

# Supporting Information

## Visible-Light-Driven Four-Component Diacylation of Styrenes with Acyl Azolium Salts: Carbonylative Synthesis of 1,4-Diketone

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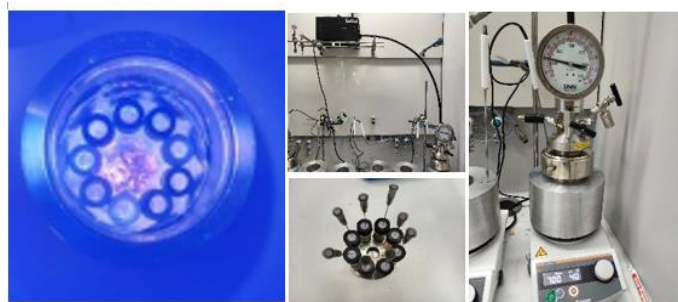
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# 1. General information

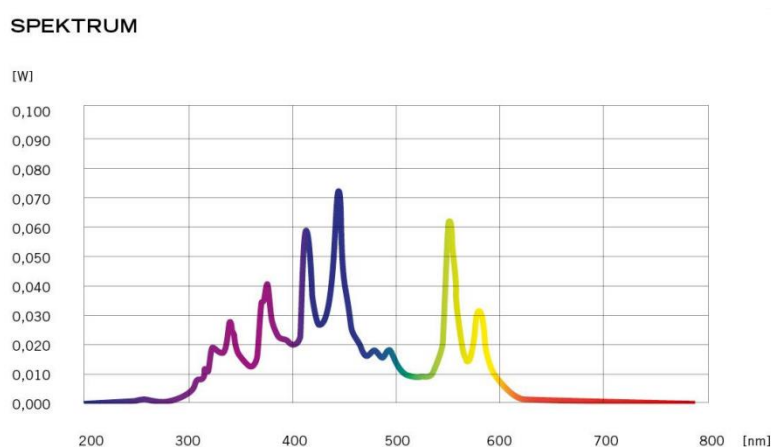
Unless otherwise noted, materials were purchased from commercial suppliers and used without further purification. Flash column chromatography was performed using 200-300 mesh silica gel.  $^1\text{H}$  NMR spectra were recorded on 300 or 400 MHz spectrophotometers. Chemical shifts are reported in ppm relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal standard ( $\text{CDCl}_3$ ;  $\delta = 7.26$  ppm).  $^{13}\text{C}$  NMR was recorded at 75 MHz or 101 MHz: chemical shifts are reported in ppm from tetramethylsilane (TMS) with the solvent resonance as the internal standard ( $\text{CDCl}_3$ ;  $\delta = 77.00$  ppm). Electron impact (EI) mass spectra were recorded on AMD 402 mass spectrometer (70 eV). High resolution mass spectra (HR-MS) were recorded on Agilent 6210. The data were given as mass units per charge ( $m/z$ ). Gas chromatography analysis was performed on an Agilent HP-5890 instrument with an FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d., 0.25  $\mu\text{m}$  film thickness) using argon as carrier gas.

The light source was positioned approximately 23 cm from the reaction vial, placed atop a manufactured autoclave (see **Figure 1**). For every reaction, the powerful light source, Portable Lumatec SUPERLITE S 04<sup>[3]</sup>, was utilized with different set filters: UV-A ( $\lambda = 400 - 500$  nm) at maximum intensity (100% power). **Figures 2** and **3** illustrate the relevant photophysical properties of the lamps.

Because of the high toxicity of carbon monoxide, all the reactions should be performed in an autoclave. The laboratory should be well-equipped with a CO detector and alarm system.



**Figure 1.** Apparatus for the Photoinduced Diacylation of Styrenes Using Carbon Monoxide as a Carbonyl Source to Synthesize 1,4-Diketones



**Figure 2.** Emission Spectrums of the Portable Lumatec SUPERLITE S 04

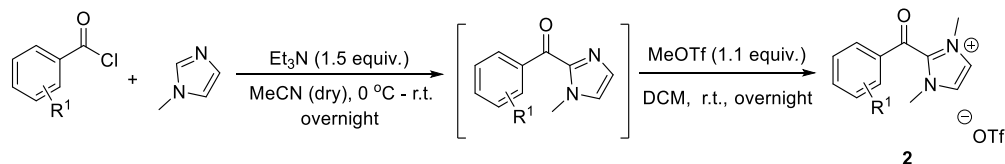
## OPTISCHE LEISTUNG

	Spektrum	Leistung	Intensität
UVA	320–400 nm	2.100 mW	10.500 mW/cm <sup>2</sup>
UVA + Blau	320–500 nm	6.900 mW	34.500 mW/cm <sup>2</sup>
Blau	400–500 nm	4.800 mW	24.000 mW/cm <sup>2</sup>
Weiß	400–700 nm	9.700 mW	48.500 mW/cm <sup>2</sup>
Violett	415 nm	2.000 mW	10.000 mW/cm <sup>2</sup>
Blau 440	440 nm	2.300 mW	11.500 mW/cm <sup>2</sup>
Blau 460	460 nm	2.000 mW	10.000 mW/cm <sup>2</sup>
Türkis	490 nm	1.200 mW	6.000 mW/cm <sup>2</sup>
Grün	550 nm	1.400 mW	7.000 mW/cm <sup>2</sup>
Gelb	570 nm	1.800 mW	9.000 mW/cm <sup>2</sup>

**Figure 3.** Technical Specifications of the Portable Lumatec SUPERLITE S 04

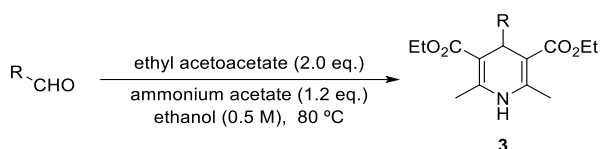
## 2. Preparation of substrates

### 2.1 Preparation of dimethylacylimidazoliums **2**



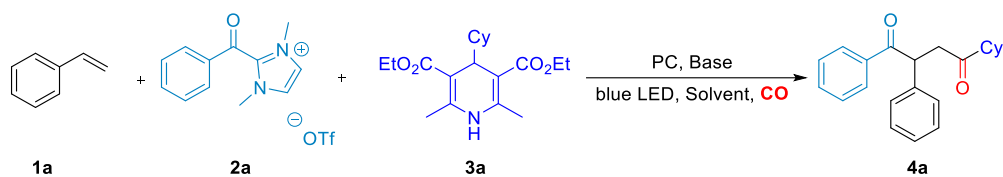
To a solution of 1-methylimidazole (411.0 mg, 350.0  $\mu$ L, 5.0 mmol, 1.0 equiv) and acid chloride derivatives (7.5 mmol, 1.5 equiv) in anhydrous acetonitrile (10.0 mL), triethylamine (0.76 g, 1.1 mL, 7.5 mmol, 1.5 equiv) was added dropwise at 0 °C, over 0.5 h. The reaction mixture was allowed to slowly warm to room temperature and stirred overnight. After the addition of water (10.0 mL), the phases were separated, and the aqueous layer was extracted with ethyl acetate (3 $\times$ 10.0 mL). The combined organic phases were washed with saturated sodium chloride solution (10.0 mL), dried over anhydrous sodium sulfate, and concentrated in vacuo. The obtained crude intermediate product, N-methyl-C2-acyl imidazoles, does not need to be purified and is directly used in the next step of the reaction. To a solution of the obtained crude intermediate product, N-methyl-C2-acyl imidazoles, in dichloromethane (10.0 mL) was added methyl triflate (900.0 mg, 622.0  $\mu$ L, 5.5 mmol, 1.1 equiv) at room temperature, and the reaction mixture was stirred overnight. The solvent was evaporated in vacuo, and the white precipitate produced by adding diethyl ether was filtered out and washed with diethyl ether (3 $\times$ 10.0 mL). The acyl azolium salts **2** was obtained by vacuum drying. GC Mass and Spectroscopic data in agreement with that reported previously.<sup>[1]</sup>

### 2.2 Preparation of Hantzsch Ester **3**



A reaction flask was charged with ethyl acetoacetate (1.3 g, 10.0 mmol, 2.0 equiv.), ammonium acetate (0.5 g, 1.2 equiv.), ethanol (10.0 mL). To the above solution, the aldehyde (5.0 mmol) was added slowly. After addition, the system was heated at 80 °C with stirring. The reaction was monitored by TLC. When the reaction was completed, the solvent was evaporated, vacuum the crude product, half an hour later add petroleum ether ultrasound, to give the corresponding Hantzsch ester. GC Mass and Spectroscopic data in agreement with that reported previously.<sup>[2]</sup>

### 3. Complementary reaction optimization Data



**Table 1. Optimization of pressure (CO)**

Entry	pressure(bar)	Yield (%)
1	30	10
2	40	32
3	50	25
4	60	28

Reaction conditions: **1a** (0.1 mmol), **2a** (0.1 mmol), **3a** (0.1 mmol), MeCN (1.0 mL), Na<sub>2</sub>CO<sub>3</sub> (0.2 mmol), [Ir]PF<sub>6</sub> (2 mol%), r.t., 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard.

**Table 2. Optimization of equivalent ratio**

Entry	<b>1a : 2a : 3a</b>	Yield (%)
1	1.0 : 1.0 : 1.0	32
2	1.0 : 1.5 : 1.0	20
3	1.0 : 1.0 : 1.5	35
4	1.0 : 1.5 : 1.5	40
5	1.0 : 2.0 : 2.0	34
6	1.0 : 1.5 : 2.0	32

Reaction conditions: CO (40 bar), MeCN (1.0 mL), Na<sub>2</sub>CO<sub>3</sub> (0.2 mmol), [Ir]PF<sub>6</sub> (2 mol%), r.t., 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard.

**Table 3. Optimization of temperature**

Entry	Temperature (°C)	Yield (%)
1	r.t.	40
2	40	48
3	50	30
4	60	trace

Reaction conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), **3a** (0.15 mmol), CO (40 bar), MeCN (1.0 mL), Na<sub>2</sub>CO<sub>3</sub> (0.2 mmol), [Ir]PF<sub>6</sub> (2 mol%), 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard.

**Table 4 Optimization of photosensitizer**

Entry	PC	Yield (%)
1 <sup>b</sup>	[Ir]PF <sub>6</sub>	48
2 <sup>b</sup>	4CzIPN	18
3 <sup>b</sup>	[Ir]PN <sub>6</sub>	trace
4 <sup>b</sup>	Eosin Y	trace
5 <sup>b</sup>	Rh(dppe) <sub>2</sub> BF <sub>4</sub>	10
6 <sup>b</sup>	Ru(bpy) <sub>3</sub> Cl <sub>2</sub> ·6H <sub>2</sub> O	trace
7 <sup>b</sup>	Co(dmgh) <sub>2</sub> Cl <sub>2</sub>	trace
8 <sup>b</sup>	Ir(ppy) <sub>3</sub>	35
1 <sup>a</sup>	[Ir]PF <sub>6</sub>	33
1 <sup>c</sup>	[Ir]PF <sub>6</sub>	40

Reaction conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), **3a** (0.15 mmol), CO (40 bar), MeCN (1.0 mL), Na<sub>2</sub>CO<sub>3</sub> (0.2 mmol), PC (1 mol%<sup>a</sup>, 2 mol%<sup>b</sup>, 3 mol%<sup>c</sup>), 40 °C, 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard.

**Table 5. Optimization of solvent**

Entry	Solvent	Yield (%)
1 <sup>a</sup>	MeCN	48
2 <sup>a</sup>	THF	32
3 <sup>a</sup>	DMF	trace
4 <sup>a</sup>	DMAc	10
5 <sup>a</sup>	DMSO	-
6 <sup>a</sup>	PhCF <sub>3</sub>	trace
7 <sup>a</sup>	Toluene	trace
8 <sup>a</sup>	MeOH	-
9 <sup>b</sup>	MeCN	41
10 <sup>c</sup>	MeCN	30

Reaction conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), **3a** (0.15 mmol), CO (40 bar), Solvent (0.1M<sup>a</sup>, 0.2M<sup>b</sup>, 0.05 M<sup>c</sup>), Na<sub>2</sub>CO<sub>3</sub> (0.2 mmol), [Ir]PF<sub>6</sub> (2 mol%), 40 °C, 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard.

**Table 6. Optimization of base**

Entry	Base	Yield (%)
1 <sup>b</sup>	Na <sub>2</sub> CO <sub>3</sub>	48
2 <sup>b</sup>	K <sub>2</sub> CO <sub>3</sub>	10
3 <sup>b</sup>	CS <sub>2</sub> CO <sub>3</sub>	trace
4 <sup>b</sup>	Li <sub>2</sub> CO <sub>3</sub>	13
5 <sup>b</sup>	NaHCO <sub>3</sub>	32
6 <sup>b</sup>	KHCO <sub>3</sub>	20
7 <sup>b</sup>	K <sub>2</sub> HPO <sub>4</sub>	21
8 <sup>b</sup>	KH <sub>2</sub> PO <sub>4</sub>	8
9 <sup>b</sup>	Na <sub>2</sub> HPO <sub>4</sub>	19
10 <sup>b</sup>	NaH <sub>2</sub> PO <sub>4</sub>	24
11 <sup>b</sup>	Na <sub>3</sub> PO <sub>4</sub>	60
12 <sup>b</sup>	<sup>t</sup> BuONa	trace
13 <sup>b</sup>	DBU	-
14 <sup>b</sup>	NEt <sub>3</sub>	-
15 <sup>b</sup>	No	-

16 <sup>c</sup>	Na <sub>3</sub> PO <sub>4</sub>	68
17 <sup>d</sup>	Na <sub>3</sub> PO <sub>4</sub>	50
18 <sup>a</sup>	Na <sub>3</sub> PO <sub>4</sub>	35

Reaction conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), **3a** (0.15 mmol), CO (40 bar), MeCN (1.0 mL), Base (0.1 mmol <sup>a</sup>, 0.2 mmol <sup>b</sup>, 0.3 mmol <sup>c</sup>, 0.4 mmol <sup>d</sup>), [Ir]PF<sub>6</sub> (2 mol%), 40 °C, 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard.

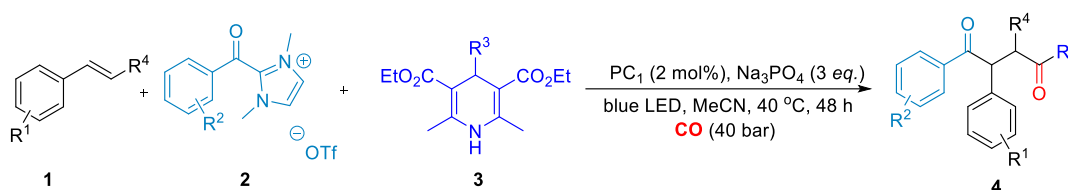
**Table 7. Optimization of time**

Entry	Time (h)	Yield (%)
1	12	40
2	20	50
3	24	68
4	36	70
5	48	85 (78)
6	55	83

Reaction conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), **3a** (0.15 mmol), CO (40 bar), MeCN (1.0 mL), Na<sub>3</sub>PO<sub>4</sub> (0.3 mmol), [Ir]PF<sub>6</sub> (2 mol%), 40 °C, 400-500 nm. Determined by GC with hexadecane as internal standard. Isolated yield is shown in parentheses.

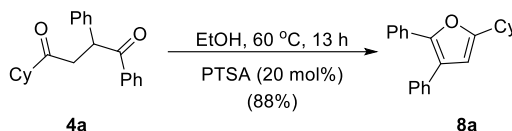
## 4. Characterization and procedure of 1,4-dione products 4 and synthetic transformations products 8

### 4.1 General diacylation procedure for the synthesis of 1,4-diones 4



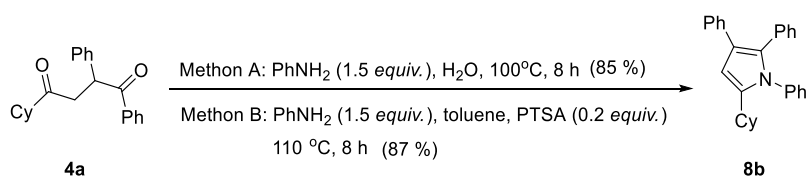
A 4 mL screw-cap vial was charged with Hantzsch ester **3** (1.5 equiv), acyl azolium salts **2** (1.5 equiv),  $[\text{Ir}]\text{PF}_6$  (4.4 mg, 2 mol%), and an oven-dried stirring bar. The vial was closed with a Teflon septum and cap and connected to the atmosphere via a needle. After replacing the nitrogen in the vial three times, acetonitrile (2 mL) was added. Then, styrenes (0.2 mmol, 1.0 equiv) was added using a microinjector. The vial was then moved to a cannula and transferred into a 300 mL photoautoclave (manufactured by Parr Instrument Company®), under a nitrogen atmosphere. At room temperature, the autoclave was washed with CO three times and charged with 40 bar of CO. The autoclave was placed on a heating plate equipped with a magnetic stirrer and an aluminum block. The reaction mixture was allowed to react at 40 °C under UV-A (400-500 nm) for 48 hours. After the reaction was complete, the pressure of the autoclave was carefully released, and the residual CO was washed away with nitrogen. The solvent was removed under vacuum, and the product was purified by column chromatography on silica gel using petroleum ether and ethyl acetate (30:1 - 20:1) to afford the corresponding product **4**.

### 4.2 General procedure for the synthesis of 5-cyclohexyl-2,3-diphenylfuran **8a**<sup>[6]</sup>



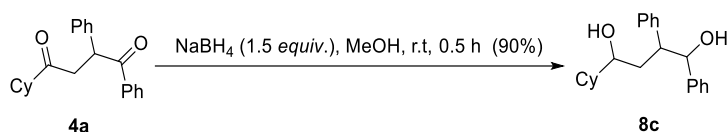
To a stirred solution of **4a** (32.0 mg, 0.1 mmol) in toluene (2 mL) at room temperature was added *p*-toluenesulfonic acid (3.5 mg, 0.02 mmol). The mixture was stirred vigorously for 13 hours at 60 °C. The crude product was purified by column chromatography on silica gel using petroleum ether and ethyl acetate (50:1) as to afford **8a** (26.6 mg, 88% yield) as a yellow solid.

### 4.3 General procedure for the synthesis of 5-cyclohexyl-1,2,3-triphenyl-1H-pyrrole **8b**<sup>[7]</sup>



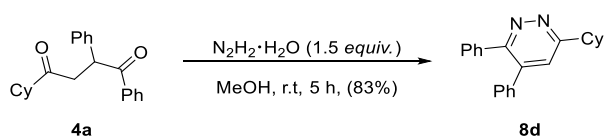
Methon A: The mixture of **4a** (32.0 mg, 0.1 mmol) and aniline (14.0 mg, 0.15 mmol) in water (1 mL) at 100 °C for 8 hours; Methon B: The mixture of **4a** (32.0 mg, 0.1 mmol), *p*-toluenesulfonic acid (3.5 mg, 0.02 mmol) and aniline (14.0 mg, 0.15 mmol) in toluene (1 mL) at 110 °C for 8 hours. The mixture was cooled to room temperature and diluted with ethyl acetate (1 mL) and washed with saturated  $\text{NaHCO}_3$  solution (1 mL) and saturated  $\text{NaCl}$  solution. The organic phase was dried over anhydrous magnesium sulfate and concentrated. The product was purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate (50:1) as eluent to give **8b** (32.1 mg, 85% yield<sup>A</sup>; 32.8 mg, 87% yield<sup>B</sup>) as a brown solid.

### 4.4 General procedure for the synthesis of 4-cyclohexyl-1,2-diphenylbutane-1,4-diol **8c**<sup>[8]</sup>



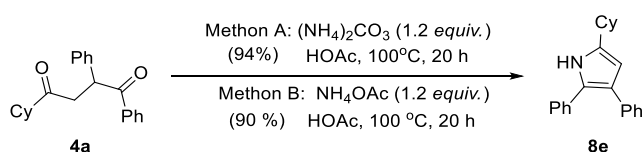
The mixture of **4a** (32.0 mg, 0.1 mmol) was added dropwise to a solution of sodium borohydride (6.0 mg, 0.15 mmol, 1.5 equiv) in MeOH (1 mL) at 0 °C for 1 hour, then room temperature for 2 hours. The reaction was diluted with ethyl acetate (1 mL), quenched with saturated  $\text{NH}_4\text{Cl}$  solution (1 mL), washed by saturated  $\text{NaCl}$  solution (1 mL). The organic phase was dried over anhydrous magnesium sulfate and concentrated. The product was purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate (20:1) as eluent to give **8c** (29.2 mg, 90% yield) as a white solid.

### 4.5 General procedure for the synthesis of 6-cyclohexyl-3,4-diphenylpyridazine **8d**<sup>[9]</sup>

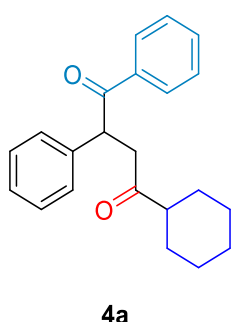


**4a** (32.0 mg, 0.1 mmol) was dissolved in hydrazine hydrate (15.0 uL, 0.15 mmol, 1.5 equiv) and MeOH (1 mL) was added. The reaction mixture was stirred at room temperature for 5 hours. The crude product was purified by flash chromatography on silica gel with petroleum ether/ethyl acetate (30:1) as eluent to afford **8d** (26.0 mg, 83% yield) as a yellow solid.

## 4.6 General procedure for the synthesis of 5-cyclohexyl-2,3-diphenyl-1H-pyrrole **8e**<sup>[10]</sup>



Methon A: The solution of **4a** (32.0 mg, 0.1 mmol) and  $(\text{NH}_4)_2\text{CO}_3$  (12.0 mg, 0.12 mmol) in acetic acid (1 mL) at 100 °C for 20 hours; Methon B: The solution of **4a** (32.0 mg, 0.1 mmol) and  $\text{NH}_4\text{OAc}$  (9.3 mg, 0.12 mmol) in acetic acid (1 mL) at 100 °C for 20 hours. The reaction mixture was diluted with ethyl acetate (1 mL) and washed with saturated  $\text{NaHCO}_3$  solution (1 mL). The organic phase was dried over magnesium sulfate and concentrated. The product was purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate (50:1) as eluent to give **8e** (28.2 mg, 95% yield<sup>A</sup>; 27.0 mg, 90% yield<sup>B</sup>) as a white solid.



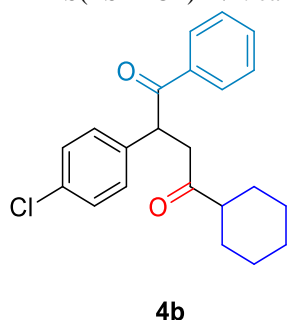
### 4-cyclohexyl-1,2-diphenylbutane-1,4-dione (**4a**)

Chromatography Pentane/EA = 20:1 (v/v), 50.0 mg (78%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.98 – 7.17 (m, 10H), 5.12 (dd,  $J$  = 10.1, 3.9 Hz, 1H), 3.63 (dd,  $J$  = 17.9, 10.1 Hz, 1H), 2.76 (dd,  $J$  = 17.9, 3.9 Hz, 1H), 2.43 – 2.33 (m, 1H), 1.95 – 1.15 (m, 10H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  212.1, 199.1, 138.7, 136.4, 132.8, 129.1, 128.8, 128.4, 128.1, 127.2, 50.7, 48.5, 45.4, 28.3, 25.8, 25.6, 25.6.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{22}\text{H}_{24}\text{O}_2$  343.1668, found: 343.1667.



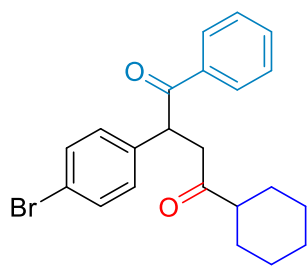
### 2-(4-chlorophenyl)-4-cyclohexyl-1-phenylbutane-1,4-dione (**4b**)

Chromatography Pentane/EA = 20:1 (v/v), 42.5 mg (60%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.88 – 7.11 (m, 9H), 5.04 (dd,  $J$  = 9.8, 4.2 Hz, 1H), 3.51 (dd,  $J$  = 18.0, 9.9 Hz, 1H), 2.68 (dd,  $J$  = 18.0, 4.2 Hz, 1H), 2.35 – 2.23 (m, 1H), 1.88 – 1.11 (m, 10H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  211.8, 198.8, 137.2, 136.1, 133.2, 133.01, 129.5, 129.2, 128.8, 128.5, 50.6, 47.7, 45.2, 28.3, 25.8, 25.6, 25.5.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{22}\text{H}_{23}\text{ClO}_2$  377.1279, found: 377.1284.



**4c**

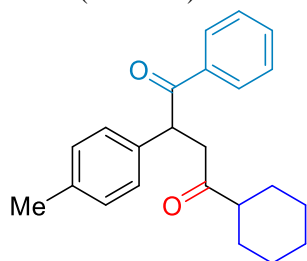
### 2-(4-bromophenyl)-4-cyclohexyl-1-phenylbutane-1,4-dione (4c)

Chromatography Pentane/EA = 20:1 (v/v), 45.6 mg (57%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.89 – 7.04 (m, 9H), 5.02 (dd,  $J = 9.8, 4.1$  Hz, 1H), 3.51 (dd,  $J = 18.0, 9.8$  Hz, 1H), 2.67 (dd,  $J = 17.9, 4.2$  Hz, 1H), 2.35 – 2.23 (m, 1H), 1.88 – 1.07 (m, 10H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  211.7, 198.7, 137.7, 136.1, 133.0, 132.2, 129.8, 128.8, 128.5, 121.3, 50.6, 47.8, 45.2, 28.3, 25.8, 25.6, 25.5.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{22}\text{H}_{23}\text{BrO}_2$  421.0773, found: 421.0777.



**4d**

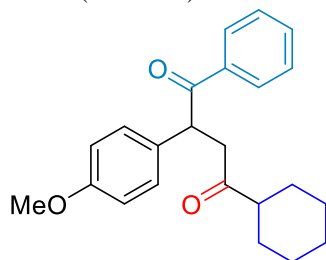
### 4-cyclohexyl-1-phenyl-2-(p-tolyl)butane-1,4-dione (4d)

Chromatography Pentane/EA = 20:1 (v/v), 43.4 mg (65%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.90 – 6.98 (m, 9H), 5.01 (dd,  $J = 10.1, 3.9$  Hz, 1H), 3.53 (dd,  $J = 17.9, 10.1$  Hz, 1H), 2.65 (dd,  $J = 17.9, 3.9$  Hz, 1H), 2.31 – 2.22 (m, 1H), 2.19 (s, 3H), 1.89 – 1.11 (m, 10H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  212.2, 199.1, 136.9, 136.4, 135.6, 132.7, 129.8, 128.8, 128.4, 127.9, 50.6, 48.1, 45.4, 28.3, 28.3, 25.8, 25.6, 25.6, 21.0.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{23}\text{H}_{26}\text{O}_2$  357.1825, found: 357.1826.



**4e**

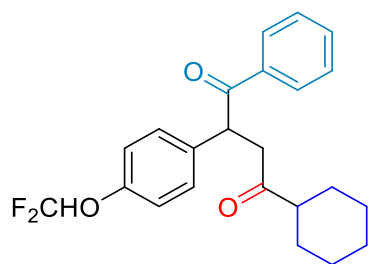
### 4-cyclohexyl-2-(4-methoxyphenyl)-1-phenylbutane-1,4-dione (4e)

Chromatography Pentane/EA = 30:1 (v/v), 42.8 mg (61%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.90 – 6.70 (m, 9H), 5.00 (dd,  $J = 10.0, 4.1$  Hz, 1H), 3.66 (s, 3H), 3.51 (dd,  $J = 17.9, 10.0$  Hz, 1H), 2.67 (dd,  $J = 17.9, 4.1$  Hz, 1H), 2.34 – 2.25 (m, 1H), 1.87 – 1.12 (m, 10H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  212.3, 199.3, 158.7, 136.5, 132.7, 130.6, 129.1, 128.8, 128.4, 114.5, 55.2, 50.7, 47.6, 45.4, 28.3, 28.3, 25.8, 25.6, 25.6.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{23}\text{H}_{26}\text{O}_3$  351.1995, found: 351.1960.



**4f**

**4-cyclohexyl-2-(4-(difluoromethoxy)phenyl)-1-phenylbutane-1,4-dione (4f)**

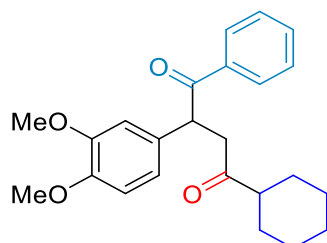
Chromatography Pentane/EA = 30:1 (v/v), 37.8 mg (49%), white solid.

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.90 – 6.93 (m, 9H), 6.37 (t, *J* = 73.8, 1H), 5.06 (dd, *J* = 9.9, 4.1 Hz, 1H), 3.53 (dd, *J* = 18.0, 9.9 Hz, 1H), 2.69 (dd, *J* = 18.0, 4.1 Hz, 1H), 2.35 – 1.09 (m, 10H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 211.9, 198.9, 150.2, 136.2, 135.8, 133.0, 129.5, 128.8, 128.5, 120.1, 119.2, 115.7, 112.3, 50.6, 47.6, 45.3, 28.3, 25.8, 25.6, 25.5.

<sup>19</sup>F NMR (282 MHz, CDCl<sub>3</sub>) δ -80.76, -81.02.

HRMS(ESI-TOF) *m/z*: calcd for [M<sup>+</sup>]H<sup>+</sup> C<sub>23</sub>H<sub>24</sub>F<sub>2</sub>O<sub>3</sub> 387.1766, found: 387.1770.



**4g**

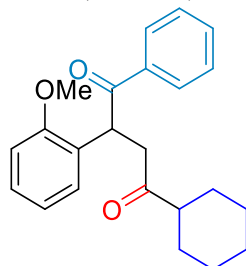
**4-cyclohexyl-2-(3,4-dimethoxyphenyl)-1-phenylbutane-1,4-dione (4g)**

Chromatography Pentane/EA = 30:1 (v/v), 32.6 mg (43%), white solid.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.98 – 7.35 (m, 5H), 6.81 (dd, *J* = 8.3, 2.0 Hz, 1H), 6.77 (s, 1H), 6.74 (d, *J* = 2.1 Hz, 1H), 5.06 (dd, *J* = 9.9, 4.0 Hz, 1H), 3.83 (s, 3H), 3.81 (s, 3H), 3.60 (dd, *J* = 17.9, 10.0 Hz, 1H), 2.75 (dd, *J* = 17.9, 4.0 Hz, 1H), 2.41 – 2.35 (m, 1H), 1.94 – 1.16 (m, 10H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 212.3, 199.3, 149.3, 148.2, 136.5, 132.8, 131.1, 128.8, 128.4, 120.5, 111.6, 110.8, 55.90, 55.8, 50.7, 48.1, 45.5, 28.4, 28.3, 25.8, 25.6, 25.6.

HRMS(ESI-TOF) *m/z*: calcd for [M<sup>+</sup>]H<sup>+</sup> C<sub>24</sub>H<sub>28</sub>O<sub>4</sub> 381.2061, found: 381.2067.



**4h**

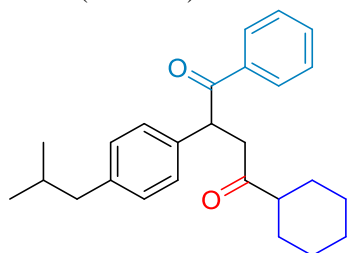
**4-cyclohexyl-2-(2-methoxyphenyl)-1-phenylbutane-1,4-dione (4h)**

Chromatography Pentane/EA = 30:1 (v/v), 37.4 mg (54%), pale yellow solid.

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.89 – 6.72 (m, 9H), 5.46 (dd, *J* = 10.4, 3.4 Hz, 1H), 3.80 (s, 3H), 3.45 (dd, *J* = 17.7, 10.4 Hz, 1H), 2.56 (dd, *J* = 17.7, 3.4 Hz, 1H), 2.38 – 2.29 (m, 1H), 1.92 – 1.11 (m, 10H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  212.3, 199.7, 155.9, 136.4, 132.6, 128.7, 128.6, 128.3, 128.2, 127.2, 121.0, 110.9, 55.4, 50.7, 43.8, 41.3, 28.4, 28.4, 25.9, 25.7, 25.6.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{23}\text{H}_{26}\text{O}_3$  351.1955, found: 351.1964.



**4i**

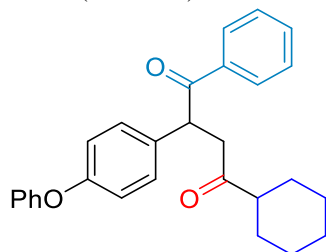
#### **4-cyclohexyl-2-(4-isobutylphenyl)-1-phenylbutane-1,4-dione (4i)**

Chromatography Pentane/EA = 30:1 (v/v), 51.2 mg (68%), white solid.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.98 – 7.03 (m, 9H), 5.09 (dd,  $J$  = 10.1, 4.0 Hz, 1H), 3.60 (dd,  $J$  = 17.9, 10.1 Hz, 1H), 2.76 (dd,  $J$  = 17.9, 4.0 Hz, 1H), 2.43 – 2.28 (m, 4H), 1.93 – 1.20 (m, 10H), 0.85 (d,  $J$  = 6.6 Hz, 6H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  212.3, 199.2, 140.7, 136.5, 135.8, 132.7, 129.8, 128.9, 128.4, 127.8, 50.7, 48.2, 45.5, 44.9, 30.1, 28.3, 28.3, 25.8, 25.6, 25.6, 22.3, 22.3.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{26}\text{H}_{32}\text{O}_2$  377.2475, found: 377.2484.



**4j**

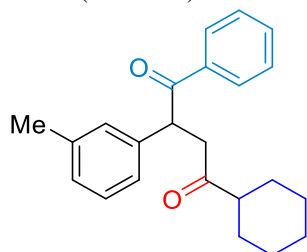
#### **4-cyclohexyl-2-(4-phenoxyphenyl)-1-phenylbutane-1,4-dione (4j)**

Chromatography Pentane/EA = 30:1 (v/v), 43.6 mg (53%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92 – 6.80 (m, 14H), 7.31 (dd,  $J$  = 9.97, 4.09, 1H), 3.53 (dd,  $J$  = 17.9, 10.0 Hz, 1H), 2.70 (dd,  $J$  = 17.9, 4.1 Hz, 1H), 7.17 – 7.10 (m, 2H), 7.04 – 6.95 (m, 1H), 6.95 – 6.71 (m, 4H), 5.04 (dd,  $J$  = 10.0, 4.1 Hz, 1H), 3.53 (dd,  $J$  = 17.9, 10.0 Hz, 1H), 2.70 (dd,  $J$  = 17.9, 4.1 Hz, 1H), 2.35 – 2.26 (m, 1H), 1.88 – 1.11 (m, 10H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  212.1, 199.1, 156.8, 156.5, 136.4, 133.2, 132.8, 129.7, 129.4, 128.9, 128.4, 123.4, 119.1, 119.0, 50.7, 47.7, 45.5, 28.3, 28.3, 25.8, 25.6, 25.6.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{28}\text{H}_{28}\text{O}_3$  413.2112, found: 413.2124.



**4k**

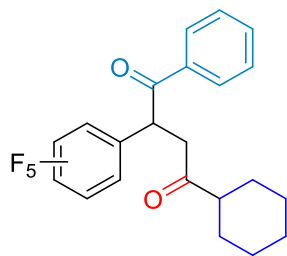
#### **4-cyclohexyl-1-phenyl-2-(m-tolyl)butane-1,4-dione (4k)**

Chromatography Pentane/EA = 20:1 (v/v), 42.0 mg (63%), white solid.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.91 – 6.91 (m, 9H), 5.00 (dd,  $J$  = 10.3, 3.8 Hz, 1H), 3.55 (dd,  $J$  = 18.0, 10.3 Hz, 1H), 2.65 (dd,  $J$  = 18.0, 3.8 Hz, 1H), 2.33 – 2.26 (m, 1H), 2.20 (s, 3H), 1.87 – 1.10 (m, 10H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  212.1, 199.1, 138.7, 138.6, 136.4, 132.7, 128.9, 128.8, 128.6, 128.4, 127.9, 125.2, 50.6, 48.4, 45.5, 28.3, 25.8, 25.6, 25.5, 21.4, 21.3.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{Na}^+$   $\text{C}_{23}\text{H}_{26}\text{O}_2$  357.1825, found: 357.1827.



4l

#### 4-cyclohexyl-1,2-diphenylbutane-1,4-dione (4l)

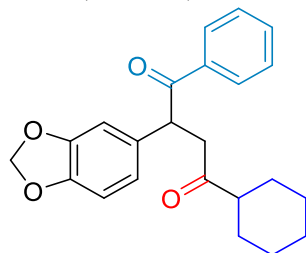
Chromatography Pentane/EA = 20:1 (v/v), 54.2 mg (63%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.79 – 7.29 (m, 5H), 5.34 (dd,  $J$  = 8.9, 4.8 Hz, 1H), 3.59 (dd,  $J$  = 17.8, 8.8 Hz, 1H), 2.63 (dd,  $J$  = 17.8, 4.8 Hz, 1H), 2.44 – 2.35 (m, 1H), 1.93 – 1.13 (m, 10H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  210.4, 195.2, 135.3, 133.5, 128.9, 128.8, 128.2, 128.0, 50.8, 40.4, 38.3, 28.4, 28.4, 25.77, 25.6, 25.5.

$^{19}\text{F}$  NMR (282 MHz,  $\text{CDCl}_3$ )  $\delta$  -140.83 – -143.75 (m), -153.29 – -158.92 (m), -160.91 – -162.82 (m).

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{22}\text{H}_{19}\text{F}_5\text{O}_2$  411.1378, found: 411.1384.



4m

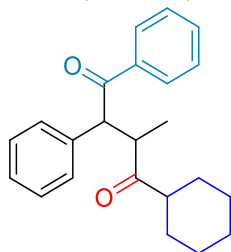
#### 2-(benzo[d][1,3]dioxol-5-yl)-4-cyclohexyl-1-phenylbutane-1,4-dione (4m)

Chromatography Pentane/EA = 20:1 (v/v), 48.8 mg (67%), pale yellow solid.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.00 – 6.71 (m, 8H), 5.93 – 5.89 (m, 2H), 5.06 (dd,  $J$  = 9.9, 4.0 Hz, 1H), 3.59 (dd,  $J$  = 17.9, 10.0 Hz, 1H), 2.75 (dd,  $J$  = 17.9, 4.0 Hz, 1H), 2.43 – 2.36 (m, 1H), 1.96 – 1.18 (m, 10H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  212.1, 199.0, 148.1, 146.7, 136.3, 132.8, 132.2, 128.8, 128.4, 121.4, 108.7, 108.3, 101.0, 50.6, 48.0, 45.4, 28.3, 25.8, 25.6, 25.5.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{23}\text{H}_{24}\text{O}_4$  365.1747, found: 365.1756.



4n

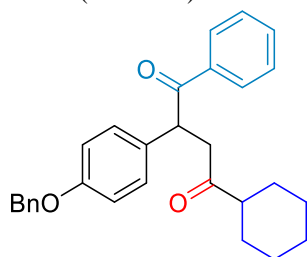
#### 1-cyclohexyl-2-methyl-3,4-diphenylbutane-1,4-dione (4n)

Chromatography Pentane/EA = 30:1 (v/v), 36.2 mg (54%), white solid. dr = 9:1, slightly sticky white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.94 – 6.98 (m, 10H), 5.03 – 4.73 (m, 1H), 3.67 (dd,  $J$  = 10.6, 6.7 Hz, 0.1/1H), 3.53 (dd,  $J$  = 17.9, 10.1 Hz, 0.9/1H), 2.65 (dd,  $J$  = 17.9, 3.9 Hz, 1H), 2.18 (s, 3H), 1.88 – 1.04 (m, 10H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  216.3(minor), 212.1, 199.1, 198.7(minor), 136.8, 136.4, 135.6, 133.1(minor), 132.7, 129.7, 129.0(minor), 128.8, 128.7(minor), 128.6(minor), 128.5(minor), 128.3, 128.1(minor), 127.9, 127.3(minor), 55.9(minor), 51.6(minor), 50.6, 48.3(minor), 48.0, 45.4, 28.3, 27.8(minor), 26.9(minor), 25.8, 25.6, 25.5, 25.3(minor), 20.9, 16.8(minor).

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{23}\text{H}_{26}\text{O}_2$  335.2006, found: 335.2013.



**4o**

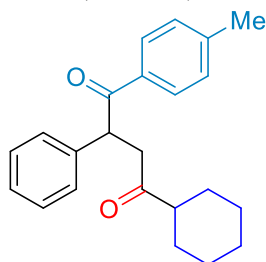
### 2-(4-(benzyloxy)phenyl)-4-cyclohexyl-1-phenylbutane-1,4-dione (4o)

Chromatography Pentane/EA = 30:1 (v/v), 42.6 mg (50%), white solid.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.01–6.89 (m, 14H), 5.11 (dd,  $J = 10.0, 4.0$  Hz, 1H), 5.01 (s, 2H), 3.62 (dd,  $J = 17.9, 10.0$  Hz, 1H), 2.77 (dd,  $J = 17.9, 4.0$  Hz, 1H), 2.46–2.37 (m, 1H), 1.97–1.16 (m, 10H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  212.3, 199.2, 157.9, 136.8, 136.4, 132.7, 130.8, 129.2, 128.8, 128.5, 128.4, 128.0, 127.4, 115.4, 69.9, 50.7, 47.6, 45.4, 28.3, 28.3, 25.8, 25.6, 25.6.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{29}\text{H}_{30}\text{O}_3$  427.2268, found: 427.2277.



**4p**

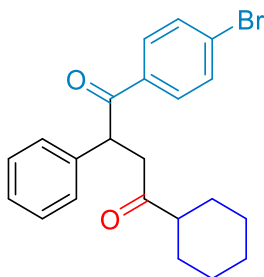
### 4-cyclohexyl-2-phenyl-1-(p-tolyl)butane-1,4-dione (4p)

Chromatography Pentane/EA = 20:1 (v/v), 46.2 mg (69%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.80 (d,  $J = 8.3$  Hz, 2H), 7.19 (d,  $J = 4.4$  Hz, 4H), 7.12–7.07 (m, 3H), 5.03 (dd,  $J = 10.0, 4.0$  Hz, 1H), 3.53 (dd,  $J = 17.9, 10.0$  Hz, 1H), 2.67 (dd,  $J = 17.9, 4.0$  Hz, 1H), 2.33–2.27 (m, 1H), 2.25 (s, 3H), 1.88–1.14 (m, 10H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  212.1, 198.6, 143.5, 139.0, 133.8, 129.1, 129.0, 129.0, 128.0, 127.1, 50.7, 48.4, 45.3, 28.3, 28.3, 25.8, 25.6, 25.5, 21.5.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{23}\text{H}_{26}\text{O}_2$  357.1825, found: 357.1834.



**4q**

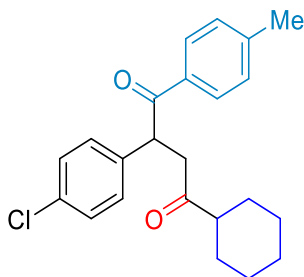
**1-(4-bromophenyl)-4-cyclohexyl-2-phenylbutane-1,4-dione (4q)**

Chromatography Pentane/EA = 20:1 (v/v), 40.0 mg (50%), white solid.

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.77 – 7.10 (m, 9H), 4.97 (dd, *J* = 10.2, 3.8 Hz, 1H), 3.54 (dd, *J* = 18.0, 10.2 Hz, 1H), 2.68 (dd, *J* = 18.0, 3.8 Hz, 1H), 2.34 – 2.26 (m, 1H), 1.87 – 1.12 (m, 10H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 212.1, 198.1, 138.3, 135.1, 131.7, 130.4, 129.2, 128.0, 128.0, 127.4, 50.6, 48.5, 45.4, 28.3, 25.8, 25.6, 25.5.

HRMS(ESI-TOF) *m/z*: calcd for [M<sup>+</sup>]<sup>+</sup> C<sub>22</sub>H<sub>23</sub>O<sub>2</sub>Br 421.0773, found: 421.0778.



**4r**

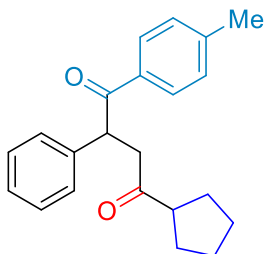
**2-(4-chlorophenyl)-4-cyclohexyl-1-(p-tolyl)butane-1,4-dione (4r)**

Chromatography Pentane/EA = 20:1 (v/v), 52.0 mg (72%), white solid.

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.77 (d, *J* = 8.3 Hz, 2H), 7.18 – 7.08 (m, 6H), 5.02 (dd, *J* = 9.7, 4.3 Hz, 1H), 3.49 (dd, *J* = 17.9, 9.7 Hz, 1H), 2.65 (dd, *J* = 17.9, 4.3 Hz, 1H), 2.26 (s, 3H), 1.87 – 1.07 (m, 11H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 211.8, 198.3, 143.8, 137.5, 133.6, 133.0, 129.5, 129.4, 129.3, 129.2, 129.2, 128.9, 128.7, 50.7, 47.6, 45.1, 28.3, 25.8, 25.6, 25.5, 21.6.

