

## Supporting Information

### Nickel-Catalyzed Electrochemical Reductive Relay Cross-Coupling of Alkyl Halides with Alkyl Carboxylic Acids

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## 1 General Information

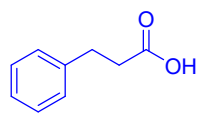
All the electrochemical reduction were performed in an undivided cell equipped with Nickel foam (1.5×2.5 cm<sup>2</sup>) and an iron rod as sacrificial anode unless otherwise noted. Solvents and commercially available reagents were used without purification. Column chromatography was performed using either 100-200 Mesh or 300-400 Mesh silica gel. Visualization of spots on TLC plate was accomplished with UV light (254 nm).

All the nickel foams were purchased from T-mall, China. The potentiostat was purchased from Shiqiang Telecom Co., Ltd, Shengzhen, China. The All commercial reagents were purchased from TCI, Sigma-Aldrich, Adamas-beta chemistry and Energy Chemical of the highest purity grade. They were used without further purification unless specified.

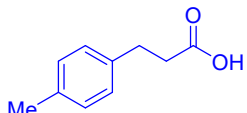
<sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on Agilent AV 400, Varian Inova 400 (400 MHz and 101 MHz, respectively). <sup>19</sup>F NMR spectra were recorded on Agilent AV 400, Varian Inova 400 (376 MHz) instrument. The peaks were internally referenced to TMS (0.00 ppm) or residual undeuterated solvent signal. The following abbreviations were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br = broad. Infrared spectra were obtained on a Bio-Rad FTS-185 instrument. High resolution mass spectra were recorded at the Center for Mass Spectrometry, Shanghai Institute of Organic Chemistry. Analytical and spectral data of all those known compounds are exactly matching with the reported values.

## 2 Structures of Starting Materials

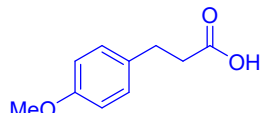
### Alkyl acids



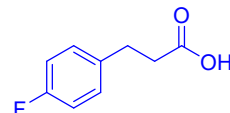
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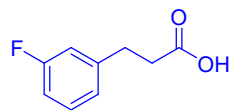
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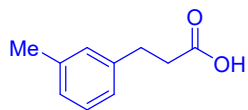
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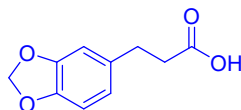
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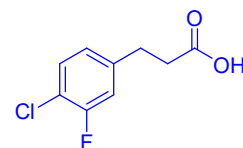
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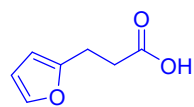
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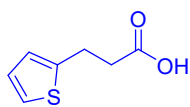
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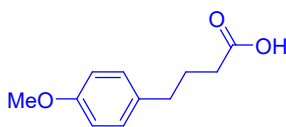
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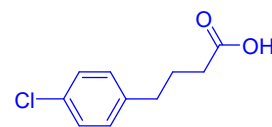
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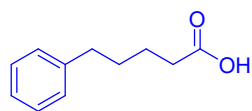
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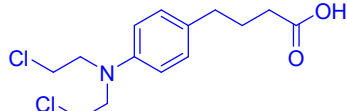
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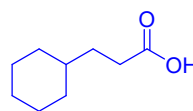
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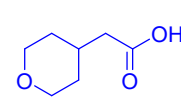
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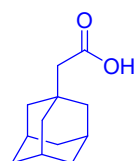
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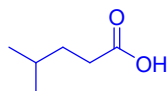
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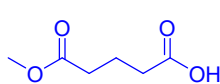
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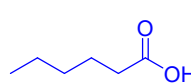
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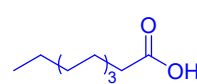
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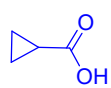
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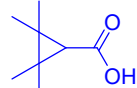
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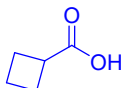
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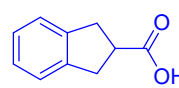
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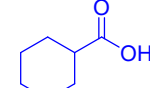
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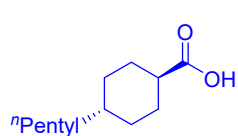
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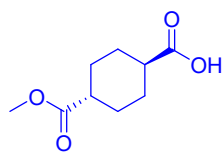
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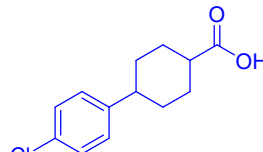
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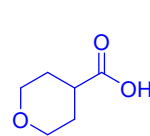
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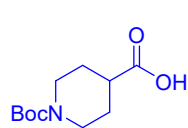
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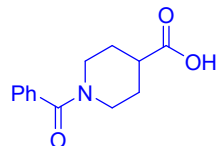
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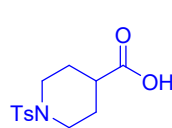
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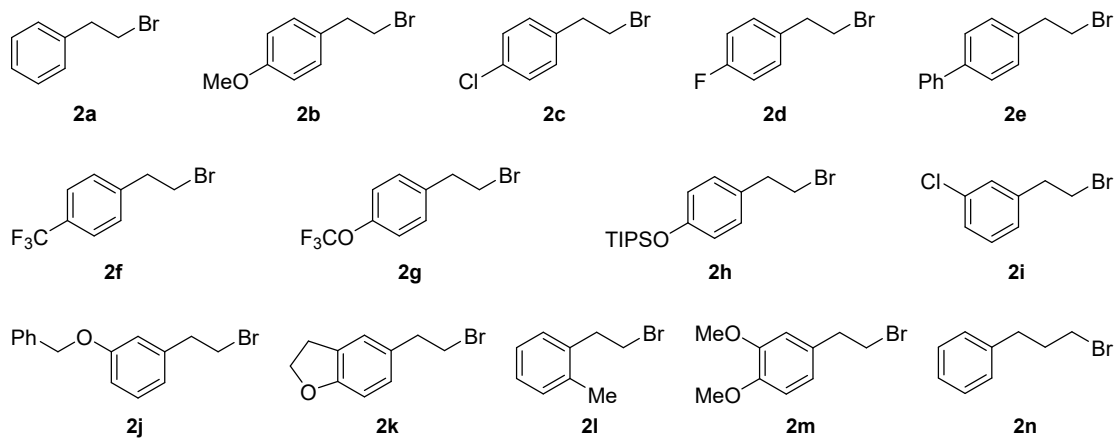


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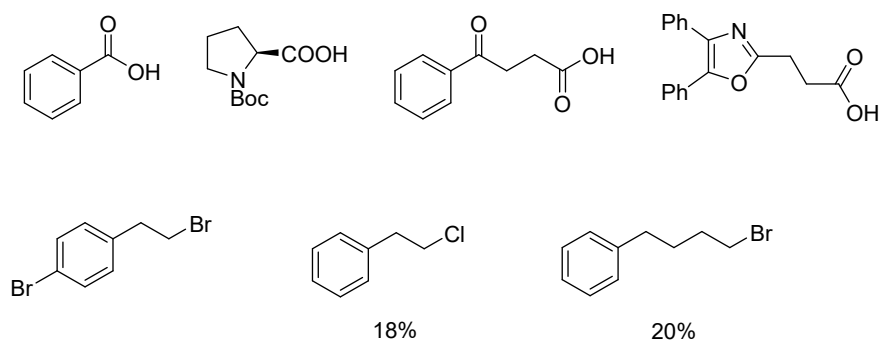


1ag

## Alkyl bromides

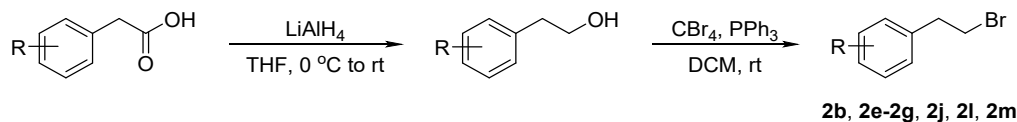


## Failed substrates



### 3 Preparation of alkyl bromide substrates

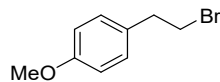
General procedure for the synthesis of **2b**, **2e-2g**, **2j**, **2l** and **2m**.



**Procedure A:** LiAlH<sub>4</sub> (1.2 equiv.) and anhydrous THF (c = 1.25 M) were added to the flask. Then a solution of acid (1.0 equiv.) in anhydrous THF (c = 1.25 M) was added dropwise under the ice bath. The mixture was stirred at room temperature for 1 h. then the reaction was quenched with MeOH and NaOH (10% in aqueous), followed by workup with hydrochloric acid (1 M). The mixture was extracted with EtOAc (3 equal volume) and the organic layer was washed with brine, dried with Na<sub>2</sub>SO<sub>4</sub>, and concentrated by flash evaporation for the next step without further purification.

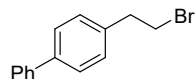
**Procedure B:** To a stirred solution of alcohol (1.0 equiv.) and CBr<sub>4</sub> (1.2 equiv.) in DCM (0.3 M) was added triphenylphosphine (1.2 equiv.). After stirring at room temperature for 5 h, the mixture was concentrated by flash evaporation. The residue was added with petroleum ether and filtered. The solution was concentrated by flash evaporation and the crude product was purified by flash column chromatography on silica to afford the product.

1-(2-bromoethyl)-4-methoxybenzene (**2b**)<sup>[1]</sup>



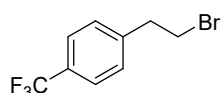
From 2-(4-methoxyphenyl)acetic acid (3.32 g, 20 mmol), following the general procedure A and B, the title compound (**2b**) was obtained (2.6 g, 60% in two steps) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.18 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 3.84 (s, 3H), 3.58 (t, *J* = 7.6 Hz, 2H), 3.15 (t, *J* = 7.6 Hz, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 158.51, 130.98, 129.66, 113.97, 55.23, 38.56, 33.47.

4-(2-bromoethyl)-1,1'-biphenyl (**2e**)<sup>[1]</sup>



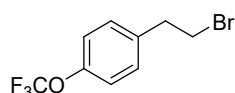
From 2-([1,1'-biphenyl]-4-yl)acetic acid (4.25 g, 20 mmol), following the general procedure A and B, the title compound (**2e**) was obtained (4.06 g, 78% in two steps) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.64 (d, *J* = 7.2 Hz, 2H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.43 – 7.37 (m, 1H), 7.33 (d, *J* = 8.0 Hz, 2H), 3.64 (t, *J* = 7.6 Hz, 2H), 3.25 (t, *J* = 7.6 Hz, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 140.91, 140.02, 138.08, 129.26, 128.95, 127.50, 127.44, 127.20, 39.19, 33.08.

1-(2-bromoethyl)-4-(trifluoromethyl)benzene (**2f**) [2]



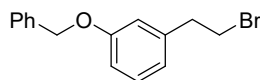
From 2-(4-(trifluoromethyl)phenyl)ethan-1-ol (796 mg, 4.2 mmol), following the general procedure B, the title compound (**2f**) was obtained (780 mg, 73%) as a colorless oil. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.62 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 3.62 (t, *J* = 7.6 Hz, 2H), 3.26 (t, *J* = 7.6 Hz, 2H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 142.81 (br m), 129.29 (q, *J* = 32.0 Hz), 129.06, 125.55 (q, *J* = 3.8 Hz), 124.21 (q, *J* = 270.1 Hz), 38.88, 32.15. **<sup>19</sup>F NMR** (376 MHz, CDCl<sub>3</sub>) δ -62.46.

1-(2-bromoethyl)-4-(trifluoromethoxy)benzene (**2g**) [3]



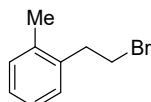
From 2-(4-(trifluoromethoxy)phenyl)acetic acid (2.20 g, 10 mmol), following the general procedure A and B, the title compound (**2g**) was obtained (1.56 g, 58% in two steps) as a colorless oil. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.27 (d, *J* = 8.4 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 3.59 (t, *J* = 7.6 Hz, 2H), 3.20 (t, *J* = 7.6 Hz, 2H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 148.19, 137.56, 130.05, 121.15, 120.49 (q, *J* = 256.1 Hz), 38.52, 32.58. **<sup>19</sup>F NMR** (376 MHz, CDCl<sub>3</sub>) δ -57.91.

1-(benzyloxy)-3-(2-bromoethyl)benzene (**2j**)



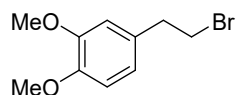
From 2-(3-(benzyloxy)phenyl)acetic acid (2.42 g, 10 mmol), following the general procedure A and B, the title compound (**2j**) was obtained (2.08 g, 71% in two steps) as a colorless oil. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.52 – 7.37 (m, 5H), 7.30 (t, *J* = 7.6 Hz, 1H), 6.97 – 6.86 (m, 3H), 5.11 (s, 2H), 3.61 (t, *J* = 7.6 Hz, 2H), 3.19 (t, *J* = 7.6 Hz, 2H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 159.16, 140.65, 137.11, 129.83, 128.79, 128.18, 127.71, 121.44, 115.61, 113.25, 70.12, 39.60, 32.99. **HRMS** (EI) calculated for C<sub>15</sub>H<sub>15</sub>BrO [M]<sup>+</sup> 290.0301, measured: 290.0302. **IR** (neat) 3031, 1592, 1491, 1259, 1018, 740, 696, 626, 540, 448 cm<sup>-1</sup>.

1-(2-bromoethyl)-2-methylbenzene (**2i**) [4]



From 2-(*o*-tolyl)acetic acid (3.00 g, 20 mmol), following the general procedure A and B, the title compound (**2l**) was obtained (2.20 g, 55% in two steps) as a yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.29 – 7.22 (m, 4H), 3.60 (t, *J* = 7.6 Hz, 2H), 3.26 (t, *J* = 7.6 Hz, 2H), 2.42 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 137.21, 136.17, 130.62, 129.40, 127.20, 126.32, 37.04, 31.76, 19.37.

4-(2-bromoethyl)-1,2-dimethoxybenzene (**2m**) [5]



From 2-(3,4-dimethoxyphenyl)ethan-1-ol (1.82 g, 10 mmol), following the general procedure B, the title compound (**2m**) was obtained (2.25 g, 91%) as a white solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.84 (d, *J* = 8.0 Hz, 1H), 6.79 – 6.74 (m, 2H), 3.90 (s, 3H), 3.89 (s, 3H), 3.57 (t, *J* = 7.6 Hz, 2H), 3.12 (t, *J* = 7.6 Hz, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 148.94, 147.97, 131.51, 120.69, 111.86, 111.23, 55.91, 55.88, 39.10, 33.31.

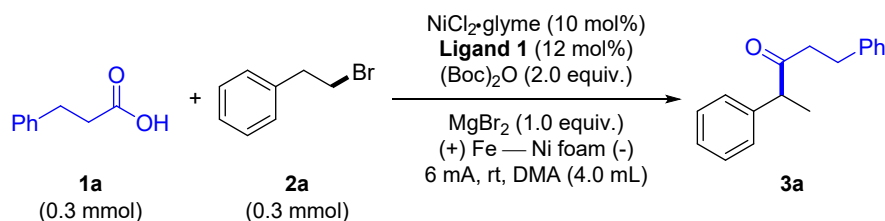
(4-(2-bromoethyl)phenoxy)triisopropylsilane (**2h**) [6]



To a solution of 4-(2-bromoethyl)phenol (1.0 g, 4.97 mmol, 1.0 equiv.) in anhydrous CH<sub>2</sub>Cl<sub>2</sub> (30 mL) was added imidazole (0.36 g, 5.22 mmol, 1.05 equiv.), followed by dropwise addition of TIPSCl (1.06 mL, 4.97 mmol, 1.0 equiv.). After stirring for 16 h, the cloudy reaction mixture was transferred to a separatory funnel and washed with saturated aqueous NH<sub>4</sub>Cl and H<sub>2</sub>O. The aqueous phase was extracted twice with CH<sub>2</sub>Cl<sub>2</sub>, and the combined extracts were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated. The crude product was purified by flash column chromatography on silica to afford the title product (**2h**) (1.30 g, 73%) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.08 (d, *J* = 8.4 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 2H), 3.55 (t, *J* = 7.6 Hz, 2H), 3.11 (t, *J* = 7.6 Hz, 2H), 1.33 – 1.23 (m, 3H), 1.13 (d, *J* = 7.2 Hz, 18H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 155.02, 131.38, 129.59, 119.99, 38.81, 33.36, 17.95, 12.67.

## 4 Conditions Screening of the Reaction<sup>a</sup>

Table S1

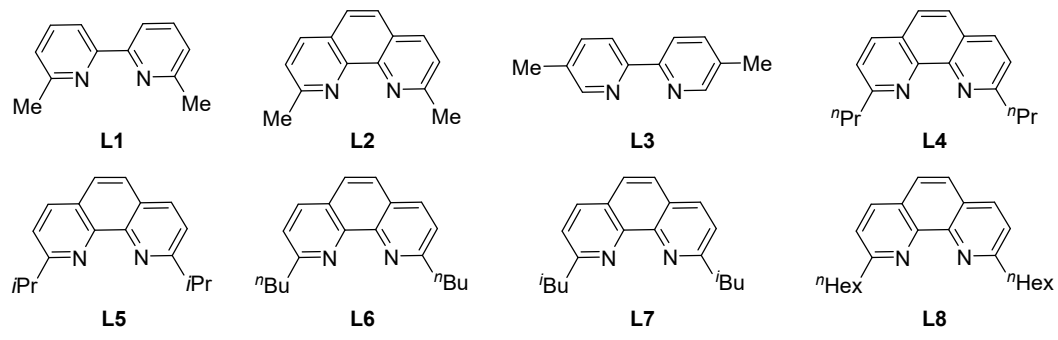


entry	deviation from above condition	F/mol	yield(%) <sup>b</sup>
1	no	4.5	77(72) <sup>c</sup>
2	without electricity	0	NP
3	$\text{NiI}_2$ as the catalyst	4.5	21
4	$\text{Ni}(\text{BF}_4)_2 \cdot 6\text{H}_2\text{O}$ as the catalyst	4.5	58
5	$\text{Ni}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ as the catalyst	4.5	50
6	<b>L3</b> as the ligand	4.5	NP
7	<b>L4</b> as the ligand	4.5	64
8	<b>L5</b> as the ligand	4.5	66
9	<b>L6</b> as the ligand	4.5	40
10	<b>L7</b> as the ligand	4.5	31
11	<b>L8</b> as the ligand	4.5	40
12	[Ni]/ <b>L1</b> (5/6 mol%)	4.5	57
13	$\text{MgBr}_2$ (2.0 equiv.)	4.5	33
14	$\text{MgBr}_2$ (0.5 equiv.)	4.5	50
15	<sup>n</sup> $\text{Bu}_4\text{NBF}_4$ in lieu of $\text{MgBr}_2$	4.5	0
16	Zn as the anode	4.5	46
17	5 mA	3.8	70
18	7 mA	5.3	64
19	8 mA	6.0	57
20	DMA( 3.0 mL)	4.5	56
21	<b>2a</b> (1.2 equiv)	4.5	70
22	<b>2a</b> (2.0 equiv)	4.5	35
23	60 °C	4.5	18
24	40 °C	4.5	35
25	$\text{NaI}$ (1.0 equiv.) as additive	4.5	72
26	$\text{LiI}$ (1.0 equiv.) as additive	4.5	73
27	$\text{ZnI}_2$ (1.0 equiv.) as additive	4.5	25

<sup>a</sup>Reaction conditions: **1a** (0.3 mmol), **2a** (0.3 mmol),  $\text{NiCl}_2 \cdot \text{glyme}$  (10 mol%), **Ligand 1** (12 mol%),  $\text{MgBr}_2$  (0.3 mmol, 1.0 equiv.) and DMA (4 mL) in an undivided cell with a Ni foam cathode and an iron rod as sacrificial anode. <sup>b</sup>The yields were determined by <sup>1</sup>H-NMR with  $\text{CH}_2\text{Br}_2$  as the internal standard.

<sup>c</sup>Isolated yield of **3a**.



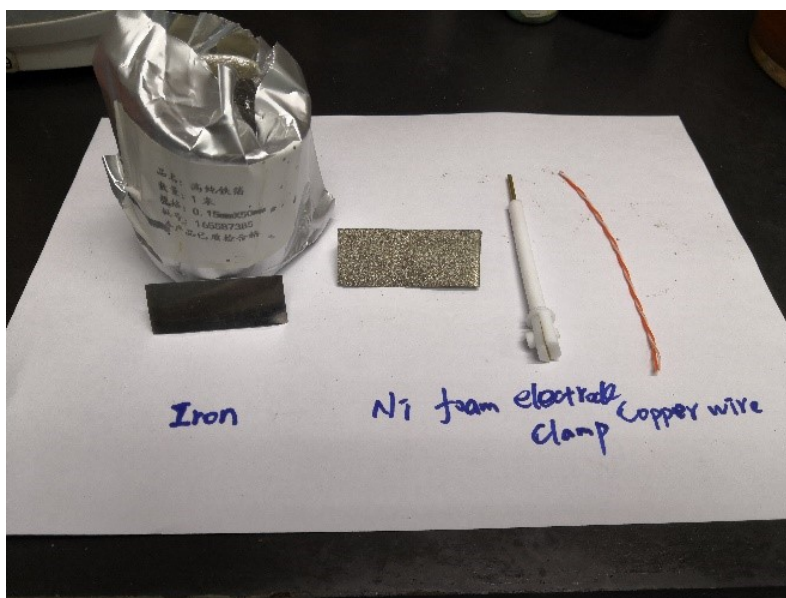


## 5 Photographic Guide for Electrochemical Reductive Relay Cross-Couplings

### 1 Easily hand-made electrochemical cell

#### Step 0. Overview of materials used.

From left to right: 1) The iron rod 2) The nickel foam cathode 3) The electrode clamp. 4) copper wire



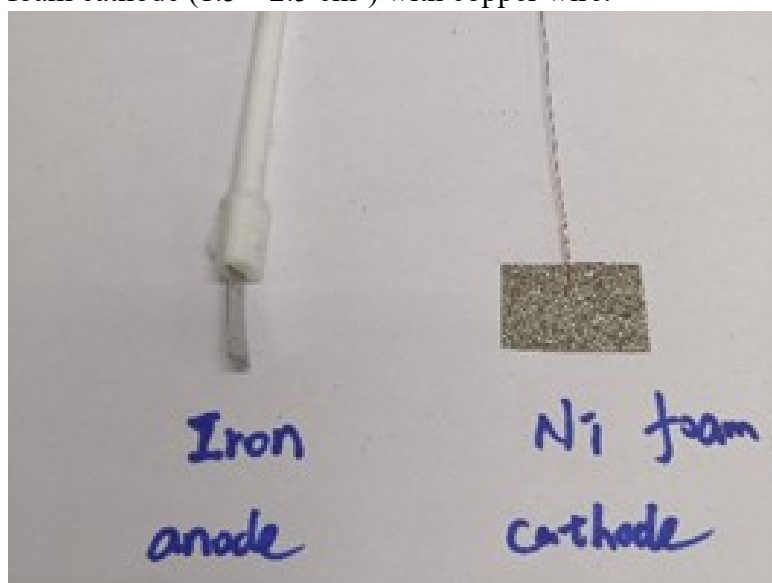
#### Step 1. Preparation of two electrodes.

Cut an iron rod about  $0.5 \times 1.5 \text{ cm}^2$  with a scissors.

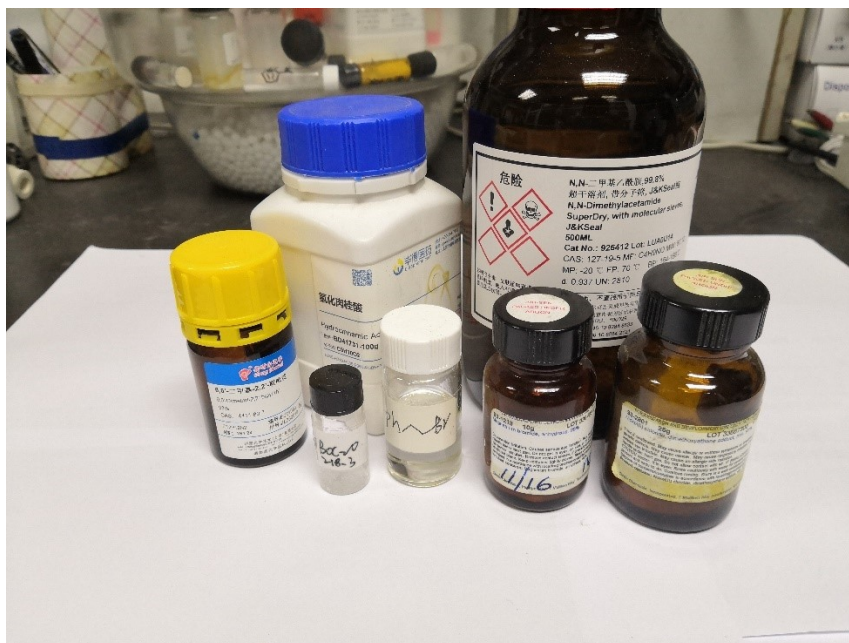
Attach the iron with the electrode clamp.

Strip the protective skin of the copper wire with a tweezer.

Wrap the Ni foam cathode ( $1.5 \times 2.5 \text{ cm}^2$ ) with copper wire.



## 2 Graphical Guide for Electrochemical Reductive Relay Cross-Couplings.



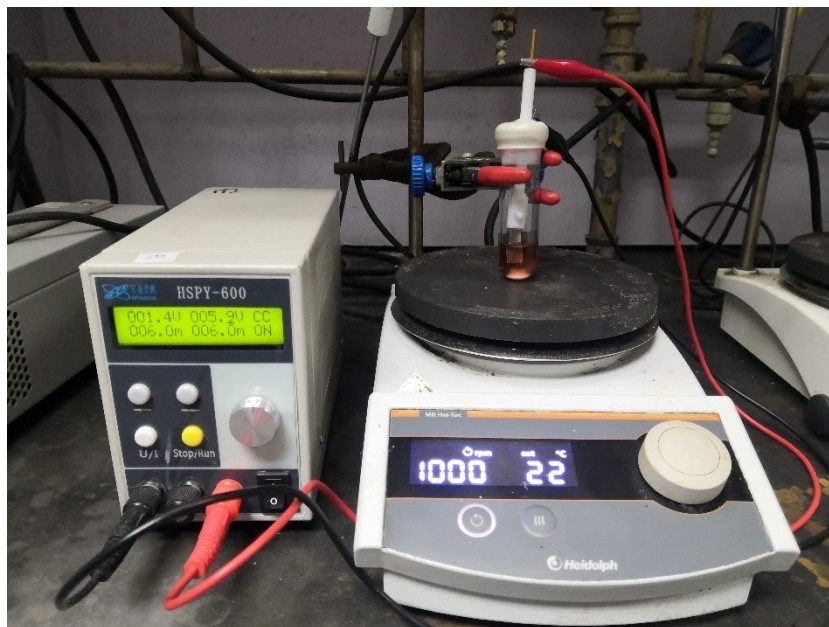
Materials used in the reaction.

Step 1. Weight the ligand and the hydrocinnamic acid in a 10 mL hydrogenation tube.

Step 2. Transfer the vial to a Nitrogen-filled glove box. Weight the  $\text{MgBr}_2$  and Nickel catalyst, then install the two electrodes. Remove the tube from the glove box.



- Step 3. The alkyl bromide and  $\text{Boc}_2\text{O}$  dissolved in anhydrous DMA (4.0 mL) was injected into the tube with a 5 mL syringe. And the tube was sealed with parafilm.
- Step 4. After stirring for 30 minutes, attached to electrode (the red (+) to the electrode clamp, the black (-) to the copper wire). The reaction was electrolyzed for 6 h under a constant-current electrolysis at 6 mA.

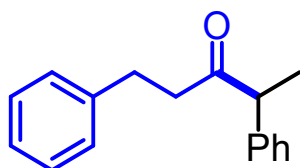


## 6 General Procedure for the Electrolysis

To a 10 mL hydrogenation tube charged with a stir bar was added the acids and **ligand 1** (6.6 mg, 12 mol%). The vial was then introduced in a Nitrogen-filled glove box. Weigh the NiCl<sub>2</sub>·glyme (6.6 mg, 10 mol%) and MgBr<sub>2</sub> (55.2 mg, 1.0 equiv) into the tube and install a Ni foam cathode (2.5 x 1.5 cm<sup>2</sup>) and an iron rod anode. The tube was then removed from the glove box. Next the Boc<sub>2</sub>O and the alkyl bromide dissolved in anhydrous DMA (4.0 mL) was injected into the tube with a 5 mL syringe. The reaction mixture was stirred at about 1000 rpm for 30 minutes. After that, the reaction mixture was electrolyzed for 6 h under a constant-current electrolysis at 6 mA. After the reaction was completed, the mixture was diluted with EtOAc (about 40 mL) and washed with sat. NH<sub>4</sub>Cl (3 x equal volume), dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated *in vacuo*. The crude product was purified by column chromatography to furnish the desired product.

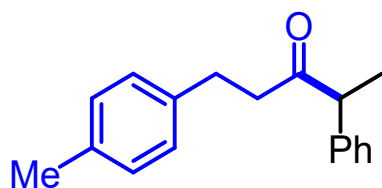
### Characterization Data for the Products

#### 1,4-diphenylpentan-3-one (**3a**)<sup>[7]</sup>



The title product was prepared according to the general procedure with 3-phenylpropionic acid (**1a**) (45.0 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3a** (51.5 mg, 72%) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.30 (t, *J* = 7.2 Hz, 2H), 7.24 – 7.18 (m, 3H), 7.17 – 7.14 (m, 3H), 7.06 (d, *J* = 7.2 Hz, 2H), 3.70 (q, *J* = 6.8 Hz, 1H), 2.89 – 2.71 (m, 2H), 2.70 – 2.61 (m, 2H), 1.37 (d, *J* = 6.8 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 209.9, 141.1, 140.5, 128.9, 128.4, 128.3, 127.9, 127.2, 126.0, 53.2, 42.6, 30.0, 17.3.

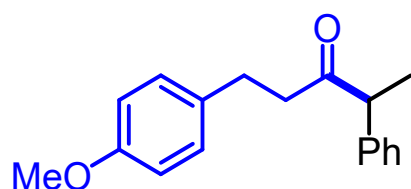
#### 4-phenyl-1-(p-tolyl)pentan-3-one (**3b**)



The title product was prepared according to the general procedure with 3-(p-

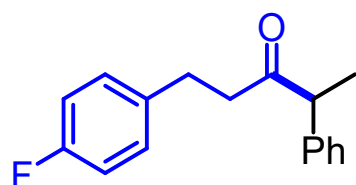
tolyl)propanoic acid (**1b**) (49.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3b** ((35.4 mg, 52%) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.21 (t, *J* = 7.2 Hz, 2H), 7.18 – 7.12 (m, 1H), 7.08 (d, *J* = 6.8 Hz, 2H), 6.94 (d, *J* = 8.0 Hz, 2H), 6.87 (d, *J* = 8.0 Hz, 2H), 3.61 (q, *J* = 6.8 Hz, 1H), 2.77 – 2.60 (m, 2H), 2.59 – 2.52 (m, 2H), 2.20 (s, 3H), 1.29 (d, *J* = 6.8 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 209.0, 139.5, 136.9, 134.4, 128.0, 127.9, 127.1, 126.8, 126.1, 52.2, 41.7, 28.5, 19.9, 16.3. HRMS (EI) calculated for C<sub>18</sub>H<sub>20</sub>O [M]<sup>+</sup> 252.1509, measured: 252.1507. IR (neat) 2927, 1711, 1514, 1451, 1259, 1020, 808, 699, 528, 481 cm<sup>-1</sup>.

### 1-(4-methoxyphenyl)-4-phenylpentan-3-one (**3c**)<sup>[8]</sup>



The title product was prepared according to the general procedure with 3-(4-methoxyphenyl)propanoic acid (**1c**) (54.1 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3c** (56.4 mg, 70%) as a pale yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.34 – 7.22 (m, 3H), 7.16 (d, *J* = 7.2 Hz, 2H), 6.99 (d, *J* = 8.8 Hz, 2H), 6.77 (d, *J* = 8.4 Hz, 2H), 3.77 (s, 3H), 3.70 (q, *J* = 6.8 Hz, 1H), 2.84 – 2.69 (m, 2H), 2.67 – 2.56 (m, 2H), 1.38 (d, *J* = 6.8 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 210.0, 157.9, 140.5, 133.1, 129.2, 128.9, 127.9, 127.1, 113.8, 55.2, 53.2, 42.9, 29.1, 17.3.

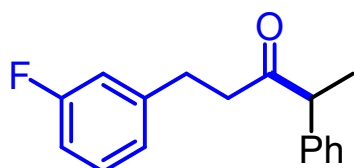
### 1-(4-fluorophenyl)-4-phenylpentan-3-one (**3d**)



The title product was prepared according to the general procedure with 3-(4-fluorophenyl)propanoic acid (**1d**) (50.5 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3d** (50.0 mg, 65%) as a yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ

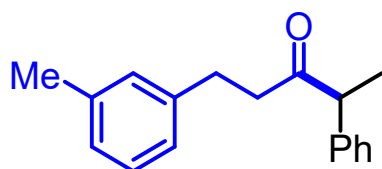
7.32 – 6.86 (m, 9H), 3.70 (q,  $J = 6.8$  Hz, 1H), 2.86 – 2.71 (m, 2H), 2.69 – 2.56 (m, 2H), 1.38 (d,  $J = 6.8$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  209.7, 161.4 (d,  $J = 243.7$  Hz), 140.4, 136.7 (d,  $J = 3.2$  Hz), 129.8 (d,  $J = 7.8$  Hz), 129.1, 128.0, 127.3, 115.2 (d,  $J = 21.1$  Hz), 53.3, 42.6, 29.2, 17.4.  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -117.45. HRMS (EI) calculated for  $\text{C}_{17}\text{H}_{17}\text{FO}$   $[\text{M}]^+$  256.1258, measured: 256.1257. IR (neat) 2930, 1711, 1508, 1219, 1156, 825, 699, 530, 481  $\text{cm}^{-1}$ .

#### 1-(3-fluorophenyl)-4-phenylpentan-3-one (3e)



The title product was prepared according to the general procedure with 3-(3-fluorophenyl)propanoic acid (**1e**) (50.5 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3e** (45.0 mg, 58%) as a pale yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.34 – 7.21 (m, 3H), 7.20 – 7.11 (m, 3H), 6.86 – 6.79 (m, 2H), 6.76 – 6.70 (m, 1H), 3.70 (q,  $J = 6.8$  Hz, 1H), 2.88 – 2.72 (m, 2H), 2.71 – 2.57 (m, 2H), 1.38 (d,  $J = 6.8$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  209.4, 162.8 (d,  $J = 245.4$  Hz), 143.6 (d,  $J = 7.3$  Hz), 140.3, 129.8 (d,  $J = 8.3$  Hz), 129.0, 127.9, 127.2, 124.0 (d,  $J = 2.8$  Hz), 115.1 (d,  $J = 21.0$  Hz), 112.9 (d,  $J = 21.0$  Hz), 53.2, 42.1, 29.6, 17.3.  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -113.64. HRMS (EI) calculated for  $\text{C}_{17}\text{H}_{17}\text{FO}$   $[\text{M}]^+$  256.1258, measured: 256.1257. IR (neat) 2931, 1712, 1587, 1489, 1449, 1247, 1139, 782, 699, 537  $\text{cm}^{-1}$ .

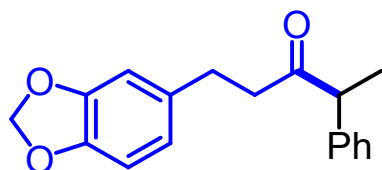
#### 4-phenyl-1-(m-tolyl)pentan-3-one (3f)



The title product was prepared according to the general procedure with 3-(m-tolyl)propanoic acid (**1f**) (49.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3f** (45.5 mg, 60%) as a yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.32 – 7.27 (m, 2H), 7.25 – 7.21 (m, 1H), 7.17 – 7.15 (m, 2H), 7.12 – 7.08 (m, 1H), 6.96 (d,

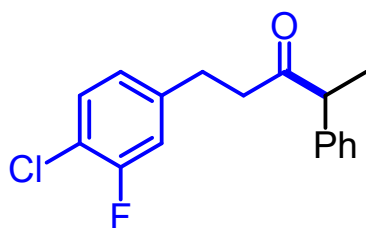
$J = 7.2$  Hz, 1H), 6.86 – 6.85 (m, 2H), 3.70 (q,  $J = 6.8$  Hz, 1H), 2.85 – 2.70 (m, 2H), 2.69 – 2.61 (m, 2H), 2.27 (s, 3H), 1.37 (d,  $J = 6.8$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  210.0, 141.0, 140.5, 138.0, 129.1, 128.9, 128.3, 127.9, 127.2, 122.8, 125.3, 53.2, 42.7, 29.9, 21.4, 17.4. HRMS (EI) calculated for  $\text{C}_{18}\text{H}_{20}\text{O}$   $[\text{M}]^+$  252.1509, measured: 252.1509. IR (neat) 2928, 1712, 1608, 1492, 1451, 1122, 782, 699, 548  $\text{cm}^{-1}$ .

### 1-(benzo[d][1,3]dioxol-5-yl)-4-phenylpentan-3-one (3g)



The title product was prepared according to the general procedure with 3-(benzo[d][1,3]dioxol-5-yl)propanoic acid (**1g**) (58.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3g** (52.5 mg, 62%) as a pale yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.36 – 7.31 (m, 2H), 7.30 – 7.25 (m, 1H), 7.22 – 7.16 (m, 2H), 6.69 (d,  $J = 7.6$  Hz, 1H), 6.57 (s, 1H), 6.54 (d,  $J = 8.0$  Hz, 1H), 5.91 (s, 2H), 3.73 (q,  $J = 6.8$  Hz, 1H), 2.84 – 2.59 (m, 4H), 1.41 (d,  $J = 6.8$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  209.8, 147.5, 145.8, 140.4, 134.9, 129.0, 127.9, 127.2, 121.1, 108.8, 108.2, 100.8, 53.3, 42.8, 29.7, 17.3. HRMS (ESI) calculated for  $\text{C}_{18}\text{H}_{18}\text{O}_3\text{Na}$   $[\text{M}+\text{Na}]^+$  305.1148, measured: 305.1148. IR (neat) 2892, 1710, 1488, 1442, 1243, 1037, 928, 808, 731, 699, 545  $\text{cm}^{-1}$ .

### 1-(4-chloro-3-fluorophenyl)-4-phenylpentan-3-one (3h)

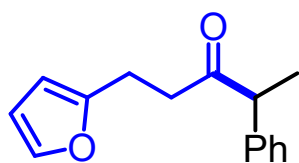


The title product was prepared according to the general procedure with 3-(4-chloro-3-fluorophenyl)propanoic acid (**1h**) (60.8 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3h** (43.6 mg, 50%) as a pale yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



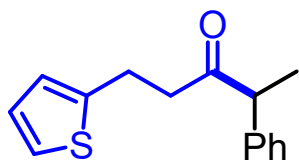
$\delta$  7.34 – 7.25 (m, 3H), 7.21 (t,  $J$  = 8.0 Hz, 1H), 7.18 – 7.12 (m, 2H), 6.83 – 6.77 (m, 2H), 3.73 (q,  $J$  = 6.8 Hz, 1H), 2.93 – 2.51 (m, 4H), 1.40 (d,  $J$  = 6.8 Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  209.0, 157.8 (d,  $J$  = 248.3 Hz), 142.0 (d,  $J$  = 6.5 Hz), 140.0, 130.3, 129.0, 127.8, 127.3, 124.8 (d,  $J$  = 3.5 Hz), 118.3 (d,  $J$  = 17.6 Hz), 116.5 (d,  $J$  = 20.7 Hz), 53.4, 41.8, 29.1, 17.2.  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -115.93. HRMS (EI) calculated for  $\text{C}_{17}\text{H}_{16}\text{ClFO}$   $[\text{M}]^+$  290.0868, measured: 290.0870. IR (neat) 2932, 1712, 1580, 1490, 1373, 1242, 1062, 867, 699, 500  $\text{cm}^{-1}$ .

### 1-(furan-2-yl)-4-phenylpentan-3-one (3i)



The title product was prepared according to the general procedure with 3-(furan-2-yl)propanoic acid (**1i**) (42.0 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3i** (43.2 mg, 63%) as a colorless oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.32 (t,  $J$  = 7.2 Hz, 2H), 7.27 – 7.24 (m, 1H), 7.23 – 7.21 (m, 1H), 7.21 – 7.16 (m, 2H), 6.26 – 6.17 (m, 1H), 5.87 (d,  $J$  = 3.2 Hz, 1H), 3.74 (q,  $J$  = 6.8 Hz, 1H), 2.92 – 2.77 (m, 2H), 2.72 – 2.65 (m, 2H), 1.39 (d,  $J$  = 6.8 Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  209.4, 154.6, 141.0, 140.4, 129.0, 127.9, 127.2, 110.1, 105.1, 53.1, 39.2, 22.3, 17.4. HRMS (EI) calculated for  $\text{C}_{15}\text{H}_{16}\text{O}_2$   $[\text{M}]^+$  228.1145, measured: 228.1143. IR (neat) 2930, 1712, 1598, 1493, 1359, 1147, 1009, 729, 699, 598, 484  $\text{cm}^{-1}$ .

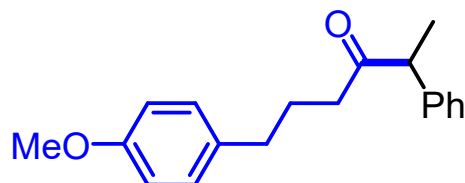
### 4-phenyl-1-(thiophen-2-yl)pentan-3-one (3j)



The title product was prepared according to the general procedure with 3-(thiophen-2-yl)propanoic acid (**1j**) (46.9 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3j** (40.0 mg, 54%) as a pale yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.34 (t,  $J$  = 7.2 Hz, 2H), 7.32 – 7.25 (m, 1H), 7.21 (d,  $J$  = 6.8 Hz, 2H), 7.09 (d,  $J$  = 5.2 Hz, 1H), 6.93 – 6.84 (m, 1H), 6.71 (d,  $J$  = 4.0 Hz, 1H), 3.76 (q,  $J$  = 6.8 Hz, 1H), 3.16 – 2.97

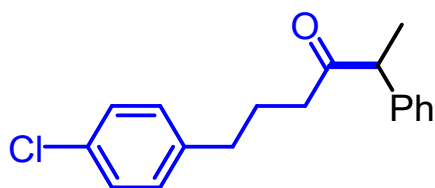
(m, 2H), 2.80 – 2.72 (m, 2H), 1.42 (d,  $J = 6.8$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  209.3, 143.7, 140.4, 129.0, 127.9, 127.2, 126.8, 124.5, 123.3, 53.2, 42.7, 24.1, 17.4. HRMS (EI) calculated for  $\text{C}_{15}\text{H}_{16}\text{OS}$   $[\text{M}]^+$  244.0916, measured: 244.0916. IR (neat) 2929, 1711, 1493, 1451, 1121, 1030, 847, 824, 694, 544  $\text{cm}^{-1}$ .

### 6-(4-methoxyphenyl)-2-phenylhexan-3-one (3k)



The title product was prepared according to the general procedure with 4-(4-methoxyphenyl)butanoic acid (**1k**) (58.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3k** (70.3 mg, 83%) as a pale yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.36 (t,  $J = 7.2$  Hz, 2H), 7.32 – 7.26 (m, 1H), 7.26 – 7.21 (m, 2H), 6.98 (d,  $J = 8.4$  Hz, 2H), 6.80 (d,  $J = 8.4$  Hz, 2H), 3.80 (s, 3H), 3.75 (q,  $J = 6.8$  Hz, 1H), 2.54 – 2.35 (m, 4H), 1.90 – 1.73 (m, 2H), 1.41 (d,  $J = 6.8$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  210.7, 157.8, 140.7, 133.8, 129.3, 128.9, 127.9, 127.1, 113.7, 55.3, 53.0, 40.2, 34.0, 25.6, 17.5. HRMS (ESI) calculated for  $\text{C}_{19}\text{H}_{22}\text{O}_2\text{Na}$   $[\text{M}+\text{Na}]^+$  305.1512, measured: 305.1511. IR (neat) 2931, 1710, 1510, 1452, 1243, 1176, 1034, 810, 700, 544  $\text{cm}^{-1}$ .

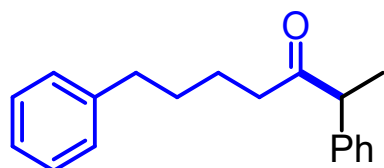
### 6-(4-chlorophenyl)-2-phenylhexan-3-one (3l)



The title product was prepared according to the general procedure with 4-(4-methoxyphenyl)butanoic acid (**1l**) (59.6 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3l** (61.1 mg, 71%) as a pale yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.36 (t,  $J = 7.2$  Hz, 2H), 7.33 – 7.28 (m, 1H), 7.25 – 7.18 (m, 4H), 6.97 (d,  $J = 8.4$  Hz, 2H), 3.75 (q,  $J = 6.8$  Hz, 1H), 2.55 – 2.31 (m, 4H), 1.86 – 1.77 (m, 2H), 1.41 (d,  $J = 6.8$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  210.4, 140.6, 140.1, 131.5, 129.8, 129.0, 128.4, 127.9, 127.2, 53.1, 39.9, 34.2, 25.1, 17.4. HRMS (EI) calculated for  $\text{C}_{18}\text{H}_{19}\text{ClO}$   $[\text{M}]^+$  286.1119, measured: 286.1119. IR (neat) 2929, 1711,

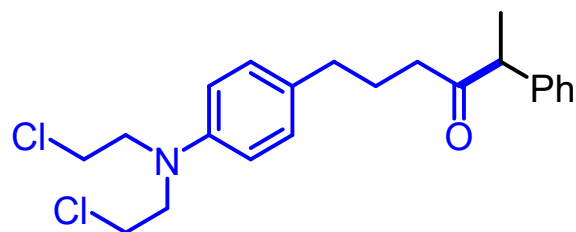
1491, 1451, 1089, 1013, 799, 759, 699, 522  $\text{cm}^{-1}$ .

### 2,7-diphenylheptan-3-one (3m)



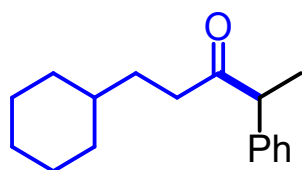
The title product was prepared according to the general procedure with 5-phenylpentanoic acid (**1m**) (53.5 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3m** (58.3 mg, 73%) as a yellow oil. **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.36 (t,  $J = 7.2$  Hz, 2H), 7.31 – 7.27 (m, 3H), 7.26 – 7.17 (m, 3H), 7.14 (d,  $J = 7.2$  Hz, 2H), 3.77 (q,  $J = 6.8$  Hz, 1H), 2.55 (t,  $J = 7.2$  Hz, 2H), 2.43 – 2.38 (m, 2H), 1.62 – 1.47 (m, 4H), 1.42 (d,  $J = 6.8$  Hz, 3H). **<sup>13</sup>C NMR** (101 MHz,  $\text{CDCl}_3$ )  $\delta$  210.8, 142.3, 140.7, 128.9, 128.4, 128.3, 127.9, 127.2, 125.7, 53.0, 40.8, 35.7, 30.8, 23.5, 17.5. **HRMS** (EI) calculated for  $\text{C}_{19}\text{H}_{22}\text{O}$   $[\text{M}]^+$  266.1665, measured: 266.1669. **IR** (neat) 2930, 1711, 1600, 1493, 1451, 1028, 745, 697, 701, 545  $\text{cm}^{-1}$ .

### 6-(4-(bis(2-chloroethyl)amino)phenyl)-2-phenylhexan-3-one (3n)



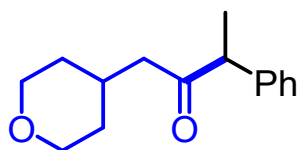
The title product was prepared according to the general procedure with chlorambucil (**1n**) (91.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3n** (70.6 mg, 60%) as a yellow oil. **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.37 (t,  $J = 7.2$  Hz, 2H), 7.34 – 7.28 (m, 1H), 7.27 – 7.22 (m, 2H), 6.98 (d,  $J = 8.4$  Hz, 2H), 6.61 (d,  $J = 8.8$  Hz, 2H), 3.82 – 3.75 (m, 1H), 3.72 (t,  $J = 6.4$  Hz, 4H), 3.64 (t,  $J = 6.4$  Hz, 4H), 2.52 – 2.34 (m, 4H), 1.86 – 1.76 (m, 2H), 1.42 (d,  $J = 6.8$  Hz, 3H). **<sup>13</sup>C NMR** (101 MHz,  $\text{CDCl}_3$ )  $\delta$  210.7, 144.1, 140.7, 131.0, 129.7, 129.0, 128.0, 127.2, 112.2, 53.7, 53.0, 40.5, 40.3, 33.8, 25.6, 17.5. **HRMS** (EI) calculated for  $\text{C}_{22}\text{H}_{27}\text{Cl}_2\text{NO}$   $[\text{M}]^+$  391.1464, measured: 391.1467. **IR** (neat) 2930, 1710, 1614, 1516, 1353, 1179, 802, 741, 701, 545  $\text{cm}^{-1}$ .

### 1-cyclohexyl-4-phenylpentan-3-one (**3o**)



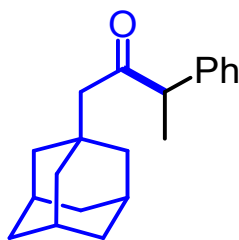
The title product was prepared according to the general procedure with 3-cyclohexylpropanoic acid (**1o**) (46.9 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3o** (36.7 mg, 50%) as a colorless oil. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.32 (t, *J* = 7.2 Hz, 2H), 7.28 – 7.23 (m, 1H), 7.23 – 7.19 (m, 2H), 3.75 (q, *J* = 7.2 Hz, 1H), 2.46 – 2.25 (m, 2H), 1.68 – 1.49 (m, 5H), 1.38 (d, *J* = 7.2 Hz, 3H), 1.41 – 1.34 (m, 2H), 1.19 – 0.99 (m, 4H), 0.84 – 0.68 (m, 2H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 211.3, 140.8, 128.9, 127.9, 127.1, 53.0, 38.6, 37.1, 33.2, 32.9, 31.3, 26.5, 26.2, 26.2, 17.5. **HRMS** (EI) calculated for C<sub>17</sub>H<sub>24</sub>O [M]<sup>+</sup> 244.1822, measured: 244.1823. **IR** (neat) 2920, 2849, 1712, 1493, 1449, 1070, 757, 699, 542 cm<sup>-1</sup>.

### 3-phenyl-1-(tetrahydro-2H-pyran-4-yl)butan-2-one (**3p**)



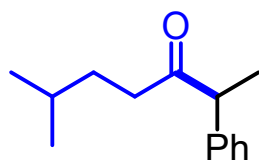
The title product was prepared according to the general procedure with 2-(tetrahydro-2H-pyran-4-yl)acetic acid (**1p**) (43.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3p** (48.7 mg, 70%) as a white solid. m.p. 51-53 °C. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.38 – 7.32 (m, 2H), 7.30 – 7.27 (m, 1H), 7.21 (d, *J* = 6.8 Hz, 2H), 3.92 – 3.79 (m, 2H), 3.73 (q, *J* = 6.8 Hz, 1H), 3.42 – 3.30 (m, 2H), 2.30 (d, *J* = 6.4 Hz, 2H), 2.10 – 1.98 (m, 1H), 1.56 – 1.50 (m, 1H), 1.44 – 1.42 (m, 1H), 1.40 (d, *J* = 6.8 Hz, 3H), 1.26 – 1.12 (m, 1H), 1.11 – 0.97 (m, 1H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 209.7, 140.3, 129.0, 127.9, 127.3, 67.8, 53.6, 47.8, 32.8, 32.4, 30.7, 17.2. **HRMS** (ESI) calculated for C<sub>15</sub>H<sub>21</sub>O<sub>2</sub> [M+H]<sup>+</sup> 233.1536, measured: 233.1536. **IR** (neat) 2927, 2839, 1710, 1493, 1451, 1092, 1013, 852, 760, 700, 545 cm<sup>-1</sup>.

### 1-((3*r*,5*r*,7*r*)-adamantan-1-yl)-3-phenylbutan-2-one (**3q**)



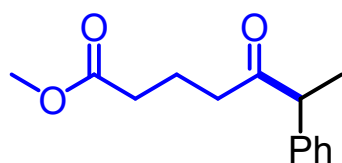
The title product was prepared according to the general procedure with 2-((3*r*,5*r*,7*r*)-adamantan-1-yl)acetic acid (**1q**) (58.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3q** (53.0 mg, 63%) as a colorless oil. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.32 (t, *J* = 7.2 Hz, 2H), 7.27 – 7.22 (m, 1H), 7.21 – 7.16 (m, 2H), 3.70 (q, *J* = 6.8 Hz, 1H), 2.22 (d, *J* = 14.4 Hz, 1H), 1.98 (d, *J* = 14.4 Hz, 1H), 1.92 (brs, 3H), 1.73 – 1.51 (m, 12H), 1.34 (d, *J* = 6.8 Hz, 3H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 210.2, 140.6, 128.9, 128.1, 127.0, 55.0, 54.5, 42.4, 36.8, 33.6, 28.6, 17.4. **HRMS** (ESI) calculated for C<sub>22</sub>H<sub>26</sub>ONa [M+Na]<sup>+</sup> 305.1876, measured: 305.1871. **IR** (neat) 2898, 2846, 1707, 1450, 1029, 755, 698, 537 cm<sup>-1</sup>.

### 6-methyl-2-phenylheptan-3-one (**3r**)



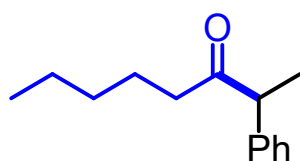
The title product was prepared according to the general procedure with 4-methylpentanoic acid (**1r**) (34.9 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3r** (42.9 mg, 70%) as a colorless oil. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.25 (t, *J* = 7.2 Hz, 2H), 7.20 – 7.16 (m, 1H), 7.15 – 7.11 (m, 2H), 3.69 (q, *J* = 6.8 Hz, 1H), 2.27 (t, *J* = 8.0 Hz, 2H), 1.40 – 1.23 (m, 6H), 0.71 (d, *J* = 6.0 Hz, 3H), 0.68 (d, *J* = 6.0 Hz, 3H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 211.2, 140.8, 128.9, 127.9, 127.1, 53.0, 39.1, 32.8, 27.5, 22.4, 22.1, 17.5. **HRMS** (EI) calculated for C<sub>14</sub>H<sub>20</sub>O [M]<sup>+</sup> 204.1509, measured: 204.1508. **IR** (neat) 2956, 2870, 1712, 1453, 1136, 757, 700, 548 cm<sup>-1</sup>.

### methyl 5-oxo-6-phenylheptanoate (**3s**)



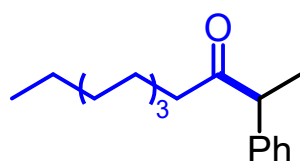
The title product was prepared according to the general procedure with 5-methoxy-5-oxopentanoic acid (**1s**) (43.8 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3s** (45.6 mg, 65%) as a pale yellow oil. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.32 (t, *J* = 7.2 Hz, 2H), 7.28 – 7.23 (m, 1H), 7.20 (d, *J* = 7.2 Hz, 2H), 3.75 (q, *J* = 6.8 Hz, 1H), 3.60 (s, 3H), 2.42 (t, *J* = 7.2 Hz, 2H), 2.29 – 2.12 (m, 2H), 1.90 – 1.74 (m, 2H), 1.39 (d, *J* = 6.8 Hz, 3H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 210.0, 173.6, 140.5, 129.0, 127.8, 127.2, 53.0, 51.5, 39.7, 32.9, 18.9, 17.4. **HRMS** (ESI) calculated for C<sub>14</sub>H<sub>18</sub>O<sub>3</sub>Na [M+Na]<sup>+</sup> 257.1148, measured: 257.1150. **IR** (neat) 2951, 1733, 1711, 1493, 1451, 1372, 1171, 761, 700, 545 cm<sup>-1</sup>.

### 2-phenyloctan-3-one (**3t**)<sup>[1]</sup>



The title product was prepared according to the general procedure with hexanoic acid (**1t**) (34.9 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3t** (33.0 mg, 54%) as a pale yellow oil. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.35 (t, *J* = 7.6 Hz, 2H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.23 (d, *J* = 7.2 Hz, 2H), 3.77 (q, *J* = 6.8 Hz, 1H), 2.36 (t, *J* = 7.2 Hz, 2H), 1.59 – 1.45 (m, 2H), 1.41 (d, *J* = 6.8 Hz, 3H), 1.29 – 1.12 (m, 4H), 0.85 (t, *J* = 7.2 Hz, 3H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 211.1, 140.8, 128.9, 127.9, 127.1, 53.0, 41.0, 31.3, 23.6, 22.4, 17.5, 13.9.

