Supporting Information

N-Heterocyclic Carbene/Photoredox-Catalyzed Regioselective Carbonylation of Alkenes

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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}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 ($CDCl_3$: δ = 7.26 ppm). ^{13}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 ($CDCl_3$: δ = 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 μm 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[3], was utilized with different set filters: UV-A (λ = 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.

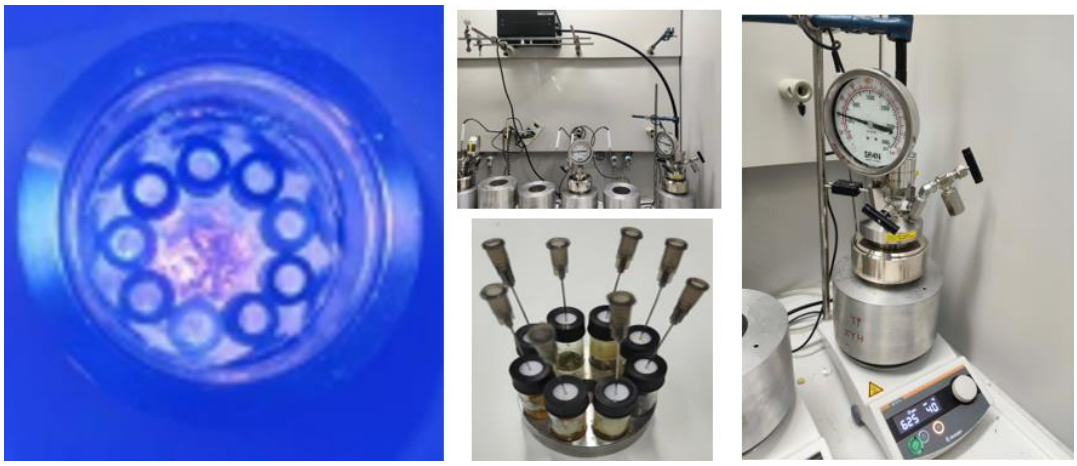


Figure 1. Apparatus for the Photoinduced Diacylation of Styrenes with Carboxylic Acids Using Carbon Monoxide as a Carbonyl Source to Form 1,4-Diketones

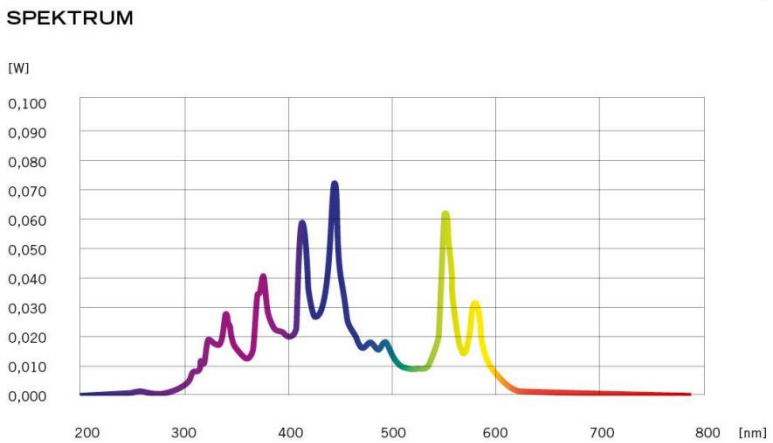


Figure 2. Emission Spectrums of the Portable Lumatec SUPERLITE S 04

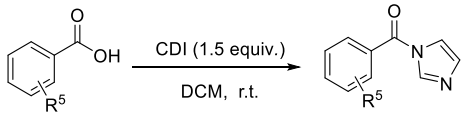
OPTISCHE LEISTUNG

Spektrum		Leistung	Intensität
UVA	320–400 nm	2.100 mW	10.500 mW/cm²
UVA+Blau	320–500 nm	6.900 mW	34.500 mW/cm²
Blau	400–500 nm	4.800 mW	24.000 mW/cm²
Weiß	400–700 nm	9.700 mW	48.500 mW/cm²
Violett	415 nm	2.000 mW	10.000 mW/cm²
Blau 440	440 nm	2.300 mW	11.500 mW/cm²
Blau 460	460 nm	2.000 mW	10.000 mW/cm²
Türkis	490 nm	1.200 mW	6.000 mW/cm²
Grün	550 nm	1.400 mW	7.000 mW/cm²
Gelb	570 nm	1.800 mW	9.000 mW/cm²

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

2. Preparation of substrates

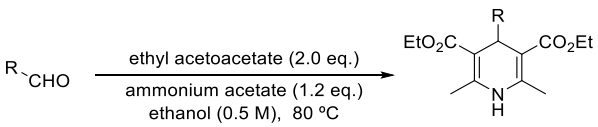
2.1 General procedure for the synthesis of acyl imidazoles 2



2

Acyl imidazoles **2** were prepared based on the literature[1]: The appropriate acid (6 mmol, 1.0 equiv) was dissolved in dry dichloromethane (20 ml, 0.3 M), and CDI (Carbonyldiimidazole, 9 mmol, 1.5 equiv) was added slowly. The resulting mixture was stirred at room temperature, overnight. Upon completion, the solution was transferred to a separatory funnel and washed with deionized water (2 × 10 mL). The organic layer was then dried over $MgSO_4$. Concentration under reduced pressure yielded the acyl imidazole, which was used in the subsequent reaction without further purification.

2.2 Preparation of Hantzsch Ester 3



3

Hantzsch ester **3** were prepared based on the literature[2]: 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, 0.5 M). To the above solution, the aldehyde (5.0 mmol) was added slowly. After addition, the system was heated at 80 ºC 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.

3. Complementary reaction optimization Data

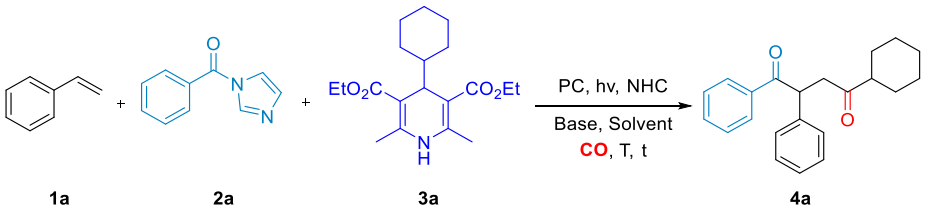
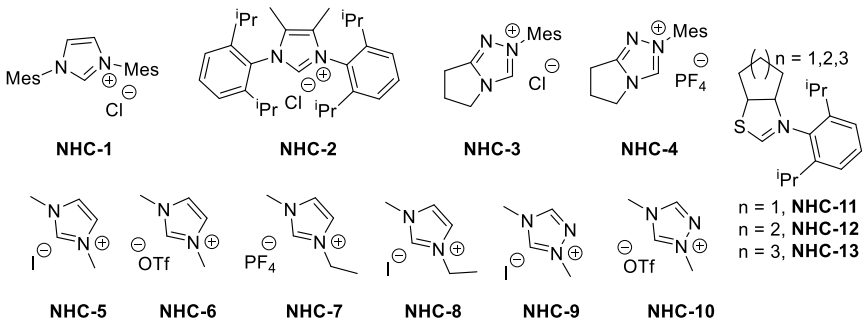


Table 1. Optimization of Carbene (NHC)

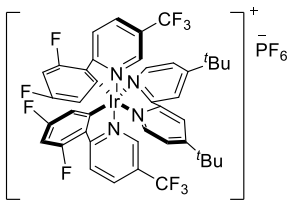
Entry	NHC	Yield (%)
1 [a]	**NHC-1**	-
2 [a]	**NHC-2**	-
3 [a]	**NHC-3**	15
4 [a]	**NHC-4**	26
5 [a]	**NHC-5**	trace
6 [a]	**NHC-6**	13
7 [a]	**NHC-7**	trace
8 [a]	**NHC-8**	10
9 [a]	**NHC-9**	38
10 [a]	**NHC-10**	30
11 [a]	**NHC-11**	-
12 [a]	**NHC-12**	-
13 [a]	**NHC-13**	-
14 [b]	**NHC-9**	40
15 [c]	**NHC-9**	52
16 [d]	**NHC-9**	43
17 [e]	**NHC-9**	20
18 [f]	**NHC-9**	-



Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), CO (40 bar), MeCN (1.0 mL), K_2CO_3 (0.2 mmol), **PC-1** (2 mol%), **NHC** (10 mol%[a], 20 mol%[b], 25 mol%[c], 30 mol%[d], 50 mol%[e], 100 mol%[f]), r.t., 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard. NHC Preparation Method. [4]

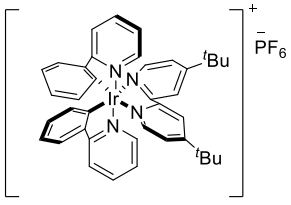
Table 2 Optimization of photosensitizer (PC)

Entry	**PC**	Yield (%)
1[c]	**PC-1**	52
2[c]	**PC-2**	30
3[c]	**PC-3**	22
4[c]	**PC-4**	trace
5[c]	**PC-5**	10
6[c]	**PC-6**	-
7[a]	**PC-1**	48
8[b]	**PC-1**	59
9[d]	**PC-1**	56
10	**-**	-



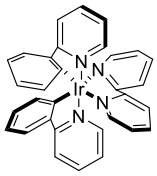
$Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$

PC-1



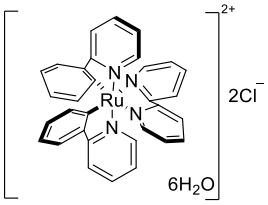
$[Ir(dtbbpy)(ppy)_2]PF_6$

PC-2



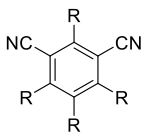
fac-$Ir(ppy)_3$

PC-3



$Ru(bpy)_3Cl_2 \cdot 6H_2O$

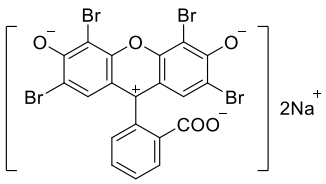
PC-4



R = carbazole

4CzIPN

PC-5



Eosin Y

PC-6

Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), CO (40 bar), MeCN (1.0 mL), K_2CO_3 (0.2 mmol), **PC** (1 mol%[a], 1.5 mol%[b], 2 mol%[c], 3 mol%[d]), **NHC-9** (25 mol%), r.t, 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard.

Table 3. Optimization of wavelength

Entry	Wavelength (nm)	Yield (%)
1	320-400	-
2	415	32
3	440	42
4	460	48
5	490	55

6	550	-
7	400-500	59
8	400-700	40

Reaction conditions: **1a** (0.1 mmol), **2a** (0.1 mmol), **3a** (0.15 mmol), CO (40 bar), MeCN (1.0 mL), K_2CO_3 (0.2 mmol), **PC-1** (1.5 mol%), **NHC-9** (25 mol%), r.t., 24 h. Determined by GC with hexadecane as internal standard.

Table 4. Optimization of pressure (CO)

Entry	Pressure(bar)	Yield (%)
1	30	32
2	35	40
3	40	59
4	45	58
5	50	55
6	60	40

Reaction conditions: **1a** (0.1 mmol), **2a** (0.1 mmol), **3a** (0.15 mmol), MeCN (1.0 mL), K_2CO_3 (0.2 mmol), **PC-1** (1.5 mol%), **NHC-9** (25 mol%), r.t., 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard.

Table 5. Optimization of temperature

Entry	Temperature (^oC)	Yield (%)
1	r.t.	59
2	40	65
3	50	40
4	60	trace

Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), CO (40 bar), MeCN (1.0 mL), K_2CO_3 (0.2 mmol), **PC-1** (1.5 mol%), **NHC-9** (25 mol%), 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard.

Table 6. Optimization of solvent

Entry	Solvent	Yield (%)
1[a]	MeCN	65
2[a]	MeCN/H_2O (9/1)	-
3[a]	DMF	-
4[a]	DMAc	-
5[a]	DMSO	-

6[a]	DCM	-
7[a]	DCE	-
8[a]	THF	42
9[a]	$PhCF_3$	trace
10[a]	Tolune	trace
11[b]	MeCN	55
12[c]	MeCN	60

Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), CO (40 bar), Solvent (0.1M[a], 0.2M[b], 0.05 M[c]), K_2CO_3 (0.2 mmol), **PC-1** (1.5 mol%), **NHC-9** (25 mol%), 40 °C, 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard.

Table 7. Optimization of base

Entry	Base	Yield (%)
1[c]	Na_2CO_3	50
2[c]	K_2CO_3	65
3[c]	Cs_2CO_3	-
4[c]	Li_2CO_3	-
5[c]	$NaHCO_3$	trace
6[c]	$KHCO_3$	trace
7[c]	K_3PO_4	45
8[c]	KH_2PO_4	34
9[c]	Na_2HPO_4	22
10[c]	NaH_2PO_4	20
11[c]	Na_3PO_4	30
12[c]	Na_2HPO_4	32
13[c]	NaH_2PO_4	10
14[c]	^{t}BuONa	-
15[c]	DBU	-
16[c]	NEt_3	-
17[a]	K_2CO_3	25
18[b]	K_2CO_3	40
19[d]	K_2CO_3	60
20[e]	K_2CO_3	62
21	-	-

Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), CO (40 bar), MeCN (1.0 mL), Base (0.05 mmol [a], 0.1 mmol [b], 0.2 mmol [c], 0.25 mmol [d], 0.3 mmol [e]), **PC-1** (1.5 mol%), **NHC-9** (25 mol%), 40 °C, 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard.

Table 8. Optimization of equivalent ratio

Entry	**1a** : **2a** : **3a**	Yield (%)
1	1.0 : 1.0 : 1.0	10
2	1.0 : 1.5 : 1.0	22
3	1.0 : 1.0 : 1.5	40
4	1.0 : 1.5 : 1.5	51
5	1.0 : 2.0 : 1.5	65
6	1.0 : 3.0 : 1.5	64
7	1.0 : 2.0 : 2.0	69
8	1.0 : 2.0 : 2.5	75
9	1.0 : 2.0 : 3.0	72

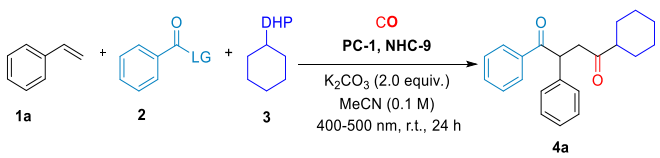
Reaction conditions: CO (40 bar), MeCN (1.0 mL), K_2CO_3 (0.2 mmol), **PC-1** (1.5 mol%), **NHC-9** (25 mol%), 40 °C, 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard.

Table 9. Optimization of time

Entry	Time (h)	Yield (%)
1	20	40
2	24	70
3	30	86
4	36	90 (82)
5	48	87

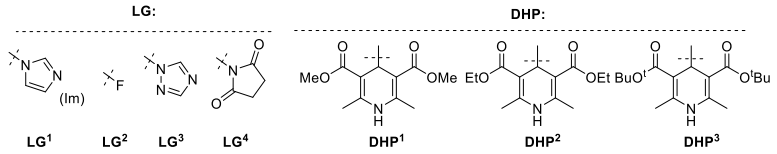
Reaction conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), **3a** (0.25 mmol), CO (40 bar), MeCN (1.0 mL), K_2CO_3 (0.2 mmol), **PC-1** (1.5 mol%), **NHC-9** (25 mol%), 40 °C, 400-500 nm. Determined by GC with hexadecane as internal standard. Isolated yield is shown in parentheses.

Table 10. Optimization of LG and DHP



Entry	**LG**	**DHP**	Yield (%)
1	**LG-1**	**DHP-1**	20
2	**LG-1**	**DHP-2**	52
3	**LG-1**	**DHP-3**	30

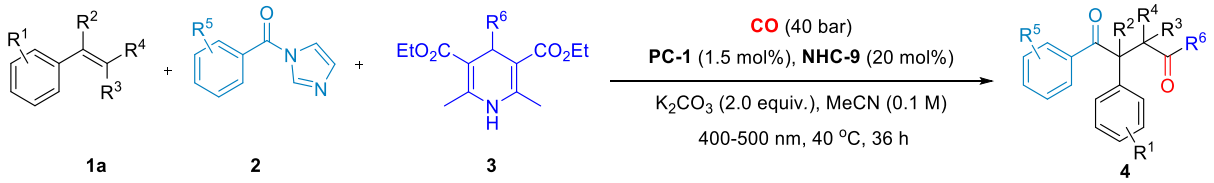
4	**LG-2**	**DHP-1**	trace
5	**LG-2**	**DHP-2**	22
6	**LG-2**	**DHP-3**	trace
7	**LG-3**	**DHP-1**	15
8	**LG-3**	**DHP-2**	35
9	**LG-3**	**DHP-3**	trace
10	**LG-4**	**DHP-1**	trace
11	**LG-4**	**DHP-2**	32
12	**LG-4**	**DHP-3**	10



Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), CO (40 bar), MeCN (1.0 mL), K_2CO_3 (0.2 mmol), **PC-1** (1.5 mol%), **NHC-9** (25 mol%), 40 °C, 24 h, 400-500 nm. Determined by GC with hexadecane as internal standard.

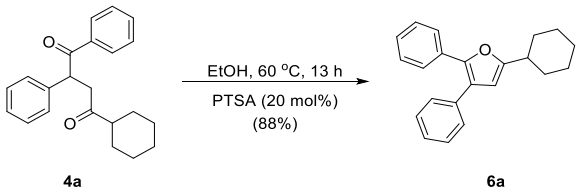
4. Characterization and procedure of 1,4-dione products 4 and synthetic transformations products 6

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



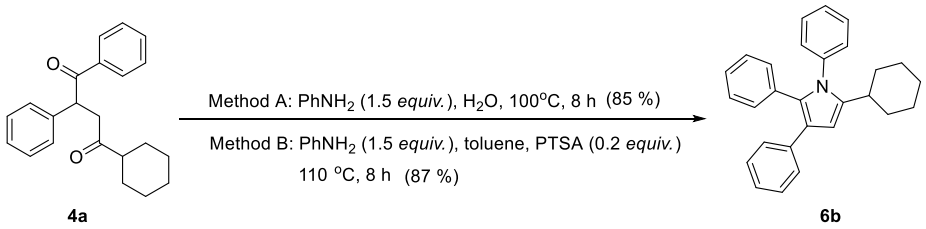
A 4 mL screw-cap vial was charged with Hantzsch ester **3** (2.5 equiv), acyl imidazoles **2** (1.5 equiv), **PC-1** (3.3 mg, 1.5 mol%), **NHC-9** (11mg, 25 mol%) and an oven-dried stirring bar. The vial was closed with a Teflon septum and cap and connected to the atmosphere via a needle. After replacing 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 36 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 6a[6]



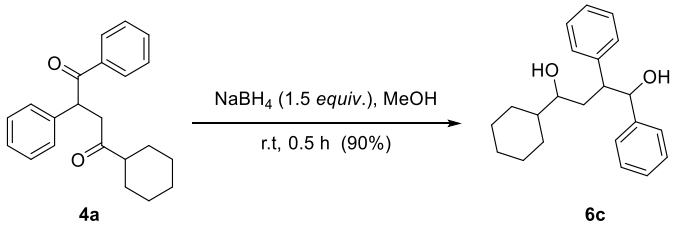
To a stirred solution of **4a** (32.0 mg, 0.1 mmol) in toluene (2 mL) at room temperature was added *p*-toluenesulfonic acid (3.5 mg, 0.02 mmol). The mixture was stirred vigorously for 13 hours at 60 °C. The crude product was purified by column chromatography on silica gel using petroleum ether and ethyl acetate (50:1) as to afford **6a** (26.6 mg, 88% yield) as a yellow solid. The **6a** was obtained by vacuum drying.

4.3 General procedure for the synthesis of 5-cyclohexyl-1,2,3-triphenyl-1H-pyrrole 6b[7]



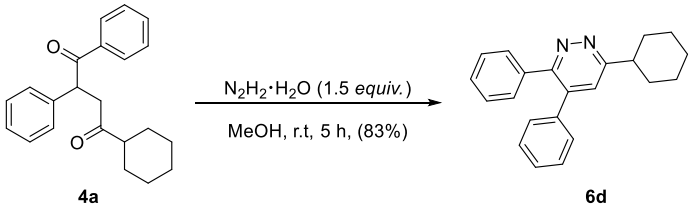
Method A: The mixture of **4a** (32.0 mg, 0.1 mmol) and aniline (14.0 mg, 0.15 mmol) in water (1 mL) at 100 °C for 8 hours; Method B: The mixture of **4a** (32.0 mg, 0.1 mmol), *p*-toluenesulfonic acid (3.5 mg, 0.02 mmol) and aniline (14.0 mg, 0.15 mmol) in toluene (1 mL) at 110 °C for 8 hours. The mixture was cooled to room temperature and diluted with ethyl acetate (1 mL) and washed with saturated $NaHCO_3$ solution (1 mL) and saturated 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[A]; 32.8 mg, 87% yield[B]) as a brown solid. The **6b** was obtained by vacuum drying.

4.4 General procedure for the synthesis of 4-cyclohexyl-1,2-diphenylbutane-1,4-diol 6c[8]



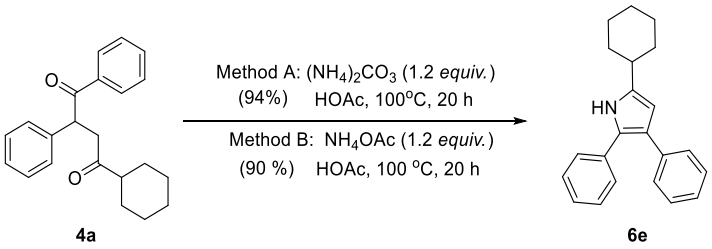
The mixture of **4a** (32.0 mg, 0.1 mmol) was added dropwise to a solution of sodium borohydride (6.0 mg, 0.15 mmol, 1.5 equiv) in MeOH (1 mL) at 0 °C for 1 hour, then room temperature for 2 hours. The reaction was diluted with ethyl acetate (1 mL), quenched with saturated NH_4Cl solution (1 mL), washed by saturated 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 **6c** (29.2 mg, 90% yield) as a white solid. The **6c** was obtained by vacuum drying.

4.5 General procedure for the synthesis of 6-cyclohexyl-3,4-diphenylpyridazine 6d[9]



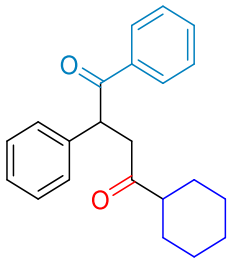
4a (32.0 mg, 0.1 mmol) was dissolved in hydrazine hydrate (15.0 uL, 0.15 mmol, 1.5 equiv) and MeOH (1 mL) was added. The reaction mixture was stirred at room temperature for 5 hours. The crude product was purified by flash chromatography on silica gel with petroleum ether/ethyl acetate (30:1) as eluent to afford **8d** (26.0 mg, 83% yield) as a yellow solid. The **6d** was obtained by vacuum drying.

4.6 General procedure for the synthesis of 5-cyclohexyl-2,3-diphenyl-1H-pyrrole 6e[10]



4a 6e

Method A: The solution of **4a** (32.0 mg, 0.1 mmol) and $(NH_4)_2CO_3$ (12.0 mg, 0.12 mmol) in acetic acid (1 mL) at 100 °C for 20 hours; Method B: The solution of **4a** (32.0 mg, 0.1 mmol) and NH_4OAc (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 $NaHCO_3$ 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 **6e** (28.2 mg, 95% yield[A]; 27.0 mg, 90% yield[B]) as a white solid. The **6e** was obtained by vacuum drying.

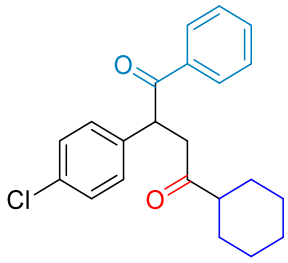


4a

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

Chromatography Pentane/EA = 20:1 (v/v), 52.6 mg (82%), white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.88 (m, 2H), 7.41 – 7.09 (m, 8H), 5.05 (dd, *J* = 10.1, 3.9 Hz, 1H), 3.55 (dd, *J* = 17.9, 10.1 Hz, 1H), 2.69 (dd, *J* = 18.0, 3.9 Hz, 1H), 2.33 – 1.08 (m, 11H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 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, 29.7, 28.3, 25.8, 25.6, 25.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{22}H_{24}O_2$ 343.1668, found: 343.1676.



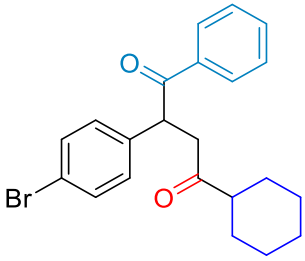
4b

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

Chromatography Pentane/EA = 20:1 (v/v), 49.6 mg (70%), white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.86 (m, 2H), 7.43 – 7.11 (m, 7H), 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), 2.36 – 1.11 (m, 11H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 211.8, 198.8, 137.2, 136.2, 133.2, 133.1, 129.5, 129.3, 128.8, 128.5, 50.7, 47.7, 45.3, 28.4, 25.8, 25.6, 25.5.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{22}H_{23}ClO_2$ 355.1459, found: 355.1458.

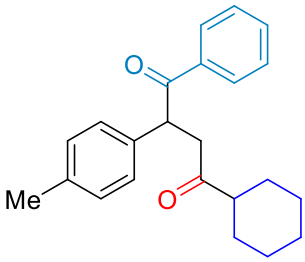


4c

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

Chromatography Pentane/EA = 20:1 (v/v), 58.4 mg (73%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 – 7.85 (m, 2H), 7.45 – 7.04 (m, 7H), 5.02 (dd, *J* = 9.8, 4.1 Hz, 1H), 3.51 (dd, *J* = 18.0, 9.8 Hz, 1H), 2.67 (dd, *J* = 18.0, 4.2 Hz, 1H), 2.35 – 1.08 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 211.8, 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 $[M^+]Na^+$ $C_{22}H_{23}BrO_2$ 421.0774, found: 421.0777.

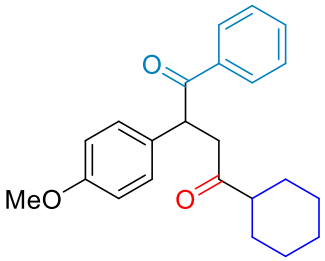


4d

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

Chromatography Pentane/EA = 20:1 (v/v), 55.6 mg (83%), white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 – 7.87 (m, 2H), 7.40 – 6.71 (m, 7H), 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), 2.33 – 1.11 (m, 11H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 212.3, 199.3, 158.7, 136.5, 132.8, 130.6, 129.2, 128.9, 128.4, 114.5, 55.2, 50.7, 47.6, 45.4, 28.4, 28.4, 25.9, 25.7, 25.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{26}O_2$ 335.2006, found: 335.2010.



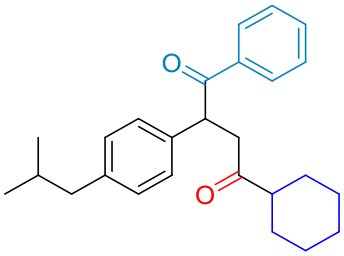
4e

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

Chromatography Pentane/EA = 30:1 (v/v), 54.6 mg (78%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91 – 7.87 (m, 2H), 7.42 – 6.71 (m, 7H), 5.00 (dd, *J* = 9.9, 4.1 Hz, 1H), 3.67 (s, 3H), 3.52 (dd, *J* = 17.9, 10.0 Hz, 1H), 2.67 (dd, *J* = 17.9, 4.1 Hz, 1H), 2.34 – 1.08 (m, 11H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 212.3, 199.3, 158.7, 136.4, 132.7, 130.6, 129.2, 128.8, 128.4, 114.5, 55.2, 50.7, 47.6, 45.4, 28.4, 28.3, 25.8, 25.6, 25.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{26}O_3$ 351.1995, found: 351.1960.

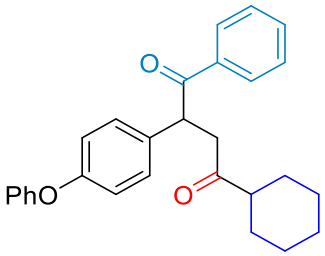


4f

4-cyclohexyl-2-(4-isobutylphenyl)-1-phenylbutane-1,4-dione (4f)

Chromatography Pentane/EA = 30:1 (v/v), 52.8 mg (70%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91 – 7.88 (m, 2H), 7.41 – 6.94 (m, 2H), 5.01 (dd, *J* = 10.1, 4.0 Hz, 8H), 3.53 (dd, *J* = 17.8, 10.1 Hz, 1H), 2.68 (dd, *J* = 17.9, 4.0 Hz, 1H), 2.31 (d, *J* = 7.2 Hz, 2H), 1.87 – 1.12 (m, 12H), 0.77 (d, *J* = 6.6 Hz, 6H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.3, 199.2, 140.7, 136.5, 135.8, 132.7, 129.8, 128.8, 128.4, 127.8, 50.7, 48.1, 45.5, 44.9, 30.1, 28.3, 28.3, 25.8, 25.6, 25.5, 22.3.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{26}H_{32}O_2$ 399.2295, found: 399.2297.

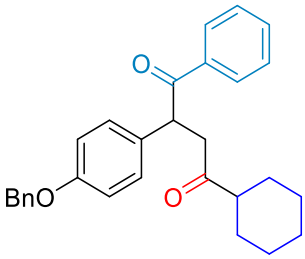


4g

4-cyclohexyl-2-(4-phenoxyphenyl)-1-phenylbutane-1,4-dione (4g)

Chromatography Pentane/EA = 30:1 (v/v), 49.6 mg (60%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.93 – 7.89 (m, 2H), 7.44 – 6.80 (m, 12H), 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), 2.36 – 1.08 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.1, 199.1, 156.8, 156.5, 136.4, 133.6, 132.8, 129.7, 129.4, 128.9, 128.5, 123.4, 119.1, 119.0, 50.7, 47.6, 45.5, 28.3, 28.3, 25.8, 25.6, 25.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{28}H_{28}O_3$ 435.1931, found: 435.1942.



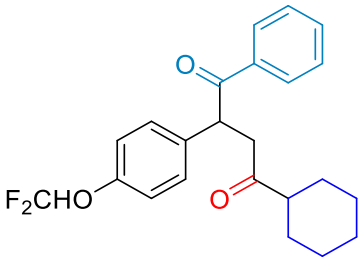
4h

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

Chromatography Pentane/EA = 30:1 (v/v), 52.8 mg (62%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91 – 7.87 (m, 2H), 7.42 – 6.78 (m, 12H), 5.00 (dd, *J* = 10.0, 4.1 Hz, 1H), 4.91 (s, 2H), 3.51 (dd, *J* = 17.9, 10.0 Hz, 1H), 2.67 (dd, *J* = 17.9, 4.1 Hz, 1H), 2.33 – 1.12 (m, 11H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 212.3, 199.2, 157.9, 136.8, 136.4, 132.7, 130.9, 129.2, 128.8, 128.6, 128.4, 127.9, 127.4, 115.4, 69.9, 50.7, 47.6, 45.4, 28.3, 25.8, 25.6, 25.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{29}H_{30}O_3$ 449.2087, found: 449.2098.

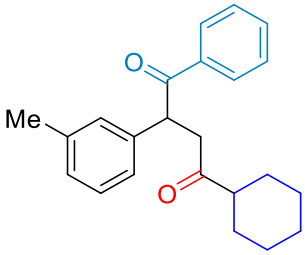


4i

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

Chromatography Pentane/EA = 30:1 (v/v), 43.2 mg (56%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91 – 7.87 (m, 2H), 7.45 – 6.90 (m, 7H), 6.62 – 6.12 (t, 1H), 5.06 (dd, *J* = 9.9, 4.1 Hz, 1H), 3.53 (dd, *J* = 17.9, 9.9 Hz, 1H), 2.69 (dd, *J* = 17.9, 4.2 Hz, 1H), 2.34 – 1.05 (m, 11H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 211.9, 198.9, 150.2, 136.2, 135.8, 133.0, 129.5, 128.8, 128.5, 120.2, 115.7, 50.7, 47.6, 45.3, 28.3, 25.8, 25.6, 25.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ -80.79, -80.98.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{23}H_{24}F_2O_3$ 409.1586, found: 409.1591.

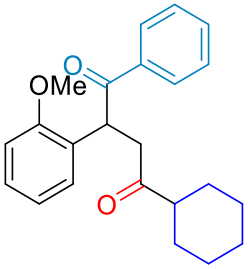


4j

4-cyclohexyl-1-phenyl-2-(m-tolyl)butane-1,4-dione (4j)

Chromatography Pentane/EA = 20:1 (v/v), 53.6 mg (80%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92 – 7.88 (m, 2H), 7.42 – 6.91 (m, 7H), 6.93 (dd, *J* = 2.3, 1.3 Hz, 1H), 5.01 (dd, *J* = 10.3, 3.8 Hz, 1H), 3.55 (dd, *J* = 17.9, 10.3 Hz, 1H), 2.66 (dd, *J* = 17.9, 3.8 Hz, 1H), 2.34 – 2.25 (m, 1H), 2.21 (s, 3H), 1.89 – 1.11 (m, 10H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.2, 199.1, 138.8, 138.6, 136.5, 132.8, 128.9, 128.9, 128.6, 128.4, 127.9, 125.2, 50.7, 48.4, 45.5, 28.4, 25.8, 25.6, 25.6, 21.4.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{26}O_2$ 335.2006, found: 335.2008.

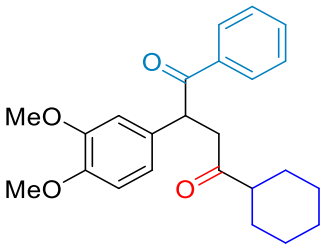


4k

4-cyclohexyl-2-(2-methoxyphenyl)-1-phenylbutane-1,4-dione (4k)

Chromatography Pentane/EA = 30:1 (v/v), 42.0 mg (60%), pale yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.90 – 7.86 (m, 2H), 7.39 – 6.72 (m, 7H), 5.46 (dd, *J* = 10.4, 3.4 Hz, 1H), 3.81 (s, 3H), 3.45 (dd, *J* = 17.7, 10.4 Hz, 1H), 2.56 (dd, *J* = 17.7, 3.4 Hz, 1H), 2.39 – 1.09 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.4, 199.7, 155.9, 136.4, 132.6, 128.7, 128.6, 128.4, 128.2, 127.2, 120.9, 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 $[M^+]Na^+$ $C_{23}H_{26}O_3$ 373.1774, found: 373.1784.

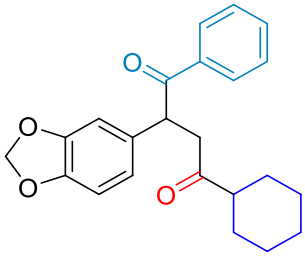


4l

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

Chromatography Pentane/EA = 30:1 (v/v), 50.2 mg (66%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92 – 7.88 (m, 2H), 7.43 – 6.67 (m, 6H), 4.99 (dd, *J* = 10.0, 4.0 Hz, 1H), 3.75 (d, *J* = 6.4 Hz, 6H), 3.53 (dd, *J* = 17.9, 9.9 Hz, 1H), 2.69 (dd, *J* = 17.9, 4.0 Hz, 1H), 2.35 – 1.08 (m, 11H).
^{13}C NMR (101 MHz, $CDCl_3$) 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, 55.9, 55.8, 50.7, 48.2, 45.5, 28.4, 28.3, 25.8, 25.6, 25.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{24}H_{28}O_4$ 403.1880, found: 403.1886.

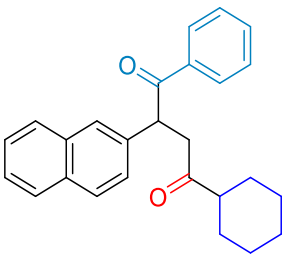


4m

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

Chromatography Pentane/EA = 20:1 (v/v), 51.0 mg (70%), pale yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92 – 7.87 (m, 2H), 7.43 – 5.81 (m, 6H), 4.96 (dd, *J* = 10.0, 4.0 Hz, 1H), 3.50 (dd, *J* = 17.9, 10.0 Hz, 1H), 2.66 (dd, *J* = 17.9, 4.0 Hz, 1H), 2.34 – 1.07 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.2, 199.0, 148.1, 146.8, 136.3, 132.8, 132.3, 128.8, 128.4, 121.5, 108.8, 108.3, 101.08, 50.7, 48.0, 45.4, 28.3, 25.8, 25.6, 25.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{23}H_{24}O_4$ 387.1567, found: 387.1571.

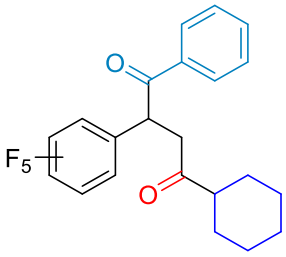


4n

4-cyclohexyl-2-(naphthalen-2-yl)-1-phenylbutane-1,4-dione (4n)

Chromatography Pentane/EA = 20:1 (v/v), 37.0 mg (50%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.71 – 7.23 (m, 10H), 5.22 (dd, *J* = 10.0, 3.9 Hz, 1H), 3.64 (dd, *J* = 18.0, 10.0 Hz, 1H), 2.76 (dd, *J* = 18.0, 3.9 Hz, 1H), 2.37 – 1.07 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.0, 199.0, 136.4, 136.2, 133.6, 132.8, 132.4, 128.9, 128.9, 128.4, 127.7, 127.6, 126.9, 126.3, 126.0, 125.9, 50.7, 48.6, 45.5, 28.4, 28.4, 25.8, 25.6, 25.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{26}H_{26}O_2$ 393.1825, found: 393.1829.

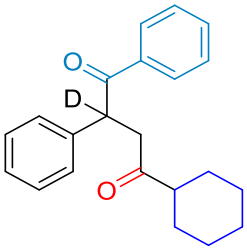


4o

4-cyclohexyl-2-(perfluorophenyl)-1-phenylbutane-1,4-dione (4o)

Chromatography Pentane/EA = 20:1 (v/v), 50.8mg (59%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.75 – 7.07 (m, 2H), 7.47 – 7.07 (m, 3H), 4.95 (dd, *J* = 10.0, 4.0 Hz, 1H), 3.50 (dd, *J* = 18.0, 10.0 Hz, 1H), 2.67 (dd, *J* = 18.0, 4.0 Hz, 1H), 2.33 – 1.11 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 211.8, 197.8, 136.8, 134.9, 133.4, 131.9, 130.3, 129.4, 129.4, 128.3, 50.6, 47.8, 45.2, 28.4, 28.4, 25.8, 25.6, 25.6.
^{19}F NMR (282 MHz, $CDCl_3$) δ -140.72 – -140.85 (m, 2F), -154.29 (t, *J* = 20.9 Hz, 1F), -160.78 – -160.96 (m, 2F).
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{22}H_{19}F_5O_2$ 453.1823, found: 453.1830.

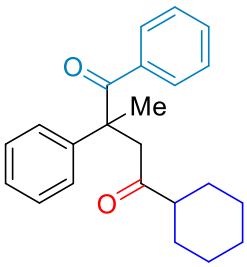


4p

4 4-cyclohexyl-1,2-diphenylbutane-1,4-dione-2-d (4p)

Chromatography Pentane/EA = 20:1 (v/v), 51.4 mg (80%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92 – 7.88 (m, 2H), 7.41 – 7.08 (m, 8H), 3.55 (d, *J* = 17.9 Hz, 1H), 2.68 (d, *J* = 18.0 Hz, 1H), 2.34 – 1.07 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.1, 199.5, 138.6, 136.4, 132.8, 129.1, 128.9, 128.8, 128.4, 128.1, 128.1, 127.2, 50.6, 48.5, 45.3, 28.3, 25.8, 25.6, 25.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{22}H_{23}DO_2$ 322.1912, found: 322.1915.

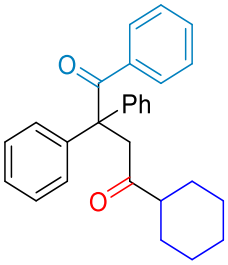


4q

4-cyclohexyl-2-methyl-1,2-diphenylbutane-1,4-dione (4q)

Chromatography Pentane/EA = 20:1 (v/v), 56.8 mg (85%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 – 7.09 (m, 10H), 3.31 (dd, *J* = 17.2, 0.8 Hz, 1H), 2.93 (d, *J* = 17.2 Hz, 1H), 2.15 – 2.06 (m, 1H), 1.70 (s, 3H), 1.66 – 1.07 (m, 10H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 211.40, 203.19, 143.05, 137.50, 131.15, 129.08, 128.92, 127.81, 127.18, 126.06, 53.04, 51.39, 51.20, 28.32, 28.14, 25.77, 25.58, 25.54, 24.19.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{26}O_2$ 335.2006, found: 335.2010.

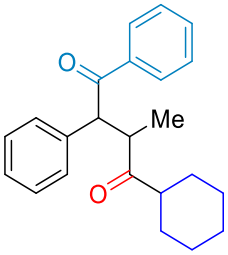


4r

4-cyclohexyl-1,2,2-triphenylbutane-1,4-dione (4r)

Chromatography Pentane/EA = 20:1 (v/v), 47.6 mg (60%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 – 7.33 (m, 2H), 7.26 – 7.07 (m, 13H), 3.67 (s, 2H), 2.09 – 0.88 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 210.1, 201.2, 142.5, 139.0, 130.7, 129.2, 129.1, 128.2, 127.6, 126.9, 61.7, 51.8, 51.1, 27.9, 25.7, 25.4.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{28}H_{28}O_2$ 419.1982, found: 419.1980.

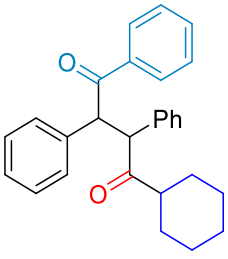


4s

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

Chromatography Pentane/EA = 30:1 (v/v), 30.8 mg (46%), white solid. dr = 7:3, slightly sticky white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.04 – 7.84 (m, 2H), 7.45 – 7.08 (m, 8H), 4.81 (d, *J* = 10.6 Hz, 0.69H), 4.76 (d, *J* = 10.6 Hz, 0.31H), 3.73 – 3.48 (m, 1H), 1.92 – 0.86 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 217.3(minor), 200.0(minor), 198.8, 137.1, 137.0(minor), 136.9, 136.7(minor), 133.1, 132.7(minor), 129.9, 129.1(minor), 129.0, 128.9(minor), 128.8, 128.7(minor), 128.6, 128.4, 128.4(minor), 127.4, 56.4(minor), 55.9, 51.7(minor), 49.7, 48.3, 30.2(minor), 29.1(minor), 28.3, 27.9(minor), 26.9, 26.0(minor), 25.9, 25.7(minor), 25.6, 25.6(minor), 25.4, 16.9, 15.1(minor).
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{23}H_{26}O_2$ 357.1825, found: 357.1827.

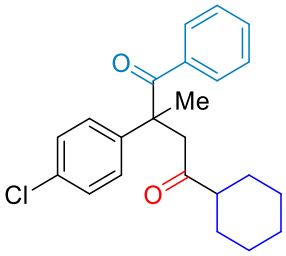


4t

1-cyclohexyl-2,3,4-triphenylbutane-1,4-dione (4t).

Chromatography Pentane/EA = 30:1 (v/v), 39.6 mg (50%), white solid. dr = 8:2, slightly sticky white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.04 – 7.73 (m, 2H), 7.43 – 6.85 (m, 13H), 5.52 – 4.61 (m, 2H), 2.43 – 0.91 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.99, 211.4, 199.6, 198.1, 136.7, 136.6, 135.8, 132.8, 132.7, 129.3, 129.0, 128.7, 128.7, 128.6, 128.6, 128.5, 128.4, 128.2, 127.4, 127.3, 127.2, 126.9, 61.4, 60.1, 57.3, 55.8, 51.0, 50.0, 29.3, 28.3, 27.7, 27.6, 25.9, 25.8, 25.6, 25.6, 25.3, 25.1.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{28}H_{28}O_2$ 397.2162, found: 397.2164.

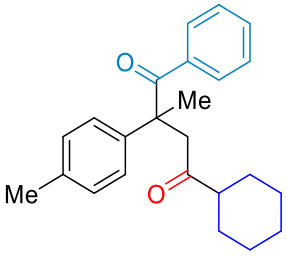


4u

2-(4-chlorophenyl)-4-cyclohexyl-2-methyl-1-phenylbutane-1,4-dione (4u)

Chromatography Pentane/EA = 30:1 (v/v), 53.2 mg (72%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33 – 7.05 (m, 9H), 3.29 (d, *J* = 17.4 Hz, 1H), 2.91 (d, *J* = 17.4 Hz, 1H), 2.17 – 2.07 (m, 1H), 1.69 (s, 3H), 1.68 – 1.06 (m, 10H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 210.9, 202.8, 141.6, 137.2, 133.2, 131.3, 129.2, 128.8, 127.9, 127.6, 52.6, 51.3, 51.1, 28.3, 28.2, 25.7, 25.5, 24.2.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{25}ClO_2$ 369.1616, found: 369.1618.

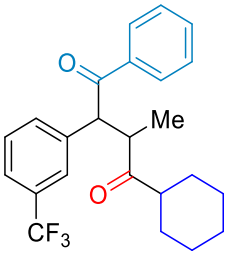


4v

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

Chromatography Pentane/EA = 30:1 (v/v), 48.8 mg (70%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 – 7.09 (m, 9H), 3.31 (d, *J* = 17.2 Hz, 1H), 2.89 (d, *J* = 17.2 Hz, 1H), 2.28 (s, 3H), 2.14 – 2.08 (m, 1H), 1.67 (s, 3H), 1.65 – 1.05 (m, 10H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 211.45, 203.4, 140.0, 137.7, 136.9, 131.0, 129.8, 128.9, 127.8, 125.9, 52.7, 51.4, 51.2, 28.3, 28.2, 25.8, 25.6, 25.6, 24.2, 20.9.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{24}H_{28}O_2$ 349.2162, found: 349.2171.

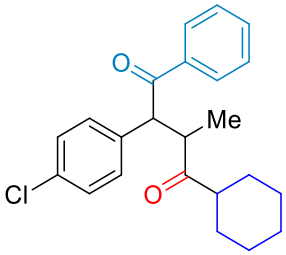


4w

1-cyclohexyl-2-methyl-4-phenyl-3-(3-(trifluoromethyl)phenyl)butane-1,4-dione (4w)

Chromatography Pentane/EA = 30:1 (v/v), 41.0 mg (51%), white solid. dr = 6:4, slightly sticky white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.04 – 7.83 (m, 2H), 7.55 – 7.28 (m, 7H), 4.93 (d, *J* = 10.6 Hz, 0.4H), 4.84 (d, *J* = 10.6 Hz, 0.6H), 3.74 – 3.51 (m, 1H), 2.64 – 0.86 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 216.6, 215.6, 199.5, 198.5, 138.3, 138.1, 136.7, 136.4, 133.5, 133.0, 132.8, 132.3, 129.9, 129.5, 129.2, 128.8, 128.7, 128.6, 128.6, 128.5, 125.5, 125.4, 124.4, 124.4, 55.8, 54.9, 51.3, 49.6, 48.6, 48.4, 29.1, 28.2, 27.7, 27.2, 25.9, 25.8, 25.6, 25.5, 25.4, 16.9, 15.1, 14.2.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{24}H_{25}F_3O_2$ 403.1879, found: 403.1881.

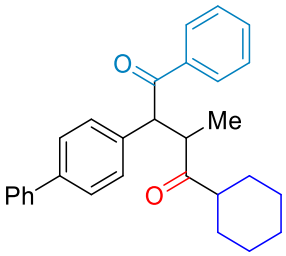


4x

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

Chromatography Pentane/EA = 30:1 (v/v), 31.8 mg (43%), white solid. dr = 8:2, slightly sticky white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92 – 7.81 (m, 2H), 7.48 – 7.11 (m, 7H), 4.83 (d, *J* = 10.6 Hz, 0.8H), 4.74 (d, *J* = 10.6 Hz, 0.2H), 3.69 – 3.45 (m, 1H), 2.02 – 0.65 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 216.7, 215.9(minor), 199.7(minor), 198.6, 138.6, 136.7(minor), 136.4(minor), 136.4, 135.7(minor), 135.5, 133.3, 132.9, 131.9(minor), 130.4, 130.2(minor), 129.9(minor), 129.5(minor), 129.3, 129.2, 129.0(minor), 128.9, 128.7(minor), 128.6, 128.6(minor), 128.5(minor), 128.4, 55.5(minor), 54.8, 51.3(minor), 50.4, 48.3(minor), 46.4, 38.5, 31.4(minor), 28.1(minor), 28.1(minor), 27.8(minor), 27.2(minor), 25.8, 25.6, 25.5, 17.1, 16.9(minor), 15.0.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{25}ClO_2$ 369.1616, found: 369.1622.

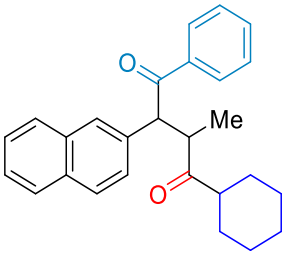


4y

2-([1,1'-biphenyl]-4-yl)-4-cyclohexyl-3-methyl-1-phenylbutane-1,4-dione (4y)

Chromatography Pentane/EA = 30:1 (v/v), 47.6 mg (58%), white solid. dr = 8:2, slightly sticky white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.97 – 7.87 (m, 2H), 7.47 – 7.23 (m, 12H), 4.86 (d, *J* = 10.6 Hz, 0.8H), 3.30 (d, *J* = 10.6 Hz, 0.2H), 3.76 – 3.52 (m, 1H), 1.97 – 0.69 (m, 14H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 217.3(minor), 216.3, 199.9(minor), 198.7, 140.5, 140.4(minor), 140.2, 136.9, 136.1, 135.9(minor), 133.2, 132.7(minor), 129.4, 129.3(minor), 129.1, 128.7(minor), 128.6(minor), 128.4, 127.6(minor), 127.4(minor), 127.3, 127.3(minor), 126.9, 55.9(minor), 55.5, 51.7, 49.7(minor), 48.3, 29.1(minor), 28.3(minor), 27.9, 27.1, 25.9(minor), 25.8(minor), 25.7, 25.6, 25.4, 16.9, 15.2(minor).
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{29}H_{30}O_2$ 411.2319, found: 411.2327.

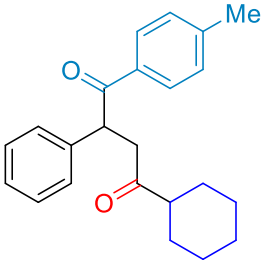


4z

1-cyclohexyl-2-methyl-3-(naphthalen-2-yl)-4-phenylbutane-1,4-dione (4z)

Chromatography Pentane/EA = 30:1 (v/v), 30.8 mg (40%), white solid. dr = 9:1, slightly sticky white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.38 – 7.11 (m, 12H), 7.46 – 3.58 (m, 3H), 1.79 – 0.50 (m, 14H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 217.4(minor), 216.0, 198.9(minor), 198.2, 139.9, 137.0, 134.3(minor), 133.4, 132.9(minor), 132.6, 129.5, 128.9, 128.5, 128.4, 128.3, 126.7, 125.8, 125.7, 125.2, 123.7, 51.3, 29.2(minor), 28.1, 26.9(minor), 26.0, 25.9, 25.7, 25.6, 25.6, 25.2, 16.8.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{27}H_{28}O_2$ 385.2162, found: 385.2164.

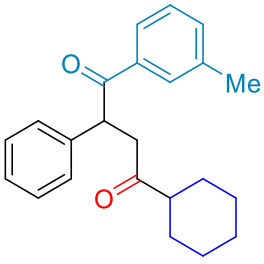


4aa

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

Chromatography Pentane/EA = 20:1 (v/v), 55.6 mg (83%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.82 – 7.78 (m, 2H), 7.20 – 7.06 (m, 7H), 5.03 (dd, *J* = 10.0, 4.1 Hz, 1H), 3.53 (dd, *J* = 17.9, 10.0 Hz, 1H), 2.67 (dd, *J* = 17.9, 4.1 Hz, 1H), 2.35 – 2.28 (m, 1H), 2.26 (s, 3H), 1.88 – 1.07 (m, 10H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.1, 198.6, 143.6, 139.0, 133.8, 129.1, 129.0, 129.0, 128.1, 127.1, 50.7, 48.4, 45.3, 28.4, 28.3, 25.8, 25.6, 25.6, 21.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{26}O_2$ 335.2006, found: 335:2008.



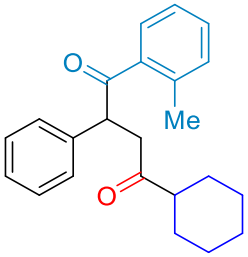
4ab

4-cyclohexyl-2-phenyl-1-(m-tolyl)butane-1,4-dione (4ab)

Chromatography Pentane/EA = 20:1 (v/v), 46.8 mg (70%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 – 7.67 (m, 2H), 7.20 – 7.07 (m, 7H), 5.04 (dd, *J* = 10.1, 3.9 Hz, 1H), 3.55 (dd, *J* = 17.9, 10.1 Hz, 1H), 2.67 (dd, *J* = 17.9, 3.9 Hz, 1H), 2.37 – 2.27 (m, 1H), 2.25 (s, 3H), 1.87 – 1.08 (m, 10H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 212.1, 199.2, 138.7, 138.1, 136.4, 133.6, 129.3, 129.0, 128.2, 128.1, 127.1, 126.1, 50.6, 48.4, 45.4, 28.3, 25.8, 25.6, 25.5, 21.3.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{26}O_2$ 335.2006, found: 335.2003.

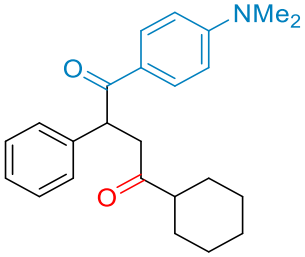


4ac

4-cyclohexyl-2-phenyl-1-(o-tolyl)butane-1,4-dione (4ac)

Chromatography Pentane/EA = 20:1 (v/v), 53.6 mg (80%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ7.76 – .08 (m, 8), 7.03 (dd, *J* = 10.7, 3.5 Hz, 1H), 4.86 (dd, *J* = 10.7, 3.5 Hz, 1H), 3.63 (dd, *J* = 18.0, 10.7 Hz, 1H), 2.65 (dd, *J* = 18.0, 3.5 Hz, 1H), 2.37 – 1.08 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.4, 202.9, 138.4, 137.9, 137.6, 131.3, 130.7, 128.9, 128.3, 128.3, 127.3, 125.4, 51.55, 50.6, 44.7, 28.4, 25.8, 25.6, 25.6, 20.4.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{26}O_2$ 335.2006, found: 335.2006.

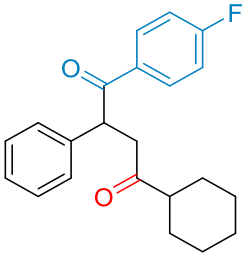


4ad

4-cyclohexyl-1-(4-(dimethylamino)phenyl)-2-phenylbutane-1,4-dione (4ad)

Chromatography Pentane/EA = 20:1 (v/v), 36.4 mg (50%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.85 – 6.47 (m, 9H), 5.01 (dd, *J* = 9.6, 4.4 Hz, 1H), 3.50 (dd, *J* = 17.7, 9.6 Hz, 1H), 2.91 (s, 6H), 2.64 (dd, *J* = 17.7, 4.4 Hz, 1H), 2.34 – 1.03 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.35, 196.68, 153.15, 140.16, 131.11, 128.87, 128.00, 126.77, 124.20, 110.62, 50.84, 47.77, 45.24, 39.95, 28.33, 28.26, 25.86, 25.67, 25.56.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{24}H_{29}NO_2$ 364.2271, found: 364.2274.



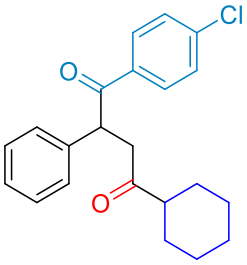
4ae

4-cyclohexyl-1-(4-fluorophenyl)-2-phenylbutane-1,4-dione (4ae)

Chromatography Pentane/EA = 20:1 (v/v), 46.0 mg (68%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95 – 7.89 (m, 2H), 7.24 – 6.92 (m, 7H), 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), 2.35 – 1.08 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.1, 197.5, 138.5, 131.5, 131.4, 129.2, 128.0, 127.3, 115.7, 115.4, 50.6, 48.5, 45.5, 28.4, 25.8, 25.6, 25.6.

^{19}F NMR (376 MHz, $CDCl_3$) δ -105.59 (qd, *J* = 8.2, 5.6 Hz, 1F).
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{22}H_{23}FO_2$ 361.1574, found: 361.1582.

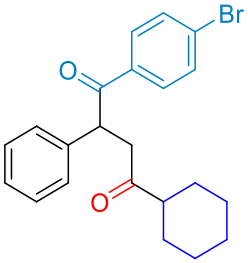


4af

1-(4-chlorophenyl)-4-cyclohexyl-2-phenylbutane-1,4-dione (4af)

Chromatography Pentane/EA = 20:1 (v/v), 48.2 mg (66%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.85 – 7.80 (m, 2H), 7.29 – 7.10 (m, 7H), 4.98 (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), 2.35 – 1.12 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.1, 197.9, 139.2, 138.3, 134.7, 130.3, 129.2, 128.7, 128.0, 127.4, 50.6, 48.6, 45.4, 28.4, 25.8, 25.6, 25.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{22}H_{23}ClO_2$ 355.1460, found: 355.1468.

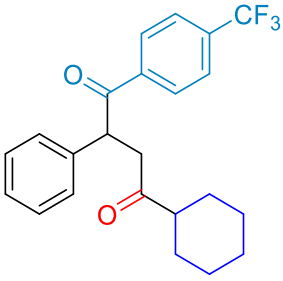


4ag

1-(4-bromophenyl)-4-cyclohexyl-2-phenylbutane-1,4-dione (4ag)

Chromatography Pentane/EA = 20:1 (v/v), 55.8 mg (70%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 – 7.72 (m, 2H), 7.45 – 7.09 (m, 7H), 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 – 1.12 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.0, 198.1, 138.3, 135.2, 131.7, 130.4, 129.2, 128.0, 127.9, 127.4, 50.6, 48.6, 45.4, 28.3, 25.8, 25.6, 25.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{22}H_{23}BrO_2$ 421.0773, found: 421.0774.



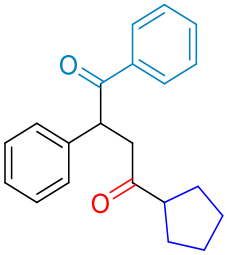
4ah

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

Chromatography Pentane/EA = 20:1 (v/v), 38.8 mg (50%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00 – 7.12 (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 – 1.08 (m, 11H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 212.1, 198.3, 139.3, 137.8, 129.3, 129.1, 128.1, 127.5, 125.6, 125.5, 125.5, 125.43, 50.56, 48.88, 45.51, 28.38, 28.36, 25.79, 25.59, 25.55.
^{19}F NMR (282 MHz, $CDCl_3$) δ -63.19.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{23}F_3O_2$ 389.1723, found: 389.1727.

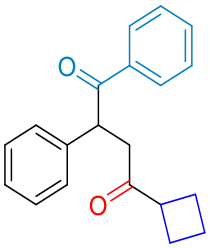


4ai

4-cyclopentyl-1,2-diphenylbutane-1,4-dione (4ai)

Chromatography Pentane/EA = 20:1 (v/v), 48.4 mg (79%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92 – 7.88 (m, 2H), 7.41 – 7.09 (m, 8H), 5.07 (dd, *J* = 10.1, 4.0 Hz, 1H), 3.56 (dd, *J* = 17.9, 10.1 Hz, 1H), 2.83 (p, *J* = 8.0 Hz, 1H), 2.72 (dd, *J* = 17.9, 4.0 Hz, 1H), 1.80 – 1.46 (m, 8H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 211.2, 199.0, 138.6, 136.4, 132.8, 129.1, 128.8, 128.5, 128.4, 128.3, 128.1, 127.2, 51.2, 48.5, 46.5, 28.7, 28.7, 25.9, 25.9.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{21}H_{22}O_2$ 307.1693, found: 307.1690.

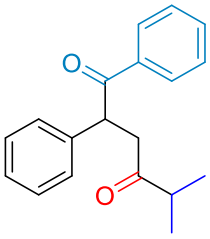


4aj

4-cyclobutyl-1,2-diphenylbutane-1,4-dione (4aj)

Chromatography Pentane/EA = 20:1 (v/v), 23.8 mg (40%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.93 – 7.89 (m, 2H), 7.43 – 7.07 (m, 8H), 5.07 (dd, *J* = 10.0, 4.1 Hz, 1H), 3.44 (dd, *J* = 17.9, 10.0 Hz, 1H), 3.25 – 2.66 (m, 1H), 2.62 (dd, *J* = 17.8, 4.1 Hz, 1H), 2.28 – 1.19 (m, 7H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 209.9, 199.0, 138.7, 136.4, 132.8, 129.1, 128.8, 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 $[M^+]H^+$ $C_{20}H_{20}O_2$ 293.1536, found: 293.1540.

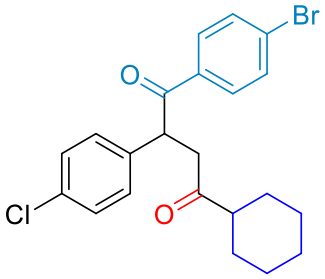


4ak

5-methyl-1,2-diphenylhexane-1,4-dione (4ak)

Chromatography Pentane/EA = 20:1 (v/v), 49.7 mg (88%), pale yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92 – 7.88 (m, 2H), 7.42 – 7.09 (m, 8H), 5.06 (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), 2.58 (dq, *J* = 13.9, 6.9 Hz, 1H), 1.06 (d, *J* = 6.9 Hz, 3H), 1.00 (d, *J* = 7.0 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 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 $[M^+]Na^+$ $C_{19}H_{20}O_2$ 303.1356, found: 303.1364.

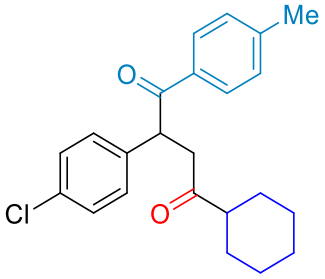


4al

1-(4-bromophenyl)-2-(4-chlorophenyl)-4-cyclohexylbutane-1,4-dione (4al)

Chromatography Pentane/EA = 20:1 (v/v), 60.6 mg (70%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 – 7.70 (m, 2H), 7.47 – 7.08 (m, 6H), 4.95 (dd, *J* = 10.0, 4.0 Hz, 1H), 3.50 (dd, *J* = 18.0, 10.0 Hz, 1H), 2.67 (dd, *J* = 18.0, 4.0 Hz, 1H), 2.33 – 1.13 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 211.7, 197.8, 136.7, 134.9, 133.4, 131.8, 130.3, 129.4, 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 $[M^+]H^+$ $C_{22}H_{22}BrClO_2$ 433.0565, found: 433.0577.

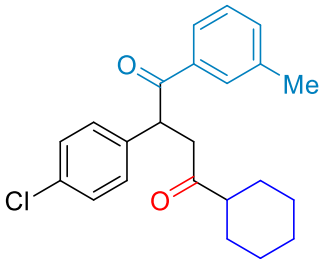


4am

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

Chromatography Pentane/EA = 20:1 (v/v), 53.8 mg (73%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79 – 7.75 (m, 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), 2.66 (dd, *J* = 17.9, 4.3 Hz, 1H), 2.36 – 2.29 (m, 1H), 2.27 (s, 3H), 1.90 – 1.11 (m, 10H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 211.8, 198.3, 143.9, 137.8, 133.6, 133.1, 129.4, 129.2, 128.9, 50.7, 47.6, 45.1, 28.3, 25.8, 25.6, 25.5, 21.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{25}ClO_2$ 391.1616, found: 391.1620.

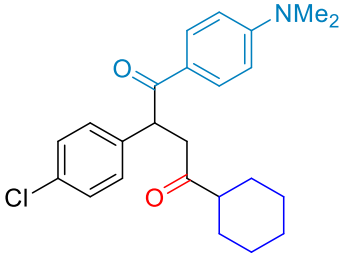


4an

2-(4-chlorophenyl)-4-cyclohexyl-1-(m-tolyl)butane-1,4-dione (4an)

Chromatography Pentane/EA = 30:1 (v/v), 50.8 mg (69%), white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 – 7.65 (m, 2H), 7.24 – 7.11 (m, 6H), 5.03 (dd, *J* = 9.9, 4.2 Hz, 1H), 3.51 (dd, *J* = 18.0, 9.8 Hz, 1H), 2.67 (dd, *J* = 18.0, 4.2 Hz, 1H), 2.33 – 2.28 (m, 1H), 2.28 (s, 3H), 1.86 – 1.06 (m, 10H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 211.9, 199.0, 138.3, 137.3, 136.2, 133.8, 129.5, 129.3, 129.2, 128.4, 126.1, 50.7, 47.7, 45.2, 28.3, 25.8, 25.6, 25.5, 21.3.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{23}H_{25}ClO_2$ 391.1435, found: 391.1440.

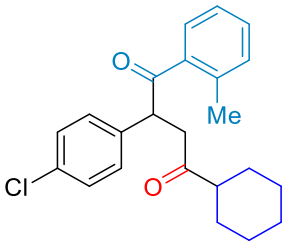


4ao

2-(4-chlorophenyl)-4-cyclohexyl-1-(4-(dimethylamino)phenyl)butane-1,4-dione (4ao)

Chromatography Pentane/EA = 20:1 (v/v), 36.0 mg (46%), white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 – 6.59 (m, 8H), 5.09 (dd, *J* = 9.2, 4.7 Hz, 1H), 3.54 (dd, *J* = 17.8, 9.2 Hz, 1H), 3.03 (s, 6H), 2.73 (dd, *J* = 17.8, 4.7 Hz, 1H), 2.41 – 1.20 (m, 11H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 212.1, 196.3, 153.2, 138.6, 132.7, 131.1, 129.4, 129.0, 124.0, 110.8, 50.8, 46.9, 45.0, 40.1, 28.3, 28.3, 25.8, 25.7, 25.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{24}H_{28}ClNO_2$ 420.1701, found: 420.1700.

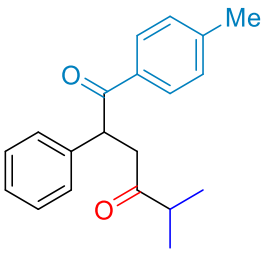


4ap

2-(4-chlorophenyl)-4-cyclohexyl-1-(o-tolyl)butane-1,4-dione (4ap)

Chromatography Pentane/EA = 20:1 (v/v), 59.0 mg (80%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.23 – 7.03 (m, 7H), 4.85 (dd, *J* = 10.4, 3.7 Hz, 1H), 3.58 (dd, *J* = 18.0, 10.4 Hz, 1H), 2.63 (dd, *J* = 18.0, 3.7 Hz, 1H), 2.38 – 2.29 (m, 1H), 2.16 (s, 3H), 1.90 – 1.09 (m, 10H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.0, 202.5, 138.1, 137.9, 136.2, 133.2, 131.4, 130.9, 129.6, 129.1, 128.3, 125.5, 50.8, 50.6, 44.5, 28.4, 28.4, 25.8, 25.6, 25.5, 20.5.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{25}ClO_2$ 369.1616, found: 369.1619.

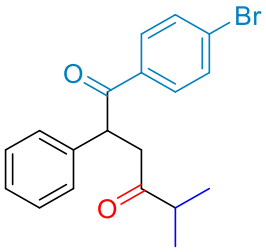


4aq

5-methyl-2-phenyl-1-(p-tolyl)hexane-1,4-dione (4aq)

Chromatography Pentane/EA = 20:1 (v/v), 52.4 mg (89%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.82 – 7.78 (m, 2H), 7.20 – 7.07 (m, 7H), 5.04 (dd, *J* = 9.9, 4.1 Hz, 1H), 3.54 (dd, *J* = 17.8, 9.9 Hz, 1H), 2.70 (dd, *J* = 17.8, 4.1 Hz, 1H), 2.57 (dq, *J* = 13.9, 6.9 Hz, 1H), 2.26 (s, 3H), 1.05 (d, *J* = 6.9 Hz, 3H), 1.00 (d, *J* = 7.0 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.8, 198.5, 143.6, 138.9, 133.8, 129.1, 129.1, 128.9, 128.1 127.1, 48.4, 45.1, 40.8, 21.6, 18.1.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{20}H_{22}O_2$ 295.1693, found: 295.1691.

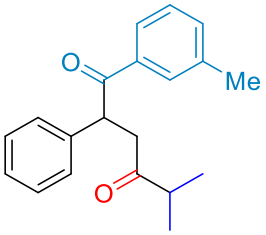


4ar

1-(4-bromophenyl)-5-methyl-2-phenylhexane-1,4-dione (4ar)

Chromatography Pentane/EA = 20:1 (v/v), 52.6 mg (73%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 – 7.73 (m, 2H), 7.45 – 7.10 (m, 7H), 4.97 (dd, *J* = 10.1, 3.9 Hz, 1H), 3.55 (dd, *J* = 18.0, 10.2 Hz, 1H), 2.71 (dd, *J* = 18.0, 3.9 Hz, 1H), 2.57 (dq, *J* = 13.9, 6.9 Hz, 1H), 1.06 (d, *J* = 6.9 Hz, 3H), 1.00 (d, *J* = 7.0 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.7, 198.0, 138.2, 135.1, 131.7, 130.4, 129.2, 129.0, 128.3, 128.0, 127.9, 127.4, 48.6, 45.1, 40.7, 18.1.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{19}H_{19}BrO_2$ 359.0641, found: 359.0640.



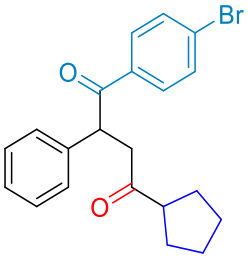
4as

5-methyl-2-phenyl-1-(m-tolyl)hexane-1,4-dione (4as)

Chromatography Pentane/EA = 20:1 (v/v), 47.0 mg (80%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 – 7.67 (m, 2H), 7.23 – 7.08 (m, 7H), 5.05 (dd, *J* = 10.1, 4.0 Hz, 1H), 3.55 (dd, *J* = 17.9, 10.1 Hz, 1H), 2.70 (dd, *J* = 17.9, 4.0 Hz, 1H), 2.60 – 2.49 (m, 1H), 2.26 (s, 3H), 1.02 (dd, *J* = 15.7, 6.9 Hz, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 212.8, 199.2, 138.7, 138.1, 136.4, 133.6, 129.3, 129.1, 128.3, 128.1, 127.2, 126.1, 48.5, 45.2, 40.8, 21.3, 18.9.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{20}H_{22}O_2$ 295.1693, found: 295.1696.

