.11 – 7.06 (m, 2H), 6.83 – 6.74 (m, 4H), 6.66 – 6.60 (m, 2H), 4.96 (d,  $J = 8.3$  Hz, 1H), 3.97 (t,  $J = 7.9$  Hz, 1H), 2.57 (q,  $J = 7.6$  Hz, 2H), 1.89 – 1.75 (m, 1H), 1.17 (t,  $J = 7.6$  Hz, 3H), 0.90 (d,  $J = 6.7$  Hz, 3H), 0.67 (d,  $J = 6.7$  Hz, 3H).

#### 4-(1-(phenylsulfonamido)ethyl)phenyl 4-methylpentanoate (**53**)



**53**

A dried 10 mL Schlenk tube was charged with 4-ethylphenyl 4-methylpentanoate (**1ze**, 132.2 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced

pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **53** as a pale yellow oil (35.3 mg, 0.094 mmol, 47% yield, > 20:1 *r.r.*), site-selectivity was determined by <sup>1</sup>H NMR analysis.

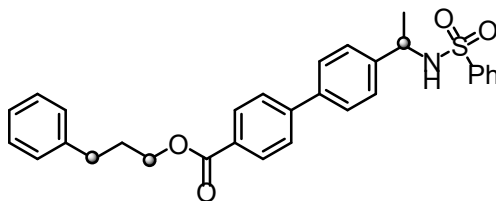
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.71 (d, *J* = 7.8 Hz, 2H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.08 (d, *J* = 8.4 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 5.03 (d, *J* = 7.0 Hz, 1H), 4.53 – 4.48 (m, 1H), 2.59 – 2.46 (m, 2H), 1.70 – 1.60 (m, 4H), 1.41 (d, *J* = 6.9 Hz, 3H), 0.95 (d, *J* = 5.8 Hz, 6H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 172.4, 150.0, 140.6, 139.3, 132.4, 128.9, 127.2, 127.0, 121.6, 53.2, 33.7, 32.5, 27.7, 23.5, 22.2.

IR (film): ν (cm<sup>-1</sup>) 3439, 2957, 1752, 1654, 1508, 1447, 1324, 1205, 1159, 1089, 1017, 719.

HRMS (ESI-TOF, *m/z*) calcd for C<sub>20</sub>H<sub>25</sub>NNaO<sub>4</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 398.1397, found: 398.1407.

### 3-phenylpropyl 4'-(1-(phenylsulfonamido)ethyl)-[1,1'-biphenyl]-4-carboxylate (**54**)



**54**

A dried 10 mL Schlenk tube was charged with 3-phenylpropyl 4'-ethyl-[1,1'-biphenyl]-4-carboxylate (**1zf**, 206.7 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **54** as a white solid (62.0 mg, 0.124 mmol, 62% yield, > 20:1 *r.r.*), site-selectivity was determined by <sup>1</sup>H NMR analysis.

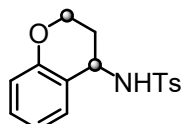
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.64 – 7.59 (m, 2H), 7.47 – 7.39 (m, 1H), 7.31 (t, *J* = 7.8 Hz, 2H), 7.18 – 7.10 (m, 2H), 6.89 – 6.77 (m, 4H), 6.74 – 6.67 (m, 2H), 4.97 (d, *J* = 8.2 Hz, 1H), 4.05 (t, *J* = 7.9 Hz, 1H), 2.64 (q, *J* = 7.6 Hz, 2H), 1.95 – 1.86 (m, 1H), 1.30 – 1.14 (m, 6H), 0.97 (d, *J* = 6.7 Hz, 3H), 0.74 (d, *J* = 6.7 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  166.4, 145.0, 141.9, 141.2, 140.7, 139.2, 132.3, 130.1, 129.2, 128.8, 128.5, 128.5, 127.4, 127.1, 126.9, 126.8, 126.1, 64.4, 53.4, 32.4, 30.3, 23.5.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3629, 1701, 1654, 1605, 1560, 1495, 1445, 1274, 1159, 1090.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{30}\text{H}_{29}\text{NNaO}_4\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 522.1710, found: 522.1711.

#### ***N*-(chroman-4-yl)-4-methylbenzenesulfonamide (55)**



**55**

A dried 10 mL Schlenk tube was charged with chromane (**1zg**, 80.5 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford the amination product **55** as a white solid (24.9 mg, 0.082 mmol, 41% yield, > 20:1 *r.r.*), site-selectivity was determined by  $^1\text{H}$  NMR analysis.

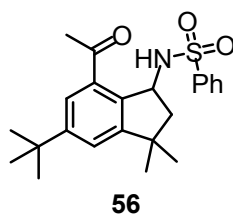
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.03 – 7.89 (m, 2H), 7.71 – 7.63 (m, 1H), 7.63 – 7.55 (m, 2H), 7.13 (m, 1H), 6.74 – 6.79 (m, 2H), 6.65 (dd,  $J = 7.8, 1.7$  Hz, 1H), 4.71 (d,  $J = 6.5$  Hz, 1H), 4.41 (q,  $J = 4.9$  Hz, 1H), 4.29 – 4.04 (m, 2H), 2.18 – 1.96 (m, 2H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  155.0, 140.6, 133.0, 129.8, 129.4, 129.1, 127.1, 120.8, 120.6, 117.4, 62.3, 47.7, 29.9.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3288, 1735, 1654, 1582, 1491, 1448, 1333, 1267, 1224, 1159, 1071, 928, 829, 752, 719, 689, 422.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{16}\text{H}_{17}\text{NNaO}_3\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 326.0821, found: 326.0821.

***N*-(7-acetyl-5-(*tert*-butyl)-3,3-dimethyl-2,3-dihydro-1*H*-inden-1-yl)benzenesulfonamide (56)**



A dried 10 mL Schlenk tube was charged with Celestolide (**1zh**, 73.3 mg, 0.30 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **56** as a white solid (65.5 mg, 0.164 mmol, 82% yield).

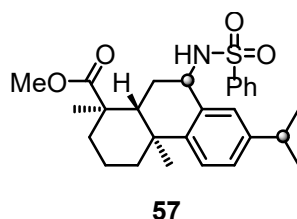
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.91 – 7.86 (m, 2H), 7.51 (d,  $J = 1.8$  Hz, 1H), 7.50 – 7.39 (m, 3H), 7.26 (d,  $J = 1.8$  Hz, 1H), 5.73 (d,  $J = 2.1$  Hz, 1H), 4.41 (dt,  $J = 7.2, 2.3$  Hz, 1H), 2.58 (dd,  $J = 13.7, 2.4$  Hz, 1H), 2.40 (s, 3H), 1.81 (dd,  $J = 13.7, 7.3$  Hz, 1H), 1.25 (s, 3H), 1.23 (s, 9H), 1.12 (s, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  201.7, 155.4, 152.9, 139.4, 137.1, 134.4, 132.5, 128.9, 128.0, 125.7, 124.2, 57.3, 45.1, 43.2, 34.9, 31.3, 30.6, 29.4, 28.2.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3436, 2958, 2101, 1752, 1637, 1447, 1332, 1239, 1164, 1093.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>23</sub>H<sub>29</sub>NNaO<sub>3</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 422.1760, found: 422.1774.

**methyl(1*R*,4*aS*,10*aR*)-7-isopropyl-1,4*a*-dimethyl-9-(phenylsulfonamido)-1,2,3,4,4*a*,9,10,10*a*-octahydrophenanthrene-1-carboxylate (57)**



A dried 10 mL Schlenk tube was charged with Dehydroabietic acid methyl ester (**1zi**, 188.7 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk

tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1, v: v) to afford **57** as a white solid (72.3 mg, 0.154 mmol, 77% yield, 11:1 *d.r.*), site-selectivity was determined by  $^1\text{H}$  NMR analysis.

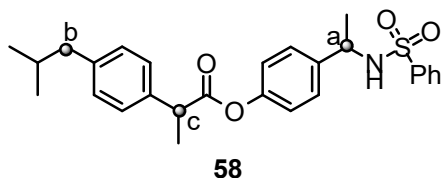
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.00 – 7.95 (m, 2H), 7.66 – 7.59 (m, 1H), 7.59 – 7.53 (m, 2H), 7.13 (d,  $J = 8.2$  Hz, 1H), 7.08 – 7.03 (m, 1H), 6.57 (d,  $J = 2.0$  Hz, 1H), 5.11 (d,  $J = 7.7$  Hz, 1H), 4.43 – 4.39 (m, 1H), 3.74 (s, 3H), 2.73 – 2.64 (m, 1H), 2.34 (d,  $J = 12.7$  Hz, 1H), 2.26 (d,  $J = 13.0$  Hz, 1H), 2.02 – 1.92 (m, 1H), 1.76 – 1.67 (m, 4H), 1.49 (d,  $J = 13.2$  Hz), 1.45 – 1.37 (m, 1H), 1.22 (s, 3H), 1.13 (s, 3H), 1.09 (d,  $J = 4.0$  Hz, 3H), 1.08 (d,  $J = 4.0$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  178.4, 147.0, 146.8, 141.1, 133.3, 132.5, 129.1, 127.6, 127.3, 126.7, 124.5, 52.5, 52.4, 47.2, 40.5, 37.9, 37.2, 36.5, 33.3, 29.5, 24.2, 23.8, 23.7, 18.5, 16.3.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3726, 3284, 2927, 1719, 1654, 1560, 1497, 1446, 1333, 1255, 1166, 1055, 755, 720, 690.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{27}\text{H}_{35}\text{NNaO}_4\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 492.2179, found: 492.2185.

#### 4-(1-(phenylsulfonamido)ethyl)phenyl 2-(4-isobutylphenyl)propanoate (**58**)



A dried 10 mL Schlenk tube was charged with 4-ethylphenyl 2-(4-isobutylphenyl)propanoate (**1zj**, 186.3 mg, 0.60 mmol), benzenesulfonamide (**2b**, 31.4 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **58** as a pale yellow oil (56.8 mg, 0.122 mmol, 61% yield, a:b:c = 6.1:1:0 *r.r.*), site-selectivity was determined by  $^1\text{H}$  NMR analysis.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.72 – 7.65 (m, 2H), 7.49 – 7.43 (m, 1H), 7.40 – 7.34 (m, 2H), 7.30 –



7.27 (m, 2H), 7.14 (d,  $J = 1.8$  Hz, 2H), 7.07 – 7.01 (m, 2H), 6.83 – 6.77 (m, 2H), 5.13 (d,  $J = 7.0$  Hz, 1H), 4.51 – 4.43 (m, 1H), 3.94 – 3.88 (m, 1H), 2.48 (d,  $J = 7.2$  Hz, 2H), 1.92 – 1.82 (m, 1H), 1.59 (d,  $J = 7.2$  Hz, 3H), 1.38 (d,  $J = 6.9$  Hz, 3H), 0.92 (d,  $J = 6.6$  Hz, 6H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  173.0, 150.1, 140.8, 140.5, 139.3, 137.1, 132.4, 129.5, 128.8, 127.1, 127.1, 126.9, 121.4, 53.1, 45.2, 45.0, 30.1, 23.4, 22.4, 18.4.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3284, 2956, 2869, 1756, 1654, 1605, 1560, 1508, 1448, 1378, 1326, 1205, 1163, 1090, 1018, 962, 897, 848, 754, 720, 689.

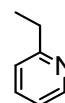
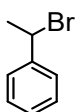
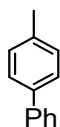
HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{27}\text{H}_{31}\text{NNaO}_4\text{S}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 488.1866, found: 488.1891.

### 4.3 Unsuccessful Substrates

- unreactive substrates



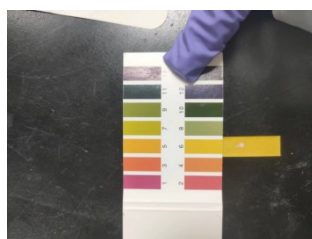
- substrates giving complex mixture



Under the standard condition, tetrahydrofuran and cycloalkanes such as cyclohexane and methylcyclohexane, were found to be unreactive substrates.

Under the standard condition, primary  $\text{C}(\text{sp}^3)\text{-H}$  such as 4-methyl-1,1'-biphenyl, bromine-substituent benzylic substrates such as (1-bromoethyl)benzene, 2-ethylpyridine failed in the reaction to construct the sulfonamidation product and gave a very complex mixture.

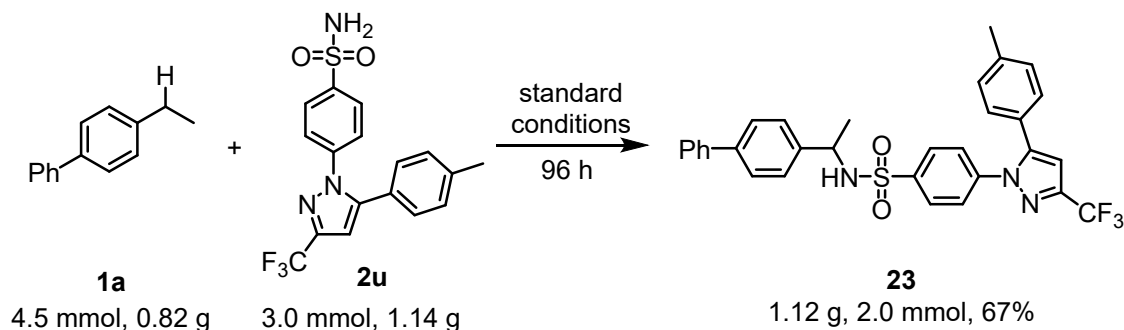
### 4.4 Determination of pH Value after the Reaction



The pH value of the reaction  $1\mathbf{a}+2\mathbf{a}\rightarrow\mathbf{3}$  under standard conditions was determined as  $\sim 6.0$  through pH test strips. This result suggests that the photochemical reaction is relatively neutral.

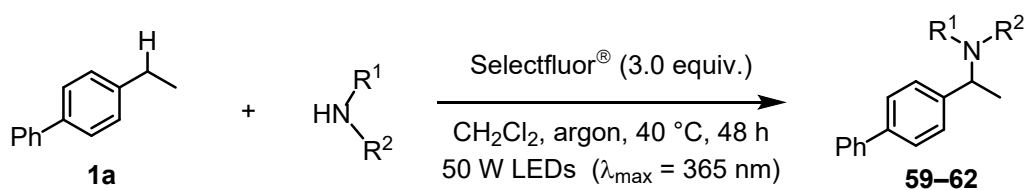
## 5. Synthetic Utility

### 5.1 A Scale-up Reaction

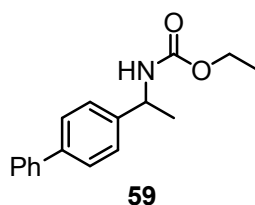


A dried 25 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 0.82 g, 4.5 mmol), Celecoxib (**2u**, 1.14 g, 3.0 mmol), Selectfluor<sup>®</sup> (3.19 g, 9.0 mmol) and dichloromethane (15 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 96 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **23** as a white solid (1.12 g, 2.0 mmol, 67% yield).

### 5.2 Scope of Other *N*-Containing Reagents



#### ethyl (1-([1,1'-biphenyl]-4-yl)ethyl)carbamate (**59**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 72.9 mg, 0.40 mmol), ethyl carbamate (17.8 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL).

The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs lamp ( $\lambda_{\text{max}} = 365 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred at 40 °C for 48 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **59** as a white solid (41.5 mg, 0.0154 mmol, 77% yield).

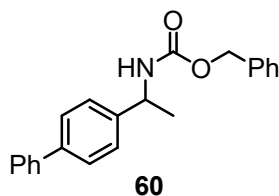
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.62 – 7.54 (m, 4H), 7.44 (t,  $J = 7.7 \text{ Hz}$ , 2H), 7.39 (d,  $J = 7.9 \text{ Hz}$ , 2H), 7.37 – 7.31 (m, 1H), 5.07 – 4.79 (m, 2H), 4.17 – 4.08 (m, 2H), 1.52 (d,  $J = 6.8 \text{ Hz}$ , 3H), 1.24 (t,  $J = 7.1 \text{ Hz}$ , 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  155.9, 142.8, 140.8, 140.3, 128.8, 127.4, 127.3, 127.1, 126.4, 60.8, 50.3, 22.5, 14.6.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3302, 2980, 2929, 1715, 1689, 1542, 1485, 1453, 1406, 1369, 1314, 1253, 1153, 1104, 1066, 1025, 836, 763, 728, 693.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{17}\text{H}_{20}\text{NO}_2^+$  ( $\text{M}+\text{H}$ ) $^+$ : 270.1489, found: 270.1498.

#### benzyl (1-([1,1'-biphenyl]-4-yl)ethyl)carbamate (**60**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 72.9 mg, 0.40 mmol), benzyl carbamate (30.2 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs lamp ( $\lambda_{\text{max}} = 365 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred at 40 °C for 48 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford **60** as a white solid (57.0 mg, 0.172 mmol, 86% yield).

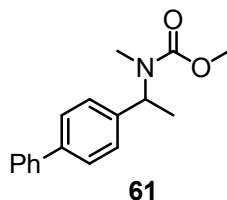
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 (t,  $J = 7.8 \text{ Hz}$ , 4H), 7.45 (t,  $J = 7.6 \text{ Hz}$ , 2H), 7.41 – 7.30 (m, 7H), 5.19 – 5.04 (m, 3H), 4.99 – 4.85 (m, 1H), 1.53 (d,  $J = 6.9 \text{ Hz}$ , 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  155.6, 142.6, 140.8, 140.4, 136.5, 128.8, 128.5, 128.2, 128.1, 127.4, 127.3, 127.1, 126.4, 66.8, 50.6, 22.5.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3330, 3030, 2971, 1696, 1529, 1486, 1453, 1407, 1246, 1058, 1007, 913, 839, 749, 765, 696.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>22</sub>H<sub>22</sub>NO<sub>2</sub><sup>+</sup> (M+H)<sup>+</sup>: 332.1645, found: 332.1642

**methyl (1-([1,1'-biphenyl]-4-yl)ethyl)(methyl)carbamate (61)**



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 72.9 mg, 0.40 mmol), methyl methylcarbamate (17.8 mg, 0.20 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs lamp ( $\lambda_{\text{max}} = 365$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 48 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford the amination product **61** as a colorless oil (30.7 mg, 0.114 mmol, 57% yield).

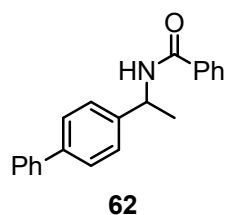
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.61 – 7.55 (m, 4H), 7.44 (t,  $J = 7.7$  Hz, 2H), 7.40 – 7.32 (m, 3H), 5.62 (s, 1H), 3.78 (s, 3H), 2.67 (s, 3H), 1.56 (d,  $J = 7.0$  Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  157.2, 140.7, 140.1, 139.9, 128.7, 127.4, 127.3, 127.1, 127.0, 52.9, 52.7, 28.5, 16.3.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3445, 2954, 1697, 1487, 1452, 1392, 1321, 1190, 1153, 1077, 1007, 842, 766, 738, 697.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>17</sub>H<sub>20</sub>NO<sub>2</sub><sup>+</sup> (M+H)<sup>+</sup>: 270.1489, found: 270.1487.

***N*--(1-([1,1'-biphenyl]-4-yl)ethyl)benzamide (62)**



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 74.9 mg, 0.40 mmol),

benzamide (24.2 mg, 0.2 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs lamp ( $\lambda_{\text{max}} = 365 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred at 40 °C for 48 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford the amination product **62** as a white solid (27.1 mg, 0.090 mmol, 45% yield).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.83 – 7.77 (m, 2H), 7.61 – 7.56 (m, 4H), 7.52 – 7.39 (m, 7H), 7.38 – 7.32 (m, 1H), 6.44 (d,  $J = 7.8 \text{ Hz}$ , 1H), 5.43 – 5.36 (m, 1H), 1.65 (d,  $J = 6.9 \text{ Hz}$ , 3H).

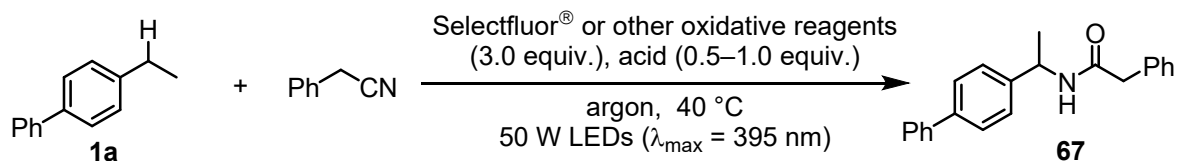
<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  166.6, 142.2, 140.8, 140.5, 134.6, 131.5, 128.8, 128.6, 127.5, 127.3, 127.1, 127.0, 126.7, 49.0, 21.7.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3726, 3316, 1735, 1654, 1632, 1560, 1541, 1105, 420.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>42</sub>H<sub>38</sub>N<sub>2</sub>NaO<sub>2</sub><sup>+</sup> (2M+Na)<sup>+</sup>: 625.2825, found: 625.2830.

### 5.3 Optimization of Conditions for the C(sp<sup>3</sup>)-H Amidation Reaction with Nitriles

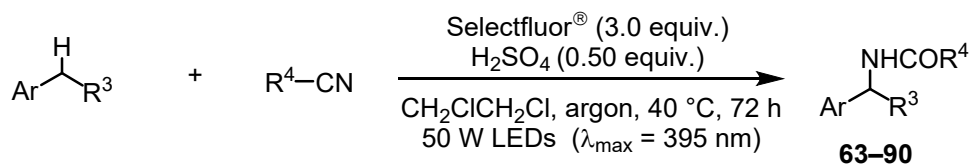
**General procedure.** A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> or other oxidative reagents (0.60 mmol), acid (0.10 mmol or 0.20 mmol) and the indicated solvent (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred at 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **67** as a white solid.

**Table S2.** Optimization of conditions for the C(sp<sup>3</sup>)-H amidation reaction with nitriles <sup>a</sup>

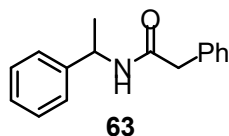
entry	Selectfluor <sup>®</sup> or other oxidative reagents	acid	solvent	time (h)	yield (%)
1	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	CH <sub>2</sub> Cl <sub>2</sub>	72	70
2	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	ClCH <sub>2</sub> CH <sub>2</sub> Cl	72	78
3	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	CHCl <sub>3</sub>	72	48
4	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	Cl <sub>2</sub> CHCHCl <sub>2</sub>	72	64
5	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	THF	72	n.a.
6	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	DMSO	72	n.a.
7	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	THF	72	n.a.
8	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	MeCN	72	trace
9	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	ethyl acetate	72	11
10	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	DMF	72	n.a.
11	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	DMSO	72	n.a.
12	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	acetone	48	12
13	NFSI	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	ClCH <sub>2</sub> CH <sub>2</sub> Cl	48	31
14	TBHP	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	ClCH <sub>2</sub> CH <sub>2</sub> Cl	48	n.a.
15	(NH <sub>4</sub> ) <sub>2</sub> S <sub>2</sub> O <sub>8</sub>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	ClCH <sub>2</sub> CH <sub>2</sub> Cl	48	9
16	PhI(OAc) <sub>2</sub>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	ClCH <sub>2</sub> CH <sub>2</sub> Cl	48	47
17	Selectfluor <sup>®</sup>	TFA (1.0 equiv.)	ClCH <sub>2</sub> CH <sub>2</sub> Cl	48	26
18	Selectfluor <sup>®</sup>	CF <sub>3</sub> SO <sub>2</sub> H (1.0 equiv.)	ClCH <sub>2</sub> CH <sub>2</sub> Cl	48	13
19	Selectfluor <sup>®</sup>	HCl (1.0 equiv.) <sup>c</sup>	ClCH <sub>2</sub> CH <sub>2</sub> Cl	48	trace
20	none	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	ClCH <sub>2</sub> CH <sub>2</sub> Cl	48	trace
21 <sup>d</sup>	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	ClCH <sub>2</sub> CH <sub>2</sub> Cl	48	41
22 <sup>e</sup>	Selectfluor <sup>®</sup>	H <sub>2</sub> SO <sub>4</sub> (0.5 equiv.)	ClCH <sub>2</sub> CH <sub>2</sub> Cl	48	55
23	Selectfluor <sup>®</sup>	none	ClCH <sub>2</sub> CH <sub>2</sub> Cl	48	0

<sup>a</sup> Reaction conditions: **1a** (36.5 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> or other oxidative reagents (3.0 equiv.), acid (0.5 or 1.0 equiv.) and the indicated solvent (1.0 mL), 40 °C, 50 W LEDs ( $\lambda_{\text{max}} = 395 \text{ nm}$ ), under argon. <sup>b</sup> Isolated yield. <sup>c</sup> HCl (2 M in Et<sub>2</sub>O). <sup>d</sup> Selectfluor<sup>®</sup> (2.0 equiv.). <sup>e</sup> Selectfluor<sup>®</sup> (2.5 equiv.). n.a. = not applicable.

## 5.4 Substrate Scope of the C(sp<sup>3</sup>)-H Amidation Reaction with Nitriles



### 2-phenyl-*N*-(1-phenylethyl)acetamide (**63**)



A dried 10 mL Schlenk tube was charged with ethylbenzene (**1b**, 21.2 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs (λ<sub>max</sub> = 395 nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **63** as a white solid (24.9 mg, 0.104 mmol, 40% yield).

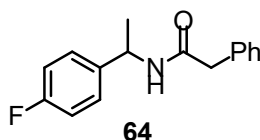
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.26 – 7.29 (m, 2H), 7.24 – 7.13 (m, 6H), 7.11 (d, *J* = 7.1 Hz, 2H), 5.55 (d, *J* = 7.6 Hz, 1H), 5.09 – 5.01 (m, 1H), 3.50 (s, *J* = 1.3 Hz, 2H), 1.32 (d, *J* = 6.9 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 170.0, 143.1, 134.9, 129.4, 129.0, 128.6, 127.4, 127.3, 125.9, 48.7, 43.9, 21.8.

IR (film): ν (cm<sup>-1</sup>) 3727, 3296, 2923, 1735, 1701, 1639, 1560, 1546, 1438, 763, 401.

HRMS (ESI-TOF, *m/z*) calcd for C<sub>16</sub>H<sub>18</sub>NO<sup>+</sup> (*M*+*H*)<sup>+</sup>: 240.1383, found: 240.1385.

### *N*-(1-(4-fluorophenyl)ethyl)-2-phenylacetamide (**64**)



A dried 10 mL Schlenk tube was charged with 1-ethyl-4-fluorobenzene (**1c**, 24.8 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-

thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **64** as a white solid (18.0 mg, 0.070 mmol, 35% yield).

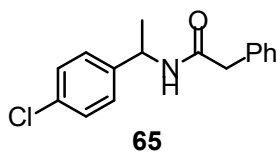
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.38 – 7.32 (m, 2H), 7.32 – 7.27 (m, 1H), 7.26 – 7.21 (m, 2H), 7.18 – 7.12 (m, 2H), 7.00 – 6.93 (m, 2H), 5.59 (d,  $J = 7.1$  Hz, 1H), 5.13 – 5.05 (m, 1H), 3.57 (s, 2H), 1.37 (d,  $J = 7.0$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.0, 161.9 (d,  $J = 147.4$  Hz), 138.9, 138.9, 134.8, 129.2 (d,  $J = 35.3$  Hz), 127.6 (d,  $J = 7.6$  Hz), 127.4, 115.4 (d,  $J = 21.4$  Hz), 48.1, 43.9, 21.8.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3696, 3386, 2949, 2843, 1752, 1735, 1654, 1560, 1512, 1455, 1225, 1113, 1053, 1033, 1017.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{16}\text{H}_{16}\text{FNNaO}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 280.1108, found: 280.1111.

#### ***N*-(1-(4-chlorophenyl)ethyl)-2-phenylacetamide (65)**



A dried 10 mL Schlenk tube was charged with 1-chloro-4-ethylbenzene (**1d**, 28.1 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated  $\text{H}_2\text{SO}_4$  (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **65** as a white solid (25.7 mg, 0.094 mmol, 47% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.30 – 7.33 (m, 2H), 7.29 – 7.24 (m, 1H), 7.23 – 7.18 (m, 4H), 7.09 – 7.06 (m, 2H), 5.62 – 5.48 (m, 1H), 5.07 – 4.99 (m, 1H), 3.53 (s, 2H), 1.33 (d,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.1, 141.7, 134.8, 133.0, 129.3, 129.1, 128.7, 127.4, 127.3, 48.2, 43.8, 21.7.

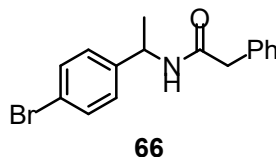
IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3311, 2949, 2843, 1644, 1536, 1492, 1452, 1411, 1247, 1102, 1053, 1033, 1015,



823, 430.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $C_{16}H_{16}ClNNaO^+$  ( $M+Na$ ) $^+$ : 296.0813, found: 296.0815.

#### *N*-(1-(4-bromophenyl)ethyl)-2-phenylacetamide (**66**)



A dried 10 mL Schlenk tube was charged with 1-bromo-4-ethylbenzene (**1e**, 37.0 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max}$  = 395 nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **66** as a white solid (26.7 mg, 0.084 mmol, 42% yield).

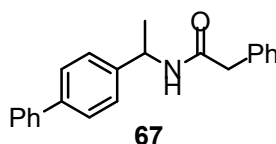
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.43 – 7.38 (m, 2H), 7.38 – 7.33 (m, 2H), 7.32 – 7.27 (m, 1H), 7.26 – 7.22 (m, 2H), 7.07 – 7.03 (m, 2H), 5.58 (d,  $J$  = 7.8 Hz, 1H), 5.09 – 5.02 (m, 1H), 3.57 (s, 2H), 1.36 (d,  $J$  = 7.0 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  170.1, 142.2, 134.8, 131.7, 129.3, 129.1, 127.7, 127.4, 121.1, 48.3, 43.8, 21.7.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3388, 2949, 2837, 2520, 2216, 1645, 1536, 1451, 1411, 1113, 1032, 429.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $C_{16}H_{16}BrNNaO^+$  ( $M+Na$ ) $^+$ : 340.0307, found: 340.0310.

#### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-2-phenylacetamide (**67**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max}$  = 395 nm) with

a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **67** as a white solid (49.2 mg, 0.156 mmol, 78% yield).

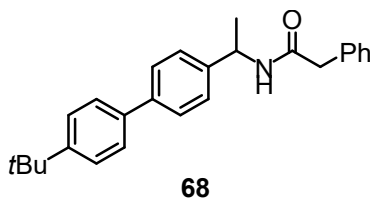
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.55 – 7.51 (m, 2H), 7.51 – 7.47 (m, 2H), 7.42 – 7.37 (m, 2H), 7.35 – 7.29 (m, 3H), 7.29 – 7.26 (m, 1H), 7.26 – 7.21 (m, 4H), 5.75 (d, *J* = 8.1 Hz, 1H), 5.18 – 5.10 (m, 1H), 3.56 (s, 2H), 1.40 (d, *J* = 6.9 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 170.0, 142.1, 140.7, 140.2, 134.9, 129.3, 129.0, 128.7, 127.3, 127.2, 127.0, 126.4, 48.4, 43.8, 21.7.

IR (film): ν (cm<sup>-1</sup>) 3438, 2964, 2108, 1734, 1735, 1638, 1261, 763.

HRMS (ESI-TOF, *m/z*) calcd for C<sub>22</sub>H<sub>21</sub>NNaO<sup>+</sup> (*M*+Na)<sup>+</sup>: 338.1515, found: 338.1517.

#### *N*-(1-(4'-(*tert*-butyl)-[1,1'-biphenyl]-4-yl)ethyl)-2-phenylacetamide (**68**)



A dried 10 mL Schlenk tube was charged with 4-(*tert*-butyl)-4'-ethyl-1,1'-biphenyl (**1m**, 47.7 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}}$  = 395 nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **68** as a white solid (37.9 mg, 0.102 mmol, 51% yield).

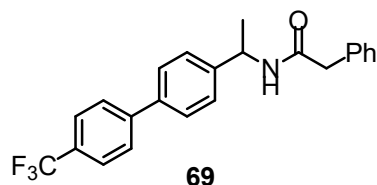
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.49 – 7.43 (m, 4H), 7.40 (d, *J* = 8.5 Hz, 2H), 7.28 – 7.33 (m, 2H), 7.25 – 7.17 (m, 5H), 5.63 (d, *J* = 8.2 Hz, 1H), 5.15 – 5.08 (m, 1H), 3.54 (s, 2H), 1.38 (d, *J* = 6.9 Hz, 3H), 1.31 (s, 9H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 170.1, 150.3, 141.8, 140.1, 137.8, 135.0, 129.4, 129.0, 127.4, 127.2, 126.7, 126.4, 125.7, 48.5, 43.9, 34.5, 31.4, 21.7.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3727, 3409, 2963, 1735, 1640, 1546, 1496, 1452, 1112, 821.

HRMS (ESI-TOF, m/z) calcd for C<sub>26</sub>H<sub>29</sub>NNaO<sup>+</sup> (M+Na)<sup>+</sup>: 394.2141, found: 394.2157.

**2-phenyl-N-(1-(4'-(trifluoromethyl)-[1,1'-biphenyl]-4-yl)ethyl)acetamide (69)**



A dried 10 mL Schlenk tube was charged with 4-ethyl-4'-(trifluoromethyl)-1,1'-biphenyl (50.1 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}}$  = 395 nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **69** as a white solid (46.8 mg, 0.122 mmol, 61% yield).

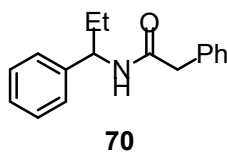
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.66 (q,  $J$  = 8.4 Hz, 4H), 7.54 – 7.50 (m, 2H), 7.40 – 7.34 (m, 2H), 7.33 – 7.27 (m, 5H), 5.65 (d,  $J$  = 7.8 Hz, 1H), 5.20 – 5.13 (m, 1H), 3.61 (s, 2H), 1.43 (d,  $J$  = 7.0 Hz, 3H).

<sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  170.1, 144.2, 143.3, 138.7, 134.9, 129.4, 129.3 (q,  $J$  = 31.7 Hz), 129.1, 127.5, 127.4, 127.3, 126.6, 125.7 (q,  $J$  = 3.0 Hz), 124.2 (q,  $J$  = 271.8 Hz), 48.5, 43.9, 21.8.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3726, 3582, 3305, 1735, 1701, 1638, 1560, 1541, 1495, 1438, 1336, 1163, 1116, 1073, 821, 696.

HRMS (ESI-TOF, m/z) calcd for C<sub>23</sub>H<sub>20</sub>F<sub>3</sub>NNaO<sup>+</sup> (M+Na)<sup>+</sup>: 406.1389, found: 406.1392.

### 2-phenyl-*N*-(1-phenylpropyl)acetamide (**70**)



A dried 10 mL Schlenk tube was charged with propylbenzene (**1q**, 24.0 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **70** as a white solid (20.3 mg, 0.080 mmol, 40% yield).

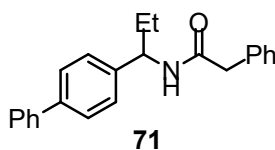
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, TMS signal at 0.0 ppm as a standard)  $\delta$  7.24 – 7.30 (m, 2H), 7.24 – 7.12 (m, 6H), 7.08 – 7.03 (m, 2H), 5.62 (d,  $J = 7.9$  Hz, 1H), 4.80 (q,  $J = 7.6$  Hz, 1H), 3.58 – 3.39 (m, 2H), 1.66 – 1.57 (m, 2H), 0.72 (t,  $J = 7.4$  Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  170.2, 142.1, 135.0, 129.4, 129.0, 128.6, 127.2, 127.4, 126.4, 54.8, 43.9, 29.2, 10.5.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3405, 2951, 2844, 2078, 1644, 1546, 1495, 1454, 1054, 1033, 1017, 699.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>17</sub>H<sub>20</sub>NO<sup>+</sup> (M+H)<sup>+</sup>: 254.1539, found: 254.1543.

### *N*-(1-([1,1'-biphenyl]-4-yl)propyl)-2-phenylacetamide (**71**)



A dried 10 mL Schlenk tube was charged with 4-propyl-1,1'-biphenyl (**1t**, 39.3 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **71** as a white solid (45.5 mg, 0.138 mmol, 69% yield).

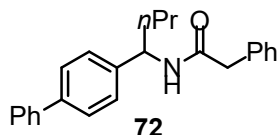
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 – 7.54 (m, 2H), 7.54 – 7.50 (m, 2H), 7.45 – 7.41 (m, 2H), 7.39 – 7.35 (m, 2H), 7.35 – 7.31 (m, 2H), 7.30 – 7.25 (m, 2H), 7.23 – 7.19 (m, 2H), 5.66 (d,  $J = 8.4$  Hz, 1H), 4.93 (q,  $J = 7.6$  Hz, 1H), 3.69 – 3.49 (m, 2H), 1.81 – 1.68 (m, 2H), 0.84 (t,  $J = 7.4$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  141.1, 140.8, 140.2, 135.0, 129.4, 129.1, 128.8, 127.4, 127.3, 127.3, 127.1, 126.8, 54.5, 44.0, 29.2, 10.6.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3424, 2093, 1640, 1542, 1494, 1451, 1264, 761, 694.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{23}\text{H}_{23}\text{NNaO}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 352.1672, found: 352.1676.

### *N*-(1-([1,1'-biphenyl]-4-yl)butyl)-2-phenylacetamide (**72**)



A dried 10 mL Schlenk tube was charged with 4-butyl-1,1'-biphenyl (**1u**, 42.1 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated  $\text{H}_2\text{SO}_4$  (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **72** as a white solid (52.2 mg, 0.152 mmol, 76% yield).

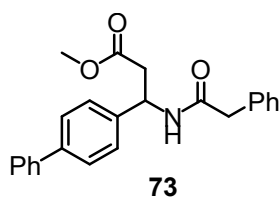
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.55 – 7.50 (m, 2H), 7.50 – 7.46 (m, 2H), 7.39 (t,  $J = 7.7$  Hz, 2H), 7.36 – 7.26 (m, 4H), 7.25 – 7.16 (m, 4H), 5.73 (d,  $J = 8.3$  Hz, 1H), 4.98 (q,  $J = 7.6$  Hz, 1H), 3.65 – 3.41 (m, 2H), 1.62 – 1.70 (m, 2H), 1.35 – 1.08 (m, 3H), 0.86 (t,  $J = 7.4$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.2, 141.5, 140.8, 140.2, 135.0, 129.4, 129.0, 128.8, 127.4, 127.3, 127.3, 127.1, 126.8, 52.9, 43.9, 38.4, 19.4, 13.8.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3427, 2078, 1640, 1546, 1455, 1055, 1033, 1014, 762, 436.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{24}\text{H}_{25}\text{NNaO}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 366.1828, found: 366.1835.

**methyl 3-([1,1'-biphenyl]-4-yl)-3-(2-phenylacetamido)propanoate (73)**



A dried 10 mL Schlenk tube was charged with methyl 3-([1,1'-biphenyl]-4-yl)propanoate (**1v**, 48.1 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **73** as a white solid (35.1 mg, 0.094 mmol, 47% yield).

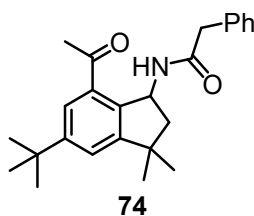
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.56 – 7.52 (m, 2H), 7.52 – 7.49 (m, 2H), 7.44 – 7.28 (m, 8H), 7.24 – 7.20 (m, 2H), 6.52 (d,  $J = 8.6 \text{ Hz}$ , 1H), 5.48 – 5.42 (m, 1H), 3.63 (s, 2H), 3.55 (s, 3H), 2.95 – 2.68 (m, 2H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  171.3, 170.3, 140.6, 140.5, 139.4, 134.8, 129.4, 129.0, 128.8, 127.4, 127.4, 127.0, 126.5, 51.8, 49.3, 43.9, 39.8.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3726, 3284, 2922, 2851, 1735, 1652, 1542, 1486, 1437, 1261, 1166, 765, 731, 696.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>24</sub>H<sub>23</sub>NNaO<sub>3</sub><sup>+</sup> (M+Na)<sup>+</sup>: 396.1570, found: 396.1573.

***N*-(7-acetyl-5-(*tert*-butyl)-3,3-dimethyl-2,3-dihydro-1*H*-inden-1-yl)-2-phenylacetamide (74)**



A dried 10 mL Schlenk tube was charged with Celestolide (**1zh**, 48.9 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395 \text{ nm}$ ) with a DC

fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **74** as a white solid (37.0 mg, 0.098 mmol, 49% yield).

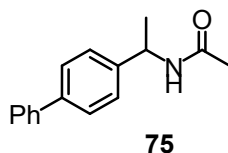
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.53 (d, *J* = 1.8 Hz, 1H), 7.31 (d, *J* = 1.8 Hz, 1H), 7.30 – 7.27 (m, 2H), 7.25 – 7.20 (m, 3H), 5.66 – 5.60 (m, 1H), 5.57 (d, *J* = 7.2 Hz, 1H), 3.51 – 3.60 (m, 2H), 2.47 (s, 3H), 2.41 – 2.34 (m, 1H), 1.92 – 1.87 (m, 1H), 1.33 (s, 9H), 1.23 (d, *J* = 11.3 Hz, 7H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 200.7, 170.3, 154.8, 152.5, 136.8, 135.7, 134.9, 129.5, 128.9, 127.2, 124.8, 123.0, 53.1, 49.3, 43.9, 42.4, 34.9, 31.4, 30.5, 29.7, 28.8.

IR (film): ν (cm<sup>-1</sup>) 3432, 2957, 1640, 1560, 1535, 1495, 1459, 1364, 1234.

HRMS (ESI-TOF, *m/z*) calcd for C<sub>25</sub>H<sub>32</sub>NO<sub>2</sub><sup>+</sup> (M+H)<sup>+</sup>: 378.2428, found: 378.2429.

#### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)acetamide (**75**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), acetonitrile (41.1 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs (λ<sub>max</sub> = 395 nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **75** as a white solid (28.2 mg, 0.118 mmol, 59% yield).

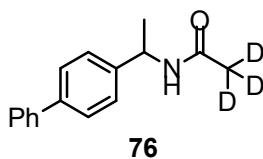
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.57 (d, *J* = 8.4, 4H), 7.45 – 7.41(m, 2H), 7.41 – 7.37 (m, 2H), 7.37 – 7.31 (m, 1H), 5.75 (d, *J* = 7.0 Hz, 1H), 5.22 – 5.14 (m, 1H), 2.01 (s, 3H), 1.53 (d, *J* = 6.9 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 169.1, 142.2, 140.7, 140.4, 128.8, 127.4, 127.3, 127.1, 126.6, 48.6, 23.5, 21.7.

IR (film): ν (cm<sup>-1</sup>) 3424, 1654, 1560, 1438, 1055, 1033, 1014.

HRMS (ESI-TOF, *m/z*) calcd for C<sub>16</sub>H<sub>17</sub>NNaO<sup>+</sup> (M+Na)<sup>+</sup>: 262.1202, found: 262.1204.

***N*-(1-([1,1'-biphenyl]-4-yl)ethyl)acetamide-2,2,2-*d*<sub>3</sub> (76)**



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), acetonitrile-*d*<sub>3</sub> (44.1 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **76** as a white solid (20.8 mg, 0.086 mmol, 43% yield).

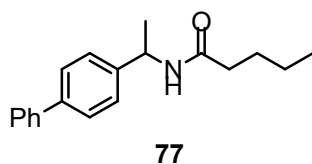
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.60 – 7.54 (m, 4H), 7.43 (t, *J* = 7.7 Hz, 2H), 7.41 – 7.37 (m, 2H), 7.37 – 7.31 (m, 1H), 5.76 (d, *J* = 7.9 Hz, 1H), 5.22 – 5.14 (m, 1H), 1.53 (d, *J* = 6.9 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  169.1, 142.2, 140.7, 140.4, 128.8, 127.4, 127.3, 127.1, 126.6, 48.5, 21.7.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3409, 2950, 2843, 1640, 1546, 1454, 1408, 1113, 1053, 1033, 1018, 422.

HRMS (ESI-TOF, *m/z*) calcd for C<sub>16</sub>H<sub>15</sub>D<sub>3</sub>NO<sup>+</sup> (*M*+*H*)<sup>+</sup>: 243.1571, found: 243.1575.

***N*-(1-([1,1'-biphenyl]-4-yl)ethyl)pentanamide (77)**



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), pentanenitrile (83.1 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **77** as a white solid (35.5 mg, 0.126 mmol, 63% yield).



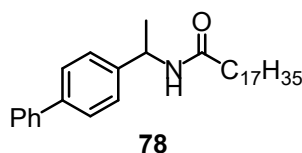
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60 – 7.52 (m, 4H), 7.43 (t,  $J = 7.7$  Hz, 2H), 7.41 – 7.36 (m, 2H), 7.36 – 7.31 (m, 1H), 5.67 (d,  $J = 7.9$  Hz, 1H), 5.23 – 5.15 (m, 1H), 2.28 – 2.14 (m, 2H), 1.67 – 1.62 (m, 2H), 1.53 (d,  $J = 6.9$  Hz, 3H), 1.40 – 1.32 (m, 2H), 0.92 (t,  $J = 7.4$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  172.2, 142.4, 140.8, 140.3, 128.8, 127.4, 127.3, 127.1, 126.6, 48.3, 36.7, 27.8, 22.4, 21.7, 13.8.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3298, 1735, 1685, 1640, 1560, 1546, 1438, 1261, 736.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{19}\text{H}_{23}\text{NNaO}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 304.1672, found: 304.1677.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)stearamide (**78**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), stearonitrile (265.5 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated  $\text{H}_2\text{SO}_4$  (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **78** as a white solid (56.6 mg, 0.122 mmol, 61% yield).

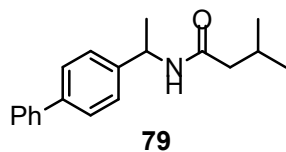
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.62 – 7.52 (m, 4H), 7.46 – 7.41 (m, 2H), 7.41 – 7.37 (m, 2H), 7.37 – 7.32 (m, 1H), 5.73 (d,  $J = 7.9$  Hz, 1H), 5.23 – 5.16 (m, 1H), 2.19 (t,  $J = 7.5$  Hz, 2H), 1.67 – 1.60 (m, 2H), 1.53 (d,  $J = 6.9$  Hz, 3H), 1.39 – 1.18 (m, 28H), 0.88 (t,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  172.2, 142.4, 140.8, 140.3, 128.8, 127.4, 127.3, 127.1, 126.6, 48.3, 37.0, 31.9, 29.7, 29.7, 29.6, 29.5, 29.4, 29.3, 25.8, 22.7, 21.7, 14.1.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3356, 2948, 2834, 2522, 2052, 1654, 1560, 1453, 1413, 1114, 1033.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{32}\text{H}_{49}\text{NNaO}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 486.3706, found: 486.3711.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-3-methylbutanamide (**79**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), 3-methylbutanenitrile (83.1 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **79** as a white solid (37.1 mg, 0.132 mmol, 66% yield).

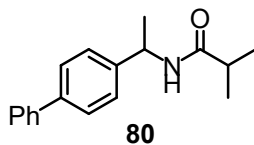
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.60 – 7.52 (m, 4H), 7.46 – 7.41 (m, 2H), 7.41 – 7.37 (m, 2H), 7.37 – 7.31 (m, 1H), 5.69 (d,  $J = 7.9$  Hz, 1H), 5.24 – 5.17 (p,  $J = 7.1$  Hz, 1H), 2.09 – 2.18 (m, 1H), 2.08 – 2.01 (m, 2H), 1.53 (d,  $J = 6.9$  Hz, 3H), 0.96 (dd,  $J = 9.4, 6.5$  Hz, 6H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  171.6, 142.3, 140.8, 140.3, 128.8, 127.4, 127.3, 127.1, 126.6, 48.3, 46.3, 26.2, 22.5, 22.5, 21.7.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3727, 3298, 2957, 1735, 1640, 1541, 1485, 838, 764, 696, 425.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>19</sub>H<sub>23</sub>NNaO<sup>+</sup> (M+Na)<sup>+</sup>: 304.1672, found: 304.1676.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)isobutyramide (**80**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), isobutyronitrile (69.1 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **80** as a white solid (34.2 mg, 0.128 mmol, 64% yield).

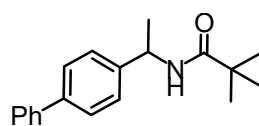
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60 – 7.54 (m, 4H), 7.46 – 7.41 (m, 2H), 7.40 – 7.37 (m, 2H), 7.37 – 7.32 (m, 1H), 5.66 (d,  $J = 7.7$  Hz, 1H), 5.21 – 5.14 (m, 1H), 2.40 – 2.32 (m, 1H), 1.53 (d,  $J = 6.9$  Hz, 3H), 1.19 (d,  $J = 6.9$  Hz, 3H), 1.17 (d,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  176.0, 142.5, 140.8, 140.3, 128.8, 127.4, 127.3, 127.1, 126.6, 48.2, 35.7, 21.7, 19.6, 19.6.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3583, 1735, 1654, 1560, 1438, 422.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{18}\text{H}_{22}\text{NO}^+$  ( $\text{M}+\text{H}$ ) $^+$ : 268.1696, found: 268.1703.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)pivalamide (**81**)



**81**

A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), pivalonitrile (83.1 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated  $\text{H}_2\text{SO}_4$  (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **81** as a white solid (18.6 mg, 0.066 mmol, 33% yield).

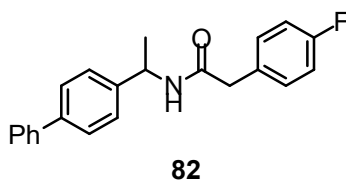
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60 – 7.53 (m, 4H), 7.46 – 7.40 (m, 2H), 7.38 – 7.31 (m, 3H), 5.83 (d,  $J = 7.6$  Hz, 1H), 5.19 – 5.11 (m, 1H), 1.52 (d,  $J = 6.9$  Hz, 3H), 1.22 (s, 9H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  177.5, 142.6, 140.8, 140.2, 128.8, 127.4, 127.3, 127.1, 126.5, 48.2, 38.6, 27.6, 21.7.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3341, 2967, 1735, 1632, 1529, 1484, 1207, 839, 762, 729.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{19}\text{H}_{24}\text{NO}^+$  ( $\text{M}+\text{H}$ ) $^+$  282.1852, found: 282.1861.

***N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-2-(4-fluorophenyl)acetamide (82)**



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), 2-(4-fluorophenyl)acetonitrile (135.1 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **82** as a white solid (50.0 mg, 0.150 mmol, 75% yield).

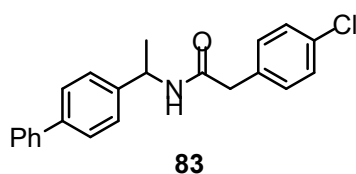
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.60 – 7.50 (m, 4H), 7.43 (dd,  $J = 8.4, 6.9$  Hz, 2H), 7.38 – 7.32 (m, 1H), 7.30 – 7.27 (m, 2H), 7.26 – 7.20 (m, 2H), 7.08 – 7.01 (m, 2H), 5.72 (d,  $J = 8.1$  Hz, 1H), 5.20 – 5.13 (m, 1H), 3.55 (s, 2H), 1.46 (d,  $J = 6.9$  Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  169.8, 162.1 (d,  $J = 252.0$  Hz), 142.0, 140.7, 140.4, 130.9 (d,  $J = 7.6$  Hz), 130.7 (d,  $J = 3.8$  Hz), 128.8, 127.4, 127.3, 127.1, 126.4, 115.9 (d,  $J = 1.3$  Hz), 48.6, 42.9, 21.7.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3696, 3402, 2950, 2844, 1639, 1560, 1546, 1512, 1455, 1054, 1033, 1016, 764, 429.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>22</sub>H<sub>21</sub>FNO<sup>+</sup> (M+H)<sup>+</sup>: 334.1602, found: 334.1606.

***N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-2-(4-chlorophenyl)acetamide (83)**



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), 2-(4-chlorophenyl)acetonitrile (151.6 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then

concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **83** as a white solid (49.7 mg, 0.142 mmol, 71% yield).

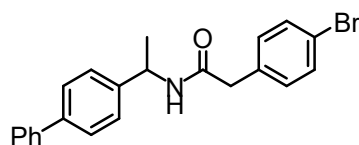
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 – 7.51 (m, 4H), 7.46 – 7.41 (m, 2H), 7.37 – 7.27 (m, 5H), 7.23 – 7.16 (m, 2H), 5.69 (d,  $J$  = 8.0 Hz, 1H), 5.20 – 5.12 (m, 1H), 3.54 (s, 2H), 1.46 (d,  $J$  = 6.9 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  169.4, 141.9, 140.7, 140.4, 133.4, 133.3, 130.7, 129.1, 128.8, 127.4, 127.4, 127.1, 126.5, 48.7, 43.1, 21.7.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3853, 3696, 3681, 3372, 2949, 2844, 1735, 1685, 1654, 1560, 1455, 1054, 1033, 1016, 433.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{22}\text{H}_{21}\text{ClNO}^+$  ( $\text{M}+\text{H}$ ) $^+$ : 350.1306, found: 350.1308.

#### ***N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-2-(4-bromophenyl)acetamide (84)**



**84**

A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), 2-(4-bromophenyl)acetonitrile (196.0 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated  $\text{H}_2\text{SO}_4$  (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}}$  = 395 nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **84** as a white solid (43.4 mg, 0.110 mmol, 55% yield).

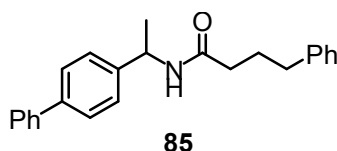
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 – 7.52 (m, 4H), 7.50 – 7.45 (m, 2H), 7.46 – 7.40 (m, 2H), 7.38 – 7.32 (m, 1H), 7.31 – 7.27 (m, 2H), 7.19 – 7.10 (m, 2H), 5.71 (d,  $J$  = 8.0 Hz, 1H), 5.20 – 5.12 (m, 1H), 3.52 (s, 2H), 1.46 (d,  $J$  = 6.9 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  169.3, 141.9, 140.7, 140.4, 133.9, 132.1, 131.0, 128.8, 127.4, 127.4, 127.1, 126.5, 121.4, 48.7, 43.2, 21.7.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3377, 2950, 2838, 2522, 2212, 1644, 1536, 1451, 1413, 1113, 1032, 433.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>22</sub>H<sub>20</sub>BrNNaO<sup>+</sup> (M+Na)<sup>+</sup>: 416.0620, found: 416.0624.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-4-phenylbutanamide (**85**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), 4-phenylbutanenitrile (145.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **85** as a white solid (45.3 mg, 0.132 mmol, 66% yield).

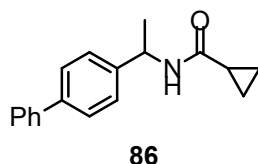
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.59 – 7.54 (m, 4H), 7.46 – 7.40 (m, 2H), 7.40 – 7.32 (m, 3H), 7.29 – 7.26 (m, 2H), 7.21 – 7.14 (m, 3H), 5.63 (d,  $J = 8.0$  Hz, 1H), 5.23 – 5.15 (m, 1H), 2.66 (t,  $J = 7.5$  Hz, 2H), 2.25 – 2.11 (m, 2H), 2.03 – 1.98 (m, 2H), 1.52 (d,  $J = 6.9$  Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  171.7, 142.3, 141.5, 140.8, 140.4, 128.8, 128.5, 128.4, 127.4, 127.3, 127.1, 126.6, 126.0, 48.4, 36.0, 35.2, 27.1, 21.7.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3727, 3285, 1735, 1640, 1560, 1546, 1484, 697.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>24</sub>H<sub>26</sub>NO<sup>+</sup> (M+H)<sup>+</sup>: 344.2009, found: 344.2013.

### *N*-(1-([1,1'-biphenyl]-4-yl)ethyl)cyclopropanecarboxamide (**86**)



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), cyclopropanecarbonitrile (67.1 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three

freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **86** as a white solid (35.6 mg, 0.134 mmol, 67% yield).

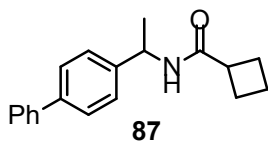
$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60 – 7.54 (m, 4H), 7.48 – 7.38 (m, 4H), 7.37 – 7.31 (m, 1H), 5.93 (d,  $J = 7.9 \text{ Hz}$ , 1H), 5.23 – 5.16 (m, 1H), 1.54 (d,  $J = 6.9 \text{ Hz}$ , 3H), 1.38 – 1.32 (m, 1H), 1.09 – 0.92 (m, 2H), 0.84 – 0.68 (m, 2H).

$^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  172.6, 142.5, 140.8, 140.3, 128.8, 127.4, 127.3, 127.1, 126.6, 48.6, 21.8, 14.9, 7.2.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 2950, 2842, 2522, 2178, 1652, 1455, 1404, 1112, 1033, 1014, 479, 471, 444, 430.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{18}\text{H}_{20}\text{NO}^+$  ( $\text{M}+\text{H}$ ) $^+$ : 266.1539, found: 266.1544.

#### ***N*-(1-([1,1'-biphenyl]-4-yl)ethyl)cyclobutanecarboxamide (87)**



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), cyclobutanecarbonitrile (81.1 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated  $\text{H}_2\text{SO}_4$  (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **87** as a white solid (36.9 mg, 0.132 mmol, 66% yield).

$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60 – 7.54 (m, 4H), 7.43 (t,  $J = 7.7 \text{ Hz}$ , 2H), 7.40 – 7.36 (m, 2H), 7.36 – 7.32 (m, 1H), 5.61 (d,  $J = 8.0 \text{ Hz}$ , 1H), 5.22 – 5.14 (m, 1H), 3.05 – 2.96 (m, 1H), 2.36 – 2.24 (m, 2H), 2.20 – 2.10 (m, 2H), 2.01 – 1.91 (m, 1H), 1.91 – 1.83 (m, 1H), 1.52 (d,  $J = 6.9 \text{ Hz}$ , 3H).

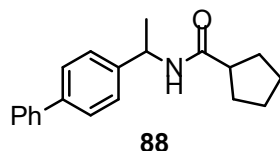
$^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  174.0, 142.5, 140.8, 140.3, 128.8, 127.4, 127.3, 127.1, 126.6, 48.2,

40.0, 25.3, 25.3, 21.8, 18.2.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3293, 2110, 1639, 1541, 1449, 1389, 1255, 1015, 840, 763, 729, 693, 433.

HRMS (ESI-TOF, m/z) calcd for C<sub>19</sub>H<sub>21</sub>NNaO<sup>+</sup> (M+Na)<sup>+</sup>: 302.1515, found: 302.1517.

***N*-(1-([1,1'-biphenyl]-4-yl)ethyl)cyclopentanecarboxamide (88)**



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), cyclopentanecarbonitrile (95.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}}$  = 395 nm) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **88** as a white solid (40.5 mg, 0.138 mmol, 69% yield).

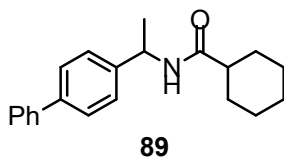
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.60 – 7.53 (m, 4H), 7.45 – 7.40 (m, 2H), 7.40 – 7.37 (m, 2H), 7.37 – 7.31 (m, 1H), 5.69 (d, *J* = 7.8 Hz, 1H), 5.22 – 5.14 (m, 1H), 2.52 (q, *J* = 8.1 Hz, 1H), 1.93 – 1.68 (m, 6H), 1.62 – 1.54 (m, 2H), 1.52 (d, *J* = 6.9 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  175.3, 142.5, 140.8, 140.3, 128.8, 127.4, 127.3, 127.1, 126.6, 48.3, 45.9, 30.4, 30.4, 25.9, 21.8.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3727, 3293, 2963, 2868, 1735, 1638, 1541, 1485, 1448, 1389, 1235., 1114, 840, 763, 728, 694.

HRMS (ESI-TOF, m/z) calcd for C<sub>20</sub>H<sub>23</sub>NNaO<sup>+</sup> (M+Na)<sup>+</sup>: 316.1672, found: 316.1674.

***N*-(1-([1,1'-biphenyl]-4-yl)ethyl)cyclohexanecarboxamide (89)**



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol),



cyclohexanecarbonitrile (109.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **89** as a white solid (39.4 mg, 0.128 mmol, 64% yield).

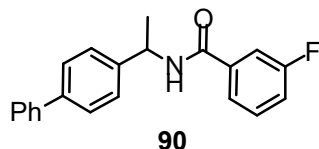
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.59 – 7.53 (m, 4H), 7.43 (t,  $J = 7.7 \text{ Hz}$ , 2H), 7.40 – 7.31 (m, 3H), 5.67 (d,  $J = 7.9 \text{ Hz}$ , 1H), 5.21 – 5.14 (m, 1H), 2.09 (tt,  $J = 11.8, 3.5 \text{ Hz}$ , 1H), 1.92 – 1.83 (m, 2H), 1.83 – 1.76 (m, 2H), 1.70 – 1.64 (m, 1H), 1.52 (d,  $J = 6.9 \text{ Hz}$ , 3H), 1.46 (td,  $J = 12.3, 3.5 \text{ Hz}$ , 2H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  175.51, 142.5, 140.8, 140.3, 128.8, 127.4, 127.3, 127.1, 126.5, 48.0, 45.6, 29.7, 29.7, 25.7, 25.7, 21.7.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3449, 1735, 1637, 1560, 1546.

HRMS (ESI-TOF,  $m/z$ ) calcd for C<sub>21</sub>H<sub>25</sub>NNaO<sup>+</sup> (M+Na)<sup>+</sup>: 330.1828, found: 330.1841.

#### ***N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-3-fluorobenzamide (90)**



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), 3-fluorobenzonitrile (121.1 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol), concentrated H<sub>2</sub>SO<sub>4</sub> (9.8 mg, 0.10 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred 40 °C for 72 h, then concentrated to dryness under reduced pressure. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 3:1) to afford **90** as a white solid (19.8 mg, 0.062 mmol, 31% yield).

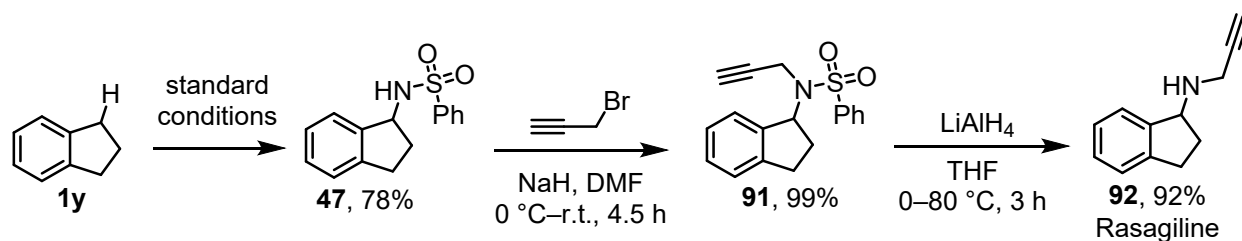
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.42 – 7.37 (m, 4H), 7.36 – 7.30 (m, 2H), 7.29 – 7.18 (m, 5H), 7.18 – 7.12 (m, 1H), 7.03 – 6.98 (m, 1H), 6.14 (d,  $J = 7.7 \text{ Hz}$ , 1H), 5.22 – 5.14 (m, 1H), 1.46 (d,  $J = 6.9 \text{ Hz}$ , 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  165.3 (d,  $J = 2.5$  Hz), 163.8, 161.8, 141.9, 140.6 (d,  $J = 11.3$  Hz), 136.8 (d,  $J = 7.6$  Hz), 130.2 (d,  $J = 7.6$  Hz), 128.8, 127.5, 127.3, 127.1, 126.7, 122.4 (d,  $J = 2.5$  Hz), 118.5 (d,  $J = 20.2$  Hz), 114.4 (d,  $J = 22.7$  Hz), 49.2, 21.6.

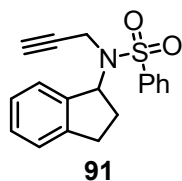
IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3423, 2982, 1735, 1654, 1560, 1483, 1055, 1033, 1014.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{21}\text{H}_{18}\text{FNNaO}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 342.1265, found: 342.1268.

## 5.5 Synthesis of Rasagiline



### *N*-(2,3-dihydro-1H-inden-1-yl)-*N*-(prop-2-yn-1-yl)benzenesulfonamide (**91**)



A dried 10 mL Schlenk tube was charged with *N*-(2,3-dihydro-1H-inden-1-yl)benzenesulfonamide (**47**, 136.7 mg, 0.50 mmol) and DMF (2.0 mL), then cooled down to 0 °C. NaH (24.0 mg, 1.0 mmol) was added, and the resultant mixture was stirred at 0 °C for 30 min. Propargyl bromide (89.2 mg, 0.75 mmol, 80% purity) was added dropwise. The reaction was warmed up to room temperature and stirred for additional 4 h, then quenched with  $\text{H}_2\text{O}$  (10 mL) and extracted with ethyl acetate (10 mL  $\times$  3). The combined organic layers were dried over  $\text{Na}_2\text{SO}_4$  and concentrated under vacuum. The residue was subjected to flash chromatography on silica gel (eluted with PE to PE: EA = 5:1) to afford the **91** as a yellow solid (154.1 mg, 0.495 mmol, 99% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.02 (d,  $J = 7.8$ , 2H), 7.64 – 7.58 (m, 1H), 7.57 – 7.51 (m, 2H), 7.25 – 7.20 (m, 2H), 7.18 – 7.14 (m, 1H), 7.05 (d,  $J = 7.6$  Hz, 1H), 5.55 (t,  $J = 7.7$  Hz, 1H), 4.25 – 4.17 (m, 1H), 3.60 – 3.50 (m, 1H), 2.98 – 3.04 (m, 1H), 2.83 – 2.74 (m, 1H), 2.29 – 2.15 (m, 2H), 2.06 (t,  $J = 2.5$  Hz, 1H).

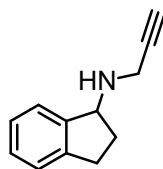
$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  143.7, 140.8, 139.5, 132.7, 128.9, 128.5, 127.6, 126.9, 125.0, 124.7,

79.9, 72.1, 63.5, 32.6, 30.2, 29.4.

IR (film):  $\nu$  (cm<sup>-1</sup>) 3439, 2924, 2121, 1640, 1447, 1322, 1157, 1094.

HRMS (ESI-TOF, m/z) calcd for C<sub>18</sub>H<sub>17</sub>NNaO<sub>2</sub>S<sup>+</sup> (M+Na)<sup>+</sup>: 344.0872, found: 344.0883.

***N*-(prop-2-yn-1-yl)-2,3-dihydro-1*H*-inden-1-amine (92)**



**92**

A dried 5.0 mL Schlenk tube was charged with LiAlH<sub>4</sub> (2.5 M in THF, 0.50 mmol) in anhydrous tetrahydrofuran (1.0 mL), and the mixture was cooled down to 0 °C. A solution of **91** (31.1 mg, 0.10 mmol) in anhydrous tetrahydrofuran (1.0 mL) was added dropwise over 10 minutes under Ar. The mixture was heated at reflux for 3 h, then cooled to 0 °C. An aqueous solution of HCl (36% in H<sub>2</sub>O) was added until no H<sub>2</sub> evolution. Added H<sub>2</sub>O (5 mL) to the mixture and extracted with CH<sub>2</sub>Cl<sub>2</sub> (5 mL × 3). After filtration over Na<sub>2</sub>SO<sub>4</sub> and evaporation of the solvent under reduced pressure, Rasagiline **92** was obtained as a yellow oil (15.8 mg, 0.092 mmol, 92% yield).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.33 – 7.35 (m, 1H), 7.26 – 7.16 (m, 3H), 4.41 (t, *J* = 6.9, 5.2 Hz, 1H), 3.60 – 3.37 (m, 2H), 3.01 – 3.07 (m, 1H), 2.79 – 2.85 (m, 1H), 2.45 – 2.32 (m, 1H), 2.25 (t, *J* = 2.4 Hz, 1H), 1.93 – 1.78 (m, 1H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  144.5, 143.8, 127.6, 126.3, 124.9, 124.2, 82.5, 71.4, 61.9, 36.2, 33.3, 30.5.

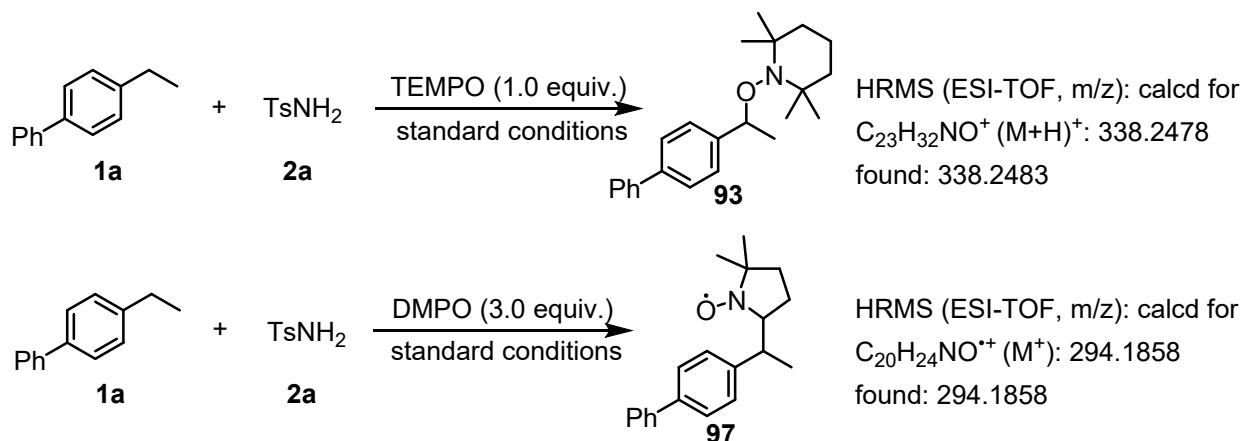
IR (film):  $\nu$  (cm<sup>-1</sup>) 3726, 3405, 2964, 1735, 1685, 1654, 1617, 1560, 1027.

HRMS (ESI-TOF, m/z) calcd for C<sub>12</sub>H<sub>14</sub>N<sup>+</sup> (M+H)<sup>+</sup>: 172.1121, found: 172.1124.

## 6. Mechanistic Investigations

### 6.1 Radical Trapping Experiments

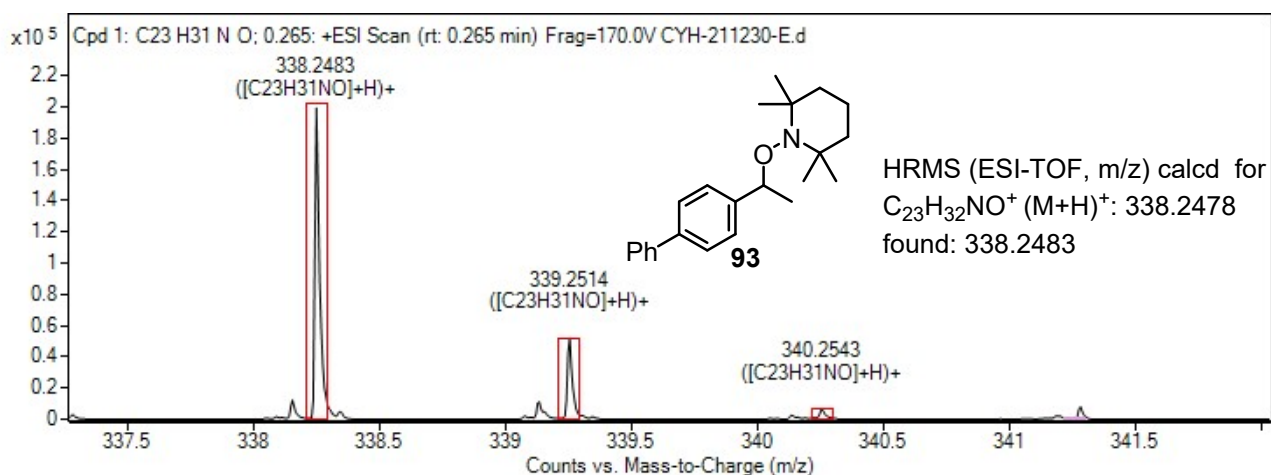
#### 6.1.1 Trapping of C-centered Radicals in the Photochemical C(sp<sup>3</sup>)-H Sulfonylamidation Reaction



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *p*-toluenesulfonamide (**2a**, 34.2 mg, 0.20 mmol), radical scavenger 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (31.2 mg, 0.20 mmol) or 5,5-dimethyl-1-pyrroline *N*-oxide (DMPO) (67.9 mg, 0.60 mmol). The mixtures in dichloromethane (1.0 mL) was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 12 h. The reaction mixture was analyzed by HRMS.

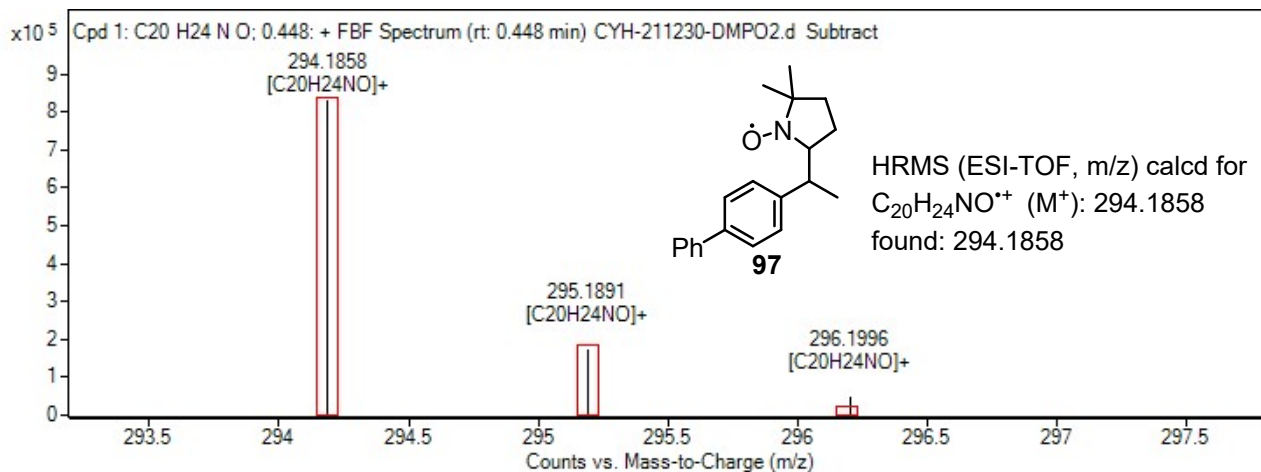
#### Results:

Compound **93**, HRMS (ESI-TOF, m/z) calcd for C<sub>23</sub>H<sub>32</sub>NO<sup>+</sup> (M+H)<sup>+</sup>: 338.2478, found: 338.2483.



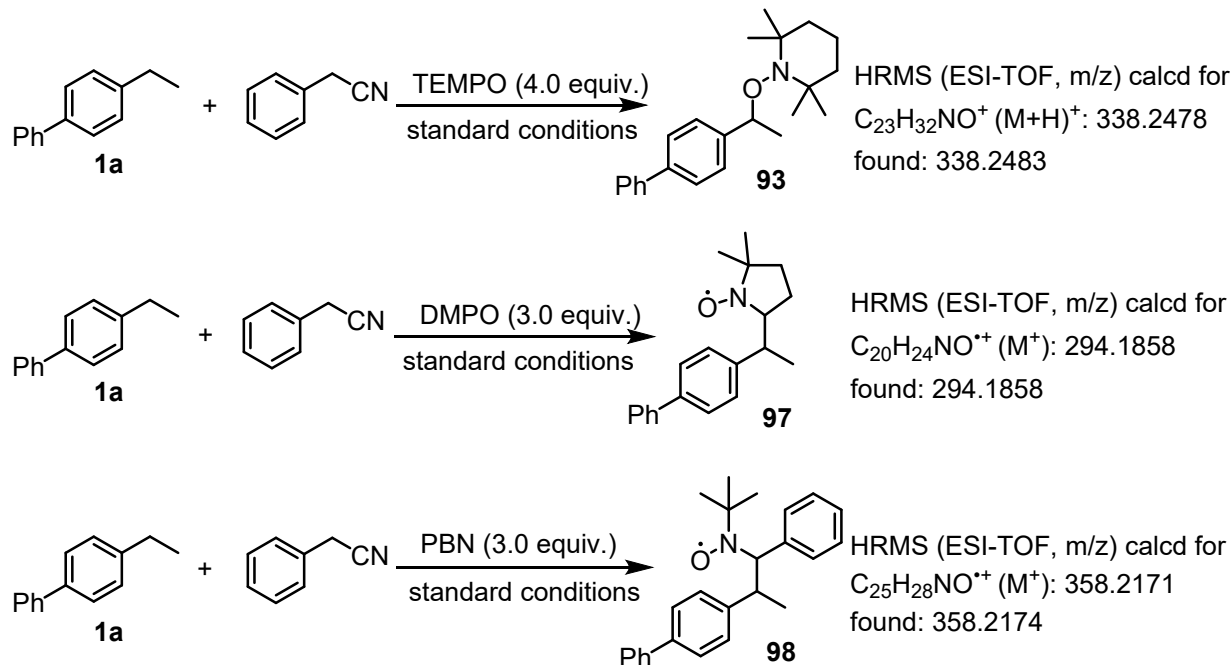
**Figure S2.** HRMS data of compound **93**.

Compound **97**, HRMS (ESI-TOF,  $m/z$ ) calcd for  $C_{20}H_{24}NO^+$  ( $M^+$ ): 294.1858, found: 294.1858.



**Figure S3.** HRMS data of compound **97**.

### 6.1.2 Trapping of C-centered Radicals in the Photochemical $C(sp^3)$ -H Amidation Reaction with Nitriles



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), concentrated  $H_2SO_4$  (9.8 mg, 0.10 mmol), radical scavenger 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (125.0 mg, 0.80 mmol), 5,5-dimethyl-1-pyrroline *N*-oxide

(DMPO) (67.9 mg, 0.60 mmol) or *N*-Benzylidene-*tert*-butylamine *N*-Oxide (PBN) (106.3 mg, 0.60 mmol). The mixtures in 1,2-dichloroethane (1.0 mL) was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395 \text{ nm}$ ) with a DC fan cooling. The reaction was stirred at 40 °C for 8 h. The reaction mixture was analyzed by HRMS.

## Results:

Compound **93**, HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{23}\text{H}_{32}\text{NO}^+$  ( $\text{M}+\text{H}$ ) $^+$ : 338.2478, found: 262.2483.

Compound **97**, HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{20}\text{H}_{24}\text{NO}^+$  ( $\text{M}^+$ ): 294.1858, found: 294.1858.

Compound **98**, HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{25}\text{H}_{28}\text{NO}^+$  ( $\text{M}^+$ ): 358.2171, found: 358.2174.

