Supporting Information

Decatungstate-mediated desulfonylative allylation and diacrylation using aldehydes/alkanes and allylic sulfones

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Table of Contents

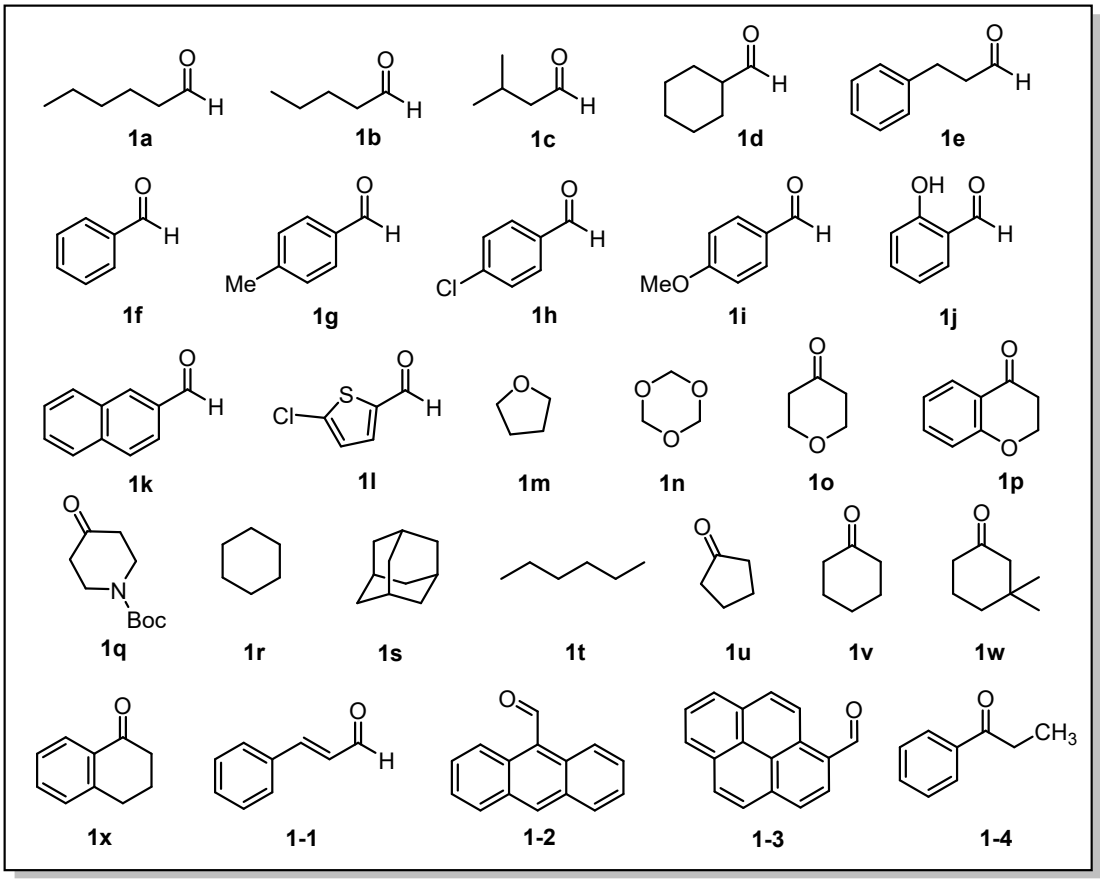
1. General Information

Commercially available reagents were used as received without further purification. All reactions were carried out in 10 mL sealed tubes filled with nitrogen, using 365-370 nm LED (12 W) purchased from SHANGHAI 3S TECHNOLOGY for light irradiation. Reactions were monitored by thin layer chromatography (TLC) using glass 0.25 mm silica gel plates with visualization under UV light (254 nm and 365 nm), or $KMnO_4$ staining solution as chromogenic agents. Organic solutions were concentrated under reduced pressure on a Heidolph rotary evaporator. Chromatographic purification of products was accomplished by flash chromatography on Merck Silica Gel 60F-254 (200-400 mesh). TBADT (tetrabutylammonium decatungstate) was synthesized according to the reported methods[1].

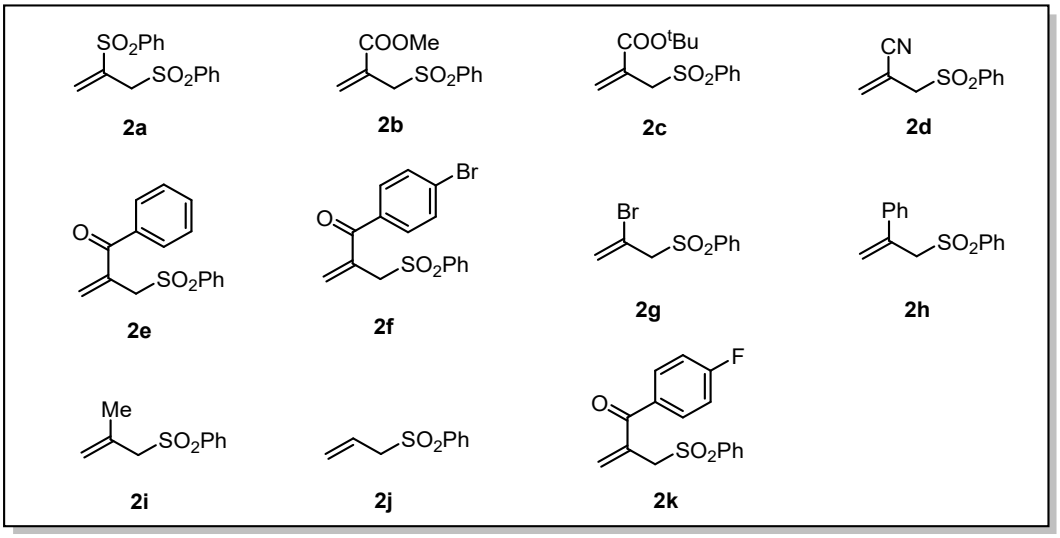
^1H NMR and ^{13}C NMR spectra were recorded on a Bruker Advance III-400 in solvents as indicated. Chemical shift were reported in ppm from TMS with the solvent resonance as internal standard ($CDCl_3$: ^1H-NMR: δ 7.26; ^{13}C-NMR: δ 77.0). Data for ^1H NMR and ^{19}F were reported as follows: chemical shift referred to TMS (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad singlet, dd = doublet of doublets, td = triplet of doublets etc.), coupling constants (*J*) were reported in units of hertz (Hz). Data for ^{13}C NMR were reported in terms of chemical shift. HRMS were obtained on a Q-TOF micro spectrometer.

2. Starting Materials

H–Donors

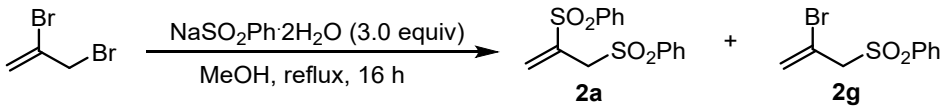


allylic sulfones



All of the aldehydes and alkanes **1** were purchased and used directly from commercial sources. Allylic sulfones were synthesized according to the literatures[2], and all of the spectra were in full accordance with the data in the literatures.

Perparaing of starting materials 2a and 2g



Sodium benzenesulfinate dihydrate (9.0 g, 45 mmol) and 2,3-dibromopropene (3.0 g, 15 mmol) were dissolved in MeOH (60 mL) in a dried round bottom flask equipped with a magnetic stir bar. The mixture was stirred at 70 °C for 16 h. After cooling to room temperature, the mixture was concentrated under reduced pressure and diluted with ethyl acetate, washed with water and brine. Then the organic layer was separated, dried with Na_2SO_4 and concentrated under reduced pressure. The residue was further purified by flash chromatography on silica gel (petroleum ether/EtOAc = 5:1–3:1) affording 1,2-bis(phenylsulfonyl)-2-propene **2a** (2.81 g) in 58% yield as a white solid and ((2-bromoallyl)sulfonyl)benzene **2g** (1.21 g) in 31% yield as a white solid.

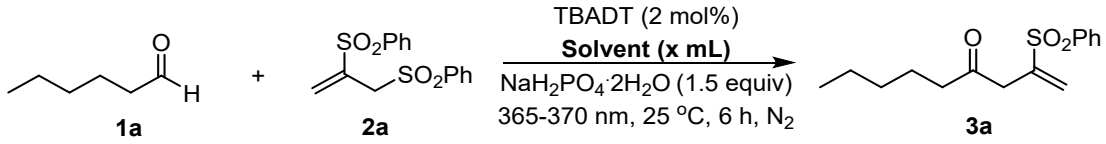
3. Optimization of Reaction Conditions

3.1 General Procedure A for the synthesis of 3a

A 10 mL oven-dried sealed tube equipped with a magnetic stir bar was charged with TBADT (13.4 mg, 2 mol%), base and allyl sulfone **2a** (128.8 mg, 0.4 mmol, 2.0 equiv). The tube was evacuated and backfilled with nitrogen (three times). Next, hexanal **1a** (25 μL, 0.2 mmol) and solvent were subsequently injected into the tube by syringe under nitrogen atmosphere. The reaction mixture was stirred and irradiated using 365-370 nm LED lamps (1.0 cm from the bulb, with adequate fans and a water bath to keep the reaction at room temperature) for 6 h at 25 °C. After exposing to air for 10 minutes, the mixture was diluted with ethyl acetate (10 mL), washed with water (4 mL) and brine (4 mL). The organic layer was then separated, dried with Na_2SO_4 and concentrated under reduced pressure. The crude product was purified through column chromatography (silica gel, petroleum ether/ethyl acetate) to afford the product **3a**.

3.2 Optimization A for the synthesis of 3a

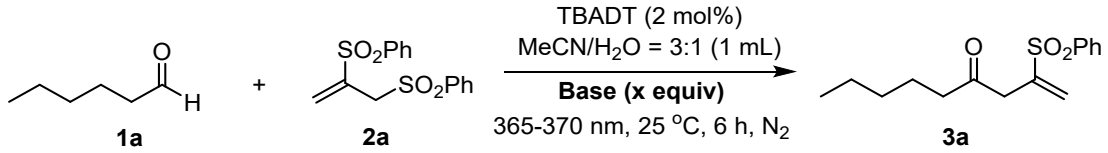
Table S1 Optimization of 3a——*Screening of Solvent*



Entry	Solvent	Yield[a] (%)
1	MeCN (1 mL)	28
2	MeCN/H_2O = 1:1 (1 mL)	30
3	MeCN/H_2O = 2:1 (1 mL)	50
4	MeCN/H_2O = 3:1 (1 mL)	**73(68)**
5	MeCN/H_2O = 4:1 (1 mL)	46
6	Acetone/H_2O = 3:1 (1 mL)	44
7	DMF/H_2O = 3:1 (1 mL)	＜5
8	MeCN/H_2O = 3:1 (2 mL)	55

[a]Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), TBADT (2 mol%), $NaH_2PO_4·2H_2O$ (1.5 equiv), solvent (x mL), 365-370 nm UV-LEDs, 25 °C, 6 h, N_2. Yields were determined by crude ^{1}H NMR spectra using 1,3,5-trimethoxybenzene as an internal standard. Isolated yields were depicted in the parentheses.

Screening of Base

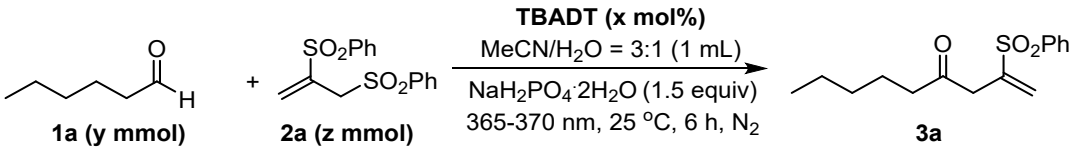


1a **2a** **3a**

Entry	Base	Yield[a] (%)
1	$NaH_2PO_4·2H_2O$ (1.5 equiv)	**73(68)**
2	NaOH (1.5 equiv)	＜5
3	K_2CO_3 (1.5 equiv)	＜5
4	NaOAc (1.5 equiv)	46
5	Cs_2CO_3 (1.5 equiv)	＜5
6	$NaHCO_3$ (1.5 equiv)	59
7	$NaH_2PO_4·2H_2O$ (3 equiv)	53
8	iPr_2Net (1.5 equiv)	n.r.
9	Et_3N (1.5 equiv)	n.r.
10	Without base	＜5

[a]Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), TBADT (2 mol%), base (x equiv), $MeCN/H_2O$ = 3:1 (1 mL), 365-370 nm UV-LEDs, 25 °C, 6 h, N_2. Yields were determined by crude ^1H NMR spectra using 1,3,5-trimethoxybenzene as an internal standard. Isolated yields were depicted in the parentheses. n. r. = no reaction.

Others



1a (y mmol) **2a (z mmol)** **3a**

Entry	x (mol%)	y (mmol)	z (mmol)	Yield[a] (%)
1	-	0.2	0.4	＜5[b]
2	1	0.2	0.4	55
3	2	0.2	0.4	**73(68)**
4	4	0.2	0.4	41
5	2	0.2	0.2	32
6	2	0.2	0.3	58
7	2	0.2	0.6	50
8	2	0.4	0.2	(15)[c]

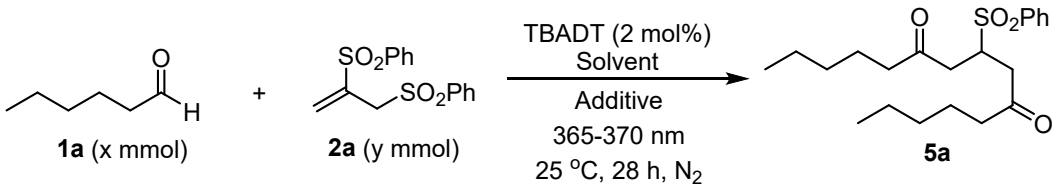
[a]Reaction conditions: **1a** (y mmol), **2a** (z mmol), TBADT (x mol%), $NaH_2PO_4·2H_2O$ (1.5 equiv), $MeCN/H_2O$ = 3:1 (1 mL), 365-370 nm UV-LEDs, 25 °C, 6 h, N_2. Yields were determined by crude ^1H NMR spectra using 1,3,5-trimethoxybenzene as an internal standard. Isolated yields were depicted in the parentheses. [b]The reaction was conducted in the absence of light or TBADT. [c]27% of **5a** was isolated.

3.3 General Procedure B for Optimization of 5a

A 10 mL oven-dried sealed tube equipped with a magnetic stir bar was charged with TBADT (13.4 mg, 2 mol%) and allyl sulfone **2a** (64.4 mg, 0.2 mmol). The tube was evacuated and backfilled with nitrogen (three times). Next, hexanal **1a** and solvent (2 mL) were subsequently injected into the tube by syringe under nitrogen atmosphere. The reaction mixture was stirred and irradiated using 365-370 nm LED lamps (1.0 cm from the bulb, with adequate fans and a water bath to keep the reaction at room temperature) for 28 h at 25 °C. After exposing to air for 10 minutes, the mixture was diluted with ethyl acetate (10 mL), washed with water (4 mL) and brine (4 mL). The organic layer was then separated, dried with Na_2SO_4 and concentrated under reduced pressure. The crude product was purified through column chromatography (silica gel, petroleum ether/ethyl acetate) to afford the product **5a**.

3.4 Optimization B for the synthesis of 5a

Table S2 Optimization of 5a

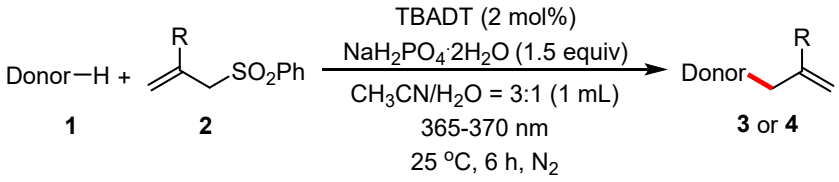


Entry	x (mmol)	y (mmol)	Solvent (mL)	Additive (equiv)	Yield[a] (%)
1	0.2	0.2	MeCN (2 mL)	-	21
2	0.5	0.2	MeCN (2 mL)	-	59
3	1	0.2	MeCN (2 mL)	-	**66**
4	2	0.2	MeCN (2 mL)	-	64
5	1	0.2	Acetone (2 mL)	-	47
6	1	0.2	DMF (2 mL)	-	13
8	1	0.2	DCM (2 mL)	-	35
9	1	0.2	MeCN/H_2O = 3:1 (2 mL)	-	41
10	1	0.2	MeCN (2 mL)	K_2CO_3 (2 equiv)	39
11	1	0.2	MeCN/H_2O = 3:1 (2 mL)	K_2CO_3 (2 equiv)	30

[a]Reaction conditions: **1a** (x mmol), **2a** (y mmol), TBADT (2 mol%), additive (2.0 equiv), solvent (2 mL), 365-370 nm UV-LEDs, 25 °C, 28 h, N_2. Yields of isolated product.

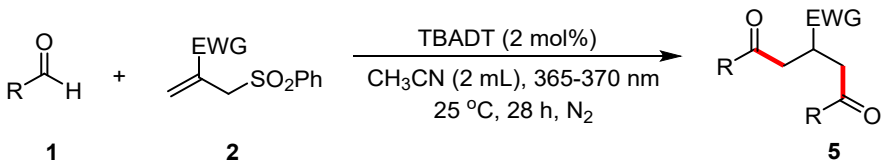
4. Decatungstate-mediated desulfonylative allylation and diacrylation using aldehydes/alkanes and allylic sulfones

4.1 General Procedure for Schemes 2 and 3 (General Procedure C)



A 10 mL oven-dried sealed tube equipped with a magnetic stir bar was charged with TBADT (13.4 mg, 0.004 mmol, 2 mol%), H–donors **1** (if solid, 0.2 mmol), $NaH_2PO_4 \cdot 2H_2O$ (46.8 mg, 0.3 mmol, 1.5 equiv) and allyl sulfones **2** (0.4 mmol, 2.0 equiv). The tube was evacuated and backfilled with nitrogen (three times). Next, H–donors **1** (if liquid, 0.2 mmol), MeCN (0.75 mL) and distilled water (0.25 mL) were subsequently injected into the tube by syringe under nitrogen atmosphere. The reaction mixture was stirred and irradiated using 365-370 nm LED lamps (1.0 cm from the bulb, with adequate fans and a water bath to keep the reaction at room temperature) for 6 h at 25 °C. After the reaction was completed, the mixture was diluted with ethyl acetate (10 mL), washed with water (4 mL) and brine (4 mL). The organic layer was then separated, dried with Na_2SO_4 and concentrated under reduced pressure. The crude product was purified through column chromatography (silica gel, petroleum ether/ethyl acetate) to afford the desired products **3** or **4** listed in Scheme 2 and Scheme 3.

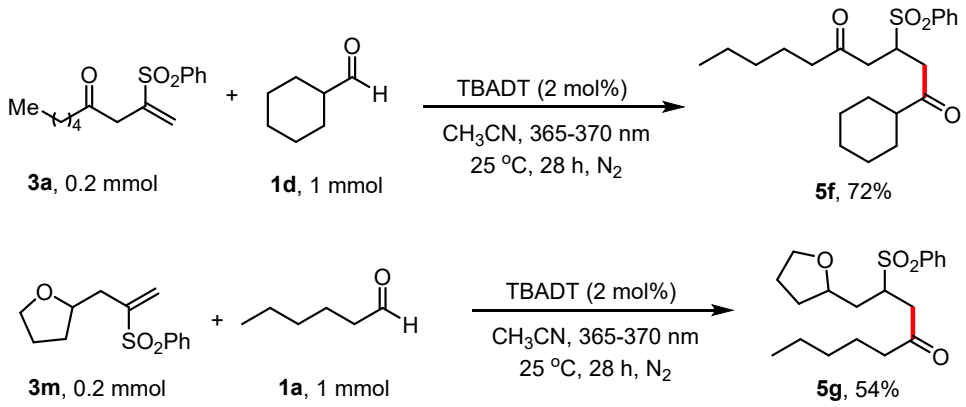
4.2 General Procedure for Scheme 4 (General Procedure D)



A 10 mL oven-dried sealed tube equipped with a magnetic stir bar was charged with TBADT (13.4 mg, 0.004 mmol, 2 mol%), allyl sulfones **2** (0.2 mmol). The tube was evacuated and backfilled with nitrogen (three times). Next, aldehydes (1.0 mmol, 5.0 equiv) and 2 mL of MeCN were subsequently injected into the tube by syringe under

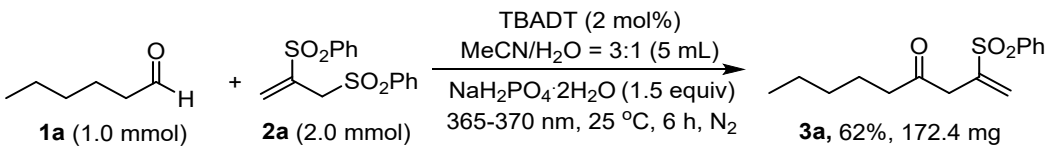
nitrogen atmosphere. The reaction mixture was stirred and irradiated using 365-370 nm LED lamps (1.0 cm from the bulb, with adequate fans and a water bath to keep the reaction at room temperature) for 28 h at 25 °C. After the reaction was completed, the mixture was diluted with ethyl acetate (10 mL), washed with water (4 mL) and brine (4 mL). The organic layer was then separated, dried with Na_2SO_4 and concentrated under reduced pressure. The crude product was purified through column chromatography (silica gel, petroleum ether/ethyl acetate) to afford the desired products **5** listed in Scheme 4.

4.3 General Procedure for Synthesis of 5f and 5g (General Procedure E)



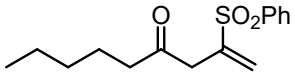
A 10 mL oven-dried sealed tube equipped with a magnetic stir bar was charged with TBADT (13.4 mg, 0.004 mmol, 2 mol%), allylation products **3** (0.2 mmol). The tube was evacuated and backfilled with nitrogen (three times). Next, aldehydes (1.0 mmol, 5.0 equiv) and 2 mL of MeCN were subsequently injected into the tube by syringe under nitrogen atmosphere. The reaction mixture was stirred and irradiated using 365-370 nm LED lamps (1.0 cm from the bulb, with adequate fans and a water bath to keep the reaction at room temperature) for 28 h at 25 °C. After the reaction was completed, the mixture was diluted with ethyl acetate (10 mL), washed with water (4 mL) and brine (4 mL). The organic layer was then separated, dried with Na2SO4 and concentrated under reduced pressure. The crude products was purified through column chromatography (silica gel, petroleum ether/ethyl acetate) to afford the desired products **5f** and **5g**.

4.4 General Procedure for Synthesis of 3a on 1 mmol scale (General Procedure F)

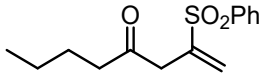


A 10 mL oven-dried sealed tube equipped with a magnetic stir bar was charged with TBADT (66.8 mg, 0.02 mmol, 2 mol%), $NaH_2PO_4 \cdot 2H_2O$ (234 mg, 1.5 mmol, 1.5 equiv) and allyl sulfone **2a** (644 mg, 2.0 mmol, 2.0 equiv). The tube was evacuated and backfilled with nitrogen (three times). Next, hexanal **1a** (125 μL, 1.0 mmol), MeCN (3.75 mL) and distilled water (1.25 mL) were subsequently injected into the tube by syringe under nitrogen atmosphere. The reaction mixture was stirred and irradiated using 365-370 nm LED lamps (1.0 cm from the bulb, with adequate fans and a water bath to keep the reaction at room temperature) for 6 h at 25 °C. After exposing to air for 10 minutes, the mixture was diluted with ethyl acetate, washed with water and brine. The organic layer was then separated, dried with Na_2SO_4 and concentrated under reduced pressure. The crude product was purified through column chromatography (silica gel, petroleum ether/ethyl acetate) to afford the product **3a** (172.4 mg) in 62% yield.

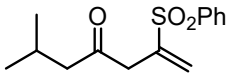
4.4 Characterization of Products



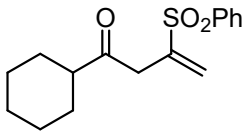
2-(Phenylsulfonyl)non-1-en-4-one (3a): Colorless oil; (68%, 37.8 mg); Rf 0.45 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 7.6 Hz, 2H), 7.62 (t, *J* = 7.1 Hz, 1H), 7.53 (t, *J* = 7.8 Hz, 2H), 6.51 (s, 1H), 5.93 (s, 1H), 3.33 (s, 2H), 2.33 (t, *J* = 7.4 Hz, 2H), 1.48-1.37 (m, 2H), 1.29-1.10 (m, 4H), 0.84 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.2, 143.2, 138.2, 133.7, 129.2, 128.4, 127.9, 42.5, 42.4, 31.1, 23.1, 22.3, 13.8; HRMS (ESI) calcd for $C_{15}H_{21}O_3S$ $[M+H]^+$ 281.1206, found 281.1201.



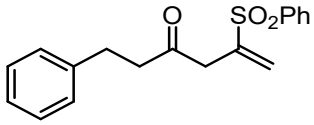
2-(Phenylsulfonyl)oct-1-en-4-one (3b): Colorless oil; (61%, 32.6 mg); Rf 0.45 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.2 Hz, 2H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 2H), 6.52 (s, 1H), 5.94 (s, 1H), 3.34 (s, 2H), 2.35 (t, *J* = 7.2 Hz, 2H), 1.47-1.38 (m, 2H), 1.25-1.17 (m, 2H), 0.85 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.2, 143.6, 138.2, 133.8, 129.3, 128.4, 127.9, 42.4, 42.6, 25.5, 22.1, 13.8; HRMS (ESI) calcd for $C_{14}H_{19}O_3S$ $[M+H]^+$ 267.1049, found 267.1050.



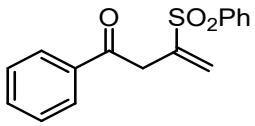
6-Methyl-2-(phenylsulfonyl)hept-1-en-4-one (3c): Colorless oil; (57%, 30.1 mg); Rf 0.45 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.6 Hz, 2H), 7.63 (t, *J* = 7.2 Hz, 1H), 7.54 (t, *J* = 7.2 Hz, 2H), 6.53 (s, 1H), 5.94 (s, 1H), 3.32 (s, 2H), 2.23 (d, *J* = 6.8 Hz, 2H), 2.05-1.95 (m, 1H), 0.83 (s, 3H), 0.81 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 202.7, 142.5, 137.3, 132.8, 128.3, 127.4, 126.9, 50.4, 41.8, 23.3, 21.4; HRMS (ESI) calcd for $C_{14}H_{19}O_3S$ $[M+H]^+$ 267.1049, found 267.1046.



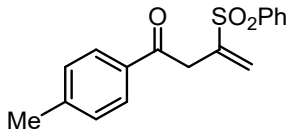
1-Cyclohexyl-3-(phenylsulfonyl)but-3-en-1-one (3d): Colorless oil; (58%, 33.8 mg); Rf 0.45 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.2 Hz, 2H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.54 (t, *J* = 8.0 Hz, 2H), 6.53 (s, 1H), 5.92 (s, 1H), 3.41 (s, 2H), 2.34-2.18 (m, 1H), 1.75-1.60 (m, 5H), 1.26-1.10 (m, 5H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.9, 143.5, 138.3, 133.7 129.2, 128.4, 128.0, 50.6, 40.2, 28.2, 25.6, 25.4; HRMS (ESI) calcd for $C_{16}H_{21}O_3S$ $[M+H]^+$ 293.1206, found 293.1193.



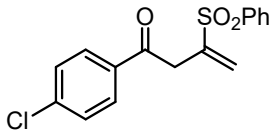
1-Phenyl-5-(phenylsulfonyl)hex-5-en-3-one (3e): Colorless oil; (68%, 42.7 mg); Rf 0.4 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 7.6 Hz, 2H), 7.61 (t, *J* = 7.2 Hz, 1H), 7.51 (t, *J* = 7.2 Hz, 2H), 7.24 (t, *J* = 7.2 Hz, 2H), 7.16 (t, *J* = 7.2 Hz, 1H), 7.09 (d, *J* = 7.2 Hz, 2H), 6.46 (s, 1H), 5.84 (s, 1H), 3.28 (s, 2H), 2.76 (t, *J* = 7.2 Hz, 2H), 2.67 (t, *J* = 7.2 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.2, 143.5, 140.4, 138.1, 133.8, 129.3, 128.5, 128.4, 128.3, 128.0, 126.2, 43.9, 42.8, 29.5; HRMS (ESI) calcd for $C_{18}H_{19}O_3S$ $[M+H]^+$ 315.1049, found 315.1047.



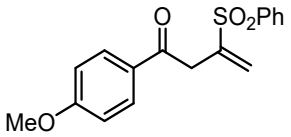
1-Phenyl-3-(phenylsulfonyl)but-3-en-1-one (3f): Colorless oil; (55%, 31.3 mg); Rf 0.35 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.85 (m, 2H), 7.81-7.79 (m, 2H), 7.64-7.49 (m, 4H), 7.42 (t, *J* = 8.0 Hz, 2H), 6.57 (s, 1H), 5.92 (s, 1H), 3.93 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.0, 144.0, 135.6, 133.8, 133.7, 129.3, 128.8, 128.5, 128.4, 127.8, 99.9, 38.4; HRMS (ESI) calcd for $C_{16}H_{15}O_3S$ $[M+H]^+$ 287.0736, found 287.0735.



3-(Phenylsulfonyl)-1-(*p*-tolyl)but-3-en-1-one (3g): Colorless oil; (61%, 36.6 mg); Rf 0.35 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.2 Hz, 2H), 7.69 (d, *J* = 7.6 Hz, 2H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.51 (t, *J* = 7.2 Hz, 2H), 7.21 (d, *J* = 7.2 Hz, 2H), 6.55 (s, 1H), 5.90 (s, 1H), 3.90 (s, 2H), 2.39 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.6, 144.7, 144.2, 138. 2, 133.7, 133.2, 129.4, 129.3, 128.5, 128.4, 127.6, 38.3, 21.7; HRMS (ESI) calcd for $C_{17}H_{17}O_3S$ $[M+H]^+$ 301.0893, found 301.0892.

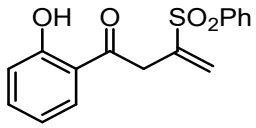


1-(4-Chlorophenyl)-3-(phenylsulfonyl)but-3-en-1-one (3h): Yellowish oil; (54%, 34.5 mg); Rf 0.35 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.2 Hz, 2H), 7.75 (d, *J* = 8.4 Hz, 2H), 7.63 (t, *J* = 7.2 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 2H), 7.40 (d, *J* = 8.4 Hz, 2H), 6.55 (s, 1H), 5.91 (s, 1H), 3.89 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.9, 143.9, 140.3, 138.1, 133.9, 133.9, 129.8, 129.3, 129.1, 128.5, 127.9, 38.4; HRMS (ESI) calcd for $C_{16}H_{14}ClO_3S$ $[M+H]^+$ 321.0347, found 321.0346.

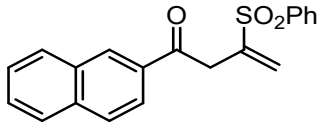


1-(4-Methoxyphenyl)-3-(phenylsulfonyl)but-3-en-1-one (3i): Colorless oil; (59%, 37.2 mg); Rf 0.35 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.6 Hz, 2H), 7.79 (d, *J* = 7.2 Hz, 2H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.51 (t, *J* = 6.8 Hz, 2H), 6.88 (d, *J* = 7.6 Hz, 2H), 6.53 (s, 1H), 5.89 (s, 1H), 3.86 (s, 5H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.5, 163.0, 143.4, 137.3, 132.7, 129.8, 128.3, 127.7, 127.4, 126.4, 112.9, 54.5, 37.1; HRMS (ESI) calcd for $C_{17}H_{17}O_4S$ $[M+H]^+$ 317.0842, found

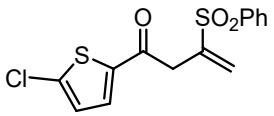
317.0836.



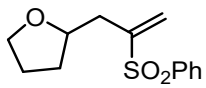
1-(2-Hydroxyphenyl)-3-(phenylsulfonyl)but-3-en-1-one (3j): Colorless oil; (51%, 30.5 mg); Rf 0.3 (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.68 (s, 1H), 7.87 (d, *J* = 7.6 Hz, 2H), 7.65-7.56 (m, 2H), 7.53-7.45 (m, 3H), 6.94 (d, *J* = 8.4 Hz, 1H), 6.86 (t, *J* = 7.6 Hz, 1H), 6.59 (s, 1H), 5.92 (s, 1H), 3.96 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.9, 162.7, 143.9, 138.1, 137.1, 133.9, 130.0, 129.3, 128.5, 128.1, 119.3, 118.7, 118.4, 38.3; HRMS (ESI) calcd for $C_{16}H_{15}O_4S$ $[M+H]^+$ 303.0686, found 303.0678.



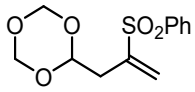
1-(Naphthalen-2-yl)-3-(phenylsulfonyl)but-3-en-1-one (3k): Colorless oil; (45%, 30.2 mg); Rf 0.4 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (s, 1H), 7.93-7.84 (m, 6H), 7.64-7.48 (m, 5H), 6.59 (s, 1H), 5.96 (s, 1H), 4.07 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.9, 144.1, 138.2, 135.7, 133.8, 132.9, 132.3, 130.4, 129.7, 129.3, 128.9, 128.6, 128.5, 127.8, 127.7, 127.0, 123.7, 38.4; HRMS (ESI) calcd for $C_{20}H_{17}O_3S$ $[M+H]^+$ 337.0893, found 337.0890.



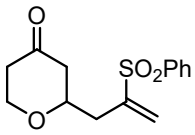
1-(5-Chlorothiophen-2-yl)-3-(phenylsulfonyl)but-3-en-1-one (3l): Brown oil; (41%, 26.7 mg); Rf 0.5 (EtOAc/petroleum ether = 1:4); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.0 Hz, 2H), 7.63 (t, *J* = 6.8 Hz, 1H), 7.52 (t, *J* = 7.2 Hz, 2H), 7.46-7.37 (m, 1H), 6.93-6.92 (m, 1H), 6.55 (s, 1H), 5.99 (s, 1H), 3.78 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 185.8, 143.5, 141.3, 141.1, 138.0, 133.9, 132.7, 129.3, 128.4, 127.9, 127.8, 38.2; HRMS (ESI) calcd for $C_{14}H_{12}ClO_3S_2$ $[M+H]^+$ 326.9911, found 326.9907.



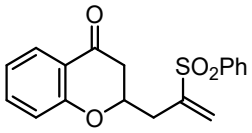
2-(2-(Phenylsulfonyl)allyl)tetrahydrofuran (3m): Colorless oil; (52%, 26.1 mg); Rf 0.5 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 6.8 Hz, 2H), 7.62 (t, *J* = 7.2 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 2H), 6.43 (s, 1H), 5.97 (s, 1H), 4.03-3.94 (m, 1H), 3.78-3.64 (m, 2H), 2.47-2.33 (m, 2H), 2.02-1.90 (m, 1H), 1.88-1.77 (m, 2H), 1.48-1.41 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.6, 138.7, 133.5, 129.2, 128.3, 125.3, 76.6, 67.9, 35.0, 31.2, 25.4; HRMS (ESI) calcd for $C_{13}H_{17}O_3S$ $[M+H]^+$ 253.0893, found 253.0892.



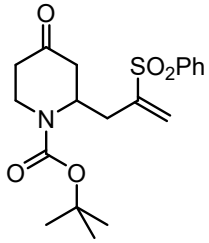
2-(2-(Phenylsulfonyl)allyl)-1,3,5-trioxane (3n): Colorless oil; (51%, 27.5 mg); Rf 0.5 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 7.2 Hz, 2H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.55 (t, *J* = 8.0 Hz, 2H), 6.48 (s, 1H), 5.98 (s, 1H), 5.14 (d, *J* = 6.4 Hz, 2H), 5.04 (t, *J* = 5.2 Hz, 1H), 5.00 (d, *J* = 6.8 Hz, 2H), 2.61 (d, *J* = 5.2 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.6, 138.4, 133.7, 129.3, 128.4, 128.3, 99.2, 93.2, 35.1; HRMS (ESI) calcd for $C_{12}H_{15}O_5S$ $[M+H]^+$ 271.0635, found 271.0632.



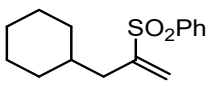
2-(2-(Phenylsulfonyl)allyl)tetrahydro-4*H*-pyran-4-one (3o): Colorless oil; (60%, 33.5 mg); Rf 0.4 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.2 Hz, 2H), 7.69-7.53 (m, 3H), 6.48 (s, 1H), 5.96 (s, 1H), 4.20-4.14 (m, 1H), 3.77-3.64 (m, 1H), 3.48 (t, *J* = 12.0 Hz, 1H), 2.59-2.42 (m, 3H), 2.37-2.16 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 205.5, 145.8, 138.6, 133.7, 129.3, 128.2, 126.9, 75.2, 66.2, 47.7, 41.8, 36.2; HRMS (ESI) calcd for $C_{14}H_{17}O_4S$ $[M+H]^+$ 281.0842, found 281.0839.



2-(2-(Phenylsulfonyl)allyl)chroman-4-one (3p): Colorless oil; (47%, 30.8 mg); Rf 0.35 (EtOAc/petroleum ether = 1:4); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.6 Hz, 2H), 7.85 (d, *J* = 7.6 Hz, 1H), 7.64 (t, *J* = 7.2 Hz, 1H), 7.56 (t, *J* = 8.0 Hz, 2H), 7.48-7.42 (m, 1H), 7.01 (t, *J* = 8.0 Hz, 1H), 6.87 (d, *J* = 8.0 Hz, 1H), 6.54 (s, 1H), 6.03 (s, 1H), 4.66 (ddd, *J* = 12.4, 8.5, 4.3 Hz, 1H), 2.82 (dd, *J* = 15.7, 8.3 Hz, 1H), 2.72-2.55 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.1, 160. 7, 145.5, 138.4, 136.1, 133.8, 129.4, 128.3, 127.3, 127.0, 121.7, 120.9, 117.8, 75.1, 42.4, 34.8; HRMS (ESI) calcd for $C_{18}H_{17}O_4S$ $[M+H]^+$ 329.0842, found 329.0828.

