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Supporting Information

Trans-Selective Semireduction of Activated Alkynes via Phosphine Redox Catalysis

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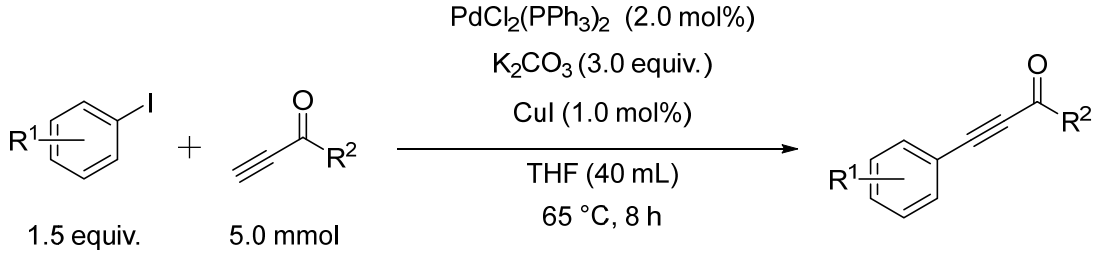
General Remarks

All reactions were conducted in oven-dried glassware under nitrogen atmosphere. Unless otherwise noted, chemical reagents were purchased from commercial suppliers and used without further purification. 1,4-dioxane was distilled from sodium and benzophenone and stored under nitrogen atmosphere. Analytical thin layer chromatography (TLC) was performed on silica gel plates (300-400 mesh) and visualized with a UV light (254 or 366 nm) or fresh $KMnO_4$ solution. The column chromatography was performed using silica gel (300-400 mesh) or performed on the CombiFlash NextGen 300 flash chromatography system. NMR spectra were recorded on a Bruker AVANCE III HD 400 (^1H 400 MHz, ^{13}C 100 MHz), AVANCE III HD 600 (^1H 600 MHz, ^{13}C 150 MHz), JEOL JNM-ECZ400S or JEOL JNM-ECZ600R spectrometers, using $CDCl_3$ as solvent at ambient temperature. The chemical shifts are reported in parts per million (ppm) and calibrated to tetramethylsilane (0 ppm) or the residual ^1H and ^{13}C signals of the solvents ($CDCl_3$: 7.26 ppm for ^1H NMR and 77.16 ppm for ^{13}C NMR). Peak multiplicities are abbreviated as follows: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, td = triplet of doublets, m = multiplet, br = broad. High-Resolution Mass Spectrometry (HRMS) analyses were performed by the instrumentation center at Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, on an Impact II Q-TOF mass spectrometry equipped with an ESI source from Bruker.

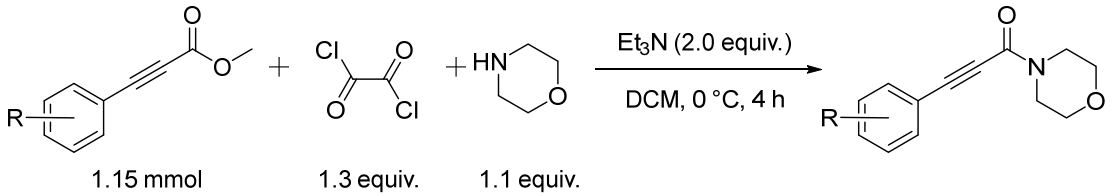
Syntheses of Starting Materials

General Procedure A



The corresponding iodobenzene (7.5 mmol, 1.5 equiv.), alkyne (5.0 mmol, 1.0 equiv.), and K_2CO_3 (15 mmol, 3.0 equiv.) were dissolved in THF (40 mL). To this solution was added $PdCl_2(PPh_3)_2$ (0.10 mmol, 2.0 mol%). After the mixture was stirred for 5 min, CuI (0.05 mmol, 1 mol%) was added. The resulting mixture was heated at 65 °C under a nitrogen atmosphere for 8 h. The reaction mixture was cooled to room temperature, and the solid was removed by filtration. The filtrate was concentrated under reduced pressure. The residue was then purified by silica gel column chromatography using a mixture of petroleum ether and ethyl acetate as the eluent.[1]

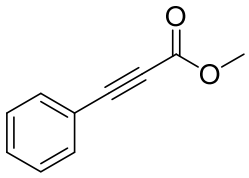
General Procedure B



The corresponding alkynoate (1.15 mmol, 2.0 equiv.) was dissolved in the corresponding alcohol (10 mL), and 1 M aqueous NaOH solution (5.0 mL) was added. The mixture was stirred at room temperature for 3 h. The reaction mixture was acidified with 1 M hydrochloric acid (10 mL) and extracted with dichloromethane (4 × 25 mL). The combined organic phases were washed with brine (10.0 mL), dried over anhydrous sodium sulfate, and the solvent was evaporated under reduced pressure. The resulting residue was used directly without further purification. The residue was suspended in dichloromethane (50 mL), and triethylamine (1.27 mL, 2.3

mmol, 2.0 equiv.) was added. The solution was cooled to 0 °C, and oxalyl chloride (128 μL, 1.49 mmol, 1.3 equiv.) was added dropwise, followed by morpholine (109 μL, 1.26 mmol, 1.1 equiv.). The resulting solution was stirred at room temperature for 4 h. The reaction mixture was extracted successively with saturated aqueous ammonium chloride solution (2 × 10 mL) and brine (10 mL). The organic phase was dried over sodium sulfate, and the solvent was removed under reduced pressure to obtain the title compounds.[2]

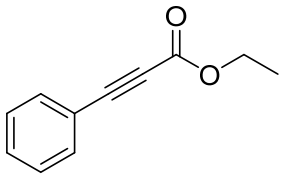
Methyl 3-phenylpropiolate (1a)



According to General Procedure A, **1a** was prepared from iodobenzene (0.840 mL, 7.50 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.00 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the title compound was obtained as colorless oil. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60-7.57 (m, 2H), 7.50-7.35 (m, 3H), 3.86 (s, 3H).

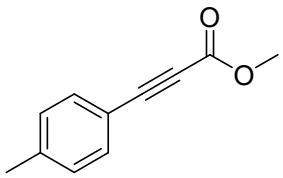
Ethyl 3-phenylpropiolate (1b)



According to General Procedure A, **1b** was prepared from iodobenzene (0.84 mL, 7.5 mmol, 1.5 equiv.) and ethyl propiolate (0.51 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the title compound was obtained as yellow oil. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60-7.52 (m, 2H), 7.50-7.35 (m, 3H), 4.30 (q, J = 7.0 Hz, 2H), 1.37 (t, J = 7.0 Hz, 3H).

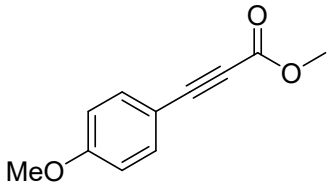
Methyl 3-(p-tolyl) propiolate (1d)



According to General Procedure A, **1d** was prepared from 4-iodotoluene (1.64 g, 7.5 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 9:1), the title compound was obtained as white solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50 (d, *J* = 9.0 Hz, 2H), 7.18 (d, *J* = 9.0 Hz, 2H), 3.85 (s, 3H), 2.37 (s, 3H).

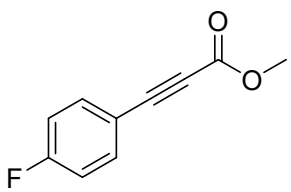
Methyl 3-(4-methoxyphenyl) propiolate (1e)



According to General Procedure A, **1e** was prepared from 1-bromo-3-iodobenzene (1.76 mg, 7.5 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 9:1), the title compound was obtained as yellow solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (d, *J* = 8.6 Hz, 2H), 6.88 (d, *J* = 8.7 Hz, 2H), 3.84 (s, 3H), 3.82 (s, 3H).

Methyl 3-(4-fluorophenyl) propiolate (1f)

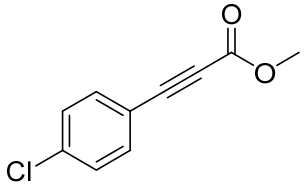


According to General Procedure A, **1f** was prepared from 1-fluoro-4-iodobenzene

(0.86 mL, 7.5 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the title compound was obtained as white solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64-7.53 (m, 2H), 7.11-7.03 (m, 2H), 3.82 (s, 3H).

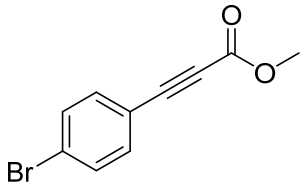
Methyl 3-(4-chlorophenyl) propiolate (1g)



According to General Procedure A, **1g** was prepared from 1-chloro-4-iodobenzene (1.78 g, 7.5 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 9:1), the title compound was obtained as white solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55-7.51 (m, 2H), 7.38-7.33 (m, 2H), 3.86 (s, 3H).

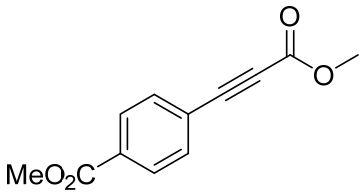
Methyl 3-(4-bromophenyl) propiolate (1h)



According to General Procedure A, **1h** was prepared from 1-bromo-4-iodobenzene (2.12 g, 7.5 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 9:1), the title compound was obtained as yellow solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53-7.50 (m, 2H), 7.44-7.42 (m, 2H), 3.86 (s, 3H).

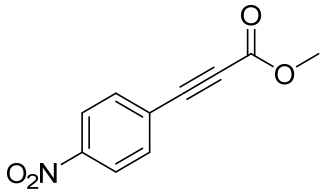
Methyl 4-(3-methoxy-3-oxoprop-1-yn-1-yl) benzoate (1i)



According to General Procedure A, **1i** was prepared from 4-iodobenzoate (1.96 g, 7.5 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 6:4), the title compound was obtained as yellow solid. The NMR data are in agreement with those reported in the literature.[3]

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10-7.98 (m, 2H), 7.63 (d, *J* = 8.4 Hz, 2H), 3.94 (s, 3H), 3.86 (s, 3H).

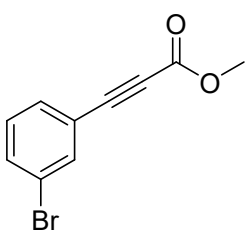
Methyl 3-(4-nitrophenyl) propiolate (1j)



According to General Procedure A, **1j** was prepared from 1-iodo-4-nitrobenzene (1.87 mg, 7.5 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 9:1), the title compound was obtained as yellow solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25 (d, *J* = 9.0 Hz, 2H), 7.76 (d, *J* = 9.1 Hz, 2H), 3.89 (s, 3H).

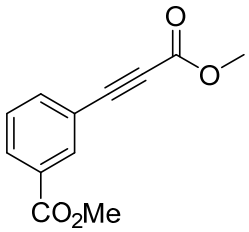
Methyl 3-(3-bromophenyl) propiolate (1k)



According to General Procedure A, **1k** was prepared from 1-bromo-3-iodobenzene (0.96 mL, 7.5 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 9:1), the title compound was obtained as white solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75-7.72 (m, 1H), 7.62-7.55 (m, 1H), 7.53-7.49 (m, 1H), 7.28-7.25 (m, 1H), 3.86 (s, 3H).

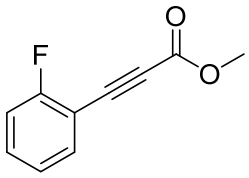
Methyl 3-(3-methoxy-3-oxoprop-1-yn-1-yl) benzoate (1l)



According to General Procedure A, **1l** was prepared from methyl 3-iodobenzoate (1.96 g, 7.5 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 7:3), the title compound was obtained as colorless oil. The NMR data are in agreement with those reported in the literature.[4]

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.20 (m, 1H), 8.05 (d, 1H, *J* = 8.0 Hz), 7.67 (d, 1H, *J* = 7.7 Hz), 7.40 (t, 1H, *J* = 7.6 Hz), 3.86 (s, 3H), 3.80 (s, 3H).

Methyl 3-(2-fluorophenyl)propiolate (1m)

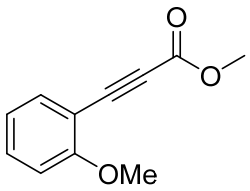


According to General Procedure A, **1m** was prepared from 1-fluoro-2-iodobenzene (0.87 mL, 7.5 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the title compound was obtained as

white solid. The NMR data are in agreement with those reported in the literature.[5]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56-7.49 (m, 1H), 7.45-7.49 (m, 1H), 7.19-7.05 (m, 2H), 3.81 (s, 3H).

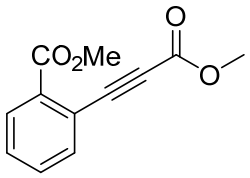
Methyl 3-(2-methoxyphenyl) propiolate (1n)



According to General Procedure A, **1n** was prepared from 2-iodoanisole (0.98 mL, 7.5 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 9:1), the title compound was obtained as yellow solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (dd, *J* = 7.5, 1.8 Hz, 1H), 7.40-7.33 (m, 1H), 6.92-6.83 (m, 2H), 3.83 (s, 3H), 3.80 (s, 3H).

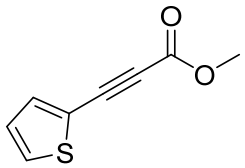
Methyl [2-(methylcarboxy)phenyl]-propynoate (1o)



According to General Procedure A, **1o** was prepared from methyl 2-iodobenzoate (1.96 g, 7.5 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 7:3), the title compound was obtained as yellow oil. The NMR data are in agreement with those reported in the literature.[6]

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03-8.06 (m, 1H), 7.69-7.72 (m, 1H), 7.50-7.58 (m, 2H), 3.97 (s, 3H), 3.95 (s, 3H).

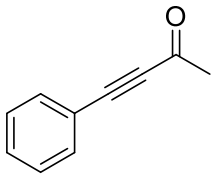
Methyl 3-(thiophen-2-yl) propiolate (1p)



According to General Procedure A, **1p** was prepared from 2-iodothiophene (0.98 mL, 7.5 mmol, 1.5 equiv.) and methyl propiolate (0.44 mL, 5.0 mmol, 1.0 equiv.). After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 9:1), the title compound was obtained as colorless oil. The NMR data are in agreement with those reported in the literature.[5]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51-7.44 (m, 2H), 7.04 (dd, *J* = 5.1, 3.9 Hz, 1H), 3.82 (s, 3H).

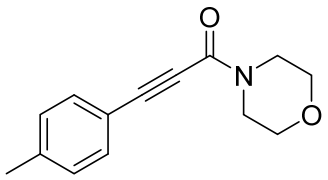
4-Phenyl-3-butyne-2-one (1r)



CuI (48 mg, 0.05 equiv.) was added to dry THF (0.30 M) in a flask under nitrogen. Et_3N (0.69 mL, 2.0 equiv.) was added dropwise, followed by the alkyne (0.55 mL, 1.0 equiv.) and acid chloride (1.06 mL, 2.0 equiv.). The reaction mixture was stirred at room temperature overnight (18 h). The solution was diluted with Et_2O and washed with water followed by separation and extraction twice. The organic layers were combined and dried over $MgSO_4$. The mixture was filtered. The solvents were removed under vacuum. The crude product was purified by flash chromatography (petroleum ether/ethyl acetate = 50:1) to provide the product. The product was obtained as a yellow oil. The NMR data are in agreement with those reported in the literature.[7]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 (ddd, *J* = 6.9, 2H), 7.50-7.42 (m, 1H), 7.40-7.37 (m, 2H), 2.46 (s, 3H).

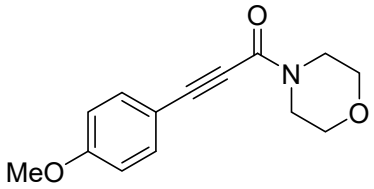
1-Morpholino-3-(p-tolyl)prop-2-yn-1-one (1ad)



According to General Procedure B, **1ad** was prepared from **1d**. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 6:4), the title compound was obtained as yellow solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (d, *J* = 8.1 Hz, 2H), 7.17 (d, *J* = 7.9 Hz, 2H), 3.87-3.82 (m, 2H), 3.76-3.75 (m, 2H), 3.70 (s, 4H), 2.37 (s, 3H).

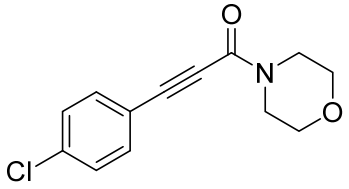
3-(4-Methoxyphenyl)-1-morpholinoprop-2-yn-1-one (1ae)



According to General Procedure B, **1ae** was prepared from **1e**. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 6:4), the title compound was obtained as yellow solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51-7.49 (m, 2H), 6.91-6.87 (m, 2H), 3.85-3.83 (m, 5H), 3.75-3.73 (m, 2H), 3.71 (s, 4H).

2-(4-Chlorophenyl)-1-morpholinoprop-2-yn-1-one (1af)

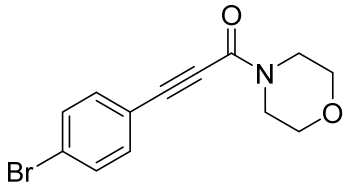


According to General Procedure B, **1af** was prepared from **1g**. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl

acetate = 7:3), the title compound was obtained as yellow solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50-7.47 (m, 2H), 7.38-7.34 (m, 2H), 3.83-3.81 (m, 2H), 3.79-3.72 (m, 2H), 3.70 (s, 4H).

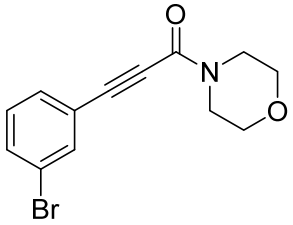
2-(4-Bromophenyl)-1-morpholinoprop-2-yn-1-one (1ag)



According to General Procedure B, **1ag** was prepared from **2h**. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 6:4), the title compound was obtained as yellow solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52-7.49 (m, 2H), 7.41-7.39 (m, 2H), 3.82-3.81 (m, 2H), 3.74-3.70 (m, 2H), 3.71 (s, 4H).

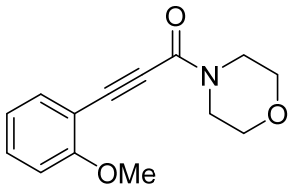
3-(3-Bromophenyl)-1-morpholinoprop-2-yn-1-one (1ah)



According to General Procedure B, **1ah** was prepared from **1k**. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 8:2), the title compound was obtained as yellow solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (t, J = 1.8 Hz, 1H), 7.57-7.55 (m, 1H), 7.50 (dt, J = 7.8, J = 1.2 Hz, 1H), 7.26 (t, J = 7.9 Hz, 1H), 3.85-3.81 (m, 2H), 3.77-3.75 (m, 2H), 3.70 (s, 4H).

3-(2-Methoxyphenyl)-1-morpholinoprop-2-yn-1-one (1ai)

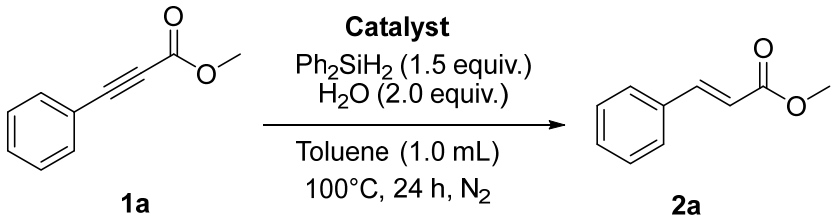


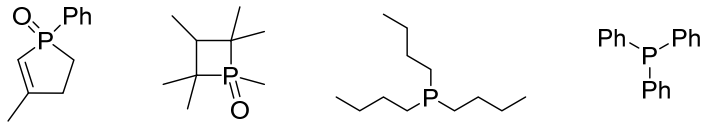
According to General Procedure B, **1ai** was prepared from **1n**. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 6:4), the title compound was obtained as yellow solid. The NMR data are in agreement with those reported in the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50 (dd, *J* = 7.6, 1.8 Hz, 1H), 7.40-7.37 (m, 1H), 7.96-7.89 (m, 2H), 3.93-3.91 (m, 2H), 3.89 (s, 3H), 3.78-3.74 (m, 2H), 3.70 (s, 4H).

Optimization of Reaction Conditions

Effect of catalyst loading

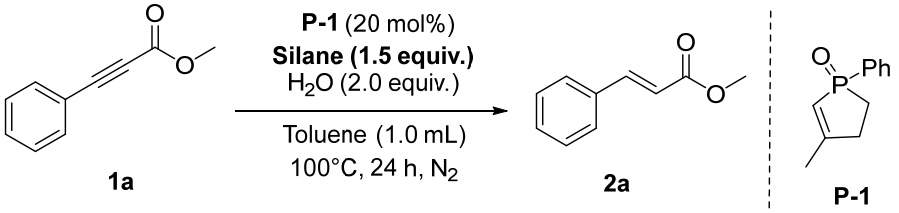




Entry[a]	Catalyst	Yield of 2a (%)[b]
1	**P-1** (15 mol%)	55
2	**P-1** (10 mol%)	51
3	**P-1** (5 mol%)	45
4	**P-2** (20 mol%)	<5
5	**P-3** (20 mol%)	36
6	**P-4** (20 mol%)	17

[a]Reaction conditions: methyl 3-phenylpropiolate **1a** (0.2 mmol), Ph_2SiH_2 (1.5 equiv.), H_2O (2.0 equiv.), toluene (1.0 mL), N_2, 100 °C, 24 h. [b]Yields were determined by GC analysis of the crude reaction mixture using *n*-dodecane as an internal standard.

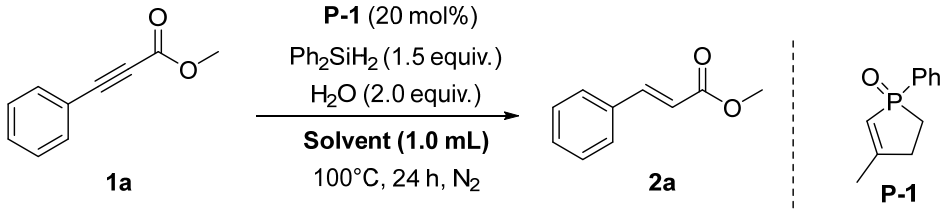
Screening of silane



Entry[a]	Silane	Yield of 2a (%)[b]
1	PMHS	<5
2	Et_3SiH	N.D.
3	$PhSiH_3$	73
4	Ph_2SiH_2	86

[a]Reaction conditions: methyl 3-phenylpropiolate **1a** (0.2 mmol), **P-1** (20 mmol%), H_2O (2.0 equiv.), toluene (1.0 mL), N_2, 100 °C, 24 h. [b]Yields were determined by GC analysis of the crude reaction mixture using *n*-dodecane as an internal standard. N.D. = not detected.

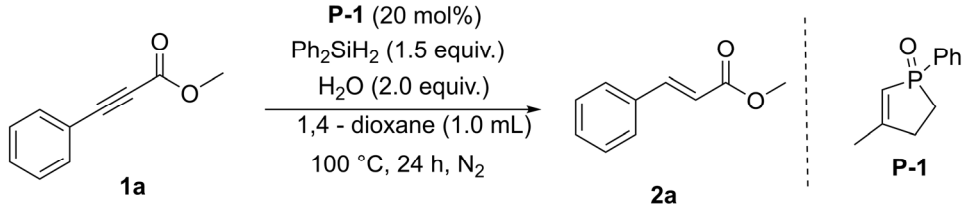
Screening of solvents



Entry[a]	Solvent (1.0 mL)	Yield of 2a (%)[b]
1	Toluene	86
2	MeCN	38
3	DCE	33
4	1,4-dioxane	98[c]

[a]Reaction conditions: methyl 3-phenylpropiolate **1a** (0.2 mmol), **P-1** (20 mmol%), Ph_2SiH_2 (1.5 equiv.), H_2O (2.0 equiv.), N_2, 100 °C, 24 h. [b]Yields were determined by GC analysis of the crude reaction mixture using *n*-dodecane as an internal standard. [c]Isolated yield.

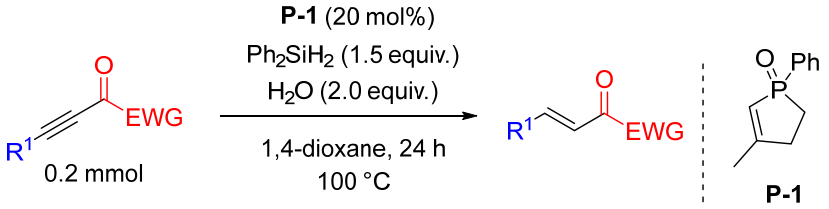
Further screening experiments



Entry[a]	Deviations from standard conditions	Yield of 2a (%)[b]
1	**P-1** (15 mol%)	62
2	reaction conducted at 80 °C	44
3	w/o **P-1**	N.D.
4	w/o Ph_2SiH_2	N.D.
5	w/o water	28

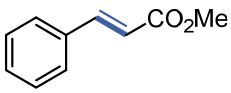
[a]Reaction conditions: methyl 3-phenylpropiolate **1a** (0.2 mmol), **P-1** (20 mmol%), Ph_2SiH_2 (1.5 equiv.), H_2O (2.0 equiv.), 1,4-dioxane (1.0 mL), N_2, 100 °C, 24 h. [b]Yields were determined by GC analysis of the crude reaction mixture using *n*-dodecane as an internal standard. w/o = without. N.D. = not detected.

General Procedure and Product Characterization



In the glovebox, the alkynoate (0.2 mmol, 1.0 equiv.) was dissolved in 1,4-dioxane (1.0 mL). The tube was moved out of the glovebox after being sealed with a rubber septum and then H_2O (7.2 μL, 0.4 mmol, 2.00 equiv.) and Ph_2SiH_2 (56 μL, 0.3 mmol, 1.5 equiv.) were added sequentially, followed by **P-1** (7.6 mg, 0.04 mmol, 20 mol%). The resulting solution was stirred at 100 °C for 24 h. The solvent was removed under reduced pressure, and the residue was purified by flash column chromatography to afford the desired product.

Methyl (*E*)-3-phenylacrylate (2a)

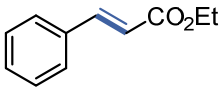


According to General Procedure, the title compound was synthesized from **1a** (29 μL, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2a** was obtained as a white solid (31.7 mg, 98% yield). The NMR data are in agreement with those reported in the literature.[8]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, J = 16.0 Hz, 1H), 7.56-7.49 (m, 2H), 7.39 (m, 3H), 6.45 (d, J = 16.0 Hz, 1H), 3.80 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.5, 144.9, 134.4, 130.3, 128.9, 128.1, 117.8, 51.7.

