Supporting Information

Visible-light or sunlight photoredox-catalyzed β-selective acylation of alkenes to access α, β-unsaturated ketones

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Table of Contents

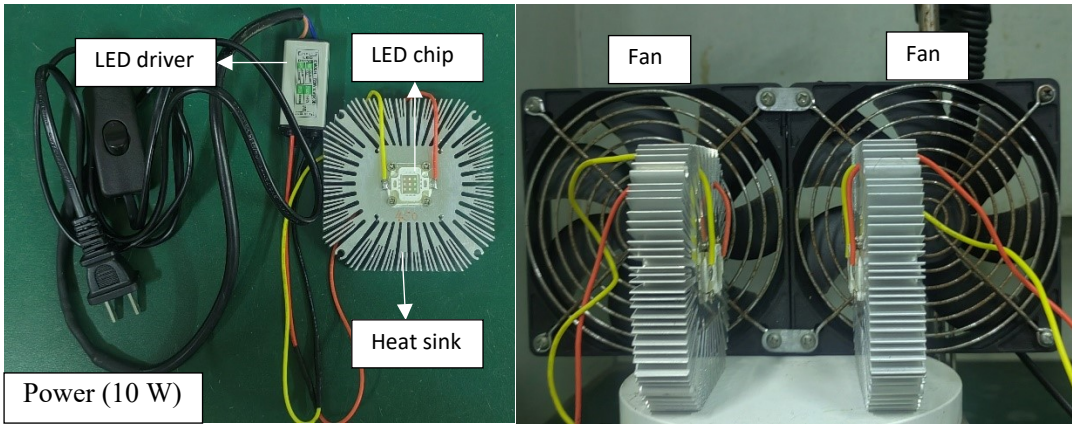
1. General Information

^{1}H NMR spectra were recorded on Bruker 600 MHz spectrometer and the chemical shifts were reported in parts per million (δ) relative to internal solvent signal (7.261 ppm in $CDCl_3$). The peak patterns are indicated as follows: s, singlet; d, doublet; dd, doublet of doublet; t, triplet; q, quartet; m, multiplet. The coupling constants, J, are reported in Hertz (Hz). ^{13}C NMR spectra were obtained at Bruker 126 MHz and referenced to the internal solvent signals (central peak is 77.000 ppm in $CDCl_3$). $CDCl_3$ was used as the NMR solvent. Thermo Q Exactive was used for HRMS and ESI-MS.

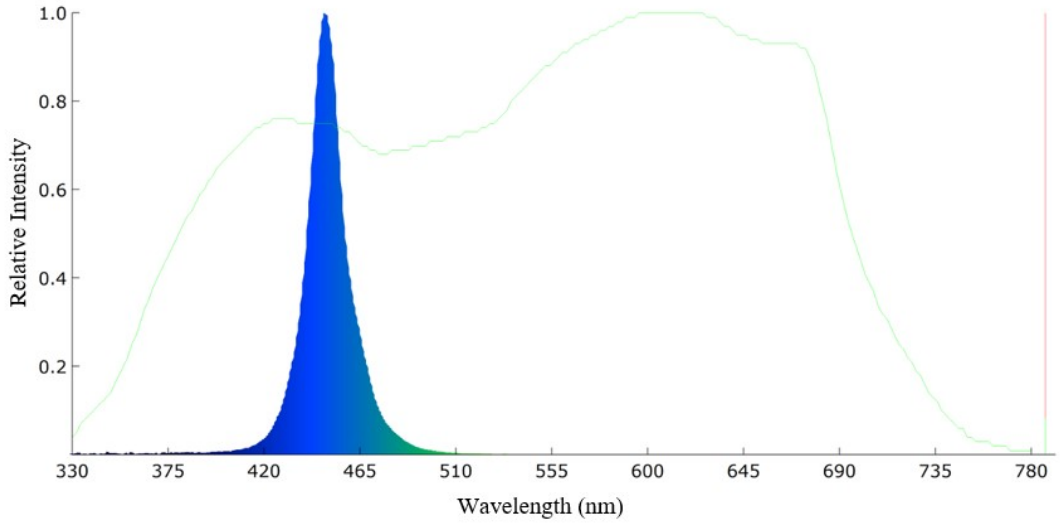
Unless otherwise noted, all reagents were purchased from commercial suppliers (Energy-Chemical, Bidepharm, Heowns, or TCI) and used without further purification. Flash column chromatography was performed over silica gel 200-300. The reagents were weighed and handled in a glove box.

2. Device of Photoredox Reaction

Device purchased from Taobao: https://m.tb.cn/h.gMpfnvu1y5LSsmt



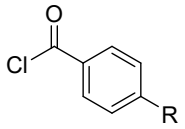
Relative Intensity vs. Wavelength:



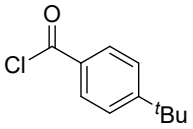
Features of LEDs: Central wavelength = 449.4 nm; Peak wavelength = 449.7 nm.

3. General Procedure for Preparation of Substrates

3.1 List and Preparation of Aroyl Chlorides 1a – 1u



R = H ,**1a**
R = Me ,**1b**



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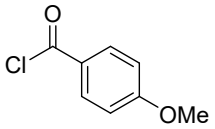

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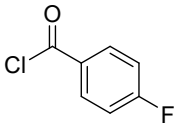

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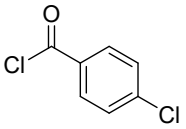

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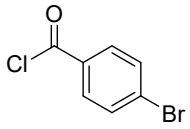

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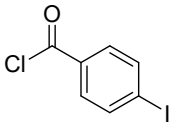

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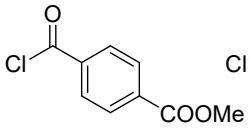

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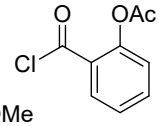

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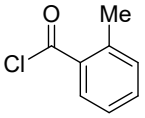

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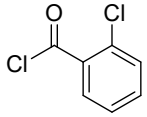

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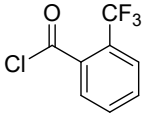

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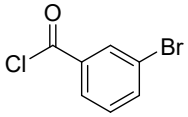

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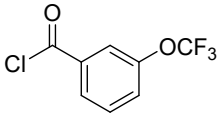

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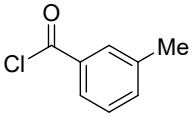

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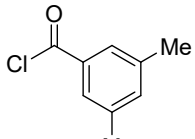

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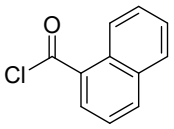

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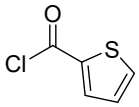

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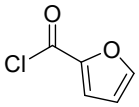
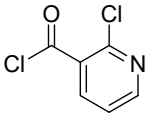


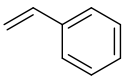
1t



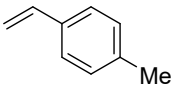
1u

Aroyl chlorides **1a** – **1u** are commercial purchases.

3.2 List and Preparation of Alkenes 2a – 2t



2a



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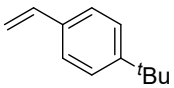

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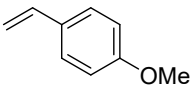

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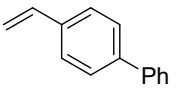

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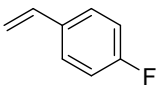

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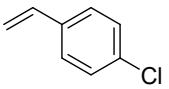

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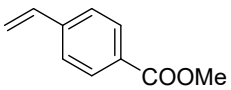

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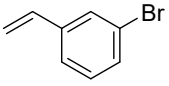

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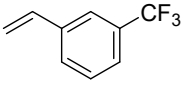

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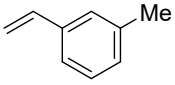

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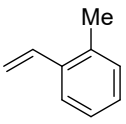

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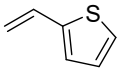

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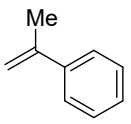

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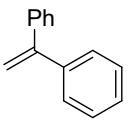

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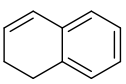

2p

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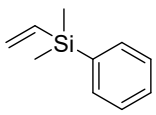

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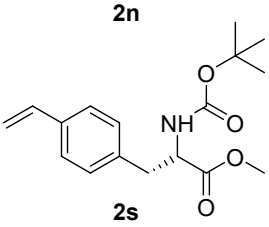

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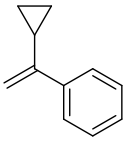
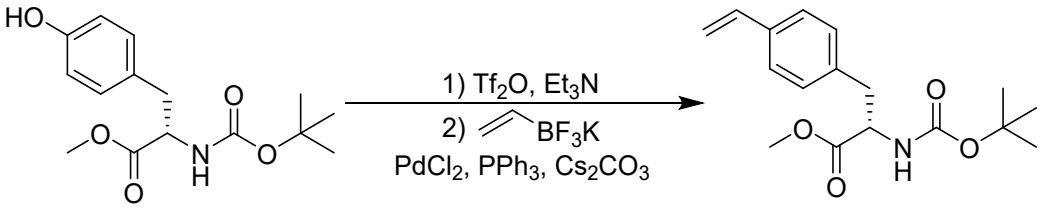


2t

Alkenes **2a** – **2r** are commercial purchases. Compounds **2s**[1] and **2t**[2] were prepared according to the literatures.

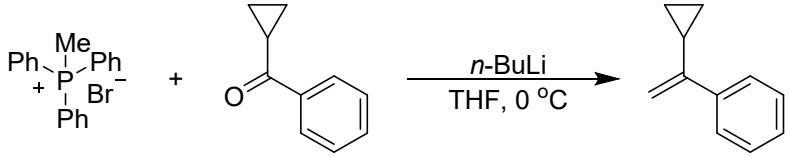
Method for the synthesis of 2s:



1) A dry round-bottomed flask equipped with a magnetic stirring bar was charged with Boc-L Tyrosine methyl ester (0.56 g, 2.0 mmol, 1.0 equiv) and DCM (7.0 mL). The mixture was cooled to 0 °C, after which Et_3N (0.57 mL, 4.0 mmol, 2.0 equiv) and Tf_2O (0.37 mL, 2.2 mmol, 1.1 equiv) were added dropwise. The resulting brown mixture was allowed to warm to rt and further stirred for 3 h. A saturated aqueous solution of $NaHCO_3$ was added and the mixture was extracted three times with DCM. Combined organic phases were dried over anhydrous Na_2SO_4, concentrated in vacuum and the crude product was subjected to column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1 to 5/1, v/v), giving (methyl (*S*)-2-((isopropoxycarbonyl)amino)-3-(4-(((trifluoromethyl)sulfonyl)oxy)phenyl)propanoate) (0.73 g, 90%) as a white solid.

2) In a glovebox, a flame-dried reaction tube equipped with a magnetic stir bar was charged with (methyl(*S*)-2-((isopropoxycarbonyl)amino)-3-(4-(((trifluoromethyl)sulfonyl)oxy)phenyl)propanoate) (413 mg, 1.0 mmol), potassium vinyltrifluoroborate (268 mg, 2.0 mmol, 2.0 equiv), $PdCl_2$ (17.7 mg, 0.1 mmol, 0.1 equiv), PPh_3 (31.4 mg, 0.12 mmol, 0.12 equiv) and Cs_2CO_3 (975 mg, 3.0 mmol, 3.0 equiv). Anhydrous THF (3 mL) and distilled water (600 uL) were added. Then the resulting solution was stirred at 85 °C for 12 h. More water was added and the mixture was extracted three times with DCM. The combined organic phases were dried over anhydrous Na_2SO_4, and concentrated in vacuum, and the crude product was subjected to column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1 and 5/1, v/v) to give **2s** (171 mg, yield: 56%) as a white solid.

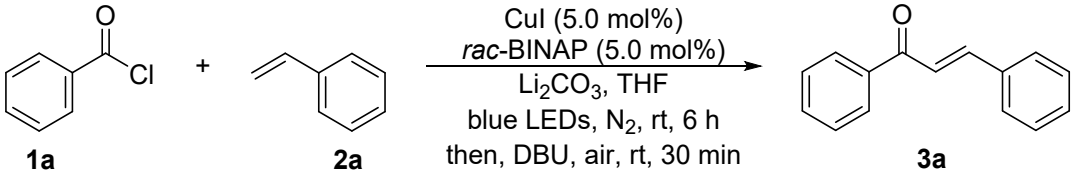
Method for the synthesis of 2t:



To an anhydrous THF (13 mL) solution of methyltriphenylphosphonium bromide (2.1 g, 6.0 mmol) at 0 °C was added *n*-butyl lithium (3.8 mL, 1.6 M, 6.0 mmol) dropwise under a nitrogen atmosphere, and the mixture was stirred for 1 h. After adding cyclopropyl(phenyl)methanone (5.0 mmol), the mixture was stirred at rt for 3 h. Concentrated in vacuum, and the crude product was subjected to column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (100/1, v/v) to give **2t** (713 mg, 98%) as a colorless liquid.

4. Optimization of the Reaction Conditions

Table S1. Optimization of the reaction conditions [a,b]

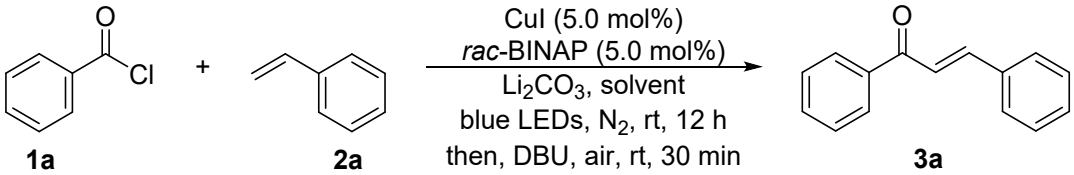


1a **2a** **3a**

entry	Variations of the standard conditions	**3a**/yield/%
1	None	88
2	Sunlight instead of blue LEDs	67
3	No light	0
4	No CuI and *rac*-BINAP	0
5	No light, 60 °C	0
6	No Li_2CO_3	19
7	DBU instead of Li_2CO_3	0
8	THF/DCE(1:1) instead of THF	83
9	4CzIPN instead of CuI and *rac*-BINAP	17
10	air instead of N_2	0

[a] Reaction conditions: **1a** (0.30 mmol), **2a** (0.20 mmol), CuI (5.0 mol%), *rac*-BINAP (5.0 mol%), and Li_2CO_3 (1.0 equiv) in THF (1.0 mL) irradiated with blue LEDs for 6 h at rt (25-30 °C). Afterward, 3.0 equiv of DBU was added to the mixture, followed by stirring for 30 min; [b] Isolated yields.

Table S2. Screening of solvent for **3a** [a,b]

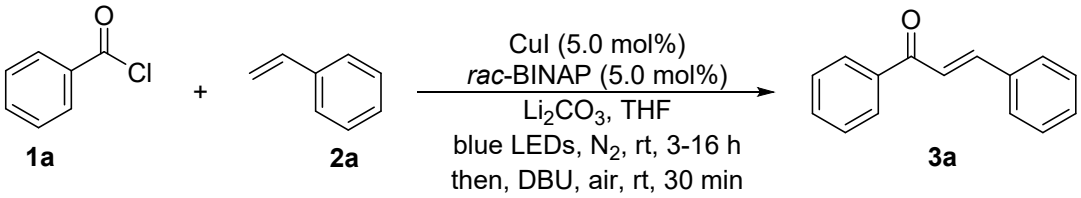


1a **2a** **3a**

entry	solvent	**3a**/yield/%
1	DMA	51
2	THF	72
3	CPME	61
4	2-MeTHF	46
5	$PhCF_3$	27
6	PhF	31
7	DCE	24
8	DMSO	0
9	Dioxane	24
10	MeCN	28

[a] Reaction conditions: **1a** (0.30 mmol), **2a** (0.20 mmol), CuI (5.0 mol%), *rac*-BINAP (5.0 mol%), and Li_2CO_3 (2.0 equiv) in solvent (1.0 mL) irradiated with blue LEDs for 6 h at rt (25-30 °C). Afterward, 3.0 equiv of DBU was added to the mixture, followed by stirring for 30 min; [b] Isolated yields.

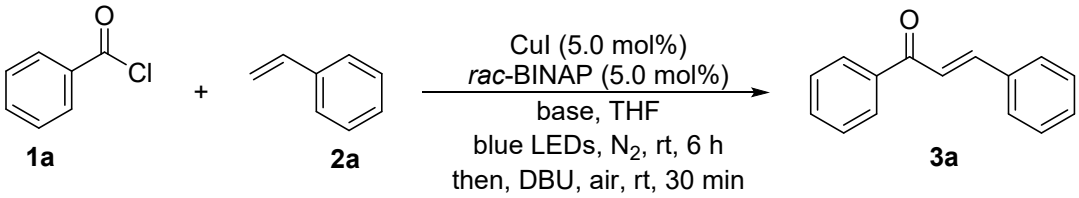
Table S3. Screening of time for **3a** [a,b]



entry	time	**3a**/yield/%
1	16	70
2	12	72
3	9	72
4	6	73
5	3	39

[a] Reaction conditions: **1a** (0.30 mmol), **2a** (0.20 mmol), CuI (5.0 mol%), *rac*-BINAP (5.0 mol%), and Li_2CO_3 (2.0 equiv) in THF (1.0 mL) irradiated with blue LEDs for 3-16 h at rt (25-30 ^oC). Afterward, 3.0 equiv of DBU was added to the mixture, followed by stirring for 30 min; [b] Isolated yields.

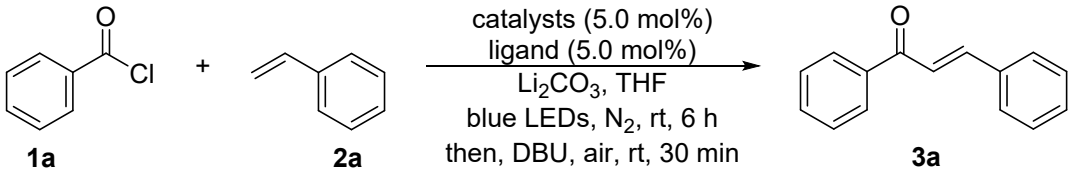
Table S4. Screening of base for **3a** [a,b]



entry	base	**3a**/yield/%
1	Li_2CO_3	73
2	^{t}BuOK	0
3	^{t}BuOLi	0
4	Rb_2CO_3	0
5	Cs_2CO_3	trace
6	K_2HPO_4	38
7	Na_2CO_3	31
8	$NaHCO_3$	40
9	Et_3N	16
10	2,6-di^{t}Bu-4-MePy	20
11	DBU	0
12	Li_2CO_3	88[c]
13	Ag_2CO_3	65[c]

[a] Reaction conditions: **1a** (0.30 mmol), **2a** (0.20 mmol), CuI (5.0 mol%), *rac*-BINAP (5.0 mol%), and base (2.0 equiv) in THF (1.0 mL) irradiated with blue LEDs for 6 h at rt (25-30 °C). Afterward, 3.0 equiv of DBU was added to the mixture, followed by stirring for 30 min; [b] Isolated yields; [c] base (1.0 equiv).

Table S5. Screening of catalysts and ligands for **3a** [a,b]



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

entry	catalyst	ligand	**3a**/yield/%
1	CuCl	*rac*-BINAP	72
2	CuBr	*rac*-BINAP	82
3	CuI	*rac*-BINAP	88
4	Cu_2O	*rac*-BINAP	75
5	CuO	*rac*-BINAP	46
6	CuI	dppf	trace
7	CuI	XantPhos	trace
8	CuI	dppe	trace
9	CuI	DPEPhos	trace
10 [c]	4CzIPN	-	17

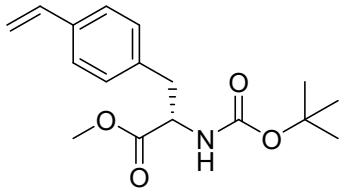
[a] Reaction conditions: **1a** (0.30 mmol), **2a** (0.20 mmol), catalyst (5.0 mol%), ligand (5.0 mol%), and Li_2CO_3 (1.0 equiv) in THF (1.0 mL) irradiated with blue LEDs for 6 h at rt (25-30 °C). Afterward, 3.0 equiv of DBU was added to the mixture, followed by stirring for 30 min; [b] Isolated yields; [c] catalyst (2.0 mol%).

5. General Procedure for Products 3 and 4

In a glovebox, a quartz test tube (20*165 mm, 30 mL) equipped with a magnetic stir bar was charged with CuI (1.9 mg, 5.0 mol%), *rac*-BINAP (6.2 mg, 5.0 mol%), aroyl chlorides **1** (0.3 mmol), alkenes **2** (0.2 mmol), Li_2CO_3 (0.2 mmol), and THF (1.0 mL) before being sealed with a rubber stopper. Then, the test tube was placed in the photoreactor about 1.5 cm away from 10 W blue LEDs light irradiation with a fan to maintain the temperature at rt. After 6 h, DBU (0.6 mmol) was added to the reaction mixture in air and stirred for 30 minutes at rt without irradiation. Finally, the reaction mixture was concentrated under reduced pressure and the crude product was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate) to give products **3** and **4**.

6. Characterization Data of Products

methyl (*S*)-2-((*tert*-butoxycarbonyl)amino)-3-(4-vinylphenyl)propanoate (2s)

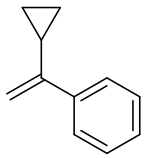


Following the method for the synthesis of **2s**, the title compound was obtained as a white solid in 56% (171 mg) yield. The spectral data were in accordance with those reported in the literature.[3]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.33 (d, *J* = 8.1 Hz, 2H), 7.08 (d, *J* = 7.8 Hz, 2H), 6.68 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.72 (dd, *J* = 17.6, 0.9 Hz, 1H), 5.22 (dd, *J* = 10.9, 0.9 Hz, 1H), 4.98 (d, *J* = 8.1 Hz, 1H), 4.58 (q, *J* = 6.2 Hz, 1H), 3.71 (s, 3H), 3.13 – 3.02 (m, 2H), 1.42 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 172.3, 155.0, 136.4, 136.3, 135.6, 129.4, 126.3, 113.6, 79.9, 54.3, 52.2, 38.0, 28.3.

(1-cyclopropylvinyl)benzene (2t)

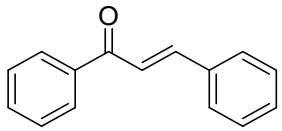


Following the method for the synthesis of **2t**, the title compound was obtained as a colorless liquid in 98% (713 mg) yield. The spectral data were in accordance with those reported in the literature.[2]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.64 – 7.60 (m, 2H), 7.36 (dd, *J* = 8.2, 6.8 Hz, 2H), 7.32 – 7.28 (m, 1H), 5.29 (s, 1H), 4.95 (s, 1H), 1.70 – 1.65 (m, 1H), 0.87 – 0.84 (m, 2H), 0.63 – 0.60 (m, 2H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 149.4, 141.6, 128.1, 127.4, 126.1, 109.0, 15.6, 6.7.

(*E*)-chalcone (3a)

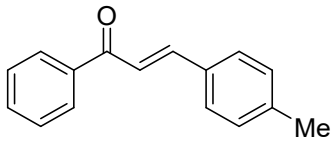


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow solid in 88% (36.5 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.05 – 8.02 (m, 2H), 7.82 (d, *J* = 15.7 Hz, 1H), 7.65 (dd, *J* = 6.4, 3.2 Hz, 2H), 7.61 – 7.57 (m, 1H), 7.56 – 7.50 (m, 3H), 7.45 – 7.40 (m, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.5, 144.8, 138.1, 134.8, 132.8, 130.5, 128.9, 128.6, 128.5, 128.4, 122.1.

(*E*)-1-phenyl-3-(*p*-tolyl)prop-2-en-1-one (3b)

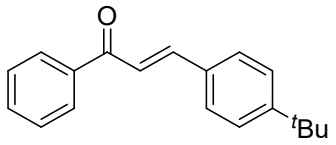


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow solid in 78% (34.7 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.04 – 8.00 (m, 2H), 7.80 (d, *J* = 15.7 Hz, 1H), 7.56 (dd, *J* = 16.0, 7.8 Hz, 3H), 7.53 – 7.47 (m, 3H), 7.23 (d, *J* = 7.8 Hz, 2H), 2.40 (s, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.7, 144.9, 141.1, 138.3, 132.6, 132.1, 129.7, 128.6, 128.5, 128.5, 121.1, 21.5.

(*E*)-3-(4-(*tert*-butyl)phenyl)-1-phenylprop-2-en-1-one (3c)

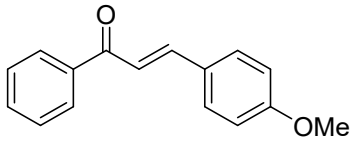


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow solid in 74% (39.1 mg) yield. The spectral data were in accordance with those reported in the literature.[5]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.03 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.82 (d, *J* = 15.7 Hz, 1H), 7.59 (dd, *J* = 12.8, 7.9 Hz, 3H), 7.53 – 7.49 (m, 3H), 7.45 (d, *J* = 8.4 Hz, 2H), 1.35 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.7, 154.2, 144.8, 138.3, 132.6, 132.1, 128.6, 128.4, 128.3, 125.9, 121.3, 34.9, 31.1.

(*E*)-3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one (3d)

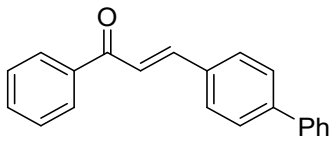


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 72% (34.3 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 7.0 Hz, 2H), 7.79 (d, *J* = 15.6 Hz, 1H), 7.61 – 7.55 (m, 3H), 7.50 (t, *J* = 7.7 Hz, 2H), 7.42 (d, *J* = 15.7 Hz, 1H), 6.94 (d, *J* = 8.8 Hz, 2H), 3.85 (s, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.5, 161.6, 144.7, 138.5, 132.5, 130.2, 128.5, 128.4, 127.6, 119.7, 114.4, 55.4.

(*E*)-3-([1,1'-biphenyl]-4-yl)-1-phenylprop-2-en-1-one (3e)

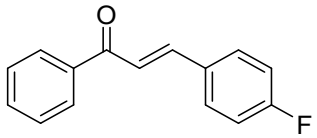


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow solid in 72% (40.9 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.06 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.88 (d, *J* = 15.7 Hz, 1H), 7.73 (d, *J* = 8.3 Hz, 2H), 7.68 – 7.63 (m, 4H), 7.62 – 7.58 (m, 2H), 7.53 (t, *J* = 7.6 Hz, 2H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.41 – 7.38 (m, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.4, 144.3, 143.3, 140.1, 138.2, 133.8, 132.7, 128.9, 128.9, 128.6, 128.5, 127.9, 127.5, 127.0, 121.8.

(*E*)-3-(4-Fluorophenyl)-1-phenylprop-2-en-1-one (3f)



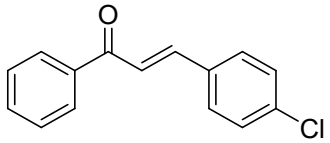
Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 73% (33.0 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.03 – 8.00 (m, 2H), 7.77 (d, *J* = 15.7 Hz, 1H), 7.65 – 7.61 (m, 2H), 7.60 – 7.57 (m, 1H), 7.50 (t, *J* = 7.7 Hz, 2H), 7.46 (d, *J* = 15.7 Hz, 1H), 7.10 (t, *J* = 8.6 Hz, 2H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.2, 164.0(d, $J^1_{C\text{-}F}$ = 251.868), 143.4, 138.1, 132.8, 131.1, 130.3 (d, $J^3_{C\text{-}F}$ = 8.758), 128.6, 128.4, 121.7(d, $J^4_{C\text{-}F}$ = 2.265), 116.1 (d, $J^2_{C\text{-}F}$ = 21.895).

^{19}F NMR (565 MHz, Chloroform-*d*) δ -107.57.

(*E*)-3-(4-chlorophenyl)-1-phenylprop-2-en-1-one (3g)

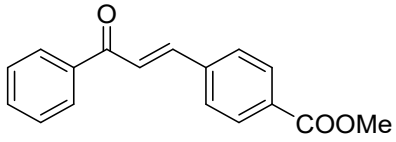


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 74% (35.9 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.04 – 7.99 (m, 2H), 7.76 (d, *J* = 15.7 Hz, 1H), 7.61 – 7.55 (m, 3H), 7.53 – 7.49 (m, 3H), 7.39 (d, *J* = 8.5 Hz, 2H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.2, 143.3, 138.0, 136.4, 133.4, 132.9, 129.6, 129.2, 128.6, 128.5, 122.4.

methyl (*E*)-4-(3-oxo-3-phenylprop-1-en-1-yl)benzoate (3h)

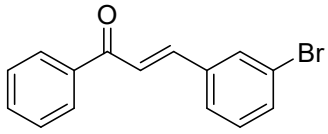


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow solid in 47% (25.0 mg) yield. The spectral data were in accordance with those reported in the literature.[6]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.07 (d, *J* = 8.3 Hz, 2H), 8.04 – 8.01 (m, 2H), 7.80 (d, *J* = 15.7 Hz, 1H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.62 – 7.58 (m, 2H), 7.51 (t, *J* = 7.7 Hz, 2H), 3.93 (s, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.1, 166.4, 143.2, 139.1, 137.8, 133.0, 131.5, 130.1, 128.7, 128.5, 128.2, 124.1, 52.3.

(*E*)-3-(3-bromophenyl)-1-phenylprop-2-en-1-one (3i)

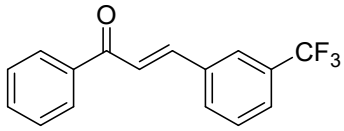


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 60% (34.3 mg) yield. The spectral data were in accordance with those reported in the literature.[7]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.04 – 8.00 (m, 2H), 7.79 (s, 1H), 7.72 (d, *J* = 15.7 Hz, 1H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.55 – 7.50 (m, 5H), 7.29 (t, *J* = 7.8 Hz, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.0, 142.9, 137.9, 137.0, 133.2, 133.0, 130.8, 130.4, 128.7, 128.5, 127.2, 123.2, 123.1.

(*E*)-1-phenyl-3-(3-(trifluoromethyl)phenyl)prop-2-en-1-one (3j)



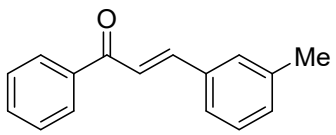
Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 73% (40.3 mg) yield. The spectral data were in accordance with those reported in the literature.[8]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.04 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.90 – 7.79 (m, 3H), 7.66 (d, *J* = 8.9 Hz, 1H), 7.62 – 7.51 (m, 5H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.0, 142.8, 137.8, 135.7, 133.1, 131.6, 131.5 (q, $J^2_{C\text{-}F}$ = 32.314), 128.7, 128.5, 126.8 (q, $J^4_{C\text{-}F}$ = 3.926), 124.5(q, $J^3_{C\text{-}F}$ = 4.530), 123.8(q, $J^1_{C\text{-}F}$ = 272.706), 123.6.

^{19}F NMR (565 MHz, Chloroform-*d*) δ -62.83.

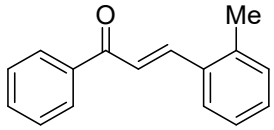
(*E*)-1-phenyl-3-(*m*-tolyl)prop-2-en-1-one (3k)



Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow solid in 84% (37.4 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

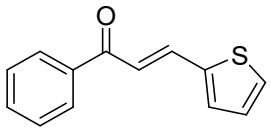
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.03 (d, *J* = 8.3 Hz, 2H), 7.80 (d, *J* = 15.7 Hz, 1H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.55 – 7.50 (m, 3H), 7.46 (d, *J* = 7.8 Hz, 2H), 7.32 (t, *J* = 7.9 Hz, 1H), 7.24 (d, *J* = 7.6 Hz, 1H), 2.40 (s, 3H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.6, 145.0, 138.6, 138.2, 134.8, 132.7, 131.4, 129.0, 128.8, 128.6, 128.5, 125.7, 121.8, 21.3.

(*E*)-1-phenyl-3-(*o*-tolyl)prop-2-en-1-one (3l)



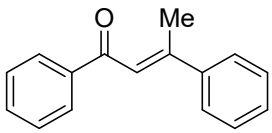
Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow solid in 67% (29.8 mg) yield. The spectral data were in accordance with those reported in the literature.[4]
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.13 (d, *J* = 15.5 Hz, 1H), 8.04 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.71 (d, *J* = 7.8 Hz, 1H), 7.61 – 7.57 (m, 1H), 7.52 (t, *J* = 7.7 Hz, 2H), 7.47 (d, *J* = 15.5 Hz, 1H), 7.32 (td, *J* = 7.4, 1.4 Hz, 1H), 7.28 – 7.23 (m, 2H), 2.49 (s, 3H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.4, 142.4, 138.3, 138.2, 133.9, 132.8, 130.9, 130.2, 128.6, 128.5, 126.4, 126.3, 123.1, 19.8.

(*E*)-1-phenyl-3-(thiophen-2-yl)prop-2-en-1-one (3m)



Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 35% (15.0 mg) yield. The spectral data were in accordance with those reported in the literature.[4]
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.01 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.95 (d, *J* = 15.3 Hz, 1H), 7.60 – 7.56 (m, 1H), 7.50 (t, *J* = 7.7 Hz, 2H), 7.42 (d, *J* = 5.0 Hz, 1H), 7.36 (d, *J* = 3.6 Hz, 1H), 7.34 (d, *J* = 15.3 Hz, 1H), 7.09 (dd, *J* = 5.1, 3.6 Hz, 1H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 189.9, 140.4, 138.1, 137.3, 132.8, 132.1, 128.8, 128.7, 128.4, 128.4, 120.8.

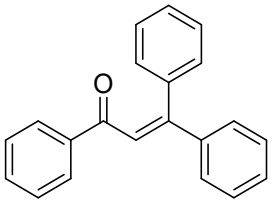
(*E*)-1,3-diphenylbut-2-en-1-one (3n)



Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 49% (21.8 mg) yield. The spectral data were in accordance with those reported in the literature.[9]
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.03 – 7.98 (m, 2H), 7.59 – 7.54 (m, 3H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.45 – 7.39 (m, 3H), 7.18 (d, *J* = 1.4 Hz, 1H), 2.61 (d, *J* = 1.3 Hz, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 191.9, 155.1, 142.8, 139.3, 132.5, 129.1, 128.6, 128.5, 128.3, 126.5, 122.1, 18.9.

1,3,3-triphenylprop-2-en-1-one (3o)

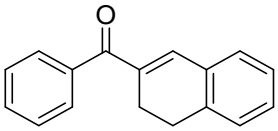


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 73% (41.5 mg) yield. The spectral data were in accordance with those reported in the literature.[10]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.93 (d, *J* = 8.3 Hz, 2H), 7.51 – 7.48 (m, 1H), 7.40 (dt, *J* = 12.5, 5.6 Hz, 7H), 7.29 (d, *J* = 6.7 Hz, 3H), 7.21 (dt, *J* = 7.8, 1.9 Hz, 2H), 7.14 (d, *J* = 1.5 Hz, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.7, 154.7, 141.3, 139.0, 138.2, 132.6, 129.7, 129.3, 128.7, 128.5, 128.4, 128.3, 128.0, 124.0.

(3,4-Dihydronaphthalen-2-yl)(phenyl)methanone (3p)

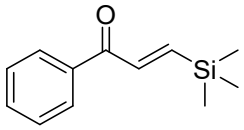


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 76% (35.6 mg) yield. The spectral data were in accordance with those reported in the literature.[11]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.78 – 7.73 (m, 2H), 7.58 – 7.55 (m, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.28 (td, *J* = 7.4, 1.4 Hz, 1H), 7.24 – 7.19 (m, 2H), 7.14 (d, *J* = 7.5 Hz, 2H), 2.96 (t, *J* = 8.2 Hz, 2H), 2.78 (t, *J* = 8.6 Hz, 2H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 197.4, 140.3, 138.3, 137.4, 137.3, 132.5, 131.6, 129.9, 129.1, 128.8, 128.2, 127.8, 126.7, 27.5, 22.7.

(*E*)-1-phenyl-3-(trimethylsilyl)prop-2-en-1-one (3q)

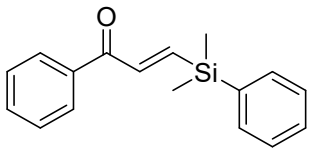


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a yellow oil in 56% (22.9 mg) yield. The spectral data were in accordance with those reported in the literature.[12]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.94 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.58 – 7.55 (m, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.33 – 7.23 (m, 2H), 0.20 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.6, 149.7, 138.0, 137.5, 132.7, 128.8, 128.5, -1.8.

(*E*)-3-(dimethyl(phenyl)silyl)-1-phenylprop-2-en-1-one (3r)

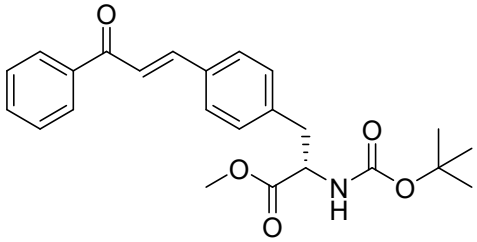


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a yellow oil in 60% (32.0 mg) yield. The spectral data were in accordance with those reported in the literature.[12]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.93 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.58 – 7.55 (m, 3H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.46 – 7.36 (m, 5H), 7.31 (d, *J* = 18.7 Hz, 1H), 0.50 (s, 6H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.3, 147.4, 139.4, 137.4, 136.5, 133.9, 132.8, 129.5, 128.8, 128.5, 128.0, -3.0.

Methyl (*S,E*)-2-((*tert*-butoxycarbonyl)amino)-3-(4-(3-oxo-3-phenylprop-1-en-1-yl)phenyl)propanoate (3s)



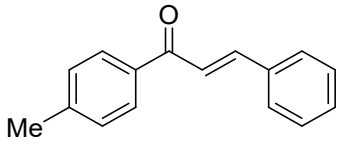
Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 50 % (41.0 mg) yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 7.9 Hz, 2H), 7.78 (d, *J* = 15.7 Hz, 1H), 7.57 (d, *J* = 8.0 Hz, 3H), 7.52 – 7.48 (m, 3H), 7.19 (d, *J* = 7.9 Hz, 2H), 5.05 (d, *J* = 6.2 Hz, 1H), 4.61 (q, *J* = 6.8 Hz, 1H), 3.72 (s, 3H), 3.12 (ddd, *J* = 61.0, 13.8, 6.1 Hz, 2H), 1.41 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.5, 172.1, 155.0, 144.4, 138.9, 138.2, 133.6, 132.7, 130.0, 129.9, 128.6, 128.4, 121.8, 80.0, 54.2, 52.3, 38.3, 28.2.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for ($C_{24}H_{27}NO_5Na)^+$, 432.1781; found: 432.1760.

(*E*)-3-phenyl-1-(p-tolyl)prop-2-en-1-one (4a)

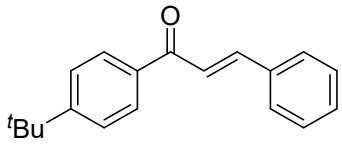


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow solid in 79% (33.0 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.2 Hz, 2H), 7.81 (d, *J* = 15.7 Hz, 1H), 7.65 (dd, *J* = 7.3, 2.2 Hz, 2H), 7.55 (d, *J* = 15.7 Hz, 1H), 7.42 (dd, *J* = 5.0, 2.3 Hz, 3H), 7.31 (d, *J* = 7.6 Hz, 2H), 2.44 (s, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 189.9, 144.3, 143.6, 135.6, 134.9, 130.4, 129.3, 128.9, 128.6, 128.3, 122.0, 21.6.

(*E*)-1-(4-(*tert*-butyl)phenyl)-3-phenylprop-2-en-1-one (4b)

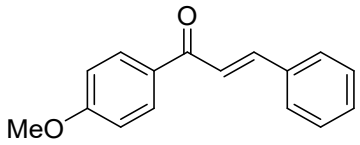


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow solid in 67% (35.3 mg) yield. The spectral data were in accordance with those reported in the literature.[13]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.5 Hz, 2H), 7.82 (d, *J* = 15.7 Hz, 1H), 7.68 – 7.64 (m, 2H), 7.58 – 7.51 (m, 3H), 7.44 – 7.41 (m, 3H), 1.37 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.0, 156.5, 144.3, 135.5, 135.0, 130.4, 128.9, 128.5, 128.4, 125.6, 122.1, 35.1, 31.1.

(*E*)-1-(4-methoxyphenyl)-3-phenylprop-2-en-1-one (4c)

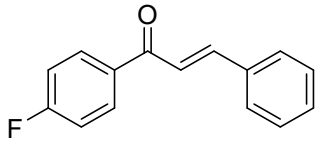


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 72% (34.3 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.05 (d, *J* = 8.9 Hz, 2H), 7.81 (d, *J* = 15.7 Hz, 1H), 7.64 (dd, *J* = 7.4, 2.1 Hz, 2H), 7.55 (d, *J* = 15.6 Hz, 1H), 7.43 – 7.38 (m, 3H), 6.98 (d, *J* = 8.9 Hz, 2H), 3.89 (s, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 188.7, 163.4, 143.9, 135.0, 131.0, 130.8, 130.3, 128.9, 128.3, 121.8, 113.8, 55.4.

(*E*)-1-(4-fluorophenyl)-3-phenylprop-2-en-1-one (4d)



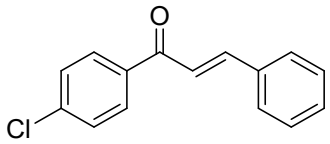
Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 79% (35.6 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.08 – 8.03 (m, 2H), 7.82 (d, *J* = 15.7 Hz, 1H), 7.64 (dd, *J* = 6.7, 2.8 Hz, 2H), 7.51 (d, *J* = 15.7 Hz, 1H), 7.46 – 7.38 (m, 3H), 7.18 (t, *J* = 8.6 Hz, 2H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 188.8, 165.6($J^1_{C\text{-}F}$ = 254.586), 145.0, 134.7, 134.5($J^4_{C\text{-}F}$ = 2.869), 131.1($J^4_{C\text{-}F}$ = 9.211), 130.6, 128.9, 128.4, 121.6, 115.0($J^2_{C\text{-}F}$ = 21.744).

^{19}F NMR (565 MHz, Chloroform-*d*) δ -105.54.

(*E*)-1-(4-chlorophenyl)-3-phenylprop-2-en-1-one (4e)

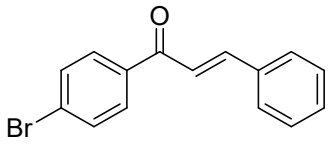


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 56% (27.3 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 8.5 Hz, 2H), 7.82 (d, *J* = 15.6 Hz, 1H), 7.64 (dd, *J* = 6.4, 3.2 Hz, 2H), 7.52 – 7.45 (m, 3H), 7.44 – 7.40 (m, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 189.1, 145.3, 139.2, 136.5, 134.7, 130.7, 129.9, 129.0, 128.9, 128.5, 121.5.

(*E*)-1-(4-bromophenyl)-3-phenylprop-2-en-1-one (4f)

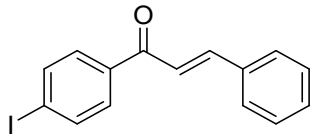


Following the general procedure, solvent changed to THF/DCE (1:1), the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow solid in 72% (41.5 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 8.5 Hz, 2H), 7.82 (d, *J* = 15.7 Hz, 1H), 7.65 (d, *J* = 8.5 Hz, 4H), 7.48 (d, *J* = 15.7 Hz, 1H), 7.45 – 7.41 (m, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 189.4, 145.4, 136.9, 134.7, 131.9, 130.7, 130.0, 129.0, 128.5, 127.9, 121.4.

(*E*)-1-(4-iodophenyl)-3-phenylprop-2-en-1-one (4g)

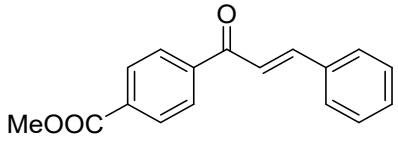


Following the general procedure, solvent changed to THF/DCE (1:1), the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=10:1) as a light yellow solid in 46% (30.7 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.87 (d, *J* = 8.4 Hz, 2H), 7.81 (d, *J* = 15.7 Hz, 1H), 7.74 – 7.72 (m, 2H), 7.65 – 7.63 (m, 2H), 7.46 (d, *J* = 15.7 Hz, 1H), 7.44 – 7.41 (m, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 189.7, 145.5, 138.0, 137.5, 134.7, 130.8, 129.9, 129.0, 128.6, 121.5, 100.6.

methyl 4-cinnamoylbenzoate (4h)

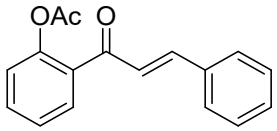


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=10:1) an a light yellow oil in 45% (24.1 mg) yield. The spectral data were in accordance with those reported in the literature.[13]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.16 (d, *J* = 8.4 Hz, 2H), 8.05 (d, *J* = 8.4 Hz, 2H), 7.82 (d, *J* = 15.7 Hz, 1H), 7.65 (dd, *J* = 6.8, 2.8 Hz, 2H), 7.51 (d, *J* = 15.7 Hz, 1H), 7.45 – 7.41 (m, 3H), 3.96 (s, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.1, 166.3, 145.8, 141.6, 134.6, 133.5, 130.8, 129.8, 129.0, 128.6, 128.3, 121.8, 52.4.

(*E*)-2-cinnamoylphenyl acetate (4i)

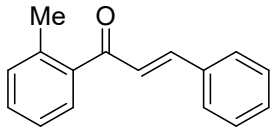


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=10:1) as a light yellow oil in 41% (22.0 mg) yield. The spectral data were in accordance with those reported in the literature.[14]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.70 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.60 – 7.53 (m, 4H), 7.41 (hept, *J* = 3.8 Hz, 3H), 7.36 (td, *J* = 7.6, 1.2 Hz, 1H), 7.21 – 7.15 (m, 2H), 2.24 (s, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 191.6, 169.4, 148.7, 145.5, 134.5, 132.5, 132.2, 130.7, 129.8, 129.0, 128.4, 126.0, 125.3, 123.5, 21.0.