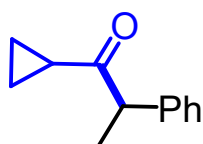
### 2-phenyloctan-3-one (**3u**)



The title product was prepared according to the general procedure with decanoic acid

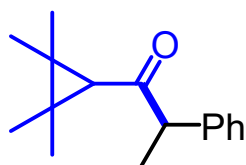
(**1u**) (51.7 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3u** (41.5 mg, 53%) as a yellow oil. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.25 (t, *J* = 7.2 Hz, 2H), 7.20 – 7.16 (m, 1H), 7.13 (d, *J* = 7.2 Hz, 2H), 3.67 (q, *J* = 7.2 Hz, 1H), 2.26 (t, *J* = 8.0 Hz, 2H), 1.47 – 1.35 (m, 2H), 1.31 (d, *J* = 7.2 Hz, 3H), 1.22 – 1.07 (m, 12H), 0.79 (t, *J* = 6.8 Hz, 3H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 211.1, 140.8, 128.9, 127.9, 127.1, 53.0, 41.1, 31.9, 29.4, 29.3, 29.2, 29.1, 23.9, 22.7, 17.5, 14.1. **HRMS** (EI) calculated for C<sub>18</sub>H<sub>28</sub>O [M]<sup>+</sup> 260.2135, measured: 260.2134. **IR** (neat) 2923, 2853, 1713, 1493, 1453, 1373, 759, 699, 546 cm<sup>-1</sup>.

### 1-cyclopropyl-2-phenylpropan-1-one (**3v**)



The title product was prepared according to the general procedure with cyclopropanecarboxylic acid (**1v**) (25.8 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3v** (26.0 mg, 50%) as a pale yellow oil. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.34 (t, *J* = 7.2 Hz, 2H), 7.30 – 7.21 (m, 3H), 3.90 (q, *J* = 7.2 Hz, 1H), 1.90 – 1.81 (m, 1H), 1.41 (d, *J* = 7.2 Hz, 3H), 1.09 – 0.90 (m, 2H), 0.83 – 0.74 (m, 1H), 0.73 – 0.66 (m, 1H). **<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 210.9, 140.9, 128.9, 128.1, 127.0, 53.8, 19.7, 17.6, 11.4, 11.3. **HRMS** (EI) calculated for C<sub>12</sub>H<sub>14</sub>O [M]<sup>+</sup> 174.1039, measured: 174.1039. **IR** (neat) 2975, 2931, 1689, 1452, 1378, 1041, 1016, 796, 699 cm<sup>-1</sup>.

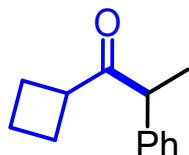
### 2-phenyl-1-(2,2,3,3-tetramethylcyclopropyl)propan-1-one (**3w**)



The title product was prepared according to the general procedure with chrysanthemum acid (**1w**) (42.7 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3w** (47.7 mg, 69%) as a colorless oil. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.24 (t, *J* = 7.2 Hz,

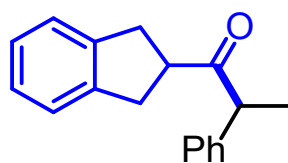
2H), 7.18 – 7.12 (m, 1H), 7.11 – 7.06 (m, 2H), 3.70 (q,  $J = 6.8$  Hz, 1H), 1.30 – 1.26 (m, 4H), 1.17 (s, 3H), 1.00 (s, 3H), 0.90 (s, 3H), 0.74 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  207.7, 140.1, 127.6, 126.9, 125.7, 54.3, 43.1, 33.4, 32.9, 22.8, 21.9, 15.9, 15.4, 15.1. HRMS (ESI) calculated for  $\text{C}_{16}\text{H}_{23}\text{O}$   $[\text{M}]^+$  231.1743, measured: 231.1740. IR (neat) 2925, 1683, 1492, 1451, 1378, 1106, 1010, 698, 524  $\text{cm}^{-1}$ .

### 1-cyclobutyl-2-phenylpropan-1-one (3x)



The title product was prepared according to the general procedure with cyclobutanecarboxylic acid (**1x**) (30.0 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3x** (41.8mg, 74%) as a yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.33 (t,  $J = 7.2$  Hz, 2H), 7.29 – 7.25 (m, 1H), 7.24 – 7.18 (m, 2H), 3.76 (q,  $J = 7.2$  Hz, 1H), 3.30 (m, 1H), 2.22 – 2.13 (m, 2H), 2.09 (m, 1H), 1.94 – 1.71 (m, 3H), 1.40 (d,  $J = 7.2$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  211.8, 140.7, 128.8, 128.0, 127.0, 50.8, 44.3, 25.4, 24.4, 17.7, 17.6. HRMS (EI) calculated for  $\text{C}_{13}\text{H}_{16}\text{O}$   $[\text{M}]^+$  188.1196, measured: 188.1196. IR (neat) 2928, 1706, 1453, 1130, 968, 699, 504  $\text{cm}^{-1}$ .

### 1-(2,3-dihydro-1H-inden-2-yl)-2-phenylpropan-1-one (3y)

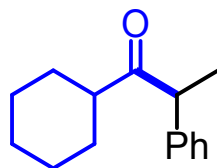


The title product was prepared according to the general procedure with 2,3-dihydro-1H-indene-2-carboxylic acid (**1y**) (48.7 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3y** (41.3 mg, 55%) as a pale yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35 (t,  $J = 7.2$  Hz, 2H), 7.30 – 7.25 (m, 3H), 7.17 – 7.09 (m, 4H), 3.95 (q,  $J = 6.8$  Hz, 1H), 3.50 (p,  $J = 8.8$  Hz, 1H), 3.21 – 3.09 (m, 3H), 2.79 – 2.67 (m, 1H), 1.45 (d,  $J = 6.8$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  211.8, 142.0, 141.1, 140.4, 129.0, 128.0, 127.2, 126.6, 126.4, 124.3, 124.2, 52.3, 49.8, 36.5, 35.5, 17.9. HRMS (EI) calculated for  $\text{C}_{16}\text{H}_{18}\text{O}$   $[\text{M}]^+$  250.1352, measured: 250.1352. IR (neat)



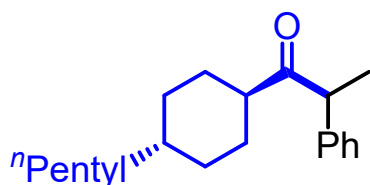
2929, 1707, 1485, 1451, 743, 699, 509 cm<sup>-1</sup>.

**1-cyclohexyl-2-phenylpropan-1-one (3z)** [7]



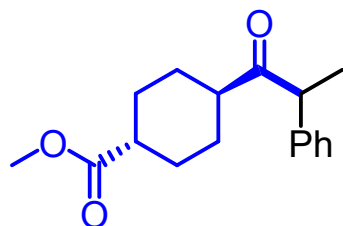
The title product was prepared according to the general procedure with cyclohexanecarboxylic acid (**1z**) (38.5 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3z** (38.9 mg, 60%) as a pale yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.31 (t, *J* = 7.2 Hz, 2H), 7.28 – 7.23 (m, 1H), 7.23 – 7.19 (m, 2H), 3.90 (q, *J* = 6.8 Hz, 1H), 2.45 – 2.35 (m, 1H), 1.87 – 1.81 (m, 1H), 1.77 – 1.72 (m, 1H), 1.69 – 1.58 (m, 2H), 1.52 – 1.41 (m, 1H), 1.36 (d, *J* = 6.8 Hz, 3H), 1.31 – 1.03 (m, 5H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 212.8, 139.7, 127.8, 127.0, 126.0, 50.1, 48.5, 28.4, 27.3, 24.9, 24.7, 24.3, 17.2.

**1-((1s,4r)-4-pentylcyclohexyl)-2-phenylpropan-1-one (3aa)**



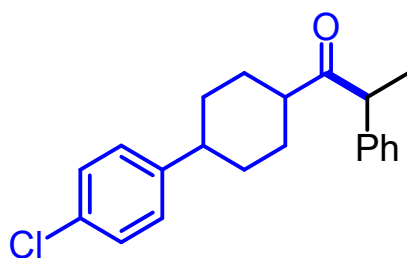
The title product was prepared according to the general procedure with trans-4-pentylcyclohexane-1-carboxylic acid (**1aa**) (59.5 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3aa** (36.0 mg, 42%) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.37 – 7.20 (m, 5H), 3.92 (q, *J* = 6.8 Hz, 1H), 2.45 – 2.30 (m, 1H), 1.95 – 1.85 (m, 1H), 1.83 – 1.76 (m, 1H), 1.73 – 1.64 (m, 1H), 1.55 – 1.41 (m, 2H), 1.38 (d, *J* = 6.8 Hz, 3H), 1.34 – 1.21 (m, 7H), 1.19 – 1.12 (m, 3H), 0.95 – 0.83 (m, 4H), 0.78 – 0.68 (m, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 214.1, 140.7, 128.8, 128.0, 127.0, 51.3, 49.7, 37.2, 36.9, 32.7, 32.2, 32.1, 29.5, 28.4, 26.5, 22.7, 18.2, 14.1. HRMS (ESI) calculated for C<sub>20</sub>H<sub>30</sub>ONa [M+Na]<sup>+</sup> 309.2189, measured: 309.2183. IR (neat) 2922, 2852, 1706, 1449, 1374, 951, 729, 698, 555 cm<sup>-1</sup>.

**methyl (1r,4r)-4-(2-phenylpropanoyl)cyclohexane-1-carboxylate (3ab)**



The title product was prepared according to the general procedure with trans-4-(methoxycarbonyl)cyclohexane-1-carboxylic acid (**1ab**) (55.9 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3ab** (57.6 mg, 70%) as a yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.34 (t, *J* = 7.2 Hz, 2H), 7.25 – 7.23 (m, 1H), 7.25 – 7.19 (m, 2H), 3.91 (q, *J* = 6.8 Hz, 1H), 3.65 (s, 3H), 2.50 – 2.36 (m, 1H), 2.34 – 2.20 (m, 1H), 2.05 – 2.01 (m, 1H), 1.99 – 1.91 (m, 2H), 1.58 – 1.49 (m, 1H), 1.44 – 1.24 (m, 7H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 213.2, 176.0, 140.4, 128.9, 128.0, 127.2, 51.6, 51.5, 48.4, 42.4, 28.5, 28.3, 27.8, 27.4, 18.0. HRMS (ESI) calculated for C<sub>17</sub>H<sub>22</sub>O<sub>3</sub>Na [M+Na]<sup>+</sup> 297.1461, measured: 297.1462. IR (neat) 2933, 2861, 1731, 1704, 1451, 1247, 1017, 896, 730, 699, 555 cm<sup>-1</sup>.

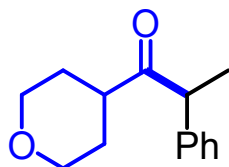
**1-(4-(4-chlorophenyl)cyclohexyl)-2-phenylpropan-1-one (3ac)**



The title product was prepared according to the general procedure with 4-(4-chlorophenyl)cyclohexane-1-carboxylic acid (**1ac**) (71.6 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3ac** (78.4 mg, 80%) as a pale yellow solid. m.p. 59-61 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.37 (t, *J* = 7.2 Hz, 2H), 7.31 – 7.23 (m, 5H), 7.10 (d, *J* = 8.4 Hz, 2H), 3.96 (q, *J* = 6.8 Hz, 1H), 2.57 – 2.41 (m, 2H), 2.08 – 1.99 (m, 1H), 1.98 – 1.91 (m, 1H), 1.89 – 1.79 (m, 1H), 1.70 – 1.55 (m, 3H), 1.51 – 1.39 (m, 5H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 213.5, 145.4, 140.5, 131.6, 128.9, 128.4, 128.1, 128.0, 127.2, 51.5, 48.8, 43.0, 33.6, 33.0, 29.7, 28.6, 18.1. HRMS (EI)

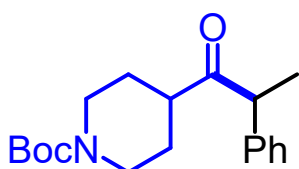
calculated for  $C_{21}H_{23}ClO$   $[M]^+$  326.1432, measured: 326.1432. **IR** (neat) 2927, 2854, 1704, 1492, 1449, 1091, 972, 822, 699, 530  $cm^{-1}$ .

### 2-phenyl-1-(tetrahydro-2H-pyran-4-yl)propan-1-one (3ad)



The title product was prepared according to the general procedure with tetrahydro-2H-pyran-4-carboxylic acid (**1ad**) (39.0 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3ad** (37.9 mg, 58%) as a pale yellow oil.  **$^1H$  NMR** (400 MHz,  $CDCl_3$ )  $\delta$  7.53 – 7.11 (m, 5H), 4.00 – 3.85 (m, 3H), 3.42 – 3.31 (m, 1H), 3.31 – 3.20 (m, 1H), 2.70 – 2.57 (m, 1H), 1.79 – 1.58 (m, 3H), 1.39 (d,  $J = 6.8$  Hz, 3H), 1.37 – 1.29 (m, 1H).  **$^{13}C$  NMR** (101 MHz,  $CDCl_3$ )  $\delta$  211.8, 140.3, 129.0, 127.9, 127.2, 67.3, 67.0, 51.0, 46.1, 29.0, 28.1, 18.2. **HRMS** (ESI) calculated for  $C_{14}H_{19}O_2$   $[M+H]^+$  219.1380, measured: 219.1380. **IR** (neat) 2950, 2844, 1705, 1449, 1115, 1088, 1016, 732, 699, 559, 506  $cm^{-1}$ .

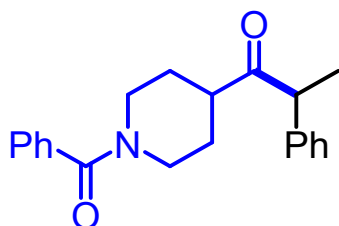
### tert-butyl 4-(2-phenylpropanoyl)piperidine-1-carboxylate (3ae)



The title product was prepared according to the general procedure with 1-(tert-butoxycarbonyl)piperidine-4-carboxylic acid (**1ae**) (68.8 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3ae** (57.2 mg, 60%) as a yellow solid. m.p. 77-79 °C.  **$^1H$  NMR** (400 MHz,  $CDCl_3$ )  $\delta$  7.35 – 7.30 (m, 2H), 7.29 – 7.25 (m, 1H), 7.23 – 7.17 (m, 2H), 4.20 – 3.96 (m, 2H), 3.90 (q,  $J = 6.8$  Hz, 1H), 2.68 (t,  $J = 11.8$  Hz, 1H), 2.59 – 2.47 (m, 2H), 1.84 – 1.71 (m, 1H), 1.59 – 1.46 (m, 3H), 1.43 (s, 9H), 1.37 (d,  $J = 6.8$  Hz, 3H).  **$^{13}C$  NMR** (101 MHz,  $CDCl_3$ )  $\delta$  212.2, 154.7, 140.3, 129.1, 128.0, 127.3, 79.6, 51.3, 47.2, 43.2 (br,  $NCH_2$ ), 28.5, 27.6, 18.2. **HRMS** (ESI) calculated for  $C_{19}H_{27}NO_3Na$   $[M+Na]^+$  340.1883, measured: 340.1881. **IR** (neat) 2930, 1688, 1450,

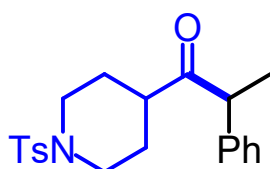
1421, 1233, 1168, 1012, 972, 701, 555  $\text{cm}^{-1}$ .

### 1-(1-benzoylpiperidin-4-yl)-2-phenylpropan-1-one (**3af**)



The title product was prepared according to the general procedure with 1-benzoylpiperidine-4-carboxylic acid (**1af**) (70.0 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3af** (61.7 mg, 64%) as a yellow oil.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.34 – 7.23 (m, 7H), 7.21 – 7.16 (m, 1H), 7.15 – 7.11 (m, 2H), 4.51 (br, 1H), 3.84 – 3.81 (m, 1H), 3.59 (br, 1H), 2.76 – 2.55 (br m, 3H), 1.86 – 1.74 (m, 1H), 1.52 (br, 3H), 1.31 (d,  $J = 6.8$  Hz, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  210.6, 169.3, 139.1, 135.0, 128.6, 128.0, 127.4, 126.9, 126.3, 125.8, 50.4, 45.9, 40.6 (br m), 27.3 (br m), 17.1. **HRMS** (ESI) calculated for  $\text{C}_{21}\text{H}_{24}\text{NO}_2$   $[\text{M}+\text{H}]^+$  322.1802, measured: 322.1796. **IR** (neat) 2947, 2859, 1704, 1626, 1430, 1278, 972, 789, 700, 556  $\text{cm}^{-1}$ .

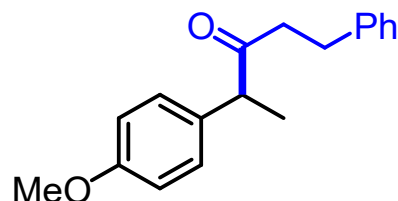
### 2-phenyl-1-(1-tosylpiperidin-4-yl)propan-1-one (**3ag**)



The title product was prepared according to the general procedure with 1-tosylpiperidine-4-carboxylic acid (**1ag**) (85.0 mg, 0.3 mmol) and (2-bromoethyl)benzene (**2a**) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **3ag** (71.3 mg, 64%) as a white solid. m.p. 151-153  $^{\circ}\text{C}$ .  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60 (d,  $J = 8.4$  Hz, 2H), 7.36 – 7.21 (m, 5H), 7.14 (d,  $J = 6.8$  Hz, 2H), 3.84 (q,  $J = 6.8$  Hz, 1H), 3.75 – 3.68 (m, 1H), 3.66 – 3.59 (m, 1H), 2.43 (s, 3H), 2.36 – 2.24 (m, 2H), 2.22 – 2.13 (m, 1H), 1.93 – 1.85 (m, 1H), 1.78 – 1.61 (m, 2H), 1.51 – 1.45 (m, 1H), 1.34 (d,  $J = 6.8$  Hz, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  211.4, 143.6, 140.1, 133.0, 129.7, 129.1, 127.9, 127.7, 127.3, 51.3, 45.8, 45.7, 45.3, 27.9, 27.0, 21.6, 18.1. **HRMS** (DART) calculated for  $\text{C}_{21}\text{H}_{26}\text{NSO}_2$

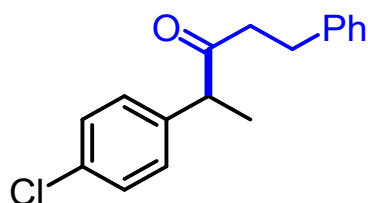
$[M]^+$  372.1628, measured: 372.1625. **IR** (neat) 2844, 1704, 1594, 1490, 1159, 932, 798, 719, 545  $\text{cm}^{-1}$ .

#### 4-(4-methoxyphenyl)-1-phenylpentan-3-one (**4a**) <sup>[7]</sup>



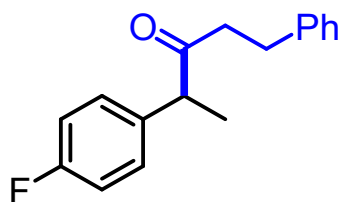
The title product was prepared according to the general procedure with 3-phenylpropionic acid (**1a**) (45.0 mg, 0.3 mmol) and 1-(2-bromoethyl)-4-methoxybenzene (**2b**) (64.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4a** (33.8 mg, 42%) as a pale yellow oil. **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.29 – 7.23 (m, 2H), 7.22 – 7.16 (m, 1H), 7.13 – 7.07 (m, 4H), 6.86 (d,  $J = 8.4$  Hz, 2H), 3.82 (s, 3H), 3.68 (q,  $J = 7.2$  Hz, 1H), 2.94 – 2.61 (m, 4H), 1.38 (d,  $J = 7.2$  Hz, 3H). **<sup>13</sup>C NMR** (101 MHz,  $\text{CDCl}_3$ )  $\delta$  210.2, 158.7, 141.1, 132.5, 128.9, 128.4, 128.3, 126.0, 114.3, 55.3, 52.3, 42.5, 30.0, 17.4.

#### 4-(4-chlorophenyl)-1-phenylpentan-3-one (**4b**)



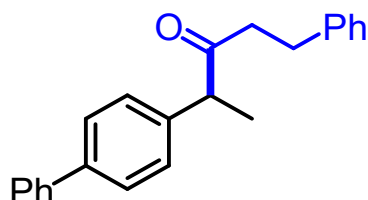
The title product was prepared according to the general procedure with 3-phenylpropionic acid (**1a**) (45.0 mg, 0.3 mmol) and 1-(2-bromoethyl)-4-chlorobenzene (**2c**) (65.9 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4b** (49.8 mg, 61%) as a colorless oil. **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.33 – 7.17 (m, 5H), 7.13 – 7.07 (m, 4H), 3.71 (q,  $J = 7.2$  Hz, 1H), 2.93 – 2.78 (m, 2H), 2.73 – 2.64 (m, 2H), 1.39 (d,  $J = 7.2$  Hz, 3H). **<sup>13</sup>C NMR** (101 MHz,  $\text{CDCl}_3$ )  $\delta$  209.4, 140.9, 138.8, 133.1, 129.2, 129.1, 128.5, 128.3, 126.1, 52.5, 42.7, 29.9, 17.4. **HRMS** (EI) calculated for  $\text{C}_{17}\text{H}_{17}\text{ClO}$   $[M]^+$  272.0962, measured: 272.0963. **IR** (neat) 2930, 1712, 1490, 1453, 1091, 1013, 830, 748, 698, 505  $\text{cm}^{-1}$ .

#### 4-(4-fluorophenyl)-1-phenylpentan-3-one (**4c**)<sup>[7]</sup>



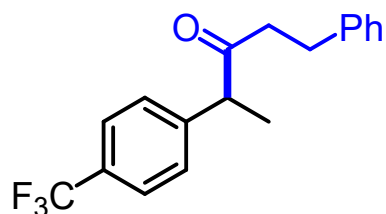
The title product was prepared according to the general procedure with 3-phenylpropionic acid (**1a**) (45.0 mg, 0.3 mmol) and 1-(2-bromoethyl)-4-fluorobenzene (**2d**) (60.9 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4c** (43.8 mg, 57%) as a pale yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.19 – 7.12 (m, 2H), 7.10 – 7.06 (m, 1H), 7.05 – 6.96 (m, 4H), 6.93 – 6.86 (m, 2H), 3.61 (q, *J* = 7.2 Hz, 1H), 2.81 – 2.66 (m, 2H), 2.63 – 2.55 (m, 2H), 1.28 (d, *J* = 7.2 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 209.6, 162.0 (d, *J* = 245.7 Hz), 140.9, 136.1 (d, *J* = 3.3 Hz), 129.4 (d, *J* = 8.0 Hz), 128.4, 128.3, 126.1, 115.8 (d, *J* = 21.4 Hz), 52.3, 42.6, 29.9, 17.5. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -115.52.

#### 4-([1,1'-biphenyl]-4-yl)-1-phenylpentan-3-one (**4d**)<sup>[7]</sup>



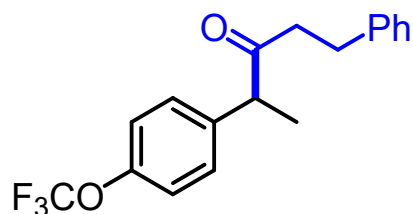
The title product was prepared according to the general procedure with 3-phenylpropionic acid (**1a**) (45.0 mg, 0.3 mmol) and 4-(2-bromoethyl)-1,1'-biphenyl (**2e**) (78.4 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4d** (58.4 mg, 62%) as a white solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.62 (d, *J* = 7.2 Hz, 2H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.39 (t, *J* = 7.2 Hz, 1H), 7.31 – 7.23 (m, 4H), 7.23 – 7.17 (m, 1H), 7.12 (d, *J* = 7.2 Hz, 2H), 3.79 (q, *J* = 6.8 Hz, 1H), 2.99 – 2.62 (m, 4H), 1.46 (d, *J* = 6.8 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 209.9, 141.1, 140.7, 140.1, 139.4, 128.8, 128.4, 128.3, 128.3, 127.7, 127.4, 127.1, 126.1, 52.9, 42.7, 30.0, 17.4.

### 1-phenyl-4-(4-(trifluoromethyl)phenyl)pentan-3-one (**4e**)<sup>[7]</sup>



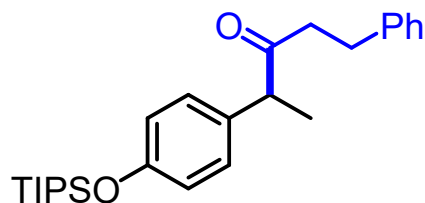
The title product was prepared according to the general procedure with 3-phenylpropionic acid (**1a**) (45.0 mg, 0.3 mmol) and 1-(2-bromoethyl)-4-(trifluoromethyl)benzene (**2f**) (75.9 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4e** (51.0 mg, 64%) as a pale yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.45 (d, *J* = 8.4 Hz, 2H), 7.20 – 7.05 (m, 5H), 6.97 (d, *J* = 7.2 Hz, 2H), 3.69 (q, *J* = 6.8 Hz, 1H), 2.83 – 2.68 (m, 2H), 2.60 (t, *J* = 7.6 Hz, 2H), 1.31 (d, *J* = 6.8 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 208.8, 144.2, 140.7, 129.5 (q, *J* = 32.5 Hz), 128.4, 128.3, 128.2, 126.1, 125.9 (q, *J* = 3.8 Hz), 124.1 (q, *J* = 272.0 Hz), 53.0, 42.8, 29.8, 17.4. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -62.52.

### 1-phenyl-4-(4-(trifluoromethoxy)phenyl)pentan-3-one (**4f**)



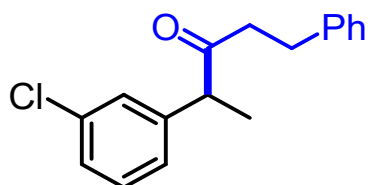
The title product was prepared according to the general procedure with 3-phenylpropionic acid (**1a**) (45.0 mg, 0.3 mmol) and 1-(2-bromoethyl)-4-(trifluoromethoxy)benzene (**2g**) (80.7 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4f** (54.7 mg, 56%) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.28 – 7.23 (m, 2H), 7.22 – 7.13 (m, 5H), 7.09 (d, *J* = 7.2 Hz, 2H), 3.75 (q, *J* = 6.8 Hz, 1H), 2.91 – 2.79 (m, 2H), 2.75 – 2.67 (m, 2H), 1.40 (d, *J* = 6.8 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 209.3, 148.3 (d, *J* = 1.8 Hz), 140.8, 139.0, 129.2, 128.4, 128.3, 126.1, 121.4, 120.5 (q, *J* = 257.1 Hz), 52.4, 42.7, 29.9, 17.5. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.86. HRMS (EI) calculated for C<sub>18</sub>H<sub>17</sub>F<sub>3</sub>O<sub>2</sub> [M]<sup>+</sup> 322.1175, measured: 322.1176. IR (neat) 2932, 1706, 1509, 1449, 1263, 1244, 1148, 1025, 811, 690, 552 cm<sup>-1</sup>.

#### 1-phenyl-4-(4-((triisopropylsilyloxy)phenyl)pentan-3-one (4g)



The title product was prepared according to the general procedure with 3-phenylpropionic acid (**1a**) (45.0 mg, 0.3 mmol) and 4-(2-bromoethyl)phenoxytriisopropylsilane (**2h**) (107.2 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4g** (67.8 mg, 55%) as a colorless oil.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.30 – 7.23 (m, 2H), 7.18 (t,  $J = 7.2$  Hz, 1H), 7.09 (d,  $J = 7.2$  Hz, 2H), 7.03 (d,  $J = 8.4$  Hz, 2H), 6.84 (d,  $J = 8.4$  Hz, 2H), 3.67 (q,  $J = 6.8$  Hz, 1H), 2.91 – 2.74 (m, 2H), 2.73 – 2.58 (m, 2H), 1.38 (d,  $J = 6.8$  Hz, 3H), 1.33 – 1.22 (m, 3H), 1.13 (d,  $J = 7.2$  Hz, 18H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  210.3, 155.2, 141.2, 132.9, 128.8, 128.4, 128.3, 126.0, 120.3, 52.4, 42.4, 30.1, 17.9, 17.2, 12.7. **HRMS** (EI) calculated for  $\text{C}_{26}\text{H}_{38}\text{O}_2\text{Si}$   $[\text{M}]^+$  410.2636, measured: 410.2635. **IR** (neat) 2943, 2866, 1713, 1605, 1508, 1262, 882, 837, 683, 553  $\text{cm}^{-1}$ .

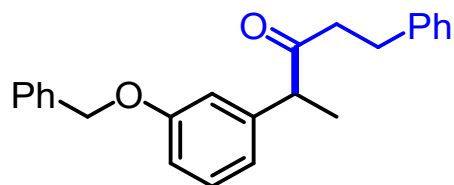
#### 4-(3-chlorophenyl)-1-phenylpentan-3-one (4h)



The title product was prepared according to the general procedure with 3-phenylpropionic acid (**1a**) (45.0 mg, 0.3 mmol) and 1-(2-bromoethyl)-3-chlorobenzene (**2i**) (65.9 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4h** (49.0 mg, 60%) as a pale yellow oil.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.29 – 7.23 (m, 4H), 7.22 – 7.17 (m, 2H), 7.11 (d,  $J = 6.8$  Hz, 2H), 7.07 – 7.01 (m, 1H), 3.70 (q,  $J = 6.8$  Hz, 1H), 2.94 – 2.79 (m, 2H), 2.74 – 2.67 (m, 2H), 1.39 (d,  $J = 6.8$  Hz, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  209.1, 142.3, 140.8, 134.7, 130.2, 128.5, 128.3, 128.1, 127.4, 126.1, 126.1, 52.8, 42.8, 29.9, 17.3. **HRMS** (EI) calculated for  $\text{C}_{17}\text{H}_{17}\text{ClO}$   $[\text{M}]^+$  272.0962, measured: 272.0964. **IR** (neat) 2929, 1713, 1593, 1453, 1080, 780, 748, 695, 553, 443  $\text{cm}^{-1}$ .

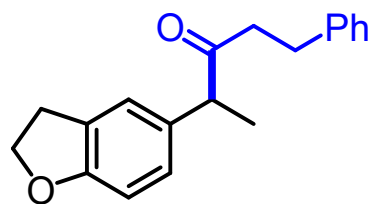


#### 4-(3-(benzyloxy)phenyl)-1-phenylpentan-3-one (4i)



The title product was prepared according to the general procedure with 3-phenylpropionic acid (**1a**) (45.0 mg, 0.3 mmol) and 1-(benzyloxy)-3-(2-bromoethyl)benzene (**2j**) (87.4 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4i** (51.7 mg, 50%) as a pale yellow oil.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.51 (d,  $J = 7.2$  Hz, 2H), 7.46 (t,  $J = 7.2$  Hz, 2H), 7.42 – 7.37 (m, 1H), 7.35 – 7.26 (m, 3H), 7.26 – 7.20 (m, 1H), 7.15 (d,  $J = 7.2$  Hz, 2H), 6.95 (d,  $J = 8.0$  Hz, 1H), 6.86 (d,  $J = 7.6$  Hz, 2H), 5.09 (s, 2H), 3.75 (q,  $J = 7.2$  Hz, 1H), 2.98 – 2.83 (m, 2H), 2.80 – 2.66 (m, 2H), 1.45 (d,  $J = 7.2$  Hz, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  209.7, 159.2, 142.1, 141.1, 136.9, 130.1, 128.7, 128.5, 128.4, 128.1, 127.7, 126.1, 120.7, 114.5, 113.5, 70.0, 53.2, 42.5, 30.0, 17.3. **HRMS** (EI) calculated for  $\text{C}_{24}\text{H}_{24}\text{O}_2$   $[\text{M}]^+$  344.1771, measured: 344.1772. **IR** (neat) 2930, 1710, 1581, 1451, 1258, 1156, 1025, 735, 695, 554, 456  $\text{cm}^{-1}$ .

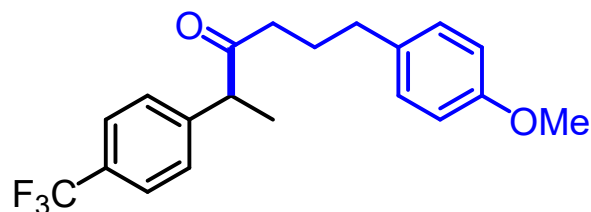
#### 4-(2,3-dihydrobenzofuran-5-yl)-1-phenylpentan-3-one (4j)



The title product was prepared according to the general procedure with 3-phenylpropionic acid (**1a**) (45.0 mg, 0.3 mmol) and 5-(2-bromoethyl)-2,3-dihydrobenzofuran (**2k**) (68.1 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4j** (38.0 mg, 39%) as a colorless oil.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.21 – 7.12 (m, 2H), 7.07 (t,  $J = 7.2$  Hz, 1H), 6.99 (d,  $J = 7.2$  Hz, 2H), 6.87 (s, 1H), 6.82 (d,  $J = 8.0$  Hz, 1H), 6.62 (d,  $J = 8.0$  Hz, 1H), 4.47 (t,  $J = 8.8$  Hz, 2H), 3.55 (q,  $J = 7.2$  Hz, 1H), 3.07 (t,  $J = 8.8$  Hz, 2H), 2.82 – 2.68 (m, 2H), 2.67 – 2.51 (m, 2H), 1.26 (d,  $J = 7.2$  Hz, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  210.3,

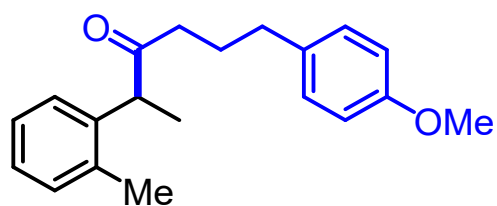
159.3, 141.1, 132.4, 128.4, 128.3, 127.7, 127.7, 126.0, 124.2, 109.5, 71.3.

**6-(4-methoxyphenyl)-2-(4-(trifluoromethyl)phenyl)hexan-3-one (4k)**



The title product was prepared according to the general procedure with 4-(4-methoxyphenyl)butanoic acid (**1k**) (58.3 mg, 0.3 mmol) and 1-(2-bromoethyl)-4-(trifluoromethyl)benzene (**2f**) (75.9 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4k** (51.0 mg, 60%) as a pale yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.61 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 6.97 (d, *J* = 8.8 Hz, 2H), 6.80 (d, *J* = 8.8 Hz, 2H), 3.86 – 3.81 (m, 1H), 3.80 (s, 3H), 2.57 – 2.42 (m, 2H), 2.39 (t, *J* = 7.2 Hz, 2H), 1.88 – 1.78 (m, 2H), 1.43 (d, *J* = 6.8 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 209.7, 157.9, 144.6, 133.5, 129.5 (q, *J* = 32.4 Hz), 129.3, 128.3, 125.8 (q, *J* = 3.7 Hz), 124.1 (q, *J* = 272.1 Hz), 113.8, 55.2, 52.7, 40.4, 33.9, 25.3, 17.5. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -62.47. HRMS (EI) calculated for C<sub>20</sub>H<sub>21</sub>F<sub>3</sub>O<sub>2</sub> [M]<sup>+</sup> 350.1488, measured: 350.1486. IR (neat) 2925, 1714, 1613, 1511, 1323, 1244, 1163, 1119, 1068, 840, 605 cm<sup>-1</sup>.

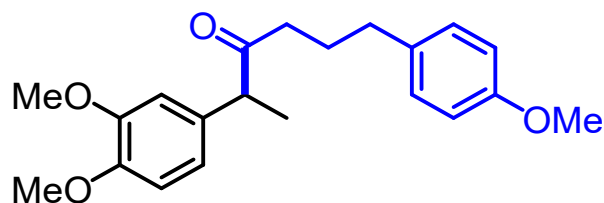
**6-(4-methoxyphenyl)-2-(o-tolyl)hexan-3-one (4l)**



The title product was prepared according to the general procedure with 4-(4-methoxyphenyl)butanoic acid (**1k**) (58.3 mg, 0.3 mmol) and 1-(2-bromoethyl)-2-methylbenzene (**2l**) (59.7 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4l** (43.0 mg, 48%) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.24 – 7.17 (m, 3H), 7.10 – 7.03 (m, 1H), 6.99 (d, *J* = 8.8 Hz, 2H), 6.80 (d, *J* = 8.8 Hz, 2H), 3.94 (q, *J* = 7.2 Hz, 1H), 3.80 (s, 3H), 2.61 – 2.46 (m, 1H), 2.45 – 2.40 (m, 1H), 2.38 (s, 3H), 2.35 – 2.27 (m, 2H), 1.87 – 1.74 (m, 2H), 1.36 (d, *J* = 7.2 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 211.1, 157.8, 139.2, 135.8,

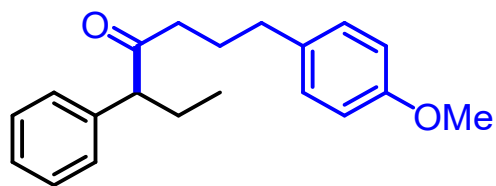
133.8, 130.9, 129.3, 126.99, 126.98, 126.7, 113.7, 55.3, 48.9, 40.1, 34.1, 25.7, 19.8, 16.9. **HRMS** (EI) calculated for C<sub>20</sub>H<sub>24</sub>O<sub>2</sub> [M]<sup>+</sup> 296.1771, measured: 296.1775. **IR** (neat) 2930, 1710, 1611, 1510, 1454, 1243, 1176, 1034, 811, 758, 728, 554, 455 cm<sup>-1</sup>.

#### 2-(3,4-dimethoxyphenyl)-6-(4-methoxyphenyl)hexan-3-one (4m)



The title product was prepared according to the general procedure with 4-(4-methoxyphenyl)butanoic acid (**1k**) (58.3 mg, 0.3 mmol) and 4-(2-bromoethyl)-1,2-dimethoxybenzene (**2m**) (73.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4m** (41.5 mg, 40%) as a white solid. m.p. 75-77 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.98 (d, *J* = 8.4 Hz, 2H), 6.86 – 6.75 (m, 4H), 6.70 (s, 1H), 3.89 (s, 3H), 3.87 (s, 3H), 3.78 (s, 3H), 3.68 (q, *J* = 7.2 Hz, 1H), 2.54 – 2.30 (m, 4H), 1.88 – 1.77 (m, 2H), 1.38 (d, *J* = 7.2 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 211.0, 157.8, 149.2, 148.2, 133.7, 133.1, 129.3, 120.1, 113.7, 111.4, 110.7, 55.9, 55.2, 52.5, 39.9, 34.0, 25.6, 17.5. **HRMS** (DART) calculated for C<sub>21</sub>H<sub>27</sub>O<sub>4</sub> [M]<sup>+</sup> 343.1904, measured: 343.1902. **IR** (neat) 2921, 1714, 1507, 1454, 1255, 1209, 1158, 1018, 747, 698, 549 cm<sup>-1</sup>.

#### 1-(4-methoxyphenyl)-5-phenylheptan-4-one (4n)



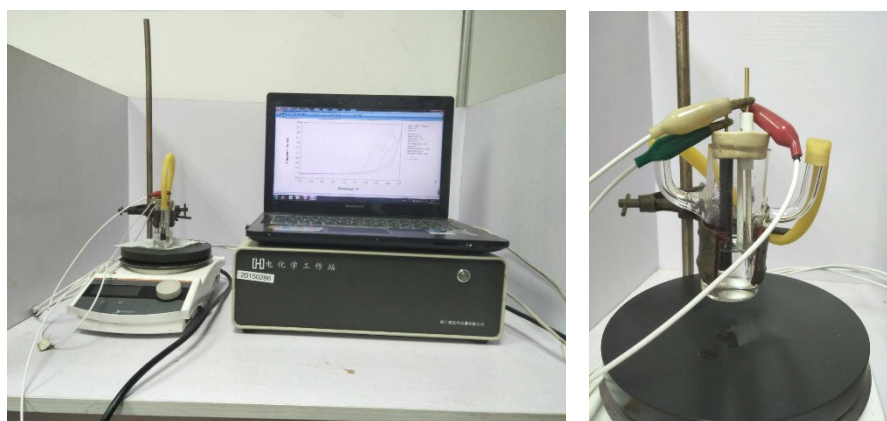
The title product was prepared according to the general procedure with 4-(4-methoxyphenyl)butanoic acid (**1k**) (58.3 mg, 0.3 mmol) and (3-bromopropyl)benzene (**2n**) (59.7 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford **4n** (46.3 mg, 52%) as a yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.35 (t, *J* = 7.2 Hz, 2H), 7.32 – 7.28 (m, 1H), 7.22 (d, *J* = 6.8 Hz, 2H), 6.97 (d, *J* = 8.4 Hz, 2H), 6.80 (d, *J* = 8.4 Hz, 2H), 3.80 (s, 3H), 3.52 (t, *J* = 7.2 Hz, 1H), 2.54 – 2.32 (m, 4H), 2.18 – 2.01 (m, 1H), 1.88 – 1.77 (m, 2H), 1.77 – 1.68 (m, 1H),

0.84 (t,  $J = 7.2$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  210.5, 157.8, 139.0, 133.8, 129.3, 128.8, 128.4, 127.2, 113.7, 60.9, 55.3, 41.0, 34.0, 25.5, 25.3, 12.2. HRMS (EI) calculated for  $\text{C}_{20}\text{H}_{24}\text{O}_2$   $[\text{M}]^+$  296.1771, measured: 296.1772. IR (neat) 2925, 1708, 1611, 1510, 1453, 1243, 1033, 807, 755, 699, 544  $\text{cm}^{-1}$ .

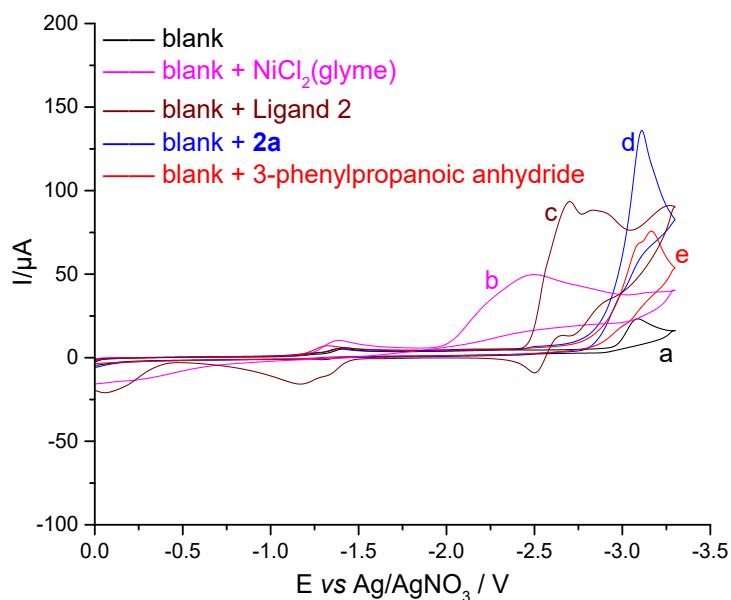
## 7 Mechanism Experiments

### 7.1 Cyclic Voltammetry

Cyclic voltammograms were recorded with a CHI660E potentiostat at room temperature in DMAc.  $n\text{Bu}_4\text{NBr}$  (0.1 M) was used as the supporting electrolyte, and a Glass Carbon electrode was used as the working electrode. The auxiliary electrode was a Pt sheet. All potentials are referenced against the  $\text{Ag}/\text{AgNO}_3$  redox couple. The scan rate was  $100 \text{ mV s}^{-1}$ .

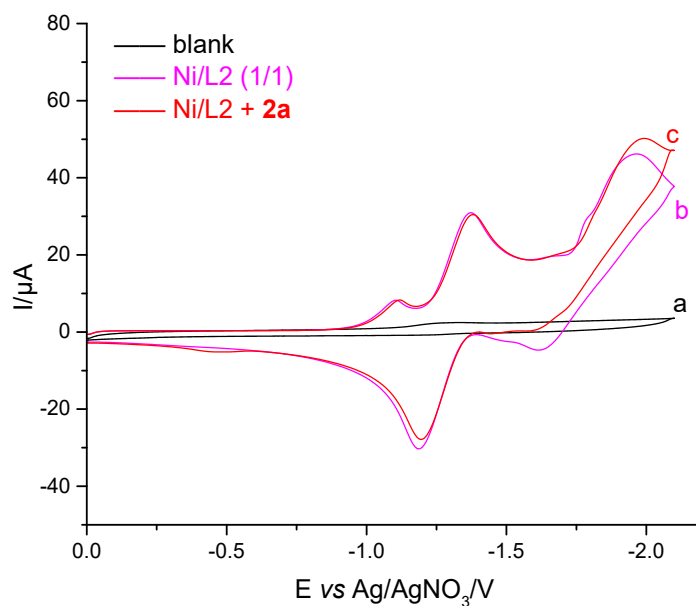


**Figure S1:** Photograph of setup used for cyclic voltammetry.

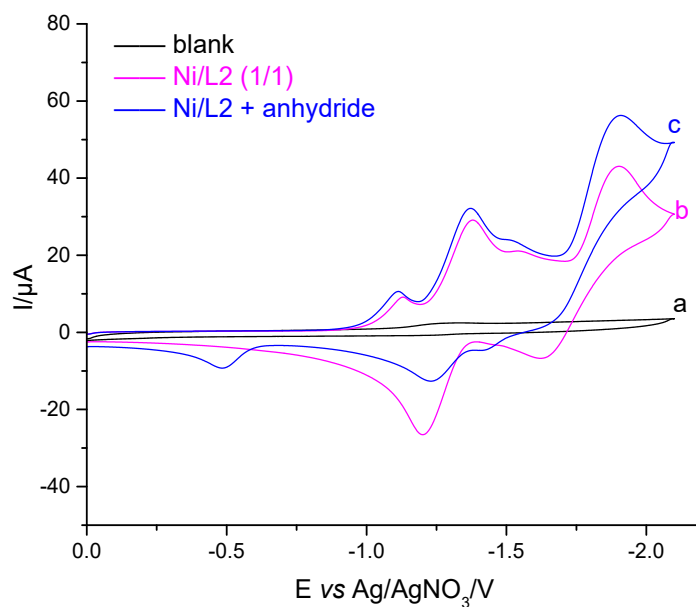


**Figure S2:** Cyclic voltammograms recorded on a glassy carbon electrode at  $100 \text{ mVs}^{-1}$  in: (a) DMA containing 0.1 M of  $n\text{Bu}_4\text{NBr}$ ; (b) solution (a) with 10 mM of  $\text{NiCl}_2 \cdot \text{glyme}$  added; (c) solution (a) with 10 mM of 2,9-Dimethyl-1,10-phenanthroline (**Ligand 2**) added; (d) solution (a) with 10 mM of (2-Bromoethyl)benzene (**2a**) added; (e) solution (a) with 10 mM of 3-phenylpropanoic anhydride

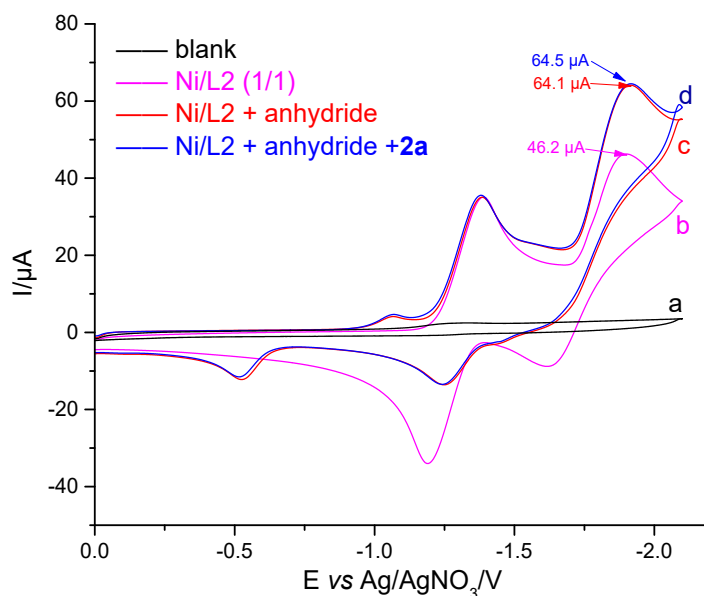
added.



**Figure S3.** Cyclic voltammograms recorded on a glassy carbon electrode at  $100 \text{ mVs}^{-1}$  in: (a) DMA containing  $0.1 \text{ M}$  of  $n\text{Bu}_4\text{NBr}$ ; (b) solution (a) with  $7.5 \text{ mM}$  of  $\text{NiCl}_2 \cdot \text{glyme}$  and 2,9-Dimethyl-1,10-phenanthroline ( $\text{Ni/L} = 1/1$ ) added; (c) solution (b) with  $10 \text{ mM}$  of **2a** added;



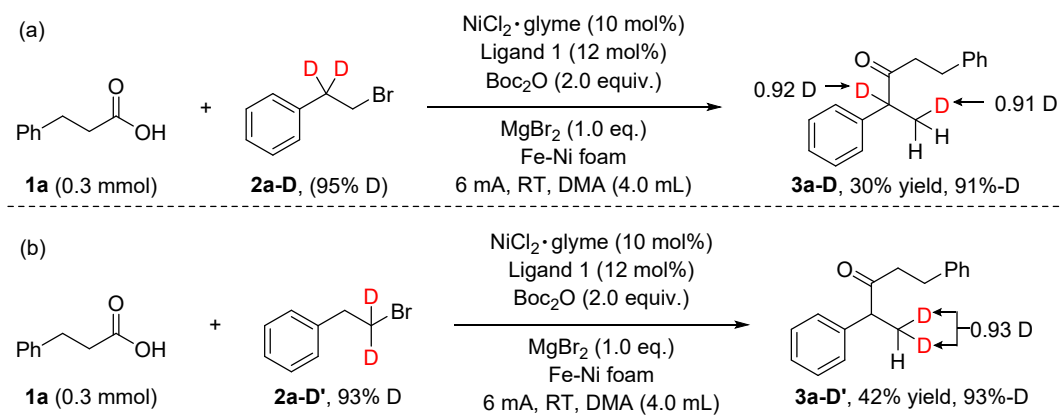
**Figure S4.** Cyclic voltammograms recorded on a glassy carbon electrode at  $100 \text{ mVs}^{-1}$  in: (a) DMA containing  $0.1 \text{ M}$  of  $n\text{Bu}_4\text{NBr}$ ; (b) solution (a) with  $7.5 \text{ mM}$  of  $\text{NiCl}_2 \cdot \text{glyme}$  and 2,9-Dimethyl-1,10-phenanthroline ( $\text{Ni/L} = 1/1$ ) added; (c) solution (b) with  $10 \text{ mM}$  of 3-phenylpropanoic anhydride added.



**Figure S5.** Cyclic voltammograms recorded on a glassy carbon electrode at 100 mVs<sup>-1</sup>: (a) DMA containing 0.1 M of *n*-Bu<sub>4</sub>NBr; (b) solution (a) with 7.5 mM of NiCl<sub>2</sub>·glyme and **Ligand 2** added; (c) solution (b) with 10 mM of 3-phenylpropanoic anhydride added; (d) solution (c) with 10 mM of **2a** added.

## 7.2 Deuterium-labeled Experiments

To gain further insight into this electrochemical reductive relay cross-coupling system, deuterium-labeled **2a-D** and **2a-D'** were prepared and subjected to the reactions. As shown in Scheme S1a, 91% deuterium incorporation was observed in the methyl group of **3a-D** and the H/D scrambling between methyl and benzyl groups was not observed in Scheme S1b. These results indicate that the styrene intermediate may be generated in the migratory process and the β-H elimination/reductive elimination sequence is irreversible in the formation of product **3a-D** or **3a-D'**.