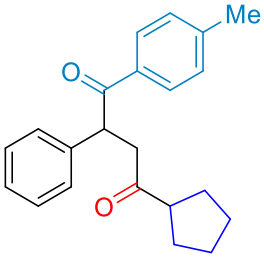


4at

1-(4-bromophenyl)-4-cyclopentyl-2-phenylbutane-1,4-dione (4at)

Chromatography Pentane/EA = 20:1 (v/v), 53.8 mg (70%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 – 7.73 (m, 2H), 7.46 – 7.10 (m, 7H), 4.98 (dd, *J* = 10.1, 3.9 Hz, 1H), 3.55 (dd, *J* = 18.0, 10.1 Hz, 1H), 2.88 – 2.78 (m, 1H), 2.71 (dd, *J* = 18.0, 3.9 Hz, 1H), 1.79 – 1.48 (m, 8H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 211.2, 198.1, 138.2, 135.2, 131.7, 130.4, 129.2, 128.0, 127.9, 127.4, 51.2, 48.6, 46.4, 28.8, 28.7, 26.0, 25.9.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{21}H_{21}BrO_2$ 407.0617, found: 407.0620.

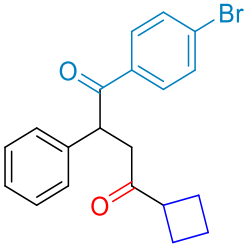


4au

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

Chromatography Pentane/EA = 20:1 (v/v), 45.6 mg (71%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.82 – 7.78 (m, 2H), 7.21 – 7.07 (m, 7H), 5.05 (dd, *J* = 10.0, 4.1 Hz, 1H), 3.54 (dd, *J* = 17.9, 9.9 Hz, 1H), 2.87 – 2.77 (m, 1H), 2.70 (dd, *J* = 17.9, 4.1 Hz, 1H), 2.26 (s, 3H), 1.80 – 1.45 (m, 8H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 211.2, 198.6, 143.6, 138.9, 133.8, 129.1, 129.0, 128.9, 128.1, 127.1, 51.3, 48.4, 46.4, 28.7, 28.7, 25.9, 25.9, 21.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{22}H_{24}O_2$ 343.1668, found: 343.1669.

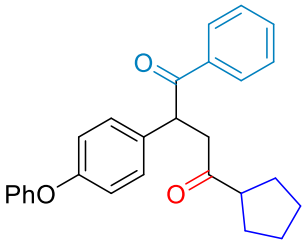


4av

1-(4-bromophenyl)-4-cyclobutyl-2-phenylbutane-1,4-dione (4av)

Chromatography Pentane/EA = 20:1 (v/v), 26.0 mg (35%), white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.74 (m, 2H), 7.47 – 7.13 (m, 7H), 4.98 (dd, *J* = 10.1, 4.0 Hz, 1H), 3.43 (dd, *J* = 17.9, 10.1 Hz, 1H), 3.27 – 73.16 (m, 1H), 2.61 (dd, *J* = 18.0, 4.0 Hz, 1H), 2.30 - 1.72 (m, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 209.8, 198.0, 138.2, 135.1, 131.8, 130.4, 129.2, 128.0, 127.4, 48.5, 45.2, 44.6, 24.3, 24.1, 17.8.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{20}H_{19}BrO_2$ 393.0461, found: 393.0460.

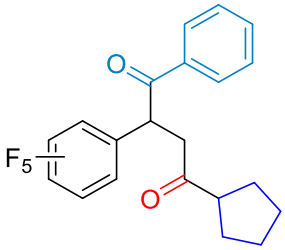


4aw

4-cyclopentyl-2-(4-phenoxyphenyl)-1-phenylbutane-1,4-dione (4aw)

Chromatography Pentane/EA = 20:1 (v/v), 55.8 mg (70%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.93 – 7.89 (m, 2H), 7.44 – 6.81 (m, 12H), 5.05 (dd, *J* = 9.9, 4.1 Hz, 1H), 3.54 (dd, *J* = 17.9, 9.9 Hz, 1H), 2.88 – 2.78 (m, 1H), 2.73 (dd, *J* = 17.9, 4.2 Hz, 1H), 1.82 – 1.45 (m, 8H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 211.2, 199.1, 156.7, 156.5, 136.4, 133.1, 132.9, 129.7, 129.4, 128.9, 128.5, 123.5, 119.1, 119.1, 51.2, 47.7, 46.5, 30.9, 28.7, 25.9, 25.9.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{27}H_{26}O_3$ 399.1955, found: 399.1954.

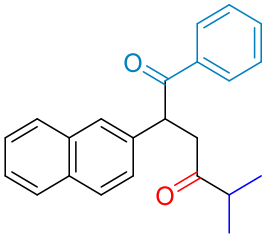


4ax

4-cyclopentyl-2-(perfluorophenyl)-1-phenylbutane-1,4-dione (4ax)

Chromatography Pentane/EA = 20:1 (v/v), 50.0 mg (60%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80 – 7.76 (m, 2H), 7.48 – 7.19 (m, 3H), 5.35 (dd, *J* = 8.8, 4.8 Hz, 1H), 3.60 (dd, *J* = 17.8, 8.8 Hz, 1H), 2.99 – 2.87 (m, 1H), 2.71 – 2.57 (m, 1H), 1.86 – 1.18 (m, 8H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 209.6, 195.2, 135.3, 133.5, 128.8, 128.2, 128.1, 51.4, 41.5, 38.4, 28.9, 28.8, 25.9, 25.9.
^{19}F NMR (282 MHz, $CDCl_3$) δ -136.69 – -146.03 (m, 2F), -154.21 – -157.65 (m, 1F), -160.77 – -162.88 (m, 2F).
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{21}H_{17}F_5O_2$ 439.1667, found: 439.1663.

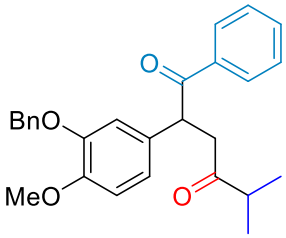


4ay

5-methyl-2-(naphthalen-2-yl)-1-phenylhexane-1,4-dione (4ay)

Chromatography Pentane/EA = 20:1 (v/v), 34.4 mg (52%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.72 – 7.25 (m, 10H), 5.22 (dd, *J* = 9.9, 4.0 Hz, 1H), 3.65 (dd, *J* = 17.9, 10.0 Hz, 1H), 2.79 (dd, *J* = 17.9, 4.1 Hz, 1H), 2.65 – 2.51 (m, 10H), 1.04 (dd, *J* = 19.1, 6.9 Hz, 6H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.7, 198.9, 136.4, 136.1, 133.6, 132.9, 132.4, 128.9, 128.9, 128.5, 127.7, 127.6, 126.9, 126.3, 126.0, 48.7, 45.2, 40.8, 18.1.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{22}O_2$ 331.1693, found: 331.1693.

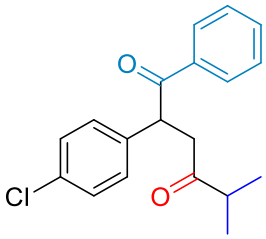


4az

2-(3-(benzyloxy)-4-methoxyphenyl)-5-methyl-1-phenylhexane-1,4-dione (4az)

Chromatography Pentane/EA = 20:1 (v/v), 48.4 mg (58%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.06 – 6.68 (m, 8H), 5.00 (s, 2H), 4.99 – 4.93 (m, 1H), 3.76 (s, 3H), 3.52 (dd, *J* = 17.9, 9.9 Hz, 1H), 2.70 (dd, *J* = 17.9, 4.1 Hz, 1H), 2.73 – 2.51 (m, 1H), 1.02 (dd, *J* = 15.0, 6.9 Hz, 6H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.95, 199.19, 149.97, 147.41, 136.99, 136.46, 133.70, 132.79, 131.51, 130.16, 128.80, 128.50, 128.46, 128.40, 127.81, 127.20, 120.46, 114.24, 111.35, 70.94, 56.02, 48.13, 45.20, 40.81, 18.09.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{27}H_{28}O_4$ 417.2060, found: 417.2061.

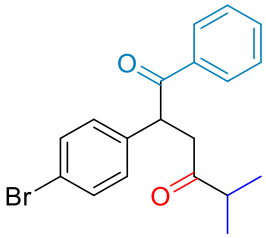


4ba

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

Chromatography Pentane/EA = 20:1 (v/v), 50.4 mg (80%), white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 – 7.86 (m, 2H), 7.43 – 7.12 (m, 7H), 5.04 (dd, *J* = 9.8, 4.3 Hz, 1H), 3.52 (dd, *J* = 17.9, 9.8 Hz, 1H), 2.70 (dd, *J* = 17.9, 4.2 Hz, 1H), 2.61 – 2.51 (m, 1H), 1.05 (d, *J* = 6.9 Hz, 3H), 1.00 (d, *J* = 7.0 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 212.5, 198.7, 137.1, 136.1, 133.2, 133.0, 129.5, 129.3, 128.8, 128.5, 47.8, 44.9, 40.8, 181.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{19}H_{19}ClO_2$ 337.0966, found: 337.0967.

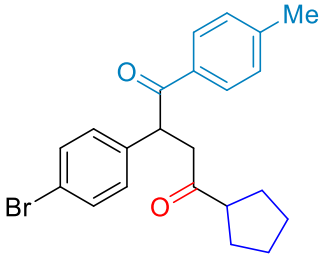


4bb

2-(4-bromophenyl)-5-methyl-1-phenylhexane-1,4-dione (4bb)

Chromatography Pentane/EA = 20:1 (v/v), 59.0 mg (82%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.89 – 7.85 (m, 2H), 7.44 – 7.06 (m, 7H), 5.03 (dd, *J* = 9.8, 4.2 Hz, 1H), 3.52 (dd, *J* = 17.9, 9.8 Hz, 1H), 2.70 (dd, *J* = 17.9, 4.2 Hz, 1H), 2.74 – 2.49 (m, 1H), 1.03 (dd, *J* = 15.2, 6.9 Hz, 6H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.4, 198.6, 137.6, 136.1, 133.0, 132.2, 129.8, 128.8, 128.5, 121.3, 47.8, 44.9, 40.8, 18.1.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{19}H_{19}BrO_2$ 381.0461, found: 381.0466.

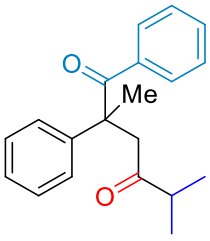


4bc

2-(4-bromophenyl)-4-cyclopentyl-1-(p-tolyl)butane-1,4-dione (4bc)

Chromatography Pentane/EA = 20:1 (v/v), 54.8 mg (70%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79 – 7.75 (m, 2H), 7.34 – 7.05 (m, 6H), 5.02 (dd, *J* = 9.6, 4.3 Hz, 1H), 3.49 (dd, *J* = 17.9, 9.7 Hz, 1H), 2.82 (p, *J* = 7.9 Hz, 1H), 2.69 (dd, *J* = 17.9, 4.3 Hz, 1H), 2.27 (s, 3H), 1.78 – 1.18 (m, 8H),
^{13}C NMR (75 MHz, $CDCl_3$) δ 210.9, 198.2, 143.9, 137.9, 133.6, 132.2, 129.8, 129.2, 128.9, 121.2, 51.3, 47.7, 46.1, 28.7, 28.7, 25.9, 25.9, 21.6.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{22}H_{23}BrO_2$ 399.0954, found: 399.0958.

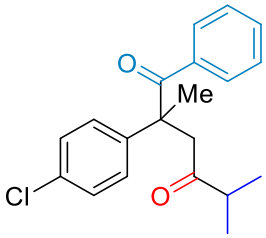


4bd

2,5-dimethyl-1,2-diphenylhexane-1,4-dione (4bd)

Chromatography Pentane/EA = 20:1 (v/v), 51.2 mg (87%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 – 7.11 (m, 10H), 3.32 (dd, *J* = 17.2, 0.8 Hz, 1H), 2.93 (d, *J* = 17.2 Hz, 1H), 2.43 – 2.33 (m, 1H), 1.30 – 0.87 (m, 6H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 212.1, 203.2, 142.9, 137.4, 131.2, 129.1, 128.9, 127.8, 127.2, 126.1, 53.1, 51.0, 41.4, 24.1, 18.1, 17.9.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{20}H_{22}O_2$ 317.1512, found: 317.1505.

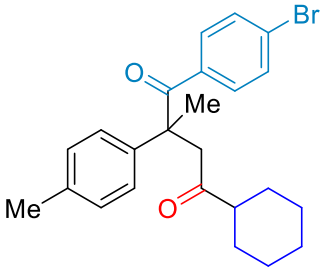


4be

2-(4-chlorophenyl)-2,5-dimethyl-1-phenylhexane-1,4-dione (4be)

Chromatography Pentane/EA = 20:1 (v/v), 51.8 mg (79%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 – 7.13 (m, 9H), 3.29 (dd, *J* = 17.4, 0.8 Hz, 1H), 2.92 (d, *J* = 17.4 Hz, 1H), 2.45 – 2.36 (m, 1H), 1.70 (s, 3H), 0.90 (dd, *J* = 6.9, 3.5 Hz, 6H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 211.7, 202.8, 141.6, 137.1, 133.2, 131.4, 129.2, 128.9, 127.9, 127.6, 52.7, 50.9, 41.3, 24.1, 18.1, 17.9.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{20}H_{21}ClO_2$ 329.1303, found: 329.1308.

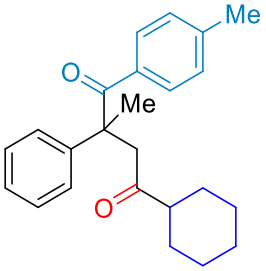


4bf

1-(4-bromophenyl)-4-cyclohexyl-2-methyl-2-(p-tolyl)butane-1,4-dione (4bf)

Chromatography Pentane/EA = 20:1 (v/v), 51.2 mg (60%), white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.02 (m, 8H), 3.26 (d, *J* = 17.7 Hz, 1H), 2.91 (d, *J* = 17.3 Hz, 1H), 2.28 (s, 3H), 2.16 – 1.05 (m, 14H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 211.4, 202.3, 139.6, 137.1, 136.3, 131.1, 130.6, 129.9, 125.9, 125.9, 52.6, 51.4, 51.1, 28.3, 28.2, 25.8, 25.6, 24.2, 20.9.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{24}H_{27}BrO_2$ 427.1267, found: 427.1269.

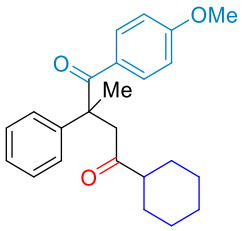


4bg

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

Chromatography Pentane/EA = 20:1 (v/v), 34.8 mg (50%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 – 6.91 (m, 9H), 3.40 – 3.08 (m, 1H), 2.90 (d, *J* = 16.9 Hz, 1H), 2.21 (s, 3H), 2.13 – 1.07 (m, 14H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 211.6, 202.5, 143.4, 141.8, 134.3, 129.3, 129.0, 128.5, 127.1, 126.1, 53.3, 51.5, 51.3, 28.3, 28.1, 25.8, 25.6, 25.6, 24.0, 21.4.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{22}H_{24}O_2$ 371.1982, found: 371.1987.

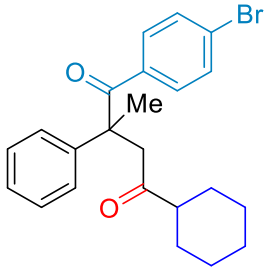


4bh

4-cyclohexyl-1-(4-methoxyphenyl)-2-methyl-2-phenylbutane-1,4-dione (4bh)

Chromatography Pentane/EA = 20:1 (v/v), 47.4 mg (65%), white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 6.70 (m, 9H), 3.78 (s, 3H), 3.30 (d, *J* = 16.8 Hz, 1H), 2.99 (d, *J* = 16.8 Hz, 1H), 2.21.34 – 1.13 (m, 14H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 211.8, 201.1, 162.1, 143.7, 131.7, 129.3, 129.1, 127.1, 126.1, 113.1, 55.3, 53.4, 51.7, 51.4, 28.4, 28.2, 25.8, 25.7, 25.6, 24.1.
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{24}H_{28}O_3$ 387.1931, found: 387.1938.

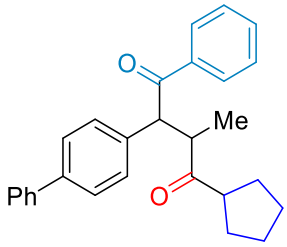


4bi

1-(4-bromophenyl)-4-cyclohexyl-2-methyl-2-phenylbutane-1,4-dione (4bi)

Chromatography Pentane/EA = 20:1 (v/v), 62.0 mg (75%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34 – 7.12 (m, 9H), 3.28 (dd, *J* = 17.2, 0.8 Hz, 1H), 2.93 (d, *J* = 17.2 Hz, 1H), 2.16 – 1.09 (m, 14H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 211.3, 202.1, 162.2, 142.7, 136.2, 131.1, 130.5, 129.2, 127.4, 126.0, 53.0, 51.4, 51.1, 31.5, 31.4, 30.2, 30.1, 28.3, 28.2, 25.8, 25.6, 25.6, 24.1.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{23}H_{25}BrO_2$ 413.1111, found: 413.1115.

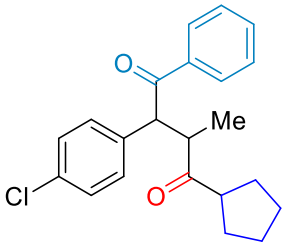


4bj

2-([1,1'-biphenyl]-4-yl)-4-cyclopentyl-3-methyl-1-phenylbutane-1,4-dione (4bj)

Chromatography Pentane/EA = 20:1 (v/v), 31.0 mg (39%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98 – 7.19 (m, 9H), 4.89 (d, *J* = 10.6 Hz, 0.7H), 4.81 (d, *J* = 10.6 Hz, 0.3H), 3.72 – 0.93 (m, 13H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 216. 9(minor), 215.9, 199.9(minor), 198.8, 140.4, 140.4(minor), 140.2(minor), 136.9, 136.1(minor), 133.2, 132.7, 129.4, 129.3(minor), 128.7, 128.6(minor), 128.4, 127.6, 127.4(minor), 127.3, 126.9, 55.9(minor), 55.4, 52.0, 50.1, 49.5(minor), 49.4, 30.2(minor), 28.7(minor), 28.1, 27.9(minor), 26.1, 25.9(minor), 25.8, 25.5(minor), 16.9, 15.2(minor).
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{28}H_{28}O_2$ 397.2162, found: 397.2162.

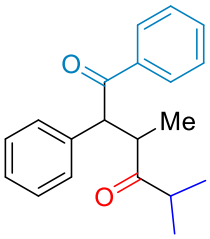


4bk

2-(4-chlorophenyl)-4-cyclopentyl-3-methyl-1-phenylbutane-1,4-dione (4bk)

Chromatography Pentane/EA = 20:1 (v/v), 29.8 mg (42%), white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 – 7.12 (m, 9H), 4.80 (dd, *J* = 30.4, 10.6 Hz, 1H), 3.64 – 0.88 (m, 13H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 216.6(minor), 215.1, 199.7(minor), 198.6, 136.7, 136.5(minor), 135.7, 135.5(minor), 133.4, 132.9(minor), 130.4, 130.2(minor), 129.3, 129.2(minor), 129.0, 128.9, 128.7, 128.6, 128.5, 128.5(minor), 55.5(minor), 54.7, 51.8, 50.0(minor), 49.4, 49.3(minor), 30.9, 30.2(minor), 28.7, 28.2(minor), 27.9, 26.1(minor), 25.9(minor), 25.8, 25.6, 16.8, 15.0(minor).
HRMS(ESI-TOF) m/z: calcd for $[M^+]Na^+$ $C_{22}H_{23}ClO_2$ 377.1279, found: 377.1276.

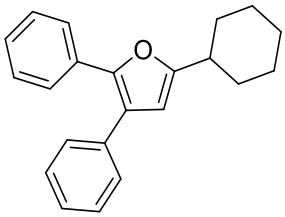


4bl

3,5-dimethyl-1,2-diphenylhexane-1,4-dione (4bl)

Chromatography Pentane/EA = 20:1 (v/v), 30.0 mg (35%), white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.06 (m, 10H), 4.81 (d, *J* = 10.6 Hz, 0.7H), 4.76 (d, *J* = 10.6 Hz, 0.3H), 3.74 – 0.46 (d, *J* = 6.9 Hz, 11H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 218.1(minor), 217.1, 199.9(minor), 198.7, 137.0, 136.9(minor), 136.9, 136.6(minor), 133.1, 132.7(minor), 129.9, 129.4(minor), 129.1, 129.0(minor), 128.9, 128.9(minor), 128.8, 128.7(minor), 128.6, 128.5(minor), 128.4, 128.4(minor), 127.4, 127.4(minor), 56.4(minor), 56.0, 48.3, 41.7, 39.5(minor), 19.2(minor), 18.9(minor), 18.2, 17.6(minor), 16.9, 16.7, 15.2(minor).
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{20}H_{22}O_2$ 295.1693, found: 295.1696.

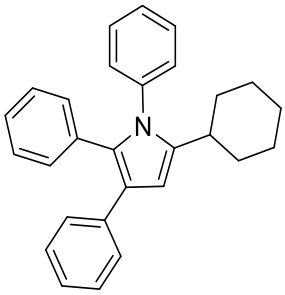


5-cyclohexyl-2,3-diphenylfuran (6a)

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

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 – 7.07 (m, 10H), 6.04 (s, 1H), 2.66 – 1.17 (m, 11H).

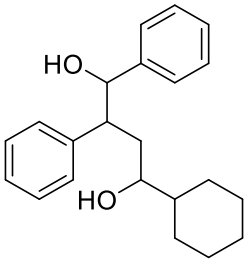
^{13}C NMR (75 MHz, $CDCl_3$) δ 160.2, 146.4, 135.0, 131.8, 128.7, 128.7, 128.4, 127.1, 127.0, 126.1, 122.9, 107.5, 37.4, 31.7, 26.3, 26.1.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{22}H_{22}O$ 303.1743, found: 303.1744.



5-cyclohexyl-1,2,3-triphenyl-1H-pyrrole (6b)

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

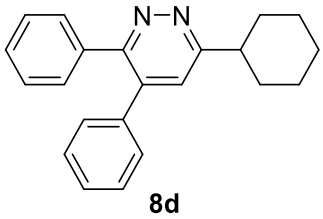
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 – 6.90 (m, 15H), 6.23 (s, 1H), 2.37 – 1.03 (m, 11H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 141.1, 138.8, 136.7, 133.0, 131.2, 129.1, 128.5, 128.1, 128.0, 127.7, 127.4, 126.3, 125.0, 122.2, 104.8, 35.7, 34.0, 26.6, 26.1.
HRMS(ESI-TOF) m/z: calcd for $[M^+]H^+$ $C_{28}H_{27}N$ 378.2216, found: 378.2220.



4-cyclohexyl-1,2-diphenylbutane-1,4-diol (6c)

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

^{1}H NMR (400 MHz, $CDCl_3$) δ 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}C NMR (101 MHz, $CDCl_3$) δ 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 $[M^+]Na^+$ $C_{22}H_{28}O_2$ 347.1981, found: 347.1989.

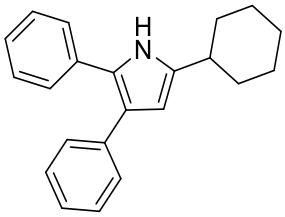


8d

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

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

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 – 7.09 (m, 11H), 3.03 – 2.93 (m, 1H), 2.08 – 1.17 (m, 10H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 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 $[M^+]H^+$ $C_{22}H_{22}N_2$ 315.1856, found: 315.1851.

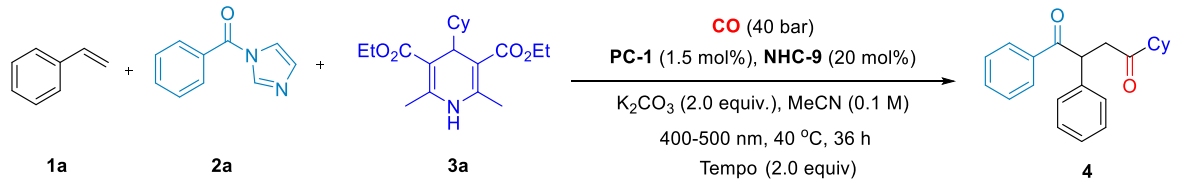


5-cyclohexyl-2,3-diphenyl-1H-pyrrole (6e)

^{1}H NMR (300 MHz, $CDCl_3$) δ 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}C NMR (75 MHz, $CDCl_3$) δ 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 $[M^+]H^+$ $C_{22}H_{23}N$ 302.1903, found: 302.1906.

5. Mechanistic investigation

5.1 Radical trapping experiment by 2,2,6,6-Tetramethylpiperidinyloxyl (TEMPO)



Scheme 1 Radical capture experiments by TEMPO

A 4 mL screw-cap vial was charged Hantzsch ester **3a** (83.0 mg, 0.25 mmol, 2.5 equiv), acyl imidazoles **2a** (34.4 mg, 0.2 mmol, 2.0 equiv), PC-1 (2.6 mg, 1.5 mol%), **NHC-9** (5.6 mg, 25 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 ℃ under UV-A (400-500 nm) for 36 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 TEMPO were added to the reaction, no products were detected, and alkyl radical was trapped by TEMPO. Data in agreement with that reported previously.[5]

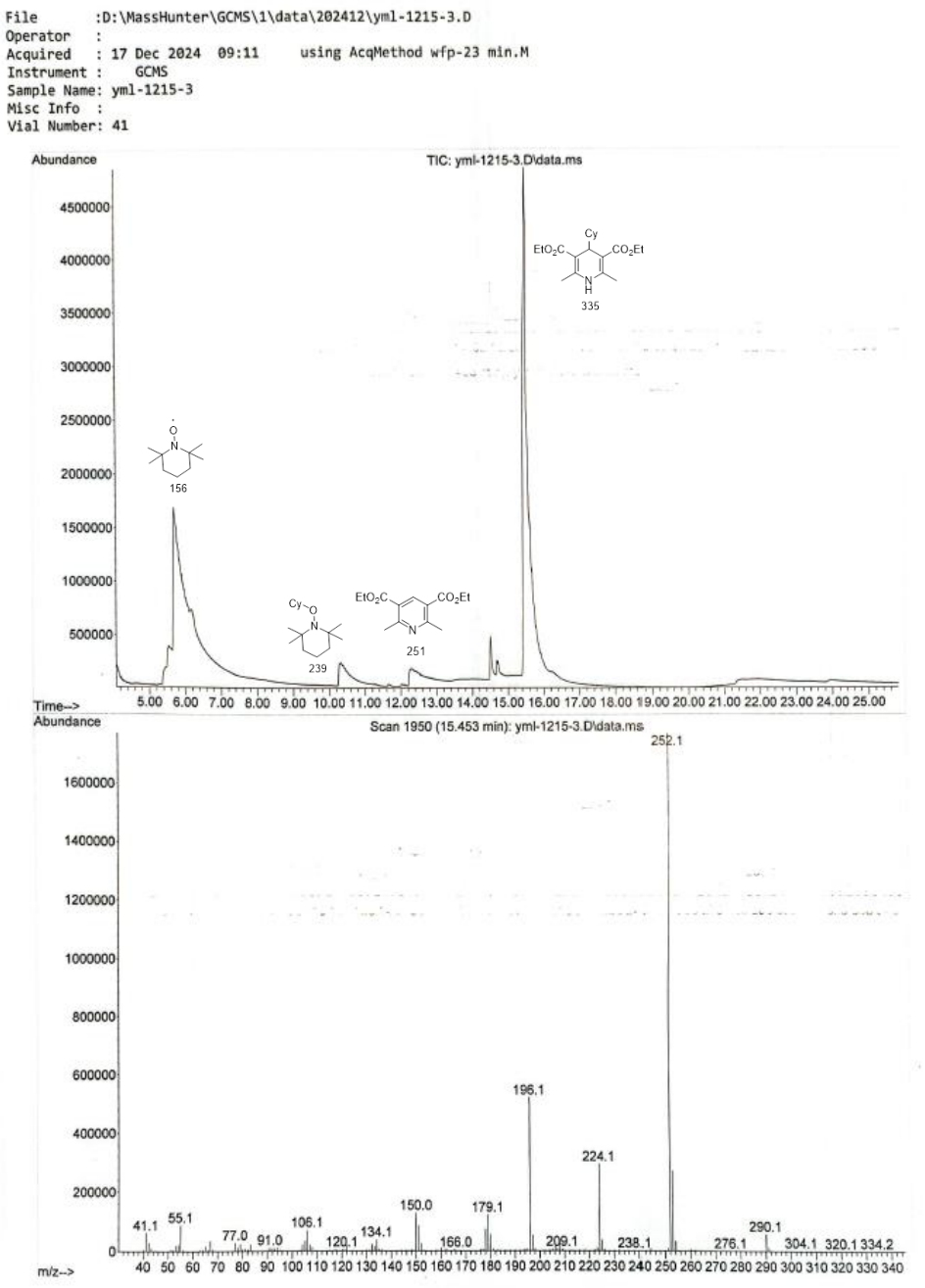


Figure 4 GC-MS of reaction system

6. References

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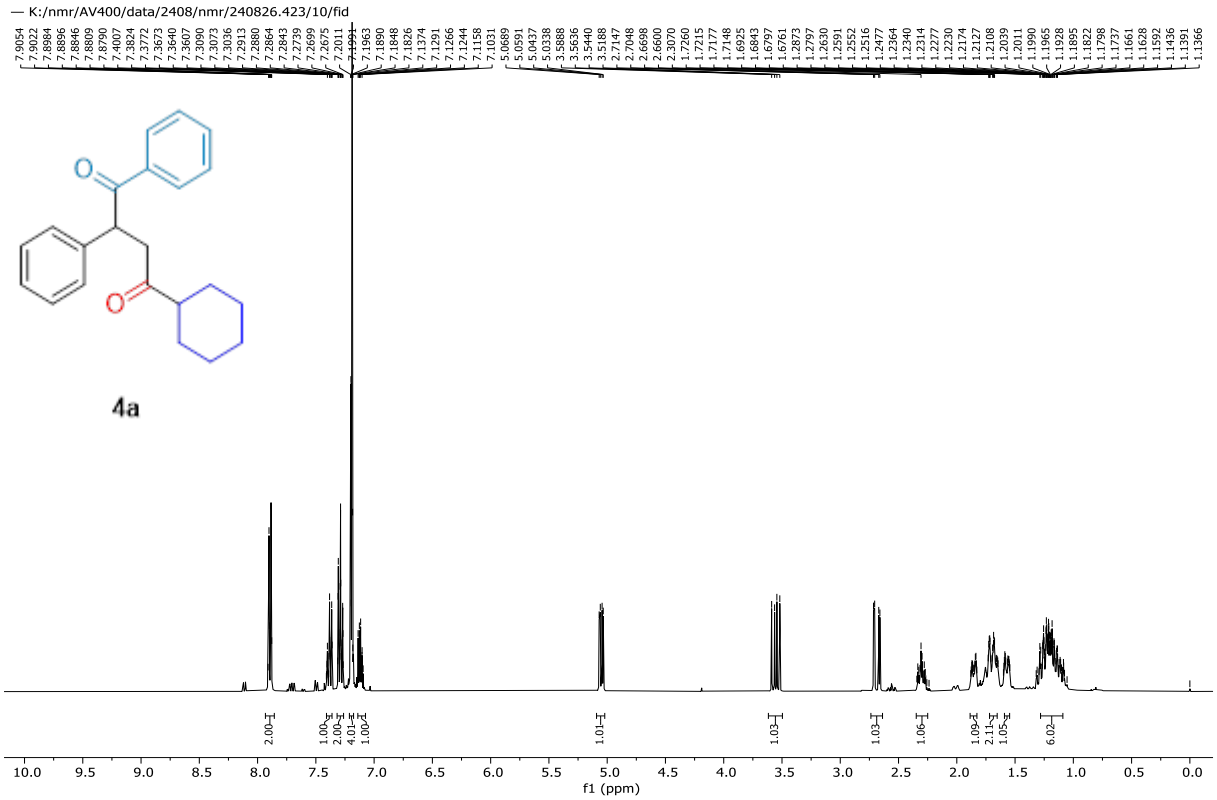
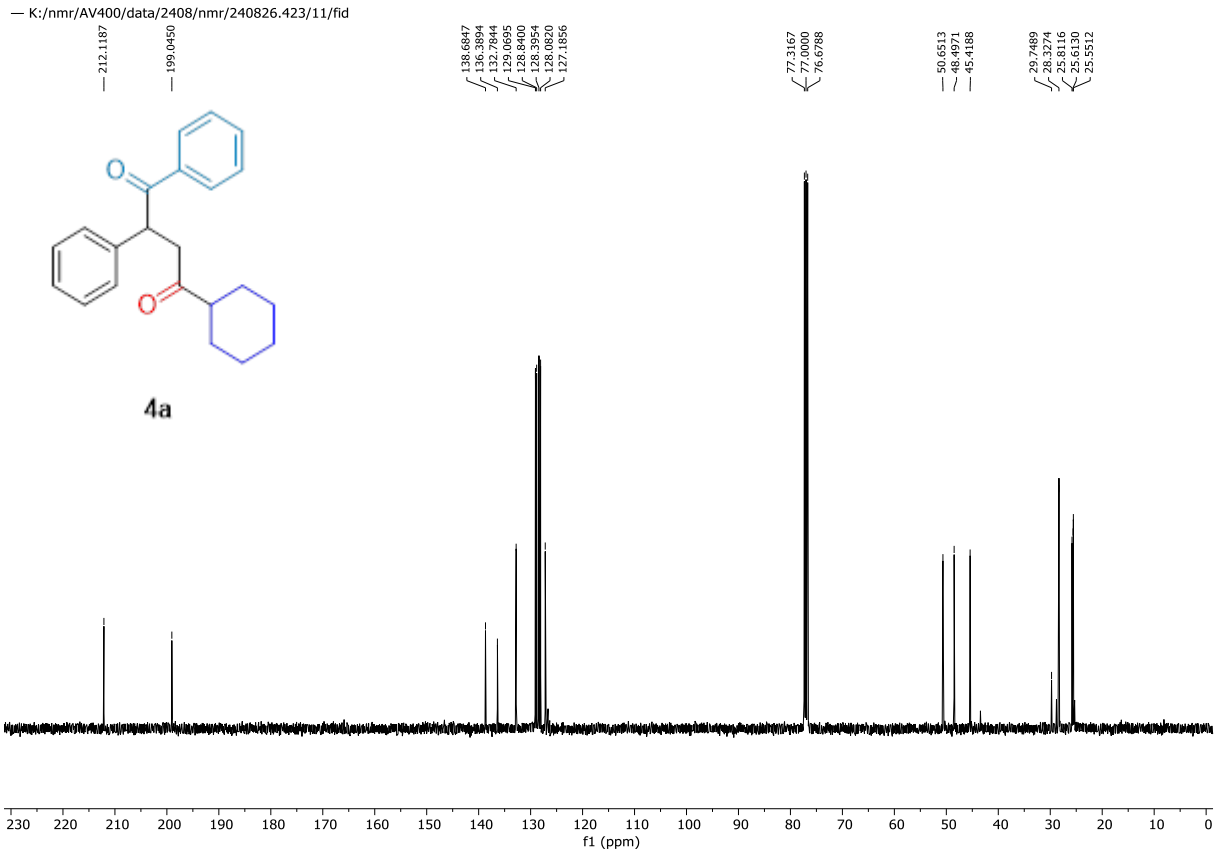
7. NMR Spectra

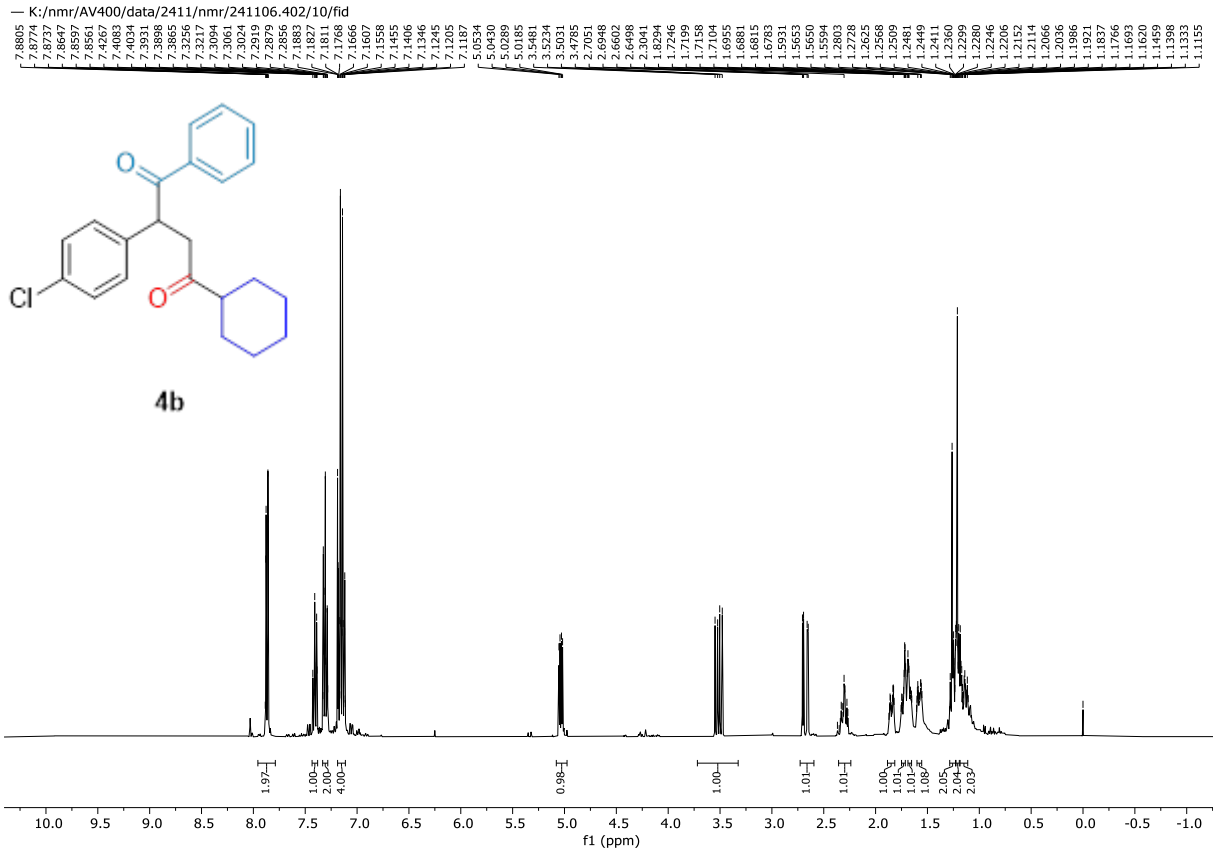
4a ^{1}H NMR (400 MHz, $CDCl_3$)



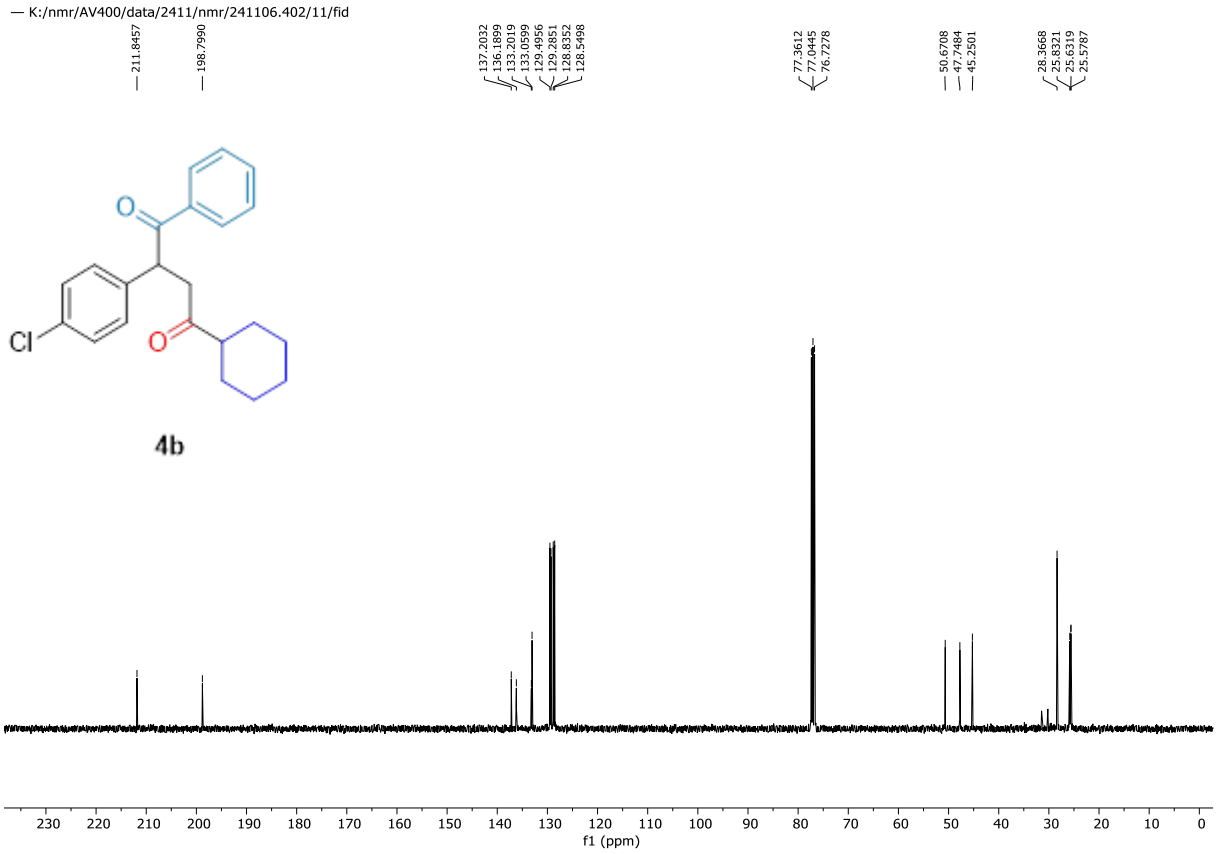
4a ^{13}C NMR (101 MHz, $CDCl_3$)



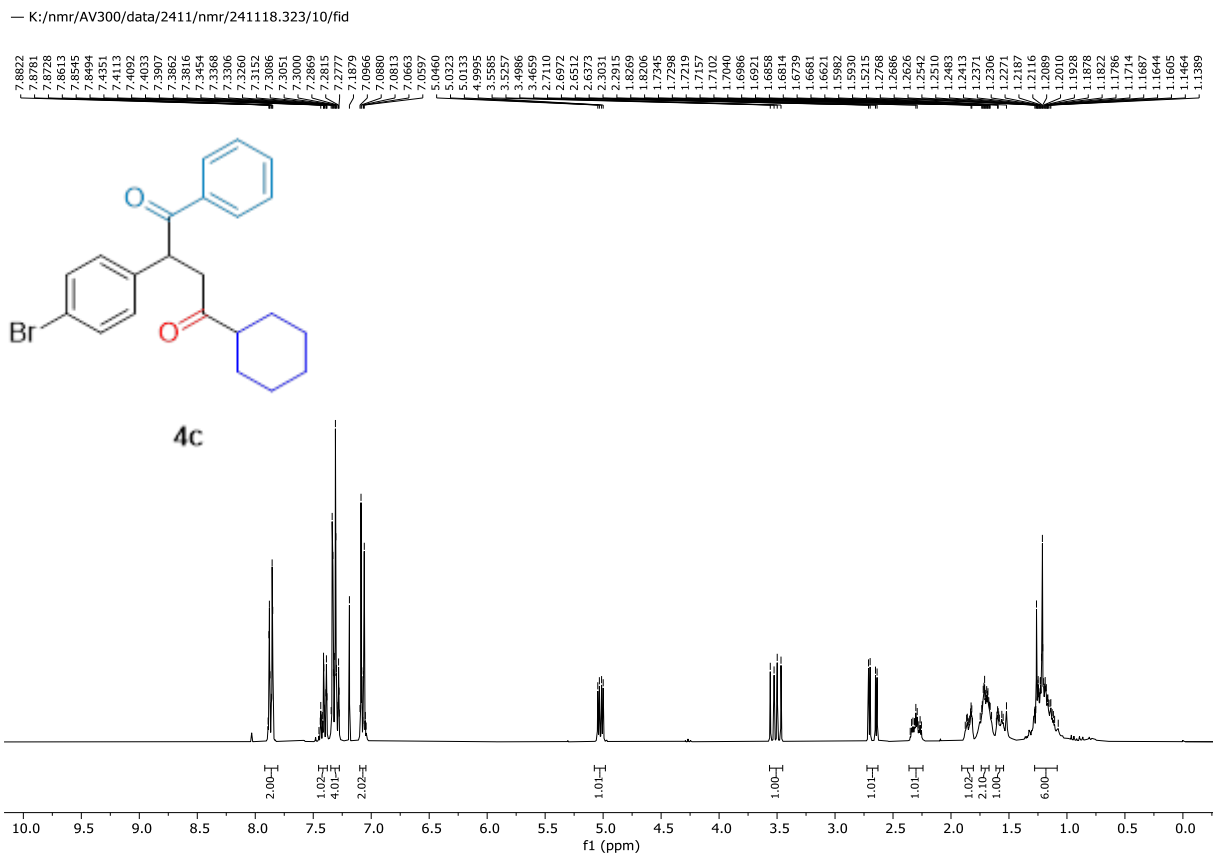
4b ^{1}H NMR (400 MHz, $CDCl_3$)



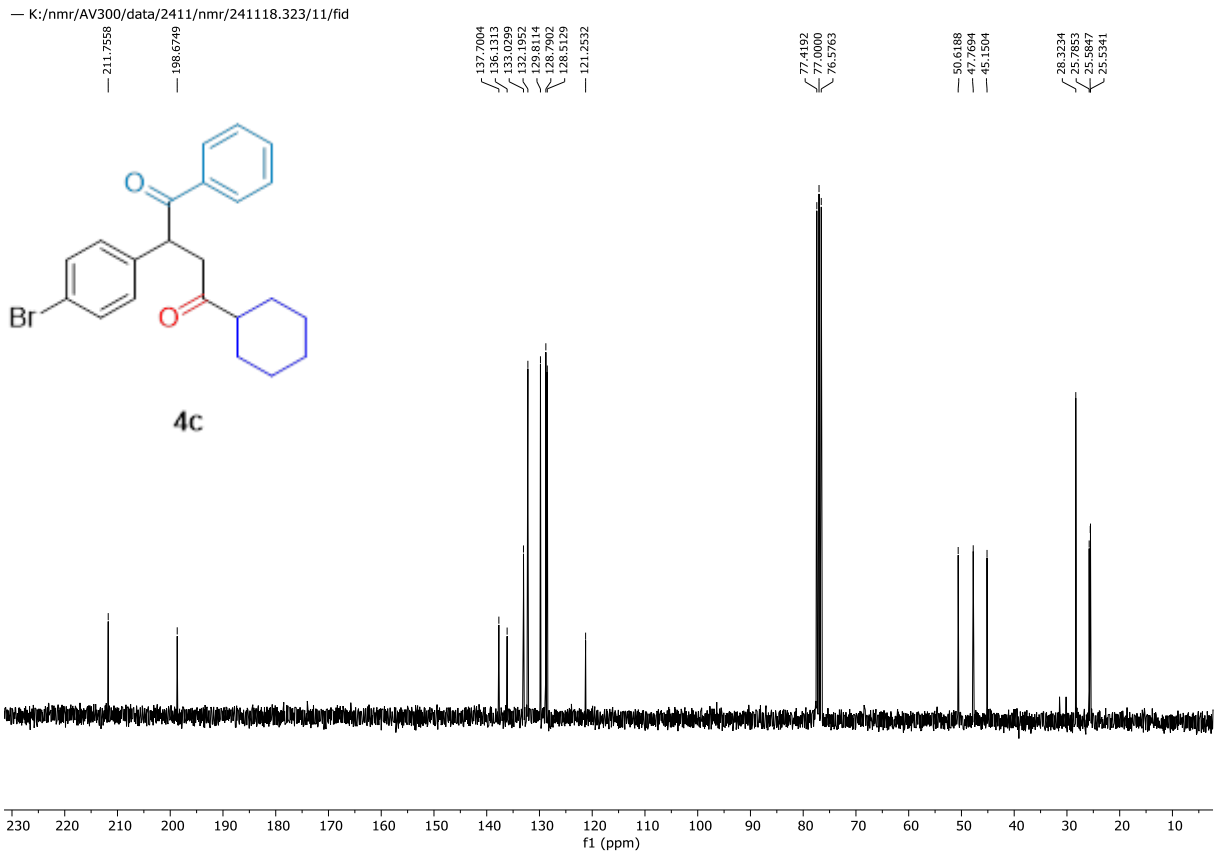
4b ^{13}C NMR (101 MHz, $CDCl_3$)



4c ^{1}H NMR (300 MHz, $CDCl_3$)



4c ^{13}C NMR (75 MHz, $CDCl_3$)



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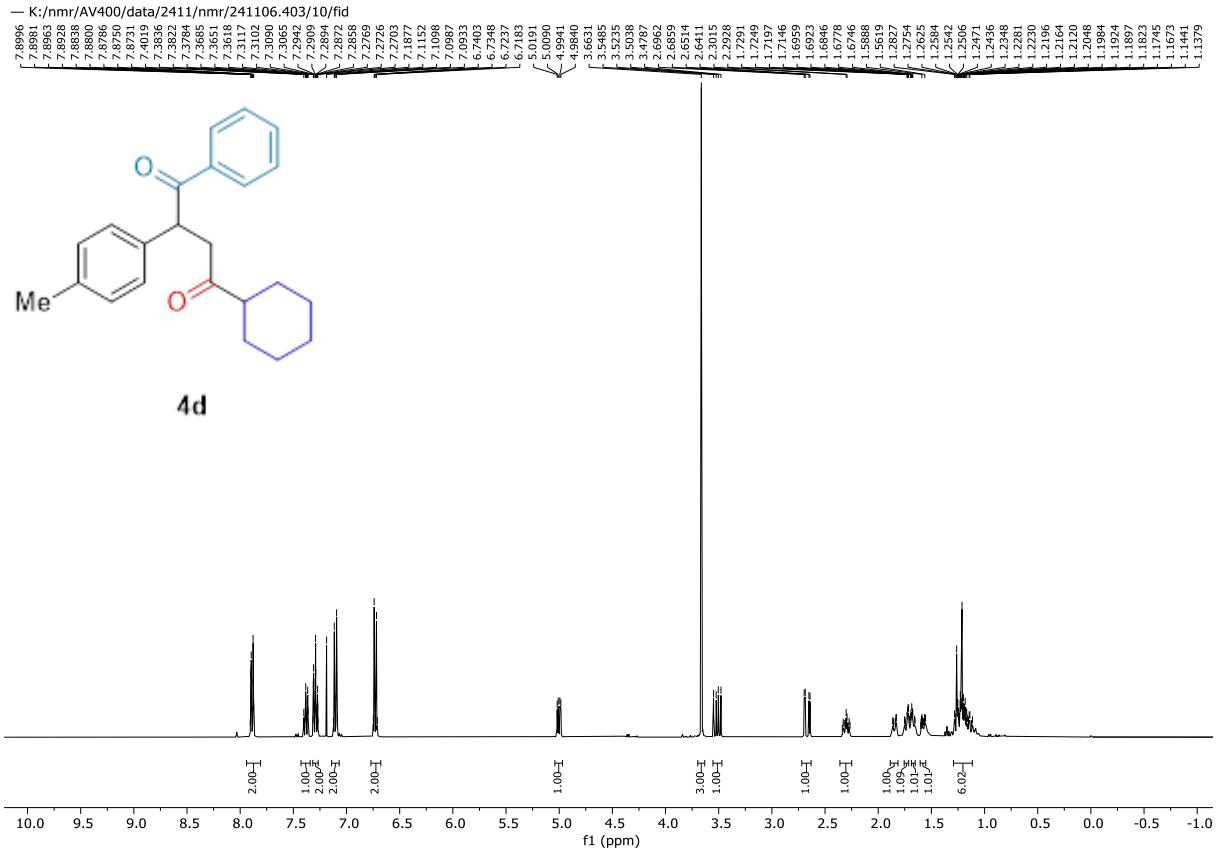
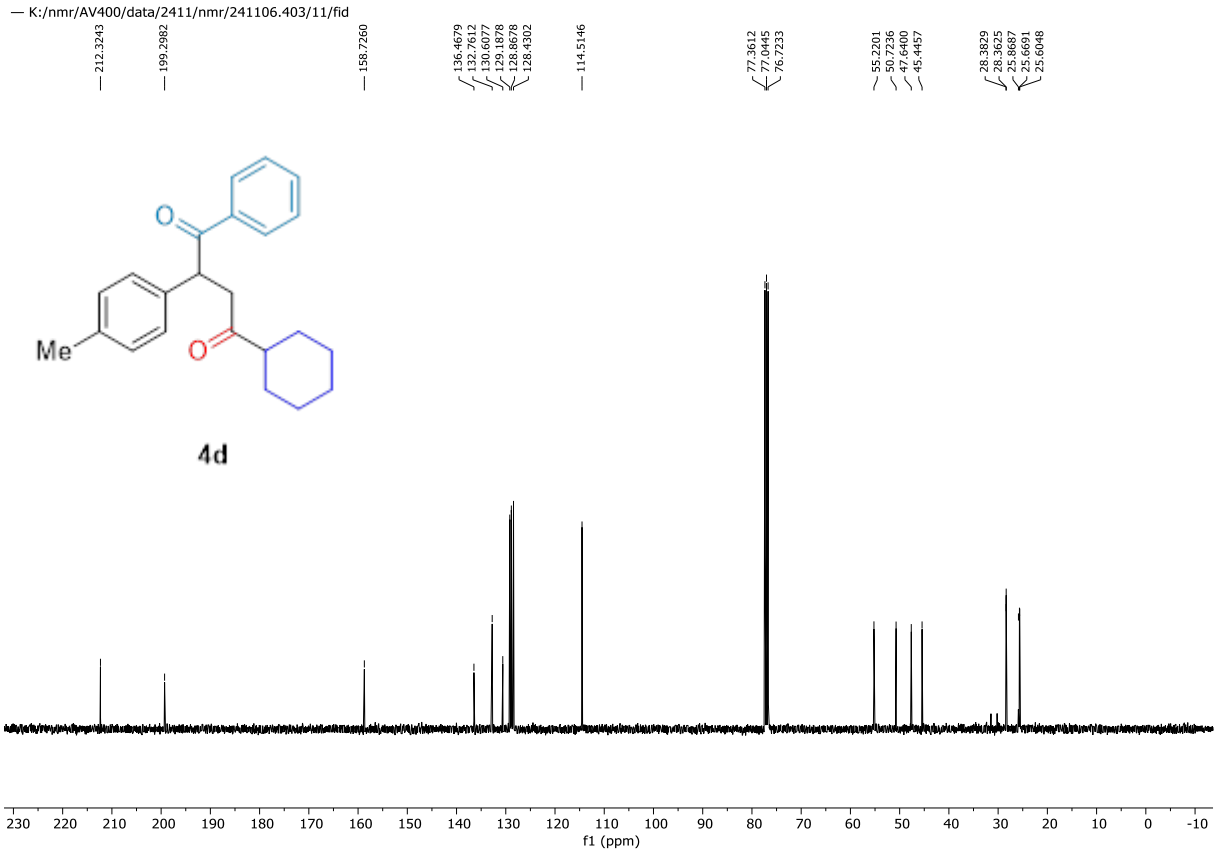
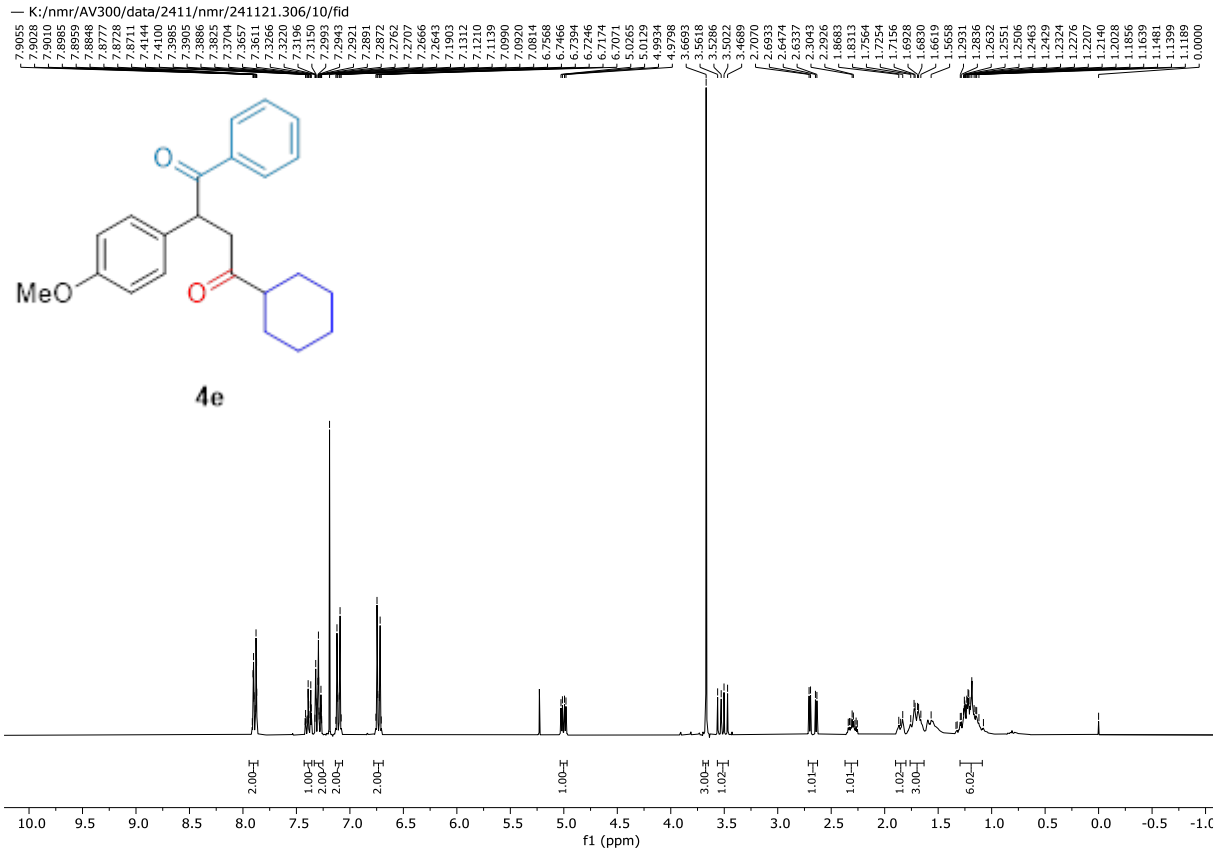


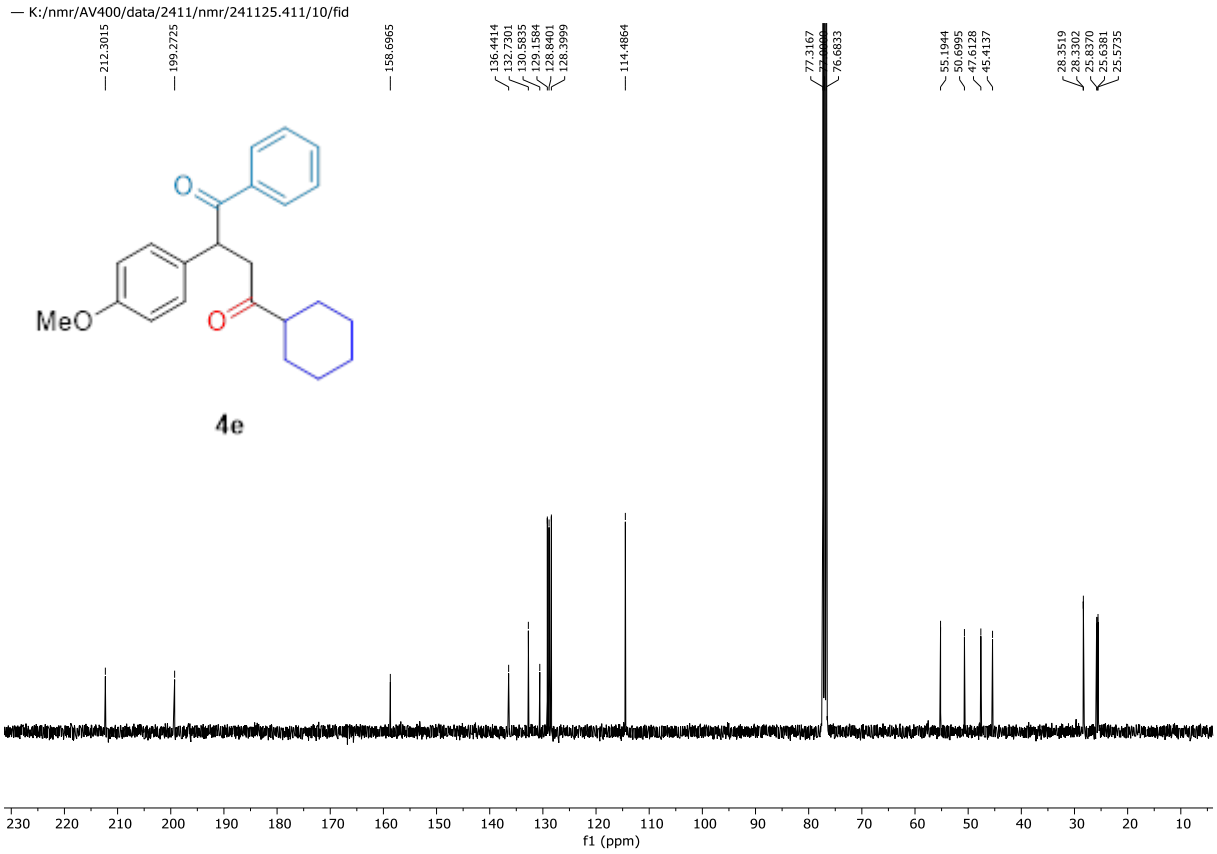
4d ^{13}C NMR (101 MHz, $CDCl_3$)



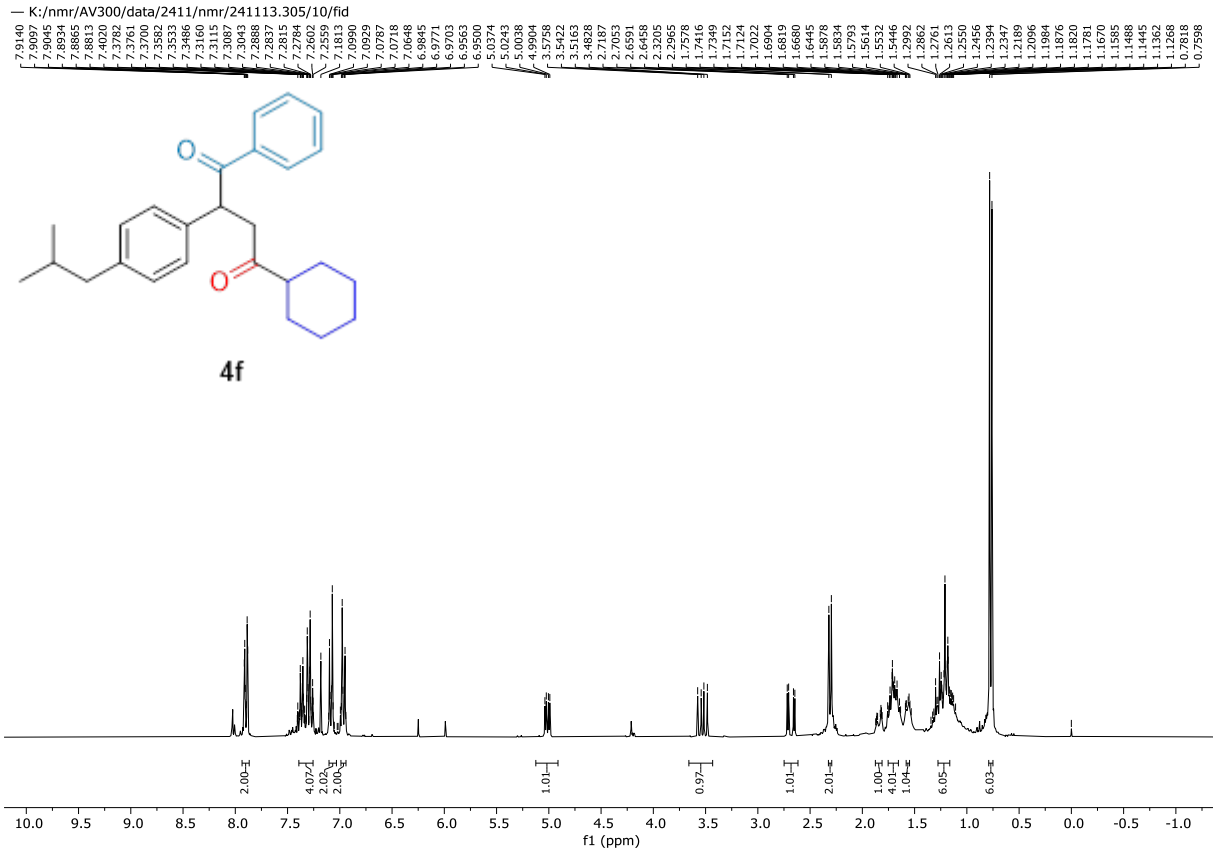
4e ^{1}H NMR (300 MHz, $CDCl_3$)



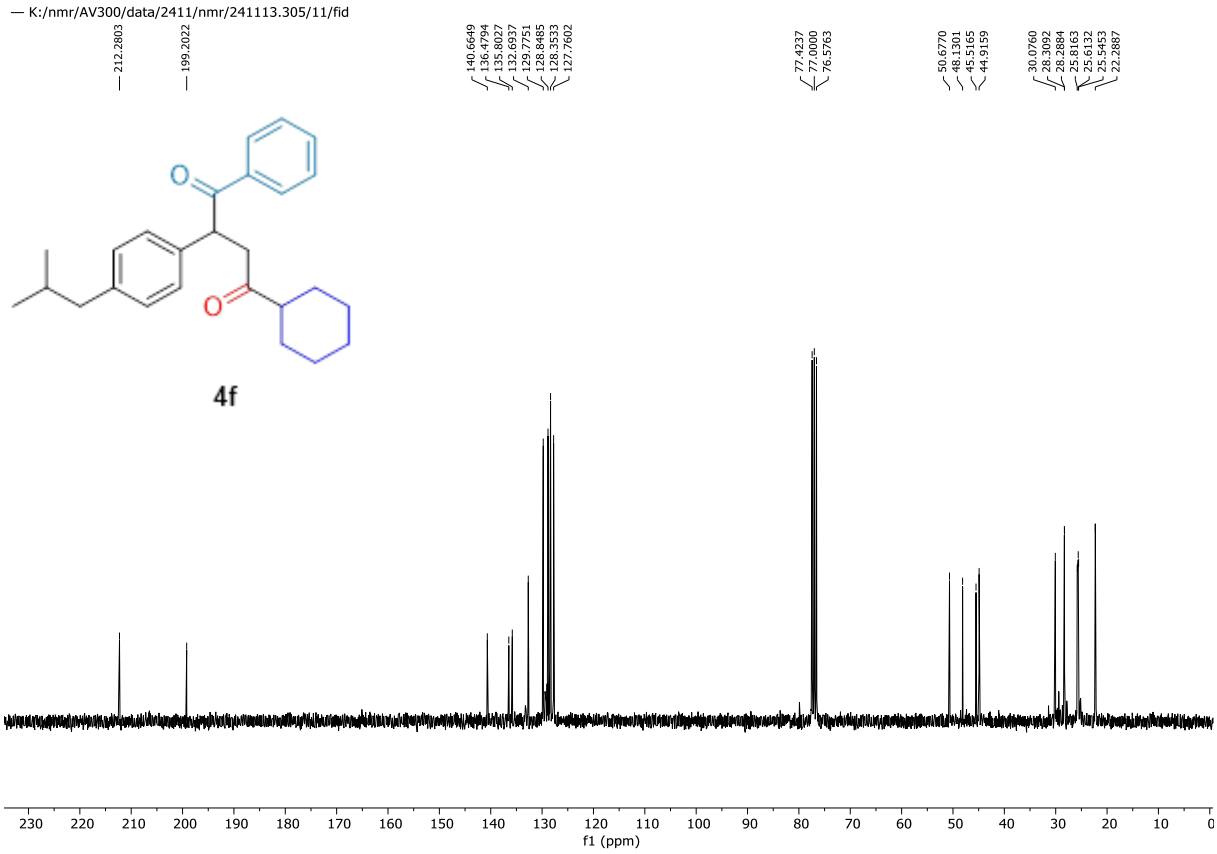
4e ^{13}C NMR (101 MHz, $CDCl_3$)



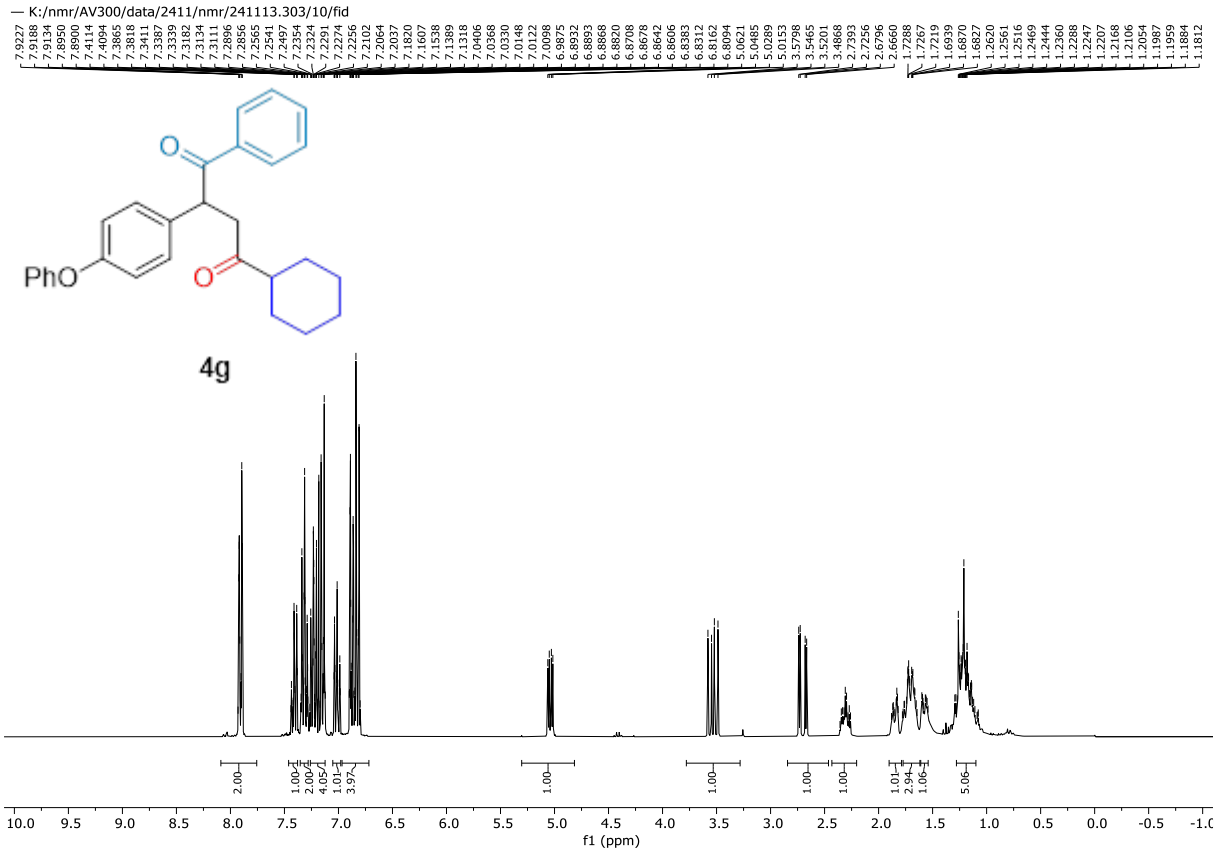
4f ^{1}H NMR (300 MHz, $CDCl_3$)