(*E*)-3-phenyl-1-(*o*-tolyl)prop-2-en-1-one (4j)

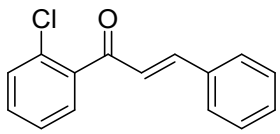


Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow solid in 84% (37.5 mg) yield. The spectral data were in accordance with those reported in the literature.[4]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.57 (dd, *J* = 6.8, 2.9 Hz, 2H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.48 (d, *J* = 16.1 Hz, 1H), 7.42 – 7.37 (m, 4H), 7.29 (d, *J* = 7.5 Hz, 2H), 7.15 (d, *J* = 16.1 Hz, 1H), 2.46 (s, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 196.5, 145.9, 139.0, 136.9, 134.6, 131.3, 130.6, 130.4, 128.9, 128.4, 128.0, 126.7, 125.4, 20.2.

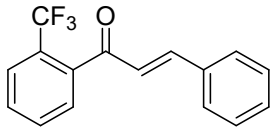
(*E*)-1-(2-chlorophenyl)-3-phenylprop-2-en-1-one (4k)



Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 79% (38.3 mg) yield. The spectral data were in accordance with those reported in the literature.[15]

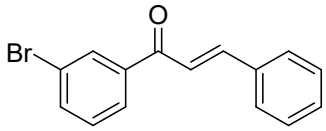
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.56 (dd, *J* = 7.4, 2.3 Hz, 2H), 7.50 – 7.45 (m, 3H), 7.44 – 7.39 (m, 4H), 7.36 (td, *J* = 7.4, 1.3 Hz, 1H), 7.14 (d, *J* = 16.1 Hz, 1H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 193.8, 146.3, 139.0, 134.4, 131.4, 131.2, 130.8, 130.2, 129.3, 128.9, 128.5, 126.8, 126.2.

(*E*)-3-phenyl-1-(2-(trifluoromethyl)phenyl)prop-2-en-1-one (4l)



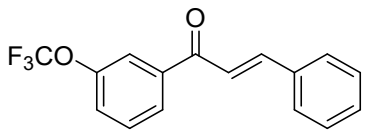
Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a yellow oil in 96% (53.0 mg) yield. The spectral data were in accordance with those reported in the literature.[16]
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.77 (d, *J* = 9.2 Hz, 1H), 7.65 (t, *J* = 6.9 Hz, 1H), 7.60 (t, *J* = 7.8 Hz, 1H), 7.53 (dd, *J* = 7.7, 1.9 Hz, 2H), 7.47 (d, *J* = 6.9 Hz, 1H), 7.43 – 7.37 (m, 3H), 7.30 (d, *J* = 16.2 Hz, 1H), 7.05 (d, *J* = 16.3 Hz, 1H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 195.1, 147.8, 138.8, 134.1, 131.6, 131.0, 129.8, 129.0, 128.5, 128.1, 127.8 (q, $J^2_{C\text{-}F}$ =32.3 Hz), 126.8, 126.7 (q, $J^3_{C\text{-}F}$ =4.8 Hz), 123.6 (q, $J^1_{C\text{-}F}$ = 273.9 Hz).

(*E*)-1-(3-bromophenyl)-3-phenylprop-2-en-1-one (4m)



Following the general procedure, 3-bromobenzoyl chloride (0.4 mmol) and stirred for 12 h, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a yellow oil in 53% (30.5 mg) yield. The spectral data were in accordance with those reported in the literature.[4]
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.14 (t, *J* = 1.8 Hz, 1H), 7.95 – 7.93 (m, 1H), 7.83 (d, *J* = 15.7 Hz, 1H), 7.71 (ddd, *J* = 7.9, 2.0, 1.1 Hz, 1H), 7.66 – 7.64 (m, 2H), 7.48 – 7.43 (m, 4H), 7.39 (t, *J* = 7.8 Hz, 1H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 189.0, 145.7, 140.0, 135.6, 134.6, 131.5, 130.8, 130.2, 129.0, 128.6, 127.0, 122.9, 121.4.

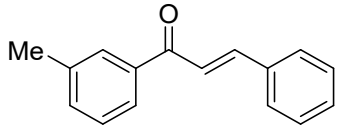
(*E*)-3-phenyl-1-(3-(trifluoromethoxy)phenyl)prop-2-en-1-one (4n)



Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 47% (27.5 mg) yield. The spectral data were in accordance with those reported in the literature.[17]

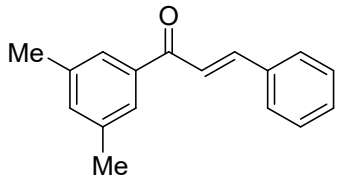
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.95 (dt, *J* = 7.7, 1.3 Hz, 1H), 7.84 (d, *J* = 15.8 Hz, 2H), 7.66 (dd, *J* = 6.6, 3.1 Hz, 2H), 7.55 (t, *J* = 8.0 Hz, 1H), 7.48 (d, *J* = 15.7 Hz, 1H), 7.44 (q, *J* = 3.6 Hz, 4H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 188.9, 149.5, 145.9, 140.0, 134.5, 130.9, 130.1, 129.0, 128.6, 126.7, 125.0, 121.3, 120.9, 120.2 (q, J_{C-F} = 258.2).

(*E*)-3-phenyl-1-(*o*-tolyl)prop-2-en-1-one (4o)



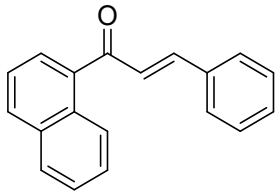
Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow solid in 81% (36.1 mg) yield. The spectral data were in accordance with those reported in the literature.[4]
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.85 – 7.79 (m, 3H), 7.65 (dd, *J* = 6.5, 3.1 Hz, 2H), 7.54 (d, *J* = 15.7 Hz, 1H), 7.44 – 7.38 (m, 5H), 2.45 (s, 3H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.7, 144.6, 138.4, 138.2, 134.9, 133.5, 130.4, 129.0, 128.9, 128.4, 128.4, 125.7, 122.2, 21.4.

(*E*)-1-(3,5-dimethylphenyl)-3-phenylprop-2-en-1-one (4p)



Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow solid in 81% (37.6 mg) yield. The spectral data were in accordance with those reported in the literature.[18]
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.81 (d, *J* = 15.7 Hz, 1H), 7.68 – 7.63 (m, 4H), 7.53 (d, *J* = 15.7 Hz, 1H), 7.45 – 7.40 (m, 3H), 7.22 (s, 1H), 2.41 (s, 6H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.8, 144.4, 138.3, 138.2, 134.9, 134.4, 130.4, 128.9, 128.4, 126.2, 122.3, 21.2.

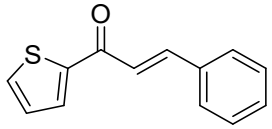
(*E*)-1-(naphthalen-1-yl)-3-phenylprop-2-en-1-one (4q)



Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a yellow oil in 78% (40.3 mg) yield. The spectral data were in accordance with those reported in the literature.[19]

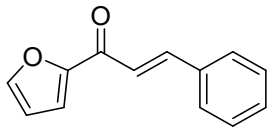
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.36 (d, *J* = 8.5 Hz, 1H), 8.01 (d, *J* = 8.2 Hz, 1H), 7.94 – 7.91 (m, 1H), 7.63 (d, *J* = 16.1 Hz, 1H), 7.59 – 7.53 (m, 5H), 7.41 (dd, *J* = 5.1, 2.0 Hz, 3H), 7.33 (d, *J* = 16.0 Hz, 1H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 195.7, 145.9, 137.0, 134.6, 133.8, 131.6, 130.7, 130.5, 128.9, 128.5, 128.4, 127.4, 127.1, 127.1, 126.4, 125.6, 124.5.

(*E*)-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (4r)



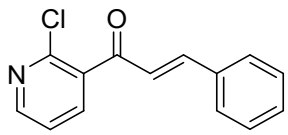
Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a yellow oil in 73% (31.4 mg) yield. The spectral data were in accordance with those reported in the literature.[4]
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.89 – 7.84 (m, 2H), 7.68 (dd, *J* = 4.9, 1.1 Hz, 1H), 7.64 (dd, *J* = 6.4, 3.2 Hz, 2H), 7.45 – 7.41 (m, 4H), 7.19 (dd, *J* = 4.9, 3.8 Hz, 1H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 182.0, 145.5, 144.0, 134.6, 133.9, 131.8, 130.6, 128.9, 128.4, 128.2, 121.6.

(E)-1-(furan-2-yl)-3-phenylprop-2-en-1-one (4s)



Following the general procedure, the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a yellow oil in 67% (26.5 mg) yield. The spectral data were in accordance with those reported in the literature.[4]
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.88 (d, *J* = 15.8 Hz, 1H), 7.64 (dd, *J* = 6.6, 2.2 Hz, 3H), 7.45 (d, *J* = 15.8 Hz, 1H), 7.41 (dd, *J* = 5.0, 1.9 Hz, 3H), 7.33 (d, *J* = 2.8 Hz, 1H), 6.59 (dd, *J* = 3.6, 1.7 Hz, 1H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 178.0, 153.6, 146.5, 143.9, 134.7, 130.6, 128.9, 128.5, 121.1, 117.5, 112.5.

(*E*)-1-(2-chloropyridin-3-yl)-3-phenylprop-2-en-1-one (4t)



Following the general procedure, 2-chloronicotinoyl chloride (0.40 mmol) and stirred for 12 h，the title compound was isolated by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) as a light yellow oil in 40% (19.6 mg) yield. The spectral data were in accordance with those reported in the literature.[20]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.52 (dd, *J* = 4.8, 2.0 Hz, 1H), 7.83 (dd, *J* = 7.5, 2.0 Hz, 1H), 7.57 (dd, *J* = 7.7, 1.9 Hz, 2H), 7.50 (d, *J* = 16.1 Hz, 1H), 7.41 (d, *J* = 7.8 Hz, 3H), 7.37 (dd, *J* = 7.5, 4.8 Hz, 1H), 7.16 (d, *J* = 16.0 Hz, 1H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 191.9, 151.0, 147.8, 146.9, 138.4, 135.4, 134.1, 131.2, 129.1, 128.7, 125.5, 122.5.

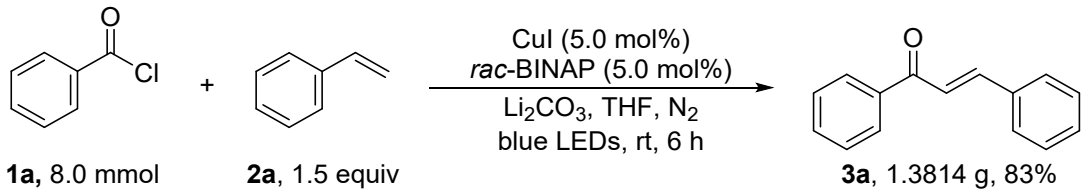
7. Sunlight Experiment

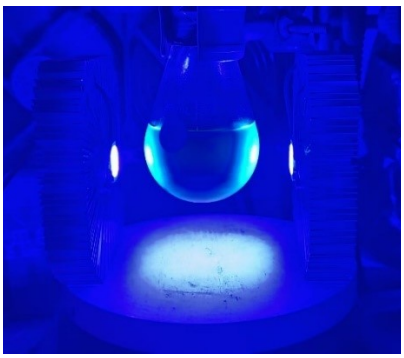
Device of sunlight experiment

The rubber plug in the head is impervious to light, and to ensure that sunlight can be illuminated at midday experiments were conducted utilizing mirrored soft film reflections. The device was located on the south side of Building 2, School of Chemistry and Chemical Engineering, Guangxi University (longitude:108.28 E, latitude:22.83 N)

In a glovebox, a quartz test tube (20*165 mm, 30 mL) equipped with a magnetic stir bar was charged with CuI (1.9 mg, 5 mol%), *rac*-BINAP (6.2 mg, 5 mol%), aroyl chlorides **1** (0.30 mmol), alkenes **2** (0.20 mmol), Li_2CO_3 (0.20 mmol), and THF (1.0 mL) before being sealed with a rubber stopper. Then, the test tube was then placed approximately 1.5 cm from the reflector lens. After 6 h (August, 10 a.m.-4 p.m., the outside temperature is about 30 ^{o}C), DBU (0.6 mmol) was added to the reaction mixture in air and stirred for 30 minutes at rt without irradiation. Finally, the reaction mixture was concentrated under reduced pressure and the crude product was purified by silica gel plate to give the corresponding products **3a** (27.9 mg, 67%), **3d** (32.0 mg, 67%), **3f** (22.0 mg, 49%), **4a** (24.8 mg, 56%), and **4j** (50.5 mg, 90%).

8. Gram-scale Experiments



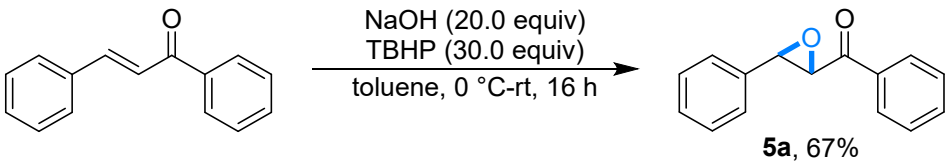


Device of gram-scale experiment

In a glovebox, a cigar-shaped vial (50 mL) equipped with a magnetic stir bar was charged with CuI (76.0 mg, 5.00 mol%), *rac*-BINAP (248 mg, 5.00 mol%), aroyl chloride **1a** (1.68 g, 12.0 mmol, 1.50 equiv), alkene **2a** (0.832 g, 8.0 mmol), Li_2CO_3 (584 mg, 8.00 mmol), and THF (30.0 mL) before being sealed with a rubber stopper. Then, the vial was placed in the photoreactor about 1.0 cm away from 10 W blue LEDs light irradiation with a fan to maintain the temperature at rt. After 24 h, DBU (3.65 g, 24 mmol, 3.0 equiv) was added to the reaction mixture in air and stirred for 30 minutes at rt without irradiation. Finally, the reaction mixture was concentrated under reduced pressure and the crude product was purified by column chromatographye on silica gel eluting with petroleum ether/ethyl acetate=20:1 to give products as a yellow solid (1.38 g, 83% yield).

9. Procedures for Diverse Derivatizations of 3a

phenyl((*2S*,*3R*)-3-phenyloxiran-2-yl)methanone (5a)

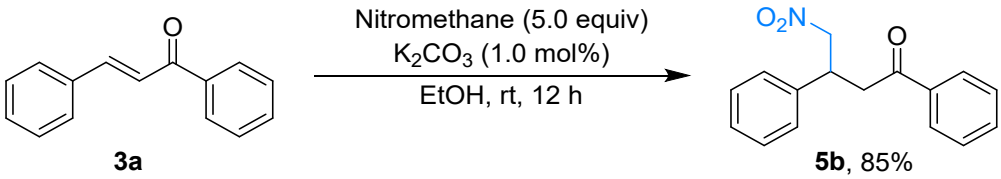


Phenyl((*2S*,*3R*)-3-phenyloxiran-2-yl)methanone was synthesized according to a literature procedure.[16] A dry round-bottomed flask equipped with a magnetic stirring bar was charged with chalcone (42 mg, 0.20 mmol), dissolved in toluene (2.0 mL). The mixture was allowed to 0 °C stirred for 10 min. Then, TBHP (0.80 mL, 70 wt%, 30 equiv) was added dropwise and the mixture was stirred at rt for 16 h until the chalcone was fully consumed. The reaction mixture was concentrated in vacuum and the crude product was subjected to column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1, v/v), giving **5a** (30 mg, 67%) as a colorless oil. The spectral data were in accordance with those reported in the literature.[21]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.03 – 7.99 (m, 2H), 7.62 (t, *J* = 7.5 Hz, 1H), 7.49 (t, *J* = 7.8 Hz, 2H), 7.43 – 7.36 (m, 5H), 4.31 (d, *J* = 1.9 Hz, 1H), 4.08 (d, *J* = 1.9 Hz, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 193.0, 135.4, 135.4, 133.9, 129.0, 128.8, 128.7, 128.3, 125.7, 60.9, 59.3.

4-nitro-1,3-diphenylbutan-1-one (5b)



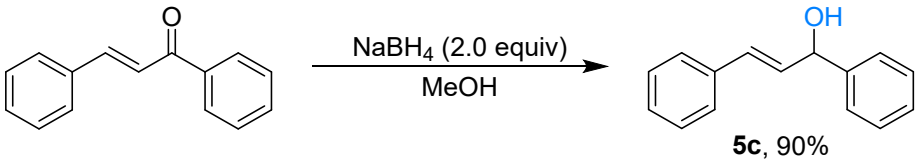
3a **5b**, 85%

4-nitro-1,3-diphenylbutan-1-one was synthesized according to a literature procedure.[22] A dry round-bottomed flask equipped with a magnetic stirring bar was charged with chalcone (416 mg, 2.0 mmol), nitromethane (610 mg, 10 mmol, 5.0 equiv), and K_2CO_3 (2.8 mg, 1.0 mmol%), dissolved in ethanol (2.0 mL). The mixture was stirred for 12 h at rt. A saturated aqueous solution of $NaHCO_3$ was added and the mixture was extracted three times with DCM. Concentrated in vacuum and the crude product was subjected to column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1 to 5/1, v/v), giving **5b** (0.46 g, 85 %) as a white solid. The spectral data were in accordance with those reported in the literature.[22]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.92 (d, *J* = 7.5 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 2H), 7.35 – 7.27 (m, 5H), 4.83 (dd, *J* = 12.5, 6.6 Hz, 1H), 4.69 (dd, *J* = 12.5, 8.0 Hz, 1H), 4.24 (ddd, *J* = 14.2, 7.8, 6.5 Hz, 1H), 3.50 – 3.41 (m, 2H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 196.8, 139.1, 136.3, 133.5, 129.0, 128.7, 128.0, 127.8, 127.4, 79.5, 41.5, 39.2.

(*E*)-1,3-diphenylprop-2-en-1-ol (5c)



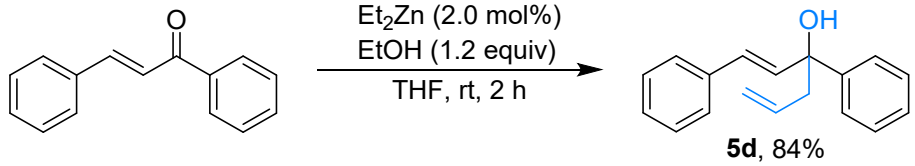
5c, 90%

(*E*)-1,3-diphenylprop-2-en-1-ol was synthesized according to a literature procedure.[23] A dry round-bottomed flask equipped with a magnetic stirring bar was charged with chalcone (208 mg, 1.0 mmol, 1.0 equiv), sodium borohydride (68 mg, 2.0 mmol, 2.0 equiv), THF (3.0 mL) and MeOH (3.0 mL). The resulting solution was stirred at rt for 30 min. The reaction mixture was concentrated in vacuum and the crude product was subjected to column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1, v/v), giving **5c** (189 mg, 90%) as a colorless liquid. The spectral data were in accordance with those reported in the literature.[23]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.47 – 7.43 (m, 2H), 7.41 – 7.37 (m, 4H), 7.34 – 7.30 (m, 3H), 7.28 – 7.24 (m, 1H), 6.70 (dd, *J* = 15.9, 1.3 Hz, 1H), 6.40 (dd, *J* = 15.9, 6.5 Hz, 1H), 5.39 (d, *J* = 6.5 Hz, 1H), 2.25 (s, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 142.7, 136.5, 131.5, 130.5, 128.6, 128.5, 127.8, 127.7, 126.6, 126.3, 75.1.

(*E*)-1,3-diphenylhexa-1,5-dien-3-ol (5d)



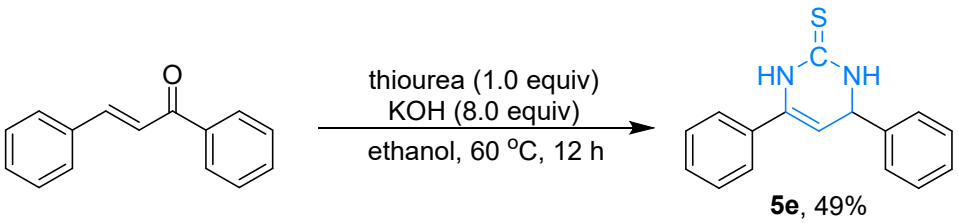
5d, 84%

(*E*)-1,3-diphenylhexa-1,5-dien-3-ol was synthesized according to a literature procedure.[24] In a glovebox, A dry round-bottomed flask equipped with a magnetic stirring bar was charged with diethyl zinc (0.40 mL, 2.0 mol%, 0.02 mmol, 1.0 M in toluene) and ethanol (164 mg, 1.2 equiv, 3.6 mmol), dissolved in THF (0.10 M) before being sealed with a rubber stopper. The mixture was allowed to rt stirred for 10 min. Then, allyl boronic acid pinacol ester (0.64 mL, 3.3 mmol, 1.1 equiv) and chalcone (0.64 g, 3.0 mmol, 1.0 equiv, in 1.5 mL THF) were added dropwise and the mixture was stirred at rt for 2 h. Concentrated in vacuum and the crude product was subjected to column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10/1, v/v), giving **5d** (0.67 g, 84%) as a colorless oil. The spectral data were in accordance with those reported in the literature.[24]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.55 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.43 – 7.37 (m, 4H), 7.35 – 7.23 (m, 4H), 6.69 (d, *J* = 16.0 Hz, 1H), 6.56 (d, *J* = 16.0 Hz, 1H), 5.76 (ddt, *J* = 17.4, 10.2, 7.3 Hz, 1H), 5.27 – 5.20 (m, 2H), 2.84 (qd, *J* = 13.9, 7.3 Hz, 2H), 2.37 (s, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 145.2, 136.7, 135.1, 133.1, 128.5, 128.3, 127.5, 127.0, 126.5, 125.4, 120.1, 75.6, 47.1.

4,6-diphenyl-3,4-dihydropyrimidine-2(1*H*)-thione (5e)

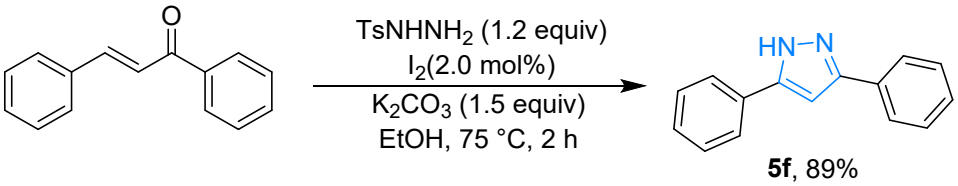


A dry round-bottomed flask equipped with a magnetic stirring bar was charged with chalcone (208 mg, 1.0 mmol, 1.0 equiv), thiourea (91 mg, 1.2 mmol, 1.2 equiv), and potassium hydroxide (0.50 g, 8.0 mmol, 8.0 equiv), dissolved in ethanol (4.0 ml). The mixture was allowed to 60 °C stirred for 12 h. The excess solvent was removed under reduced pressure and the reaction mixture was cooled in an ice bath. The products precipitated out at low temperature, were washed with water, recrystallized from ethanol, giving **5e** (130 mg, 49%) as a white solid. The spectral data were in accordance with those reported in the literature.[25]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 (s, 1H), 7.51 – 7.28 (m, 11H), 5.21 (d, *J* = 40.5 Hz, 2H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 174.8, 142.2, 133.9, 133.1, 129.4, 129.0, 128.8, 128.4, 126.8, 125.2, 100.6, 56.9.

3,5-diphenyl-1*H*-pyrazole (5f)



3,5-diphenyl-1*H*-pyrazole was synthesized according to a literature procedure.[26] A dry round-bottomed flask equipped with a magnetic stirring bar was charged with chalcone (104 mg, 0.50 mmol, 1.0 equiv), $TsNHNH_2$ (112 mg, 0.60 mmol, 1.2 equiv), and iodine (2.5 mg, 2.0 mol%), dissolved in ethanol (2.0 ml). The mixture was allowed to 75 °C stirred for 10 min. Then, K_2CO_3

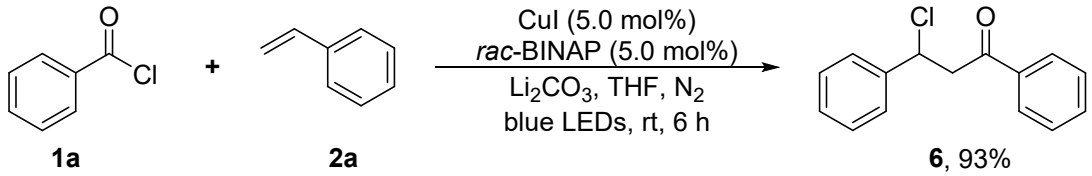
(207 mg, 1.5 mmol, 1.5 equiv) was added and the mixture was stirred for 2 h. The reaction mixture was concentrated in vacuum and the crude product was subjected to column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (5/1, v/v), giving **5f** (98 mg, 89%) as a white solid.[26]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.70 (d, *J* = 7.3 Hz, 4H), 7.35 – 7.28 (m, 6H), 6.81 (s, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 128.8, 128.1, 125.6, 100.0.

10. Mechanistic Studies

β-chloro-ketone intermediates:



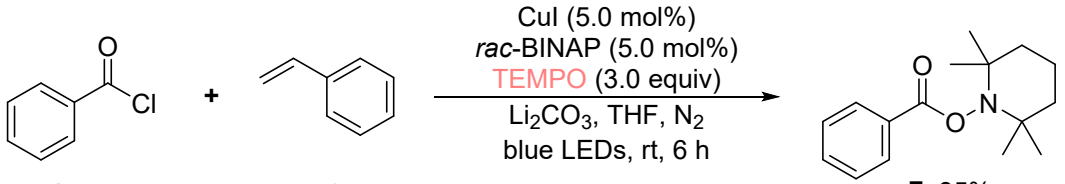
1a **2a** **6**, 93%

In a glovebox, a quartz test tube (20*165 mm, 30 mL) equipped with a magnetic stir bar was charged with CuI (1.9 mg, 5.0 mol%), *rac*-BINAP (6.2 mg, 5.0 mol%), benzoyl chloride (42 mg, 0.30 mmol), styrene (21 mg, 0.20 mmol), Li_2CO_3 (0.20 mmol), and THF (1.0 mL) before being sealed with a rubber stopper. Then, the tube was placed in the photoreactor about 1.5 cm away from 10 W blue LEDs light irradiation with a fan maintain the temperature at rt for 6 h. The reaction mixture was concentrated in vacuum and the crude product was subjected to column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (30/1, v/v), giving **6** (45 mg, 93%) as a white solid. The spectral data were in accordance with those reported in the literature.[27](The column chromatography of the β-chloro-ketones resulted in an impure spectrum due to the facile dehydrochlorination.[27])

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.97 (dd, *J* = 8.4, 1.4 Hz, 2H), 7.60 – 7.57 (m, 1H), 7.52 – 7.45 (m, 4H), 7.38 (t, *J* = 7.5 Hz, 2H), 7.32 (t, *J* = 7.2 Hz, 1H), 5.63 (dd, *J* = 8.1, 5.6 Hz, 1H), 3.97 (dd, *J* = 17.3, 8.1 Hz, 1H), 3.62 (dd, *J* = 17.3, 5.6 Hz, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 195.7, 141.0, 136.4, 133.5, 128.7, 128.7, 128.5, 128.1, 127.0, 57.5, 48.3.

TEMPO inhibition:



1a **2a** **7**, 85% by NMR

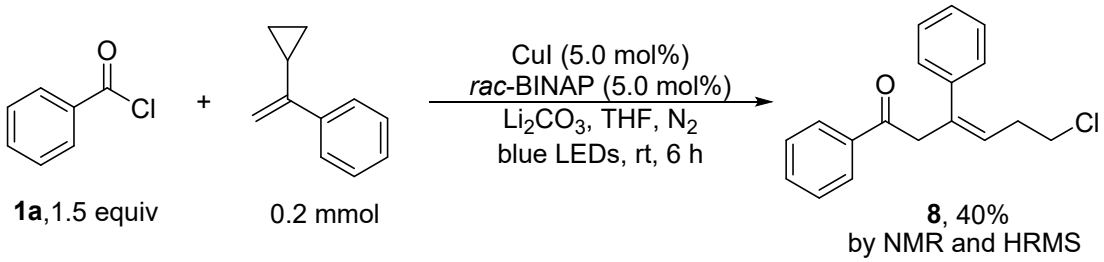
In a glovebox, a quartz test tube (20*165 mm, 30 mL) equipped with a magnetic stir bar was charged with CuI (1.9 mg, 5.0 mol%), *rac*-BINAP (6.2 mg, 5.0 mol%), benzoyl chloride (42 mg, 0.30 mmol), styrene (21 mg, 0.20 mmol), TEMPO (94 mg, 0.60 mmol), Li_2CO_3 (0.20 mmol), and THF (1.0 mL) before being sealed with a rubber stopper. Then, the tube was placed in the photoreactor about 1.5 cm away from 10 W blue LEDs light irradiation with a fan maintain the temperature at rt for 6 h. The reaction mixture was concentrated in vacuum and the crude product

was subjected to column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (20/1, v/v), giving **7** (67 mg, 85%) as a reddish brown solid. The spectral data were in accordance with those reported in the literature.[28]

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.13 (d, *J* = 4.4 Hz, 2H), 7.62 (s, 1H), 7.51 (s, 2H), 1.85 – 1.51 (m, 7H), 1.32 (s, 6H), 1.17 (s, 6H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.0, 132.5, 129.4, 129.2, 128.1, 60.1, 38.7, 31.6, 20.5, 16.7.

Radical clock experiment:



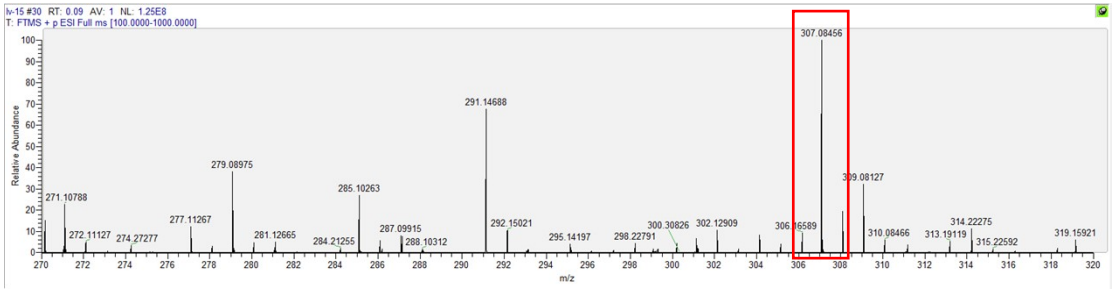
In a glovebox, a quartz test tube (20*165 mm, 30 mL) equipped with a magnetic stir bar was charged with CuI (1.9 mg, 5 mol%), *rac*-BINAP (6.2 mg, 5 mol%), benzoyl chloride (42.0 mg, 0.3 mmol), styrene (20.8 mg, 0.20 mmol), TEMPO (93.6 mg, 0.6 mmol), Li_2CO_3 (0.20 mmol), and THF (1.0 mL) before being sealed with a rubber stopper. Then, the tube was placed in the photoreactor about 1.5 cm away from 10 W blue LEDs light irradiation with a fan maintain the temperature at rt for 6 h. The reaction mixture was concentrated in vacuum and the crude product was subjected to column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (30/1, v/v), giving **8** (23 mg ,40%) as a yellow liquid.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.00 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.61 – 7.57 (m, 1H), 7.48 (t, *J* = 7.8 Hz, 2H), 7.33 – 7.27 (m, 4H), 7.25 – 7.21 (m, 1H), 6.04 (t, *J* = 7.2 Hz, 1H), 4.23 (s, 2H), 3.65 (t, *J* = 6.9 Hz, 2H), 2.65 (q, *J* = 7.0 Hz, 2H).

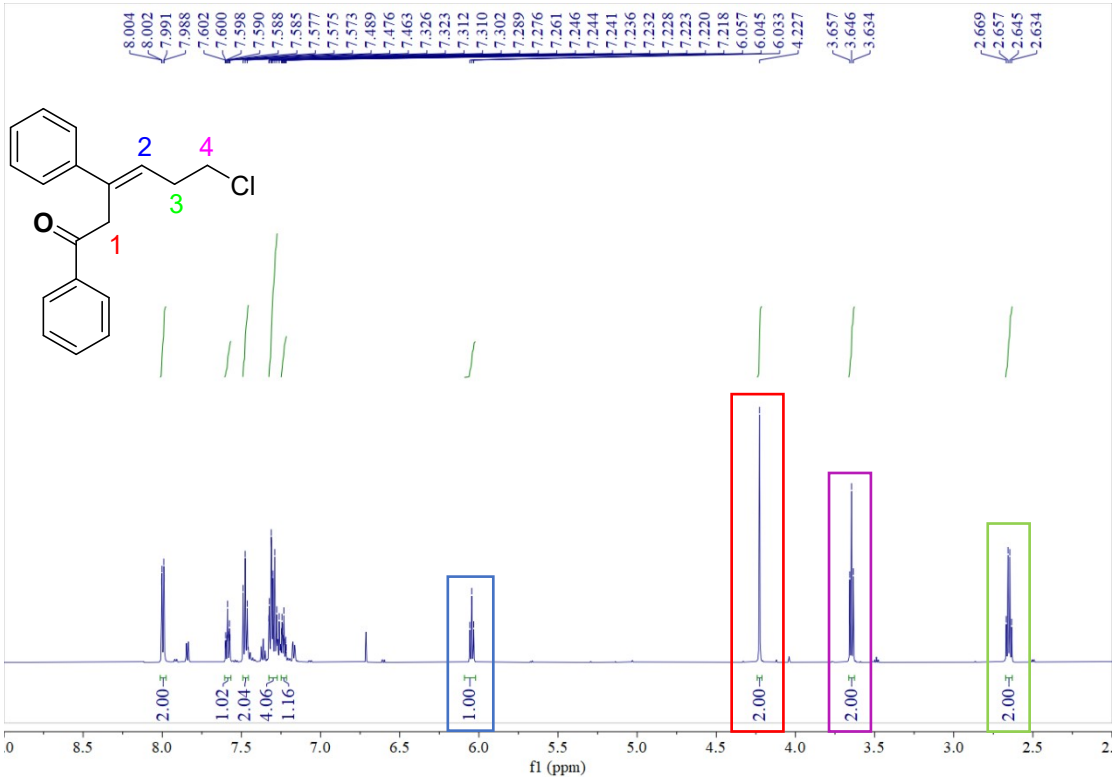
^{13}C NMR (151 MHz, Chloroform-*d*) δ 196.6, 142.3, 136.6, 135.8, 133.3, 128.6, 128.3, 128.1, 127.9, 127.1, 126.0, 43.8, 40.8, 32.5.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $(C_{18}H_{17}ClONa)^+$, 307.0860; found: 307.0846.

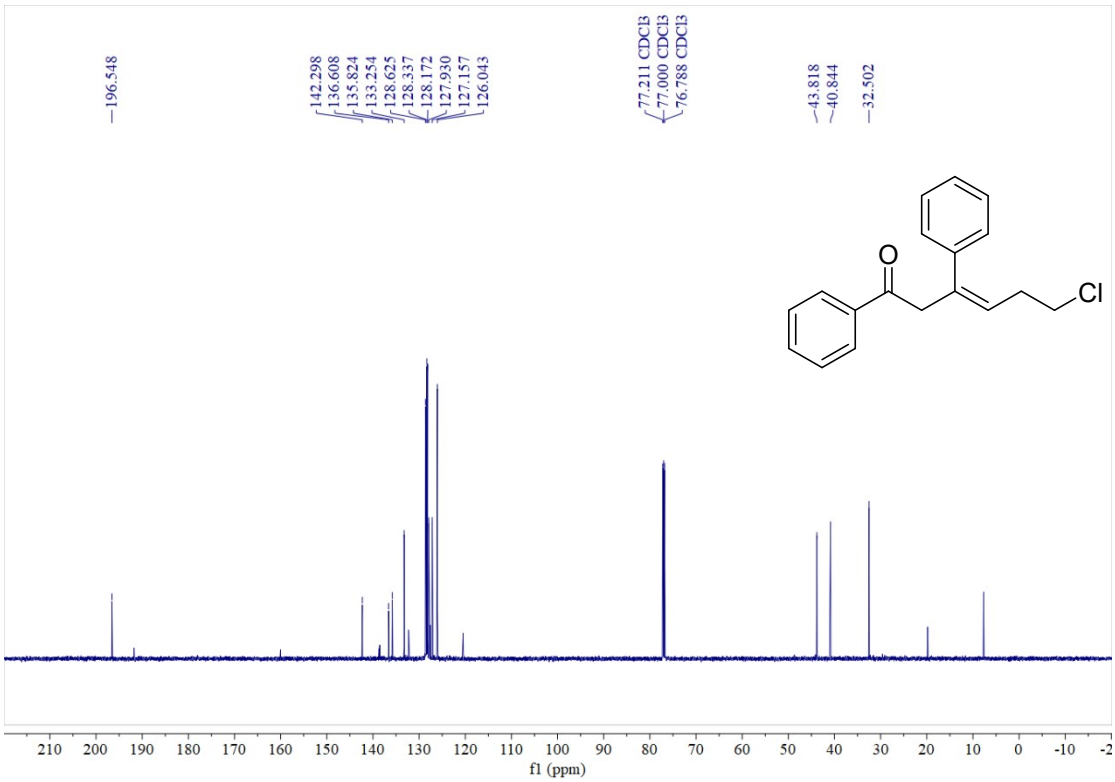
HRMS Spectrum of **8**



^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **8**



^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **8**



11. References

1 W. Xiao, L. Ning, S. Xin, S. Dong, X. Liu, X. Feng, *Angew. Chem. Int. Ed.*, 2022, **61**, e202211596.

2 J. Cao, D. Lv, F. Yu, M.-F. Chiou, Y. Li, H. Bao, *Org. Lett.*, 2021, **23**, 3184−3189.

3 C. Xu, J. Chang, Z. Dong, L. Song, L.-A. Chen, *Org. Chem. Front.*, 2023, **10**, 5592−5600.

4 Q. Jiang, J. Jia, B. Xu, A. Zhao, C.-C. Guo, *J. Org. Chem.*, 2015, **80**, 3586−3596.

5 U. Farid, F. Malmedy, R. Claveau, L. Albers, T. Wirth, *Angew. Chem. Int. Ed.*, 2013, **52**, 7018−7022.

6 I. Kazi, S. Guha, G. Sekar, *Org. Lett.*, 2017, 19, 1244−1247.

7 T. Wang, G. Chen, Y.-J. Lu, Q. Chen, Y. Huo, X. Li, *Adv. Synth. Catal.*, 2019, **361**, 3886−3892.

8 B. Kurpil, K. Otte, A. Mishchenko, P. Lamagni, W. Lipiński, N. Lock, M. Antonietti, A. Savateev, *Nat. Commun.*, 2019, **10**, 945.

9 Y. Nishikawa, H. Yamamoto, *J. Am. Chem. Soc.*, 2011, **133**, 8432−8435.

10 S. Martinez-Erro, A. Sanz-Marco, A. Bermejo Gómez, A. Vázquez-Romero, M. S. G. Ahlquist, B. Martín-Matute, *J. Am. Chem. Soc.*, 2016, **138**, 13408−13414.

11 Y. Liu, Z. Chen, Q.-L. Wang, C.-S. Zhou, B.-Q. Xiong, C.-A. Yang, K.-W. Tang, *J. Org. Chem.*, 2019, **84**, 9984−9994.

12 Y. Zhang, J. Huang, Y. Guo, L. Li, Z. Fu, W. Huang, *Angew. Chem. Int. Ed.*, 2018, **57**, 4594−4598.

13 S. Tripathi, R. Kapoor, L. D. S. Yadav, *Adv. Synth. Catal.*, 2018, **360**, 1407−1413.

14 Y. Norikane, H. Itoh, T. Arai, *J. Phys. Chem. A*, 2002, **106**, 2766−2776.

15 M.-J. Zhang, H.-X. Li, D. J. Young, H.-Y. Li, J.-P. Lang, *Org. Biomol. Chem.*, 2019, **17**, 3567−3574.

16 Z. Lei, A. Banerjee, E. Kusevska, E. Rizzo, P. Liu, M.-Y. Ngai, *Angew. Chem. Int. Ed.*, 2019, **58**, 7318−7323.

17 A. Shrestha, A. Shrestha, P.-H. Park, E.-S. Lee, *Bull. Korean Chem. Soc.*, 2019, **40**, 729-734.

18 X.-F. Wu, H. Neumann, A. Spannenberg, T. Schulz, H. Jiao, M. Beller, *J. Am. Chem. Soc.*, 2010, **132**, 14596−14602.

19 M. Vellakkaran, M. M. S. Andappan, K. Nagaiah, *RSC Adv.*, 2014, **4**, 45490−45494.

20 K. Kobayashi, A. Imaoka, *Helv. Chim. Acta*, 2013, **96**, 624−632.

21 G. F. P. de Souza, J. A. Bonacin, A. G. Salles, Jr., *J. Org. Chem.*, 2018, **83**, 8331−8340.

22 R. Gresser, H. Hartmann, M. Wrackmeyer, K. Leo, M. Riede, Tetrahedron, 2011, 67, 7148-7155.

23 X. Yan, B. Yu, H. Zhang, H. Huang, *J. Org. Chem.*, 2022, **87**, 16918−16927.

24 K. R. Fandrick, D. R. Fandrick, J. J. Gao, J. T. Reeves, Z. Tan, W. Li, J. J. Song, B. Lu, N. K. Yee, C. H. Senanayake, *Org. Lett.*, 2010, **12**, 3748−3751.

25 S. A. Abbass, G. A. I. Moustafa, H. A. Hassan, G. E.-D. A. Abuo-Rahma, *Synth. Commun.*, 2019, **49**, 2995−3000.

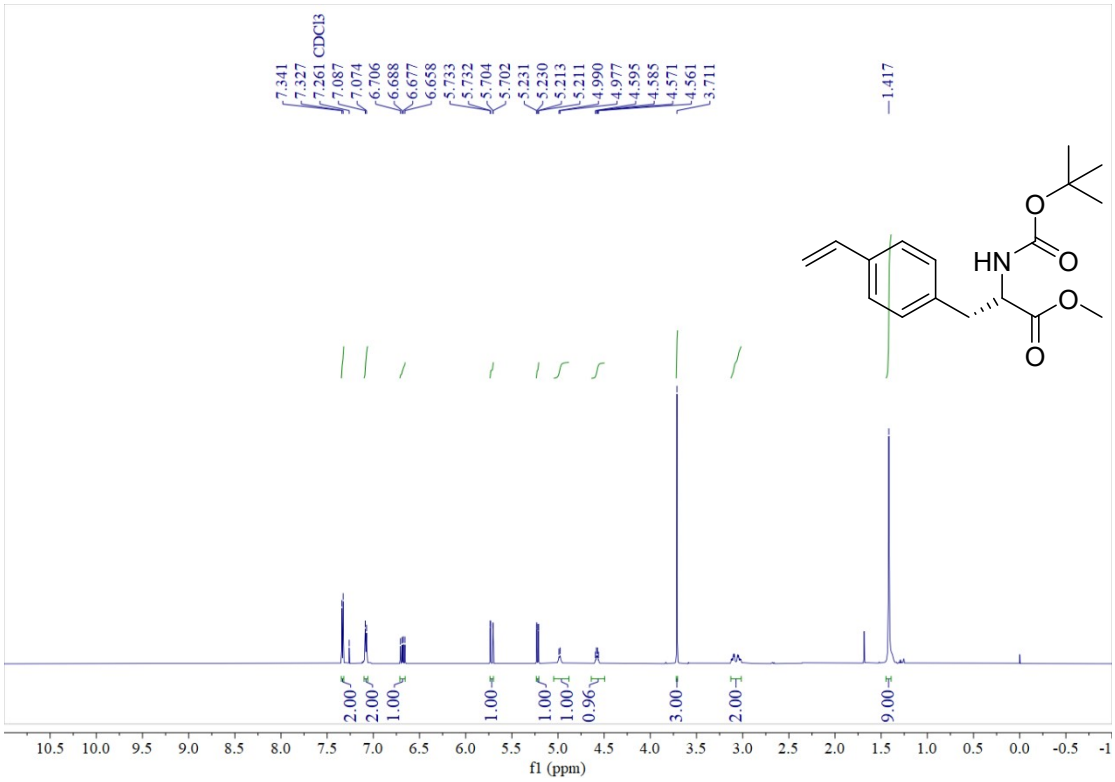
26 H. Zhang, Q. Wei, G. Zhu, J. Qu, B. Wang, *Tetrahedron Lett.*, 2016, **57**, 2633−2637.