HRMS(ESI-TOF) *m/z*: calcd for [M<sup>+</sup>]<sup>+</sup> C<sub>23</sub>H<sub>25</sub>ClO<sub>2</sub> 391.1435, found: 391.1436.



**4s**

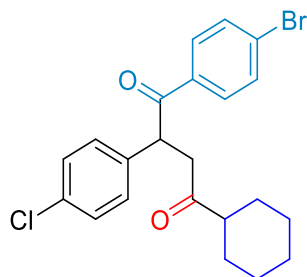
**4-cyclopentyl-2-phenyl-1-(p-tolyl)butane-1,4-dione (4s)**

Chromatography Pentane/EA = 20:1 (v/v), 50.6 mg (79%), white solid.

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.80 (d, *J* = 8.3 Hz, 2H), 7.22 – 7.06 (m, 7H), 5.05 (dd, *J* = 9.9, 4.1 Hz, 1H), 3.54 (dd, *J* = 17.9, 9.9 Hz, 1H), 2.88 – 2.77 (m, 1H), 2.70 (dd, *J* = 17.9, 4.1 Hz, 1H), 2.25 (s, 3H), 1.80 – 1.18 (m, 8H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  211.2, 198.6, 143.6, 139.0, 133.9, 129.1, 129.0, 129.0, 128.1, 127.1, 51.3, 48.4, 46.4, 28.7, 28.7, 26.0, 25.9, 21.5.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{22}\text{H}_{24}\text{O}_2$  321.1849, found: 321.1852.



**4t**

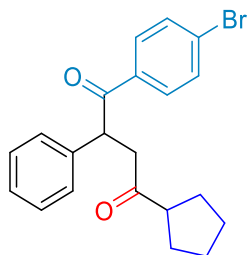
### **1-(4-bromophenyl)-2-(4-chlorophenyl)-4-cyclohexylbutane-1,4-dione (4t)**

Chromatography Pentane/EA = 20:1 (v/v), 41.6 mg (48%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75–7.08 (m, 8H), 4.95 (dd,  $J$  = 10.0, 4.0 Hz, 1H), 3.51 (dd,  $J$  = 18.0, 10.0 Hz, 1H), 2.67 (dd,  $J$  = 18.0, 4.0 Hz, 1H), 2.34–2.25 (m, 1H), 1.87–1.12 (m, 10H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  211.8, 197.8, 136.7, 134.9, 133.4, 131.8, 130.3, 129.4, 128.2, 50.6, 47.7, 45.2, 28.3, 28.3, 25.8, 25.6, 25.5.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{Na}^+$   $\text{C}_{22}\text{H}_{22}\text{BrClO}_2$  455.0384, found: 455.0386.



**4u**

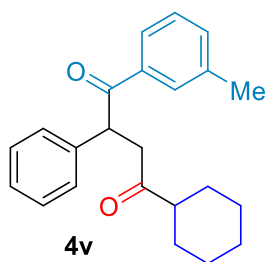
### **1-(4-bromophenyl)-4-cyclopentyl-2-phenylbutane-1,4-dione (4u)**

Chromatography Pentane/EA = 20:1 (v/v), 41.2 mg (55%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75 (d,  $J$  = 8.6 Hz, 2H), 7.44–7.09 (m, 7H), 4.98 (dd,  $J$  = 10.2, 3.9 Hz, 1H), 3.54 (dd,  $J$  = 18.0, 10.2 Hz, 1H), 2.87–2.77 (m, 1H), 2.71 (dd,  $J$  = 18.0, 3.9 Hz, 1H), 1.80–1.43 (m, 8H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  211.1, 198.0, 138.2, 135.1, 131.7, 130.3, 129.2, 128.0, 127.9, 127.4, 51.1, 48.6, 46.4, 28.7, 28.6, 25.9, 25.9.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{21}\text{H}_{21}\text{BrO}_2$  385.0798, found: 385.0802.



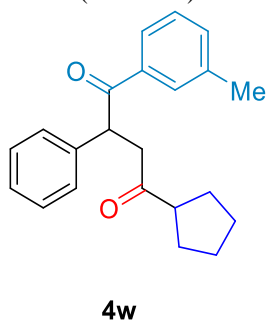
#### 4-cyclohexyl-2-phenyl-1-(*m*-tolyl)butane-1,4-dione (4v)

Chromatography Pentane/EA = 20:1 (v/v), 40.0 mg (60%), white solid.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.72 – 7.09 (m, 9H), 5.04 (dd,  $J = 10.1, 3.9$  Hz, 1H), 3.55 (dd,  $J = 17.9, 10.1$  Hz, 1H), 2.68 (dd,  $J = 17.9, 4.0$  Hz, 1H), 2.34 – 2.28 (m, 1H), 2.26 (s, 3H), 1.88 – 1.08 (m, 10H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  212.2, 199.3, 138.6, 138.1, 136.4, 133.6, 129.3, 129.0, 128.3, 128.1, 127.1, 126.1, 50.67, 48.5, 45.5, 28.3, 25.8, 25.6, 21.3.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{23}\text{H}_{26}\text{O}_2$  335.2006, found: 355.2011.



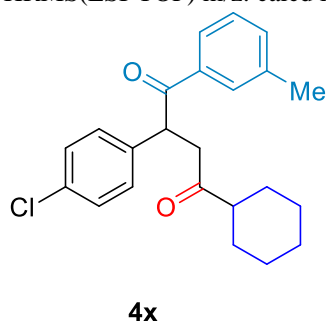
#### 4-cyclopentyl-2-phenyl-1-(*m*-tolyl)butane-1,4-dione (4w)

Chromatography Pentane/EA = 20:1 (v/v), 41.6 mg (65%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.71 – 7.08 (m, 9H), 5.06 (dd,  $J = 10.1, 4.0$  Hz, 1H), 3.55 (dd,  $J = 17.9, 10.1$  Hz, 1H), 2.87 – 2.77 (m, 1H), 2.71 (dd,  $J = 17.9, 4.0$  Hz, 1H), 2.25 (s, 3H), 1.80 – 1.21 (m, 8H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  211.2, 199.2, 138.7, 138.1, 136.4, 133.6, 129.3, 129.0, 128.3, 128.1, 127.1, 126.1, 51.2, 48.5, 46.5, 28.7, 28.6, 26.0, 25.9, 21.3.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{22}\text{H}_{24}\text{O}_2$  321.1849, found: 321.1853.



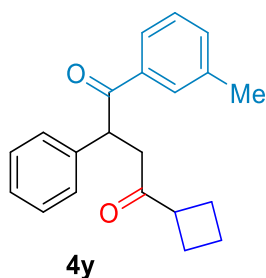
#### 2-(4-chlorophenyl)-4-cyclohexyl-1-(*m*-tolyl)butane-1,4-dione (4x)

Chromatography Pentane/EA = 20:1 (v/v), 43.0 mg (61%), white solid.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.69 – 7.11 (m, 8H), 5.03 (dd,  $J = 9.9, 4.1$  Hz, 1H), 3.51 (dd,  $J = 18.0, 9.9$  Hz, 1H), 2.67 (dd,  $J = 18.0, 4.2$  Hz, 1H), 2.34 – 2.29 (m, 1H), 2.27 (s, 3H), 1.86 – 0.98 (m, 10H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  211.9, 199.00, 138.3, 137.2, 136.2, 133.8, 133.1, 129.4, 129.3, 129.2, 128.4, 126.1, 50.6, 47.7, 45.2, 28.3, 25.8, 25.6, 25.5, 21.3.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{23}\text{H}_{25}\text{ClO}_2$  369.1616, found: 369.1622.



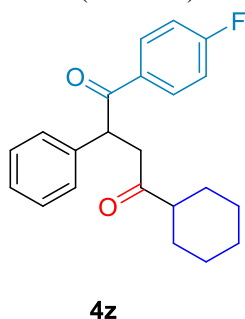
#### 4-cyclobutyl-2-phenyl-1-(*m*-tolyl)butane-1,4-dione (4y)

Chromatography Pentane/EA = 20:1 (v/v), 25.8 mg (42%), white solid.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.73 – 7.10 (m, 9H), 5.06 (dd,  $J$  = 10.0, 4.1 Hz, 1H), 3.43 (dd,  $J$  = 17.8, 10.0 Hz, 1H), 3.26 – 3.17 (m, 1H), 2.62 (dd,  $J$  = 17.8, 4.1 Hz, 1H), 2.27 (s, 3H), 2.16 – 1.18 (m, 6H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  209.9, 199.3, 138.7, 138.2, 136.4, 133.7, 129.3, 129.1, 128.3, 128.1, 127.2, 126.1, 48.4, 45.3, 44.6, 24.3, 24.1, 21.3, 17.7.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{21}\text{H}_{22}\text{O}_2$  307.1693, found: 307.1700.



#### 4-cyclohexyl-1-(4-fluorophenyl)-2-phenylbutane-1,4-dione (4z)

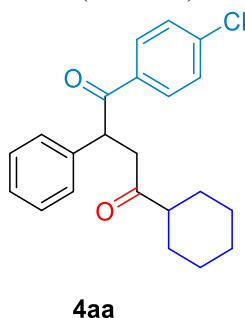
Chromatography Pentane/EA = 20:1 (v/v), 32.4 mg (48%), white solid.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.94 – 6.93 (m, 9H), 4.99 (dd,  $J$  = 10.2, 3.8 Hz, 1H), 3.55 (dd,  $J$  = 18.0, 10.2 Hz, 1H), 2.68 (dd,  $J$  = 18.0, 3.8 Hz, 1H), 2.34 – 2.27 (m, 1H), 1.87 – 1.05 (m, 10H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  212.3, 197.6, 166.9, 164.4, 138.7, 132.9, 132.9, 131.7, 131.6, 129.3, 128.2, 127.5, 115.8, 115.6, 50.8, 48.6, 45.6, 28.5, 26.0, 25.8, 25.7.

$^{19}\text{F}$  NMR (282 MHz,  $\text{CDCl}_3$ )  $\delta$  -105.53 – -105.67 (m).

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{22}\text{H}_{23}\text{FO}_2$  339.1755, found: 339.1763.



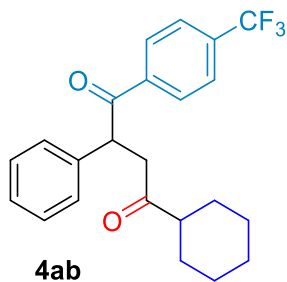
#### 1-(4-chlorophenyl)-4-cyclohexyl-2-phenylbutane-1,4-dione (4aa)

Chromatography Pentane/EA = 20:1 (v/v), 44.0 mg (62%), white solid.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.84 – 7.10 (m, 9H), 4.97 (dd,  $J$  = 10.2, 3.8 Hz, 1H), 3.54 (dd,  $J$  = 18.0, 10.2 Hz, 1H), 2.68 (dd,  $J$  = 18.0, 3.8 Hz, 1H), 2.33 – 2.26 (m, 1H), 1.87 – 1.08 (m, 10H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  212.0, 197.8, 139.2, 138.3, 134.7, 130.2, 129.2, 128.7, 128.0, 127.4, 50.6, 48.5, 45.4, 28.4, 28.3, 25.8, 25.6, 25.5.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{22}\text{H}_{23}\text{ClO}_2$  355.1459, found: 355.1450.



**4-cyclohexyl-2-phenyl-1-(4-(trifluoromethyl)phenyl)butane-1,4-dione (4ab)**

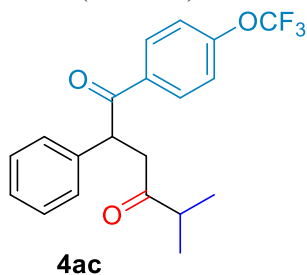
Chromatography Pentane/EA = 20:1 (v/v), 31.0 mg (40%), white solid.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.00 – 7.13 (m, 9H), 5.01 (dd,  $J = 10.4, 3.7$  Hz, 1H), 3.58 (dd,  $J = 18.1, 10.4$  Hz, 1H), 2.72 (dd,  $J = 18.1, 3.7$  Hz, 1H), 2.35 – 2.28 (m, 1H), 1.88 – 1.12 (m, 10H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  212.1, 198.3, 139.3, 137.8, 134.2, 133.8, 129.3, 129.1, 128.1, 127.5, 125.6, 125.5, 125.5, 125.4, 122.2, 50.6, 48.9, 45.5, 28.4, 28.5, 25.8, 25.6, 25.6.

$^{19}\text{F}$  NMR (282 MHz,  $\text{CDCl}_3$ )  $\delta$  -63.19.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{22}\text{H}_{24}\text{F}_3\text{O}_2$  389.1723, found: 389.1729.



**5-methyl-2-phenyl-1-(4-(trifluoromethoxy)phenyl)hexane-1,4-dione (4ac)**

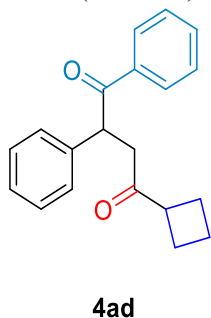
Chromatography Pentane/EA = 20:1 (v/v), 21.8 mg (30%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97 – 7.11 (m, 9H), 4.99 (dd,  $J = 10.2, 3.8$  Hz, 1H), 3.57 (dd,  $J = 18.0, 10.2$  Hz, 1H), 2.72 (dd,  $J = 18.0, 3.8$  Hz, 1H), 2.61 – 2.52 (m, 1H), 1.06 (d,  $J = 6.9$  Hz, 3H), 1.01 (d,  $J = 7.0$  Hz, 3H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  212.8, 197.5, 138.1, 134.7, 130.8, 129.3, 128.0, 127.5, 120.2, 54.8, 48.7, 45.3, 40.7, 18.1.

$^{19}\text{F}$  NMR (282 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.60.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{20}\text{H}_{19}\text{F}_3\text{O}_3$  365.1359, found: 365.1366.



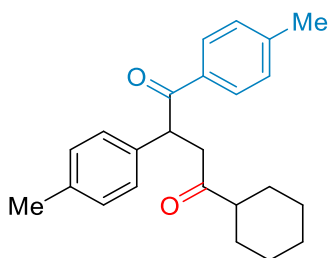
**4-cyclobutyl-1,2-diphenylbutane-1,4-dione (4ad)**

Chromatography Pentane/EA = 20:1 (v/v), 30.7 mg (53%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.93 – 7.09 (m, 10H), 5.07 (dd,  $J = 10.0, 4.1$  Hz, 1H), 3.44 (dd,  $J = 17.9, 10.0$  Hz, 1H), 3.27 – 3.16 (m, 1H), 2.66 – 1.29 (m, 7H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  209.9, 199.0, 138.6, 136.4, 132.8, 129.1, 128.9, 128.4, 128.1, 127.2, 48.5, 45.3, 44.6, 24.3, 24.1, 17.7.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{20}\text{H}_{20}\text{O}_2$  293.1536, found: 293.1535.



**4ae**

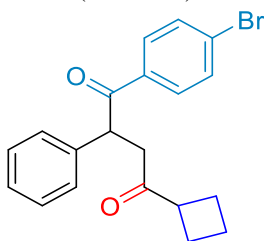
#### **4-cyclohexyl-1,2-di-*p*-tolylbutane-1,4-dione (4ae)**

Chromatography Pentane/EA = 20:1 (v/v), 48.8 mg (70%), white solid.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.83 – 7.76 (m, 2H), 7.11 – 7.05 (m, 4H), 6.99 (d,  $J$  = 7.9 Hz, 2H), 5.00 (dd,  $J$  = 10.0, 4.0 Hz, 1H), 3.52 (dd,  $J$  = 17.9, 10.0 Hz, 1H), 2.64 (dd,  $J$  = 17.9, 4.1 Hz, 1H), 2.33 – 2.89 (m, 1H), 2.26 (s, 3H), 2.19 (s, 3H), 1.86 – 1.08 (m, 10H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  212.3, 198.8, 143.58, 136.818, 1360.0, 133.9, 129.8, 129.1, 129.0, 128.2, 128.0, 50.8, 48.0, 45.4, 28.4, 28.4, 25.9, 25.7, 25.6, 21.6, 21.0.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{24}\text{H}_{28}\text{O}_2$  349.2162, found: 349.2171.



**4af**

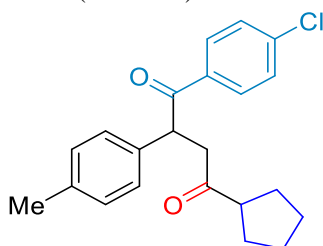
#### **1-(4-bromophenyl)-4-cyclobutyl-2-phenylbutane-1,4-dione (4af)**

Chromatography Pentane/EA = 20:1 (v/v), 33.4 mg (45%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.78 – 7.74 (m, 2H), 7.46 – 7.42 (m, 2H), 7.24 – 7.10 (m, 5H), 4.98 (dd,  $J$  = 10.1, 4.0 Hz, 1H), 3.43 (dd,  $J$  = 17.9, 10.1 Hz, 1H), 3.27 – 3.16 (m, 1H), 2.61 (dd,  $J$  = 17.9, 4.0 Hz, 1H), 2.27 – 1.18 (m, 6H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  209.8, 198.0, 138.2, 135.2, 131.8, 130.4, 129.2, 128.0, 128.0, 127.4, 48.5, 45.2, 44.5, 24.3, 24.1, 17.8.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{20}\text{H}_{19}\text{BrO}_2$  371.0641, found: 371.0643.



**4ag**

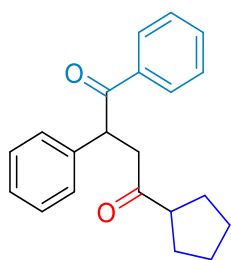
#### **1-(4-chlorophenyl)-4-cyclopentyl-2-(4-methylphenyl)butane-1,4-dione (4ag)**

Chromatography Pentane/EA = 20:1 (v/v), 39.6 mg (58%), white solid.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.85 – 7.80 (m, 2H), 7.28 – 6.99 (m, 6H), 4.96 (dd,  $J$  = 8.6, 5.0 Hz, 1H), 3.58 – 3.49 (m, 1H), 2.87 – 2.78 (m, 1H), 2.69 (dd,  $J$  = 17.9, 3.6 Hz, 1H), 2.19 (s, 3H), 1.80 – 1.46 (m, 8H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  211.2, 197.9, 139.1, 137.1, 135.2, 134.7, 130.2, 129.9, 128.7, 127.9, 51.2, 48.2, 46.4, 28.7, 28.6, 26.0, 25.9, 20.9.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{22}\text{H}_{23}\text{ClO}_2$  355.1460, found: 355.1469.



**4ah**

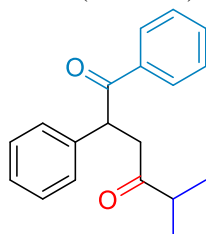
**4-cyclopentyl-1,2-diphenylbutane-1,4-dione (4ah)**

Chromatography Pentane/EA = 20:1 (v/v), 43.6 mg (71%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92 – 7.88 (m, 2H), 7.42 – 7.09 (m, 8H), 5.07 (dd,  $J = 10.1, 4.0$  Hz, 1H), 3.56 (dd,  $J = 17.9, 10.1$  Hz, 1H), 2.88 – 2.78 (m, 1H), 2.72 (dd,  $J = 17.9, 4.0$  Hz, 1H), 1.81 – 1.46 (m, 8H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  211.22, 199.05, 138.64, 136.40, 132.80, 129.08, 128.84, 128.41, 128.09, 127.20, 51.23, 48.56, 46.47, 28.72, 28.67, 25.98, 25.95.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{21}\text{H}_{22}\text{O}_2$  329.1512, found: 329.1511.



**4ai**

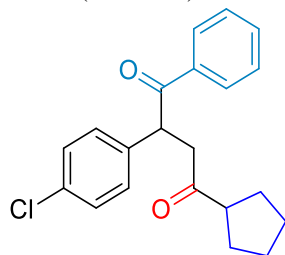
**5-methyl-1,2-diphenylhexane-1,4-dione (4ai)**

Chromatography Pentane/EA = 20:1 (v/v), 42.0 mg (75%), pale yellow solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.91 – 7.88 (m, 2H), 7.41 – 7.09 (m, 10H), 5.05 (dd,  $J = 10.1, 4.0$  Hz, 1H), 3.56 (dd,  $J = 17.9, 10.0$  Hz, 1H), 2.71 (dd,  $J = 17.9, 4.0$  Hz, 1H), 2.61 – 2.49 (m, 1H), 1.05 (d,  $J = 7.0$  Hz, 3H), 1.00 (d,  $J = 7.0$  Hz, 3H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  212.8, 199.0, 138.6, 136.4, 132.8, 129.1, 128.8, 128.4, 128.1, 127.2, 48.6, 45.2, 40.8, 18.1.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{19}\text{H}_{20}\text{O}_2$  303.1355, found: 303.1364.



**4aj**

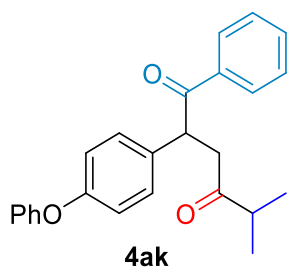
**2-(4-chlorophenyl)-4-cyclopentyl-1-phenylbutane-1,4-dione (4aj)**

Chromatography Pentane/EA = 20:1 (v/v), 38.8 mg (57%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.89 – 7.85 (m, 2H), 7.43 – 7.11 (m, 9H), 5.05 (dd,  $J = 9.8, 4.2$  Hz, 1H), 3.51 (dd,  $J = 17.9, 9.8$  Hz, 1H), 2.88 – 2.76 (m, 1H), 2.70 (dd,  $J = 18.0, 4.2$  Hz, 1H), 1.81 – 1.44 (m, 8H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  210.9, 198.7, 137.1, 136.2, 133.2, 133.0, 129.4, 129.2, 128.8, 128.5, 51.2, 47.8, 46.2, 28.7, 28.7, 26.0, 25.9.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{21}\text{H}_{21}\text{ClO}_2$  363.1122, found: 363.1129.



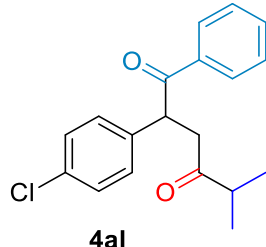
### 5-methyl-2-(4-phenoxyphenyl)-1-phenylhexane-1,4-dione (4ak)

Chromatography Pentane/EA = 20:1 (v/v), 37.2 mg (50%), pale yellow solid.

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.99 – 7.85 (m, 2H), 7.43 – 6.81 (m, 12H), 5.04 (dd,  $J = 9.9, 4.1$  Hz, 1H), 3.54 (dd,  $J = 17.9, 10.0$  Hz, 1H), 2.72 (dd,  $J = 17.9, 4.1$  Hz, 1H), 2.60 – 2.52 (m, 1H), 1.05 (d,  $J = 6.9$  Hz, 3H), 1.01 (d,  $J = 7.0$  Hz, 3H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  212.8, 199.1, 156.7, 156.6, 136.3, 133.1, 132.9, 129.7, 129.4, 128.8, 128.5, 123.5, 119.1, 119.1, 47.7, 45.2, 40.8, 18.1.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{25}\text{H}_{24}\text{O}_3$  373.1798, found: 373.1802.



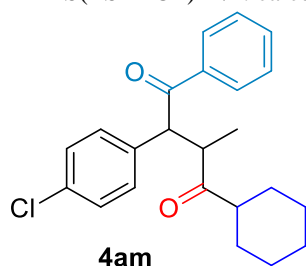
### 2-(4-chlorophenyl)-5-methyl-1-phenylhexane-1,4-dione (4al)

Chromatography Pentane/EA = 20:1 (v/v), 45.4 mg (72%), pale yellow solid.

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.94 – 7.80 (m, 2H), 7.43 – 7.12 (m, 9H), 5.04 (dd,  $J = 9.8, 4.3$  Hz, 1H), 3.51 (dd,  $J = 17.9, 9.8$  Hz, 1H), 2.70 (dd,  $J = 17.9, 4.3$  Hz, 1H), 2.59 – 2.52 (m, 1H), 1.05 (d,  $J = 6.9$  Hz, 3H), 1.00 (d,  $J = 7.0$  Hz, 3H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  212.5, 198.7, 137.1, 136.1, 133.2, 133.0, 129.4, 129.3, 128.8, 128.5, 47.8, 44.9, 40.8, 18.1.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{19}\text{H}_{19}\text{ClO}_2$  315.1147, found: 315.1151.



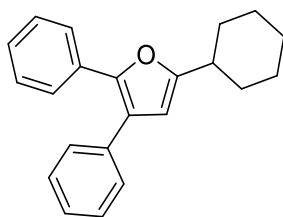
### 2-(4-chlorophenyl)-4-cyclohexyl-3-methyl-1-phenylbutane-1,4-dione (4am)

Chromatography Pentane/EA = 30:1 (v/v), 44.2 mg (60%), dr = 4:1, slightly sticky white solid.

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.98 – 7.76 (m, 2H), 7.50 – 7.07 (m, 7H), 4.83 (d,  $J = 10.6$  Hz, 0.79CH), 4.74 (d,  $J = 10.6$  Hz, 0.2CH), 3.68 – 3.60 (m, 0.8CH), 3.54 – 3.47 (m, 0.2CH), 2.06 – 1.48 (m, 4H), 1.28 – 1.08 (m, 4H), 1.06 (d,  $J = 6.7$  Hz, 2.7/3H), 1.01 – 0.95 (m, 2H), 0.87 (d,  $J = 7.3$  Hz, 0.3/3H), 0.73 – 0.63 (m, 1H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  216.9, 215.9 (minor), 199.7, 198.6 (minor), 136.7, 136.4 (minor), 135.7, 135.5 (minor), 133.4, 133.3, 132.9 (minor), 130.4, 130.2 (minor), 129.2 (minor), 128.9, 128.7, 128.6, 128.5 (minor), 55.5 (minor), 54.8, 51.3, 49.6 (minor), 48.3, 48.2 (minor), 29.1, 28.2 (minor), 27.8, 27.2 (minor), 25.9 (minor), 25.8 (minor), 25.7, 25.5 (minor), 25.4, 25.4, 16.9, 15.0 (minor).

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{23}\text{H}_{25}\text{Cl}_2\text{O}_2$  369.1616, found: 369.1620.



**8a**

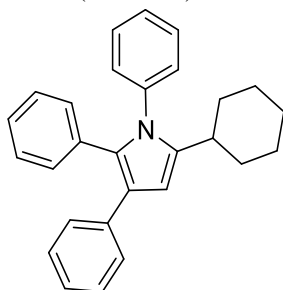
**5-cyclohexyl-2,3-diphenylfuran (8a)**

Chromatography Pentane/EA = 50:1 (v/v), 26.6 mg (88%), yellow solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.44 – 7.07 (m, 10H), 6.04 (s, 1H), 2.66 – 1.17 (m, 11H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  160.2, 146.4, 135.0, 131.8, 128.7, 128.7, 128.4, 127.1, 127.0, 126.1, 122.9, 107.5, 37.4, 31.7, 26.3, 26.1.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{22}\text{H}_{22}\text{O}$  303.1743, found: 303.1744.



**8b**

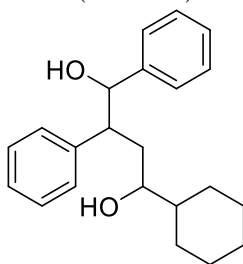
**5-cyclohexyl-1,2,3-triphenyl-1H-pyrrole (8b)**

Chromatography Pentane/EA = 20:1 (v/v), Method A: 32.0 mg (85%); Method B: 32.9 mg (87%), white solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.22 – 6.90 (m, 15H), 6.23 (s, 1H), 2.37 – 1.03 (m, 11H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  141.1, 138.8, 136.7, 133.0, 131.2, 129.1, 128.5, 128.1, 128.0, 127.7, 127.4, 126.3, 125.0, 122.2, 104.8, 35.7, 34.0, 26.6, 26.1.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{28}\text{H}_{27}\text{N}$  378.2216, found: 378.2220.



**8c**

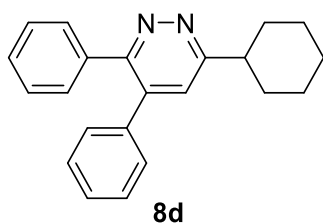
**4-cyclohexyl-1,2-diphenylbutane-1,4-diol (8c)**

Chromatography Pentane/EA = 20:1 (v/v), 29.2 mg (90%), white solid.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.26 – 7.12 (m, 10H), 4.71 (dd,  $J = 9.8, 7.4$  Hz, 1H), 3.21 – 3.15 (m, 1H), 2.96 – 2.85 (m, 1H), 1.79 – 0.69 (m, 15H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  142.4, 140.8, 129.0, 128.8, 128.6, 128.5, 128.2, 127.6, 126.9, 126.9, 78.8, 73.5, 50.5, 44.2, 36.2, 30.9, 28.9, 27.9, 26.1, 26.0.

HRMS(ESI-TOF)  $m/z$ : calcd for  $[\text{M}^+]\text{Na}^+$   $\text{C}_{22}\text{H}_{28}\text{O}_2$  347.1981, found: 347.1989.



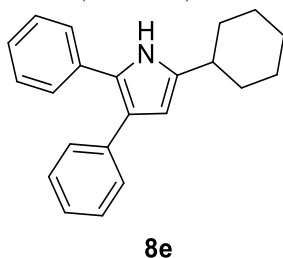
**6-cyclohexyl-3,4-diphenylpyridazine (8d)**

Chromatography Pentane/EA = 20:1 (v/v), 50.9 mg (78%), yellow solid.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.38 – 7.09 (m, 11H), 3.03 – 2.93 (m, 1H), 2.08 – 1.17 (m, 10H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  166.1, 157.8, 139.1, 137.3, 137.0, 129.9, 129.0, 128.5, 128.4, 128.4, 128.0, 125.6, 44.4, 32.7, 26.4, 25.9.

HRMS(ESI-TOF) m/z: calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{22}\text{H}_{22}\text{N}_2$  315.1856, found: 315.1851.



**5-cyclohexyl-2,3-diphenyl-1H-pyrrole (8e)**

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.86 (s, 1H), 7.29 – 7.04 (m, 10H), 6.02 (s, 1H), 2.57 – 2.48 (m, 1H), 2.03 – 1.14 (m, 10H).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  138.6, 136.9, 133.7, 128.5, 128.3, 128.2, 127.3, 126.3, 126.2, 125.5, 121.7, 106.1, 36.8, 33.1, 26.2, 26.1.

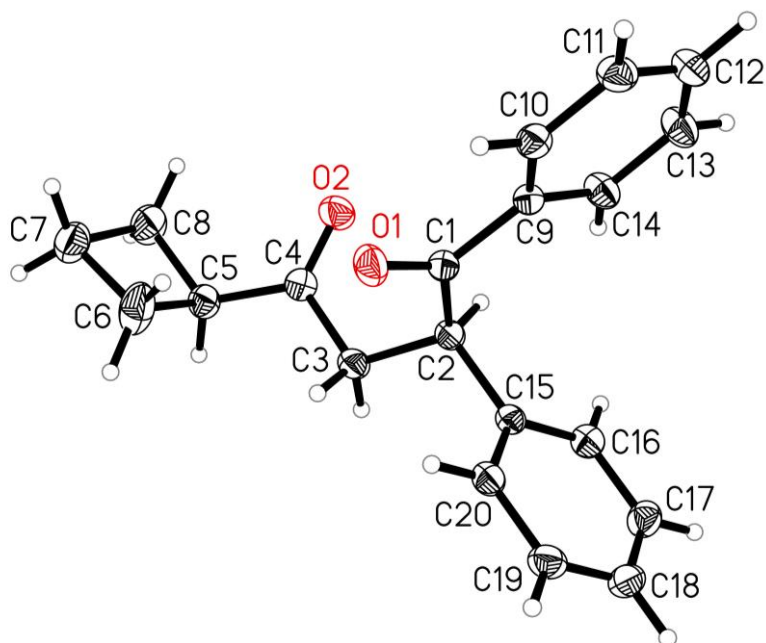
HRMS(ESI-TOF) m/z: calcd for  $[\text{M}^+]\text{H}^+$   $\text{C}_{22}\text{H}_{23}\text{N}$  302.1903, found: 302.1906.

## X-ray crystal structure analysis of **4ad**

Diffraction data were collected on a Bruker Kappa APEX II Duo diffractometer. The structure was solved by intrinsic phasing (SHELXT: Sheldrick, G. M. *Acta Cryst.* **2015**, *A71*, 3.) and refined by full-matrix least-squares procedures on  $F^2$  (SHELXL: Sheldrick, G. M. *Acta Cryst.* **2015**, *C71*, 3.). XP (Bruker AXS) was used for graphical representation.

CCDC 2386556 contains the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service [www.ccdc.cam.ac.uk/structures](http://www.ccdc.cam.ac.uk/structures).

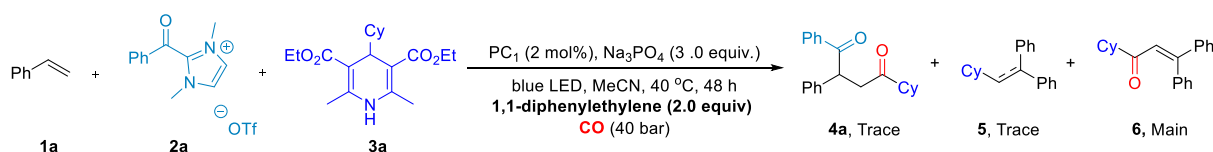
Crystal data of **4ad**:  $C_{20}H_{20}O_2$ ,  $M = 292.36$ , monoclinic, space group  $P\bar{1}$ ,  $a = 8.4568(6)$ ,  $b = 9.0832(6)$ ,  $c = 10.9280(8)$  Å,  $\beta = 77.5006(16)$ ,  $b = 83.4878(16)$ ,  $g = 74.1615(16)^\circ$ ,  $V = 787.10(10)$  Å<sup>3</sup>,  $T = 150(2)$  K,  $Z = 2$ , 27773 reflections measured, 4301 independent reflections ( $R_{int} = 0.021$ ), final  $R$  values ( $I > 2\sigma(I)$ ):  $R_1 = 0.0413$ ,  $wR_2 = 0.1111$ , final  $R$  values (all data):  $R_1 = 0.0471$ ,  $wR_2 = 0.1167$ , 199 parameters.



Molecular structure of **4ad**. Displacement ellipsoids correspond to 50% probability.

## 5. Mechanistic investigation

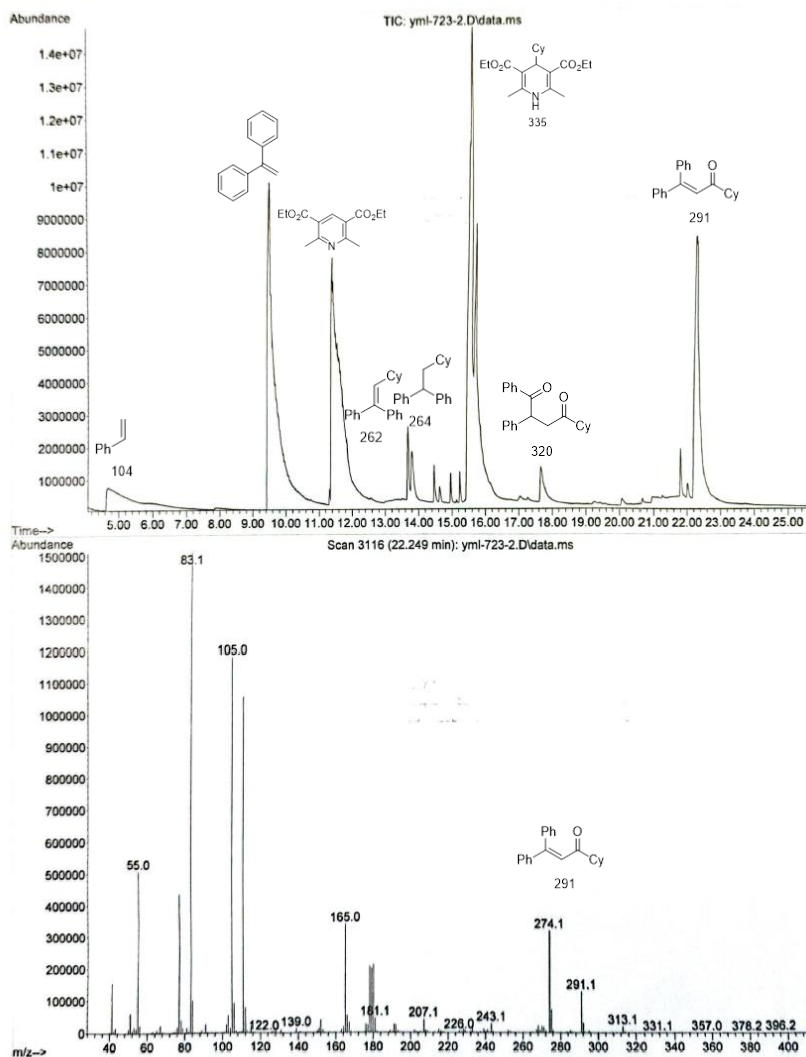
### 5.1 Radical trapping experiment by 1,1-diphenylethylene



**Scheme 1** Radical capture experiments by 1,1-diphenylethylene

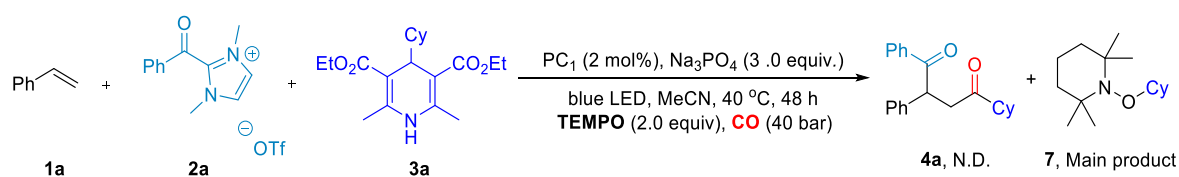
A 4 mL screw-cap vial was charged with Hantzsch ester **3a** (50.0 mg, 0.15 mmol, 1.5 equiv), acyl azolium salts **2a** (52.5 mg, 0.15 mmol, 1.5 equiv), [Ir]PF<sub>6</sub> (2.2 mg, 2 mol%), and an oven-dried stirring bar. The vial was closed with a Teflon septum and cap and connected to the atmosphere via a needle. After replacing the nitrogen in the vial three times, acetonitrile (1 mL) was added. Then, styrene (15.6 mg, 0.1 mmol, 1.0 equiv) and 1,1-diphenylethylene (35.3  $\mu$ L, 0.2 mmol, 2.0 equiv) were added using a microinjector. The vial was then moved to a cannula and transferred into a 300 mL photoautoclave (manufactured by Parr Instrument Company®), under a nitrogen atmosphere. At room temperature, the autoclave was washed with CO three times and charged with 40 bar of CO. The autoclave was placed on a heating plate equipped with a magnetic stirrer and an aluminum block. The reaction mixture was allowed to react at 40 °C under UV-A (400-500 nm) for 48 hours. After the reaction was complete, the pressure of the autoclave was carefully released, and the residual CO was washed away with nitrogen. Then, a proper amount of solvent was taken for GC-MS analysis. The result is shown in **Figure 4**. When 1,1-diphenylethylene were added to the reaction, trace products were detected, and alkyl radical and acyl radicals were trapped by 1,1-diphenylethylene. Data in agreement with that reported previously.<sup>[4]</sup>

File :D:\MassHunter\GCMS\1\data\202407\ym1-723-2.D  
 Operator :  
 Acquired : 25 Jul 2024 09:27 using AcqMethod wfp-23 min.M  
 Instrument : GCMS  
 Sample Name : ym1-723-2  
 Misc Info :  
 Vial Number : 42



**Figure 4** GC-MS of the reaction solvent

## 5.2 Radical trapping experiment by 2,2,6,6-Tetramethylpiperidinyloxy (TEMPO)



**Scheme 2** Radical capture experiments by TEMPO

A 4 mL screw-cap vial was charged Hantzsch ester **3a** (50.0 mg, 0.15 mmol, 1.5 equiv), acyl azolium salts **2a** (52.5 mg, 0.15 mmol, 1.5 equiv),  $[\text{Ir}]\text{PF}_6$  (2.2 mg, 2 mol%), TEMPO (48.0 mg, 0.2 mmol, 2.0 equiv), and an oven-dried stirring bar. The vial was closed with a Teflon septum and cap and connected to the atmosphere via a needle. After replacing the nitrogen in the vial three times, acetonitrile (1 mL) was added. Then, styrene (15.6 mg, 0.1 mmol, 1.0 equiv) was added using a microinjector. The vial was then moved to a cannula and transferred into a 300 mL photoautoclave (manufactured by Parr Instrument Company®), under a nitrogen atmosphere. At room temperature, the autoclave was washed with CO three times and charged with 40 bar of CO. The autoclave was placed on a heating plate equipped with a magnetic stirrer and an aluminum block. The reaction mixture was allowed to react at 40 °C under UV-A (400-500 nm) for 48 hours. After the reaction was complete, the pressure of the autoclave was carefully released, and the residual CO was washed away with nitrogen. Then, a proper amount of solvent was taken for GC-MS analysis. The result is shown in **Figure 5**. When TEMPO were added to the reaction, no products were detected, and alkyl radical was trapped by TEMPO. Data in agreement with that reported previously.<sup>[5]</sup>

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Vial Number: 41

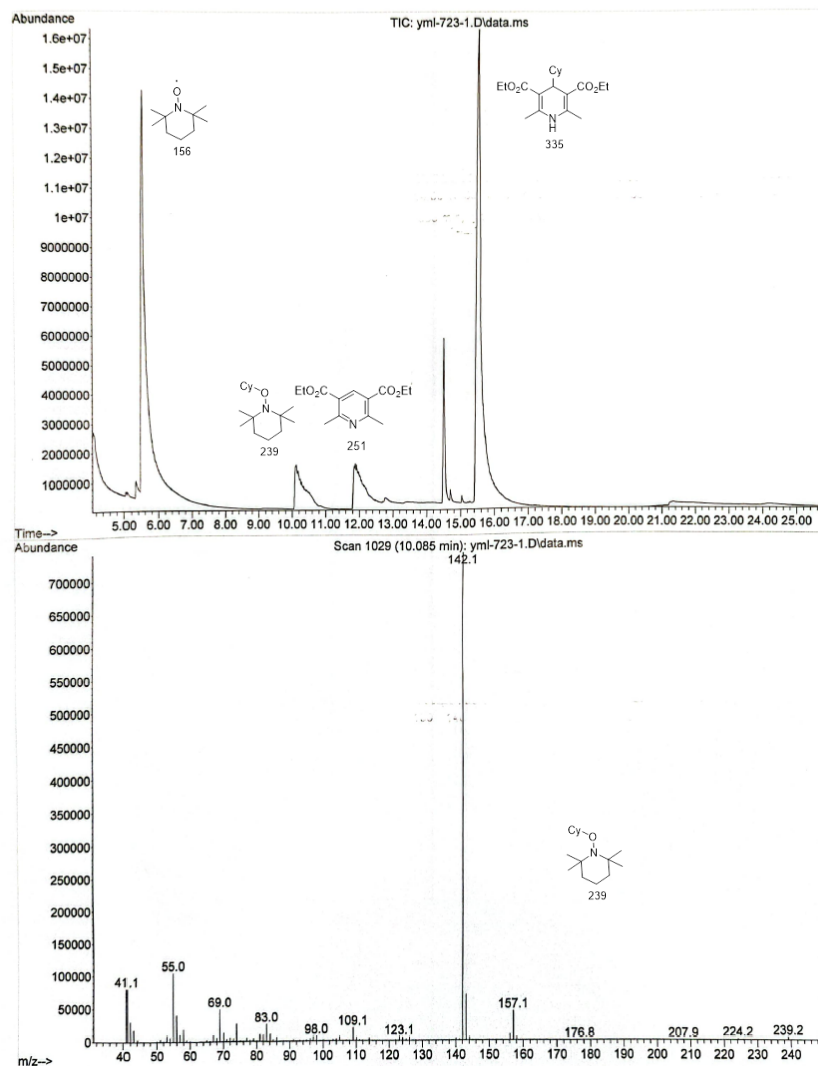


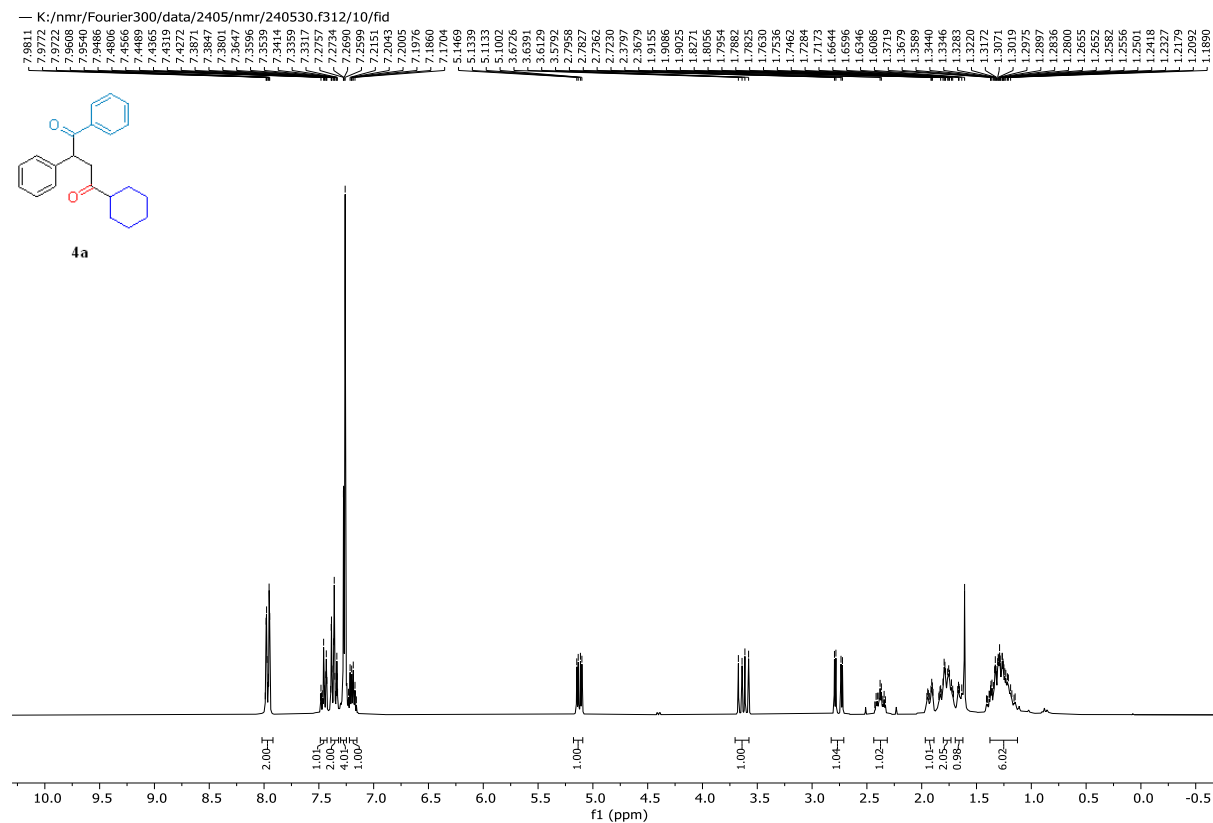
Figure 5 GC-MS of the reaction solvent

## 6. References

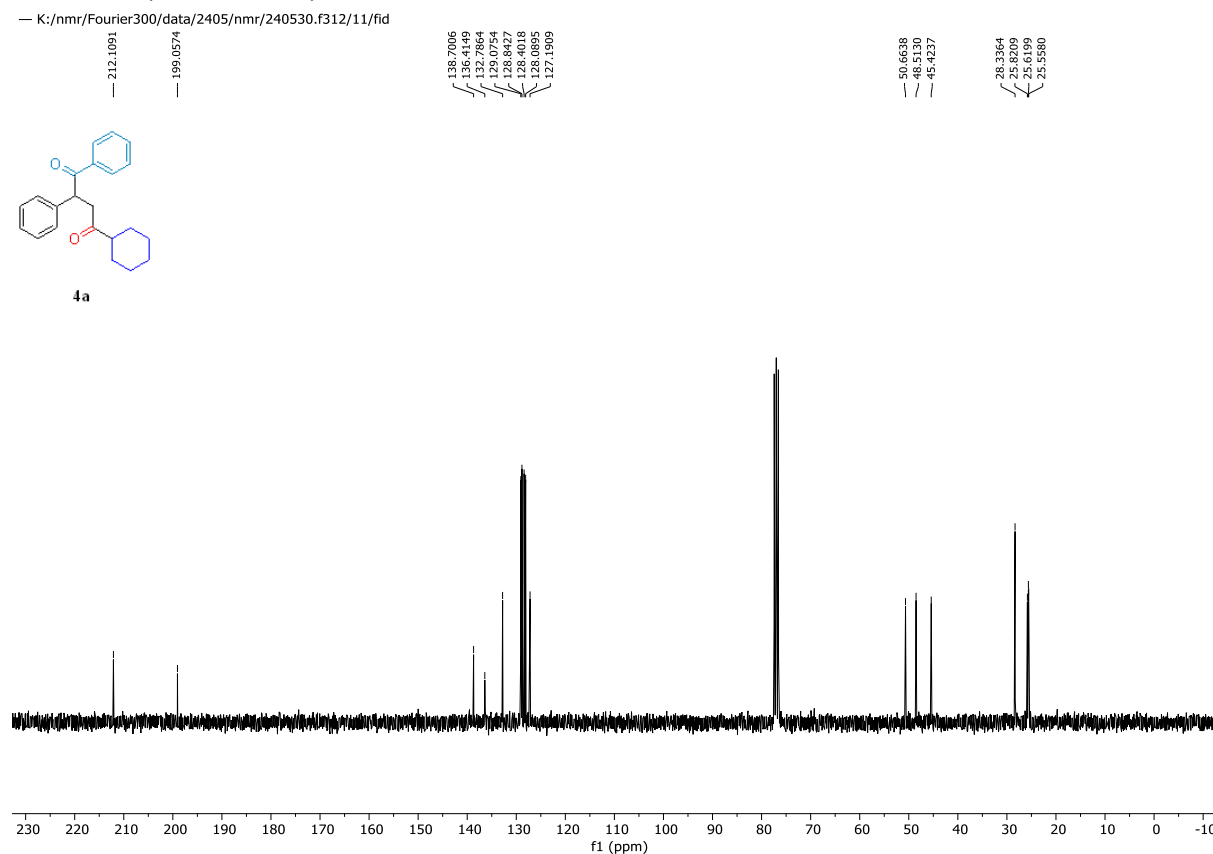
- [1]. a) M. J. Rourke, M. J. McGill, D. Yang, E. J. Farnam, J. L. Zhu, K. A. Scheidt, *J. Am. Chem. Soc.* **2023**, *145*, 1535 – 1541; b) M. J. Rourke, M. J. McGill, D. Yang, E. J. Farnam, J. L. Zhu, K. A. Scheidt, *Synlett.* **2023**, *34*, 2175 – 2180.
- [2]. G. Li, R. Chen, L. Wu, Q. Fu, X. Zhang, Z. Tang, *Angew. Chem. Int. Ed.* **2013**, *52*, 8432 – 8436.
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- [10]. R. Kuwano, M. Kashiwabara, M. Ohsumi, H. Kusano, *J. Am. Chem. Soc.* **2008**, *130*, 808 – 809.