Ethyl (*E*)-3-phenylacrylate (2b)



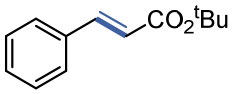
According to General Procedure, the title compound was synthesized from **1b** (35.2 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and

purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2b** was obtained as colorless oil (31.1 mg, 88% yield). The NMR data are in agreement with those reported in the literature.[8]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, *J* = 16.0 Hz, 1H), 7.53 (m, 2H), 7.38 (m, 3H), 6.44 (d, *J* = 16.0 Hz, 1H), 4.27 (q, *J* = 7.1 Hz, 2H), 1.34 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.0, 144.6, 134.5, 130.2, 128.9, 128.1, 118.3, 60.5, 14.4.

Tert-butyl cinnamate (2c)

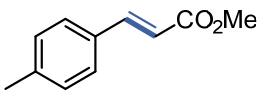


According to General Procedure, the title compound was synthesized from tert-butyl 3-phenylpropiolate[1] (39.0 μL, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 99:1), the product **2c** was obtained as colorless oil (39.9 mg, 98% yield). The NMR data are in agreement with those reported in the literature.[9]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 (d, *J* = 16.0 Hz, 1H), 7.52-7.48 (m, 2H), 7.36 (dd, *J* = 5.3, 1.9 Hz, 3H), 6.37 (d, *J* = 16.0 Hz, 1H), 1.54 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 166.4, 143.6, 134.7, 130.0, 128.8, 128.0, 120.2, 80.5, 28.2.

Methyl (*E*)-3-(*p*-tolyl)acrylate (2d)

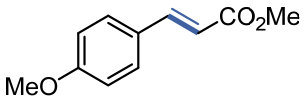


According to General Procedure, the title compound was synthesized from **1d** (34.8 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2d** was obtained as a white solid (25.7 mg, 73% yield). The NMR data are in agreement with those reported in the literature.[8]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (d, *J* = 16.0 Hz, 1H), 7.42 (d, *J* = 7.8 Hz, 2H), 7.19 (m, 2H), 6.40 (d, *J* = 16.0 Hz, 1H), 3.80 (s, 3H), 2.37 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.7, 144.9, 140.8, 131.7，129.6, 128.1, 116.7, 51.7, 21.5.

Methyl (*E*)-3-(4-methoxyphenyl)acrylate (2e)

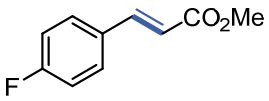


According to General Procedure, the title compound was synthesized from **1e** (38.0 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2e** was obtained as a white solid (23.8 mg, 62% yield). The NMR data are in agreement with those reported in the literature.[10]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (d, *J* = 16.0 Hz, 1H), 7.47 (d, *J* = 8.3 Hz, 2H), 6.89 (d, *J* = 8.3 Hz, 2H), 6.30 (d, *J* = 16.0 Hz, 1H), 3.83 (s, 3H), 3.78 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.9, 161.5, 144.7, 129.3, 127.2, 116.0, 114.4, 55.5, 51.1.

Methyl (*E*)-3-(4-fluorophenyl)acrylate (2f)



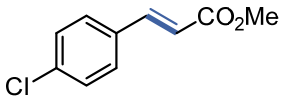
According to General Procedure, the title compound was synthesized from **1f** (35.6 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2f** was obtained as a white solid (27.4 mg, 76% yield). The NMR data are in agreement with those reported in the literature.[8]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (d, *J* = 16.0 Hz, 1H), 7.50 (dd, *J* = 8.5, 5.4 Hz, 2H), 7.06 (t, *J* = 8.5 Hz, 2H), 6.35 (d, *J* = 16.0 Hz, 1H), 3.79 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.3, 163.6 (d, J_{C-F} = 251.2 Hz), 143.6, 130.6 (d, J_{C-F} = 4.0 Hz), 130.0 (d, J_{C-F} = 9.7), 117.5 (d, J_{C-F} = 2.5 Hz), 116.1 (d, J_{C-F} = 21.8), 51.7.

^{19}F NMR (376 MHz, $CDCl_3$): δ –109.5.

Methyl (*E*)-3-(4-chlorophenyl)acrylate (2g)

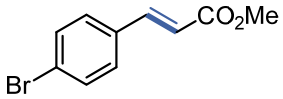


According to General Procedure, the title compound was synthesized from **1g** (38.9 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2g** was obtained as a yellow solid (27.9 mg, 71% yield). The NMR data are in agreement with those reported in the literature.[8]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, *J* = 16.0 Hz, 1H), 7.44 (d, *J* = 6.7 Hz, 2H), 7.35 (d, *J* = 6.7 Hz, 2H), 6.40 (d, *J* = 16.0 Hz, 1H), 3.80 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.2, 143.4, 136.2, 132.8, 129.2, 129.2, 118.4, 51.8.

Methyl (*E*)-3-(4-bromophenyl)acrylate (2h)

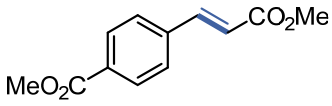


According to General Procedure, the title compound was synthesized from **1h** (47.8 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2h** was obtained as a white solid (38.5 mg, 80% yield). The NMR data are in agreement with those reported in the literature.[11]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 (d, *J* = 16.8 Hz, 1H), 7.52 (d, *J* = 6.8 Hz, 2H), 7.38 (d, *J* = 6.8 Hz, 2H), 6.43 (d, *J* = 16.8 Hz, 1H), 3.80 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.2, 143.5, 133.3, 132.2, 129.5, 124.6, 118.5, 51.8.

Methyl 4-(3-methoxy-3-oxoprop-1-yn-1-yl)benzoate (2i)

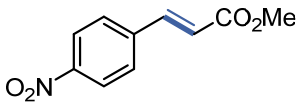


According to General Procedure, the title compound was synthesized from **1i** (43.6 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2i** was obtained as a yellow solid (31.7 mg, 72% yield). The NMR data are in agreement with those reported in the literature.[12]

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.03 (d, *J* = 8.4 Hz, 2H), 7.69 (d, *J* = 16.0 Hz, 1H), 7.56 (d, *J* = 8.4 Hz, 2H), 6.50 (d, *J* = 16.0 Hz, 1H), 3.91 (s, 3H), 3.80 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 167.1, 166.5, 143.5, 138.6, 131.5, 130.2, 128.0, 120.2, 52.4, 52.0.

Methyl (*E*)-3-(4-nitrophenyl)acrylate (2j)

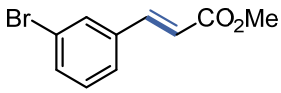


According to General Procedure, the title compound was synthesized from **1j** (41.0 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2j** was obtained as a yellow solid (21.5 mg, 52% yield). The NMR data are in agreement with those reported in the literature.[13]

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.24 (d, *J* = 8.4 Hz, 2H), 7.71 (d, *J* = 16.1 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 2H), 6.55 (d, *J* = 16.1 Hz, 1H), 3.83 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 166.5, 148.5, 141.9, 140.5, 128.7, 124.2, 122.1, 52.1.

Methyl (*E*)-3-(3-bromophenyl)acrylate (2k)



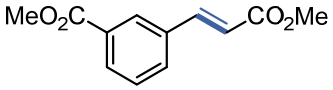
According to General Procedure, the title compound was synthesized from **1k** (47.8 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and

purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2k** was obtained as a white solid (20.7 mg, 43% yield). The NMR data are in agreement with those reported in the literature.[11]

^{1}H NMR (400 MHz, $CDCl_3$)： δ 7.66 (m, 1H), 7.63 (m, 1H), 7.51 (d, *J* = 7.9 Hz, 1H), 7.44 (d, *J* = 7.9 Hz, 1H), 7.26 (d, *J* = 16.0 Hz, 1H), 6.43 (d, *J* = 16.0 Hz, 1H), 3.81 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.0, 143.2, 136.5, 133.1, 130.8, 130.4, 126.7, 123.0, 119.3, 51.9.

Methyl (*E*)-3-(3-methoxy-3-oxoprop-1-en-1-yl)benzoate (2l)

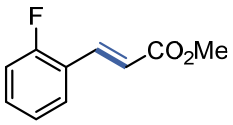


According to General Procedure, the title compound was synthesized from **1l** (43.6 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2l** was obtained as a yellow solid (43.6 mg, 99% yield). The NMR data are in agreement with those reported in the literature.[14]

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.18 (s, 1H), 8.02 (d, *J* = 7.8 Hz, 1H), 7.72-7.65 (m, 2H), 7.45 (t, *J* = 7.8 Hz, 1H), 6.50 (d, *J* = 16.0 Hz, 1H), 3.92 (s, 3H), 3.80 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 167.2, 166.6, 143.7, 134.8, 132.3, 131.2, 131.0, 129.1, 129.1, 119.2, 52.4, 51.9.

Methyl 3-(2-fluorophenyl)acrylate (2m)



According to General Procedure, the title compound was synthesized from **1m** (35.6 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2m** was obtained as a white solid (24.5 mg, 68% yield).

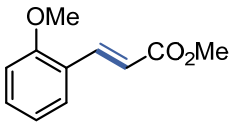
The NMR data are in agreement with those reported in the literature.[15]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, *J* = 16.2 Hz, 1H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.39-7.30 (m, 1H), 7.19-7.04 (m, 2H), 6.54 (d, *J* = 16.2 Hz, 1H), 3.81 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.3, 160.1 (d, J_{C-F} = 252.6 Hz), 137.56 (d, J_{C-F} = 2.85 Hz), 131.7 (d, J_{C-F} = 8.7 Hz), 129.1 (d, J_{C-F} = 2.7 Hz),124.4 (d, J_{C-F} = 11.9 Hz), 122.4 (d, J_{C-F} = 11.85 Hz), 120.3 (d, J_{C-F} = 6.6 Hz) 116.2 (d, J_{C-F} = 21.6 Hz), 51.8.

^{19}F NMR (376 MHz, $CDCl_3$) δ –113.98.

Methyl (*E*)-3-(2-methoxyphenyl)acrylate (2n)



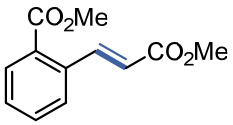
According to General Procedure, the title compound was synthesized from **1n** (33.1 μL, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2n** was obtained as a white solid (21.9 mg, 57% yield). The NMR data are in agreement with those reported in the literature.[8]

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (d, *J* = 16.1 Hz, 1H), 7.50 (d, *J* = 7.8 Hz, 1H), 7.35 (t, *J* = 7.8 Hz, 1H), 6.99-6.89 (m, 2H), 6.53 (d, *J* = 16.1 Hz, 1H), 3.88 (s, 3H), 3.80 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 168.0, 158.4, 140.3, 131.5, 128.9, 123.3, 120.7, 118.3, 111.1, 55.5, 51.6.

HRMS: Calculated for $C_{11}H_{12}NaO_3$ $[M+Na]^+$:215.0679, Found: 215.0680.

Methyl (*E*)-2-(3-methoxy-3-oxoprop-1-en-1-yl)benzoate (2o)



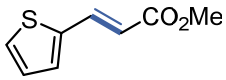
According to General Procedure, the title compound was synthesized from **1o** (35.6 μL, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and

purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2o** was obtained as colorless oil (18.1 mg, 41% yield). The NMR data are in agreement with those reported in the literature.[14]

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.45 (d, *J* = 15.9 Hz, 1H), 7.96 (d, *J* = 9.2 Hz, 1H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.56-7.51 (m, 1H), 7.47-7.42 (m, 1H), 6.30 (d, *J* = 15.9 Hz, 1H), 3.93 (s, 3H), 3.81 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.2, 167.0, 144.0, 136.4, 132.4, 130.8, 129.8, 129.4, 127.9, 120.7, 52.4, 51.8.

Methyl (*E*)-3-(thiophen-2-yl)acrylate (2p)

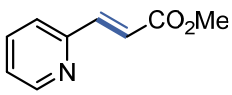


According to General Procedure, the title compound was synthesized from **1p** (26.6 μL, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2p** was obtained as a white solid (23.9 mg, 71% yield). The NMR data are in agreement with those reported in the literature.[11]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (d, *J* = 15.7 Hz, 1H), 7.38 (d, *J* = 5.2 Hz, 1H), 7.26 (d, *J* = 3.5 Hz, 1H), 7.06 (t, *J* = 3.6 Hz, 1H), 6.25 (d, *J* = 15.7 Hz, 1H), 3.80 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.4, 139.6, 137.4, 131.1, 128.6, 128.2, 116.6, 51.1.

(*E*)-Methyl-3-(pyridin-2-yl)acrylate (2q)



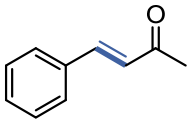
According to General Procedure, the title compound was synthesized from methyl 2-pyridylpropiolate (32.2 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 9:1), the product **2q** was obtained as a white solid

(19.6 mg, 60% yield). The NMR data are in agreement with those reported in the literature.[16]

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.65-8.60 (m, 1H), 7.75-7.65 (m, 2H), 7.42 (d, *J* = 7.9 Hz, 1H), 7.32-7.24 (m, 1H), 6.93 (d, *J* = 15.7 Hz, 1H), 3.82 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.2, 152.8, 150.1, 143.5, 136.8, 124.3, 122.0, 51.9.

(*E*)-4-Phenylbut-3-en-2-one (2r)

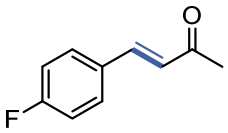


According to General Procedure, the title compound was synthesized from **1r** (29.1 μL, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 9:1), the product **2r** was obtained as a white solid (21.3 mg, 73% yield). The NMR data are in agreement with those reported in the literature.[17]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52-7.44 (m, 3H), 7.38-7.33 (m, 3H), 6.67 (d, *J* = 16.3 Hz, 1H), 2.34 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 199.4, 143.5, 134.4, 130.6, 129.0, 128.3, 127.2, 27.5.

(*E*)-4-(4-Fluorophenyl)but-3-en-2-one (2s)



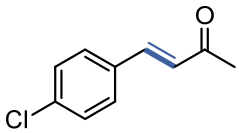
According to General Procedure, the title compound was synthesized from 4-(4-fluorophenyl)but-3-yn-2-one[18] (32.4 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2s** was obtained as colorless oil (15.7 mg, 48% yield). The NMR data are in agreement with those reported in the literature.[19]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55-7.51 (m, 2H), 7.48 (d, *J* = 16.2 Hz, 1H), 7.14-7.05 (m, 2H), 6.65 (d, *J* = 16.2 Hz, 1H), 2.38 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 198.3, 165.4 (d, *J* = 252.1 Hz), 142.1, 130.2 (d, *J* = 8.3 Hz), 127.0 (d, *J* = 2.4 Hz), 116.2 (d, *J* = 22.0 Hz), 27.7.

^{19}F NMR (565 MHz, $CDCl_3$): δ –109.0.

(*E*)-4-(4-Chlorophenyl)but-3-en-2-one (2t)

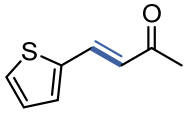


According to General Procedure, the title compound was synthesized from 4-(4-chlorophenyl)but-3-yn-2-one[20] (35.6 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2t** was obtained as a white solid (18.5 mg, 51% yield). The NMR data are in agreement with those reported in the literature.[21]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41-7.39 (m, 3H), 7.30-7.28 (m, 2H), 6.61 (d, *J* = 16.2 Hz, 1H), 2.30 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 198.2, 142.0, 136.5, 133.0, 129.5, 129.4, 127.6, 27.8.

(*E*)-4-(2-Thienyl)but-3-en-2-one (2u)

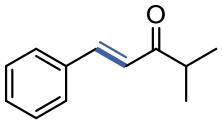


According to General Procedure, the title compound was synthesized from 4-(thiophen-2-yl)but-3-yn-2-one[18] (30.0 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2u** was obtained as yellow oil (22.2 mg, 73% yield). The NMR data are in agreement with those reported in the literature.[22]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, *J* = 15.9 Hz, 1H), 7.40 (d, *J* = 5.1 Hz, 1H), 7.29 (d, *J* = 3.8 Hz, 1H), 7.07 (t, *J* = 4.5 Hz, 1H), 6.53 (d, *J* = 15.9 Hz, 1H), 2.34 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 197.8, 139.7, 135.8, 131.6, 129.0, 128.3, 125.8, 27.7.

(*E*)-1-Cyclopropyl-3-phenylpropen-1-one (2v)

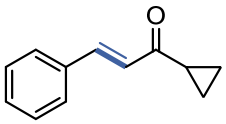


According to General Procedure, the title compound was synthesized from 4-methyl-1-phenylpent-1-yn-3-one[23] (35 μL, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2v** was obtained as yellow oil (10.4 mg, 30% yield). The NMR data are in agreement with those reported in the literature.[24]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 (d, *J* = 16.0 Hz, 1H), 7.58-7.56 (m, 2H), 7.42-7.37 (m, 3H), 6.82 (d, *J* = 16.0 Hz, 1H), 2.98-2.91 (m, 1H), 1.19 (d, *J* = 6.9 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 203.9, 142.4, 134.7, 130.4, 128.9, 128.3, 124.5, 39.3, 18.5.

(*E*)-1-Cyclopropyl-3-phenylprop-2-en-1-one(2w)



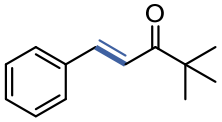
According to General Procedure, the title compound was synthesized from 1-cyclopropyl-3-phenylprop-2-yn-1-one[25] (30.4 μL, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 5:5), the product **2w** was obtained

as colorless oil (25.7 mg, 75% yield). The NMR data are in agreement with those reported in the literature.[26]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 (d, *J* = 16.1 Hz, 1H), 7.58-7.56 (m, 2H), 7.41-7.39 (m, 3H), 6.88 (d, *J* = 16.1 Hz, 1H), 2.30-2.20 (m, 1H), 1.21-1.09 (m, 2H), 1.00-0.97 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 200.3, 142.1, 134.8, 130.4, 129.0, 128.4, 126.5, 19.7, 11.5.

(*E*)-Benzylidenepinacolone (2x)

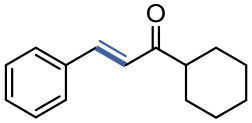


According to General Procedure, the title compound was synthesized from 4,4-dimethyl-1-phenyl-1-pentyn-3-one[27] (38 μL, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 99:1), the product **2x** was obtained as yellow oil (20.0 mg, 53% yield). The NMR data are in agreement with those reported in the literature.[28]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (d, *J* = 15.7 Hz, 1H), 7.58-7.55 (m, 2H), 7.38-7.37 (m, 3H), 7.12 (d, *J* = 15.7 Hz, 1H), 1.23 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 204.3, 142.9, 135.0, 130.2, 128.9, 128.3, 120.7, 43.3, 26.3.

(*E*)-1-Cyclohexyl-3-phenylprop-2-en-1-one (2y)



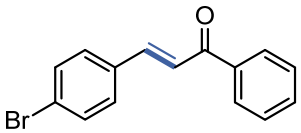
According to General Procedure, the title compound was synthesized from 1-cyclohexyl-3-phenylprop-2-yn-1-one[23] (40 μL, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on

silica gel (eluent = petroleum ether/ethyl acetate = 99:1), the product **2y** was obtained as yellow oil (36.2 mg, 84% yield). The NMR data are in agreement with those reported in the literature.[29]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, *J* = 16.0 Hz, 1H), 7.59-7.58 (m, 2H), 7.45-7.39 (m, 3H), 6.85 (d, *J* = 16.0 Hz, 1H), 2.27-2.65 (m, 1H),1.97-1.82 (m, 4H), 1.76-1.72 (m, 1H), 1.51-1.26 (m, 5H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 203.2, 142.3, 134.8, 130.3, 128.9, 128.3, 124.7, 49.4, 28.7, 25.9, 25.8.

(2*E*)-3-(4-Bromophenyl)-1-phenylprop-2-en-1-one (2z)

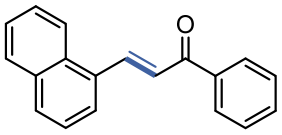


According to General Procedure, the title compound was synthesized from 3-(4-bromophenyl)-1-phenylprop-2-yn-1-one[30] (57.0 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 99:1), the product **2z** was obtained as a white solid (19.2 mg, 33% yield). The NMR data are in agreement with those reported in the literature.[31]

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (d, *J* = 7.7 Hz, 2H), 7.74 (d, *J* = 15.6 Hz, 1H), 7.66-7.47 (m, 8H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 190.3, 143.5, 138.1, 133.9, 133.1, 132.3, 129.9, 128.8, 128.6, 124.9, 122.6.

(*E*)-3-(Naphthalen-1-yl)-1-phenylprop-2-en-1-one (2aa)



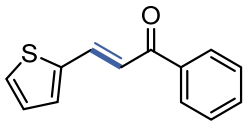
According to General Procedure, the title compound was synthesized from 3-(naphthalen-1-yl)-1-phenylprop-2-yn-1-one[32] (51.2 mg, 0.2 mmol, 1.0 equiv.) under

standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2aa** was obtained as a yellow solid (30.0 mg, 58% yield). The NMR data are in agreement with those reported in the literature.[33]

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.69 (d, *J* = 15.4 Hz, 1H), 8.27 (d, *J* = 8.3 Hz, 1H), 8.10-8.08 (m, 2H), 7.97-7.87 (m, 3H), 7.68-7.49 (m, 7H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 190.4, 141.9, 138.3, 133.8, 133.0, 132.5, 131.9, 131.0, 128.9, 128.8, 128.7, 127.1, 126.4, 125.6, 125.2, 124.8, 123.6.

(*E*)-1-Phenyl-3-(thiophen-2-yl)-2-propen-1-one (2ab)

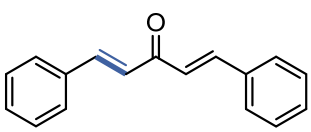


According to General Procedure, the title compound was synthesized from 1-phenyl-3-(thiophen-2-yl)prop-2-yn-1-one[34] (38 μL, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 99:1), the product **2ab** was obtained as a yellow solid (17.1 mg, 40% yield). The NMR data are in agreement with those reported in the literature.[35]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (d, *J* = 7.9 Hz, 2H), 7.89 (d, *J* = 15.3 Hz, 1H), 7.54-7.50 (m, 1H), 7.46-7.42 (m, 2H), 7.37 (d, *J* = 5.1 Hz, 1H), 7.30-7.29 (m, 2H), 7.05-7.02 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 189.9, 140.4, 138.1, 137.3, 132.8, 132.1, 128.8, 128.7, 128.4, 128.4, 120.8.

Dibenzylideneacetone (2ac)

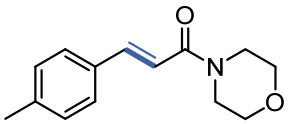


According to General Procedure, the title compound was synthesized from (E)-1,5-diphenyl-1-penten-4-yn-3-one[36] (46.4 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 99:1), the product **2ac** was obtained as a white solid (14.6 mg, 31% yield). The NMR data are in agreement with those reported in the literature.[37]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, *J* = 15.9 Hz, 2H), 7.63 (dd, *J* = 6.8, 3.0 Hz, 4H), 7.45-7.30 (m, 6H), 7.09 (d, *J* = 15.9 Hz, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 189.1, 143.5, 134.9, 130.6, 129.1, 128.5, 125.5.

(*E*)-1-Morpholino-3-(p-tolyl)prop-2-en-1-one (2ad)

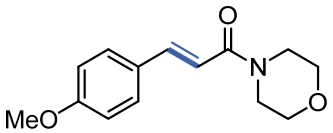


According to General Procedure, the title compound was synthesized from **1ad** (45.8 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 6:4), the product **2ad** was obtained as a yellow solid (28.2 mg, 61% yield). The NMR data are in agreement with those reported in the literature.[38]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (d, *J* = 15.4 Hz, 1H), 7.41 (d, *J* = 7.8 Hz, 2H), 7.17 (d, *J* = 7.8 Hz, 2H), 6.79 (d, *J* = 15.4 Hz, 1H), 3.70-3.66 (m, 8H), 2.35 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 165.3, 143.3, 140.2, 132.4, 129.6, 127.9, 115.5, 67.0, 46.3, 42.6, 21.5.

(*E*)-3-(4-Methoxyphenyl)-1-morpholinoprop-2-en-1-one (2ae)



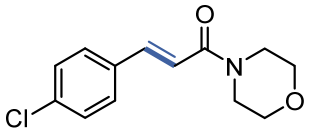
According to General Procedure, the title compound was synthesized from **1ae** (49.1 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and

purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 6:4), the product **2ae** was obtained as a white solid (32.1 mg, 65% yield). The NMR data are in agreement with those reported in the literature.[9]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (d, *J* = 15.3 Hz, 1H), 7.46-7.44 (m, 2H), 6.88-6.85 (m, 2H), 6.69 (d, *J* = 15.3 Hz, 1H), 3.80 (s, 3H), 3.69-3.65 (m, 8H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 165.9, 160.9, 142.9, 129.4, 127.8, 114.2, 114.0, 66.9, 55.4, 46.2, 42.5.

(*E*)-3-(4-Chlorophenyl)-1-morpholinoprop-2-en-1-one (2af)

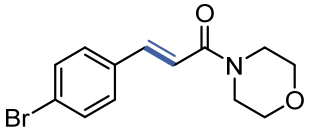


According to General Procedure, the title compound was synthesized from **1af** (49.9 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 6:4), the product **2af** was obtained as a white solid (22.7 mg, 45% yield). The NMR data are in agreement with those reported in the literature.[39]

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.63 (d, *J* = 15.4 Hz, 1H), 7.44-7.43 (m, 2H), 7.34-7.32 (m, 2H), 6.81 (d, *J* = 15.4 Hz, 1H), 3.72-3.64 (m, 8H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 165.9, 141.8, 135.6, 133.6, 129.1, 129.0, 117.1, 66.8, 46.2, 42.5.

4-[(2*E*)-3-(4-Bromophenyl)prop-2-enoyl]morpholine (2ag)



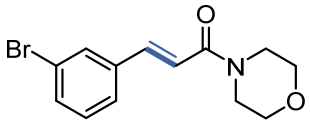
According to General Procedure, the title compound was synthesized from **1ag** (58.8 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 5:5), the product **2ag** was obtained as a white solid (47.4 mg, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61 (d, *J* = 15.4 Hz, 1H), 7.50-7.48 (m, 2H), 7.38-7.36 (m, 2H), 6.82 (d, *J* = 15.4 Hz, 1H), 3.72-3.64 (m, 8H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 165.3, 141.5, 134.0, 132.0, 129.2, 123.9, 116.5, 66.8, 46.3, 42.5.

HRMS: Calculated for $C_{13}H_{14}BrNO_2Na$ $[M+Na]^+$: 318.0100, Found: 318.0102.

