

## Electronic Supplementary Information

### Visible-light-mediated $\beta$ -acylative divergent alkene difunctionalization with Katritzky salt/CO<sub>2</sub>

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## Electronic Supplementary Information

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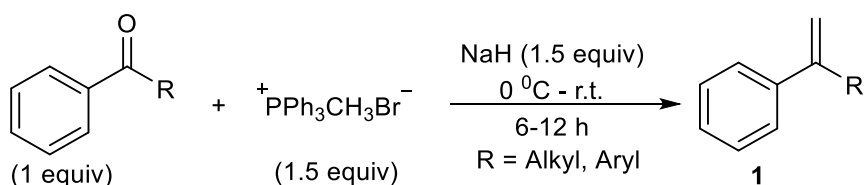
## 1. General information

Melting points were determined in open end-capillary tubes and are uncorrected. TLC was performed on silica gel plates (Merck silica gel 60, f254), and the spots were visualized with UV light (254 and 365 nm) and KMnO<sub>4</sub> stain. X-ray of crystals was recorded in Bruker D8 Venture with a Photon-III detector instrument. <sup>1</sup>H NMR was recorded at 400 MHz (JEOL-JNM-ECZ400S/L1) frequency and 600 MHz (Bruker-Avance) frequency; <sup>13</sup>C NMR spectra were recorded at 100 MHz (JEOL-JNM-ECZ400S/L1) frequency and 150 MHz (Bruker-Avance) frequency in CDCl<sub>3</sub>, DMSO-D<sub>6</sub> and (DMSO-D<sub>6</sub> + 1 drop CDCl<sub>3</sub>) solvent using TMS as the internal standard. Chemical shifts were measured in parts per million (ppm) referenced to 0.0 ppm for tetramethylsilane. The following abbreviations were used to explain multiplicities: s=singlet, d=doublet, t=triplet, q=quartet, m=multiplet. Coupling constants, *J* were reported in Hertz unit (Hz). HRMS (m/z) were measured using ESI (Q-TOF, positive ion) technique. Unless otherwise stated, all commercial reagents were used without additional purification.

## 2. Preparation of starting materials

### 2.1. General procedure for preparation of substituted olefins (1)

Slightly modifying a literature protocol<sup>1</sup>, these were prepared by Wittig reaction as follows.



An oven-dried round-bottom flask was charged with CH<sub>3</sub>PPh<sub>3</sub>Br (1.5 equiv.) and THF (carbonyl substrate concentration = 0.2 M). NaH (1.5 equiv.) was added in portion to the suspension at 0 °C. The resulting mixture was allowed to warm up to room temperature and stirred for 1 h. The yellow suspension was cooled to 0 °C again followed by addition of the carbonyl substrate (1 equiv.). Subsequently, the mixture was further stirred at room temperature for 1-12 hours. After the completion of the reaction, the solvent was removed by evaporation, the resulting reaction mixture was extracted with ethyl acetate (40 mL), water (20 mL × 2), washed with brine (20 mL), and the combined organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and the solvent was evaporated under reduced pressure. The crude product was purified via column chromatography (eluting with petroleum ether/ethyl acetate) to afford the substituted olefin substrates **1**.

## 2.2. Synthesis of $\alpha$ -oxocarboxylic acid

The  $\alpha$ -oxocarboxylic acids were prepared from oxidation of corresponding methyl ketones by SeO<sub>2</sub> according to the reported procedure.<sup>2</sup>

## 2.3. Synthesis of Katritzky salts

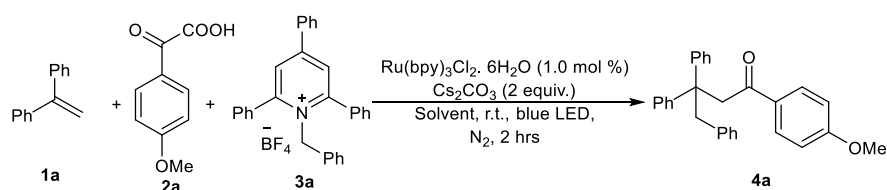
The  $\alpha$ -oxocarboxylic acids were prepared from according to the reported procedure.<sup>3</sup>

## 3. Optimization details

### 3.1. Procedure for optimization of the carbobenylation reaction

1-benzyl-2,4,6-triphenylpyridin-1-ium tetrafluoroborate **3a** (0.4 mmol, 2.0 equiv., 194 mg), 2-(4-methoxyphenyl)-2-oxoacetic acid **2a** (0.4 mmol, 2.0 equiv., 72 mg), photocatalyst and base were taken in a 7 mL screw-capped vial. Solvent was added to the mixture. The whole mixture was de-gassed and re-filled with inert gas by two consecutive freeze-pump-thaw cycles followed by the addition of 1,1-diphenylethylene **1a** (0.2 mmol, 1.0 equiv., 35  $\mu$ L). The reaction mixture was then stirred under 5W blue LED irradiation for 2 hours. After that, the solvent was evaporated under reduced pressure and the reaction mixture was extracted with ethyl acetate (30 mL), water (10 mL  $\times$  2), washed with brine (10 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography (SiO<sub>2</sub>, eluting with hexane/ethyl acetate) to afford the desired product.

**Table S1. Screening of solvents for carbobenylation**

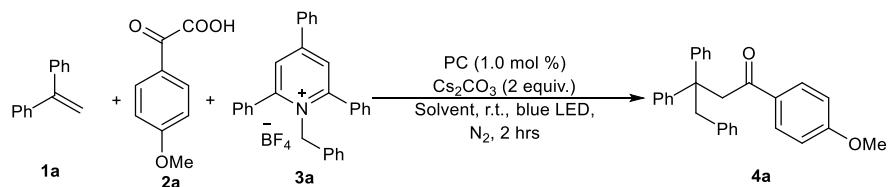


Entry	Solvent <sup>a</sup>	Yield of <b>4a</b> (%)
<b>1</b>	MeCN	<b>82</b>
2	THF	46
3	DMF	trace
5	DCE	66

6	DMSO	23
7	PhCH <sub>3</sub>	9

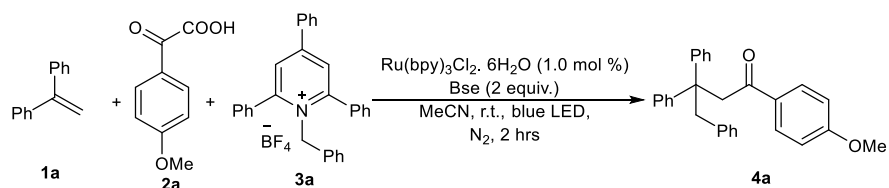
<sup>a</sup>All solvents are anhydrous.

**Table S2. Screening of photocatalysts for carbobenzylation**



Entry	Photocatalyst	Yield of <b>4a</b> (%)
<b>1</b>	<b>Ru(bpy)<sub>3</sub>Cl<sub>2</sub>·6H<sub>2</sub>O</b>	<b>82</b>
2	<i>fac</i> -Ir(ppy) <sub>3</sub>	12
5	Eosin Y	trace
6	Ir(ppy) <sub>2</sub> (dtbpy)PF <sub>6</sub>	66
7	Ir[dF(CF <sub>3</sub> )(ppy)] <sub>2</sub> (dtbpy)PF <sub>6</sub>	74
8	4-CzIPN	43
9	Ru(bpy) <sub>3</sub> (PF <sub>6</sub> ) <sub>2</sub>	76

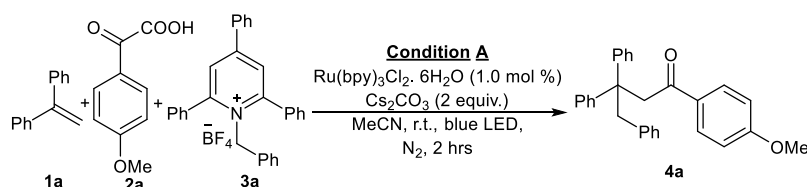
**Table S3. Screening of bases for carbobenzylation**



Entry	Base	Yield of <b>4a</b> (%)
<b>1</b>	<b>Cs<sub>2</sub>CO<sub>3</sub></b>	<b>82</b>
2	CsF	71
3	CsOAc	77
4	NaOAc	41

5	K <sub>2</sub> CO <sub>3</sub>	31
6	LiOH	18

**Table S4. Control experiments for carbobenzylation**



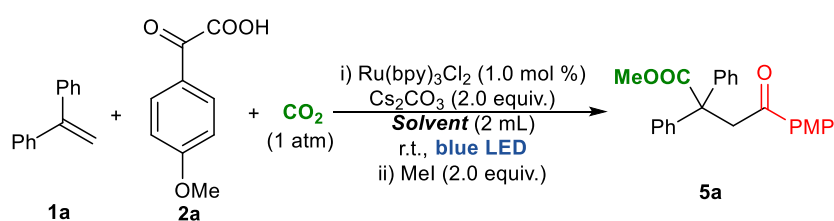
Entry	Variation from “Condition A”	Yield of <b>4a</b> (%)
<b>1</b>	<b>no variation</b>	<b>82</b>
2	no PC	0
3	no light	0
4	Ir(ppy) <sub>2</sub> (dtbpy)PF <sub>6</sub> instead of Ru(bpy) <sub>3</sub> Cl <sub>2</sub> · 6H <sub>2</sub> O	66
5	4CzIPN instead of Ru(bpy) <sub>3</sub> Cl <sub>2</sub> · 6H <sub>2</sub> O	43
6	Ir(ppy) <sub>3</sub> instead of Ru(bpy) <sub>3</sub> Cl <sub>2</sub> · 6H <sub>2</sub> O	12
7	without Cs <sub>2</sub> CO <sub>3</sub>	0
8	K <sub>2</sub> CO <sub>3</sub> instead of Cs <sub>2</sub> CO <sub>3</sub>	31
9	CsF instead of Cs <sub>2</sub> CO <sub>3</sub>	71
10	Addition of 20 mol % Cu(OTf) <sub>2</sub>	9
11	Addition of 10 mol % In(OTf) <sub>2</sub>	66

### 3.2. Procedure for optimization of the carbocarboxylation reaction

The oven-dried Schlenk tube (10 mL) containing a stirring bar was charged with **2a** (43.2 mg, 0.24 mmol, 1.2 equiv), photocatalyst and transferred to glovebox to add base. The tube was then evacuated and back-filled with CO<sub>2</sub> for 3 times. Therefore, under continuous CO<sub>2</sub> flow, solvent and **1a** (35 μL, 1.0 equiv., 0.2 mmol) were added with syringe and subsequently

the tube was sealed. The reaction was stirred with irradiating with a 30 W blue LED lamp (3 cm away, with cooling fan to keep the reaction temperature at 25~30 °C) for 12 hours. After 12 hours, the light was switched off, the Schlenk tube was opened and to the mixture, methyl iodide (24.9  $\mu$ L, 0.4 mmol, 2.0 equiv.) was added. The mixture was stirred for additional 4 hours at room temperature. After that, the reaction mixture was extracted with ethyl acetate (30 mL), water (10 mL  $\times$  2), washed with brine (10 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography (SiO<sub>2</sub>, eluting with hexane/ethyl acetate 93:7) to afford the desired product **5a**.

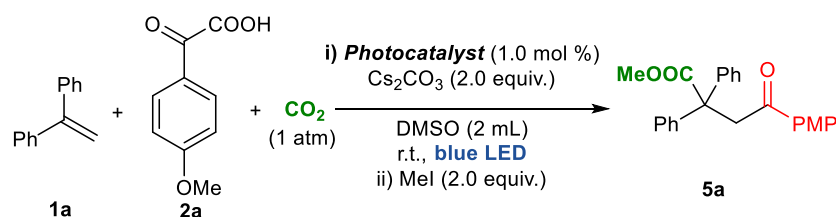
**Table S5. Screening of solvents for carbocarboxylation**



Entry	Solvent <sup>a</sup>	Yield of <b>5a</b> (%)
1	MeCN	16
2	THF	25
3	DMF	31
4	DMA	28
5	DCE	trace
<b>6</b>	<b>DMSO</b>	<b>37</b>
7	PhCH <sub>3</sub>	trace
8	HFIP	10
9	NMP	trace

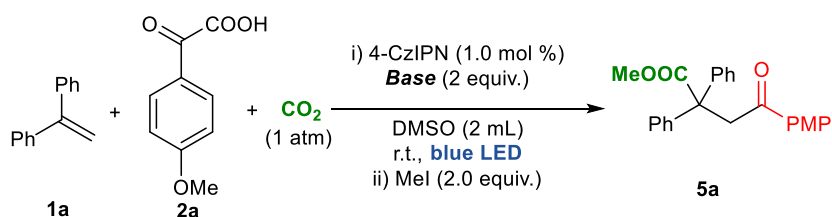
<sup>a</sup>All solvents are anhydrous.

**Table S6. Screening of photocatalysts for carbocarboxylation**



Entry	Photocatalyst	Yield of <b>5a</b> (%)
1	Ru(bpy) <sub>3</sub> Cl <sub>2</sub> ·6H <sub>2</sub> O	37
2	<i>fac</i> -Ir(ppy) <sub>3</sub>	43
3	<i>p</i> -terphenyl	ND
4	Xanthone	ND
5	Rose bengal	trace
6	Ir(ppy) <sub>2</sub> (dtbpy)PF <sub>6</sub>	70
7	Ir[dF(CF <sub>3</sub> )(ppy)] <sub>2</sub> (dtbpy)PF <sub>6</sub>	67
<b>8</b>	<b>4-CzIPN</b>	<b>81</b>
9	Ru(bpy) <sub>3</sub> (PF <sub>6</sub> ) <sub>2</sub>	32
10	4-DPAIPN	44

**Table S7. Screening of bases for carbocarboxylation**



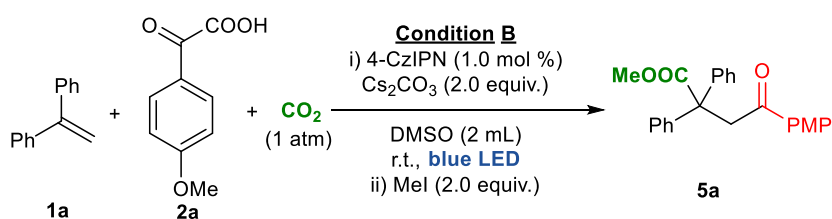
Entry	Base	Yield of <b>5a</b> (%)
<b>1</b>	<b>Cs<sub>2</sub>CO<sub>3</sub></b>	<b>81</b>
2	CsF	51
3	K <sub>2</sub> CO <sub>3</sub>	67
4	KO <sup>t</sup> Bu	41



5	Na <sub>2</sub> CO <sub>3</sub>	33
6	Li <sub>2</sub> CO <sub>3</sub>	30
7	LiCl	74
8	Et <sub>3</sub> N	trace
9	<sup>i</sup> Pr <sub>2</sub> NEt	trace
10	CsOAc	35
11 <sup>a</sup>	Cs <sub>2</sub> CO <sub>3</sub>	78

<sup>a</sup>3 equiv. of Cs<sub>2</sub>CO<sub>3</sub>.

**Table S8. Control experiments for carbocarboxylation**



Entry	Variation from “Condition B”	Yield of <b>5a</b> (%)
<b>1</b>	<b>None</b>	<b>81</b>
2	Without Cs <sub>2</sub> CO <sub>3</sub>	trace
3	Without 4-CzIPN	nd
4	Without blue LEDs	nd
5	Without 4-CzIPN, without blue LEDs	nd
6	Ar instead of CO <sub>2</sub>	30
7	O <sub>2</sub> instead of CO <sub>2</sub>	nd
8	Ar instead of CO <sub>2</sub> and 6 equiv. of <b>2a</b>	58
9	3 equiv. of <b>2a</b>	75

#### 4. General experimental procedures

##### 4.1. General procedure for carbobenzoylation (Table 2).

The Katritzky salt **3** (0.4 mmol, 2.0 equiv.),  $\alpha$ -oxocarboxylic acid **2** (0.4 mmol, 2.0 equiv.), tris(2,2'-bipyridyl)dichlororuthenium(II) hexahydrate (0.002 mmol, 1 mol %, 2.9 mg) and caesium carbonate (0.4 mmol, 2.0 equiv., 130 mg) were taken in a 7 mL screw-capped vial. 2 mL distilled acetonitrile solvent was added to the mixture. The whole mixture was de-gassed and re-filled with inert gas by two consecutive freeze-pump-thaw cycles followed by the addition of alkene **1** (0.2 mmol, 1.0 equiv.). The reaction mixture was then stirred under 5W blue LED irradiation for 2-6 hours. After completion, the solvent was evaporated under reduced pressure and the reaction mixture was extracted with ethyl acetate (30 mL), water (10 mL  $\times$  2), washed with brine (10 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography (SiO<sub>2</sub>, eluting with hexane/ethylacetate) to afford the desired product.

#### **4.2. General procedure for carbocarboxylation under blue LED irradiation (Table 3).**

The oven-dried Schlenk tube (10 mL) containing a stirring bar was charged with **1** (0.2 mmol, 1.0 equiv, if solid), 4-CzIPN (1.5 mg, 0.002 mmol, 1.0 mol %), **2** (0.24 mmol, 1.2 equiv) and transferred to glovebox to add Cs<sub>2</sub>CO<sub>3</sub> (130.3 mg, 0.4 mmol, 2.0 equiv). The tube was then evacuated and back-filled with CO<sub>2</sub> for 3 times. Therefore, under continuous CO<sub>2</sub> flow, anhydrous DMSO (3.0 mL) and **1** (if liquid) were added with syringe and subsequently the tube was sealed. The reaction was stirred with irradiating with a 30 W blue LED lamp (3 cm away, with cooling fan to keep the reaction temperature at 25~30 °C) for 12 hours. After 12 hours, the light was switched off, the schlenk tube was opened and to the mixture, methyl iodide (24.9  $\mu$ L, 0.4 mmol, 2.0 equiv.) was added. The mixture was stirred for additional 4 hours at room temperature. After that, the reaction mixture was extracted with ethyl acetate (30 mL), water (10 mL  $\times$  2), washed with brine (10 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography (SiO<sub>2</sub>, eluting with hexane/ethylacetate) to afford the desired product.

#### **4.3. General procedure for carbocarboxylation under sunlight irradiation (Table 3).**

The oven-dried Schlenk tube (10 mL) containing a stirring bar was charged with **1** (0.2 mmol, 1.0 equiv, if solid), 4-CzIPN (1.5 mg, 0.002 mmol, 1.0 mol %), **2** (0.24 mmol, 1.2 equiv) and transferred to glovebox to add Cs<sub>2</sub>CO<sub>3</sub> (130.3 mg, 0.4 mmol, 2.0 equiv). The tube was then evacuated and back-filled with CO<sub>2</sub> for 3 times. Therefore, under continuous CO<sub>2</sub> flow,

anhydrous DMSO (3.0 mL) and **1** (if liquid) were added with syringe and subsequently the tube was sealed. The reaction was stirred under direct sunlight (at Kolkata, 22.57° N, 88.36° E from 1100 hrs in December) for 5 hours. After 5 hours, the reaction was replaced from sunlight to indoor environment, the shlenk tube was opened and to the mixture, methyl iodide (24.9  $\mu$ L, 0.4 mmol, 2.0 equiv.) was added. The mixture was stirred for additional 4 hours at room temperature. After that, the reaction mixture was extracted with ethyl acetate (30 mL), water (10 mL  $\times$  2), washed with brine (10 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography (SiO<sub>2</sub>, eluting with hexane/ethylacetate) to afford the desired product.

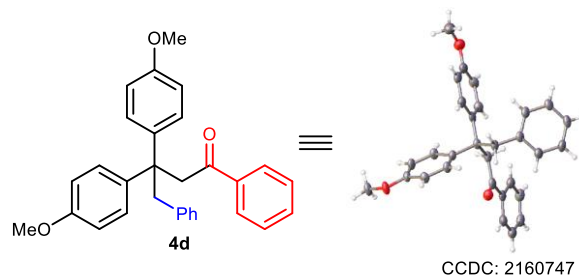
#### 4.4. General procedure for carbobenylation scale-up reaction under sunlight (Scheme 2).

4,4'-(ethene-1,1-diyl)bis(methoxybenzene) (3.16 mmol, 1 equiv), 1-benzyl-2,4,6-triphenylpyridin-1-ium tetrafluoroborate **2a** (6.32 mmol, 2.0 equiv., 3.06 g), 2-phenyl-2-oxoacetic acid **3a** (6.32 mmol, 2.0 equiv., 948 mg), tris(2,2'-bipyridyl)dichlororuthenium(II) hexahydrate (0.0316 mmol, 1 mol %, 23.6 mg) and cesium carbonate (6.32 mmol, 2.0 equiv., 2.05 g) were taken in a 50 mL round-bottomed flask connected to a nitrogen balloon *via* an adapter. 20 mL distilled acetonitrile solvent was added to the mixture. The whole mixture was de-gassed and re-filled with the inert gas by two consecutive freeze-pump-thaw cycles. Then the adapter was closed and balloon was removed. The reaction mixture was then stirred under direct sunlight for 2 hours. After that, the solvent was evaporated under reduced pressure and the reaction mixture was extracted with ethyl acetate (40 mL), water (15 mL  $\times$  2), washed with brine (15 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography (SiO<sub>2</sub>, eluting with hexane/ethylacetate) to afford the desired product.

## 5. Crystal data

### 5.1. Crystal data of **4d**.

The crystal of compound **4d** were grown in acetone-hexane solvent system by slow evaporation procedure. The crystal data was collected in X-ray spectroscopy (Bruker D8 Venture with a Photon-III detector instrument), and the data was analyzed using OLEX2 software. The structure is given below. The corresponding cif file is uploaded separately as supporting information.



Thermal ellipsoid of **4d**. Ellipsoids are represented with 50% probability.

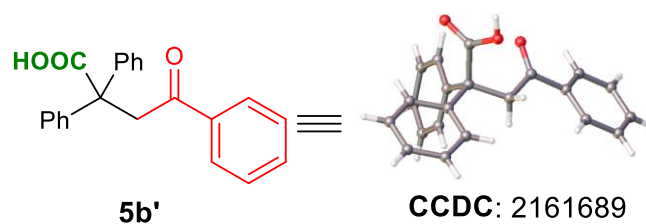
**Table S9. Crystal data and structure refinement for 4d.**

Identification code	<b>4d</b>
Empirical formula	C <sub>30</sub> H <sub>28</sub> O <sub>3</sub>
Formula weight	436.52
Temperature/K	100.0
Crystal system	triclinic
Space group	P-1
a/Å	9.7371(9)
b/Å	10.1536(10)
c/Å	12.7652(12)
α/°	102.012(3)
β/°	94.177(2)
γ/°	110.290(2)
Volume/Å <sup>3</sup>	1143.27(19)
Z	2
ρ <sub>calc</sub> /cm <sup>3</sup>	1.268
μ/mm <sup>-1</sup>	0.635
F(000)	464.0
Crystal size/mm <sup>3</sup>	0.45 × 0.34 × 0.15

Radiation	CuK $\alpha$ ( $\lambda = 1.54178$ )
2 $\Theta$ range for data collection/ $^{\circ}$	7.17 to 129.932
Index ranges	$-11 \leq h \leq 11$ , $-11 \leq k \leq 11$ , $-14 \leq l \leq 14$
Reflections collected	28980
Independent reflections	3779 [ $R_{\text{int}} = 0.0657$ , $R_{\text{sigma}} = 0.0442$ ]
Data/restraints/parameters	3779/0/300
Goodness-of-fit on $F^2$	1.045
Final R indexes [ $I \geq 2\sigma(I)$ ]	$R_1 = 0.0676$ , $wR_2 = 0.1901$
Final R indexes [all data]	$R_1 = 0.0689$ , $wR_2 = 0.1918$
Largest diff. peak/hole / $e \text{ \AA}^{-3}$	0.61/-0.29

## 5.2. Crystal data of **5b'**.

The crystal of compound **5b'** were grown in chloroform-hexane solvent system by slow evaporation procedure. The crystal data was collected in X-ray spectroscopy (Bruker D8 Venture with a Photon-III detector instrument), and the data was analyzed using OLEX2 software. The structure is given below. The corresponding cif file is uploaded separately as supporting information.



Thermal ellipsoid of **5b'**. Ellipsoids are represented with 50% probability.

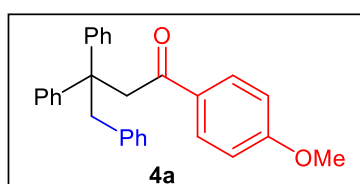
**Table S10. Crystal data and structure refinement for **5b'**.**

Identification code	<b>5b'</b>
Empirical formula	$C_{44}H_{36}O_6$
Formula weight	660.73
Temperature/K	298.0
Crystal system	triclinic

Space group	P-1
a/Å	8.9980(2)
b/Å	11.5521(2)
c/Å	18.5351(4)
$\alpha$ /°	79.7640(10)
$\beta$ /°	84.2930(10)
$\gamma$ /°	67.4670(10)
Volume/Å <sup>3</sup>	1750.20(6)
Z	2
$\rho_{\text{calc}}$ /cm <sup>3</sup>	1.254
$\mu$ /mm <sup>-1</sup>	0.663
F(000)	696.0
Crystal size/mm <sup>3</sup>	0.2 × 0.2 × 0.2
Radiation	CuK $\alpha$ ( $\lambda$ = 1.54178)
2 $\Theta$ range for data collection/°	4.848 to 136.656
Index ranges	-10 ≤ h ≤ 10, -13 ≤ k ≤ 13, -22 ≤ l ≤ 22
Reflections collected	59503
Independent reflections	6396 [ $R_{\text{int}}$ = 0.0782, $R_{\text{sigma}}$ = 0.0395]
Data/restraints/parameters	6396/0/453
Goodness-of-fit on F <sup>2</sup>	1.098
Final R indexes [ $I \geq 2\sigma(I)$ ]	$R_1$ = 0.0684, $wR_2$ = 0.1755
Final R indexes [all data]	$R_1$ = 0.0788, $wR_2$ = 0.1871
Largest diff. peak/hole / e Å <sup>-3</sup>	0.27/-0.38

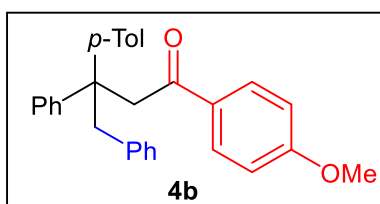
## 6. Spectral data

### 1-(4-methoxyphenyl)-3,3,4-triphenylbutan-1-one (4a)



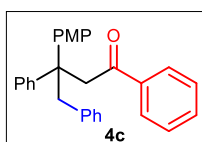
Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (71.4 mg, 82%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.75 (d, *J* = 9.2 Hz, 2H), 7.22-7.06 (m, 11H), 7.01 (t, *J* = 8.0 Hz, 2H), 6.80 (d, *J* = 8.8 Hz, 2H), 6.61-6.59 (m, 2H), 3.86 (s, 2H), 3.81 (s, 3H), 3.62 (s, 2H); δ <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 197.4, 163.2, 148.0, 138.1, 131.4, 130.9, 130.2, 128.2, 127.8, 127.5, 126.1, 126.0, 113.4, 55.5, 49.7, 43.8, 42; HRMS (ESI, *m/z*) calcd. For C<sub>29</sub>H<sub>26</sub>O<sub>2</sub>Na [M+Na]<sup>+</sup>: 429.1830; found: 429.1830.

### 1-(4-methoxyphenyl)-3,4-diphenyl-3-(*p*-tolyl)butan-1-one (4b)



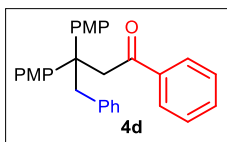
Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (48.7 mg, 58%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.76 (d, *J* = 8.8 Hz, 2H), 7.22-6.99 (m, 12H), 6.80 (d, *J* = 8.8 Hz, 2H), 6.60 (d, *J* = 7.2 Hz, 2H), 3.88-3.84 (m, 2H), 3.81 (s, 3H), 3.61 (s, 2H), 2.28 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 197.4, 163.1, 148.3, 145.1, 138.3, 135.3, 131.5, 130.9, 130.2, 128.6, 128.1, 128.0, 127.8, 127.5, 126.1, 125.9, 113.4, 55.5, 49.4, 43.8, 42.9, 21.0; HRMS (ESI, *m/z*) calcd. For C<sub>30</sub>H<sub>28</sub>O<sub>2</sub>Na [M+Na]<sup>+</sup>: 443.1987; found: 443.2007.

### 3-(4-methoxyphenyl)-1,3,4-triphenylbutan-1-one (4c)



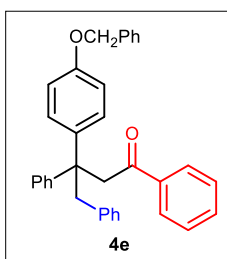
Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (58.4 mg, 72%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.74 (d, *J* = 7.6 Hz, 2H), 7.45 (t, *J* = 7.6 Hz, 1H), 7.33 (t, *J* = 8.0 Hz, 2H), 7.23-7.07 (m, 8H), 7.03 (t, *J* = 7.6 Hz, 2H), 6.75 (d, *J* = 8.8 Hz, 2H), 6.61 (d, *J* = 7.2 Hz, 2H), 3.83 (s, 2H), 3.76 (s, 3H), 3.64 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 199.1, 157.7, 148.1, 140.0, 138.5, 138.1, 132.6, 130.9, 129.2, 128.3, 128.1, 127.9, 127.8, 127.5, 126.1, 126.0, 113.2, 55.3, 49.1, 43.9, 43.6 HRMS (ESI, *m/z*) calcd. For C<sub>29</sub>H<sub>26</sub>O<sub>2</sub>Na [M+Na]<sup>+</sup>: 429.1830; found: 429.1837.

### 3,3-bis(4-methoxyphenyl)-1,4-diphenylbutan-1-one (4d)



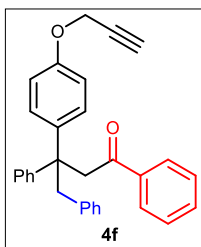
Column chromatography (SiO<sub>2</sub>, eluting with 95:5 hexane/ethyl acetate) afforded the desired product as a white solid (61.0 mg, 70%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.74 (d, *J* = 7.2 Hz, 2H), 7.45 (t, *J* = 7.2 Hz, 1H), 7.33 (t, *J* = 9.2 Hz, 2H), 7.10-7.02 (m, 7H), 6.75 (d, *J* = 8.8 Hz, 4H), 6.63 (d, *J* = 7.2 Hz, 2H), 3.79 (s, 2H), 3.76 (s, 3H), 3.60 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 199.3, 157.6, 140.2, 138.5, 138.3, 132.6, 131.0, 129.1, 128.3, 127.8, 127.5, 126.1, 113.2, 55.2, 48.6, 44.1, 43.7 HRMS (ESI, *m/z*) calcd. For C<sub>30</sub>H<sub>28</sub>O<sub>3</sub>Na [M+Na]<sup>+</sup>: 459.1936; found: 459.1952.

### 3-(4-(benzyloxy)phenyl)-1,3,4-triphenylbutan-1-one (4e)



Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (75.2 mg, 78%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.75 (d, *J* = 8.4 Hz, 2H), 7.48-7.32 (m, 8H), 7.22-7.08 (m, 8H), 7.05-7.02 (m, 2H), 6.83 (d, *J* = 8.8 Hz, 2H), 6.62 (d, *J* = 7.2 Hz, 2H), 5.02 (s, 2H), 3.84 (s, 2H), 3.65 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 199.1, 157.0, 148.1, 140.3, 138.5, 138.1, 137.2, 132.6, 130.9, 129.2, 128.6, 128.4, 128.1, 128.0, 127.9, 127.8, 127.6, 127.5, 126.2, 126.1, 114.2, 70.1, 49.2, 43.9, 43.5.

### 1,3,4-triphenyl-3-(4-(prop-2-yn-1-yloxy)phenyl)butan-1-one (4f)

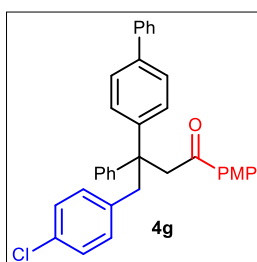


Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (56.7 mg, 66%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.74 (d, *J* = 8.8 Hz, 2H), 7.23-7.08 (m, 8H), 7.04-7.00 (m, 2H), 6.82 (dd, *J*<sub>1</sub> = 8.8 Hz, *J*<sub>2</sub> = 6 Hz, 4H), 6.62 (d, *J* =



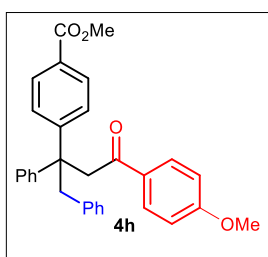
7.2 Hz, 2H), 4.63 (d,  $J = 2.4$  Hz, 2H), 3.82 (d,  $J = 1.6$  Hz, 2H), 3.81 (s, 3H), 3.59 (s, 2H), 2.50 (t,  $J = 2.4$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  197.5, 163.2, 155.7, 148.1, 141.1, 138.2, 131.5, 131.0, 130.2, 129.2, 128.1, 127.8, 127.5, 126.1, 126.0, 114.2, 113.5, 78.8, 75.5, 55.9, 55.5, 49.3, 43.9, 42.9; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{32}\text{H}_{28}\text{O}_3\text{Na}$   $[\text{M}+\text{Na}]^+$ : 483.1936; found: 483.1948.

**3-([1,1'-biphenyl]-4-yl)-4-(4-chlorophenyl)-1-(4-methoxyphenyl)-3-phenylbutan-1-one (4g)**



Column chromatography ( $\text{SiO}_2$ , eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (72.2 mg, 70%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.74 (d,  $J = 8.8$  Hz, 2H), 7.77 (d,  $J = 8.4$  Hz, 2H), 7.46-7.39 (m, 4H), 7.32 (t,  $J = 7.2$  Hz, 2H), 7.26-7.15 (m, 7H), 7.00 (d,  $J = 8.8$  Hz, 2H), 6.79 (d,  $J = 8.8$  Hz, 2H), 6.59 (d,  $J = 8.8$  Hz, 2H), 3.91-3.80 (m, 2H), 3.79 (s, 3H), 3.66-3.56 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  197.6, 163.3, 147.5, 146.8, 140.7, 138.8, 136.6, 132.3, 132.1, 131.4, 130.2, 128.8, 128.6, 128.2, 128.0, 127.7, 127.3, 127.0, 126.6, 126.3, 113.5, 55.5, 49.7, 43.1, 42.7; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{35}\text{H}_{30}\text{O}_2\text{Cl}$   $[\text{M}+\text{H}]^+$ : 517.1934; found: 517.1945.

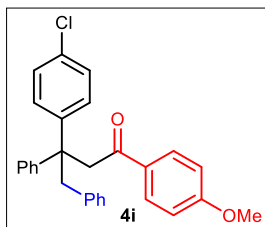
**methyl 4-(4-oxo-1,2,4-triphenylbutan-2-yl)benzoate (4h)**



Column chromatography ( $\text{SiO}_2$ , eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (31.2 mg, 36%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.88 (d,  $J = 8.8$  Hz, 2H), 7.74 (d,  $J = 9.2$  Hz, 2H), 7.26 (d,  $J = 8.8$  Hz, 2H), 7.23-7.06 (m, 6H), 7.00 (t,  $J = 7.2$  Hz, 2H), 6.81 (d,  $J = 9.2$  Hz, 2H), 6.55 (d,  $J = 8.4$  Hz, 2H), 3.91-3.86 (m, 5H), 3.81 (s, 3H), 3.65 (s, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  196.8, 167.1, 163.3, 153.7, 147.3, 137.6, 131.2, 130.8,

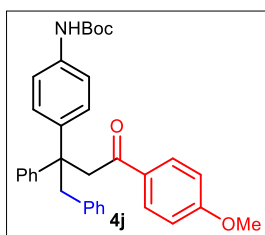
130.1, 129.2, 128.2, 128.1, 128.0, 127.8, 127.6, 126.3, 126.3, 113.6, 55.5, 52.1, 49.9, 43.7, 42.8; HRMS (ESI, m/z) calcd. For C<sub>31</sub>H<sub>29</sub>O<sub>4</sub> [M+H]<sup>+</sup>: 465.2066; found: 465.2067.

### 3-(4-chlorophenyl)-1-(4-methoxyphenyl)-3,4-diphenylbutan-1-one (4i)



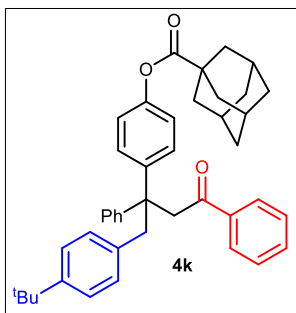
Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (54.5 mg, 62%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.76 (d, *J* = 9.2 Hz, 2H), 7.24-7.08 (m, 10H), 7.03 (t, *J* = 7.2 Hz, 2H), 6.82 (d, *J* = 9.2 Hz, 2H), 6.59 (d, *J* = 7.2 Hz, 2H), 3.88-3.78 (m, 5H), 3.60 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 197.0, 163.3, 147.5, 146.7, 137.7, 131.8, 131.3, 130.9, 130.2, 129.6, 128.1, 128.0, 127.6, 126.3, 113.6, 55.5, 49.4, 43.8, 42.8; HRMS (ESI, m/z) calcd. For C<sub>29</sub>H<sub>26</sub>O<sub>2</sub>Cl [M+H]<sup>+</sup>: 441.1621; found: 441.1626.

### tert-butyl (4-(4-(4-methoxyphenyl)-4-oxo-1,2-diphenylbutan-2-yl)phenyl)carbamate (4j)



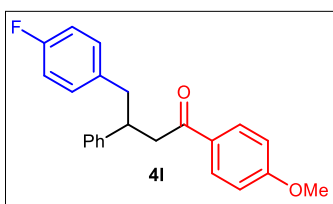
Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (50.0 mg, 48%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.74 (d, *J* = 8.8 Hz, 2H), 7.21-6.99 (m, 3H), 6.80 (d, *J* = 9.2 Hz, 2H), 6.61 (d, *J* = 6.8 Hz, 2H), 6.47 (br. s, 1H), 3.83-3.81 (m, 5H), 3.58 (s, 2H), 1.49 (s, 9H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 197.4, 163.2, 152.8, 148.0, 142.7, 138.1, 136.3, 131.4, 131.0, 130.1, 128.7, 128.1, 127.8, 127.5, 126.1, 126.0, 117.9, 113.5, 55.4, 49.3, 43.8, 42.9, 29.7, 28.4; HRMS (ESI, m/z) calcd. For C<sub>34</sub>H<sub>35</sub>O<sub>4</sub>NaN [M+Na]<sup>+</sup>: 544.2464; found: 544.2470.

### 4-(1-(4-(tert-butyl)phenyl)-4-oxo-2,4-diphenylbutan-2-yl)phenyl (1s,3s)-adamantane-1-carboxylate (4k)



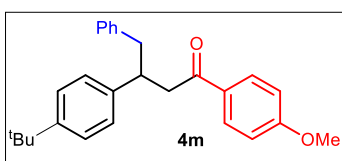
Column chromatography (SiO<sub>2</sub>, eluting with 95:5 hexane/ethyl acetate) afforded the desired product as a white solid (81.7 mg, 67%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.75 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 7.2 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.21-7.16 (m, 7H), 7.05 (d, *J* = 8.4 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 6.52 (d, *J* = 8.4 Hz, 2H), 3.89-3.78 (m, 2H), 3.73-3.63 (m, 4H), 2.08-2.04 (m, 9H), 1.77 (s, 6H), 1.24 (s, 9H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 198.7, 176.2, 149.2, 148.9, 147.8, 145.3, 138.4, 134.6, 132.6, 130.5, 129.1, 128.4, 128.2, 127.9, 127.8, 126.1, 124.5, 120.8, 49.4, 43.7, 43.5, 41.1, 38.8, 36.6, 34.4, 31.4, 28.0.

#### 4-(4-fluorophenyl)-1-(4-methoxyphenyl)-3-phenylbutan-1-one (4l)



Column chromatography (SiO<sub>2</sub>, eluting with 95:5 hexane/ethyl acetate) afforded the desired product as a white solid (36.2 mg, 52%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.85 (d, *J* = 9.2 Hz, 2H), 7.24-7.21 (m, 2H), 7.16-7.12 (m, 3H), 6.98-6.95 (m, 2H), 6.89-6.83 (m, 4H), 3.84 (s, 3H), 3.64-3.56 (m, 1H), 3.31-3.19 (m, 2H), 3.03-2.97 (m, 1H), 2.89-2.84 (m, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 197.4, 163.5, 161.4 (d, *J* = 241.8 Hz), 143.9, 135.6 (d, *J* = 3.3 Hz), 130.6 (d, *J* = 7.8 Hz), 130.3, 130.3, 128.4, 127.7, 126.5, 114.9 (d, *J* = 21.0 Hz), 113.7, 55.5, 43.9, 43.3, 42.1; HRMS (ESI, *m/z*) calcd. For C<sub>23</sub>H<sub>22</sub>O<sub>2</sub>F [M+H]<sup>+</sup>: 349.1604; found: 349.1602.

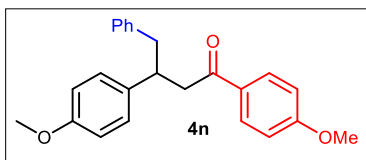
#### 3-(4-(tert-butyl)phenyl)-1-(4-methoxyphenyl)-4-phenylbutan-1-one (4m)



Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (44.0 mg, 57%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.81 (d, *J* = 8.8 Hz,

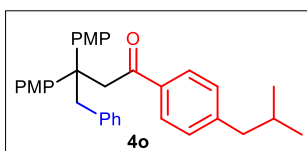
2H), 7.26-7.08 (m, 9H), 6.86 (d,  $J = 9.2$  Hz, 2H), 3.83 (s, 3H), 3.63-3.59 (m, 1H), 3.28-3.15 (s, 2H), 2.96 (d,  $J = 7.2$  Hz, 2H), 1.27 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  197.7, 163.4, 149.1, 141.3, 140.2, 130.4, 130.3, 129.4, 128.2, 127.3, 126.1, 125.3, 113.7, 55.5, 43.8, 43.0, 42.6, 34.4, 31.5; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{27}\text{H}_{31}\text{O}_2$   $[\text{M}+\text{H}]^+$ : 387.2324; found: 387.2328.

### 1,3-bis(4-methoxyphenyl)-4-phenylbutan-1-one (4n)



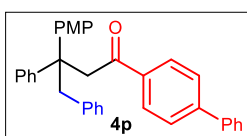
Column chromatography ( $\text{SiO}_2$ , eluting with 95:5 hexane/ethyl acetate) afforded the desired product as a white solid (38.1 mg, 53%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.82 (d,  $J = 9.2$  Hz, 2H), 7.22-7.17 (m, 2H), 7.15-7.11 (m, 1H), 7.09-7.05 (m, 4H), 6.87 (d,  $J = 9.2$  Hz, 2H), 6.76 (d,  $J = 8.8$  Hz, 2H), 3.83 (s, 3H), 3.74 (s, 3H), 3.63-3.56 (s, 1H), 3.26-3.15 (m, 2H), 3.00-2.87 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  197.7, 163.4, 158.1, 140.1, 136.3, 130.4, 129.4, 128.6, 128.2, 126.1, 113.8, 113.7, 55.5, 55.2, 44.1, 43.2, 42.5; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{24}\text{H}_{25}\text{O}_3$   $[\text{M}+\text{H}]^+$ : 361.1804; found: 361.1818.

### 1-(4-isobutylphenyl)-3,3-bis(4-methoxyphenyl)-4-phenylbutan-1-one (4o)



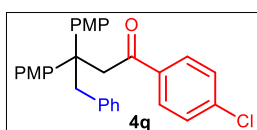
Column chromatography ( $\text{SiO}_2$ , eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (71.8 mg, 73%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.67 (d,  $J = 8.0$  Hz, 2H), 7.11-7.02 (m, 9H), 6.75 (d,  $J = 8.8$  Hz, 4H), 6.65 (d,  $J = 6.8$  Hz, 2H), 3.80 (s, 2H), 3.76 (s, 6H), 3.59 (s, 2H), 2.48 (d,  $J = 7.2$  Hz, 2H), 1.86 (m, 1H), 0.89 (s, 3H), 0.88 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  198.9, 157.6, 146.9, 140.3, 138.3, 136.3, 131.1, 129.1, 129.0, 127.9, 127.5, 126.1, 113.1, 55.2, 48.6, 45.4, 44.1, 43.5, 30.2, 22.4; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{34}\text{H}_{36}\text{O}_3\text{Na}$   $[\text{M}+\text{Na}]^+$ : 515.2562; found: 515.2566

### 1-([1,1'-biphenyl]-4-yl)-3-(4-methoxyphenyl)-3,4-diphenylbutan-1-one (4p)



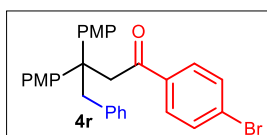
Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (65.5 mg, 68%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.84 (d, *J* = 8.4 Hz, 2H), 7.60-7.56 (m, 4H), 7.47-7.44 (m, 2H), 7.39 (t, *J* = 7.2 Hz, 1H), 7.26-7.11 (s, 3H), 7.06 (d, *J* = 8.0 Hz, 2H), 6.77 (d, *J* = 8.8 Hz, 2H), 6.66 (d, *J* = 7.2 Hz, 2H), 3.91-3.83 (m, 2H), 3.76 (s, 3H), 3.69 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 198.7, 157.7, 148.2, 145.3, 140.1, 140.0, 138.2, 137.1, 131.0, 129.2, 129.0, 128.5, 128.3, 128.2, 127.9, 127.6, 127.3, 127.0, 126.2, 126.1, 113.3, 55.3, 49.2, 44.0, 43.6; HRMS (ESI, *m/z*) calcd. For C<sub>35</sub>H<sub>30</sub>O<sub>2</sub>Na [M+Na]<sup>+</sup>: 505.2143; found: 505.2153

#### 1-(4-chlorophenyl)-3,3-bis(4-methoxyphenyl)-4-phenylbutan-1-one (4q)



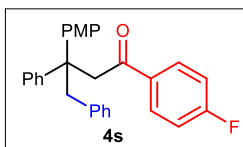
Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (58.3 mg, 62%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.64 (d, *J* = 8.8 Hz, 2H), 7.28 (d, *J* = 8.8 Hz, 2H), 7.11-7.02 (m, 7H), 6.73 (d, *J* = 8.8 Hz, 4H), 6.62 (d, *J* = 6.8 Hz, 2H), 3.74-3.74 (m, 8H), 3.53 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 198.3, 157.7, 139.9, 138.9, 138.1, 136.8, 131.0, 129.3, 129.1, 128.6, 127.5, 126.2, 113.2, 55.3, 48.8, 44.0, 43.7; HRMS (ESI, *m/z*) calcd. For C<sub>30</sub>H<sub>27</sub>O<sub>3</sub>NaCl [M+Na]<sup>+</sup>: 493.1546; found: 493.1543.

#### 1-(4-bromophenyl)-3,3-bis(4-methoxyphenyl)-4-phenylbutan-1-one (4r)



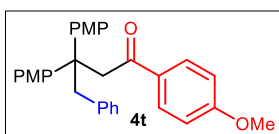
Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (47.3 mg, 46%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.56 (d, *J* = 8.4 Hz, 2H), 7.44 (d, *J* = 8.8 Hz, 2H), 7.09 (t, *J* = 7.2 Hz, 1H), 7.05-7.02 (m, 6H), 6.73 (d, *J* = 8.8 Hz, 4H), 6.62-6.61 (m, 2H), 3.75 (s, 6H), 3.74 (s, 2H), 3.52 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 198.5, 157.7, 139.9, 138.1, 137.2, 131.5, 131.0, 129.4, 129.1, 127.6, 127.5, 126.2, 113.2, 55.3, 48.7, 43.9, 43.7.

#### 1-(4-fluorophenyl)-3-(4-methoxyphenyl)-3,4-diphenylbutan-1-one (4s)



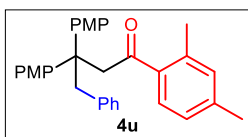
Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (56.8 mg, 67%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.77-7.74 (m, 2H), 7.23-6.96 (m, 12H), 6.75 (d, *J* = 9.2 Hz, 2H), 6.63 (d, *J* = 7.2 Hz, 2H), 3.81 (s, 2H), 3.76 (s, 3H), 3.59 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 197.7, 165.4 (d, *J* = 252.9 Hz), 157.8, 147.9, 139.8, 138.0, 134.8 (d, *J* = 2.7 Hz), 131.0, 130.5 (d, *J* = 9.0 Hz), 129.2, 128.1, 127.9, 127.5, 126.2, 126.1, 115.3 (d, *J* = 21.5 Hz), 113.2, 55.3, 49.3, 43.9, 43.4; HRMS (ESI, *m/z*) calcd. For C<sub>29</sub>H<sub>25</sub>O<sub>2</sub>NaF [M+Na]<sup>+</sup>: 447.1736; found: 447.1732.

### 1,3,3-tris(4-methoxyphenyl)-4-phenylbutan-1-one (4t)



Column chromatography (SiO<sub>2</sub>, eluting with 95:5 hexane/ethyl acetate) afforded the desired product as a white solid (69.9 mg, 75%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.74 (d, *J* = 8.8 Hz, 2H), 7.10-7.00 (m, 7H), 6.79 (d, *J* = 8.8 Hz, 2H), 6.73 (d, *J* = 8.8 Hz, 2H), 6.62 (d, *J* = 8.0 Hz, 2H), 3.81 (s, 3H), 3.78 (s, 2H), 3.75 (s, 6H), 3.53 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 197.6, 163.1, 157.6, 140.4, 138.4, 131.5, 131.0, 130.2, 129.1, 127.5, 126.0, 113.4, 113.1, 55.5, 55.3, 48.6, 44.1, 43.1; HRMS (ESI, *m/z*) calcd. For C<sub>31</sub>H<sub>30</sub>O<sub>4</sub>Na [M+Na]<sup>+</sup>: 489.2042; found: 489.2040.

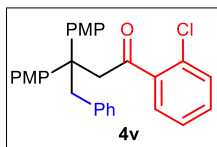
### 1-(2,4-dimethylphenyl)-3,3-bis(4-methoxyphenyl)-4-phenylbutan-1-one (4u)



Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (64.9 mg, 70%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.20 (d, *J* = 8.4 Hz, 2H), 7.13-7.03 (m, 8H), 6.93-6.91 (m, 2H), 6.75 (d, *J* = 9.2 Hz, 2H), 6.68 (d, *J* = 6.8 Hz, 2H), 3.83 (s, 2H), 3.77 (s, 6H), 3.50 (s, 2H), 2.28 (s, 3H), 2.15 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 203.7, 157.7, 140.8, 140.3, 138.4, 137.8, 137.2, 132.3, 131.1, 129.1, 127.5, 126.1, 125.9,

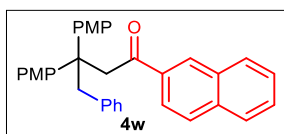
113.2, 55.3, 48.8, 46.7, 44.1, 21.3, 20.6; HRMS (ESI, m/z) calcd. For  $C_{32}H_{32}O_3Na$   $[M+Na]^+$ : 487.2249; found: 487.2251.

### 1-(2-chlorophenyl)-3,3-bis(4-methoxyphenyl)-4-phenylbutan-1-one (4v)



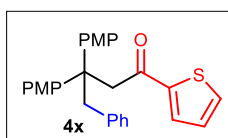
Column chromatography ( $SiO_2$ , eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (62.0 mg, 66%).  $^1H$  NMR (400 MHz,  $CDCl_3$ ):  $\delta$  7.48 (dd,  $J_1 = 4.8$  Hz,  $J_2 = 1.2$  Hz, 1H), 7.42 (dd,  $J_1 = 4.0$  Hz,  $J_2 = 1.2$  Hz, 1H), 7.10-6.94 (m, 8H), 6.95 (dd,  $J_1 = 4.8$  Hz,  $J_2 = 3.6$  Hz, 1H), 6.74 (d,  $J = 8.8$  Hz, 4H), 6.70 (d,  $J = 8.0$  Hz, 2H), 3.76-3.75 (m, 8H), 3.49 (s, 2H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ ):  $\delta$  203.3, 157.8, 141.4, 139.8, 138.0, 131.2, 130.9, 129.9, 129.9, 129.1, 128.2, 127.5, 126.6, 126.2, 113.2, 55.3, 49.3, 48.6, 43.9; HRMS (ESI, m/z) calcd. For  $C_{30}H_{27}O_3NaCl$   $[M+Na]^+$ : 493.1546; found: 493.1548.

### 3,3-bis(4-methoxyphenyl)-1-(naphthalen-2-yl)-4-phenylbutan-1-one (4w)



Column chromatography ( $SiO_2$ , eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (65.1 mg, 67%).  $^1H$  NMR (400 MHz,  $CDCl_3$ ):  $\delta$  8.21 (s, 1H), 7.87-7.76 (m, 4H), 7.57-7.48 (m, 2H), 7.13-7.03 (m, 7H), 6.75 (d,  $J = 8.8$  Hz, 4H), 6.69 (d,  $J = 7.2$  Hz, 2H), 3.84 (s, 2H), 3.74 (s, 2H), 3.72 (s, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ ):  $\delta$  199.3, 157.7, 140.2, 138.3, 135.8, 135.3, 132.4, 131.1, 129.6, 129.4, 129.2, 128.3, 128.1, 127.7, 127.5, 126.7, 126.1, 123.8, 113.2, 55.2, 48.8, 44.1, 43.8; HRMS (ESI, m/z) calcd. For  $C_{34}H_{30}O_3Na$   $[M+Na]^+$ : 509.2093; found: 509.2111.

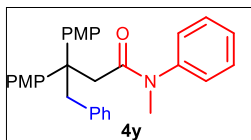
### 3,3-bis(4-methoxyphenyl)-4-phenyl-1-(thiophen-2-yl)butan-1-one (4x)



Column chromatography ( $SiO_2$ , eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (57.4 mg, 65%).  $^1H$  NMR (400 MHz,  $CDCl_3$ ):  $\delta$  7.27-7.19 (m, 1H), 7.15-7.04 (m, 4H), 7.00 (d,  $J = 8.8$  Hz, 4H), 6.73-6.68 (m, 7H), 3.76 (s, 6H), 3.74 (s, 2H), 3.58

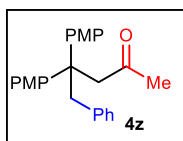
(s, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  192.2, 157.7, 145.8, 139.8, 138.1, 133.4, 131.8, 131.2, 129.2, 127.8, 127.5, 126.1, 113.2, 55.3, 49.1, 44.7, 44.1; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{28}\text{H}_{26}\text{O}_3\text{NaS}$   $[\text{M}+\text{Na}]^+$ : 465.1500; found: 465.1510.

### 3,3-bis(4-methoxyphenyl)-N-methyl-N,4-diphenylbutanamide (4y)



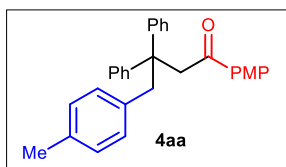
Column chromatography ( $\text{SiO}_2$ , eluting with 90:10 hexane/ethyl acetate) afforded the desired product as a white solid (55.8 mg, 60%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.30 (t,  $J = 5.2$  Hz, 2H), 7.25 (t,  $J = 4.8$  Hz, 1H), 7.13-7.07 (m, 3H), 6.85-6.81 (m, 6H), 6.76-6.73 (m, 6H), 3.82-3.80 (m, 8H), 3.06 (s, 3H), 2.72 (s, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  170.6, 157.0, 143.5, 139.7, 138.0, 130.8, 128.9, 128.7, 127.0, 126.9, 126.8, 125.3, 112.4, 54.7, 48.7, 43.5, 38.5, 36.7; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{31}\text{H}_{32}\text{O}_3\text{N}$   $[\text{M}+\text{H}]^+$ : 466.2382; found: 466.2384.

### 4,4-bis(4-methoxyphenyl)-5-phenylpentan-2-one (4z)



Column chromatography ( $\text{SiO}_2$ , eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (41.8 mg, 56%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.11-7.02 (m, 3H), 6.99 (d,  $J = 8.8$  Hz, 4H), 6.77 (d,  $J = 8.8$  Hz, 4H), 6.66 (dd,  $J_1 = 8.0$  Hz,  $J_2 = 1.2$  Hz, 2H), 3.78 (s, 6H), 3.58 (s, 2H), 2.99 (s, 2H), 1.7 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  208.8, 157.8, 139.8, 138.0, 131.2, 129.1, 127.4, 126.0, 113.2, 55.3, 48.9, 48.7, 43.5, 32.8.

### 1-(4-methoxyphenyl)-3,3-diphenyl-4-(p-tolyl)butan-1-one (4aa)

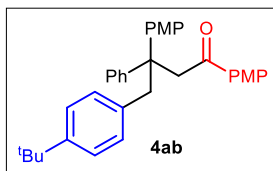


Column chromatography ( $\text{SiO}_2$ , eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (52.1 mg, 62%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.76 (d,  $J = 9.2$  Hz, 2H), 7.23-7.06 (m, 10H), 6.84-6.79 (m, 4H), 6.48 (d,  $J = 9.2$  Hz, 2H), 3.83-3.81 (m, 5H), 3.63



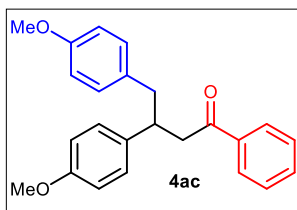
(s, 2H), 2.23 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  197.3, 163.1, 148.2, 135.5, 134.9, 131.5, 130.8, 130.2, 128.2, 128.2, 127.8, 125.9, 113.5, 55.5, 49.7, 43.4, 42.9, 21.1.

#### 4-(4-(tert-butyl)phenyl)-1,3-bis(4-methoxyphenyl)-3-phenylbutan-1-one (4ab)



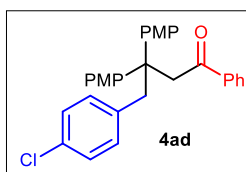
Column chromatography ( $\text{SiO}_2$ , eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (52 mg, 53%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.45 (d,  $J = 8.8$  Hz, 2H), 7.23-7.13 (m, 5H), 7.09 (d,  $J = 9.2$  Hz, 2H), 7.03 (d,  $J = 8.4$  Hz, 2H), 6.80 (d,  $J = 8.8$  Hz, 2H), 6.75 (d,  $J = 9.2$  Hz, 2H), 6.53 (d,  $J = 8.4$  Hz, 2H), 3.81-3.79 (m, 5H), 3.76 (s, 3H), 3.60 (s, 2H), 1.23 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  197.5, 163.1, 157.6, 148.8, 148.4, 140.4, 135.0, 131.6, 130.6, 130.2, 129.2, 128.2, 127.8, 125.9, 124.4, 113.4, 113.1, 55.4, 55.2, 49.1, 43.5, 43.1, 34.3, 31.4; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{34}\text{H}_{36}\text{O}_3\text{Na}$   $[\text{M}+\text{Na}]^+$ : 515.2562; found: 515.2561.

#### 3,4-bis(4-methoxyphenyl)-1-phenylbutan-1-one (4ac)



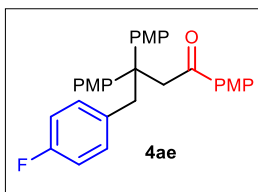
Column chromatography ( $\text{SiO}_2$ , eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (41.0 mg, 57%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.82 (d,  $J = 7.2$  Hz, 2H), 7.50 (t,  $J = 7.2$  Hz, 1H), 7.40 (d,  $J = 7.2$  Hz, 2H), 7.06 (d,  $J = 8.4$  Hz, 2H), 6.95 (d,  $J = 8.8$  Hz, 2H), 6.77-6.72 (m, 4H), 3.74 (s, 6H), 3.59-3.52 (m, 1H), 3.29-3.18 (m, 2H), 2.92-2.82 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  199.2, 158.1, 157.9, 137.3, 136.3, 132.9, 132.1, 130.3, 128.6, 128.5, 128.1, 113.8, 55.3, 44.4, 42.5, 42.3.

#### 4-(4-chlorophenyl)-3,3-bis(4-methoxyphenyl)-1-phenylbutan-1-one (4ad)



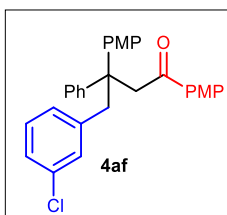
Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (52.6 mg, 56%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.71 (dd, *J*<sub>1</sub> = 8.4 Hz, *J*<sub>2</sub> = 1.2 Hz, 2H), 7.44 (t, *J* = 7.2 Hz, 1H), 7.32 (t, *J* = 7.2 Hz, 1H), 7.04 (d, *J* = 8.8 Hz, 2H), 7.00 (d, *J* = 8.4 Hz, 2H), 6.73 (t, *J* = 8.8 Hz, 4H), 6.55 (d, *J* = 8.4 Hz, 2H), 3.75 (s, 8H), 3.54 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 199.4, 157.7, 139.8, 138.3, 136.7, 132.7, 132.3, 132.0, 129.1, 128.4, 127.8, 127.6, 113.2, 55.3, 48.6, 43.4, 43.3.

#### 4-(4-fluorophenyl)-1,3,3-tris(4-methoxyphenyl)butan-1-one (4ae)



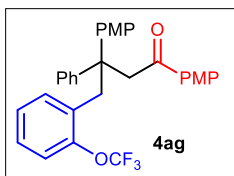
Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (77.4 mg, 80%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.72 (d, *J* = 9.2 Hz, 2H), 7.04 (d, *J* = 9.2 Hz, 4H), 6.79 (d, *J* = 8.8 Hz, 2H), 6.74-6.69 (m, 6H), 6.61-6.57 (m, 2H), 3.80 (s, 3H), 3.75 (s, 8H), 3.49 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 197.8, 163.2, 161.6 (d, *J* = 242.5 Hz), 157.7, 140.1, 133.9 (d, *J* = 2.8 Hz), 132.3 (d, *J* = 7.6 Hz), 131.5, 130.2, 129.1, 114.2 (d, *J* = 20.6 Hz), 113.4, 113.2, 55.5, 55.2, 48.7, 43.2, 42.9; HRMS (ESI, *m/z*) calcd. For C<sub>31</sub>H<sub>29</sub>O<sub>4</sub>NaF [M+Na]<sup>+</sup>: 507.1948; found: 507.1953.

#### 4-(3-chlorophenyl)-1,3-bis(4-methoxyphenyl)-3-phenylbutan-1-one (4af)



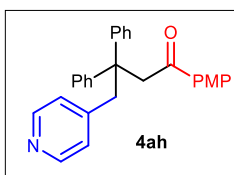
Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (45.1 mg, 48%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.69 (dd, *J*<sub>1</sub> = 8.4 Hz, *J*<sub>2</sub> = 1.2 Hz, 2H), 7.43 (td, *J*<sub>1</sub> = 7.2 Hz, *J*<sub>2</sub> = 1.2 Hz, 1H), 7.31 (t, *J* = 8.0 Hz, 2H), 7.08-7.03 (m, 5H), 6.96 (t, *J* = 7.8 Hz, 1H), 6.74 (d, *J* = 8.8 Hz, 2H), 6.57-6.53 (m, 2H), 3.75 (s, 6H), 3.73 (s, 2H), 3.54 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 199.6, 157.8, 140.4, 139.6, 138.5, 133.2, 132.6, 131.0, 129.1, 129.0, 128.6, 128.3, 127.8, 126.3, 113.3, 55.3, 48.7, 43.7, 43.4.

#### 1,3-bis(4-methoxyphenyl)-3-phenyl-4-(2-(trifluoromethoxy)phenyl)butan-1-one (4ag)



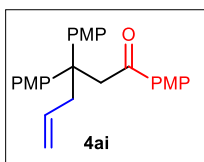
Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (42.6 mg, 41%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.79 (dd, *J*<sub>1</sub> = 8.0 Hz, *J*<sub>2</sub> = 1.2 Hz, 2H), 7.46 (td, *J*<sub>1</sub> = 7.2 Hz, *J*<sub>2</sub> = 1.2 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.14-7.01 (m, 6H), 6.93-6.88 (m, 1H), 6.73 (d, *J* = 9.2 Hz, 2H), 6.54 (dd, *J*<sub>1</sub> = 8.0 Hz, *J*<sub>2</sub> = 1.6 Hz, 1H), 3.84 (s, 2H), 3.75 (s, 6H), 3.67 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 197.6, 157.7, 148.6, 139.7, 138.2, 133.5, 132.5, 130.6, 129.1, 128.3, 127.8, 127.6, 125.6, 121.6, 120.3 (q, *J* = 256.0 Hz), 119.6, 119.0, 113.1, 55.2, 48.4, 44.1, 38.4; HRMS (ESI, *m/z*) calcd. For C<sub>31</sub>H<sub>27</sub>O<sub>4</sub>NaF<sub>3</sub> [M+Na]<sup>+</sup>: 543.1759; found: 543.1761.

#### 1-(4-methoxyphenyl)-3,3-diphenyl-4-(pyridin-4-yl)butan-1-one (4ah)



Column chromatography (SiO<sub>2</sub>, eluting with 80:20 hexane/ethyl acetate) afforded the desired product as a white solid (25.2 mg, 31%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.26 (d, *J* = 3.2 Hz, 2H), 7.68 (d, *J* = 8.8 Hz, 2H), 7.23-7.18 (m, 4H), 7.16-7.11 (m, 6H), 6.77 (d, *J* = 8.8 Hz, 2H), 6.65 (d, *J* = 6.0 Hz, 2H), 3.87 (s, 2H), 3.80 (s, 3H), 3.51 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 197.6, 163.4, 148.2, 146.9, 131.1, 130.3, 128.2, 127.9, 126.9, 126.6, 113.5, 55.5, 49.8, 43.2, 42.5; HRMS (ESI, *m/z*) calcd. For C<sub>28</sub>H<sub>26</sub>O<sub>2</sub>N [M+H]<sup>+</sup>: 408.1964; found: 408.1965.

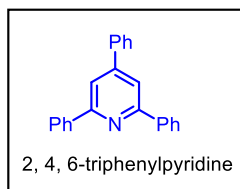
#### 1-(4-methoxyphenyl)-3,3-diphenylhex-5-en-1-one (4ai)



Column chromatography (SiO<sub>2</sub>, eluting with 96:4 hexane/ethyl acetate) afforded the desired product as a white solid (28.3 mg, 34%). <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>): δ 7.73 (d, *J* = 8.4 Hz, 2H), 7.13 (d, *J* = 9.0 Hz, 4H), 6.80 (d, *J* = 9.0 Hz, 2H), 6.77 (d, *J* = 9.0 Hz, 4H), 5.47-5.41 (m, 1H), 5.07-5.04 (m, 1H), 4.98 (dd, *J*<sub>1</sub> = 10.2 Hz, *J*<sub>2</sub> = 1.8 Hz, 1H), 3.82 (s, 3H), 3.76 (s, 6H), 3.69 (s, 2H), 3.20 (d, *J* = 7.2 Hz, 2H); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>): δ 196.7, 162.4, 157.0,

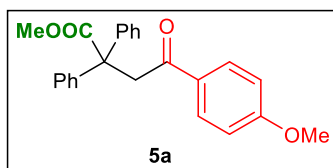
139.5, 134.5, 130.9, 129.6, 128.2, 117.9, 112.8, 112.6, 54.9, 54.7, 47.1, 43.8, 42.4; HRMS (ESI, m/z) calcd. For  $C_{27}H_{28}O_4Na$   $[M+Na]^+$ : 439.1885; found: 439.1888.

### 2,4,6-triphenylpyridine (byproduct formed from carbobenzylation reaction)



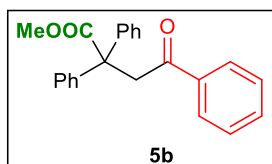
This was recovered in quantitative amount from each of the carbobenzylation reaction and purified in column chromatography ( $SiO_2$ , eluting with hexane) affording as white solid (61.5 mg, quant.).  $^1H$  NMR (400 MHz,  $CDCl_3$ ):  $\delta$  8.23-8.20 (m, 4H), 7.90 (s, 2H), 7.77-7.74 (m, 2H), 7.56-7.43 (m, 9H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ ):  $\delta$  195.02, 173.90, 163.66, 143.36, 130.37, 130.00, 128.81, 128.00, 126.87, 113.80, 57.14, 55.58, 52.51, 48.04; HRMS (ESI, m/z) calcd. For  $C_{23}H_{17}N$   $[M]^+$ : 307.1361; found: 307.1359.

### 4-(4-methoxyphenyl)-4-oxo-2,2-diphenylbutanoic acid (5a)



This was prepared following the general procedure and purified in column chromatography ( $SiO_2$ , eluting with 90:10 hexane/ethyl acetate) affording as white solid (61.3 mg, 82%).  $^1H$  NMR (400 MHz,  $CDCl_3$ ):  $\delta$  7.92 (d,  $J = 7$  Hz, 2H), 7.32-7.19 (m, 10H), 6.89 (d,  $J = 8.8$  Hz, 2H), 4.15 (s, 2H), 3.84 (s, 3H), 3.74 (s, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ ):  $\delta$  195.02, 173.90, 163.66, 143.36, 130.37, 130.00, 128.81, 128.00, 126.87, 113.80, 57.14, 55.58, 52.51, 48.04; HRMS (ESI, m/z) calcd. For  $C_{24}H_{22}O_4$   $[M]^+$ : 374.1518; found: 374.1515.

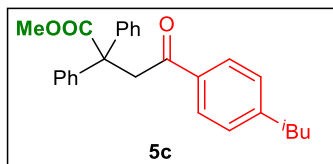
### methyl 4-oxo-2,2,4-triphenylbutanoate (5b)



This was prepared following the general procedure and purified in column chromatography ( $SiO_2$ , eluting with 90:10 hexane/ethyl acetate) affording as white solid (51.6 mg, 72%).  $^1H$  NMR (400 MHz,  $CDCl_3$ ):  $\delta$  7.95-7.92 (m, 2H), 7.54 (t,  $J = 7.2$  Hz, 1H), 7.43 (t,  $J = 8$  Hz, 2H),

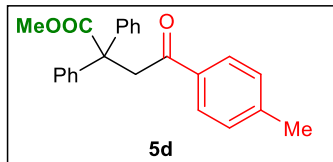
7.32-7.29 (m, 4H), 7.27-7.18 (m, 6H), 4.20 (s, 2H), 3.74 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  196.5, 173.7, 143.2, 136.9, 133.3, 128.8, 128.6, 128.0, 128.0, 126.9, 51.1, 52.5, 48.3; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{23}\text{H}_{21}\text{O}_3$   $[\text{M}+\text{H}]^+$ : 345.1491; found: 345.1494.