# 7. NMR Spectra

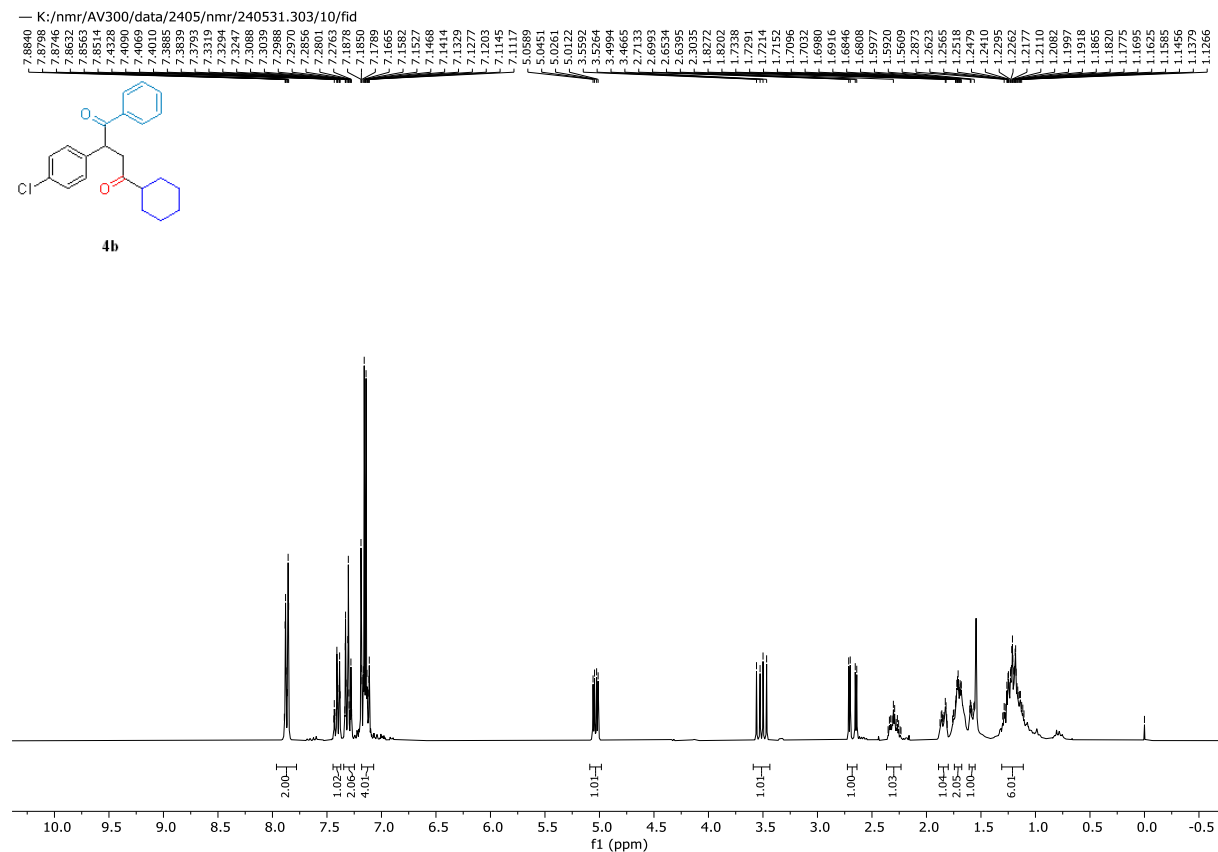
## 4a <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



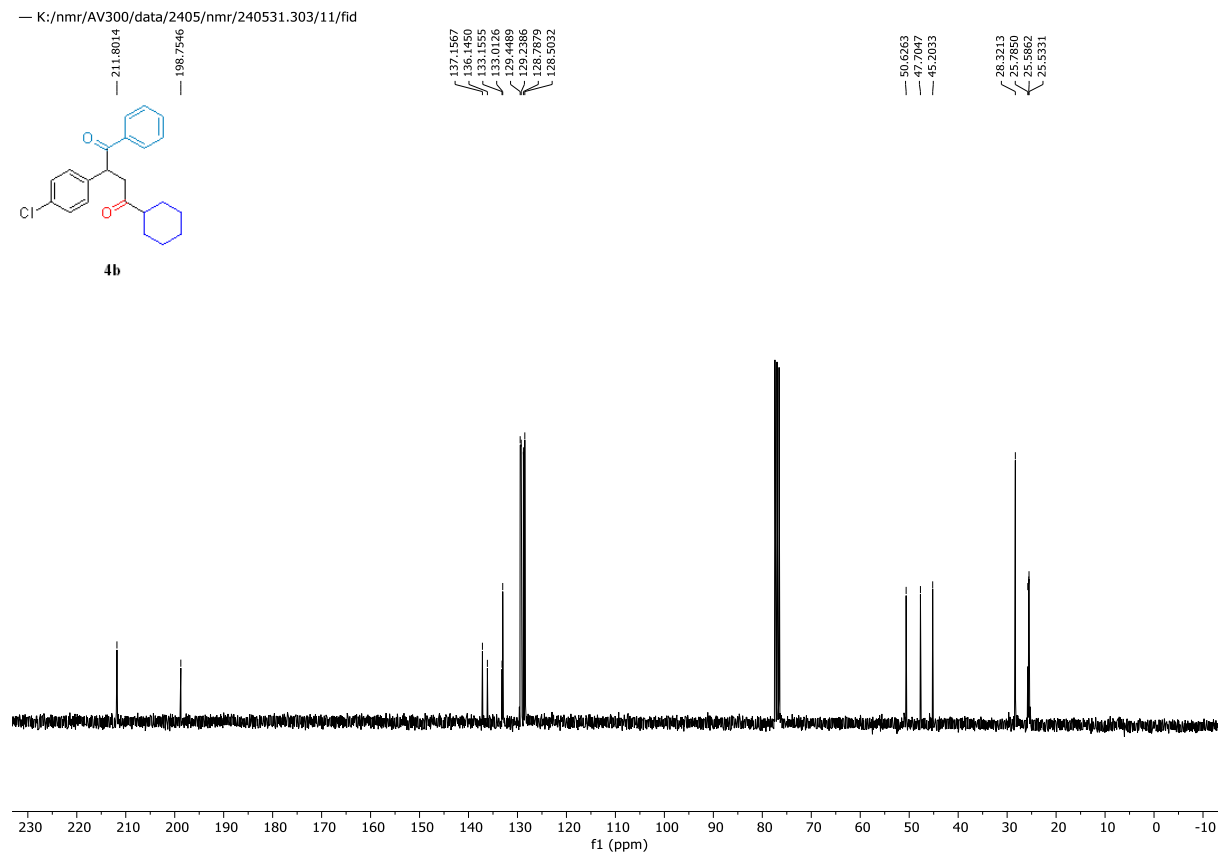
## 4a <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



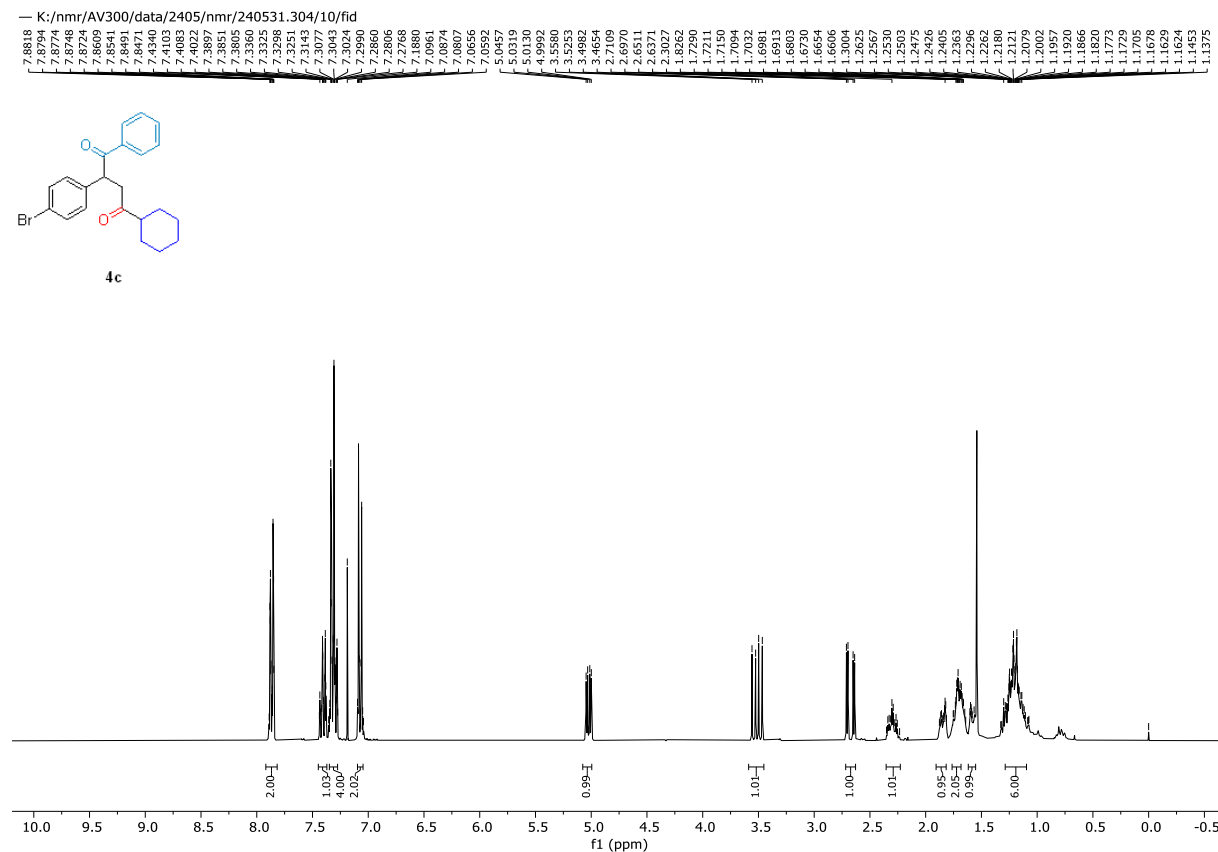
### 4b <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



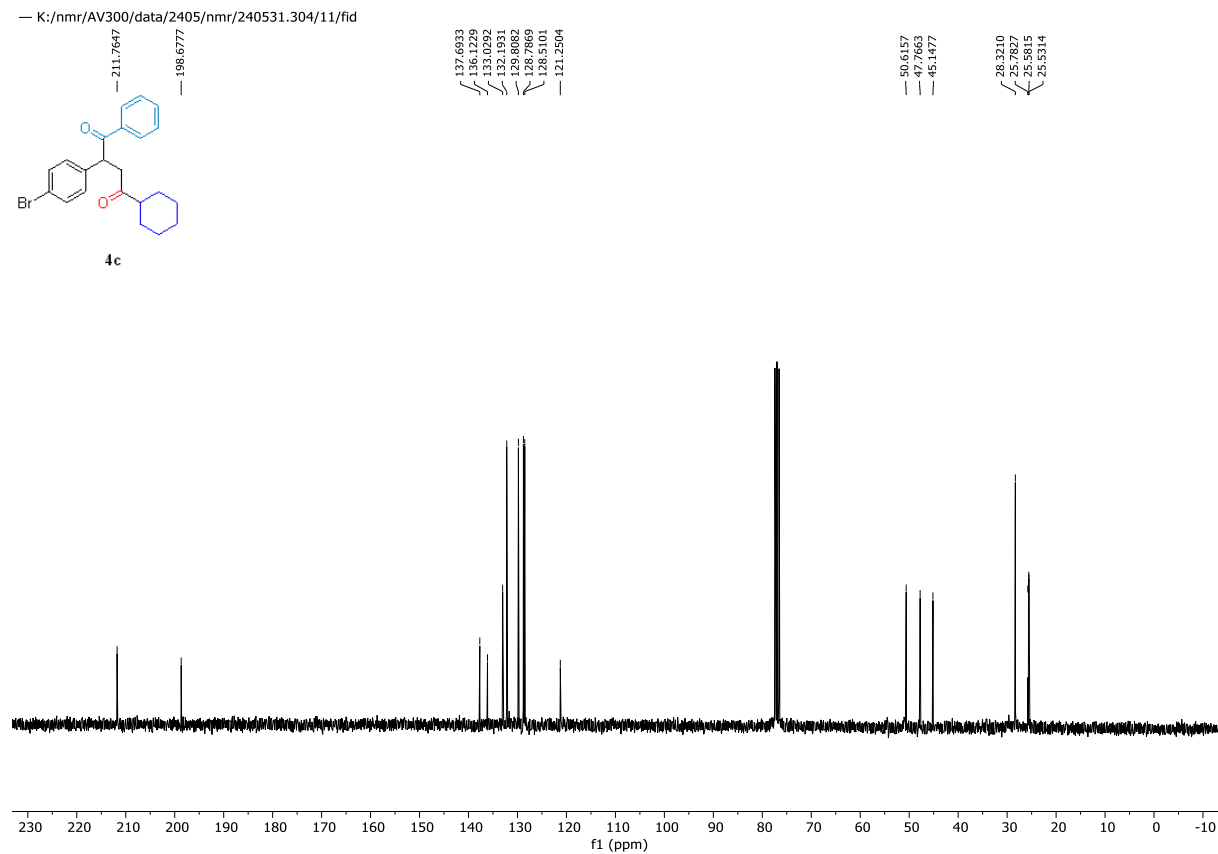
### 4b <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



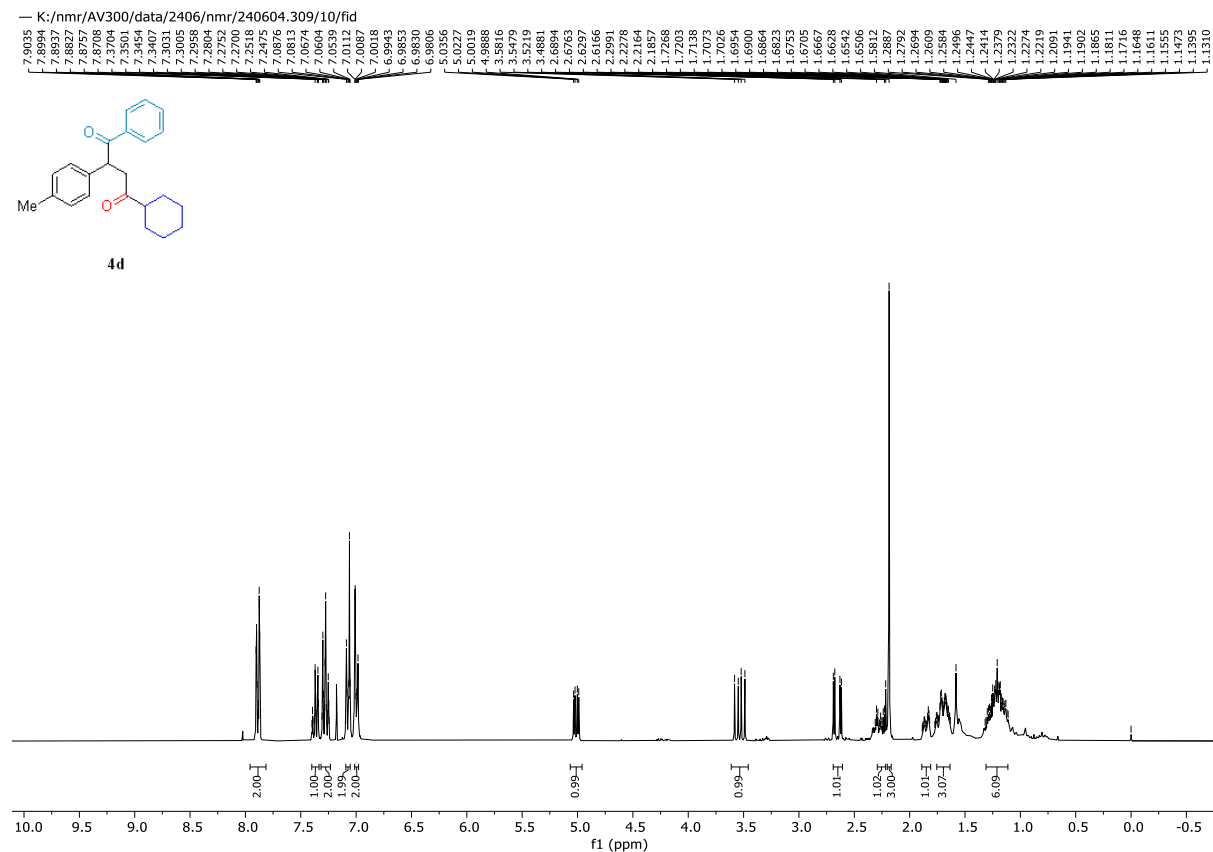
### 4c <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



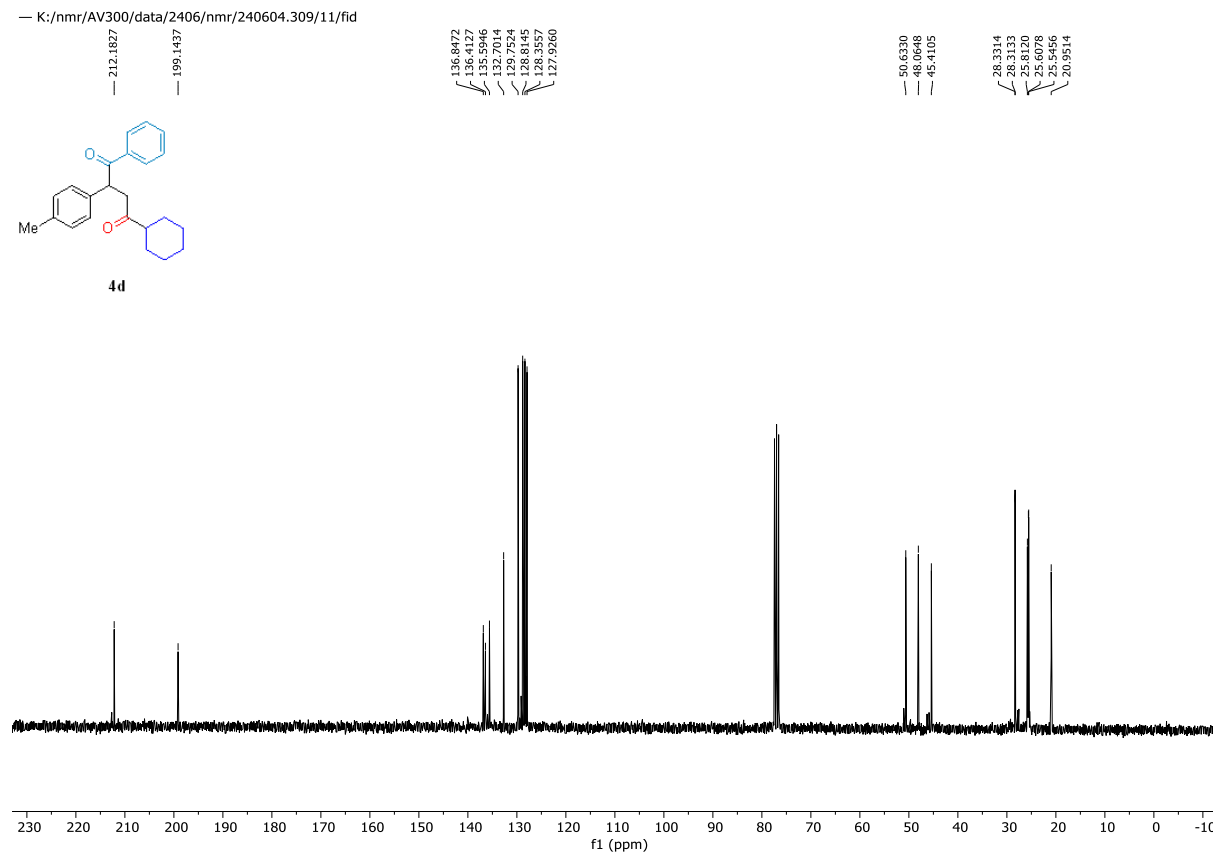
### 4c <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



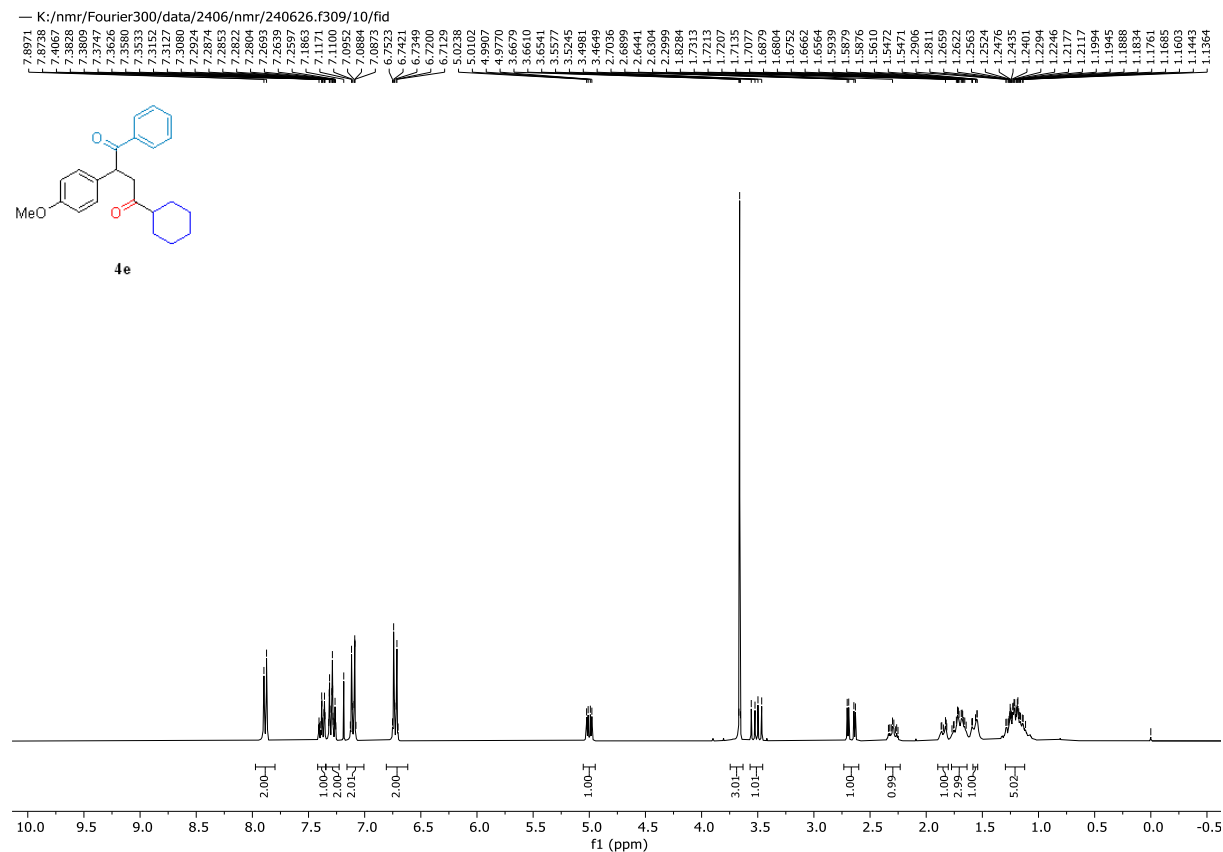
### 4d <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



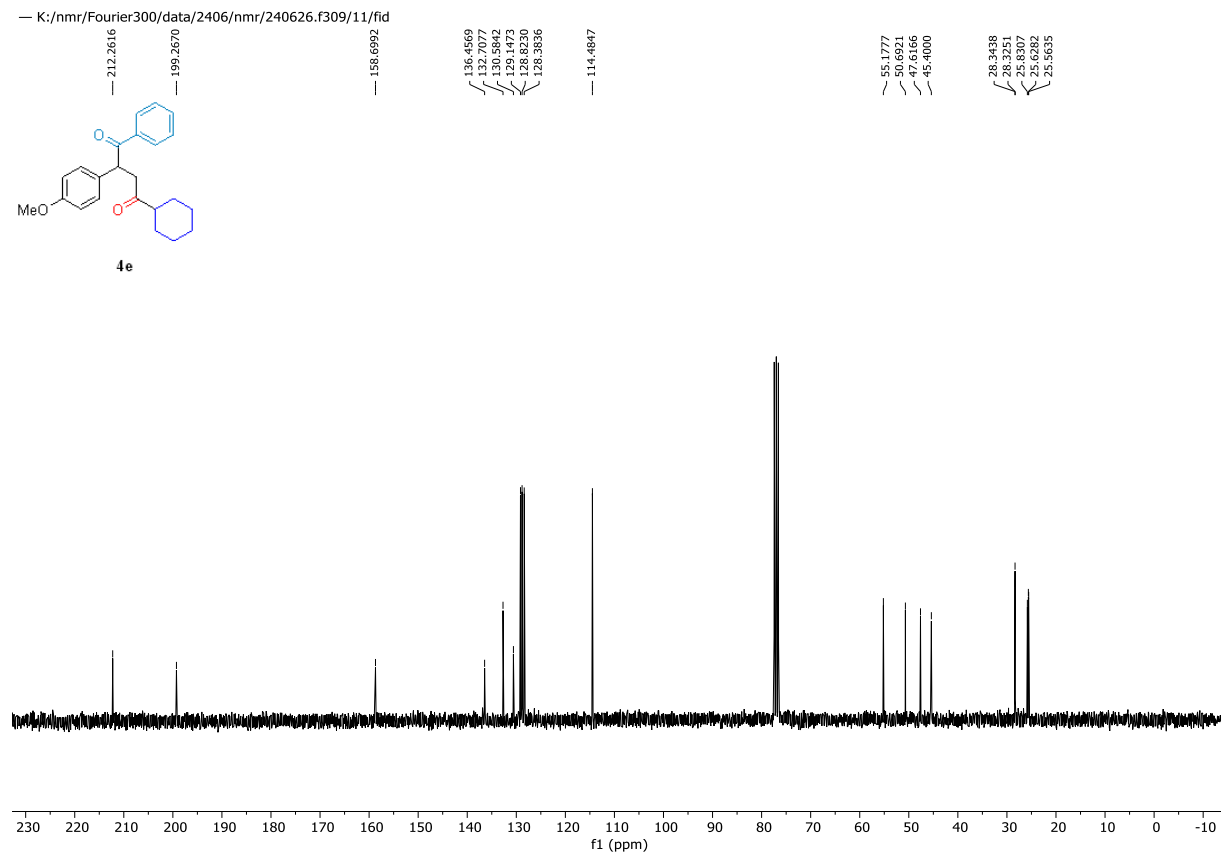
### 4d <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



### 4e <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



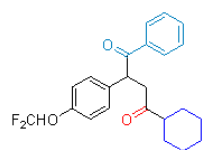
### 4e <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



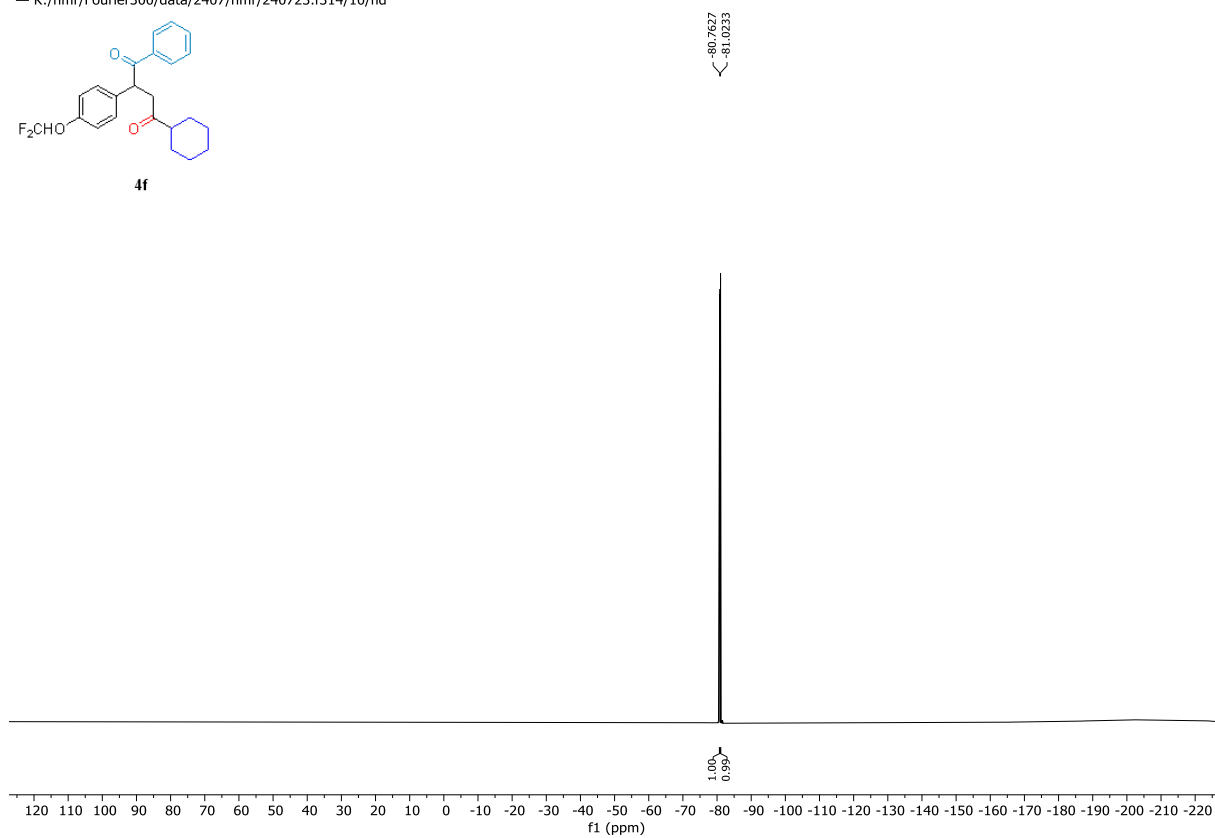


**4f**  $^{19}\text{F}$  NMR (282 MHz,  $\text{CDCl}_3$ )

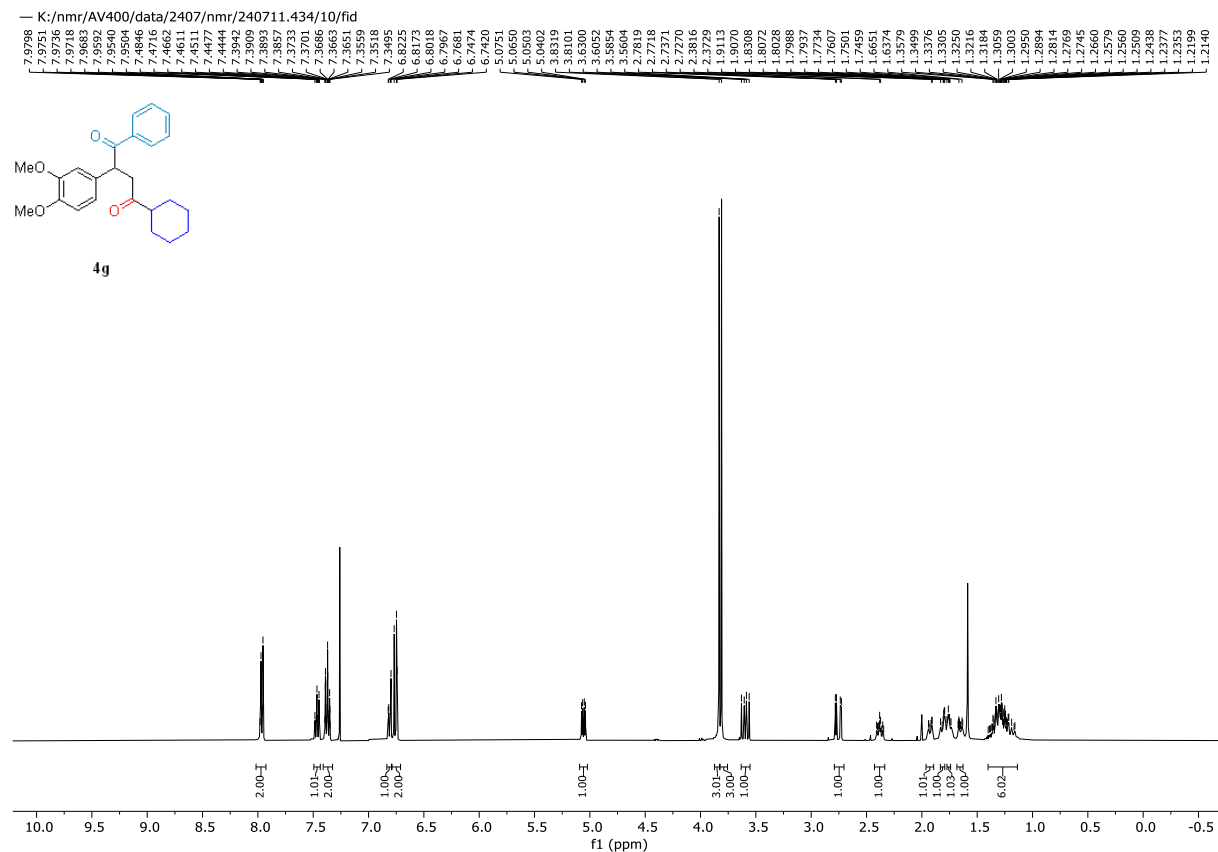
— K:/nmr/Fourier300/data/2407/nmr/240723.f314/10/fid



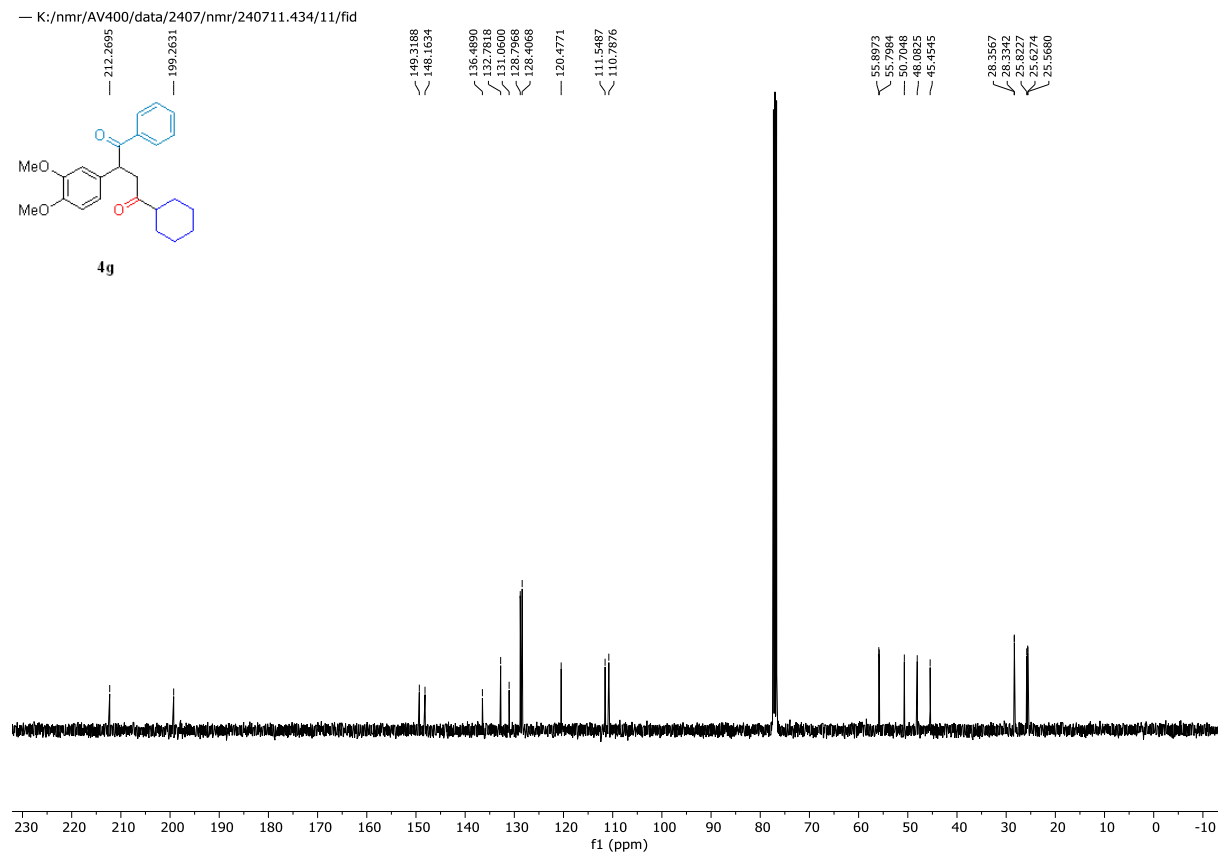
**4f**



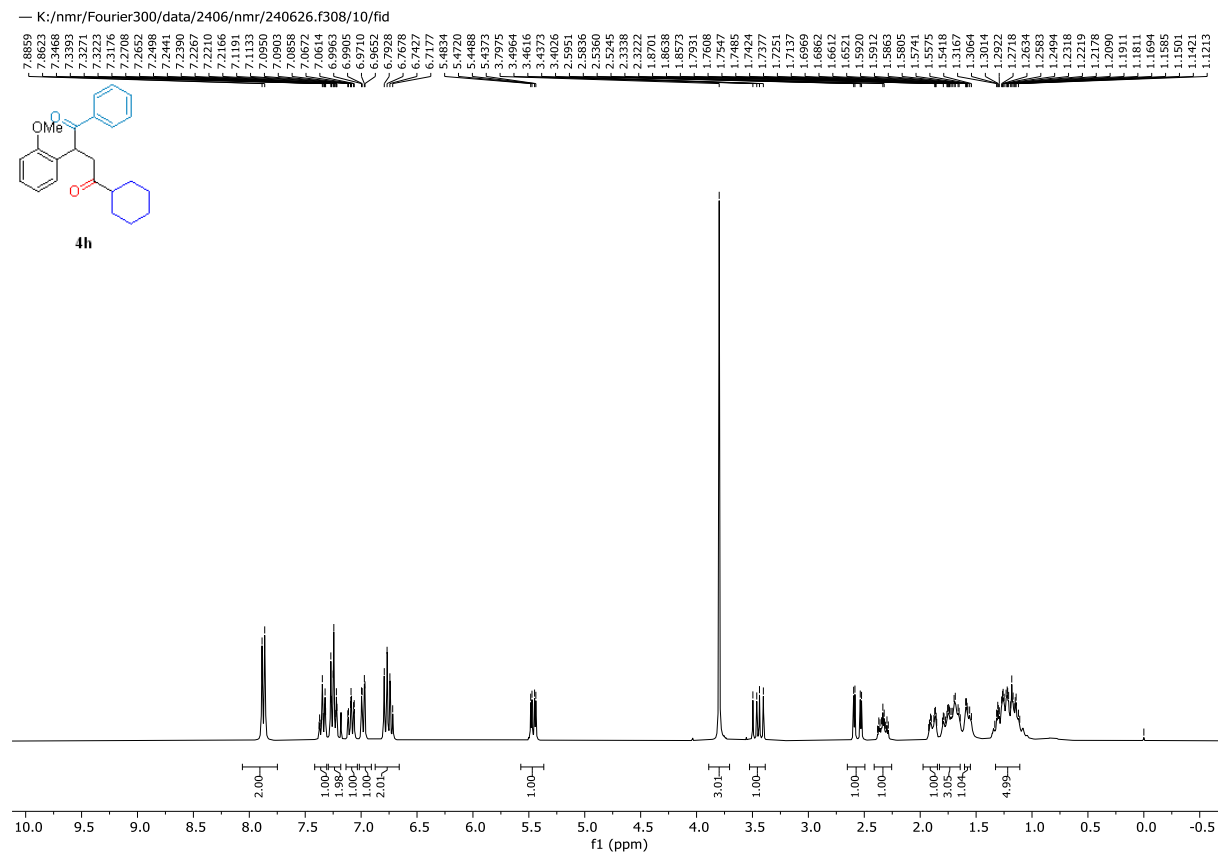
### 4g <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