4f ^{13}C NMR (75 MHz, $CDCl_3$)



4g ^{1}H NMR (300 MHz, $CDCl_3$)



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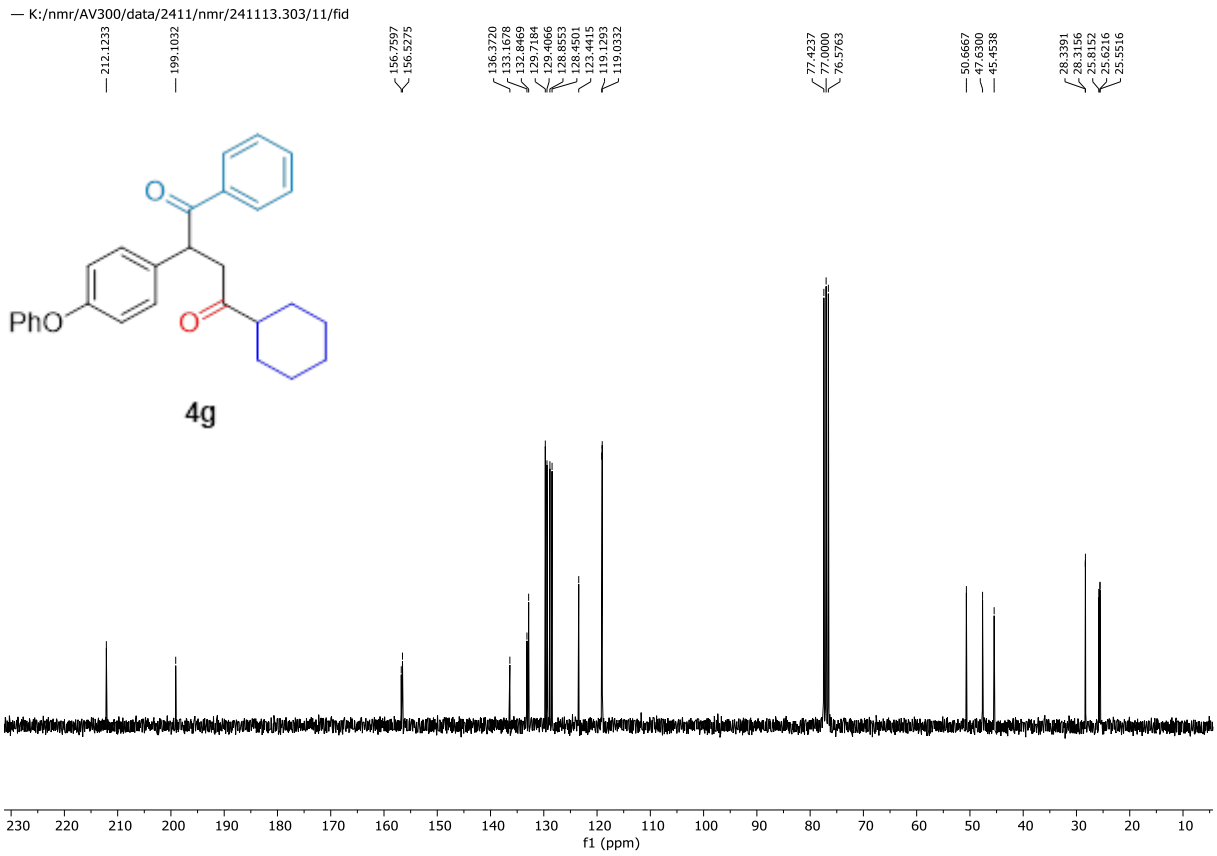
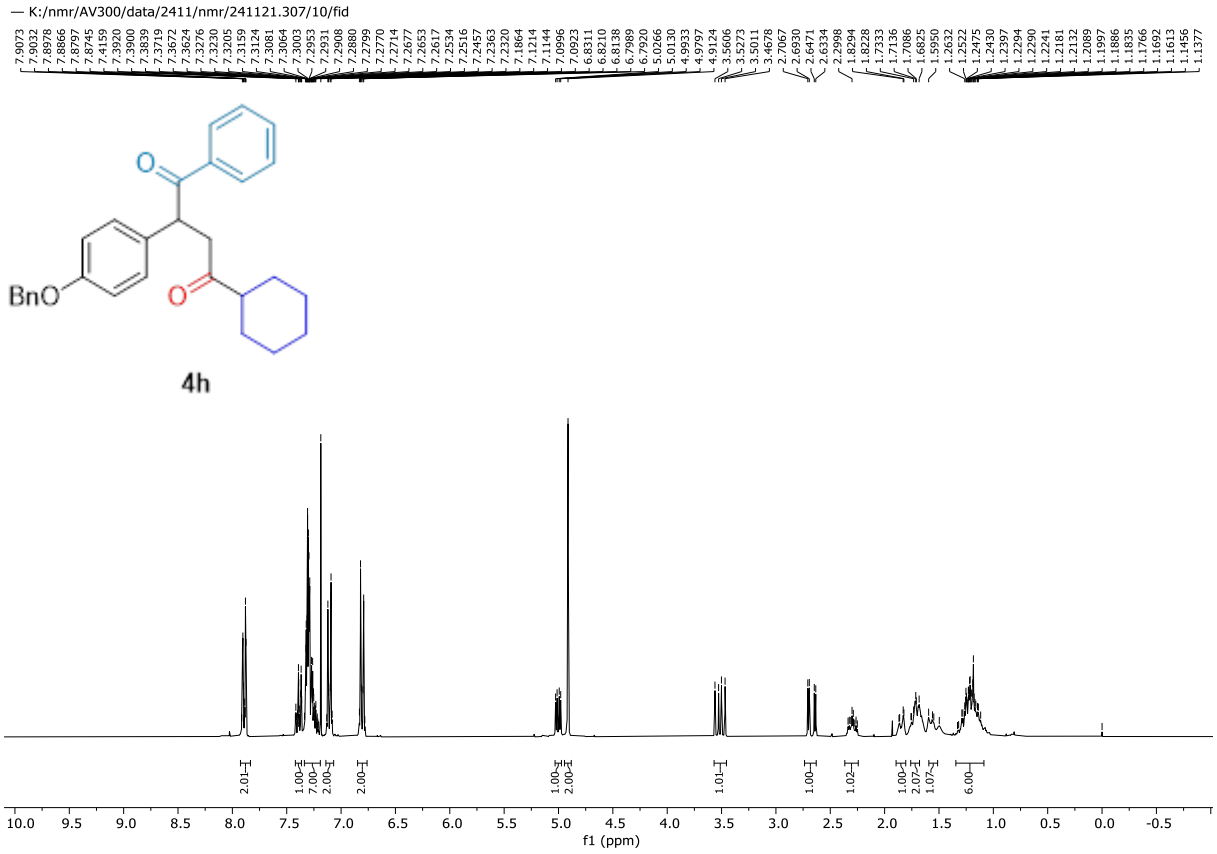
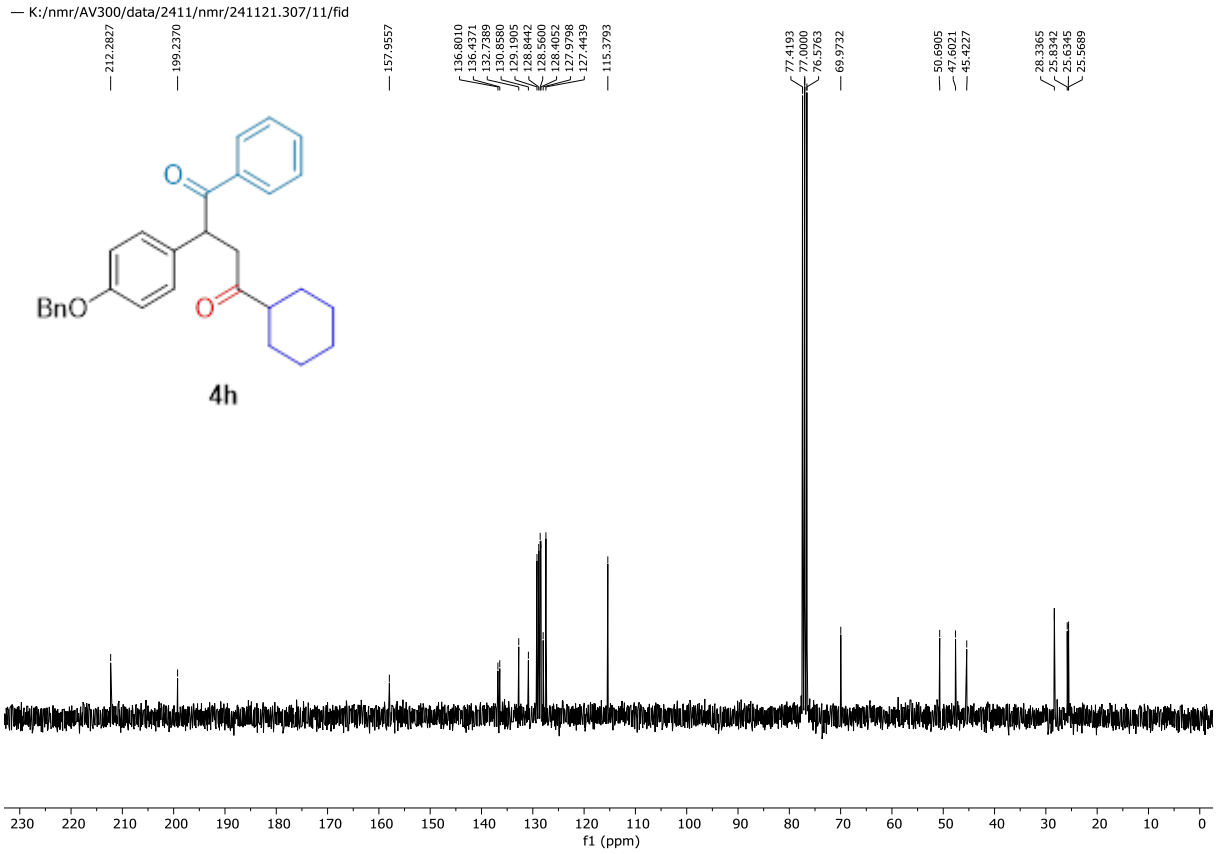


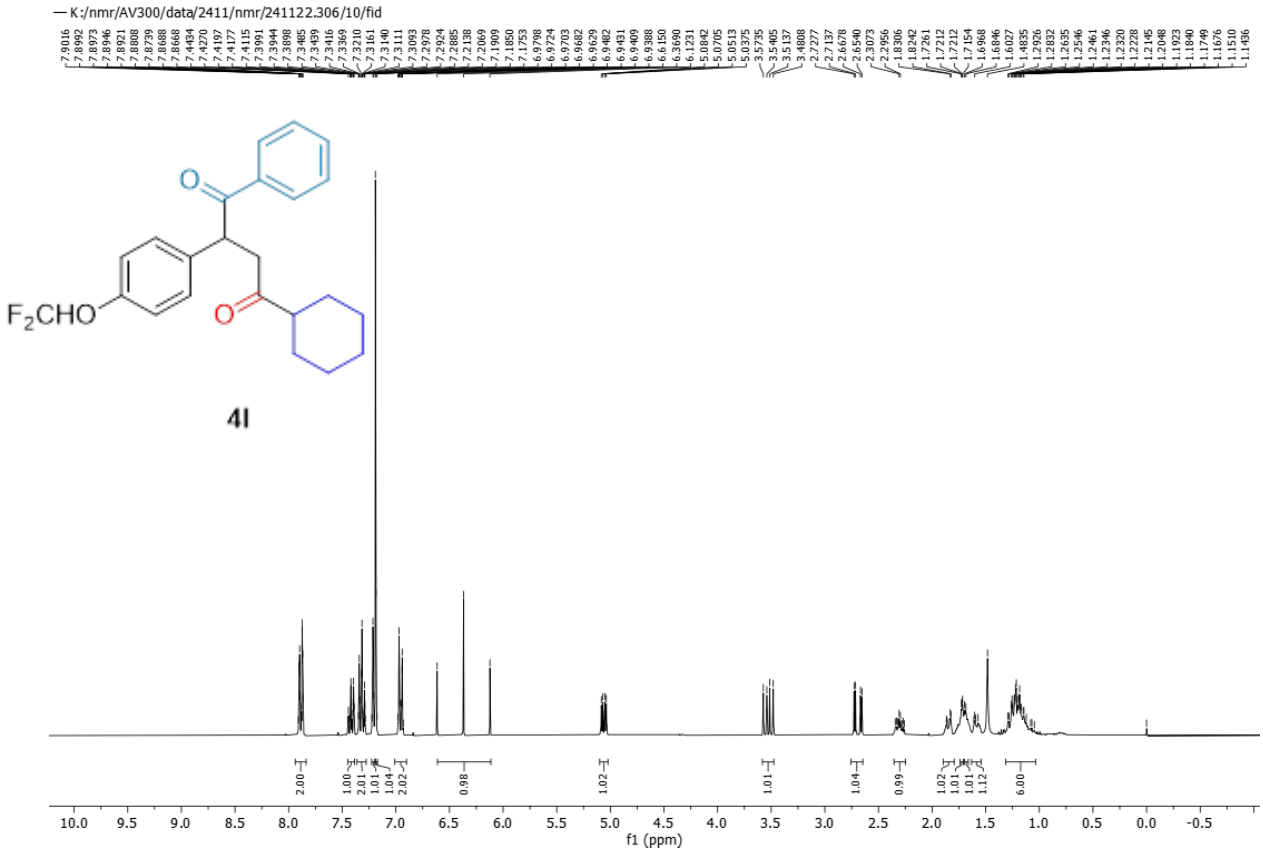
4h ^{1}H NMR (300 MHz, $CDCl_3$)



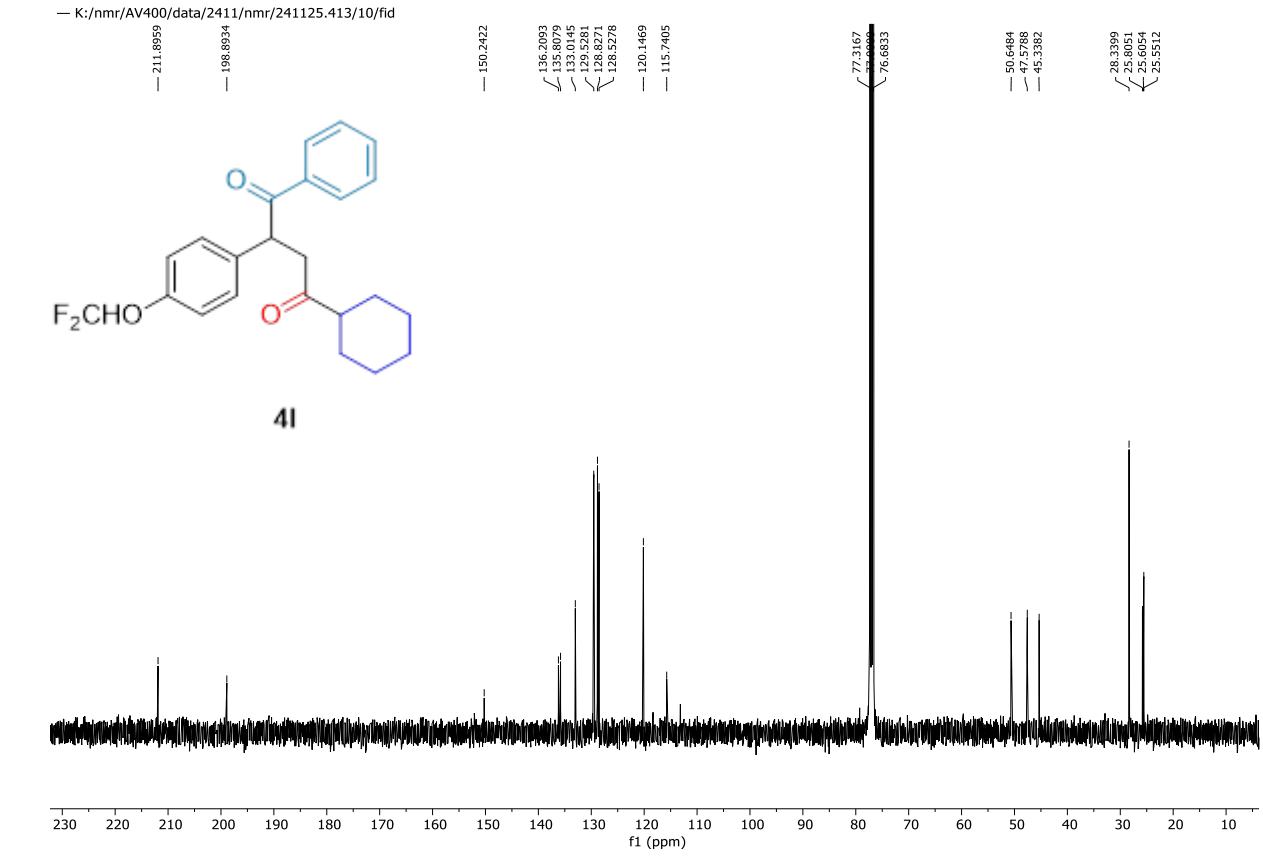
4h ^{13}C NMR (75 MHz, $CDCl_3$)



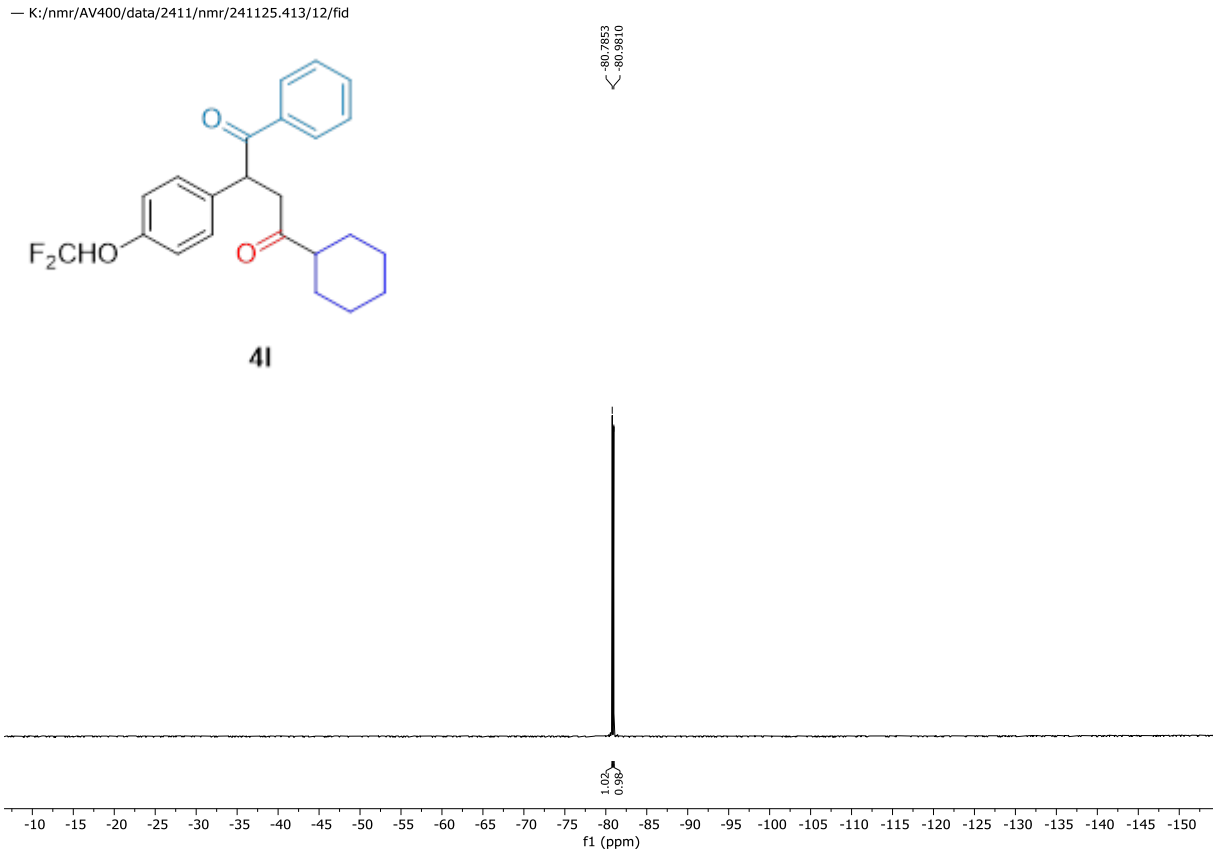
4i ^{1}H NMR (300 MHz, $CDCl_3$)



4i ^{13}C NMR (101 MHz, $CDCl_3$)



4i ^{13}F NMR (282 MHz, $CDCl_3$)



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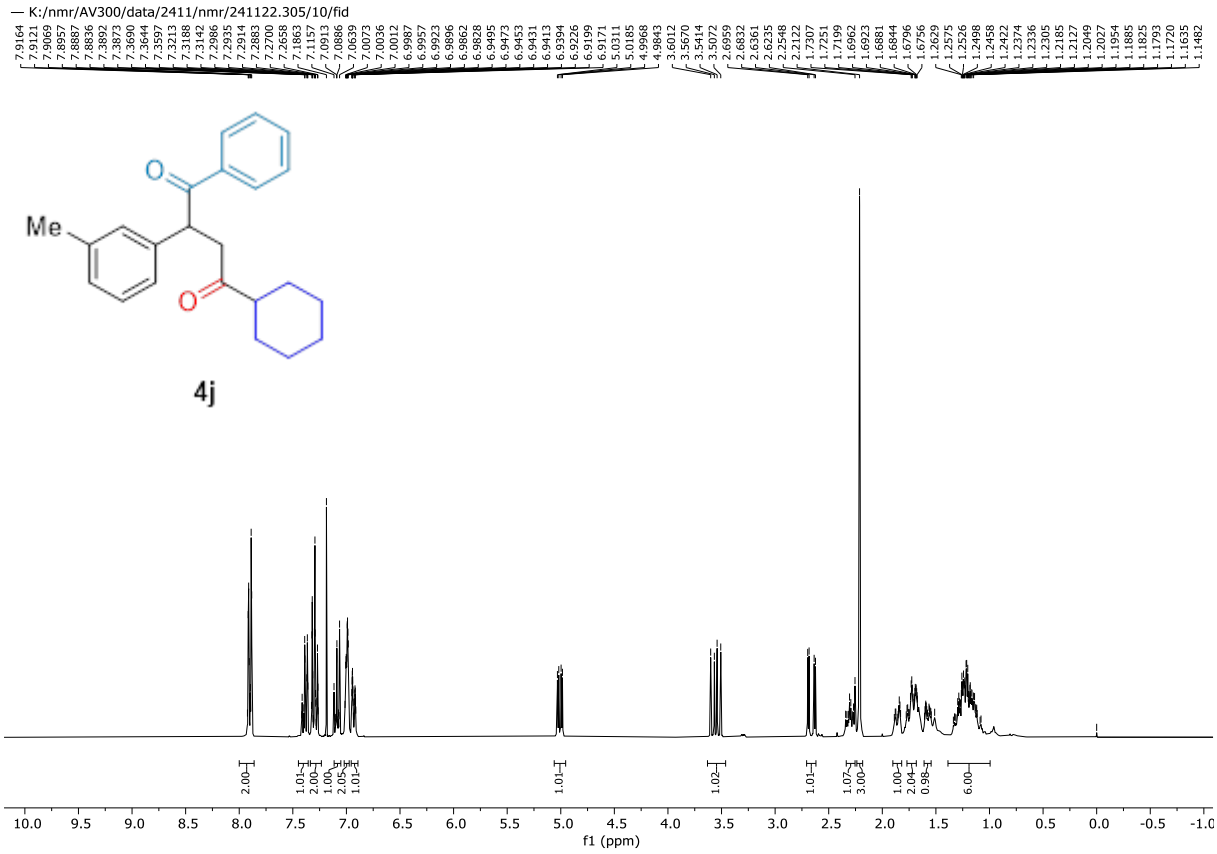

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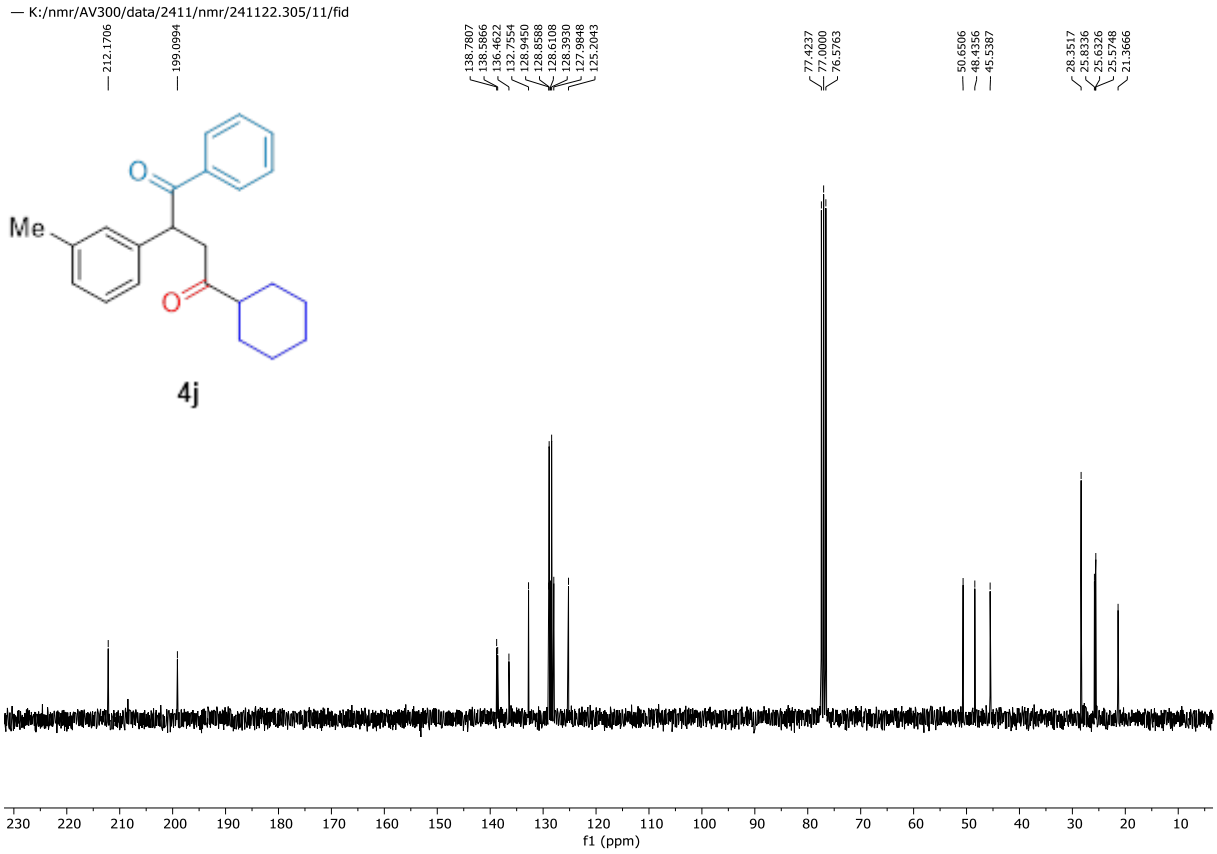

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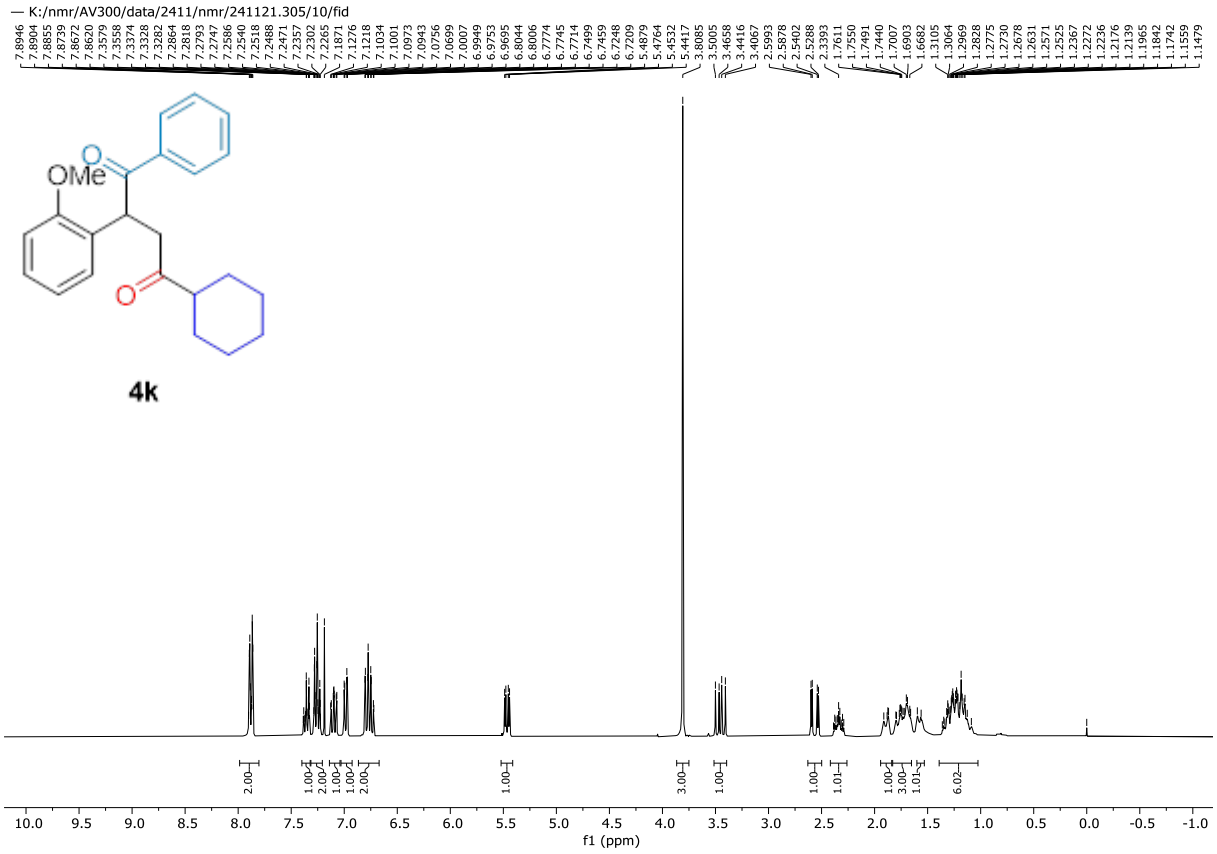

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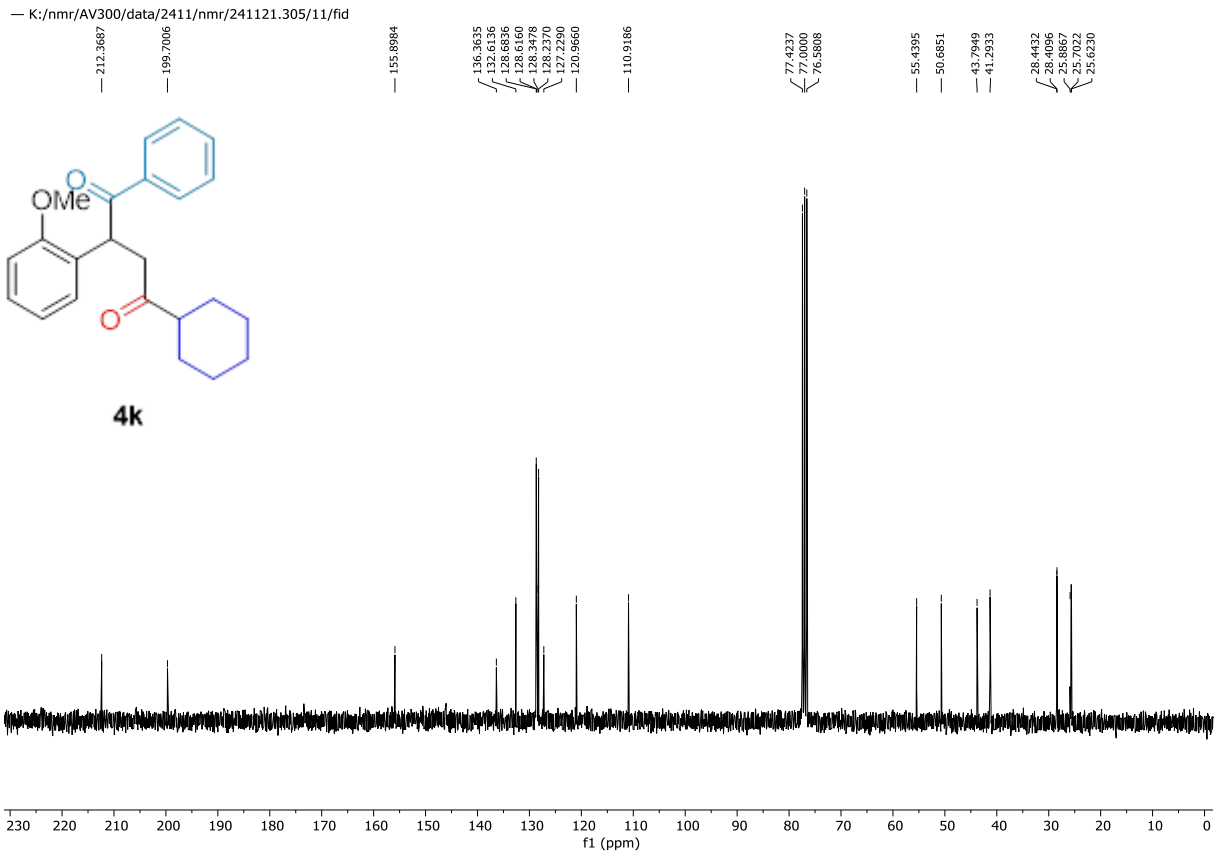

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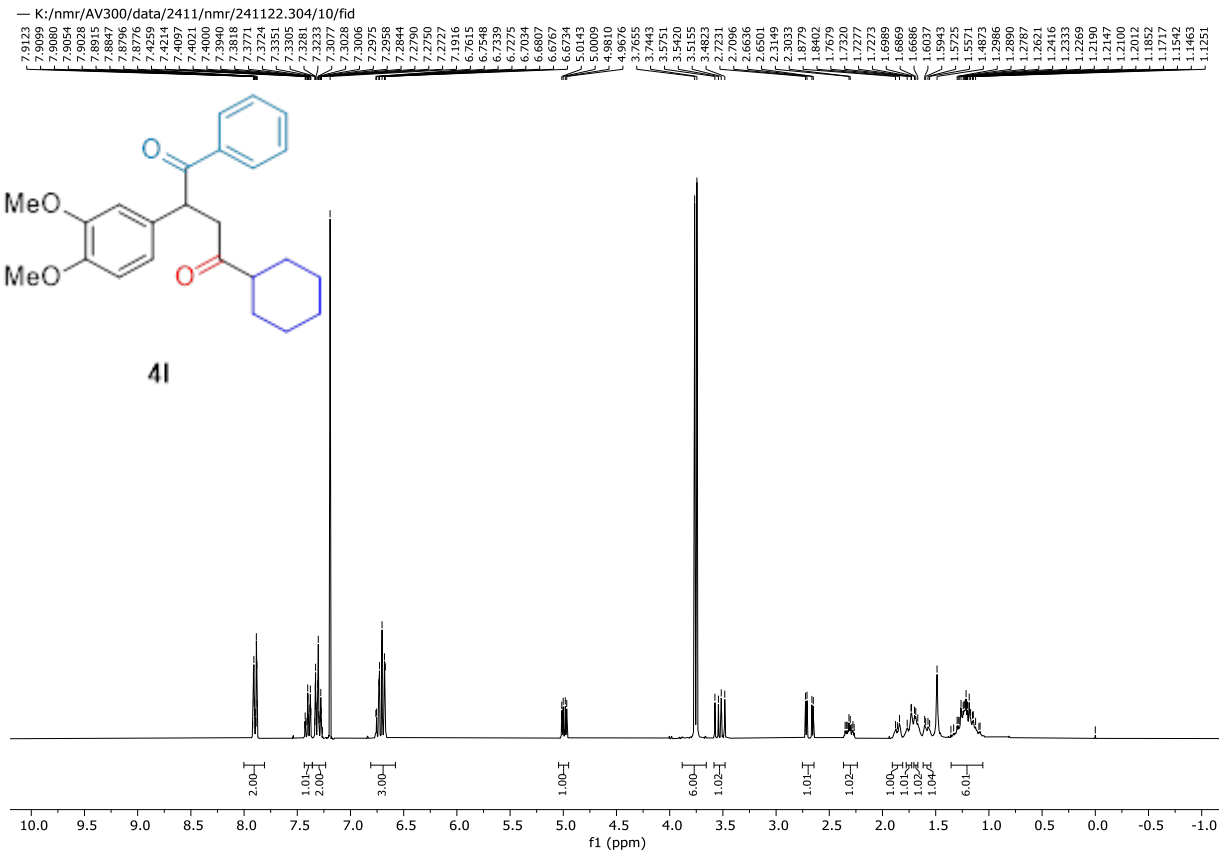
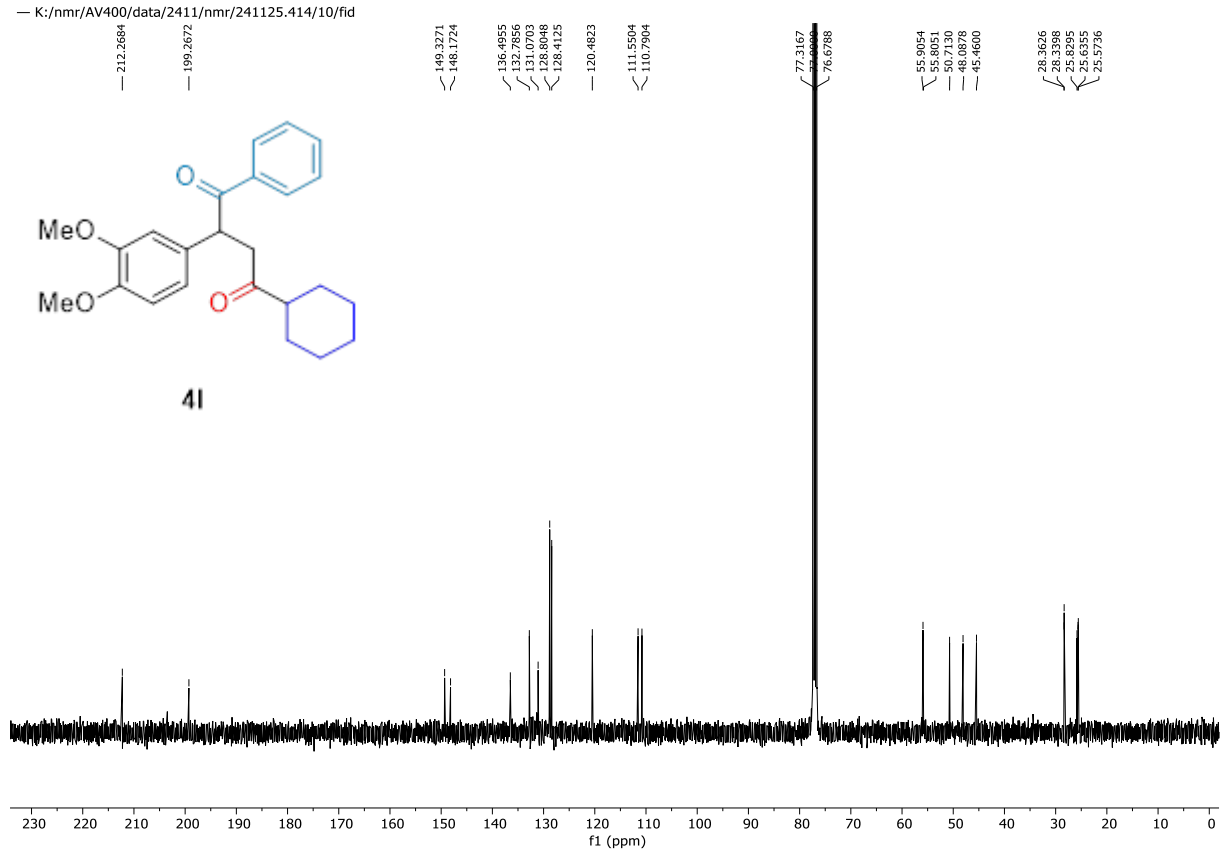
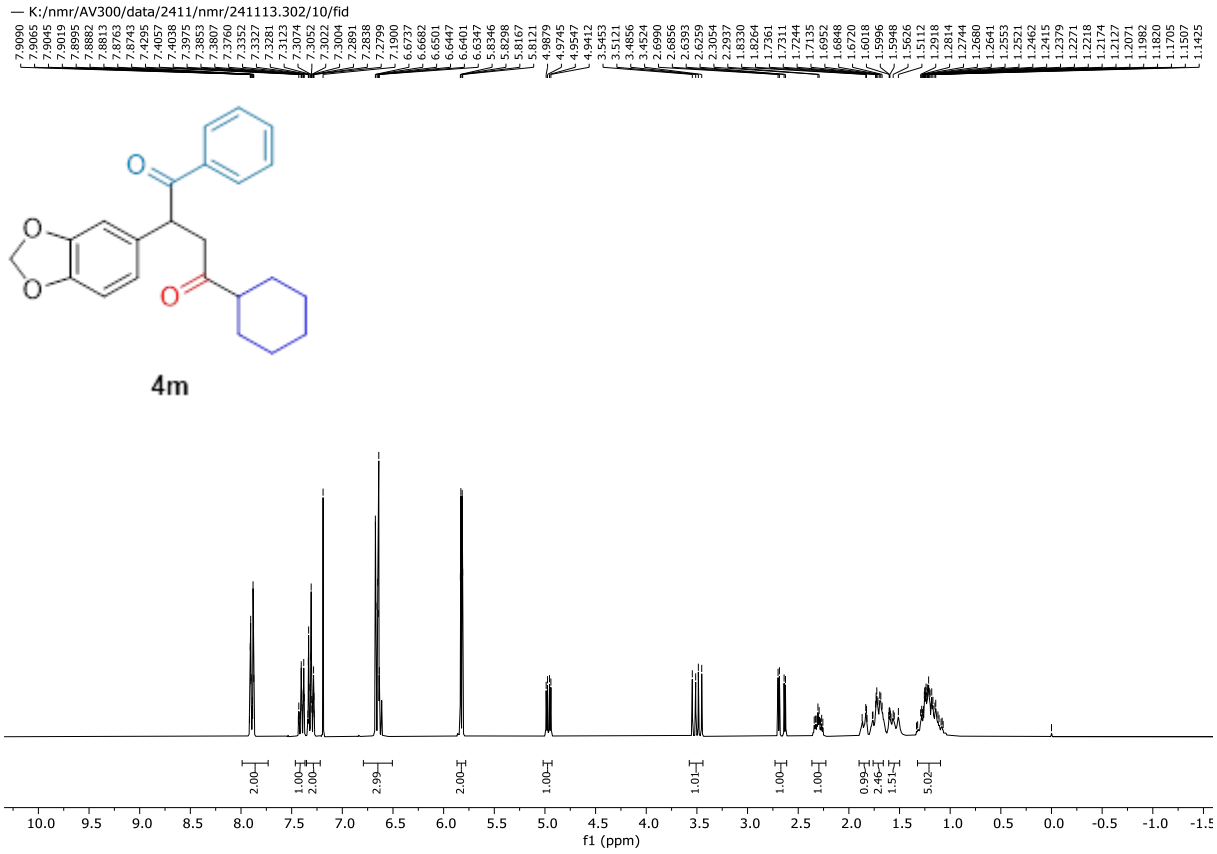


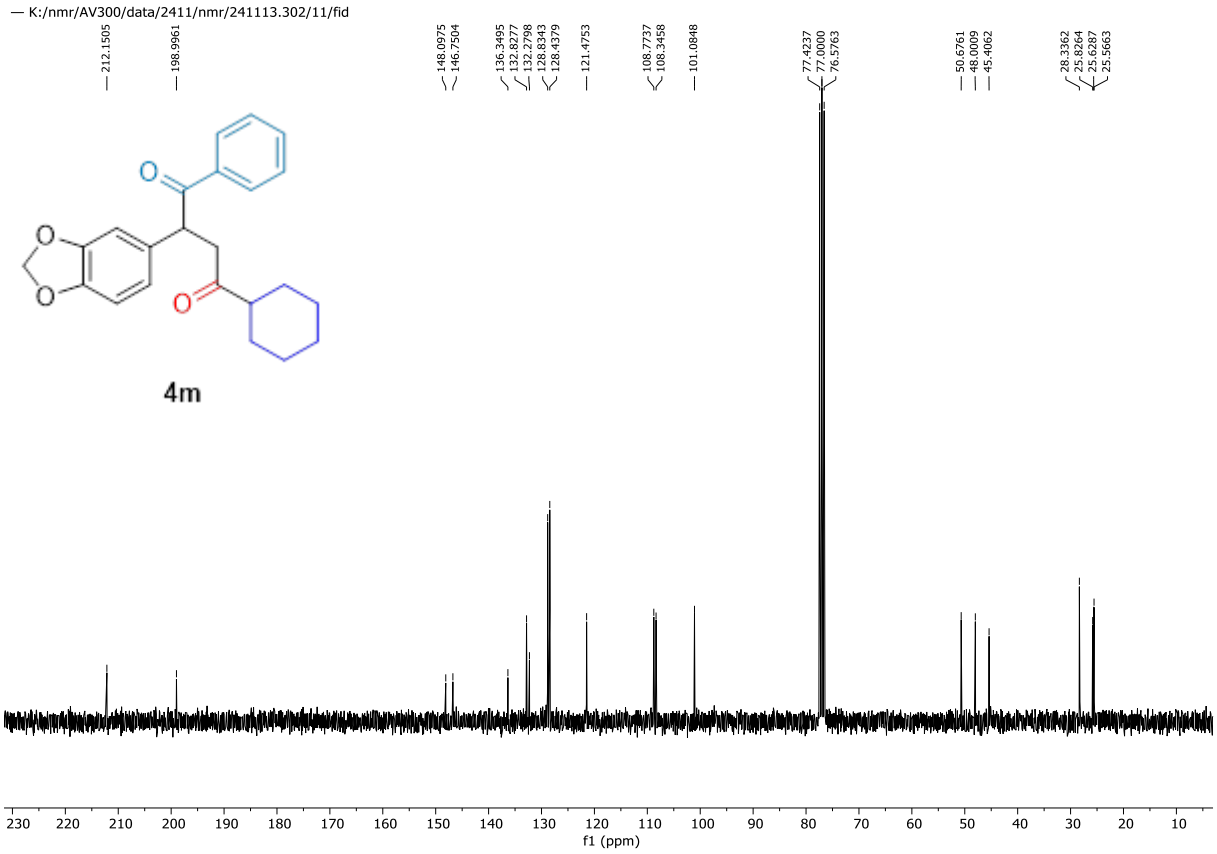
4l ^{13}C NMR (101 MHz, $CDCl_3$)



4m ^{1}H NMR (300 MHz, $CDCl_3$)



4m ^{13}C NMR (75 MHz, $CDCl_3$)



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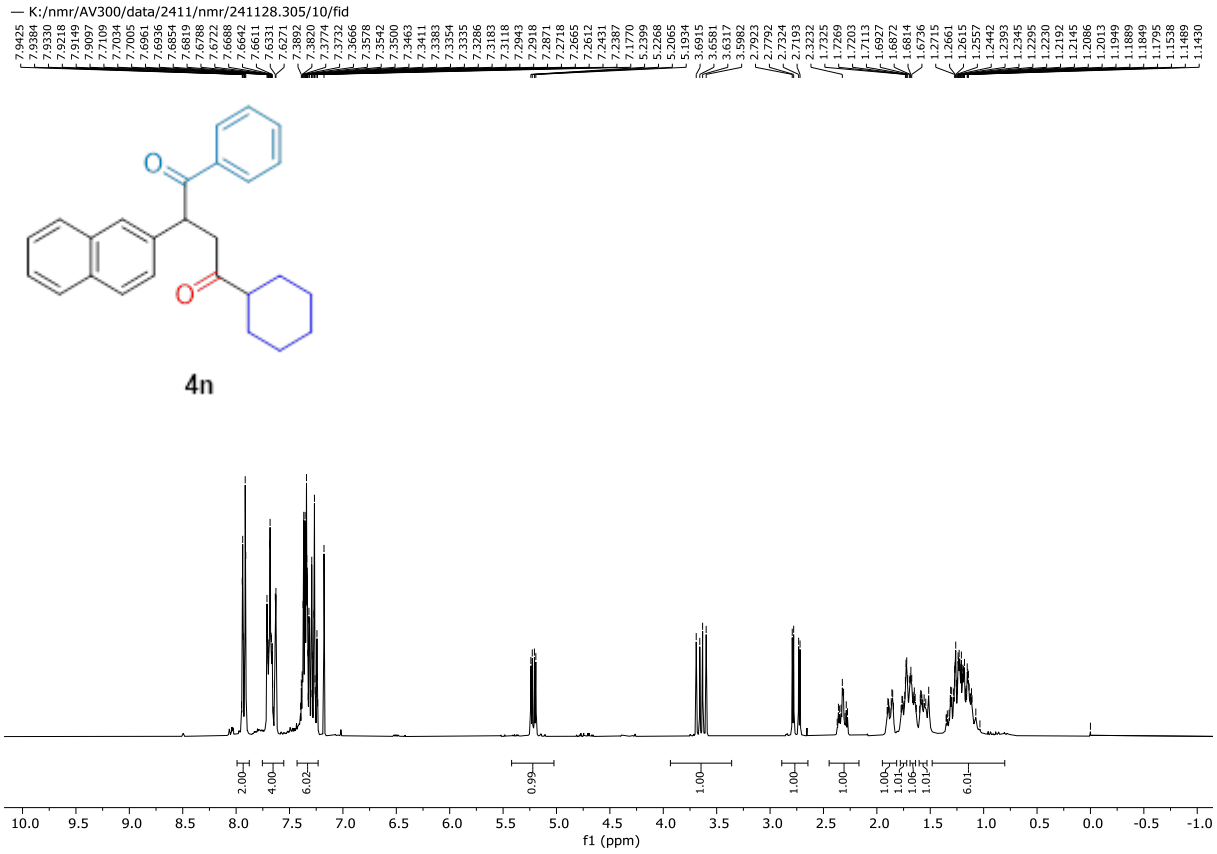

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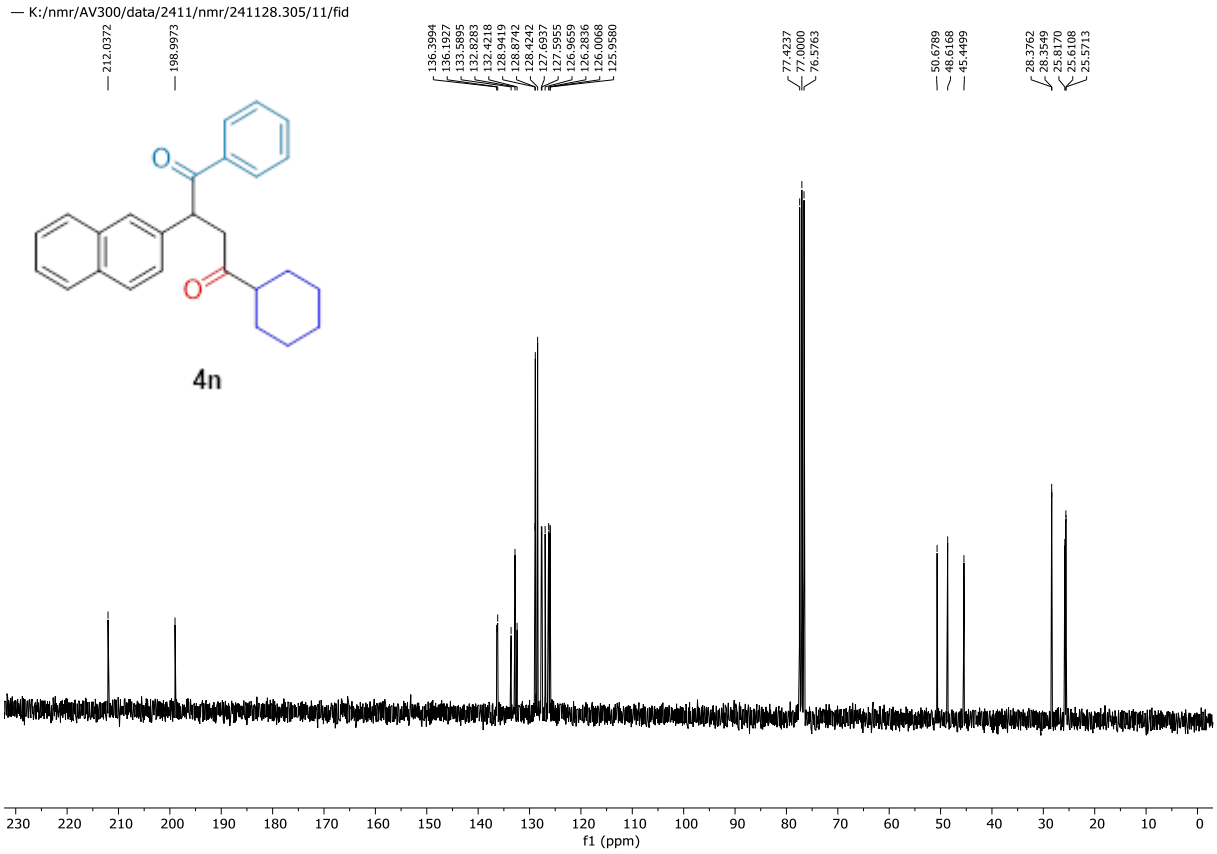
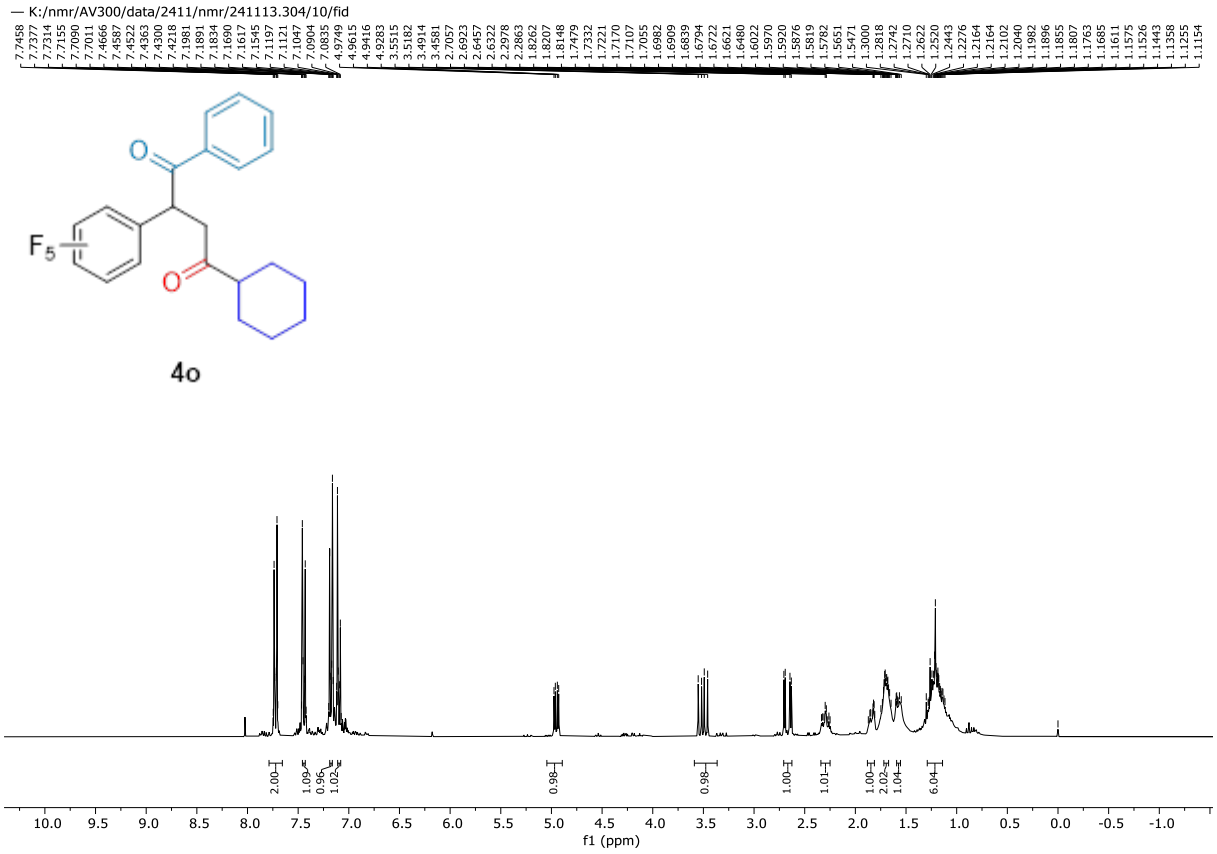
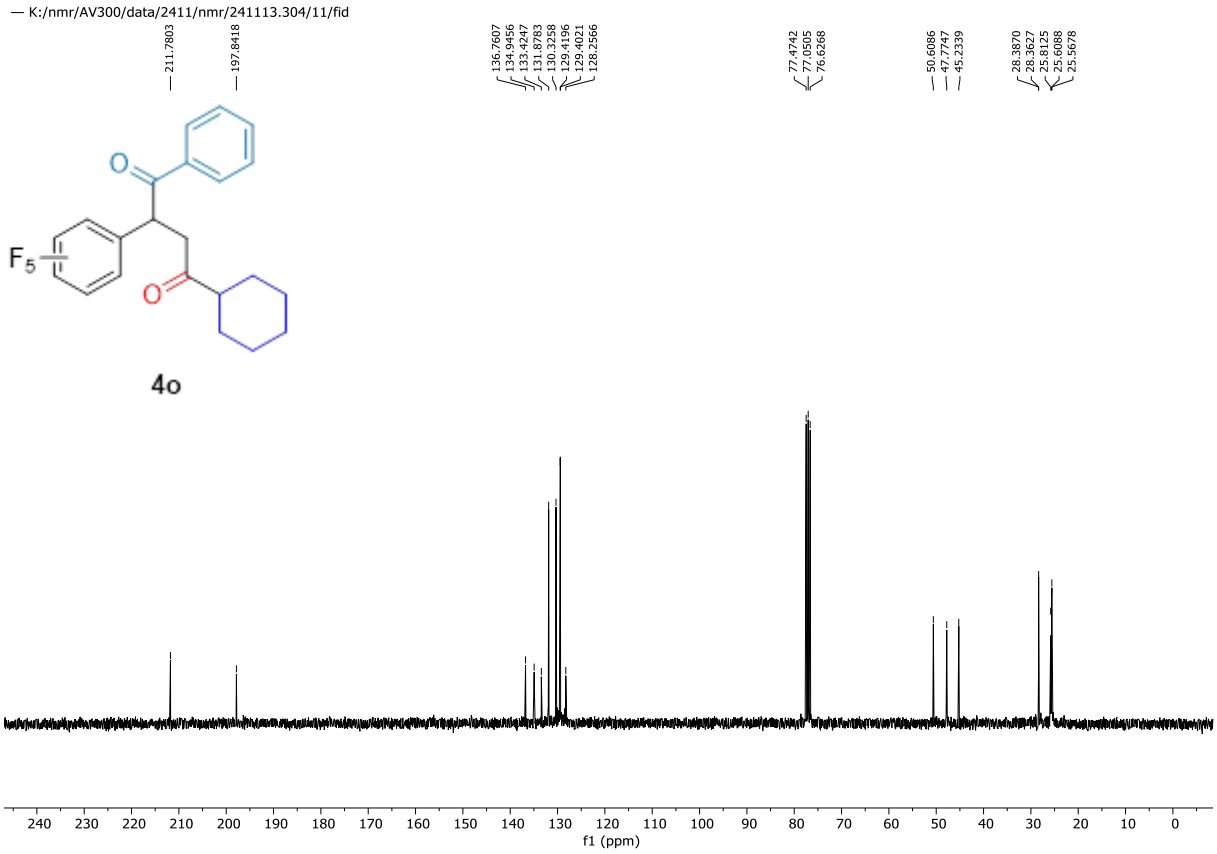


4o ^{1}H NMR (300 MHz, $CDCl_3$)



4o ^{13}C NMR (75 MHz, $CDCl_3$)



4o ^{13}F NMR (282 MHz, $CDCl_3$)

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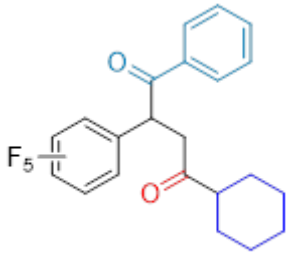
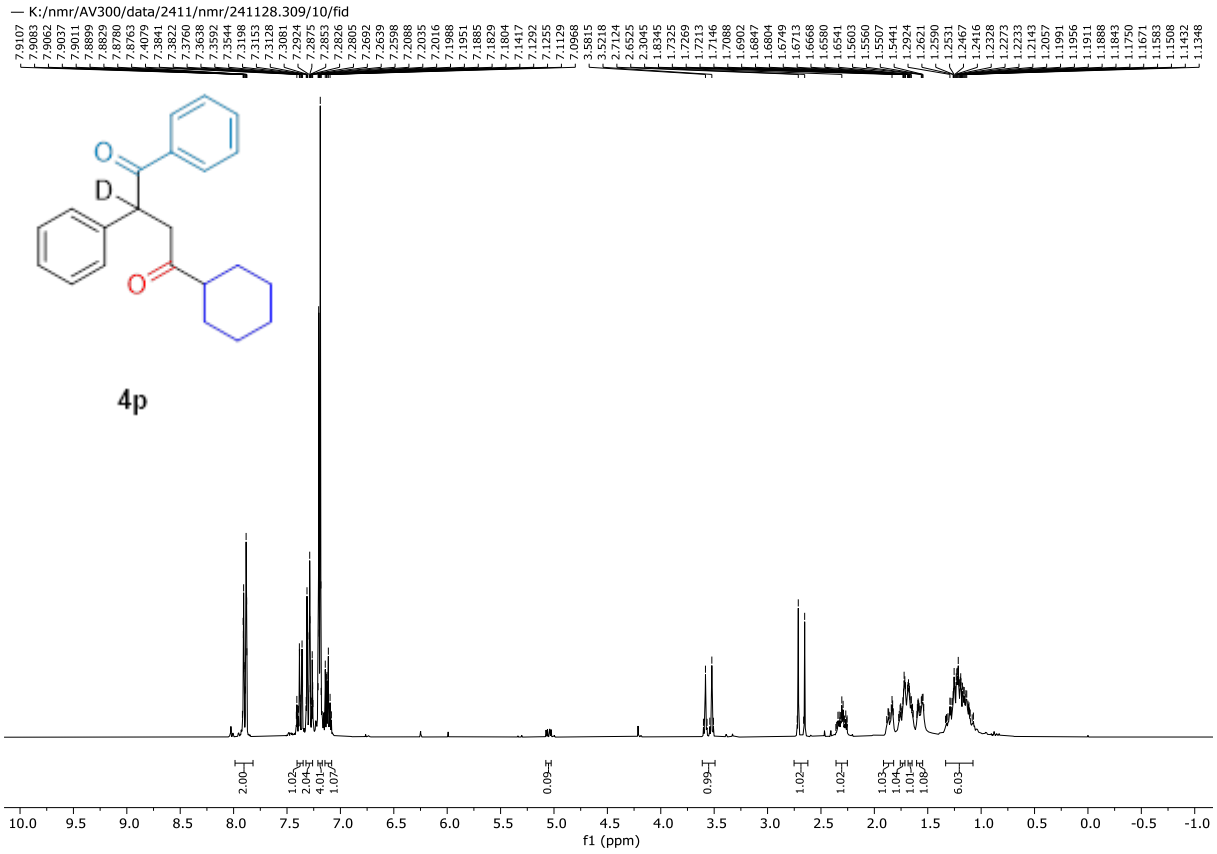
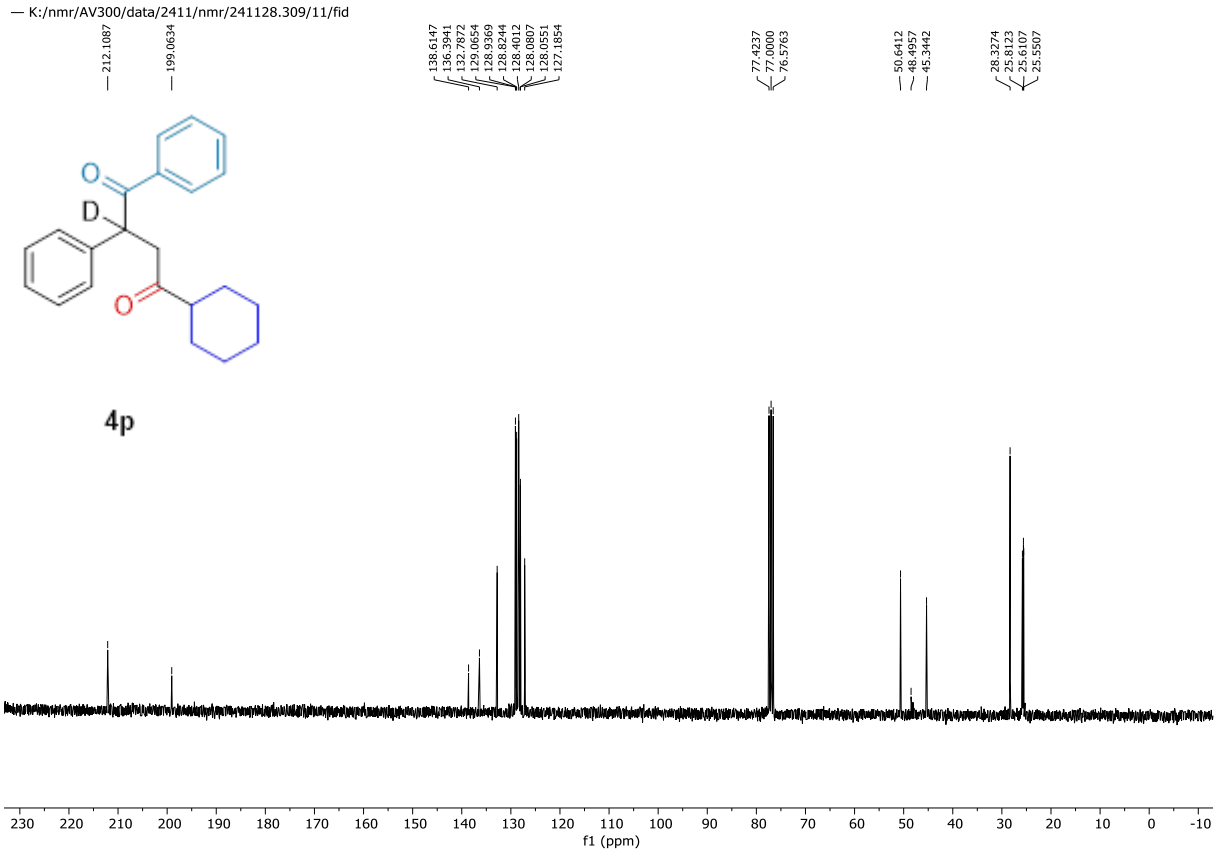


4o

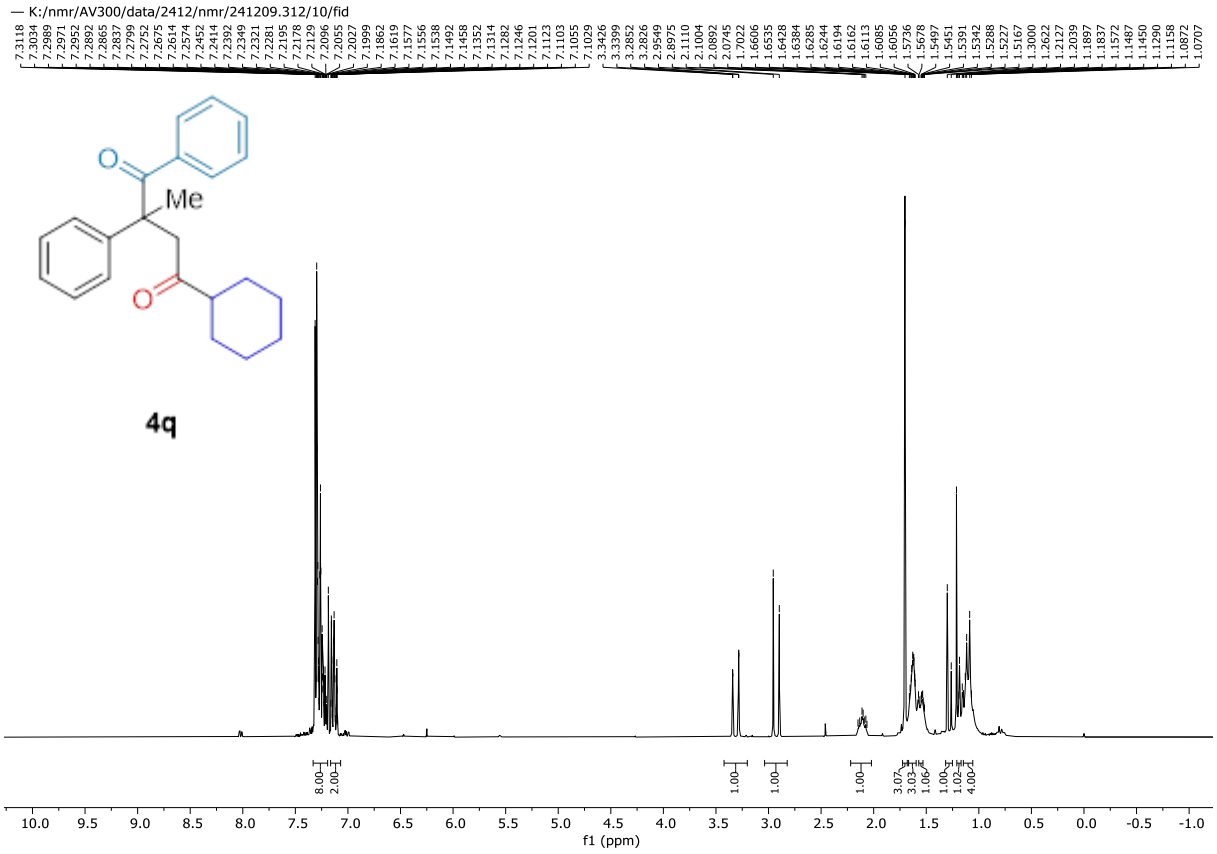
4p ^{1}H NMR (300 MHz, $CDCl_3$)