27 D. V. Patil, H. Y. Kim, K. Oh, *Org. Lett.*, 2020, **22**, 3018−3022.

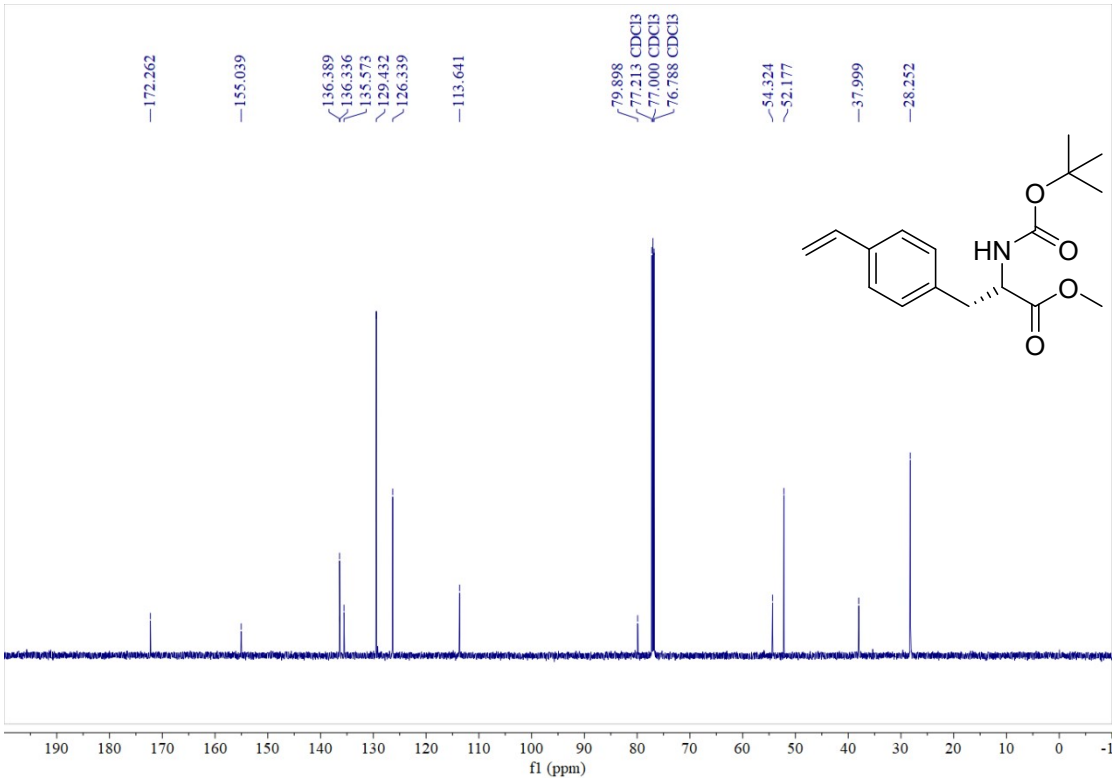
28 J. Guin, S. De Sarkar, S. Grimme, A. Studer, *Angew. Chem. Int. Ed.*, 2008, **47**, 8727−8730.

12. NMR Spectra of Substrates, Products and Derivatives

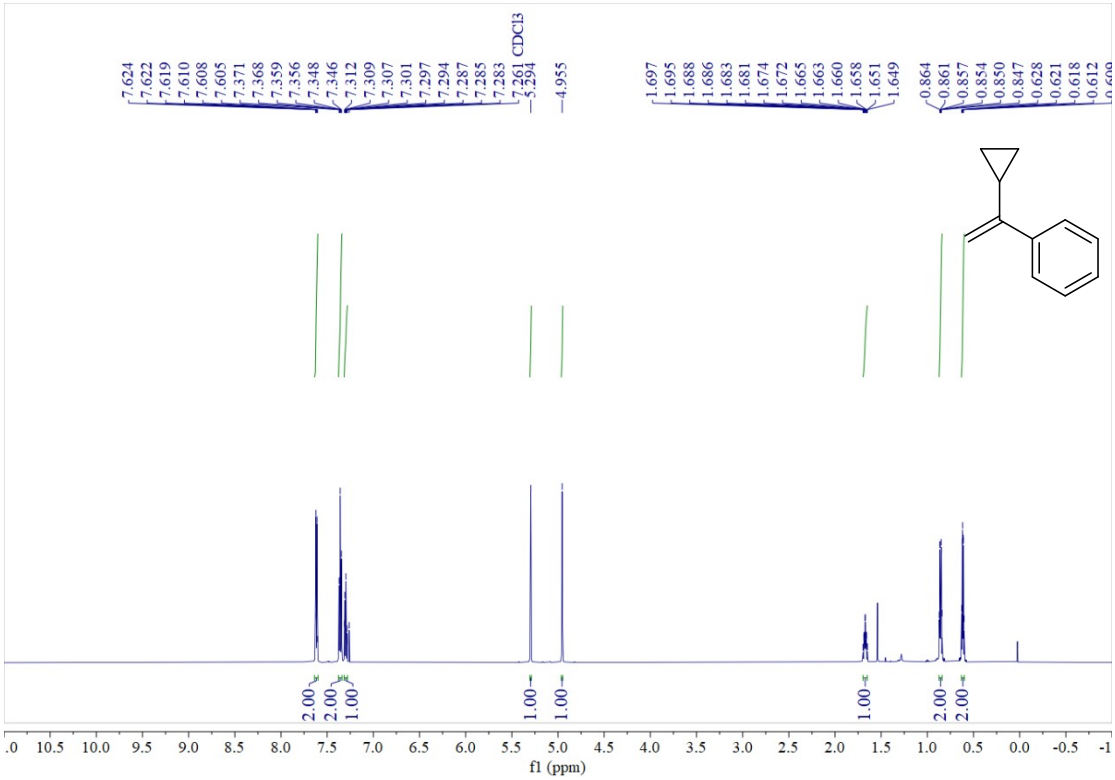
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **2s**



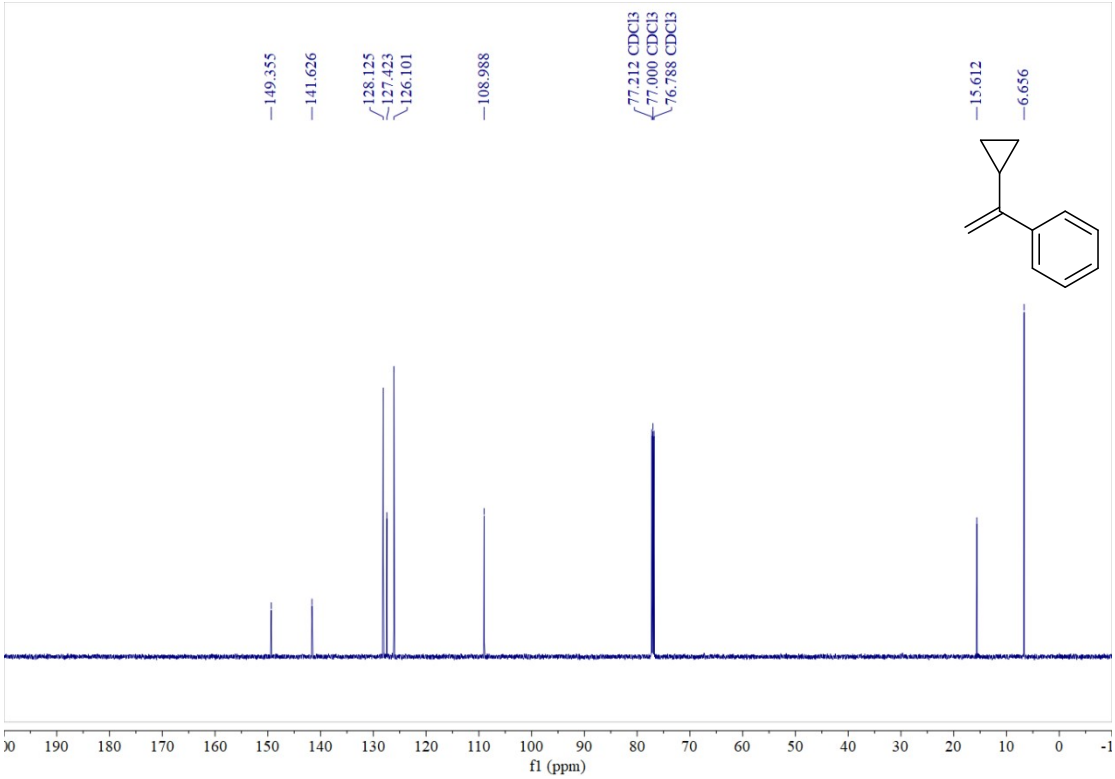
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum of **2s**



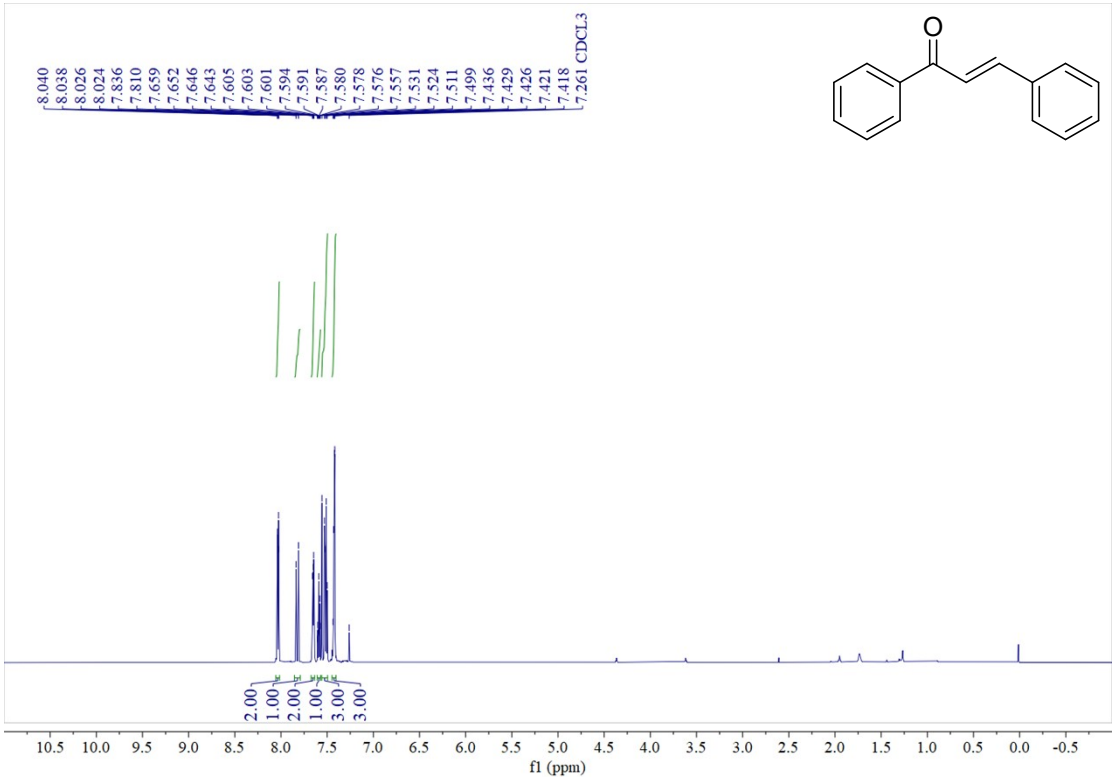
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **2t**



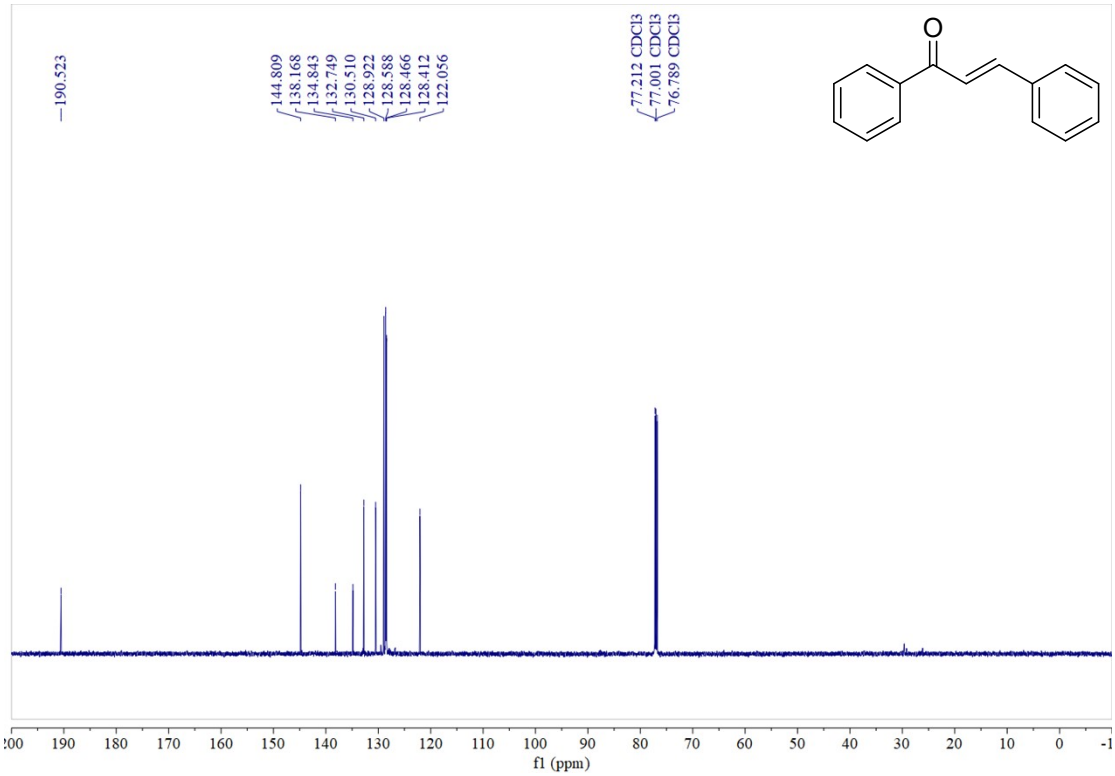
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **2t**



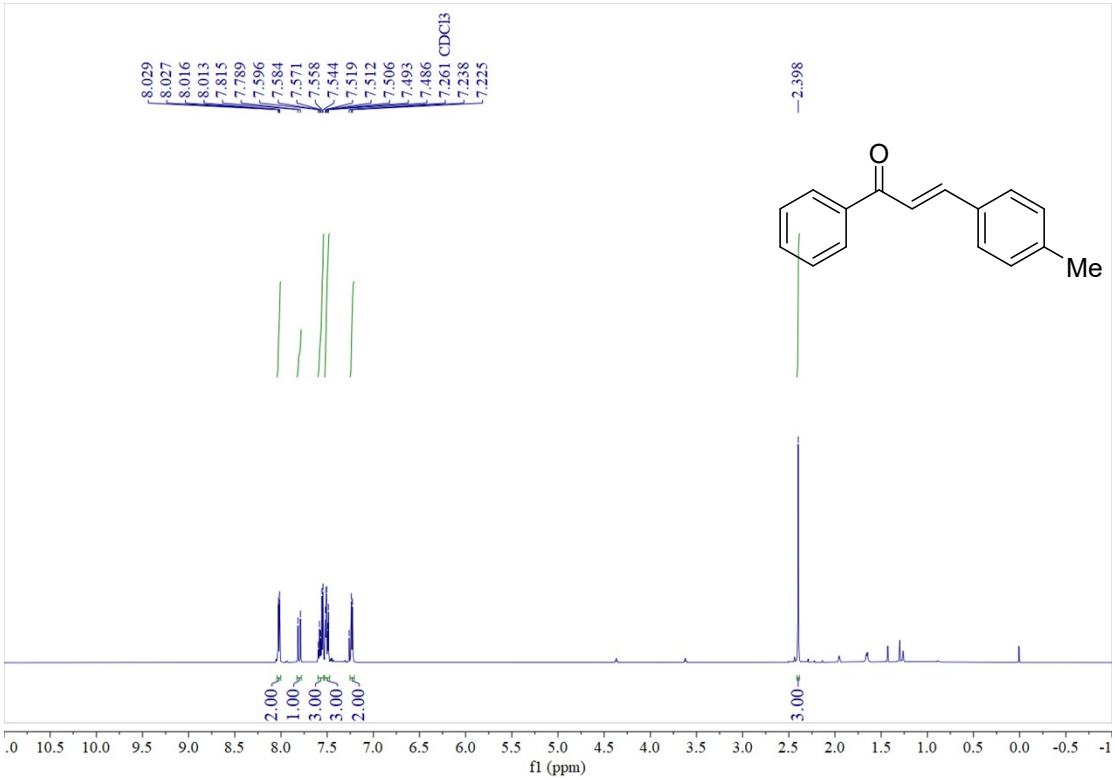
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3a**



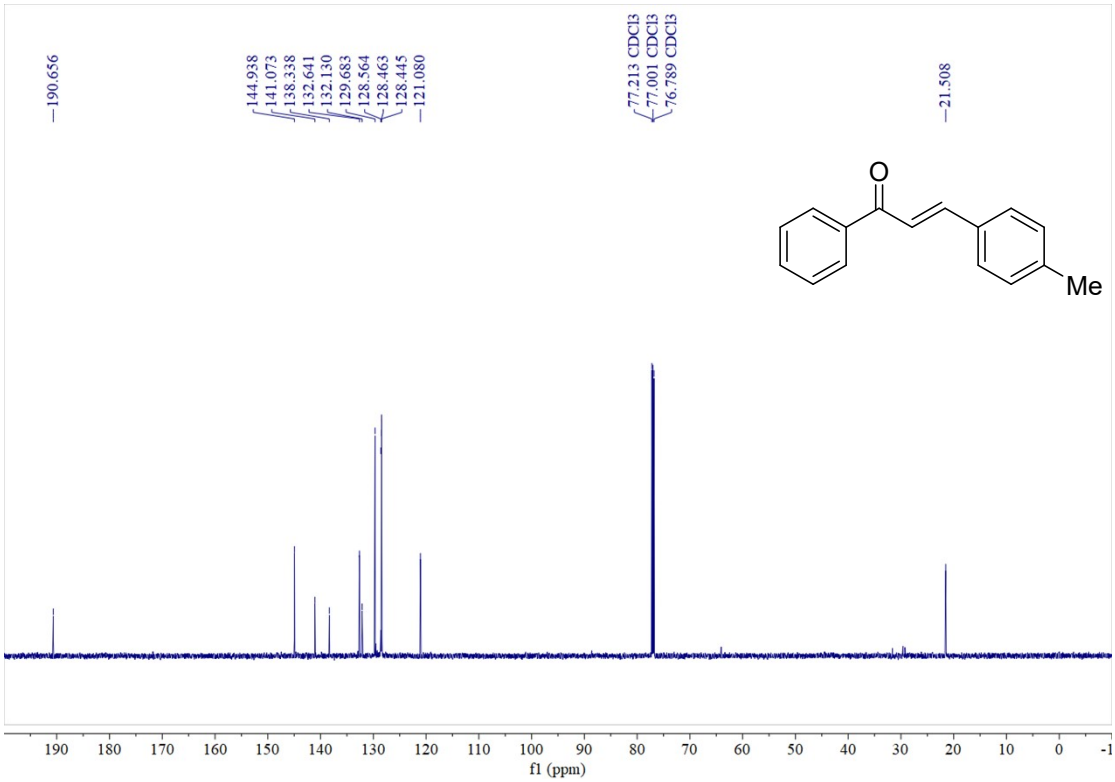
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3a**



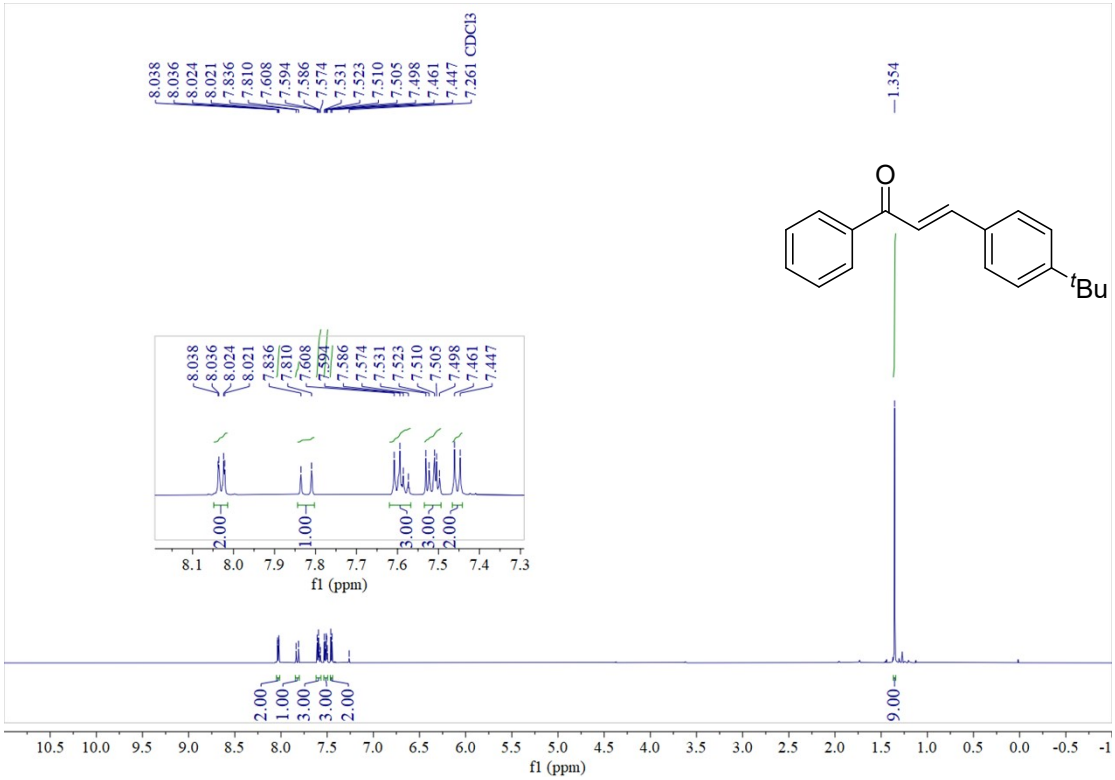
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3b**



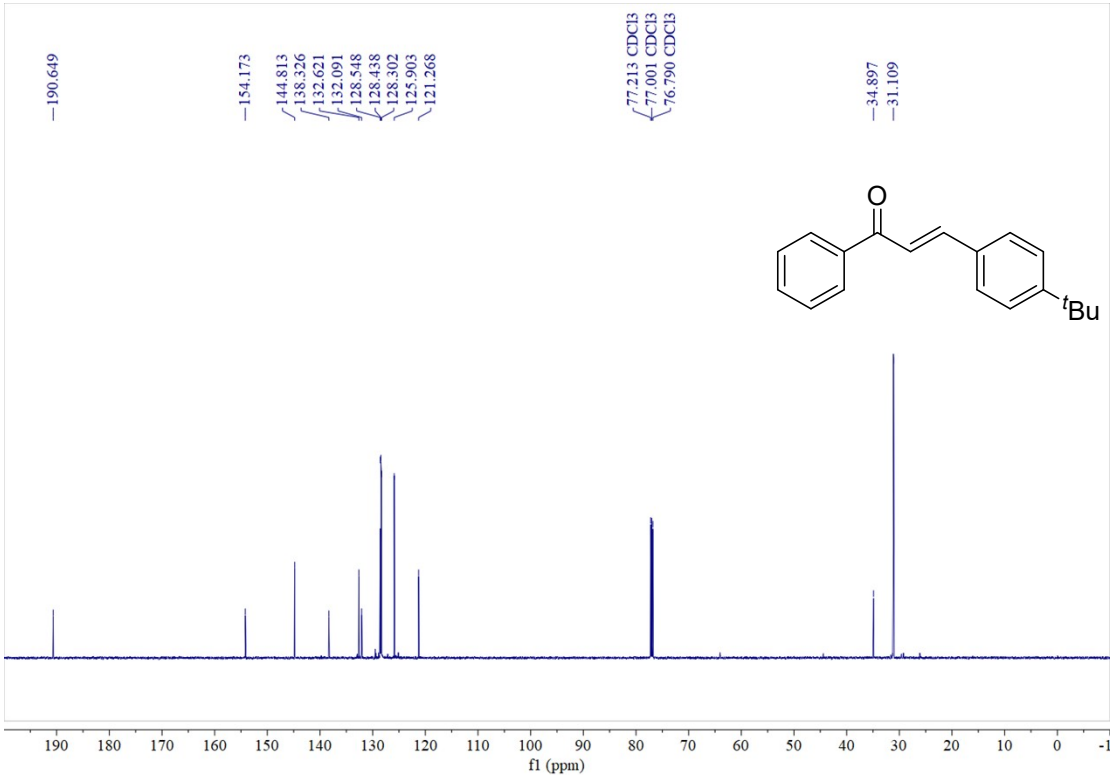
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3b**



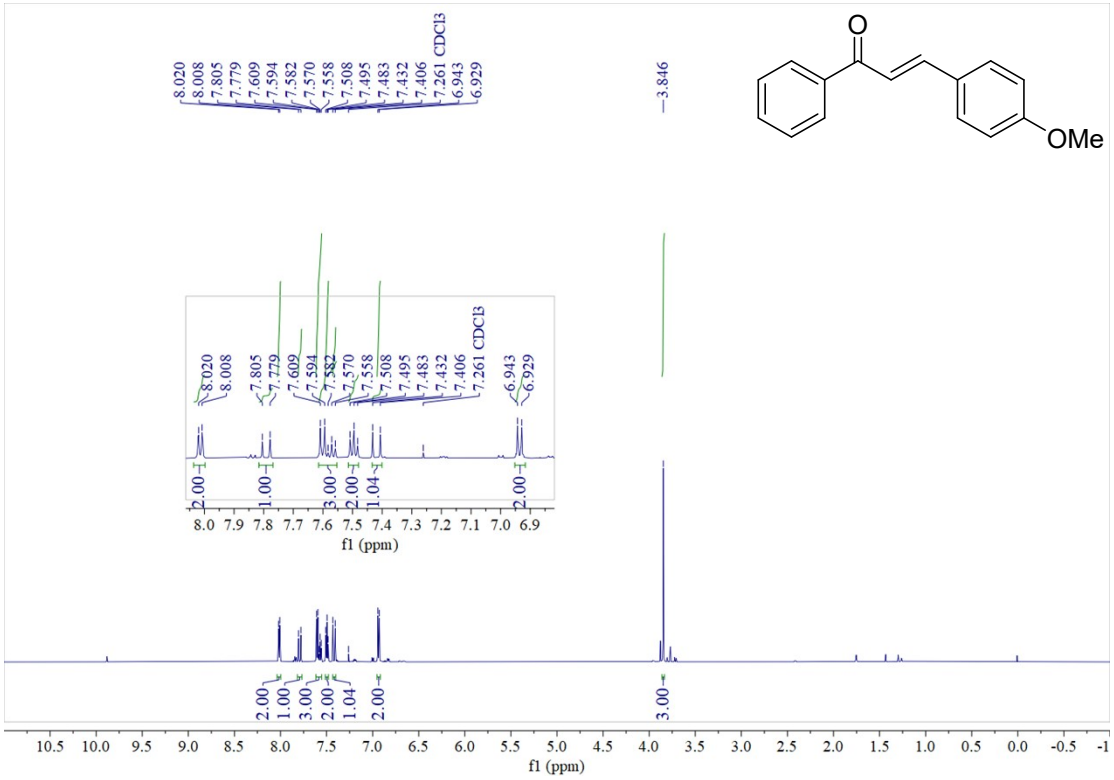
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3c**



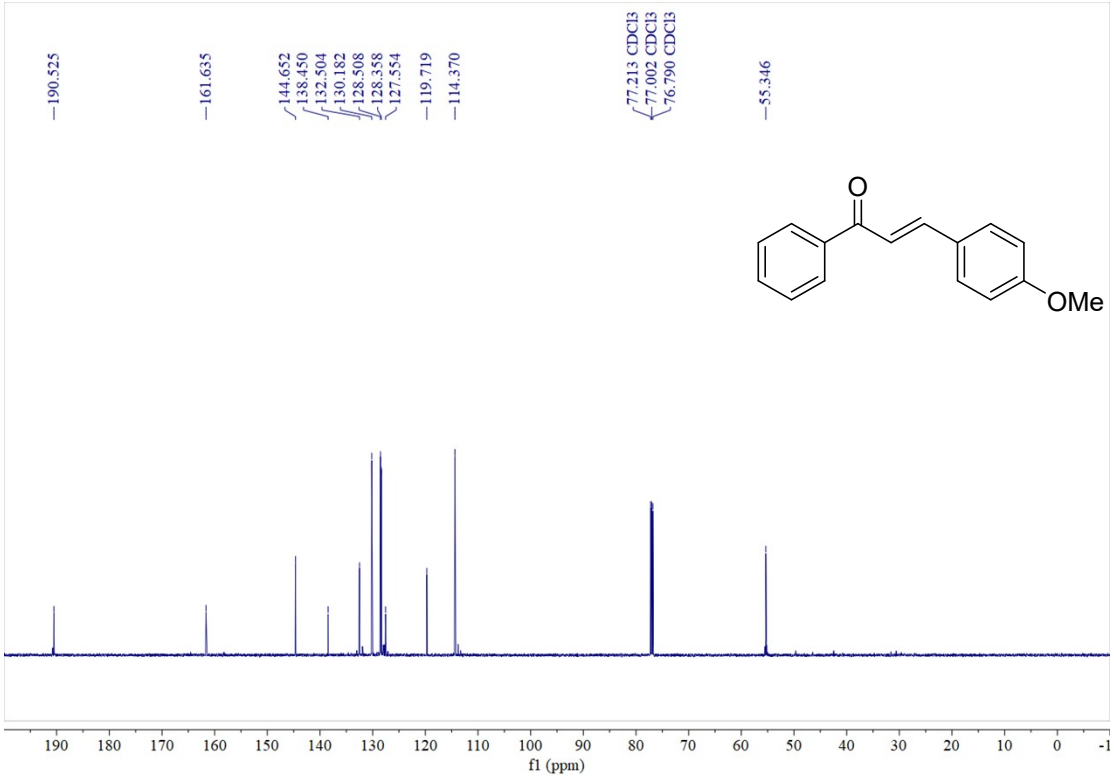
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3c**



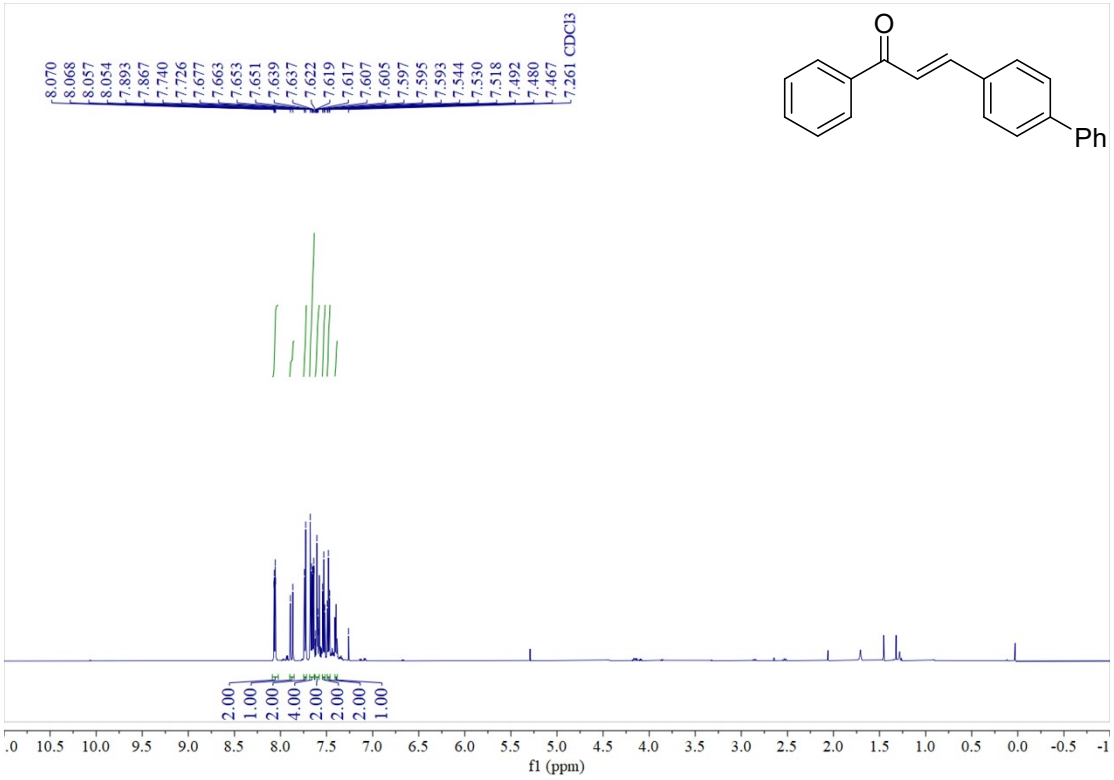
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3d**



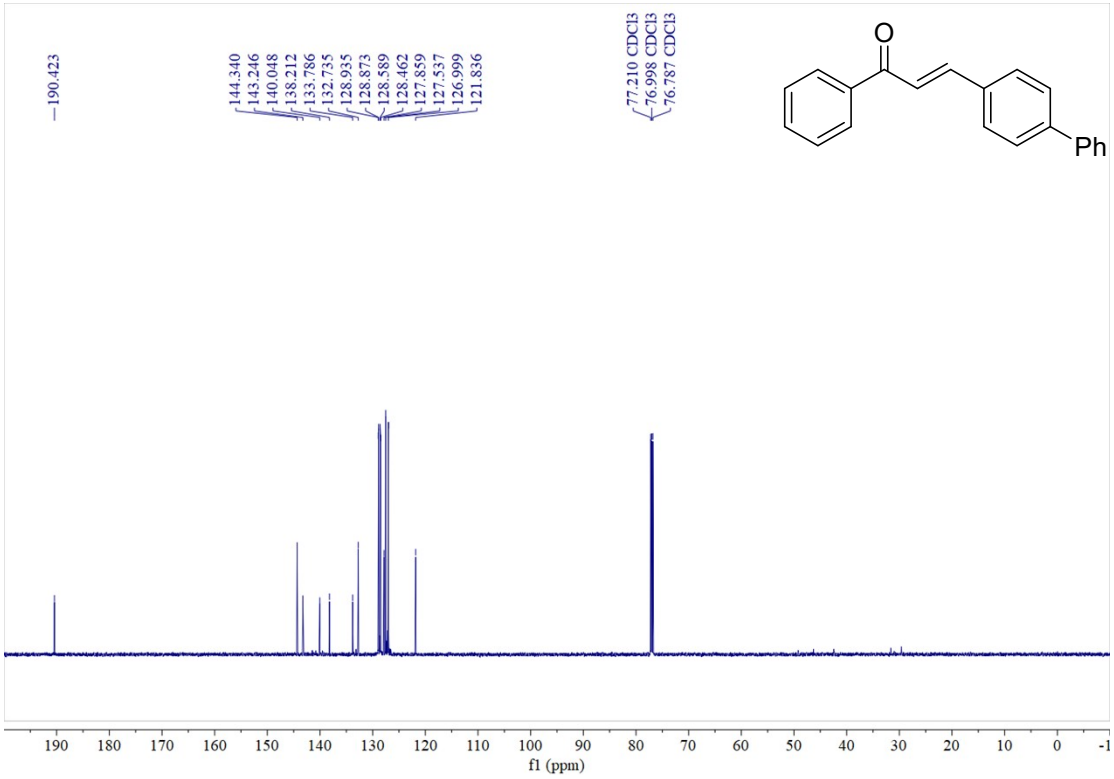
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3d**



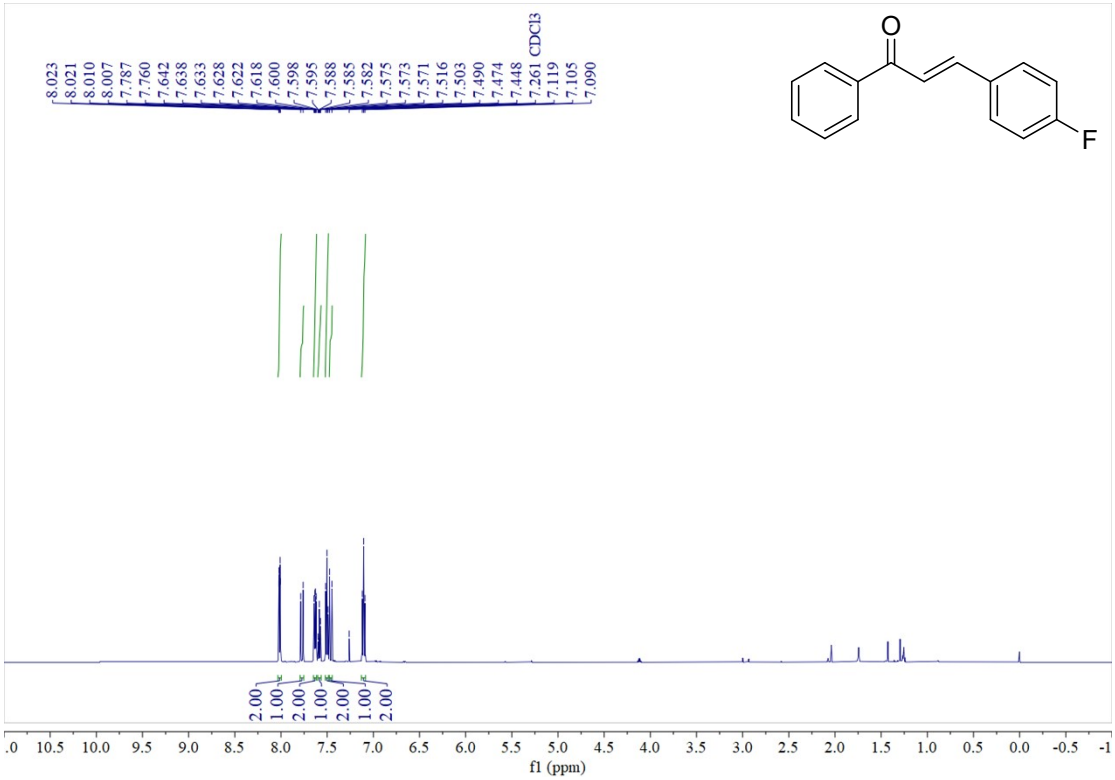
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3e**



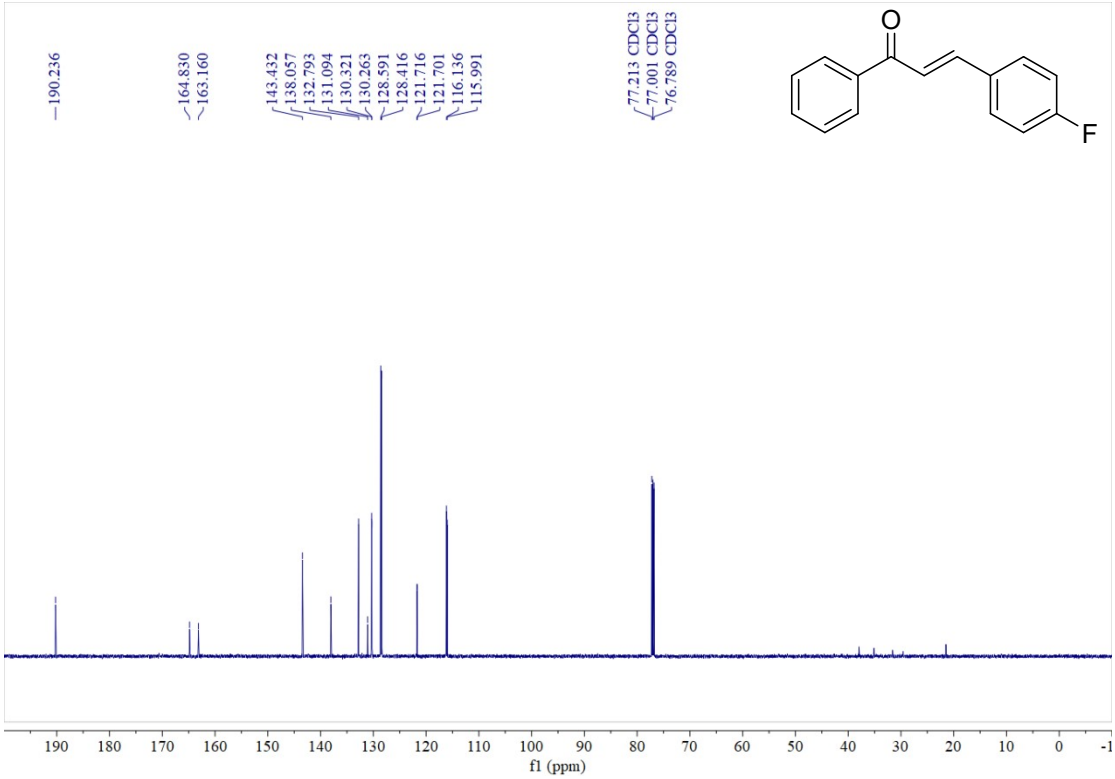
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3e**



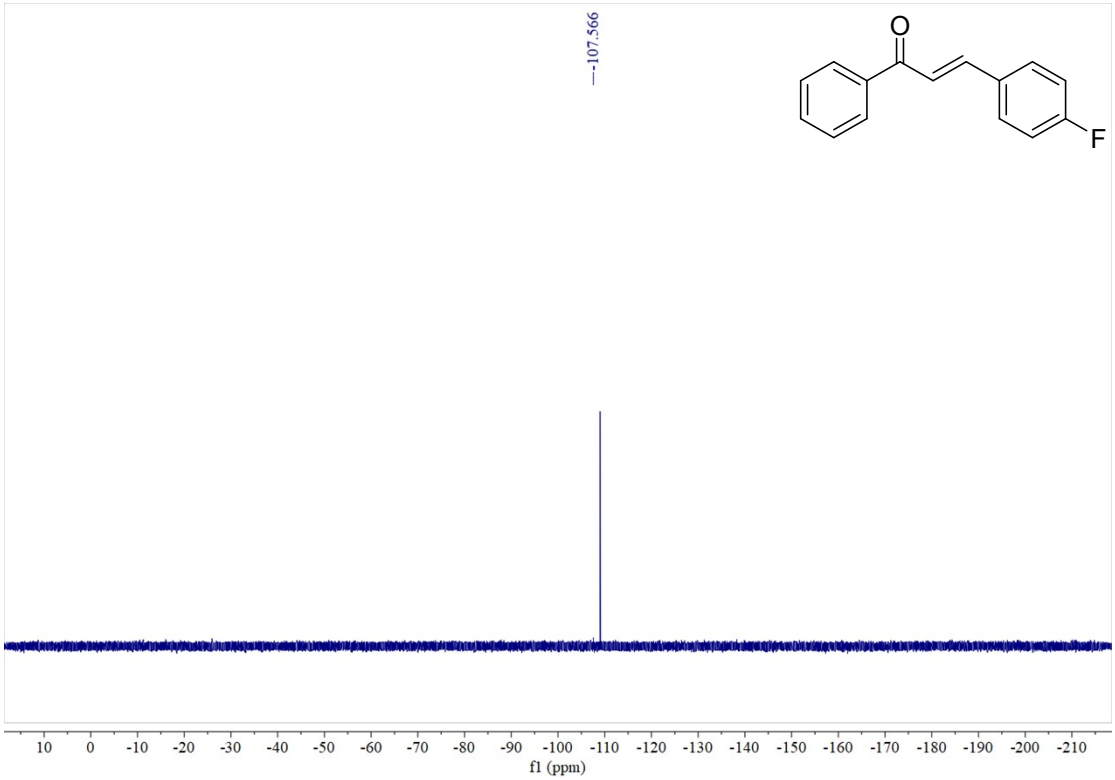
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3f**



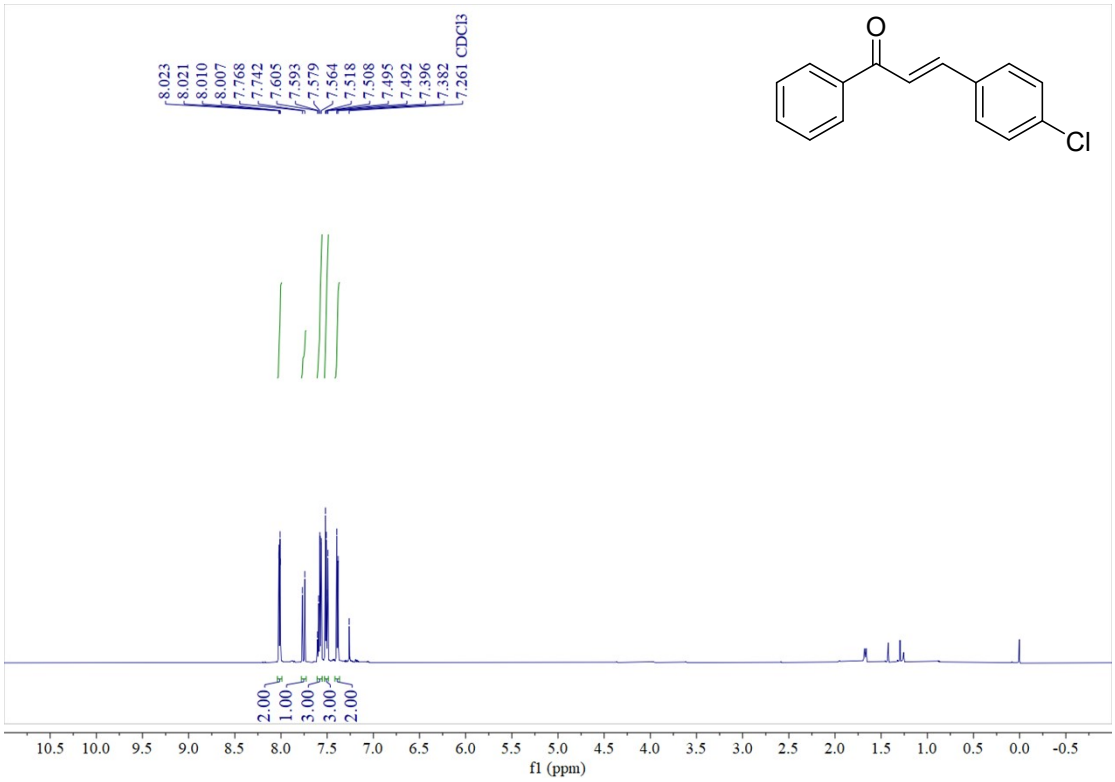
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3f**