**methyl 4-(4-isobutylphenyl)-4-oxo-2,2-diphenylbutanoate (5c)**



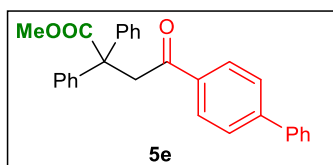
This was prepared following the general procedure and purified in column chromatography ( $\text{SiO}_2$ , eluting with 90:10 hexane/ethyl acetate) affording as yellowish white solid (64.0 mg, 80%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.85 (d,  $J = 8$  Hz, 2H), 7.32-7.25 (m, 7H), 7.23-7.18 (m, 5H), 4.18 (s, 1H), 3.73 (s, 3H), 2.51 (d,  $J = 7.6$  Hz, 2H), 1.87 (sept.,  $J = 6.8$  Hz, 1H), 0.90 (s, 3H), 0.88 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  196.14, 173.85, 147.85, 143.30, 134.68, 129.38, 128.81, 128.06, 128.00, 126.88, 57.07, 52.51, 48.30, 45.46, 30.20, 22.39; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{27}\text{H}_{29}\text{O}_4$   $[\text{M}+\text{H}]^+$ : 401.2117; found: 401.2112.

**methyl 4-oxo-2,2-diphenyl-4-(p-tolyl)butanoate (5d)**



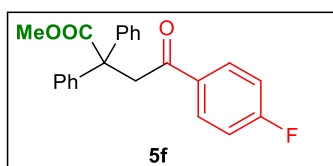
This was prepared following the general procedure and purified in column chromatography ( $\text{SiO}_2$ , eluting with 90:10 hexane/ethyl acetate) affording as white solid (55.8 mg, 78%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$   $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.83 (d,  $J = 8$  Hz, 2H), 7.31 – 7.25 (m, 7H), 7.23 – 7.17 (m, 5H), 4.17 (s, 2H), 3.74 (s, 3H), 2.39 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  196.10, 173.84, 144.13, 143.29, 134.45, 129.33, 128.80, 128.20, 128.00, 126.89, 57.11, 52.52, 48.26, 21.72; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{24}\text{H}_{23}\text{O}_3$   $[\text{M}+\text{H}]^+$ : 359.1647; found: 359.1648.

**methyl 4-([1,1'-biphenyl]-4-yl)-4-oxo-2,2-diphenylbutanoate (5e)**



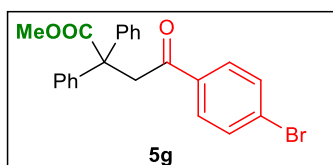
This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as white solid (51.2 mg, 61%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.02-8.00 (m, 2H), 7.66-7.64 (m, 2H), 7.61-7.59 (m, 2H), 7.48-7.44 (m, 2H), 7.41-7.39 (m, 1H), 7.34-7.26 (m, 8H), 7.23-7.19 (m, 2H), 4.23 (s, 2H), 3.75 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 196.08, 173.80, 146.02, 143.22, 139.90, 135.60, 129.05, 128.81, 128.68, 128.36, 128.04, 127.35, 127.32, 126.95, 57.16, 52.56, 48.39; HRMS (ESI, m/z) calcd. For C<sub>29</sub>H<sub>25</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 421.1804; found: 421.1799.

#### methyl 4-(4-fluorophenyl)-4-oxo-2,2-diphenylbutanoate (5f)



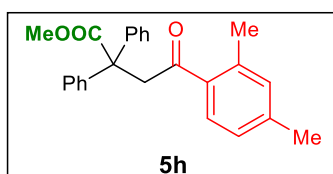
This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as brown solid (49.2 mg, 68%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.97-7.93 (m, 2H), 7.30-7.25 (m, 7H), 7.24-7.18 (m, 3H), 7.09 (t, *J* = 8.4 Hz, 2H), 4.15 (s, 2H), 3.74 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 194.98, 173.70, 165.88 (d, *J* = 254 Hz), 143.07, 133.36 (d, *J* = 3 Hz), 130.72 (d, *J* = 9 Hz), 128.76, 128.05, 126.99, 115.76 (d, *J* = 22 Hz), 57.19, 52.58, 48.21; HRMS (ESI, m/z) calcd. For C<sub>24</sub>H<sub>23</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 359.1647; found: 359.1647.

#### methyl 4-(4-bromophenyl)-4-oxo-2,2-diphenylbutanoate (5g)



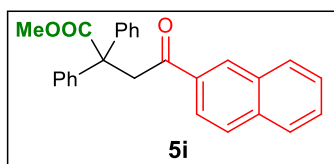
This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as yellow solid (41.4 mg, 49%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.57 (d, *J* = 4.8 Hz, 2H), 7.36-7.34 (m, 3H), 7.28-7.22 (m, 3H), 7.16-7.12 (m, 3H), 7.02 (s, 1H), 6.65 (d, *J* = 8 Hz, 2H), 3.70 (s, 2H), 3.67 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 192.72, 173.79, 154.19, 142.85, 142.44, 141.50, 139.17, 136.33, 131.05, 129.85, 129.25, 128.63, 128.49, 128.40, 128.07, 128.02, 127.85, 127.12, 124.28, 62.04, 52.39, 44.52; HRMS (ESI, m/z) calcd. For C<sub>23</sub>H<sub>20</sub>BrO<sub>3</sub> [M+H]<sup>+</sup>: 423.0596; found: 423.0602.

#### methyl 4-(2,4-dimethylphenyl)-4-oxo-2,2-diphenylbutanoate (5h)



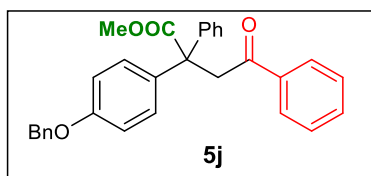
This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as white solid (54.3 mg, 73%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.49 (d, *J* = 8 Hz, 2H), 7.32-7.26 (m, 6H), 7.25-7.18 (m, 4H), 7.03-7.02 (m, 2H), 4.11 (s, 2H), 3.76 (s, 3H), 2.34 (s, 3H), 2.32 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 200.08, 173.88, 143.29, 141.99, 138.52, 135.22, 132.87, 128.81, 128.51, 128.01, 126.89, 126.30, 57.26, 52.48, 51.06, 21.43, 21.16; HRMS (ESI, *m/z*) calcd. For C<sub>25</sub>H<sub>25</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 373.1804; found: 373.1800.

#### methyl 4-(naphthalen-2-yl)-4-oxo-2,2-diphenylbutanoate (5i)



This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as yellowish white solid (51.2 mg, 65%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.46 (s, 1H), 7.99 (dd, *J*<sub>1</sub> = 8.4 Hz, *J*<sub>2</sub> = 1.6 Hz, 1H), 7.94 (d, *J* = 7.2 Hz, 1H), 7.87-7.84 (m, 2H), 7.61-7.52 (m, 2H), 7.36-7.34 (m, 4H), 7.30-7.26 (m, 4H), 7.23-7.19 (m, 2H), 4.34 (s, 2H), 3.76 (m, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 196.48, 173.83, 143.24, 135.70, 134.24, 132.52, 129.68, 129.63, 128.84, 128.63, 128.55, 128.06, 127.86, 126.96, 126.93, 123.86, 57.28, 52.59, 48.44; HRMS (ESI, *m/z*) calcd. For C<sub>27</sub>H<sub>23</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 395.1647; found: 395.1645.

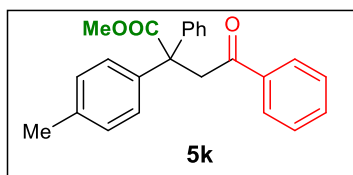
#### methyl 2-(4-(benzyloxy)phenyl)-4-oxo-2,4-diphenylbutanoate (5j)



This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as white solid (70.2 mg, 78%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.95-7.93 (m, 2H), 7.55 (t, *J* = 8 Hz, 1H), 7.46-7.26 (m, 12H), 7.23-7.21 (m, 2H), 6.89-6.86 (m, 2H), 5.02 (s, 2H), 4.19 (s, 2H), 3.74 (s, 3H); <sup>13</sup>C NMR (100

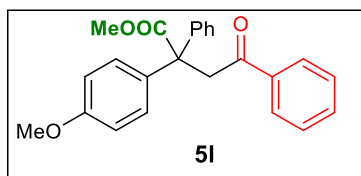
MHz, CDCl<sub>3</sub>):  $\delta$  196.59, 174.00, 157.63, 143.49, 137.07, 136.97, 135.52, 133.29, 130.04, 128.67, 128.07, 127.61, 126.93, 114.22, 70.08, 56.49, 52.54, 48.50; HRMS (ESI, m/z) calcd. For C<sub>30</sub>H<sub>27</sub>O<sub>4</sub> [M+H]<sup>+</sup>: 451.1909; found: 451.1911.

#### methyl 4-oxo-2,4-diphenyl-2-(p-tolyl)butanoate (5k)



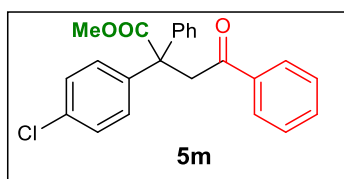
This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as white solid (58.0 mg, 81%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.95-7.93 (m, 2H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.43 (m, 2H), 7.33-7.30 (m, 2H), 7.27-7.25 (m, 1H), 7.24-7.17 (m, 4H), 4.19 (s, 2H), 3.73 (s, 3H), 2.29 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  196.55, 173.92, 143.36, 140.24, 136.97, 136.60, 133.25, 128.80, 128.76, 128.66, 128.60, 128.08, 127.98, 126.86, 56.78, 52.51, 48.41, 21.01; HRMS (ESI, m/z) calcd. For C<sub>24</sub>H<sub>23</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 359.1647; found: 359.1634.

#### methyl 2-(4-methoxyphenyl)-4-oxo-2,4-diphenylbutanoate (5l)



This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as yellow gel (57.6 mg, 77%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.95-7.92 (m, 2H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.45-7.41 (m, 2H), 7.34-7.31 (m, 2H), 7.28-7.26 (m, 2H), 7.24-7.18 (m, 3H), 6.81-6.77 (m, 2H), 4.18 (s, 2H), 3.76 (s, 3H), 3.73 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  196.58, 174.01, 158.32, 143.51, 136.97, 135.23, 133.27, 129.99, 128.68, 128.07, 128.04, 126.90, 113.33, 56.46, 55.27, 52.51, 48.49; HRMS (ESI, m/z) calcd. For C<sub>24</sub>H<sub>23</sub>O<sub>4</sub> [M+H]<sup>+</sup>: 375.1596; found: 375.1588.

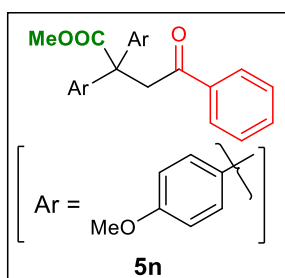
#### methyl 2-(4-chlorophenyl)-4-oxo-2,4-diphenylbutanoate (5m)





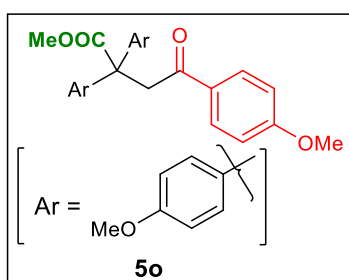
This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as white solid (55.2 mg, 73%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.94-7.91 (m, 2H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.30-7.25 (m, 5H), 7.24-7.19 (m, 4H), 4.17 (AB<sub>q</sub>, *J* = 49.6 Hz, 2H), 3.73 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 196.27, 173.45, 142.89, 141.61, 136.74, 133.45, 132.81, 130.62, 128.73, 128.38, 128.33, 128.07, 127.99, 127.28, 56.64, 52.66, 48.25; HRMS (ESI, *m/z*) calcd. For C<sub>23</sub>H<sub>20</sub>ClO<sub>3</sub> [M+H]<sup>+</sup>: 379.1101; found: 379.1094.

#### methyl 2,2-bis(4-methoxyphenyl)-4-oxo-4-phenylbutanoate (5n)



This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as white solid (50.1 mg, 62%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.94-7.91 (m, 2H), 7.53 (t, *J* = 7.8 Hz, 1H), 7.44-7.40 (m, 2H), 7.25-7.22 (m, 4H), 6.80-6.76 (m, 4H), 4.14 (s, 2H), 3.76 (s, 6H), 3.72 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 196.62, 174.19, 158.29, 137.02, 135.51, 133.22, 129.86, 128.65, 128.06, 113.32, 55.78, 55.28, 52.47, 48.59; HRMS (ESI, *m/z*) calcd. For C<sub>25</sub>H<sub>25</sub>O<sub>5</sub> [M+H]<sup>+</sup>: 405.1624; found: 405.1623.

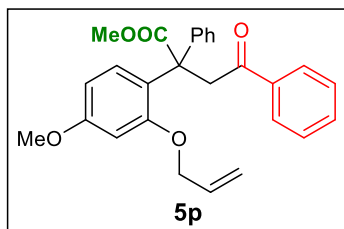
#### methyl 2,2,4-tris(4-methoxyphenyl)-4-oxobutanoate (5o)



This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as white solid (65.1 mg, 75%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.91 (d, *J* = 8.8 Hz, 2H), 7.22 (d, *J* = 8.8 Hz, 4H), 6.89 (d, *J* = 8.8 Hz, 2H), 6.78 (d, *J* = 8.8 Hz, 4H), 4.08 (s, 2H), 3.84 (s, 3H), 3.76 (s, 6H), 3.72 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 195.15, 174.32, 163.61, 158.23, 135.66, 130.34, 130.10, 129.87,

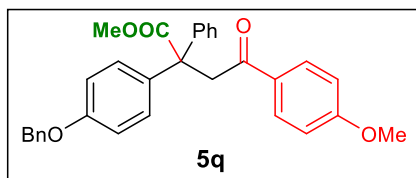
113.77, 113.29, 55.81, 55.57, 55.27, 52.44, 48.26; HRMS (ESI,  $m/z$ ) calcd. For  $C_{26}H_{27}O_6$   $[M+H]^+$ : 435.1808; found: 435.1820.

**methyl 2-(2-(allyloxy)-4-methoxyphenyl)-4-oxo-2,4-diphenylbutanoate (5p)**



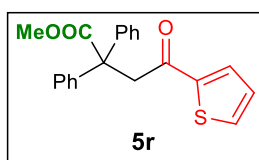
This was prepared following the general procedure and purified in column chromatography ( $SiO_2$ , eluting with 90:10 hexane/ethyl acetate) affording as yellowish white solid (58.5 mg, 68%).  $^1H$  NMR (400 MHz,  $CDCl_3$ ):  $\delta$  7.93-7.90 (m, 2H), 7.53-7.49 (m, 1H), 7.42-7.38 (m, 4H), 7.29-7.26 (m, 2H), 7.24-7.20 (m, 2H), 6.42 (dd,  $J_1 = 8.8$  Hz,  $J_2 = 2.4$  Hz, 1H), 6.35 (d,  $J = 2.4$  Hz, 1H), 5.85-5.75 (m, 1H), 5.28-5.17 (m, 2H), 4.38-4.31 (m, 4H), 3.74 (s, 3H), 3.64 (s, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ ):  $\delta$  197.28, 174.76, 159.79, 156.72, 140.26, 137.68, 132.97, 132.84, 132.00, 128.83, 128.48, 128.04, 127.81, 126.89, 124.00, 117.46, 103.80, 100.14, 69.29, 56.02, 55.31, 52.46, 42.90; HRMS (ESI,  $m/z$ ) calcd. For  $C_{26}H_{25}O_4$   $[M+H]^+$ : 401.1753; found: 401.1760.

**methyl 2-(4-(benzyloxy)phenyl)-4-(4-methoxyphenyl)-4-oxo-2-phenylbutanoate (5q)**



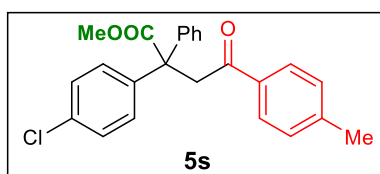
This was prepared following the general procedure and purified in column chromatography ( $SiO_2$ , eluting with 90:10 hexane/ethyl acetate) affording as yellow gum (82.6 mg, 86%).  $^1H$  NMR (400 MHz,  $CDCl_3$ ):  $\delta$  7.94-7.91 (m, 2H), 7.42-7.19 (m, 12H), 6.92-6.85 (m, 4H), 5.01 (s, 2H), 4.14-4.13 (m, 2H), 3.85 (s, 3H), 3.74 (s, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ ):  $\delta$  195.11, 174.12, 163.66, 157.59, 143.66, 137.10, 135.68, 130.38, 130.06, 130.05, 128.71, 128.67, 128.04, 127.61, 126.87, 114.18, 113.81, 70.07, 56.52, 55.59, 52.50, 48.17; HRMS (ESI,  $m/z$ ) calcd. For  $C_{31}H_{29}O_5$   $[M+H]^+$ : 481.2015; found: 481.2022.

**methyl 4-oxo-2,2-diphenyl-4-(thiophen-2-yl)butanoate (5r)**



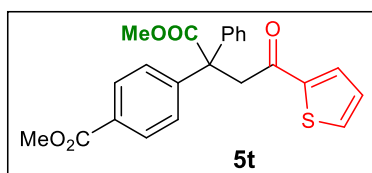
This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as yellow solid (58.1 mg, 83%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.68 (dd, *J*<sub>1</sub> = 3.6 Hz, *J*<sub>2</sub> = 1.2 Hz, 1H), 7.58 (dd, *J*<sub>1</sub> = 5.2 Hz, *J*<sub>2</sub> = 1.2 Hz, 1H), 7.33-7.26 (m, 7H), 7.25-7.19 (m, 3H), 7.08-7.06 (m, 1H), 4.13 (s, 2H), 3.74 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 189.46, 173.60, 144.08, 142.97, 133.72, 131.85, 128.80, 128.12, 128.06, 127.02, 57.26, 52.63, 48.63; HRMS (ESI, *m/z*) calcd. For C<sub>21</sub>H<sub>19</sub>O<sub>3</sub>S [M+H]<sup>+</sup>: 351.1055; found: 351.1055.

#### methyl 2-(4-chlorophenyl)-4-oxo-2-phenyl-4-(p-tolyl)butanoate (5s)



This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as colourless gum (51.7 mg, 66%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.86-7.83 (m, 2H), 7.53 (dd, *J*<sub>1</sub> = 5.6 Hz, *J*<sub>2</sub> = 2.0 Hz, 1H), 7.39-7.35 (m, 2H), 7.31-7.25 (m, 4H), 7.23-7.16 (m, 4H), 4.48 (AB<sub>q</sub>, *J* = 18.8 Hz, 2H), 3.70 (s, 3H), 2.39 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 196.09, 173.67, 144.03, 139.70, 134.69, 133.85, 133.12, 130.91, 129.31, 128.82, 128.51, 128.18, 128.10, 127.28, 125.92, 58.33, 52.86, 43.16, 21.70; HRMS (ESI, *m/z*) calcd. For C<sub>24</sub>H<sub>21</sub>ClO<sub>3</sub> [M+H]<sup>+</sup>: 393.1257; found: 393.1249.

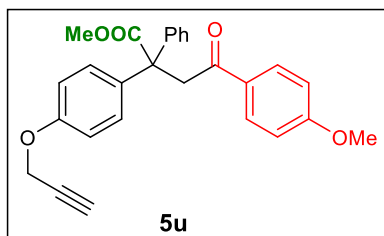
#### methyl 4-(1-methoxy-1,4-dioxo-2-phenyl-4-(thiophen-2-yl)butan-2-yl)benzoate (5t)



This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as white solid (58.8 mg, 72%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.57 (dd, *J*<sub>1</sub> = 4 Hz, *J*<sub>2</sub> = 1.2 Hz, 1H), 7.52 (dd, *J*<sub>1</sub> = 5.2 Hz, *J*<sub>2</sub> = 1.2 Hz, 1H), 7.39 (dd, *J*<sub>1</sub> = 4.8 Hz, *J*<sub>2</sub> = 0.8 Hz, 1H), 7.33-7.31 (m, 3H), 7.11 (dd, *J*<sub>1</sub> = 4 Hz, *J*<sub>2</sub> = 1.2 Hz, 1H), 7.01 (dd, *J*<sub>1</sub> = 5.2 Hz, *J*<sub>2</sub> = 4 Hz, 1H), 6.83-6.80 (m, 4H), 4.14 (s, 2H), 3.76

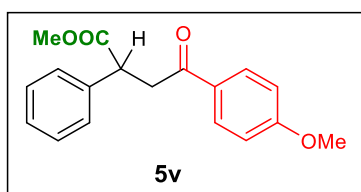
(s, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  192.71, 189.65, 158.60, 144.88, 143.24, 133.61, 133.48, 132.56, 131.68, 130.83, 129.87, 127.99, 127.32, 113.61, 61.95, 55.28, 50.25; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{23}\text{H}_{21}\text{O}_5\text{S}$   $[\text{M}+\text{H}]^+$ : 411.1266; found: 411.1269.

**methyl 4-(4-methoxyphenyl)-4-oxo-2-phenyl-2-(4-(prop-2-yn-1-yloxy)phenyl)butanoate (5u)**



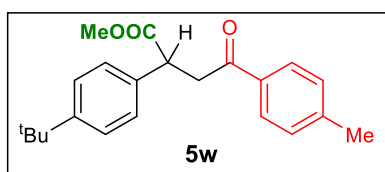
This was prepared following the general procedure and purified in column chromatography ( $\text{SiO}_2$ , eluting with 90:10 hexane/ethyl acetate) affording as colourless gum (58.3 mg, 68%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.92-7.90 (m, 2H), 7.31-7.27 (m, 3H), 7.24-7.19 (m, 4H), 6.91-6.83 (m, 4H), 4.62 (d,  $J = 7.2$  Hz, 2H), 4.11 (s, 2H), 3.85 (s, 3H), 3.73 (s, 3H), 2.49 (t,  $J = 2.4$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  195.03, 174.02, 163.65, 156.33, 143.54, 136.29, 130.35, 130.09, 130.01, 128.63, 128.04, 126.89, 114.16, 113.79, 78.67, 75.57, 56.51, 55.86, 55.57, 52.50, 48.13; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{27}\text{H}_{25}\text{O}_5$   $[\text{M}+\text{H}]^+$ : 429.1702; found: 429.1689.

**methyl 4-oxo-2,4-diphenylbutanoate (5v)**



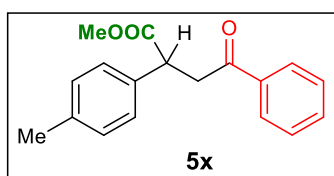
This was prepared following the general procedure and purified in column chromatography ( $\text{SiO}_2$ , eluting with 90:10 hexane/ethyl acetate) affording as brown solid (44.7 mg, 75%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.96-7.92 (m, 2H), 7.34-7.26 (m, 5H), 6.92-6.89 (m, 2H), 4.27 (dd,  $J_1 = 10.4$  Hz,  $J_2 = 4$  Hz, 1H), 3.95-3.88 (m, 1H), 3.85 (s, 3H), 3.21 (dd,  $J_1 = 17.6$  Hz,  $J_2 = 4$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  196.19, 174.05, 163.73, 138.59, 132.75, 130.45, 129.61, 128.98, 127.92, 127.59, 113.82, 55.56, 52.39, 46.51, 42.56; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{18}\text{H}_{19}\text{O}_4$   $[\text{M}+\text{H}]^+$ : 299.1283; found: 299.1287.

**methyl 2-(4-(tert-butyl)phenyl)-4-oxo-4-(p-tolyl)butanoate (5w)**



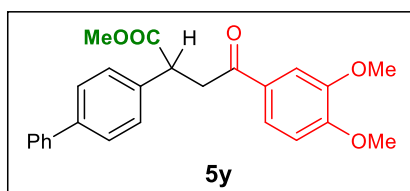
This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as colourless gum (49.3 mg, 73%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.87-7.85 (m, 2H), 7.36-7.34 (m, 2H), 7.29-7.26 (m, 2H), 7.23 (d, *J* = 8 Hz, 2H), 4.26 (dd, *J*<sub>1</sub> = 10.4 Hz, *J*<sub>2</sub> = 4 Hz, 1H), 3.91 (dd, *J*<sub>1</sub> = 18 Hz, *J*<sub>2</sub> = 10.4 Hz, 1H), 3.69 (s, 3H), 3.23 (dd, *J*<sub>1</sub> = 18 Hz, *J*<sub>2</sub> = 4 Hz, 1H), 2.39 (s, 3H), 1.31 (s, 9H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 197.48, 174.18, 150.48, 144.17, 135.42, 134.07, 129.35, 128.30, 127.52, 125.91, 52.35, 45.97, 42.90, 34.57, 31.40, 21.74; HRMS (ESI, *m/z*) calcd. For C<sub>22</sub>H<sub>27</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 339.1960; found: 339.1969.

#### methyl 4-oxo-4-phenyl-2-(*p*-tolyl)butanoate (5x)



This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as white crystalline solid (38.9 mg, 69%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.97-7.95 (m, 2H), 7.55 (tt, *J*<sub>1</sub> = 7.2 Hz, *J*<sub>2</sub> = 1.2 Hz, 1H), 7.44 (t, *J* = 8 Hz, 2H), 7.23 (d, *J* = 8 Hz, 2H), 7.15 (d, *J* = 8 Hz, 2H), 4.26 (dd, *J*<sub>1</sub> = 10 Hz, *J*<sub>2</sub> = 4 Hz, 1H), 3.92 (dd, *J*<sub>1</sub> = 17.2 Hz, *J*<sub>2</sub> = 9.6 Hz, 1H), 3.68 (s, 3H), 3.24 (dd, *J*<sub>1</sub> = 18 Hz, *J*<sub>2</sub> = 4 Hz, 1H), 2.33 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 197.81, 174.11, 137.37, 136.52, 135.43, 133.37, 129.69, 128.68, 128.17, 127.77, 52.40, 46.02, 42.93, 21.14; HRMS (ESI, *m/z*) calcd. For C<sub>18</sub>H<sub>19</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 283.1334; found: 283.1326.

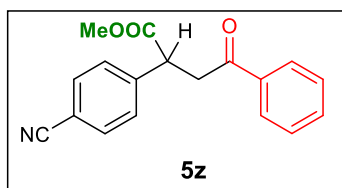
#### methyl 2-([1,1'-biphenyl]-4-yl)-4-(3,4-dimethoxyphenyl)-4-oxobutanoate (5y)



This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as white solid (52.5 mg, 65%). <sup>1</sup>H

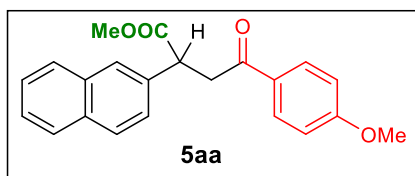
NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.61 (dd,  $J_1 = 8$  Hz,  $J_2 = 2$  Hz, 1H), 7.57 (m, 4H), 7.52 (d,  $J = 2$  Hz, 1H), 7.44-7.41 (m, 4H), 7.34 (tt,  $J_1 = 7.2$  Hz,  $J_2 = 2$  Hz, 1H), 6.86 (d,  $J = 8.4$  Hz, 1H), 4.33 (dd,  $J_1 = 10.0$  Hz,  $J_2 = 4$  Hz, 1H), 3.95-3.88 (m, 7H), 3.72 (s, 3H), 3.29 (dd,  $J_1 = 18$  Hz,  $J_2 = 4.4$  Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  196.26, 174.06, 153.59, 149.12, 140.65, 137.54, 129.73, 128.88, 128.36, 127.71, 127.48, 127.13, 122.92, 110.21, 110.12, 56.16, 56.06, 52.48, 46.27, 42.44; HRMS (ESI, m/z) calcd. For C<sub>25</sub>H<sub>25</sub>O<sub>5</sub> [M+H]<sup>+</sup>: 405.1702; found: 405.1702.

#### methyl 2-(4-cyanophenyl)-4-oxo-4-phenylbutanoate (5z)



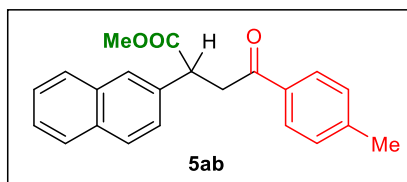
This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as white solid (36.9 mg, 63%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.96-7.92 (m, 2H), 7.64-7.61 (m, 2H), 7.56 (tt,  $J_1 = 8$  Hz,  $J_2 = 1.2$  Hz, 1H), 7.48-7.43 (m, 4H), 4.36 (q,  $J = 4.4$  Hz, 1H), 3.90 (q,  $J = 9.2$  Hz, 1H), 3.69 (s, 3H), 3.30 (dd,  $J_1 = 18$  Hz,  $J_2 = 4.8$  Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  197.4, 163.2, 148.0, 138.1, 131.4, 130.9, 130.2, 128.2, 127.8, 127.5, 126.1, 126.0, 113.4, 55.5, 49.7, 43.8, 42; HRMS (ESI, m/z) calcd. For C<sub>18</sub>H<sub>16</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 294.1130; found: 294.1133.

#### methyl 4-(4-methoxyphenyl)-2-(naphthalen-2-yl)-4-oxobutanoate (5aa)



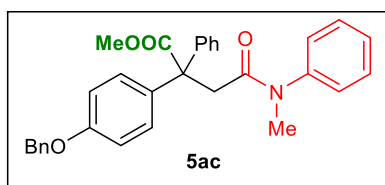
This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as reddish white solid (45.9 mg, 66%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.97-7.94 (m, 2H), 7.83-7.80 (m, 4H), 7.48-7.45 (m, 3H), 6.92-6.90 (m, 2H), 4.45 (dd,  $J_1 = 6$  Hz,  $J_2 = 4$  Hz, 1H), 3.98 (dd,  $J_1 = 18$  Hz,  $J_2 = 10.4$  Hz, 1H), 3.85 (s, 3H), 3.69 (s, 3H), 3.30 (dd,  $J_1 = 18$  Hz,  $J_2 = 4$  Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  196.13, 174.06, 163.76, 135.99, 133.57, 132.78, 130.47, 129.62, 128.73, 127.89, 127.74, 126.76, 126.42, 126.10, 125.91, 113.84, 55.56, 52.45, 46.62, 42.54; HRMS (ESI, m/z) calcd. For C<sub>22</sub>H<sub>21</sub>O<sub>4</sub> [M+H]<sup>+</sup>: 349.1440; found: 349.1444.

#### methyl 2-(naphthalen-2-yl)-4-oxo-4-(p-tolyl)butanoate (5ab)



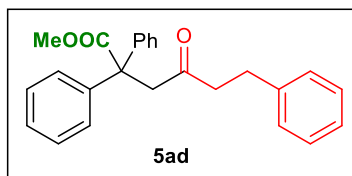
This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as white solid (47.2 mg, 71%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.89-7.87 (m, 2H), 7.83-7.80 (m, 4H), 7.49-7.44 (m, 3H), 7.24-7.23 (m, 2H), 4.46 (dd, *J*<sub>1</sub> = 10.0 Hz, *J*<sub>2</sub> = 4.0 Hz, 1H), 4.02 (dd, *J*<sub>1</sub> = 18.0 Hz, *J*<sub>2</sub> = 10.0 Hz, 1H), 3.70 (s, 3H), 3.33 (dd, *J*<sub>1</sub> = 18.0 Hz, *J*<sub>2</sub> = 4.0 Hz, 1H), 2.39 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 197.28, 174.01, 144.26, 135.93, 134.03, 133.57, 132.79, 129.38, 128.75, 128.31, 127.90, 127.74, 126.77, 126.43, 126.12, 125.90, 52.47, 46.57, 42.76, 21.74; HRMS (ESI, *m/z*) calcd. For C<sub>22</sub>H<sub>20</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 332.1412; found: 332.1413.

**methyl 2-(4-(benzyloxy)phenyl)-4-(methyl(phenyl)amino)-4-oxo-2-phenylbutanoate (5ac)**



This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as yellow solid (71.9 mg, 83%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.43-7.27 (m, 8H), 7.26-7.18 (m, 4H), 6.90-6.86 (m, 2H), 5.03 (s, 2H), 3.73 (s, 3H), 3.55 (s, 3H), 3.46 (s, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 173.65, 171.22, 157.80, 142.67, 137.01, 134.74, 129.86, 128.67, 128.54, 128.08, 127.62, 127.14, 114.26, 57.03, 52.65, 51.75, 43.88; HRMS (ESI, *m/z*) calcd. For C<sub>31</sub>H<sub>30</sub>NO<sub>5</sub> [M+H]<sup>+</sup>: 480.2175; found: 480.2169.

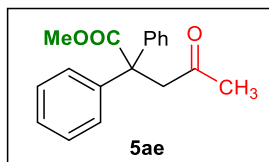
**methyl 4-oxo-2,2,6-triphenylhexanoate (5ad)**



This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as white gum (40.9 mg, 55%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.26-7.16 (m, 13H), 7.08-7.06 (m, 2H), 3.68 (s, 3H), 3.58 (s, 2H),

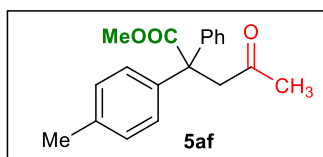
2.79 (t,  $J = 7.6$  Hz, 1H), 2.62 (t,  $J = 7.6$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  206.42, 173.61, 142.92, 140.91, 128.69, 128.52, 128.37, 128.04, 126.99, 126.15, 57.12, 52.51, 52.10, 44.88, 29.54; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{25}\text{H}_{24}\text{O}_3$   $[\text{M}+\text{H}]^+$ : 373.1804; found: 373.1801.

#### methyl 4-oxo-2,2-diphenylpentanoate (5ae)



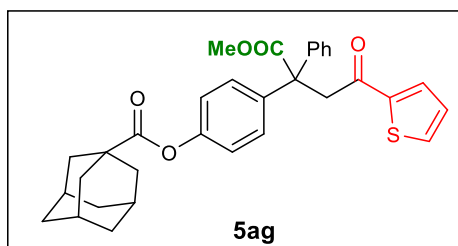
This was prepared following the general procedure and purified in column chromatography ( $\text{SiO}_2$ , eluting with 90:10 hexane/ethyl acetate) affording as colourless gummy liquid (29.9 mg, 53%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.28-7.20 (m, 10 H), 3.71 (s, 3H), 3.64 (s, 2H), 2.07 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  205.02, 173.64, 142.99, 128.69, 128.04, 127.00, 57.05, 52.54, 30.72; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{18}\text{H}_{18}\text{O}_3$   $[\text{M}+\text{H}]^+$ : 283.1334; found: 283.1334.

#### methyl 4-oxo-2-phenyl-2-(p-tolyl)pentanoate (5af)



This was prepared following the general procedure and purified in column chromatography ( $\text{SiO}_2$ , eluting with 90:10 hexane/ethyl acetate) affording as colourless gummy liquid (29.6 mg, 50%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.26-7.19 (m, 5H), 7.13-7.11 (m, 2H), 7.07-7.05 (m, 2H), 3.70 (s, 3H), 3.61 (d,  $J = 2.4$  Hz, 2H), 2.30 (s, 3H), 2.07 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  205.10, 173.77, 143.14, 139.99, 136.66, 128.76, 128.69, 128.51, 127.98, 126.92, 56.73, 52.71, 52.50, 30.74, 21.02; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{19}\text{H}_{20}\text{O}_3$   $[\text{M}+\text{H}]^+$ : 297.1491; found: 297.1499.

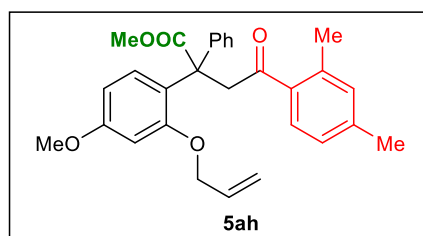
#### 4-(1-methoxy-1,4-dioxo-2-phenyl-4-(thiophen-2-yl)butan-2-yl)phenyl adamantane-1-carboxylate (5ag) (3r,5r,7r)-





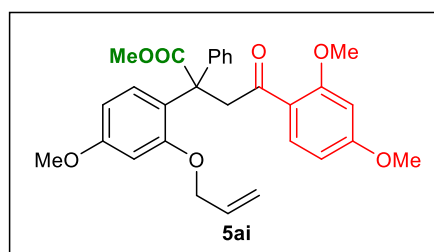
This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as ash coloured solid (88.7 mg, 84%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.67 (dd, *J*<sub>1</sub> = 4.0 Hz, *J*<sub>2</sub> = 1.2 Hz, 1H), 7.59 (dd, *J*<sub>1</sub> = 4.8 Hz, *J*<sub>2</sub> = 0.8 Hz, 1H), 7.32-7.26 (m, 6H), 7.24-7.21 (m, 1H), 7.09-7.07 (m, 1H), 6.96-6.93 (m, 2H), 4.15-4.05 (m, 2H), 3.73 (s, 3H), 2.06-2.01 (m, 9H), 1.78-1.71 (m, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 189.27, 176.10, 173.44, 149.91, 143.99, 142.78, 140.10, 133.76, 131.86, 129.99, 128.70, 128.14, 127.14, 120.93, 56.80, 52.66, 48.70, 41.11, 38.81, 36.53, 27.98; HRMS (ESI, *m/z*) calcd. For C<sub>32</sub>H<sub>32</sub>O<sub>5</sub>S [M+H]<sup>+</sup>: 529.2049; found: 529.2061.

**methyl 2-(2-(allyloxy)-4-methoxyphenyl)-4-(2,4-dimethylphenyl)-4-oxo-2-phenylbutanoate (5ah)**



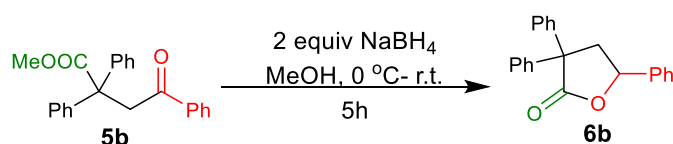
This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as yellow gel (53.1 mg, 58%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.50 (d, *J* = 8.0 Hz, 1H), 7.44-7.42 (m, 2H), 7.33-7.27 (m, 2H), 7.23-7.17 (m, 2H), 7.00-6.96 (m, 2H), 6.40 (dd, *J*<sub>1</sub> = 8.8 Hz, *J*<sub>2</sub> = 2.8 Hz, 1H), 6.34 (d, *J* = 2.4 Hz, 1H), 5.79-5.69 (m, 1H), 5.22-5.13 (m, 2H), 4.37-4.16 (m, 4H), 3.74 (s, 3H), 3.64 (s, 3H), 2.31 (s, 3H), 2.25 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 201.22, 174.69, 159.80, 156.77, 141.35, 140.68, 138.09, 136.22, 132.96, 132.59, 131.71, 128.95, 128.82, 128.52, 127.74, 126.77, 126.50, 126.11, 124.17, 117.37, 103.82, 100.28, 69.28, 56.20, 55.34, 52.40, 45.88, 21.37, 21.10; HRMS (ESI, *m/z*) calcd. For C<sub>29</sub>H<sub>30</sub>O<sub>5</sub> [M+H]<sup>+</sup>: 459.2171; found: 459.2172.

**methyl 2-(2-(allyloxy)-4-methoxyphenyl)-4-(2,4-dimethoxyphenyl)-4-oxo-2-phenylbutanoate (5ai)**



This was prepared following the general procedure and purified in column chromatography (SiO<sub>2</sub>, eluting with 90:10 hexane/ethyl acetate) affording as yellow gel (66.6 mg, 68%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.63 (dd, *J*<sub>1</sub> = 8.4 Hz, *J*<sub>2</sub> = 2.0 Hz, 1H), 7.44-7.40 (m, 3H), 7.29-7.21 (m, 4H), 6.84 (d, *J* = 8.4 Hz, 1H), 6.43 (dd, *J*<sub>1</sub> = 8.4 Hz, *J*<sub>2</sub> = 2.4 Hz, 1H), 6.37 (d, *J* = 2.8 Hz, 1H), 5.86-5.76 (m, 1H), 5.21 (qq, *J*<sub>1</sub> = 15.6 Hz, *J*<sub>2</sub> = 2.0 Hz, 2H), 4.39-4.30 (m, 4H), 3.92 (s, 3H), 3.85 (s, 3H), 3.75 (s, 3H), 3.64 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 195.86, 174.85, 159.76, 156.75, 153.12, 148.96, 140.37, 133.02, 132.01, 130.95, 128.85, 127.79, 126.85, 124.10, 122.52, 117.33, 110.29, 109.91, 103.80, 100.13, 69.27, 56.14, 55.99, 55.30, 52.45, 42.37; HRMS (ESI, *m/z*) calcd. For C<sub>29</sub>H<sub>30</sub>O<sub>7</sub> [M+H]<sup>+</sup>: 491.2070; found: 491.2077.

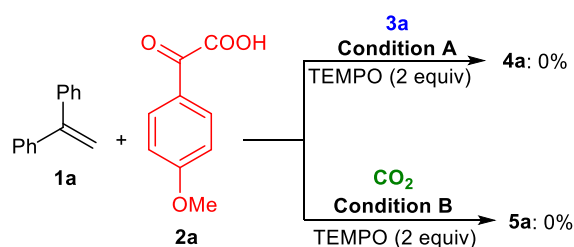
## 7. Product derivatization



In a clean 25 mL round bottom flask, methyl 4-oxo-2,2,4-triphenylbutanoate (**5b**) (68.8 mg, 0.2 mmol, 1 equiv.) was taken and to it 2 mL methanol was added. Then the solution was set up for stirring at. At 0 °C, sodium borohydride (NaBH<sub>4</sub>) (15.1 mg, 0.4 mmol, 2 equiv.) was added to the mixture and the flask was closed. Then, the reaction mixture was allowed to stir at room temperature for 5 hours. After that, 5 mL 2(N) HCl was added dropwise to the reaction mixture to quench the excess NaBH<sub>4</sub>. Therefore, reaction mixture was extracted with ethyl acetate (40 mL), water (15 mL × 2), washed with brine (15 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography (SiO<sub>2</sub>, eluting with hexane/ethylacetate 98:2) to afford **6b** as white solid (39 mg, 61%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.47-7.26 (m, 15H), 5.32 (q, *J* = 5.2 Hz, 1H), 3.30 (dd, *J*<sub>1</sub> = 12.8 Hz, *J*<sub>2</sub> = 4.8 Hz, 1H), 2.94 (dd, *J*<sub>1</sub> = 12.8 Hz, *J*<sub>2</sub> = 10.8 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 176.96, 141.73, 139.62, 138.41, 129.18, 128.87, 128.77, 128.49, 127.97, 127.81, 127.49, 127.41, 125.76, 78.07, 58.79, 46.35; HRMS (ESI, *m/z*) calcd. For C<sub>32</sub>H<sub>32</sub>O<sub>5</sub>S [M+H]<sup>+</sup>: 315.1385; found: 315.1297.

## 8. Control experiments

### 8.1. Radical inhibition experiment



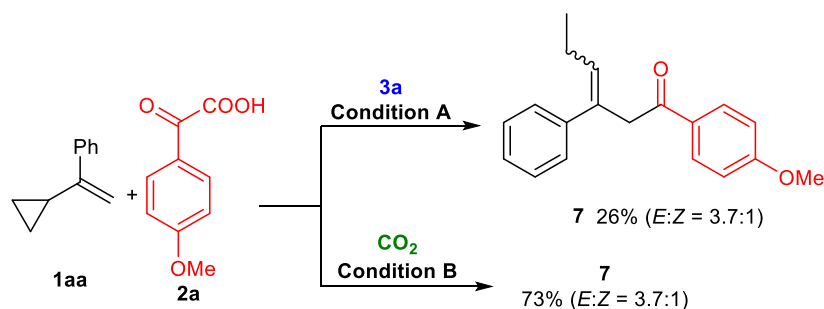
### 8.1.1. Radical inhibition experiment for carbobenzoylation (Condition A)

1-benzyl-2,4,6-triphenylpyridin-1-ium tetrafluoroborate **3a** (0.4 mmol, 2.0 equiv., 194 mg), 2-(4-methoxyphenyl)-2-oxoacetic acid **2a** (0.4 mmol, 2.0 equiv., 72 mg), tris(2,2'-bipyridyl)dichlororuthenium(II) hexahydrate (0.002 mmol, 1 mol %, 2.9 mg), caesium carbonate (0.4 mmol, 2.0 equiv., 130 mg) and TEMPO (62.5 mg, 0.4 mmol, 2.0 equiv.) were taken in a 7 mL screw-capped vial. MeCN was added to the mixture. The whole mixture was de-gassed and re-filled with inert gas by two consecutive freeze-pump-thaw cycles followed by the addition of 1,1-diphenylethylene **1a** (0.2 mmol, 1.0 equiv., 35  $\mu$ L). The reaction mixture was then stirred under 5W blue LED irradiation for 2 hours. After 2 hours, TLC was checked and no formation of the expected product **4a** was observed.

### 8.1.2. Radical inhibition experiment for carbocarboxylation (Condition B)

The oven-dried Schlenk tube (10 mL) containing a stirring bar was charged with **2a** (43.2 mg, 0.24 mmol, 1.2 equiv), 4-CzIPN (1.5 mg, 0.002 mmol, 1.0 mol %), **2** (0.24 mmol, 1.2 equiv), TEMPO (62.5 mg, 0.4 mmol, 2.0 equiv.) and transferred to glovebox to add Cs<sub>2</sub>CO<sub>3</sub> (130.3 mg, 0.4 mmol, 2.0 equiv). The tube was then evacuated and back-filled with CO<sub>2</sub> for 3 times. Therefore, under continuous CO<sub>2</sub> flow, anhydrous DMSO (3 mL) and **1a** (35  $\mu$ L, 1.0 equiv., 0.2 mmol) was added with syringe and subsequently the tube is sealed. The reaction was stirred with irradiating with a 30 W blue LED lamp (3 cm away, with cooling fan to keep the reaction temperature at 25~30 °C) for 12 hours. After 12 hours, the light is switched off, the shlenk tube is opened and to the mixture, methyl iodide (24.9  $\mu$ L, 0.4 mmol, 2.0 equiv.) is added. The mixture is stirred for additional 4 hours at room temperature. After that, TLC was checked which showed no formation of expected product **5a**.

## 8.2. Radical-clock experiment



### 8.2.1. Radical-clock experiment for carbobenzylation (Condition A)

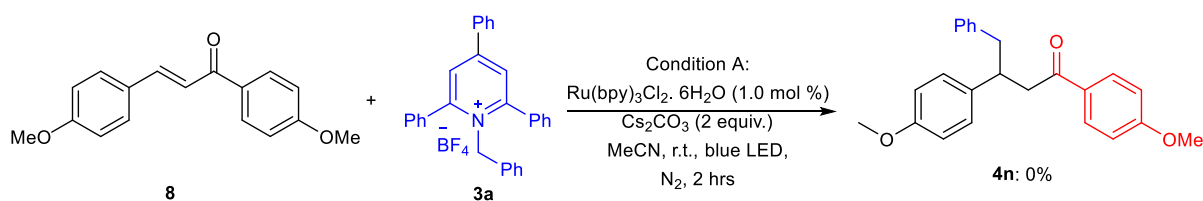
1-benzyl-2,4,6-triphenylpyridin-1-ium tetrafluoroborate **2a** (0.4 mmol, 2.0 equiv., 194 mg), 2-(4-methoxyphenyl)-2-oxoacetic acid **3a** (0.4 mmol, 2.0 equiv., 72 mg), tris(2,2'-bipyridyl)dichlororuthenium(II) hexahydrate (0.002 mmol, 1 mol %, 2.9 mg), caesium carbonate (0.4 mmol, 2.0 equiv., 130 mg) were taken in a 7 mL screw-capped vial. MeCN was added to the mixture. The whole mixture was de-gassed and re-filled with inert gas by two consecutive freeze-pump-thaw cycles followed by the addition of radical-clock substrate **1aa** (0.2 mmol, 1.0 equiv., 28.8 mg). The reaction mixture was then stirred under 5W blue LED irradiation for 2 hours. After 2 hours, TLC was checked and no formation of the expected product **4a** was observed, instead ring opening product **7** was achieved with column chromatography (eluting with petroleum ether/ethyl acetate 97:3) as colourless liquid (9 mg, 26%).

### 8.2.2. Radical-clock experiment for carbocarboxylation (Condition B)

The oven-dried Schlenk tube (10 mL) containing a stirring bar was charged with **2a** (43.2 mg, 0.24 mmol, 1.2 equiv), 4-CzIPN (1.5 mg, 0.002 mmol, 1.0 mol %) and transferred to glovebox to add Cs<sub>2</sub>CO<sub>3</sub> (130.3 mg, 0.4 mmol, 2.0 equiv). The tube was then evacuated and back-filled with CO<sub>2</sub> for 3 times. Therefore, under continuous CO<sub>2</sub> flow, anhydrous DMSO (3.0 mL) and radical-clock substrate **1aa** (0.2 mmol, 1.0 equiv., 28.8 mg) were added with syringe and subsequently the tube is sealed. The reaction was stirred with irradiating with a 30 W blue LED lamp (3 cm away, with cooling fan to keep the reaction temperature at 25~30 °C) for 12 hours. After 12 hours, the light is switched off, the shlenk tube is opened and to the mixture, methyl iodide (24.9 μL, 0.4 mmol, 2.0 equiv.) is added. The mixture is stirred for additional 4 hours at room temperature. After that, TLC was checked which showed no formation of expected product **5a**, instead ring opening product **7** was achieved with column chromatography (eluting with petroleum ether/ethyl acetate 97:3) as colourless liquid (25.3 mg, 73%).

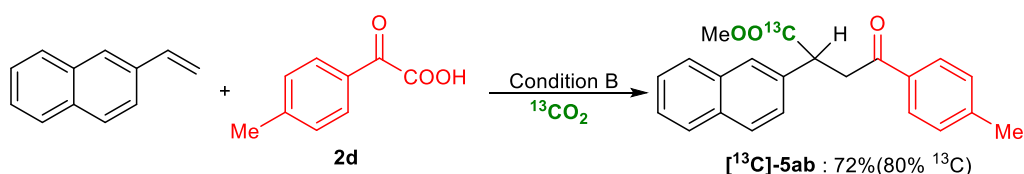
Characterization of **7** (*E* isomer):  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.98-7.94 (m, 2H), 7.31-7.23 (m, 5H), 6.93-6.91 (m, 2H), 5.99 (t,  $J = 7.2$  Hz, 1H), 4.13 (s, 2H), 3.85 (s, 3H), 2.18 (pent.,  $J = 7.6$  Hz, 2H), 1.07 (t,  $J = 7.6$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  196.86, 195.78, 163.56, 163.43, 143.02, 140.65, 134.39, 133.95, 132.81, 130.81, 130.58, 130.07, 128.51, 128.35, 128.17, 126.83, 126.75, 126.11, 126.10, 113.79, 113.69, 55.56, 55.51, 48.48, 40.45, 22.65, 22.62, 14.45, 14.03; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{32}\text{H}_{32}\text{O}_5\text{S}$   $[\text{M}+\text{H}]^+$ : 281.1542; found: 281.1544.

### 8.3. Checking the possibility of Michael addition in carbobenzoylation (Condition A)



1-benzyl-2,4,6-triphenylpyridin-1-ium tetrafluoroborate **3a** (0.4 mmol, 2.0 equiv., 194 mg), (*E*)-1,3-bis(4-methoxyphenyl)prop-2-en-1-one **8** (0.2 mmol, 1.0 equiv.), tris(2,2'-bipyridyl)dichlororuthenium(II) hexahydrate (0.002 mmol, 1 mol %, 2.9 mg), caesium carbonate (0.4 mmol, 2.0 equiv., 130 mg) were taken in a 7 mL screw-capped vial. MeCN was added to the mixture. The whole mixture was de-gassed and re-filled with inert gas by two consecutive freeze-pump-thaw cycles. The reaction mixture was then stirred under 5W blue LED irradiation for 12 hours. After 12 hours, TLC was checked and no formation of the expected product **4n** was observed.

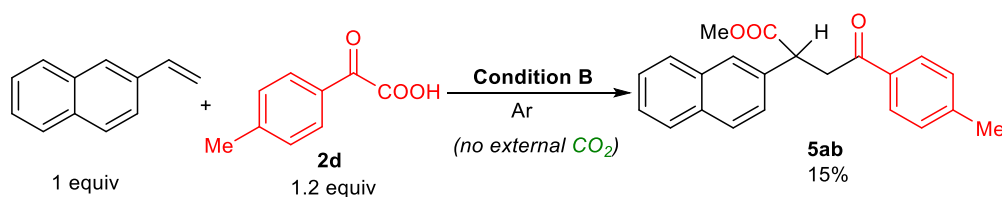
### 8.4. $^{13}\text{C}$ -labelling experiment for carbocarboxylation (Condition B)



The oven-dried Schlenk tube (10 mL) containing a stirring bar was charged with 2-vinylnaphthalene (30.8 mg, 0.20 mmol, 1.0 equiv.), **2d** (39.4 mg, 0.24 mmol, 1.2 equiv.), 4-CzIPN (1.5 mg, 0.002 mmol, 1.0 mol %) and transferred to glovebox to add  $\text{Cs}_2\text{CO}_3$  (130.3 mg, 0.4 mmol, 2.0 equiv.). The tube was then evacuated and back-filled with  $^{13}\text{CO}_2$  for 3 times. Therefore, under continuous  $^{13}\text{CO}_2$  flow, anhydrous DMSO (3.0 mL) was added with syringe and subsequently the tube is sealed. The reaction was stirred with irradiating with a 30 W blue LED lamp (3 cm away, with cooling fan to keep the reaction temperature at 25~30  $^\circ\text{C}$ ) for 12

hours. After 12 hours, the light is switched off, the shlenk tube is opened and to the mixture, methyl iodide (24.9  $\mu\text{L}$ , 0.4 mmol, 2.0 equiv.) is added. The mixture was stirred for additional 4 hours at room temperature. After that, the reaction mixture was extracted with ethyl acetate (30 mL), water (10 mL  $\times$  2), washed with brine (10 mL), dried over anhydrous  $\text{Na}_2\text{SO}_4$  and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography ( $\text{SiO}_2$ , eluting with hexane/ethylacetate 97:3) to afford the desired product [ $^{13}\text{C}$ ]-**5ab** as white solid (47.9 mg, 72% with 80%  $^{13}\text{C}$  incorporation).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.89-7.87 (m, 2H), 7.83-7.80 (m, 4H), 7.49-7.44 (m, 3H), 7.23-7.22 (m, 2H), 4.48-4.43 (m, 1H), 4.01 (qd,  $J_1 = 10.0$  Hz,  $J_2 = 2.8$  Hz, 1H), 3.69 (d,  $J = 4$  Hz, 3H), 3.33 (dq,  $J_1 = 18.0$  Hz,  $J_2 = 4.0$  Hz, 1H), 2.39 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  197.28, 174.01, 144.26, 135.93, 134.03, 133.57, 132.79, 129.38, 128.75, 128.31, 127.90, 127.74, 126.78, 126.43, 126.12, 125.89, 52.47, 46.55 (d,  $J = 57$  Hz, 46.27, 42.76, 21.75; HRMS (ESI,  $m/z$ ) calcd. For  $\text{C}_{22}\text{H}_{20}\text{O}_3$  [ $\text{M}+\text{H}$ ] $^+$ : 333.1491; found: 333.1389.

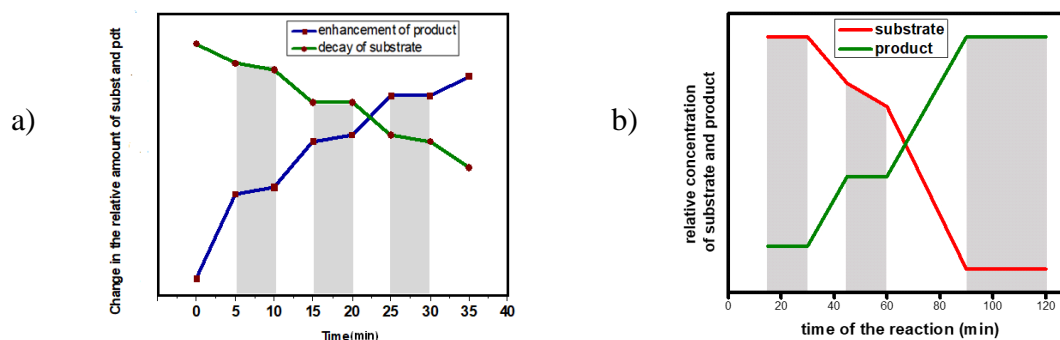
### 8.5. Carbocarboxylation reaction under Argon (Ar) atmosphere



The oven-dried Schlenk tube (10 mL) containing a stirring bar was charged with 2-vinylnaphthalene (30.8 mg, 0.20 mmol, 1.0 equiv.), **2d** (39.4 mg, 0.24 mmol, 1.2 equiv), 4-CzIPN (1.5 mg, 0.002 mmol, 1.0 mol %) and transferred to glovebox to add  $\text{Cs}_2\text{CO}_3$  (130.3 mg, 0.4 mmol, 2.0 equiv). The tube was then evacuated and back-filled with argon (Ar) for 3 times. Therefore, under continuous Ar flow, anhydrous DMSO (3.0 mL) was added with syringe and subsequently the tube is sealed. The reaction was stirred with irradiating with a 30 W blue LED lamp (3 cm away, with cooling fan to keep the reaction temperature at 25~30  $^\circ\text{C}$ ) for 12 hours. After 12 hours, the light is switched off, the shlenk tube is opened and to the mixture, methyl iodide (24.9  $\mu\text{L}$ , 0.4 mmol, 2.0 equiv.) is added. The mixture was stirred for additional 4 hours at room temperature. After that, the reaction mixture was extracted with ethyl acetate (30 mL), water (10 mL  $\times$  2), washed with brine (10 mL), dried over anhydrous  $\text{Na}_2\text{SO}_4$  and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography ( $\text{SiO}_2$ , eluting with hexane/ethylacetate 97:3) to afford the desired carboxylated product **5ab** as white solid (10.0 mg, 15%).

## 8.6. Light on-off experiment for both carbobenylation (Condition A) and carbocarboxylation (Condition B)

In both the conditions, for carbobenylation and carbocarboxylation, the standard reactions were set-up and stirred sequentially under light and in dark with a certain interval.



Light on-off experiment for a) carbobenylation (**Condition A**) and b) carbocarboxylation (**Condition B**)

The corresponding increase of product and decrease of starting material concentration was monitored using 1,2,3,4,5-pentafluoro-6-methylbenzene as internal standard. The relative concentration of the substrates and the corresponding product were calculated and presented graphically.

## 9. References

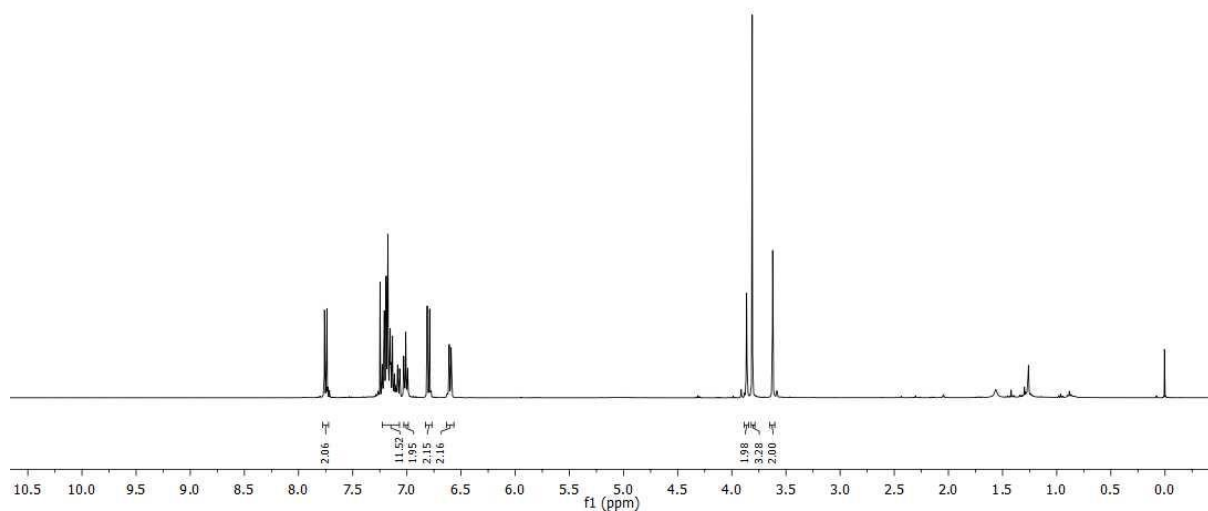
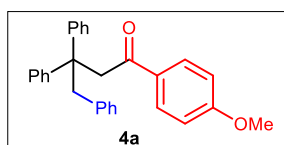
1. C. Wan, R.-J. Song and J.-H. Li, *Org. Lett.*, 2019, **21**, 2800-2803.
2. A. Hossian, K. Manna, P. Das and R. Jana, *ChemistrySelect*, 2018, **3**, 4315-4318.
3. F. J. R. Klauck, H. Yoon, M. J. James, M. Lautens and F. Glorius, *ACS Catal.*, 2019, **9**, 236-241.