**Scheme S1.** Deuterium-labeled Experiments

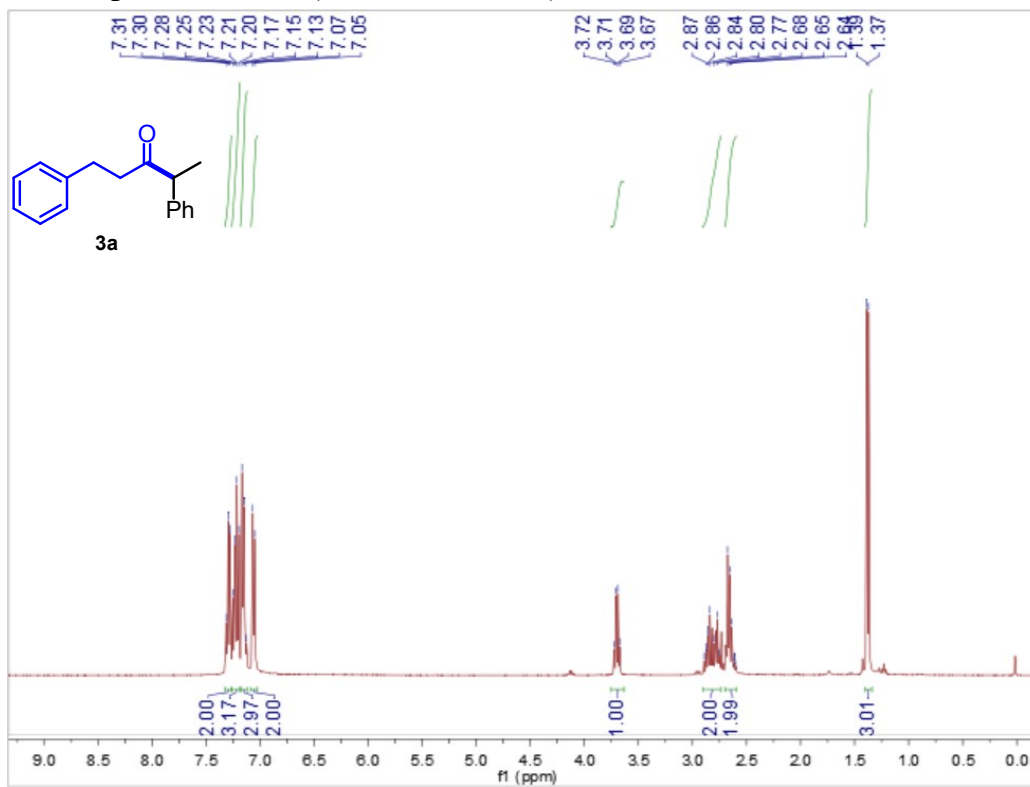
## 8 Reference

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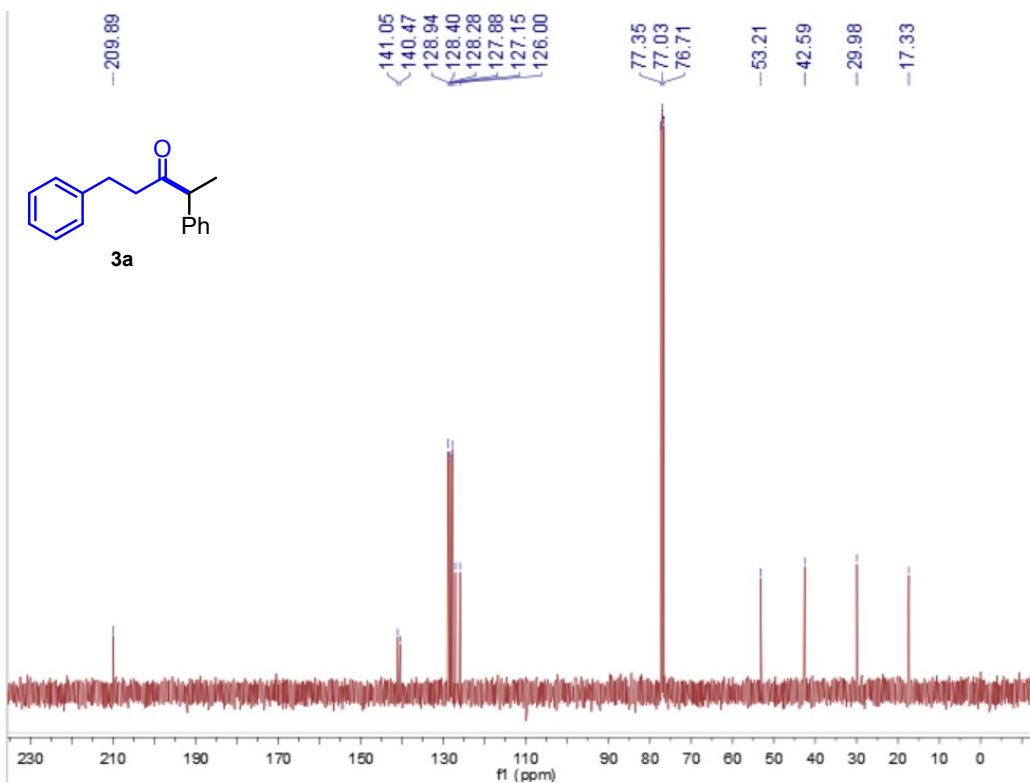


## 9 Spectra of Compounds

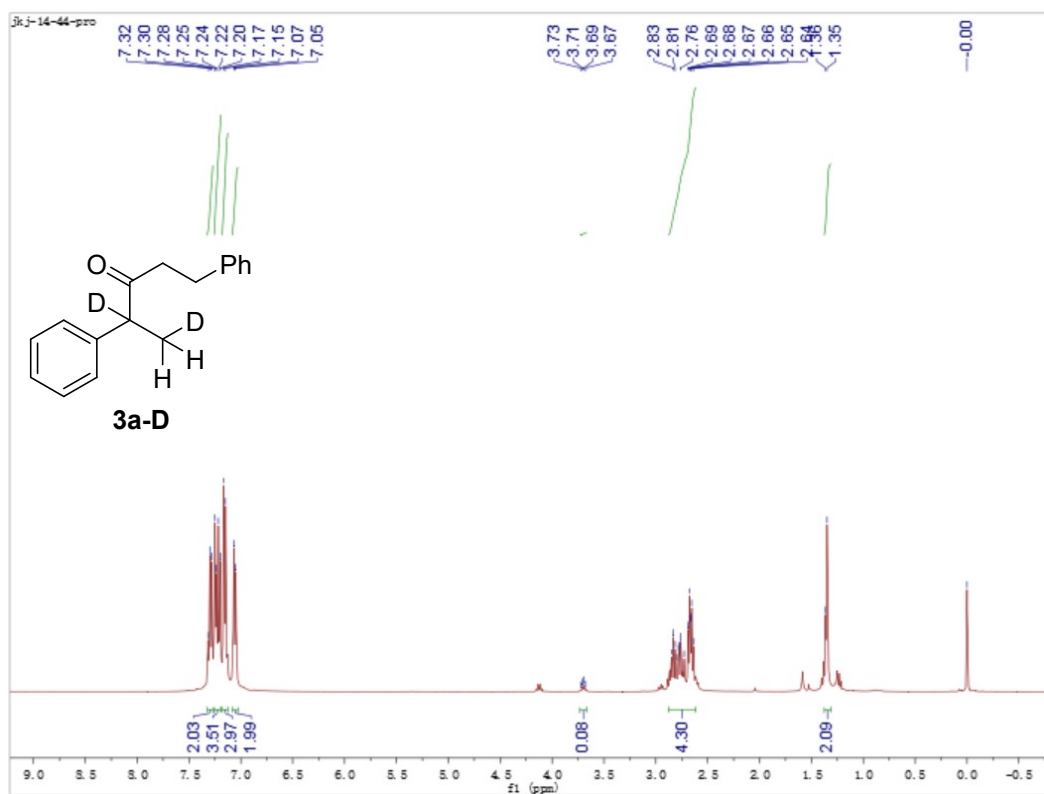
### <sup>1</sup>H NMR Spectrum of 3a (CDCl<sub>3</sub>, 400 MHz)



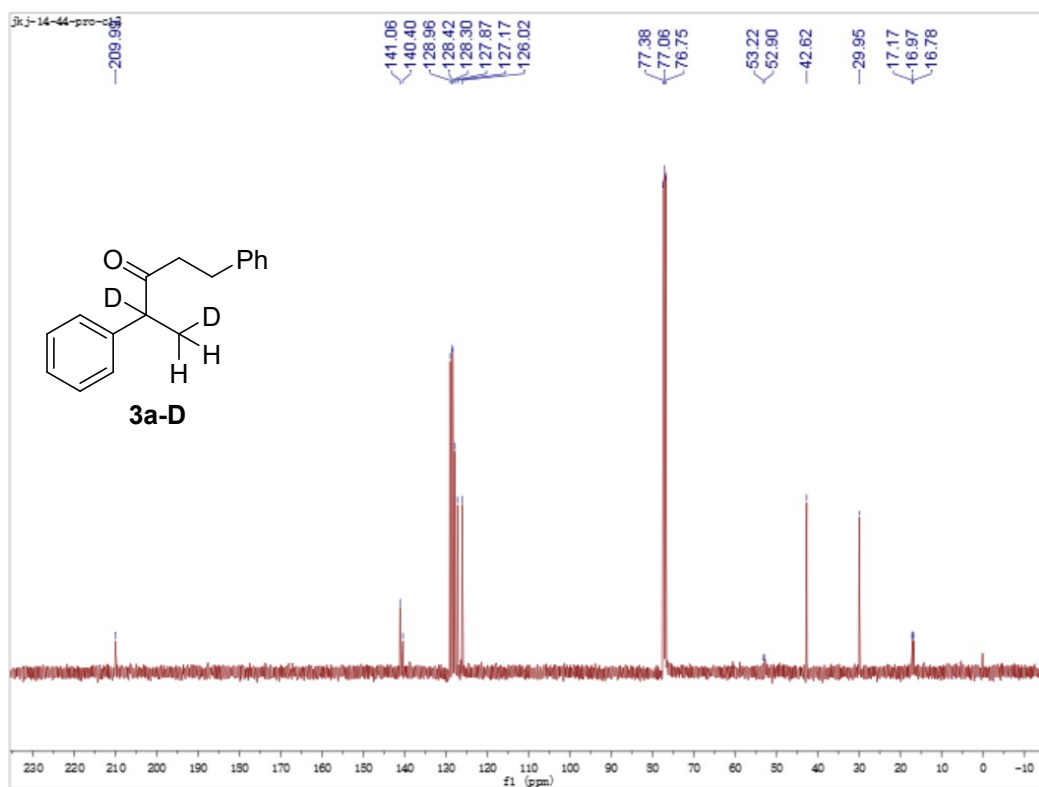
### <sup>13</sup>C NMR Spectrum of 3a (CDCl<sub>3</sub>, 101 MHz)



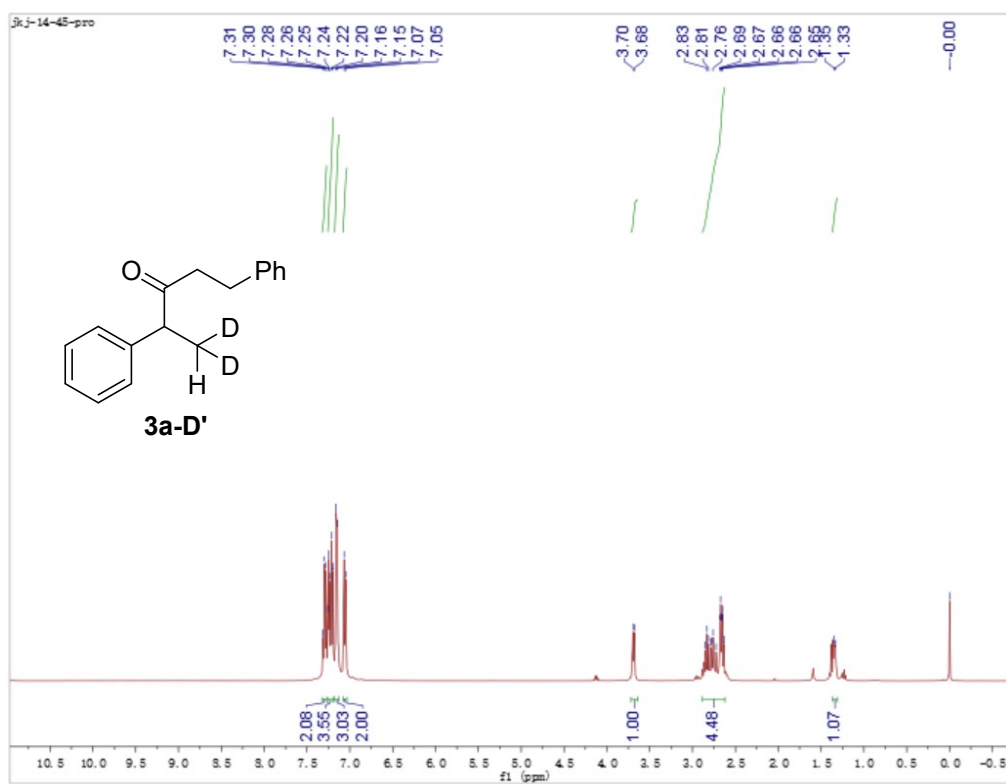
### $^1\text{H}$ NMR Spectrum of 3a-D ( $\text{CDCl}_3$ , 400 MHz)



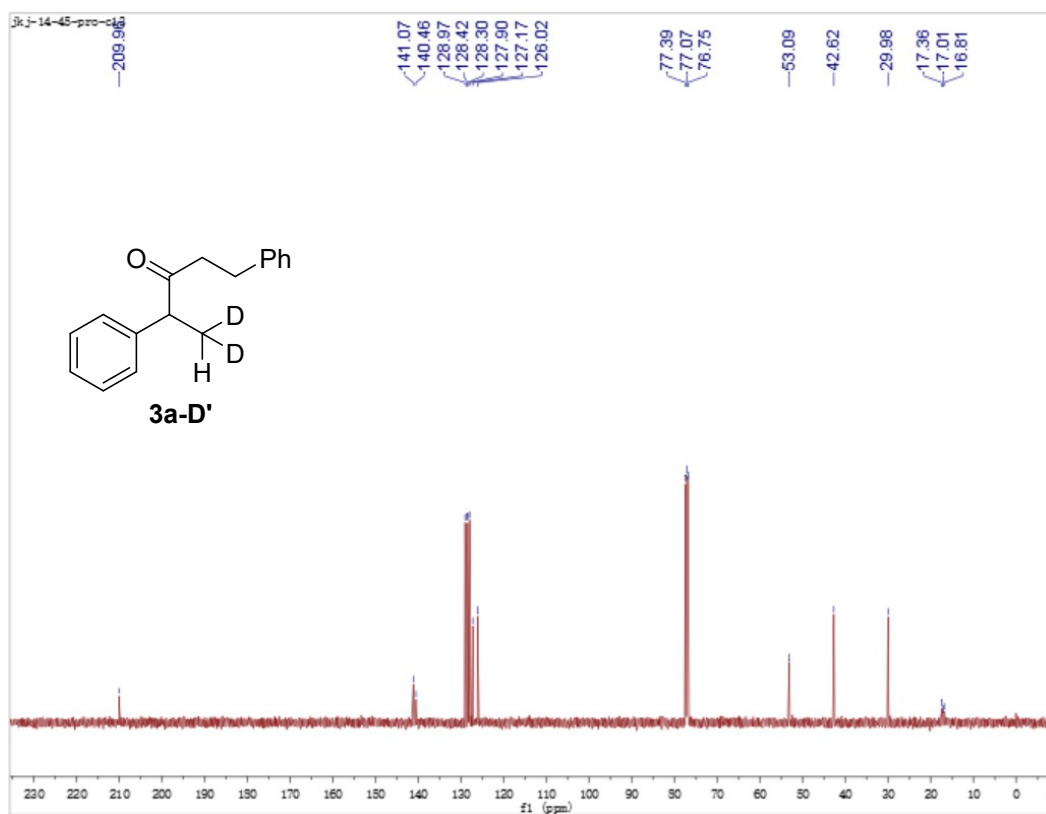
### $^{13}\text{C}$ NMR Spectrum of 3a-D ( $\text{CDCl}_3$ , 101 MHz)



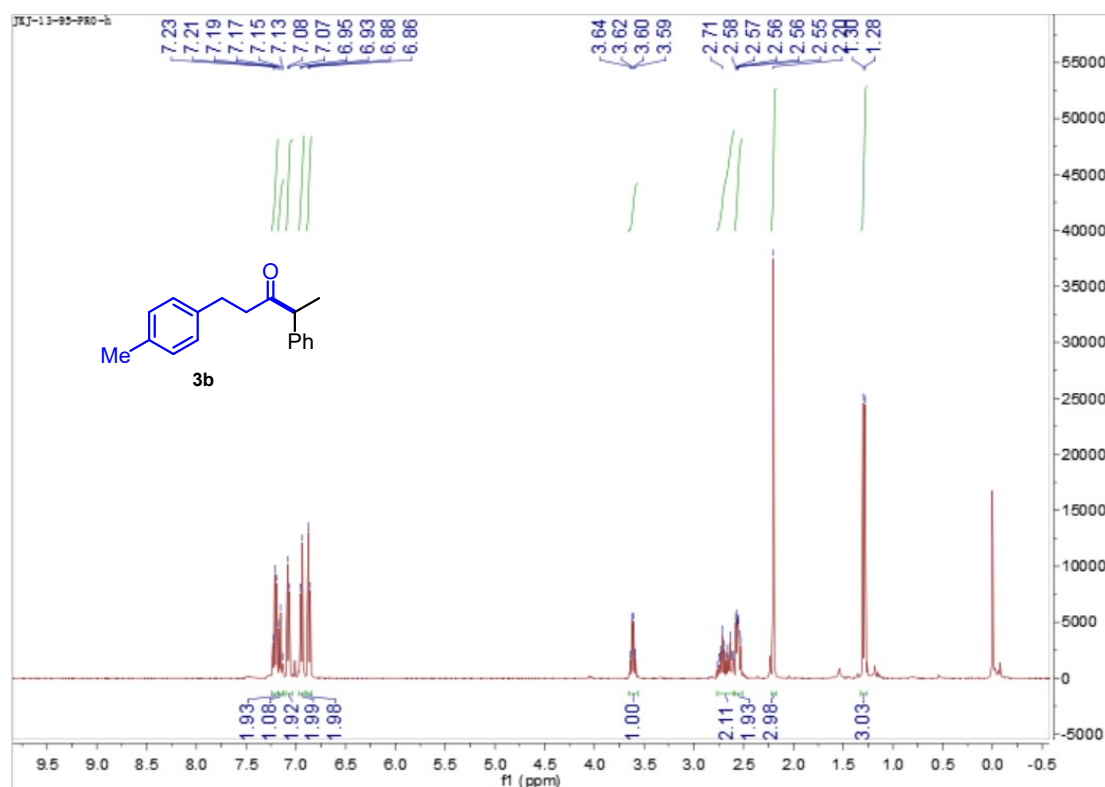
### $^1\text{H}$ NMR Spectrum of 3a-D' ( $\text{CDCl}_3$ , 400 MHz)



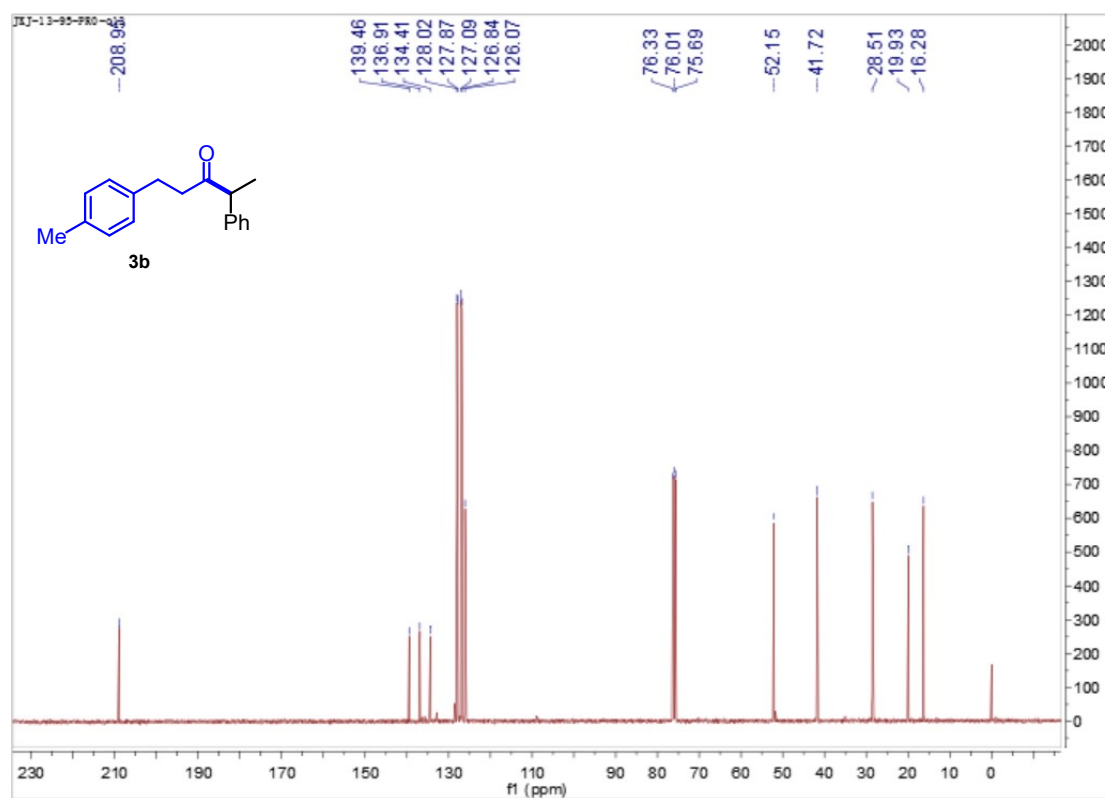
### $^{13}\text{C}$ NMR Spectrum of 3a-D' ( $\text{CDCl}_3$ , 101 MHz)



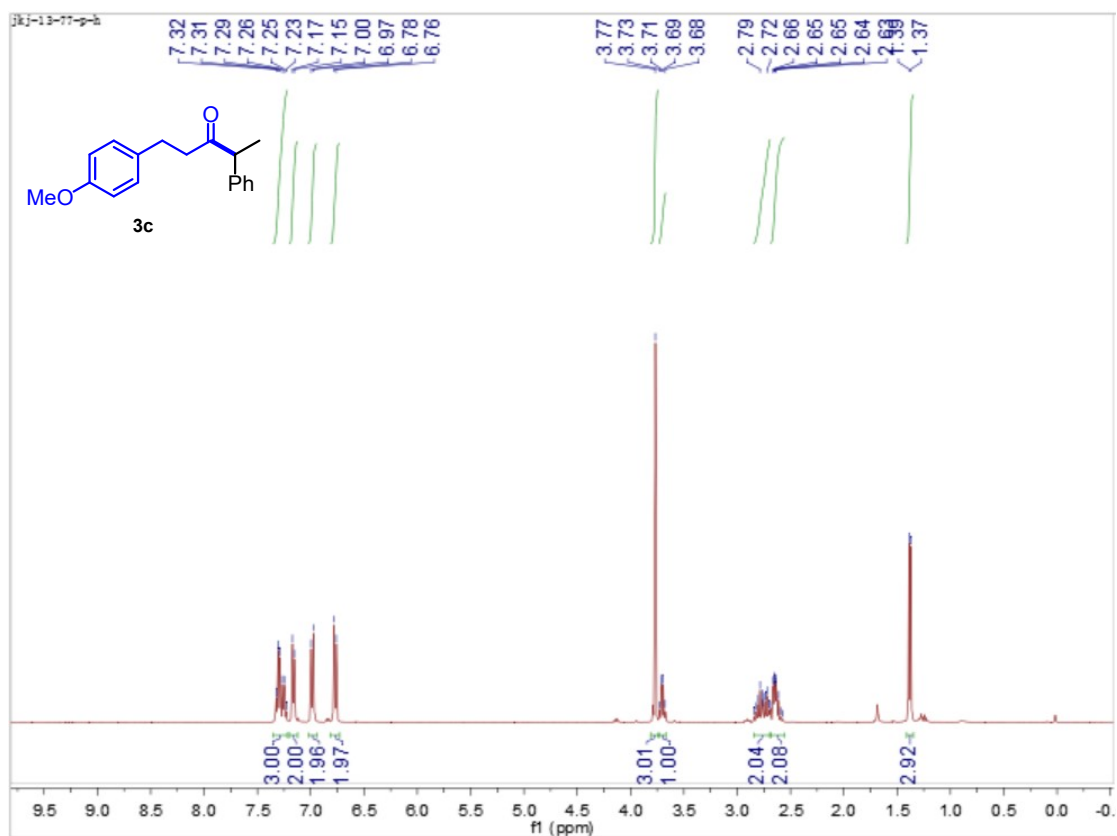
### <sup>1</sup>H NMR Spectrum of 3b (CDCl<sub>3</sub>, 400 MHz)



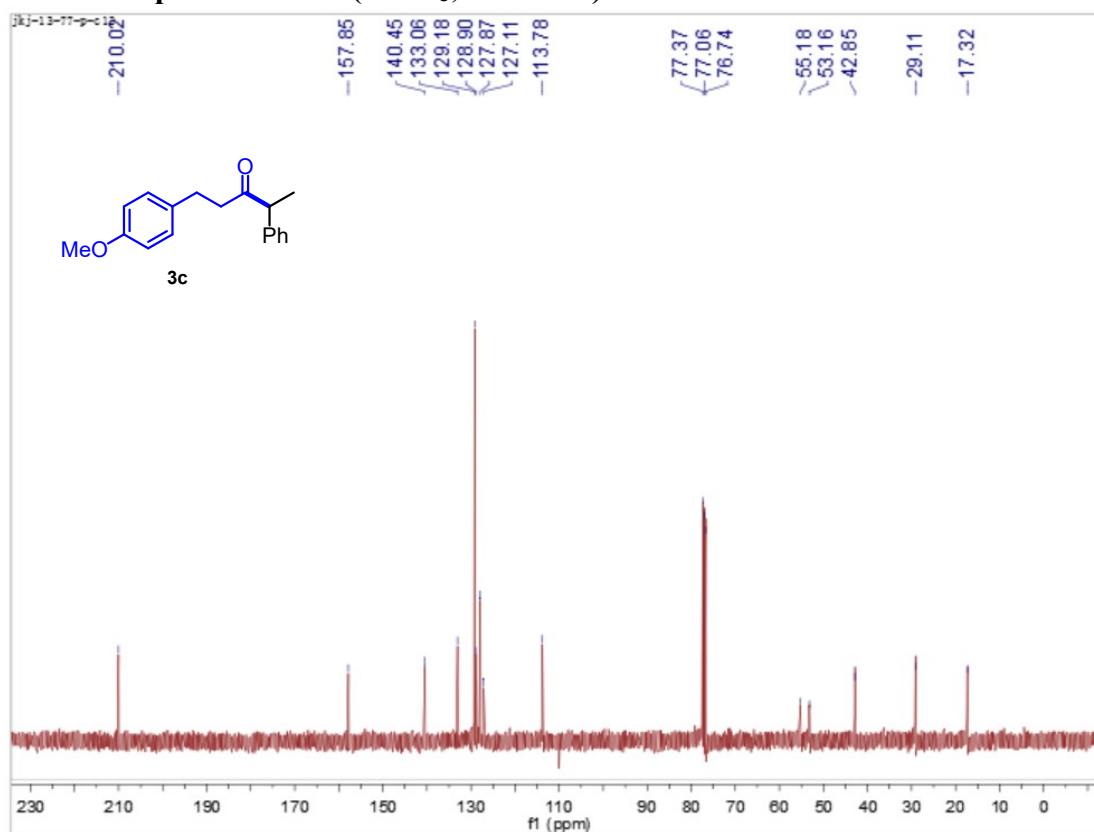
### <sup>13</sup>C NMR Spectrum of 3b (CDCl<sub>3</sub>, 101 MHz)



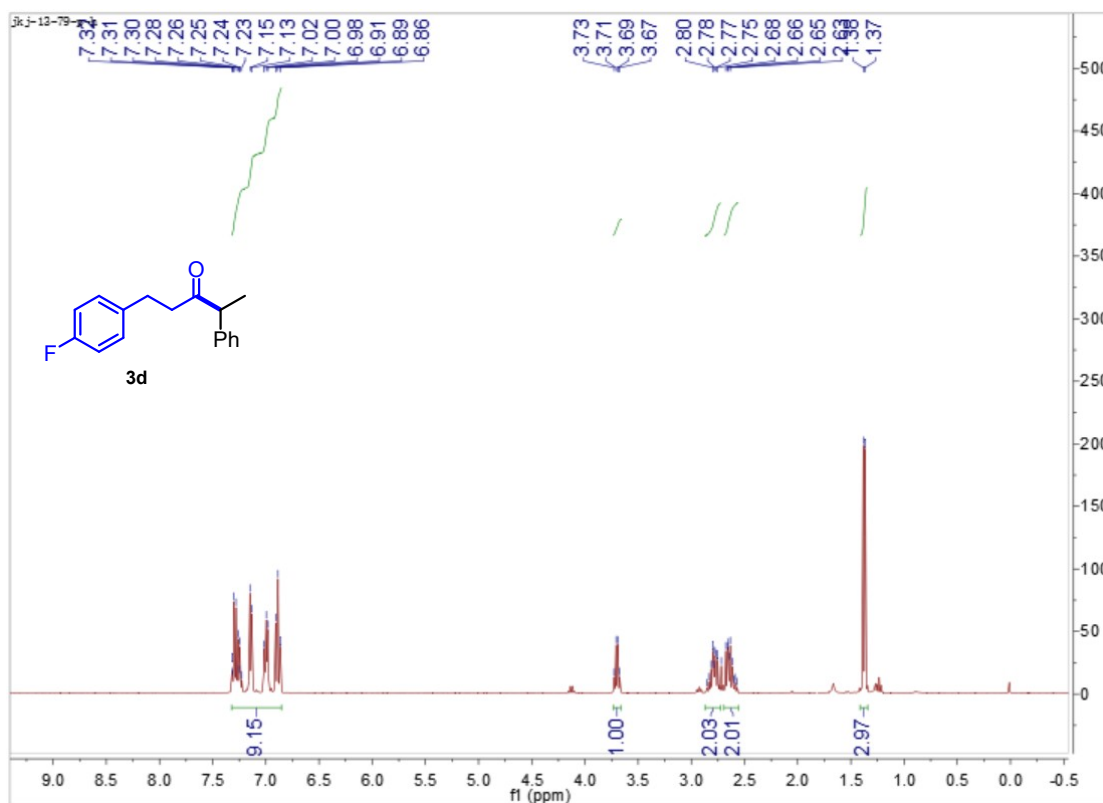
### <sup>1</sup>H NMR Spectrum of 3c (CDCl<sub>3</sub>, 400 MHz)



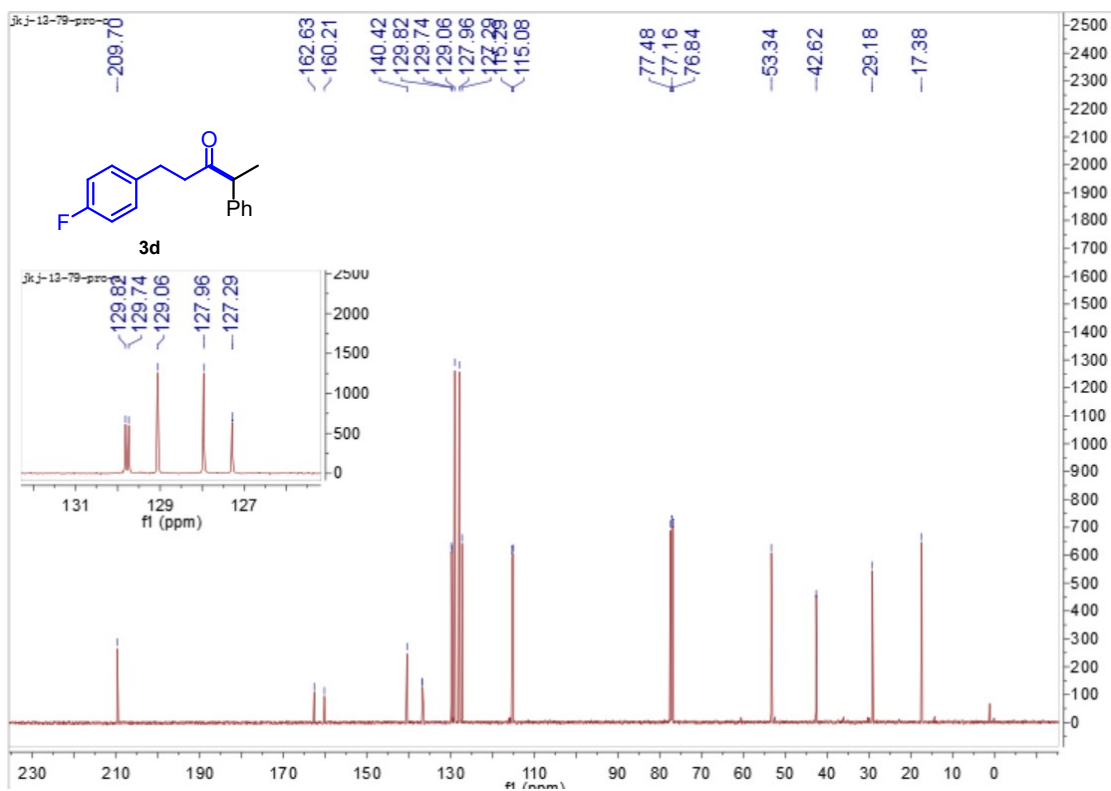
### <sup>13</sup>C NMR Spectrum of 3c (CDCl<sub>3</sub>, 101 MHz)



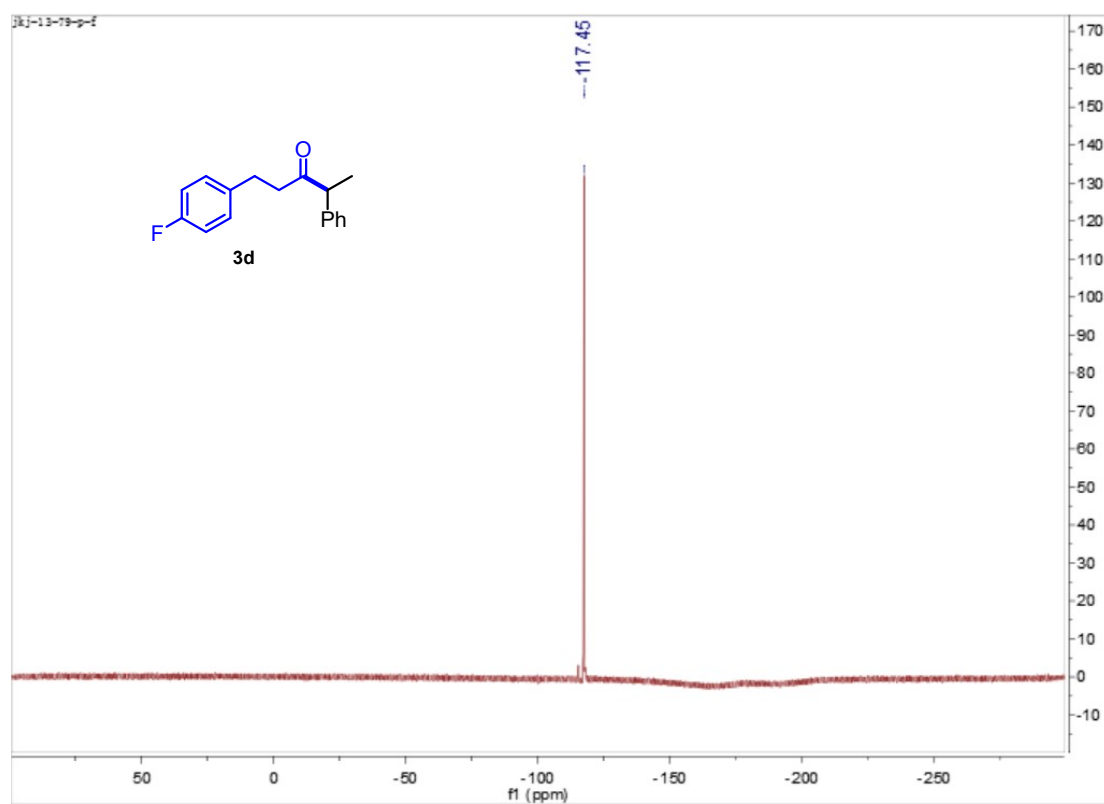
### <sup>1</sup>H NMR Spectrum of 3d (CDCl<sub>3</sub>, 400 MHz)



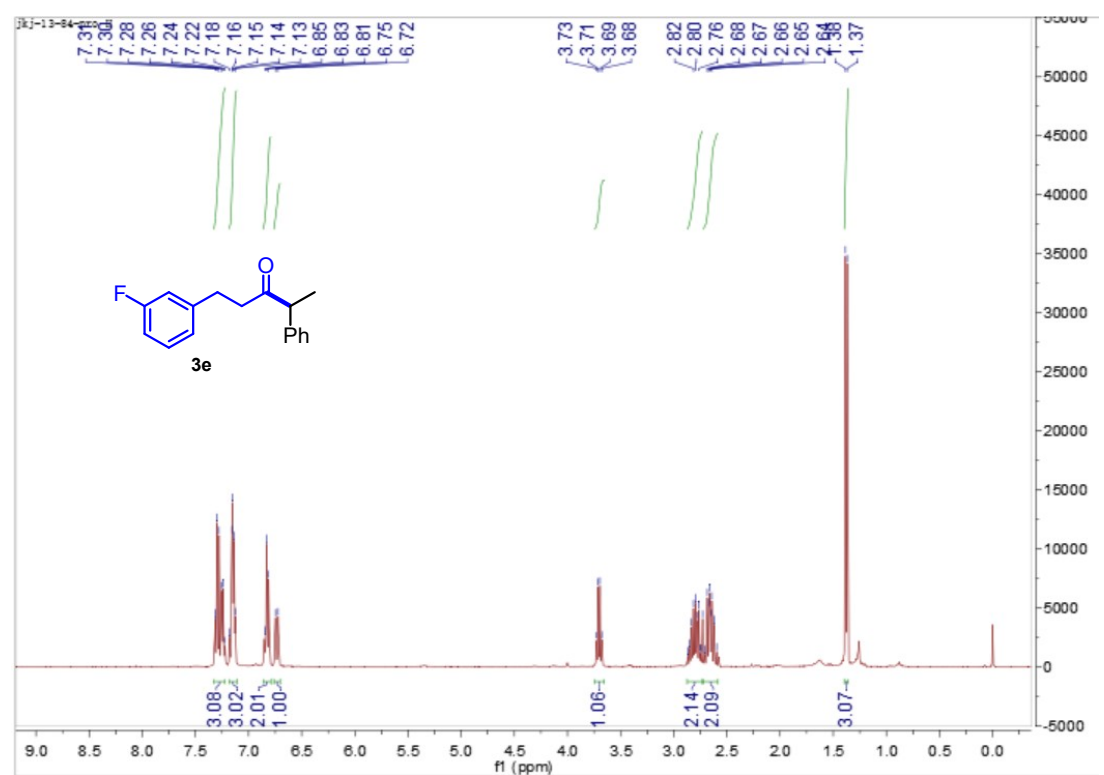
### <sup>13</sup>C NMR Spectrum of 3d (CDCl<sub>3</sub>, 101 MHz)



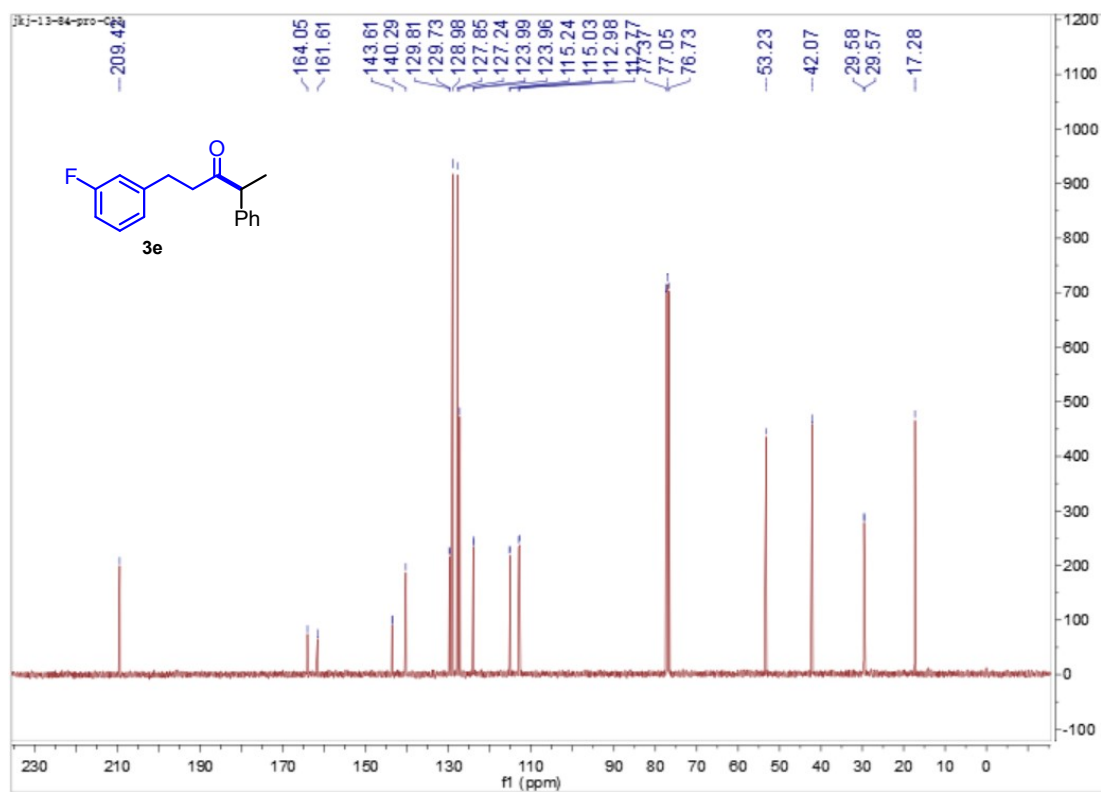
### <sup>19</sup>F NMR Spectrum of 3d (CDCl<sub>3</sub>, 376 MHz)



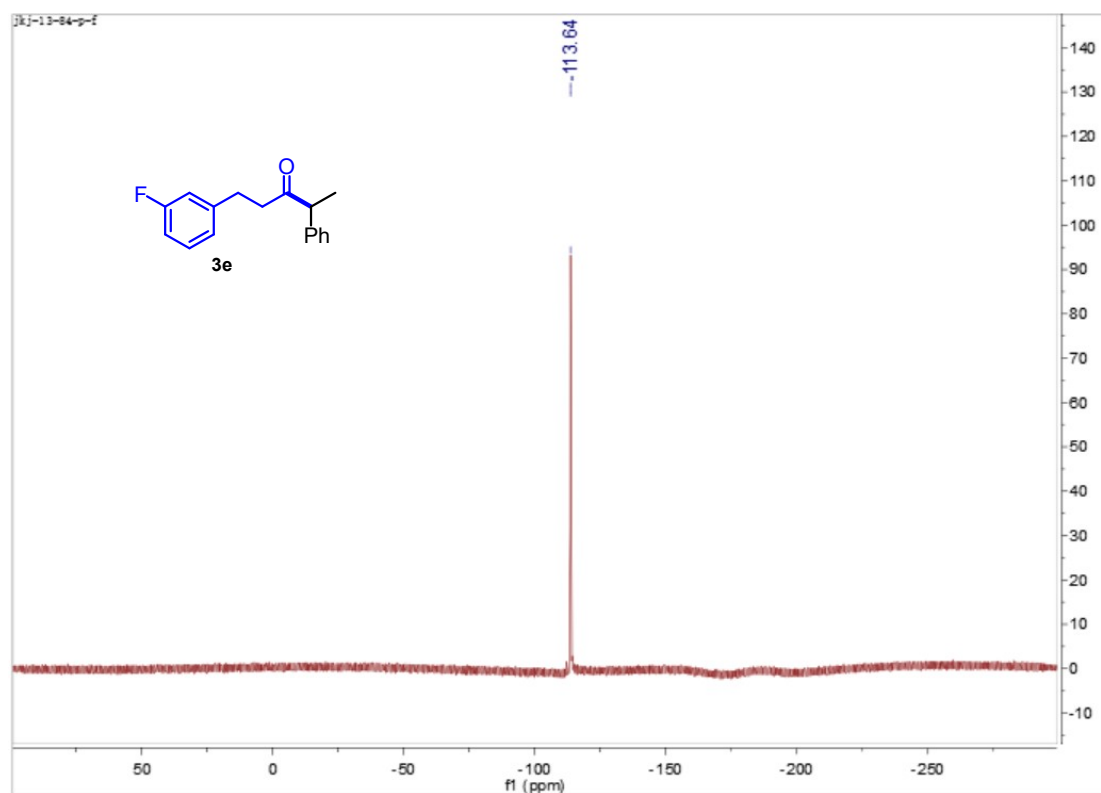
### <sup>1</sup>H NMR Spectrum of 3e (CDCl<sub>3</sub>, 400 MHz)



### <sup>13</sup>C NMR Spectrum of 3e (CDCl<sub>3</sub>, 101 MHz)

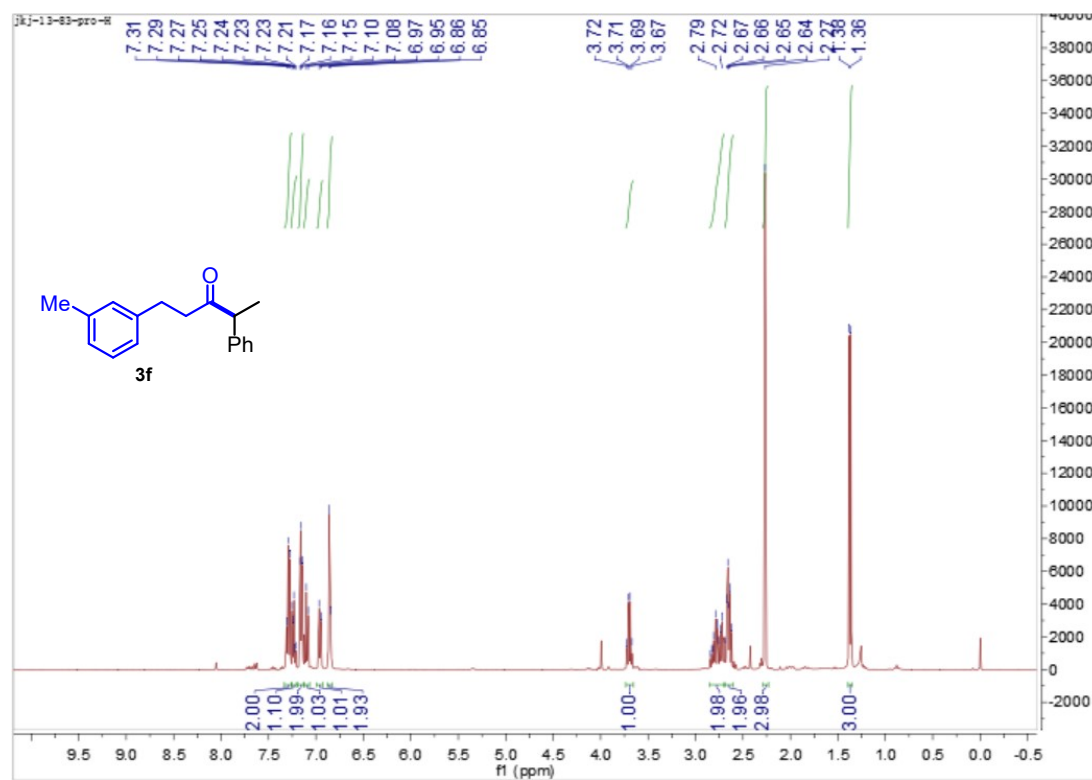


### <sup>19</sup>F NMR Spectrum of 3e (CDCl<sub>3</sub>, 376 MHz)

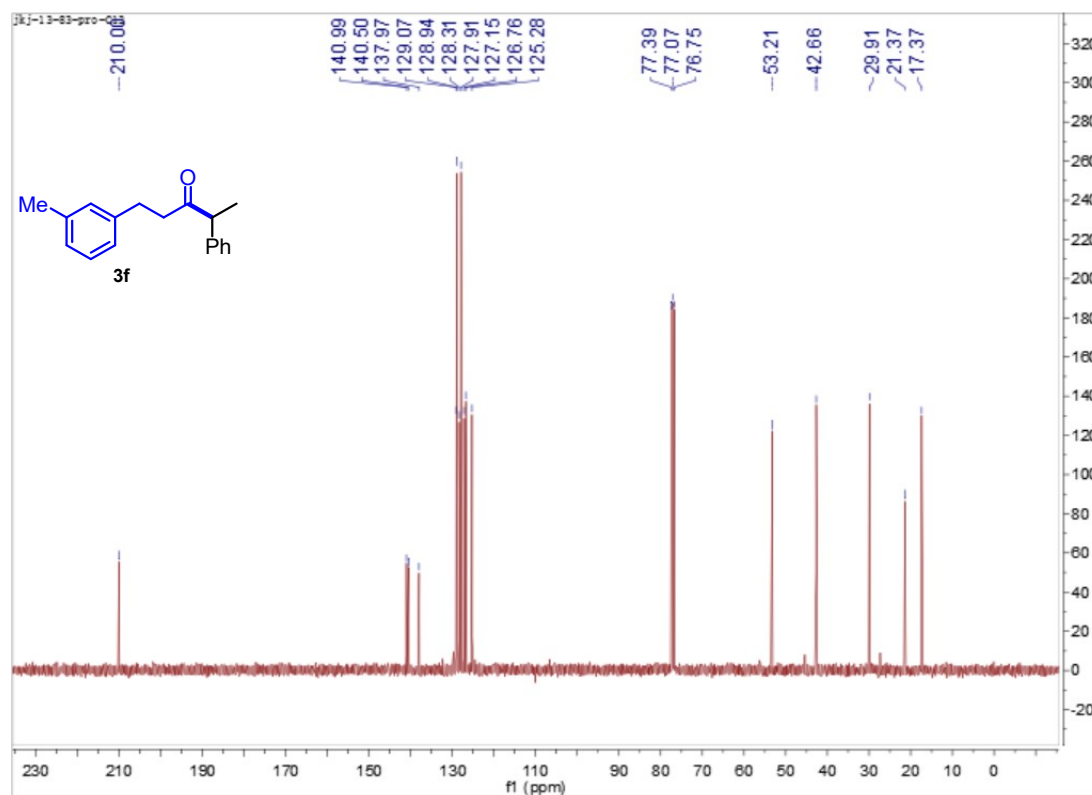




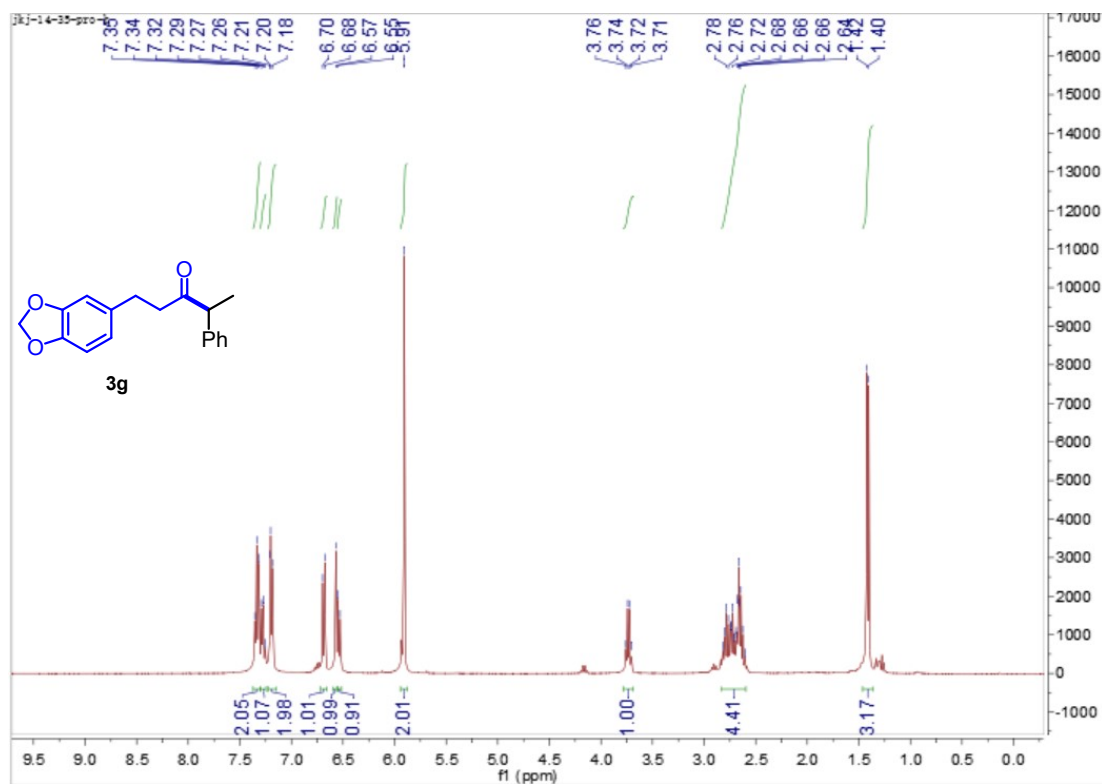
### <sup>1</sup>H NMR Spectrum of 3f (CDCl<sub>3</sub>, 400 MHz)



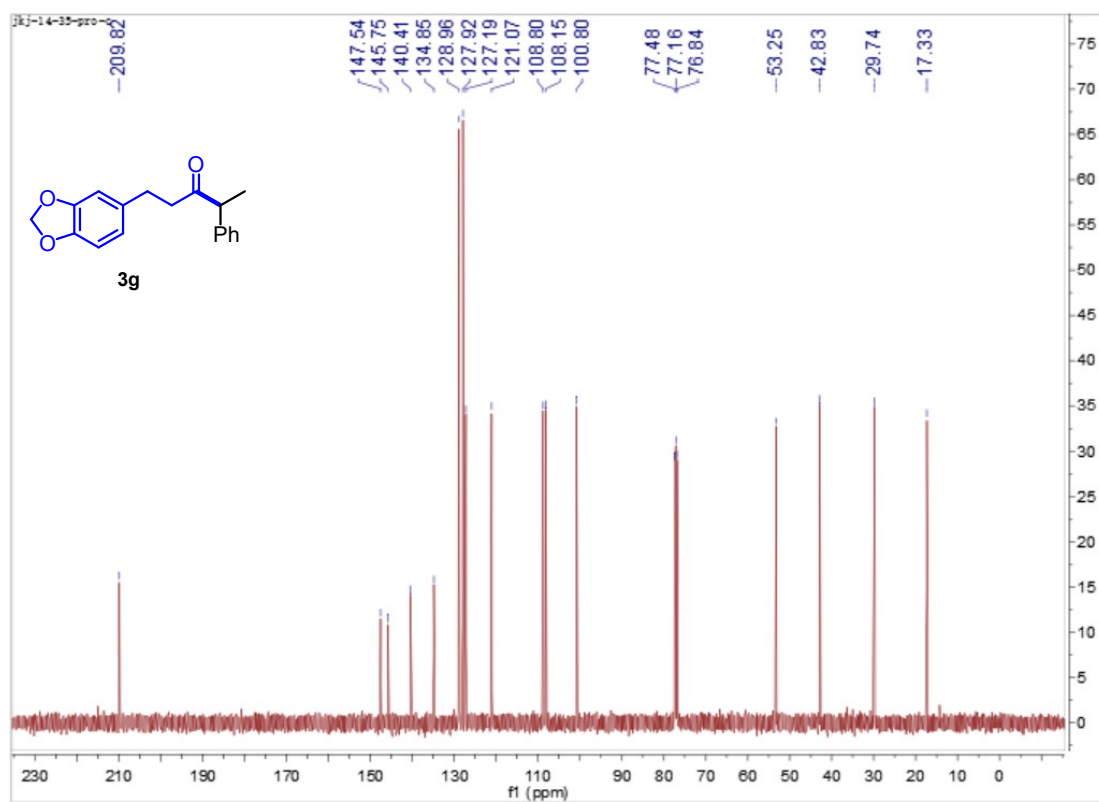
### <sup>13</sup>C NMR Spectrum of 3f (CDCl<sub>3</sub>, 101 MHz)



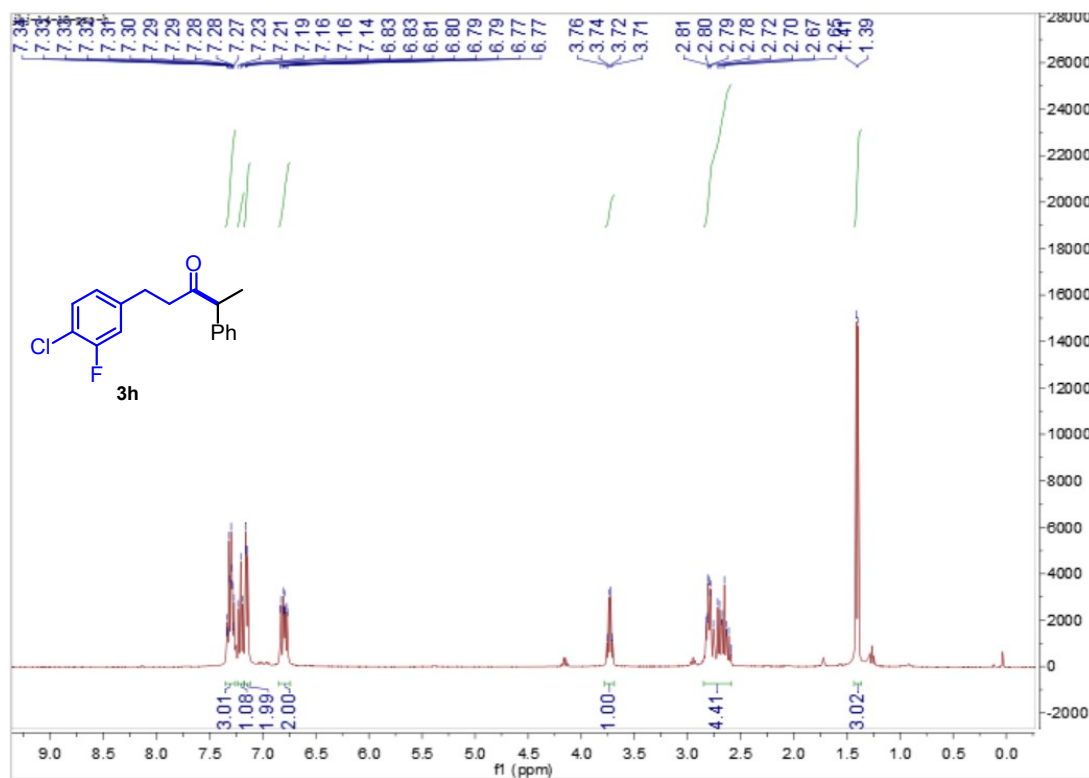
### <sup>1</sup>H NMR Spectrum of 3g (CDCl<sub>3</sub>, 400 MHz)



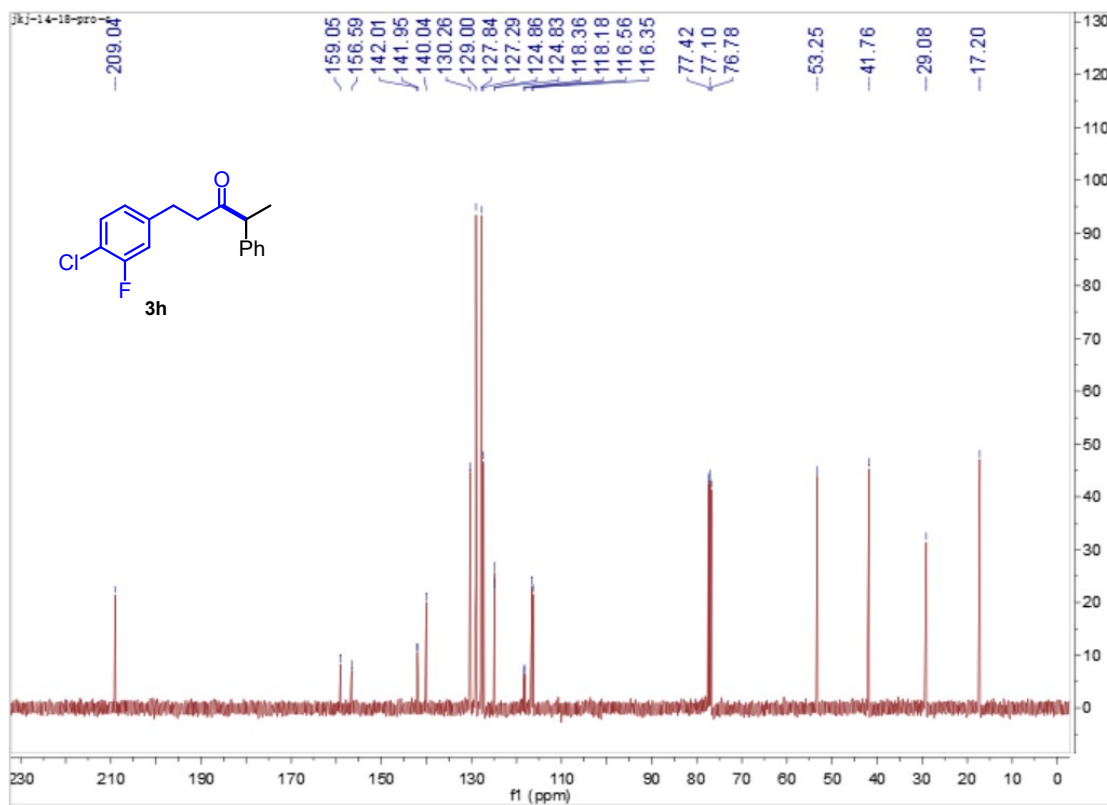
### <sup>13</sup>C NMR Spectrum of 3g (CDCl<sub>3</sub>, 101 MHz)