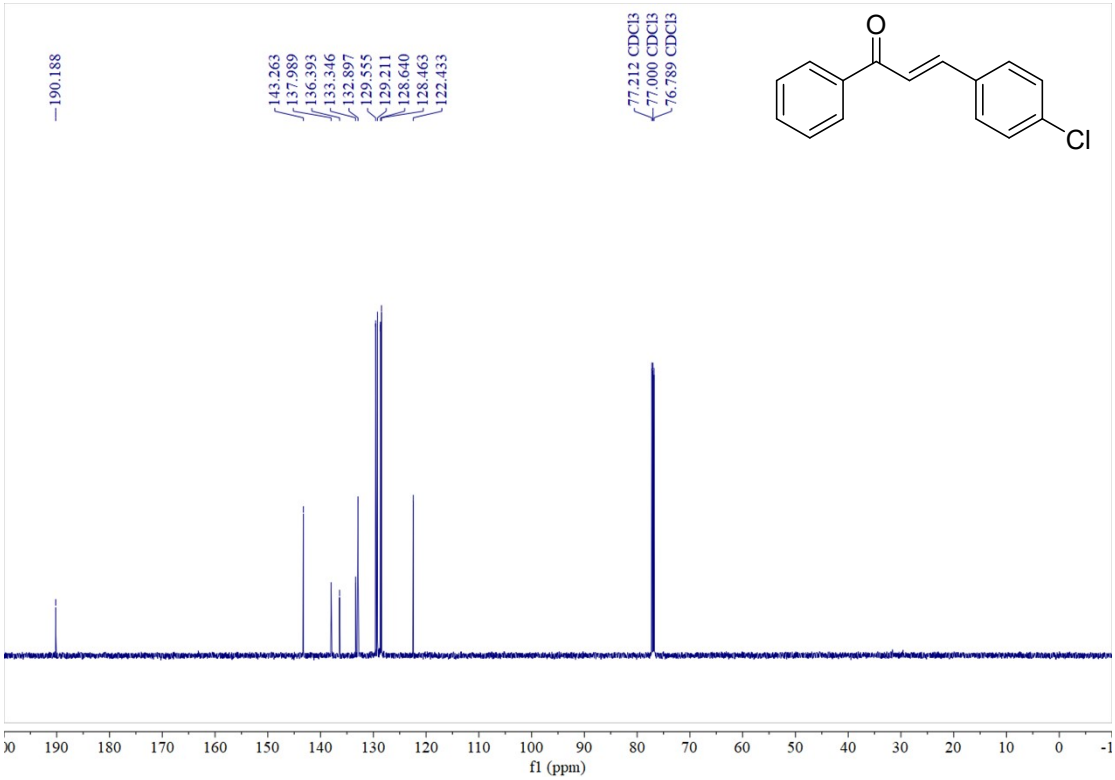
^{19}F NMR (565 MHz, $CDCl_3$) Spectrum of **3f**



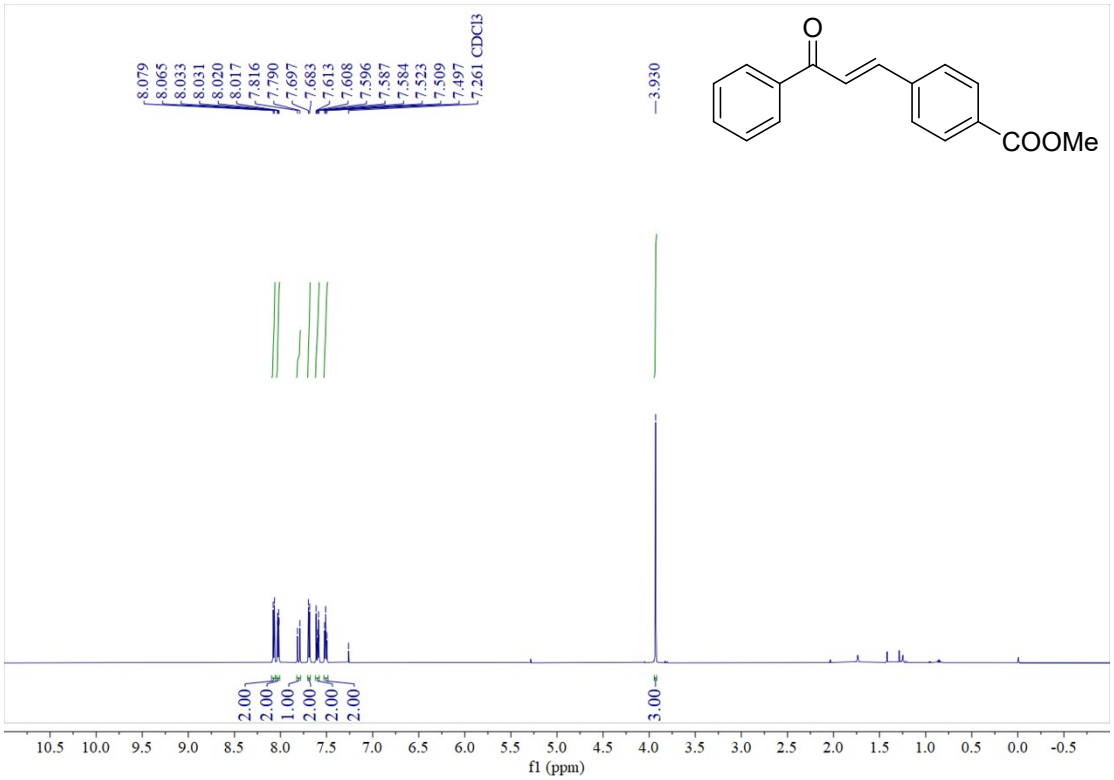
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3g**



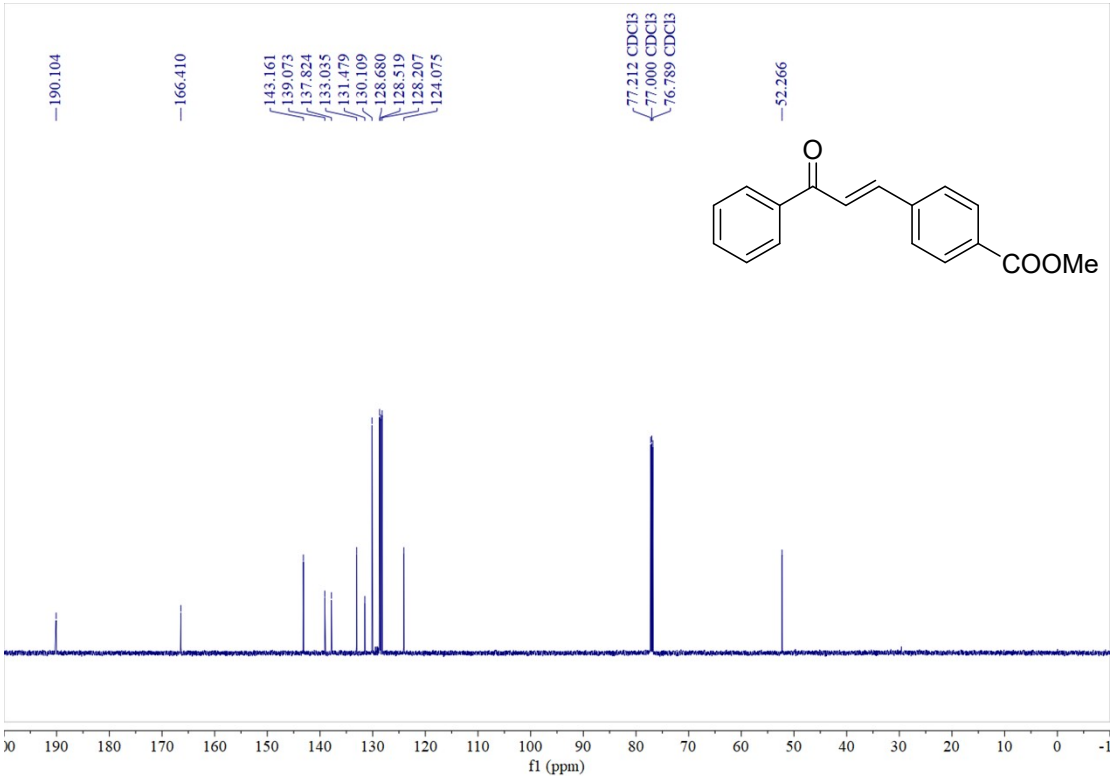
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3g**



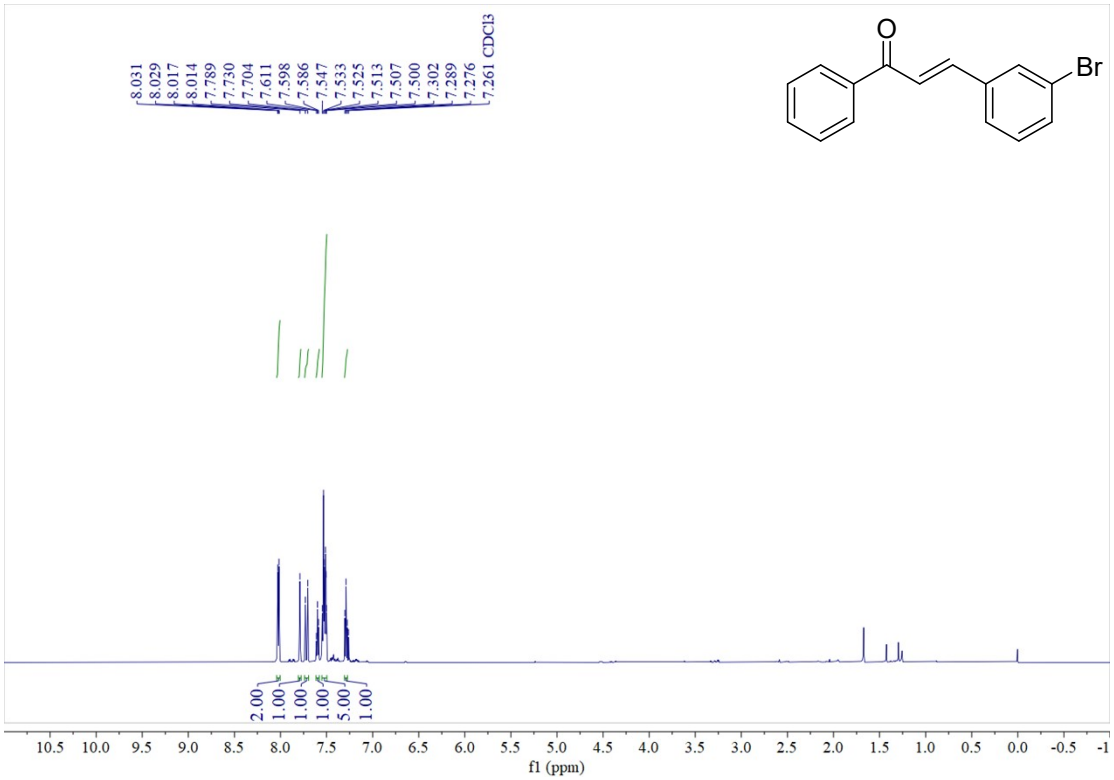
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3h**



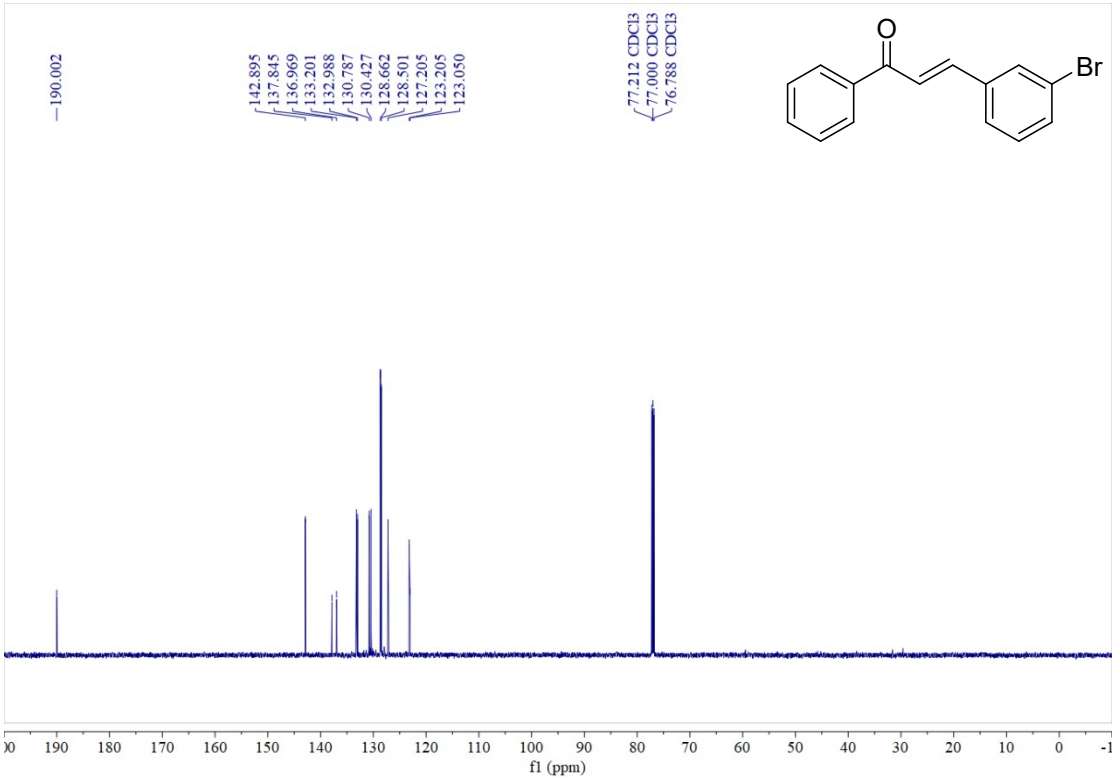
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3h**



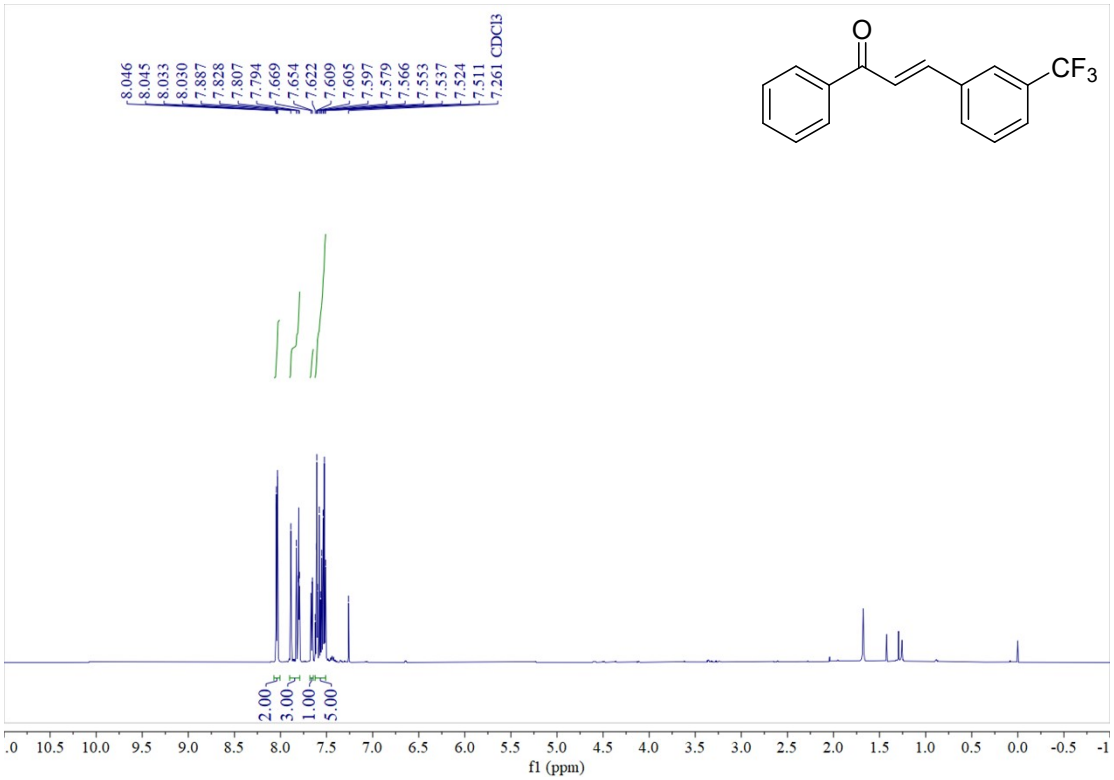
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3i**



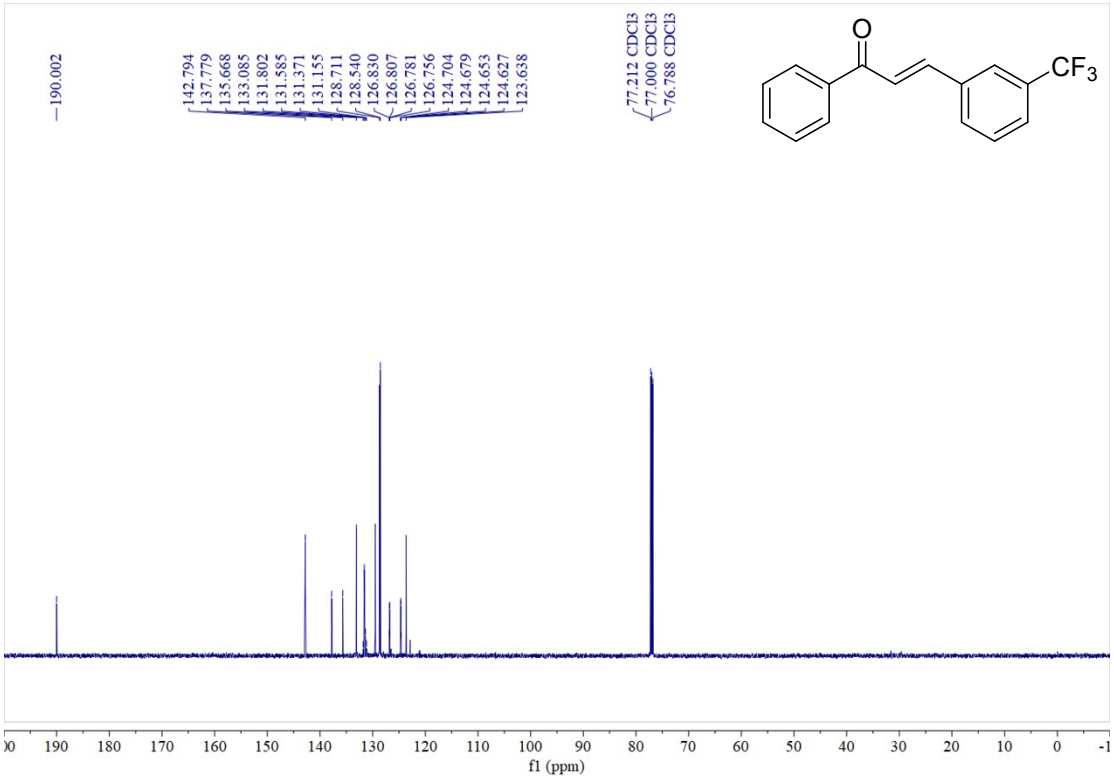
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3i**



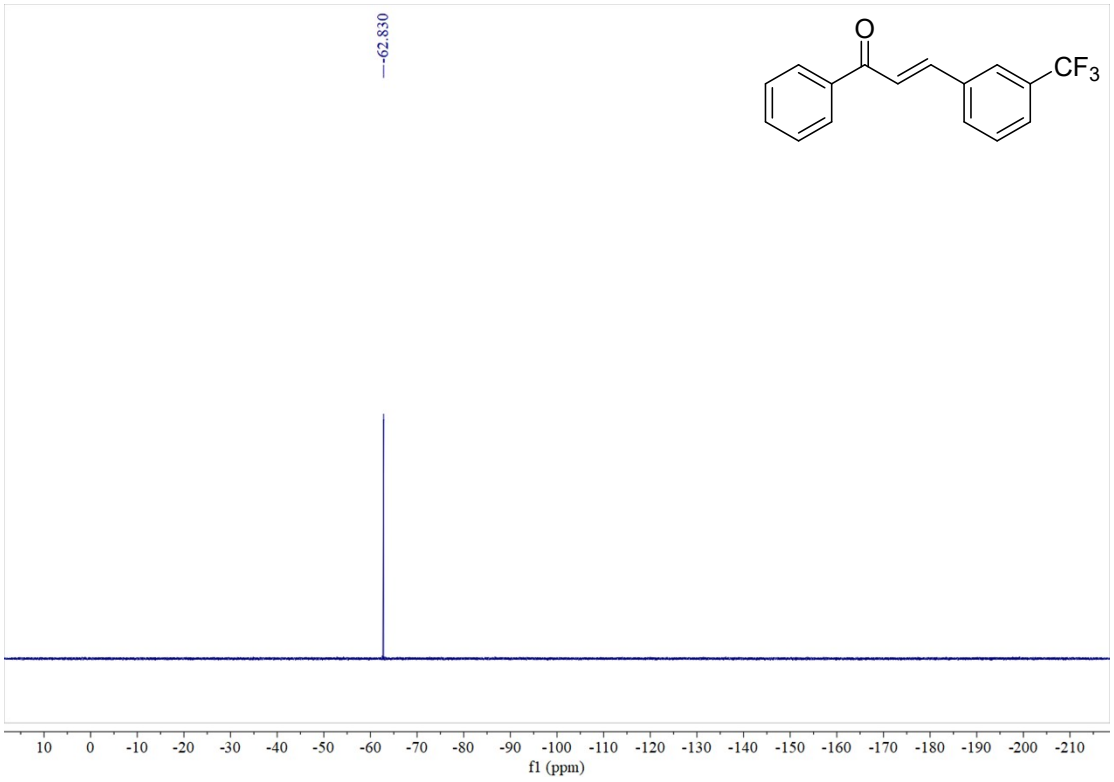
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3j**



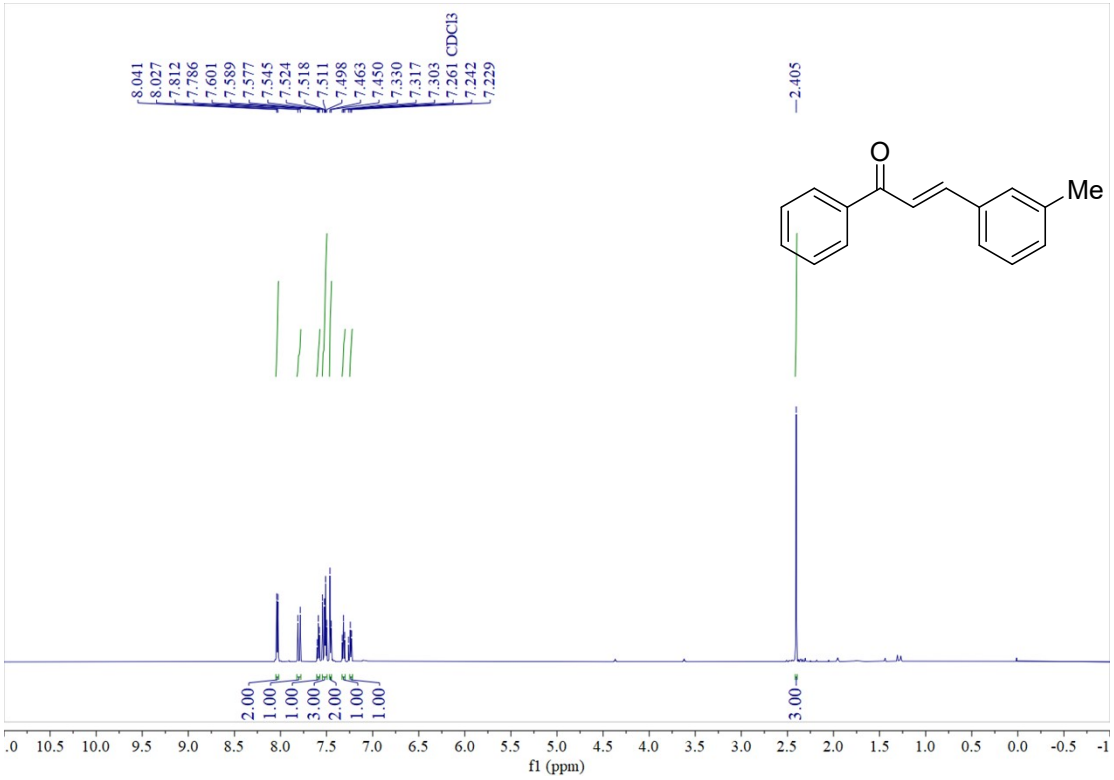
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3j**



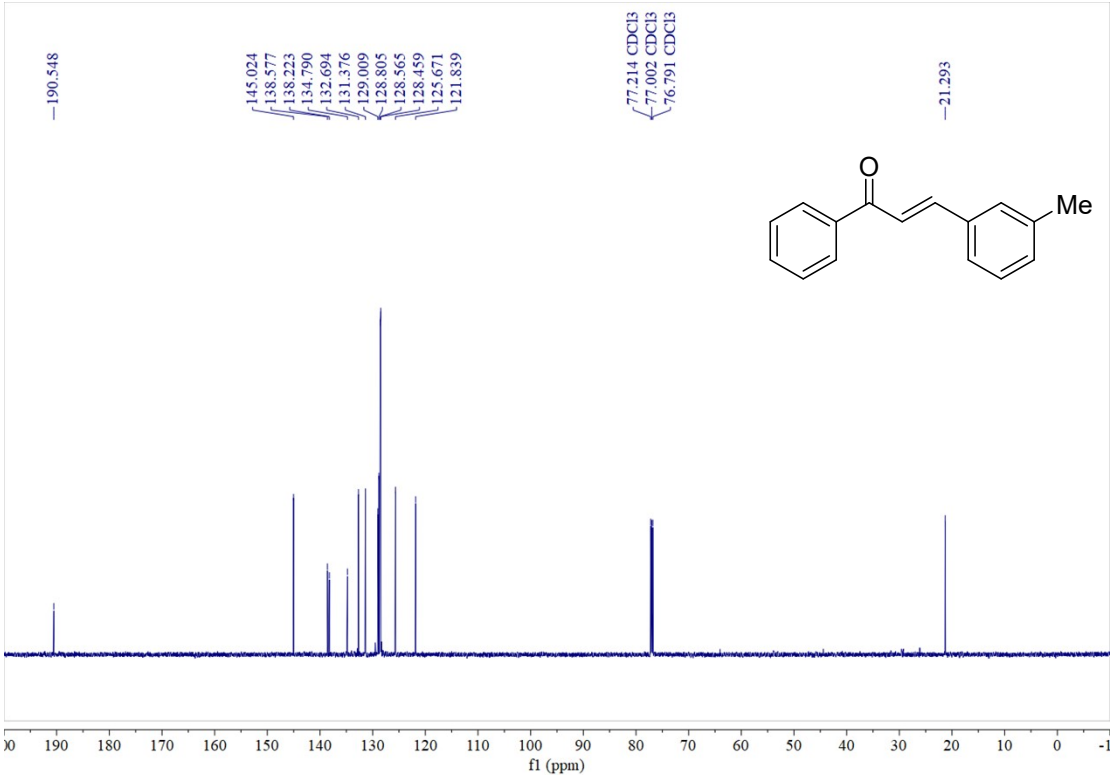
^{19}F NMR (565 MHz, $CDCl_3$) Spectrum of **3j**



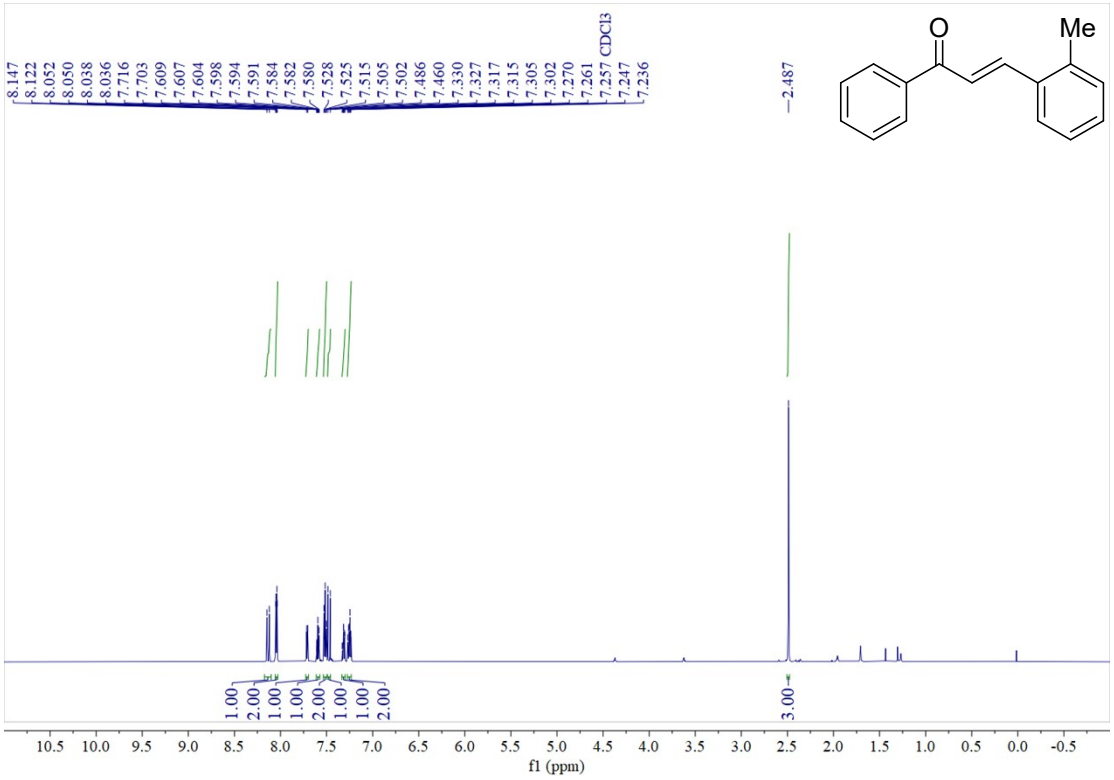
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3k**



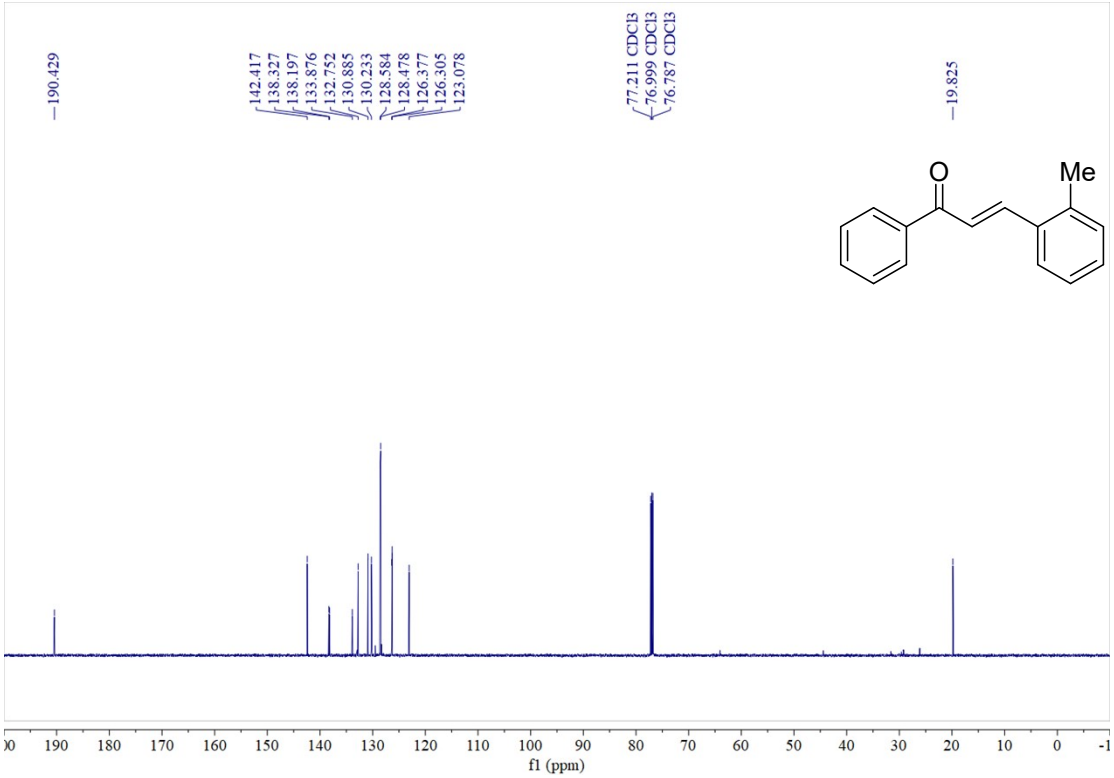
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3k**



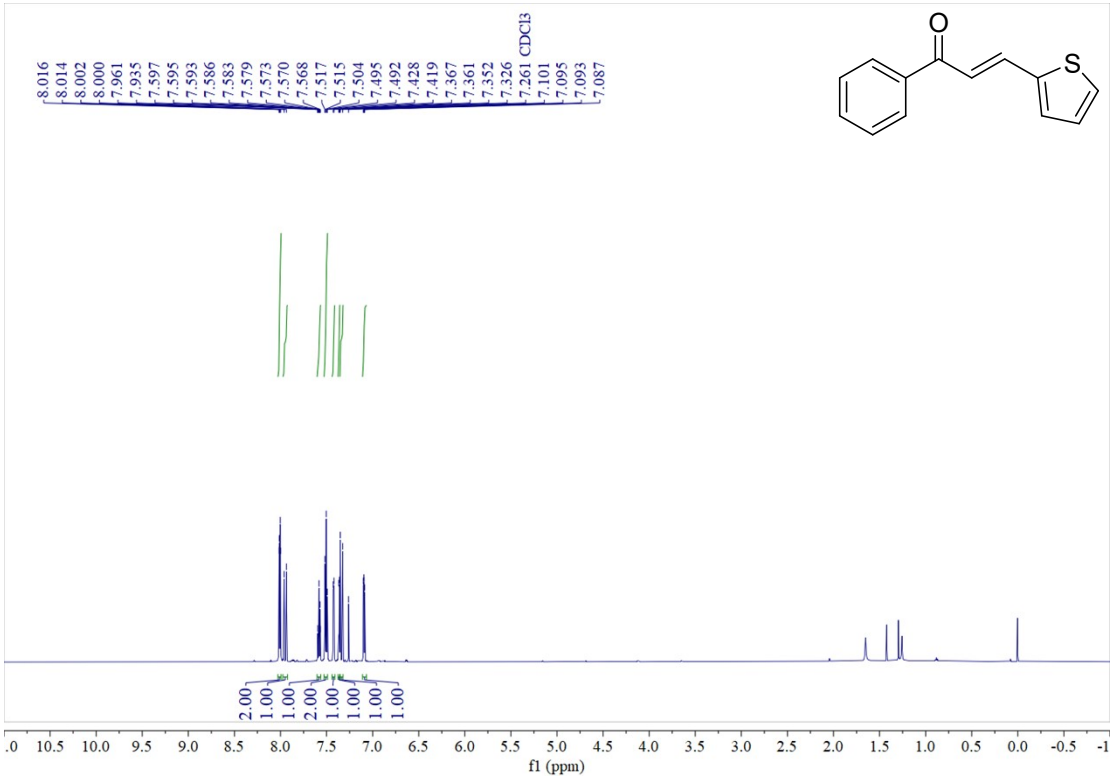
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3l**



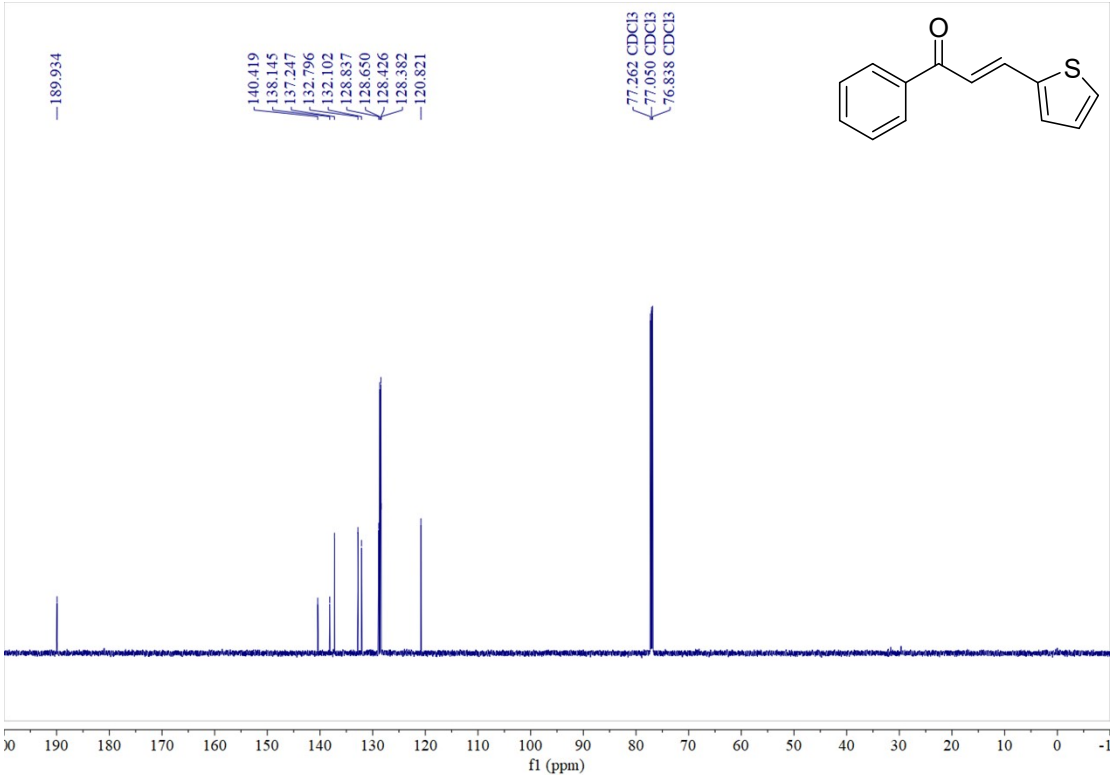
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3l**



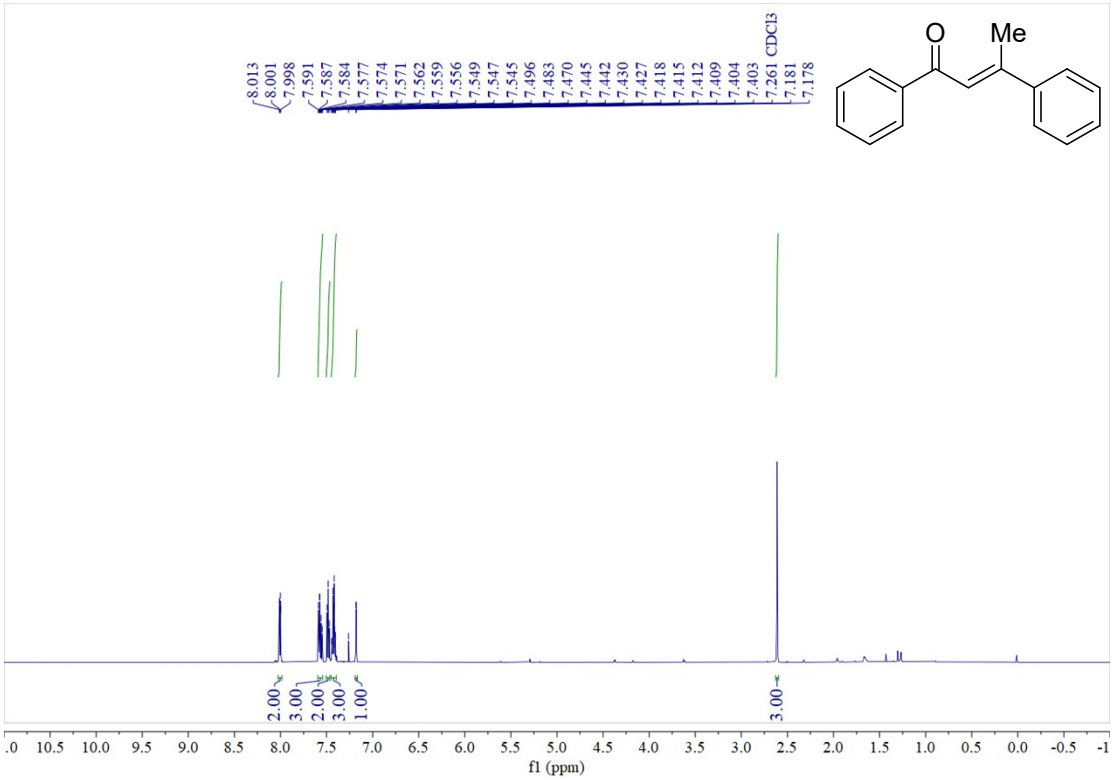
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3m**



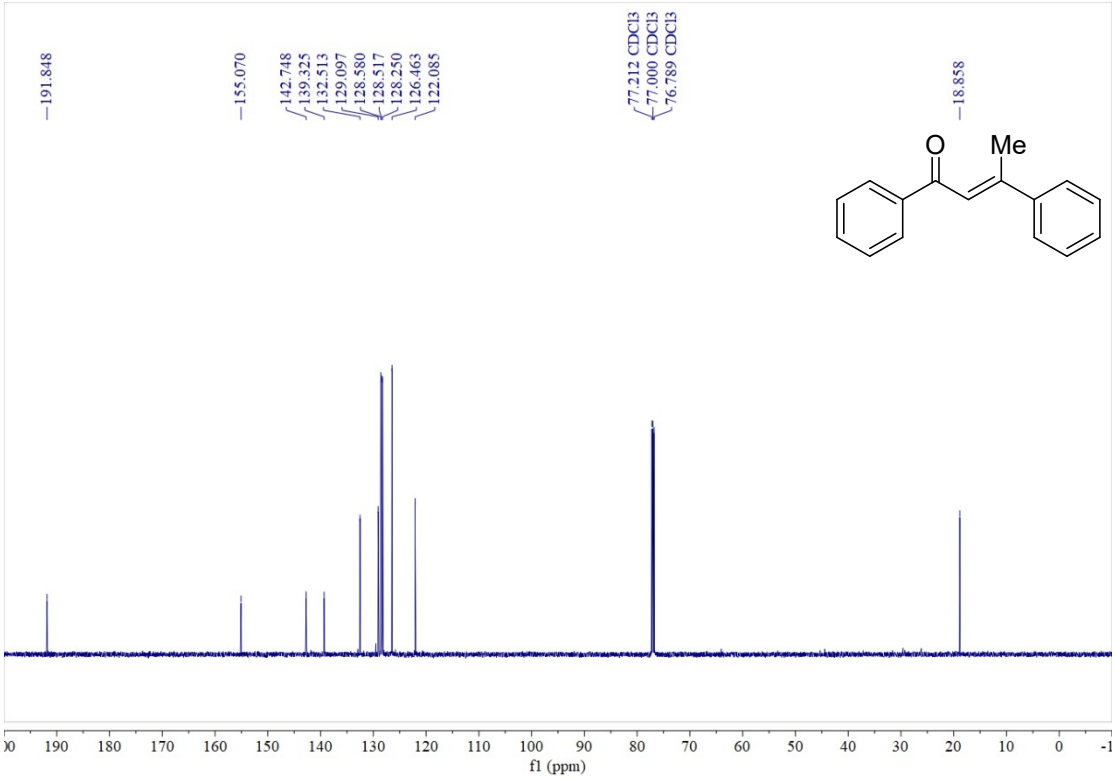
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3m**



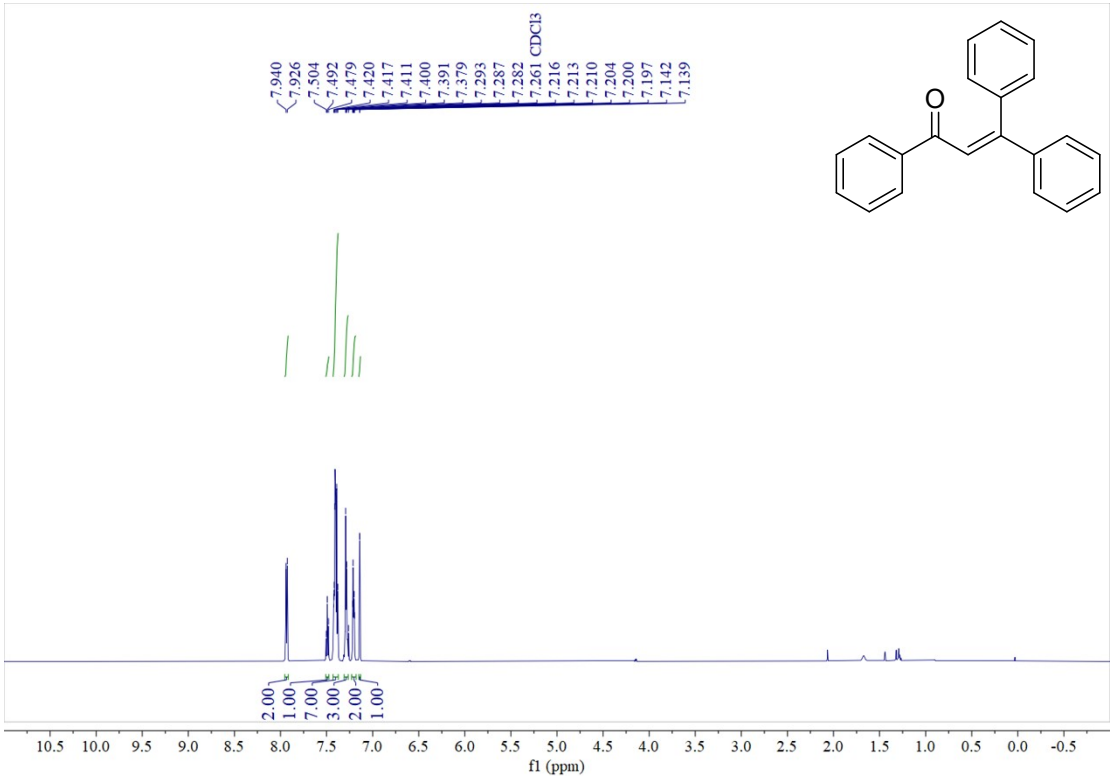
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3n**