## 10. Copies of $^1\text{H}$ and $^{13}\text{C}$ Spectra

PD-4-66  
single\_pulse

7.761  
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7.228  
7.212  
7.206  
7.206  
7.192  
7.179  
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7.158  
7.153  
7.116  
7.085  
7.067  
7.009  
7.010  
4.990  
4.812  
4.776  
4.699  
4.592

3.863  
3.812  
3.623



<sup>1</sup>H spectra of **4a**

—197.391

—163.167

148.047

138.148

131.468

130.992

130.171

128.193

127.877

127.482

126.116

126.038

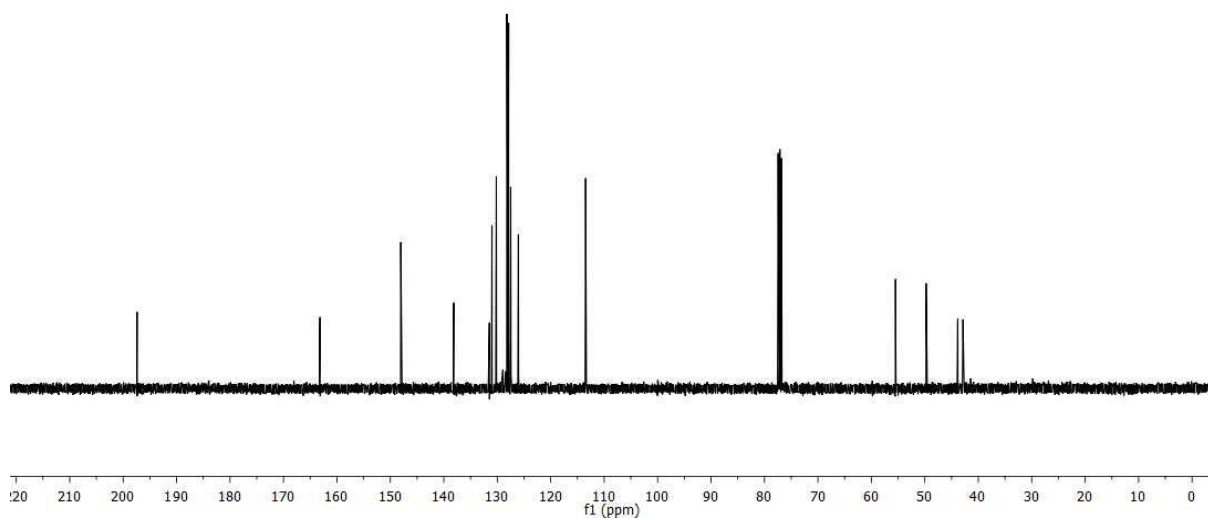
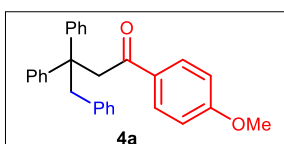
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49.764

43.837

42.862

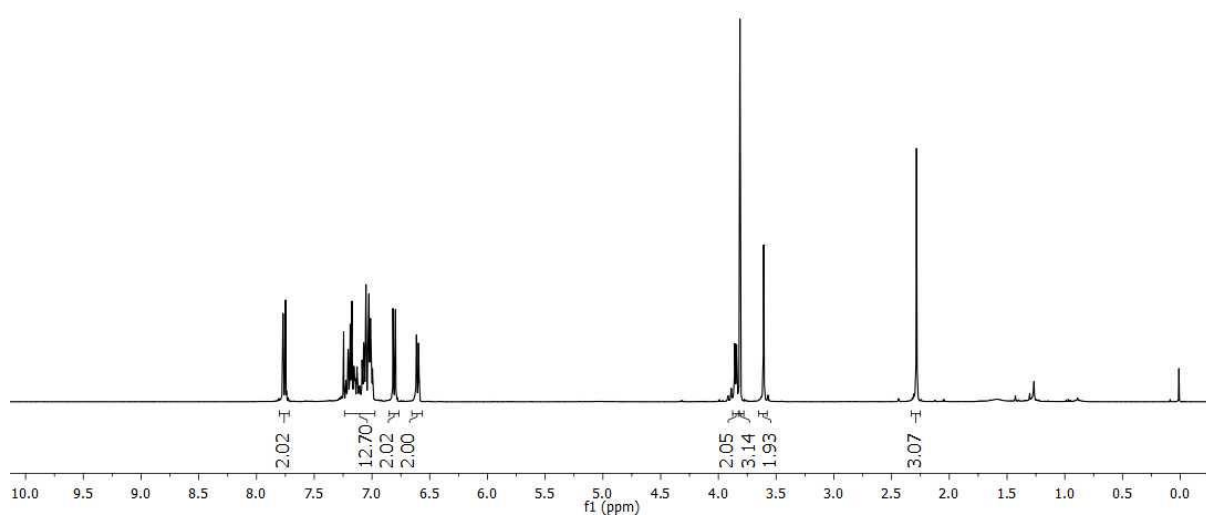
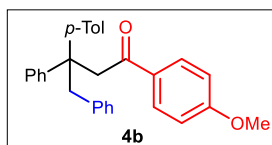


<sup>13</sup>C spectra of **4a**



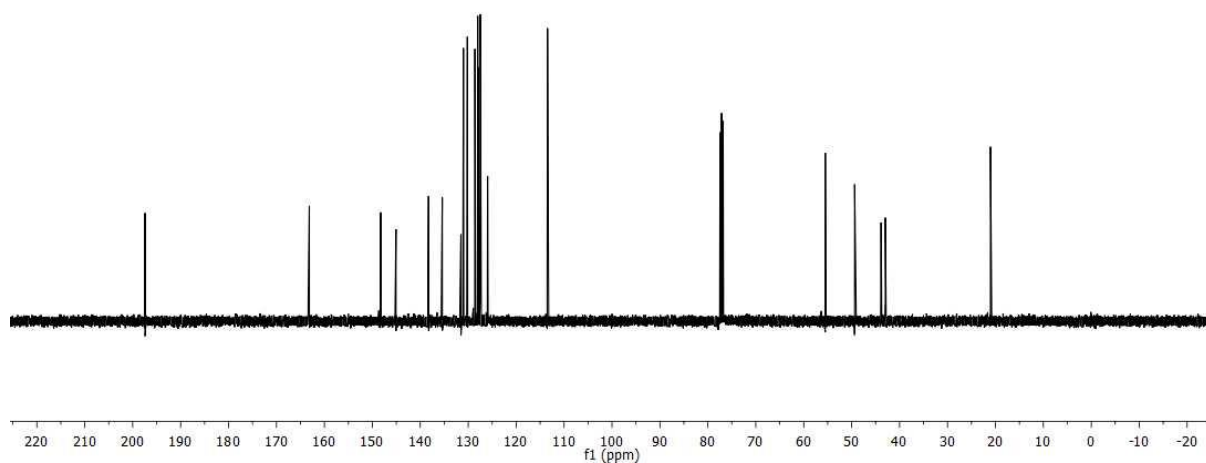
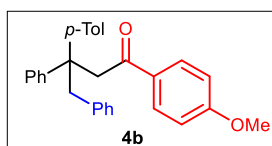
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7.013  
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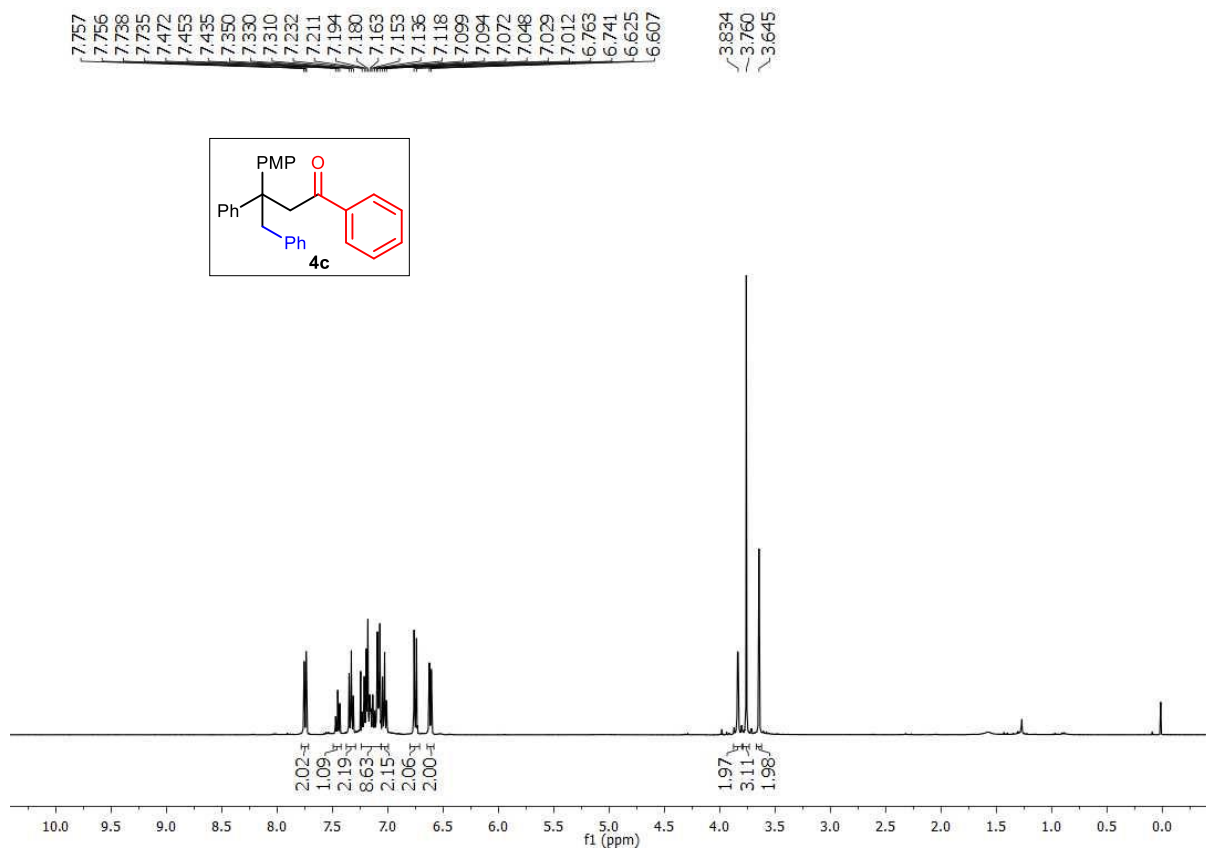


<sup>1</sup>H spectra of **4b**

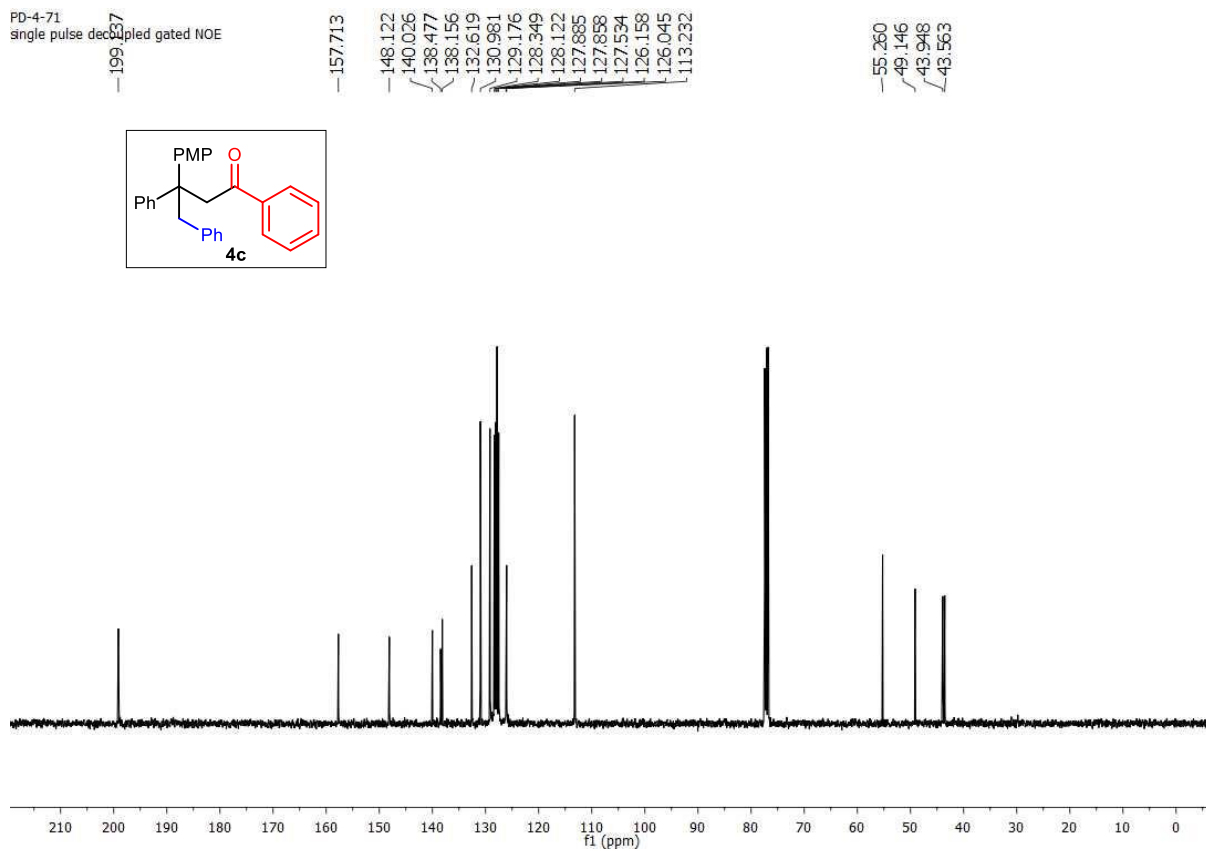
-197.424  
163.148  
148.263  
145.065  
138.281  
135.413  
131.515  
130.998  
130.174  
128.597  
128.131  
128.009  
127.849  
127.468  
126.072  
125.955  
113.453  
-55.487  
49.372  
43.862  
42.964  
-21.025



<sup>13</sup>C spectra of **4b**

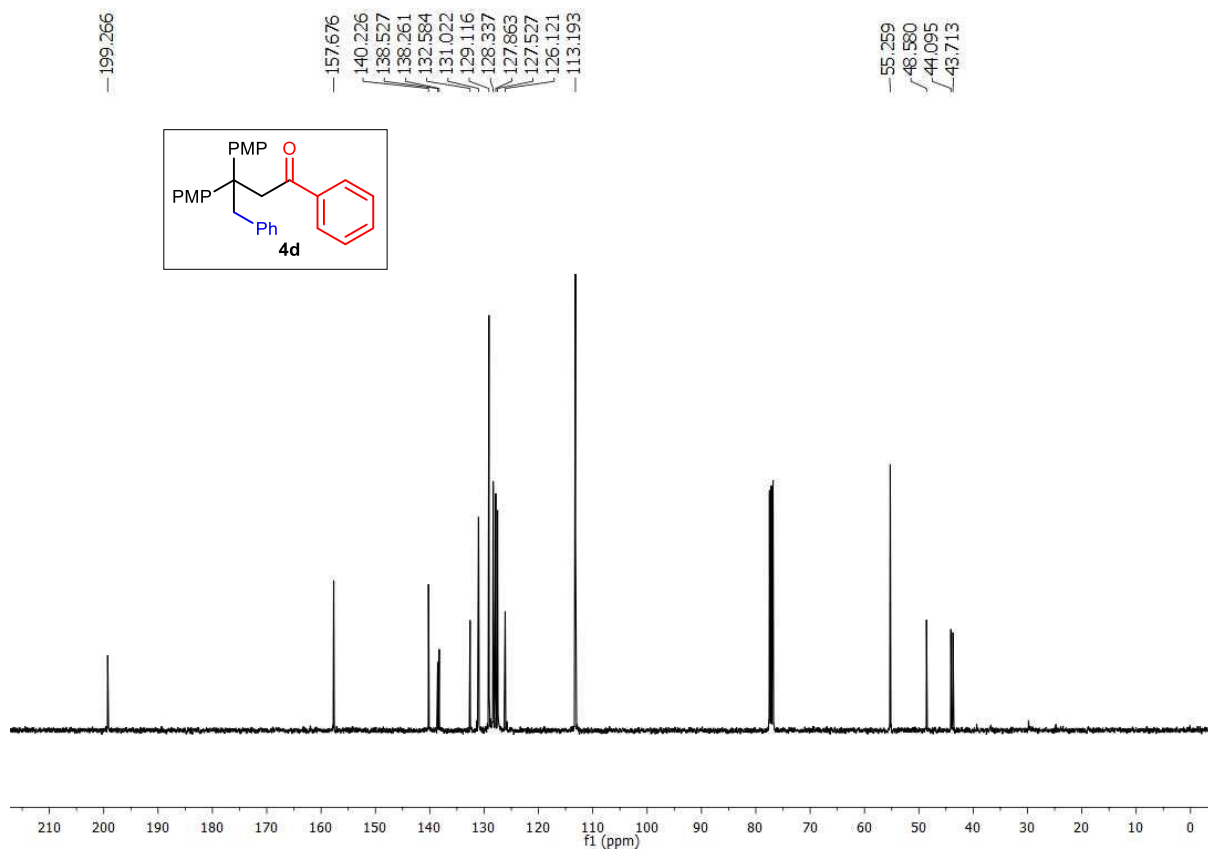
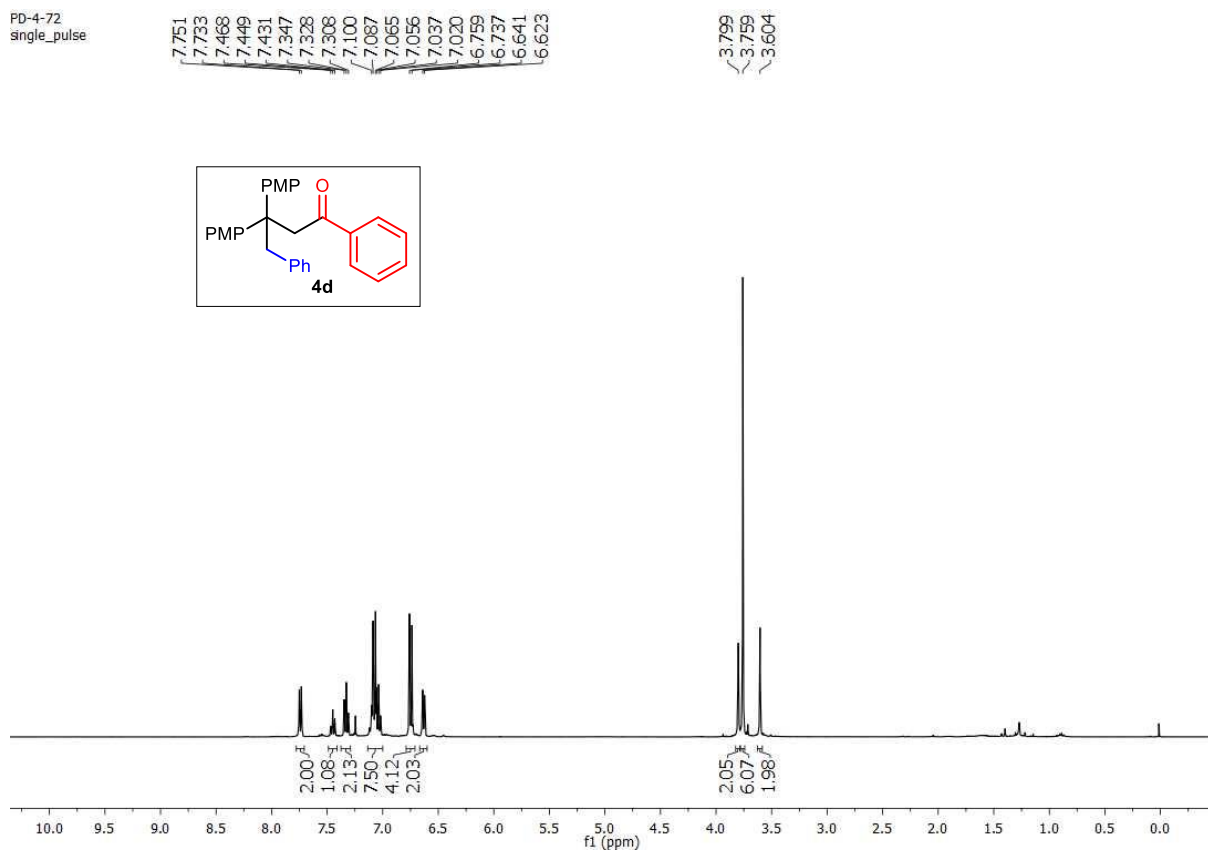


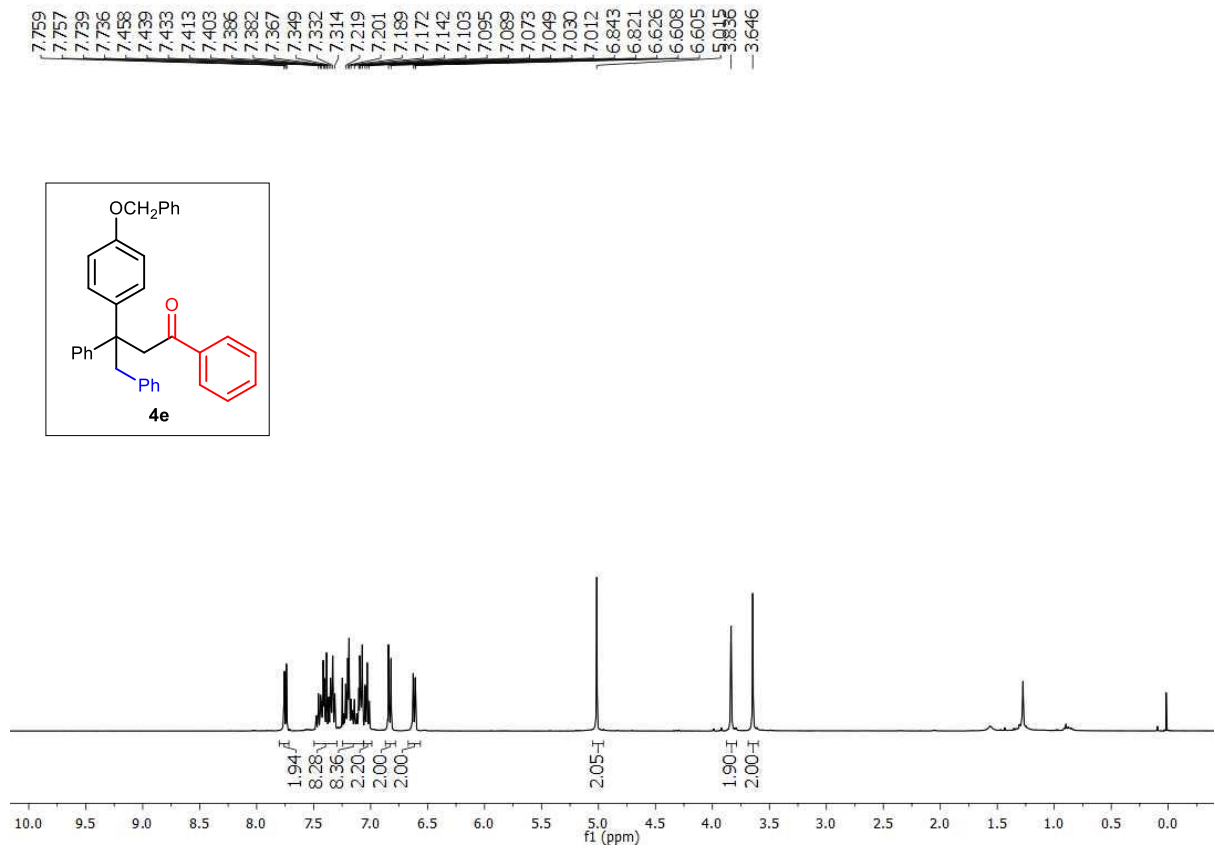
<sup>1</sup>H spectra of **4c**



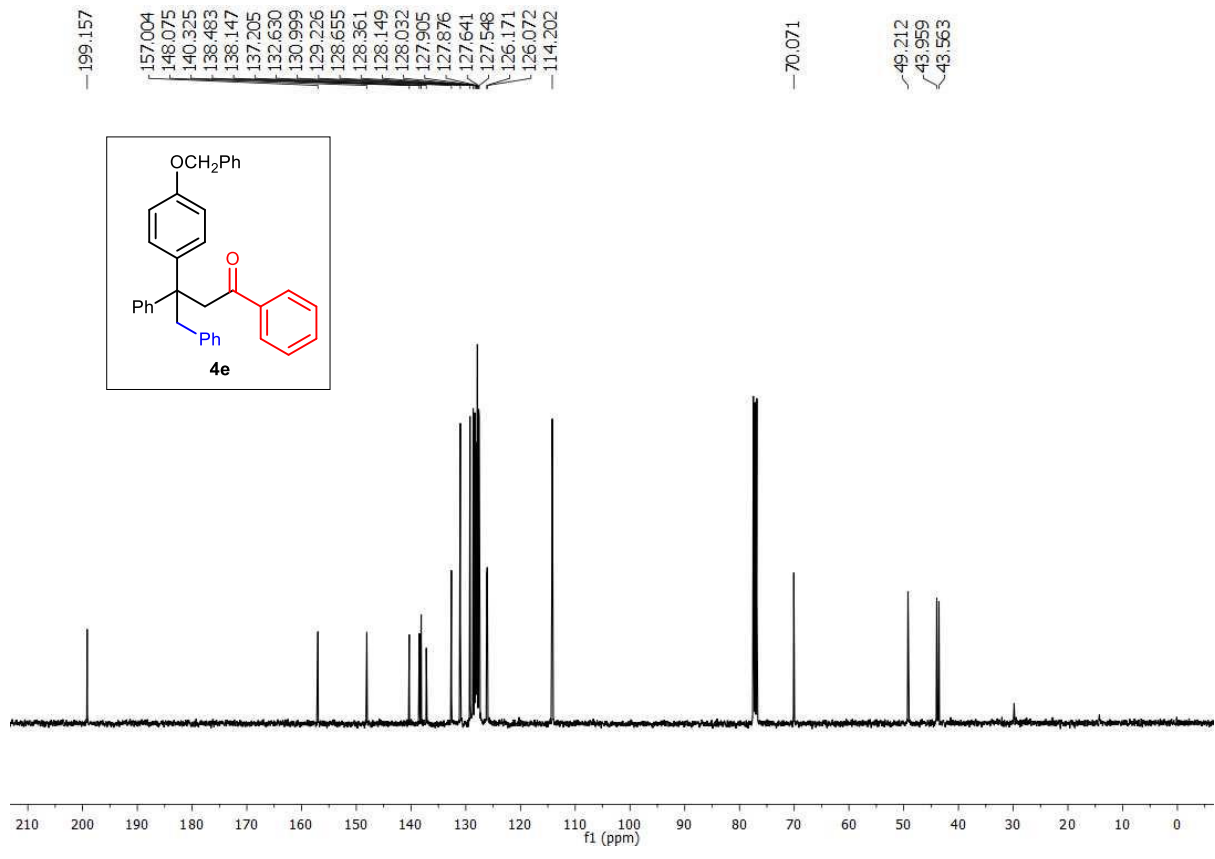
<sup>13</sup>C spectra of **4c**

PD-4-72  
single\_pulse





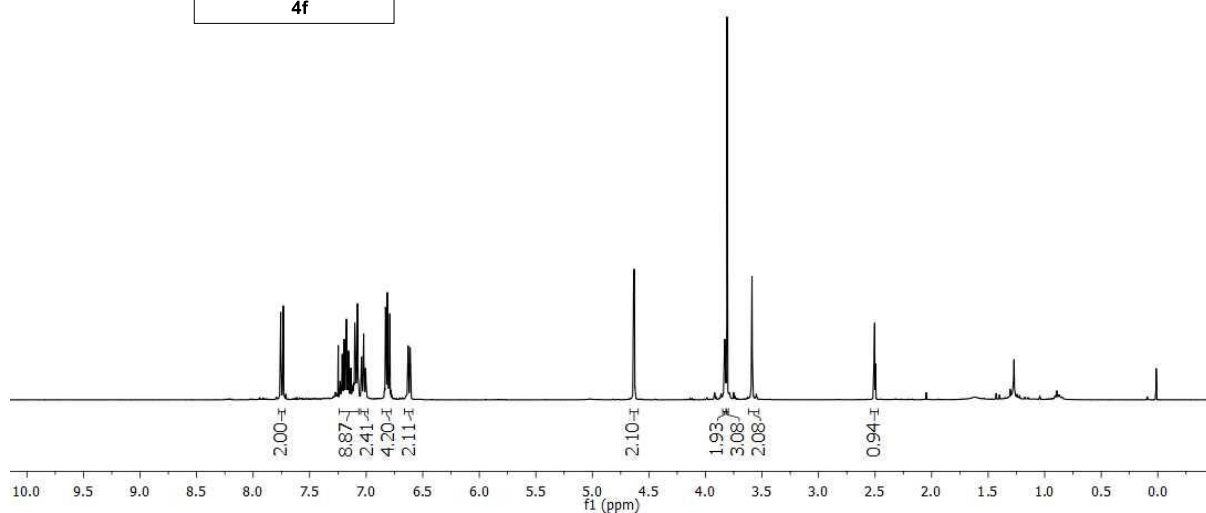
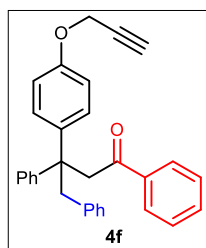
<sup>1</sup>H spectra of **4e**



<sup>13</sup>C spectra of **4e**

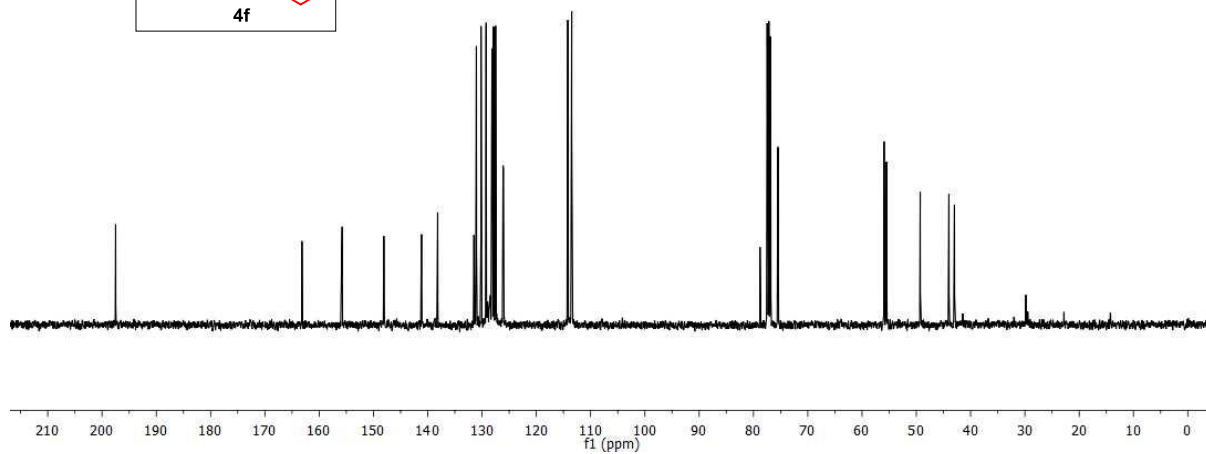
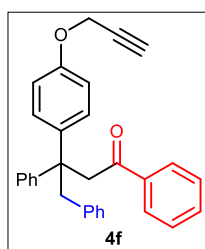
PD-4-172  
single\_pulse

7.756  
7.734  
7.730  
7.209  
7.194  
7.179  
7.174  
7.157  
7.136  
7.101  
7.093  
7.078  
7.041  
7.022  
7.005  
6.829  
6.814  
6.807  
6.791  
6.629  
6.611  
4.634  
4.628  
3.830  
3.826  
3.809  
3.589  
2.510  
2.504  
2.498

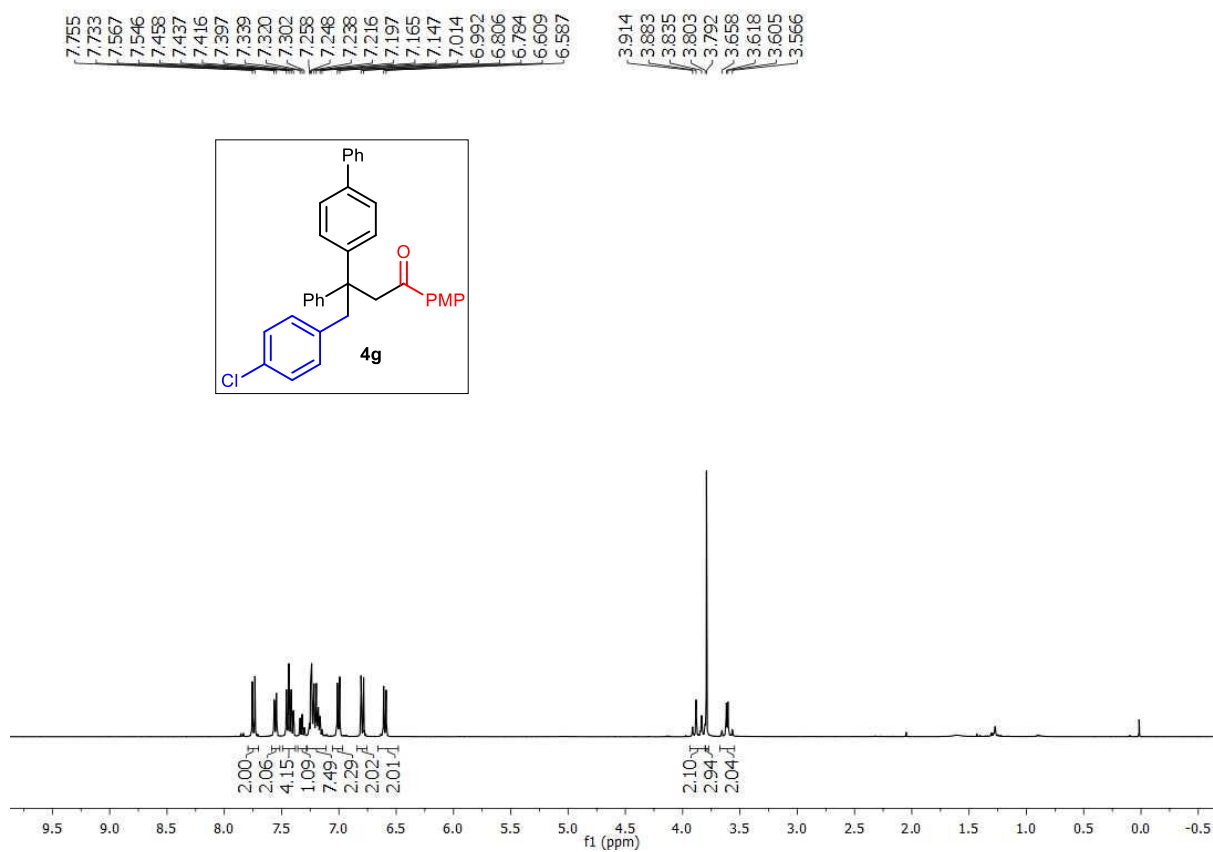


$^1\text{H}$  spectra of **4f**

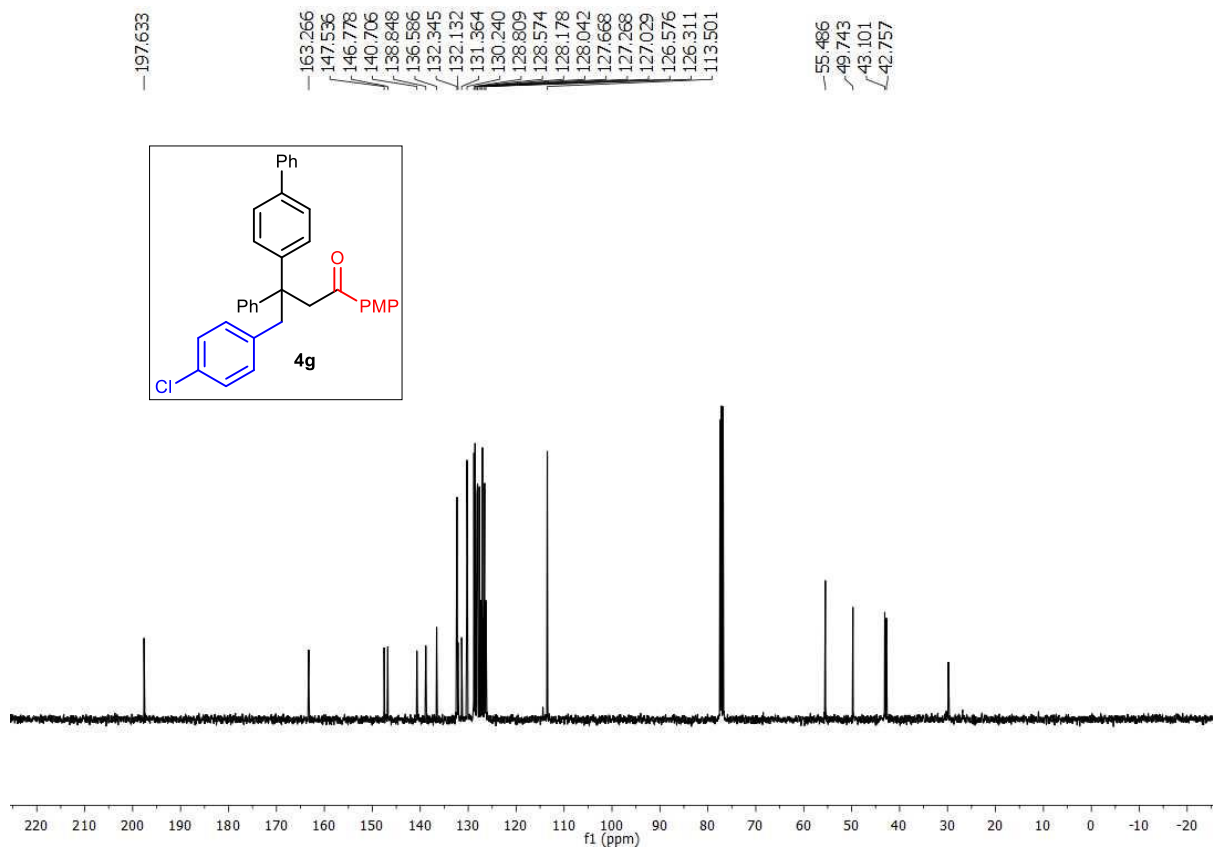
197.491  
163.164  
155.760  
148.085  
141.145  
138.166  
131.487  
131.034  
130.182  
129.244  
128.159  
127.877  
127.501  
126.121  
126.047  
114.193  
113.483  
78.794  
75.489  
55.939  
55.494  
49.282  
43.991  
42.978



$^{13}\text{C}$  spectra of **4f**

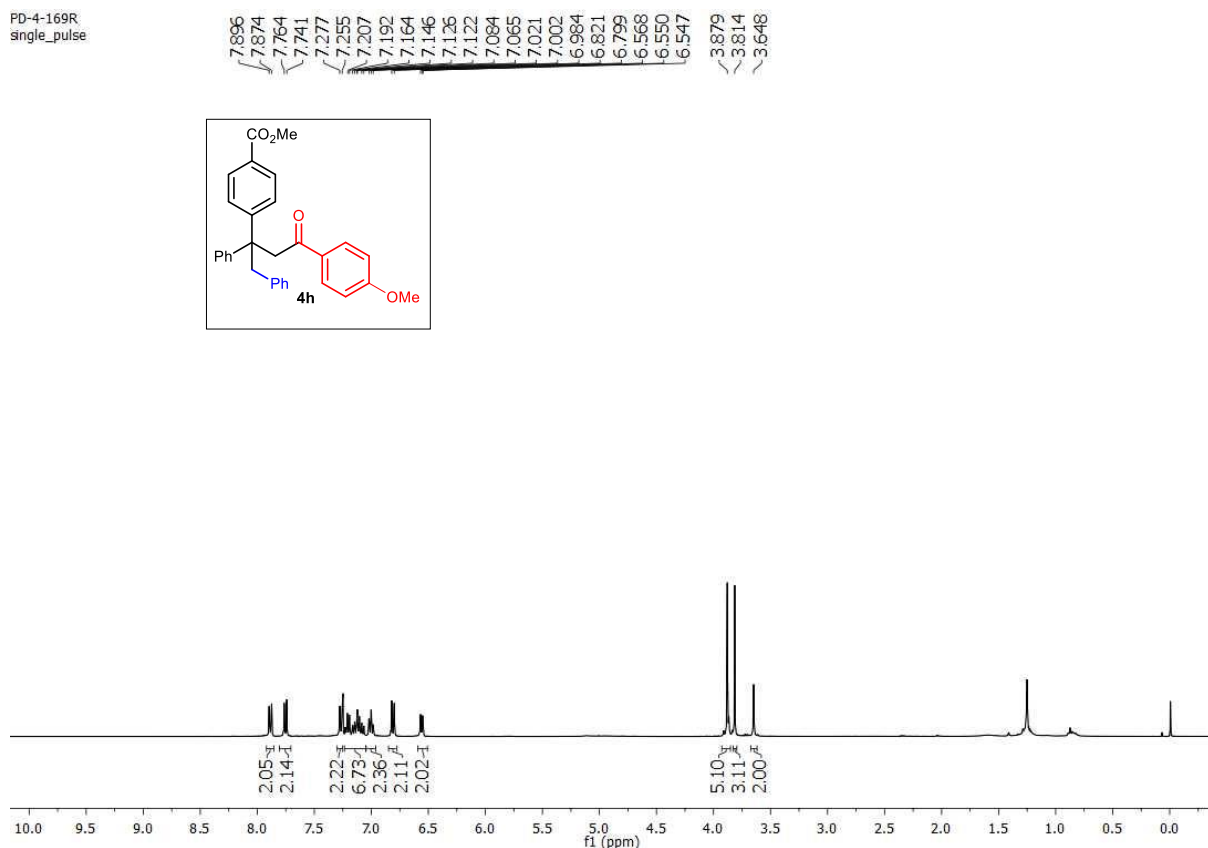


<sup>1</sup>H spectra of **4g**

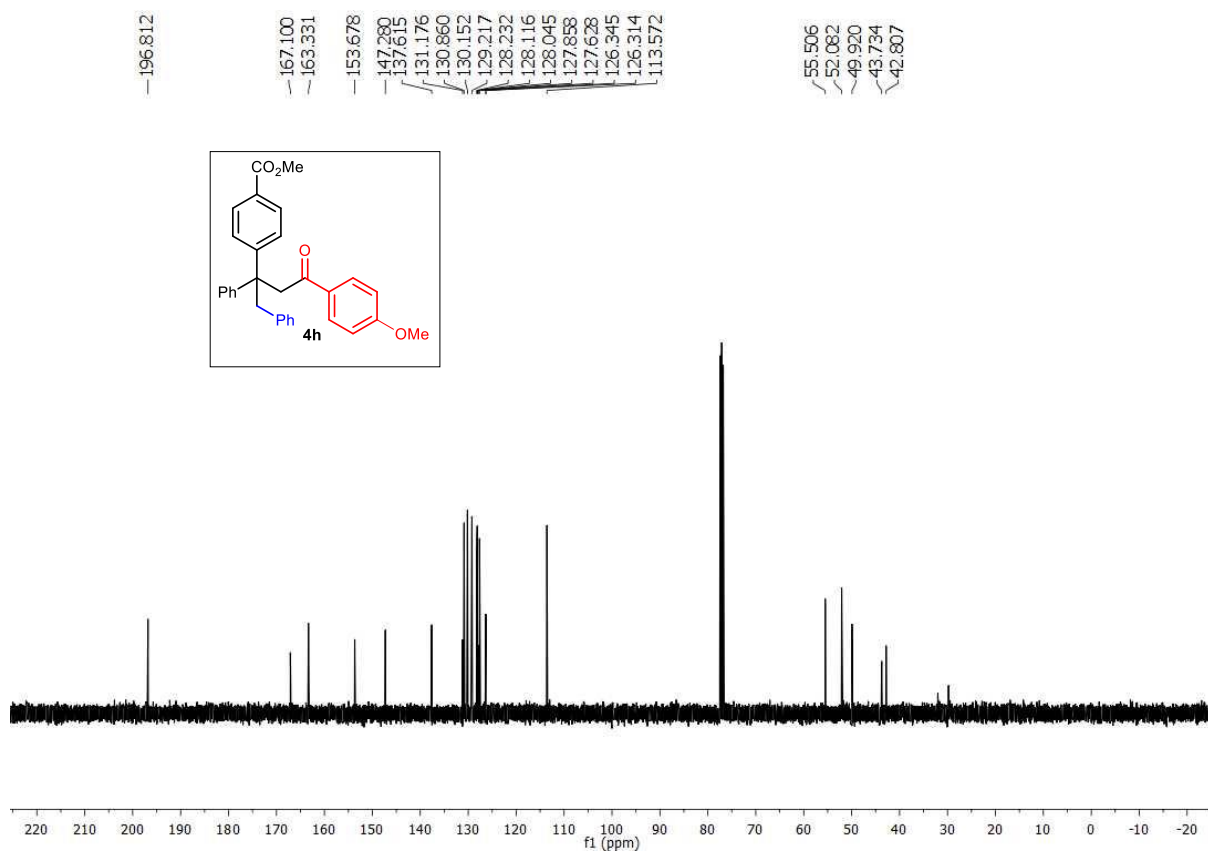


<sup>13</sup>C spectra of **4g**

PD-4-169R  
single\_pulse

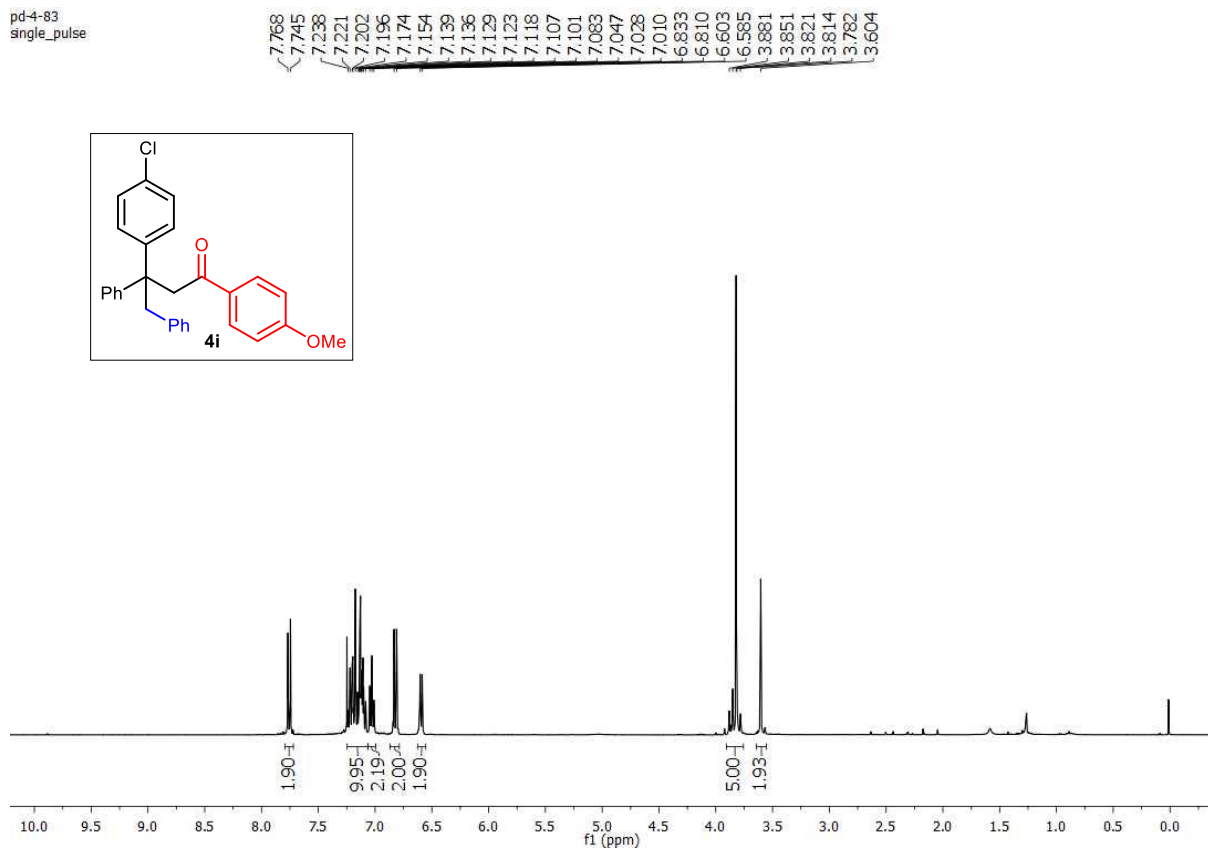


<sup>1</sup>H spectra of **4h**

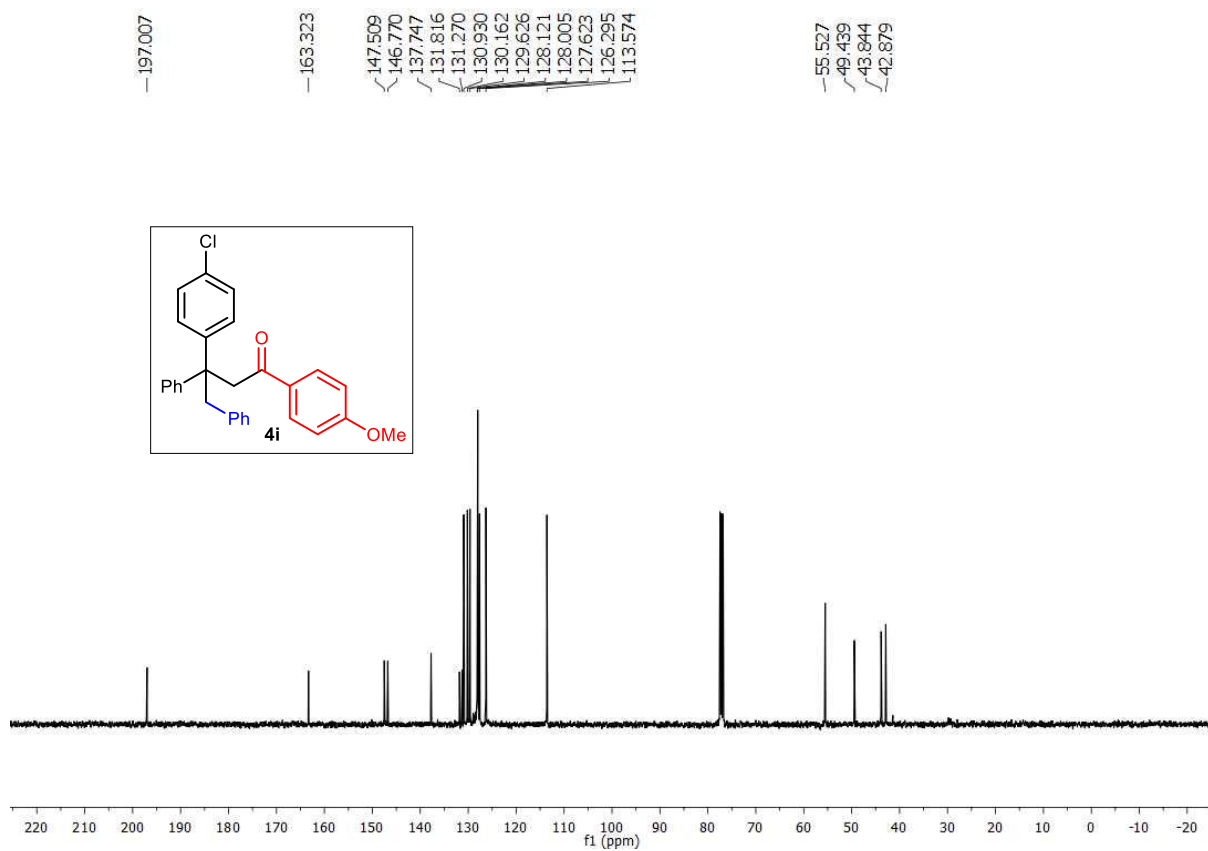


<sup>13</sup>C spectra of **4h**

pd-4-83  
single\_pulse



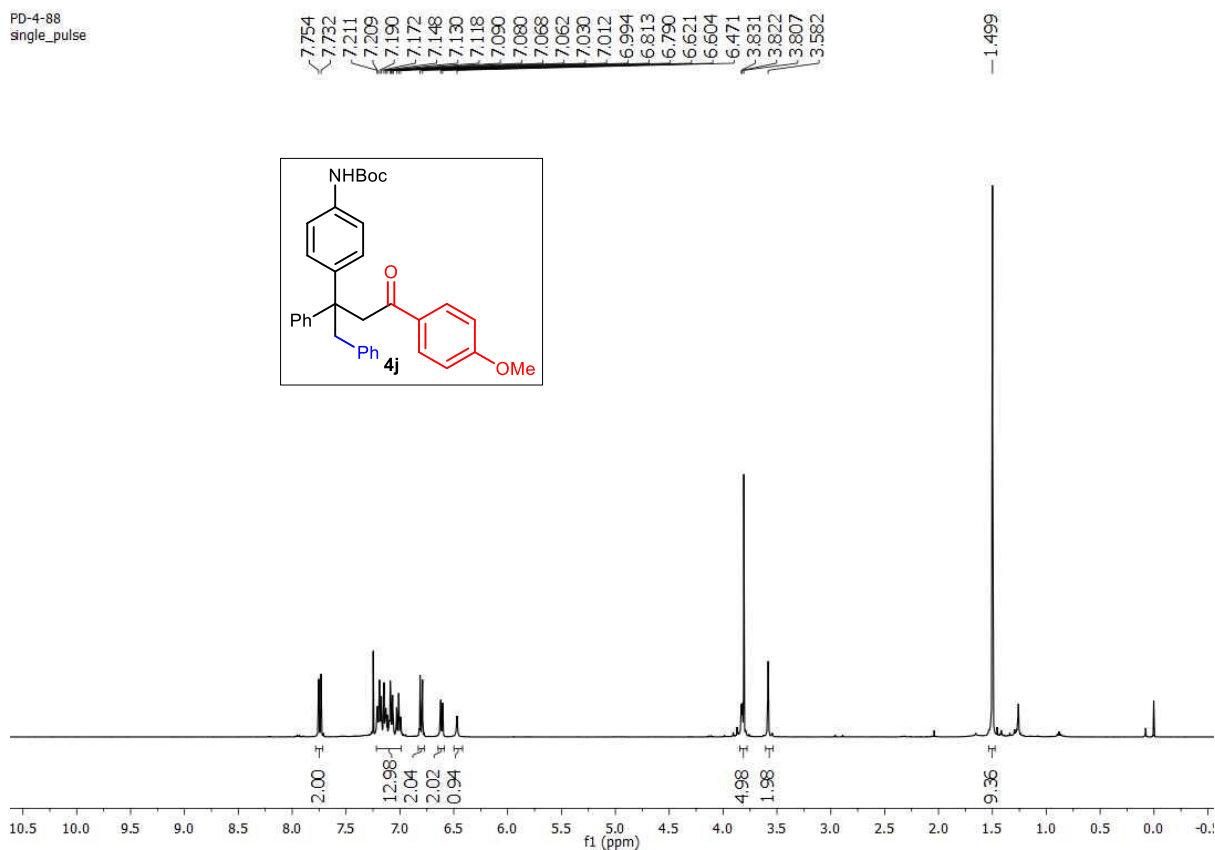
<sup>1</sup>H spectra of **4i**



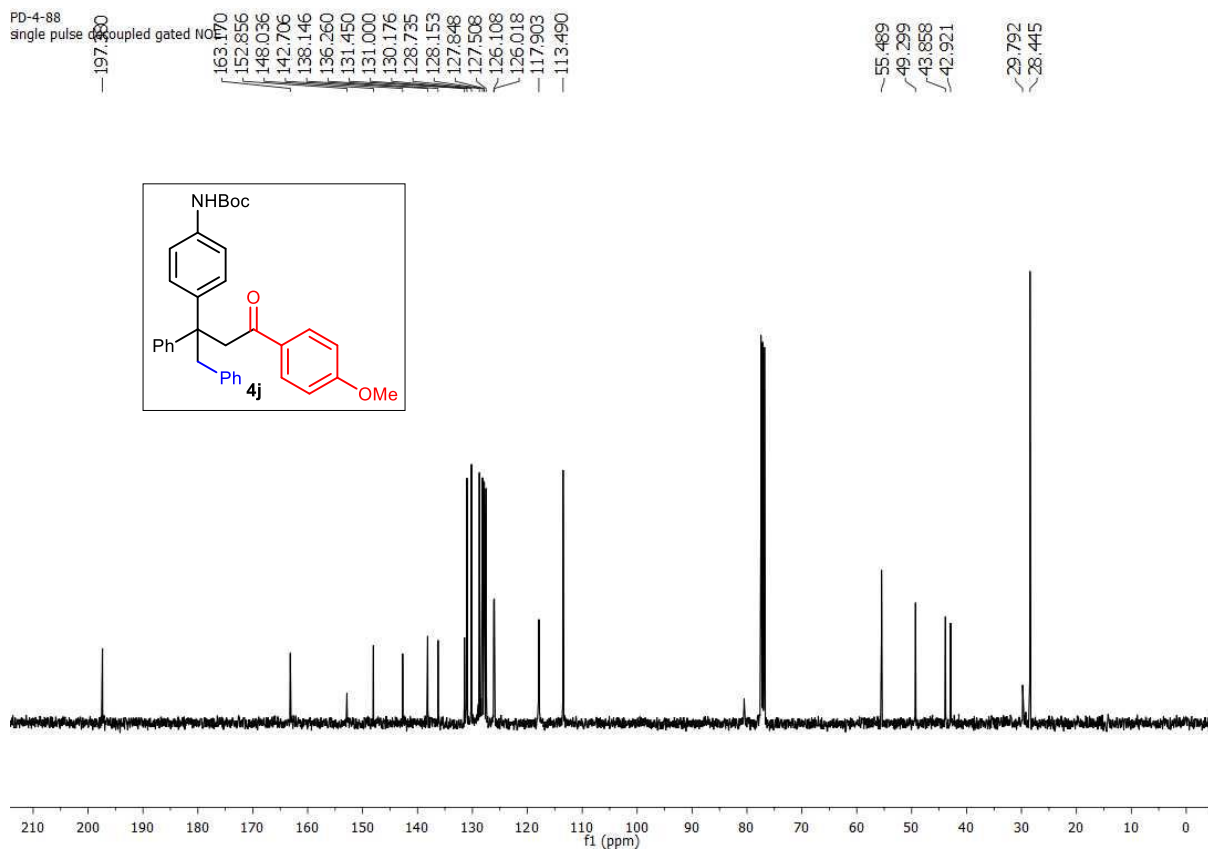
<sup>13</sup>C spectra of **4i**



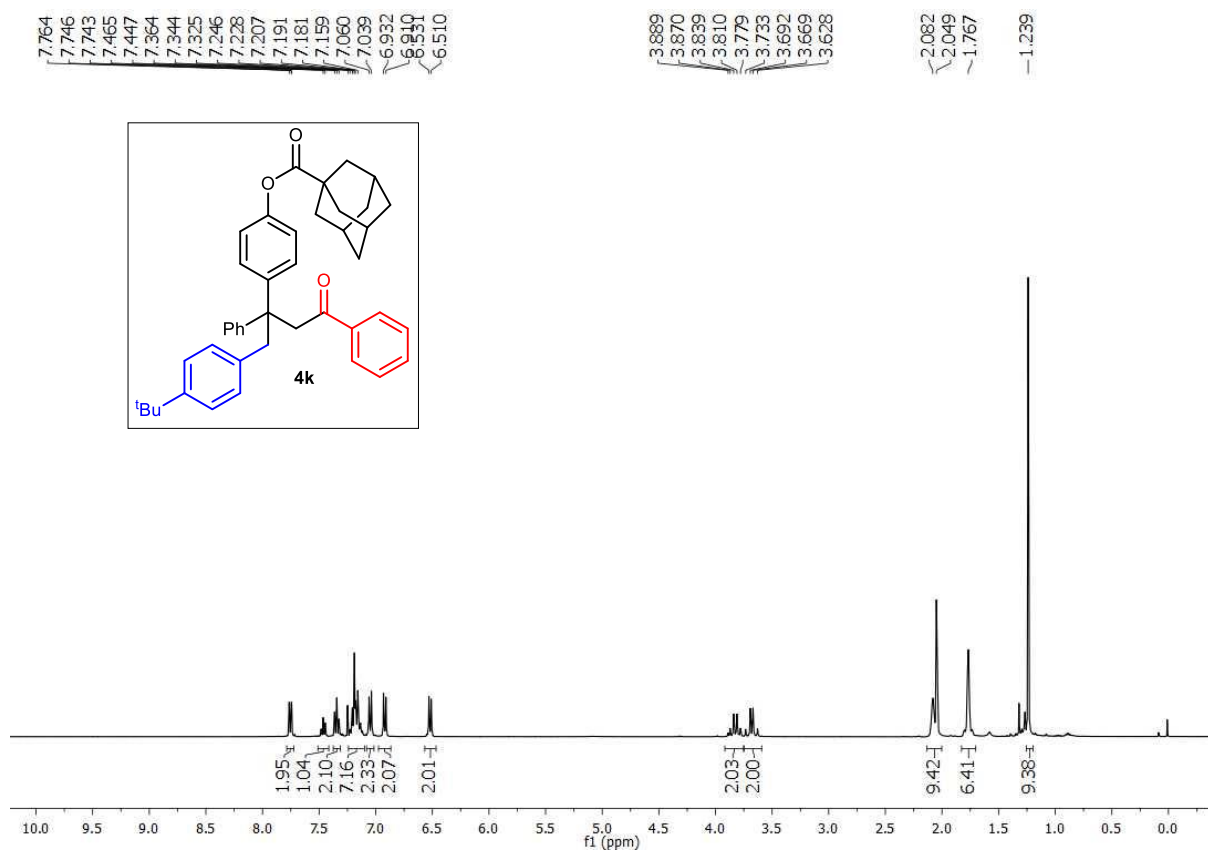
PD-4-88  
single\_pulse



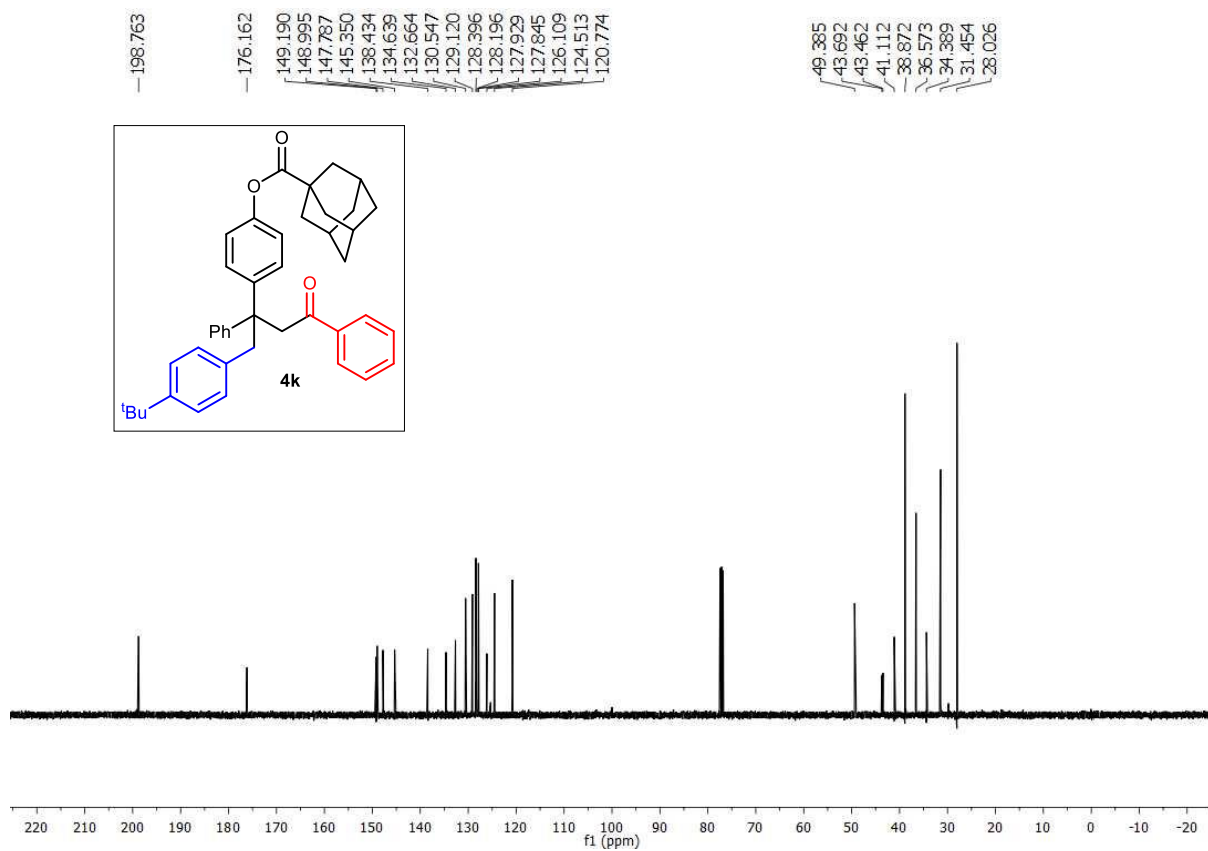
<sup>1</sup>H spectra of **4j**



<sup>13</sup>C spectra of **4j**



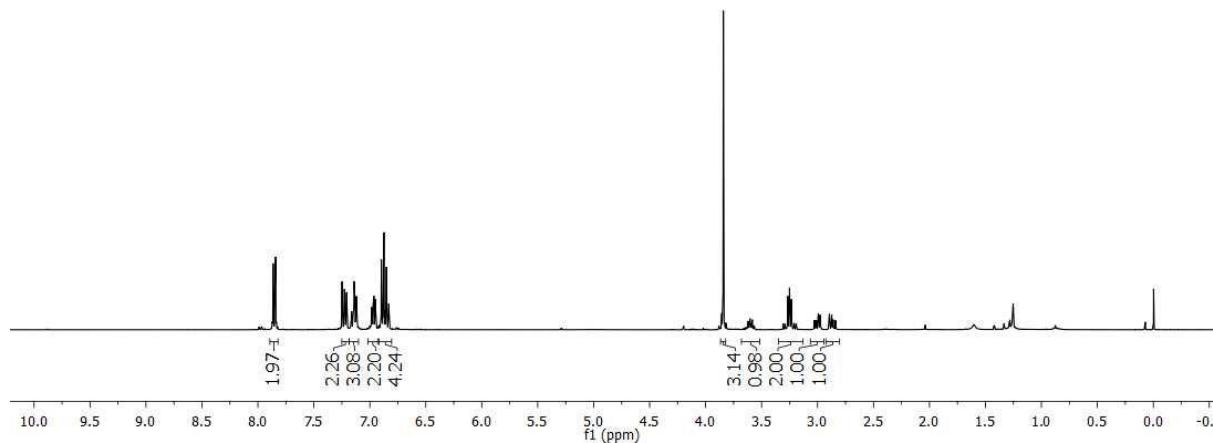
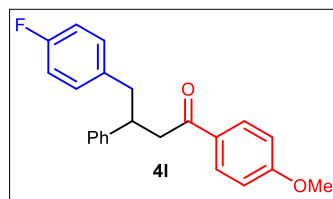
**<sup>1</sup>H spectra of 4k**



**<sup>13</sup>C spectra of 4k**

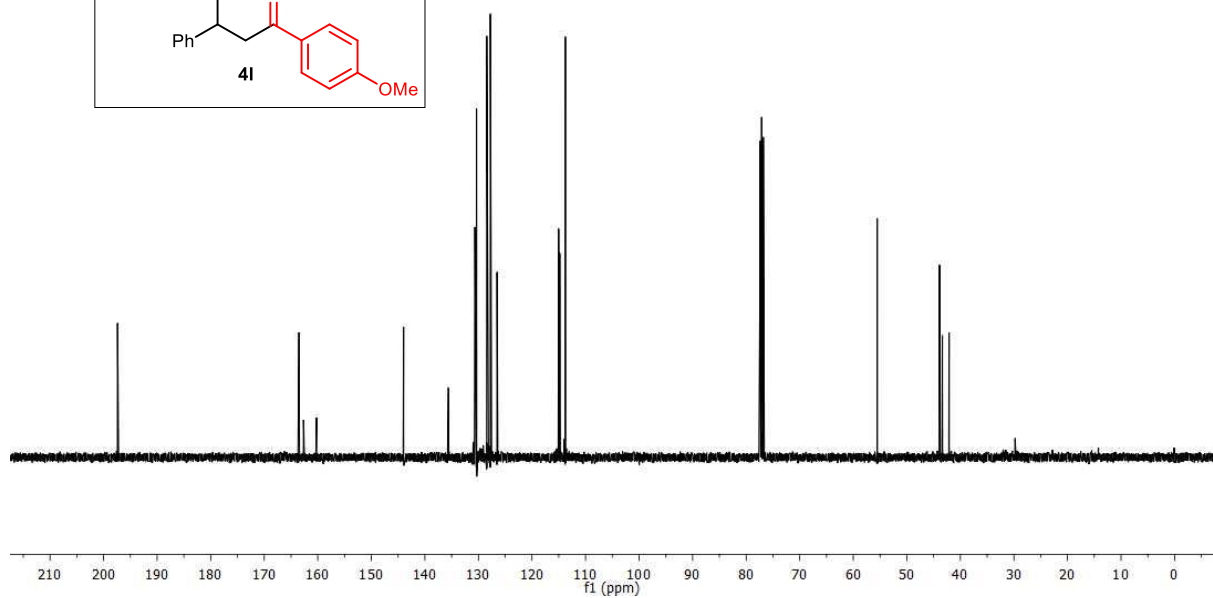
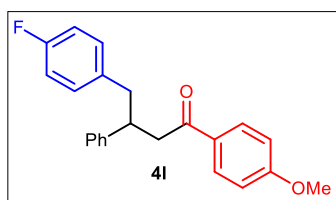
PD-4-103  
single\_pulse

7.864  
7.841  
7.226  
7.208  
7.163  
7.145  
7.137  
7.120  
6.985  
6.971  
6.963  
6.949  
6.897  
6.874  
6.852  
6.830  
3.842  
3.658  
3.637  
3.621  
3.604  
3.600  
3.583  
3.566  
3.307  
3.290  
3.265  
3.250  
3.233  
3.209  
3.191  
3.027  
3.011  
2.993  
2.977  
2.894  
2.873  
2.861  
2.840



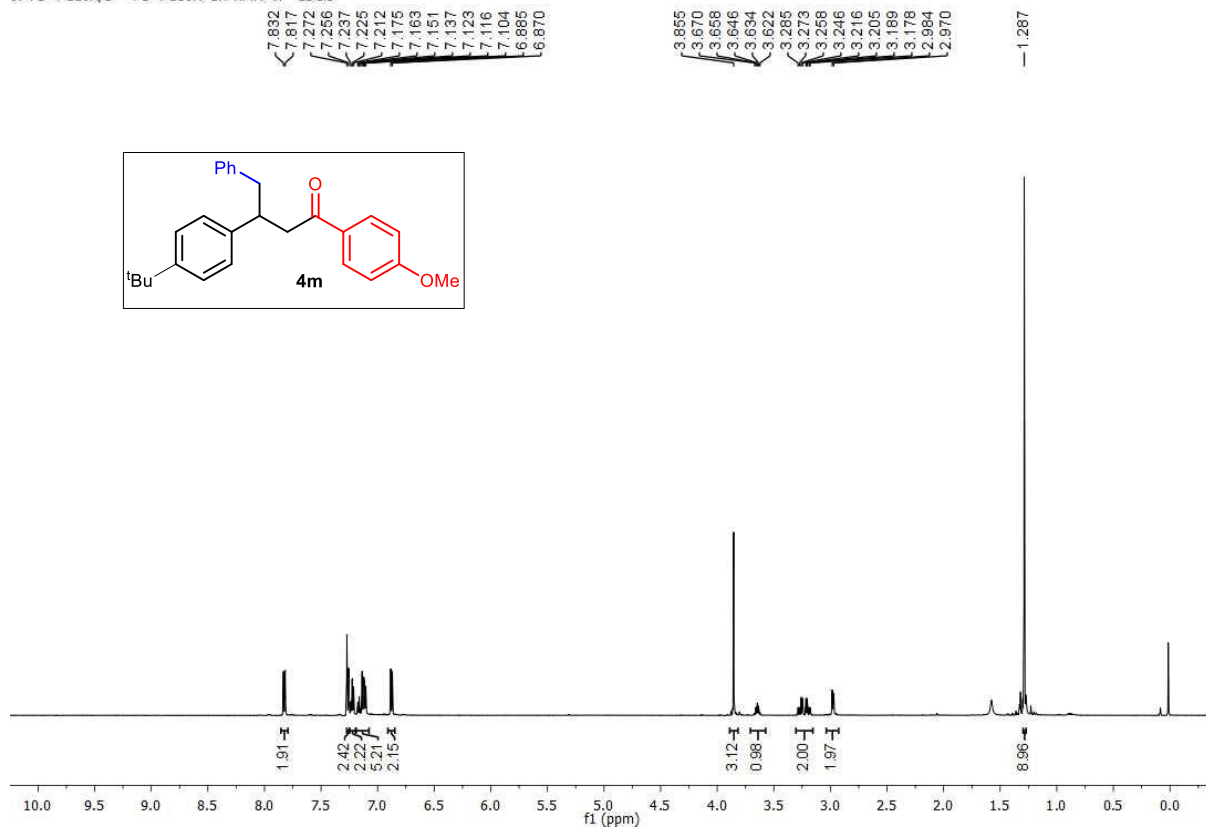
<sup>1</sup>H spectra of 4I

-197.392  
163.513  
162.658  
160.235  
143.980  
135.637  
135.604  
130.732  
130.654  
130.360  
130.331  
128.447  
127.771  
126.518  
115.026  
114.816  
113.769  
55.538  
43.905  
43.367  
42.114