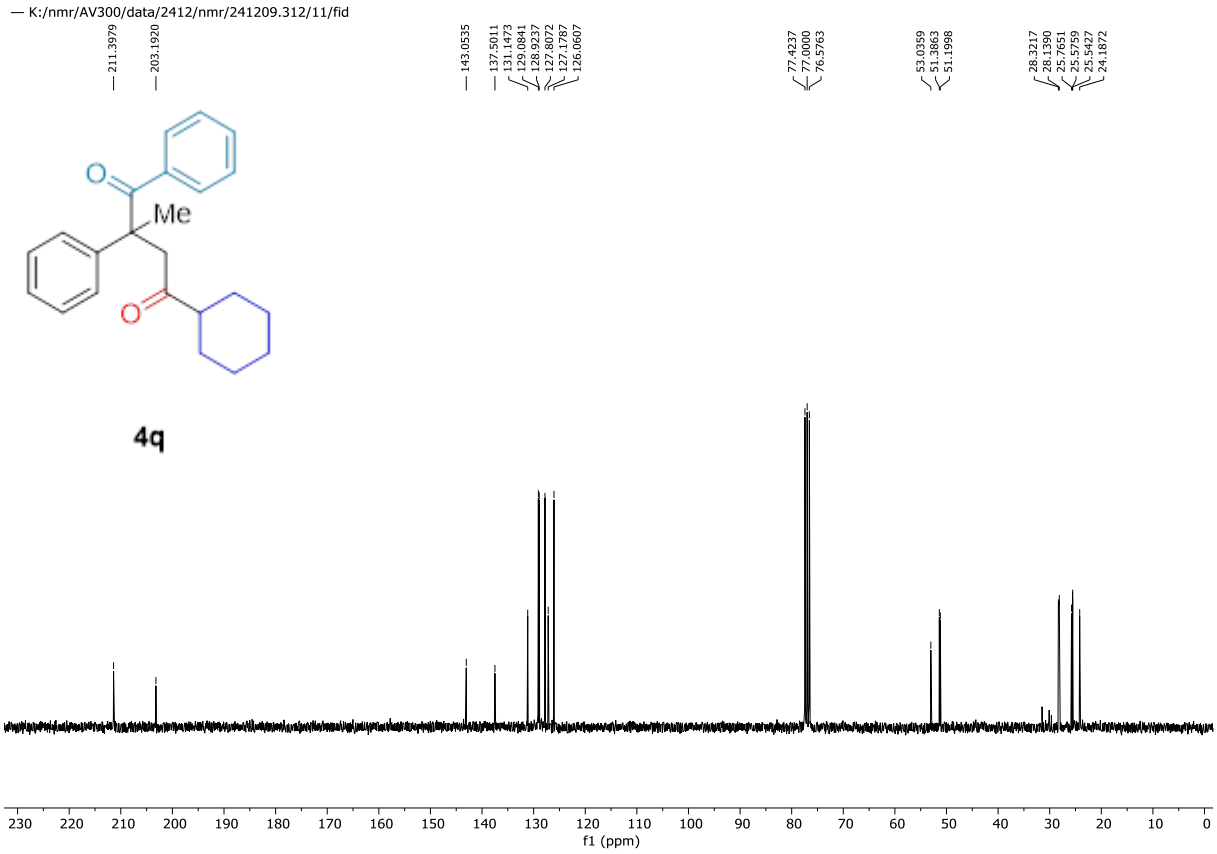
4p ^{13}C NMR (75 MHz, $CDCl_3$)



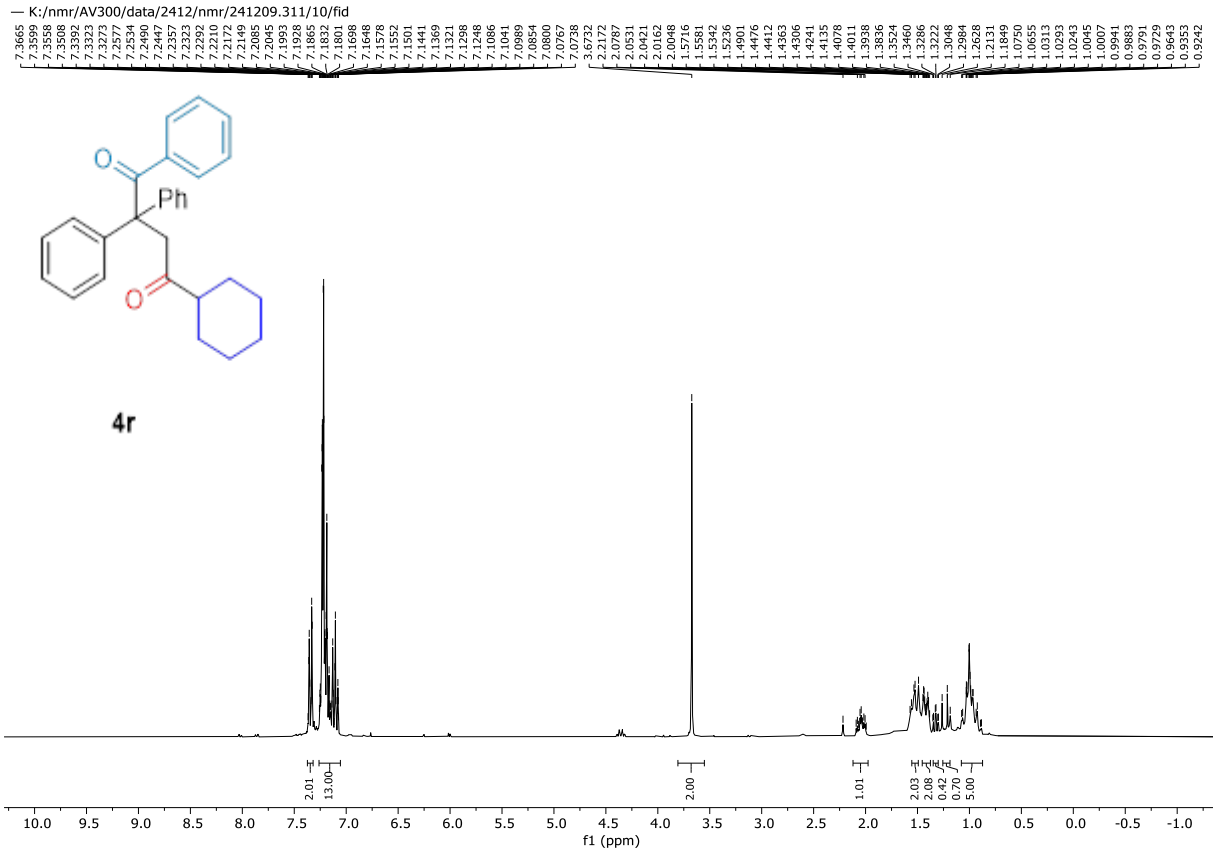
4q ^{1}H NMR (300 MHz, $CDCl_3$)



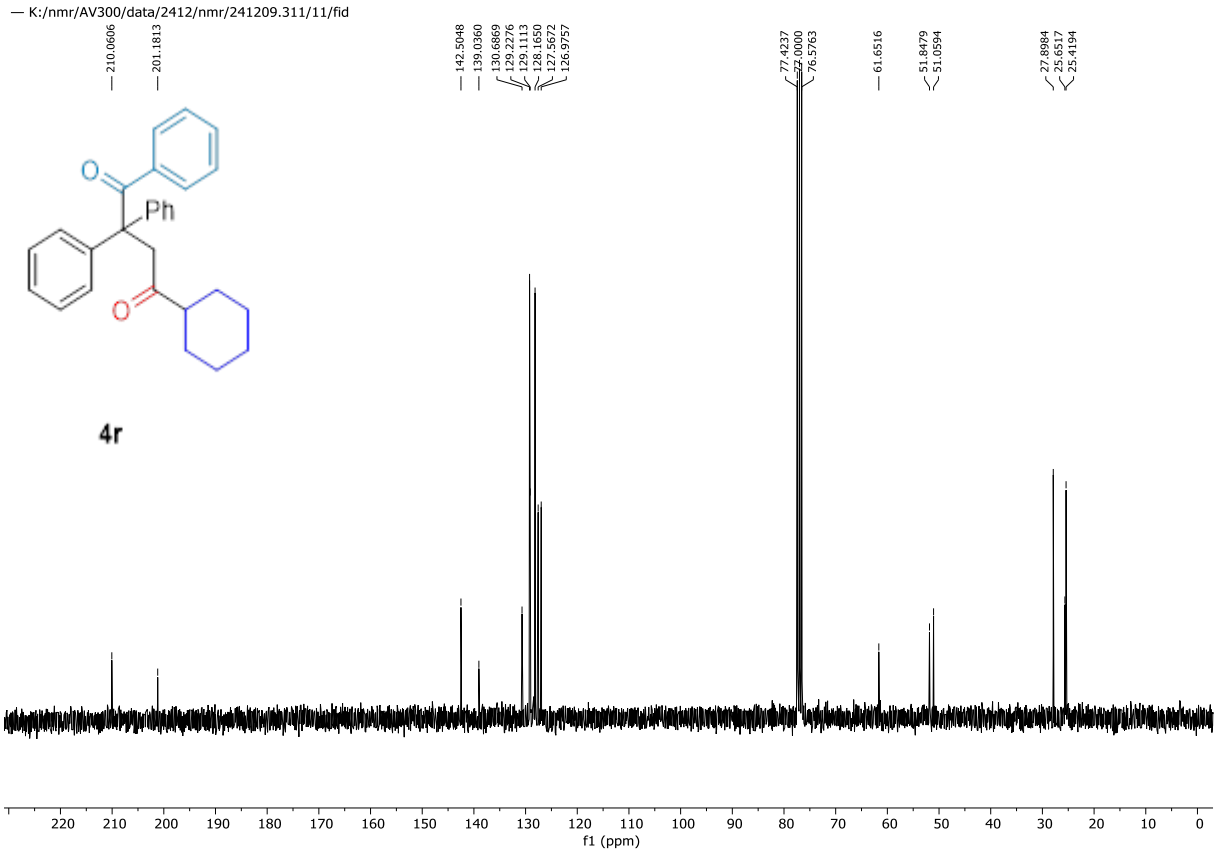
4q ^{13}C NMR (75 MHz, $CDCl_3$)



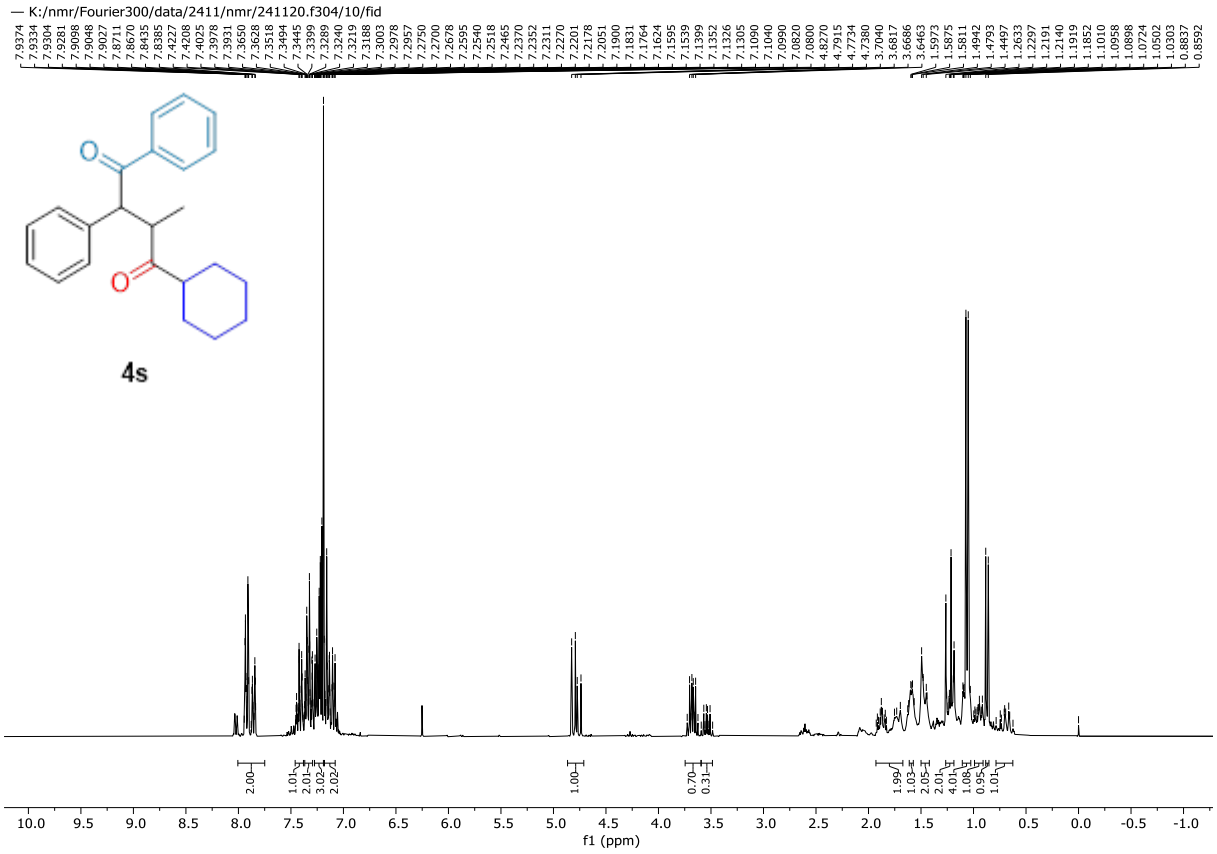
4r ^{1}H NMR (300 MHz, $CDCl_3$)



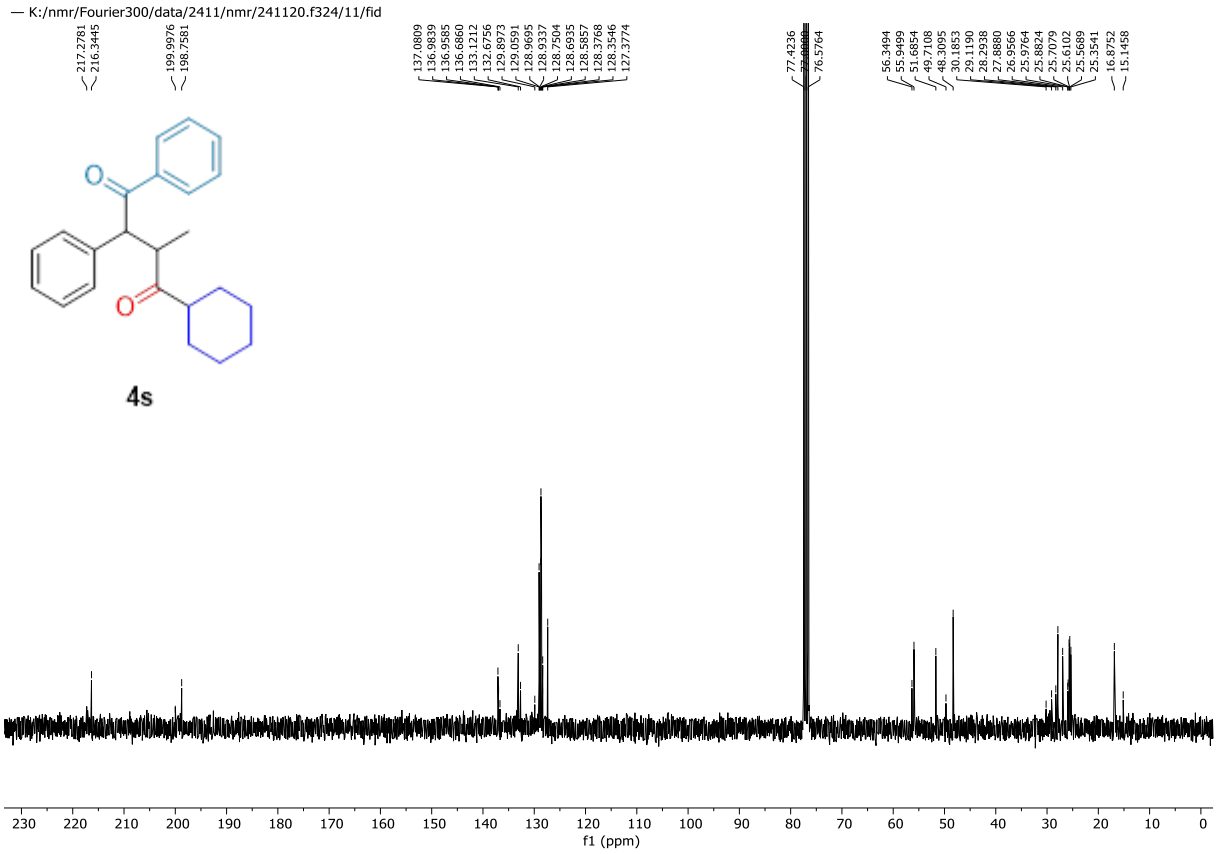
4r ^{13}C NMR (75 MHz, $CDCl_3$)



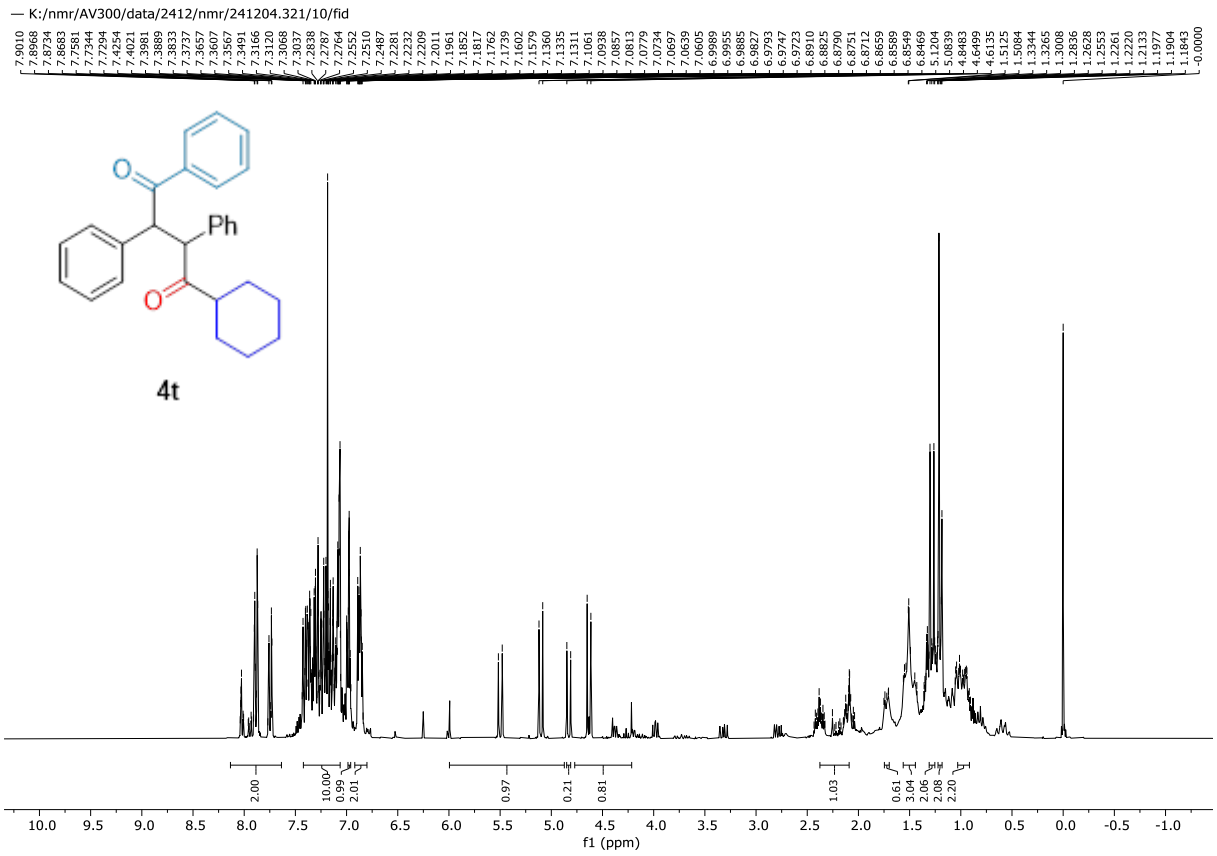
4s ^{1}H NMR (300 MHz, $CDCl_3$)



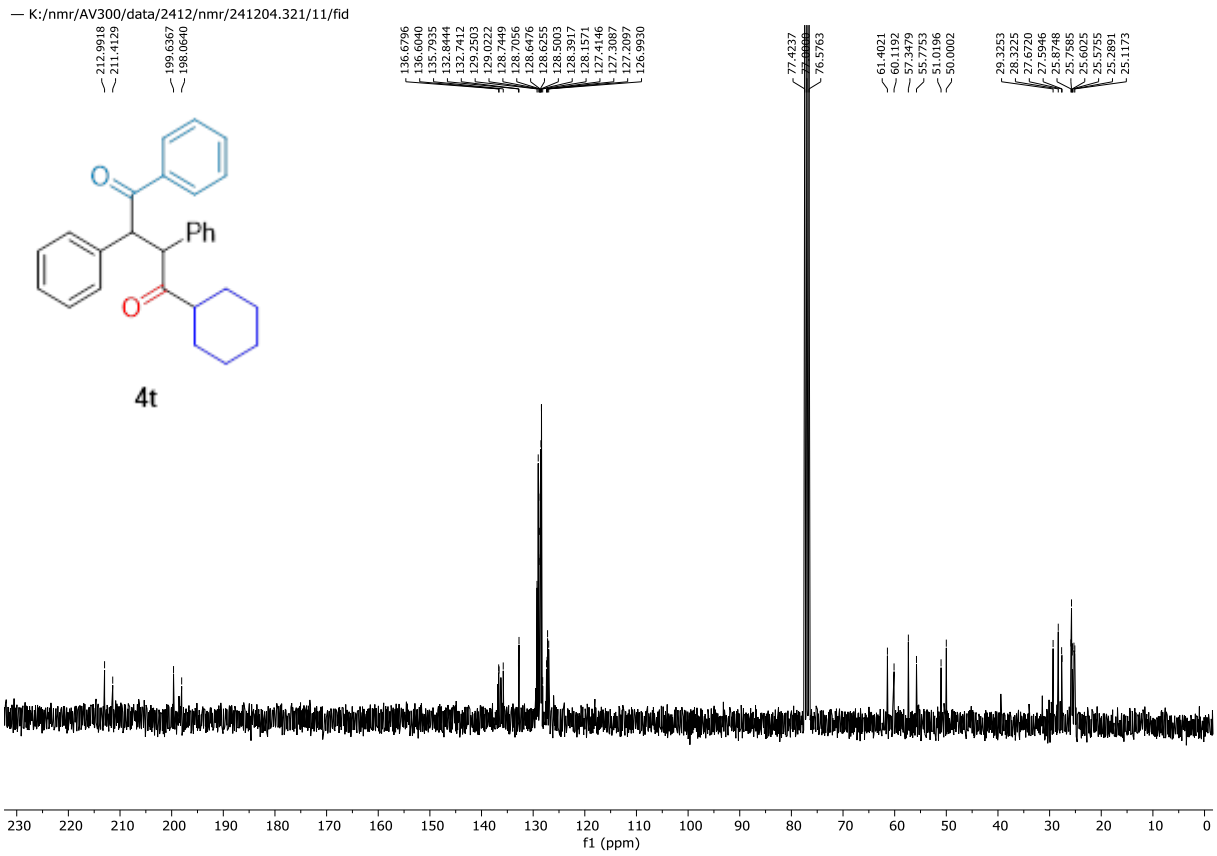
4s ^{13}C NMR (75 MHz, $CDCl_3$)



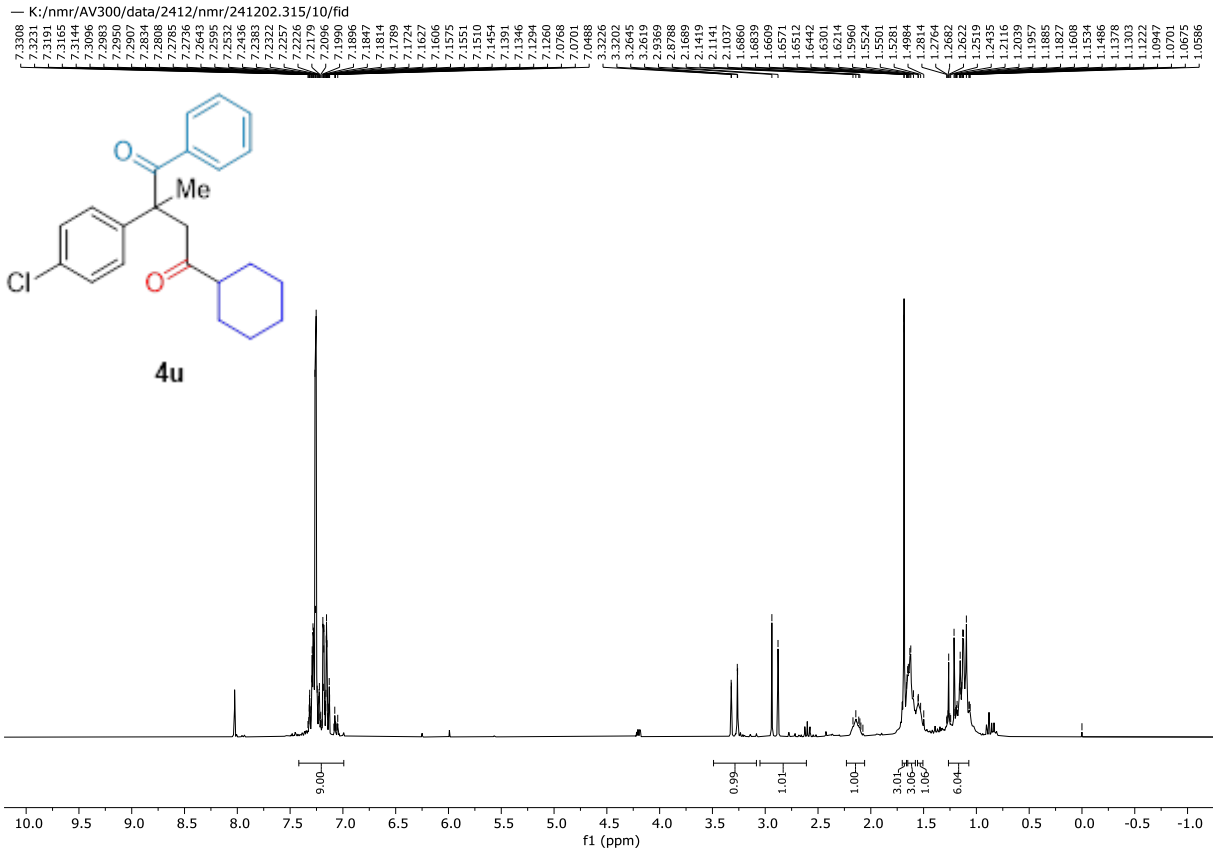
4t ^{1}H NMR (300 MHz, $CDCl_3$)



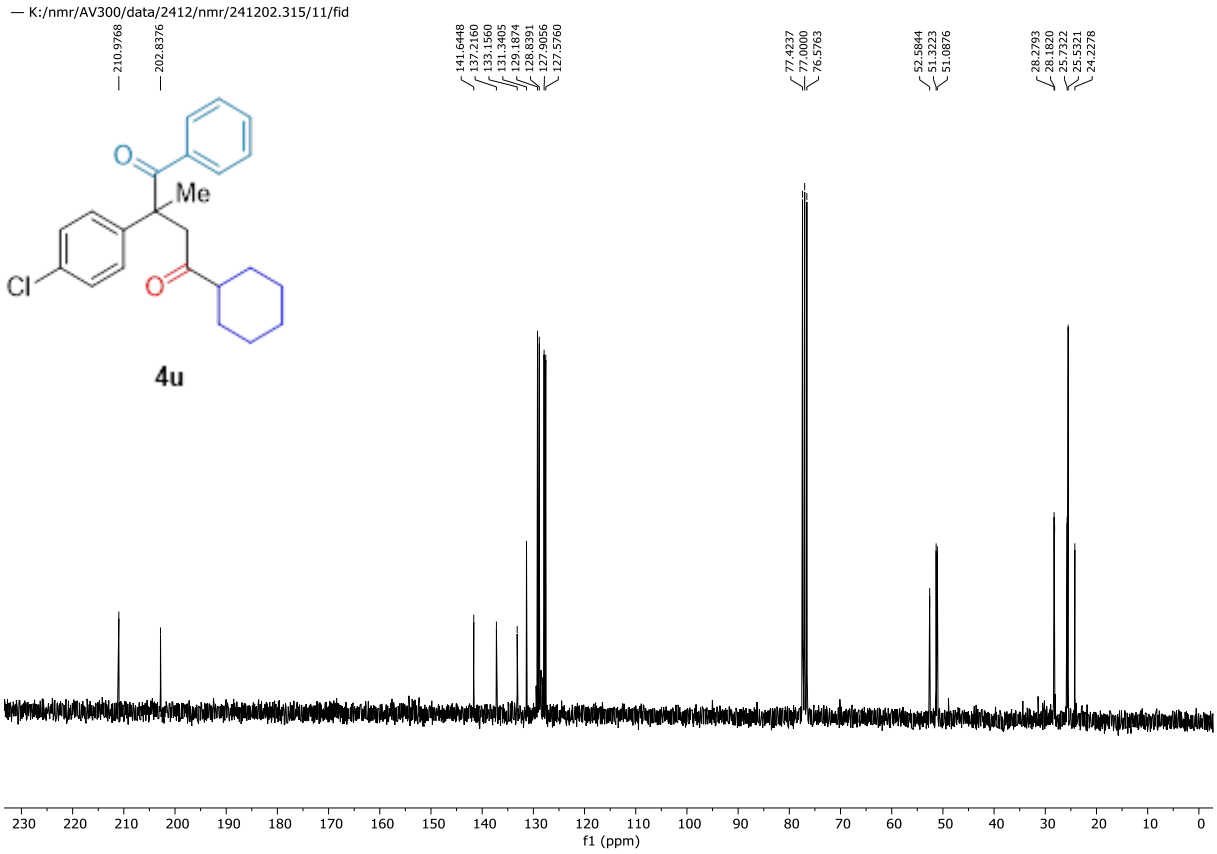
4t ^{13}C NMR (75 MHz, $CDCl_3$)



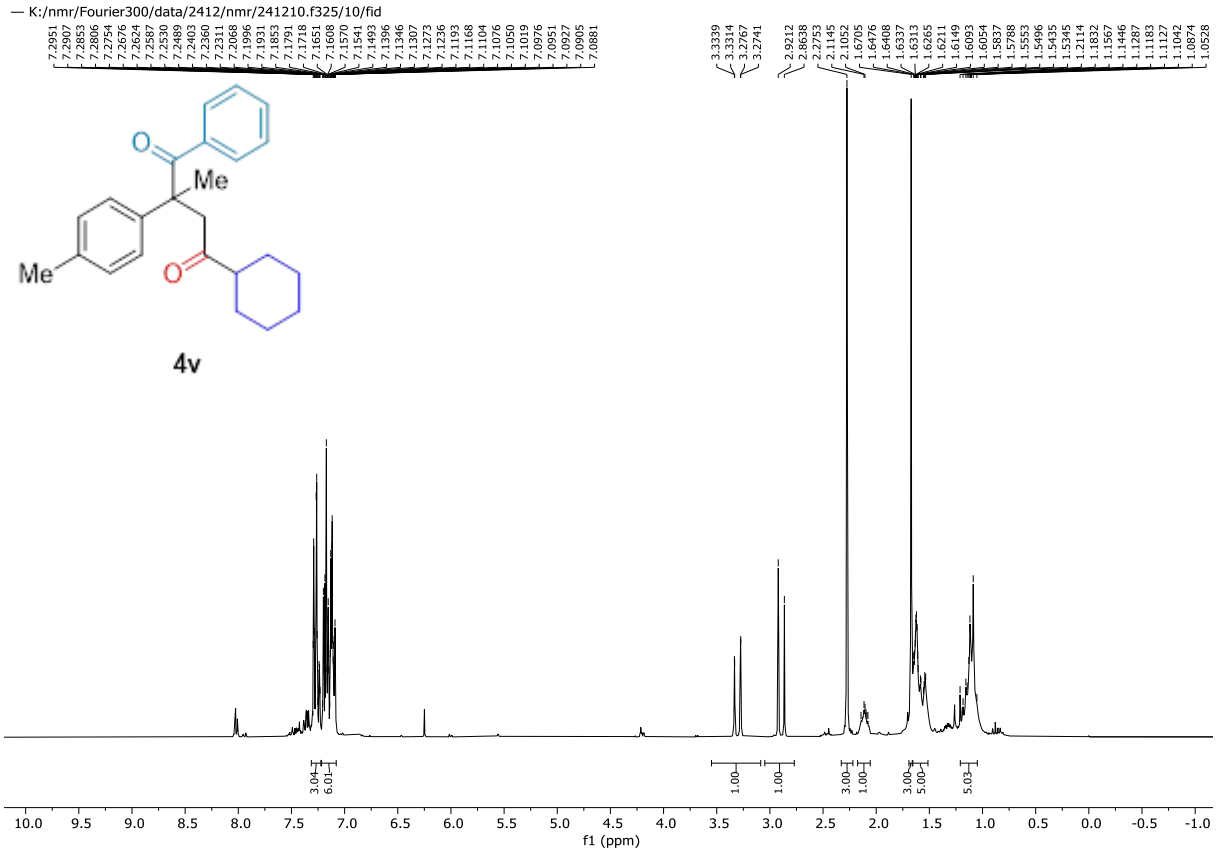
4u ^{1}H NMR (300 MHz, $CDCl_3$)



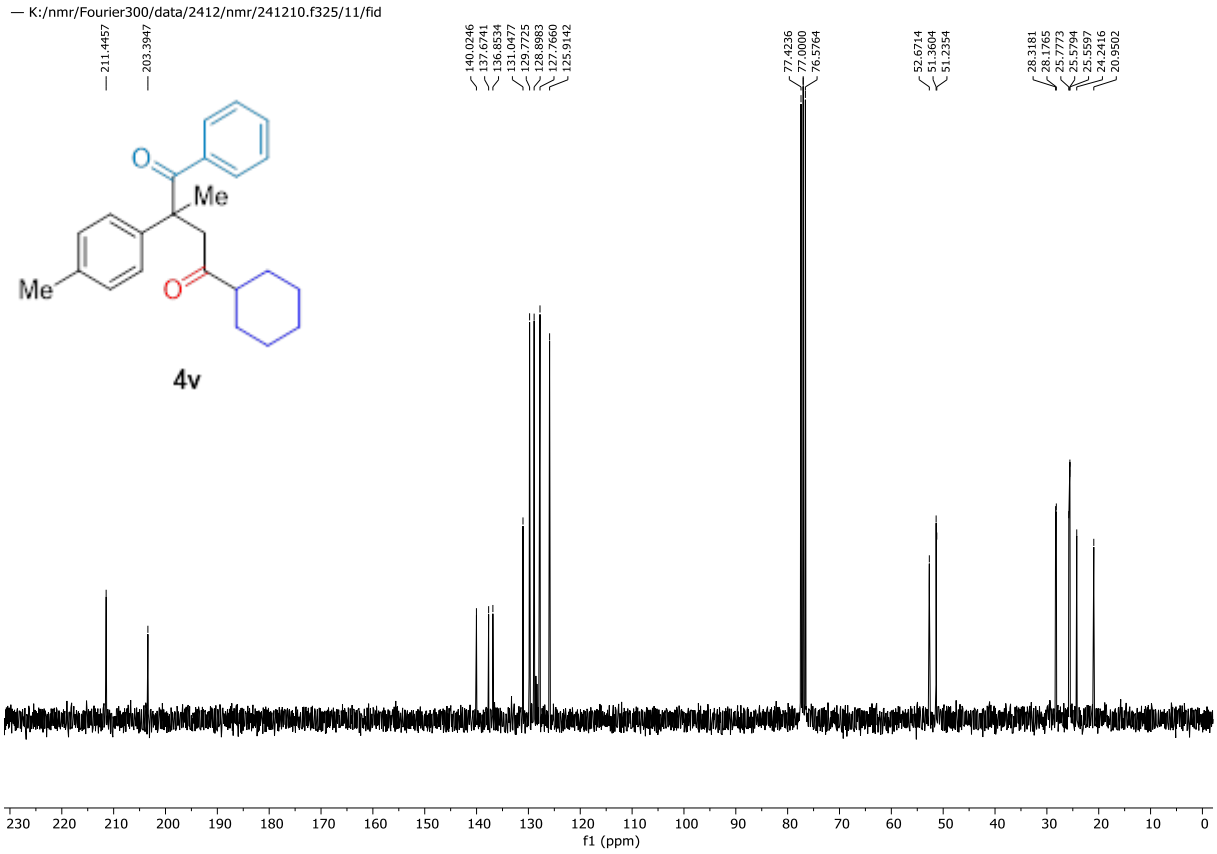
4u ^{13}C NMR (75 MHz, $CDCl_3$)



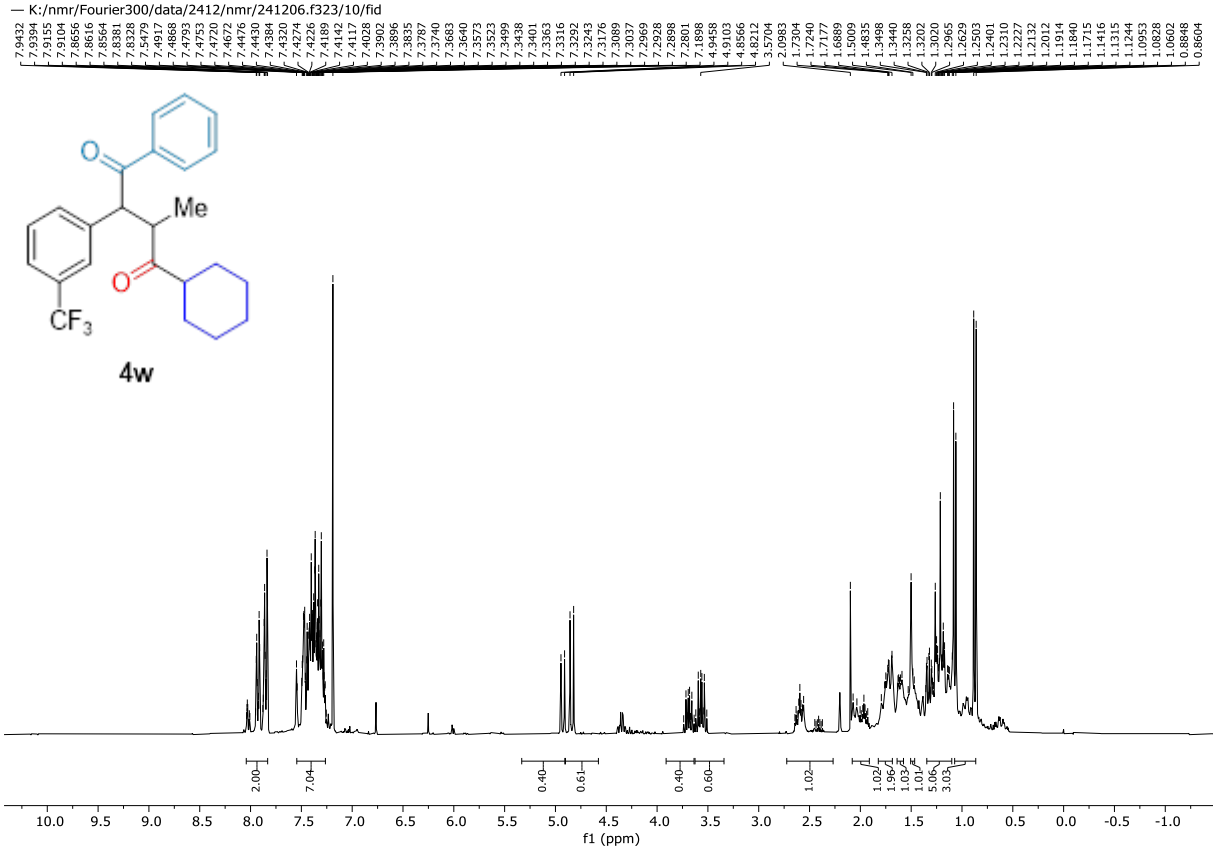
4v ^{1}H NMR (300 MHz, $CDCl_3$)



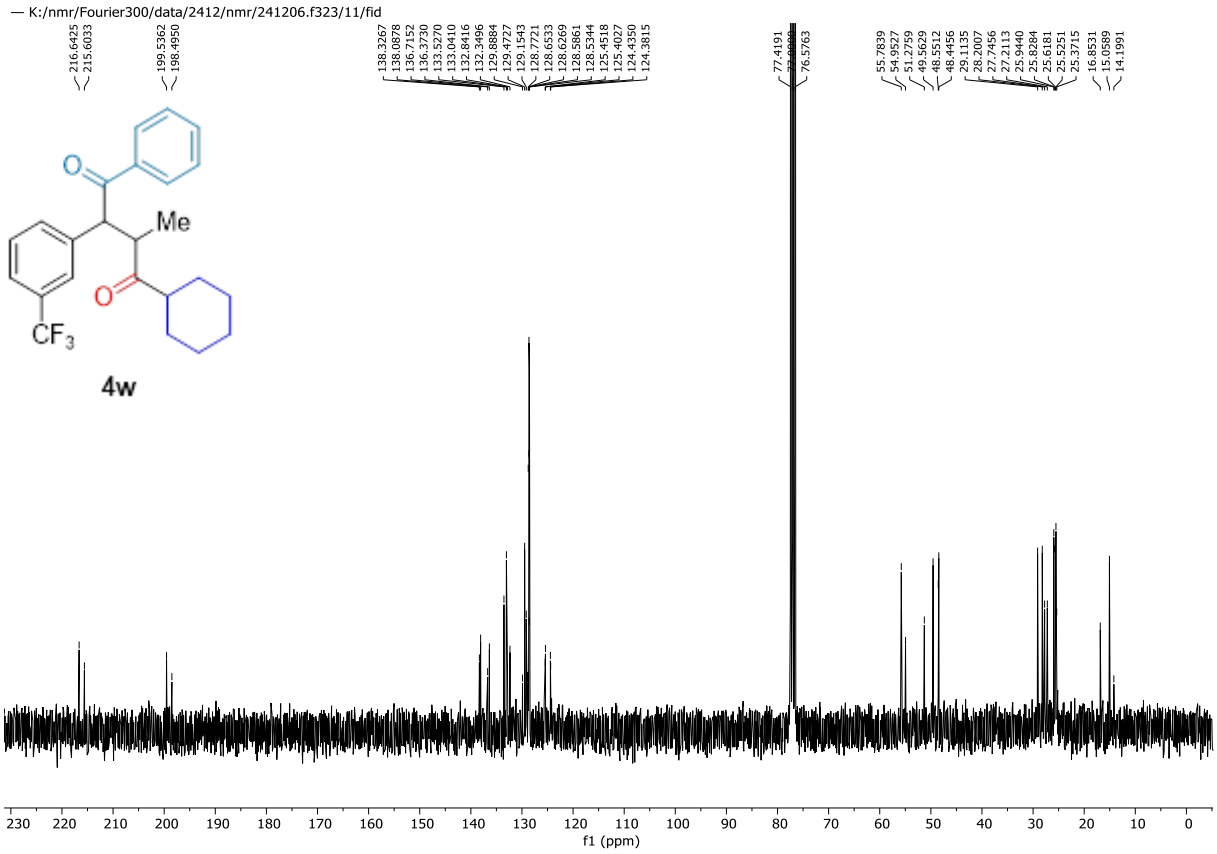
4v ^{13}C NMR (75 MHz, $CDCl_3$)



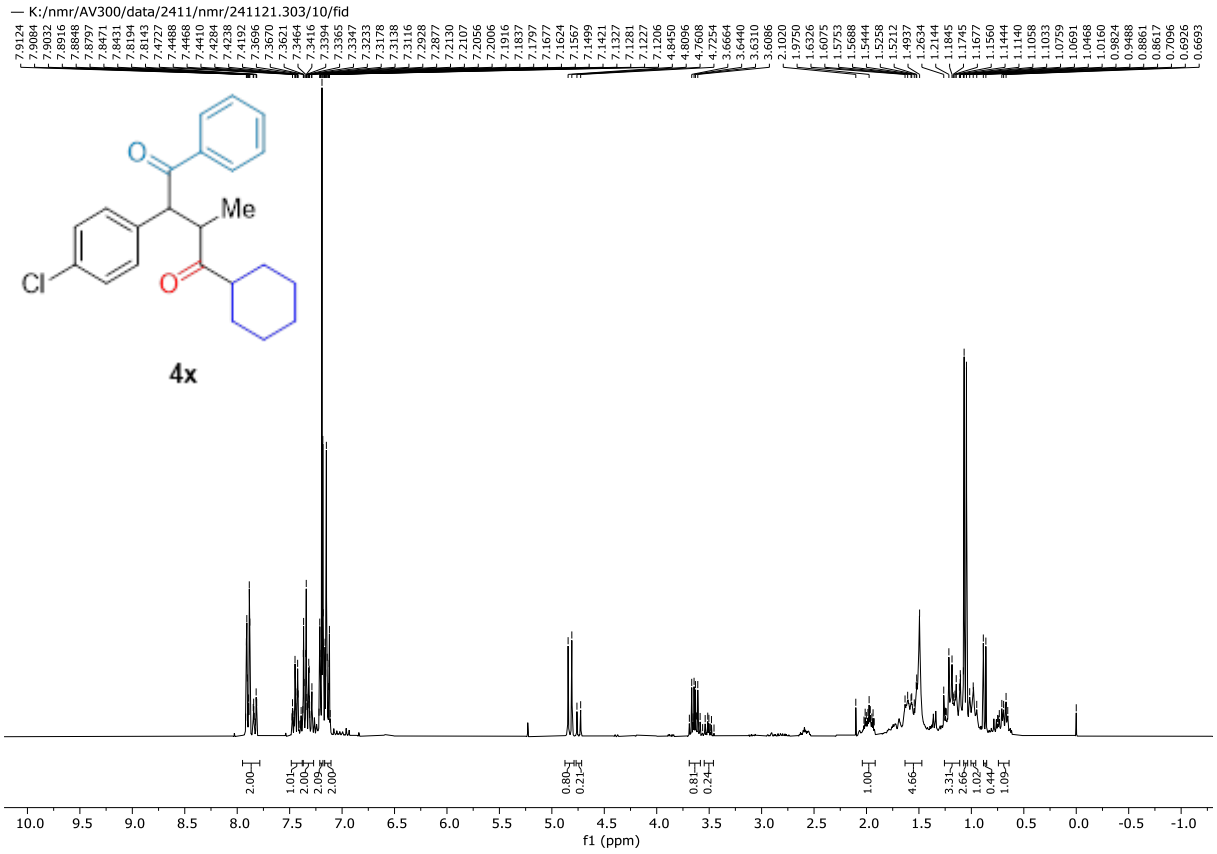
4w ^{1}H NMR (300 MHz, $CDCl_3$)



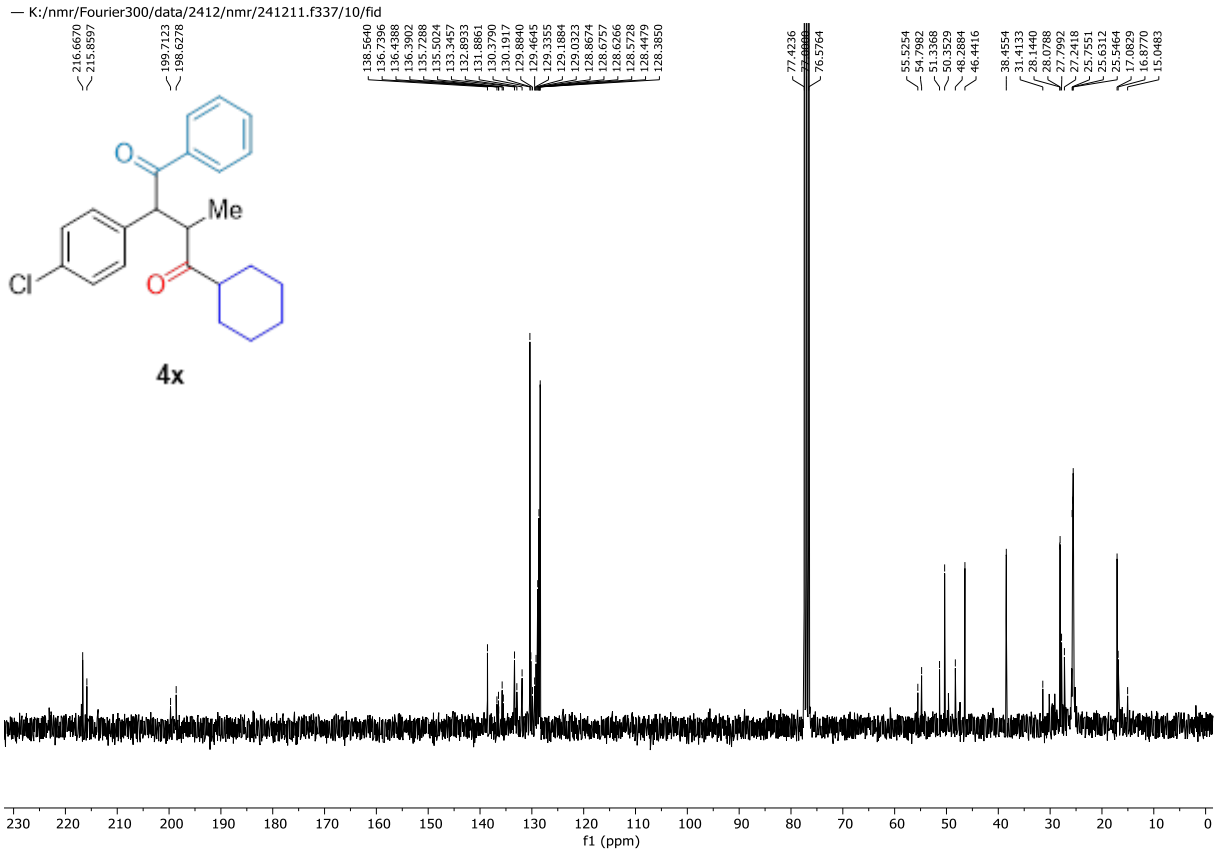
4w ^{13}C NMR (75 MHz, $CDCl_3$)



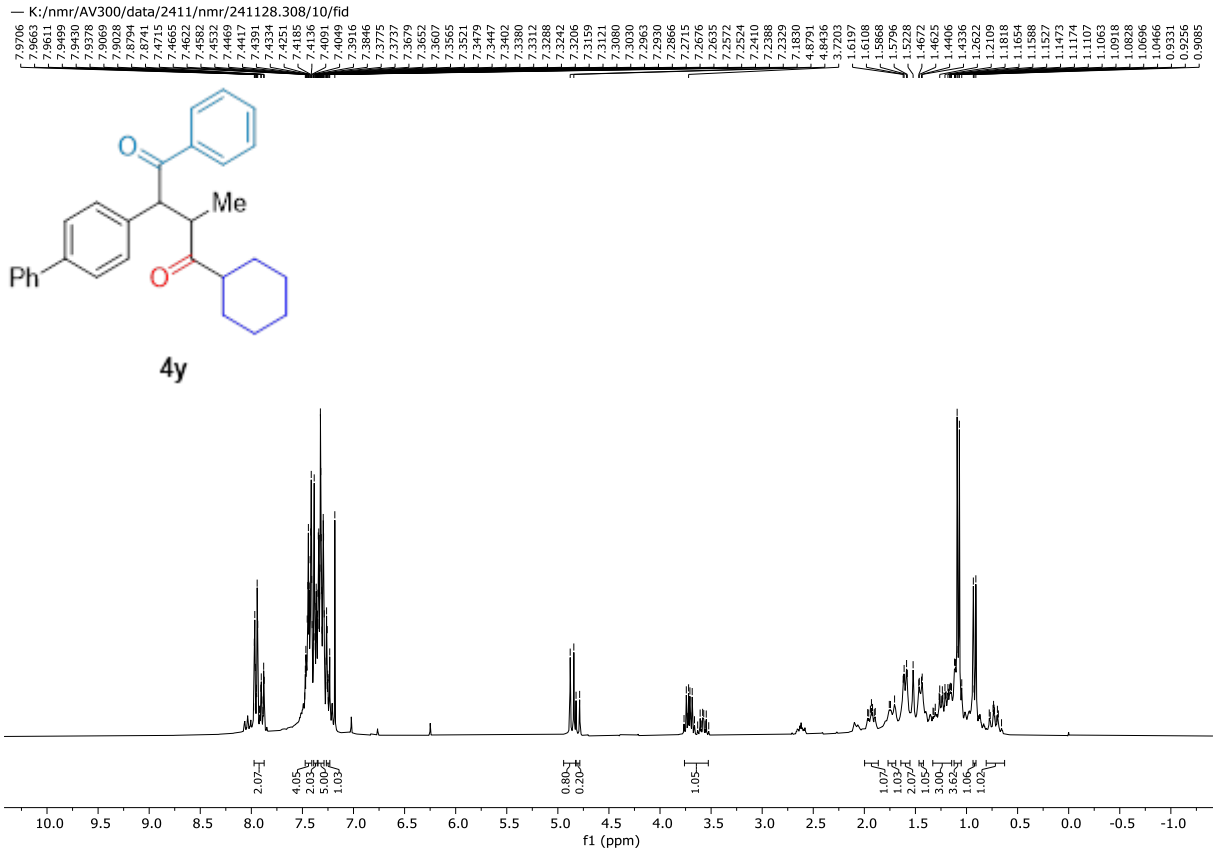
4x ^{1}H NMR (300 MHz, $CDCl_3$)



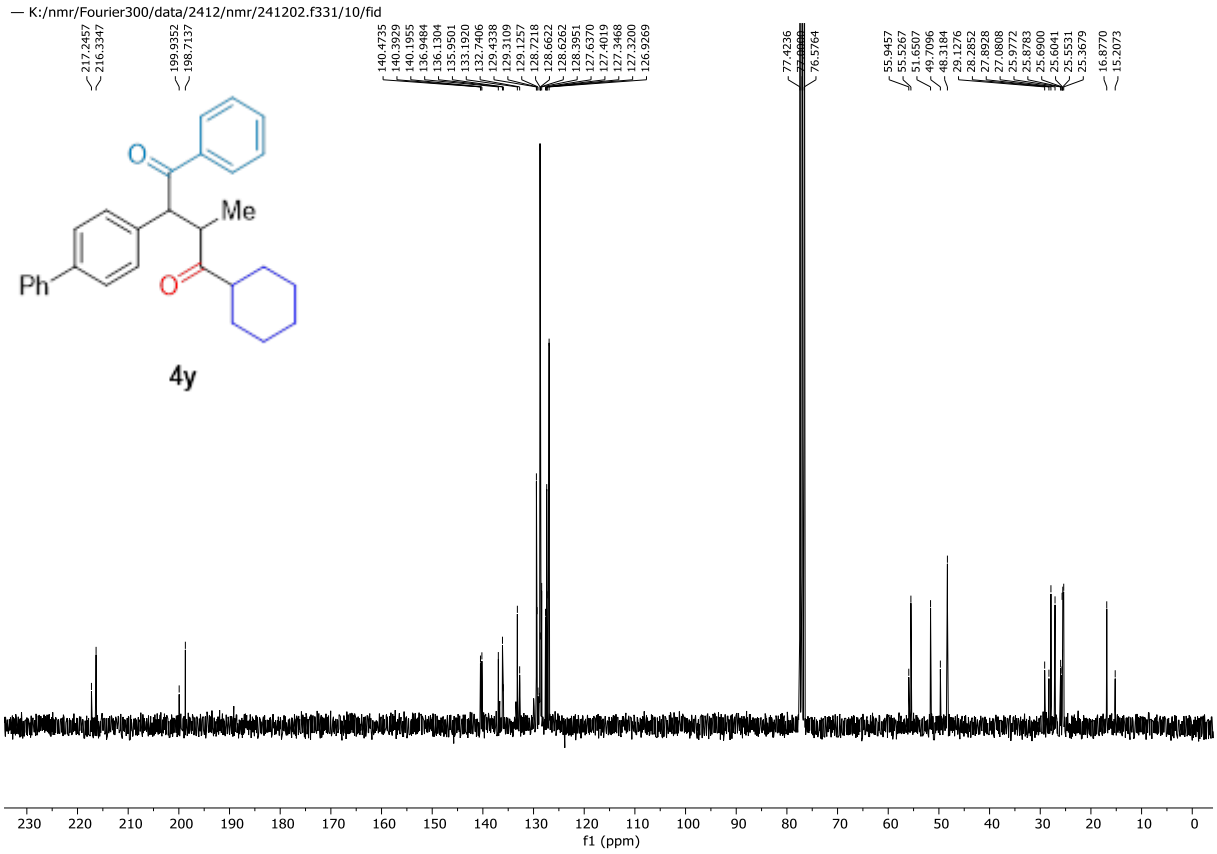
4x ^{13}C NMR (75 MHz, $CDCl_3$)



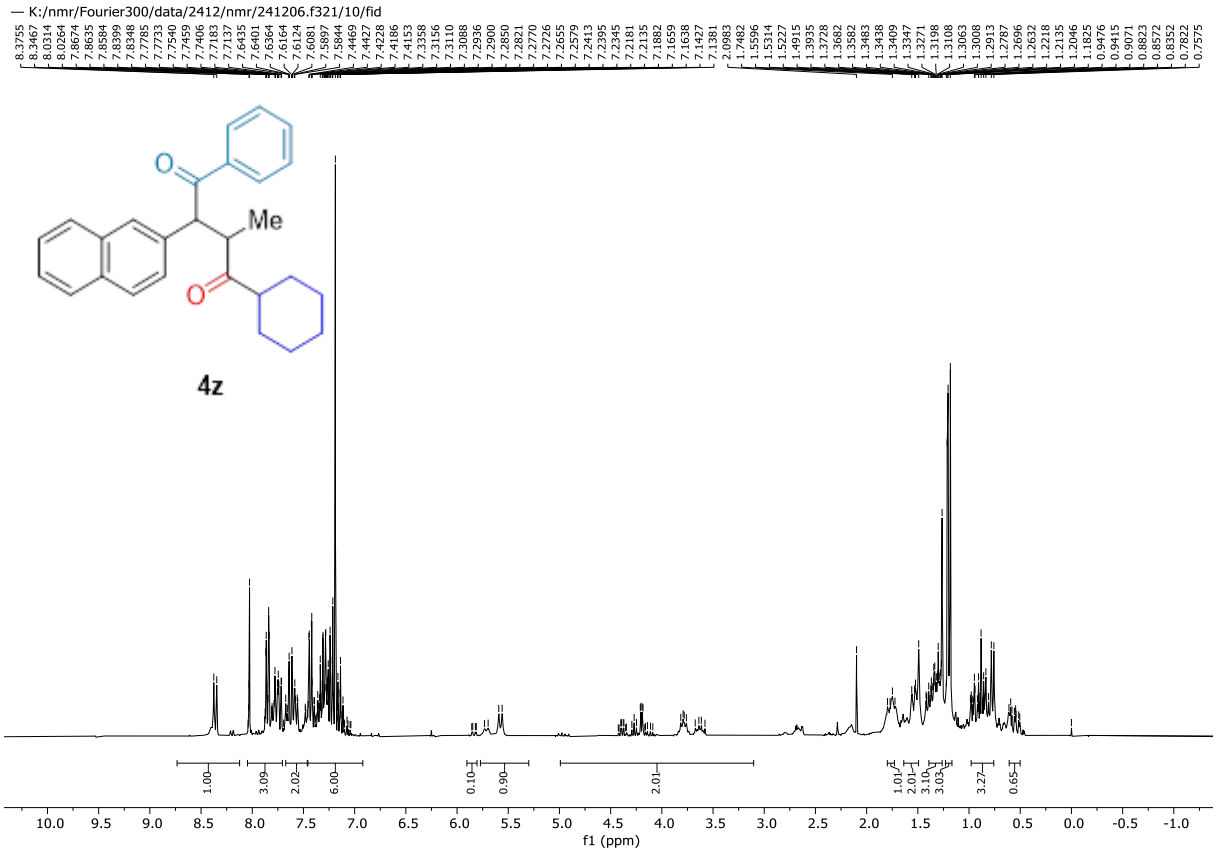
4y ^{1}H NMR (300 MHz, $CDCl_3$)



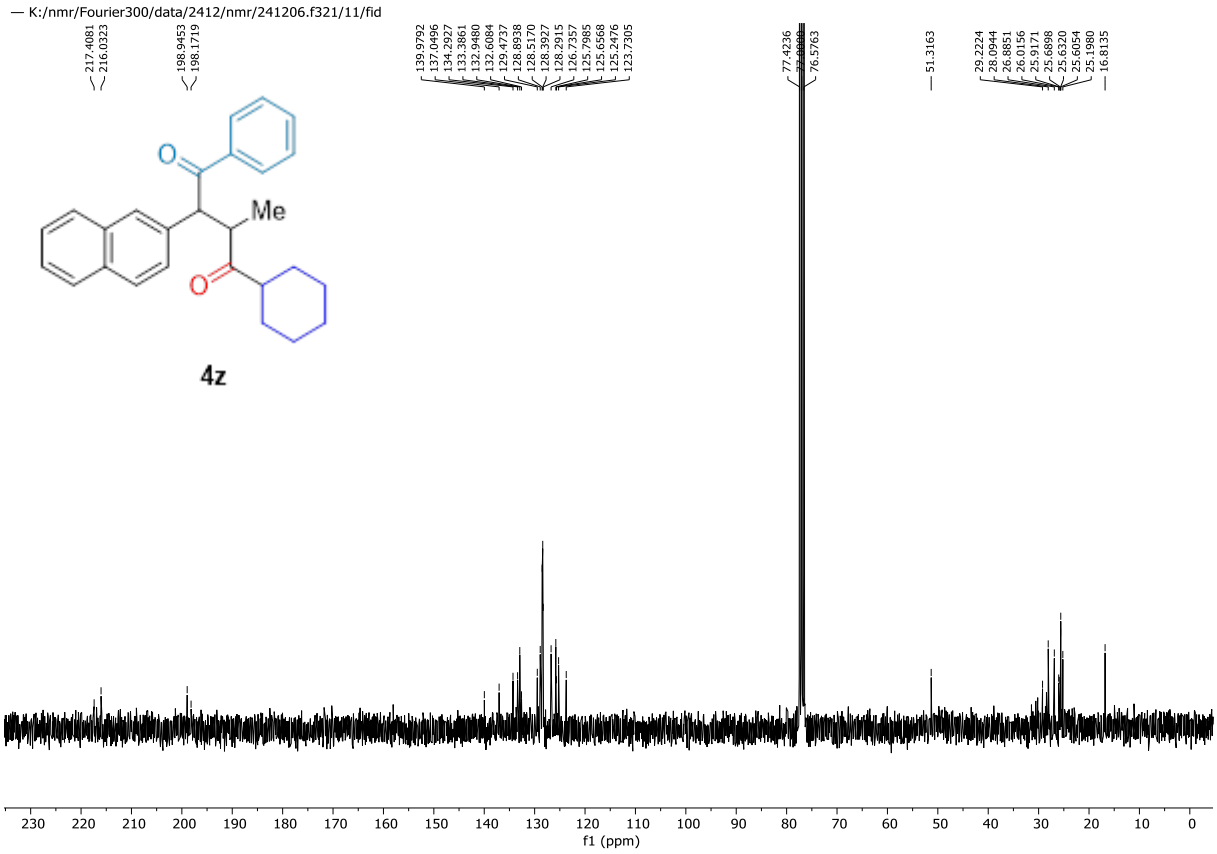
4y ^{13}C NMR (75 MHz, $CDCl_3$)



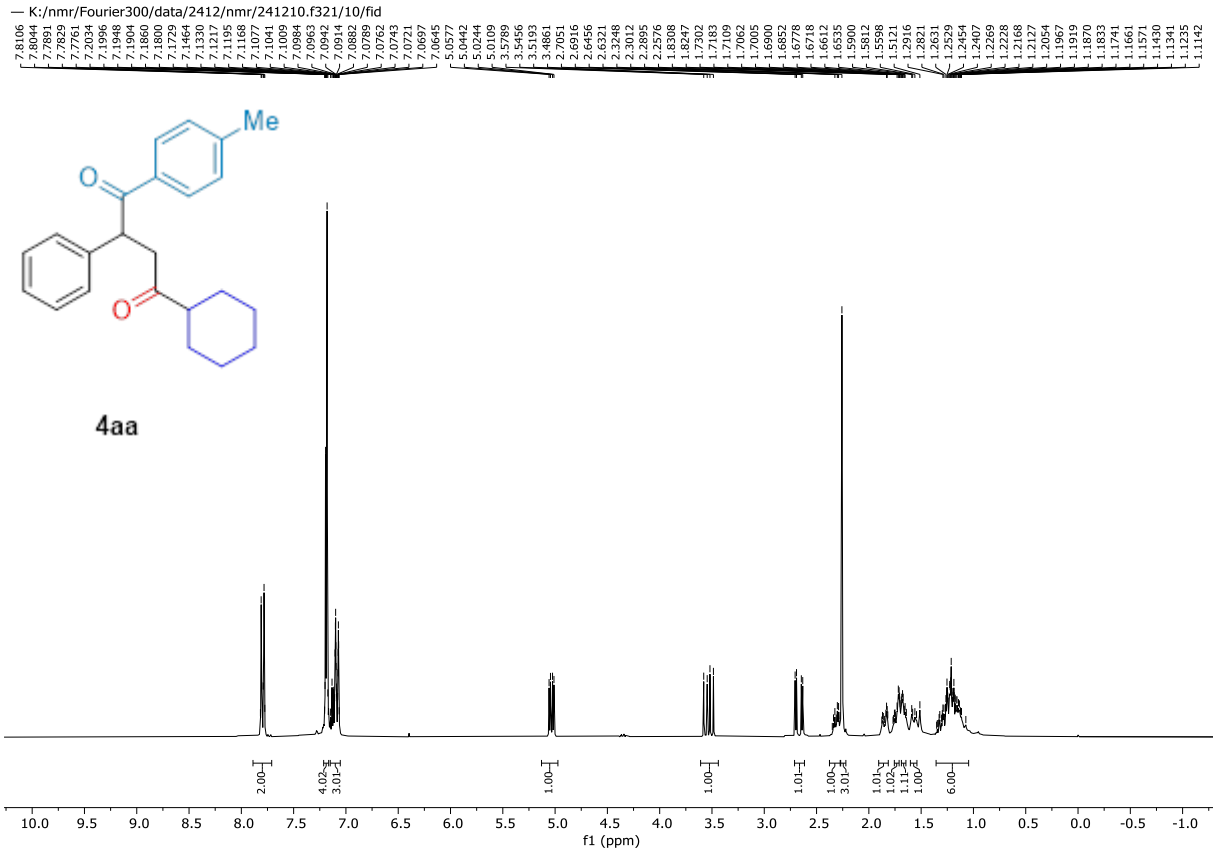
4z ^{1}H NMR (300 MHz, $CDCl_3$)



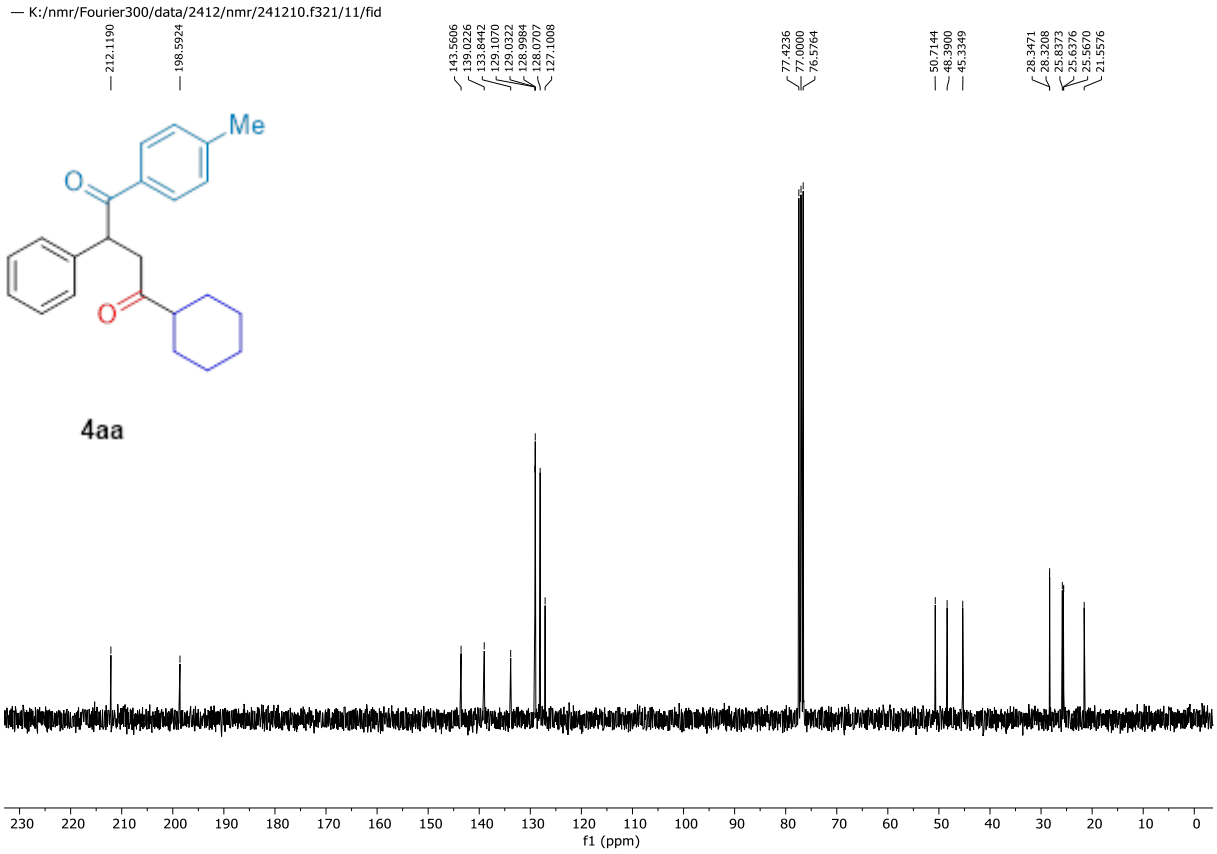
4z ^{13}C NMR (75 MHz, $CDCl_3$)