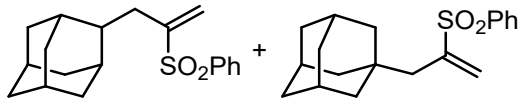


***tert*-Butyl 4-oxo-2-(2-(phenylsulfonyl)allyl)piperidine-1-carboxylate (3q):** Colorless oil; (36%, 27.3 mg); Rf 0.4 (EtOAc/petroleum ether = 1:3); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.6 Hz, 2H), 7.64 (t, *J* = 7.2 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 2H), 6.45 (s, 1H), 5.84 (brs, 1H), 5.02 (d, *J* = 6.8 Hz, 1H), 4.26 (brs, 1H), 3.02 (t, *J* = 11.6 Hz, 1H), 2.66 (dd, *J* = 14.8, 6.4 Hz, 1H), 2.47 (brs, 2H), 2.33-2.20 (m, 3H), 1.50 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.8, 207.3, 154.3, 146.2, 138.4, 133.7, 129.3, 128.3, 80.4, 44.7, 44.3, 41.1, 40.6, 28.3, 28.2; HRMS (ESI) calcd for $C_{19}H_{26}NO_5S$ $[M+H]^+$ 380.1526, found 380.1515.

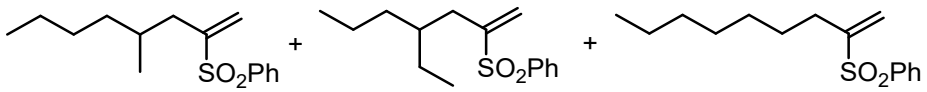


((3-Cyclohexylprop-1-en-2-yl)sulfonyl)benzene (3r): Colorless oil; (63%, 33.1 mg); Rf 0.5 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.6 Hz, 2H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.54 (t, *J* = 7.2 Hz, 2H), 6.40 (s, 1H), 5.70 (s, 1H), 2.11 (d, *J* = 7.2 Hz, 2H), 1.60 (t, *J* = 6.8 Hz, 5H), 1.43 (s, 1H), 1.17-1.02 (m, 3H), 0.79-

0.70 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.8, 139.1, 133.3, 129.1, 128.3, 124.3, 37.3, 35.7, 32.7, 26.2, 25.9; HRMS (ESI) calcd for $C_{15}H_{21}O_2S$ $[M+H]^+$ 265.1257, found 265.1262.

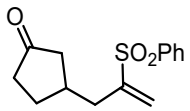


1-(2-(Phenylsulfonyl)allyl)adamantane (3s, left) + 2-(2-(phenylsulfonyl)allyl)-adamantane (3s', right): Colorless oil; a 1:1 mixture of 1- and 2-allylated products (28% total yield, 17.7 mg); Rf 0.5 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (t, *J* = 6.8 Hz, 2H), 7.61 (t, *J* = 7.2 Hz, 1H), 7.56-7.50 (m, 2H), 6.43 (s, 0.5H), 6.16 (s, 0.5H), 5.80 (s, 0.5H), 5.71 (s, 0.5H), 2.39 (d, *J* = 7.2 Hz, 1H), 2.05 (s, 1H), 1.93 (m, 1H), 1.84-1.69 (m, 3H), 1.69-1.60 (m, 4H), 1.60-1.54 (m, 3H), 1.48 (d, *J* = 2.9 Hz, 3H), 1.45-1.36 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.4, 147.5, 139.6, 139.2, 133.3, 133.2, 129.1, 129.0, 128.4, 128.2, 127.7, 124.0, 42.7, 42.3, 41.9, 38.9, 38.1, 36.7, 33.7, 32.6, 31.2, 31.1, 28.6, 27.9, 27.8; HRMS (ESI) calcd for $C_{19}H_{25}O_2S$ $[M+H]^+$ 317.1570, found 317.1560.

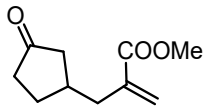


((4-Methyloct-1-en-2-yl)sulfonyl)benzene(3t, left) + ((4-ethylhept-1-en-2-yl)sulfon-yl)benzene(3t', middle) + (non-1-en-2-ylsulfonyl)benzene(3t'', right): Colorless oil; a 5.5:3.5:1 mixture of 2-, 3- and 1-allylated products (33% total yield, 17.5 mg); Rf 0.7 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.8 Hz, 2H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 2H), 6.42 (s, 1H), 5.72 (s, 1H), 2.24 (dd, *J* = 16.0, 6.4 Hz, 1H), 2.14 (d, *J* = 7.2 Hz, 0.6H), 2.00 (dd, *J* = 15.6, 8.4 Hz, 0.6H), 1.62 (brs, 0.7H), 1.53-1.40 (m, 0.7H), 1.26-1.05 (m, 6H), 1.04-0.96 (m, 0.6H), 0.89-0.70 (m, 5H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.6, 149.6, 149.4, 139.1, 139.0, 136.5, 133.4, 131.3, 129.1, 129.1, 128.3, 128.2, 126.1, 124.3, 124.3, 122.9, 121.5, 116.0, 37.1, 36.8, 36.1, 34.7, 33.8, 31.6, 31.1, 29.1, 28.9, 27.5, 26.7, 25.2, 22.7, 22.6, 22.5, 20.8, 19.4, 19.0, 14.2, 14.0, 10.4; HRMS (ESI) calcd for $C_{15}H_{23}O_2S$ $[M+H]^+$ 267.1413, found

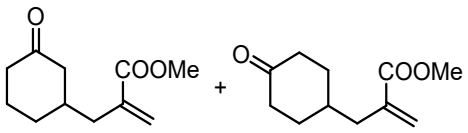
267.1397.



3-(2-(Phenylsulfonyl)allyl)cyclopentan-1-one (3u): Colorless oil; (57%, 30.1 mg); Rf 0.4 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.2 Hz, 2H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.56 (t, *J* = 8.0 Hz, 2H), 6.41 (s, 1H), 5.77 (s, 1H), 2.53-2.43 (m, 1H), 2.41-2.07 (m, 6H), 1.70 (dd, *J* = 18.0, 9.6 Hz, 1H), 1.50-1.40 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 217.7, 148.8, 138.5, 133.7, 129.3, 128.2, 124.7, 44.4, 38.1, 35.4, 35.1, 28.9; HRMS (ESI) calcd for $C_{14}H_{17}O_3S$ $[M+H]^+$ 265.0893, found 265.0893.

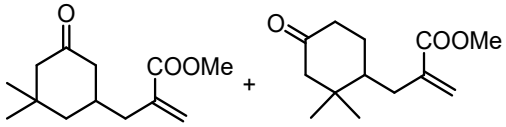


Methyl 2-((3-oxocyclopentyl)methyl)acrylate (3v): Colorless oil; (53%, 19.3 mg); Rf 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.20 (s, 1H), 5.57 (s, 1H), 3.76 (s, 3H), 2.49-2.23 (m, 5H), 2.22-2.07 (m, 2H), 1.88-1.80 (m, 1H), 1.59-1.50 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 218.9, 167.4, 138.5, 126.3, 51.9, 44.7, 38.2, 37.7, 35.9, 29.1; HRMS (ESI) calcd for $C_{10}H_{15}O_3$ $[M+H]^+$ 183.1016, found 183.1015.

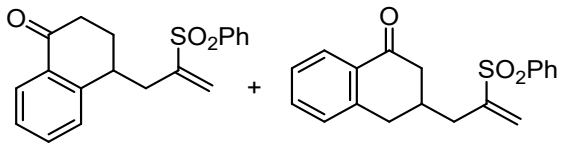


Methyl 2-((3-oxocyclohexyl)methyl)acrylate (3w, major, left) + methyl 2-((4-oxocyclo-hexyl)methyl)acrylate (3w', minor, right): Colorless oil; a 1.4:1 mixture of *β*- and *γ*-allylated products (61% total yield, 23.9 mg); Rf 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) **Major**: δ 6.20 (s, 1H), 5.54 (s, 1H), 3.76 (s, 3H); 2.55-2.15 (m, 6H), 2.08-1.88 (m, 3H), 1.74-1.53 (m, 1H), 1.46-1.25 (m, 2H); **Minor**: δ 6.20 (s, 1H), 5.52 (s, 1H), 3.73 (s, 3H); 2.55-2.15 (m, 6H), 2.08-1.88 (m, 3H), 1.74-1.53 (m, 1H), 1.46-1.25 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 211.8, 211.3, 167.5, 167.3,

138.5, 137.8, 126.9, 126.6, 51.9, 47.5, 41.3, 40.6, 39.0, 38.3, 37.8, 34.8, 32.3, 30.9, 24.9; HRMS (ESI) calcd for $C_{11}H_{17}O_3$ $[M+H]^+$ 197.1172, found 197.1173.

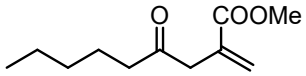


Methyl 2-((3,3-dimethyl-5-oxocyclohexyl)methyl)acrylate (3x, major, left) + methyl 2-((2,2-dimethyl-4-oxocyclohexyl)methyl)acrylate (3x', minor, right): Colorless oil; a 3.3:1 mixture of *β*- and *γ*-allylated products (53% total yield, 23.7 mg); Rf 0.3 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) **Major:** δ 6.20 (s, 1H), 5.53 (s, 1H), 3.74 (s, 3H), 2.40-2.24 (m, 3H), 2.20-2.03 (m, 3H), 1.86 (t, *J* = 12.8 Hz, 1H), 1.62 (d, *J* = 13.2 Hz, 1H), 1.28 (t, *J* = 12.8 Hz, 1H), 1.05 (s, 3H), 0.86 (s, 3H); **Minor**: δ 6.22 (s, 1H), 5.55 (s, 1H), 3.77 (s, 3H), 2.76 (d, *J* = 13.2 Hz, 1H), 2.39-2.05 (m, 2H), 1.97-1.90 (m, 2H), 1.79-1.71 (m, 2H), 1.49-1.36 (m, 2H), 1.11 (s, 3H), 0.82 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 211.8, 211.4, 167.5, 167.4, 139.3, 137.9, 126.8, 126.7, 55.8, 54.5, 51.9, 51.9, 50.8, 46.9, 44.9, 43.6, 40.6, 39.5, 35.1, 33.6, 32.6, 32.1, 29.7, 27.2, 25.7, 20.6; HRMS (ESI) calcd for $C_{13}H_{21}O_3$ $[M+H]^+$ 225.1485, found 225.1487.

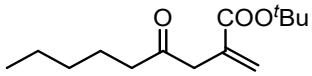


4-(2-(Phenylsulfonyl)allyl)-3,4-dihydronaphthalen-1(*2H*)-one (3y, major, left) + 3-(2-(phenylsulfonyl)allyl)-3,4-dihydronaphthalen-1(*2H*)-one (3y', minor, right): Colorless oil; a 4:1 mixture of *γ*- and *β*-allylated products (46% total yield, 29.7 mg); ^{1}H NMR (400 MHz, $CDCl_3$) **Major:** δ 8.00 (d, *J* = 7.6 Hz, 1H), 7.91 (d, *J* = 7.6 Hz, 2H), 7.65 (d, *J* = 7.2 Hz, 1H), 7.57 (t, *J* = 8.0 Hz, 2H), 7.46 (t, *J* = 7.2 Hz, 1H), 7.32 (t, *J* = 7.6 Hz, 1H), 7.21 (d, *J* = 8.0 Hz, 1H), 6.48 (s, 1H), 5.68 (s, 1H), 3.37-3.29 (m, 1H), 2.72-2.45 (m, 4H), 2.25-2.14 (m, 1H), 2.01-1.92 (m, 1H); **minor:** δ 8.00 (d, *J* = 7.6 Hz, 1H), 7.91 (d, *J* = 7.6 Hz, 2H), 7.65 (d, *J* = 7.2 Hz, 1H), 7.57 (t, *J* = 8.0 Hz, 2H), 7.46 (t, *J* = 7.2 Hz, 1H), 7.32 (t, *J* = 7.6 Hz, 1H), 7.21 (d, *J* = 8.0 Hz, 1H), 6.46 (s, 1H), 5.78 (s,

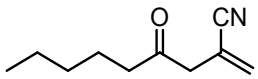
1H), 2.98 (d, *J* = 15.6 Hz, 1H), 2.72-2.45 (m, 4H), 2.36 (dd, *J* = 20.4, 7.2 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.4, 148.0, 145.9, 138.6, 133.8, 133.6, 131.8, 129.4, 128.4, 128.3, 127.5, 127.3, 126.1, 36.1, 35.2, 34.2, 25.7; HRMS (ESI) calcd for $C_{19}H_{19}O_3S$ $[M+H]^+$ 327.1049, found 327.1047.



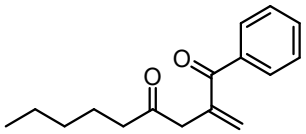
Methyl 2-methylene-4-oxononanoate (4a): Colorless oil; (64%, 25.3 mg); Rf 0.4 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.33 (s, 1H), 5.63 (s, 1H), 3.74 (s, 3H), 3.39 (s, 2H), 2.47 (t, *J* = 7.2 Hz, 2H), 1.63-1.55 (m, 2H), 1.33-1.24 (m, 4H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.4, 166.8, 134.3, 128.5, 52.0, 45.7, 42.6, 31.3, 23.4, 22.4, 13.9; HRMS (ESI) calcd for $C_{11}H_{19}O_3$ $[M+H]^+$ 199.1329, found 199.1332.



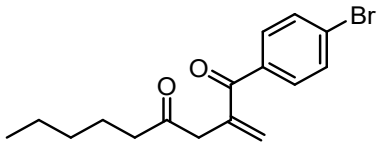
Tert-butyl 2-methylene-4-oxononanoate (4b): Colorless oil; (61%, 29.3 mg); Rf 0.4 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.24 (s, 1H), 5.54 (s, 1H), 3.34 (s, 2H), 2.46 (t, *J* = 7.2 Hz, 2H), 1.63-1.53 (m, 2H), 1.46 (s, 9H), 1.33-1.23 (m, 4H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.6, 165.5, 136.0, 127.5, 81.1, 45.9, 42.5, 31.3, 27.9, 23.4, 22.4, 13.9; HRMS (ESI) calcd for $C_{14}H_{25}O_3$ $[M+H]^+$ 241.1798, found 241.1802.



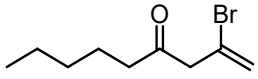
2-Methylene-4-oxononanenitrile (4c): Colorless oil; (70%, 23.1 mg); Rf 0.5 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.07 (s, 1H), 5.81 (s, 1H), 3.35 (s, 2H), 2.49 (t, *J* = 7.2 Hz, 2H), 1.64-1.56 (m, 2H), 1.35-1.24 (m, 4H), 0.89 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.5, 134.4, 118.0, 115.9, 47.0, 42.6, 31.2, 23.2, 22.3, 13.8; HRMS (ESI) calcd for $C_{10}H_{16}NO$ $[M+H]^+$ 166.1226, found 166.1227.



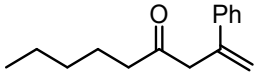
2-Methylene-1-phenylnonane-1,4-dione (4d): Colorless oil; (63%, 30.7 mg); Rf 0.5 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82-7.77 (m, 2H), 7.58-7.49 (m, 1H), 7.43 (t, *J* = 7.6 Hz, 2H), 5.91 (s, 1H), 5.77 (s, 1H), 3.61 (s, 2H), 2.52 (t, *J* = 7.6 Hz, 2H), 1.64-1.53 (m, 2H), 1.36-1.22 (m, 4H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.8, 197.3, 142.1, 137.3, 132.2, 129.7, 128.9, 128.1, 46.3, 42.7, 31.3, 23.4, 22.4, 13.8; HRMS (ESI) calcd for $C_{16}H_{21}O_2$ $[M+H]^+$ 245.1536, found 245.1540.



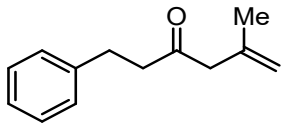
1-(4-Bromophenyl)-2-methylenenonane-1,4-dione (4e): Colorless oil; (44%, 28.3 mg); Rf 0.7 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 7.6 Hz, 2H), 7.58 (d, *J* = 8.0 Hz, 2H), 5.89 (s, 1H), 5.72 (s, 1H), 3.62 (s, 2H), 2.51 (t, *J* = 7.2 Hz, 2H), 1.63-1.56 (m, 2H), 1.33-1.24 (m, 4H), 0.88 (t, *J* = 5.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.8, 196.3, 142.0, 136.0, 131.5, 131.3, 128.6, 127.3, 46.5, 42.7, 31.3, 23.4, 22.4, 13.9; HRMS (ESI) calcd for $C_{16}H_{20}BrO_2$ $[M+H]^+$ 323.0641, found 323.0640.



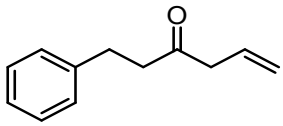
2-Bromonon-1-en-4-one (4f): Colorless oil; (42%, 18.3 mg); Rf 0.4 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.74 (s, 1H), 5.65 (s, 1H), 3.54 (s, 2H), 2.51 (t, *J* = 7.2 Hz, 2H), 1.64-1.53 (m, 2H), 1.33-1.24 (m, 4H), 0.89 (t, *J* = 5.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 205.5, 124.7, 121.4, 54.6, 42.1, 31.2, 23.2, 22.4, 13.9; HRMS (ESI) calcd for $C_9H_{16}BrO$ $[M+H]^+$ 219.0379, found 219.0381.



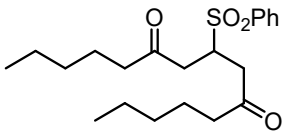
2-Phenylnon-1-en-4-one (4g): Colorless oil; (54%, 23.3 mg); Rf 0.6 (EtOAc/petroleum ether = 1:20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 7.2 Hz, 2H), 7.29-7.18 (m, 3H), 5.52 (s, 1H), 5.13 (s, 1H), 3.51 (s, 2H), 2.36 (t, *J* = 7.2 Hz, 2H), 1.50-1.40 (m, 2H), 1.28-1.07 (m, 4H), 0.78 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 208.9, 141.6, 139.9, 128.5, 127.8, 125.8, 116.5, 50.2, 41.6, 31.3, 23.4, 22.4, 13.9; HRMS (ESI) calcd for $C_{15}H_{21}O$ $[M+H]^+$ 217.1587, found 217.1588.



5-Methyl-1-phenylhex-5-en-3-one (4h): Colorless oil; (39%, 14.7 mg); Rf 0.6 (EtOAc/petroleum ether = 1:50); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 7.6 Hz, 2H), 7.24 (t, *J* = 6.4 Hz, 3H), 4.98 (s, 1H), 4.84 (s, 1H), 3.14 (s, 2H), 2.95 (d, *J* = 7.2 Hz, 2H), 2.84 (d, *J* = 7.2 Hz, 2H), 1.76 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.9, 141.0, 139.1, 128.5, 128.3, 126.1, 115.8, 52.4, 43.3, 29.8, 22.6; HRMS (ESI) calcd for $C_{13}H_{17}O$ $[M+H]^+$ 189.1274, found 189.1279.

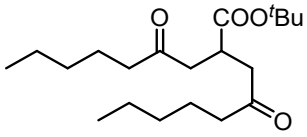


1-Phenylhex-5-en-3-one (4i)： Colorless oil; (51%, 17.7 mg); Rf 0.6 (EtOAc/petroleum ether = 1:50); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (d, *J* = 7.6 Hz, 2H), 7.15 (t, *J* = 7.6 Hz, 3H), 5.92-5.81 (m, 1H), 5.16-5.03 (m, 2H), 3.11 (d, *J* = 7.2 Hz, 2H), 2.86 (t, *J* = 7.6 Hz, 2H), 2.73 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.7, 140.9, 130.4, 128.5, 128.3, 126.1, 118.9, 47.9, 43.8, 29.7; HRMS (ESI) calcd for $C_{12}H_{15}O$ $[M+H]^+$ 175.1117, found 175.1113.

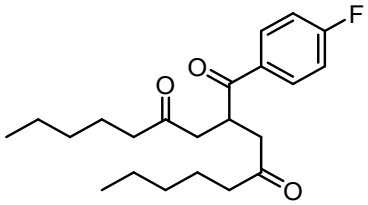


8-(Phenylsulfonyl)pentadecane-6,10-dione (5a)： Colorless oil; (66%, 50.2 mg); Rf 0.5 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.2 Hz,

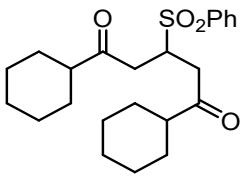
2H), 7.60 (t, *J* = 7.6 Hz, 1H), 7.50 (t, *J* = 8.0 Hz, 2H), 4.27-4.18 (m, 1H), 2.95 (dd, *J* = 17.6, 5.2 Hz, 2H), 2.58 (dd, *J* = 17.6, 7.2 Hz, 2H), 2.30 (t, *J* = 7.6 Hz, 4H), 1.47-1.36 (m, 4H), 1.26-1.07 (m, 8H), 0.80 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.0, 137.3, 134.0, 129.3, 128.8, 55.5, 42.8, 40.5, 31.1, 23.1, 22.3, 13.8; HRMS (ESI) calcd for $C_{21}H_{33}O_4S$ $[M+H]^+$ 381.2094, found 381.2085.



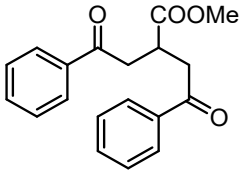
***tert*-Butyl 4-oxo-2-(2-oxoheptyl)nonanoate (5b)：** Colorless oil; (61%, 41.5 mg); Rf 0.5 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.20-3.11 (m, 1H), 2.78 (dd, *J* = 17.6, 6.4 Hz, 2H), 2.59 (dd, *J* = 17.6, 6.4 Hz, 2H), 2.38 (t, *J* = 7.2 Hz, 4H), 1.60-1.51 (m, 4H), 1.40 (s, 9H), 1.35-1.20 (m, 8H), 0.87 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 208.9, 173.2, 80.8, 43.4, 42.8, 36.5, 31.4, 27.9, 23.4, 22.4, 13.8; HRMS (ESI) calcd for $C_{20}H_{37}O_4$ $[M+H]^+$ 341.2686, found 341.2696.



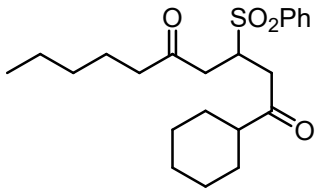
8-(4-Fluorobenzoyl)pentadecane-6,10-dione (5c): Colorless oil; (45%, 32.6 mg); Rf 0.5 (EtOAc/petroleum ether = 1:10); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (dd, *J* = 8.8, 5.6 Hz, 2H), 7.13 (t, *J* = 8.8 Hz, 2H), 4.36-4.28 (m, 1H), 2.84 (dd, *J* = 17.6, 6.8 Hz, 2H), 2.59 (dd, *J* = 17.6, 6.4 Hz, 2H), 2.37 (t, *J* = 7.2 Hz, 4H), 1.57-1.47 (m, 4H), 1.31-1.19 (m, 8H), 0.86 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 208.6, 200.8, 165.8 (d, *J* = 255.9 Hz), 132.4 (d, *J* = 2.9 Hz), 131.2 (d, *J* = 9.3 Hz), 115.7 (d, *J* = 21.9 Hz), 44.0, 42.8, 36.5, 31.3, 23.4, 22.4, 13.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -105.33; HRMS (ESI) calcd for $C_{22}H_{32}FO_3$ $[M+H]^+$ 363.2330, found 363.2319.



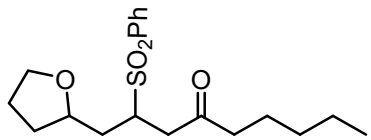
1,5-Dicyclohexyl-3-(phenylsulfonyl)pentane-1,5-dione (5d): White solid; (58%, 46.8 mg); Rf 0.5 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.6 Hz, 2H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 2H), 4.35-4.26 (m, 1H), 3.07 (dd, *J* = 18.0, 5.6 Hz, 2H), 2.66 (dd, *J* = 18.0, 7.2 Hz, 2H), 2.33-2.25 (m, 2H), 1.79-1.70 (m, 8H), 1.63 (d, *J* = 11.2 Hz, 2H), 1.26 -1.10 (m, 10H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 208.7, 137.5, 133.9, 129.2, 128.8, 55.3, 50.7, 38.7, 28.3, 28.2, 25.7, 25.5, 25.4; HRMS (ESI) calcd for $C_{23}H_{33}O_4S$ $[M+H]^+$ 405.2094, found 405.2083.



Methyl 4-oxo-2-(2-oxo-2-phenylethyl)-4-phenylbutanoate (5e): Colorless oil; (71%, 43.9 mg); Rf 0.6 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.6 Hz, 4H), 7.56 (t, *J* = 7.2 Hz, 2H), 7.45 (t, *J* = 7.6 Hz, 4H), 3.70 (s, 3H), 3.68-3.61 (m, 1H), 3.58 (dd, *J* = 18.0, 5.6 Hz, 2H), 3.38 (dd, *J* = 18.0, 6.4 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.8, 174.8, 136.5, 133.3, 128.6, 128.1, 52.6, 39.5, 35.9; HRMS (ESI) calcd for $C_{19}H_{19}O_4$ $[M+H]^+$ 311.1278, found 311.1283.



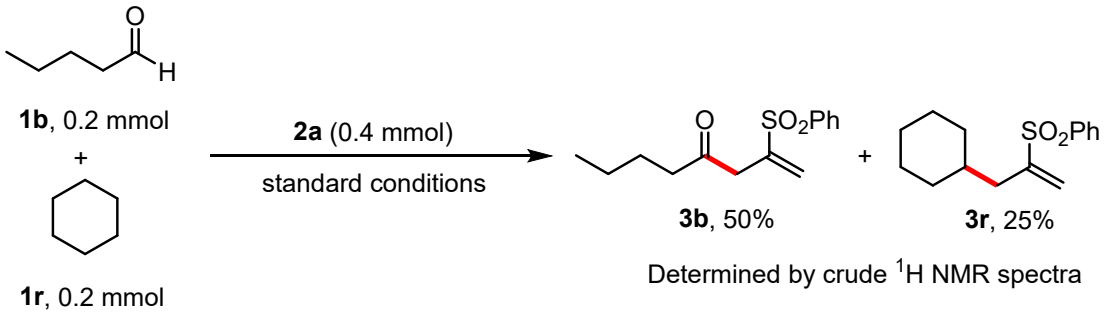
1-Cyclohexyl-3-(phenylsulfonyl)decane-1,5-dione (5f): Colorless oil; (72%, 56.6 mg); Rf 0.5 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.6 Hz, 2H), 7.64 (t, *J* = 7.2 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 2H), 4.35-4.23 (m, 1H), 3.03 (ddd, *J* = 22.8, 18.0, 5.2 Hz, 2H), 2.64 (ddd, *J* = 25.2, 18.0, 5.2 Hz, 2H), 2.34 (t, *J* = 7.2 Hz, 2H), 2.31-2.23 (m, 1H), 1.79-1.70 (m, 4H), 1.62 (d, *J* = 12.0 Hz, 1H), 1.50-1.40 (m, 2H), 1.30-1.10 (m, 9H), 0.84 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 208.8, 205.9, 137.4, 133.9, 129.2, 128.7, 55.4, 50.7, 42.7, 40.6, 38.5, 31.1, 28.3, 28.1, 25.6, 25.4, 25.4, 23.1, 22.3, 13.8; HRMS (ESI) calcd for $C_{22}H_{33}O_4S$ $[M+H]^+$ 393.2094, found 393.2096.



2-(Phenylsulfonyl)-1-(tetrahydrofuran-2-yl)nonan-4-one (5g): Colorless oil; (54%, 38.1 mg); Rf 0.4 (EtOAc/petroleum ether = 1:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 7.6 Hz, 2H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.56 (t, *J* = 7.6 Hz, 2H), 4.00-3.91 (m, 1H), 3.77-3.70 (m, 1H), 3.69-3.60 (m, 1H), 3.61-3.50 (m, 1H), 3.04-2.80 (m, 2H), 2.45-2.30 (m, 2H), 2.09 (dt, *J* = 14.0, 3.2 Hz, 1H), 1.99-1.90 (m, 1H), 1.85-1.70 (m, 2H), 1.66-1.59 (m, 1H), 1.55-1.45 (m, 2H), 1.33-1.16 (m, 5H), 0.86 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.1, 137.5, 133.8, 129.2, 128.8, 77.2, 67.7, 58.4, 42.8, 40.9, 34.5, 32.2, 31.3, 24.9, 23.1, 22.4, 13.9; HRMS (ESI) calcd for $C_{19}H_{29}O_4S$ $[M+H]^+$ 353.1781, found 353.1782.

5 Competition Experiment

We examined the relative reactivity of allylation product formation in competition experiment between aldehydic C(sp^2)–H bonds and unactivated C(sp^3)–H bonds. Reaction of pentanal **1b** and cyclohexane **1r** (in a 1:1 ratio) with allylic sulfone **2a** led to a 2:1 ratio of allylation of **3b** to **3r**. This result indicated that aldehydic C(sp^2)–H bonds allylation was preferred over aliphatic C(sp^3)–H bonds allylation. (Figure S1).



A 10 mL oven-dried sealed tube equipped with a magnetic stir bar was charged with TBADT (13.4 mg, 0.004 mmol, 2 mol%), $NaH_2PO_4 \cdot 2H_2O$ (46.8 mg, 0.3 mmol, 1.5 equiv) and allyl sulfone **2a** (0.4 mmol, 2.0 equiv). The tube was evacuated and backfilled with nitrogen (three times). Next, pentanal **1b** (22 μL, 0.2 mmol), cyclohexane **1r** (21 μL, 0.2 mmol) and MeCN (0.75 mL) and distilled water (0.25 mL) were subsequently injected into the tube by syringe under nitrogen atmosphere. The reaction mixture was stirred and irradiated using 365-370 nm LED lamps for 6 h at 25 ^oC. Next, 1,3,5-trimethoxybenzene (33.6 mg, 0.2 mmol) was added to the reaction mixture as an internal standard, and the reaction mixture was analyzed by crude ^1H NMR spectra.

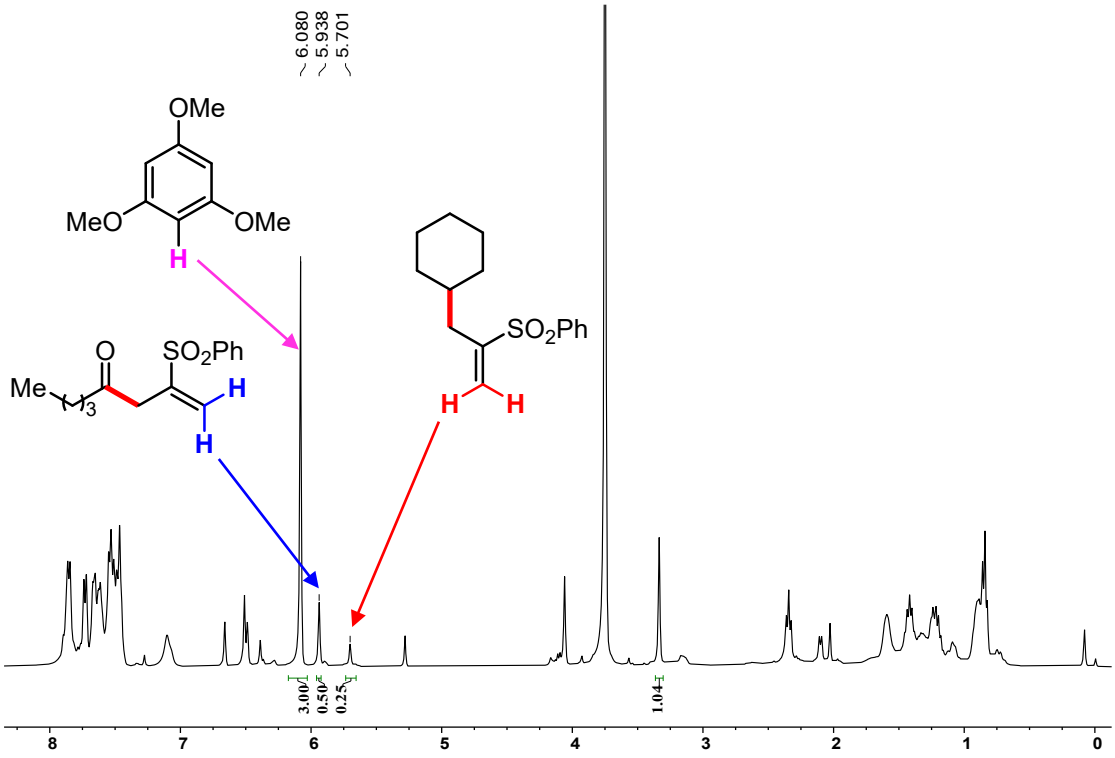
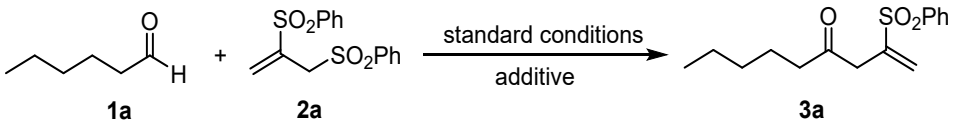


Figure S1. Competition experiment between aldehydic C(sp^2)–H bond and aliphatic C(sp^3)–H bond.

6 Mechanistic Studies

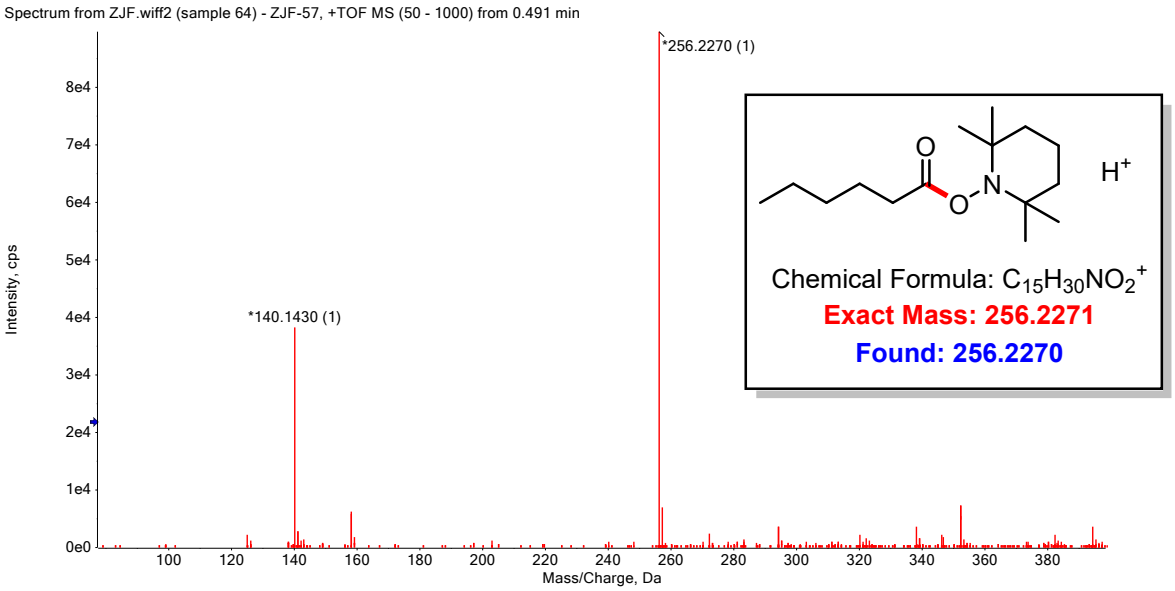
Table S3 Radical inhibiting experiment



1a 2a 3a

Entry	additive	Isolated of **3a**
1	2.0 equiv of TEMPO	0%
2	2.0 equiv of BHT	0%

When 2.0 equiv of radical scavenger TEMPO was added to the reaction of **1a** with **2a** under the standard conditions, the reaction was totally inhibited and the TEMPO–adduct intermediate could be detected by LC-MS (HRMS (ESI) calcd for $C_{15}H_{30}NO_2$ $[M+H]^+$ 256.2271, found 256.2270. In addition, this reaction was also completely suppressed in the presence of 2.0 equiv of BHT. These results strongly indicated that a radical process might be involved in this reaction.