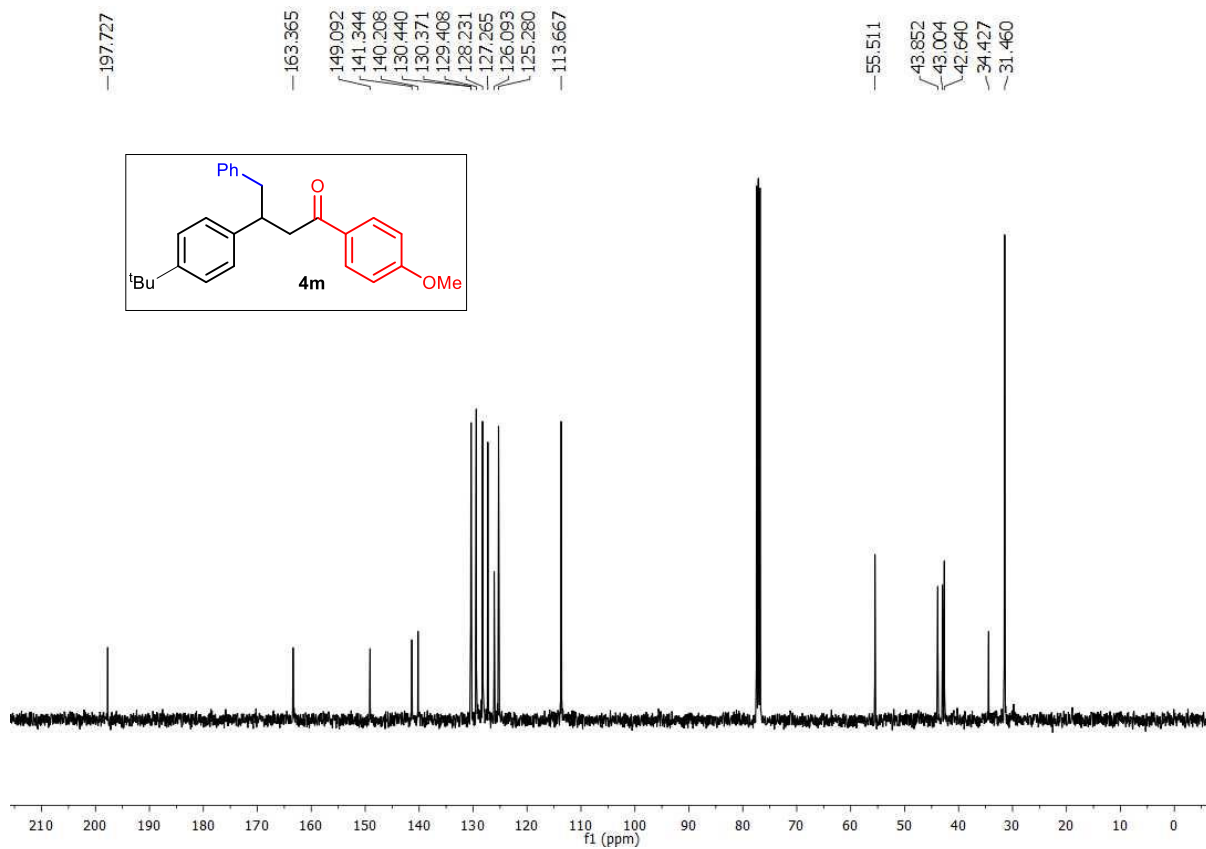


<sup>13</sup>C spectra of 4I

07-PD-4-110R/1 — PD-4-110R 1H-NMR in CDCl3

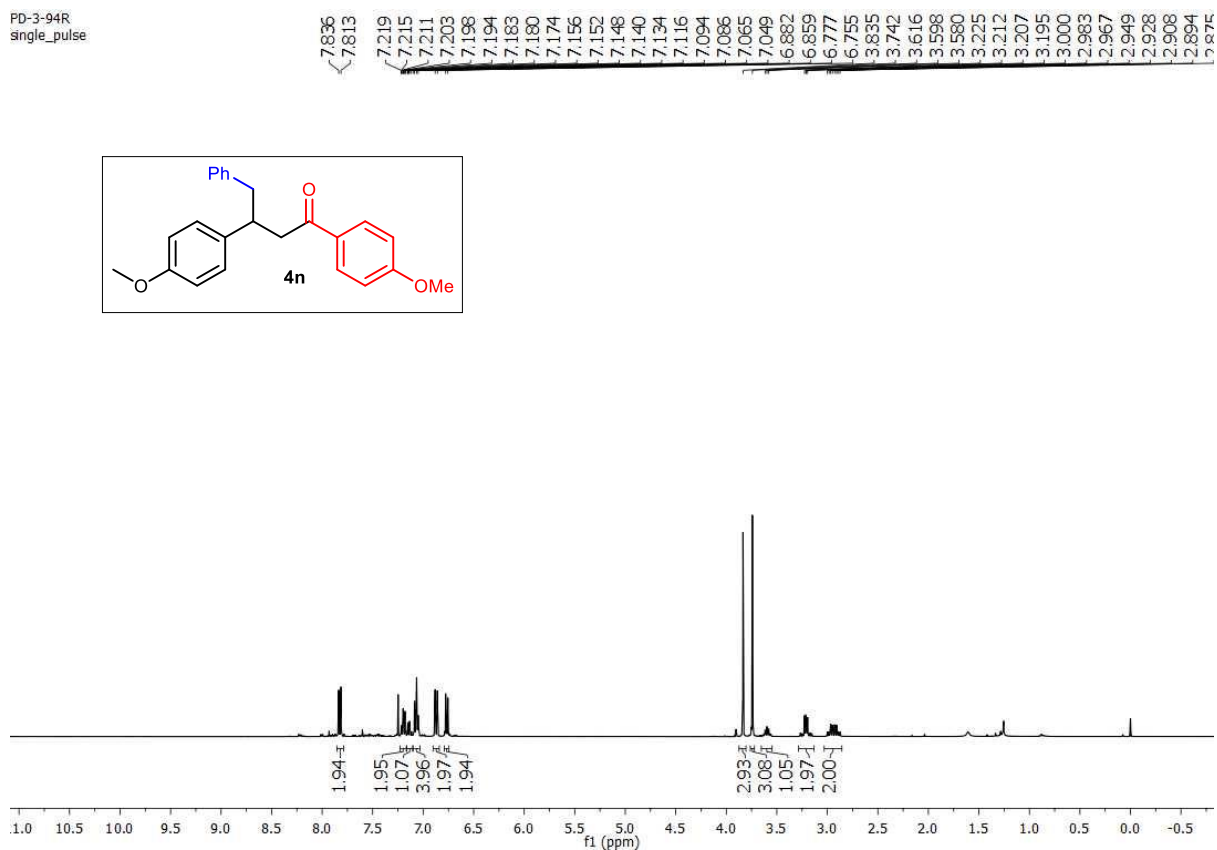


<sup>1</sup>H spectra of **4m**

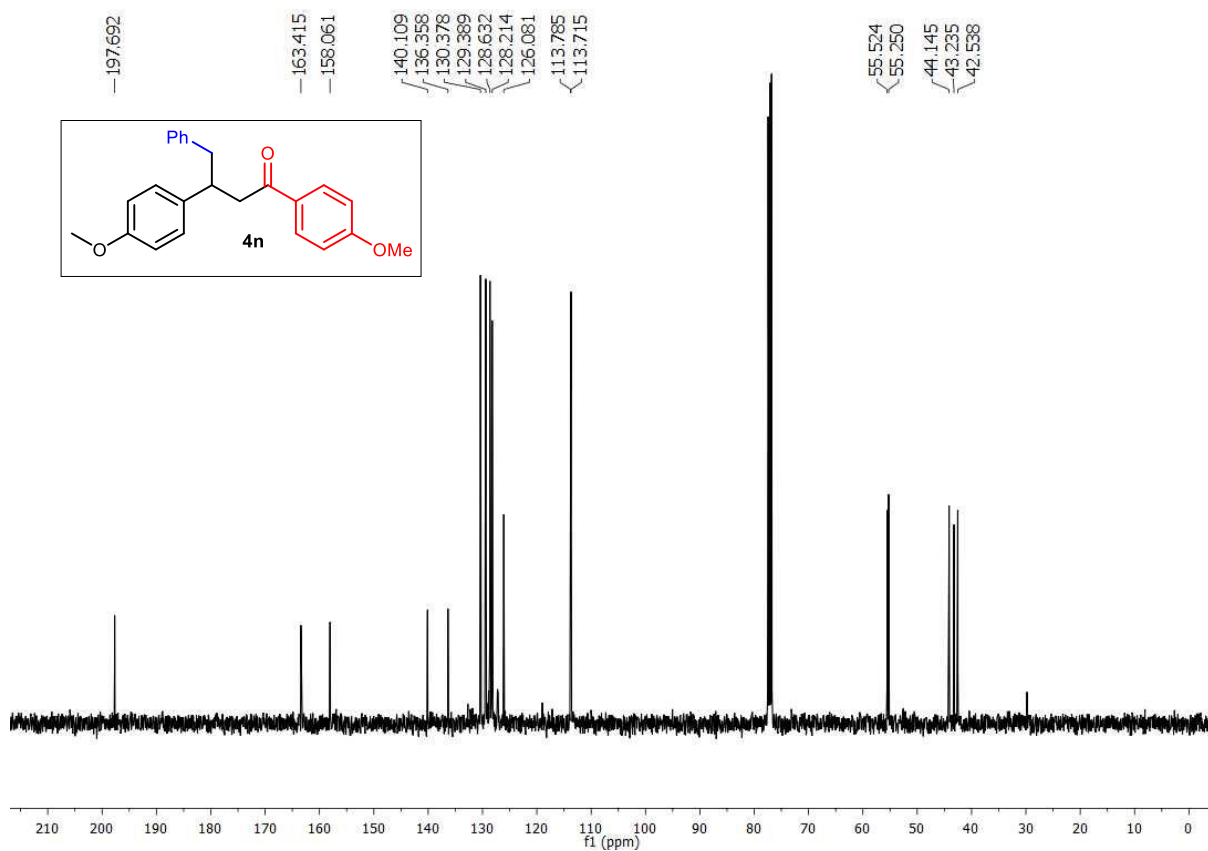


<sup>13</sup>C spectra of **4m**

PD-3-94R  
single\_pulse



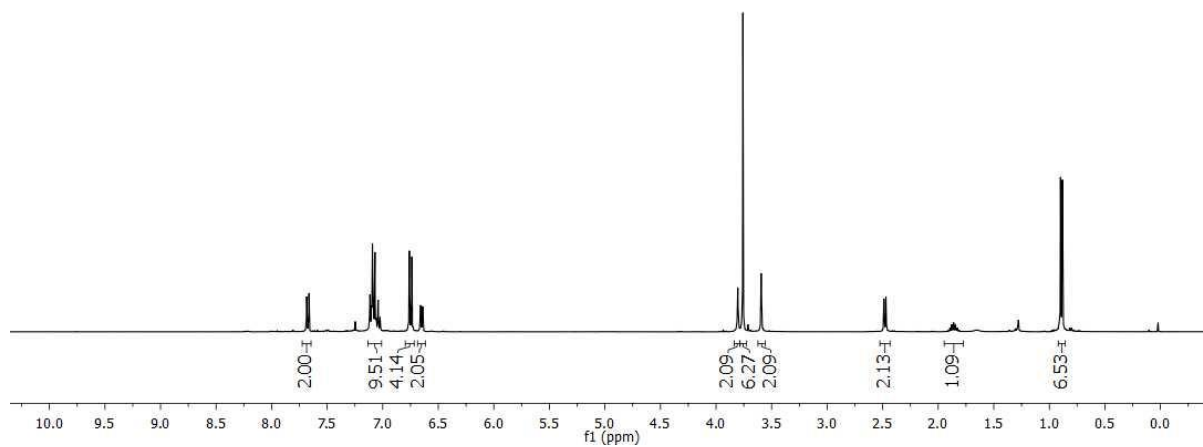
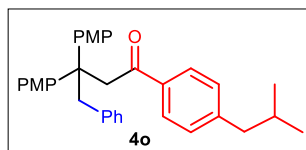
<sup>1</sup>H spectra of **4n**



<sup>13</sup>C spectra of **4n**

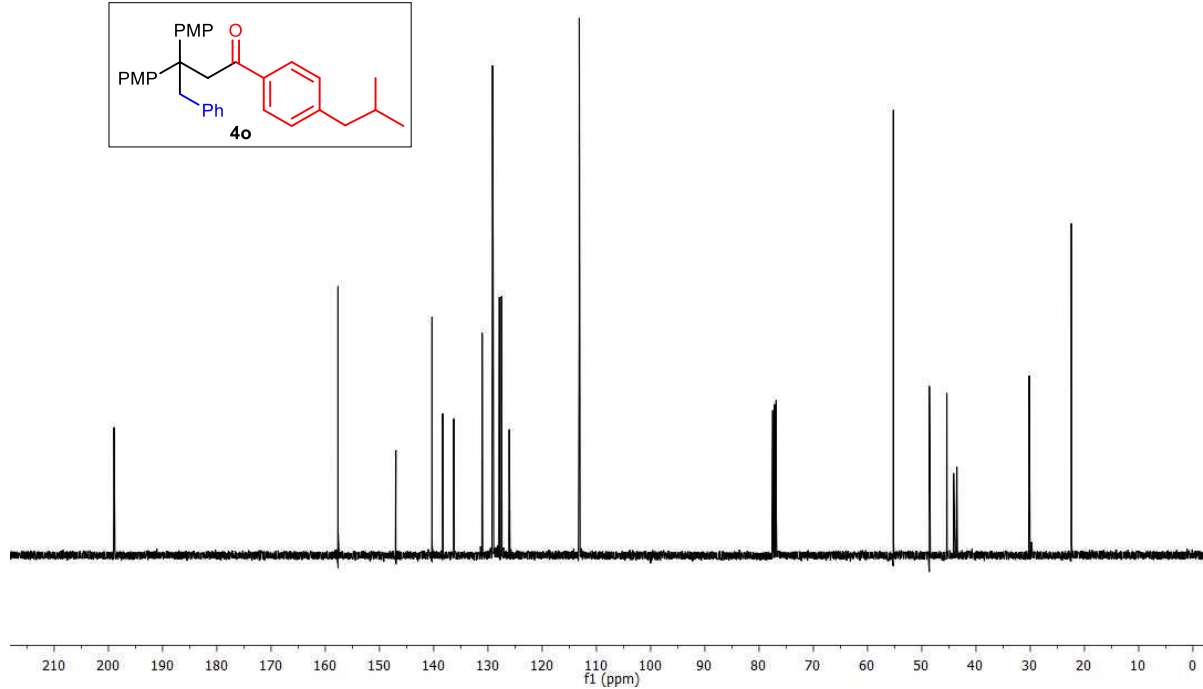
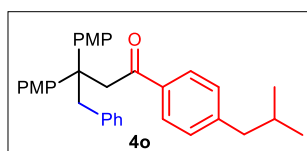
PD-4-106  
single\_pulse

7.684  
7.664  
7.115  
7.092  
7.070  
7.060  
7.041  
7.024  
6.760  
6.738  
6.658  
6.641  
3.804  
3.593  
2.489  
2.471  
1.912  
1.895  
1.878  
1.861  
1.844  
1.827  
1.810  
0.899  
0.882

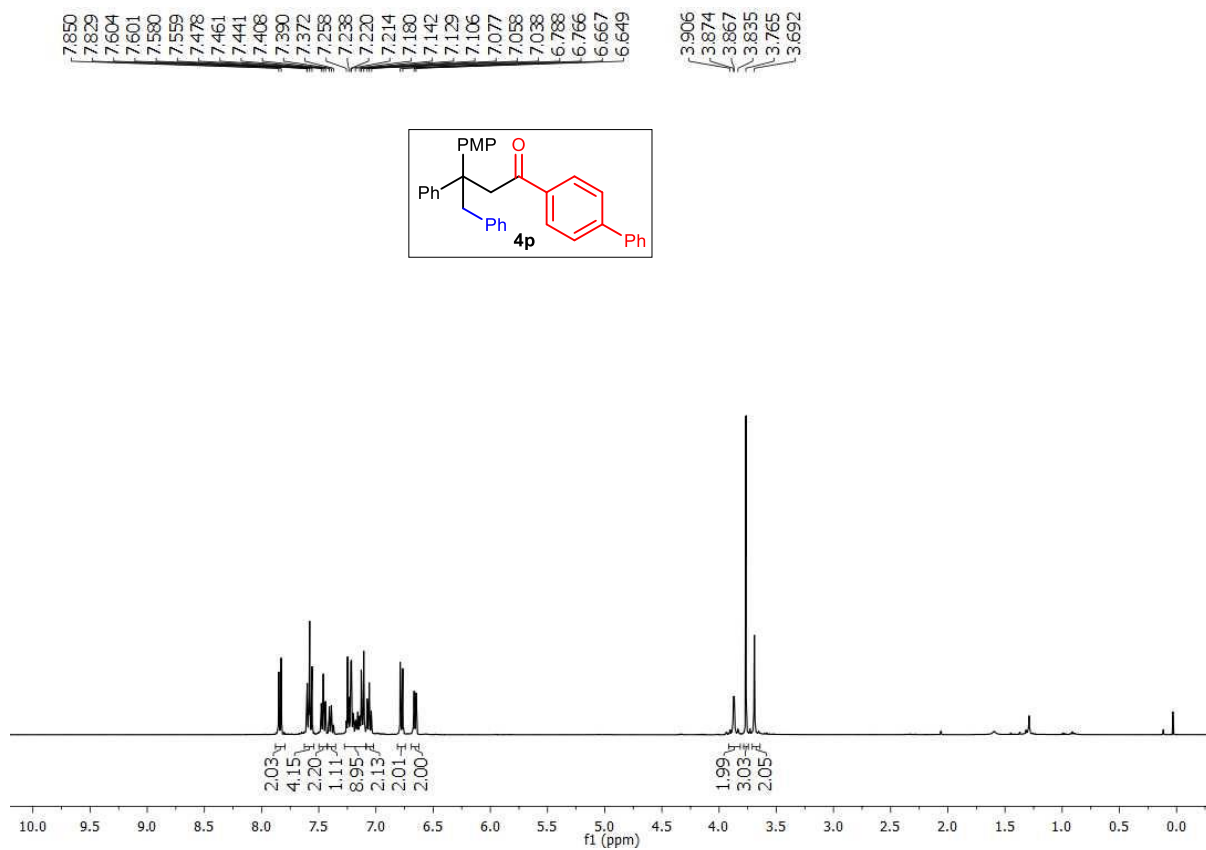


<sup>1</sup>H spectra of 4o

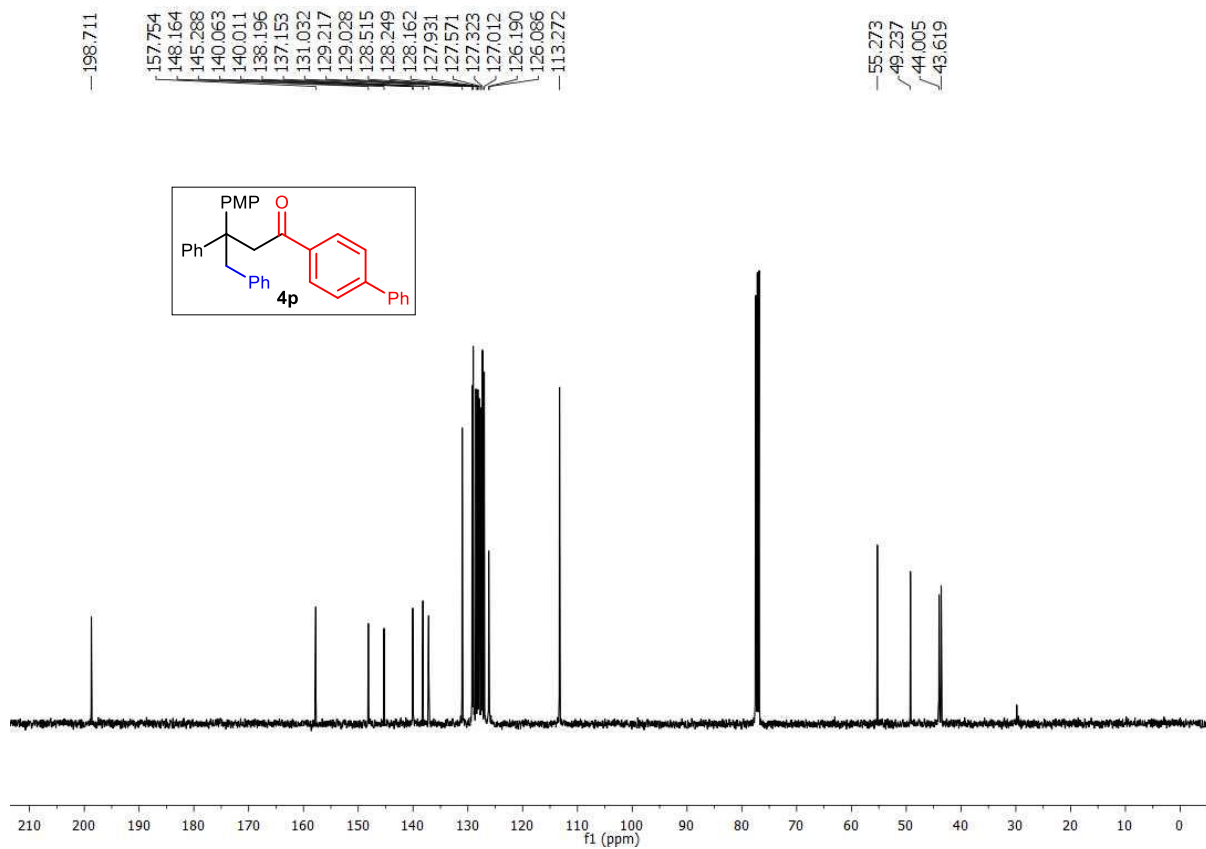
198.944  
157.649  
146.995  
140.314  
138.335  
136.294  
131.073  
129.146  
129.050  
127.892  
127.501  
126.075  
113.157  
55.231  
48.606  
45.389  
44.129  
43.540  
30.181  
22.411



<sup>13</sup>C spectra of 4o



<sup>1</sup>H spectra of **4p**

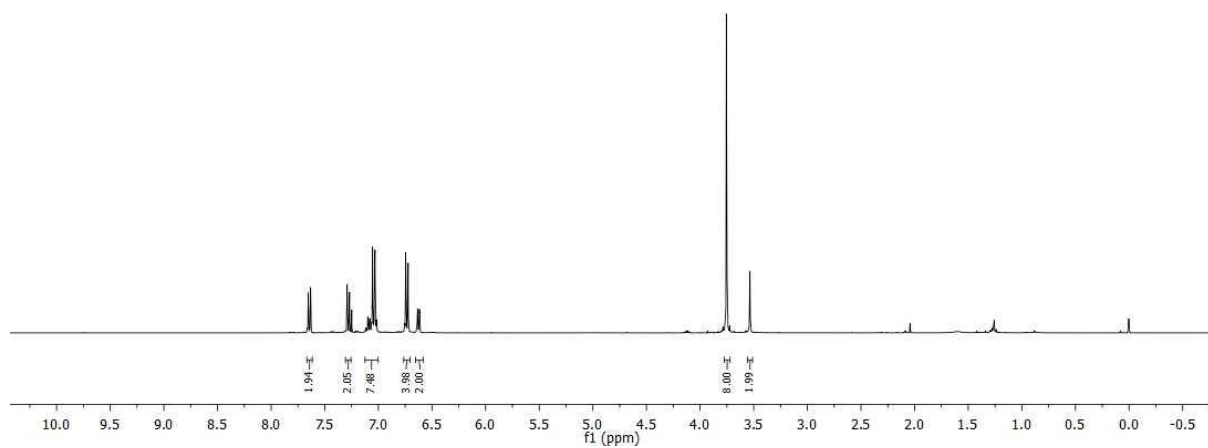
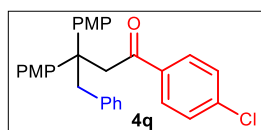


<sup>13</sup>C spectra of **4p**

PD-3-101  
single\_pulse

7.654  
7.632  
7.291  
7.269  
7.117  
7.114  
7.089  
7.084  
7.054  
7.031  
7.017  
6.746  
6.723  
6.634  
6.617

3.755  
3.748  
3.535



<sup>1</sup>H spectra of **4q**

PD-3-101  
single pulse decoupled gated NOE

157.277

157.755

139.948  
138.957  
138.095  
136.797  
131.019  
129.323  
129.099  
127.539  
126.170

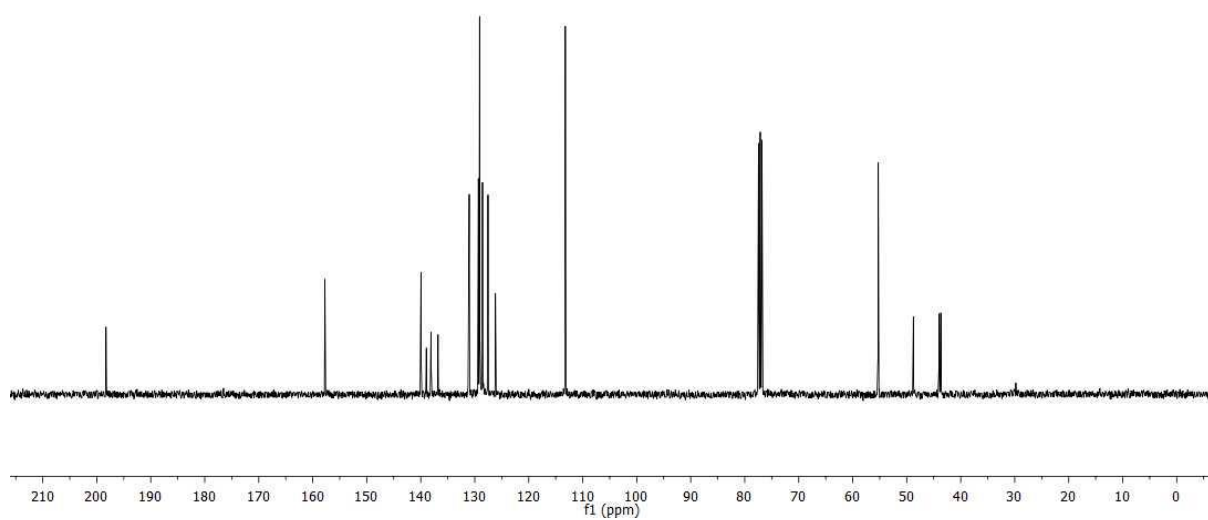
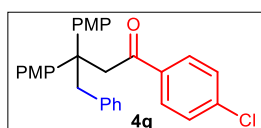
113.270

55.270

48.759

44.005

43.690



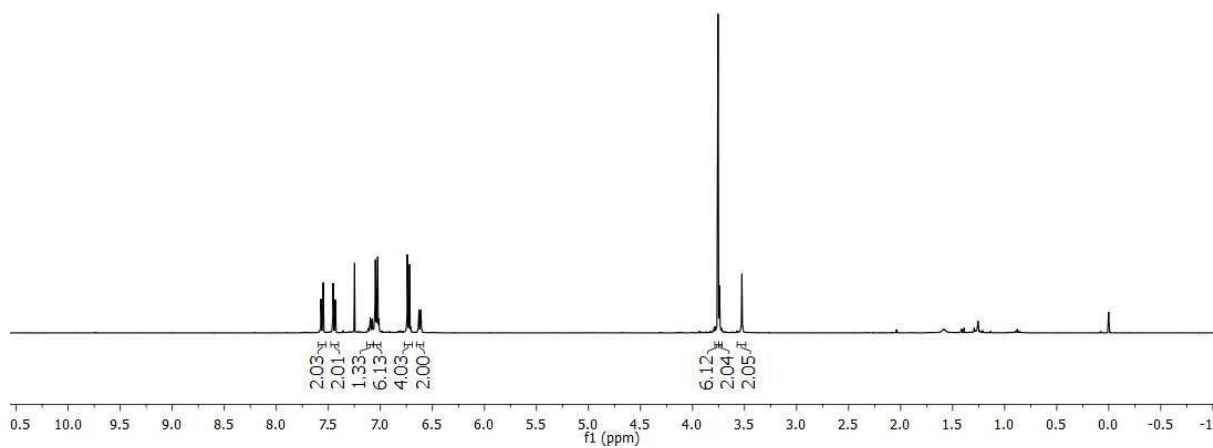
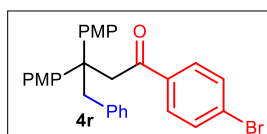
<sup>13</sup>C spectra of **4q**



PD-4-77  
single\_pulse

7.571  
7.550  
7.455  
7.433  
7.115  
7.097  
7.079  
7.048  
7.032  
7.025  
6.741  
6.719  
6.629  
6.612

3.755  
3.741  
3.525

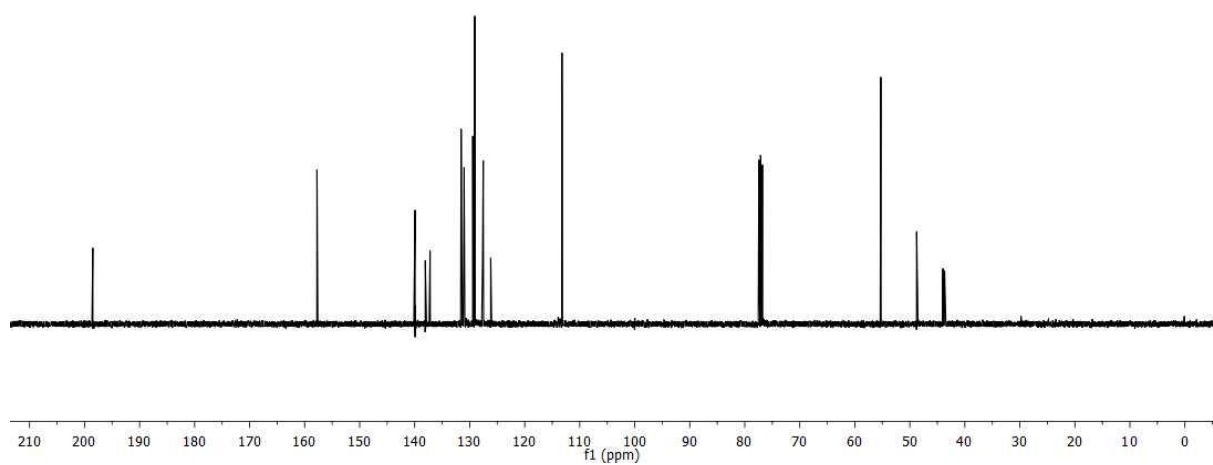
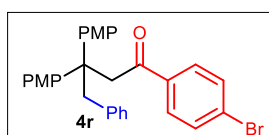


<sup>1</sup>H spectra of **4r**

-198.503

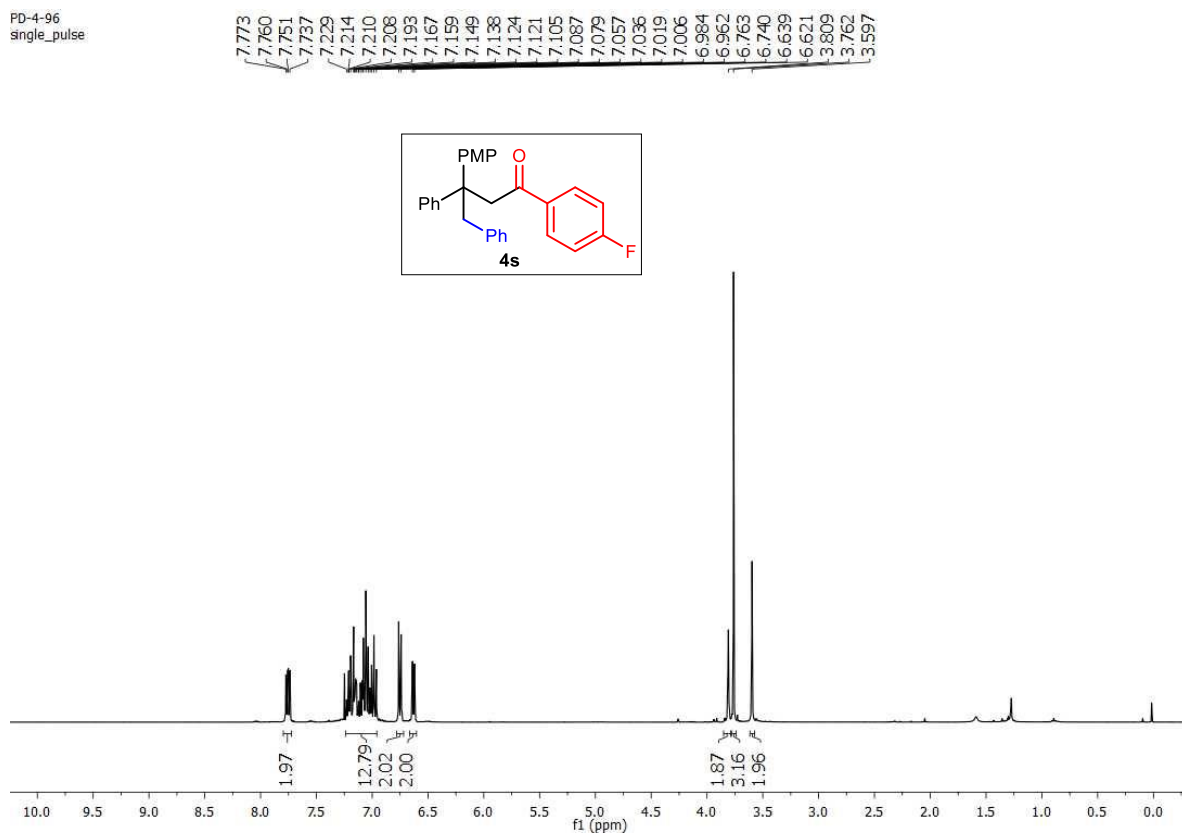
-157.759  
139.924  
138.078  
137.204  
131.556  
131.014  
129.443  
129.093  
127.664  
127.540  
126.172  
113.222

-55.274  
48.757  
43.991  
43.688



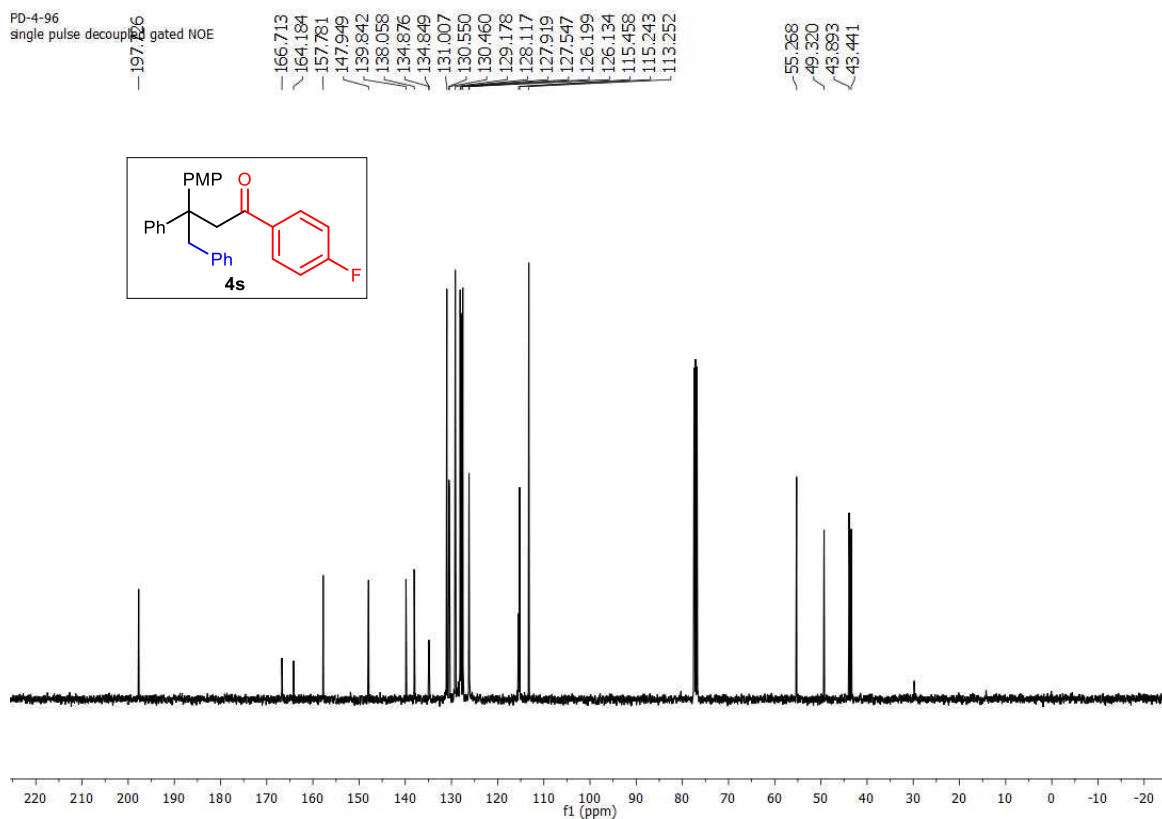
<sup>13</sup>C spectra of **4r**

PD-4-96  
single\_pulse



<sup>1</sup>H spectra of 4s

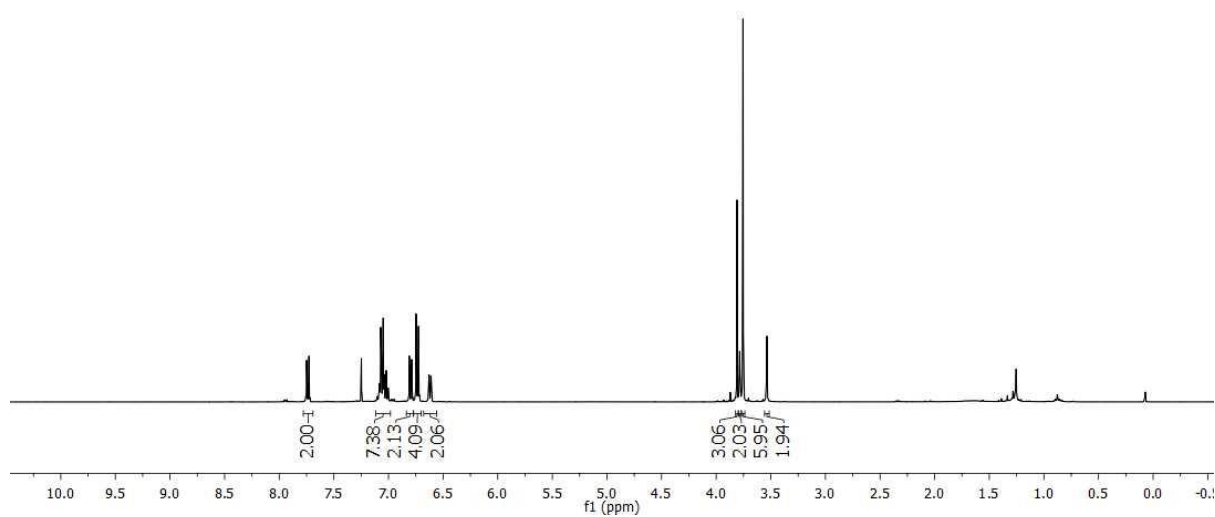
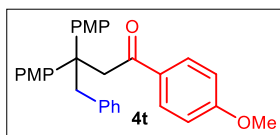
PD-4-96  
single pulse decoupled gated NOE



<sup>13</sup>C spectra of 4s

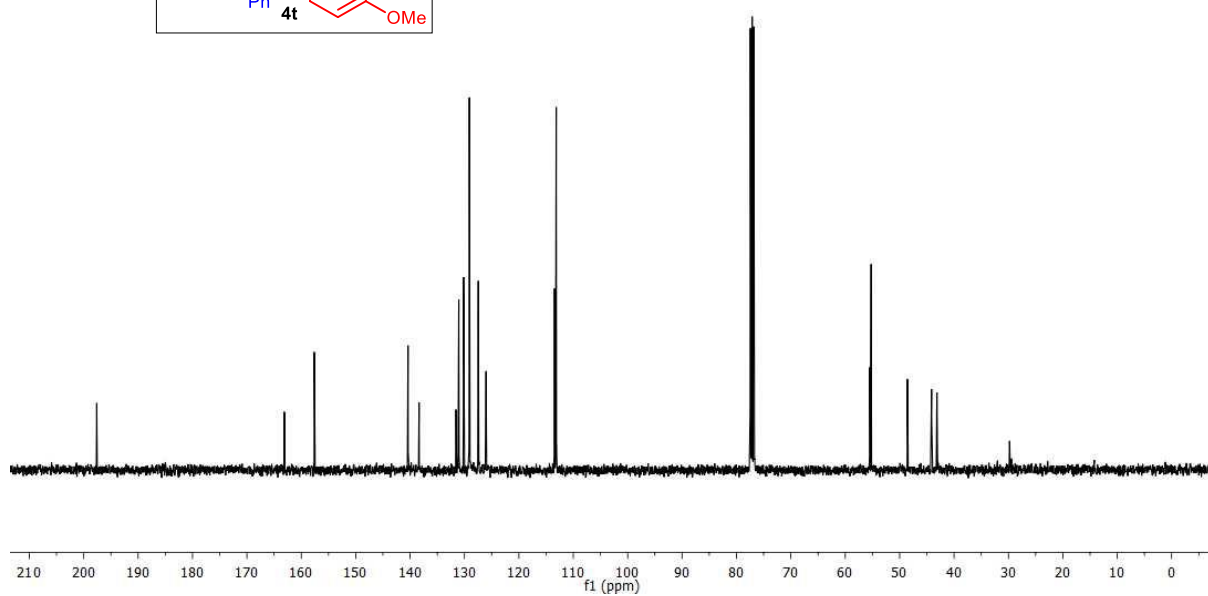
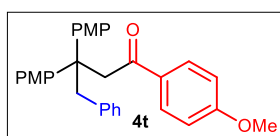
PD-4-175  
single\_pulse

7.750  
7.728  
7.102  
7.084  
7.066  
7.049  
7.039  
7.020  
7.003  
6.809  
6.787  
6.747  
6.725  
6.631  
6.611  
3.809  
3.785  
3.754  
3.536



<sup>1</sup>H spectra of **4t**

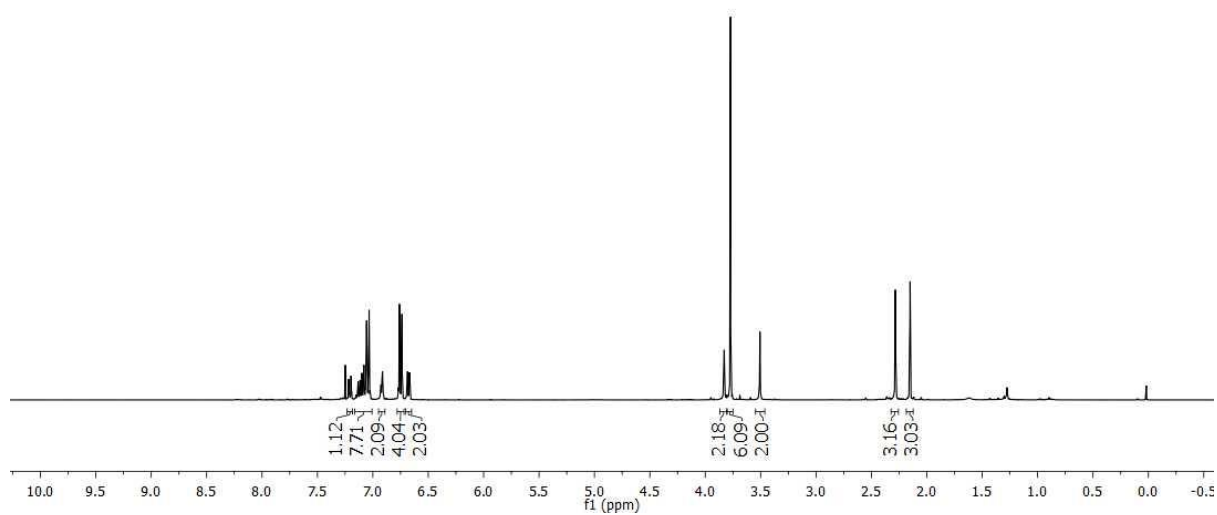
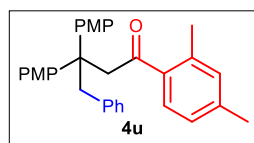
-197.606  
-163.121  
-157.607  
140.380  
138.348  
131.553  
131.051  
130.168  
129.103  
127.463  
126.035  
113.449  
113.140  
55.484  
55.244  
48.583  
44.137  
43.146



<sup>13</sup>C spectra of **4t**

PD-3-100  
single\_pulse

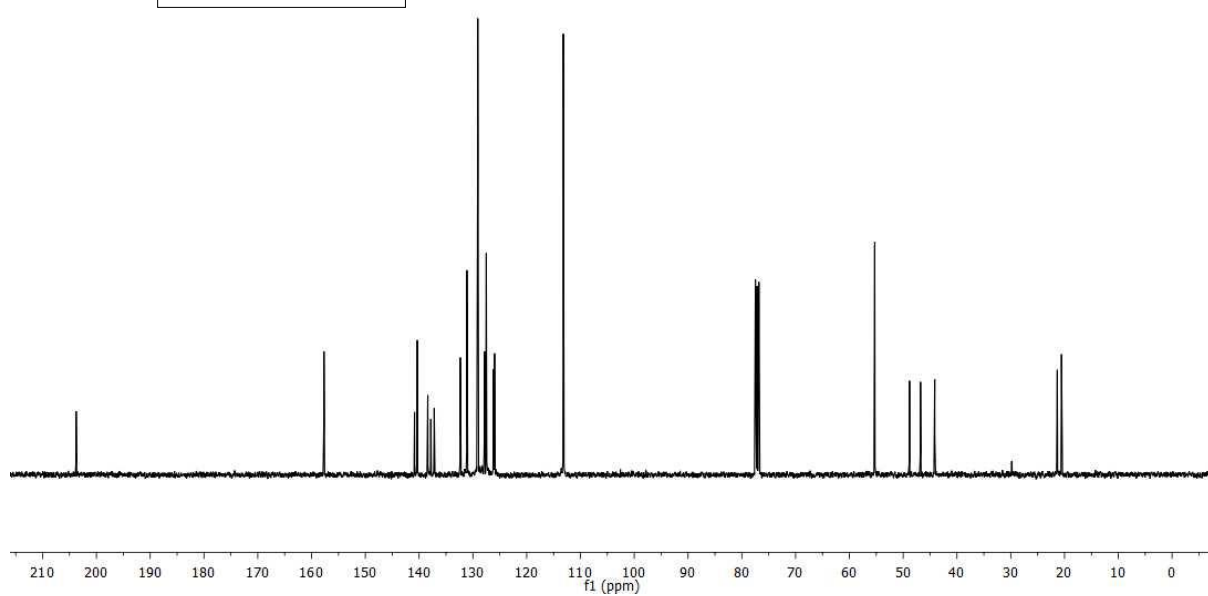
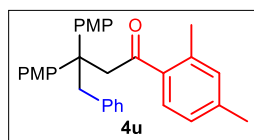
7.218  
7.197  
7.133  
7.111  
7.088  
7.080  
7.056  
7.034  
6.927  
6.912  
6.760  
6.737  
6.687  
6.670  
3.829  
3.772  
3.506  
2.284  
2.151



<sup>1</sup>H spectra of **4u**

-203.764

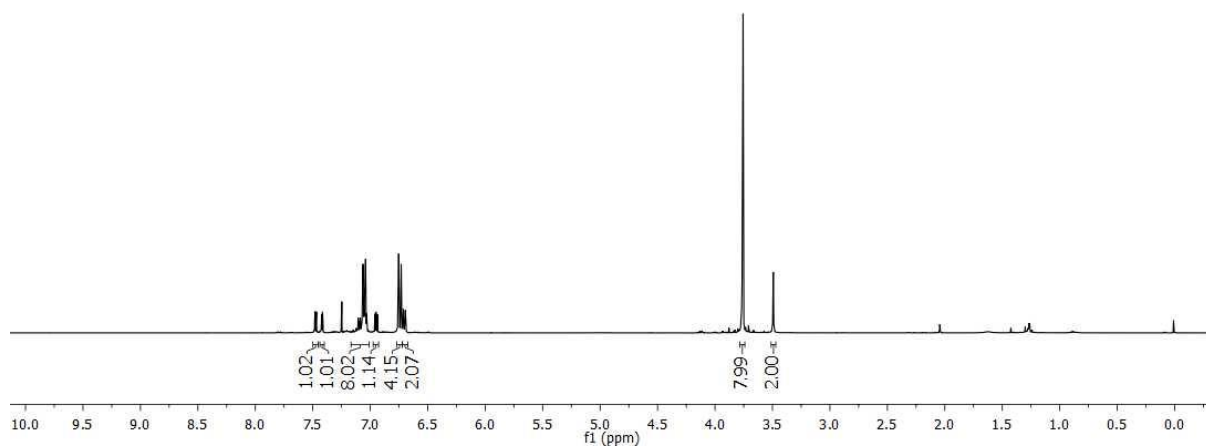
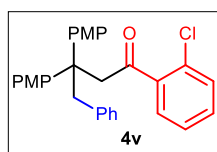
157.690  
140.847  
140.330  
138.387  
137.819  
137.190  
132.353  
131.118  
129.093  
127.510  
126.144  
125.980  
113.178  
55.292  
48.824  
46.778  
44.136  
21.345  
20.561



<sup>13</sup>C spectra of **4u**

PD-4-78  
single\_pulse

7.483  
7.481  
7.471  
7.468  
7.425  
7.422  
7.415  
7.412  
7.105  
7.087  
7.068  
7.063  
7.040  
7.032  
6.959  
6.950  
6.947  
6.937  
6.753  
6.731  
6.714  
6.711  
6.694  
6.691

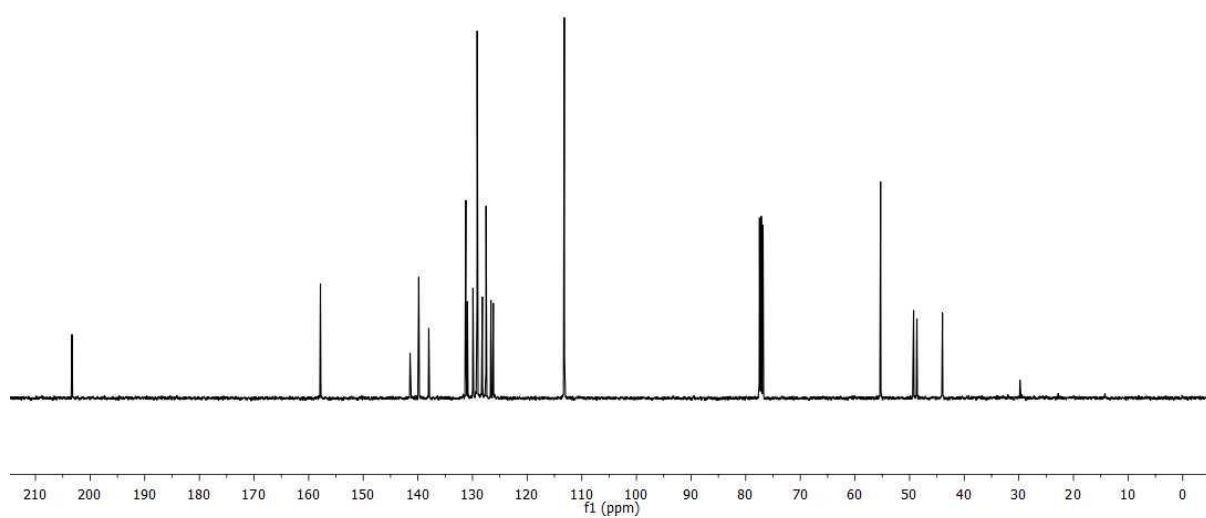
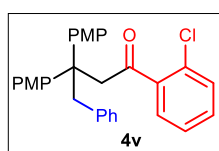


$^1\text{H}$  spectra of **4v**

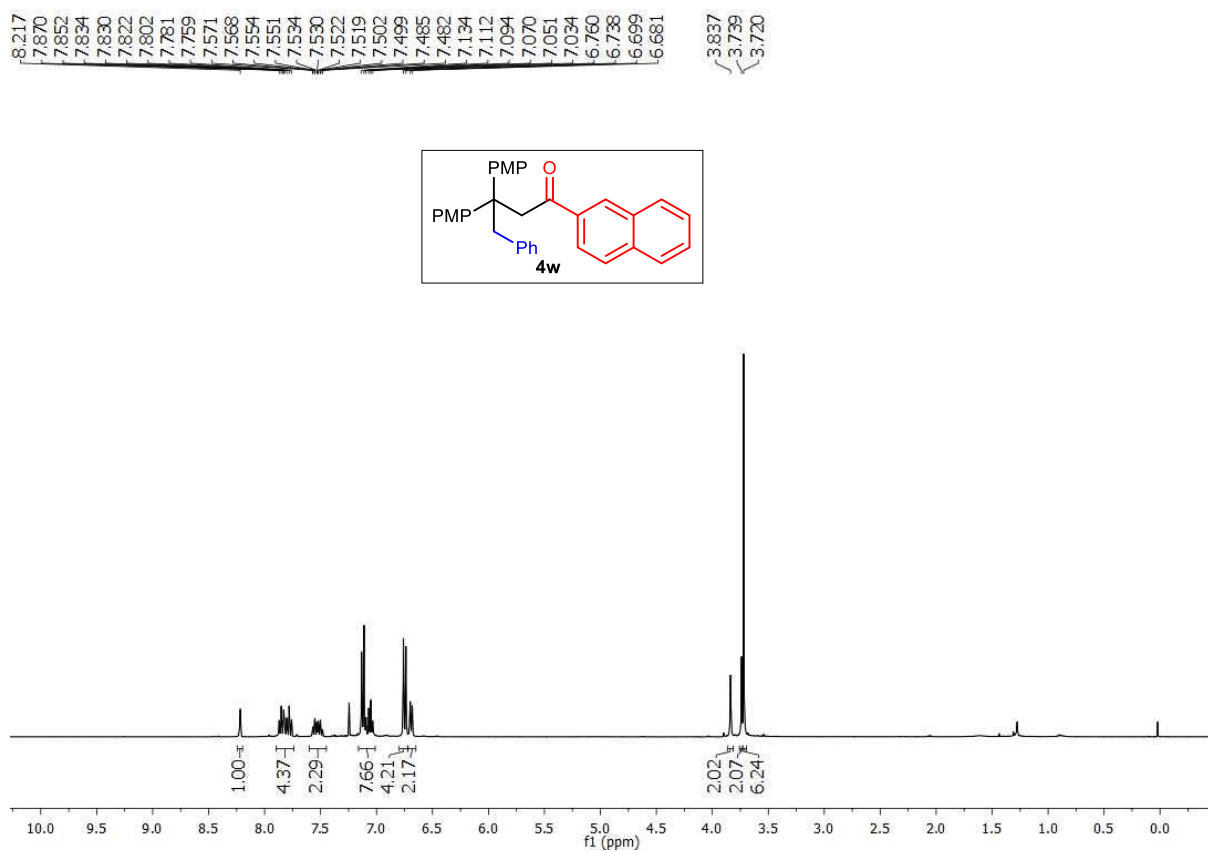
-203.316

157.841  
141.432  
139.833  
138.004  
131.228  
130.928  
129.956  
129.915  
129.152  
128.204  
127.522  
126.627  
126.197  
113.207

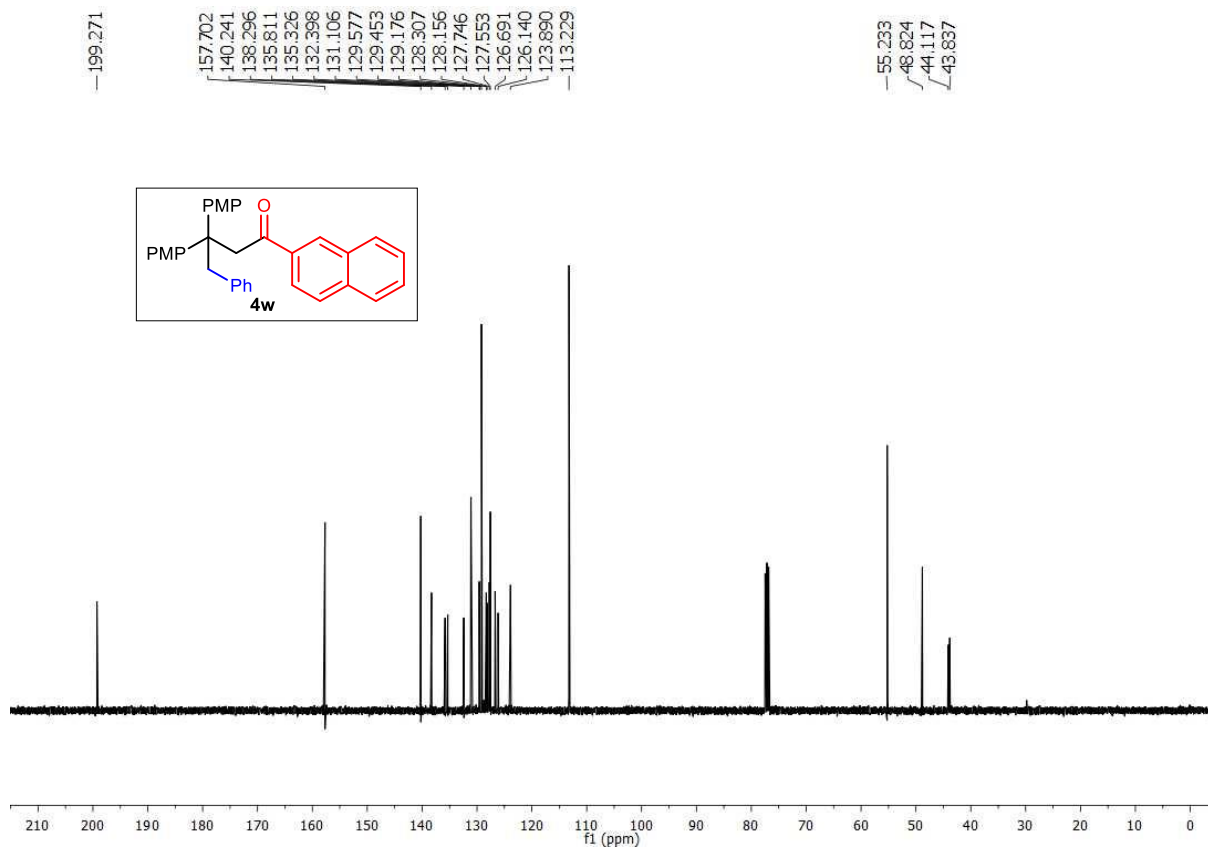
55.307  
49.262  
48.660  
43.984



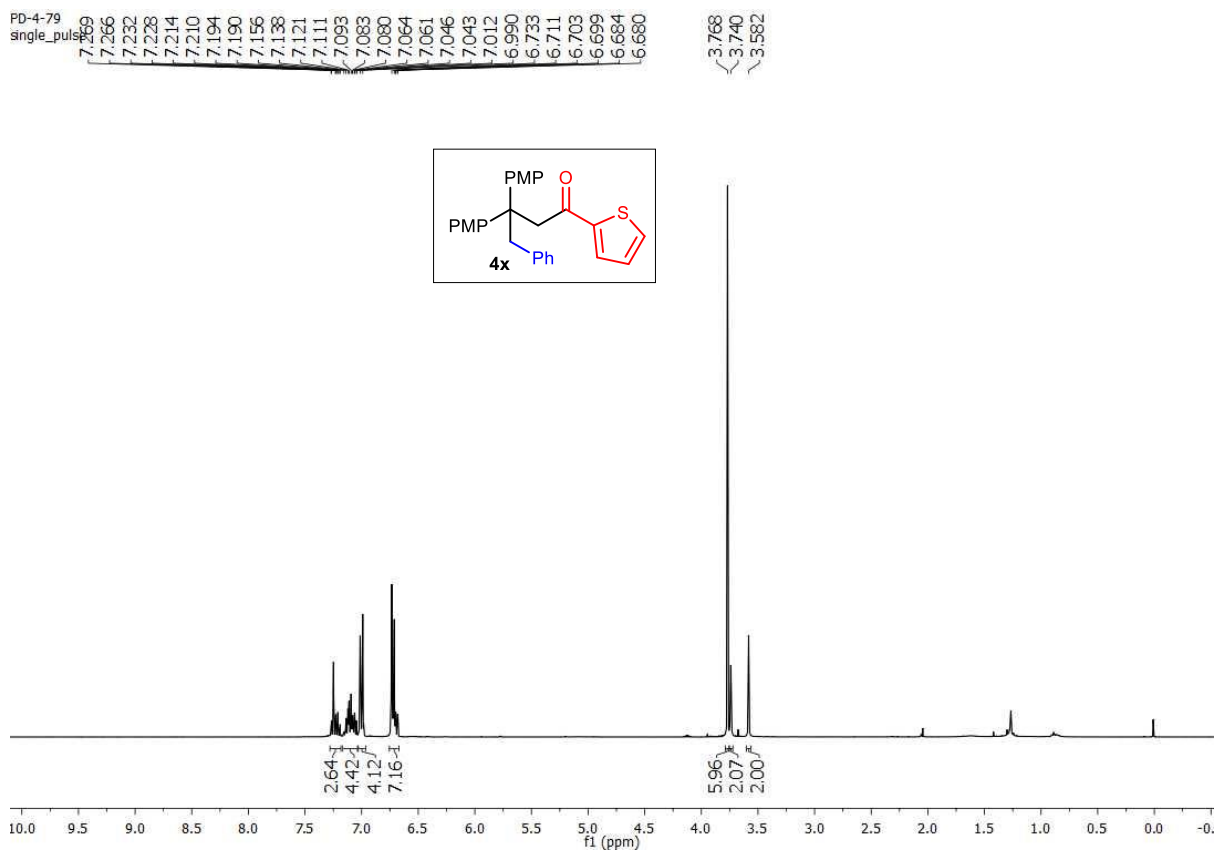
$^{13}\text{C}$  spectra of **4v**



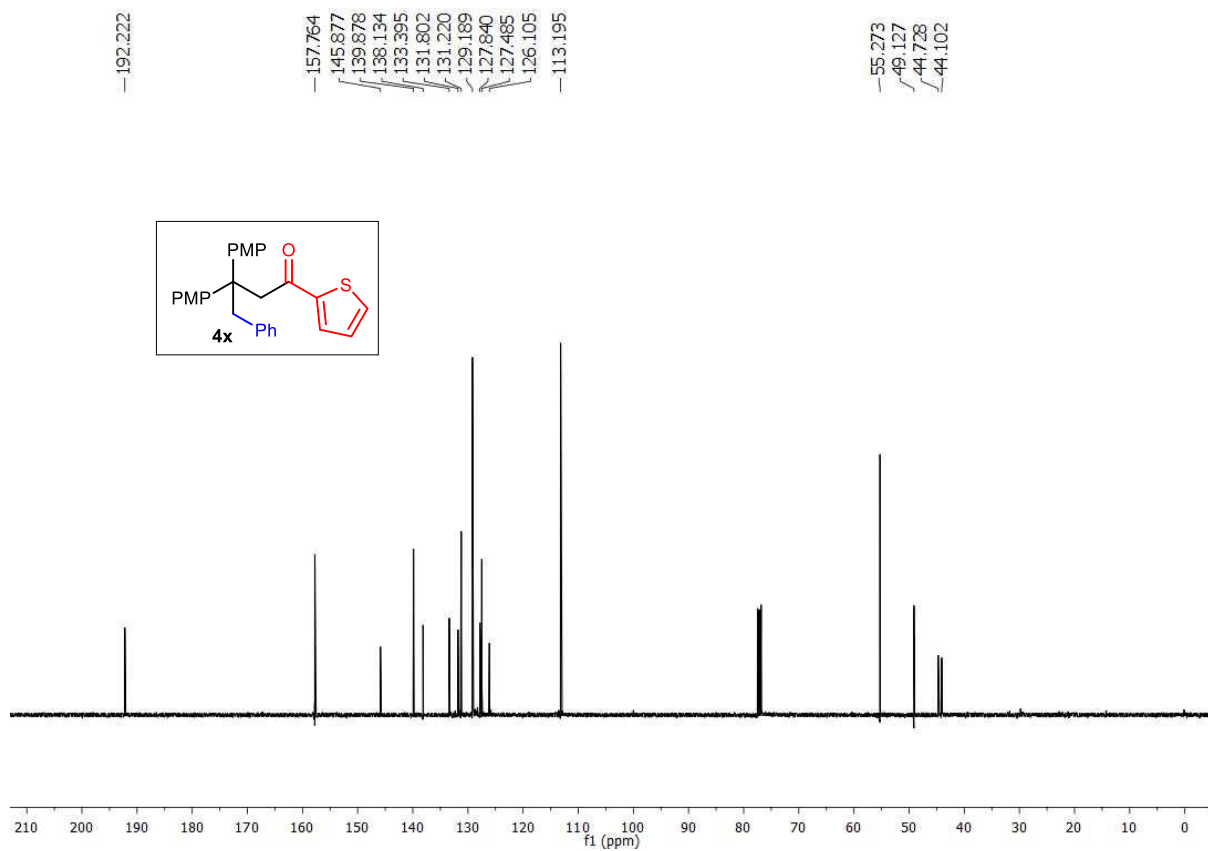
$^1\text{H}$  spectra of **4w**



$^{13}\text{C}$  spectra of **4w**

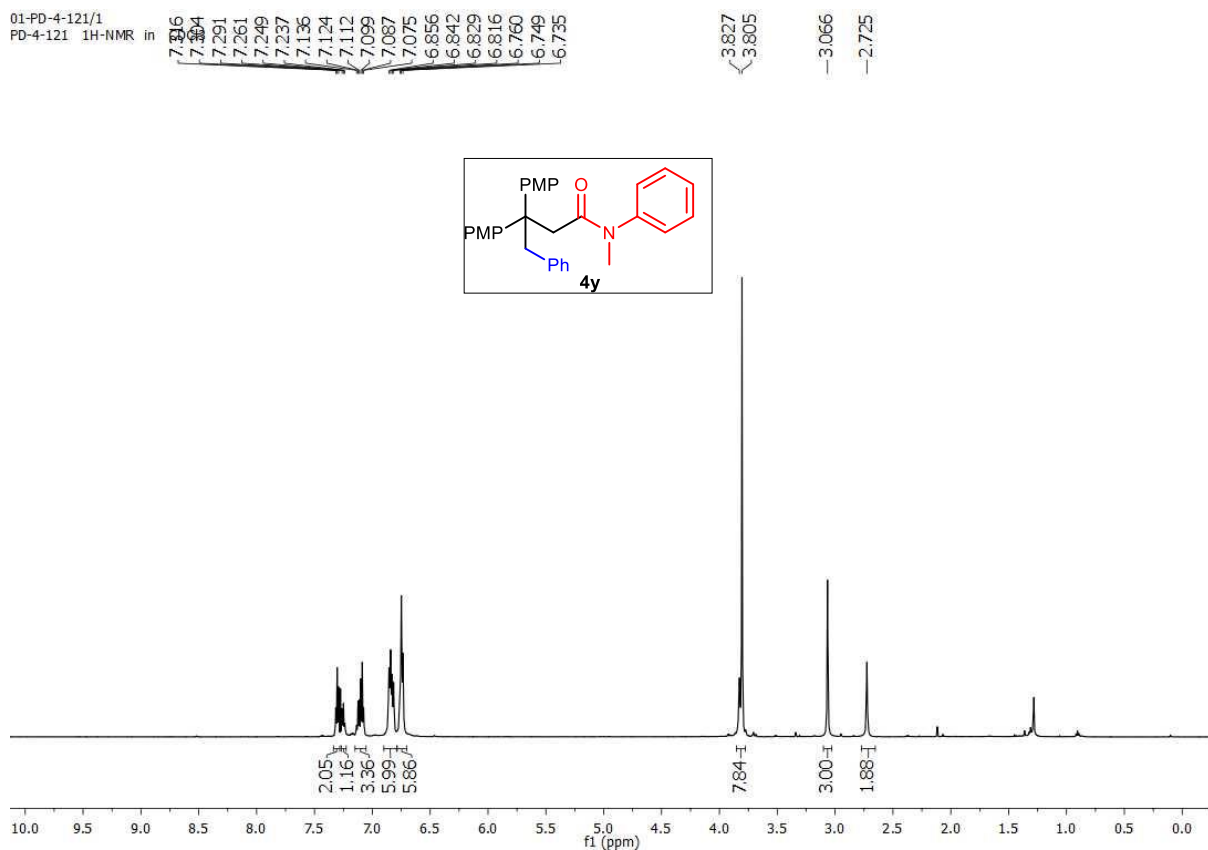


<sup>1</sup>H spectra of **4x**



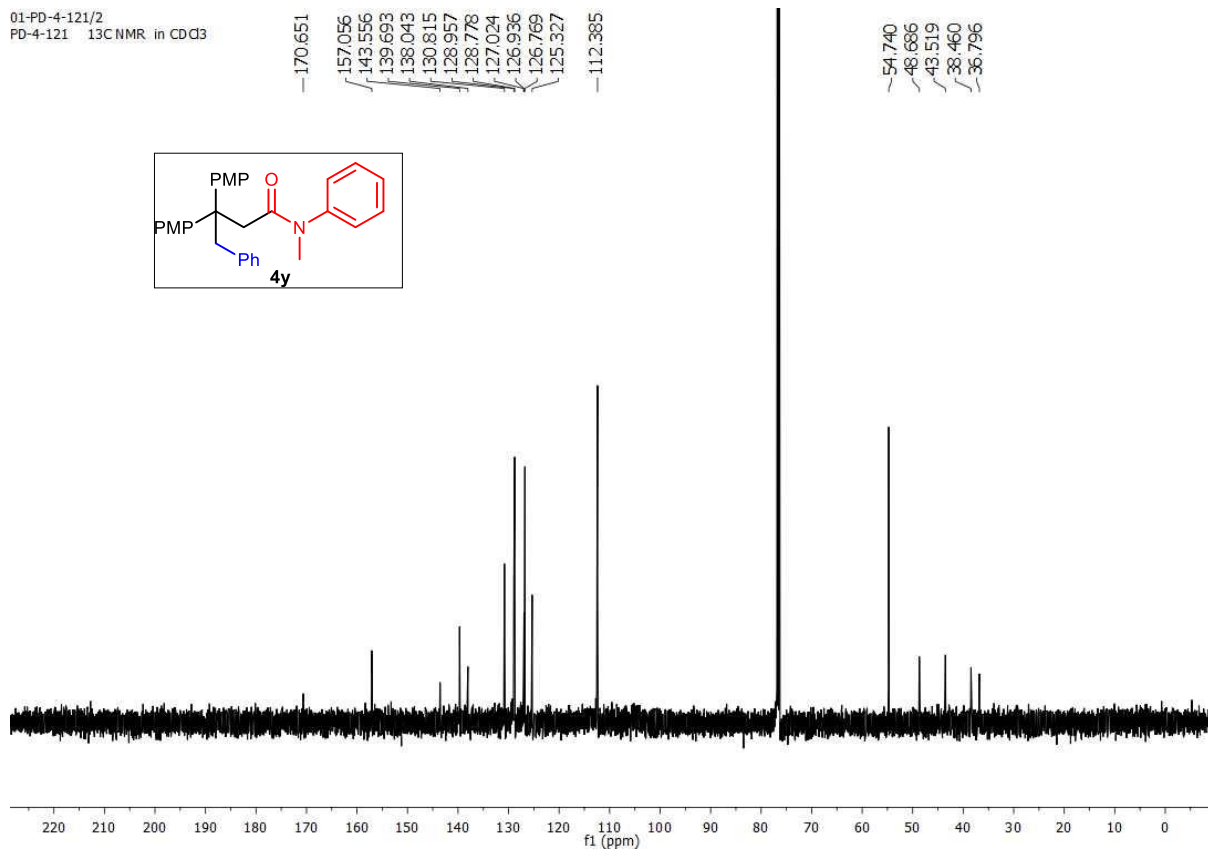
<sup>13</sup>C spectra of **4x**

01-PD-4-121/1  
PD-4-121 1H-NMR in



<sup>1</sup>H spectra of **4y**

01-PD-4-121/2  
PD-4-121 13C-NMR in CDCl<sub>3</sub>



<sup>13</sup>C spectra of **4y**



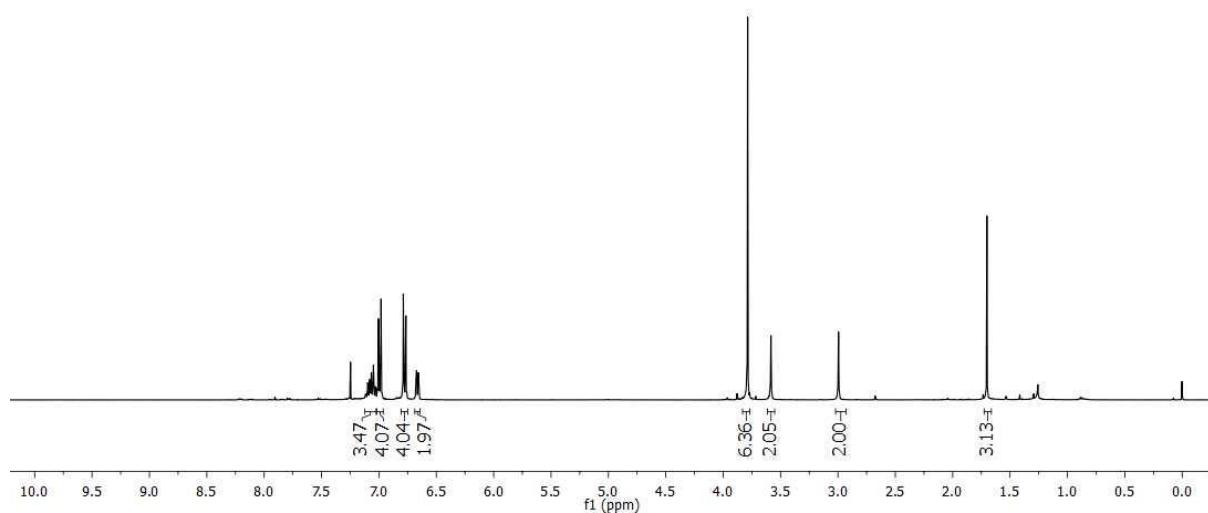
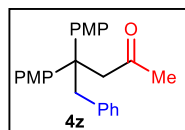
PD-4-227  
single\_pulse