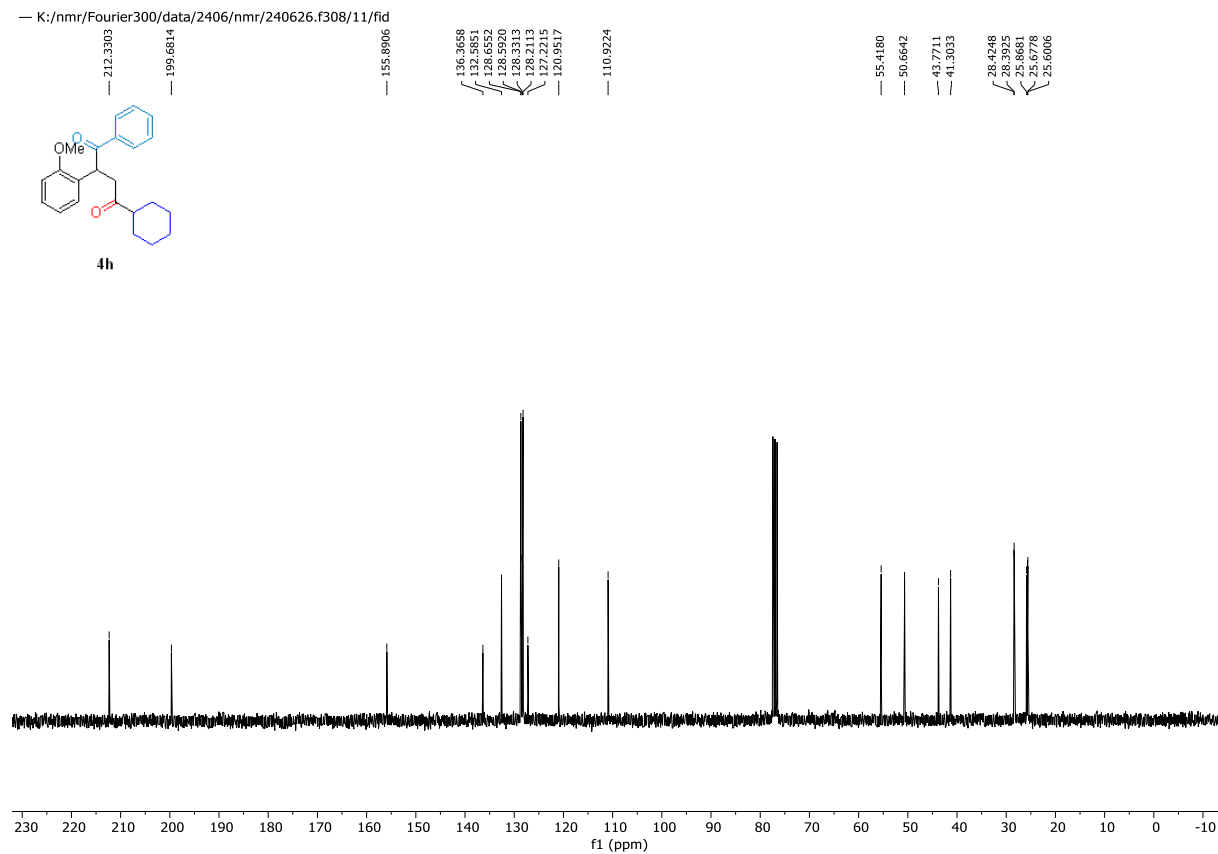
### 4g <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)



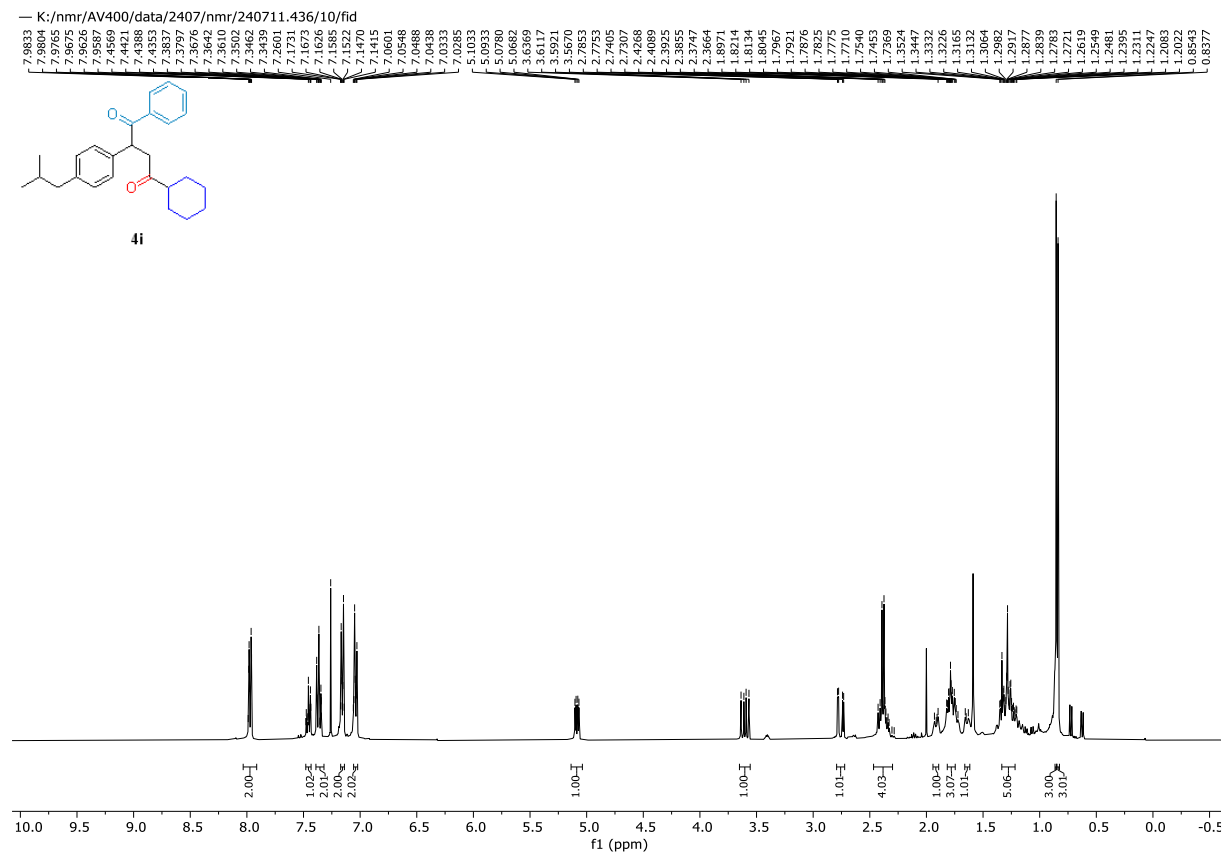
### 4h <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



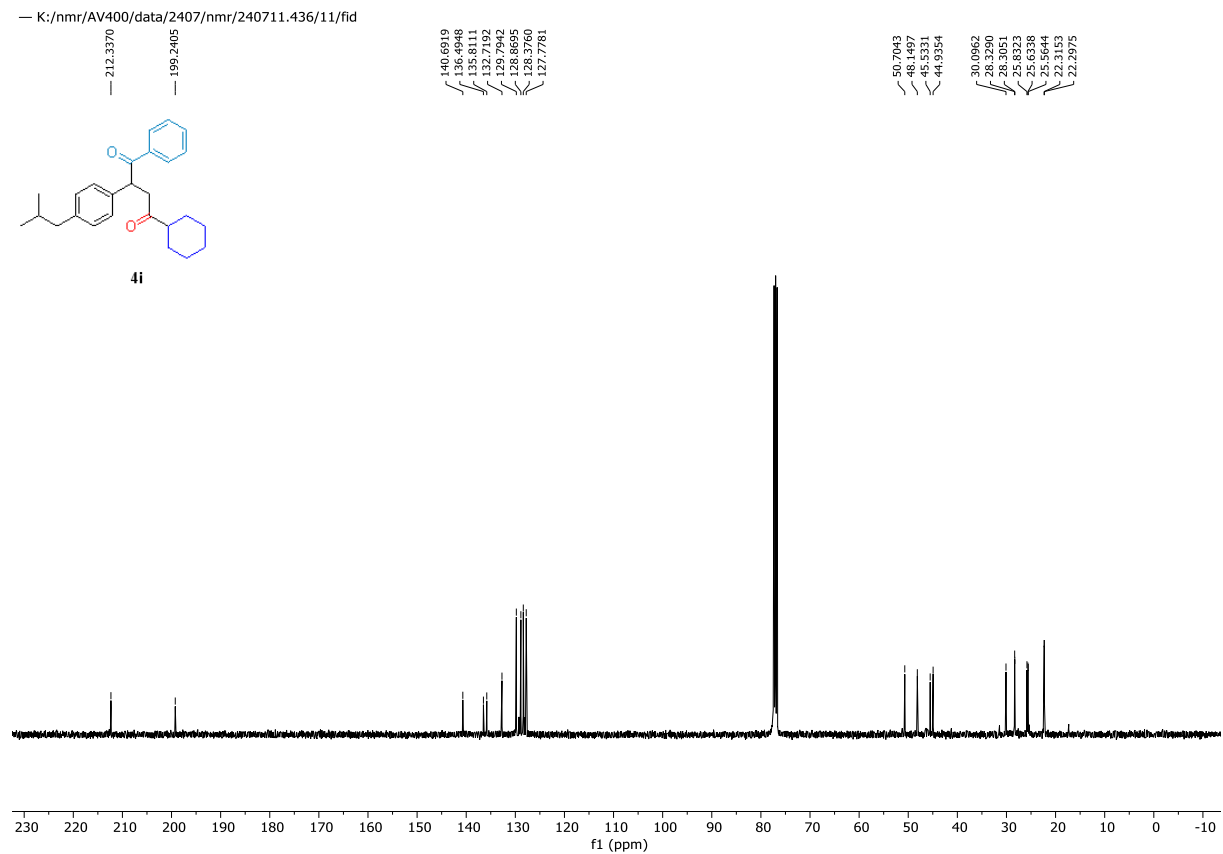
### 4h <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



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

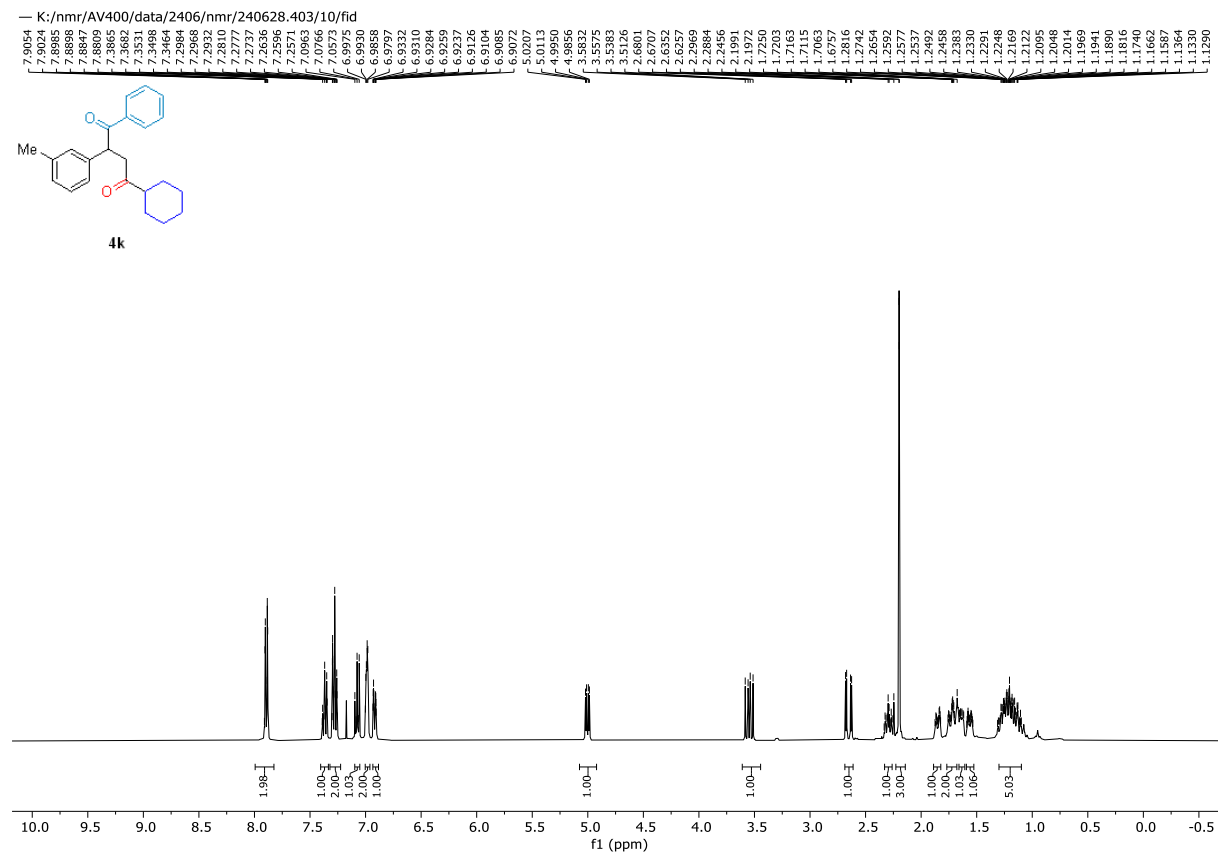


### 4i <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)

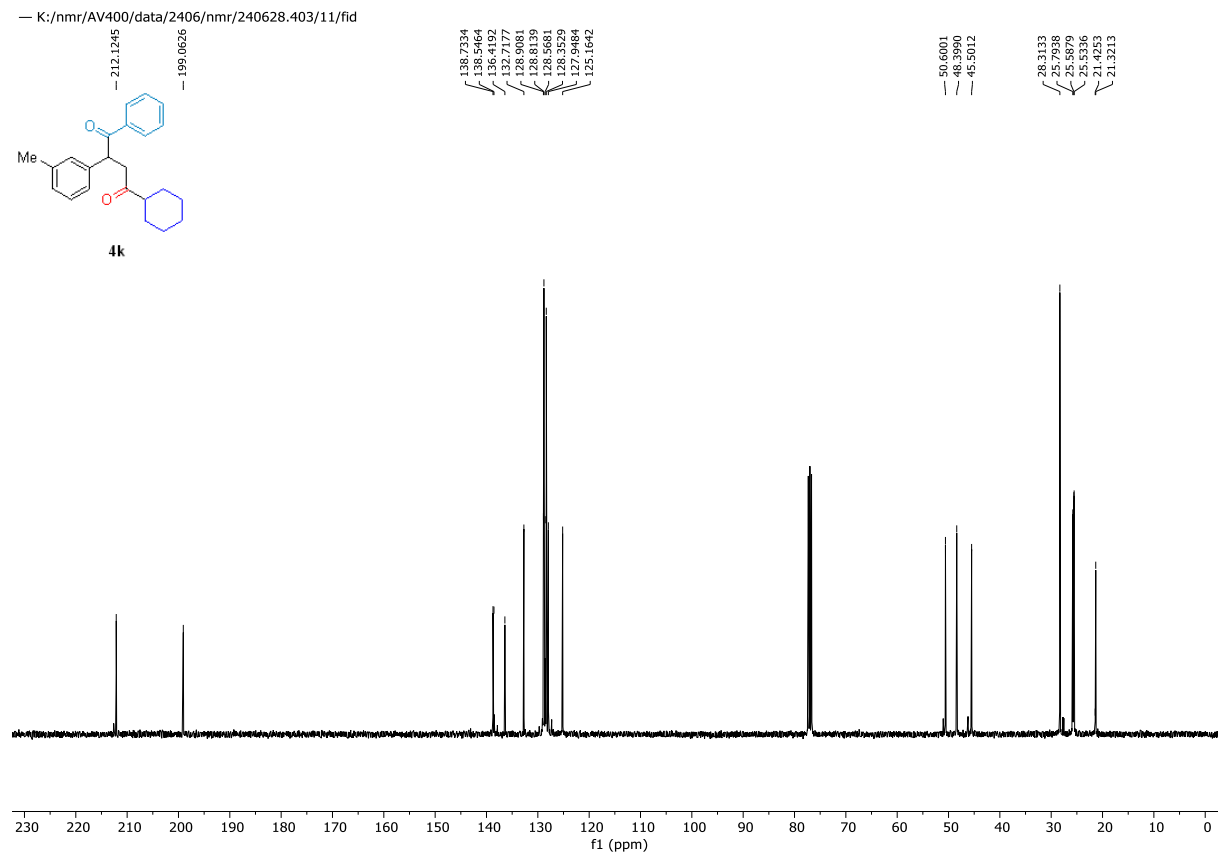




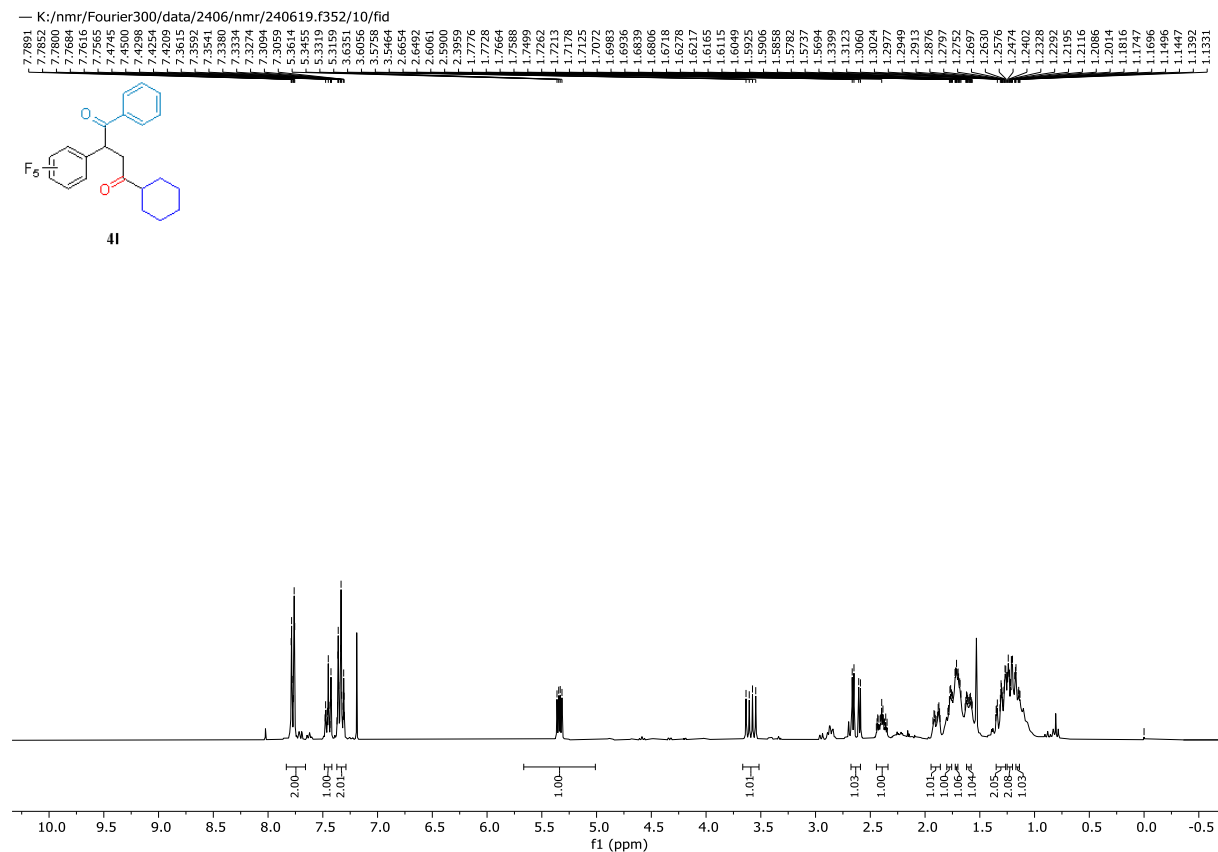
### 4k <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



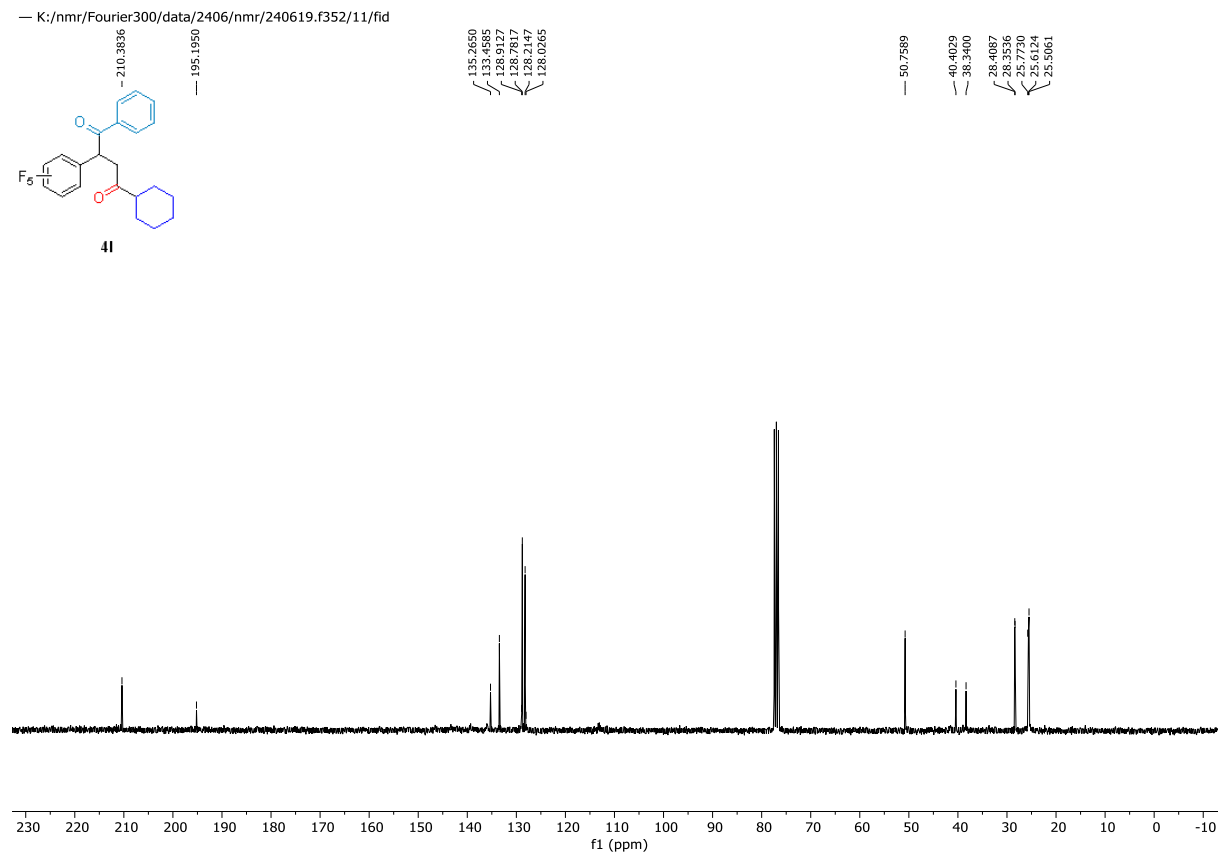
### 4k <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)



### 4i <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)

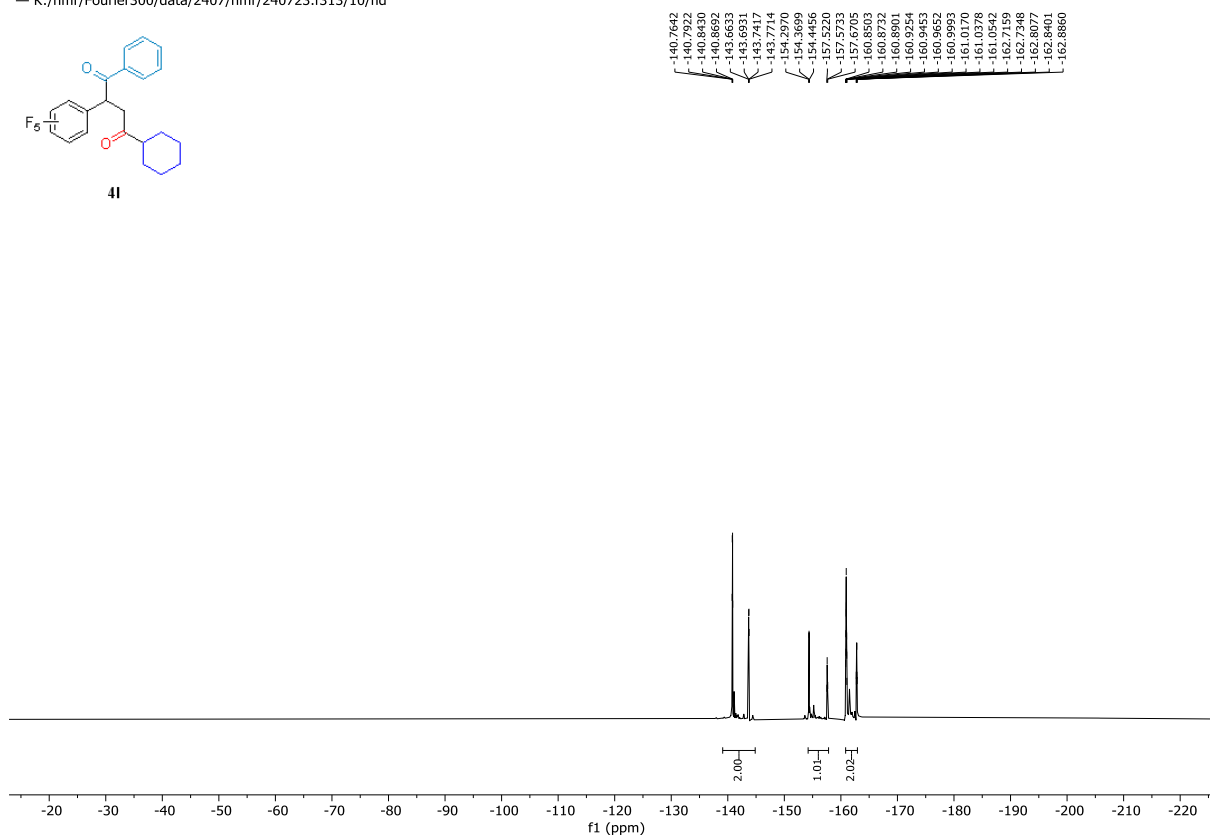
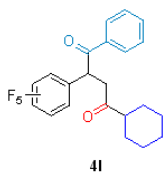


### 4i <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



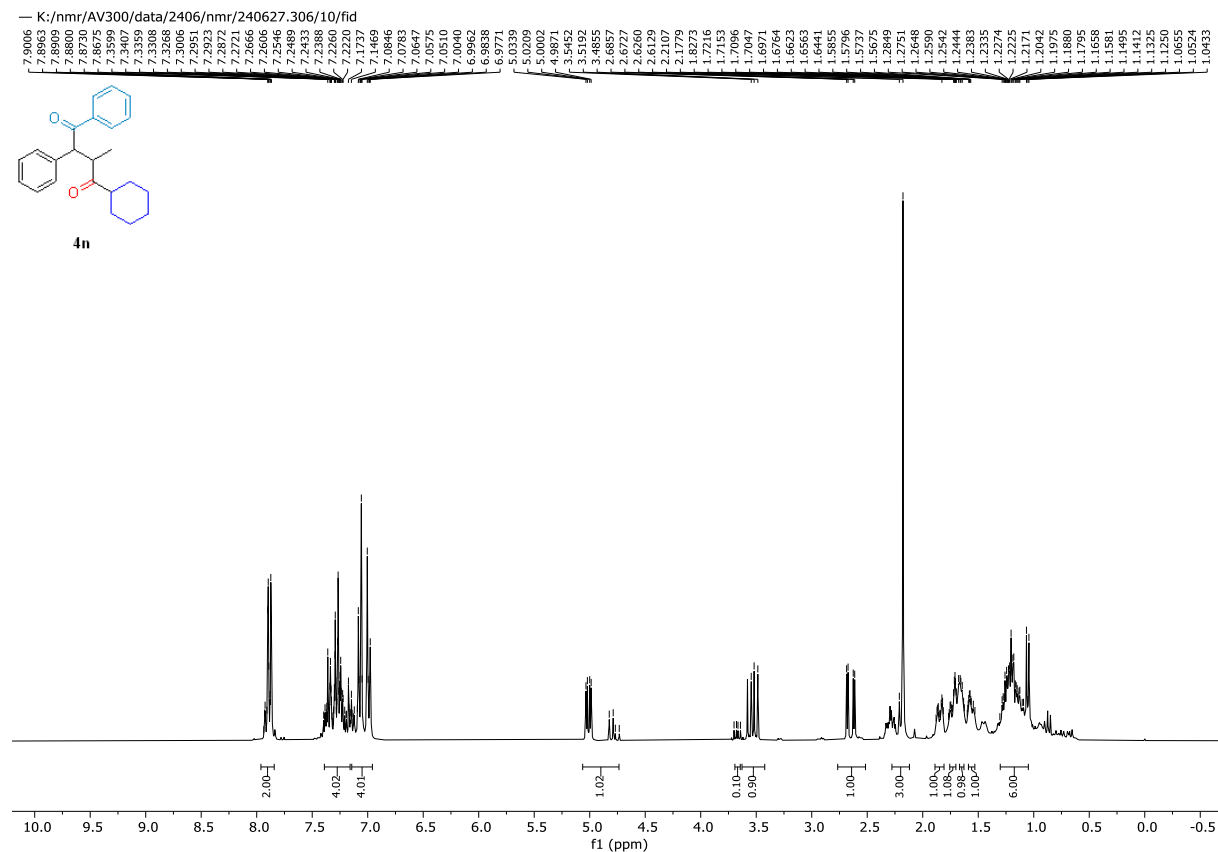
### 4i <sup>13</sup>F NMR (282 MHz, CDCl<sub>3</sub>)

K:/nmr/Fourier300/data/2407/nmr/240723.f313/10/fid

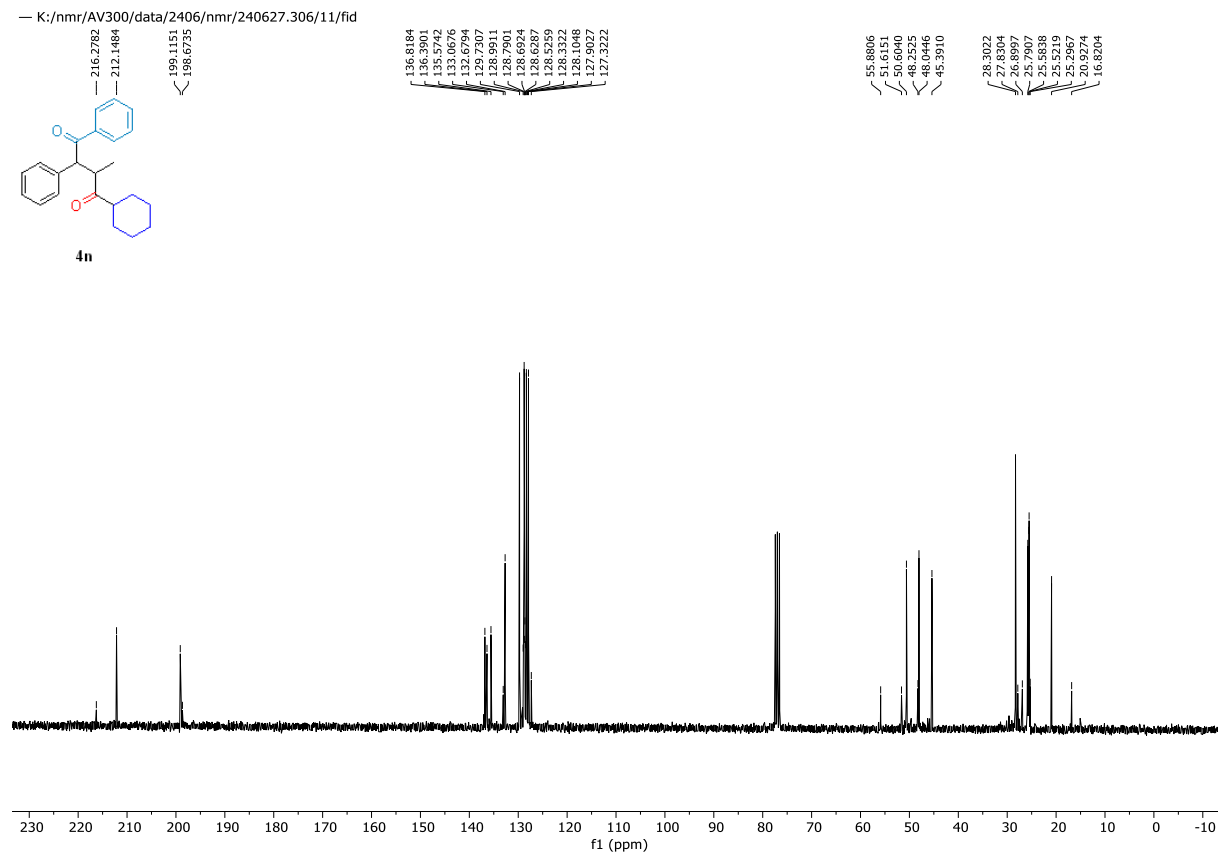




### 4n <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



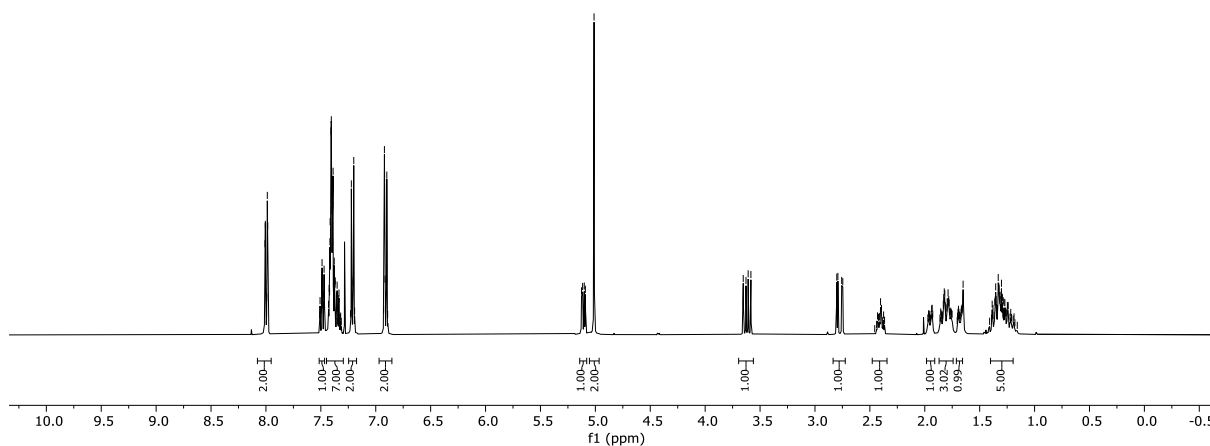
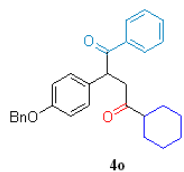
### 4n <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



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

K:/nmr/AV400/data/2407/nmr/240711.435/10/fid

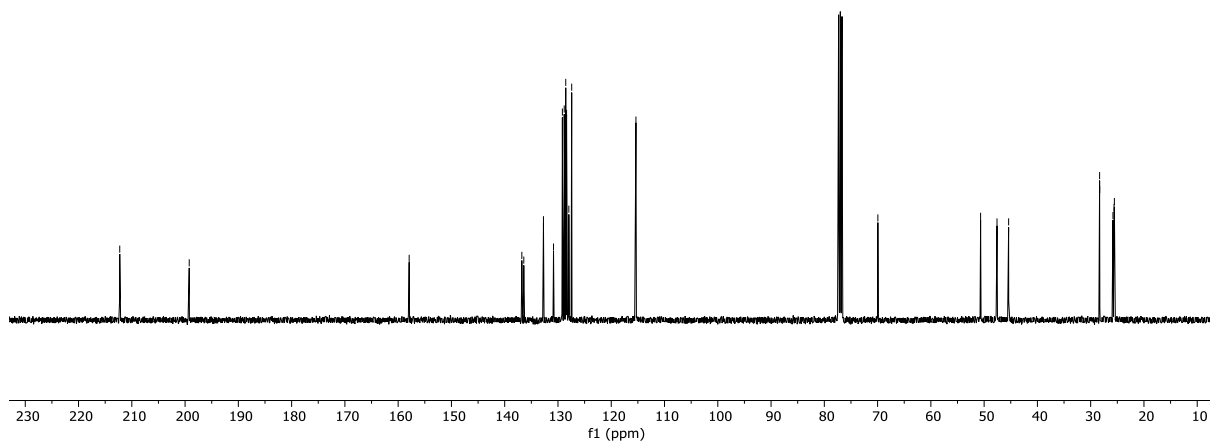
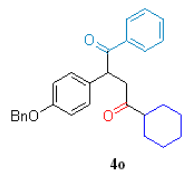
8.0085  
8.0055  
8.0016  
7.9927  
7.9876  
7.9839  
7.9826  
7.9822  
7.9837  
7.9738  
7.4705  
7.4672  
7.4595  
7.4542  
7.4505  
7.4454  
7.4154  
7.4116  
7.4077  
7.4047  
7.3996  
7.3961  
7.3926  
7.3890  
7.3855  
7.3830  
7.3780  
7.3756  
7.3739  
7.3712  
7.3699  
7.3689  
7.3659  
7.3517  
7.3435  
7.3376  
7.3348  
7.2221  
7.2167  
7.2105  
7.2002  
6.9210  
6.9157  
6.9044  
6.8992  
5.1159  
5.1100  
5.0999  
5.0920  
5.0120  
3.6532  
3.6383  
3.6085  
3.5835  
2.8013  
2.7972  
2.7567  
2.7465  
2.4028  
1.8314  
1.8272  
1.8225  
1.8176  
1.8157  
1.7955  
1.7879  
1.7814  
1.7781  
1.6508  
1.3861  
1.3817  
1.3814  
1.3841  
1.3507  
1.3310  
1.3263  
1.3232  
1.3164  
1.3157  
1.3015  
1.3015  
1.2931  
1.2853  
1.2777  
1.2705  
1.2477  
1.2406



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

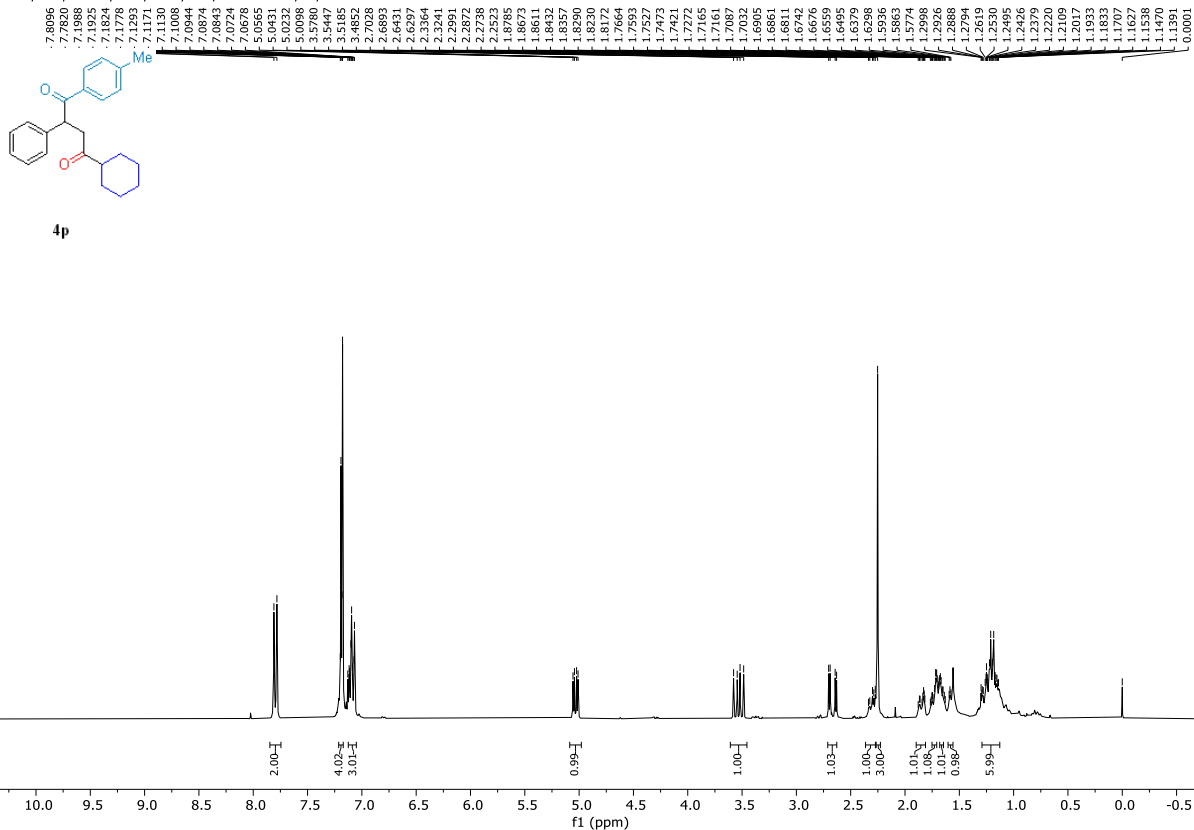
K:/nmr/AV400/data/2407/nmr/240711.435/11/fid

212.2636  
199.2204  
157.9360  
136.7826  
136.4167  
132.7244  
130.8363  
129.1728  
128.8249  
128.8249  
128.3682  
127.9595  
127.4243  
115.3634  
69.9492  
50.6532  
47.5839  
45.4038  
28.3242  
28.3078  
25.6151  
25.5489



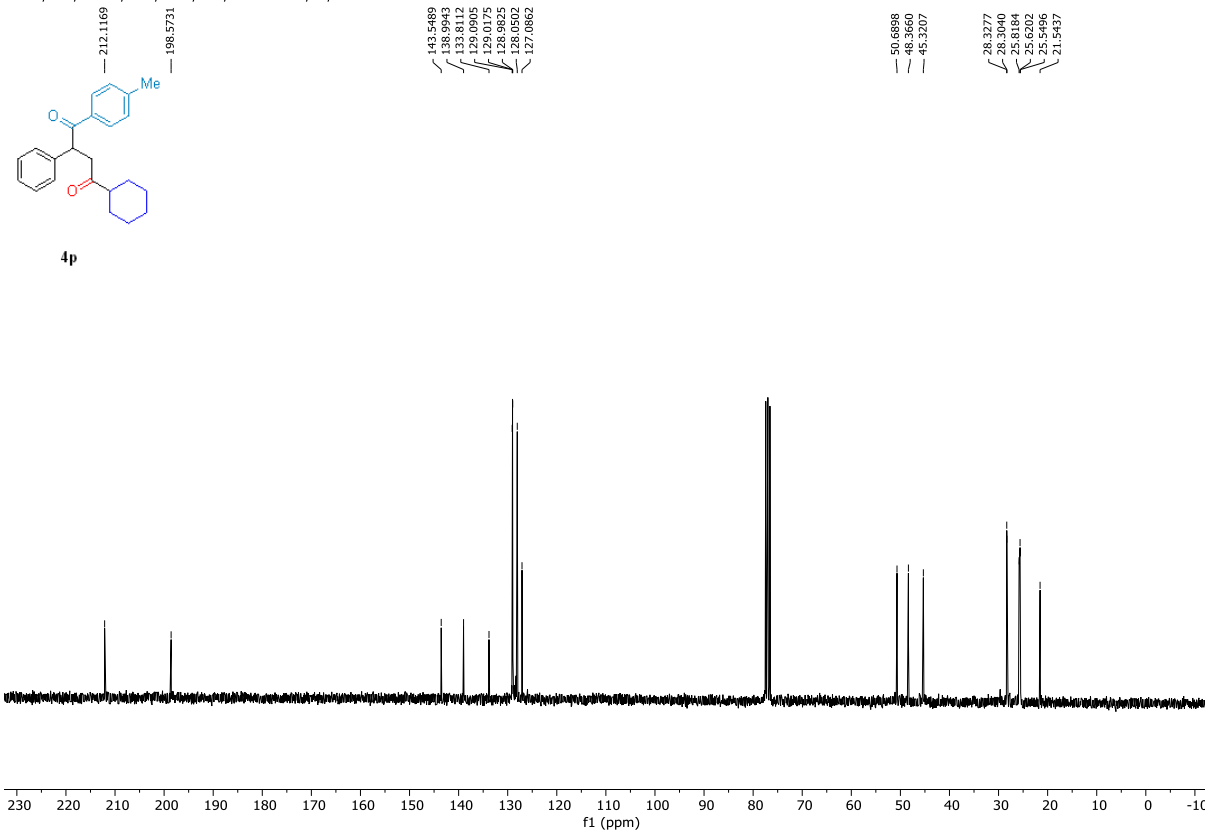
### 4p <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV300/data/2405/nmr/240531.305/10/fid