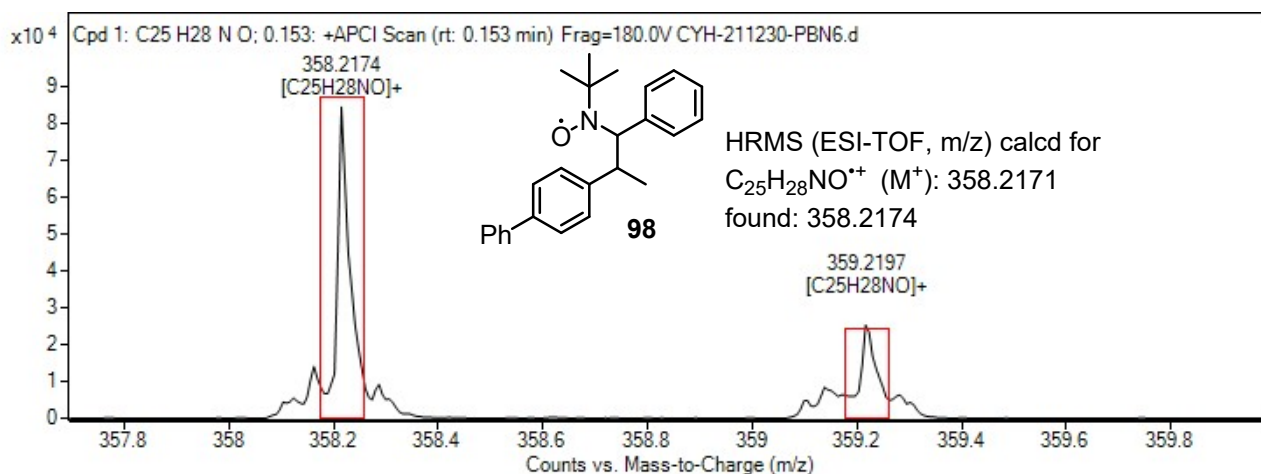
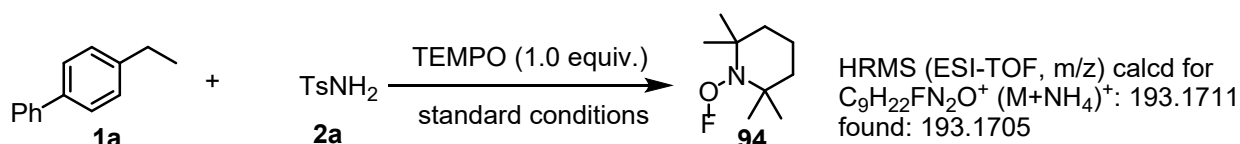


Figure S4. HRMS data of compound **98**.

### 6.1.3 Trapping of Fluorine Radicals in the Photochemical $\text{C}(\text{sp}^3)\text{-H}$ Sulfonylamidation

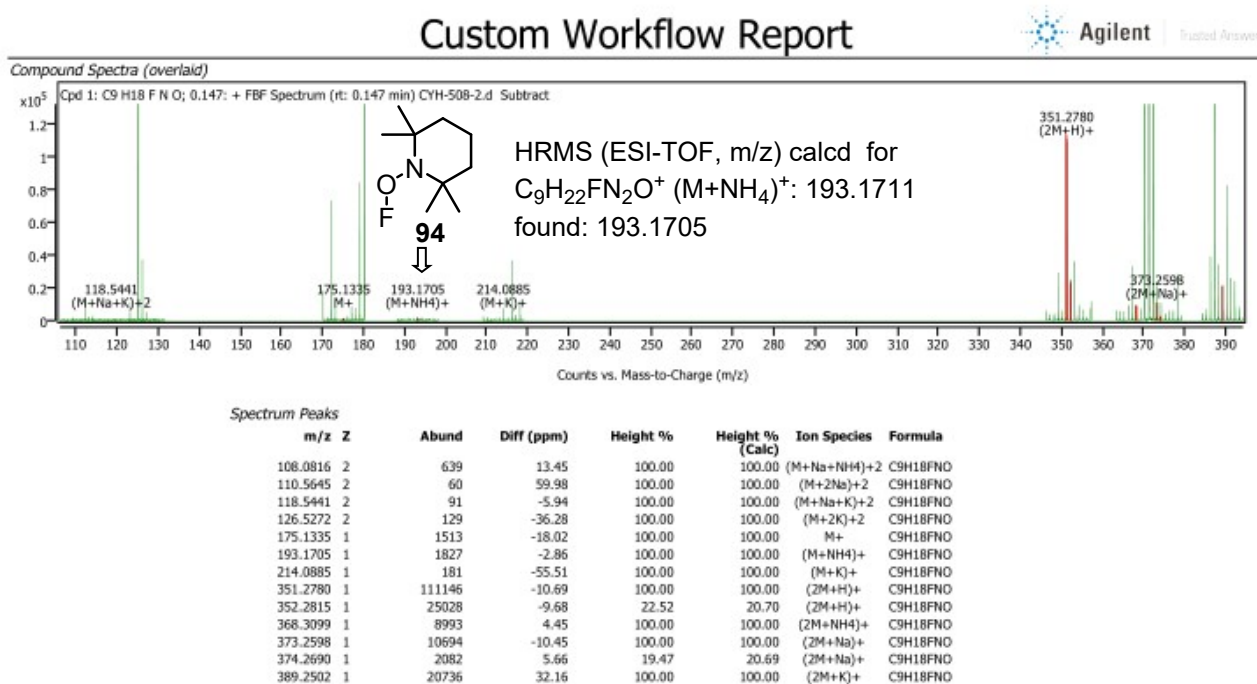


A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *p*-toluenesulfonamide (**2a**, 0.20 mmol), radical scavenger 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (31.2 mg, 0.20 mmol) and dichloromethane (1.0 mL). The mixtures was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W

LEDs ( $\lambda_{\max} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 12 h. The reaction mixture was analyzed by HRMS.

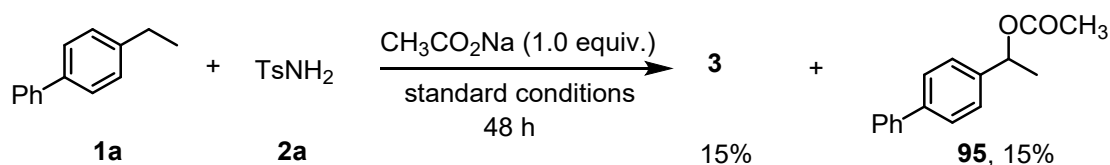
## Results:

Compound **94**, HRMS (ESI-TOF,  $m/z$ ) calcd for  $C_9H_{22}FN_2O^+$  ( $M+NH_4$ ) $^+$ : 193.1711, found: 193.1705.



**Figure S5.** HRMS data of compound **94**.

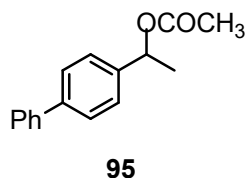
## 6.2 Probing of Carbon Cations



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 54.7 mg, 0.30 mmol), *p*-toluenesulfonamide (**2a**, 0.20 mmol), sodium acetate (16.4 mg, 0.20 mmol), Selectfluor® (212.6 mg, 0.60 mmol) and dichloromethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\max} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 48 h, then concentrated to dryness

under reduced pressure. The residue was subjected to flash chromatography on silica gel to afford **3** (10.5 mg, 0.030 mmol, 15% yield) as a white solid and **95** (7.2 mg, 0.030 mmol, 15% yield) as a white solid.

### 1-([1,1'-biphenyl]-4-yl)ethyl acetate (**95**)



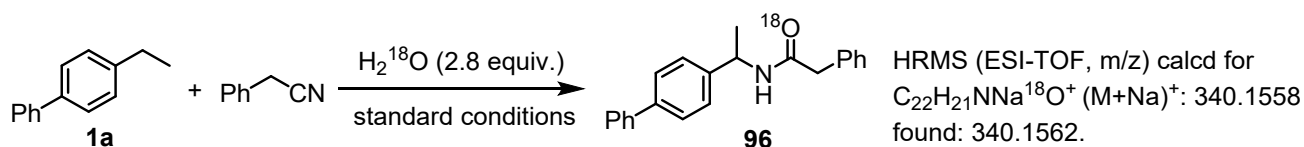
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.62 – 7.56 (m, 4H), 7.44 (t,  $J = 7.4$  Hz, 4H), 7.39 – 7.31 (m, 1H), 5.94 (q,  $J = 6.6$  Hz, 1H), 2.10 (s, 3H), 1.58 (d,  $J = 6.6$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 140.9, 140.8, 140.7, 128.8, 127.3, 127.3, 127.1, 126.6, 72.1, 22.1, 21.4.

IR (film):  $\nu$  ( $\text{cm}^{-1}$ ) 3727, 3583, 1735, 1654, 1560, 1240.

HRMS (ESI-TOF,  $m/z$ ) calcd for  $\text{C}_{16}\text{H}_{16}\text{NaO}_2^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 263.1043, found: 263.1044.

### 6.3 An Isotope Labeling Experiment



A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 36.5 mg, 0.20 mmol), benzyl cyanide (117.2 mg, 1.0 mmol), Selectfluor<sup>®</sup> (212.6 mg, 0.60 mmol),  $\text{H}_2\text{SO}_4$  (9.8 mg, 0.10 mmol),  $\text{H}_2^{18}\text{O}$  (11.2 mg, 0.56 mmol) and 1,2-dichloroethane (1.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at 40 °C for 48 h. The reaction product **96** was detected by HRMS (ESI-TOF,  $m/z$ ). Calcd for  $\text{C}_{22}\text{H}_{21}\text{NNa}^{18}\text{O}^+$  ( $\text{M}+\text{Na}$ ) $^+$ : 340.1558, found: 340.1562.



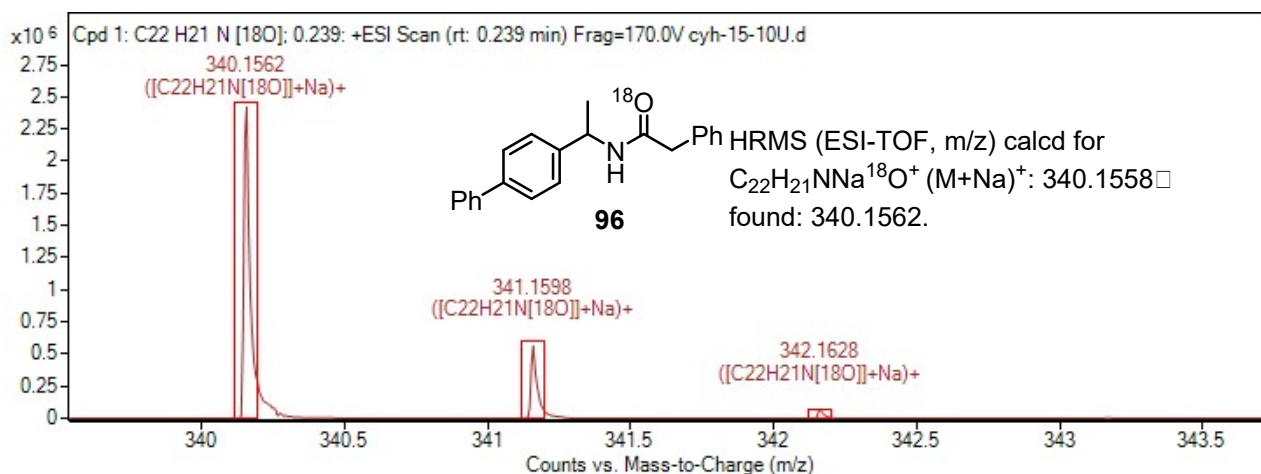
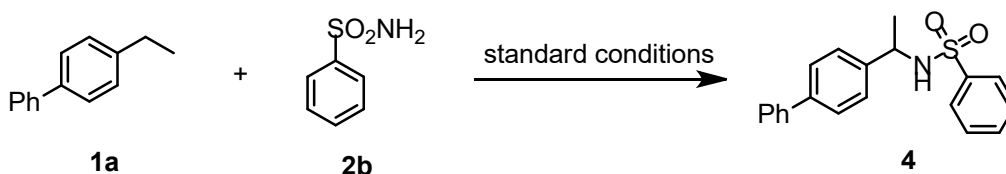
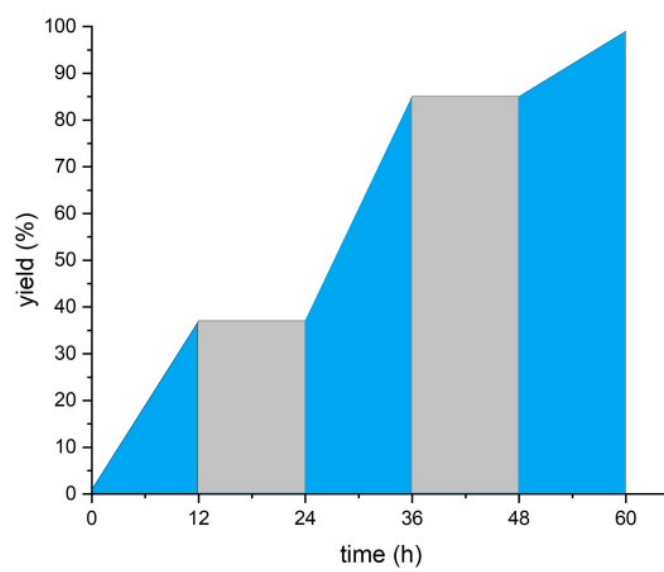


Figure S6. HRMS data of compound **96**.

#### 6.4 Light on/off Experiments

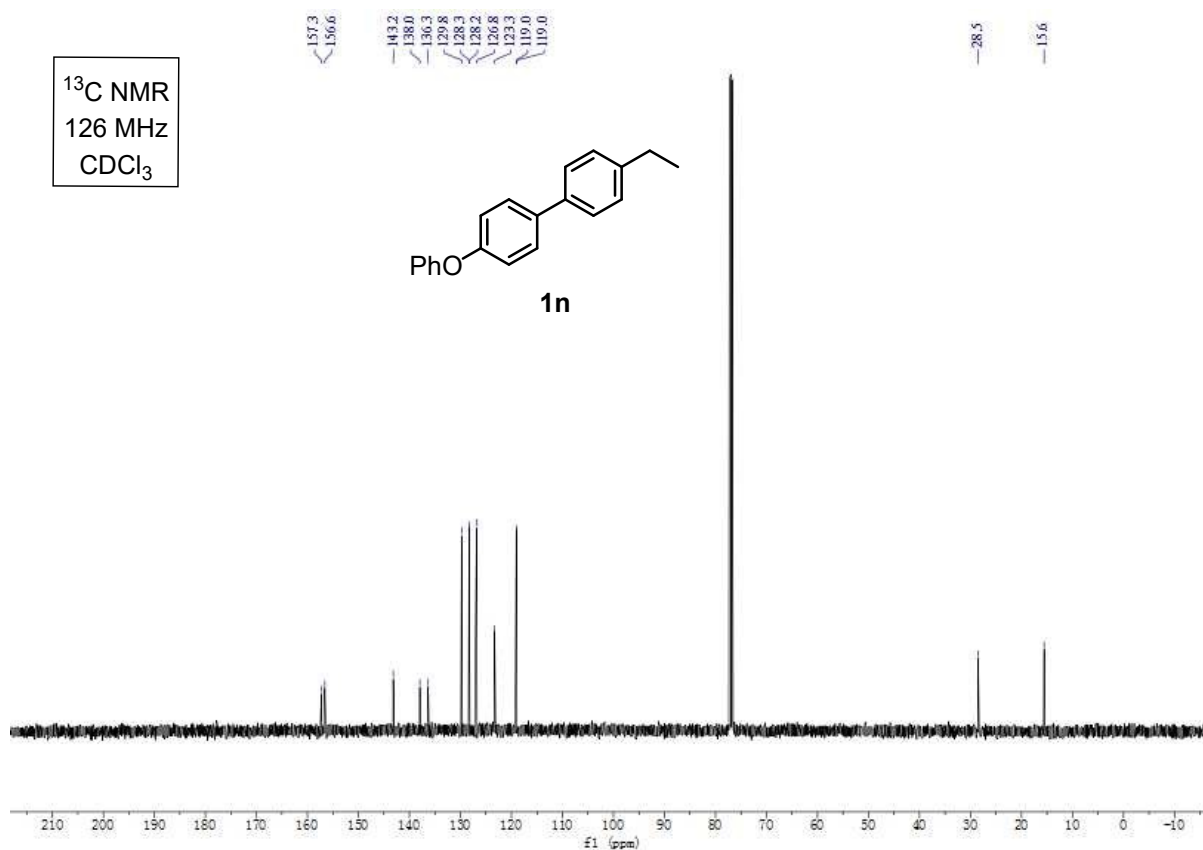
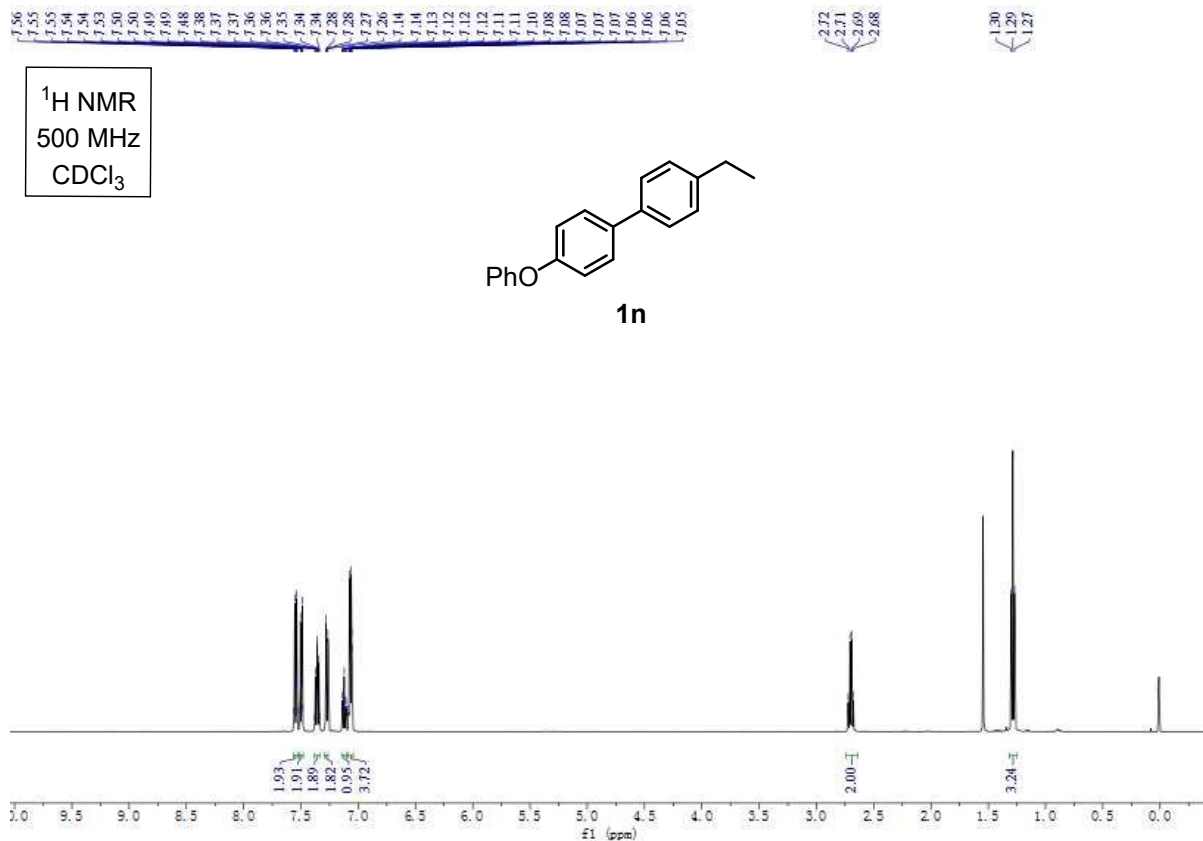


A dried 10 mL Schlenk tube was charged with 4-ethyl-1,1'-biphenyl (**1a**, 109.4 mg, 0.60 mmol), benzenesulfonamide (**2b**, 62.9 mg, 0.40 mmol), Selectfluor<sup>®</sup> (425.1 mg, 1.2 mmol) and dichloromethane (2.0 mL). The mixture was degassed *via* three freeze-pump-thaw cycles. The Schlenk tube was positioned approximately 3 cm away from a 50 W LEDs ( $\lambda_{\text{max}} = 395$  nm) with a DC fan cooling. The reaction was stirred at room temperature for the indicated time. The solvent was evaporated, and the yield analyzed by *in-situ* <sup>1</sup>H NMR using 1,1,2,2-tetrachloroethane as an internal standard. The chain reaction process was excluded.

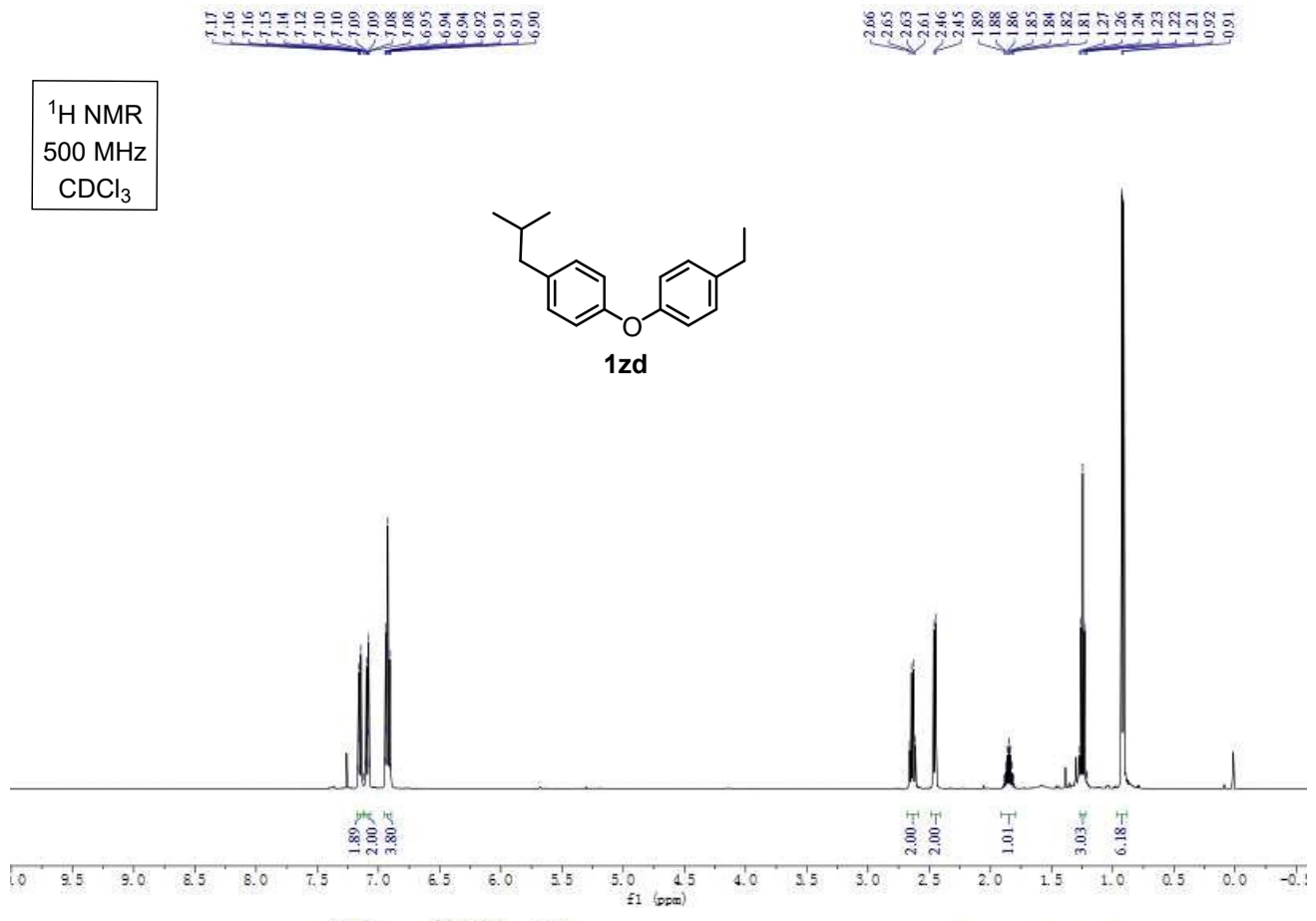
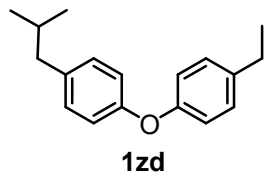


**Figure S7.** Light-off/on experiments of the reaction  $1a+2b \rightarrow 4$ .

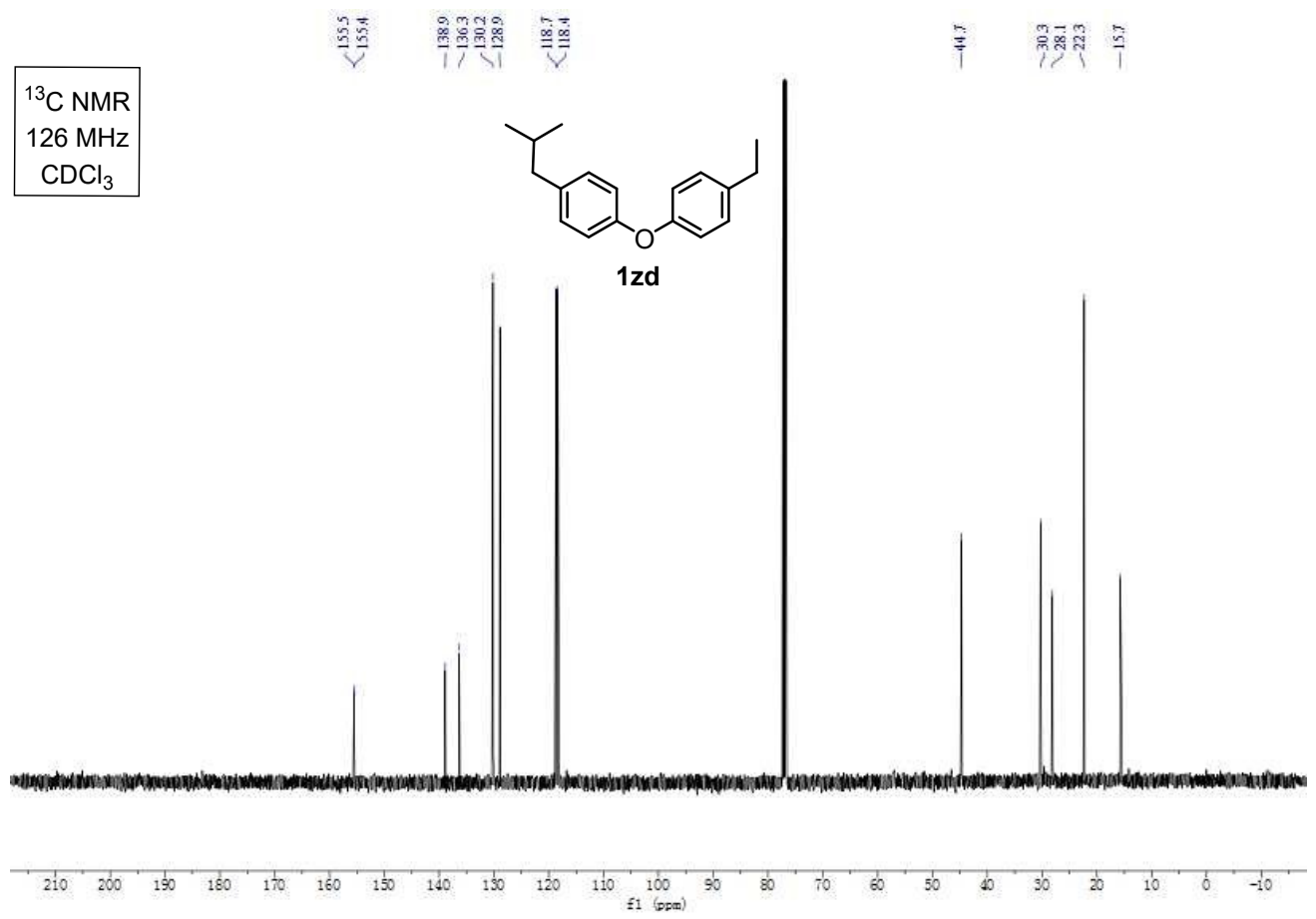
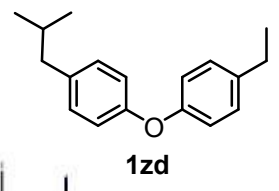
## 7. $^1\text{H}$ and $^{13}\text{C}$ NMR Spectrum



<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



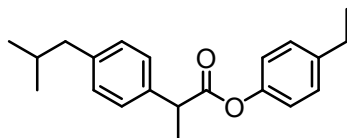
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



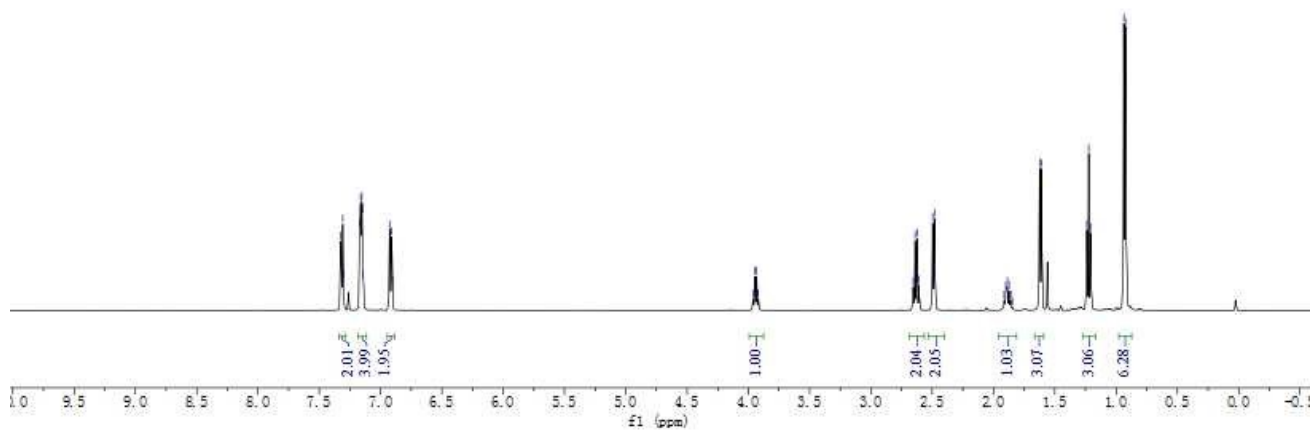
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

7.32  
7.32  
7.31  
7.30  
7.17  
7.16  
7.15  
7.15  
7.14  
6.93  
6.92  
6.91  
6.90

3.06  
3.05  
3.04  
3.03  
3.02  
2.65  
2.64  
2.62  
2.62  
2.61  
2.50  
2.49  
2.48  
2.48  
1.91  
1.90  
1.89  
1.87  
1.86  
1.85  
1.62  
1.61  
1.24  
1.22  
1.21  
0.94  
0.92



**1zj**



173.4

148.8

141.6

140.8

137.4

129.5

128.6

127.2

121.1

45.3

45.1

30.2

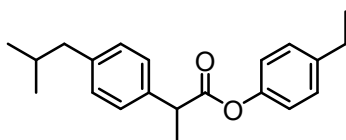
28.3

22.4

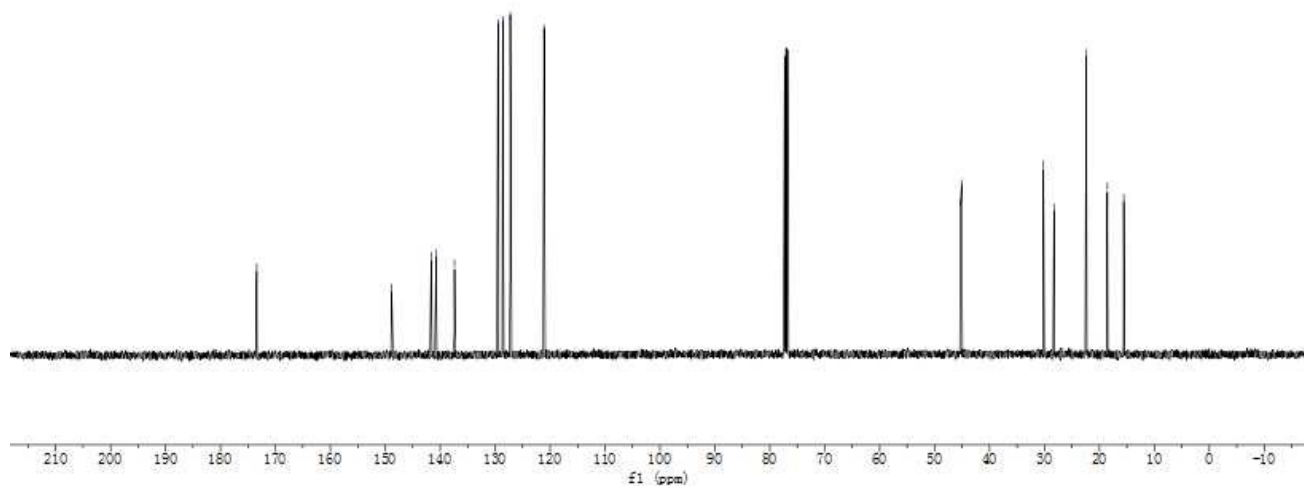
18.6

15.6

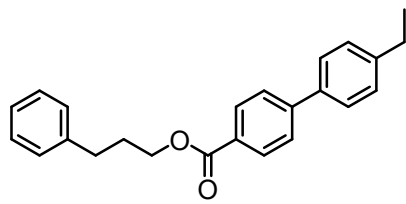
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



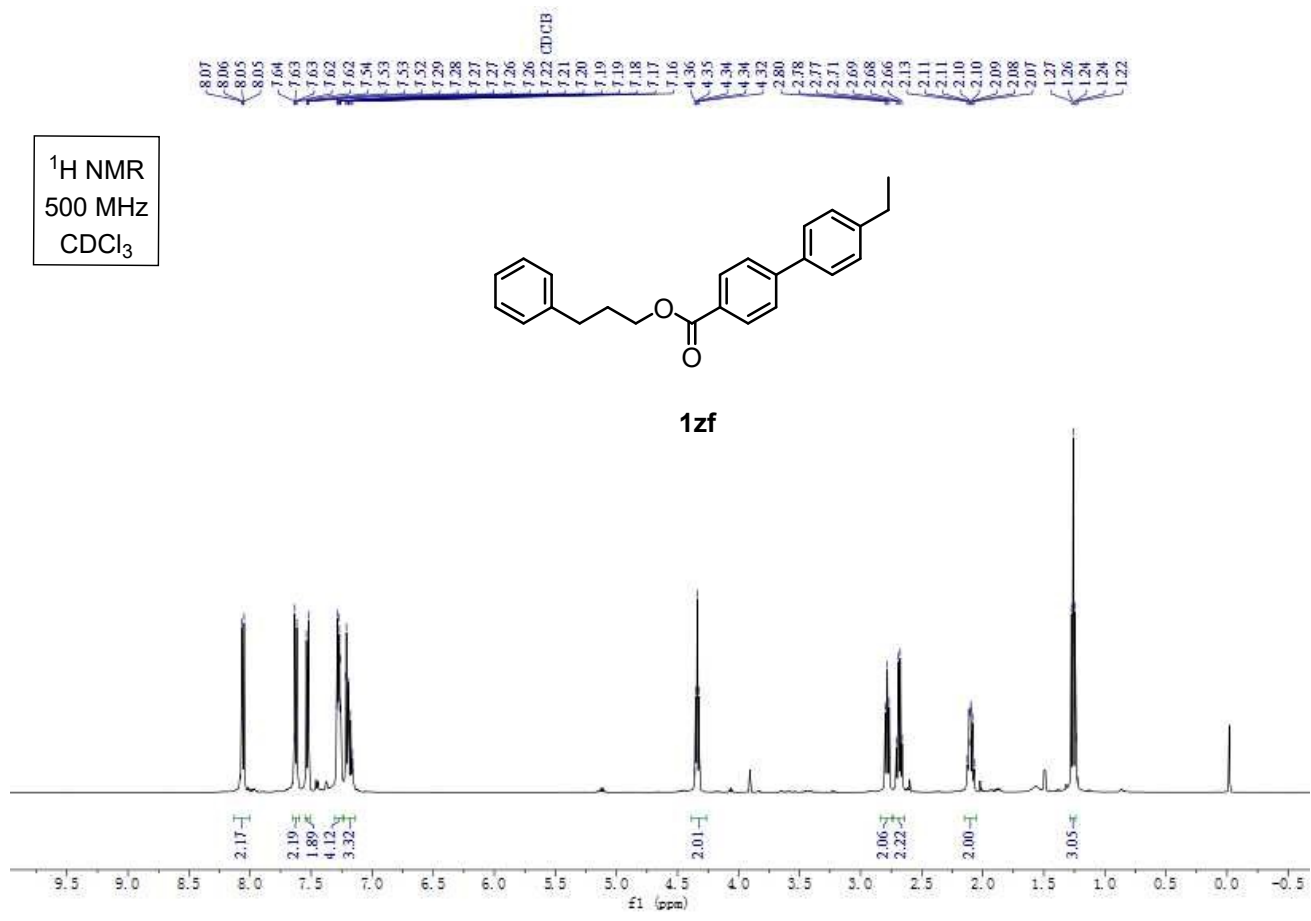
**1zj**



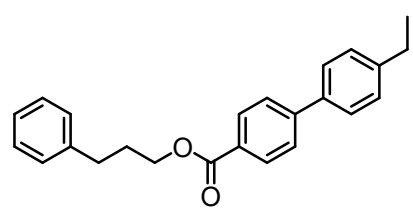
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



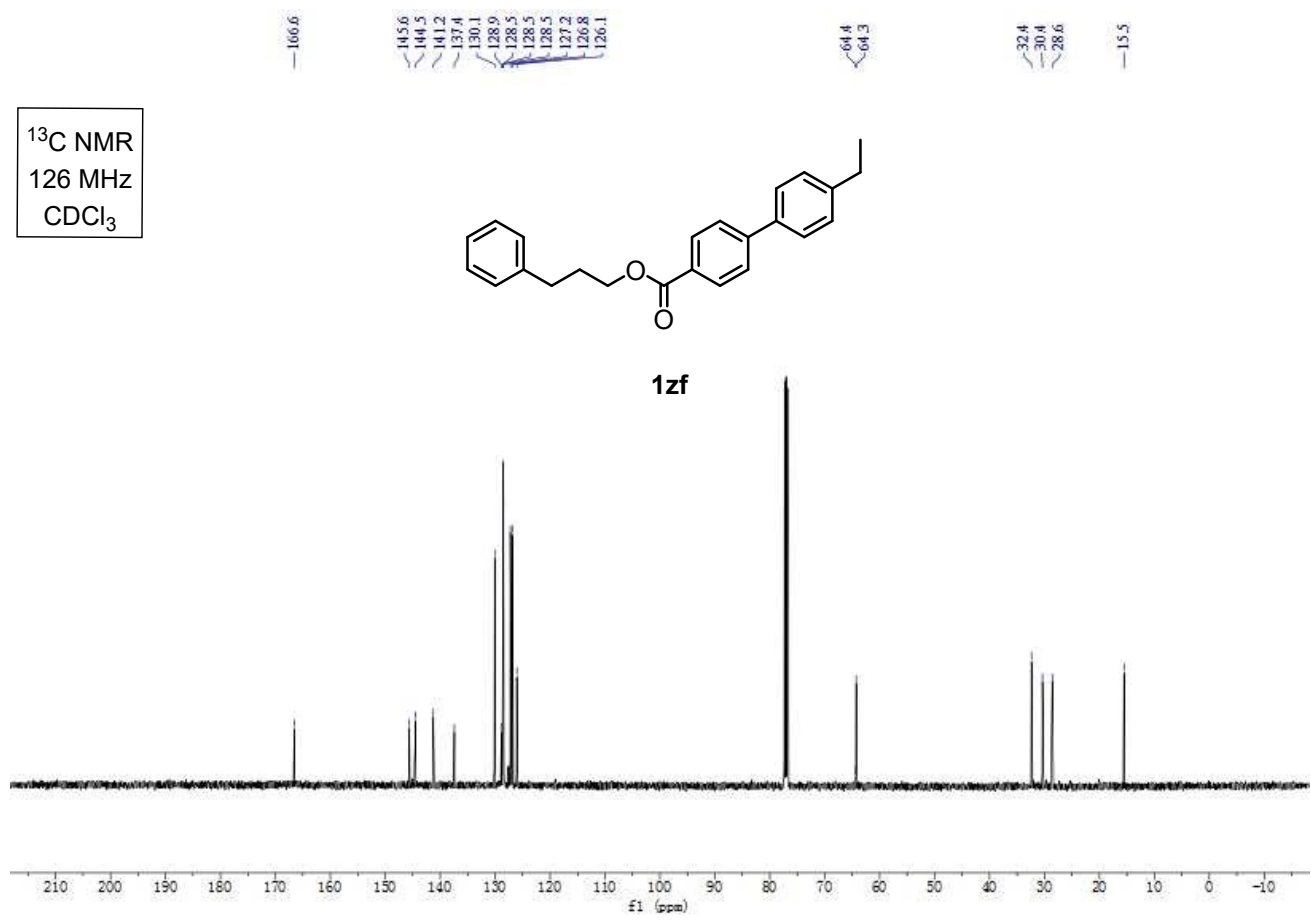
**1zf**

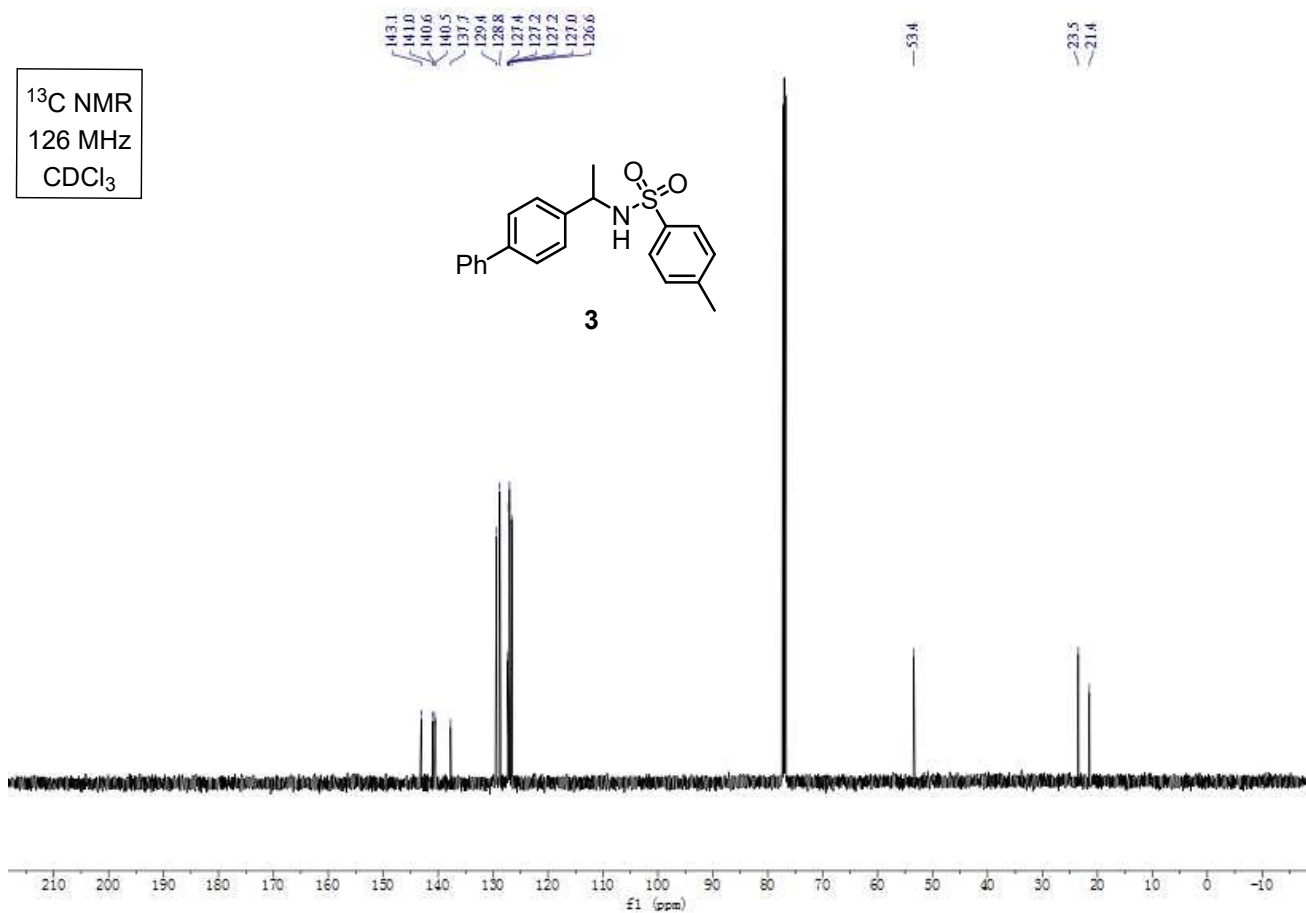
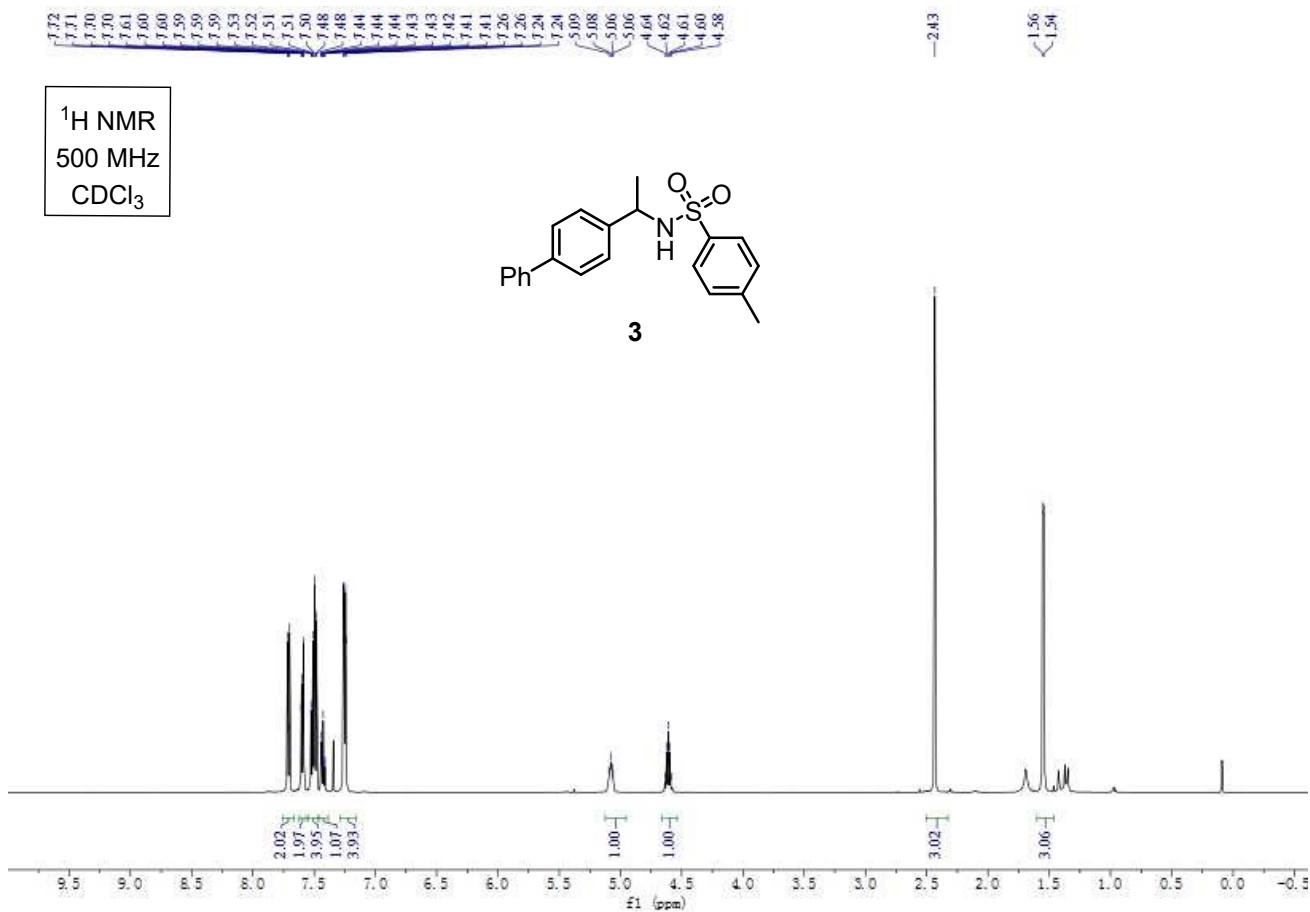


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



**1zf**

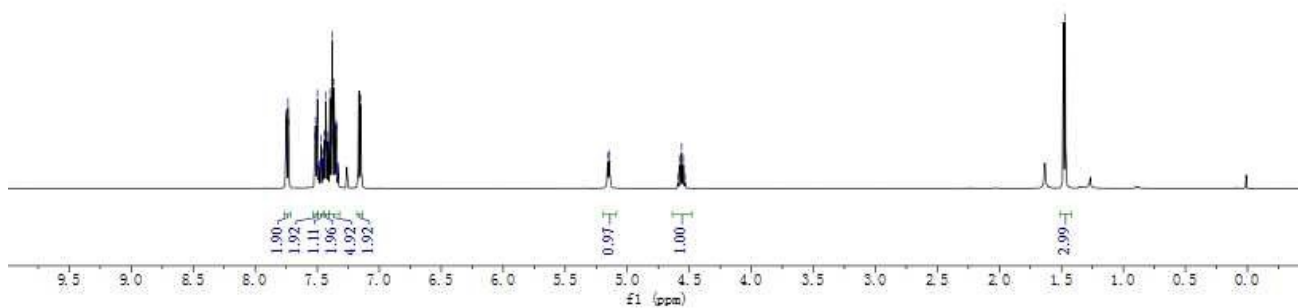
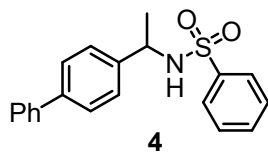




7.75  
7.75  
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7.15  
5.16  
4.58  
4.56  
4.55  
4.53

1.47

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

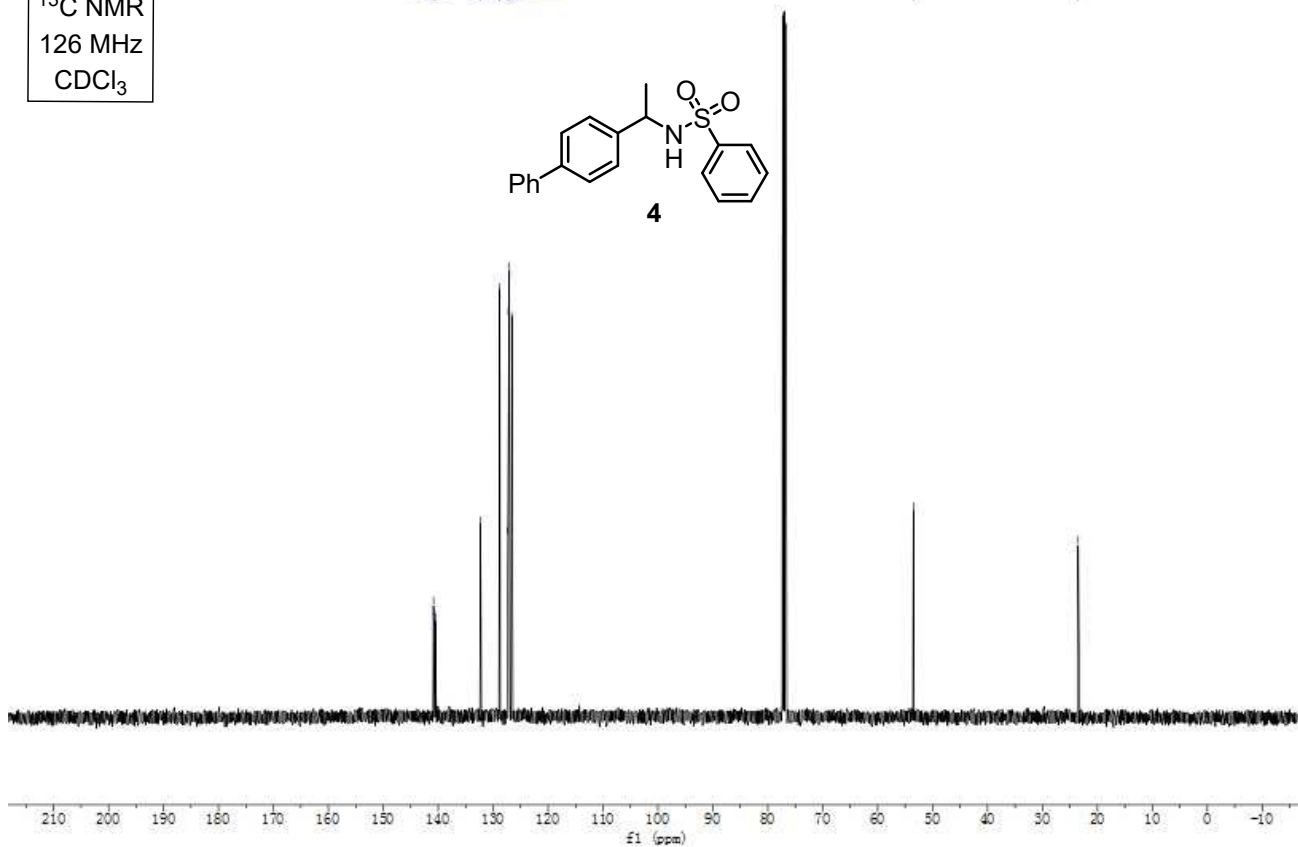
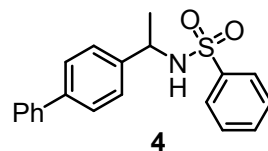


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

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140.7  
140.6  
140.5  
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128.8  
127.4  
127.2  
127.1  
127.0  
126.6

53.5

23.5



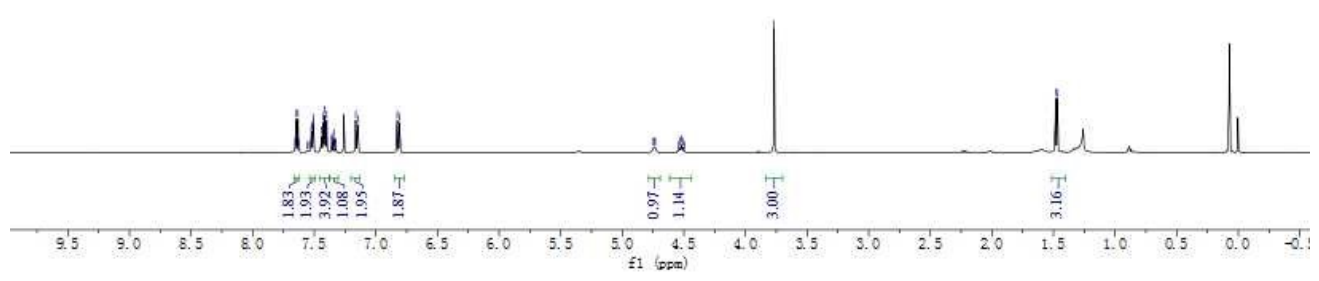
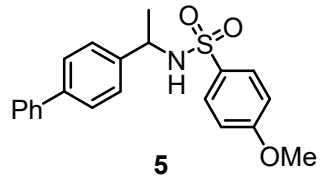


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7.42  
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7.40  
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6.81  
6.80

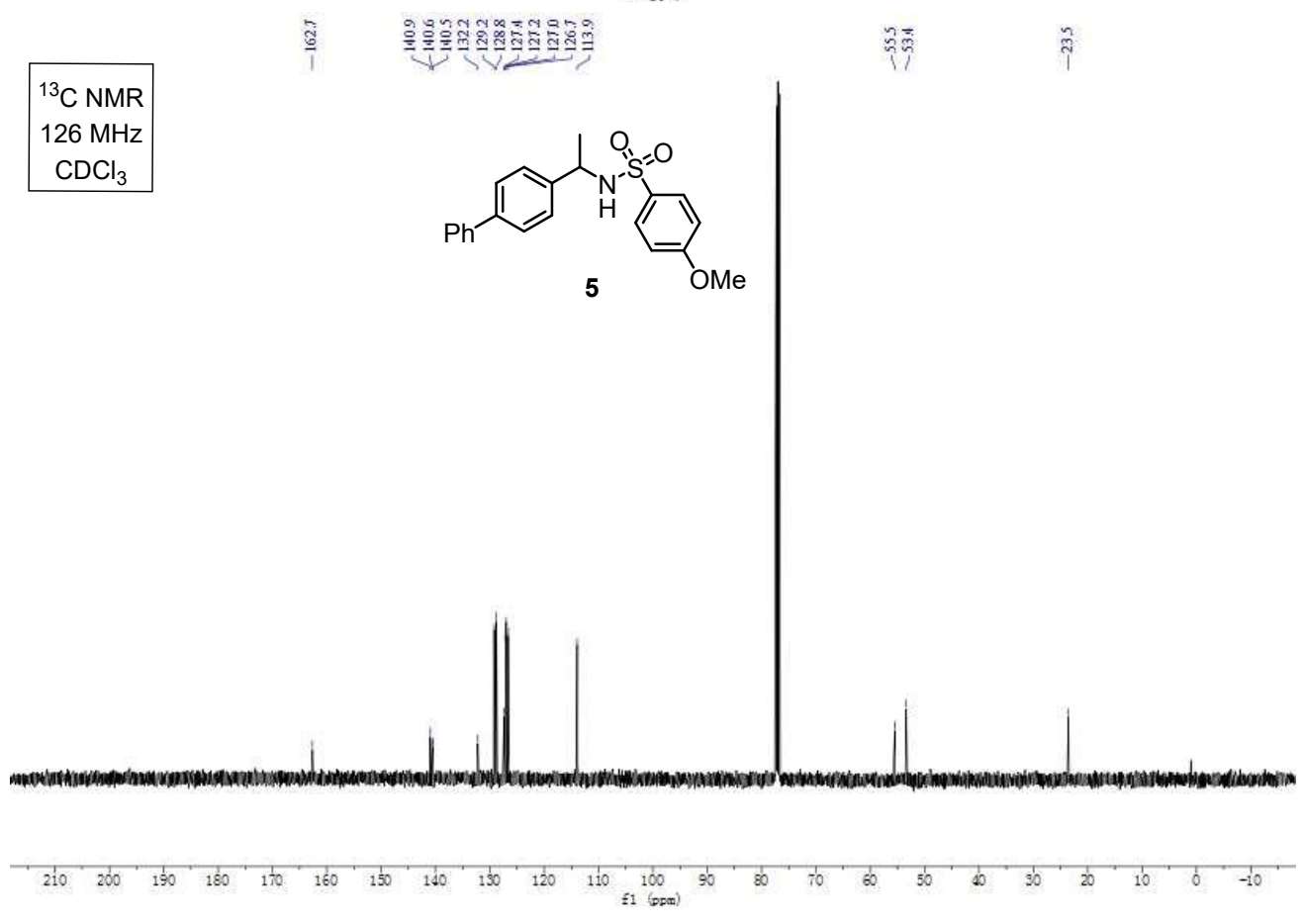
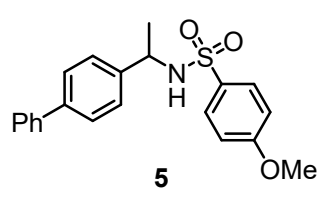
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4.52  
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4.49  
-3.77

1.48  
1.47

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



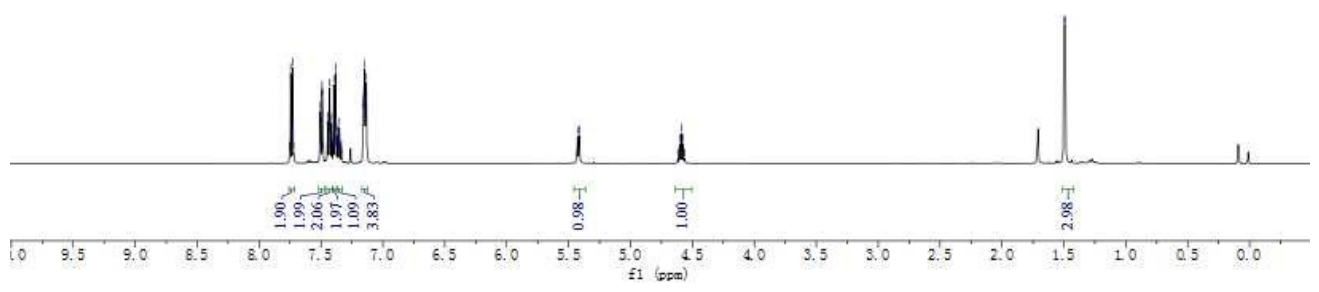
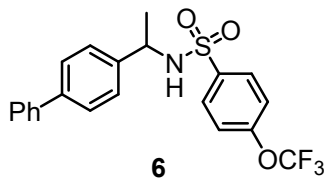
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



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7.16  
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7.15  
7.14  
7.14  
7.14  
7.13  
7.13  
5.83  
4.81  
4.60  
4.59  
4.58  
4.56

1.50  
1.49

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

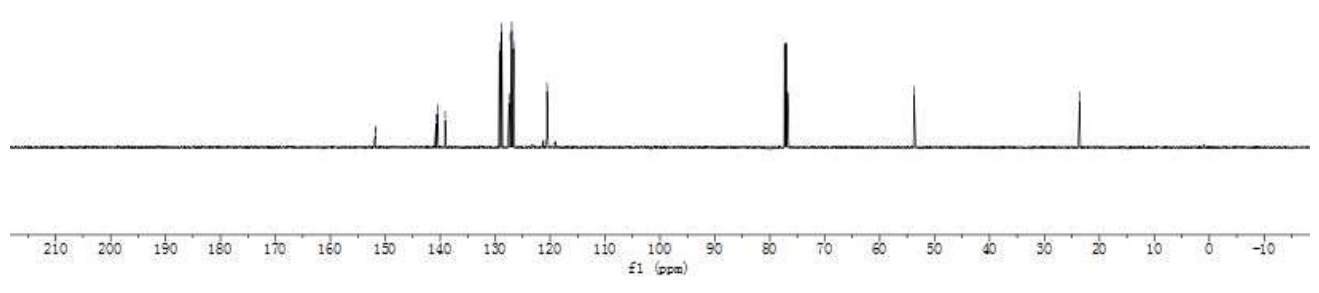
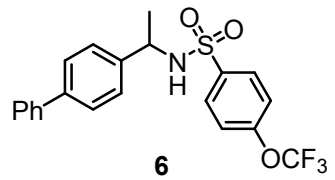


151.8  
151.8  
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140.4  
140.4  
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128.8  
127.5  
127.2  
127.0  
126.6  
120.6

-53.7

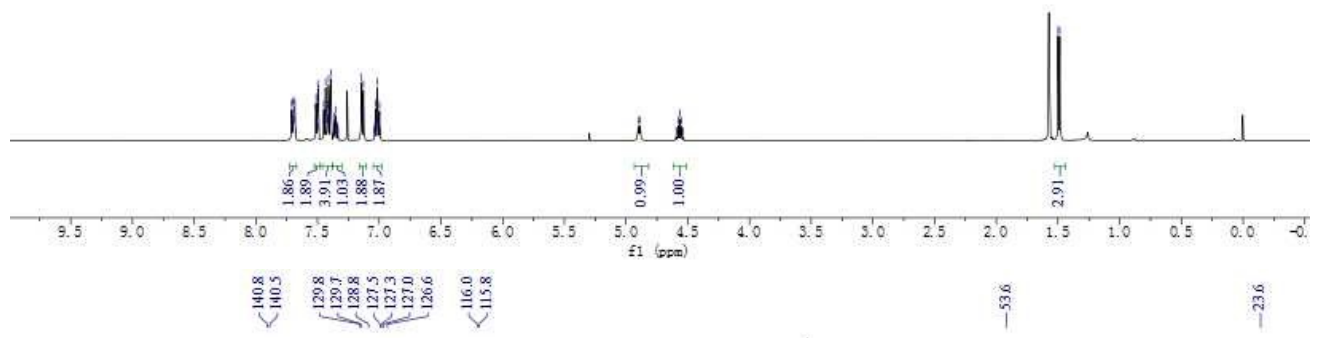
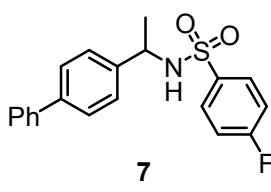
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<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

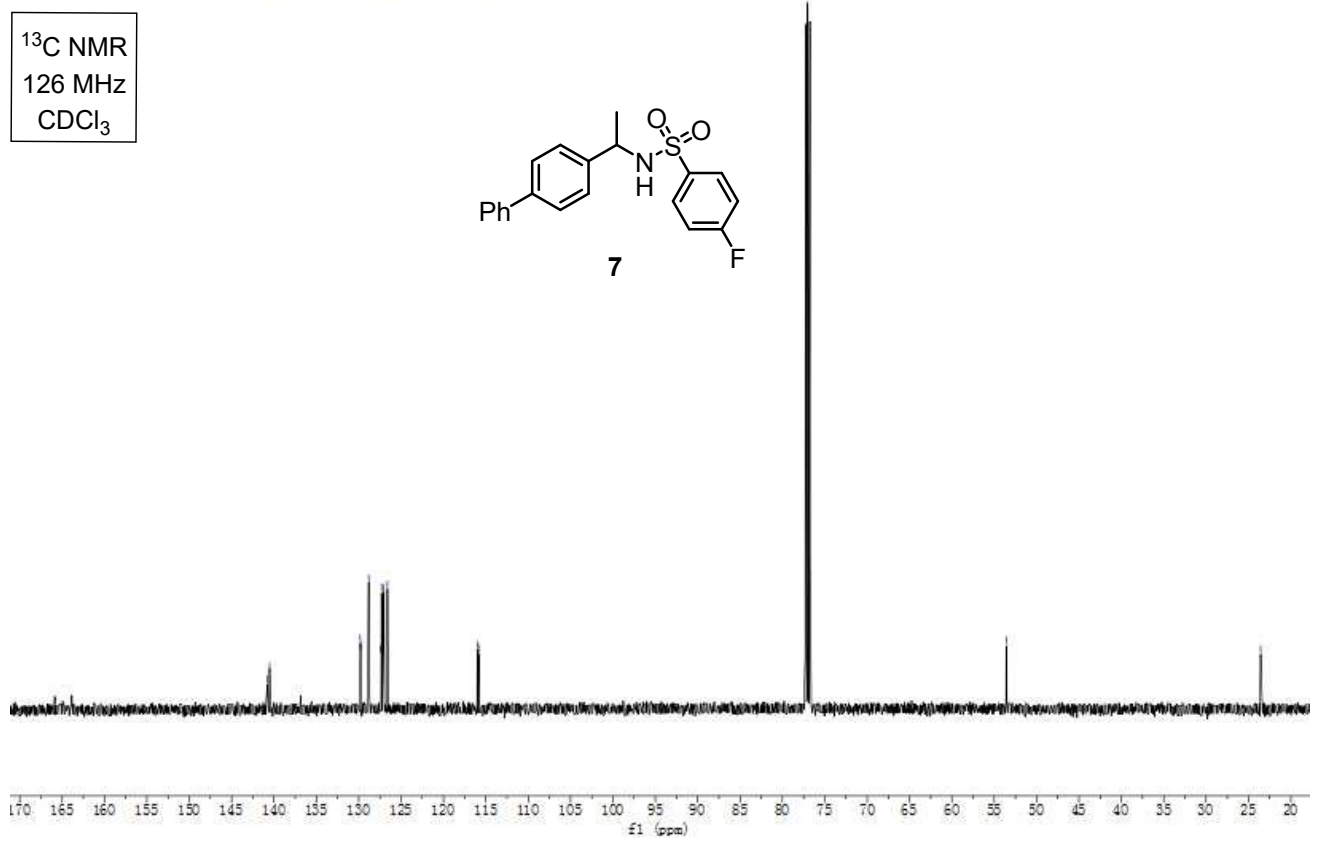
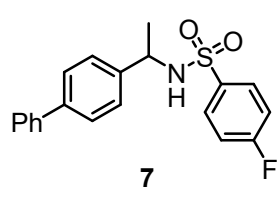


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6.99  
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4.54

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



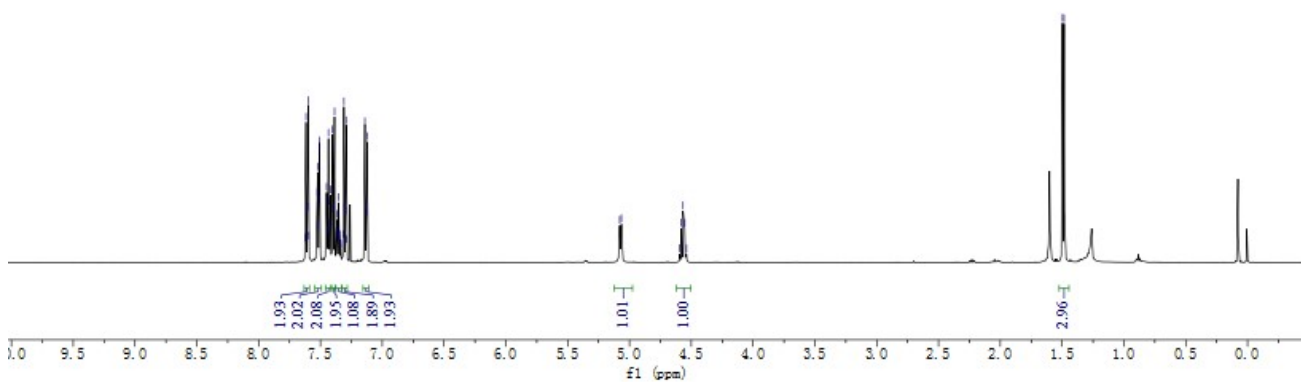
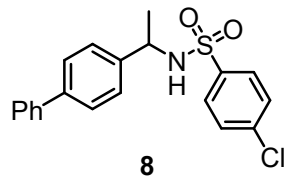
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



7.62  
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5.08  
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1.50  
1.48

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

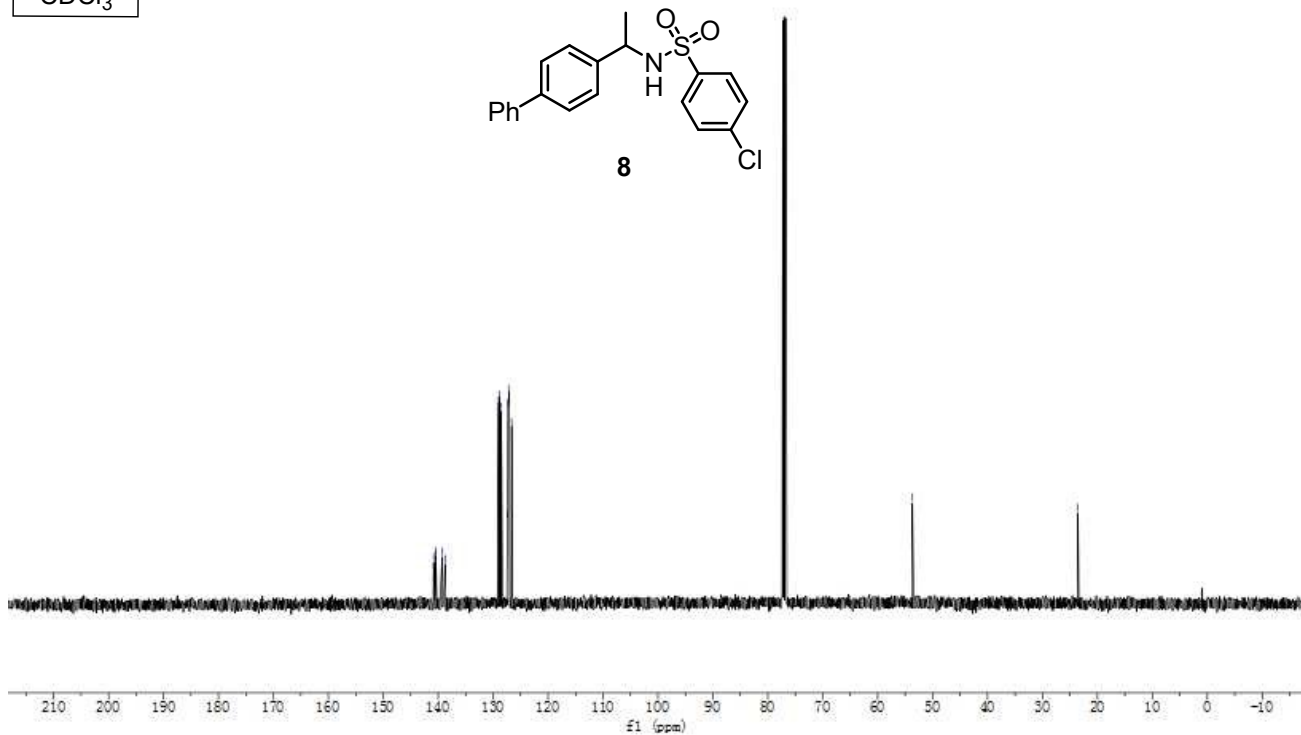
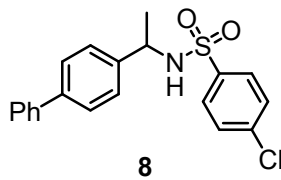


140.8  
140.5  
140.4  
139.3  
138.8  
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128.8  
128.5  
127.5  
127.3  
127.1  
126.6

-53.7

-23.5

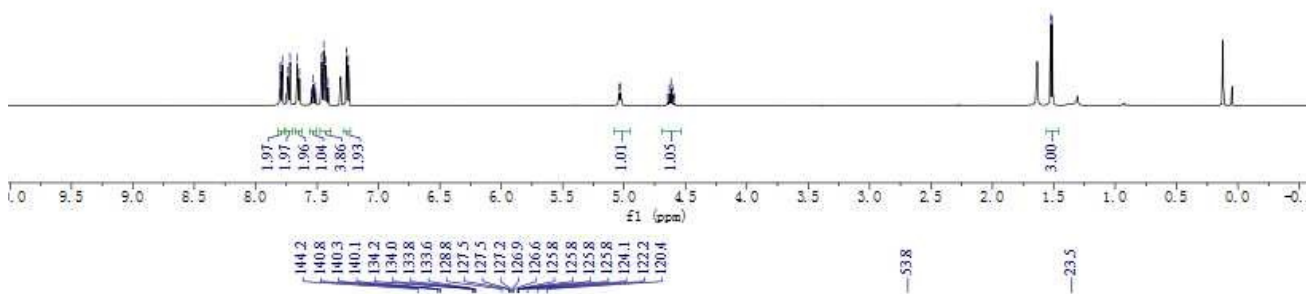
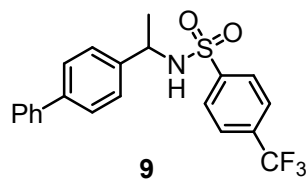
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



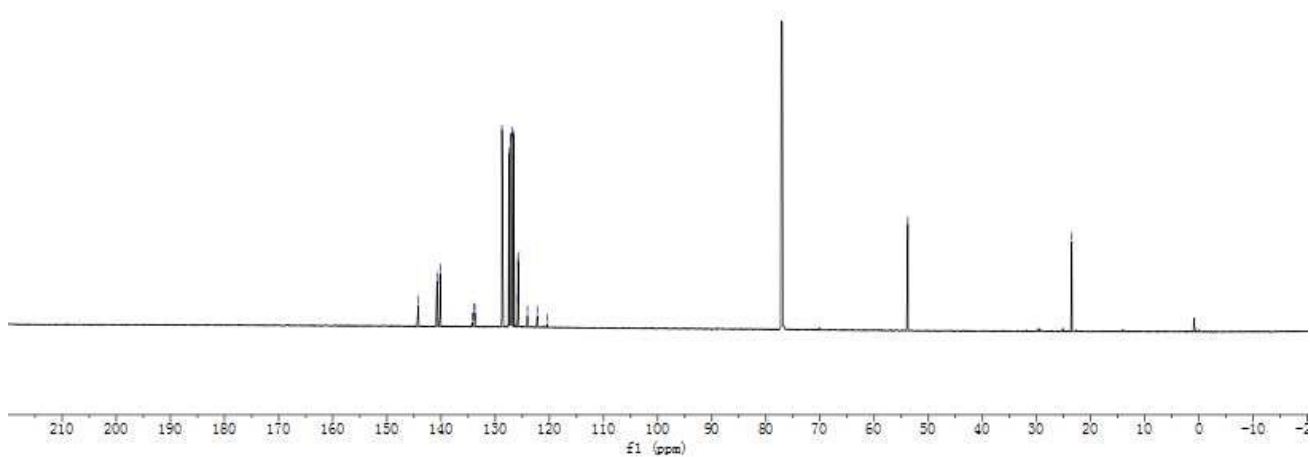
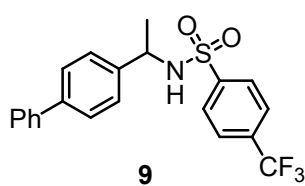
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5.03  
4.65  
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4.59

1.53  
1.51

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

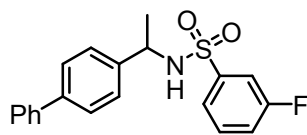


<sup>13</sup>C NMR  
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CDCl<sub>3</sub>

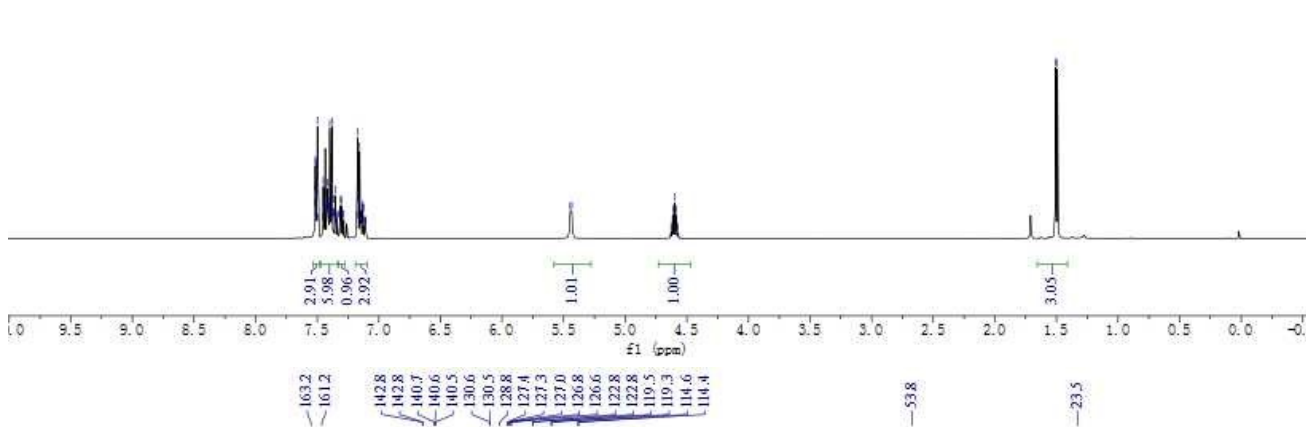


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7.12  
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7.11  
7.11  
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1.49

