Supporting Information

Iodine-Catalyzed α,β-Dehydrogenation of Ketones and Aldehydes Generating Conjugated Enones and Enals

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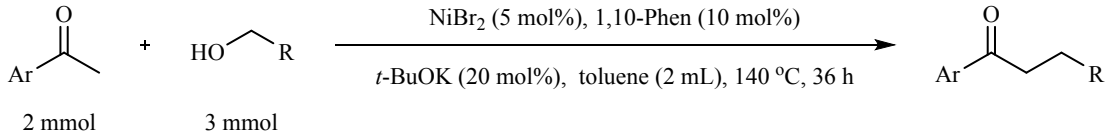
1. General Information

The reactions were carried out in schlenk tubes of 25 mL under N_2 atmosphere. Reagents were used as received unless otherwise noted, and solvents were purified according to standard operation procedure. Column chromatography was performed using Silica Gel 60 (200–300 mesh). The reactions were monitored by GC and GC-MS, GC-MS results were recorded on GCMS-QP2020, and GC analysis was performed on GC-2014 plus. The ^1H, and ^{13}C NMR spectra were recorded on a Brucker ADVANCE III spectrometer at 400 MHz, and 100 MHz respectively, and chemical shifts were reported in parts per million (ppm). All solvents and reagents were purchased from Myror Energy Chemical, Alfa Aesar, CNN, and Aladdin.

2. Experimental Procedure

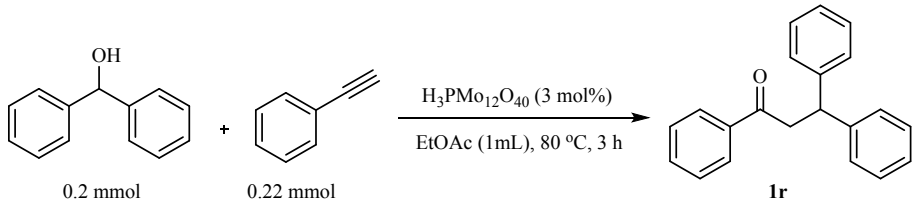
2.1 General Procedure A for the Synthesis of 1a-1q, 1s and 1t.[1]



2 mmol 3 mmol

In an oven-dried 25 mL Schlenk tube under an atmosphere of N_2 was charged with $NiBr_2$ (0.1 mmol), 1,10-Phen (o-Phenanthroline, 0.2 mmol, 10 mol%), *t*-BuOK (0.4 mmol, 20 mol%), alcohols (3mmol), then the acetophenone and toluene (2 mL) were added. The reaction mixture was reacted at 140 °C for 36 h. After then, the reaction mixture was cooled to room temperature, quenched with water, and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4 and concentrated under vacuum. The desired product was isolated by column chromatography over silica gel (200-300 mesh) using petroleum ether/ethyl acetate (50:1) as eluent.

2.2 The Synthesis of 1r from diphenylmethanol and phenylacetylene .[2]

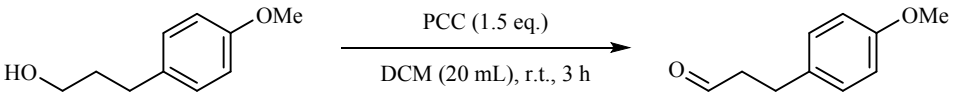


0.2 mmol 0.22 mmol **1r**

In an oven-dried 25 mL Schlenk tube was charged with $H_3PMo_{12}O_{40}$ (0.006 mmol, 11mg), diphenylmethanol

(0.2 mmol), phenylacetylene (0.22 mmol) in EtOAc (1 mL) under N_2. The reaction mixture was reacted at 80 °C for 3 h. After completion of the reaction, the mixture cooled to room temperature, quenched with water and extracted with ethyl ether. The combined organic layers were washed with saturated NaCl solution, dried over anhydrous Na_2SO_4 and concentrated under vacuum. The crude product was further purified by silica on column chromatography (petroleum ether/EtOAc = 50:1) to afford 1,3,3-triphenylpropan-1-one (0.11 mmol, 31.3mg, 55%).

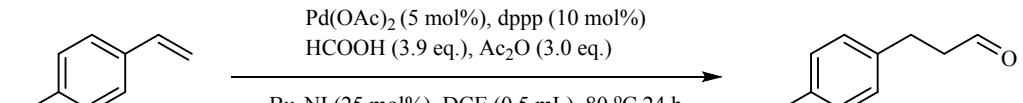
2.3 The Synthesis of 1z from 3-(4-methoxyphenyl)propan-1-ol .[3]



10 mmol **1z**

In an oven-dried 100 mL Schlenk tube was charged with alcohol (10 mmol), pyridinium chlorochromate (15 mmol) in anhydrous DCM (20 mL) under N_2. The reaction mixture was reacted at nitrogen for 3 h. After completion of the reaction, the reaction mixture was filtered and washed with DCM. The liquid was concentrated under vacuum. The crude product was isolated by column chromatography over silica gel (200-300 mesh) using petroleum ether/ethyl acetate (50:1) as eluent to obtain **1z** (pale yellow oil) (7.5 mmol, 1231.5mg, 75%).

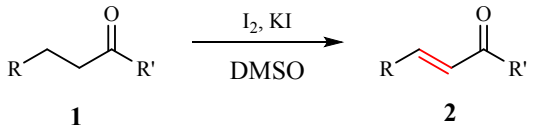
2.4 The Synthesis of 1aa from 1-chloro-4-vinylbenzene .[4]



0.5 mmol **1aa**

In an oven-dried 25 mL Schlenk tube was charged with $Pd(OAc)_2$ (0.025 mmol), dppp (0.050 mmol), *n*-Bu_4NI (0.0125 mmol), and 1,2-dichloroethane (DCE) (0.50 mL). Styrene (0.50 mmol), Ac_2O (1.50 mmol) and HCOOH (1.95 mmol) were slowly added by syringe under N_2. The reaction mixture was stirred at 80 °C for 24 h, After completion of the reaction, the mixture was cooled to room temperature and quenched with water, extracted with EtOAc. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4 and concentrated by vacuum. The crude product was isolated by silica (200-300 mesh) on column chromatography using petroleum ether/ethyl acetate (50:1) as eluent to afford **1aa** (Colorless liquid) (0.4 mmol, 67.4mg, 80%).

2.5 General Procedure B for the Synthesis of α,β-unsaturated ketones.

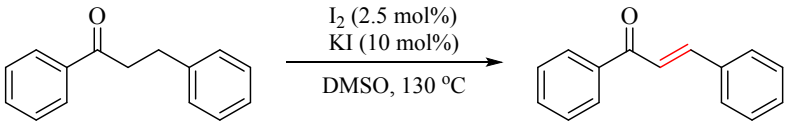


1 **2**

In an oven-dried 25 mL Schlenk tube was charged with ketones **1** (0.2 mmol), KI (0.02 mmol, 10 mol %), I_2 (0.005 mmol, 2.5 mol %) and DMSO (3 mL) in glove boxes. The reaction mixture was reacted at 130 °C for 16 h. After completion of the reaction, the reaction mixture was quenched with water and extracted with ethyl acetate The combined organic layers were washed with brine three times, dried over anhydrous Na_2SO_4 and concentrated under vacuum. The desired product was isolated by column chromatography over silica gel (200-300 mesh) using petroleum ether/ethyl acetate = 50:1 as eluent to obtain the target compounds.

2.6 **Experimental Procedure for the Preparation of chalcone.**

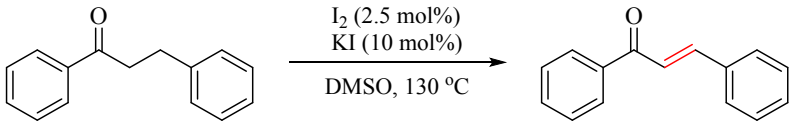
2.6.1 **Experimental Procedure for the Preparation of chalcone for 1 mmol scale.**



1a **2a**

In an oven-dried 25 mL Schlenk tube was charged with 1,3-diphenylpropan-1-one **1a** (1.0 mmol), KI (10 mol %), I_2 (2.5 mol %), and DMSO (10 mL) under N_2. The reaction mixture was reacted at 130 °C for 16 h. After completion of the reaction, the reaction mixture was washed with NaCl saturated solution, dried over anhydrous Na_2SO_4 and concentrated under vacuum. The desired product was isolated by column chromatography over silica gel (200-300 mesh) using petroleum ether/ethyl acetate = 50:1 as eluent to afford a yellow solid **2a** in 73% yield (151.8 mg).

2.6.2 **Experimental Procedure for the Preparation of chalcone for 5 mmol scale.**

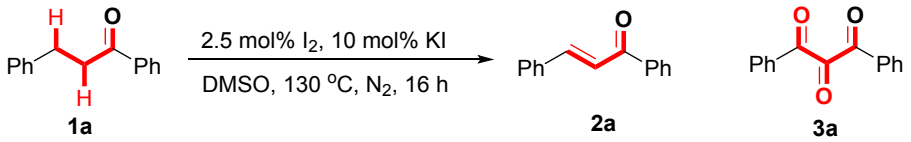


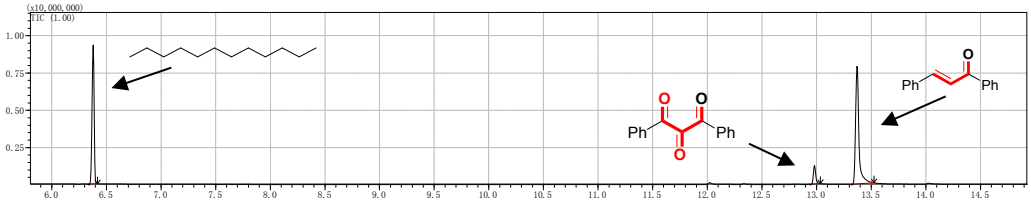
1a **2a**

In an oven dried 100 mL Schlenk tube was charged with 1,3-diphenylpropan-1-one **1a** (5.0 mmol), KI (10

mol %), I_2 (2.5 mol %), and DMSO (50 mL) under N_2. The reaction mixture was reacted at 135 °C for 16 h. After completion of the reaction, the reaction mixture was washed with NaCl saturated solution, dried over anhydrous Na_2SO_4 and concentrated under vacuum. The desired product was isolated by column chromatography over silica gel (200-300 mesh) using petroleum ether/ethyl acetate = 50:1 as eluent to afford a yellow solid **2a** in 75% yield (780 mg).

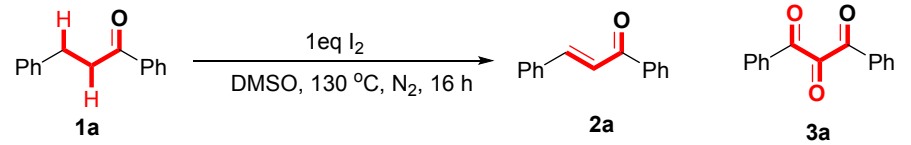
3. GC-MS data for the oxidative of ketone to three carbonyl group product

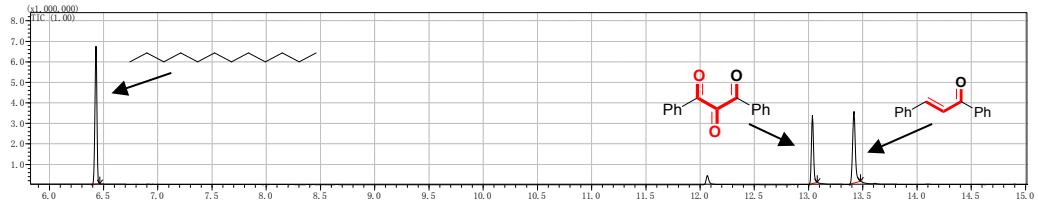




Peak Report

Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Name
1	6.380	6.340	6.420	12479626	39.89	9315320	dodecane
2	12.980	12.945	13.035	1887897	6.04	1246110	1,3-diphenylpropane-1,2,3-trione
3	13.372	13.325	13.525	16914549	54.07	7895526	chalcone





Peak Report

Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Name
1	6.430	6.395	6.465	8513307	44.25	6713272	dodecane
2	13.035	13.005	13.080	4546501	23.63	3317185	1,3-diphenylpropane-1,2,3-trione
3	13.419	13.380	13.480	6178513	32.12	3490935	chalcone

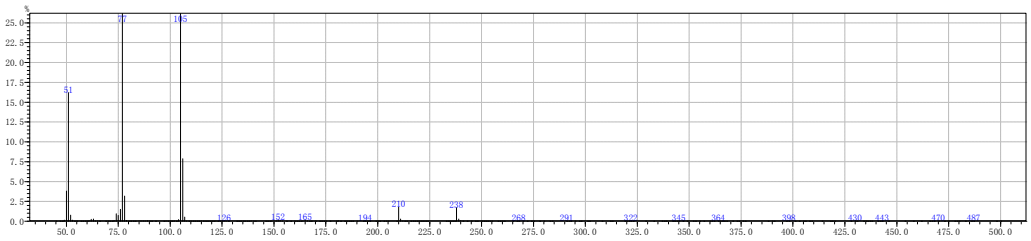
Spectrum

Line#:1 R.Time:13.035(Scan#:2008)

MassPeaks:270

RawMode:Averaged 12.975-13.215(1996-2044) BasePeak:105.05(10000)

BG Mode:Calc. from Peak Group 1 – Event 1

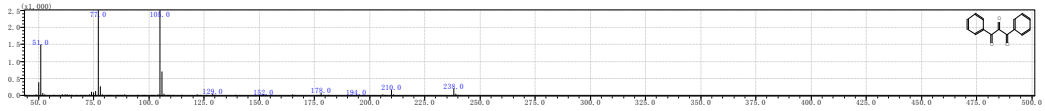


Library

Hit#:1 Entry:27332 Library:NIST17s.LIB

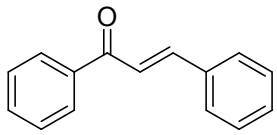
SI:97 Formula:$C_{15}H_{10}O_3$ CAS:643-75-4 Molweight:238 RetIndex:2073

CopeName:Propanetritrione, diphenyl- $$ 1,3-Diphenyl-1,2,3-propanetrione # $$



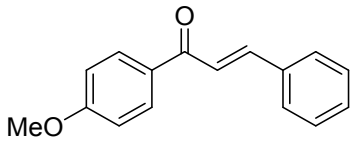
4. Characterization Data for the Products

chalcone (2a)



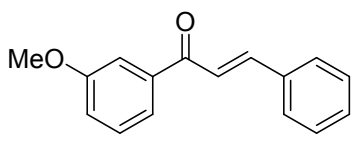
Compound **2a** was prepared from **1a** (0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a pale yellow solid in 73% yield (30.4 mg), mp 52-53 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04-8.02 (m, 2H), 7.82 (d, *J* = 15.6 Hz, 1H), 7.67-7.64 (m, 2H), 7.61-7.56 (m, 4H), 7.43-7.41 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.52, 144.81, 138.15, 134.83, 132.75, 130.51, 128.92, 128.59, 128.46, 128.41, 122.03. This compound is known.[5]

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



Compound **2b** was prepared from 1-(4-methoxyphenyl)-3-phenylpropan-1-one (**1b**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a white solid in 66% yield (31.5 mg), mp 106.6-107 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05 (d, *J* = 8.8 Hz, 2H), 7.80 (d, *J* = 15.6 Hz, 1H), 7.66-7.64 (m, 2H), 7.55 (d, *J* = 15.6 Hz, 1H), 7.43-7.41 (m, 3H), 6.99 (d, *J* = 8.8 Hz, 2H), 3.90 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.73, 163.41, 143.96, 135.06, 131.08, 130.81, 130.31, 128.91, 128.34, 121.86, 113.83, 55.49. This compound is known.[6]

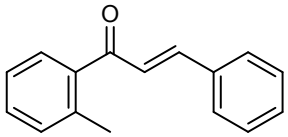
(*E*)-1-(3-methoxyphenyl)-3-phenylprop-2-en-1-one (2c)



Compound **2c** was prepared from 1-(3-methoxyphenyl)-3-phenylpropan-1-one (**1c**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a yellow oil in 75% yield (35.7 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, *J* = 15.6 Hz 1H), 7.66-7.63 (m, 2H), 7.61 (d, *J* = 8.0 Hz, 1H), 7.56-7.55 (m, 1H), 7.52 (d, *J* = 15.6 Hz, 1H), 7.43-7.39 (m, 4H), 7.15-7.12 (m, 1H), 3.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.18, 159.84, 144.79, 139.52, 134.80, 130.50,

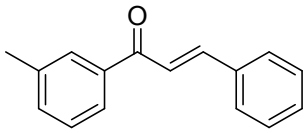
129.52, 128.90, 128.40, 122.03, 120.99, 119.24, 112.80, 55.41. This compound is known. [7]

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



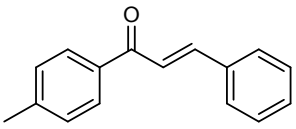
Compound **2d** was prepared from 3-phenyl-1-(o-tolyl)propan-1-one (**1d**, 0.2 mmol) according to the general procedure B and purified by silica on column chromatography using petroleum ether/ethyl acetate (50:1) as elution to afford a yellow oil in 73% yield (32.5 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70-7.67 (m, 2H), 7.63-7.57 (m, 2H), 7.53-7.49 (m, 4H), 7.42-7.38 (m, 2H), 7.26 (d, *J* = 16.4 Hz, 1H), 2.57 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.54, 145.88, 139.01, 136.89, 134.56, 131.27, 130.61, 130.42, 128.92, 128.37, 128.03, 126.69, 125.43, 20.17. This compound is known.[7]

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



Compound **2e** was prepared from 3-phenyl-1-(m-tolyl)propan-1-one (**1e**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a yellow solid in 71% yield (31.6 mg) , mp 53.6-54.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84-7.79 (m, 3H), 7.67-7.64 (m, 2H), 7.53 (d, *J* = 15.6 Hz, 1H), 7.44-7.39 (m, 5H), 2.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$):δ 190.66, 144.59, 138.41, 138.20, 134.89, 133.53, 130.44, 128.99, 128.90, 128.43, 128.39, 125.66, 122.21, 21.36. This compound is known.[7]

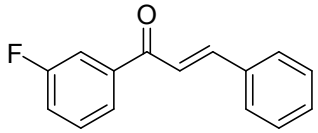
(*E*)-3-phenyl-1-(p-tolyl)prop-2-en-1-one (**2f**)



Compound **2f** was prepared from 3-phenyl-1-(p-tolyl)propan-1-one (**1f**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a pale yellow solid in 65% yield (28.9 mg), mp 53.8-55.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95

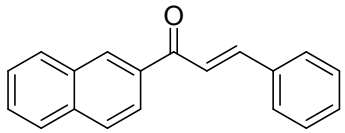
(d, *J* = 8.0 Hz, 2H), 7.82 (d, *J* = 15.6 Hz, 1H), 7.66-7.63 (m, 2H), 7.55 (d, *J* = 15.6Hz 1H), 7.42-7.41 (m, 3H), 7.30 (d, *J* = 8.0 Hz, 2H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.86, 144.27, 143.55, 135.52, 134.89, 130.33, 129.24, 128.84, 128.56, 128.31, 121.96, 21.58. This compound is known.[5]

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



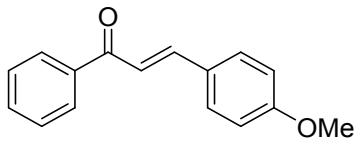
Compound **2g** was prepared from 1-(3-fluorophenyl)-3-phenylpropan-1-one (**1g**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a pale yellow solid in 55% yield (24.9 mg), mp 61.7-62.3 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70-7.64 (m, 2H), 7.57-7.48 (m, 3H), 7.36-7.27 (m, 5H), 7.16-7.11 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$):δ 189.10, 162.84 (*J* = 246.4 Hz), 145.54, 140.28 (*J* = 6.2 Hz), 134.60, 130.77, 130.25 (*J* = 7.6 Hz), 128.98, 128.51, 124.13 (*J* = 2.8 Hz), 121.46, 119.74 (*J* = 21.4 Hz), 115.26 (*J* = 22.2 Hz). This compound is known.[8]

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



Compound **2h** was prepared from 1-(naphthalen-2-yl)-3-phenylpropan-1-one (**1h**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a yellow solid in 80% yield (41.3 mg) , mp 105.1-105.4 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.54 (s, 1H), 8.13-8.11 (m, 1H), 7.99 (d, *J* = 8.0 Hz, 1H), 7.94-7.88 (m, 3H), 7.72-7.68 (m, 3H), 7.63-7.54 (m, 2H), 7.46-7.43 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.12, 144.64, 135.40, 135.37, 134.85, 132.45, 130.46, 129.86, 129.44, 128.88, 128.48, 128.41, 128.30, 127.73, 126.70, 124.39, 121.94. This compound is known.[9]

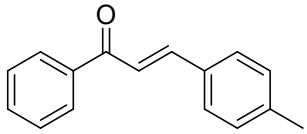
(*E*)-3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one (**2i**)



Compound **2i** was prepared from 3-(4-methoxyphenyl)-1-phenylpropan-1-one (**1i**, 0.2 mmol) according to the

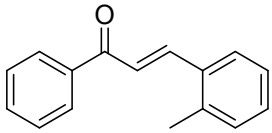
general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a pale yellow solid in 70% yield (33.4 mg), mp 72-73 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02-8.00 (m, 2H), 7.79 (d, *J* =15.6 Hz, 1H), 7.60-7.55 (m, 3H), 7.51-7.39 (m, 3H), 6.93 (d, *J* =8.8 Hz, 2H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.45, 161.59, 144.60, 138.40, 132.48, 130.15, 128.47, 128.32, 127.50, 119.65, 114.33, 55.31. This compound is known.[5]

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



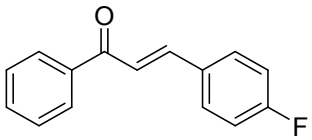
Compound **2j** was prepared from 1-phenyl-3-(p-tolyl)propan-1-one (**1j**, 0.2 mmol) in the reaction of I_2 (0.005 mmol, 2.5 mol %), DMSO (2 mL) in glove boxes. The reaction mixture was reacted at 130 °C for 16 h and purified by column chromatography on silica gel and eluant with petroleum ether/ethyl acetate (50:1) to afford a pale yellow solid in 66% yield (29.3 mg), mp 88.6-89.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03-8.01 (m, 2H), 7.80 (d, *J* =16.0 Hz,.1H), 7.60-7.48 (m, 6H), 7.23 (d, *J* =8.0 Hz, 2H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.65, 144.94, 141.08, 138.33, 132.65, 132.12, 129.68, 128.57, 128.46, 128.45, 121.06, 21.52. This compound is known.[5]

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



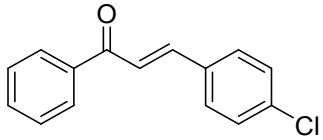
Compound **2k** was prepared from 1-phenyl-3-(o-tolyl)propan-1-one (**1k**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a yellow oil in 66% yield (29.3 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.20 (d, *J* = 15.6 Hz, 1 H), 8.12-8.10 (m, 2H), 7.76 (d, *J* = 7.2 Hz, 1H), 7.62 (dd, $J_1 = J_2$ = 7.2 Hz, 1H), 7.57-7.51 (m, 3H), 7.37-7.26 (m, 3H), 2.51 (s, 3H); ^{13}C NMR (100 MHz, CDCl3): δ 189.98, 142.05, 138.07, 137.94, 133.59, 132.54, 130.67, 130.04, 128.37, 128.26, 126.17, 126.11, 122.71, 19.58. This compound is known.[10]