7.119  
7.097  
7.080  
7.066  
7.048  
7.031  
7.036  
7.004  
6.982  
6.787  
6.765  
6.675  
6.672  
6.655  
6.651

3.787  
3.584

2.995

1.701



<sup>1</sup>H spectra of **4z**

PD-4-227  
single\_pulse  
decoupled gated NOE

208.810

157.828

139.861

138.027

131.200

129.092

127.406

126.037

113.259

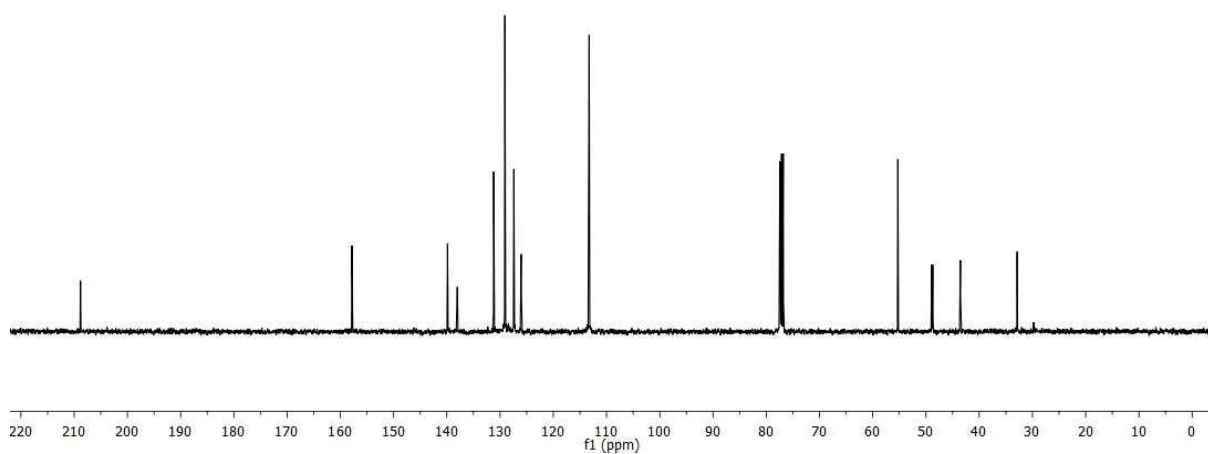
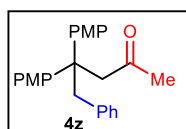
55.279

48.939

48.726

43.529

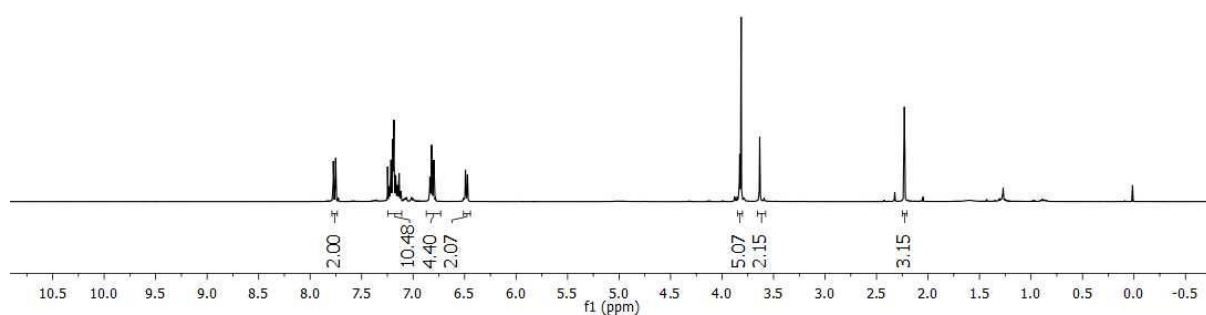
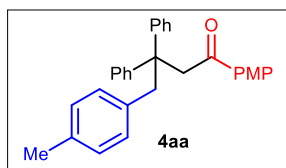
32.876



<sup>13</sup>C spectra of **4z**

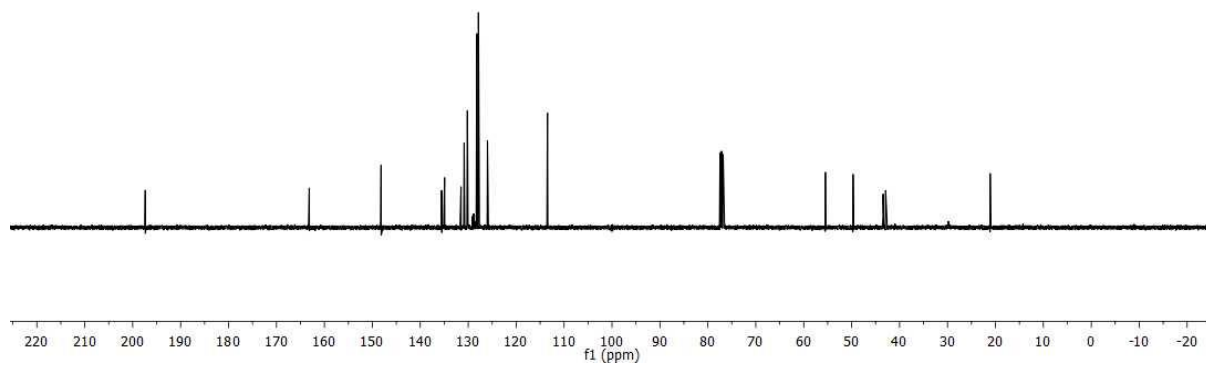
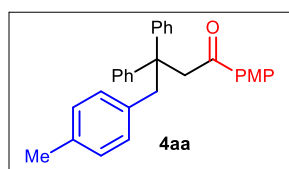
PD-4-139  
single\_pulse

7.775  
7.752  
7.234  
7.215  
7.198  
7.186  
7.169  
7.157  
7.136  
7.119  
7.064  
7.021  
6.837  
6.820  
6.798  
6.492  
6.472  
3.828  
3.815  
3.634  
-2.230



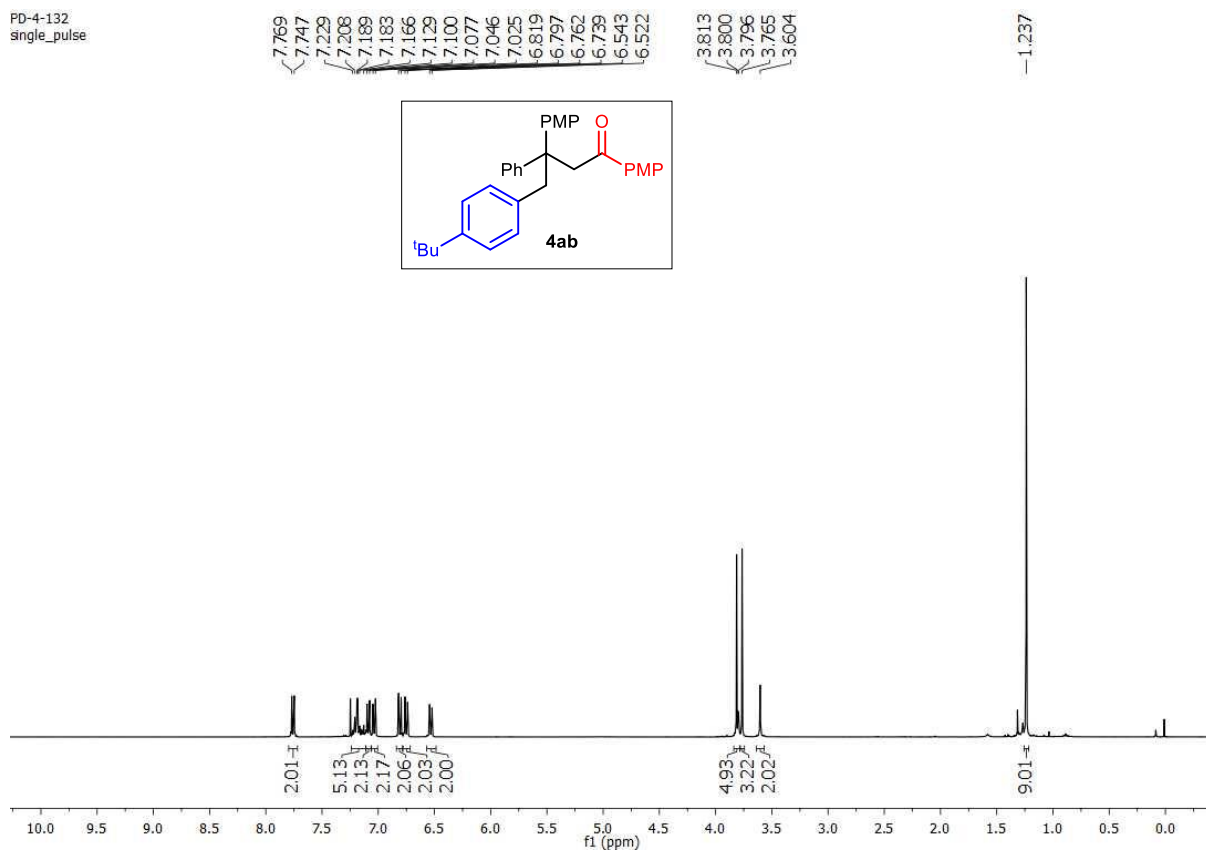
<sup>1</sup>H spectra of 4aa

-197.367  
-163.153  
148.173  
135.540  
134.924  
131.497  
130.843  
130.171  
128.230  
128.219  
127.861  
125.980  
113.463  
-55.487  
-49.710  
-43.433  
-42.908  
-21.099

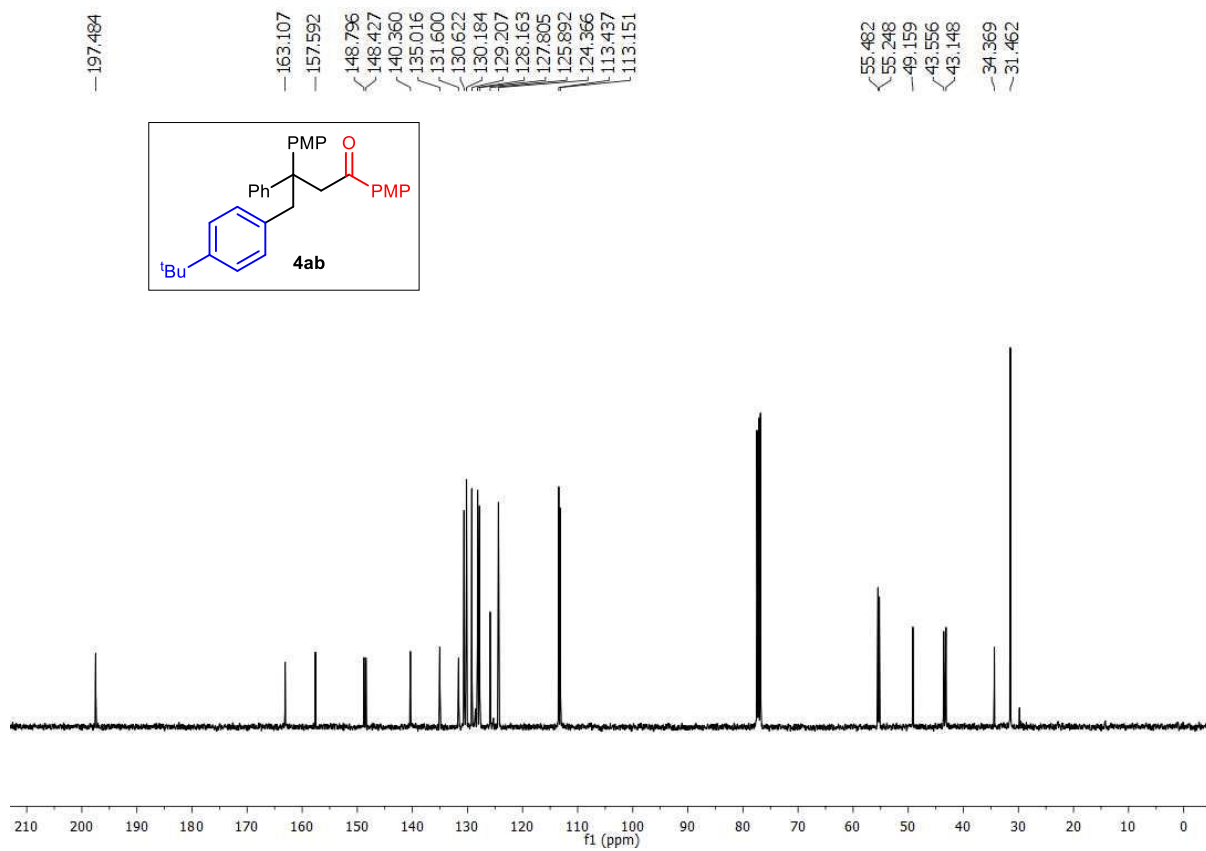


<sup>13</sup>C spectra of 4aa

PD-4-132  
single\_pulse



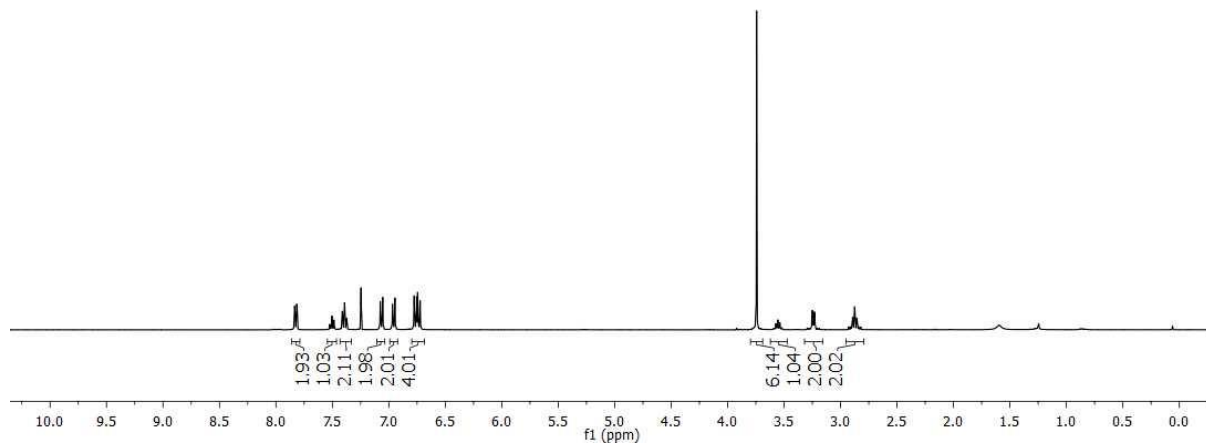
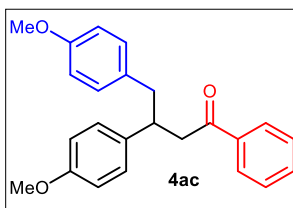
<sup>1</sup>H spectra of **4ab**



<sup>13</sup>C spectra of **4ab**

SDD-PD-2.1  
single\_pulse

7.833 7.815 7.812 7.523 7.504 7.486 7.411 7.392 7.374 7.076 7.055 6.968 6.946 6.776 6.754 6.745 6.723 3.743 3.591 3.573 3.555 3.537 3.519 3.294 3.276 3.253 3.246 3.235 3.229 3.205 3.188 2.927 2.909 2.893 2.875 2.855 2.840 2.821

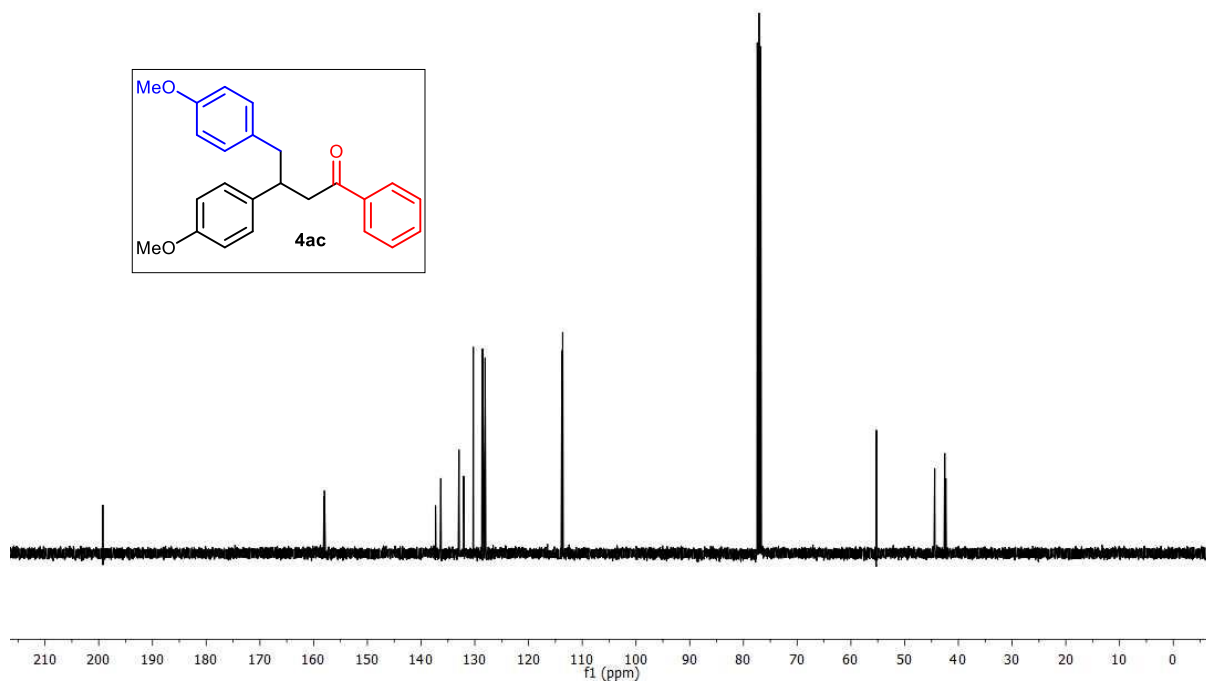
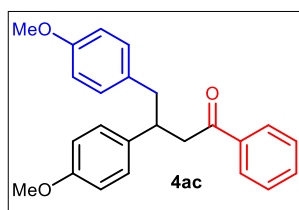


<sup>1</sup>H spectra of 4ac

—199.239

158.066 157.979 137.316 136.327 132.949 132.063 130.290 128.641 128.567 128.083 113.790

55.279 44.406 42.533 42.338

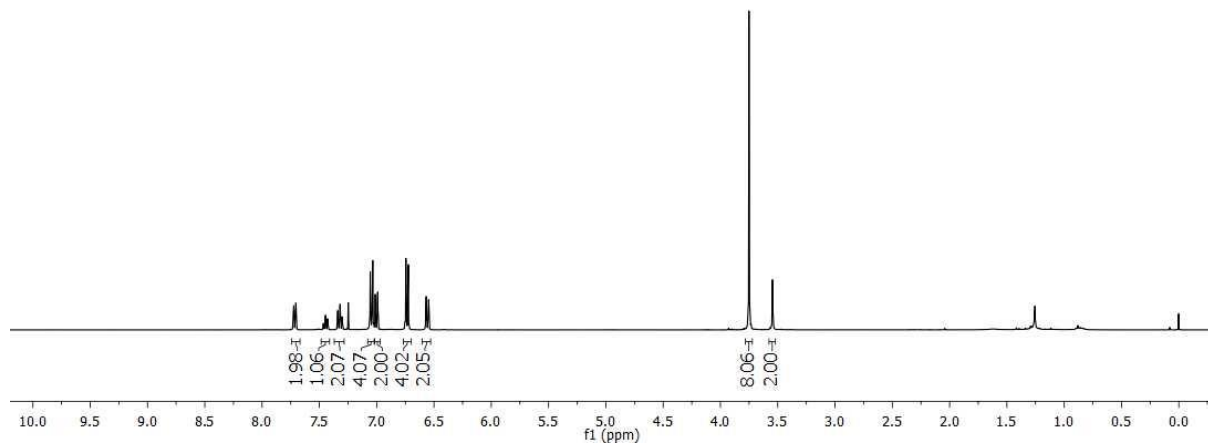
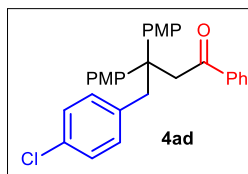


<sup>13</sup>C spectra of 4ac

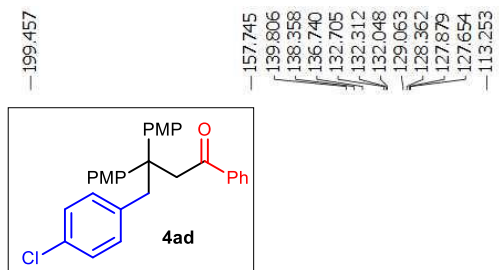
PD-4-143  
single\_pulse

7.726  
7.723  
7.705  
7.702  
7.465  
7.447  
7.428  
7.340  
7.319  
7.301  
7.056  
7.034  
7.012  
6.991  
6.745  
6.723  
6.567  
6.546

3.751  
3.545



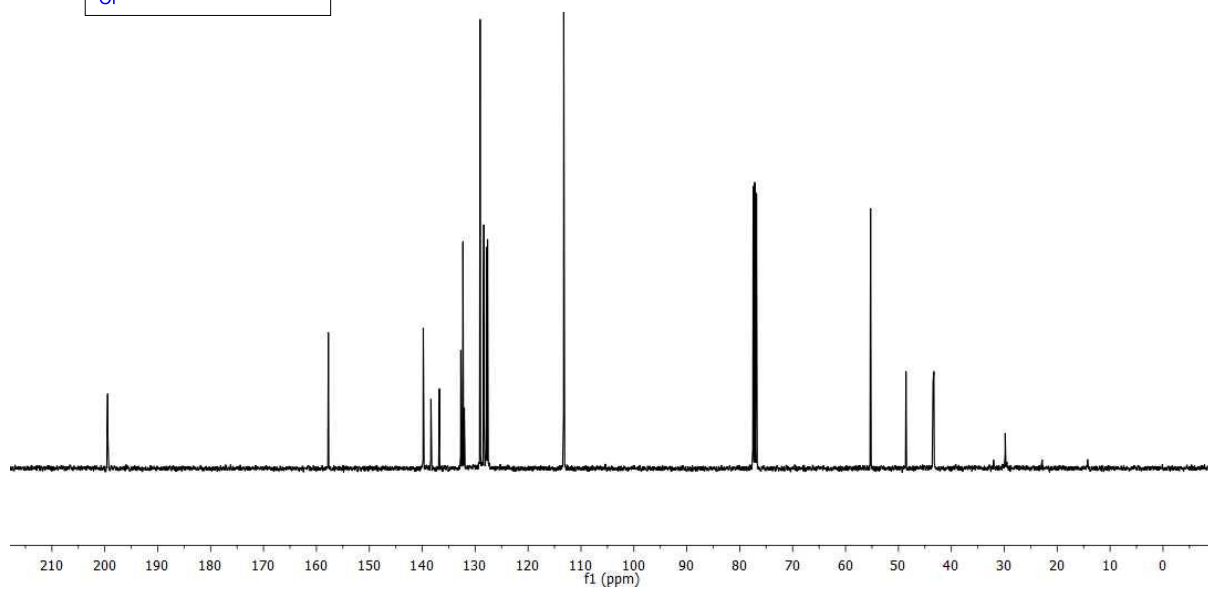
<sup>1</sup>H spectra of **4ad**



199.457

157.745  
139.806  
138.358  
136.740  
132.705  
132.312  
132.048  
129.063  
128.362  
127.879  
127.654  
113.253

55.269  
48.594  
43.459  
43.299

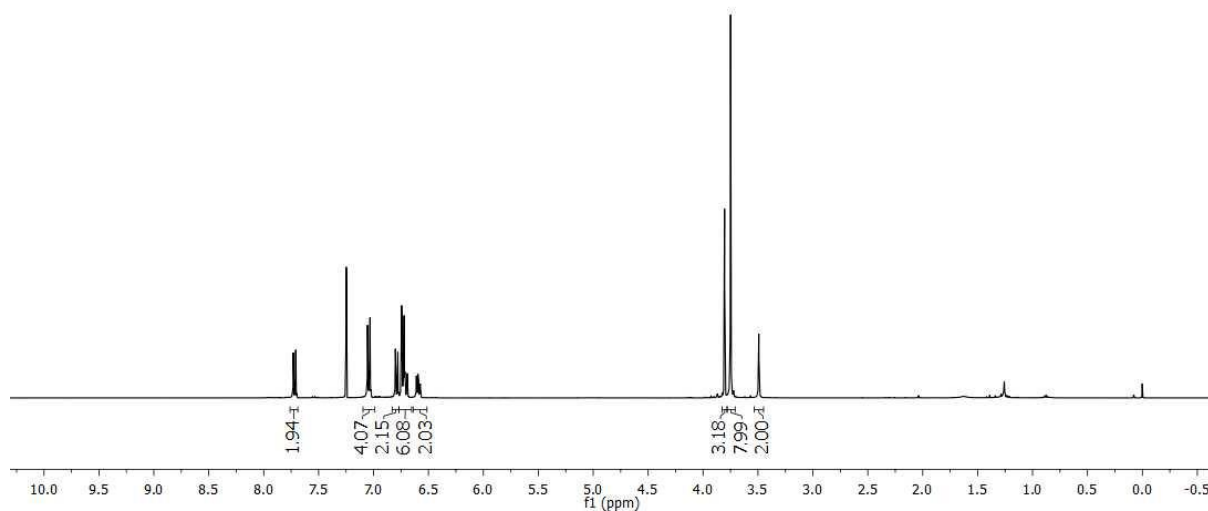
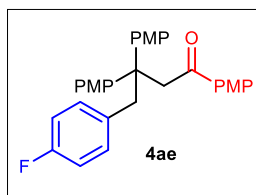


<sup>13</sup>C spectra of **4ad**

PD-4-90  
single\_pulse

7.731  
7.708  
7.055  
7.032  
6.801  
6.779  
6.744  
6.722  
6.694  
6.610  
6.596  
6.589  
6.574

3.804  
3.749  
3.492



<sup>1</sup>H spectra of 4ae

PD-4-90  
single pulse decoupled gated NOE

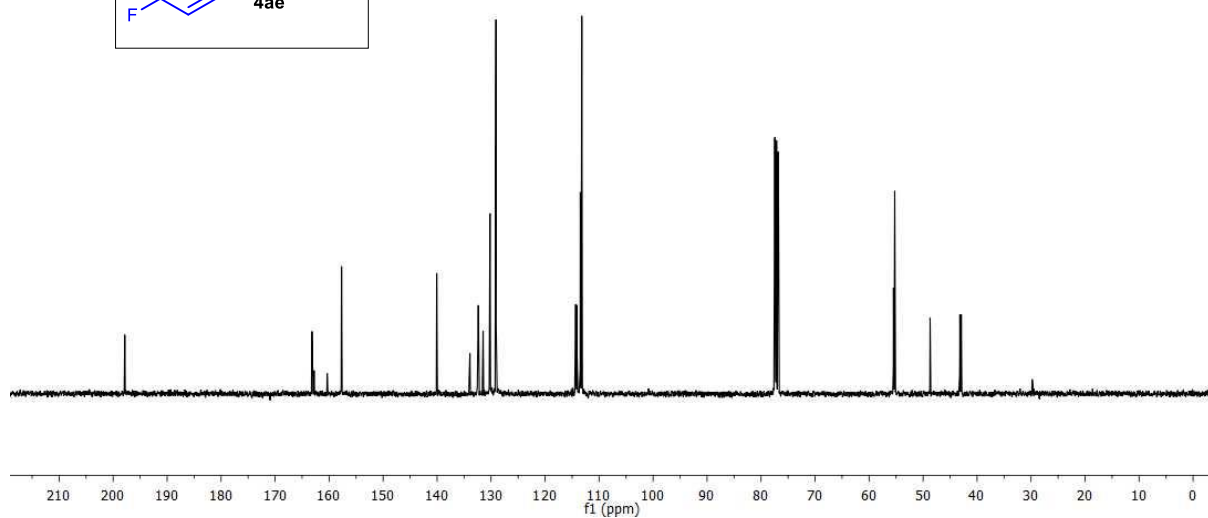
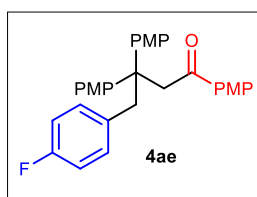
197.82

163.176  
162.785  
160.360  
157.695

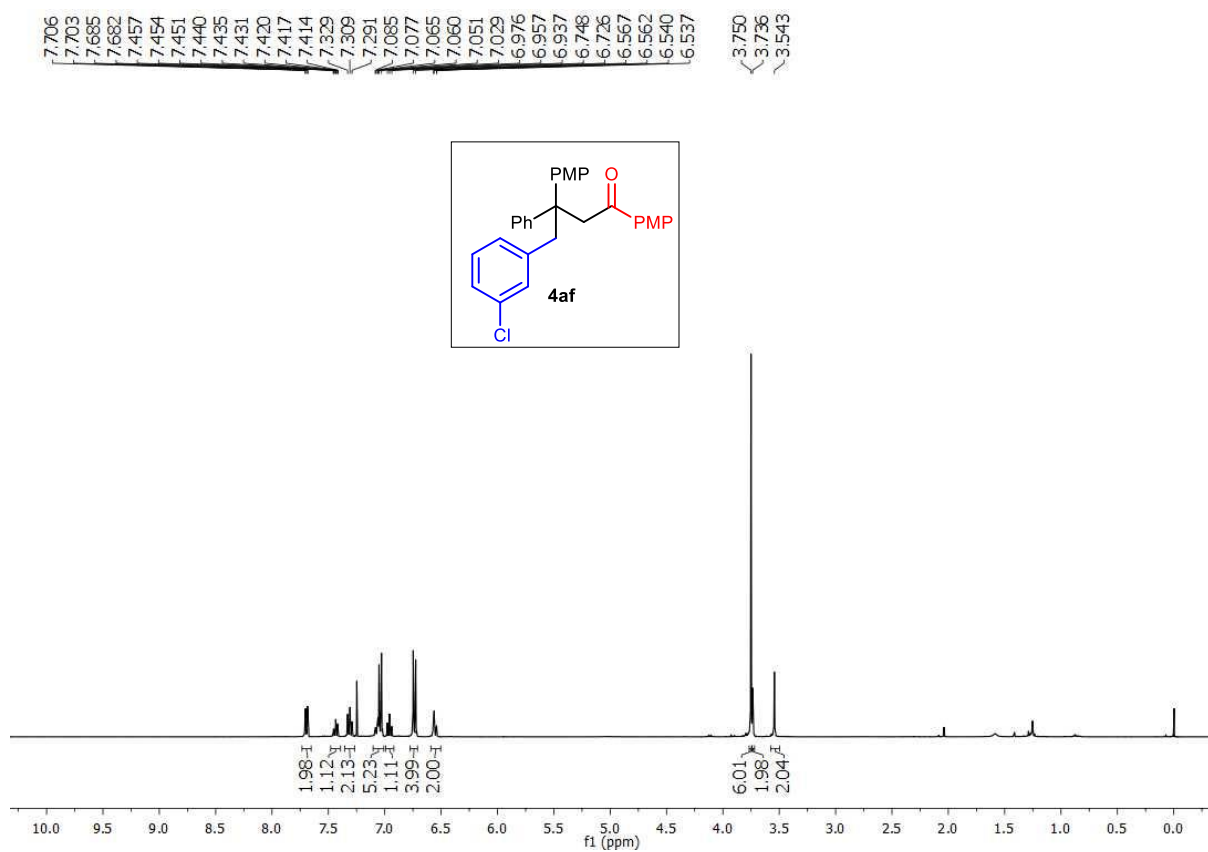
140.076  
133.950  
133.922  
132.357

114.328  
114.122  
113.462  
113.203

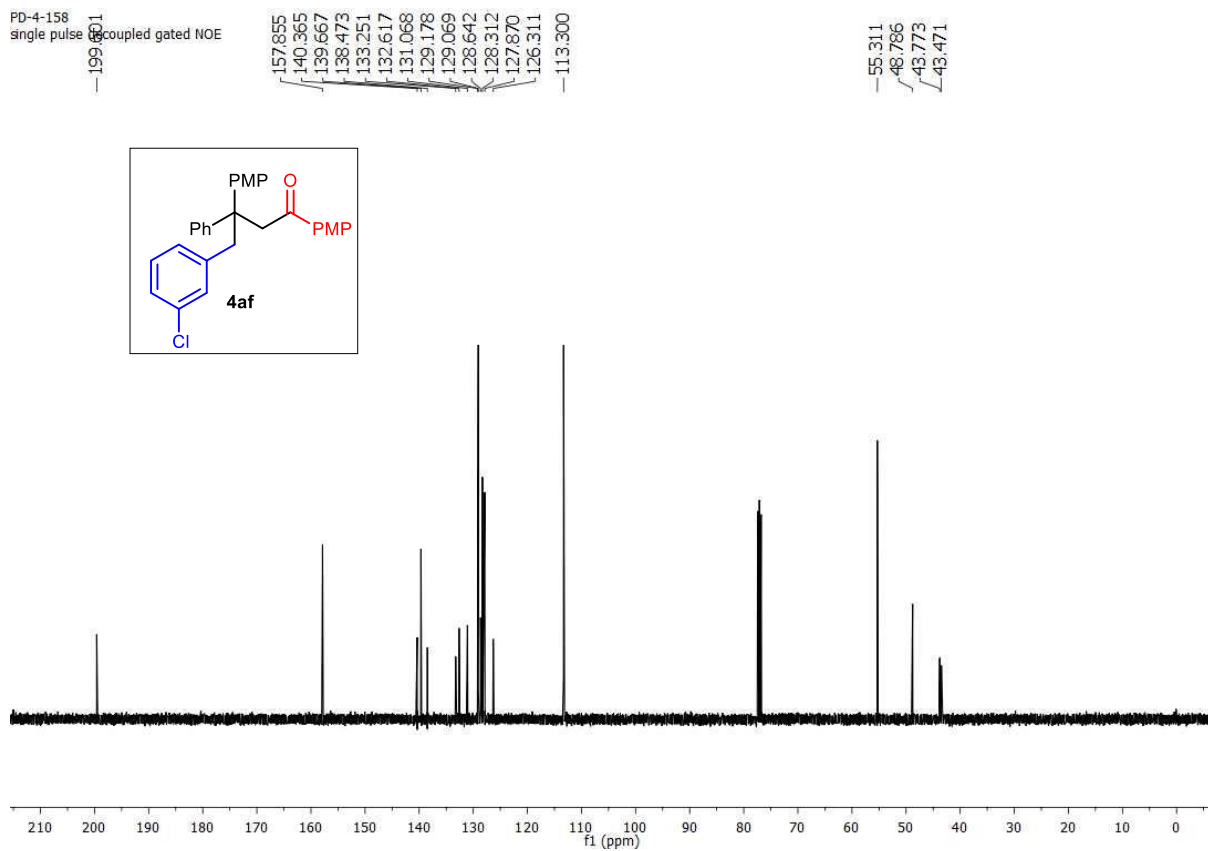
55.483  
55.247  
48.698  
43.177  
42.929



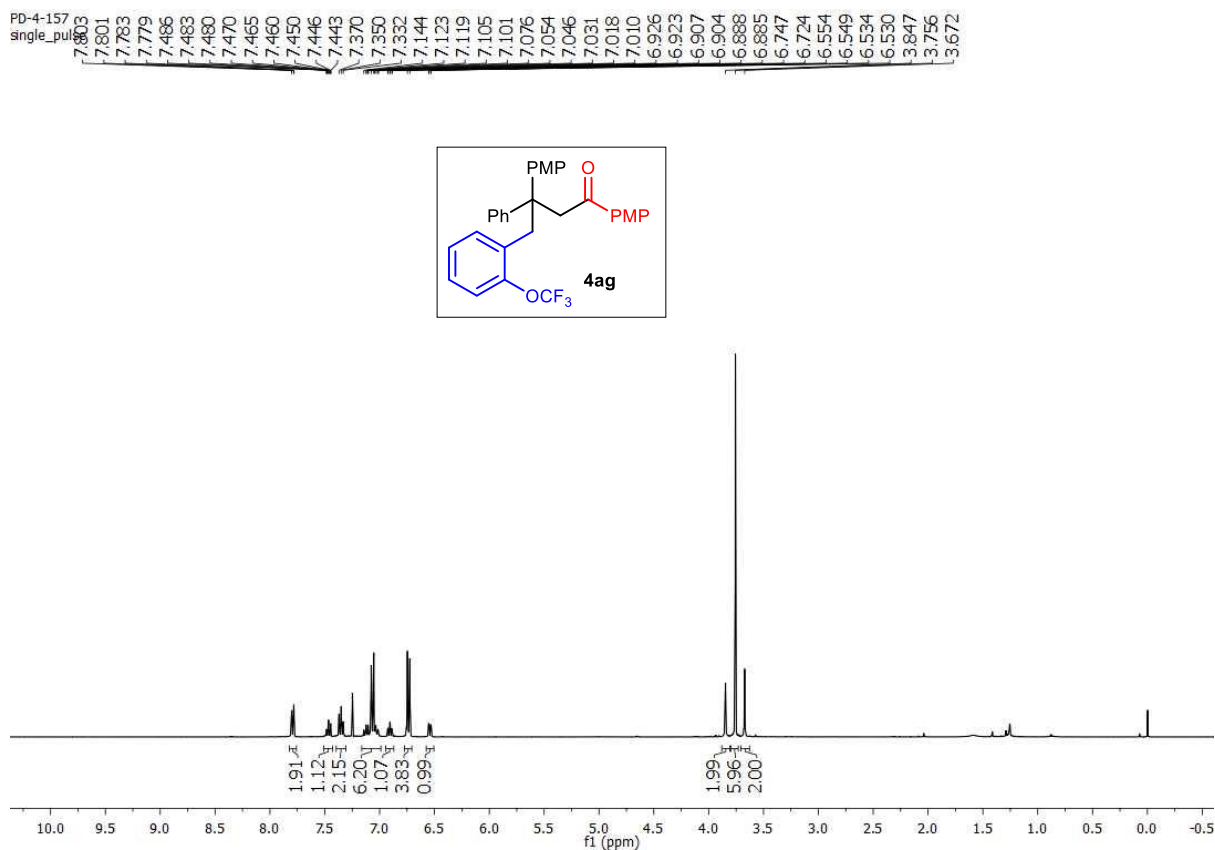
<sup>13</sup>C spectra of 4ae



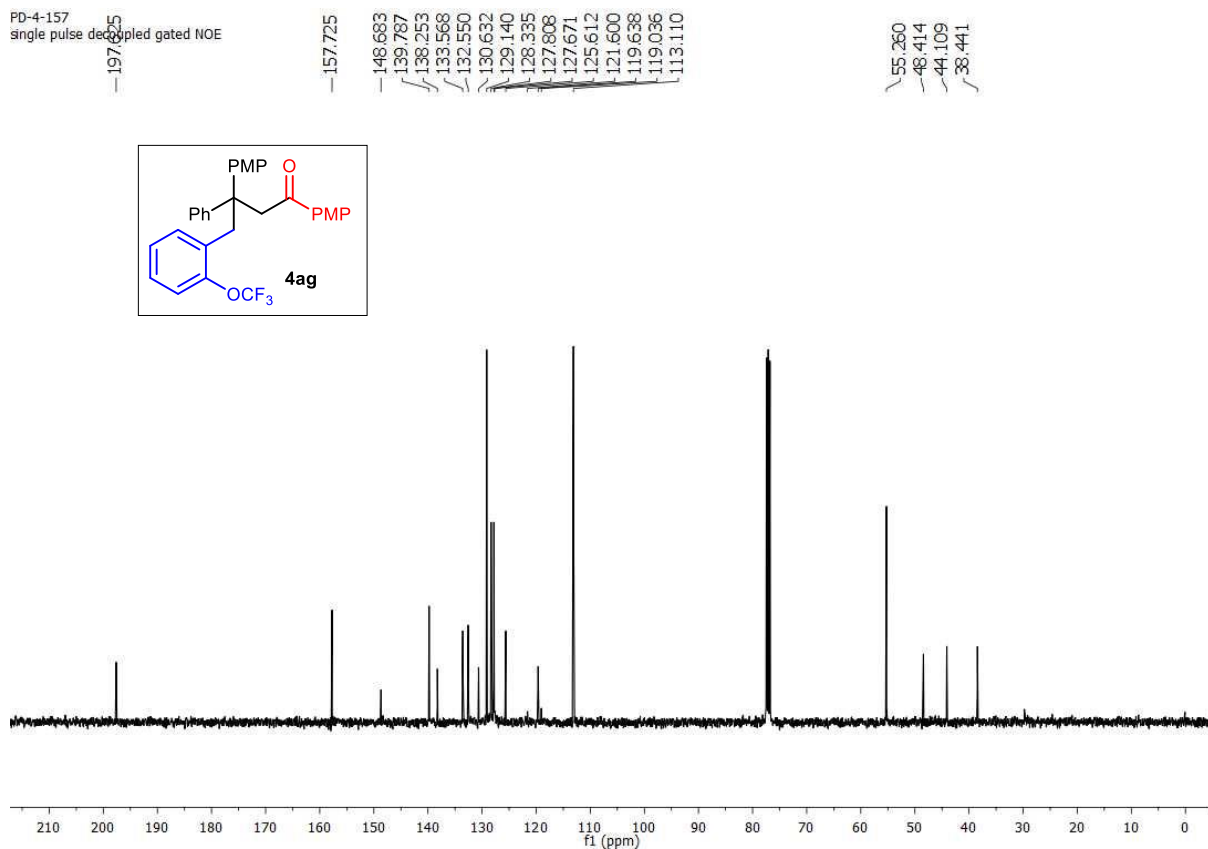
<sup>1</sup>H spectra of 4af



<sup>13</sup>C spectra of 4af



<sup>1</sup>H spectra of **4ag**

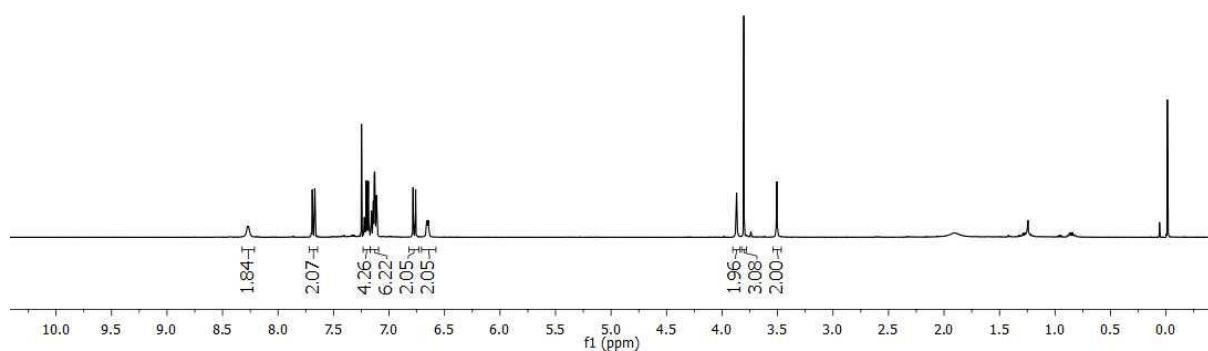
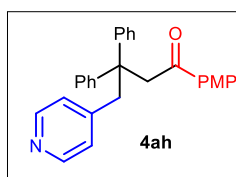


<sup>13</sup>C spectra of **4ag**



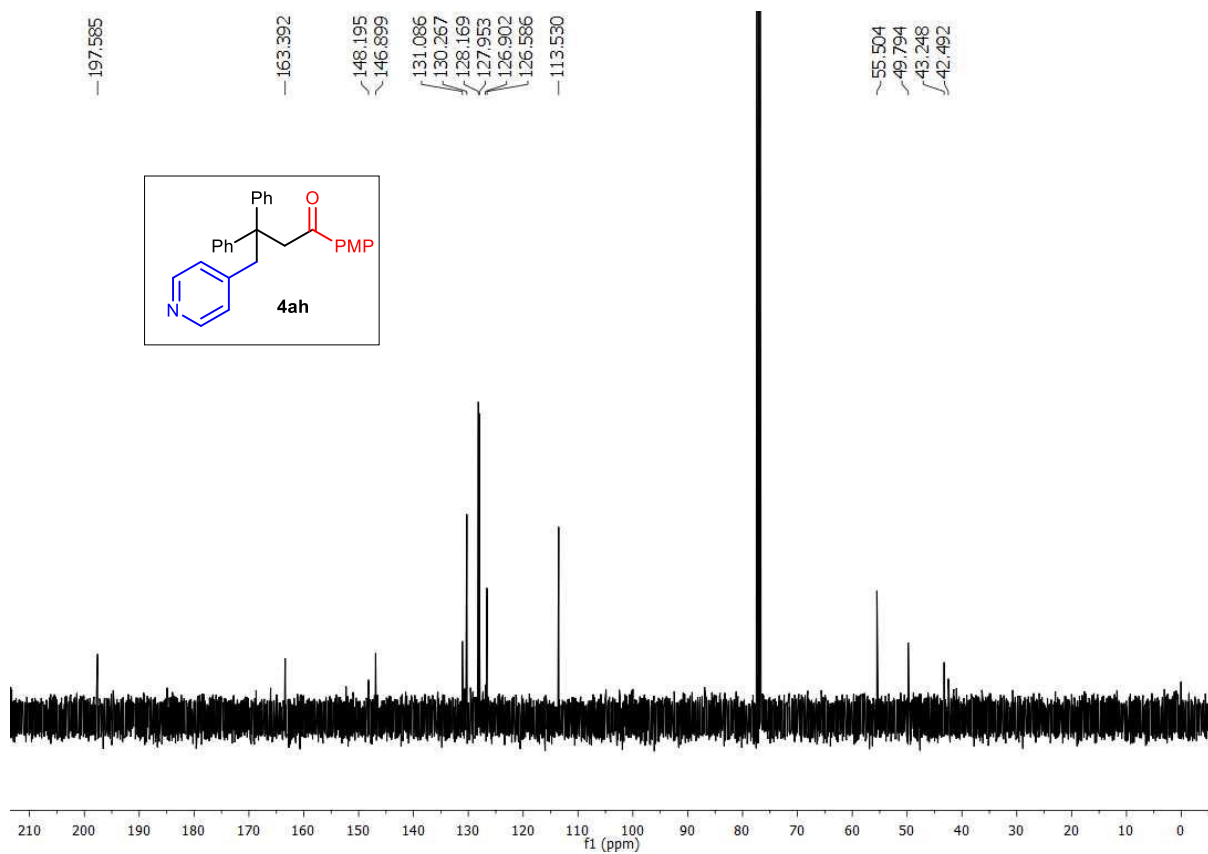
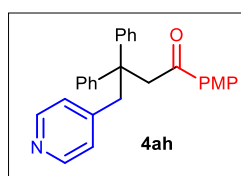
PD-4-135  
single\_pulse

8.274  
8.266  
7.691  
7.669  
7.226  
7.222  
7.218  
7.205  
7.201  
7.186  
7.158  
7.155  
7.141  
7.133  
7.129  
7.123  
7.112  
7.109  
6.783  
6.761  
6.660  
6.645  
3.869  
3.803  
3.507



<sup>1</sup>H spectra of 4ah

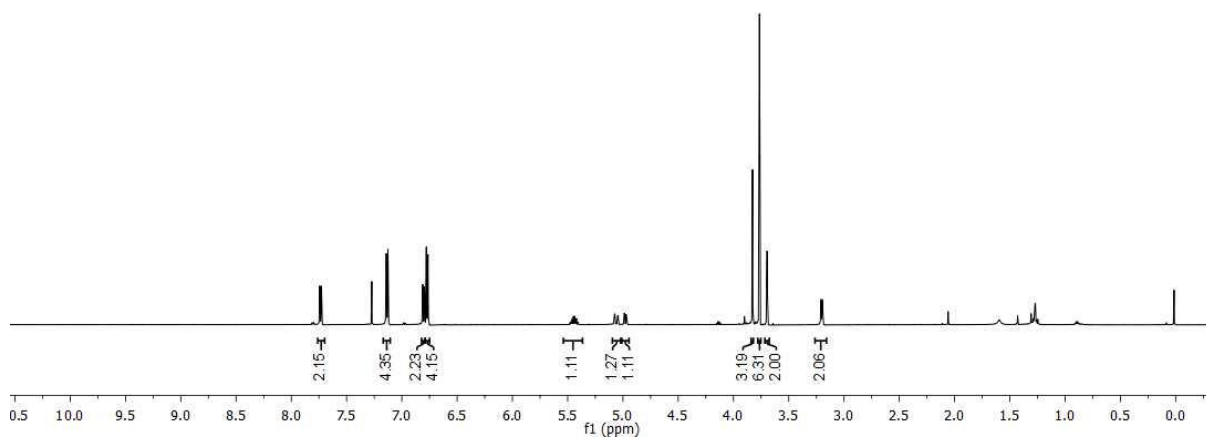
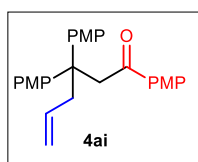
197.585  
163.392  
148.195  
146.899  
131.086  
130.267  
128.169  
127.953  
126.902  
126.586  
113.530  
55.504  
49.794  
43.248  
42.492



<sup>13</sup>C spectra of 4ah

07-PD-4-174R/1 — PD-4-174R 1H-NMR in CDCl3

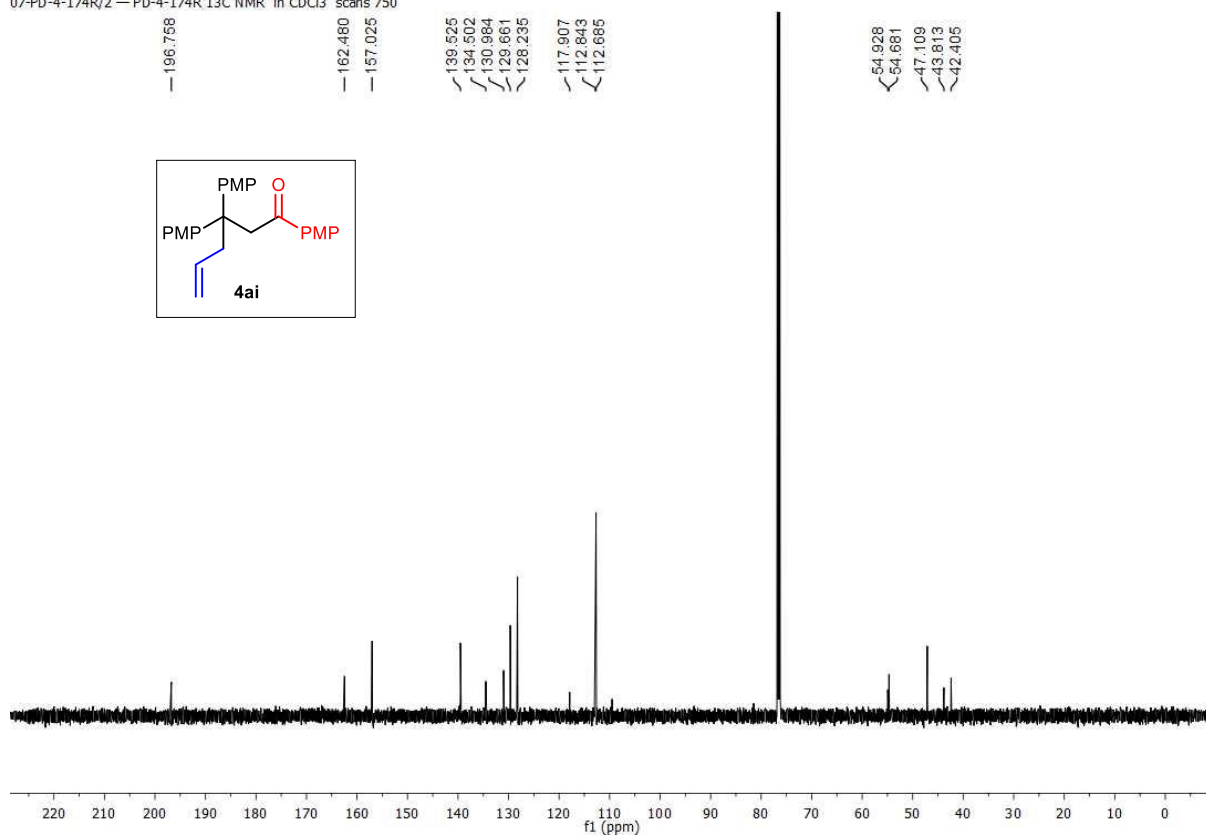
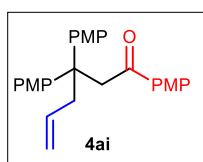
7.742  
7.728  
7.141  
6.812  
6.797  
6.779  
6.764  
5.477  
5.465  
5.448  
5.437  
5.420  
5.408  
5.076  
5.073  
5.047  
5.044  
4.988  
4.985  
4.971  
4.968  
3.828  
3.765  
3.696  
3.208  
3.196



<sup>1</sup>H spectra of 4ai

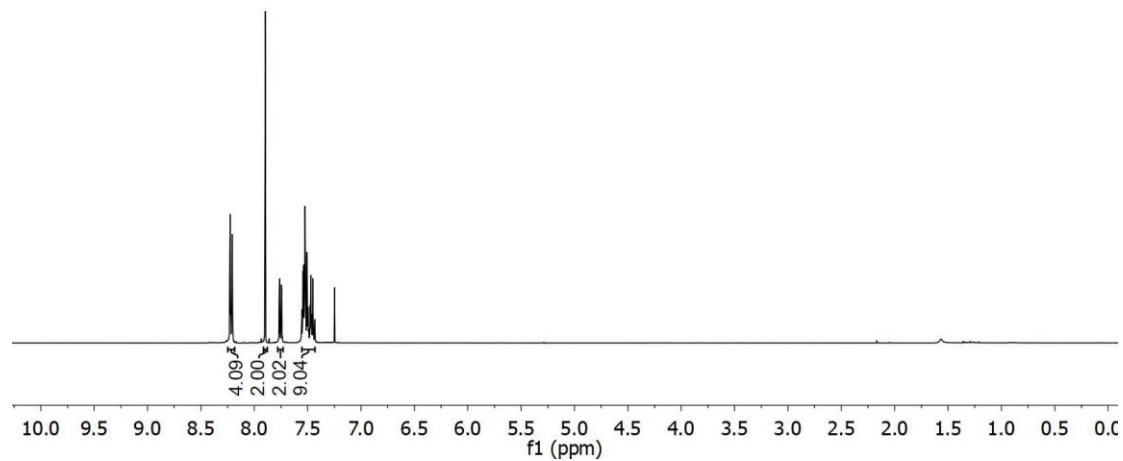
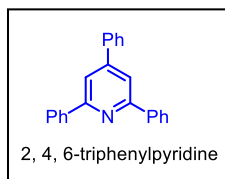
07-PD-4-174R/2 — PD-4-174R 13C NMR in CDCl3 scans 750

196.768  
162.480  
157.025  
139.525  
134.502  
130.964  
129.661  
128.235  
117.907  
112.843  
112.665  
54.928  
54.681  
47.109  
43.813  
42.405



<sup>13</sup>C spectra of 4ai

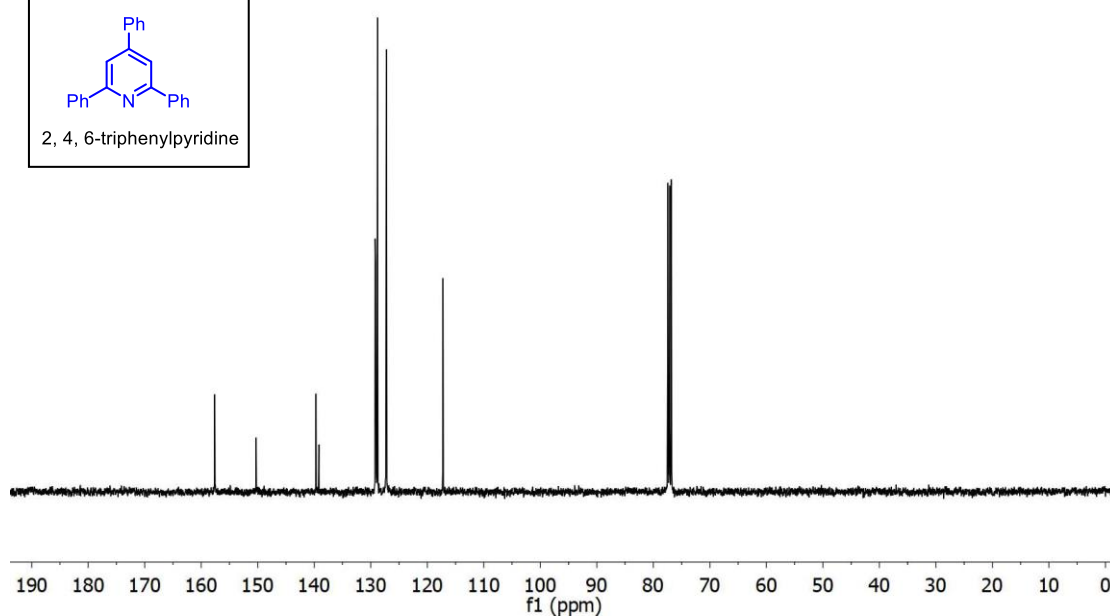
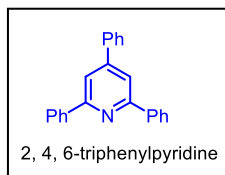
SDD-357[1] — single\_pulse  
 8.229  
 8.225  
 8.207  
 8.204  
 7.898  
 7.767  
 7.763  
 7.758  
 7.750  
 7.746  
 7.743  
 7.568  
 7.555  
 7.546  
 7.543  
 7.537  
 7.525  
 7.521  
 7.518  
 7.505  
 7.495  
 7.477  
 7.473  
 7.466  
 7.451  
 7.433



<sup>1</sup>H spectra of 2,4,6-triphenylpyridine

SDD-357[1] — single pulse decoupled gated NOE

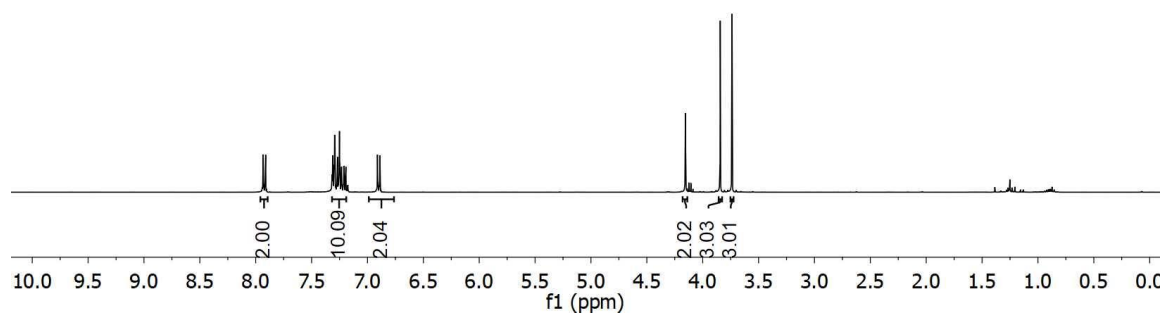
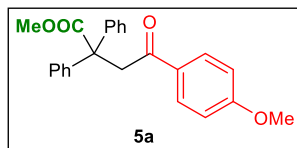
157.609  
 150.299  
 139.692  
 139.175  
 129.216  
 129.144  
 129.075  
 128.806  
 127.289  
 117.223



<sup>13</sup>C spectra of 2,4,6-triphenylpyridine

SNAN-1258E — single\_pulse

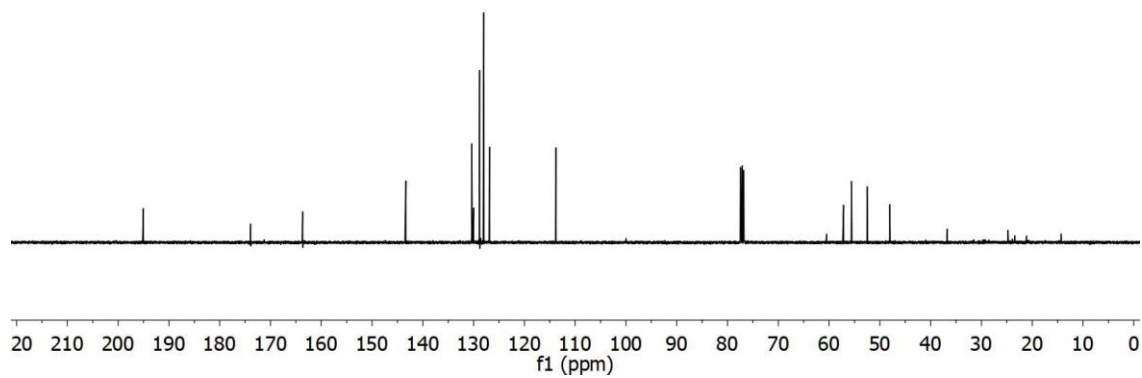
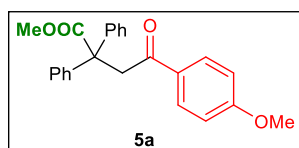
7.928  
7.911  
7.316  
7.312  
7.306  
7.297  
7.294  
7.291  
7.285  
7.271  
7.268  
7.263  
7.256  
7.252  
7.251  
7.231  
7.215  
7.211  
7.207  
7.200  
7.193  
6.911  
6.888  
4.154  
3.843  
3.738



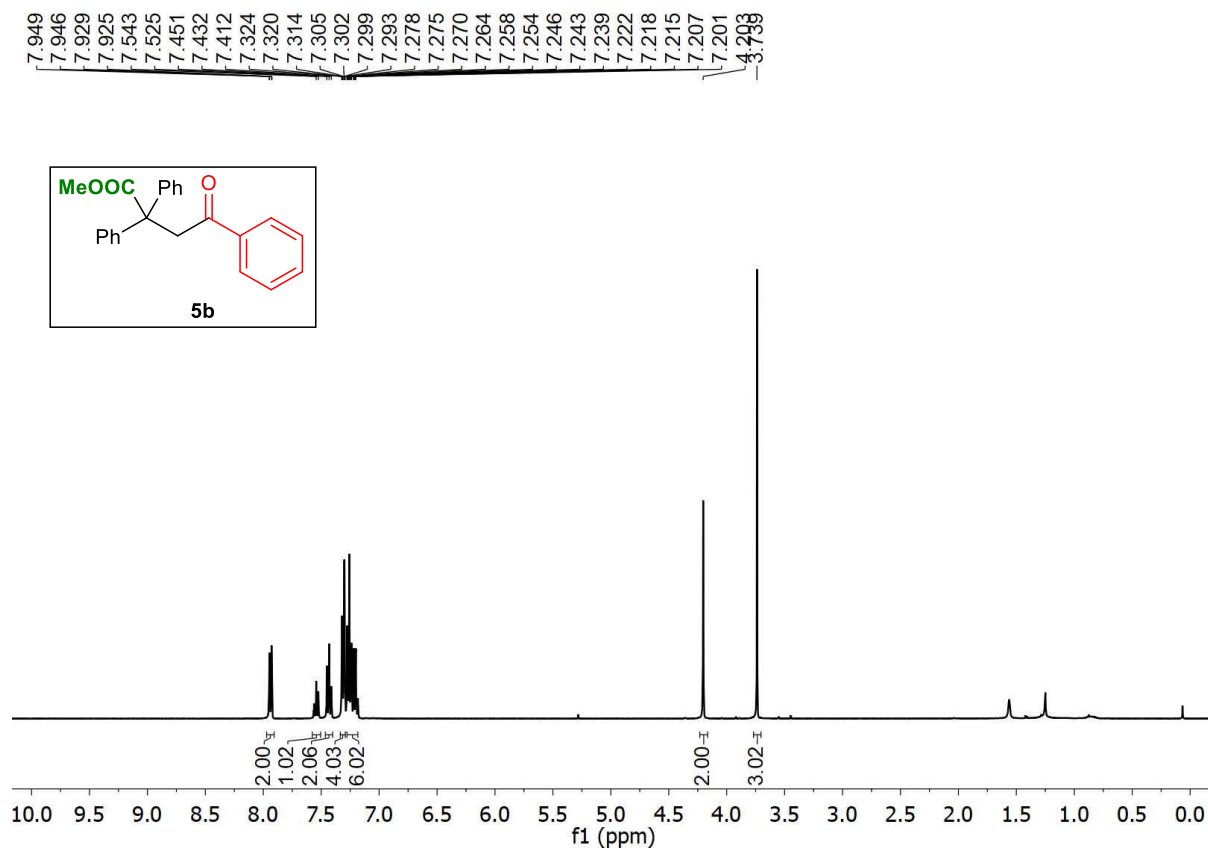
<sup>1</sup>H spectra of 5a

SNAN-1258E — single pulse decoupled gated NOE

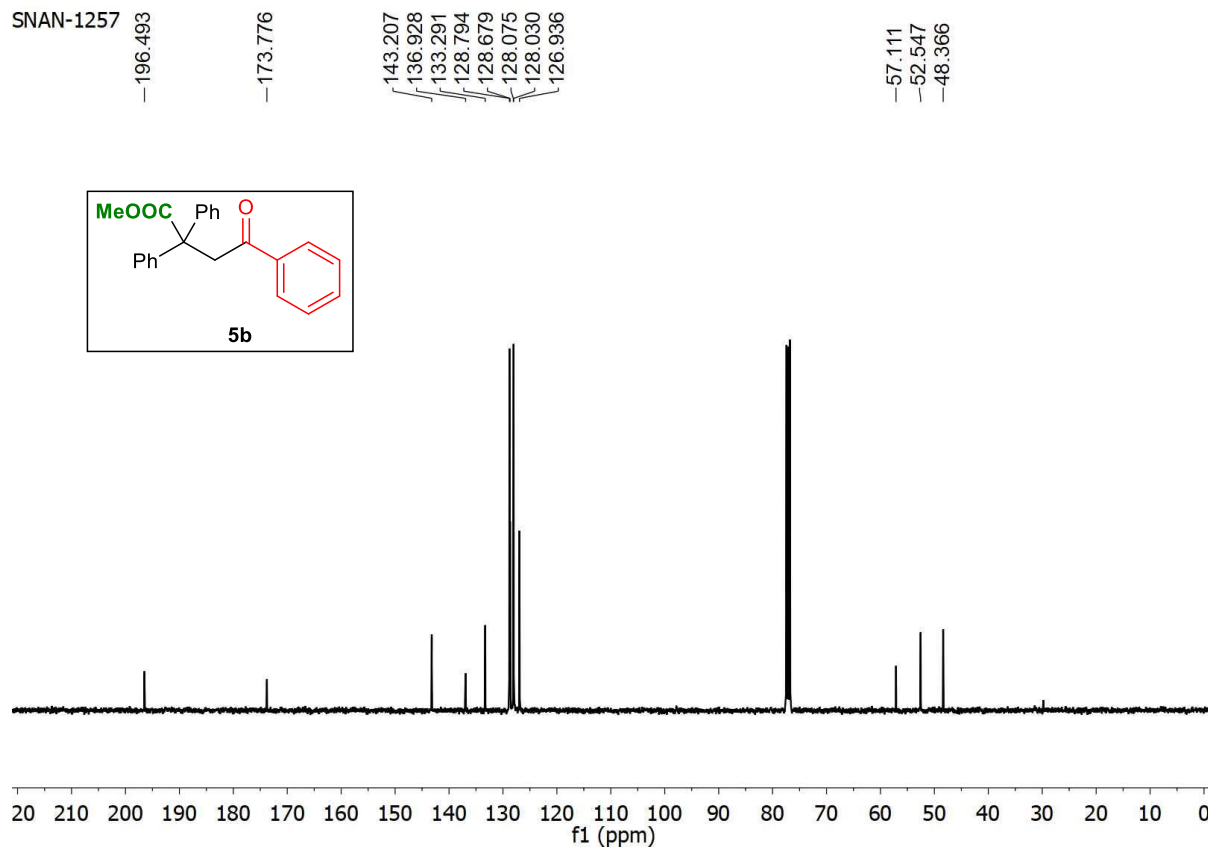
195.025  
173.903  
163.661  
143.356  
130.367  
130.003  
128.813  
127.995  
126.870  
113.799  
57.142  
55.575  
52.509  
48.038