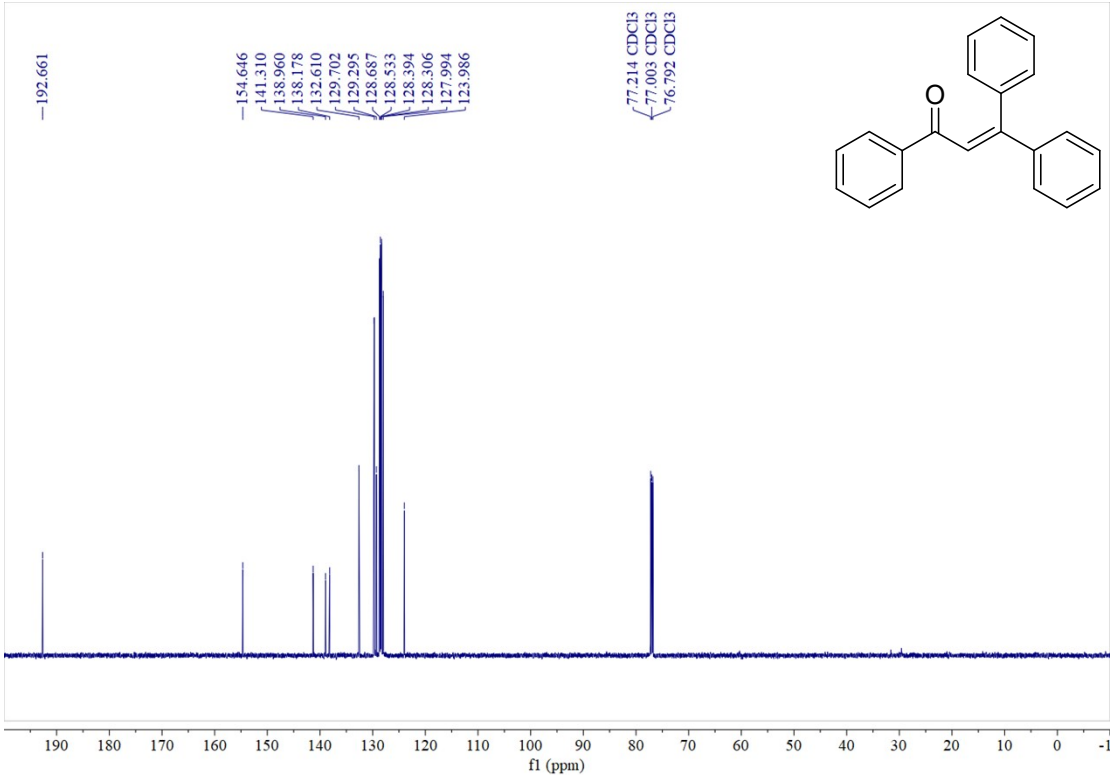
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3n**



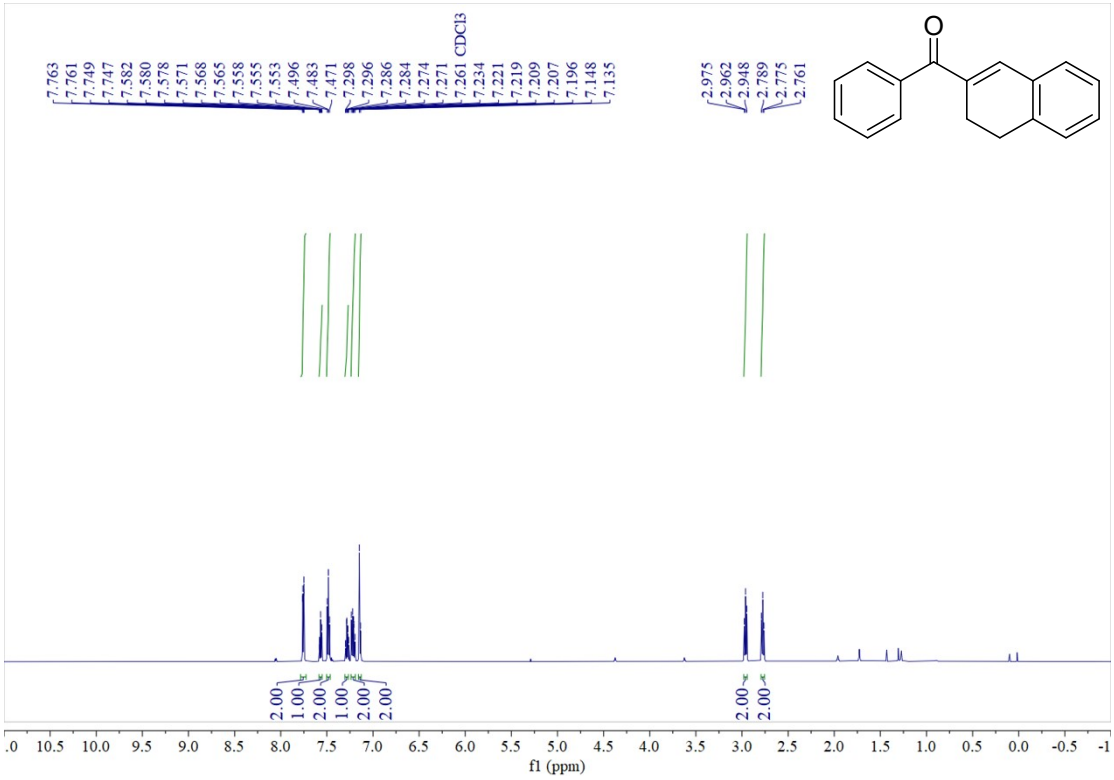
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3o**



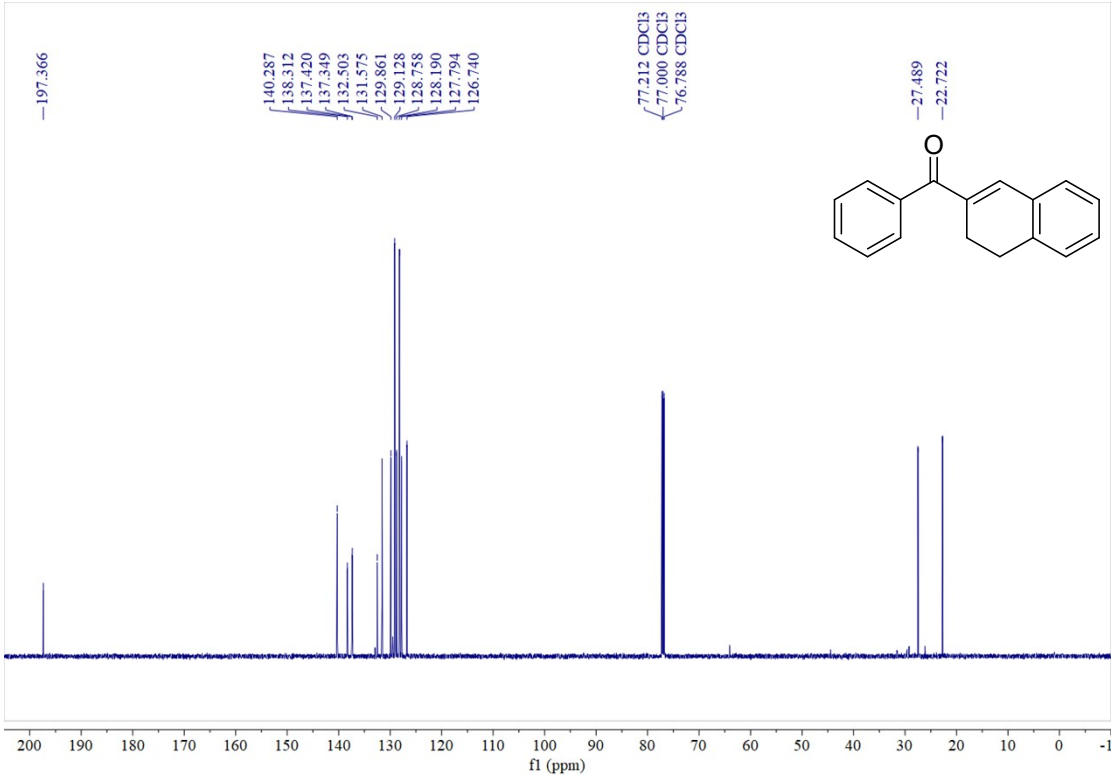
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3o**



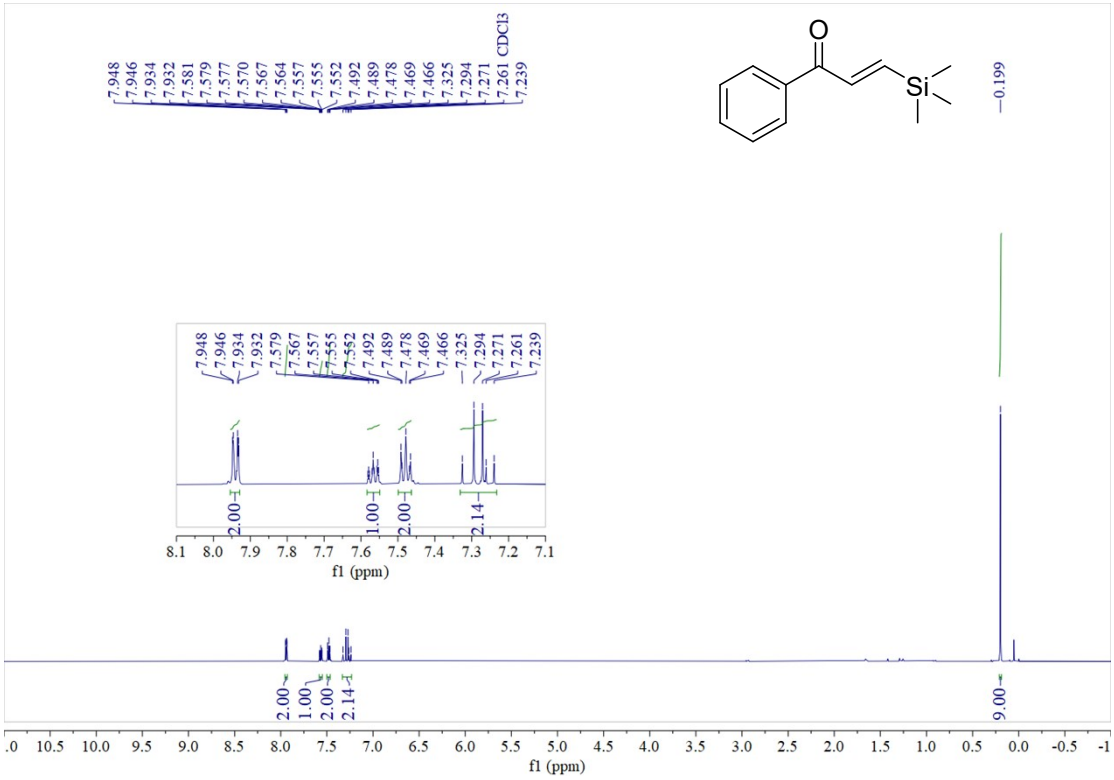
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3p**



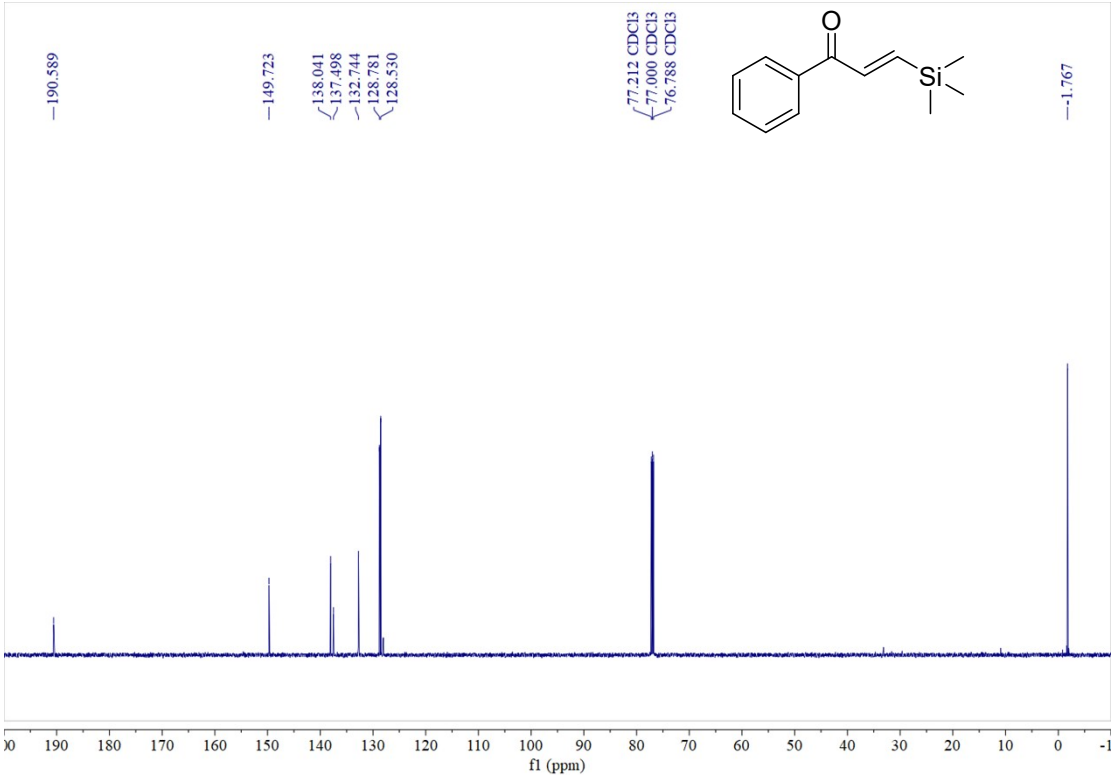
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3p**



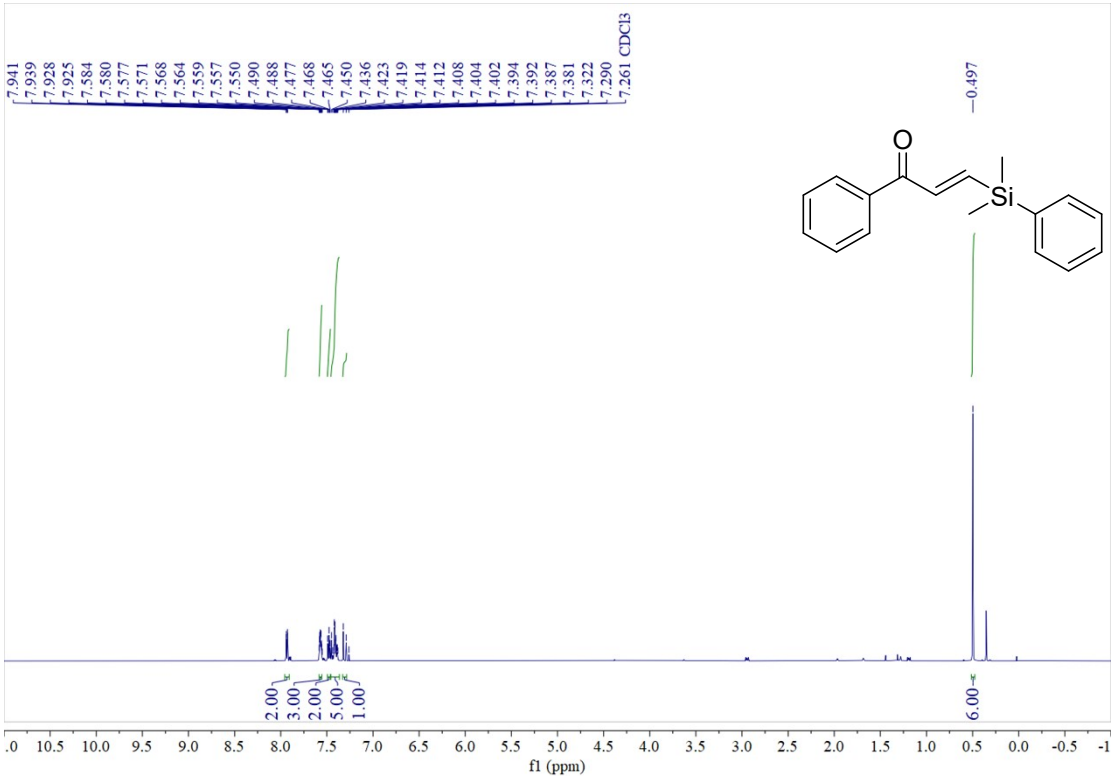
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3q**



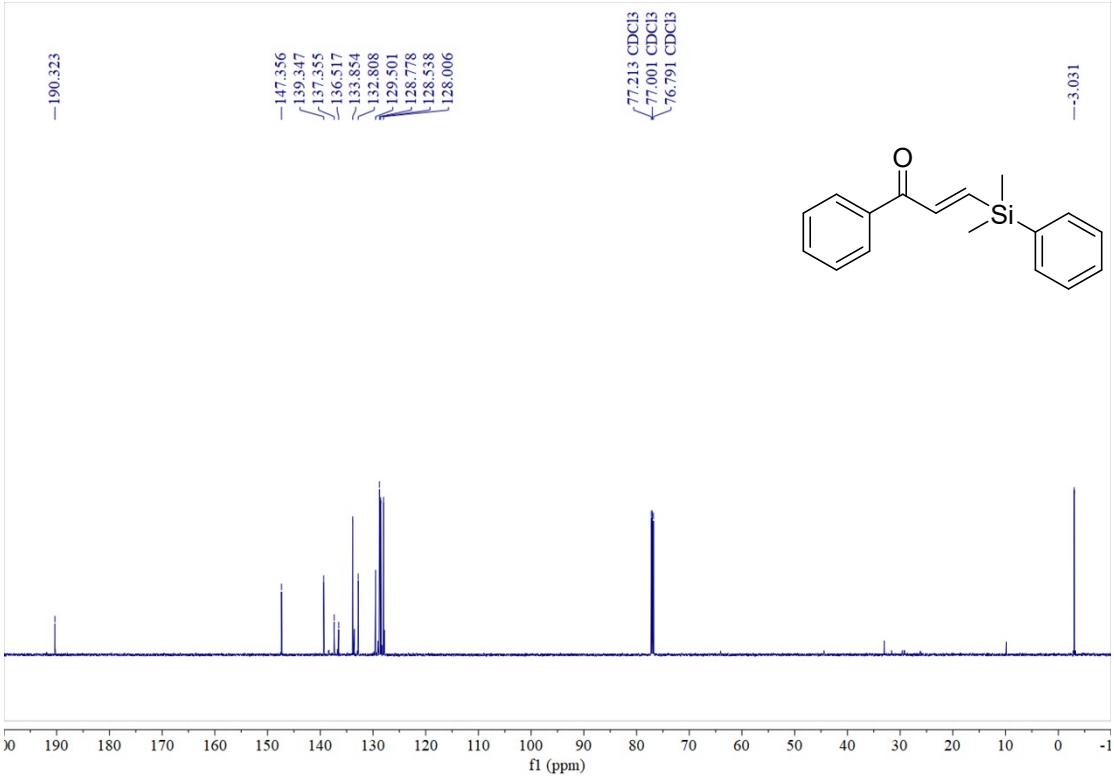
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3q**



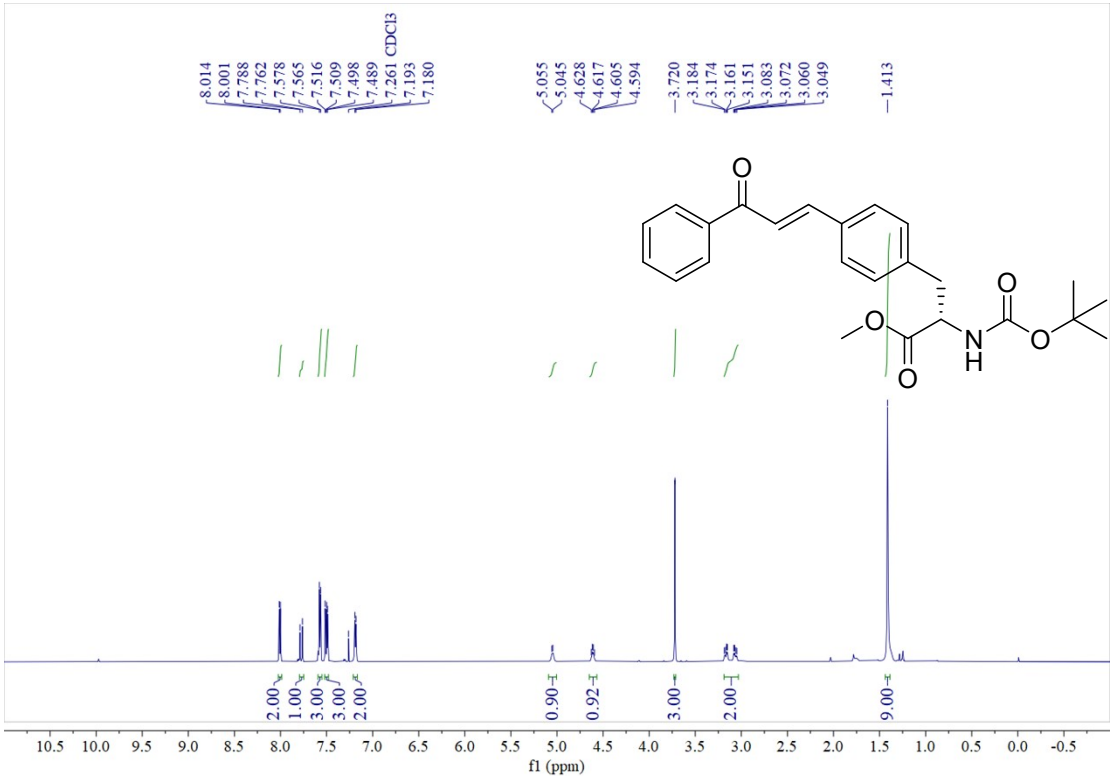
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3r**



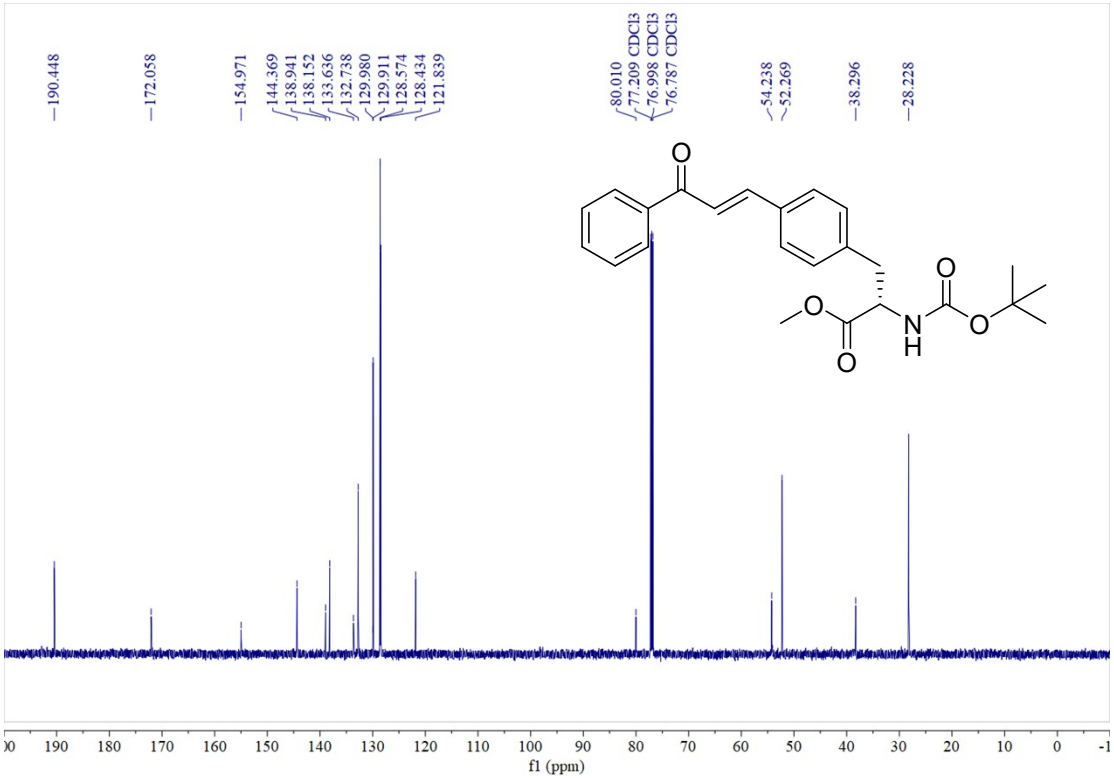
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3r**



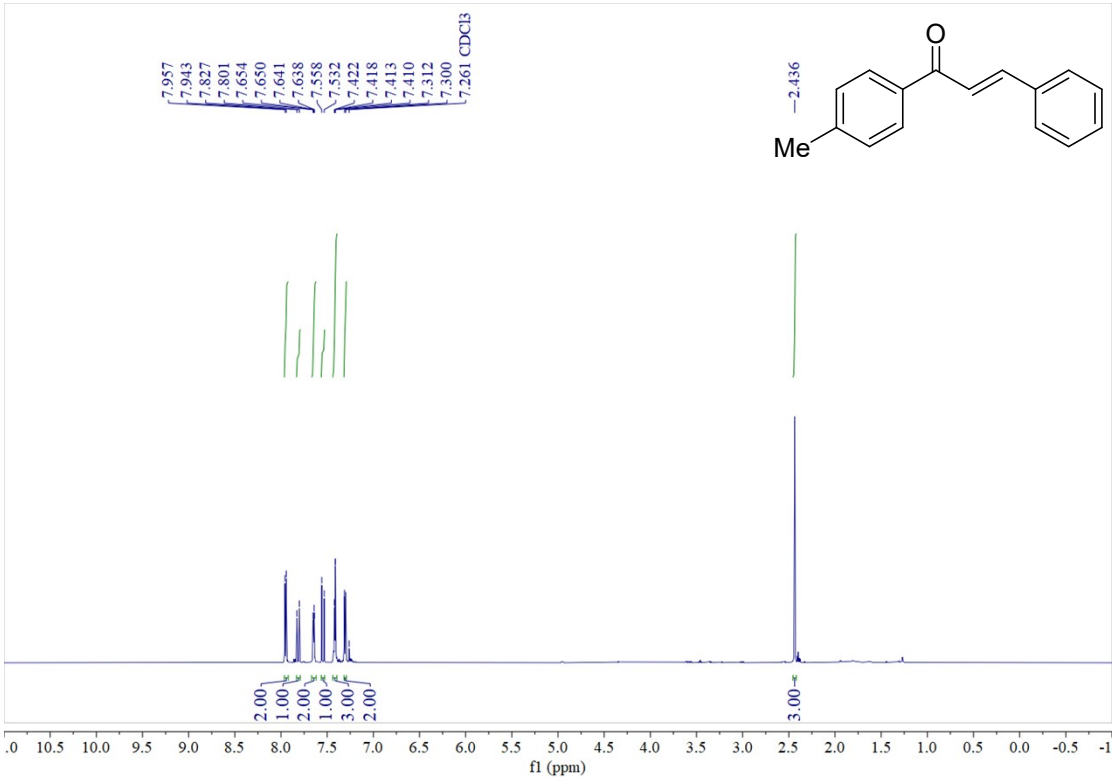
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **3s**



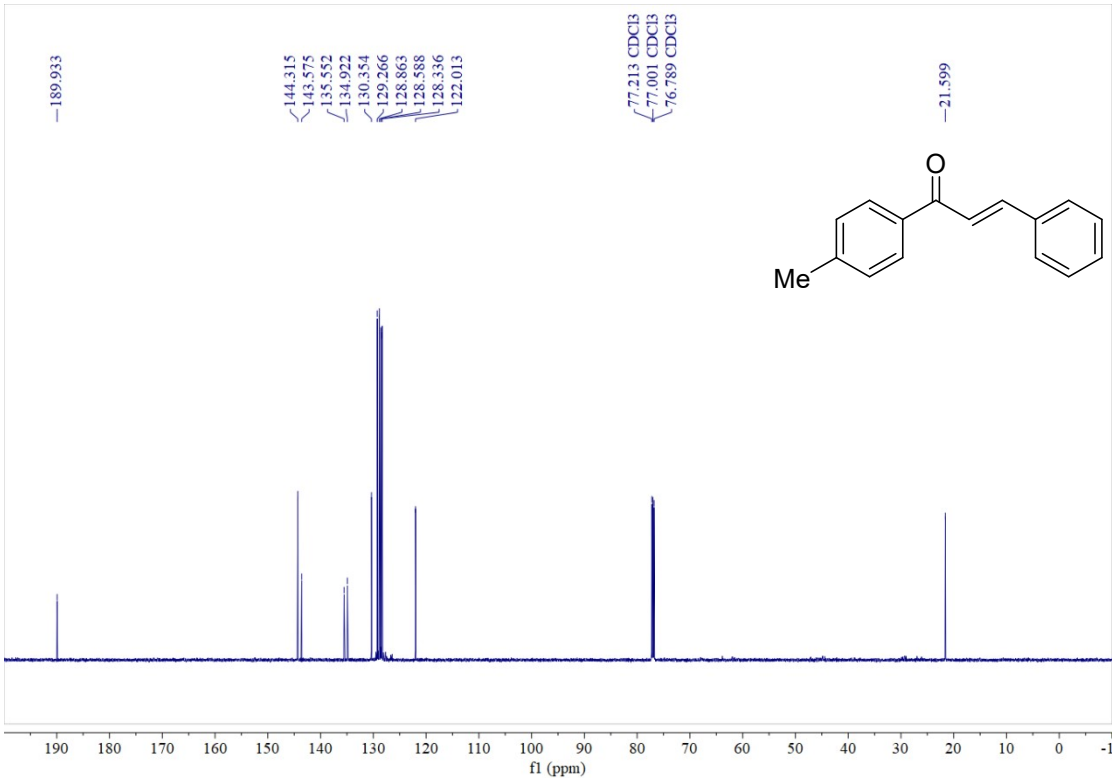
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **3s**



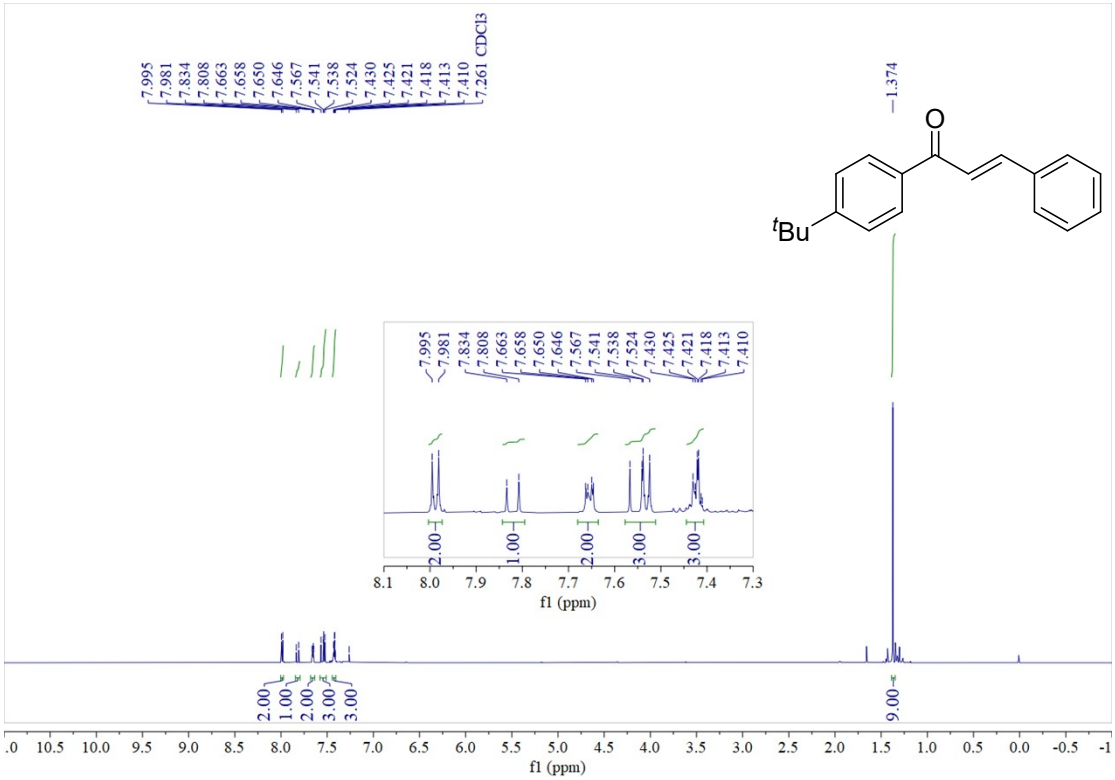
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4a**



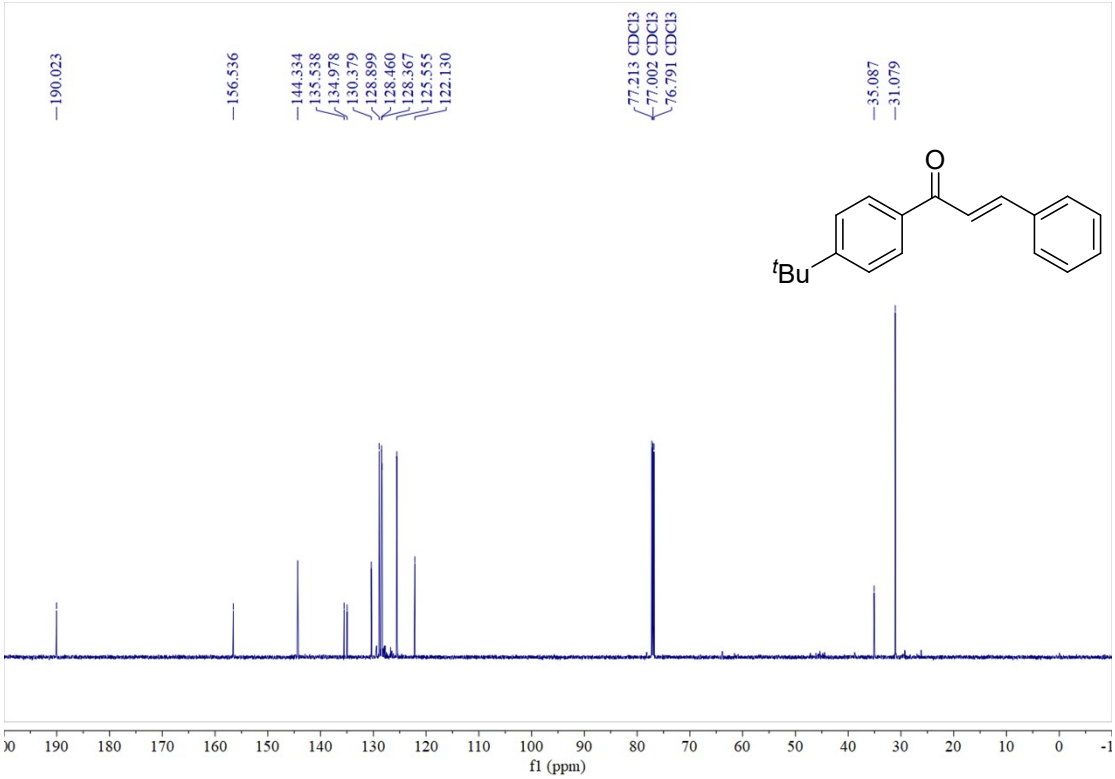
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4a**



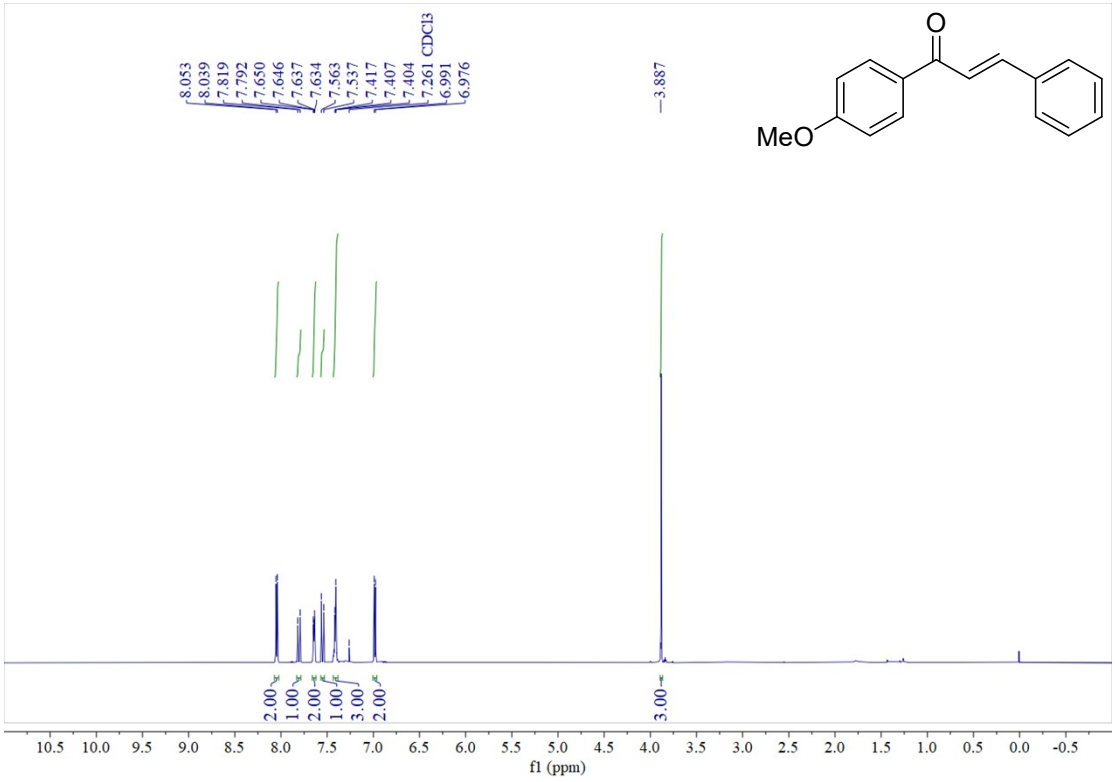
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4b**



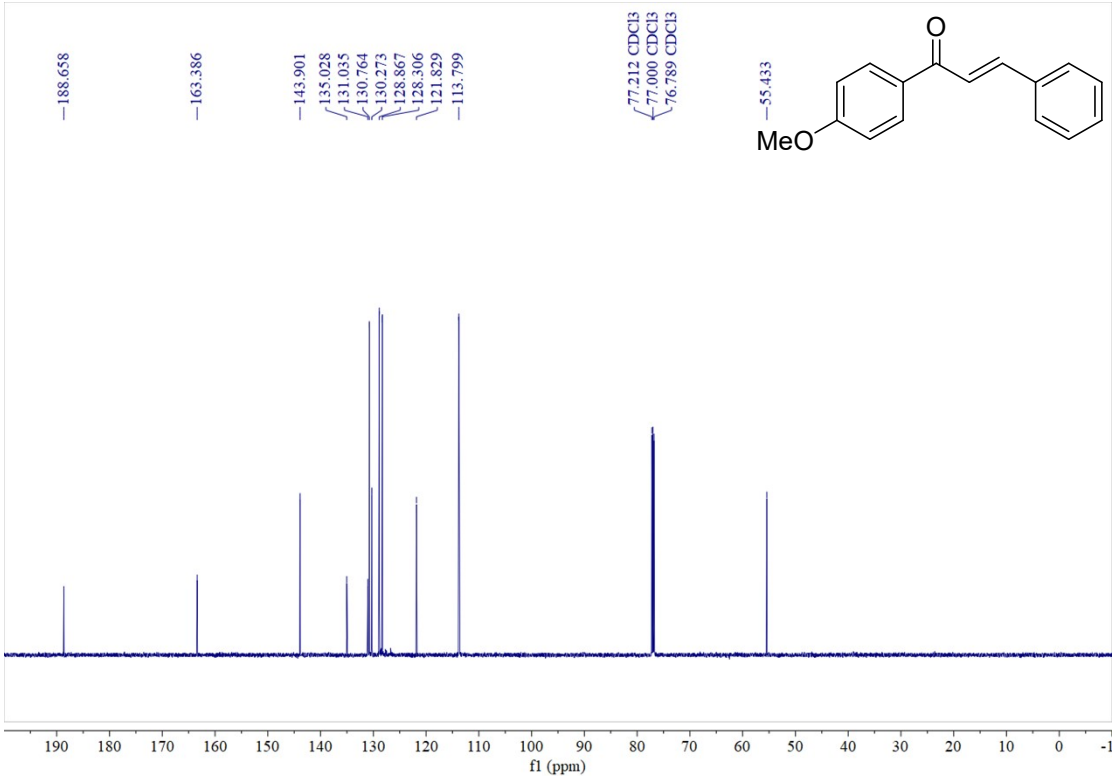
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4b**



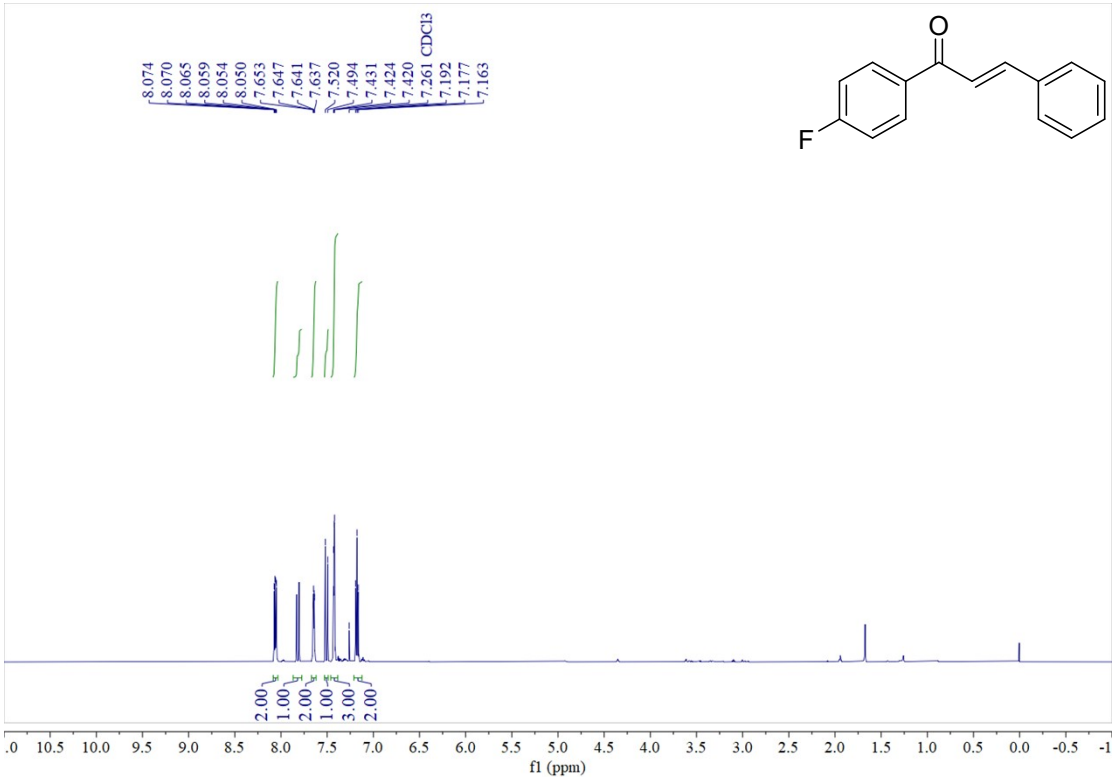
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4c**



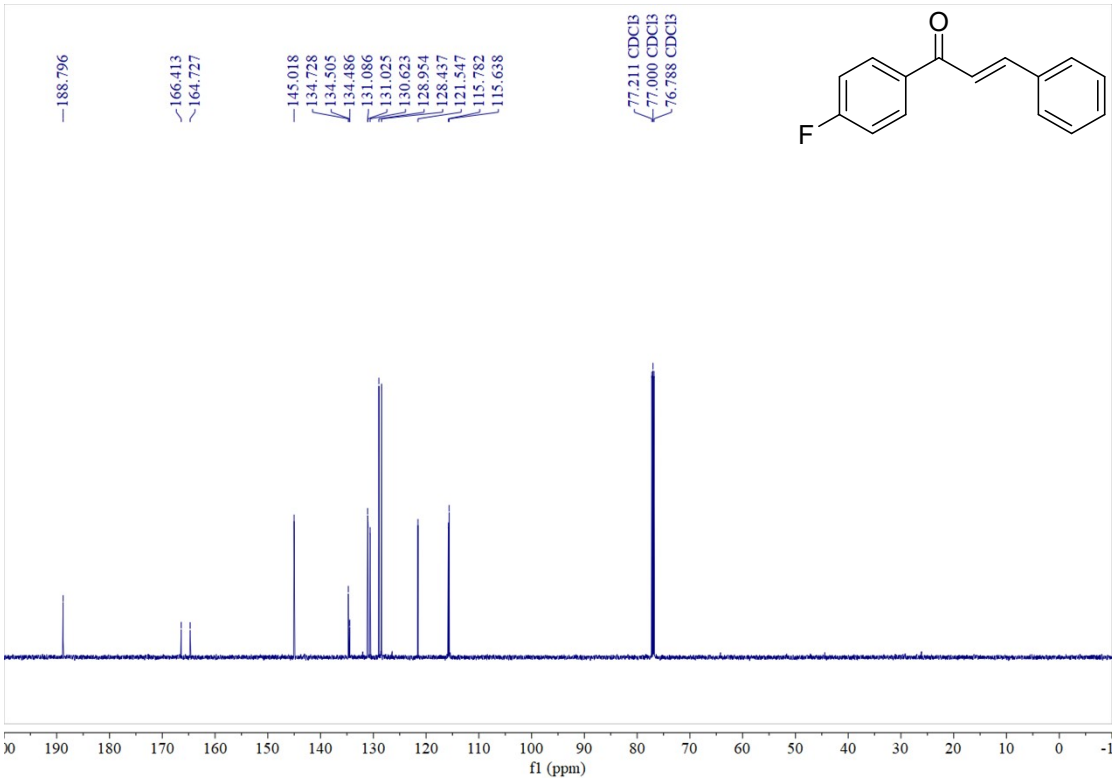
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4c**