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



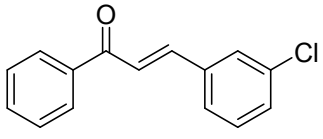
Compound **2l** was prepared from 3-(4-fluorophenyl)-1-phenylpropan-1-one (**1l**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a yellow solid in 60% yield (27.2 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02-8.00 (m, 2H), 7.77 (d, *J* = 15.6 Hz, 1H), 7.65-7.56 (m, 3H), 7.52-7.44 (m, 3H), 7.13-7.08 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.23, 163.99 (*J* = 250.2 Hz), 143.43, 138.05, 132.79, 131.07 (*J* = 3.2 Hz), 130.29 (*J* = 8.5 Hz), 128.59, 128.41, 121.69 (*J* = 2.2 Hz,), 116.06 (*J* = 21.8 Hz,). This compound is known.[9]

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



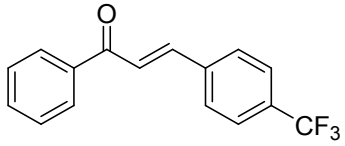
Compound **2m** was prepared from 3-(4-chlorophenyl)-1-phenylpropan-1-one (**1m**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a pale yellow solid in 54% yield (26.2 mg), mp 112.9-113.9 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02-8.00 (m, 2H), 7.75 (d, *J* = 15.6 Hz, 1H), 7.61-7.55 (m, 3H), 7.52-7.48 (m, 3H), 7.38 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.15, 143.24, 137.95, 136.36, 133.31, 132.88, 129.54, 129.18, 128.62, 128.44, 122.38. This compound is known.[5]

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



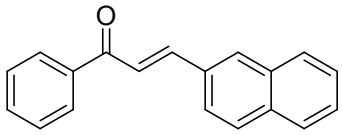
Compound **2n** was prepared from 3-(3-chlorophenyl)-1-phenylpropan-1-one (**1n**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a yellow solid in 57% yield (27.7 mg), mp 74.0-74.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03-8.01 (m, 2H), 7.72 (d, *J* = 16.0 Hz, 1H), 7.62-7.57 (m, 2H), 7.54-7.48 (m, 4H), 7.38-7.32 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.96, 142.95, 137.80, 136.64, 134.88, 132.96, 130.26, 130.13, 128.63, 128.46, 127.83, 126.73, 123.11. This compound is known.[7]

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



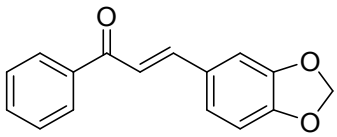
Compound **2o** was prepared from 1-phenyl-3-(4-(trifluoromethyl)phenyl)propan-1-one (**1o**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a yellow solid in 63% yield (34.8 mg), mp 122.1-123.9 °C ;^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04-8.02 (m, 2H), 7.81 (d, *J* = 15.6 Hz, 1H), 7.74 (d, *J* = 8.4 Hz, 2H), 7.67 (d, *J* = 8.4 Hz, 2H), 7.63-7.58 (m, 2H), 7.54-7.50 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.99, 142.68, 138.23, 137.74, 133.11, 131.84 (q, *J* = 32.4 Hz), 128.71, 128.53, 128.47, 125.87 (q, *J* = 3.6 Hz), 124.19, 123.80 (q, *J* = 270.5 Hz). This compound is known.[11]

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



Compound **2p** was prepared from 3-(naphthalen-2-yl)-1-phenylpropan-1-one (**1p**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a yellow solid in 72% yield (37.2 mg), mp 158.1-158.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.08-8.04 (m, 3H), 7.99 (d, *J* = 15.6 Hz, 1H), 7.90-7.79 (m, 4H), 7.67-7.59 (m, 2H), 7.55-7.51 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.53, 144.93, 138.27, 134.37, 133.34, 132.78, 132.36, 130.66, 128.72, 128.63, 128.51, 127.79, 127.37, 126.76, 123.64, 122.18. This compound is known.[12]

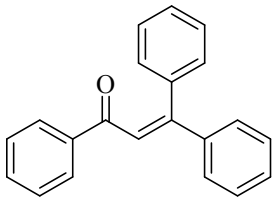
(*E*)-3-(benzo[d][1,3]dioxol-5-yl)-1-phenylprop-2-en-1-one (2q)



Compound **2q** was prepared from 3-(benzo[d][1,3]dioxol-5-yl)-1-phenylpropan-1-one (**1q**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether /ethyl acetate (50:1) to afford a pale yellow solid in 73% yield (36.8 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01-7.99 (m, 2H), 7.74 (d, *J* = 15.6 Hz, 1H), 7.59-7.48 (m, 3H), 7.37 (d, *J* = 15.6 Hz, 1H), 7.17-7.11 (m, 2H), 6.84 (d, *J* =

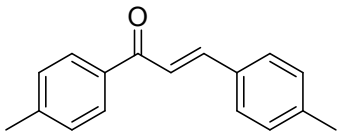
8.0 Hz, 1H), 6.02 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.33, 149.86, 148.35, 144.62, 138.31, 132.58, 129.27, 128.52, 128.35, 125.19, 120.00, 108.60, 106.58, 101.58. This compound is known.[13]

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



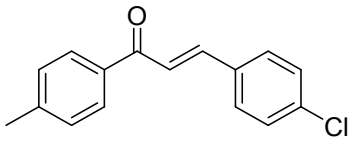
Compound **2r** was prepared from 1,3,3-triphenylpropan-1-one (**1r**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a yellow oil in 88% yield (50 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06-8.04 (m, 2H), 7.63-7.60 (m, 1H), 7.54-7.50 (m, 7H), 7.42-7.40 (m, 4H), 7.34-7.31 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 192.70, 154.70, 141.34, 138.99, 138.21, 132.64, 129.73, 129.32, 128.72, 128.57, 128.42, 128.33, 128.02, 124.01. This compound is known.[14]

(*E*)-1,3-di-p-tolylprop-2-en-1-one (2s)



Compound **2s** was prepared from 1,3-di-p-tolylpropan-1-one (**1s**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a white solid in 65% yield (30.7 mg), mp 130.9-131.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, J = 8.4 Hz, 2H), 7.80 (d, J = 16.0 Hz, 1H), 7.55-7.48 (m, 3H) 7.29 (d, J = 8.0 Hz, 2H), 7.22 (d, J = 8.0 Hz, 2H), 2.42 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.85, 144.29, 143.33, 140.76, 135.60, 132.09, 129.53, 129.14, 128.46, 128.29, 120.87, 21.50, 21.36. This compound is known.[5]

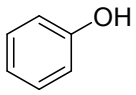
(*E*)-3-(4-chlorophenyl)-1-(p-tolyl)prop-2-en-1-one (2t)



Compound **2t** was prepared from 3-(4-chlorophenyl)-1-(p-tolyl)propan-1-one (**1t**, 0.2 mmol) according to the

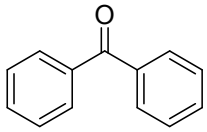
general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a white solid in 63% yield (32.3 mg), mp 150.3-150.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, *J* = 8.4 Hz, 2H), 7.75 (d, *J* = 15.6 Hz, 1H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.50 (d, *J* = 15.6 Hz, 1H), 7.39 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 2.44(s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.69, 143.80, 142.84, 136.28, 135.47, 133.51, 129.52, 129.36, 129.20, 128.64, 122.53, 21.67. This compound is known.[5]

phenol (**2u**)



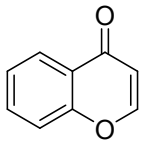
Compound **2u** was prepared from cyclohex-2-en-1-one (**1u**, 0.2 mmol) according to the general procedure B at 120 °C and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a white oil in 60% yield (11.3 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39-7.35 (m, 2H), 7.11-7.00 (m, 3H), 6.64 (b, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.16, 129.64, 120.78, 115.34. This compound is known.[15]

benzophenone (**2v**)



Compound **2v** was prepared from cyclohexyl(phenyl)methanone (**1v**, 0.2 mmol) according to the general procedure B at 150 °C for 16 h. The crude product was purified by silica gel on column chromatography and eluted with petroleum ether/ethyl acetate (50:1) to afford a white solid in 83% yield (30.2 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79-7.77 (m, 4H), 7.56-7.52 (m, 2H), 7.46-7.42 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.33, 137.30, 132.16, 129.76, 128.02. This compound is known.[16]

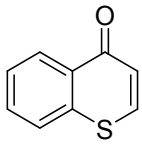
4H-chromen-4-one (**2w**)



Compound **2w** was prepared from chroman-4-one (**1w**, 0.2 mmol) in the reaction of I_2 (0.005 mmol, 2.5 mol %), KI (0.02 mmol, 10mol%), DMSO (1.5 mL) in glove boxes. The reaction mixture was reacted at 130 °C for 16 h

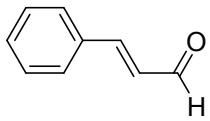
and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (3:1) to afford a pale yellow solid in 65% yield (19.0 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ8.18-8.16 (m, 1H), 7.83 (d, *J* = 6 Hz, 1H), 7.66-7.61 (m, 1H), 7.43-7.35 (m, 2H), 6.31 (d, *J* = 6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 177.54, 156.40, 155.26, 133.68, 125.67, 125.15, 124.76, 118.09, 112.87. This compound is known.[17]

4H-thiochromen-4-one (**2x**)



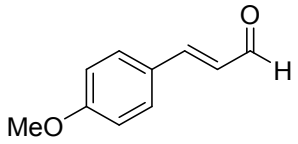
Compound **2x** was prepared from thiochroman-4-one (**1x**, 0.2 mmol) in the reaction of I_2 (0.005 mmol, 2.5 mol %), KI (0.02 mmol, 10mol%), DMSO (1.5 mL) in glove boxes. The reaction mixture was reacted at 130 °C for 16 h and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (3:1) to afford green solid in 89% yield (28.9 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ8.50 (d, *J* = 8 Hz 1H), 7.80 (d, *J* = 10.4 Hz, 1H), 7.57-7.48 (m, 3H), 6.98 (d, *J* = 10.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.59, 137.81, 137.43, 132.18, 131.32, 128.53, 127.73, 126.58, 125.74. This compound is known.[18]

cinnamaldehyde (**2y**)



Compound **2y** was prepared from 3-phenylpropanal (**1y**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10:1) to afford a yellow oil in 76% yield (20.1 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.66-9.63 (m, 1H), 7.49-7.37 (m, 6H), 6.71-6.65 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.22, 152.25, 133.36, 130.65, 128.46, 127.95, 127.74. This compound is known.[13]

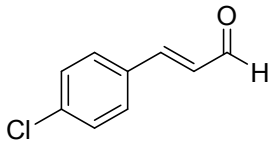
(*E*)-3-(4-methoxyphenyl)acrylaldehyde (**2z**)



Compound **2z** was prepared from 3-(4-methoxyphenyl)propanal (**1z**, 0.2 mmol) according to the general procedure

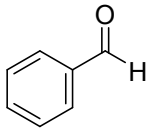
B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10:1) to afford a yellow solid in 70% yield (22.7mg), mp 52.6-53.4 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.64 (d, J = 8.0 Hz, 1H), 7.51 (d, J = 8.8 Hz, 2H), 7.41 (d, J = 16.0 Hz, 1H), 6.94 (d, J = 8.8 Hz, 2H), 6.60 (dd, J_1 = 8.0 Hz, J_2 = 16.0 Hz, 1H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.71, 162.15, 152.74, 130.31, 126.71, 126.43, 114.51, 55.39. This compound is known.[13]

(*E*)-3-(4-chlorophenyl)acrylaldehyde (2aa)



Compound **2aa** was prepared from 3-(4-chlorophenyl)propanal (**1aa**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (10:1) to afford a yellow solid in 66% yield (21.9 mg), mp 57-58.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.68 (d, J = 7.6 Hz, 1H), 7.48 (d, J = 8.4 Hz, 2H), 7.43-7.37 (m, 3H), 6.66 (dd, J_1 = 16.0 Hz, J_2 = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.26, 152.25, 133.36, 130.65, 128.46, 127.85, 127.75. This compound is known.[19]

benzaldehyde (2ab)



1) Compound **2ab** was prepared from cyclohex-3-ene-1-carbaldehyde (**1ab1**, 0.2 mmol) according to the general procedure B and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a white oil in 68% yield (14.4mg).

2) Compound **2ab** was also prepared from cyclohexanecarbaldehyde (**1ab2**, 0.2 mmol) in the reaction of I_2 (0.01 mmol, 10 mol %), DMSO (3 mL) in glove boxes. The reaction mixture was reacted at 150 °C for 16 h and purified by column chromatography on silica gel and eluted with petroleum ether/ethyl acetate (50:1) to afford a white oil in 55% yield (11.7 mg).

^{1}H NMR (400 MHz, $CDCl_3$): δ 9.83 (s, 1H), 7.70-7.68 (m, 2H), 7.45-7.40 (m, 1H), 7.34-7.30 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.97, 136.00, 134.04, 129.29, 128.59. This compound is known.[20]

5. References

(1) M. M. Wang, X. S. Ning, J. P. Qu and Y. B. Kang, *ACS. Catal.* 2017, **7**, 4000- 4003.

(2) G. P . Yang, N. Zhang, N. N. Ma, B. Yu and C. W. Hu, *Adv. Synth. Catal.* 2017, **359**, 926- 932.

(3) J. Das, M. Vellakkaran and D. Banerjee, *J. Org. Chem.* 2019, **84**, 769- 779.

(4) W. Ren, W. Chang, J. Dai, Y. Shi and J. Li, Y. Shi, *J. Am. Chem. Soc.* 2016, **138**, 14864- 14867.

(5) S. F. Yin, Y. Zhou, Z. Li, X. Yang, X. Chen, M. Li and T. Chen, *Synthesis* 2016, **48**, 231- 237.

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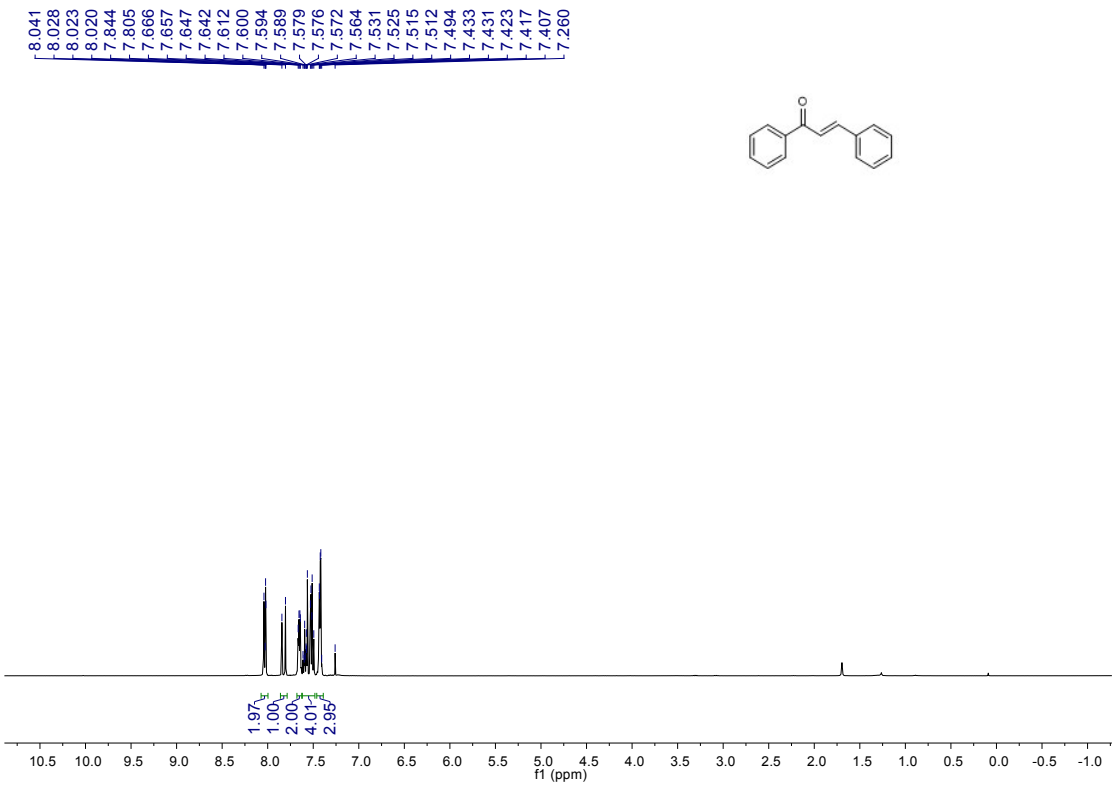
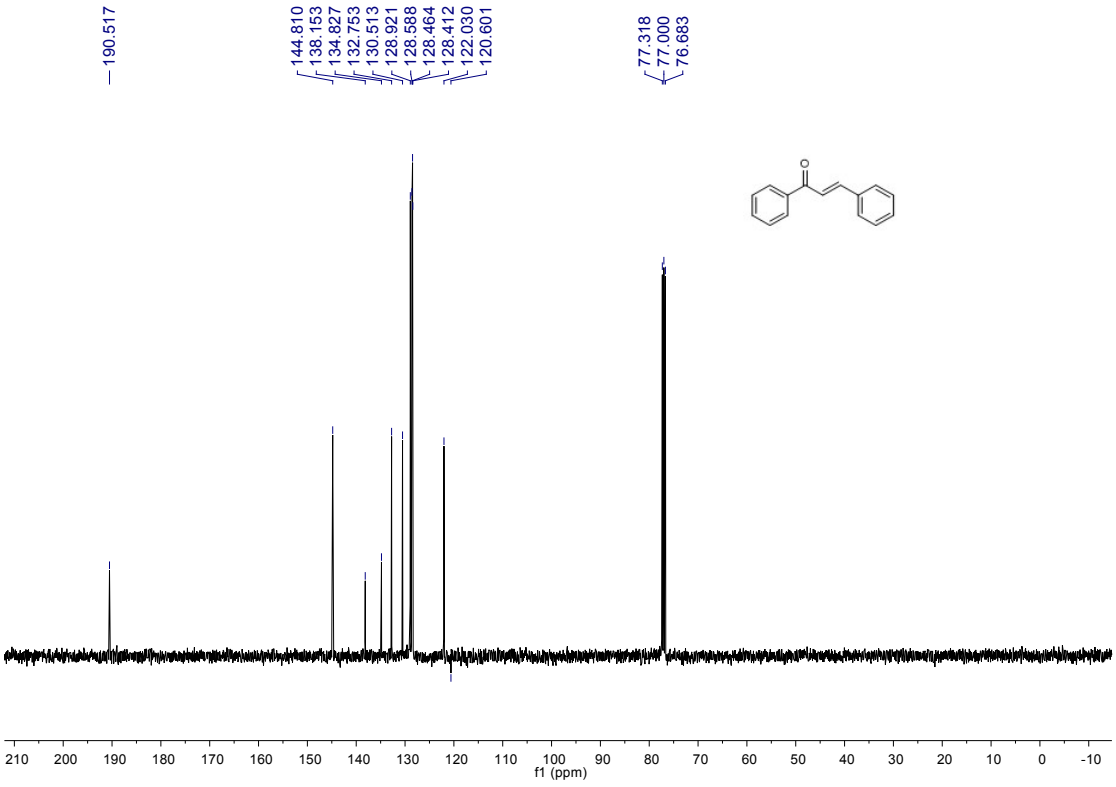
6. Copies of ^{1}H, ^{13}C NMR Spectra of the Products

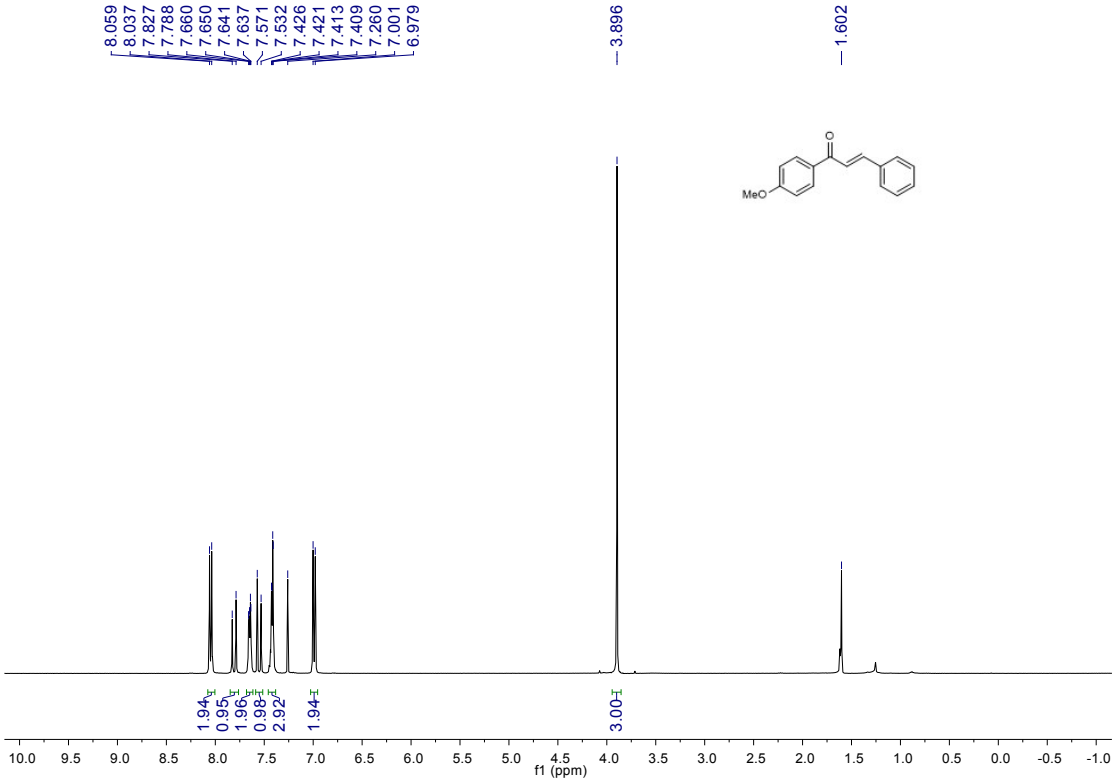
^{1}H NMR Spectrum of chalcone **(2a)**



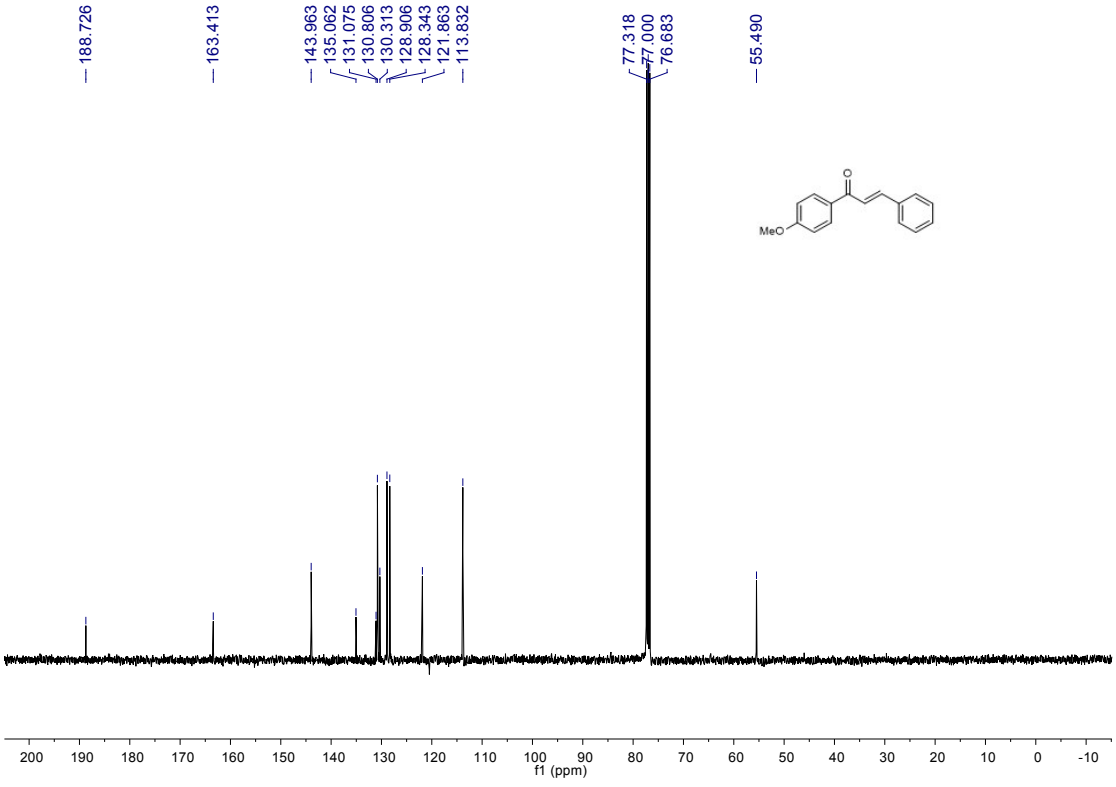
^{13}C NMR Spectrum of chalcone **(2a)**