3-(3-Bromophenyl)-1-morpholinoprop-2-en-1-one (2ah)



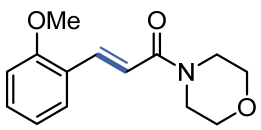
According to General Procedure, the title compound was synthesized from **1ah** (58.8 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 5:5), the product **2ah** was obtained as a white solid (58.6 mg, 99% yield).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66-7.65 (m, 1H), 7.60 (d, *J* = 15.4 Hz, 1H), 7.47-7.45 (m, 1H), 7.41-7.39 (m, 1H), 7.25-7.21 (m, 1H), 6.83 (d, *J* = 15.4 Hz, 1H), 3.72-3.64 (m, 8H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 165.1, 141.6, 137.2, 132.5, 130.4, 130.1, 126.8, 123.0, 118.0, 66.8, 46.3, 42.5.

HRMS(ESI Positive)**:** Calculated for $C_{13}H_{14}BrNaO_2$ $[M+Na]^+$: 318.0100, Found: 318.0101.

(*E*)-3-(2-Methoxyphenyl)-1-morpholin-4-ylprop-2-en-1-one (2ai)

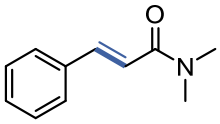


According to General Procedure, the title compound was synthesized from **1ai** (49.1 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 6:4), the product **2ai** was obtained as a white solid (47.0 mg, 95% yield).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 15.5 Hz, 1H), 7.47-7.44 (m, 1H), 7.32-7.26 (m, 1H), 6.96-6.87(m, 3H), 3.85 (s, 3H), 3.69-365 (m, 8H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 166.2, 158.2, 138.7, 130.8, 129.1, 124.1, 120.6, 117.5, 111.1, 66.9, 55.5, 46.2, 42.4.

(*E*)-*N*,*N*-dimethylcinnamamide (2aj)

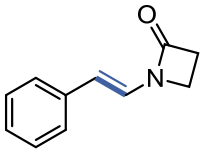


According to General Procedure, the title compound was synthesized from *N*,*N*-dimethyl-3-phenylpropiolamide[5] (34.6 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 5:5), the product **2aj** was obtained as colorless oil (34.3 mg, 98% yield). The NMR data are in agreement with those reported in the literature.[40]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (d, *J* = 15.4 Hz, 1H), 7.54 (dd, *J* = 7.4, 1.9 Hz, 2H), 7.38-7.34 (m, 3H), 6.90 (d, *J* = 15.4 Hz, 1H), 3.13 (d, *J* = 41.0 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 166.8, 142.4, 135.4, 129.6, 128.9, 127.9, 117.5, 37.5, 36.0.

2-(Phenyl)ethen-1-ylazetidinone (2ak)

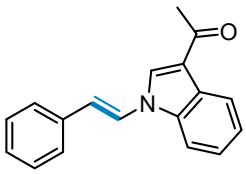


According to General Procedure, the title compound was synthesized from 1-(phenylethynyl)azetidin-2-one[41] (34.2 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2ak** was obtained as a white solid (16.6 mg, 48% yield).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29 (d, *J* = 4.3 Hz, 4H), 7.26-7.22 (m, 1H), 7.21-7.16 (m, 1H), 5.96-5.93 (m, 1H), 3.55-3.52 (m, 2H), 3.08-3.06 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 164.2, 135.7, 128.8, 126.9, 125.5, 121.3, 111.1, 38.4, 36.3.

(*E*)-1-(1-styryl-1H-indol-3-yl)ethan-1-one (2al)

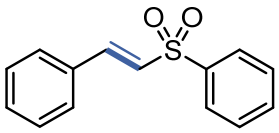


According to General Procedure, the title compound was synthesized from 1-(1-(phenylethynyl)-1H-indol-3-yl)ethan-1-one[41] (51.9 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2al** was obtained as white solid (30.3 mg, 58% yield).

^{1}H NMR (**400 MHz, $CDCl_3$**) δ 8.45-8.39 (m, 1H), 8.07 (s, 1H), 7.60 (d, *J* = 14.4 Hz, 1H), 7.56-7.28 (m, 9H), 6.87 (d, *J* = 14.4 Hz, 1H), 2.59 (s, 3H).

^{13}C NMR (**101 MHz, $CDCl_3$**) δ 193.4, 136.5, 134.9, 130.3, 129.0, 128.0, 126.5, 126.2, 124.2, 123.5, 122.9, 122.6, 119.3, 118.7, 110.0, 27.9.

Phenyl styryl sulfone (2am)

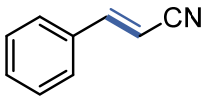


According to General Procedure, the title compound was synthesized from 2-(benzenesulfonyl)ethynylbenzene[42](48.5 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2am** was obtained as a yellow solid (22.3 mg, 46% yield).[43]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99-7.92 (m, 2H), 7.69 (d, *J* = 15.3 Hz, 1H), 7.65-7.58 (m, 1H), 7.58-7.50 (m, 2H), 7.50-7.45 (m, 2H), 7.44-7.35 (m, 3H), 6.87 (d, *J* = 15.3 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 142.5, 140.7, 133.4, 132.4, 131.3, 129.3, 129.1, 128.6, 127.7, 127.3.

Cinnamic nitrile (2an)

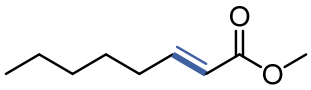


According to General Procedure, the title compound was synthesized from 2-propynenitrile[44] (25.4 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2an** was obtained as colorless oil (12.4 mg, 48% yield).[45]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48-7.35 (m, 6H), 5.88 (d, *J* = 16.6 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 150.6, 133.5, 131.3, 129.2, 127.4, 118.2, 96.4.

Methyl (E)-oct-2-enoate (2ao)

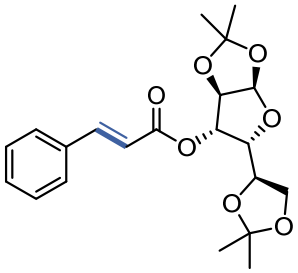


According to General Procedure, the title compound was synthesized from methyl 2-octynoate (37 μL, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = pentane/diethyl ether = 19/1), the product **2ao** was obtained as a colorless oil (16.2 mg, 52% yield).[46]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.02-6.90 (m, 1H), 5.80 (dt, *J* = 15.6, 1.5 Hz, 1H), 3.71 (s, 3H), 2.18 (q, *J* = 7.2 Hz, 2H), 1.44 (p, *J* = 7.2 Hz, 2H), 1.31-1.26 (m, 4H), 0.91-0.83 (m, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.20, 149.83, 120.78, 51.35, 32.17, 31.28, 27.68, 22.42, 13.94.

5-(2,2-Dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-6-yl cinnamate (2ar)



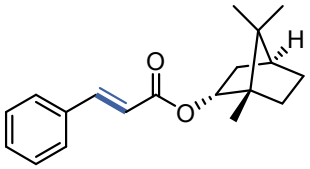
According to General Procedure, the title compound was synthesized from 5-(2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-6-yl 3-phenylpropiolate[47] (77.7 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2ar** was obtained as a white solid (59.2 mg, 76% yield).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, *J* = 16.0 Hz, 1H), 7.55-7.53 (m, 2H), 7.43-7.38 (m, 3H), 6.44 (d, *J* = 16.0 Hz, 1H), 5.94-5.93 (m, 1H), 5.40-5.39 (m, 1H), 4.59-4.58 (m, 1H), 4.37-4.26 (m, 2H), 4.11-4.08 (m, 2H), 1.55 (s, 3H), 1.43 (s, 3H), 1.32-1.31 (m, 6H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 165.6, 146.1, 134.0, 130.7, 129.0, 128.2, 117.1, 112.3, 109.4, 105.1, 83.4, 79.8, 76.2, 72.5, 67.1, 26.9, 26.7, 26.2, 25.3.

HRMS: Calculated for $C_{21}H_{26}NaO_7$ $[M+Na]^+$:413.1571, Found: 413.1573.

(1*R*)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-yl cinnamate (2as)



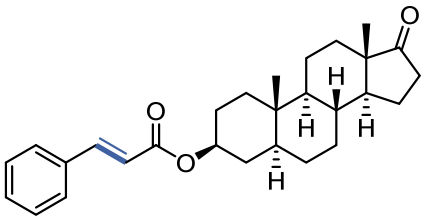
According to General Procedure, the title compound was synthesized from (1R)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl 3-phenylpropiolate[48] (56.5 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2as** was obtained as a white solid (53.9 mg, 95% yield).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (d, *J* = 16.0 Hz, 1H), 7.56-7.53 (m, 2H), 7.40-7.38 (m, 3H), 6.48 (d, *J* = 16.0 Hz, 1H), 5.04-5.00 (m, 1H), 2.11-2.00 (m, 1H), 1.81-1.76 (m, 1H), 1.72-1.70 (m, 1H), 1.43-1.23 (m, 2H), 1.09-1.04 (m, 1H), 0.95 (s, 3H), 0.90-0.89 (m, 6H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.4, 144.2, 134.6, 130.2, 128.9, 128.1, 118.8, 80.0, 49.0, 47.9, 45.0, 36.9, 28.1, 27.3, 19.8, 18.9, 13.6.

HRMS: Calculated for $C_{19}H_{24}NaO_2$ $[M+Na]^+$:307.1669, Found: 307.1667.

(3*S*,5*S*,8*R*,9*S*,10*S*,13*S*,14*S*)-10,13-Dimethyl-17-oxohexadecahydro-1H-cyclopenta[a]phenanthren-3-yl cinnamate (2at)



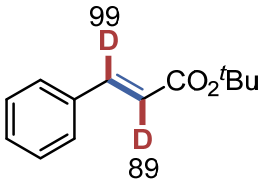
According to General Procedure, the title compound was synthesized from (3S,5S,8R,9S,10S,13S,14S)-10,13-dimethyl-17-oxohexadecahydro-1H-cyclopenta[a] phenanthren-3-yl 3-phenylpropiolate[48] (84.0 mg, 0.2 mmol, 1.0 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2at** was obtained as a white solid (75.7 mg, 90% yield).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66 (d, *J* = 16.2 Hz, 1H), 7.51-7.49 (m, 2H), 7.38-7.36 (m, 3H), 6.41 (d, *J* = 16.0 Hz, 1H), 4.83 (tt, *J* = 11.0, 4.9 Hz, 1H), 2.46-2.39 (m, 1H), 2.11-2.01 (m, 1H), 1.95-1.88 (m, 2H), 1.82-1.76 (m, 3H), 1.72-1.64 (m, 3H), 1.60-1.44 (m, 4H), 1.34-1.24 (m, 6H), 1.12-0.97 (m, 2H), 0.88-0.84 (m, 6H), 0.77-0.70 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 166.6, 144.4, 134.5, 134.4, 134.3, 130.2, 128.9, 128.05, 128.03, 127.97, 127.8, 118.7, 73.6, 54.3, 51.4, 47.8, 44.7, 36.7, 35.9, 35.7, 35.0, 34.1, 31.5, 30.8, 28.3, 27.5, 21.8, 20.5, 13.8.

HRMS: Calculated for $C_{28}H_{36}NaO_3$ $[M+Na]^+$:443.2557, Found: 443.2558.

Tert-butyl cinnamate-2,3-d_2 (2c-d_2)



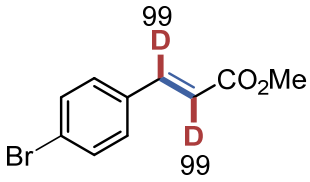
According to General Procedure, the title compound was synthesized from tert-butyl 3-phenylpropiolate[1] (39.0 μL,, 0.2 mmol, 1.0 equiv.) and D_2O (40 μL, 1.0 mmol, 5.0 equiv.) under otherwise standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2c-d_2** was obtained as colorless oil (32.1 mg, 78% yield).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59-7.44 (m, 2H), 7.41-7.37 (m, 3H), 6.42-6.22 (m, 0.11H), 1.57 (s, 6H), 1.46 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 166.4, 143.6-143.5 (m), 135.9, 128.8, 128.0, 127.9, 120.2-120.1 (m), 28.2, 28.0.

HRMS: Calculated for $C_{13}H_{14}D_2NaO_2$ $[M+Na]^+$:229.1168, Found: 229.1169.

Methyl (*E*)-3-(4-bromophenyl)acrylate-d_2 (2h-d_2)



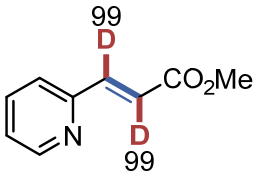
According to General Procedure, the title compound was synthesized from **1h** (47.8 mg, 0.2 mmol, 1.0 equiv.) and D_2O (40 μL, 1.0 mmol, 5.0 equiv.) under otherwise standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 98:2), the product **2h-d_2** was obtained as a white solid (38.8 mg, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51 (d, J = 8.1 Hz, 2H), 7.37 (d, J = 8.2 Hz, 2H), 3.80 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.2, 143.5-143.4 (m), 133.2, 132.1, 129.4, 124.6, 118.5-118.4 (m), 51.8.

HRMS: Calculated for $C_{10}H_7D_2BrNaO_2$ $[M+Na]^+$:264.9804, Found: 264.9804.

Methyl (*E*)-3-(pyridin-2-yl)acrylate-d_2 (2q-d_2)



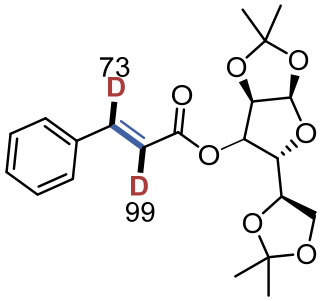
According to General Procedure, the title compound was synthesized from methyl 2-pyridylpropiolate (32.2 mg, 0.2 mmol, 1.0 equiv.) and D_2O (40 μL, 1mmol, 5 equiv.) under otherwise standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 9:1), the product **2q-*d*$_2$** was obtained as a white solid (19.4 mg, 59% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.66-8.65 (m, 1H), 7.75-7.67 (m, 1H), 7.43-7.42 (m, 1H), 7.30-7.25 (m, 1H), 3.82 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 167.2, 152.8, 150.2, 143.4-143.1 (m), 136.8, 124.3, 121.8-121.3 (m), 51.8.

HRMS: Calculated for $C_9H_7D_2NaNO_2$ $[M+Na]^+$:188.0651, Found: 118.0652.

(3a*R*,5*R*,6a*R*)-5-((*R*)-2,2-Dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro [2,3-d][1,3]dioxol-6-yl cinnamate-2,3-d_2 (2ar-d_2)



According to General Procedure, the title compound was synthesized from 5-(2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-6-yl 3-phenylpropiolate[47] (77.7 mg, 0.2 mmol, 1.0 equiv.) and D_2O (40 μL, 1mmol, 5 equiv.) under standard conditions. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ethyl acetate = 95:5), the product **2ar-*d*$_2$** was obtained as a white solid (59.5 mg, 76% yield).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75-7.65 (m, 0.27H), 7.59-7.51 (m, 2H), 7.43-7.42 (m, 3H), 5.96 (s, 1H), 5.41 (s, 1H), 4.61 (s, 1H), 4.32 (s, 2H), 4.13-4.10 (m, 2H), 1.66 (s, 1H), 1.57 (s, 3H), 1.45 (s, 3H), 1.33 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 165.5, 147.0-146.1 (m), 134.0, 130.7, 129.0, 128.2, 117.1-117.0 (m), 112.3, 109.4, 105.1, 83.4, 79.8, 76.2, 72.5, 67.1, 26.9, 26.7, 26.2, 25.3.

HRMS: Calculated for $C_{21}H_{24}D_2NaO_7$ $[M+Na]^+$:415.1696, Found: 415.1694.

References

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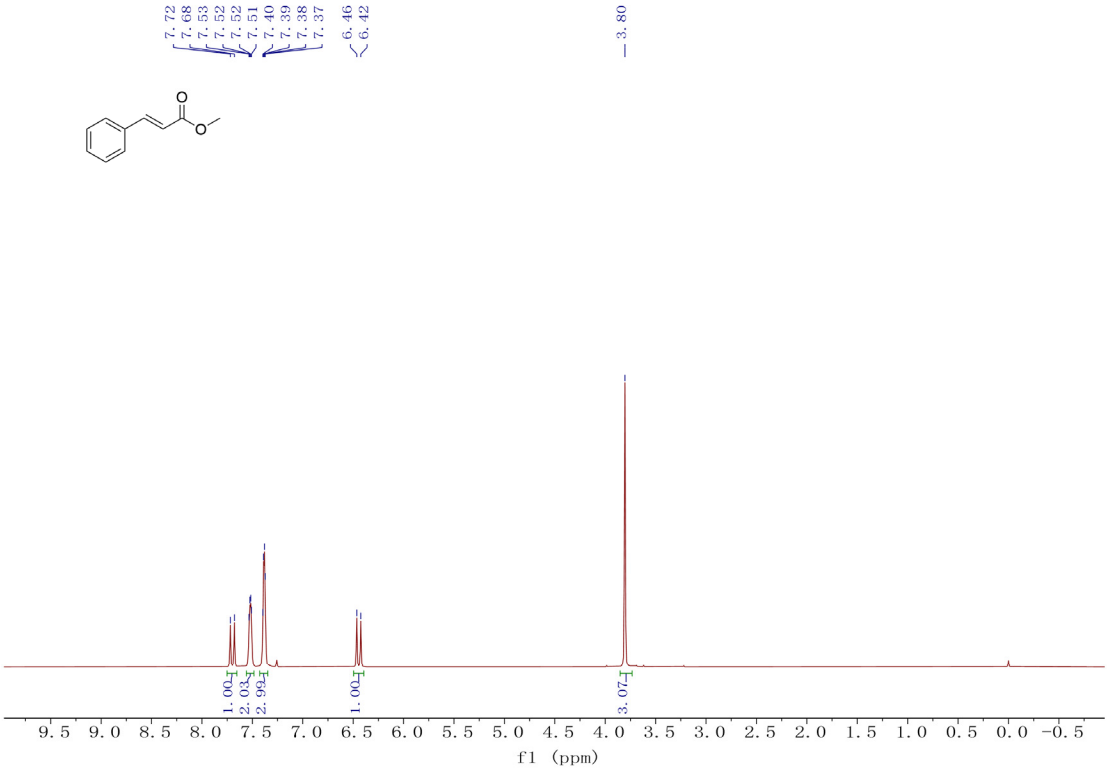
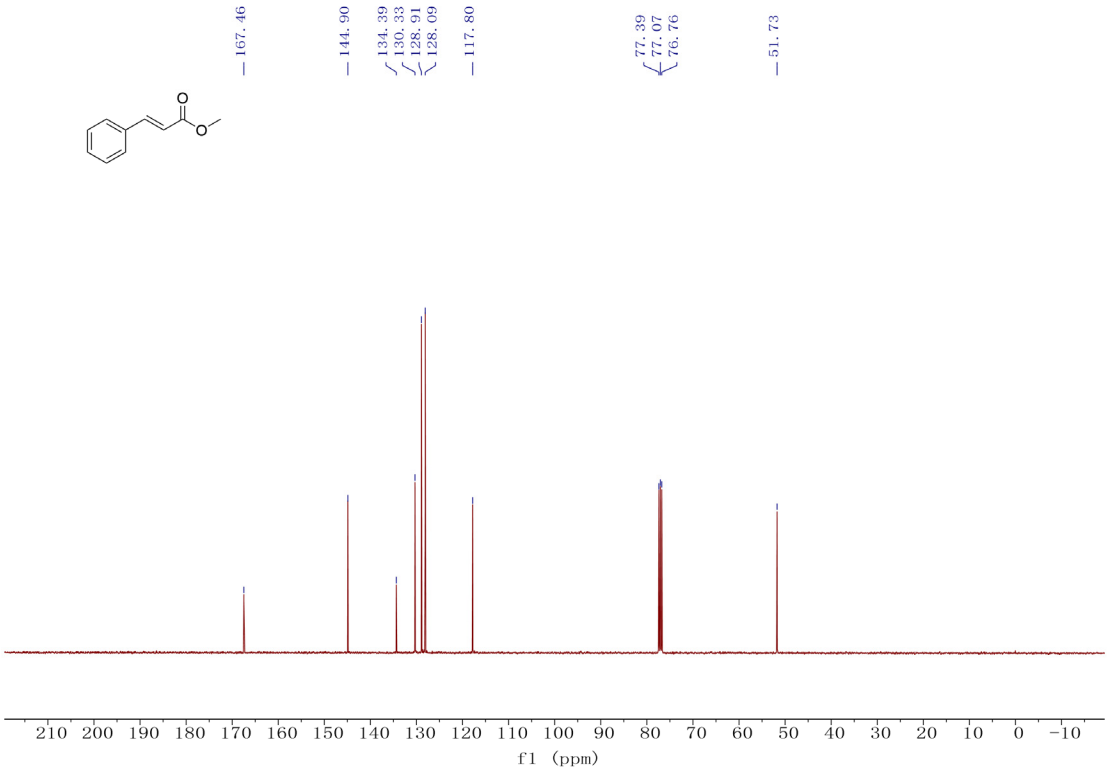
Copies of NMR Spectra

Methyl (*E*)-3-phenylacrylate (2a)



^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2a**



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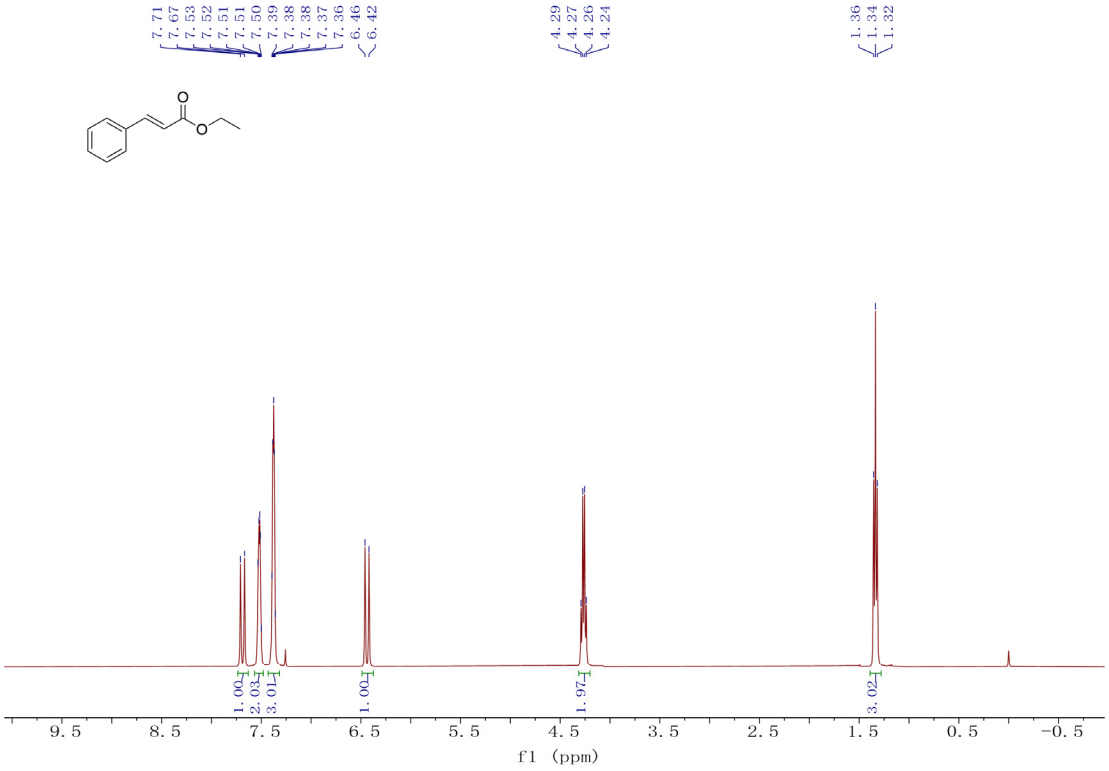

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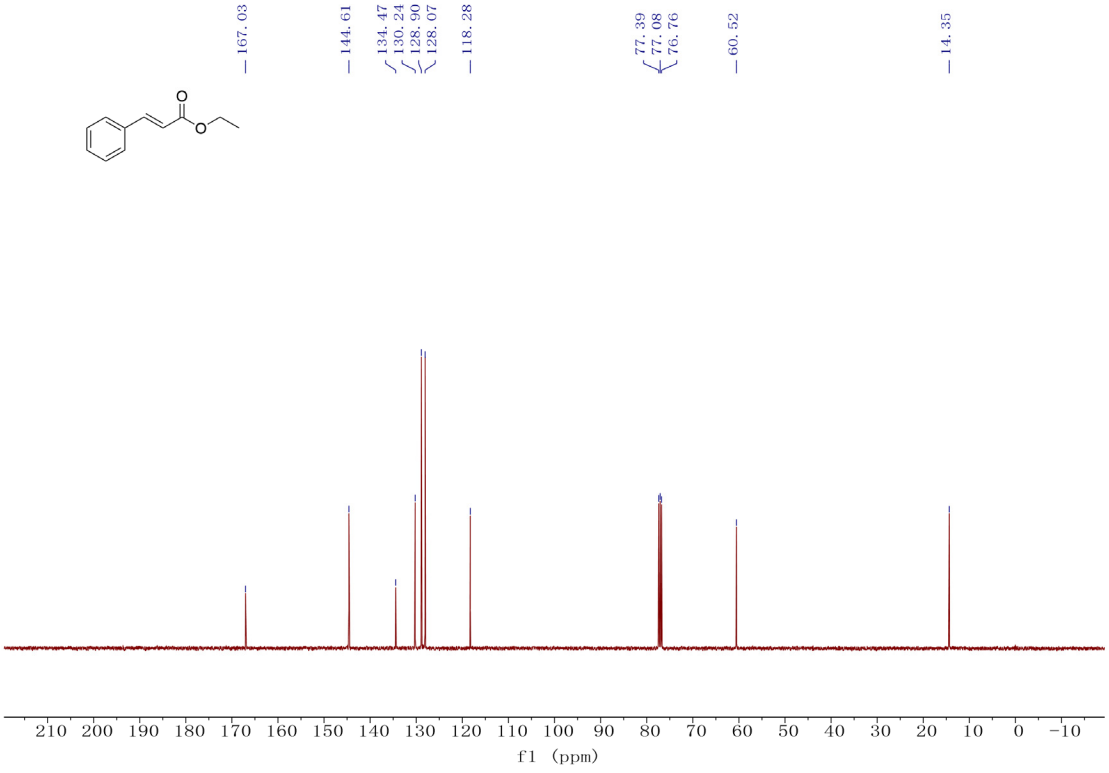