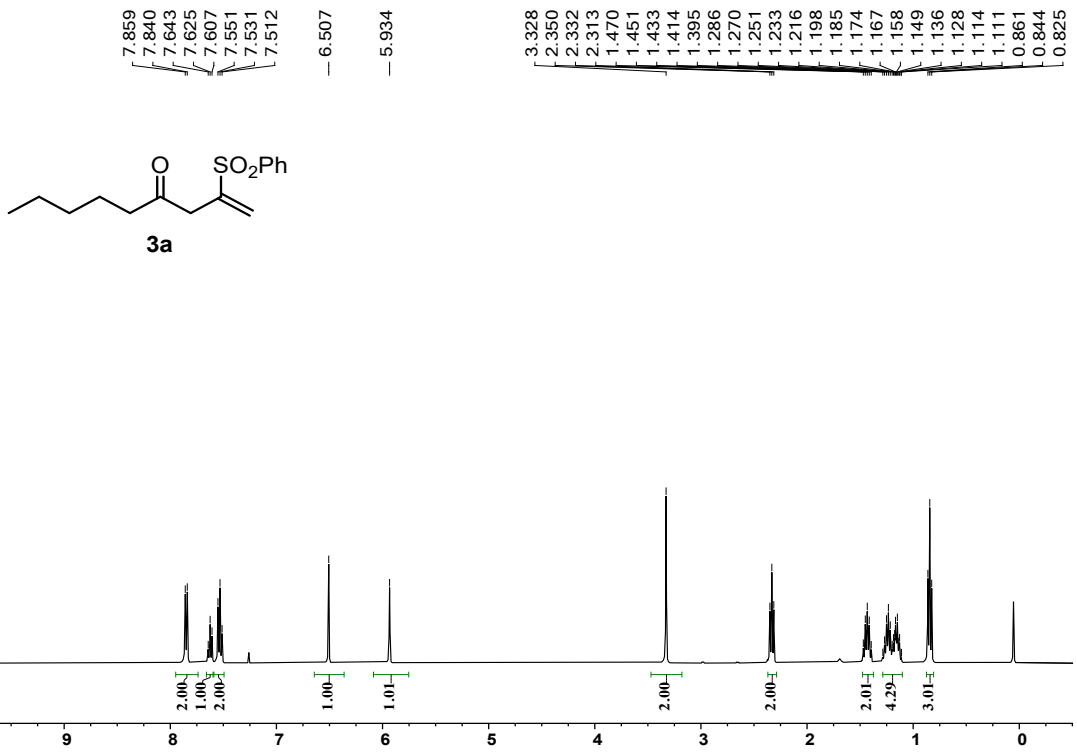


7 References

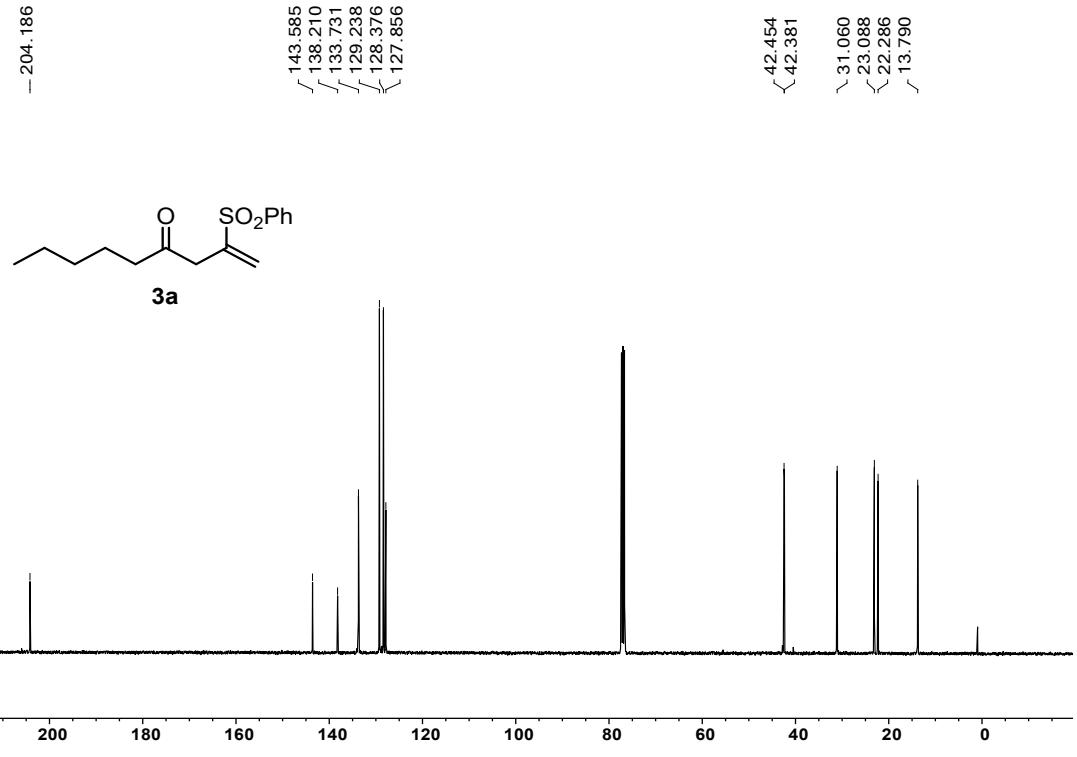
1. (a) S. Protti, D. Ravelli, M. Fagnoni and A. Albini, *Chem. Commun.*, 2009, 7351-7353; (b) I. B. Perry, T. F. Brewer, P. J. Sarver, D. M. Schultz, D. A. DiRocco and D. W. C. MacMillan, *Nature*, 2018, **560**, 70-75.
2. L. Liang, G. Guo, C. Li, S.-L. Wang, Y.-H. Wang, H.-M. Guo and H.-Y. Niu, *Org. Lett.*, 2021, **23**, 8575-8579.

8 ^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra of Products

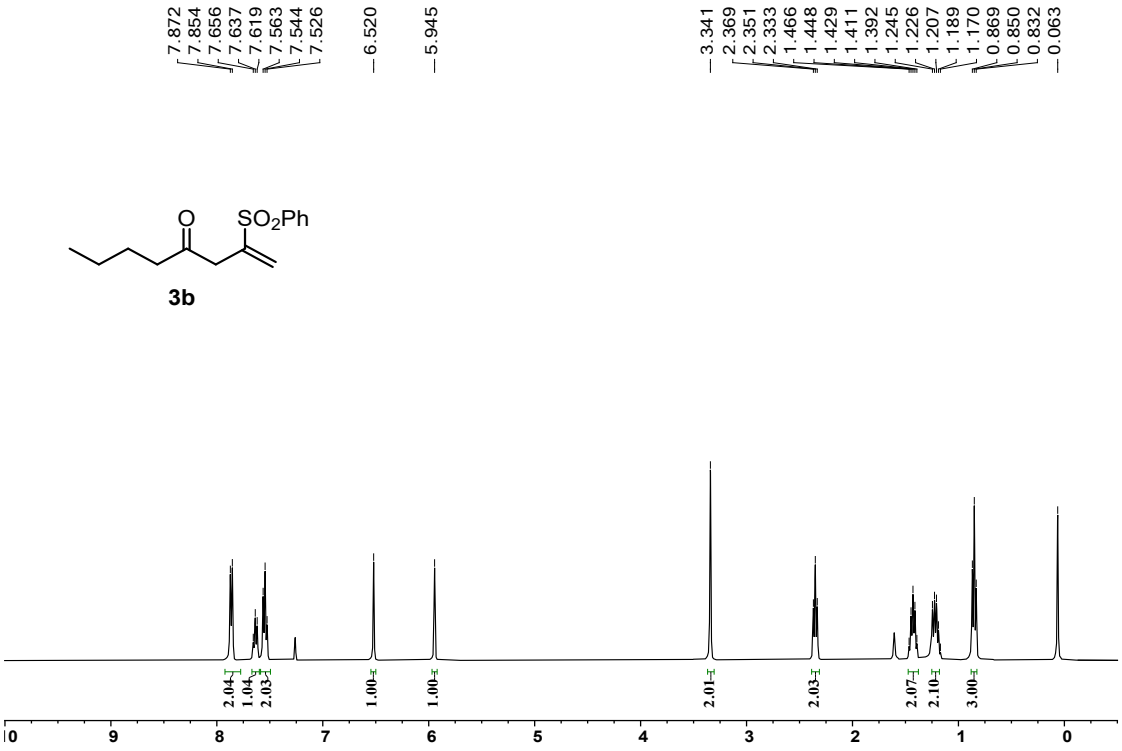
^{1}H NMR of **3a** (400 MHz, $CDCl_3$)



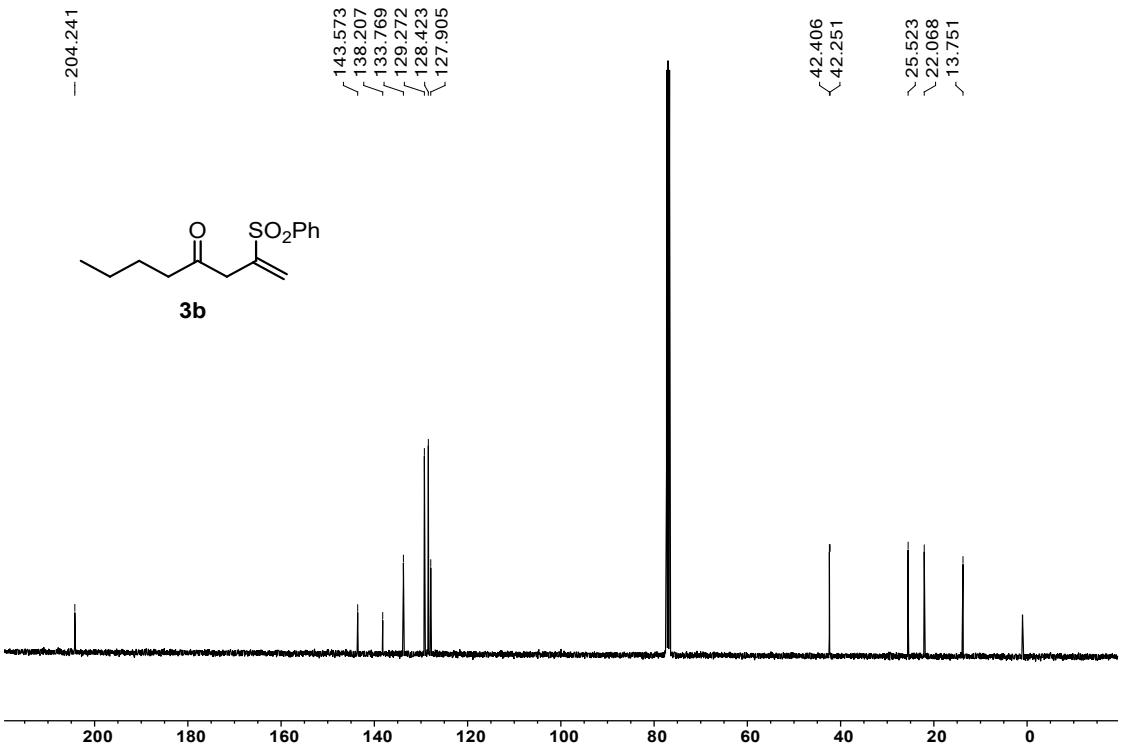
^{13}C NMR of **3a** (101 MHz, $CDCl_3$)



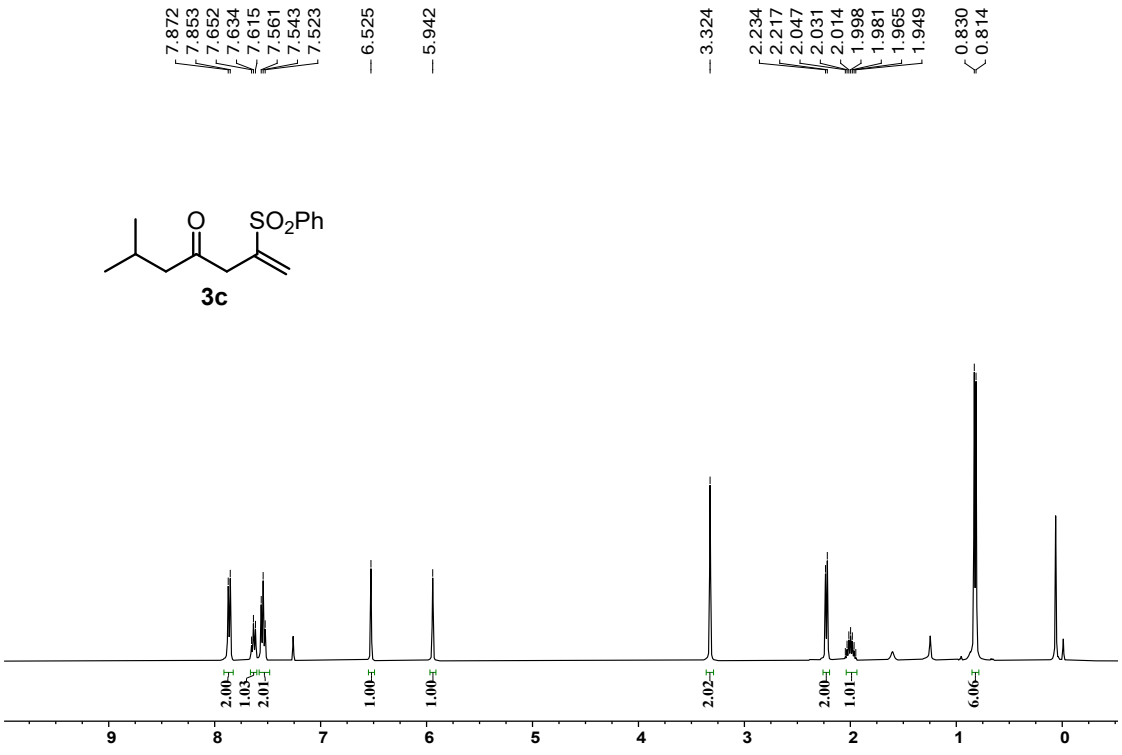
^{1}H NMR of **3b** (400 MHz, $CDCl_3$)



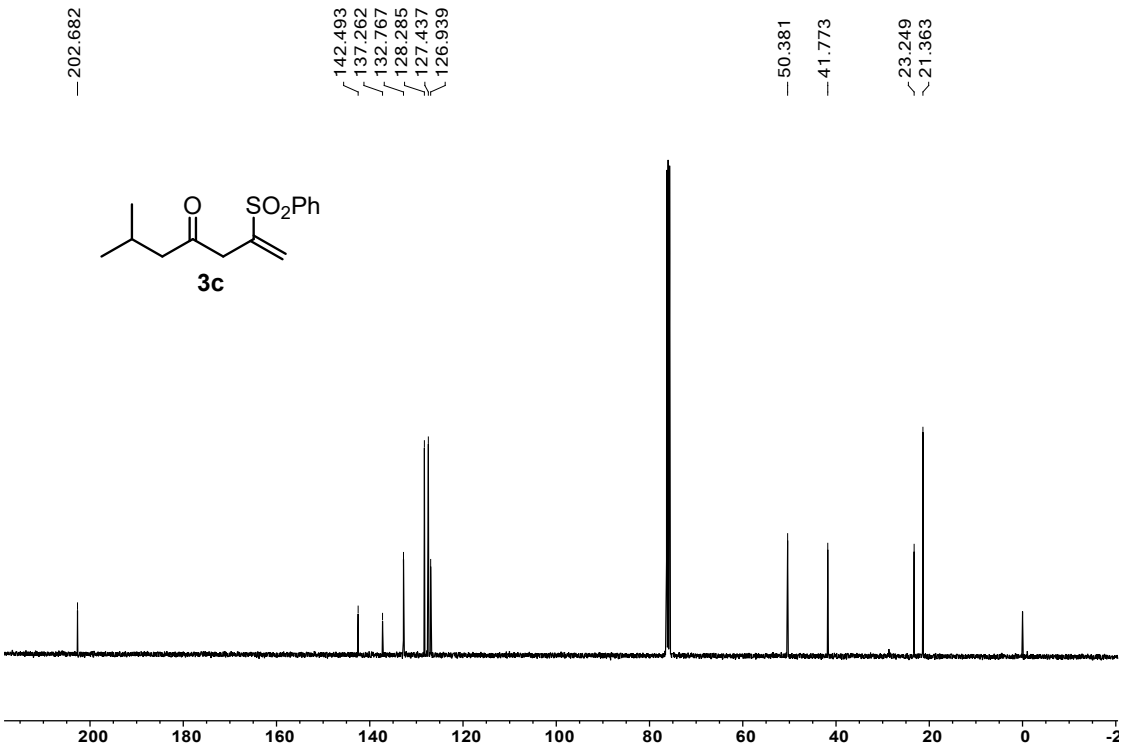
^{13}C NMR of **3b** (101 MHz, $CDCl_3$)



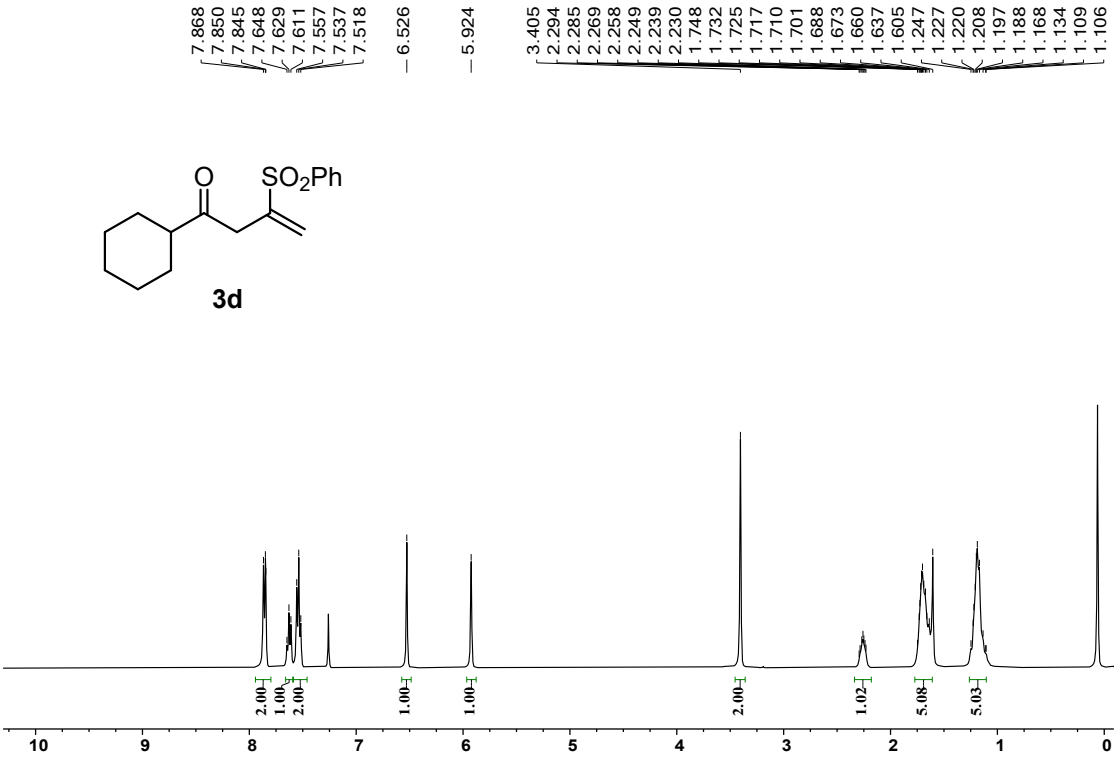
^{1}H NMR of **3c** (400 MHz, $CDCl_3$)



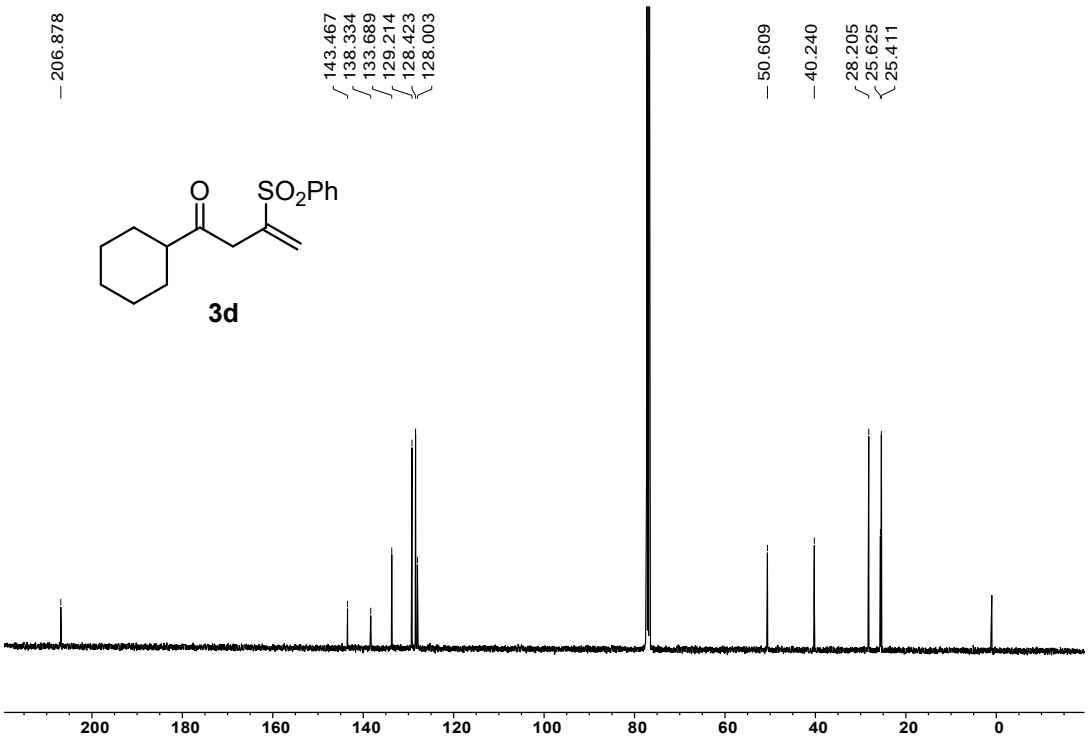
^{13}C NMR of **3c** (101 MHz, $CDCl_3$)



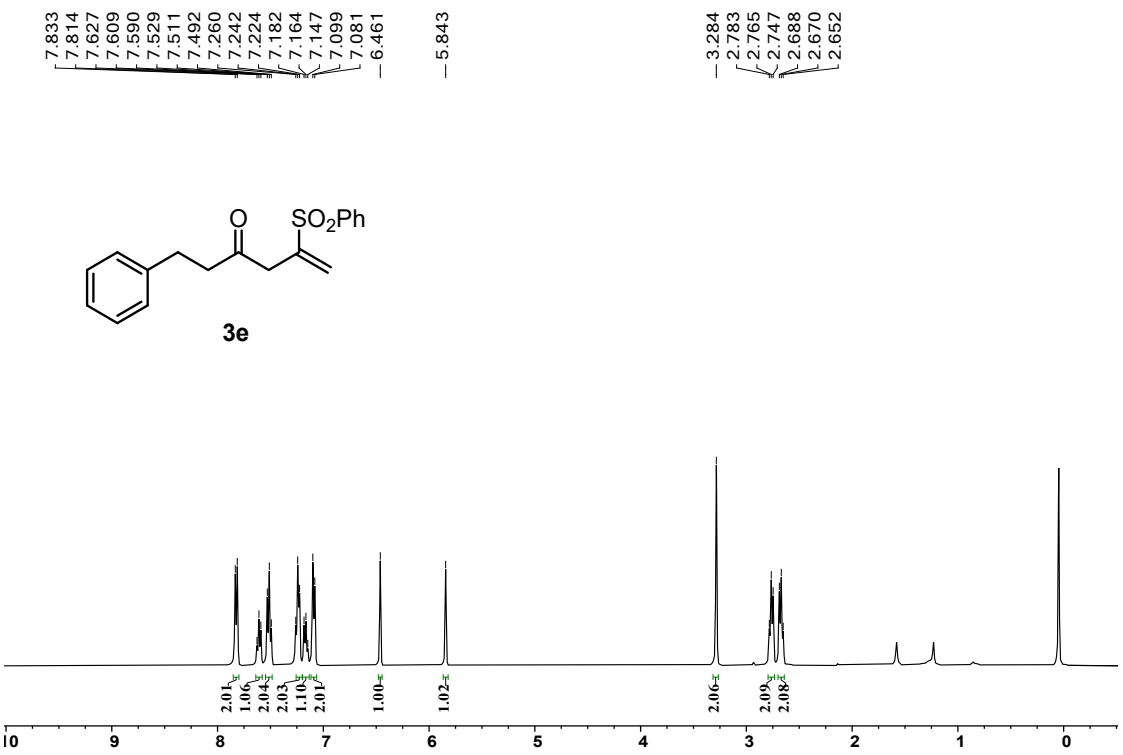
^{1}H NMR of **3d** (400 MHz, $CDCl_3$)



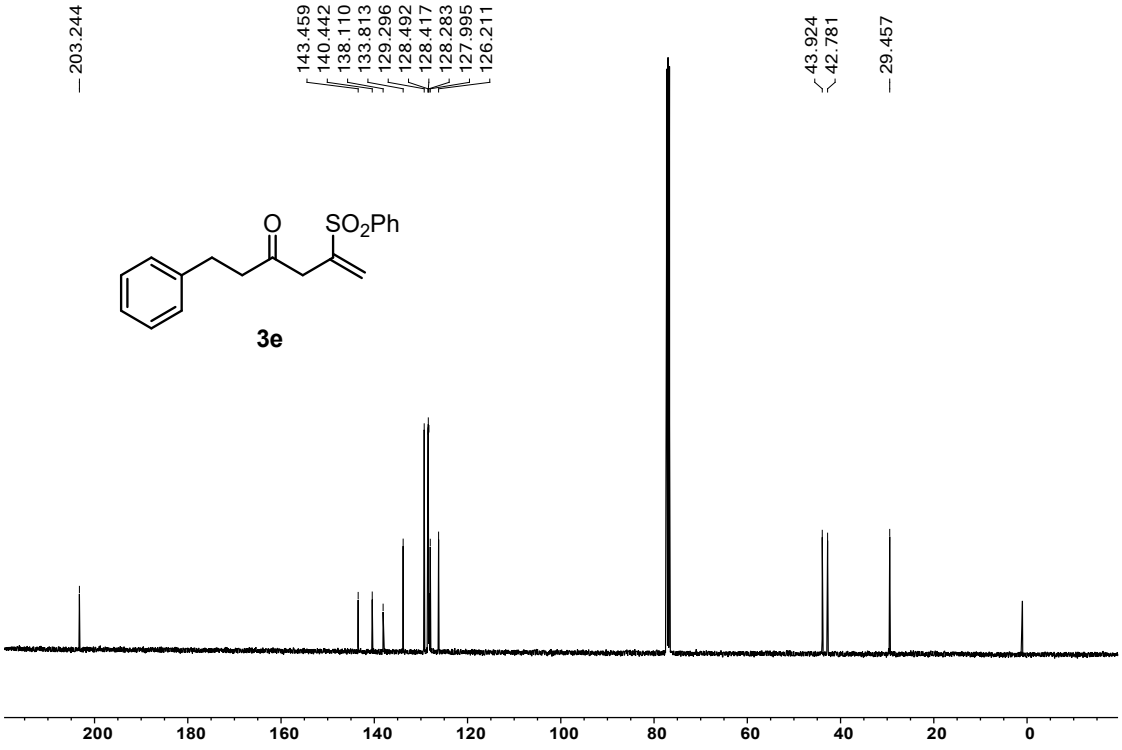
^{13}C NMR of **3d** (101 MHz, $CDCl_3$)



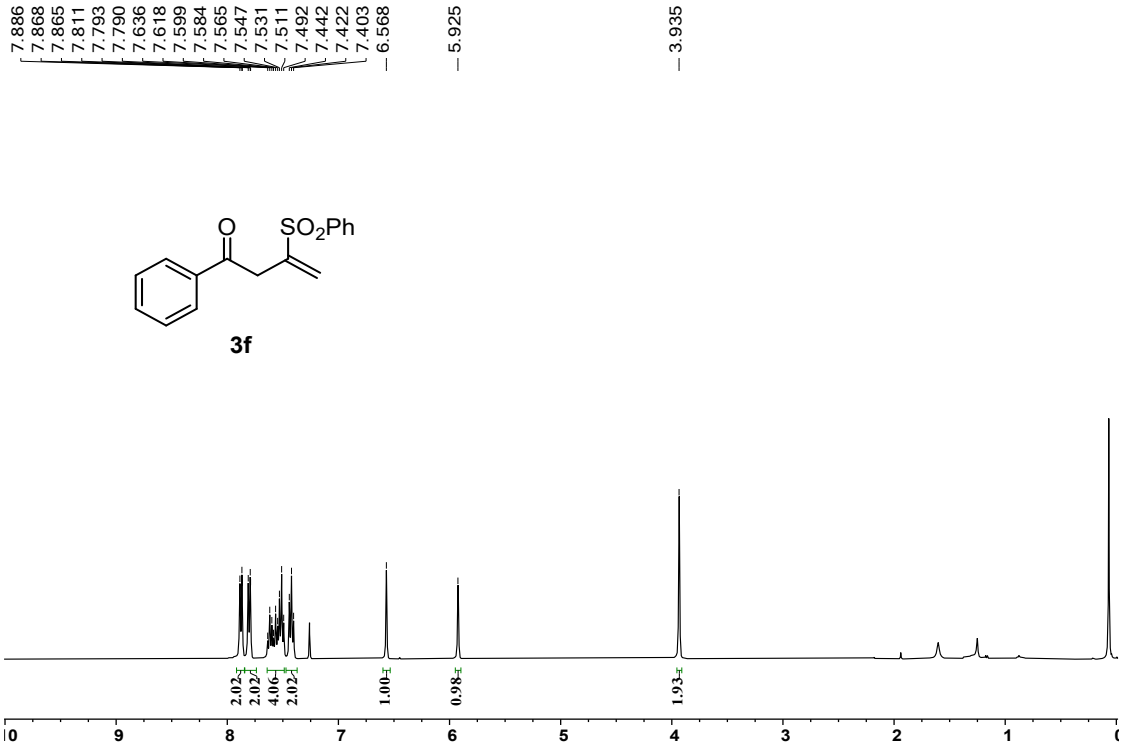
^{1}H NMR of **3e** (400 MHz, $CDCl_3$)



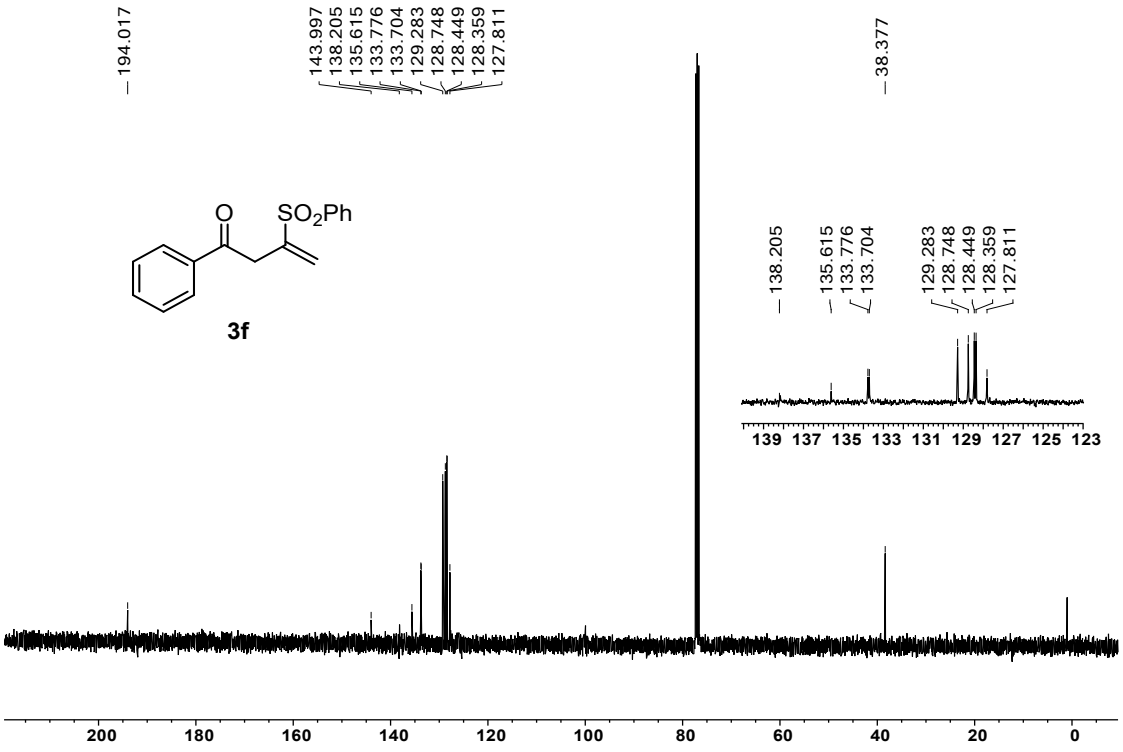
^{13}C NMR of **3e** (101 MHz, $CDCl_3$)



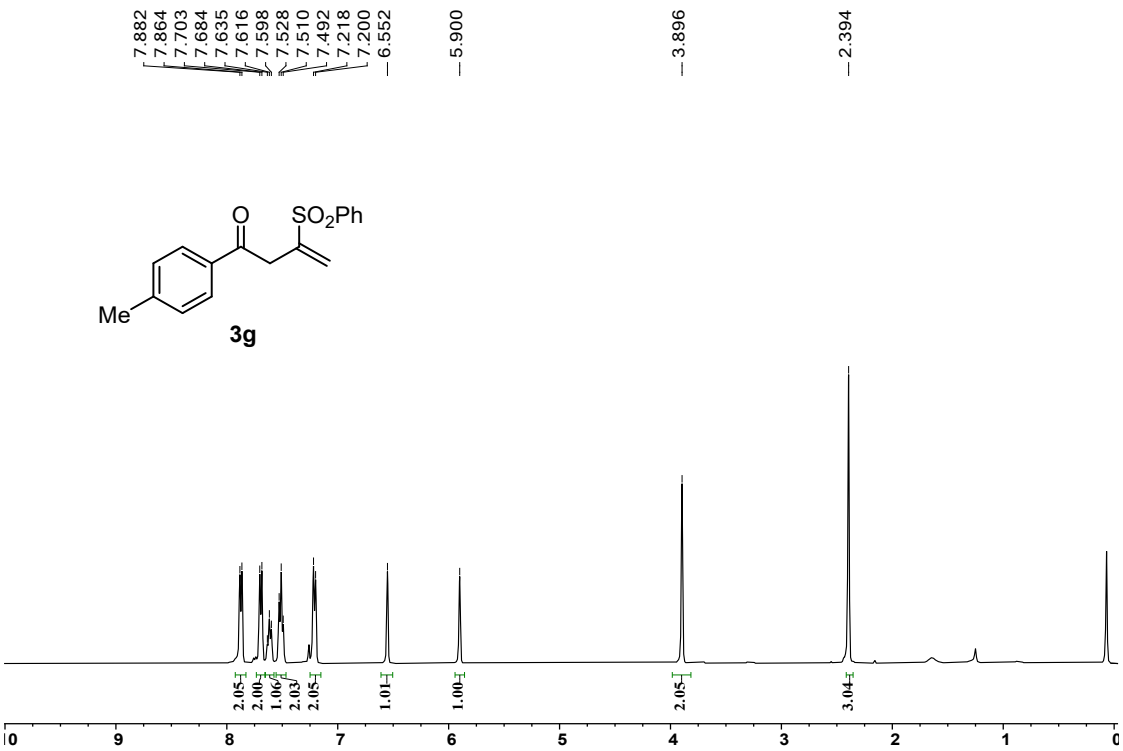
^{1}H NMR of **3f** (400 MHz, $CDCl_3$)



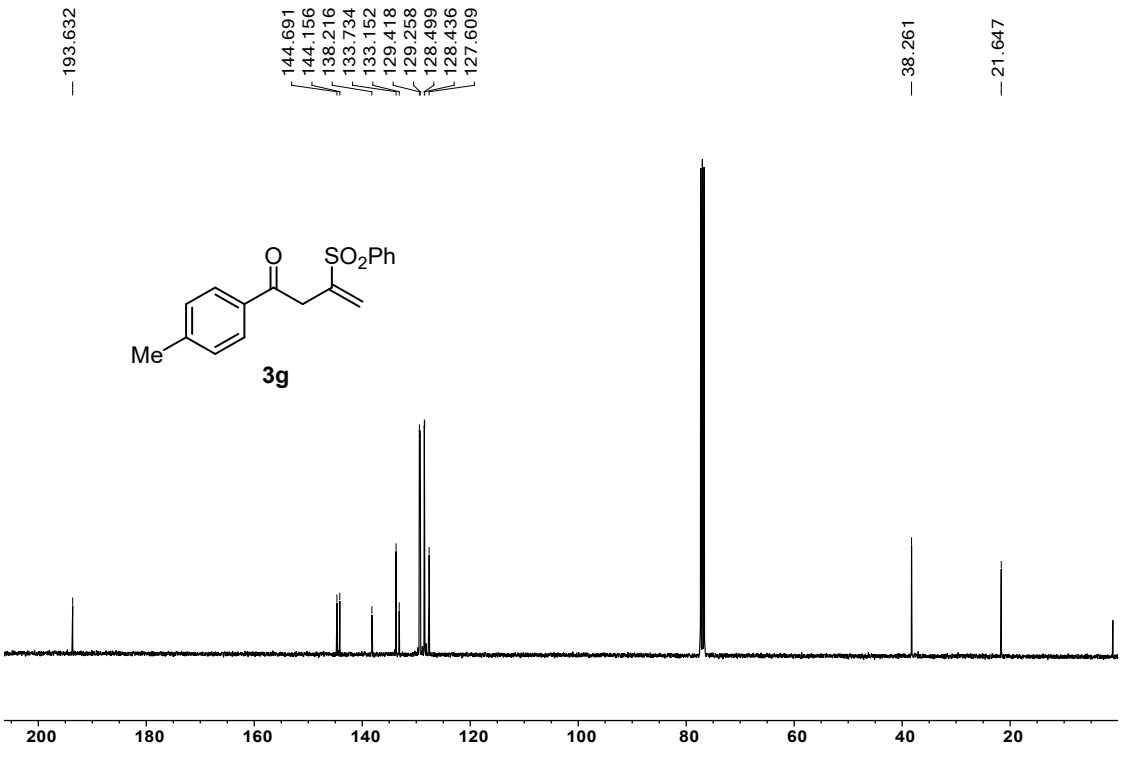
^{13}C NMR of **3f** (101 MHz, $CDCl_3$)