<sup>13</sup>C spectra of 5a

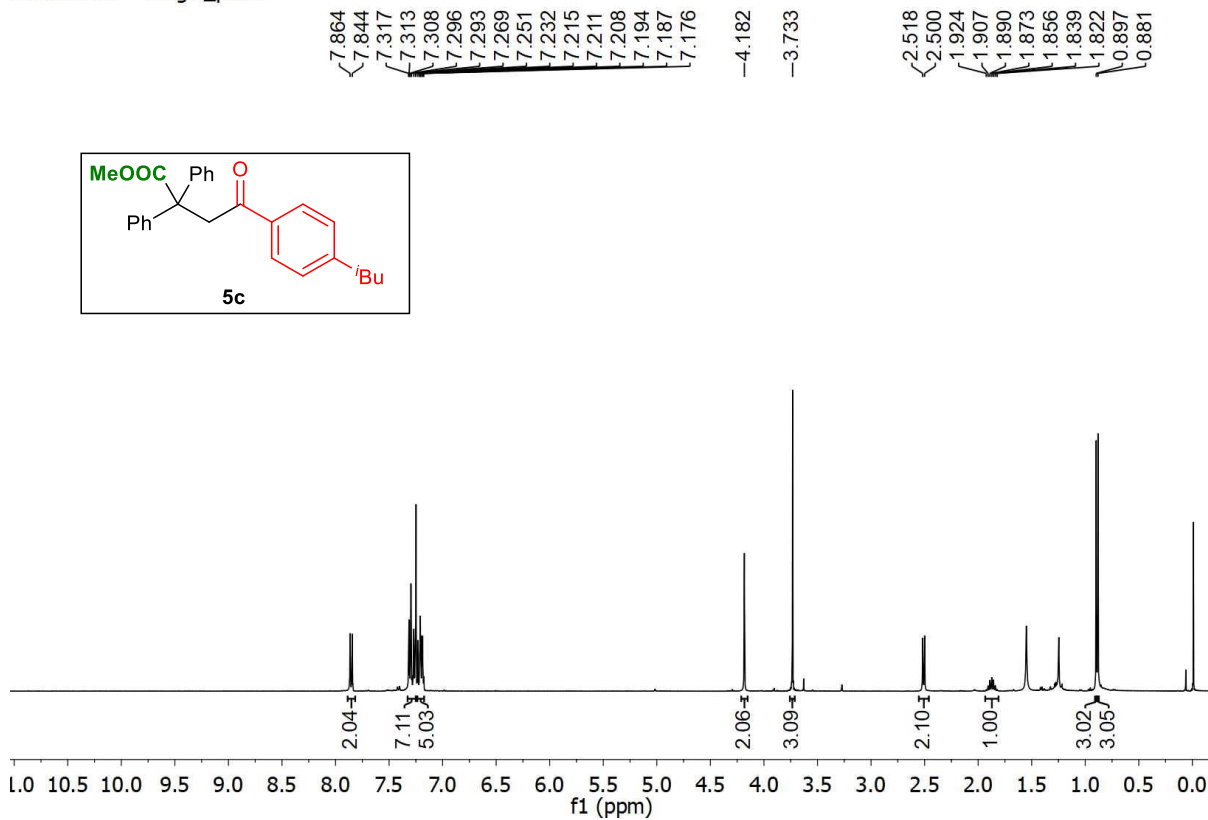


<sup>1</sup>H spectra of **5b**



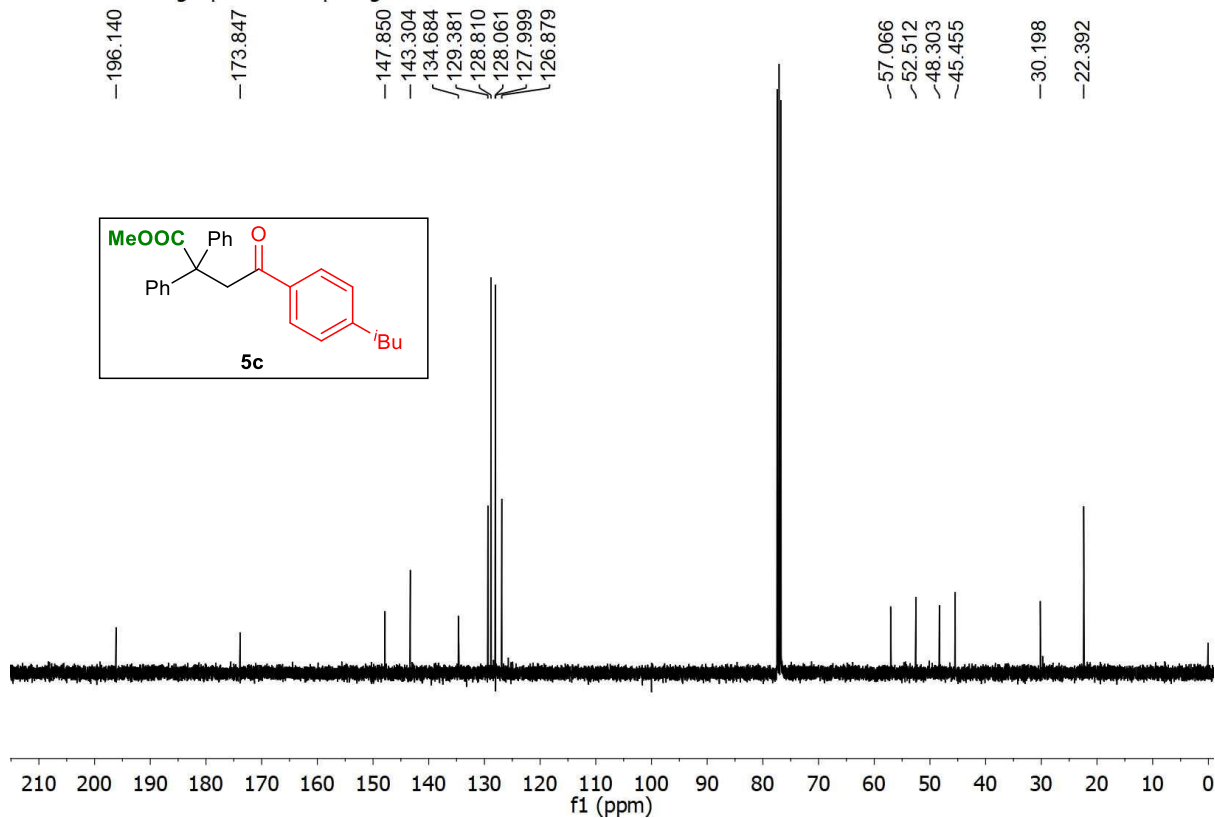
<sup>13</sup>C spectra of **5b**

SNAN-1299 — single\_pulse



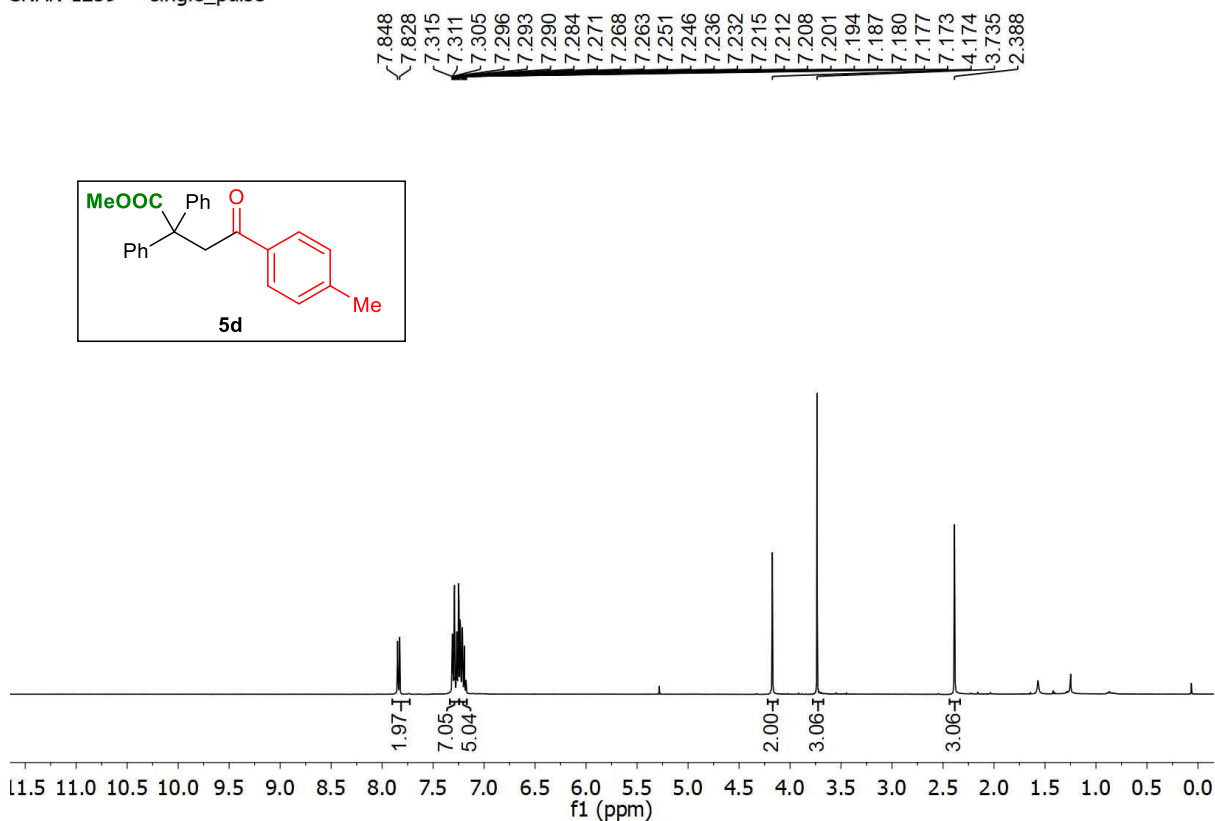
<sup>1</sup>H spectra of **5c**

SNAN-1299 — single pulse decoupled gated NOE



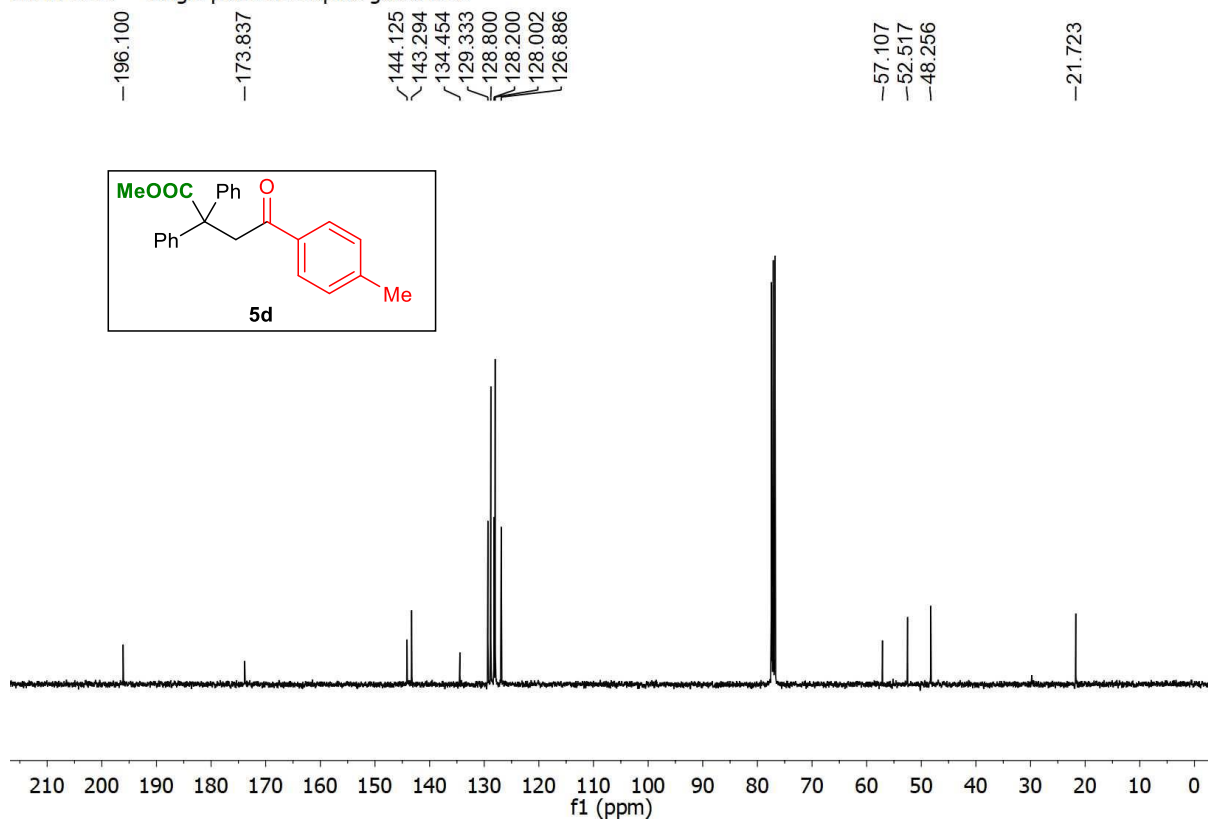
<sup>13</sup>C spectra of **5c**

SNAN-1259 — single\_pulse



<sup>1</sup>H spectra of **5d**

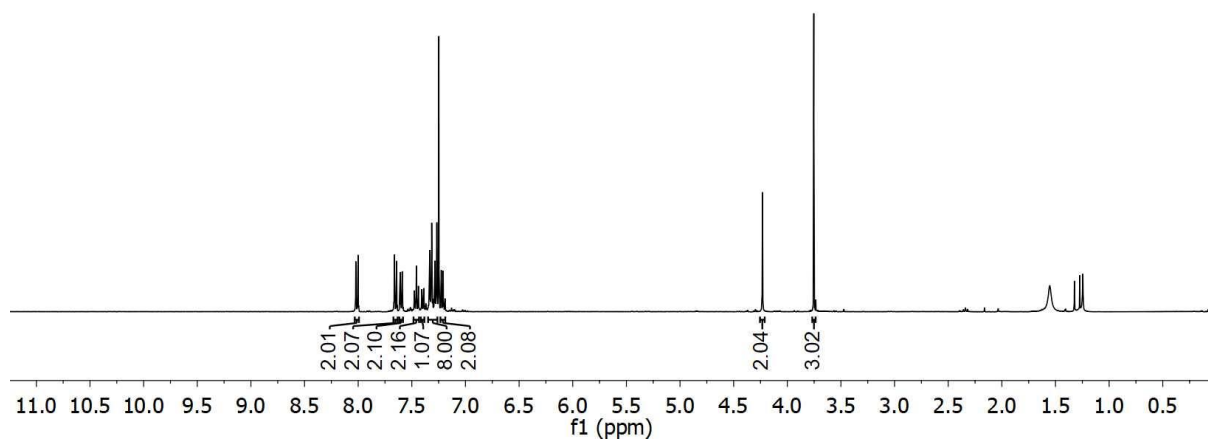
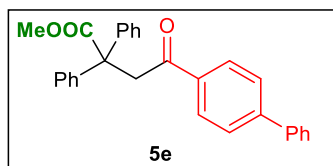
SNAN-1259 — single pulse decoupled gated NOE



<sup>13</sup>C spectra of **5d**

SNAN-1329D — single\_pulse

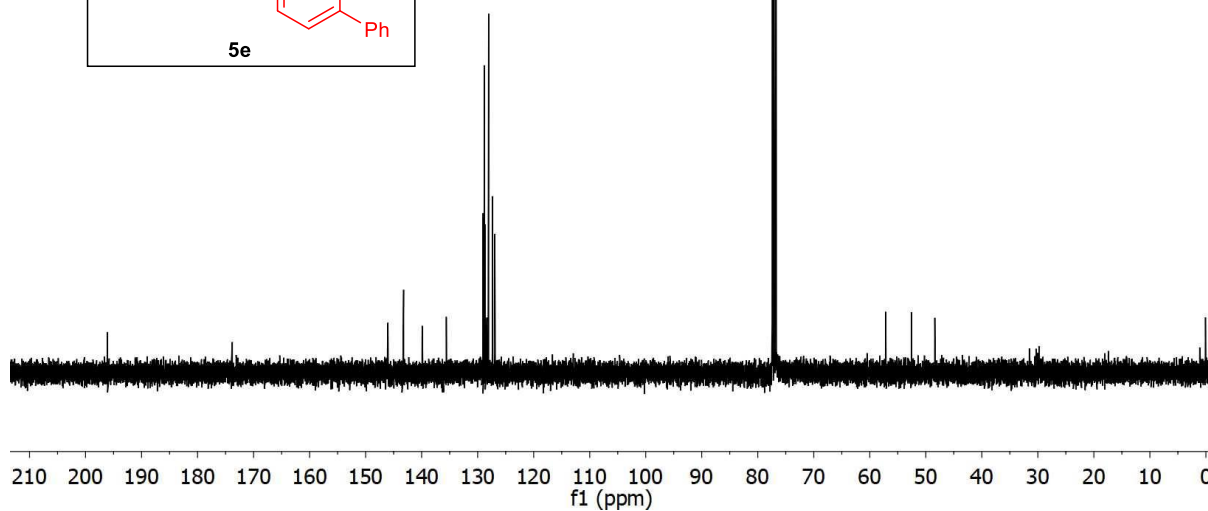
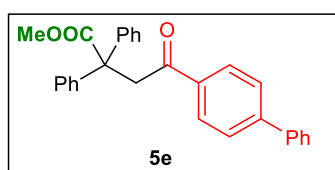
8.022  
8.006  
8.001  
7.663  
7.646  
7.642  
7.611  
7.607  
7.593  
7.590  
7.587  
7.475  
7.457  
7.454  
7.438  
7.406  
7.388  
7.336  
7.332  
7.327  
7.318  
7.315  
7.312  
7.288  
7.285  
7.279  
7.267  
7.263  
7.229  
7.226  
7.222  
7.208  
4.753



<sup>1</sup>H spectra of **5e**

SNAN-1329D — single pulse decoupled gated NOE

196.077  
173.803  
146.025  
143.221  
139.902  
135.599  
129.046  
128.806  
128.681  
128.359  
128.044  
127.349  
127.323  
126.947  
57.157  
52.565  
48.394

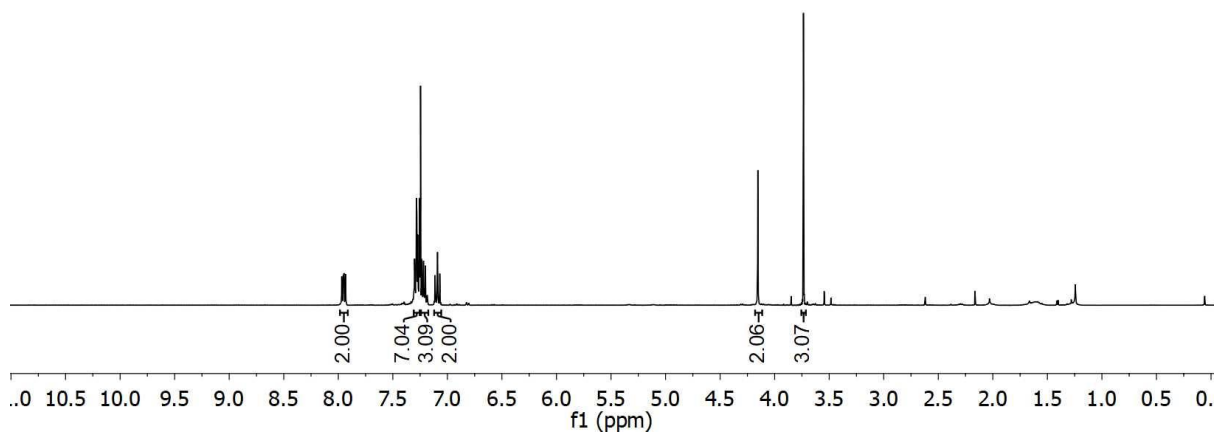
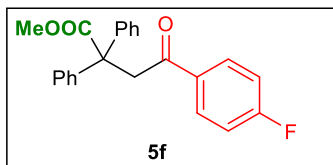


<sup>13</sup>C spectra of **5e**



SNAN-1267 — single\_pulse

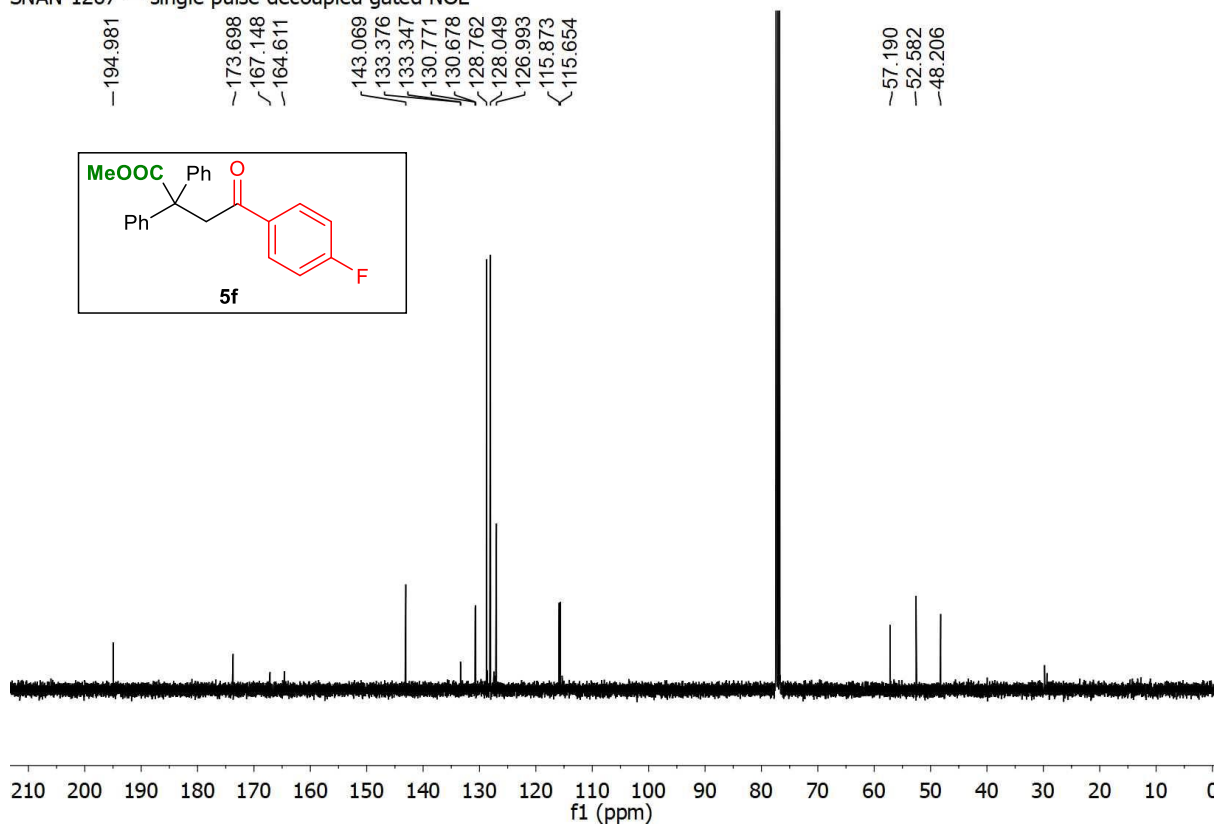
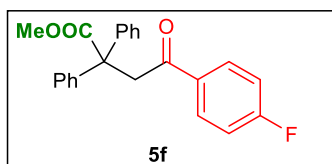
7.969  
7.955  
7.946  
7.938  
7.933  
7.306  
7.301  
7.296  
7.287  
7.284  
7.283  
7.281  
7.272  
7.257  
7.255  
7.240  
7.235  
7.223  
7.219  
7.213  
7.209  
7.201  
7.188  
7.184  
7.181  
7.113  
7.091  
7.070  
4.154  
3.736



<sup>1</sup>H spectra of **5f**

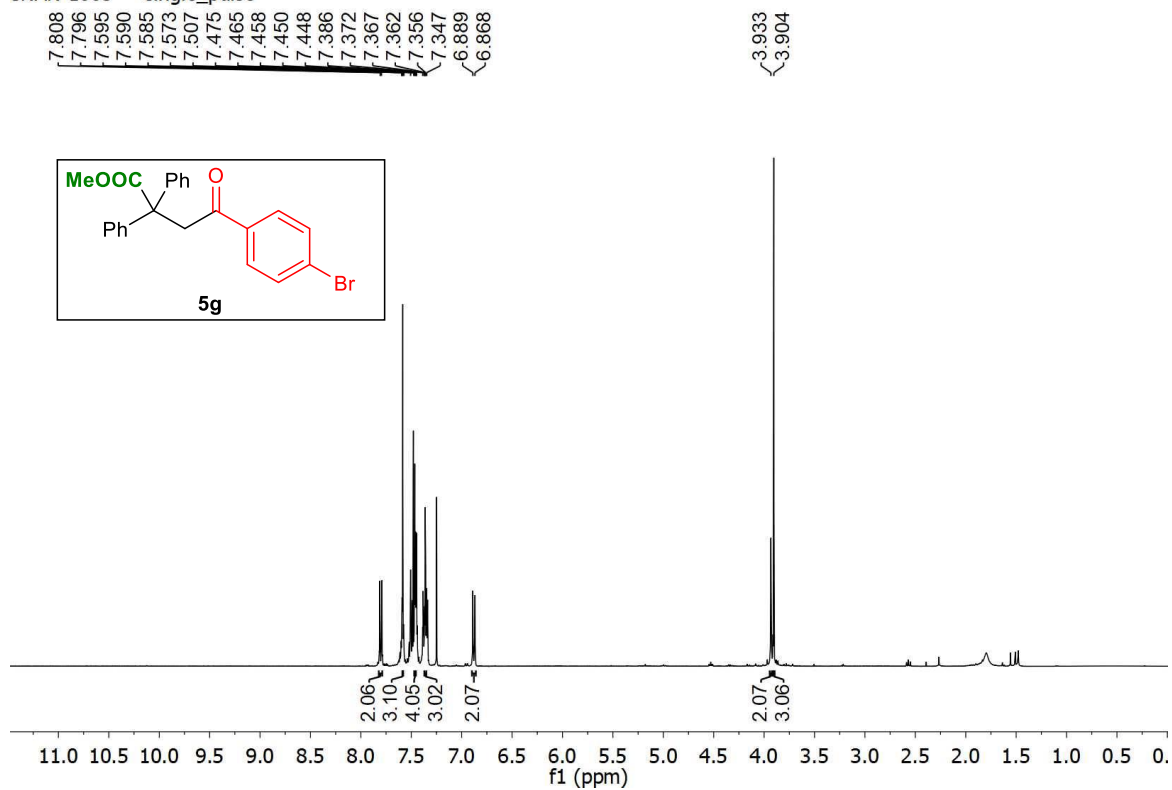
SNAN-1267 — single pulse decoupled gated NOE

194.981  
173.698  
167.148  
164.611  
143.069  
133.376  
133.347  
130.771  
130.678  
128.762  
128.049  
126.993  
115.873  
115.654  
57.190  
52.582  
48.206



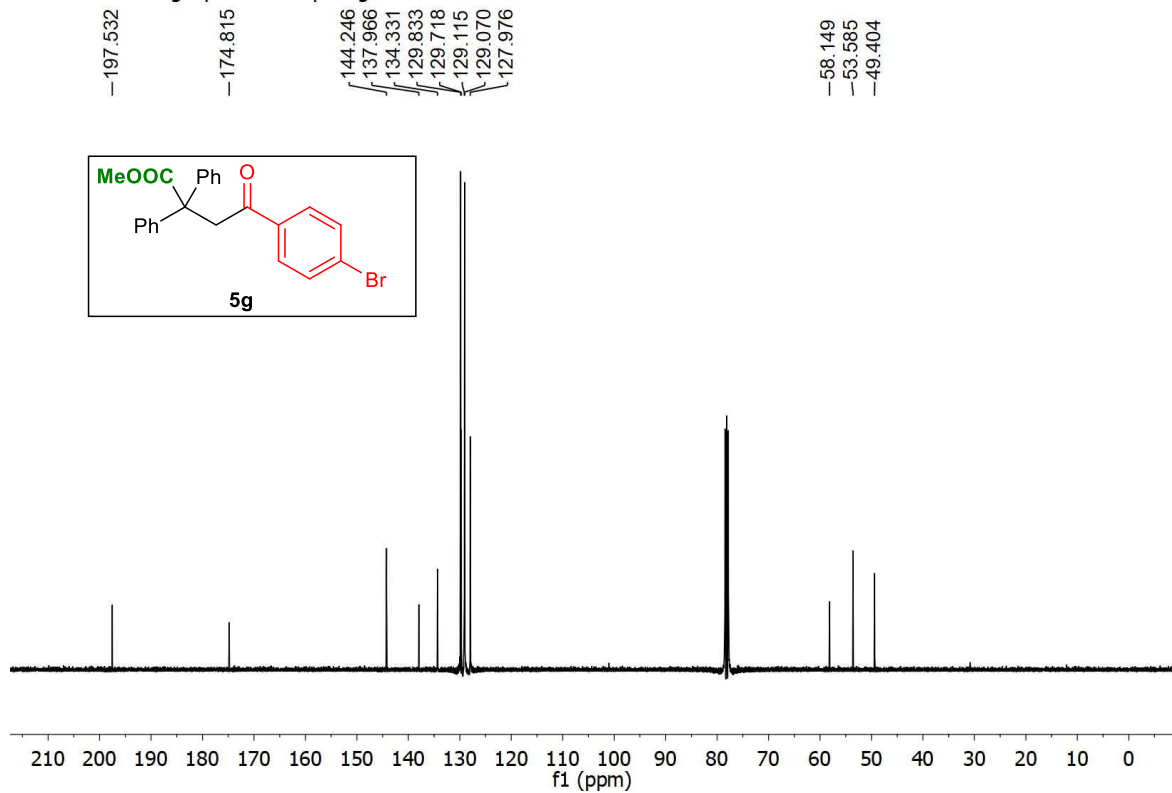
<sup>13</sup>C spectra of **5f**

SNAN-1665 — single\_pulse



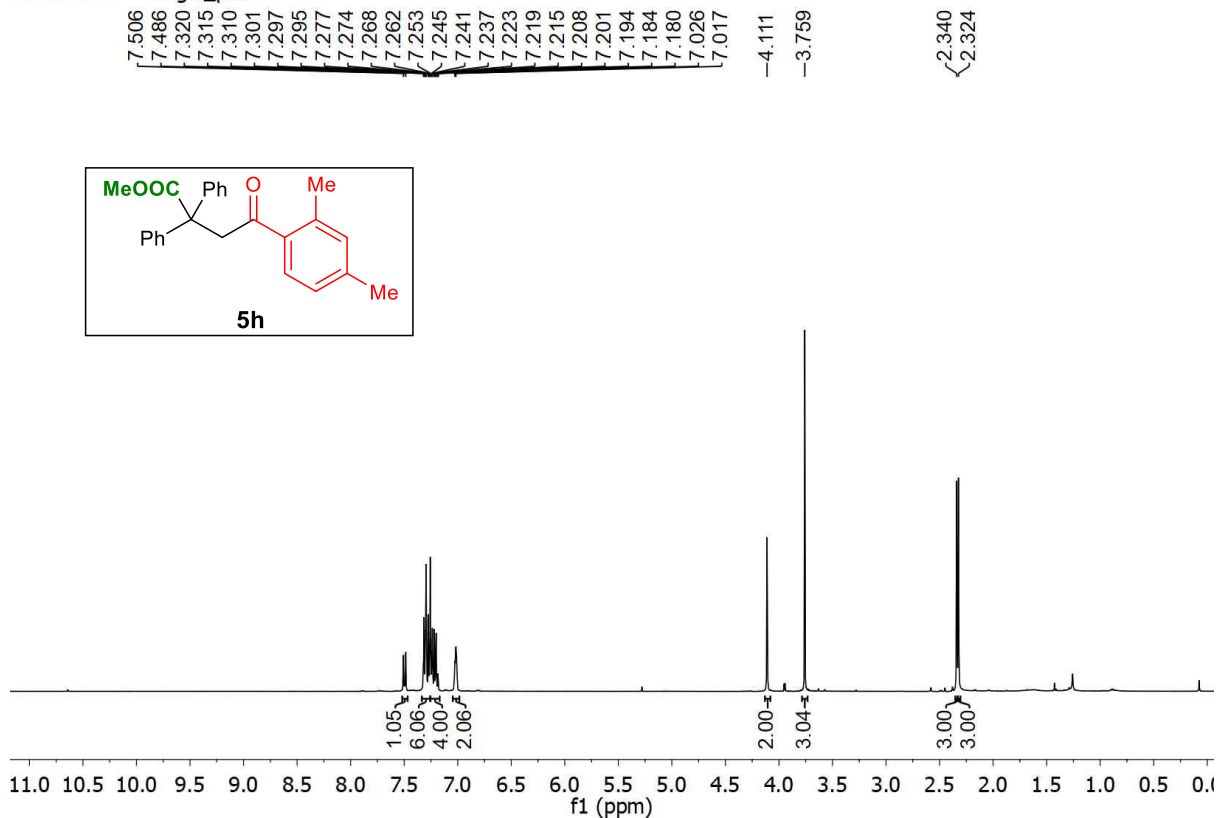
<sup>1</sup>H spectra of **5g**

SNAN-1665 — single pulse decoupled gated NOE



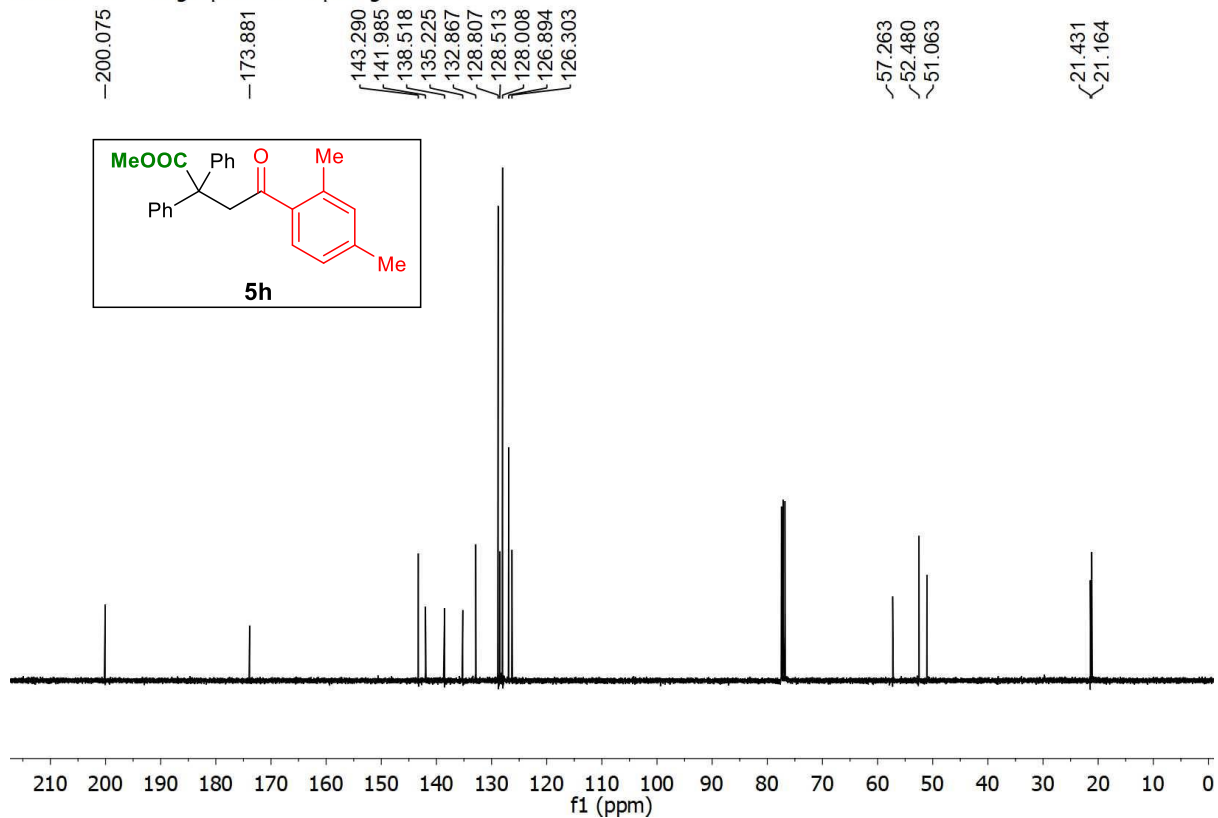
<sup>13</sup>C spectra of **5g**

SNAN-1268 — single\_pulse



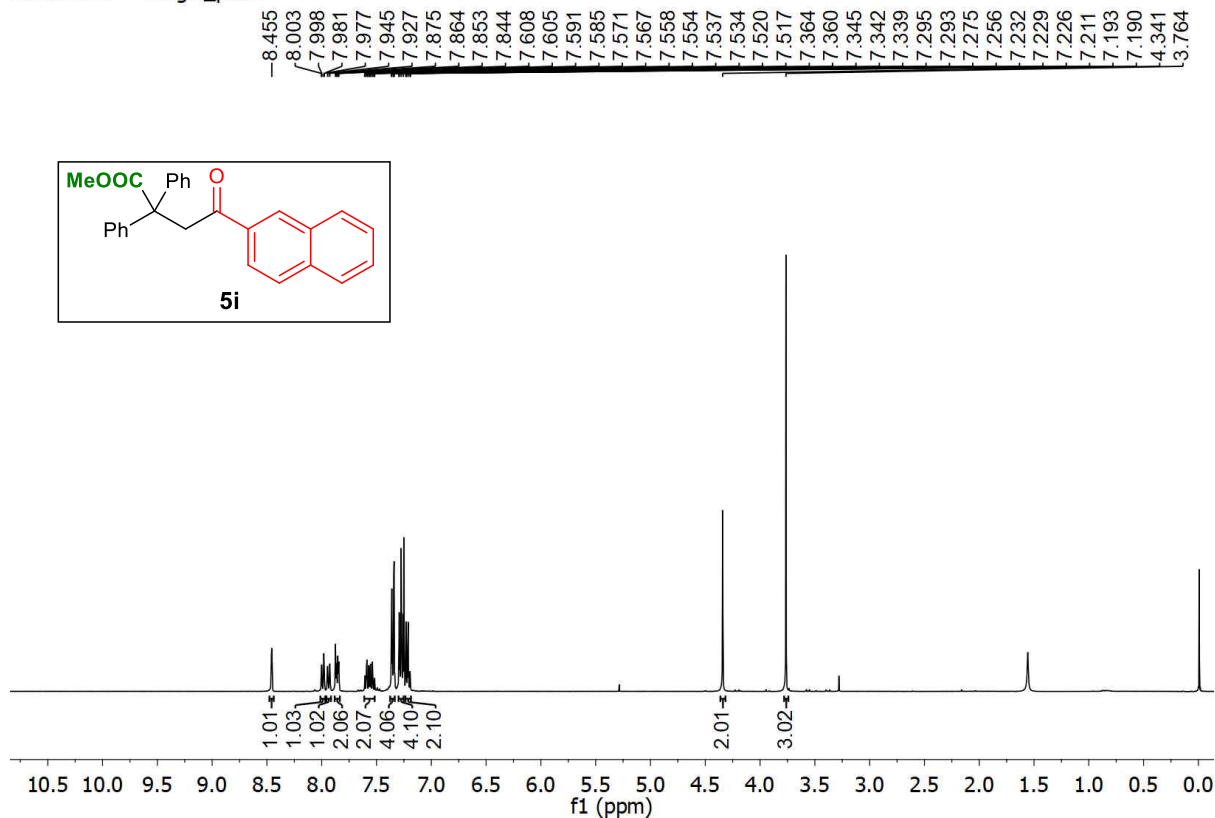
<sup>1</sup>H spectra of **5h**

SNAN-1268 — single\_pulse decoupled gated NOE



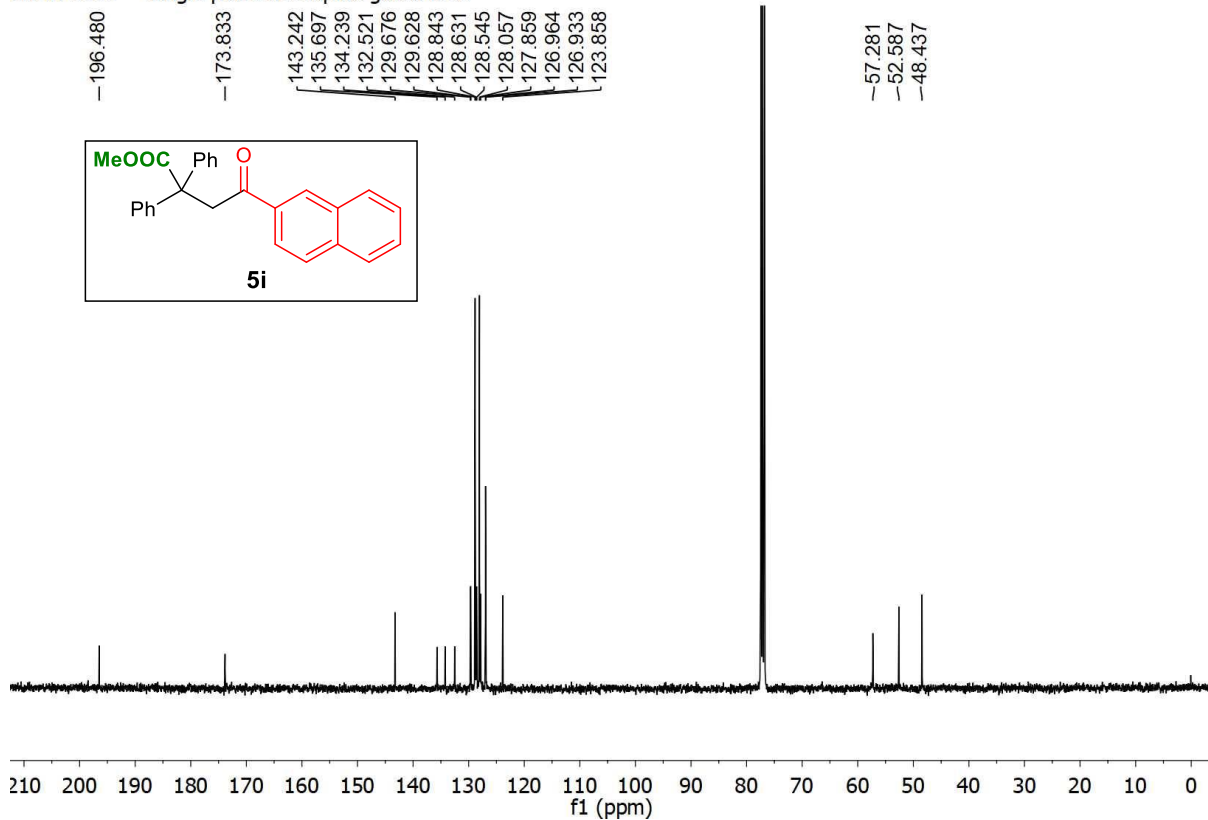
<sup>13</sup>C spectra of **5h**

SNAN-1336 — single\_pulse



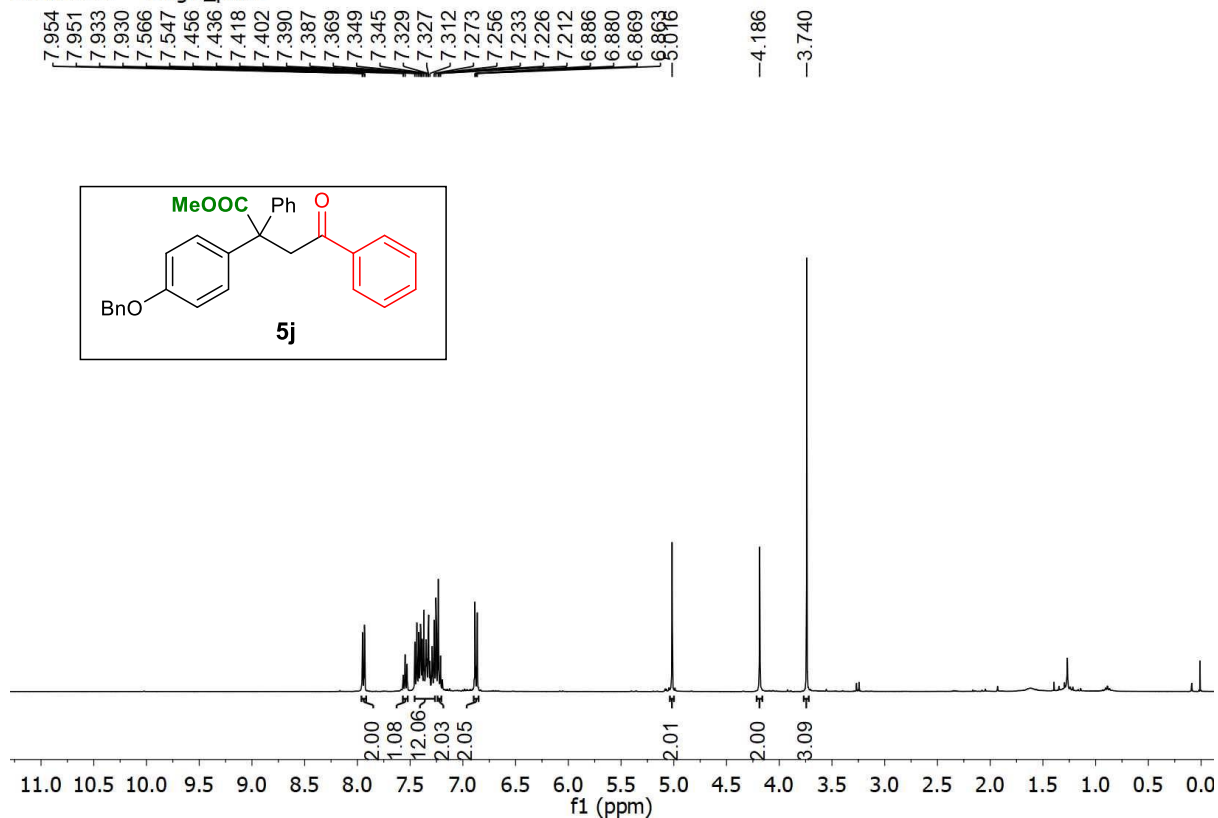
<sup>1</sup>H spectra of **5i**

SNAN-1336 — single pulse decoupled gated NOE



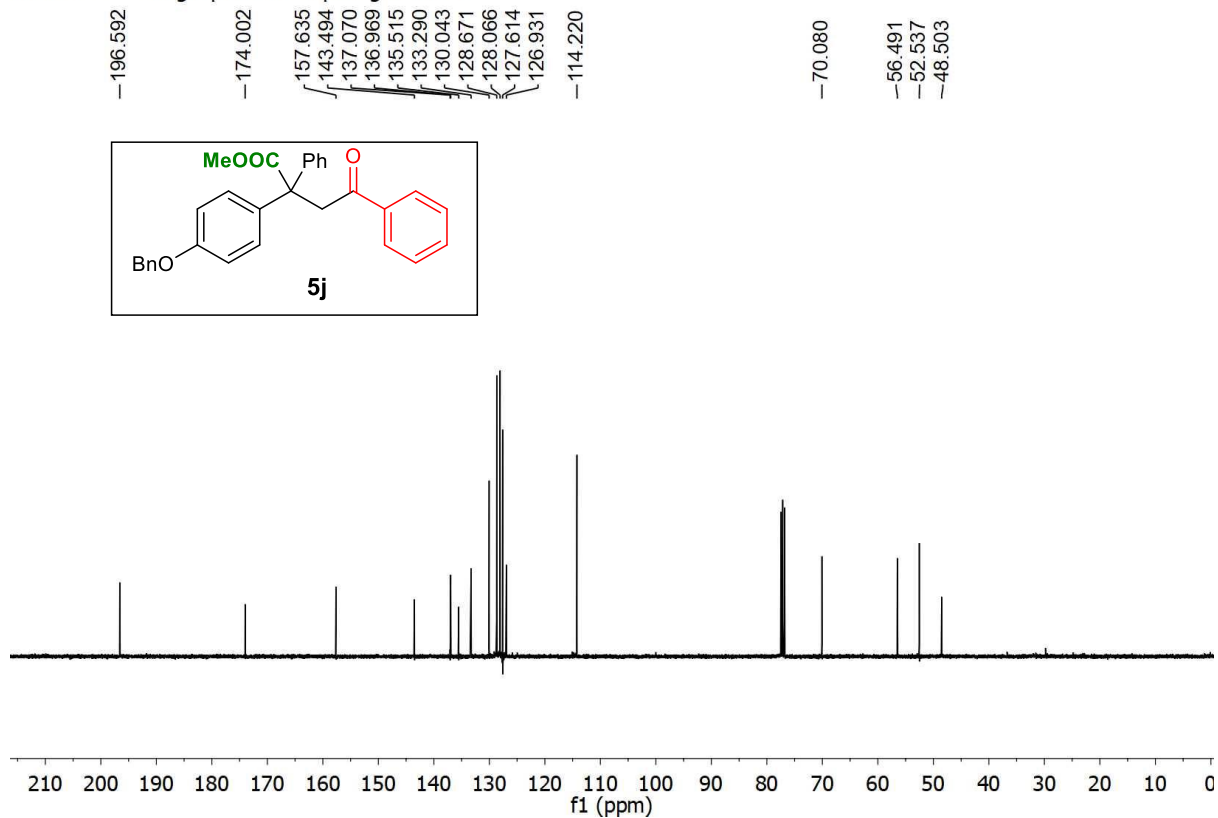
<sup>13</sup>C spectra of **5i**

SNAN-1323 — single\_pulse



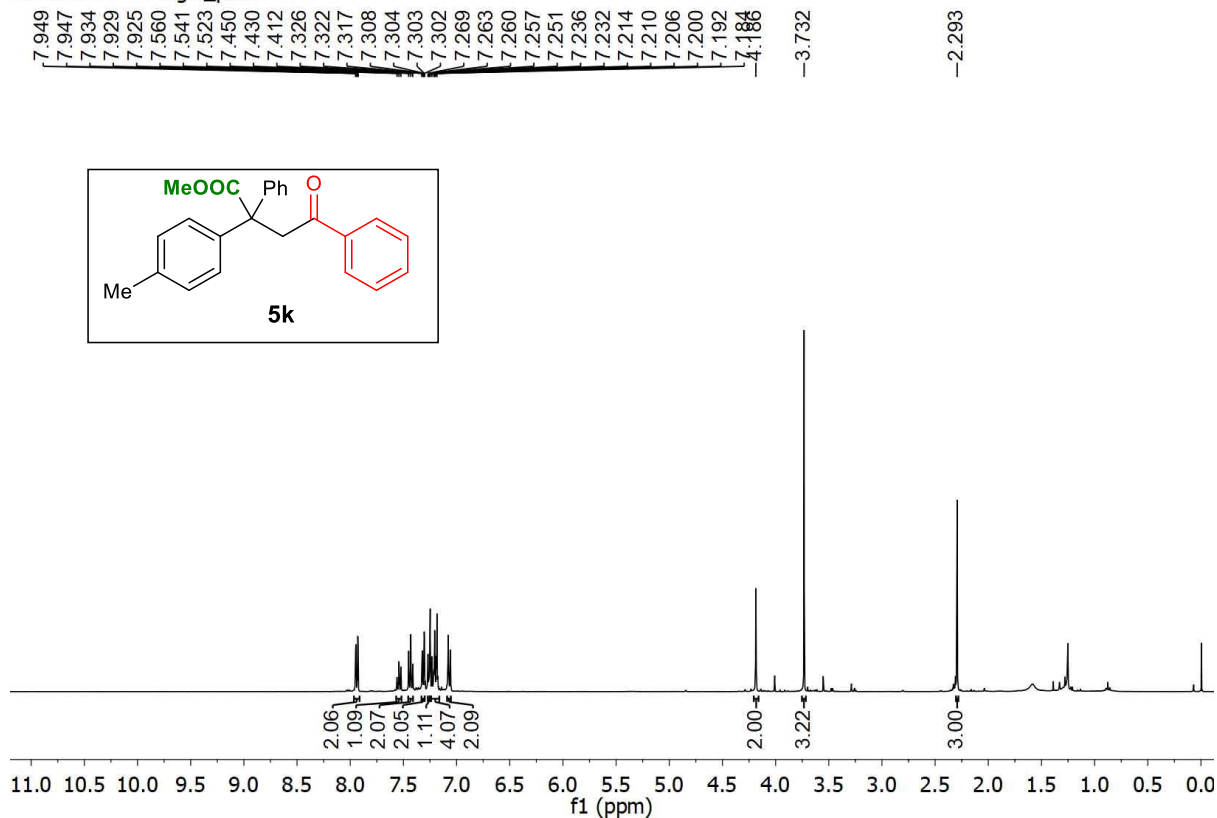
<sup>1</sup>H spectra of **5j**

SNAN-1323 — single pulse decoupled gated NOE



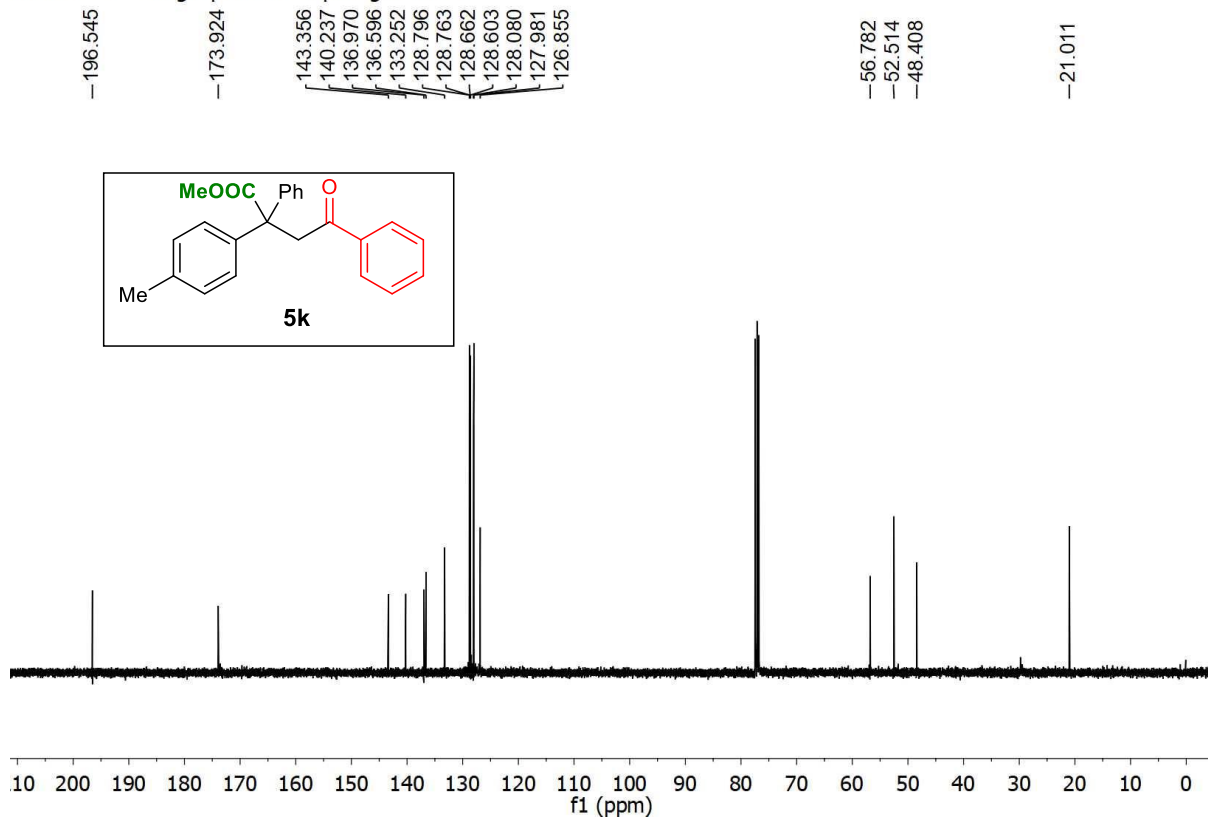
<sup>13</sup>C spectra of **5j**

SNAN-1325 — single\_pulse



<sup>1</sup>H spectra of **5k**

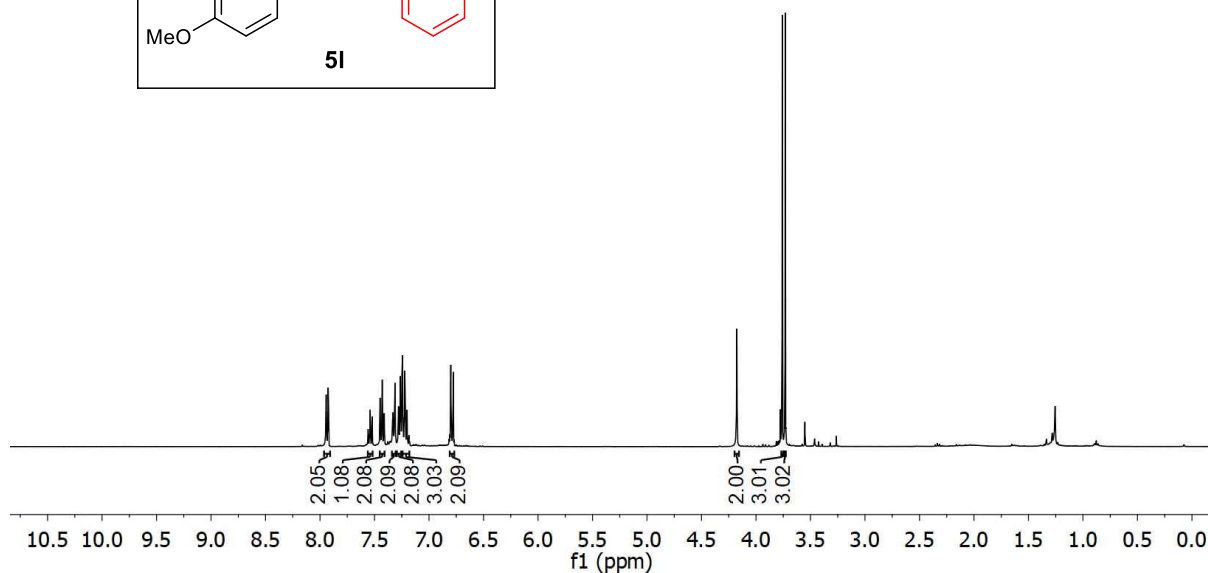
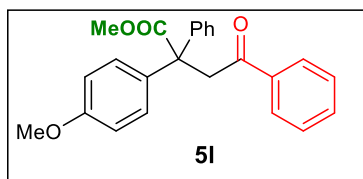
SNAN-1325 — single\_pulse decoupled gated NOE



<sup>13</sup>C spectra of **5k**

SNAN-1337 1 — single\_pulse

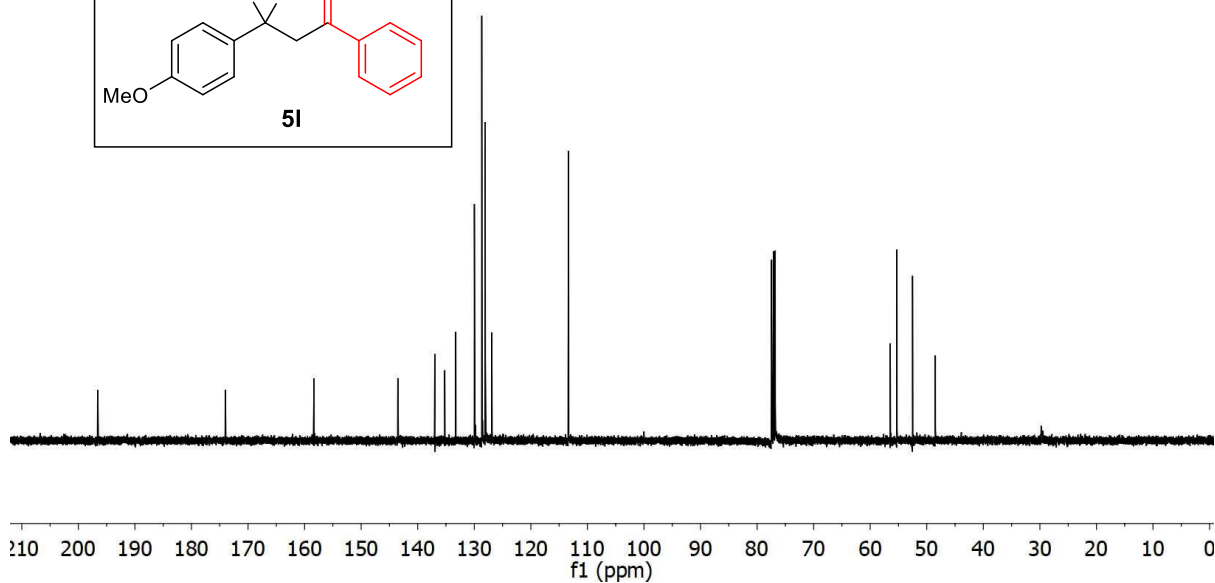
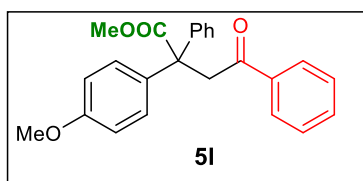
7.945  
7.944  
7.926  
7.922  
7.559  
7.540  
7.522  
7.449  
7.429  
7.411  
7.336  
7.333  
7.330  
7.325  
7.312  
7.283  
7.280  
7.275  
7.262  
7.258  
7.243  
7.237  
7.226  
7.223  
7.220  
7.215  
7.208  
7.201  
7.195  
7.184  
6.809  
6.800  
6.795  
6.783  
6.778  
6.770  
4.176  
3.759  
3.732



<sup>1</sup>H spectra of **5I**

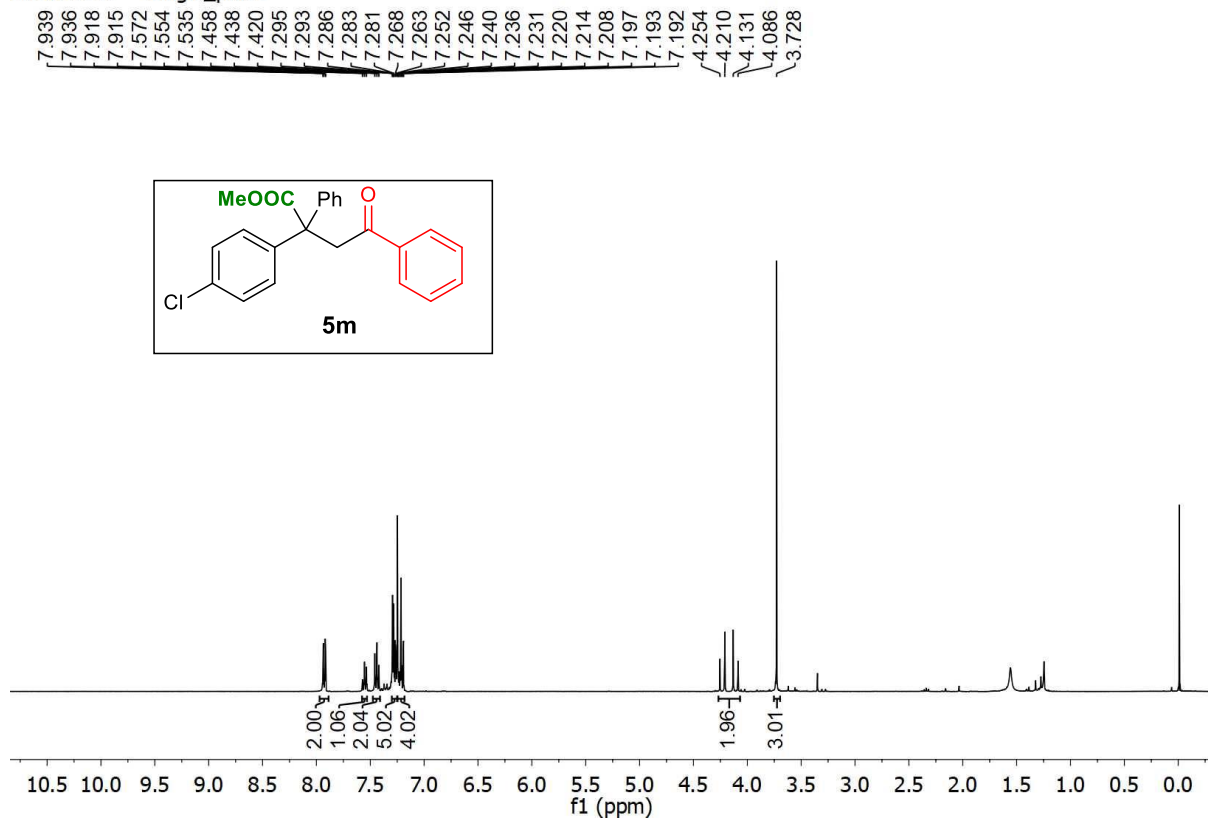
SNAN-1337 1 — single pulse decoupled gated NOE

-196.581  
-174.007  
-158.317  
143.515  
136.973  
135.226  
133.271  
129.986  
128.675  
128.071  
128.044  
126.905  
-113.334  
56.456  
55.274  
52.514  
48.486



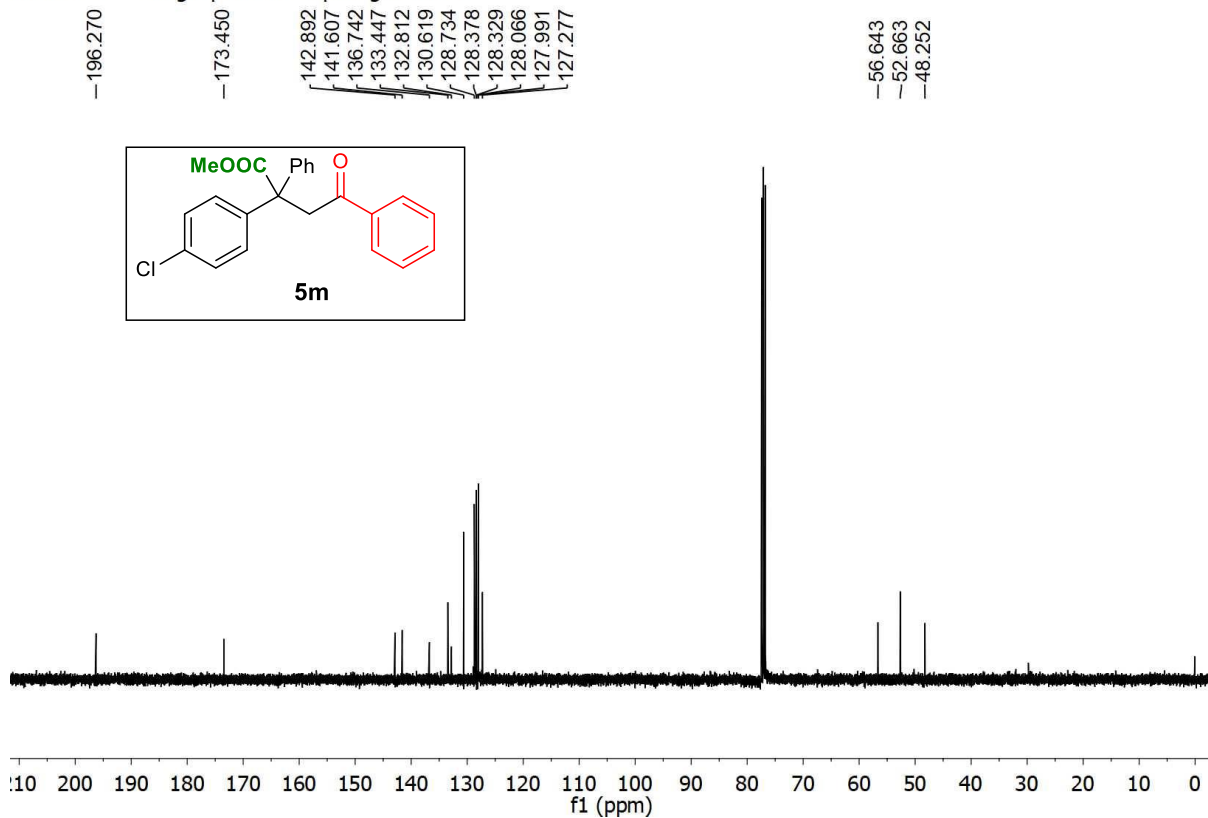
<sup>13</sup>C spectra of **5I**

SNAN-1352 — single\_pulse



<sup>1</sup>H spectra of **5m**

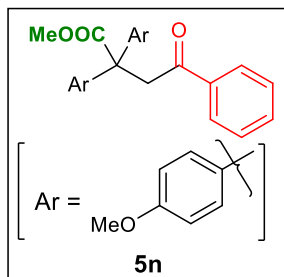
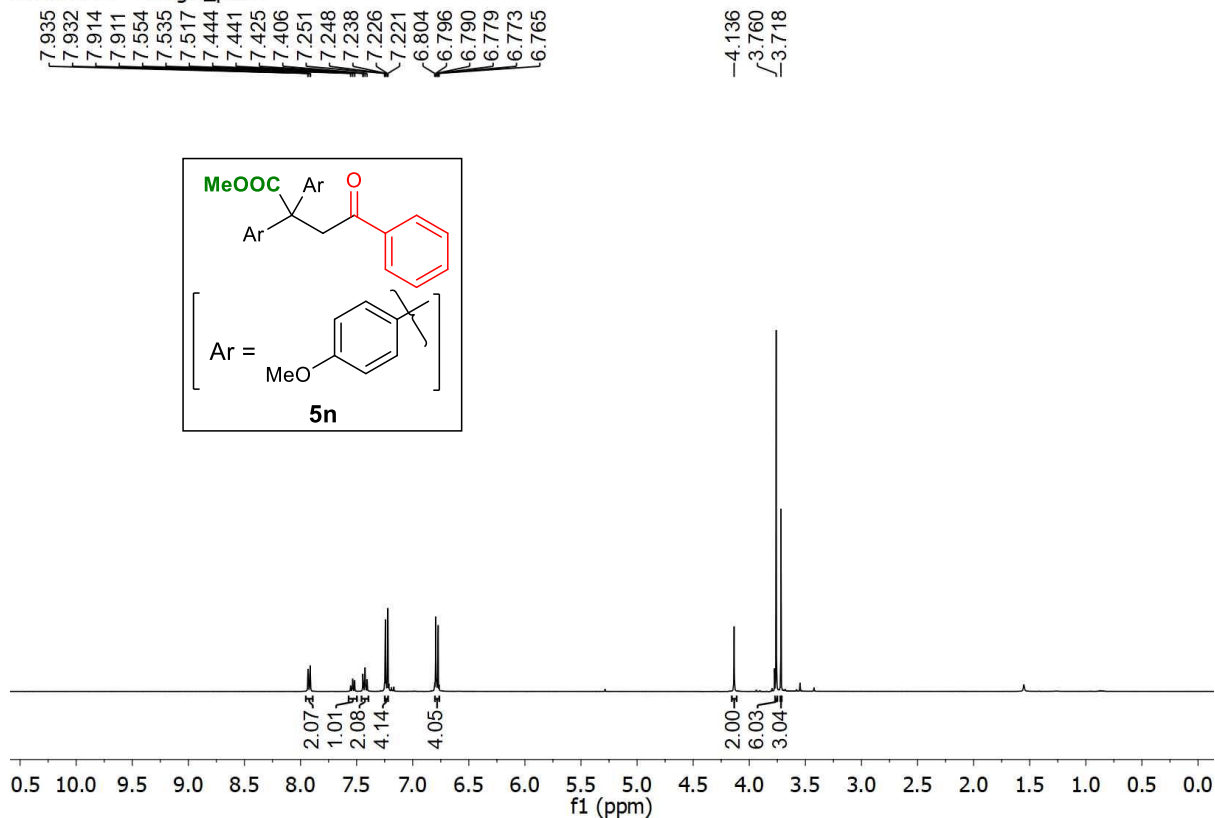
SNAN-1352 — single pulse decoupled gated NOE