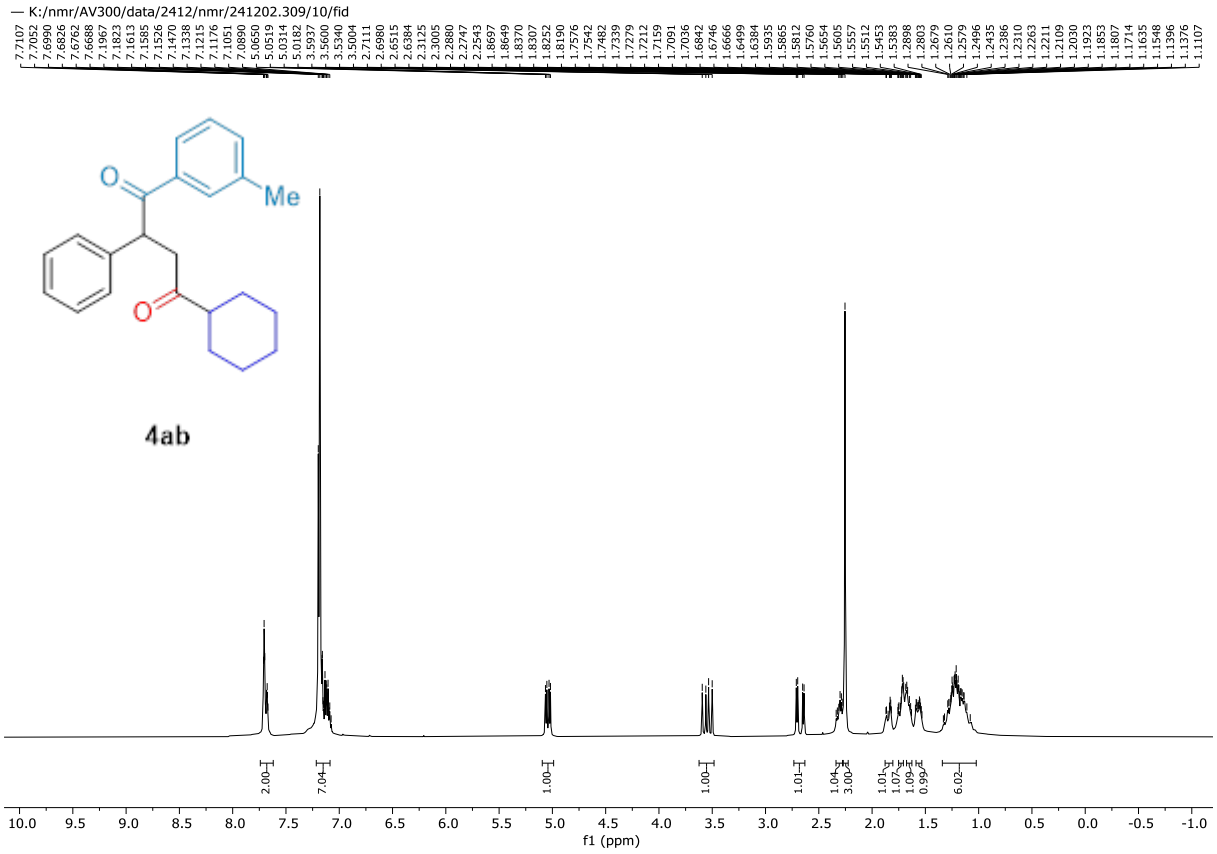
4aa ^{1}H NMR (300 MHz, $CDCl_3$)



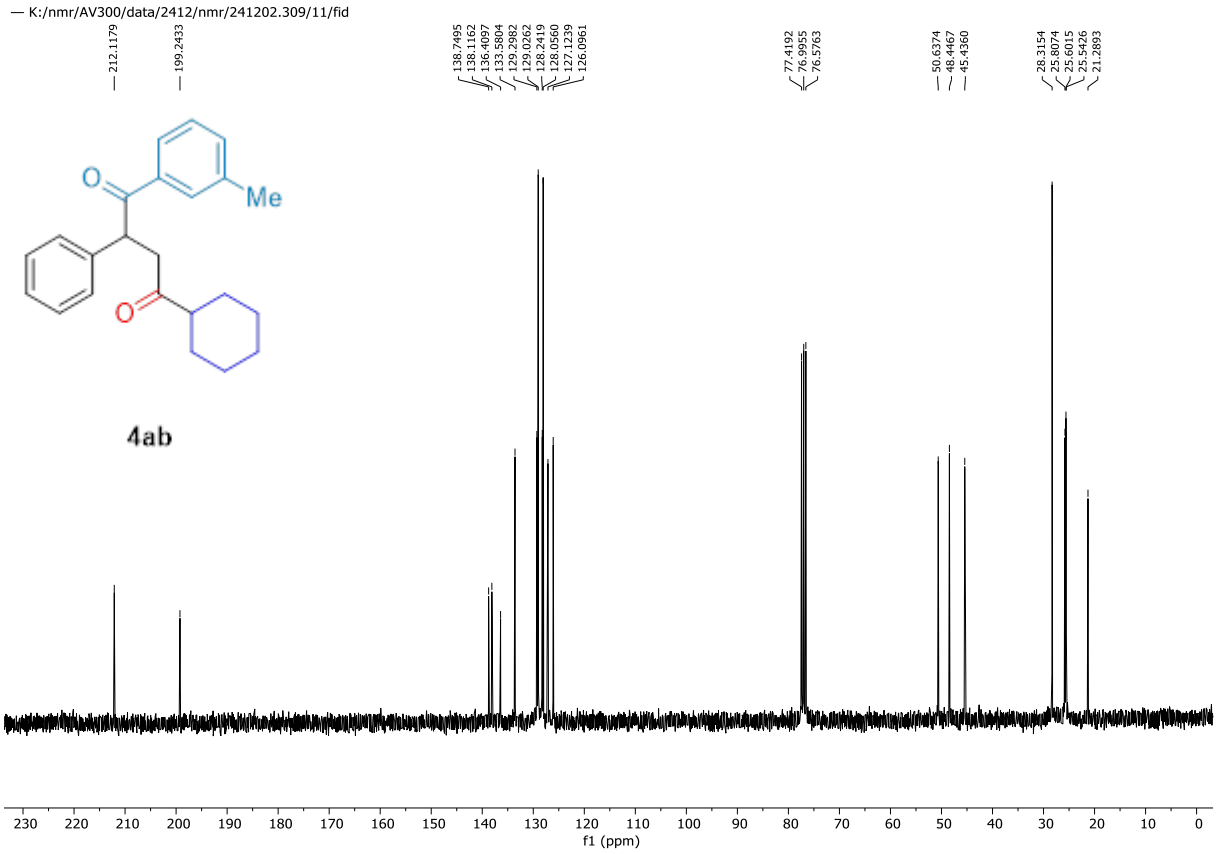
4aa ^{13}C NMR (75 MHz, $CDCl_3$)



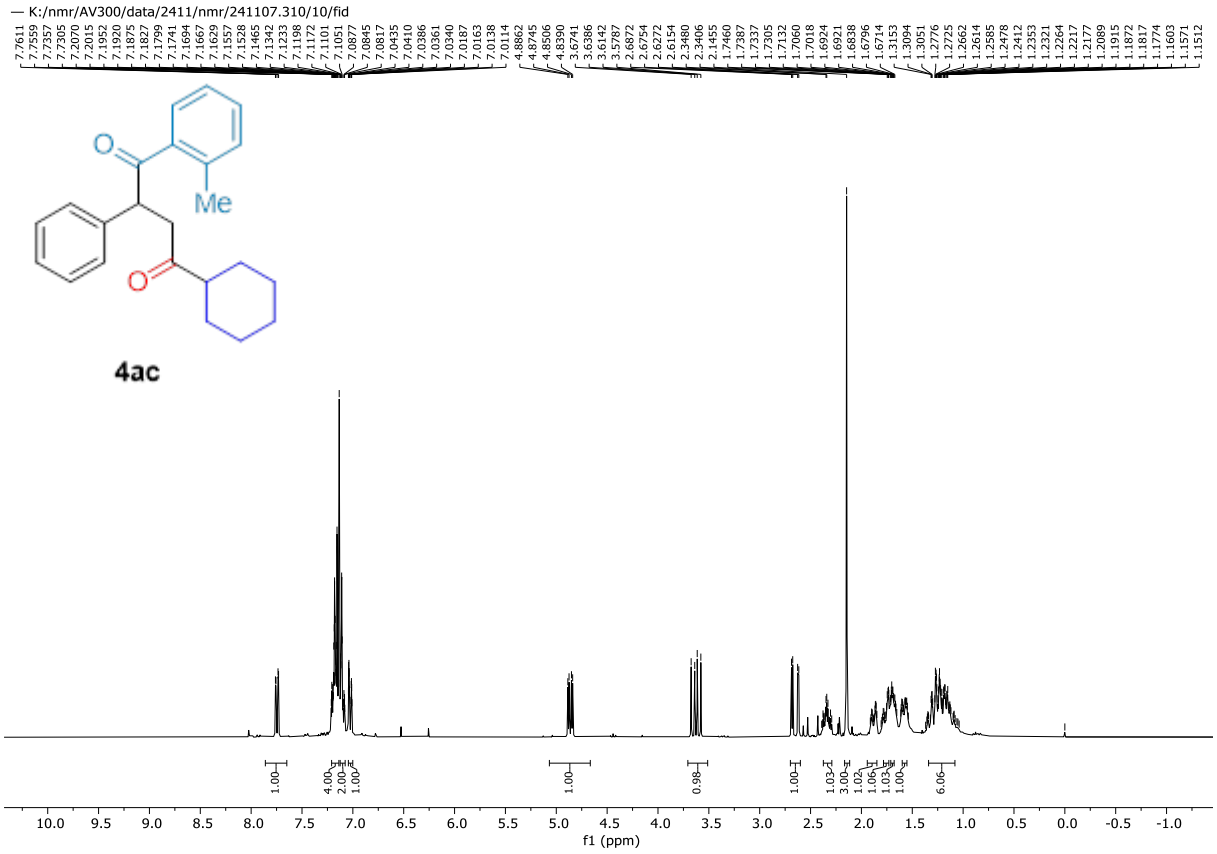
4ab ^{1}H NMR (300 MHz, $CDCl_3$)



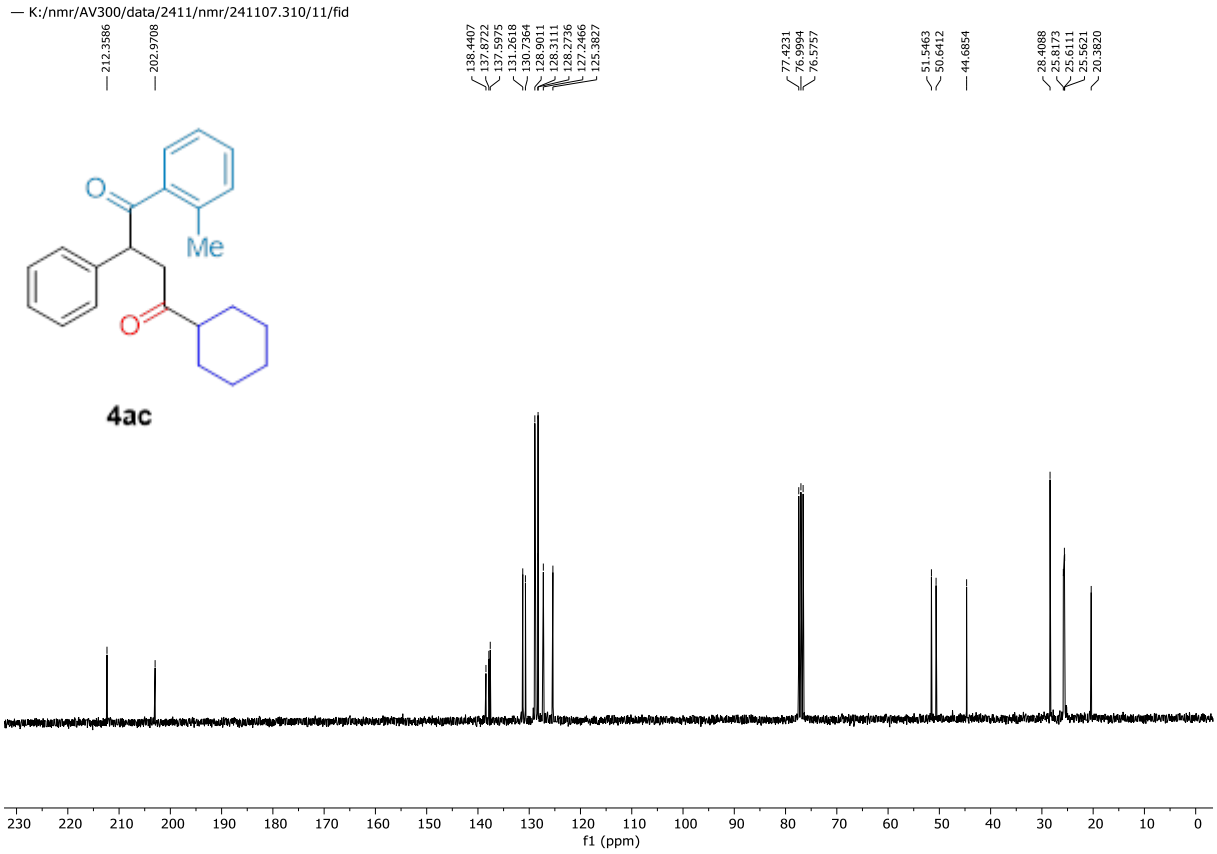
4ab ^{13}C NMR (75 MHz, $CDCl_3$)



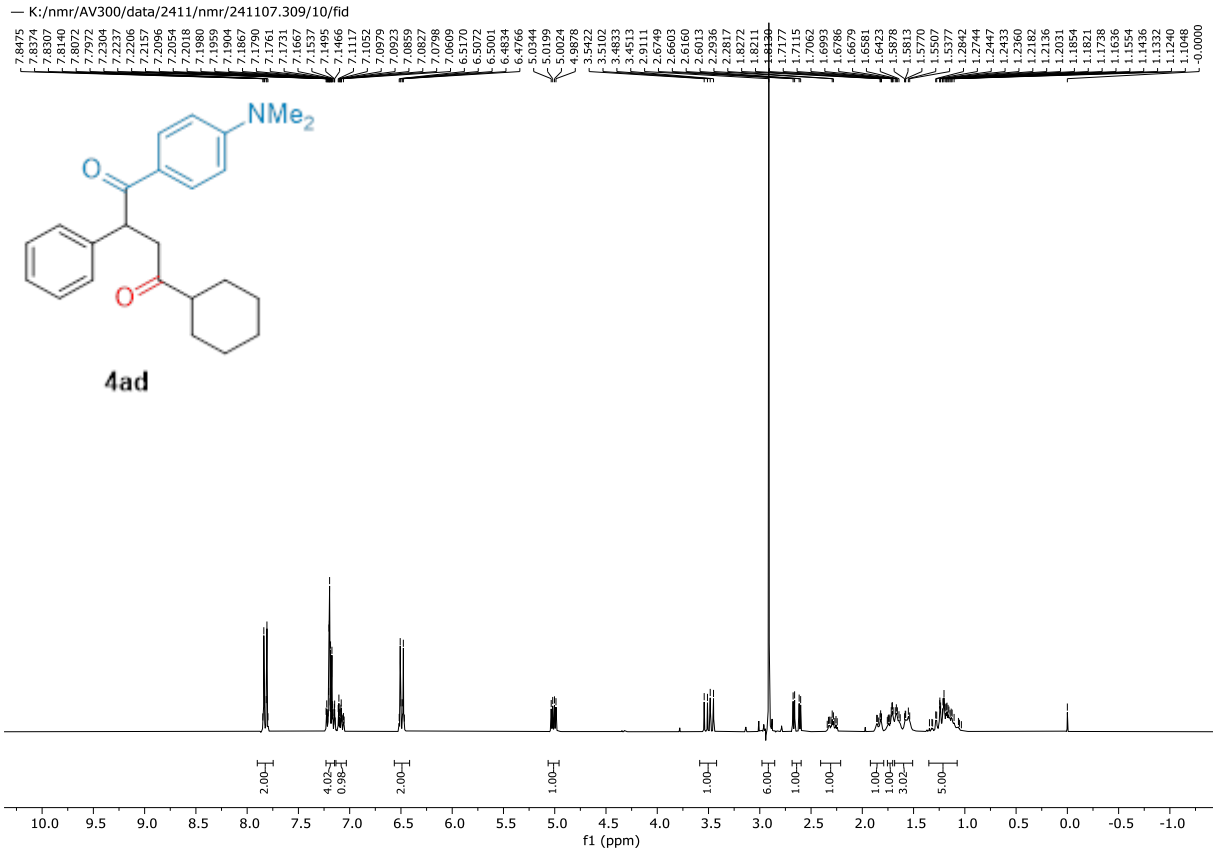
4ac ^{1}H NMR (300 MHz, $CDCl_3$)



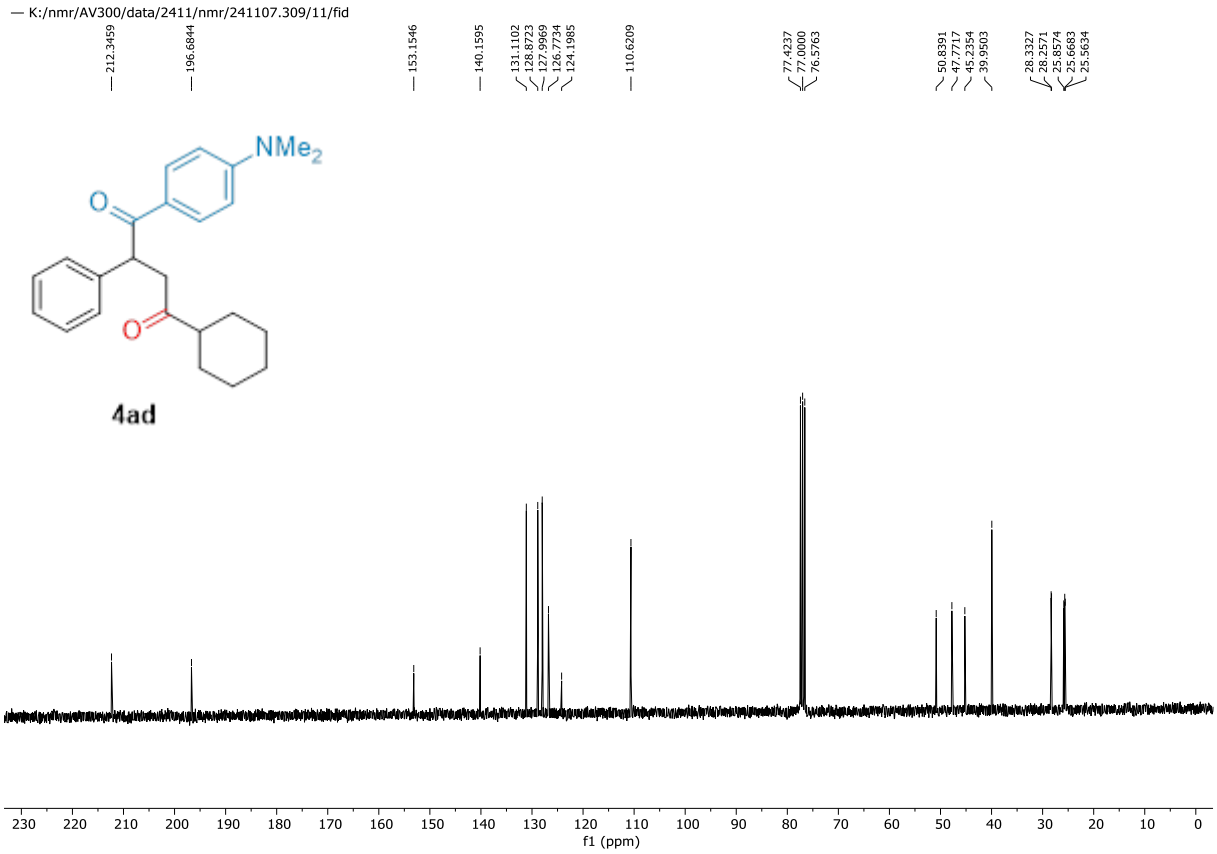
4ac ^{13}C NMR (75 MHz, $CDCl_3$)



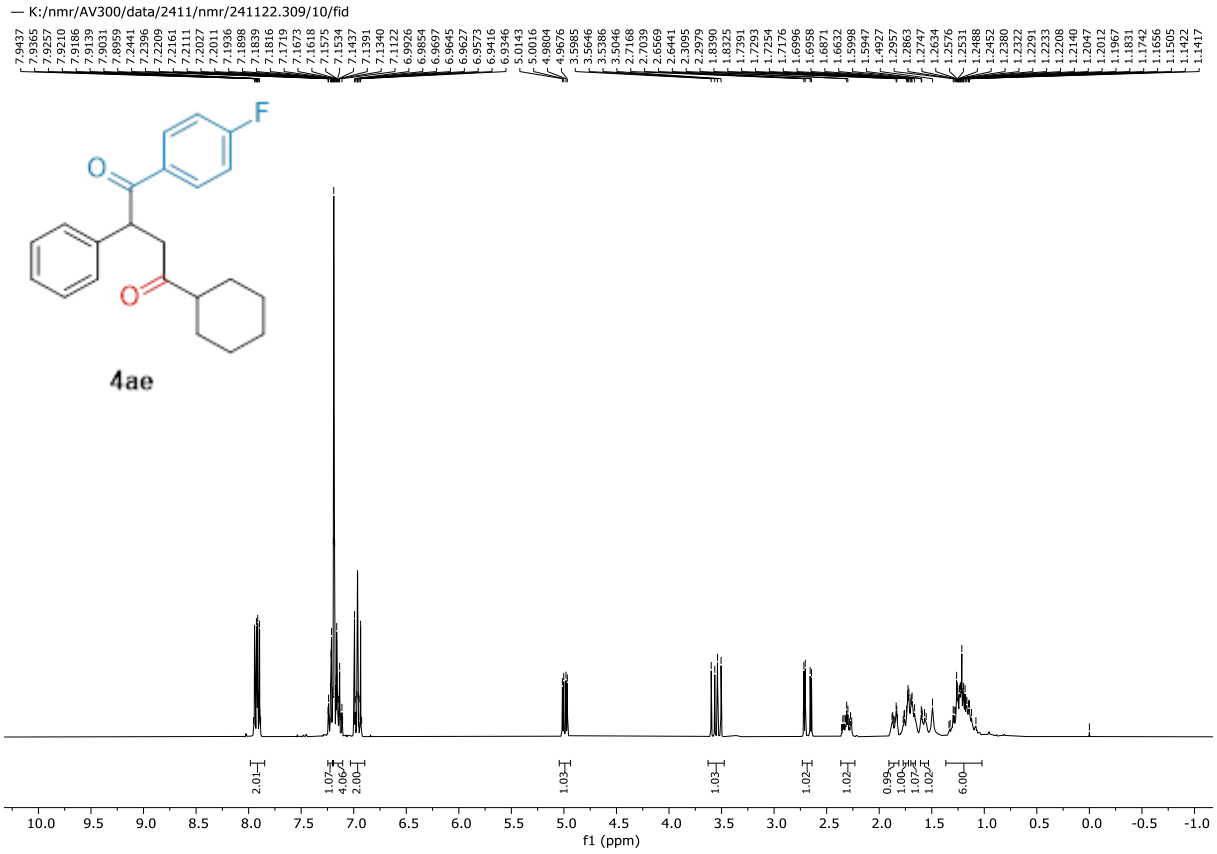
4ad ^{1}H NMR (300 MHz, $CDCl_3$)



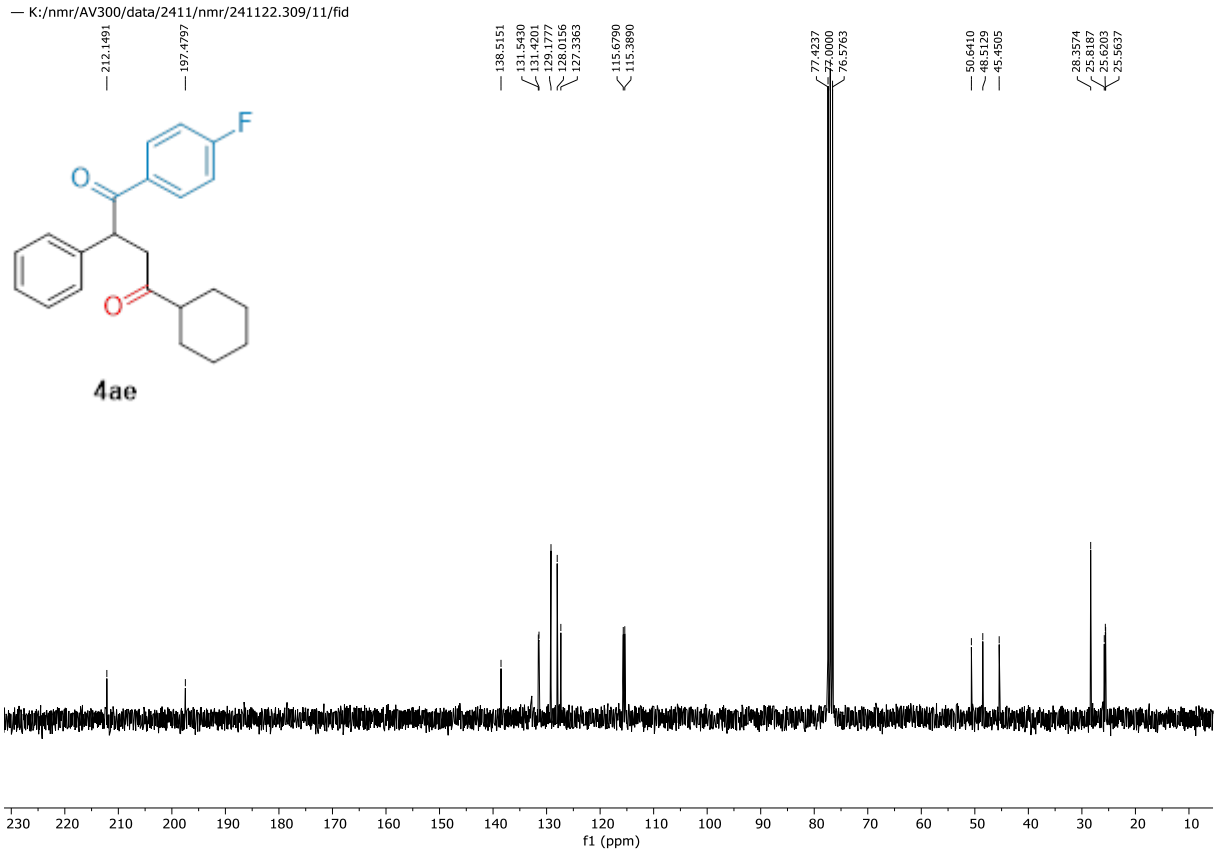
4ad ^{13}C NMR (75 MHz, $CDCl_3$)



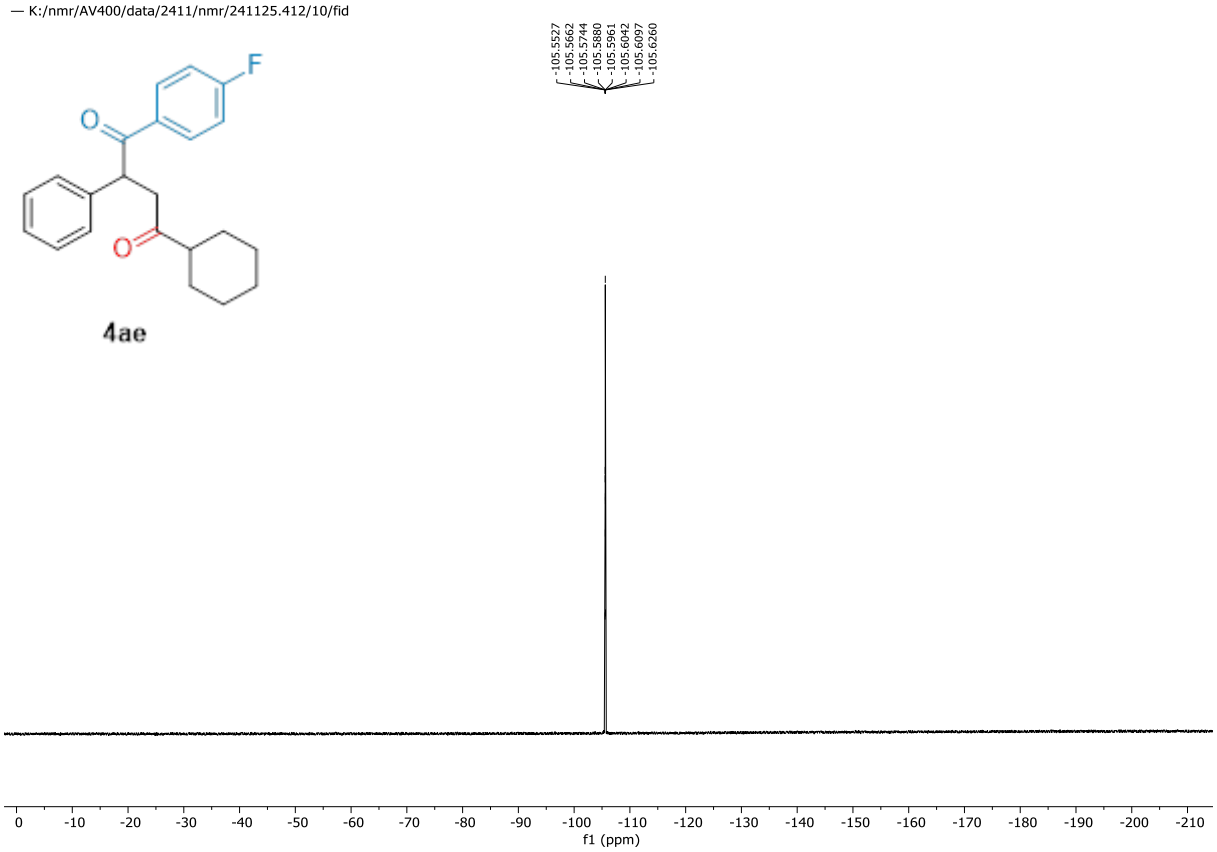
4ae ^{1}H NMR (300 MHz, $CDCl_3$)



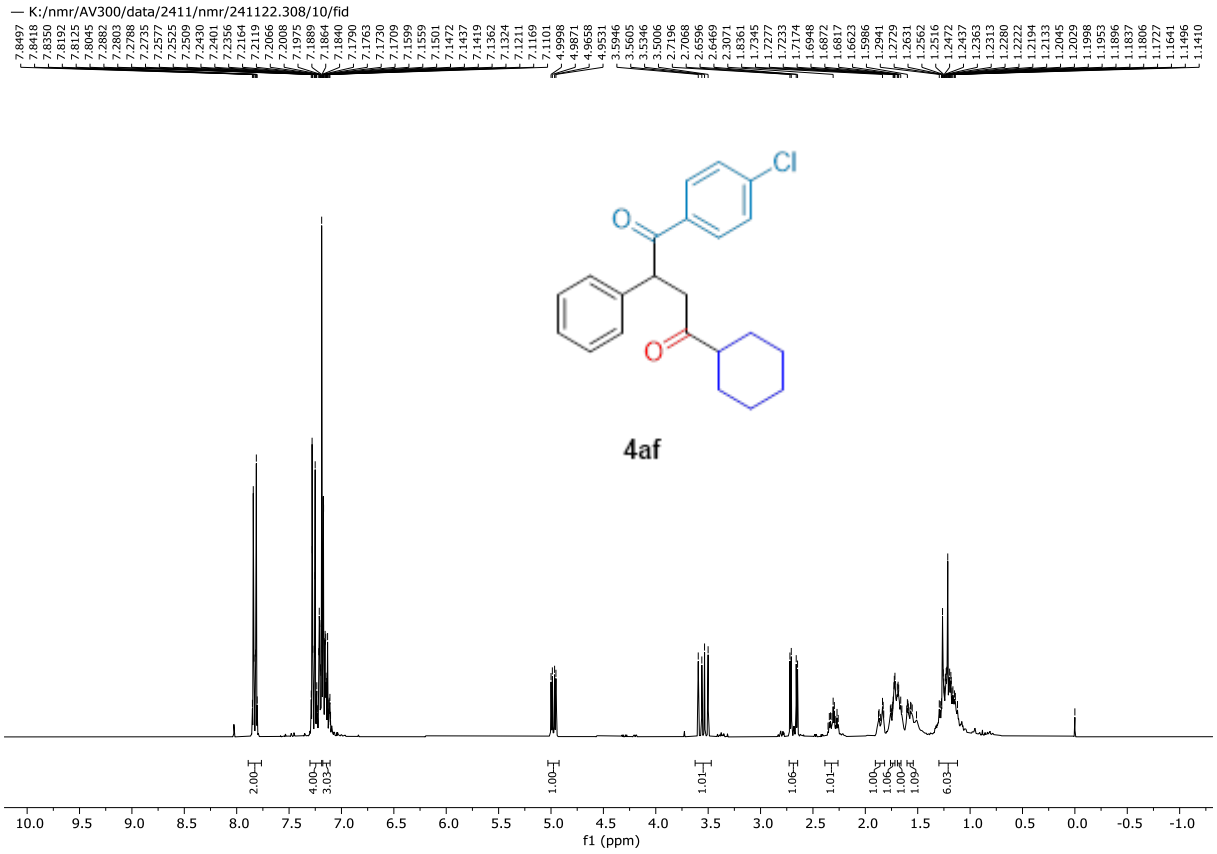
4ae ^{13}C NMR (75 MHz, $CDCl_3$)



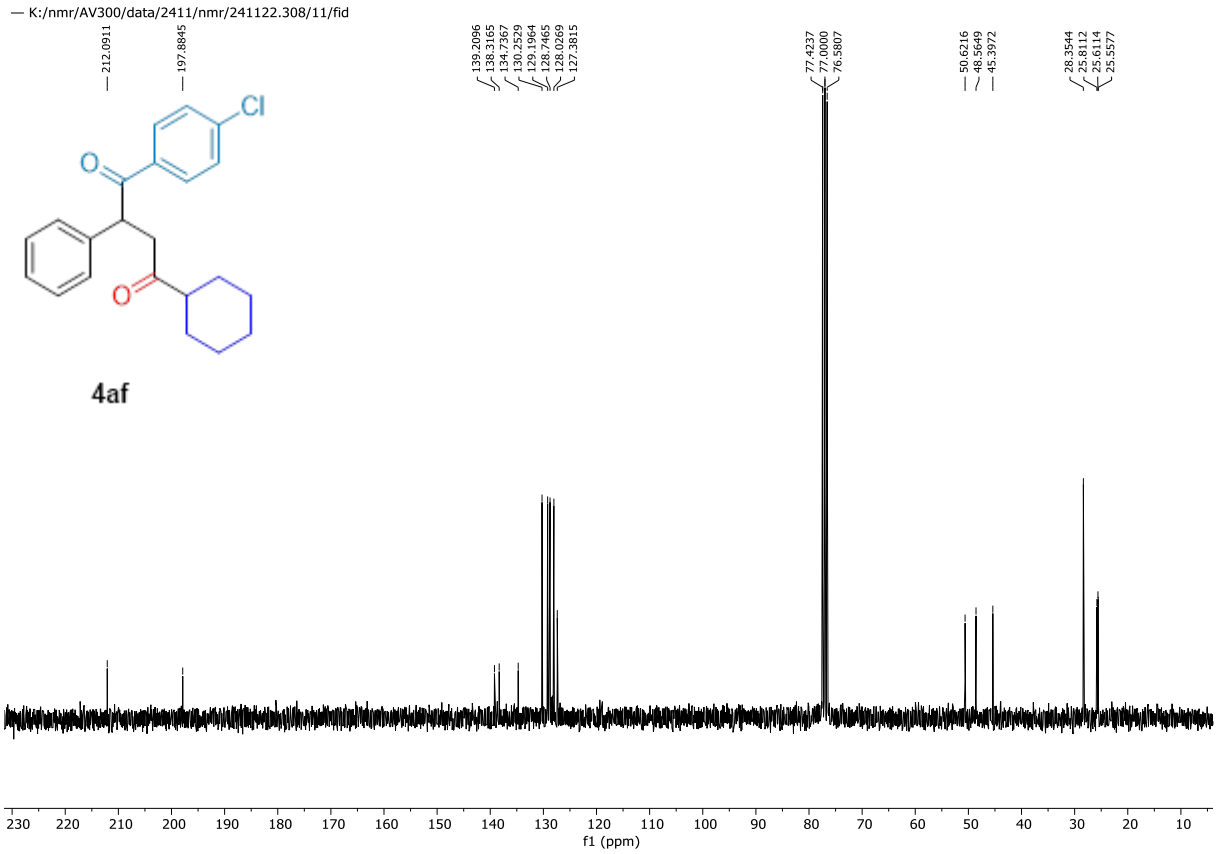
4ae ^{13}F NMR (282 MHz, $CDCl_3$)



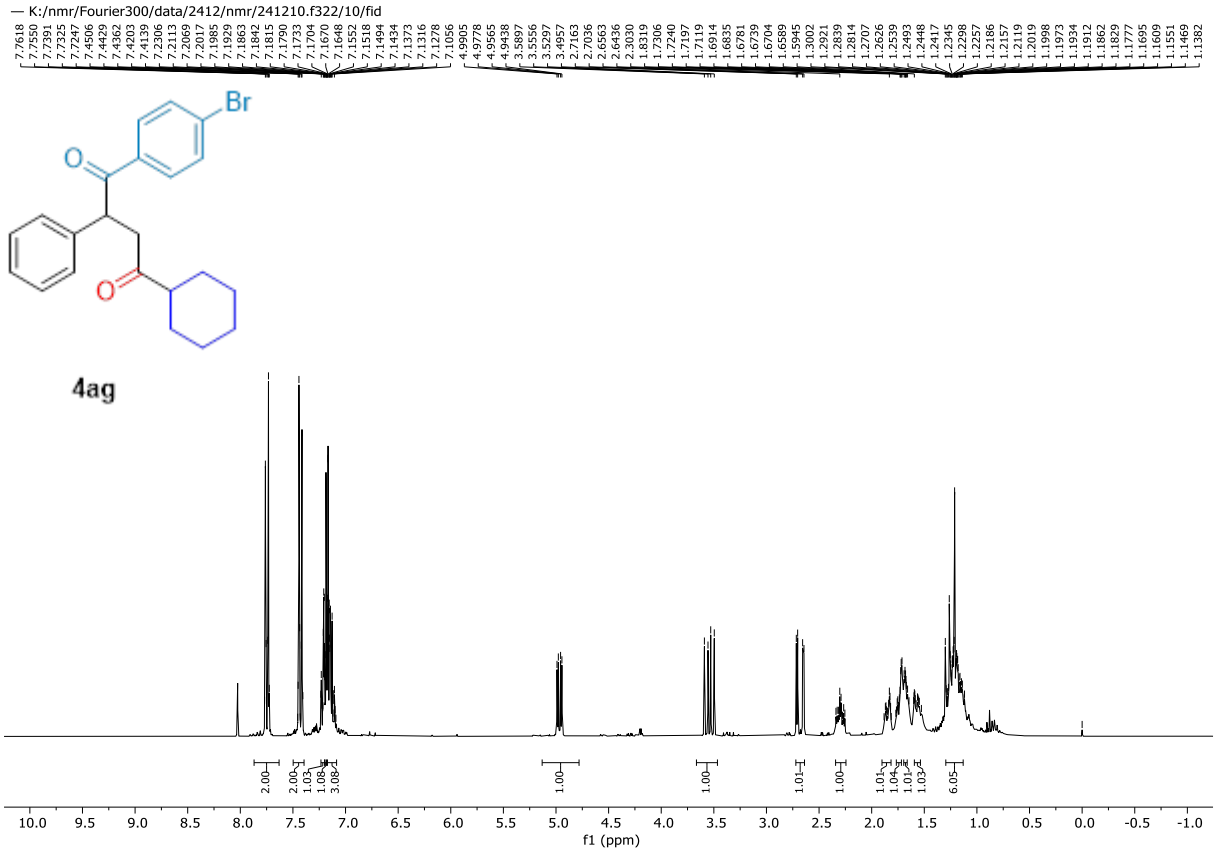
4af ^{1}H NMR (300 MHz, $CDCl_3$)



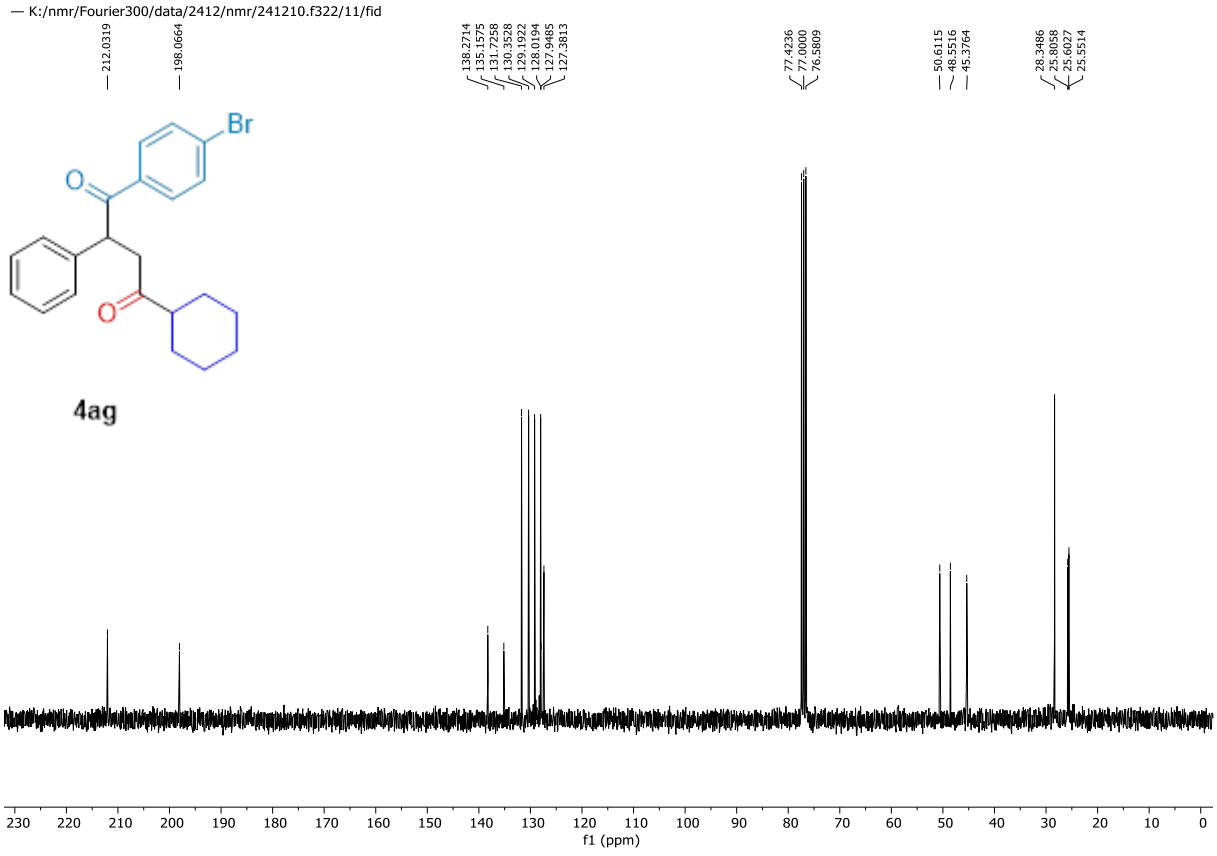
4af ^{13}C NMR (75 MHz, $CDCl_3$)



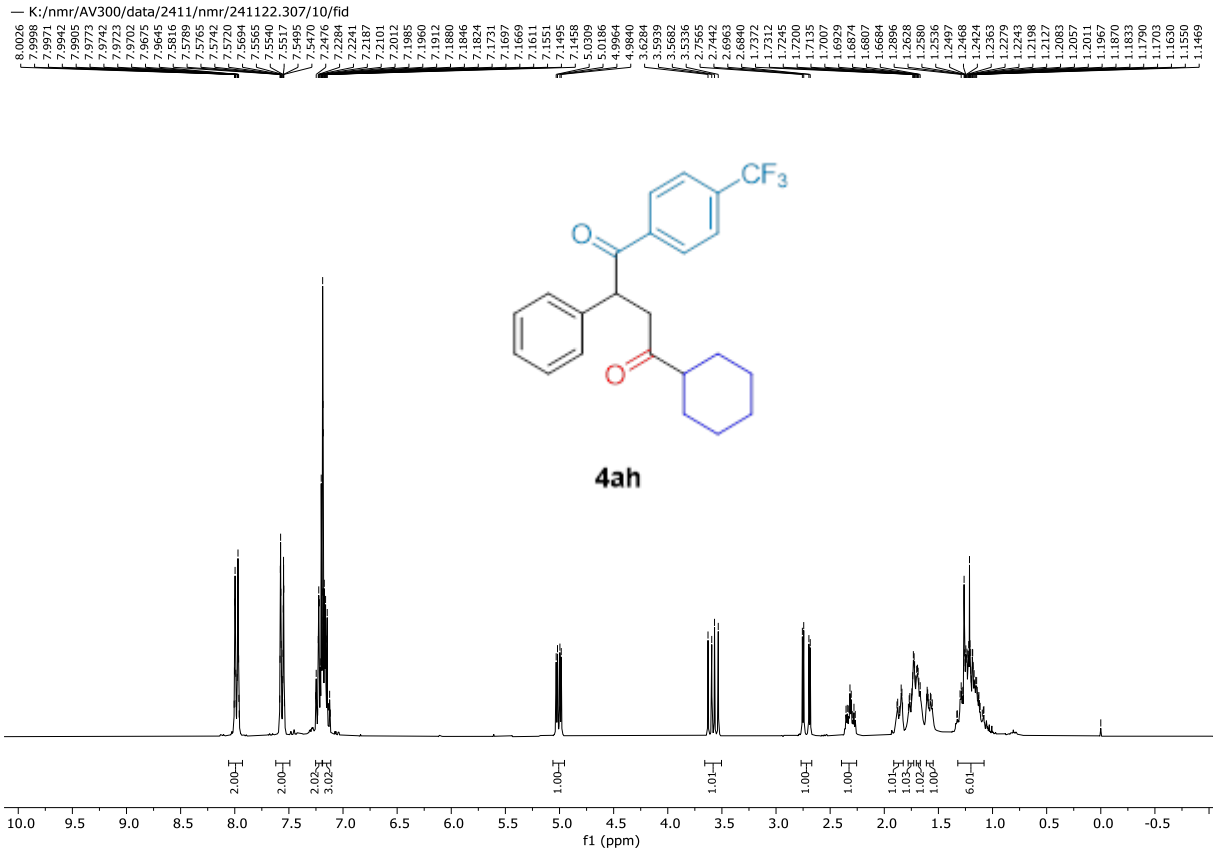
4ag ^{1}H NMR (300 MHz, $CDCl_3$)



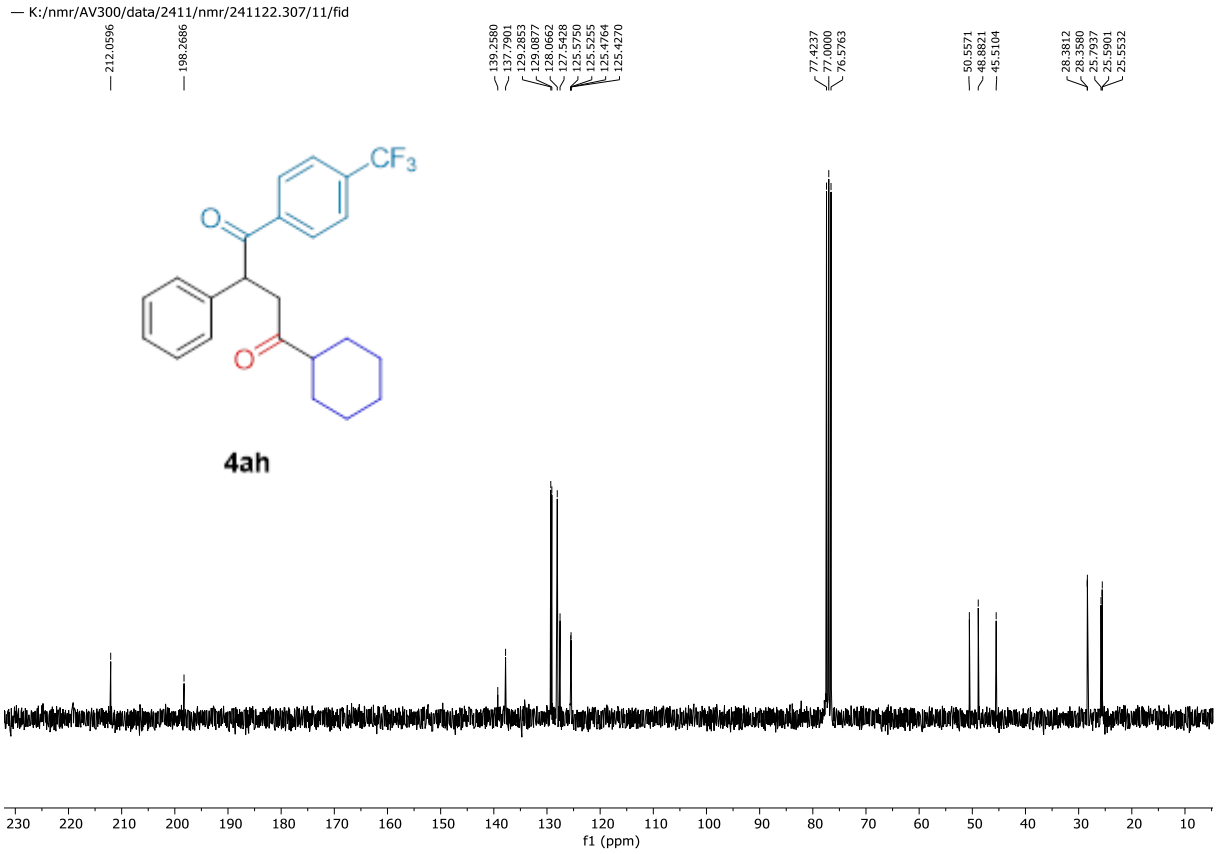
4ag ^{13}C NMR (75 MHz, $CDCl_3$)



4ah ^{1}H NMR (300 MHz, $CDCl_3$)



4ah ^{13}C NMR (75 MHz, $CDCl_3$)



4i^{13}F NMR (282 MHz, $CDCl_3$)

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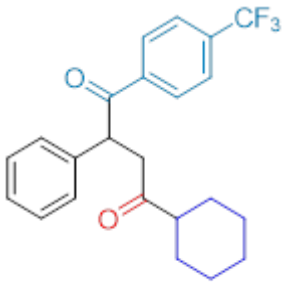
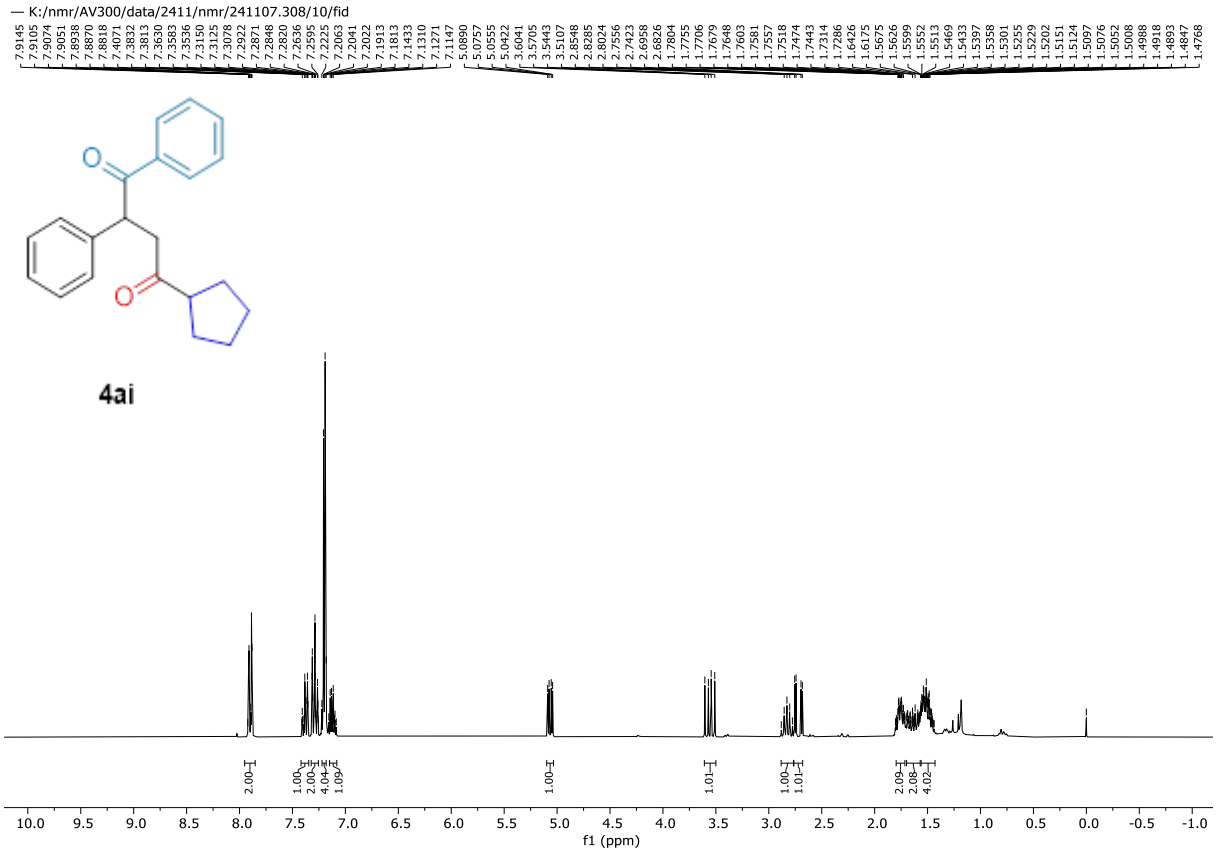
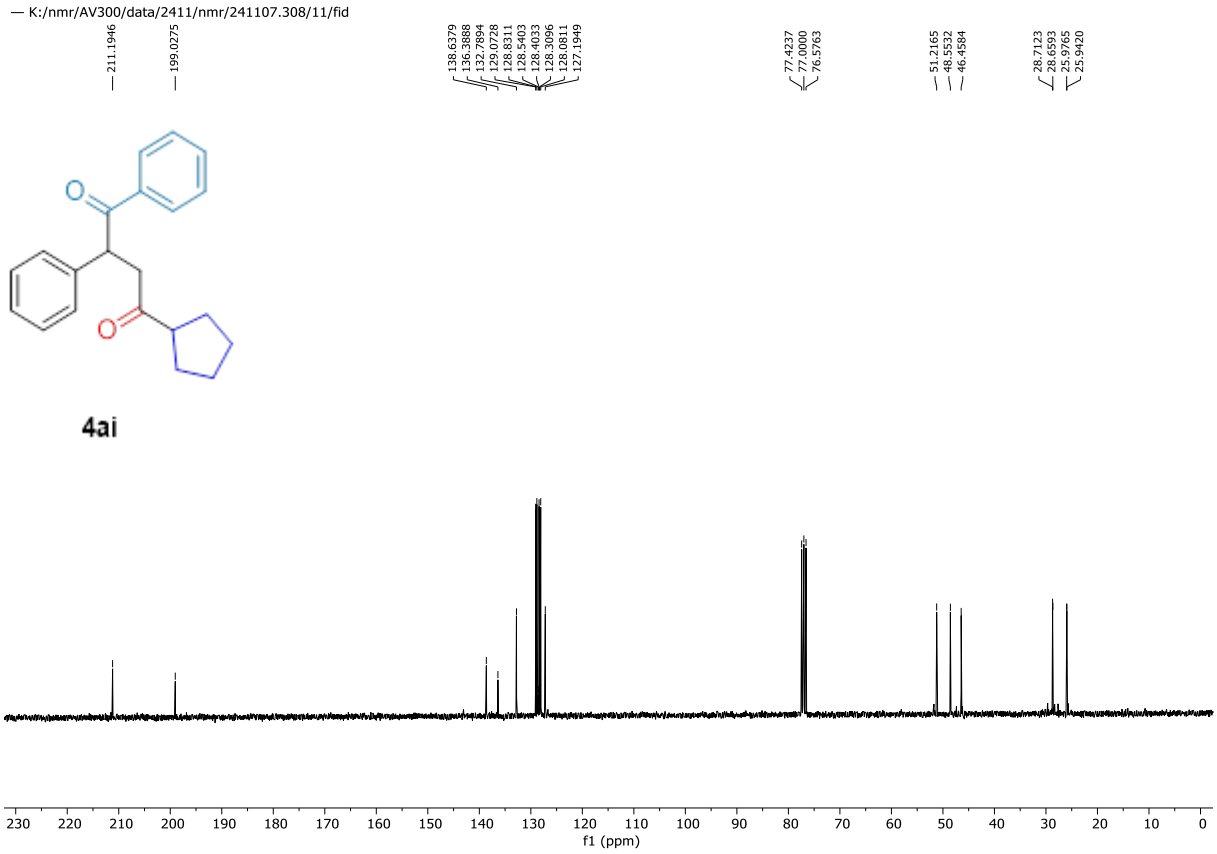


4ah

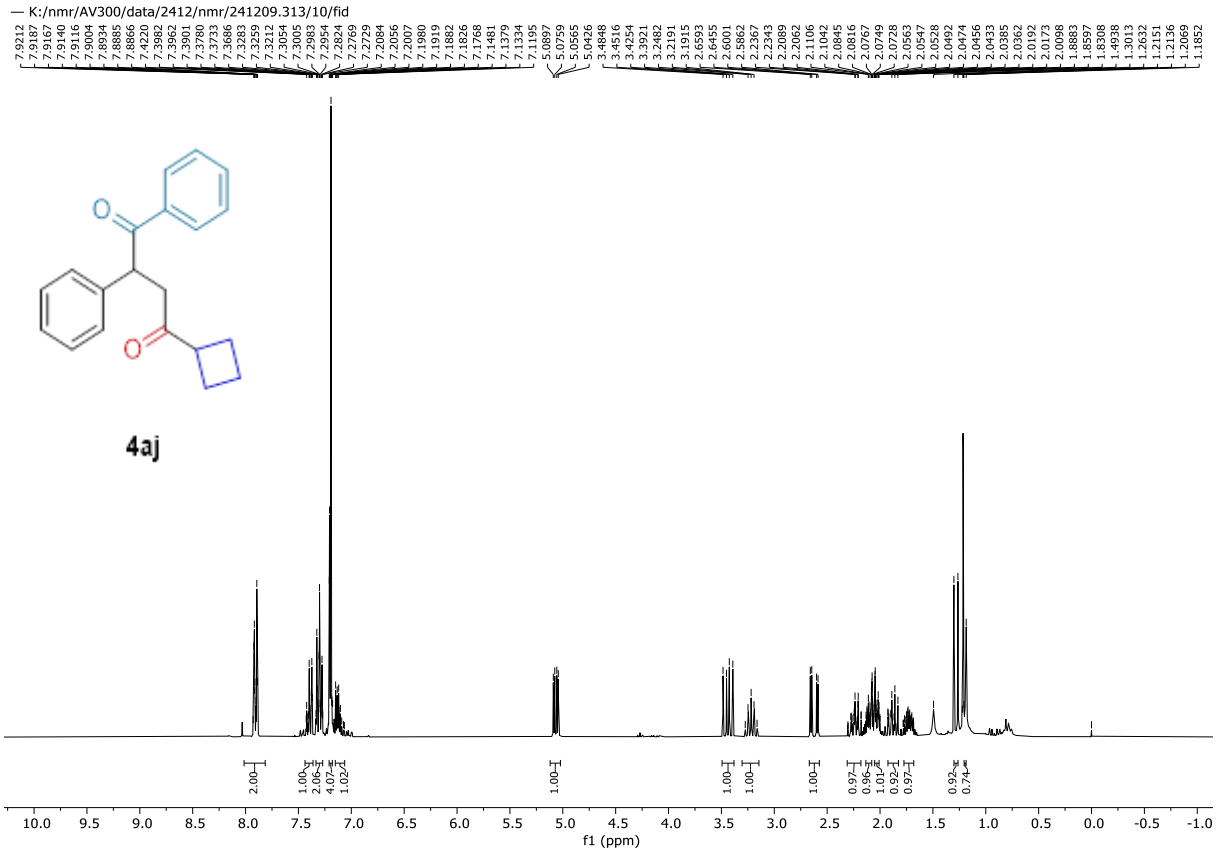
4ai ^{1}H NMR (300 MHz, $CDCl_3$)



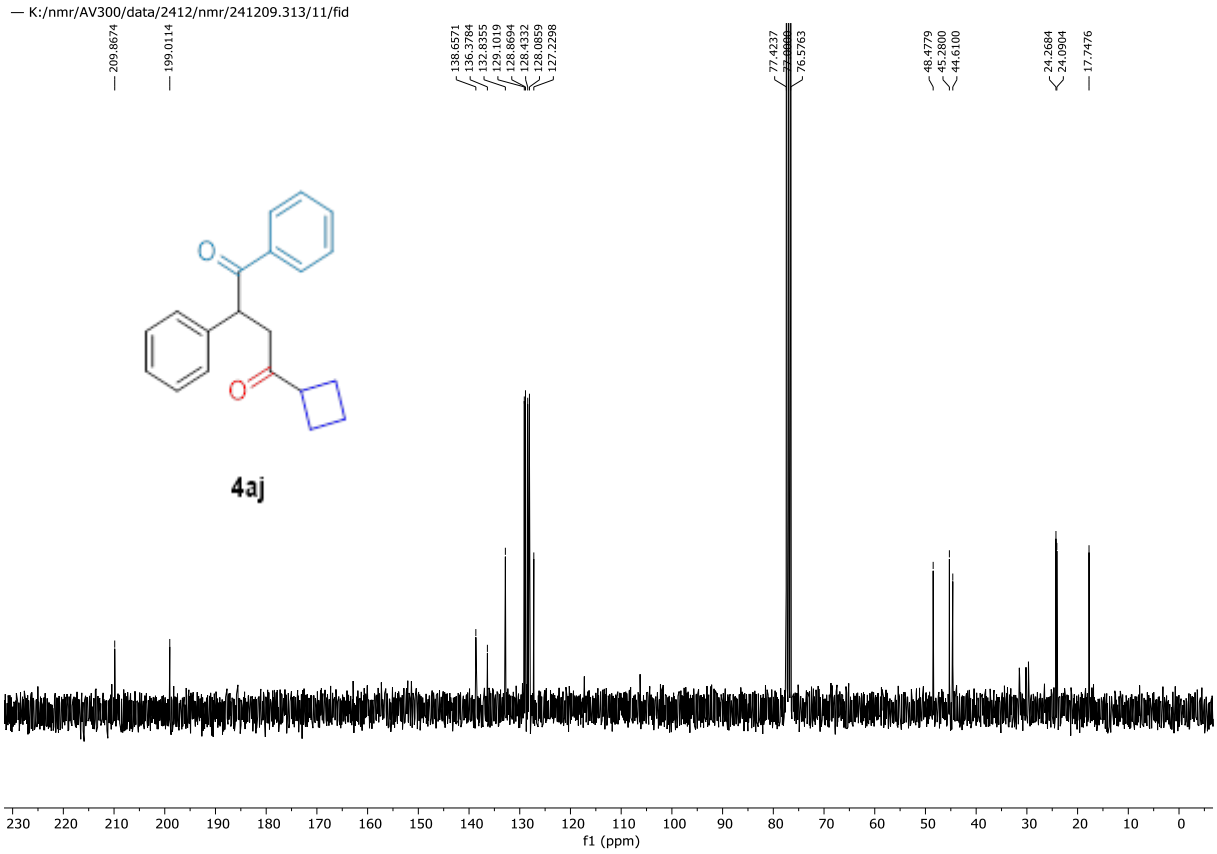
4ai ^{13}C NMR (75 MHz, $CDCl_3$)



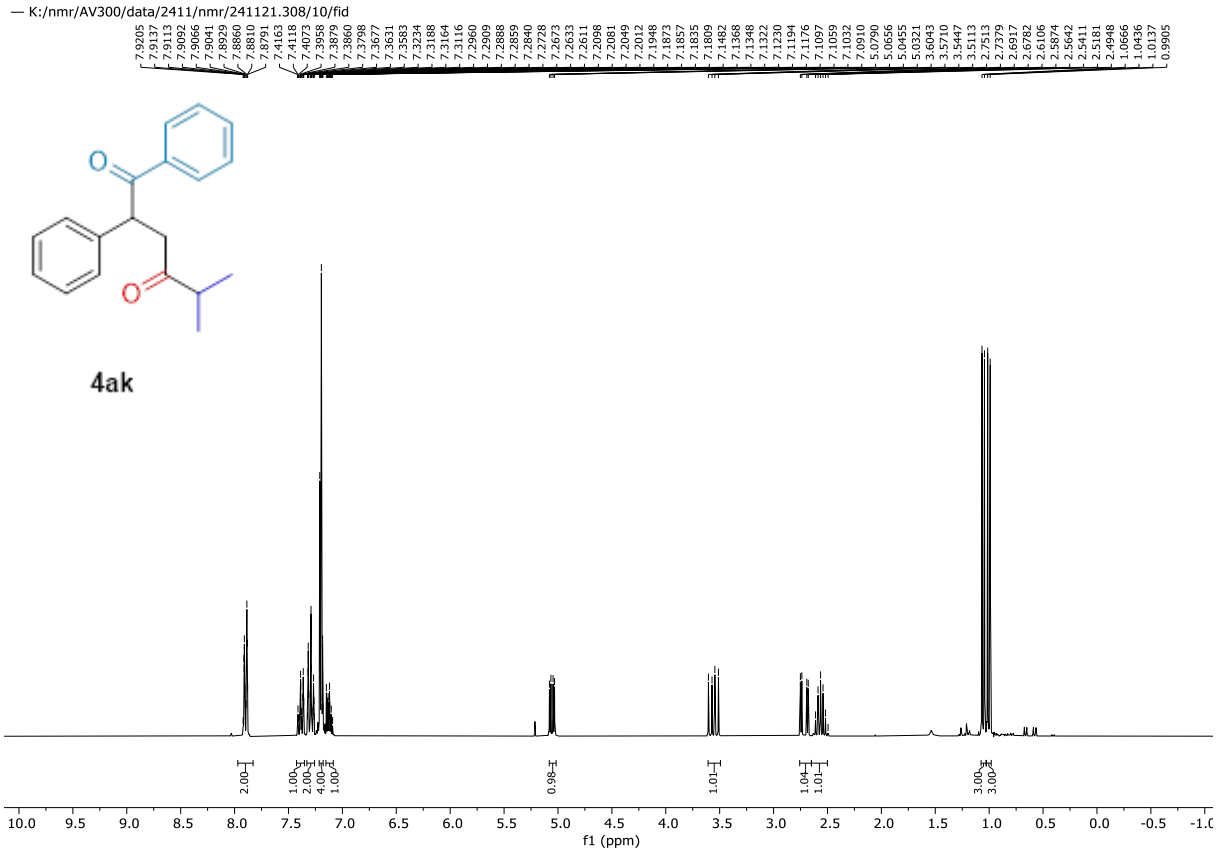
4aj ^{1}H NMR (300 MHz, $CDCl_3$)



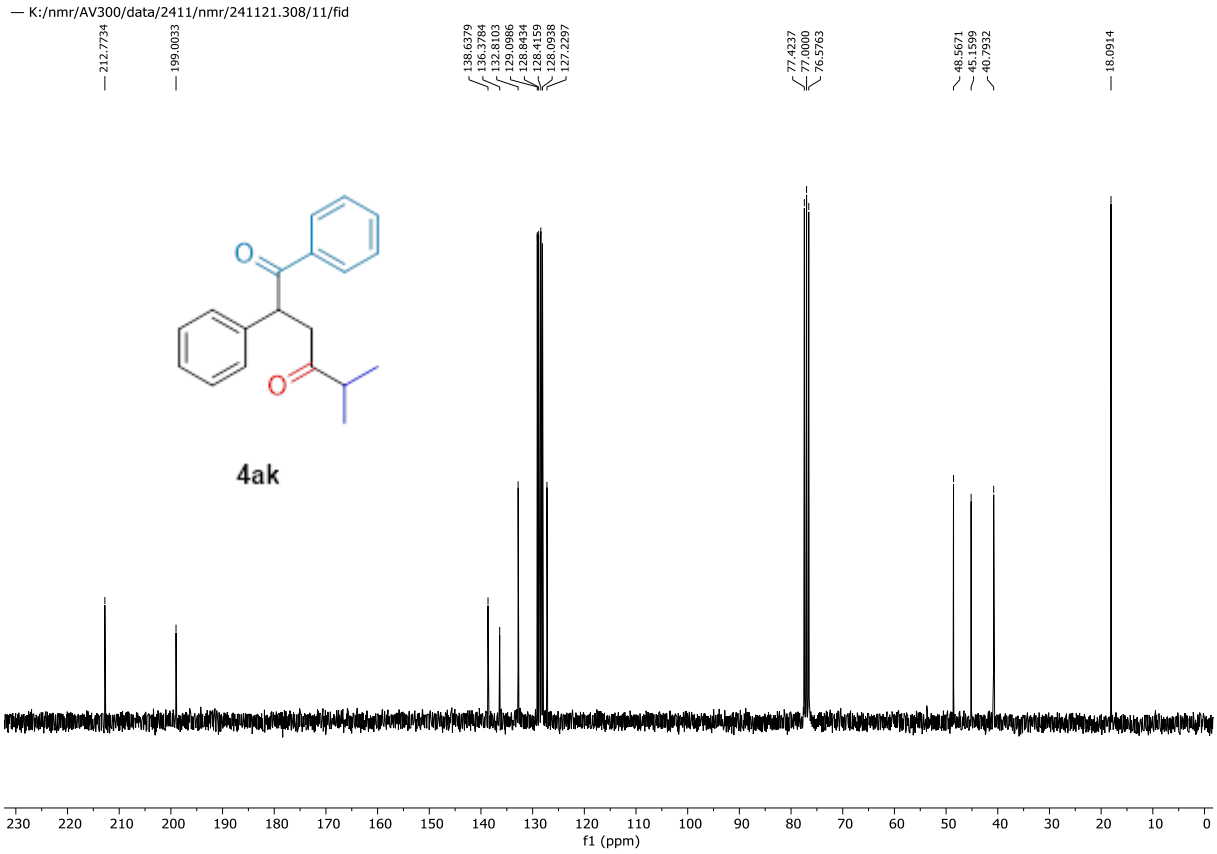
4aj ^{13}C NMR (75 MHz, $CDCl_3$)