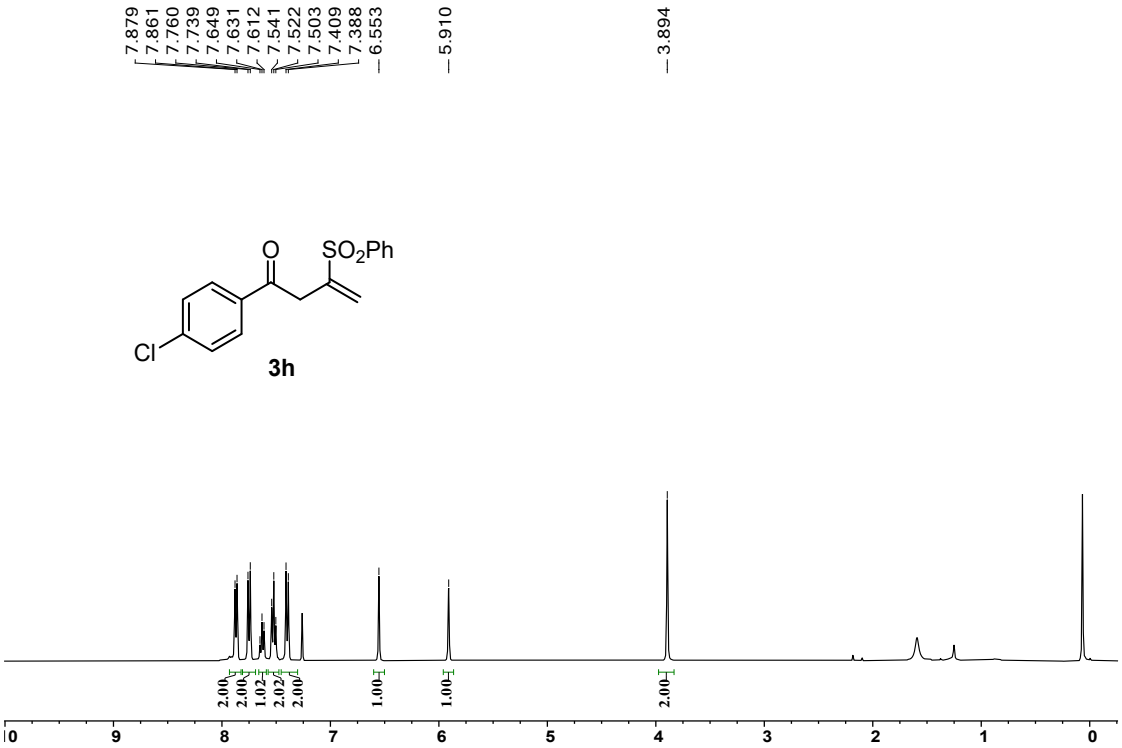
^{1}H NMR of **3g** (400 MHz, $CDCl_3$)



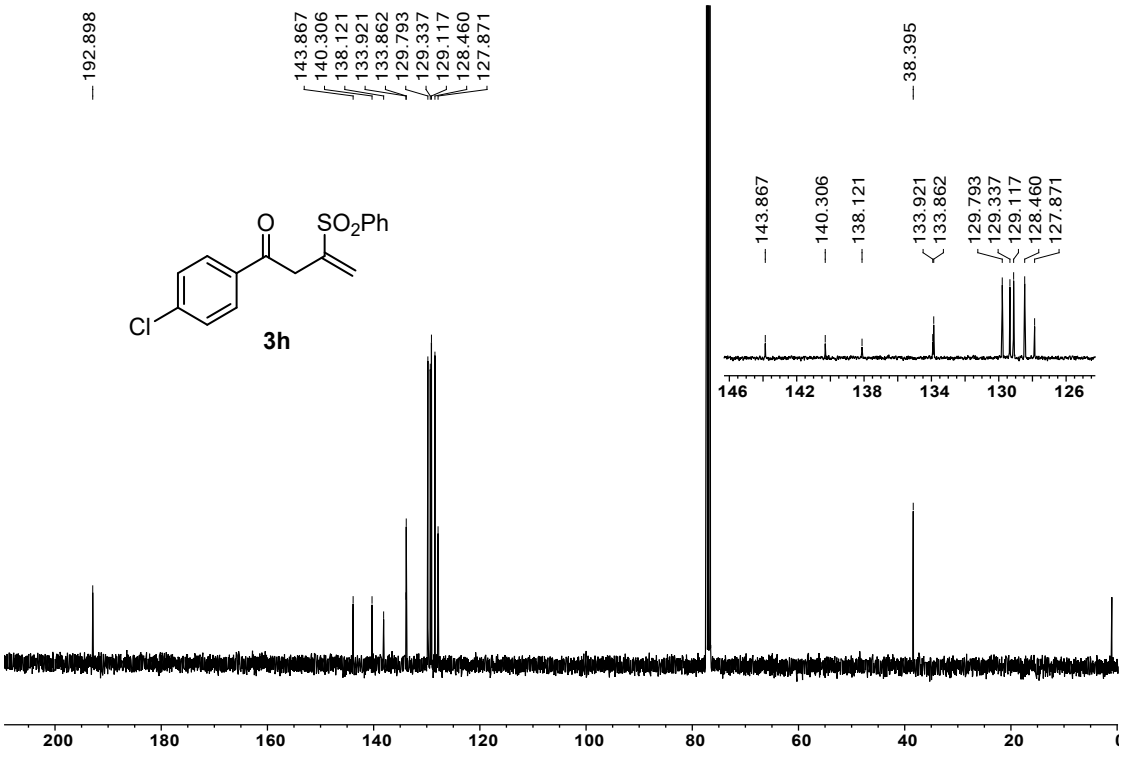
^{13}C NMR of **3g** (101 MHz, $CDCl_3$)



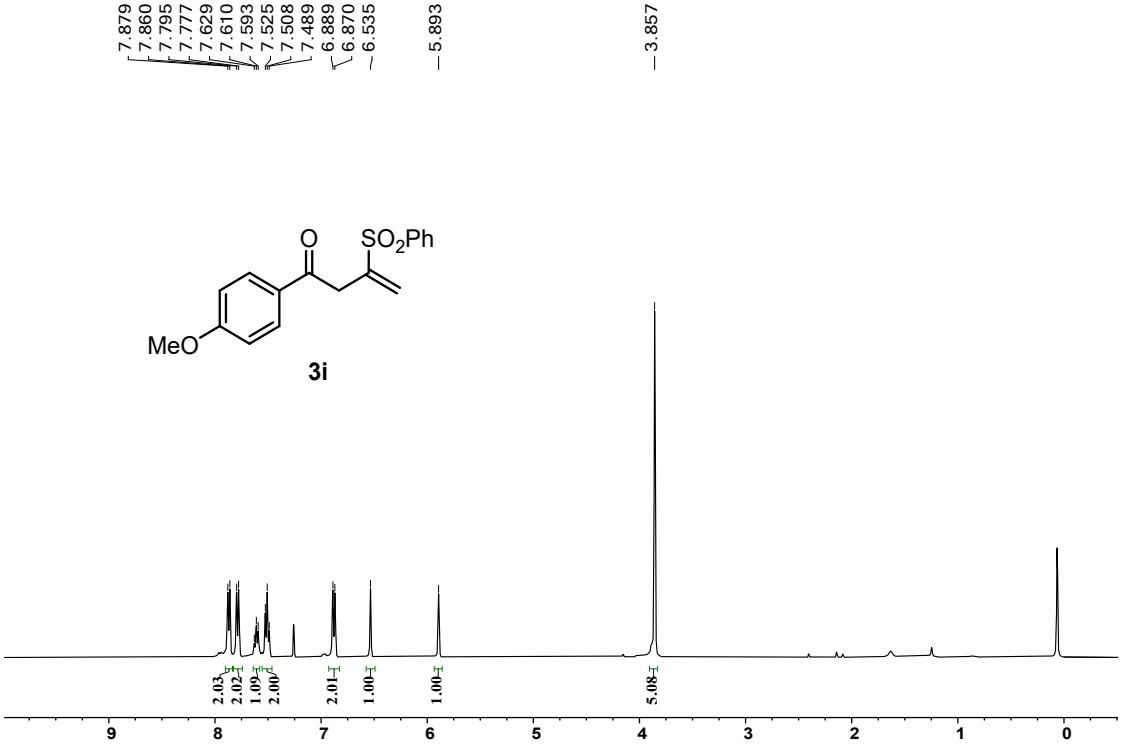
^{1}H NMR of **3h** (400 MHz, $CDCl_3$)



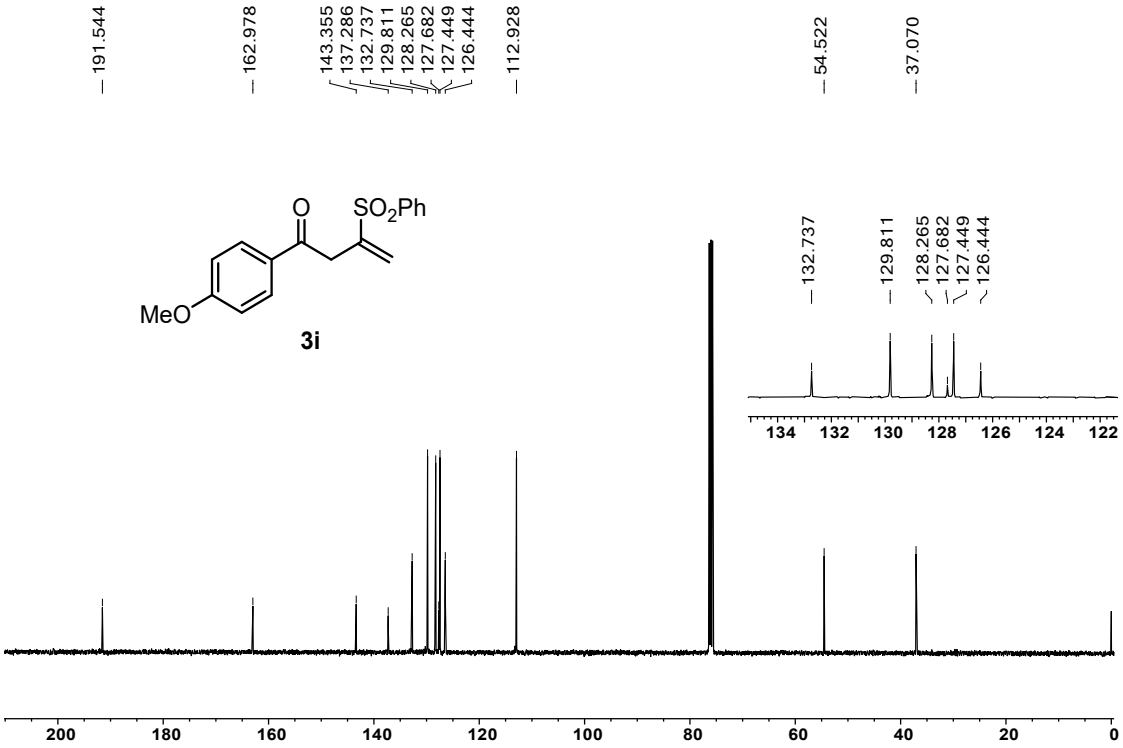
^{13}C NMR of **3h** (101 MHz, $CDCl_3$)



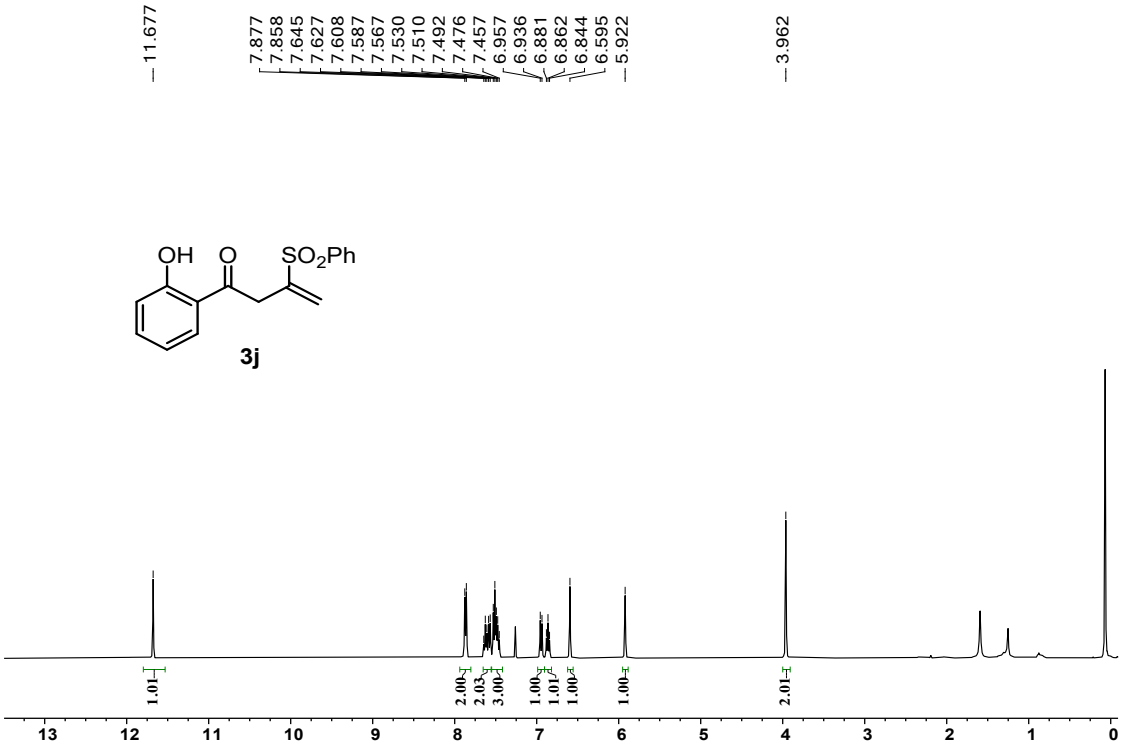
^{1}H NMR of **3i** (400 MHz, $CDCl_3$)



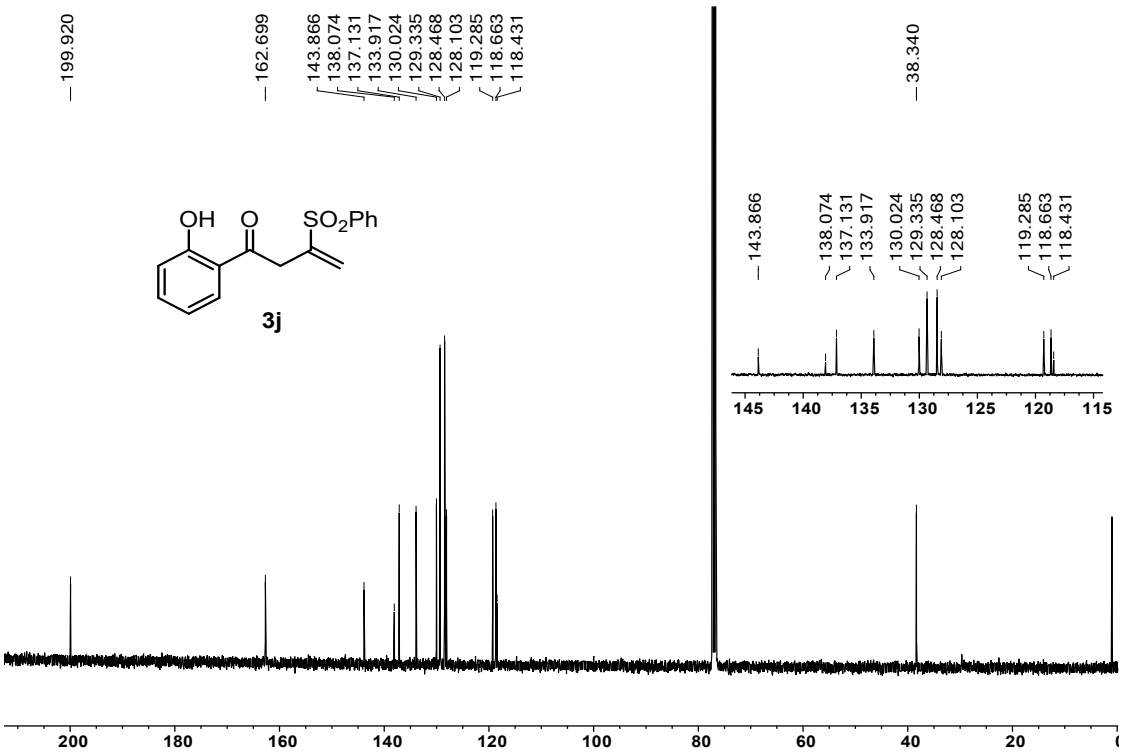
^{13}C NMR of **3i** (101 MHz, $CDCl_3$)



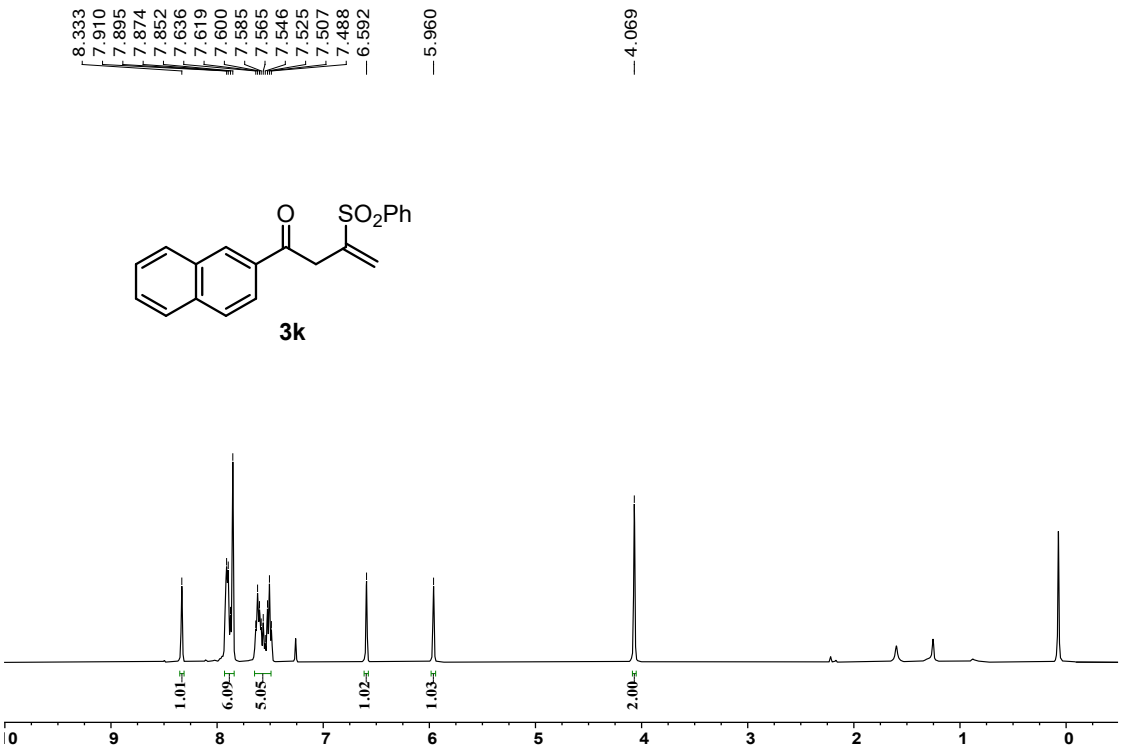
^{1}H NMR of **3j** (400 MHz, $CDCl_3$)



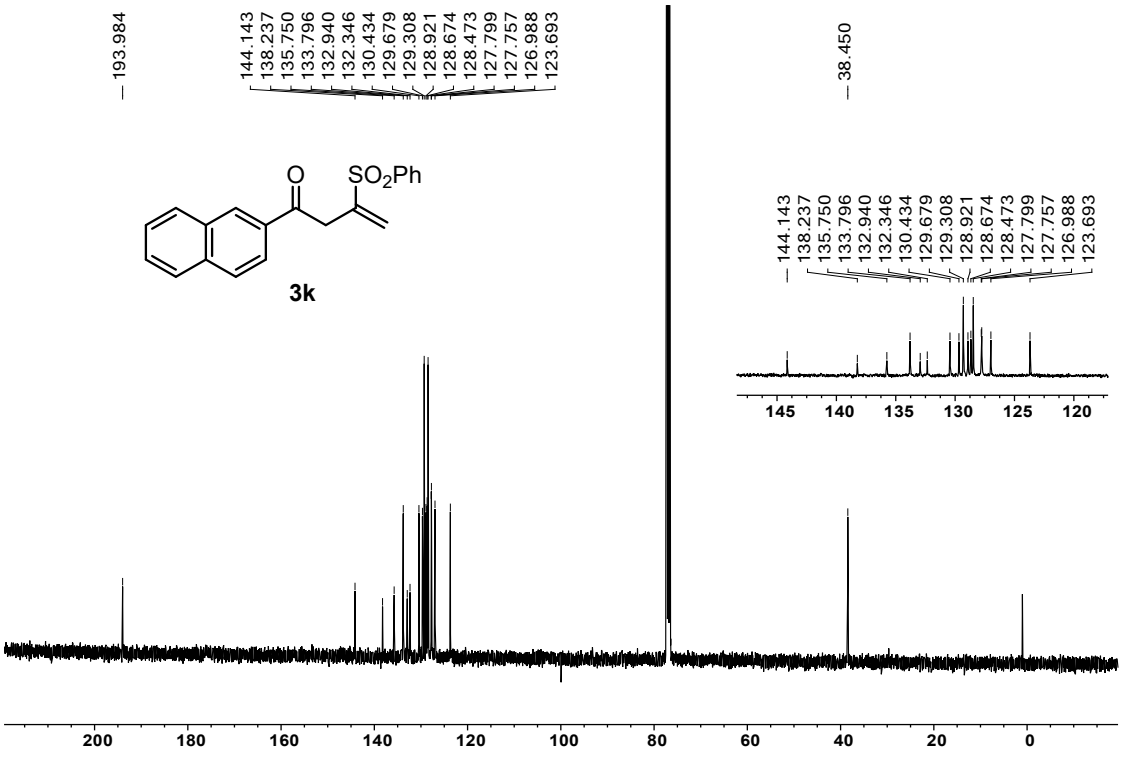
^{13}C NMR of **3j** (101 MHz, $CDCl_3$)



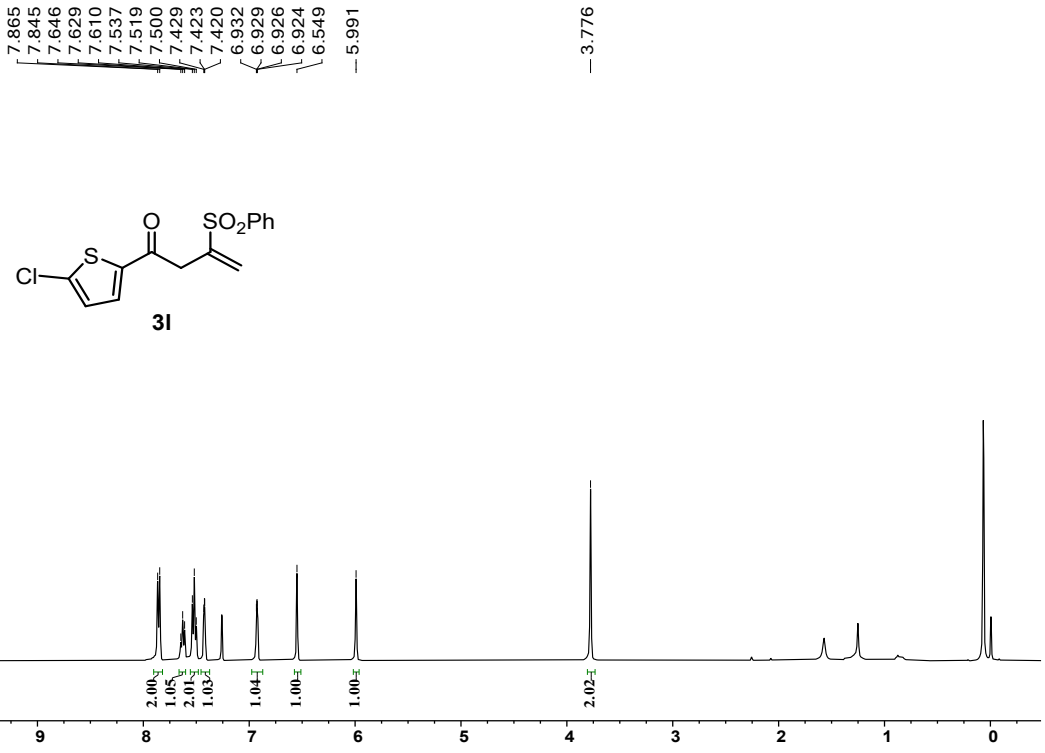
^{1}H NMR of **3k** (400 MHz, $CDCl_3$)



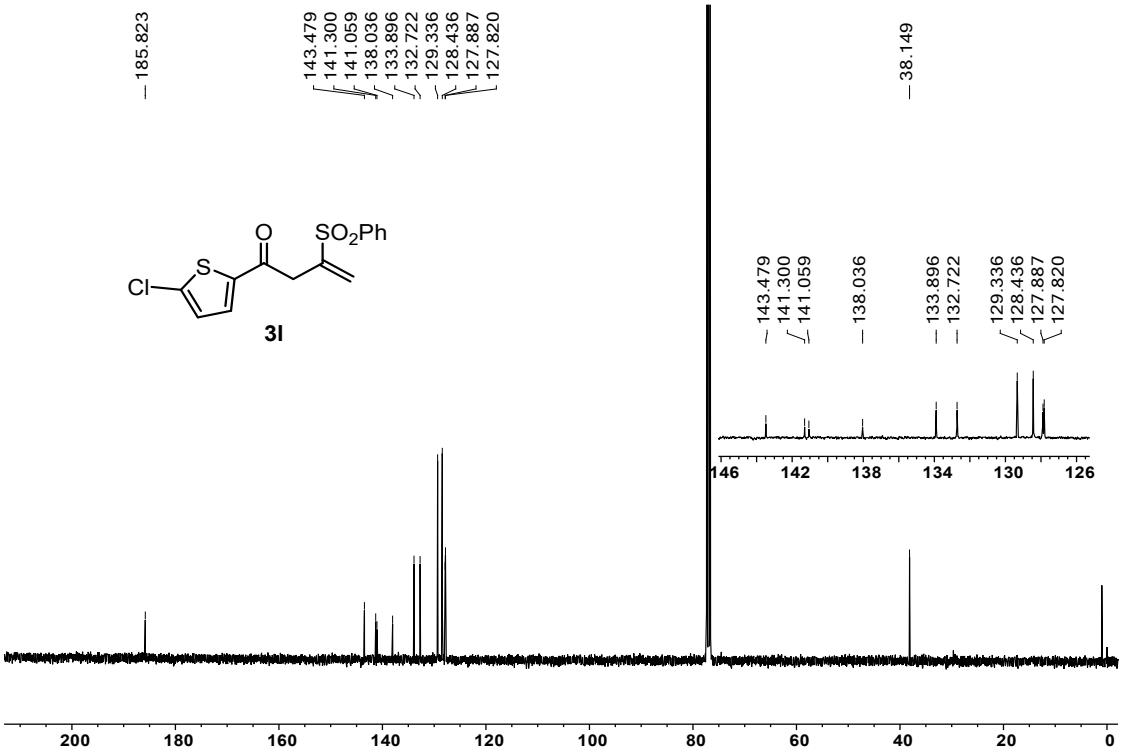
^{13}C NMR of **3k** (101 MHz, $CDCl_3$)



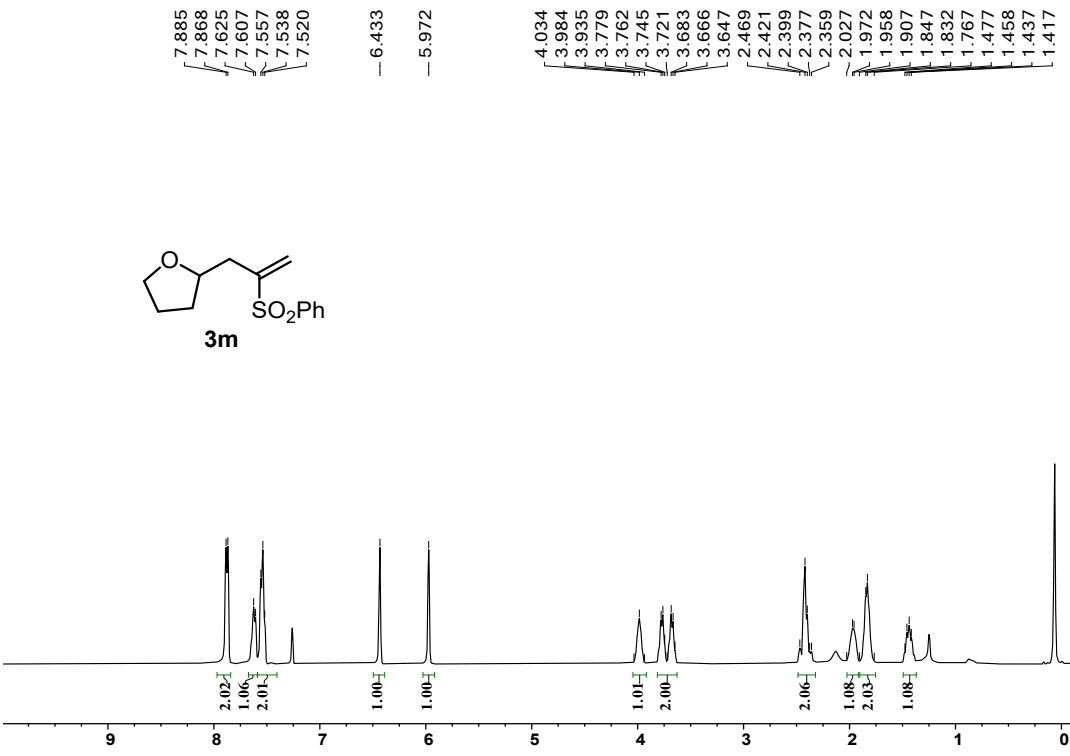
^{1}H NMR of **3l** (400 MHz, $CDCl_3$)



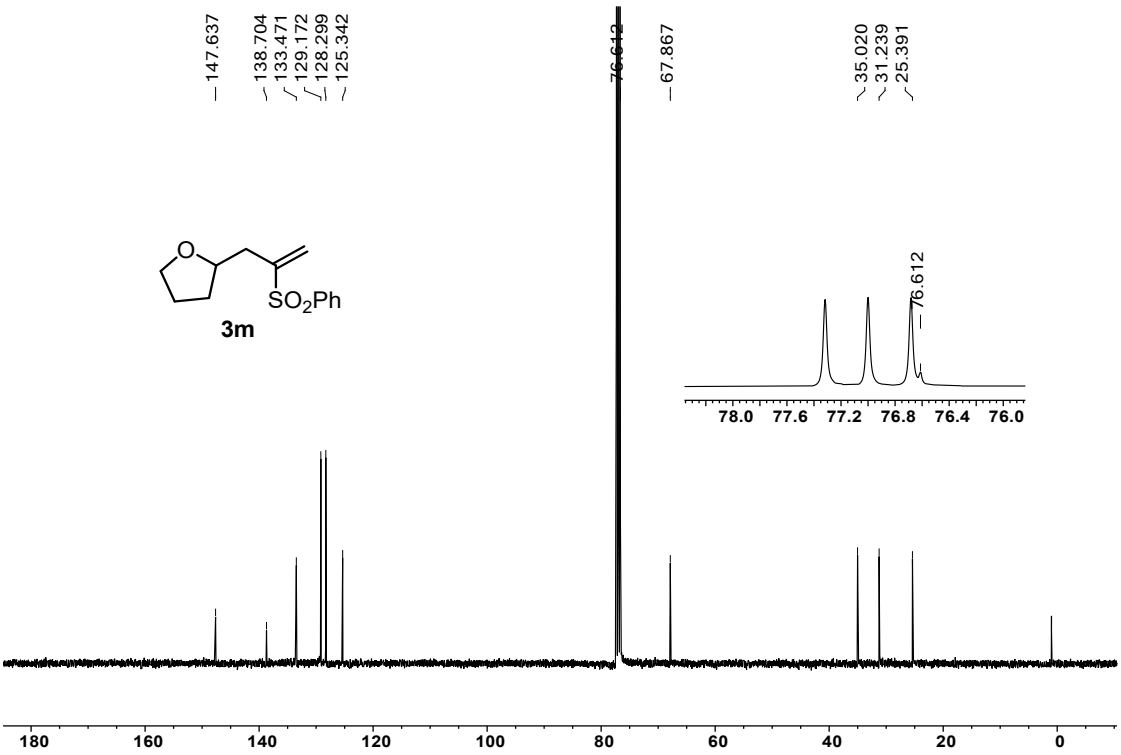
^{13}C NMR of **3l** (101 MHz, $CDCl_3$)



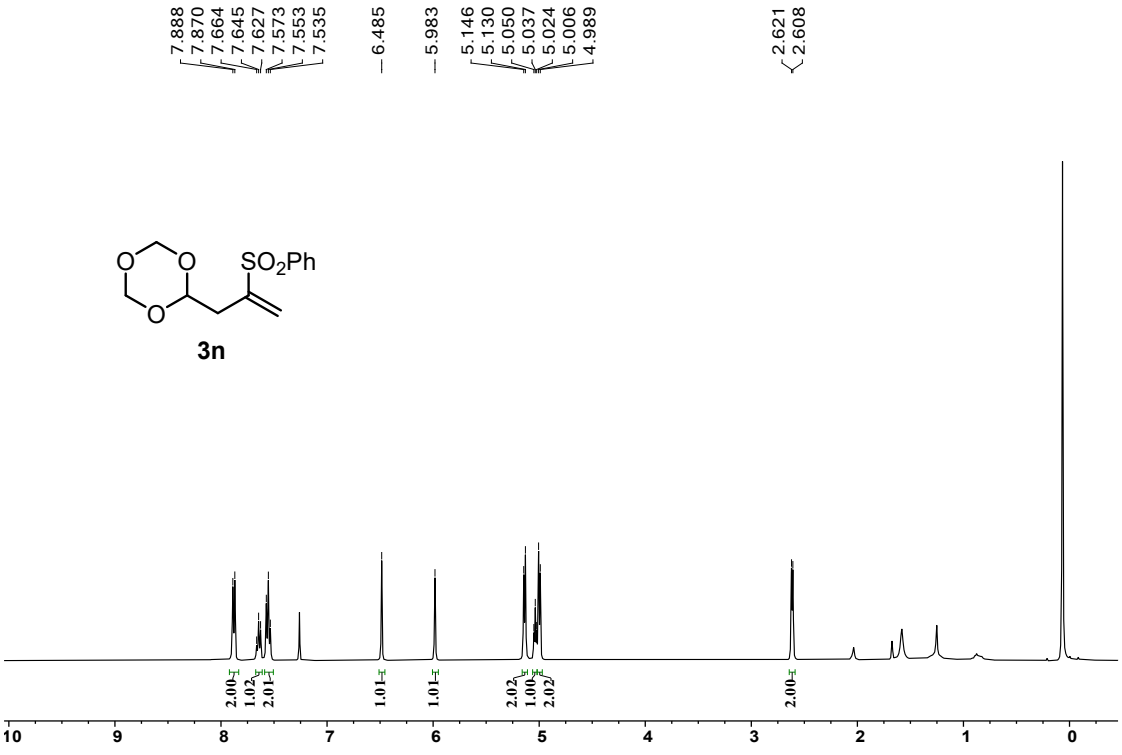
^{1}H NMR of **3m** (400 MHz, $CDCl_3$)



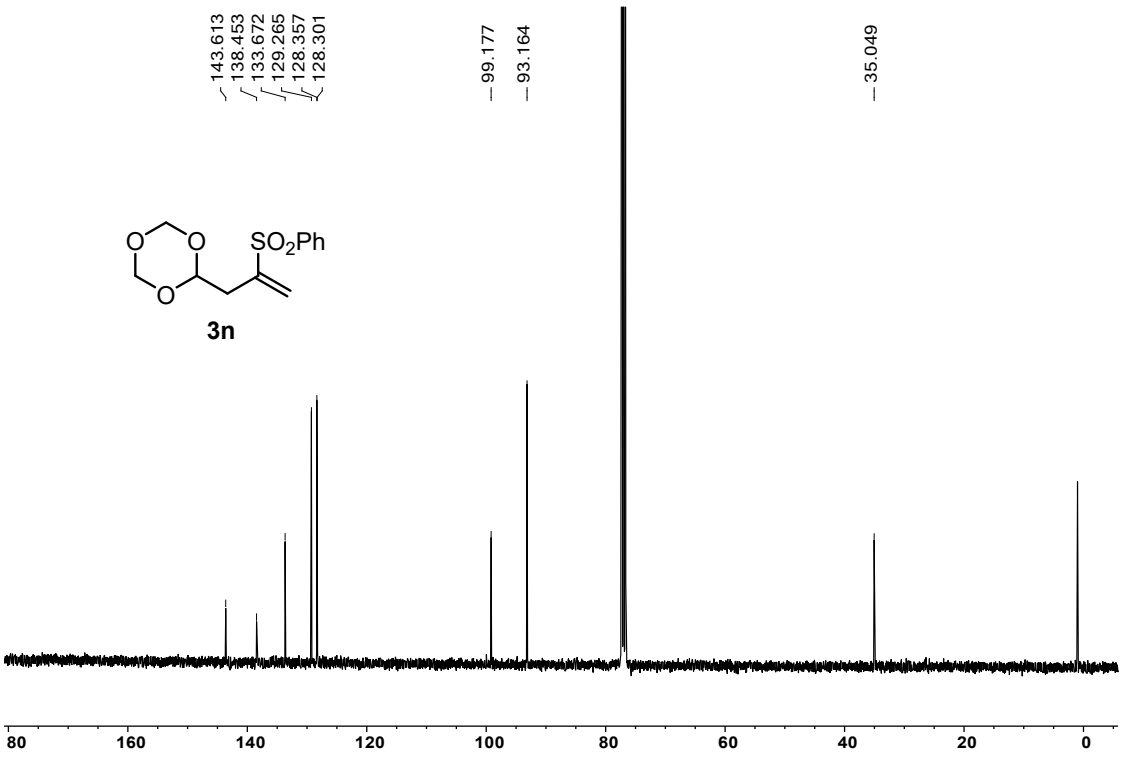
^{13}C NMR of **3m** (101 MHz, $CDCl_3$)