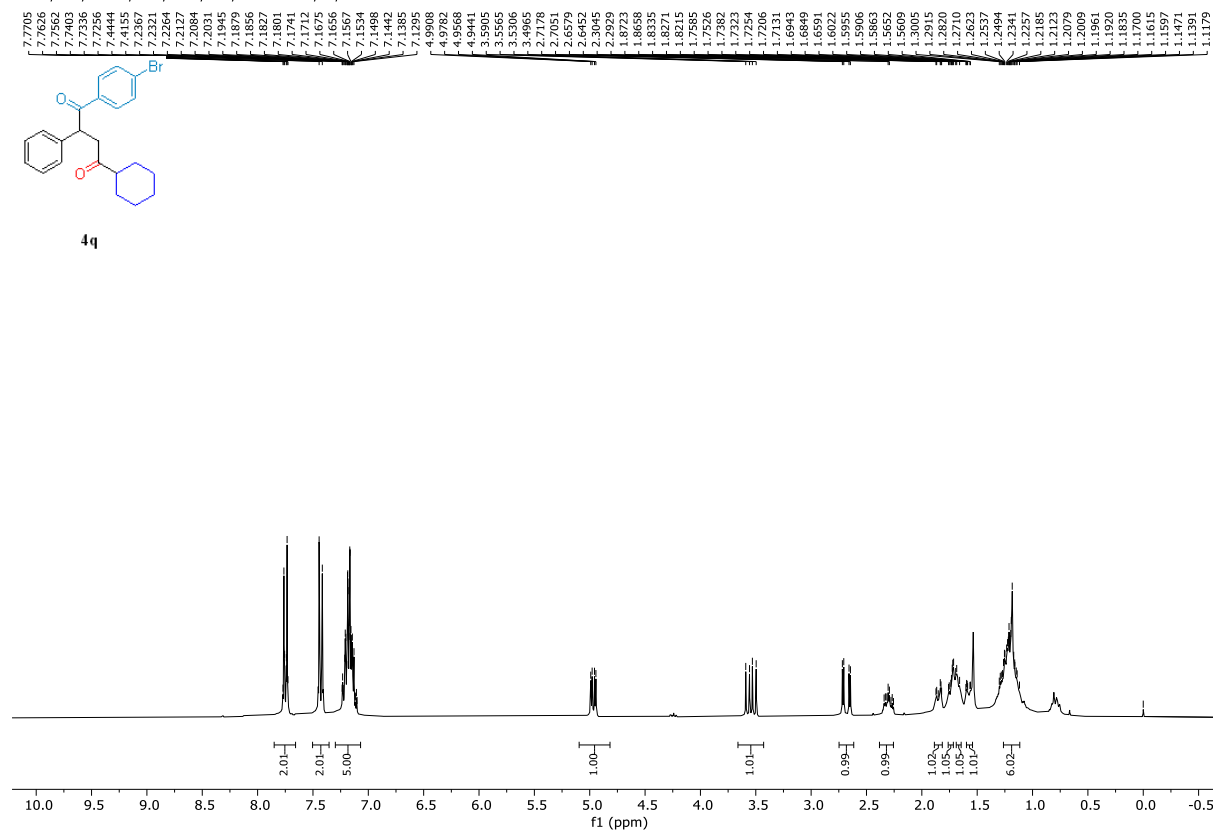
### 4p <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV300/data/2405/nmr/240531.305/11/fid



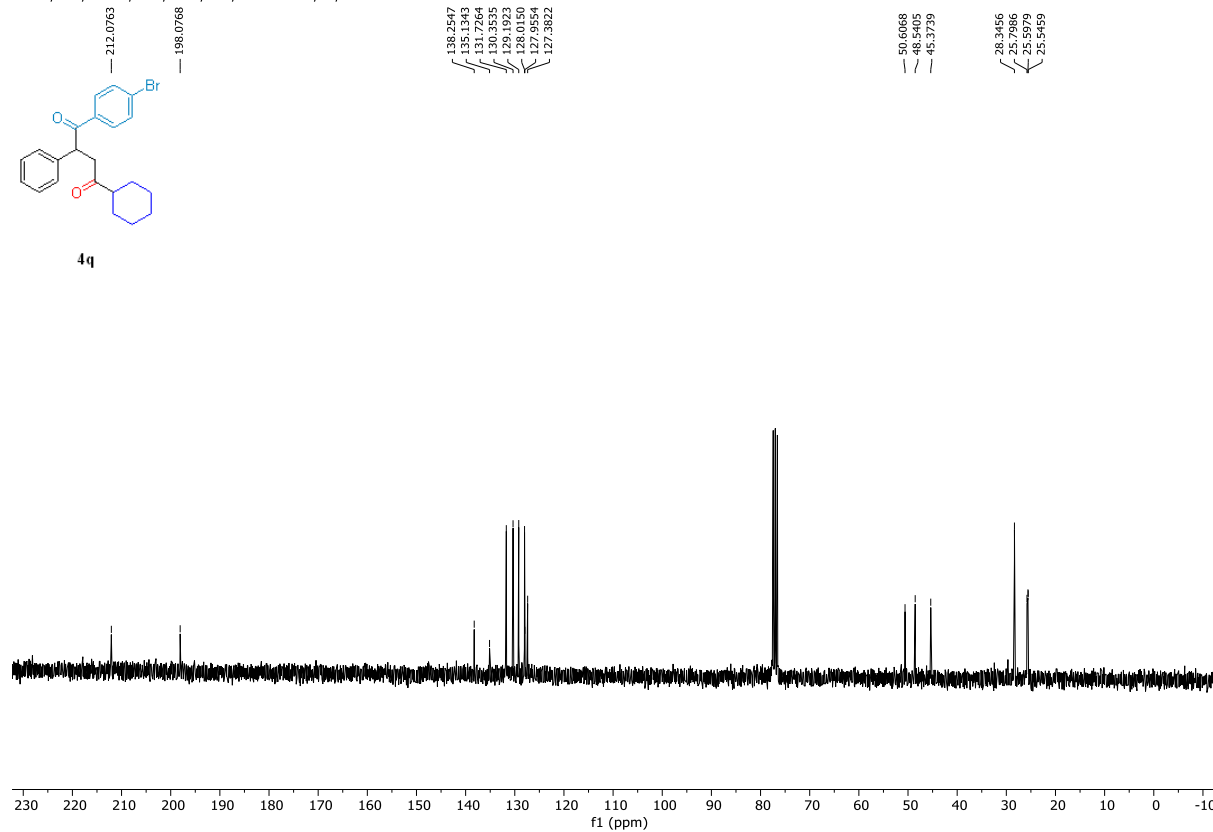
### 4q <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV300/data/2406/nmr/240603.303/10/fid

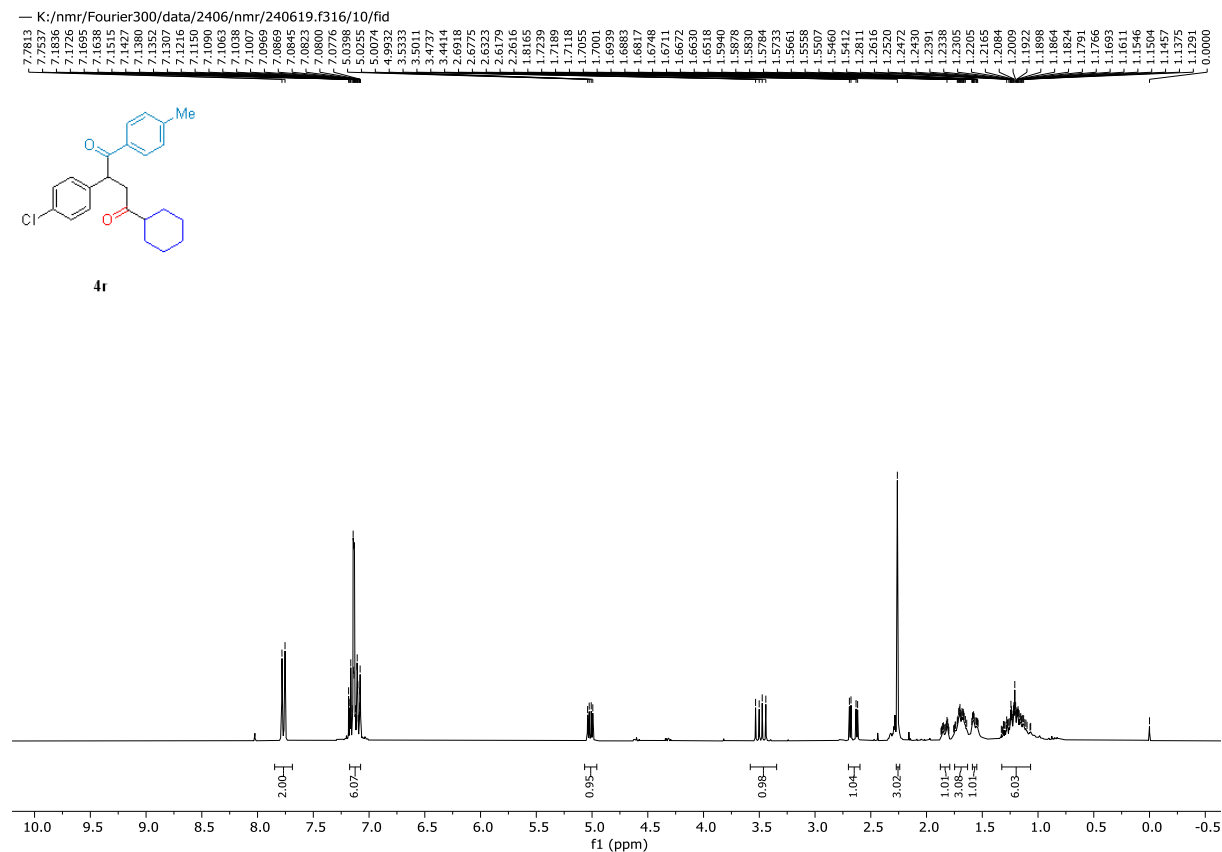


### 4q <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)

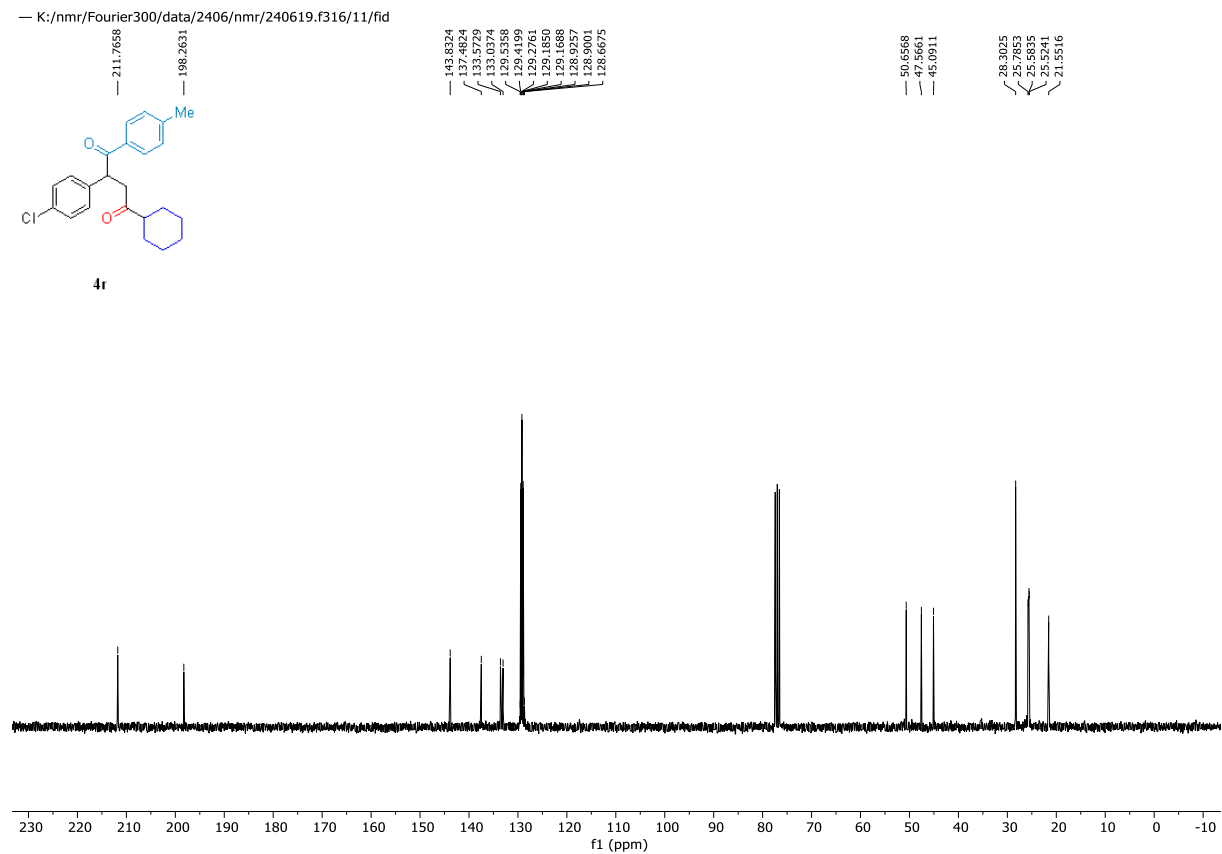
— K:/nmr/AV300/data/2406/nmr/240603.303/11/fid



### 4r <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



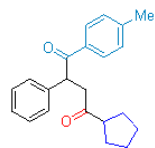
### 4r <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



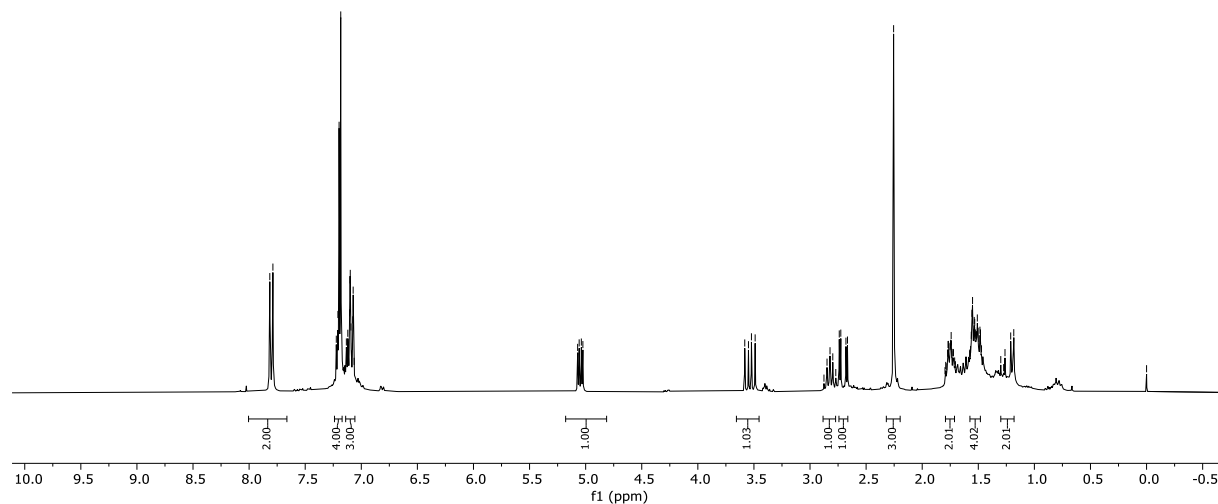
### 4s <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)

— K:/nmr/Fourier300/data/2406/nmr/240619.f315/10/fid

7.8155  
7.7879  
7.2228  
7.2083  
7.1979  
7.1831  
7.1331  
7.1277  
7.1148  
7.1050  
7.1008  
7.0984  
7.0924  
7.0866  
7.0798  
7.0759  
7.0725  
7.0698  
7.0637  
5.0716  
5.0579  
5.0483  
5.0368  
5.0248  
3.5817  
3.5487  
3.5222  
3.4891  
2.8758  
2.8484  
2.8406  
2.7921  
2.7704  
2.7393  
2.7256  
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2.6661  
2.2949  
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1.5429  
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1.5319  
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1.5193  
1.5157  
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1.4954  
1.4852  
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1.2996  
1.2740  
1.2619  
1.2111  
1.1837  
0.0000



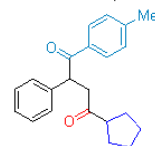
4s



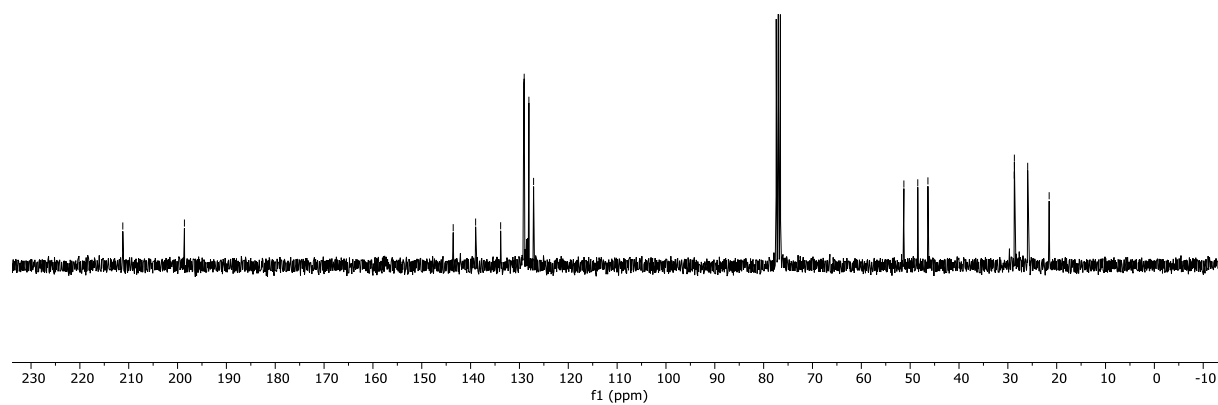
### 4s <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)

— K:/nmr/Fourier300/data/2406/nmr/240619.f315/11/fid

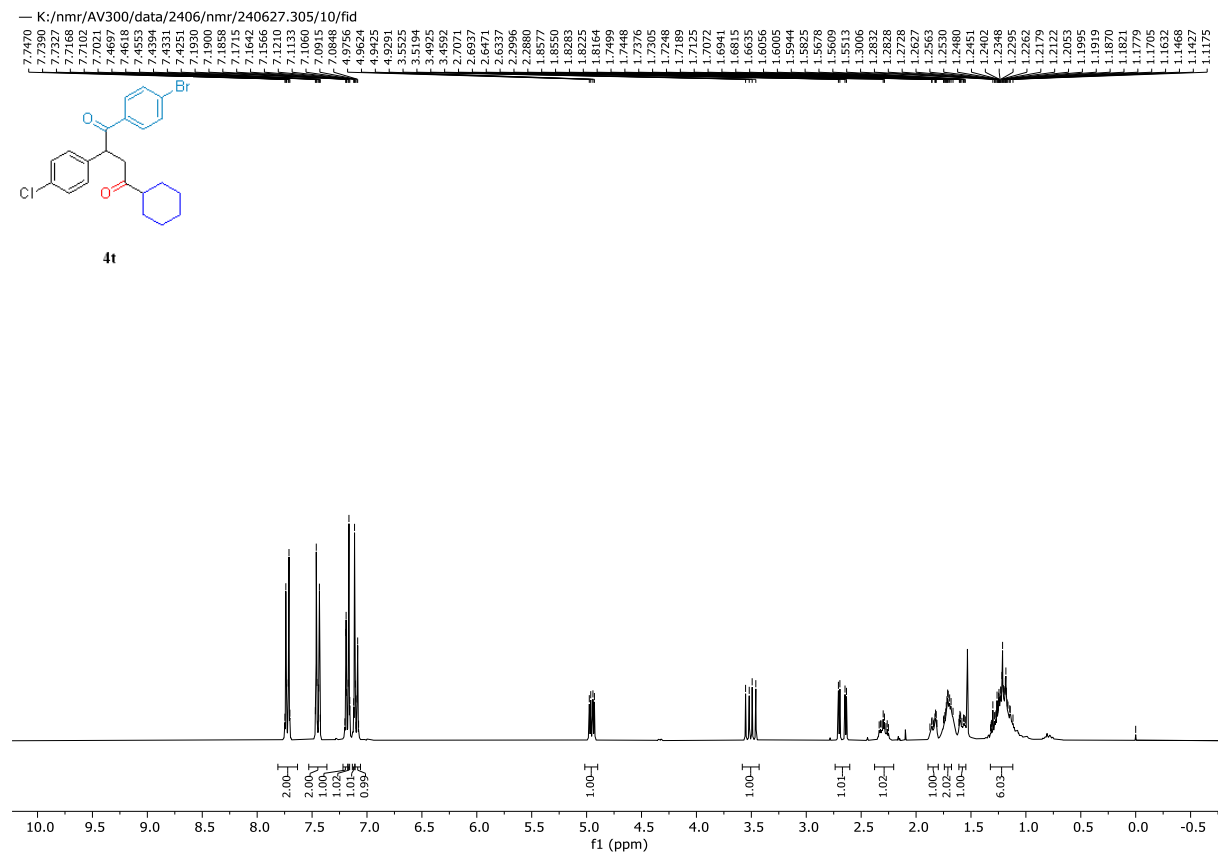
211.2114  
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143.5624  
136.9718  
133.8480  
129.1076  
129.0289  
128.9870  
128.0661  
127.1054  
51.2903  
48.4433  
46.3660  
28.6995  
28.6050  
25.9405  
21.5426



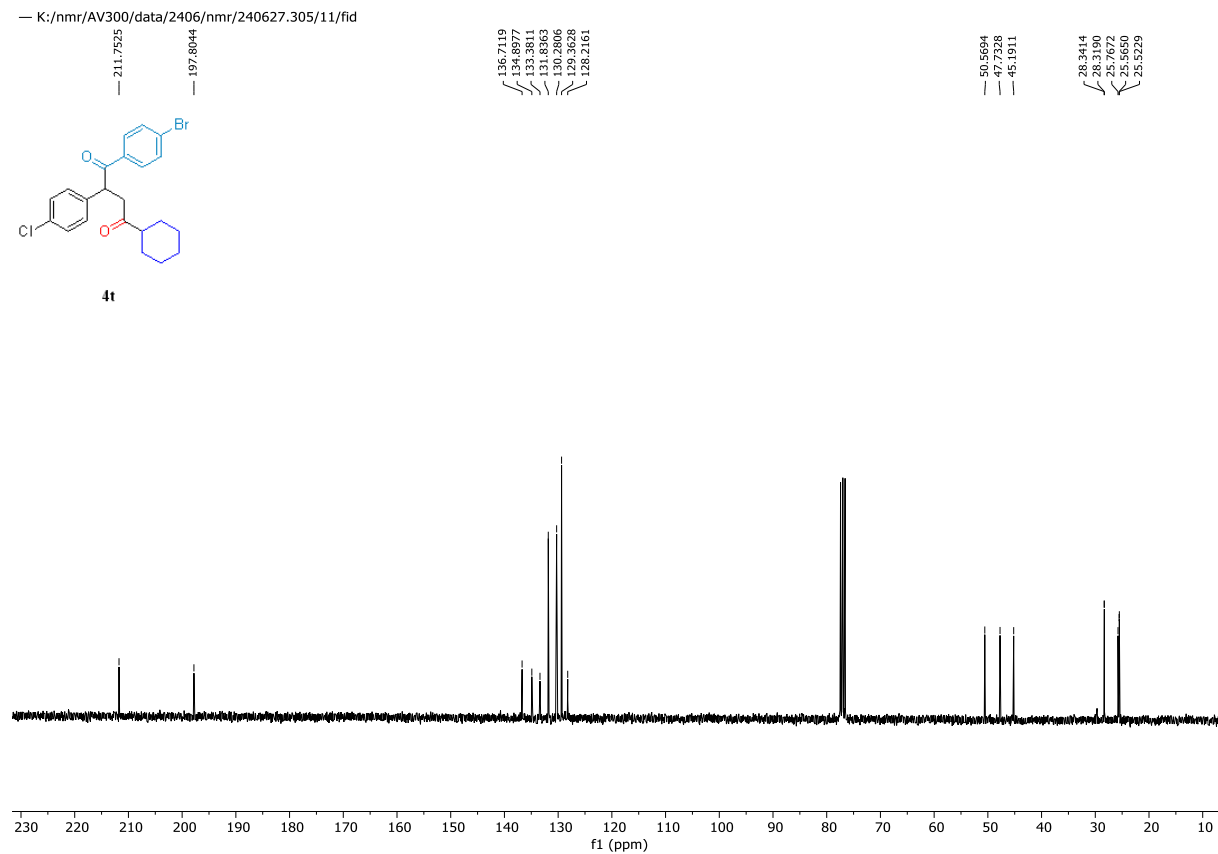
4s



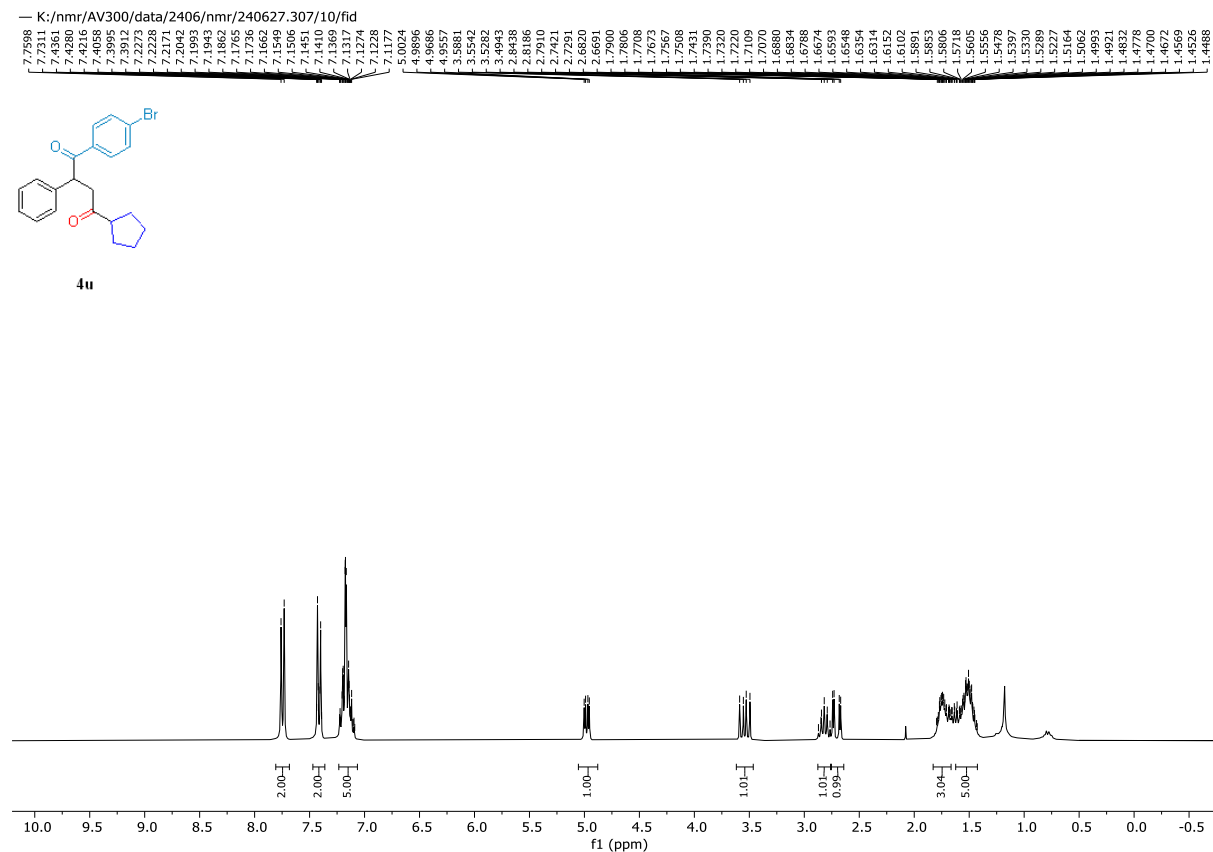
### 4t <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



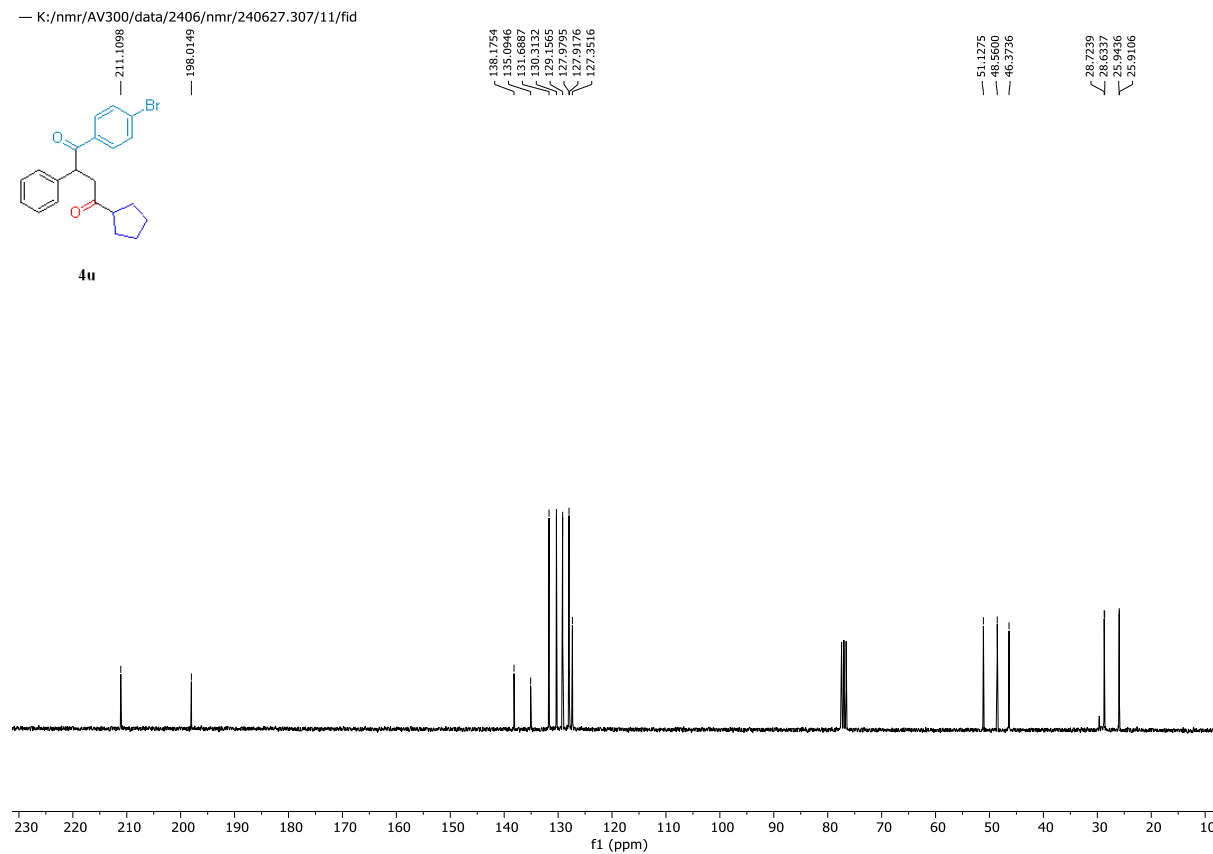
### 4t <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



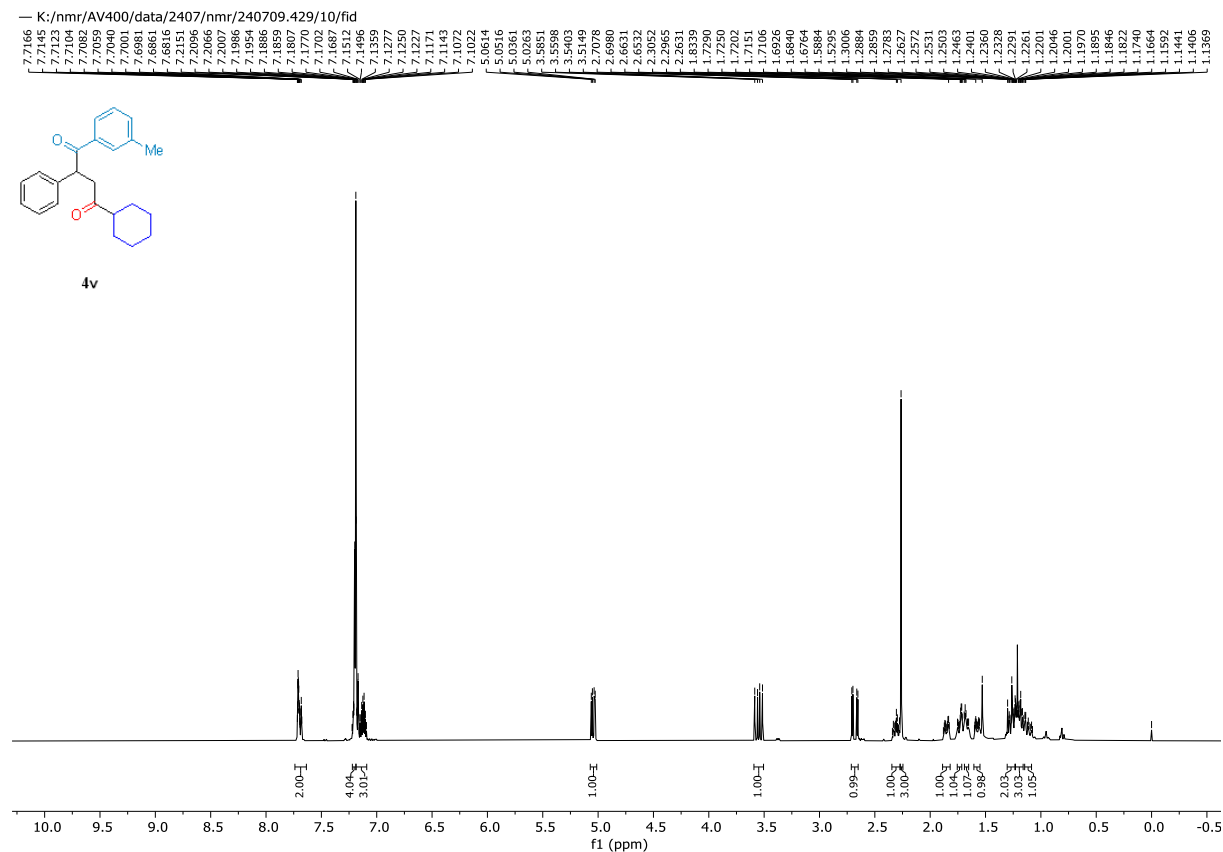
### 4u <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



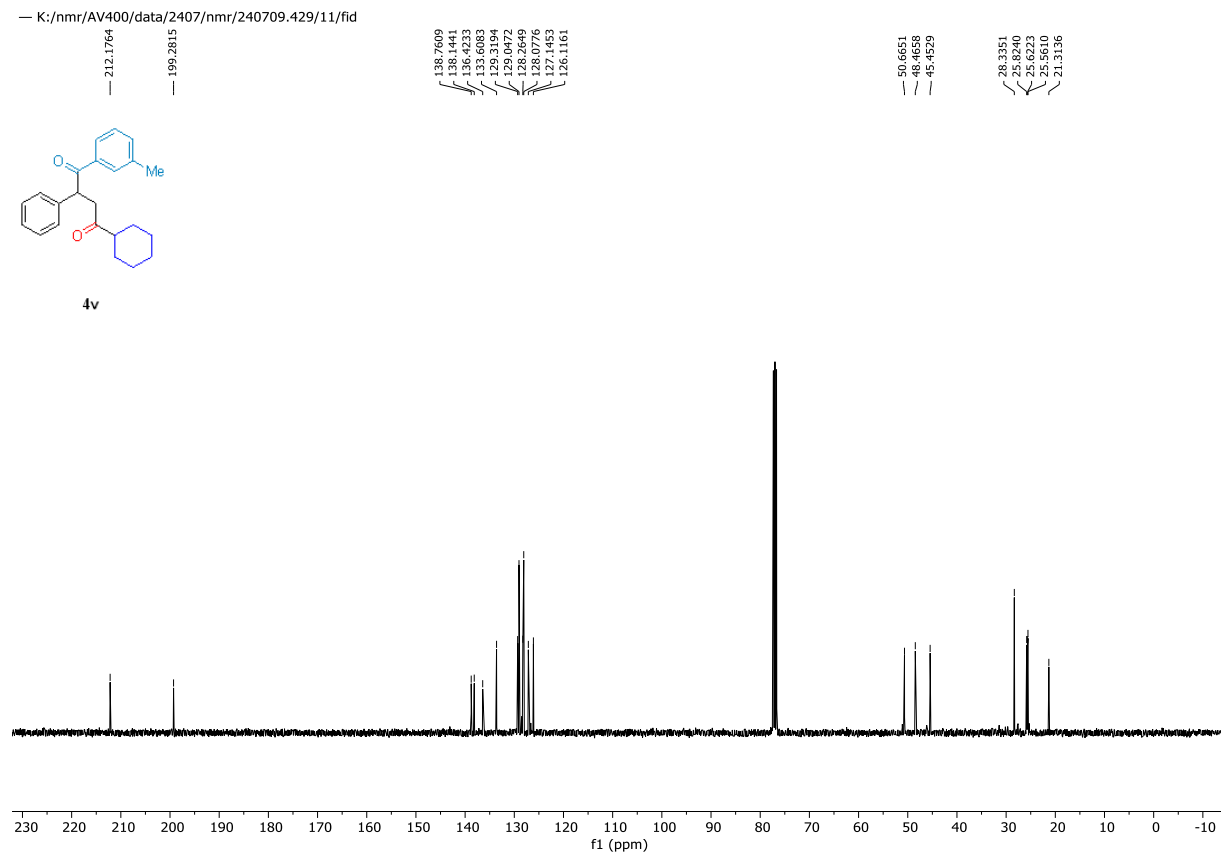
### 4u <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



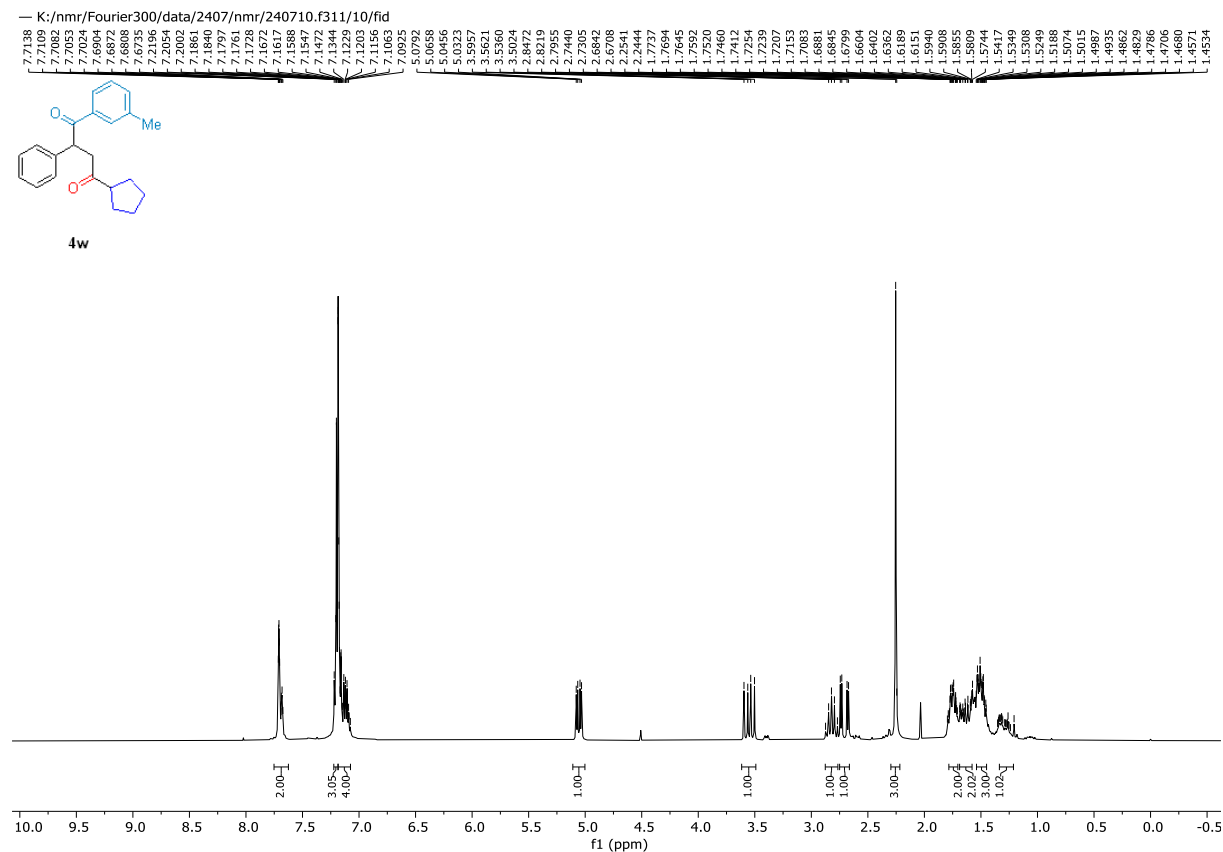
### 4v <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



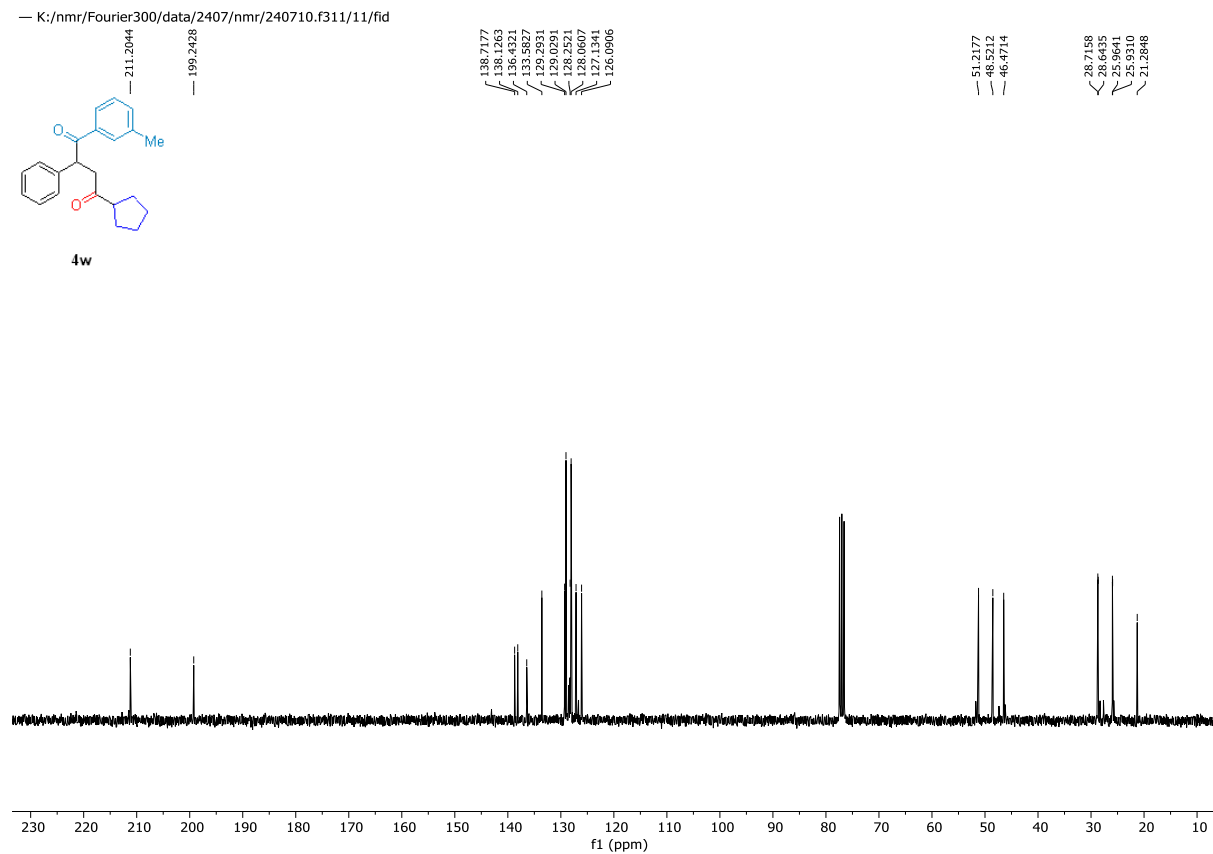
### 4v <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)