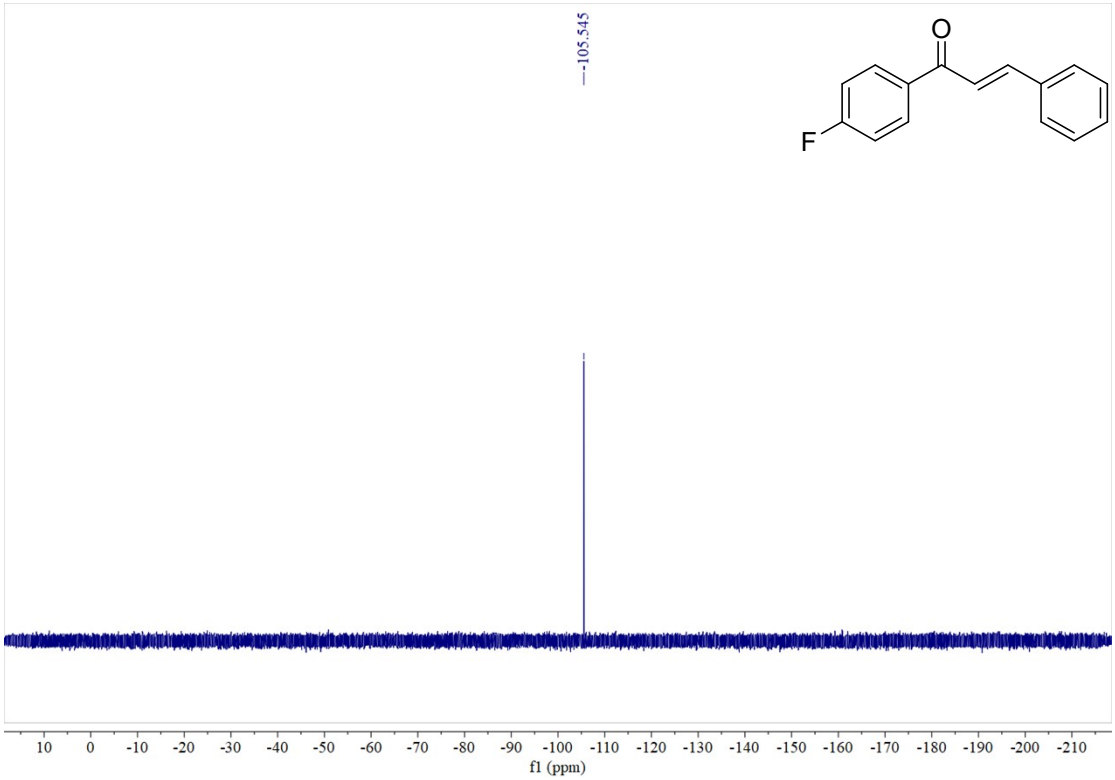
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4d**



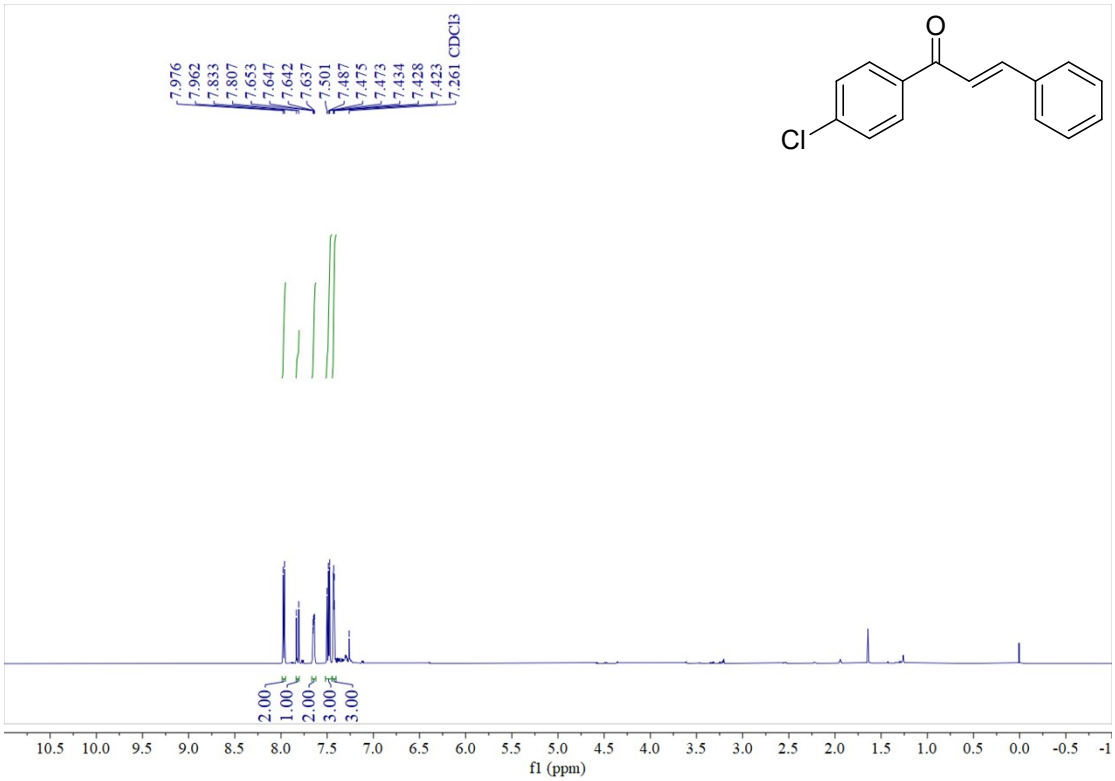
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4d**



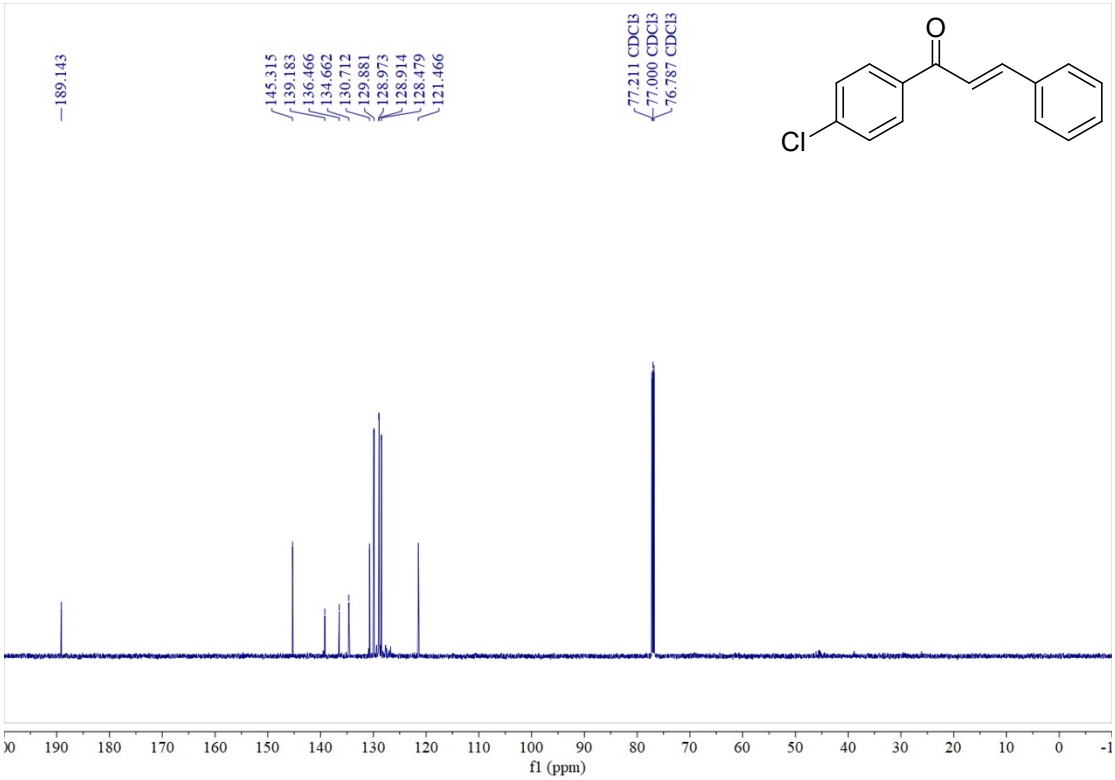
^{19}F NMR (565 MHz, $CDCl_3$) Spectrum of **4d**



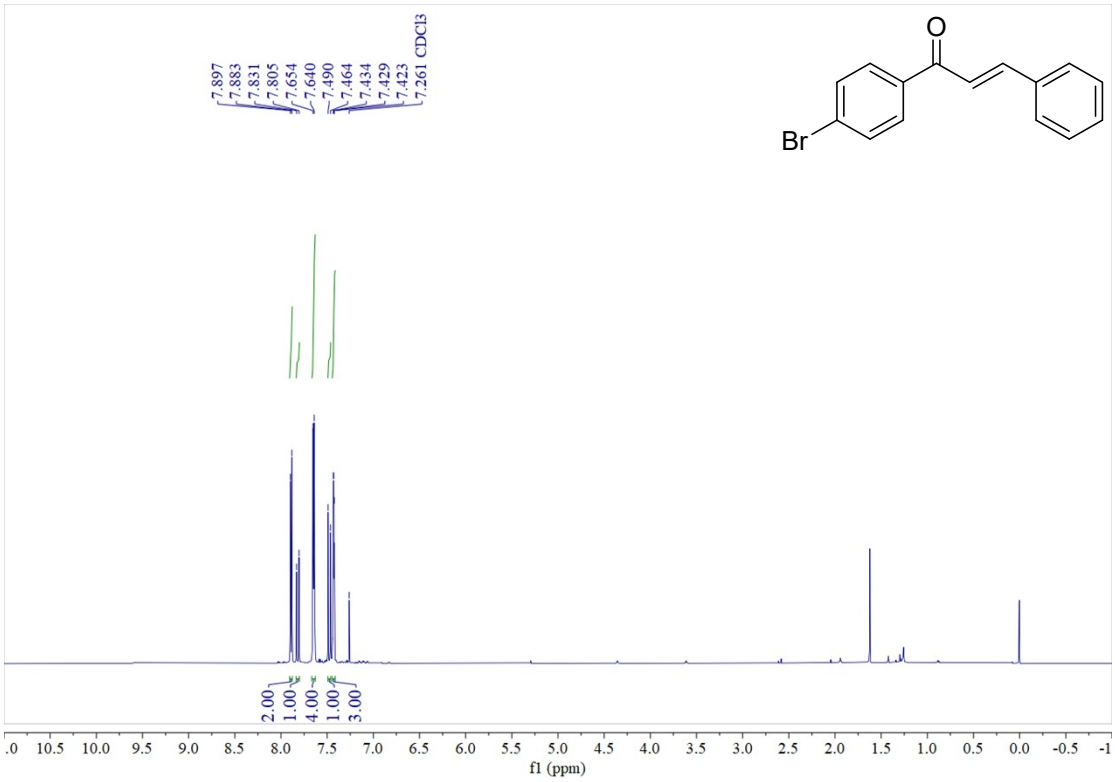
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4e**



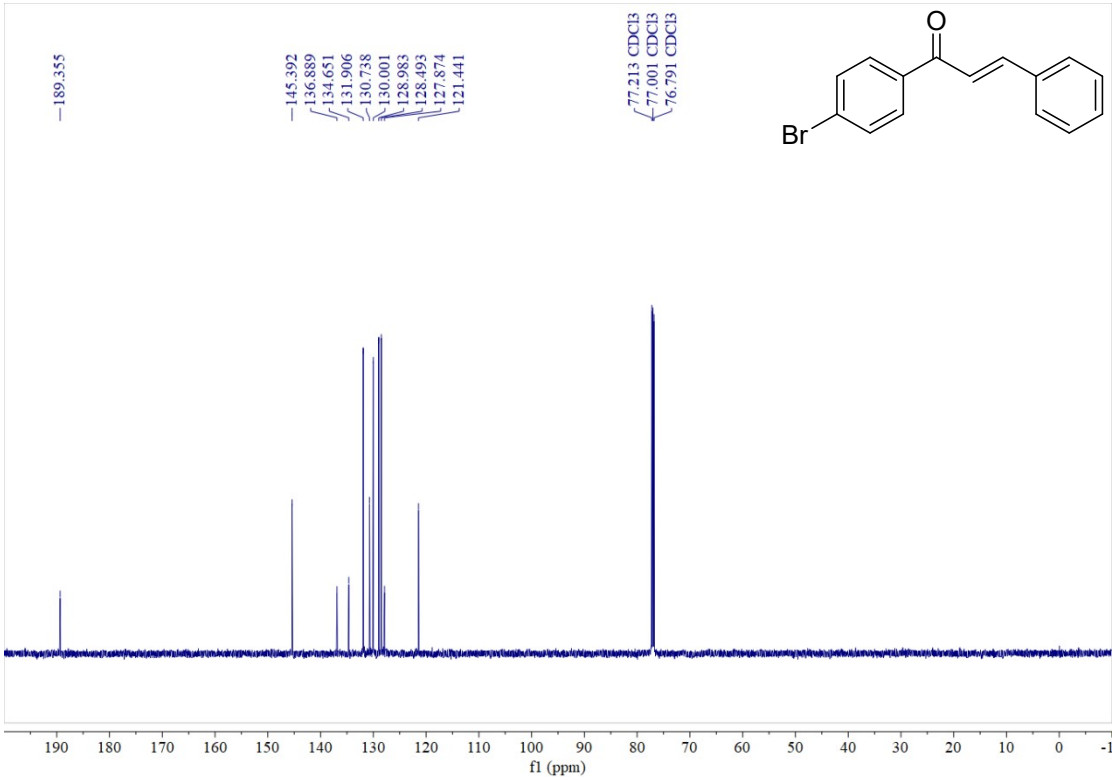
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4e**



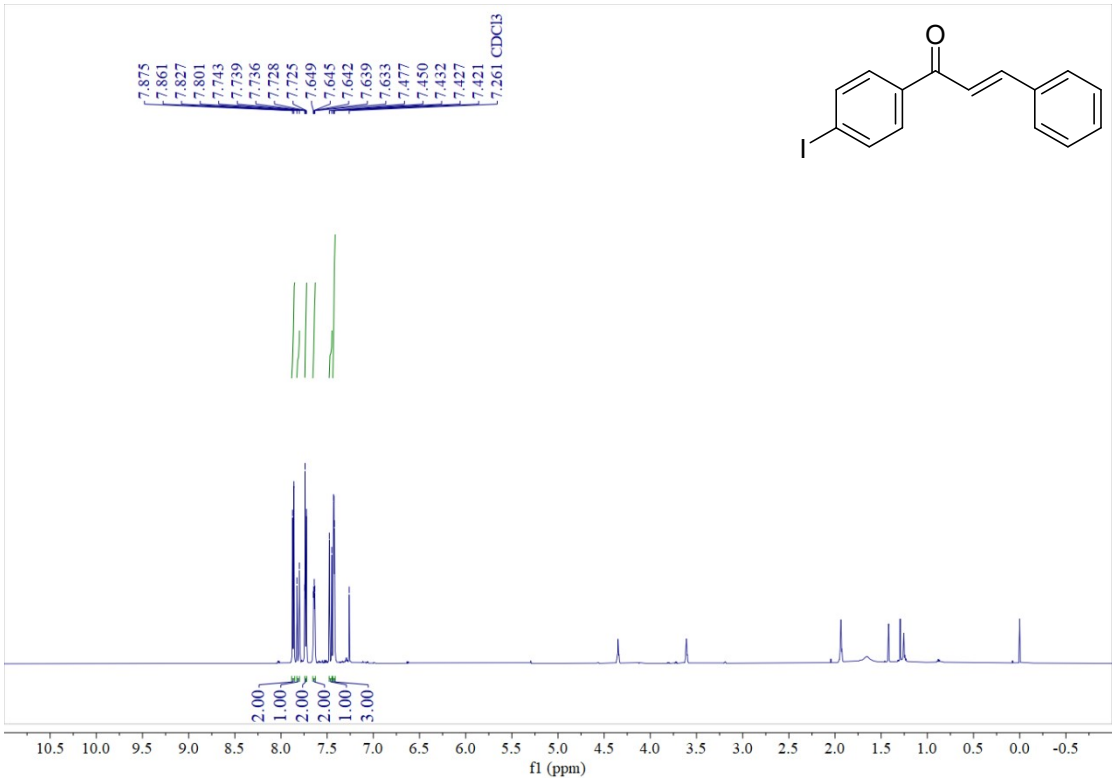
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4f**



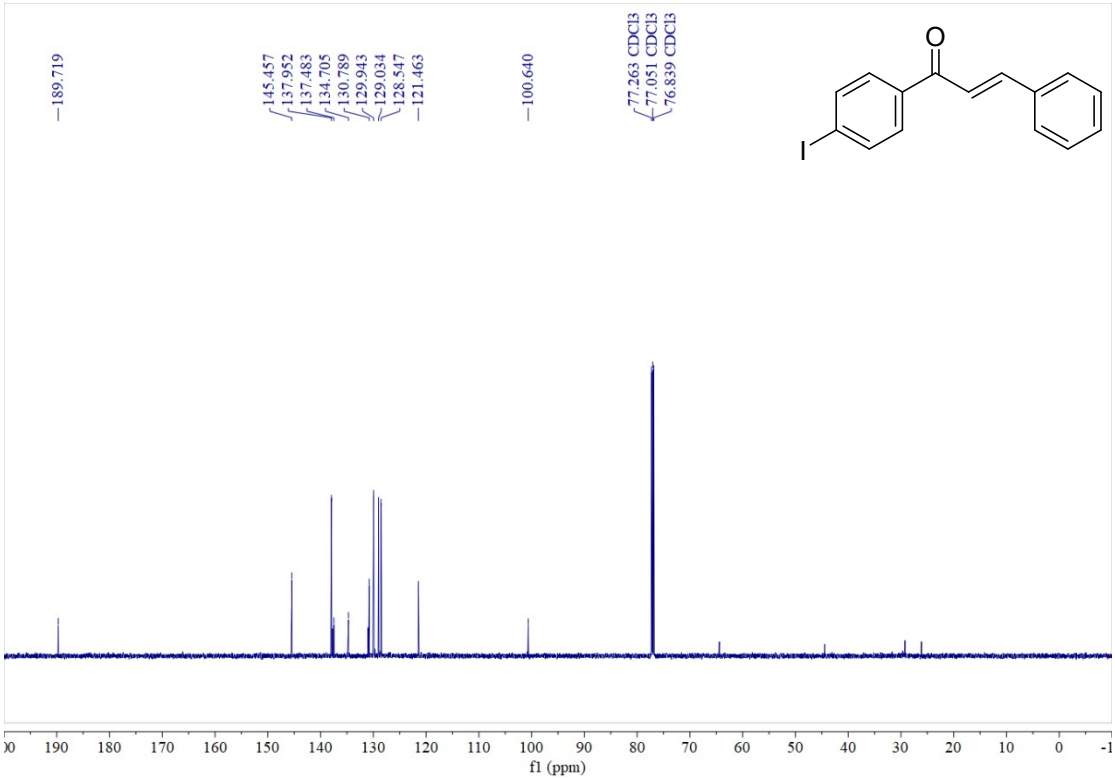
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4f**



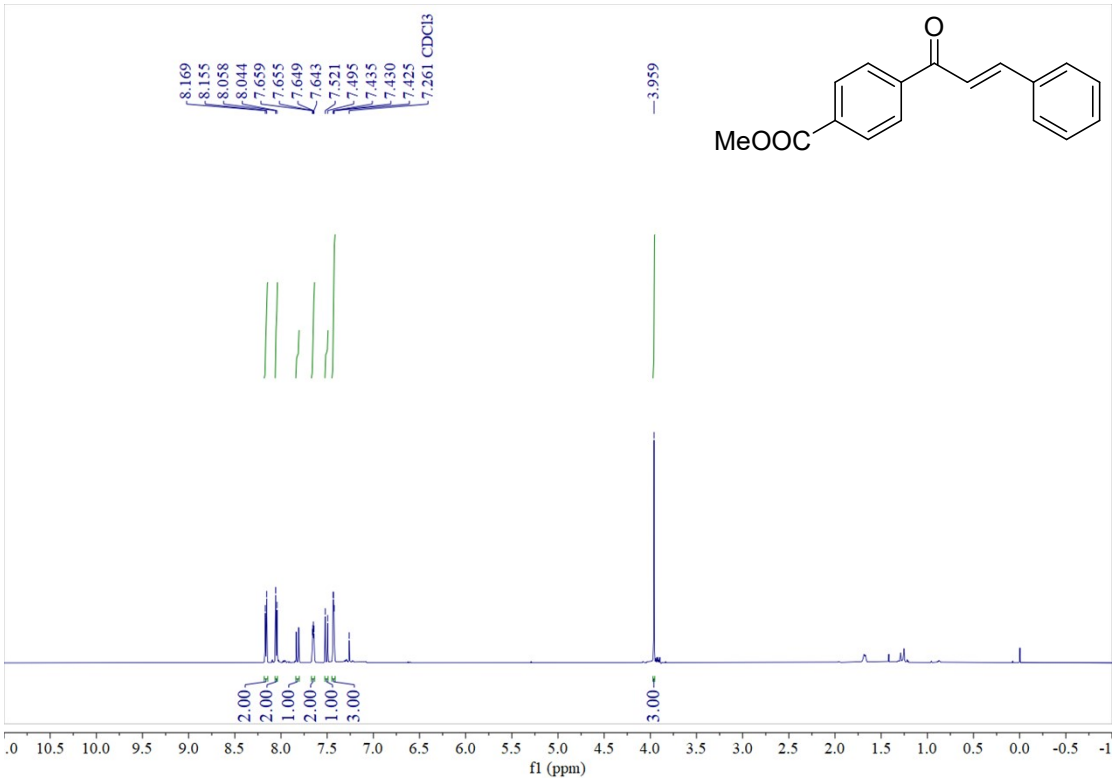
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4g**



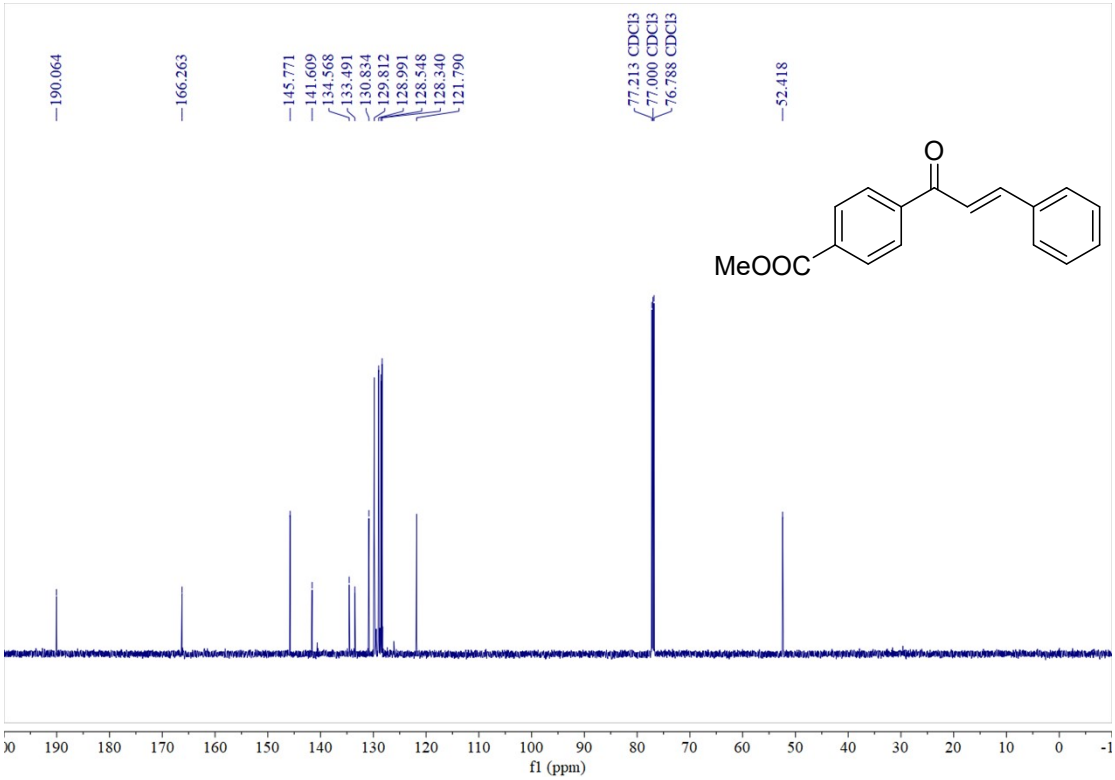
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4g**



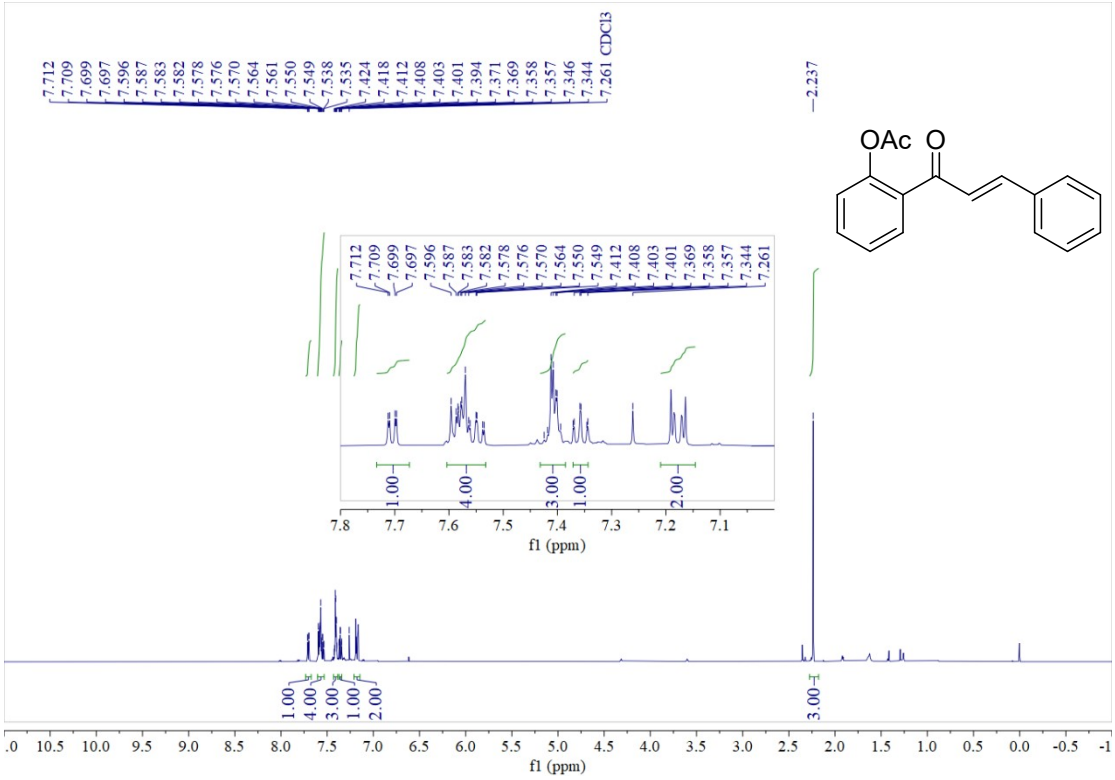
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4h**



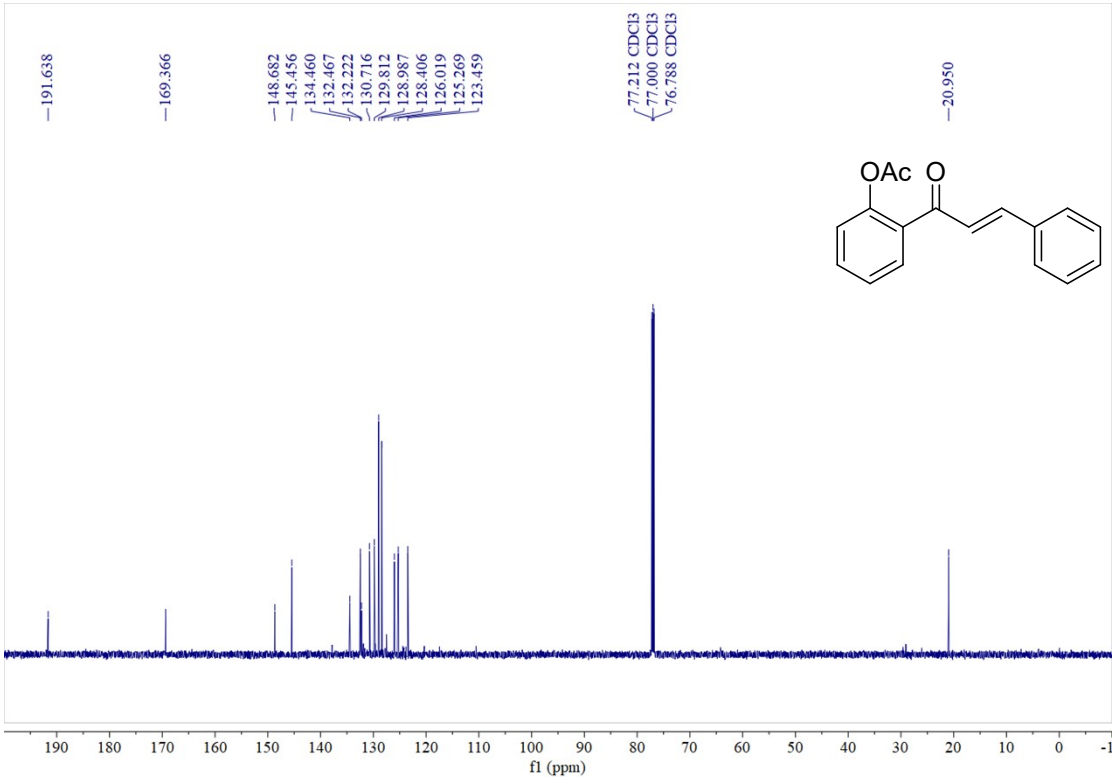
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4h**



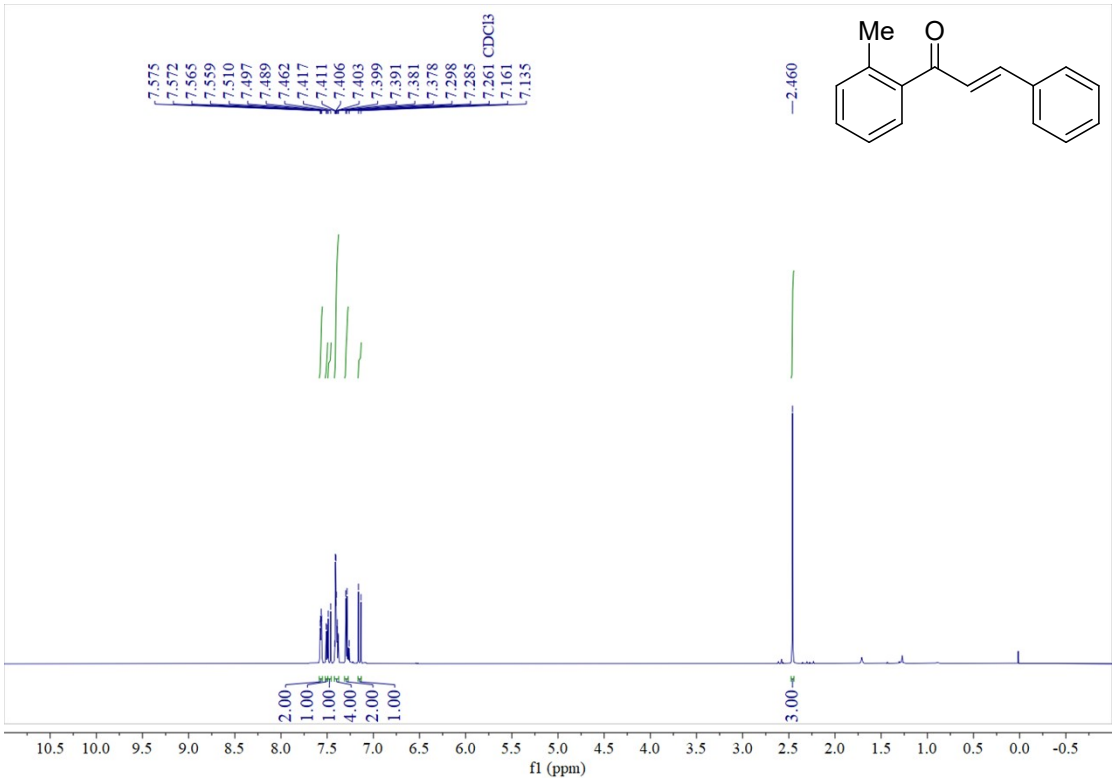
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4i**



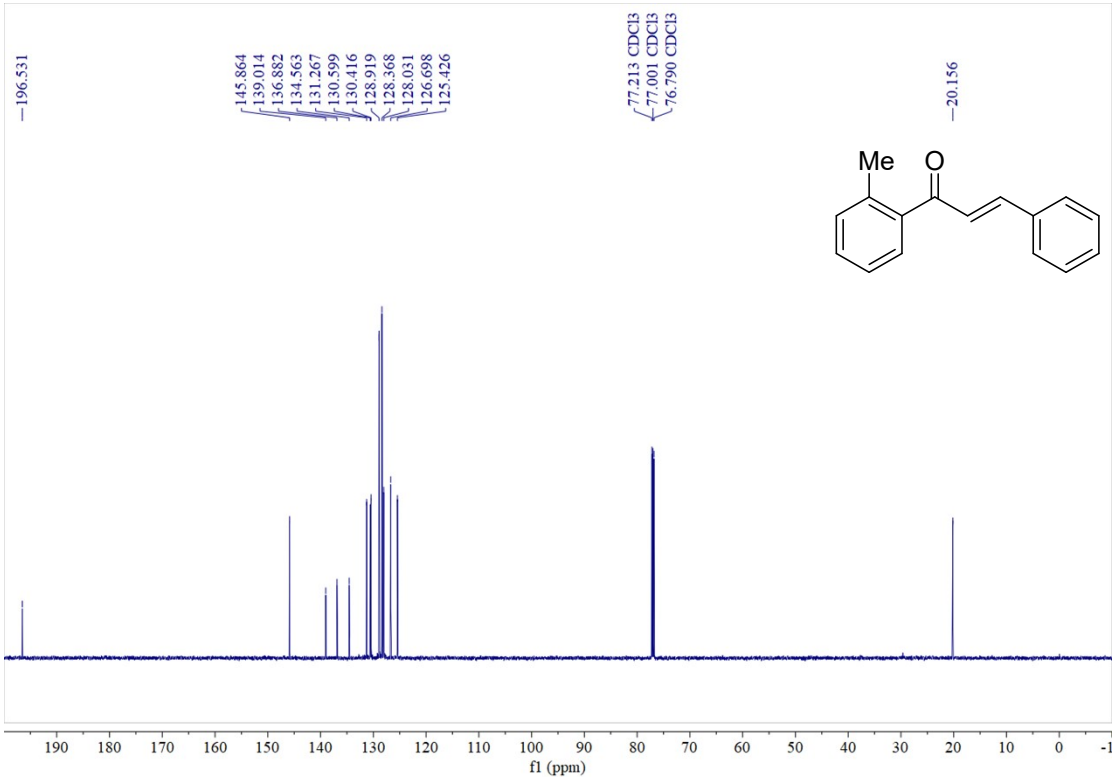
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4i**



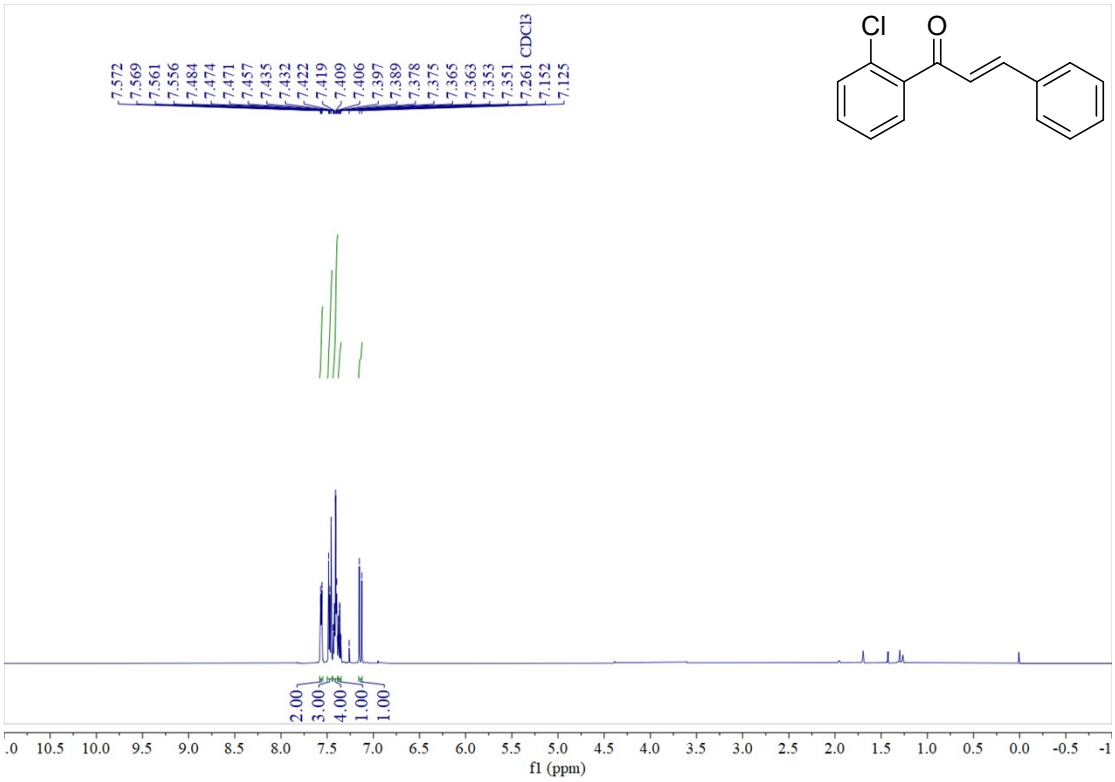
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4j**



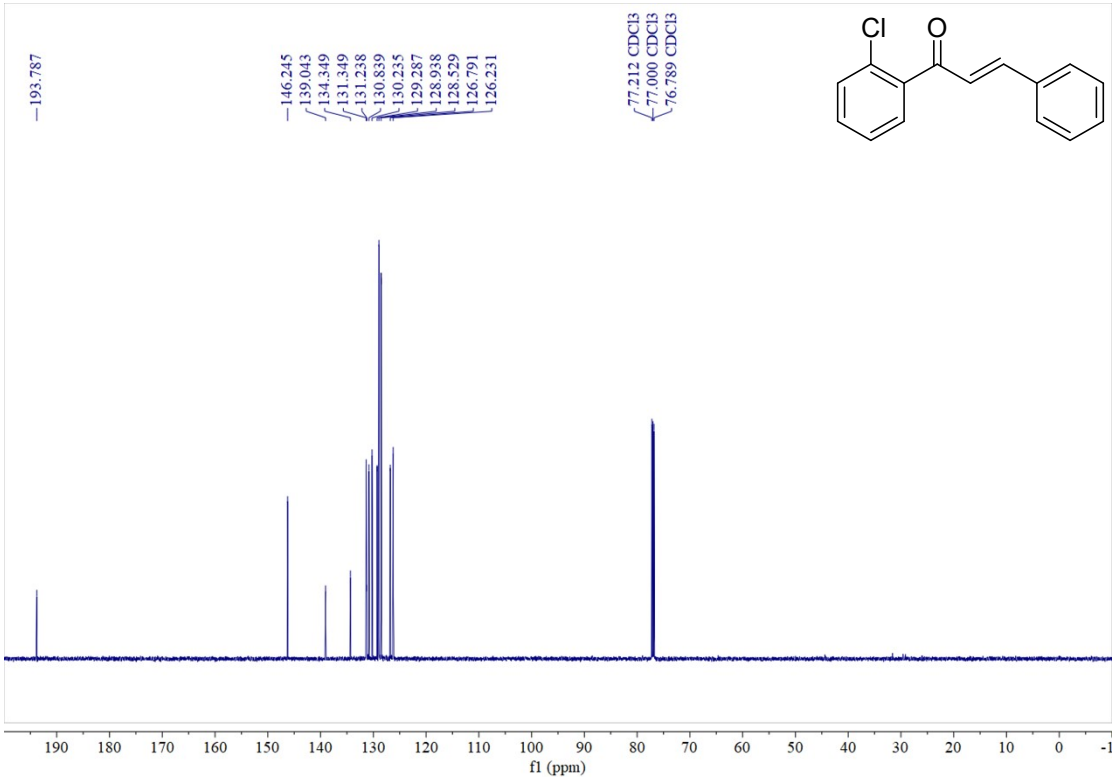
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4j**



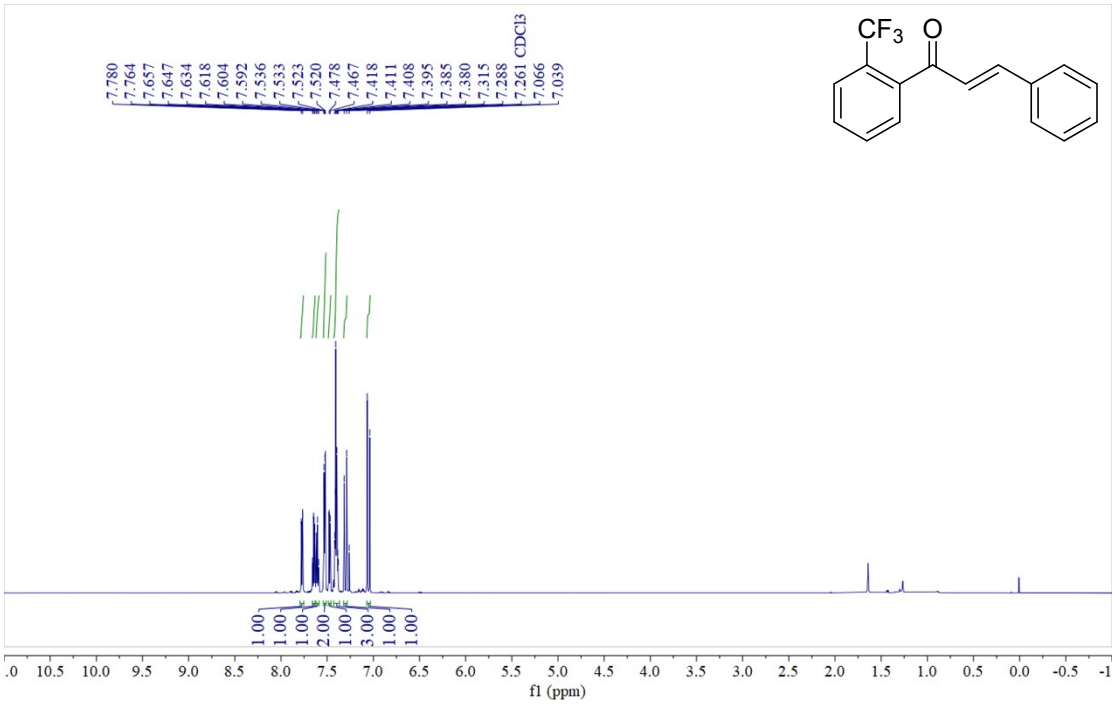
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4k**



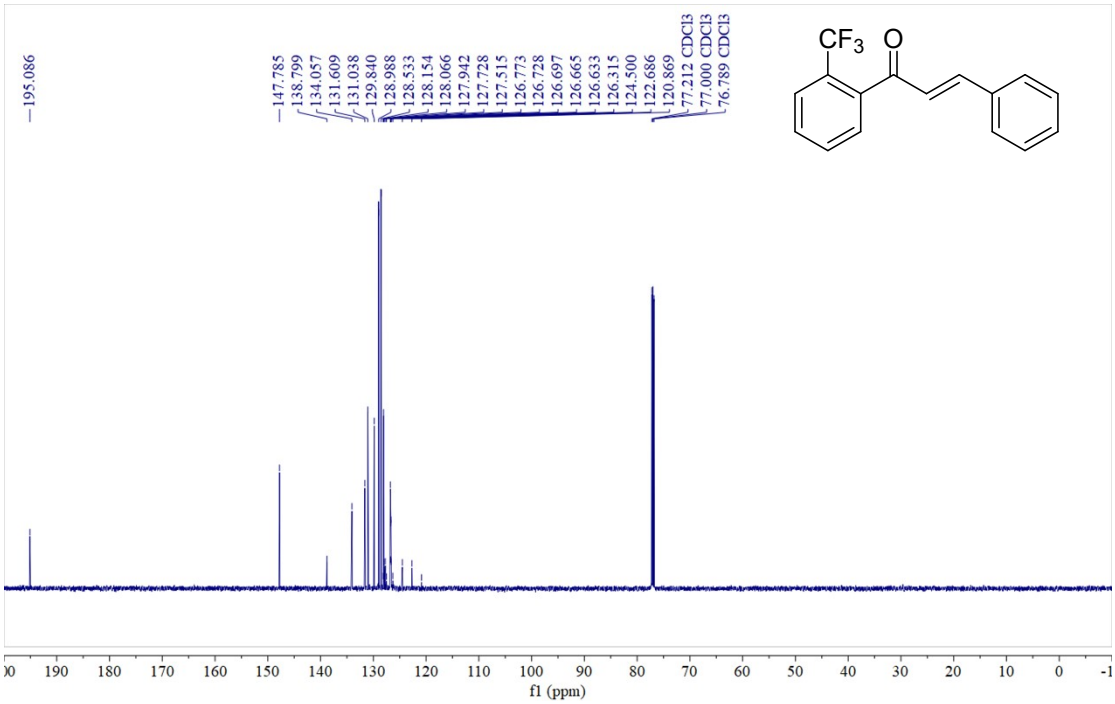
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4k**