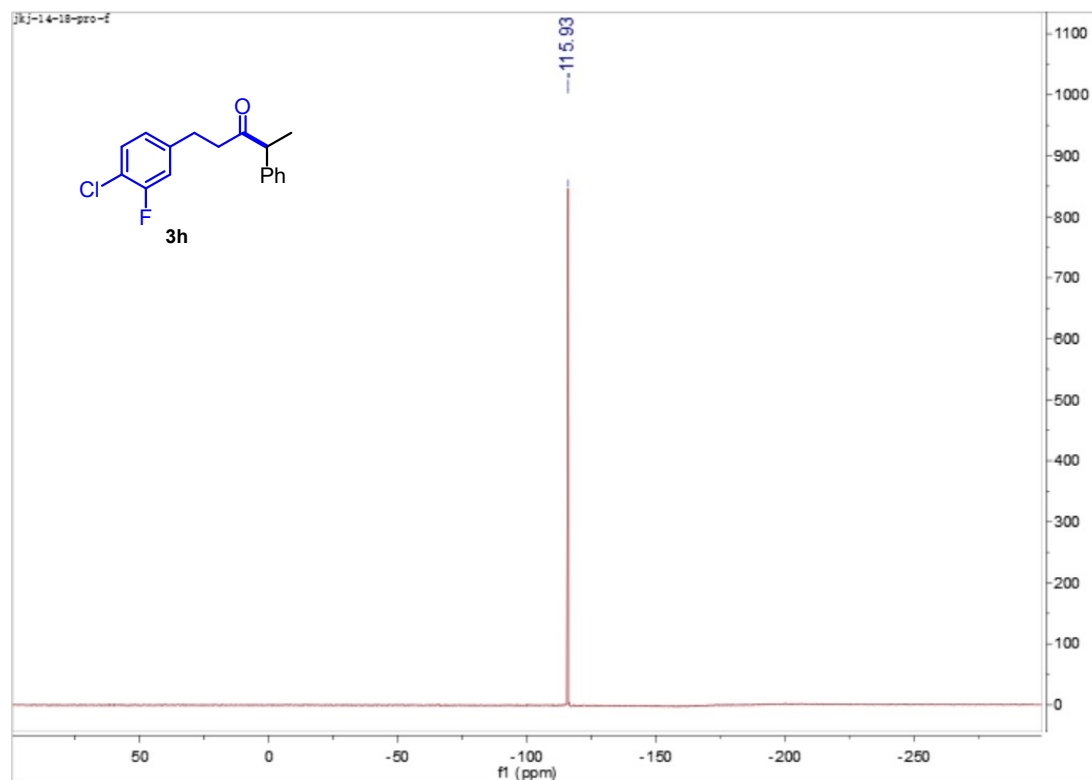
### <sup>1</sup>H NMR Spectrum of 3h (CDCl<sub>3</sub>, 400 MHz)



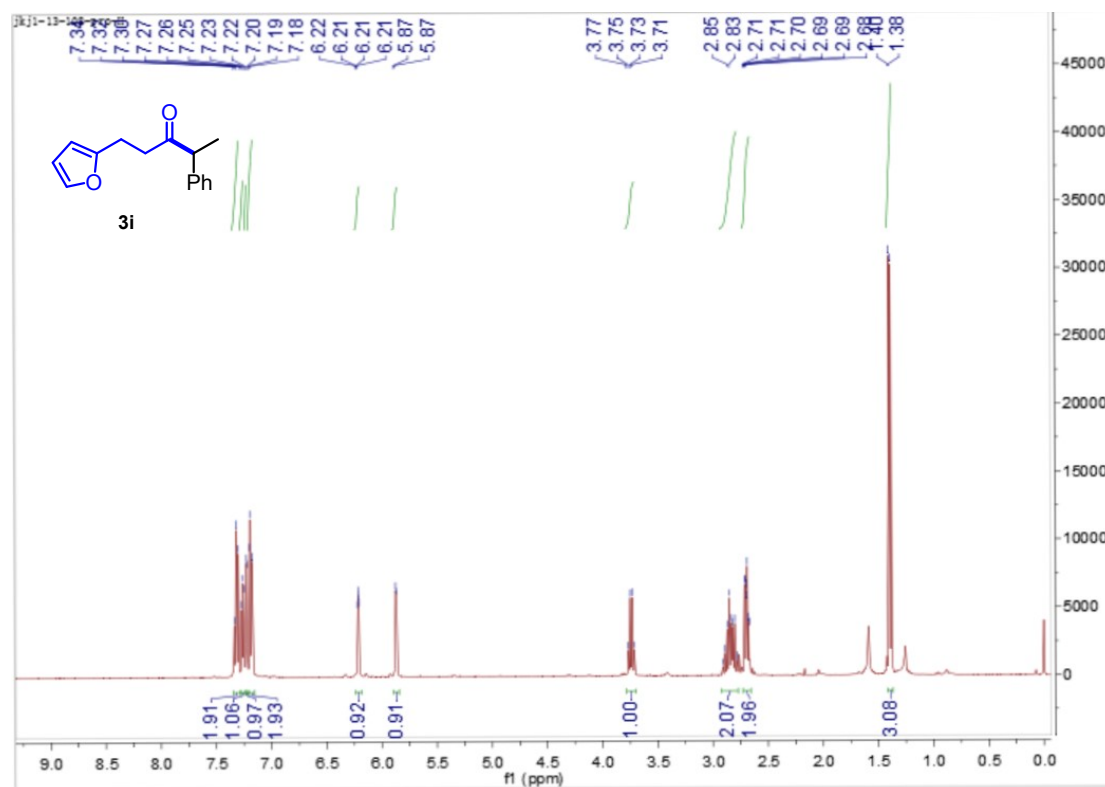
### <sup>13</sup>C NMR Spectrum of 3h (CDCl<sub>3</sub>, 101 MHz)



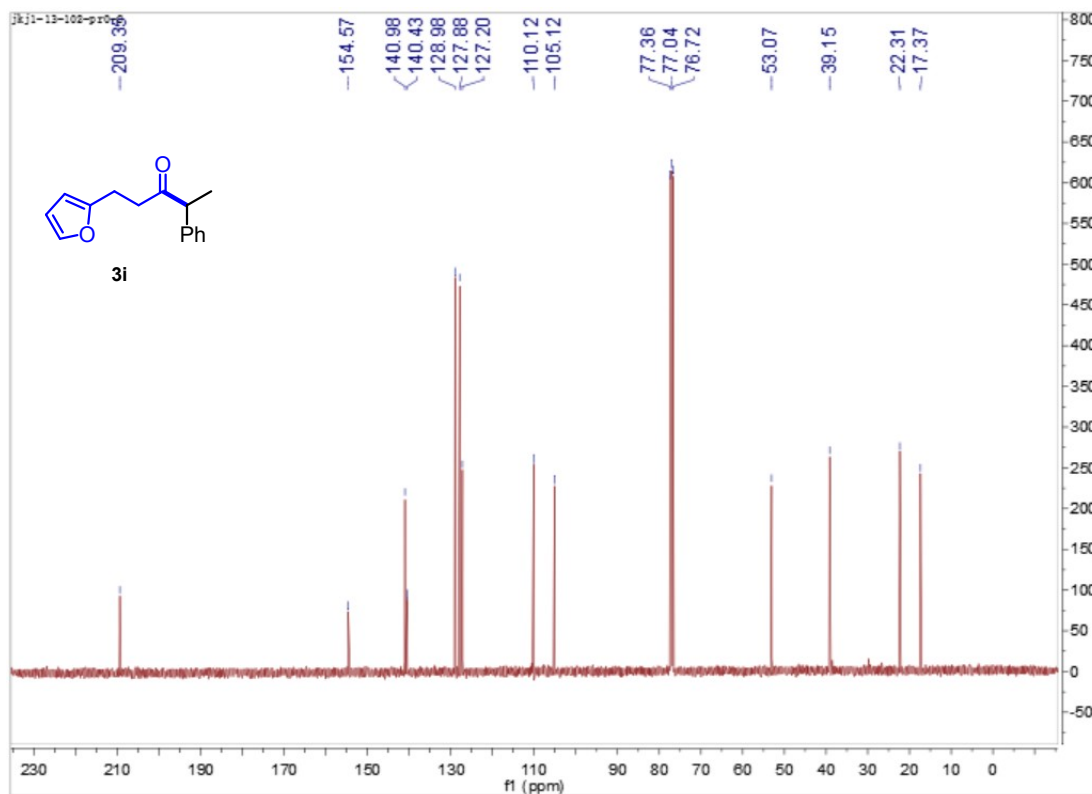
### <sup>19</sup>F NMR Spectrum of 3h (CDCl<sub>3</sub>, 376 MHz)



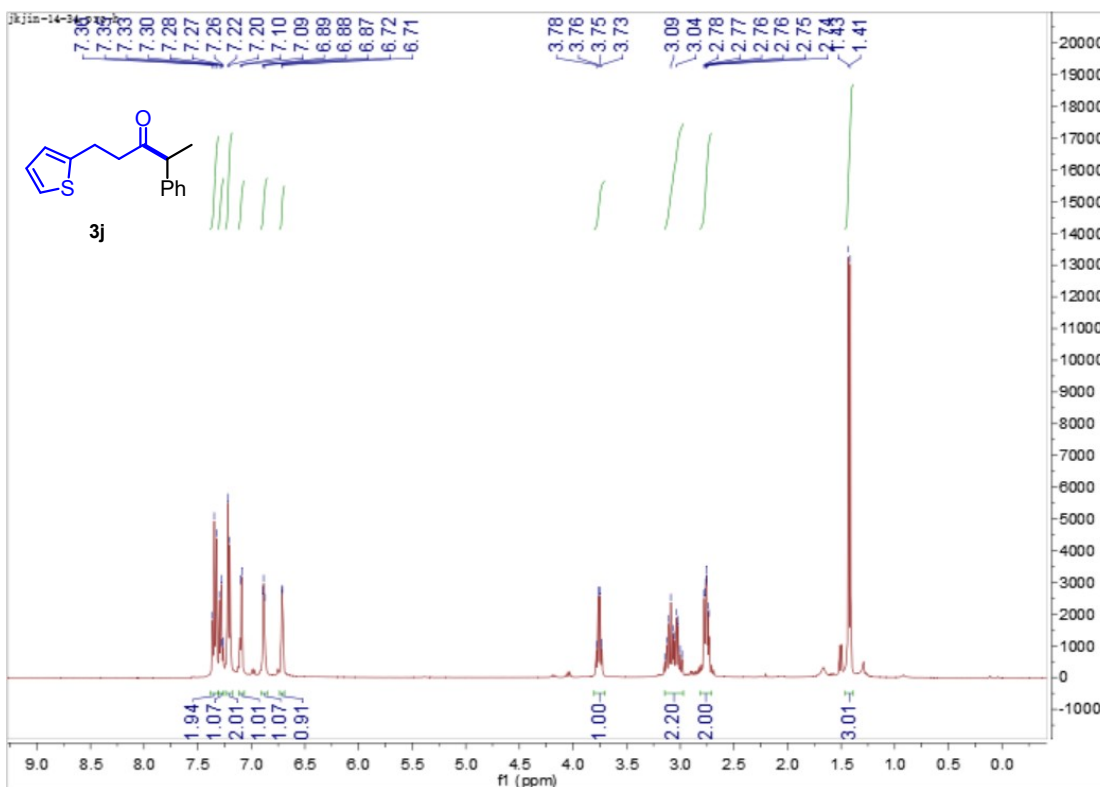
### <sup>1</sup>H NMR Spectrum of 3i (CDCl<sub>3</sub>, 400 MHz)



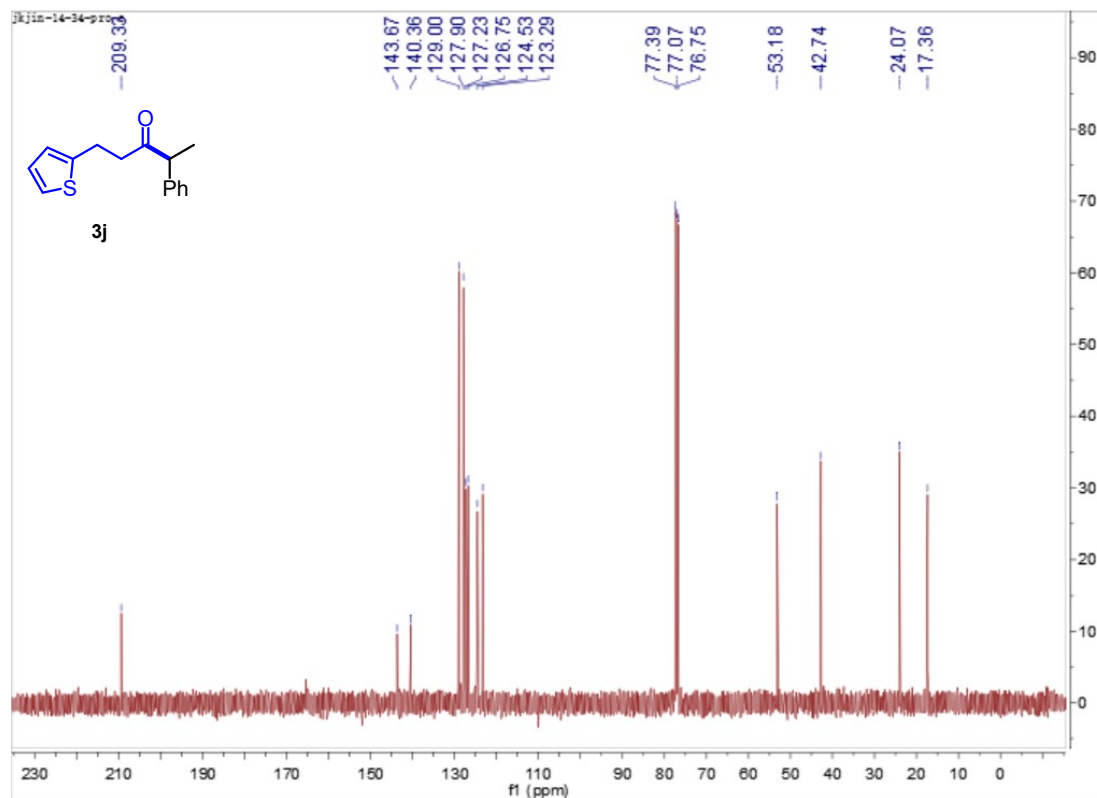
### <sup>13</sup>C NMR Spectrum of 3i (CDCl<sub>3</sub>, 101 MHz)



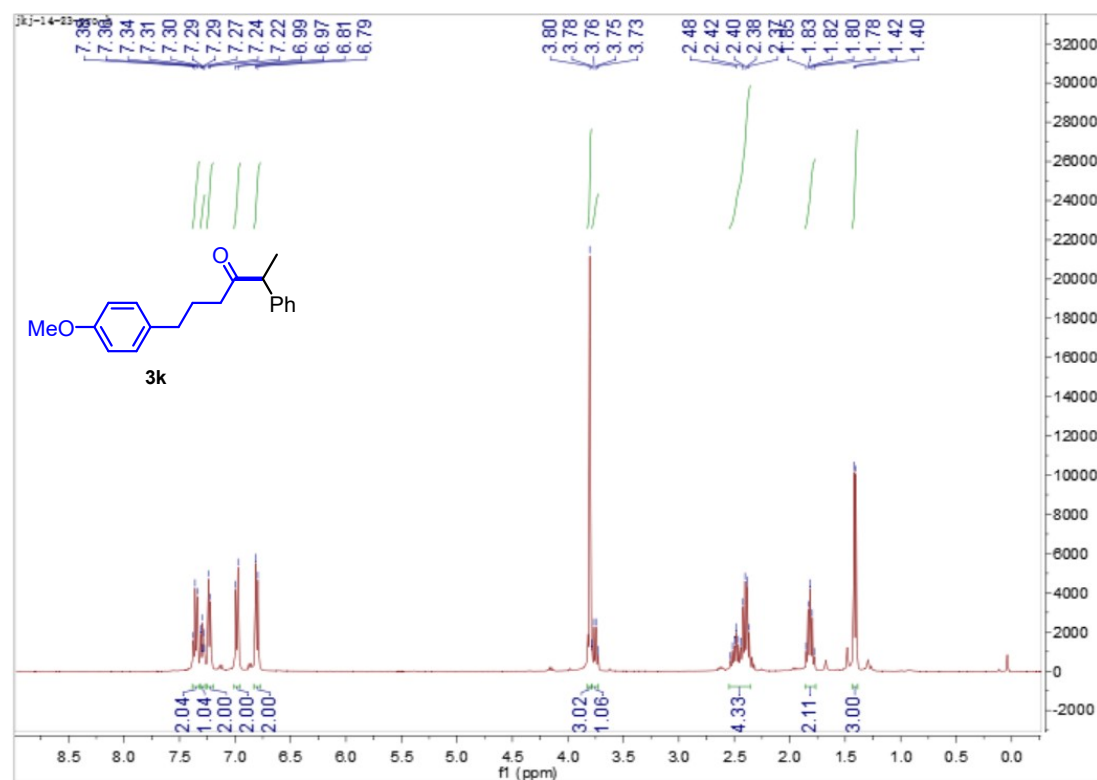
### <sup>1</sup>H NMR Spectrum of 3j (CDCl<sub>3</sub>, 400 MHz)



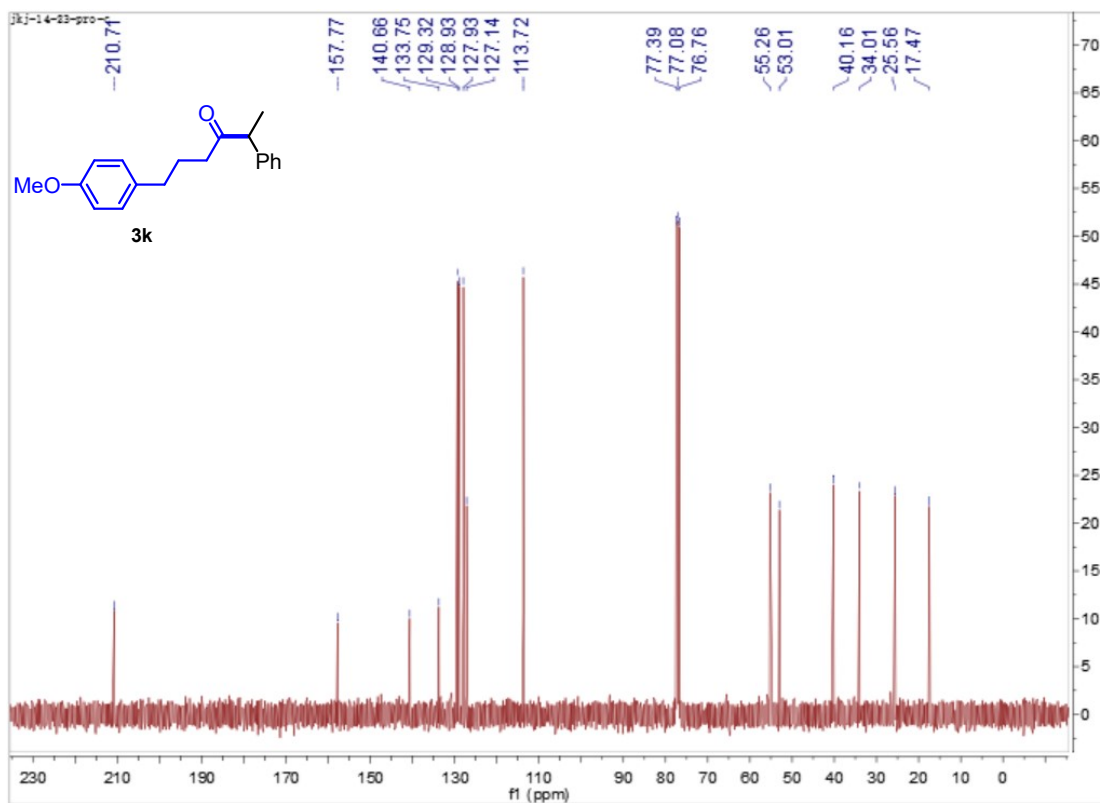
### $^{13}\text{C}$ NMR Spectrum of 3j (CDCl<sub>3</sub>, 101 MHz)



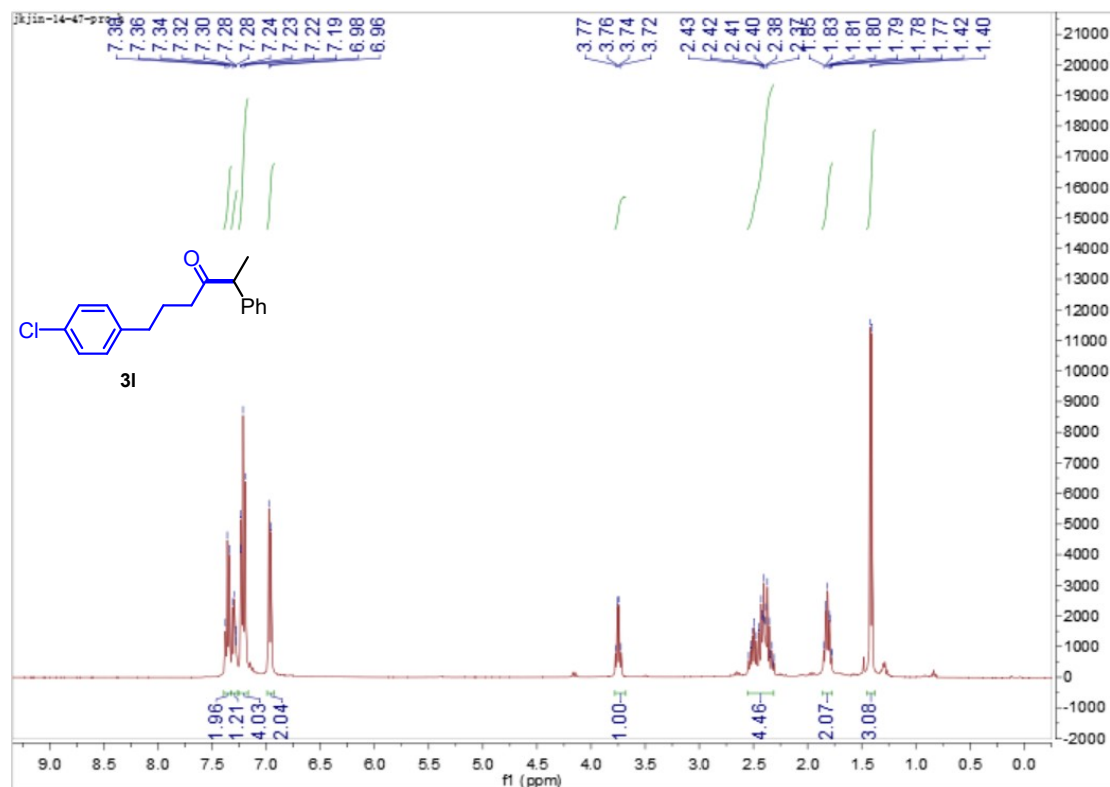
### $^1\text{H}$ NMR Spectrum of 3k (CDCl<sub>3</sub>, 400 MHz)



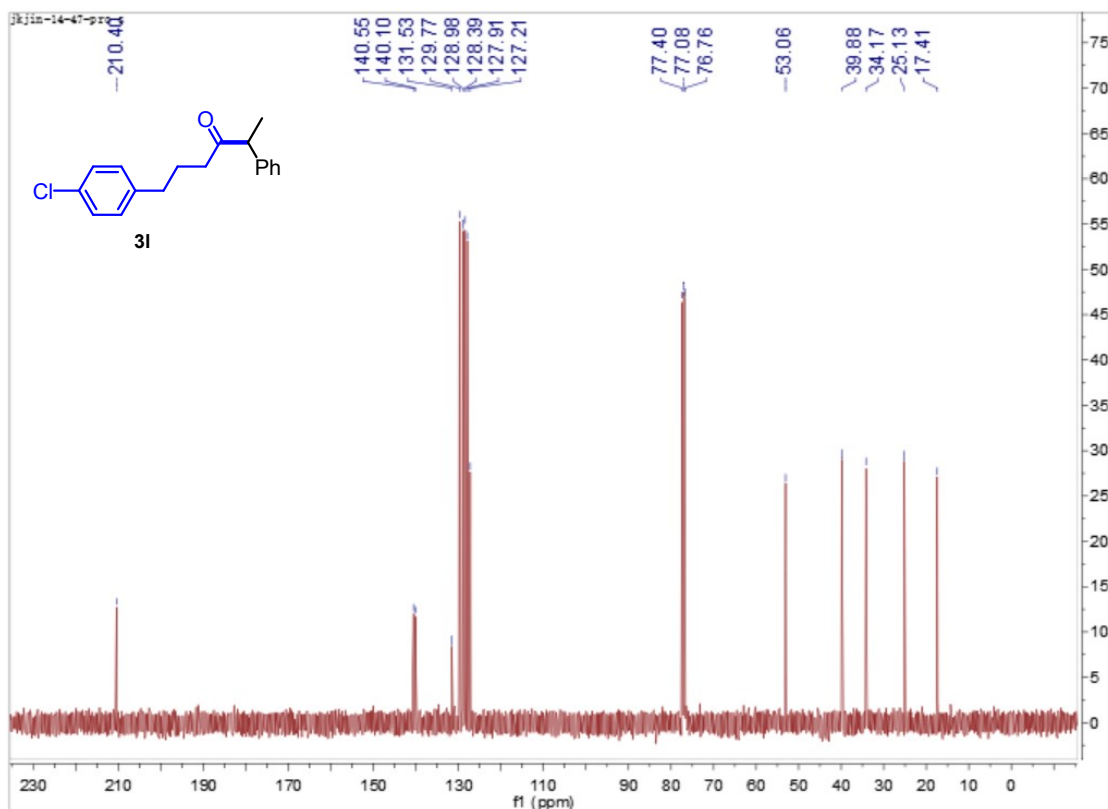
### <sup>13</sup>C NMR Spectrum of 3k (CDCl<sub>3</sub>, 101 MHz)



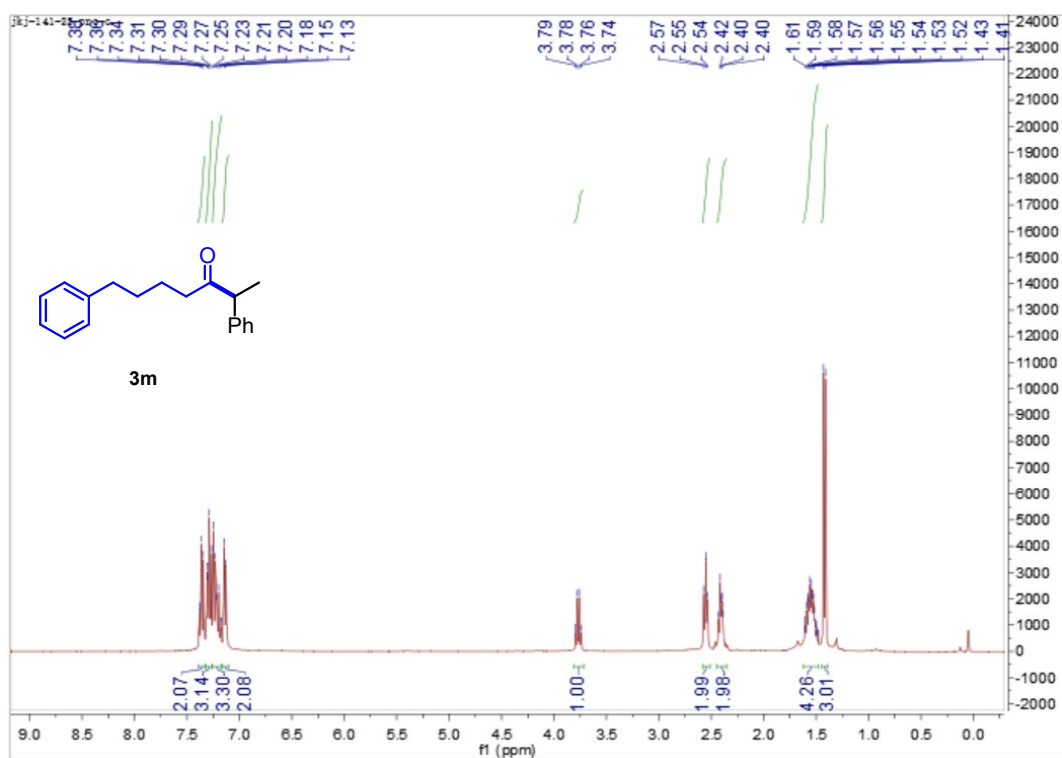
### <sup>1</sup>H NMR Spectrum of 3l (CDCl<sub>3</sub>, 400 MHz)



### <sup>13</sup>C NMR Spectrum of 3l (CDCl<sub>3</sub>, 101 MHz)

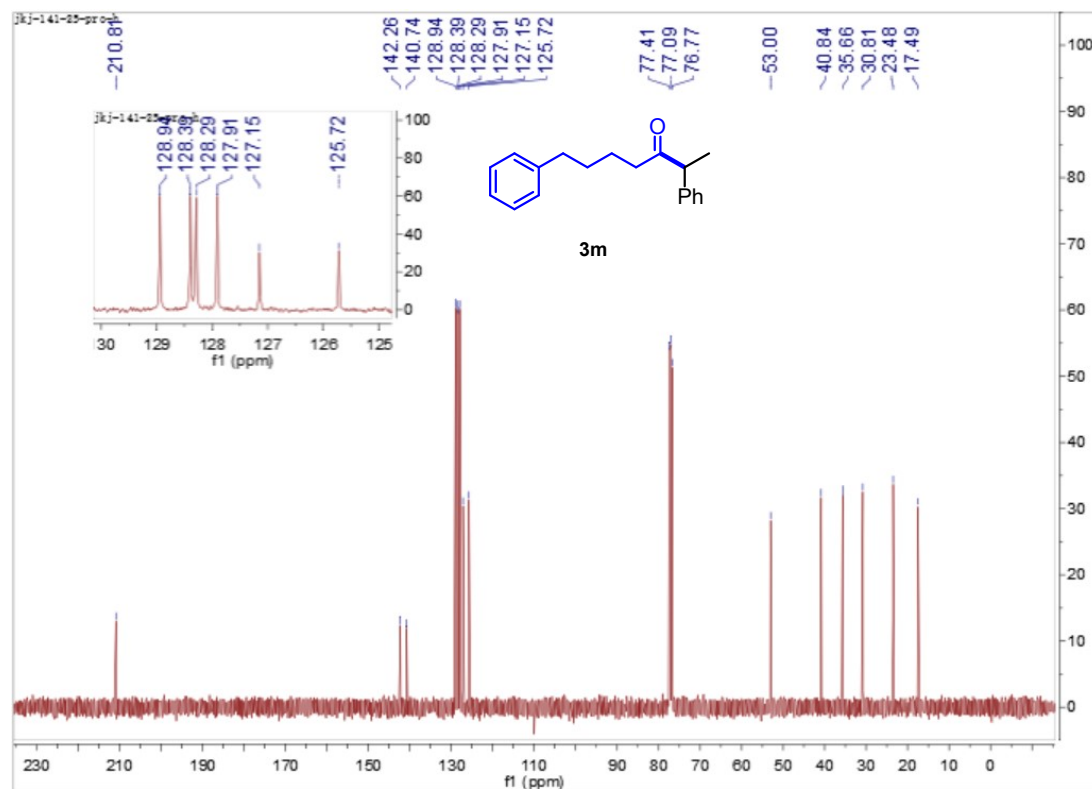


### <sup>1</sup>H NMR Spectrum of 3m (CDCl<sub>3</sub>, 400 MHz)

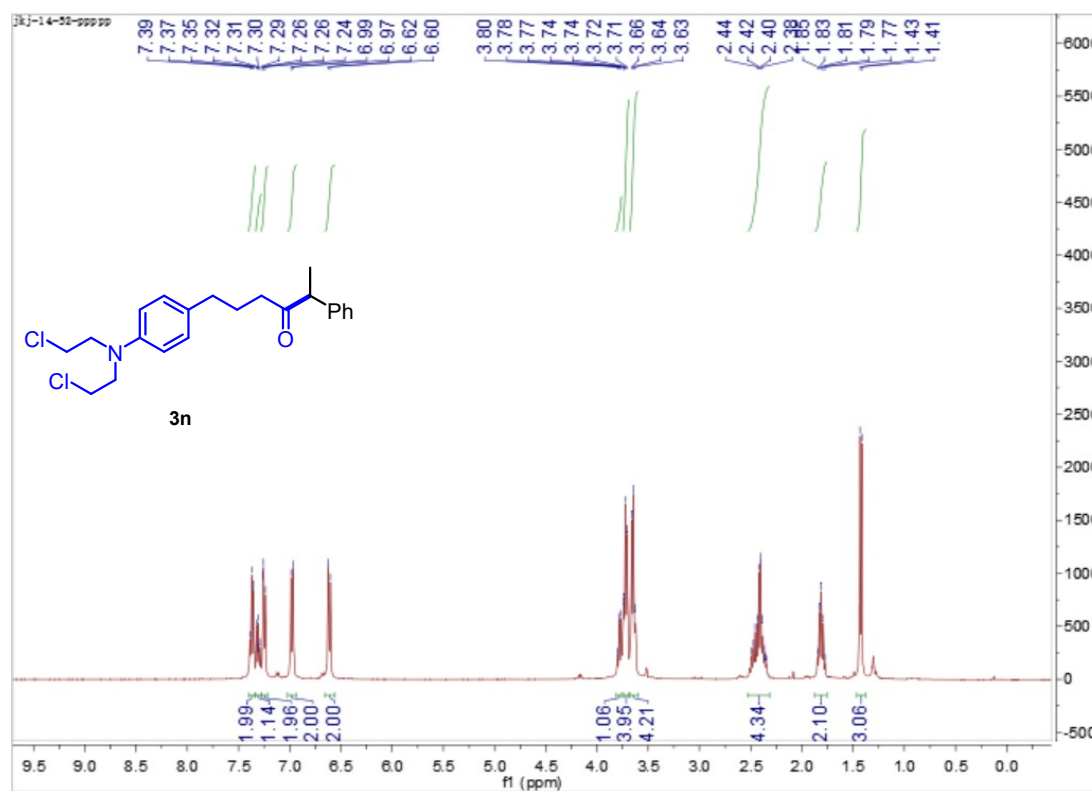




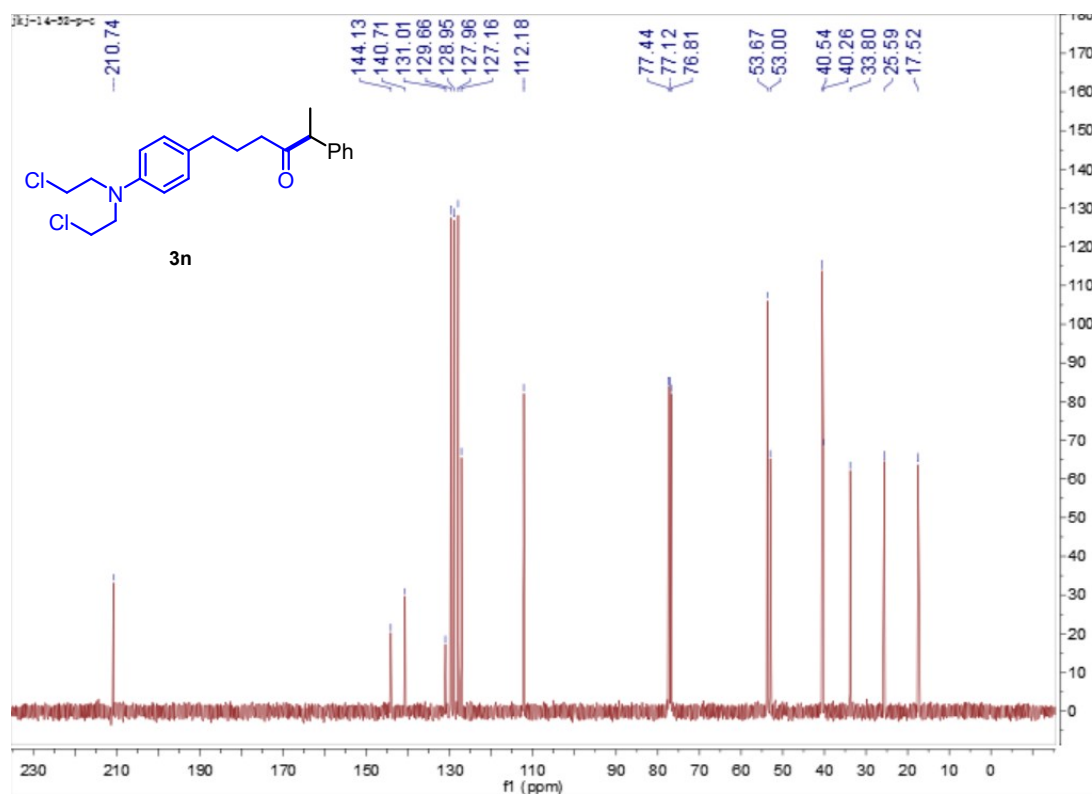
### <sup>13</sup>C NMR Spectrum of 3m (CDCl<sub>3</sub>, 101 MHz)



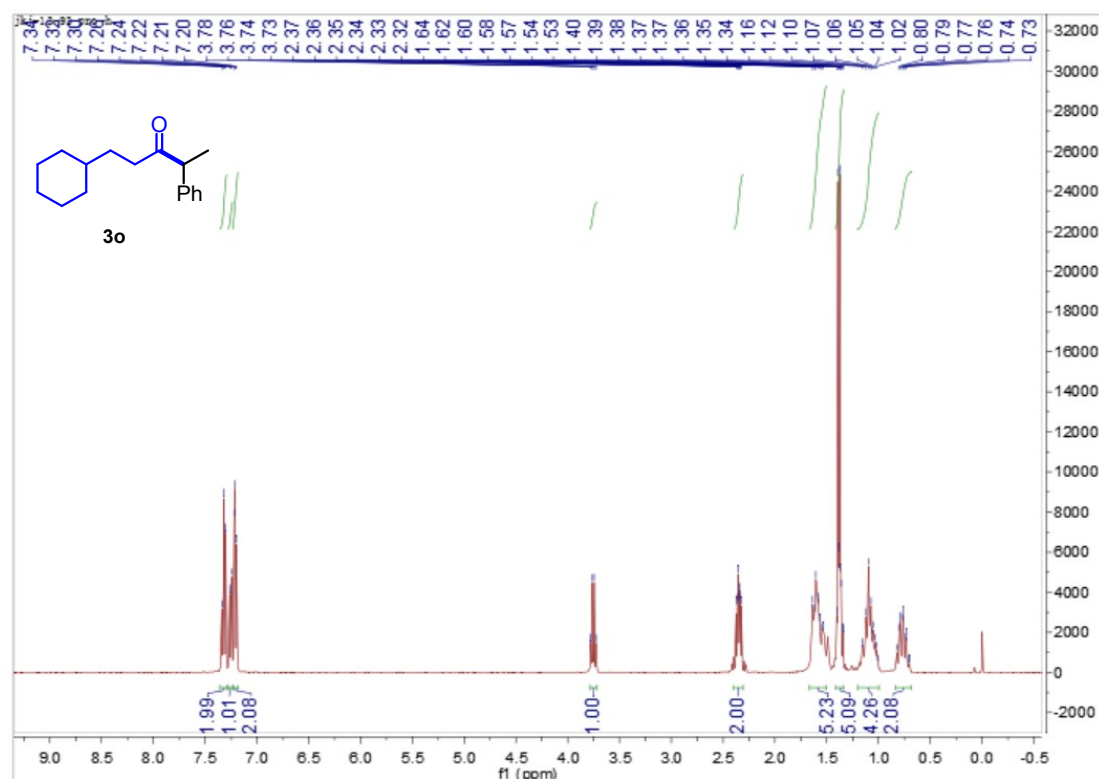
### <sup>1</sup>H NMR Spectrum of 3n (CDCl<sub>3</sub>, 400 MHz)



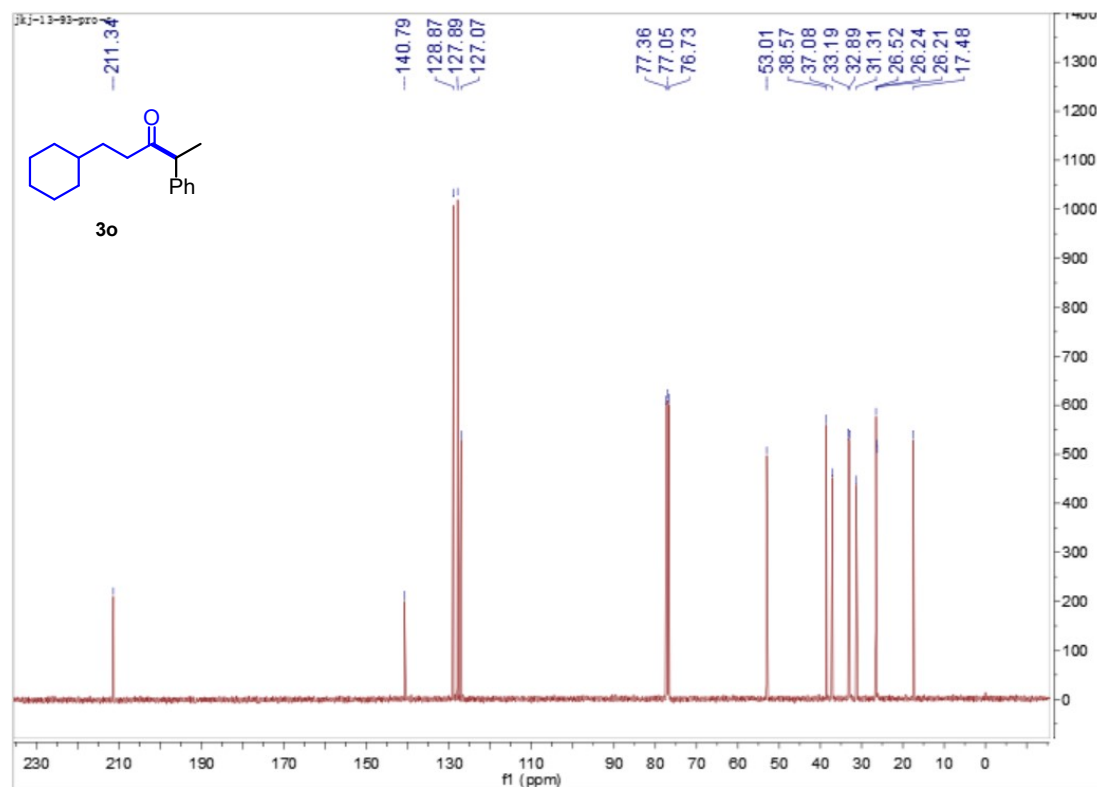
### <sup>13</sup>C NMR Spectrum of 3n (CDCl<sub>3</sub>, 101 MHz)



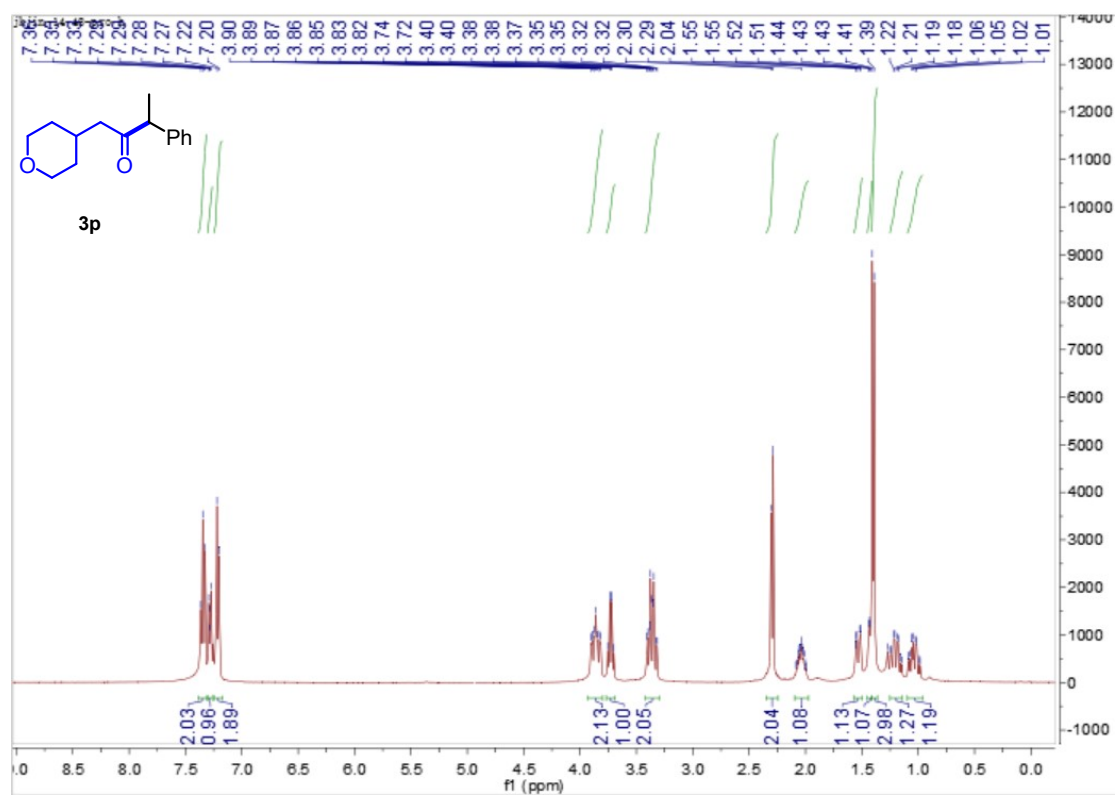
### <sup>1</sup>H NMR Spectrum of 3o (CDCl<sub>3</sub>, 400 MHz)



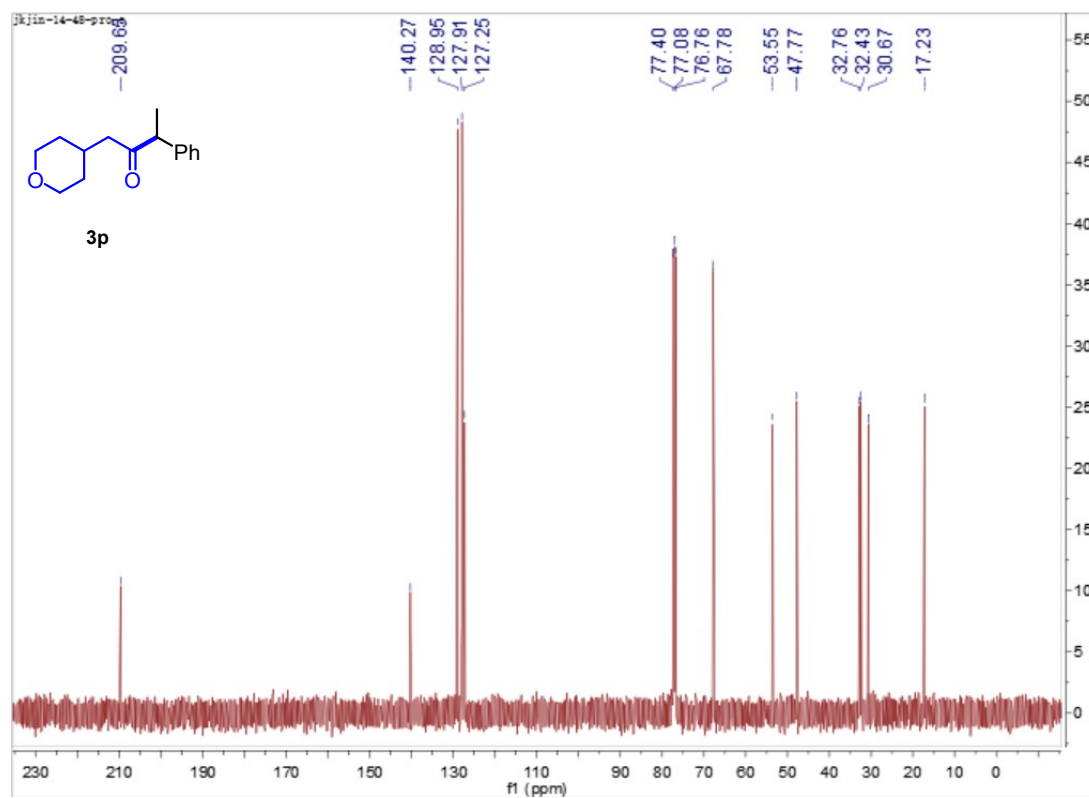
### <sup>13</sup>C NMR Spectrum of 3o (CDCl<sub>3</sub>, 101 MHz)



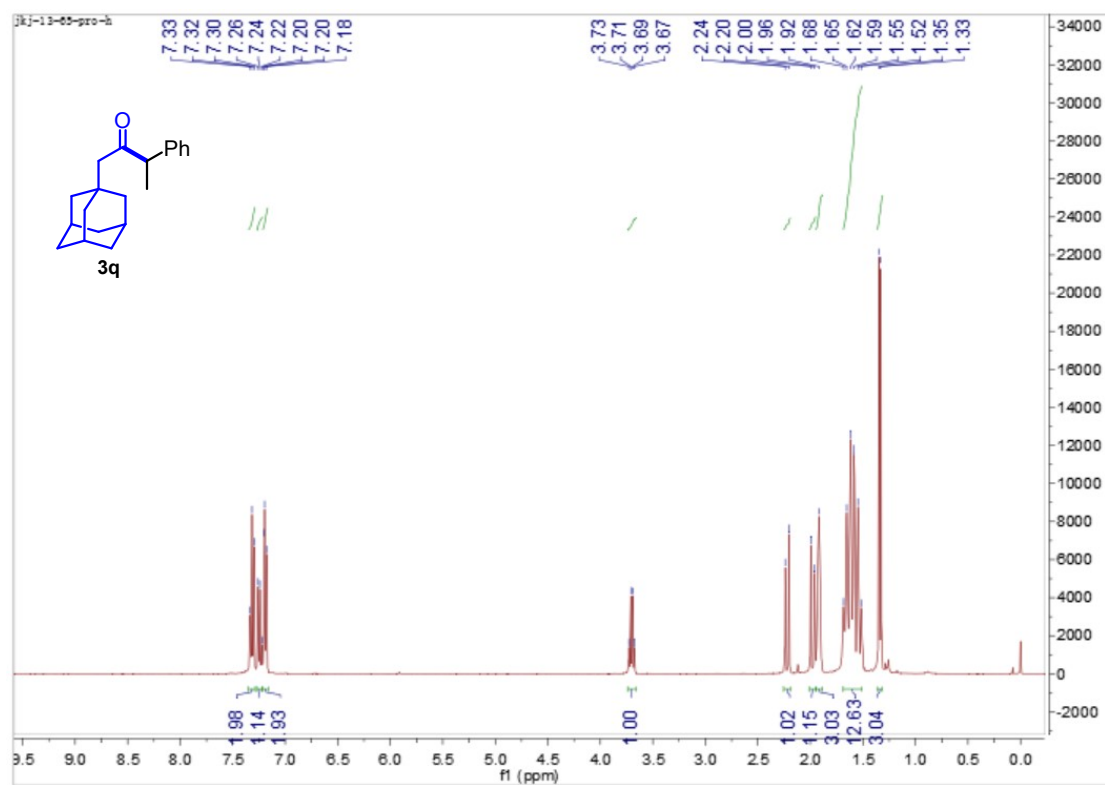
### <sup>1</sup>H NMR Spectrum of 3p (CDCl<sub>3</sub>, 400 MHz)



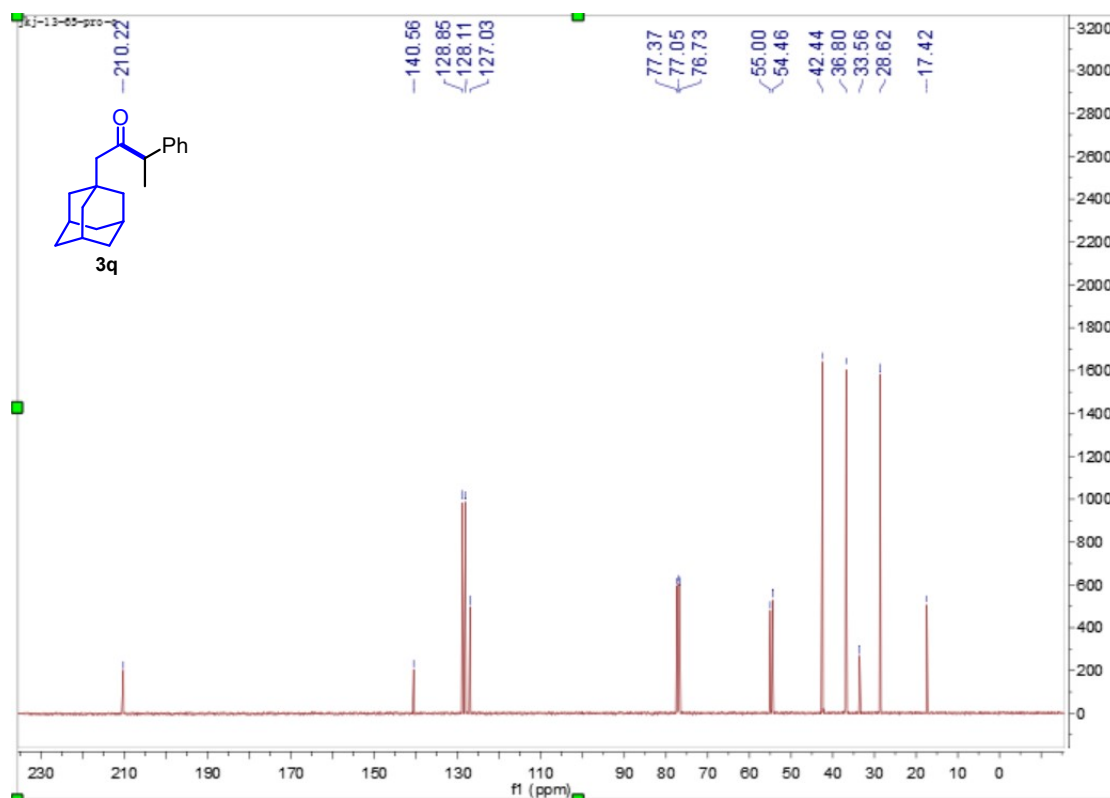
### <sup>13</sup>C NMR Spectrum of 3p (CDCl<sub>3</sub>, 101 MHz)



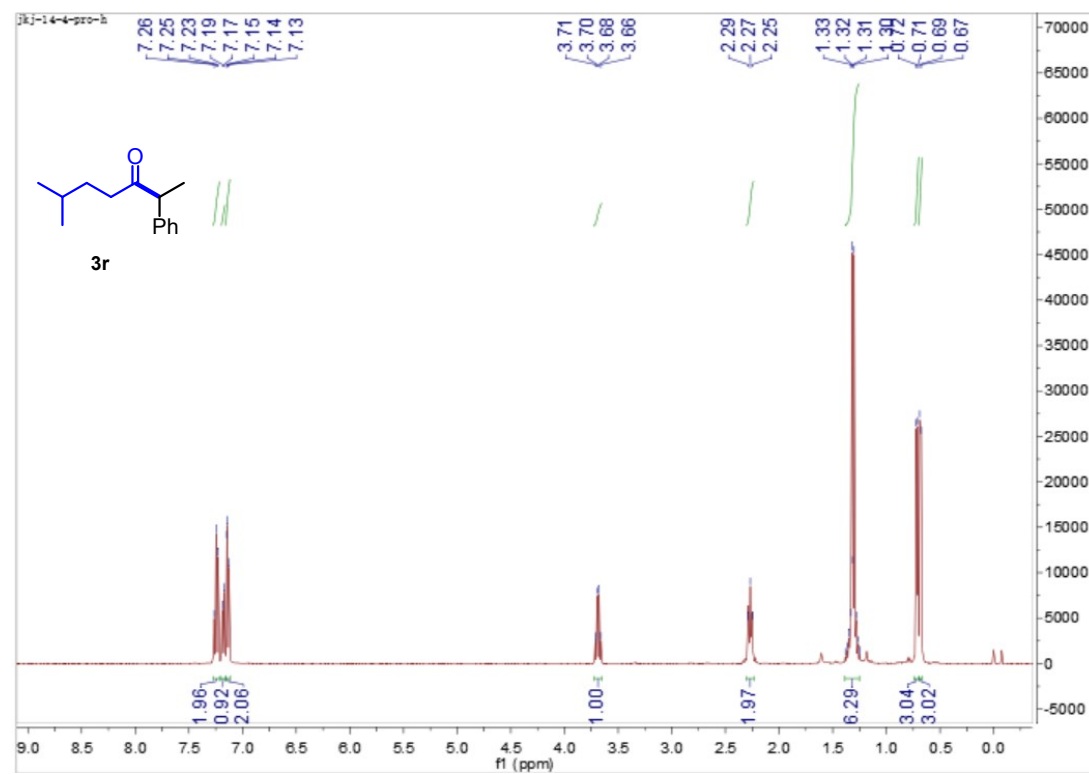
### <sup>1</sup>H NMR Spectrum of 3q (CDCl<sub>3</sub>, 400 MHz)