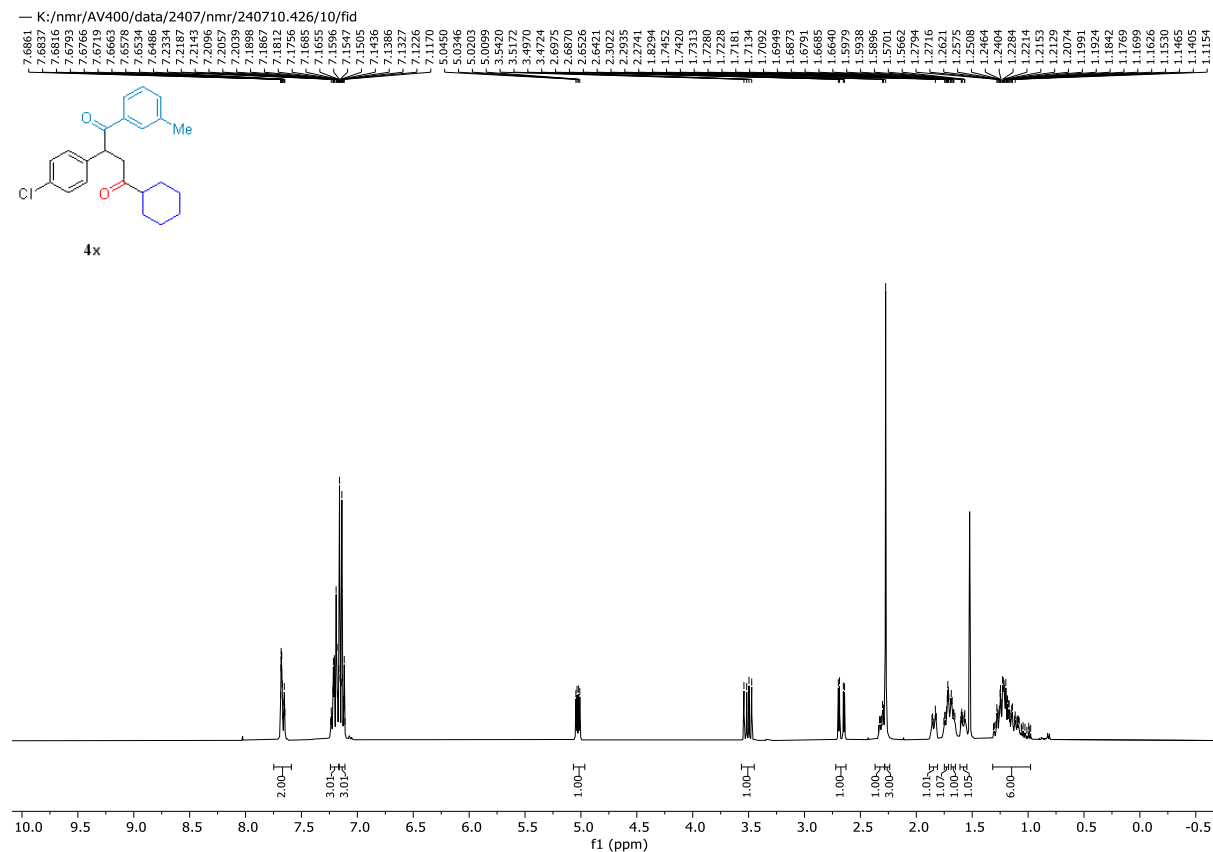
### 4w <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



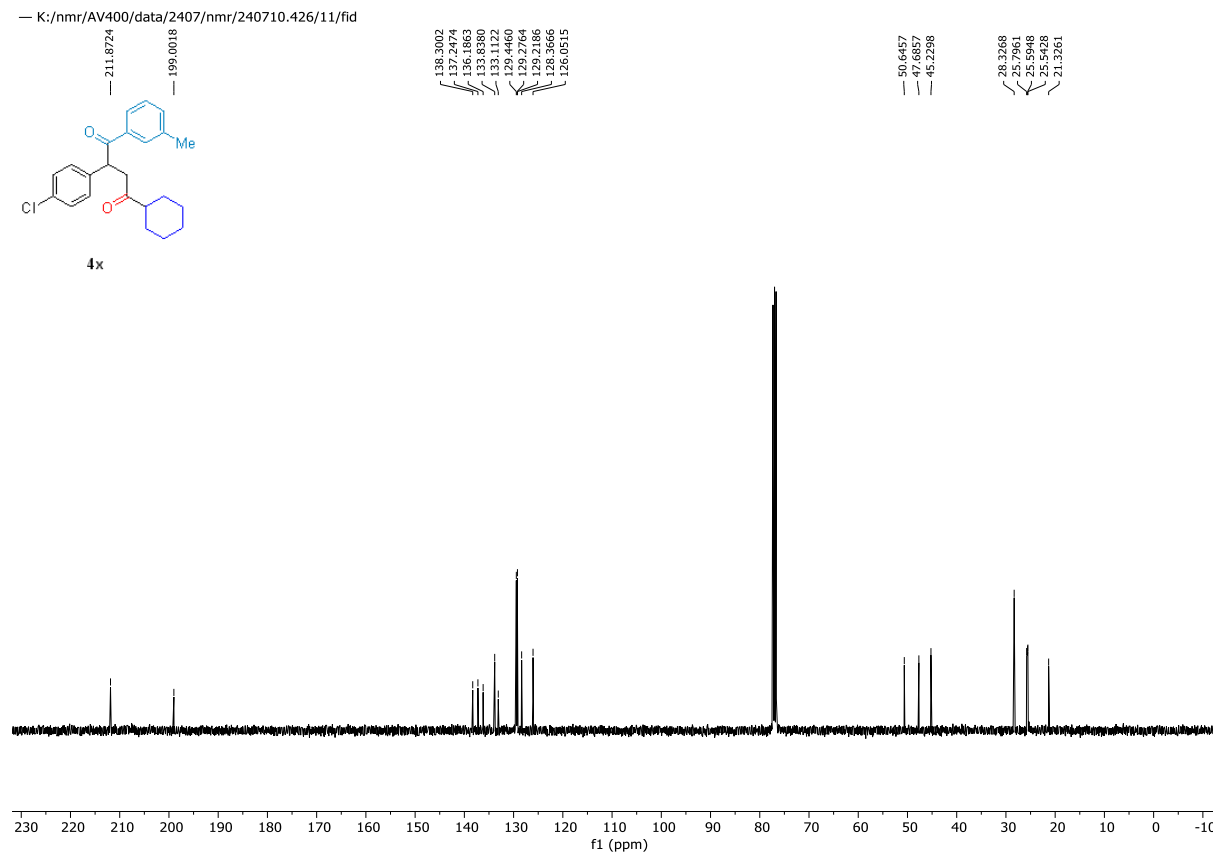
### 4w <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



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

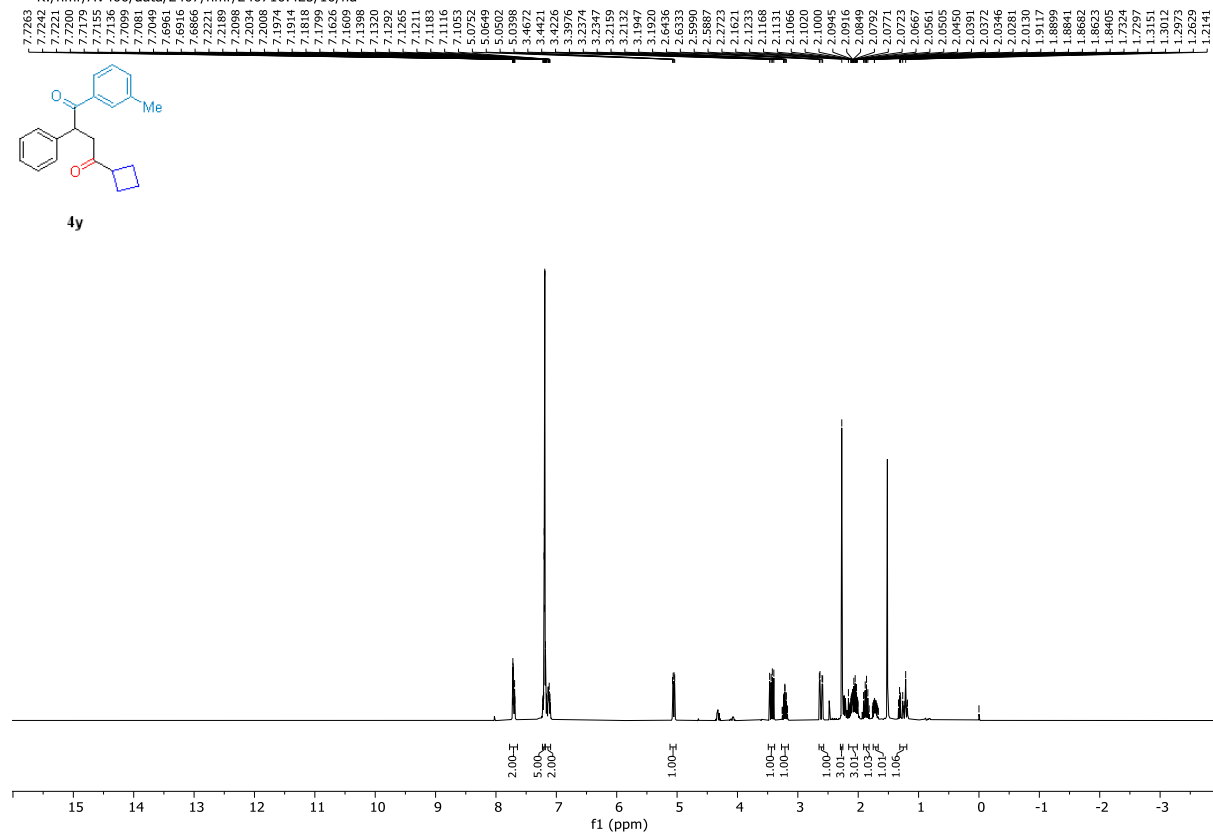


### 4x <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)



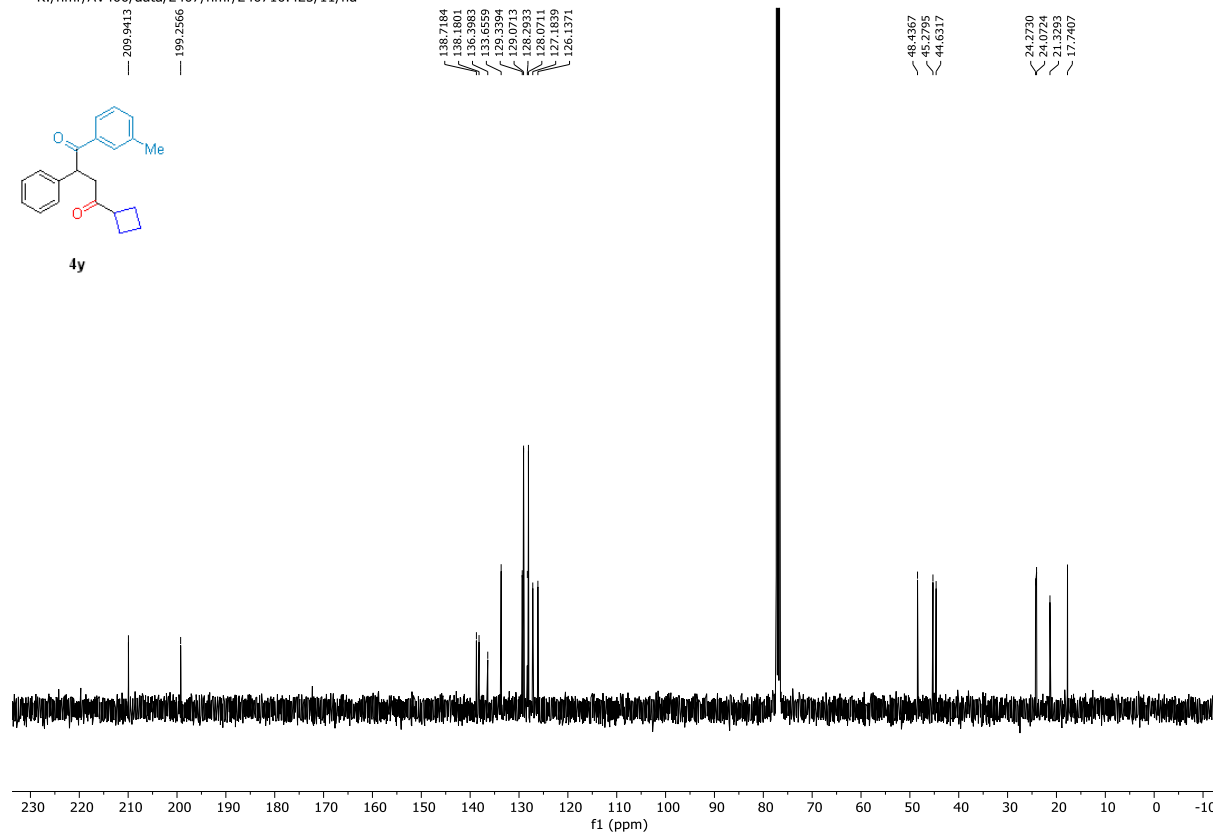
### 4y <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV400/data/2407/nmr/240710.425/10/fid



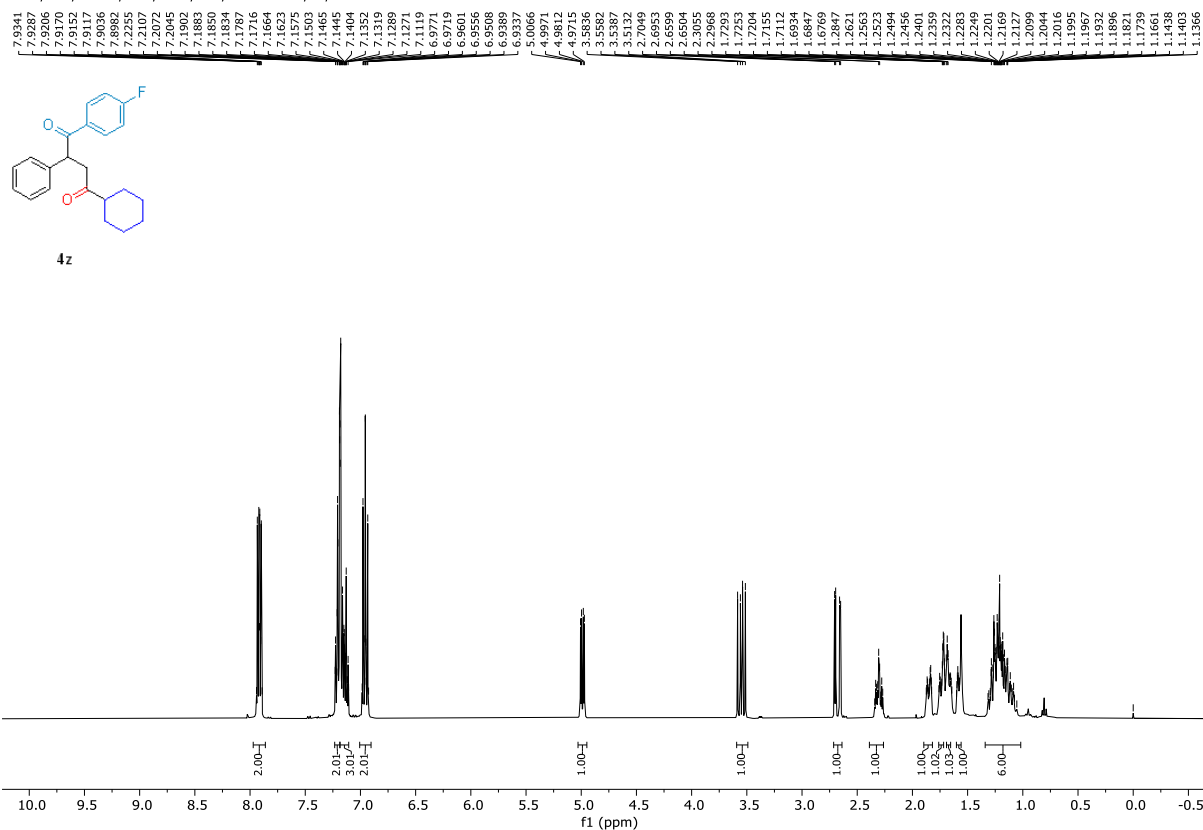
### 4y <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV400/data/2407/nmr/240710.425/11/fid



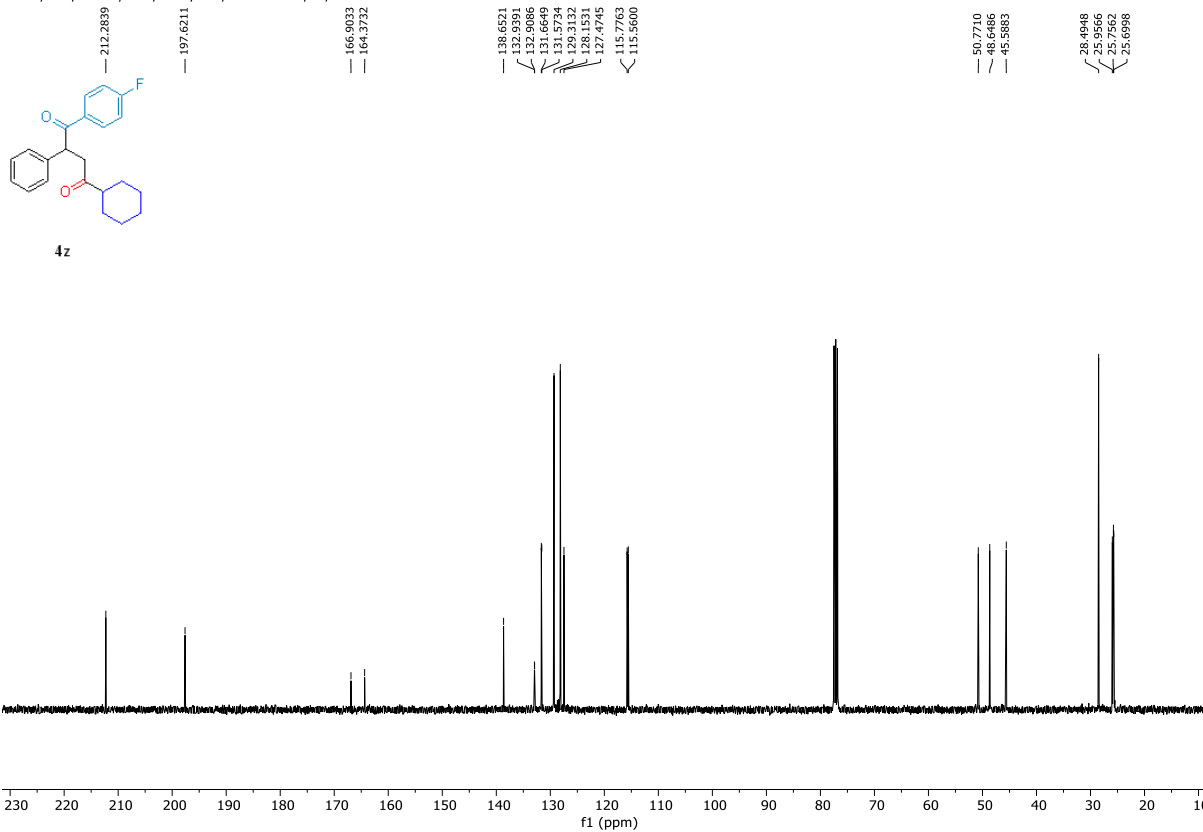
### 4z <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV400/data/2407/nmr/240709.430/10/fid



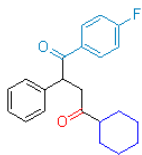
### 4z <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV400/data/2407/nmr/240709.430/11/fid

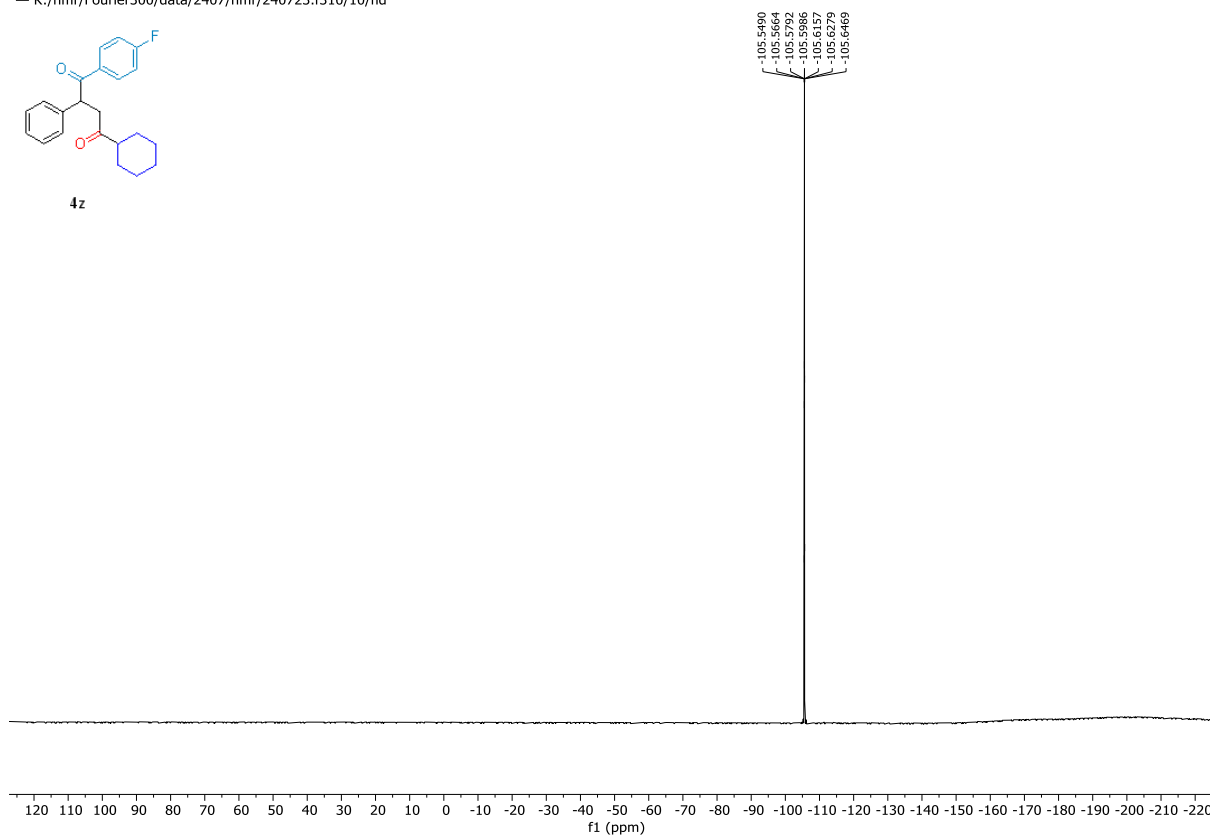


**4z**  $^{13}\text{F}$  NMR (382 MHz,  $\text{CDCl}_3$ )

— K:/nmr/Fourier300/data/2407/nmr/240723.f310/10/fid



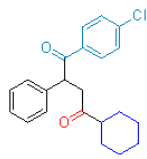
**4z**



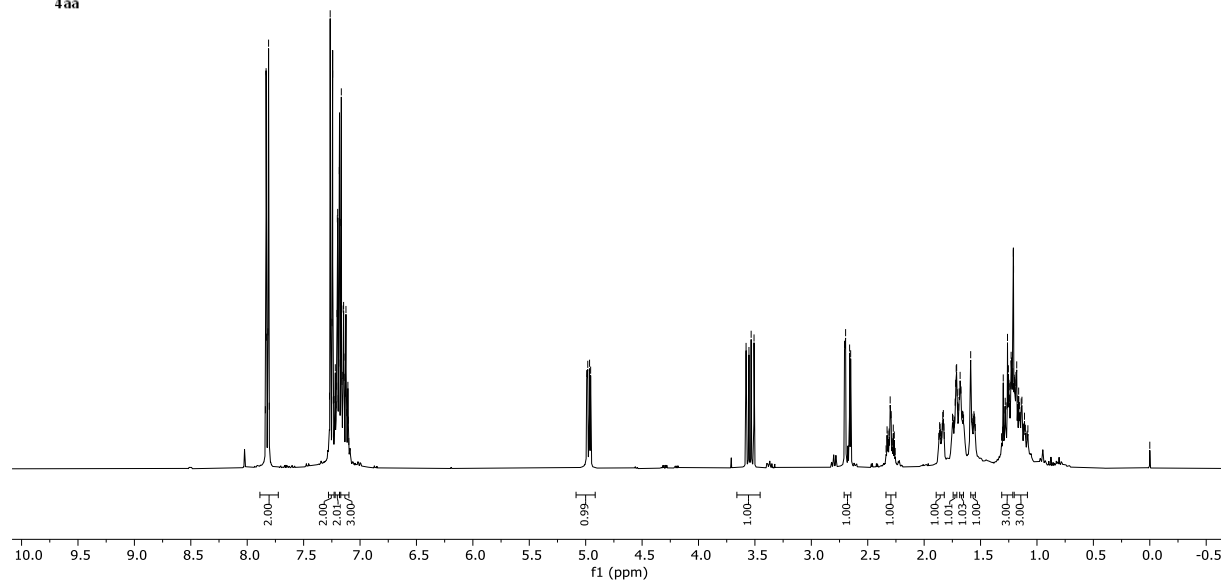
### 4aa <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV400/data/2407/nmr/240709.431/10/fid

7.8326, 7.8276, 7.8158, 7.8107, 7.8046, 7.2707, 7.2646, 7.2479, 7.2430, 7.2368, 7.2223, 7.2110, 7.2059, 7.2008, 7.2011, 7.1984, 7.1935, 7.1890, 7.1839, 7.1820, 7.1769, 7.1594, 7.1534, 7.1492, 7.1458, 7.1427, 7.1406, 7.1352, 7.1308, 7.1275, 7.1247, 7.1077, 4.9911, 4.9816, 4.9656, 4.9504, 4.9352, 3.5528, 3.5334, 3.5078, 2.7059, 2.6962, 2.6607, 2.6454, 2.3011, 1.7251, 1.7207, 1.7159, 1.7119, 1.7071, 1.7035, 1.6886, 1.6811, 1.6733, 1.5876, 1.2989, 1.2815, 1.2737, 1.2612, 1.2489, 1.2461, 1.2425, 1.2368, 1.2327, 1.2292, 1.2255, 1.2255, 1.2166, 1.2134, 1.2092, 1.2076, 1.2017, 1.1992, 1.1944, 1.1899, 1.1861, 1.1795, 1.1701, 1.1623, 1.1556, 1.1398, 1.1333

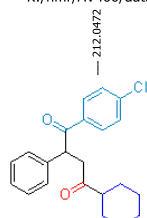


4aa

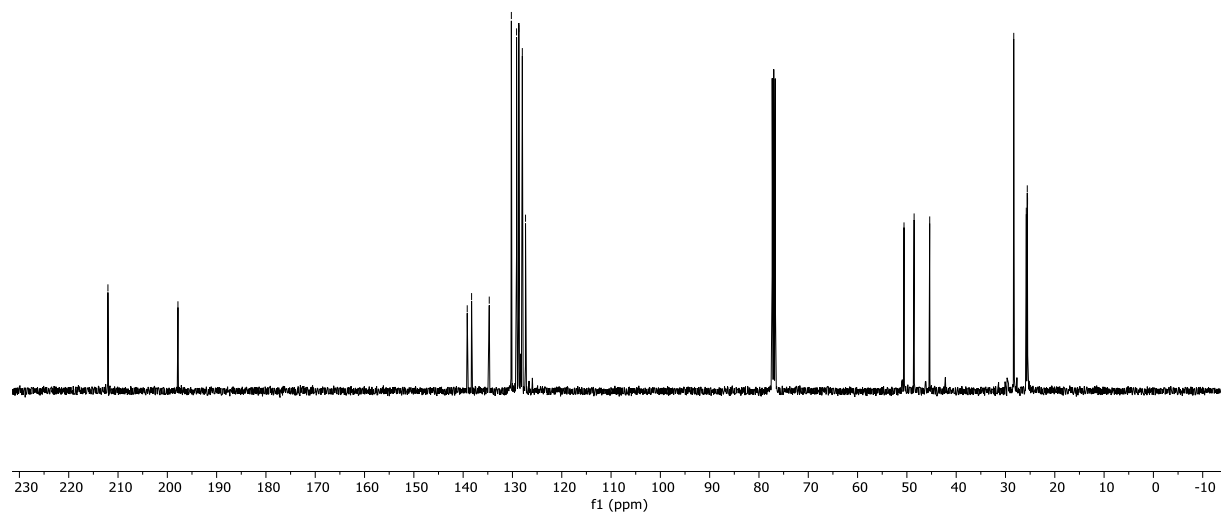


### 4aa <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)

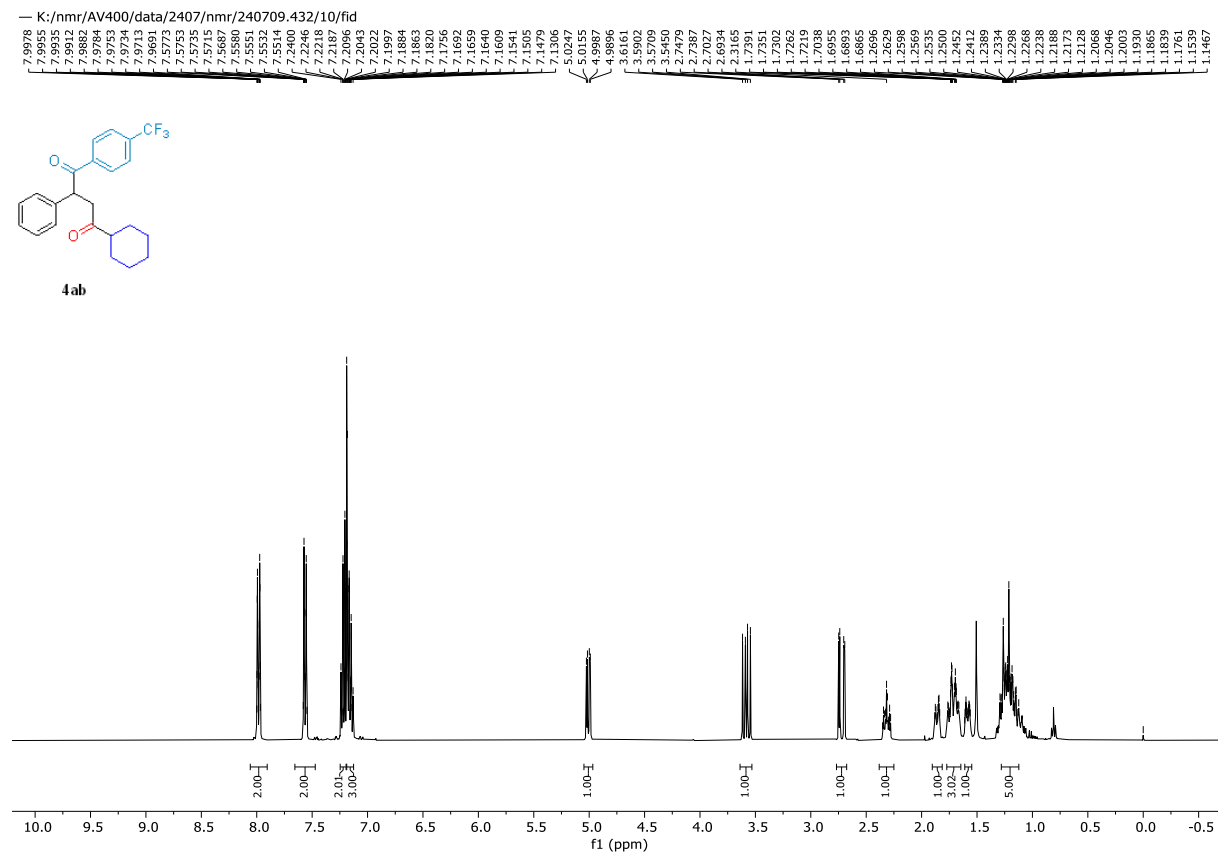
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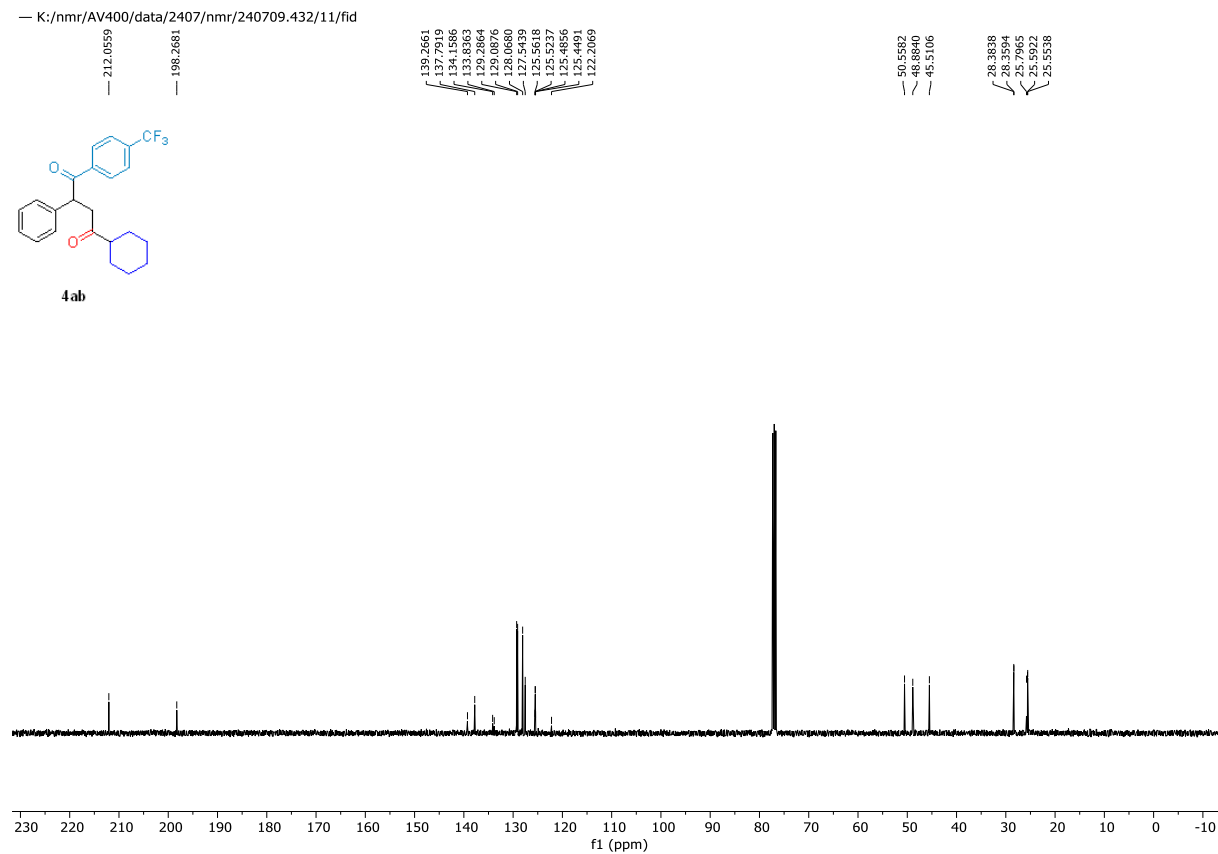
4aa



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

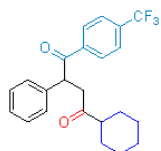


### 4ab <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)



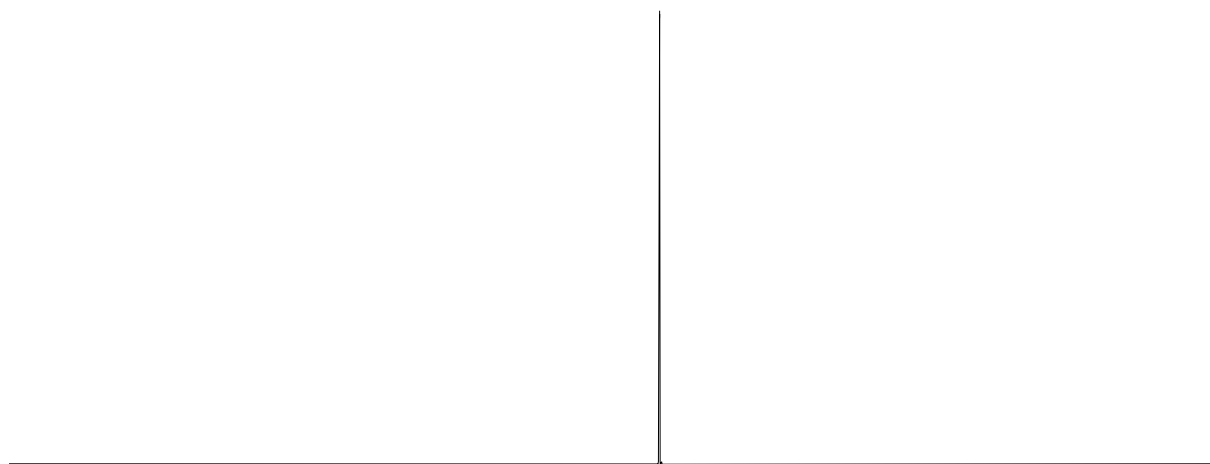
### 4ab <sup>13</sup>F NMR (282 MHz, CDCl<sub>3</sub>)

— K:/nmr/Fourier300/data/2407/nmr/240723.f311/10/fid



**4ab**

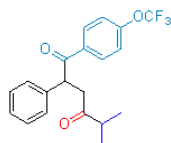
— 63.1869



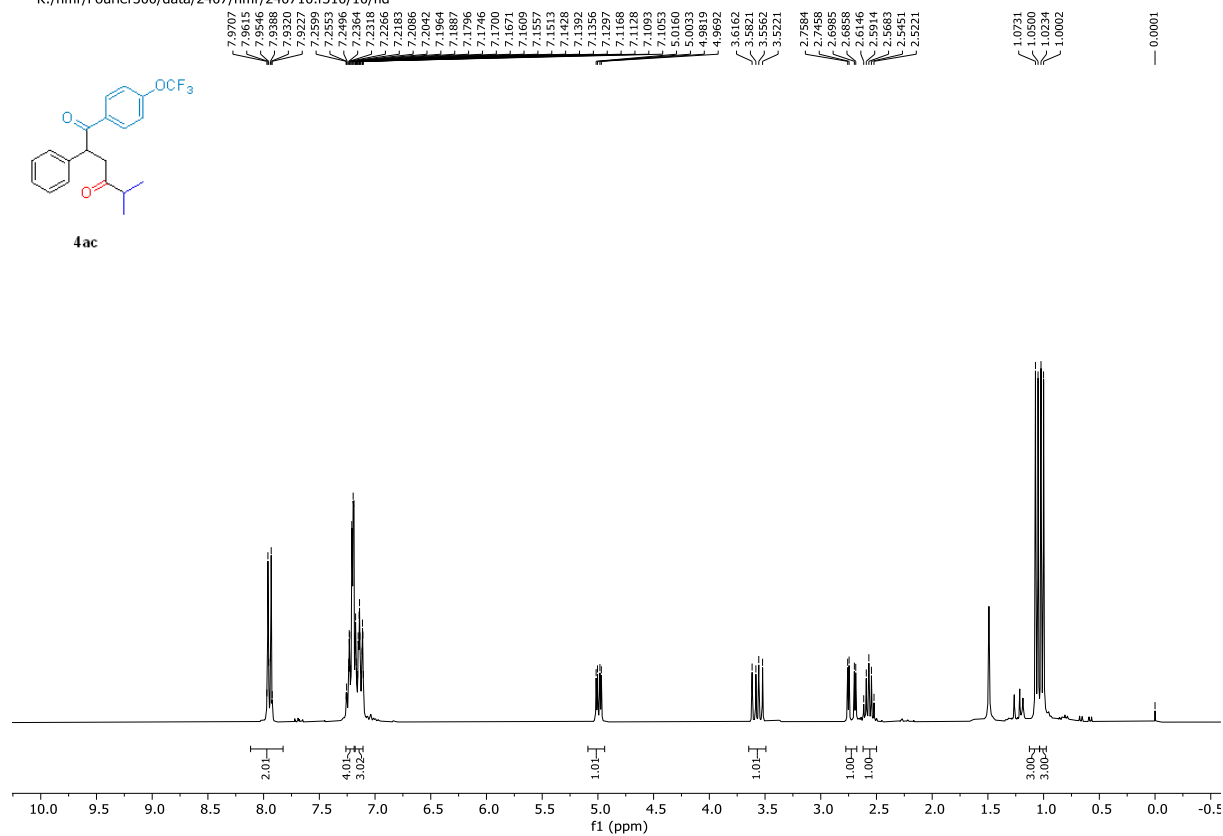
120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220  
f1 (ppm)