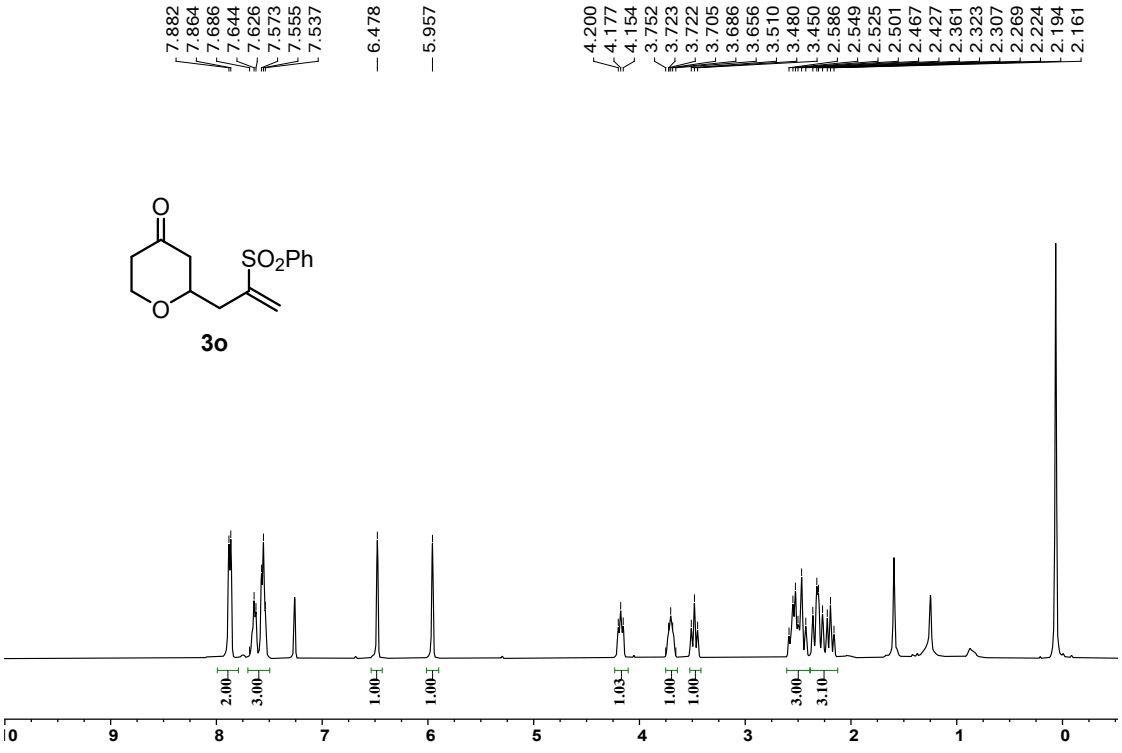
^{1}H NMR of **3n** (400 MHz, $CDCl_3$)



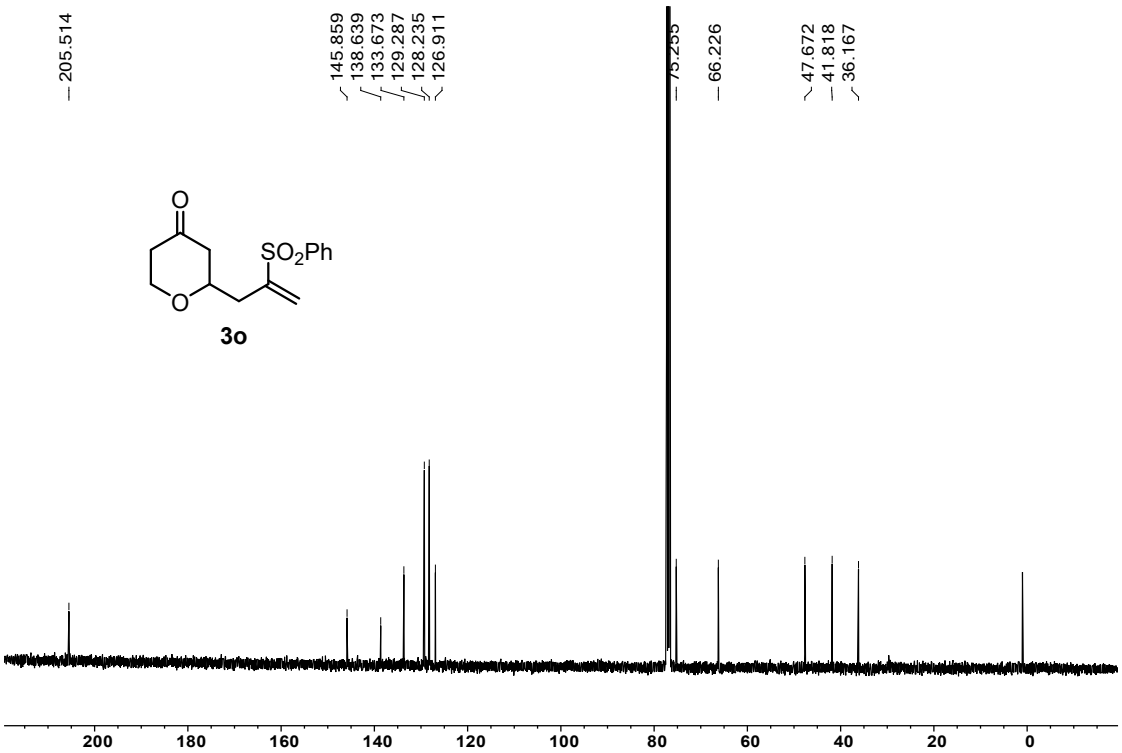
^{13}C NMR of **3n** (101 MHz, $CDCl_3$)



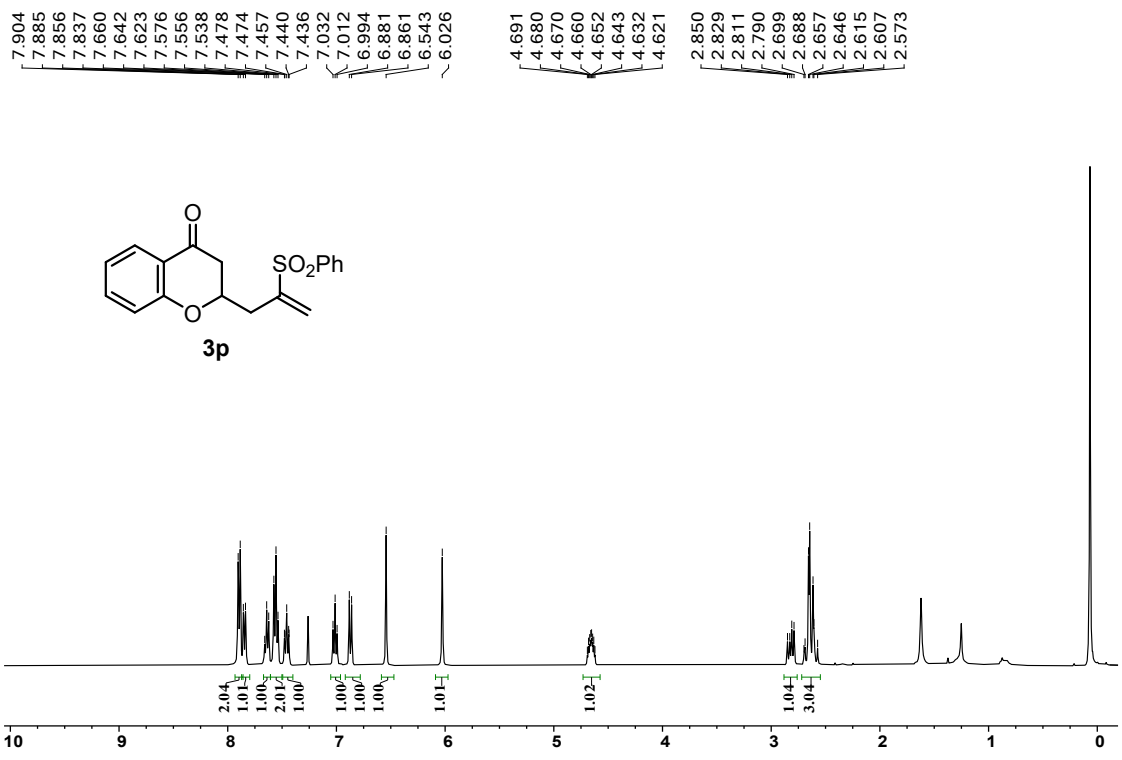
^{1}H NMR of **3o** (400 MHz, $CDCl_3$)



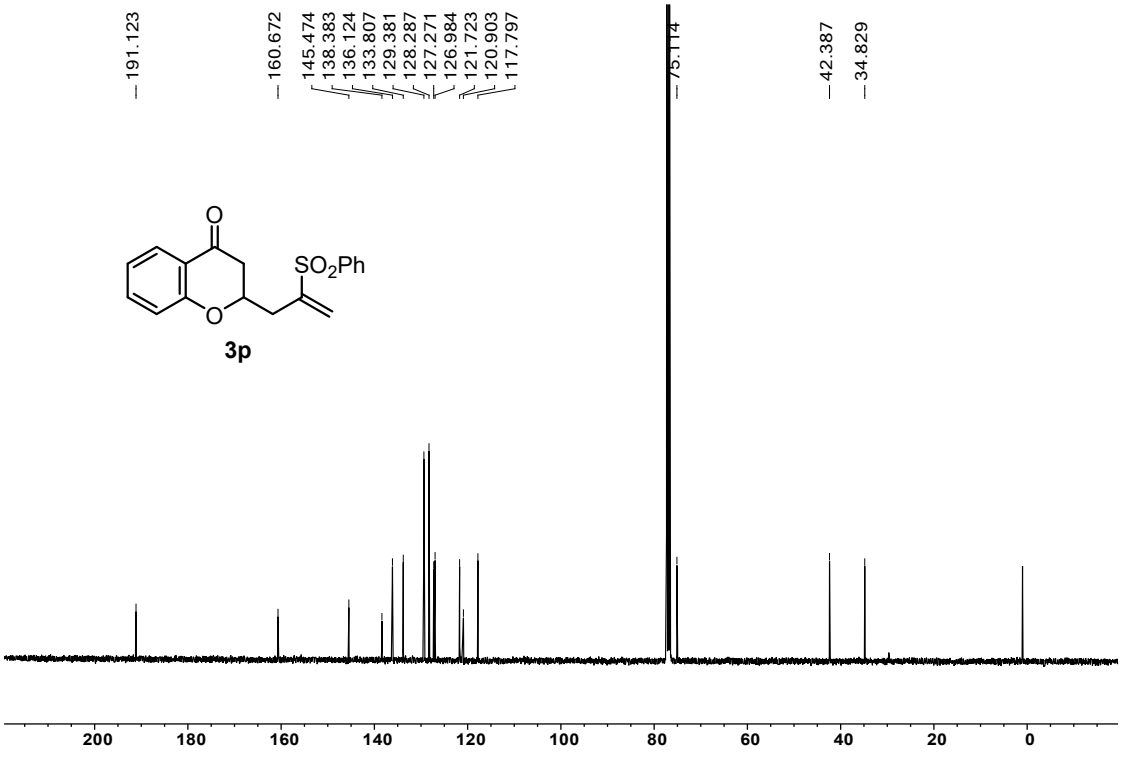
^{13}C NMR of **3o** (101 MHz, $CDCl_3$)



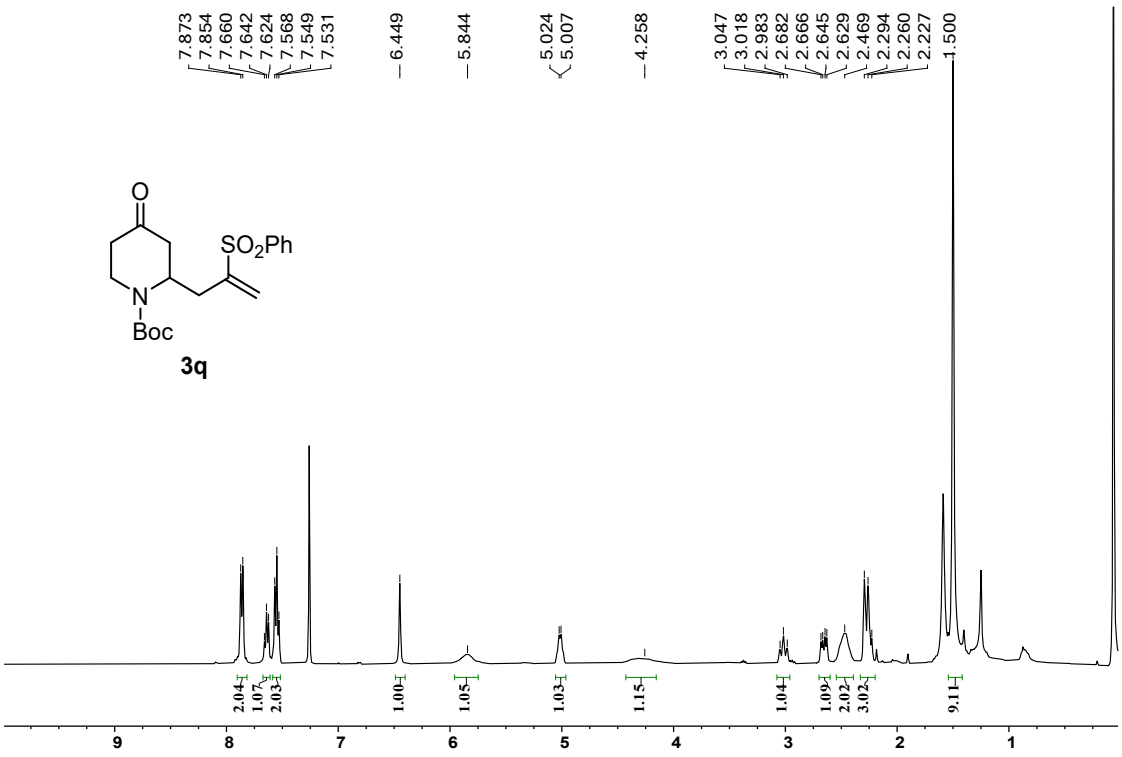
^{1}H NMR of **3p** (400 MHz, $CDCl_3$)



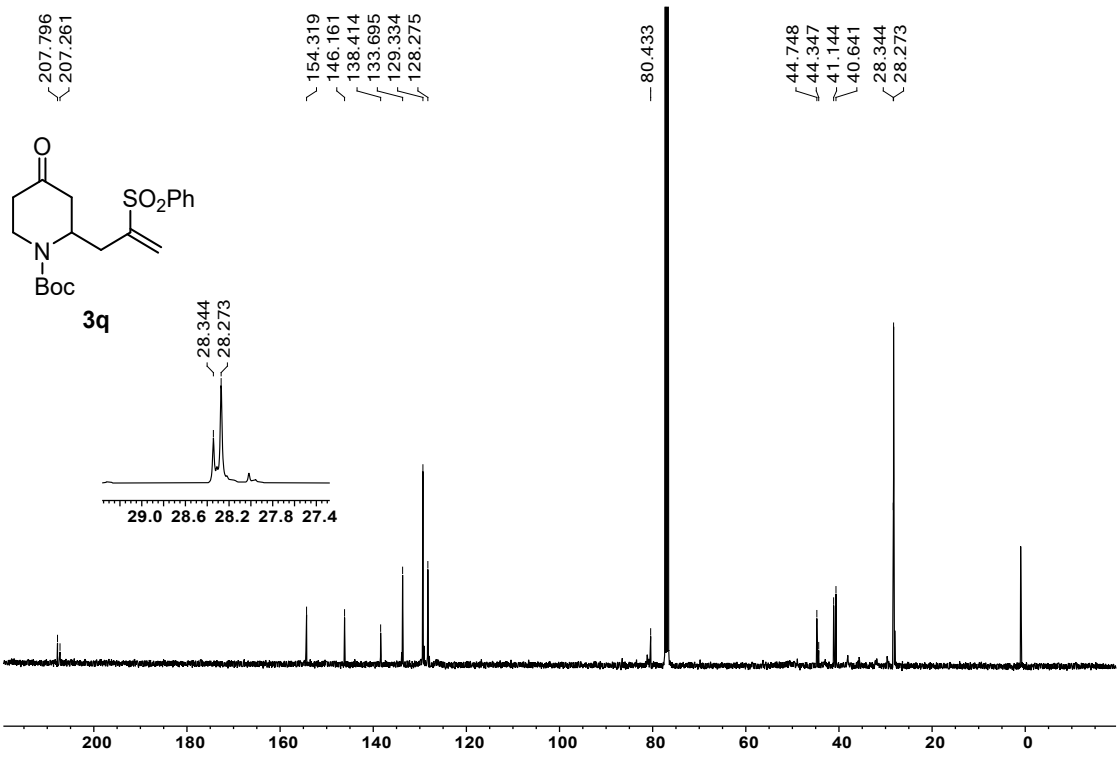
^{13}C NMR of **3p** (101 MHz, $CDCl_3$)



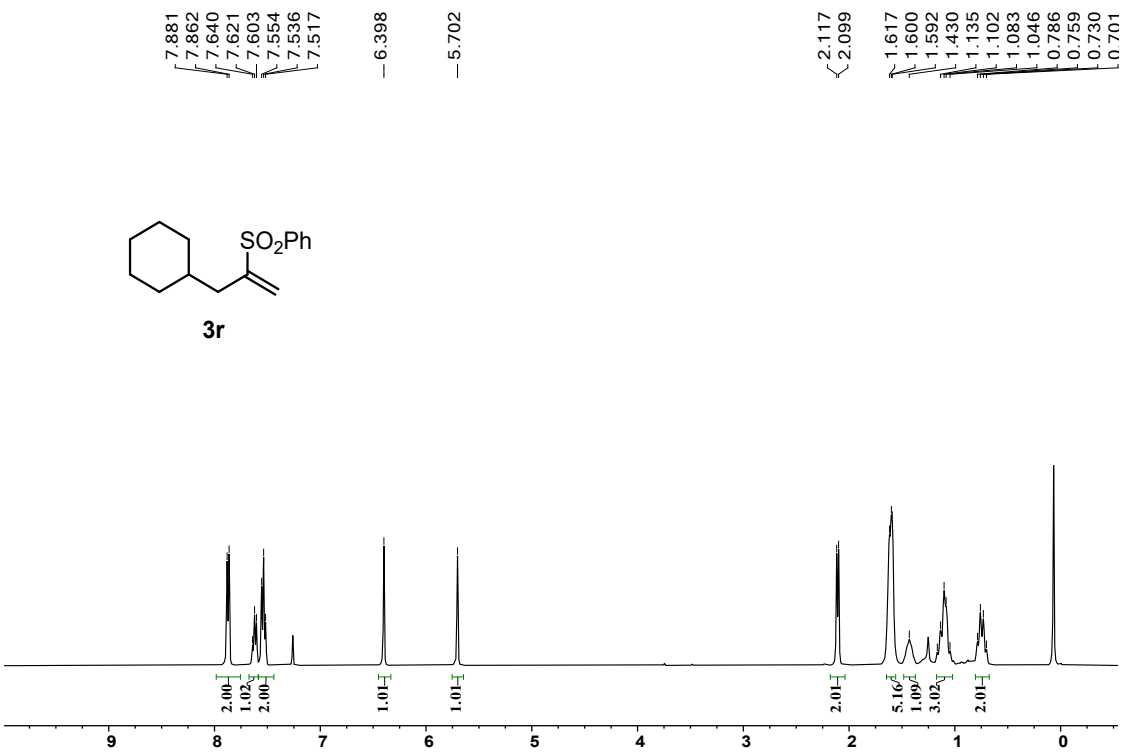
^{1}H NMR of **3q** (400 MHz, $CDCl_3$)



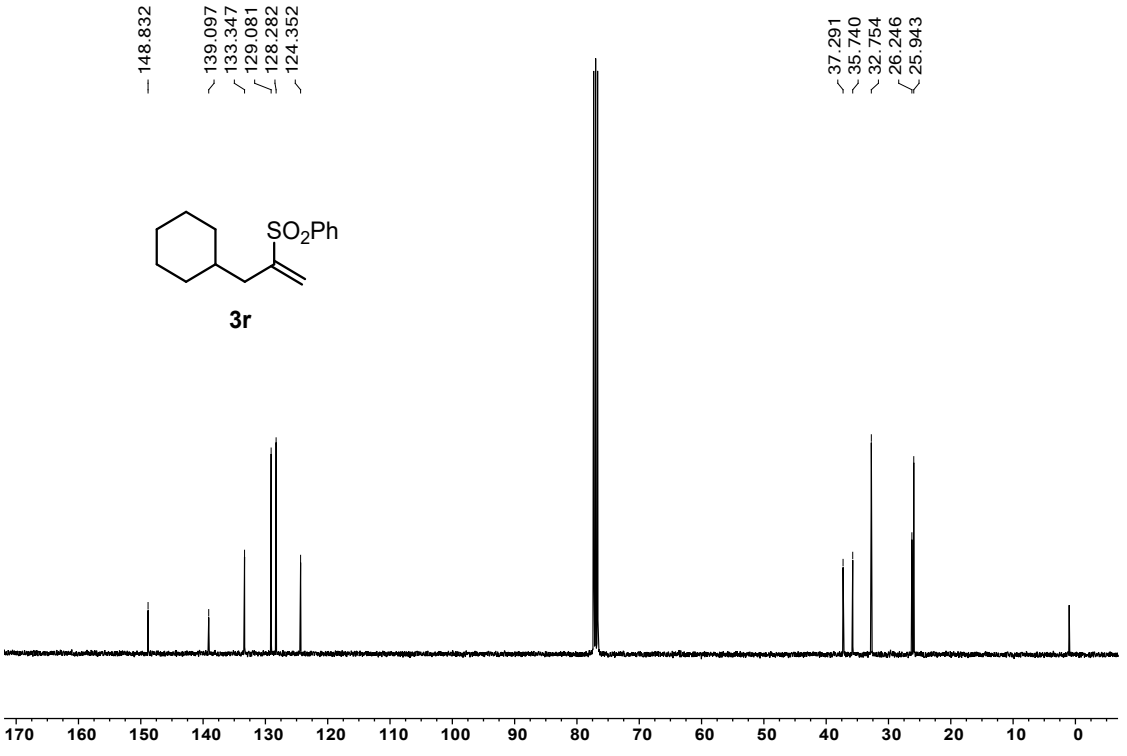
^{13}C NMR of **3q** (101 MHz, $CDCl_3$)



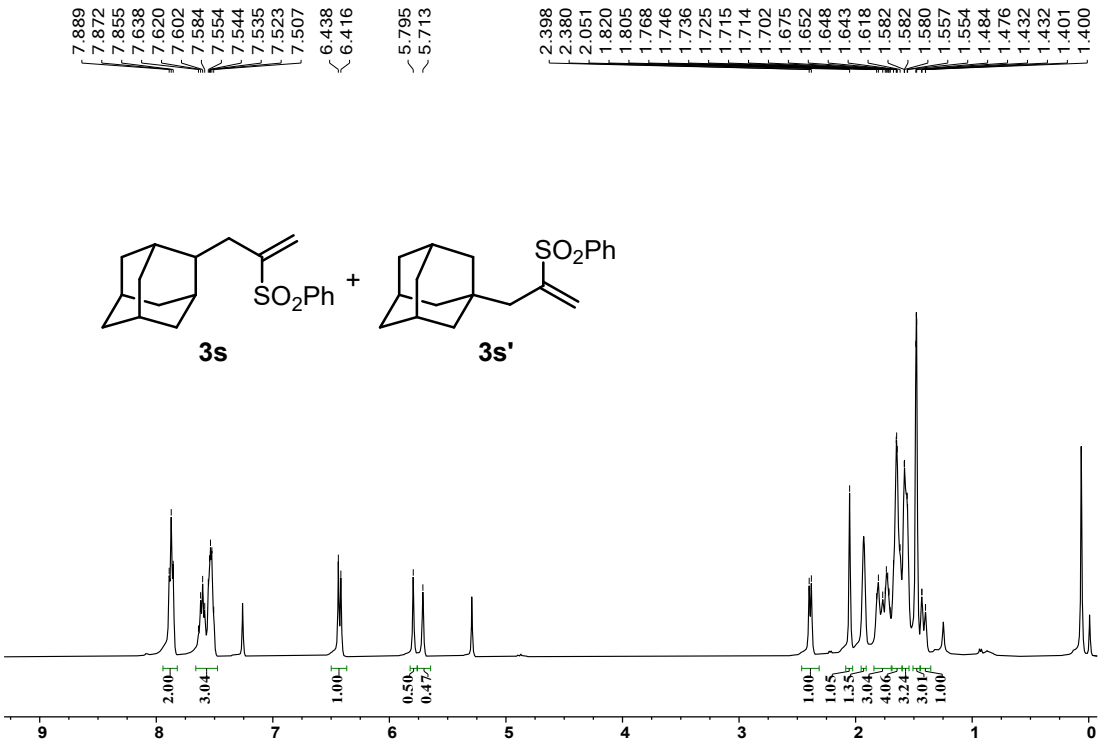
^{1}H NMR of **3r** (400 MHz, $CDCl_3$)



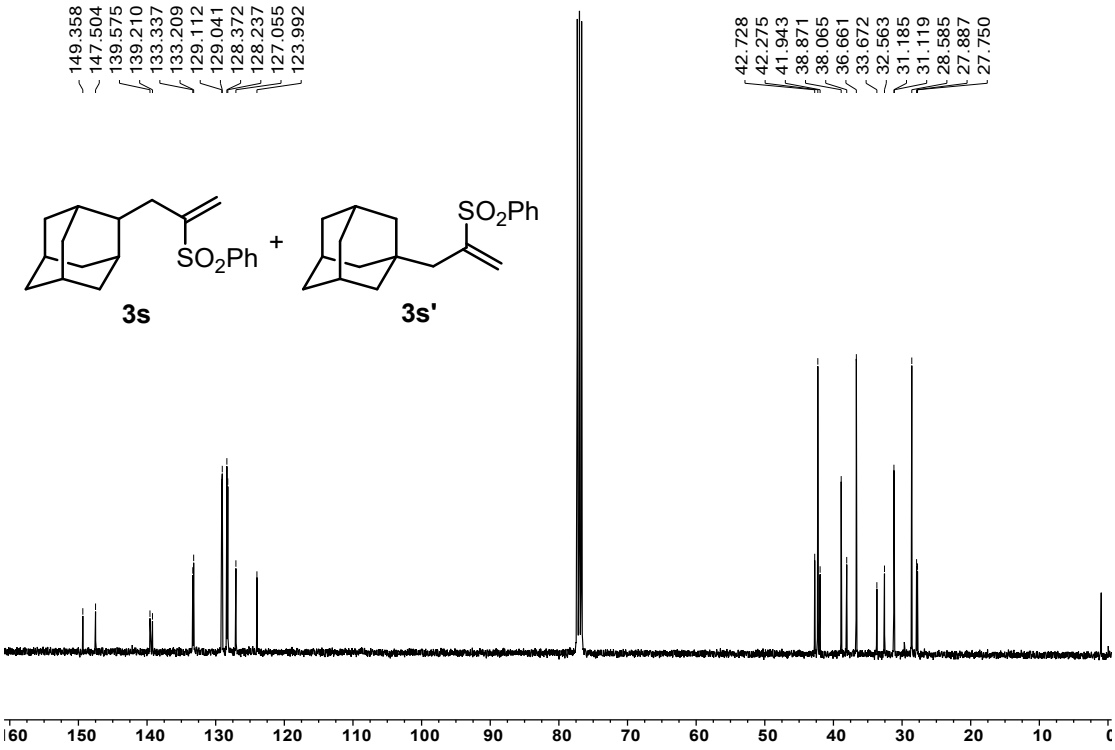
^{13}C NMR of **3r** (101 MHz, $CDCl_3$)



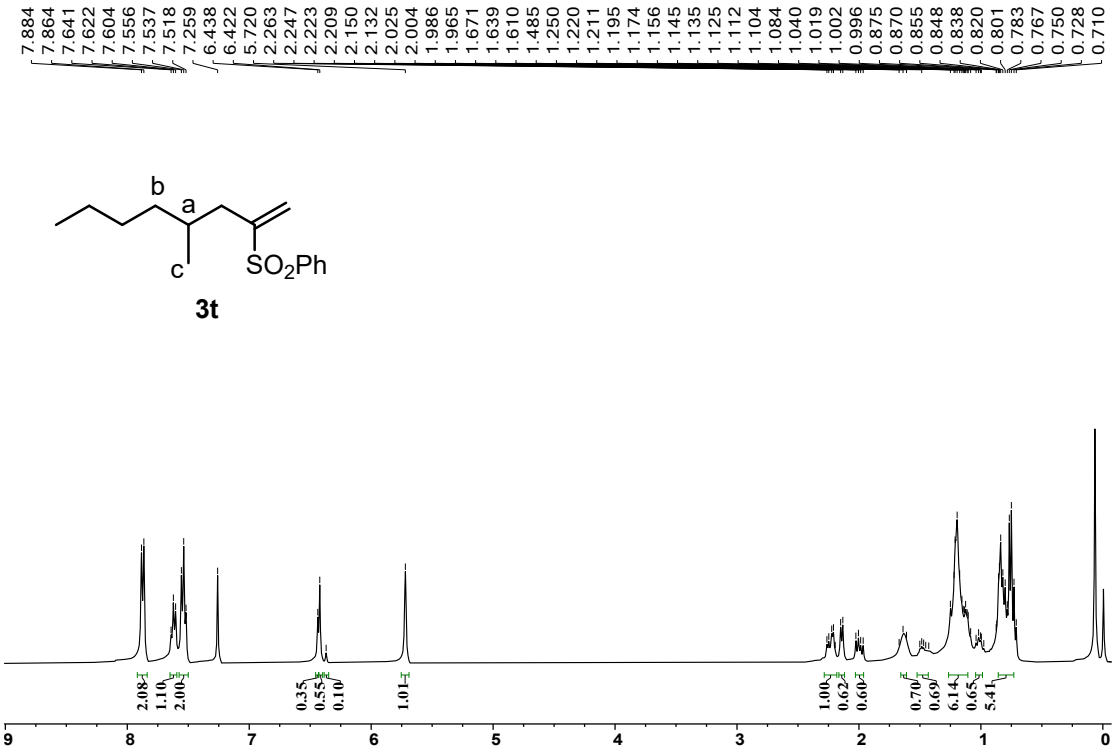
^{1}H NMR of **3s** and **3s'** (400 MHz, $CDCl_3$)



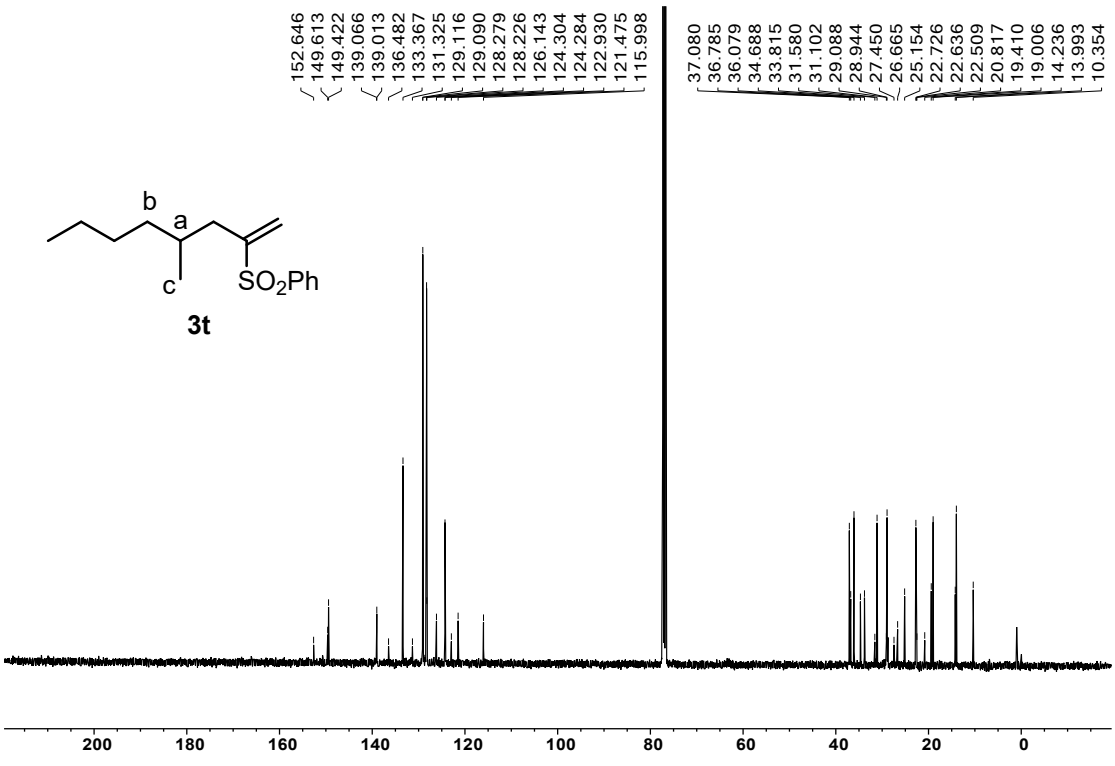
^{13}C NMR of **3s** and **3s'** (101 MHz, $CDCl_3$)



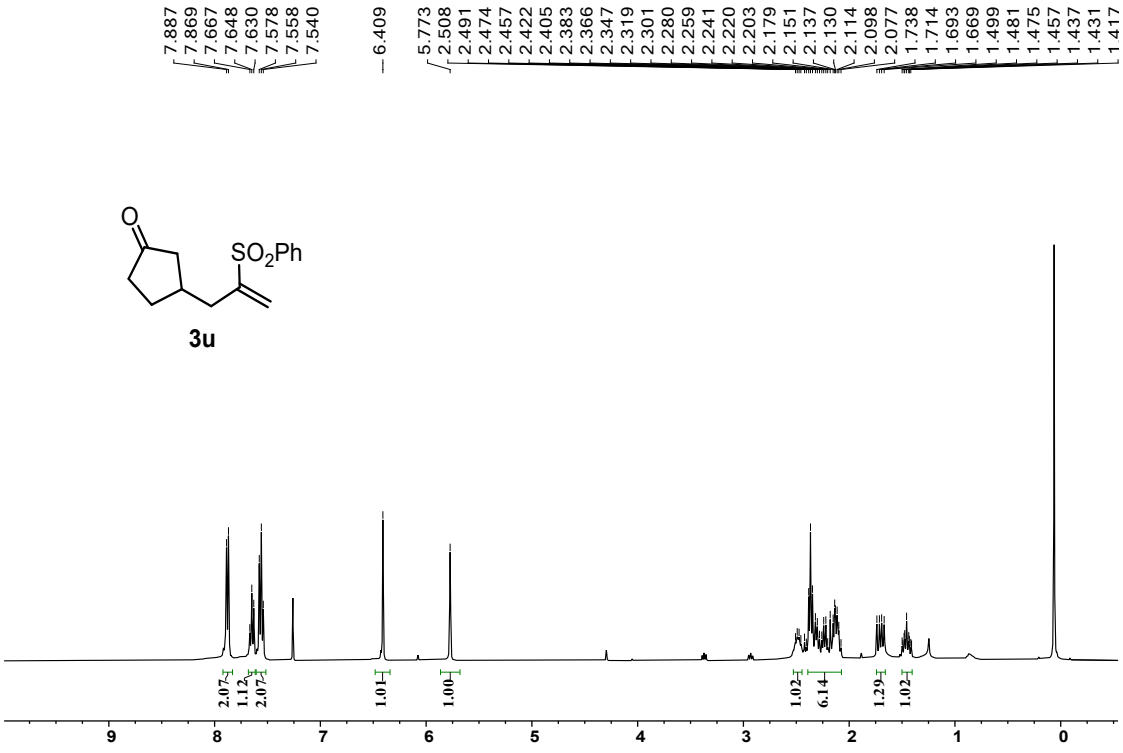
^{1}H NMR of **3t** (400 MHz, $CDCl_3$)



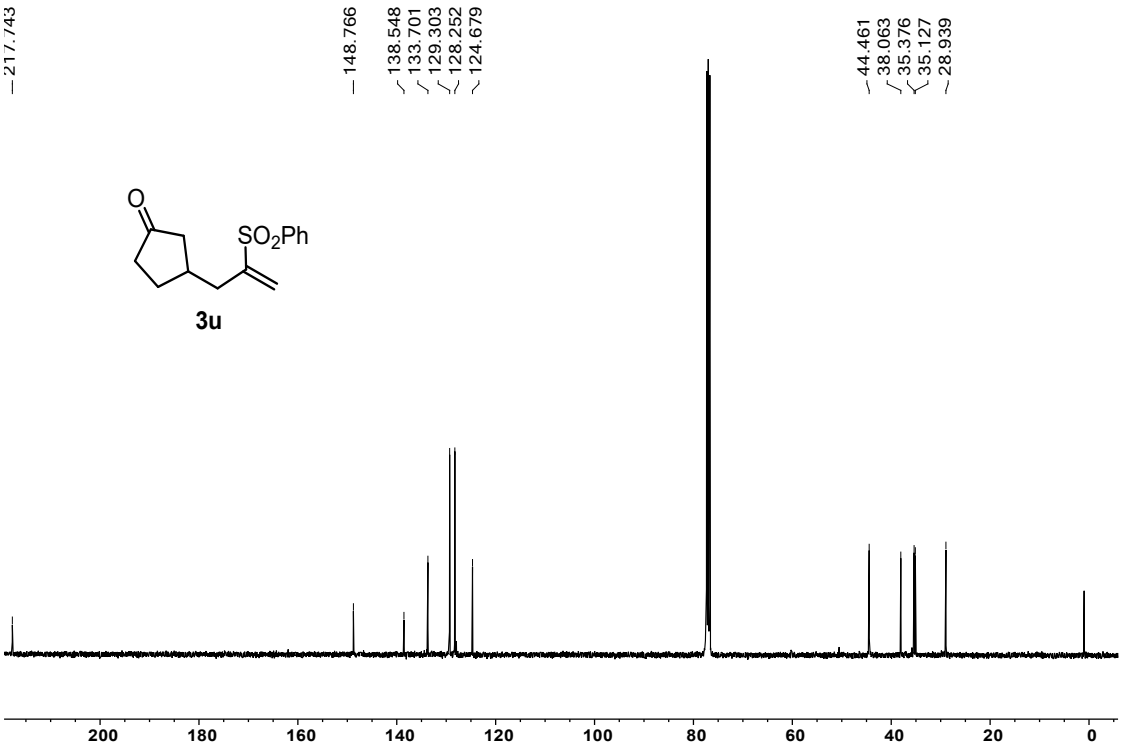
^{13}C NMR of **3t** (101 MHz, $CDCl_3$)