<sup>13</sup>C spectra of **5m**

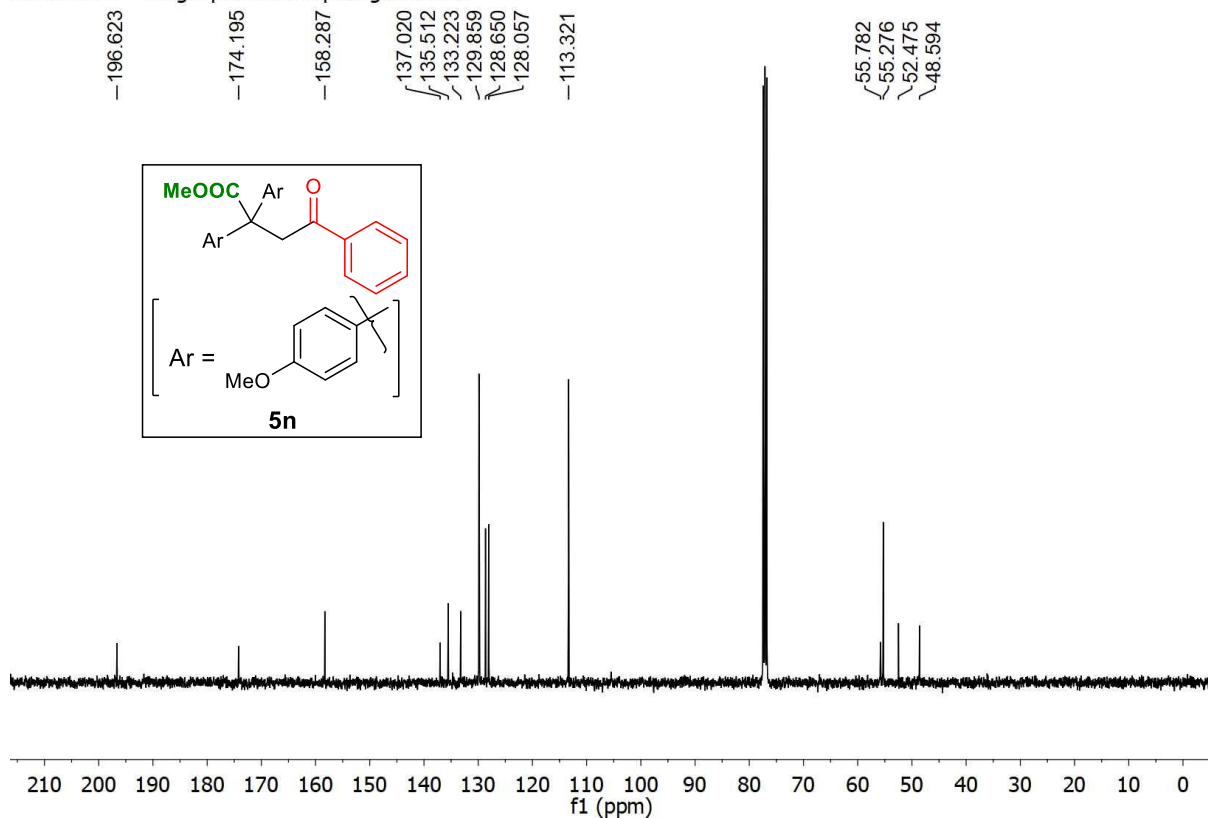


SNAN-1664 — single\_pulse



<sup>1</sup>H spectra of **5n**

SNAN-1664 — single\_pulse decoupled gated NOE

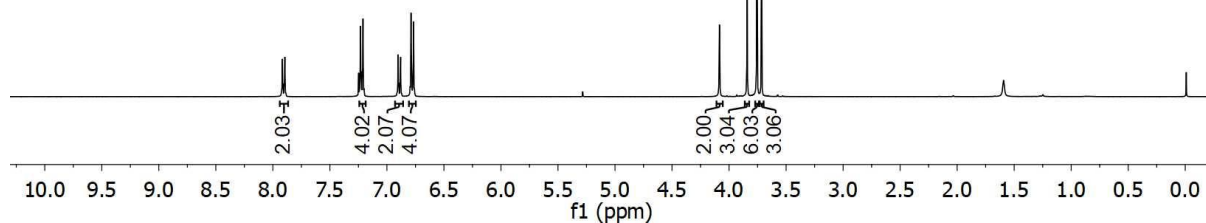
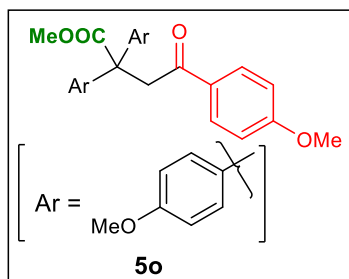


<sup>13</sup>C spectra of **5n**

SNAN-1350 — single\_pulse

7.917  
7.895  
7.232  
7.210  
6.902  
6.880  
6.789  
6.766

4.084  
3.842  
3.756  
3.715

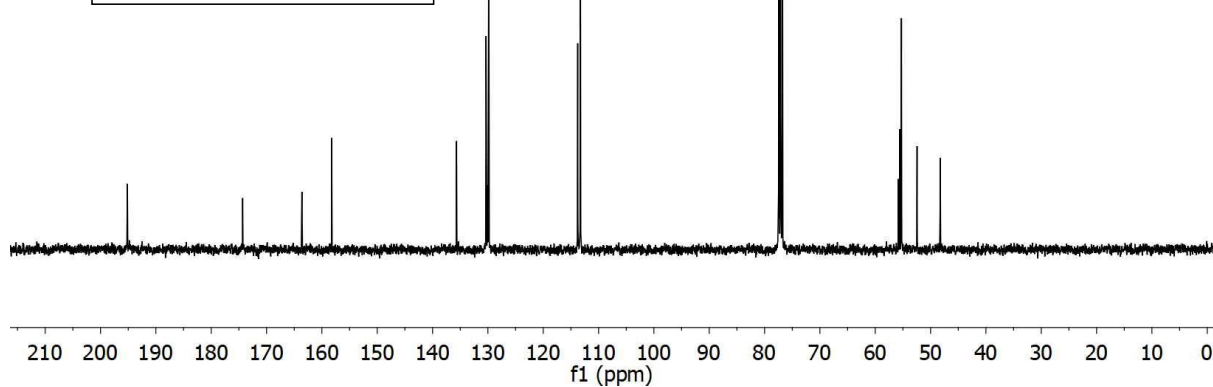
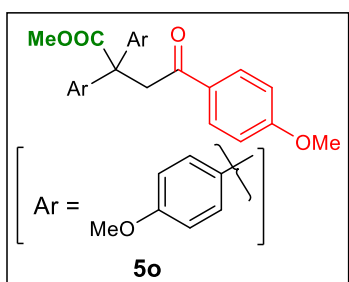


<sup>1</sup>H spectra of **5o**

SNAN-1350 — single pulse decoupled gated NOE

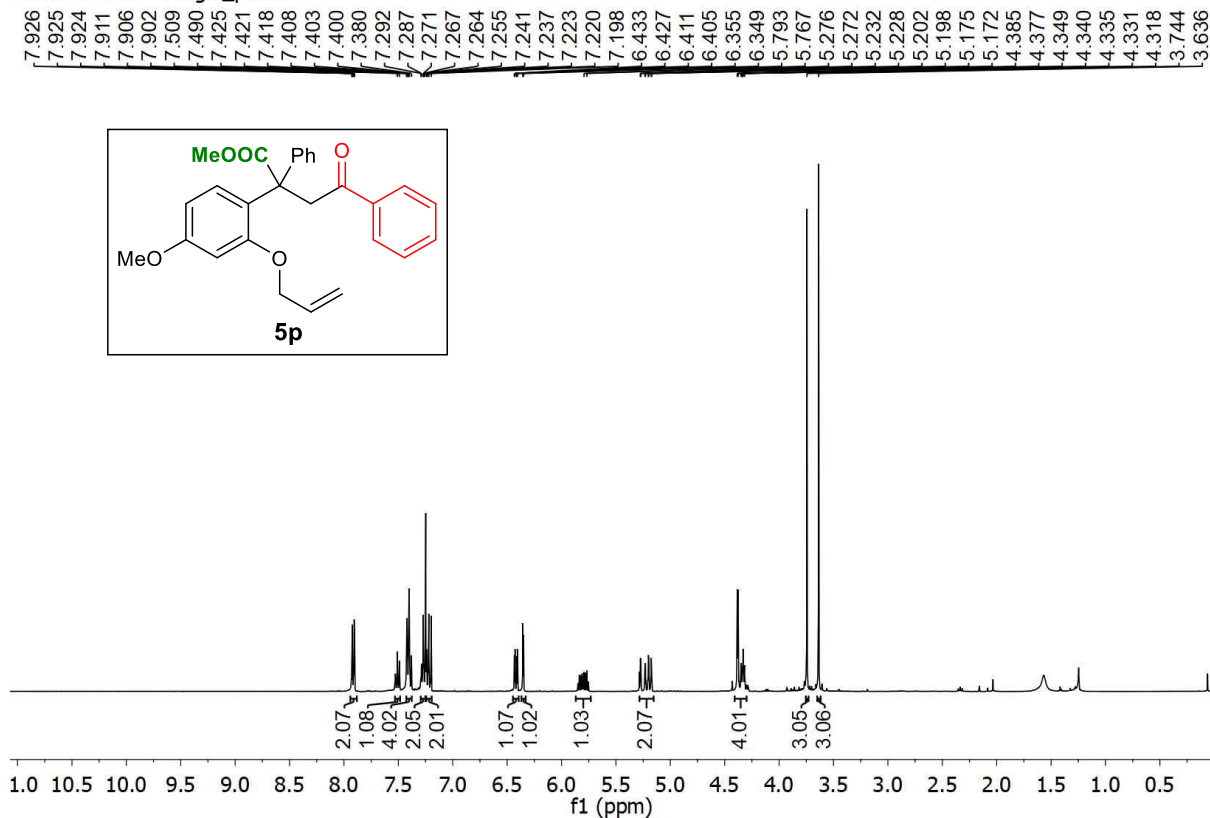
195.155  
174.320  
163.606  
158.233  
135.663  
130.337  
130.099  
129.866  
113.767  
113.289

55.808  
55.567  
55.269  
52.443  
48.264



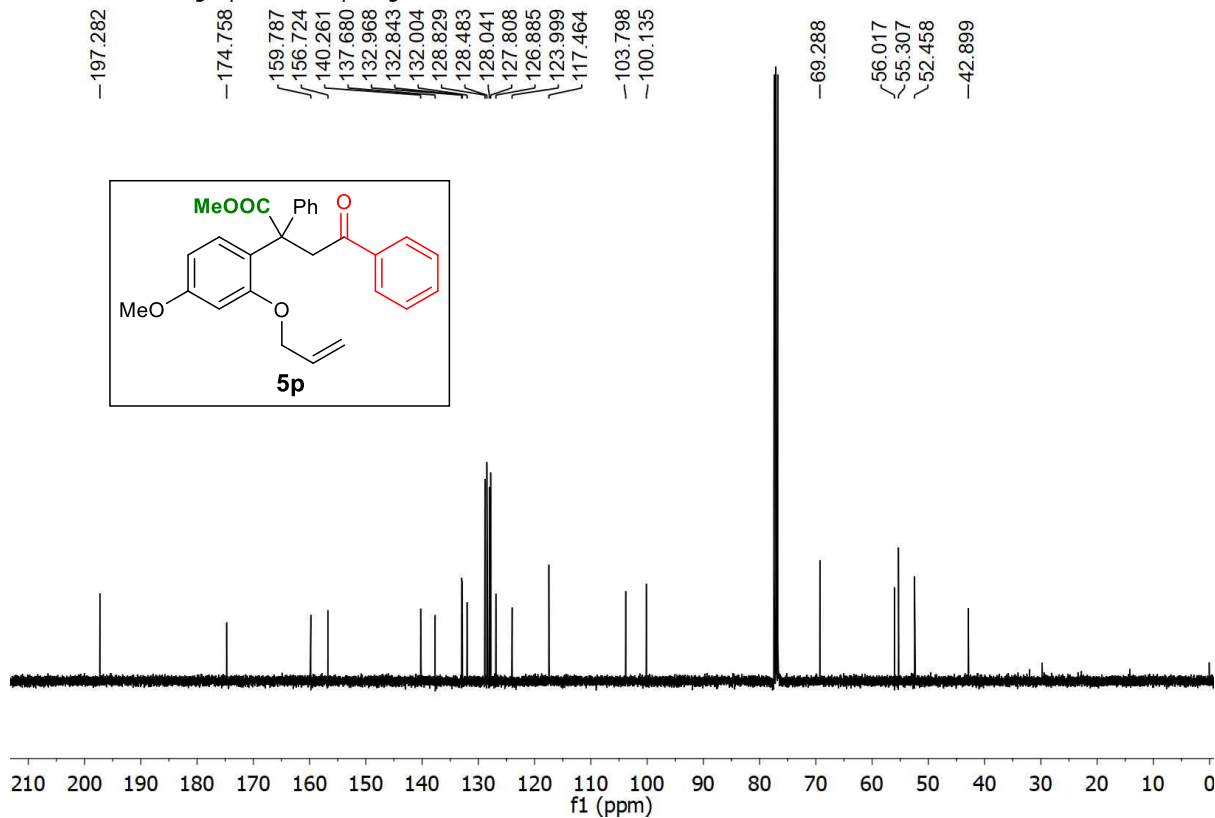
<sup>13</sup>C spectra of **5o**

SNAN-1326 1 — single\_pulse



<sup>1</sup>H spectra of 5p

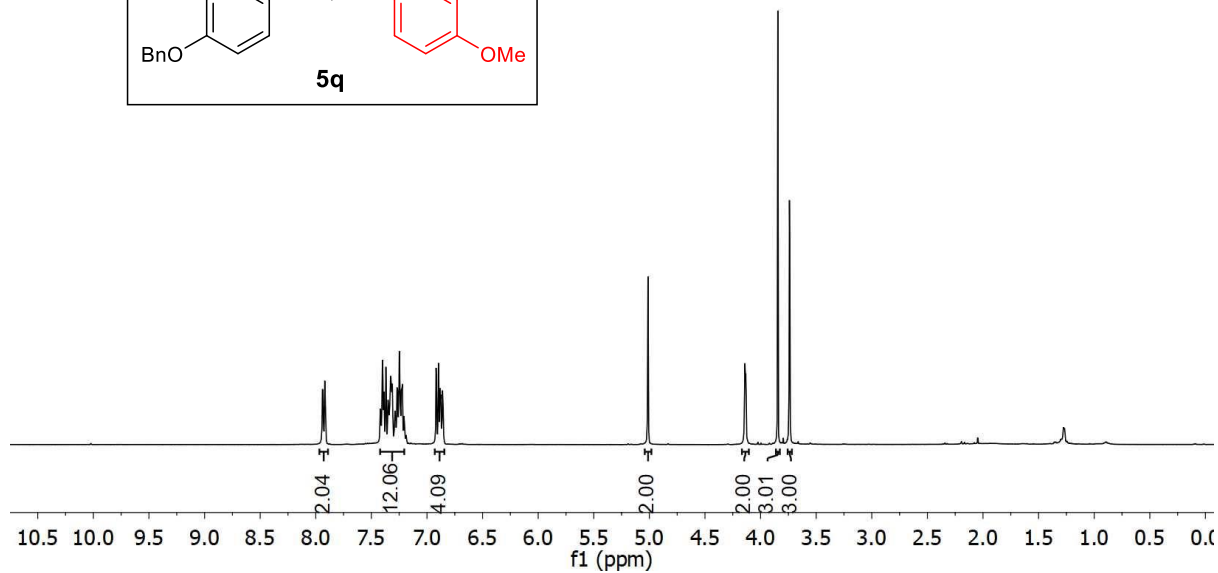
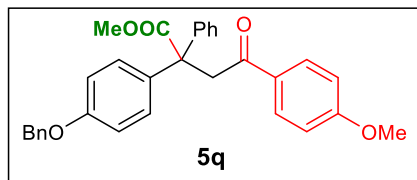
SNAN-1326 1 — single pulse decoupled gated NOE



<sup>13</sup>C spectra of 5p

SNAN-1341 — single\_pulse

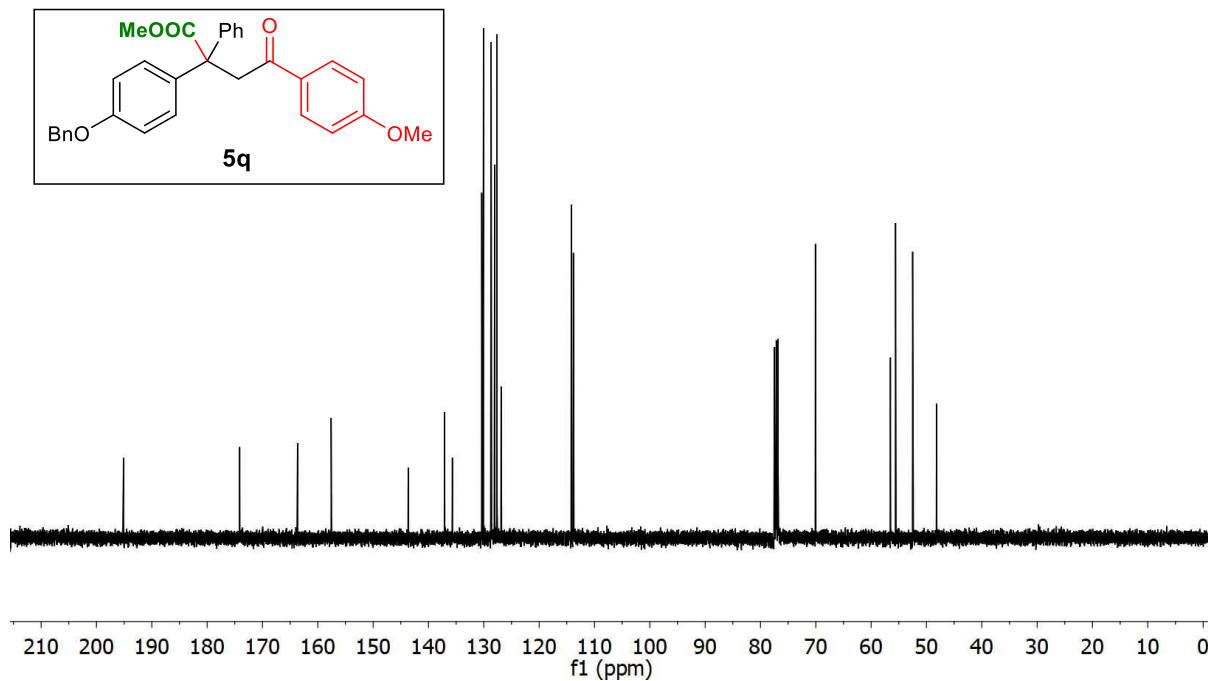
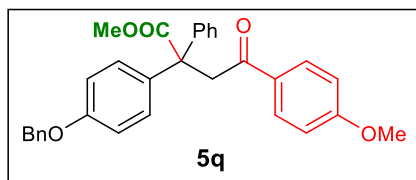
7.940  
7.936  
7.919  
7.914  
7.419  
7.399  
7.387  
7.369  
7.350  
7.332  
7.322  
7.312  
7.287  
7.269  
7.253  
7.203  
7.188  
6.918  
6.896  
6.883  
6.877  
6.861  
6.854  
5.013  
4.141  
4.139  
4.133  
3.845  
3.741



<sup>1</sup>H spectra of **5q**

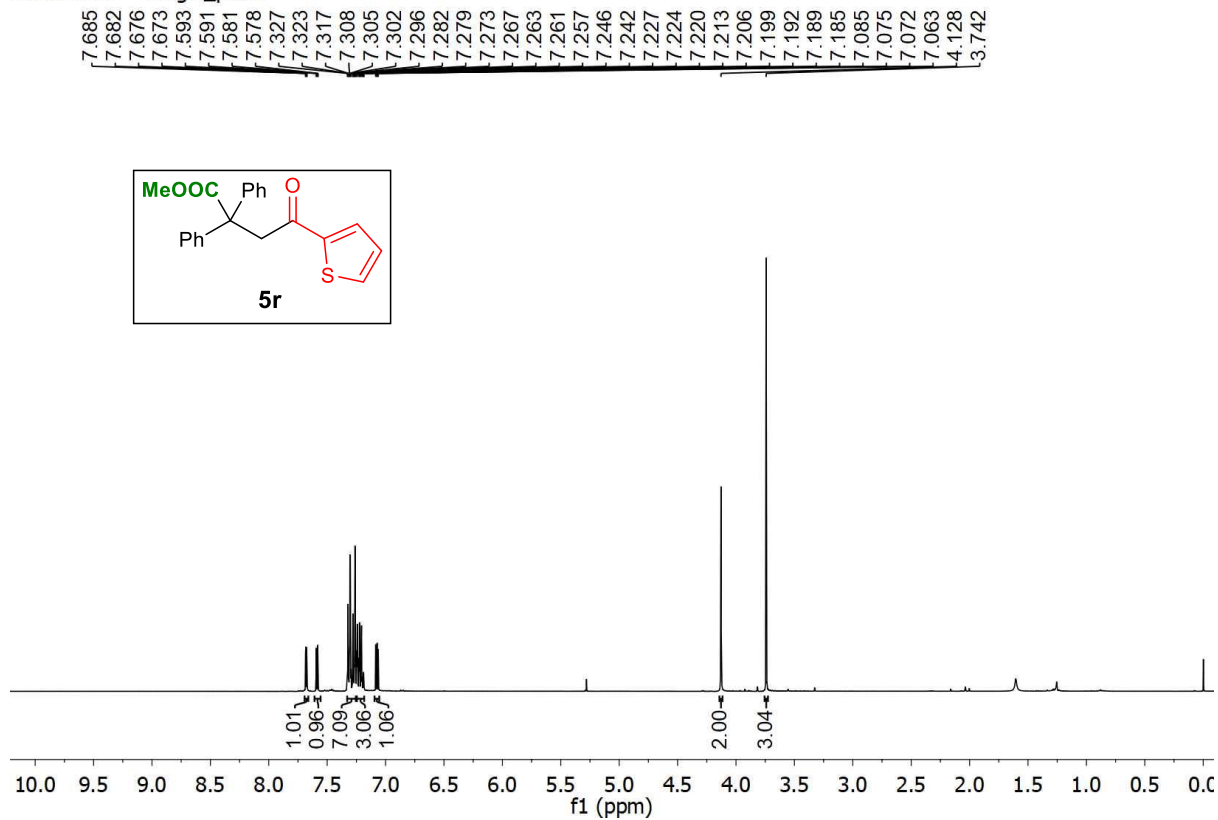
SNAN-1341 — single\_pulse decoupled gated NOE

195.110  
174.120  
163.663  
157.590  
143.656  
137.098  
135.679  
130.378  
130.061  
130.051  
128.708  
128.670  
128.037  
127.615  
126.868  
114.183  
113.811  
70.072  
56.520  
55.586  
52.498  
48.172



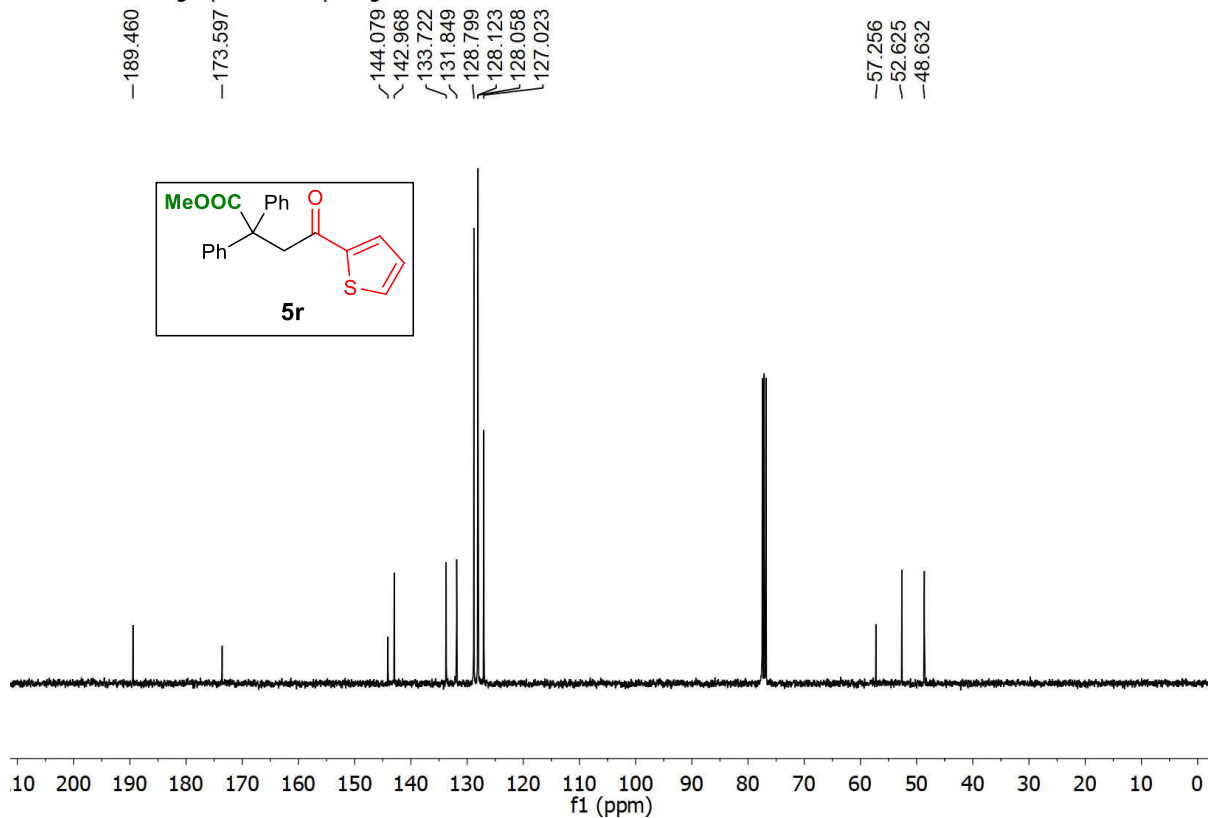
<sup>13</sup>C spectra of **5q**

SNAN-1333 — single\_pulse



<sup>1</sup>H spectra of **5r**

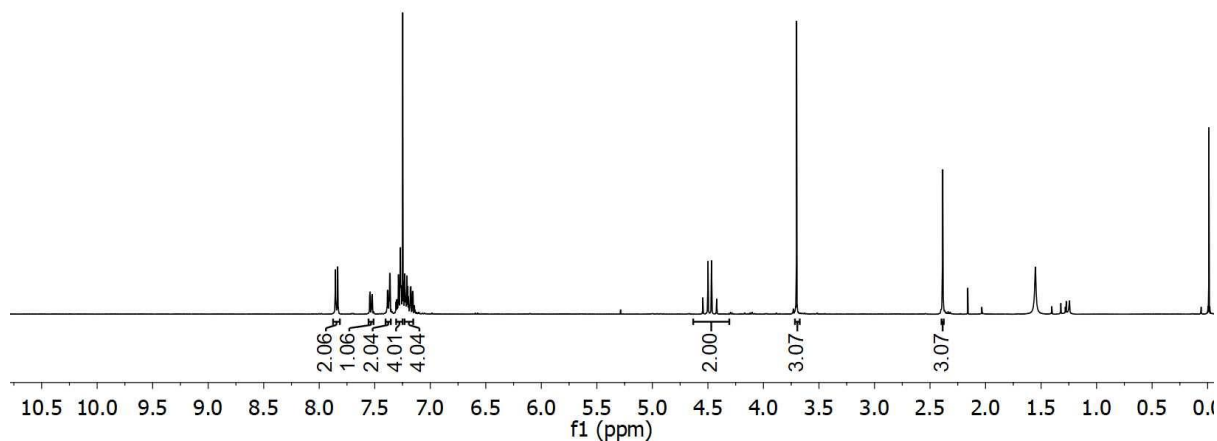
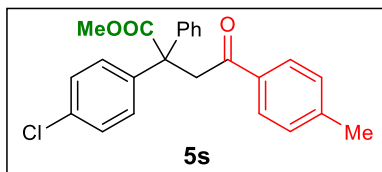
SNAN-1333 — single\_pulse decoupled gated NOE



<sup>13</sup>C spectra of **5r**

SNAN-1442 — single\_pulse

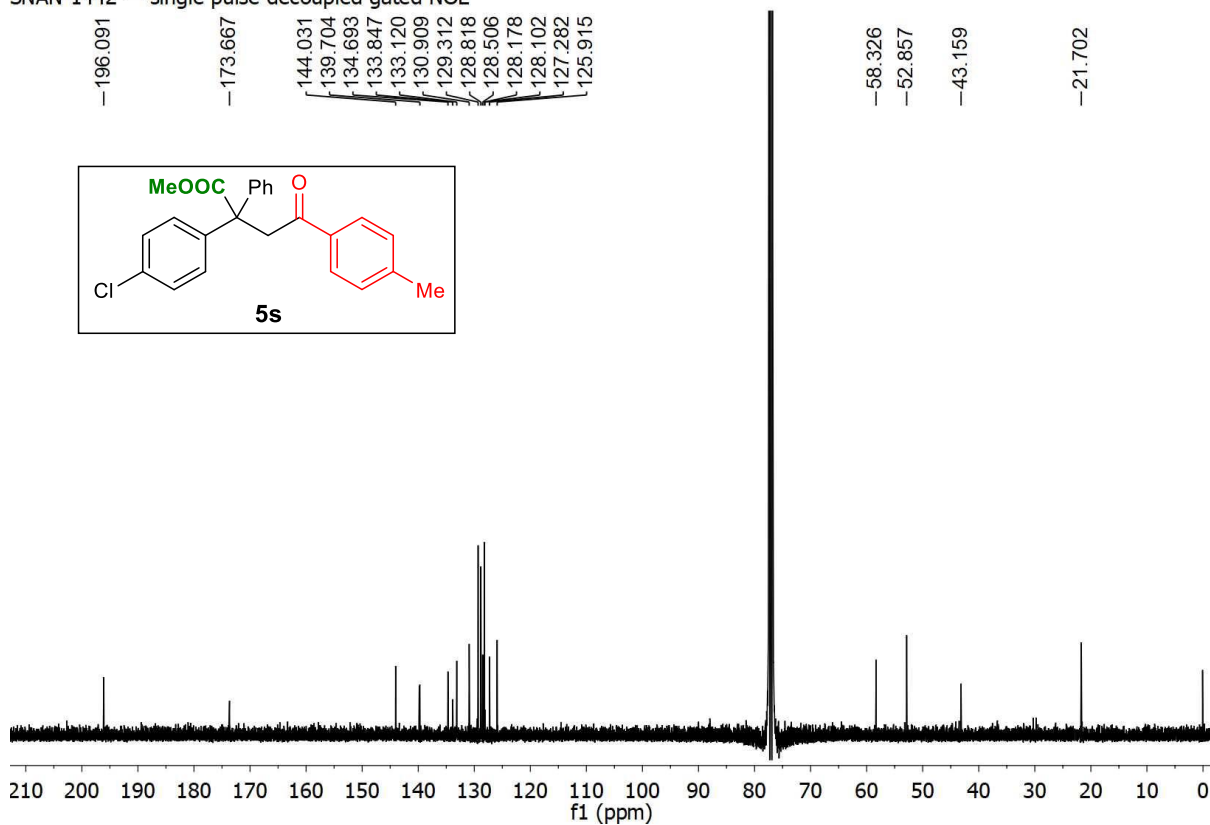
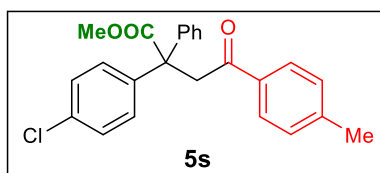
7.859  
7.854  
7.849  
7.838  
7.833  
7.828  
7.546  
7.541  
7.527  
7.522  
7.386  
7.380  
7.376  
7.365  
7.362  
7.354  
7.309  
7.301  
7.296  
7.288  
7.287  
7.283  
7.277  
7.273  
7.268  
7.265  
7.261  
7.260  
7.255  
7.251  
7.231  
7.230  
7.220  
7.215  
7.210  
7.209  
7.202  
7.197  
7.183  
7.180  
7.178  
7.174  
7.161  
7.156  
4.546  
4.499  
4.467  
4.421  
3.701  
2.385



<sup>1</sup>H spectra of **5s**

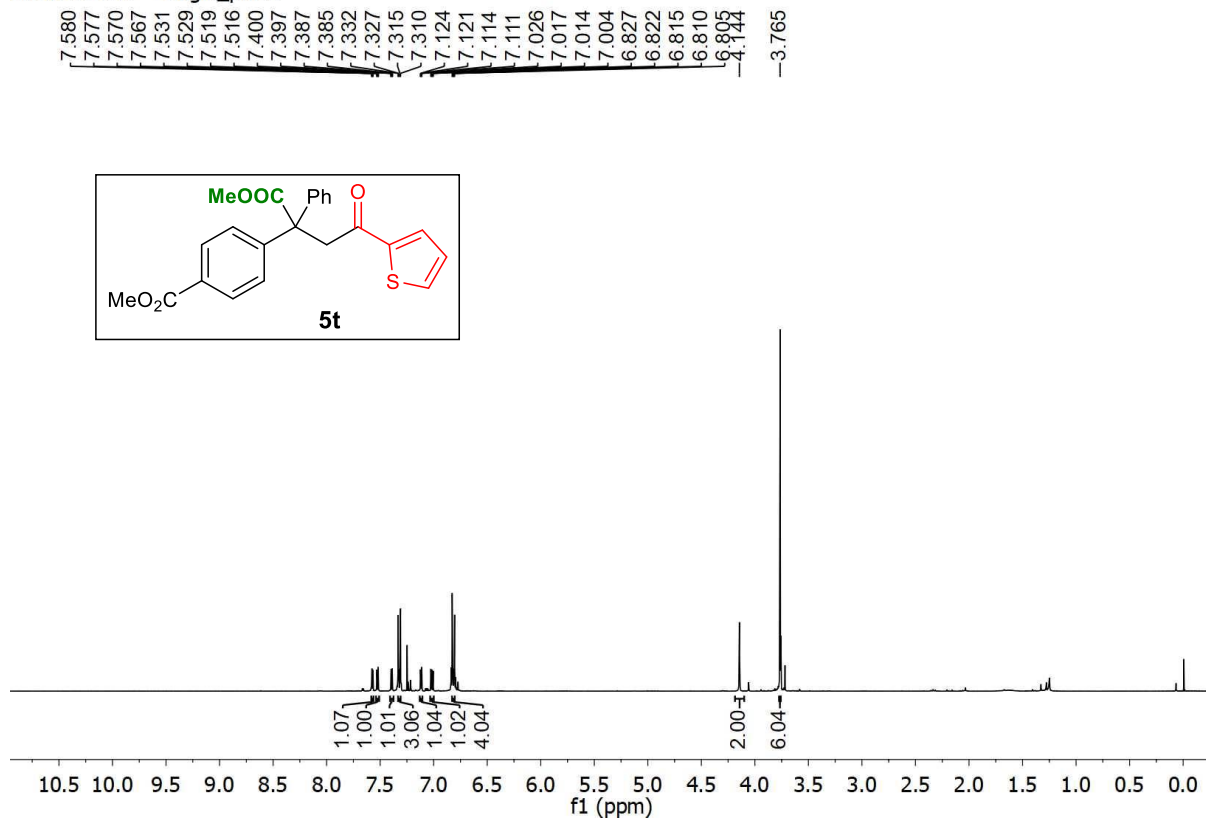
SNAN-1442 — single pulse decoupled gated NOE

-196.091  
-173.667  
144.031  
139.704  
134.693  
133.847  
133.120  
130.909  
129.312  
128.818  
128.506  
128.178  
128.102  
127.282  
125.915  
-58.326  
-52.857  
-43.159  
-21.702



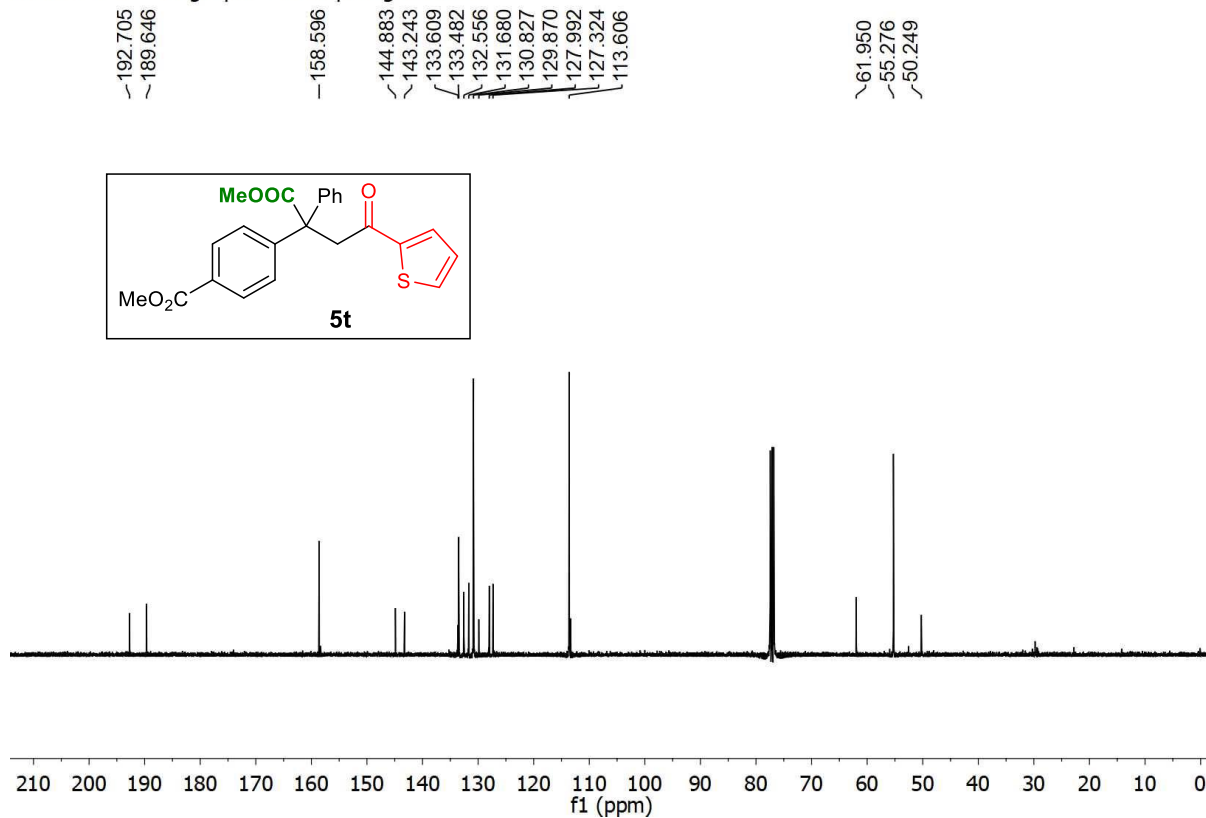
<sup>13</sup>C spectra of **5s**

SNAN-1343R — single\_pulse



<sup>1</sup>H spectra of **5t**

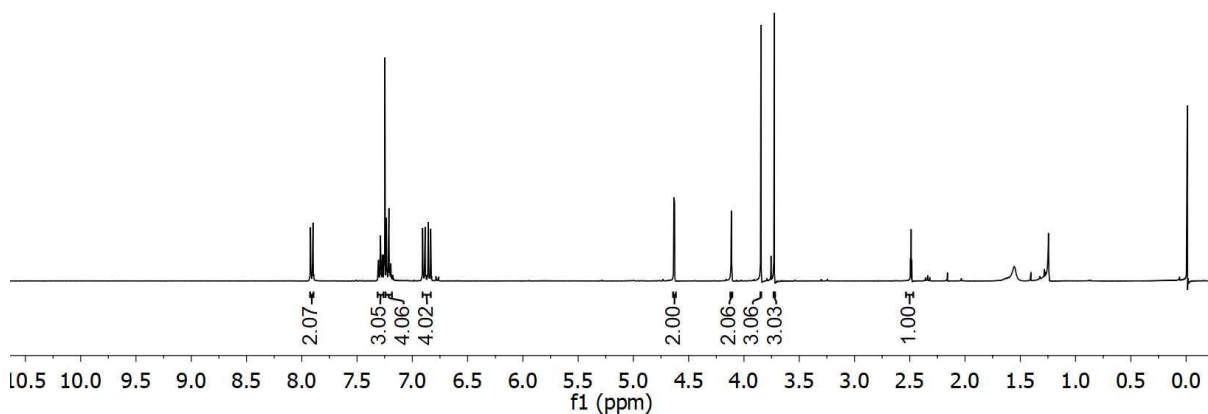
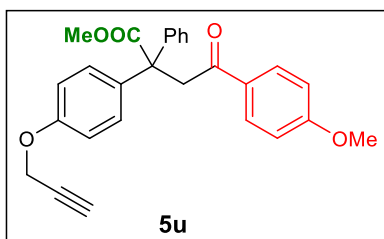
SNAN-1343R — single pulse decoupled gated NOE



<sup>13</sup>C spectra of **5t**

SNAN-1436 — single\_pulse

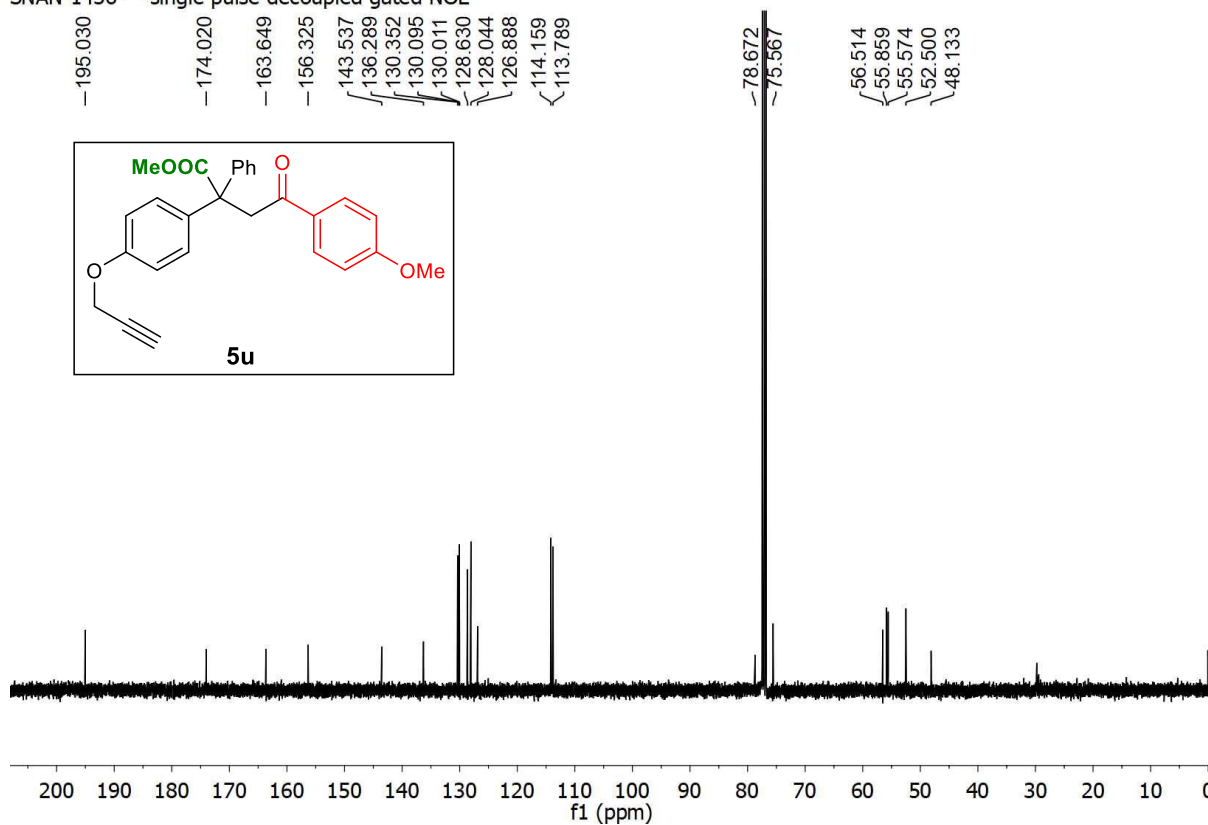
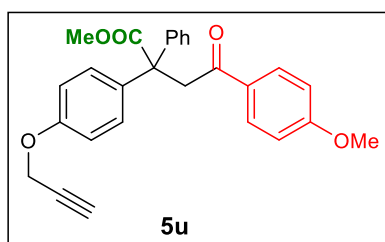
7.921  
7.916  
7.904  
7.899  
7.310  
7.305  
7.300  
7.291  
7.288  
7.284  
7.279  
7.273  
7.270  
7.265  
7.238  
7.235  
7.229  
7.218  
7.212  
7.201  
7.194  
6.907  
6.901  
6.890  
6.884  
6.877  
6.865  
6.856  
6.851  
6.839  
6.834  
4.634  
4.616  
4.114  
3.846  
3.726  
2.494  
2.488  
2.482



<sup>1</sup>H spectra of **5u**

SNAN-1436 — single\_pulse decoupled gated NOE

-195.030  
-174.020  
-163.649  
-156.325  
143.537  
136.289  
130.352  
130.095  
130.011  
128.630  
128.044  
126.888  
114.159  
113.789  
-78.672  
-75.567  
56.514  
55.859  
55.574  
52.500  
48.133

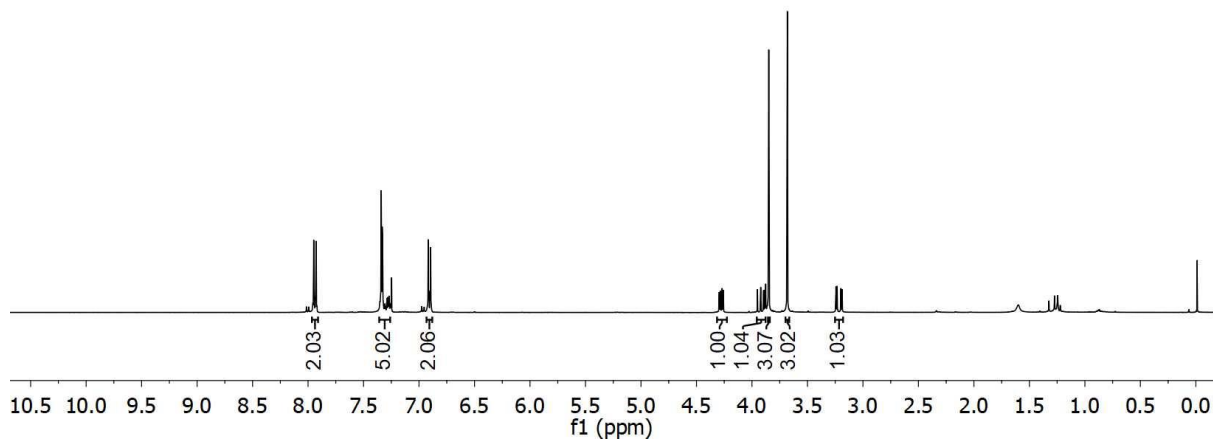
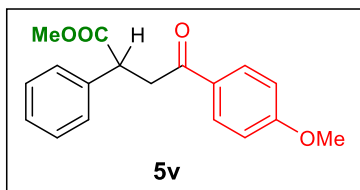


<sup>13</sup>C spectra of **5u**



SNAN-1369 — single\_pulse

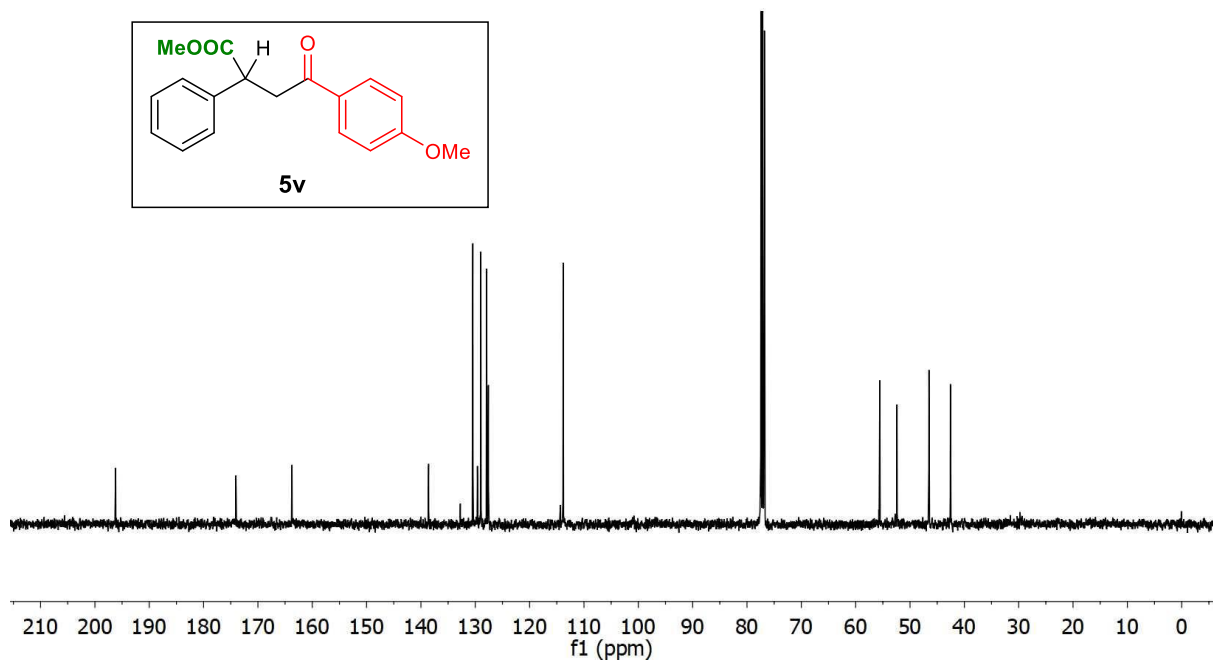
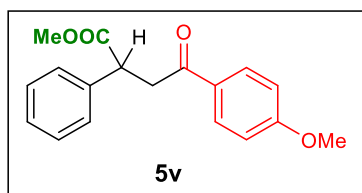
7.956  
7.949  
7.944  
7.932  
7.927  
7.919  
7.341  
7.332  
7.328  
7.309  
7.307  
7.300  
7.289  
7.282  
7.277  
7.273  
7.268  
7.261  
7.255  
6.925  
6.917  
6.912  
6.900  
6.895  
6.888  
4.295  
4.285  
4.269  
4.259  
3.951  
3.921  
3.895  
3.876  
3.848  
3.681  
3.243  
3.232  
3.198  
3.188



<sup>1</sup>H spectra of 5v

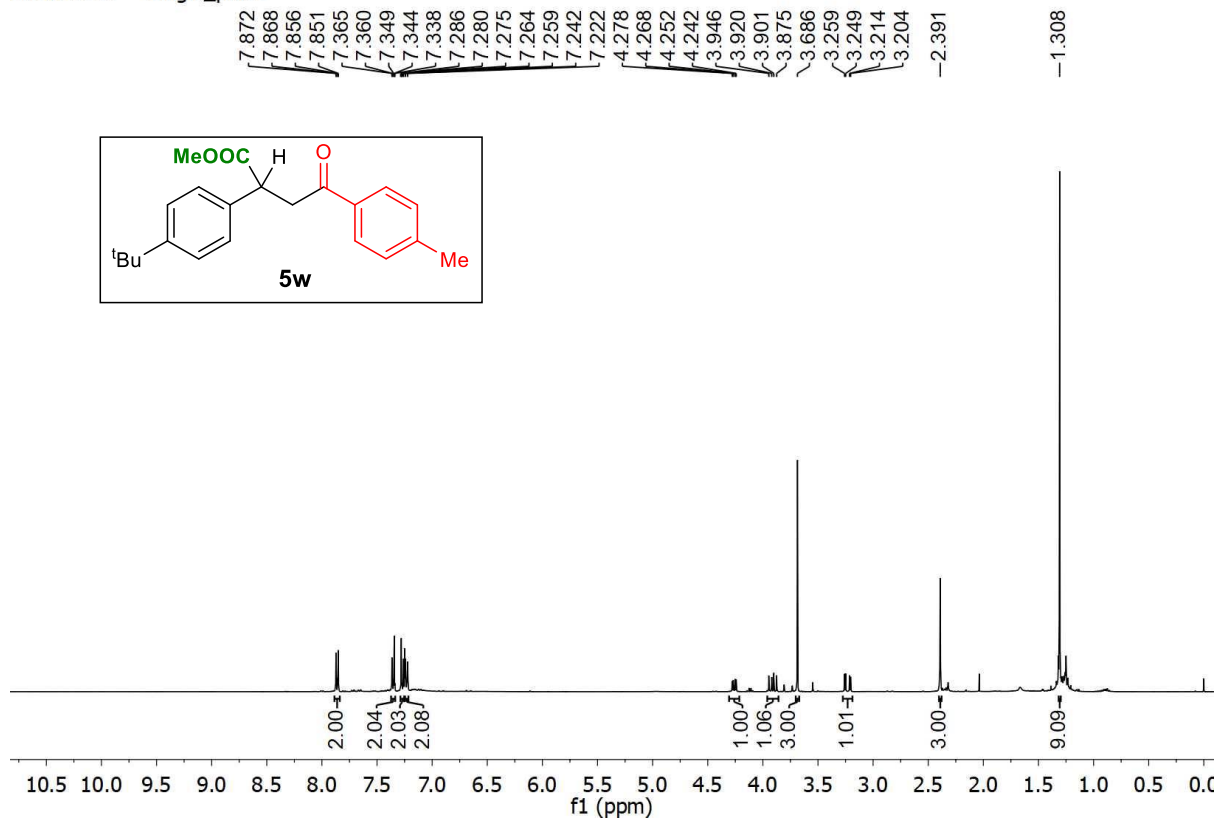
SNAN-1369 — single pulse decoupled gated NOE

-196.192  
-174.051  
-163.735  
138.591  
132.750  
130.451  
129.612  
128.975  
127.917  
127.588  
-113.823  
55.556  
52.394  
46.512  
42.562



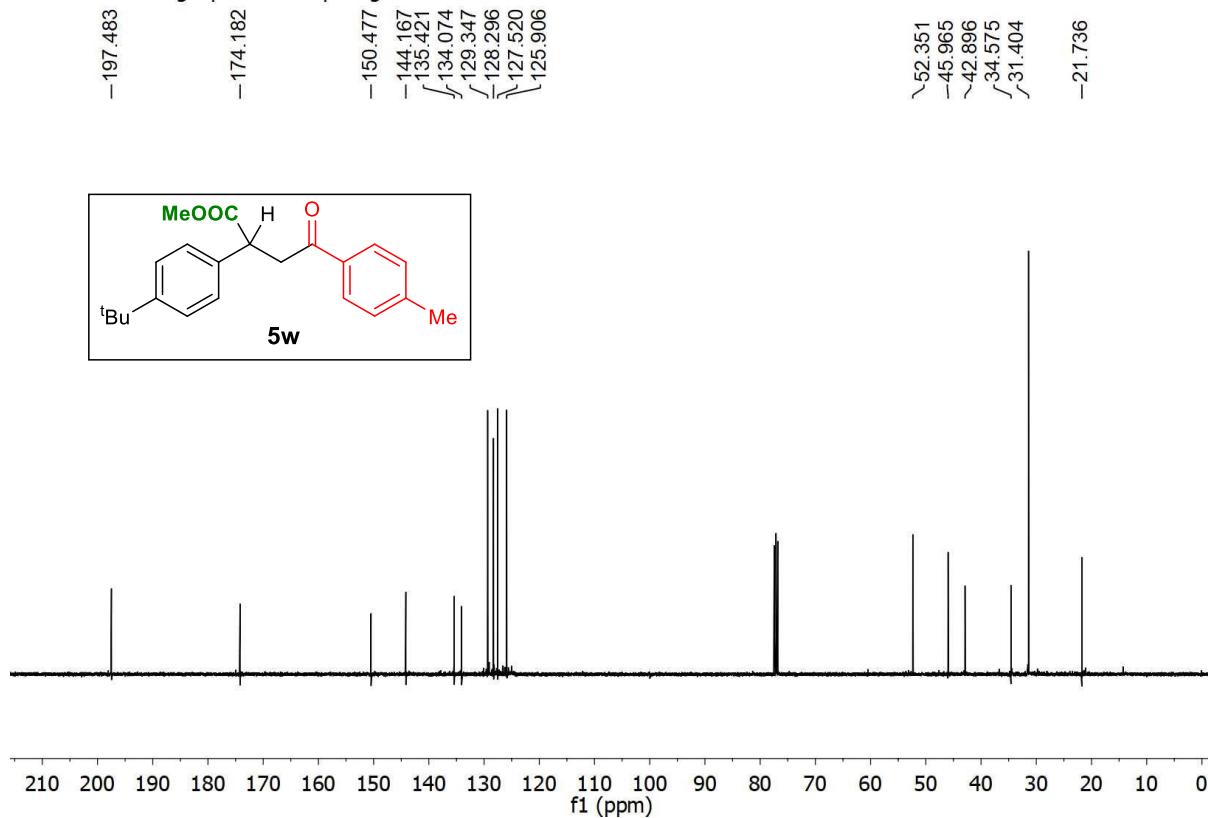
<sup>13</sup>C spectra of 5v

SNAN-1359 — single\_pulse



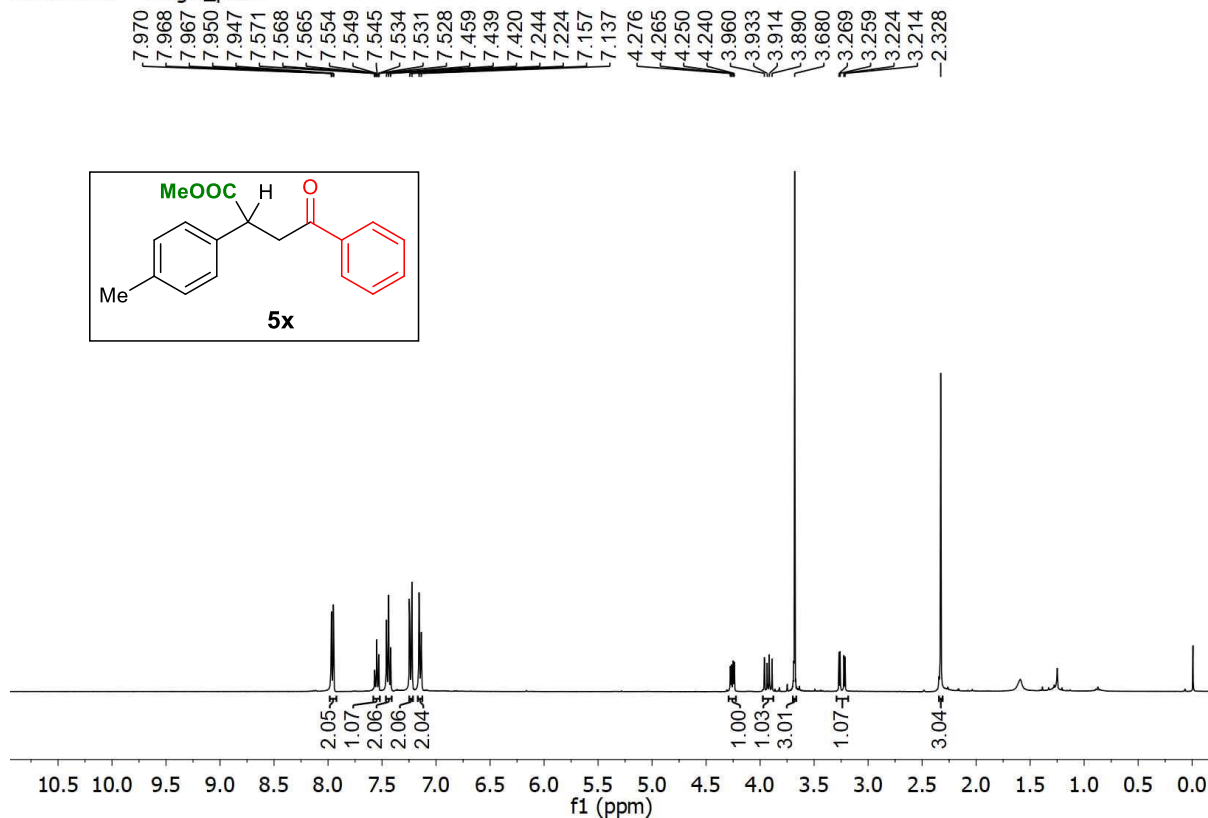
<sup>1</sup>H spectra of **5w**

SNAN-1359 — single pulse decoupled gated NOE



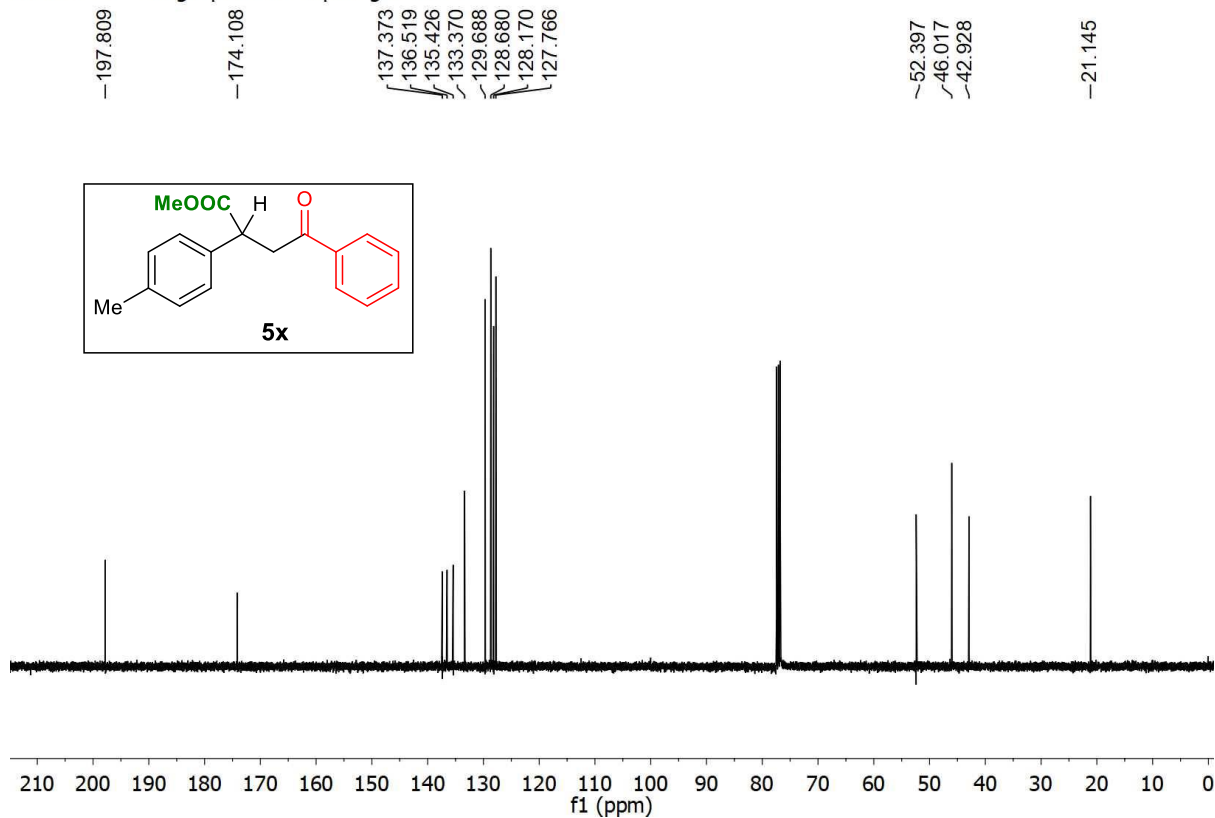
<sup>13</sup>C spectra of **5w**

SNAN-1331 — single\_pulse



<sup>1</sup>H spectra of 5x

SNAN-1331 — single\_pulse decoupled gated NOE

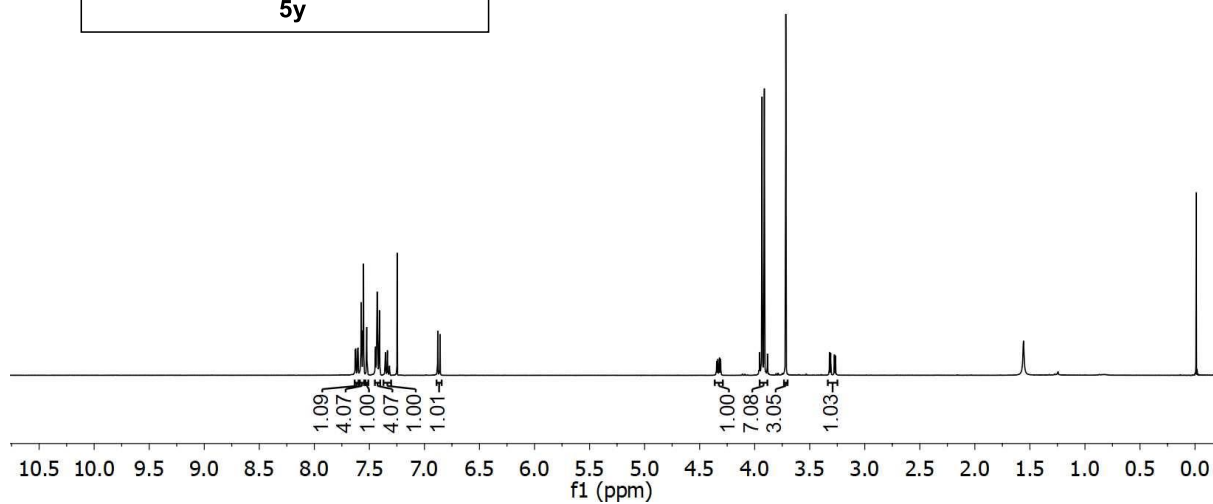
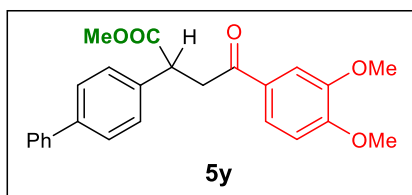


<sup>13</sup>C spectra of 5x

SNAN-1366 — single\_pulse

7.629  
7.624  
7.608  
7.603  
7.575  
7.573  
7.558  
7.554  
7.524  
7.519  
7.445  
7.430  
7.427  
7.414  
7.409  
7.354  
7.336  
6.879  
6.858

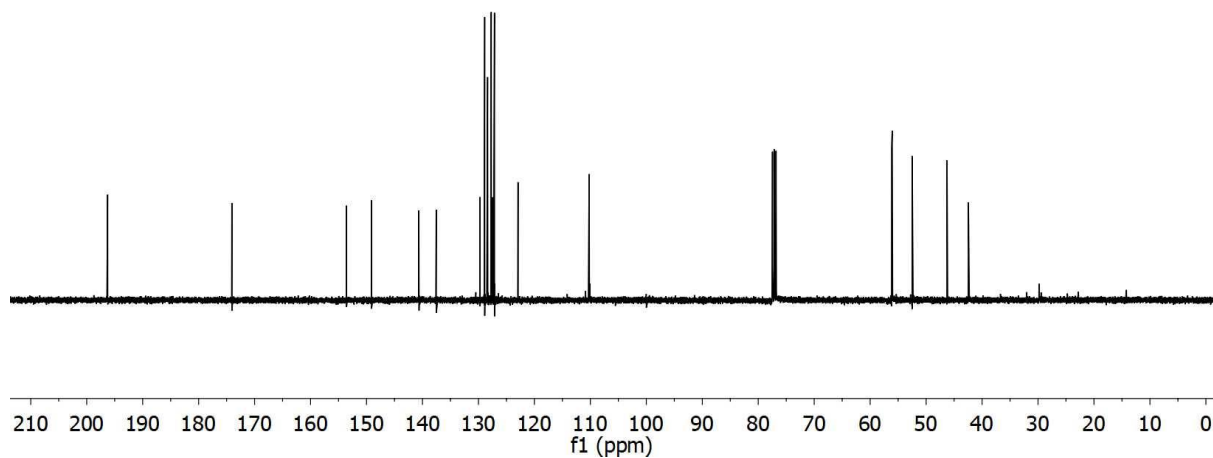
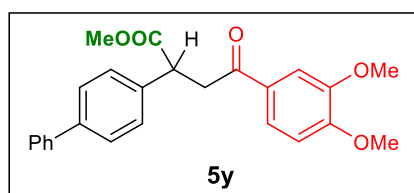
4.346  
4.335  
4.320  
4.310  
3.954  
3.933  
3.929  
3.912  
3.884  
3.717  
3.321  
3.310  
3.276  
3.265