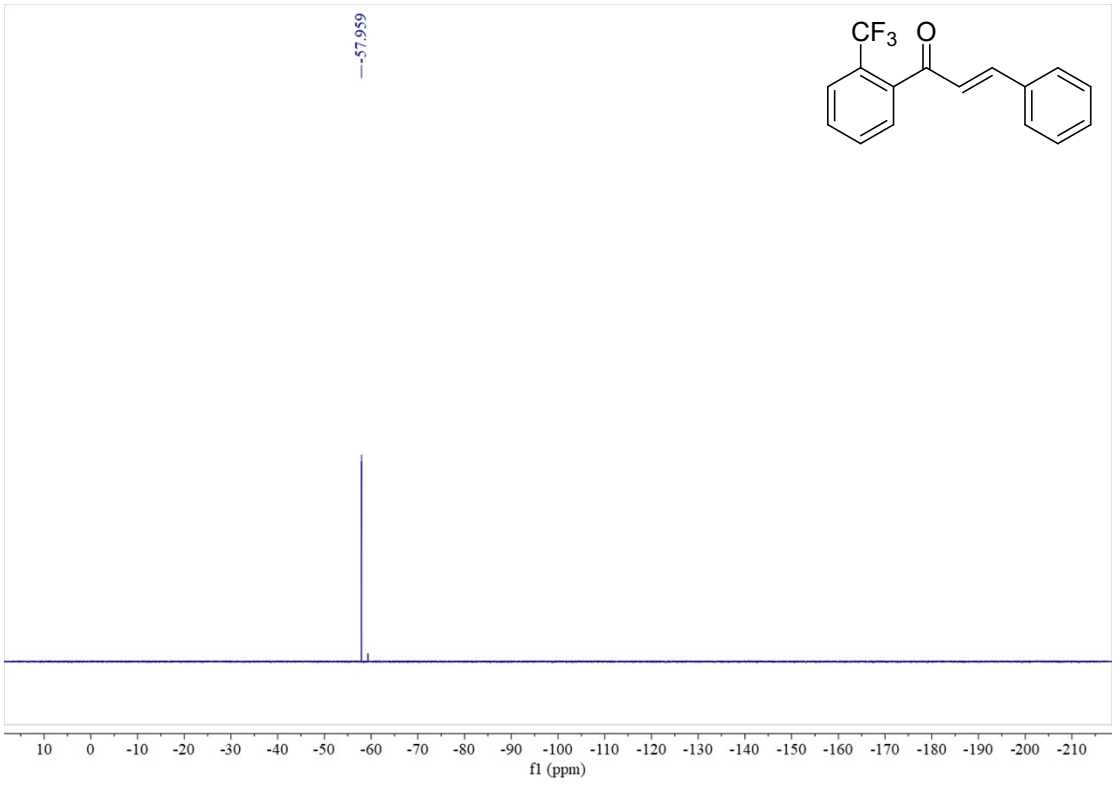
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4l**



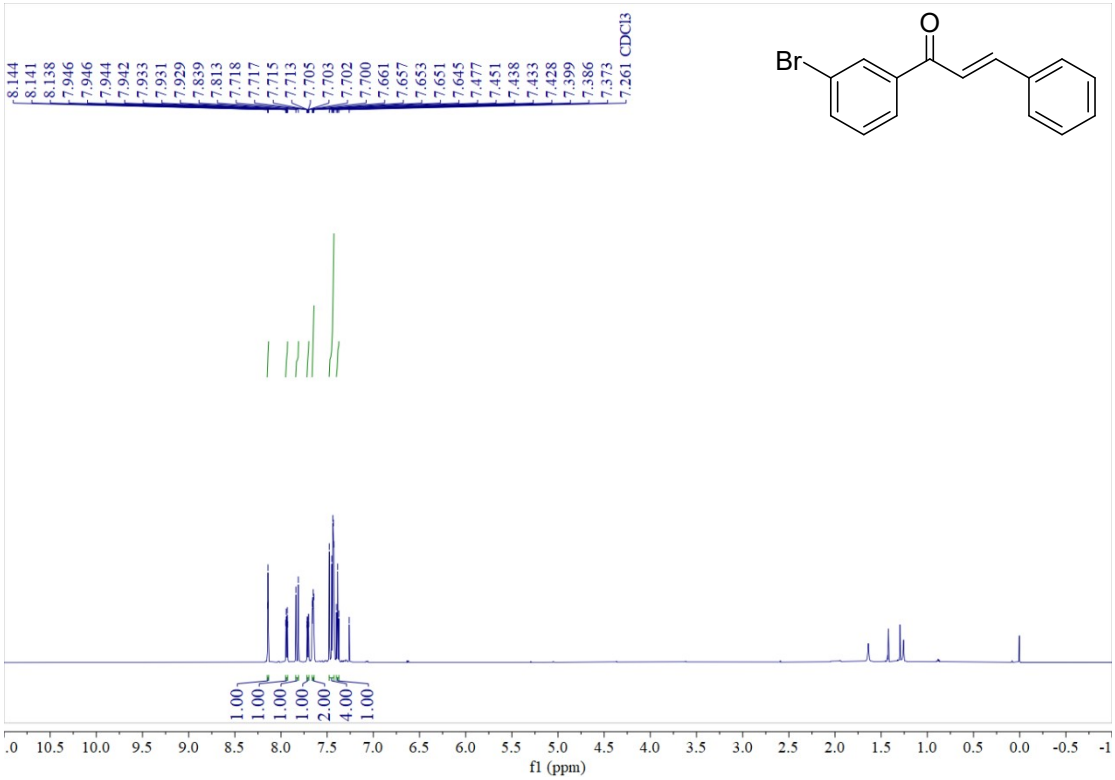
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4l**



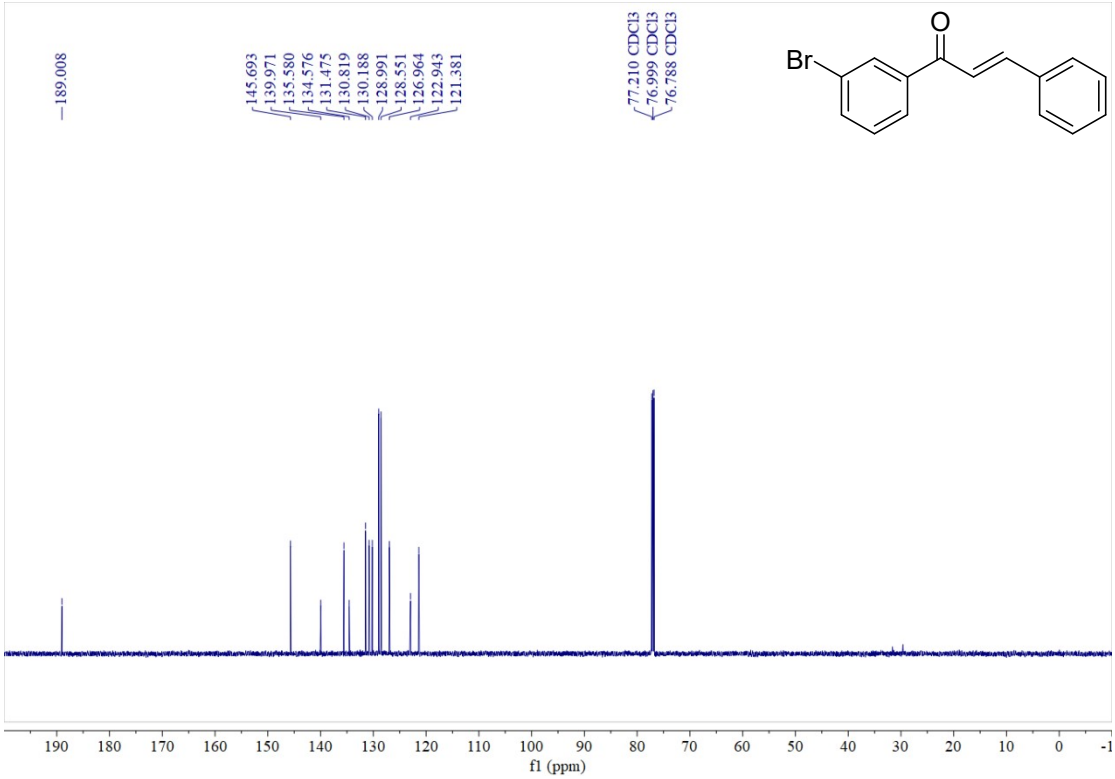
^{19}F NMR (565 MHz, $CDCl_3$) Spectrum of **4l**



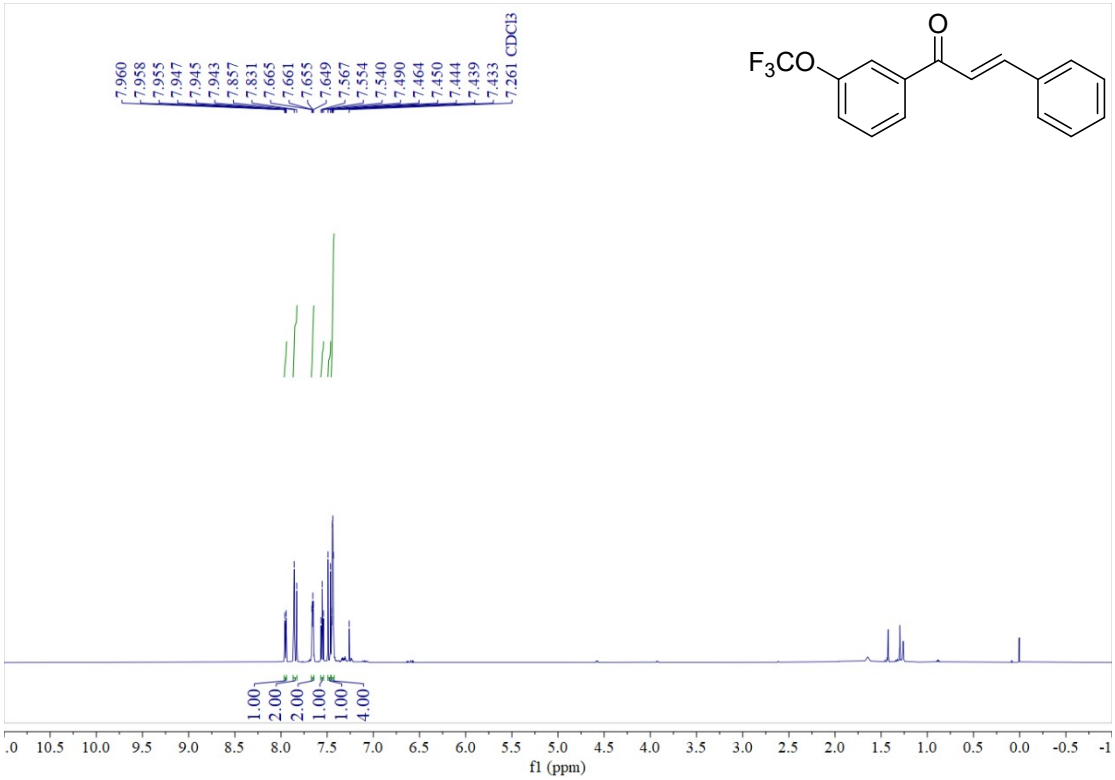
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4m**



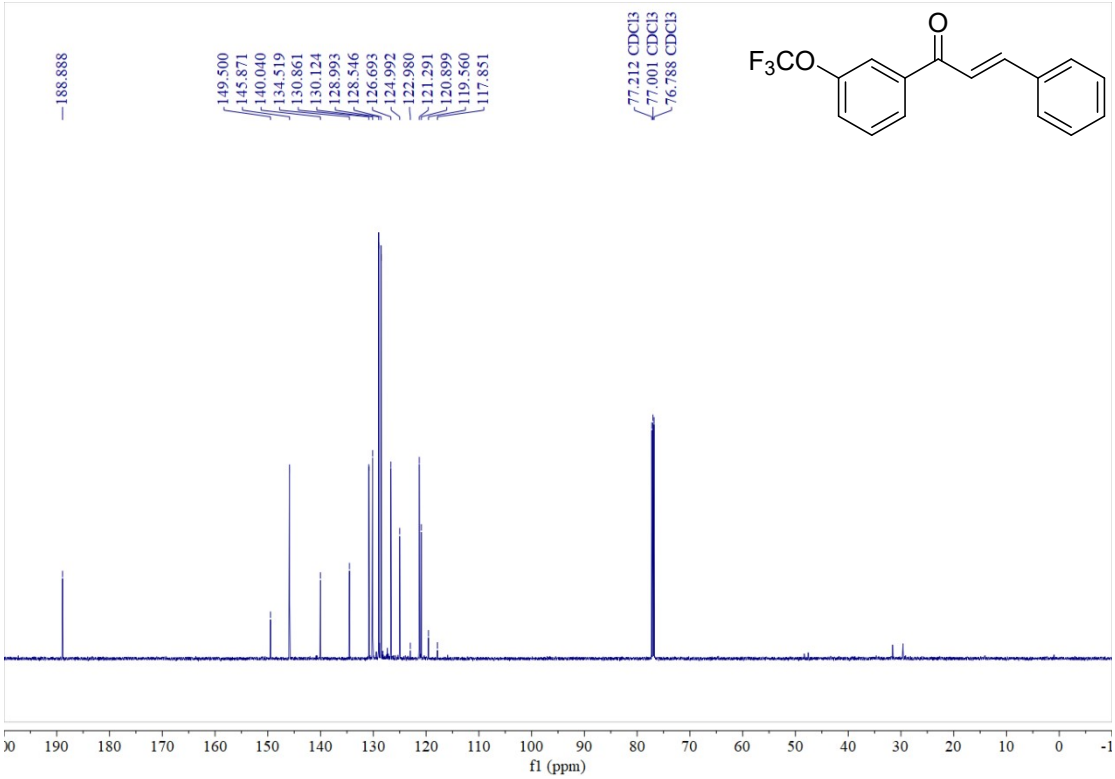
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4m**



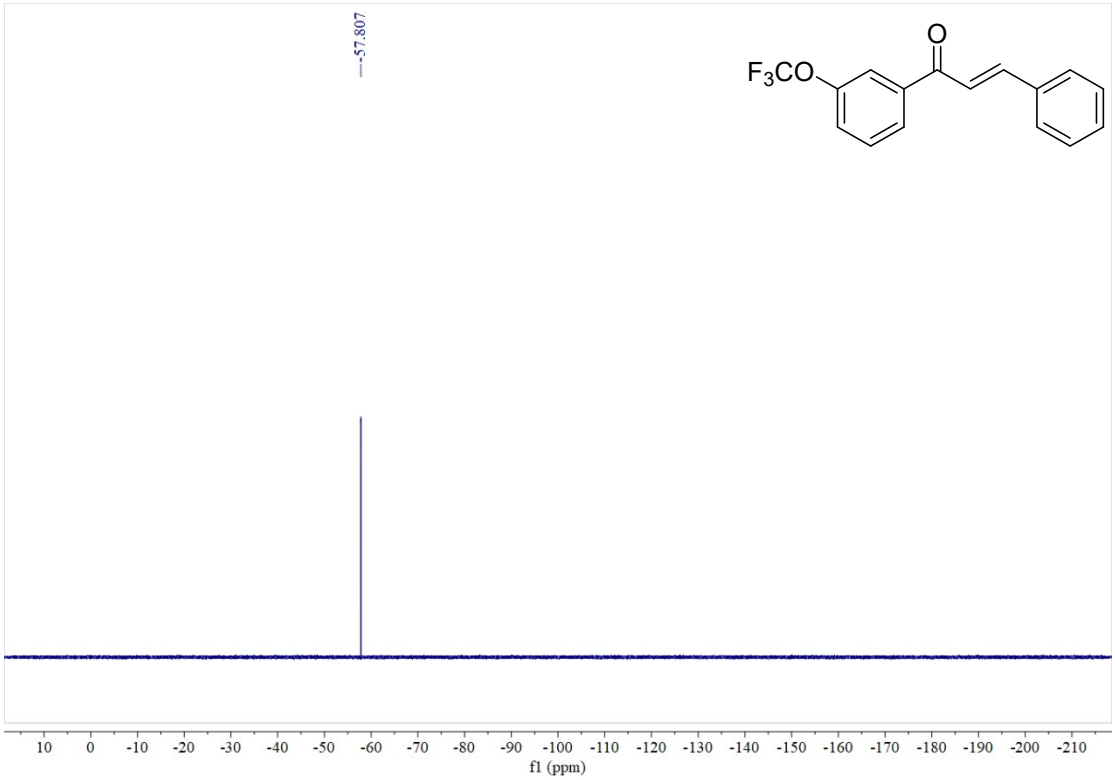
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4n**



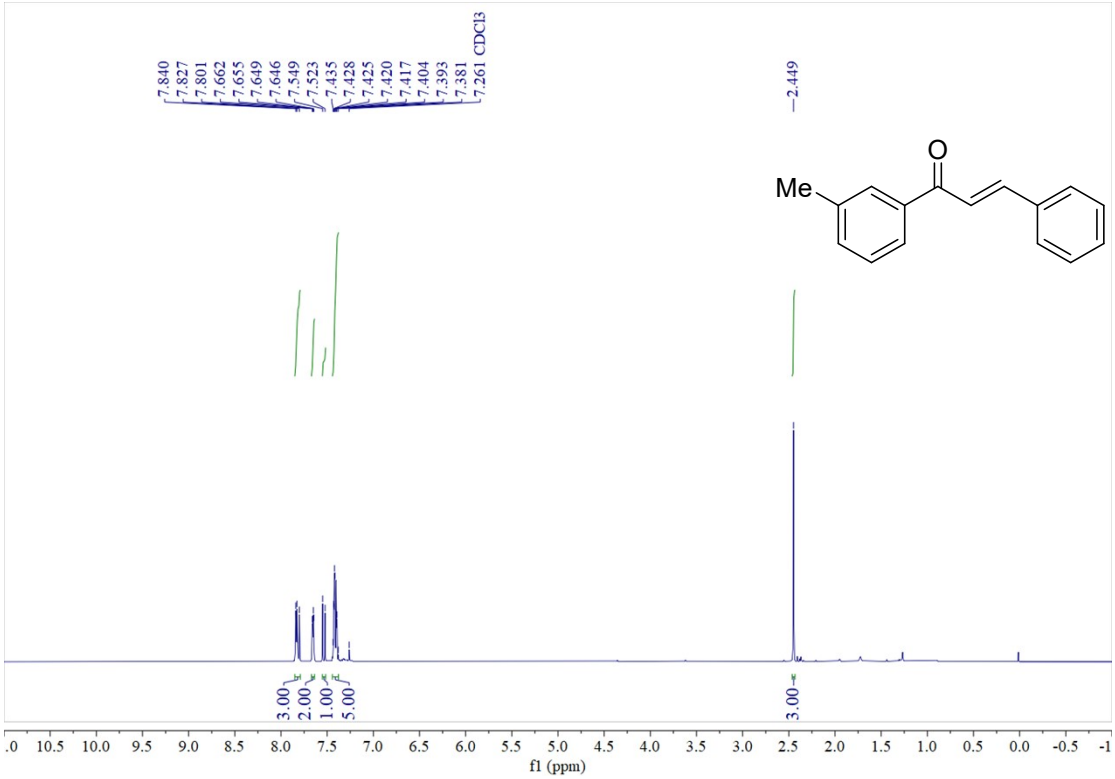
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4n**



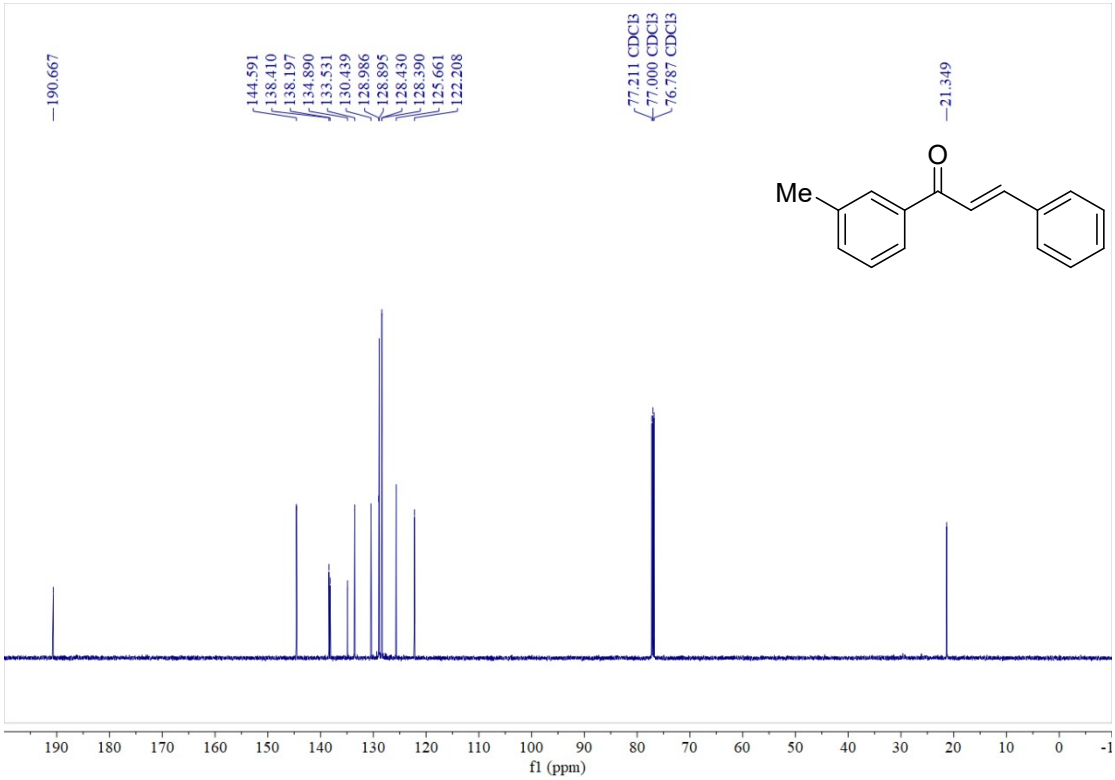
^{19}F NMR (565 MHz, $CDCl_3$) Spectrum of **4n**



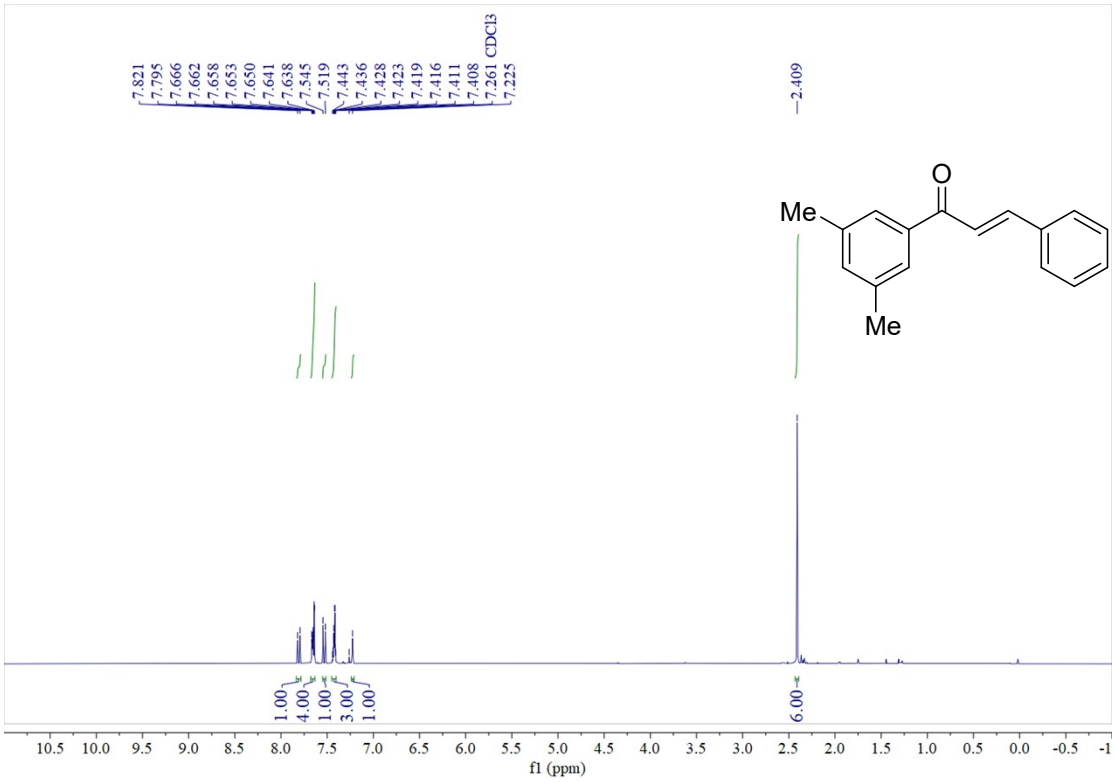
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4o**



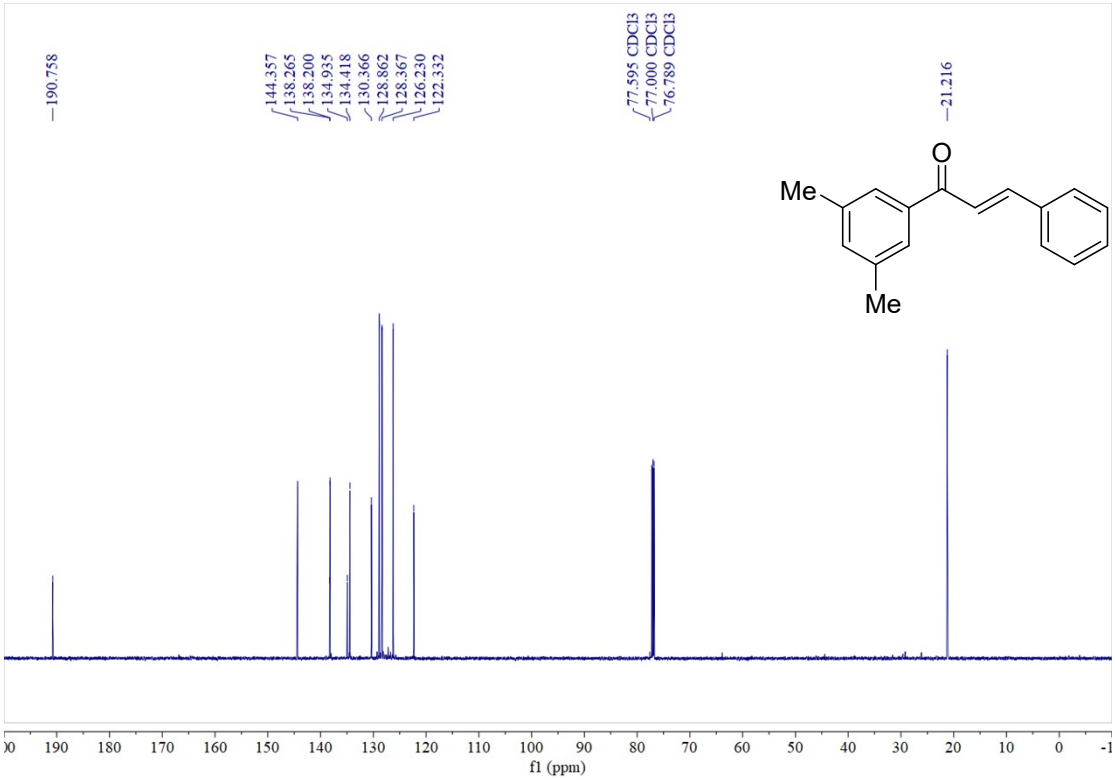
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4o**



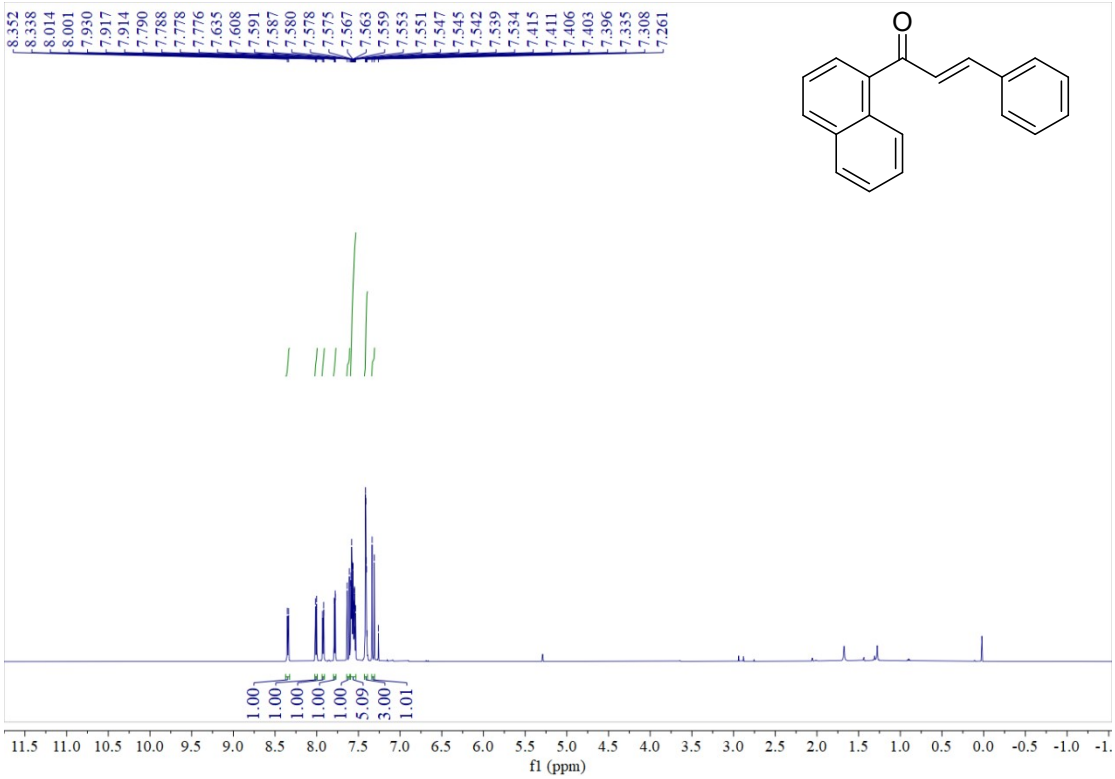
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4p**



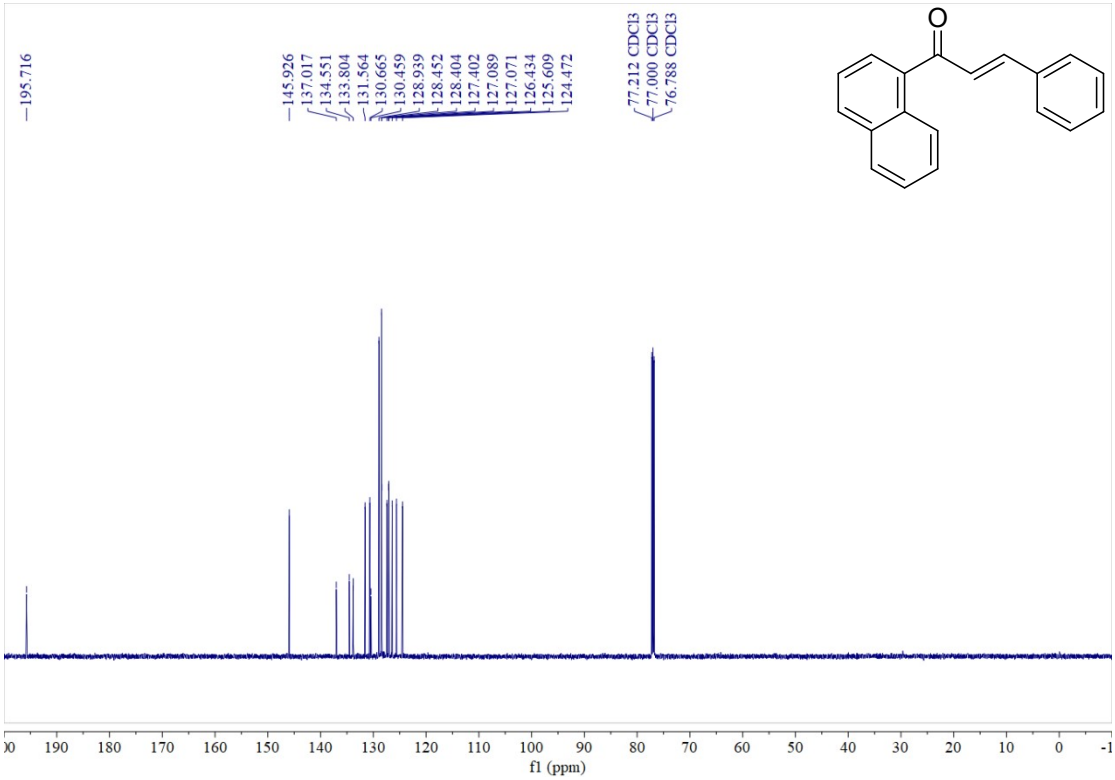
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4p**



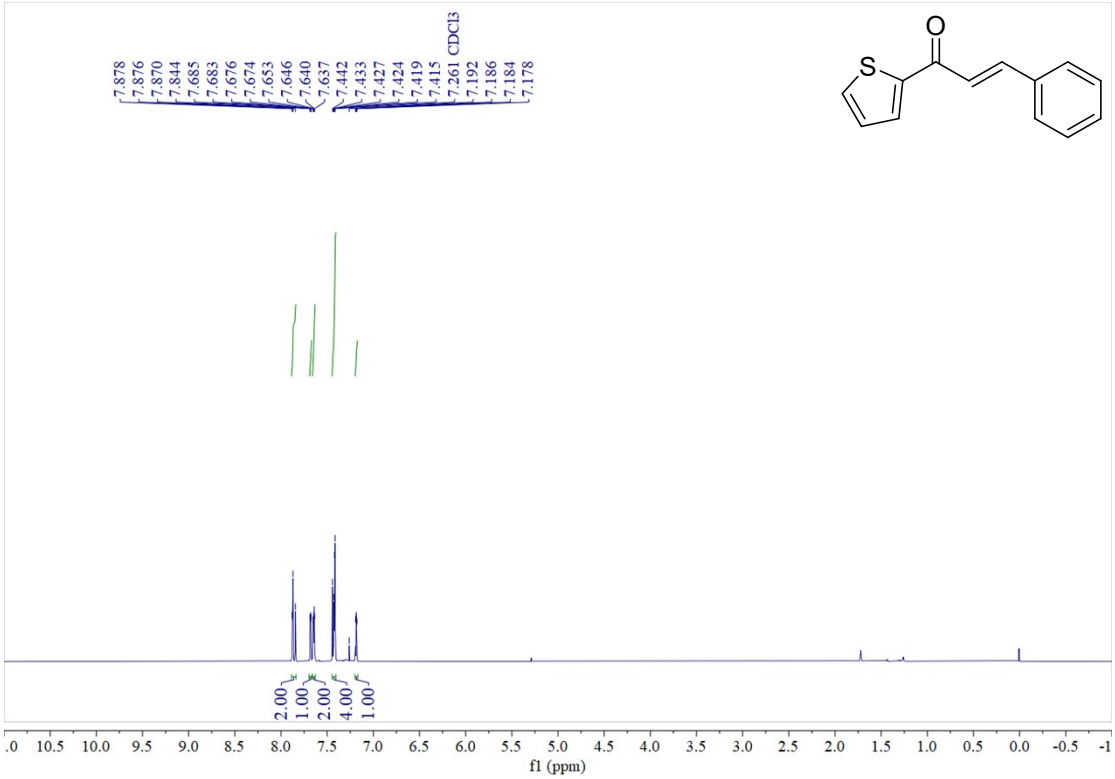
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4q**



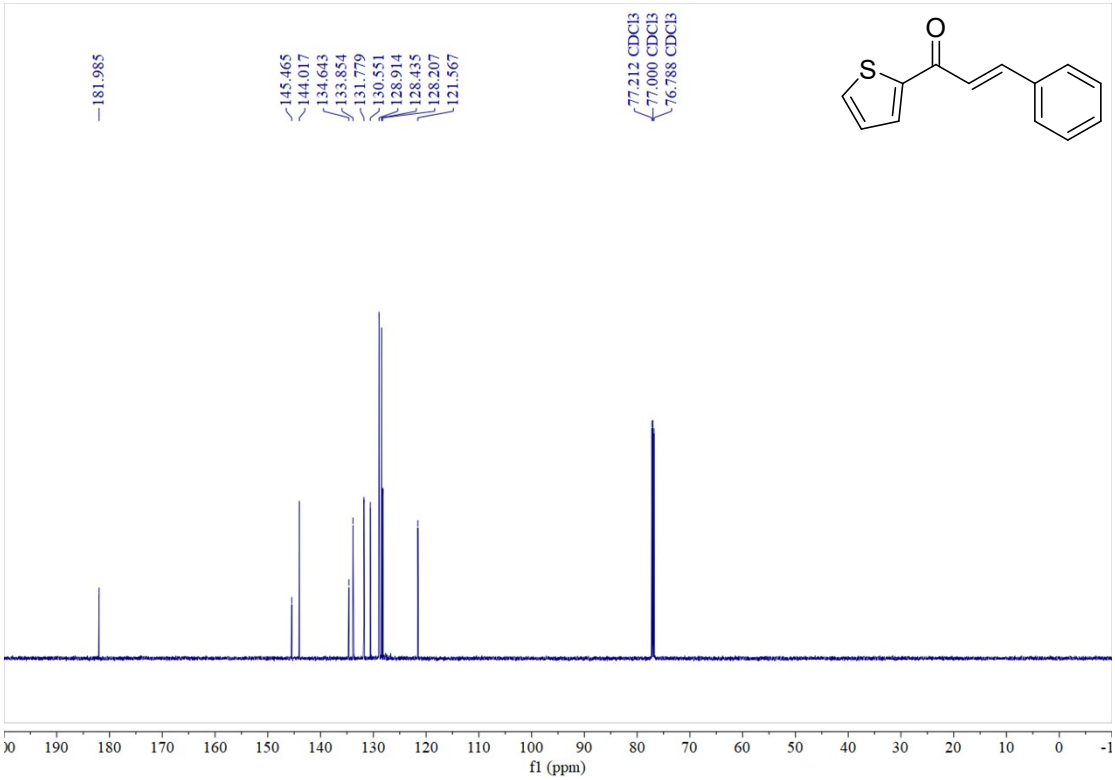
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4q**



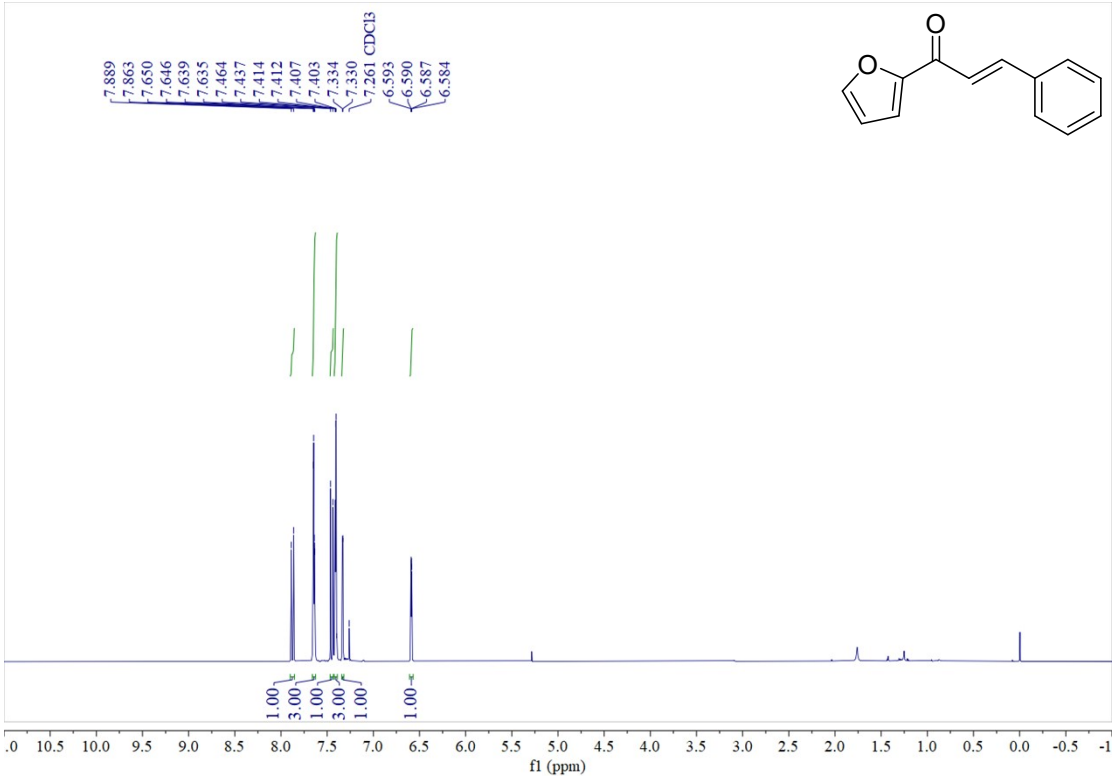
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4r**



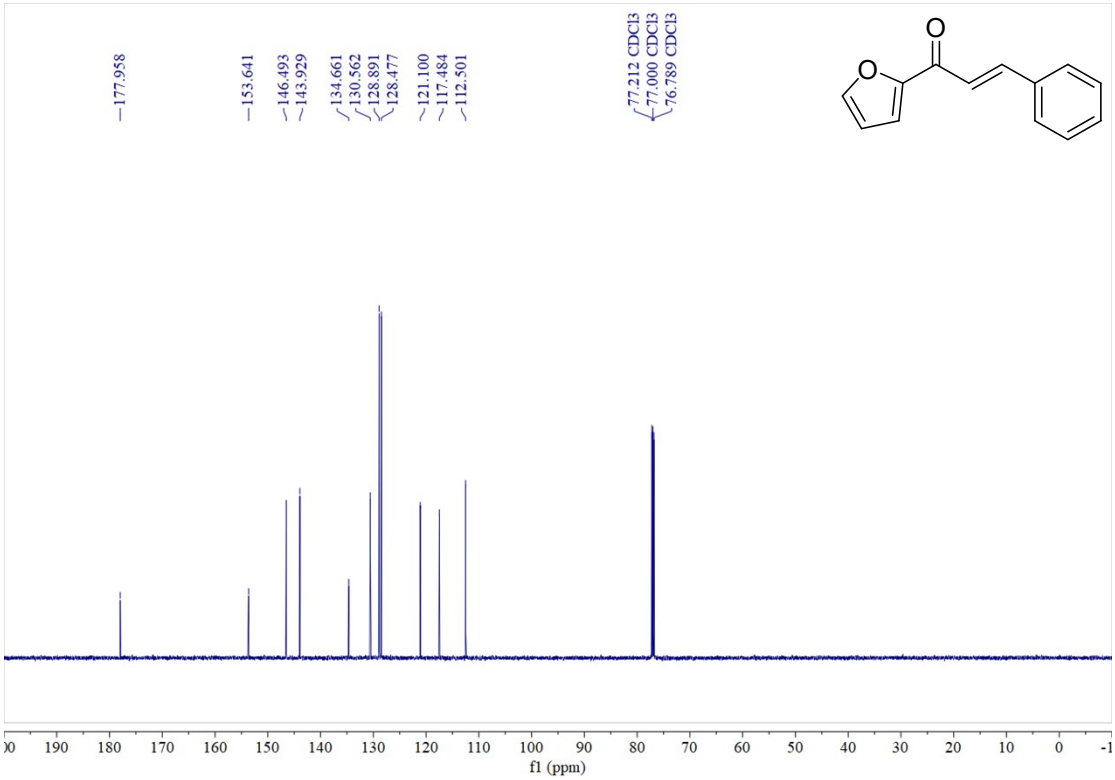
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4r**