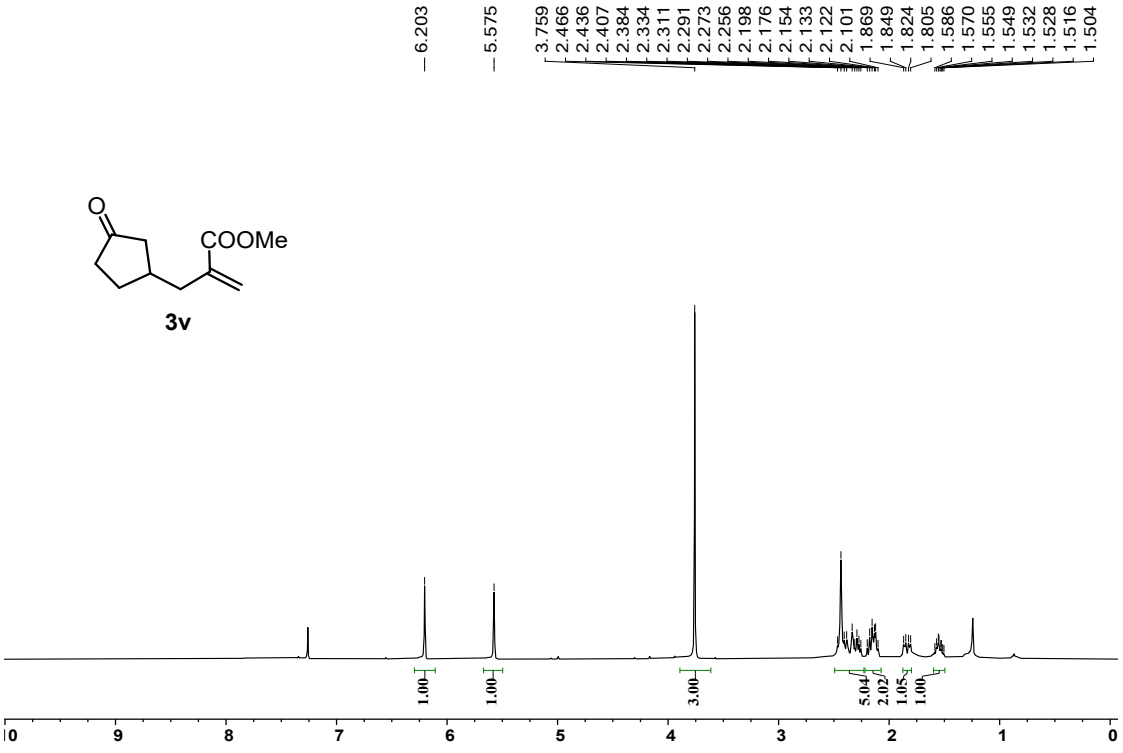
^{1}H NMR of **3u** (400 MHz, $CDCl_3$)



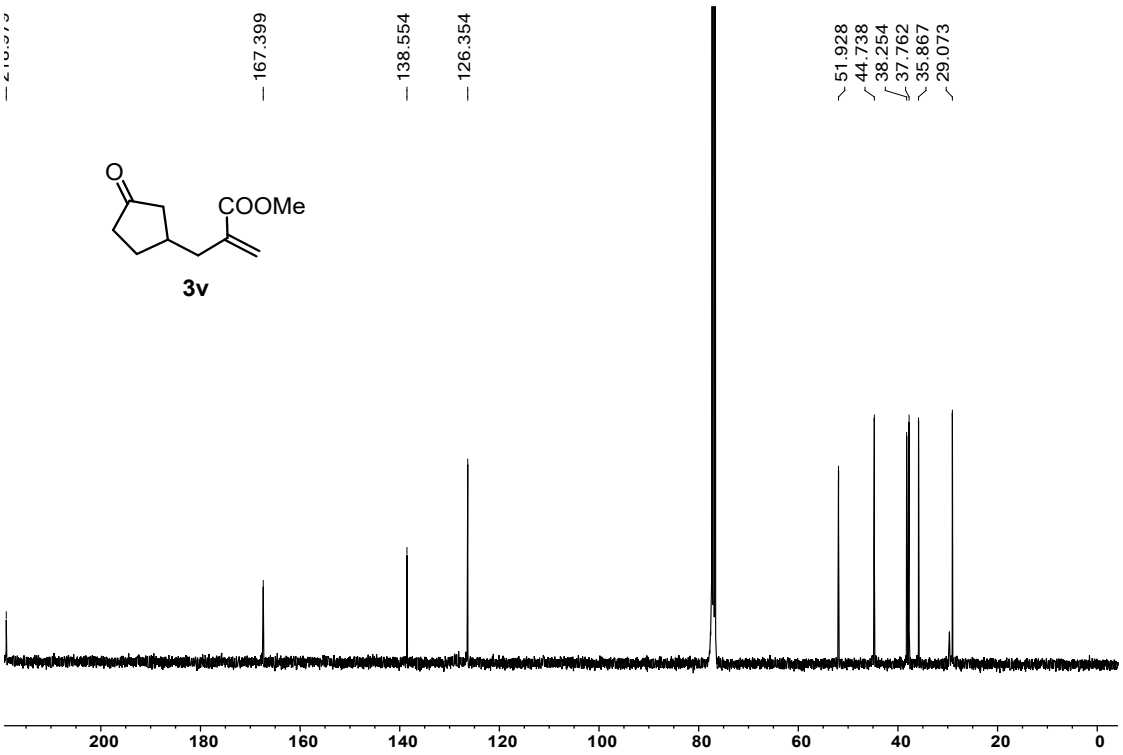
^{13}C NMR of **3u** (101 MHz, $CDCl_3$)



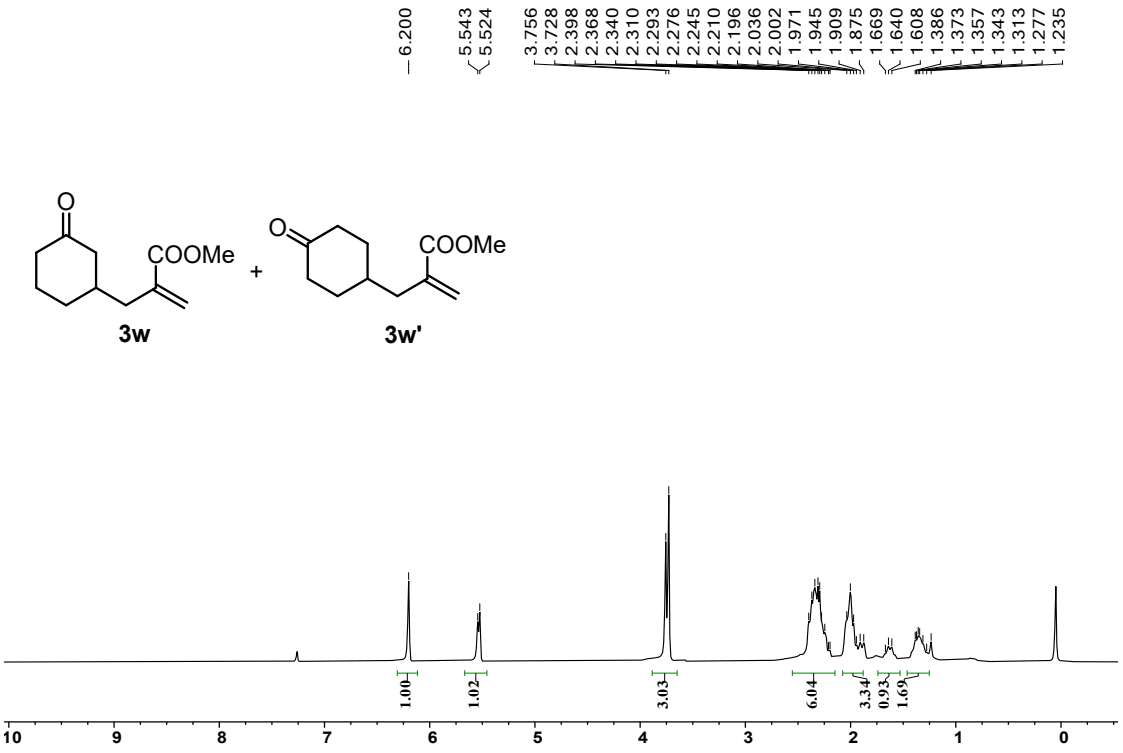
^{1}H NMR of **3v** (400 MHz, $CDCl_3$)



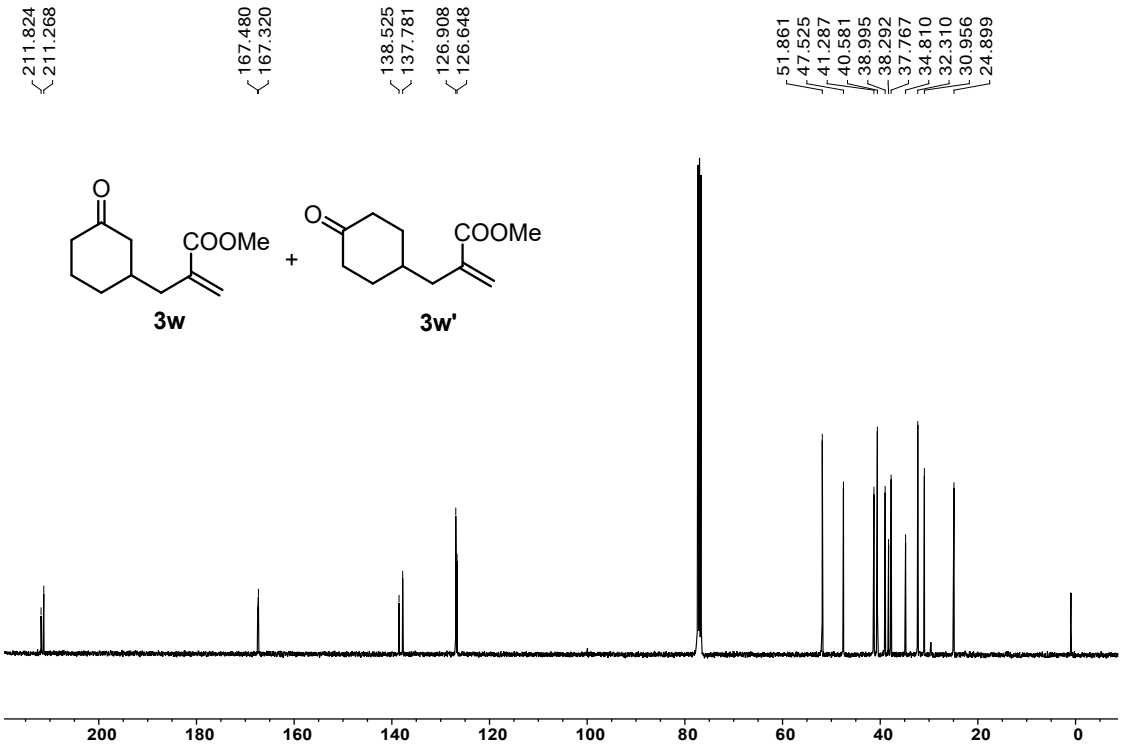
^{13}C NMR of **3v** (101 MHz, $CDCl_3$)



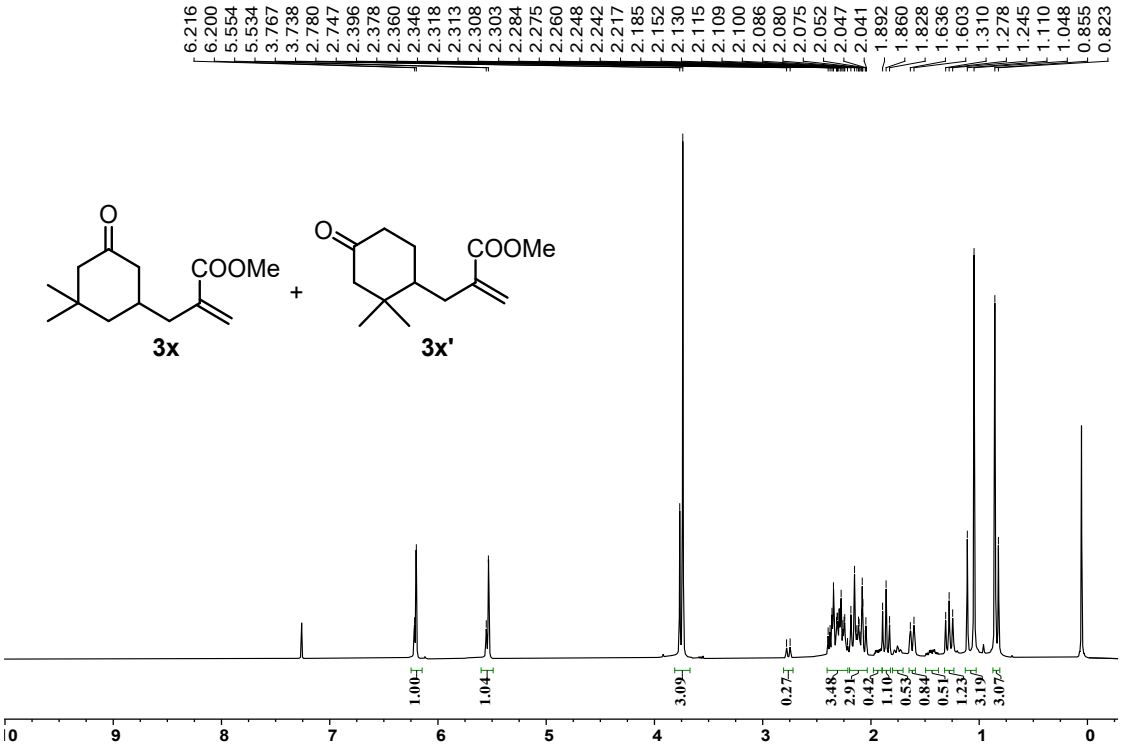
^{1}H NMR of **3w** and **3w'** (400 MHz, $CDCl_3$)



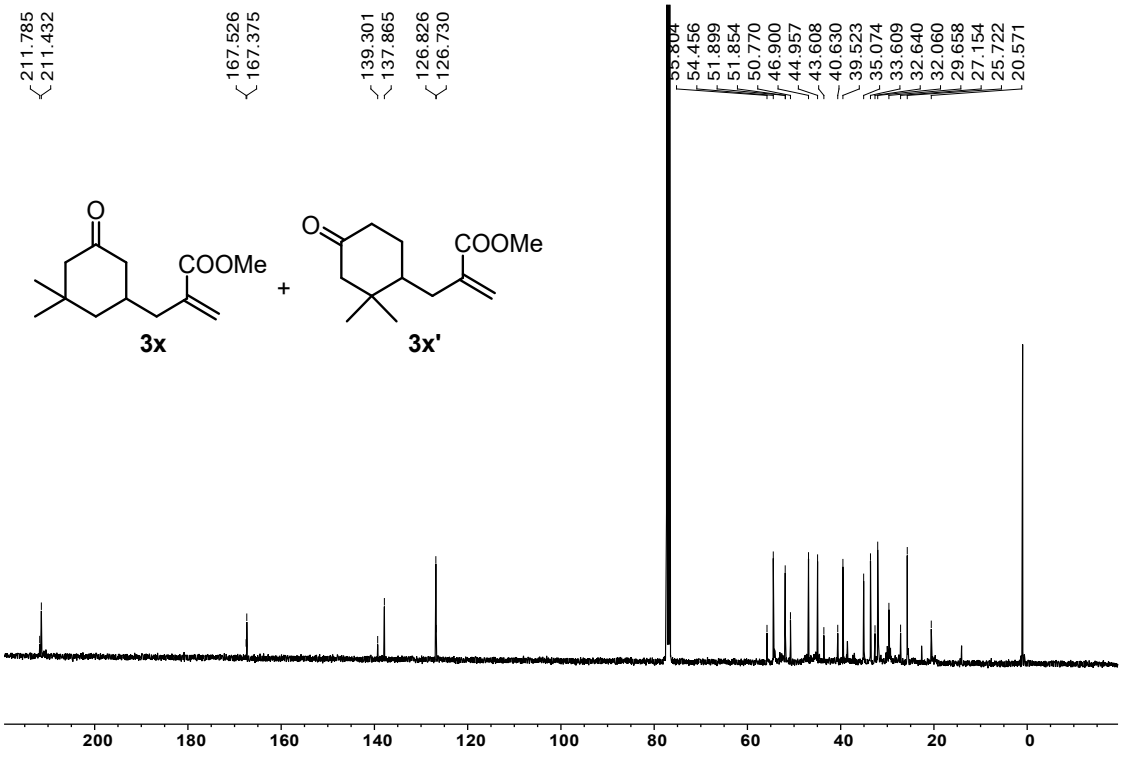
^{13}C NMR of **3w** and **3w'** (101 MHz, $CDCl_3$)



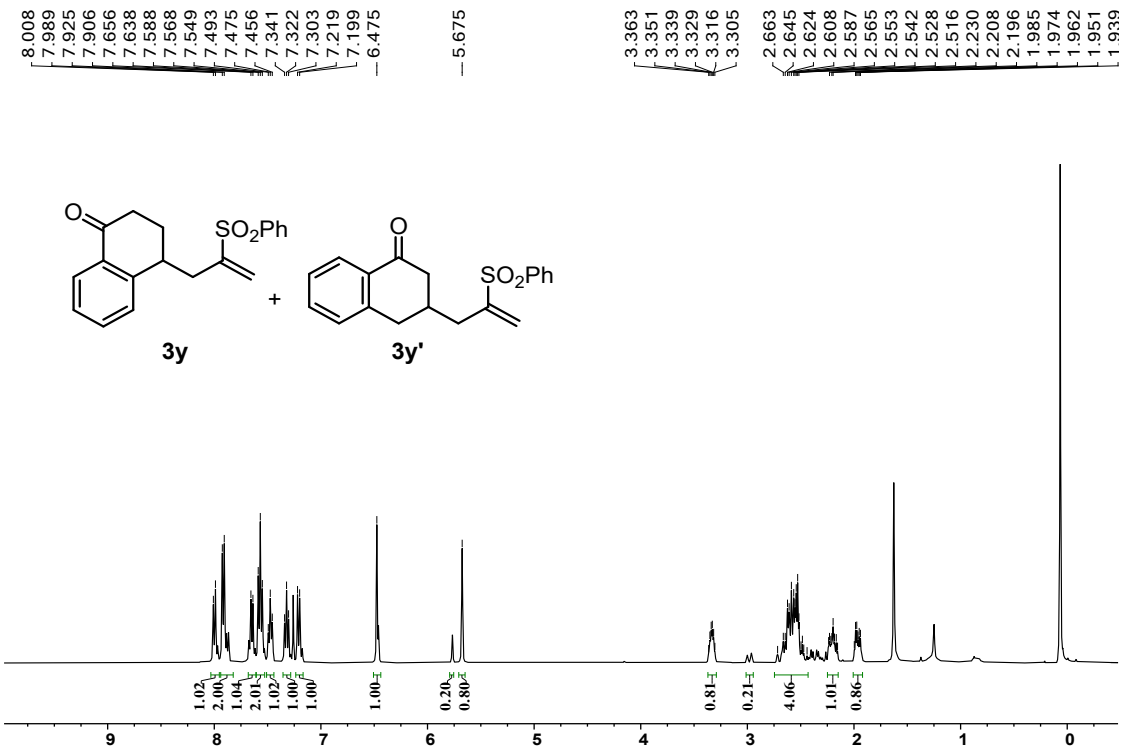
^{1}H NMR of **3x** and **3x'** (400 MHz, $CDCl_3$)



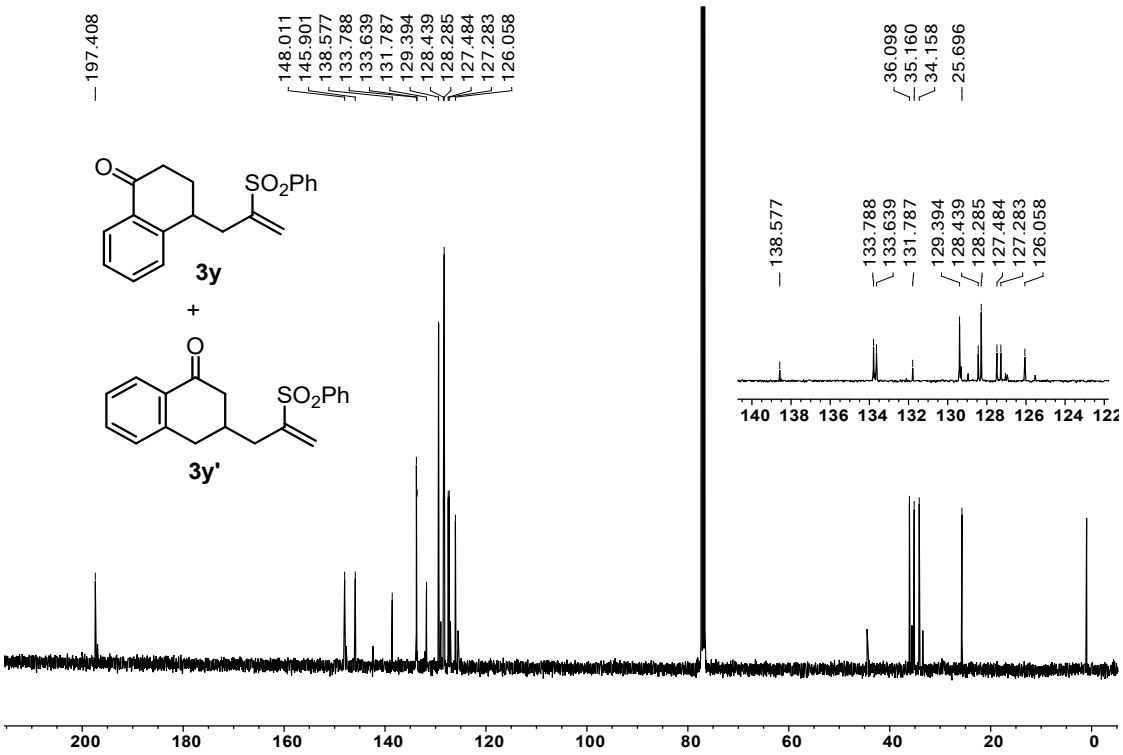
^{13}C NMR of **3x** and **3x'** (101 MHz, $CDCl_3$)



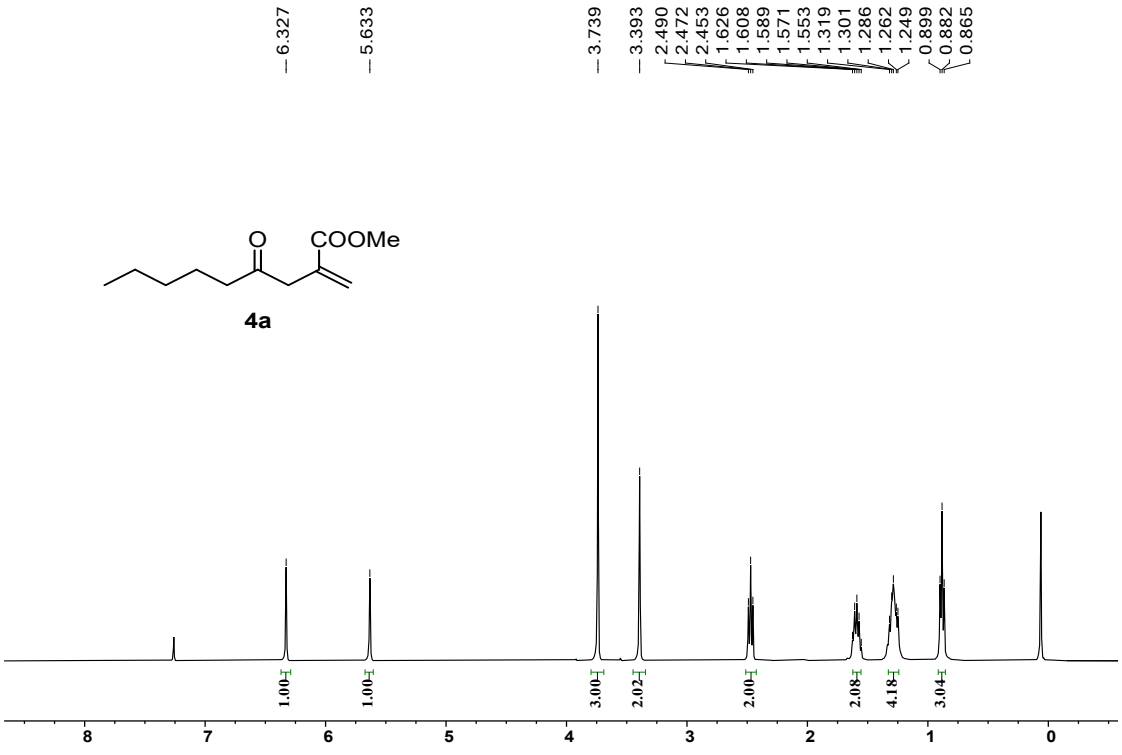
^{1}H NMR of **3y** and **3y'** (400 MHz, $CDCl_3$)



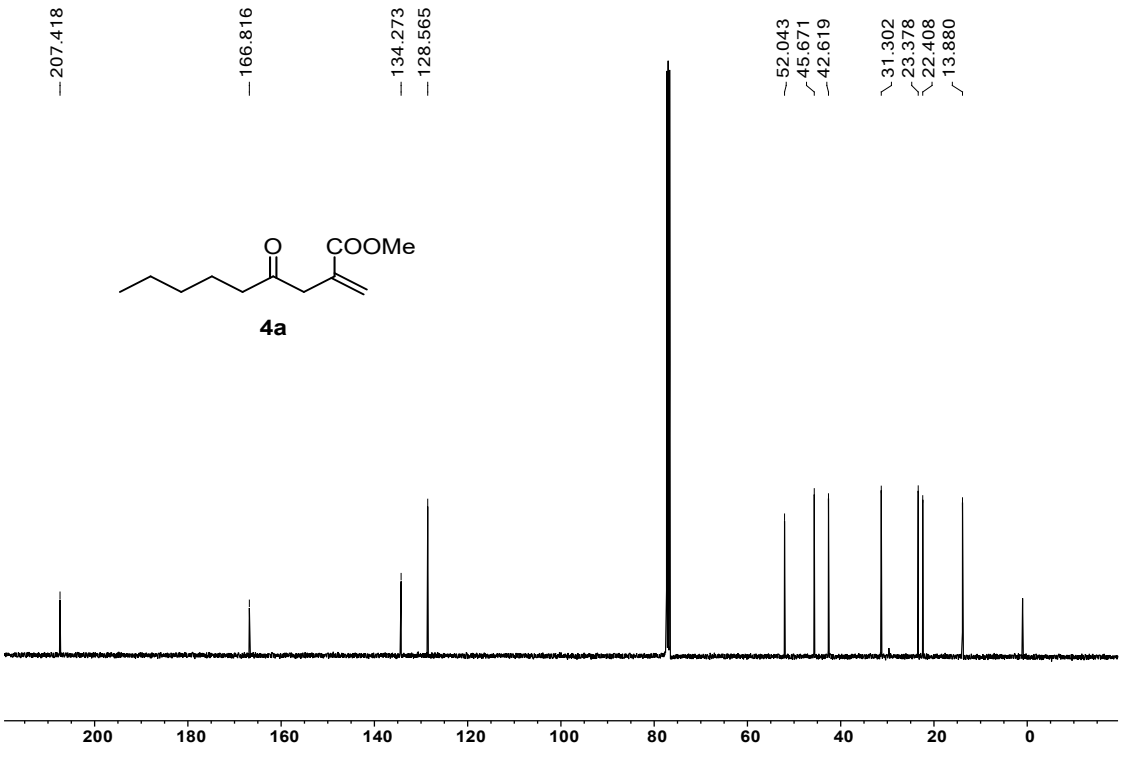
^{13}C NMR of **3y** and **3y'** (101 MHz, $CDCl_3$)



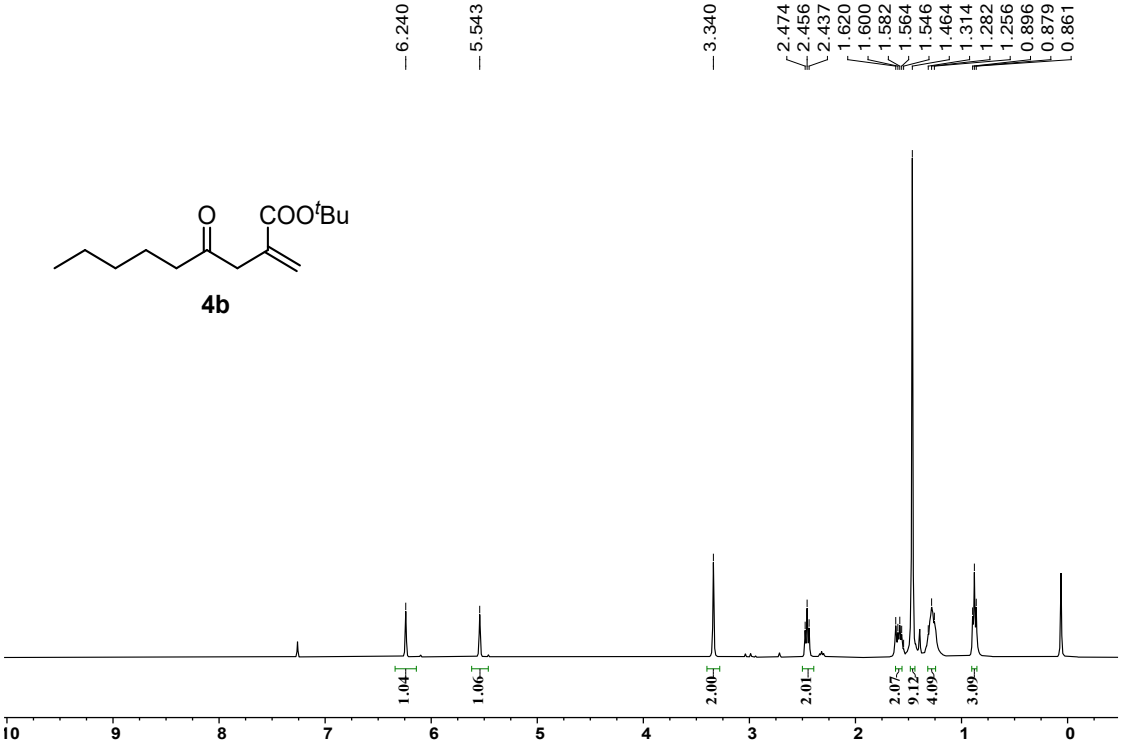
^{1}H NMR of **4a** (400 MHz, $CDCl_3$)



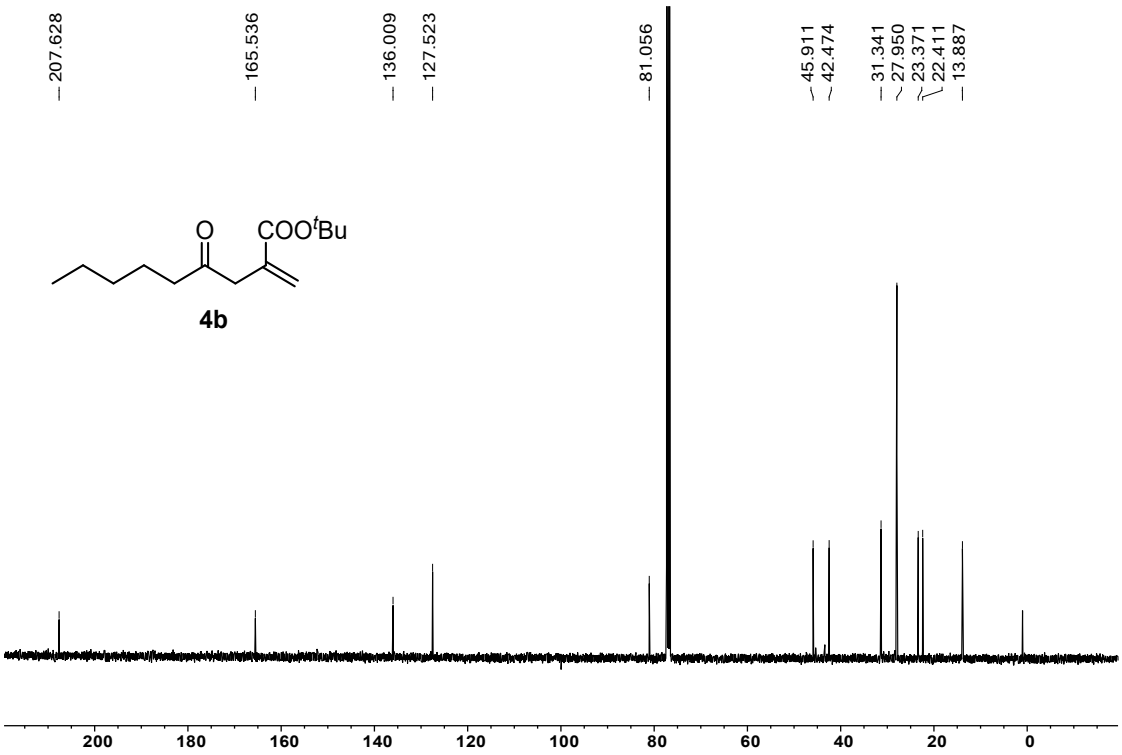
^{13}C NMR of **4a** (101 MHz, $CDCl_3$)



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

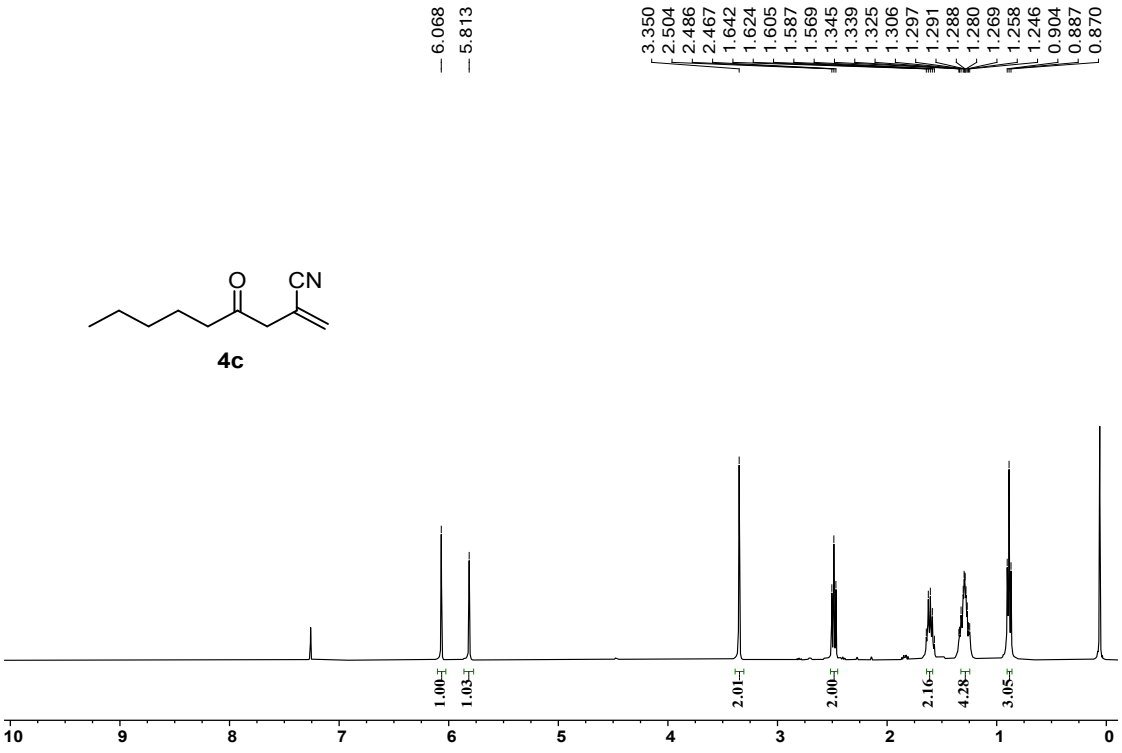


^{13}C NMR of **4b** (101 MHz, $CDCl_3$)