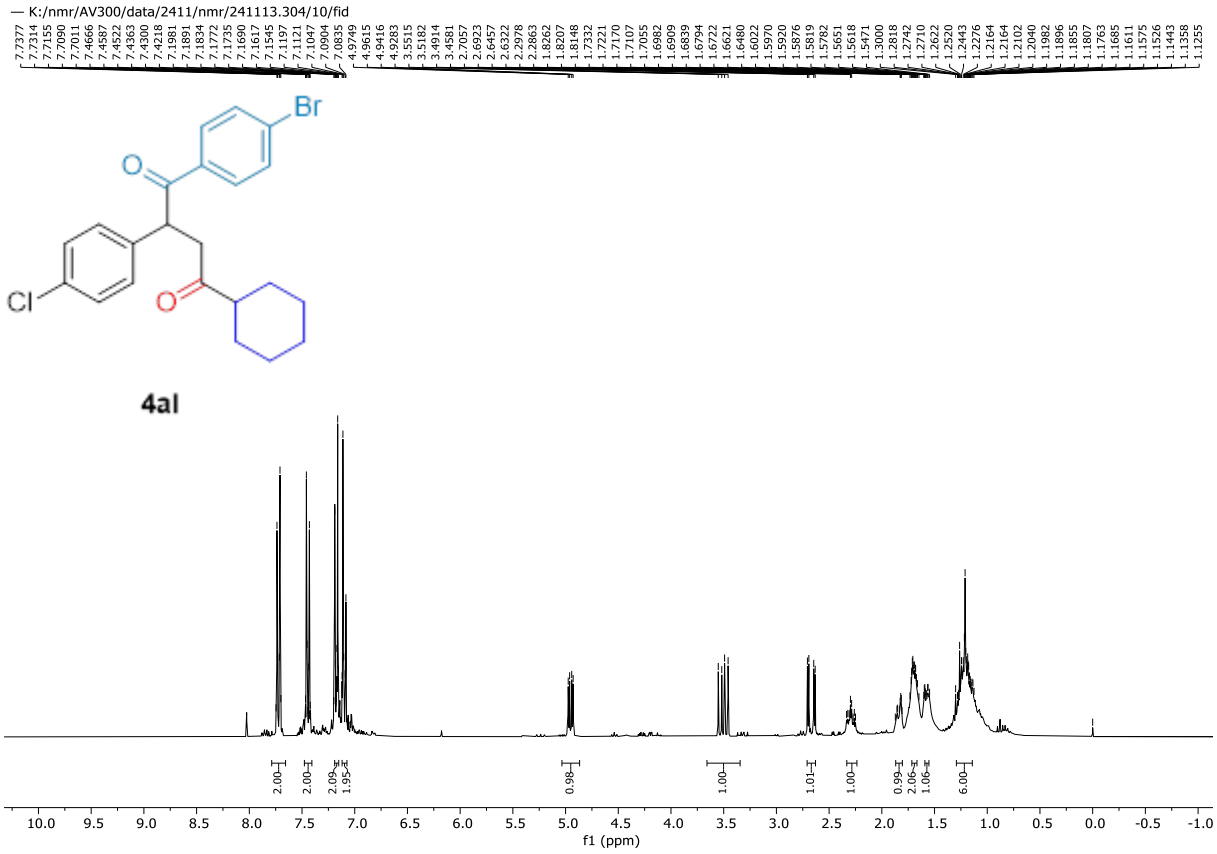
4ak ^{1}H NMR (300 MHz, $CDCl_3$)



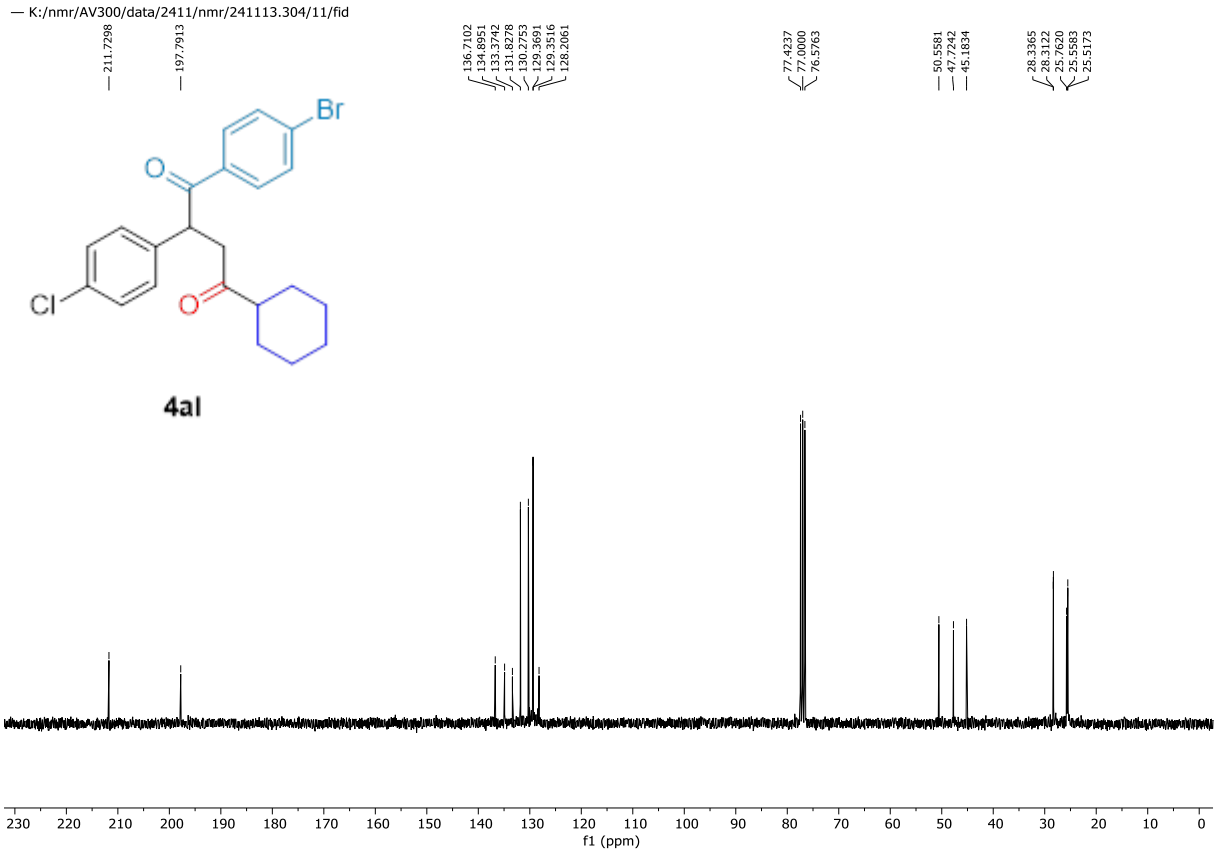
4ak ^{13}C NMR (75 MHz, $CDCl_3$)



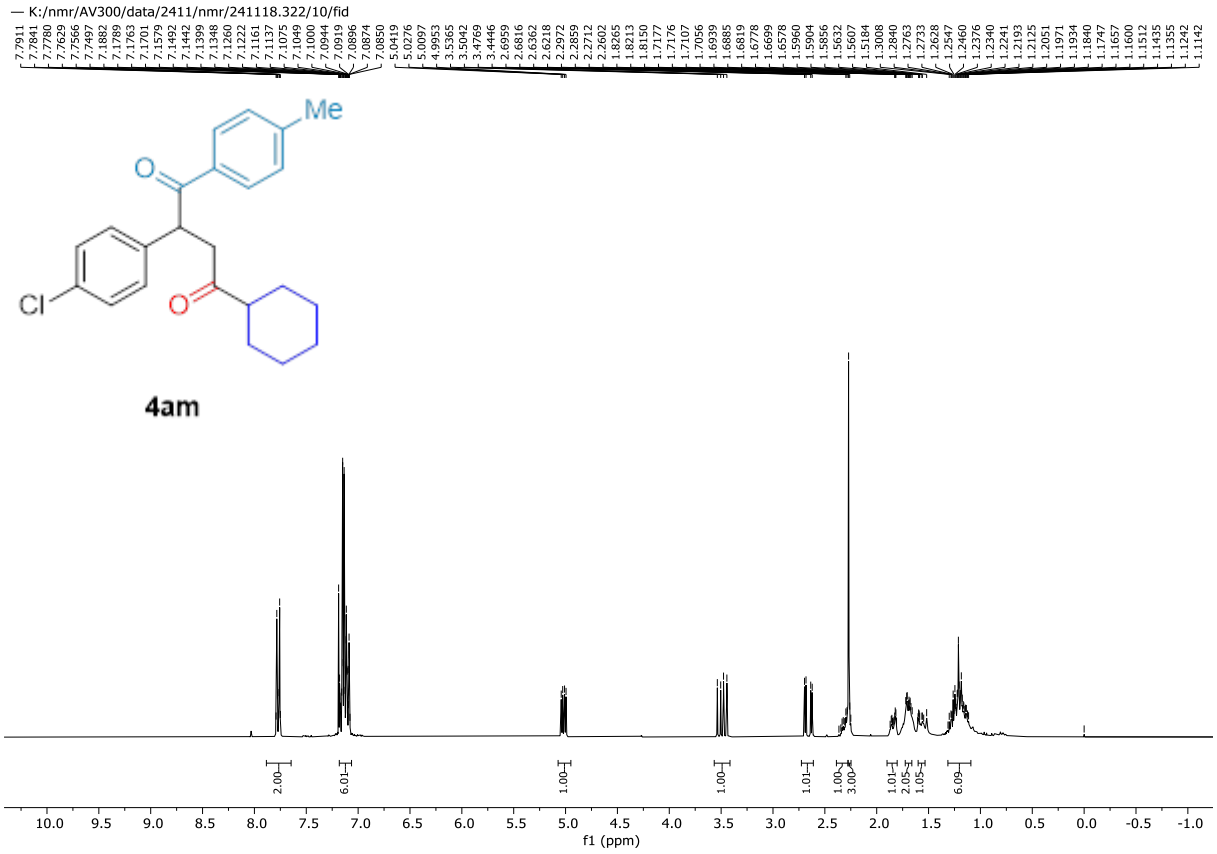
4al ^{1}H NMR (300 MHz, $CDCl_3$)



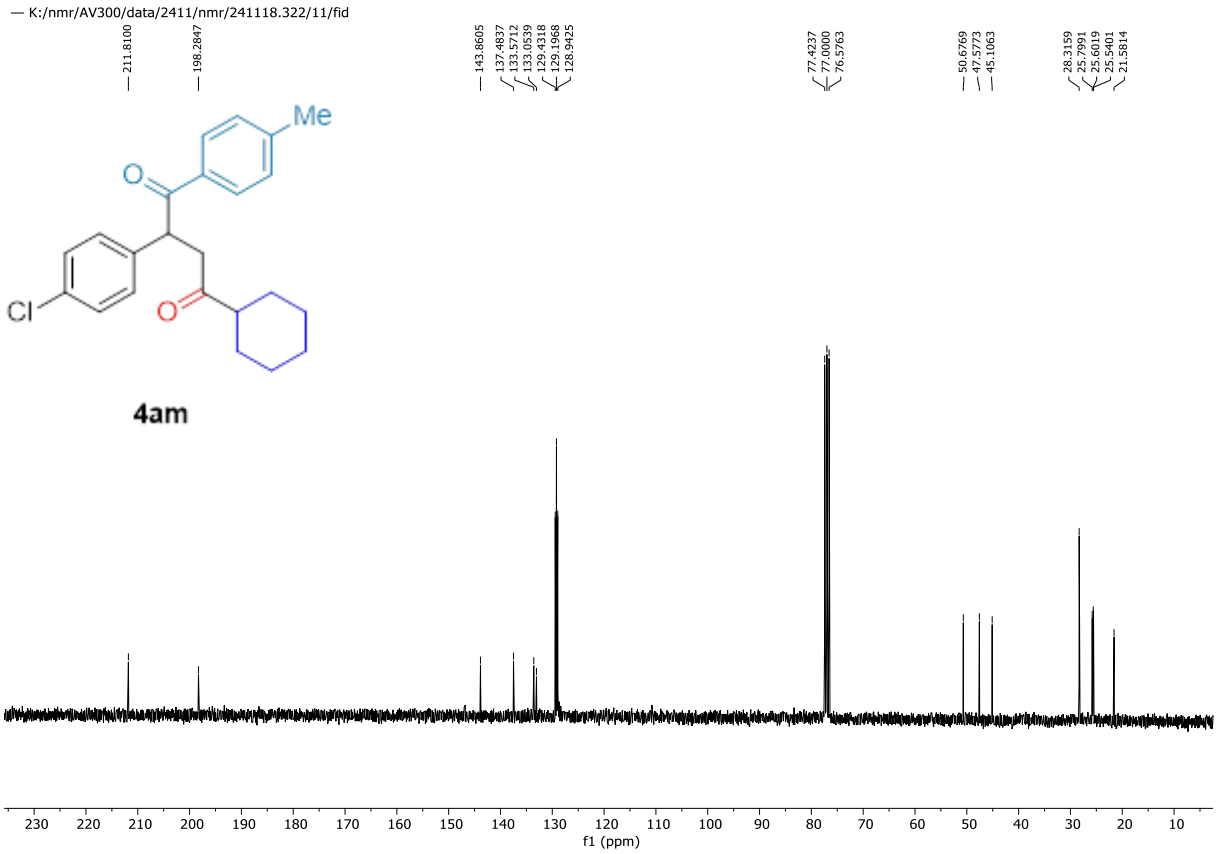
4al ^{13}C NMR (75 MHz, $CDCl_3$)



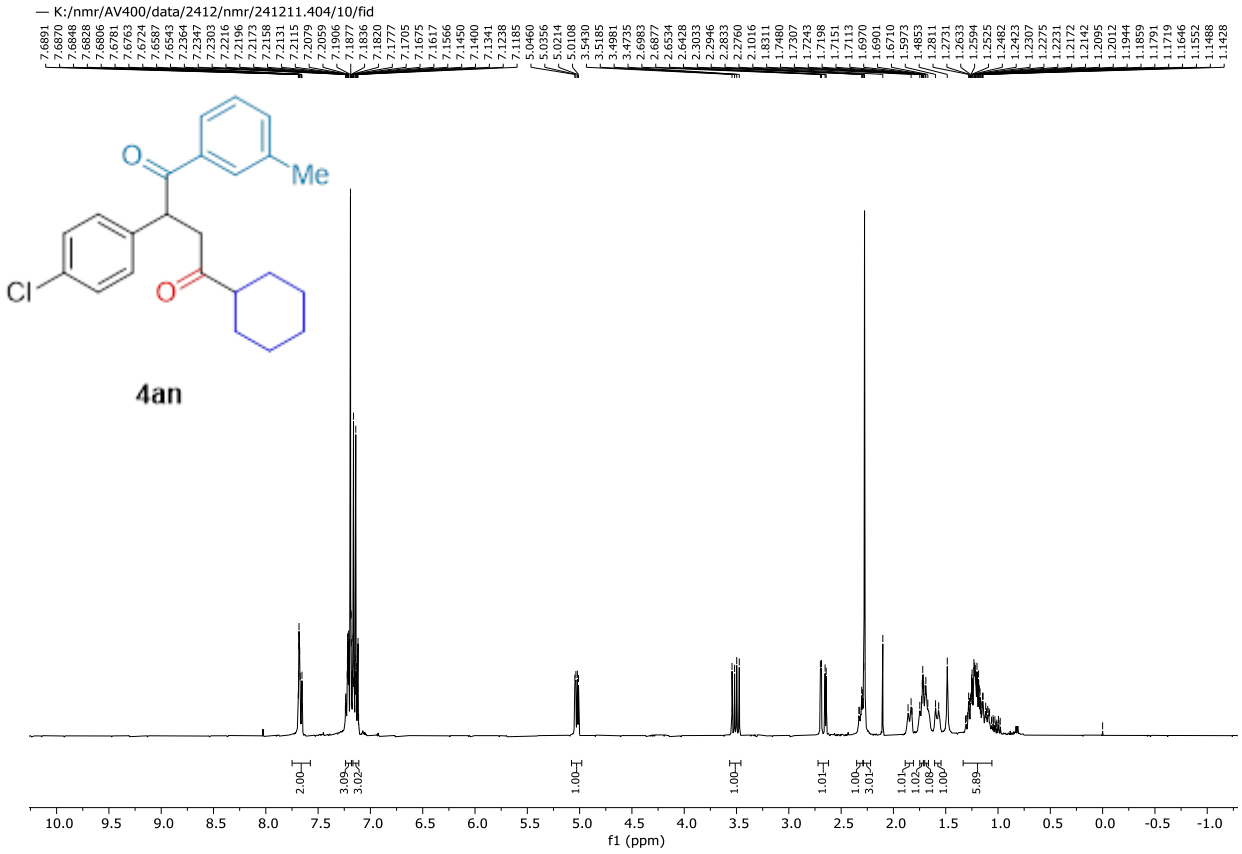
4am ^{1}H NMR (300 MHz, $CDCl_3$)



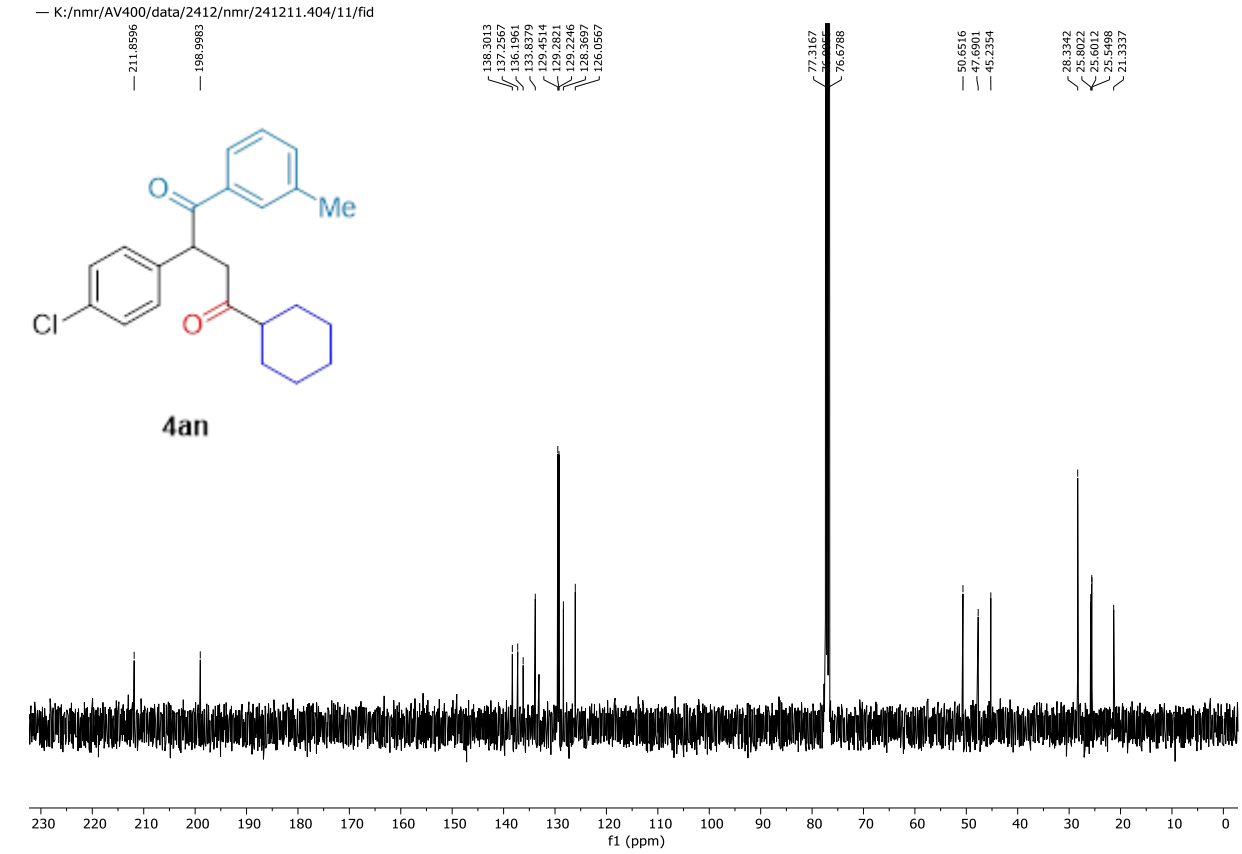
4am ^{13}C NMR (75 MHz, $CDCl_3$)



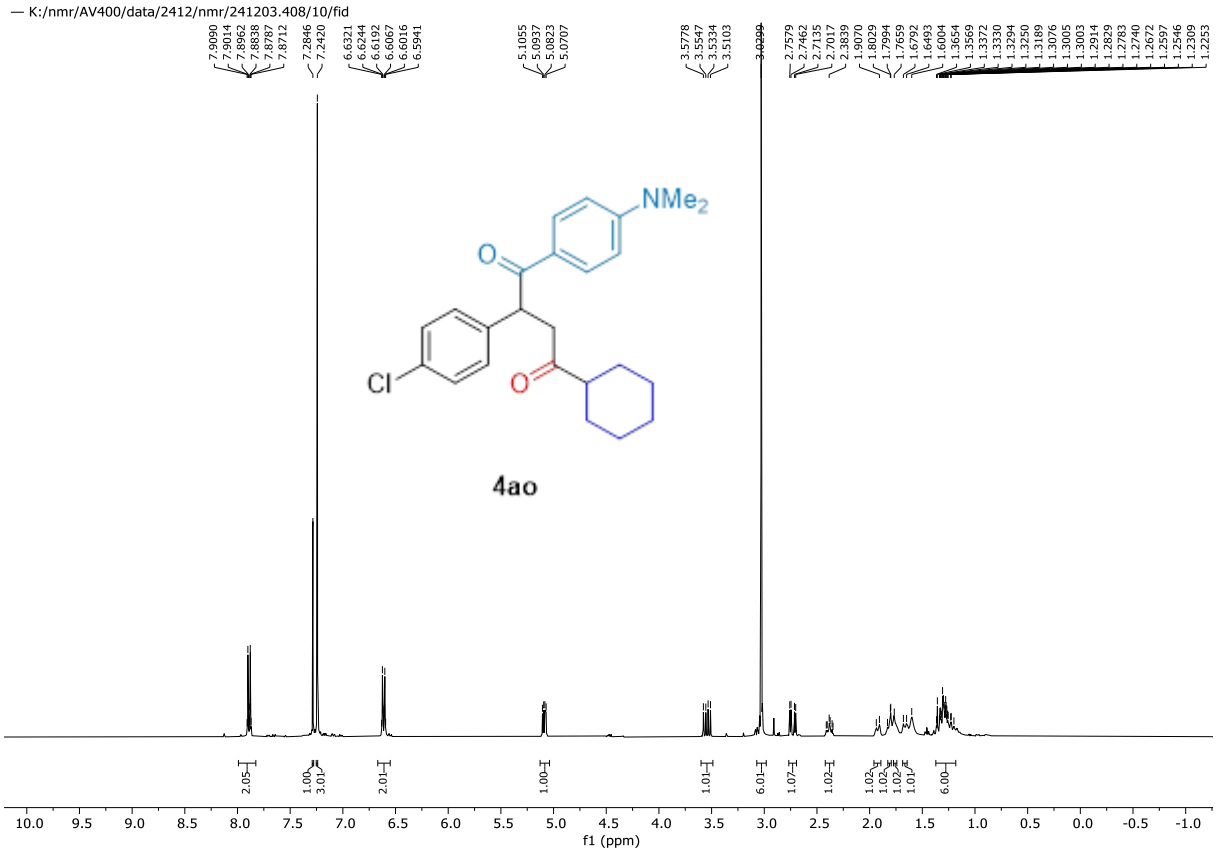
4an ^{1}H NMR (400 MHz, $CDCl_3$)



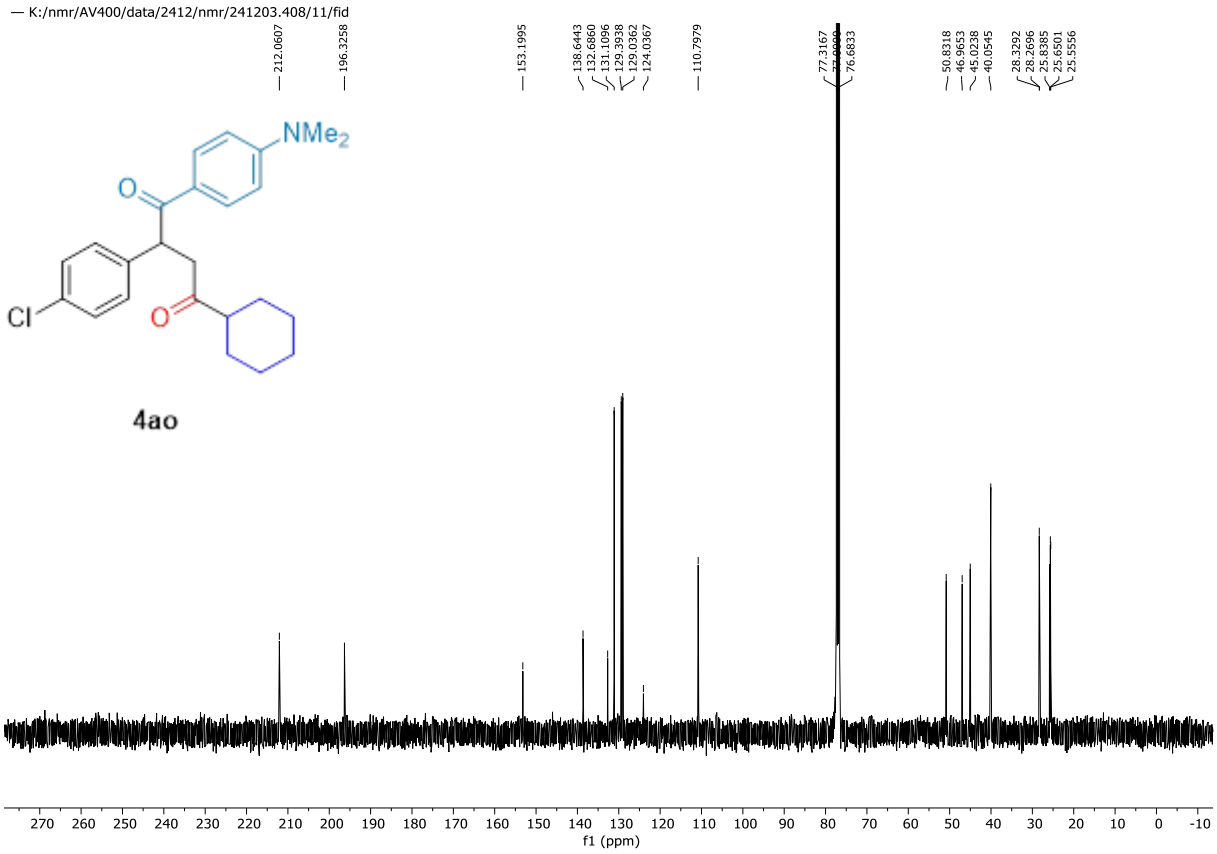
4an ^{13}C NMR (101 MHz, $CDCl_3$)



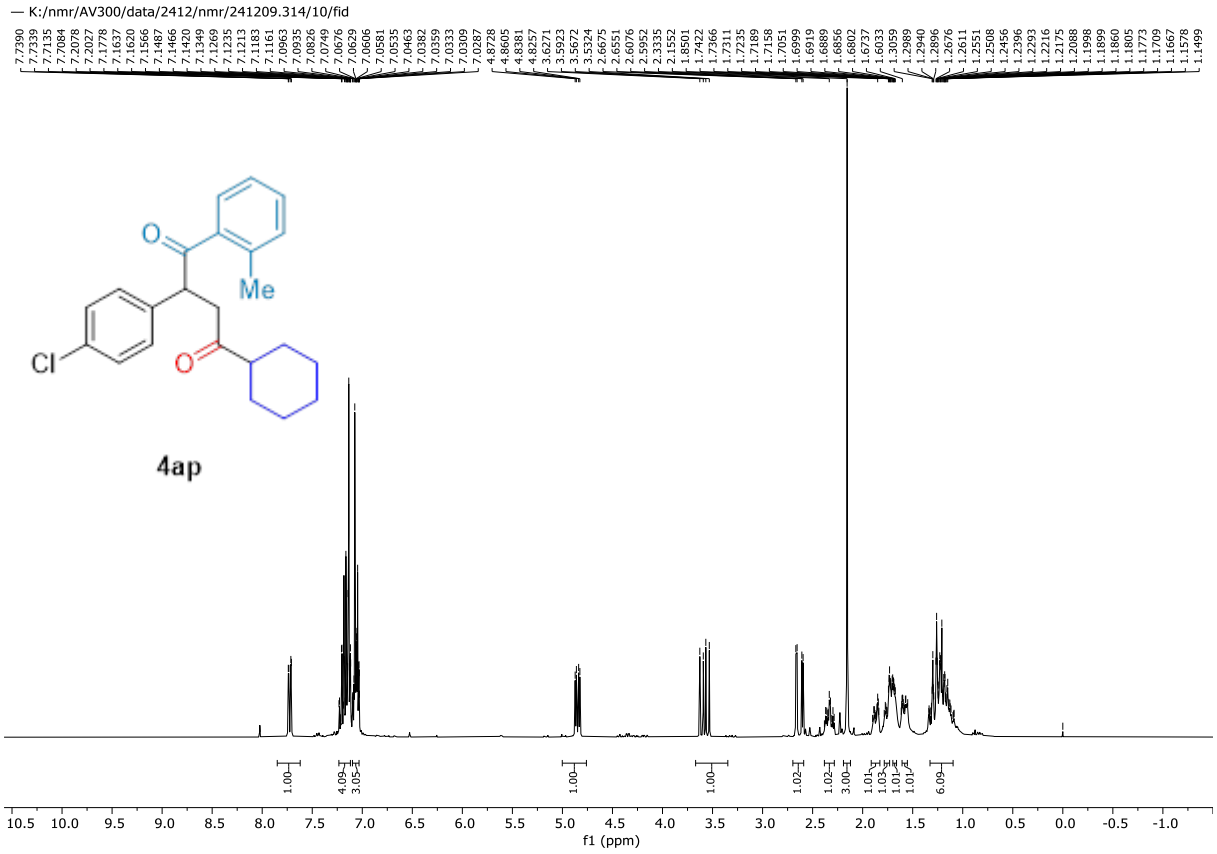
4ao ^{1}H NMR (400 MHz, $CDCl_3$)



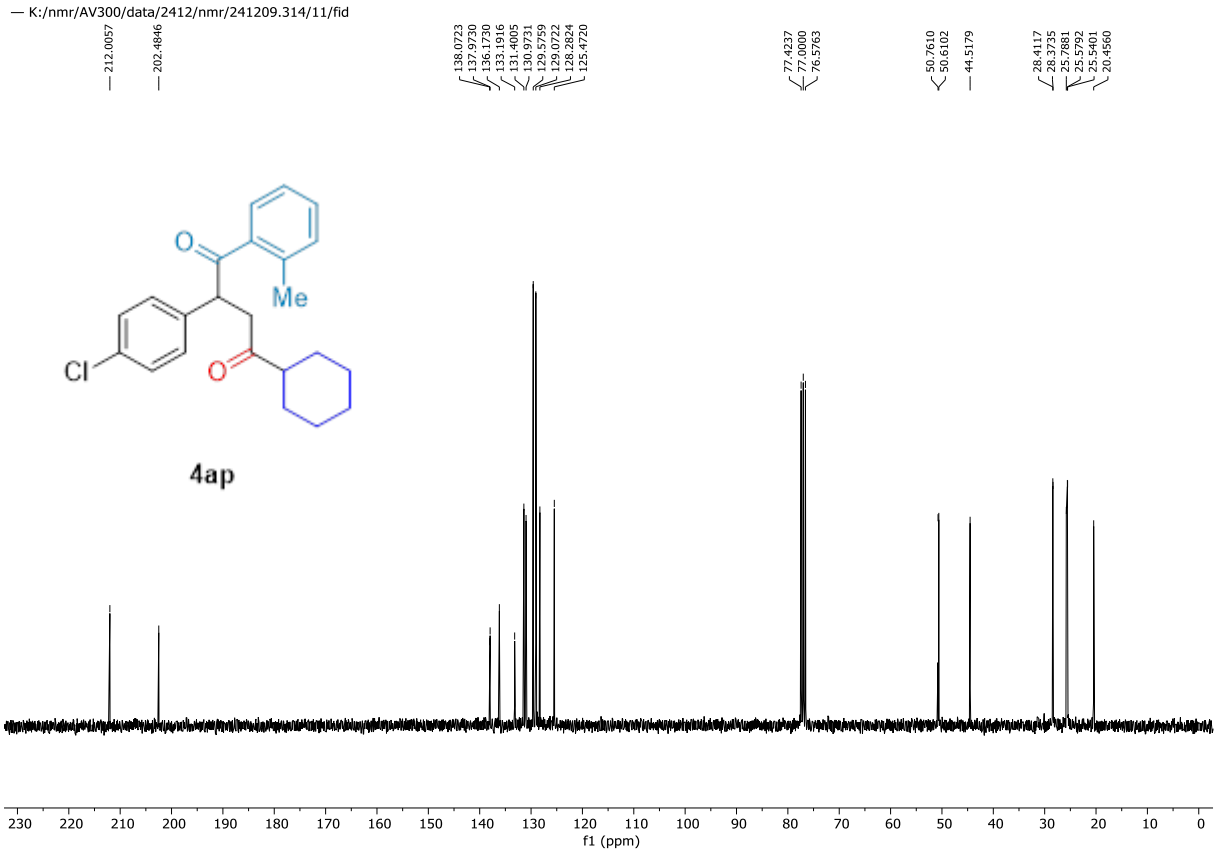
4ao ^{13}C NMR (101 MHz, $CDCl_3$)



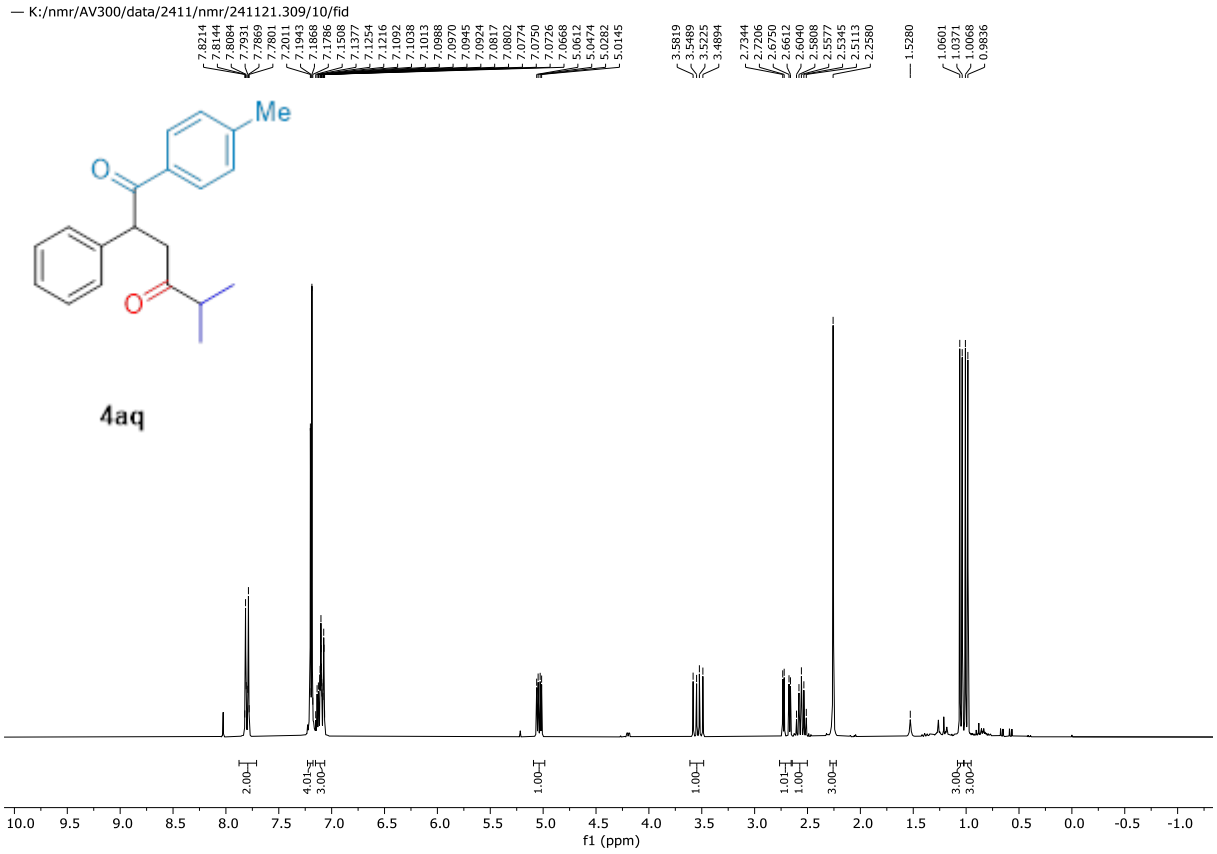
4ap ^{1}H NMR (300 MHz, $CDCl_3$)



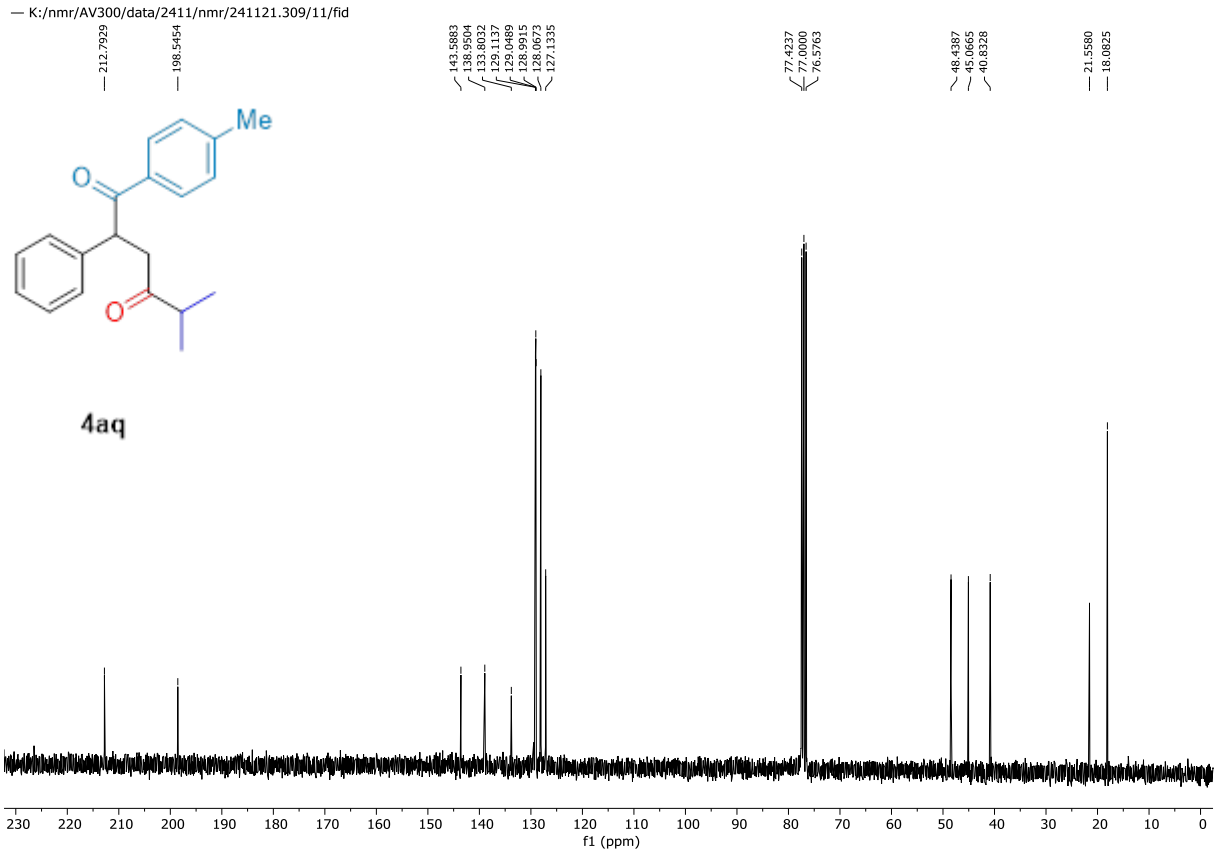
4ap ^{13}C NMR (75 MHz, $CDCl_3$)



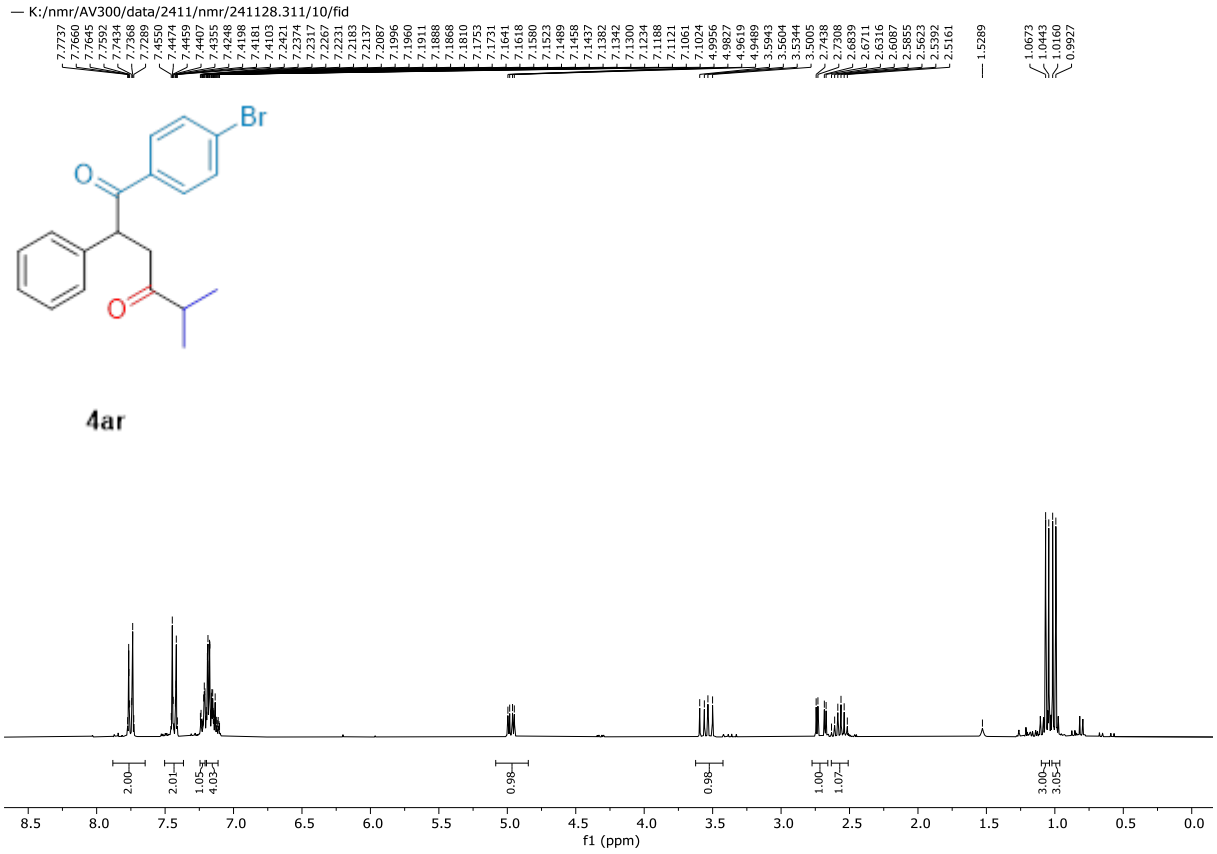
4aq ^{1}H NMR (300 MHz, $CDCl_3$)



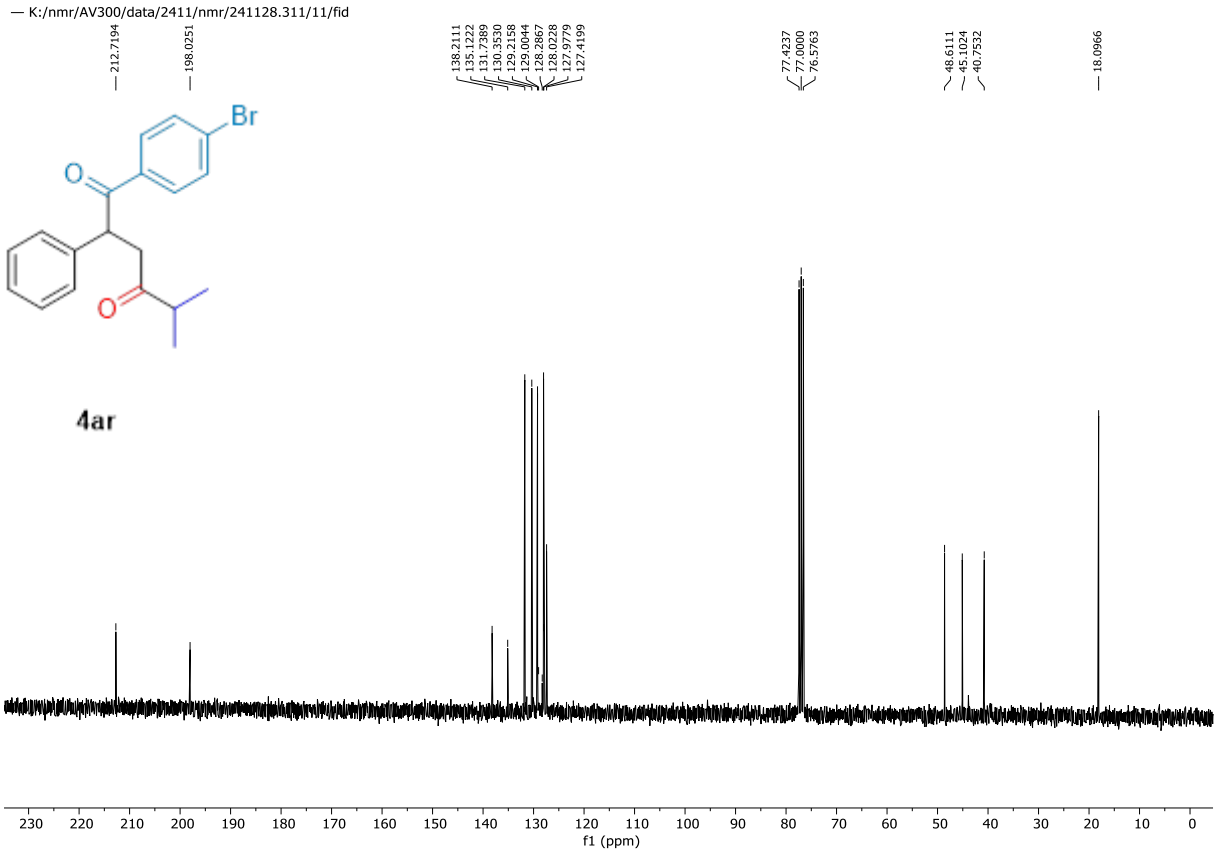
4aq ^{13}C NMR (75 MHz, $CDCl_3$)



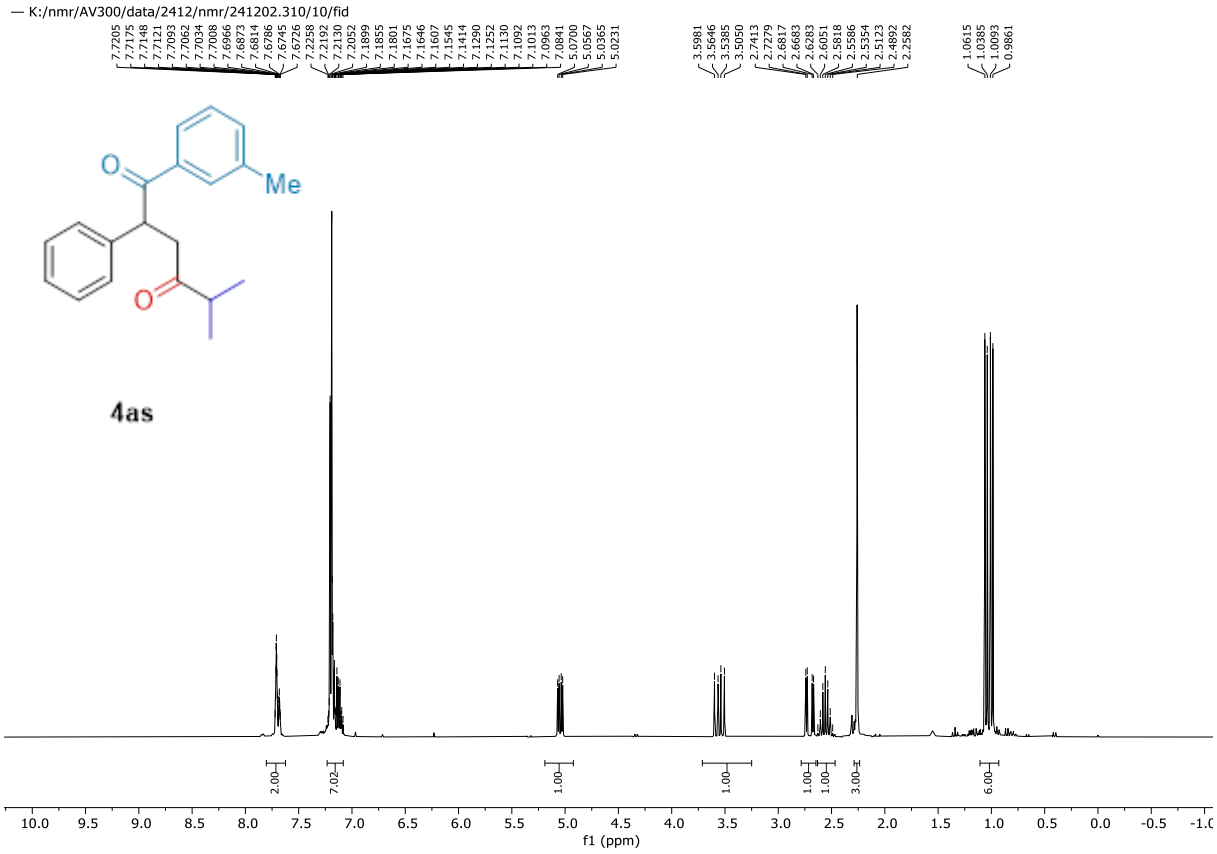
4ar ^{1}H NMR (300 MHz, $CDCl_3$)



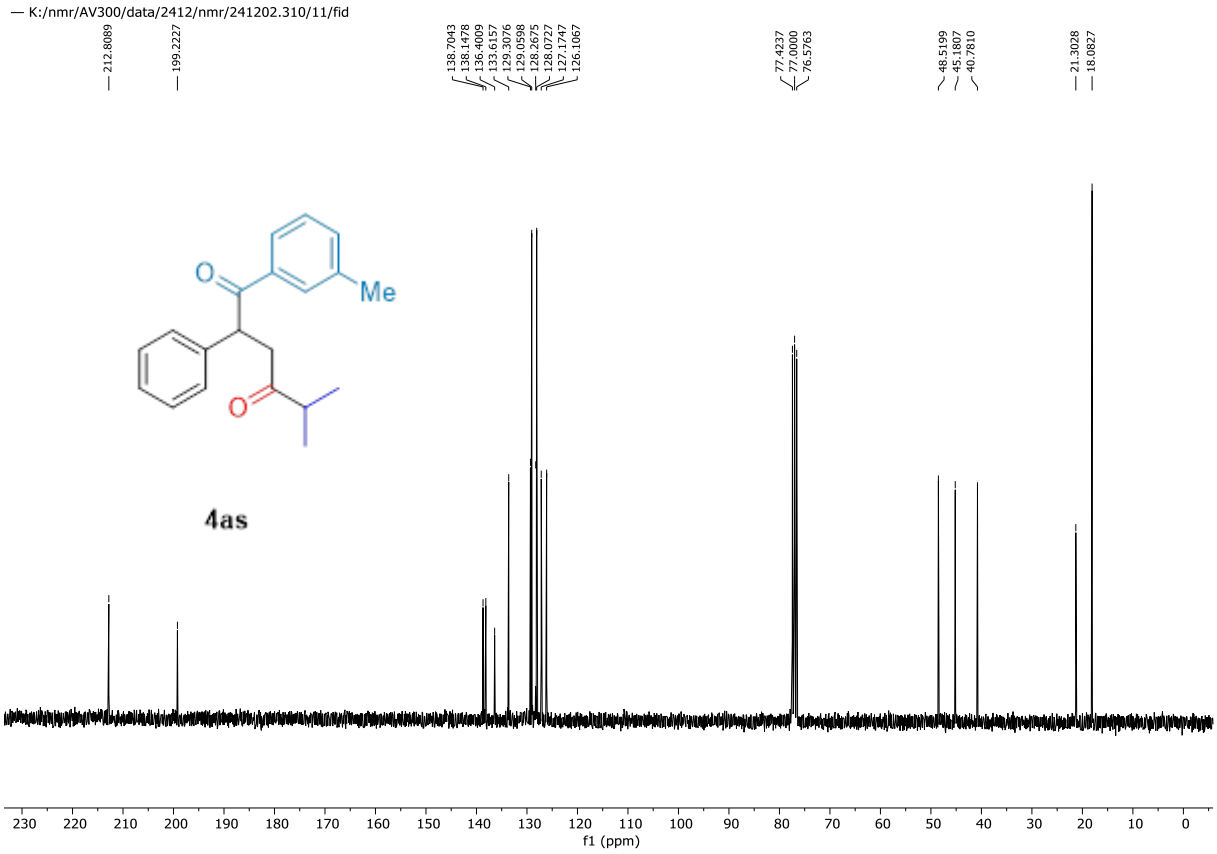
4ar ^{13}C NMR (75 MHz, $CDCl_3$)



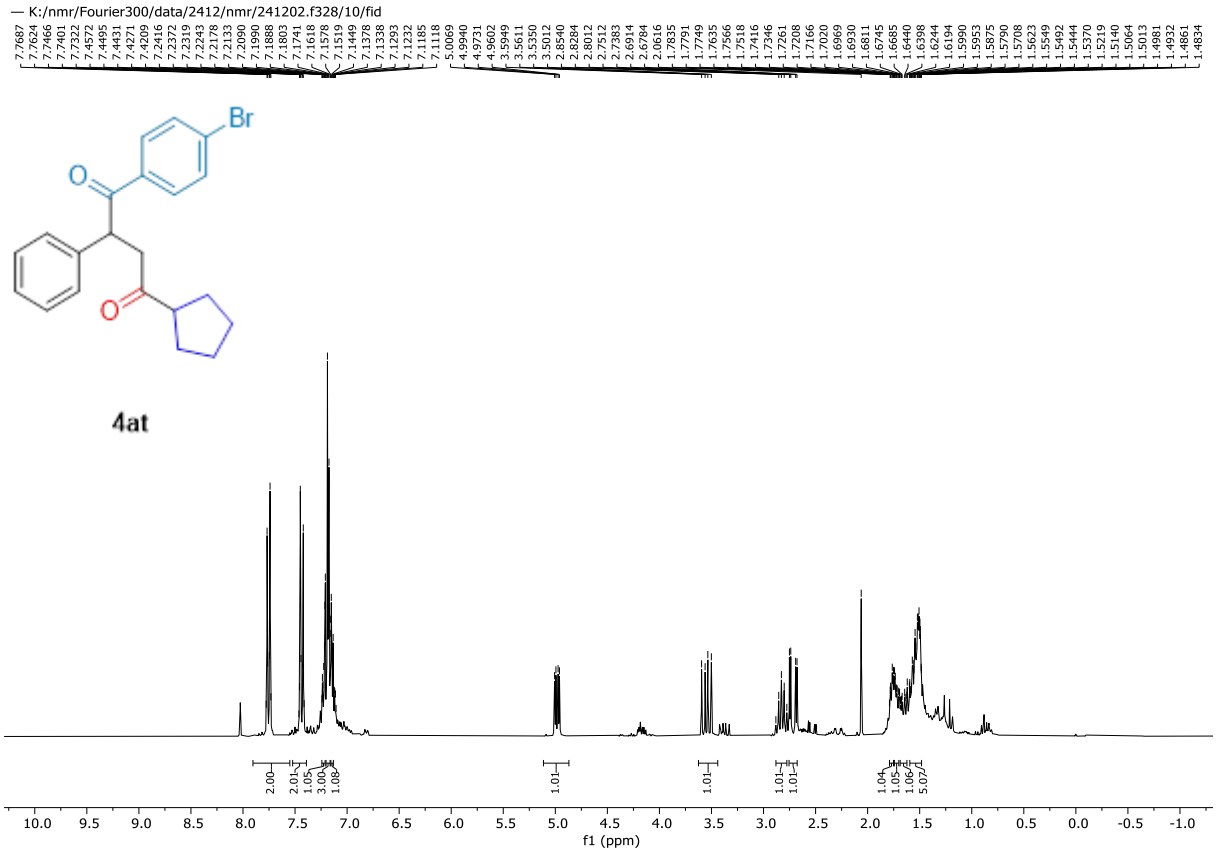
4as ^{1}H NMR (300 MHz, $CDCl_3$)



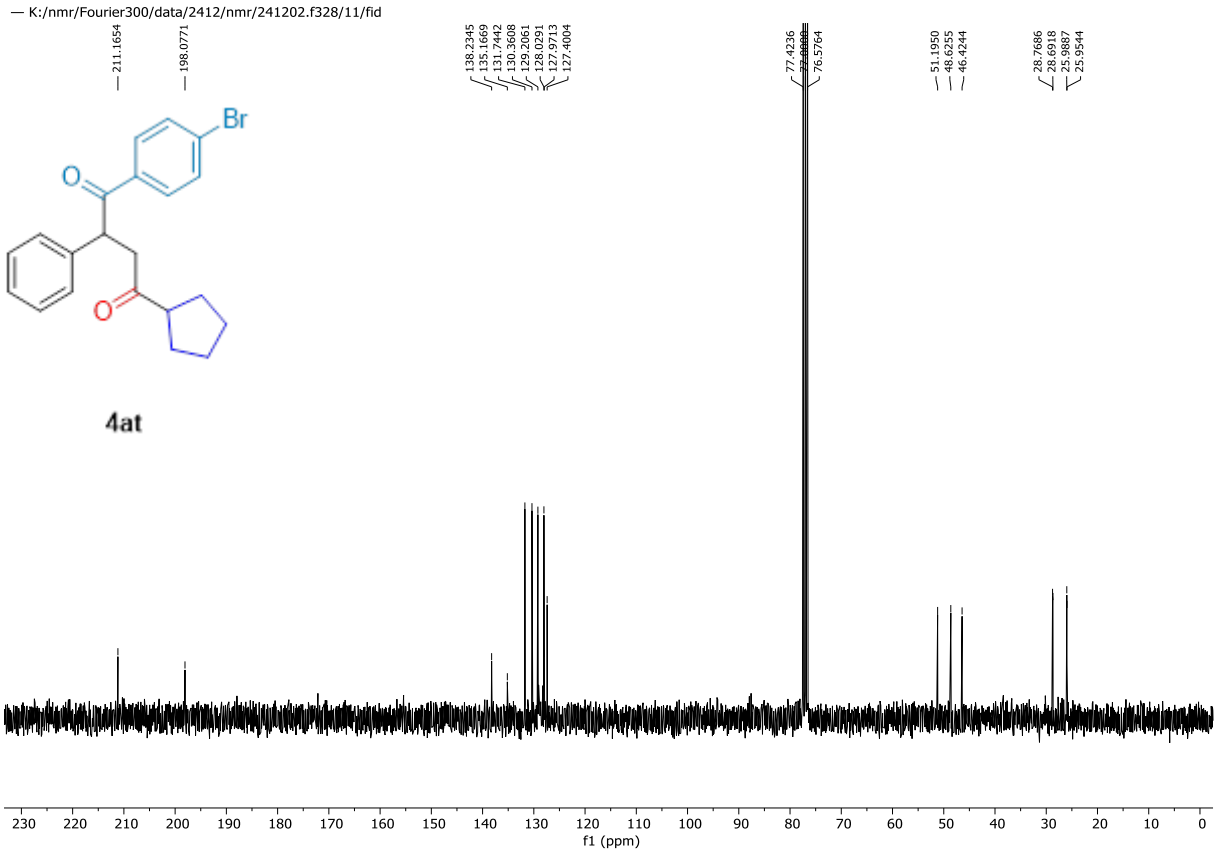
4as ^{13}C NMR (75 MHz, $CDCl_3$)



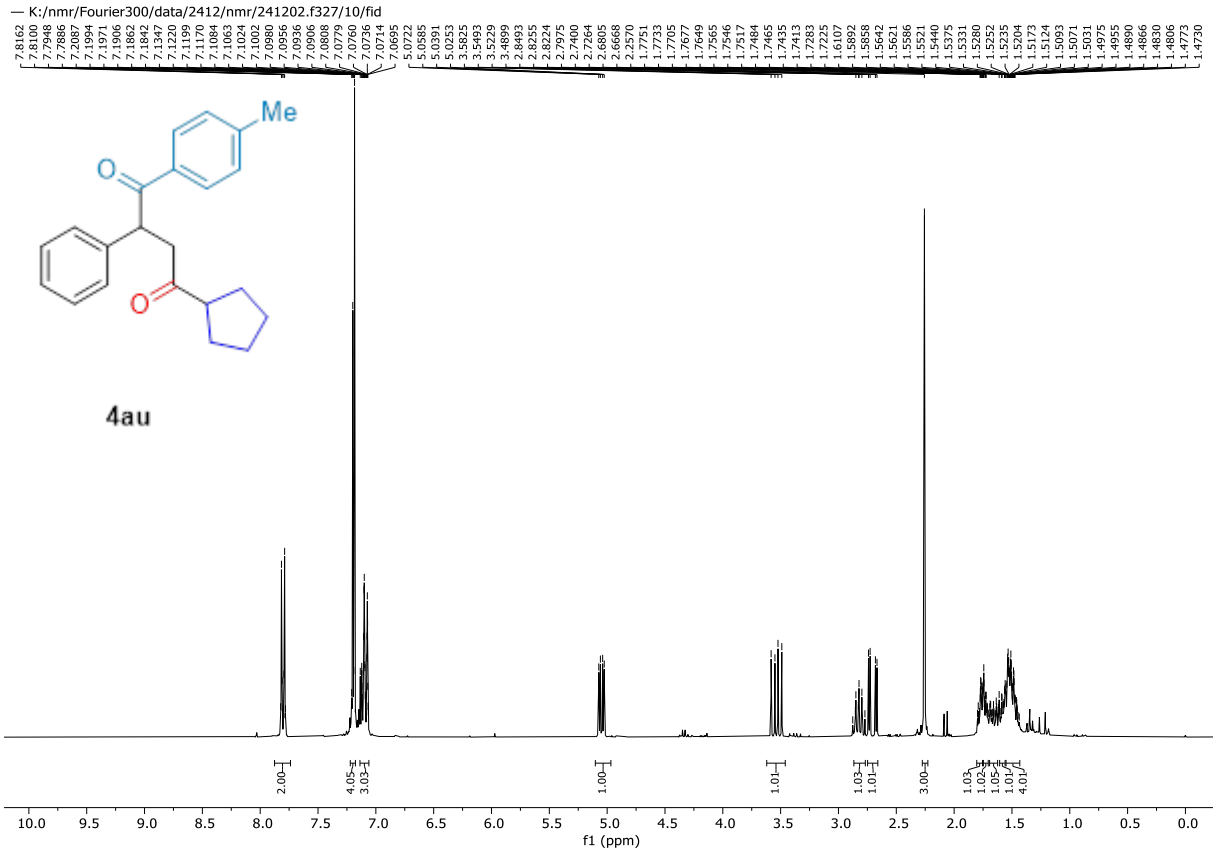
4at ^{1}H NMR (300 MHz, $CDCl_3$)



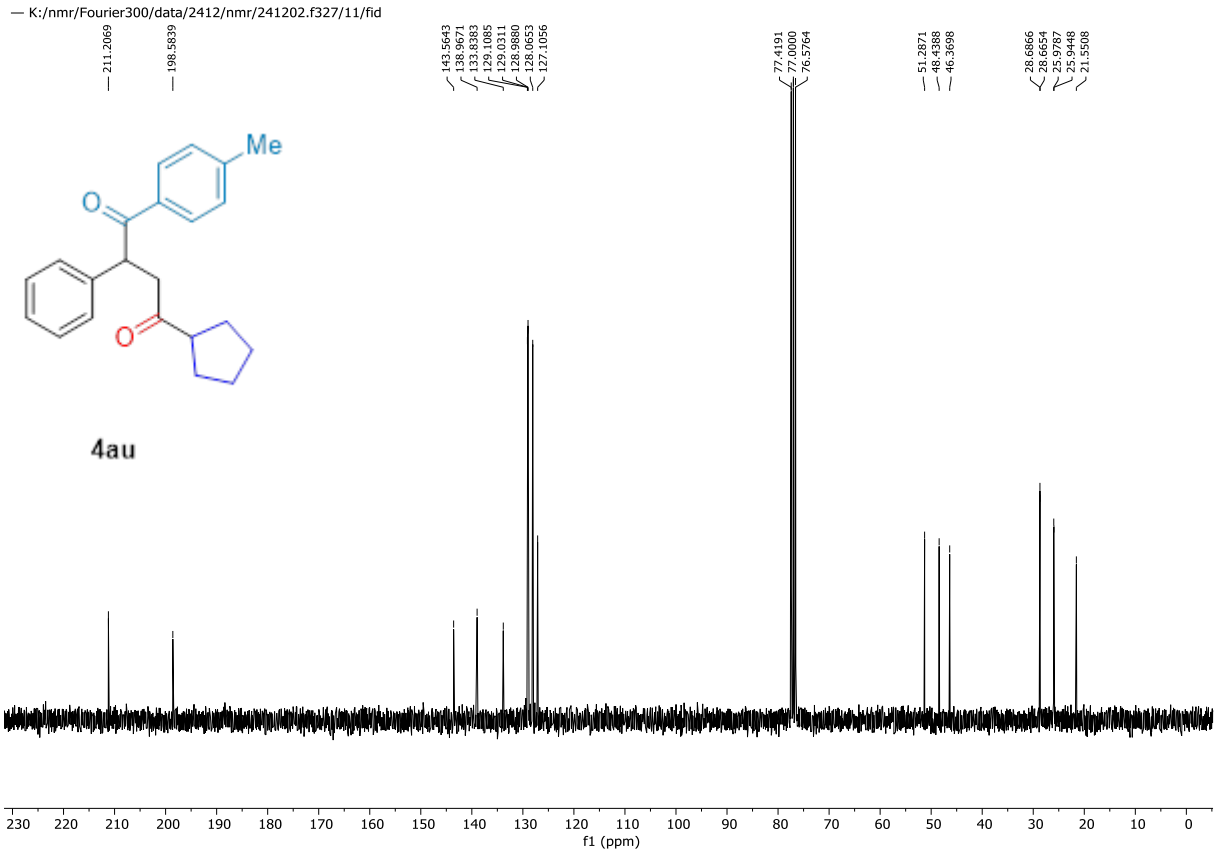
4at ^{13}C NMR (75 MHz, $CDCl_3$)



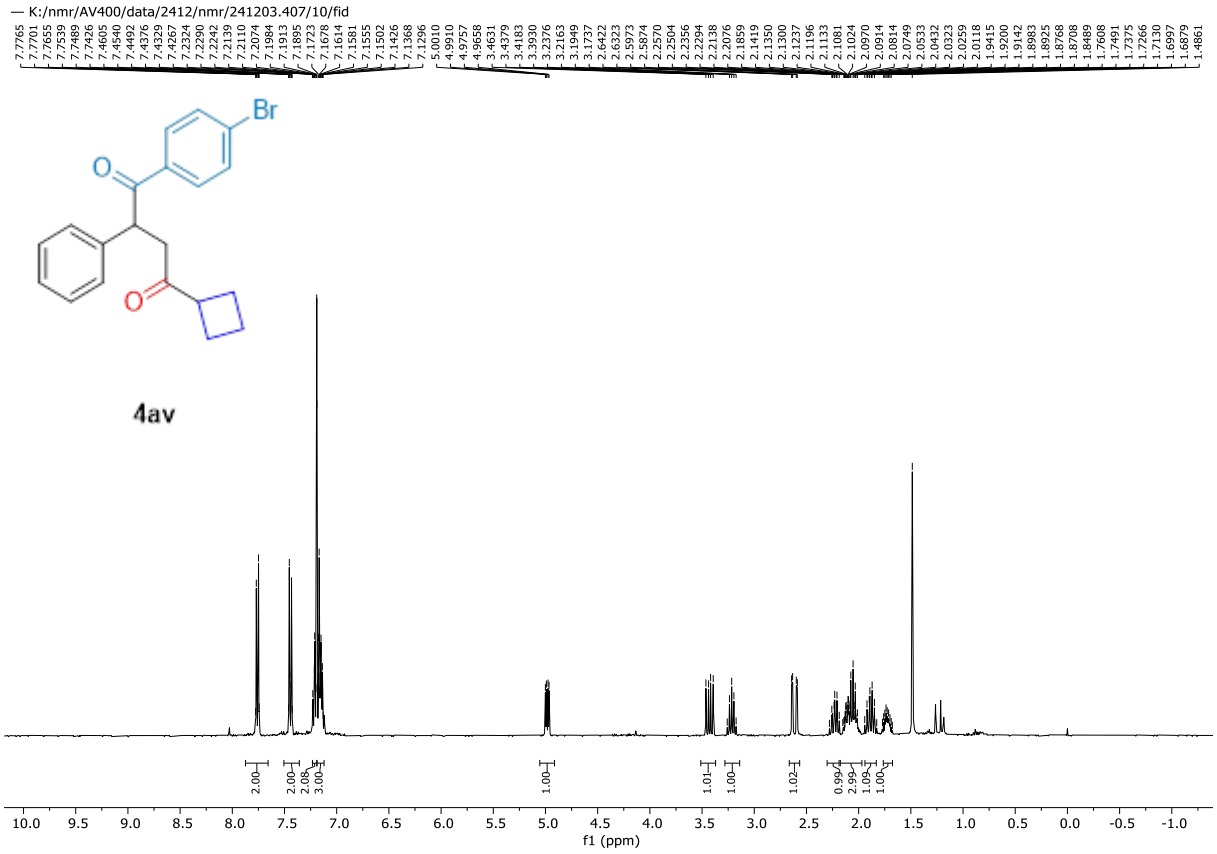
4au ^{1}H NMR (300 MHz, $CDCl_3$)



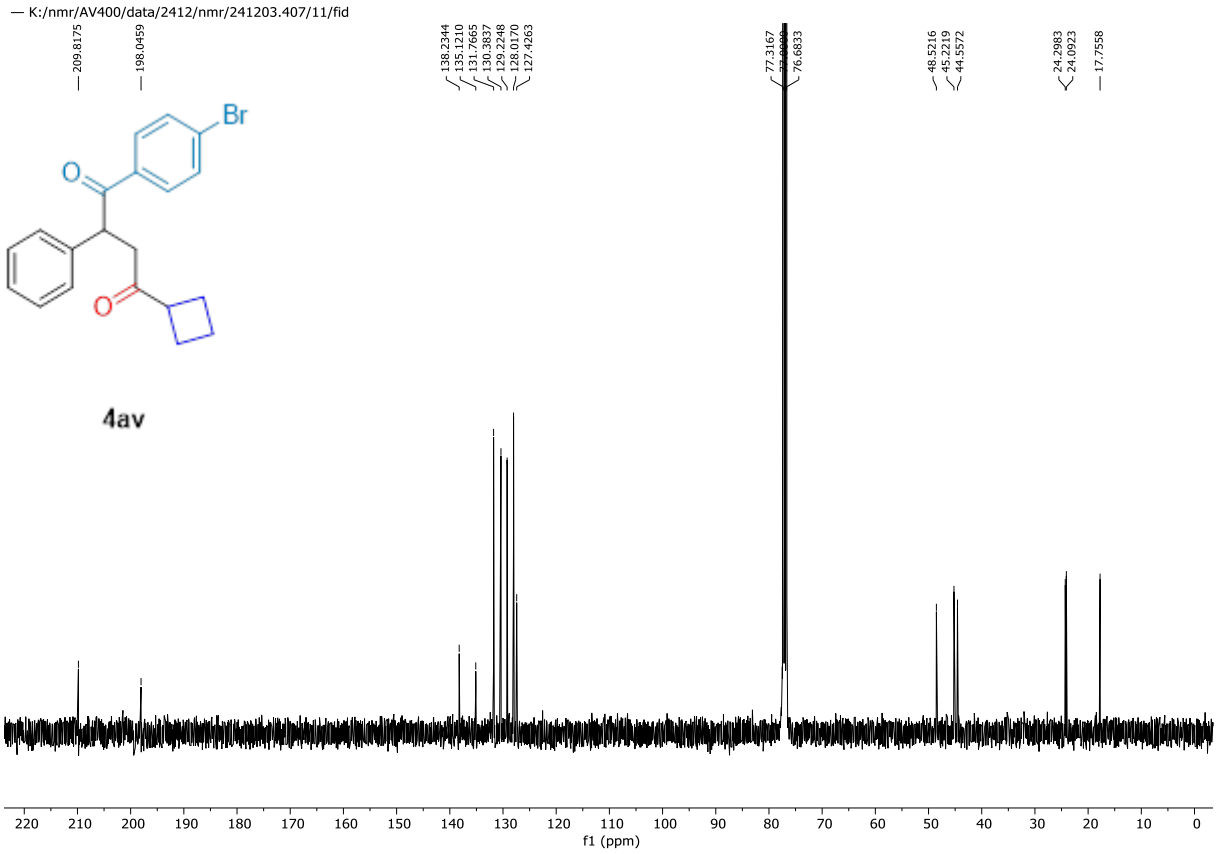
4au ^{13}C NMR 75 MHz, $CDCl_3$)