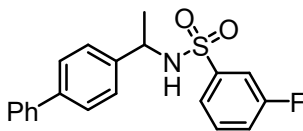
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



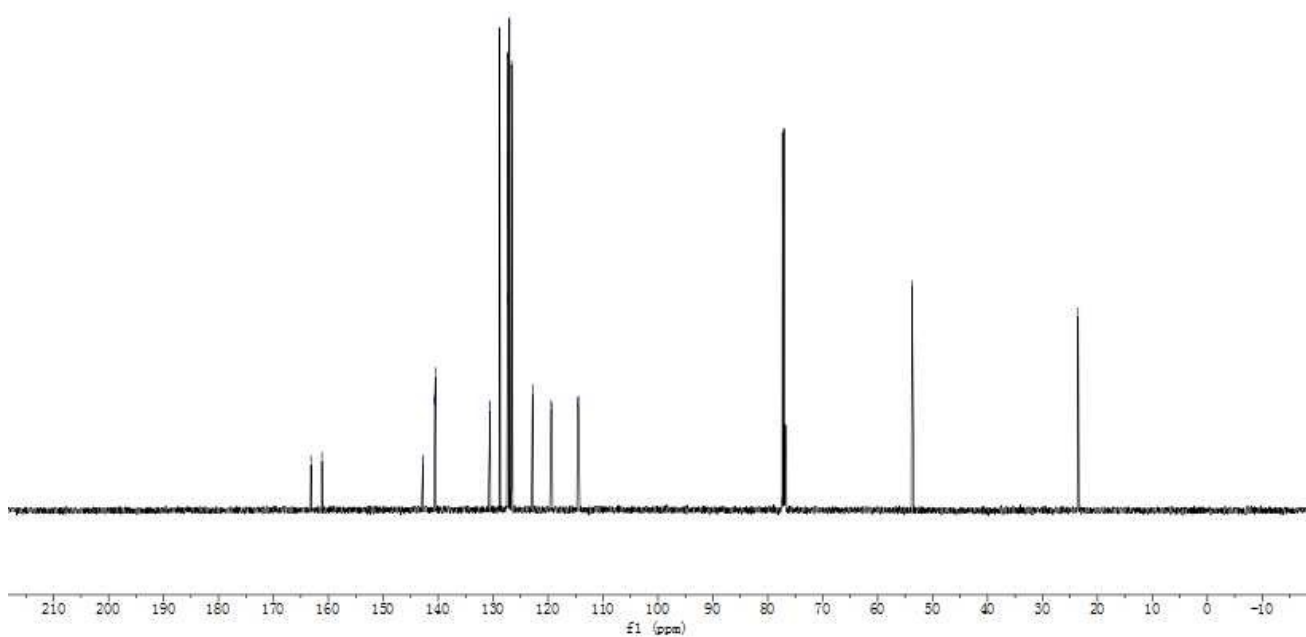
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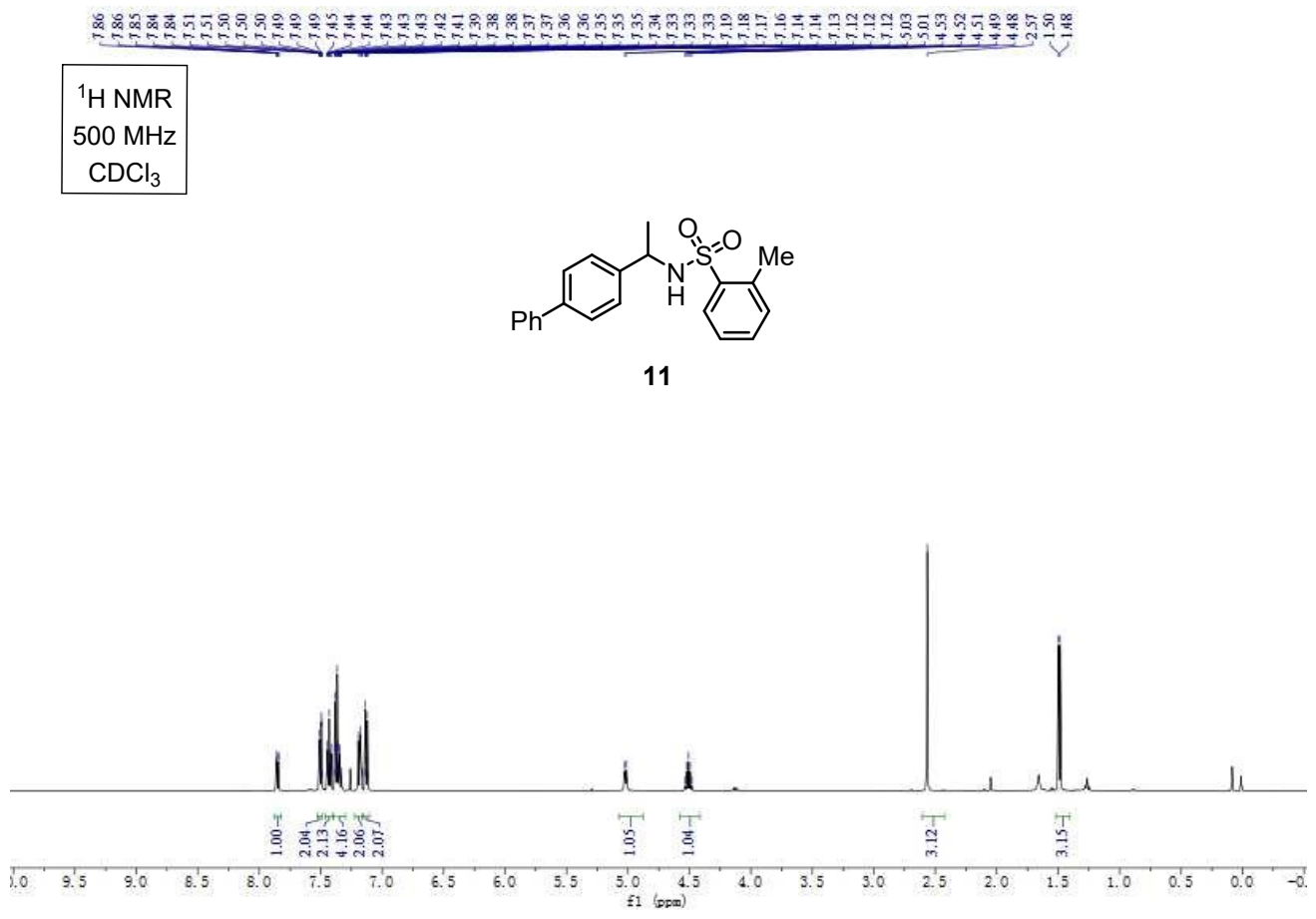
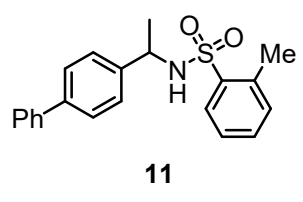
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



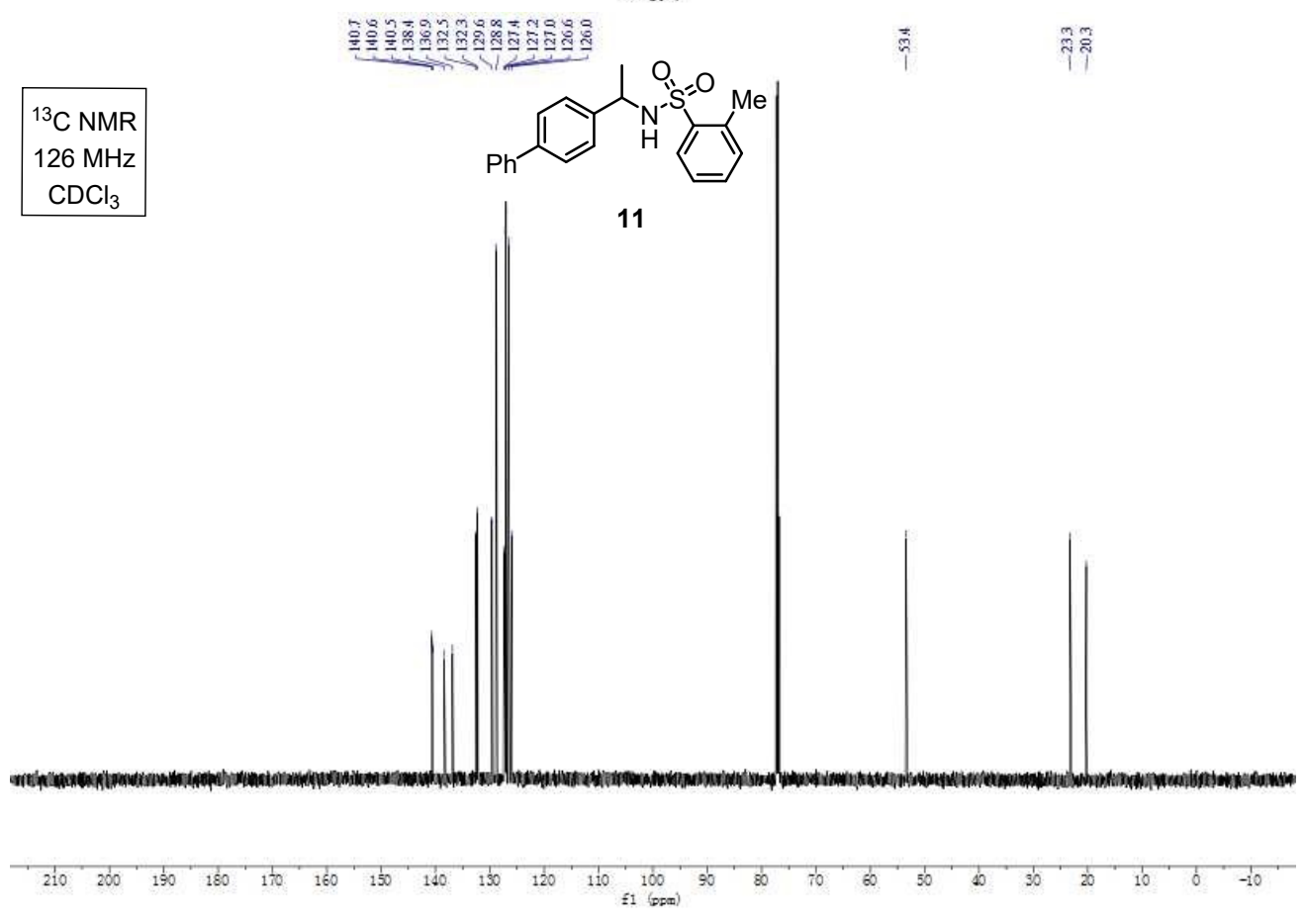
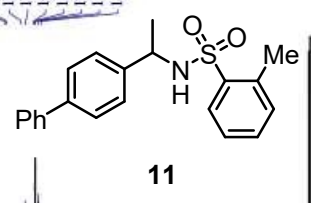
10



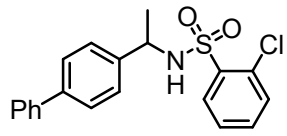
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

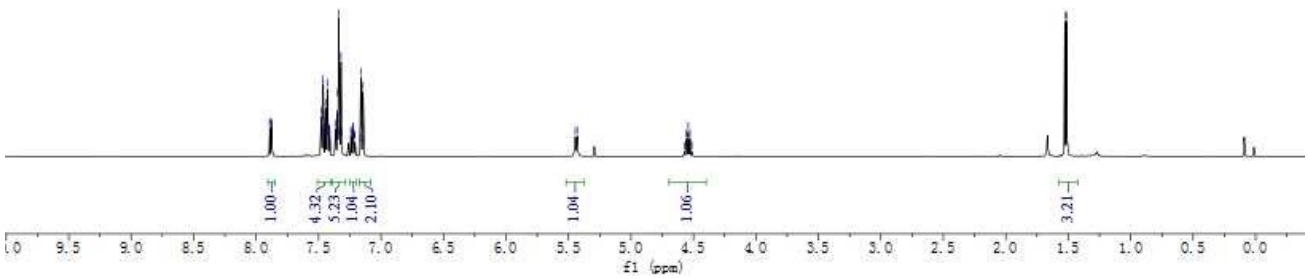


<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

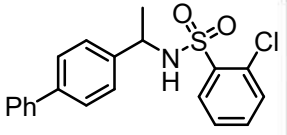


**12**

7.89  
7.89  
7.88  
7.87  
7.48  
7.48  
7.47  
7.47  
7.46  
7.46  
7.46  
7.44  
7.44  
7.44  
7.43  
7.43  
7.43  
7.42  
7.37  
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7.22  
7.22  
7.21  
7.21  
7.17  
7.16  
7.16  
7.15  
7.15  
7.14  
7.14  
5.45  
5.43  
4.57  
4.56  
4.54  
4.53  
4.51  
4.51  
1.51



<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

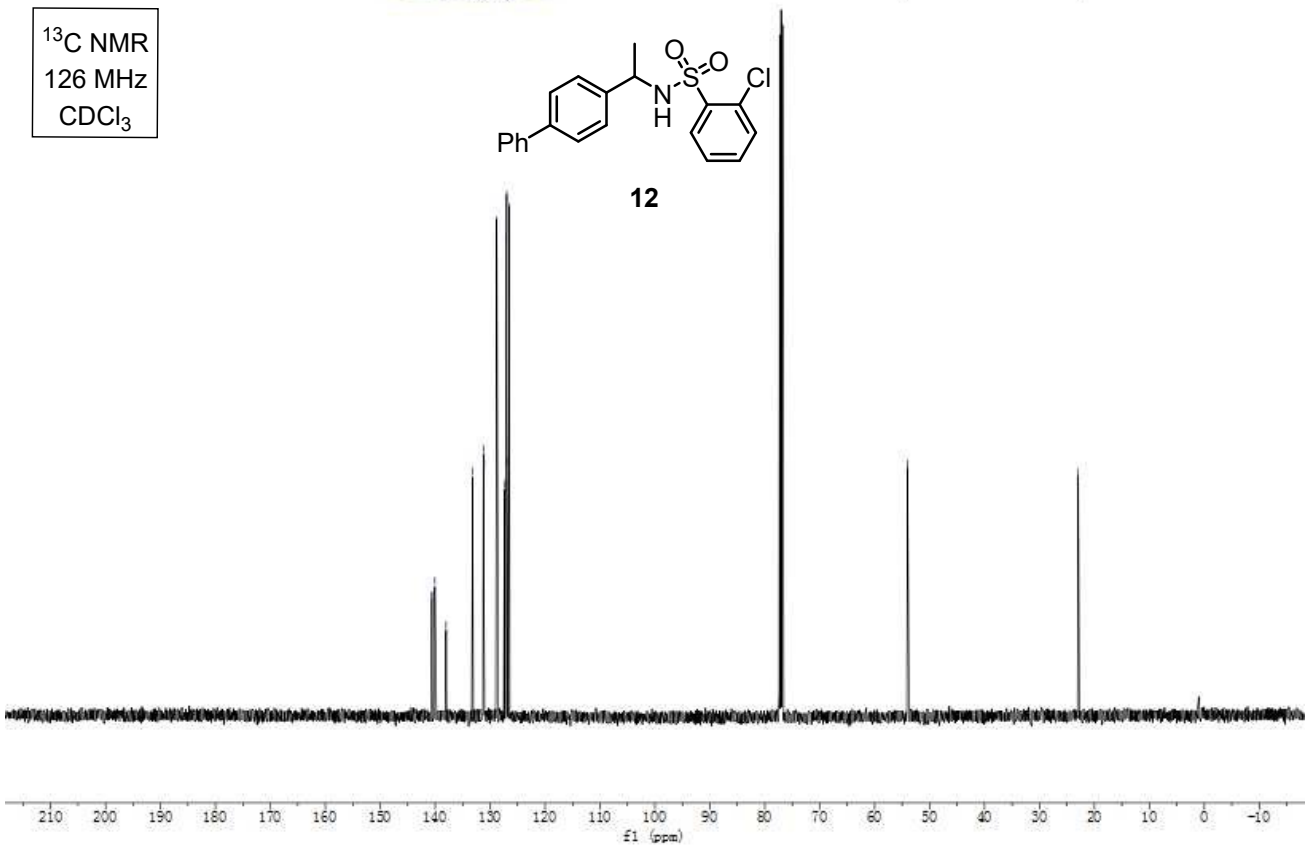


**12**

140.6  
140.6  
140.1  
138.0  
133.2  
131.3  
131.2  
131.1  
128.8  
127.4  
127.1  
127.0  
126.9  
126.5

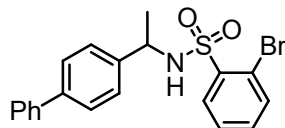
54.0

22.9



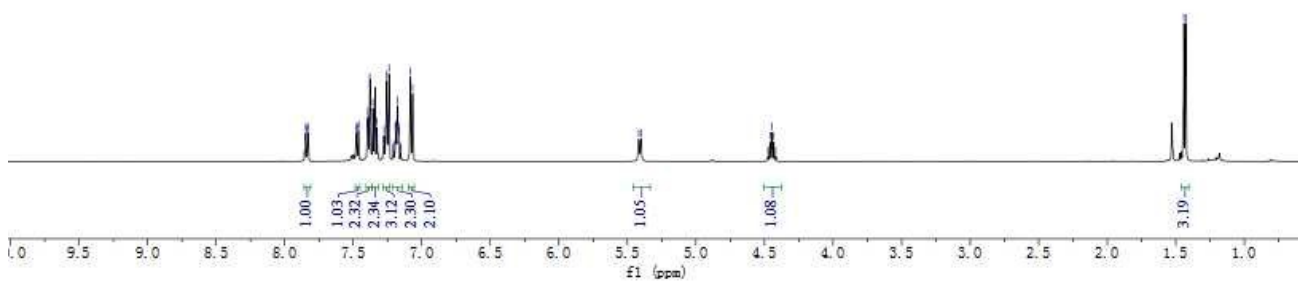


<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



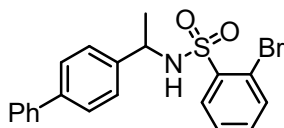
**13**

7.86  
7.85  
7.84  
7.83  
7.83  
7.83  
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7.47  
7.46  
7.46  
7.40  
7.39  
7.39  
7.38  
7.38  
7.38  
7.37  
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7.36  
7.35  
7.34  
7.34  
7.33  
7.33  
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7.27  
7.26  
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7.18  
7.17  
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7.16  
7.16  
7.15  
7.15  
7.08  
7.08  
7.07  
7.07  
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4.47  
4.46  
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4.43  
4.42  
1.44  
1.43

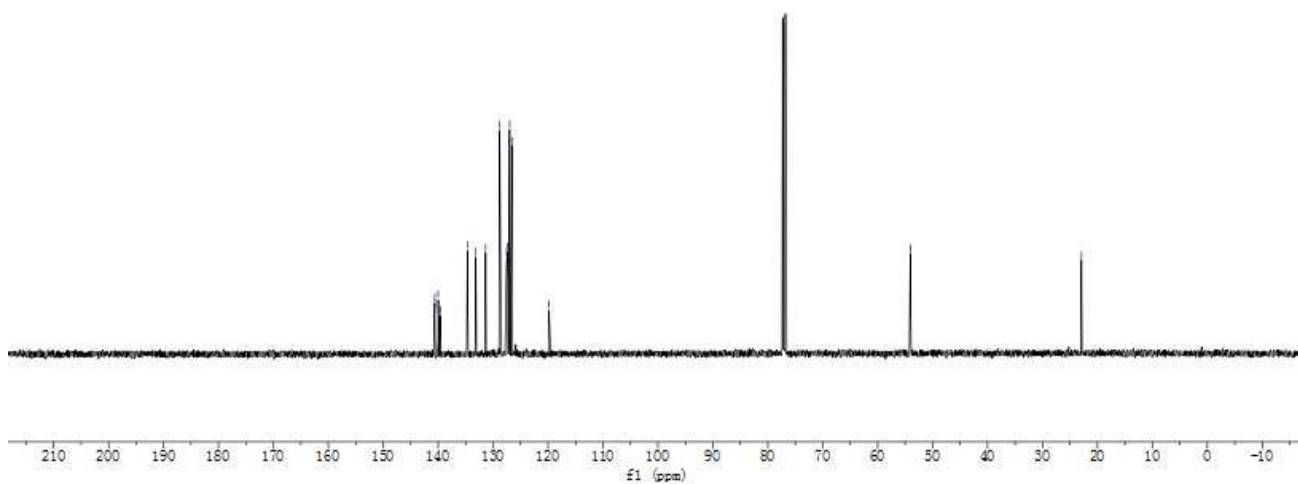


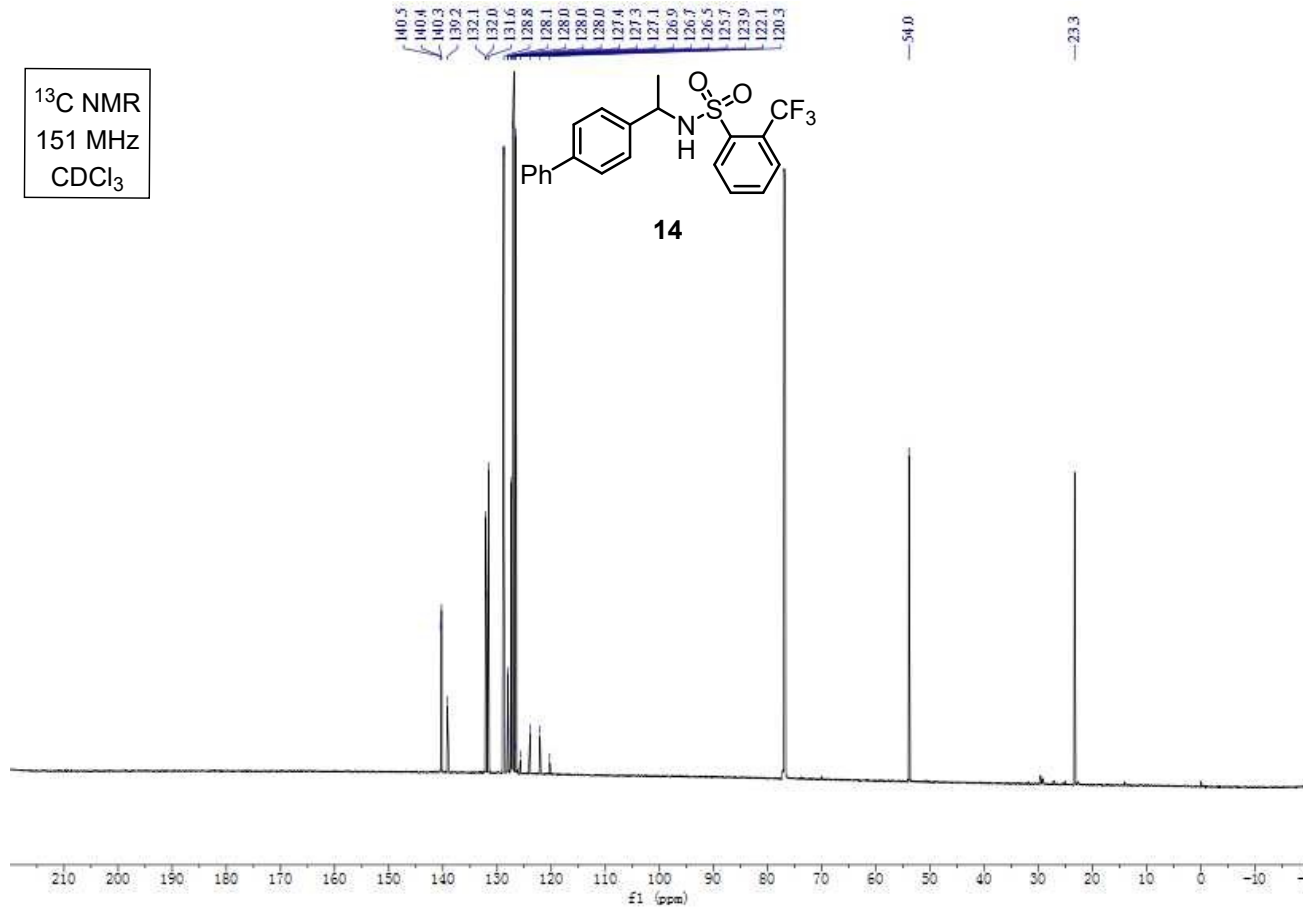
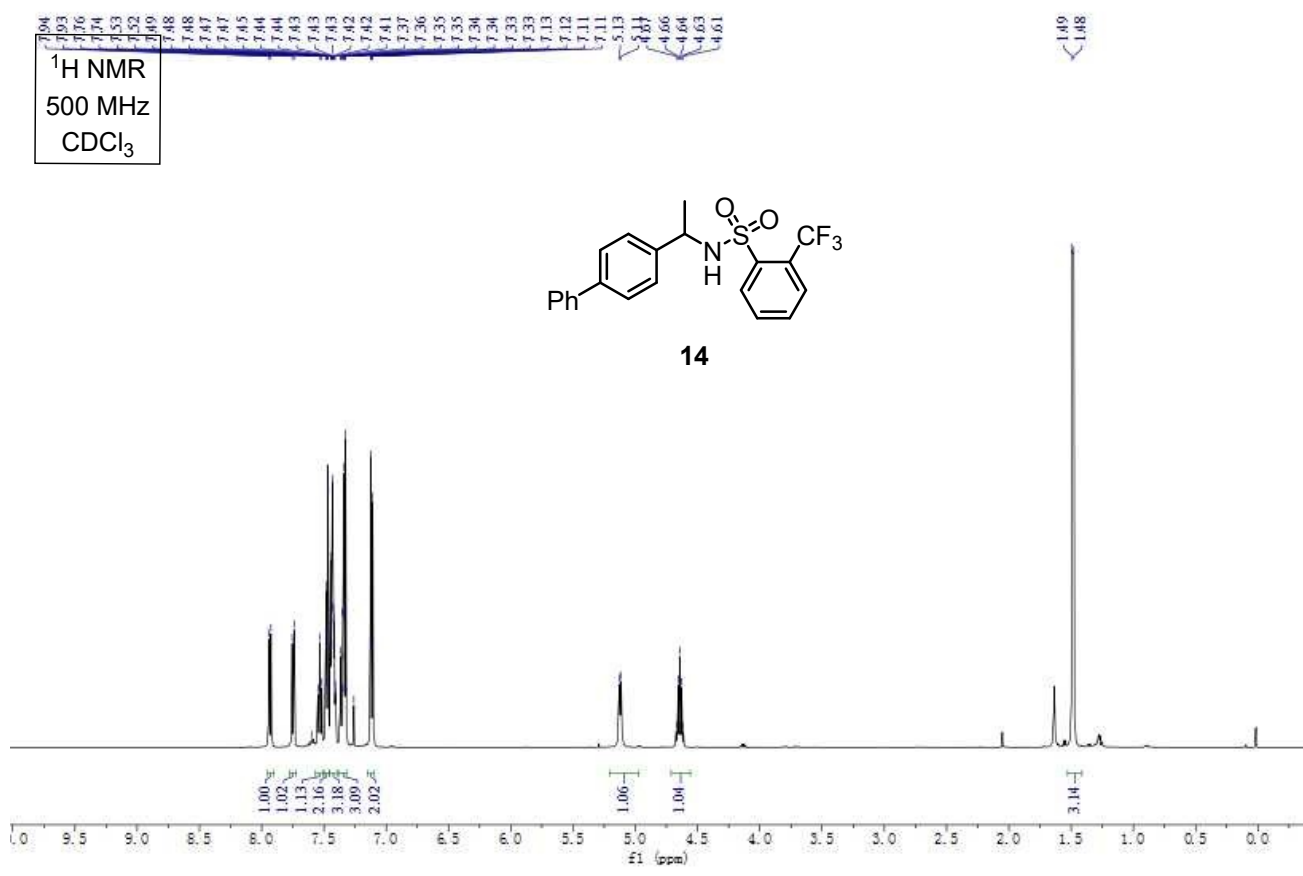
140.6  
140.6  
140.0  
139.6  
134.7  
133.1  
131.4  
128.8  
127.5  
127.4  
127.1  
127.0  
126.6  
119.8  
54.1  
22.9

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



**13**

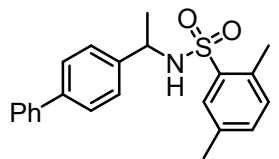




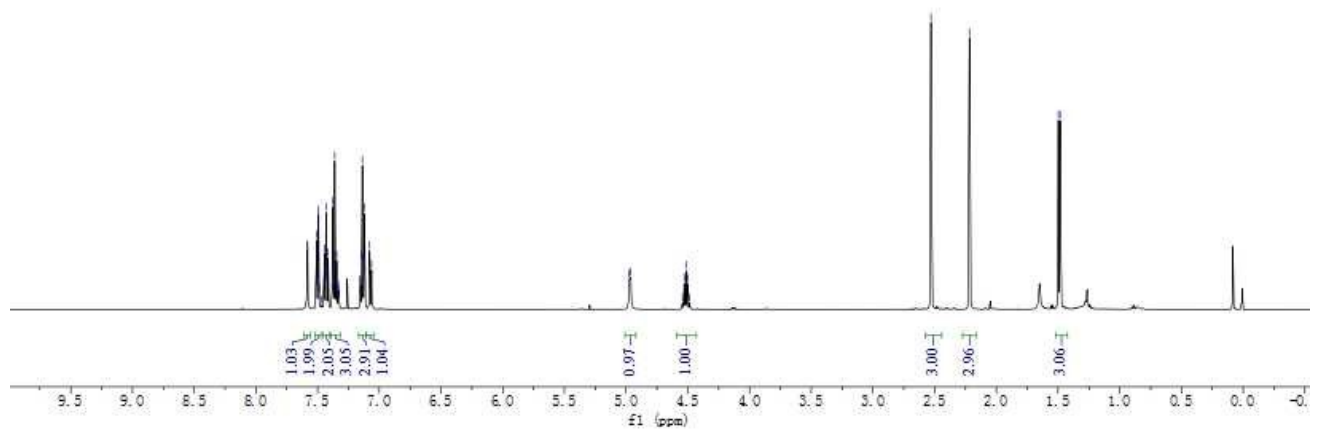
7.58  
7.58  
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7.50  
7.50  
7.49  
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7.45  
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7.43  
7.43  
7.43  
7.42  
7.42  
7.41  
7.38  
7.37  
7.36  
7.36  
7.36  
7.35  
7.35  
7.33  
7.33  
7.16  
7.15  
7.14  
7.14  
7.13  
7.12  
7.12  
7.08  
7.06  
4.98  
4.98  
4.53  
4.52  
4.50  
4.49

-2.53  
-2.22  
1.50  
1.48

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



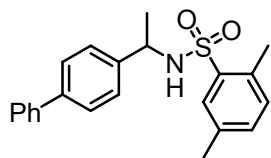
**15**



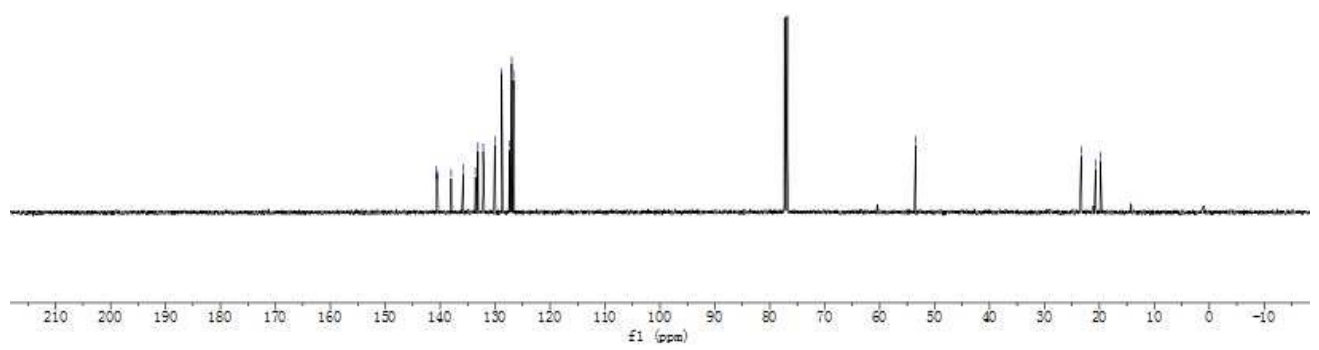
140.7  
140.6  
140.4  
138.0  
135.8  
133.6  
133.1  
132.2  
130.1  
128.8  
127.4  
127.1  
127.0  
126.7

-53.4  
23.3  
20.7  
19.8

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



**15**

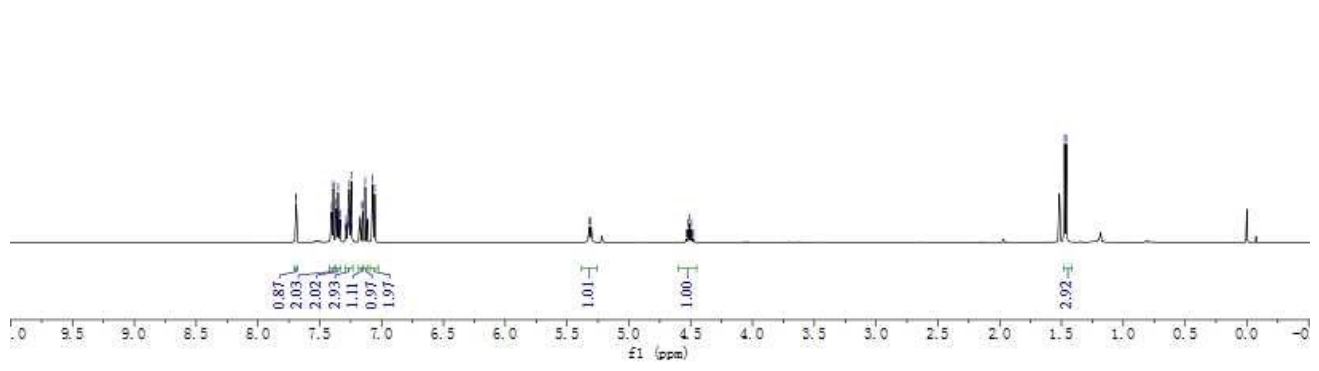
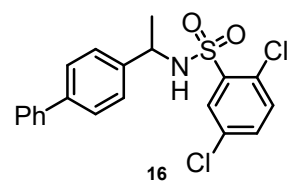


7.70  
7.69  
7.61  
7.40  
7.39  
7.37  
7.36  
7.35  
7.34  
7.34  
7.29  
7.28  
7.27  
7.27  
7.26  
7.25  
7.25  
7.18  
7.18  
7.17  
7.16  
7.15  
7.13  
7.07  
7.06  
5.96  
5.92  
5.31

4.53  
4.52  
4.51  
4.49  
4.48

1.47  
1.46

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

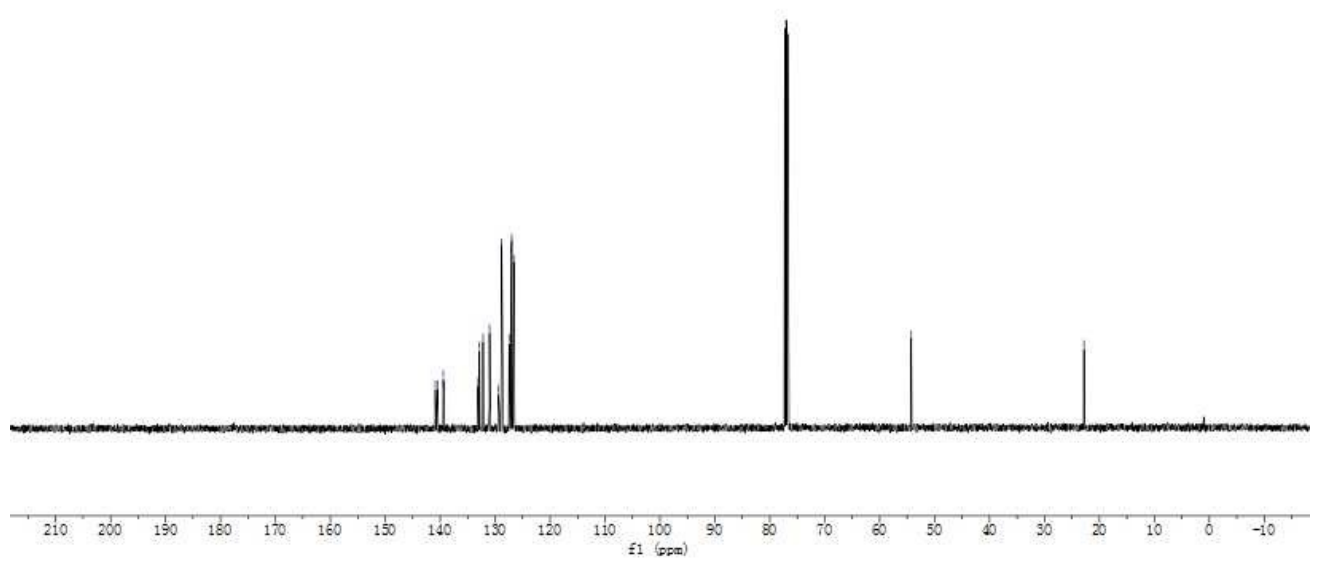
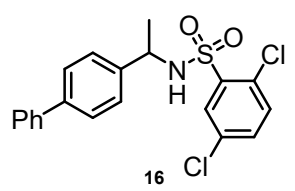


140.9  
140.4  
139.4  
133.3  
133.1  
132.8  
132.2  
131.0  
129.4  
128.8  
127.5  
127.0  
127.0  
126.6

54.2

22.8

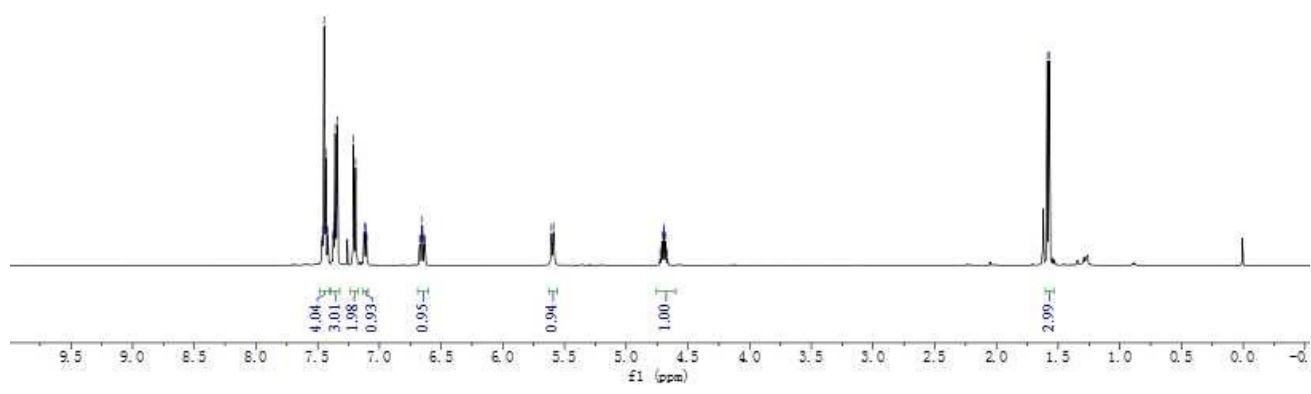
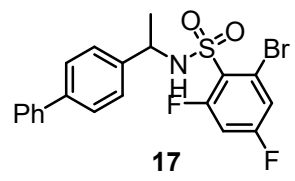
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



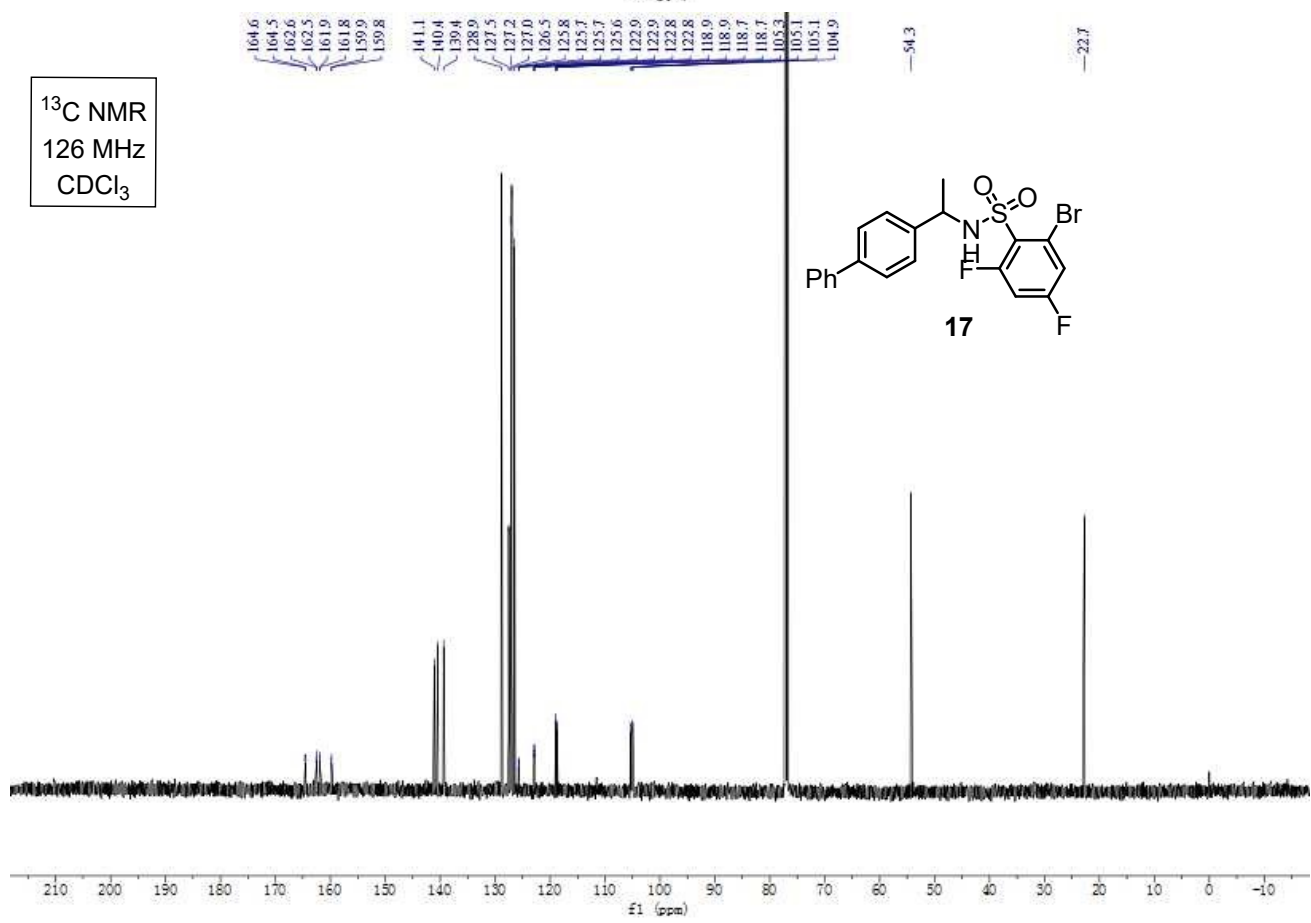
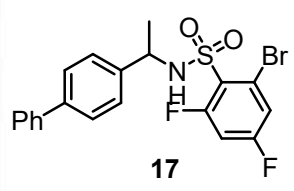
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7.43  
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7.36  
7.36  
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7.35  
7.34  
7.21  
7.21  
7.21  
7.20  
7.19  
7.12  
7.11  
6.67  
6.66  
6.65  
6.65  
6.64  
6.63  
5.61  
5.59  
4.72  
4.71  
4.70  
4.69  
4.68  
4.67

1.59  
1.57

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

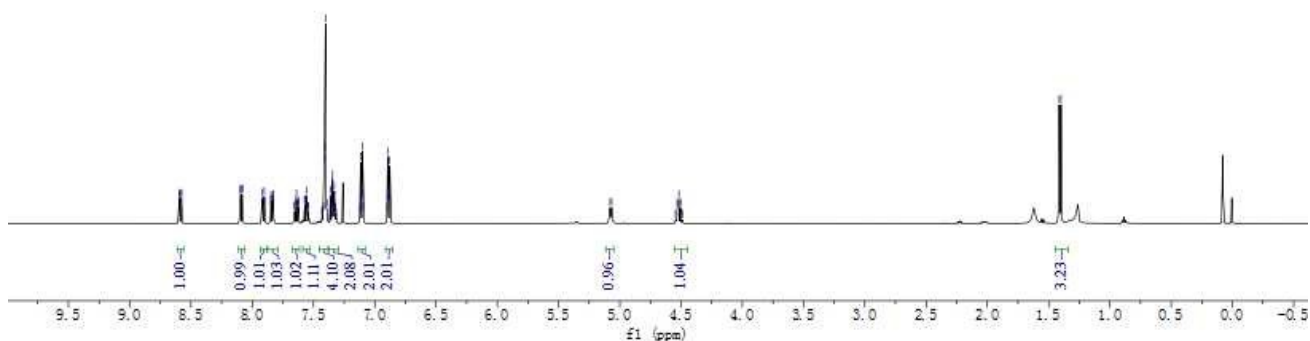
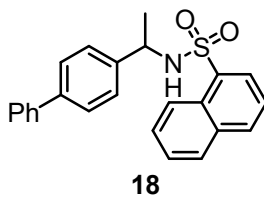


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



8.60  
8.59  
8.58  
8.58  
8.10  
8.10  
8.08  
8.08  
7.92  
7.92  
7.90  
7.90  
7.90  
7.85  
7.85  
7.84  
7.83  
7.83  
7.66  
7.65  
7.64  
7.64  
7.64  
7.62  
7.62  
7.57  
7.57  
7.56  
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7.55  
7.54  
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7.54  
7.42  
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7.35  
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7.34  
7.33  
7.33  
7.33  
7.32  
7.12  
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7.11  
7.11  
7.10  
7.10  
7.09  
7.09  
6.90  
6.89  
6.89  
6.88  
6.88  
5.08  
5.07  
4.53  
4.52  
4.50  
4.50  
1.41  
1.40

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

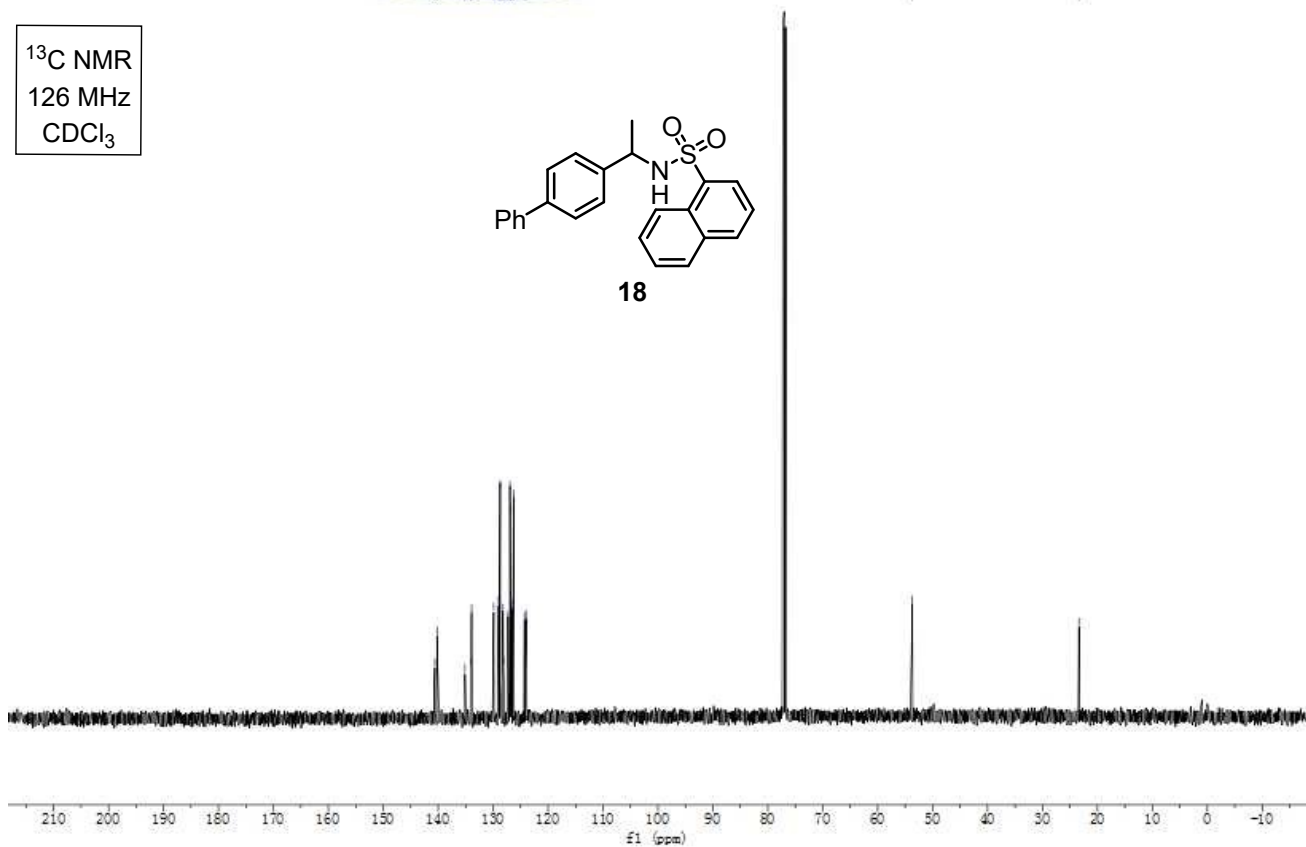
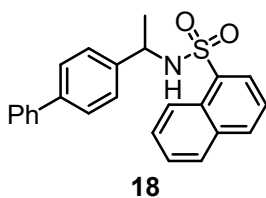


140.6  
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135.1  
134.1  
133.9  
129.9  
129.0  
128.7  
128.2  
128.1  
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126.7  
126.6  
126.3  
124.3  
124.0

-53.7

-23.4

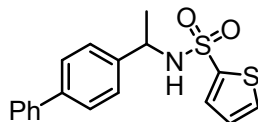
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



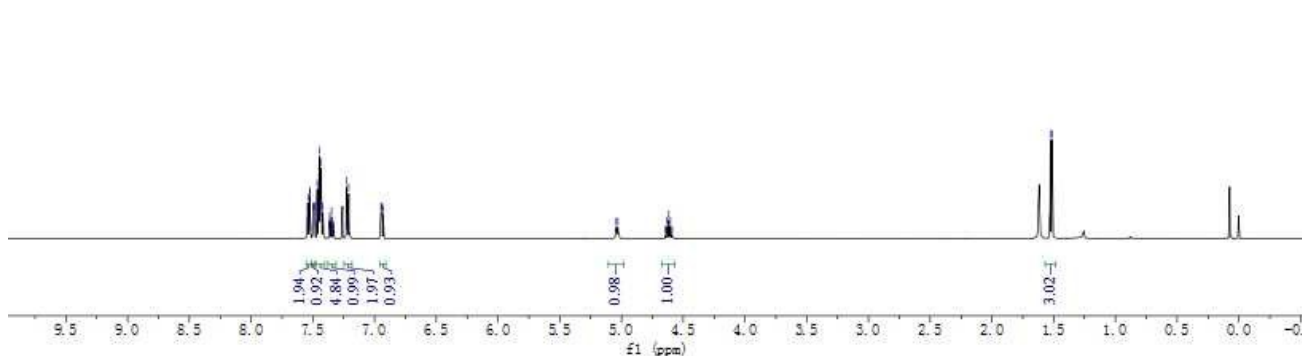
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7.42  
7.42  
7.36  
7.35  
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7.22  
7.21  
7.21  
6.95  
6.94  
6.94  
6.93  
5.04  
5.03  
4.84  
4.63  
4.62  
4.60  
4.59

1.53  
1.51

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



**19**

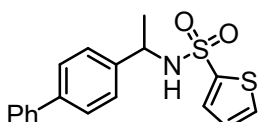


141.8  
140.8  
140.6  
140.6  
132.3  
131.7  
128.8  
127.4  
127.3  
127.1  
127.0  
126.5

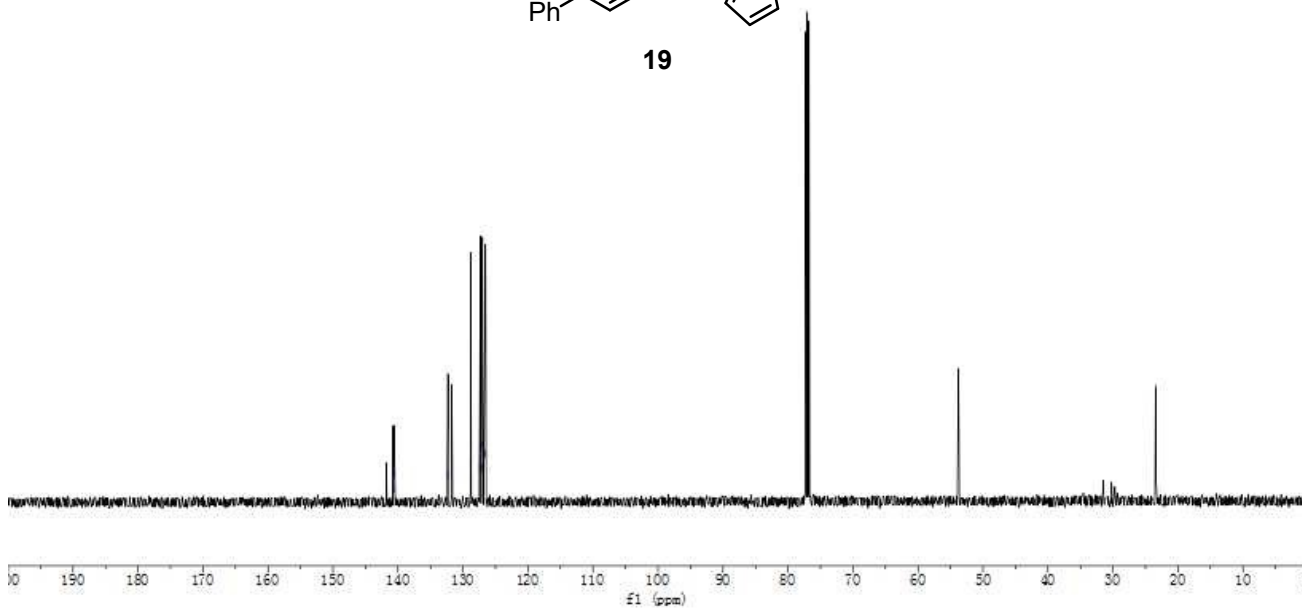
53.8

23.4

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

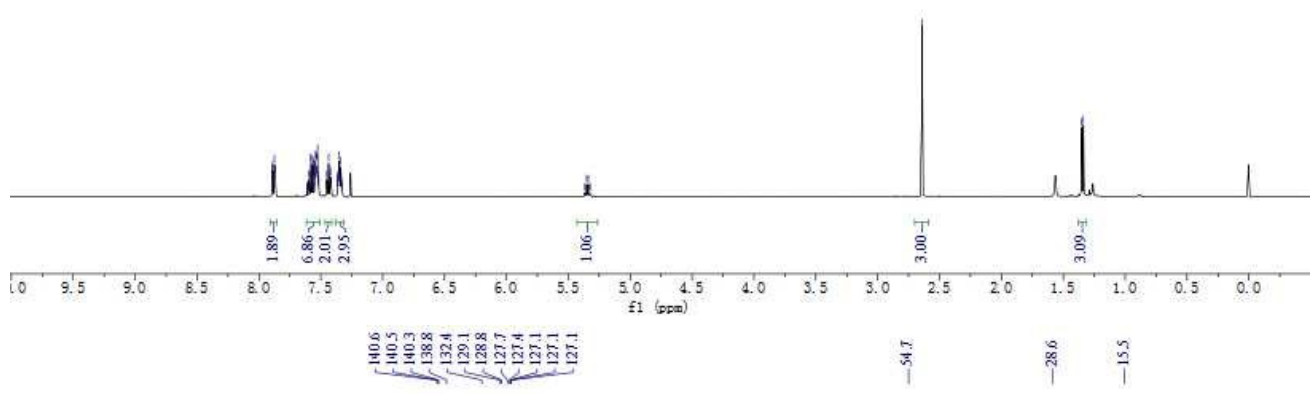
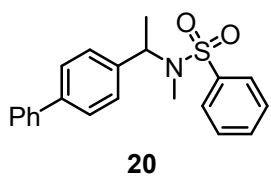


**19**

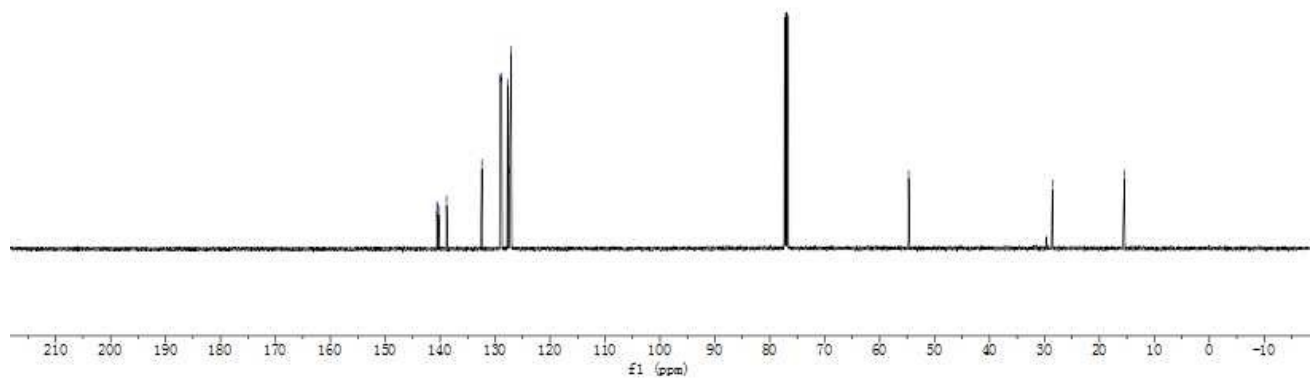
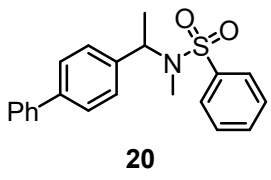


7.89  
7.89  
7.89  
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7.87  
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7.60  
7.59  
7.59  
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7.33  
7.33  
5.37  
5.35  
5.34  
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2.64  
1.35  
1.34

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

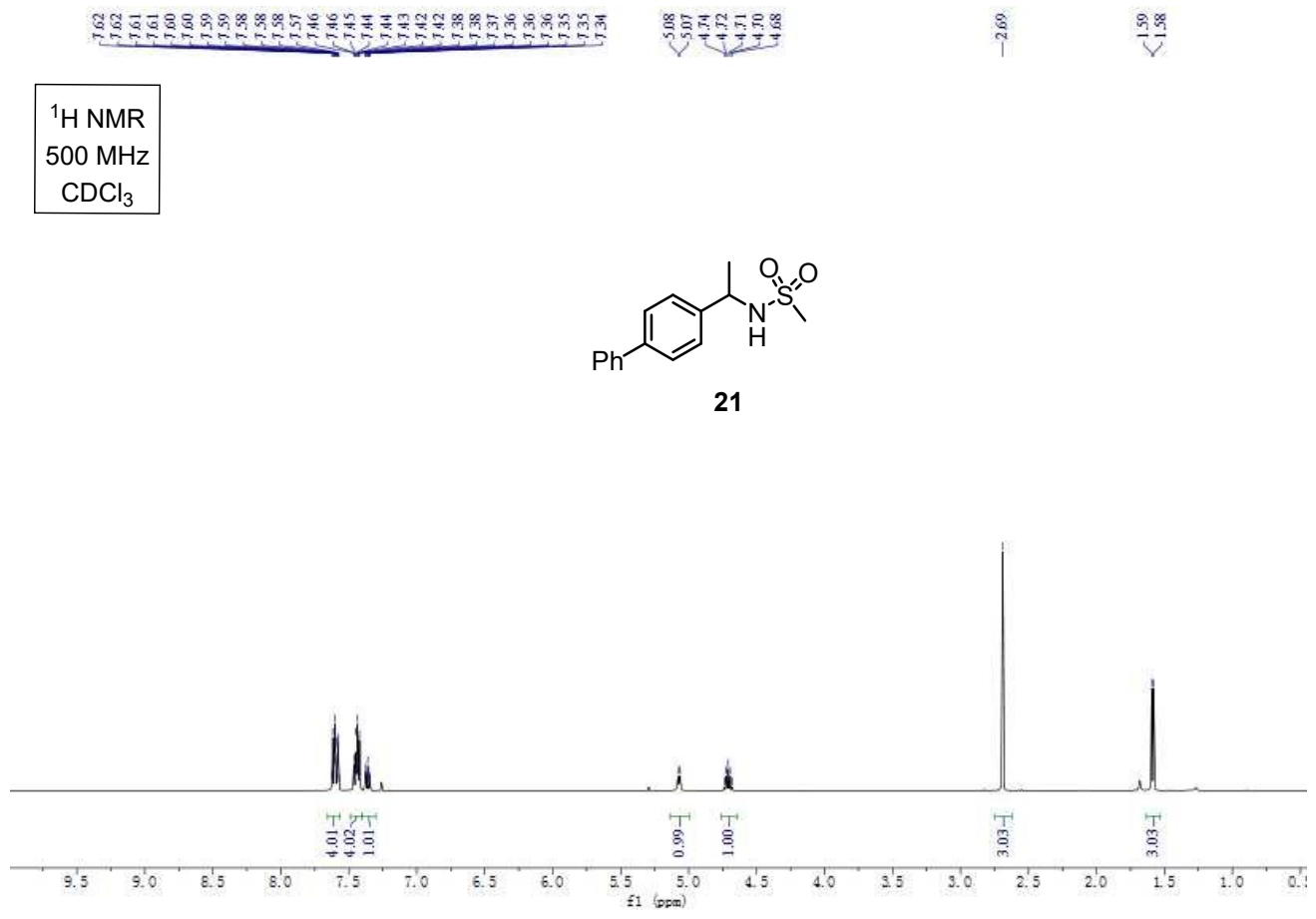
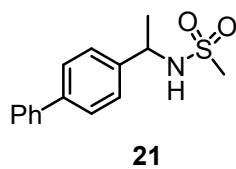


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

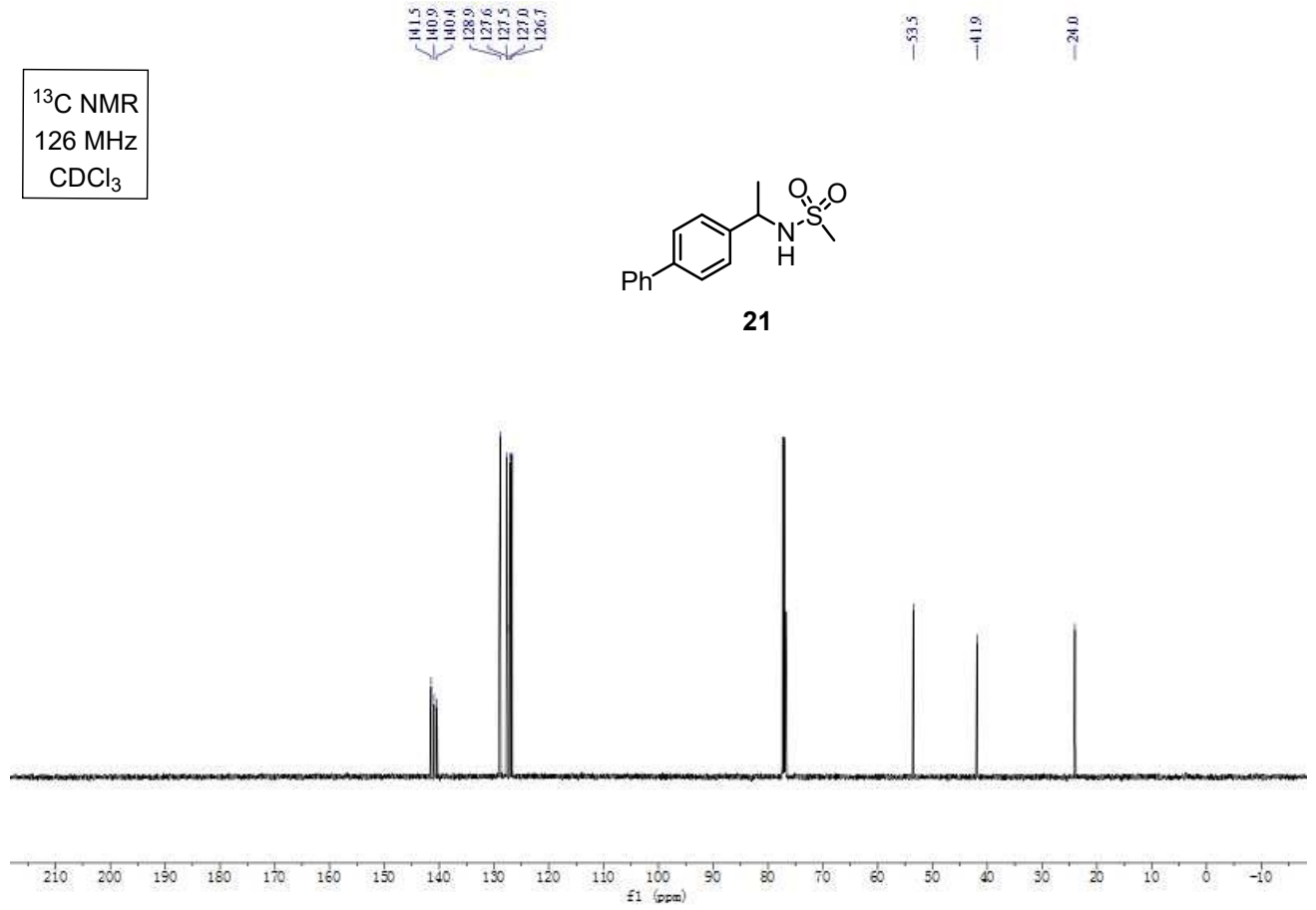
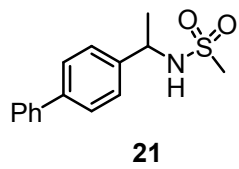




$^1\text{H}$  NMR  
500 MHz  
 $\text{CDCl}_3$



$^{13}\text{C}$  NMR  
126 MHz  
 $\text{CDCl}_3$



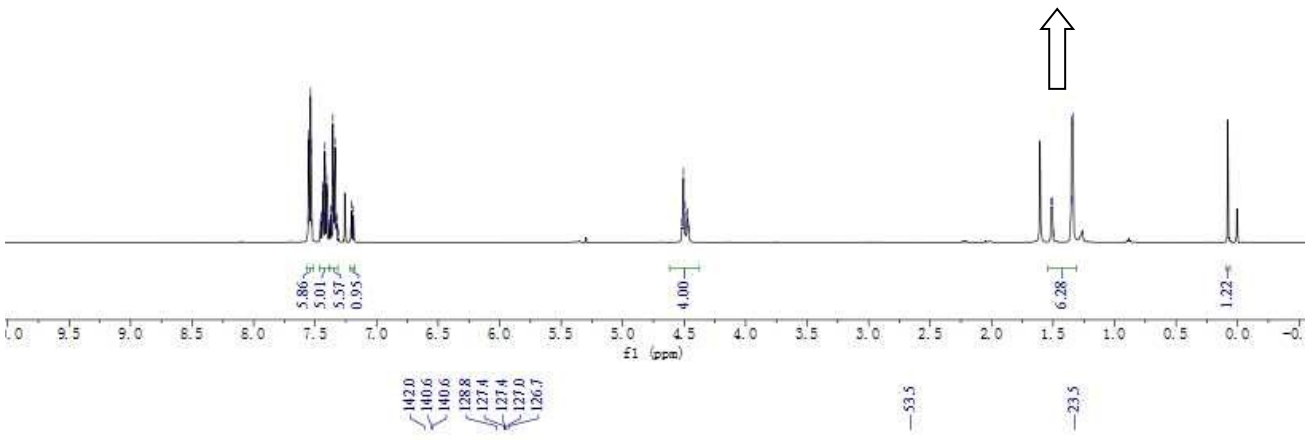
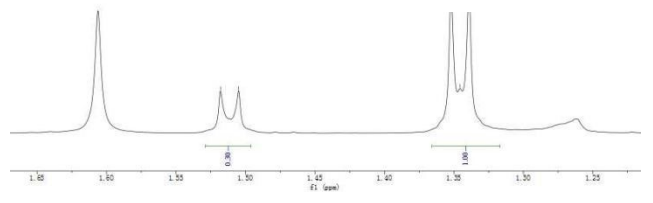
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4.47  
4.46  
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1.50  
1.35  
1.34

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

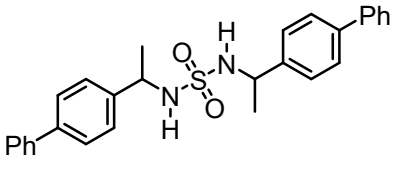


**22**

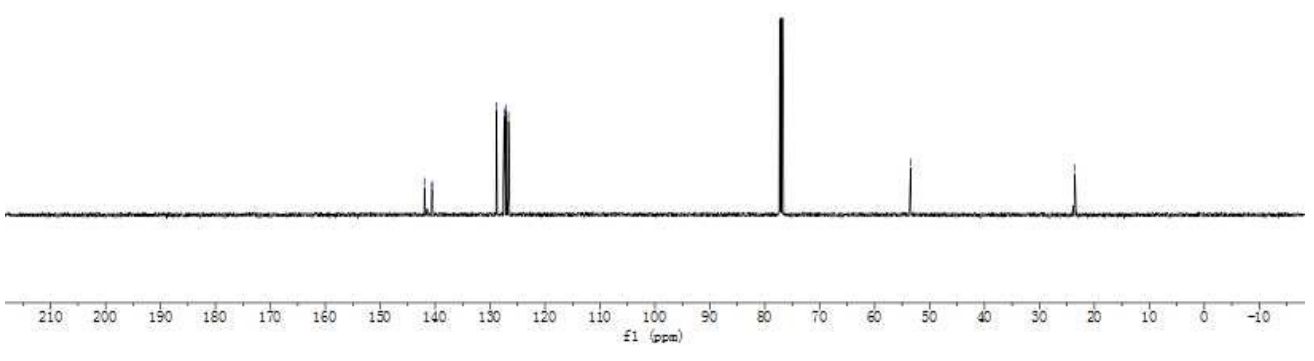
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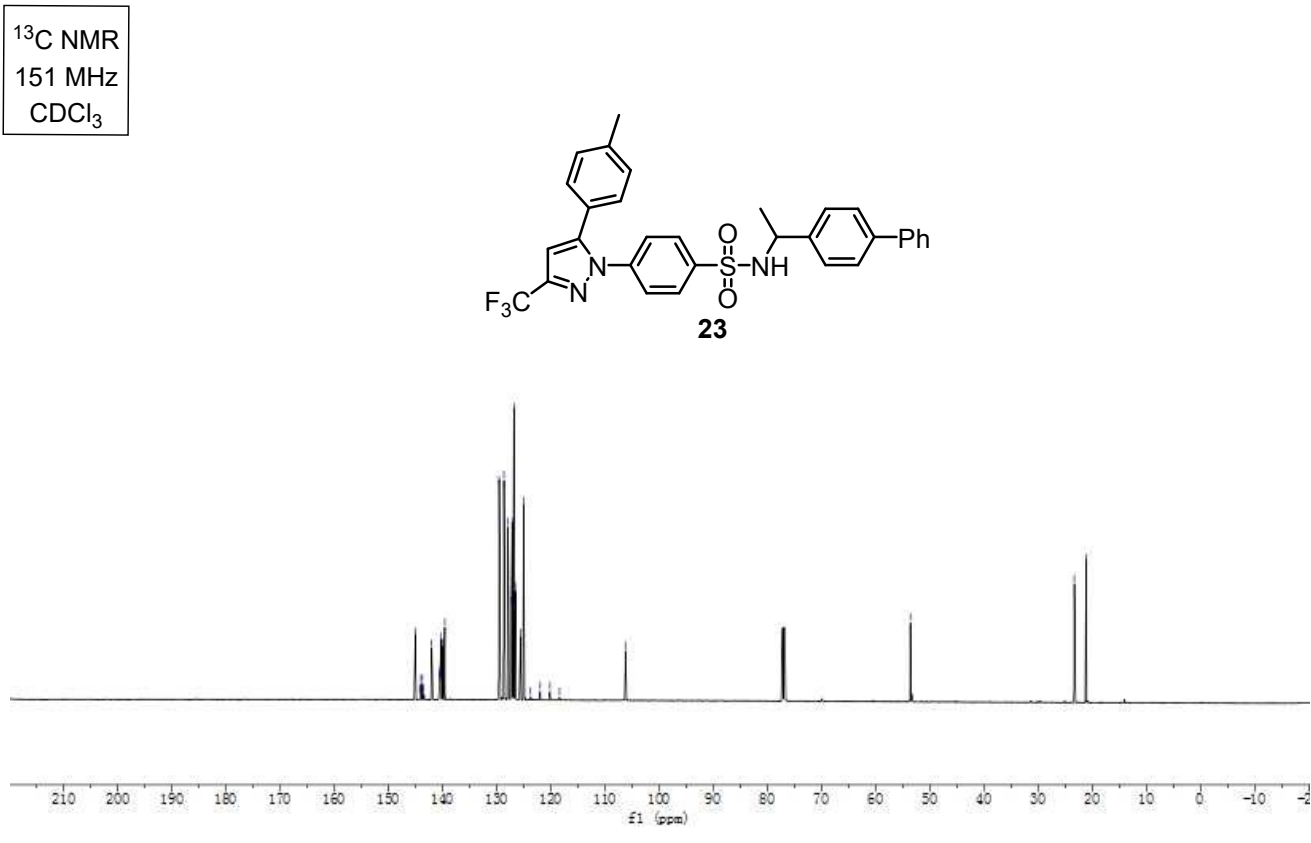
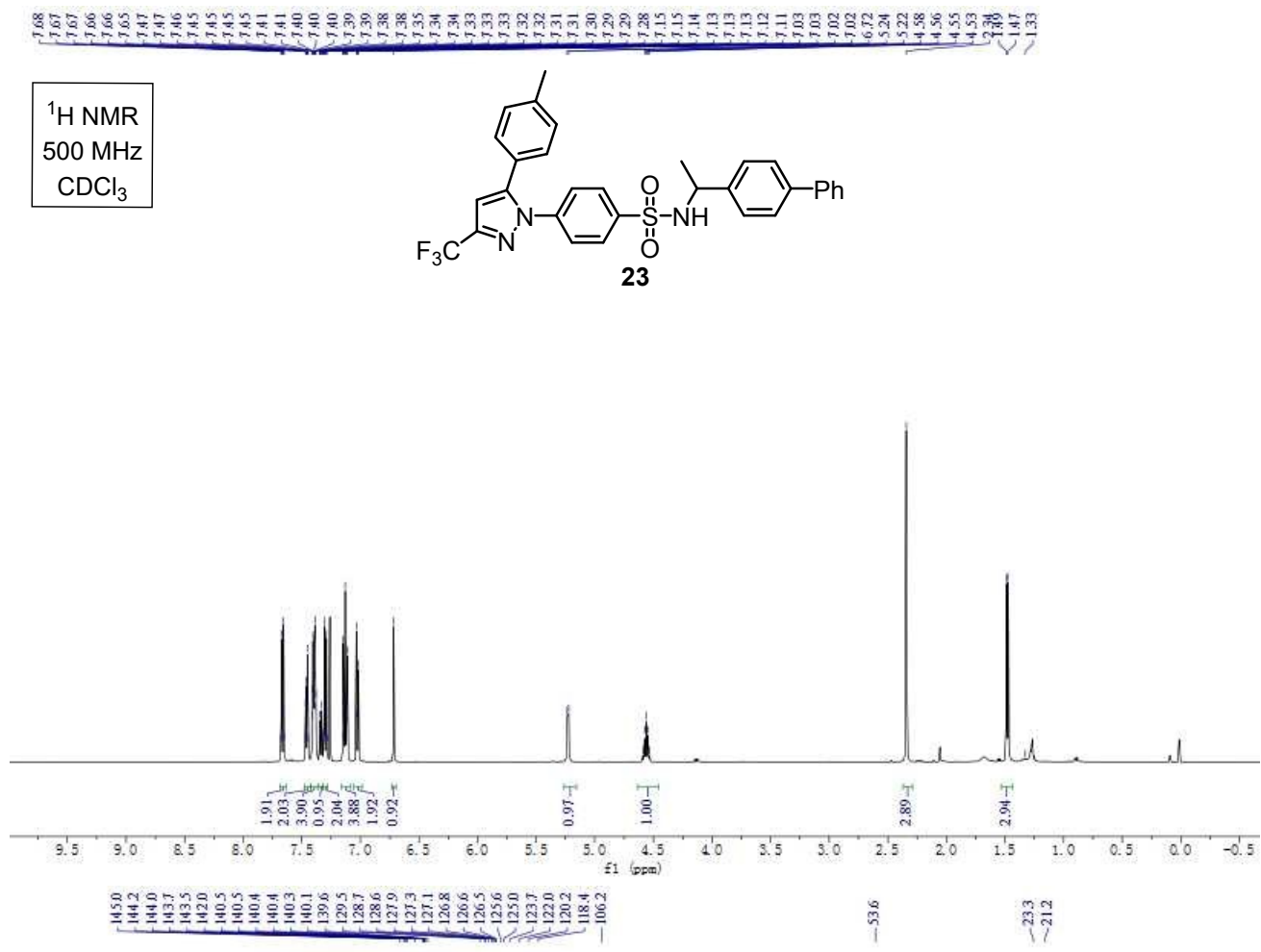


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



**22**

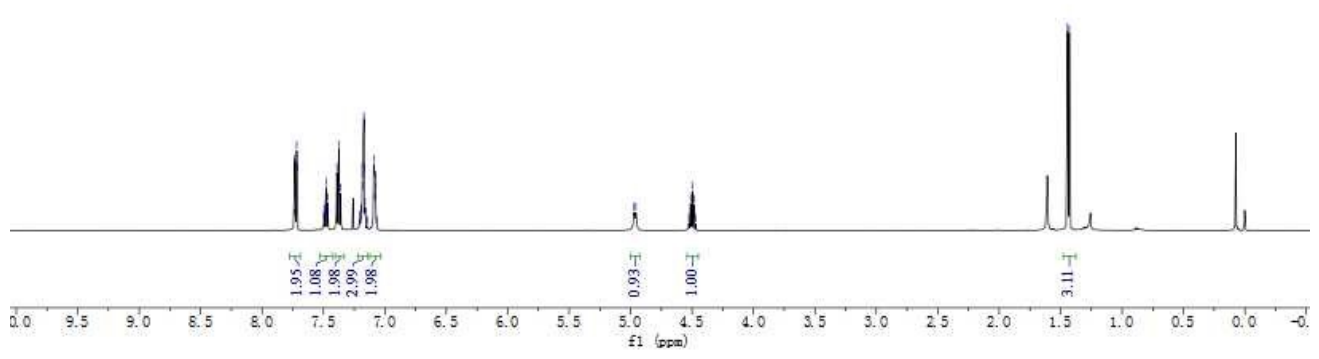
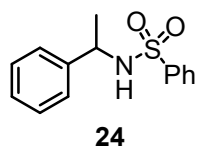