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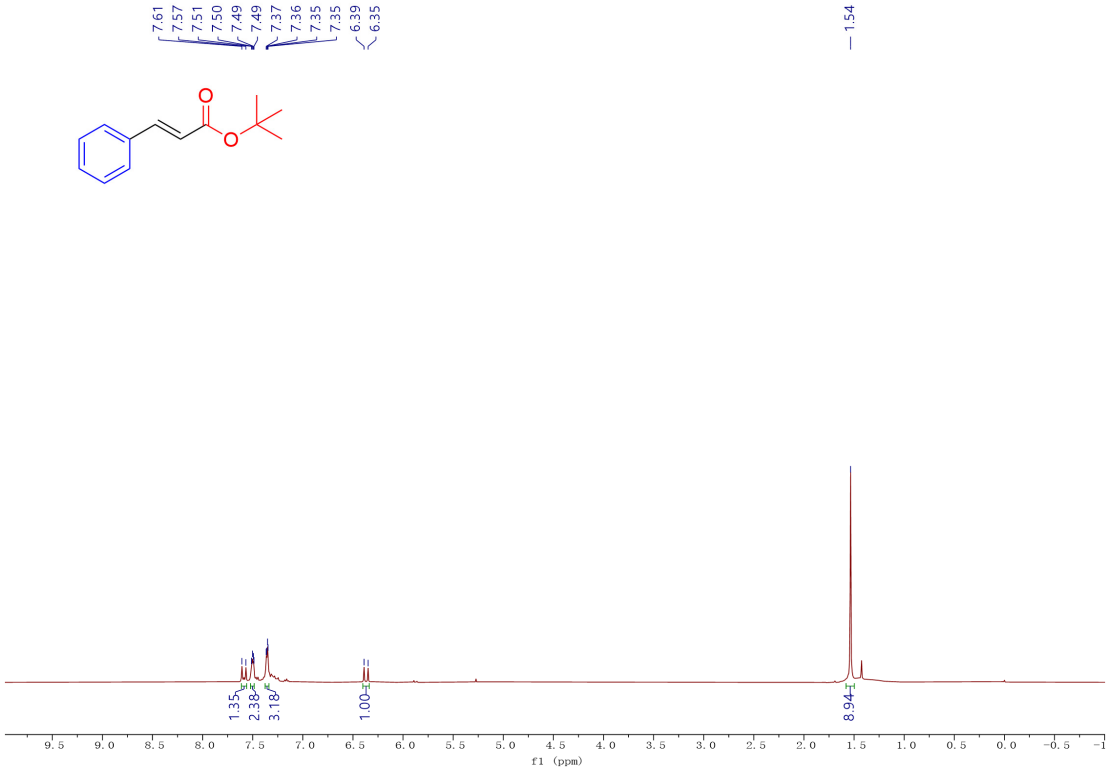
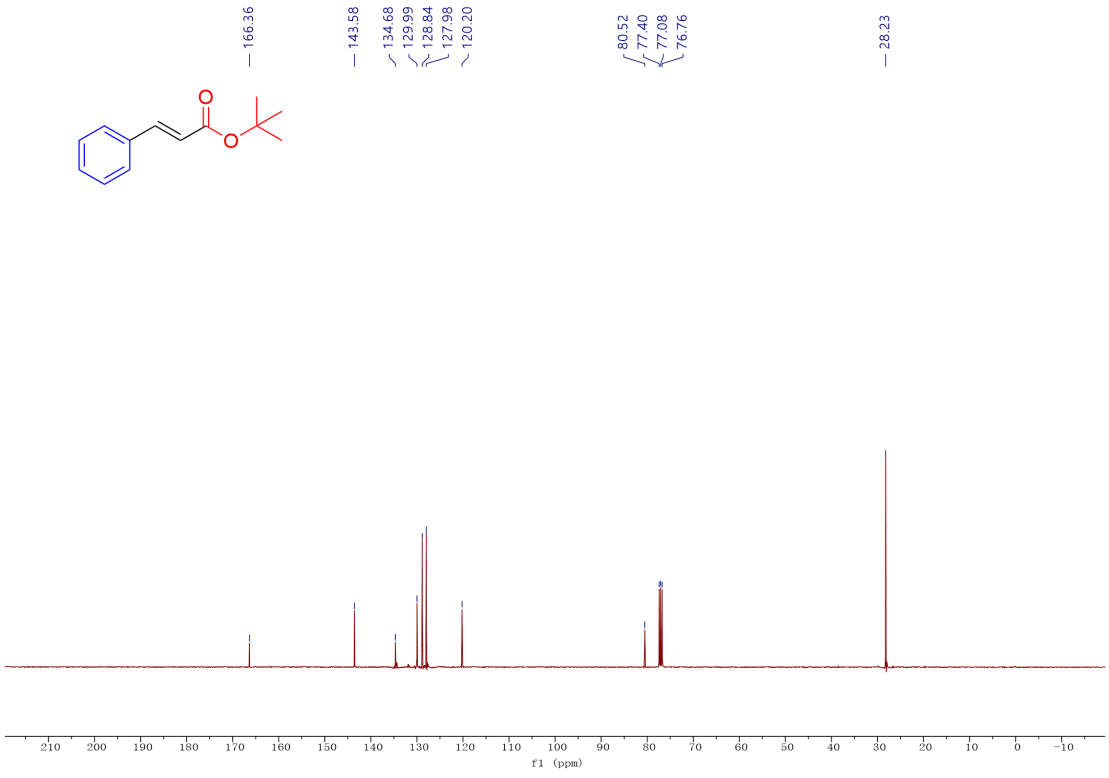
Tert-butyl cinnamate (2c)



^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2c**



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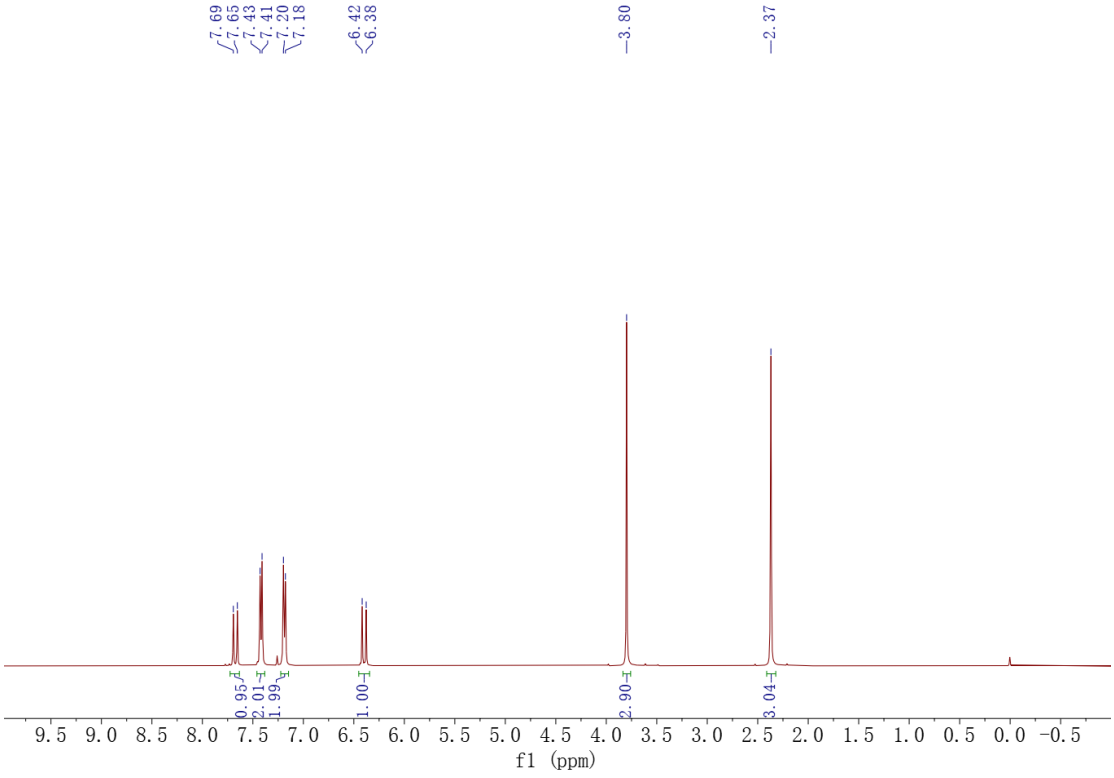
Methyl (*Z*)-3-(p-tolyl)acrylate (2d)



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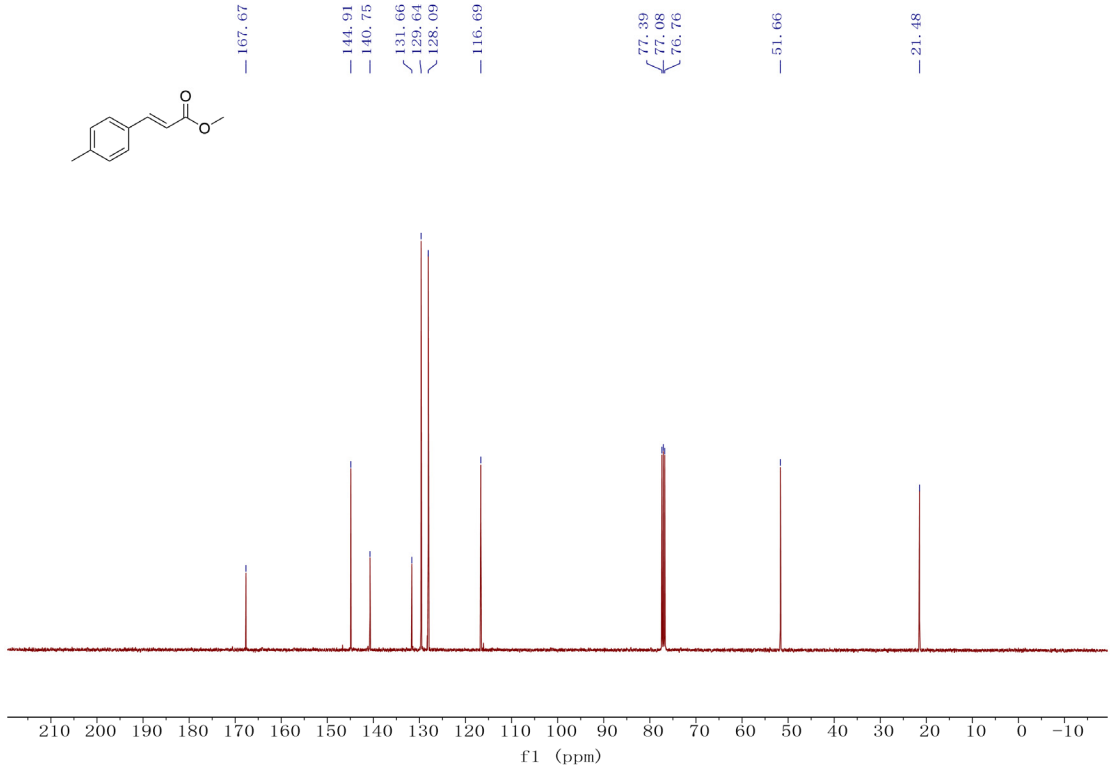

^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2d**

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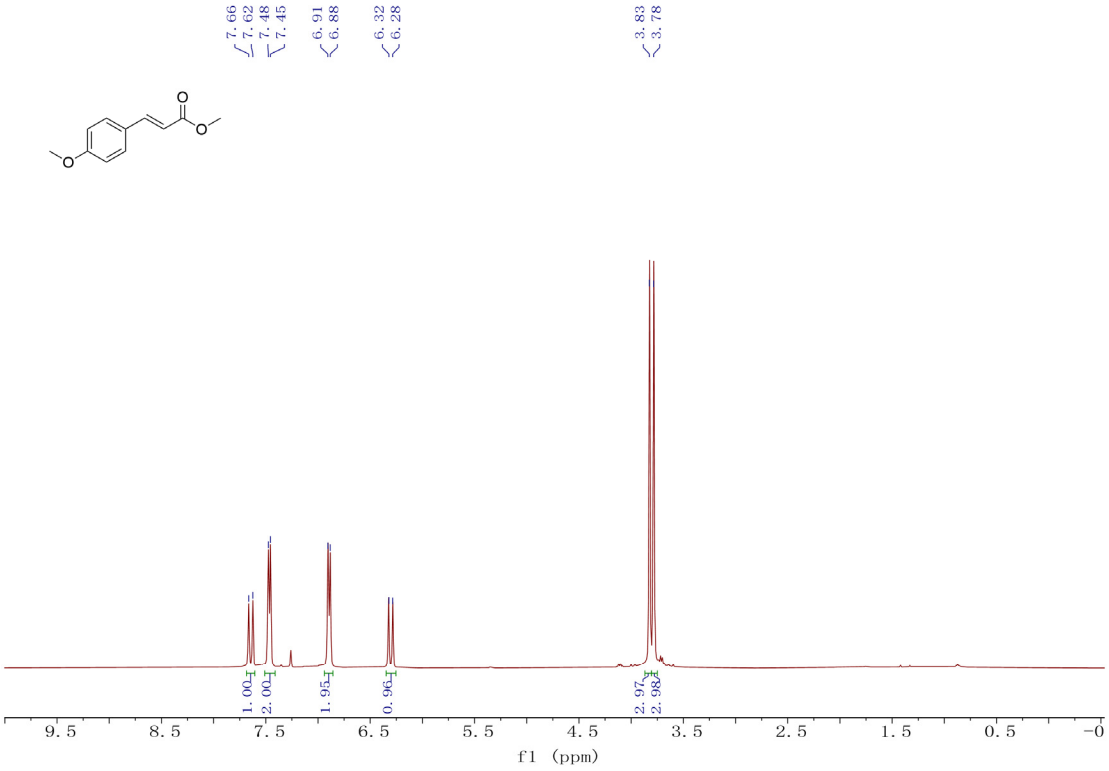

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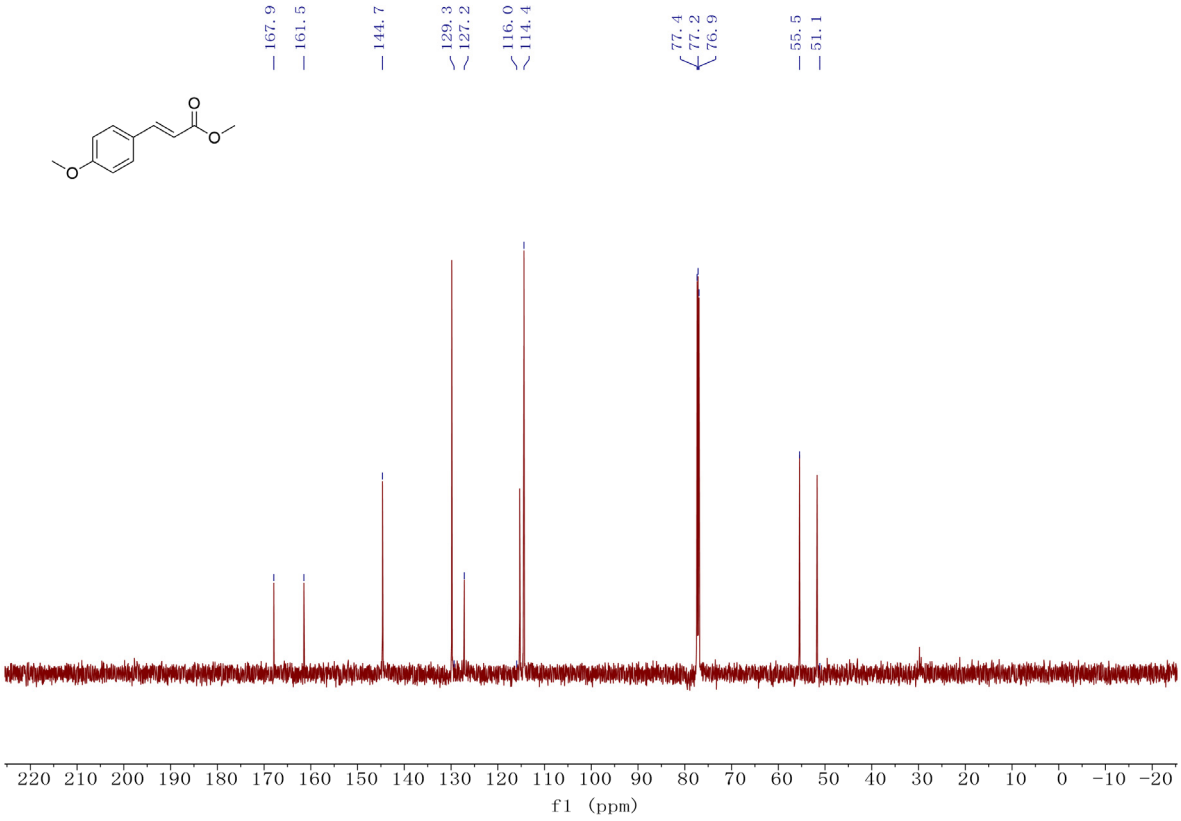
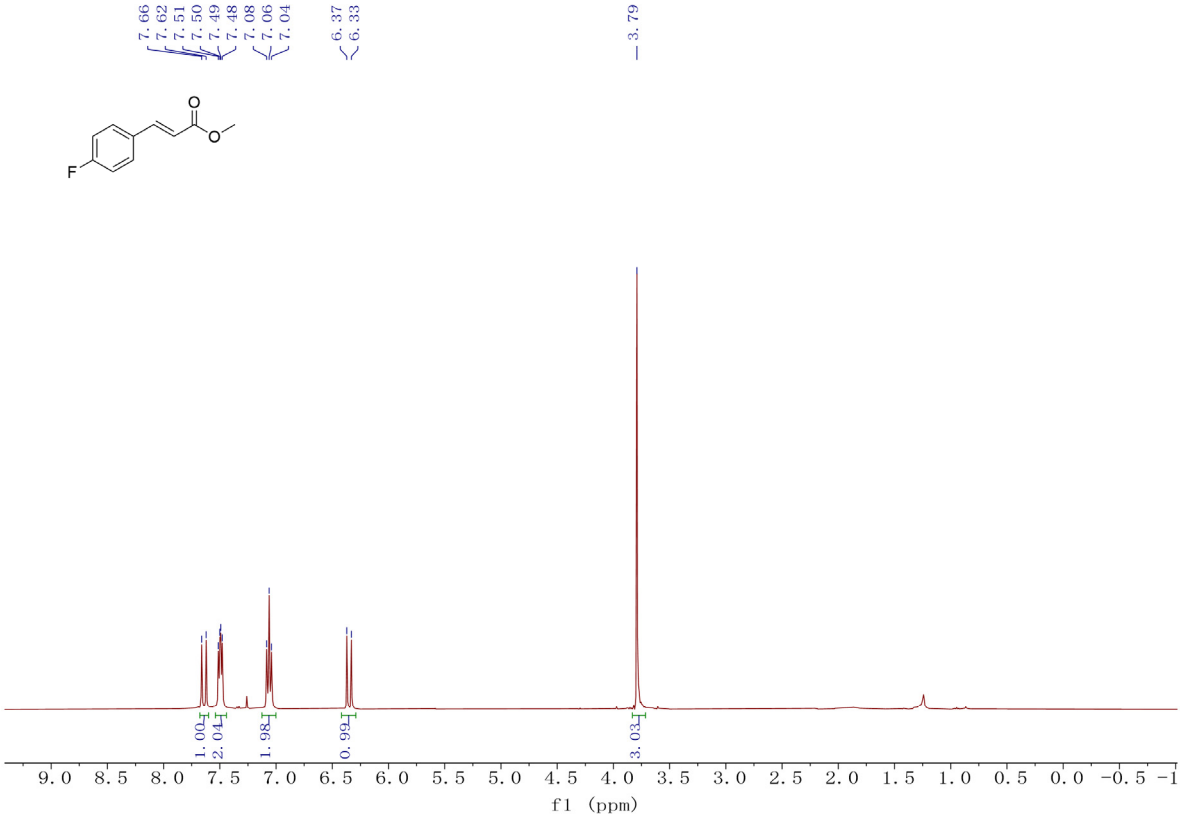
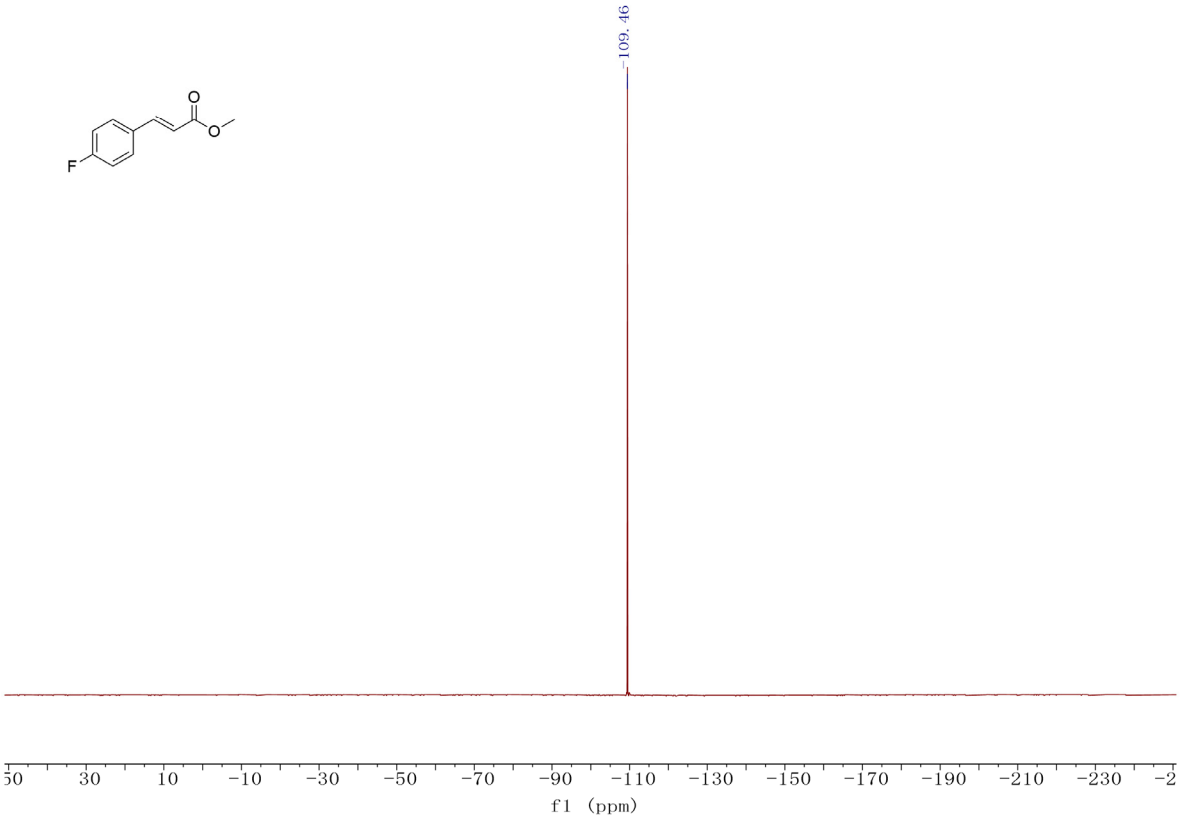


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2e**

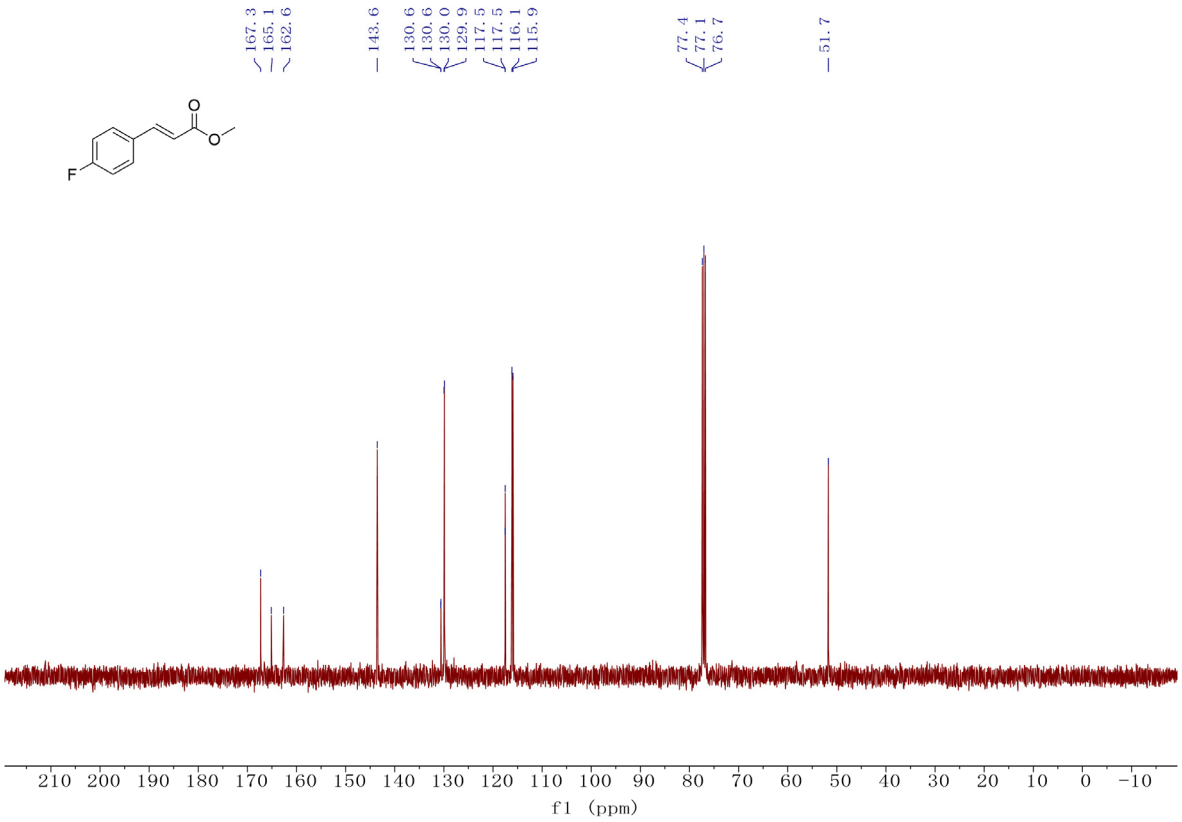
Methyl (*E*)-3-(4-fluorophenyl)acrylate (2f)