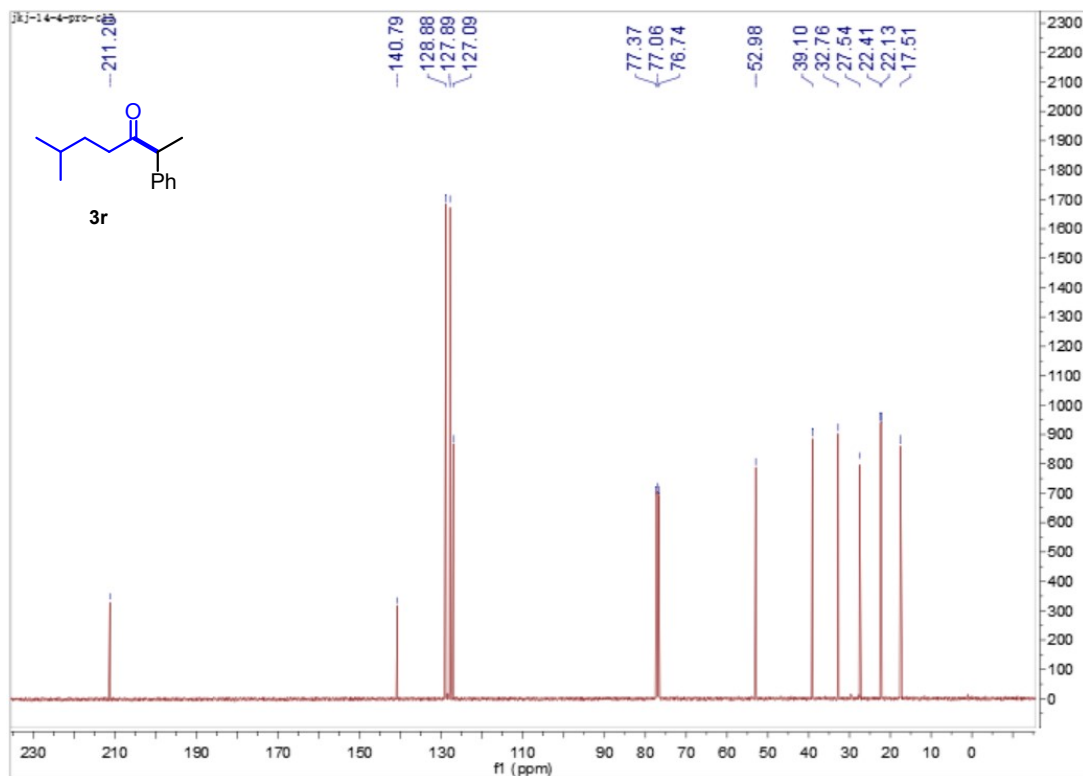
### <sup>13</sup>C NMR Spectrum of 3q (CDCl<sub>3</sub>, 101 MHz)



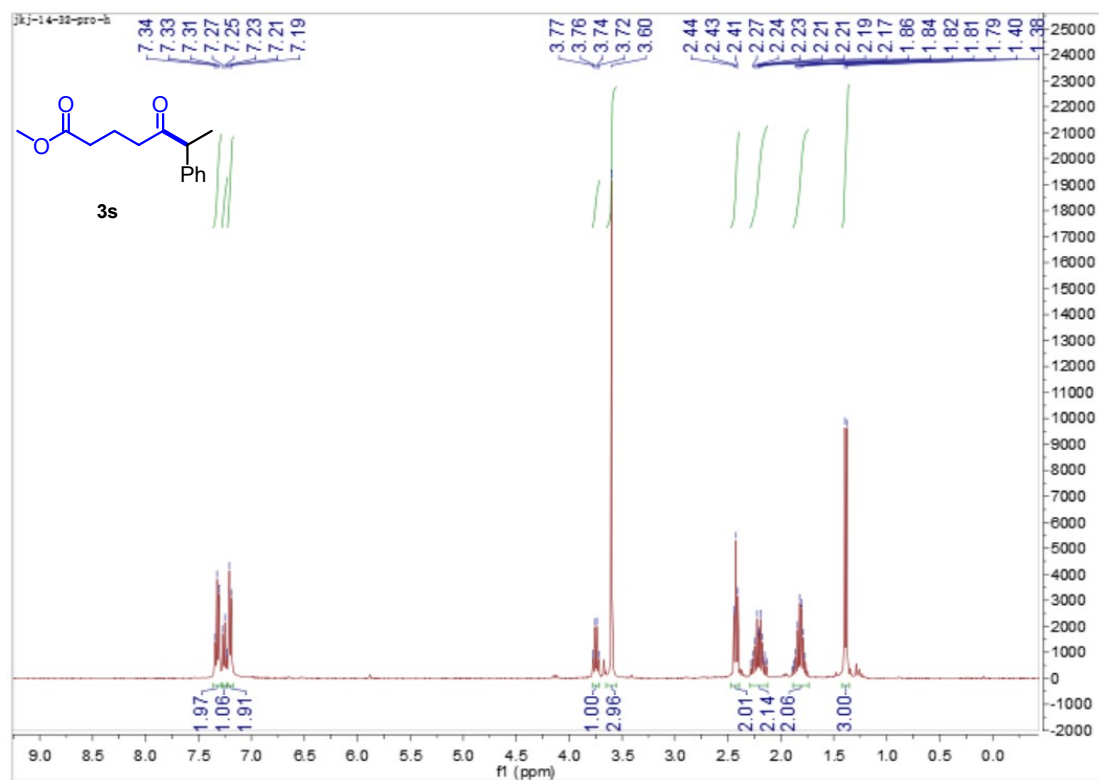
### <sup>1</sup>H NMR Spectrum of 3r (CDCl<sub>3</sub>, 400 MHz)



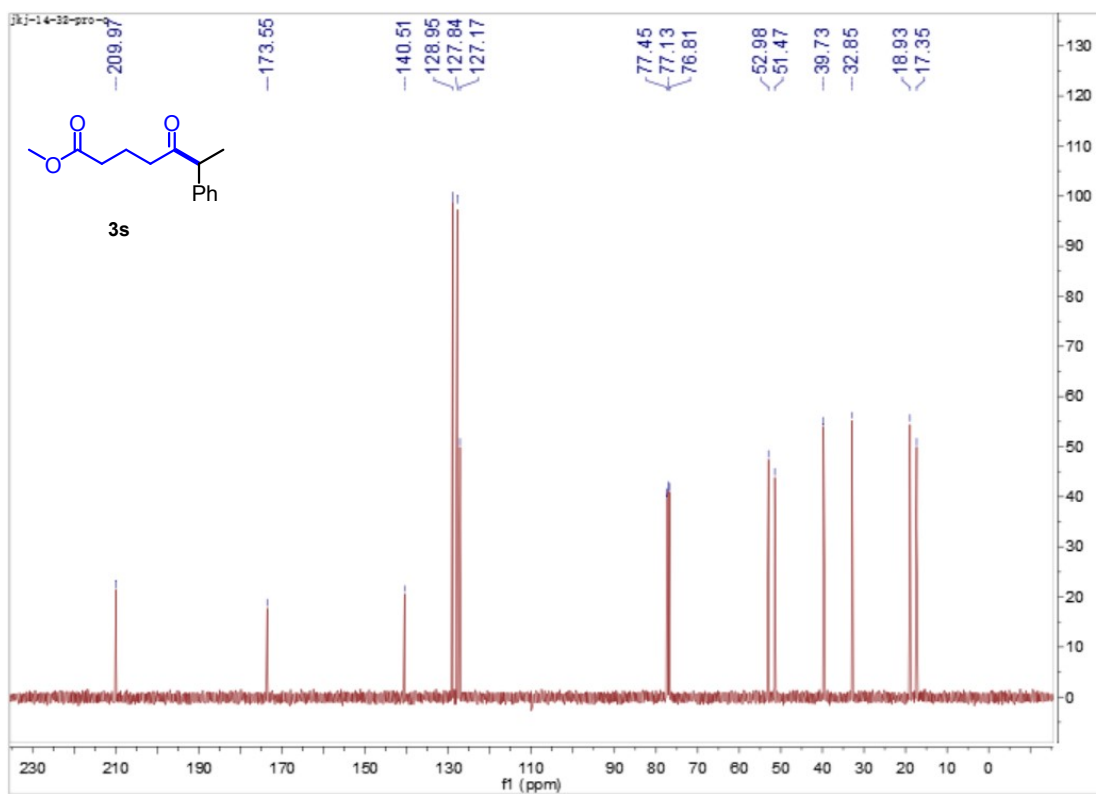
### <sup>13</sup>C NMR Spectrum of 3r (CDCl<sub>3</sub>, 101 MHz)



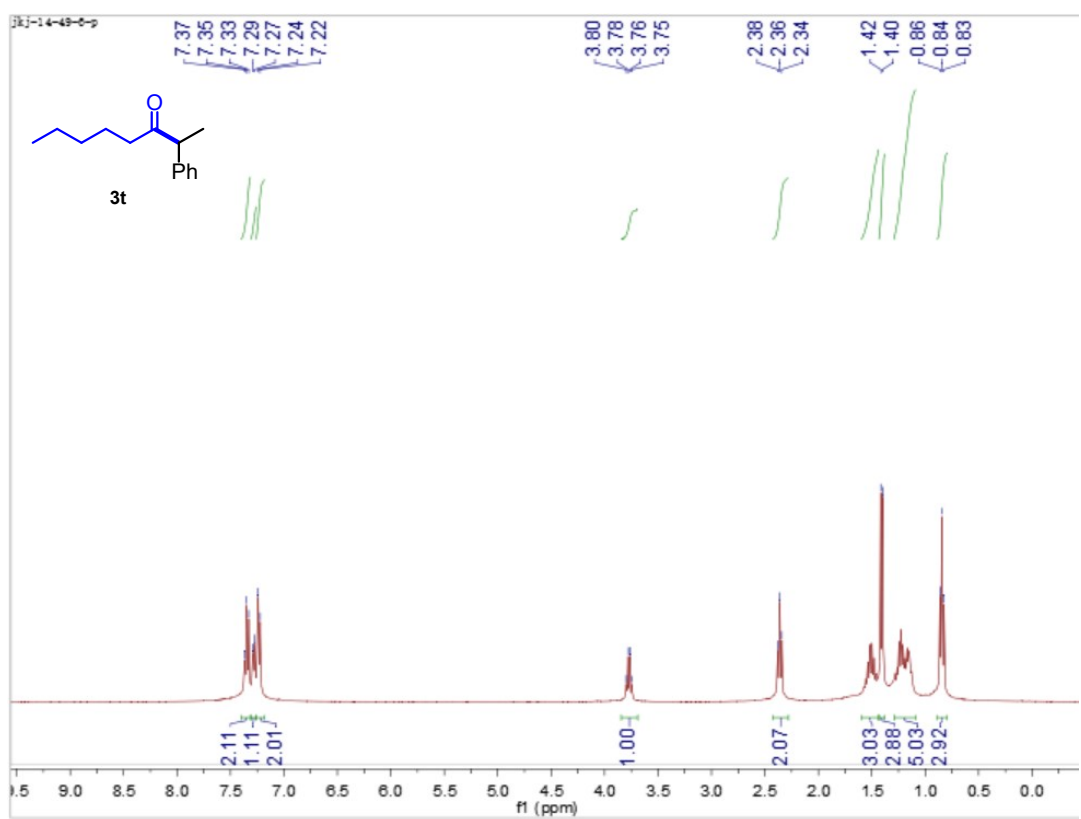
### <sup>1</sup>H NMR Spectrum of 3s (CDCl<sub>3</sub>, 400 MHz)



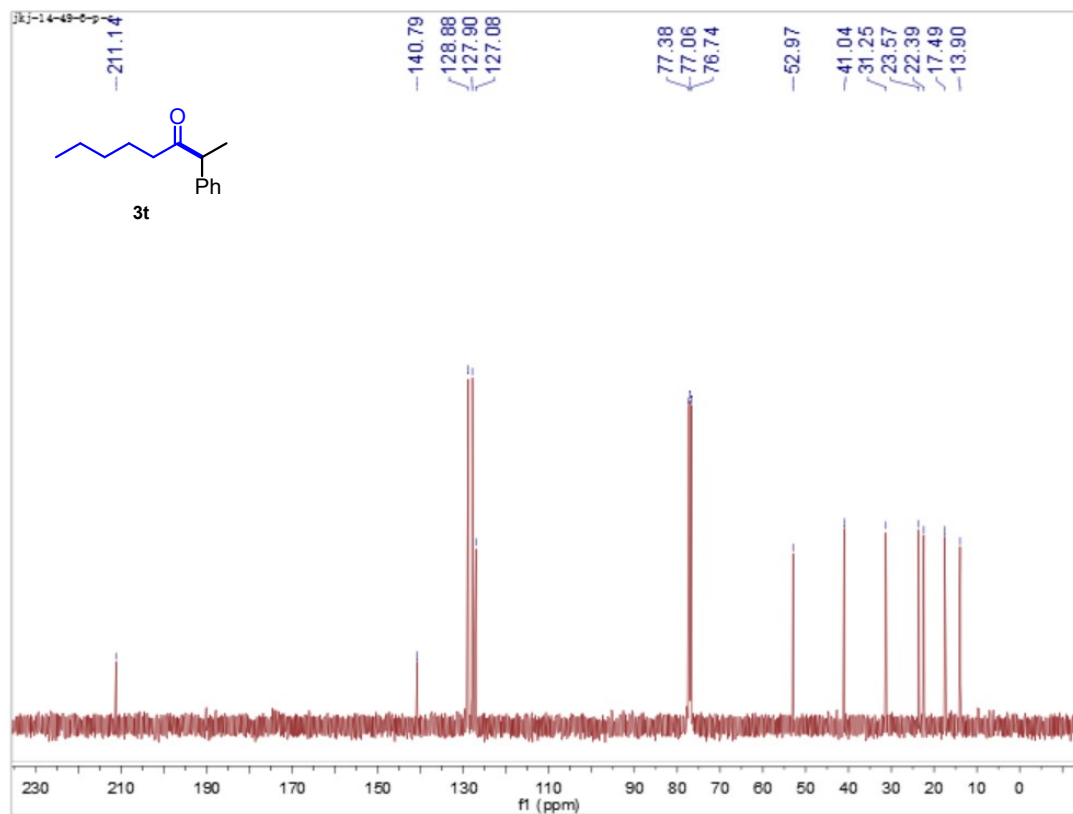
### <sup>13</sup>C NMR Spectrum of 3s (CDCl<sub>3</sub>, 101 MHz)



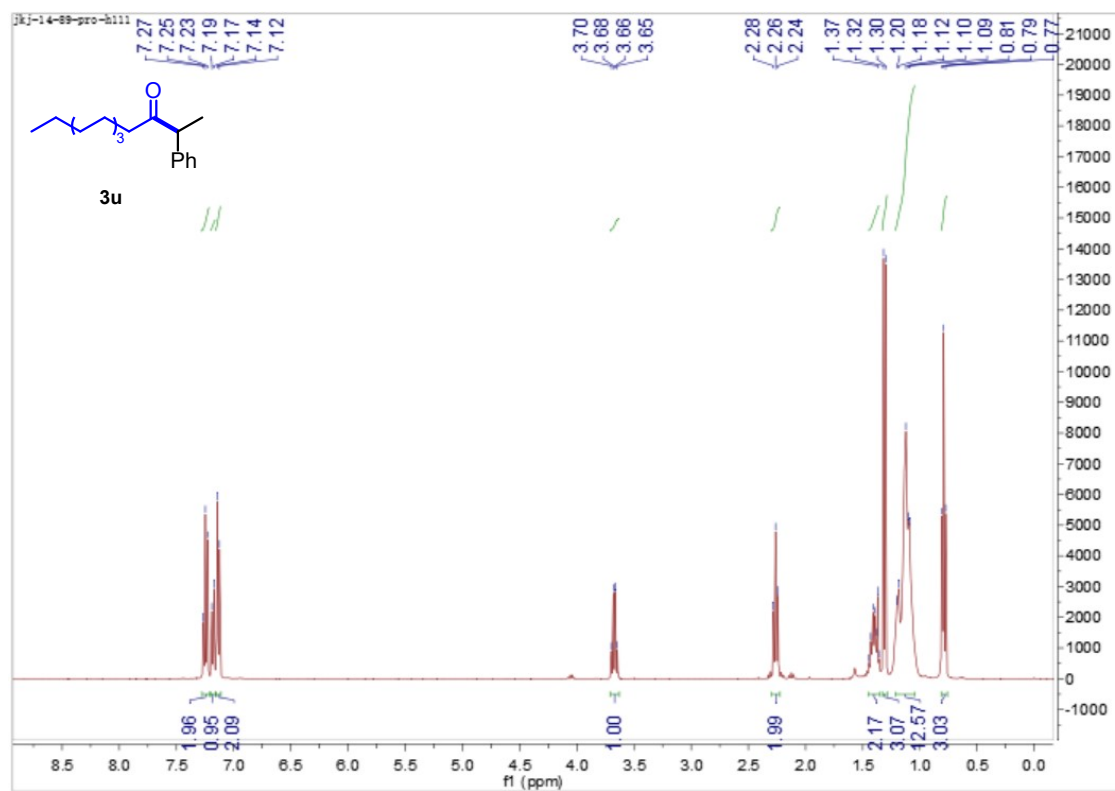
### <sup>1</sup>H NMR Spectrum of 3t (CDCl<sub>3</sub>, 400 MHz)



### <sup>13</sup>C NMR Spectrum of 3t (CDCl<sub>3</sub>, 101 MHz)

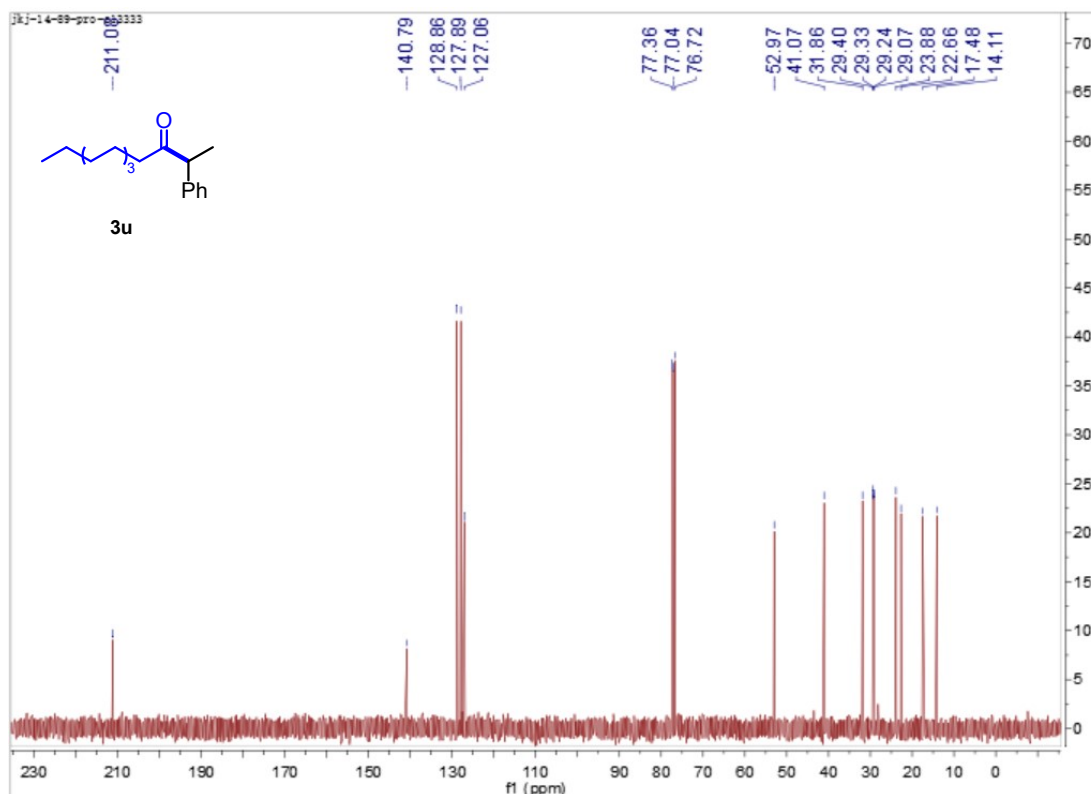


### <sup>1</sup>H NMR Spectrum of 3u (CDCl<sub>3</sub>, 400 MHz)

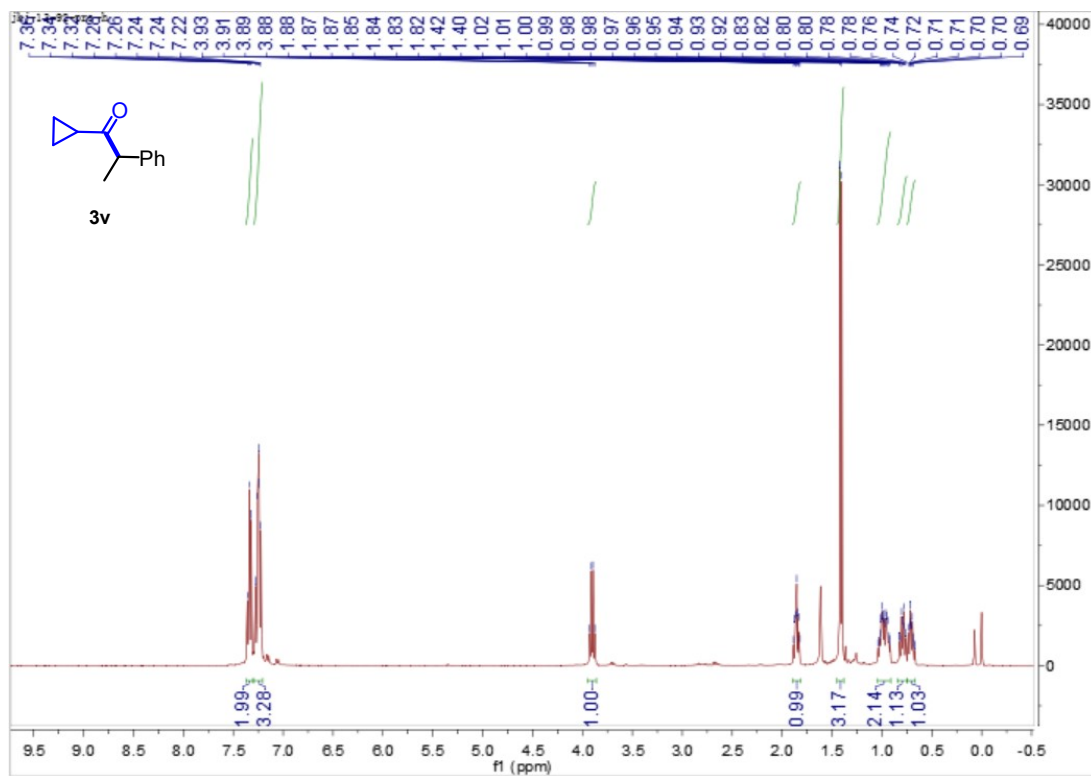




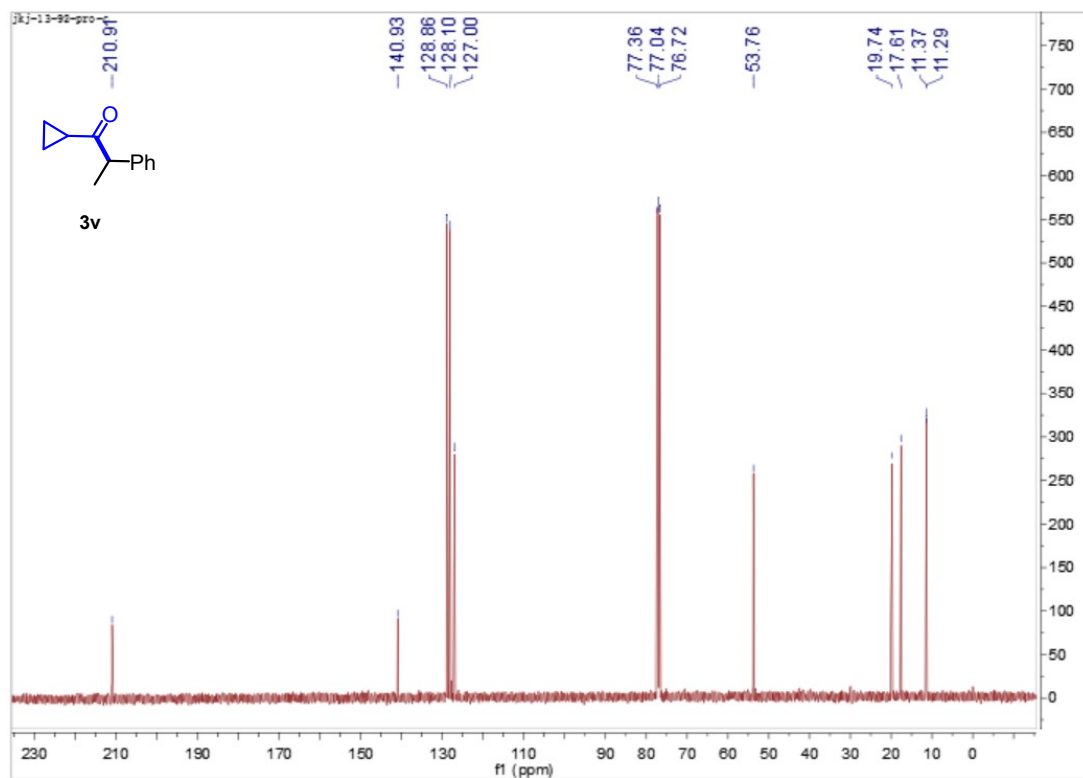
### <sup>13</sup>C NMR Spectrum of 3u (CDCl<sub>3</sub>, 101 MHz)



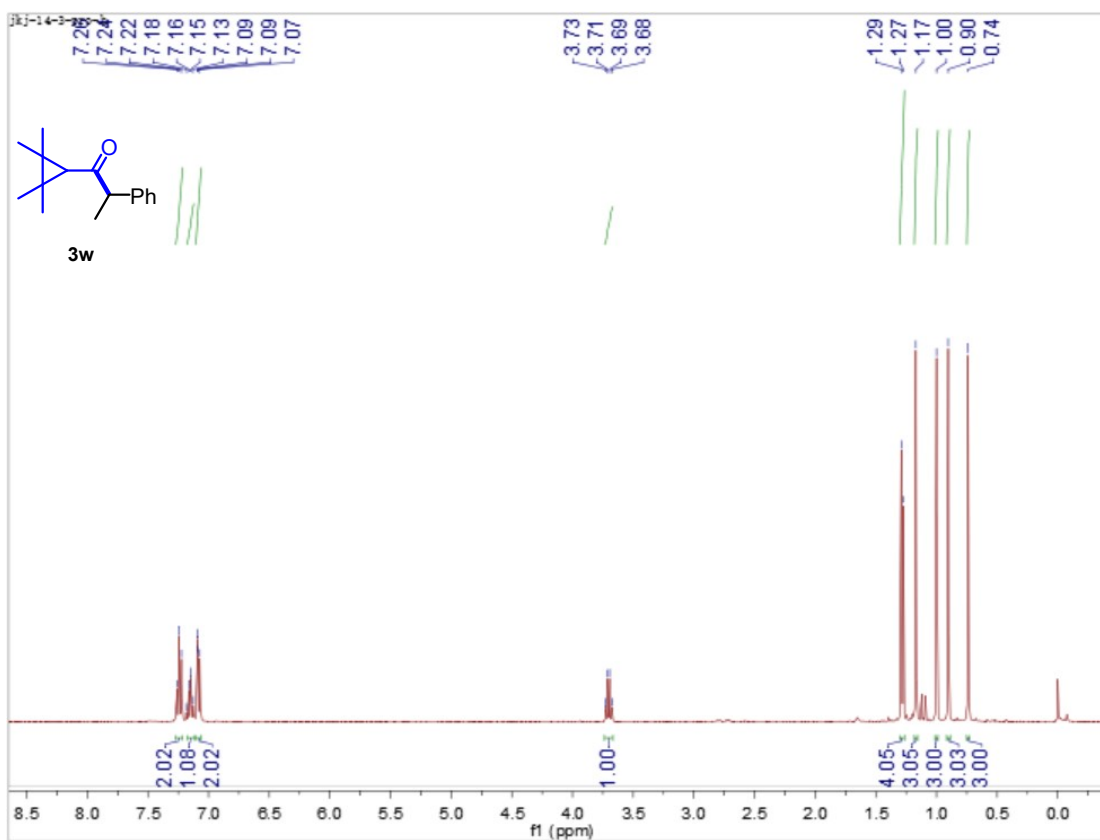
### <sup>1</sup>H NMR Spectrum of 3v (CDCl<sub>3</sub>, 400 MHz)



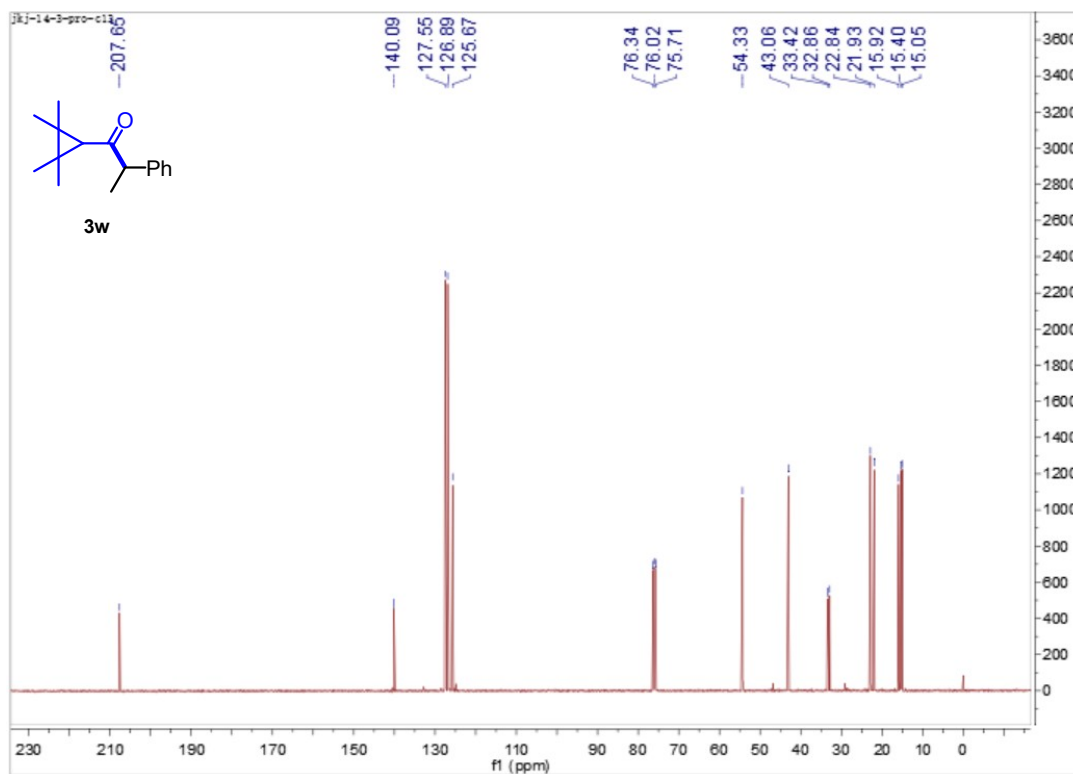
### <sup>13</sup>C NMR Spectrum of 3v (CDCl<sub>3</sub>, 101 MHz)



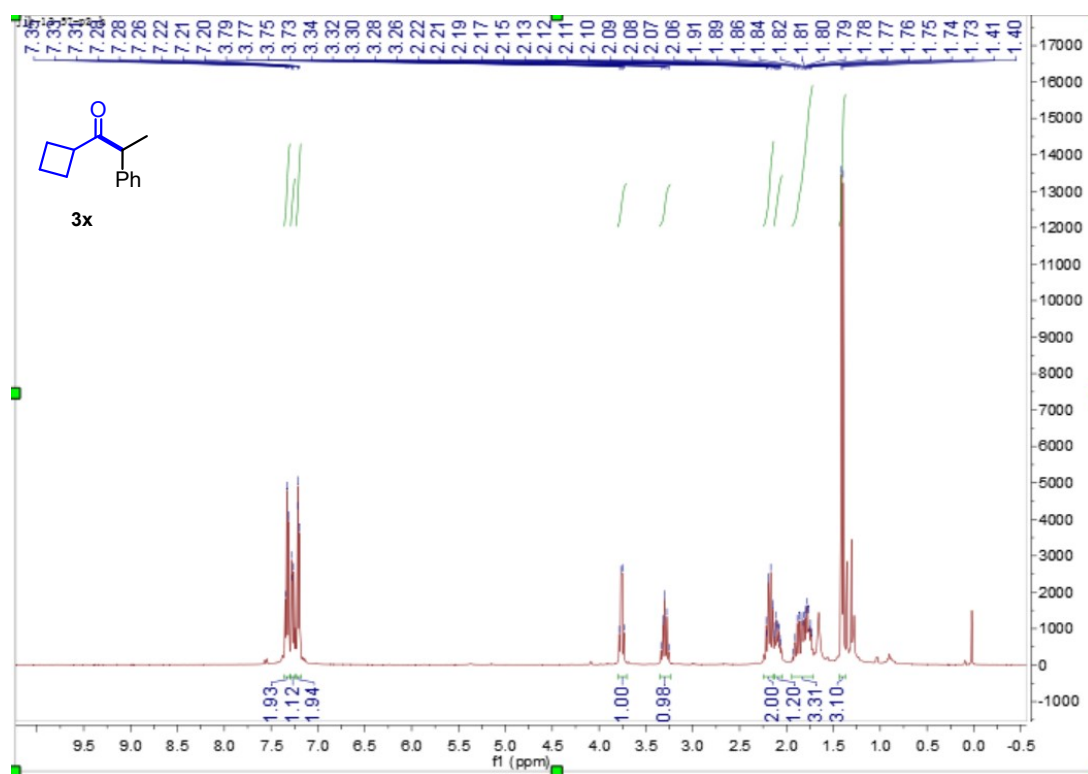
### <sup>1</sup>H NMR Spectrum of 3w (CDCl<sub>3</sub>, 400 MHz)



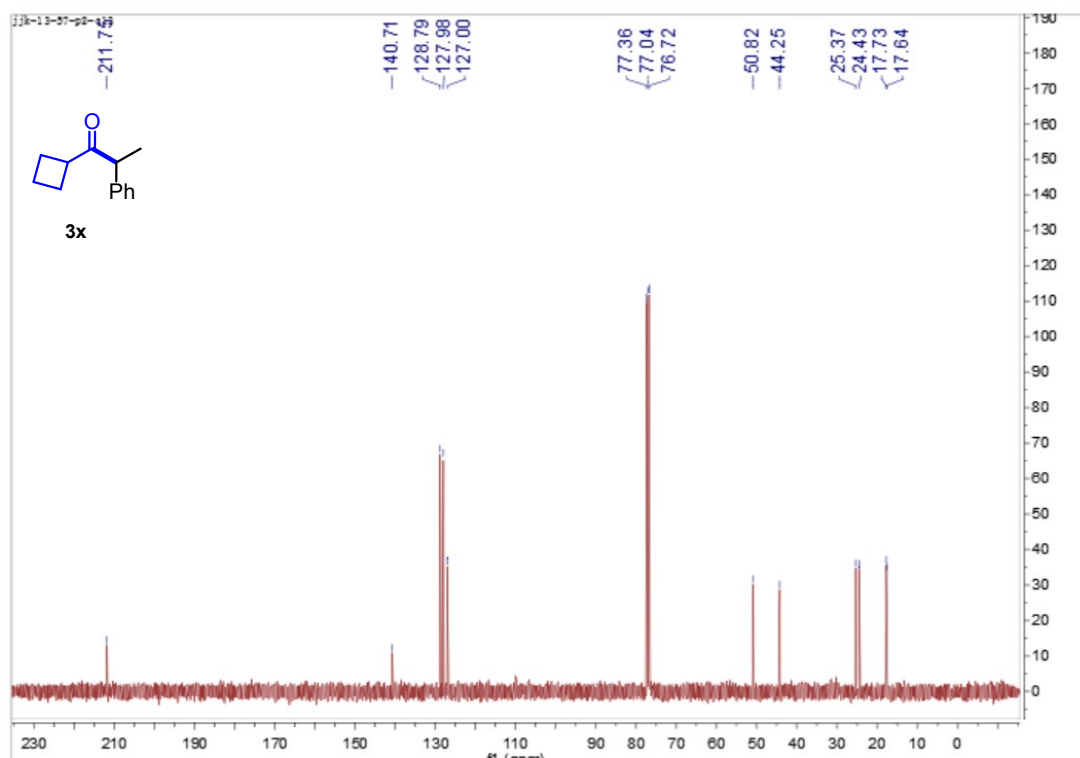
### <sup>13</sup>C NMR Spectrum of 3w (CDCl<sub>3</sub>, 101 MHz)



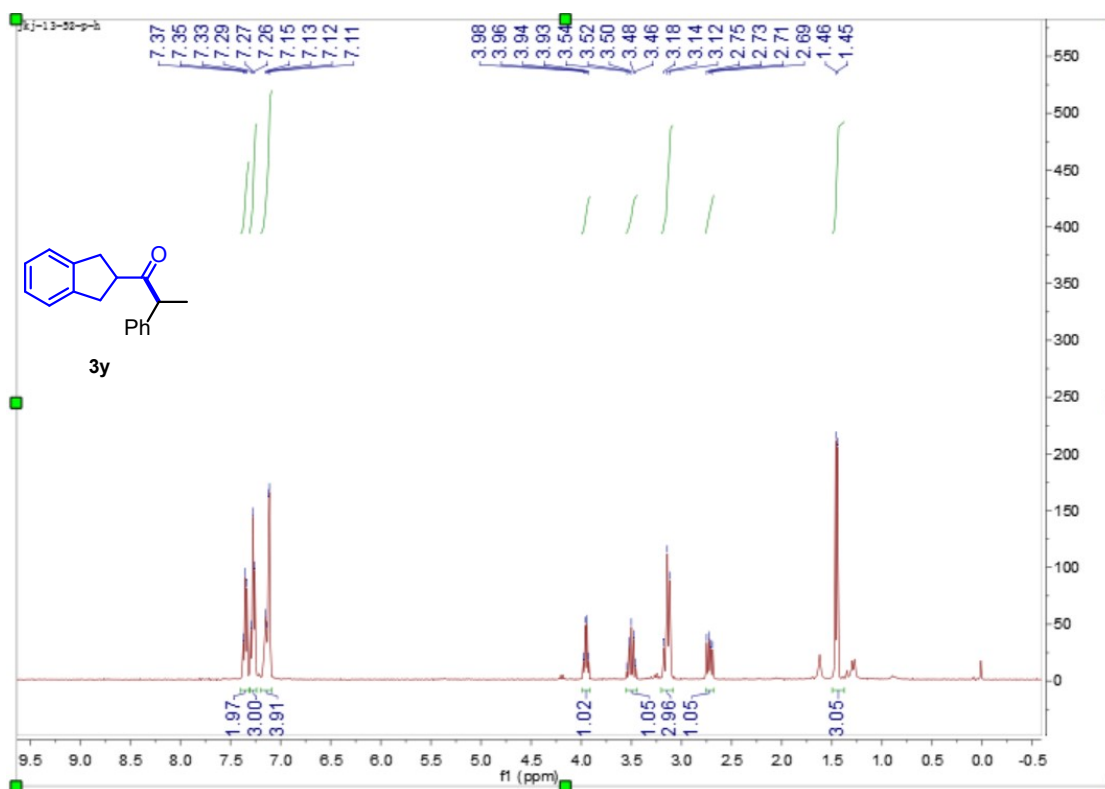
### <sup>1</sup>H NMR Spectrum of 3x (CDCl<sub>3</sub>, 400 MHz)



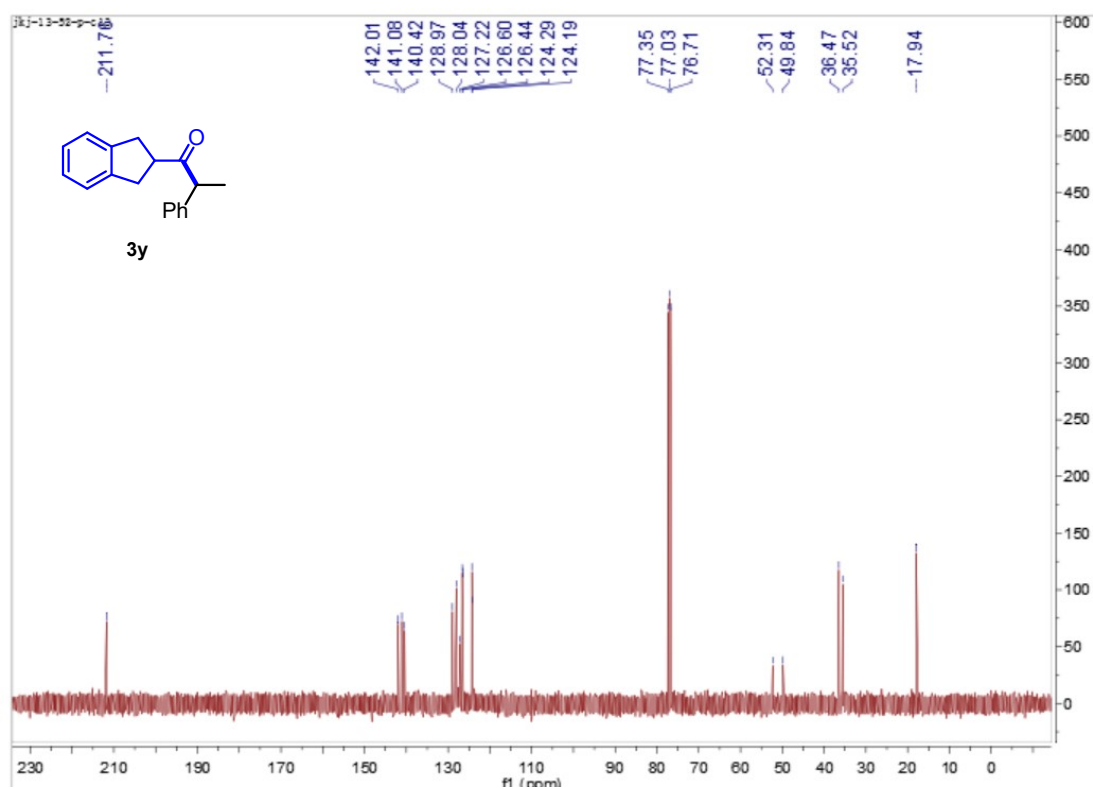
### <sup>13</sup>C NMR Spectrum of 3x (CDCl<sub>3</sub>, 101 MHz)



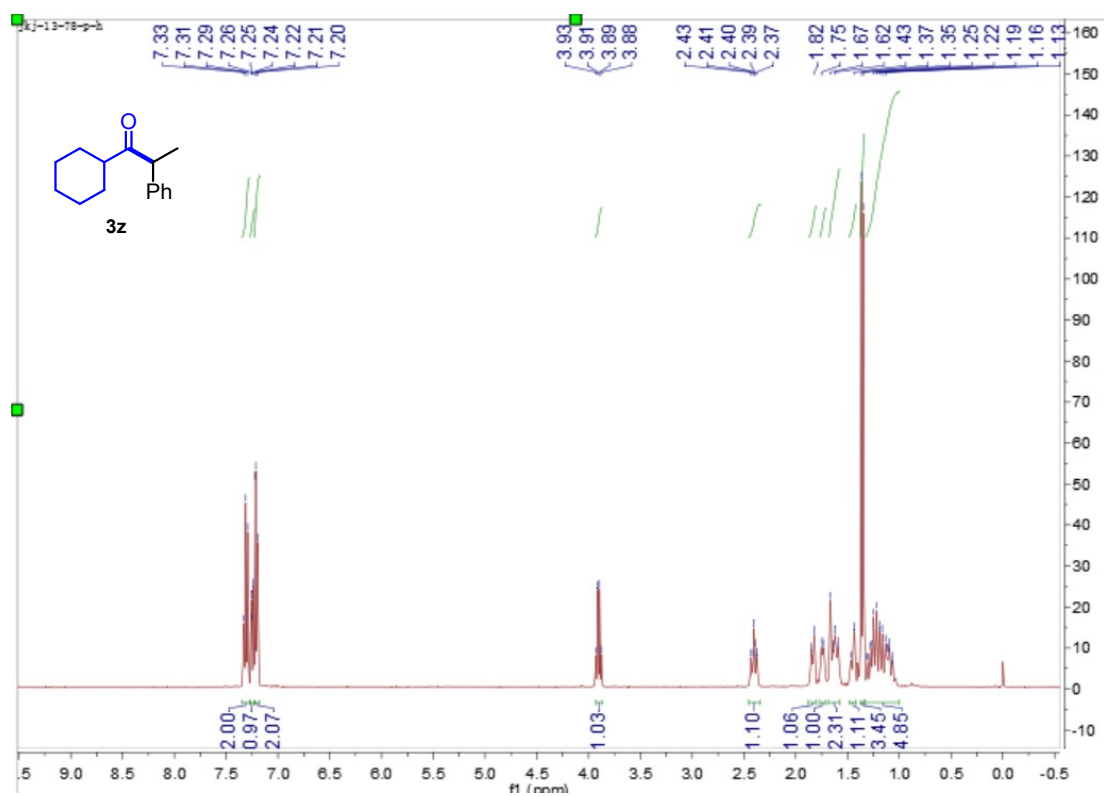
### <sup>1</sup>H NMR Spectrum of 3y (CDCl<sub>3</sub>, 400 MHz)



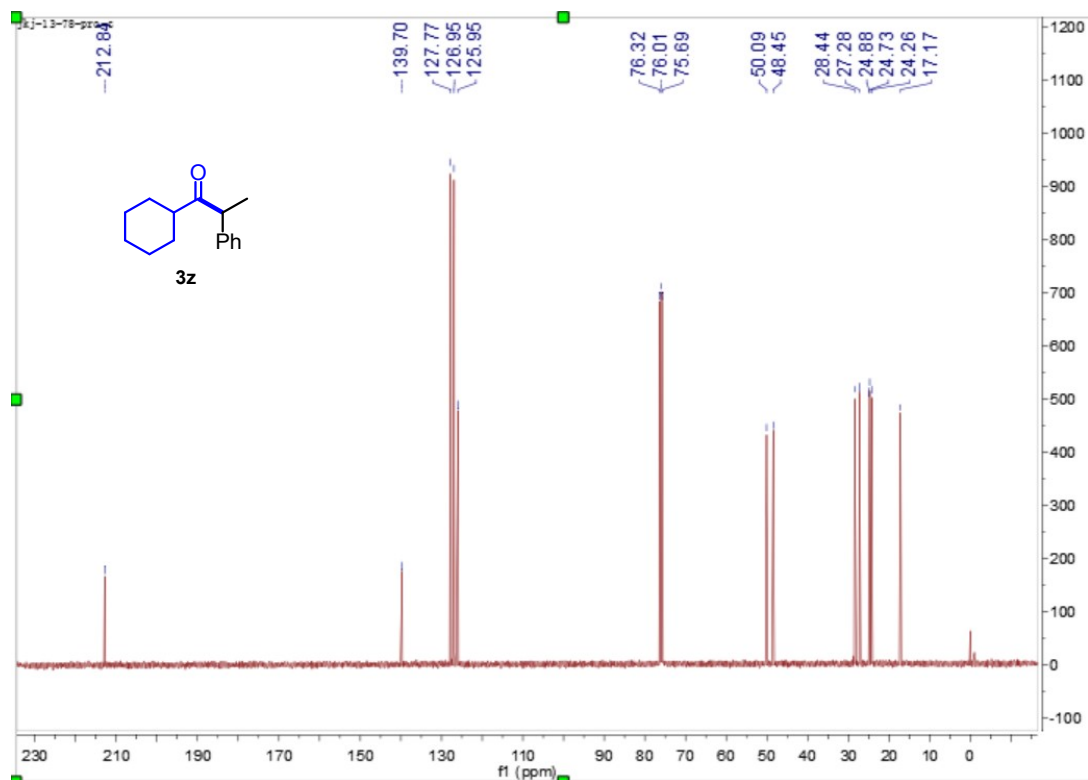
### <sup>13</sup>C NMR Spectrum of 3y (CDCl<sub>3</sub>, 101 MHz)



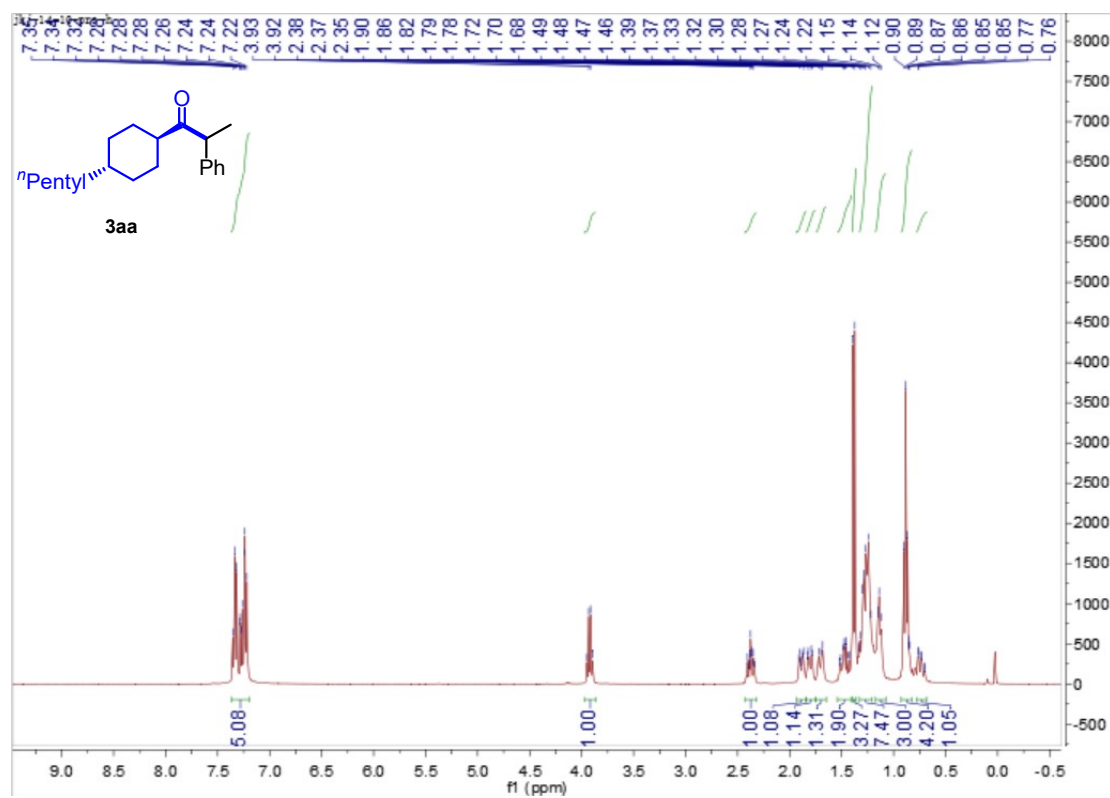
### <sup>1</sup>H NMR Spectrum of 3z (CDCl<sub>3</sub>, 400 MHz)



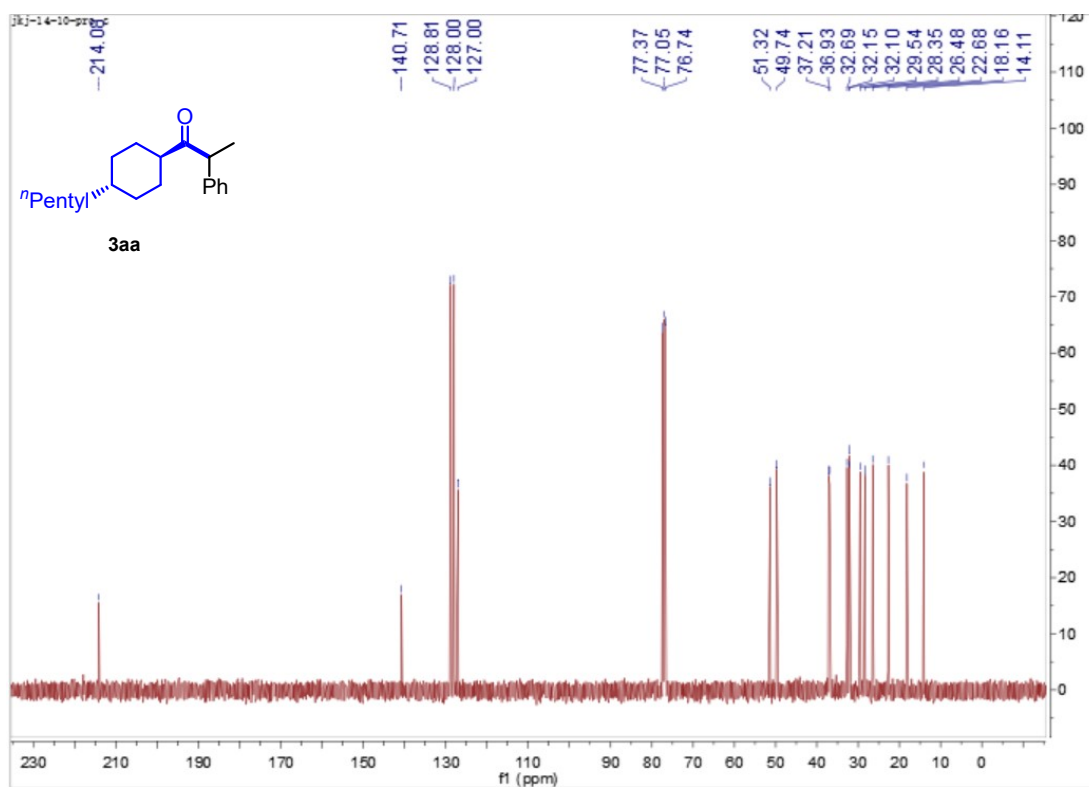
### $^{13}\text{C}$ NMR Spectrum of 3z ( $\text{CDCl}_3$ , 101 MHz)



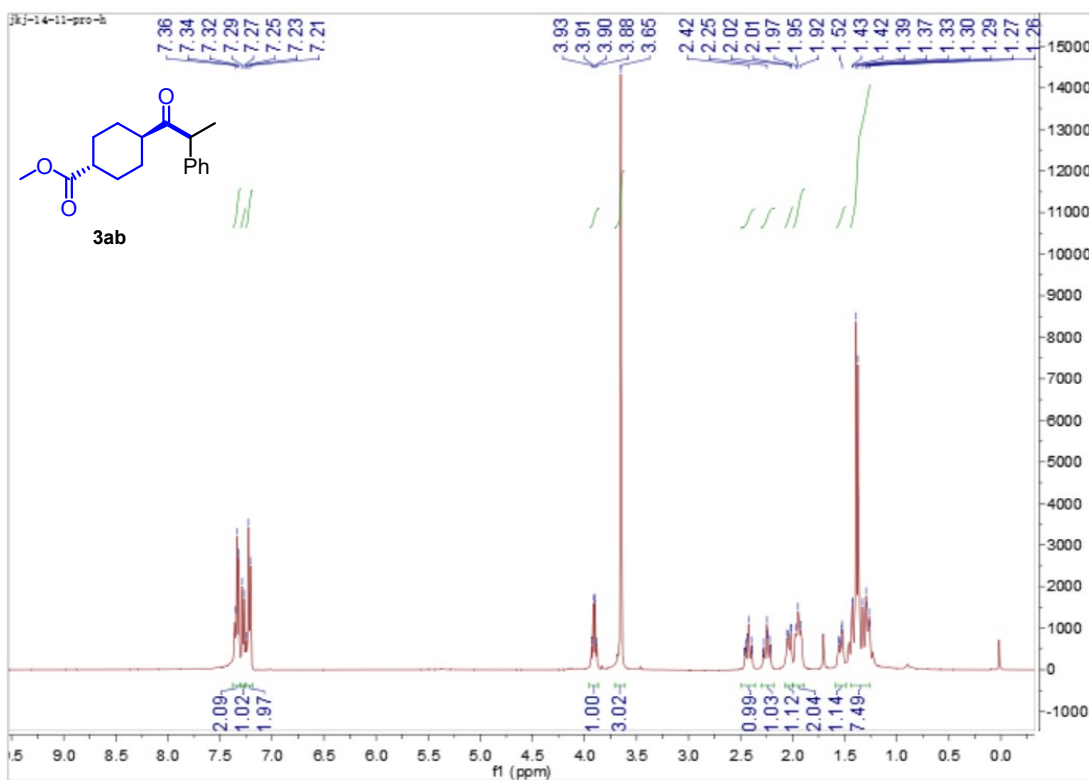
### $^1\text{H}$ NMR Spectrum of 3aa ( $\text{CDCl}_3$ , 400 MHz)



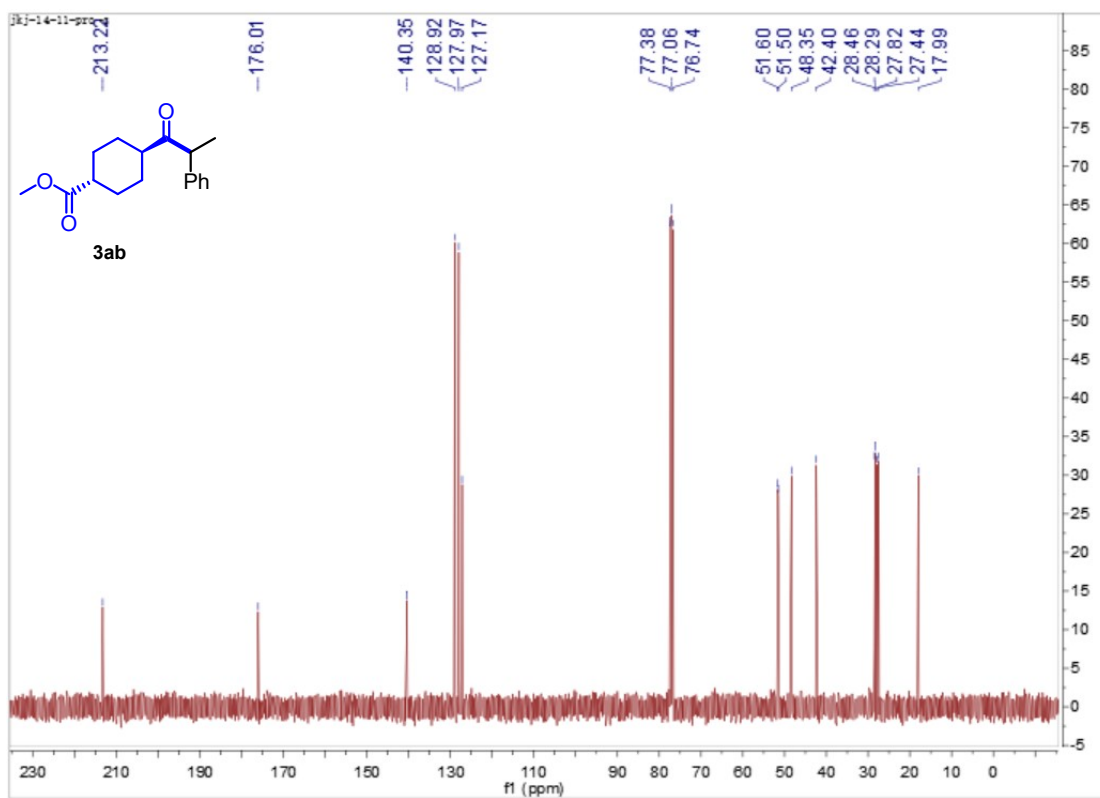
### <sup>13</sup>C NMR Spectrum of 3aa (CDCl<sub>3</sub>, 101 MHz)