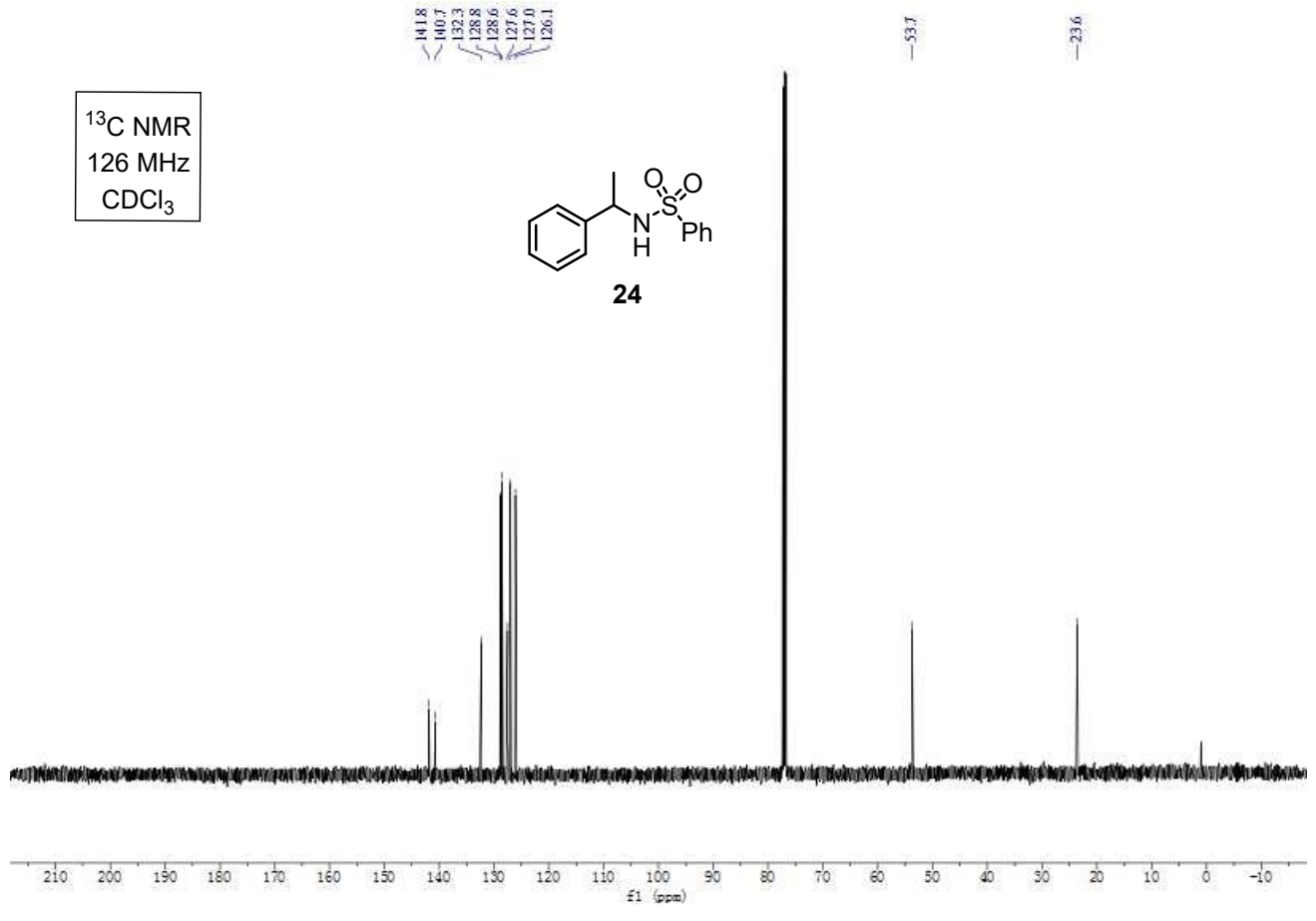
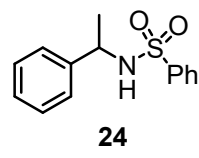
7.73  
7.73  
7.73  
7.72  
7.71  
7.49  
7.48  
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7.48  
7.48  
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7.38  
7.36  
7.36  
7.19  
7.19  
7.18  
7.18  
7.18  
7.17  
7.17  
7.17  
7.16  
7.16  
7.09  
7.09  
7.08  
7.08  
7.08  
7.07  
4.97  
4.96  
4.51  
4.50  
4.48  
4.47

144  
143

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



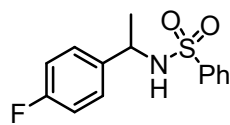
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



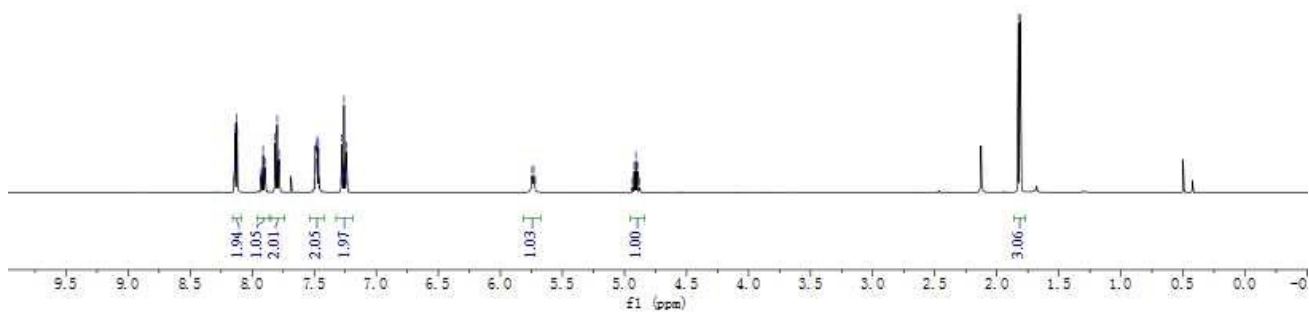
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8.13  
8.12  
8.12  
7.93  
7.93  
7.92  
7.91  
7.90  
7.90  
7.82  
7.82  
7.81  
7.80  
7.80  
7.80  
7.79  
7.78  
7.50  
7.49  
7.49  
7.48  
7.48  
7.47  
7.47  
7.46  
7.28  
7.28  
7.27  
7.26  
7.26 CDCl<sub>3</sub>  
7.25  
7.25  
7.24  
7.24  
5.75  
5.73  
5.72  
4.94  
4.92  
4.91  
4.89  
4.88

1.83  
1.81

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



25



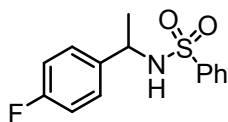
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161.0

140.6  
137.7  
137.7  
132.4  
128.8  
127.9  
127.8  
127.0  
115.4  
115.2

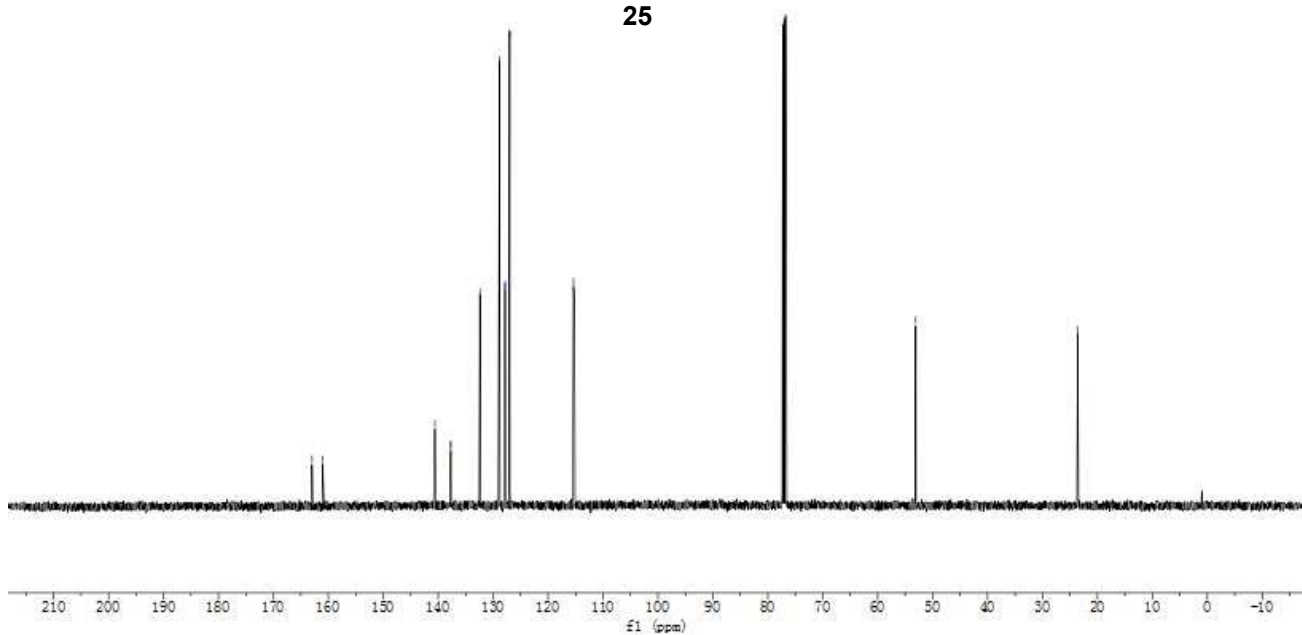
53.1

23.6

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



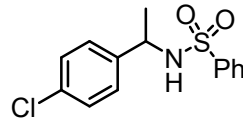
25



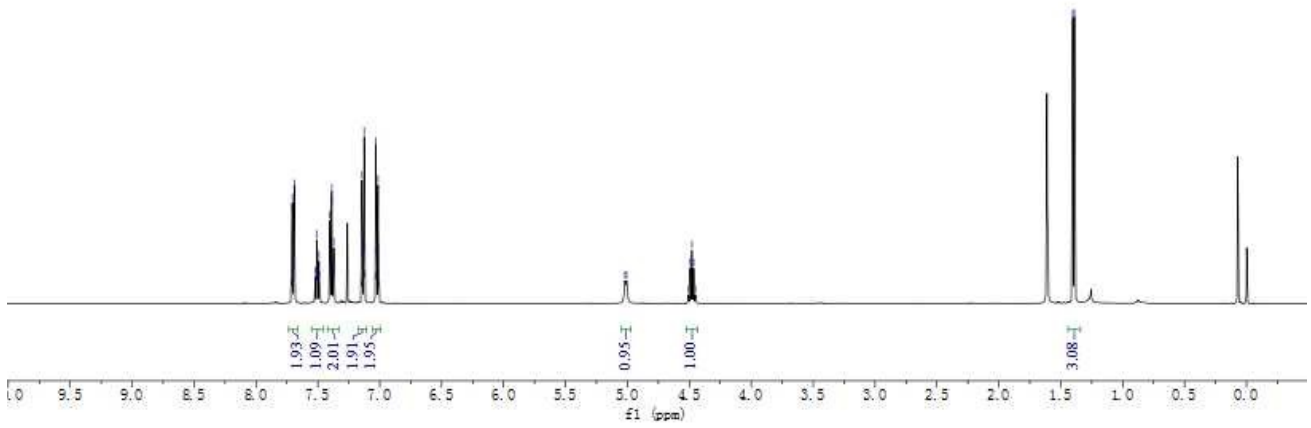
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7.52  
7.52  
7.51  
7.51  
7.50  
7.49  
7.49  
7.40  
7.40  
7.39  
7.39  
7.38  
7.38  
7.37  
7.14  
7.14  
7.13  
7.13  
7.12  
7.04  
7.03  
7.03  
7.02  
5.02  
5.01  
4.51  
4.49  
4.48  
4.46  
4.45

1.40  
1.39

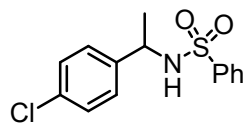
$^1\text{H}$  NMR  
500 MHz  
 $\text{CDCl}_3$



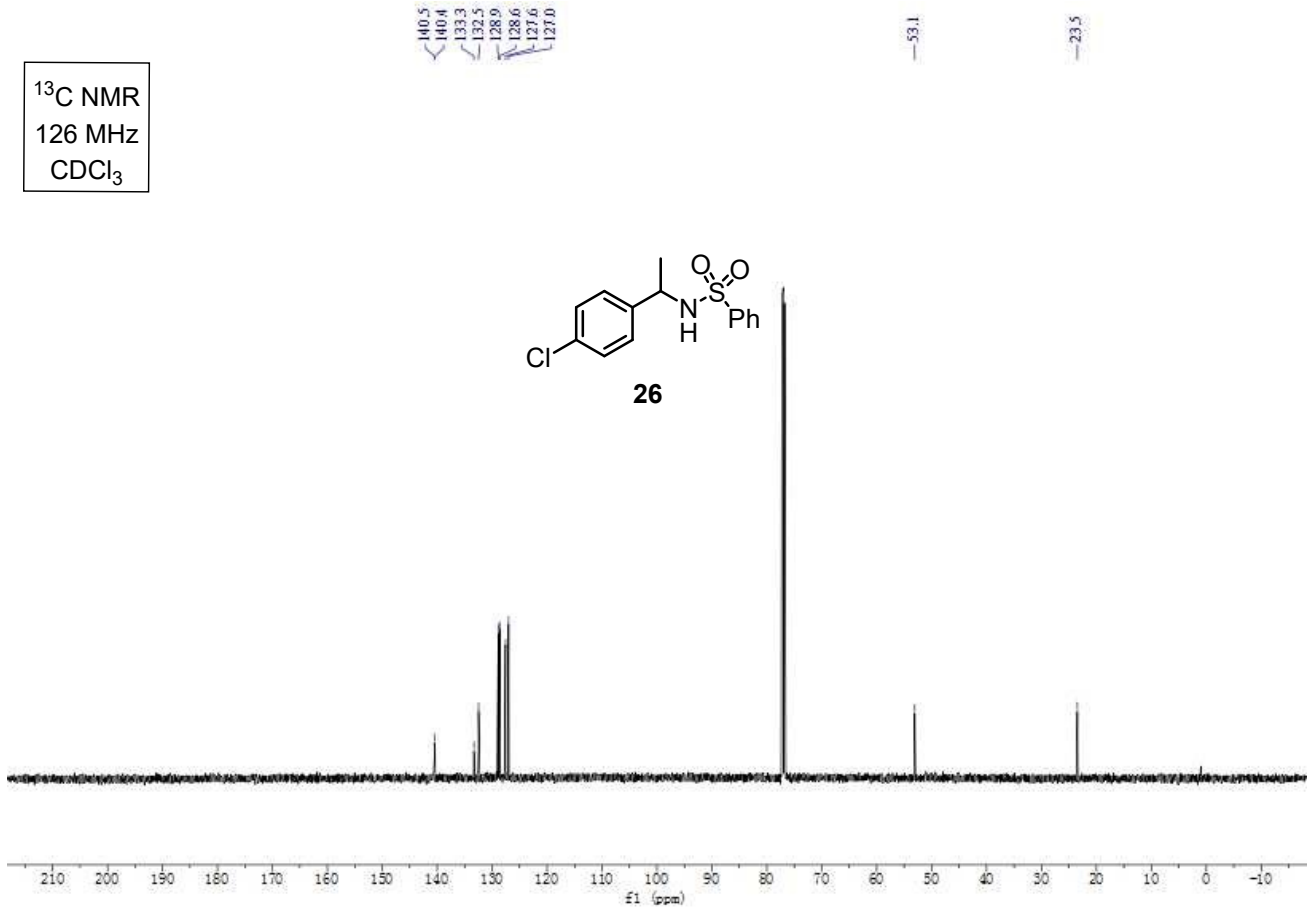
26

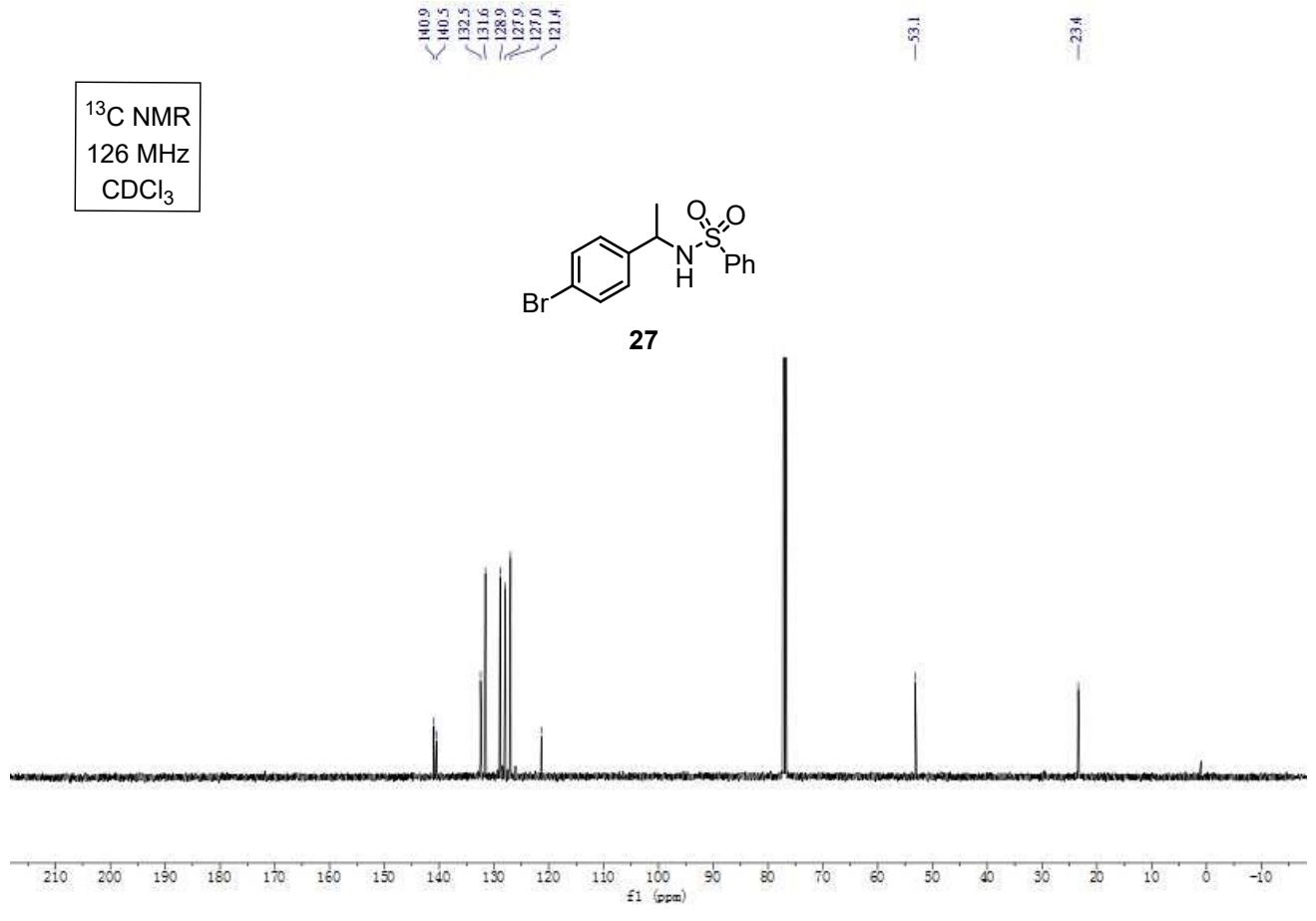
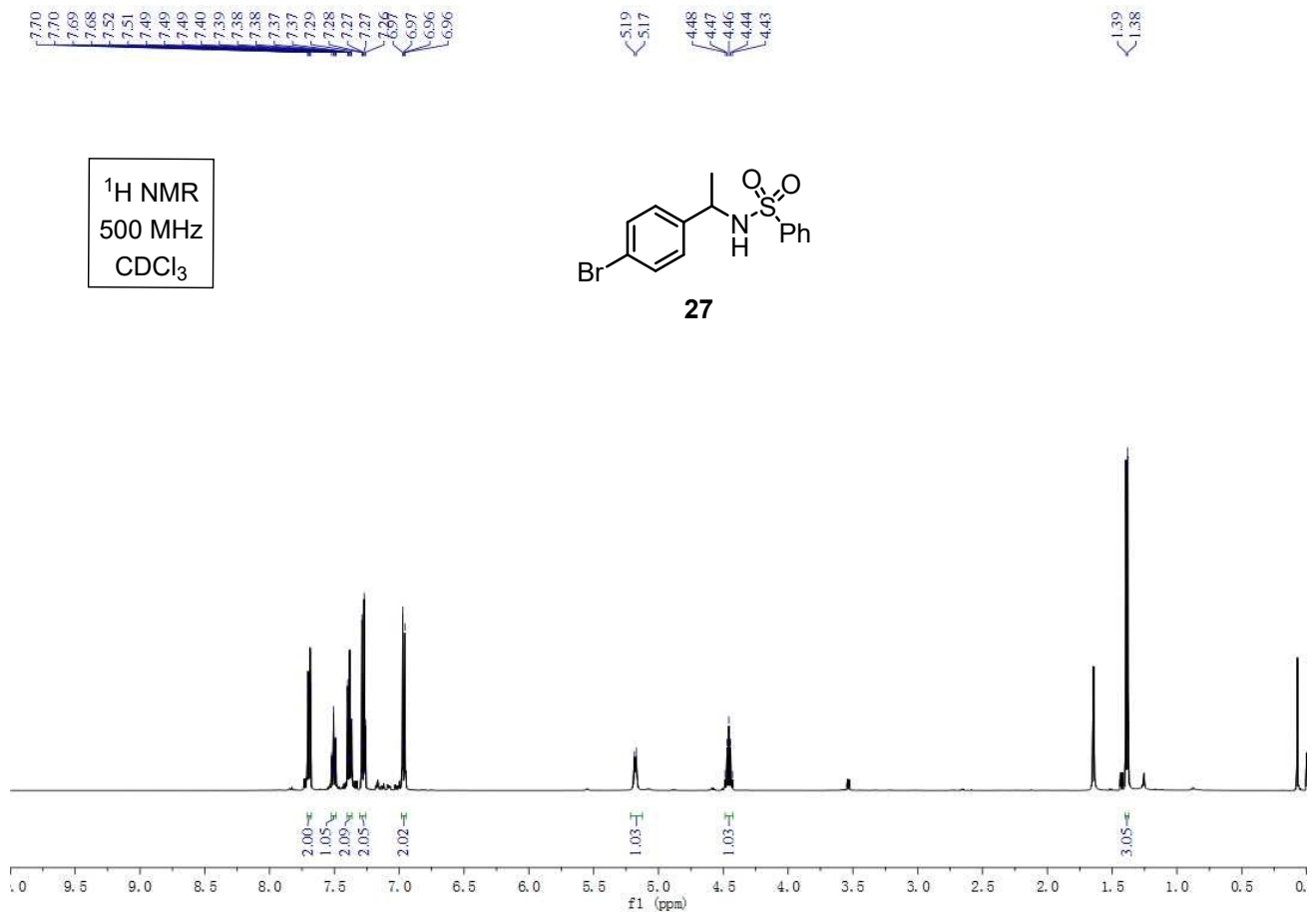


$^{13}\text{C}$  NMR  
126 MHz  
 $\text{CDCl}_3$



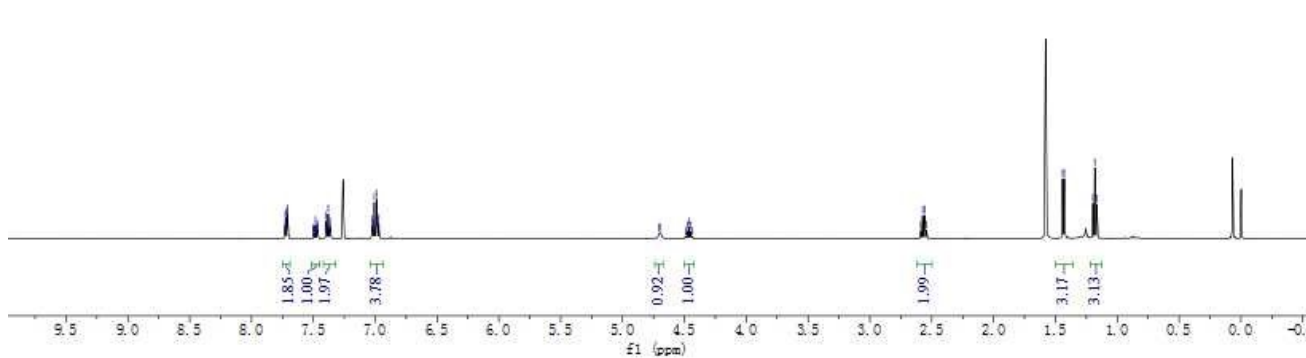
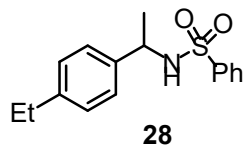
26





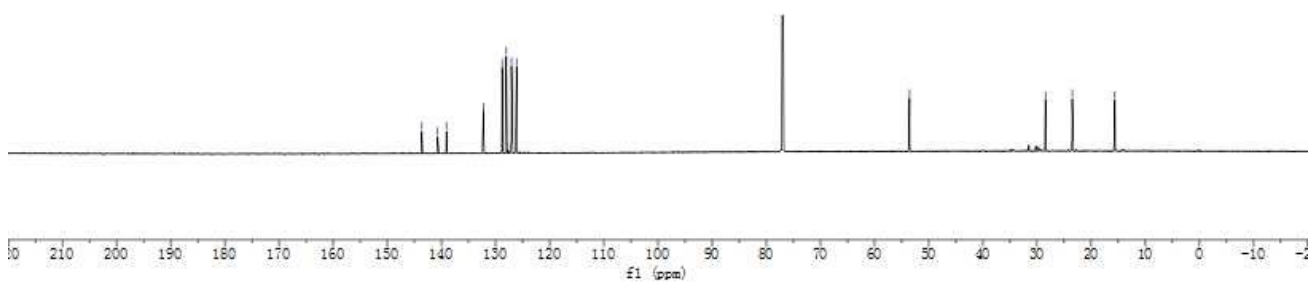
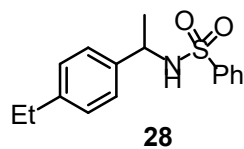
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<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

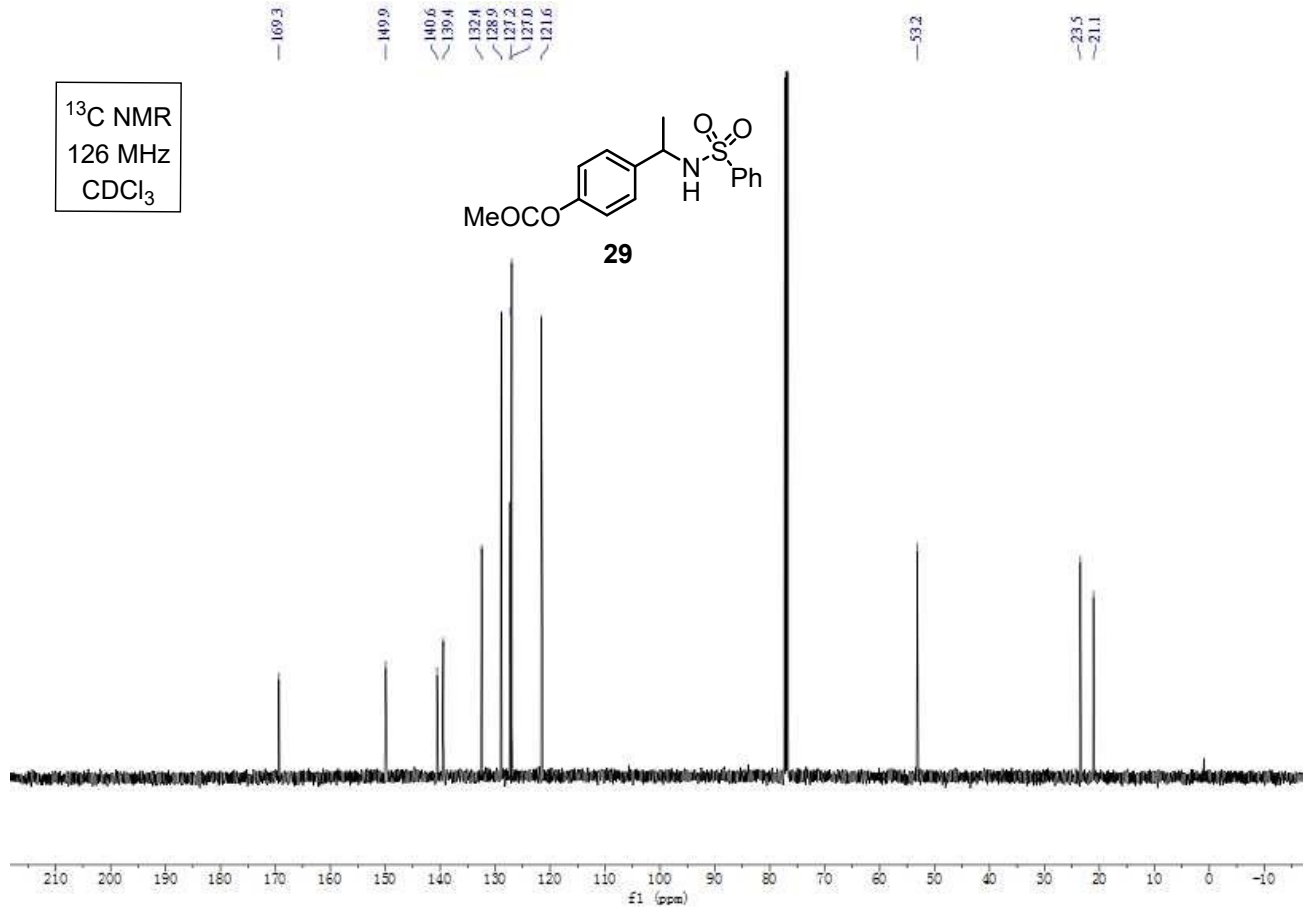
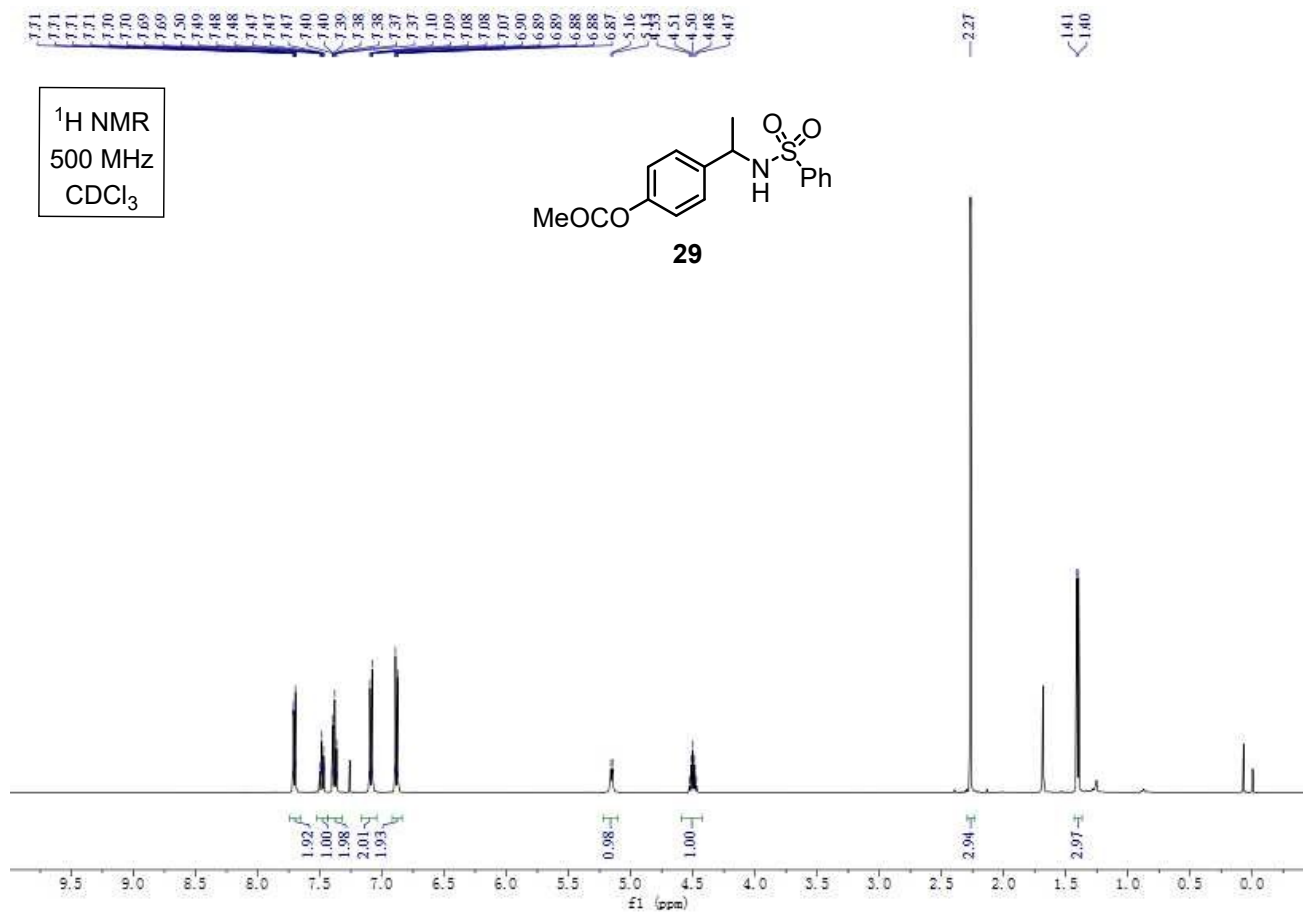


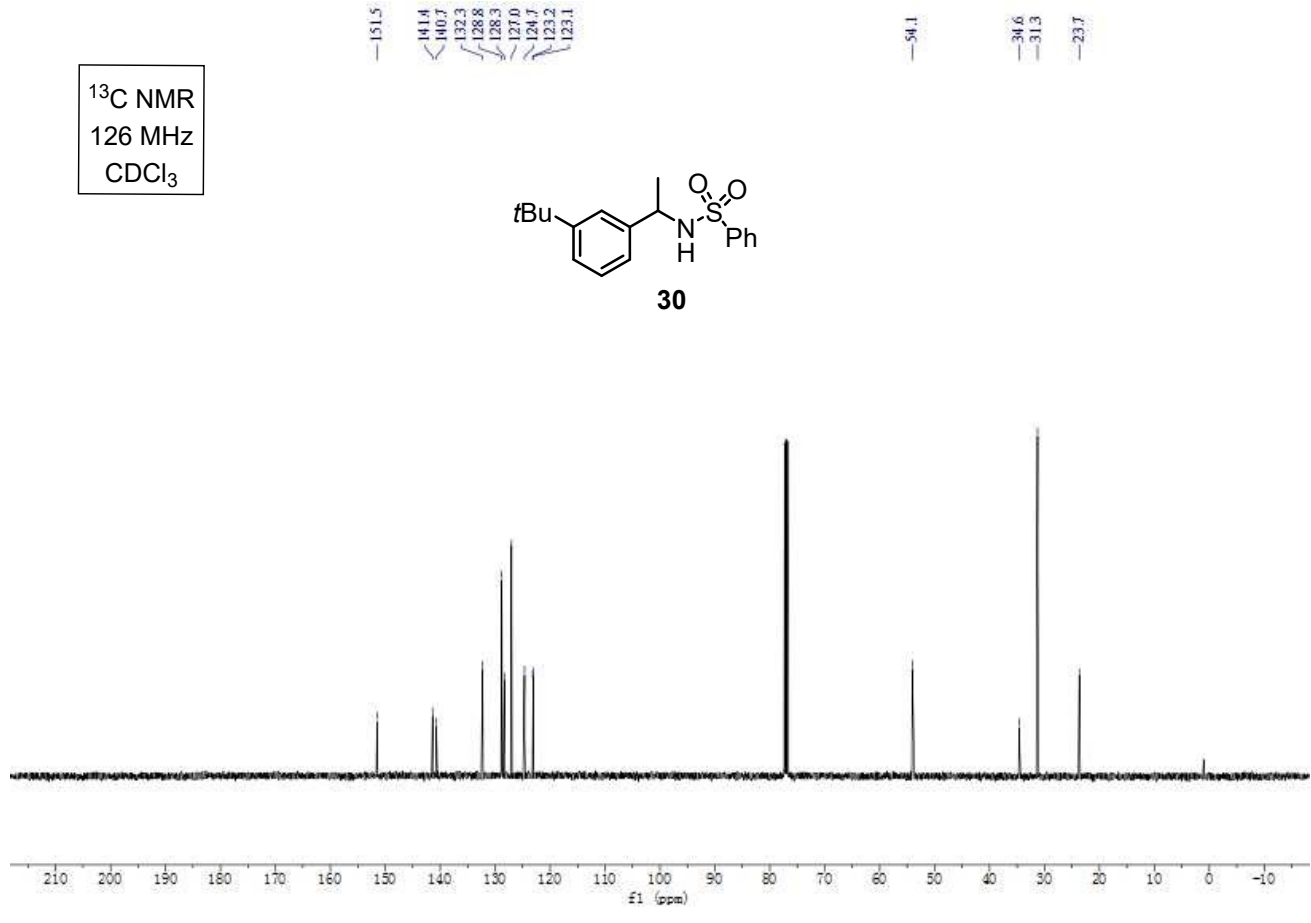
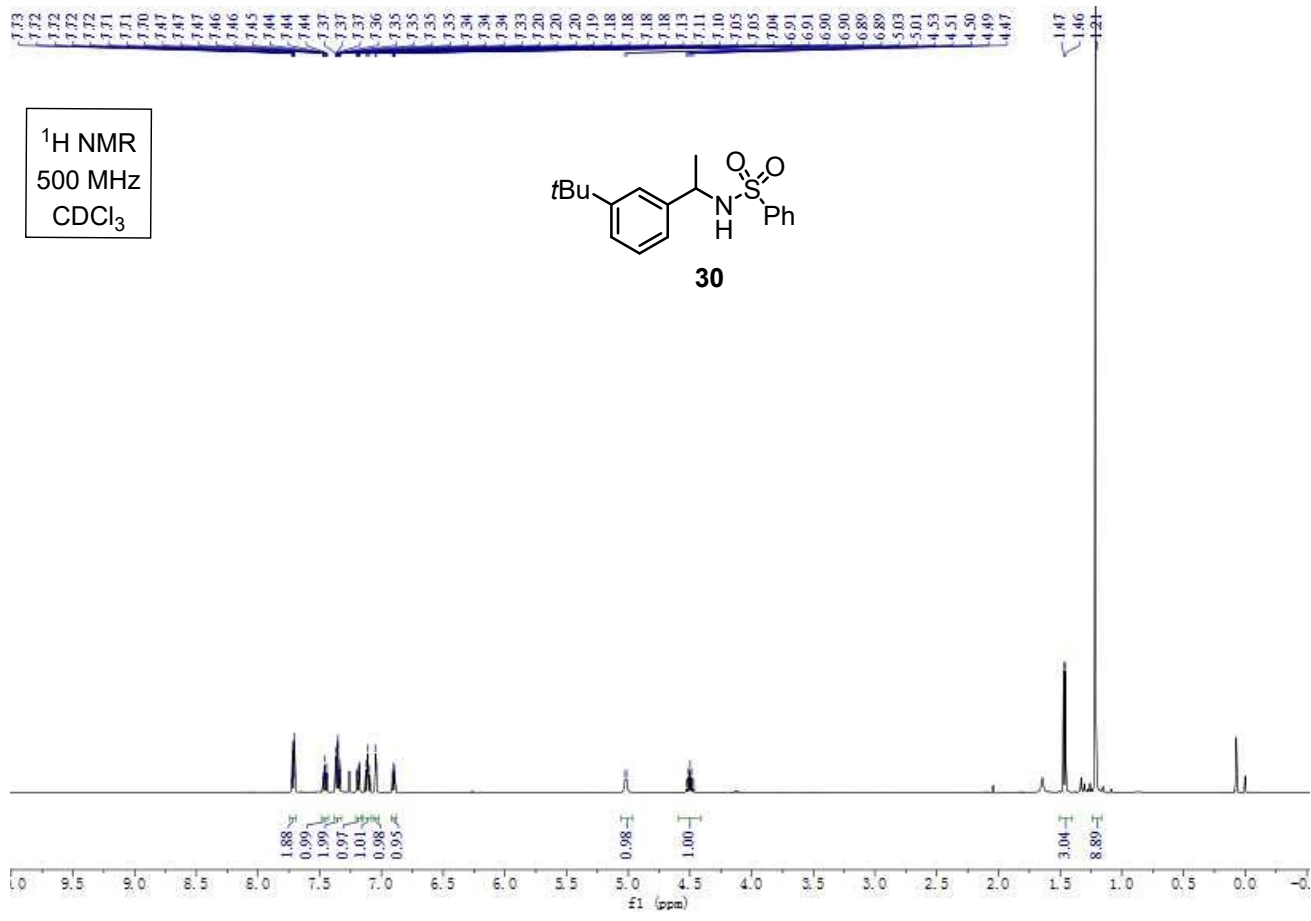
143.6 140.7 139.1 132.3 128.8 128.0 127.1 126.1

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>









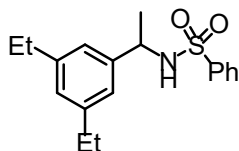
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7.72  
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7.46  
7.46  
7.45  
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7.38  
7.38  
7.38  
7.36  
7.36  
7.35  
7.35  
6.83  
6.82  
6.68  
6.68

4.88  
4.87  
4.48  
4.47  
4.45  
4.44  
4.42

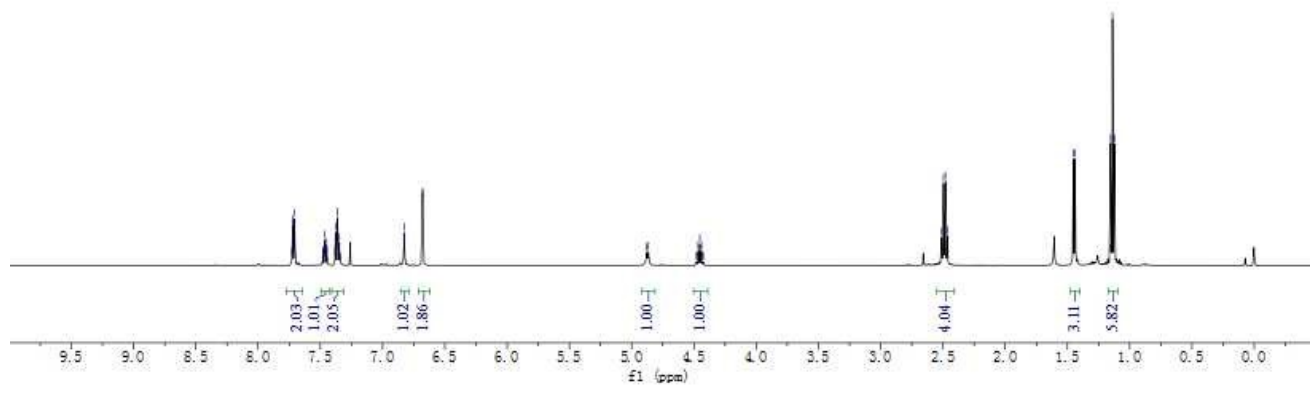
2.51  
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2.48  
2.47

1.45  
1.44  
1.16  
1.15  
1.14  
1.12

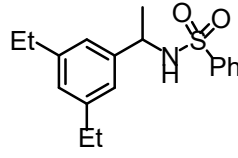
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



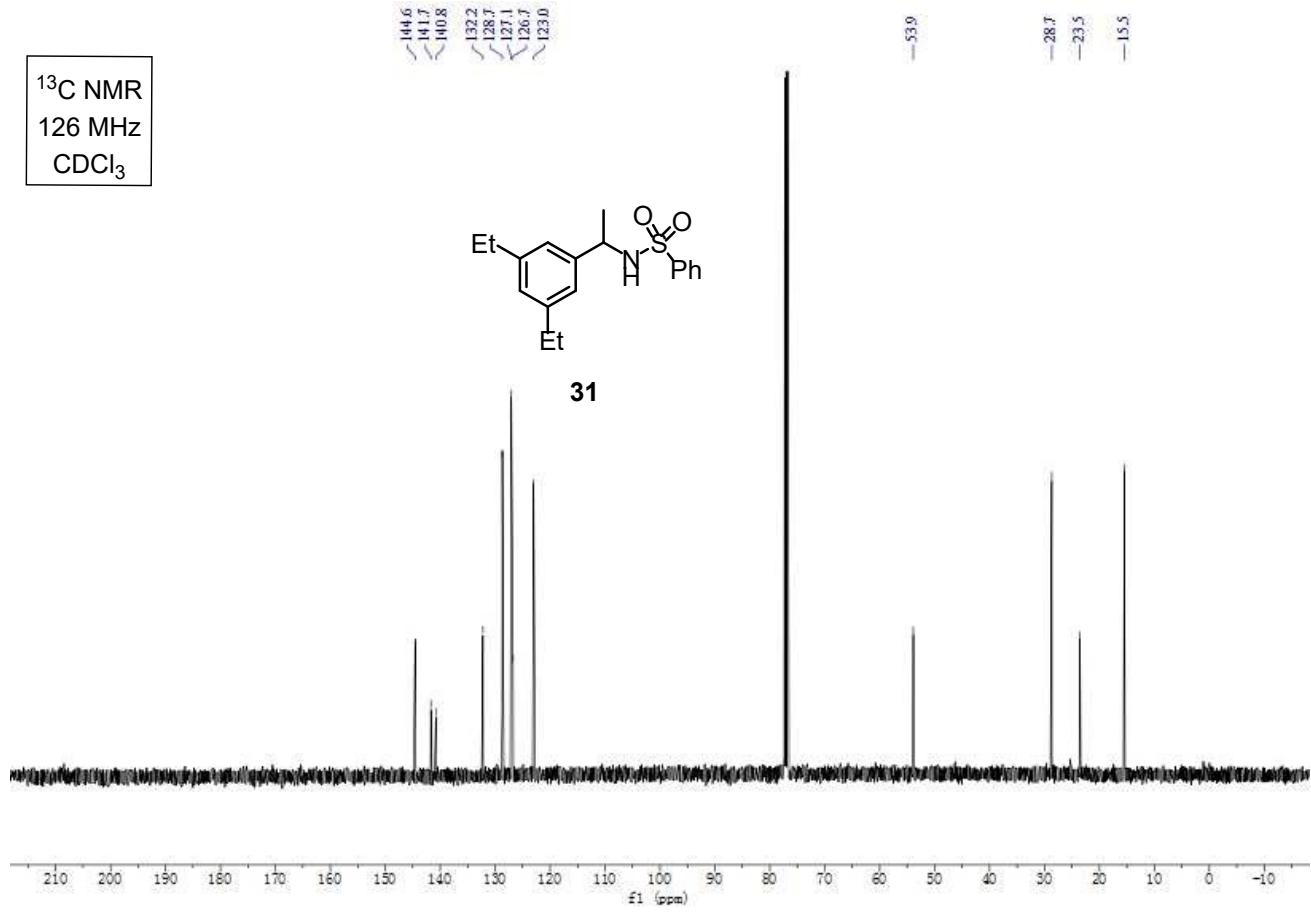
**31**



<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



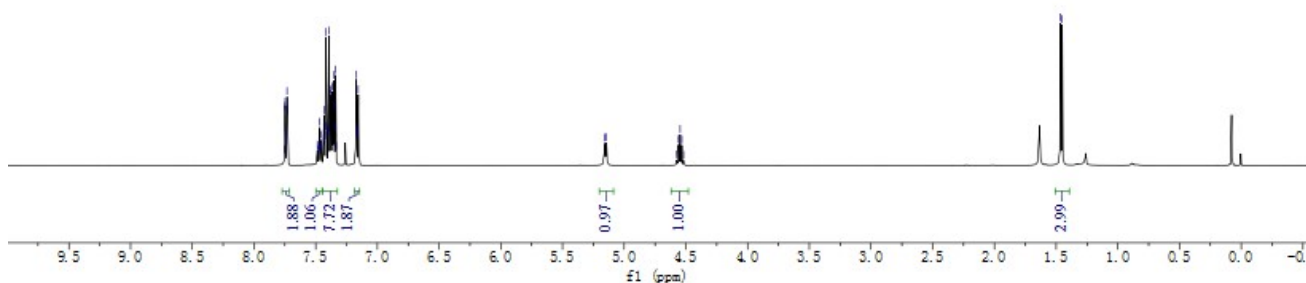
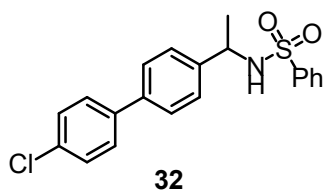
**31**



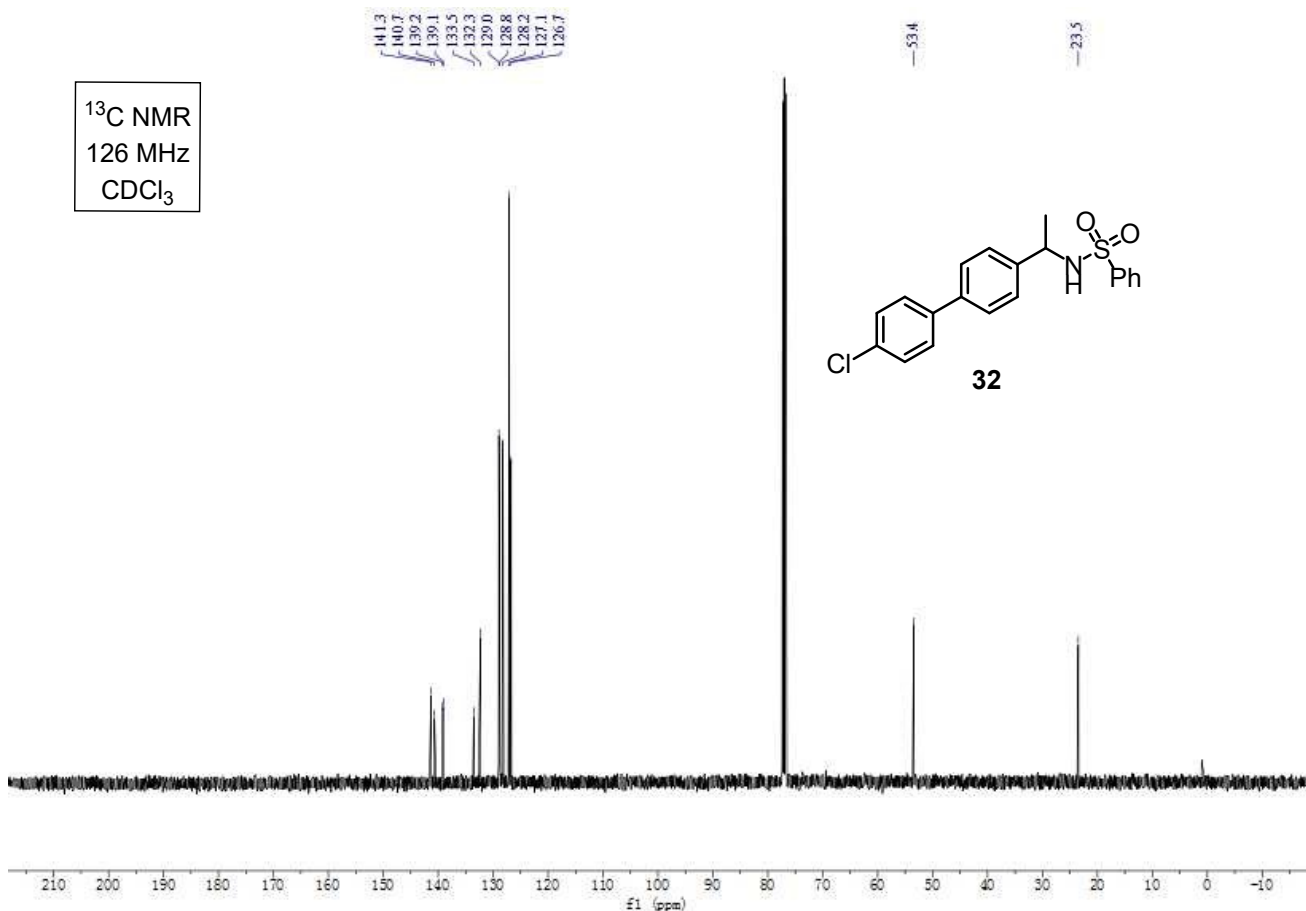
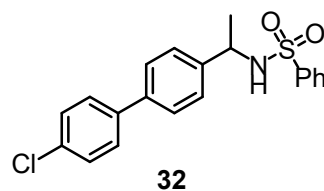
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7.49  
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7.46  
7.46  
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7.43  
7.43  
7.42  
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7.36  
7.35  
7.34  
7.34  
7.33  
7.33  
7.17  
7.17  
7.16  
5.16  
5.14  
4.56  
4.55  
4.53  
4.52

1.46  
1.45

$^1\text{H}$  NMR  
500 MHz  
 $\text{CDCl}_3$



$^{13}\text{C}$  NMR  
126 MHz  
 $\text{CDCl}_3$

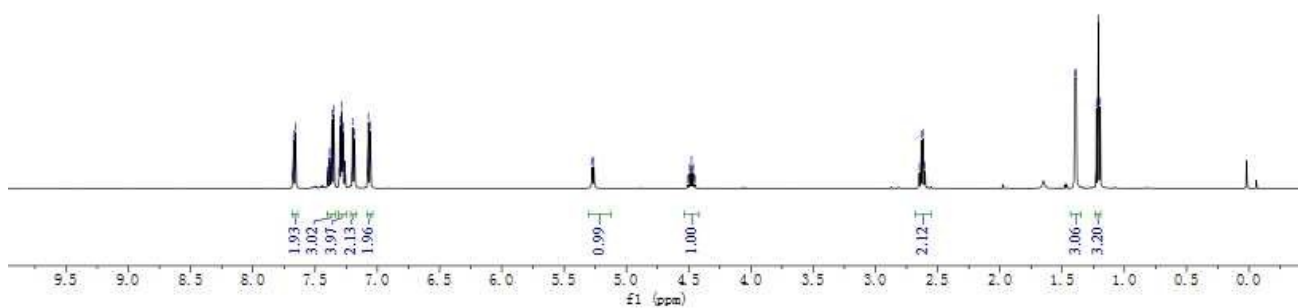
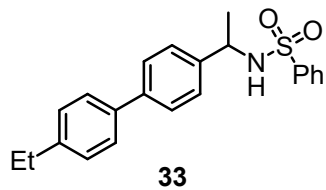


7.67  
7.67  
7.67  
7.66  
7.66  
7.66  
7.38  
7.37  
7.37  
7.37  
7.35  
7.35  
7.35  
7.30  
7.30  
7.29  
7.29  
7.28  
7.28  
7.28  
7.27  
7.27  
7.20  
7.20  
7.19  
7.18  
7.18  
7.07  
7.07  
7.06  
7.06  
5.28  
4.51  
4.51  
4.49  
4.48  
4.46  
4.45

2.65  
2.63  
2.62  
2.60

1.40  
1.39  
1.23  
1.21  
1.20  
1.19

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



143.5  
140.7  
140.6  
140.4  
138.0  
132.3  
128.8  
128.3  
127.1  
127.1  
126.9  
126.6

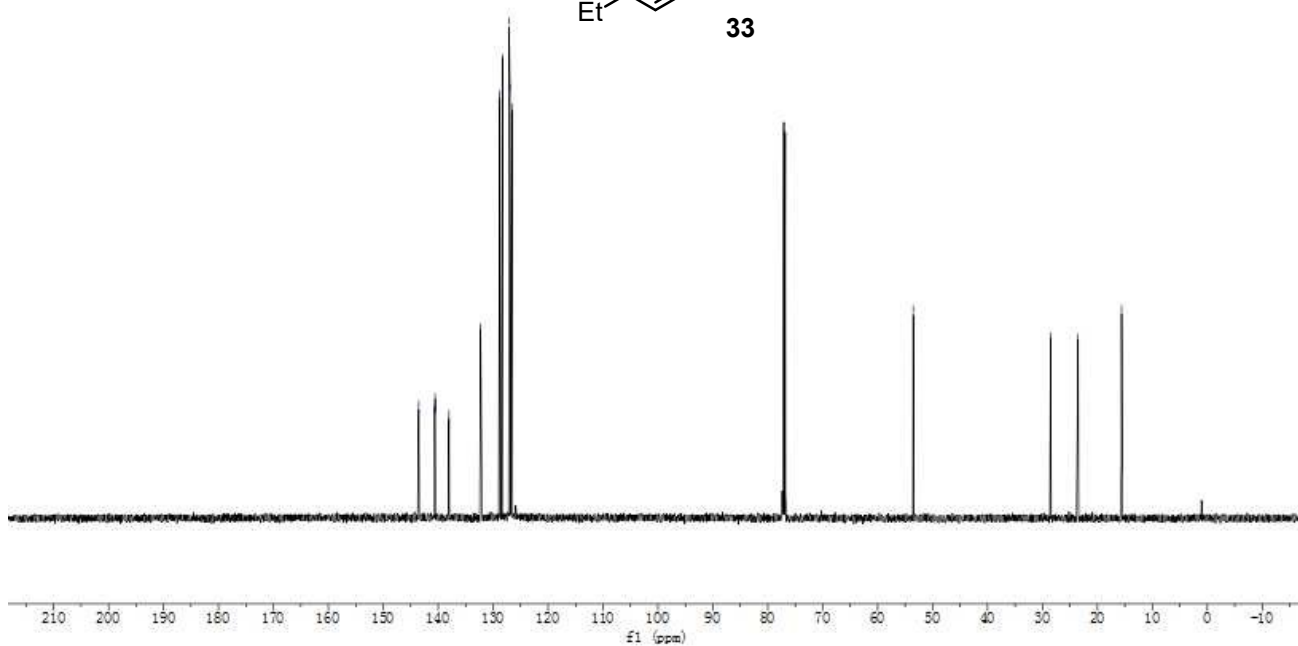
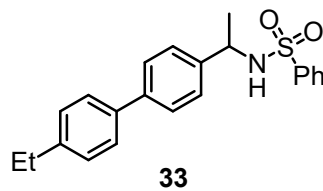
-53.5

-28.5

-23.5

-15.6

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

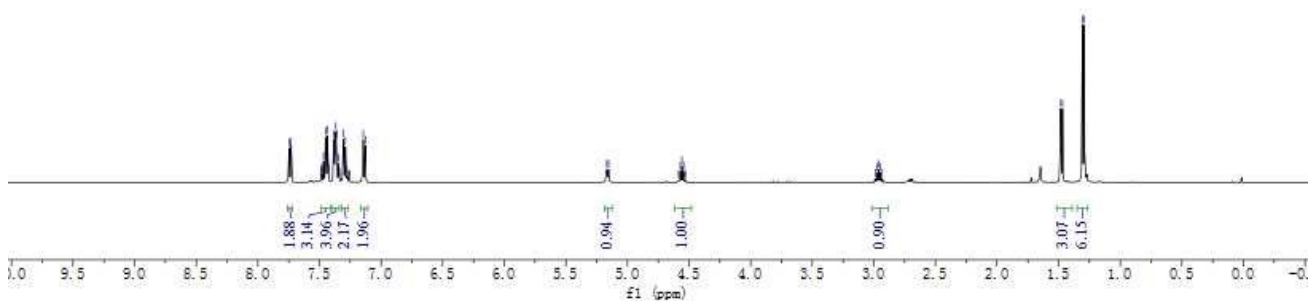
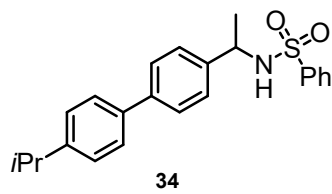


7.75  
7.74  
7.73  
7.73  
7.48  
7.48  
7.48  
7.47  
7.45  
7.44  
7.43  
7.38  
7.38  
7.37  
7.37  
7.36  
7.35  
7.34  
7.34  
7.31  
7.30  
7.29  
7.29  
7.28  
7.14  
7.13  
7.13  
5.17  
5.15  
4.58  
4.57  
4.56  
4.54  
4.53

2.99  
2.97  
2.96  
2.95  
2.93

1.48  
1.47  
1.31  
1.29

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



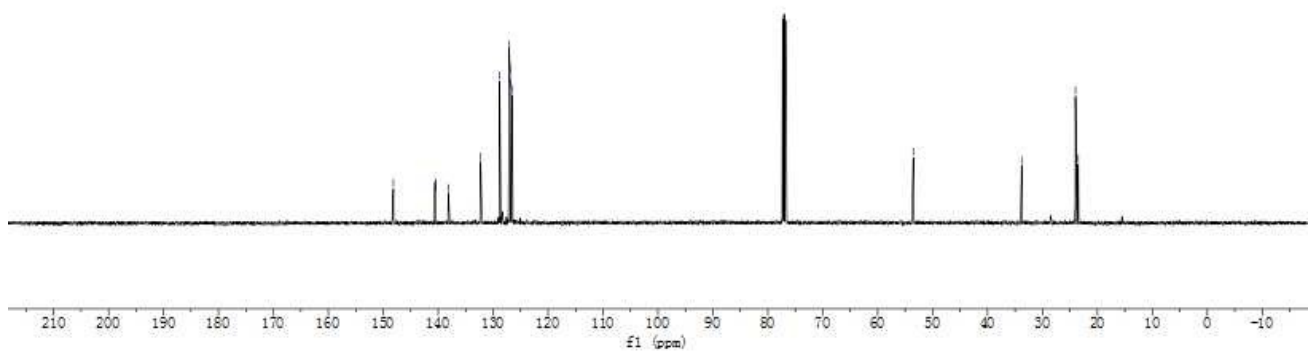
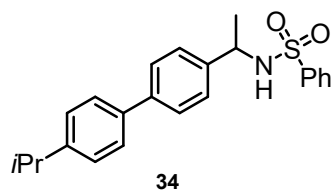
148.2  
140.7  
140.5  
140.5  
138.1  
132.3  
128.8  
127.1  
127.1  
126.9  
126.9  
126.6

53.5

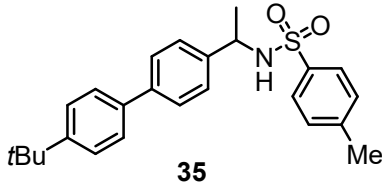
33.8

24.0  
23.5

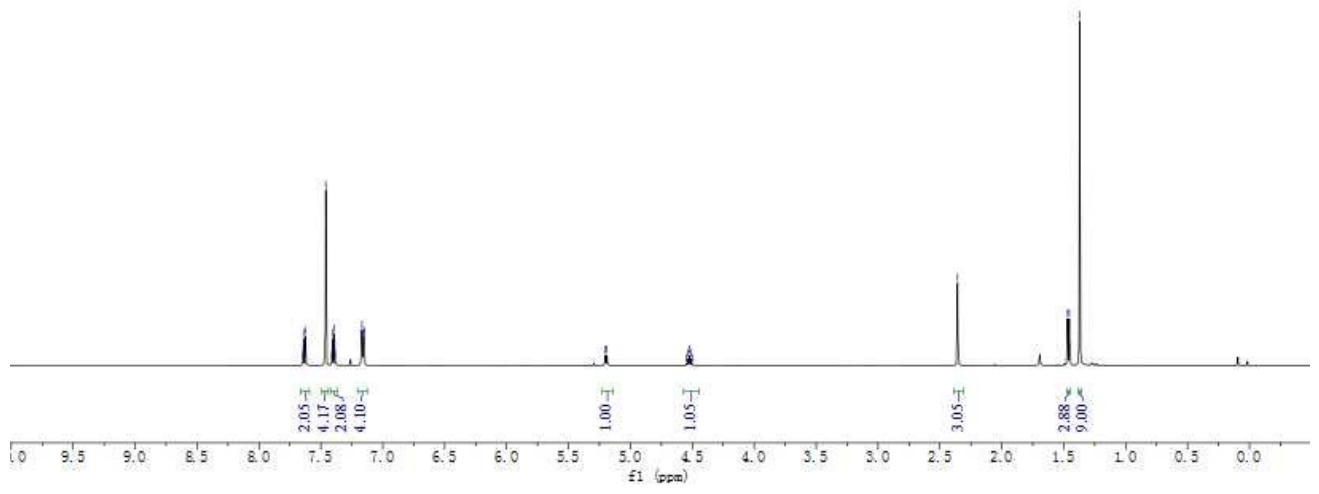
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



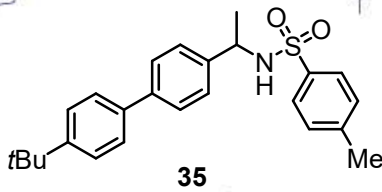
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



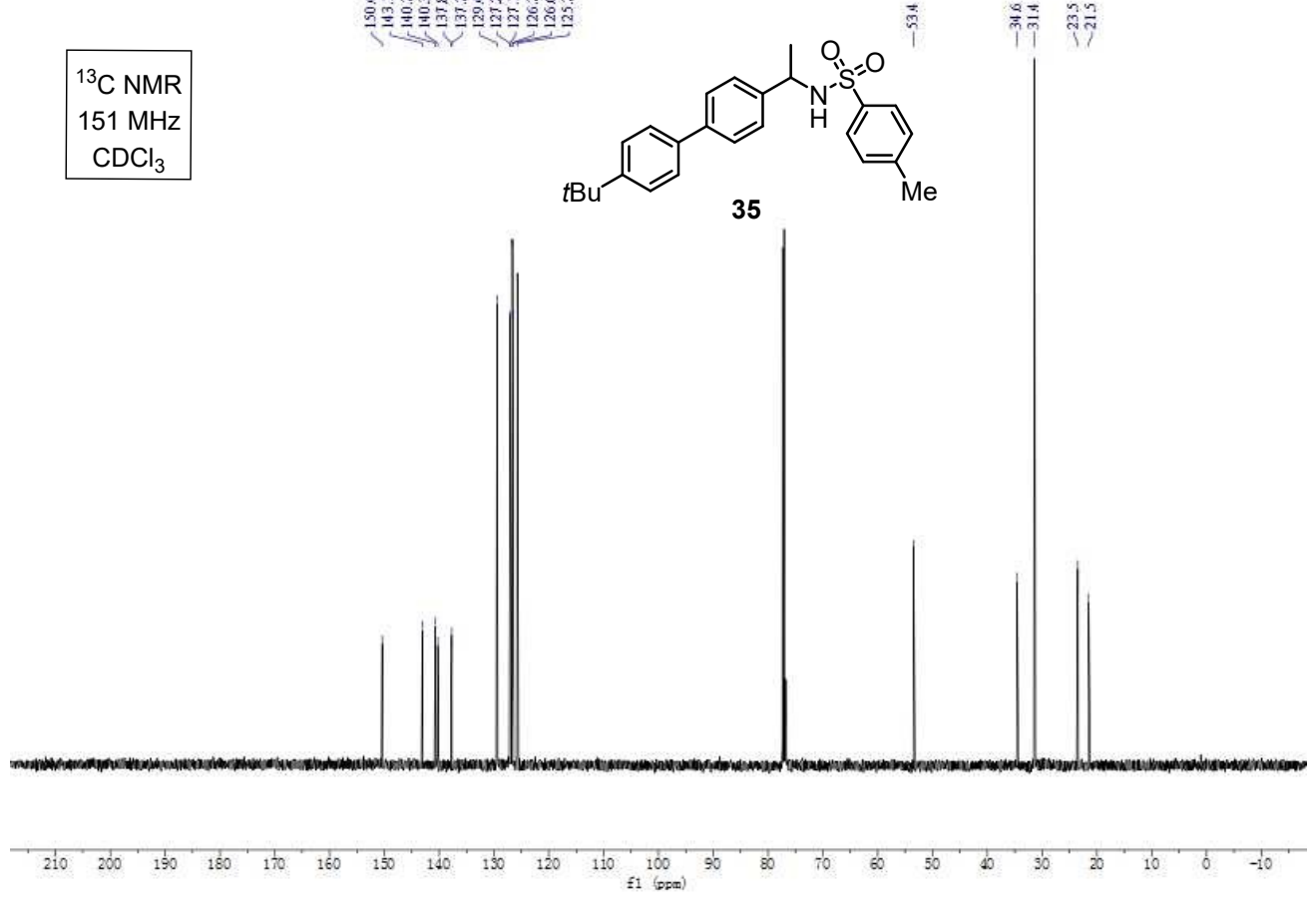
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<sup>13</sup>C NMR  
151 MHz  
CDCl<sub>3</sub>



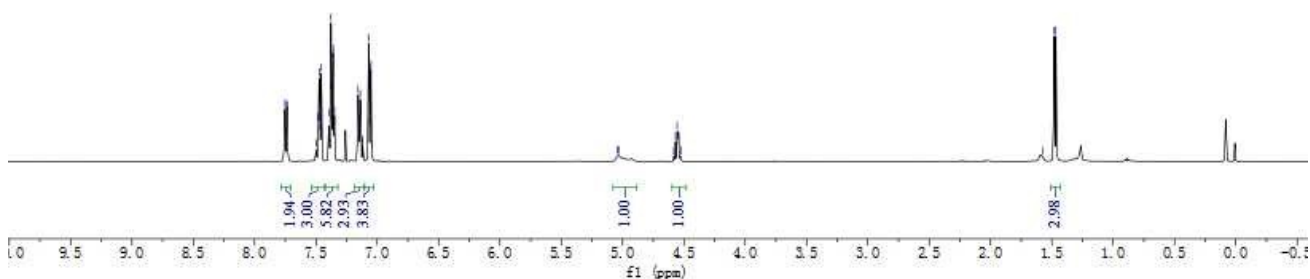
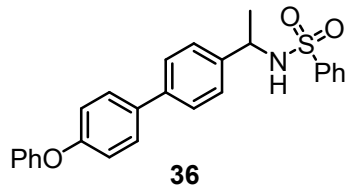
150.4, 143.1, 140.7, 140.3, 137.8, 137.7, 137.7, 129.4, 127.2, 127.1, 126.7, 126.6, 125.7, 53.4, 34.6, 31.4, 23.5, 21.5



7.75  
7.74  
7.74  
7.50  
7.48  
7.47  
7.47  
7.46  
7.46  
7.39  
7.38  
7.37  
7.36  
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7.14  
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7.05  
7.05  
7.04  
7.04  
5.04  
5.03  
4.58  
4.57  
4.55  
4.54  
4.53

1.57 H<sub>2</sub>O  
1.48  
1.47

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

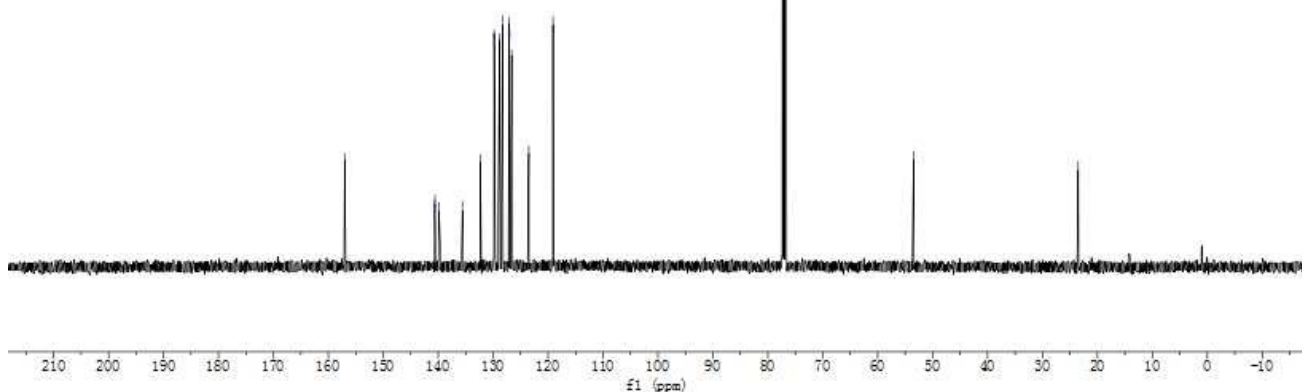
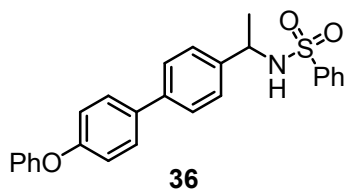


157.0  
140.7  
140.6  
139.8  
135.6  
132.3  
129.8  
128.8  
128.3  
127.1  
127.0  
126.6  
123.5  
119.1  
119.0

53.5

23.5

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

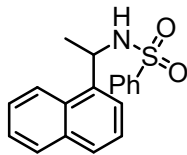




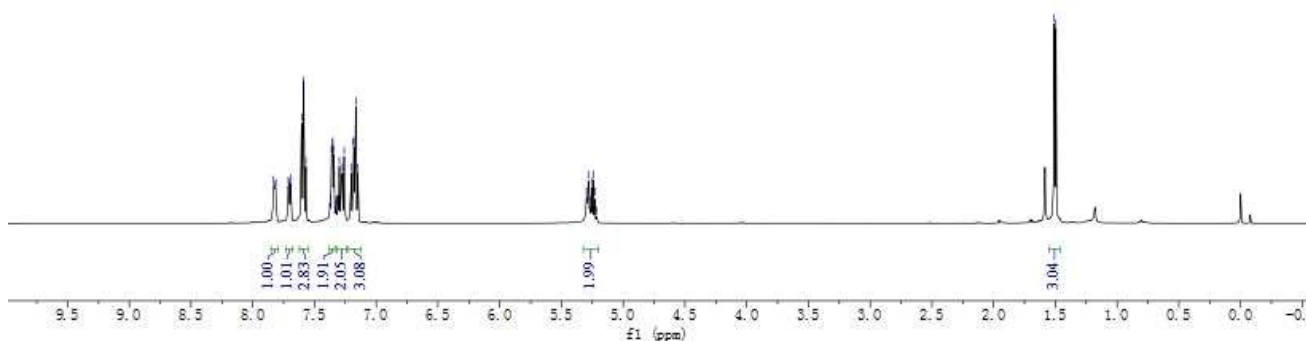
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5.30  
5.28  
5.27  
5.26  
5.24  
5.23  
5.22

1.51  
1.50

$^1\text{H}$  NMR  
500 MHz  
 $\text{CDCl}_3$



37

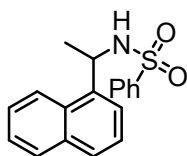


$^{13}\text{C}$  NMR  
126 MHz  
 $\text{CDCl}_3$

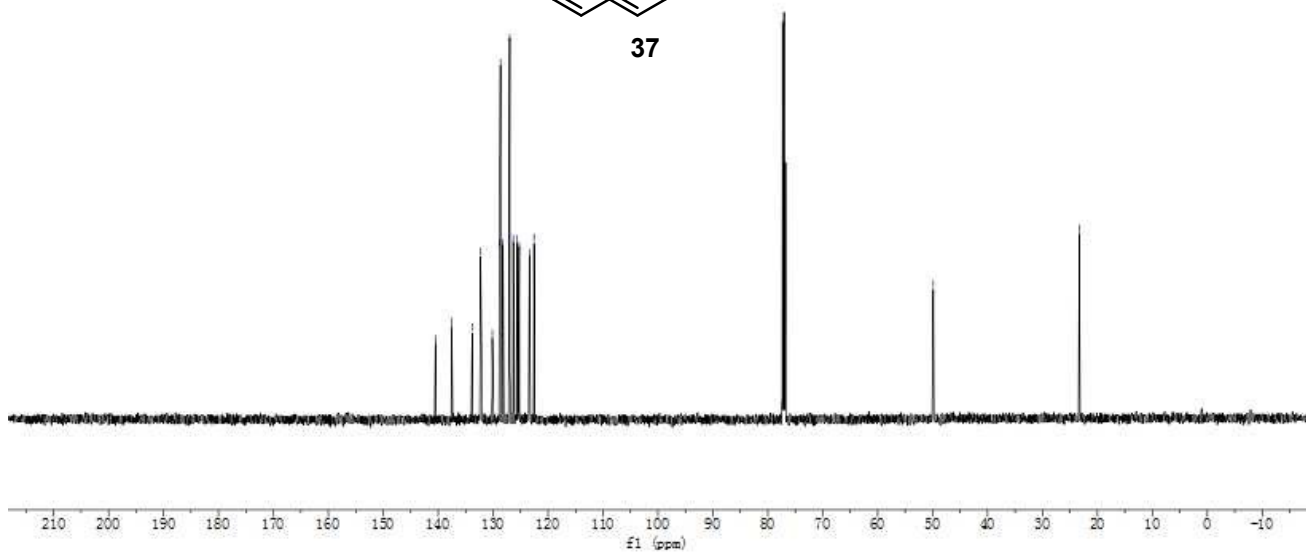
140.5  
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130.1  
128.9  
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127.0  
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125.7  
125.2  
123.4  
122.6

49.9

23.3

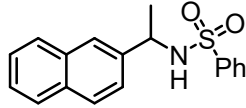


37

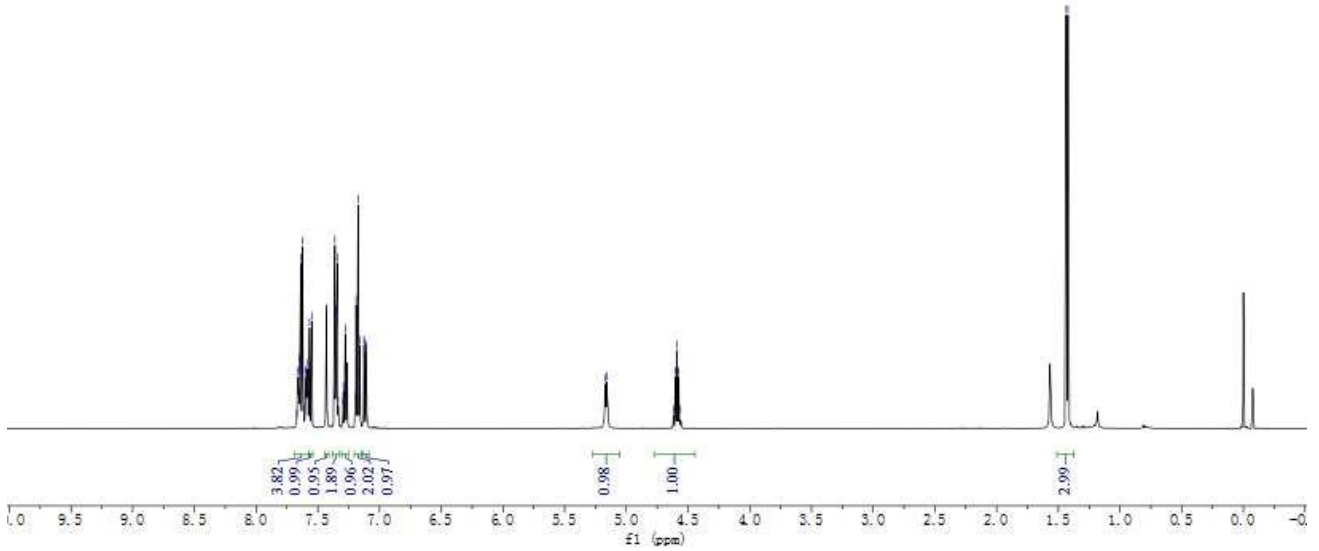


7.67  
7.66  
7.66  
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7.65  
7.64  
7.64  
7.64  
7.64  
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1.92