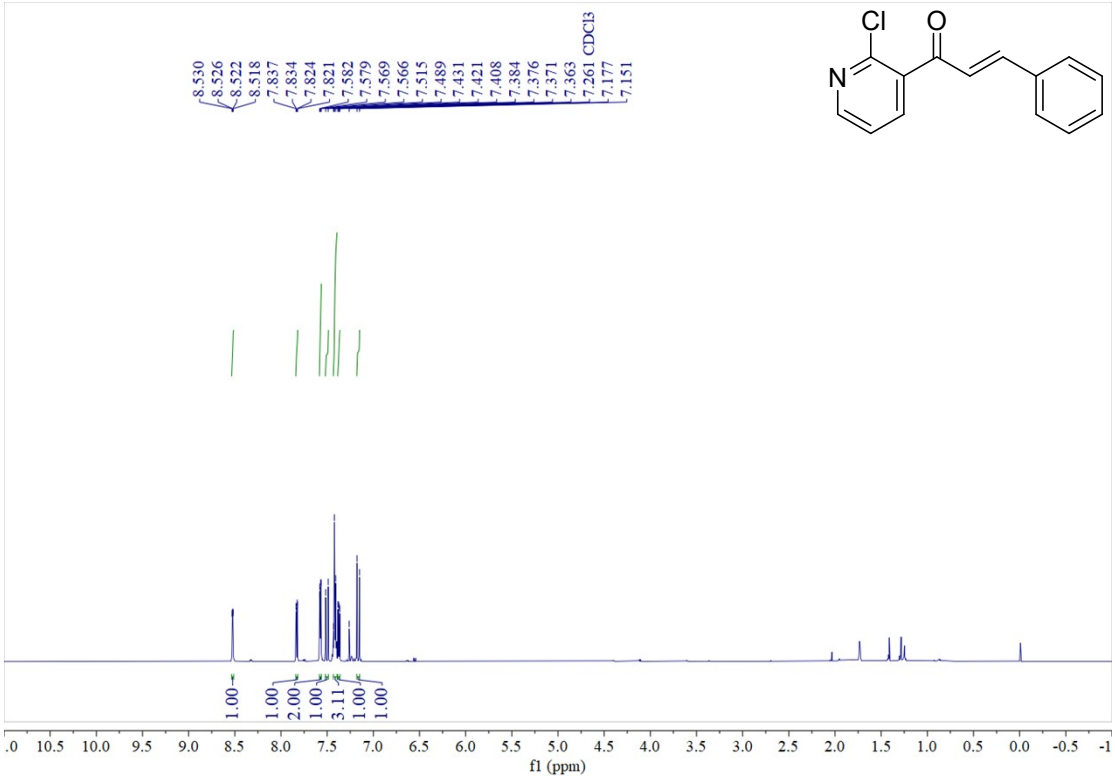
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4s**



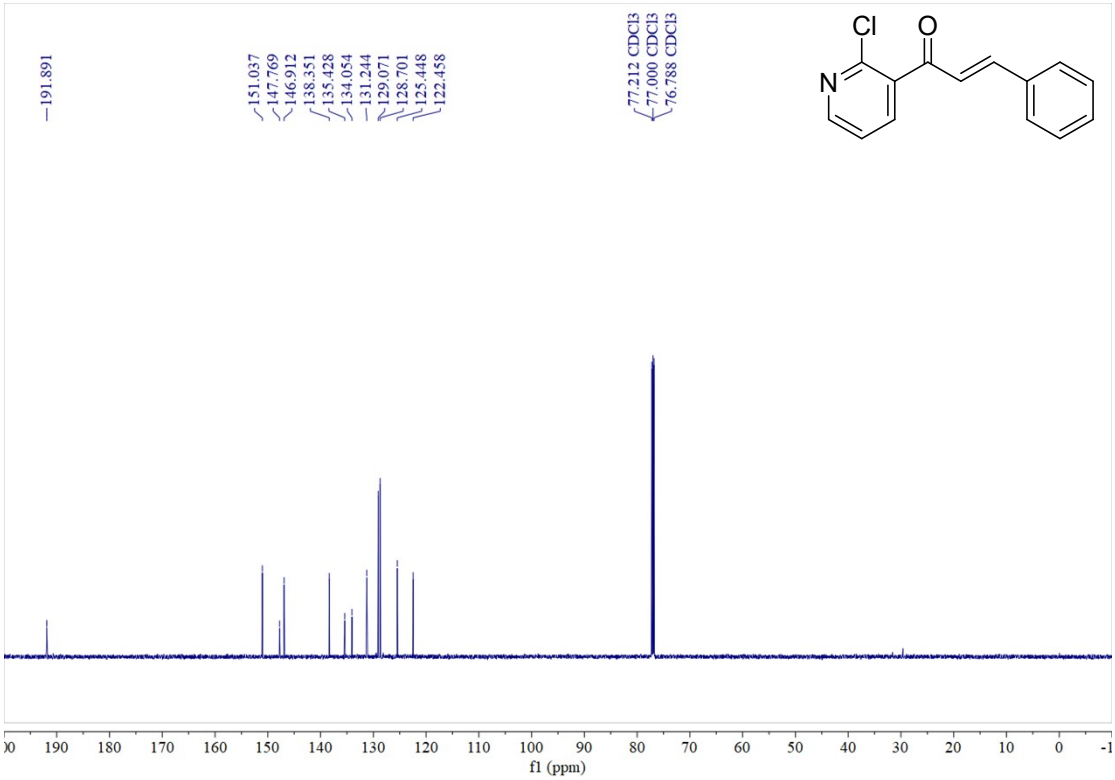
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4s**



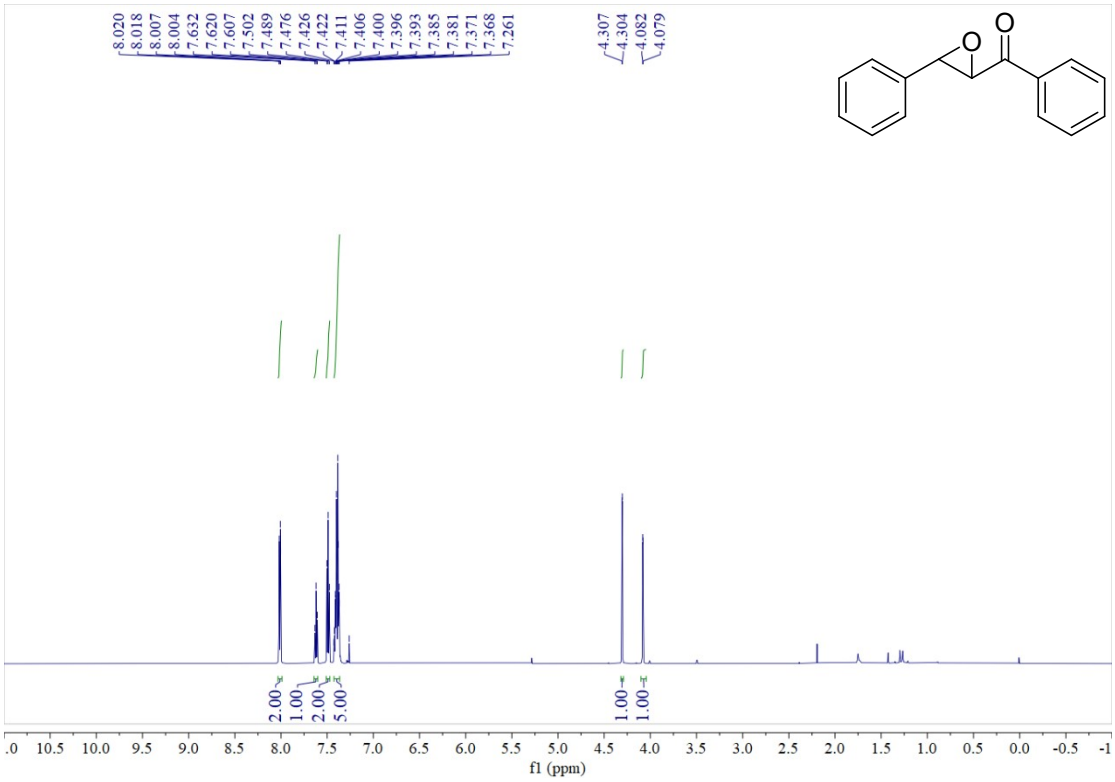
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **4t**



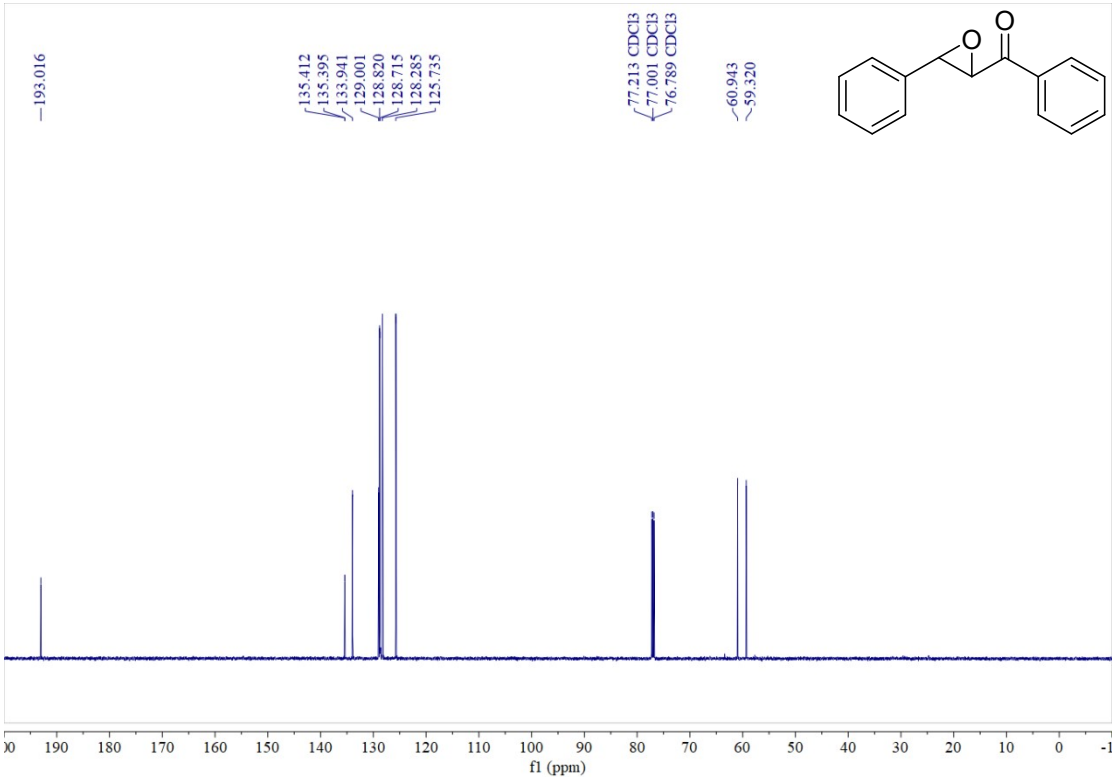
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **4t**



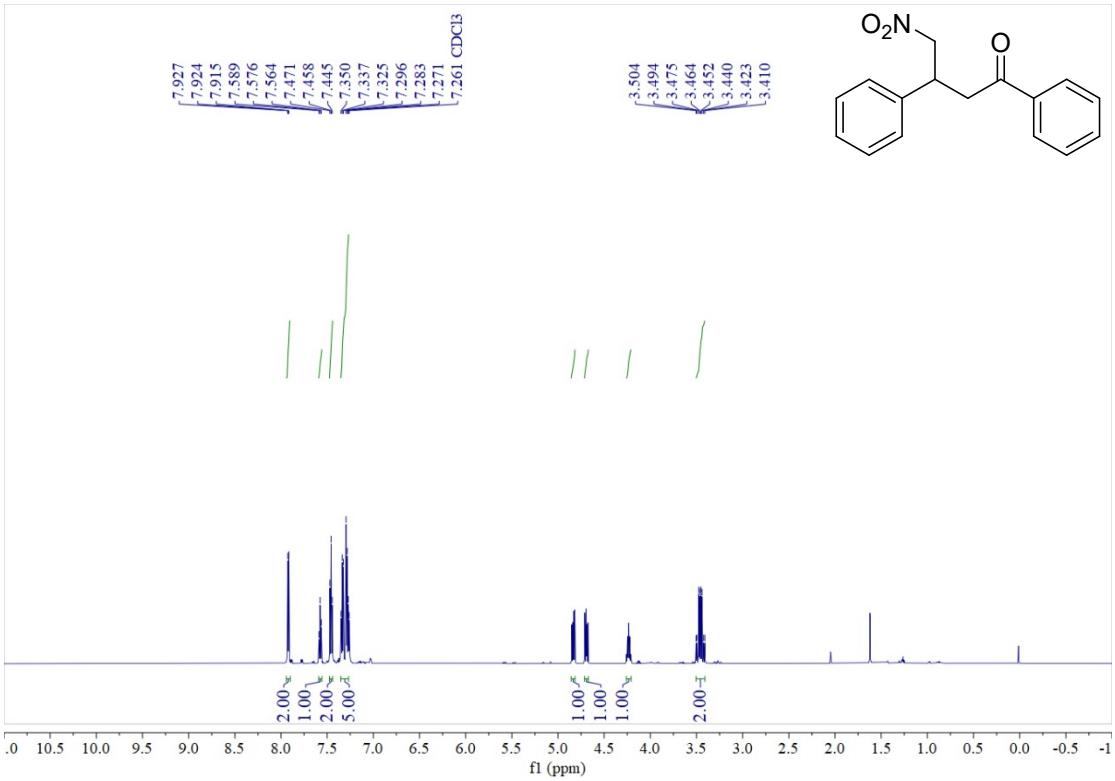
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **5a**



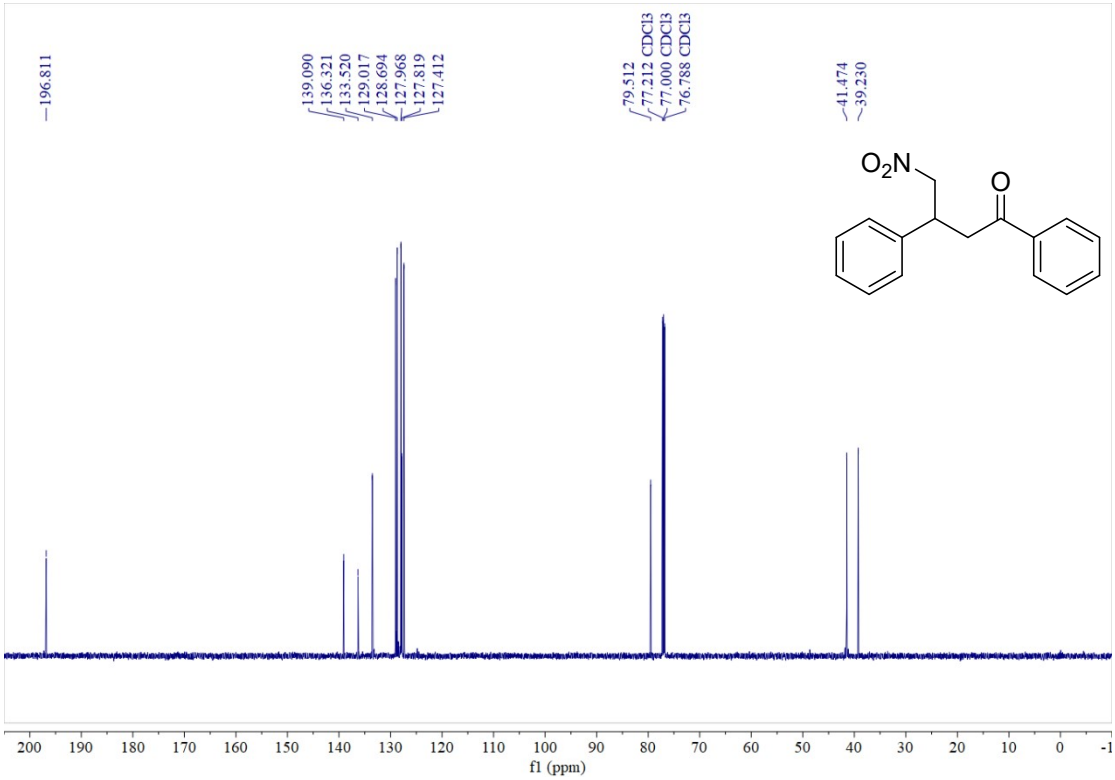
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **5a**



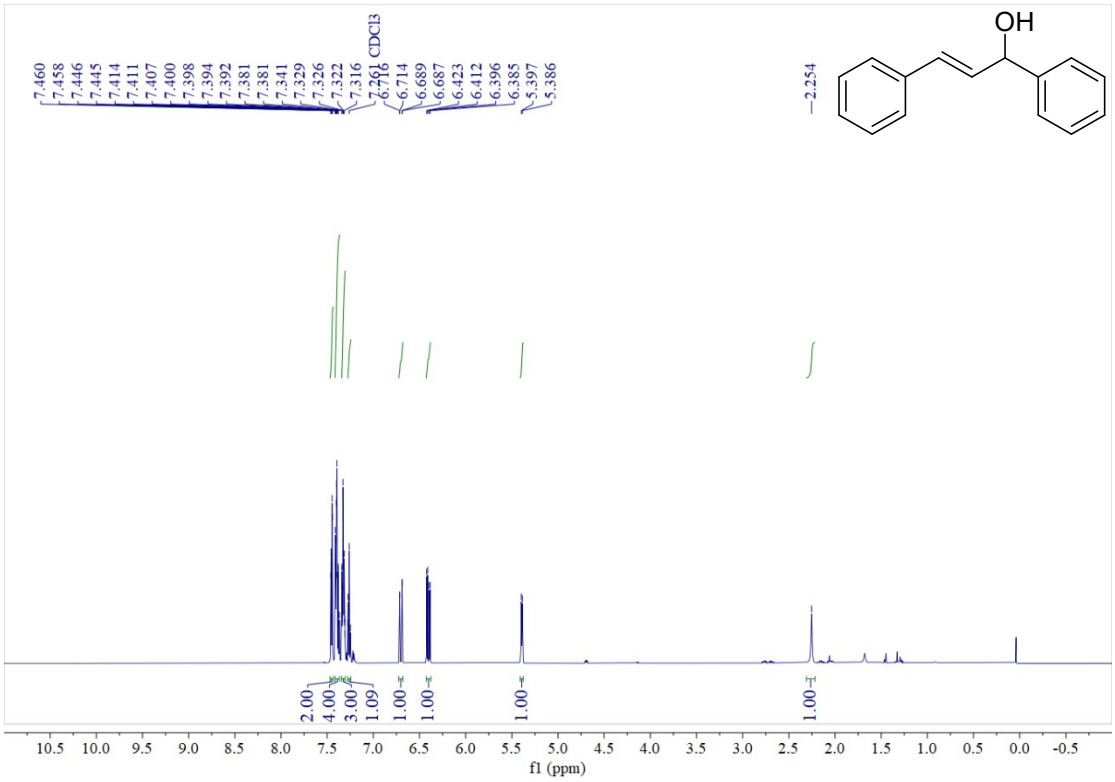
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **5b**



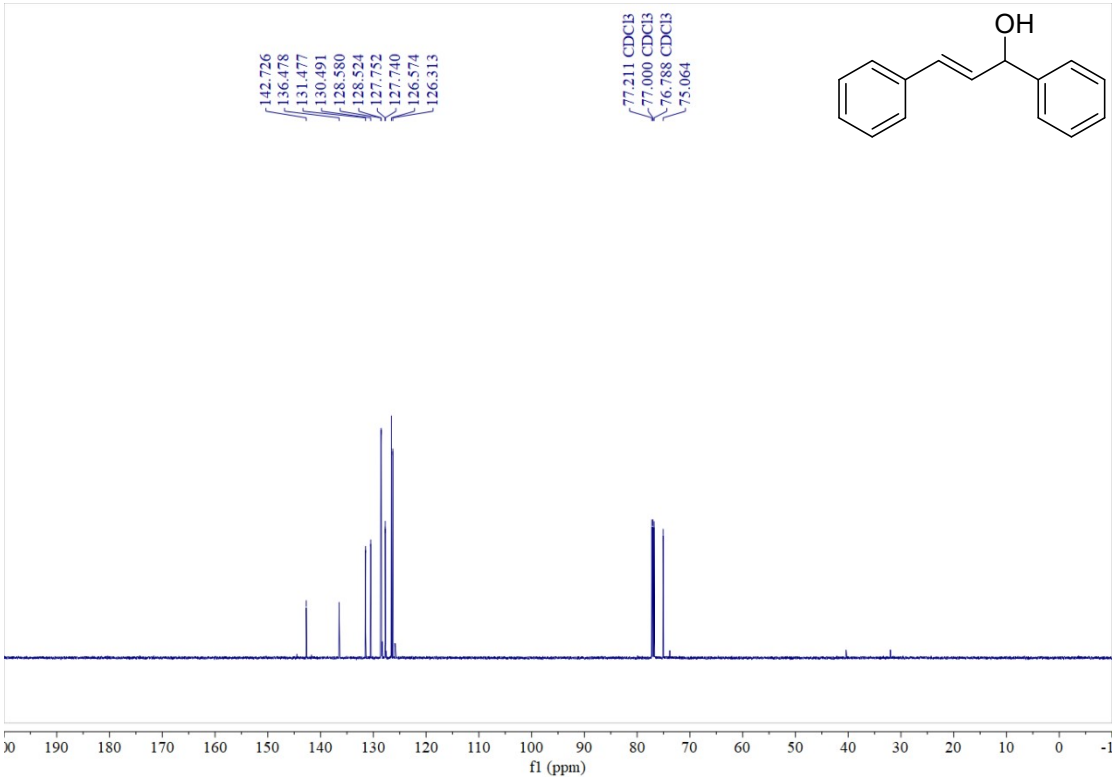
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **5b**



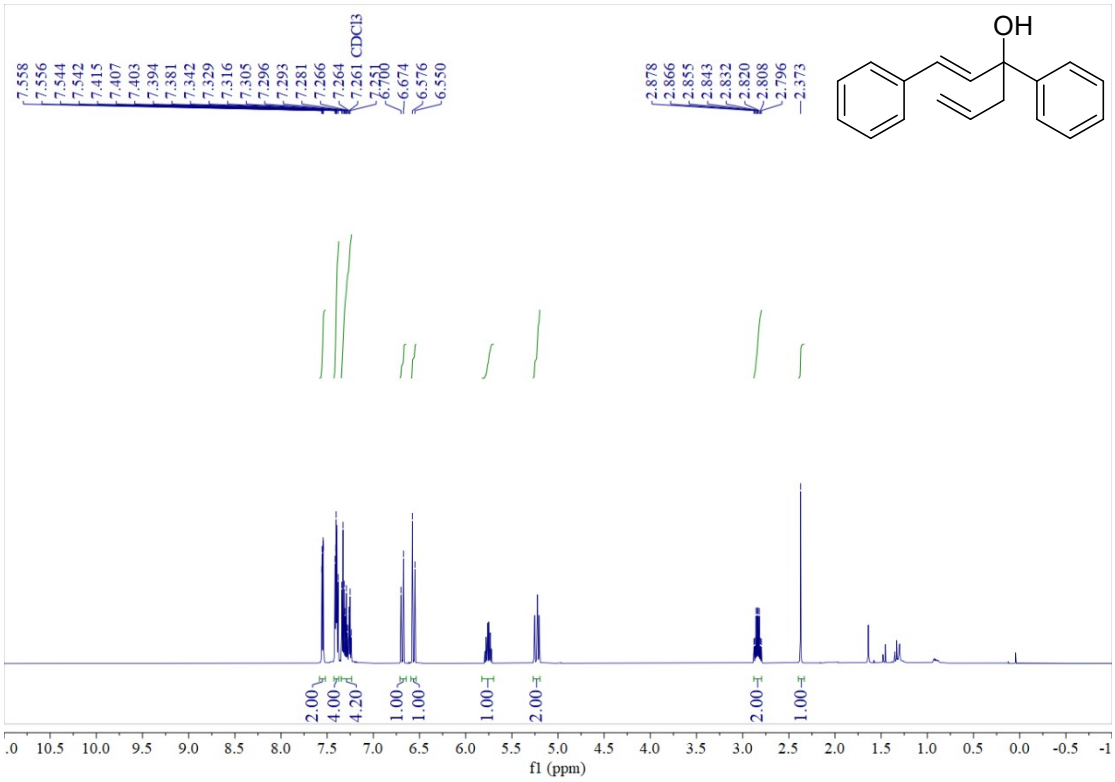
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **5c**



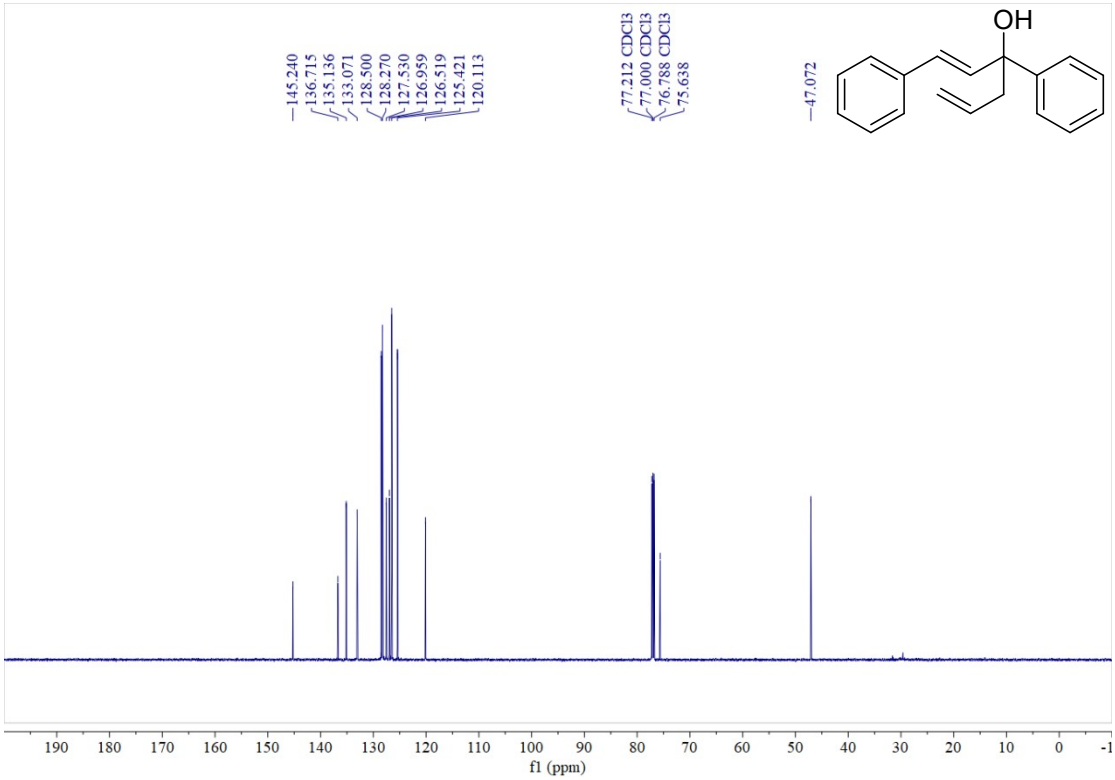
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **5c**



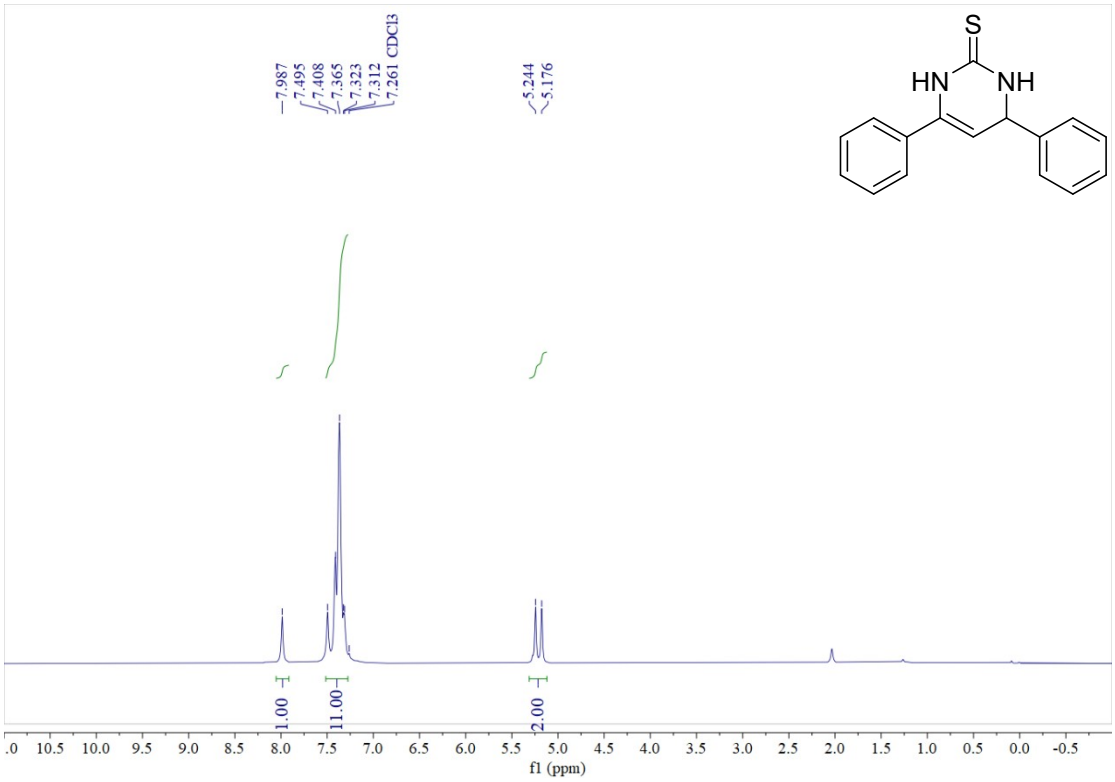
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **5d**



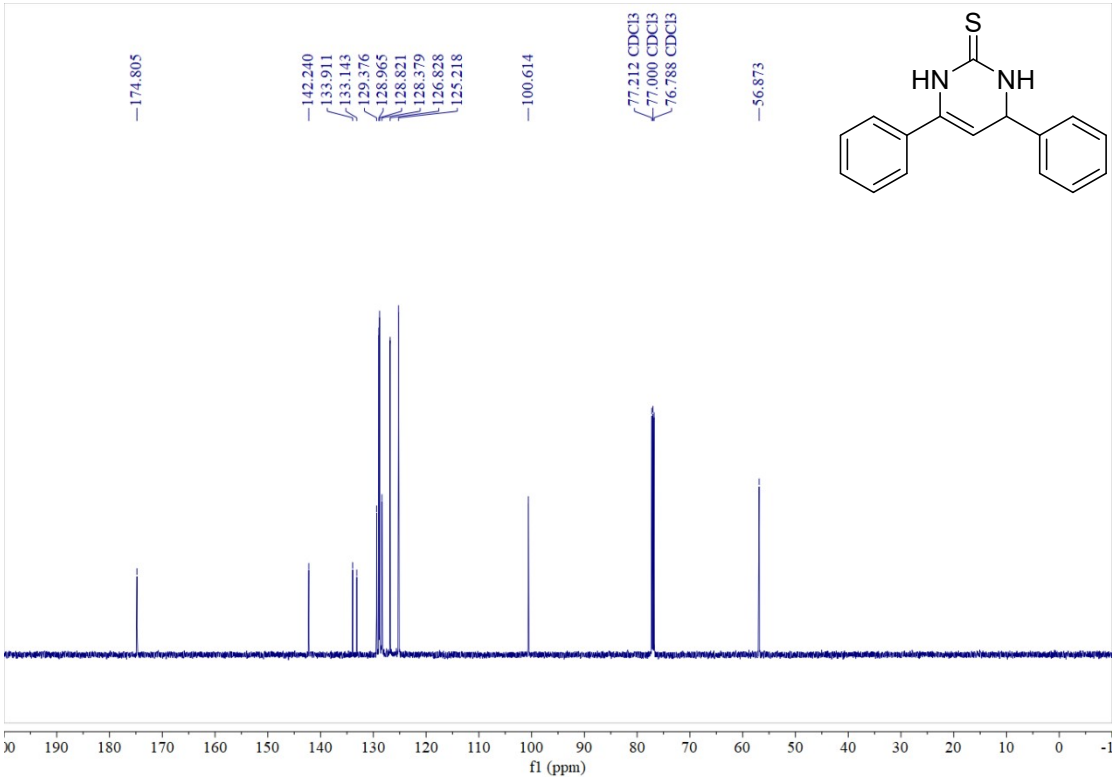
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **5d**



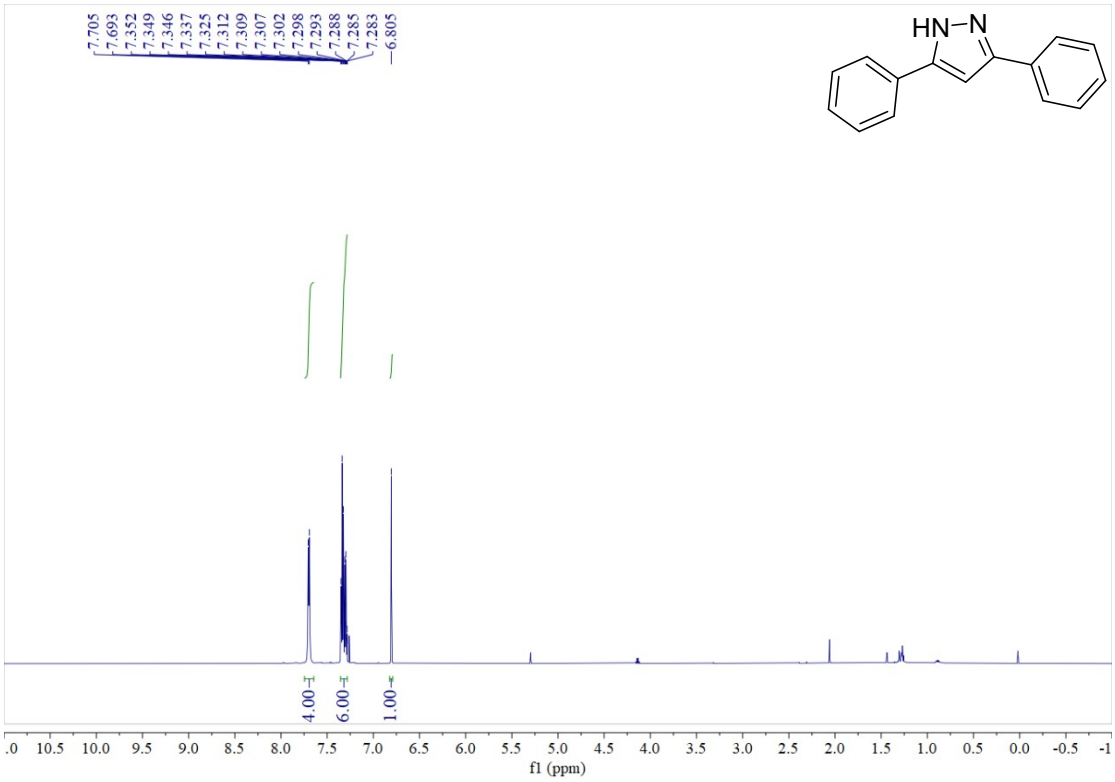
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **5e**



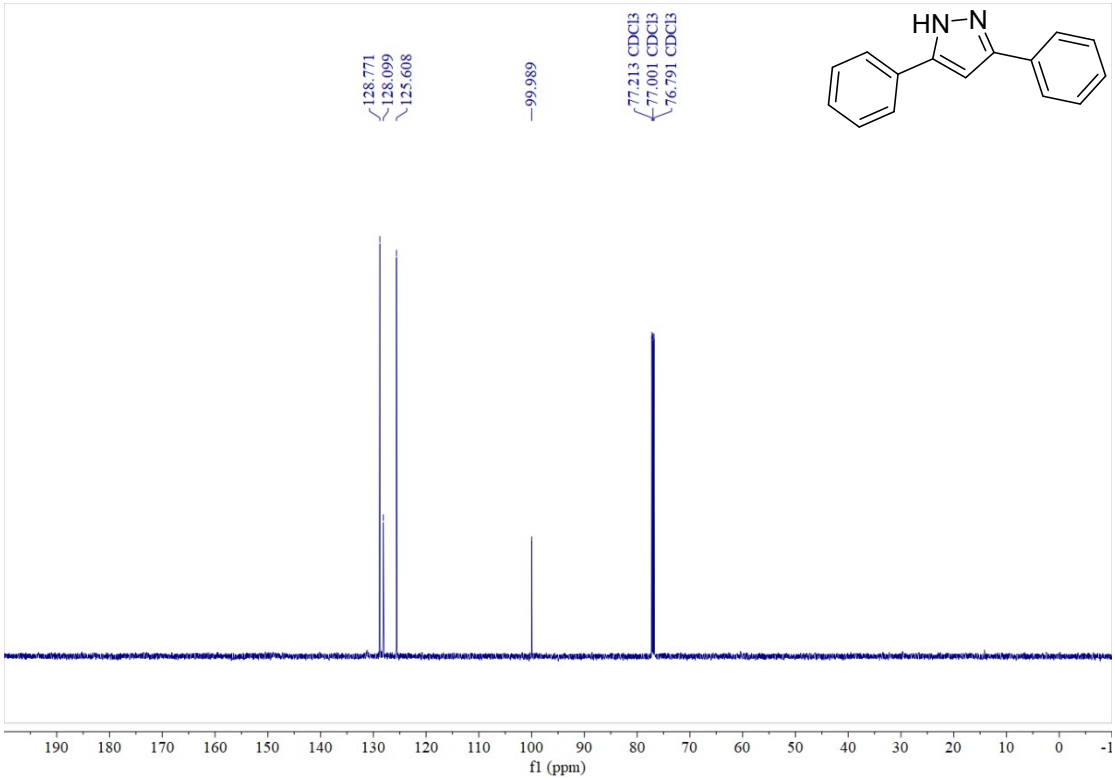
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **5e**



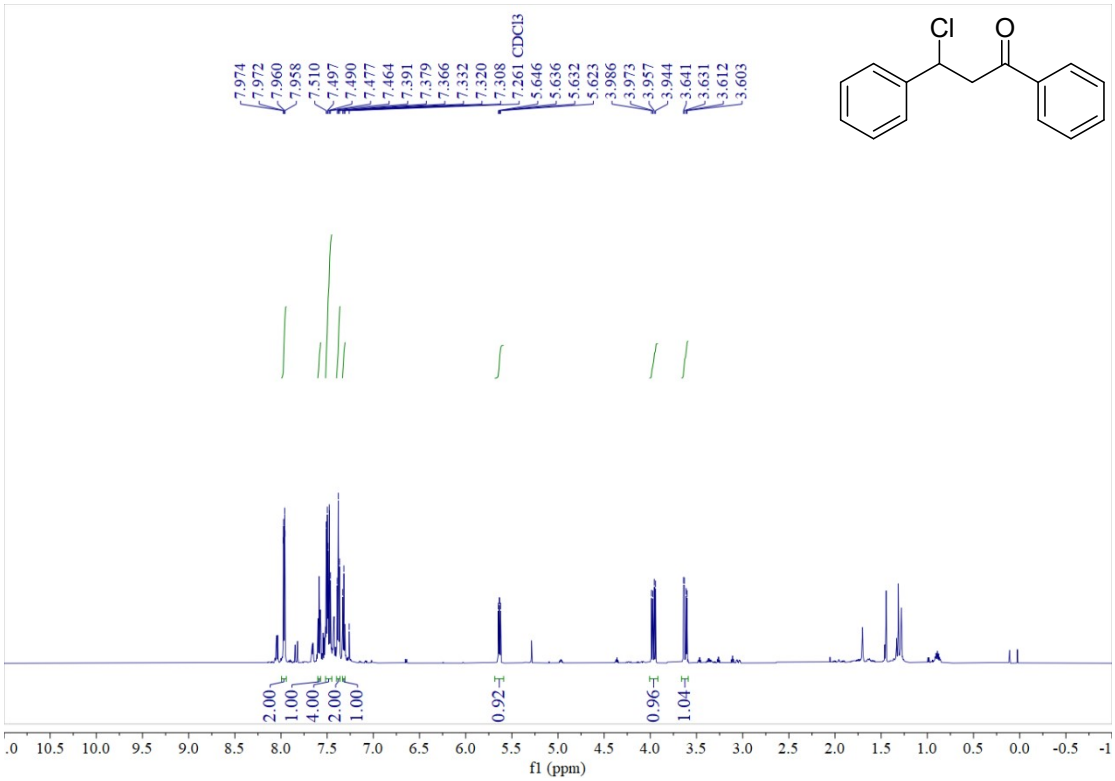
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **5f**



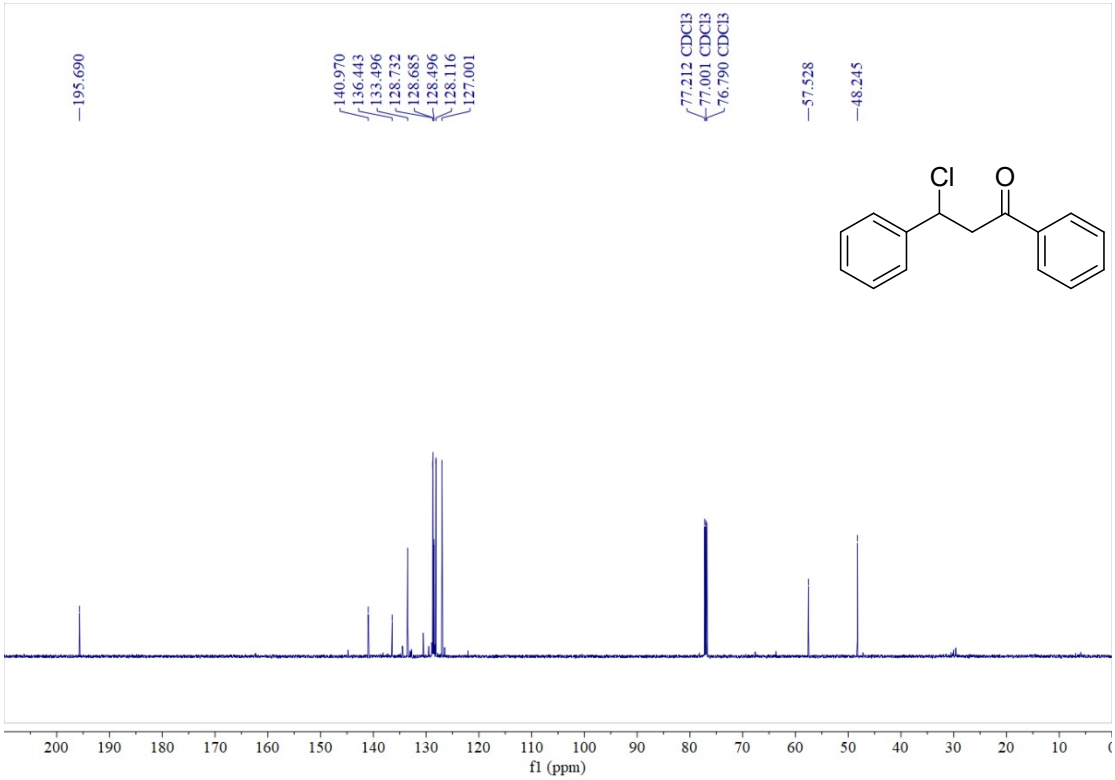
^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **5f**



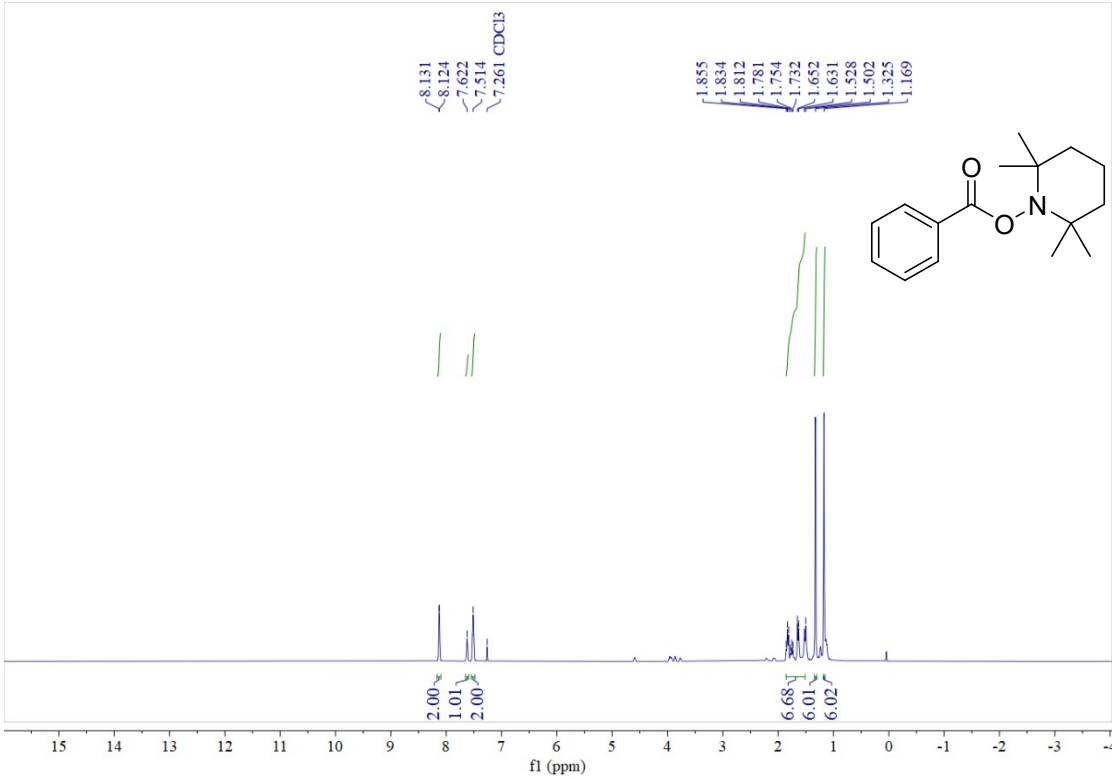
^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **6**



^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **6**



^{1}H NMR (600 MHz, $CDCl_3$) Spectrum of **7**



^{13}C NMR (151 MHz, $CDCl_3$) Spectrum **7**