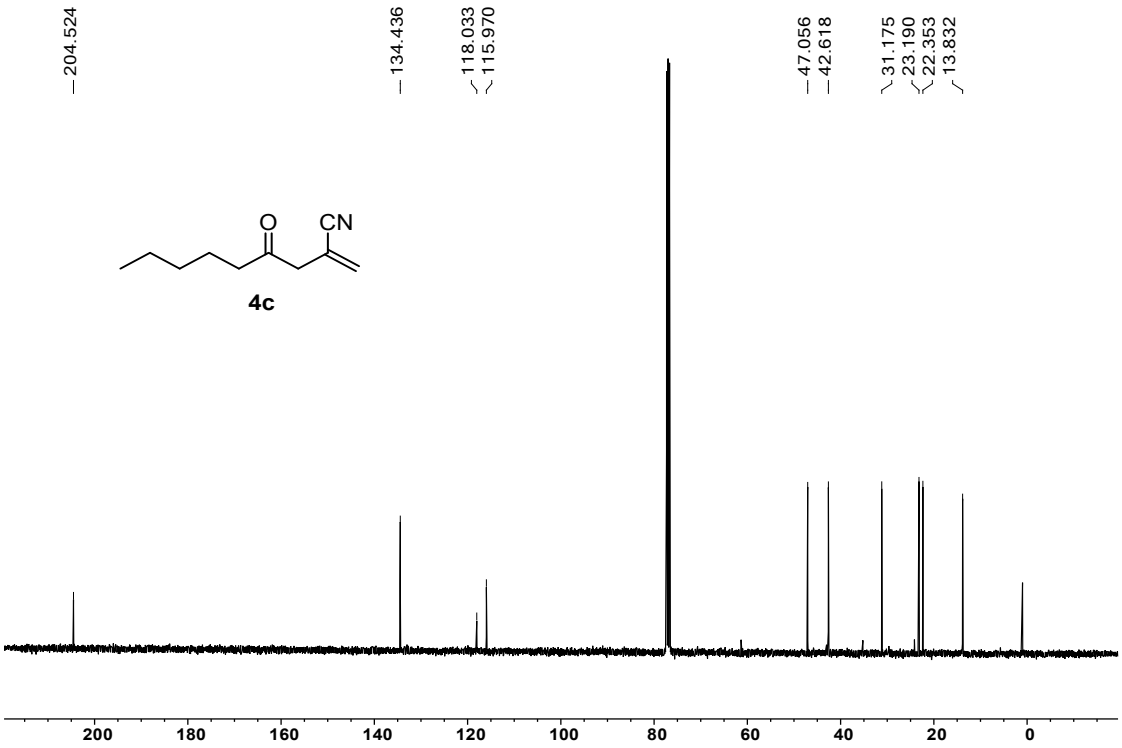
4b

^{1}H NMR of **4c** (400 MHz, $CDCl_3$)

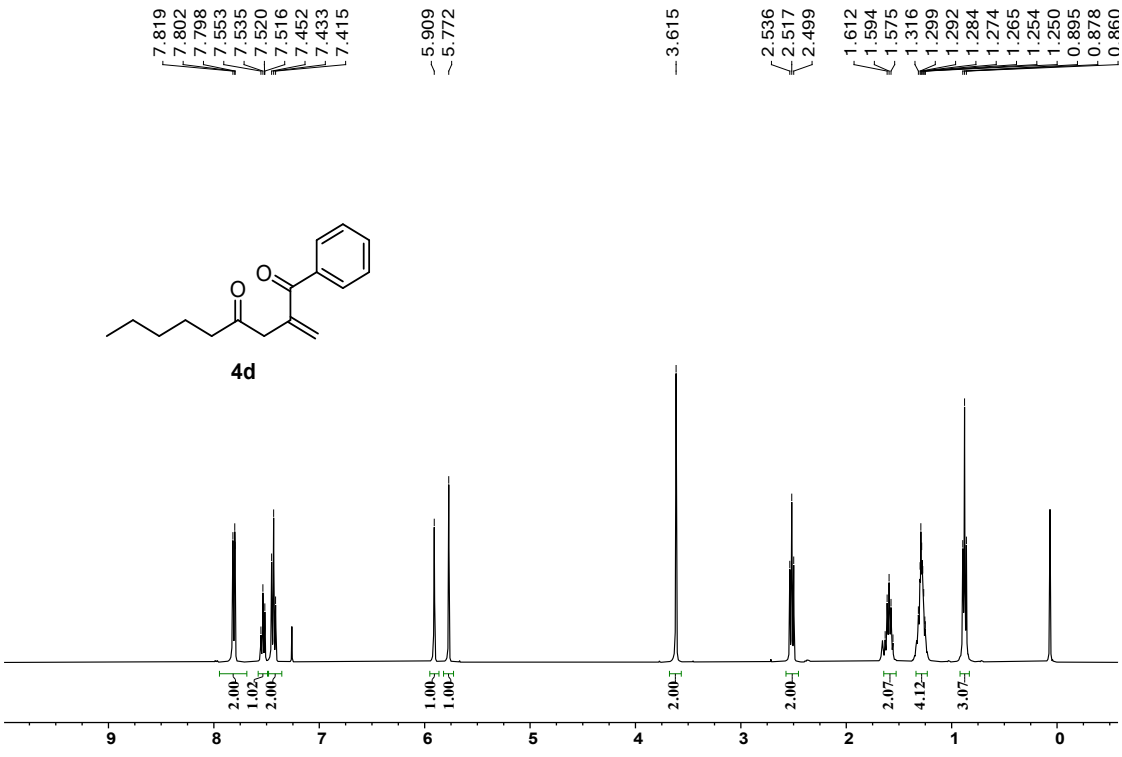


4c

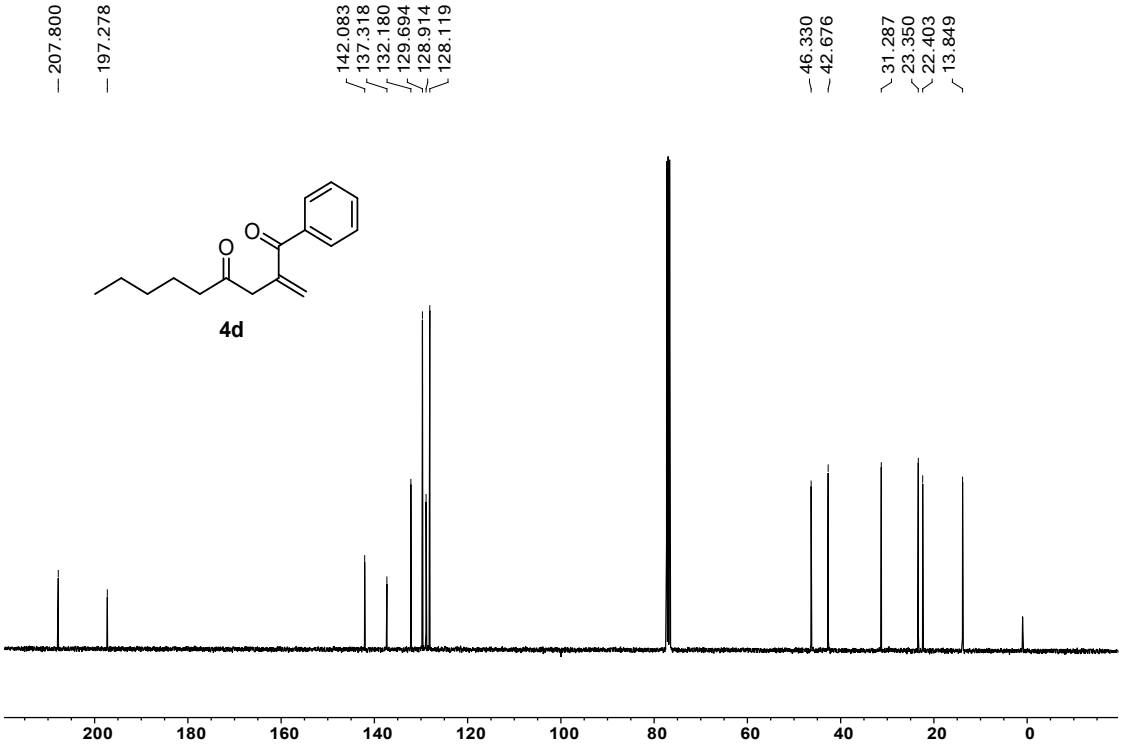
^{13}C NMR of **4c** (101 MHz, $CDCl_3$)



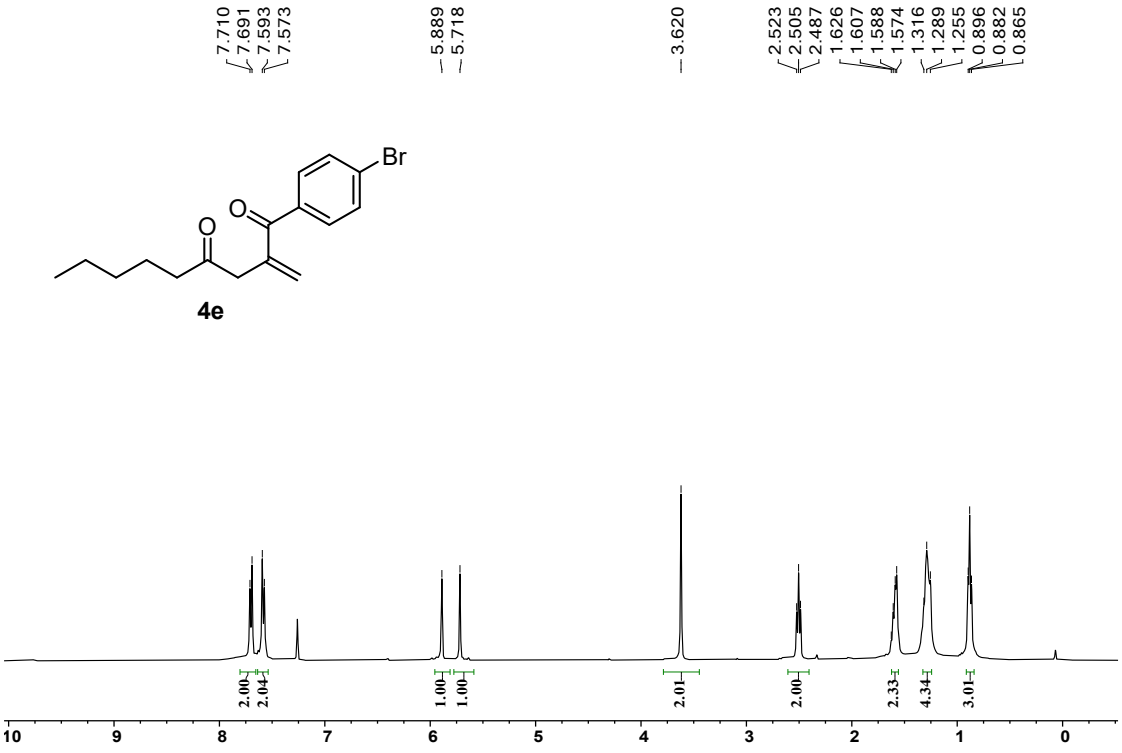
^{1}H NMR of **4d** (400 MHz, $CDCl_3$)



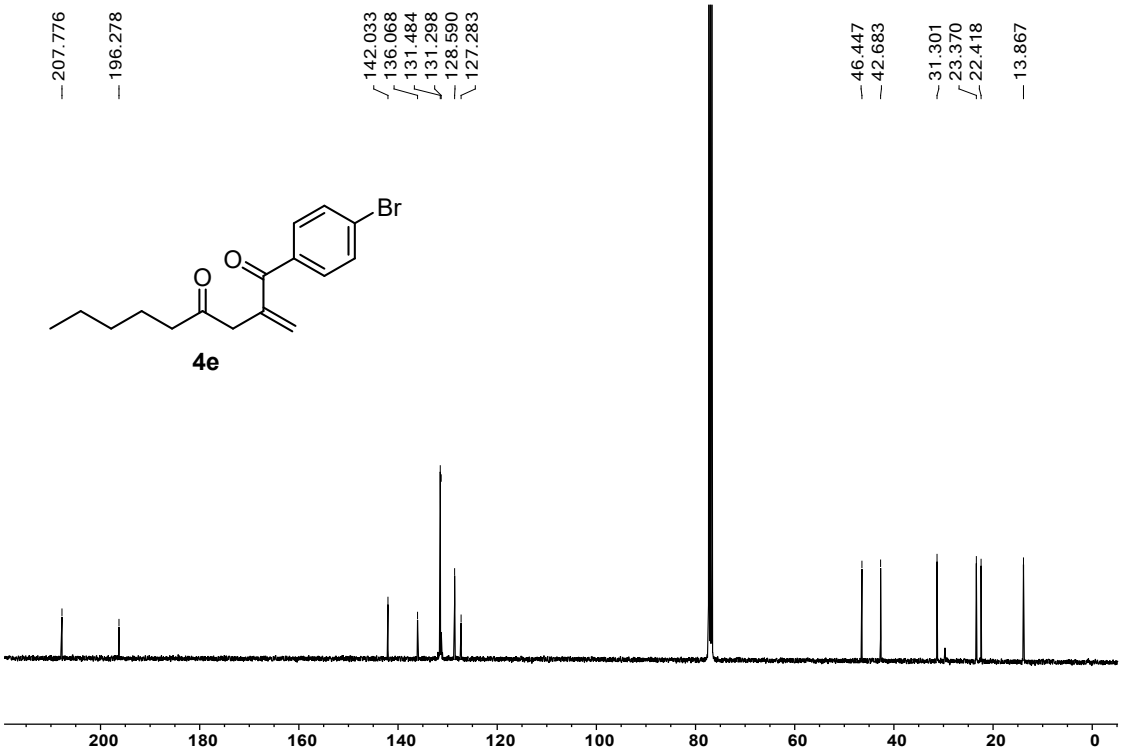
^{13}C NMR of **4d** (101 MHz, $CDCl_3$)



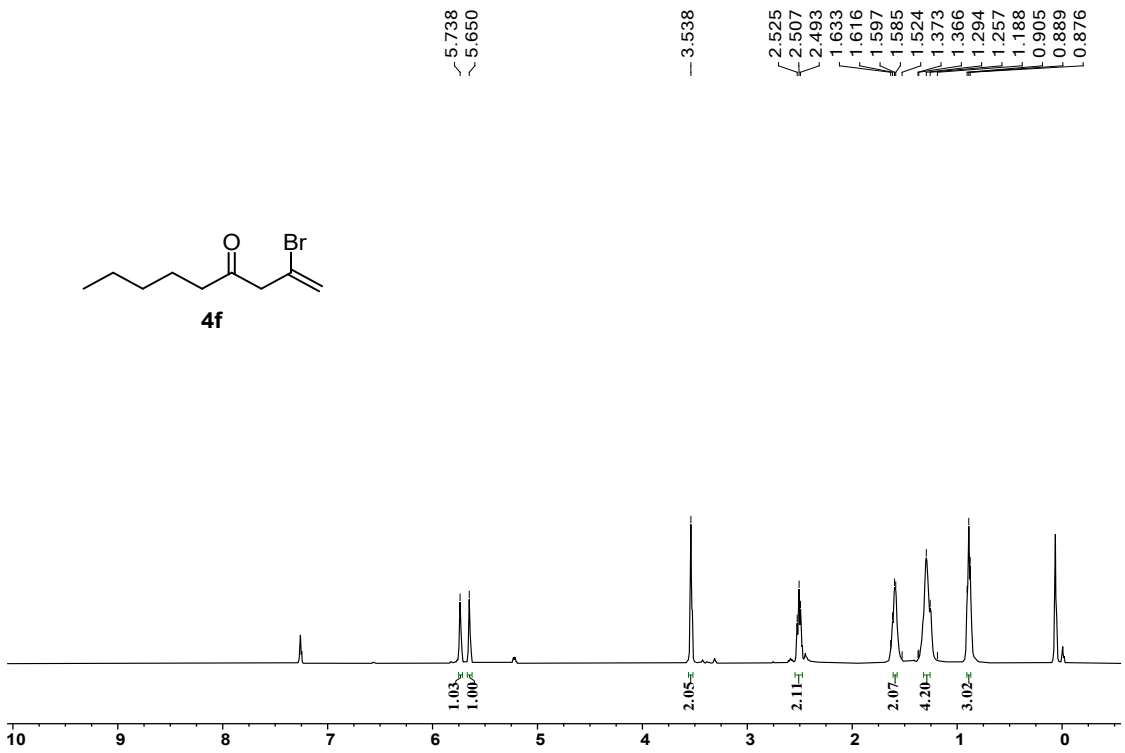
^{1}H NMR of **4e** (400 MHz, $CDCl_3$)



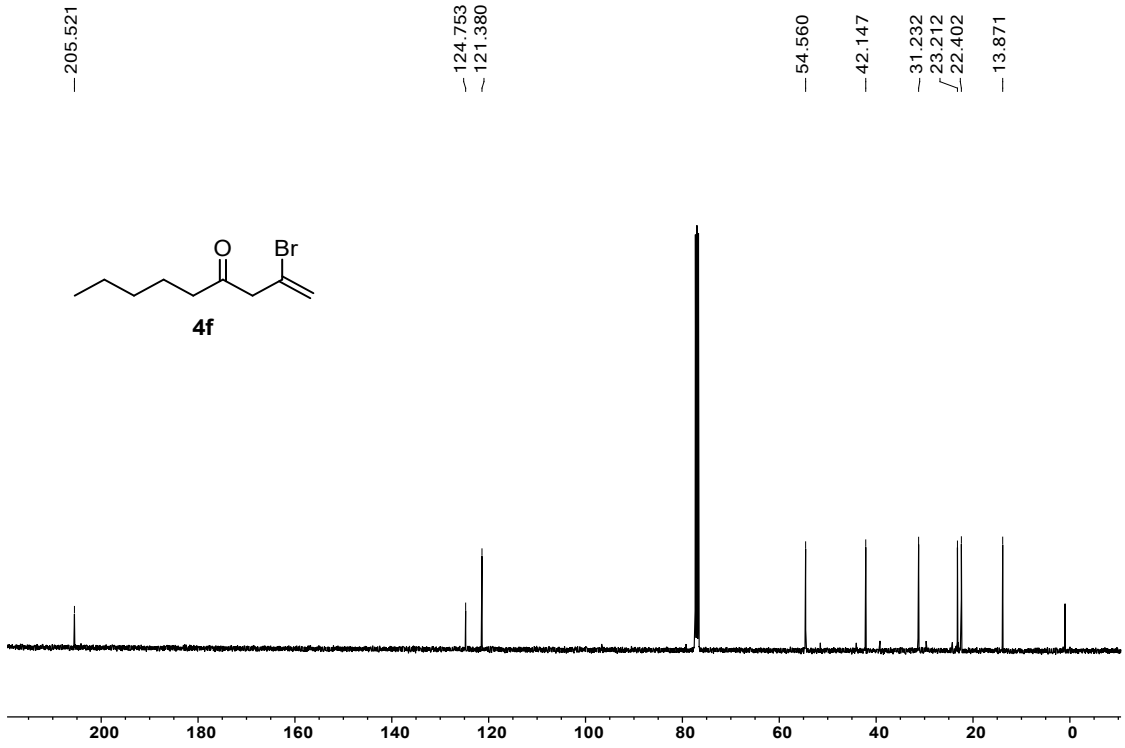
^{13}C NMR of **4e** (101 MHz, $CDCl_3$)



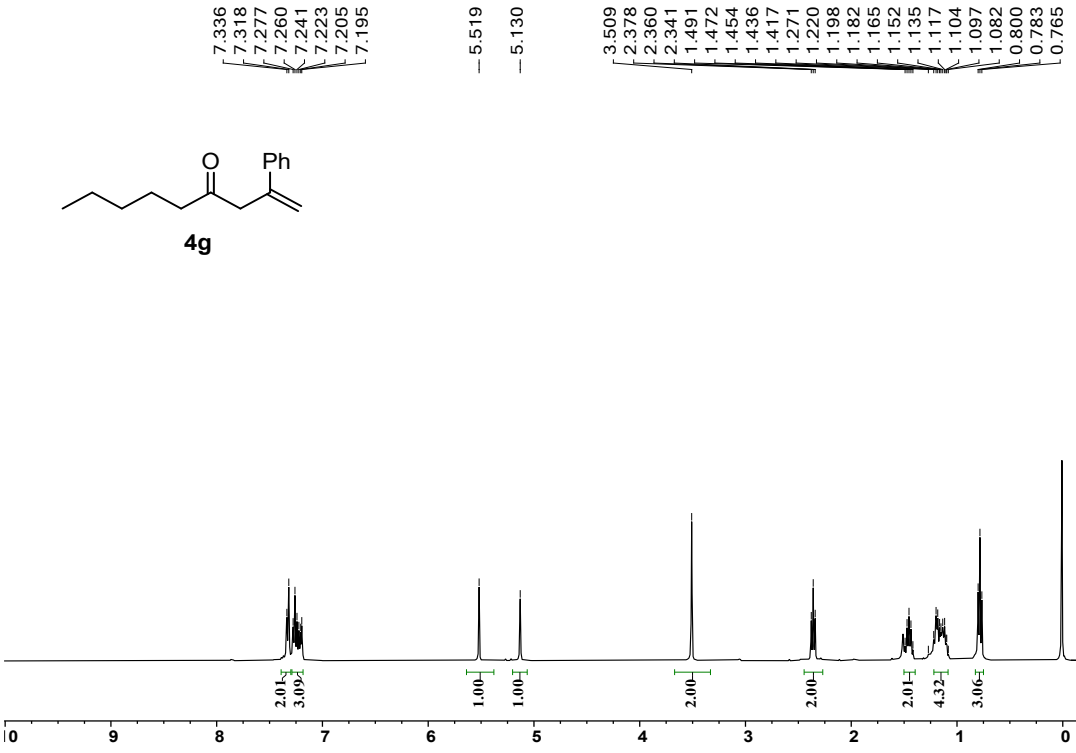
^{1}H NMR of **4f** (400 MHz, $CDCl_3$)



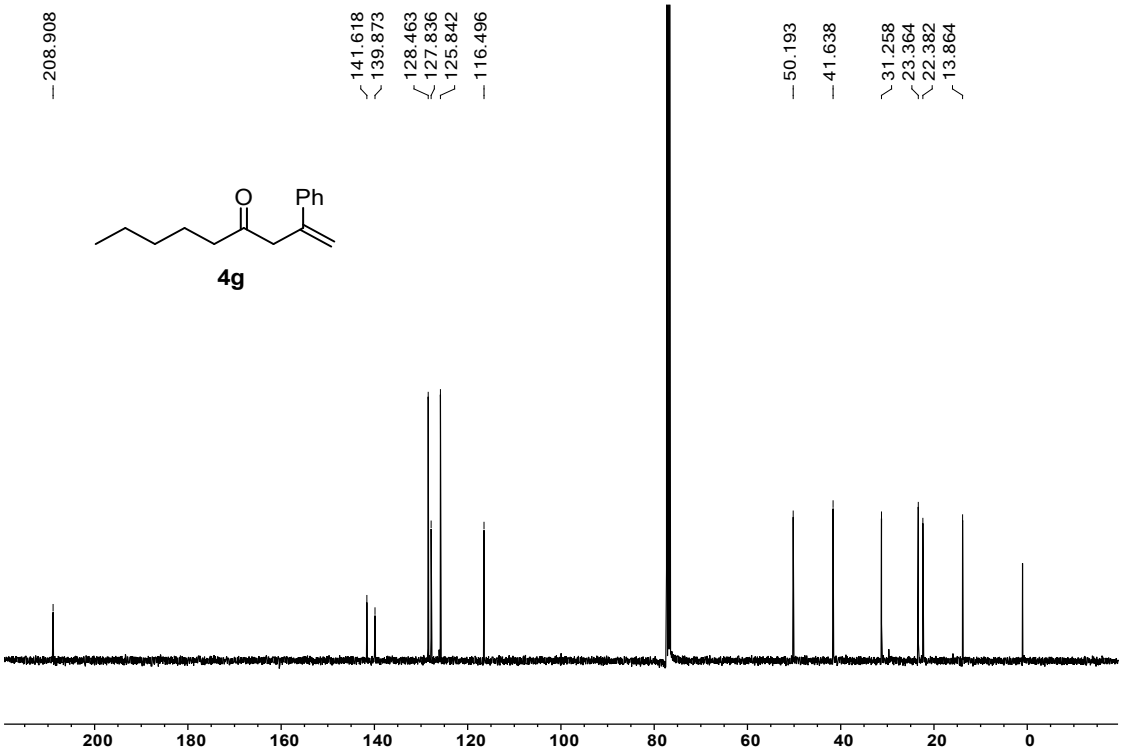
^{13}C NMR of **4f** (101 MHz, $CDCl_3$)



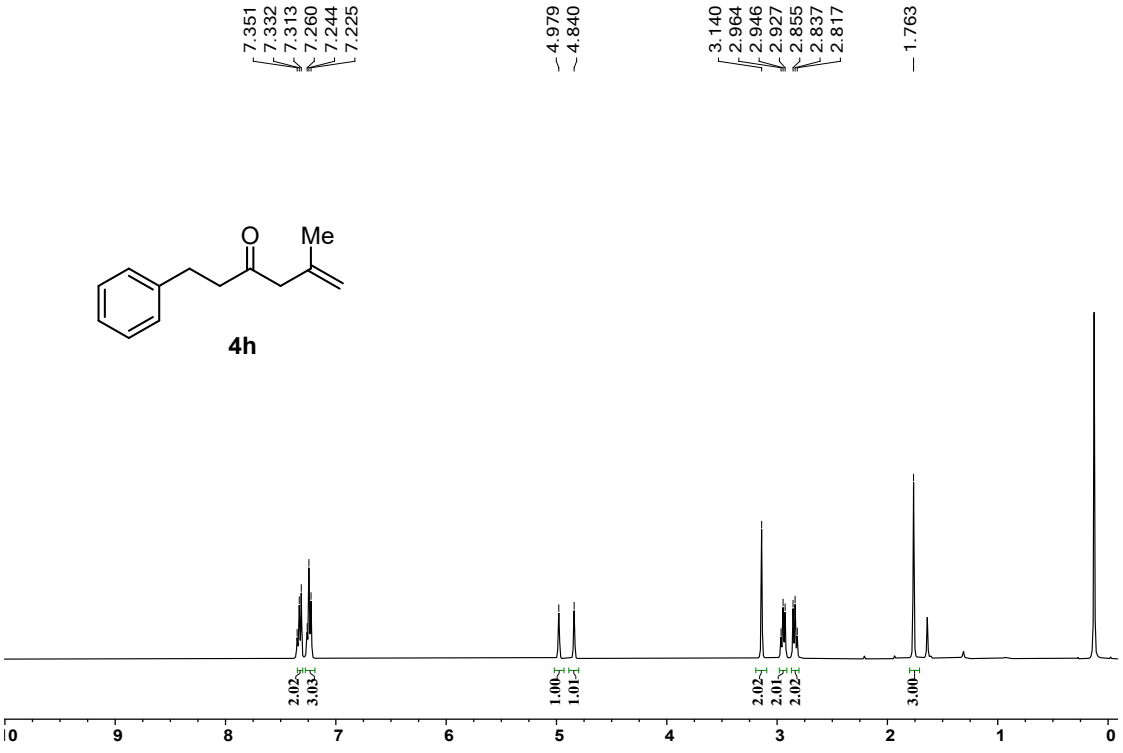
^{1}H NMR of **4g** (400 MHz, $CDCl_3$)



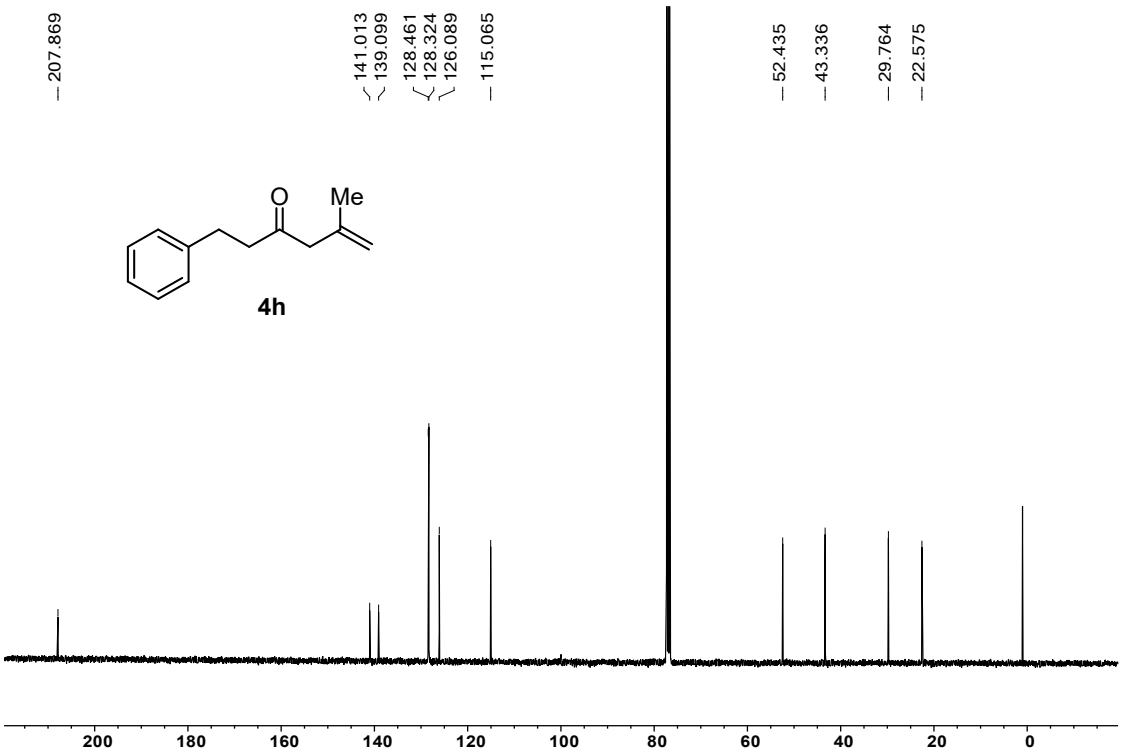
^{13}C NMR of **4g** (101 MHz, $CDCl_3$)



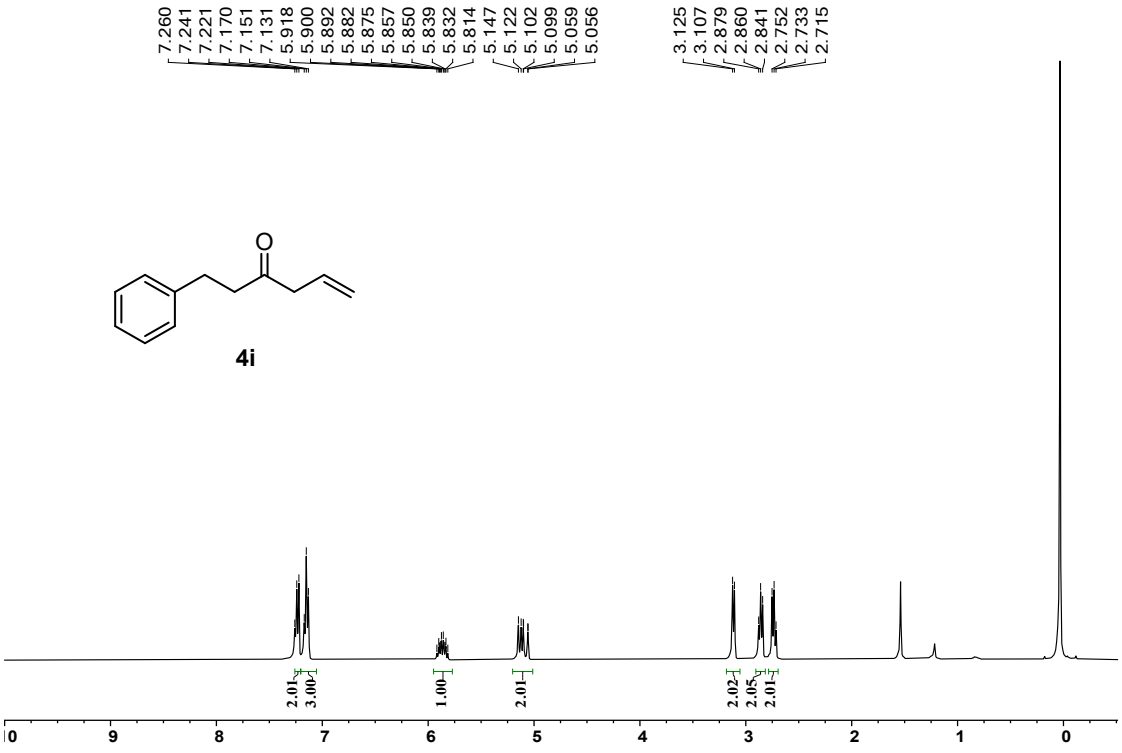
^{1}H NMR of **4h** (400 MHz, $CDCl_3$)



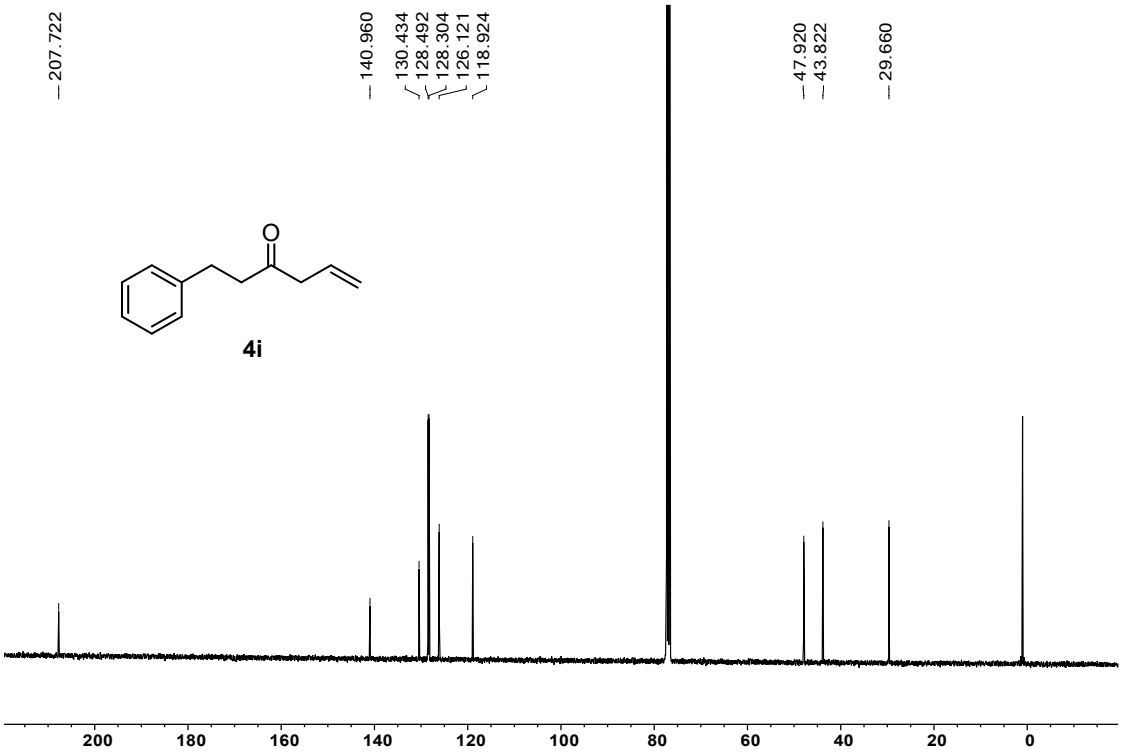
^{13}C NMR of **4h** (101 MHz, $CDCl_3$)



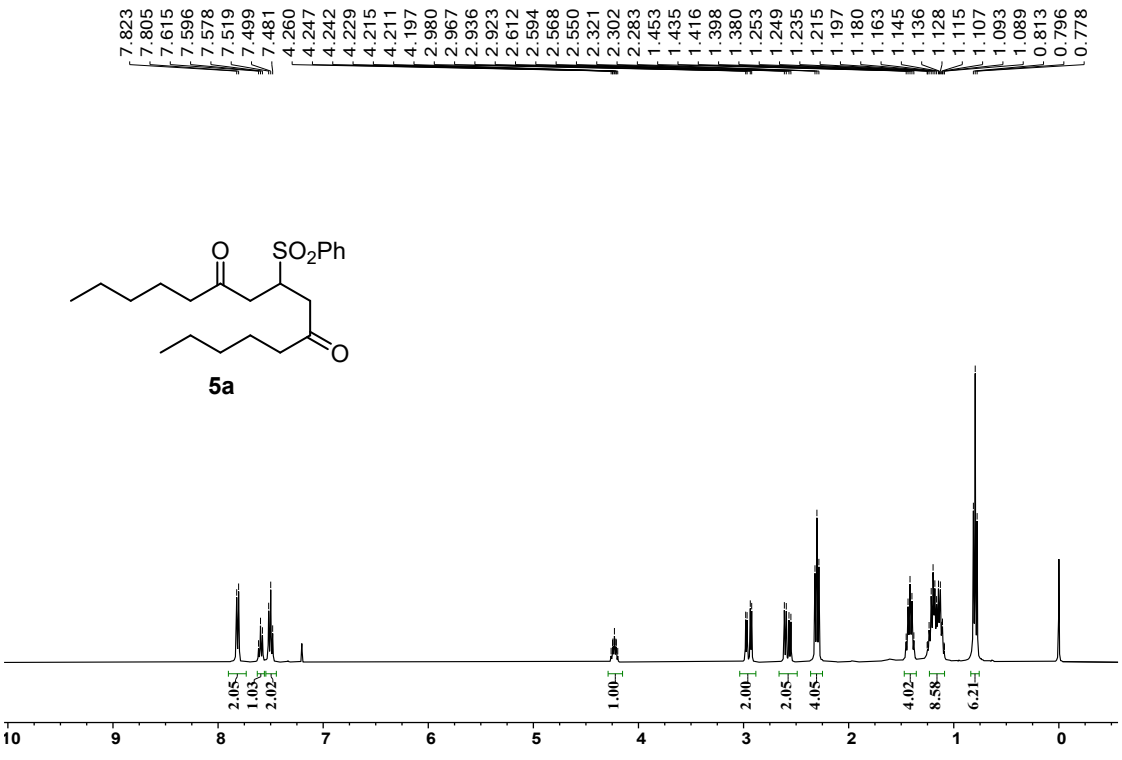
^{1}H NMR of **4i** (400 MHz, $CDCl_3$)