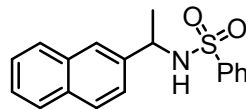
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



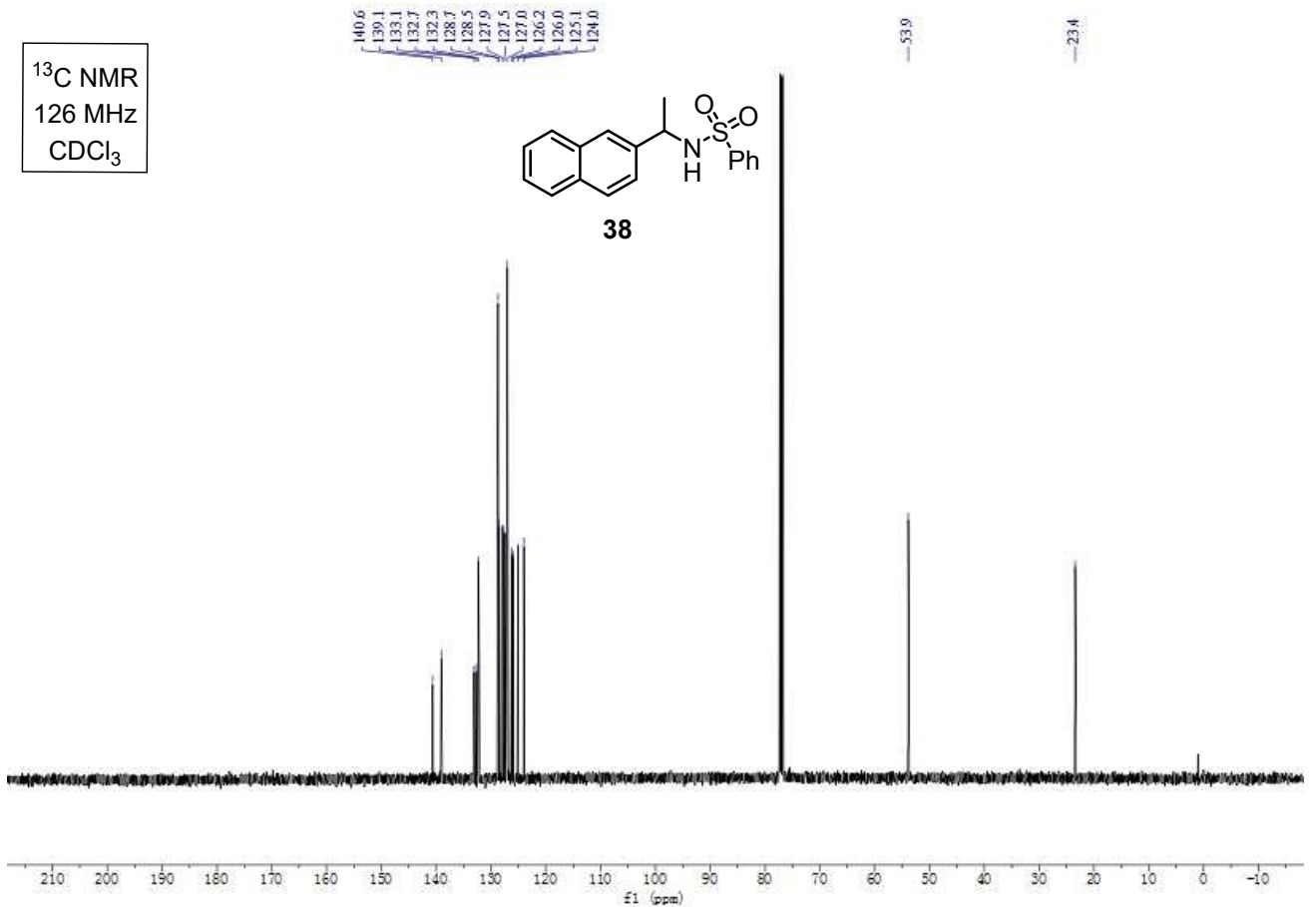
**38**



<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

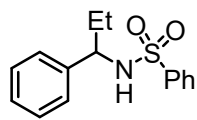


**38**

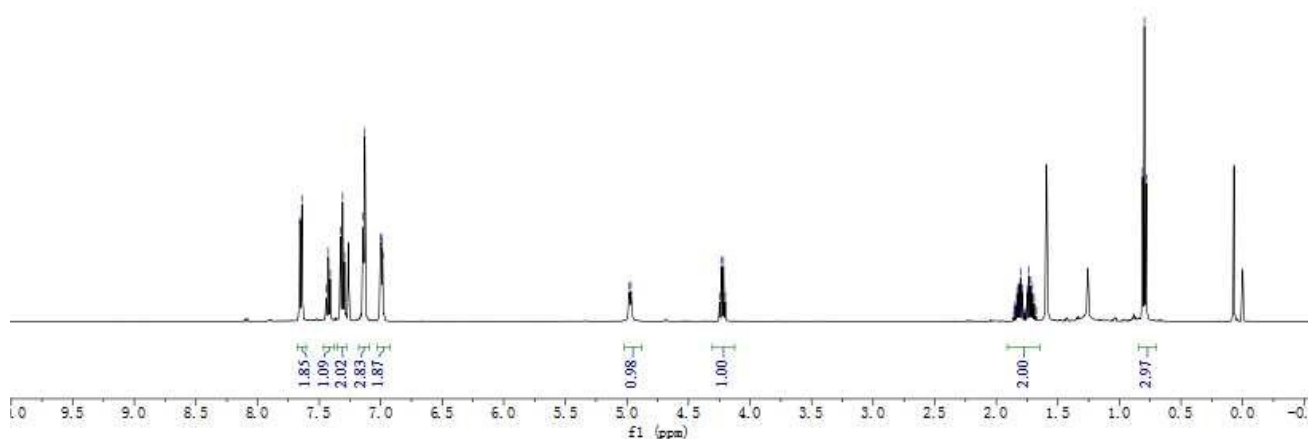


<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

7.66, 7.64, 7.44, 7.42, 7.41, 7.32, 7.31, 7.29, 7.14, 7.13, 7.00, 6.99, 6.99, 6.98, 4.98, 4.97, 4.25, 4.23, 4.22, 4.20, 1.86, 1.85, 1.83, 1.83, 1.82, 1.80, 1.79, 1.78, 1.77, 1.75, 1.72, 1.71, 1.69, 1.68, 0.81, 0.80, 0.78

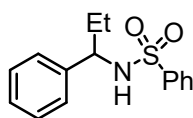


**39**

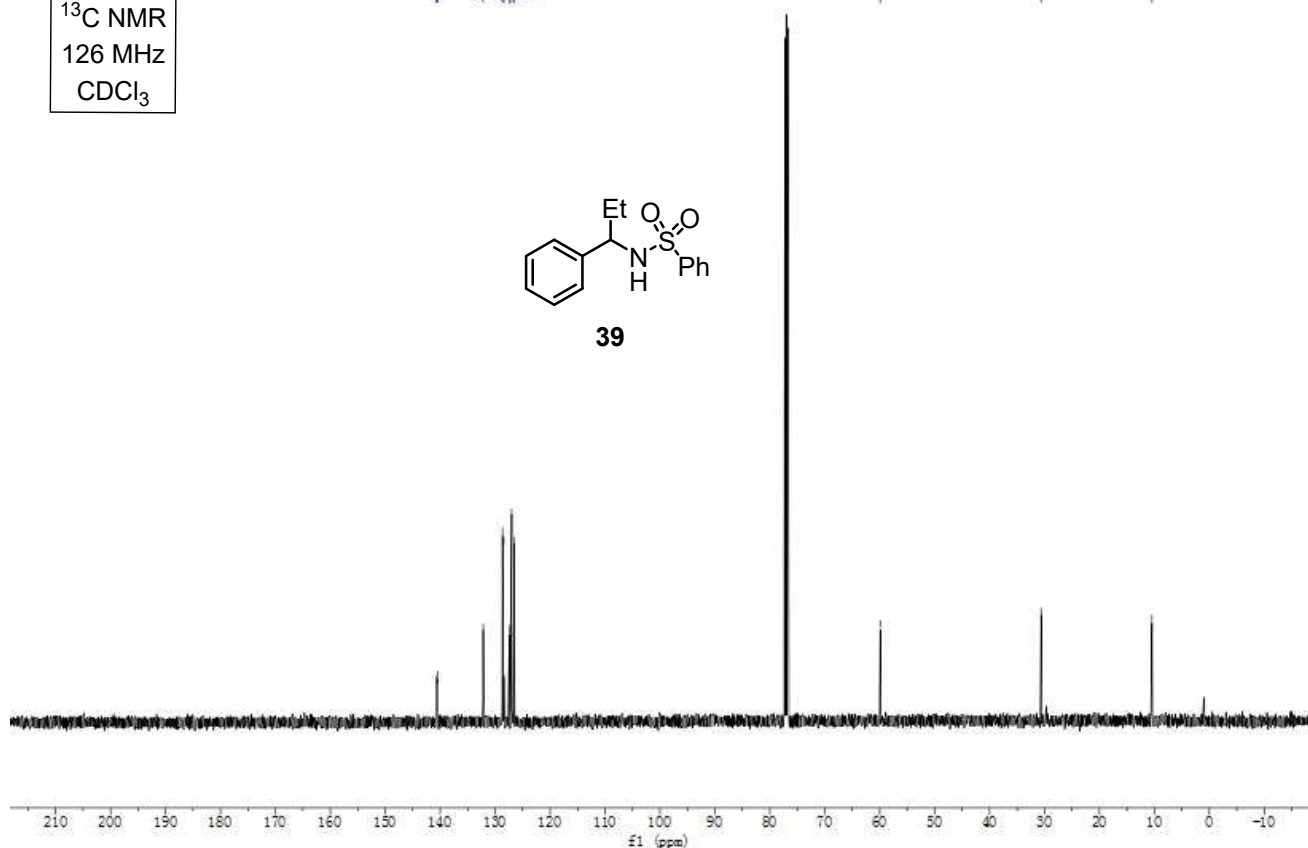


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

140.7, 140.5, 132.2, 128.7, 128.4, 127.4, 127.0, 126.5, 59.9, 30.6, 10.4

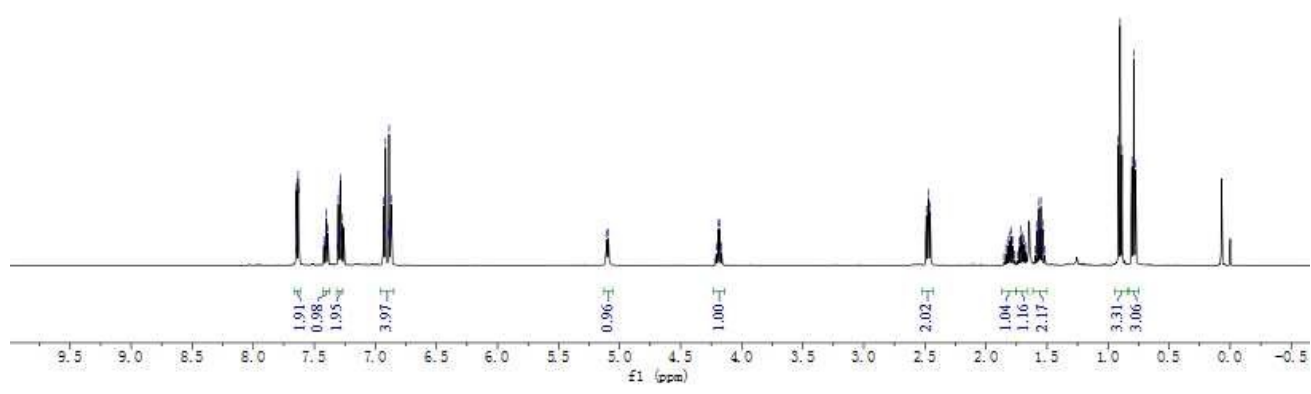
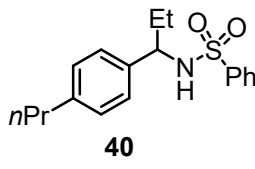


**39**



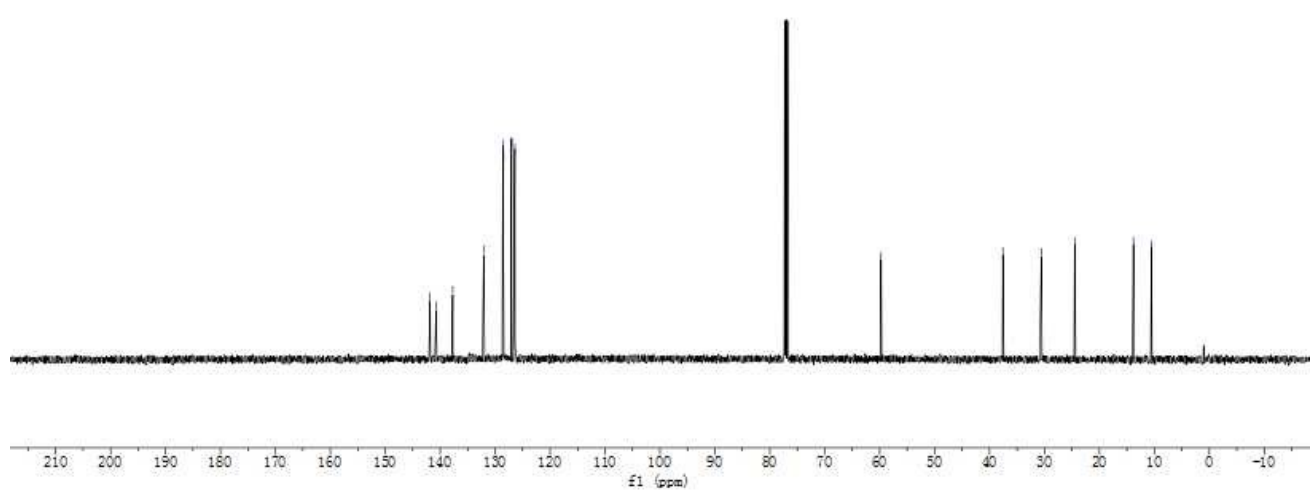
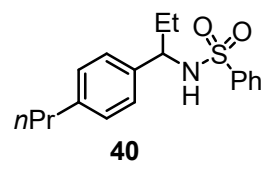
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0.78

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



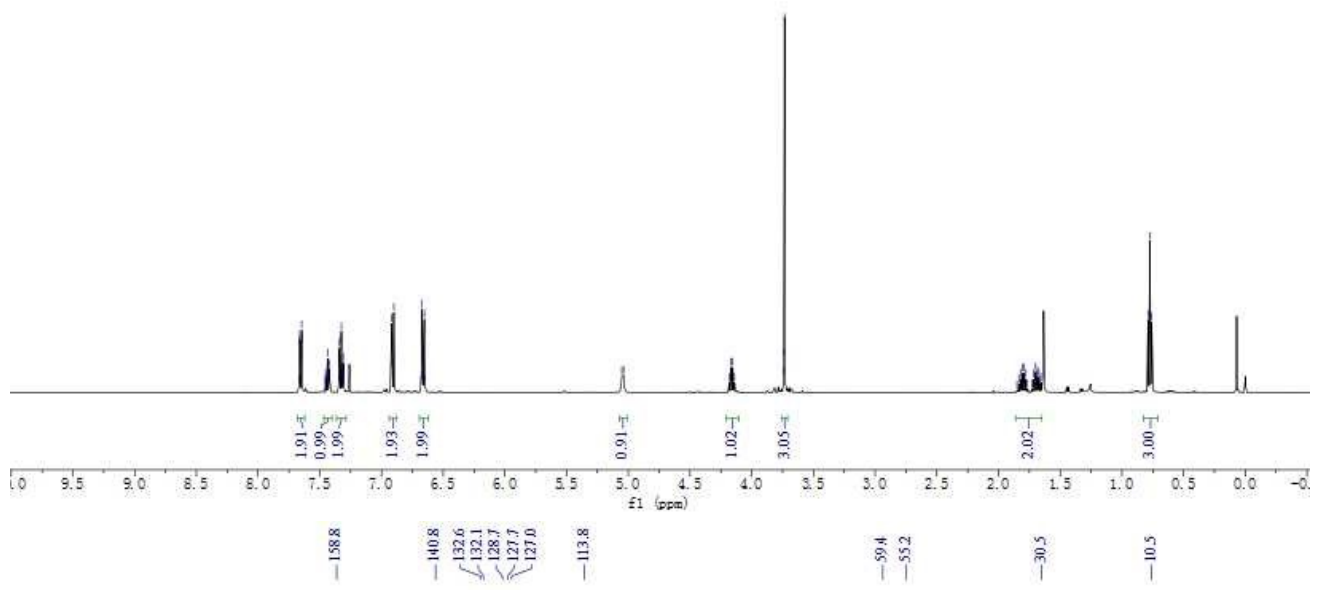
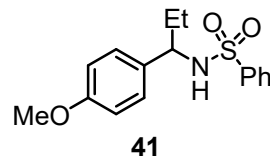
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37.6  
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24.5  
13.8  
10.5

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

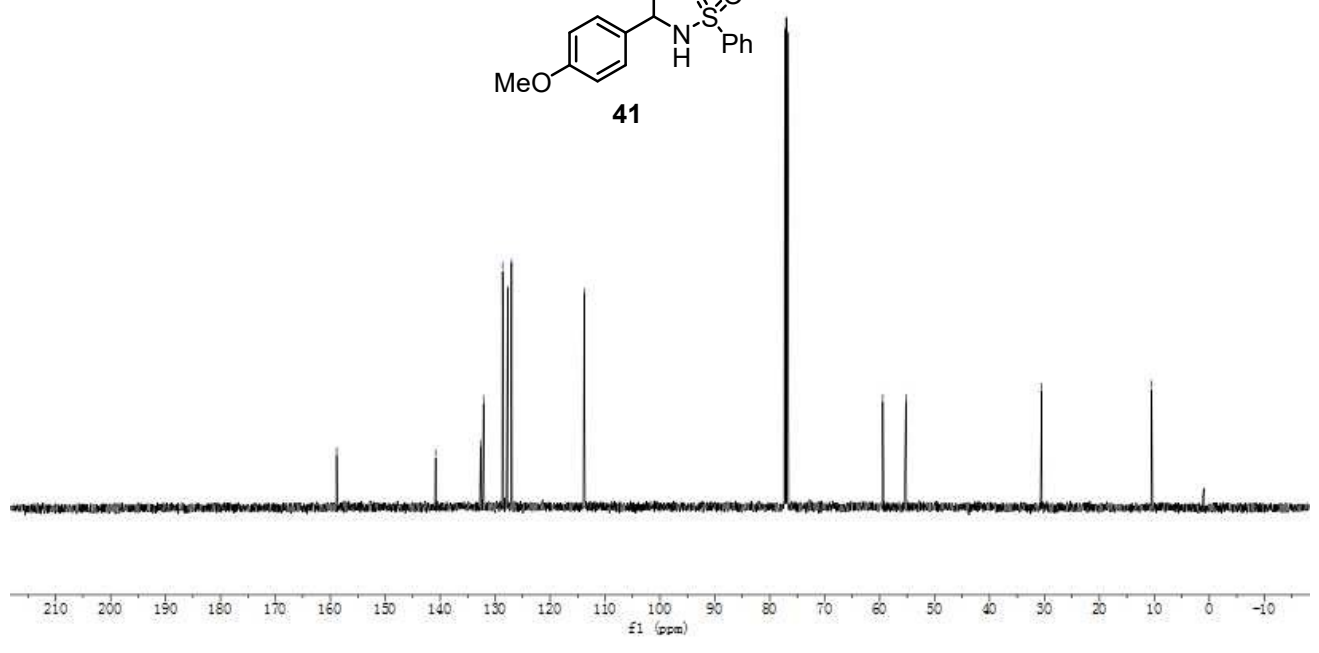
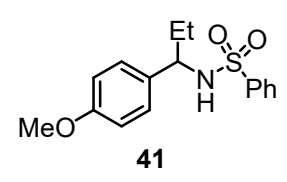


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0.77  
0.76

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



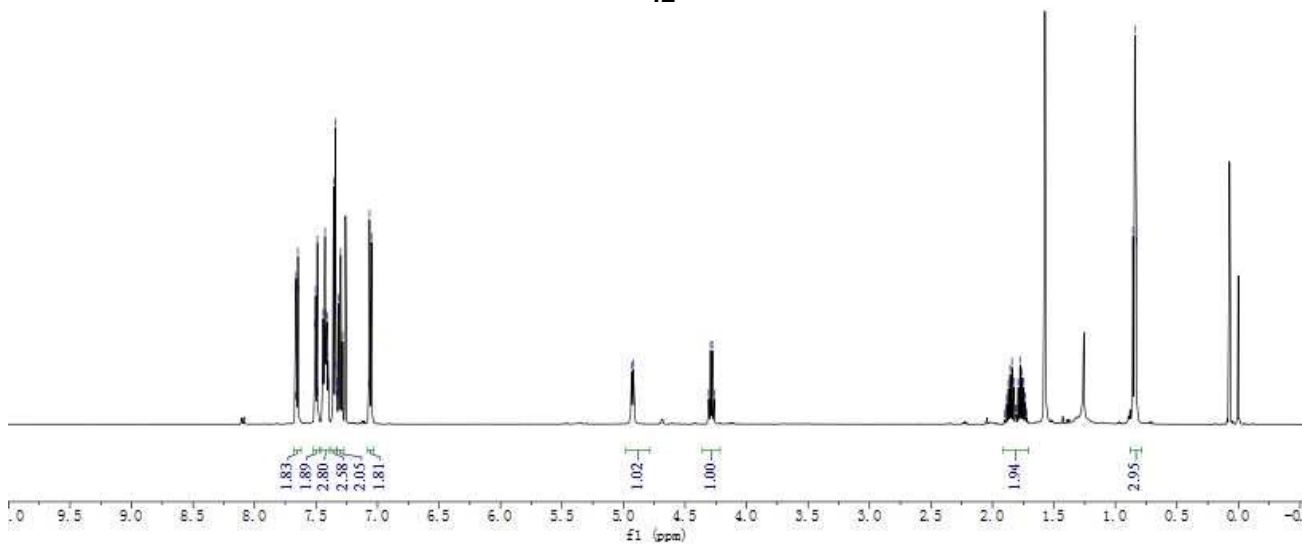
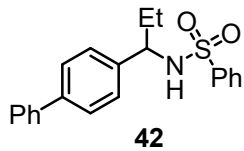
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



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0.83

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



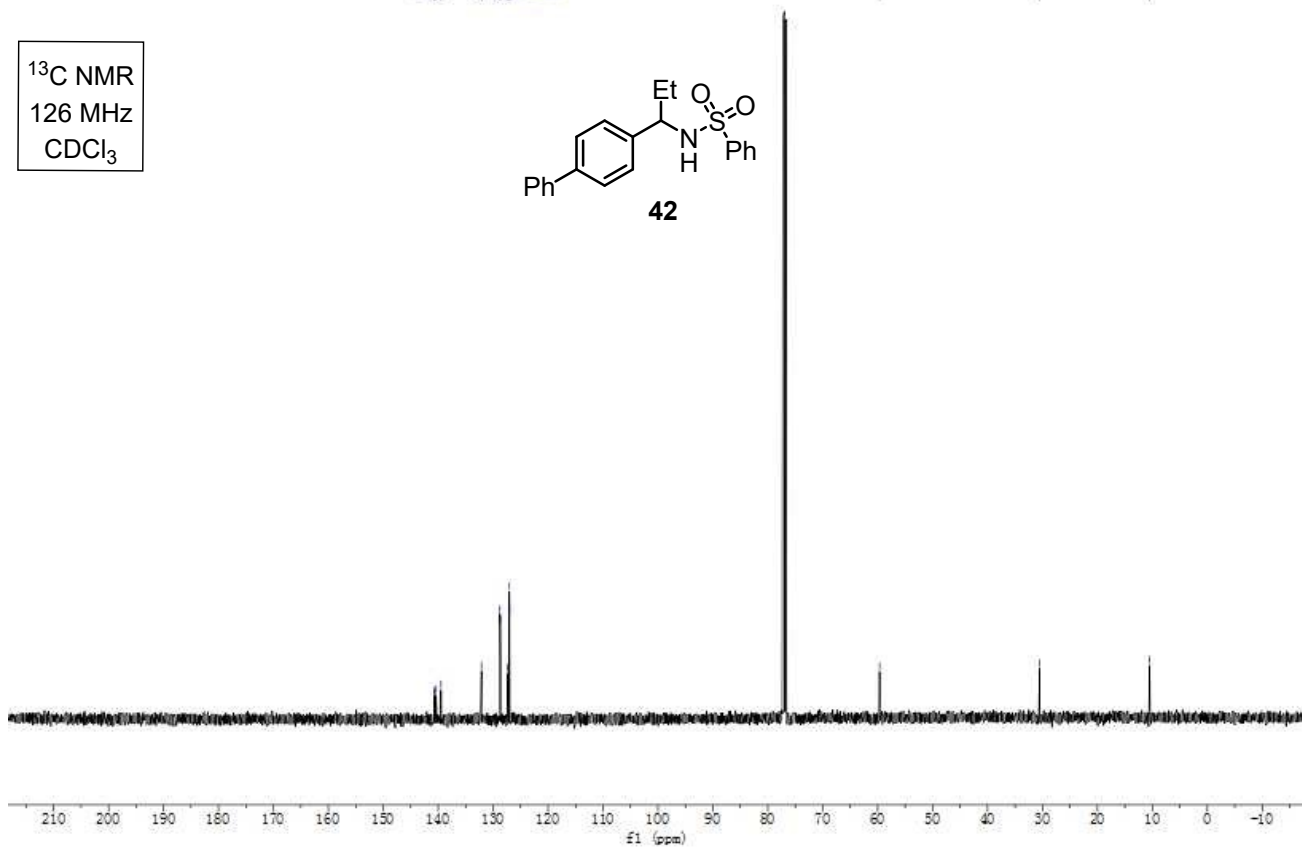
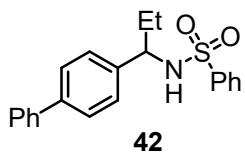
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139.5  
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128.7  
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127.1  
127.0  
127.0  
127.0

59.7

30.5

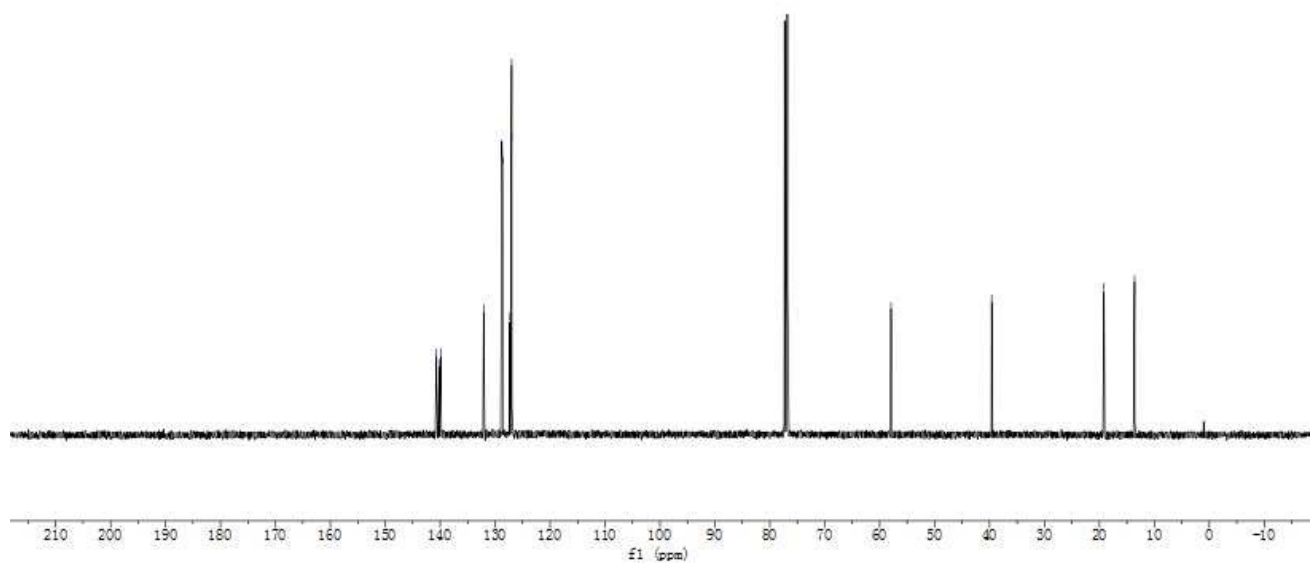
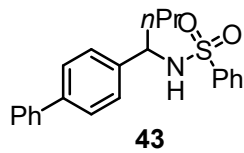
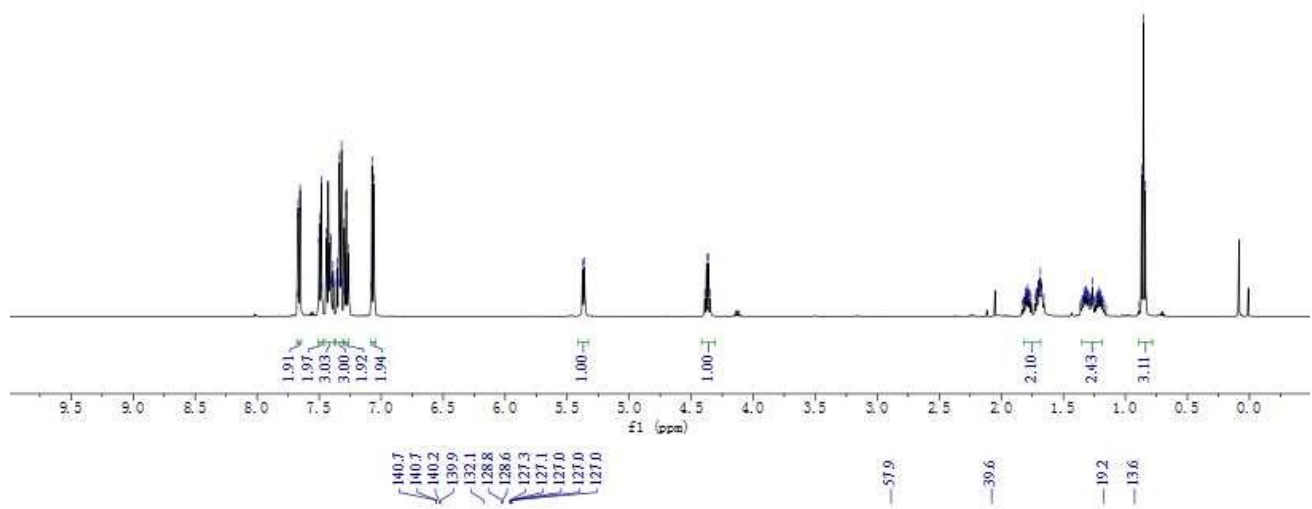
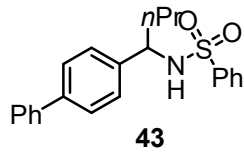
10.5

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



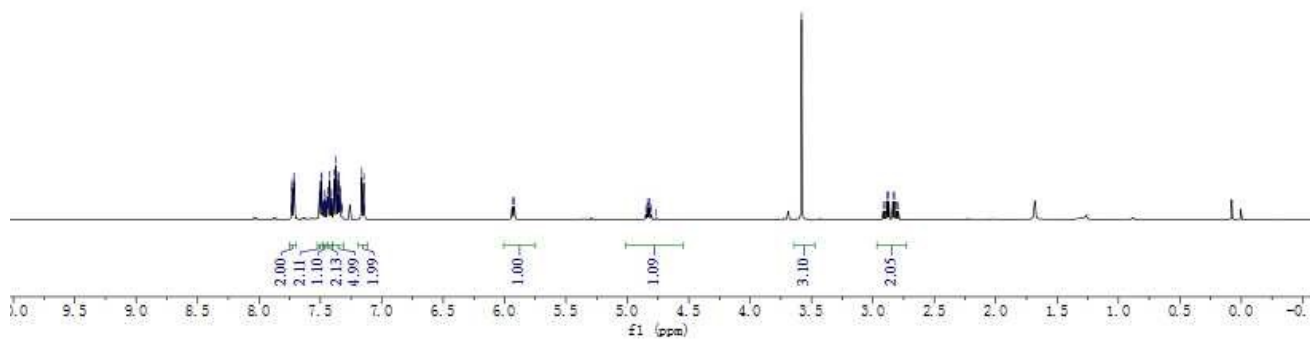
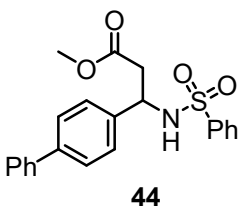
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0.84

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

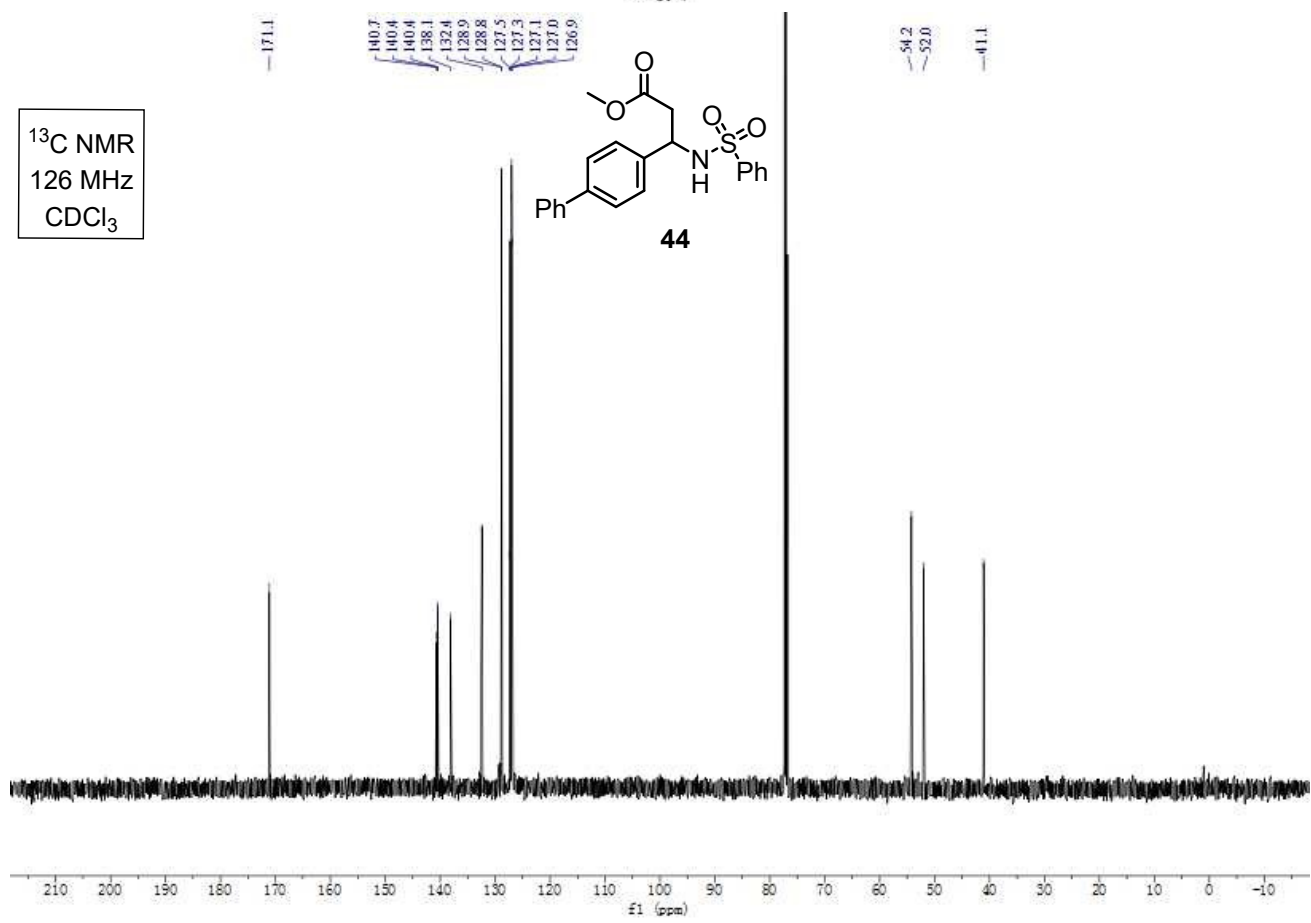
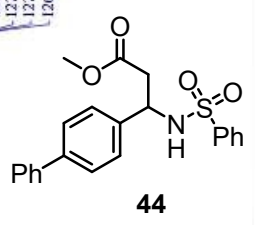


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<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



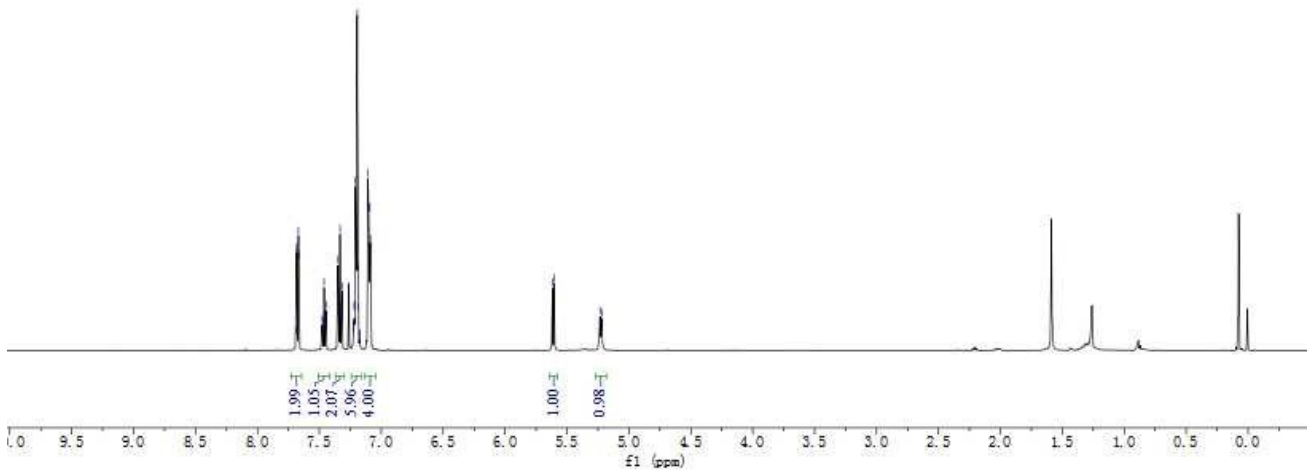


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7.09  
5.62  
5.49  
5.22

$^1\text{H}$  NMR  
500 MHz  
 $\text{CDCl}_3$



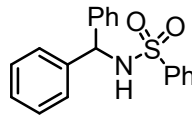
**45**



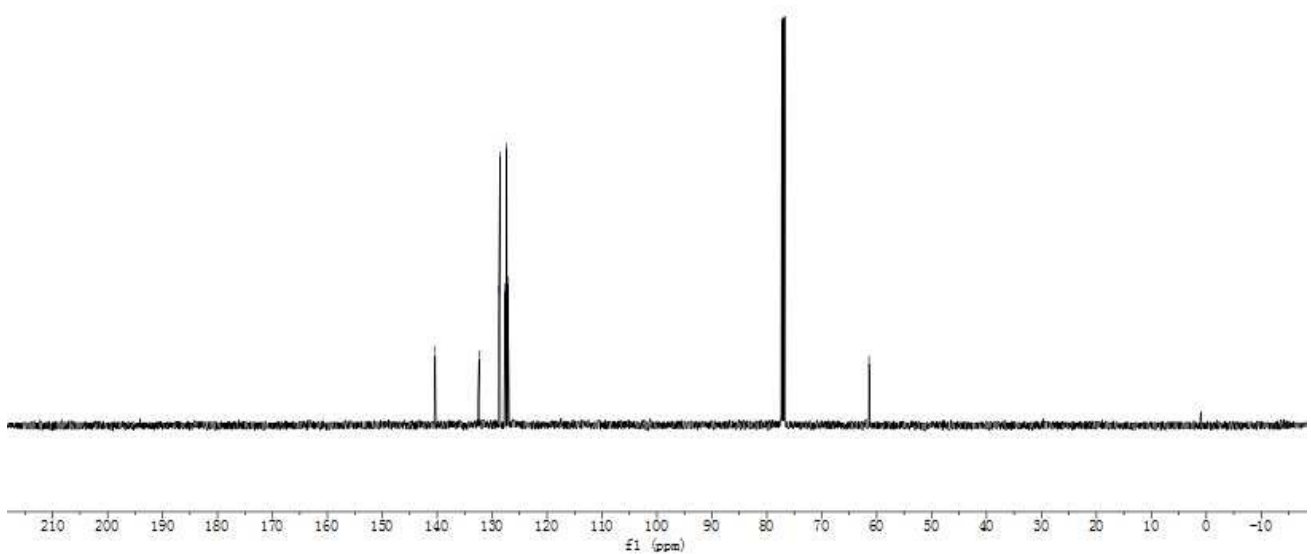
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127.4  
127.1

61.4

$^{13}\text{C}$  NMR  
126 MHz  
 $\text{CDCl}_3$

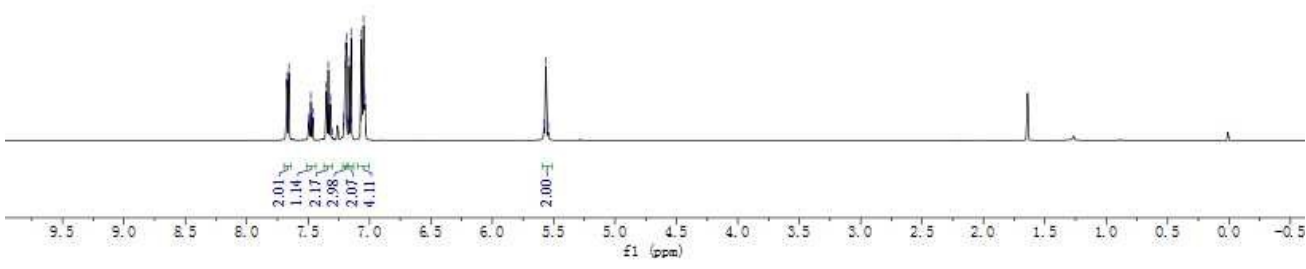
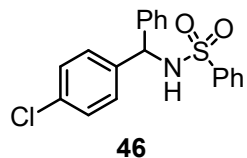


**45**



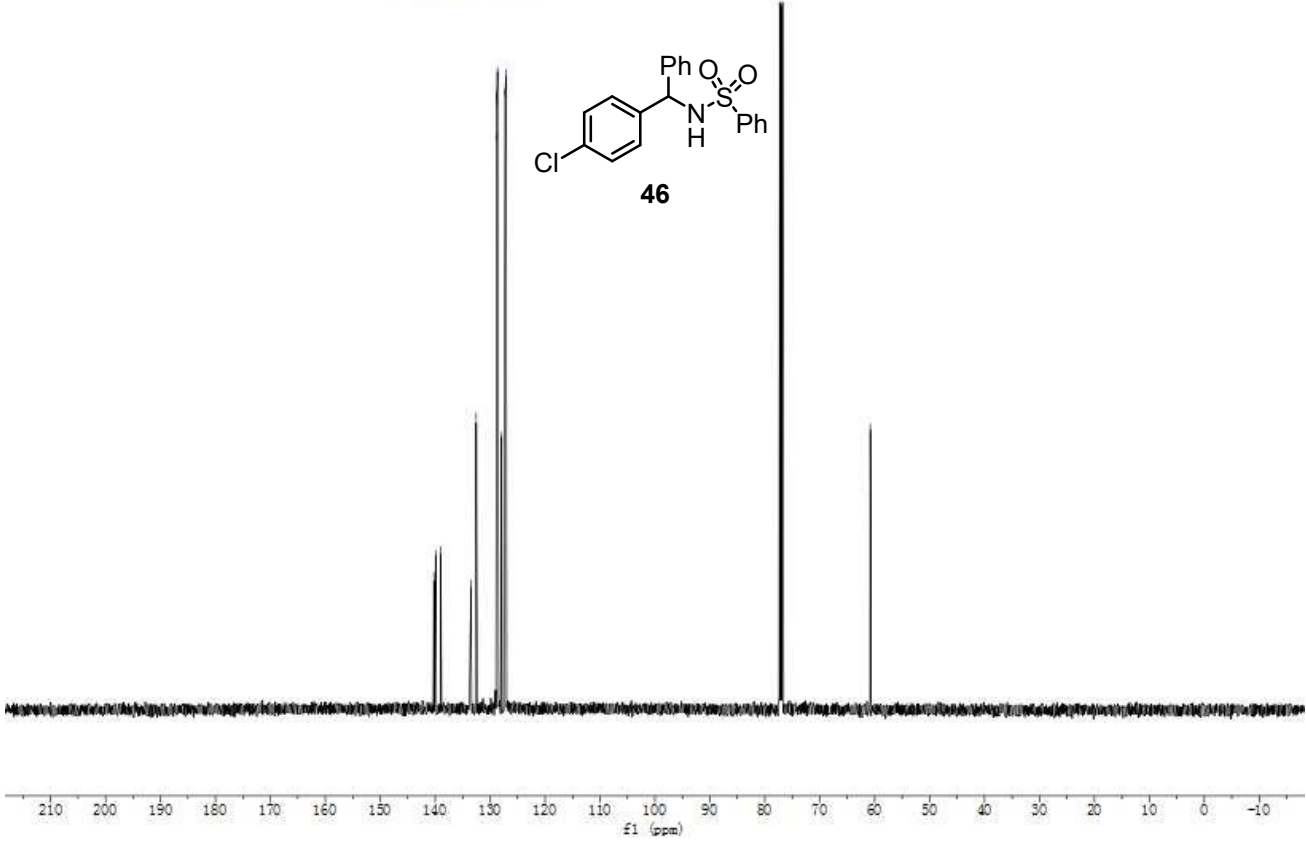
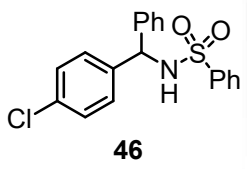
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7.03  
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5.96  
5.94

$^1\text{H NMR}$   
500 MHz  
 $\text{CDCl}_3$



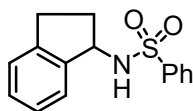
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127.1

60.8

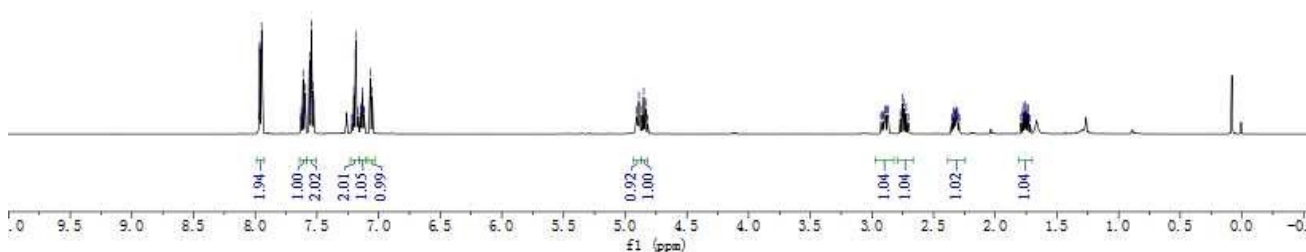


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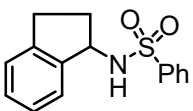
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



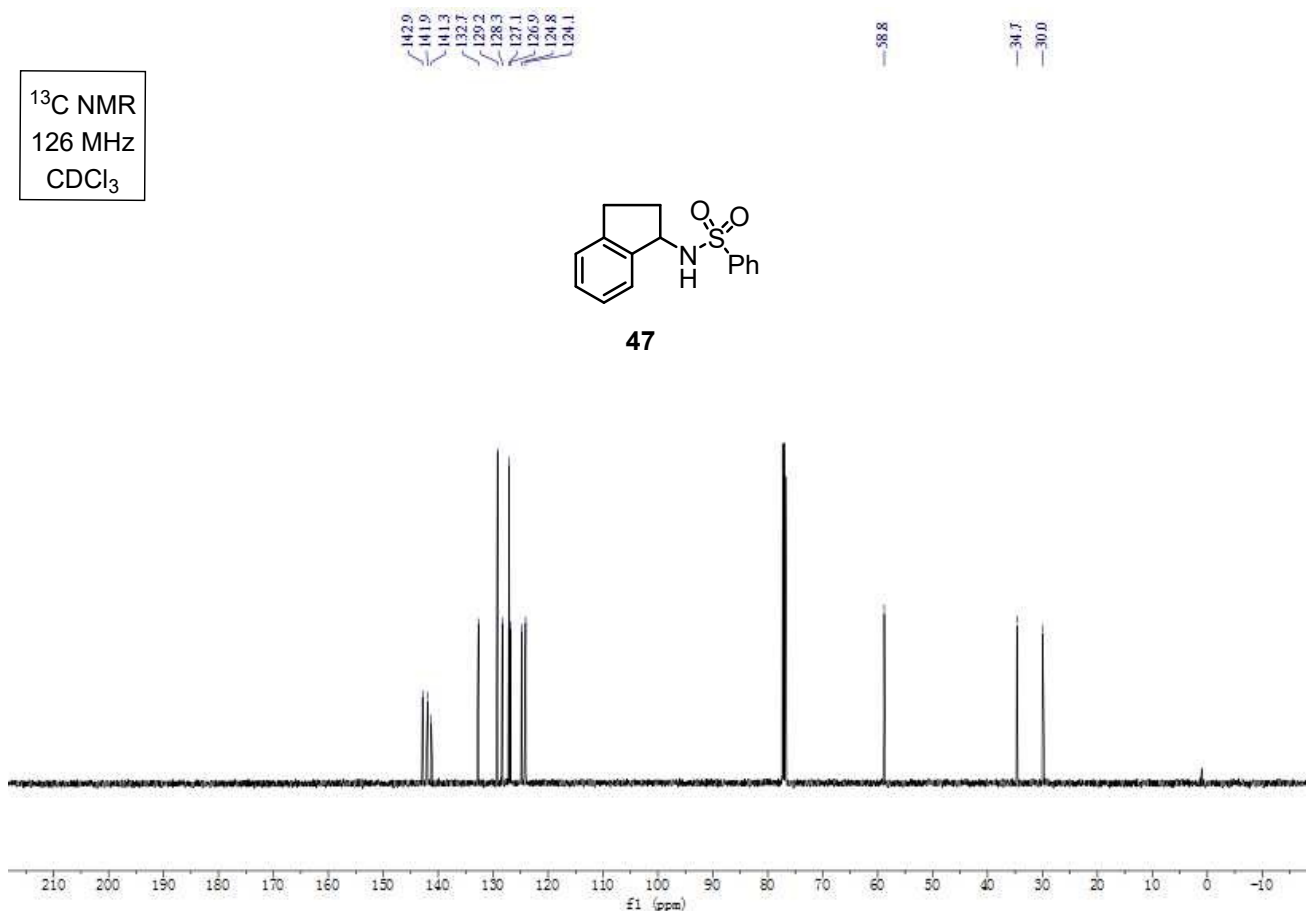
47



<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

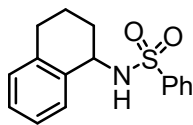


47

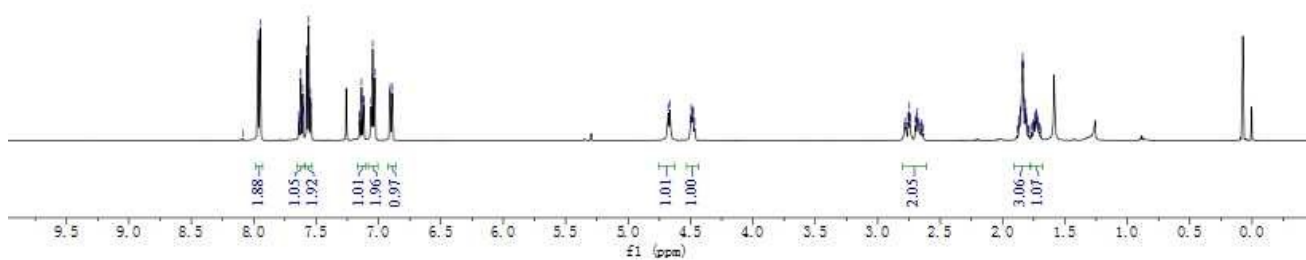


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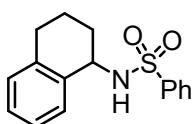
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



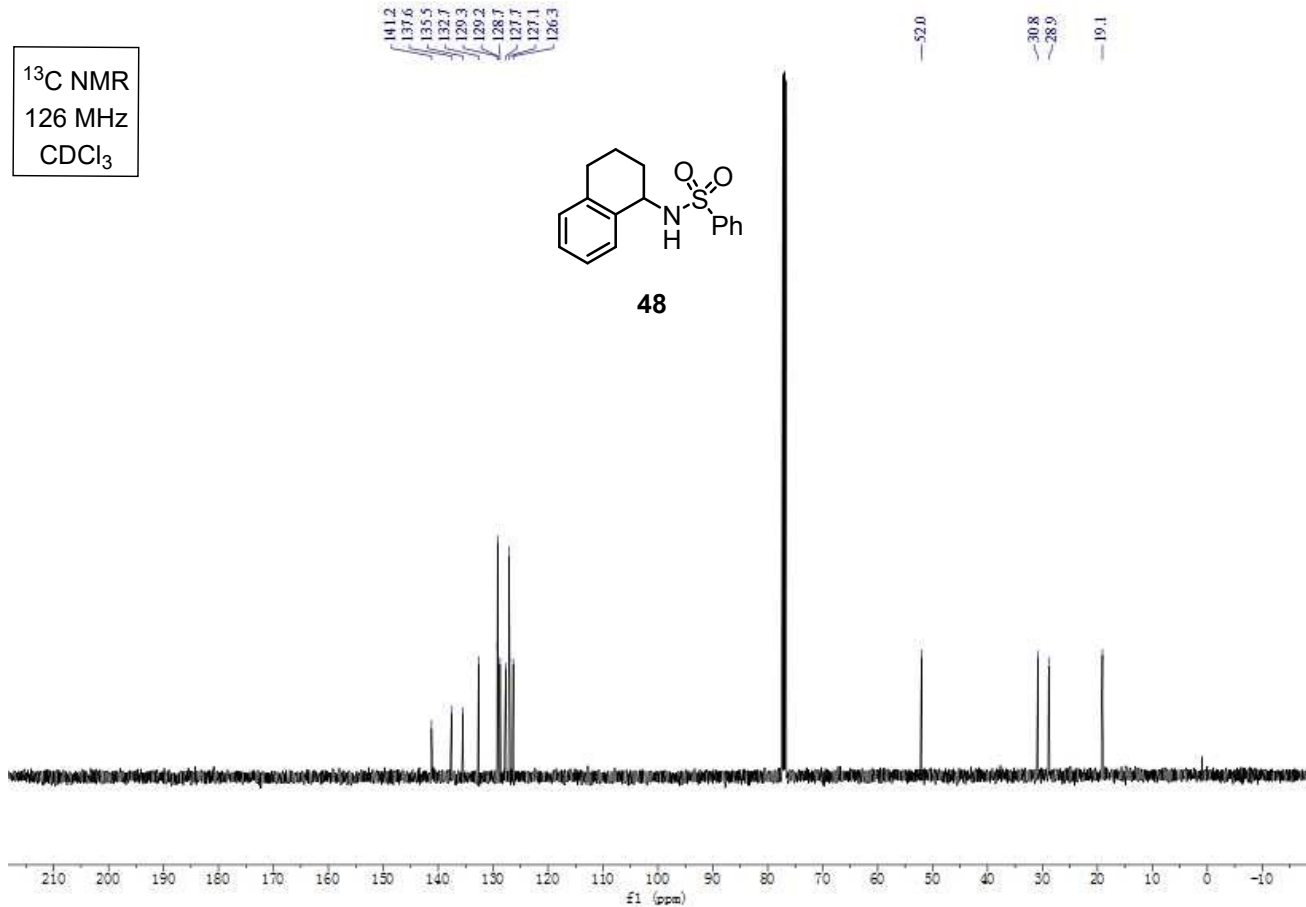
48



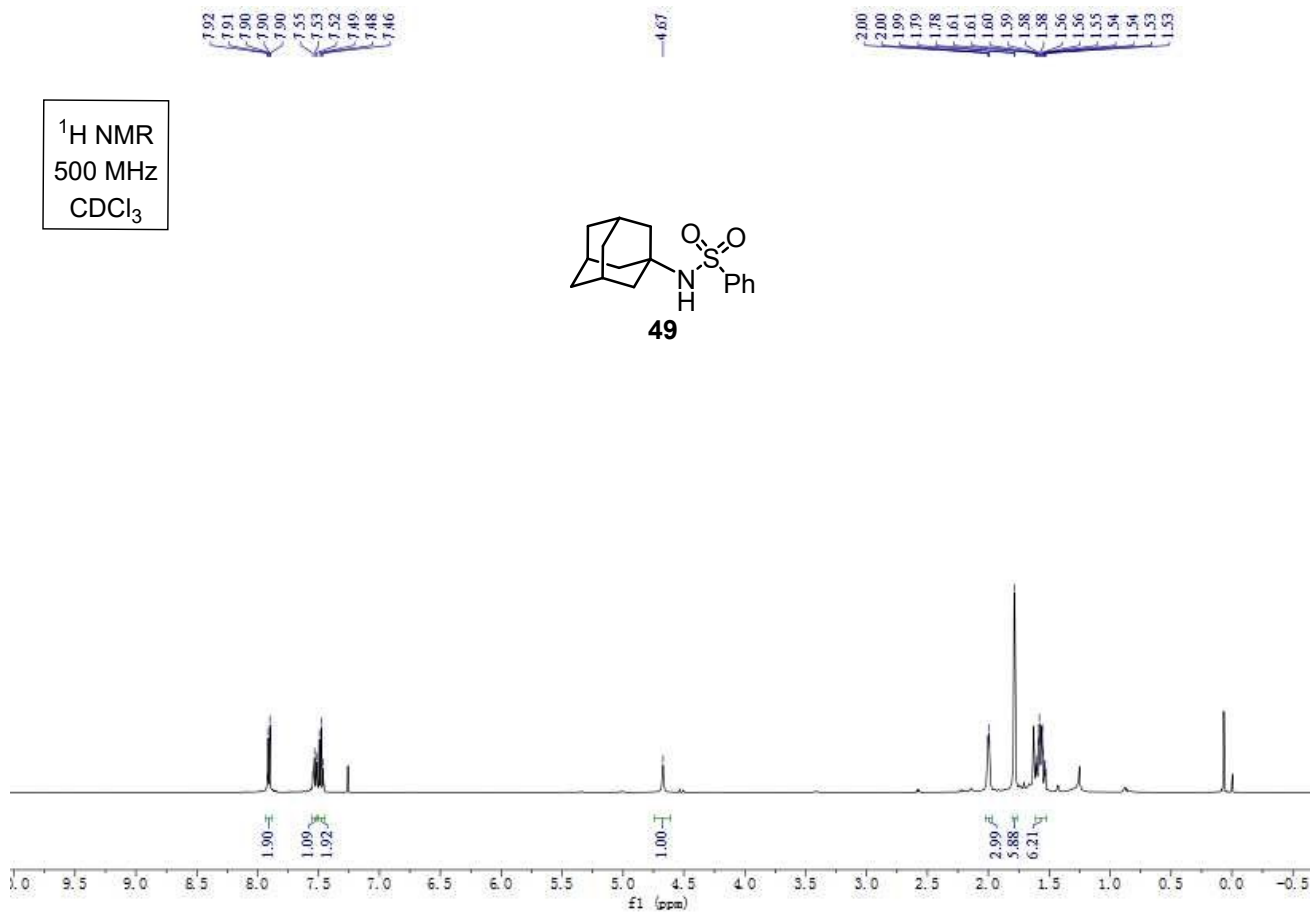
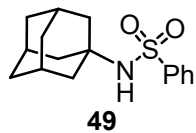
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



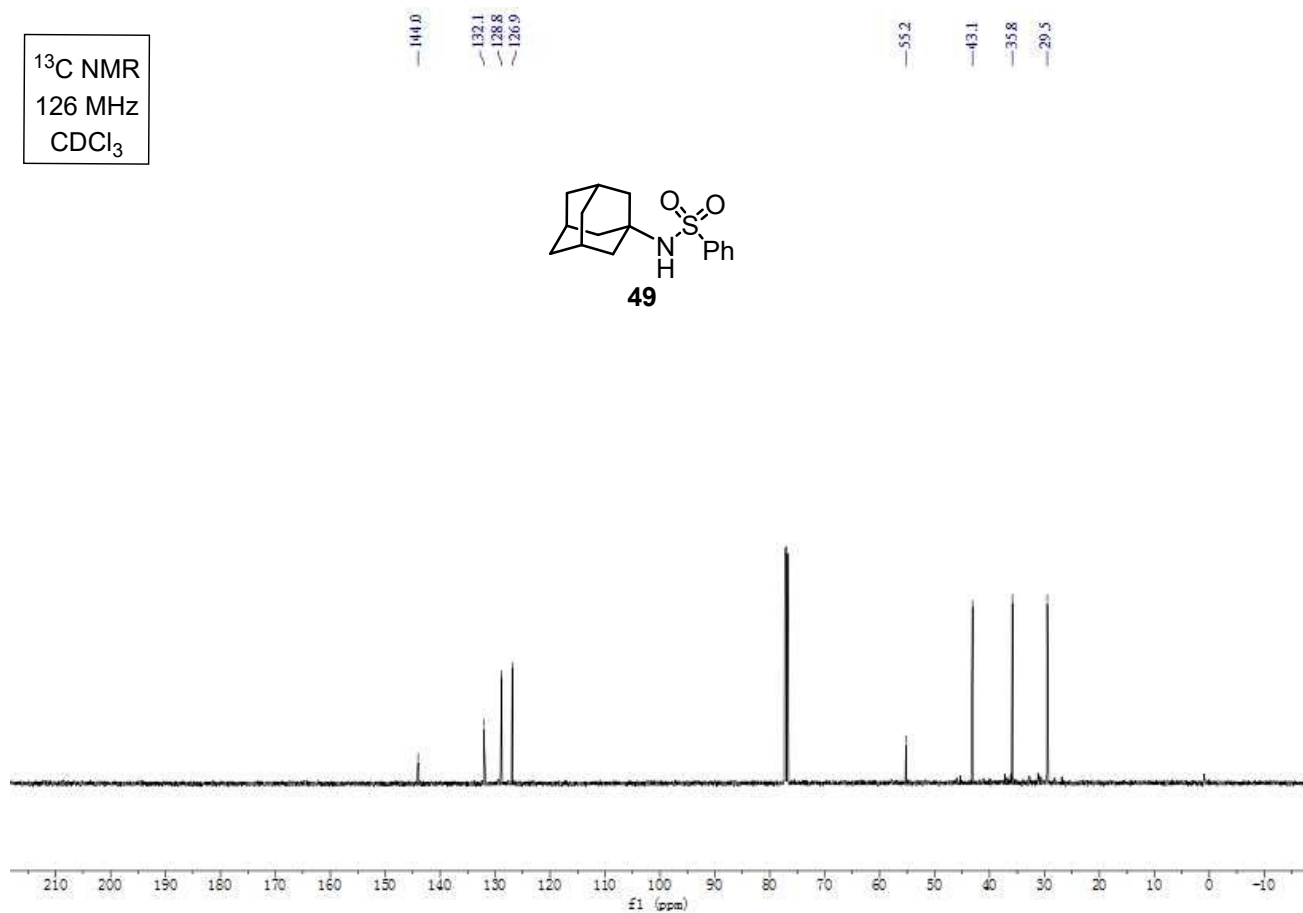
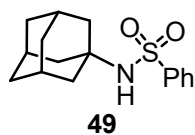
48



<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

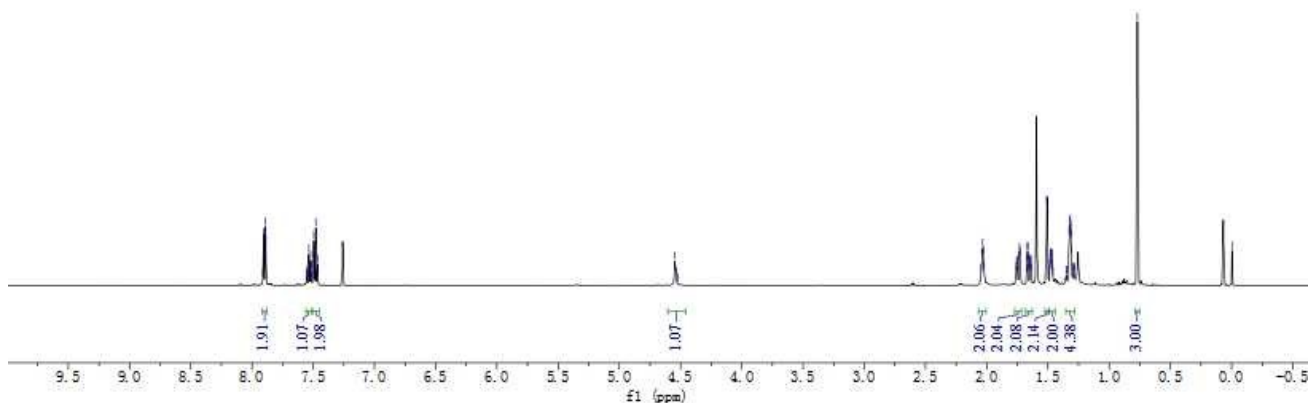
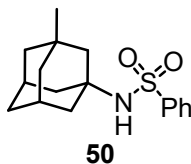


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

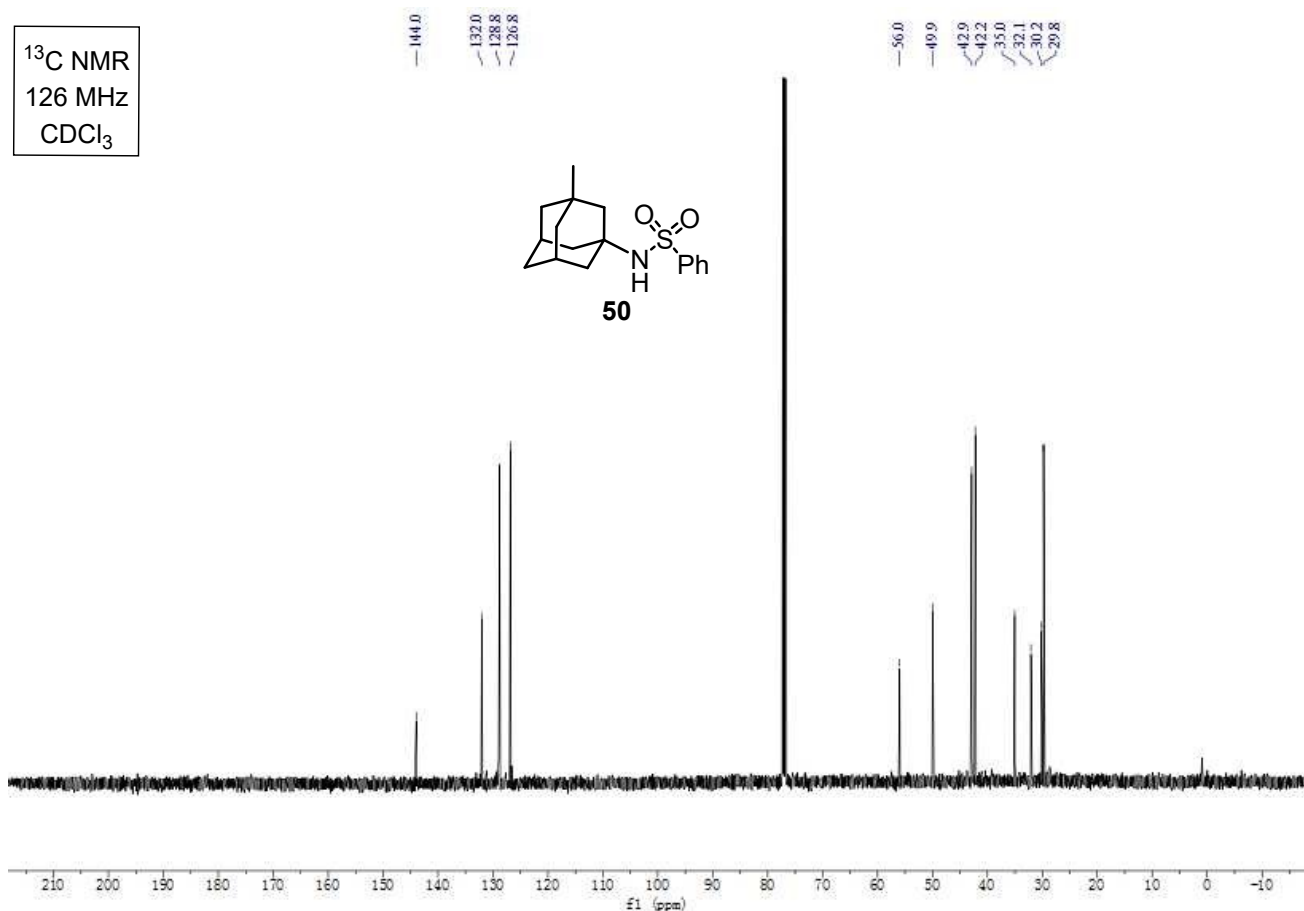
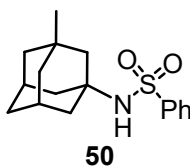


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7.90  
7.89  
7.89  
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7.46  
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4.54  
4.53  
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1.29  
1.28  
1.28  
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1.28  
0.77  
0.77  
0.00

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



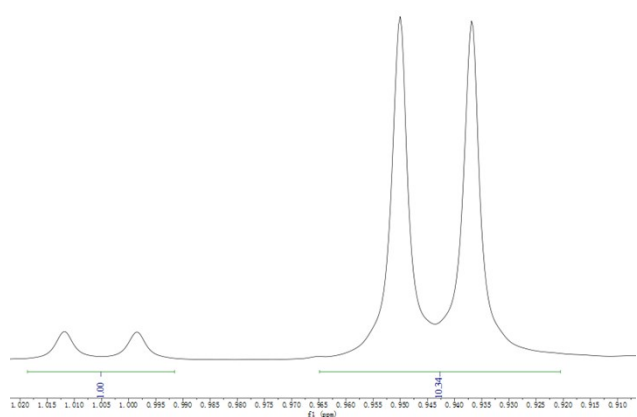
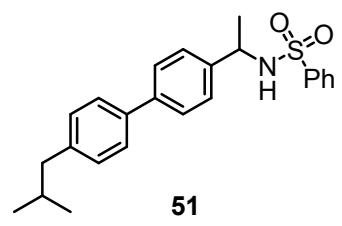
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



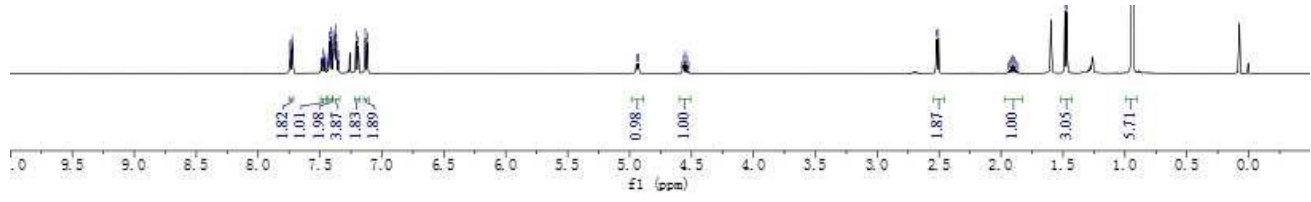
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7.43  
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4.55  
4.54  
4.52

2.52  
2.51  
1.94  
1.93  
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1.90  
1.89  
1.88  
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1.48  
1.47  
0.95  
0.93

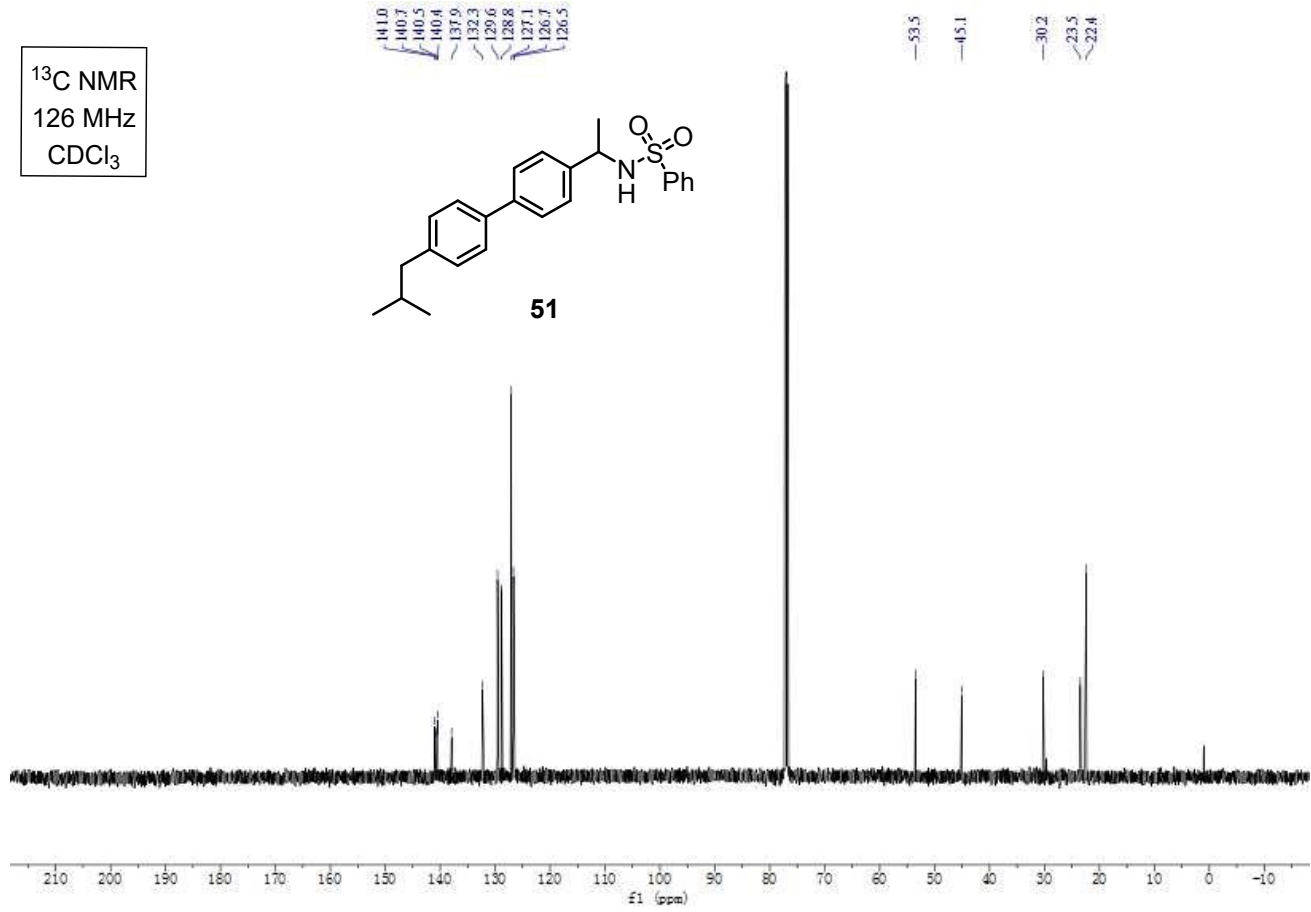
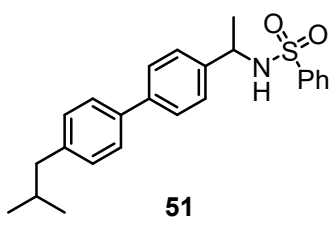
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



crude 1H NMR analysis, *r.r.* = 10:1



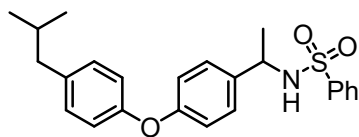
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



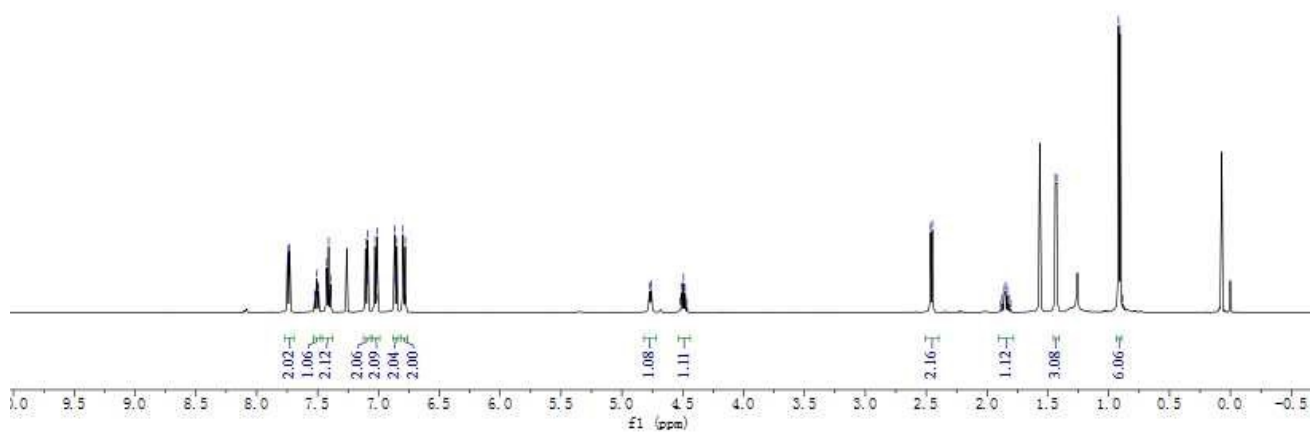
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7.41  
7.40  
7.39  
7.39  
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7.10  
7.09  
7.09  
7.03  
7.02  
7.02  
7.01  
7.01  
6.87  
6.86  
6.85  
6.85  
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6.80  
6.79  
6.79  
6.78  
6.77  
6.77  
4.77  
4.76  
4.52  
4.51  
4.51  
4.49  
4.48  
4.47

2.46  
2.45  
1.89  
1.87  
1.86  
1.85  
1.83  
1.82  
1.81  
1.44  
1.43  
0.92  
0.91  
0.88