### 4ac <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)

— K:/nmr/Fourier300/data/2407/nmr/240710.f310/10/fid

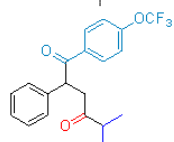


4ac

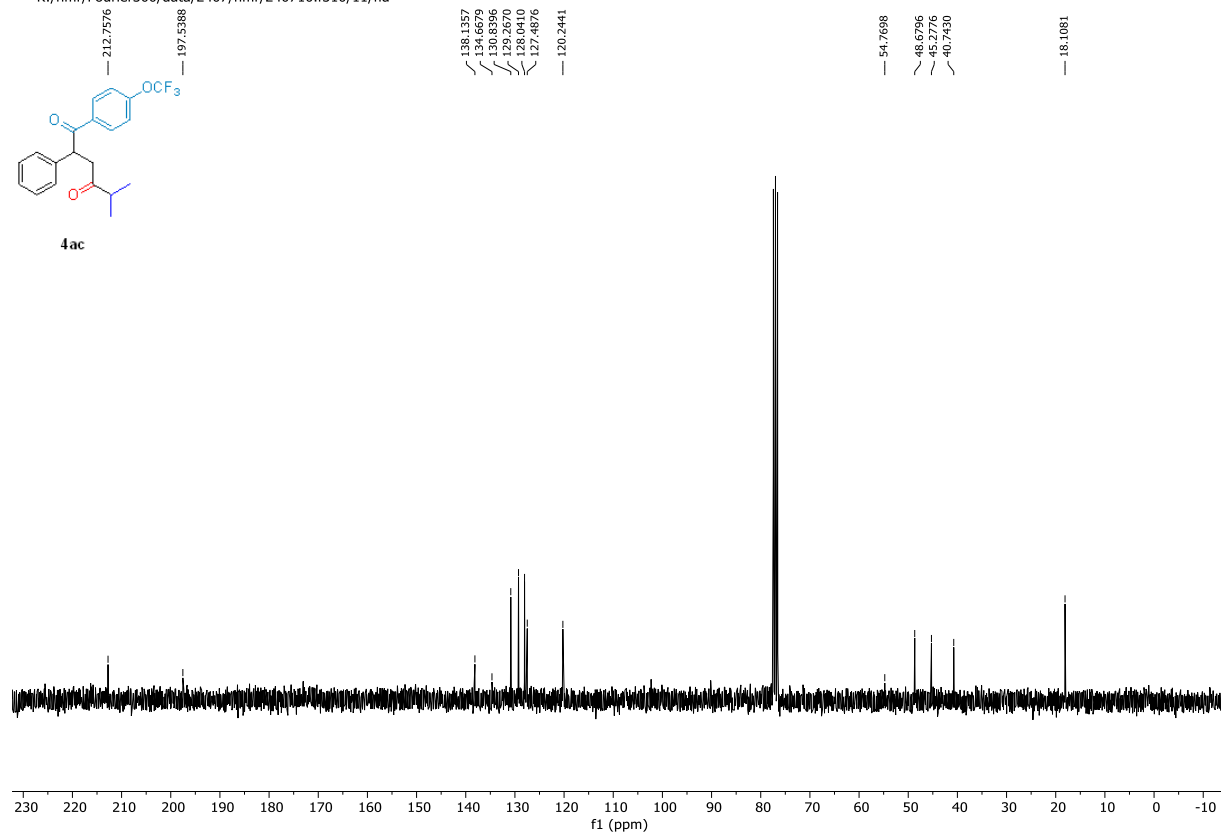


### 4ac <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)

— K:/nmr/Fourier300/data/2407/nmr/240710.f310/11/fid

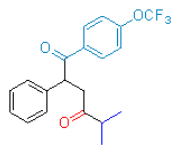


4ac

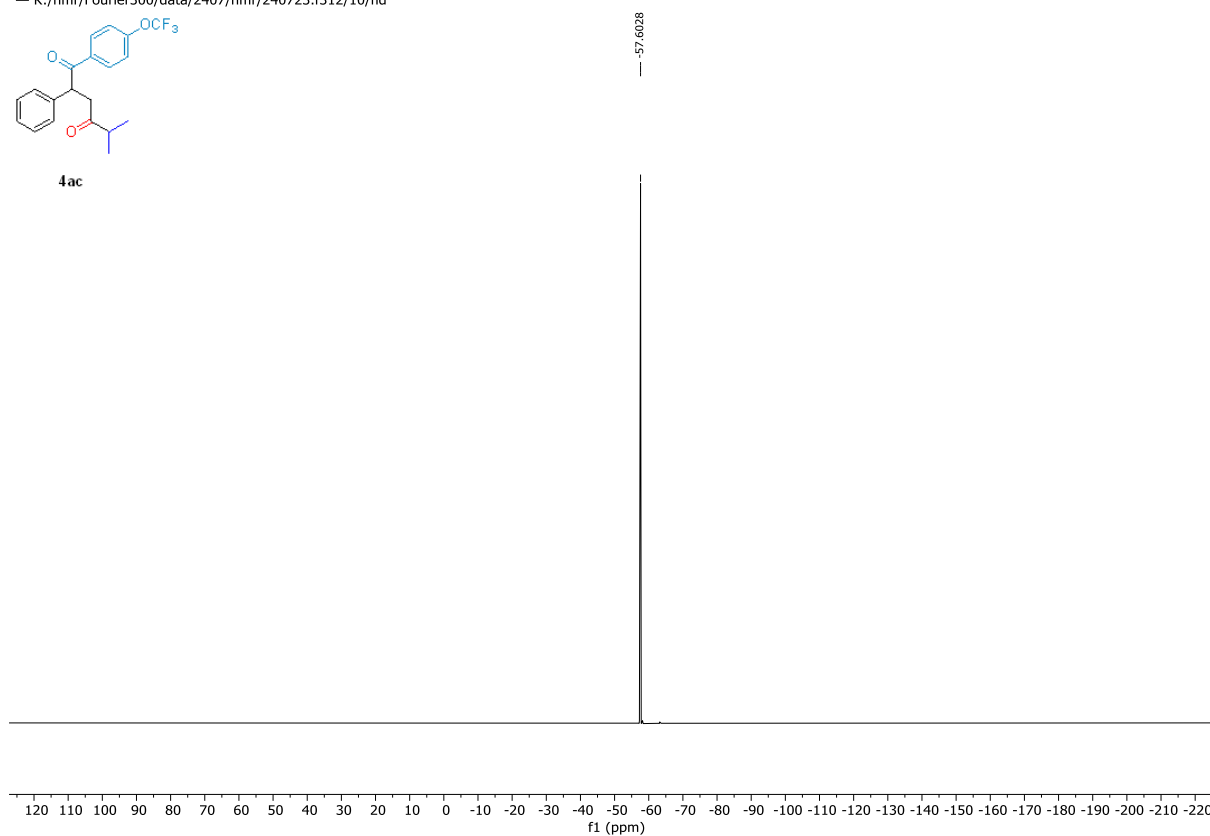


**4ac**  $^{13}\text{F}$  NMR (282 MHz,  $\text{CDCl}_3$ )

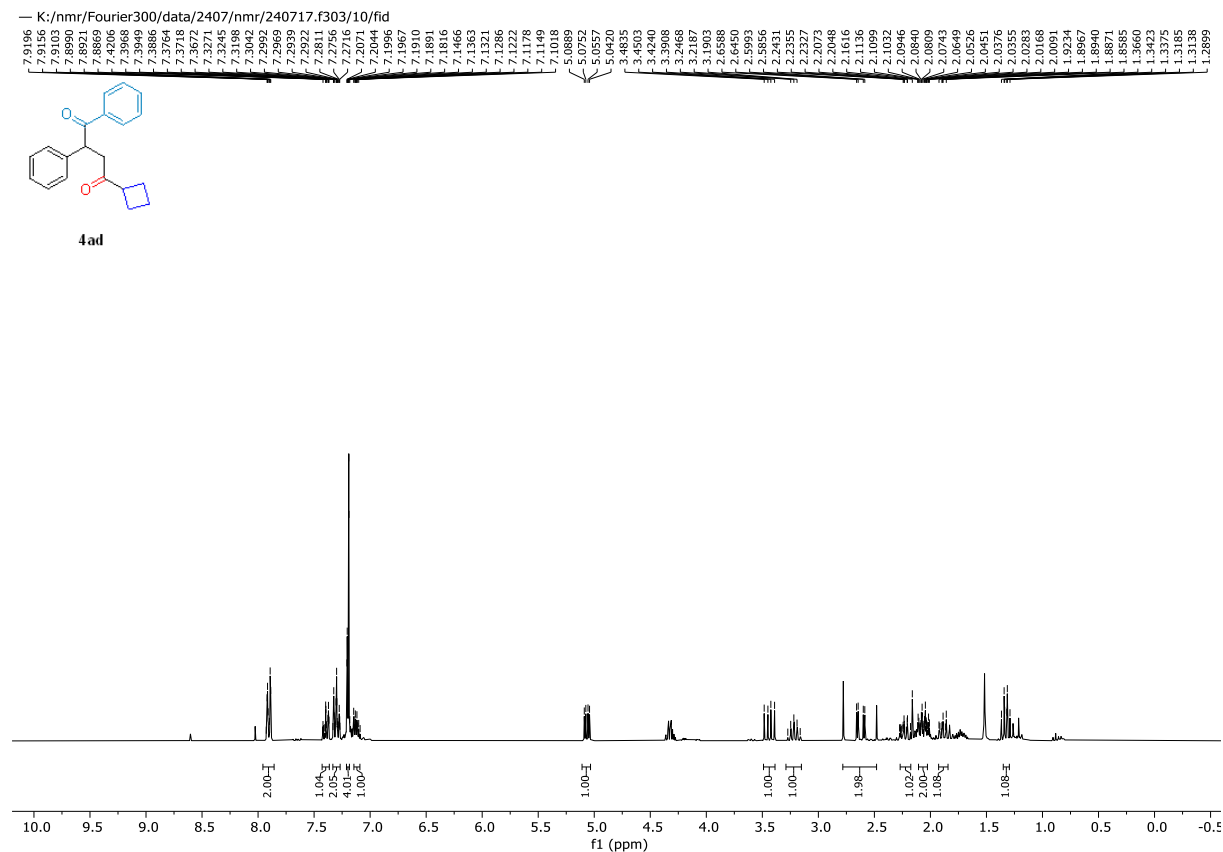
— K:/nmr/Fourier300/data/2407/nmr/240723.f312/10/fid



**4ac**



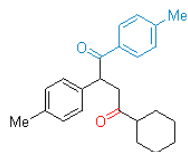
### 4ad <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



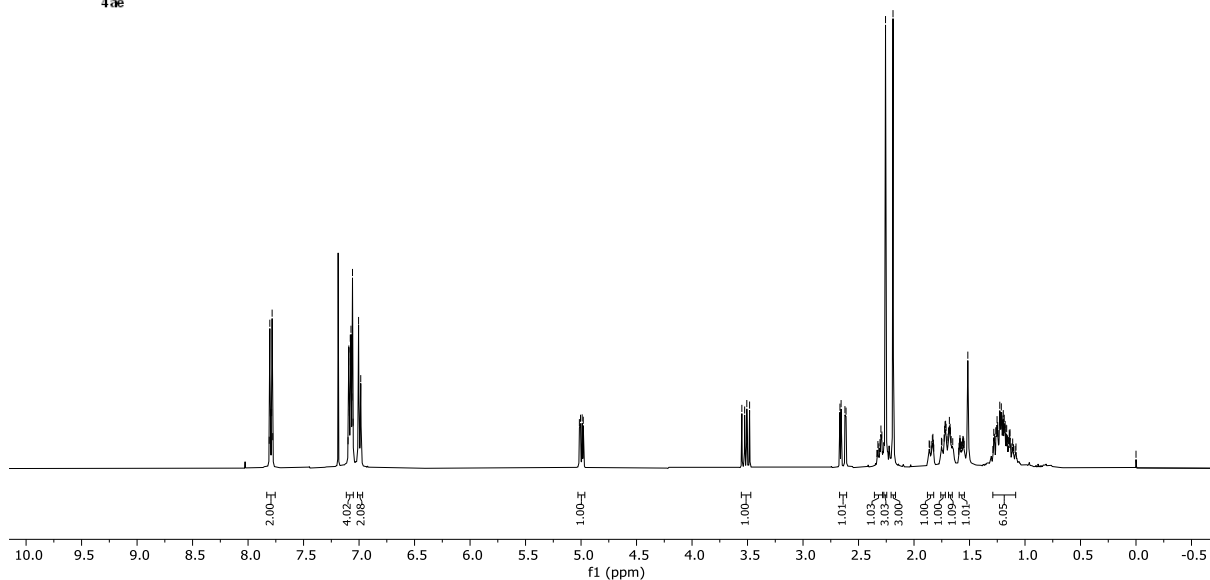
### 4ae <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV400/data/2408/nmr/240806.409/10/fid

7.8091  
7.8039  
7.7993  
7.7879  
7.7832  
7.7780  
7.0899  
7.0846  
7.0920  
7.0882  
7.0853  
7.0793  
7.0746  
7.0727  
7.0640  
7.0580  
7.0538  
7.0045  
6.9848  
5.0149  
5.0088  
4.9999  
4.9799  
3.5511  
3.5261  
3.5064  
3.4814  
2.6672  
2.6570  
2.6524  
2.6124  
2.2981  
2.2894  
2.2567  
2.1892  
1.8348  
1.8298  
1.8252  
1.7288  
1.7247  
1.7205  
1.7153  
1.7111  
1.7036  
1.6935  
1.6846  
1.6813  
1.6748  
1.6716  
1.6654  
1.6511  
1.5901  
1.5860  
1.5812  
1.5762  
1.5592  
1.5560  
1.5522  
1.5151  
1.2815  
1.2738  
1.2692  
1.2588  
1.2518  
1.2463  
1.2421  
1.2273  
1.2212  
1.2169  
1.2121  
1.2083  
1.2025  
1.1988  
1.1959  
1.1871  
1.1831  
1.1791  
1.1751  
1.1651  
1.1575  
1.1484  
1.1421  
1.1384  
1.1350  
1.1111



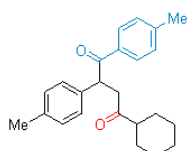
4ae



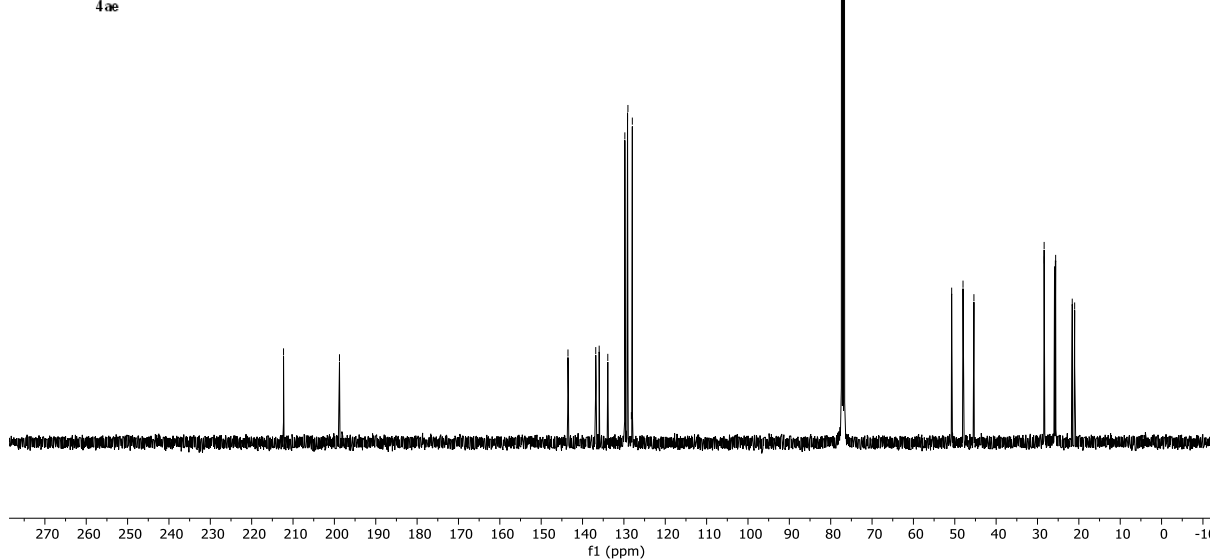
### 4ae <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV400/data/2408/nmr/240806.409/11/fid

212.2966  
196.7685  
143.5321  
136.8065  
135.9781  
133.8940  
129.7719  
128.1225  
128.0277  
128.1925  
127.9016  
50.7531  
48.0065  
45.3699  
28.3922  
28.3591  
28.3260  
25.8104  
21.5972  
21.0141



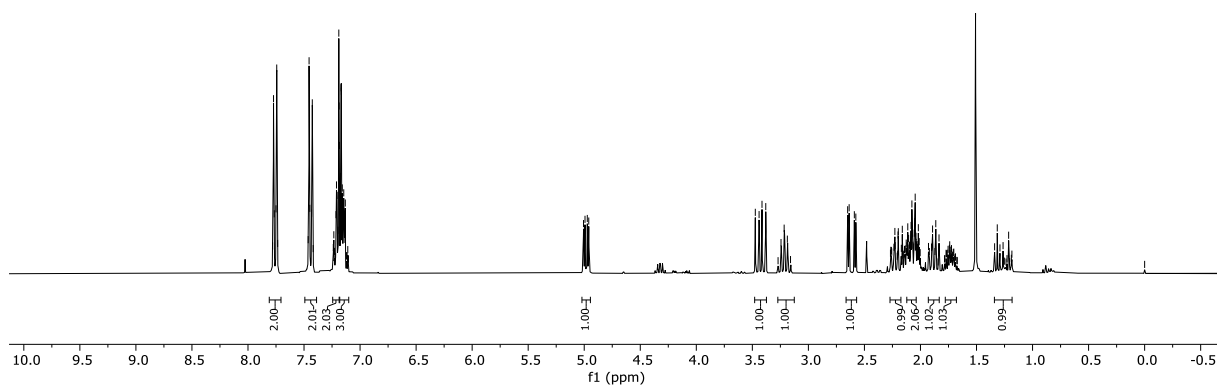
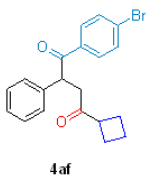
4ae



### 4af <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)

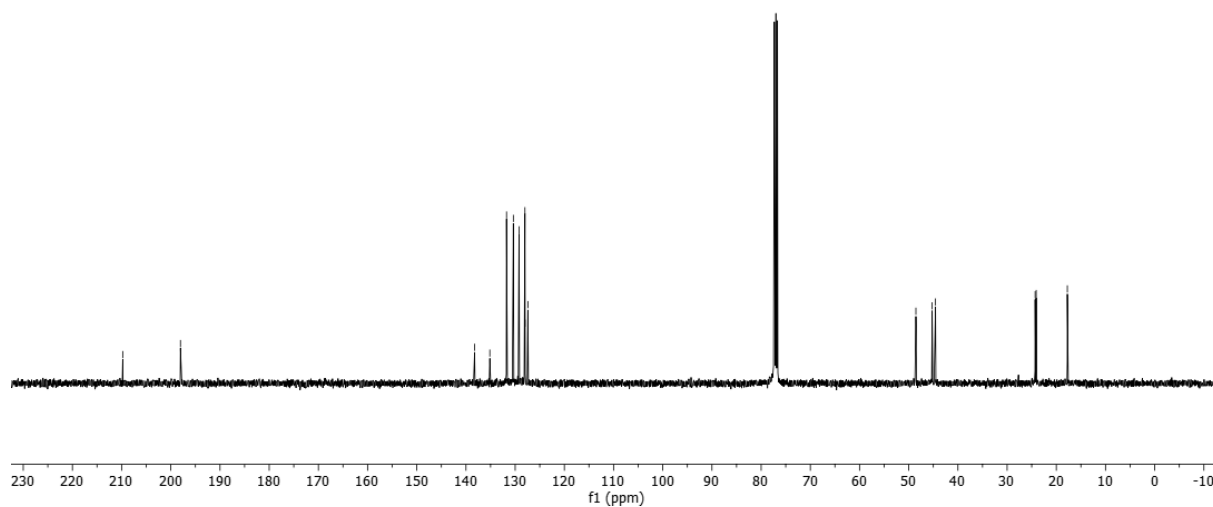
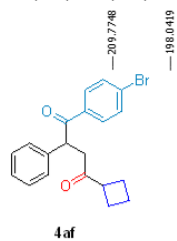
— K:\nmr\Fourier300\data\2407\nmr\240717.f304\10\fid

7.2802  
7.7722  
7.7657  
7.7499  
7.7435  
7.7355  
7.4628  
7.4483  
7.4324  
7.4262  
7.2388  
7.2341  
7.2148  
7.2115  
7.1951  
7.1921  
7.1896  
7.1872  
7.1847  
7.1820  
7.1753  
7.1728  
7.1669  
7.1669  
7.1587  
7.1558  
7.1509  
7.1456  
7.1404  
7.1352  
7.1317  
5.1063  
4.9930  
4.9727  
4.9594  
3.4736  
3.4399  
3.4159  
3.3803  
3.2483  
3.2163  
3.1877  
3.1877  
2.6496  
2.6364  
2.5900  
2.5774  
2.5264  
2.2283  
2.2253  
2.2001  
2.1977  
2.1622  
2.1558  
2.1271  
2.1271  
2.1131  
2.1058  
2.1031  
2.0979  
2.0885  
2.0839  
2.0764  
2.0744  
2.0573  
2.0602  
2.0664  
2.0554  
2.0501  
2.0471  
2.0401  
2.0378  
2.0378  
2.0263  
2.0190  
2.0115  
1.9285  
1.9014  
1.8988  
1.8934  
1.8834  
1.8545  
1.7391  
1.3386  
1.3147  
1.2909  
1.2631  
1.2134

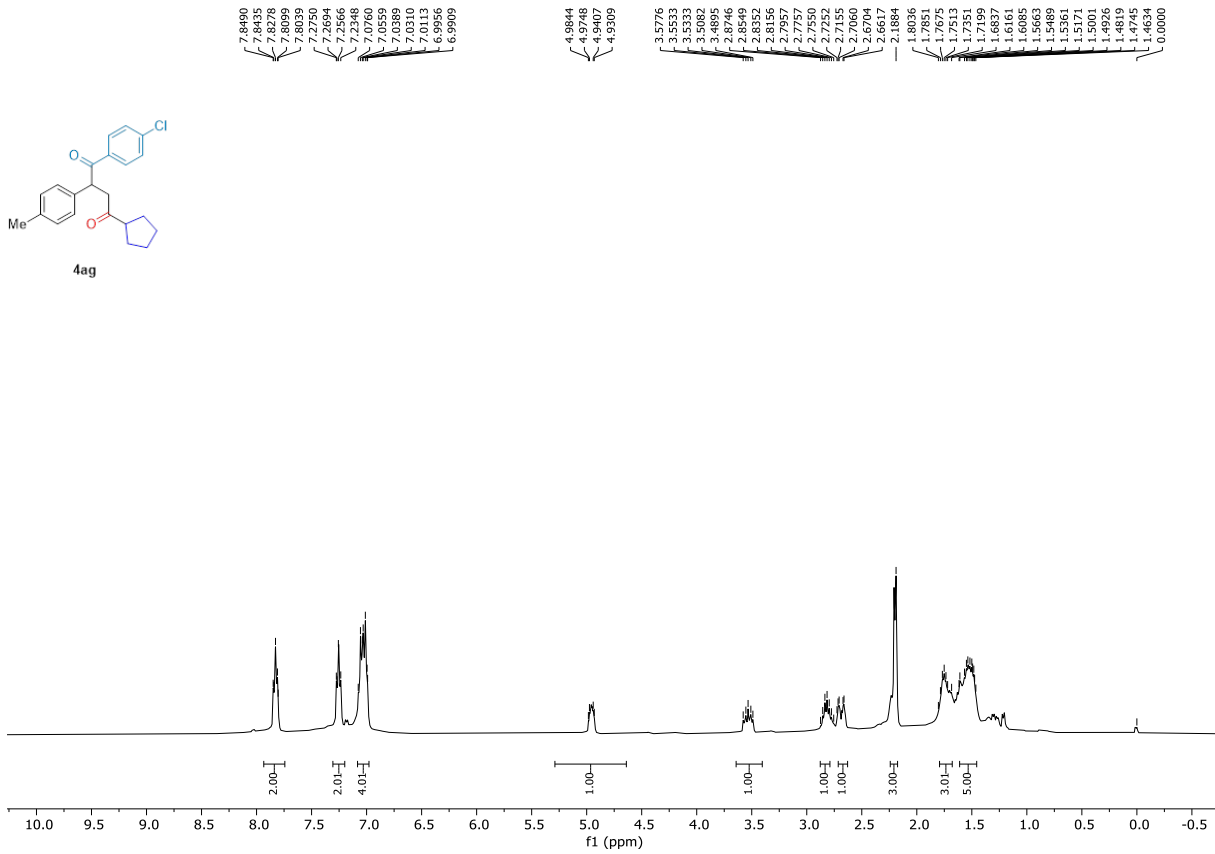
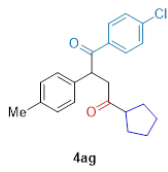


### 4af <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)

— K:\nmr\AV400\data\2407\nmr\240715.404\11\fid

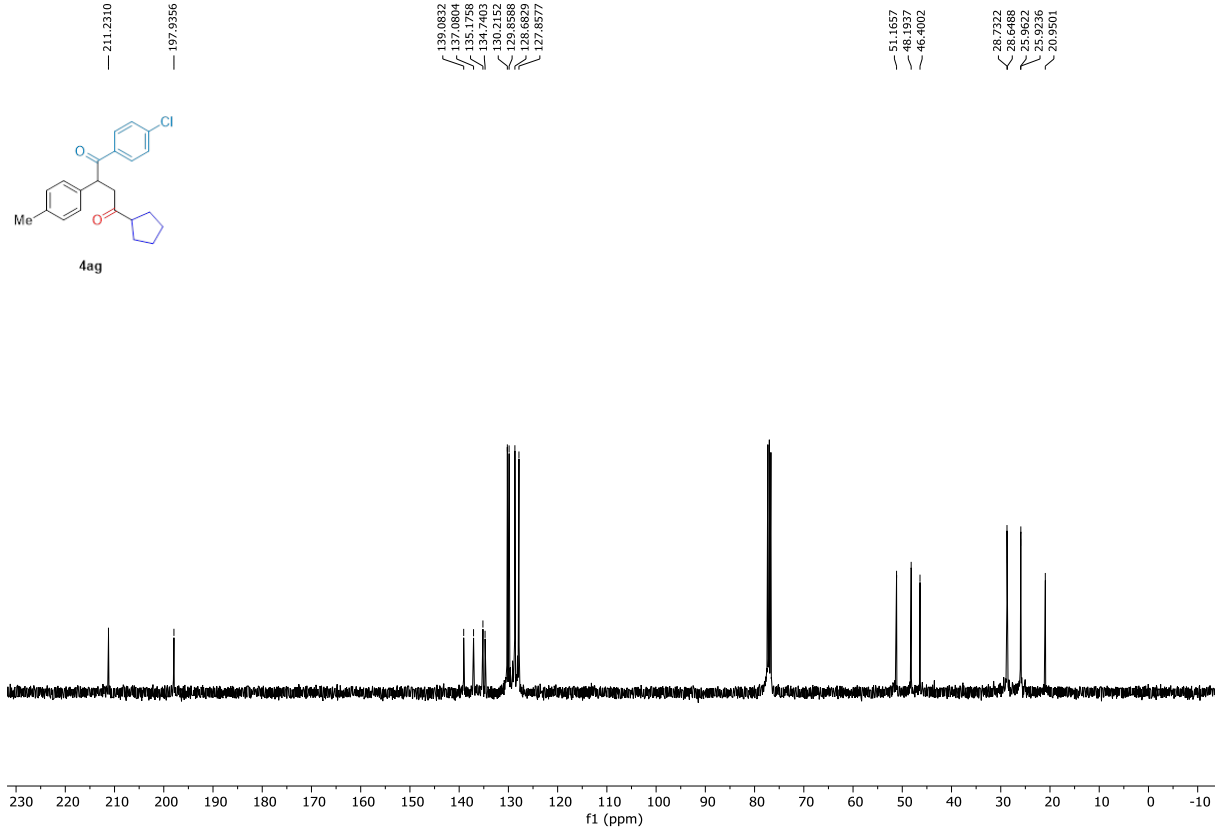


### 4ag <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



**4ag** <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)

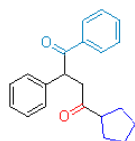
— K:/nmr/AV400/data/2407/nmr/240715.406/11/fid



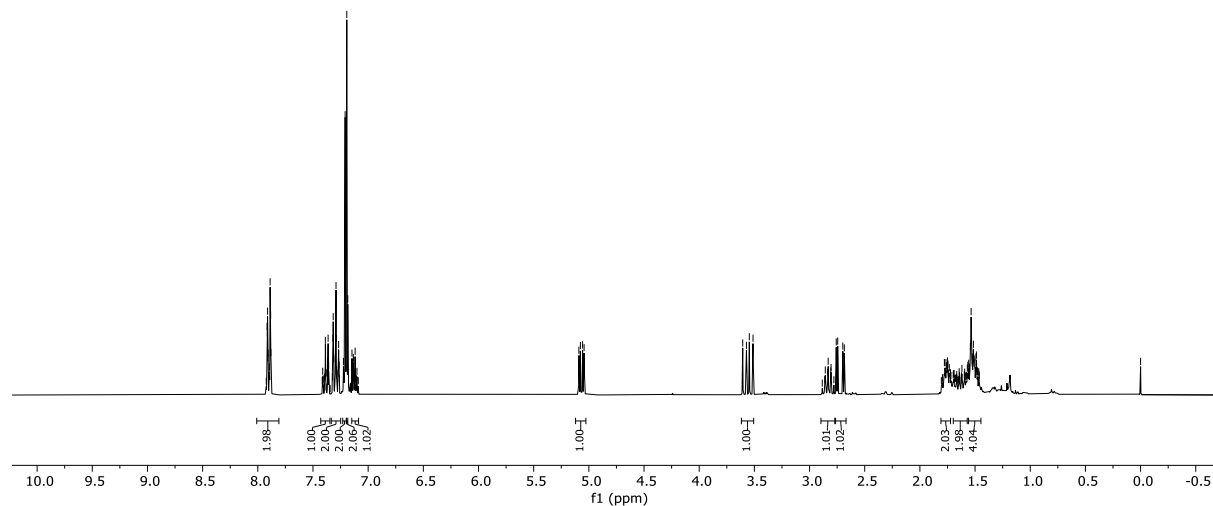
### 4ah <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV300/data/2406/nmr/240604.302/10/fid

7.9162  
7.9137  
7.9117  
7.9090  
7.9066  
7.8954  
7.8884  
7.8852  
7.8817  
7.4116  
7.3879  
7.3859  
7.3797  
7.3676  
7.3636  
7.3582  
7.3191  
7.3166  
7.3119  
7.2962  
7.2912  
7.2890  
7.2863  
7.2791  
7.2677  
7.2636  
7.2240  
7.2086  
7.1937  
7.1848  
7.1806  
7.1395  
7.1304  
7.1181  
7.1016  
5.0903  
5.0769  
5.0568  
5.0525  
3.6956  
3.5720  
3.5459  
3.5123  
2.8574  
2.8353  
2.8310  
2.8248  
2.8049  
2.7581  
2.7449  
2.6984  
2.6850  
2.6850  
1.7780  
1.7724  
1.7584  
1.7574  
1.7538  
1.7504  
1.7481  
1.7464  
1.7420  
1.7333  
1.7242  
1.6945  
1.6905  
1.6442  
1.6193  
1.5943  
1.5701  
1.5632  
1.5574  
1.5577  
1.5497  
1.5426  
1.5358  
1.5257  
1.5152  
1.5103  
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1.4830  
1.4798  
1.4770  
1.4667  
-0.0001

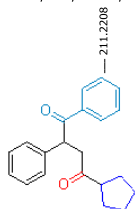


4ah

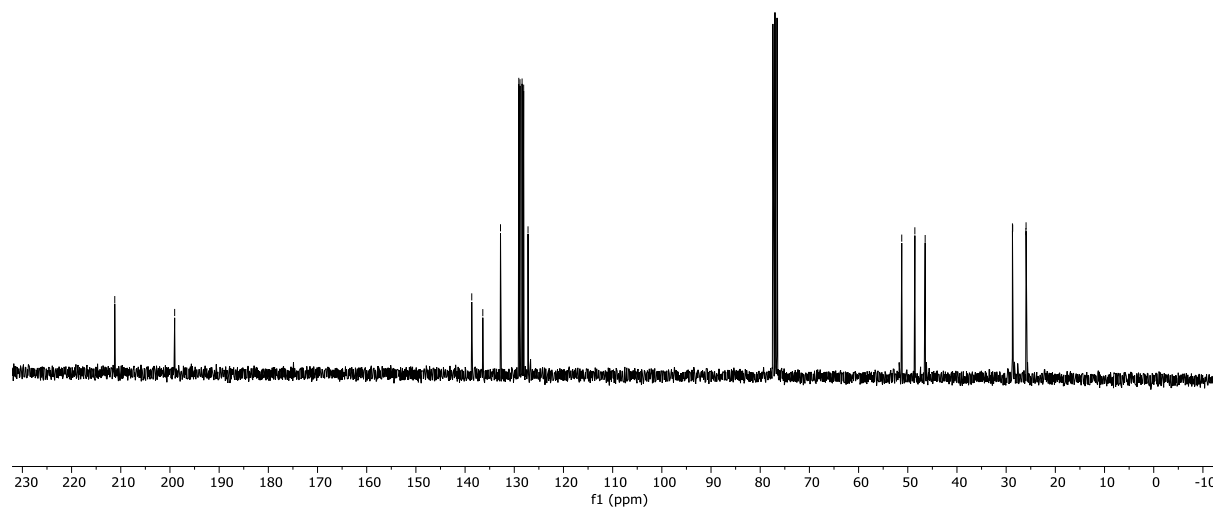


### 4ah <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV300/data/2406/nmr/240604.302/11/fid

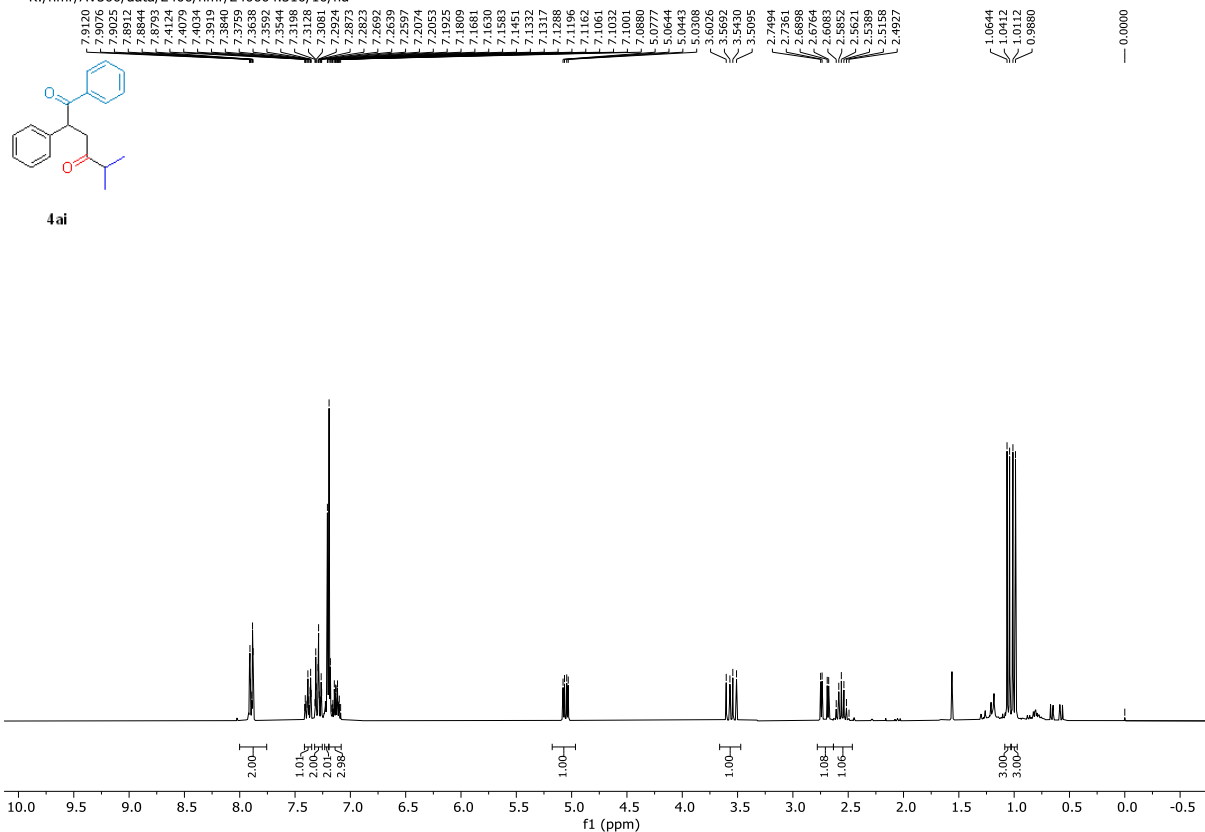


4ah



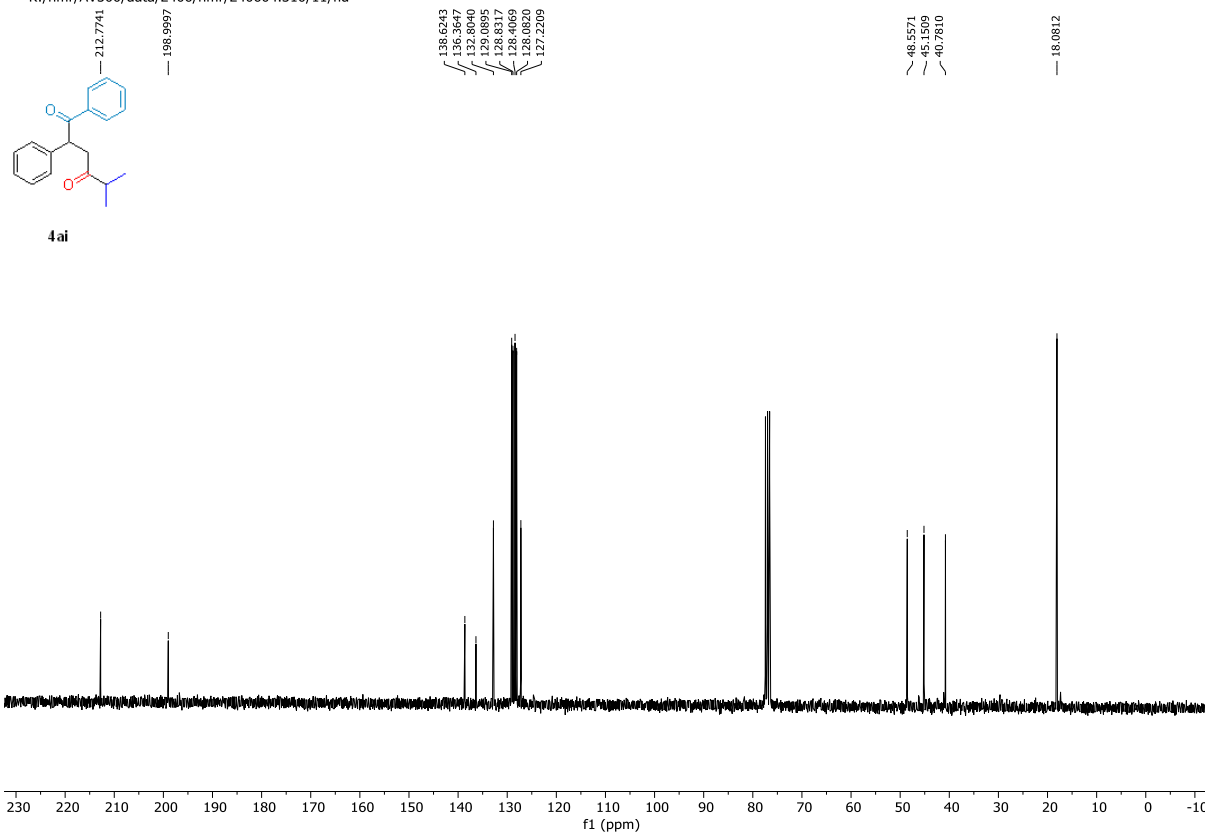
### 4ai <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV300/data/2406/nmr/240604.310/10/fid

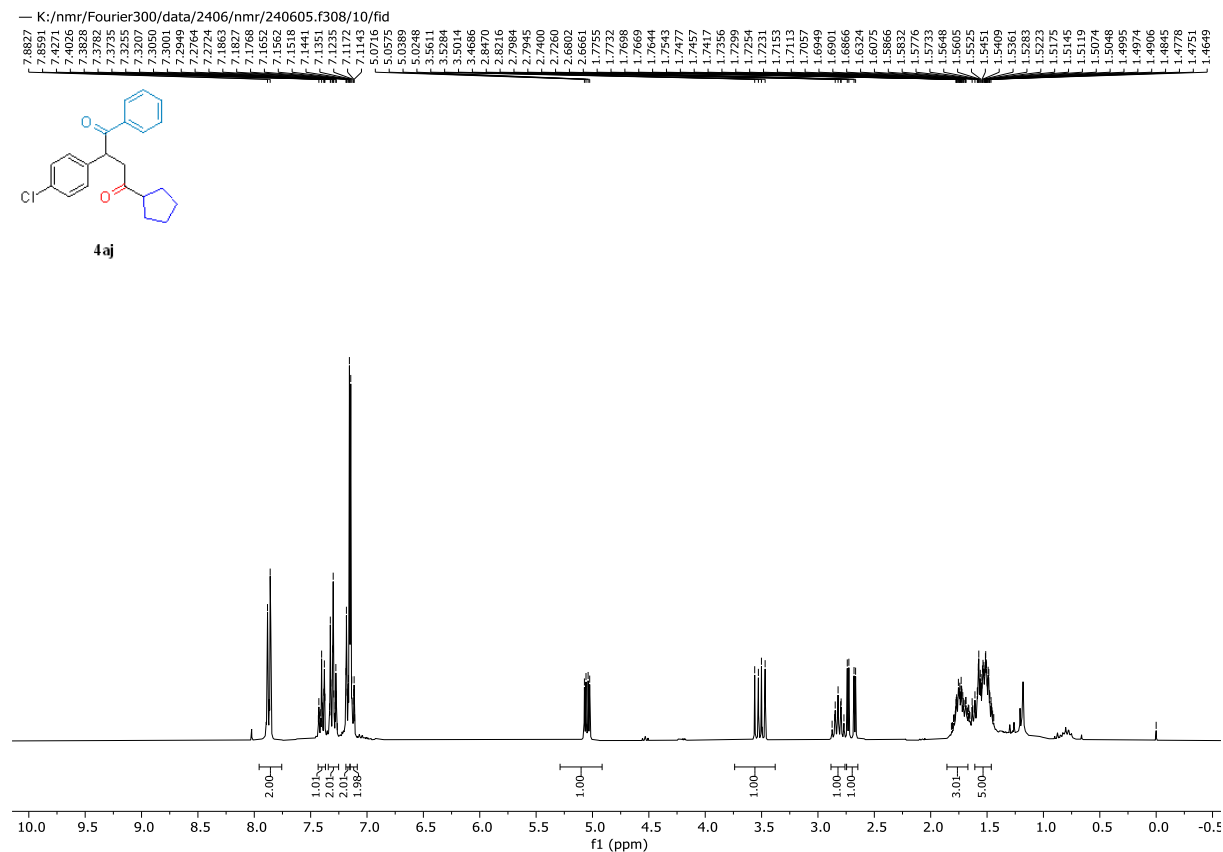


### 4ai <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)

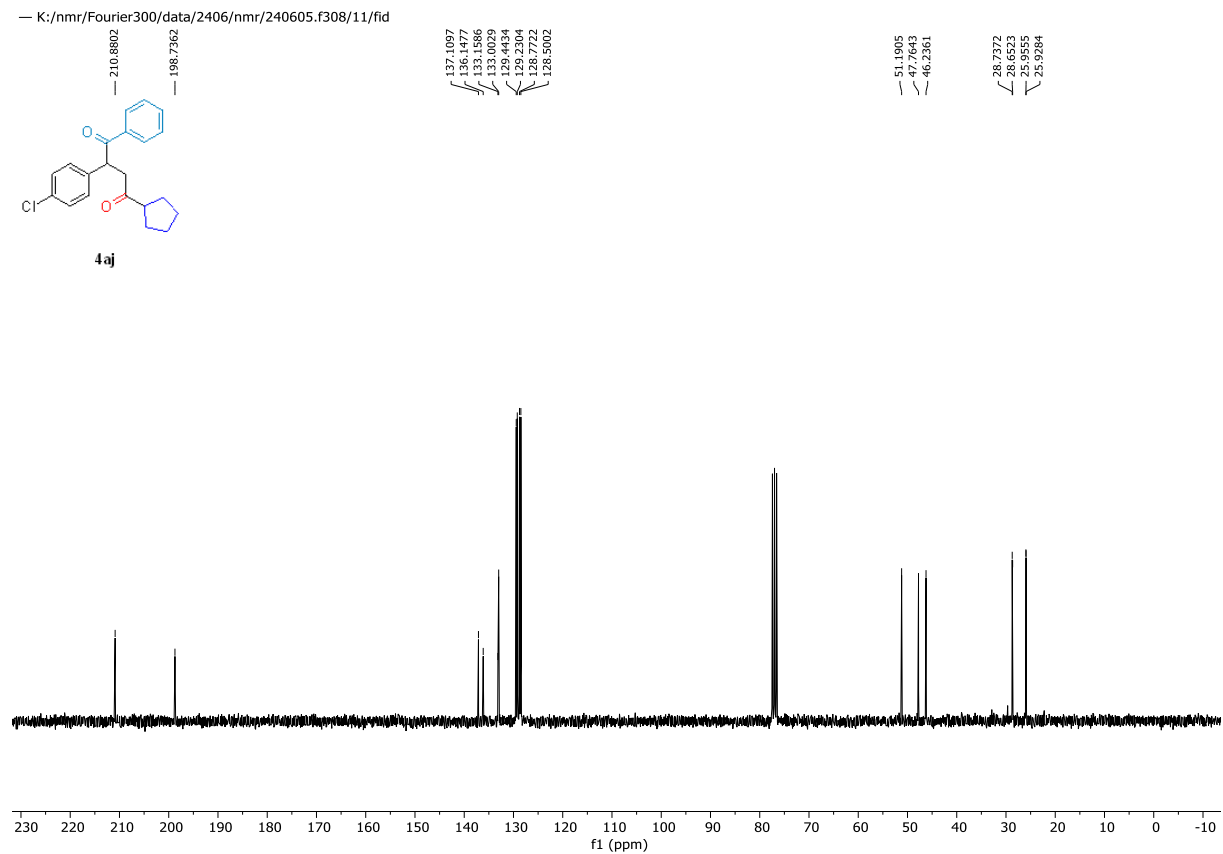
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### 4aj <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



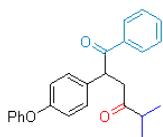
### 4aj <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



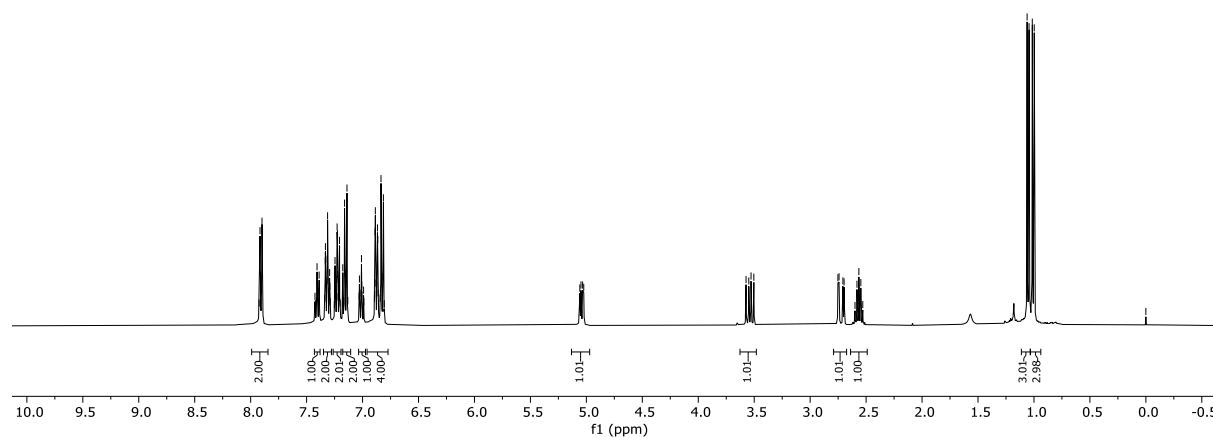
### 4ak <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV400/data/2407/nmr/240701.447/10/fid

7.9193  
7.9163  
7.9126  
7.9038  
7.8987  
7.8949  
7.4553  
7.4070  
7.4017  
7.3917  
7.3884  
7.3851  
7.3368  
7.3335  
7.3302  
7.3282  
7.3160  
7.3127  
7.3093  
7.2995  
7.2945  
7.2900  
7.2862  
7.2440  
7.2407  
7.2337  
7.2300  
7.2275  
7.2245  
7.2224  
7.2190  
7.2110  
7.2083  
7.2061  
7.1768  
7.1686  
7.1650  
7.1550  
7.1448  
7.1394  
7.1319  
7.0308  
7.0281  
7.0253  
7.0224  
7.0197  
7.0076  
7.0057  
6.9911  
6.8936  
6.8920  
6.8897  
6.8866  
6.8836  
6.8804  
6.8719  
6.8694  
6.8667  
6.8641  
6.8616  
6.8584  
6.8354  
6.8300  
6.8188  
6.8136  
5.0584  
5.0483  
5.0336  
5.0200  
3.5730  
3.5482  
3.5283  
3.5034  
2.7419  
2.7073  
2.5819  
2.5646  
2.5472  
1.0612  
1.0439  
1.0149  
0.9974