^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2f**

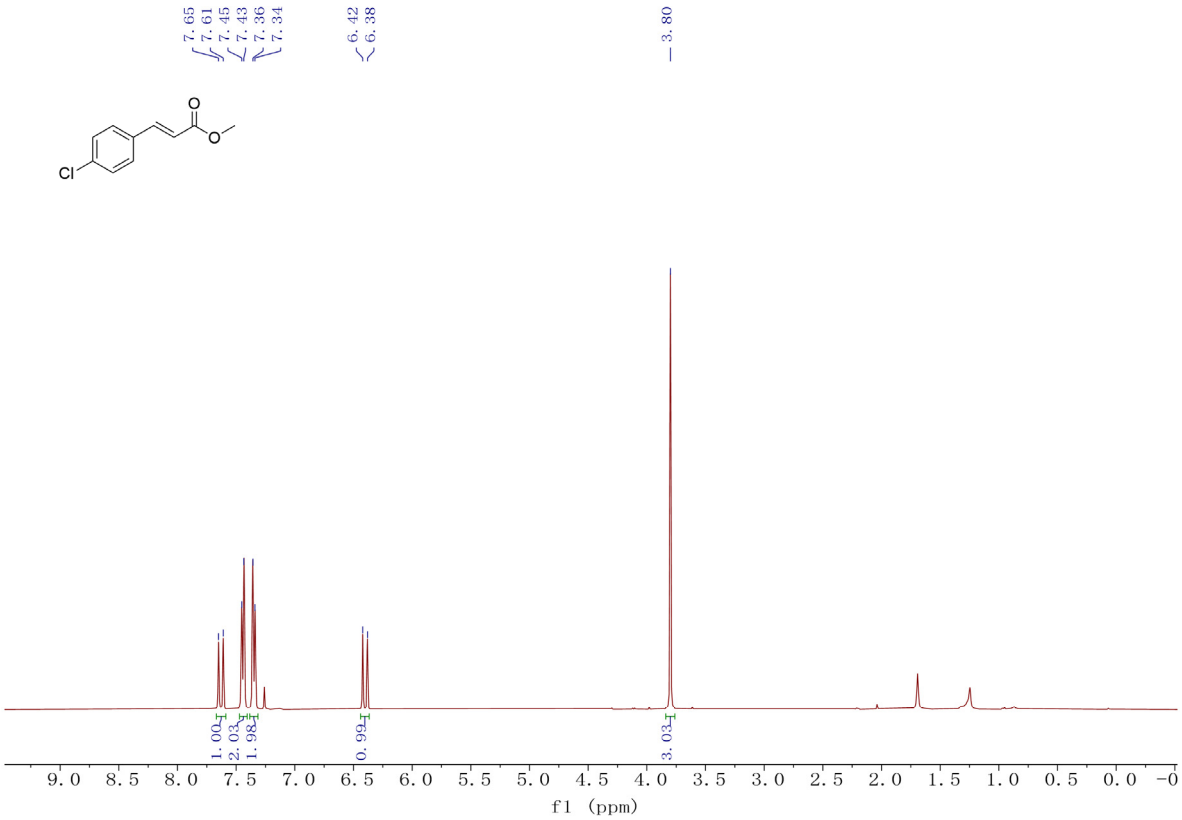


^{19}F NMR (376 MHz, $CDCl_3$) spectrum for **2f**

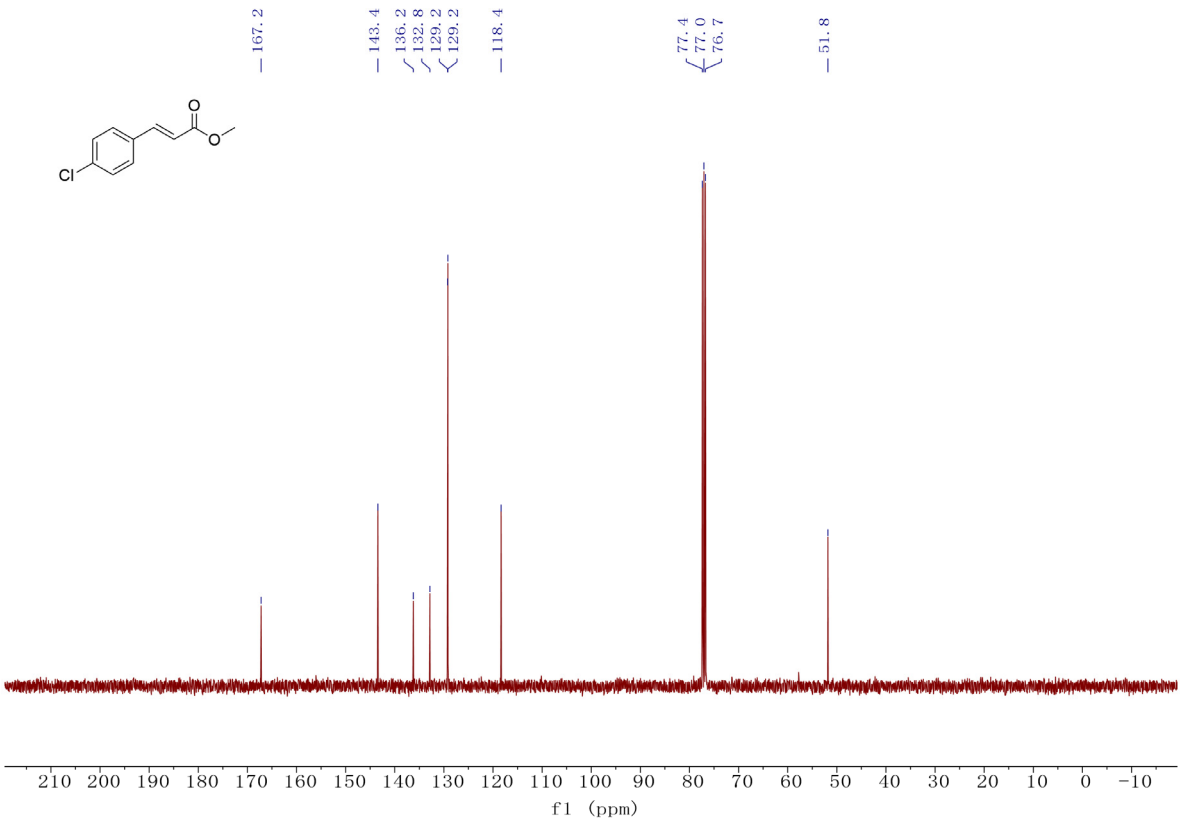


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2f**

Methyl (*E*)-3-(4-chlorophenyl)acrylate (2g)

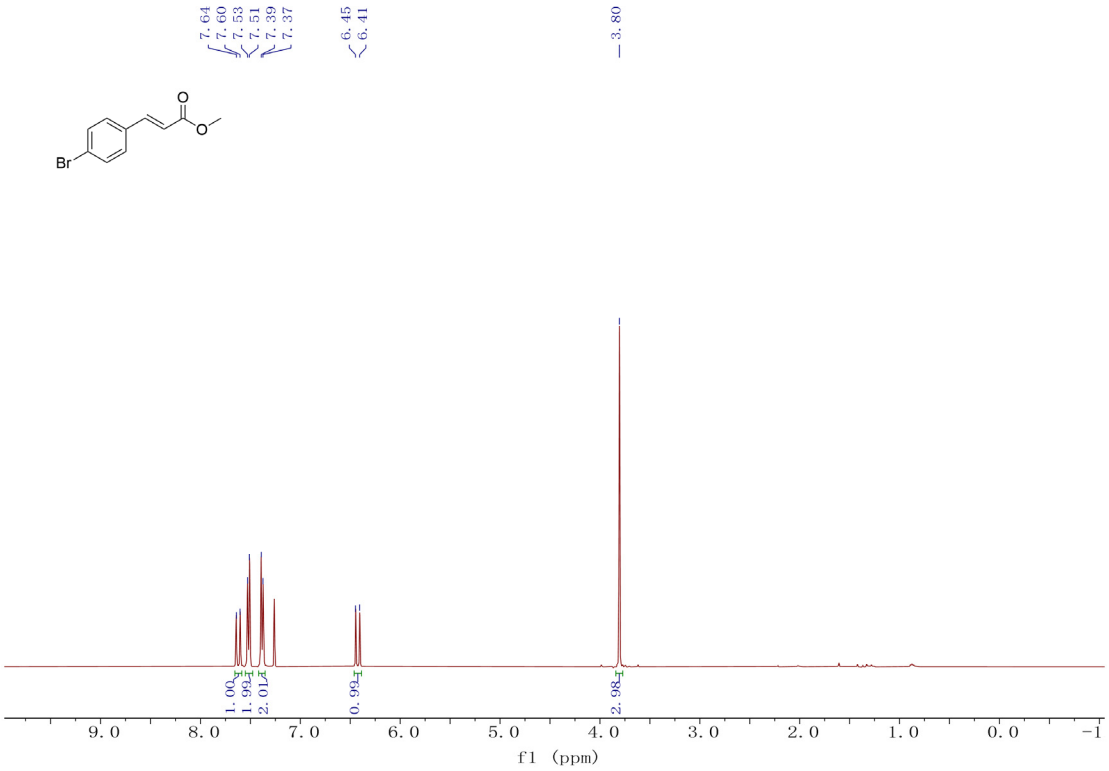


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2g**

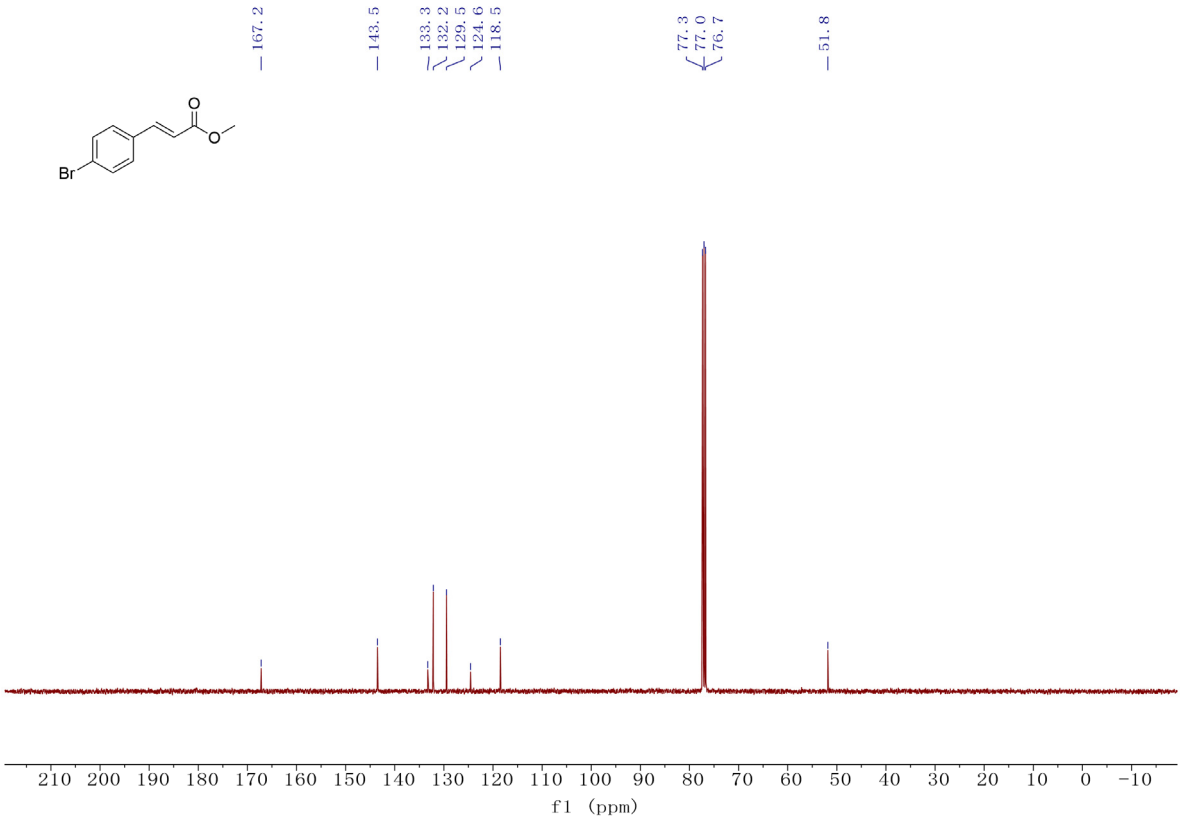


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2g**

Methyl (*E*)-3-(4-bromophenyl)acrylate (2h)

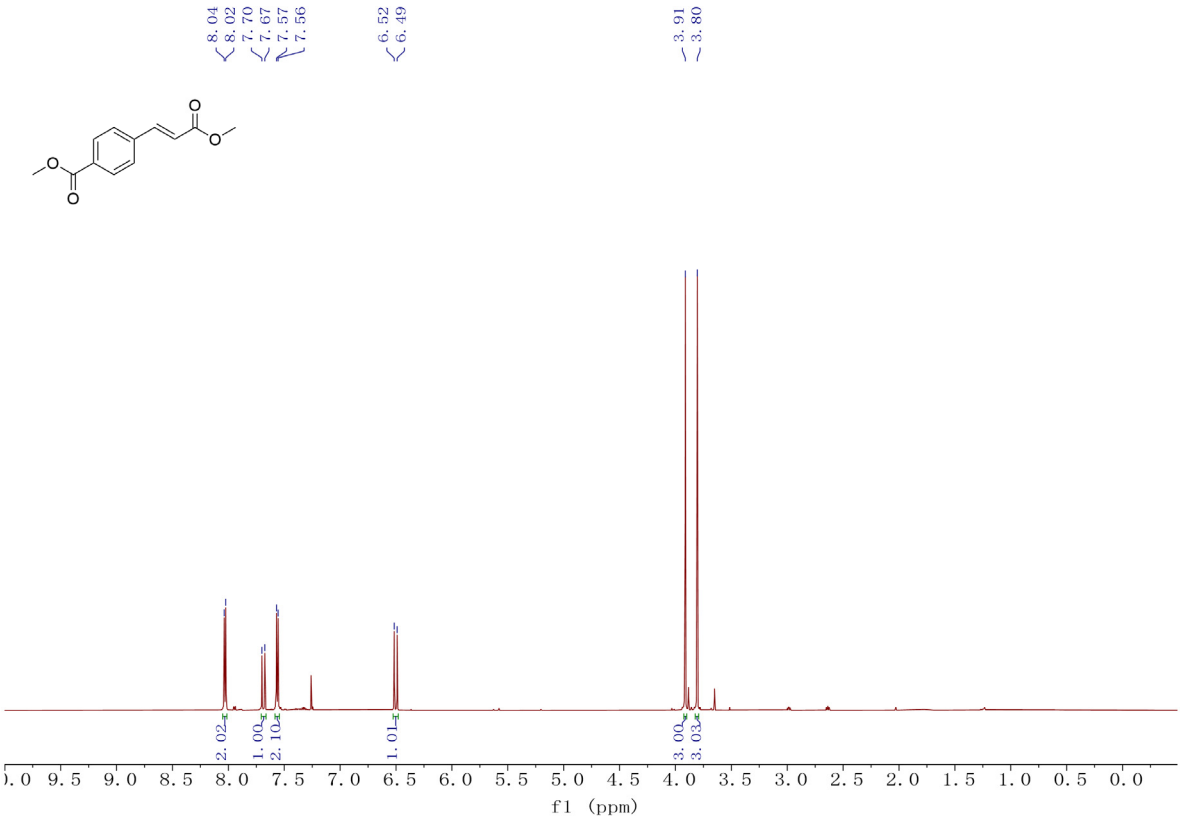


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2h**

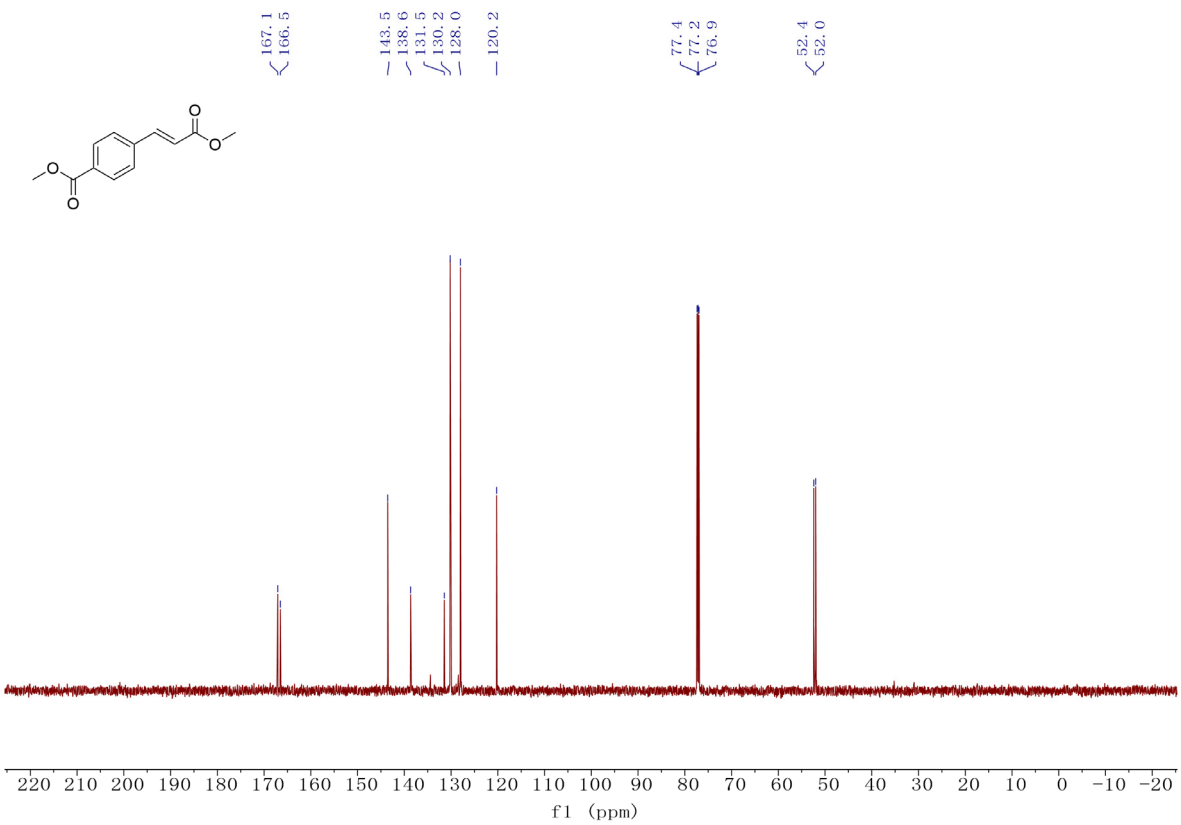


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2h**

Methyl 4-(3-methoxy-3-oxoprop-1-yn-1-yl)benzoate (2i)

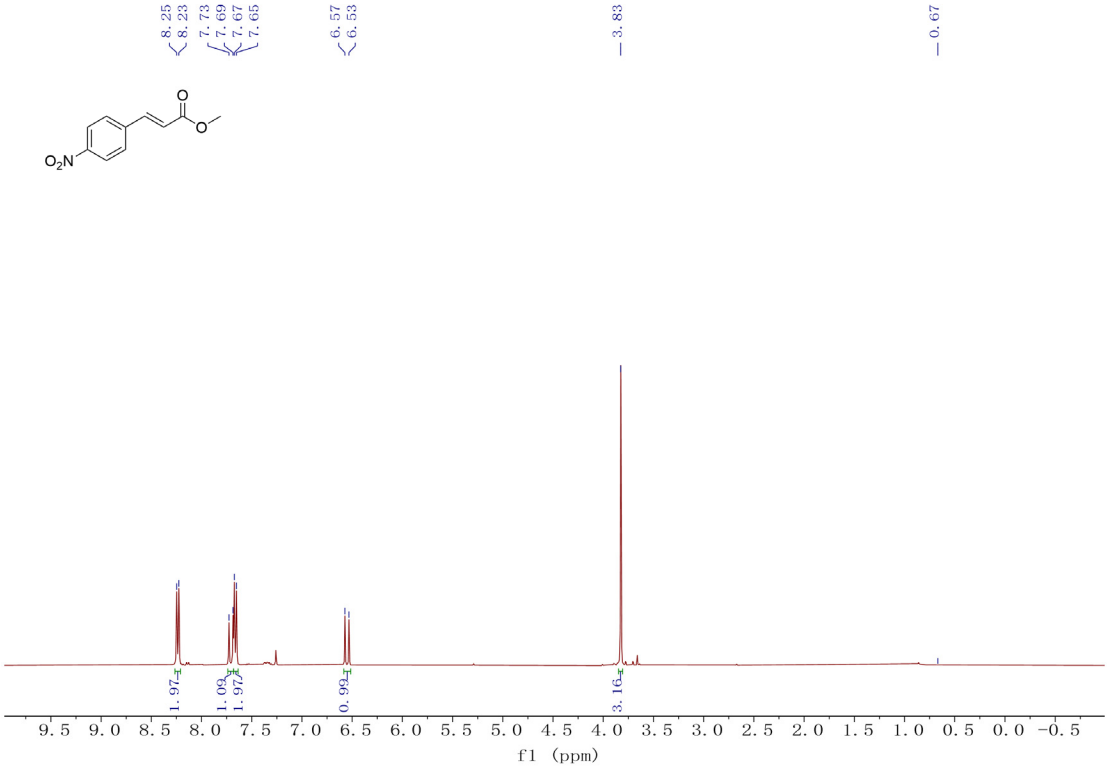


^{1}H NMR (600 MHz, $CDCl_3$) spectrum for **2i**

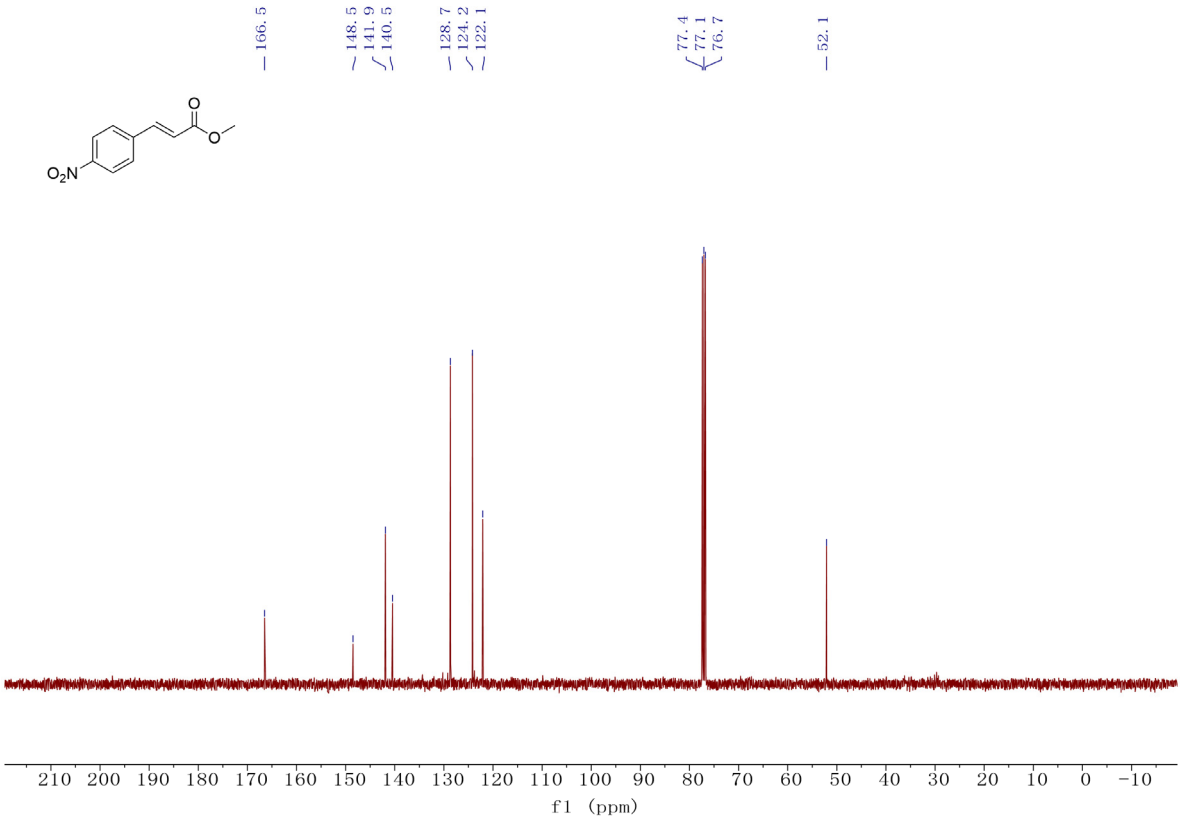


^{13}C NMR (151 MHz, $CDCl_3$) spectrum for **2i**

Methyl(*E*)-3-(4-nitrophenyl)acrylate (2j)

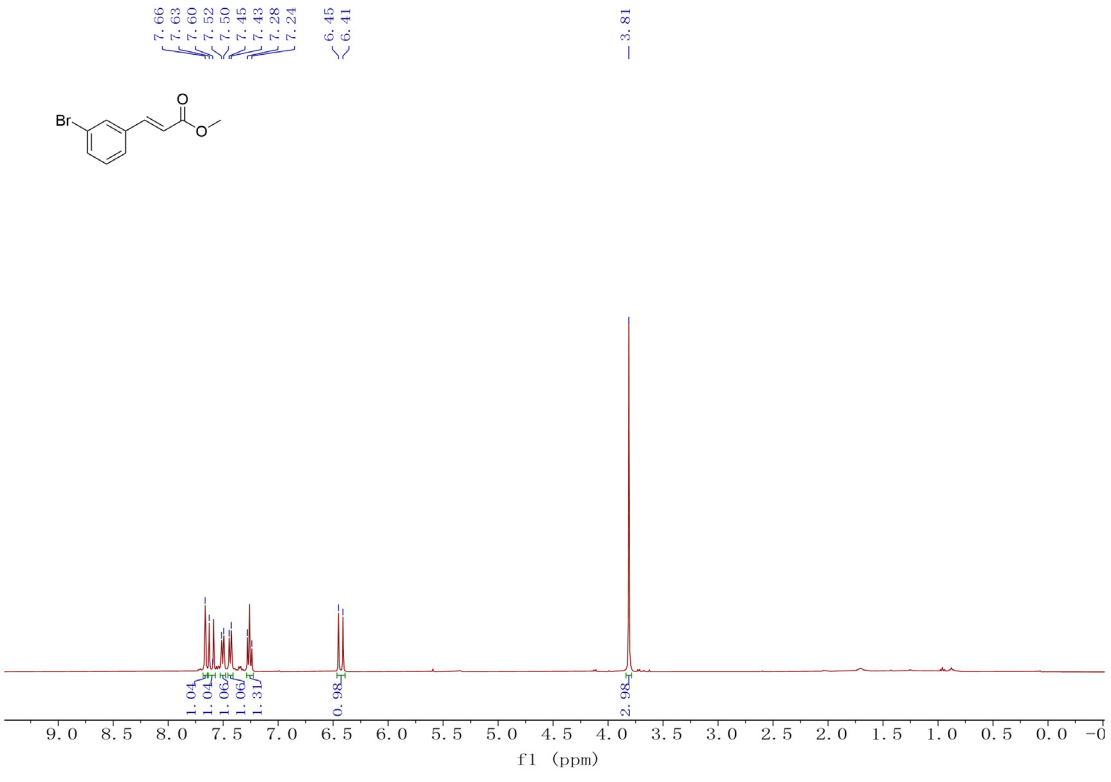


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2j**



^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2j**

Methyl (*E*)-3-(3-bromophenyl)acrylate (2k)