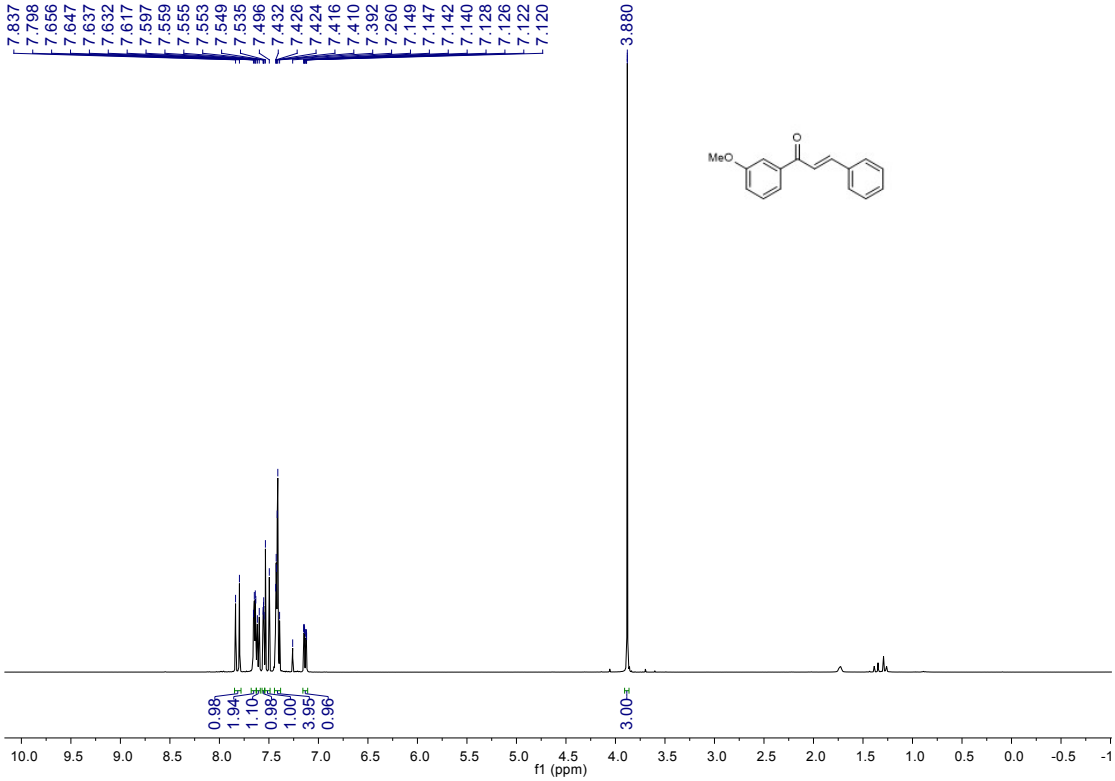
^{1}H NMR Spectrum of (*E*)-1-(4-methoxyphenyl)-3-phenylprop-2-en-1-one **(2b)**



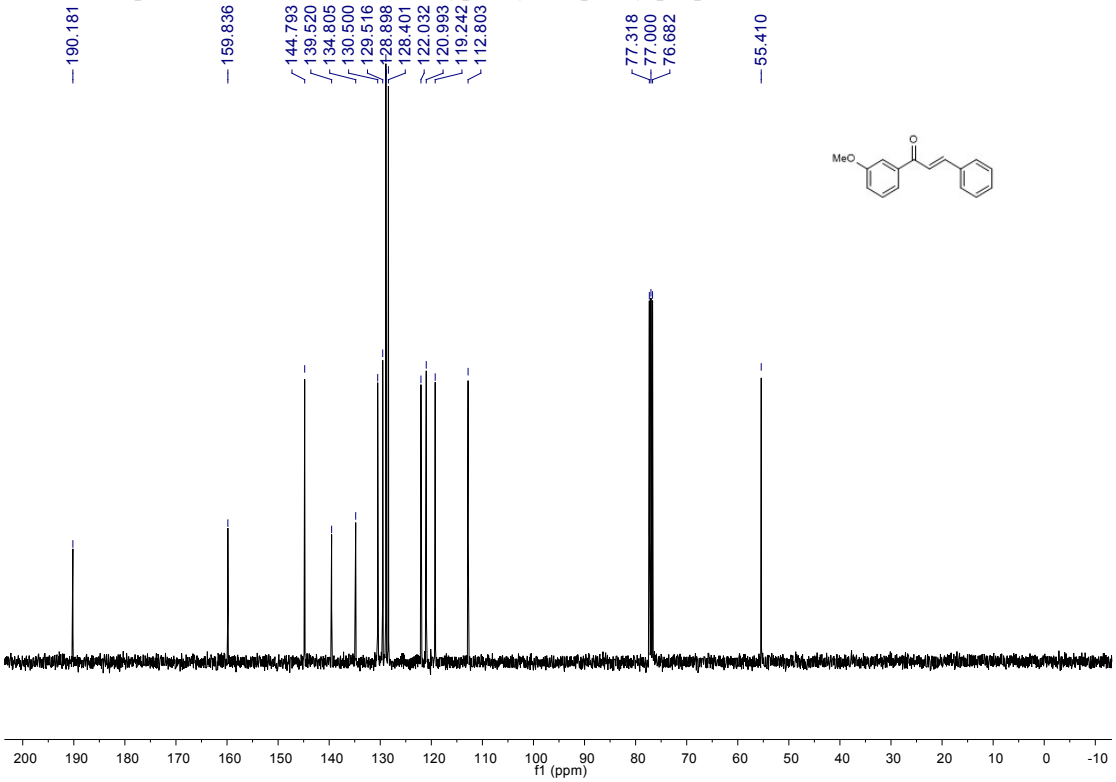
^{13}C NMR Spectrum of (*E*)-1-(4-methoxyphenyl)-3-phenylprop-2-en-1-one **(2b)**



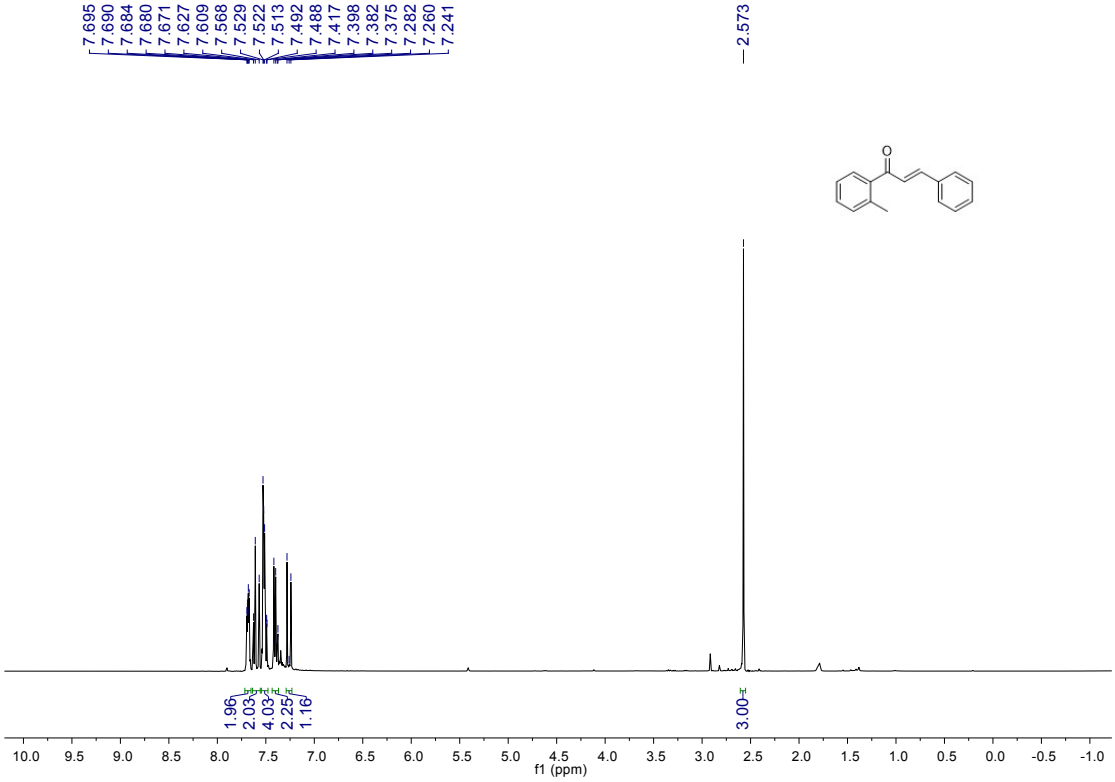
^{1}H NMR Spectrum of (*E*)-1-(3-methoxyphenyl)-3-phenylprop-2-en-1-one **(2c)**



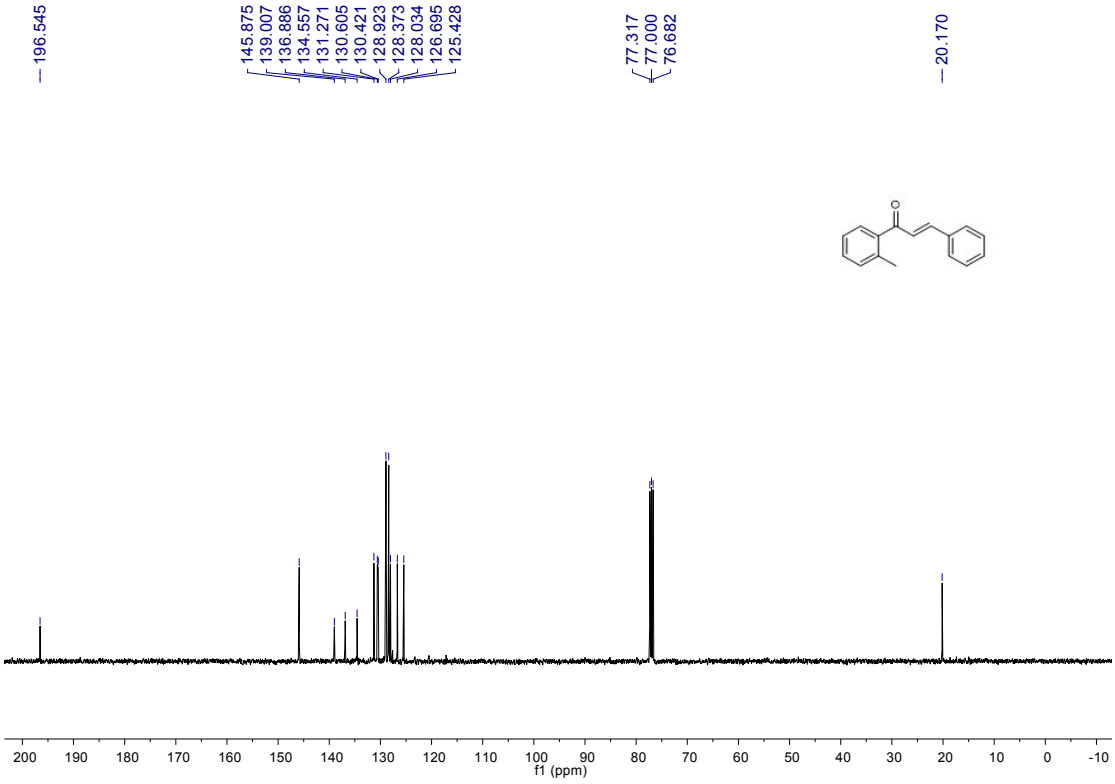
^{13}C NMR Spectrum of (*E*)-1-(3-methoxyphenyl)-3-phenylprop-2-en-1-one **(2c)**



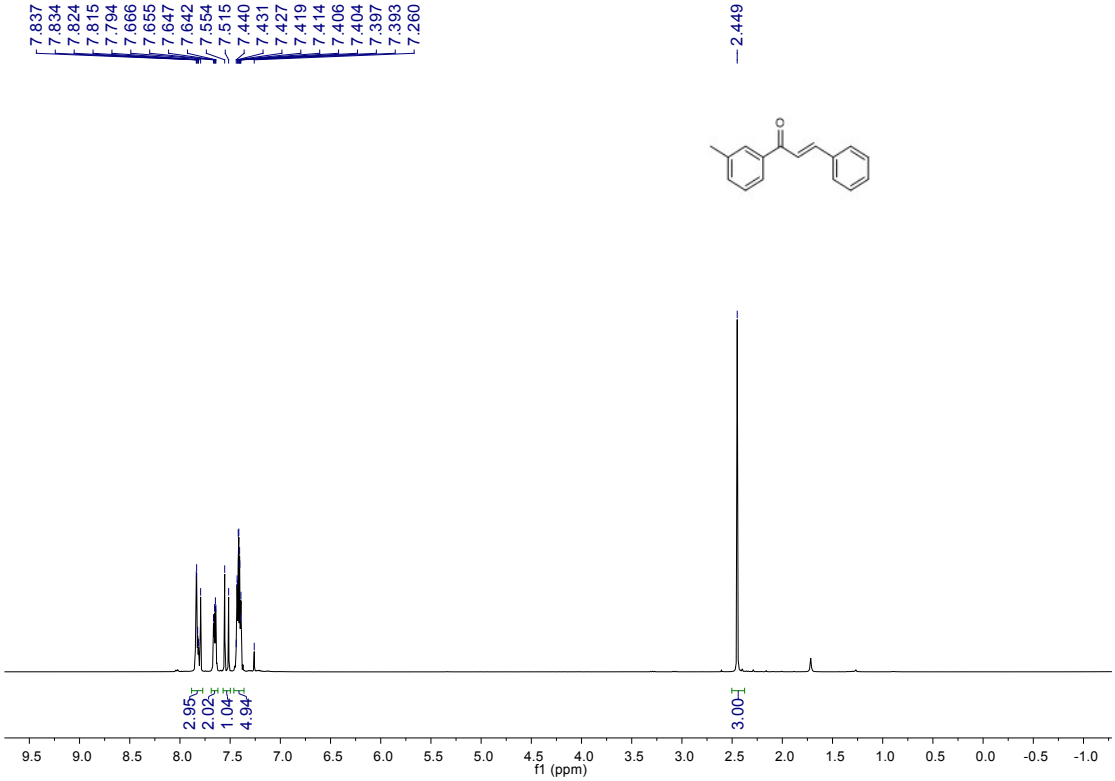
^{1}H NMR Spectrum of (*E*)-3-phenyl-1-(o-tolyl)prop-2-en-1-one **(2d)**



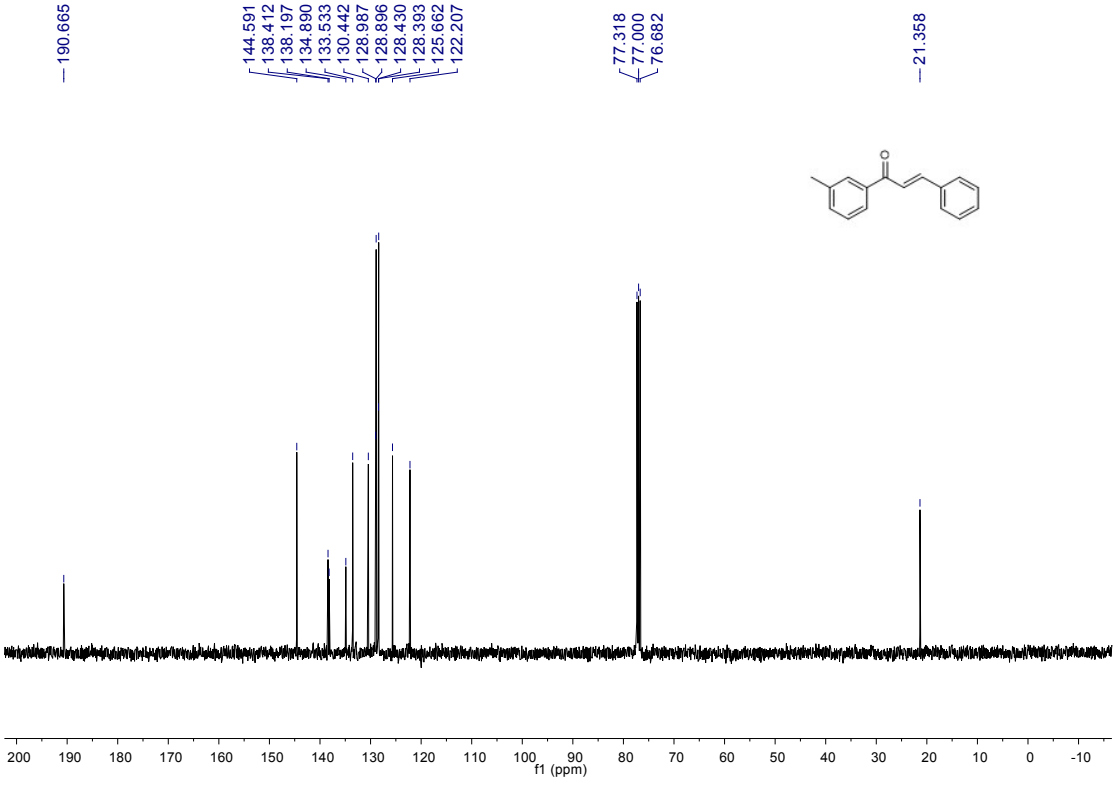
^{13}C NMR Spectrum of (*E*)-3-phenyl-1-(o-tolyl)prop-2-en-1-one **(2d)**



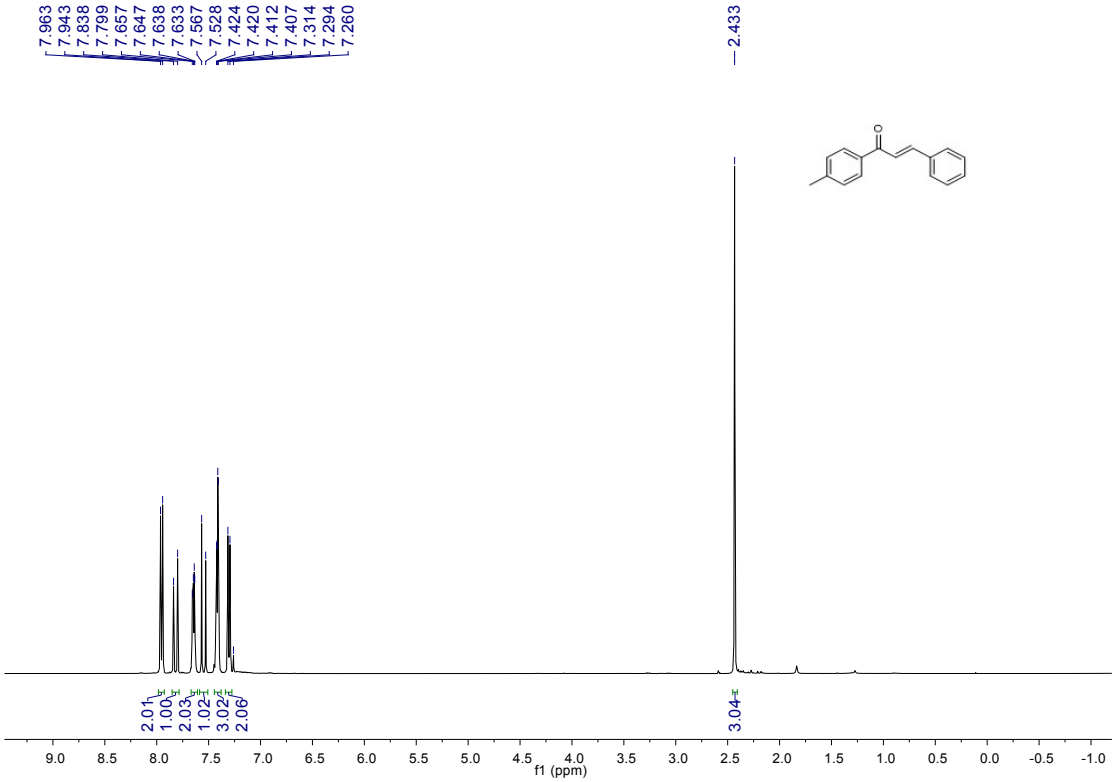
^{1}H NMR Spectrum of (*E*)-3-phenyl-1-(m-tolyl)prop-2-en-1-one **(2e)**



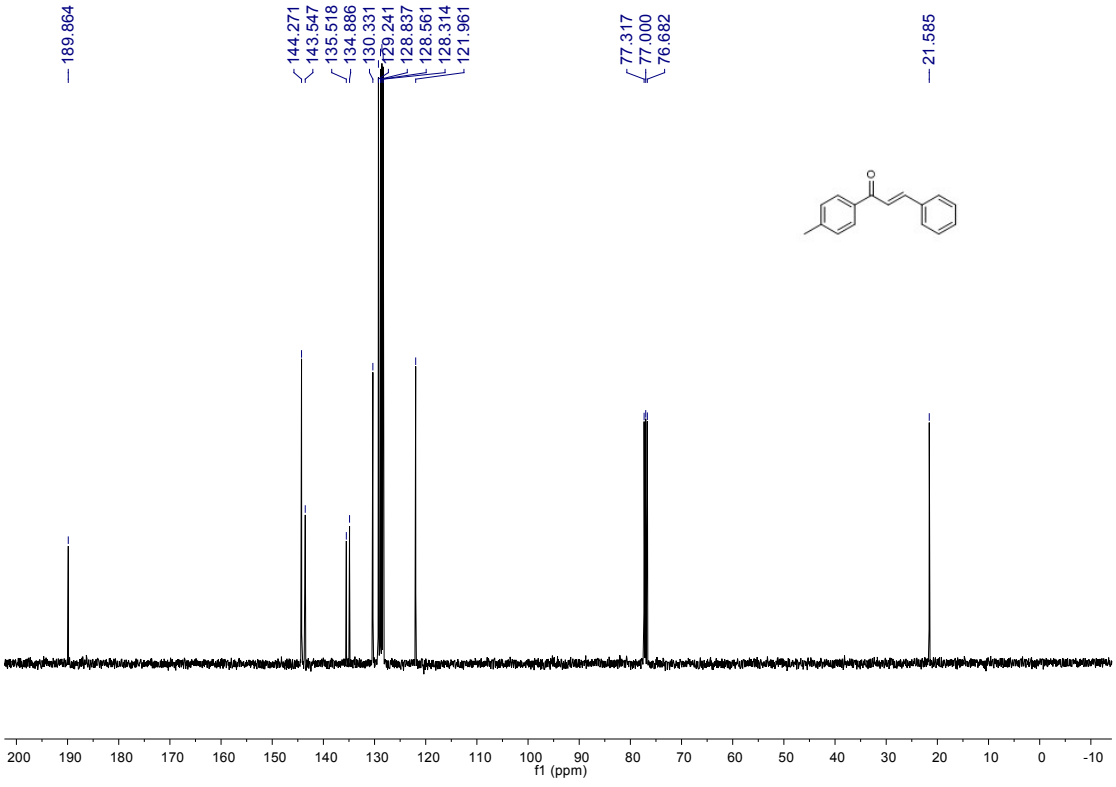
^{13}C NMR Spectrum of (*E*)-3-phenyl-1-(m-tolyl)prop-2-en-1-one **(2e)**