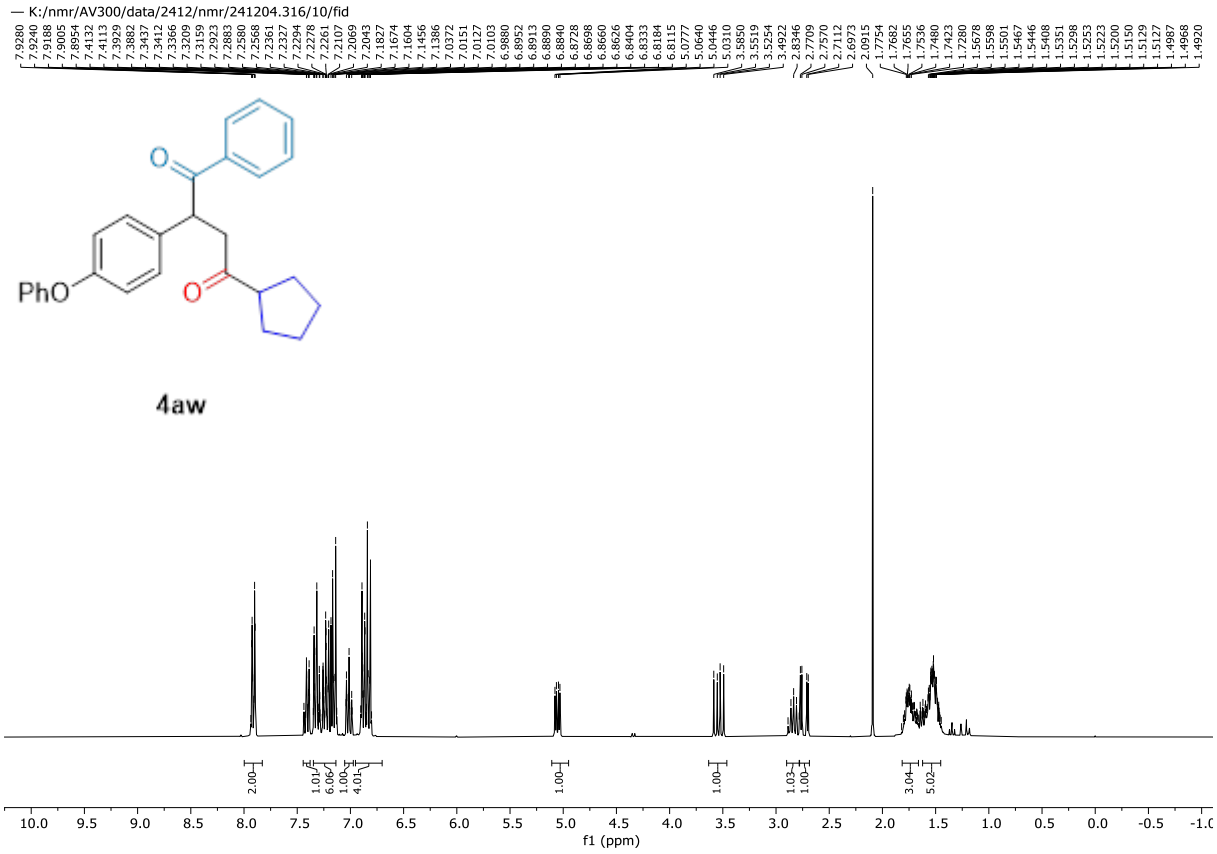
4av ^{1}H NMR (400 MHz, $CDCl_3$)



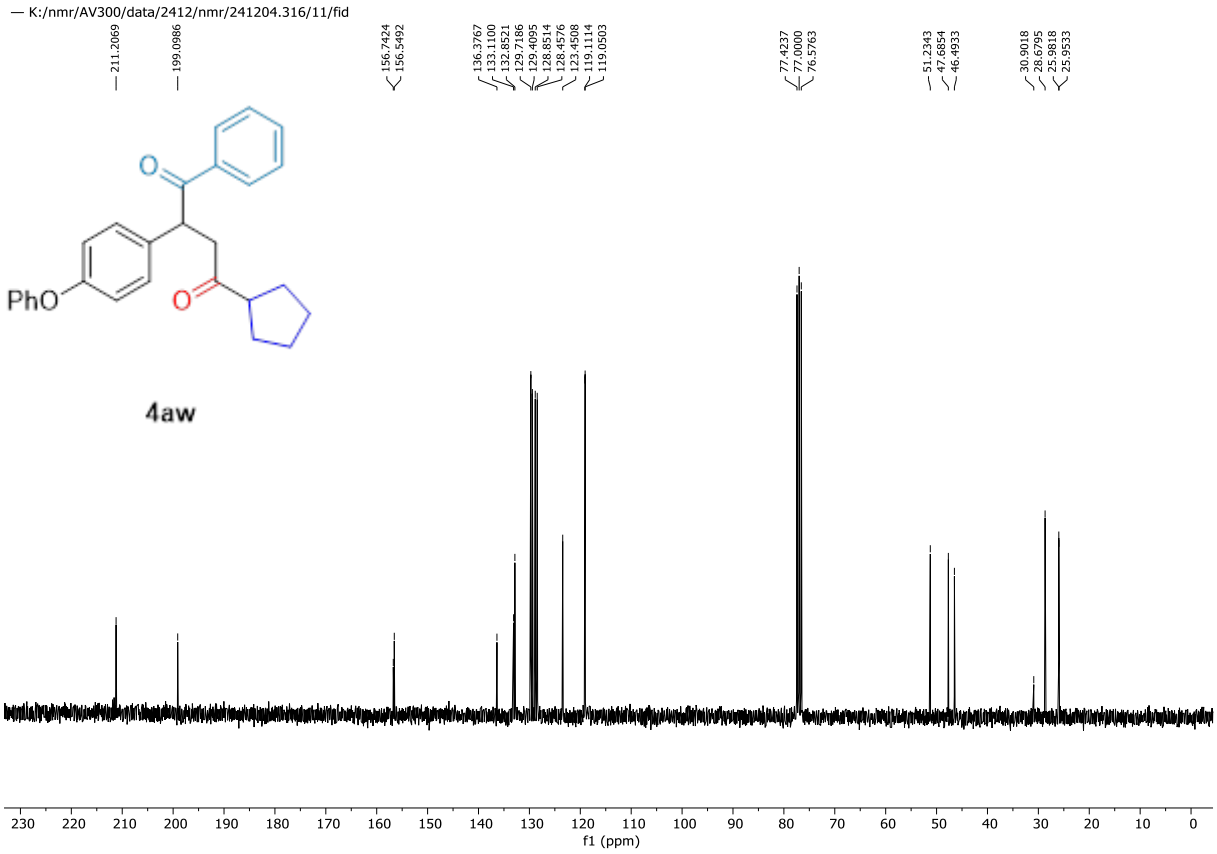
4av ^{13}C NMR (101 MHz, $CDCl_3$)



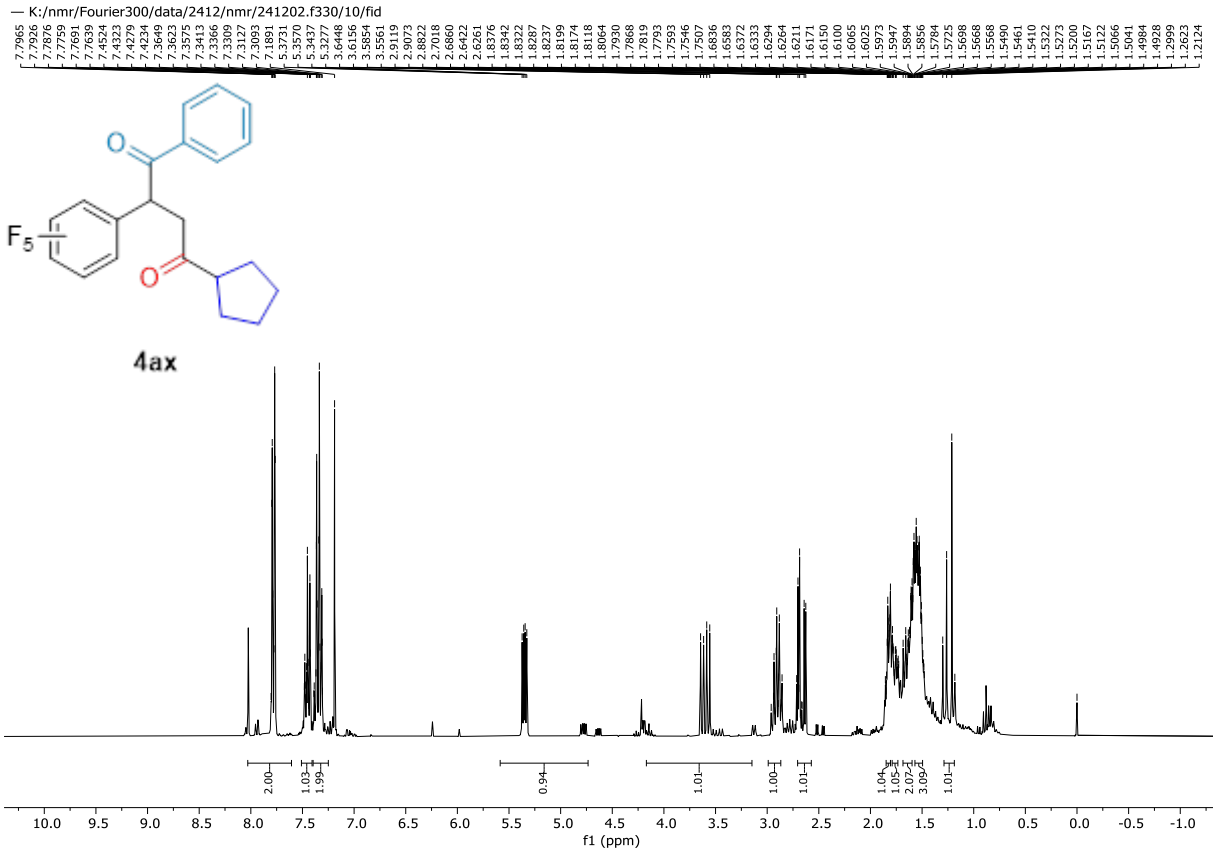
4aw ^{1}H NMR (300 MHz, $CDCl_3$)



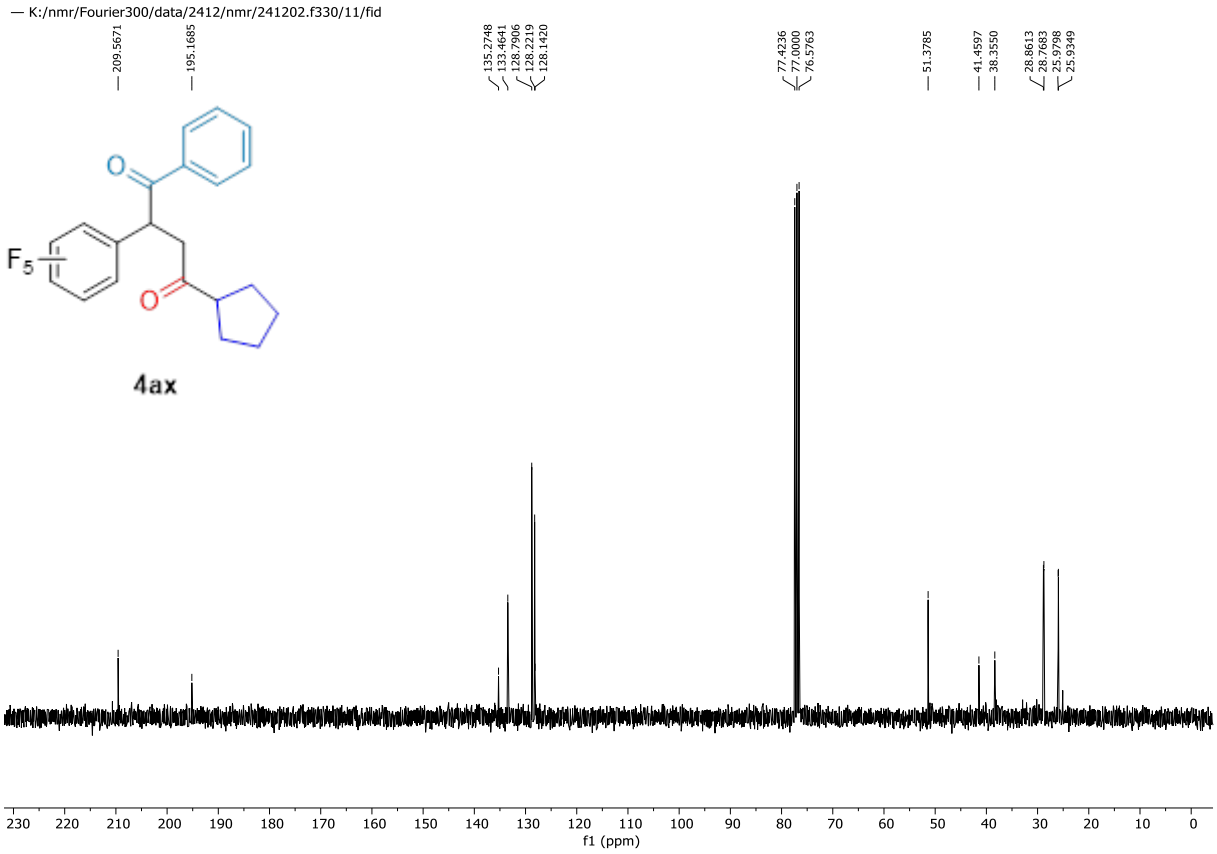
4aw ^{13}C NMR (75 MHz, $CDCl_3$)



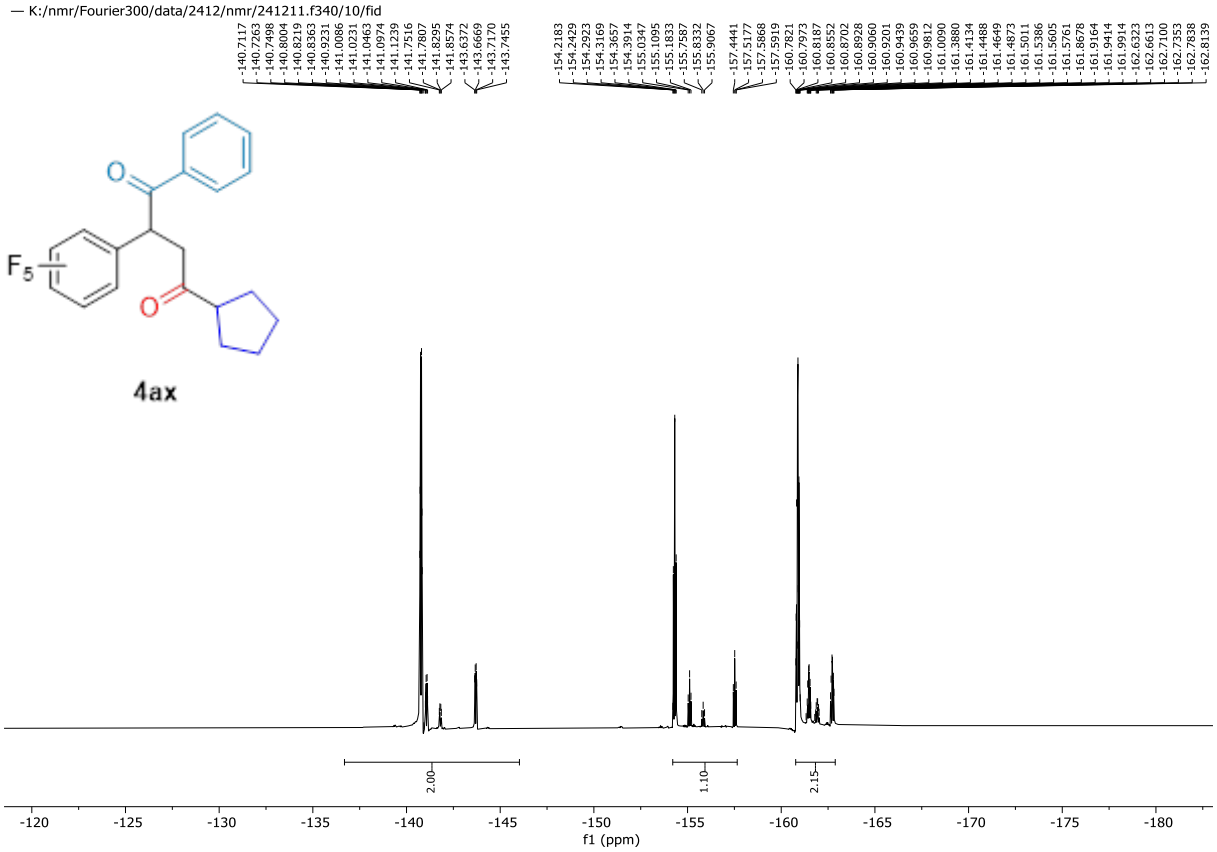
4ax ^{1}H NMR (300 MHz, $CDCl_3$)



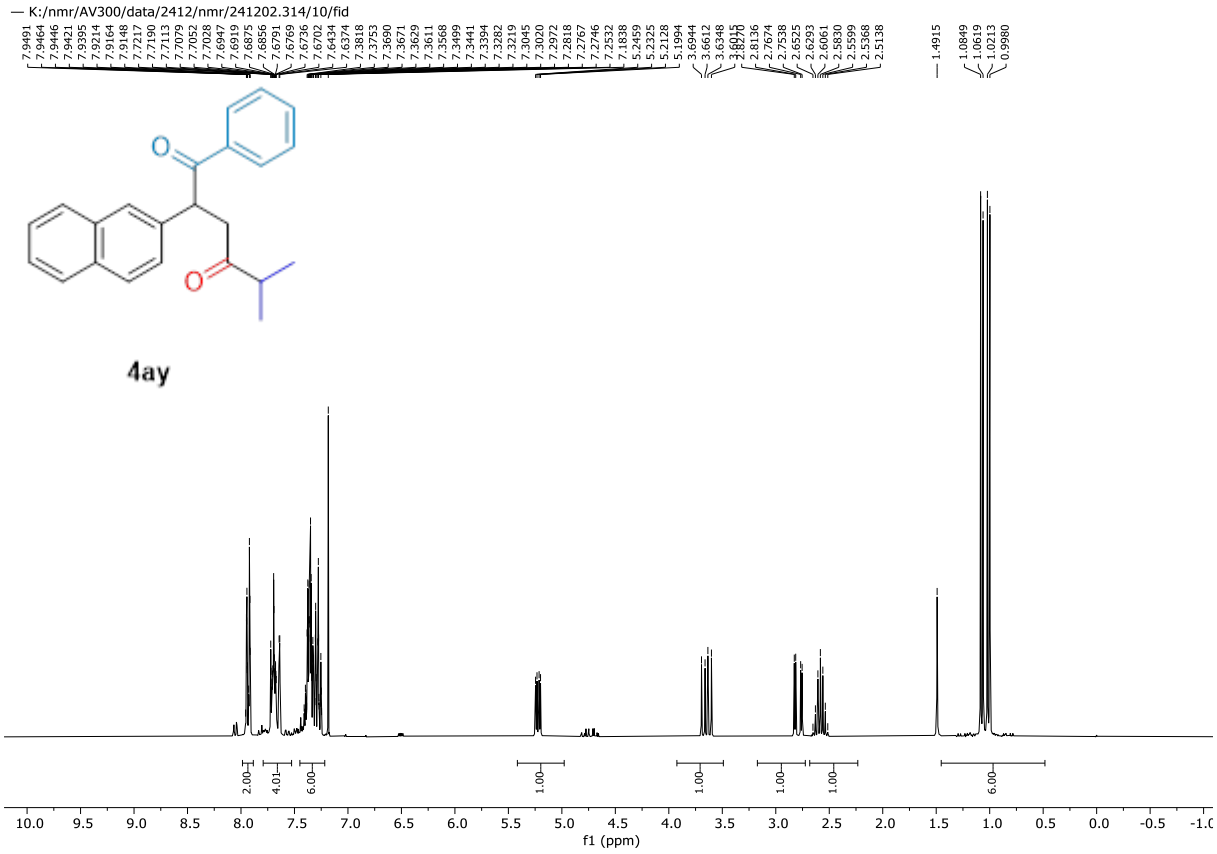
4ax ^{13}C NMR (75 MHz, $CDCl_3$)



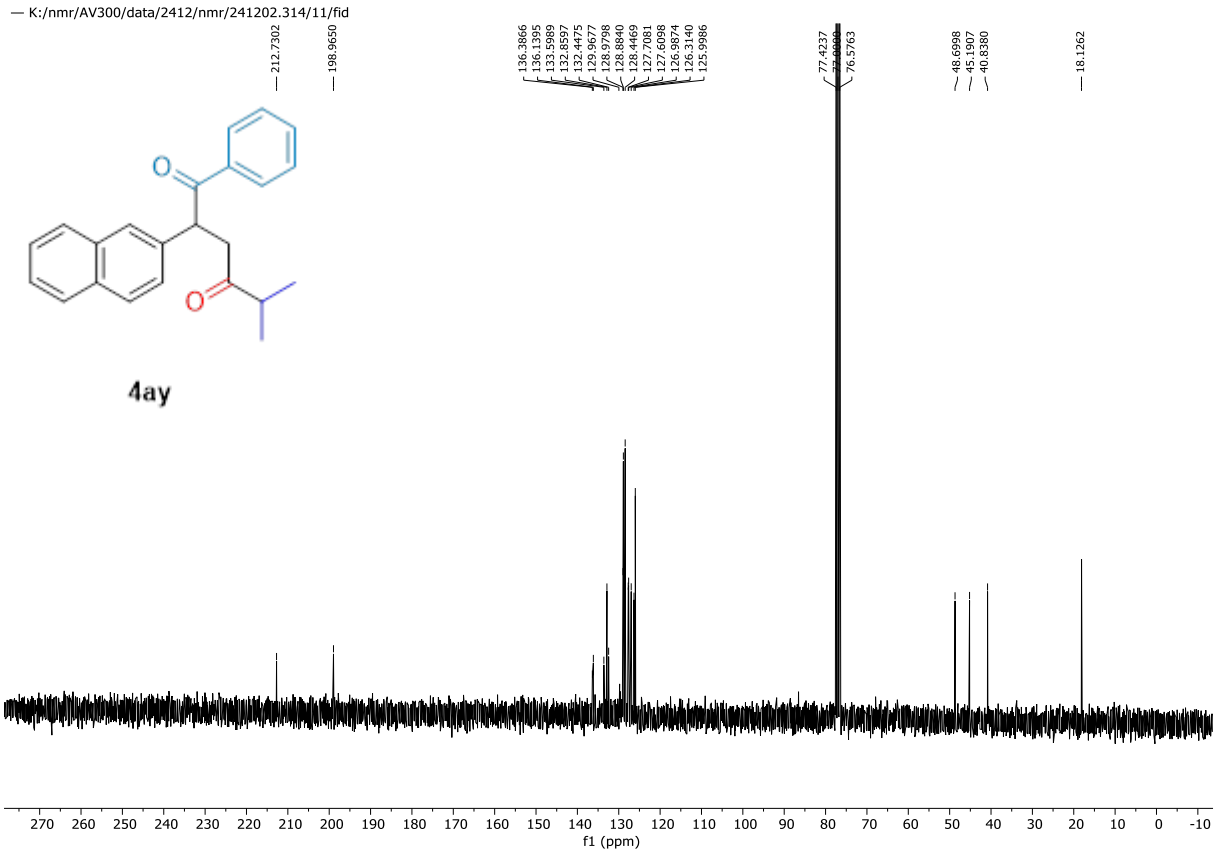
4z ^{13}F NMR (382 MHz, $CDCl_3$)



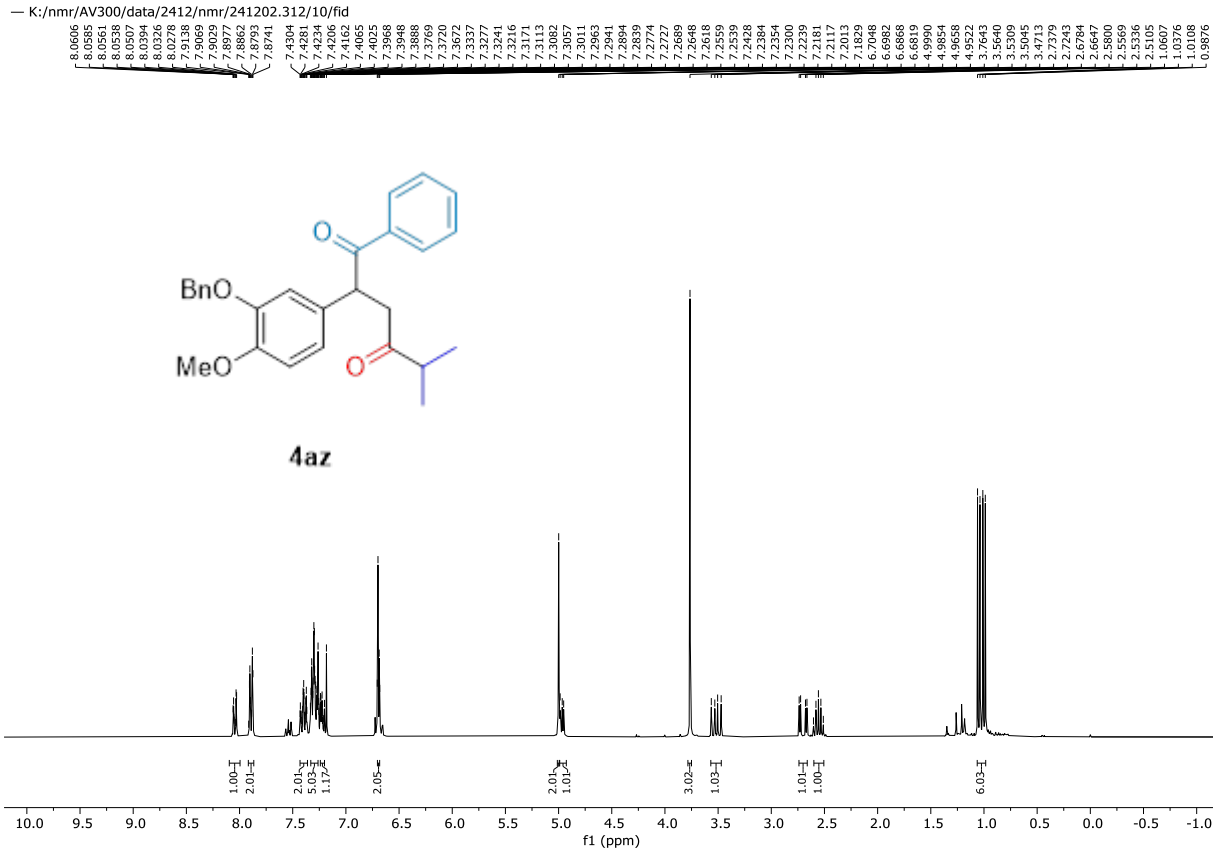
4ay ^{1}H NMR (300 MHz, $CDCl_3$)



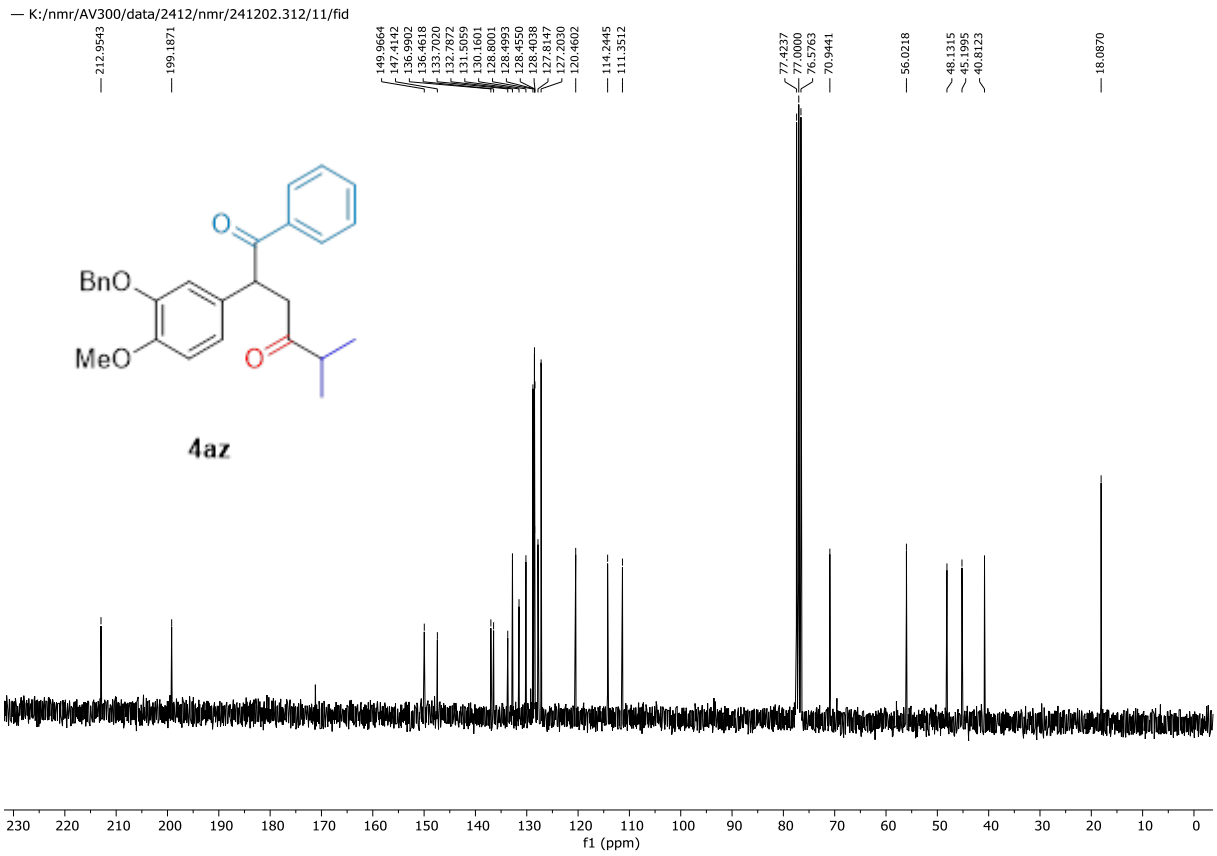
4ay ^{13}C NMR (75 MHz, $CDCl_3$)



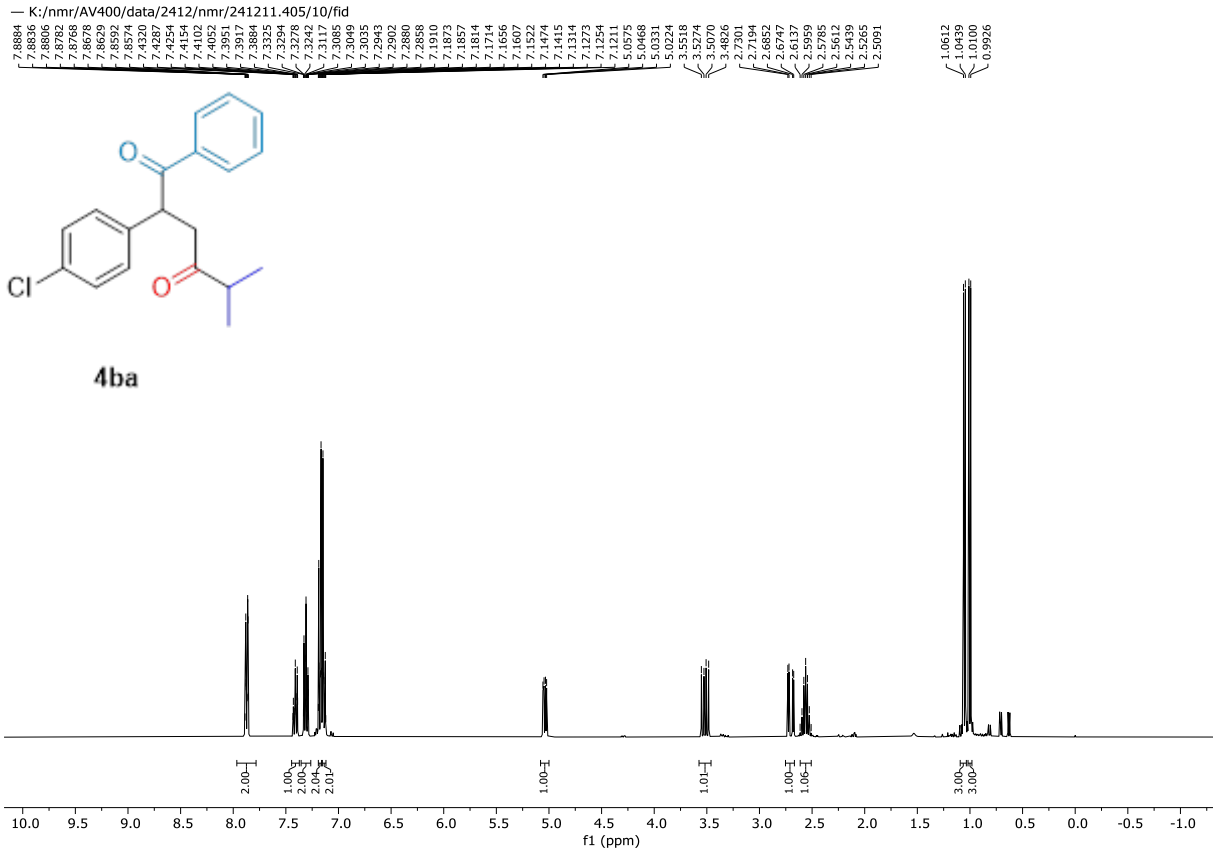
4az ^{1}H NMR (300 MHz, $CDCl_3$)



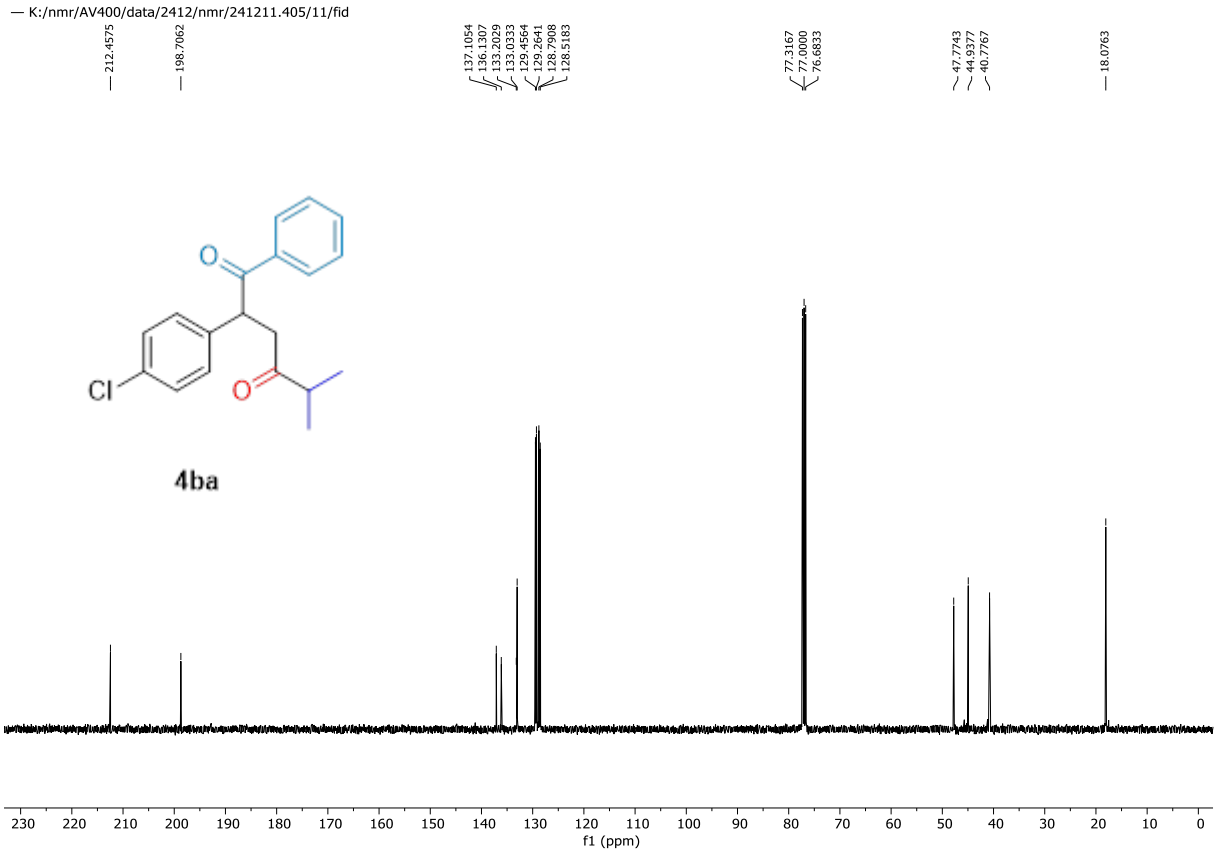
4az ^{13}C NMR (75 MHz, $CDCl_3$)



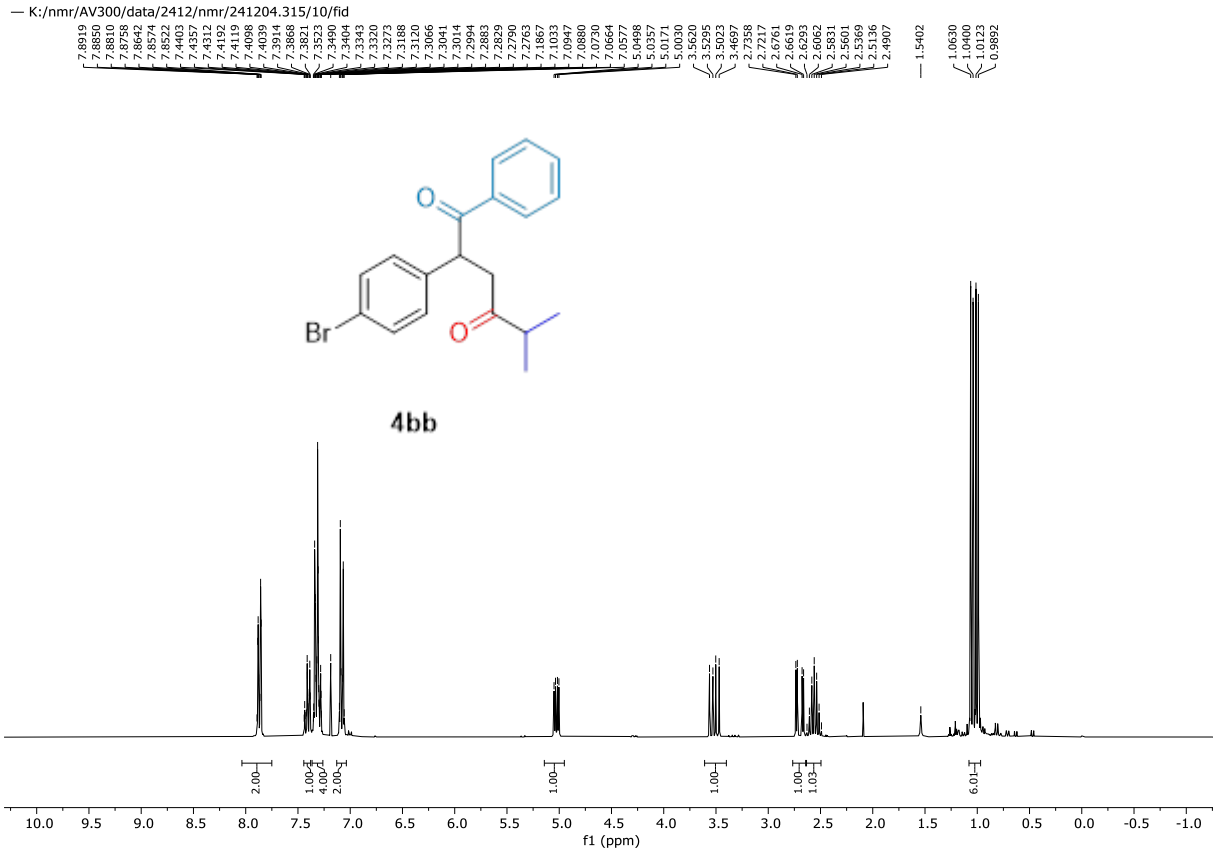
4ba ^{1}H NMR (400 MHz, $CDCl_3$)



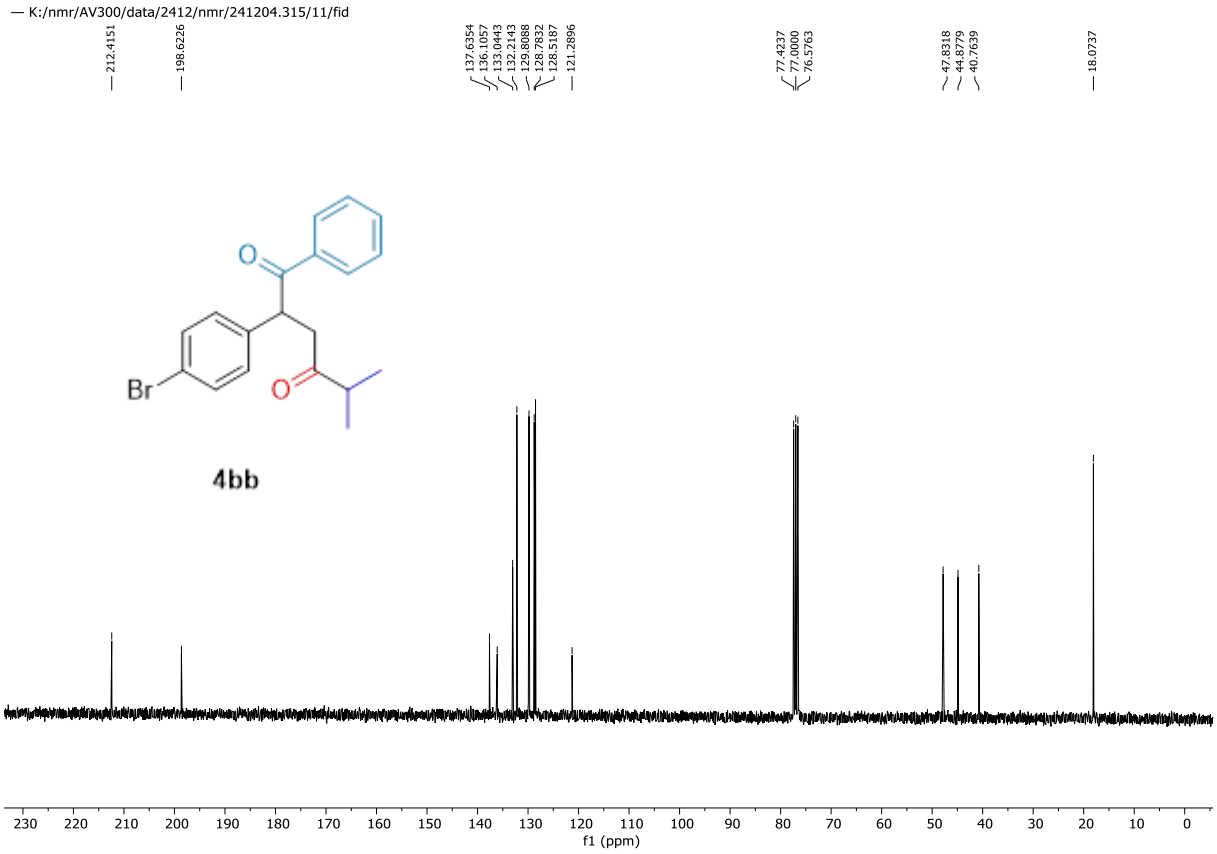
4ba ^{13}C NMR (101 MHz, $CDCl_3$)



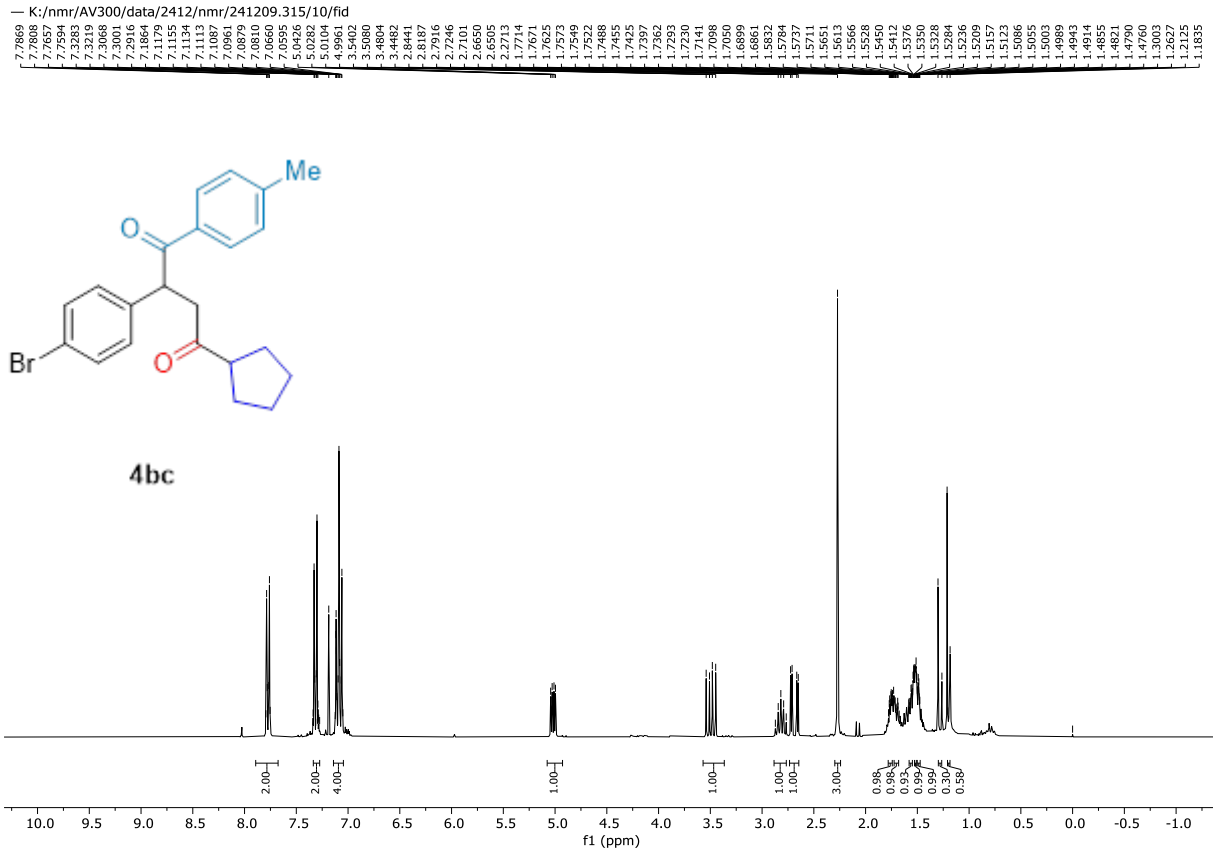
4bb ^{1}H NMR (300 MHz, $CDCl_3$)



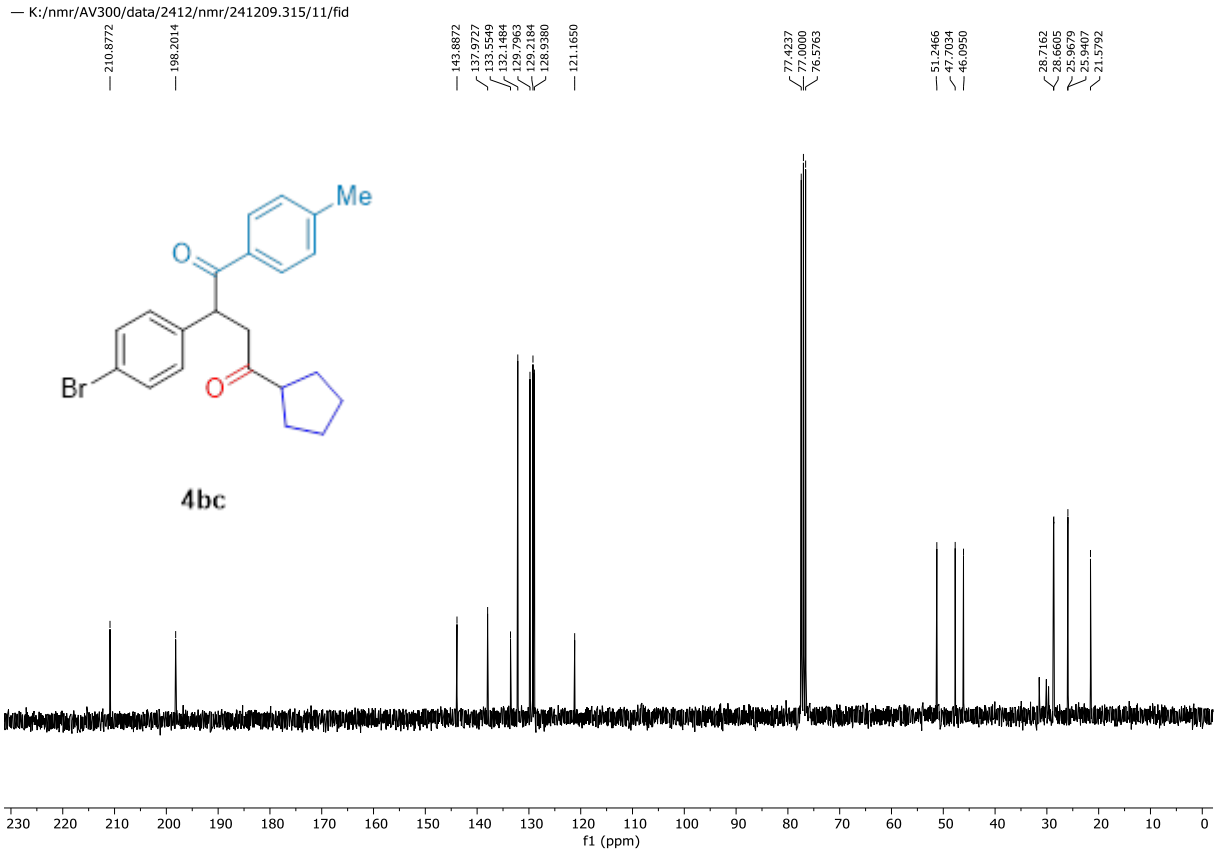
4bb ^{13}C NMR (75 MHz, $CDCl_3$)



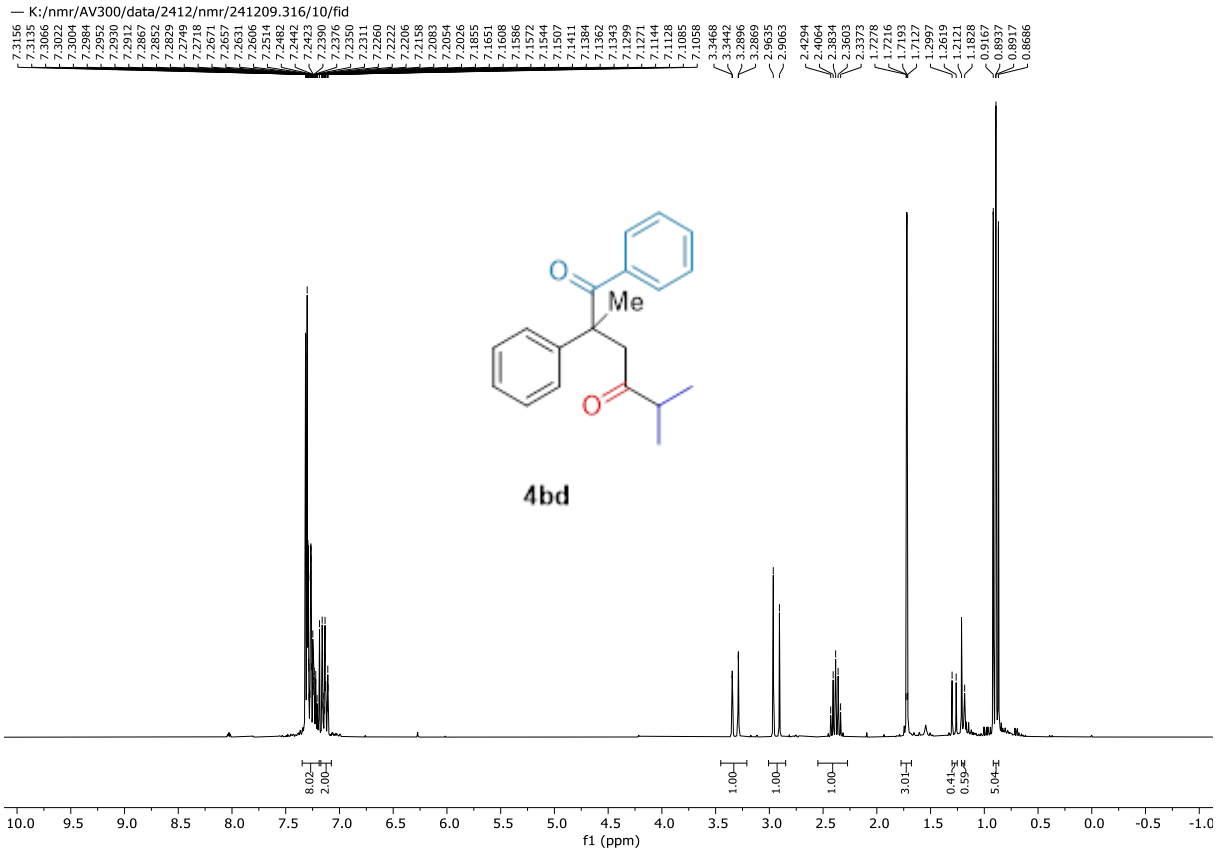
4bc ^{1}H NMR (300 MHz, $CDCl_3$)



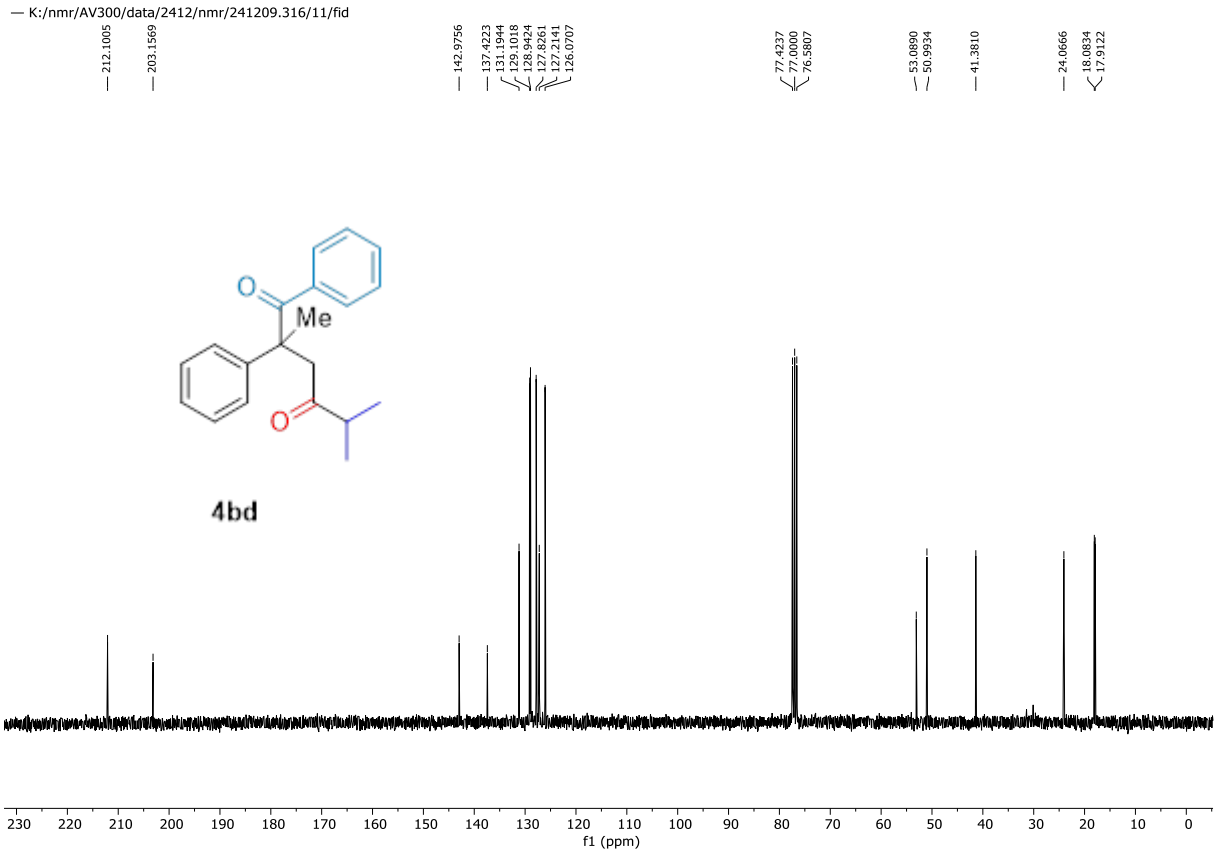
4bc ^{13}C NMR (75 MHz, $CDCl_3$)



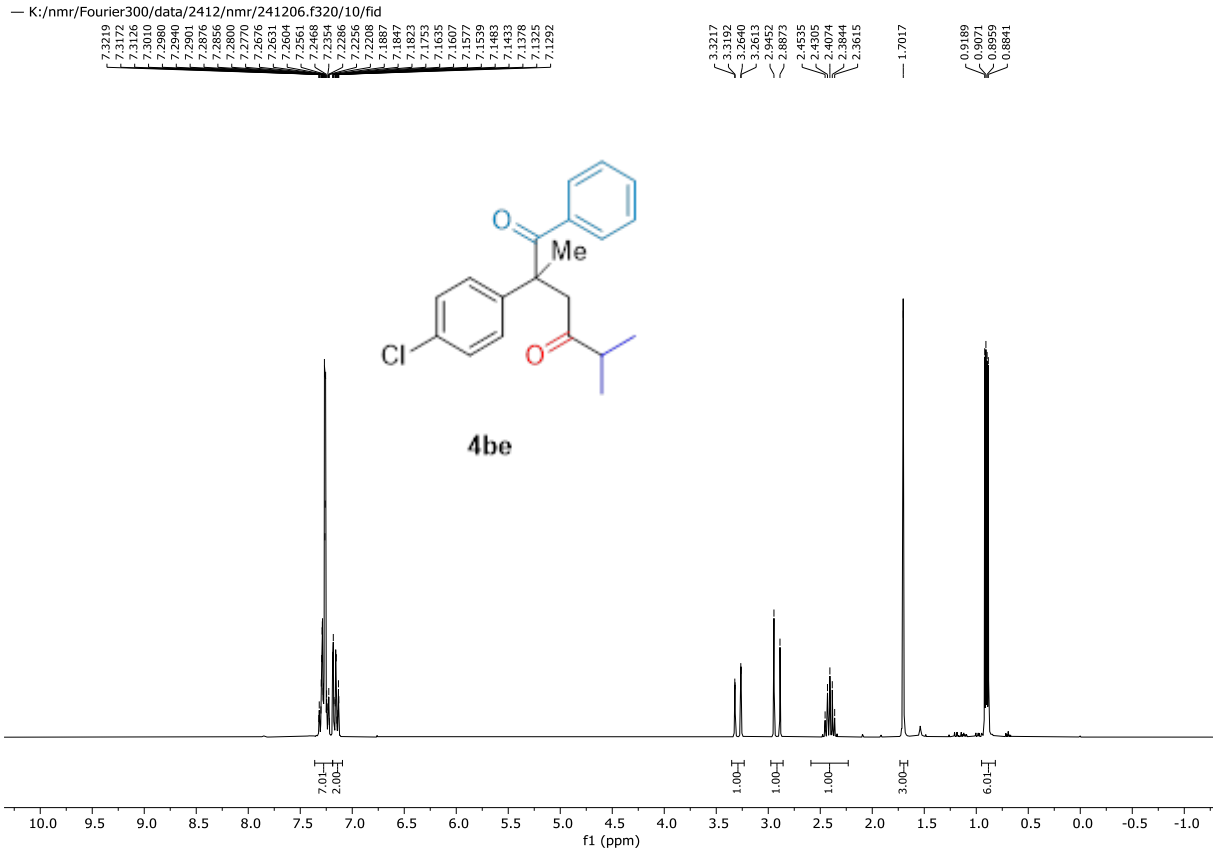
4bd ^{1}H NMR (300 MHz, $CDCl_3$)



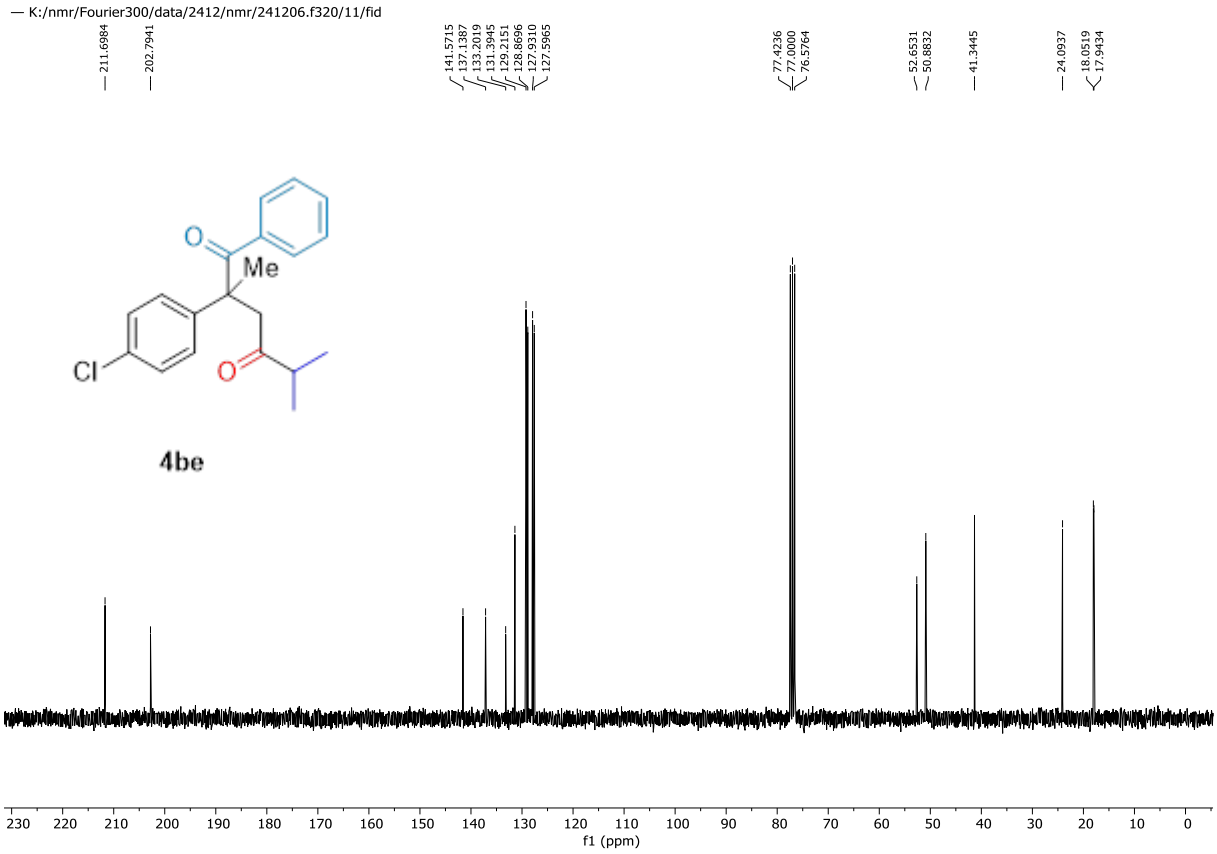
4bd ^{13}C NMR (75 MHz, $CDCl_3$)



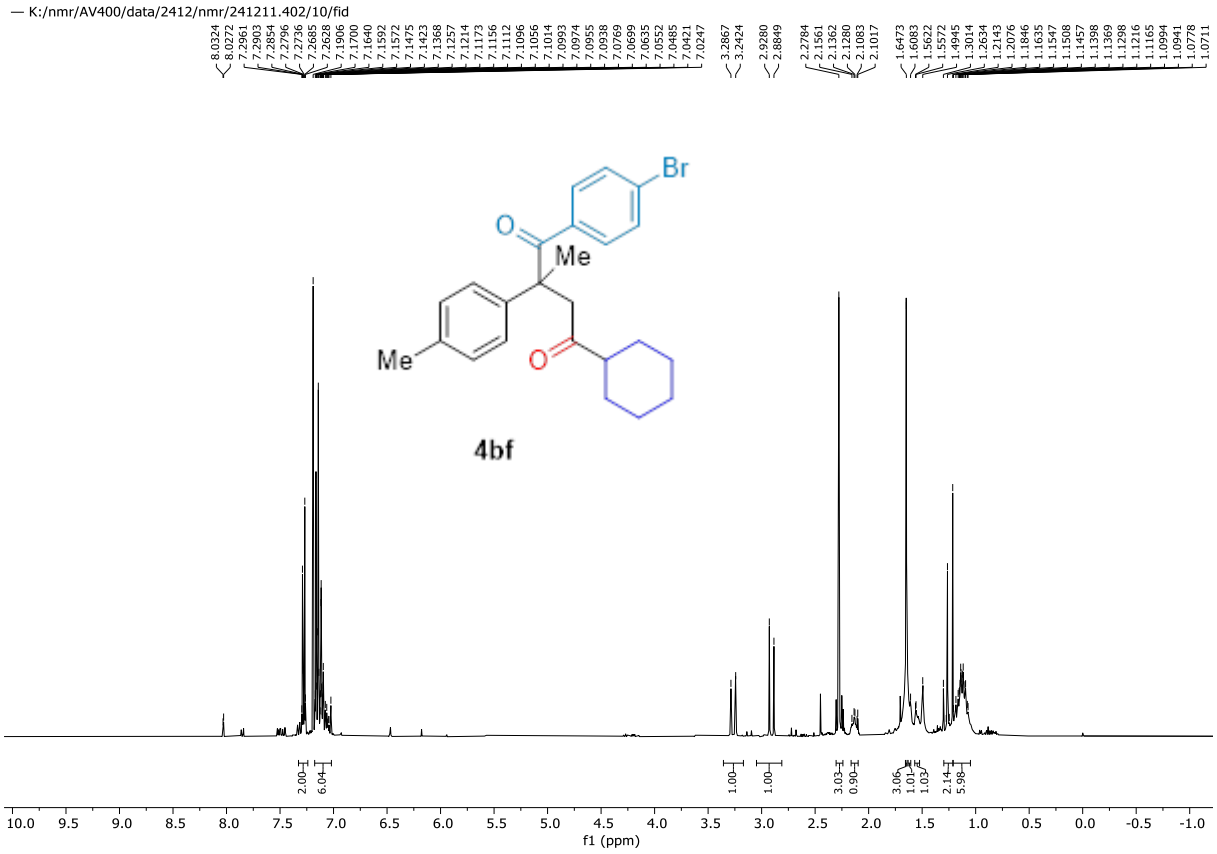
4be ^{1}H NMR (300 MHz, $CDCl_3$)



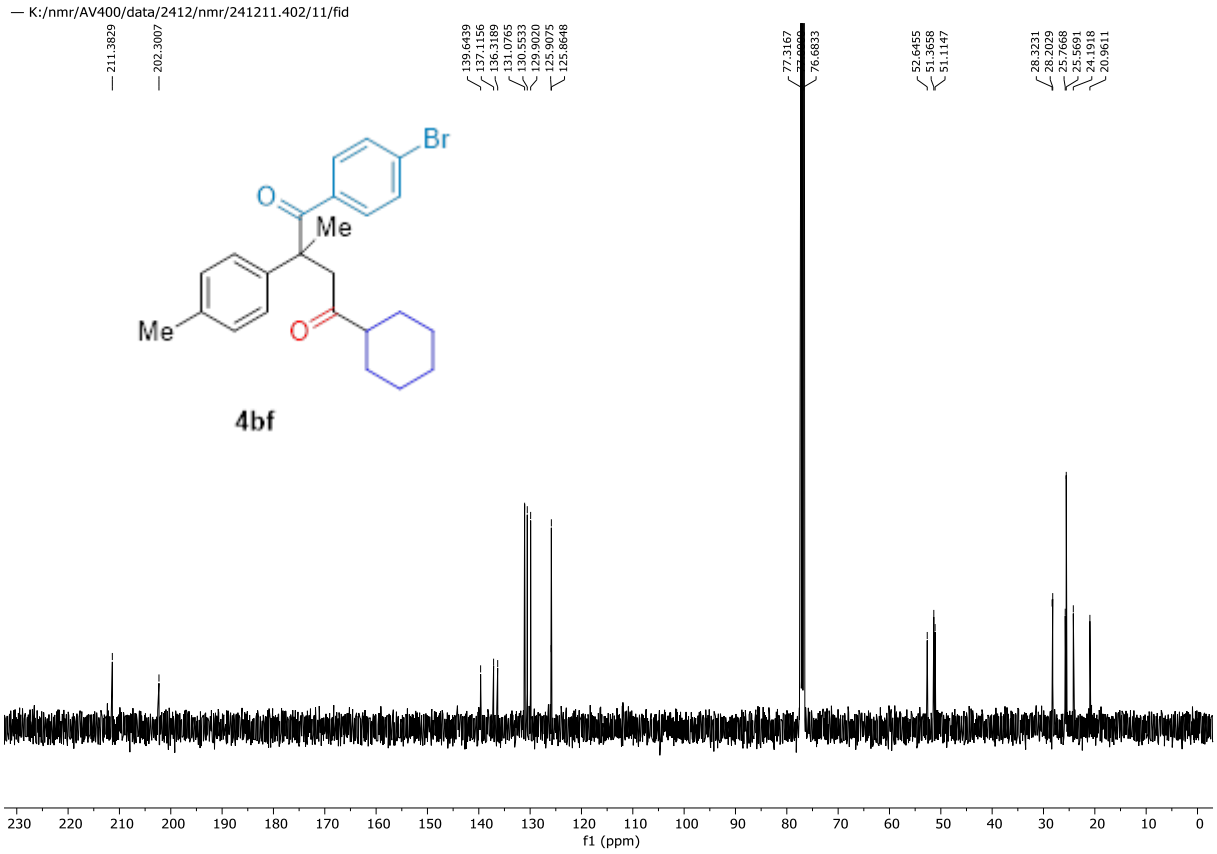
4be ^{13}C NMR (75 MHz, $CDCl_3$)