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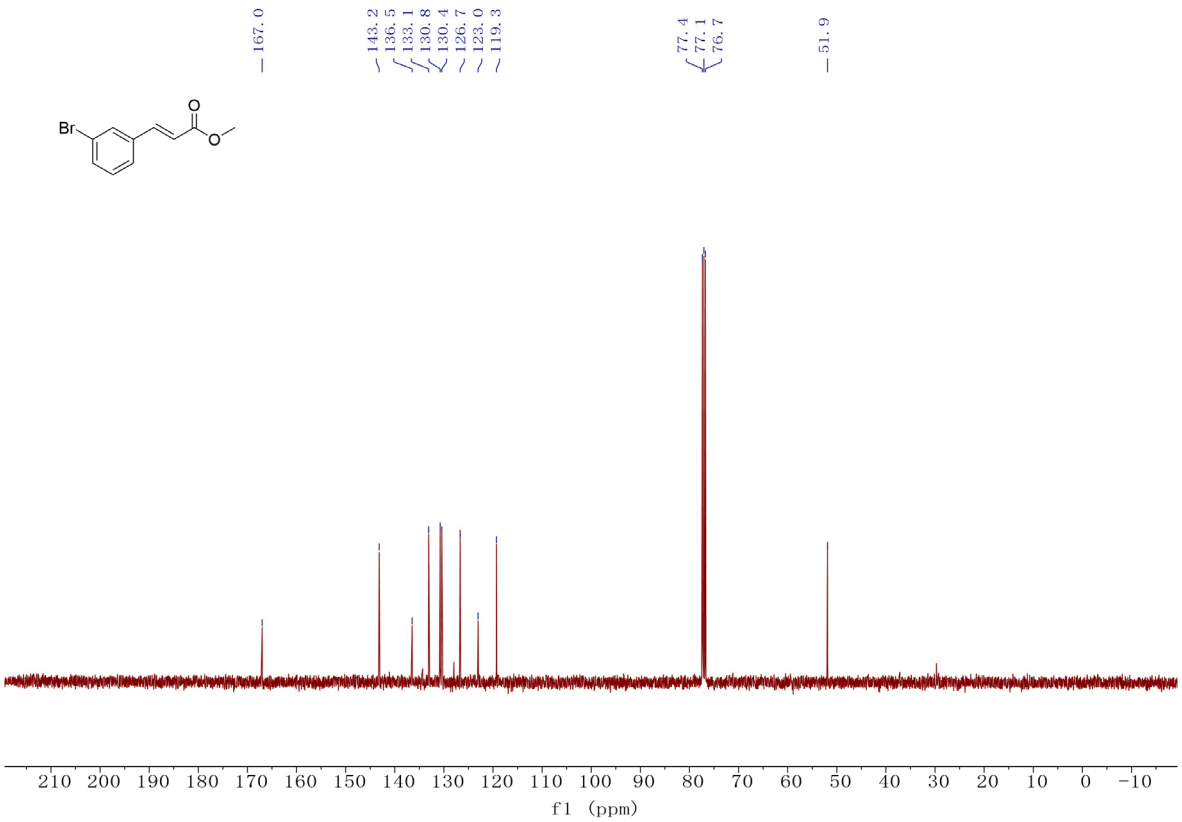
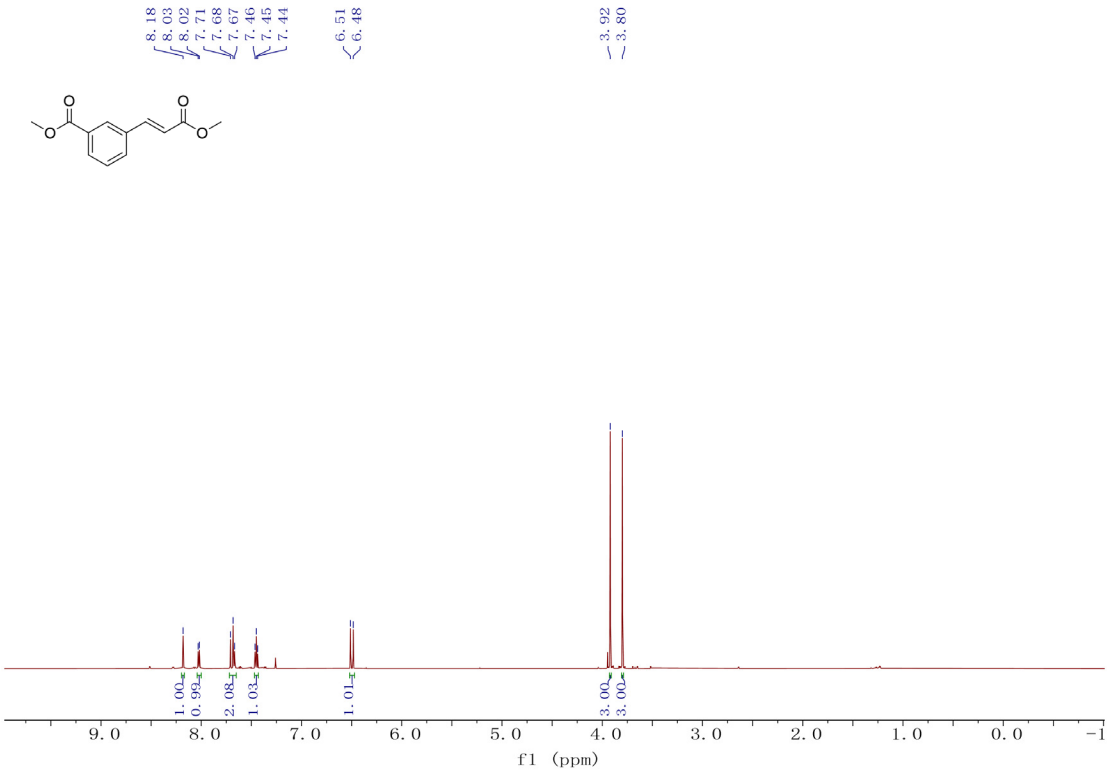
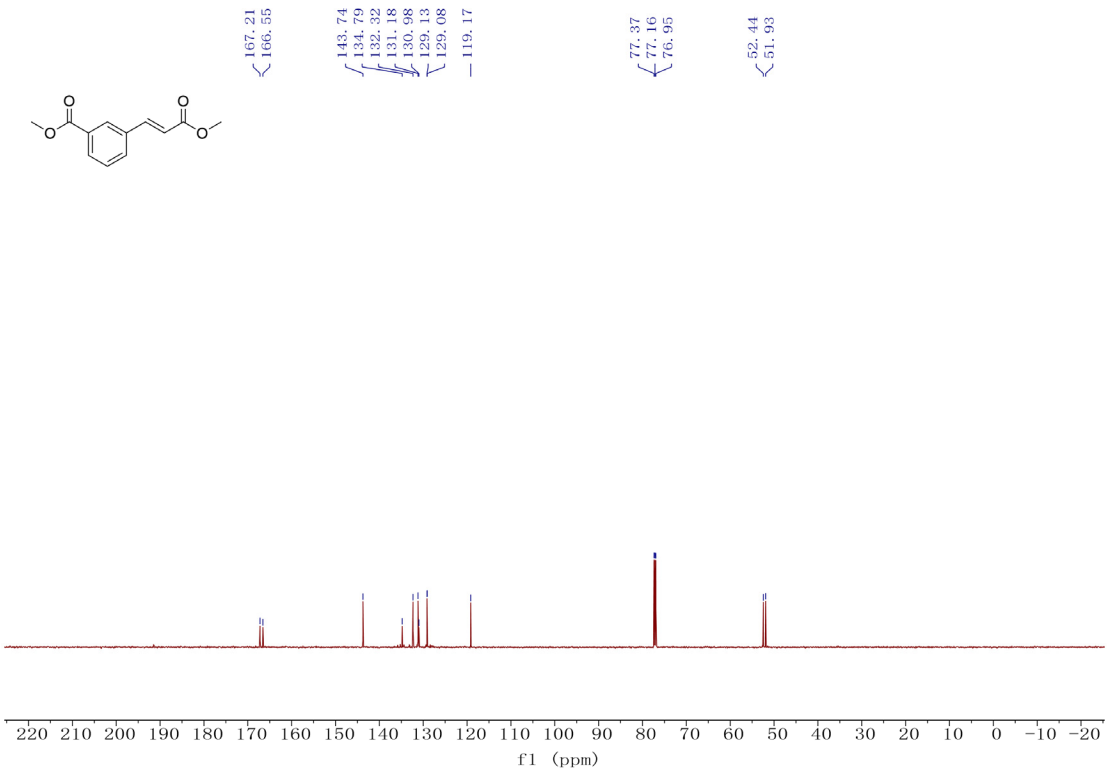


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2k**

Methyl (*E*)-3-(3-methoxy-3-oxoprop-1-en-1-yl)benzoate (2l)

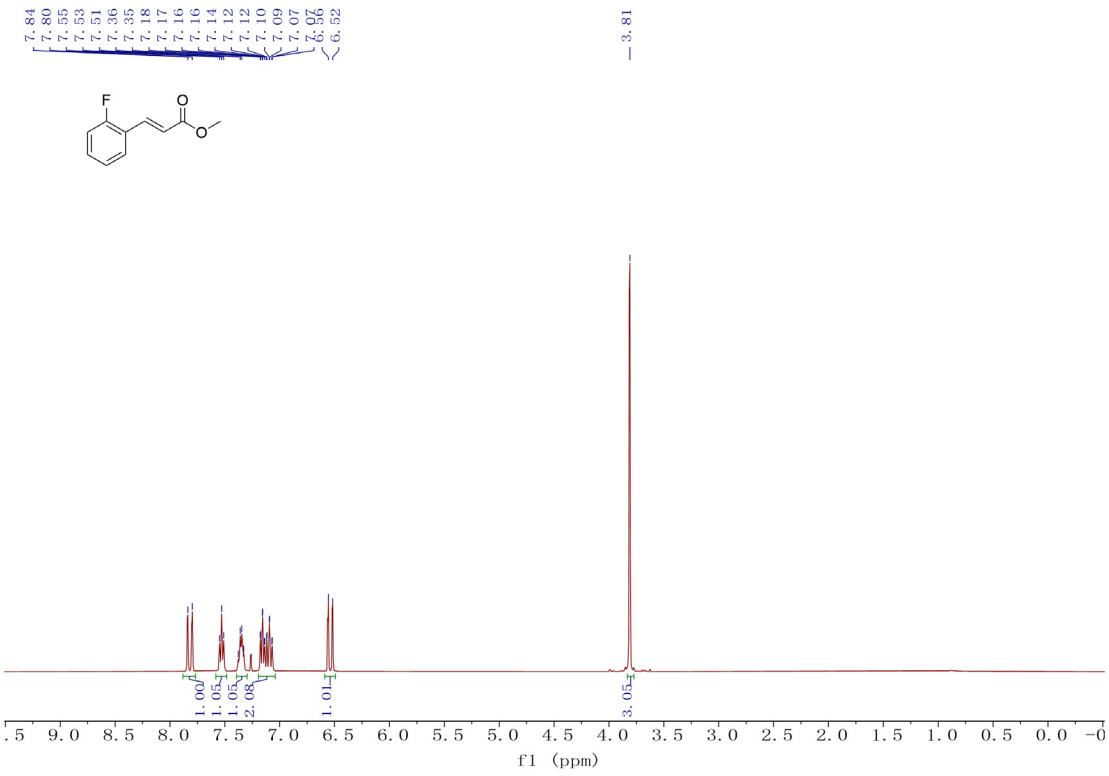


^{1}H NMR (600 MHz, $CDCl_3$) spectrum for **2l**



^{13}C NMR (151 MHz, $CDCl_3$) spectrum for **2l**

Methyl 3-(2-fluorophenyl)acrylate (2m)



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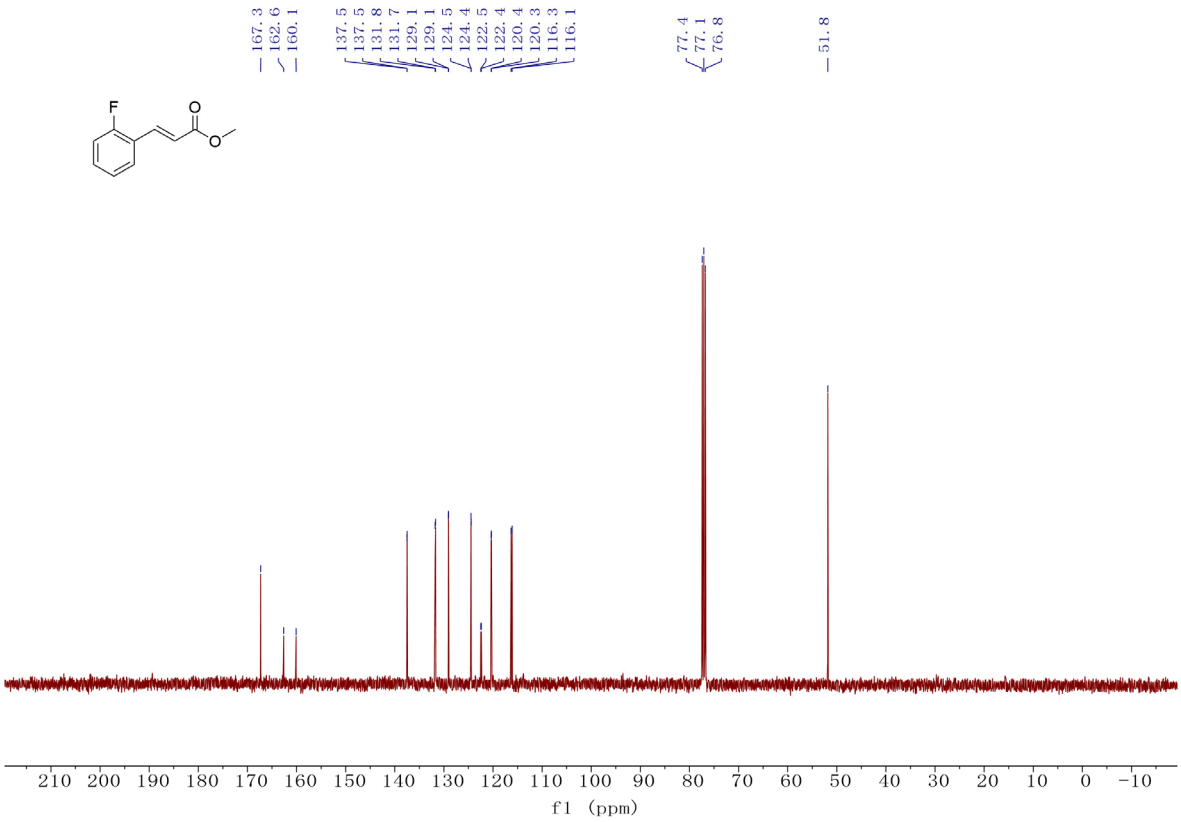

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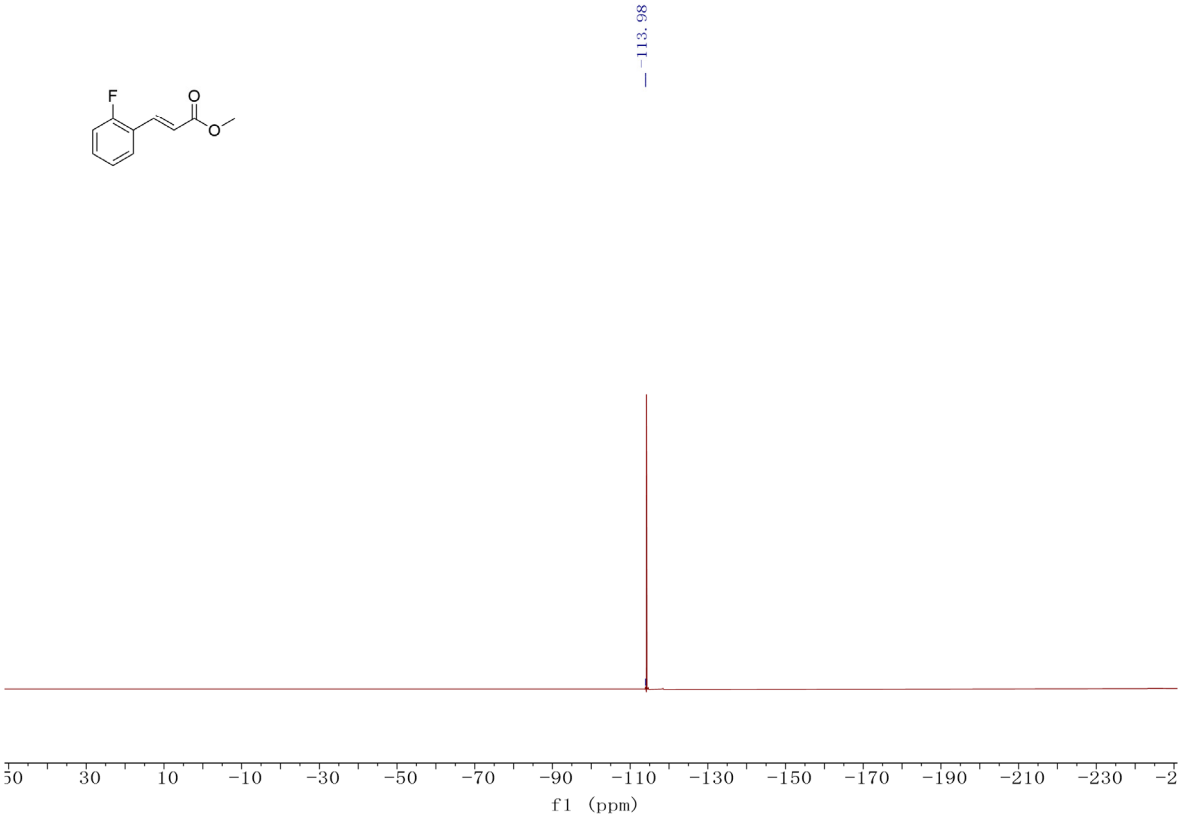
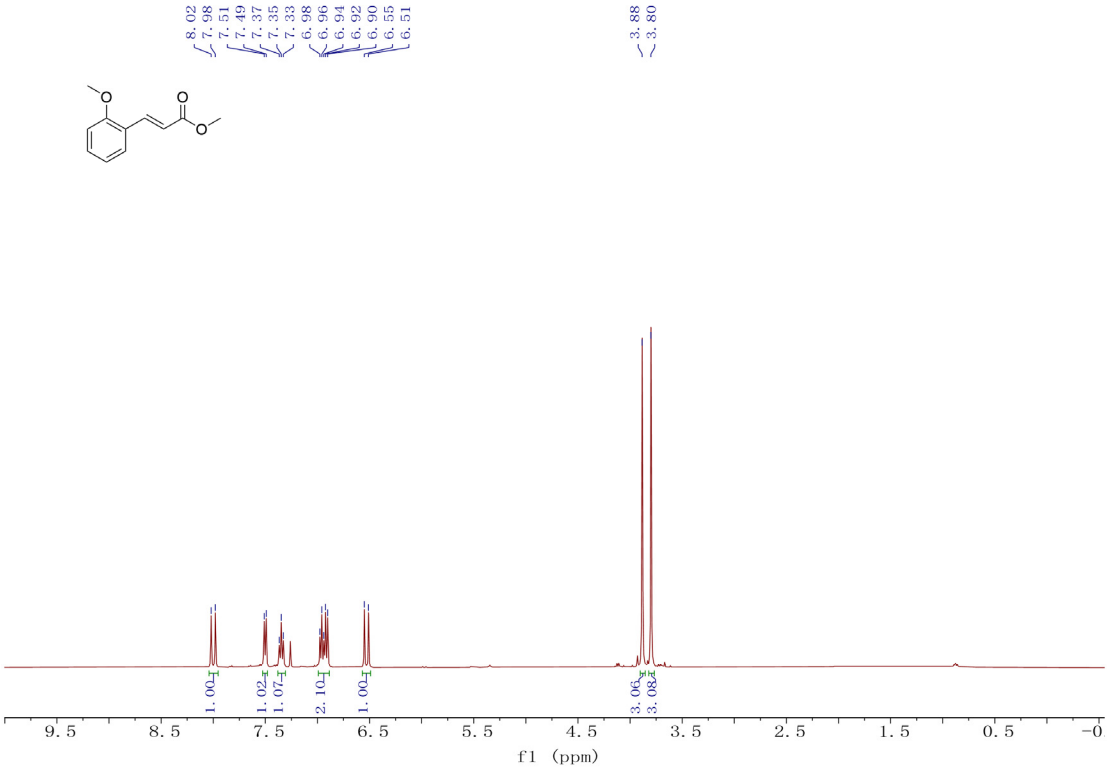


^{19}F NMR (376 MHz, $CDCl_3$) spectrum for **2m**

Ethyl (*E*)-3-(2-methoxyphenyl)acrylate (2n)



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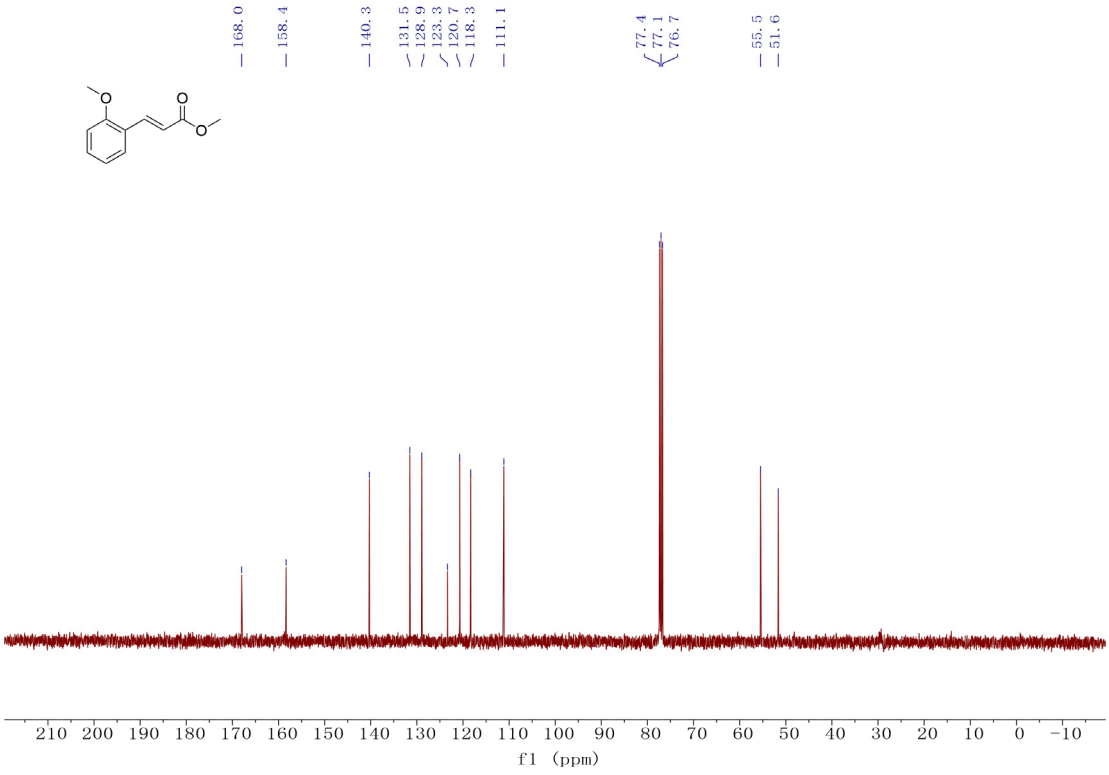
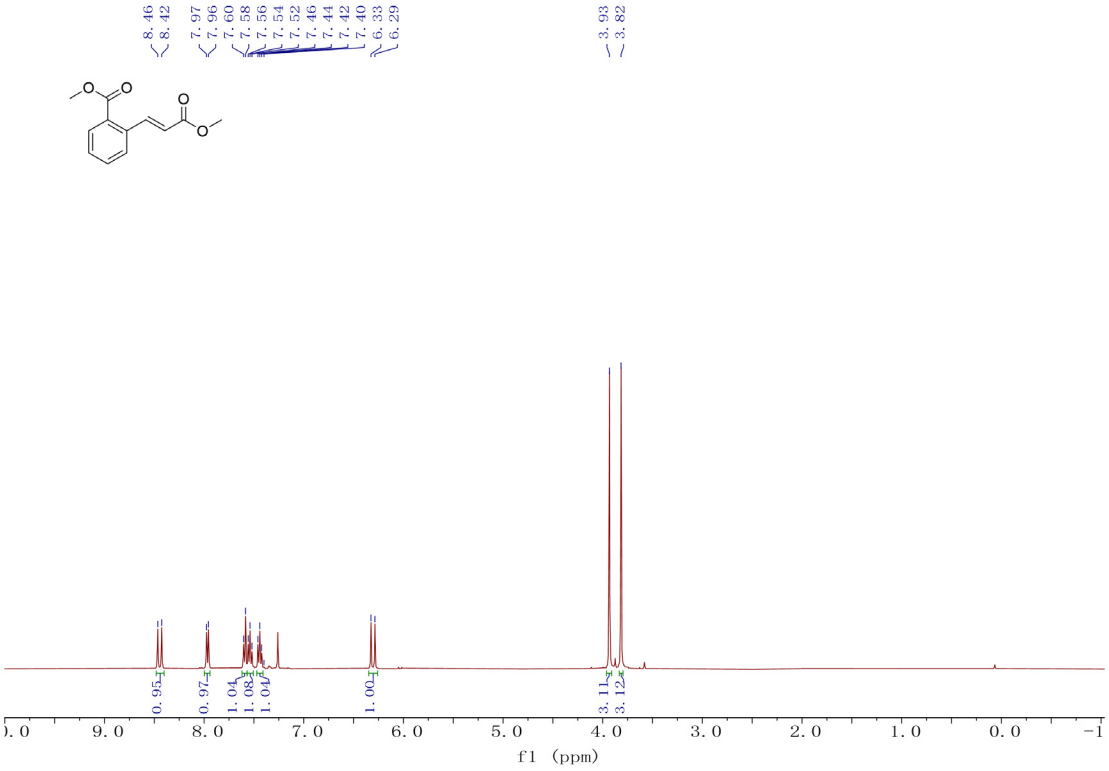


^{13}C NMR (400 MHz, $CDCl_3$) spectrum for **2n**

Methyl (*E*)-2-(3-methoxy-3-oxoprop-1-en-1-yl)benzoate (2o)