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



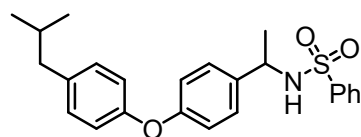
52-1



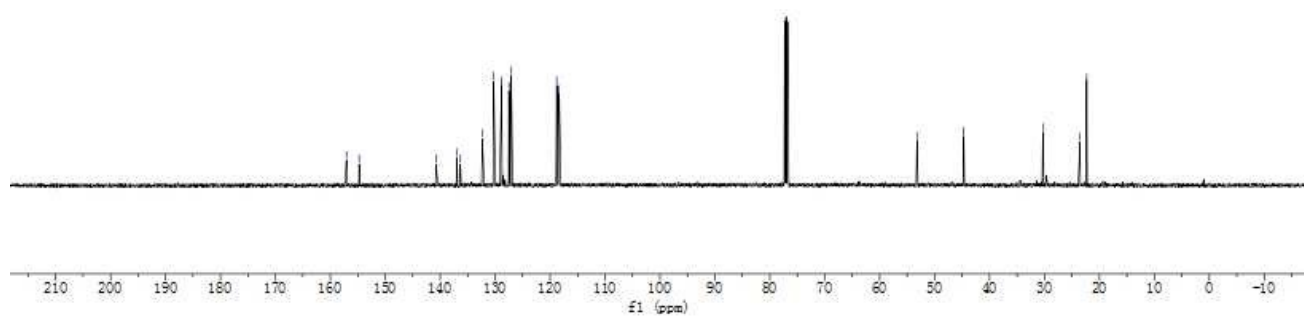
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136.3  
132.3  
130.3  
128.8  
127.5  
127.1  
118.8  
118.4

53.2  
44.7  
30.3  
23.5  
22.3

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



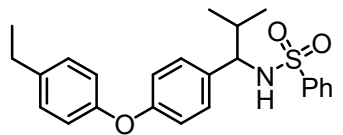
52-1



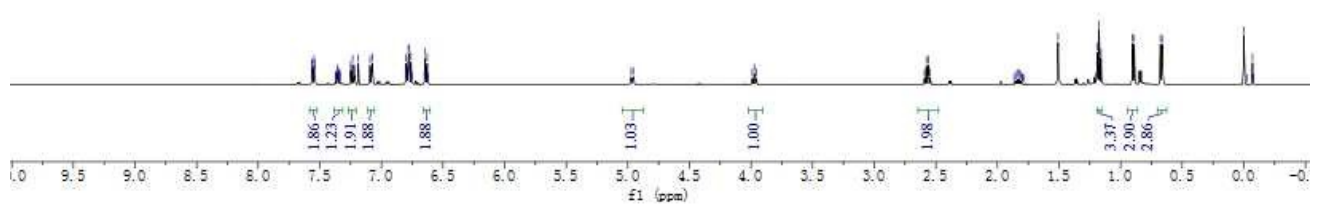


7.57  
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7.37  
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7.36  
7.35  
7.34  
7.34  
7.34  
7.25  
7.25  
7.23  
7.23  
7.22  
7.19 CDCl<sub>3</sub>  
7.09  
7.09  
7.08  
7.08  
6.80  
6.80  
6.79  
6.79  
6.78  
6.78  
6.77  
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6.76  
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6.65  
6.65  
6.64  
6.64  
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1.85  
1.84  
1.83  
1.81  
1.80  
1.80  
1.51 H<sub>2</sub>O  
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1.19  
1.18  
1.17  
1.16  
1.16  
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0.89  
0.68  
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-0.07

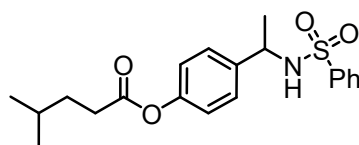
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



52-2



<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

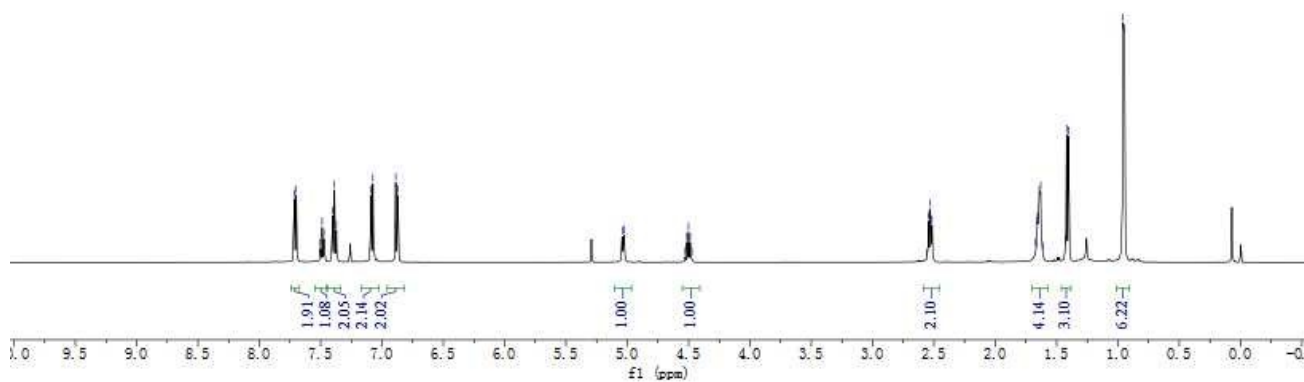


**53**

7.71  
7.70  
7.50  
7.49  
7.47  
7.40  
7.39  
7.37  
7.09  
7.07  
6.88  
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4.51  
4.50  
4.49  
4.47

2.56  
2.54  
2.53  
2.51  
1.67  
1.66  
1.66  
1.65  
1.64  
1.63  
1.61  
1.43  
1.42  
1.40  
0.96  
0.95



172.4

150.0

140.6

139.3

132.4

128.9

127.2

127.0

121.6

53.2

33.7

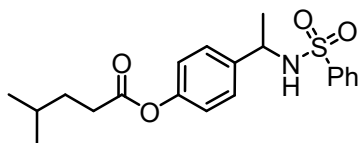
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27.7

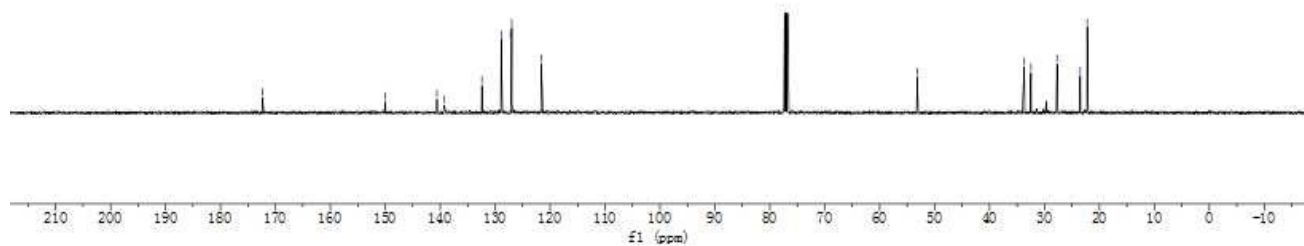
23.5

22.2

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

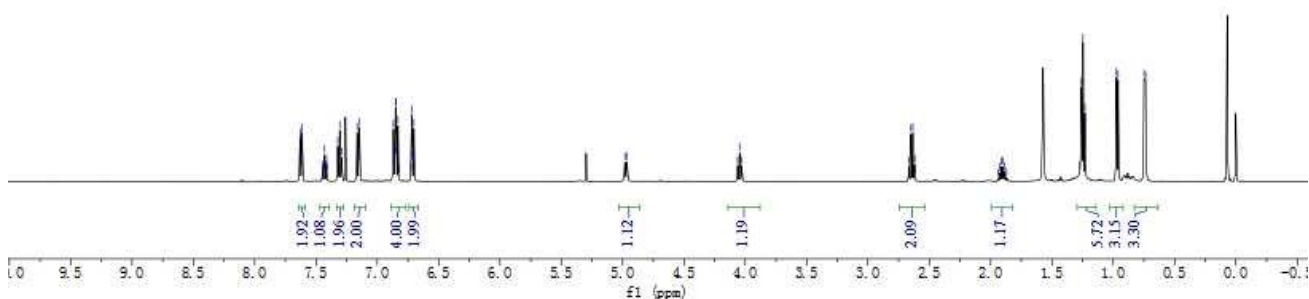
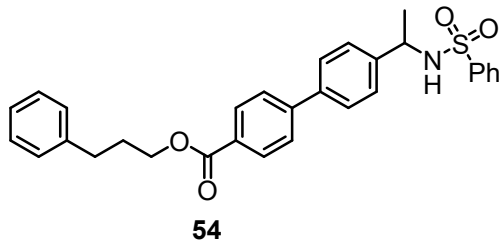


**53**

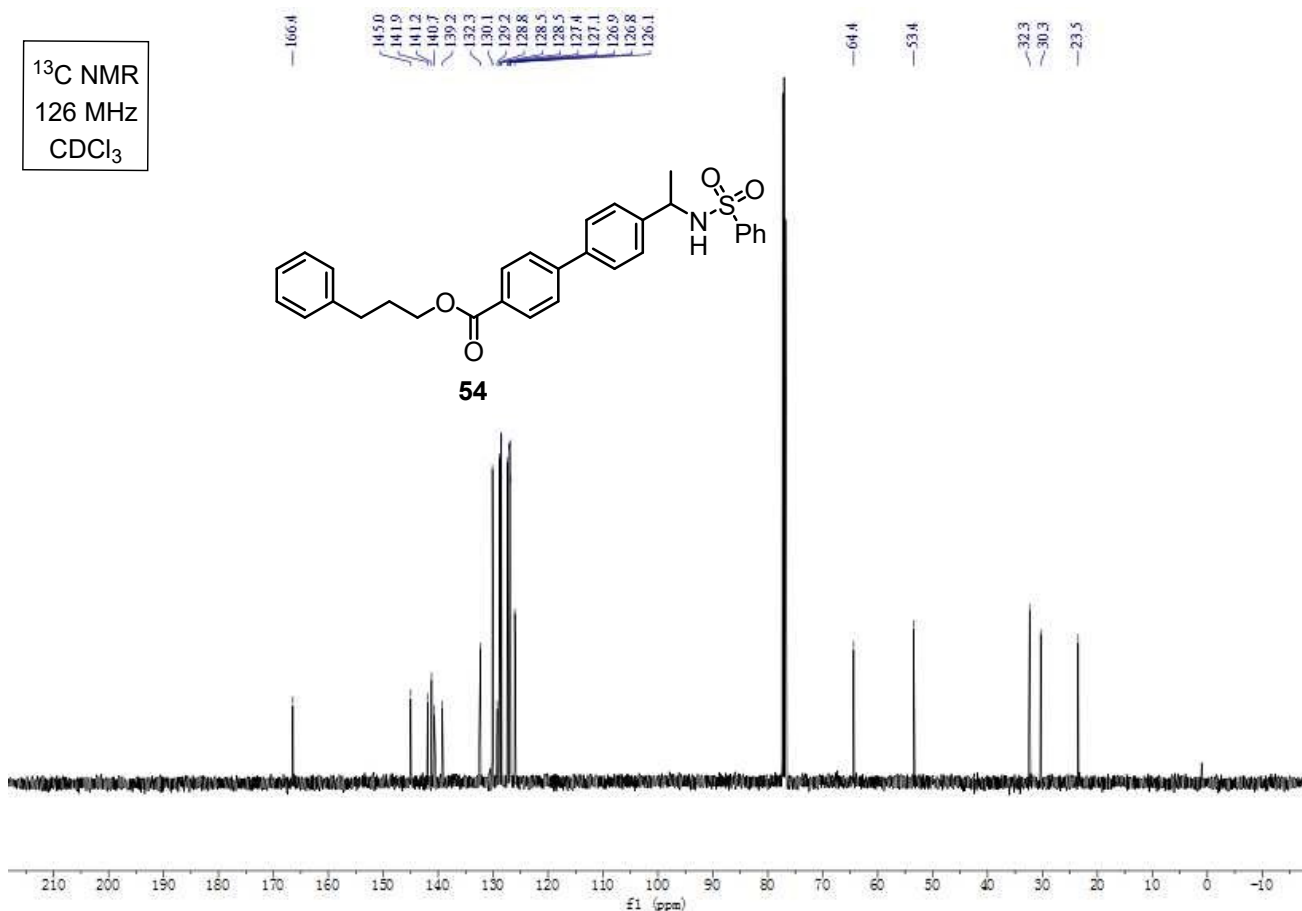
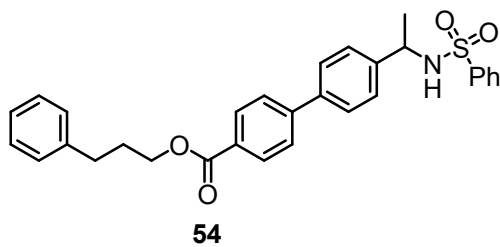


7.63, 7.63, 7.62, 7.61, 7.44, 7.43, 7.42, 7.41, 7.32, 7.32, 7.31, 7.30, 7.29, 7.16, 7.16, 7.15, 7.15, 6.87, 6.86, 6.86, 6.85, 6.85, 6.84, 6.84, 6.84, 6.83, 6.72, 6.71, 6.71, 4.98, 4.97, 4.06, 4.05, 4.03, 2.66, 2.65, 2.63, 2.62, 1.94, 1.92, 1.91, 1.90, 1.88, 1.87, 1.27, 1.26, 1.26, 1.25, 1.23, 0.97, 0.96, 0.75, 0.74

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

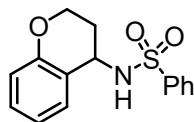


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

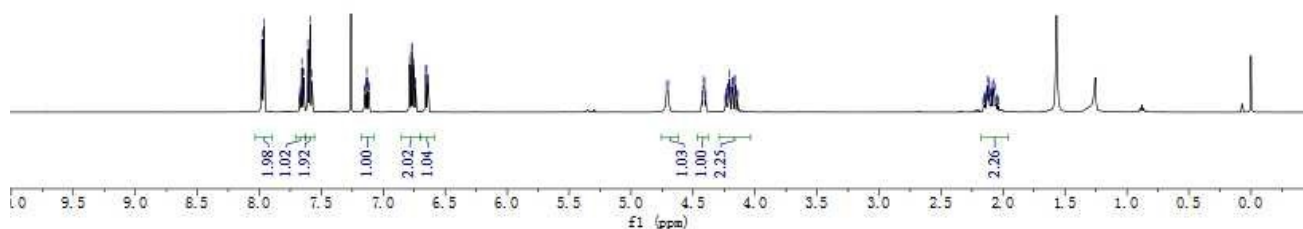


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2.07  
2.06

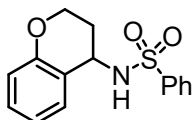
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



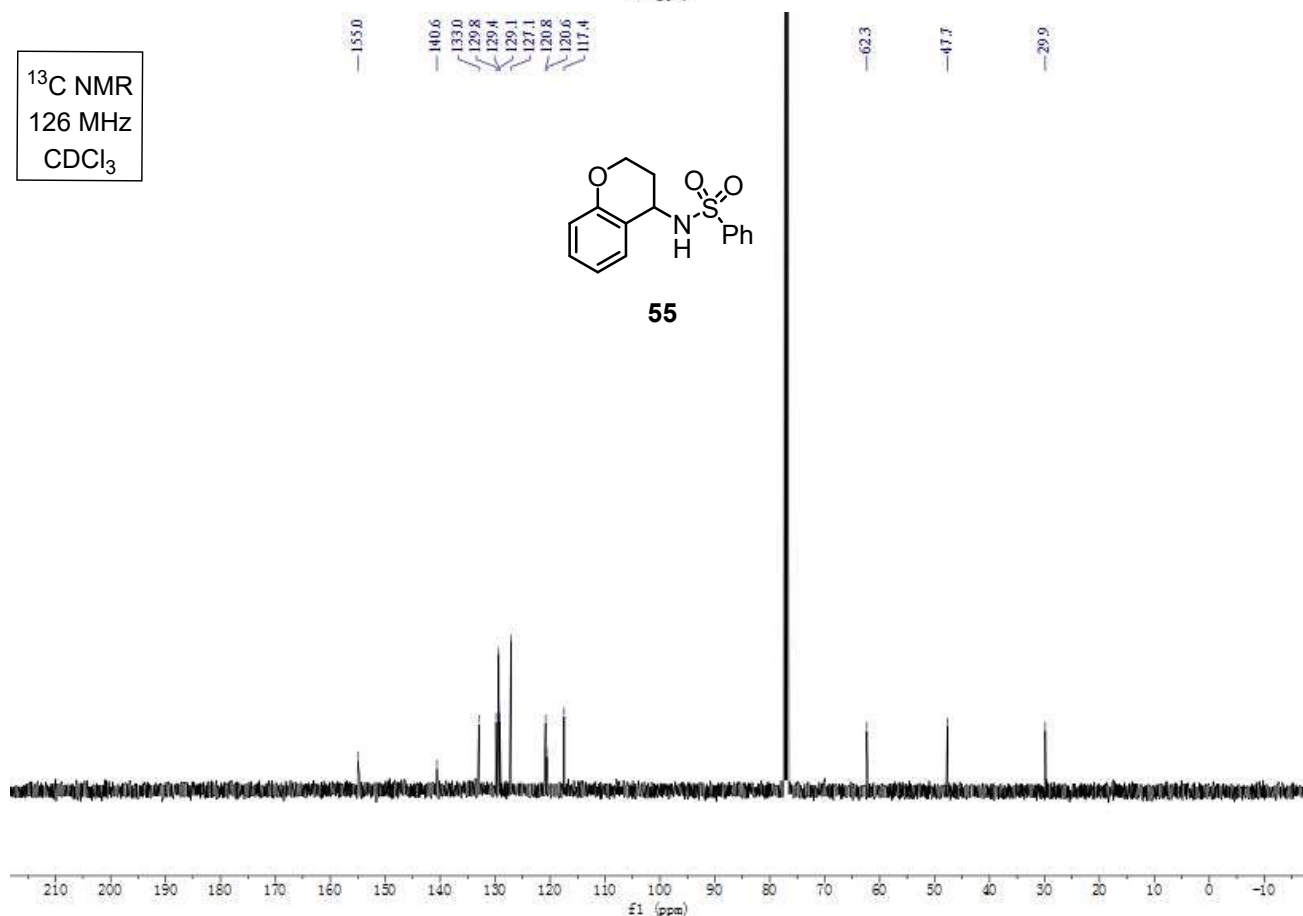
**55**



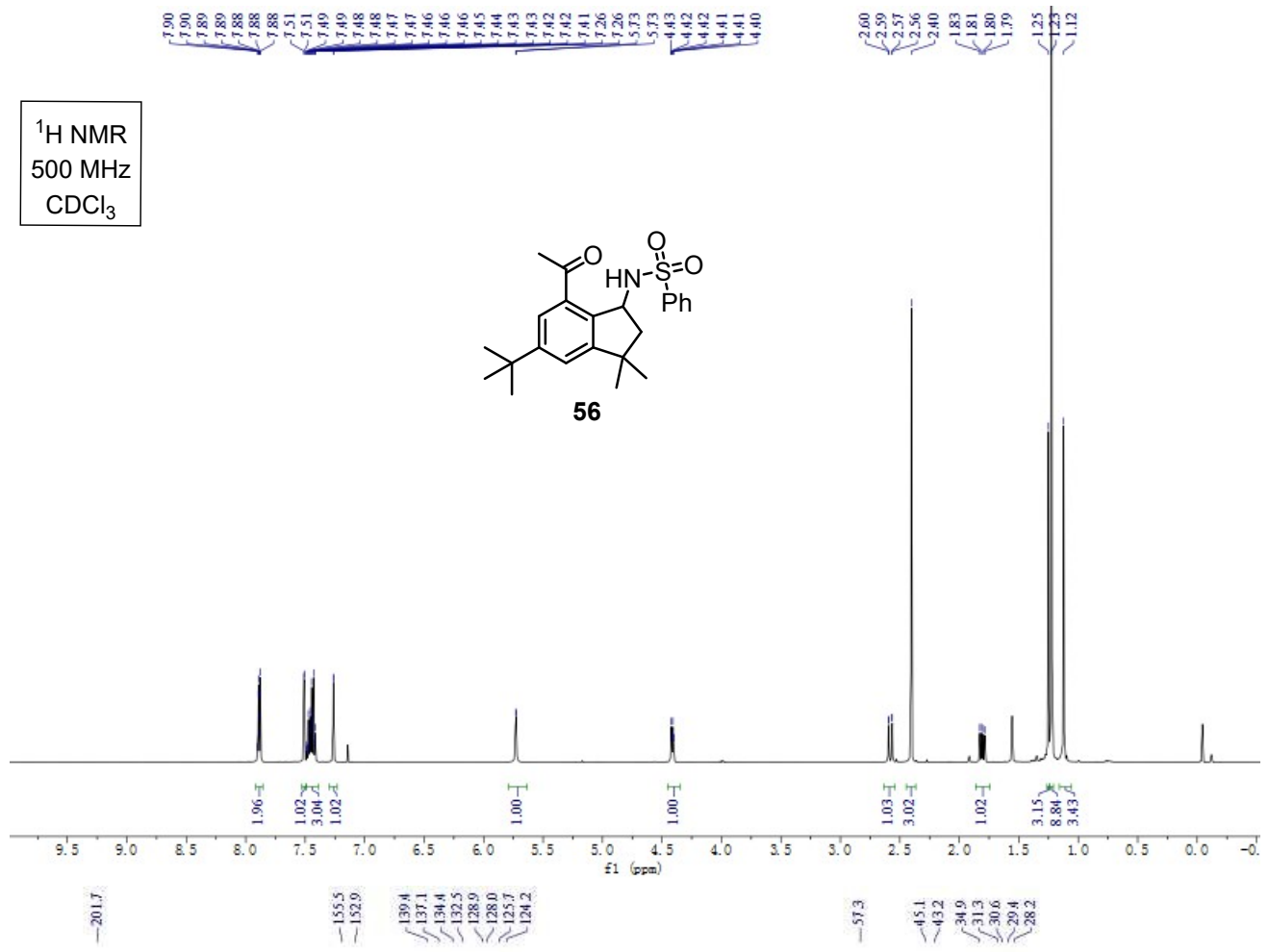
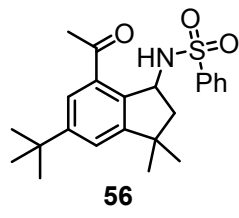
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



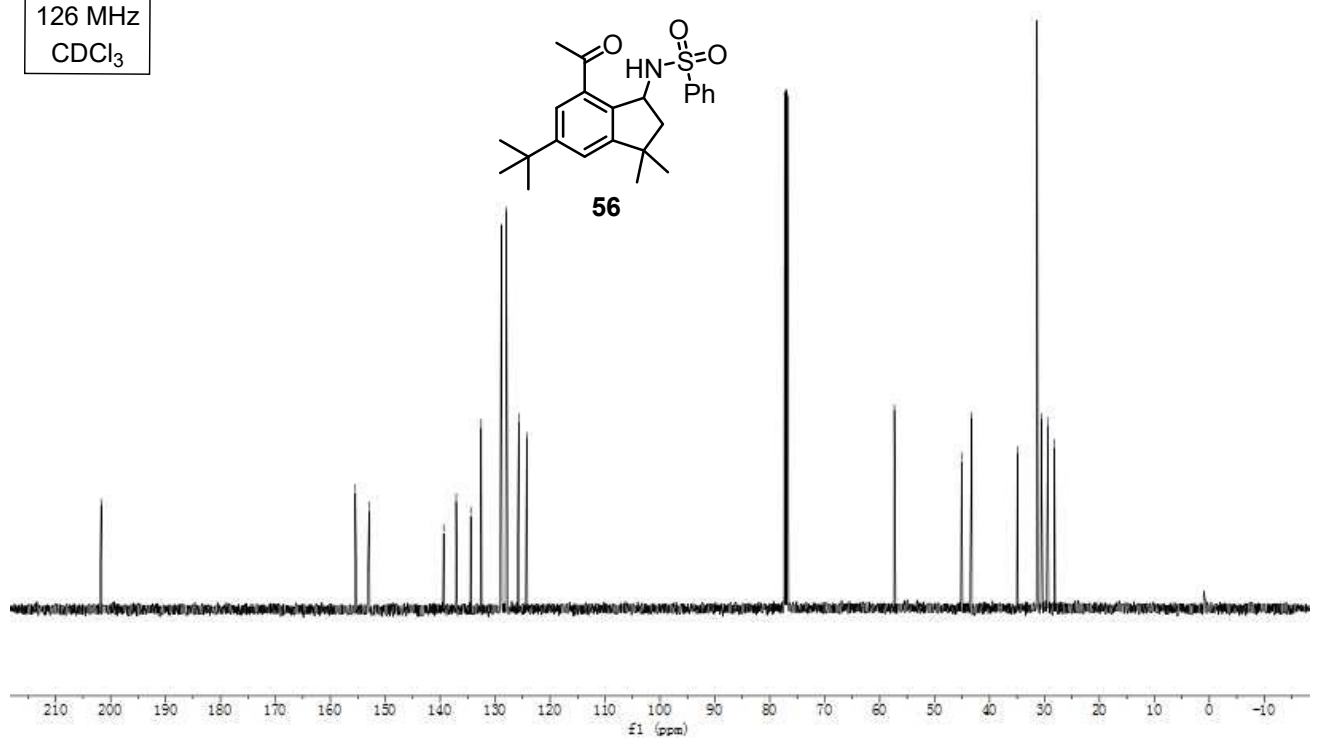
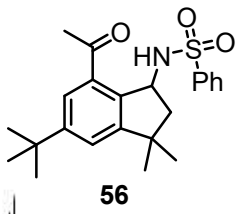
**55**



<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

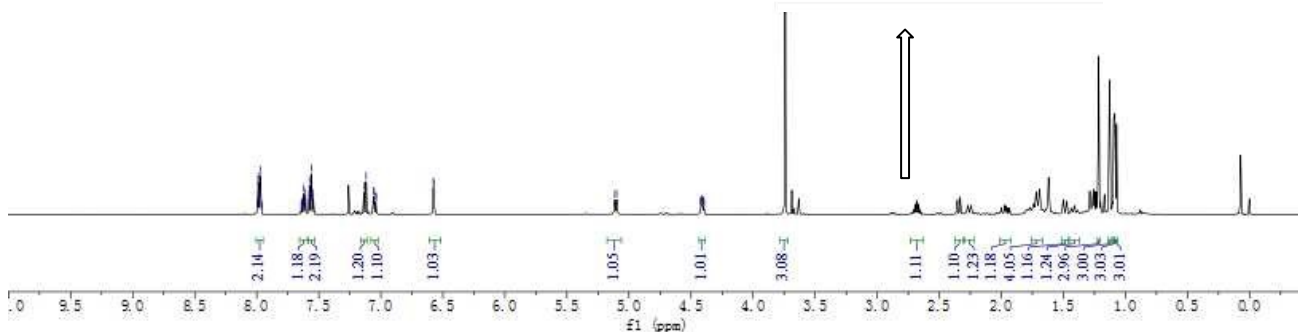
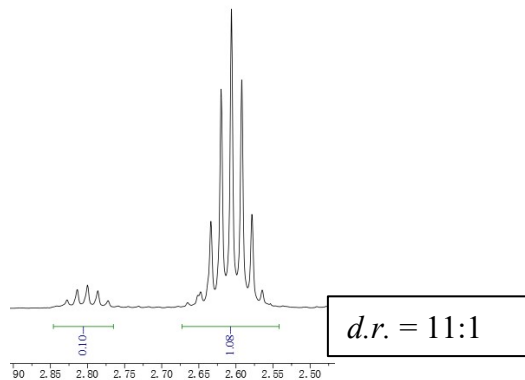
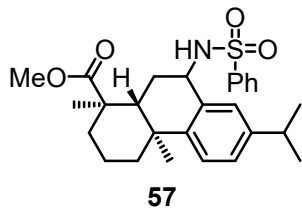


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

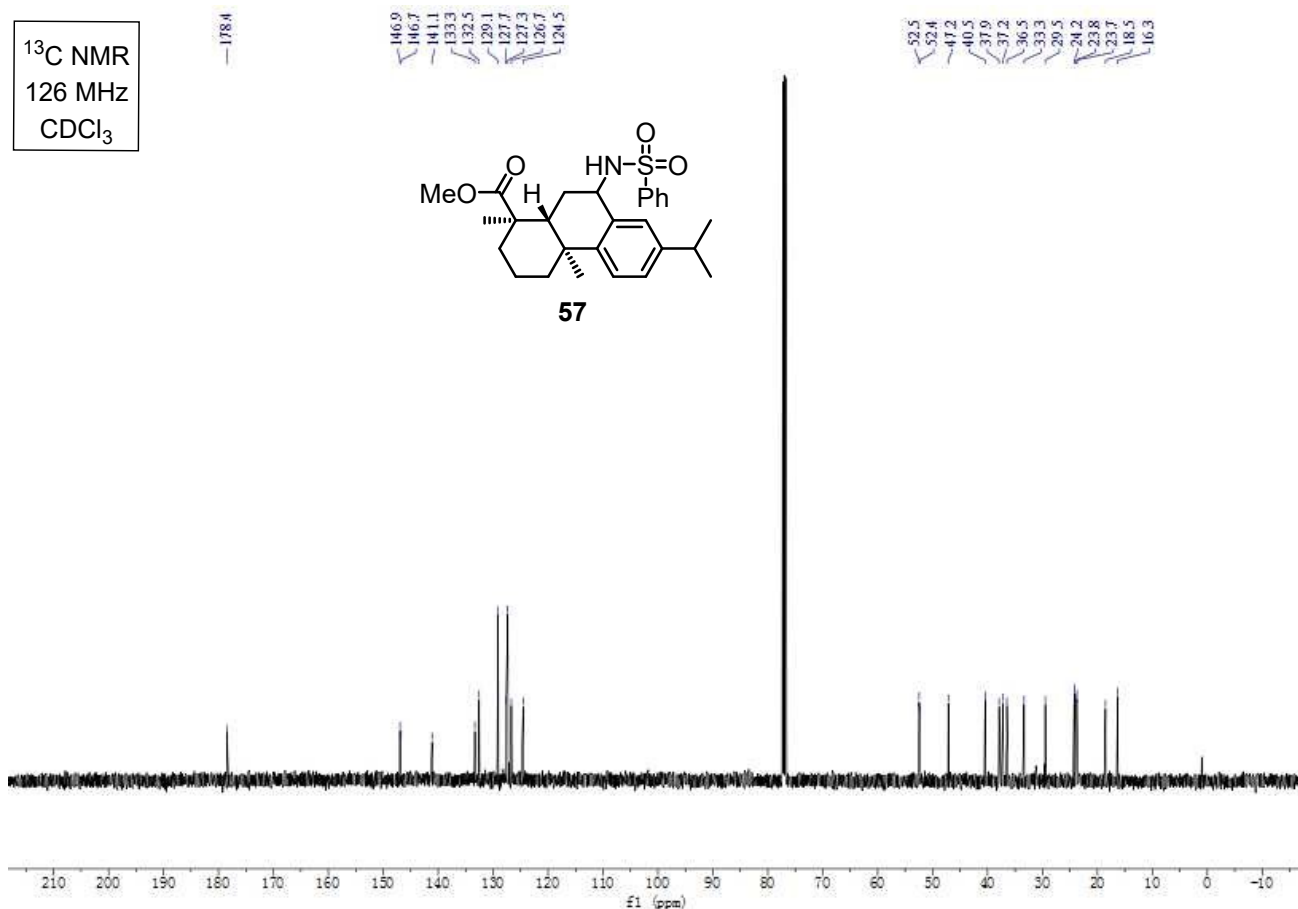
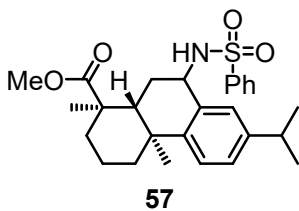


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4.82  
4.82  
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4.40  
4.39  
4.39

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



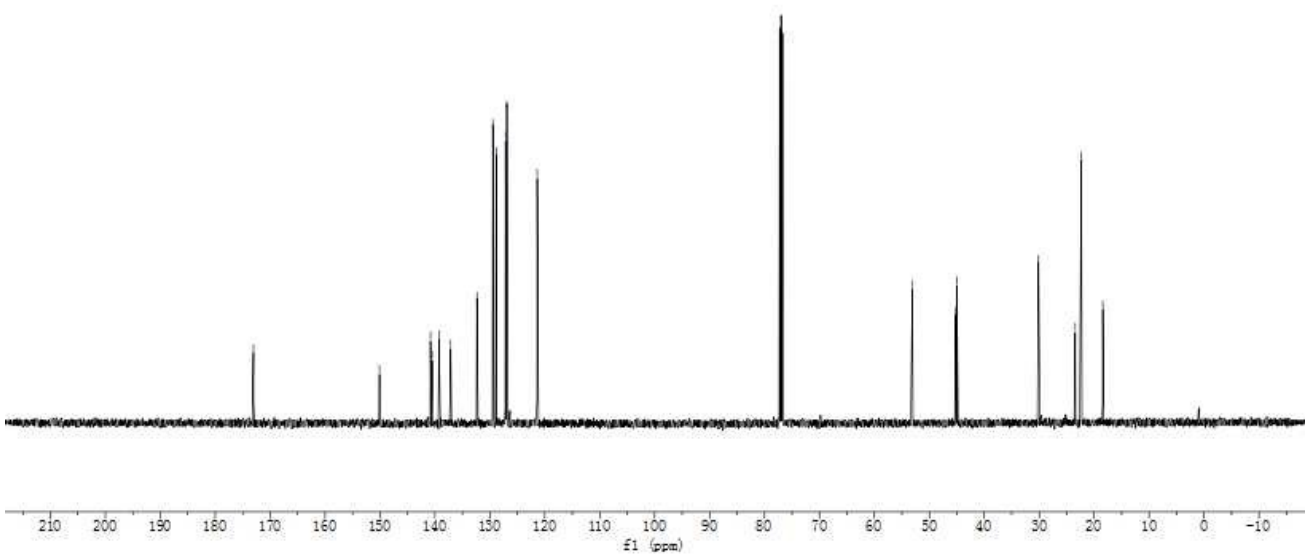
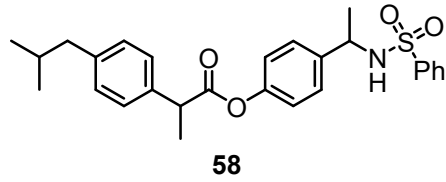
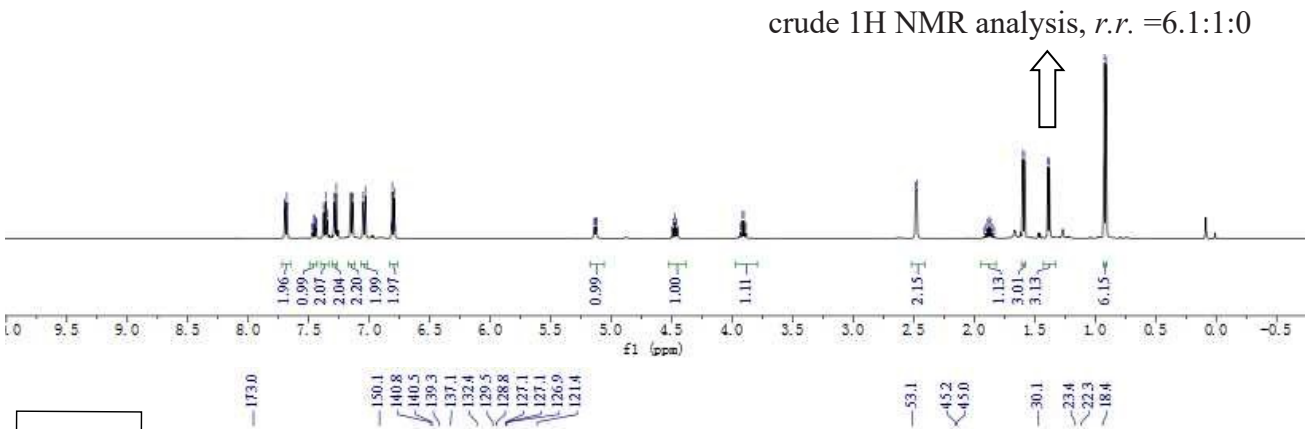
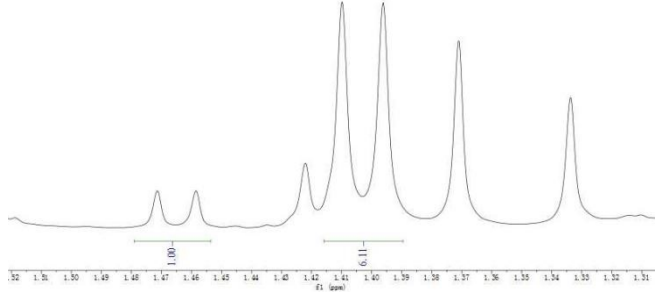
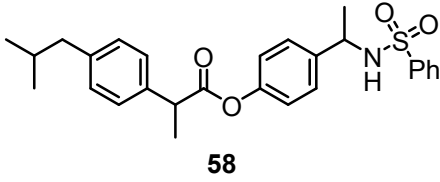
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



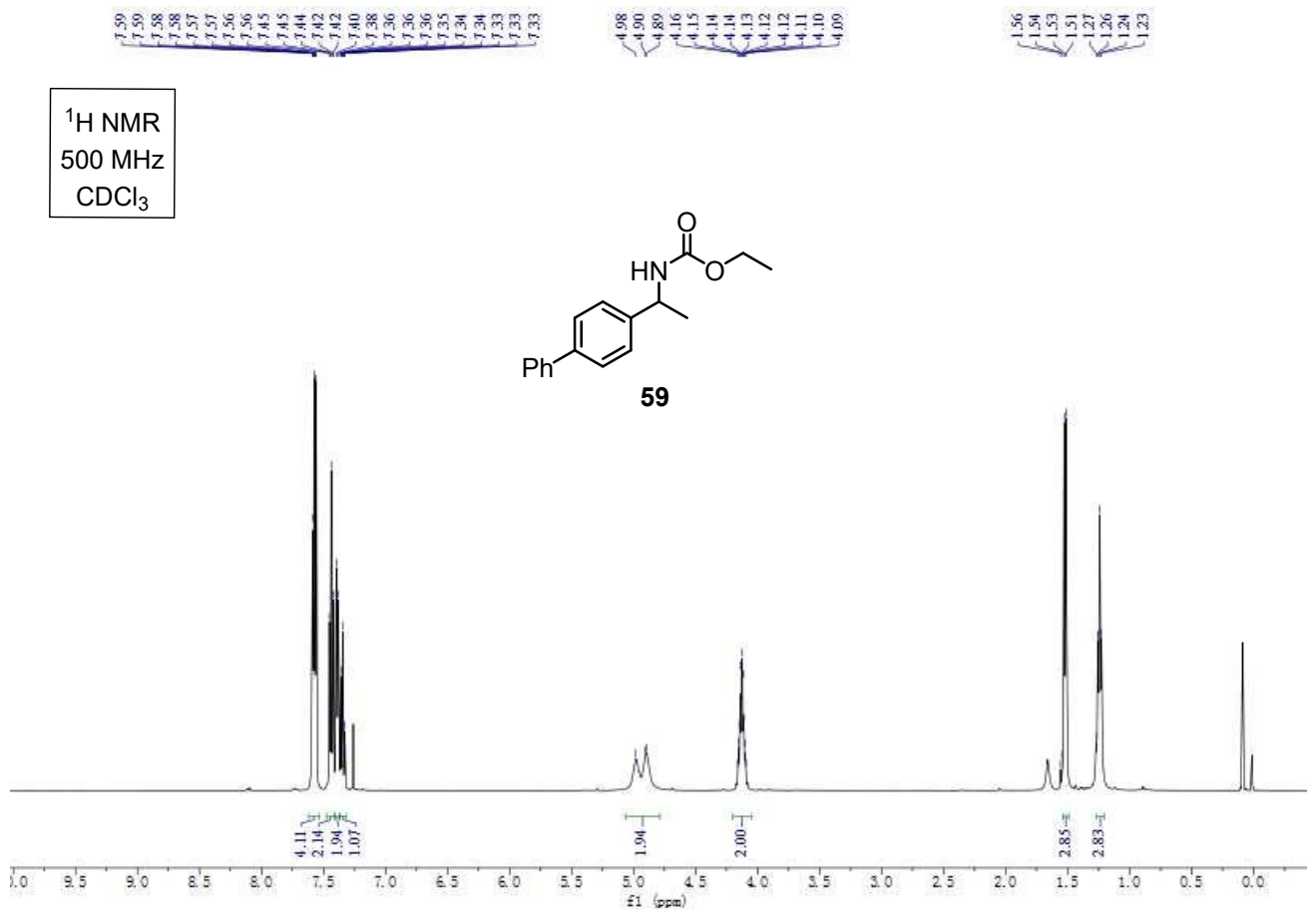
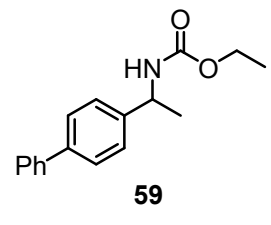
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3.89

2.49  
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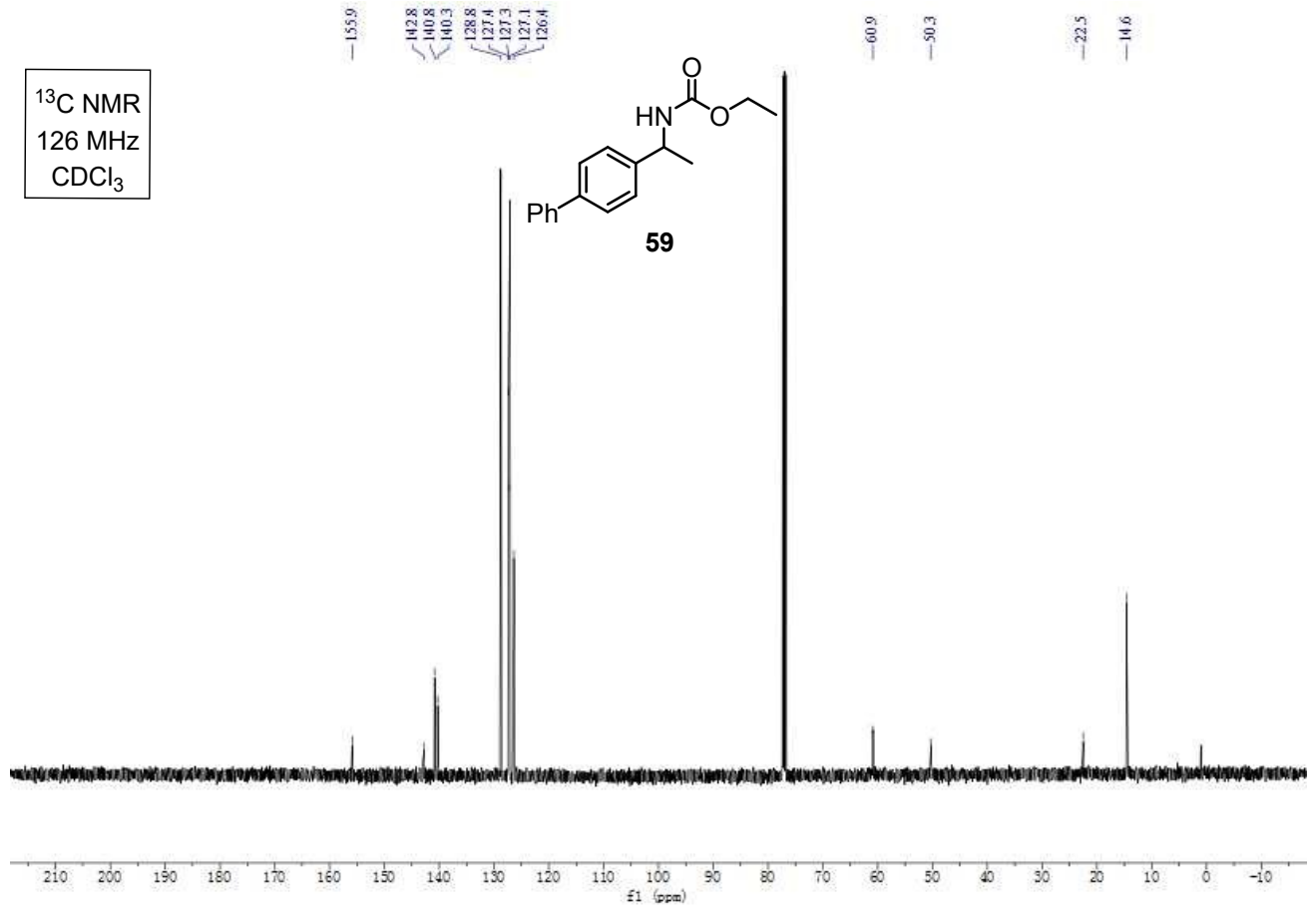
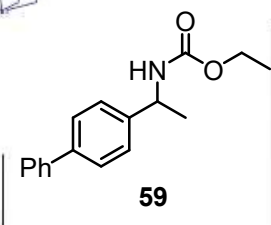
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

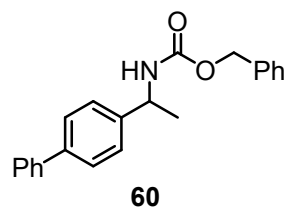


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

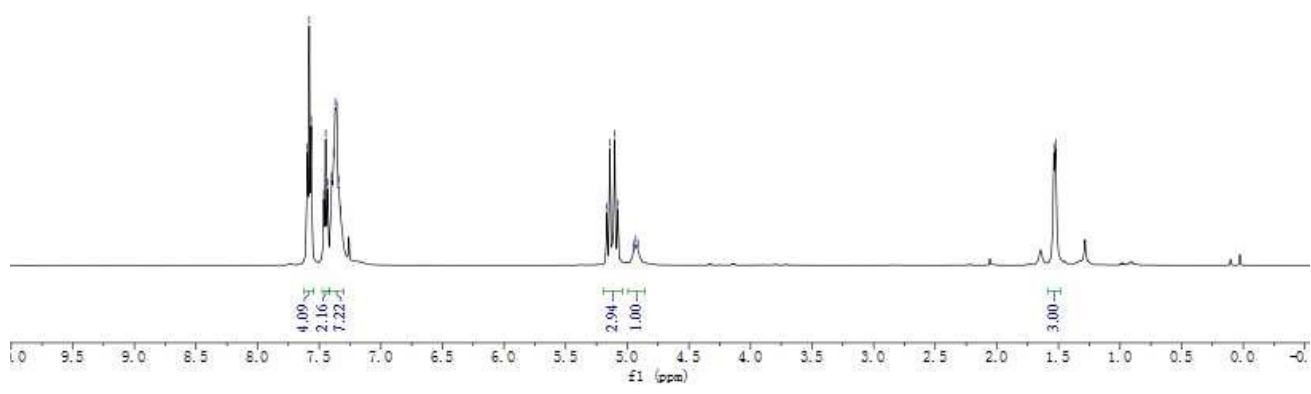




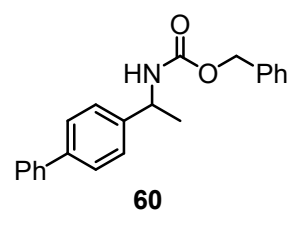
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500 MHz  
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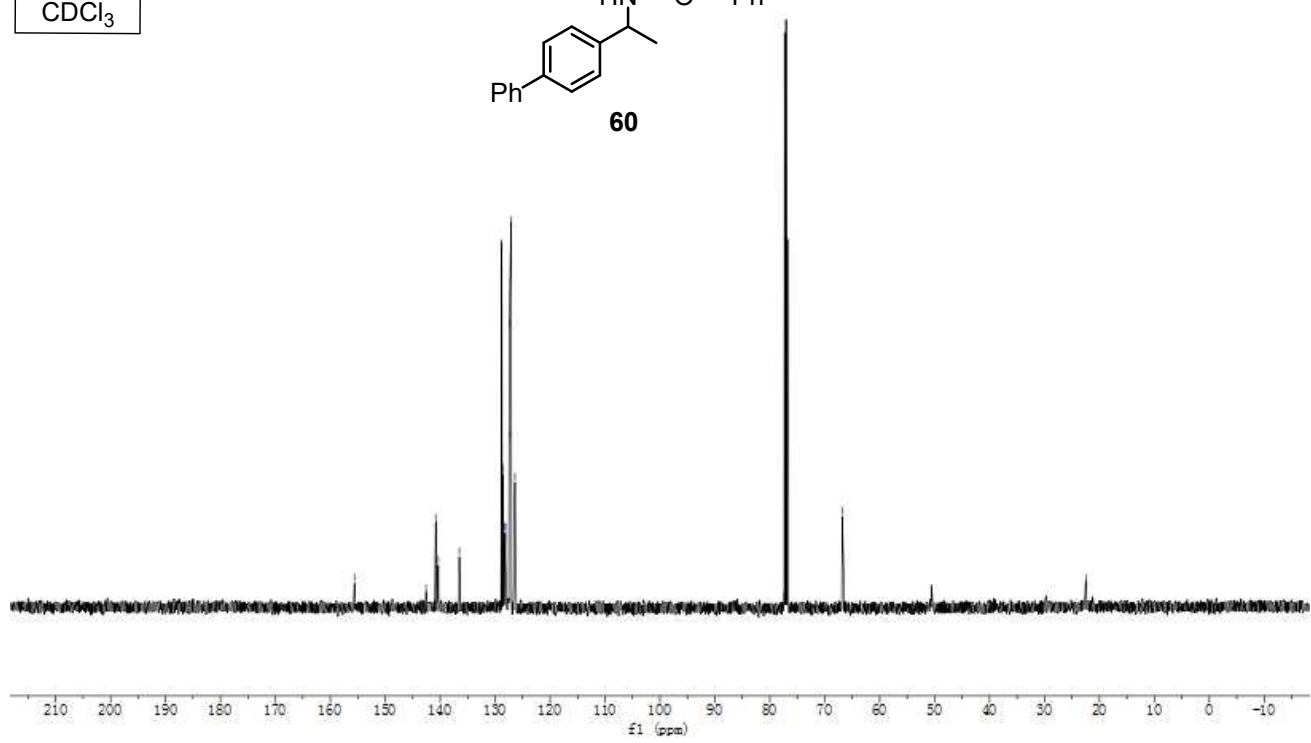
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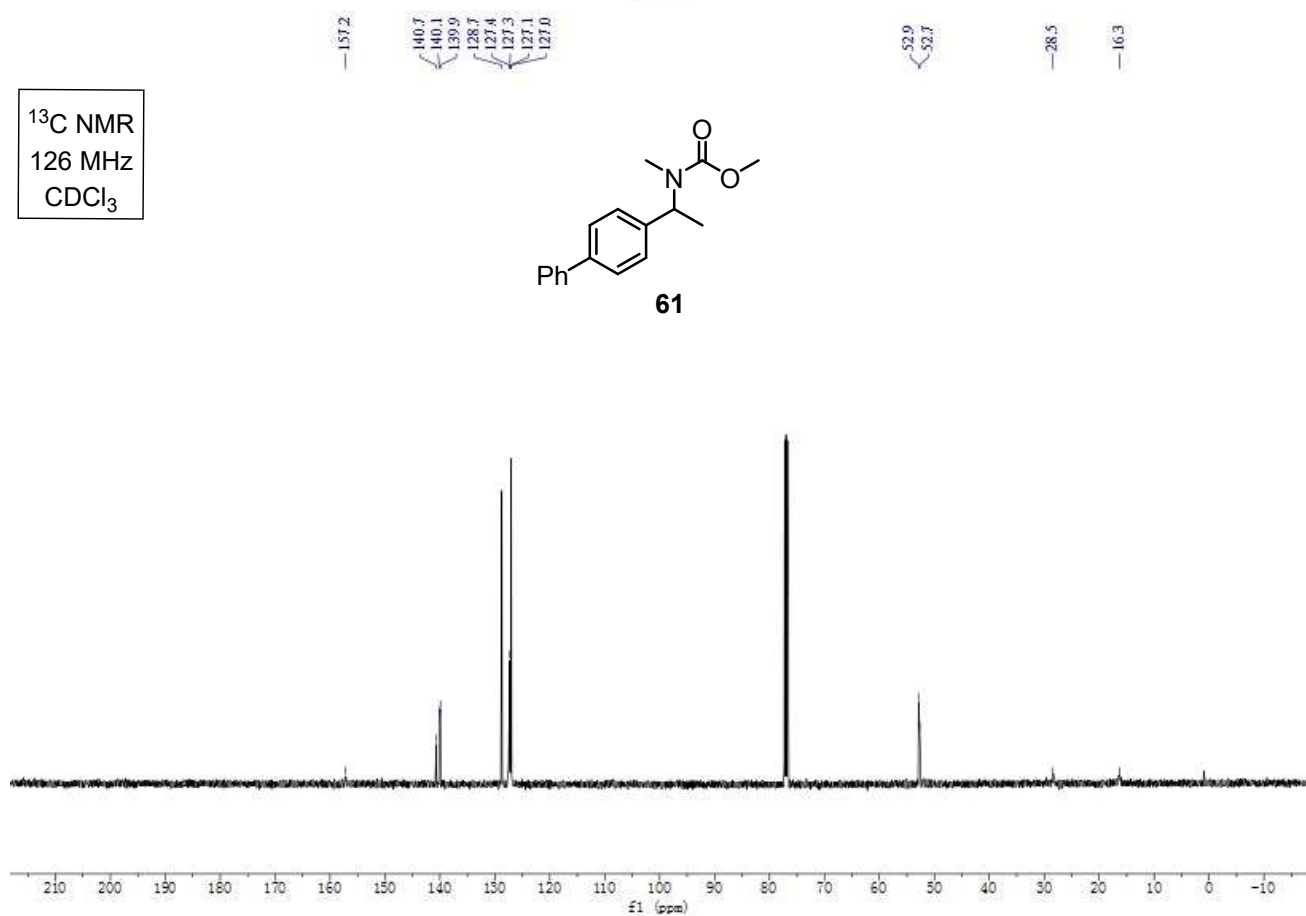
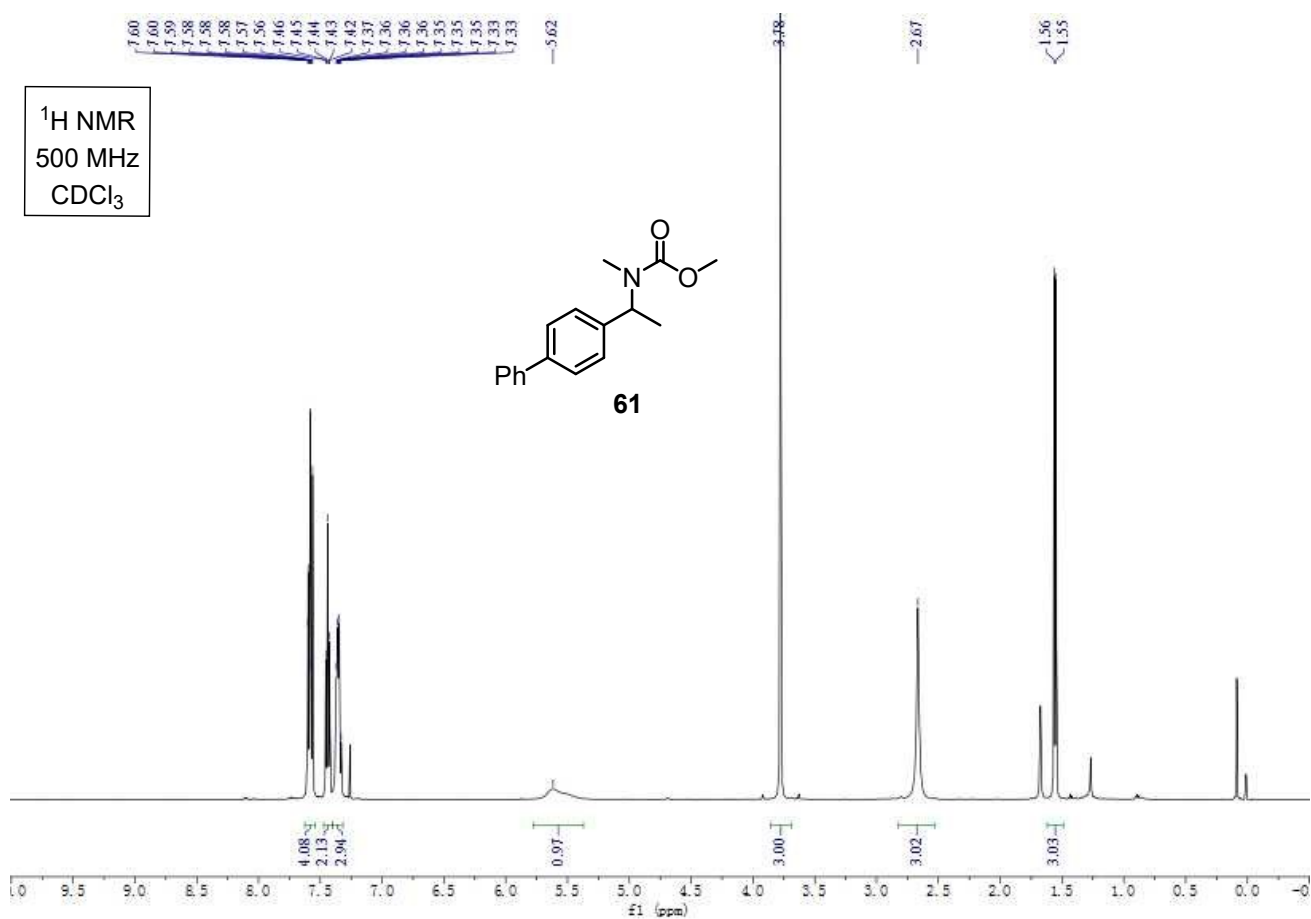


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



155.6, 142.6, 140.8, 140.3, 136.5, 128.8, 128.5, 128.2, 128.1, 127.4, 127.3, 127.1, 126.4, 66.8, 50.6, 22.5



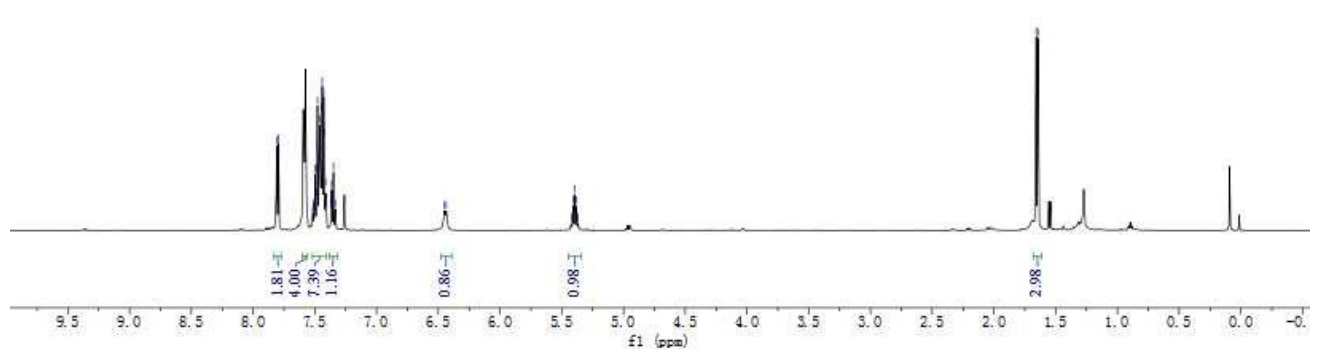
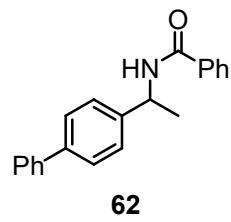


7.81  
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6.84

5.42  
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5.40  
5.38  
5.37

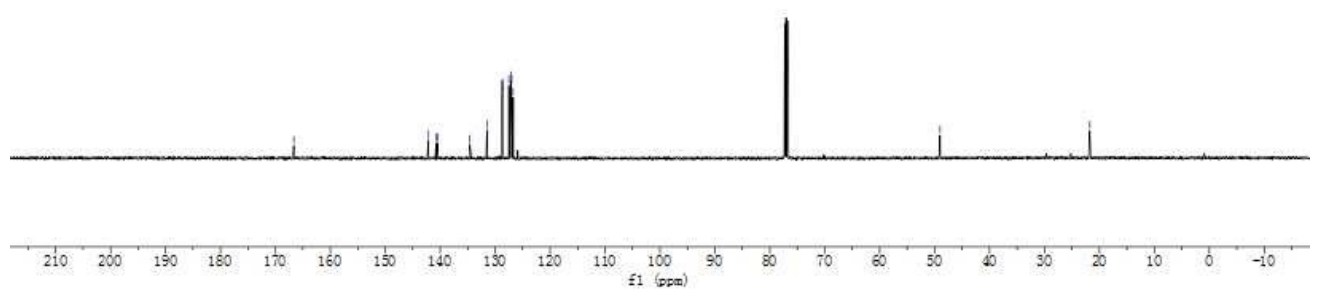
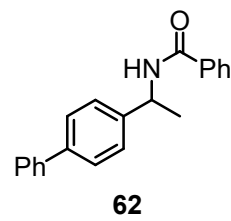
1.66  
1.64

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



166.7  
142.2  
140.8  
140.5  
134.6  
131.5  
128.8  
128.6  
127.5  
127.3  
127.1  
127.0  
126.7  
49.0  
21.7

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

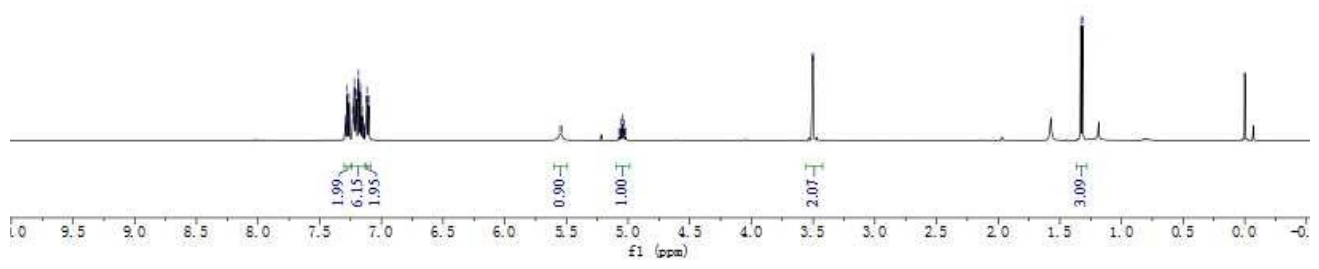
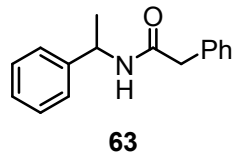


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5.54  
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5.06  
5.05  
5.03  
5.02

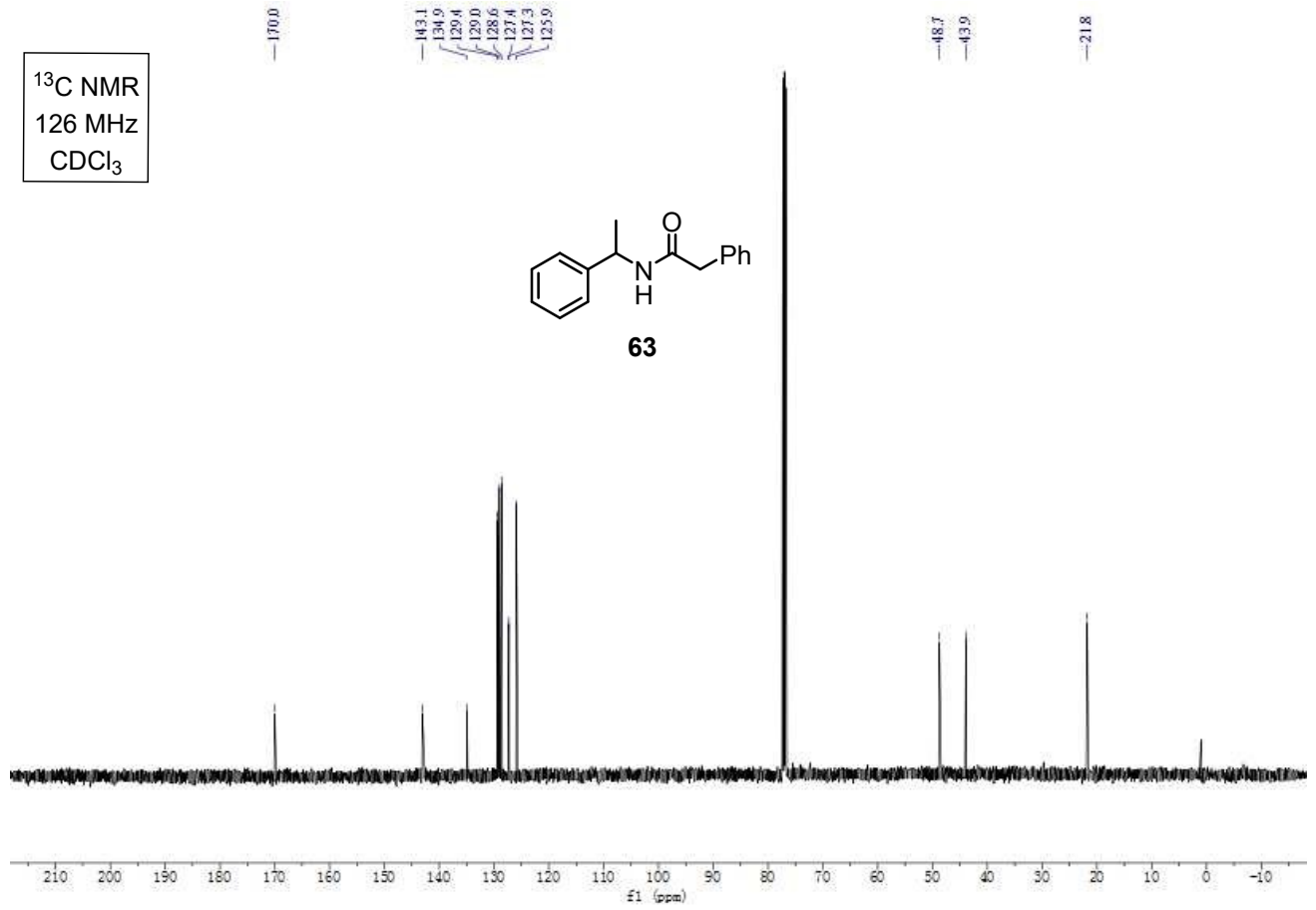
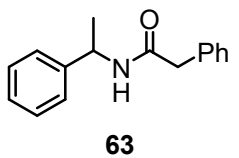
3.50  
3.50

1.33  
1.32

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

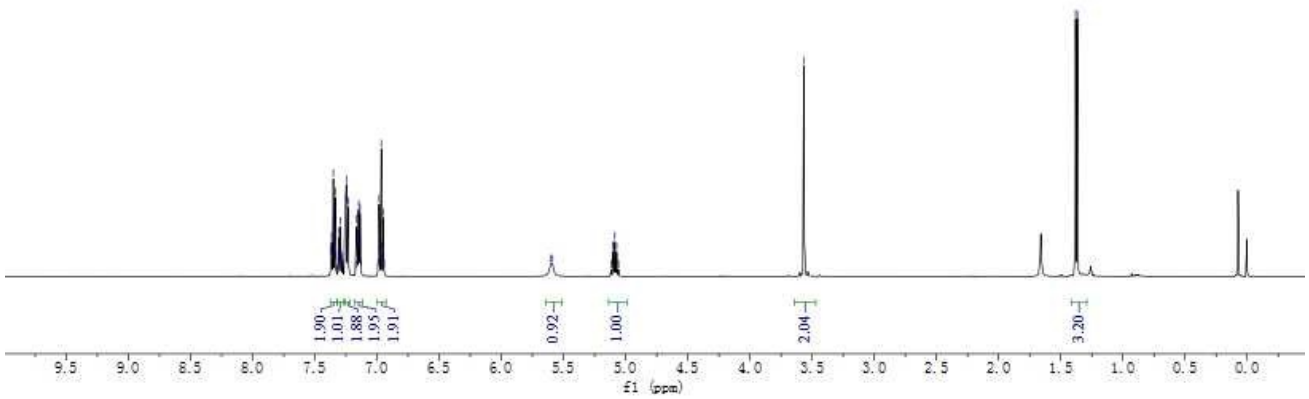
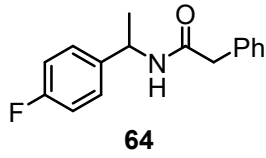


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5.07  
5.06

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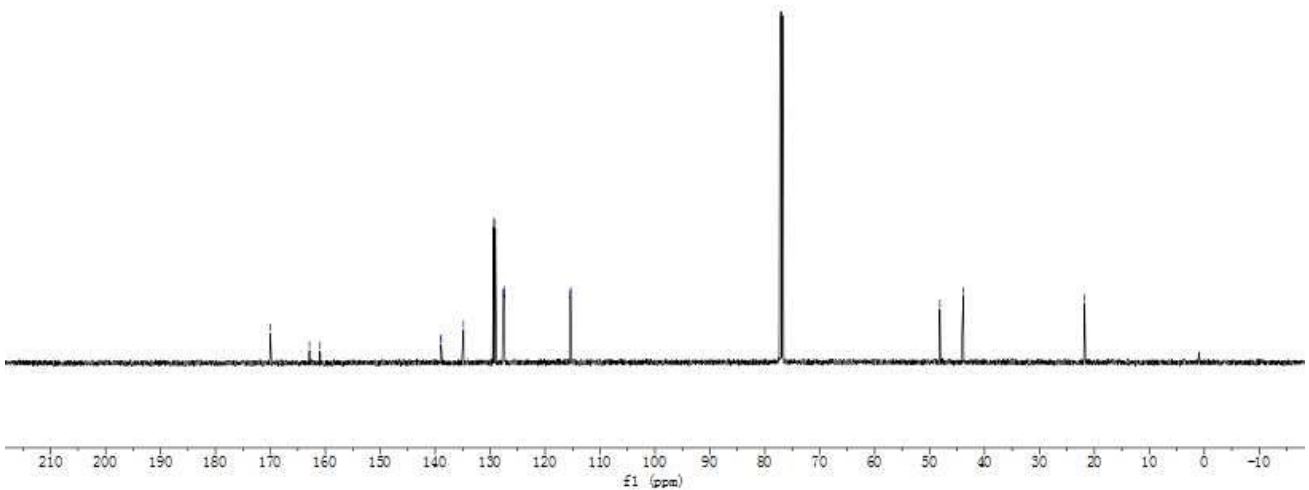
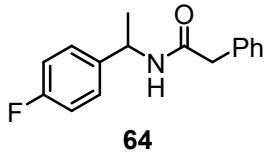
1.38  
1.37

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



170.0  
162.9  
161.0  
138.9  
138.8  
134.8  
129.3  
129.0  
127.6  
127.5  
127.4  
115.5  
115.3  
48.1  
43.9  
21.8

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

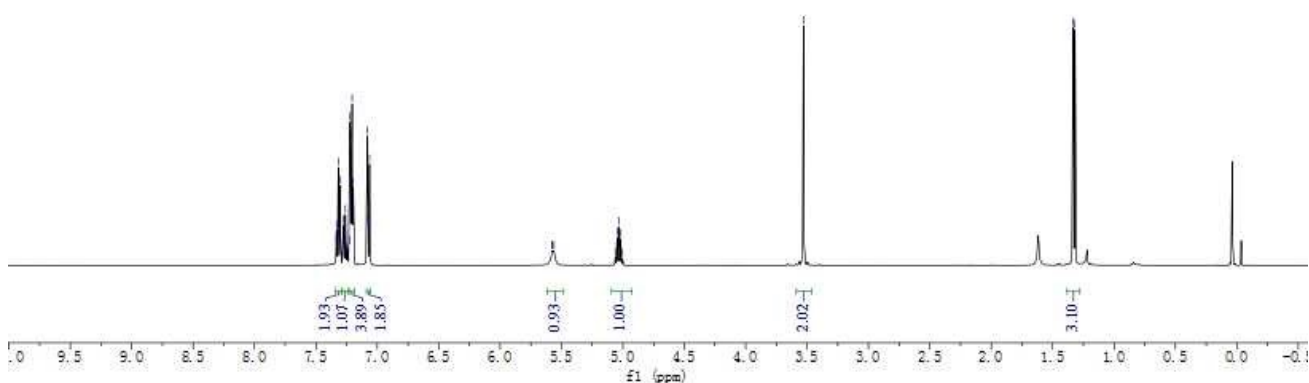
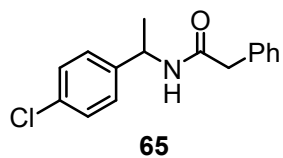


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5.57  
5.56  
5.06  
5.05  
5.03  
5.02  
5.00

-3.53

1.33  
1.32

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



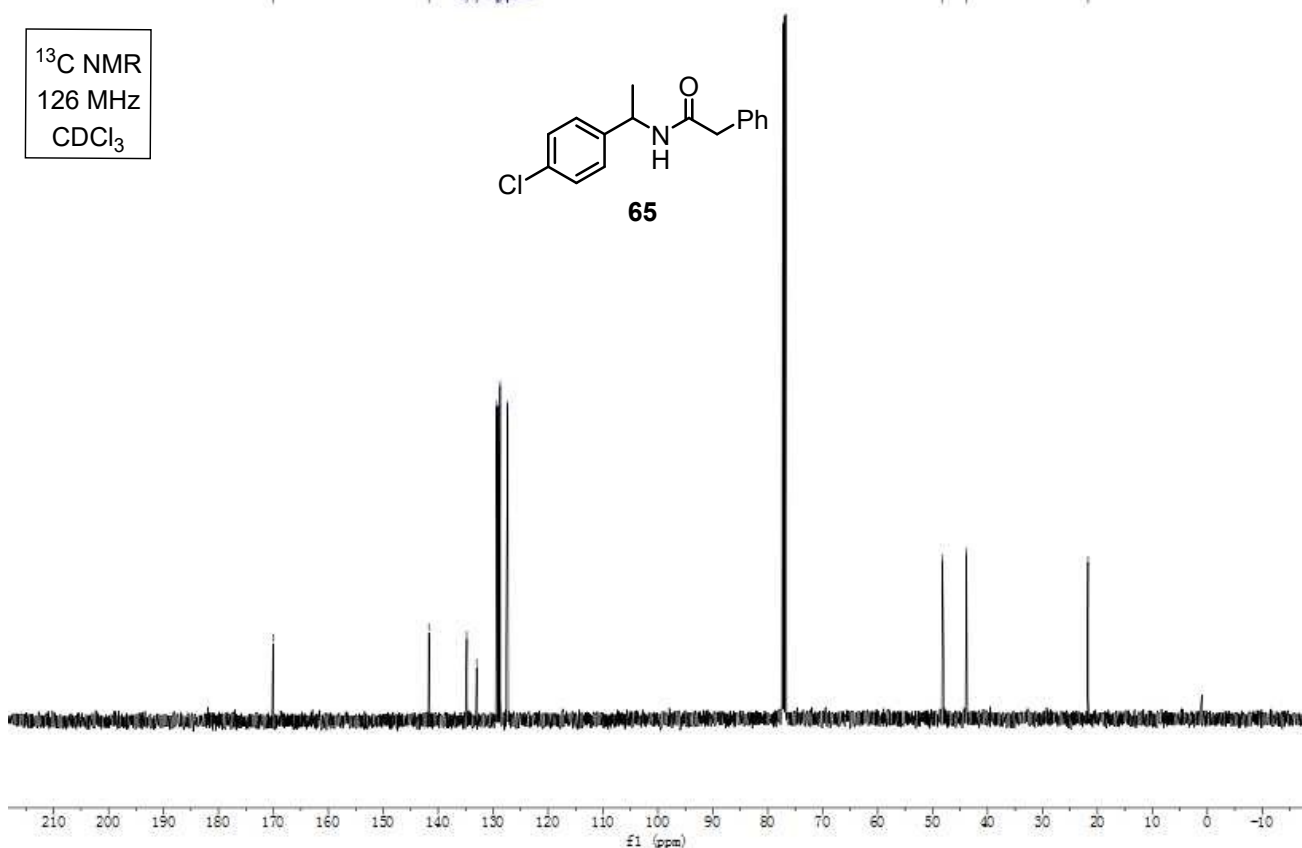
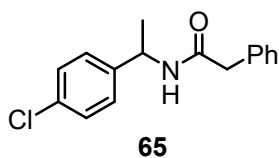
170.1

141.7  
134.8  
133.0  
129.3  
129.1  
128.7  
127.4  
127.3

48.2  
43.8

21.7

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

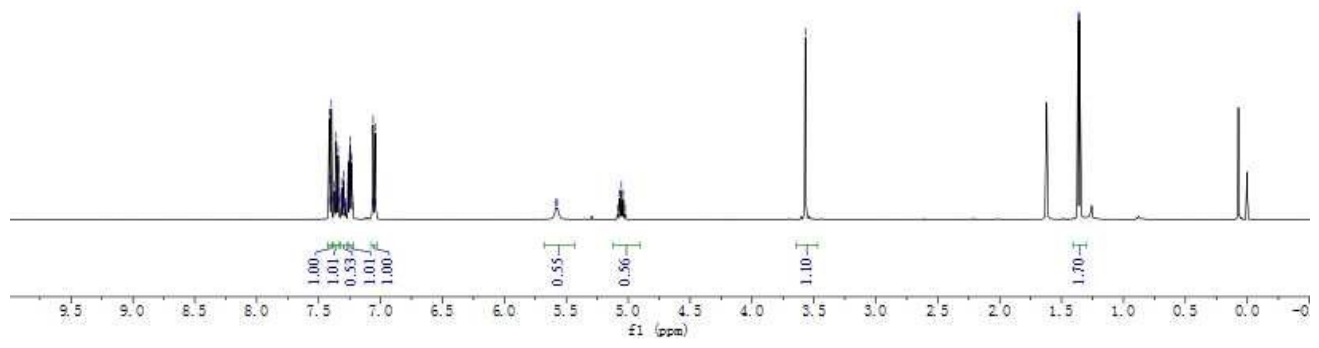
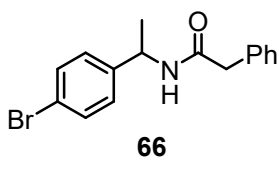


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5.04  
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5.03