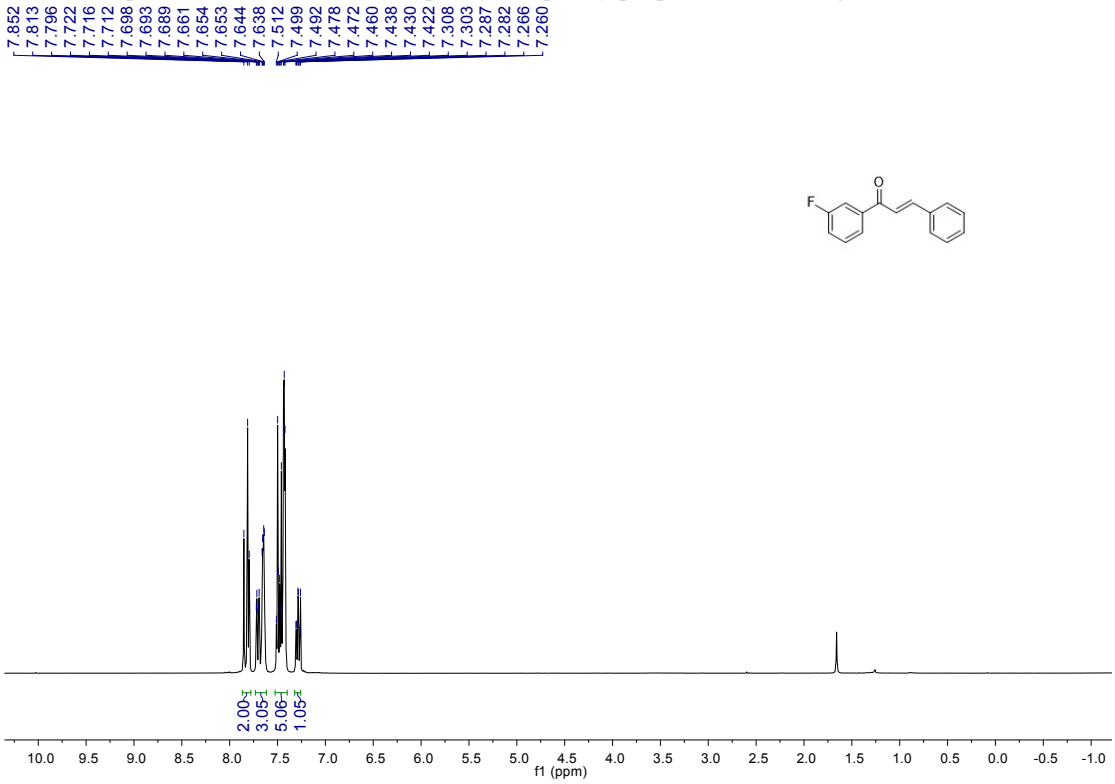
^{1}H NMR Spectrum of (*E*)-3-phenyl-1-(p-tolyl)prop-2-en-1-one **(2f)**



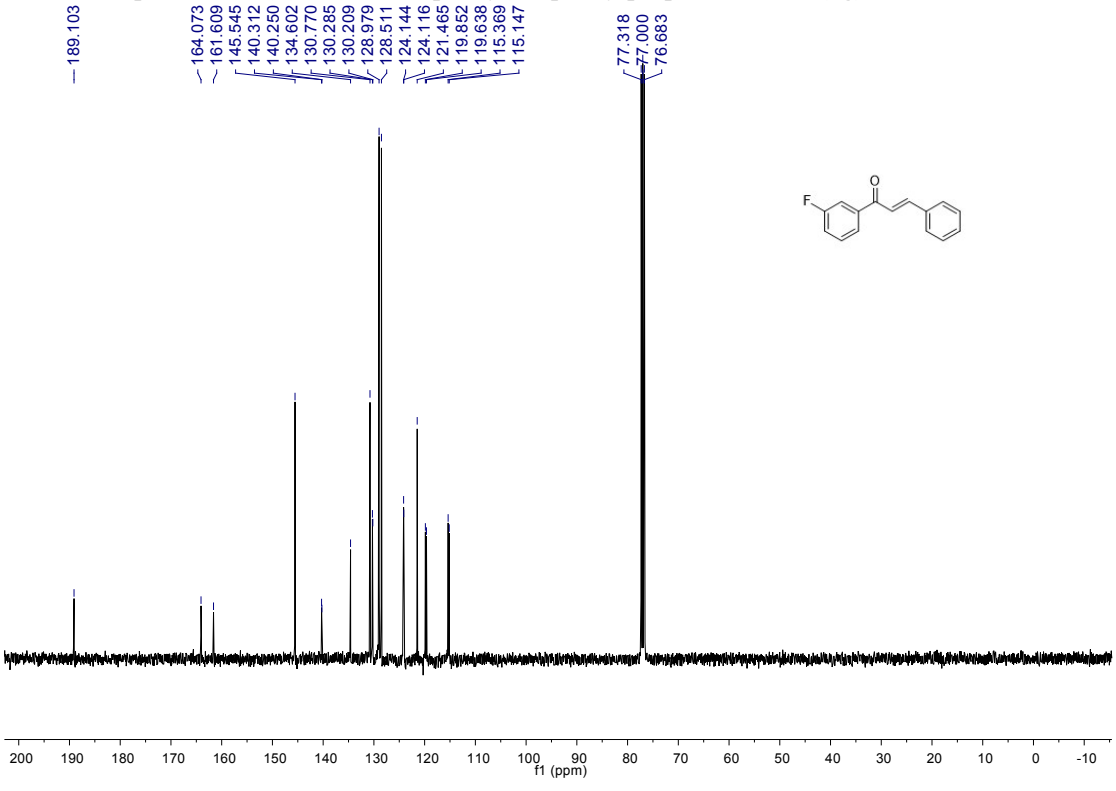
^{13}C NMR Spectrum of (*E*)-3-phenyl-1-(p-tolyl)prop-2-en-1-one **(2f)**



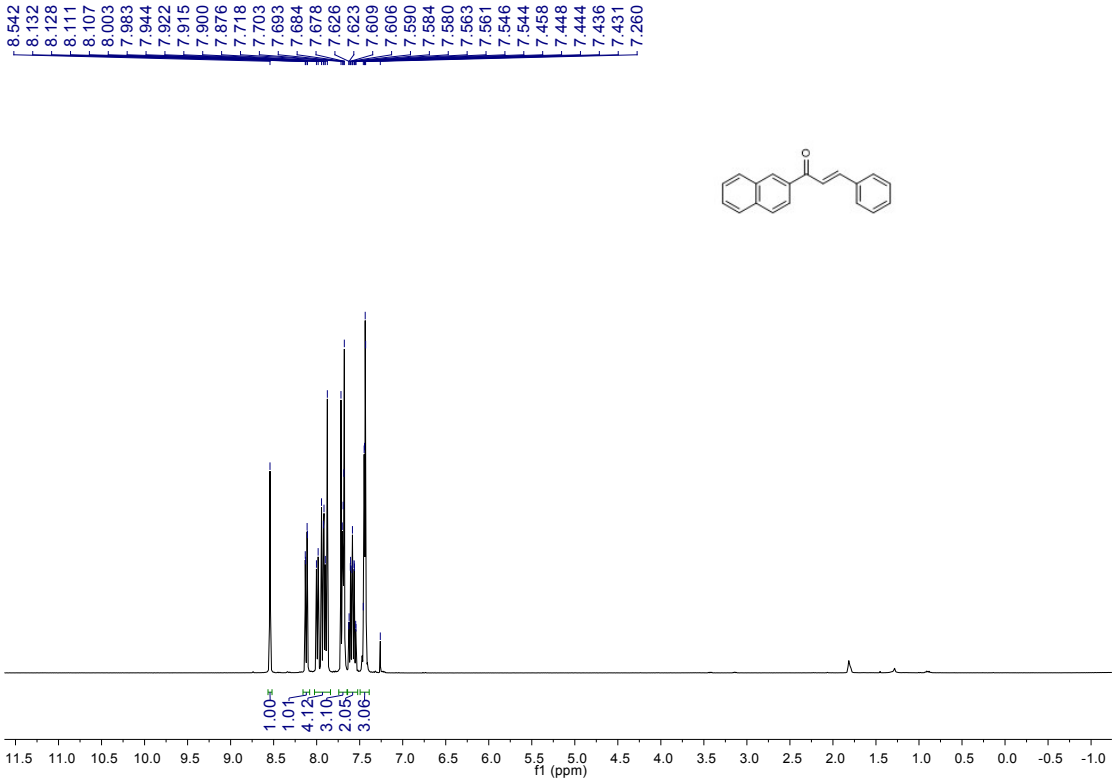
^{1}H NMR Spectrum of (*E*)-1-(3-fluorophenyl)-3-phenylprop-2-en-1-one **(2g)**



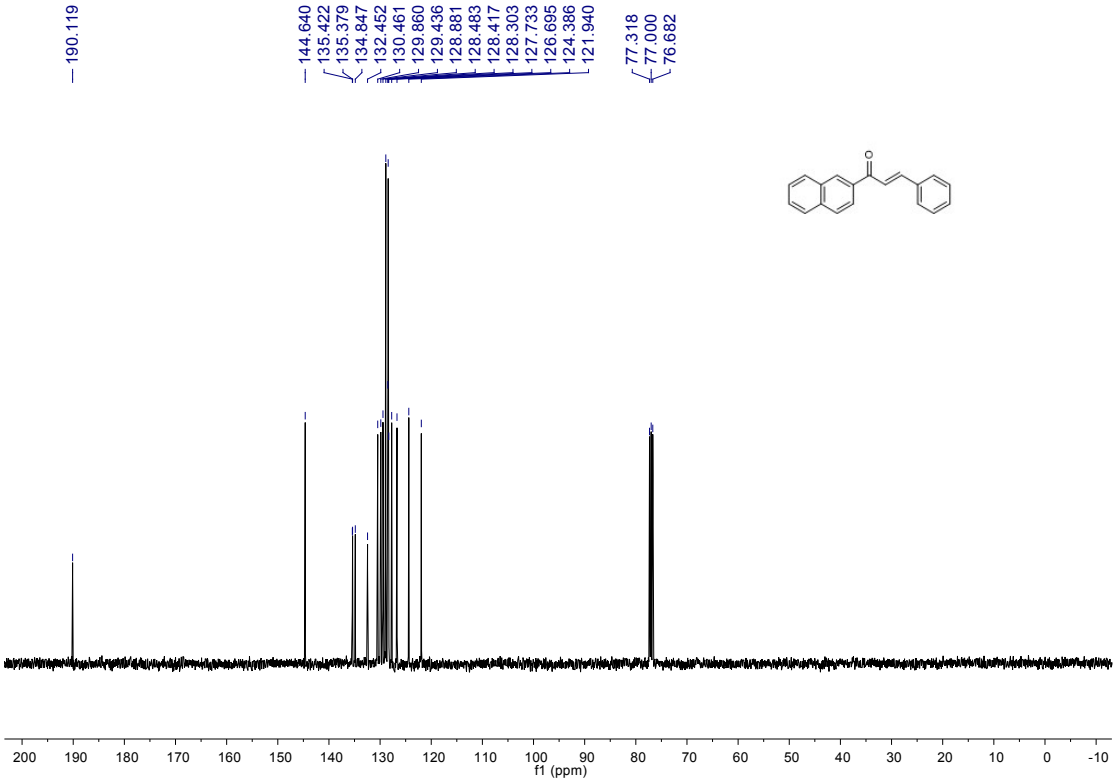
^{13}C NMR Spectrum of (*E*)-1-(3-fluorophenyl)-3-phenylprop-2-en-1-one **(2g)**



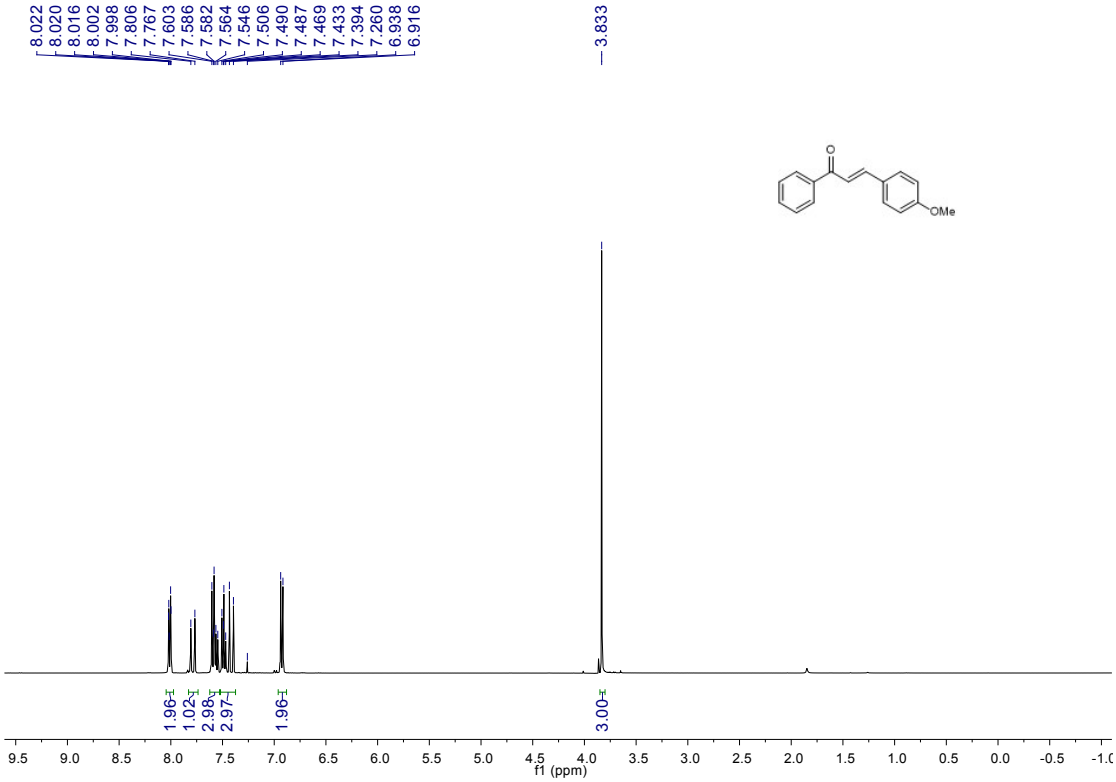
^{1}H NMR Spectrum of (*E*)-1-(naphthalen-2-yl)-3-phenylprop-2-en-1-one **(2h)**



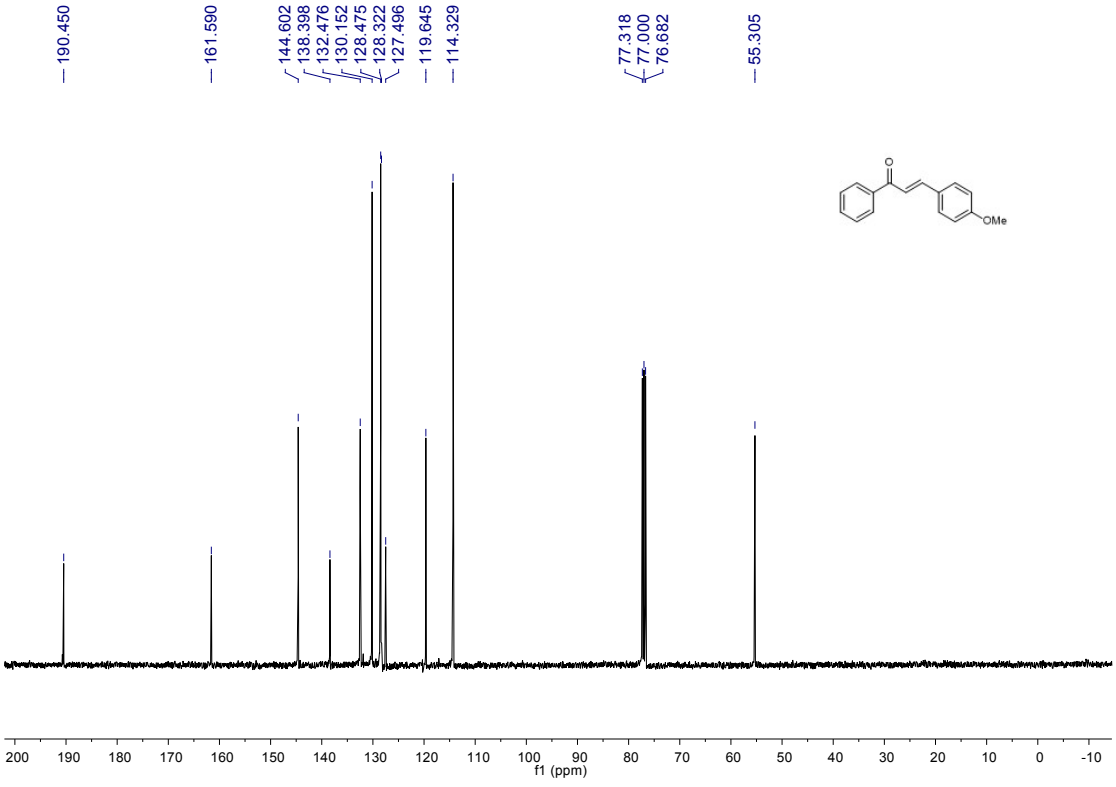
^{13}C NMR Spectrum of (*E*)-1-(naphthalen-2-yl)-3-phenylprop-2-en-1-one **(2h)**



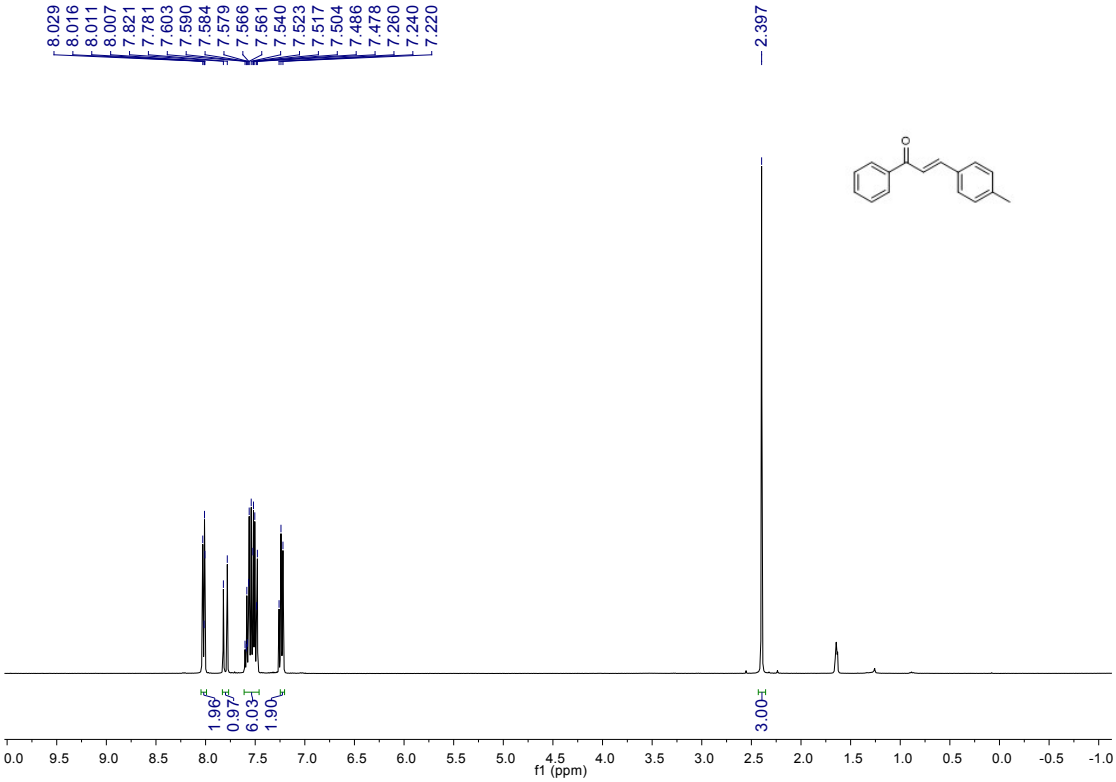
^{1}H NMR Spectrum of (*E*)-3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one **(2i)**



^{13}C NMR Spectrum of (*E*)-3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one **(2i)**