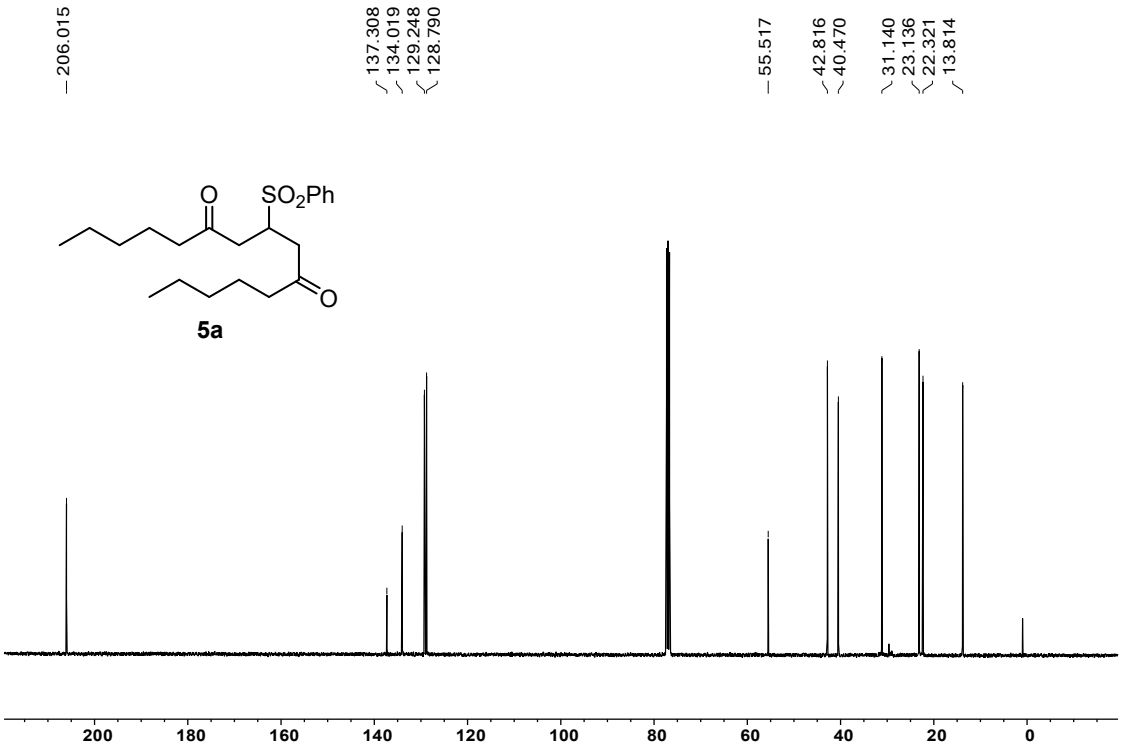
^{13}C NMR of **4i** (101 MHz, $CDCl_3$)



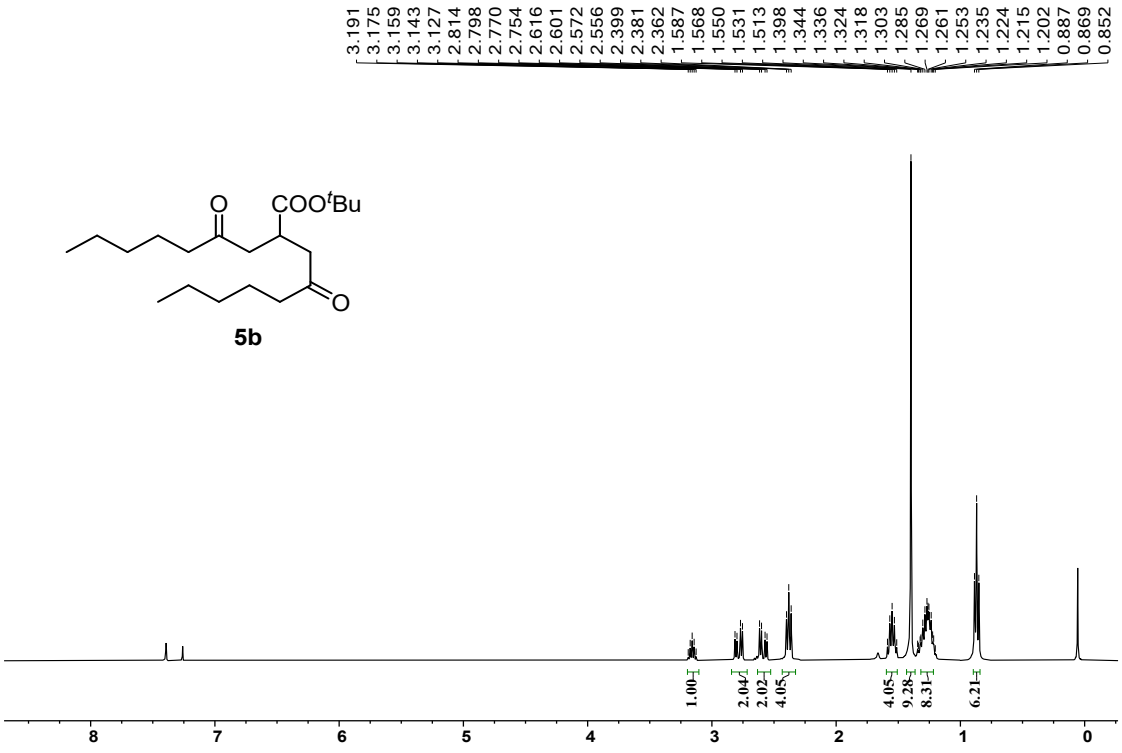
^{1}H NMR of **5a** (400 MHz, $CDCl_3$)



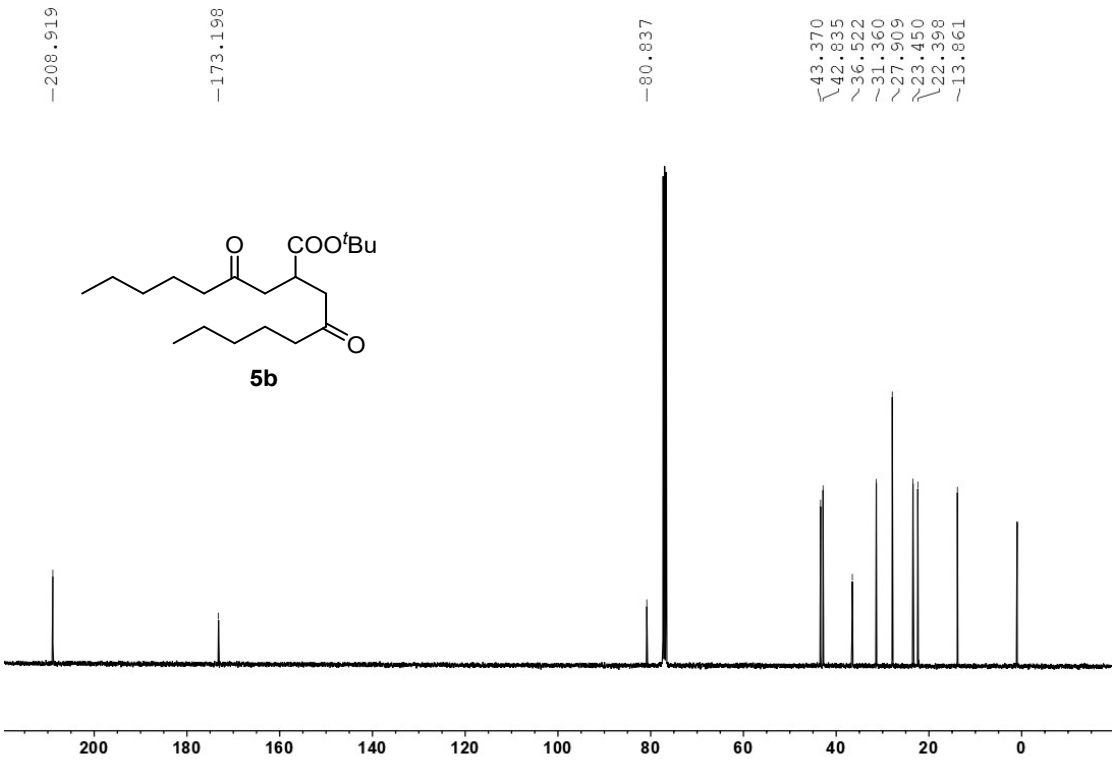
^{13}C NMR of **5a** (101 MHz, $CDCl_3$)



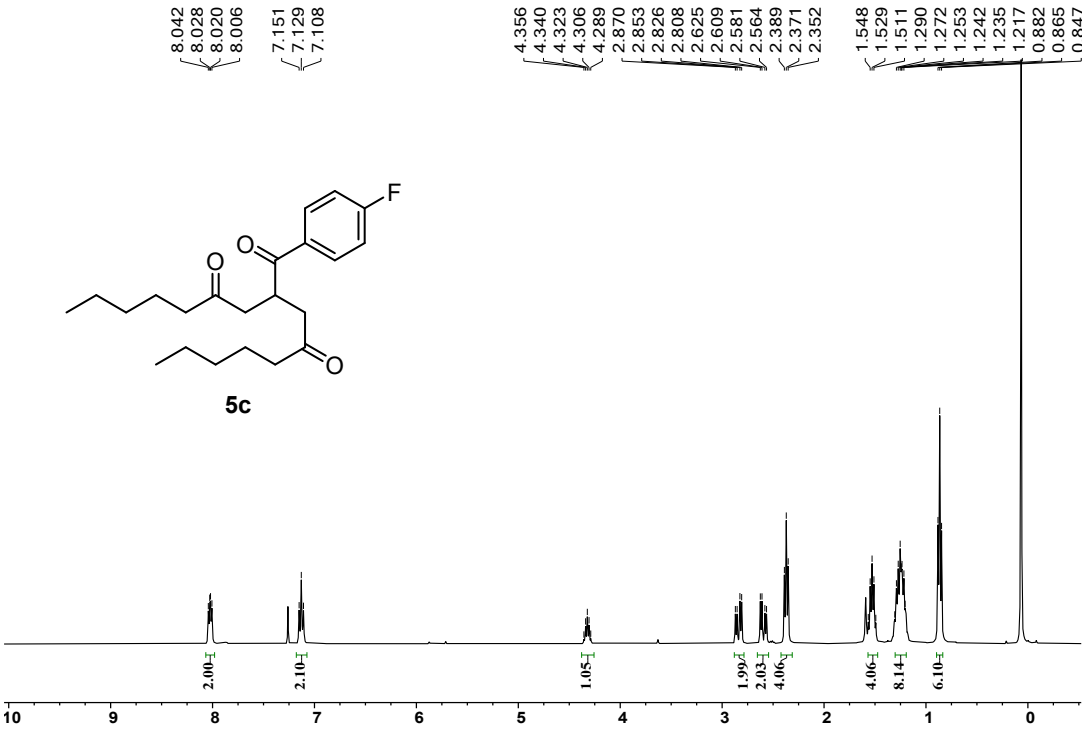
^{1}H NMR of **5b** (400 MHz, $CDCl_3$)



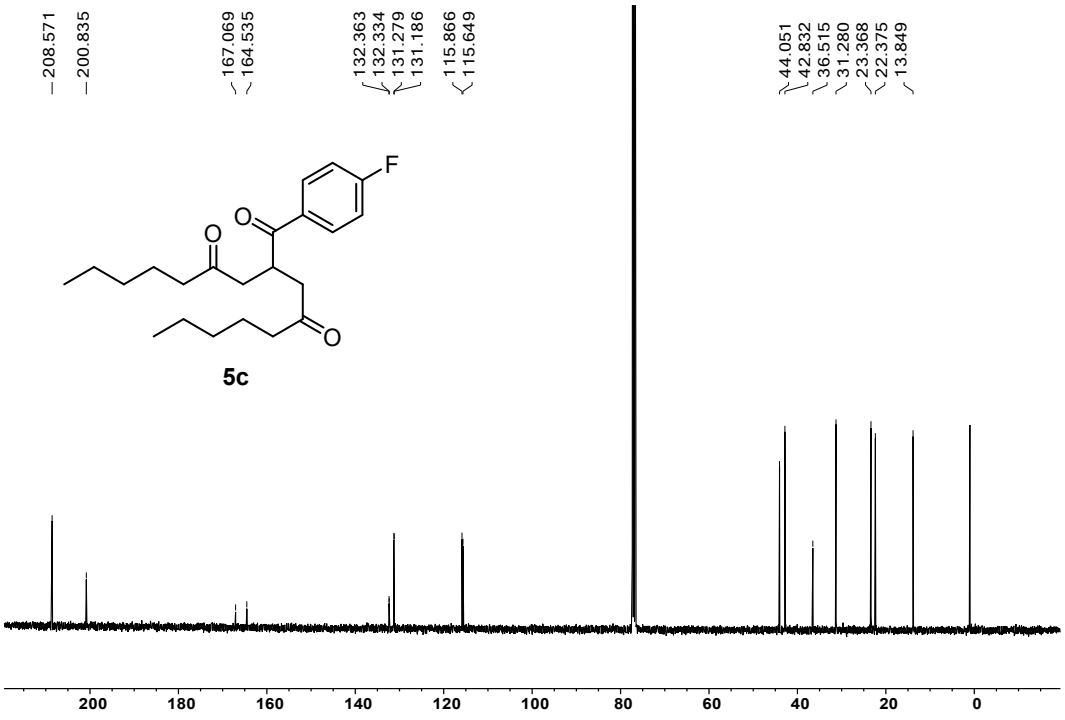
^{13}C NMR of **5b** (101 MHz, $CDCl_3$)



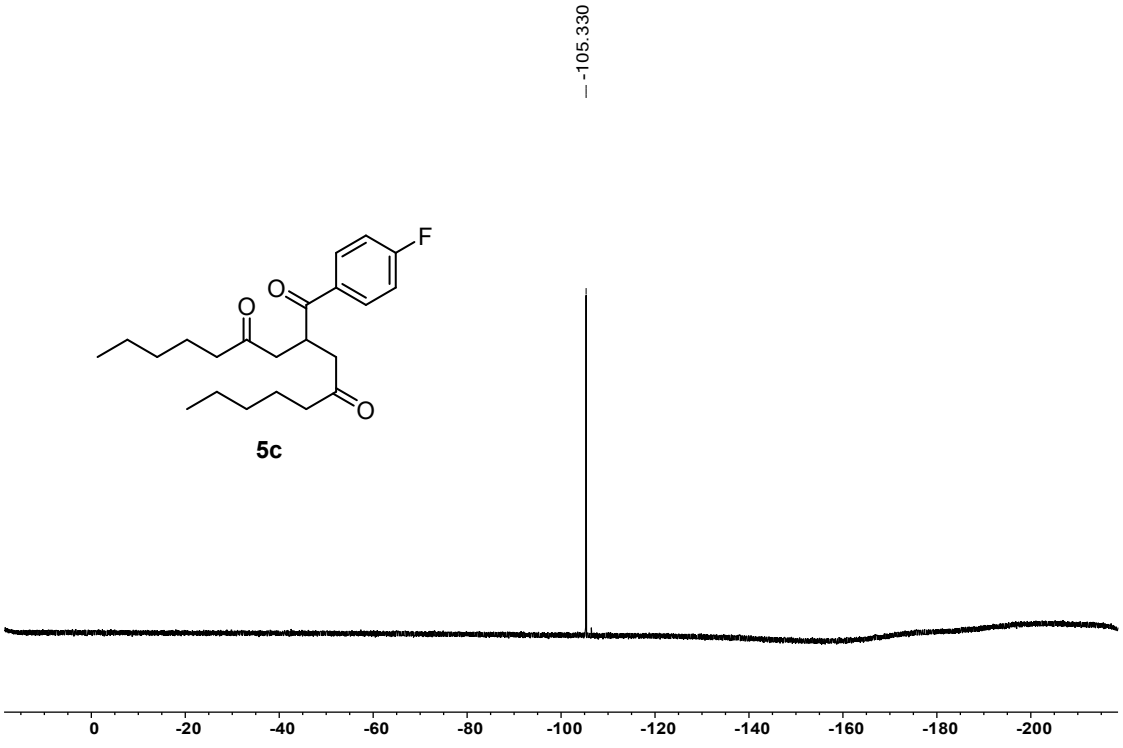
^{1}H NMR of **5c** (400 MHz, $CDCl_3$)



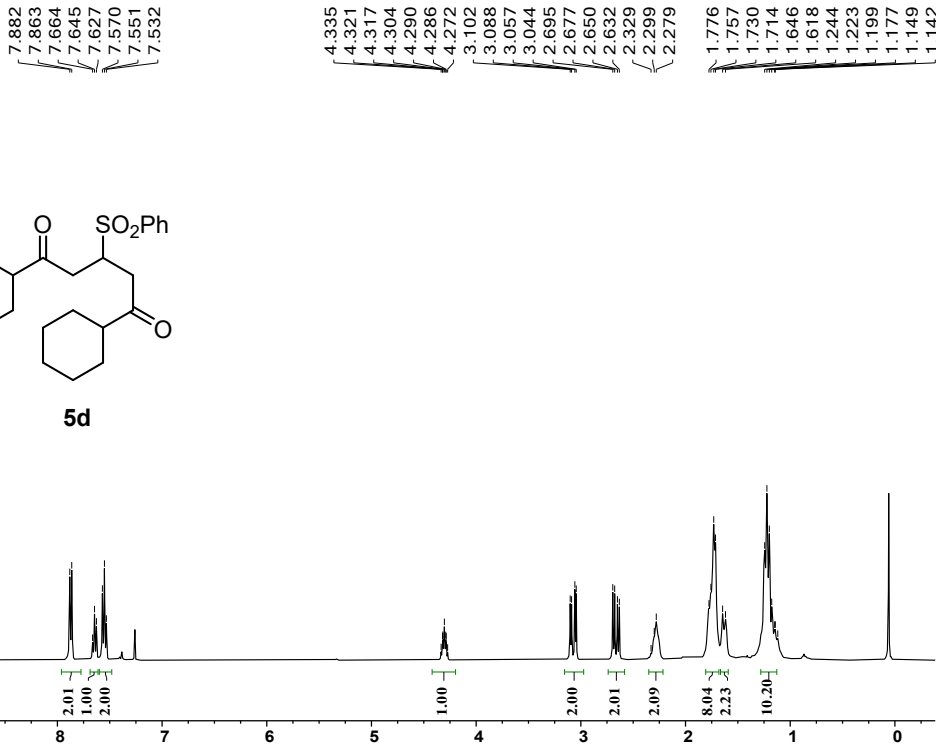
^{13}C NMR of **5c** (101 MHz, $CDCl_3$)



^{19}F NMR of **5c** (376 MHz, $CDCl_3$)

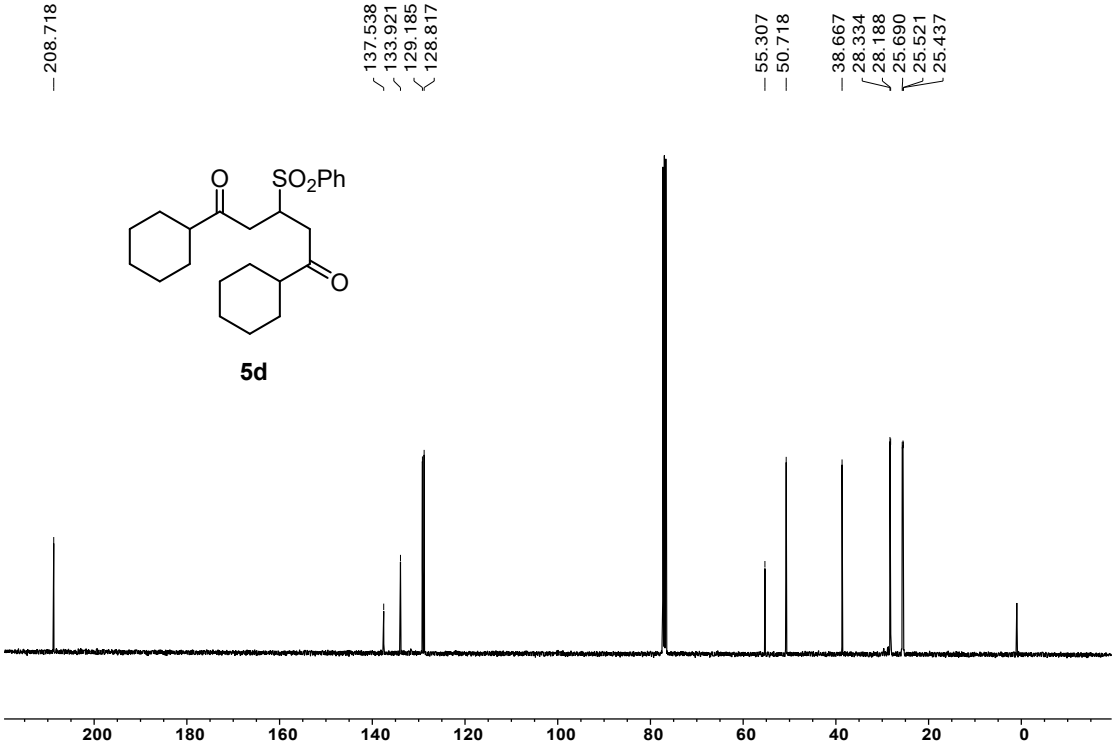


^{1}H NMR of **5d** (400 MHz, $CDCl_3$)



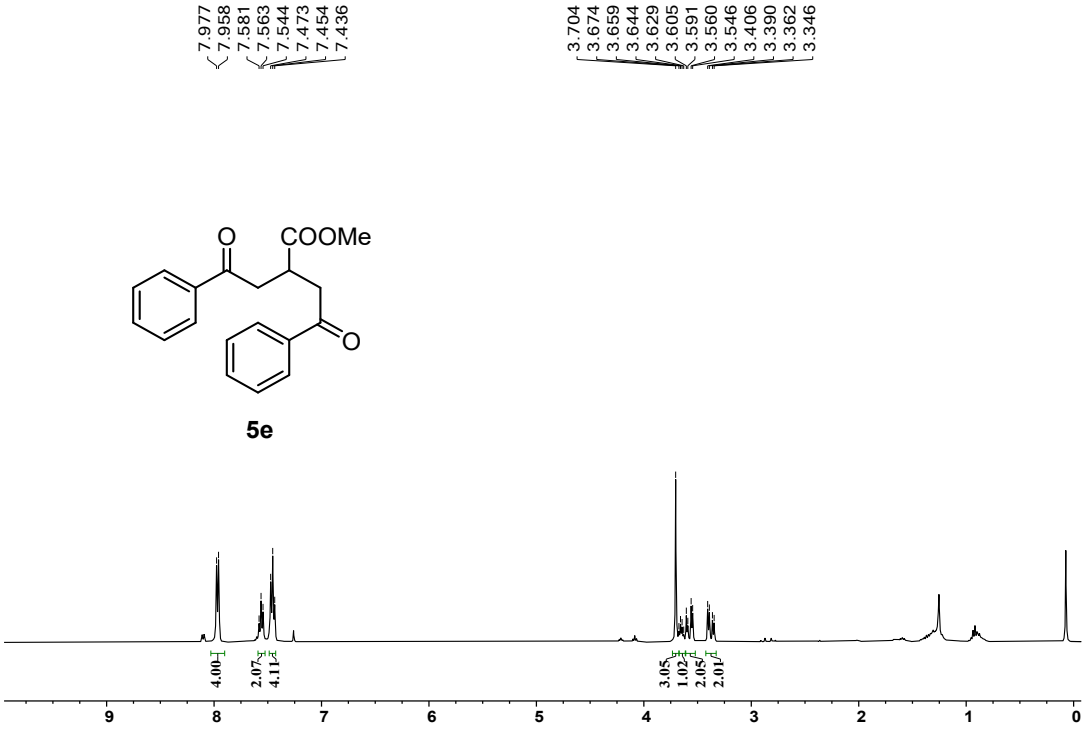
5d

^{13}C NMR of **5d** (101 MHz, $CDCl_3$)



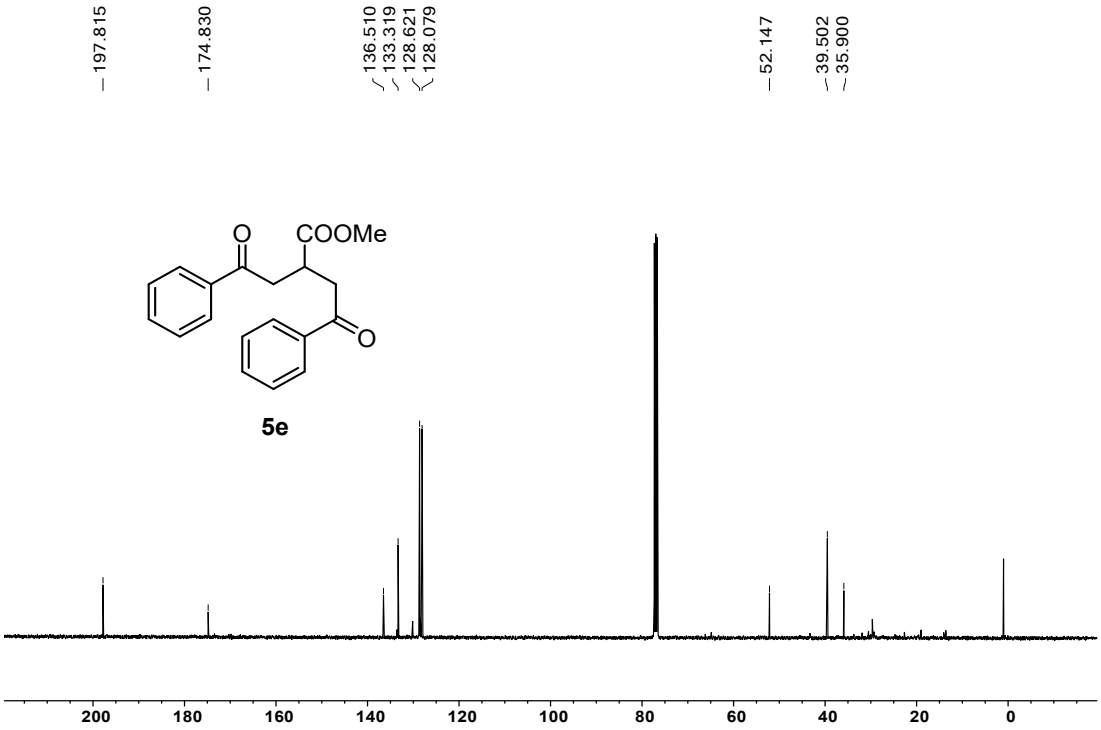
5d

^{1}H NMR of **5e** (400 MHz, $CDCl_3$)

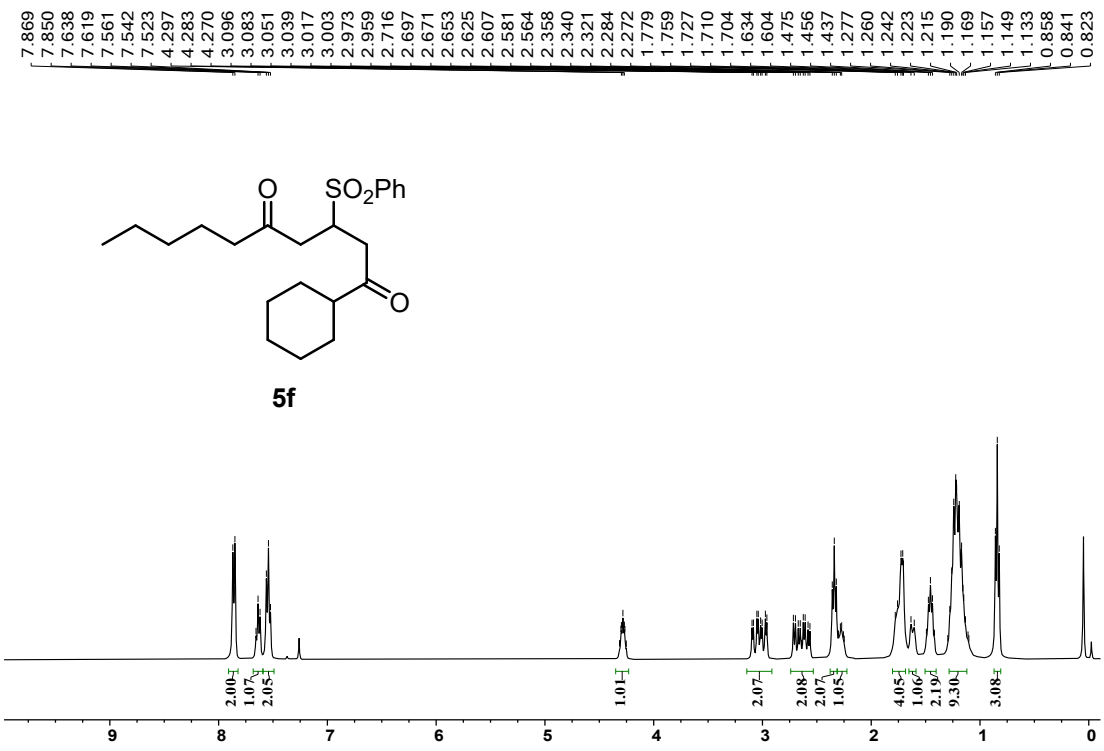


5e

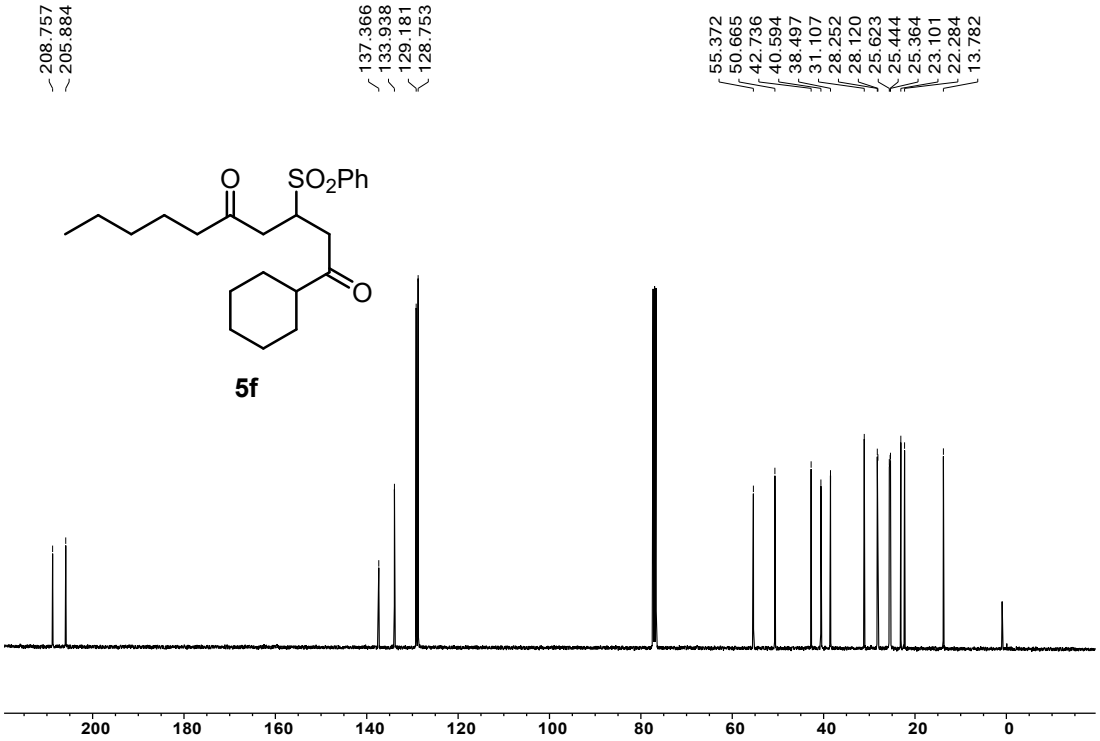
^{13}C NMR of **5e** (101 MHz, $CDCl_3$)



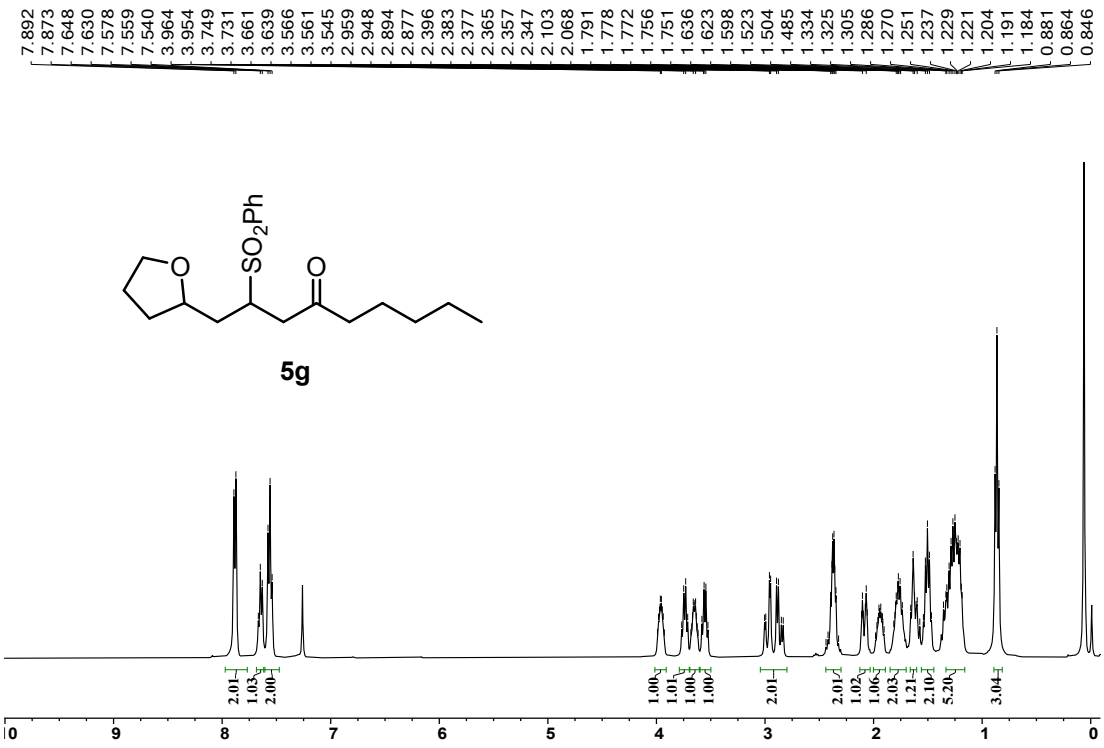
^{1}H NMR of **5f** (400 MHz, $CDCl_3$)



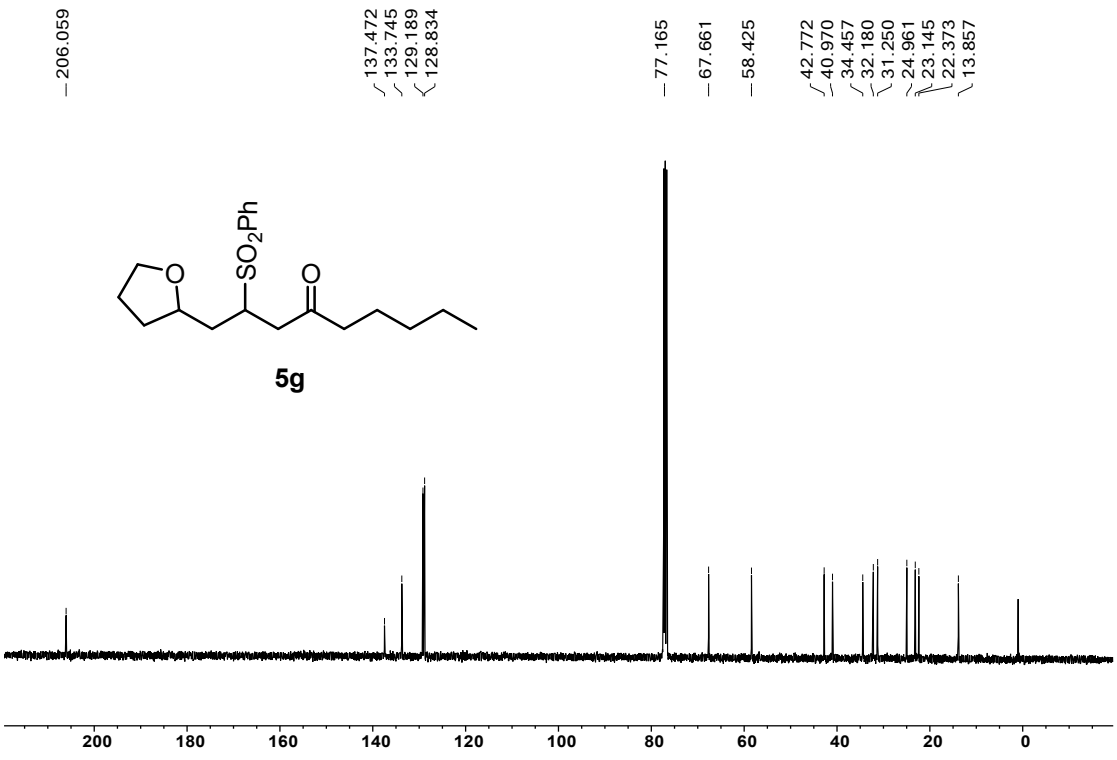
^{13}C NMR of **5f** (101 MHz, $CDCl_3$)



^{1}H NMR of **5g** (400 MHz, $CDCl_3$)



^{13}C NMR of **5g** (101 MHz, $CDCl_3$)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67.661
58.425
42.772
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34.457
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24.961
23.145
22.373
13.857
SO2Ph
O
O
200
180
160
140
120
100
80
60
40
20
0

5g