-3.57

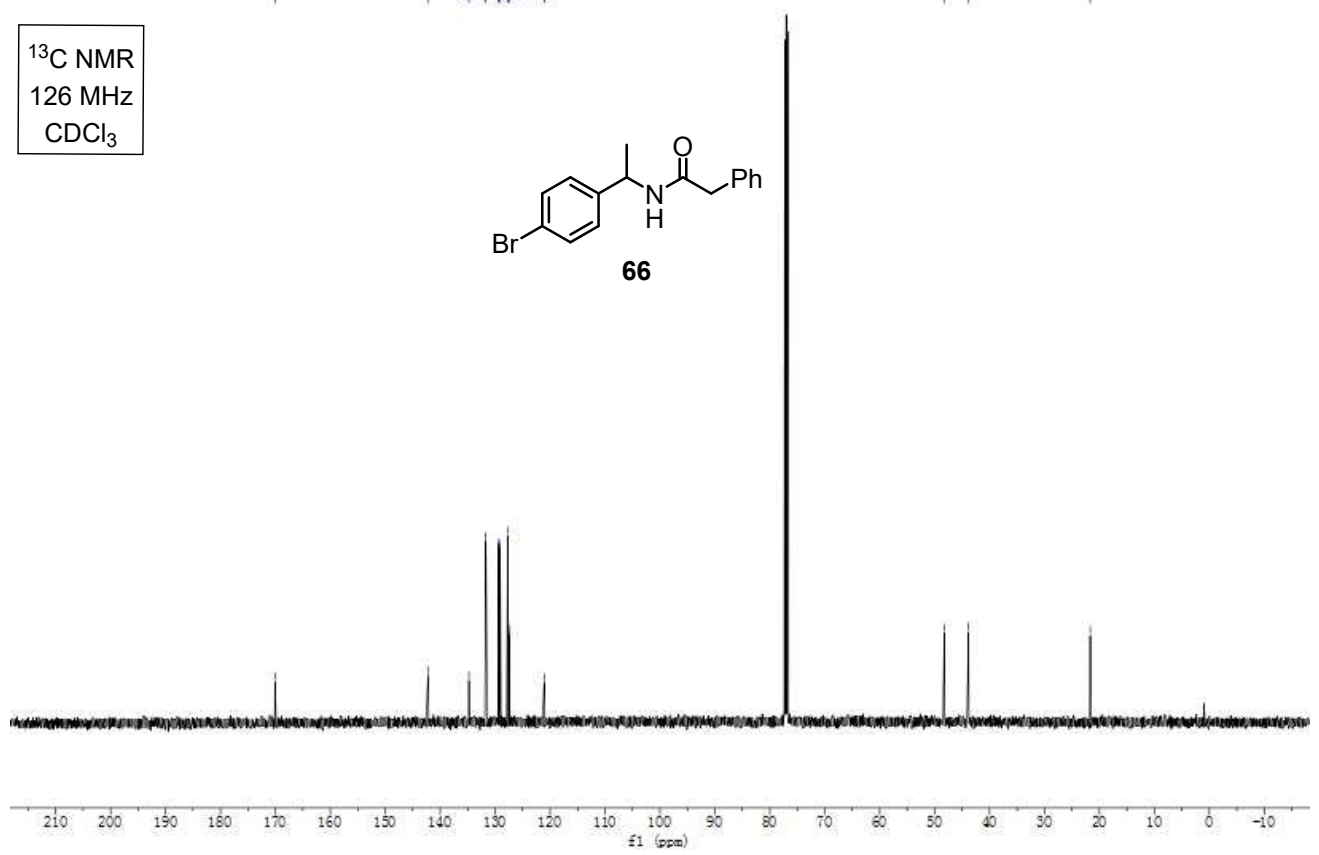
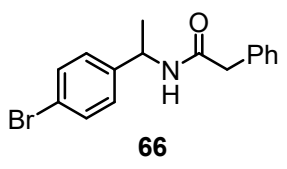
<1.37  
<1.35

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



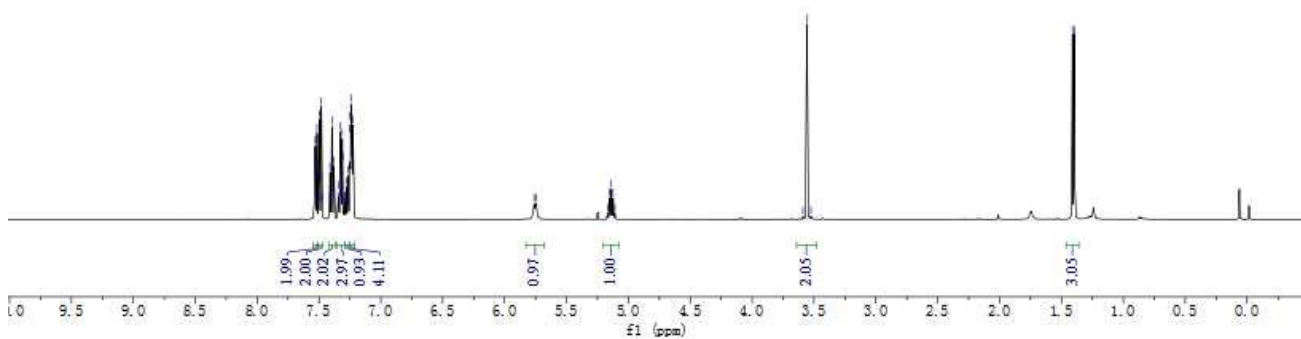
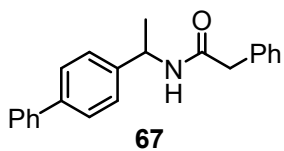
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142.2  
134.8  
131.7  
129.3  
129.1  
127.7  
127.5  
121.1  
48.3  
43.9  
21.7

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

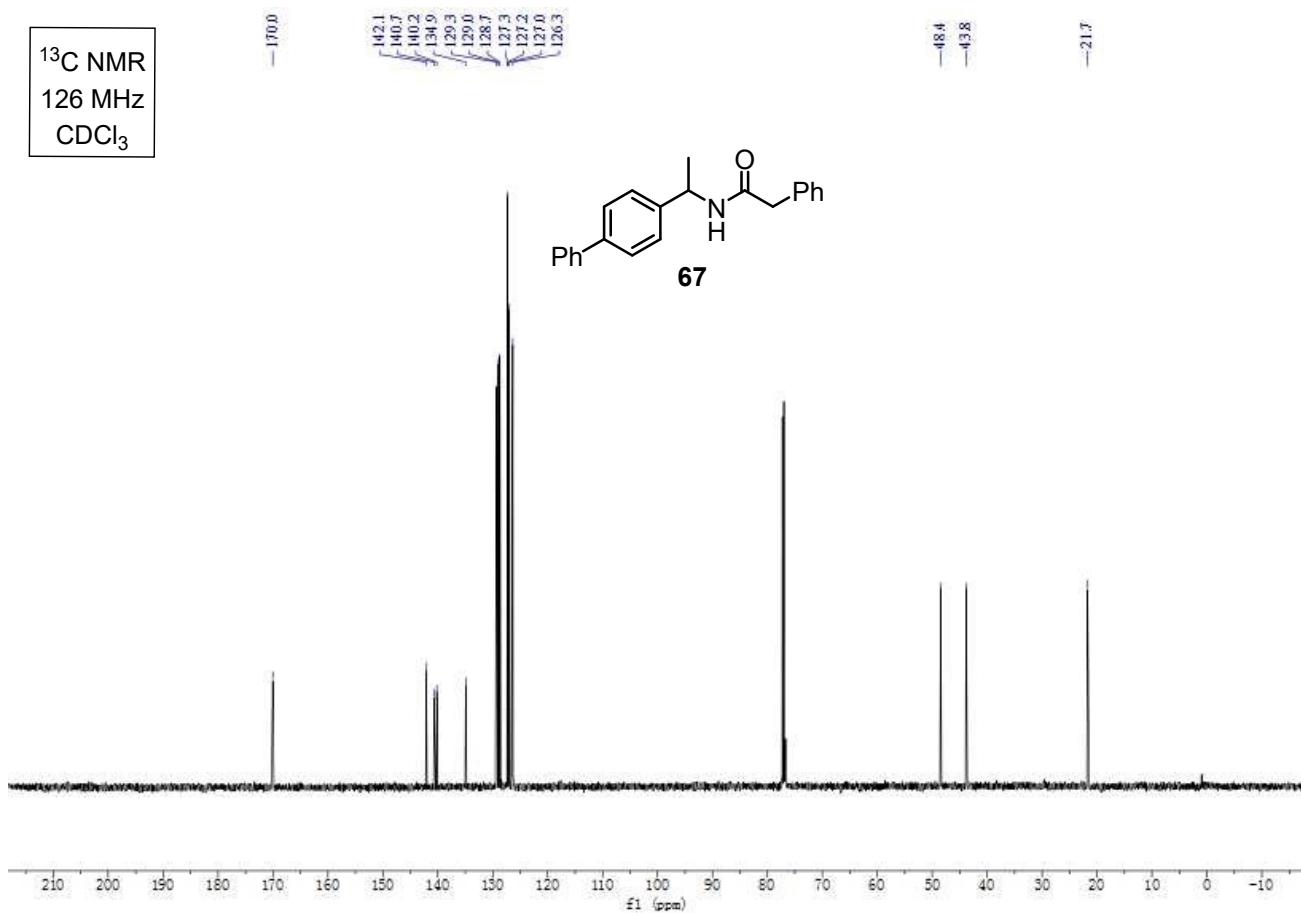
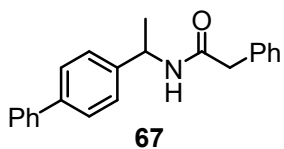


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1.41  
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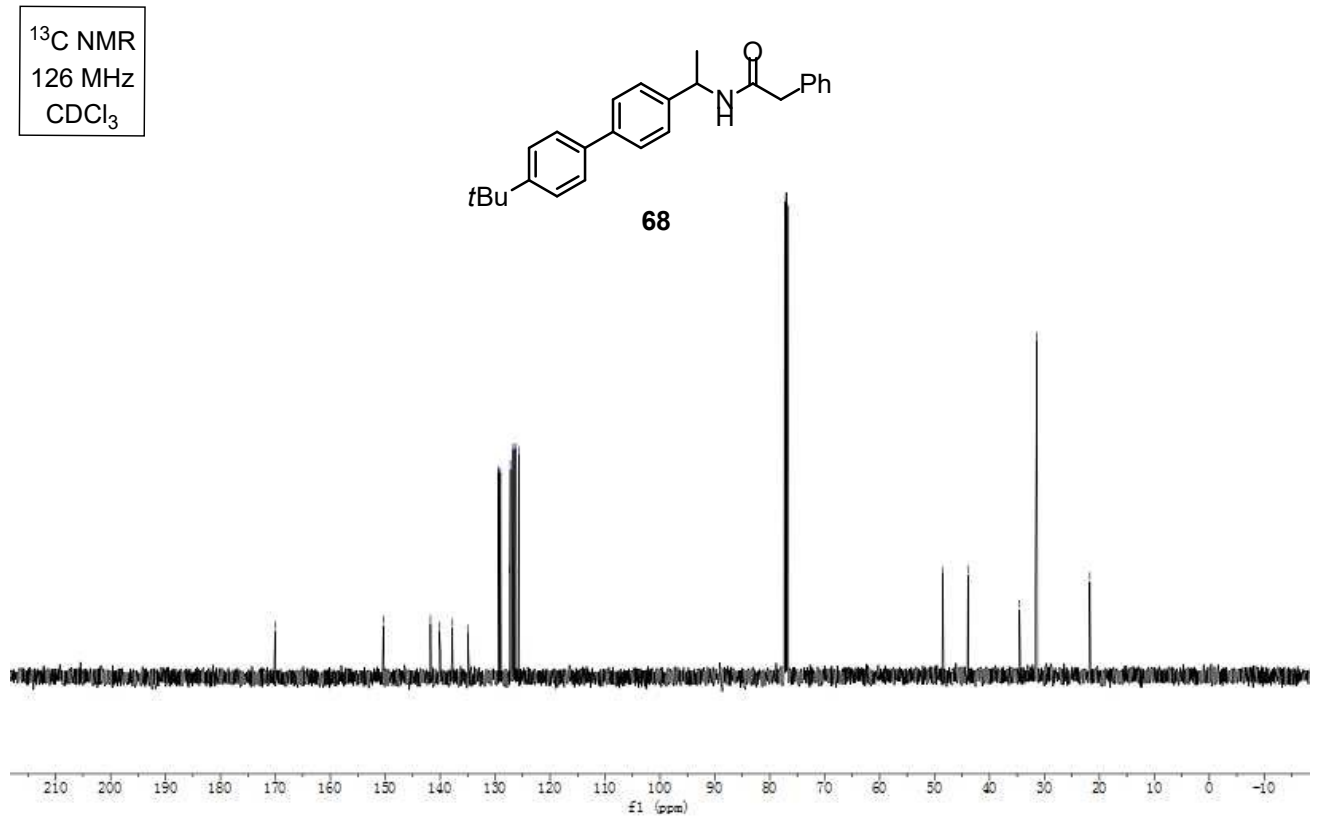
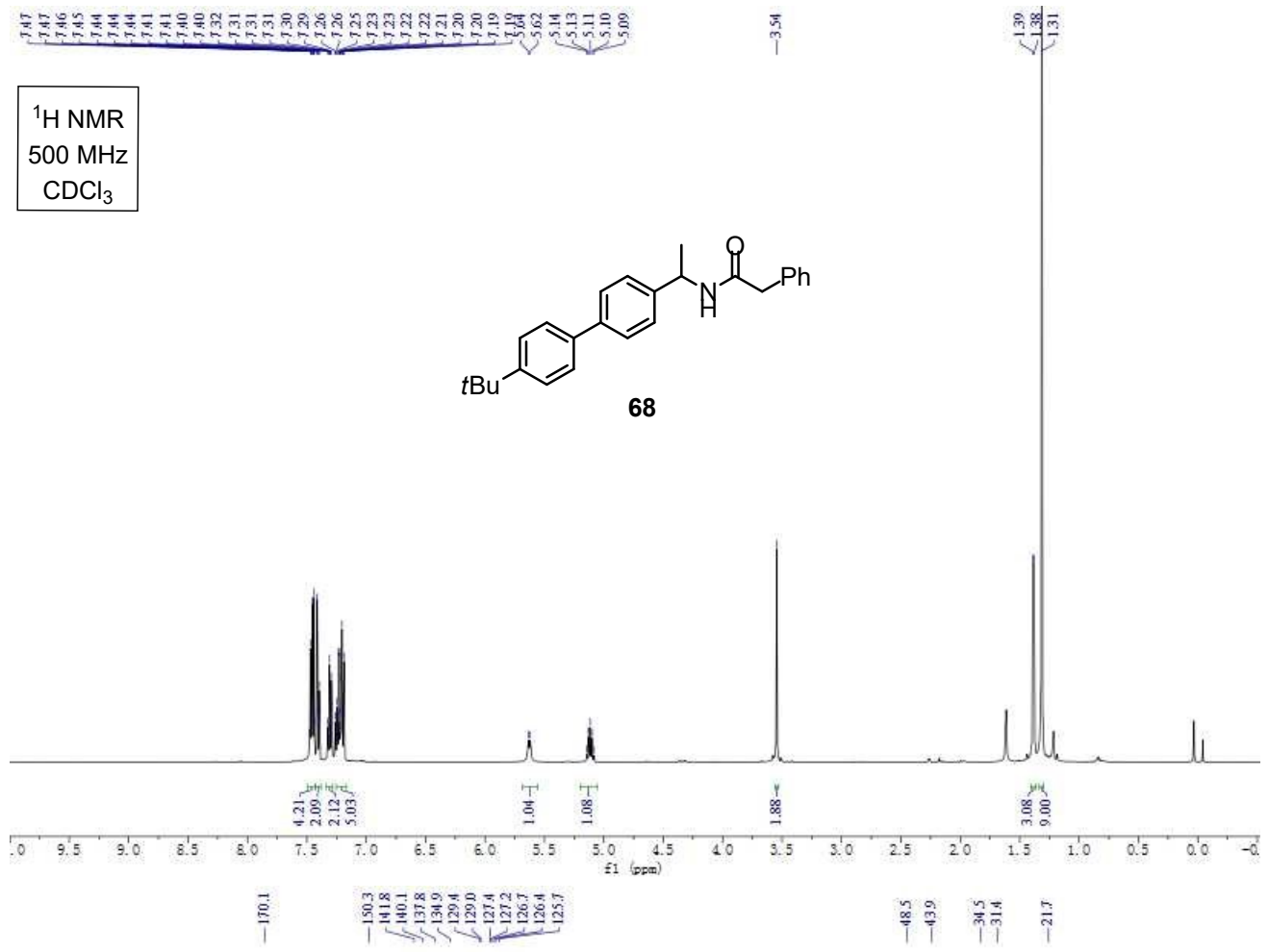
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>





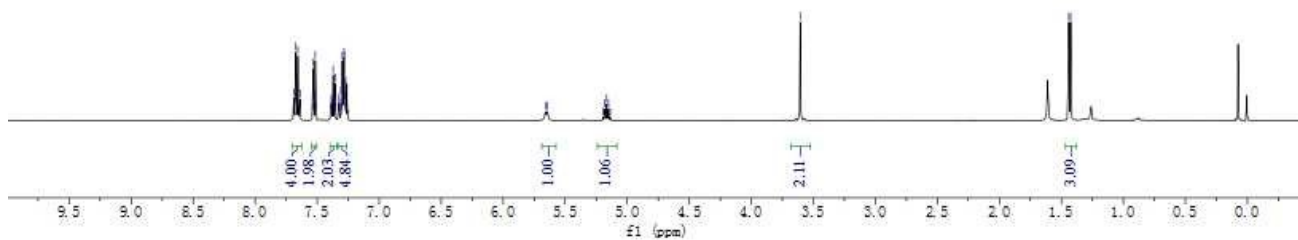
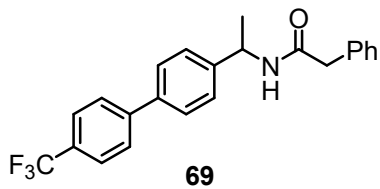


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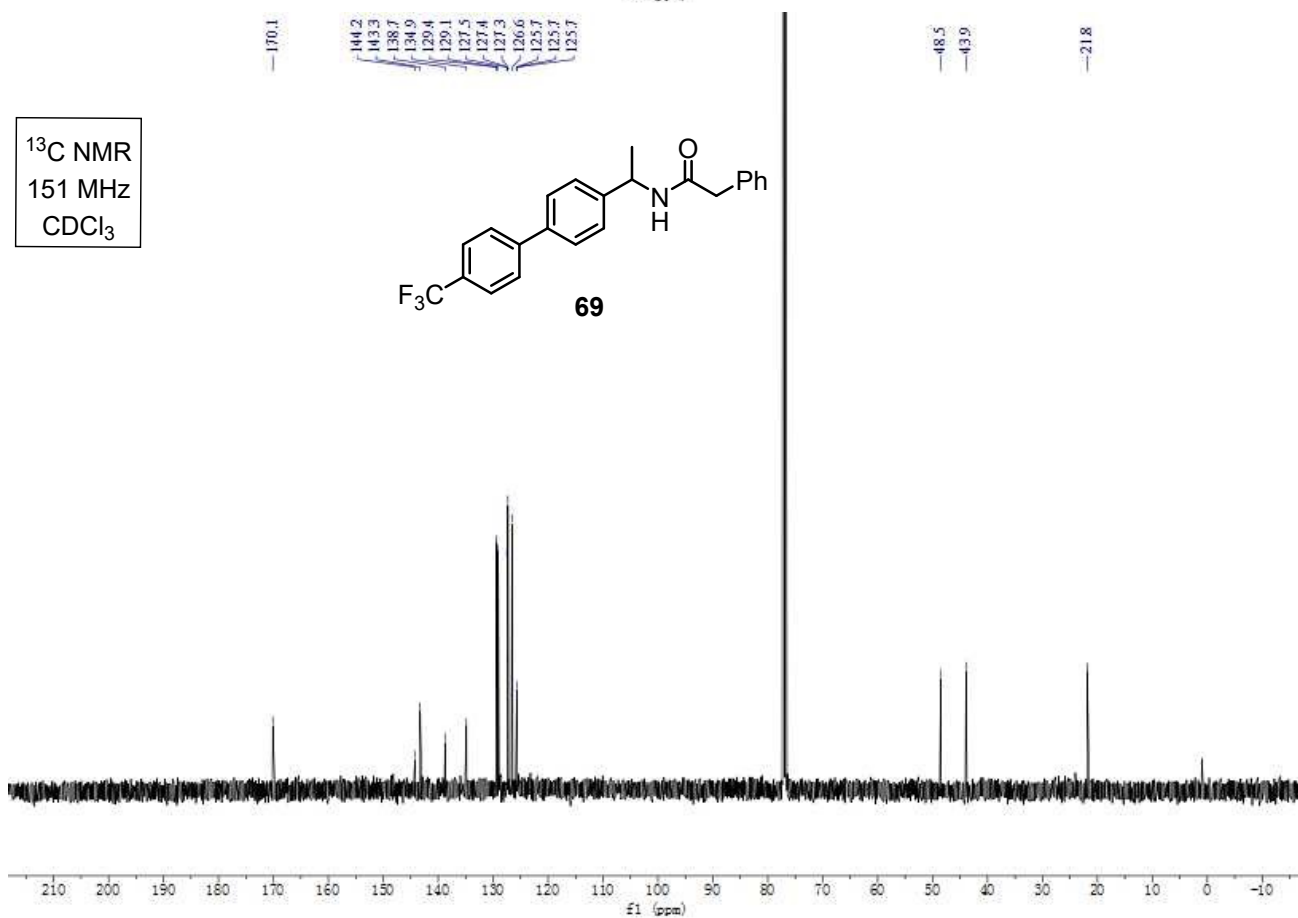
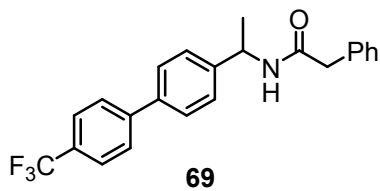
-3.61

1.44  
1.43

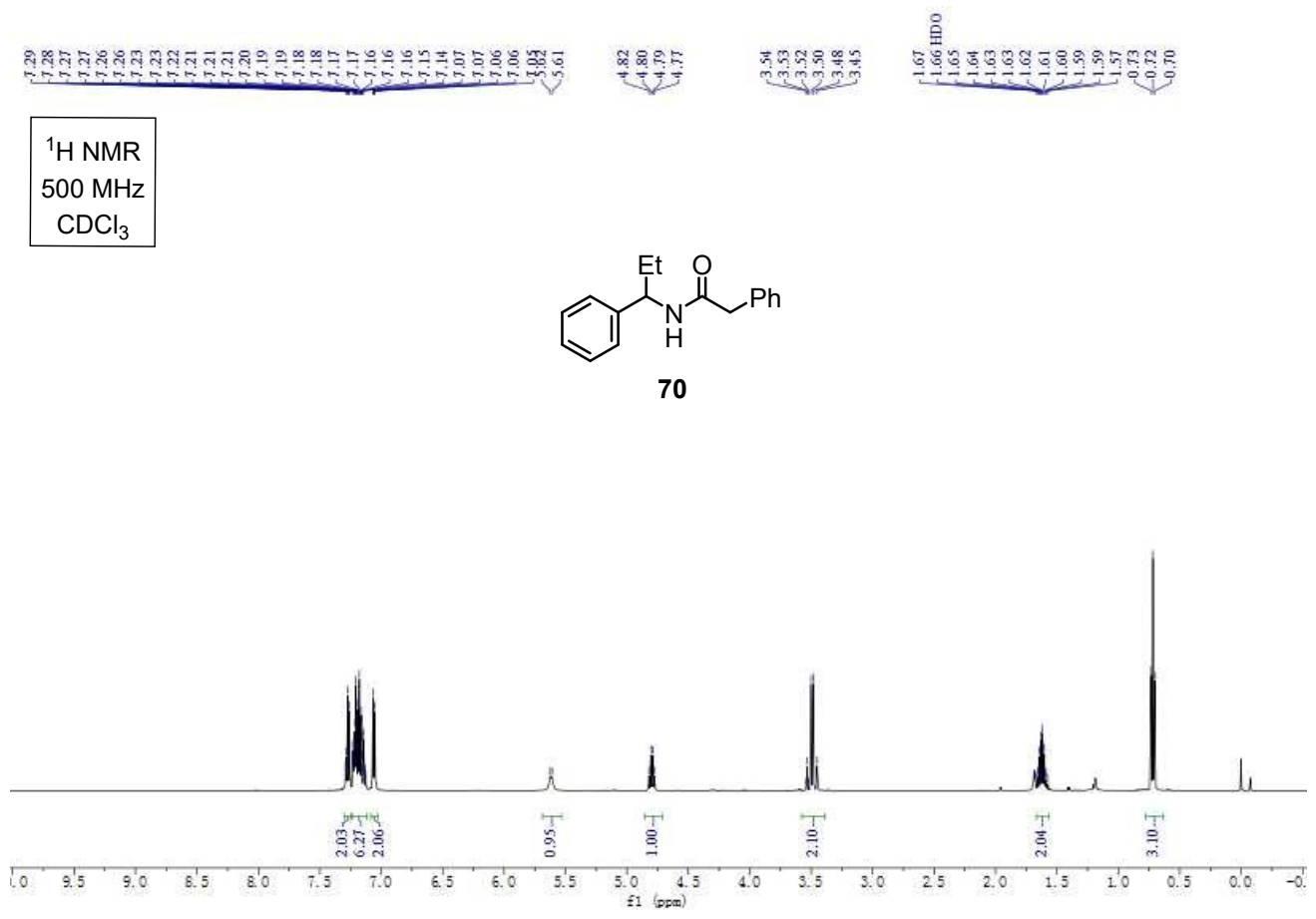
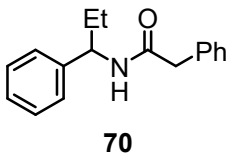
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



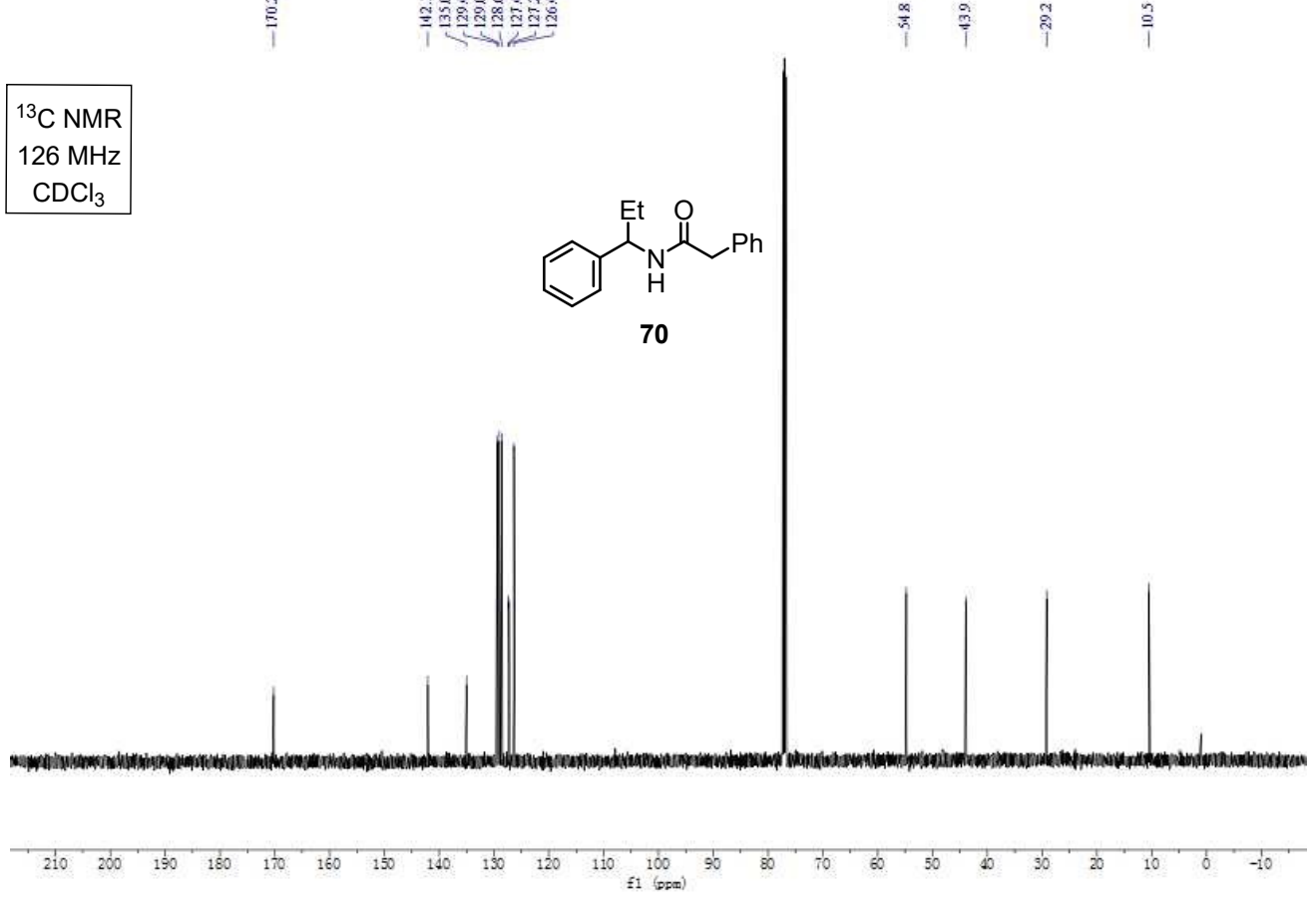
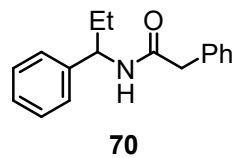
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151 MHz  
CDCl<sub>3</sub>

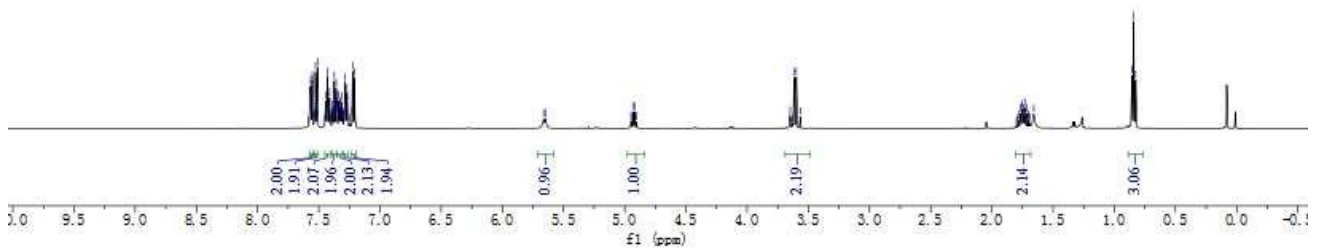
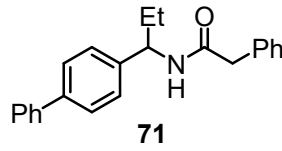
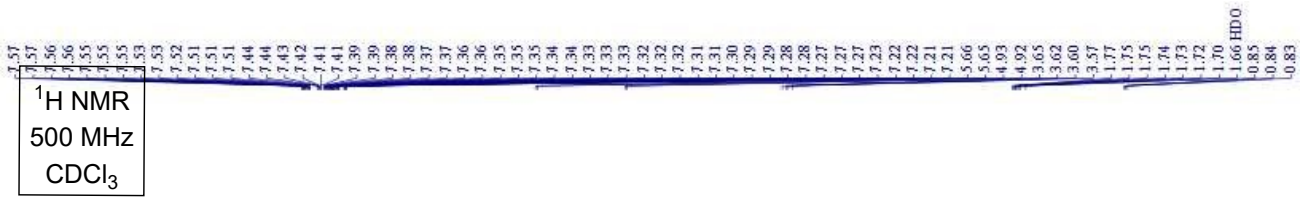


<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

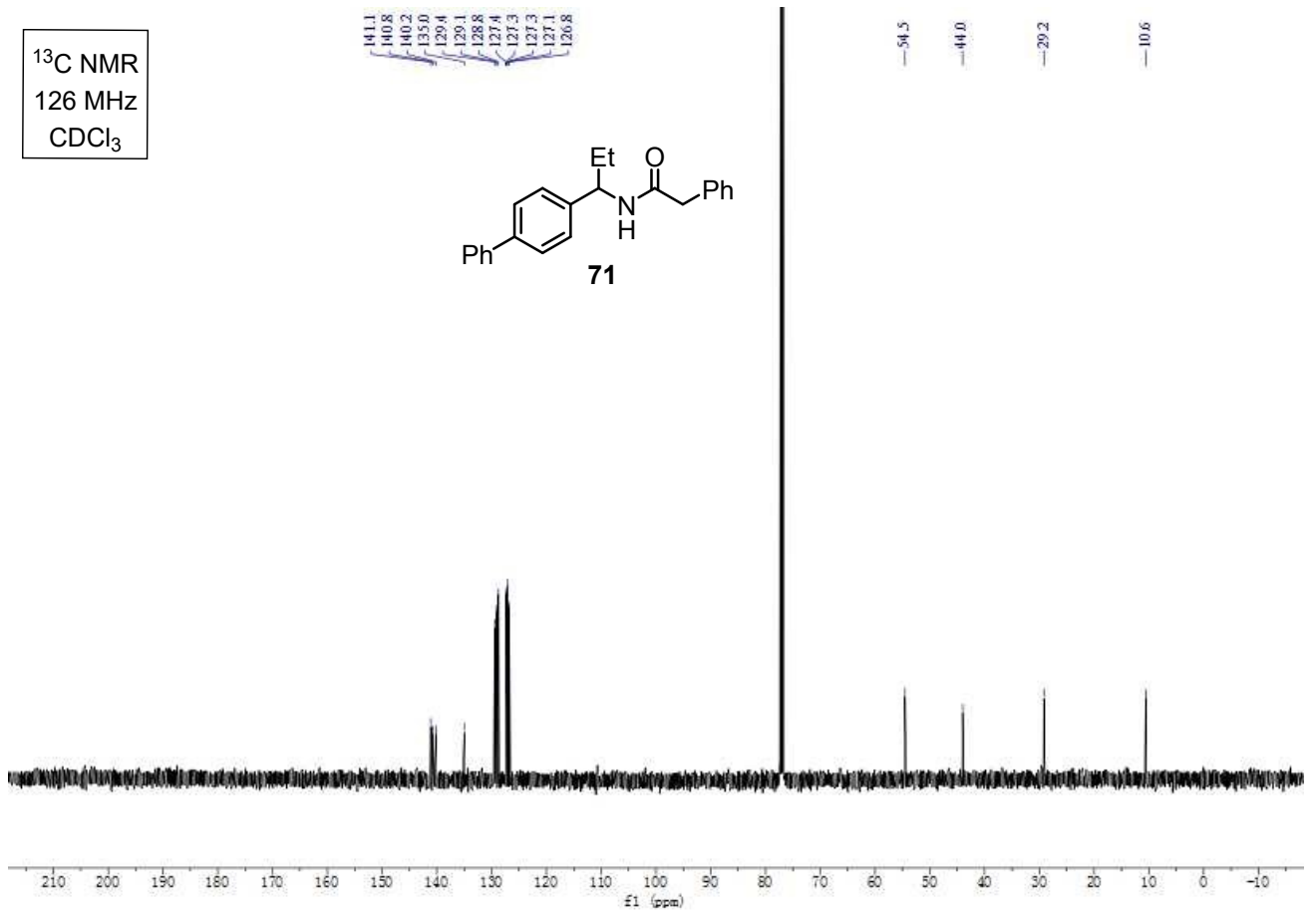
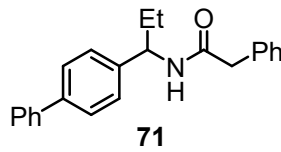


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



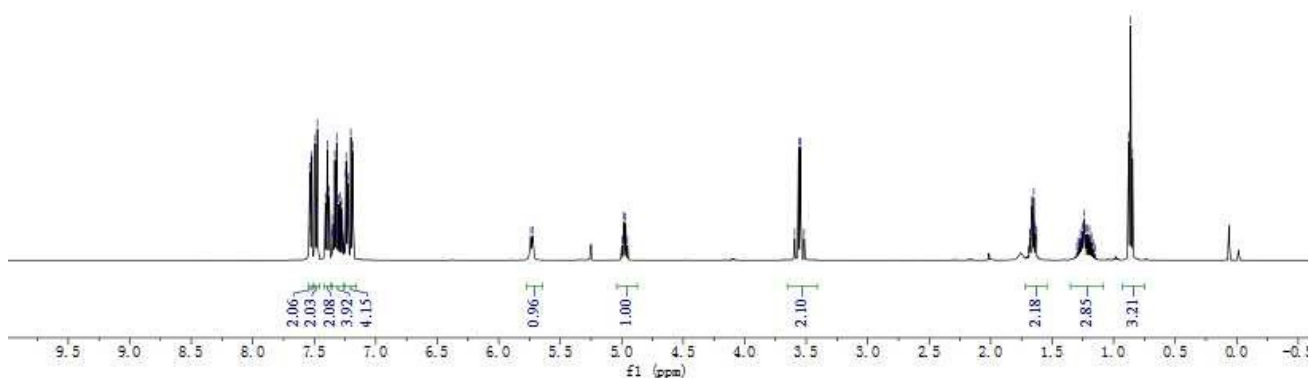
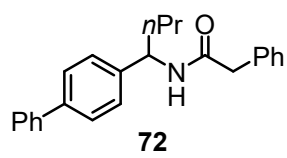


**<sup>13</sup>C NMR**  
126 MHz  
CDCl<sub>3</sub>

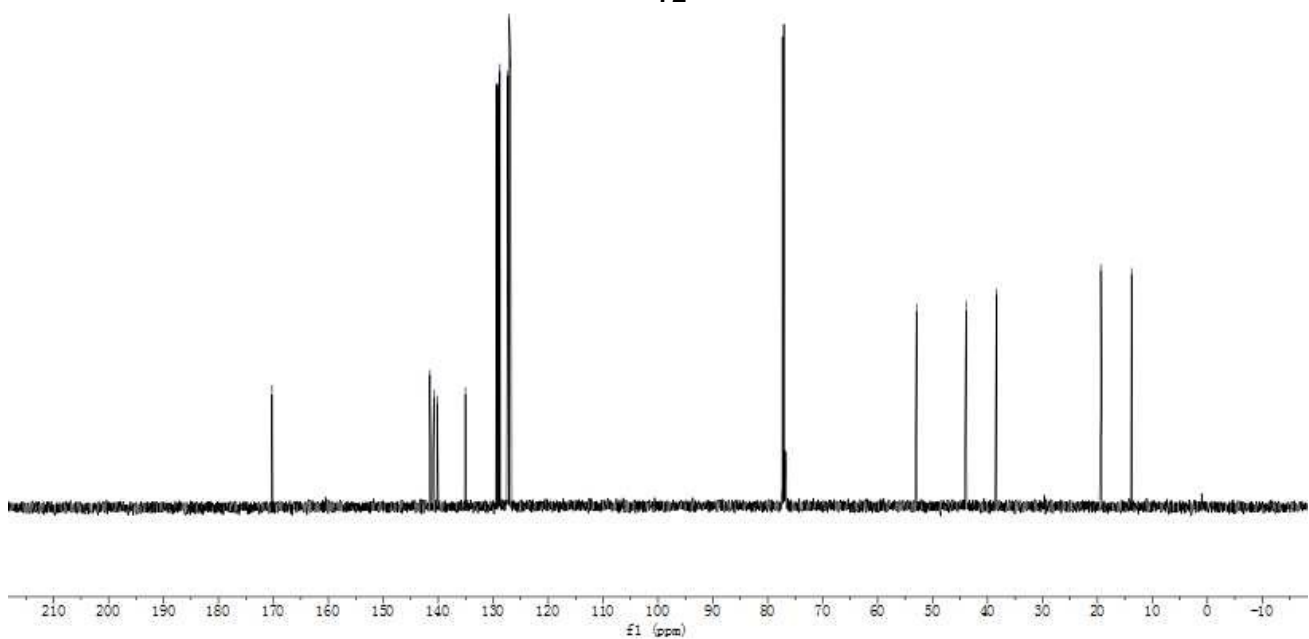
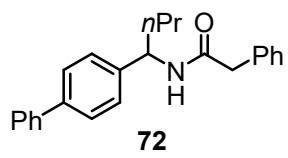


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<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

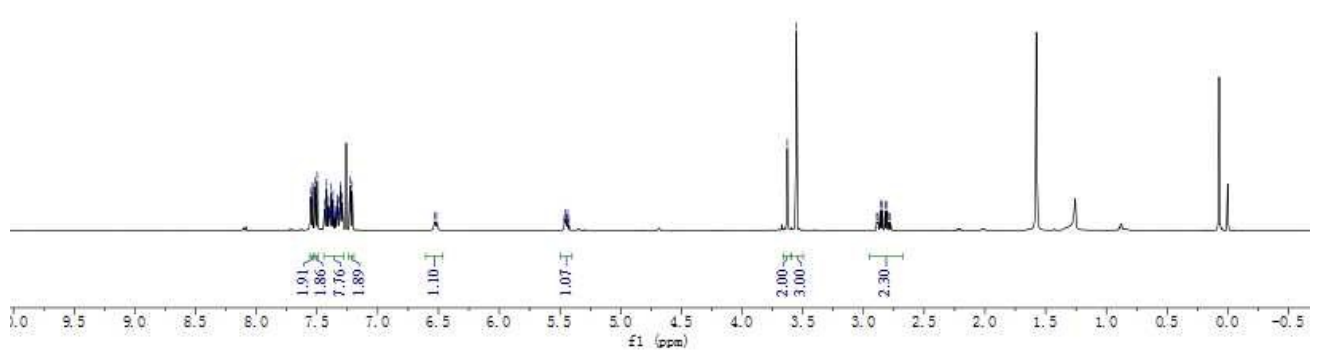
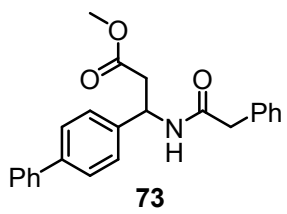


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



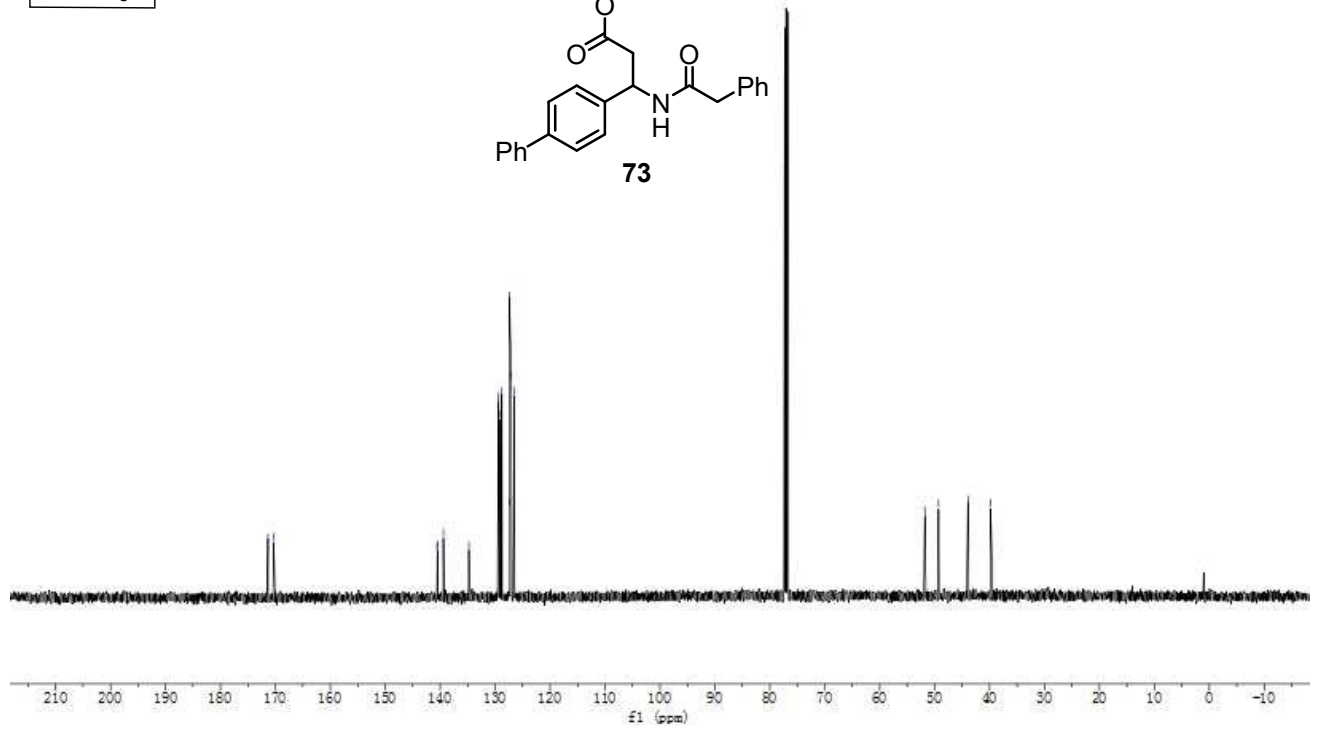
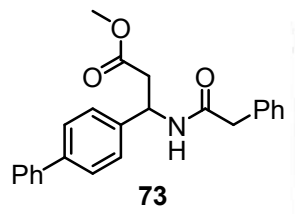
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6.51  
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5.44  
3.63  
3.63  
3.63  
2.88  
2.86  
2.85  
2.82  
2.81  
2.79  
2.78

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



171.3  
170.3  
140.6  
140.5  
139.4  
134.8  
129.4  
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127.4  
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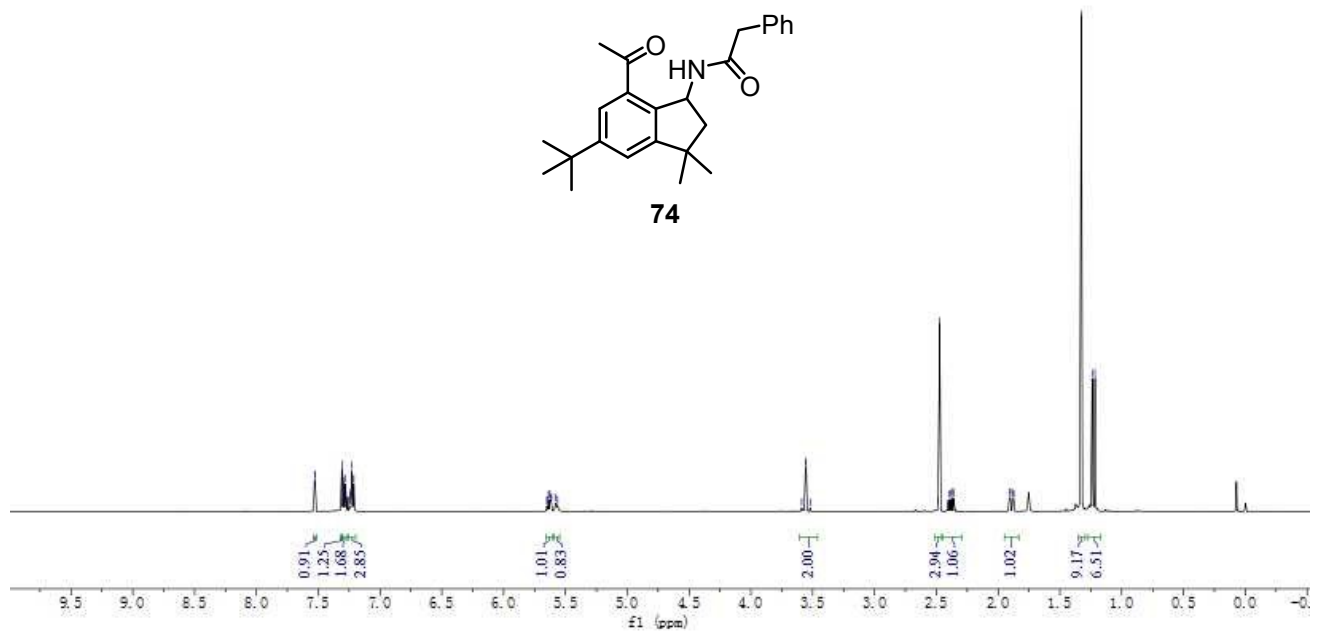
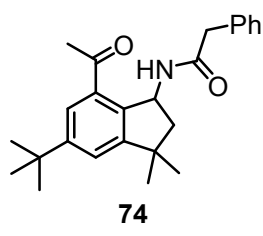
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



**<sup>1</sup>H NMR**  
500 MHz  
CDCl<sub>3</sub>

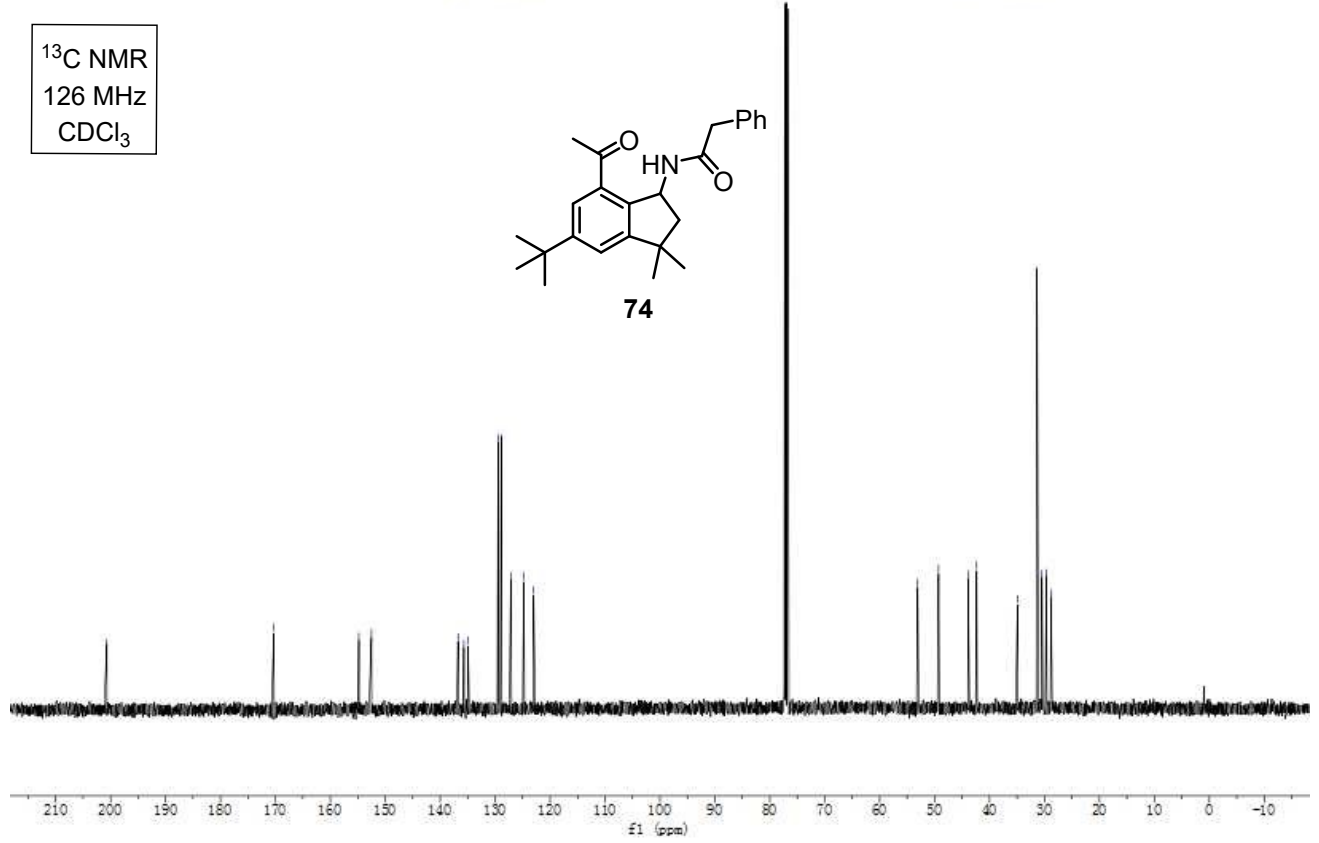
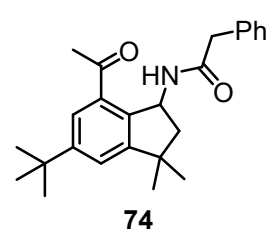
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5.64  
5.64  
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5.61  
5.58  
5.56

3.59  
3.56  
3.55  
3.52  
2.48  
2.47  
2.41  
2.40  
2.38  
2.37  
2.36  
1.91  
1.90  
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1.24  
1.22



**<sup>13</sup>C NMR**  
126 MHz  
CDCl<sub>3</sub>

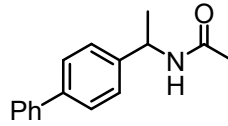
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124.8  
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31.4  
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29.7  
28.8



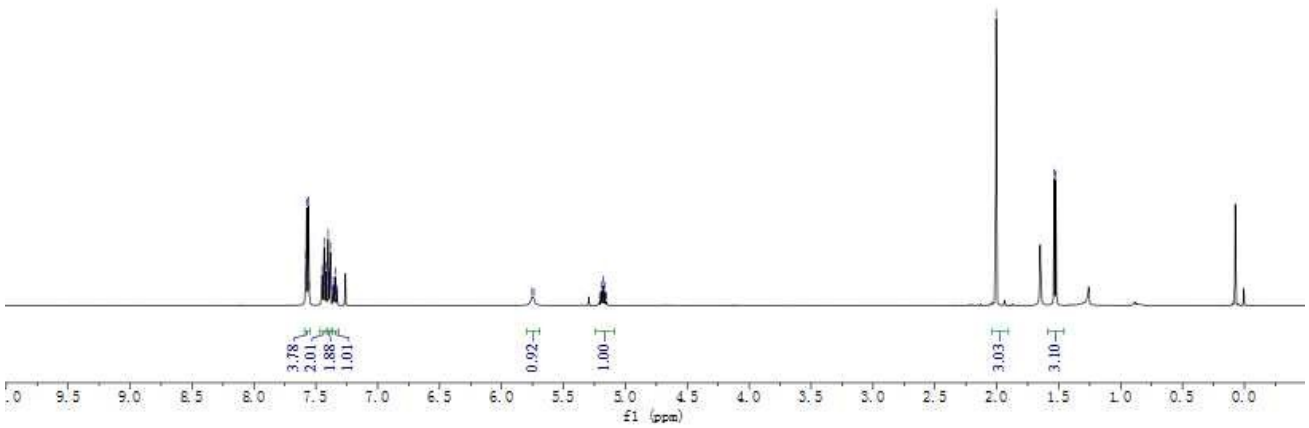
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7.33  
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5.15

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-1.54  
-1.52

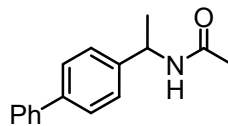
$^1\text{H}$  NMR  
500 MHz  
 $\text{CDCl}_3$



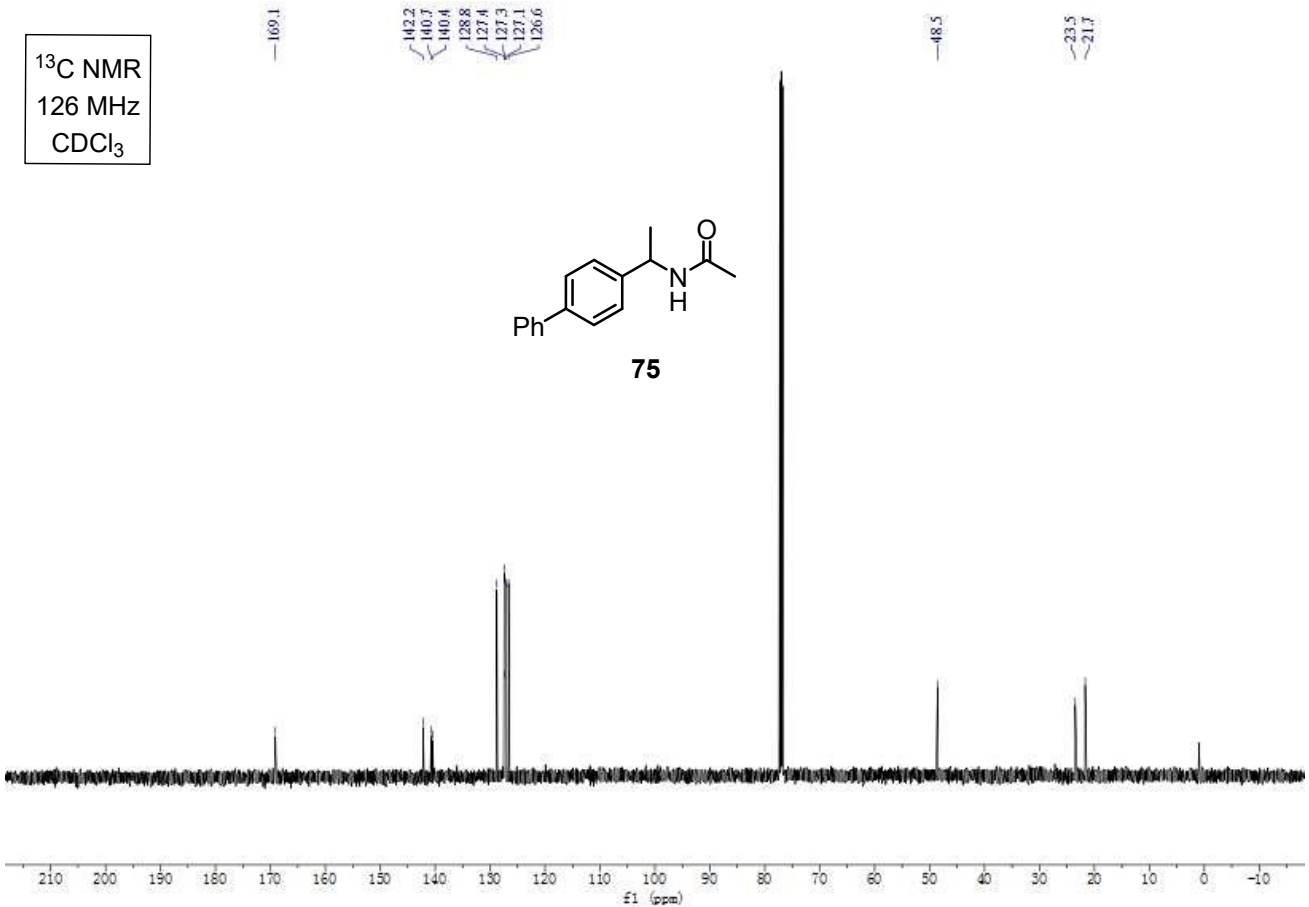
**75**



$^{13}\text{C}$  NMR  
126 MHz  
 $\text{CDCl}_3$



**75**

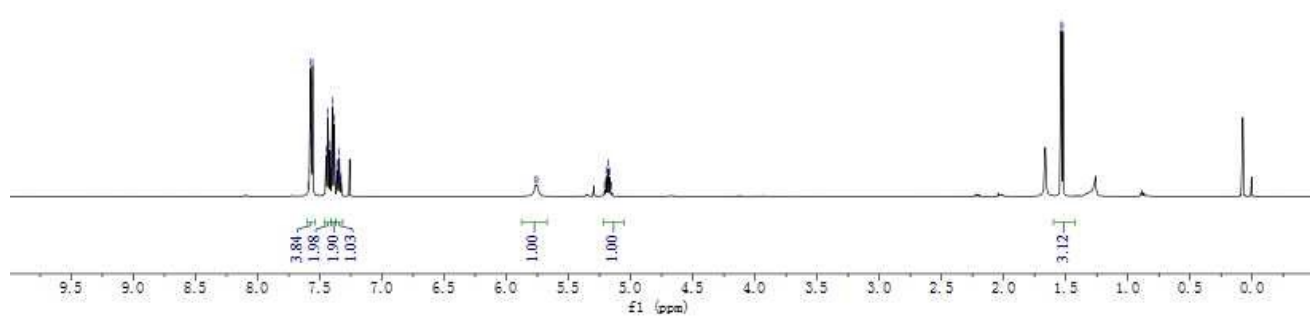
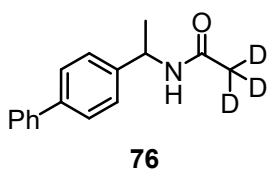




<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

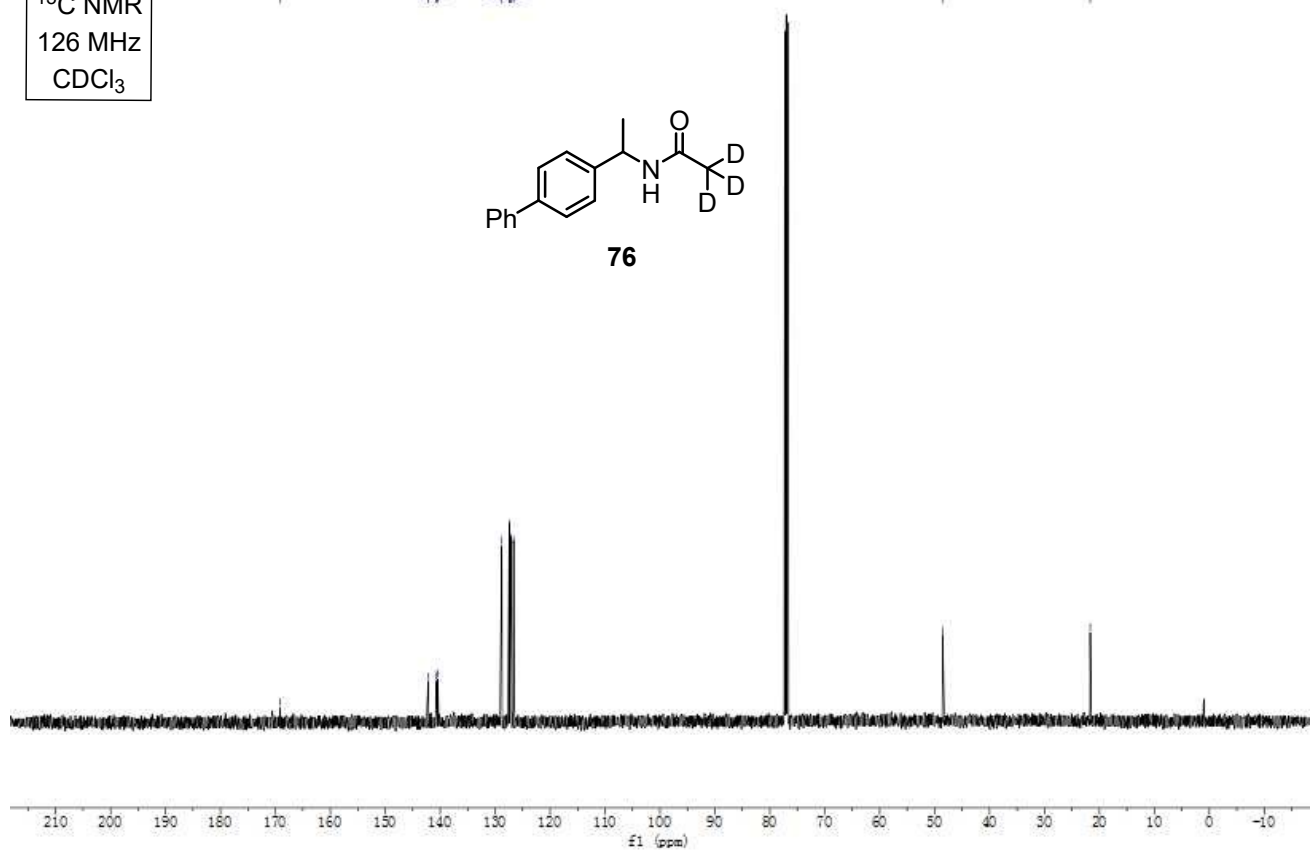
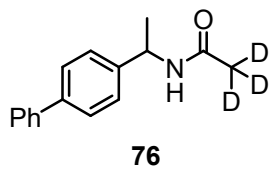
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5.18  
5.17  
5.15

1.54  
1.52

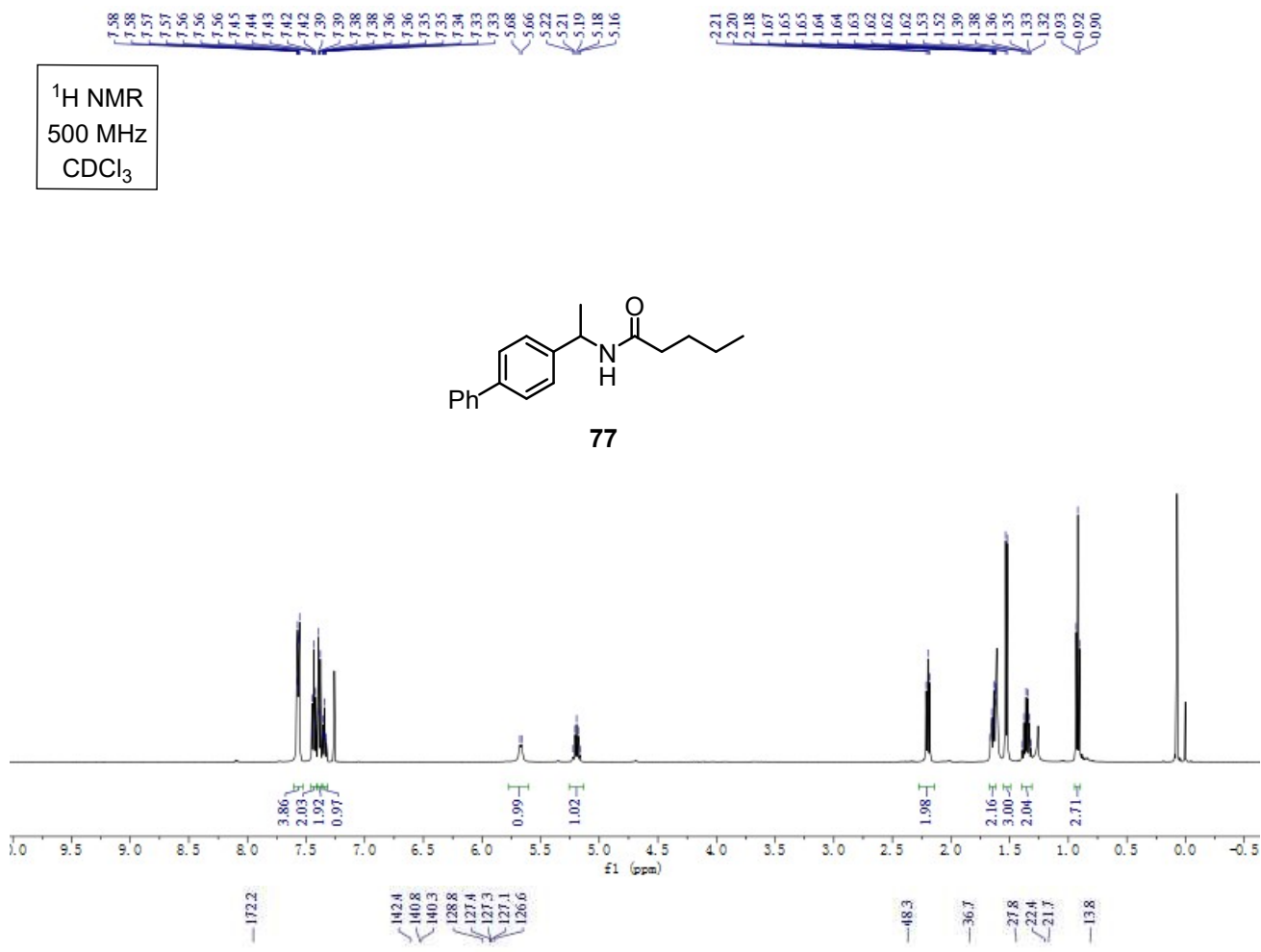
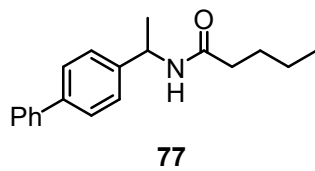


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

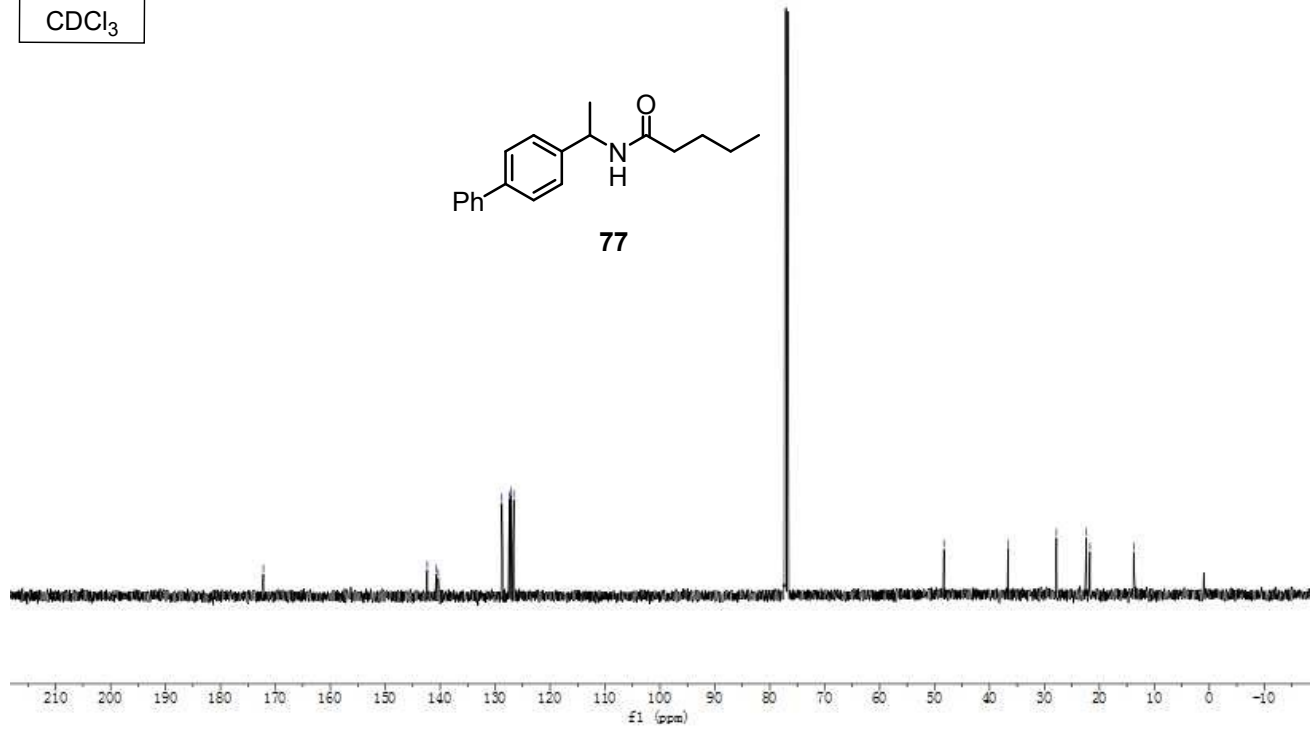
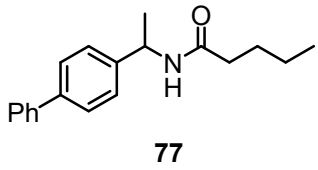
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140.4  
128.8  
127.4  
127.3  
127.1  
126.6  
48.5  
21.7



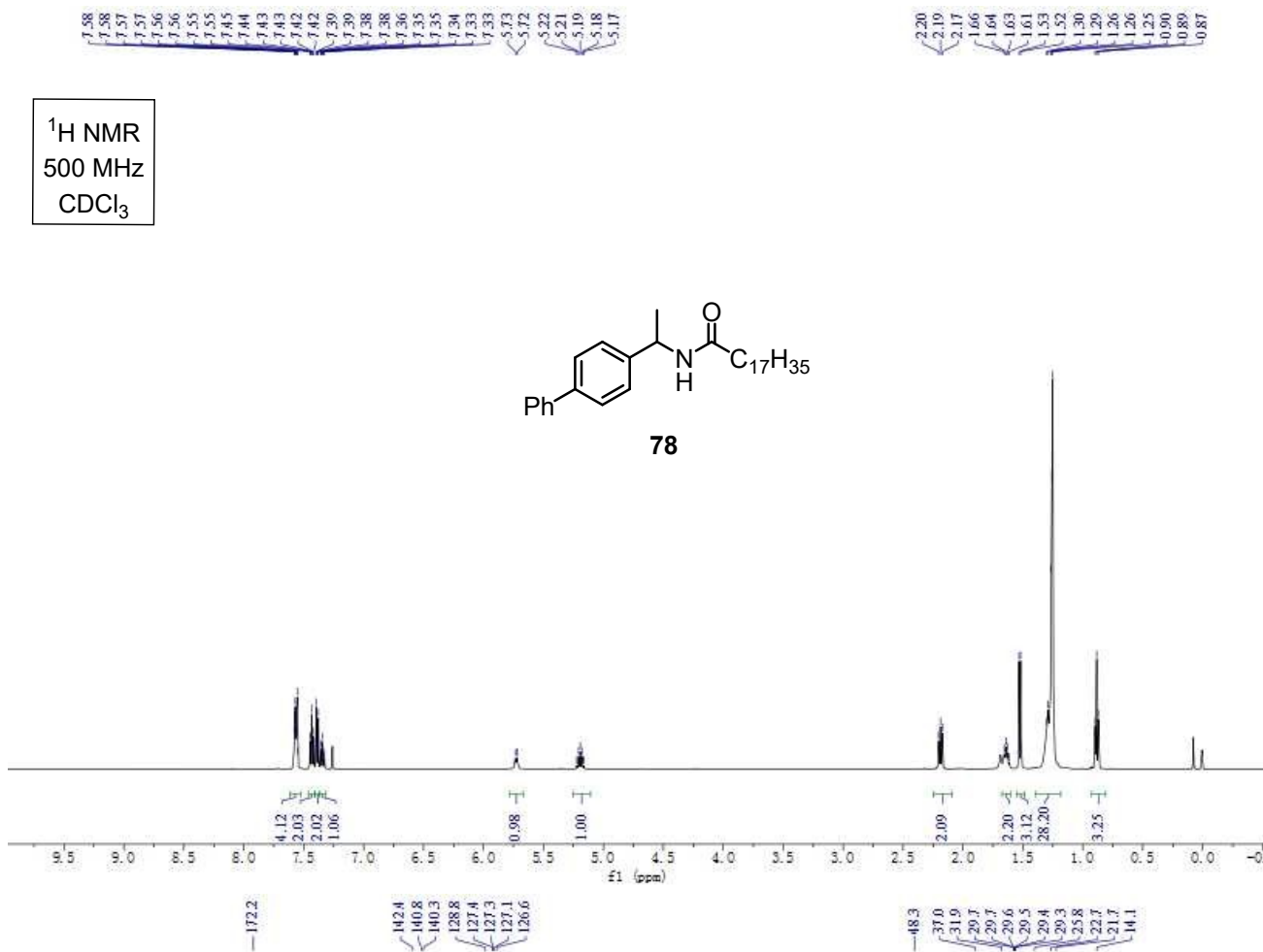
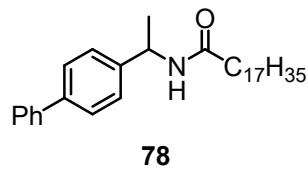
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500 MHz  
CDCl<sub>3</sub>



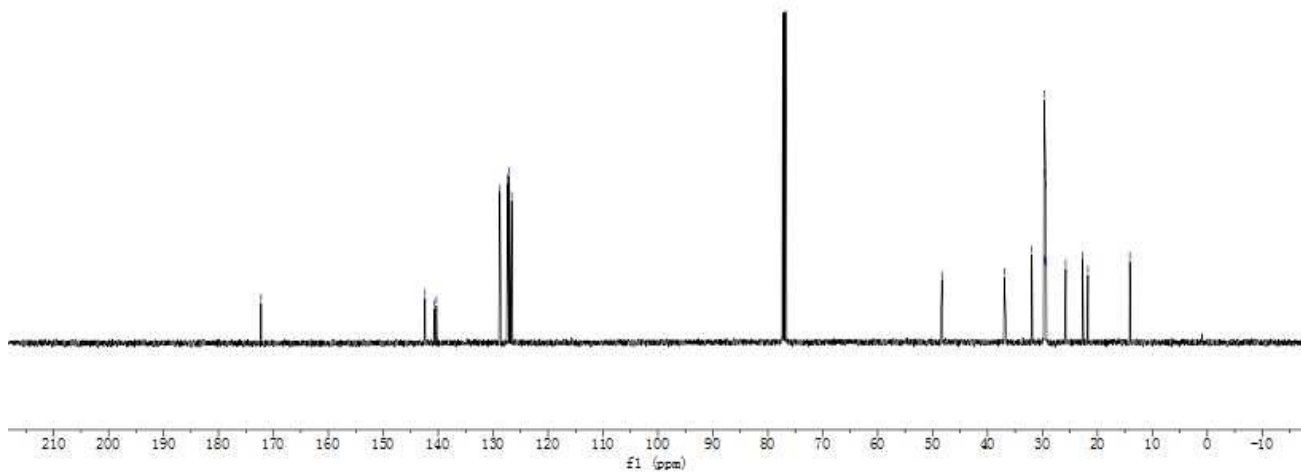
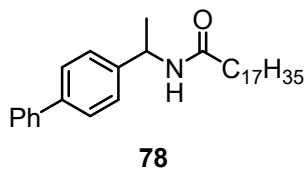
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126 MHz  
CDCl<sub>3</sub>



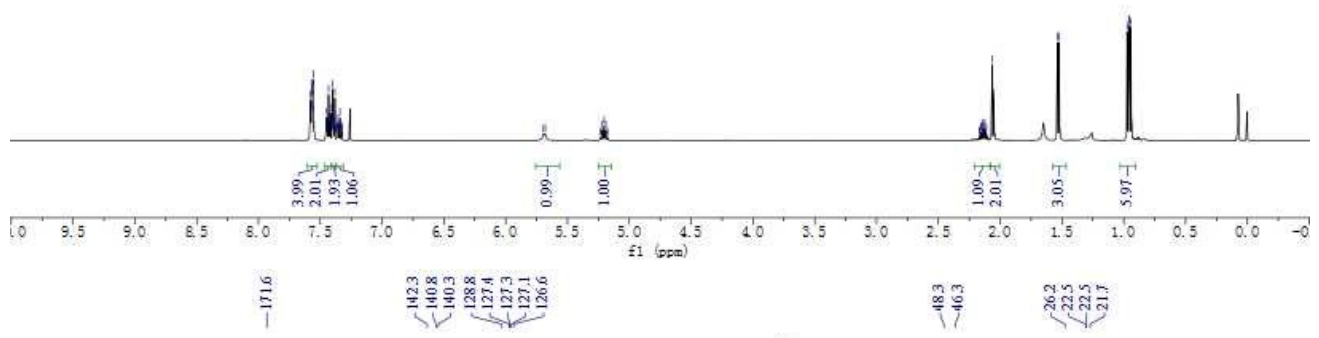
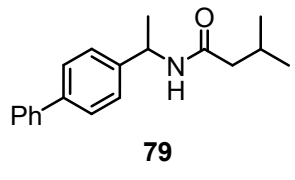
<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