^{1}H NMR Spectrum of (*E*)-1-phenyl-3-(p-tolyl)prop-2-en-1-one **(2j)**



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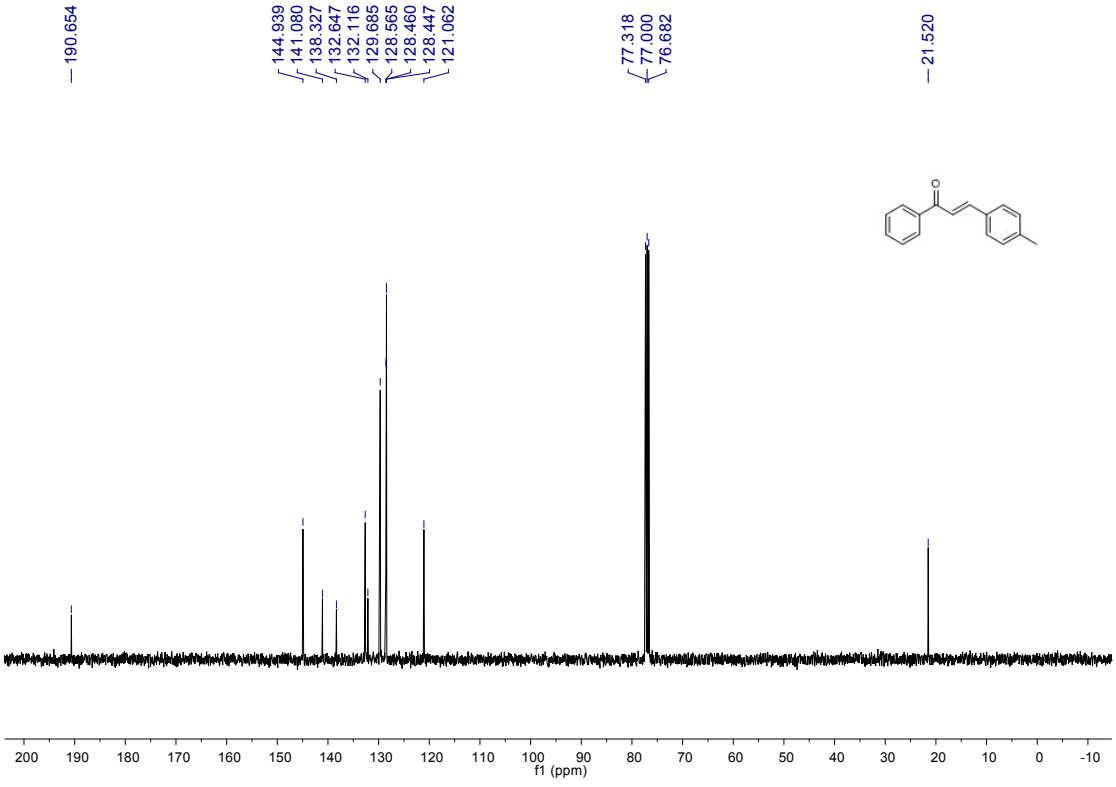
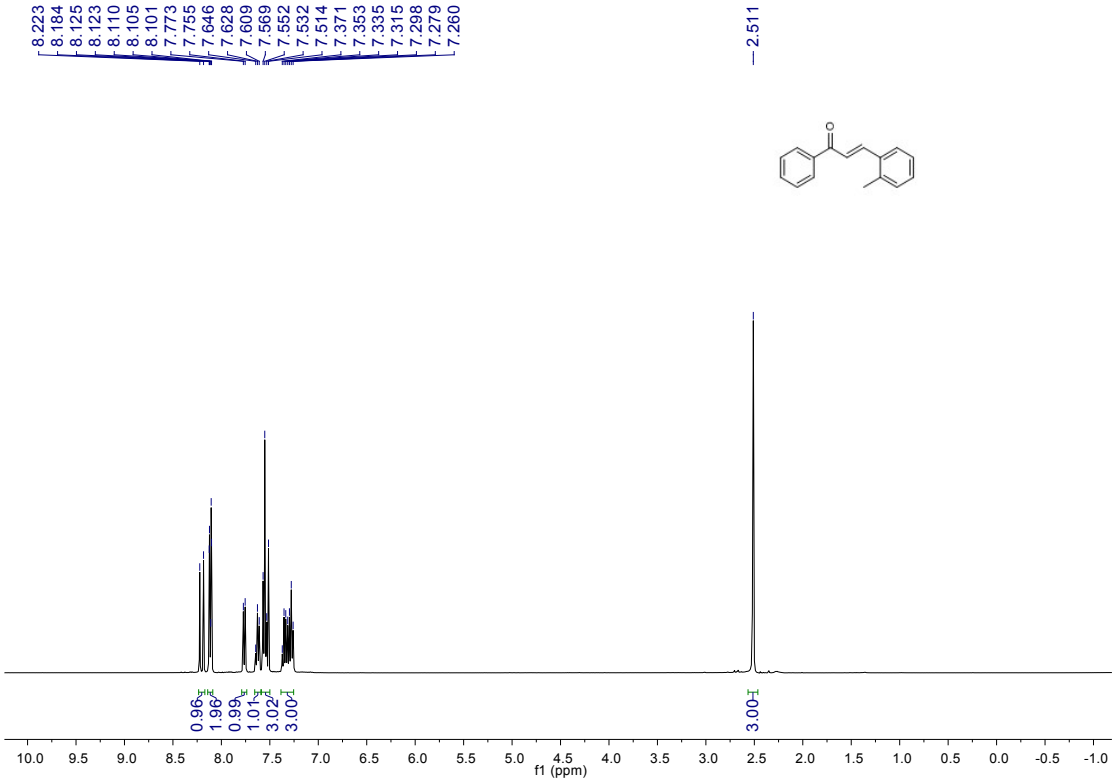
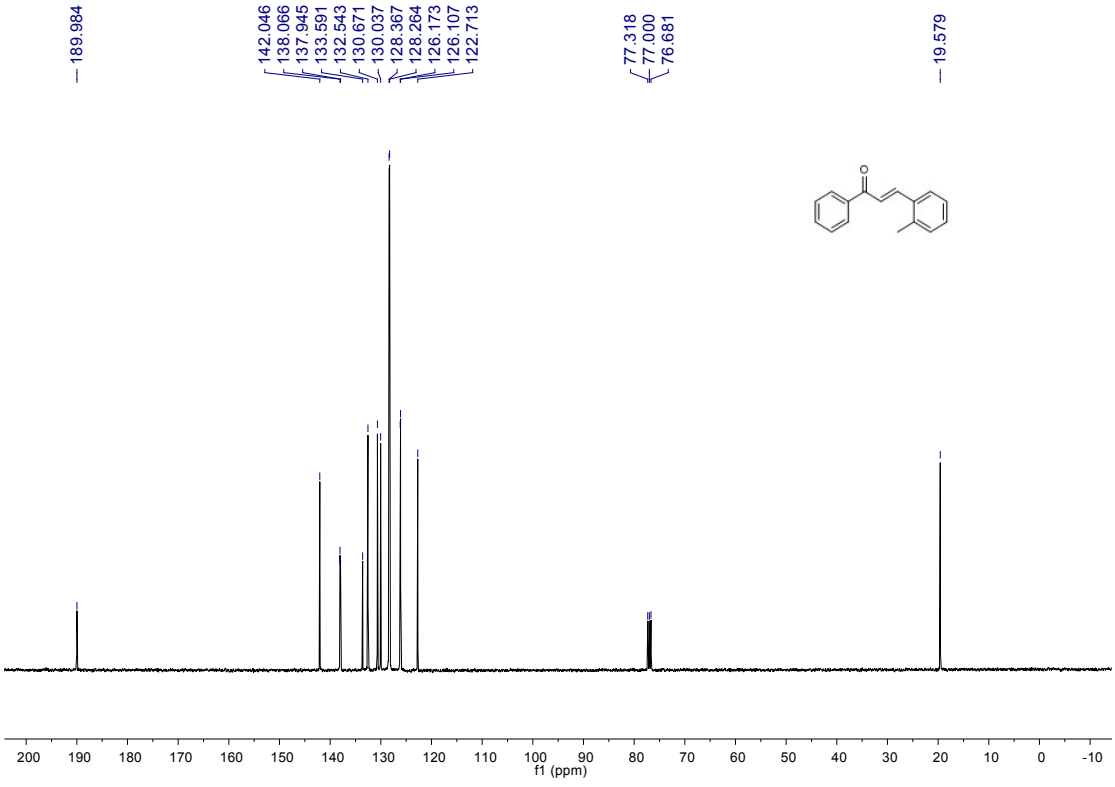


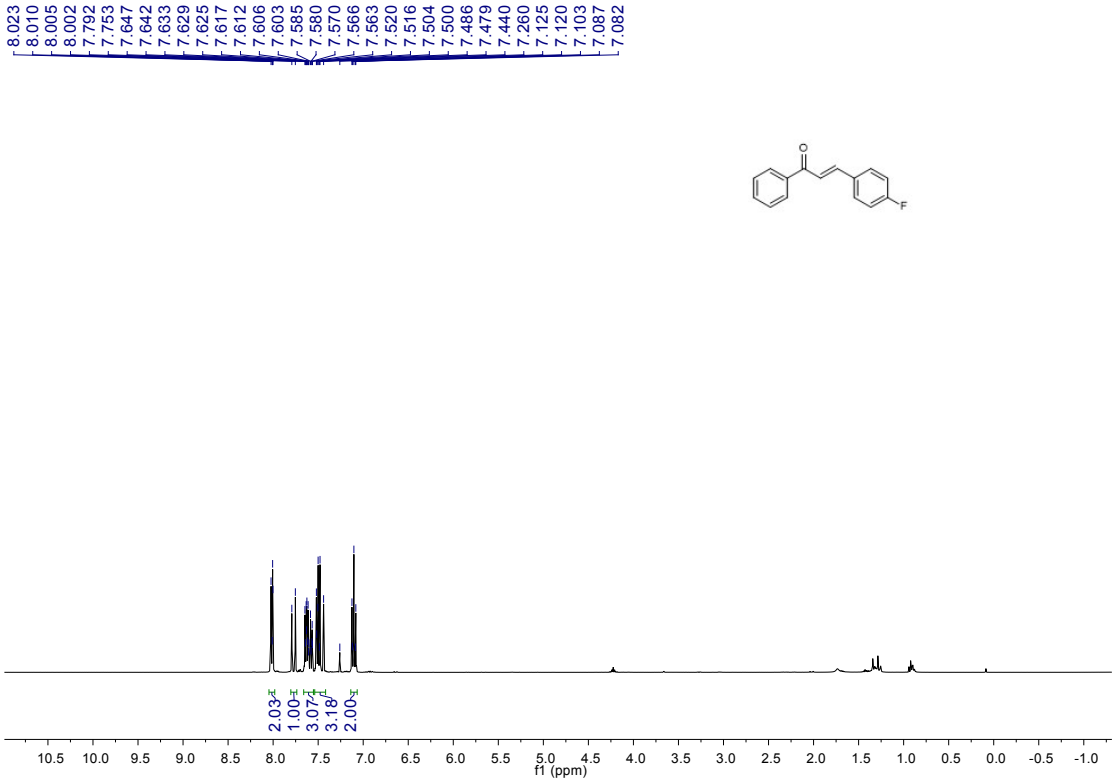
^{1}H NMR Spectrum of (*E*)-1-phenyl-3-(o-tolyl)prop-2-en-1-one **(2k)**



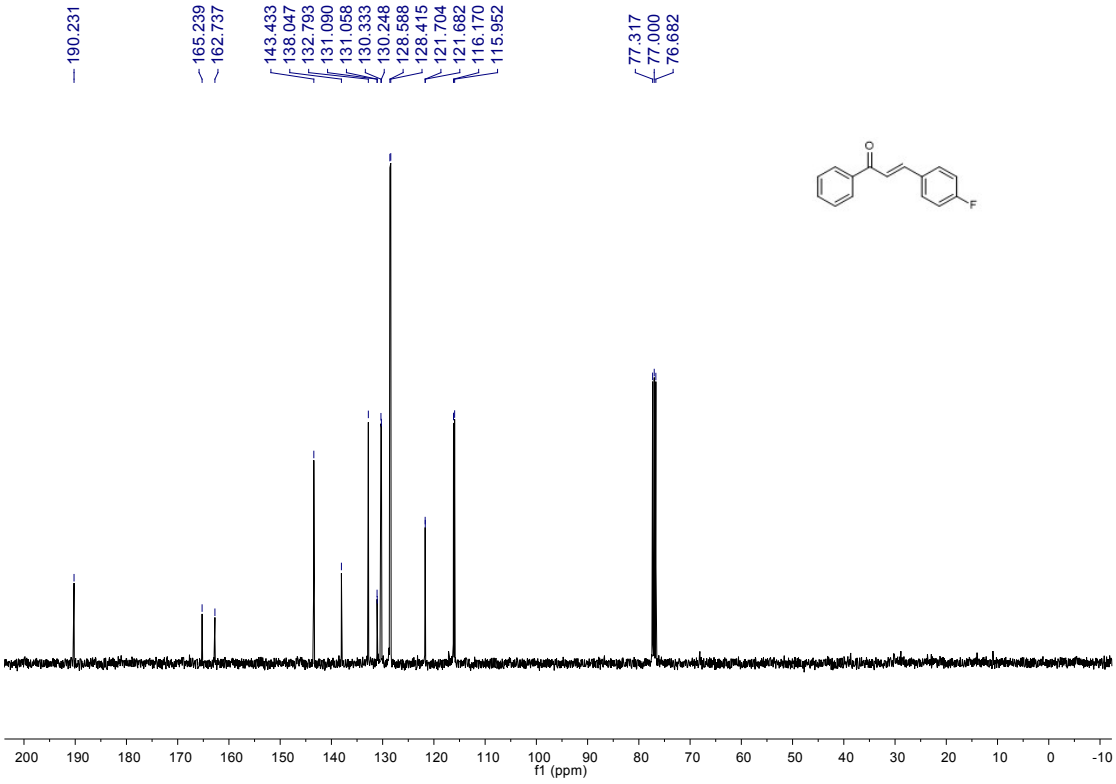
^{13}C NMR Spectrum of (*E*)-1-phenyl-3-(o-tolyl)prop-2-en-1-one **(2k)**



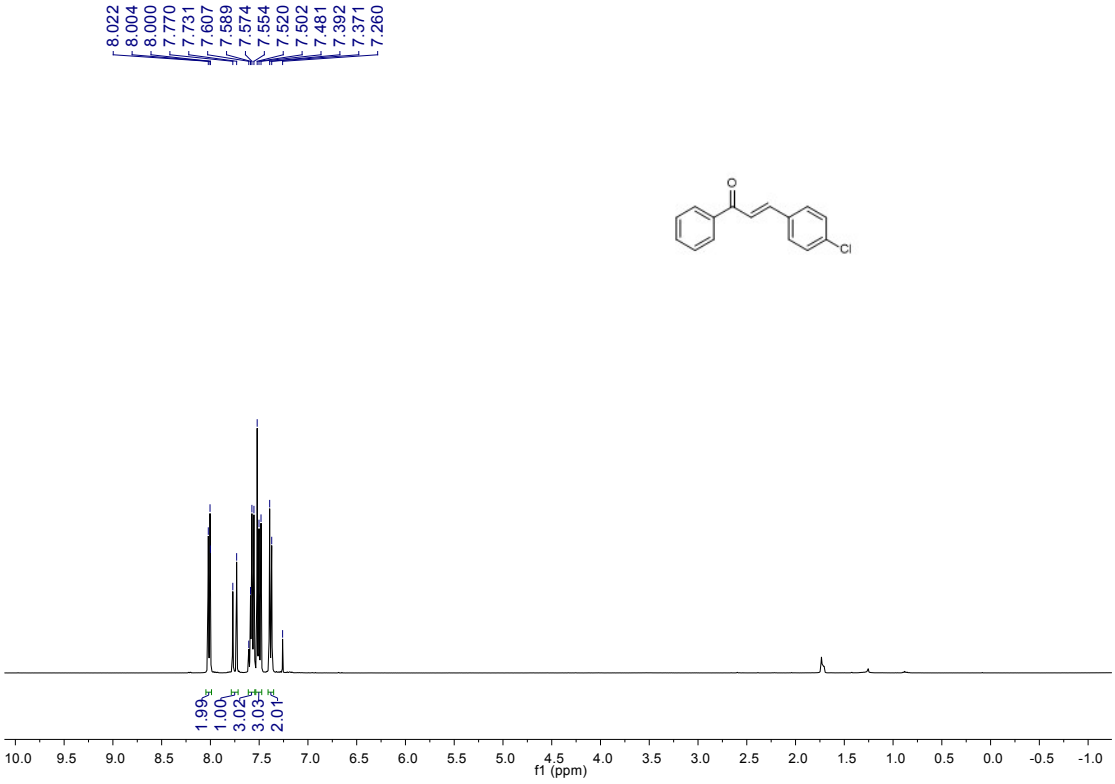
^{1}H NMR Spectrum of (*E*)-3-(4-fluorophenyl)-1-phenylprop-2-en-1-one **(2l)**



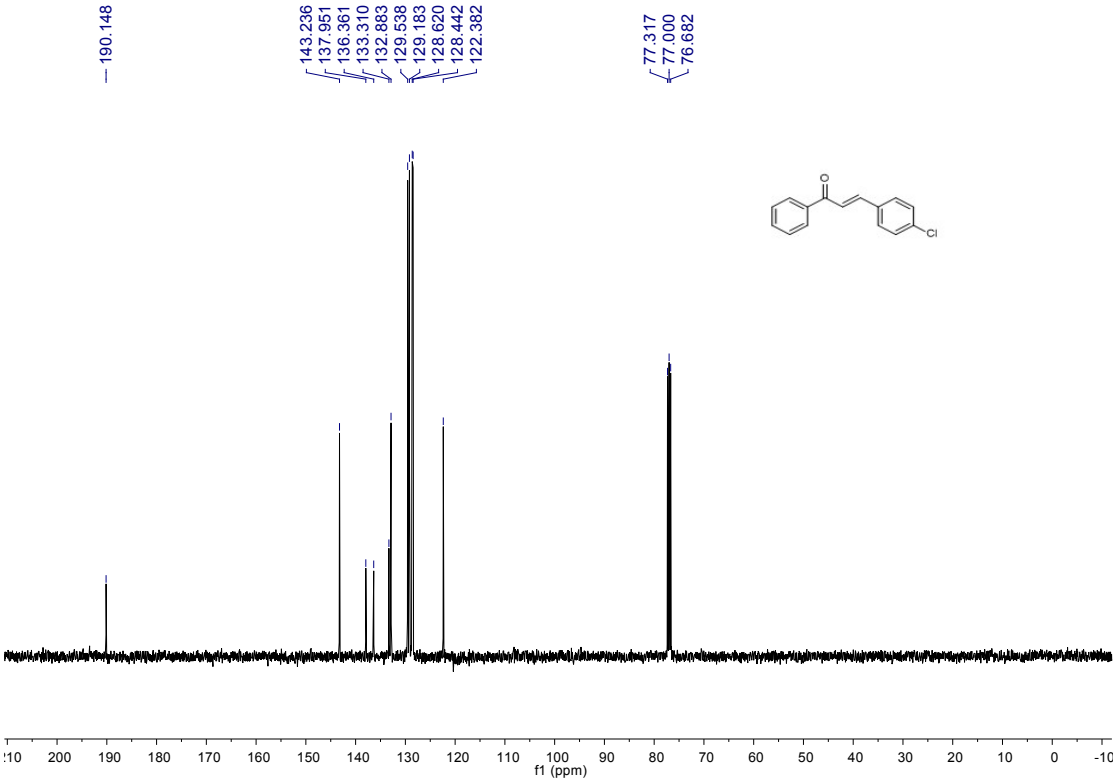
^{13}C NMR Spectrum of (*E*)-3-(4-fluorophenyl)-1-phenylprop-2-en-1-one **(2l)**



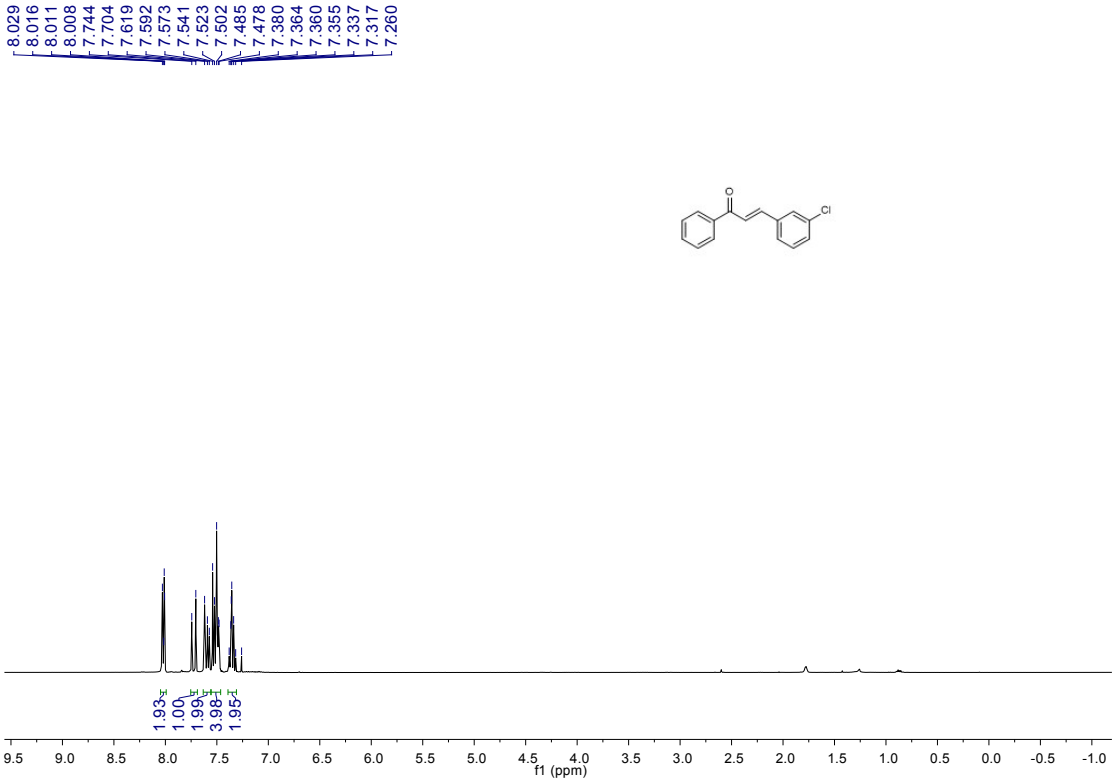
^{1}H NMR Spectrum of (*E*)-3-(4-chlorophenyl)-1-phenylprop-2-en-1-one **(2m)**



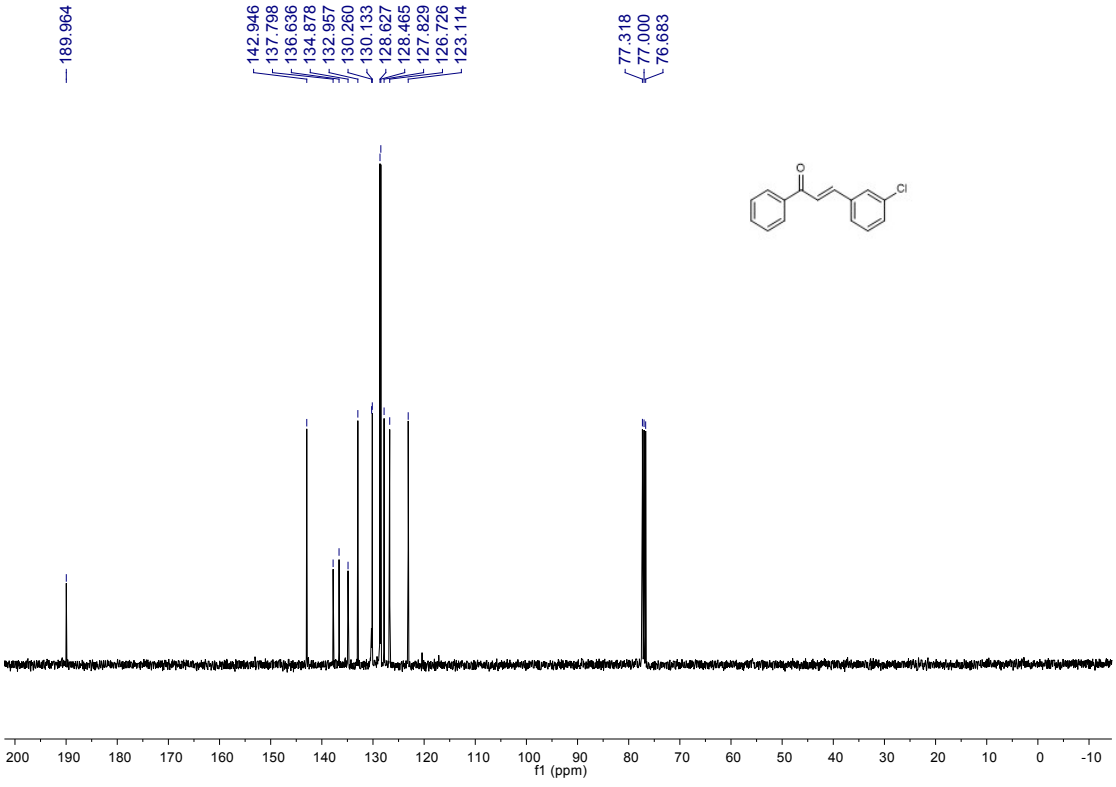
^{13}C NMR Spectrum of (*E*)-3-(4-chlorophenyl)-1-phenylprop-2-en-1-one **(2m)**



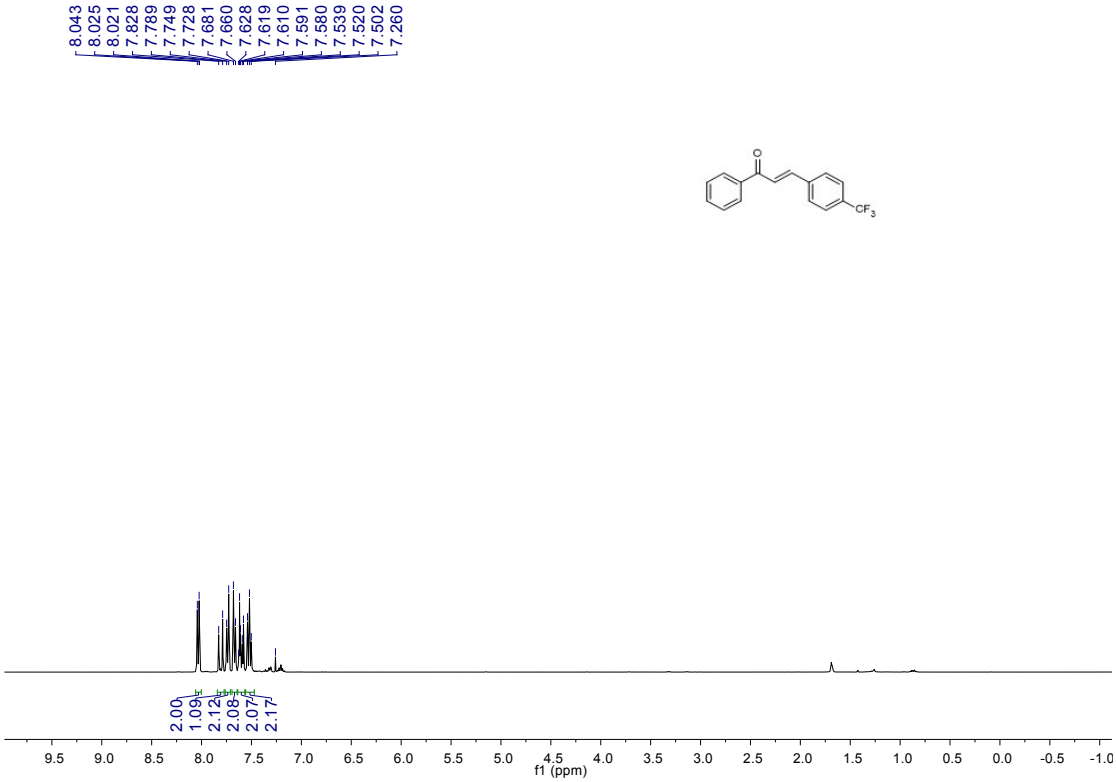
^{1}H NMR Spectrum of (*E*)-3-(3-chlorophenyl)-1-phenylprop-2-en-1-one **(2n)**