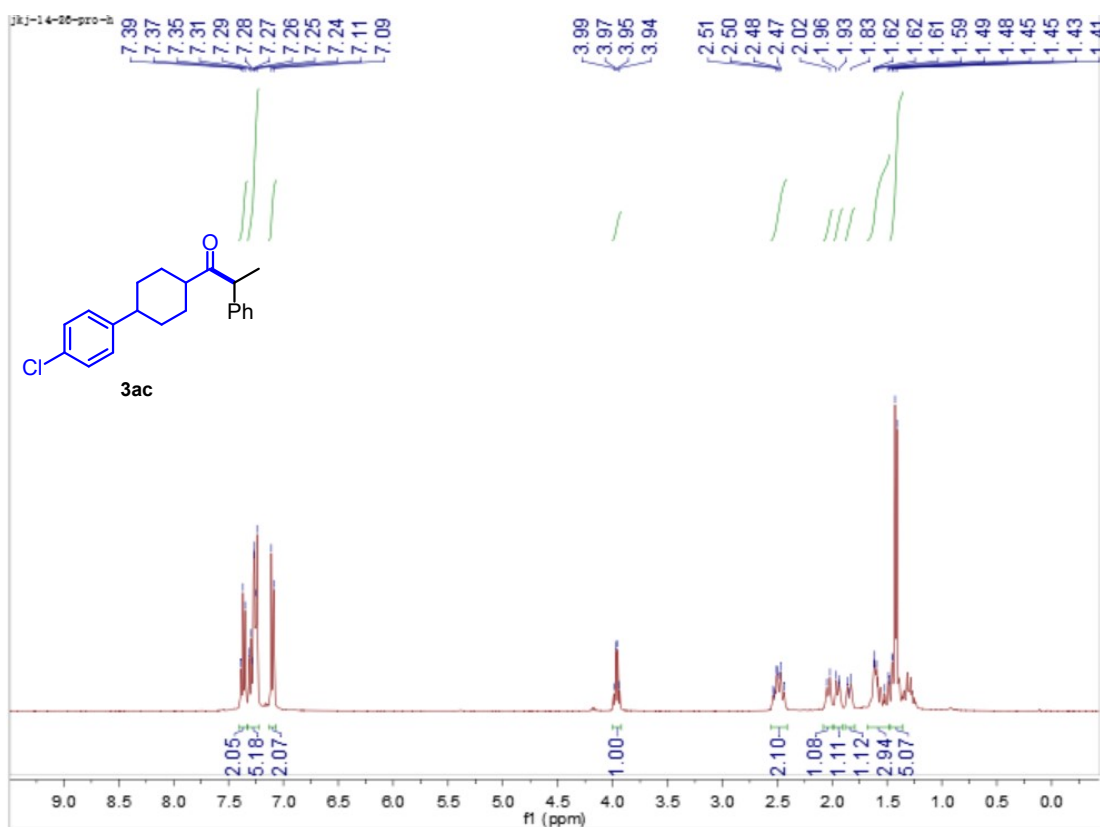
### <sup>1</sup>H NMR Spectrum of 3ab (CDCl<sub>3</sub>, 400 MHz)



### <sup>13</sup>C NMR Spectrum of 3ab (CDCl<sub>3</sub>, 101 MHz)

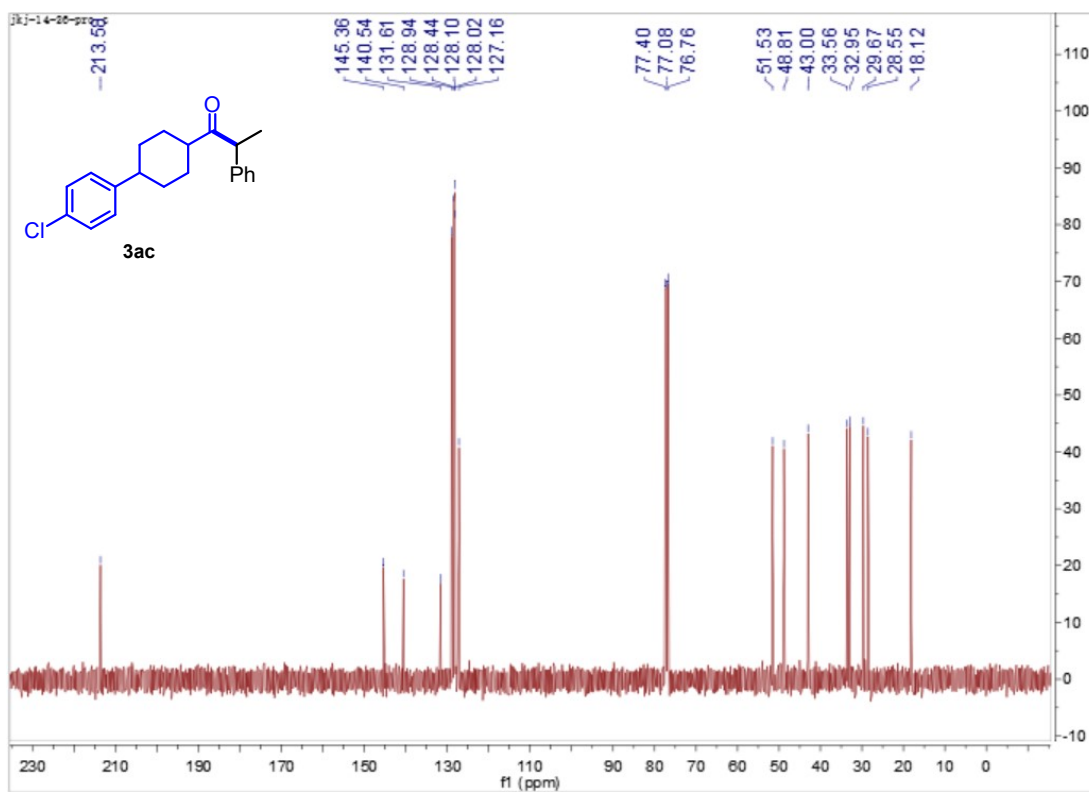


### <sup>1</sup>H NMR Spectrum of 3ac (CDCl<sub>3</sub>, 400 MHz)

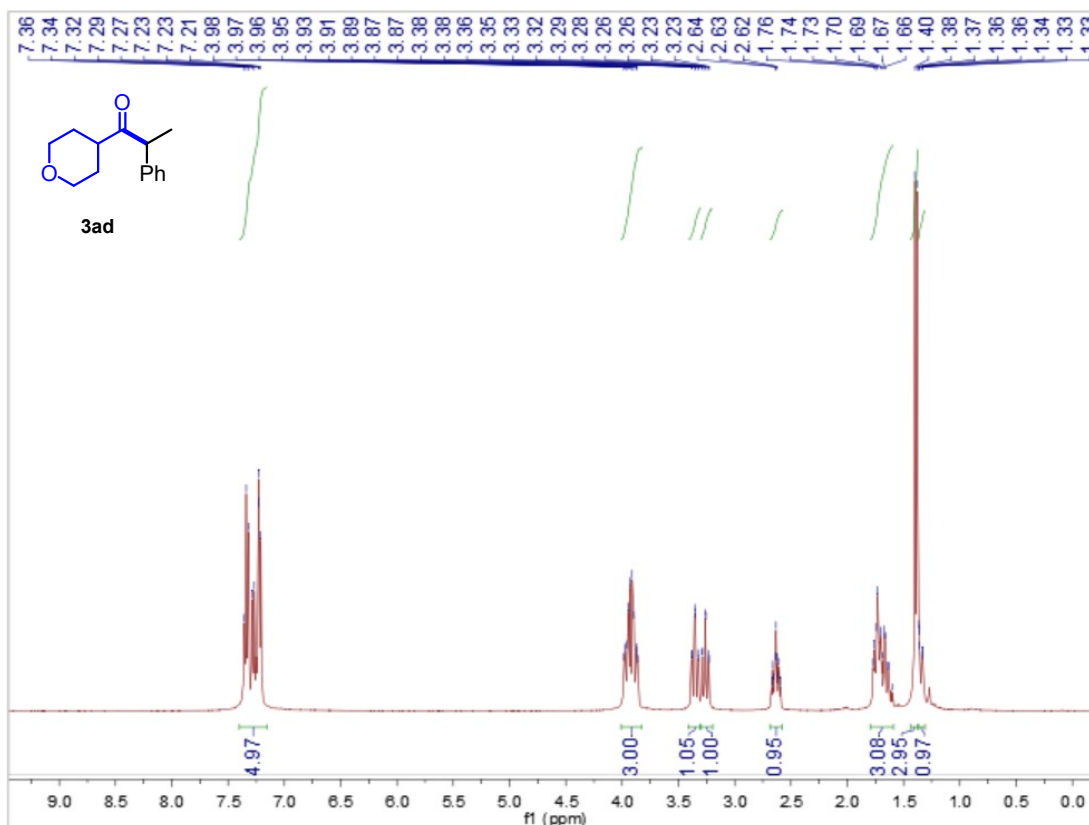




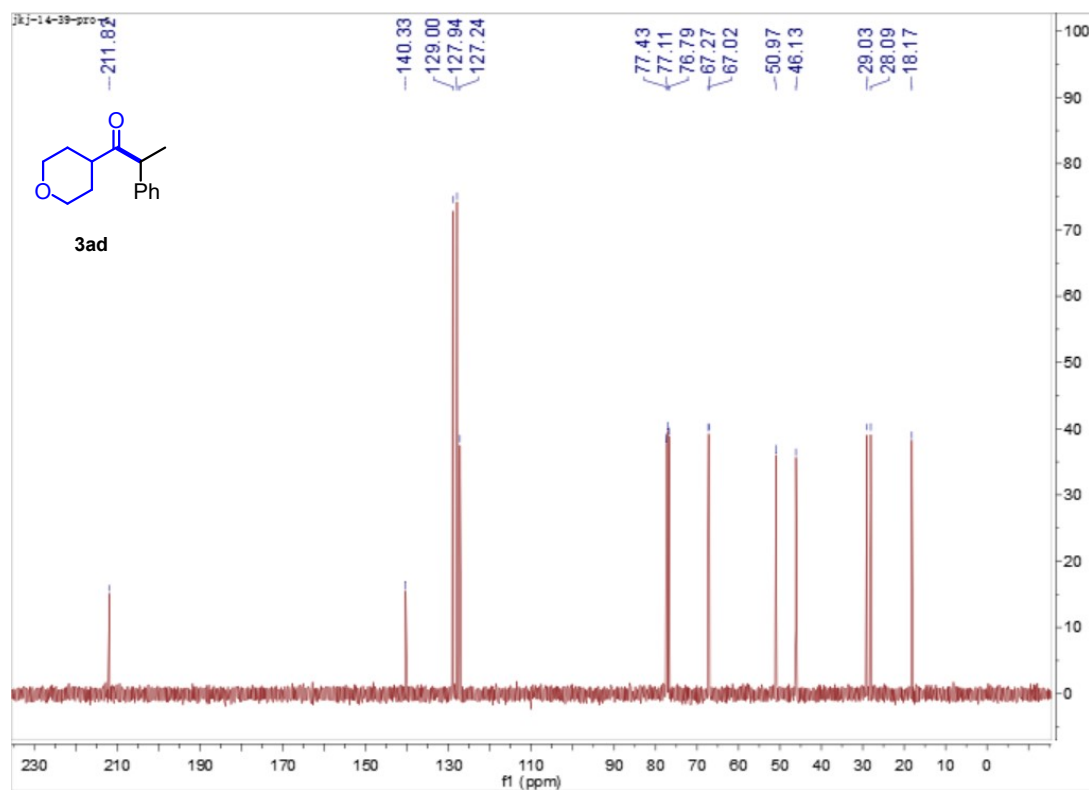
### <sup>13</sup>C NMR Spectrum of 3ac (CDCl<sub>3</sub>, 101 MHz)



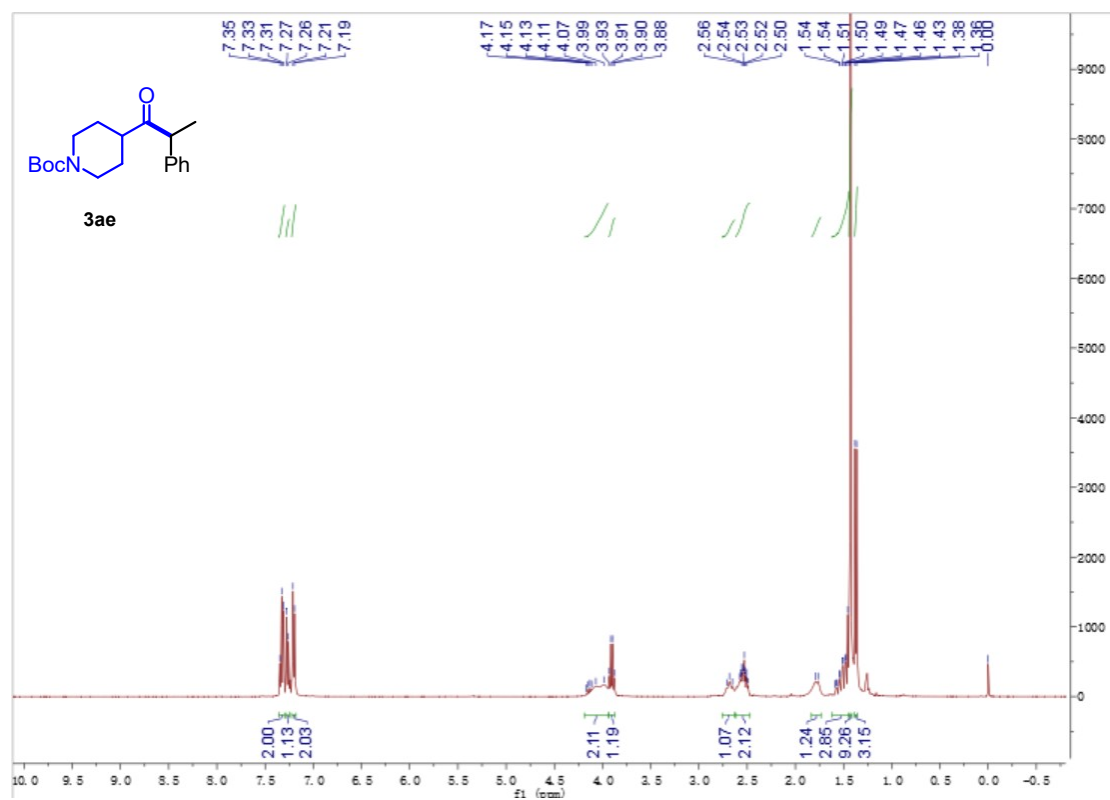
### <sup>1</sup>H NMR Spectrum of 3ad (CDCl<sub>3</sub>, 400 MHz)



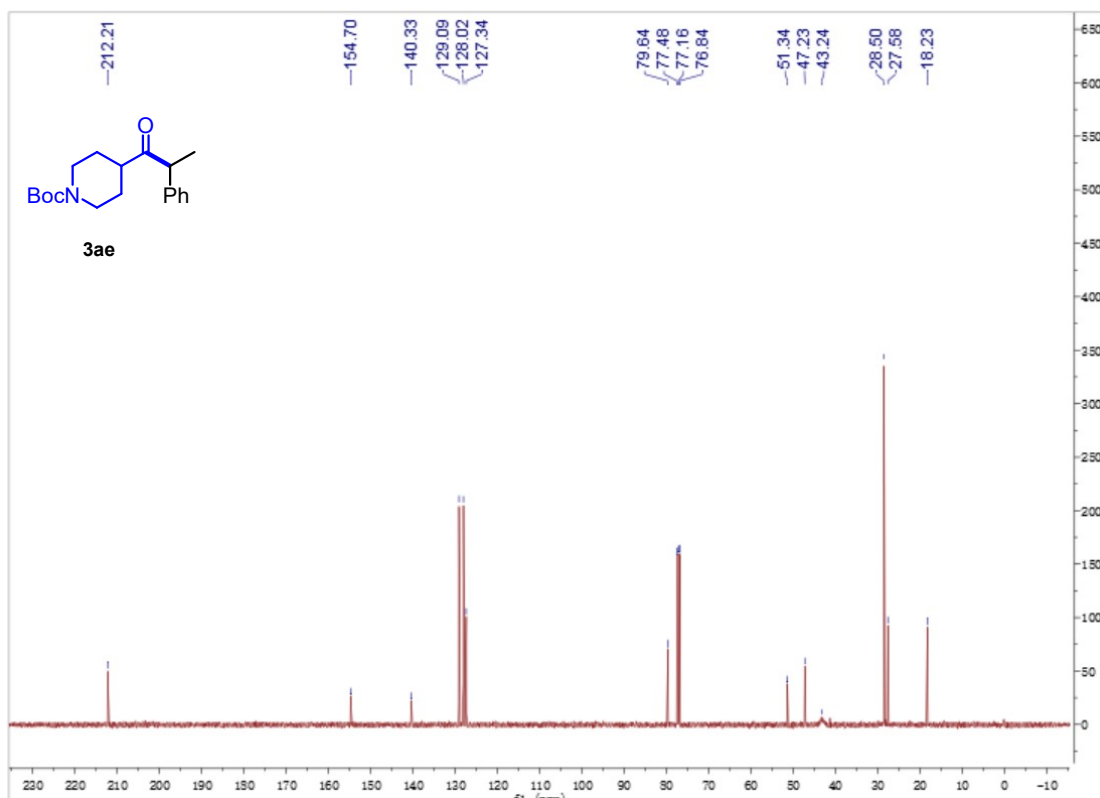
### $^{13}\text{C}$ NMR Spectrum of 3ad ( $\text{CDCl}_3$ , 101 MHz)



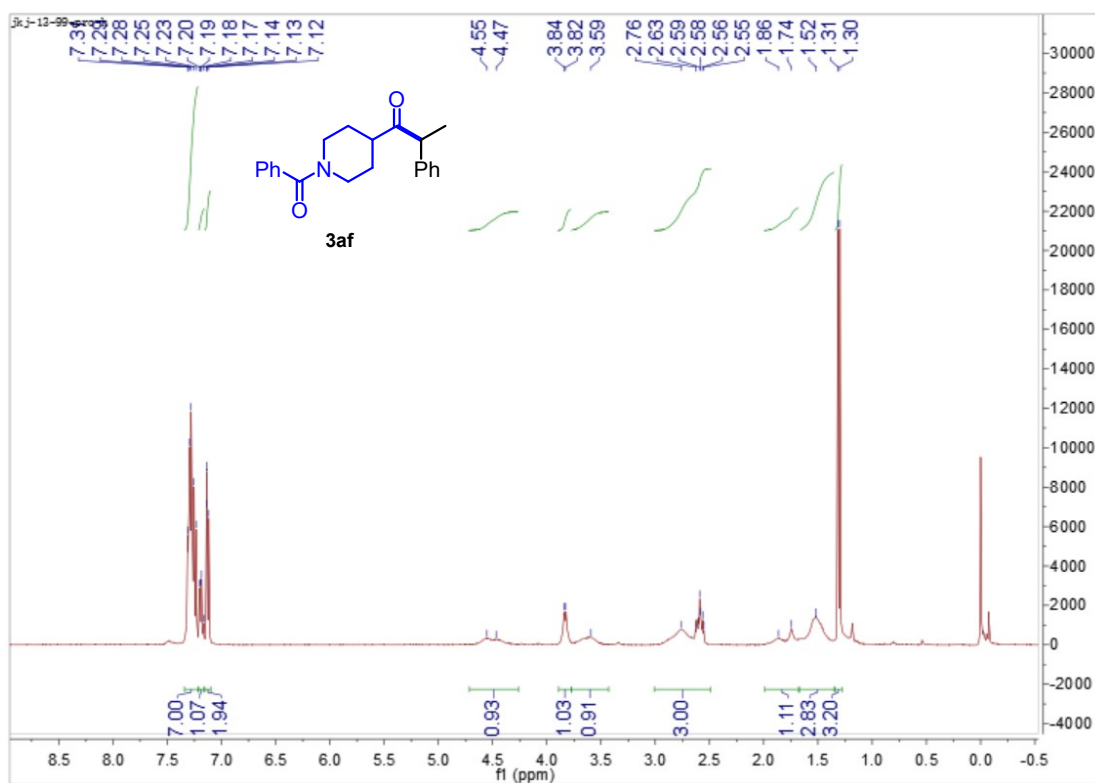
### $^1\text{H}$ NMR Spectrum of 3ae ( $\text{CDCl}_3$ , 400 MHz)



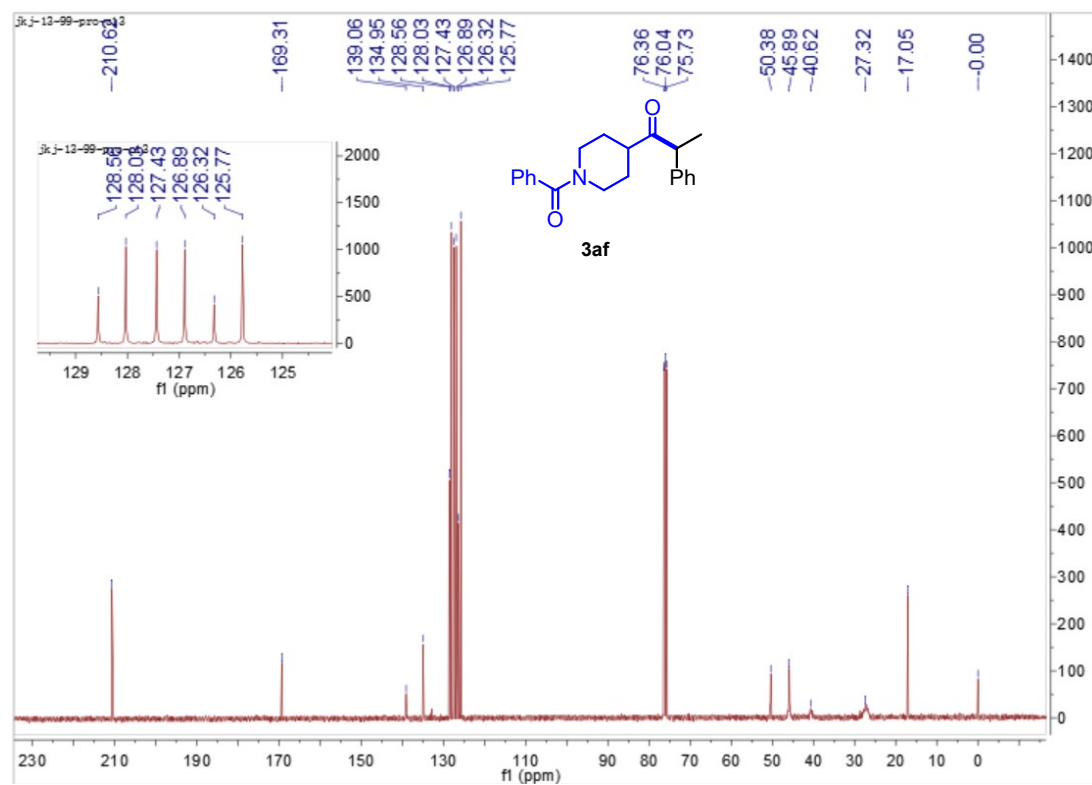
### $^{13}\text{C}$ NMR Spectrum of 3ae ( $\text{CDCl}_3$ , 101 MHz)



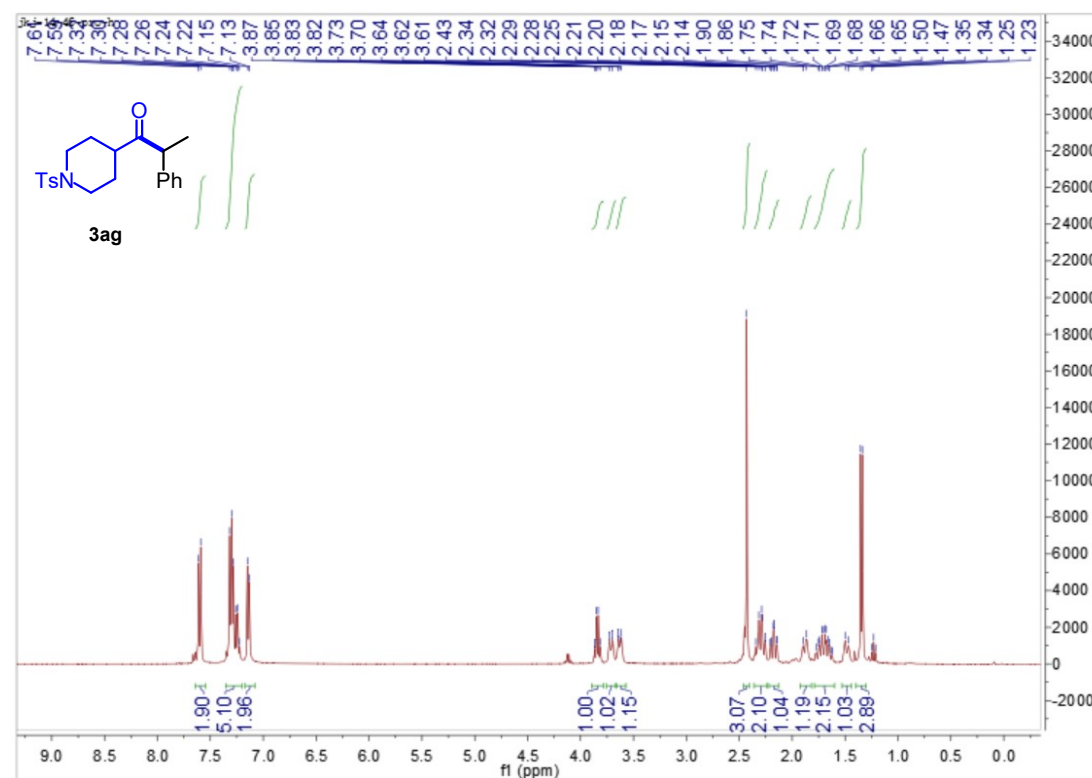
### $^1\text{H}$ NMR Spectrum of 3af ( $\text{CDCl}_3$ , 400 MHz)



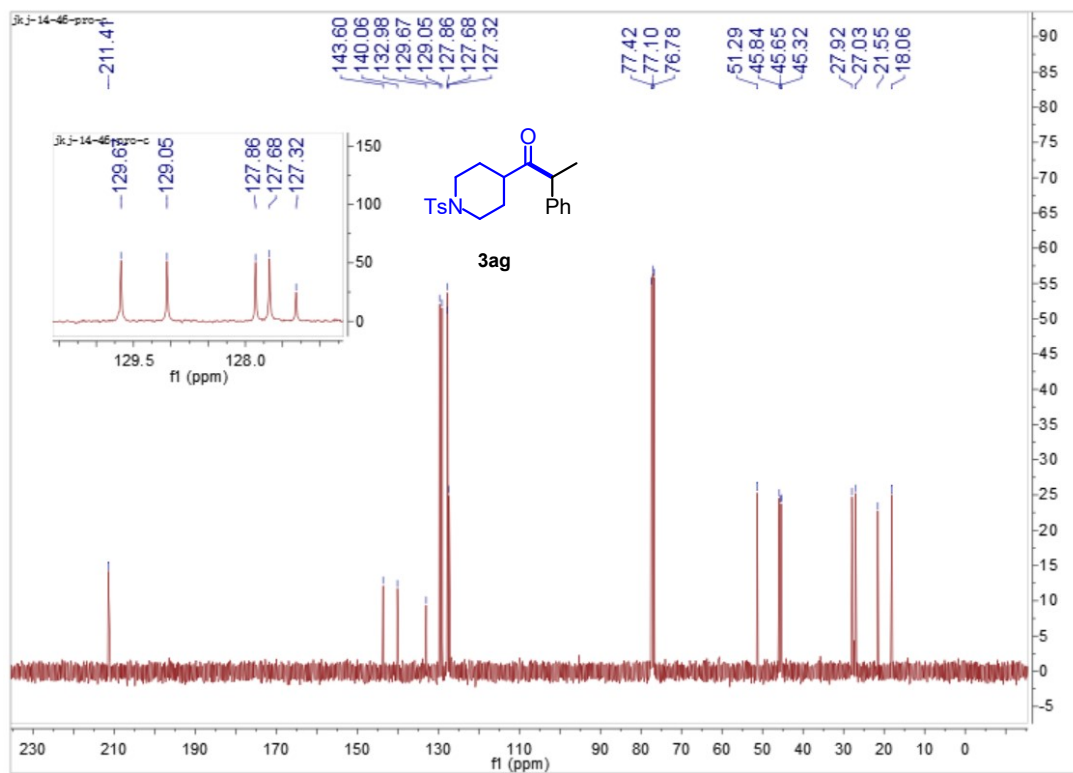
### <sup>13</sup>C NMR Spectrum of 3af (CDCl<sub>3</sub>, 101 MHz)



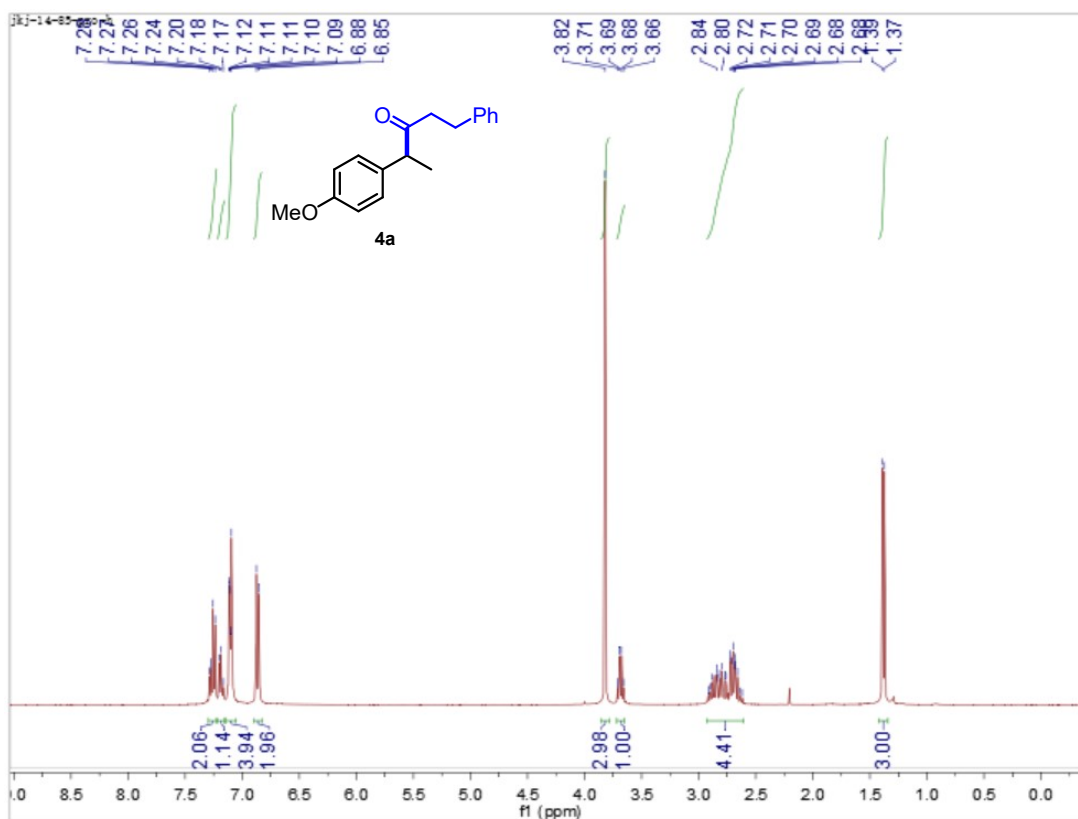
### <sup>1</sup>H NMR Spectrum of 3ag (CDCl<sub>3</sub>, 400 MHz)



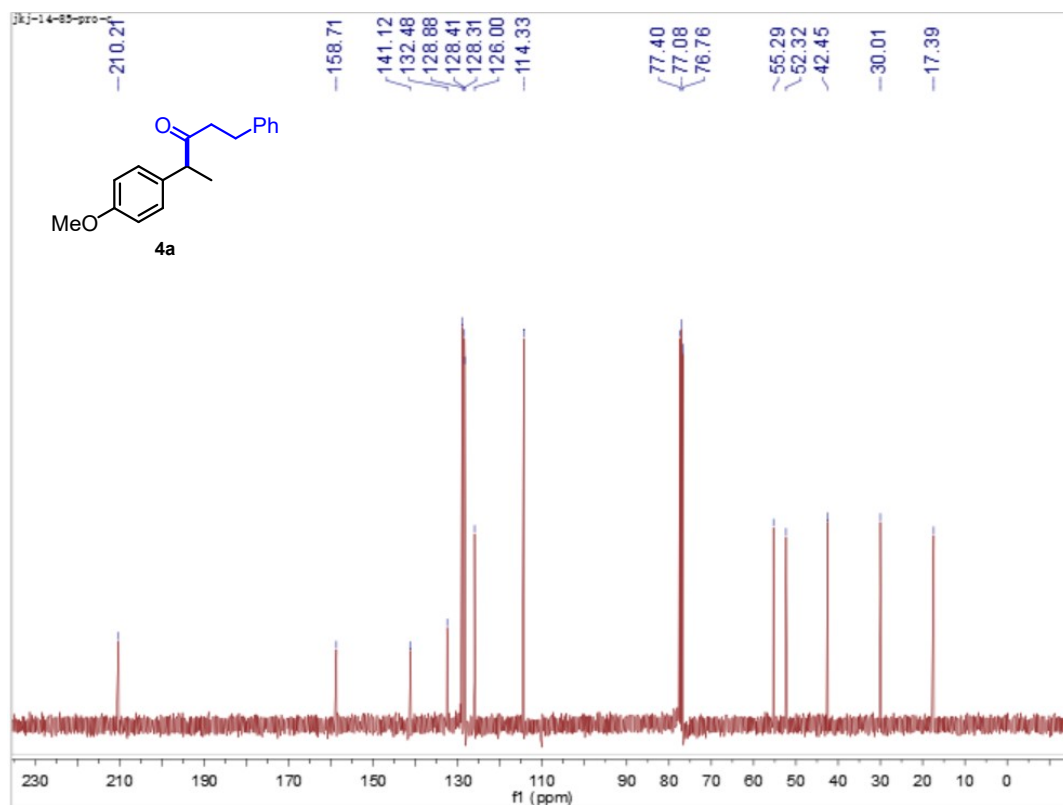
### <sup>13</sup>C NMR Spectrum of 3ag (CDCl<sub>3</sub>, 101 MHz)



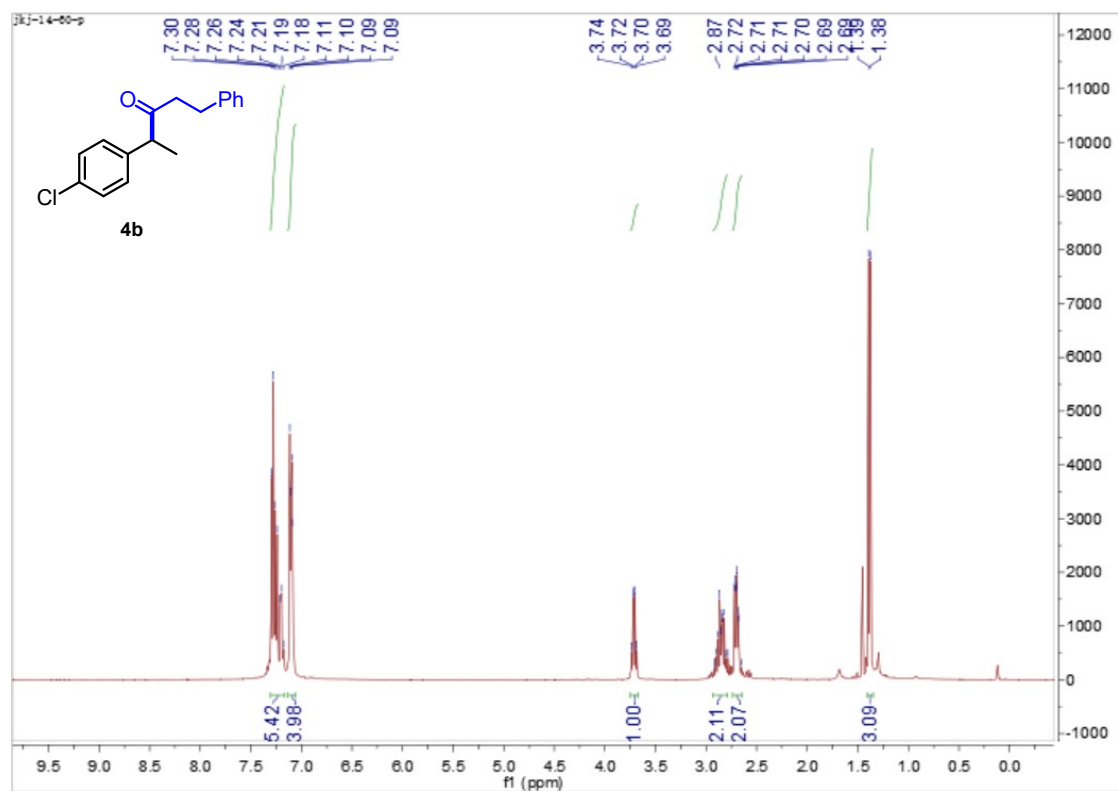
### <sup>1</sup>H NMR Spectrum of 4a (CDCl<sub>3</sub>, 400 MHz)



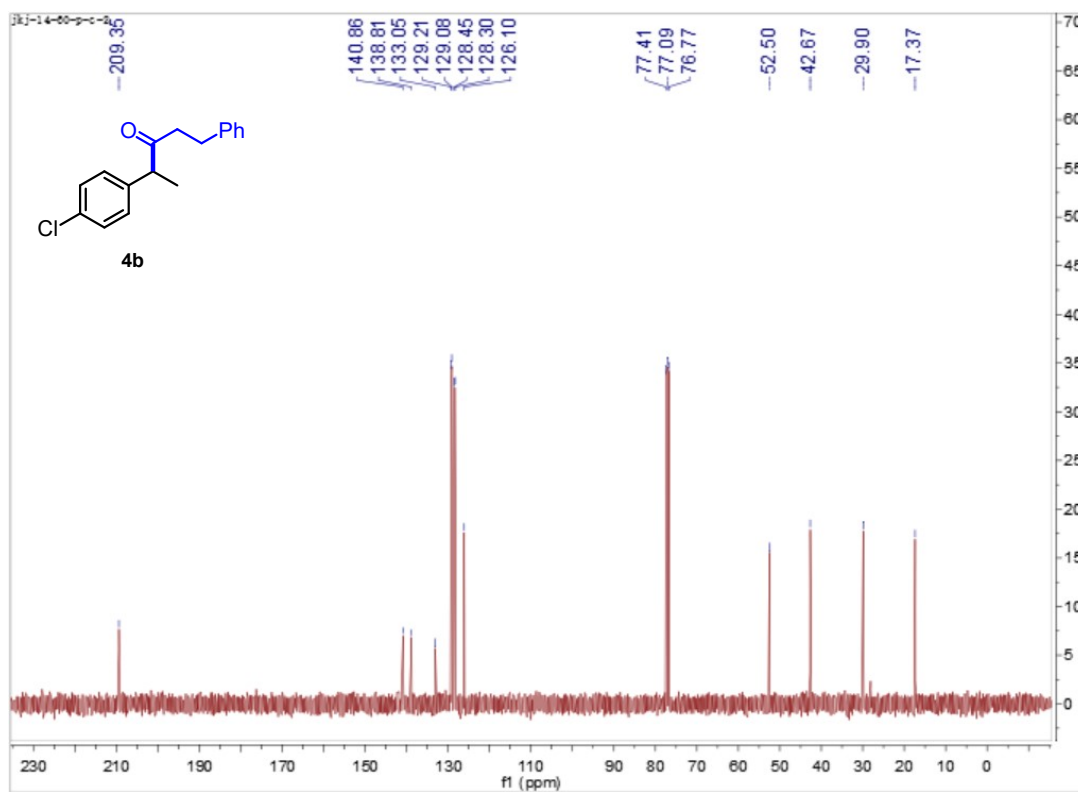
### <sup>13</sup>C NMR Spectrum of 4a (CDCl<sub>3</sub>, 101 MHz)



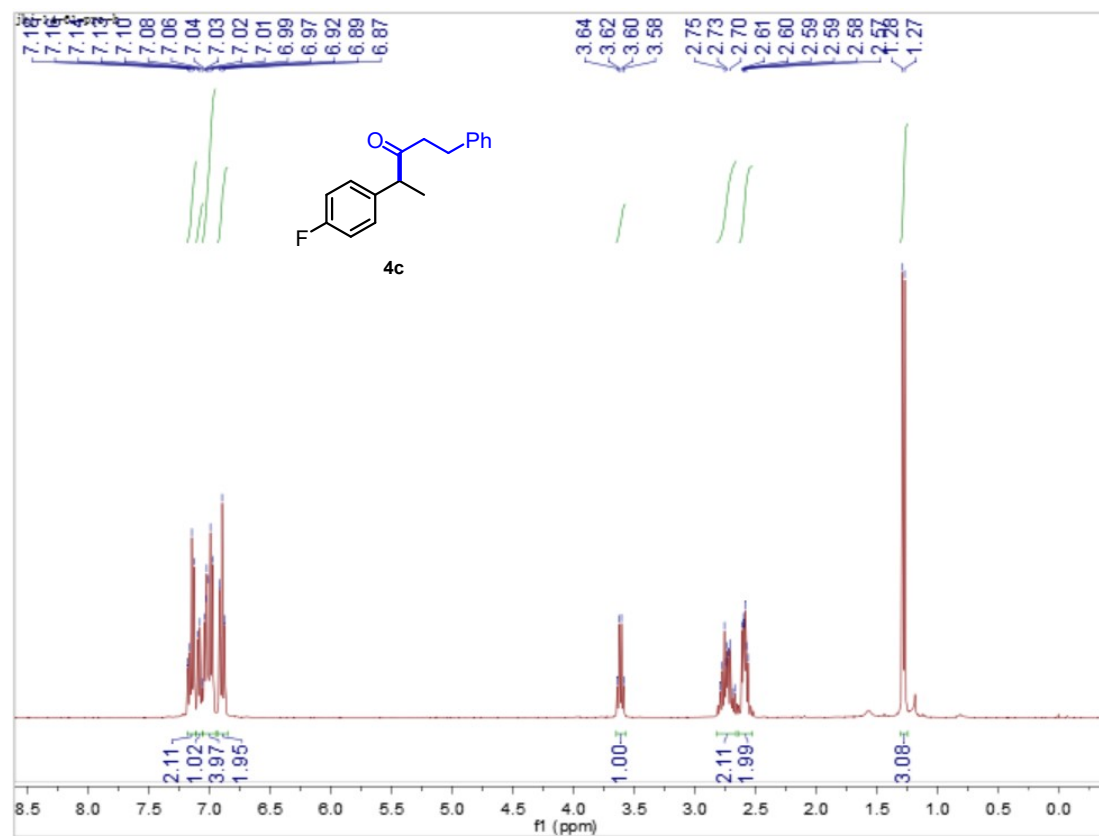
### <sup>1</sup>H NMR Spectrum of 4b (CDCl<sub>3</sub>, 400 MHz)



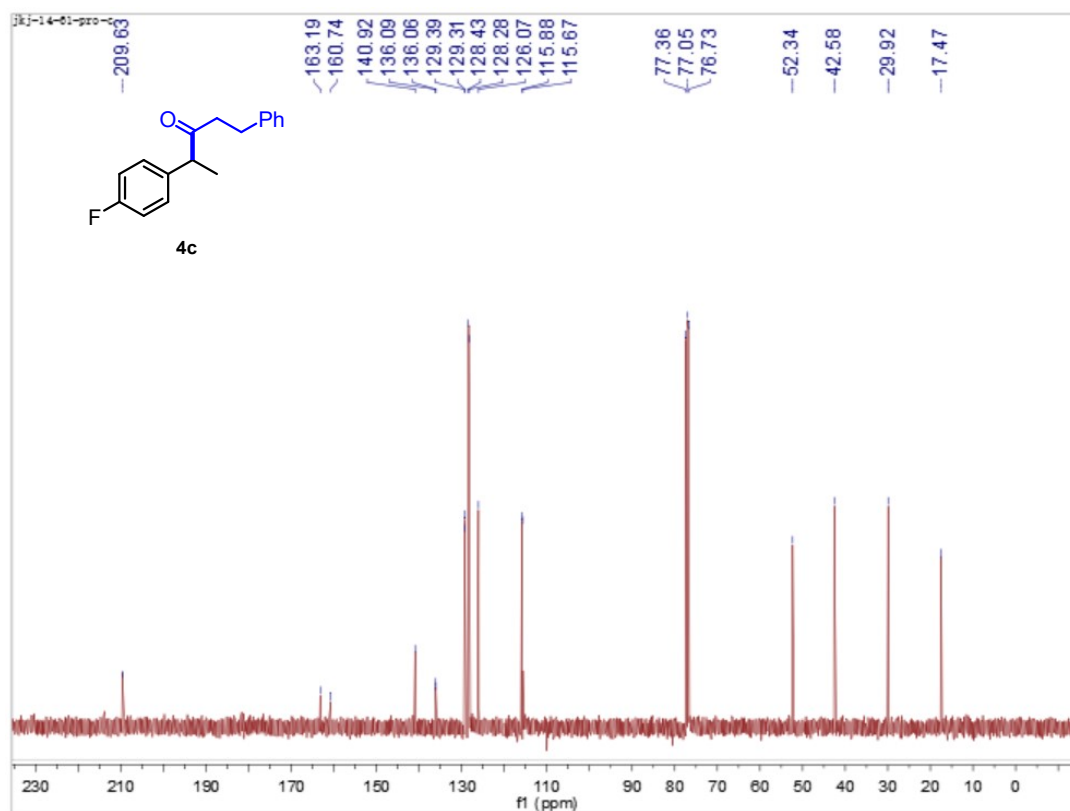
### $^{13}\text{C}$ NMR Spectrum of 4b ( $\text{CDCl}_3$ , 101 MHz)



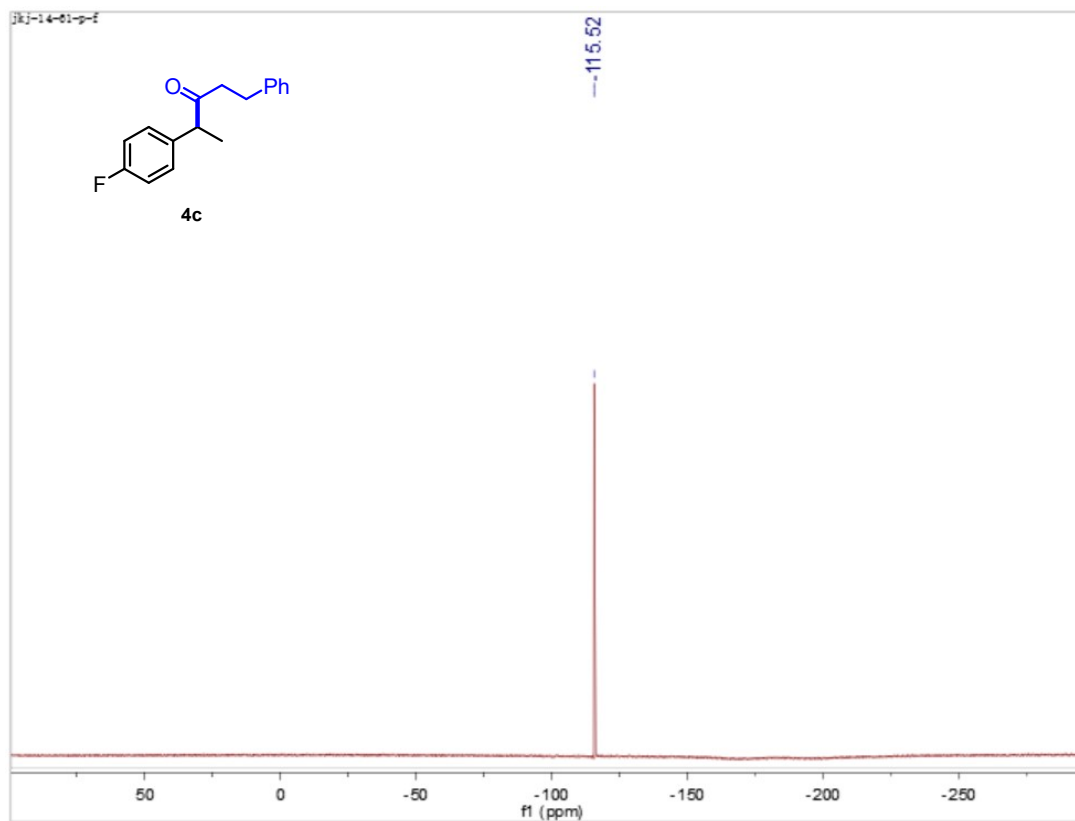
### $^1\text{H}$ NMR Spectrum of 4c ( $\text{CDCl}_3$ , 400 MHz)



### <sup>13</sup>C NMR Spectrum of 4c (CDCl<sub>3</sub>, 101 MHz)

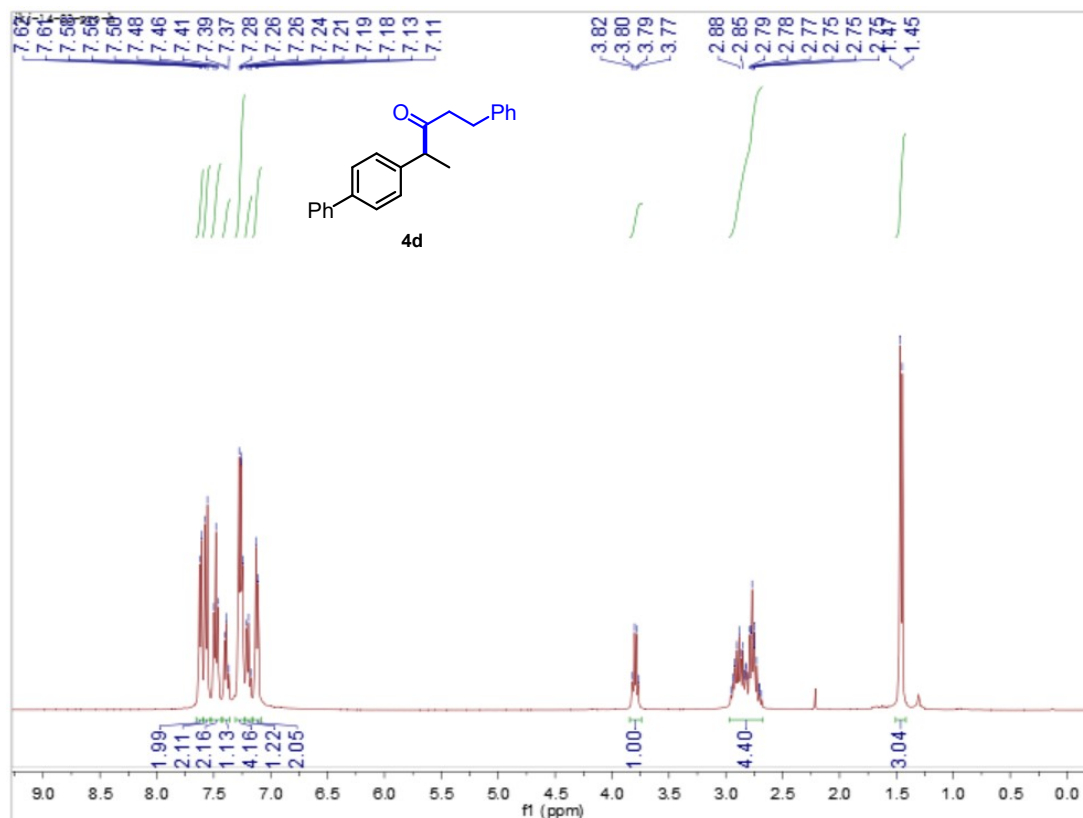


### <sup>19</sup>F NMR Spectrum of 4c (CDCl<sub>3</sub>, 376 MHz)

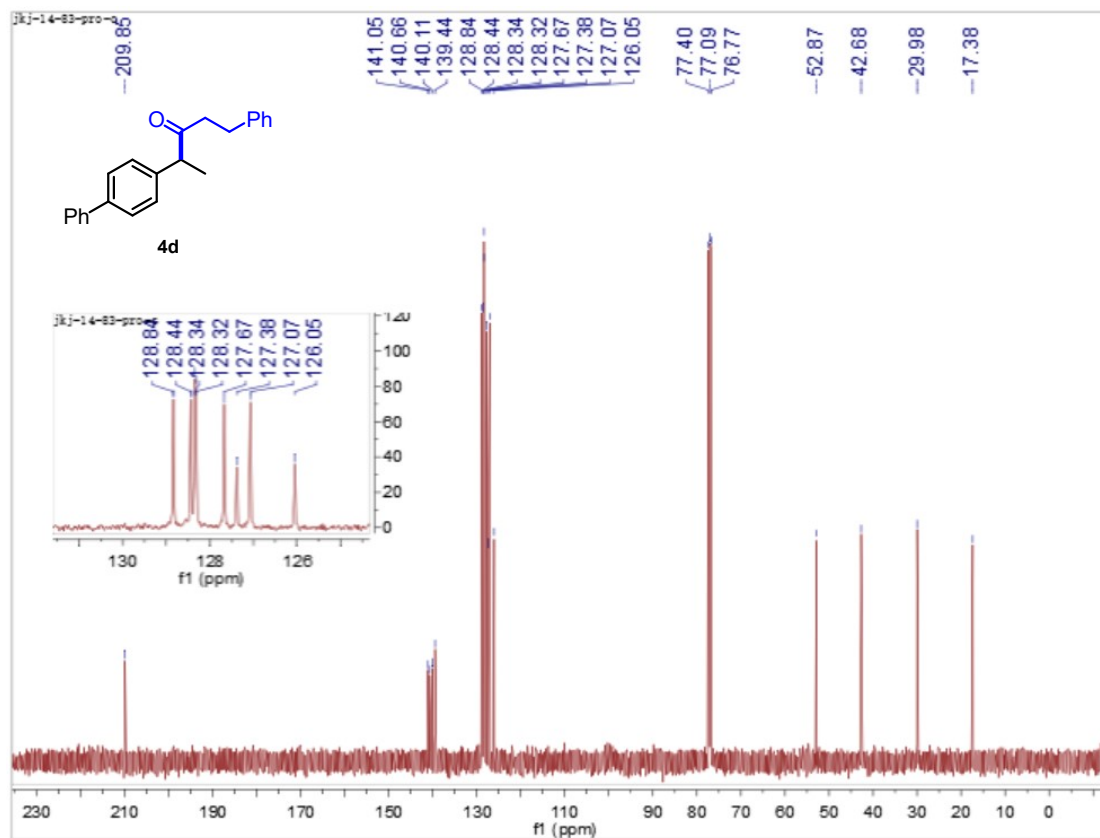




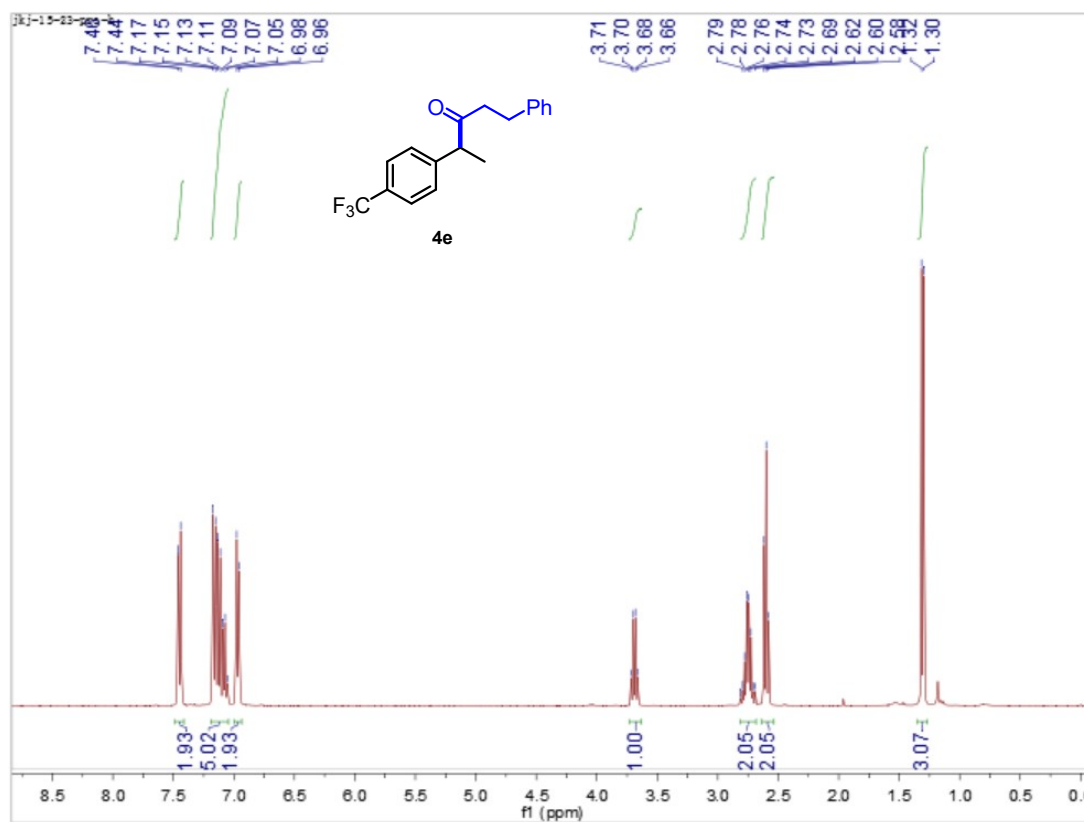
### <sup>1</sup>H NMR Spectrum of 4d (CDCl<sub>3</sub>, 400 MHz)