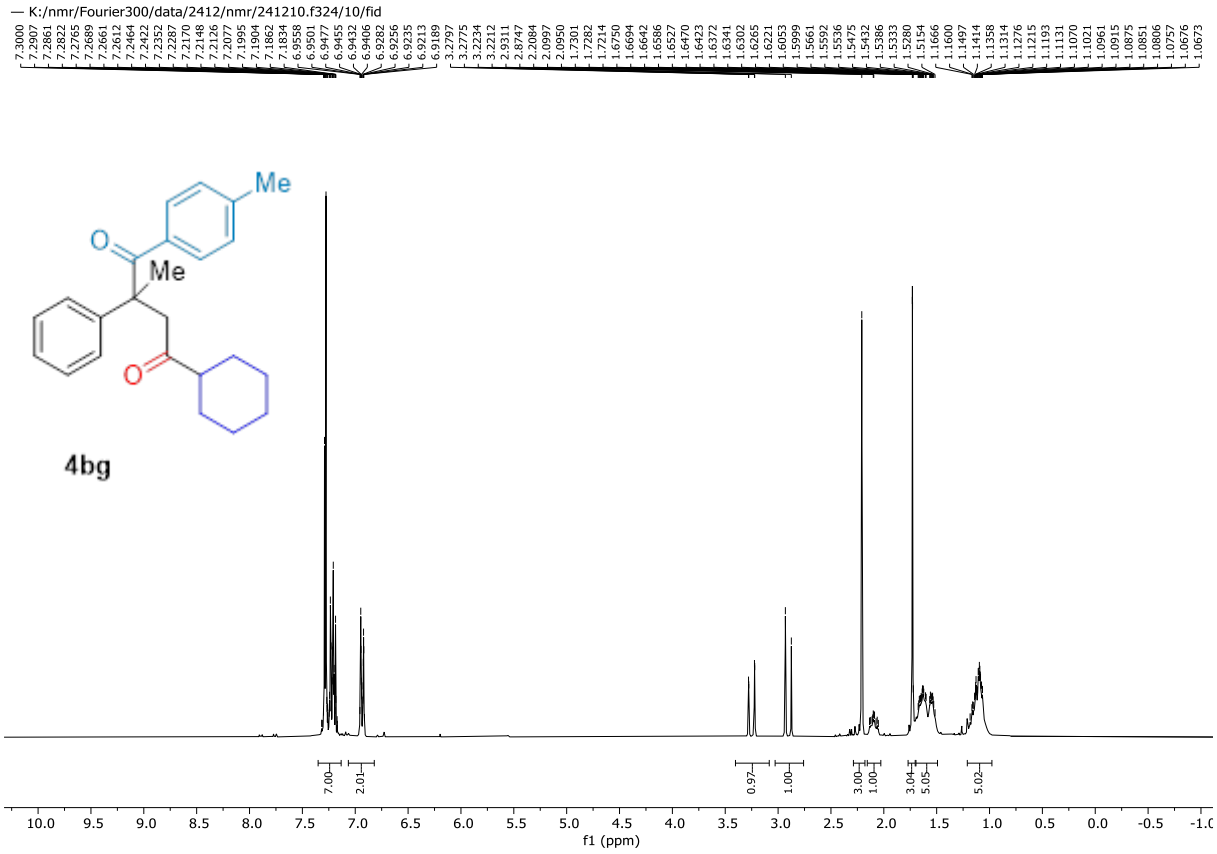
4bf ^{1}H NMR (400 MHz, $CDCl_3$)



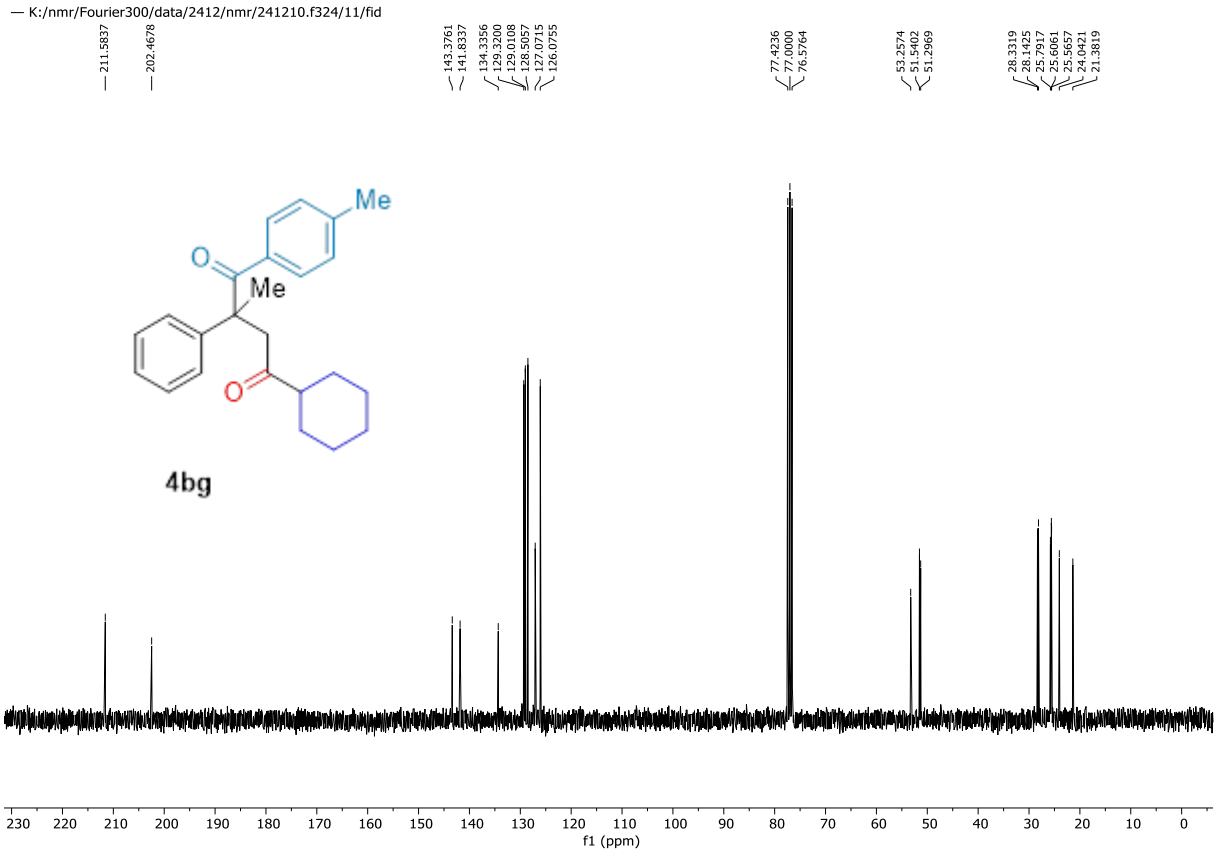
4bf ^{13}C NMR (101 MHz, $CDCl_3$)



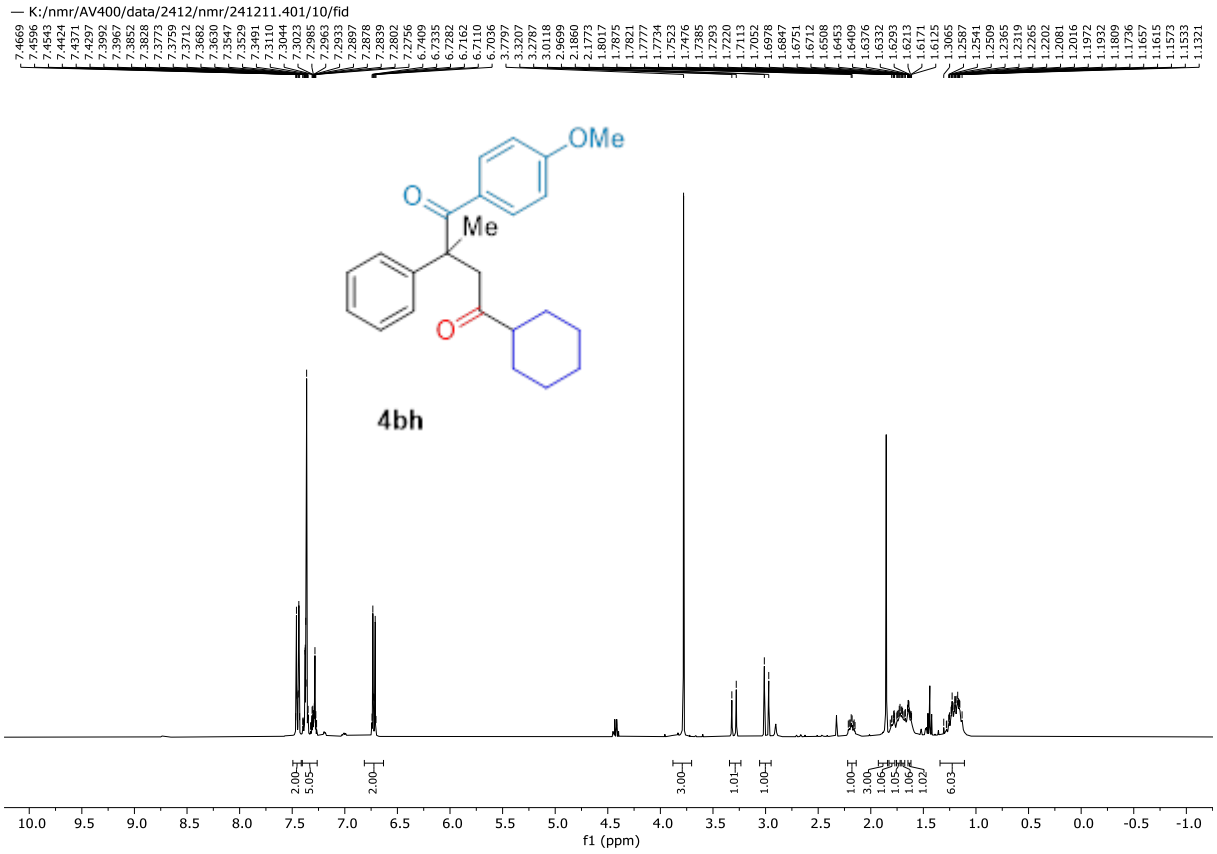
4bg ^{1}H NMR (300 MHz, $CDCl_3$)



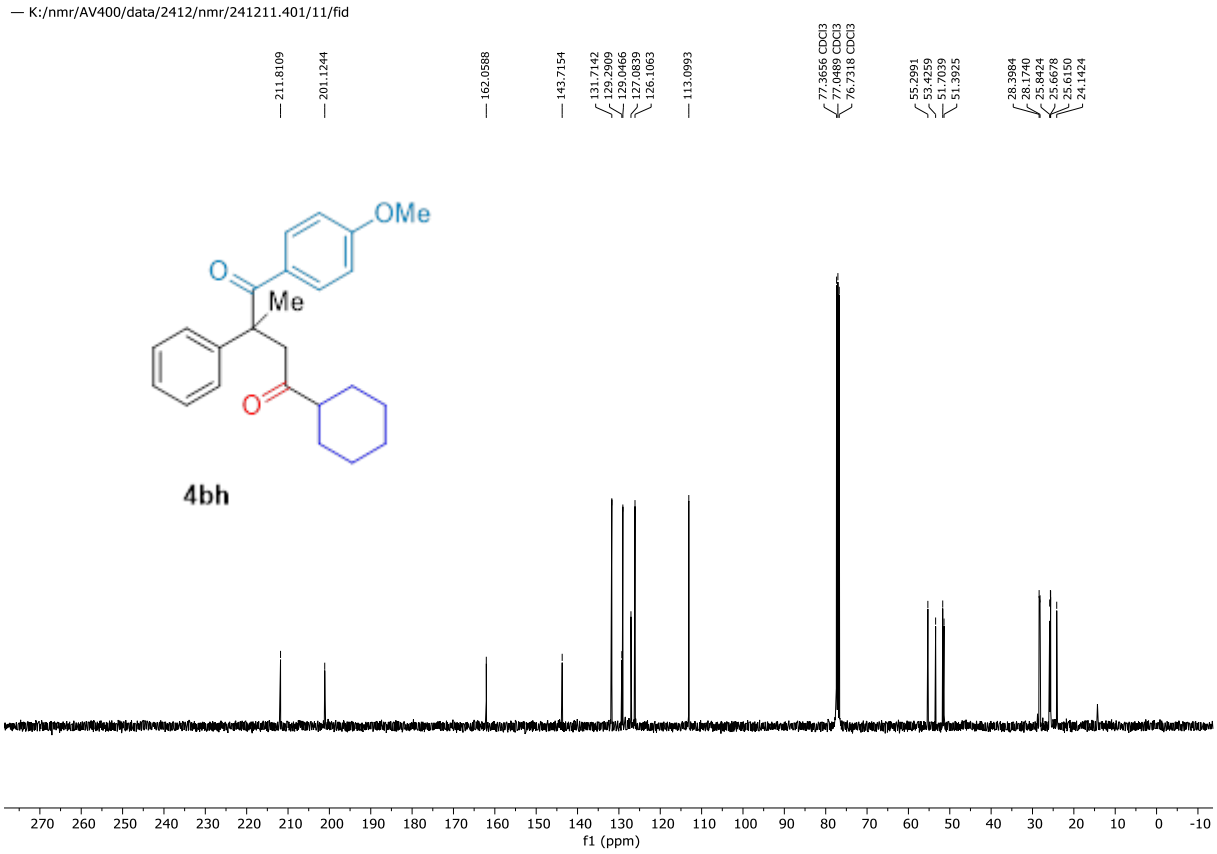
4bg ^{13}C NMR (75 MHz, $CDCl_3$)



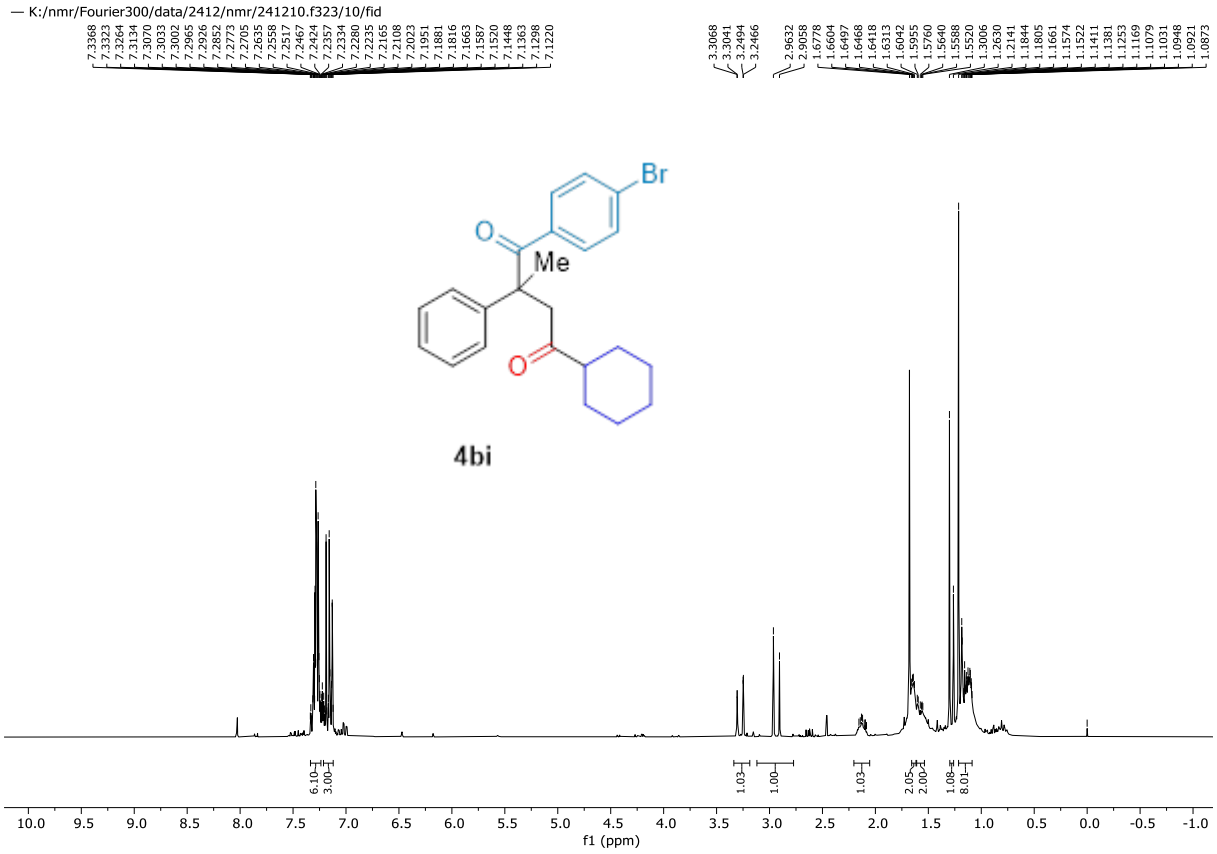
4bh ^{1}H NMR (400 MHz, $CDCl_3$)



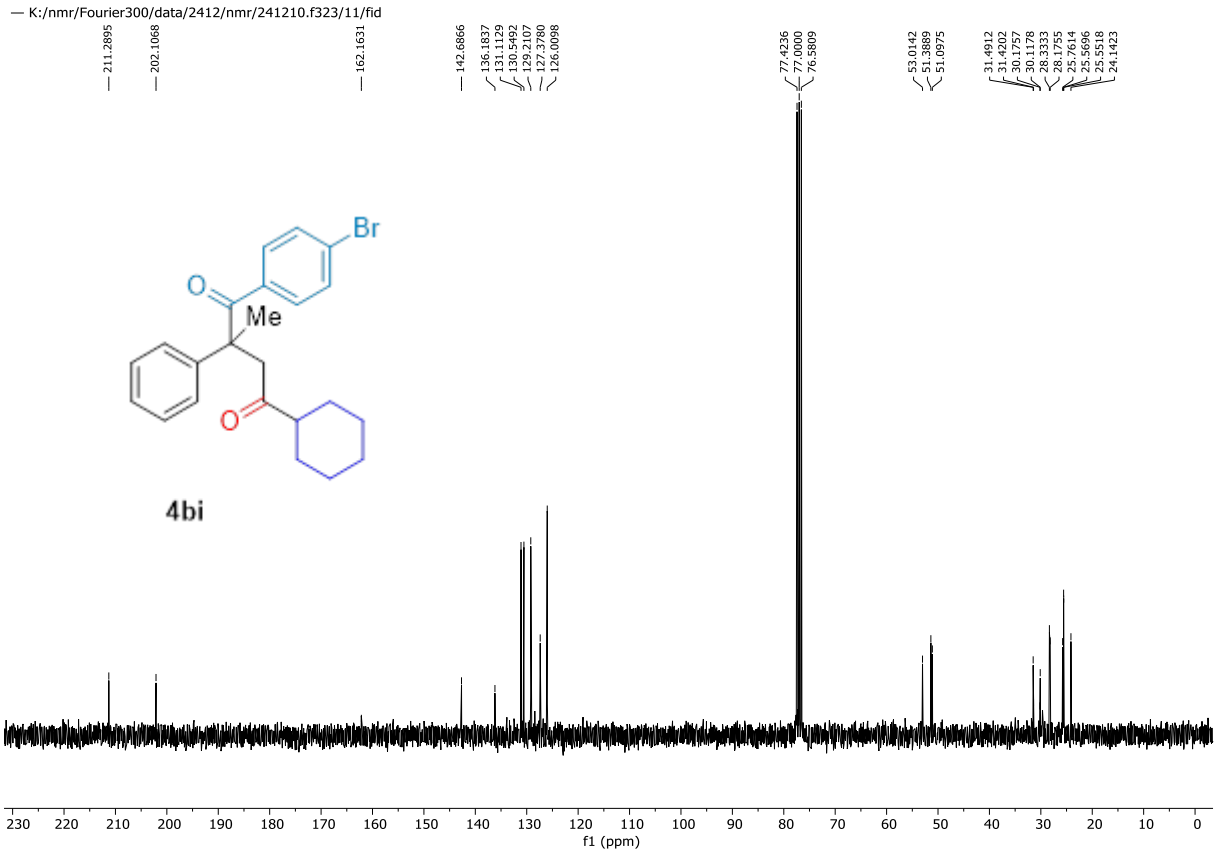
4bh ^{13}C NMR (101 MHz, $CDCl_3$)



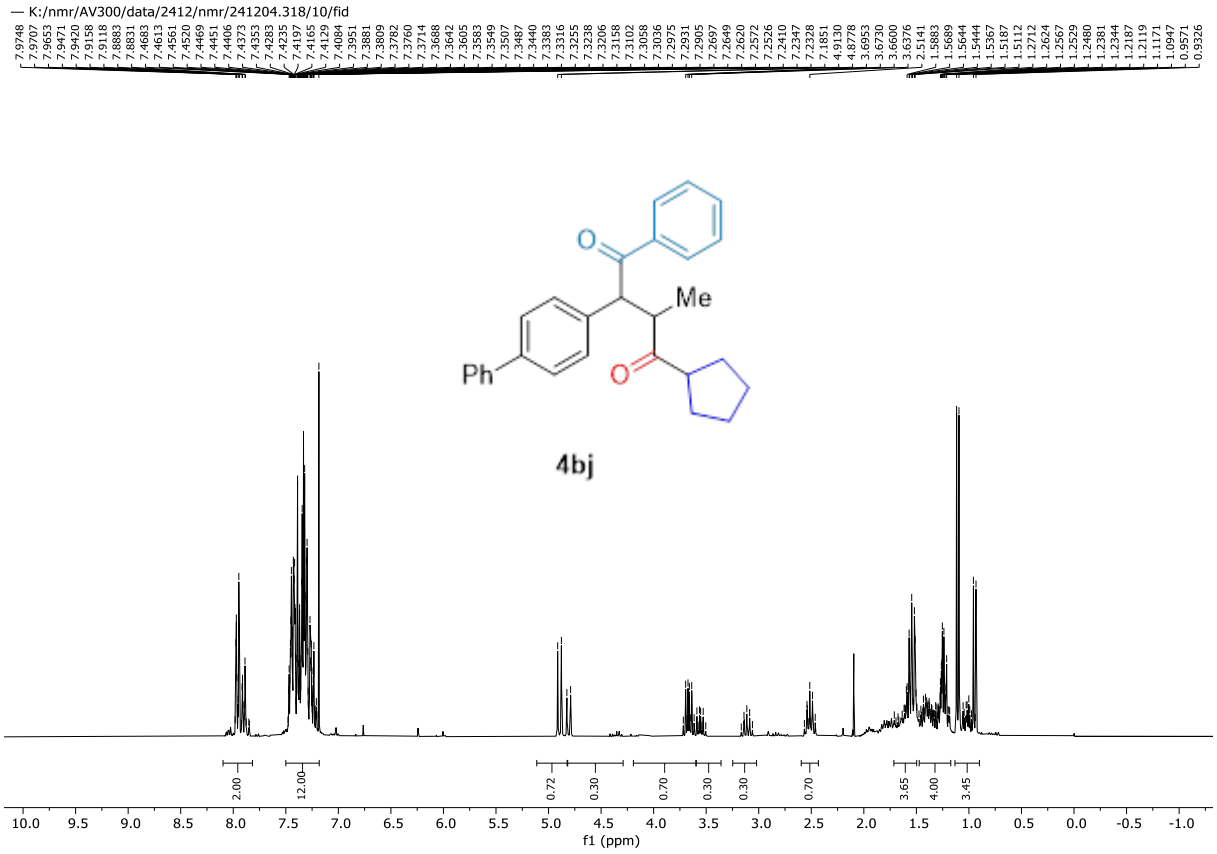
4bi ^{1}H NMR (300 MHz, $CDCl_3$)



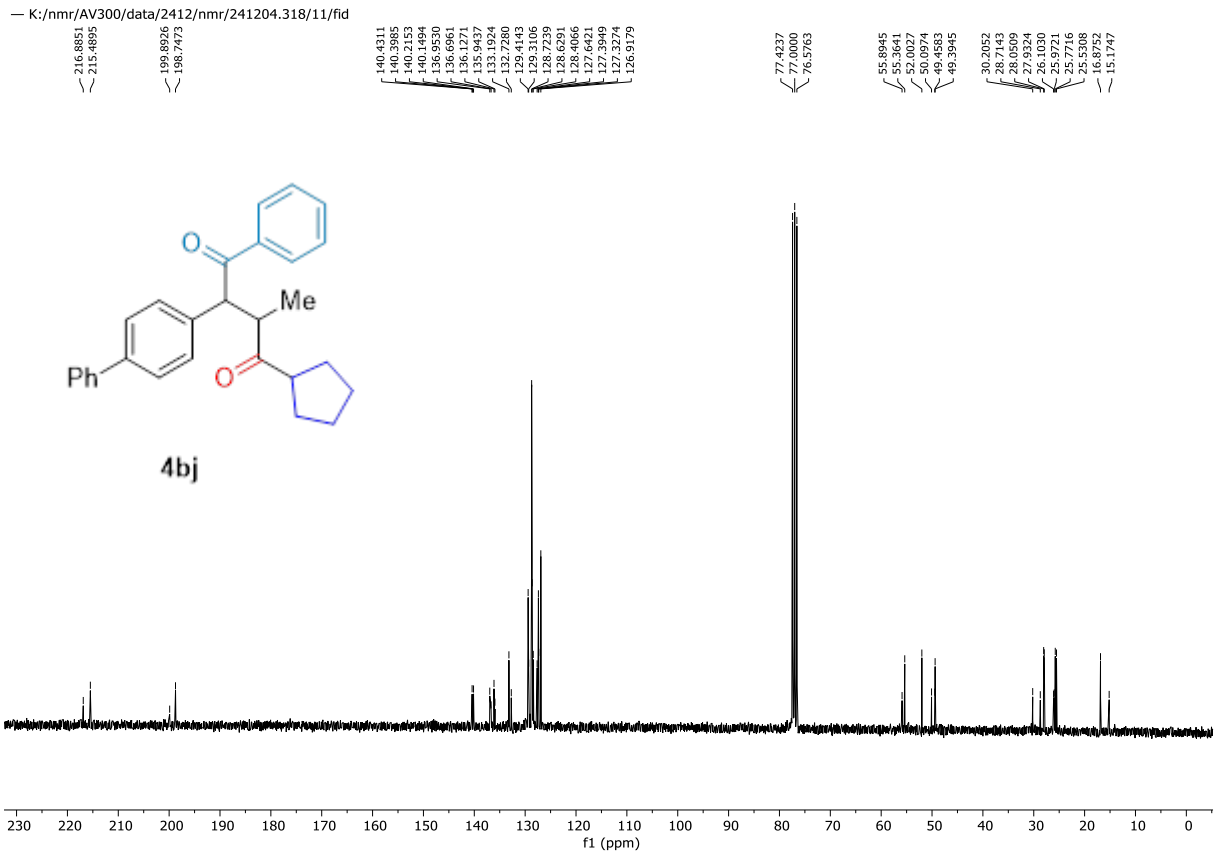
4bi ^{13}C NMR (75 MHz, $CDCl_3$)



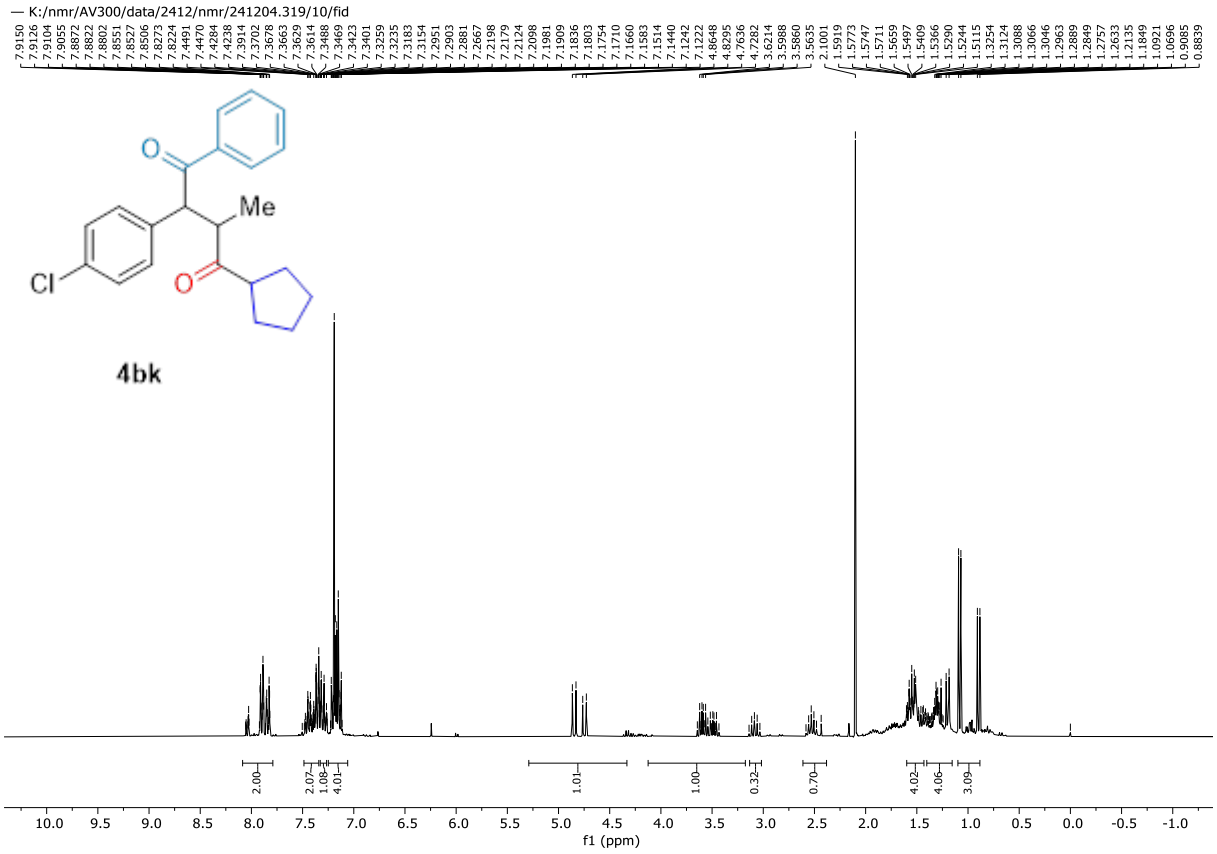
4bj ^{1}H NMR (300 MHz, $CDCl_3$)



4bj ^{13}C NMR (75 MHz, $CDCl_3$)



4bk ^{1}H NMR (300 MHz, $CDCl_3$)



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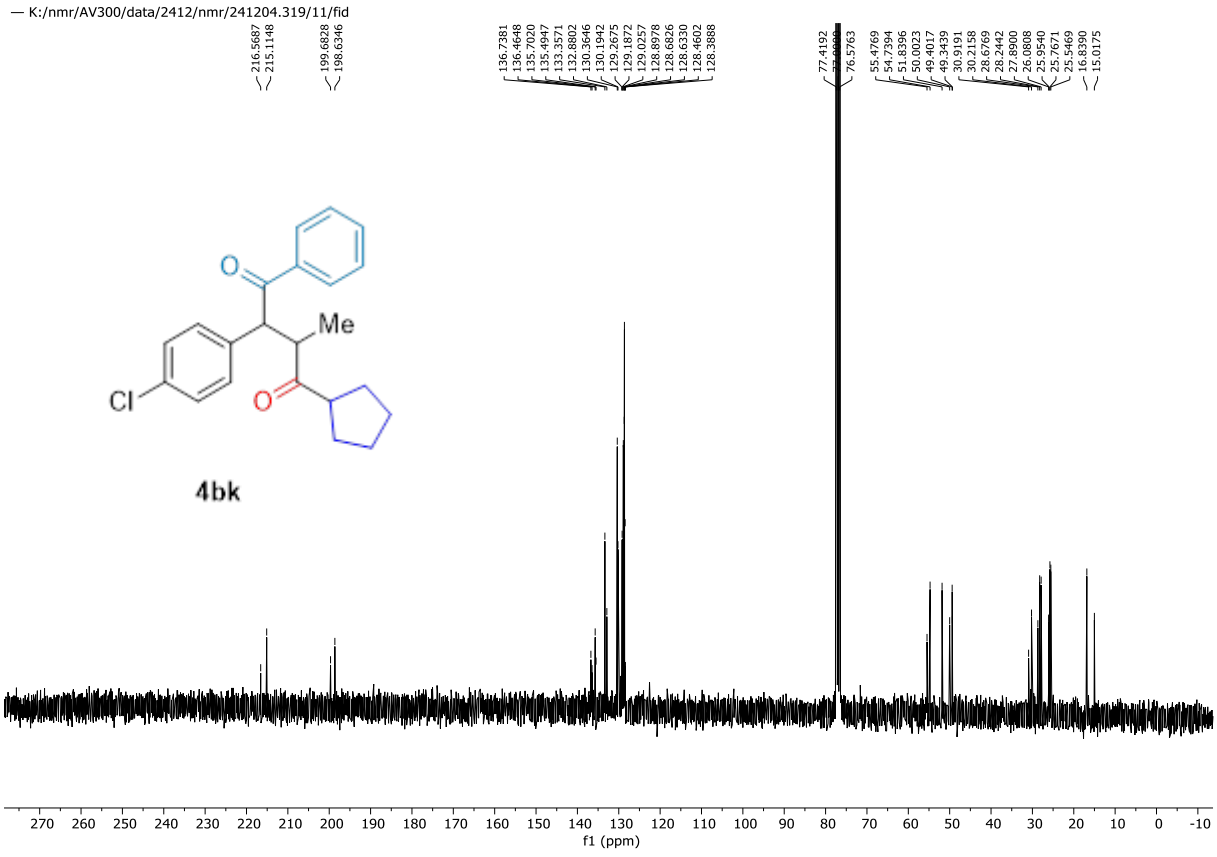
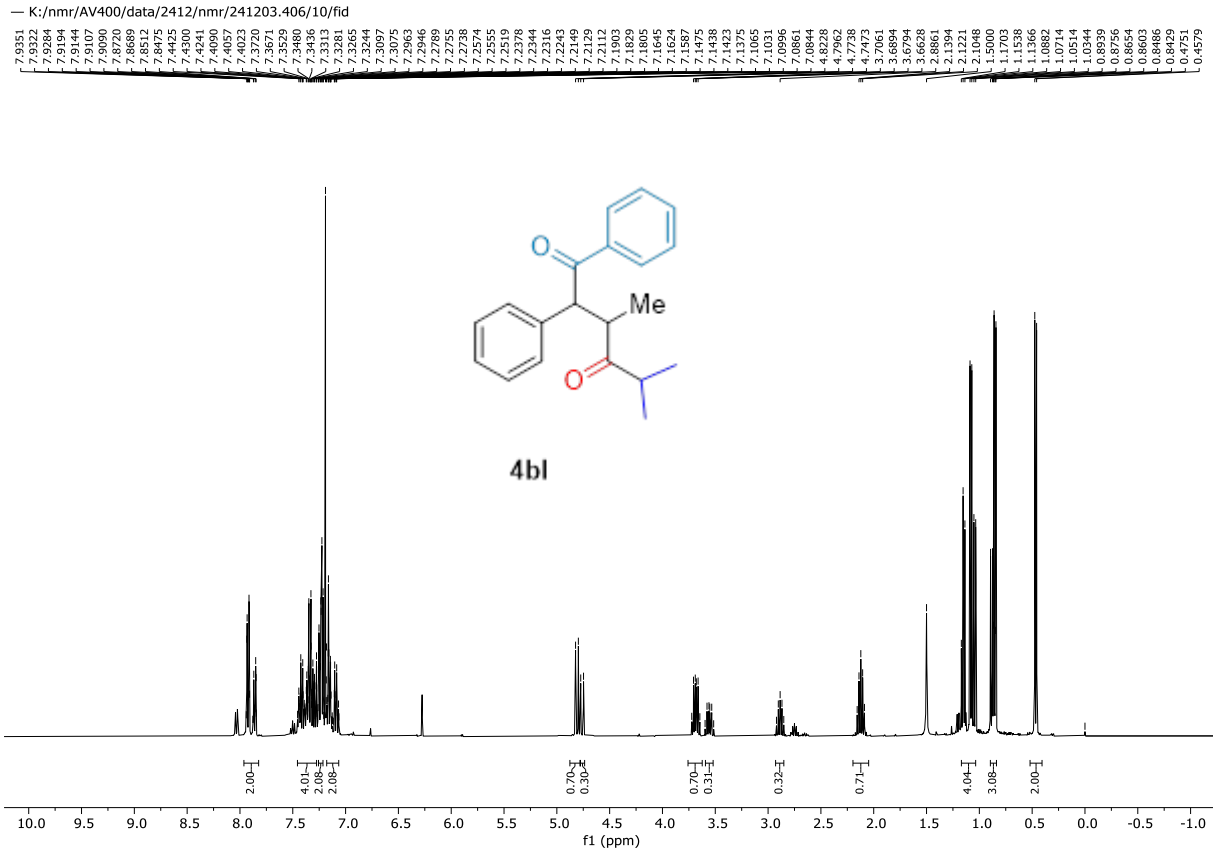
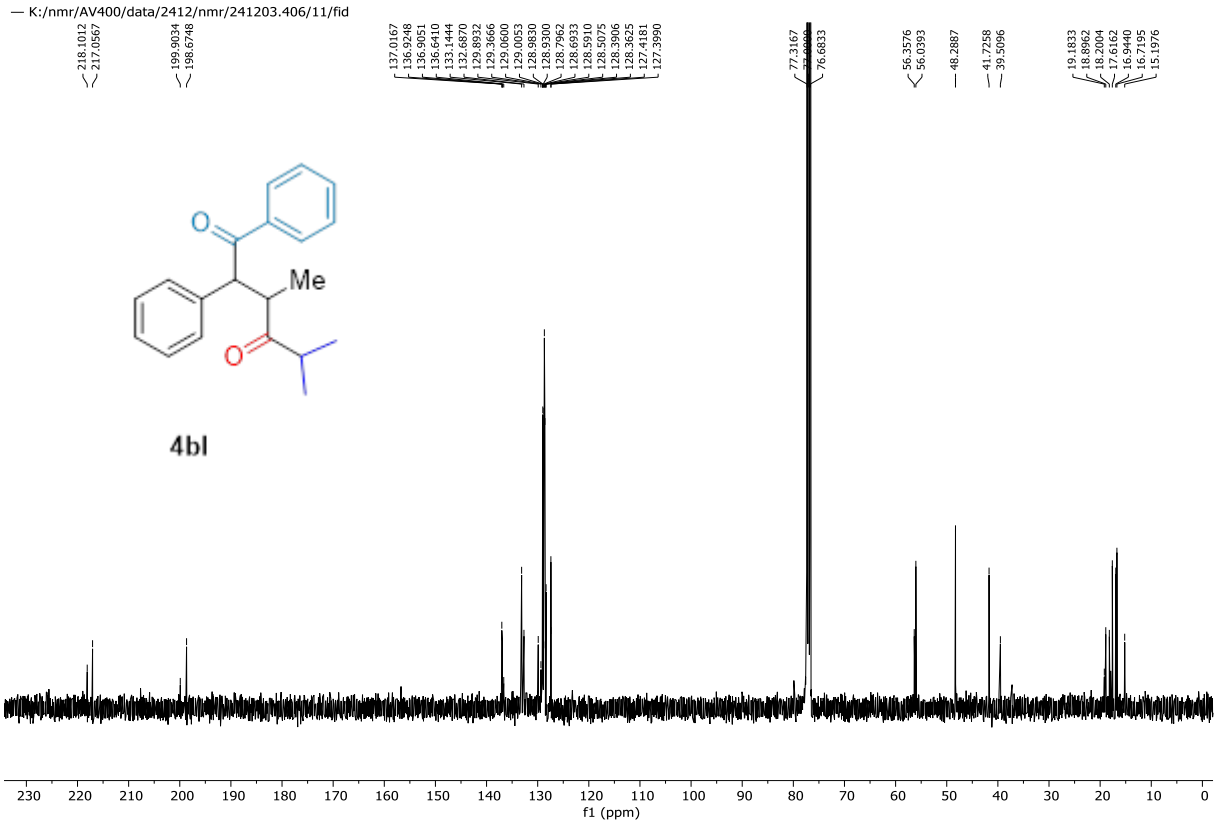


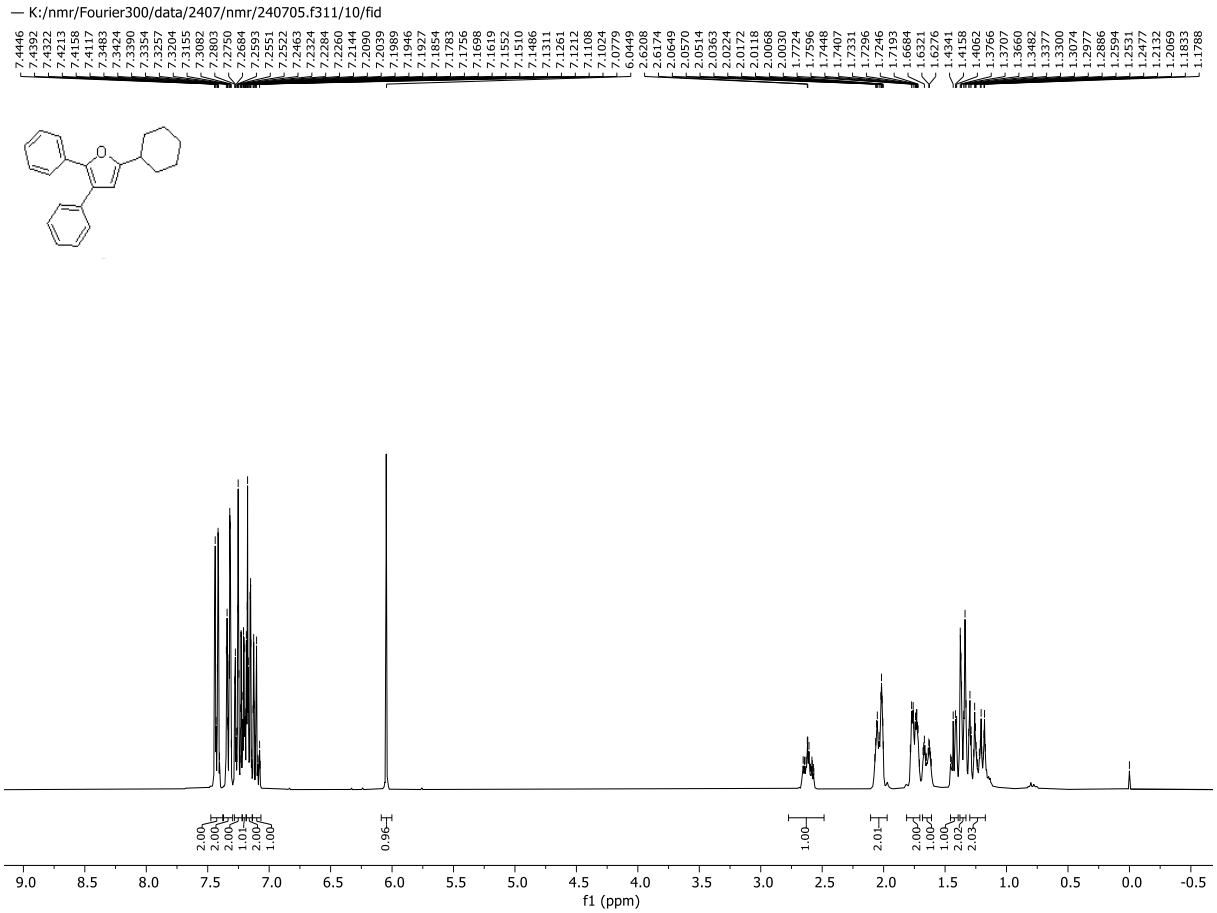
4bl ^{1}H NMR (400 MHz, $CDCl_3$)



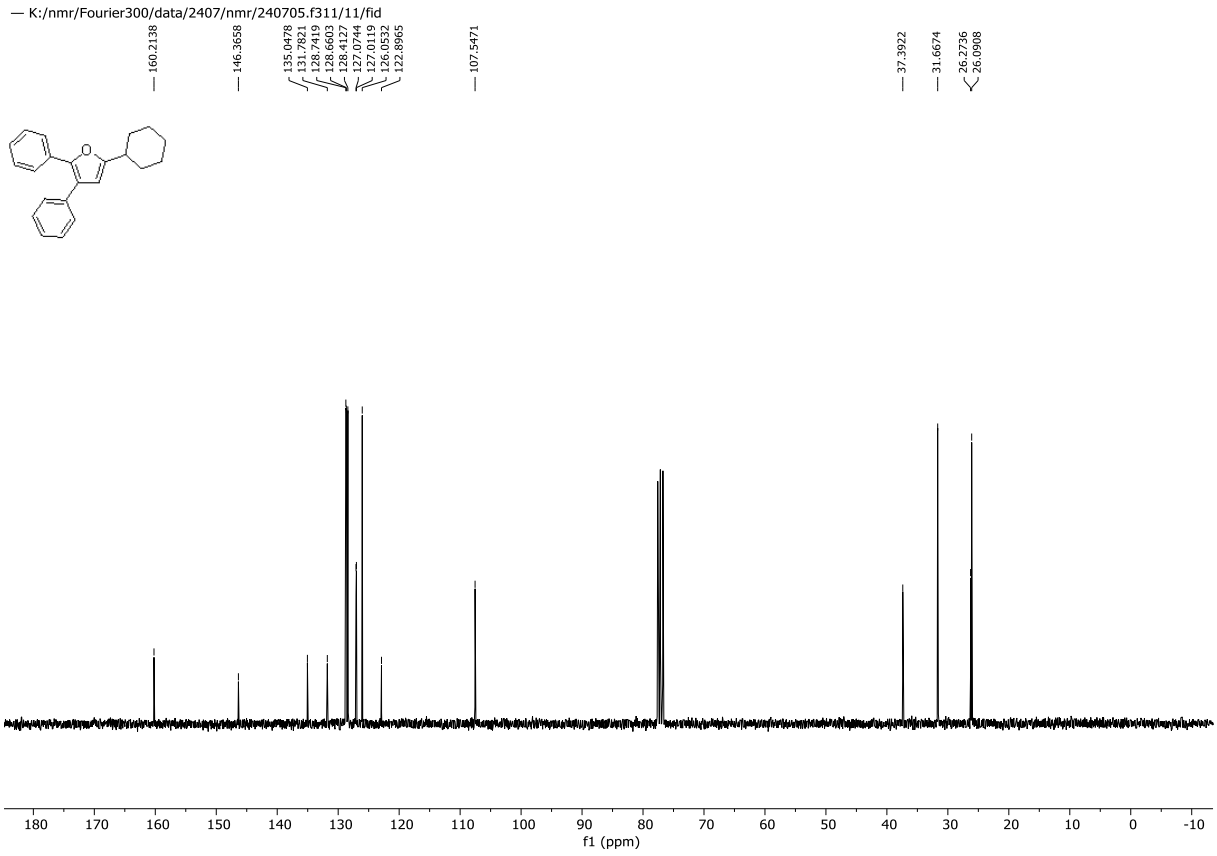
4bl ^{13}C NMR (101 MHz, $CDCl_3$)



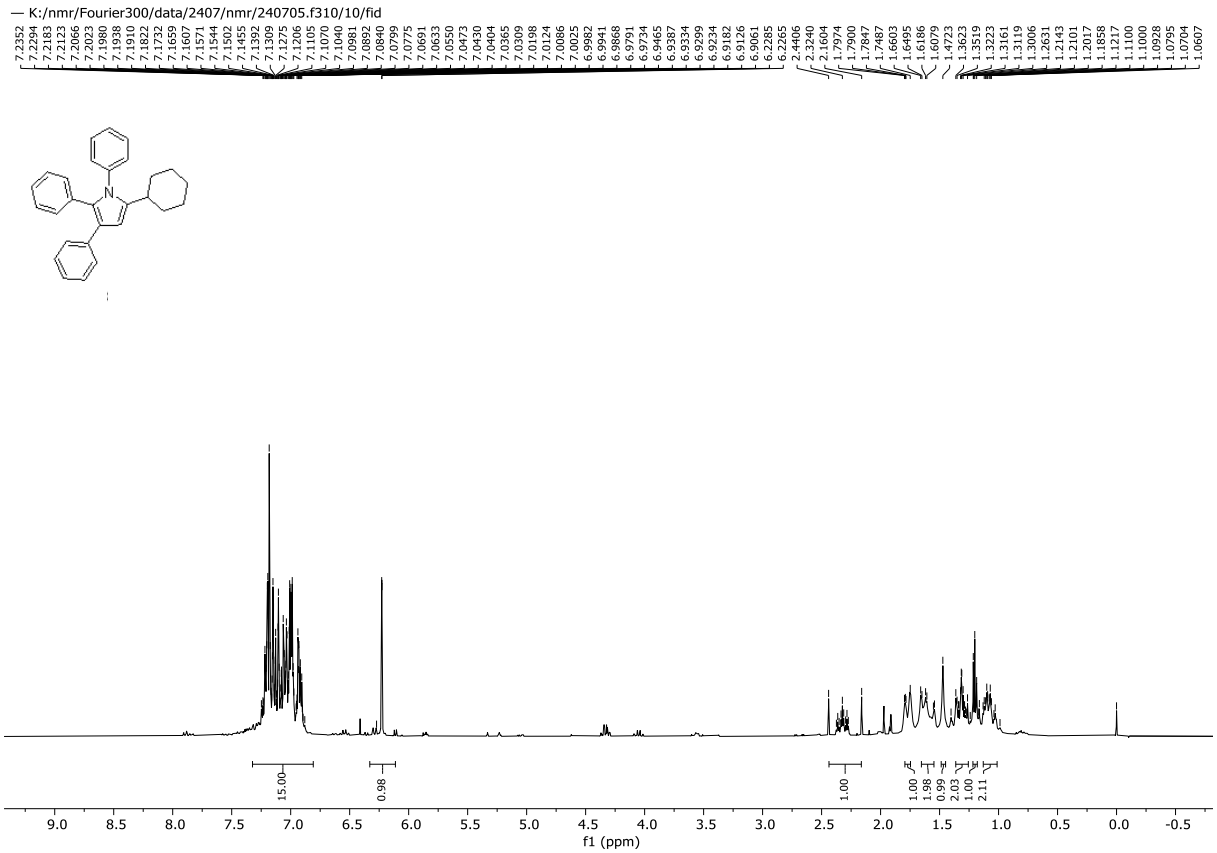
6a ^{1}H NMR (300 MHz, $CDCl_3$)



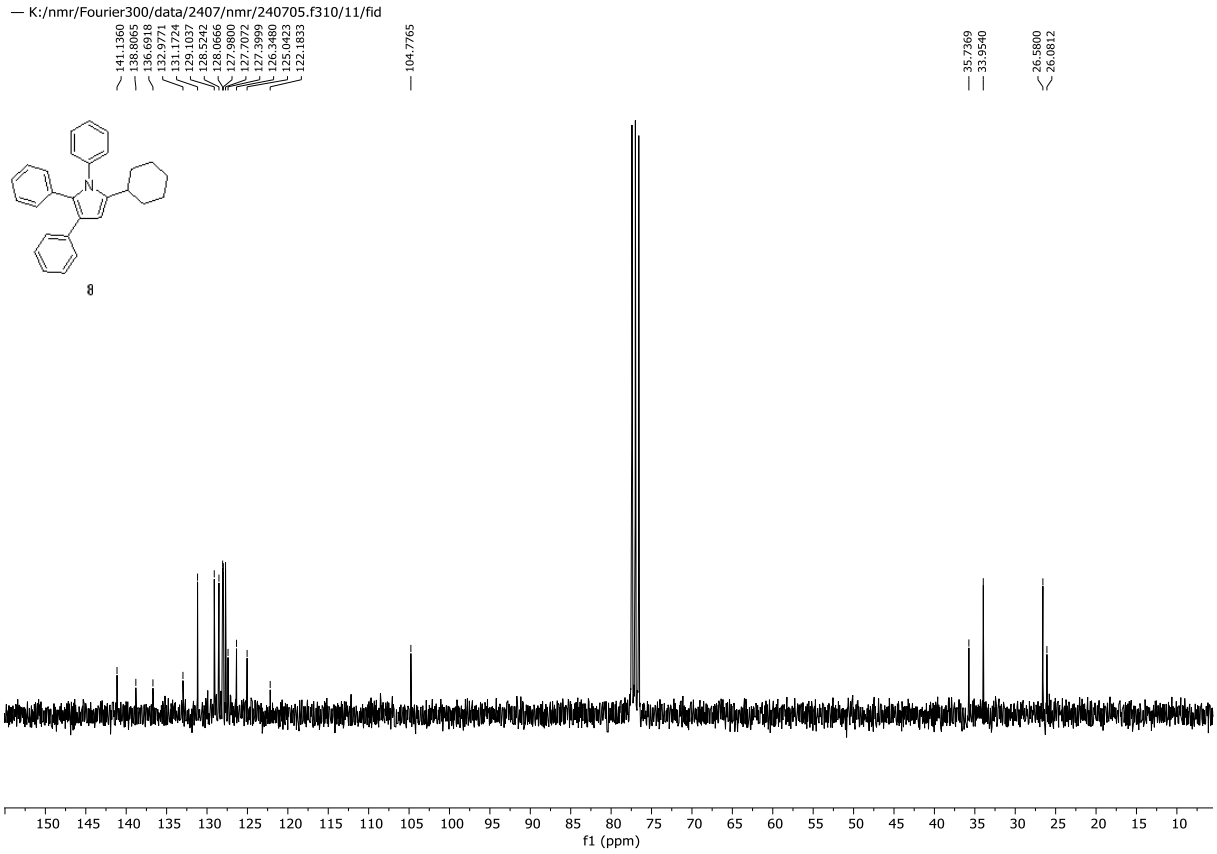
6a ^{13}C NMR (75 MHz, $CDCl_3$)



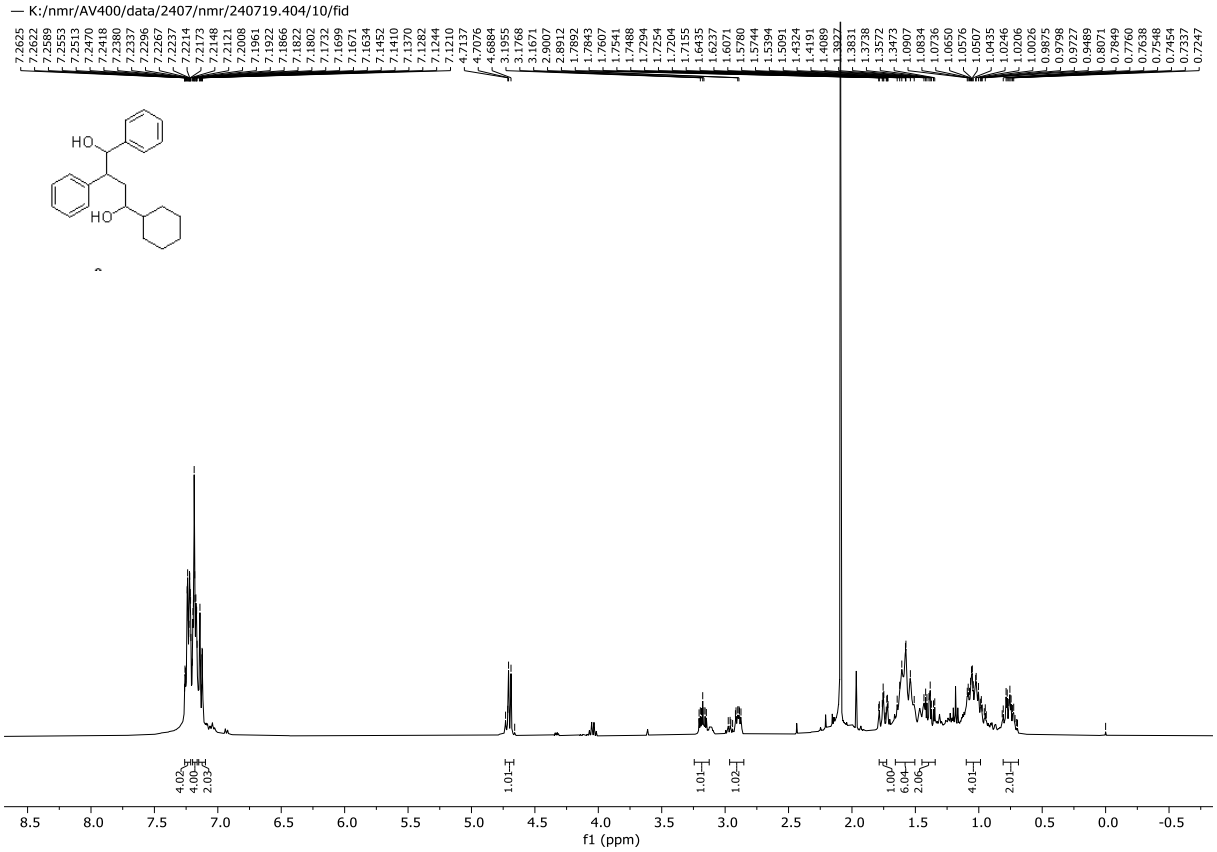
6b ^{1}H NMR (300 MHz, $CDCl_3$)



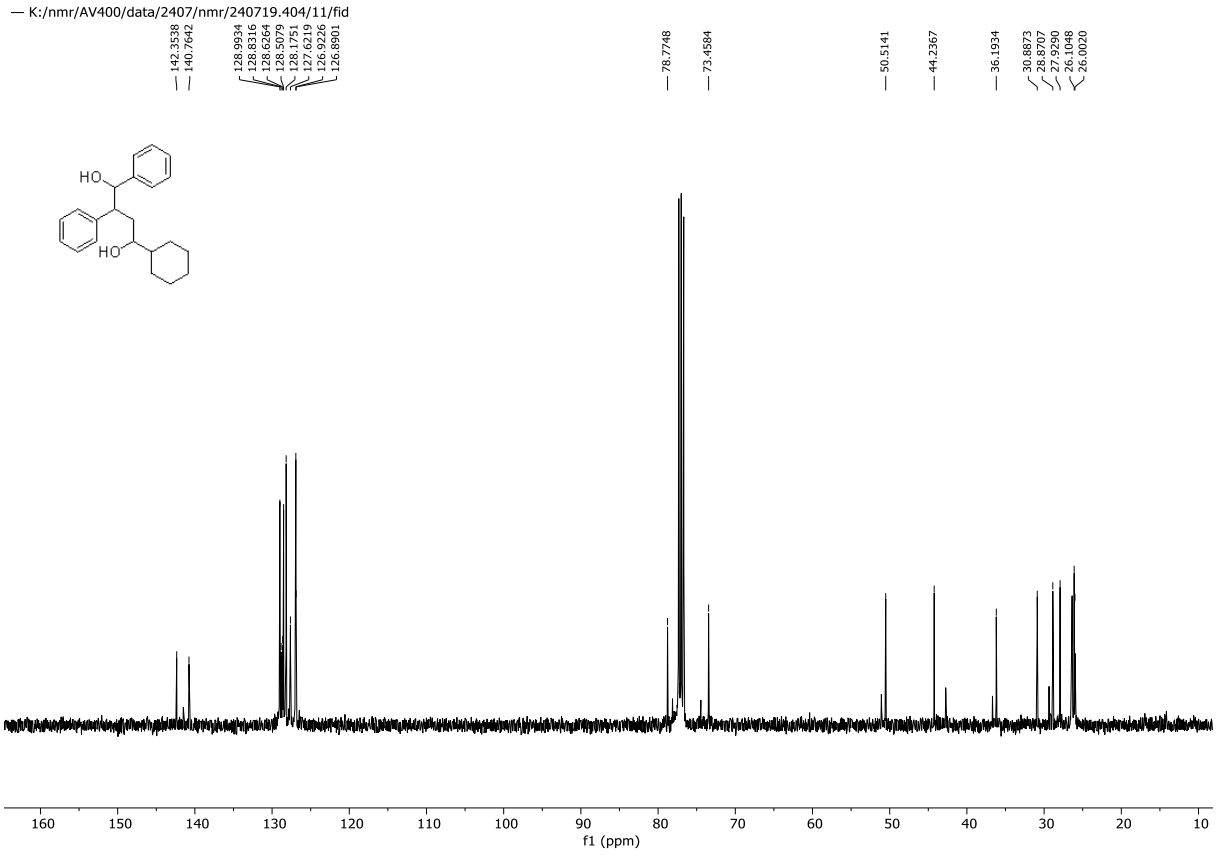
6b ^{13}C NMR (75 MHz, $CDCl_3$)



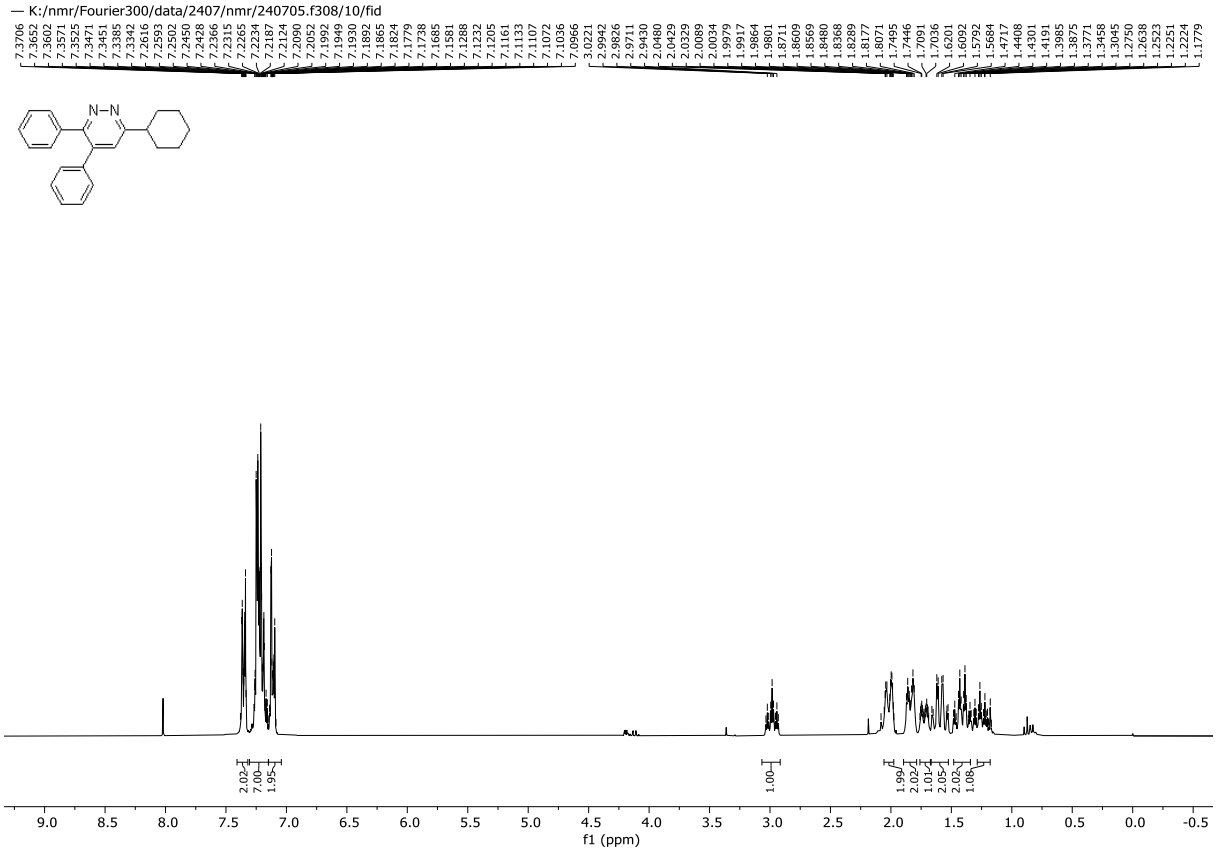
6c ^{1}H NMR (400 MHz, $CDCl_3$)



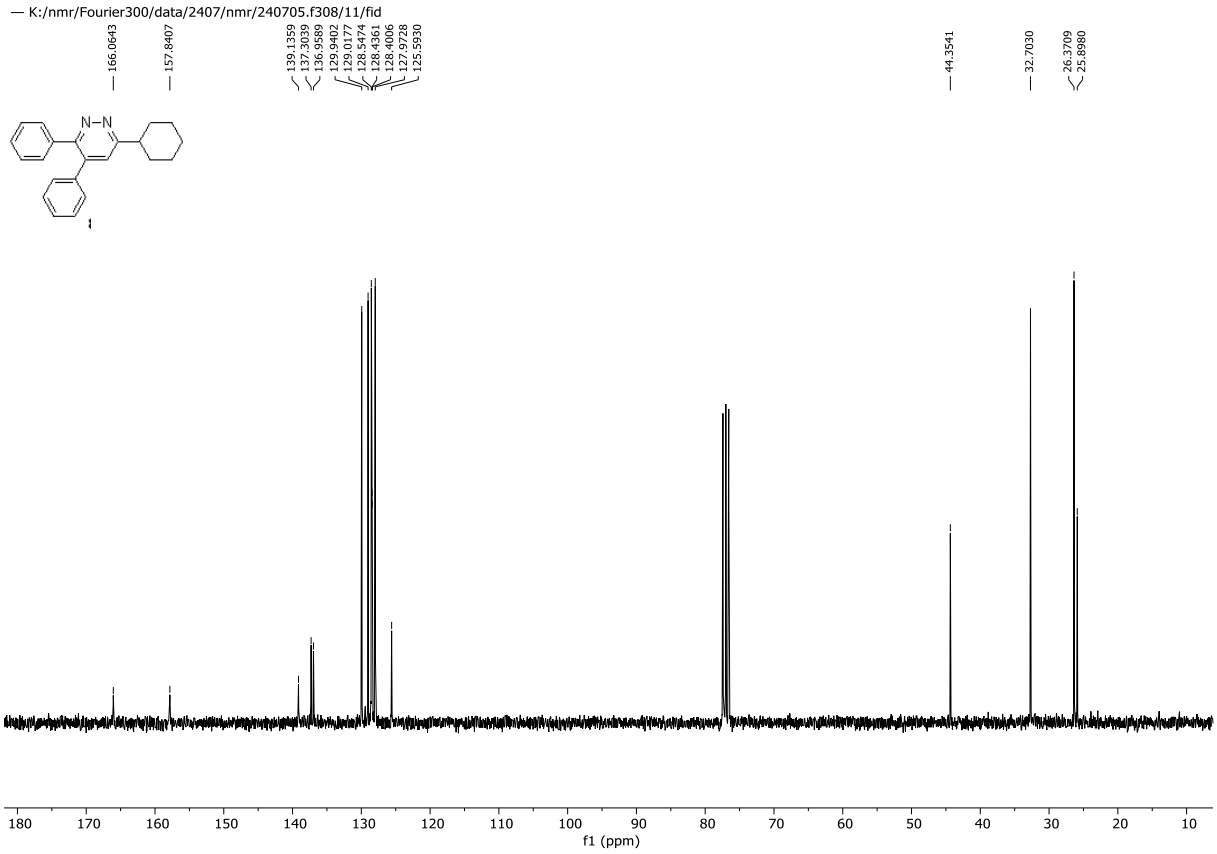
6c ^{13}C NMR (101 MHz, $CDCl_3$)



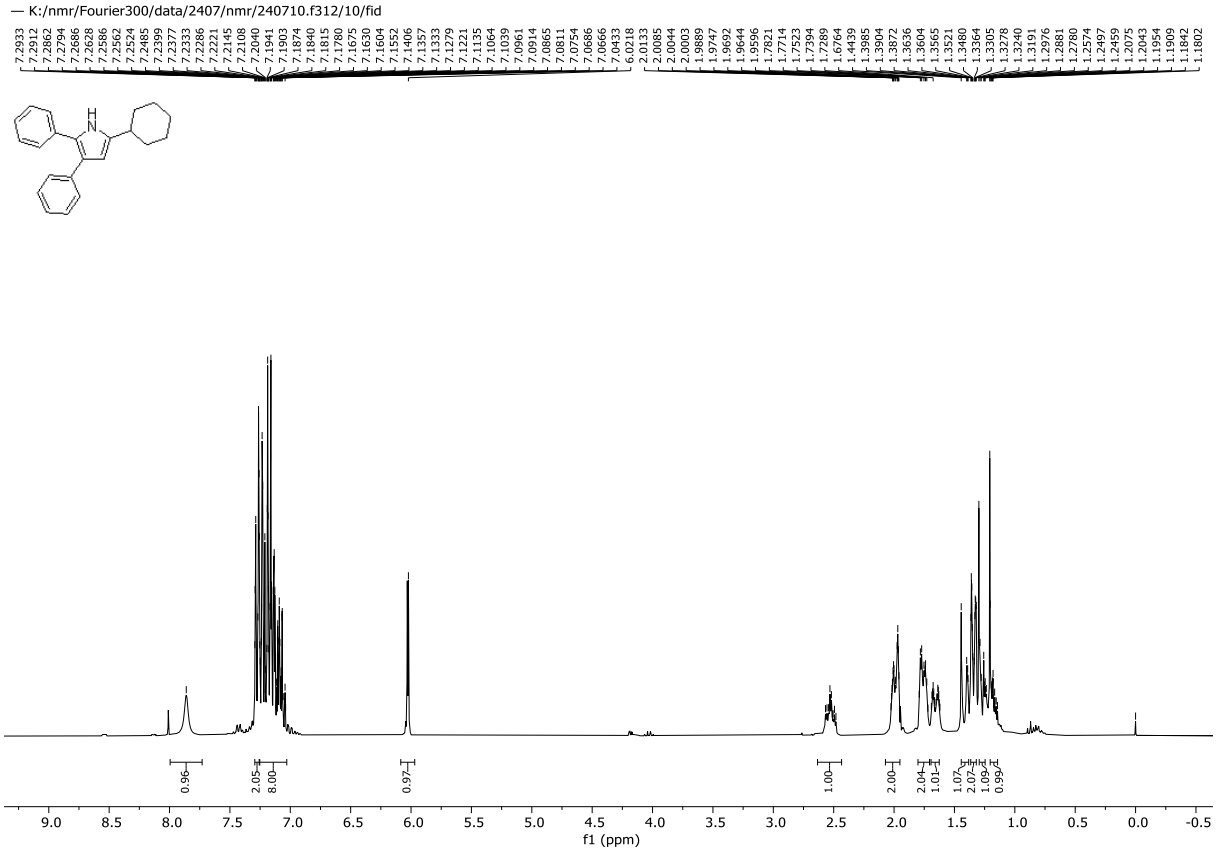
6d ^{1}H NMR (300 MHz, $CDCl_3$)



6d ^{13}C NMR (75 MHz, $CDCl_3$)



6e ^{1}H NMR (300 MHz, $CDCl_3$)



6e ^{13}C NMR (75 MHz, $CDCl_3$)