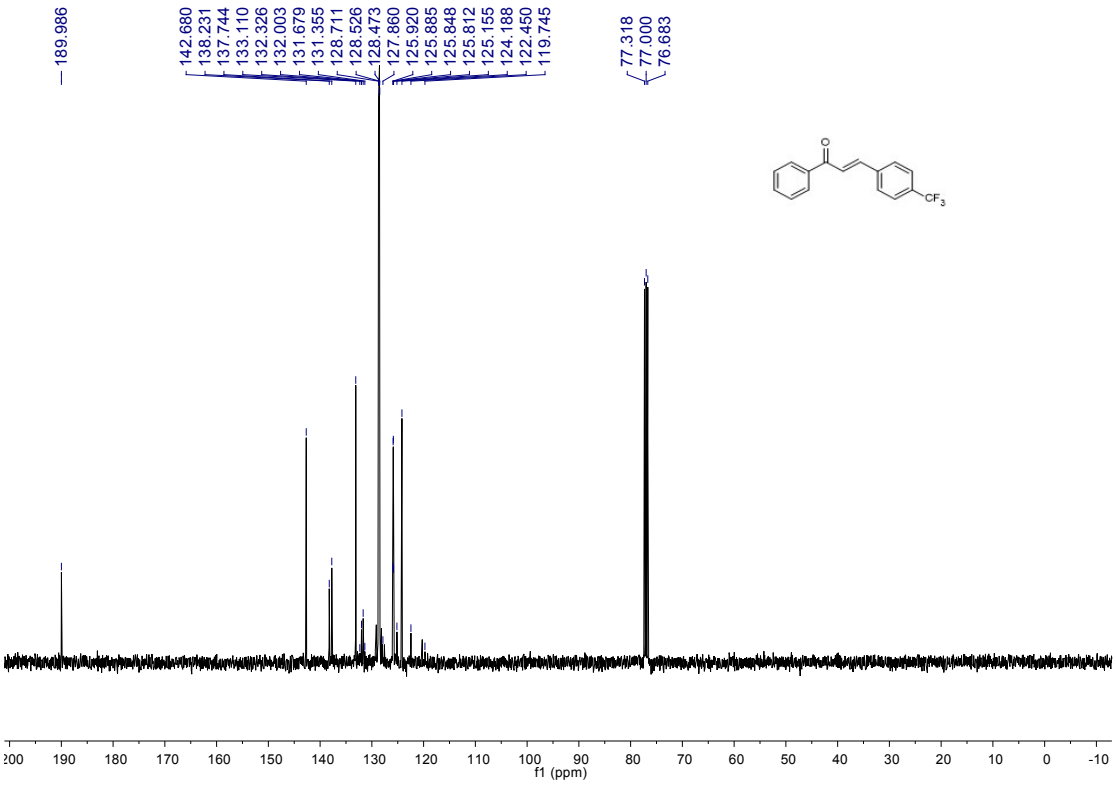
^{13}C NMR Spectrum of (*E*)-3-(3-chlorophenyl)-1-phenylprop-2-en-1-one **(2n)**



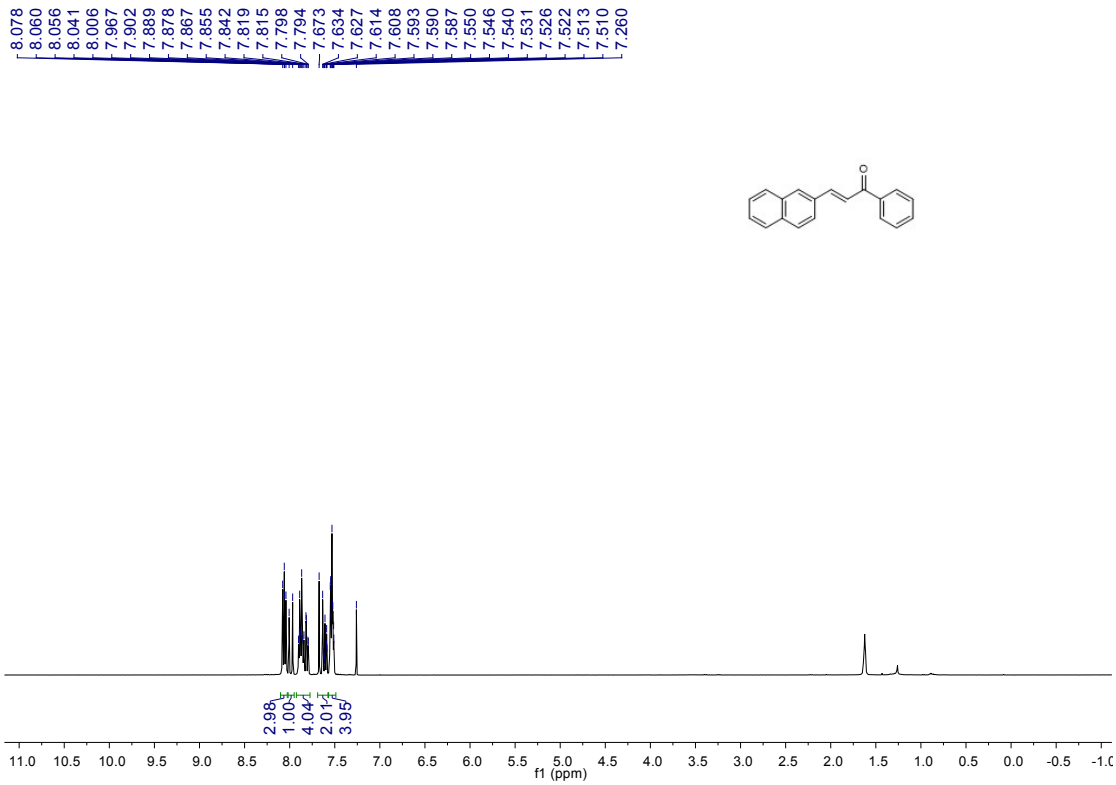
^{1}H NMR Spectrum of (*E*)-1-phenyl-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-one **(2o)**



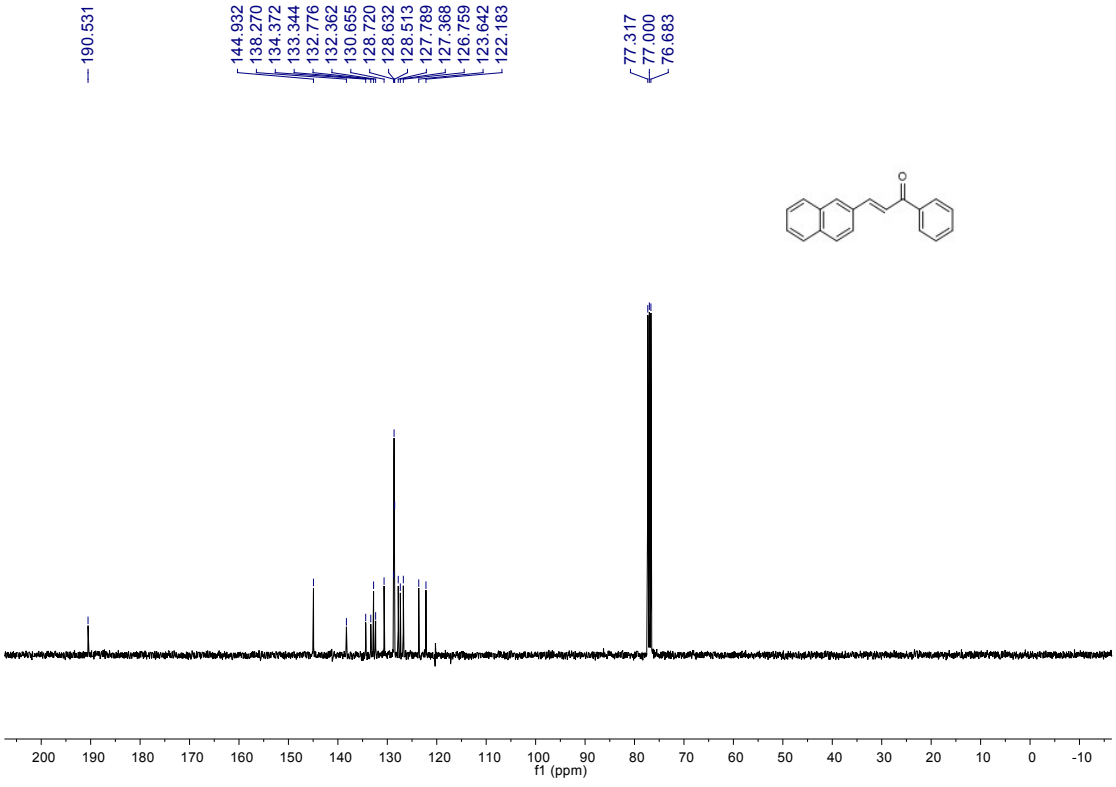
^{13}C NMR Spectrum of (*E*)-1-phenyl-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-one **(2o)**



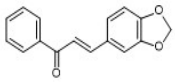
^{1}H NMR Spectrum of (*E*)-3-(naphthalen-2-yl)-1-phenylprop-2-en-1-one **(2p)**

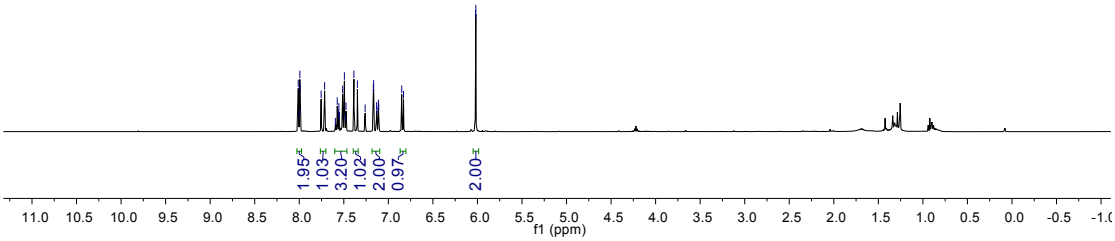


^{13}C NMR Spectrum of (*E*)-3-(naphthalen-2-yl)-1-phenylprop-2-en-1-one **(2p)**

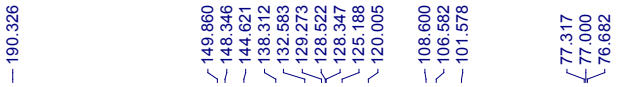


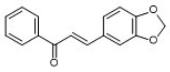
^{1}H NMR Spectrum of (*E*)-3-(benzo[d][1,3]dioxol-5-yl)-1-phenylprop-2-en-1-one **(2q)**

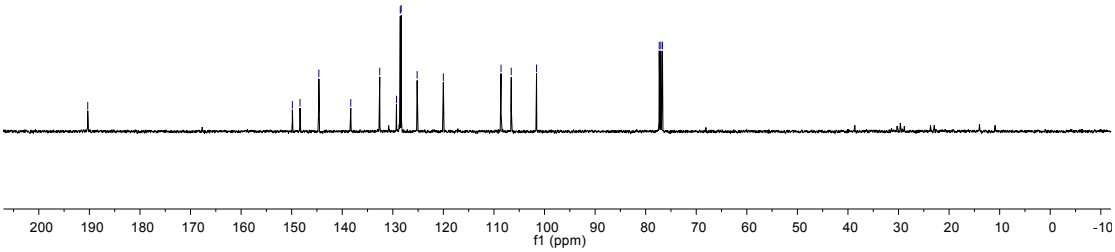




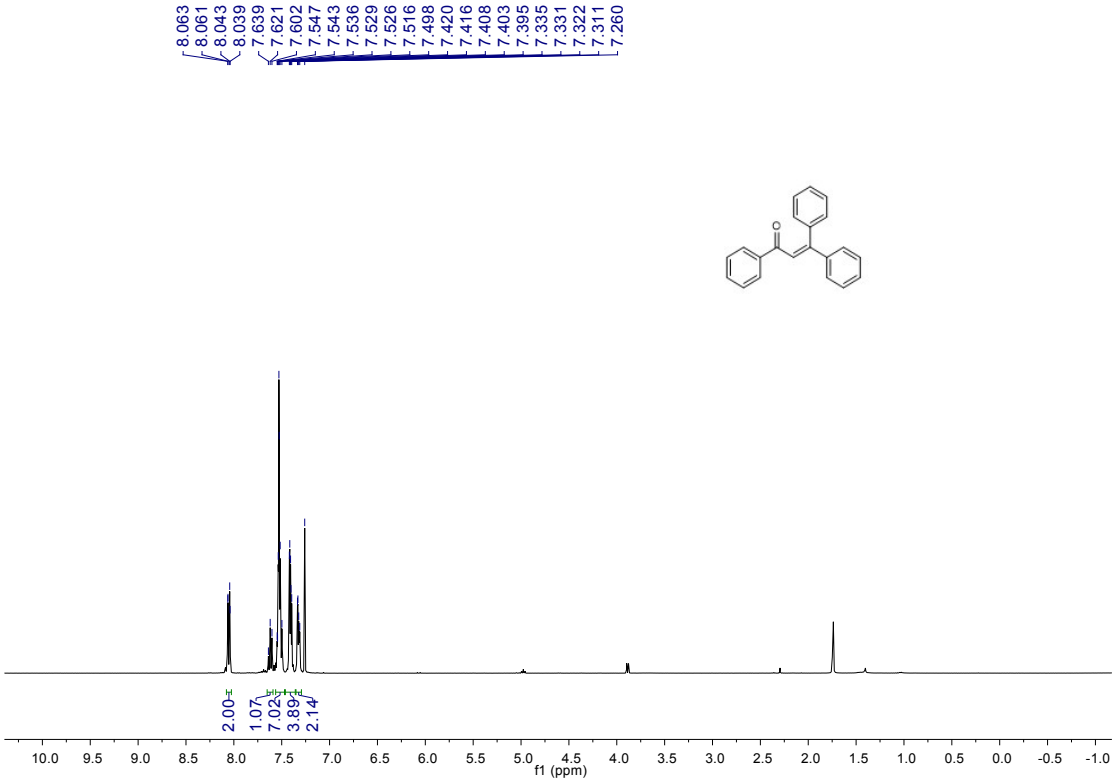
^{13}C NMR Spectrum of (*E*)-3-(benzo[d][1,3]dioxol-5-yl)-1-phenylprop-2-en-1-one **(2q)**



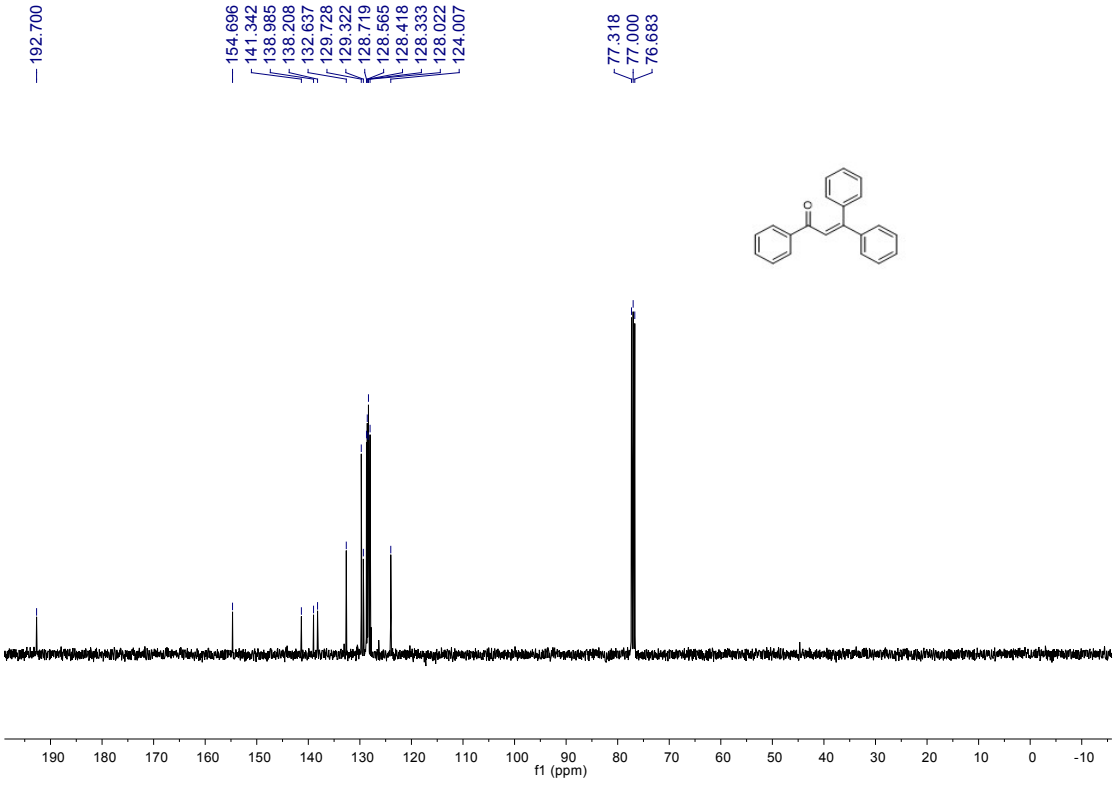




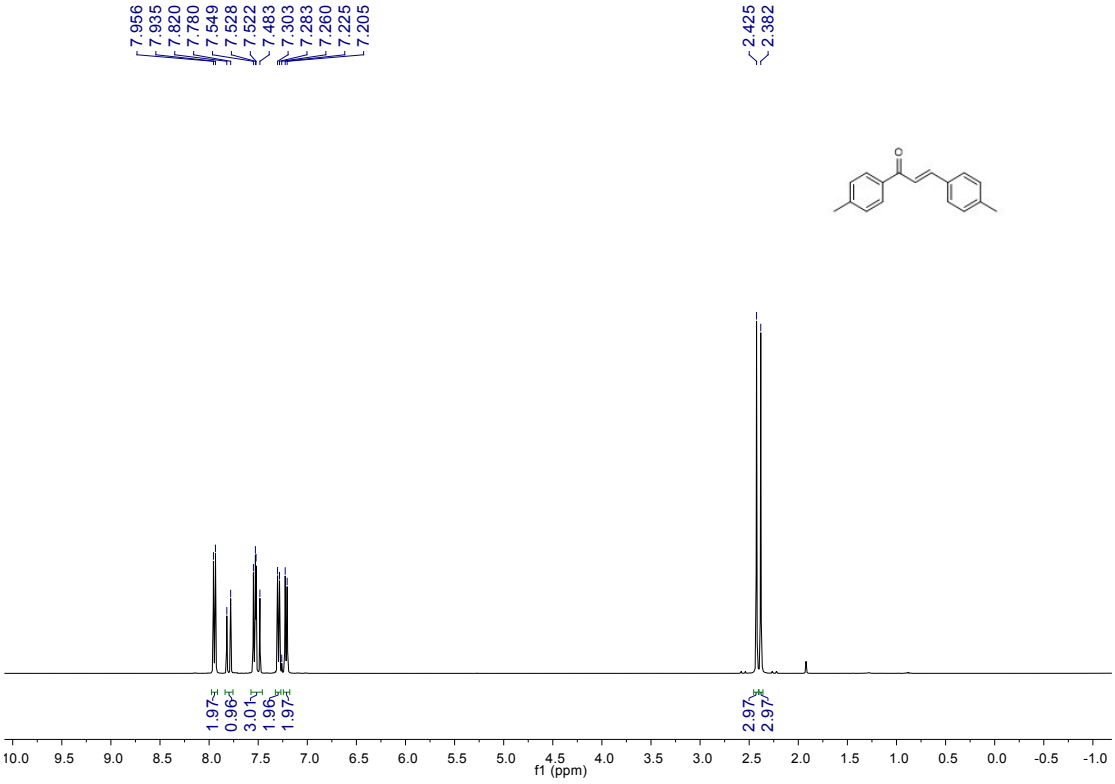
^{1}H NMR Spectrum of 1,3,3-triphenylprop-2-en-1-one **(2r)**



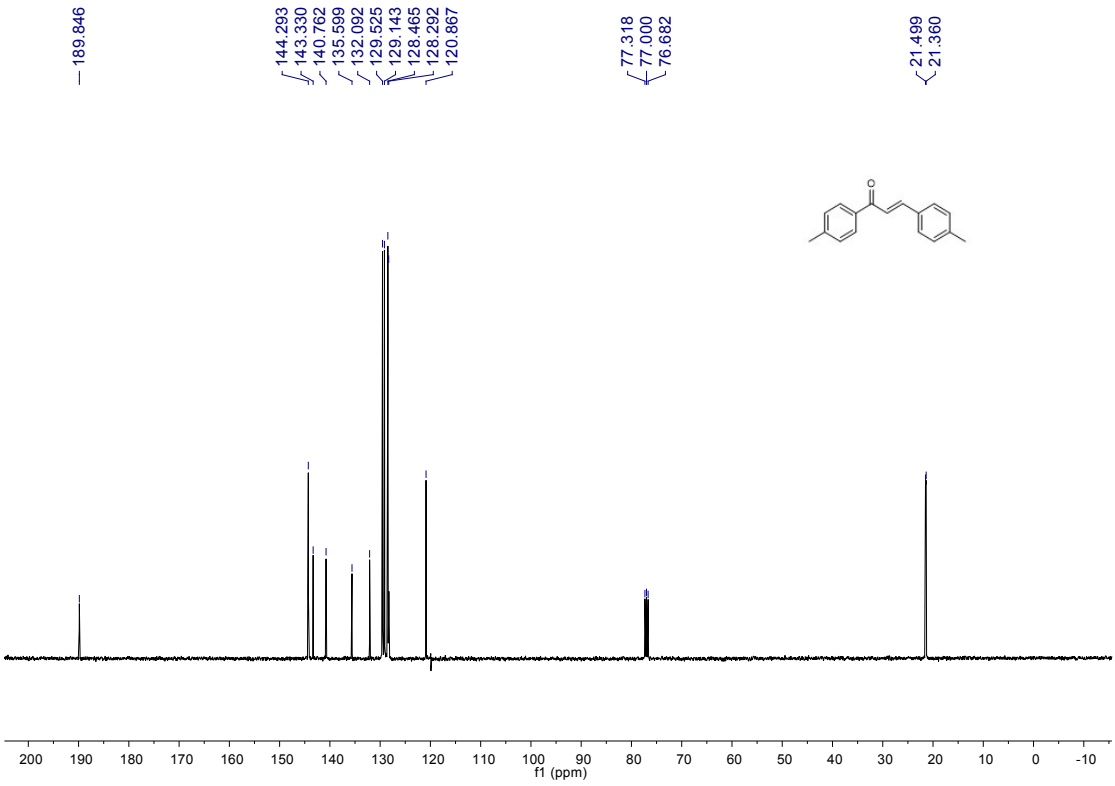
^{13}C NMR Spectrum of 1,3,3-triphenylprop-2-en-1-one **(2r)**