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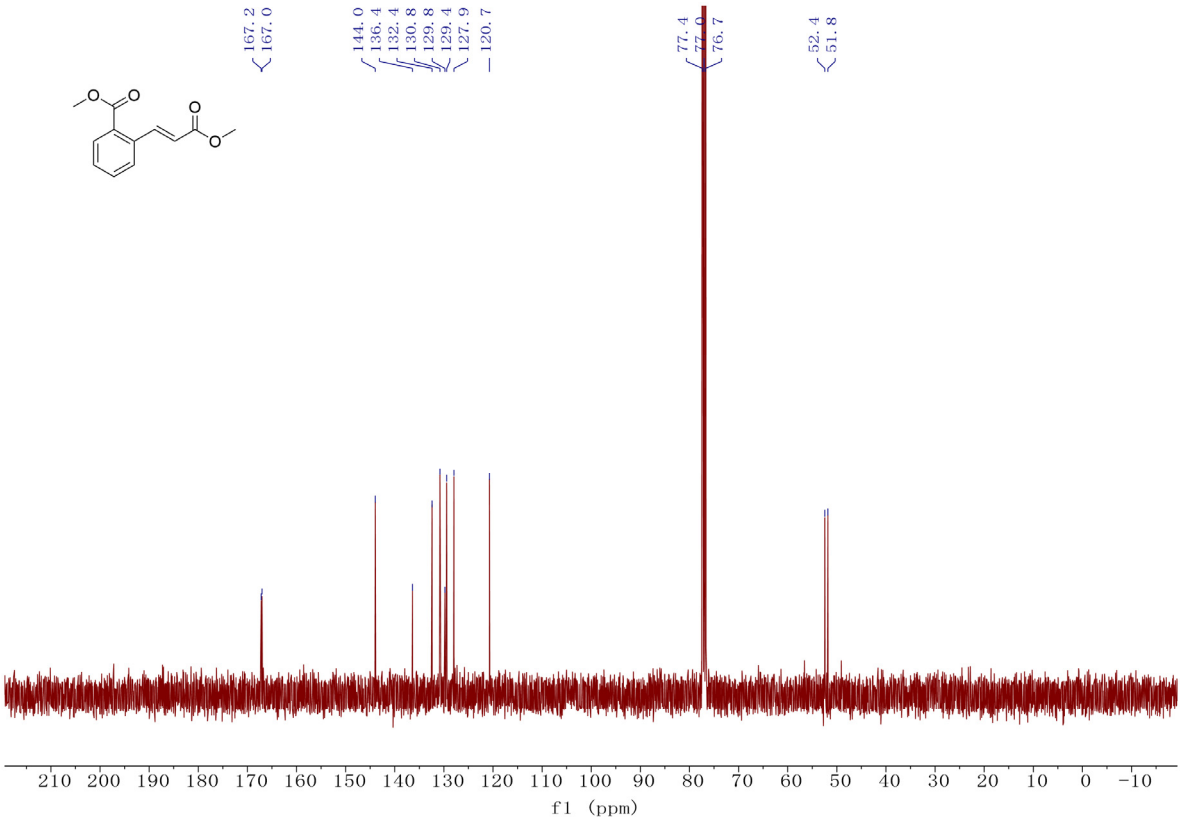

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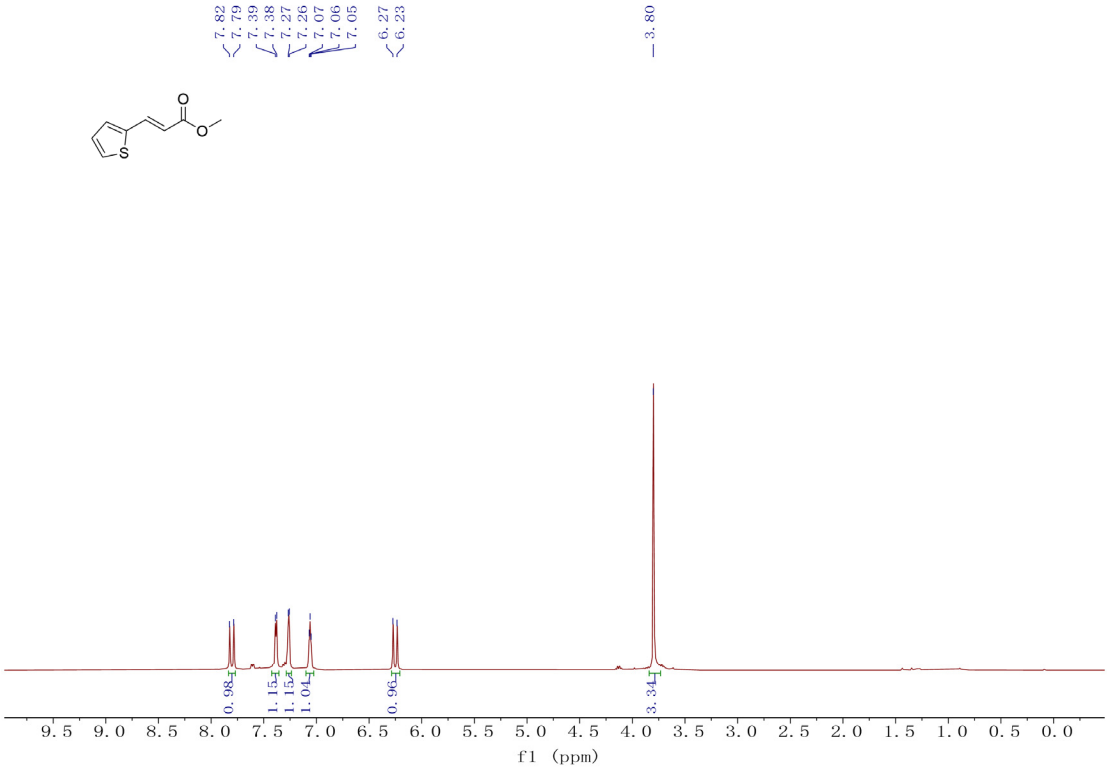

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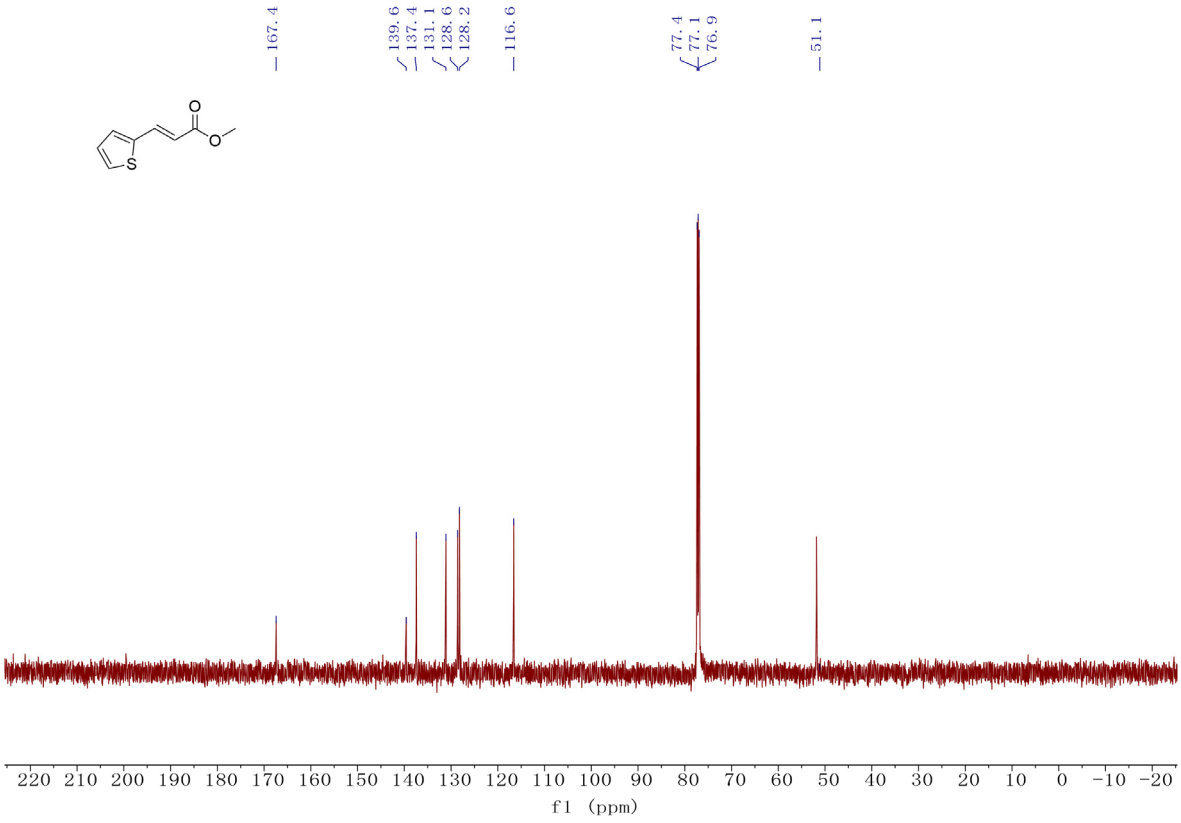
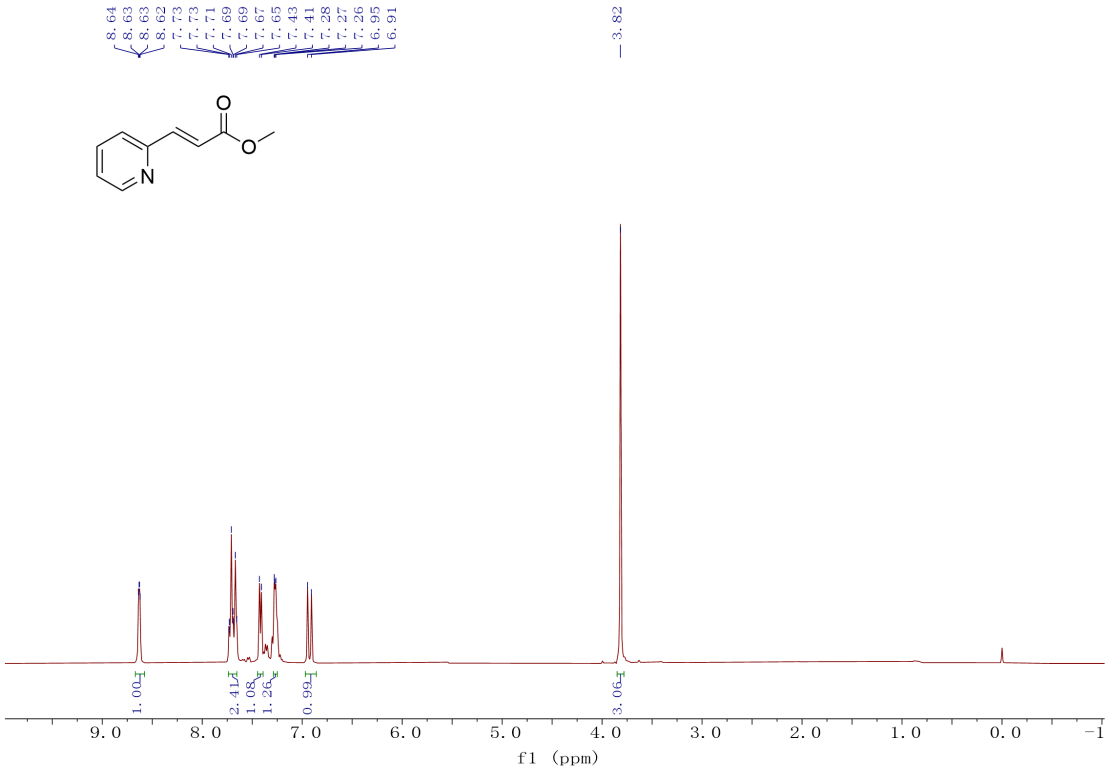
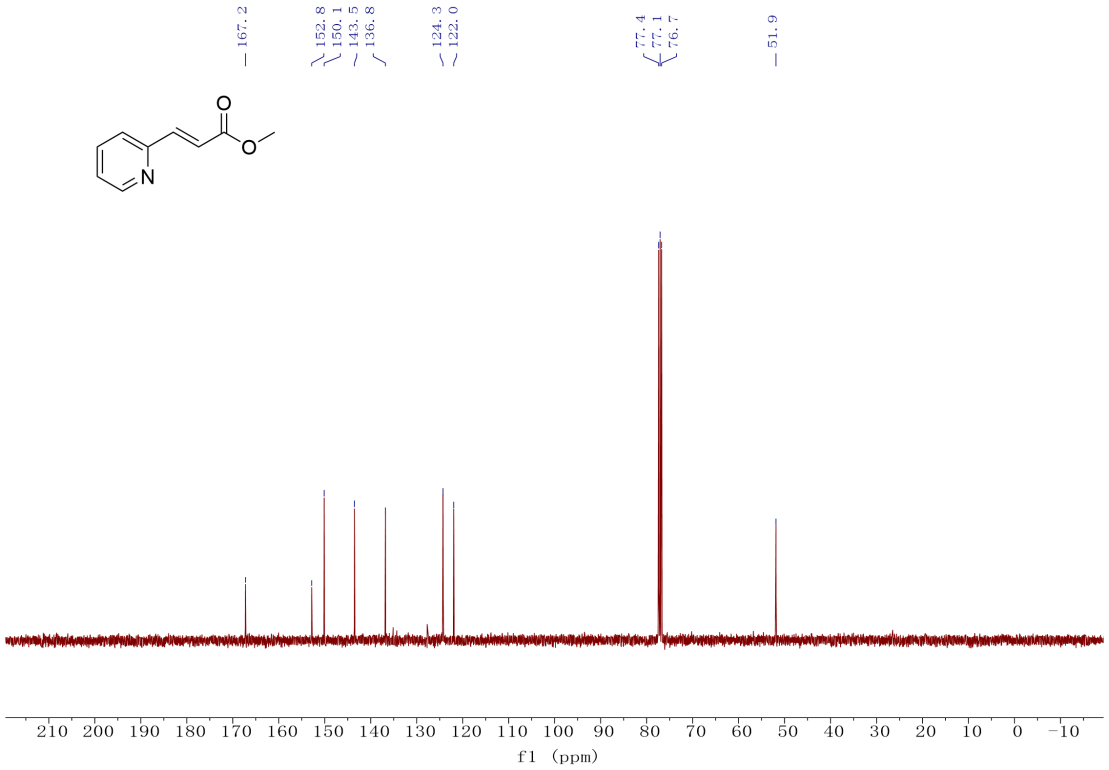


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2p**

(*E*)-methyl-3-(pyridin-2-yl)acrylate (2q)

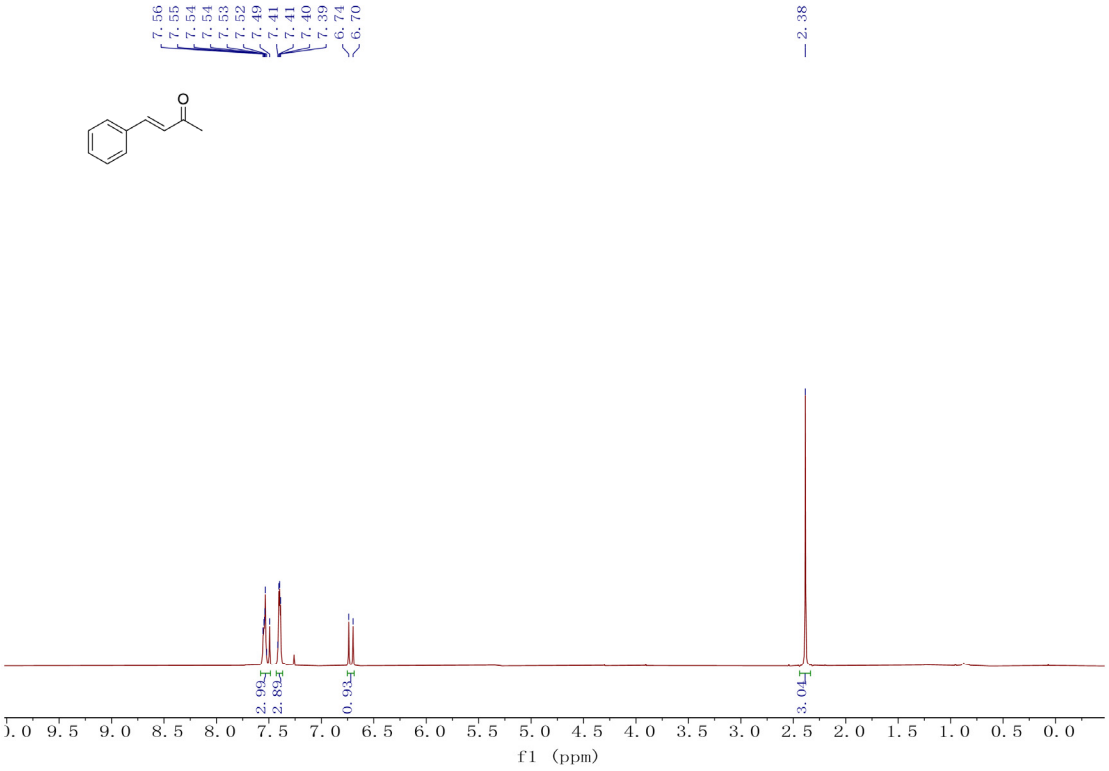


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2q**

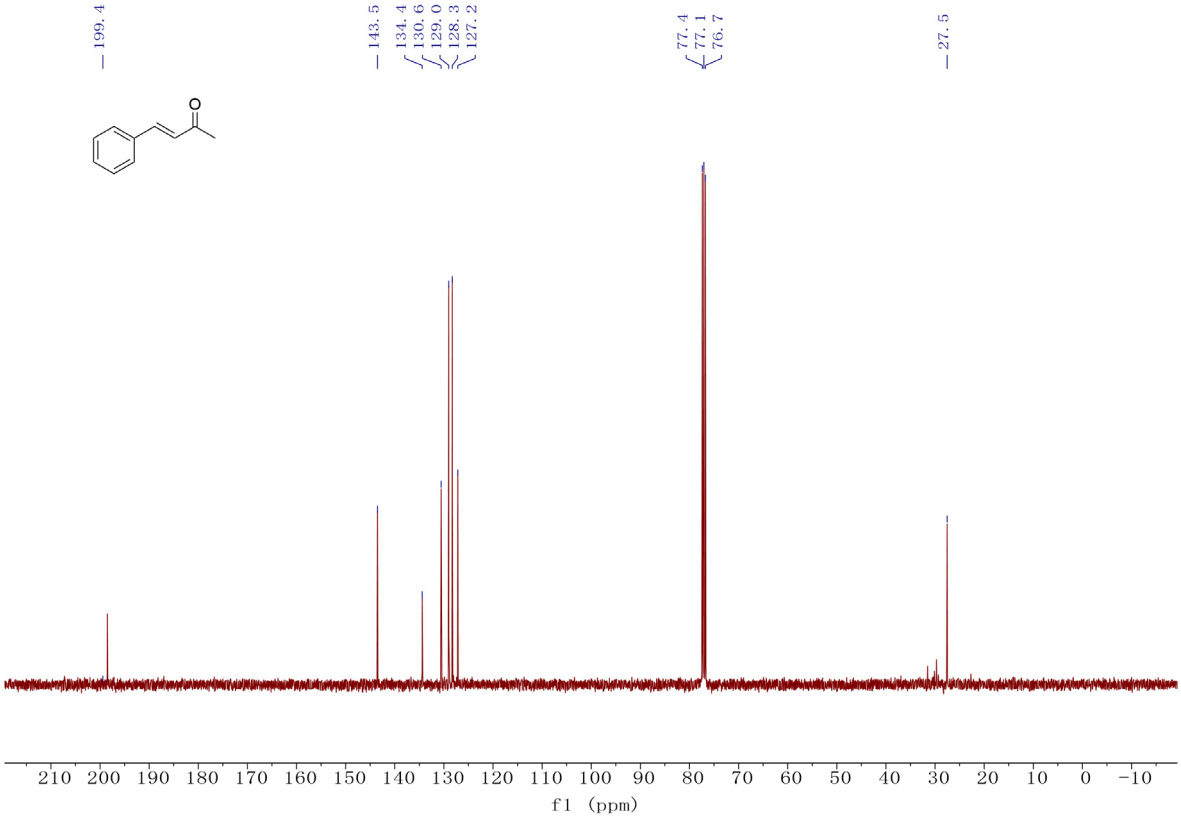


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2q**

(E)-4-phenylbut-3-en-2-one (2r)

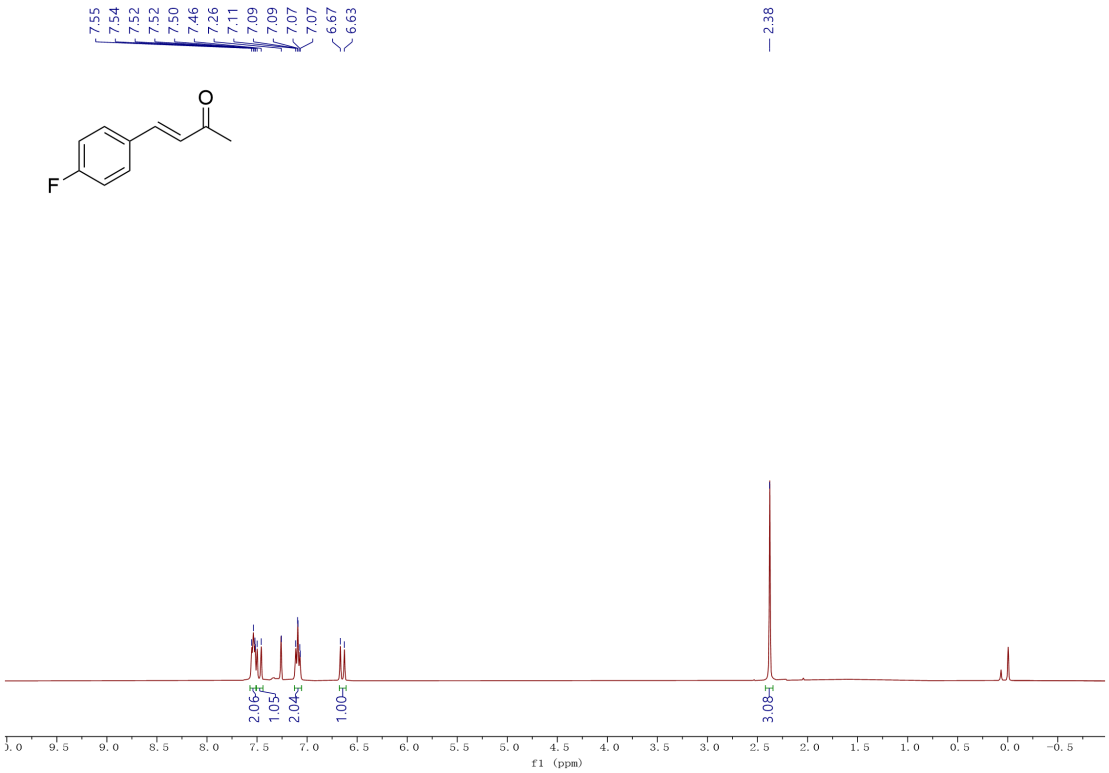


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2r**

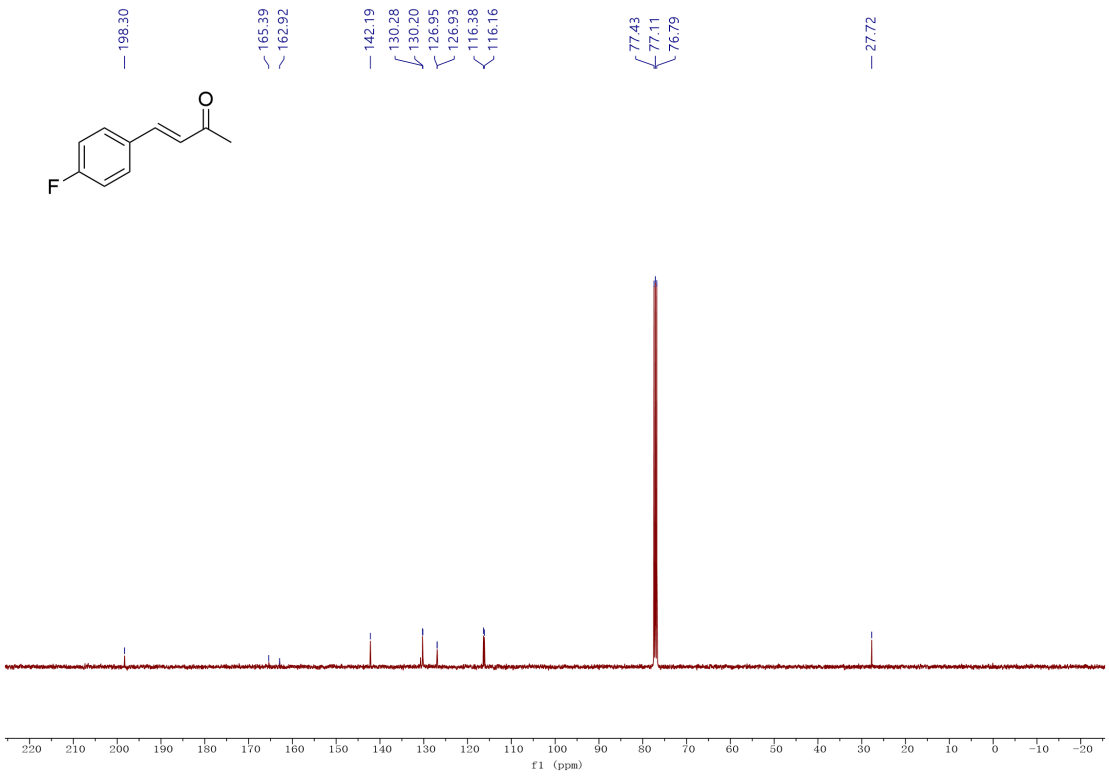


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2r**

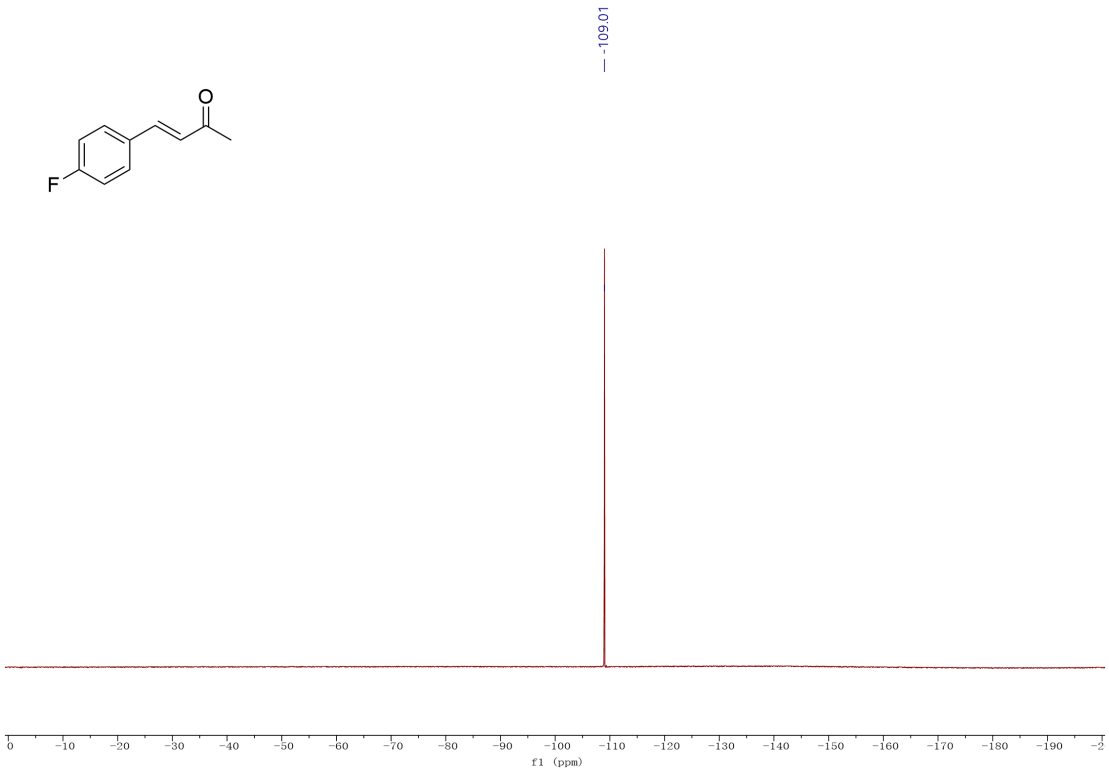
(*E*)-4-(4-fluorophenyl)but-3-en-2-one (2s)