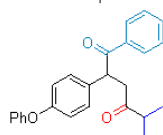
4ak



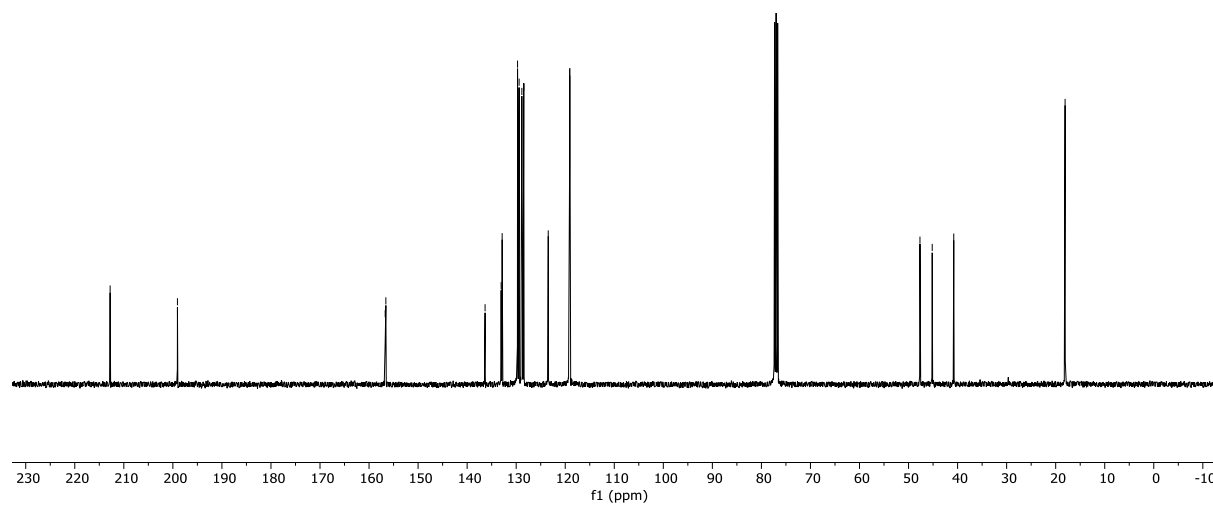
### 4ak <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV400/data/2407/nmr/240701.447/11/fid

212.7935  
195.0678  
156.7098  
156.5719  
136.3411  
132.0653  
132.9609  
132.9609  
129.4030  
128.8448  
128.4540  
123.4636  
119.0987  
119.0625  
47.6727  
45.7771  
46.7814  
18.0814

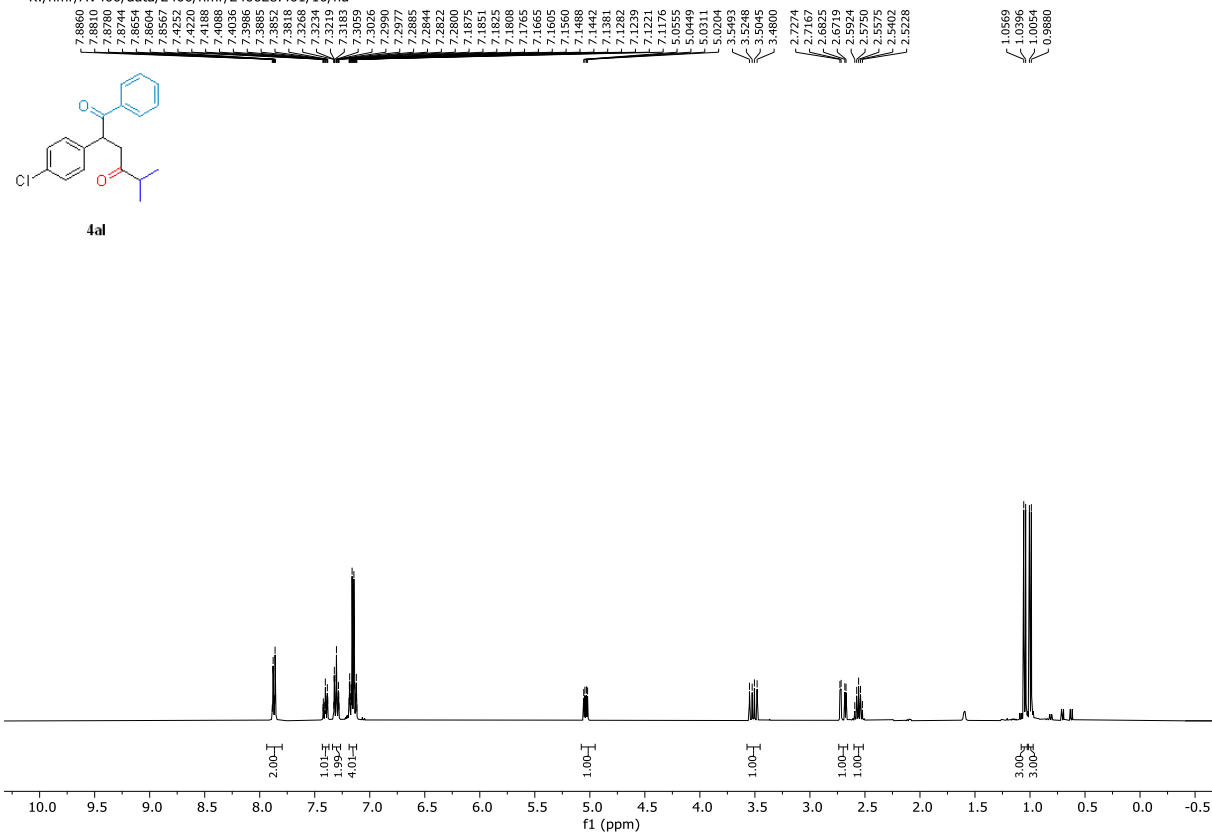


4ak



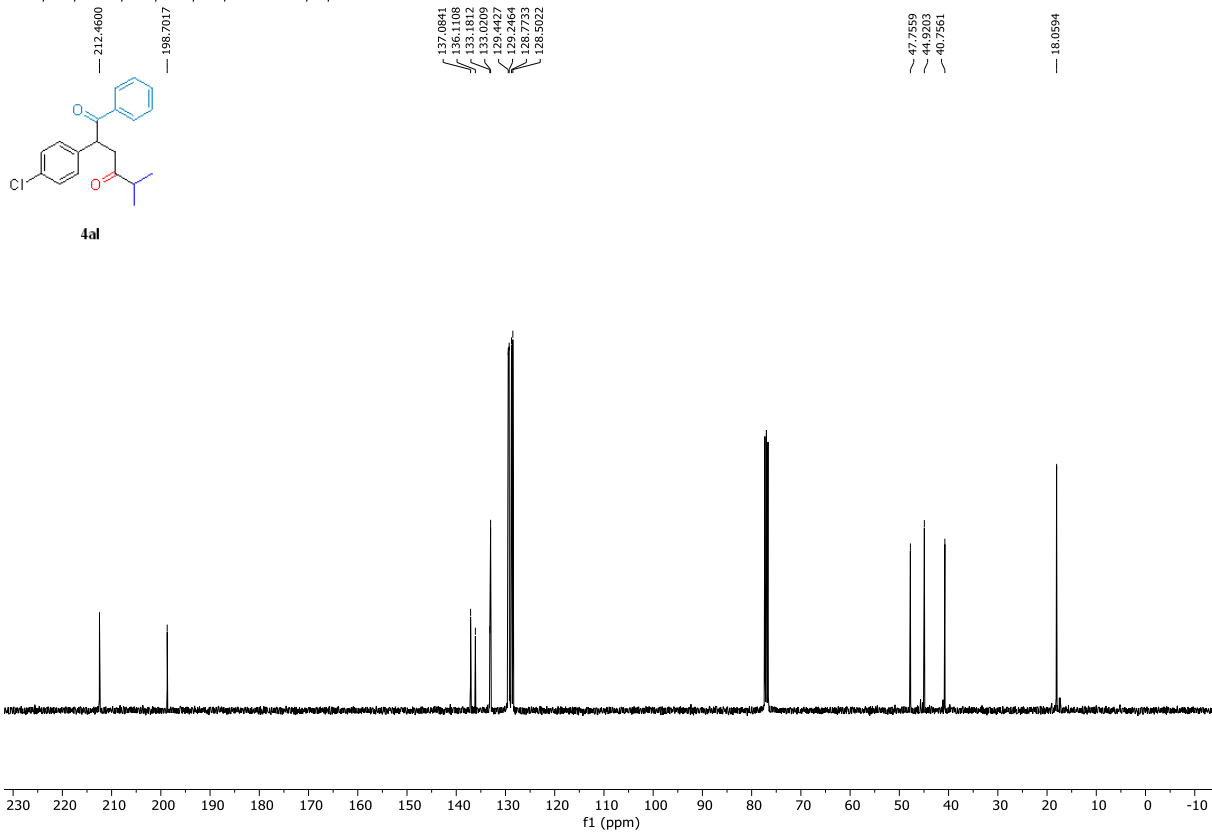
### 4al <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV400/data/2406/nmr/240628.401/10/fid



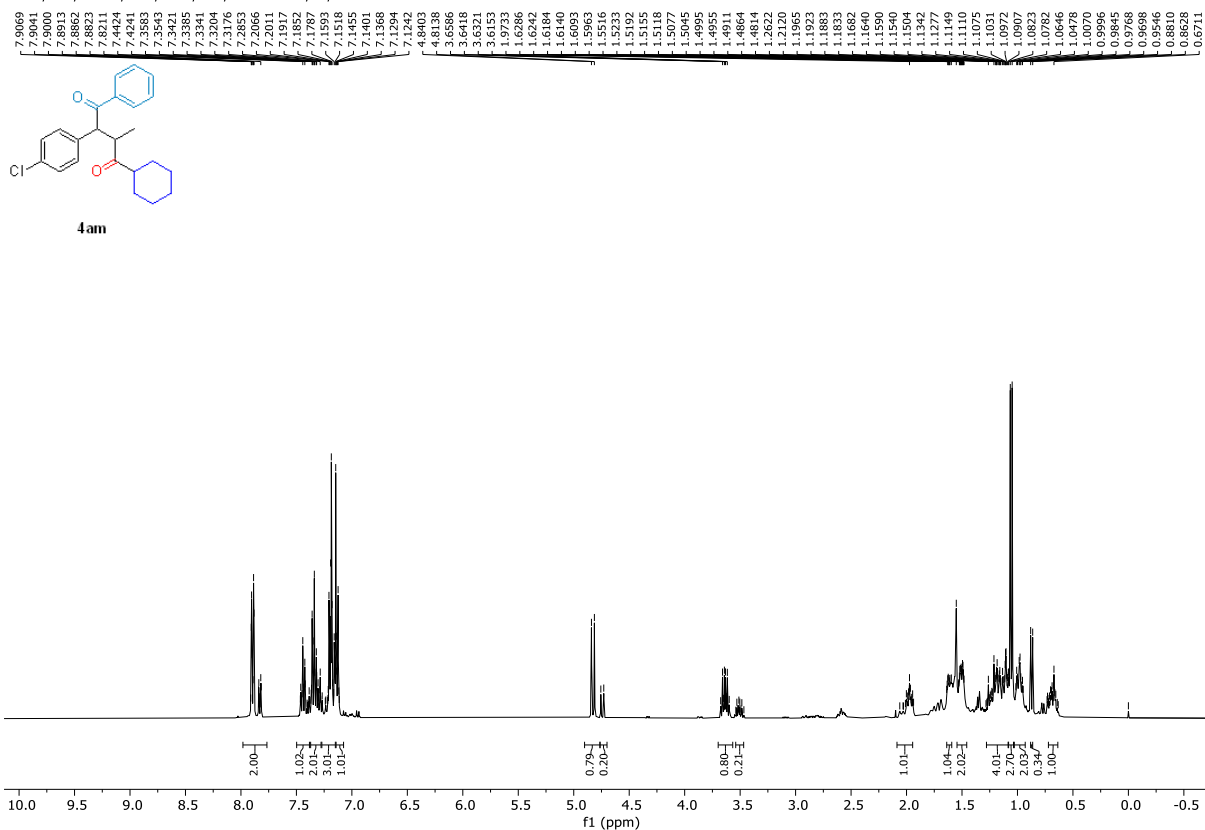
### 4al <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV400/data/2406/nmr/240628.401/11/fid



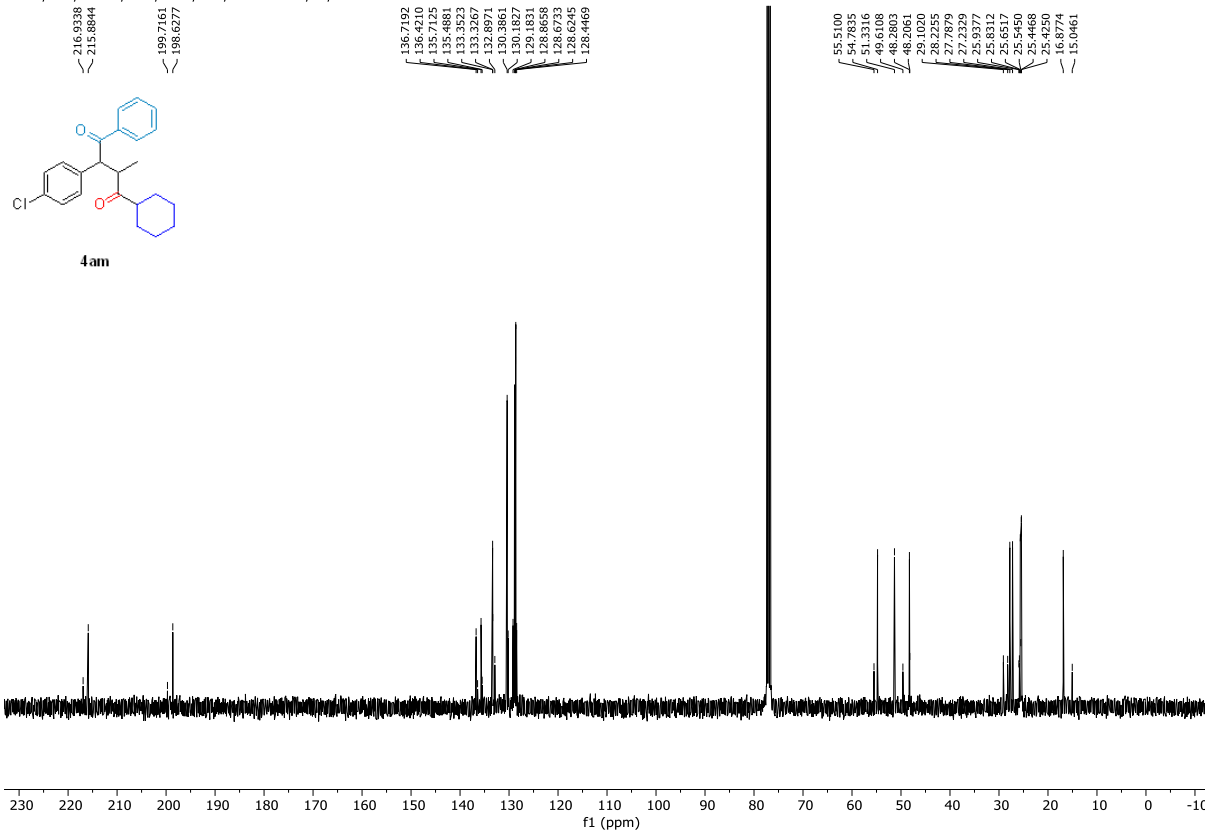
### 4am <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

— K:/nmr/AV400/data/2406/nmr/240628.402/10/fid

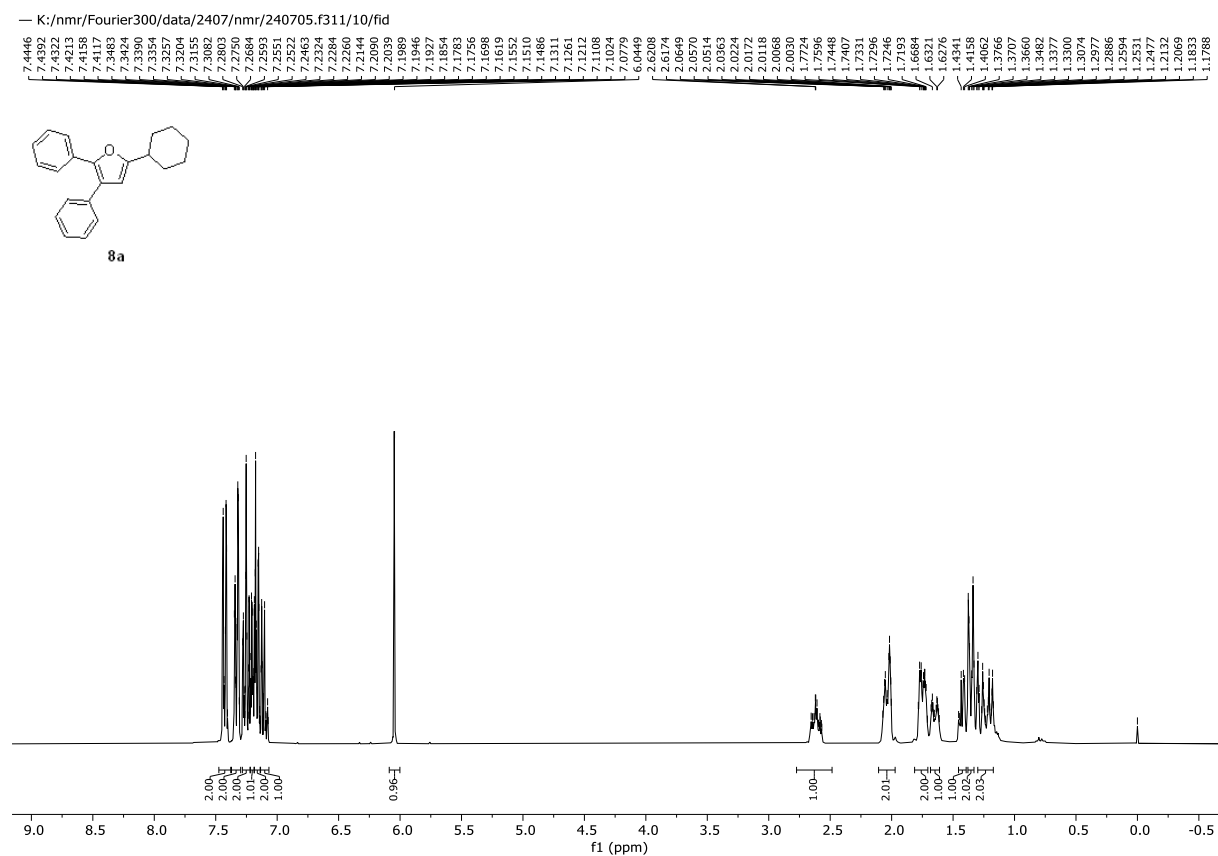


### 4am <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)

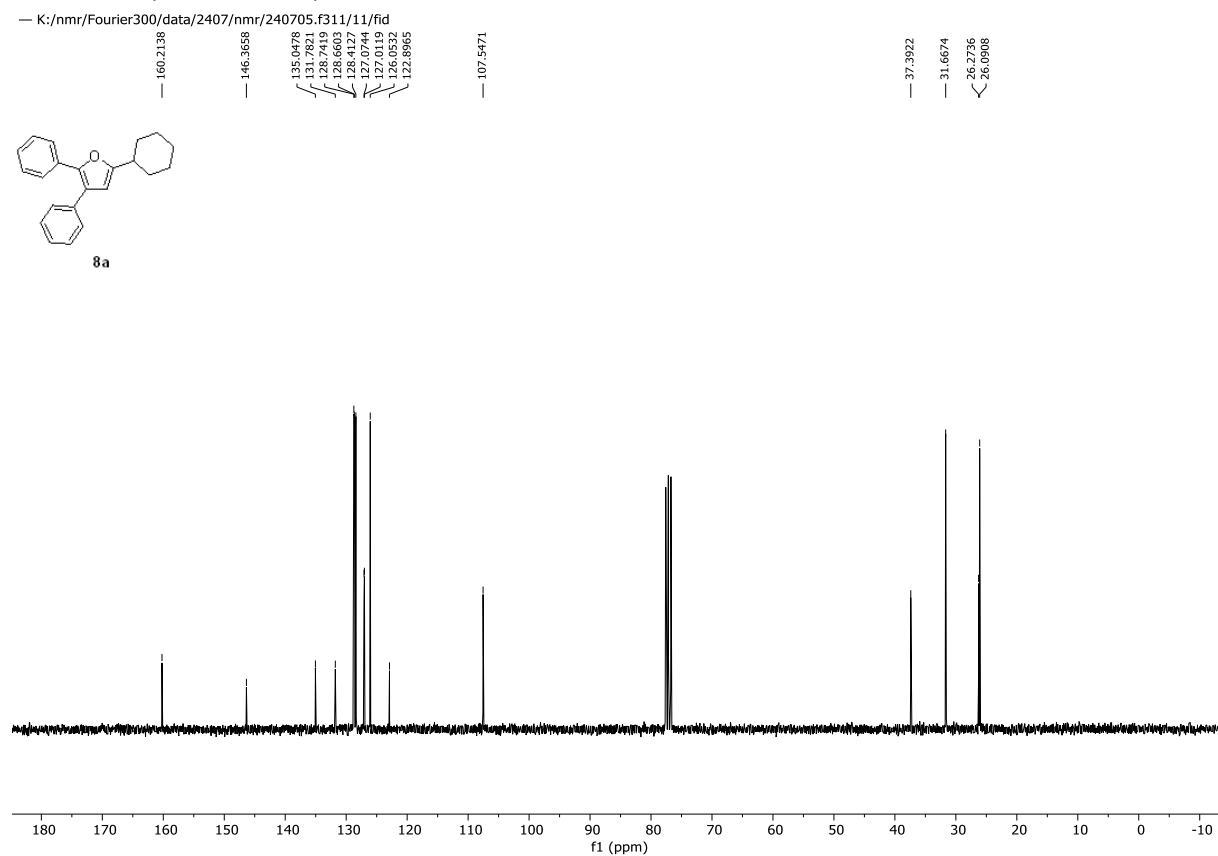
— K:/nmr/AV400/data/2406/nmr/240628.402/11/fid



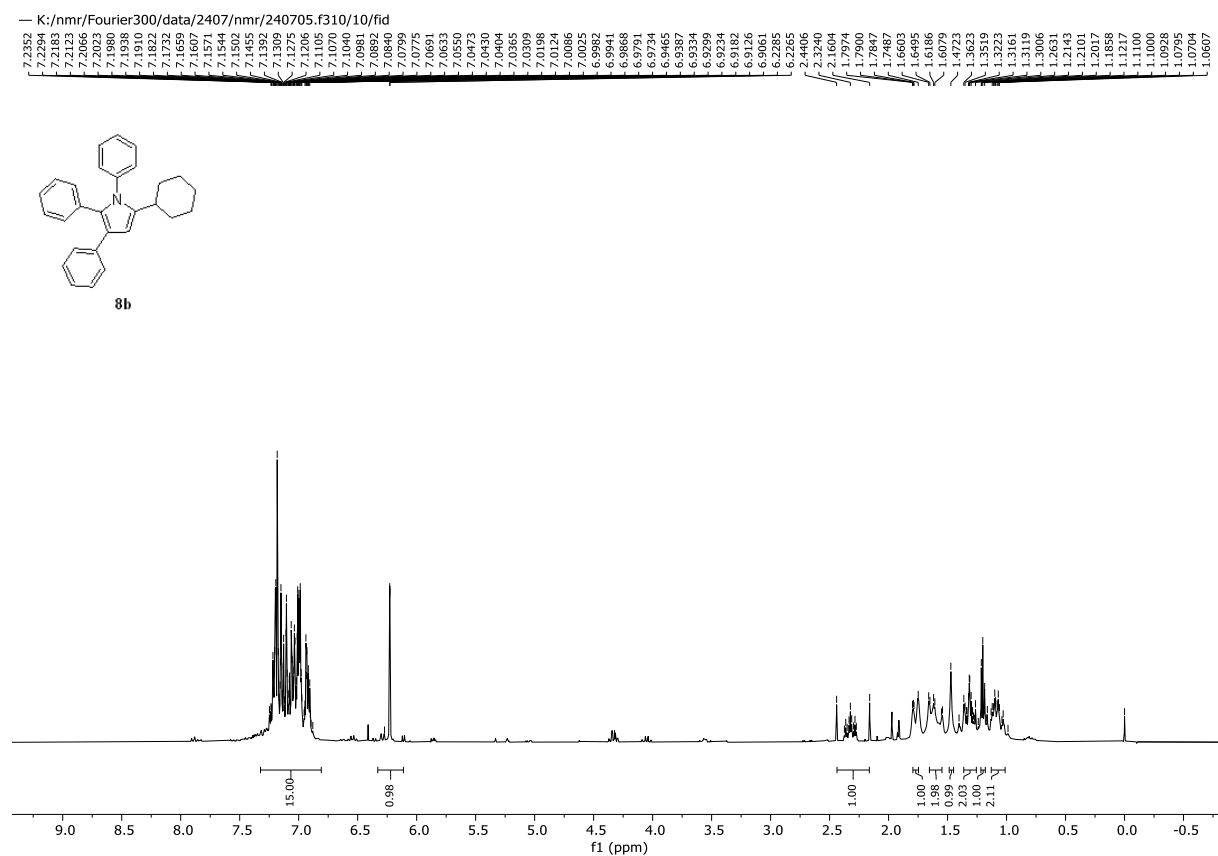
### 8a <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



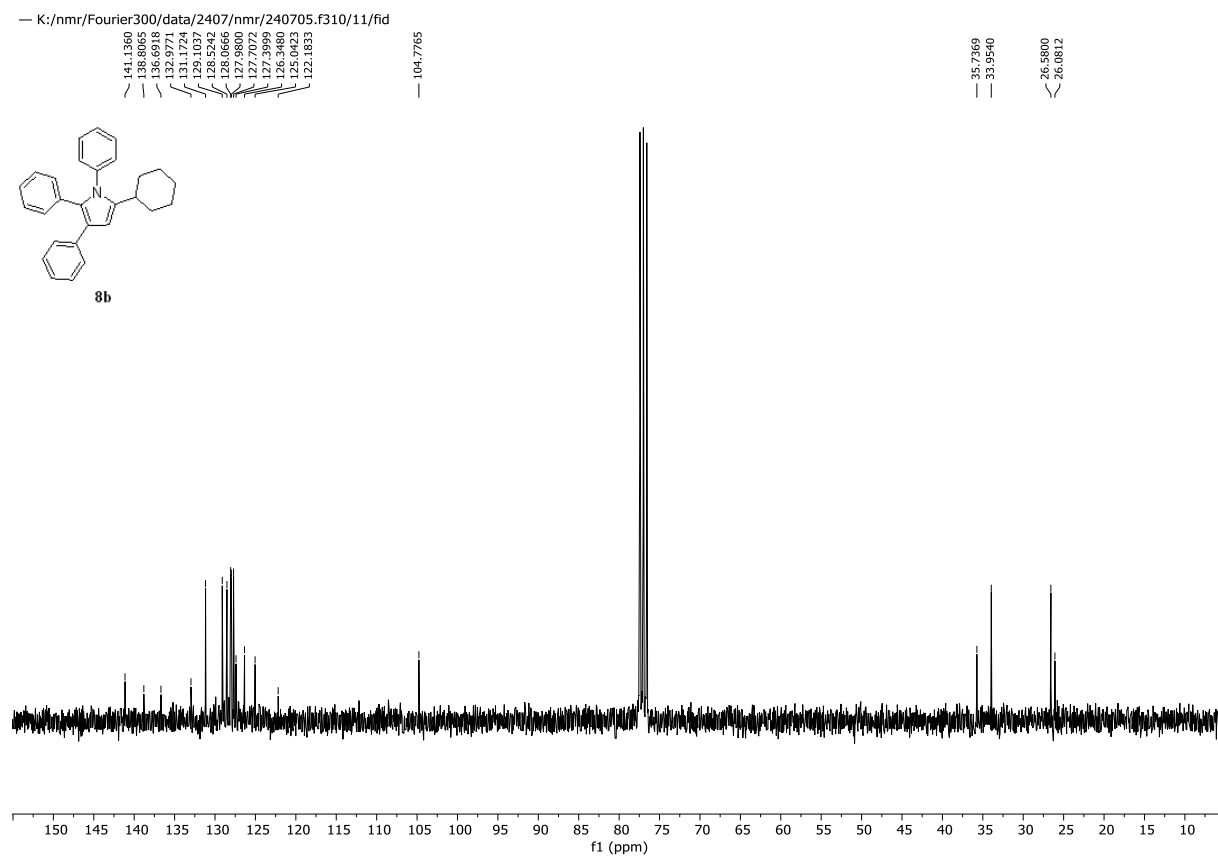
### 8a <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



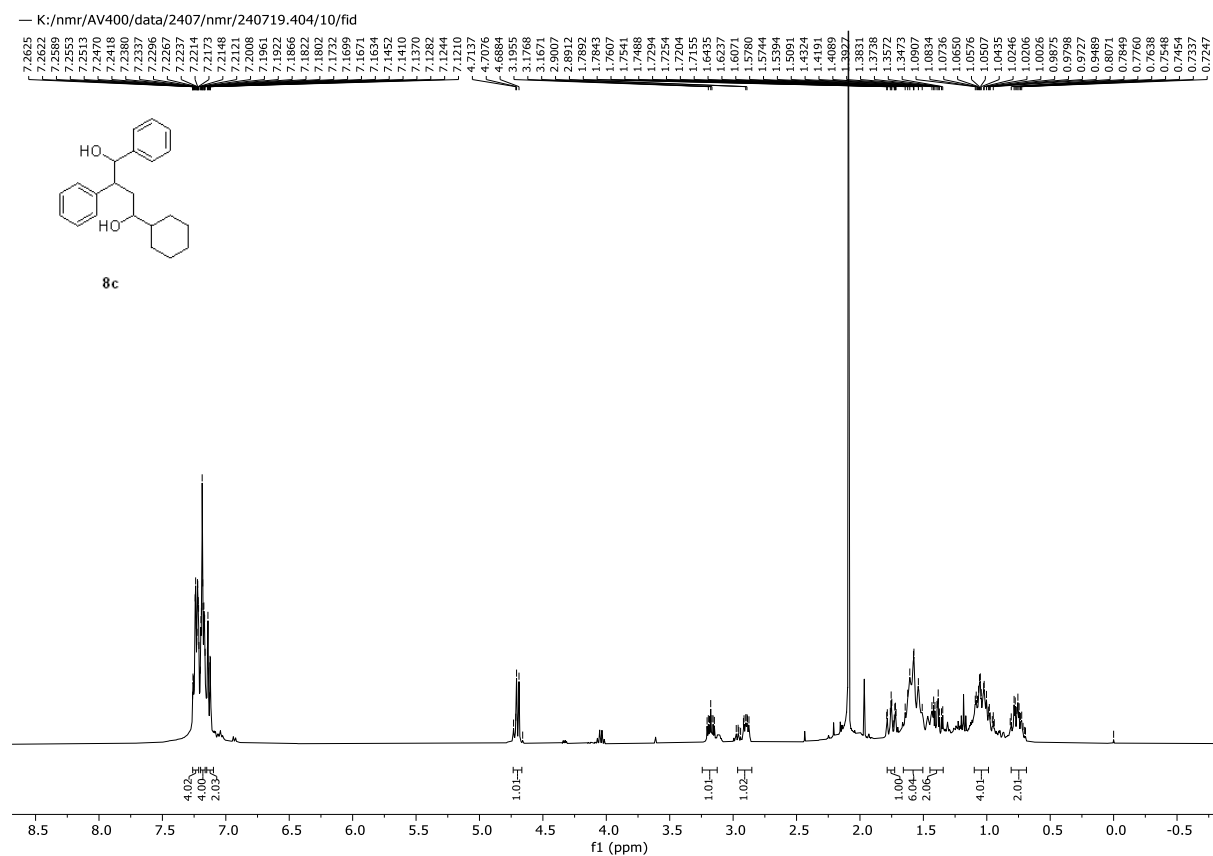
### 8b <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



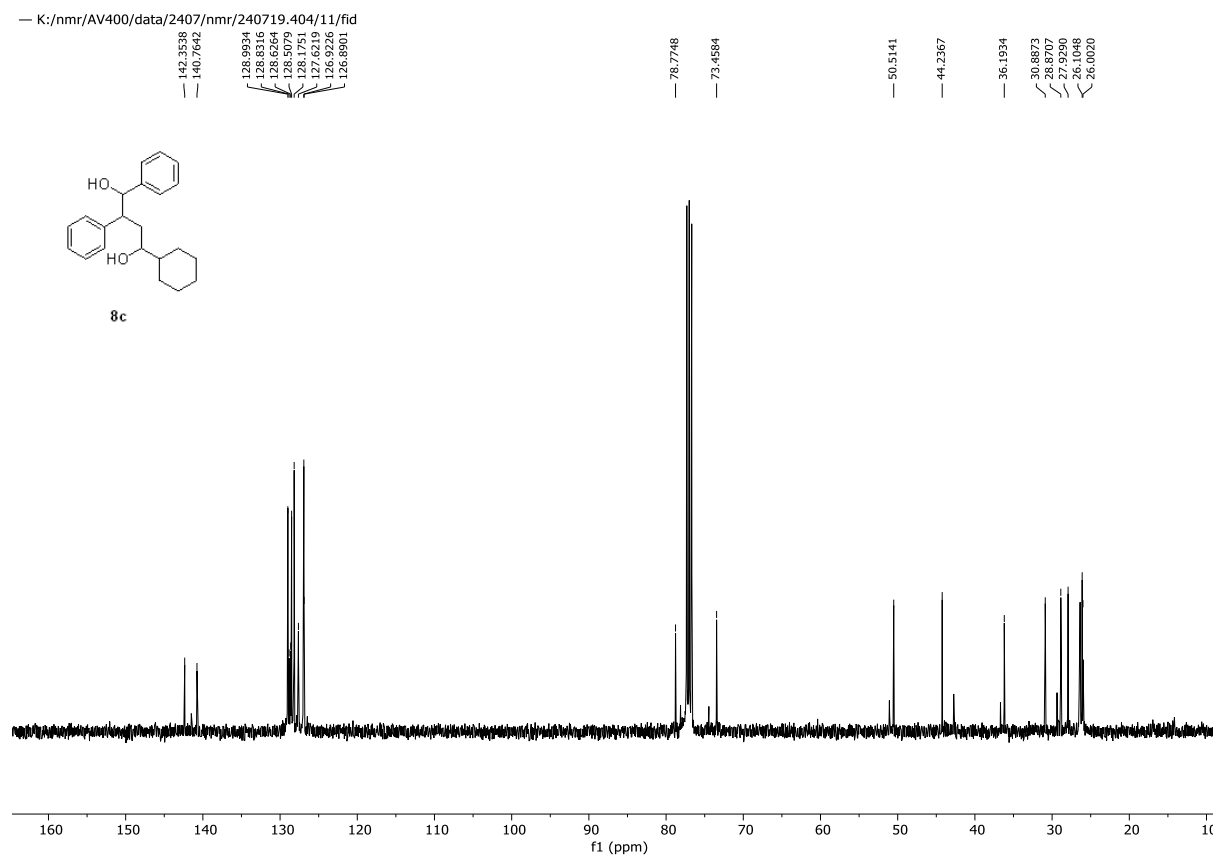
### 8b <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



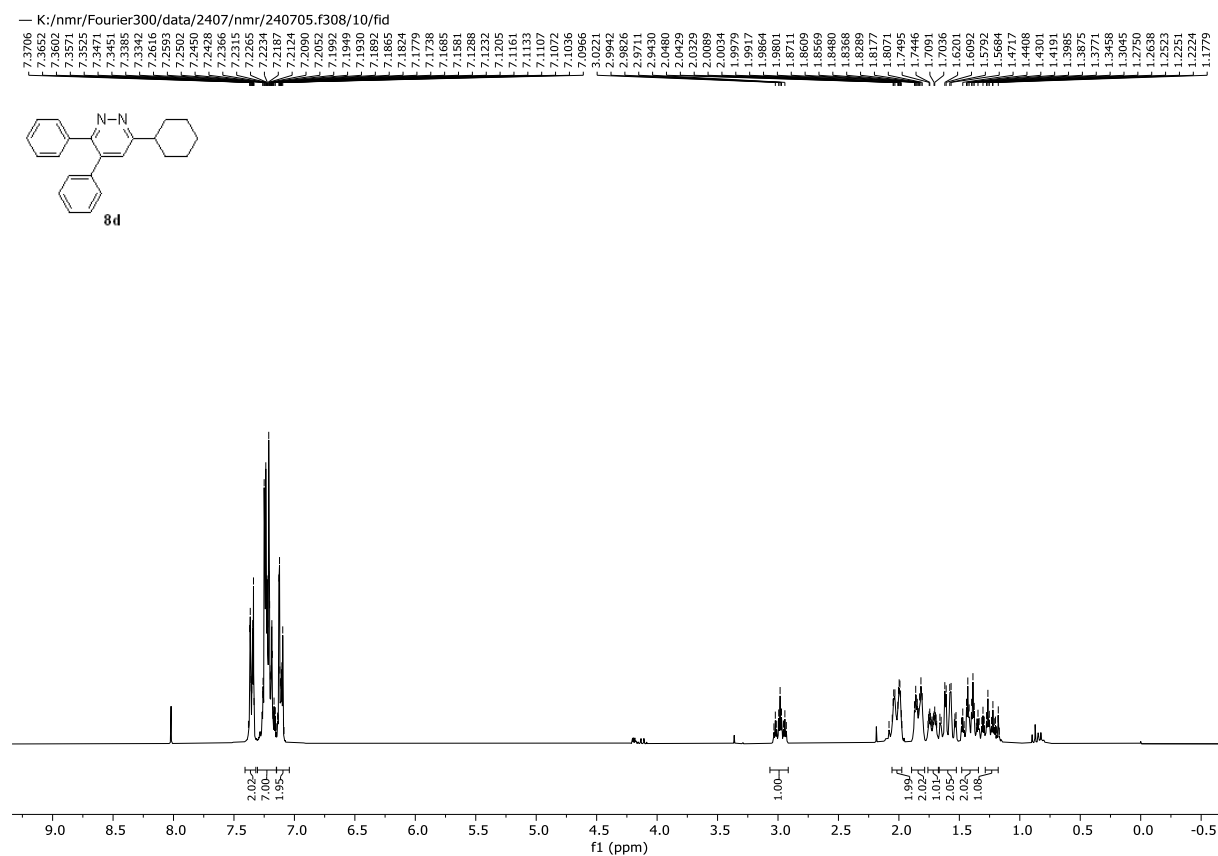
### 8c <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



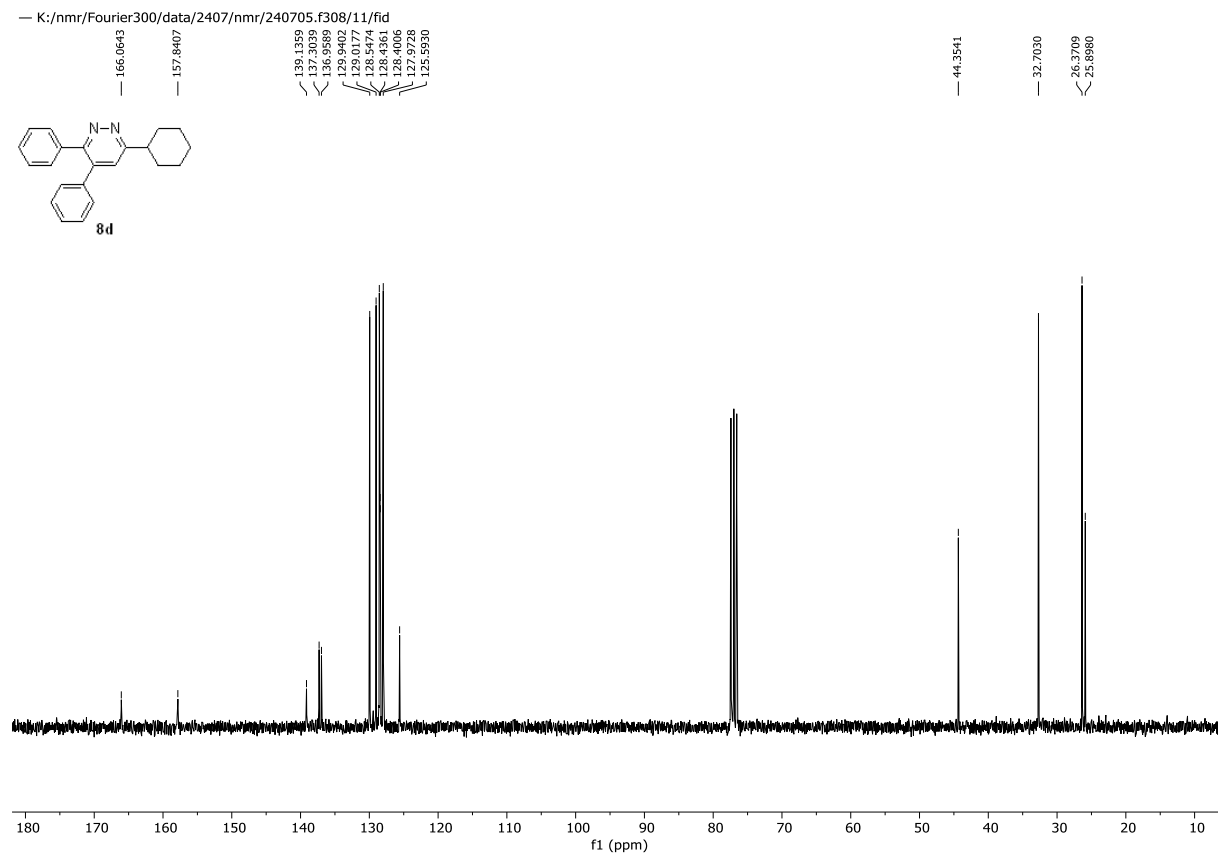
### 8c <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)



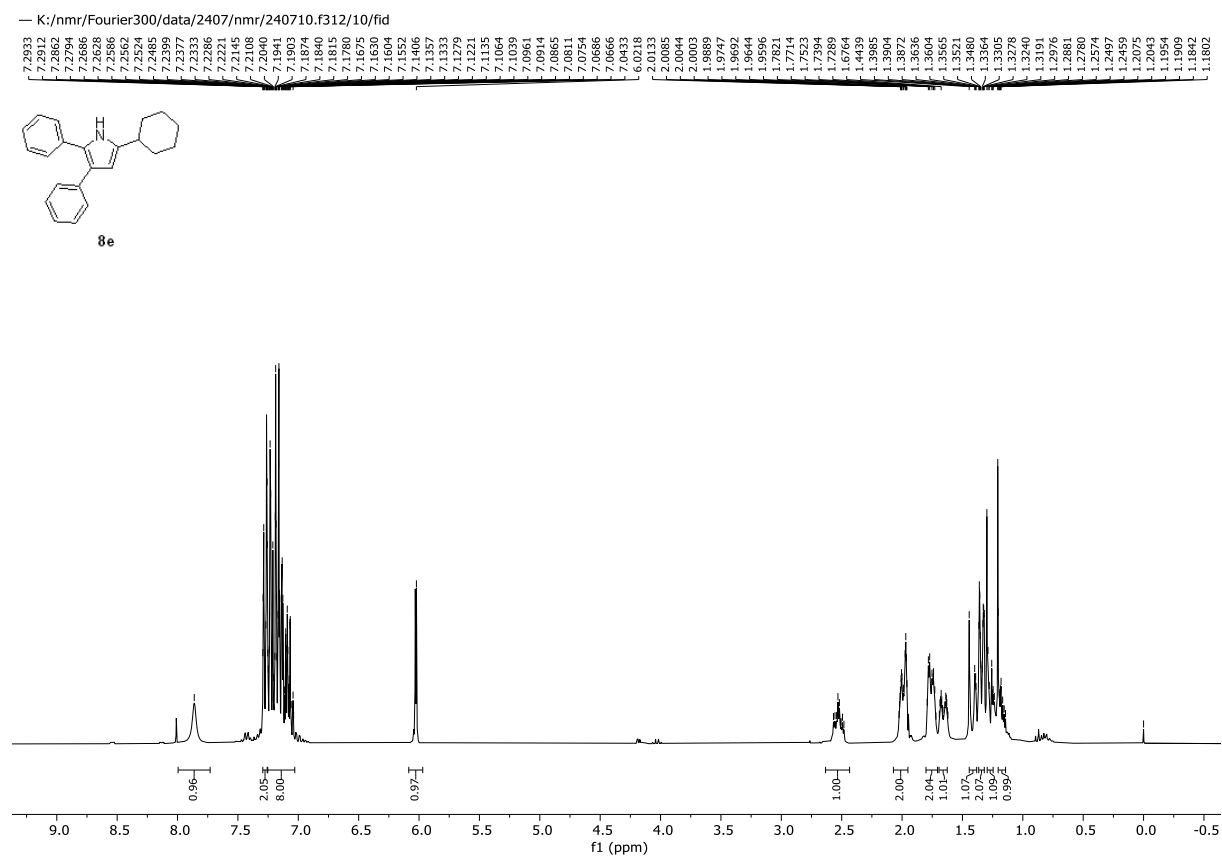
### 8d <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



### 8d <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)



### 8e <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)



### 8e <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)