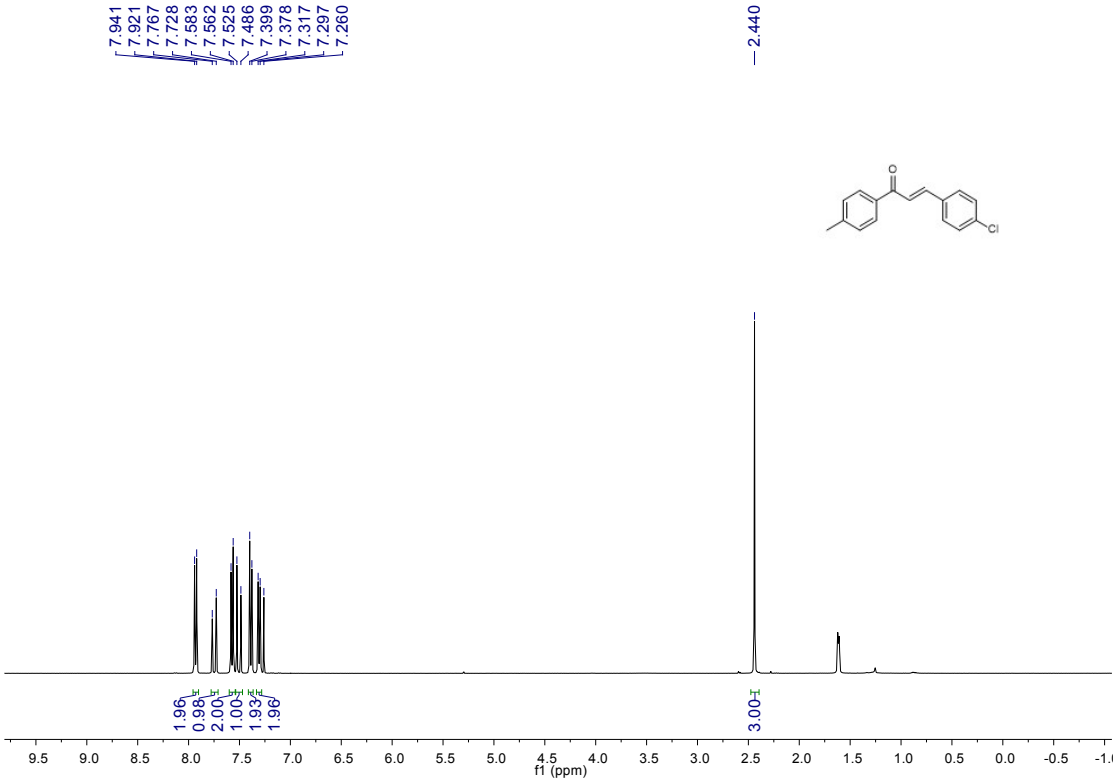
^{1}H NMR Spectrum of (*E*)-1,3-di-p-tolylprop-2-en-1-one **(2s)**



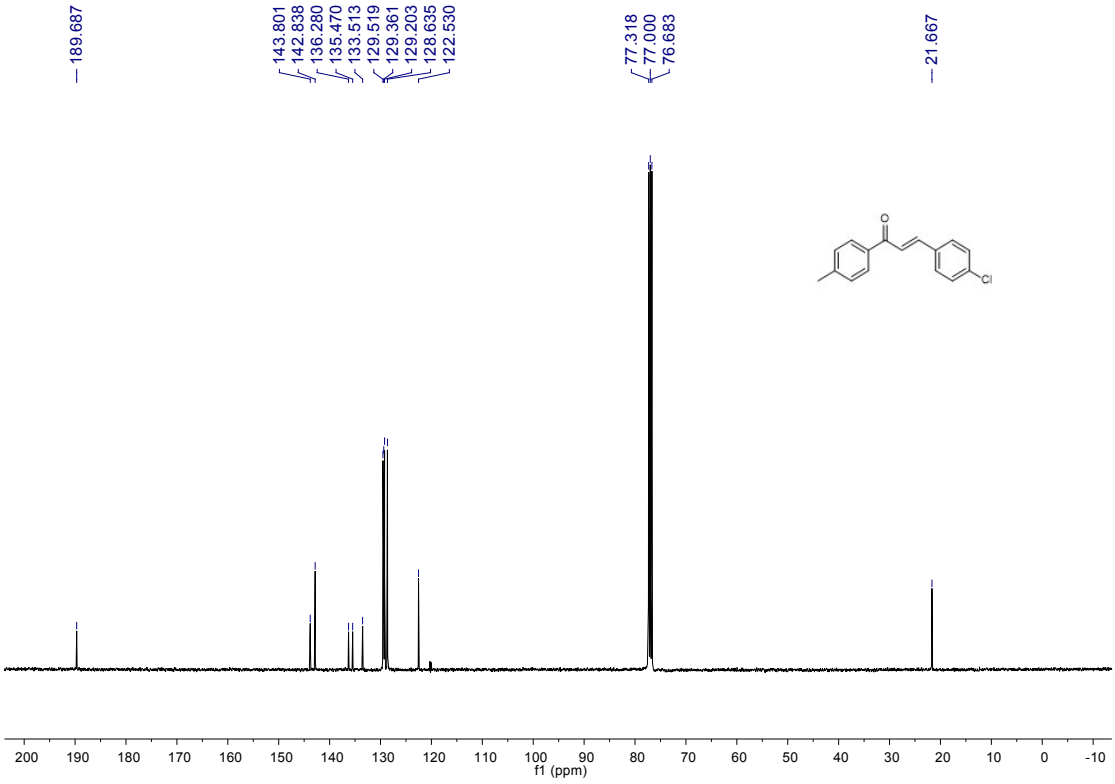
^{13}C NMR Spectrum of (*E*)-1,3-di-p-tolylprop-2-en-1-one **(2s)**



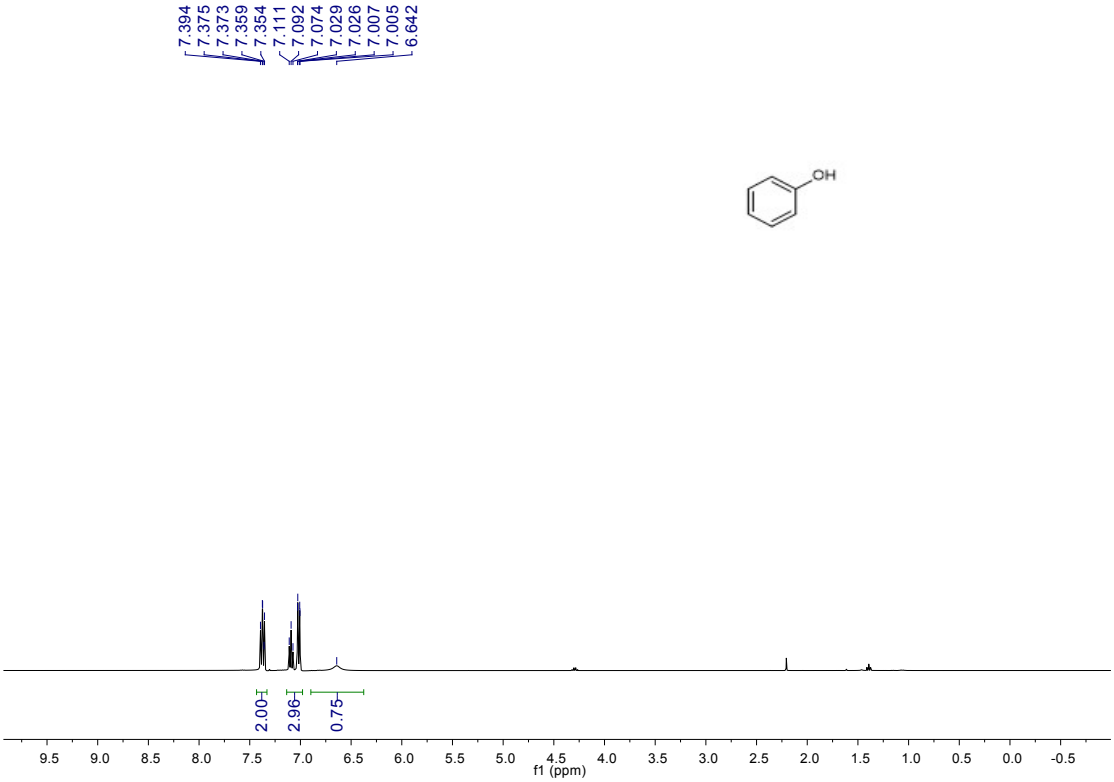
^{1}H NMR Spectrum of (*E*)-3-(4-chlorophenyl)-1-(p-tolyl)prop-2-en-1-one **(2t)**



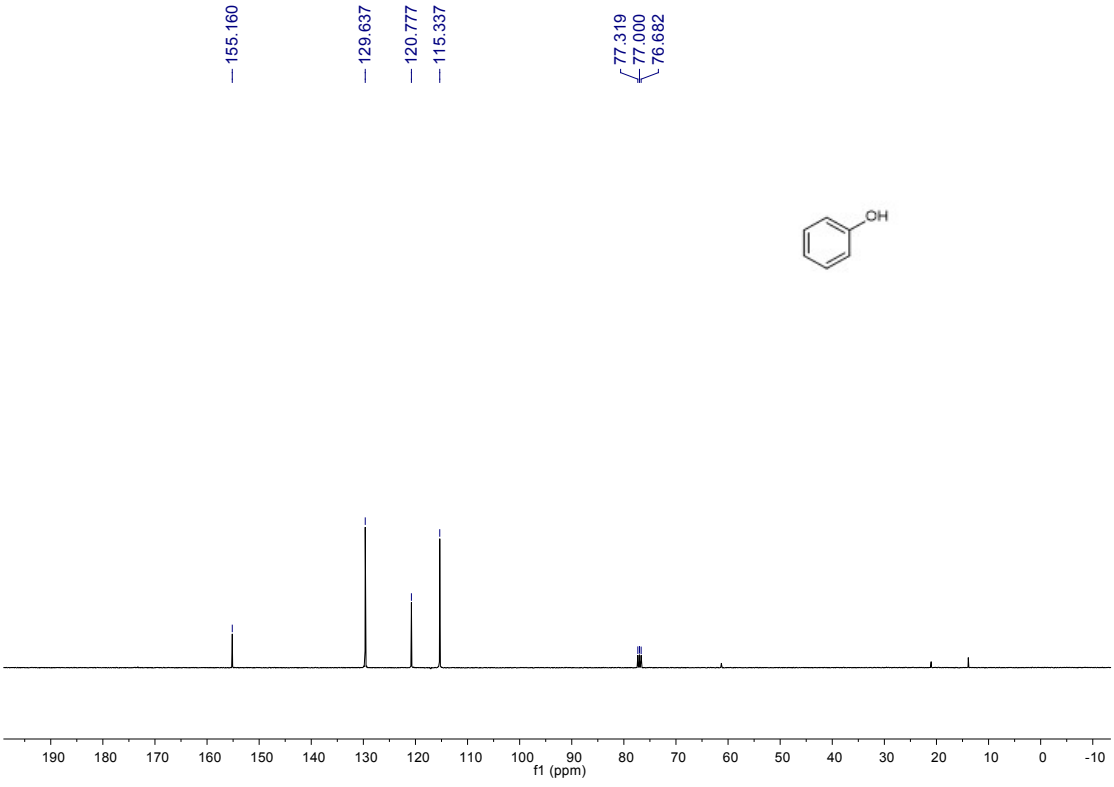
^{13}C NMR Spectrum of (*E*)-3-(4-chlorophenyl)-1-(p-tolyl)prop-2-en-1-one **(2t)**



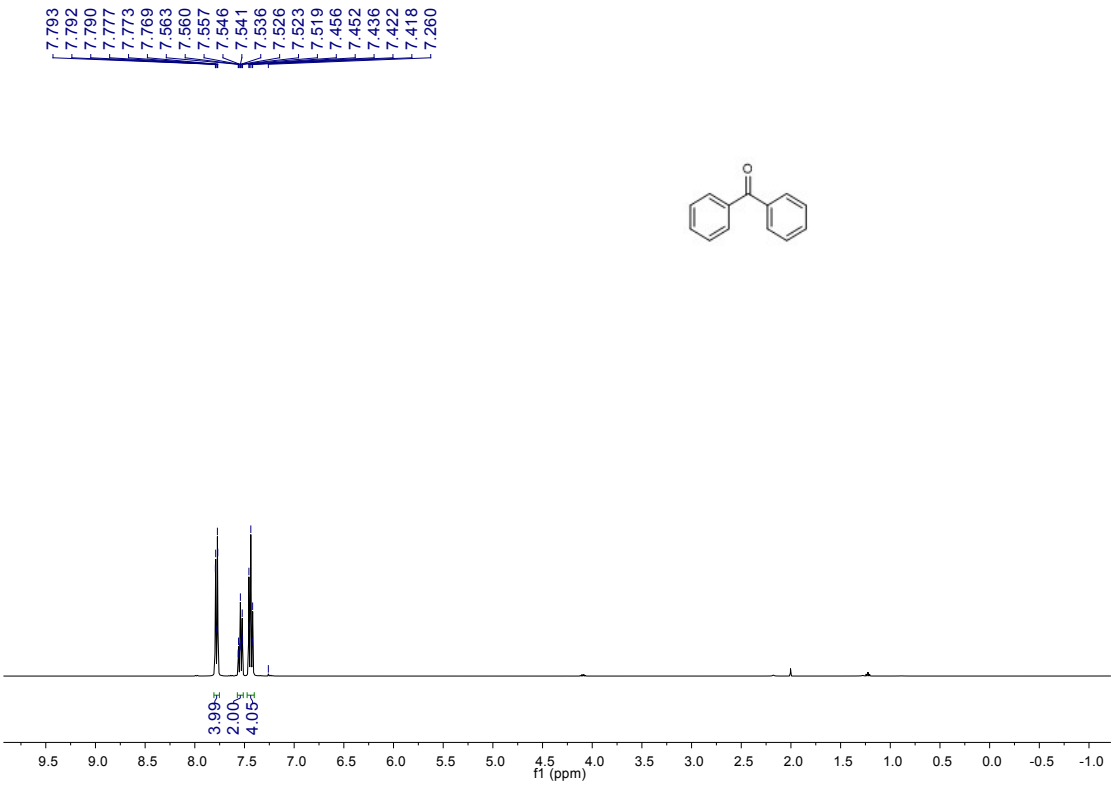
^{1}H NMR Spectrum of phenol **(2u)**



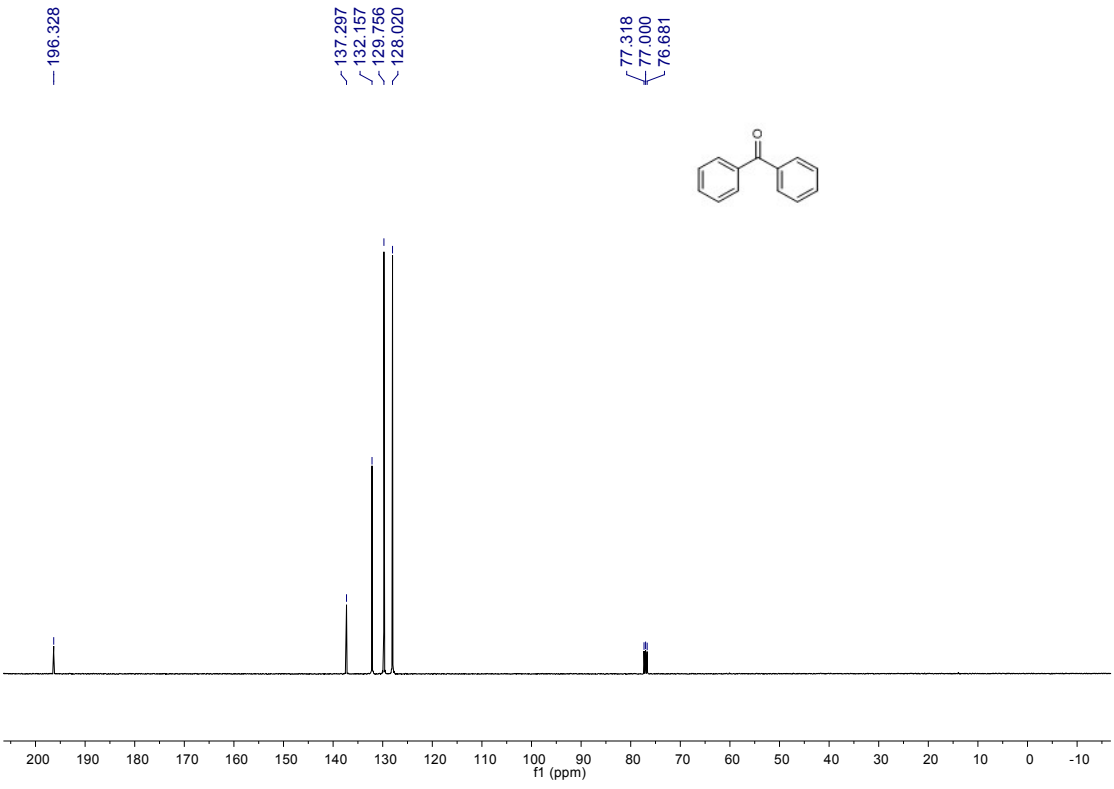
^{13}C NMR Spectrum of phenol **(2u)**



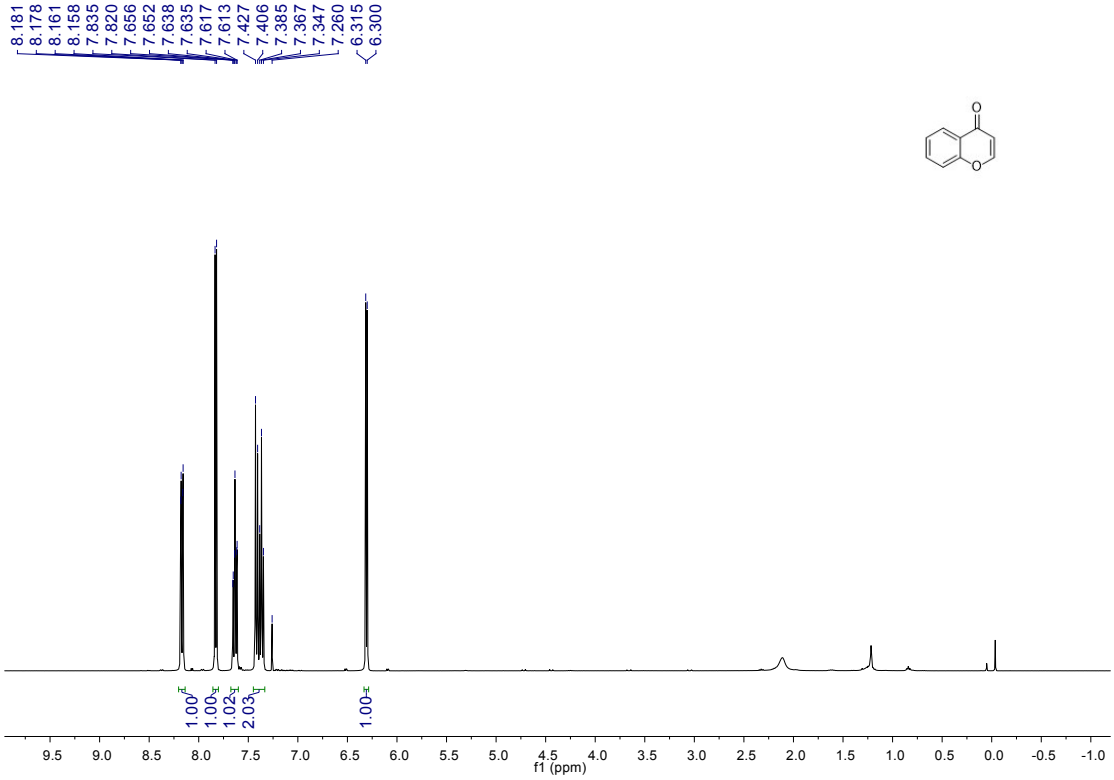
^{1}H NMR Spectrum of benzophenone **(2v)**



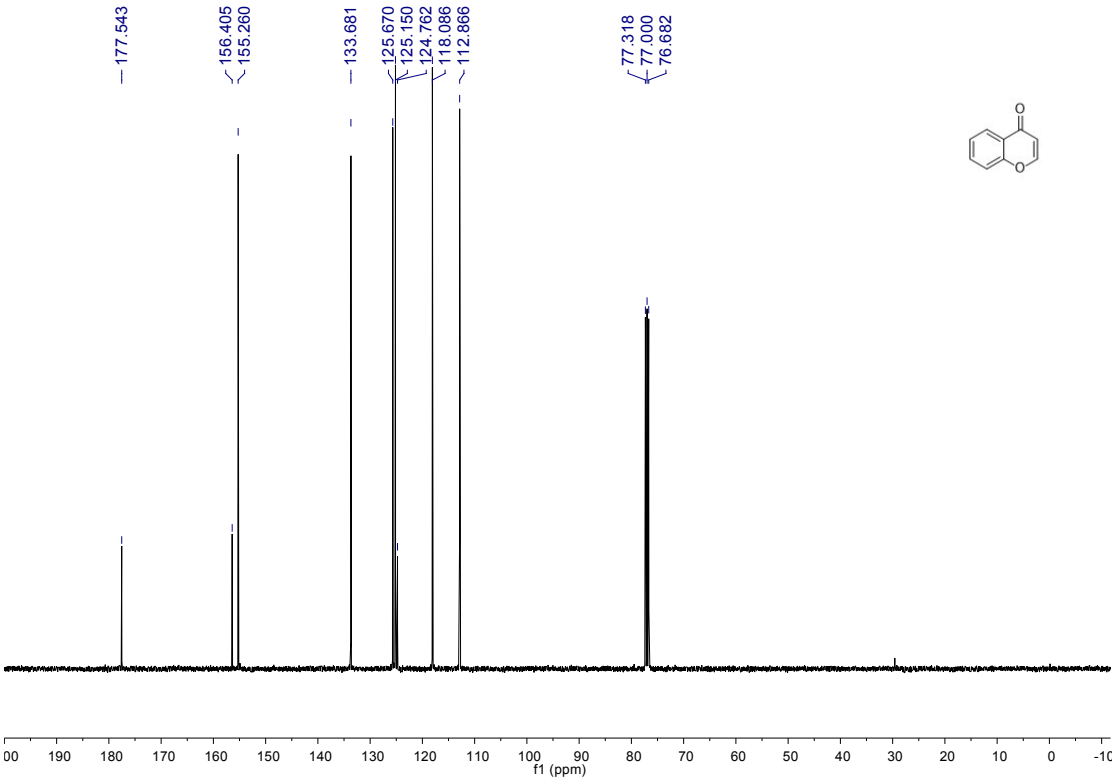
^{13}C NMR Spectrum of benzophenone **(2v)**



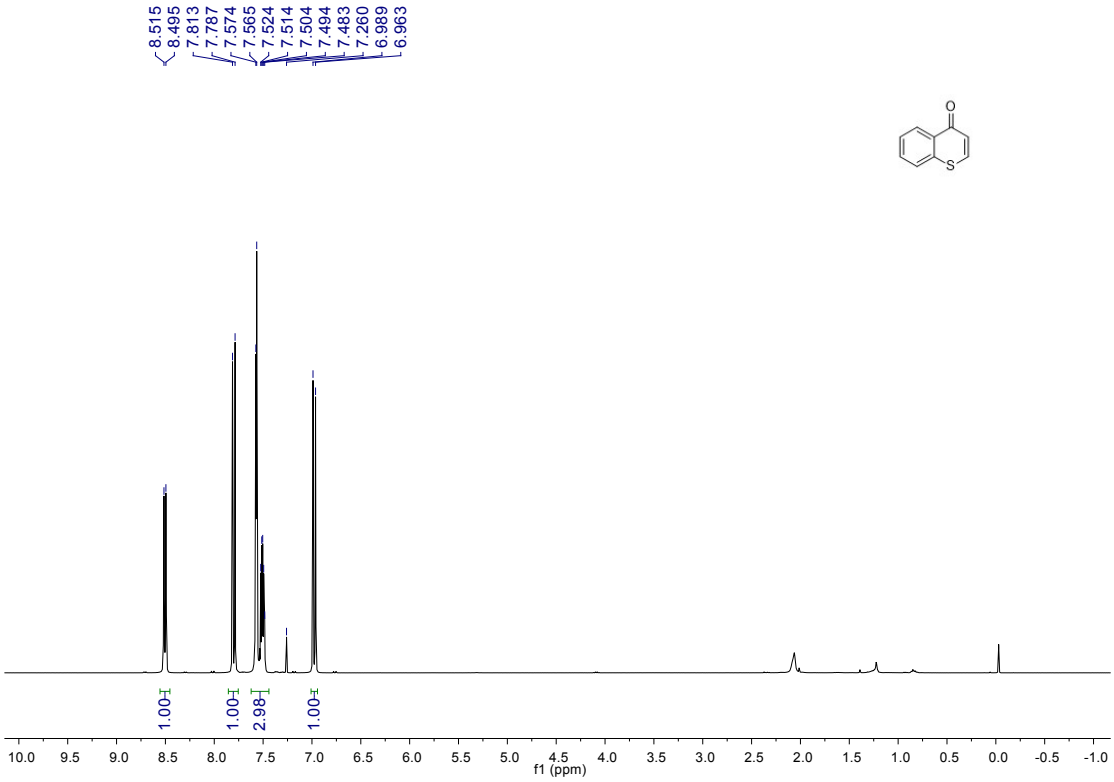
^{1}H NMR Spectrum of 4H-chromen-4-one **(2w)**