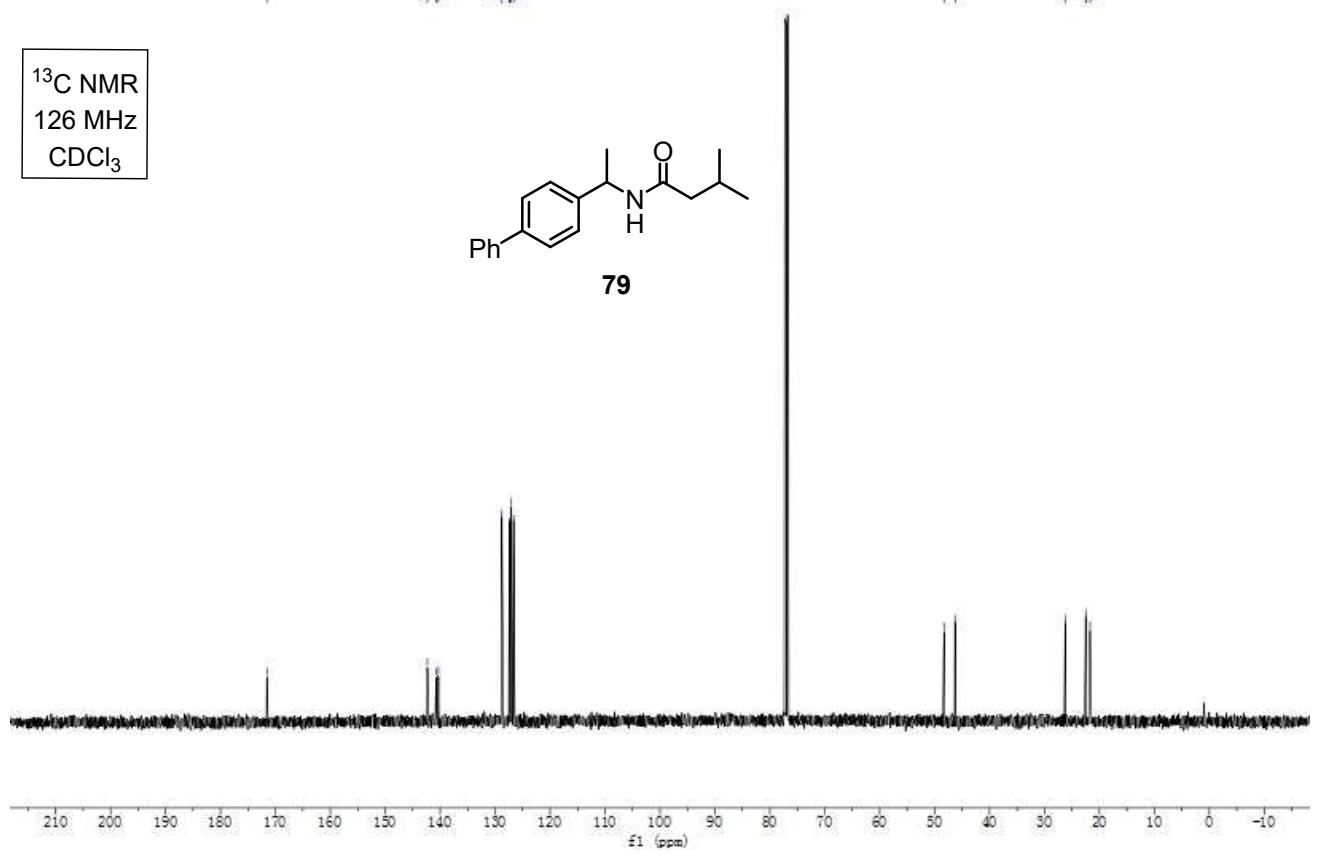
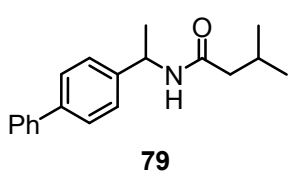
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



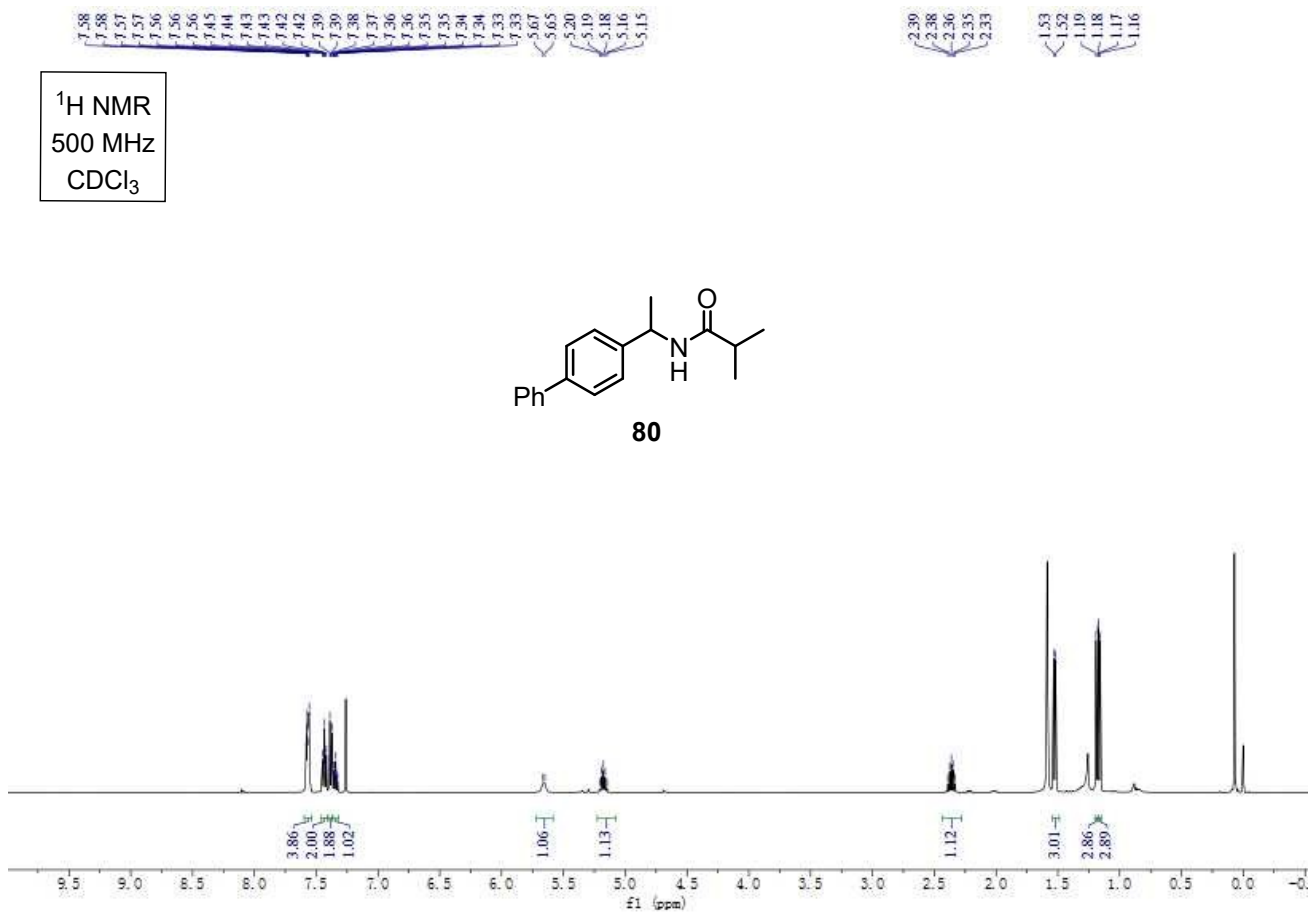
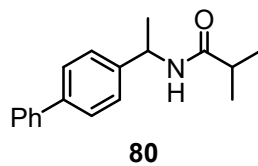
<sup>1</sup>H NMR  
 500 MHz  
 CDCl<sub>3</sub>



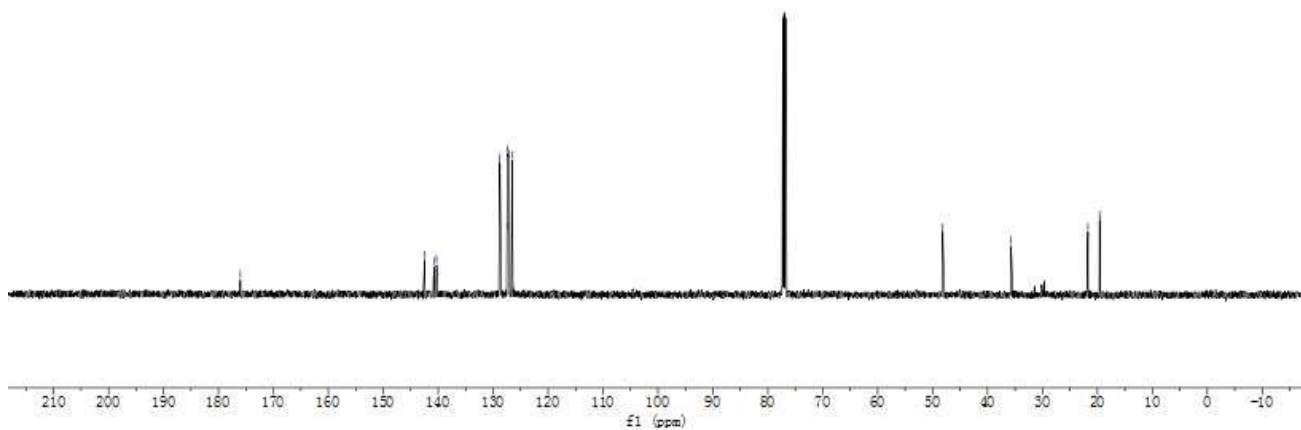
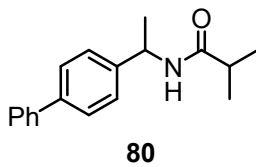
<sup>13</sup>C NMR  
 126 MHz  
 CDCl<sub>3</sub>

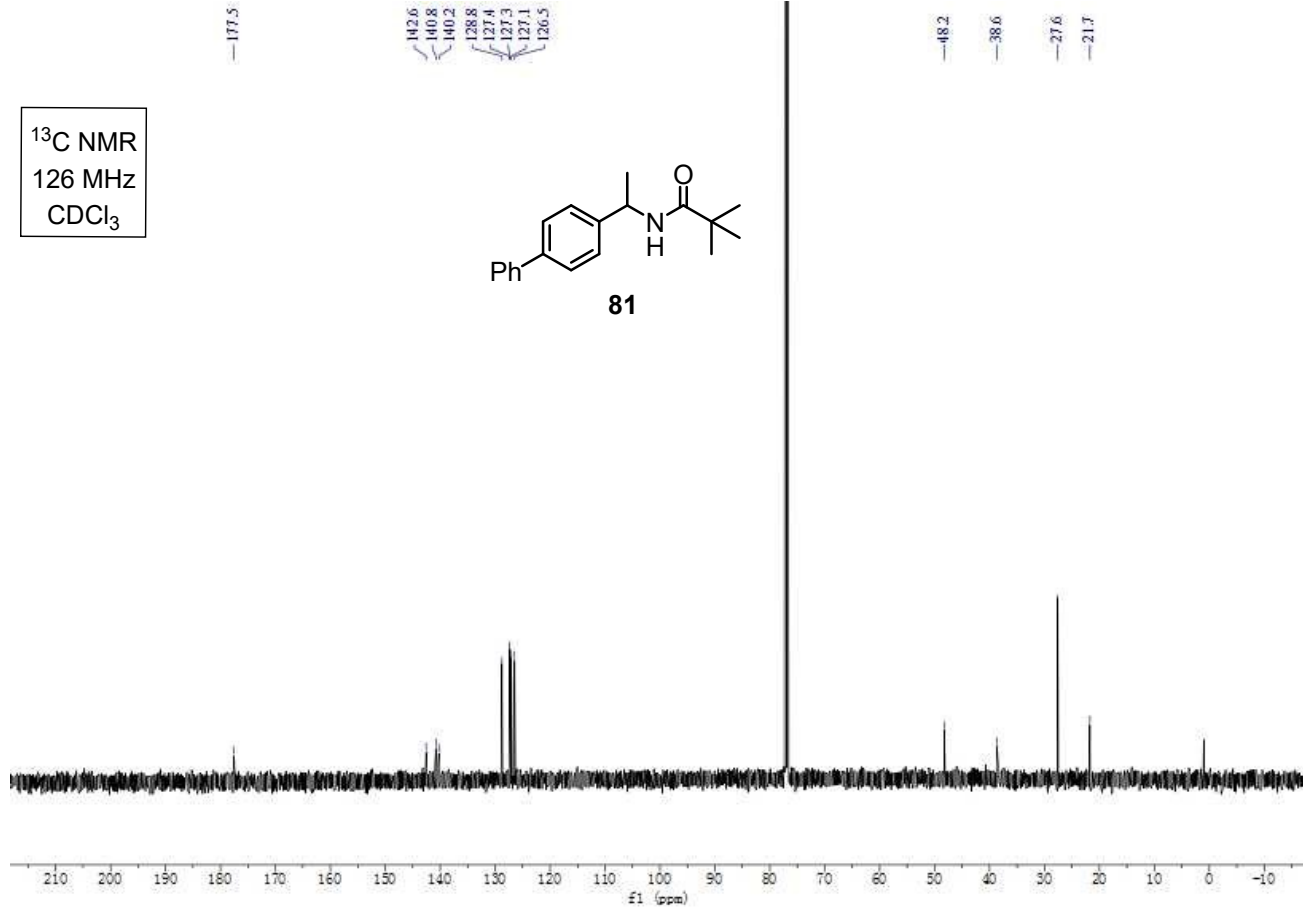
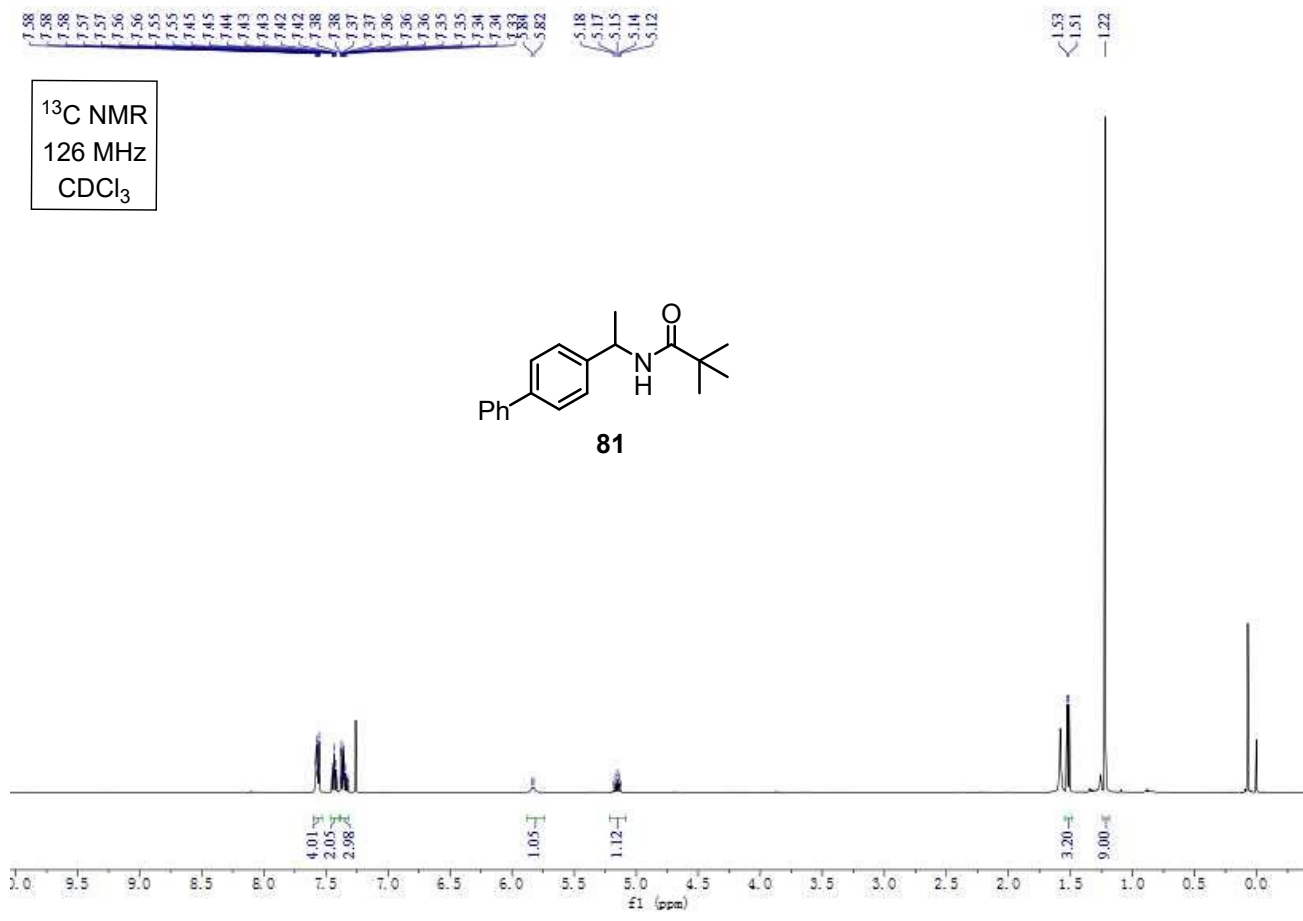


<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



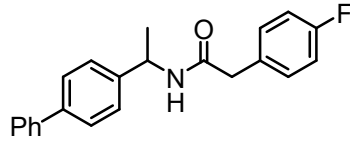
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



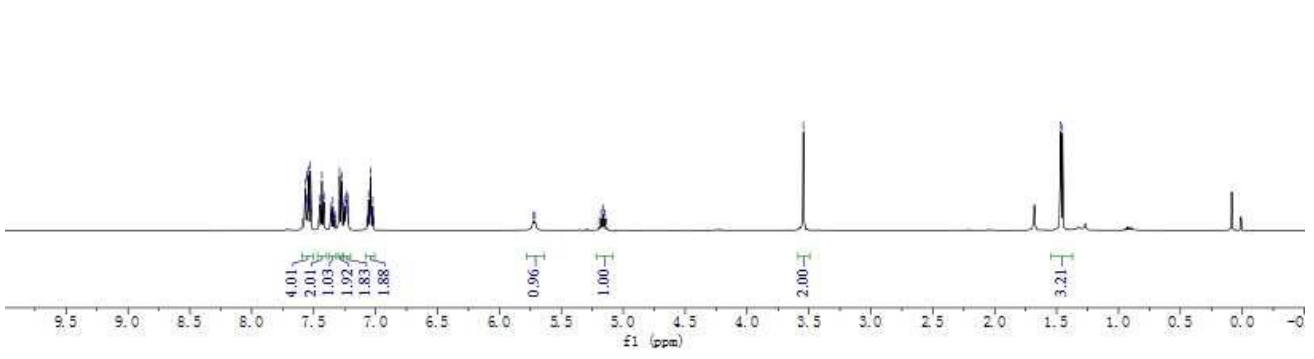


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3.95  
1.95

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

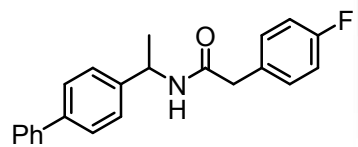


82

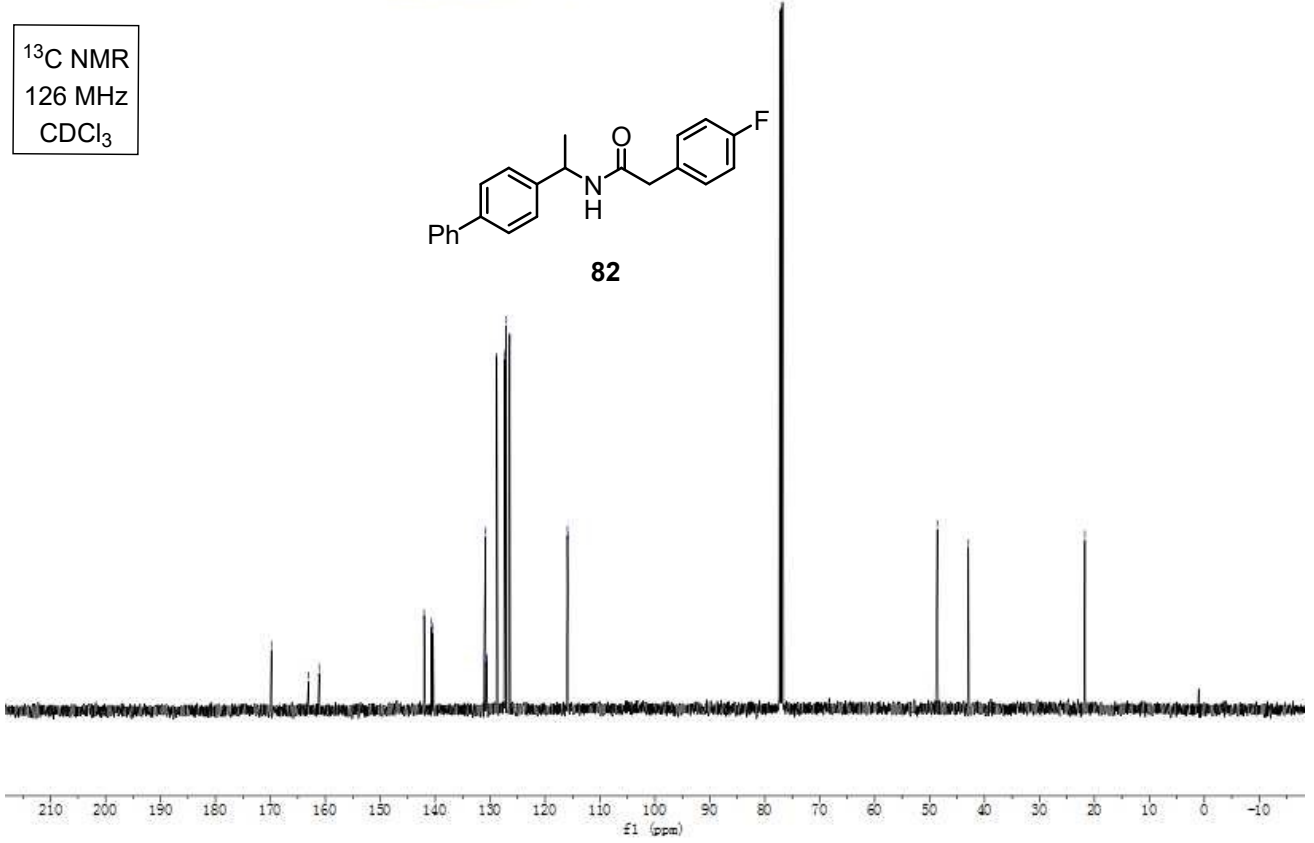


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140.4  
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130.7  
128.8  
127.4  
127.3  
127.1  
126.4  
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21.7

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



82

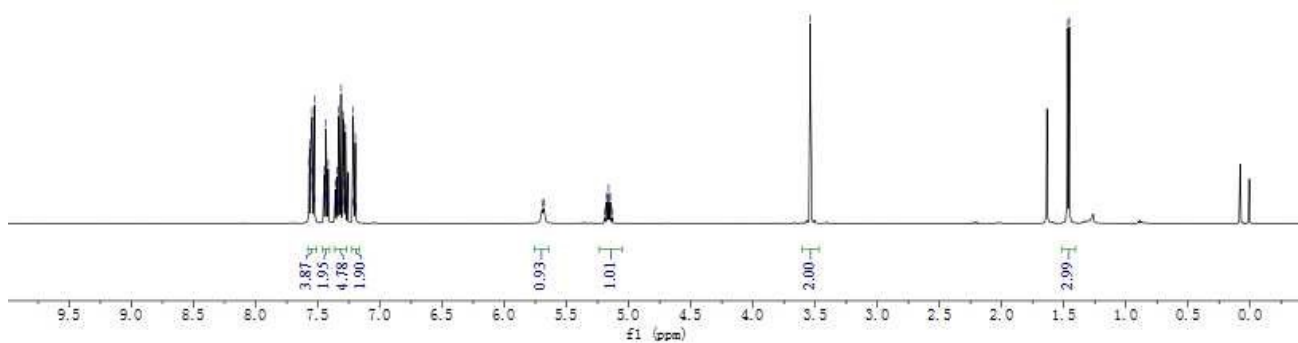
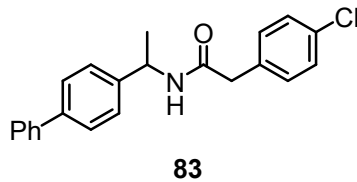


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5.18  
5.16  
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5.15

3.54

1.47  
1.46

$^1\text{H}$  NMR  
500 MHz  
 $\text{CDCl}_3$



169.4

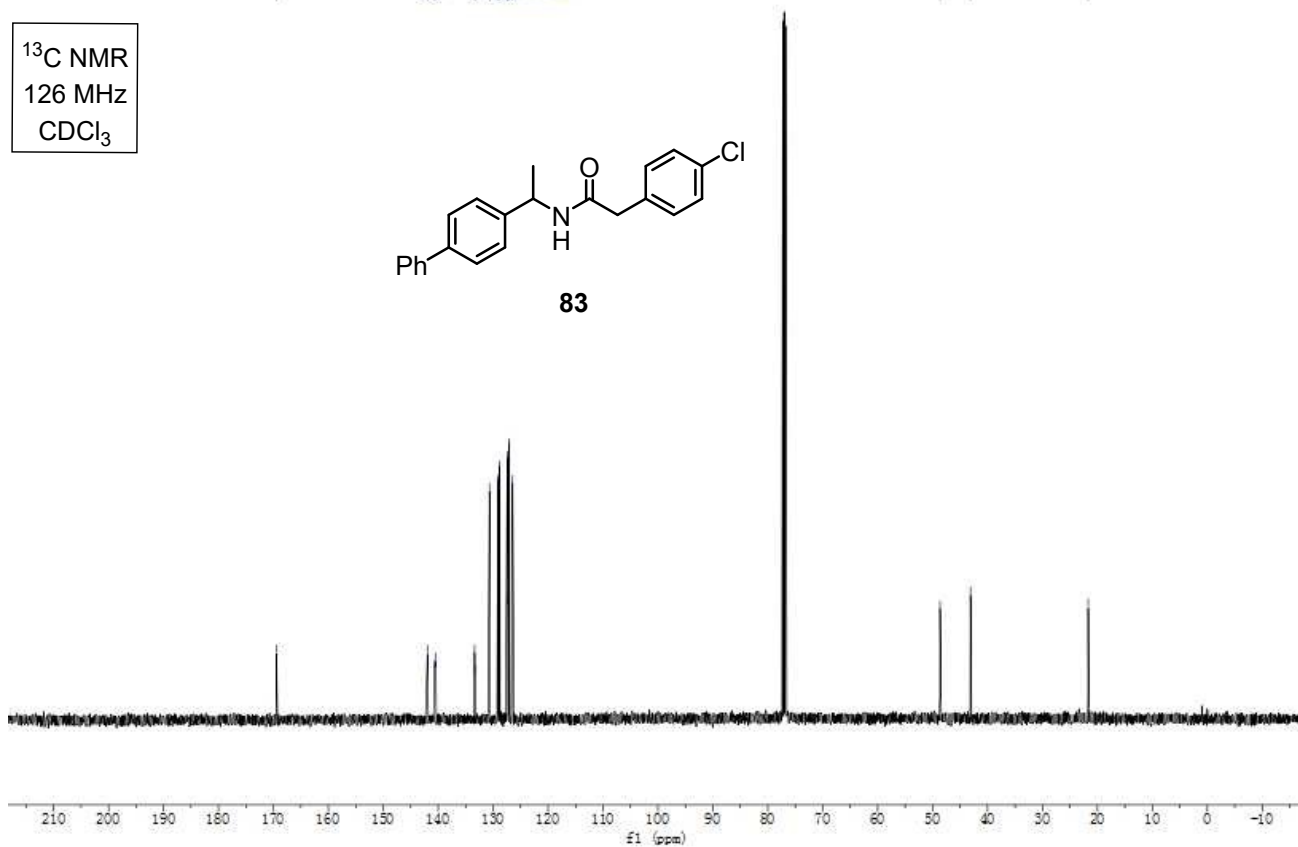
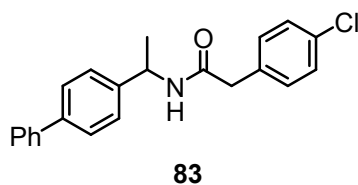
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48.7

43.1

21.7

$^{13}\text{C}$  NMR  
126 MHz  
 $\text{CDCl}_3$



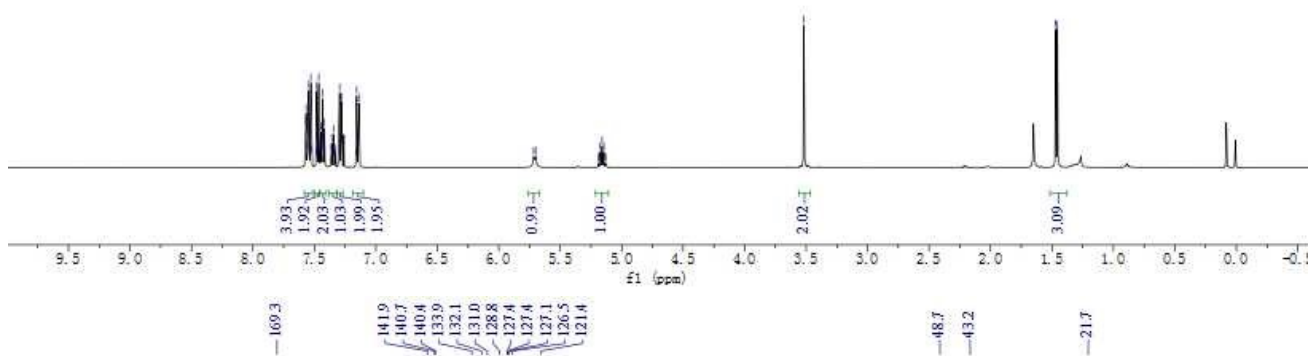
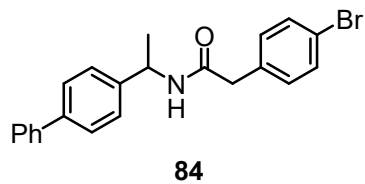


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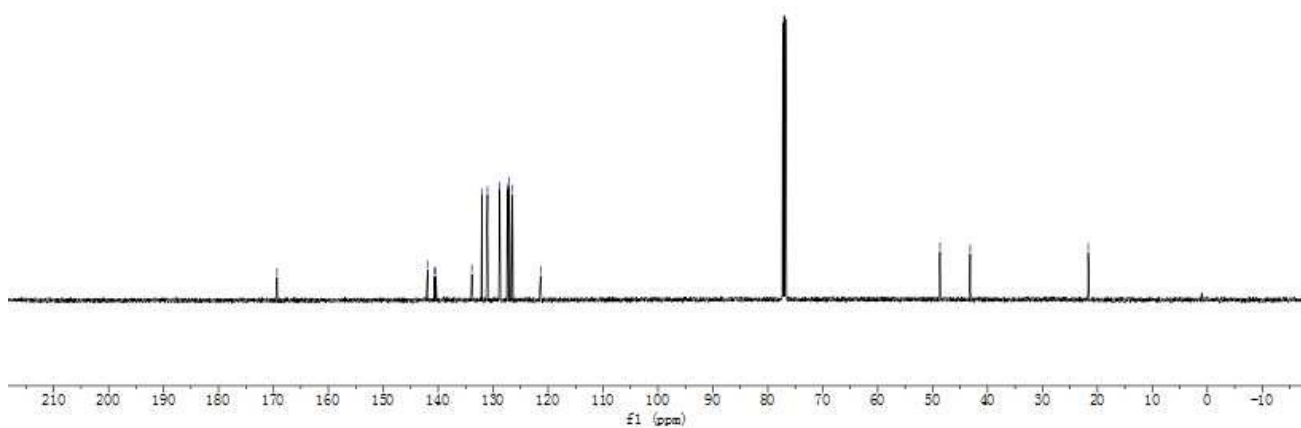
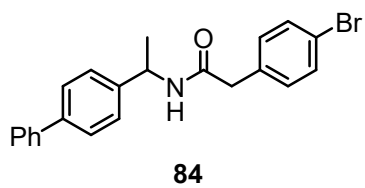
-3.52

1.87  
1.86

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



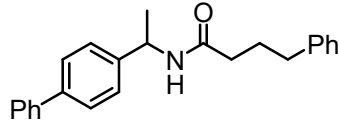
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126 MHz  
CDCl<sub>3</sub>



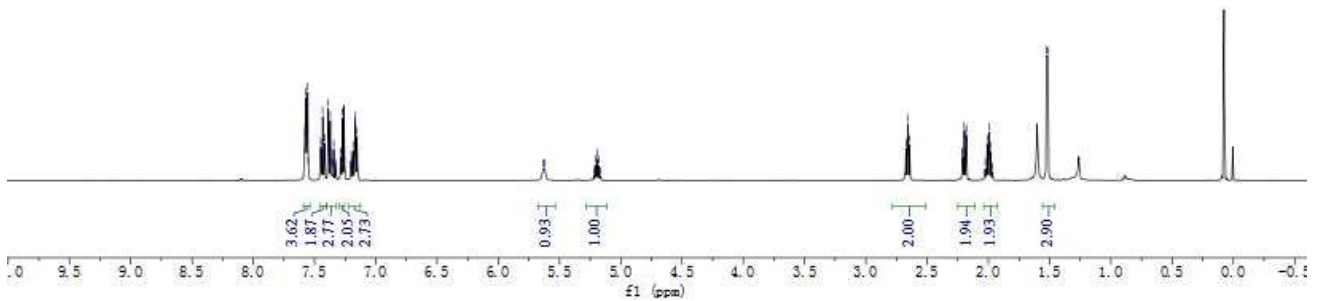
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<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

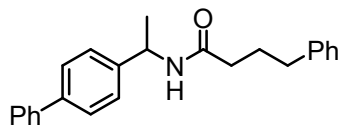


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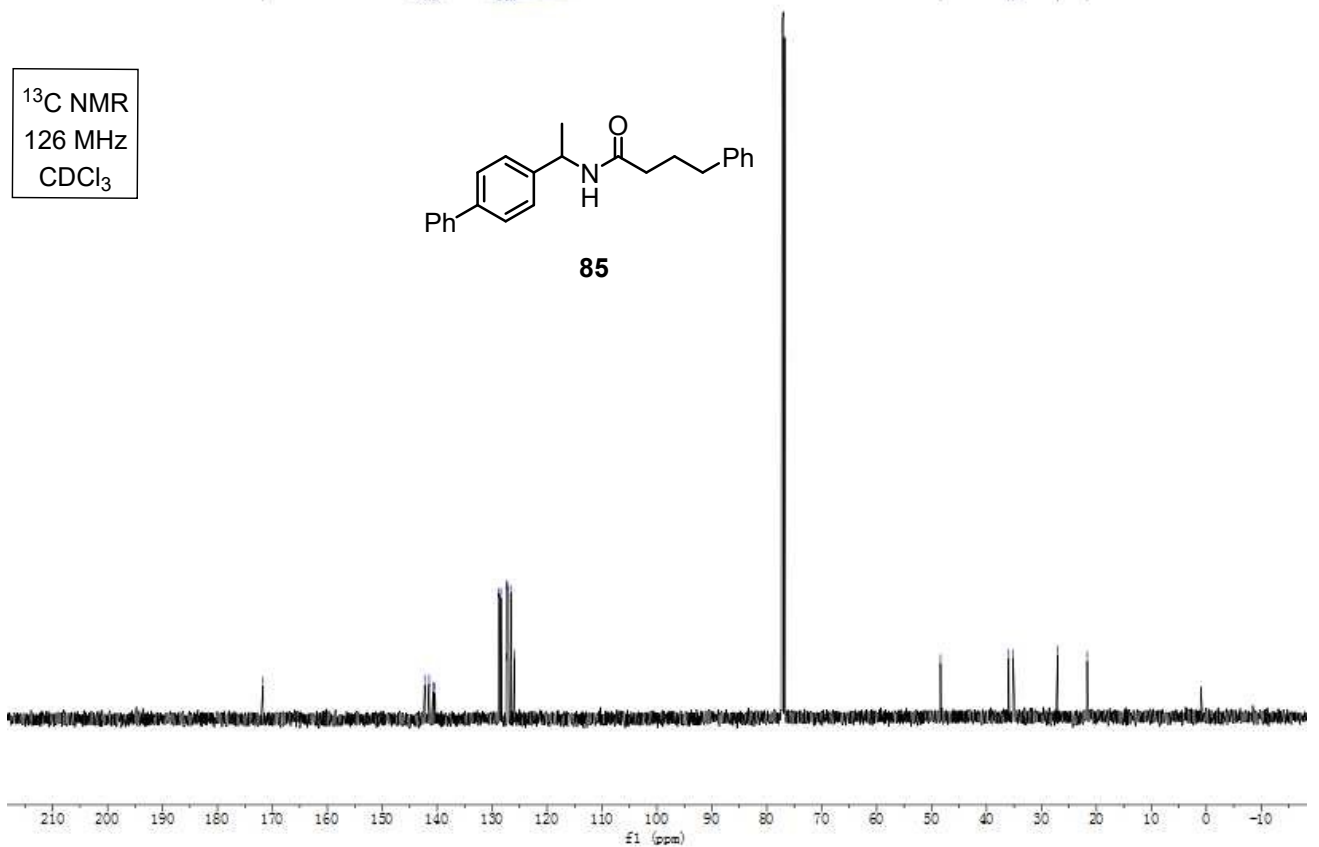


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127.1  
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35.2  
27.1  
21.7

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

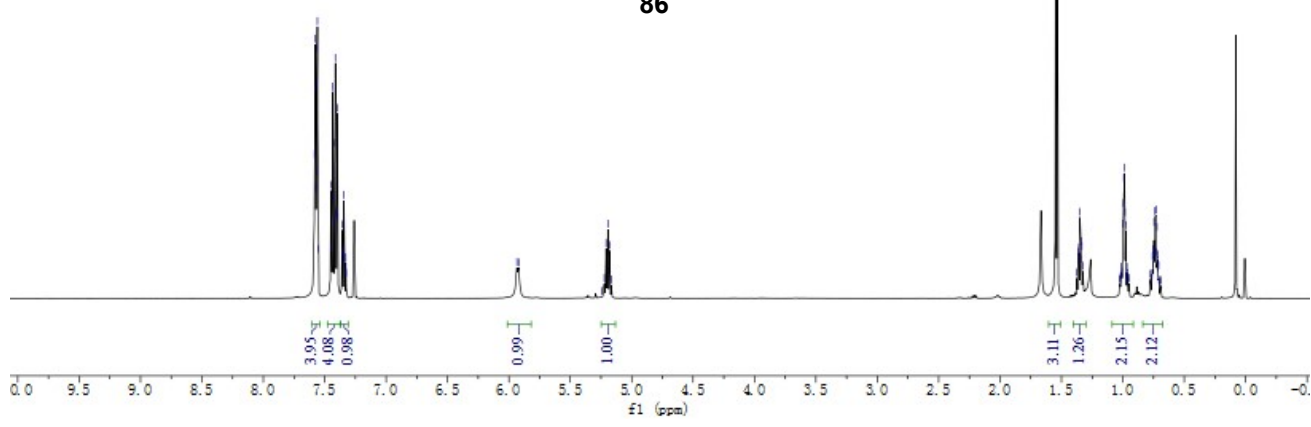
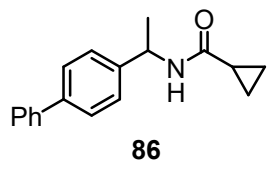


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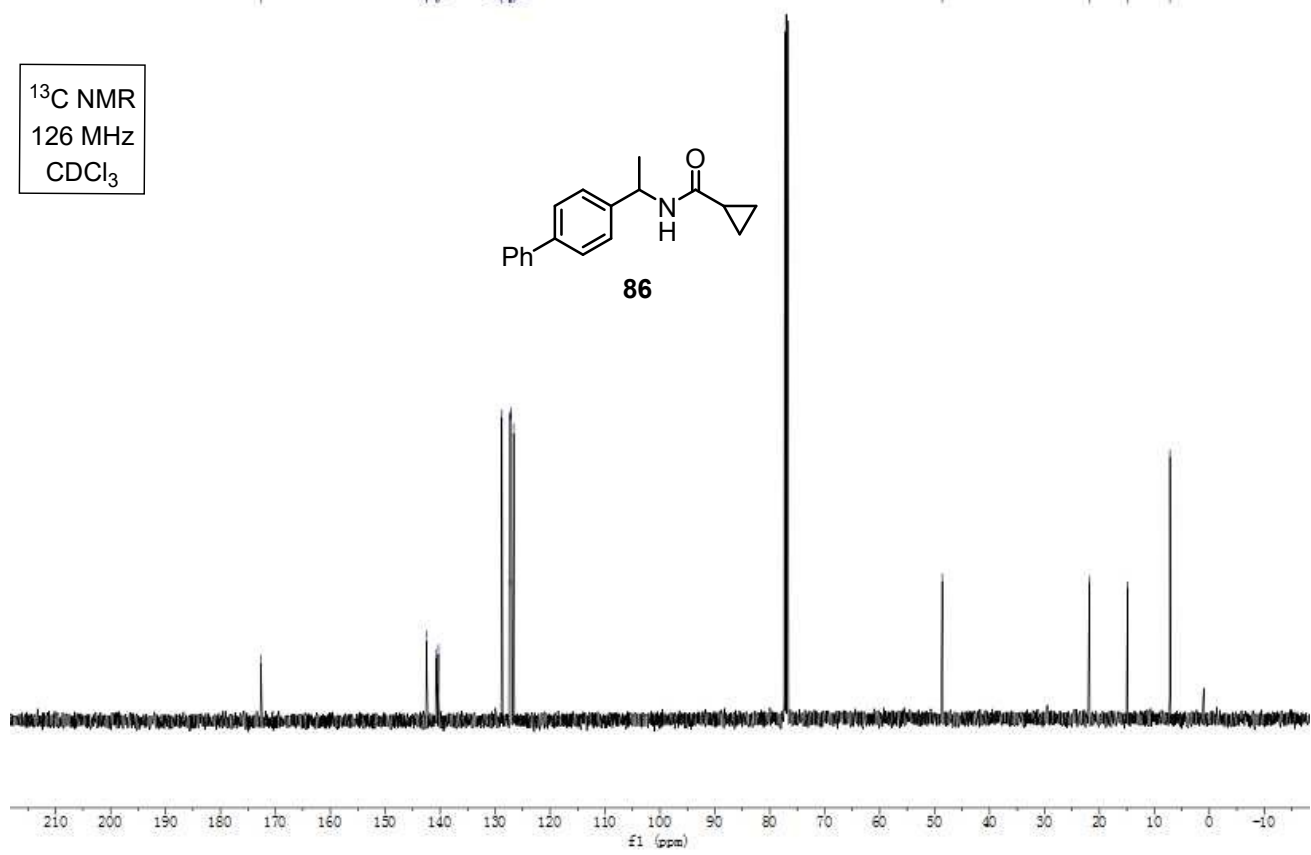
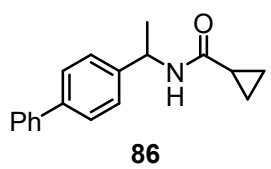
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7.43  
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7.42  
7.41  
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7.35  
7.34  
7.34  
7.33  
7.33  
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5.94  
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5.19  
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<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>



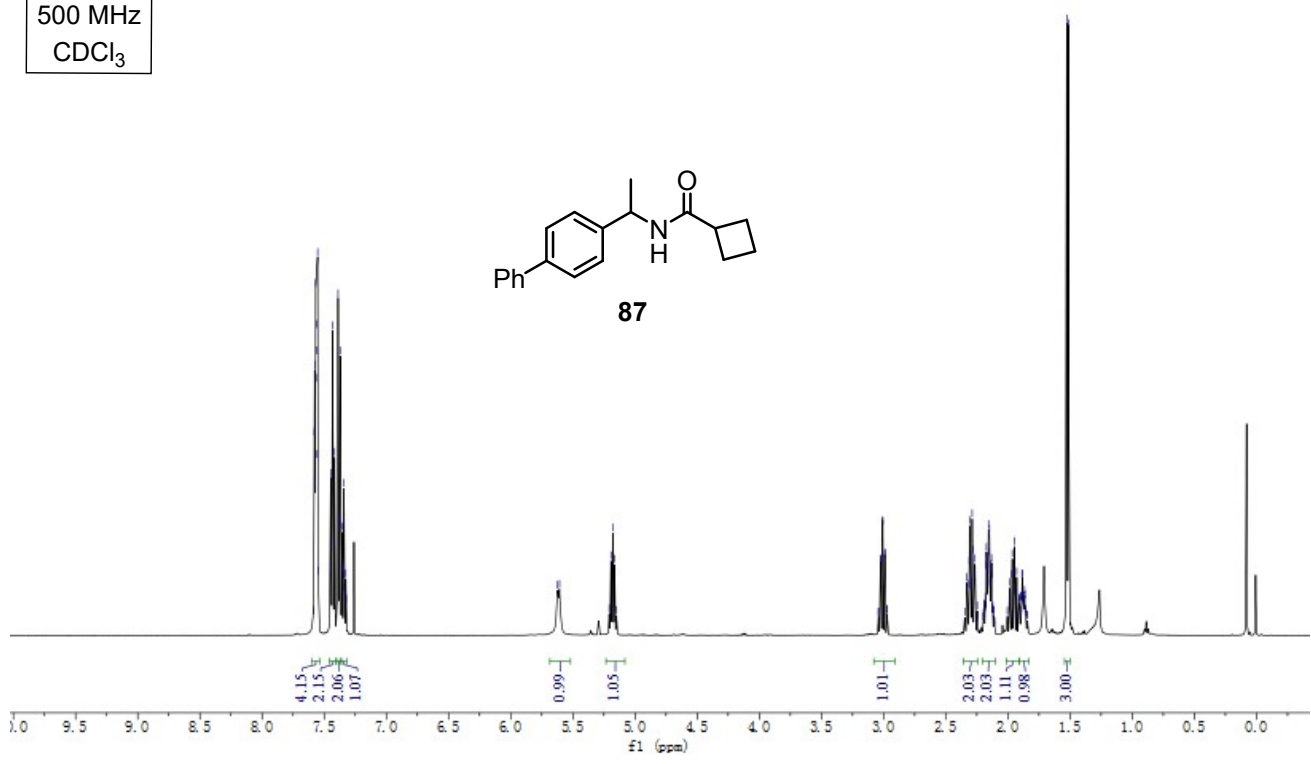
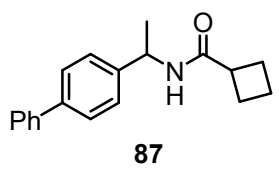
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128.8  
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127.3  
127.1  
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21.8  
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7.2

<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

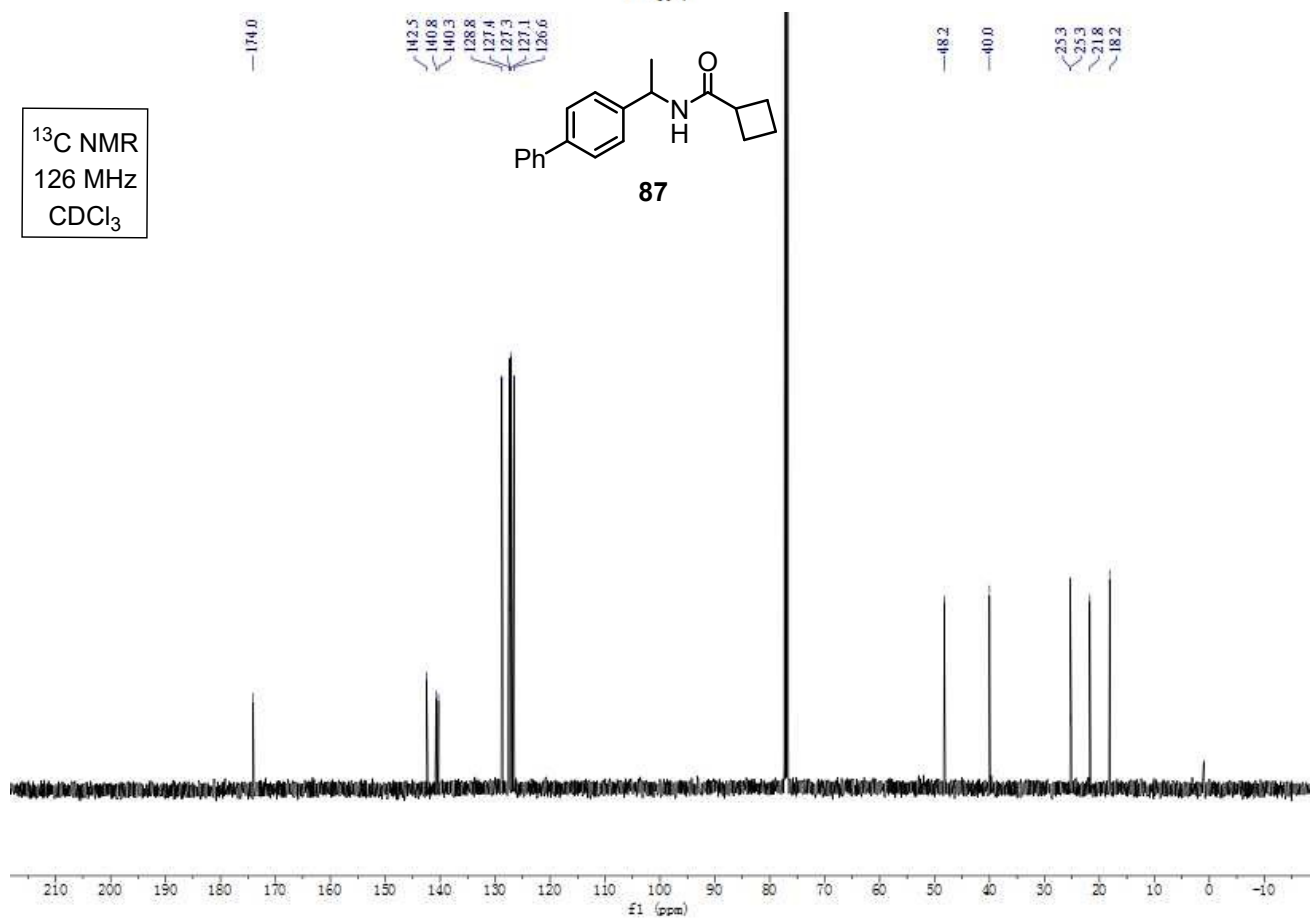
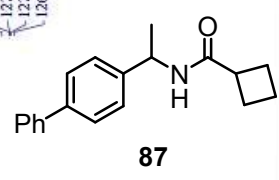


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<sup>1</sup>H NMR  
500 MHz  
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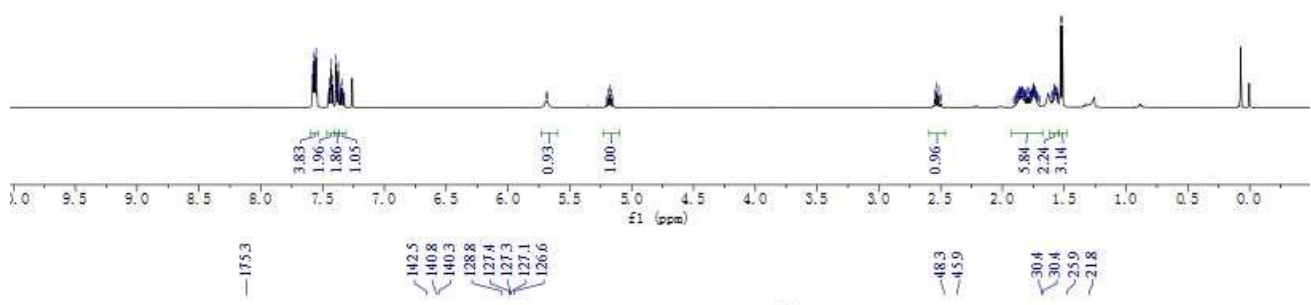
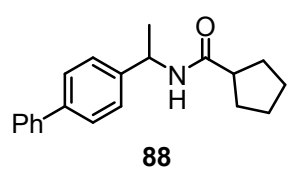


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

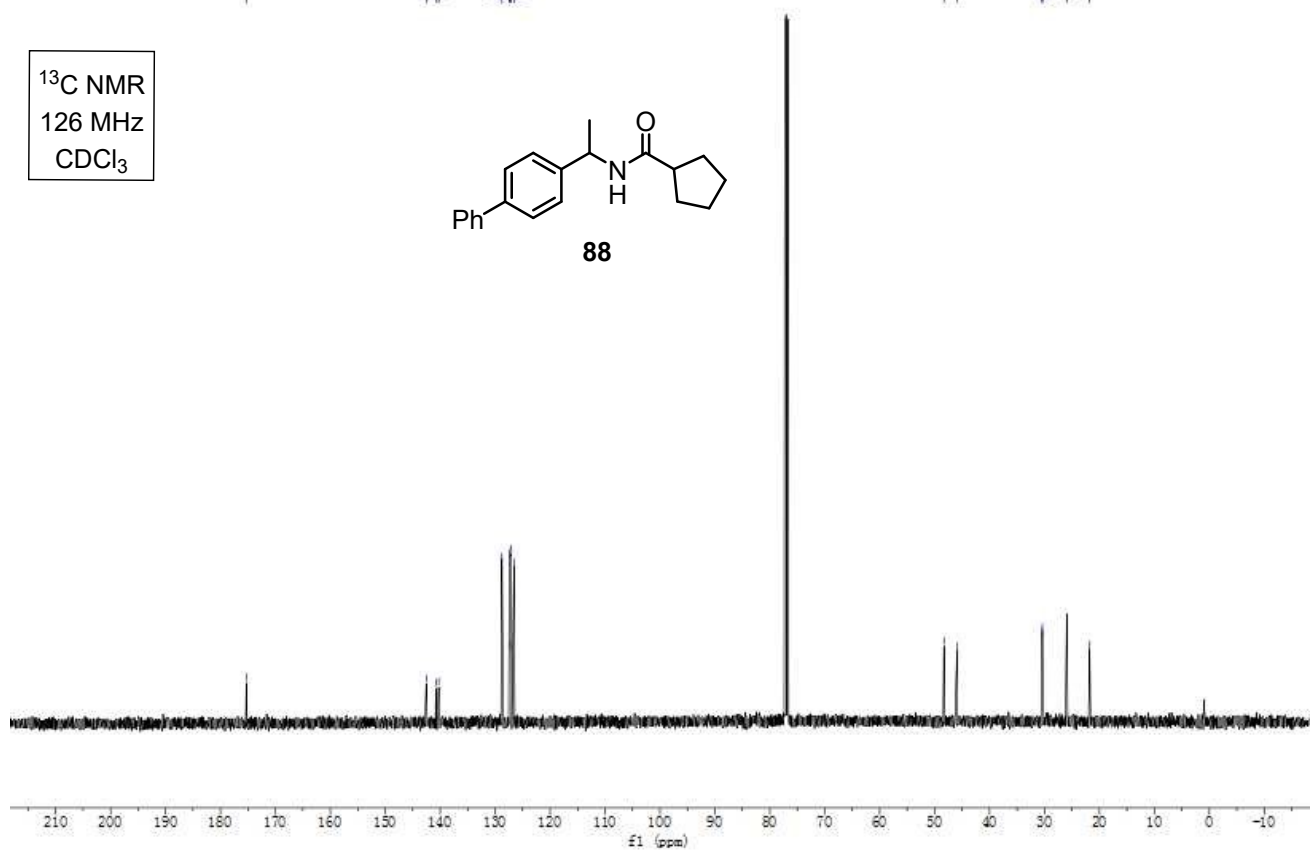
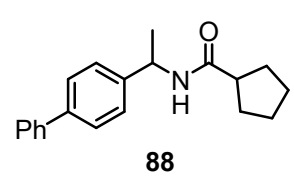


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<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

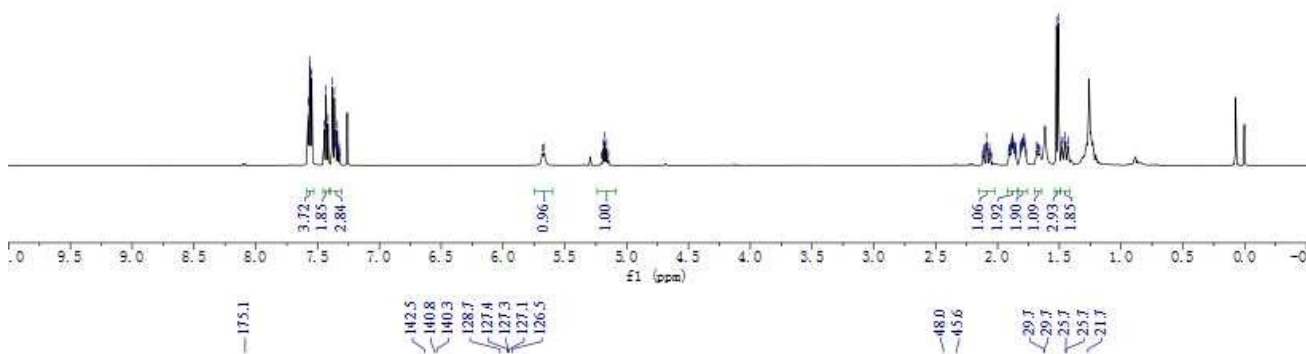
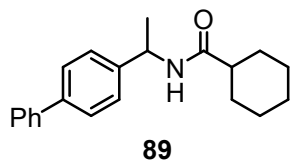


<sup>13</sup>C NMR  
126 MHz  
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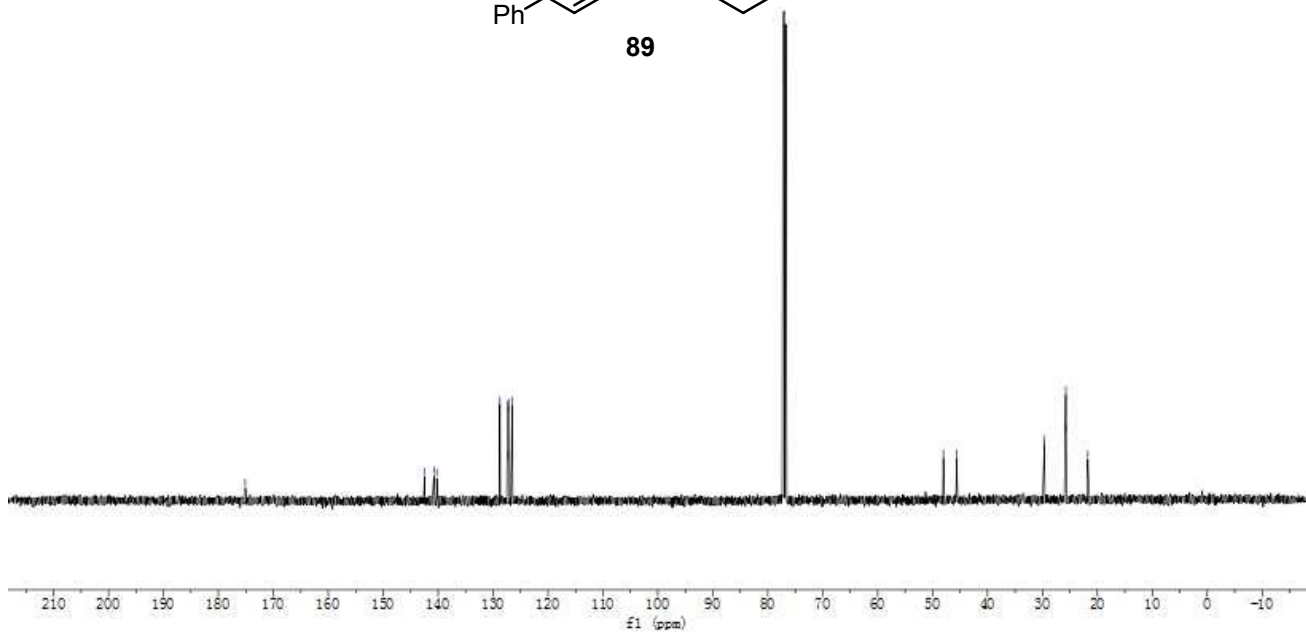
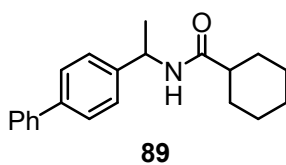


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$^{13}\text{C}$  NMR  
126 MHz  
 $\text{CDCl}_3$

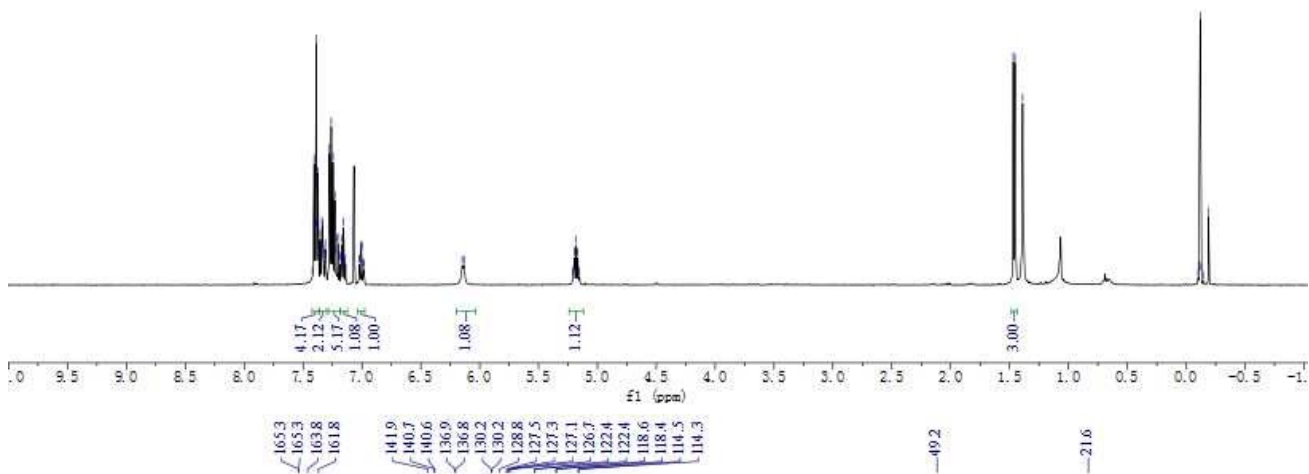
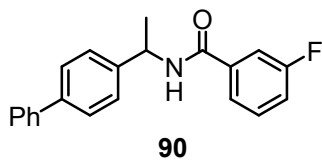


$^{13}\text{C}$  NMR  
126 MHz  
 $\text{CDCl}_3$

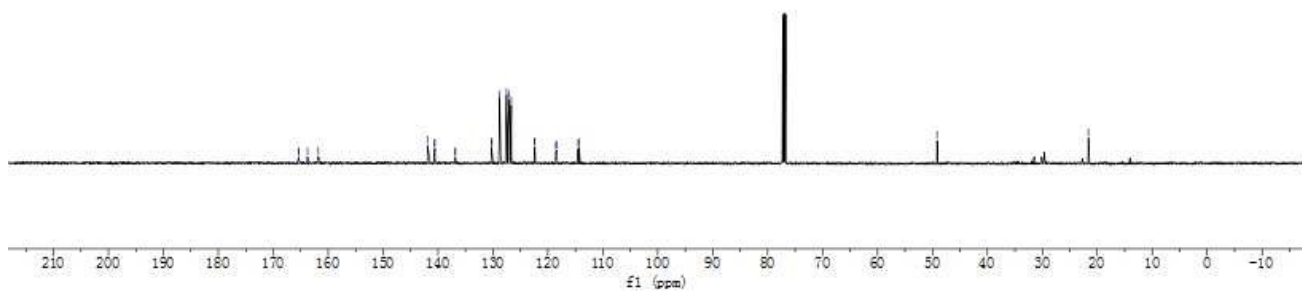
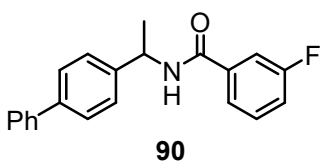


7.42 CDCl<sub>3</sub>

<sup>1</sup>H NMR  
500 MHz  
CDCl<sub>3</sub>

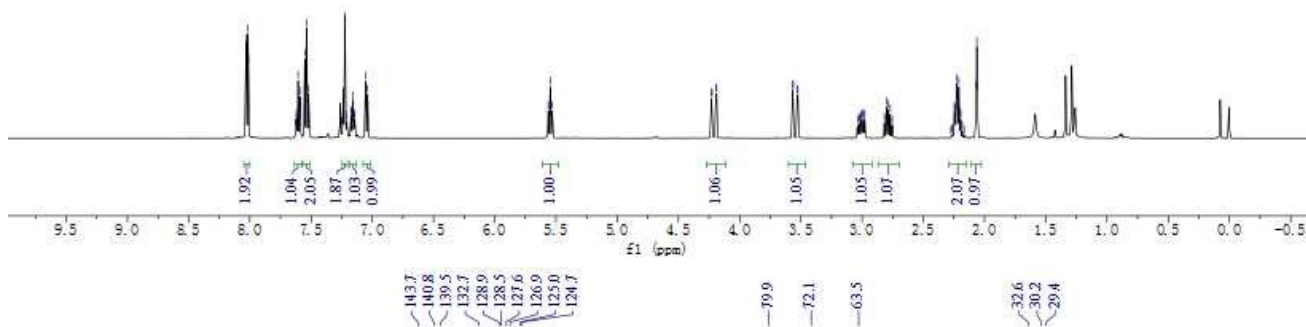
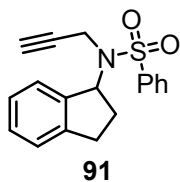


<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>

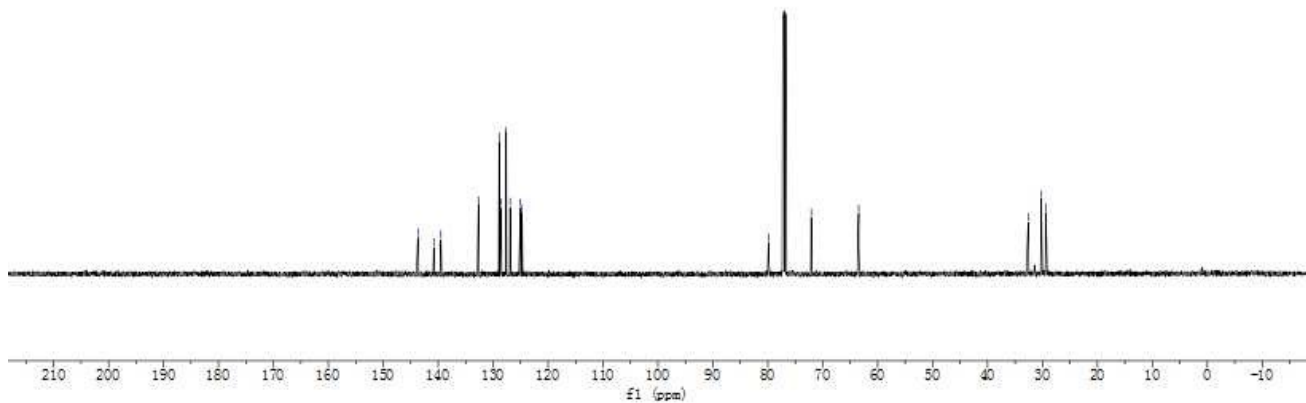
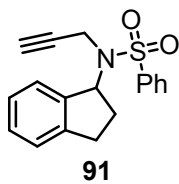


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<sup>1</sup>H NMR  
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CDCl<sub>3</sub>



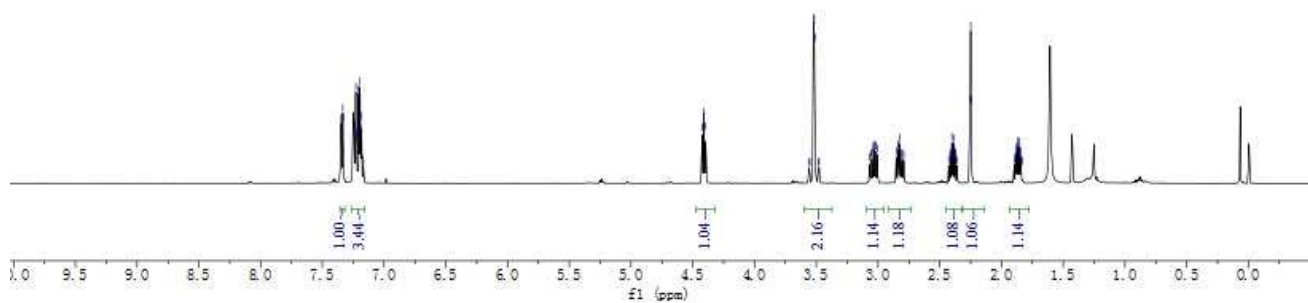
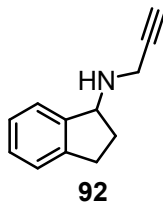
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126 MHz  
CDCl<sub>3</sub>



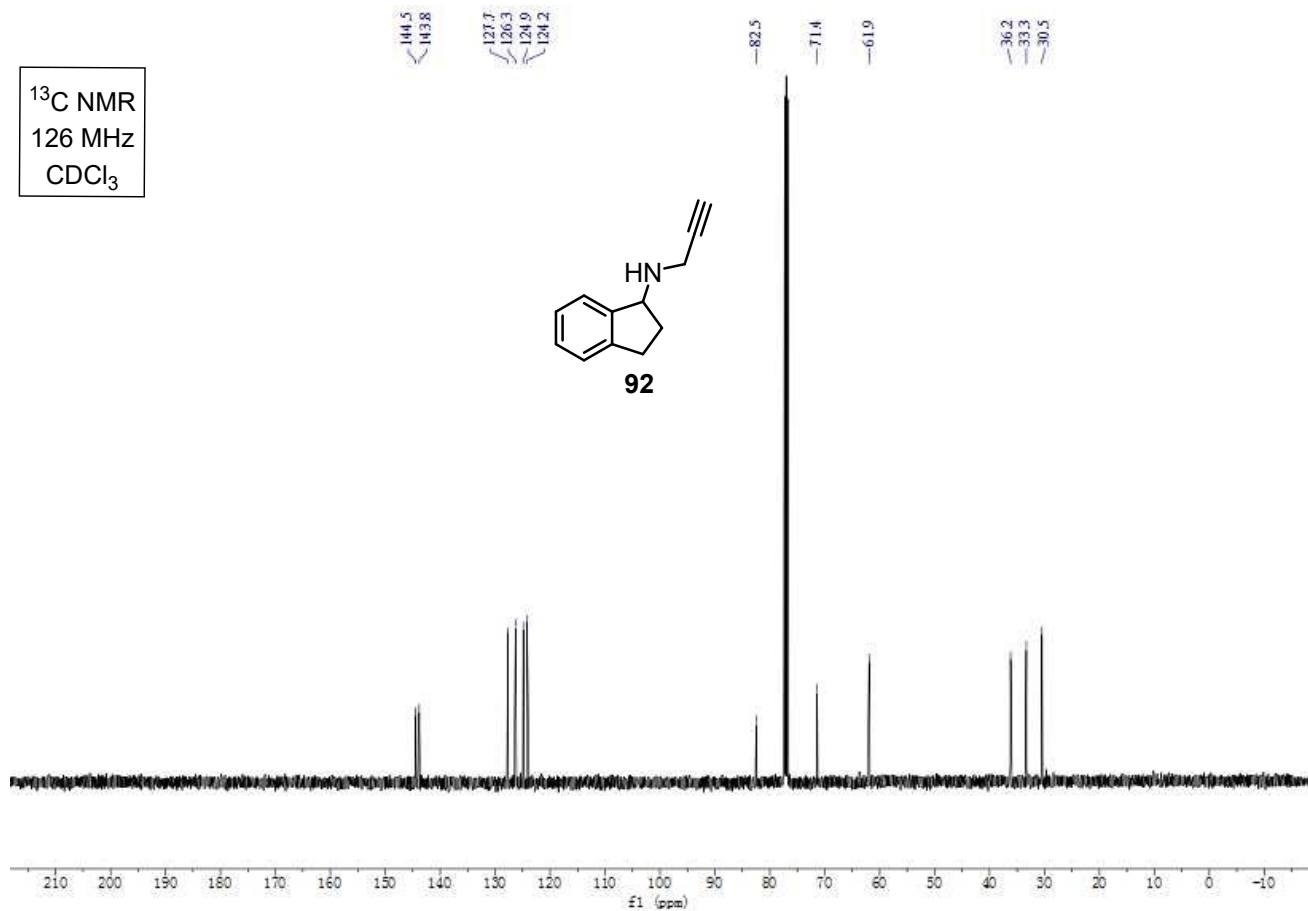
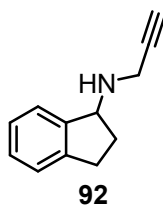


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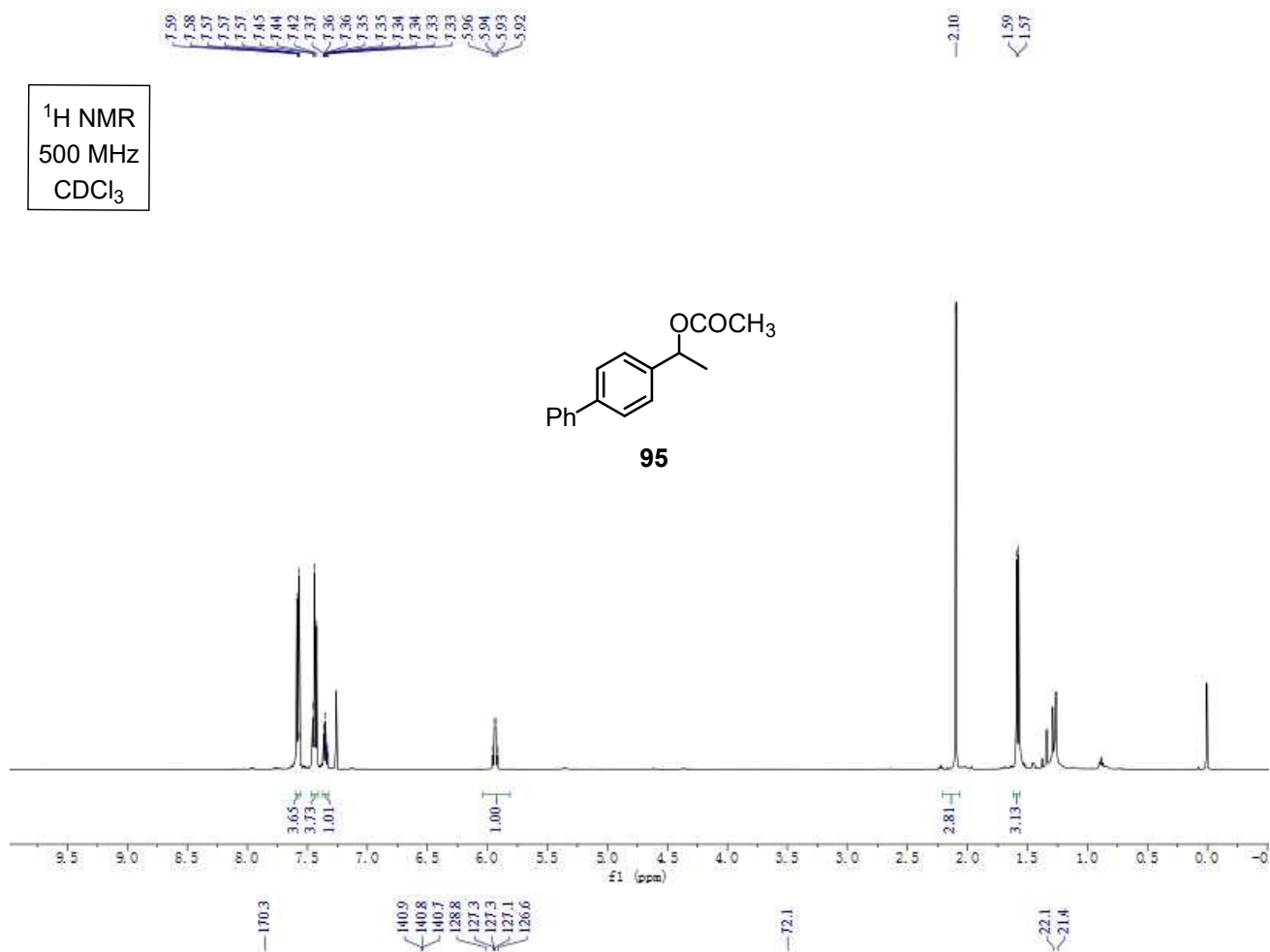
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500 MHz  
CDCl<sub>3</sub>



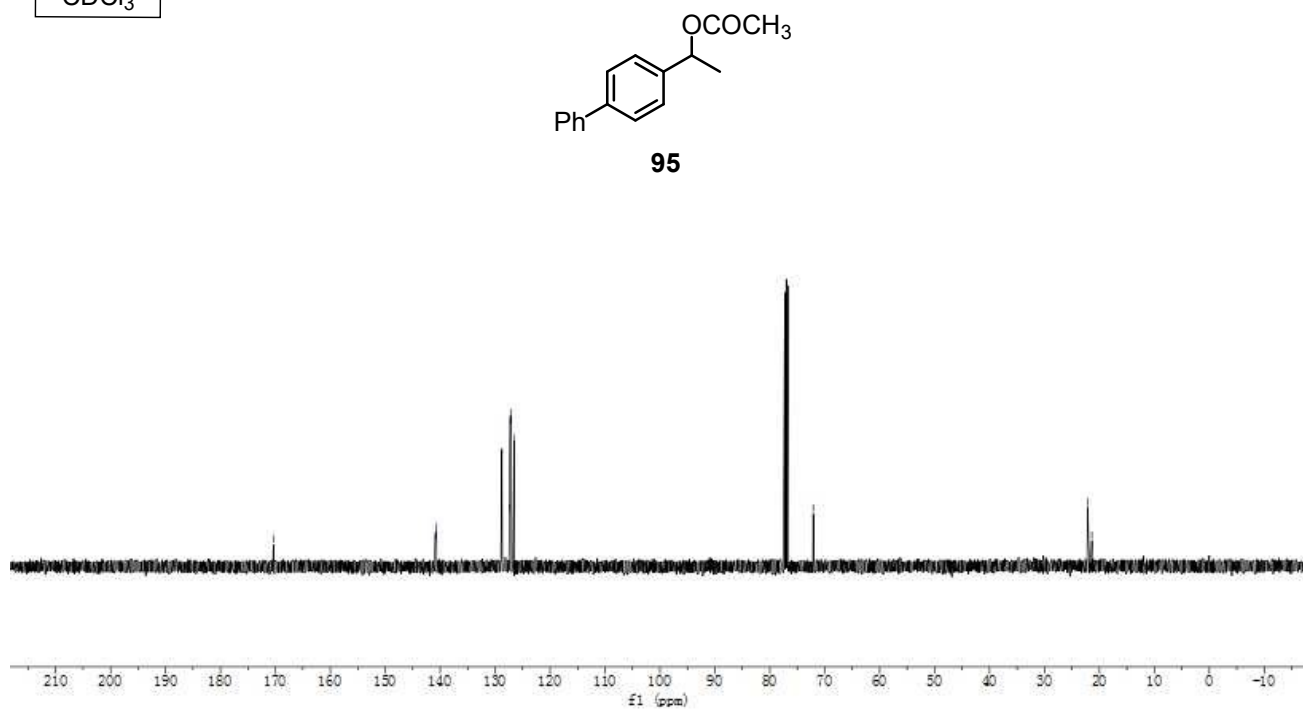
<sup>13</sup>C NMR  
126 MHz  
CDCl<sub>3</sub>



$^1\text{H}$  NMR  
500 MHz  
 $\text{CDCl}_3$



$^{13}\text{C}$  NMR  
126 MHz  
 $\text{CDCl}_3$



## 8. References

1. B. Karimi and P. F. Akhavan. *Chem. Commun.*, **2009**, 3750–3752.
2. Z.-W. Hou, D.-J. Liu, P. Xiong, X.-L. Lai, J. Song and H-C. Xu, *Angew. Chem. Int. Ed.*, 2021, **60**, 2943–2947.
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