<sup>1</sup>H spectra of **5y**

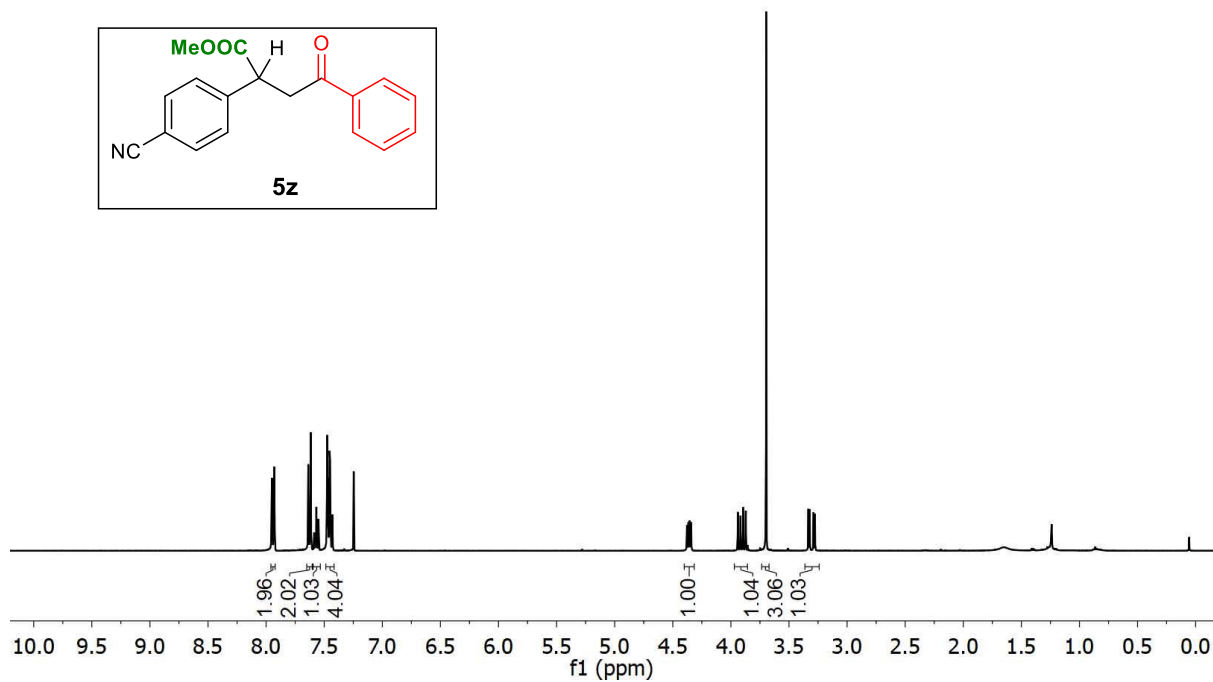
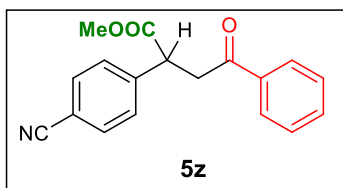
SNAN-1366 — single\_pulse decoupled gated NOE

196.262  
174.058  
153.595  
149.124  
140.649  
137.538  
129.726  
128.883  
128.357  
127.713  
127.483  
127.135  
122.921  
110.211  
110.120  
56.164  
56.060  
52.484  
46.266  
42.439



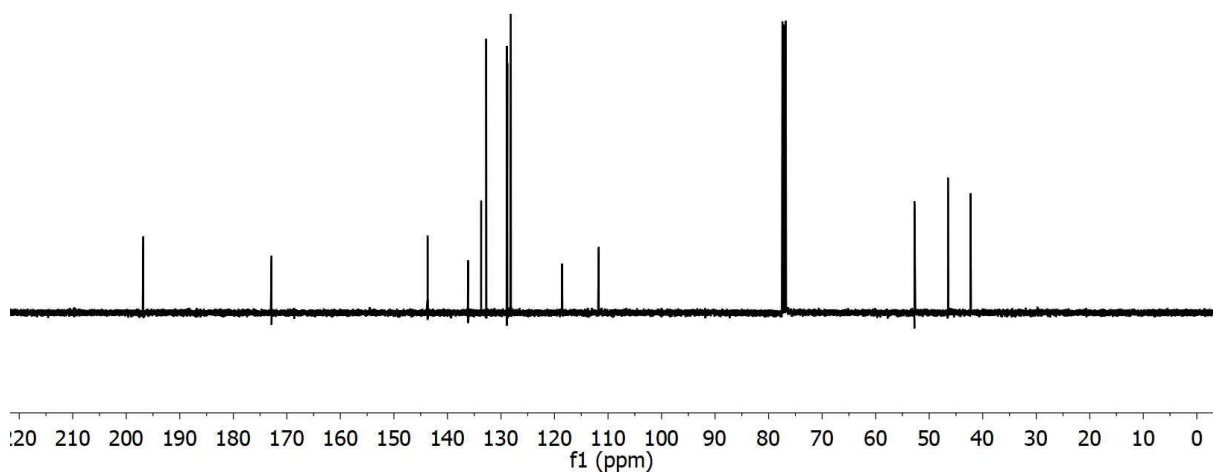
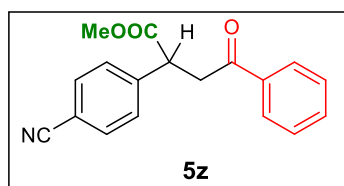
<sup>13</sup>C spectra of **5y**

SNAN-1255  
 single  
 7.9617  
 7.9375  
 7.9349  
 7.931  
 7.928  
 7.641  
 7.637  
 7.632  
 7.621  
 7.616  
 7.612  
 7.589  
 7.586  
 7.583  
 7.573  
 7.568  
 7.563  
 7.552  
 7.549  
 7.546  
 7.479  
 7.475  
 7.469  
 7.465  
 7.459  
 7.454  
 7.449  
 7.430  
 4.378  
 4.366  
 4.355  
 4.342  
 3.940  
 3.917  
 3.895  
 3.872  
 3.696  
 3.334  
 3.322  
 3.289  
 3.277



<sup>1</sup>H spectra of **5z**

SNAN-1255  
 -196.817  
 -172.867  
 143.710  
 136.152  
 133.696  
 132.759  
 128.914  
 128.802  
 128.153  
 118.581  
 111.724  
 52.752  
 46.490  
 42.265

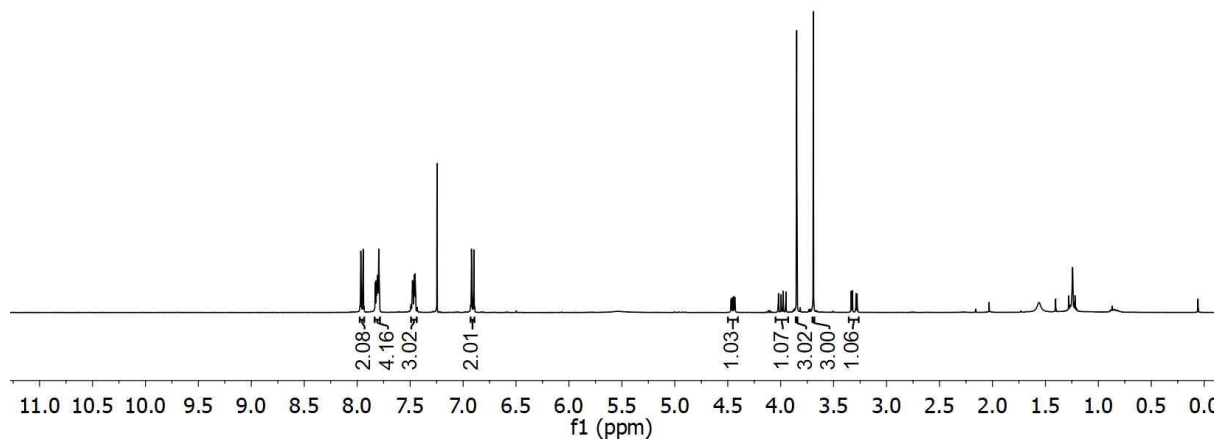
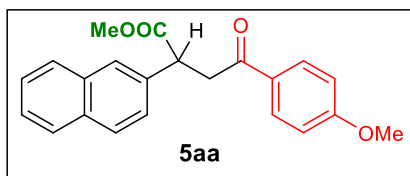


<sup>13</sup>C spectra of **5z**

SNAN-1269 1 — single\_pulse

7.966  
7.961  
7.948  
7.943  
7.829  
7.820  
7.807  
7.796  
7.476  
7.471  
7.462  
7.454  
7.447  
6.920  
6.915  
6.903  
6.898

4.468  
4.458  
4.443  
4.433  
4.022  
3.996  
3.977  
3.951  
3.849  
3.692  
3.334  
3.323  
3.289  
3.279

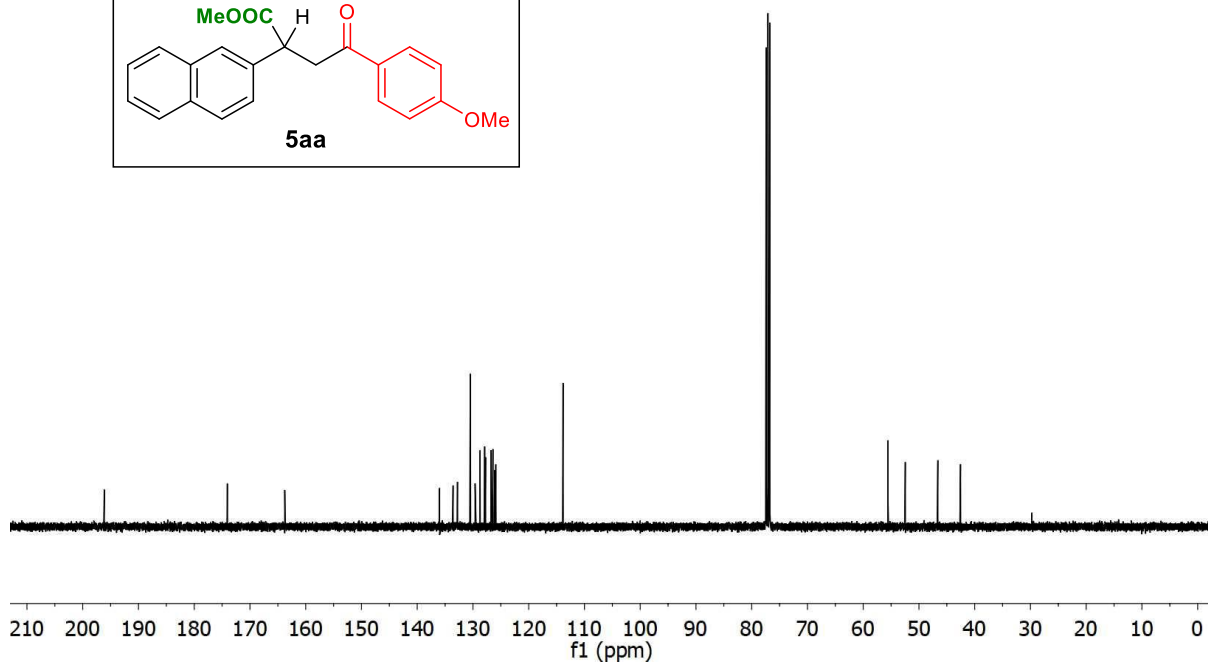
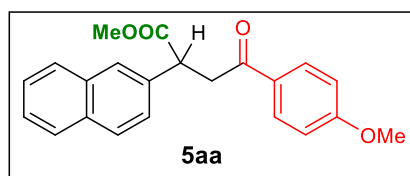


<sup>1</sup>H spectra of 5aa

SNAN-1269 1 — single pulse decoupled gated NOE

196.133  
174.059  
163.756  
135.993  
133.568  
132.777  
130.473  
129.615  
128.734  
127.895  
127.736  
126.760  
126.419  
126.104  
125.908  
113.838

55.556  
52.453  
46.617  
42.544

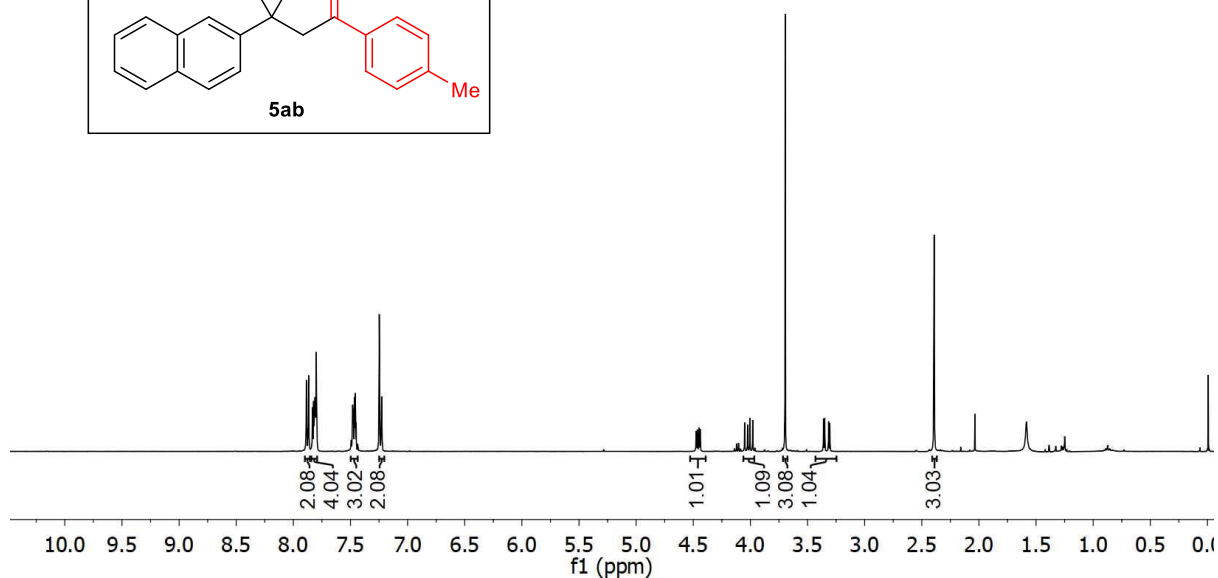
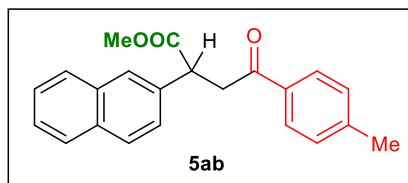


<sup>13</sup>C spectra of 5aa

SNAN-1451 — single\_pulse

7.886  
7.882  
7.870  
7.865  
7.832  
7.823  
7.810  
7.800  
7.493  
7.483  
7.481  
7.479  
7.474  
7.465  
7.457  
7.450  
7.438  
7.238  
7.227

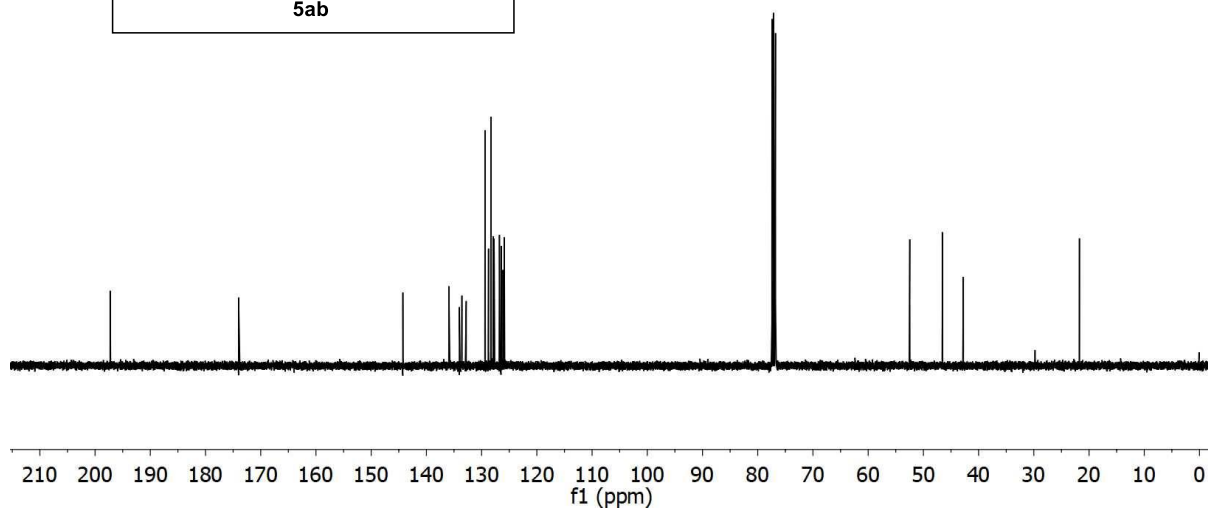
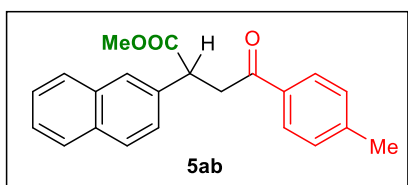
4.475  
4.465  
4.450  
4.439  
4.050  
4.025  
4.005  
3.980  
3.695  
3.360  
3.350  
3.315  
3.305  
-2.392



<sup>1</sup>H spectra of 5ab

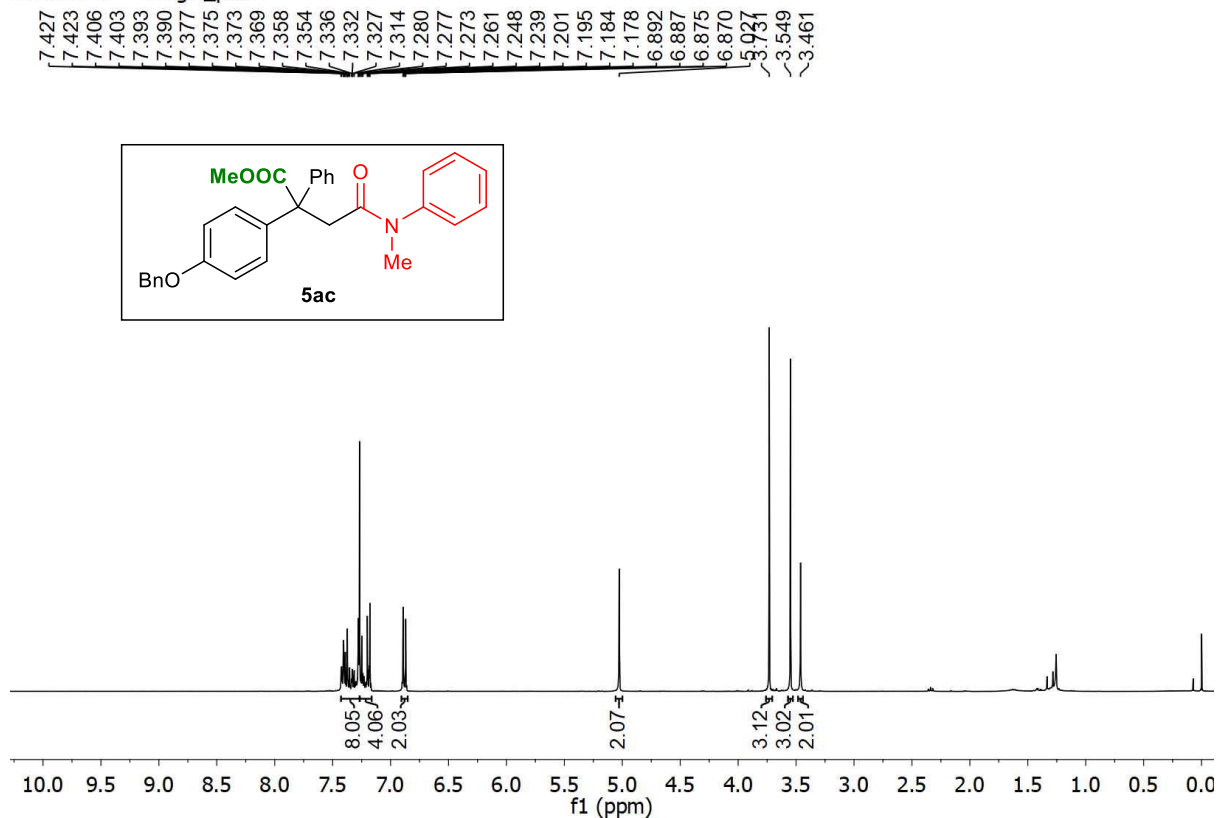
SNAN-1451 — single\_pulse decoupled gated NOE

-197.277  
-174.012  
144.261  
135.934  
134.034  
133.572  
132.787  
129.379  
128.751  
128.311  
127.900  
127.741  
126.774  
126.429  
126.120  
125.897  
52.466  
46.573  
42.761  
-21.745



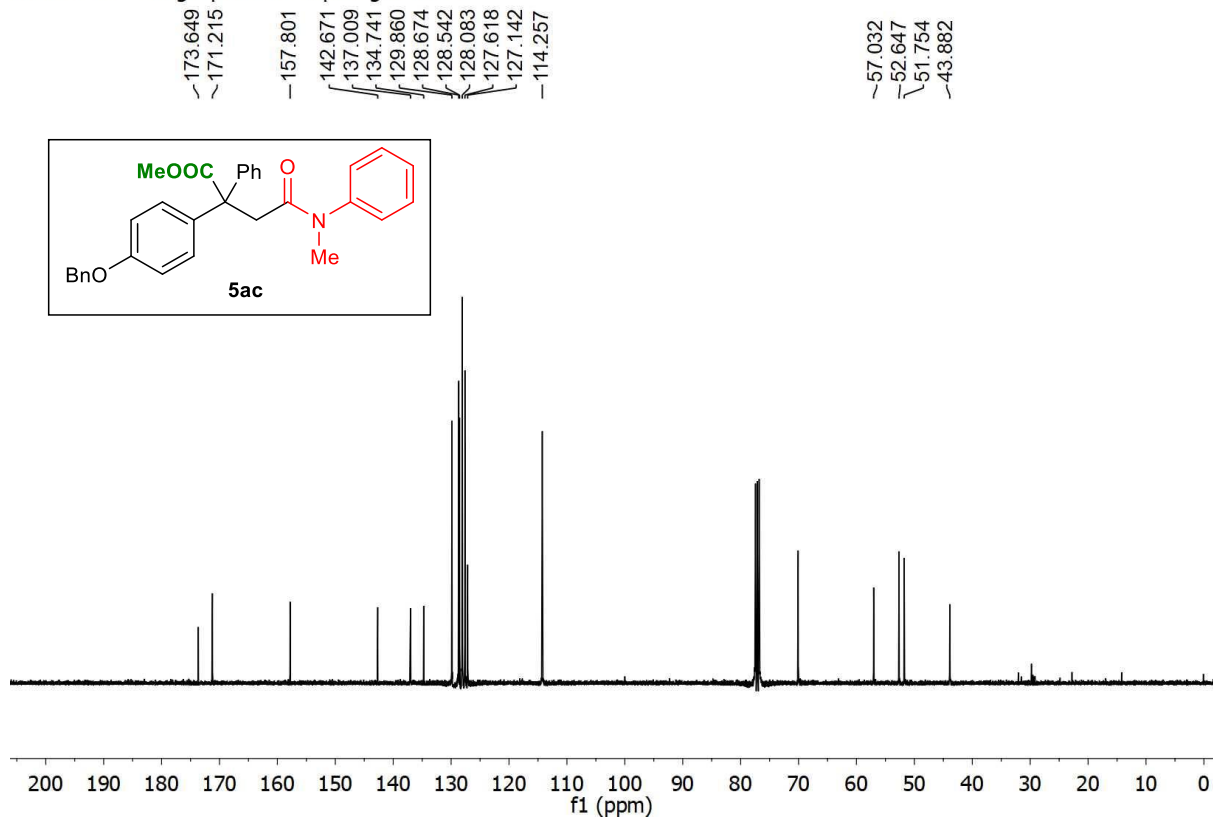
<sup>13</sup>C spectra of 5ab

SNAN-1346 — single\_pulse



<sup>1</sup>H spectra of **5ac**

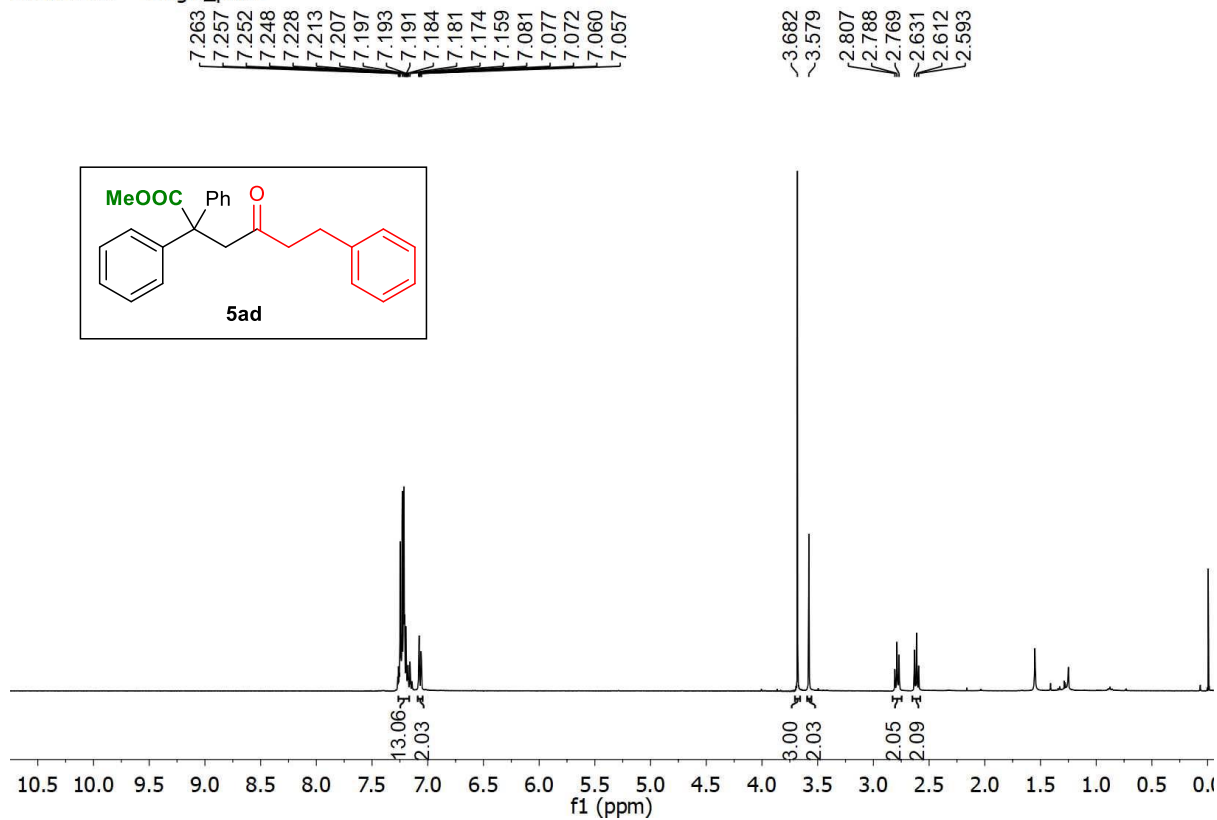
SNAN-1346 — single pulse decoupled gated NOE



<sup>13</sup>C spectra of **5ac**

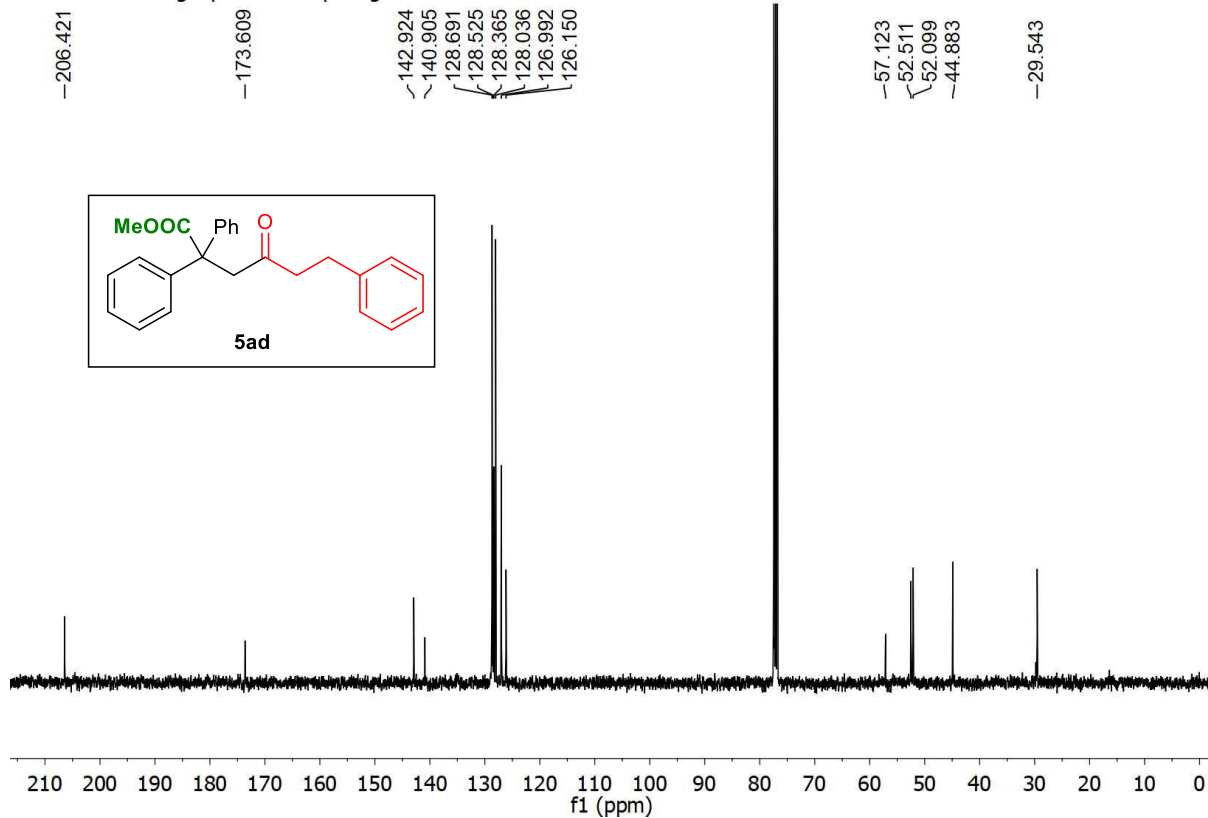


SNAN-1499 — single\_pulse



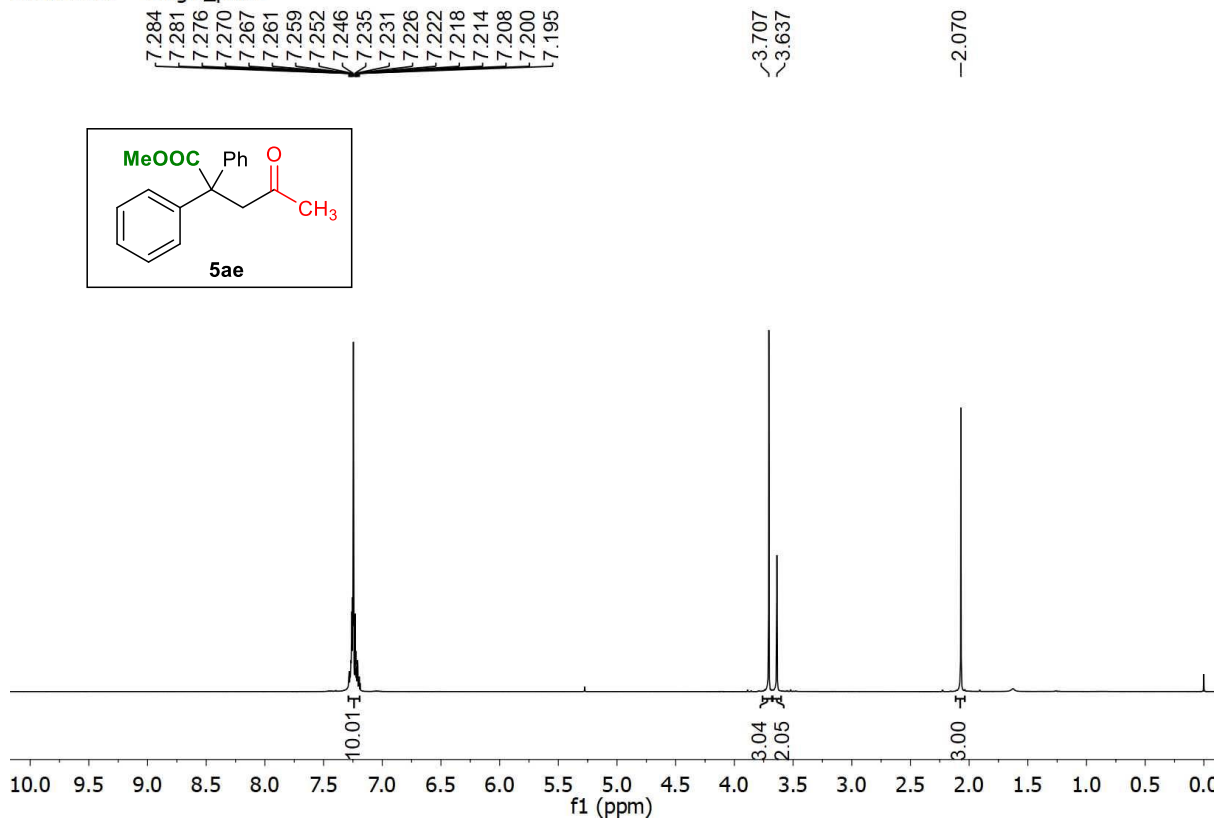
<sup>1</sup>H spectra of **5ad**

SNAN-1499 — single pulse decoupled gated NOE



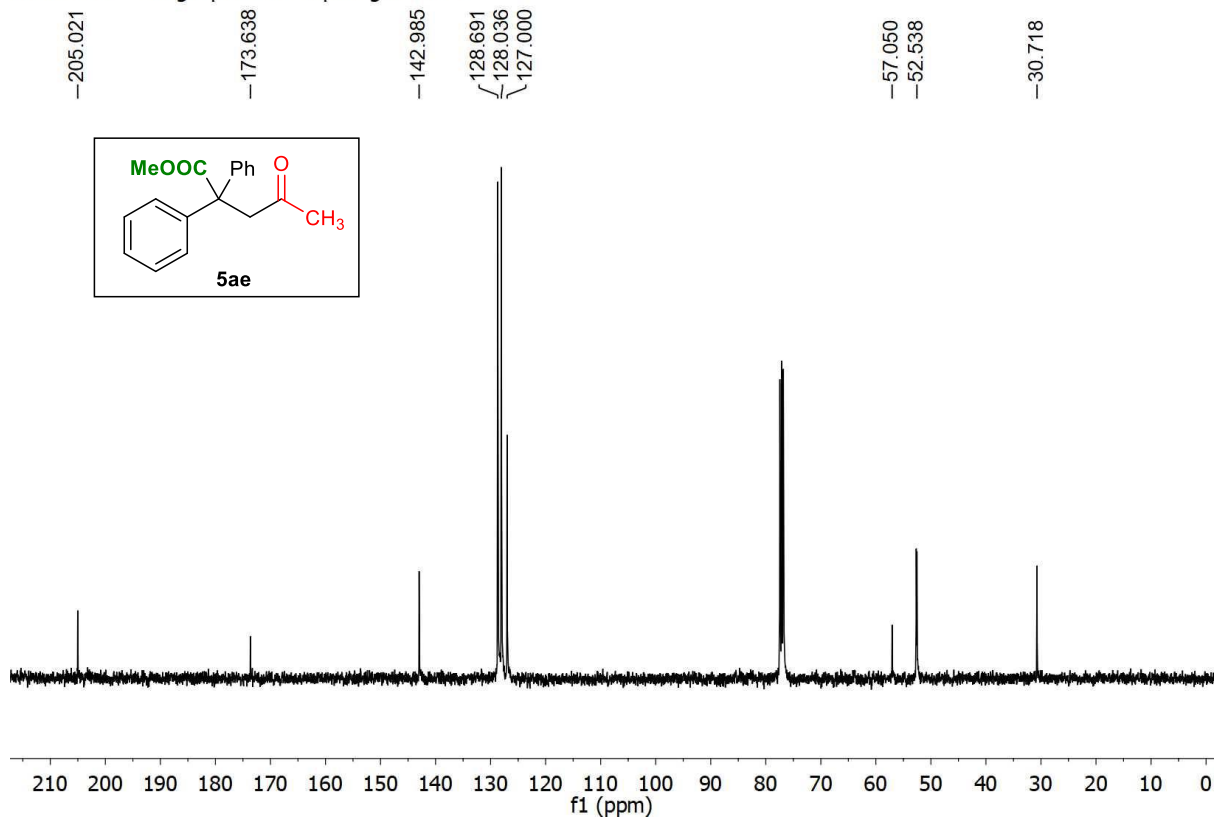
<sup>13</sup>C spectra of **5ad**

SNAN-1500 — single\_pulse



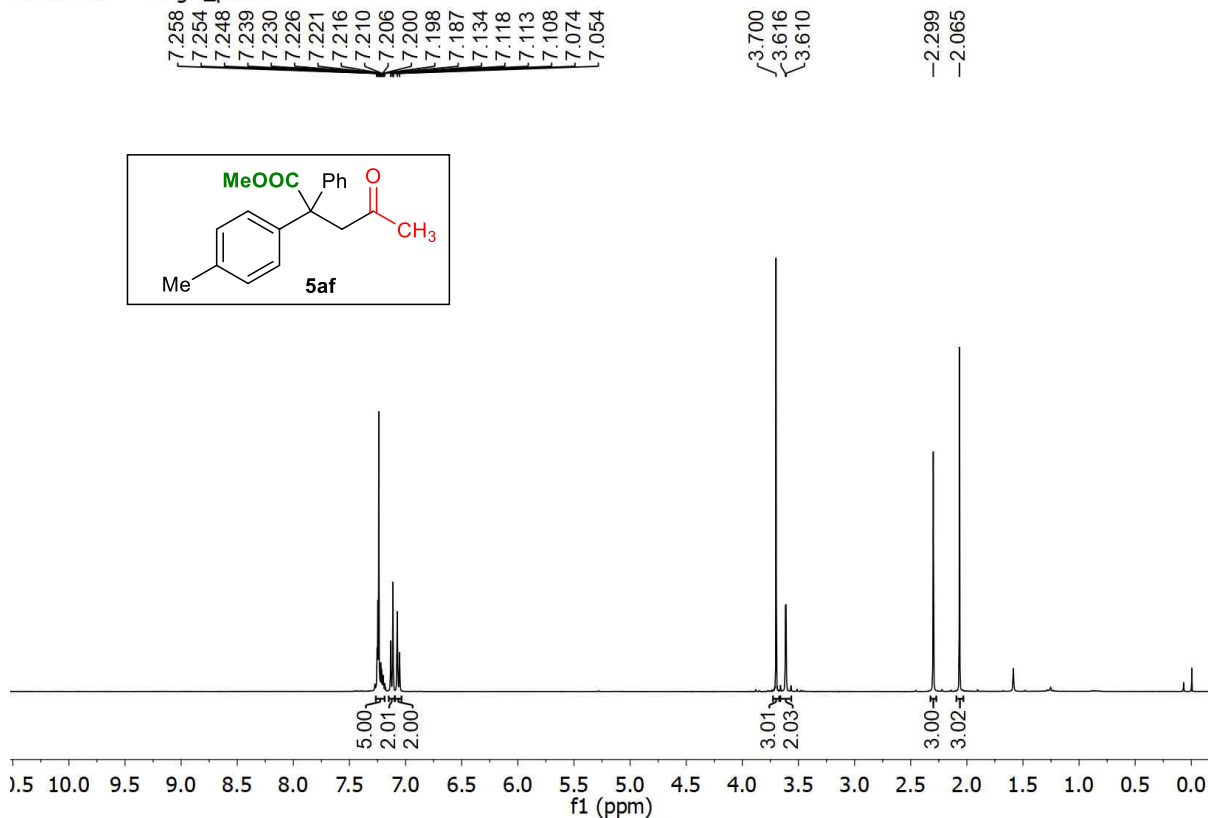
<sup>1</sup>H spectra of **5ae**

SNAN-1500 — single\_pulse decoupled gated NOE



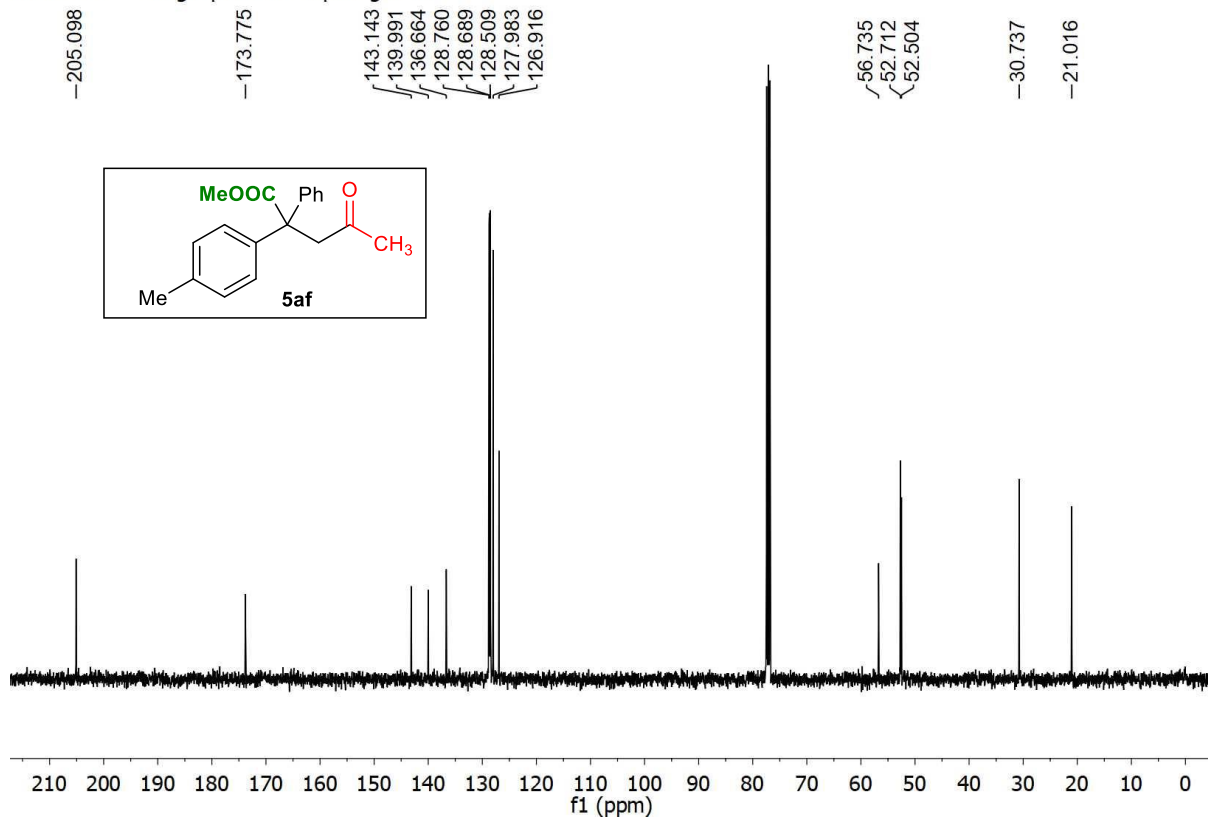
<sup>13</sup>C spectra of **5ae**

SNAN-1513 — single\_pulse



<sup>1</sup>H spectra of **5af**

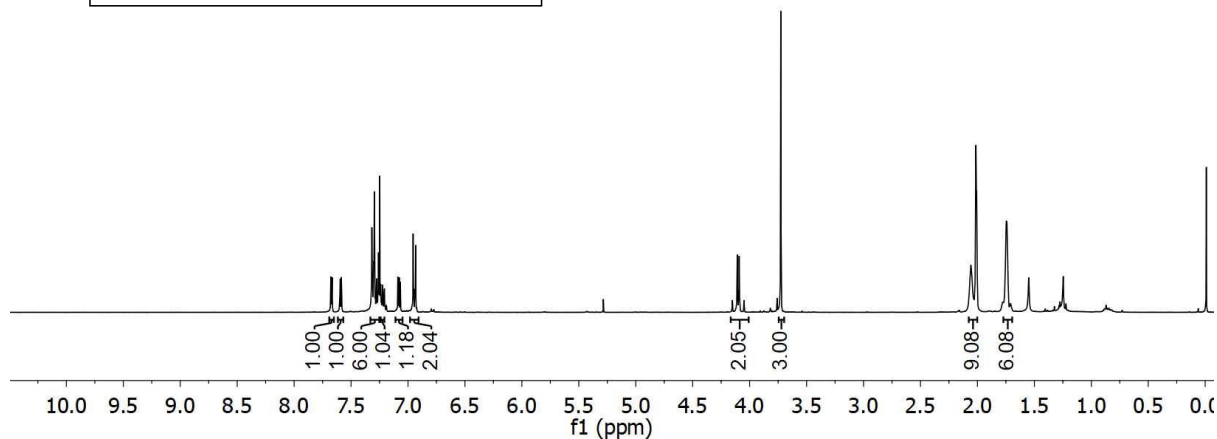
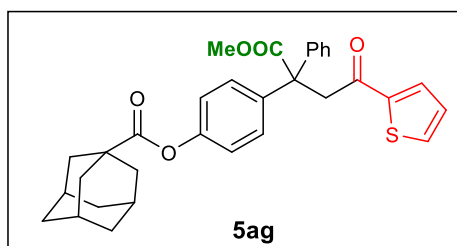
SNAN-1513 — single pulse decoupled gated NOE



<sup>13</sup>C spectra of **5af**

SNAN-1460 — single\_pulse

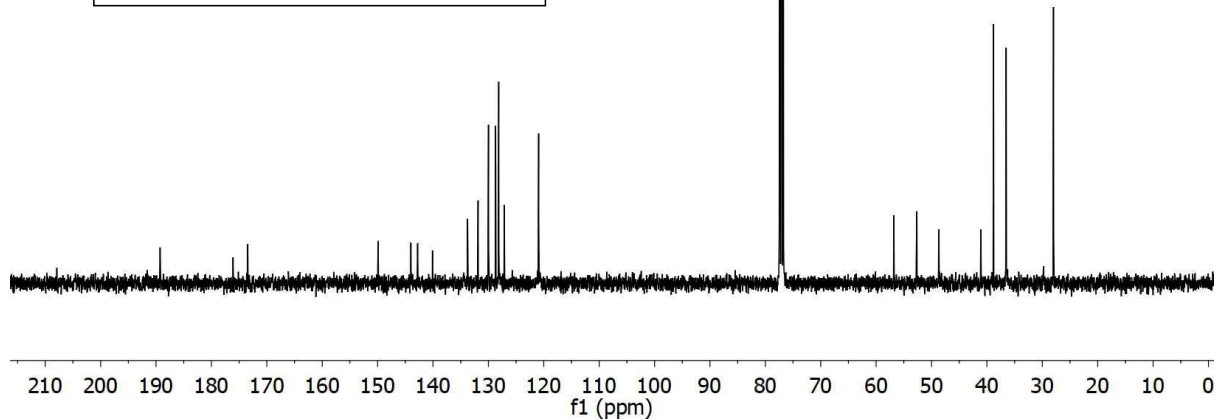
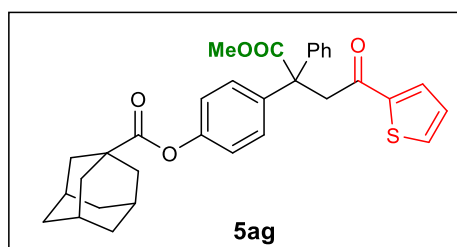
7.678  
7.675  
7.668  
7.665  
7.598  
7.595  
7.585  
7.583  
7.317  
7.300  
7.295  
7.279  
7.276  
7.259  
7.240  
7.228  
7.224  
7.220  
7.207  
7.089  
7.080  
7.077  
7.067  
6.963  
6.955  
6.950  
6.938  
6.933  
4.152  
4.109  
4.092  
4.049  
3.726  
2.057  
2.034  
2.013  
2.006  
1.779  
1.746  
1.741  
1.716  
1.708



<sup>1</sup>H spectra of **5ag**

SNAN-1460 — single pulse decoupled gated NOE

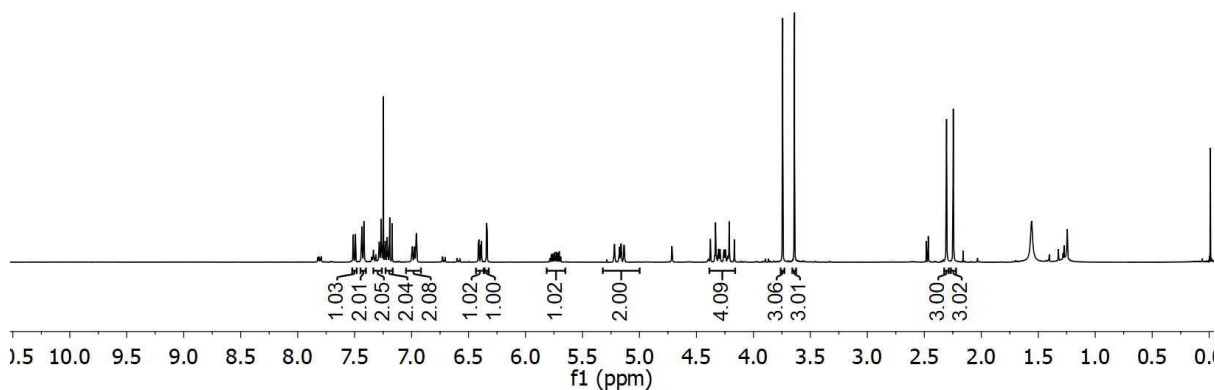
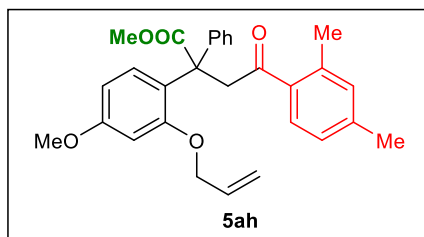
-189.266  
-176.096  
-173.443  
149.909  
143.988  
142.779  
140.096  
133.756  
131.861  
129.985  
128.698  
128.138  
127.137  
120.925  
56.802  
52.657  
48.699  
41.109  
38.815  
36.525  
27.978



<sup>13</sup>C spectra of **5ag**

SNAN-1444 — single\_pulse

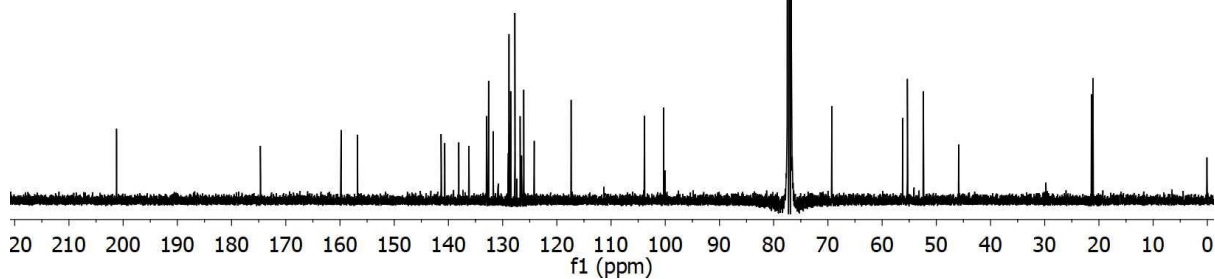
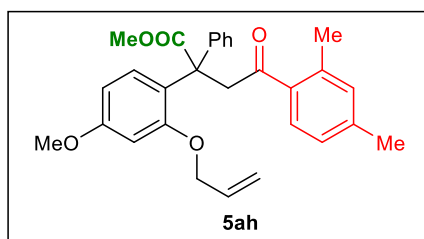
7.513  
7.493  
7.440  
7.436  
7.431  
7.422  
7.418  
7.334  
7.314  
7.288  
7.284  
7.280  
7.267  
7.232  
7.214  
7.192  
7.171  
6.997  
6.974  
6.959  
6.414  
6.408  
6.393  
6.386  
6.342  
6.336  
5.774  
5.761  
5.747  
5.731  
5.717  
5.704  
5.222  
5.218  
5.178  
5.175  
5.163  
5.160  
5.137  
5.133  
4.377  
4.332  
4.306  
4.289  
4.246  
4.212  
4.167  
3.743  
3.640  
2.305  
2.246



<sup>1</sup>H spectra of **5ah**

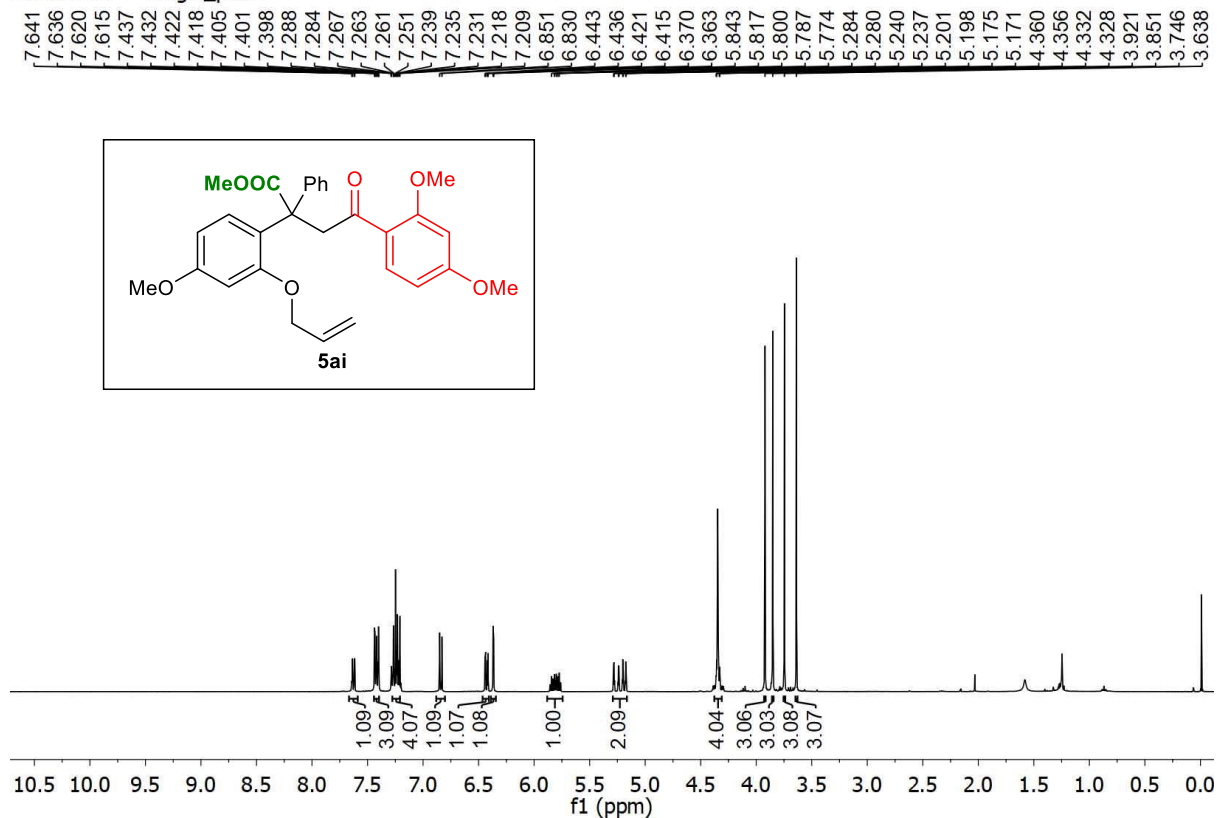
SNAN-1444 — single\_pulse decoupled gated NOE

-201.216  
-174.690  
159.796  
156.772  
141.353  
140.677  
138.088  
136.219  
132.960  
132.589  
131.712  
128.948  
128.821  
128.517  
127.741  
126.774  
126.500  
126.108  
124.173  
117.369  
103.817  
100.278  
69.275  
56.202  
55.336  
52.395  
45.883  
21.375  
21.103



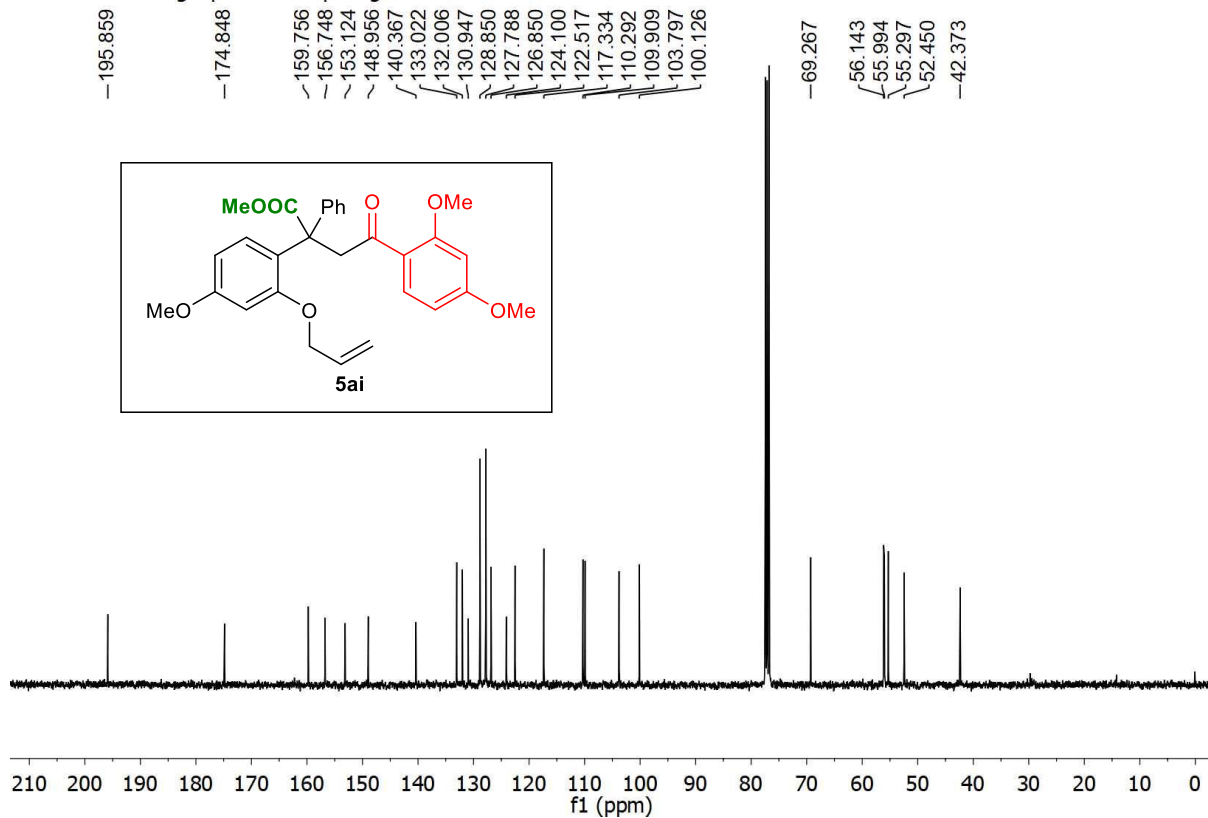
<sup>13</sup>C spectra of **5ah**

SNAN-1462 — single\_pulse



<sup>1</sup>H spectra of **5ai**

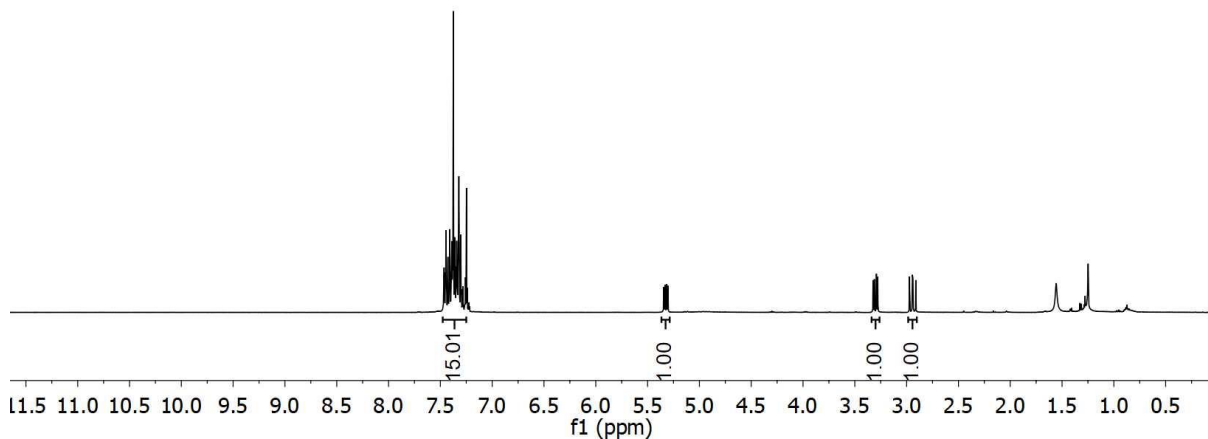
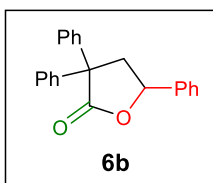
SNAN-1462 — single pulse decoupled gated NOE



<sup>13</sup>C spectra of **5ai**

SNAN-1521 — single\_pulse

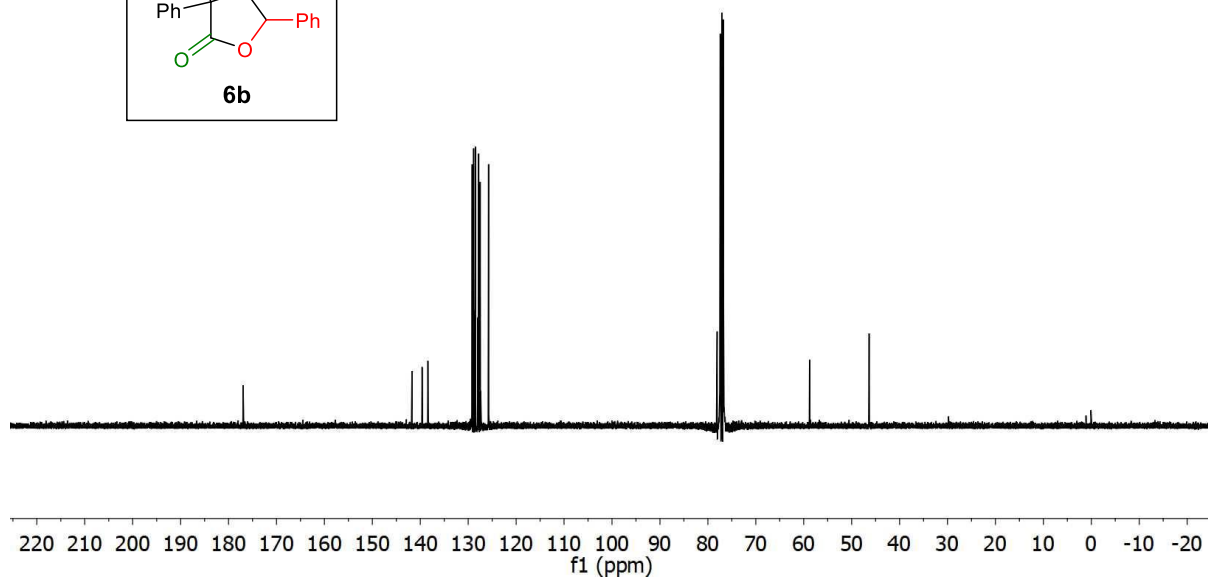
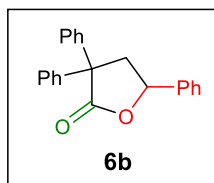
7.468  
7.464  
7.446  
7.443  
7.427  
7.409  
7.389  
7.382  
7.364  
7.361  
7.342  
7.325  
7.321  
7.302  
7.297  
7.282  
7.262  
7.258  
5.344  
5.332  
5.317  
5.304  
3.324  
3.311  
3.291  
3.279  
2.972  
2.944  
2.939  
2.912



<sup>1</sup>H spectra of **6b**

SNAN-1521 — single pulse decoupled gated NOE

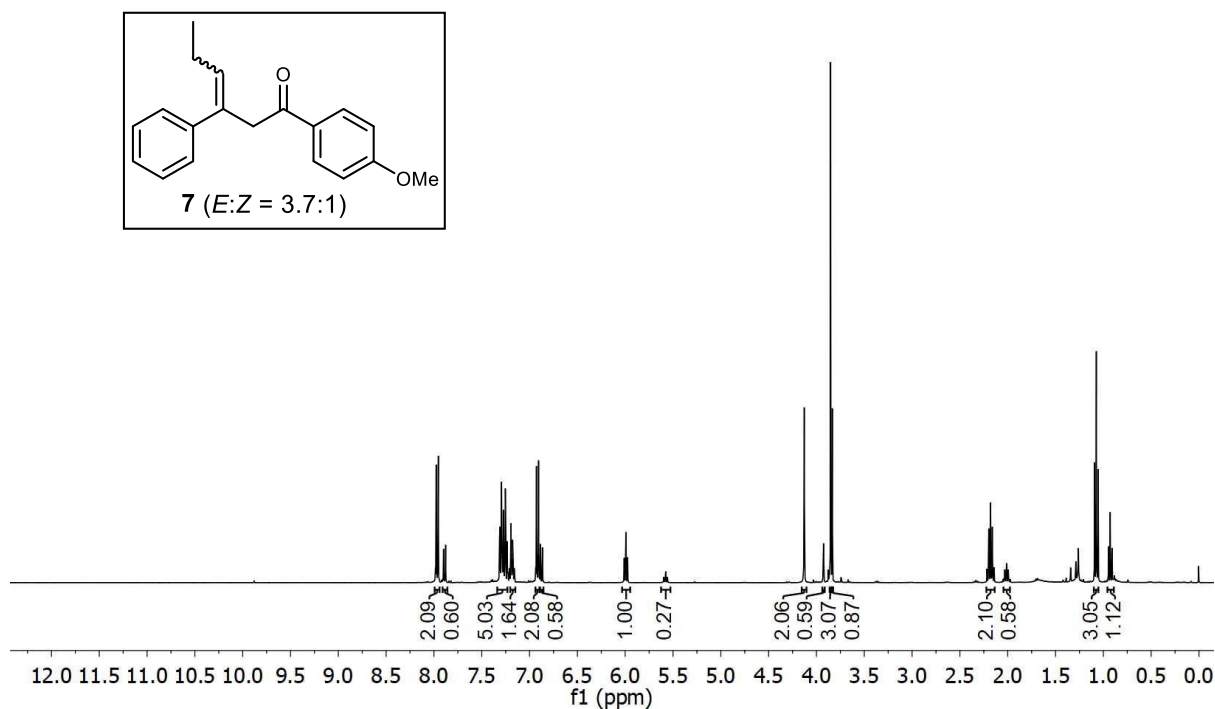
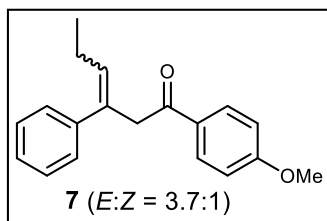
176.960  
141.731  
139.618  
138.408  
129.175  
128.868  
128.767  
128.494  
127.969  
127.813  
127.495  
127.407  
125.760  
78.065  
58.785  
46.352



<sup>13</sup>C spectra of **6b**

SNAN-1446 — single\_pulse

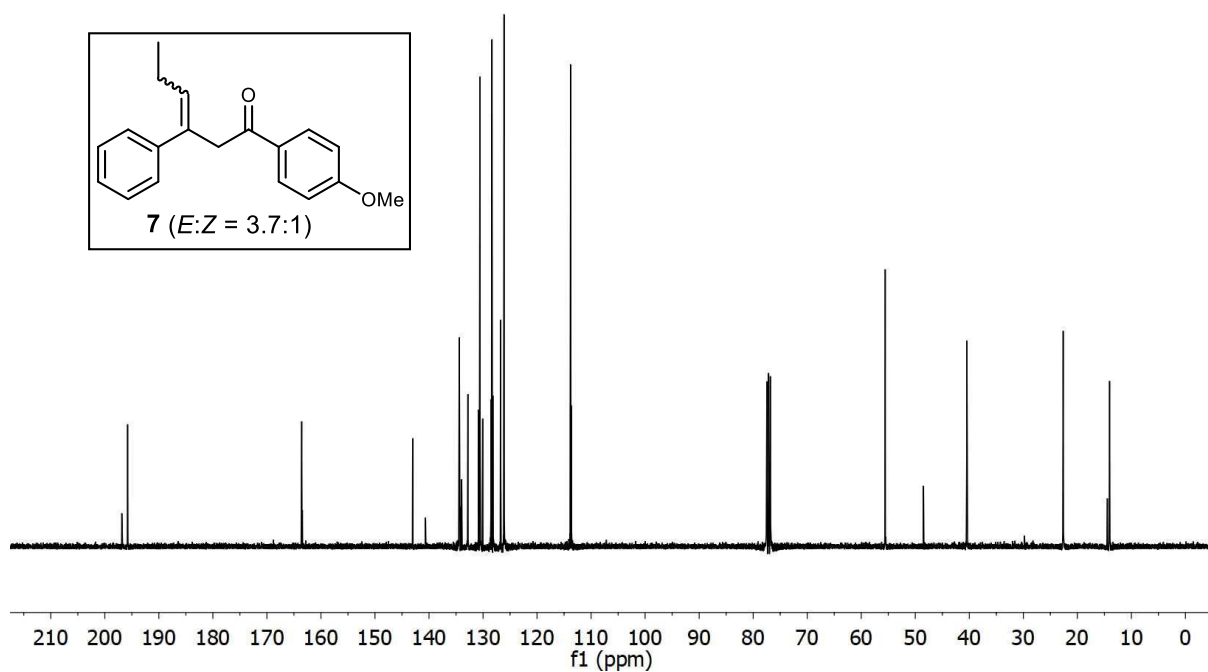
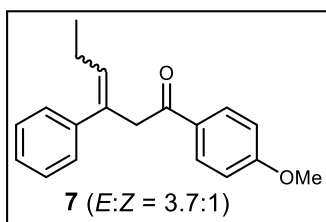
7.984  
7.976  
7.971  
7.954  
7.947  
7.900  
7.877  
7.315  
7.311  
7.290  
7.271  
7.253  
7.248  
7.233  
7.195  
7.171  
6.928  
6.923  
6.911  
6.906  
6.888  
6.865  
6.011  
5.993  
5.975  
5.596  
5.577  
5.559  
4.128  
3.852  
3.834  
2.217  
2.198  
2.179  
2.161  
2.142  
2.031  
2.012  
1.994  
1.092  
1.074  
1.055  
0.946  
0.928  
0.909



<sup>1</sup>H spectra of **7**

SNAN-1446 — single\_pulse decoupled gated NOE

196.855  
195.779  
163.557  
163.431  
143.024  
140.648  
134.394  
133.954  
132.805  
130.806  
130.576  
130.075  
128.508  
128.347  
128.174  
126.835  
126.751  
126.111  
126.099  
113.794  
113.687  
55.556  
55.512  
48.478  
40.448  
22.647  
22.624  
14.447  
14.033



<sup>13</sup>C spectra of **7**