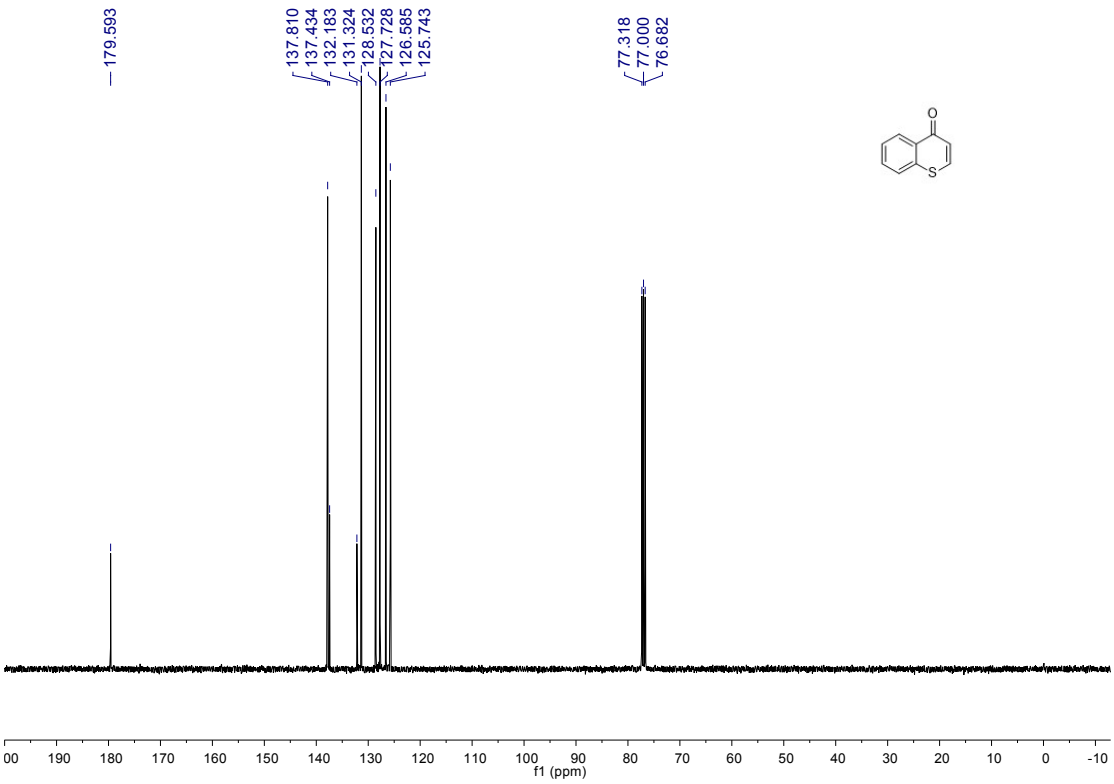
^{13}C NMR Spectrum of 4H-chromen-4-one **(2w)**



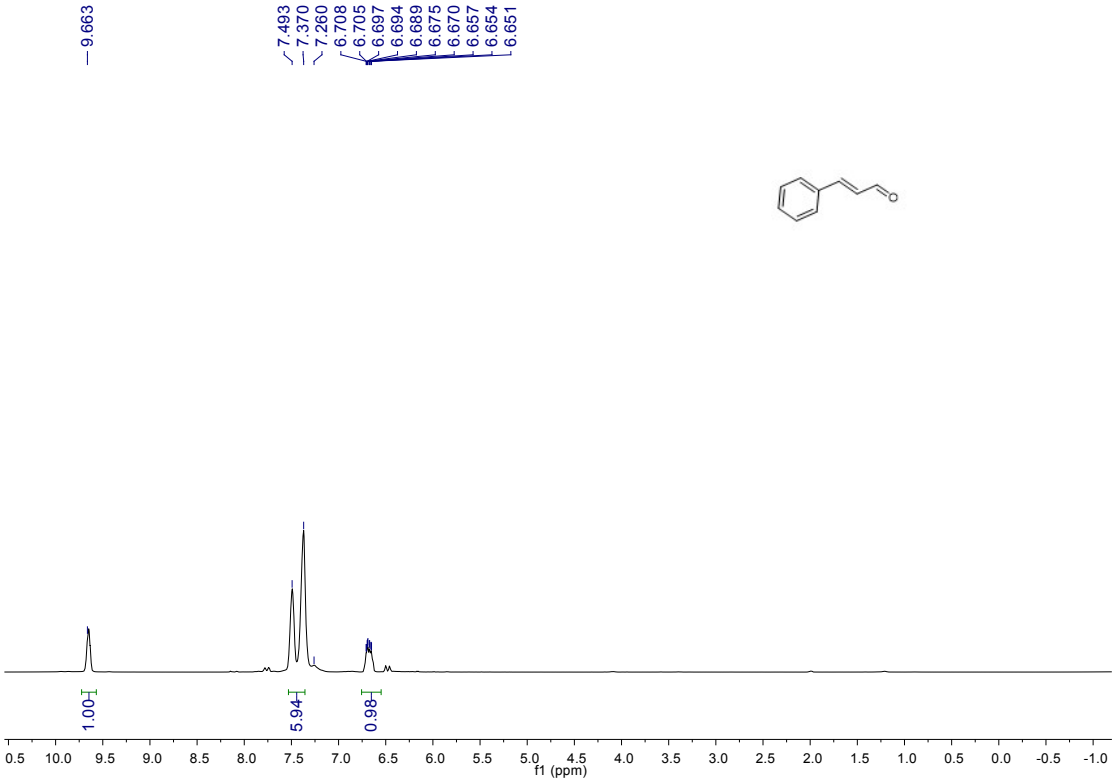
^{1}H NMR Spectrum of 4H-thiochromen-4-one **(2x)**



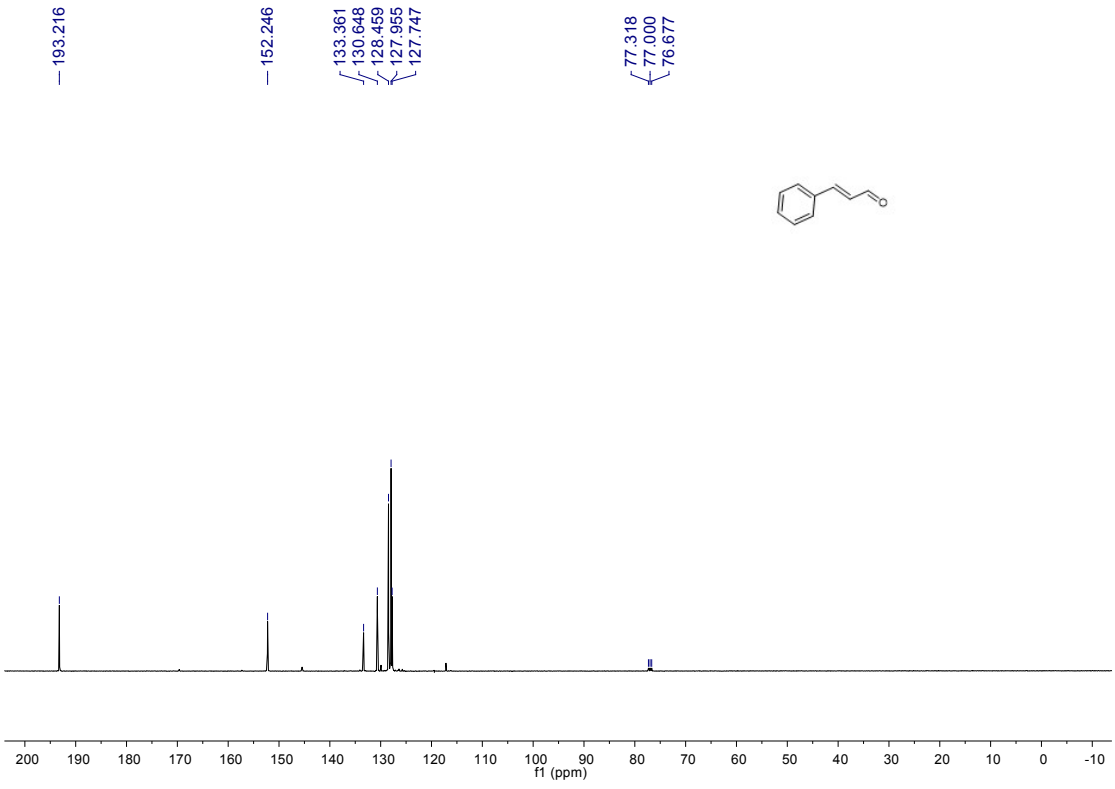
^{13}C NMR Spectrum of 4H-thiochromen-4-one **(2x)**



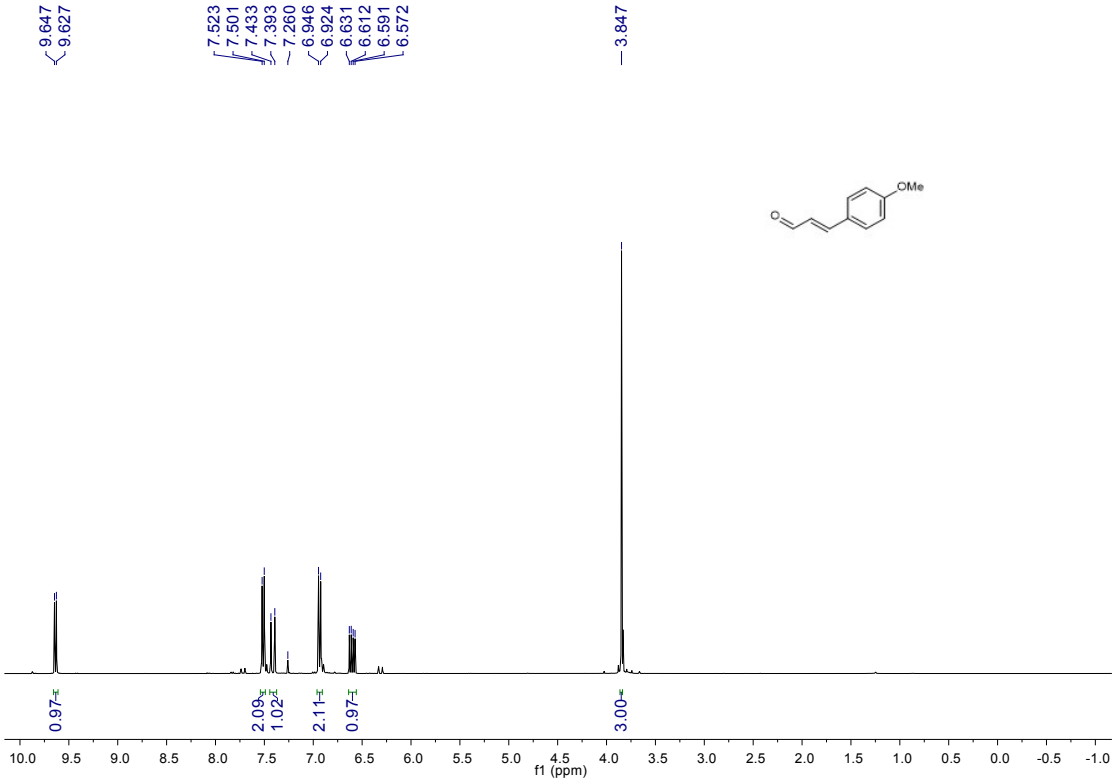
^{1}H NMR Spectrum of cinnamaldehyde **(2y)**



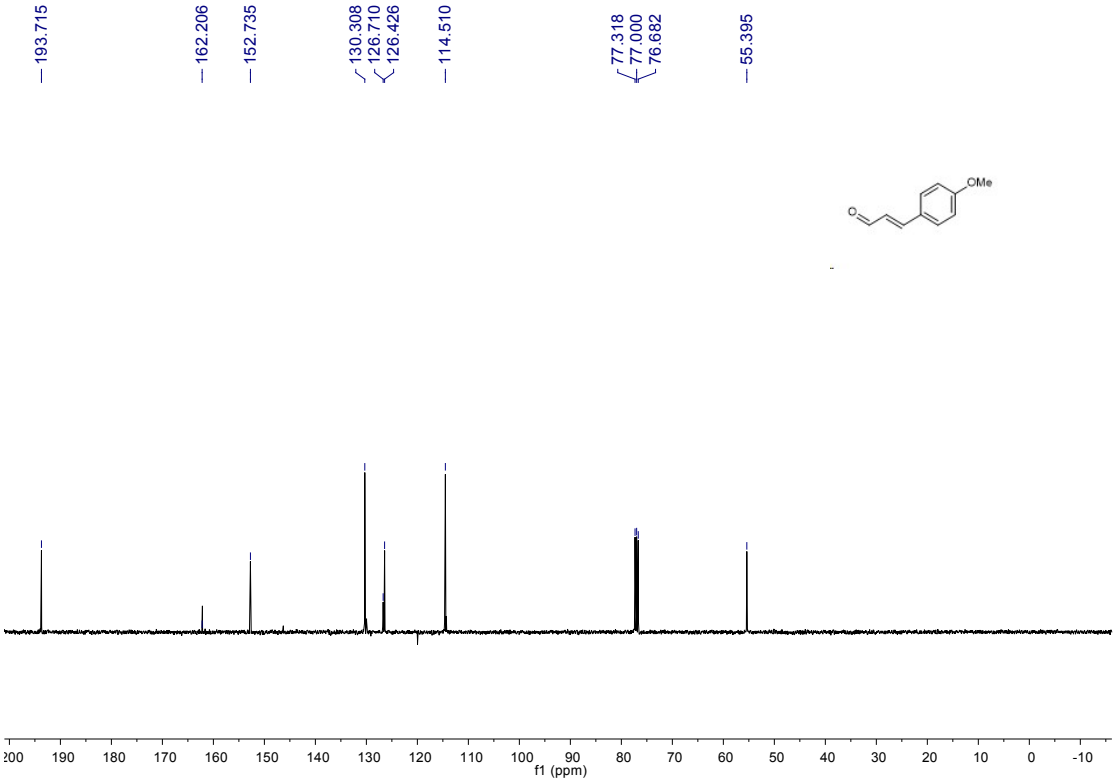
^{13}C NMR Spectrum of cinnamaldehyde **(2y)**



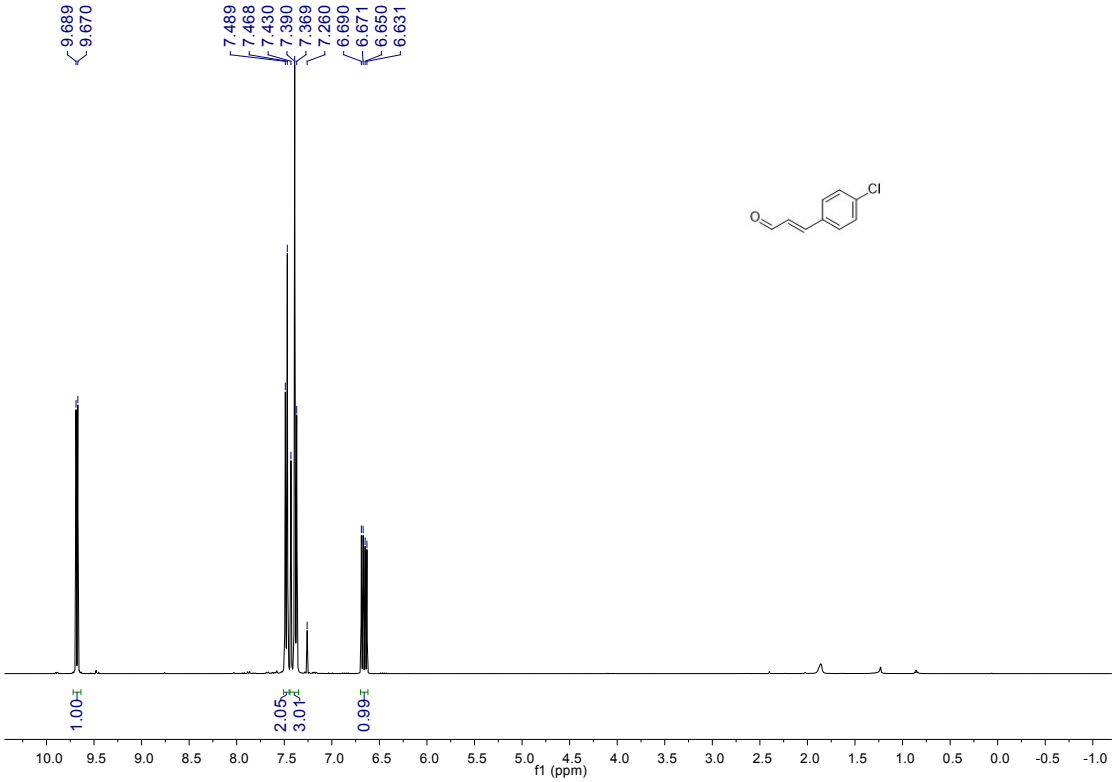
^{1}H NMR Spectrum of (*E*)-3-(4-methoxyphenyl)acrylaldehyde **(2z)**



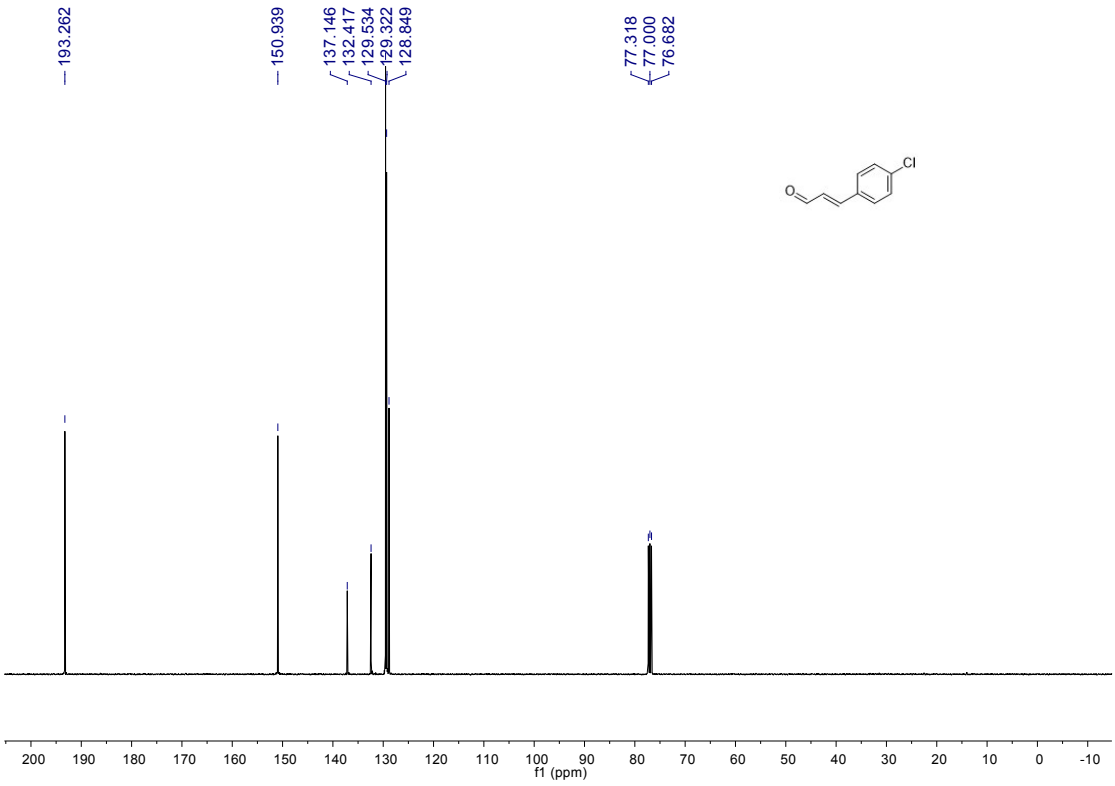
^{13}C NMR Spectrum of (*E*)-3-(4-methoxyphenyl)acrylaldehyde **(2z)**



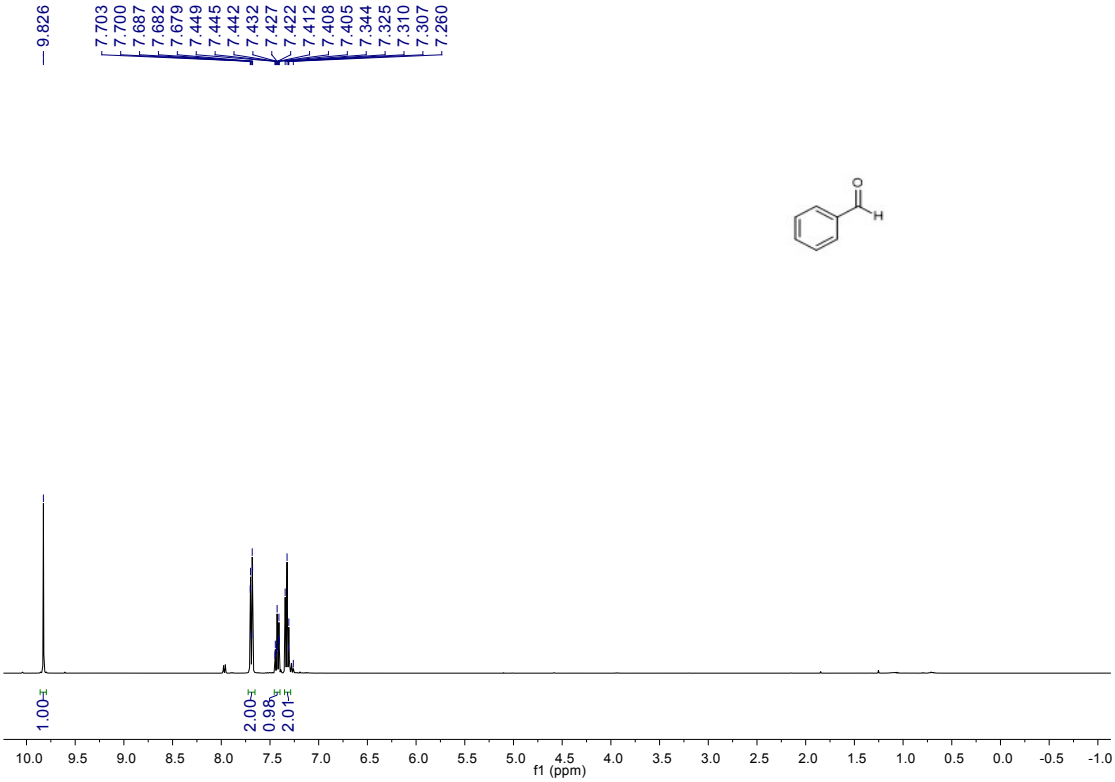
^{1}H NMR Spectrum of (*E*)-3-(4-chlorophenyl)acrylaldehyde **(2aa)**



^{13}C NMR Spectrum of (*E*)-3-(4-chlorophenyl)acrylaldehyde **(2aa)**



^{1}H NMR Spectrum of benzaldehyde **(2ab)**



^{13}C NMR Spectrum of benzaldehyde **(2ab)**