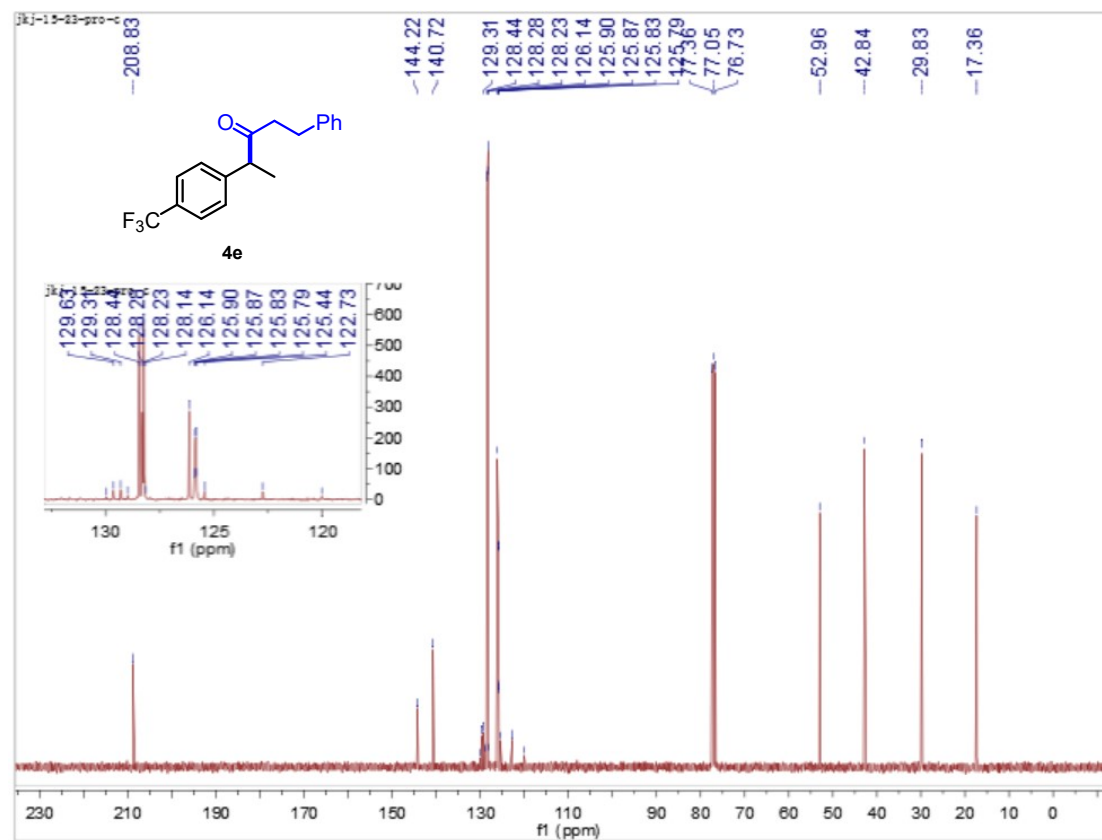
### <sup>13</sup>C NMR Spectrum of 4d (CDCl<sub>3</sub>, 101 MHz)



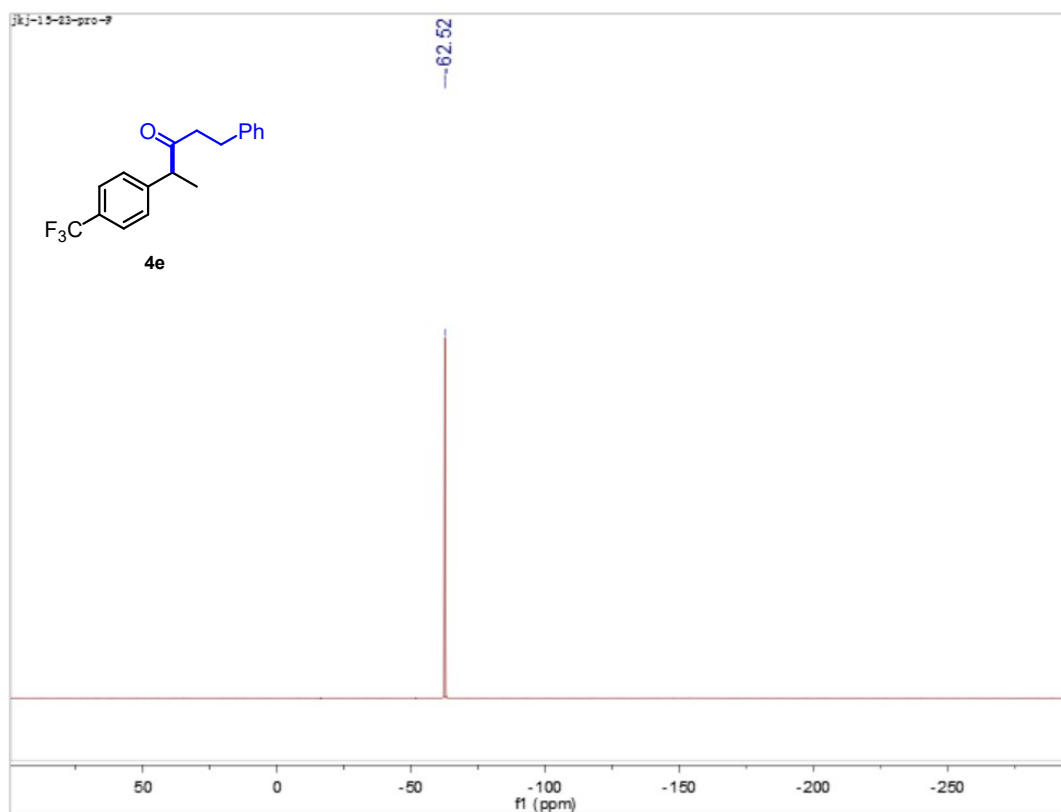
### <sup>1</sup>H NMR Spectrum of 4e (CDCl<sub>3</sub>, 400 MHz)



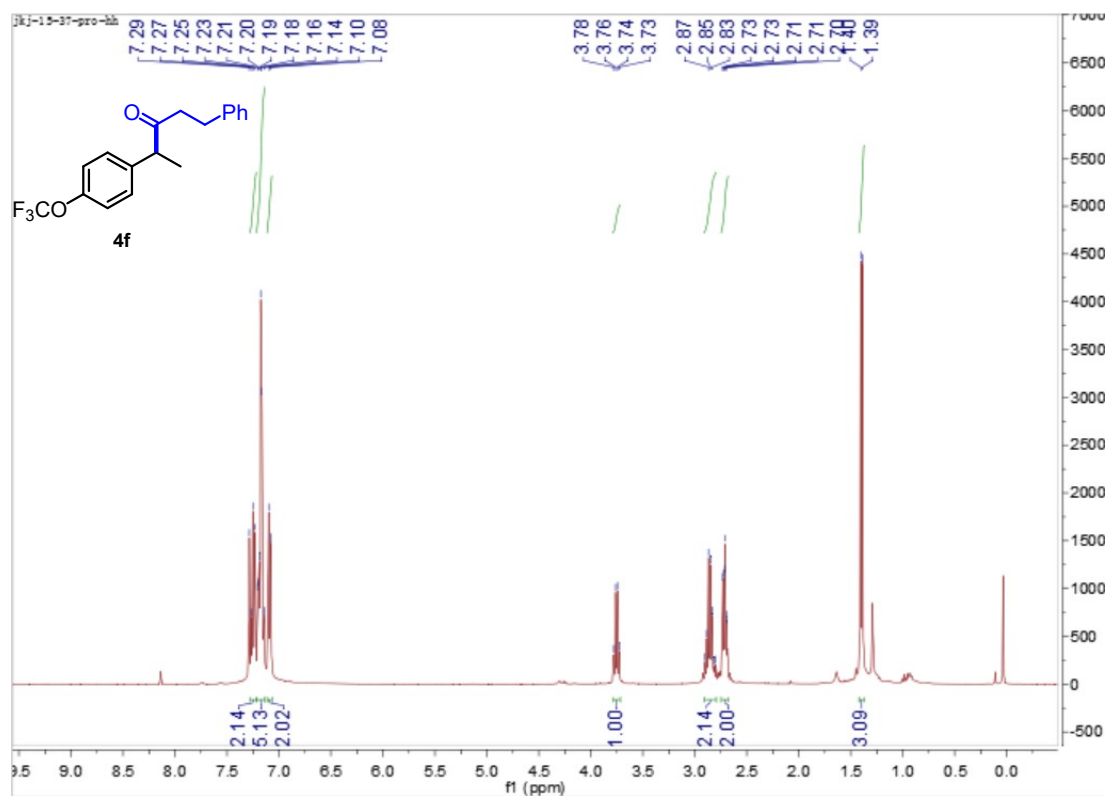
### <sup>13</sup>C NMR Spectrum of 4e (CDCl<sub>3</sub>, 101 MHz)



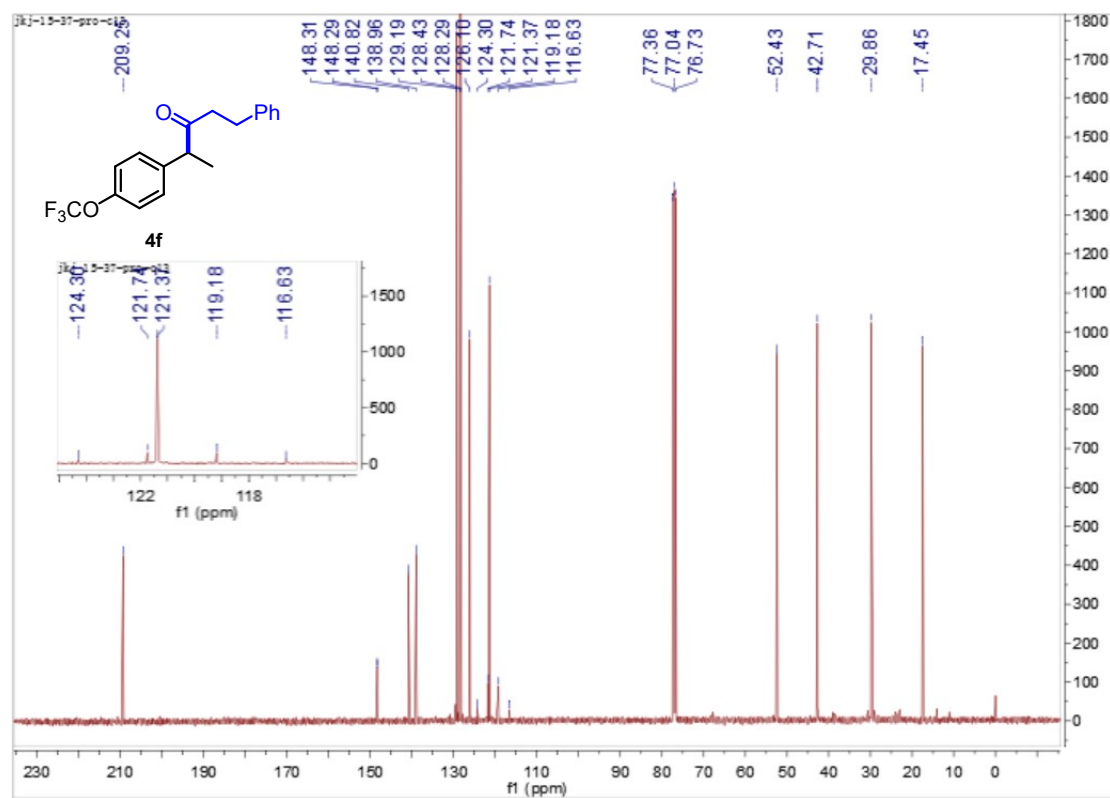
### $^{19}\text{F}$ NMR Spectrum of 4e ( $\text{CDCl}_3$ , 376 MHz)



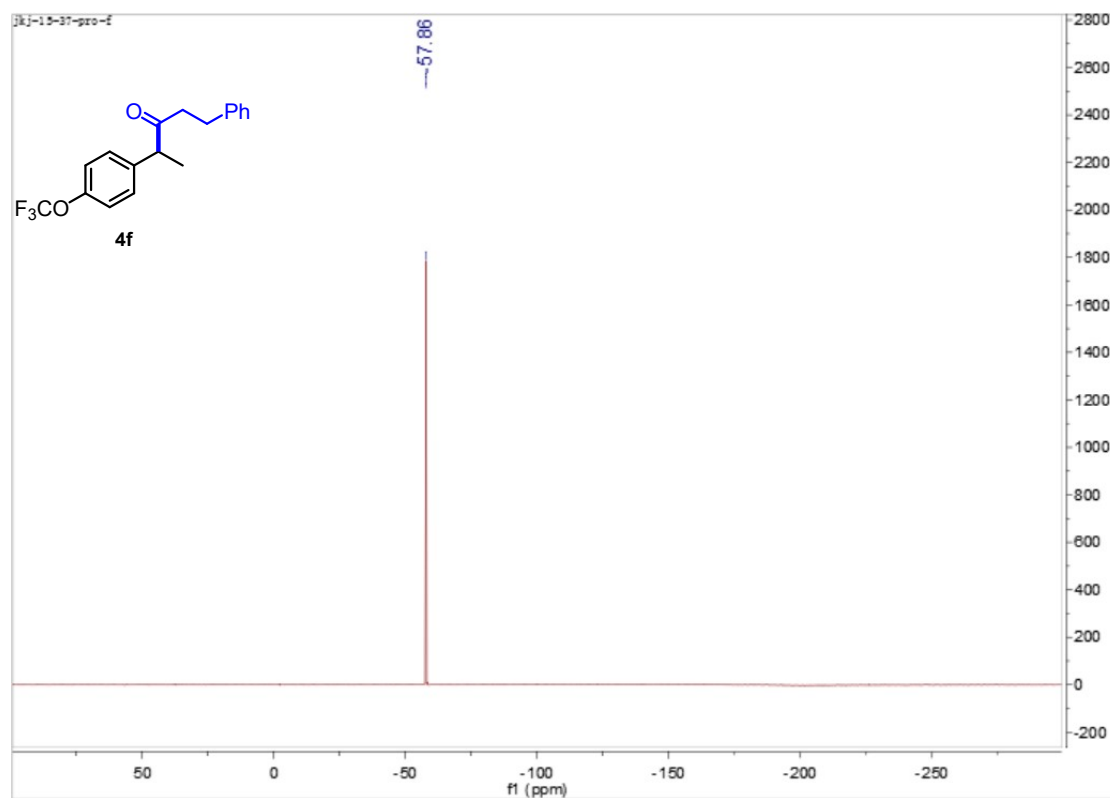
### $^1\text{H}$ NMR Spectrum of 4f ( $\text{CDCl}_3$ , 400 MHz)



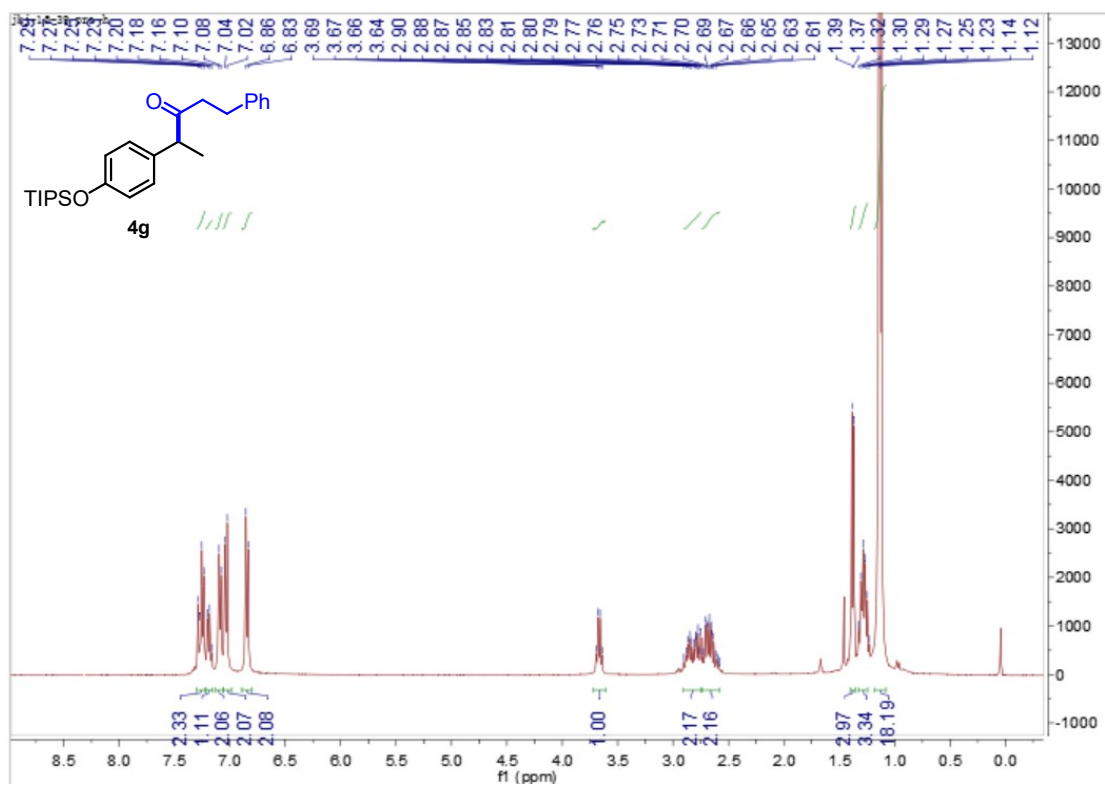
### $^{13}\text{C}$ NMR Spectrum of 4f ( $\text{CDCl}_3$ , 101 MHz)



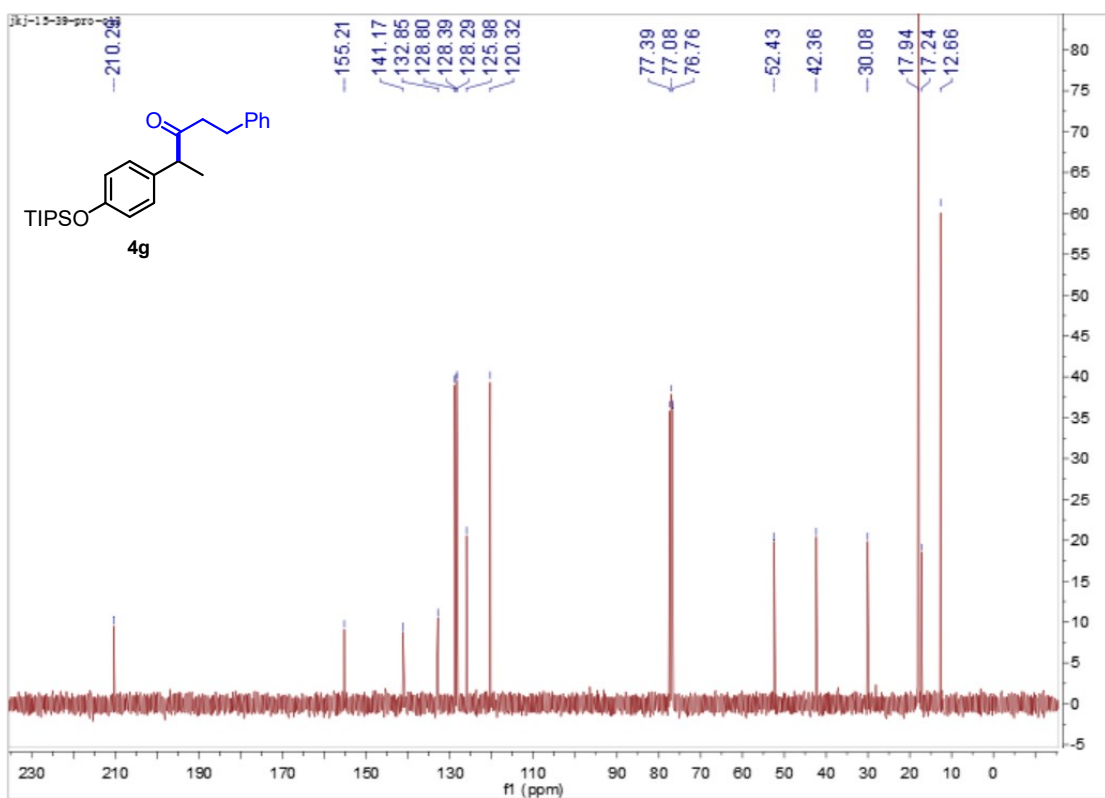
### $^{19}\text{F}$ NMR Spectrum of 4f ( $\text{CDCl}_3$ , 376 MHz)



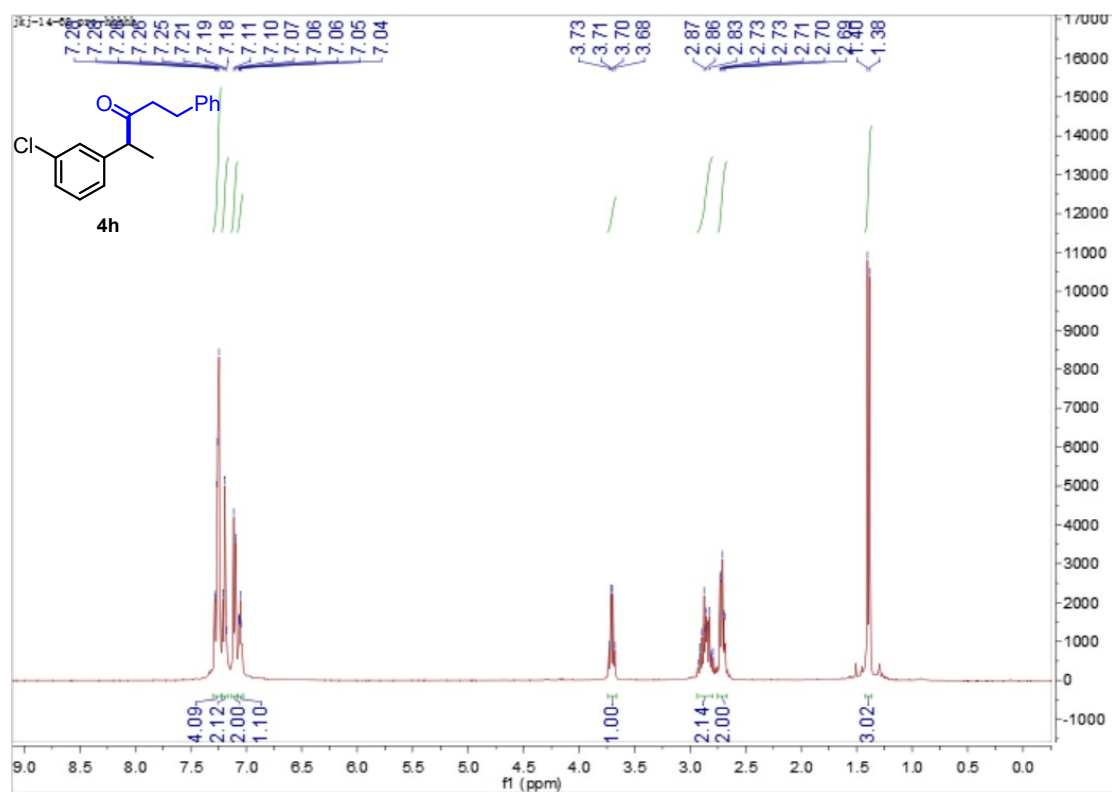
### <sup>1</sup>H NMR Spectrum of 4g (CDCl<sub>3</sub>, 400 MHz)



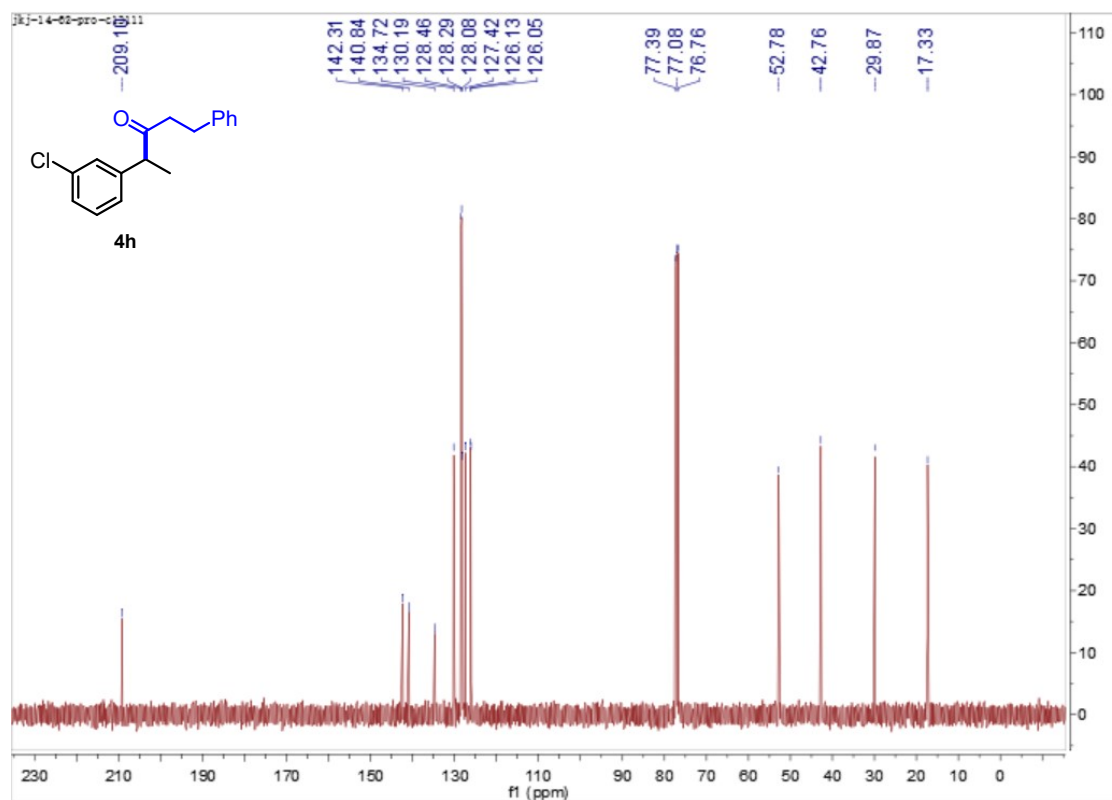
### <sup>13</sup>C NMR Spectrum of 4g (CDCl<sub>3</sub>, 101 MHz)



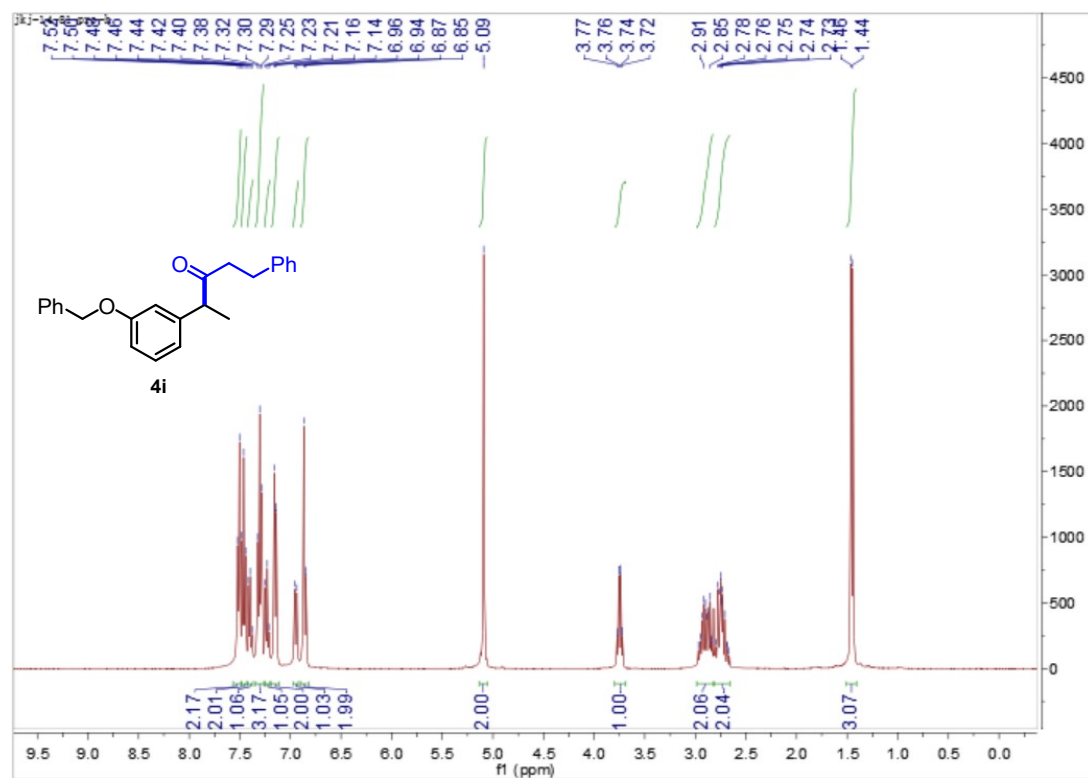
### <sup>1</sup>H NMR Spectrum of 4h (CDCl<sub>3</sub>, 400 MHz)



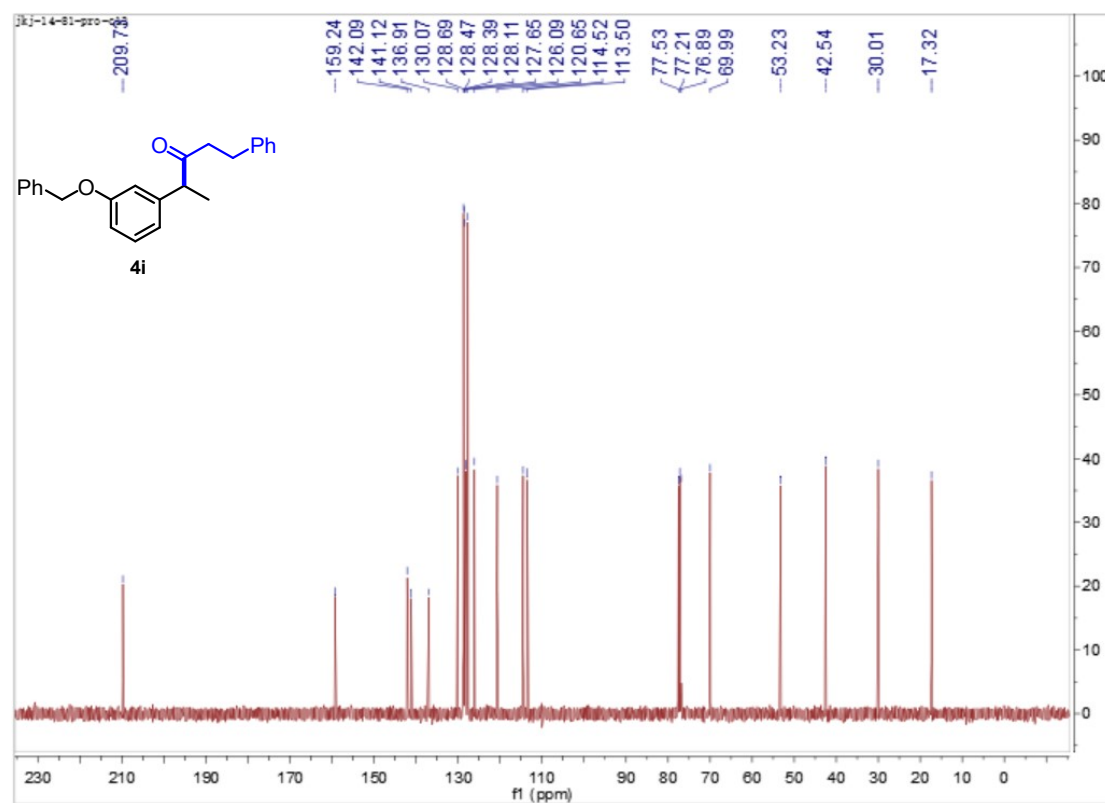
### <sup>13</sup>C NMR Spectrum of 4h (CDCl<sub>3</sub>, 101 MHz)



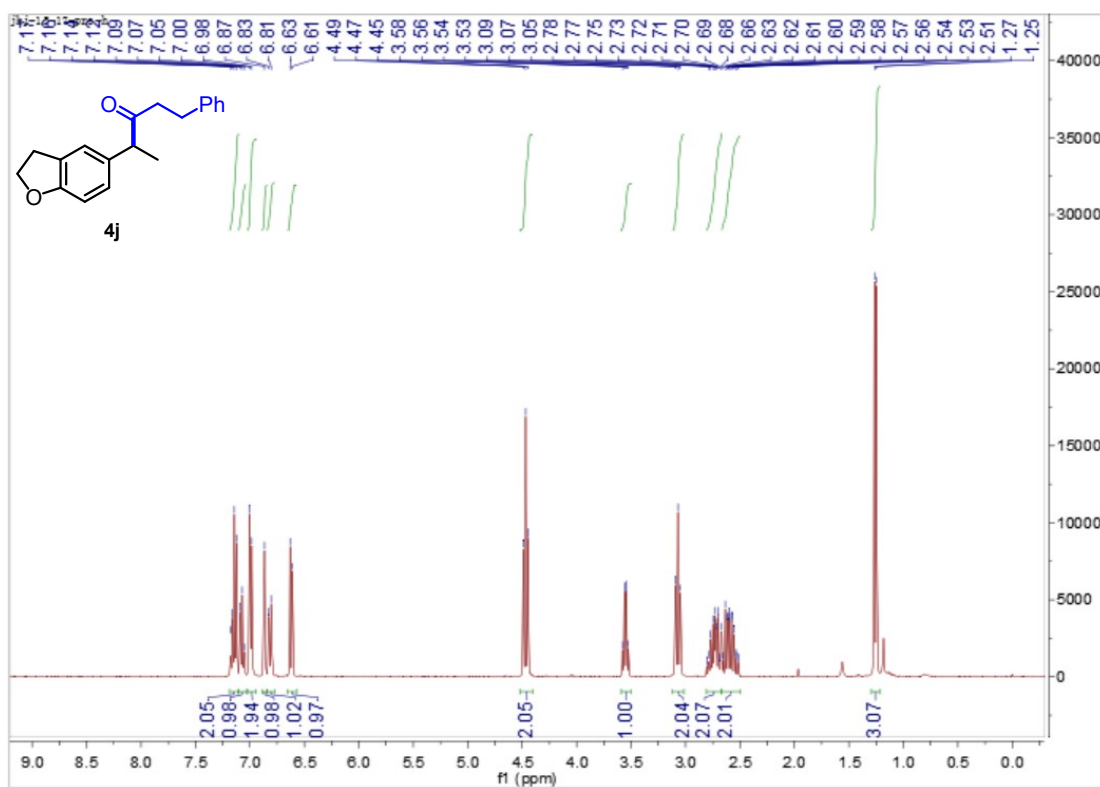
### <sup>1</sup>H NMR Spectrum of 4i (CDCl<sub>3</sub>, 400 MHz)



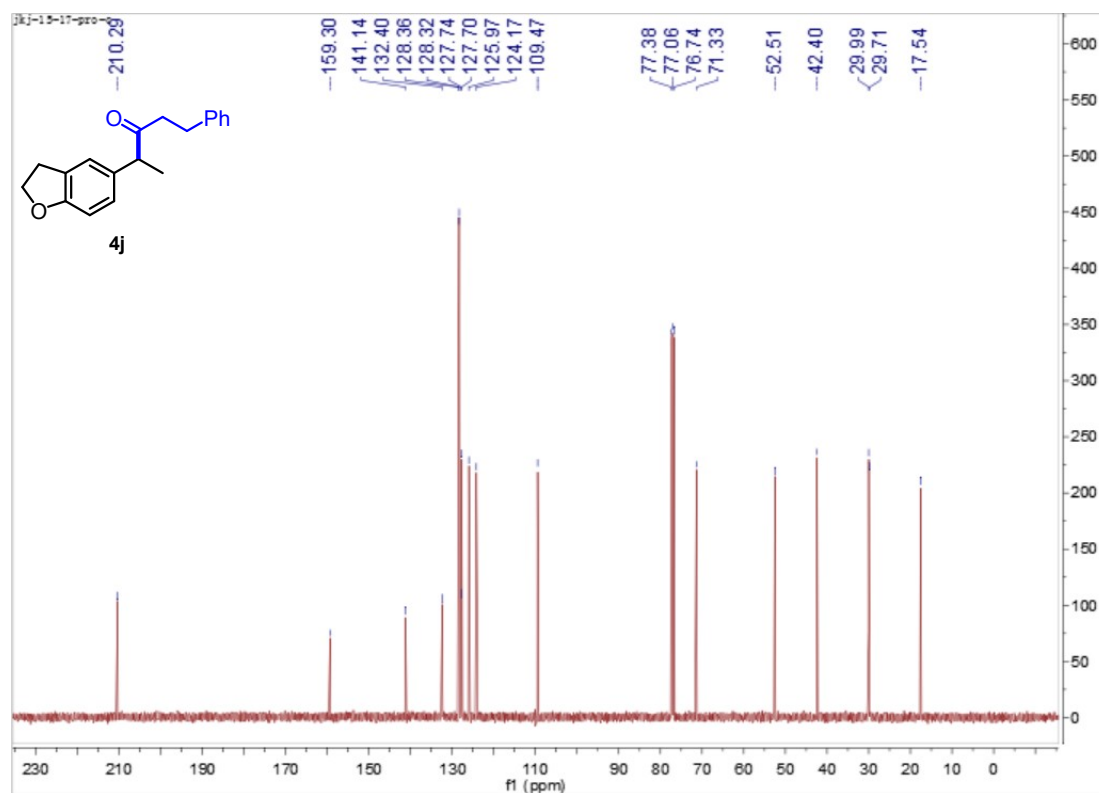
### <sup>13</sup>C NMR Spectrum of 4i (CDCl<sub>3</sub>, 101 MHz)



### <sup>1</sup>H NMR Spectrum of 4j (CDCl<sub>3</sub>, 400 MHz)

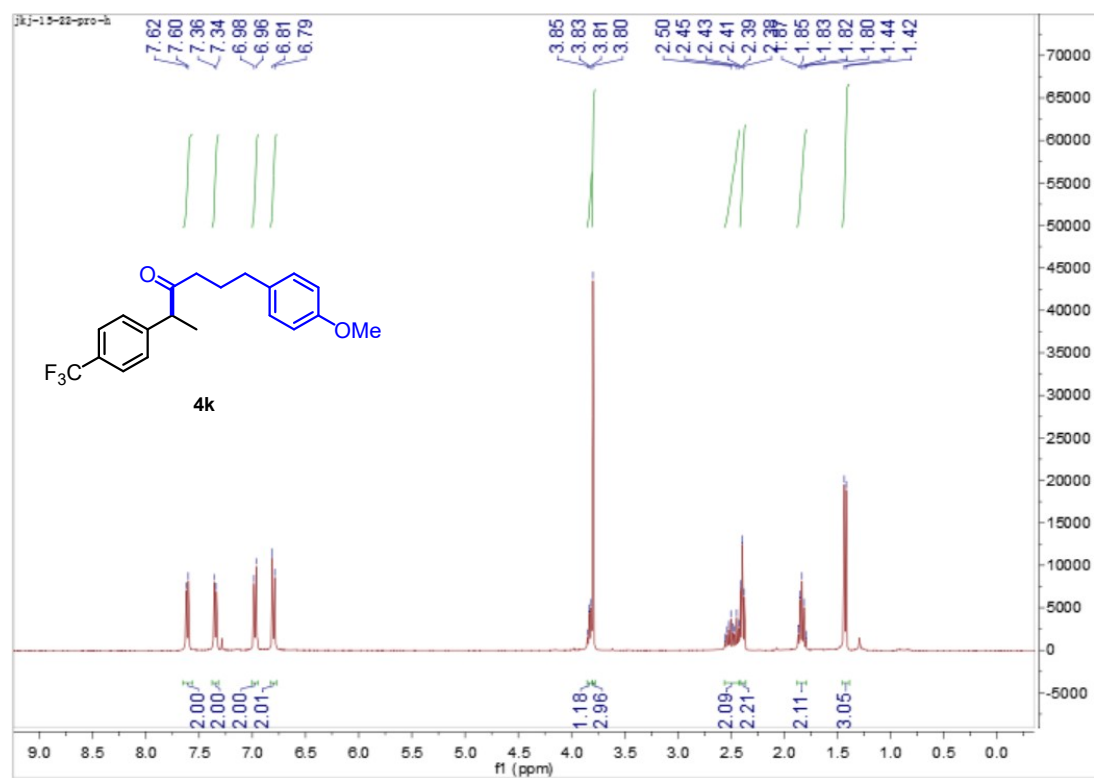


### <sup>13</sup>C NMR Spectrum of 4j (CDCl<sub>3</sub>, 101 MHz)

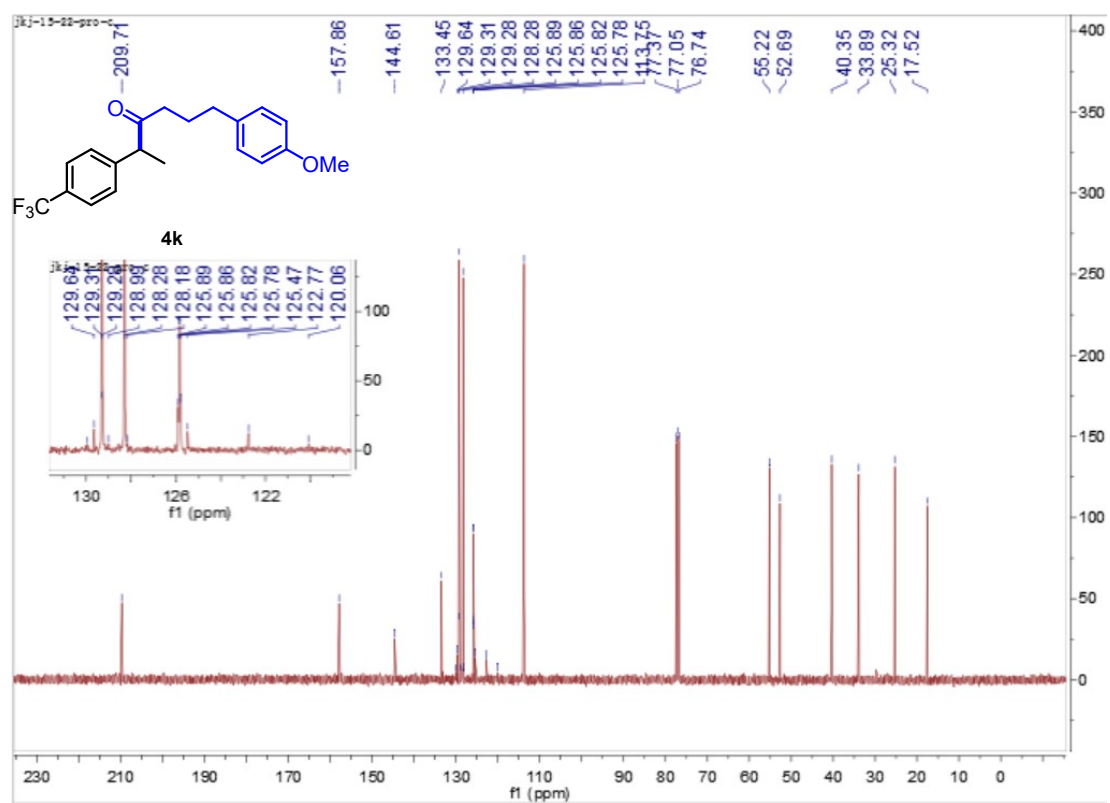




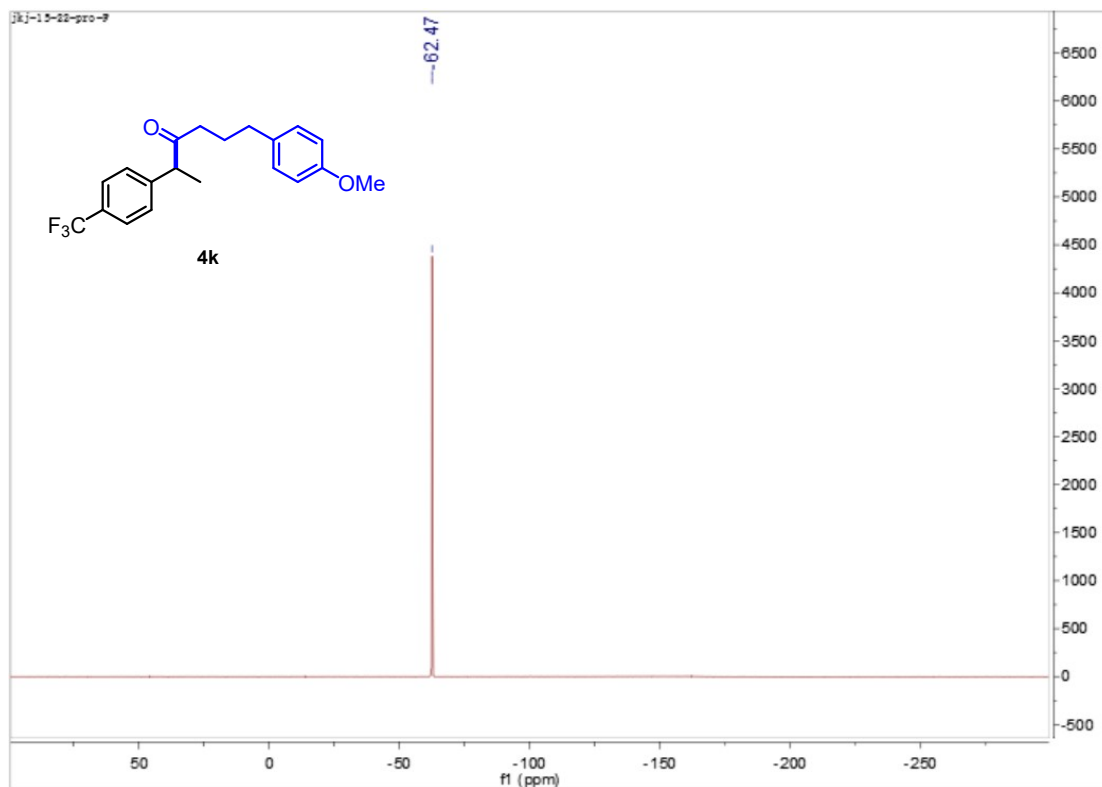
### <sup>1</sup>H NMR Spectrum of 4k (CDCl<sub>3</sub>, 400 MHz)



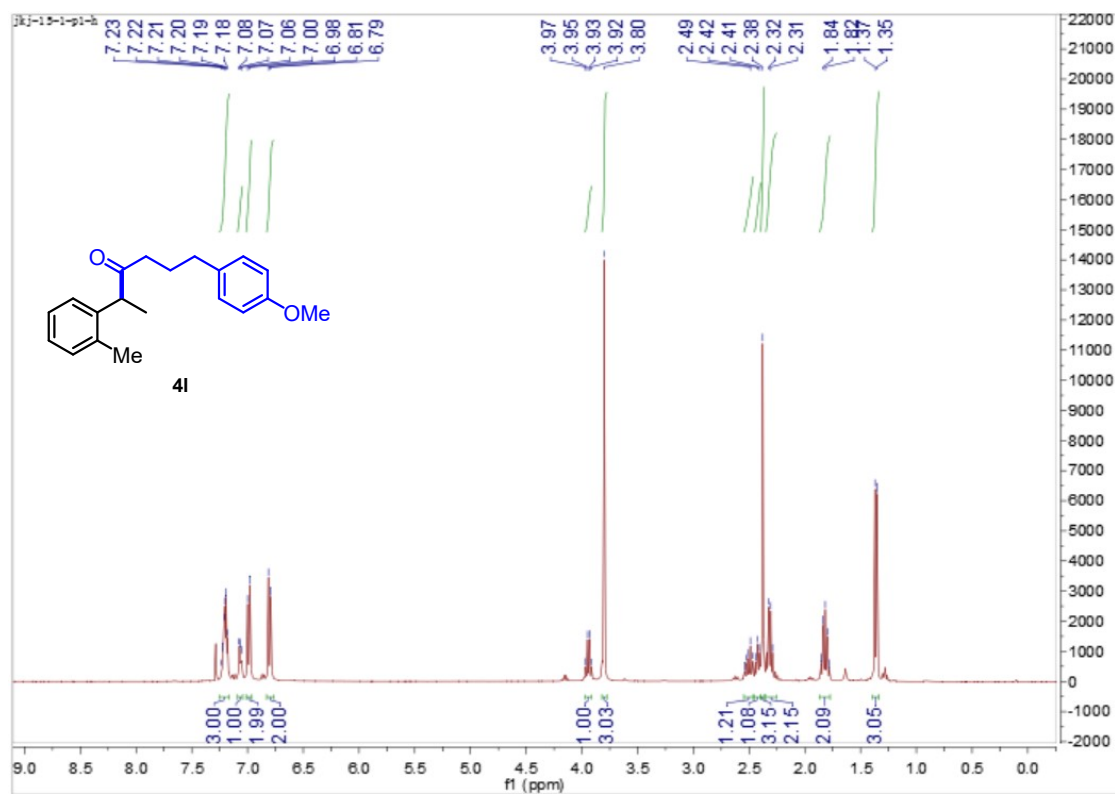
### <sup>13</sup>C NMR Spectrum of 4k (CDCl<sub>3</sub>, 101 MHz)



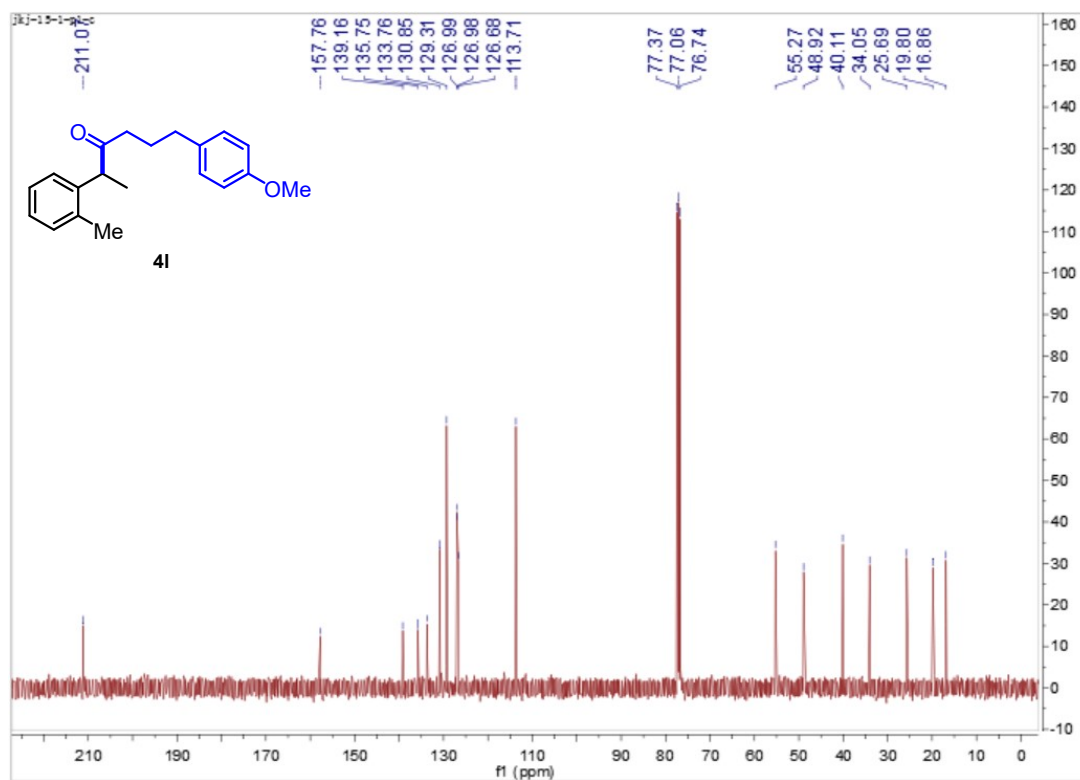
### $^{19}\text{F}$ NMR Spectrum of 4k ( $\text{CDCl}_3$ , 376 MHz)



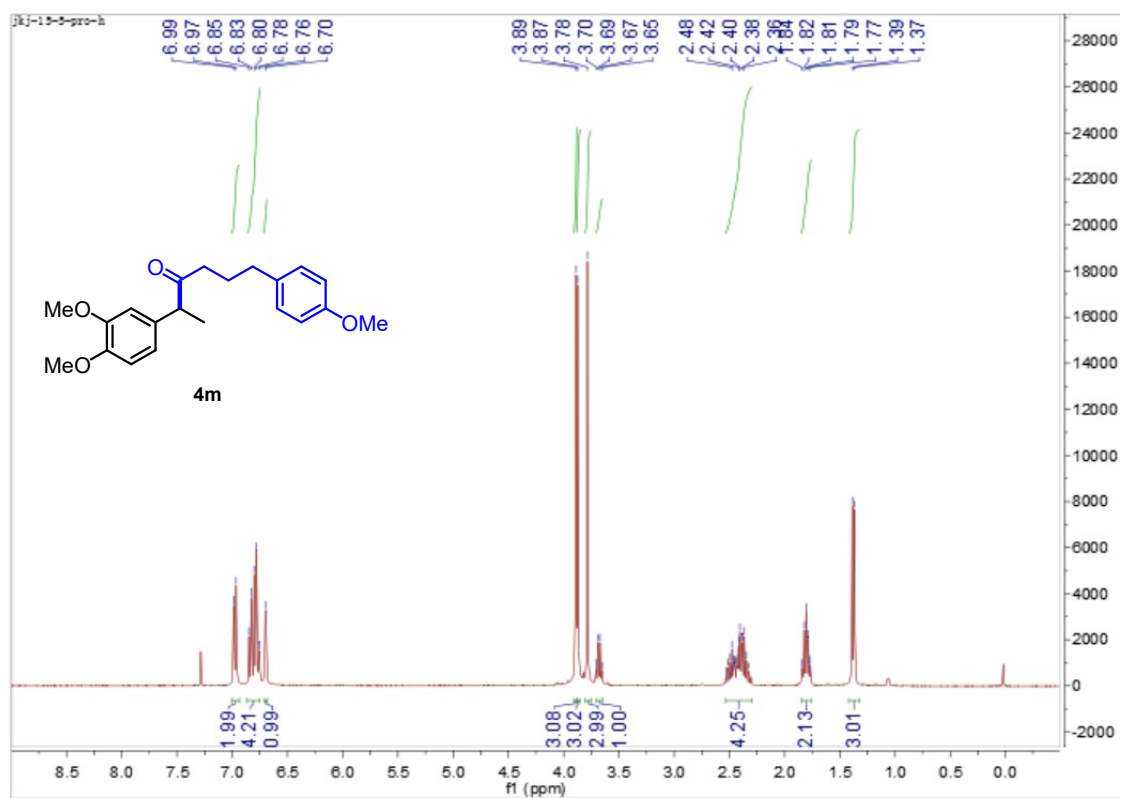
### $^1\text{H}$ NMR Spectrum of 4l ( $\text{CDCl}_3$ , 400 MHz)



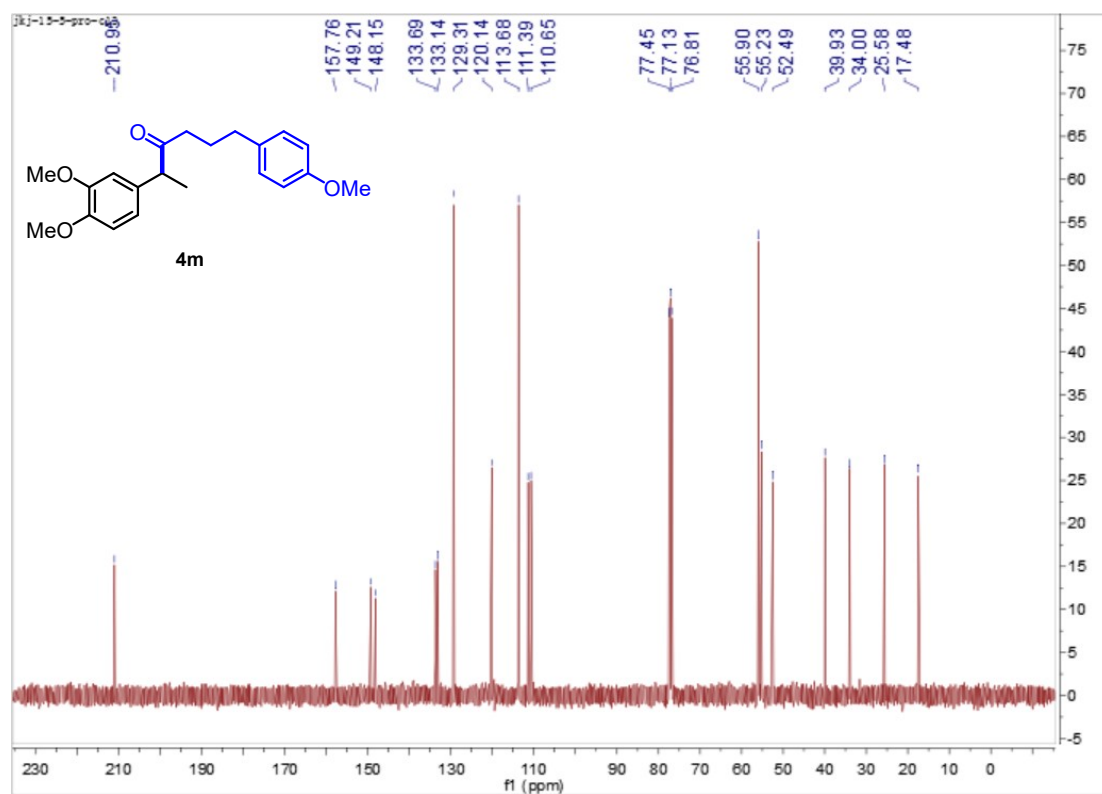
### <sup>13</sup>C NMR Spectrum of 4l (CDCl<sub>3</sub>, 101 MHz)



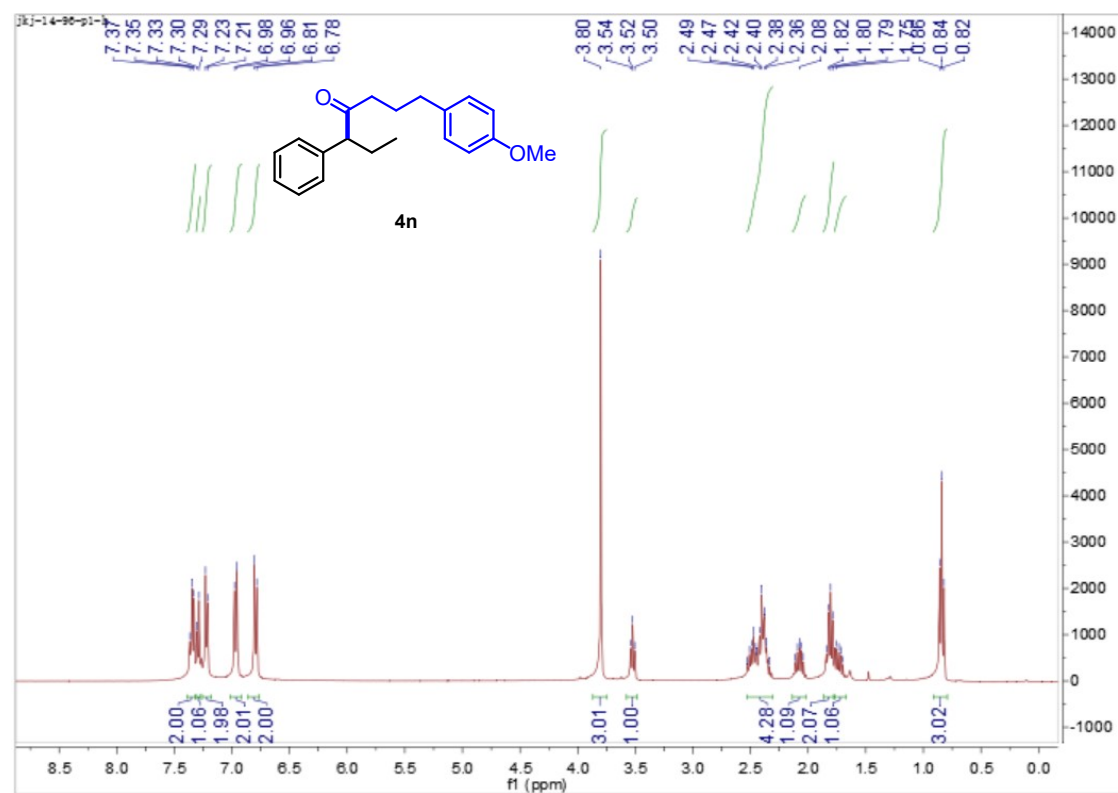
### <sup>1</sup>H NMR Spectrum of 4m (CDCl<sub>3</sub>, 400 MHz)



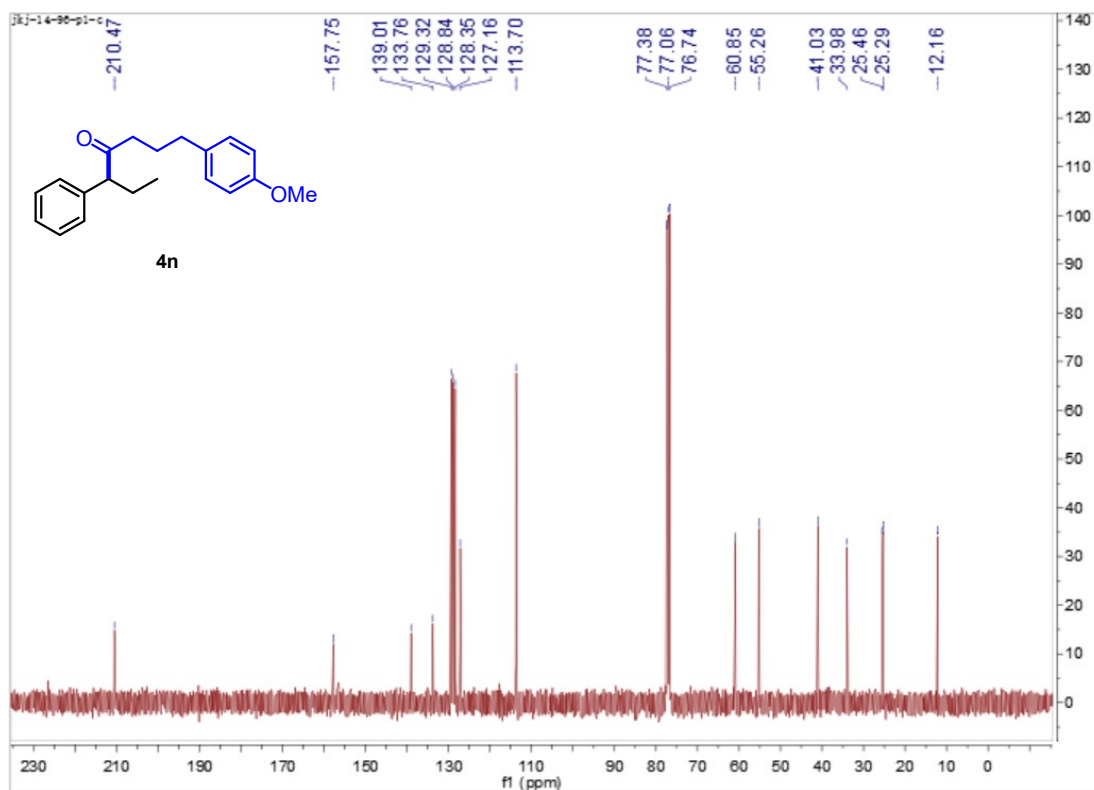
### <sup>13</sup>C NMR Spectrum of 4m (CDCl<sub>3</sub>, 101 MHz)



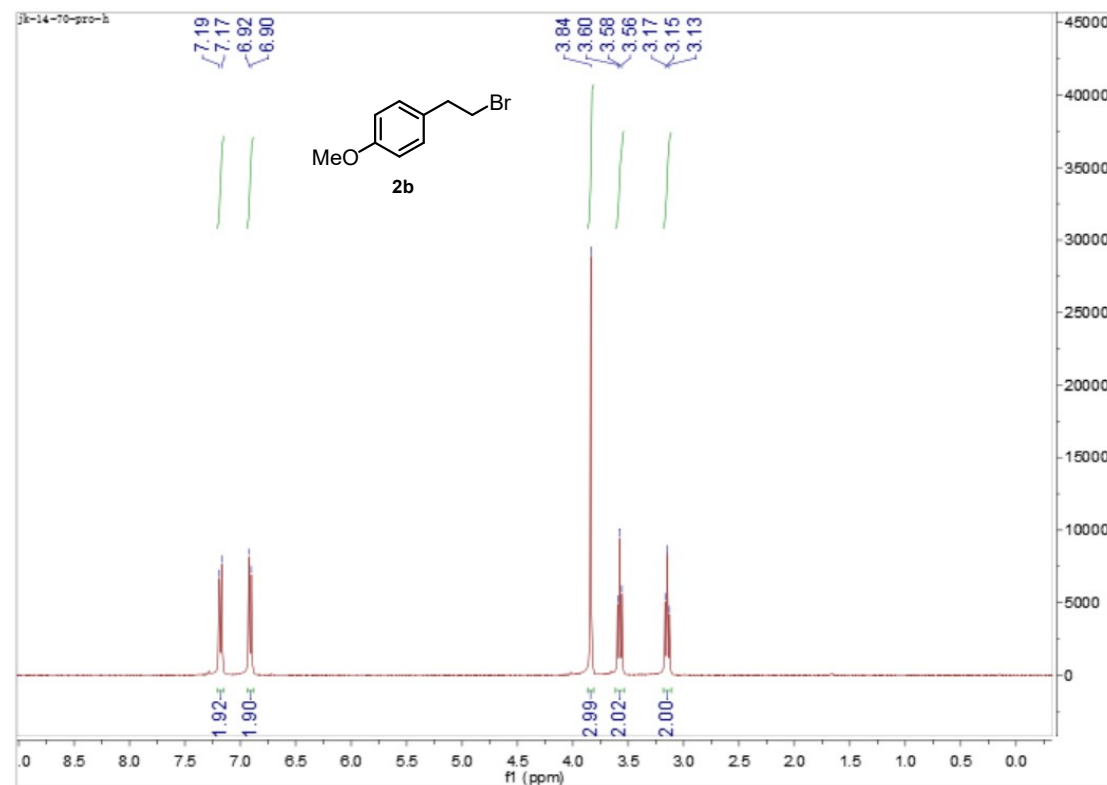
### <sup>1</sup>H NMR Spectrum of 4n (CDCl<sub>3</sub>, 400 MHz)



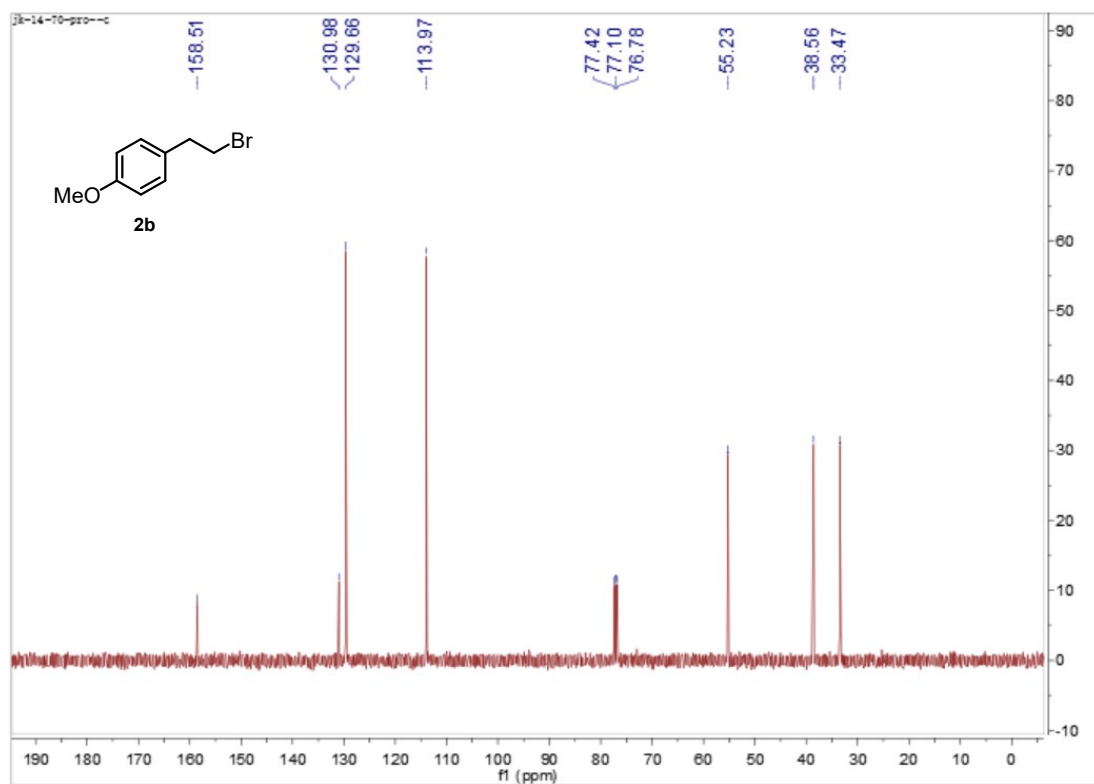
### <sup>13</sup>C NMR Spectrum of 4n (CDCl<sub>3</sub>, 101 MHz)



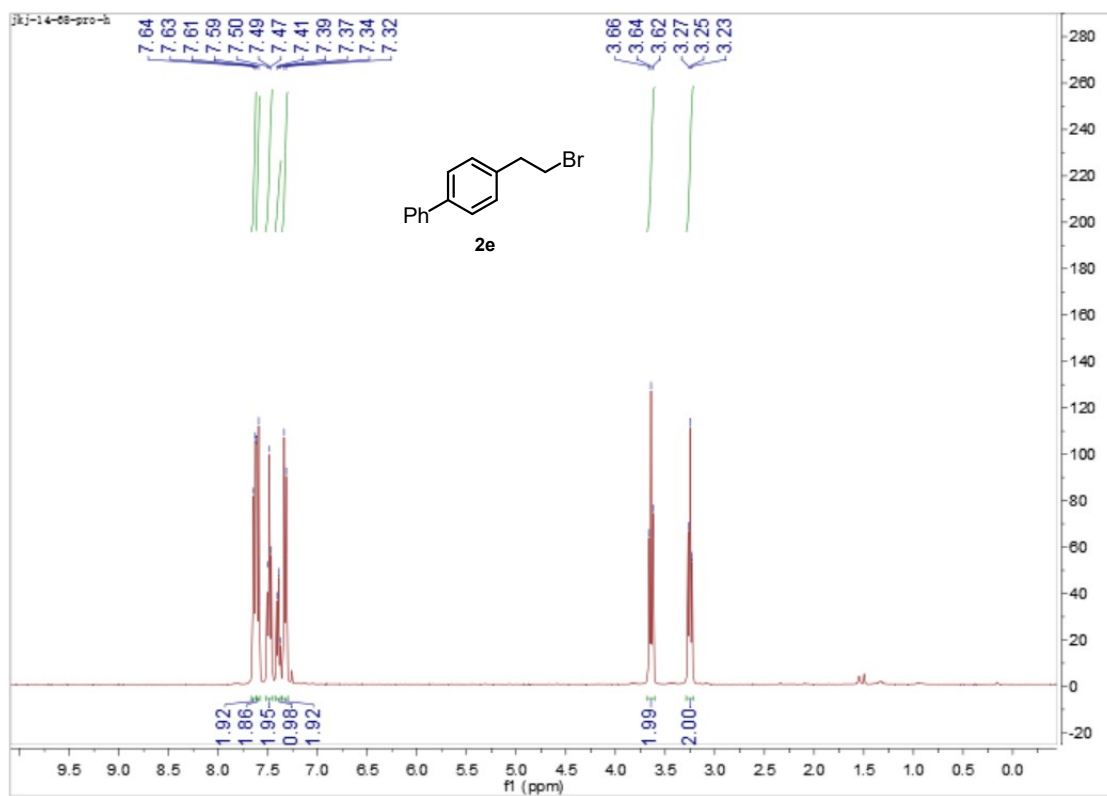
### <sup>1</sup>H NMR Spectrum of 2b (CDCl<sub>3</sub>, 400 MHz)



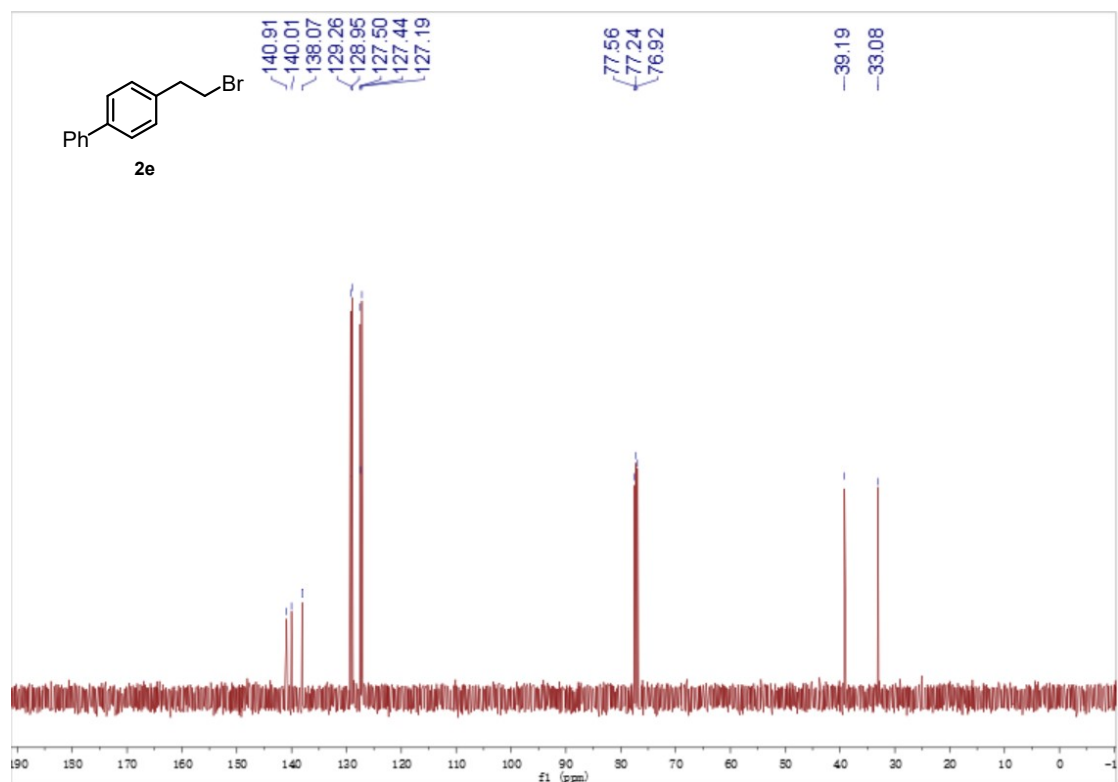
### $^{13}\text{C}$ NMR Spectrum of 2b ( $\text{CDCl}_3$ , 101 MHz)



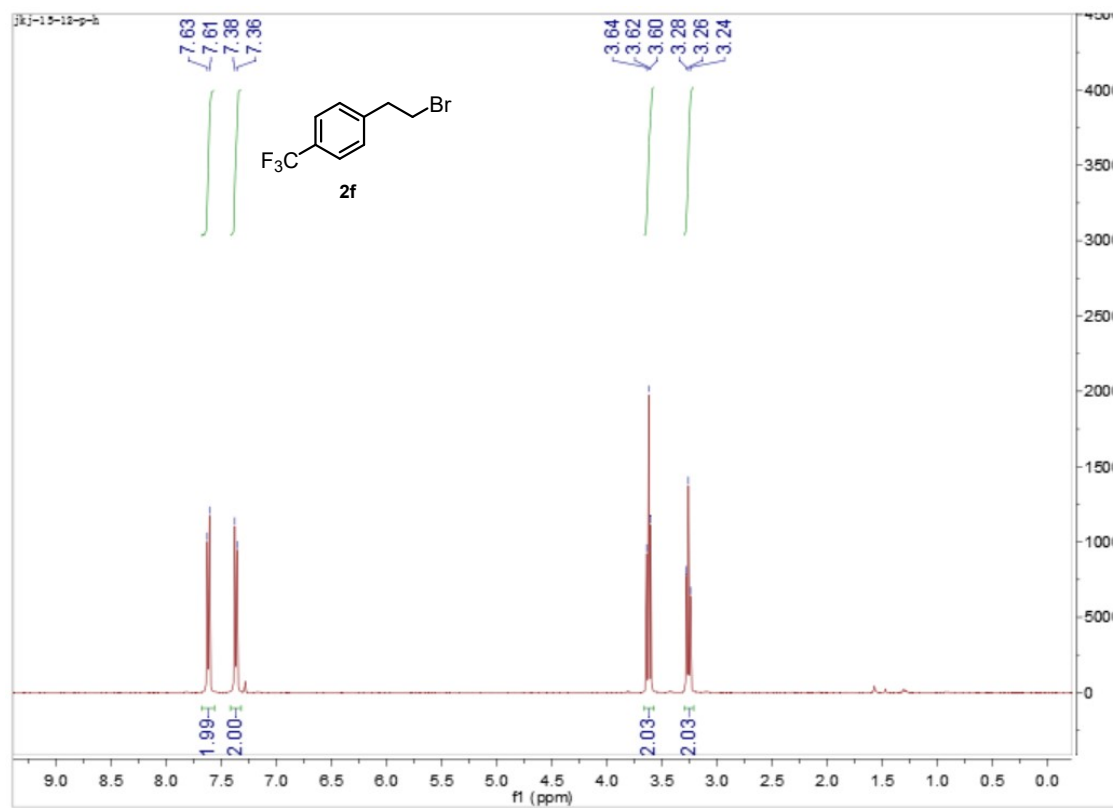
### $^1\text{H}$ NMR Spectrum of 2e ( $\text{CDCl}_3$ , 400 MHz)



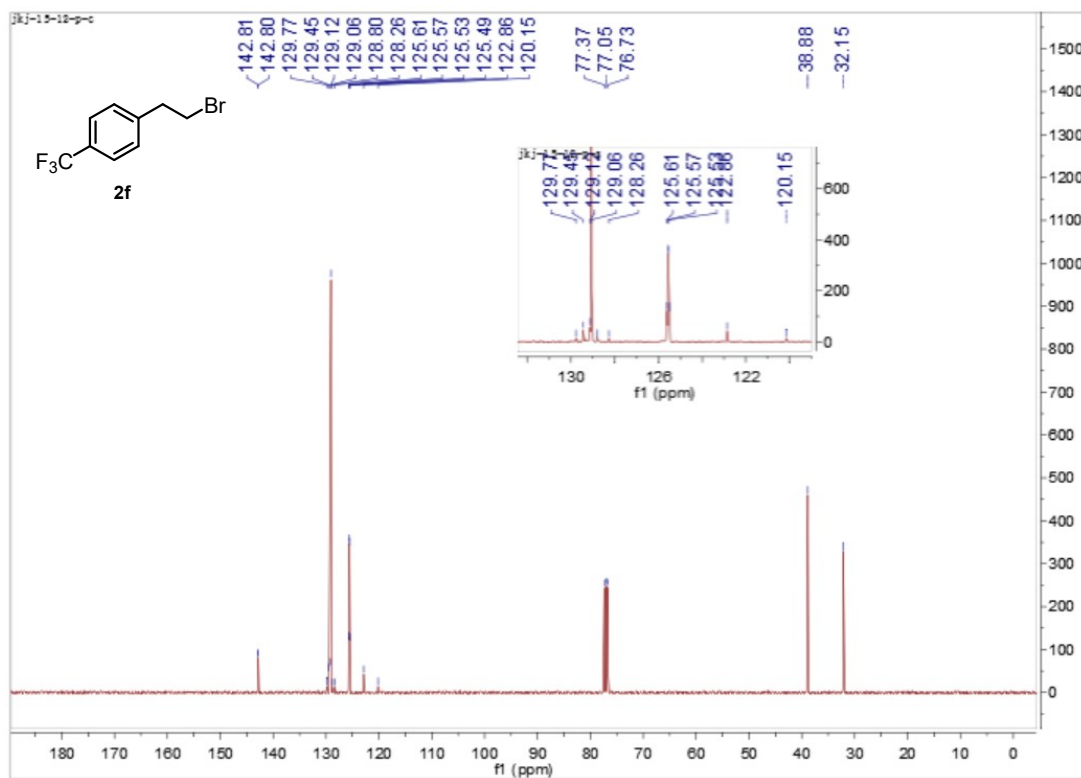
### <sup>13</sup>C NMR Spectrum of 2e (CDCl<sub>3</sub>, 101 MHz)



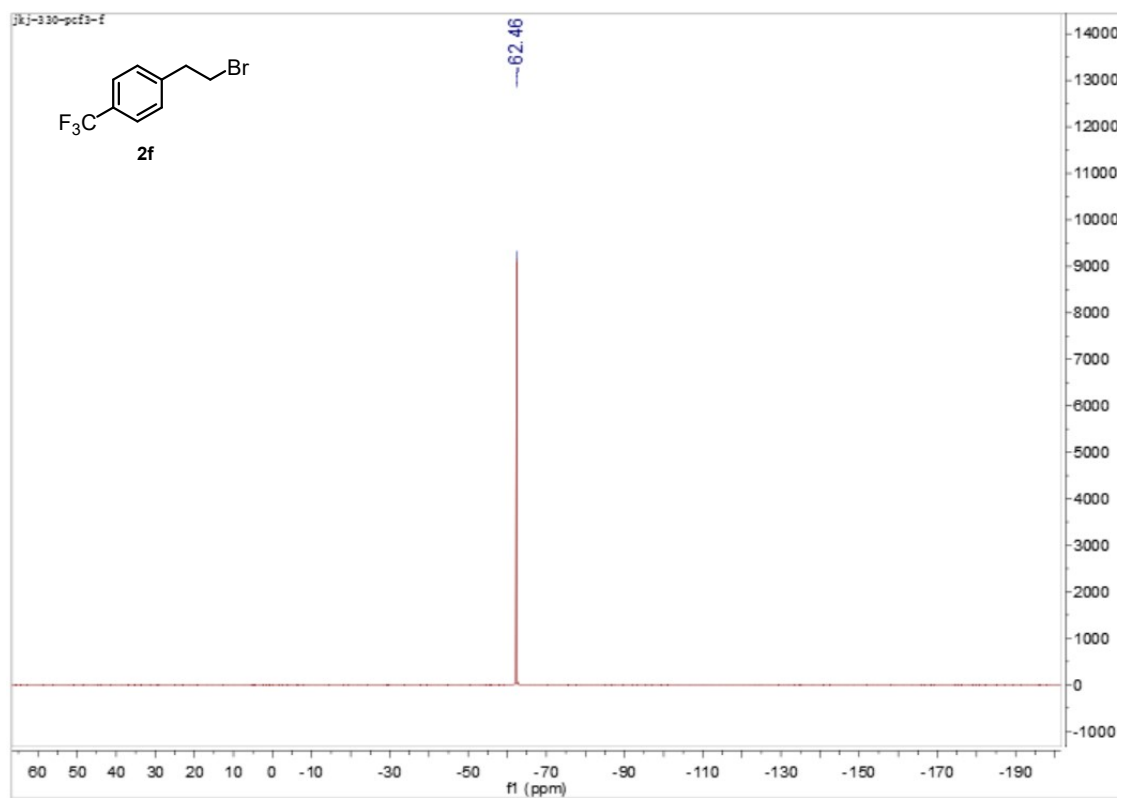
### <sup>1</sup>H NMR Spectrum of 2f (CDCl<sub>3</sub>, 400 MHz)



### <sup>13</sup>C NMR Spectrum of 2f (CDCl<sub>3</sub>, 101 MHz)

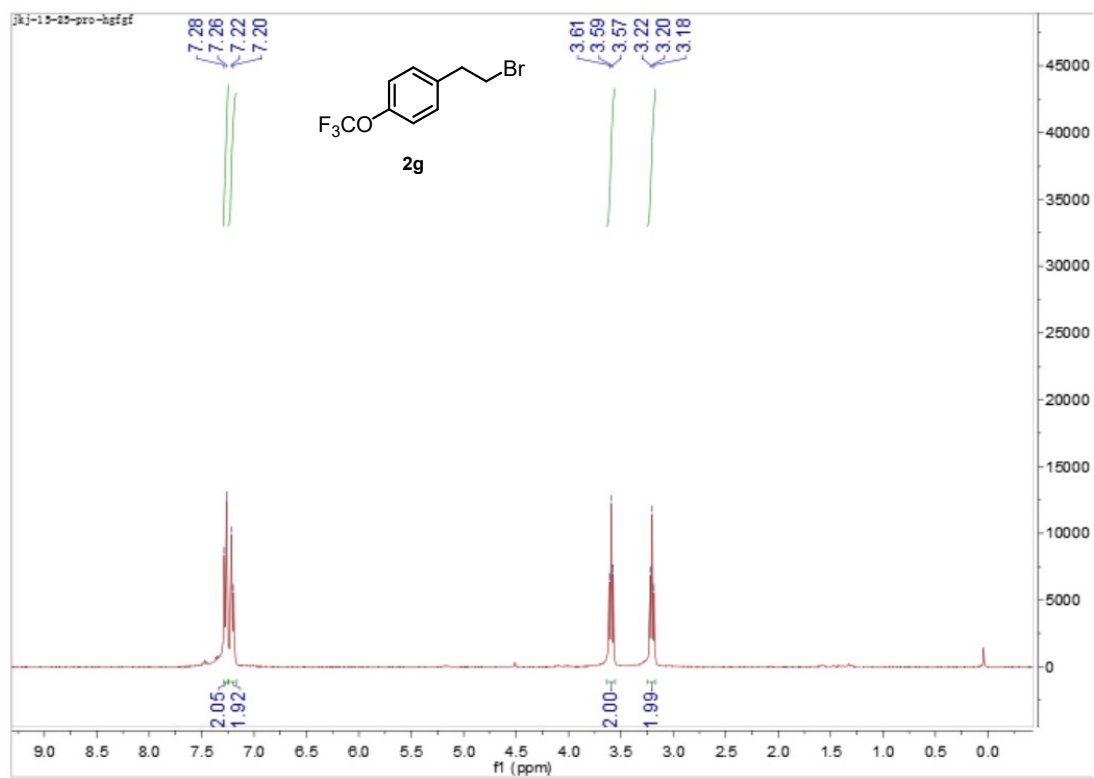


### <sup>19</sup>F NMR Spectrum of 2f (CDCl<sub>3</sub>, 376 MHz)

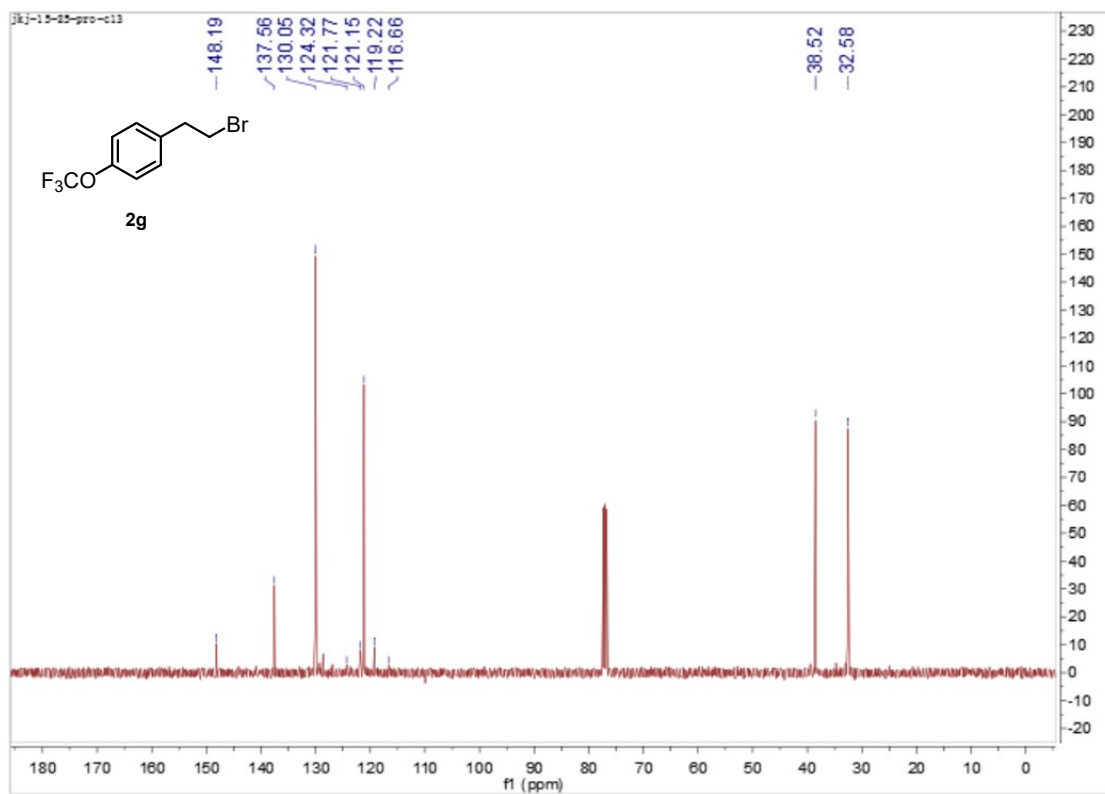




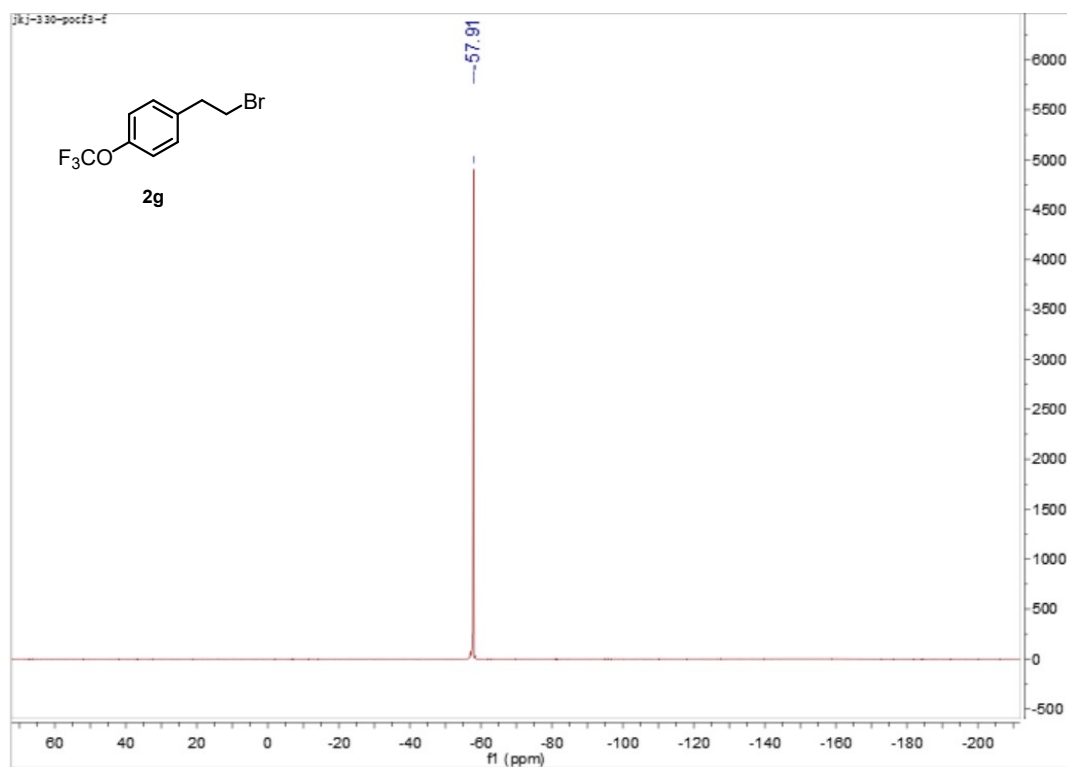
### <sup>1</sup>H NMR Spectrum of 2g (CDCl<sub>3</sub>, 400 MHz)



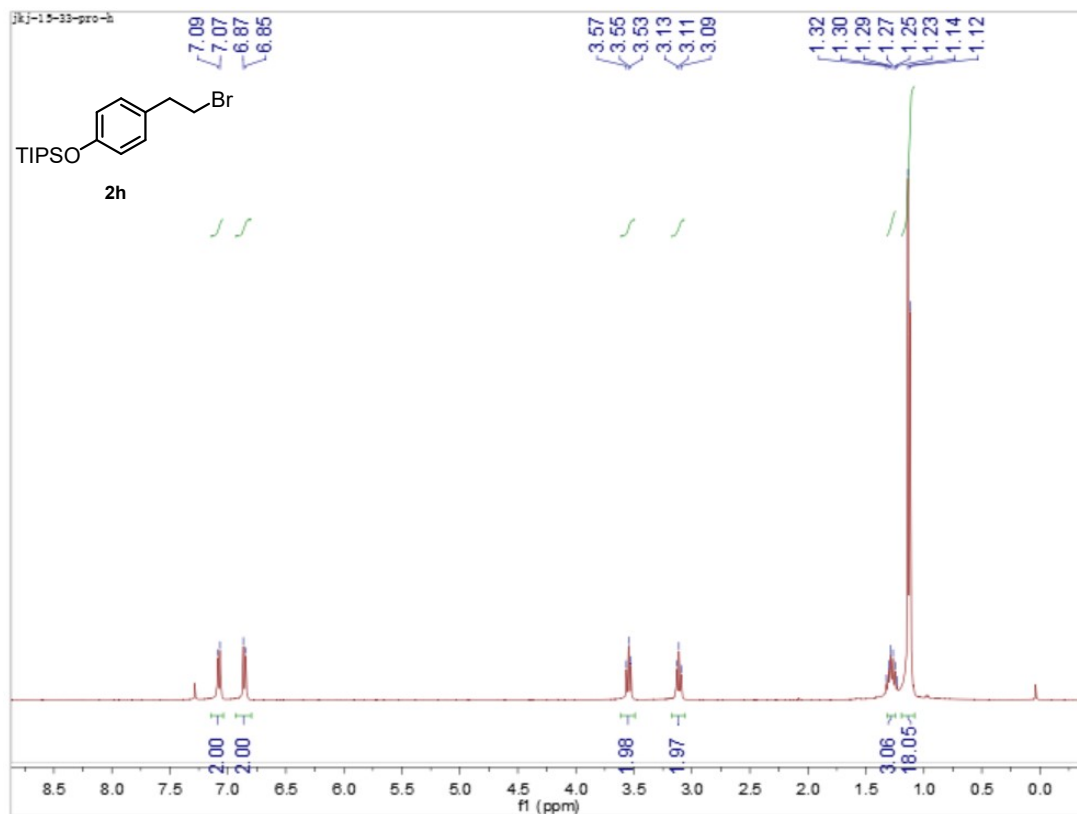
### <sup>13</sup>C NMR Spectrum of 2g (CDCl<sub>3</sub>, 101 MHz)



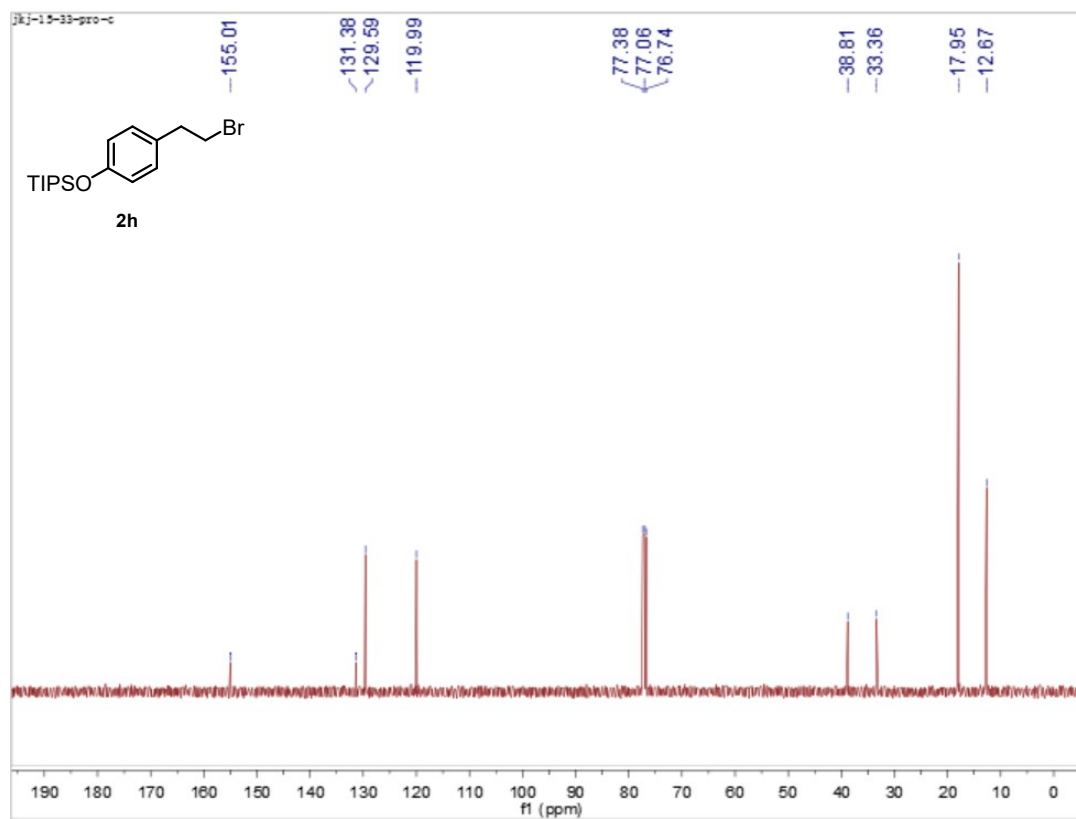
### <sup>19</sup>F NMR Spectrum of 2g (CDCl<sub>3</sub>, 376 MHz)



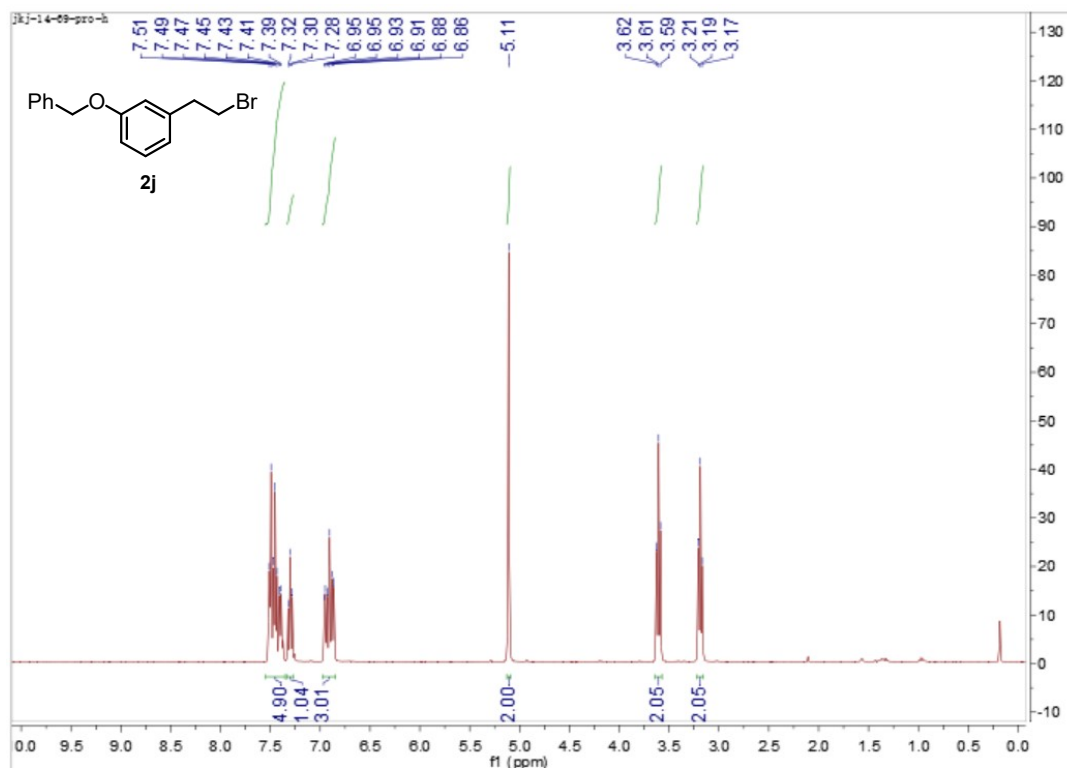
### <sup>1</sup>H NMR Spectrum of 2h (CDCl<sub>3</sub>, 400 MHz)



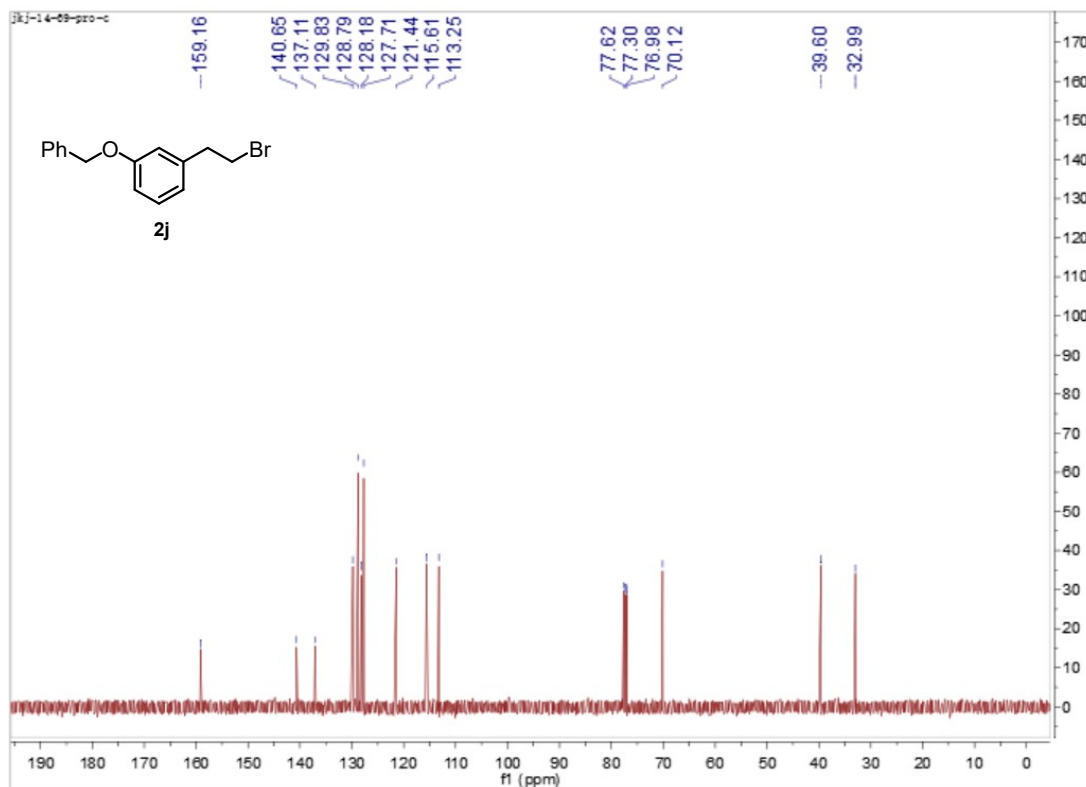
### <sup>13</sup>C NMR Spectrum of 2h (CDCl<sub>3</sub>, 101 MHz)



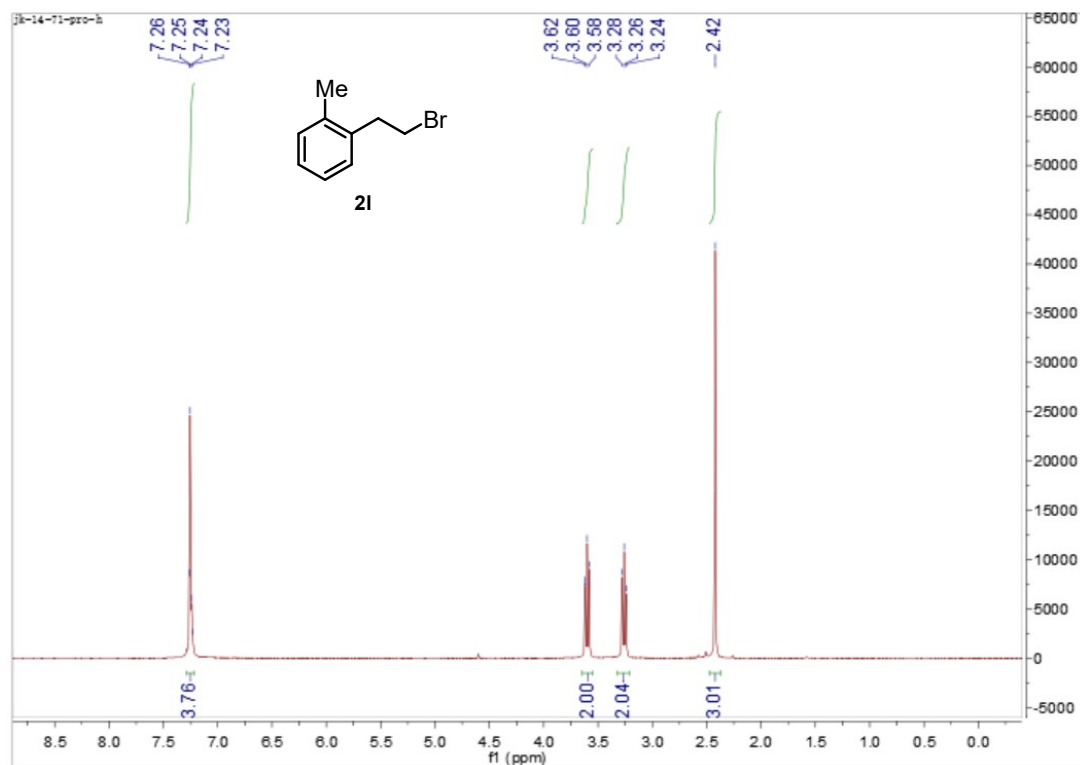
### <sup>1</sup>H NMR Spectrum of 2j (CDCl<sub>3</sub>, 400 MHz)



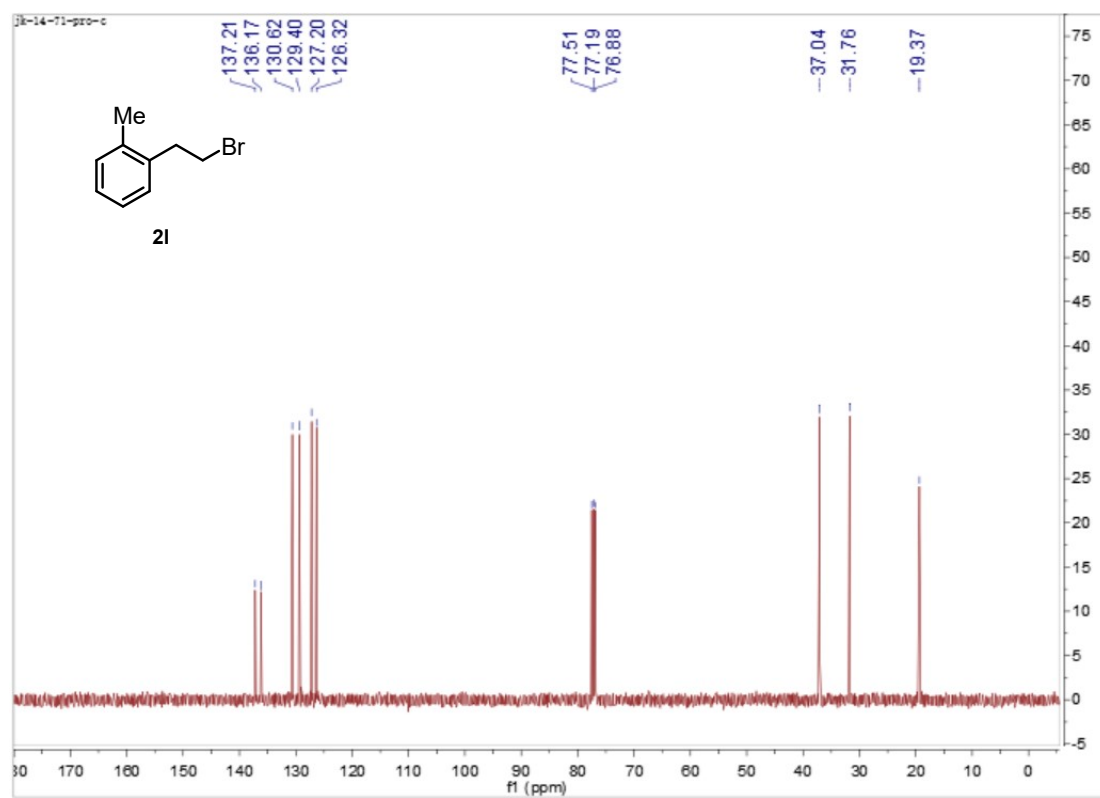
### <sup>13</sup>C NMR Spectrum of 2j (CDCl<sub>3</sub>, 101 MHz)



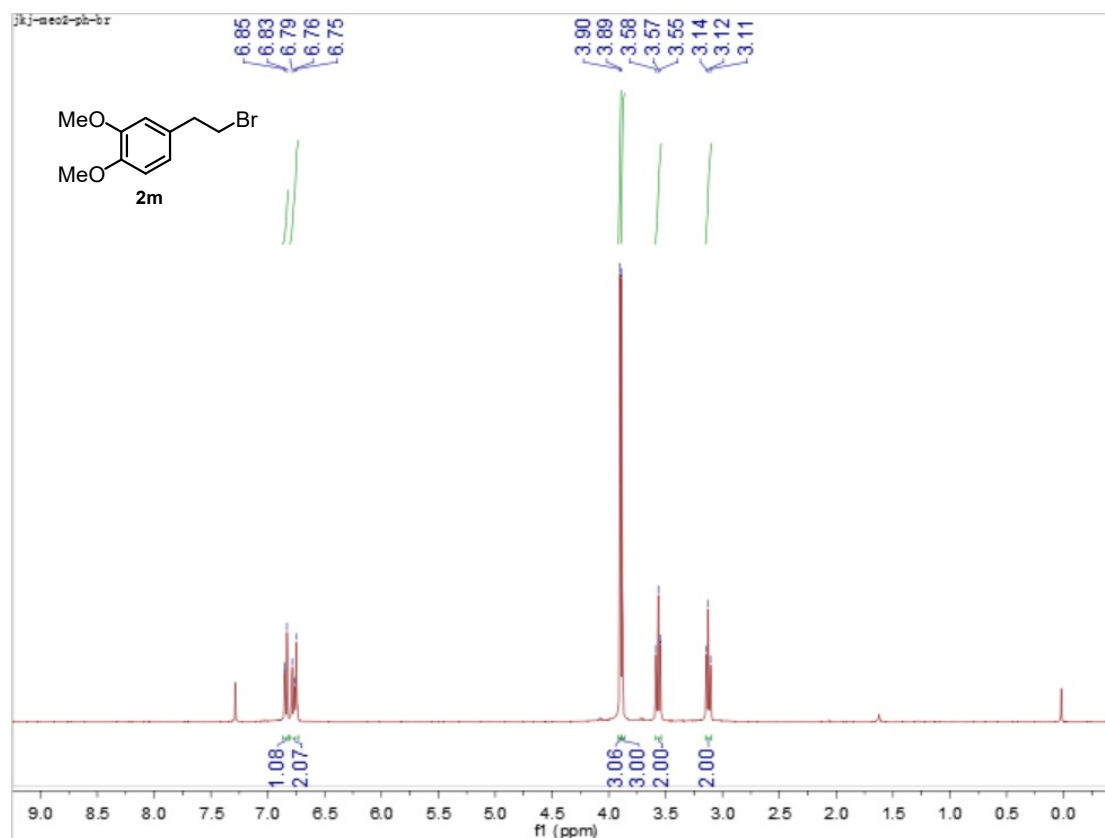
### <sup>1</sup>H NMR Spectrum of 2l (CDCl<sub>3</sub>, 400 MHz)



### <sup>13</sup>C NMR Spectrum of 2l (CDCl<sub>3</sub>, 101 MHz)



### <sup>1</sup>H NMR Spectrum of 2m (CDCl<sub>3</sub>, 400 MHz)



# <sup>13</sup>C NMR Spectrum of 2m (CDCl<sub>3</sub>, 101 MHz)