^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2s**

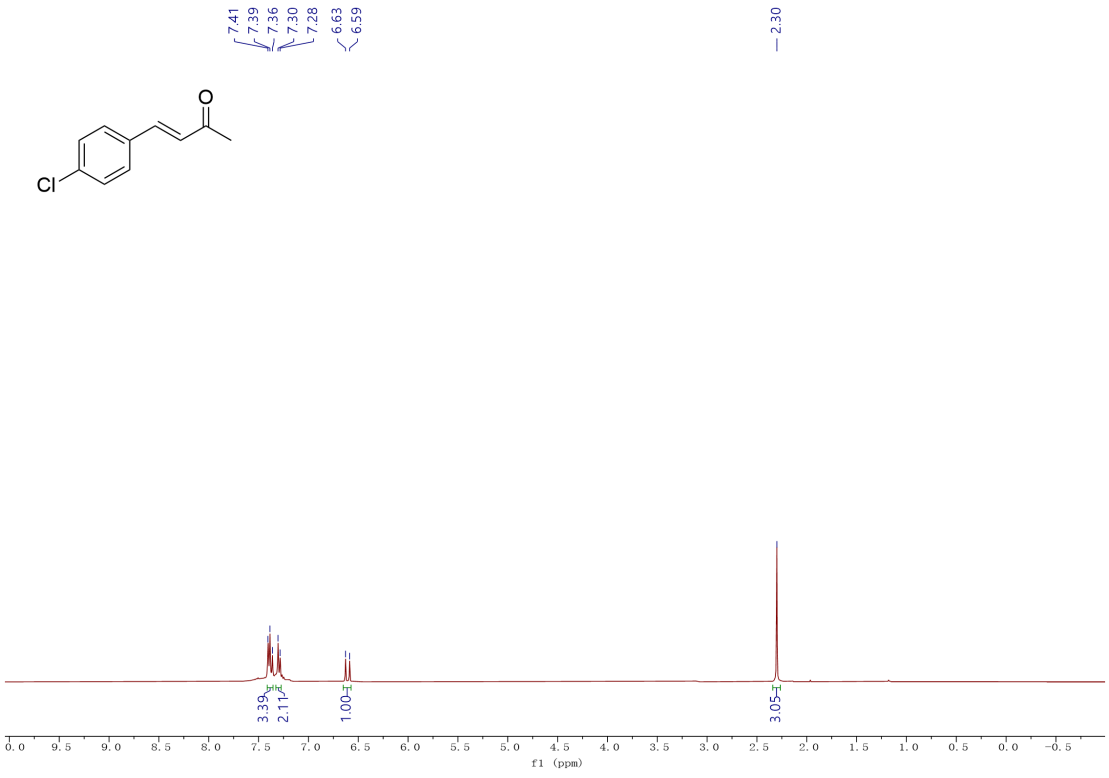


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2s**

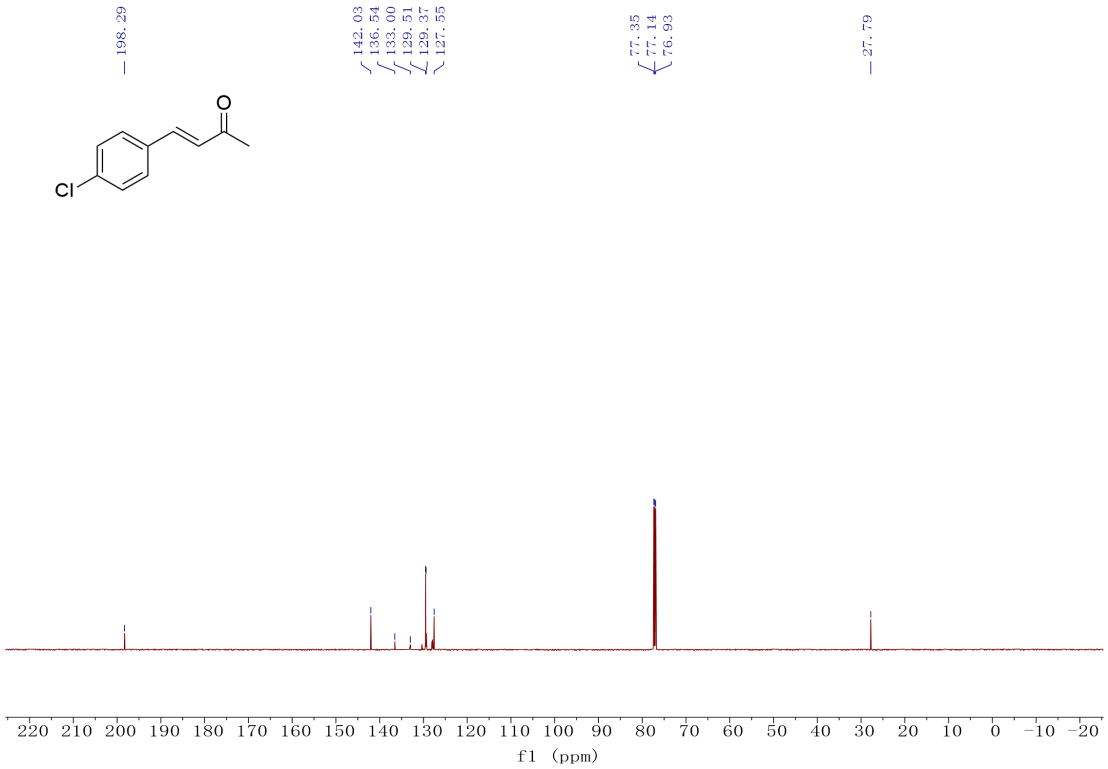


^{19}F NMR (565 MHz, $CDCl_3$) spectrum for **2s**

(*E*)-4-(4-Chlorophenyl)but-3-en-2-one (2t)

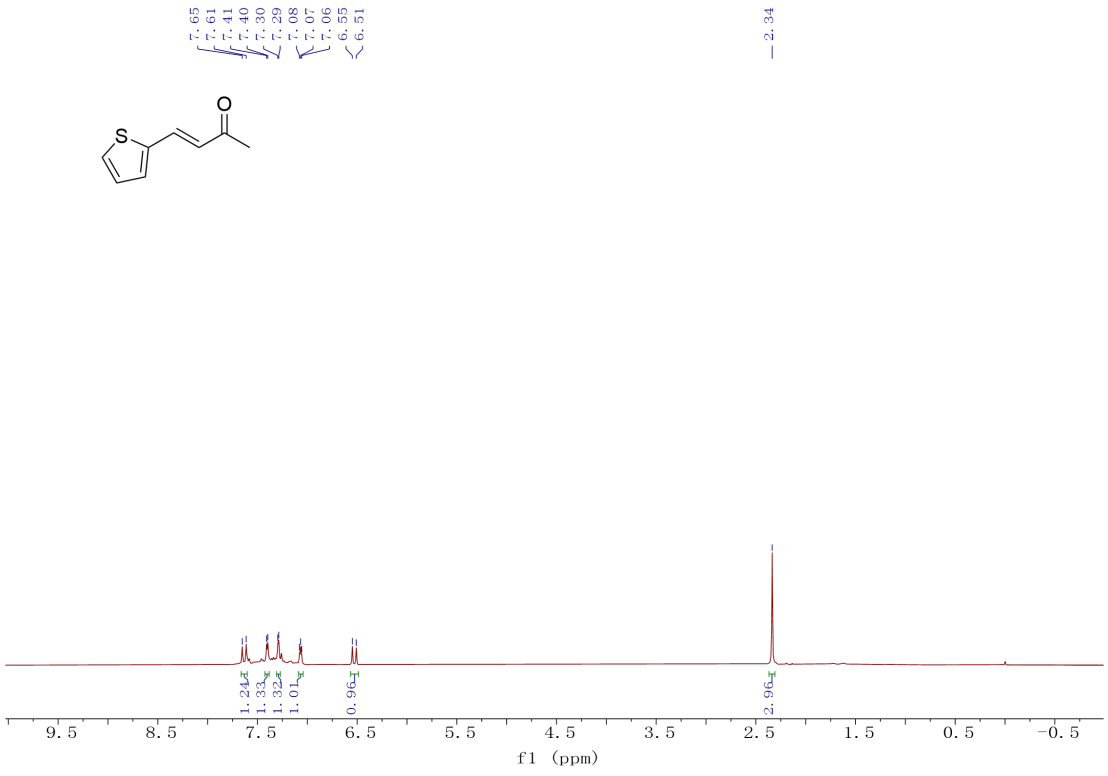


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2t**

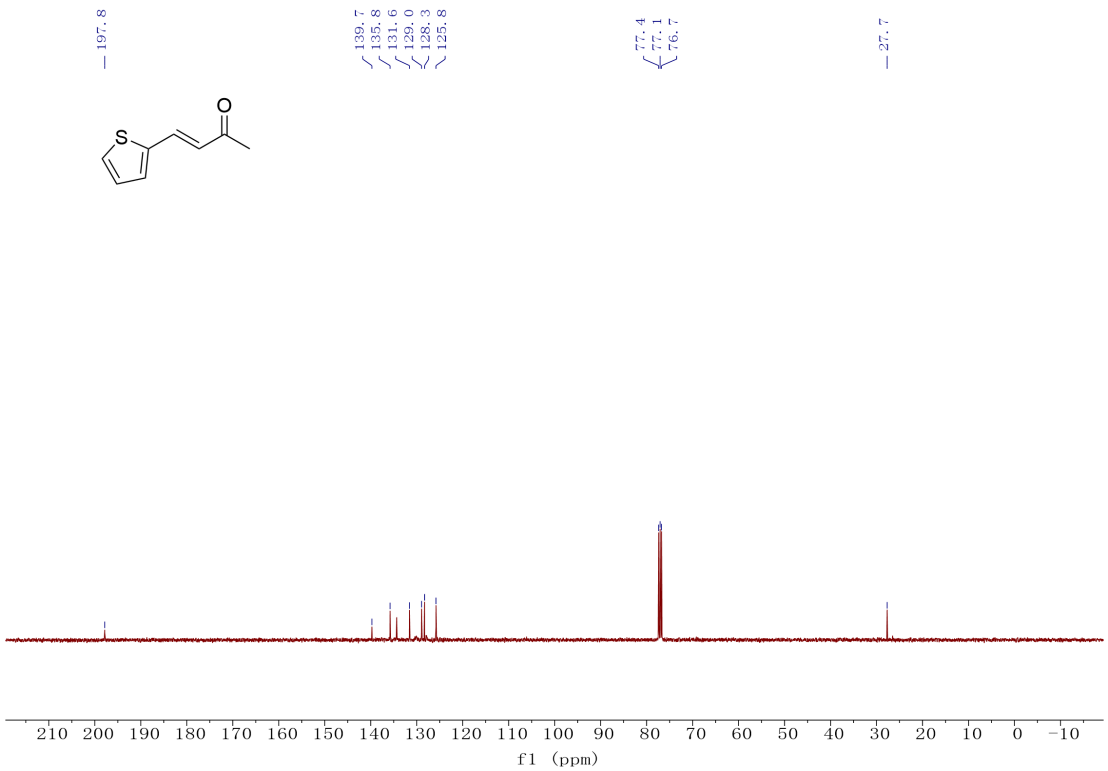


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2t**

(*E*)-4-(2-thienyl)but-3-en-2-one (2u)

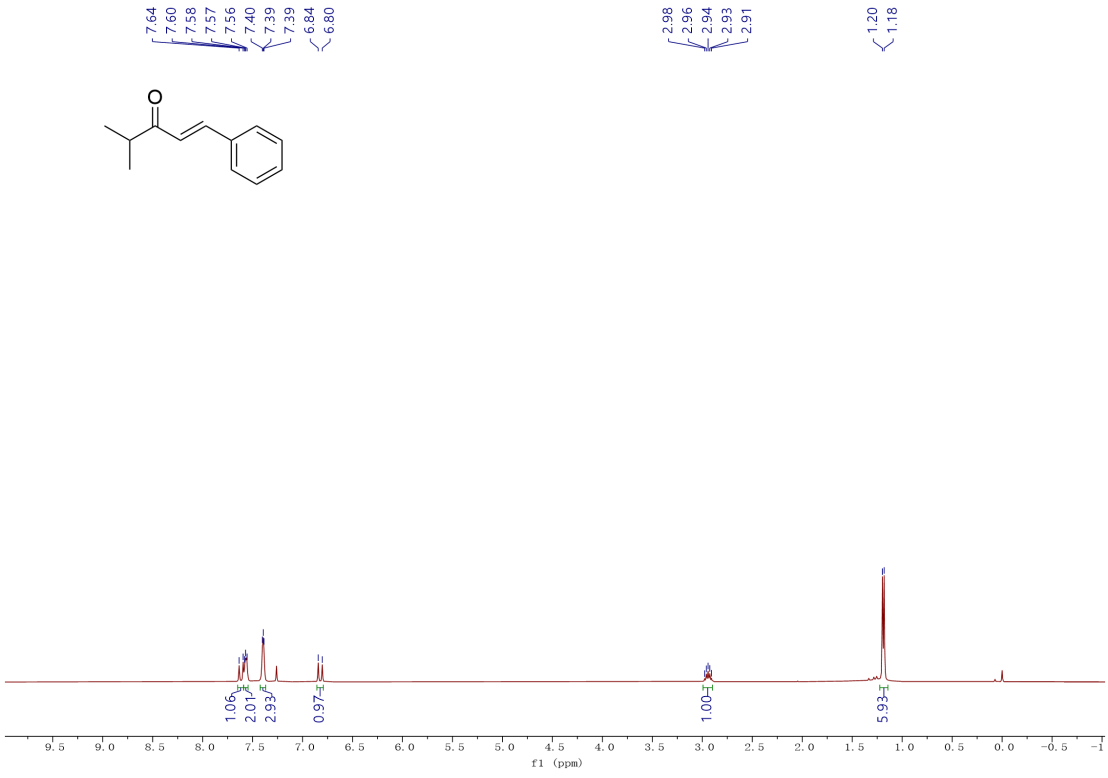


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2u**

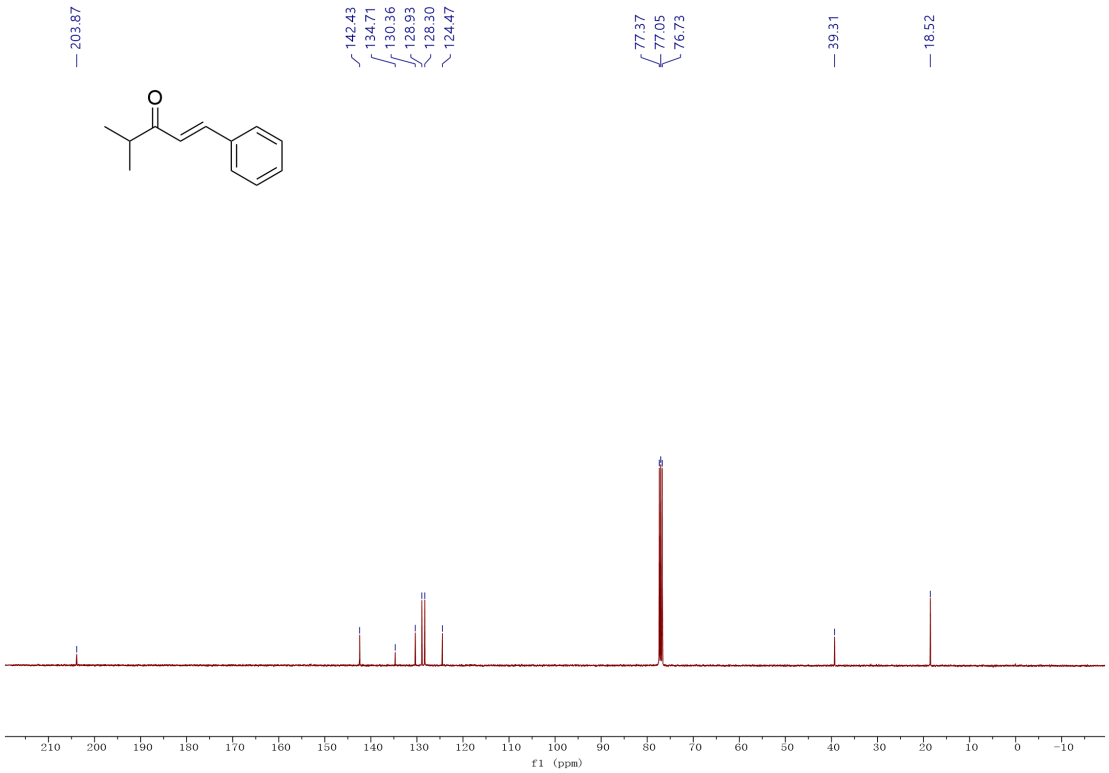


^{13}C NMR ($CDCl_3$) spectrum for **2u**

(*E*)-1-cyclopropyl-3-phenylpropen-1-one (2v)

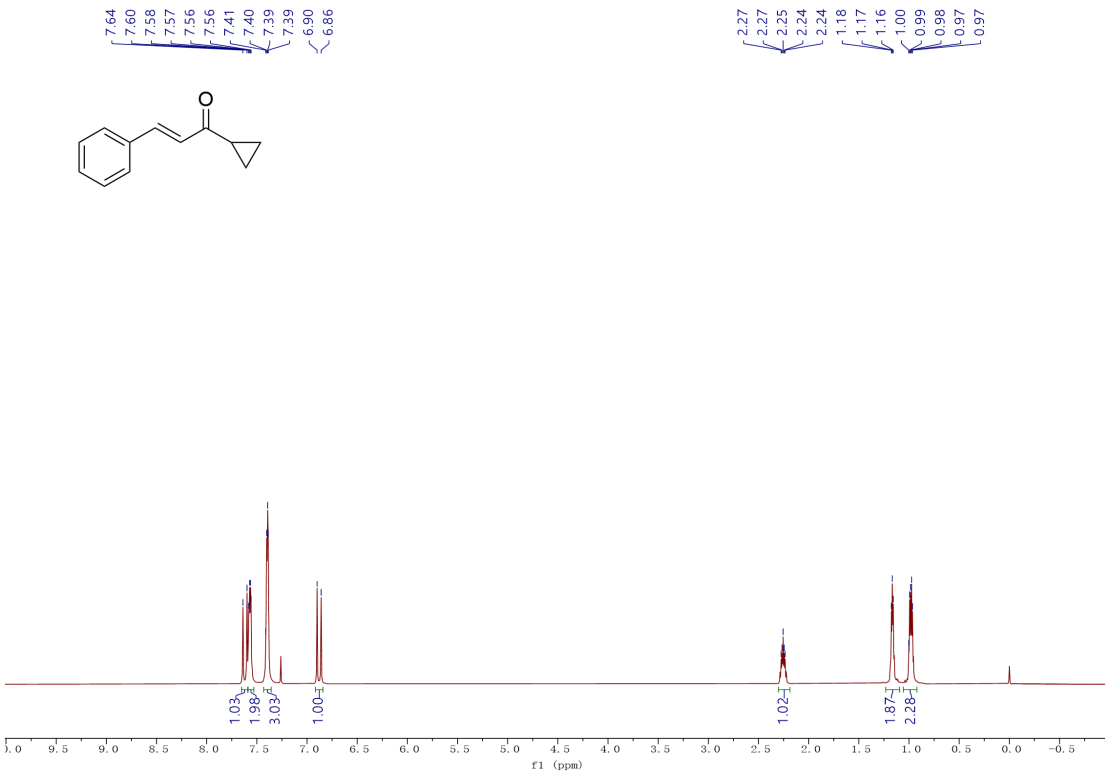


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2v**

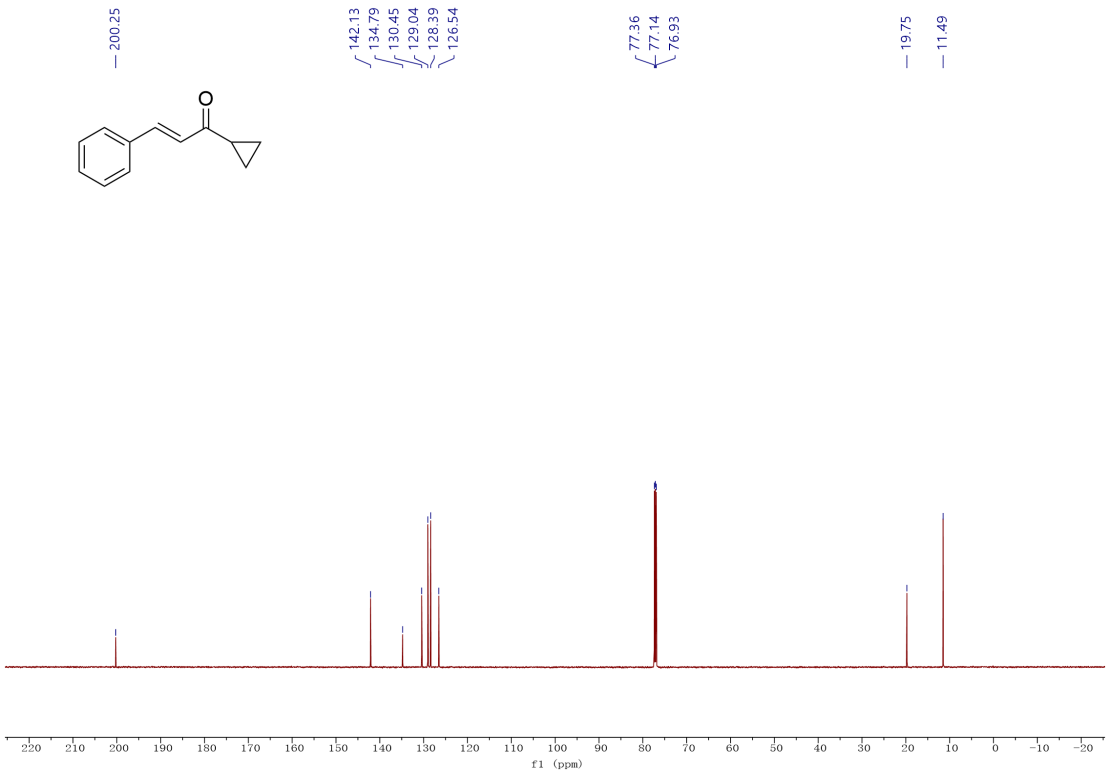


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2v**

(*E*)-1-cyclopropyl-3-phenylprop-2-en-1-one(2w)

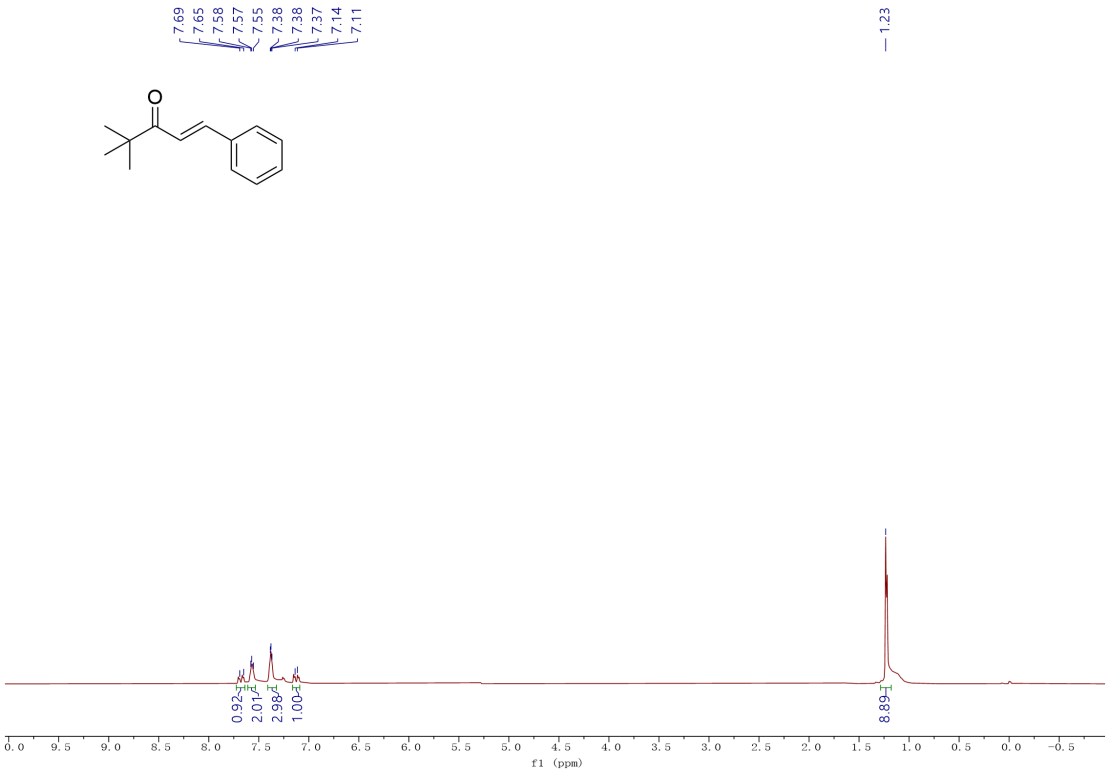


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2w**

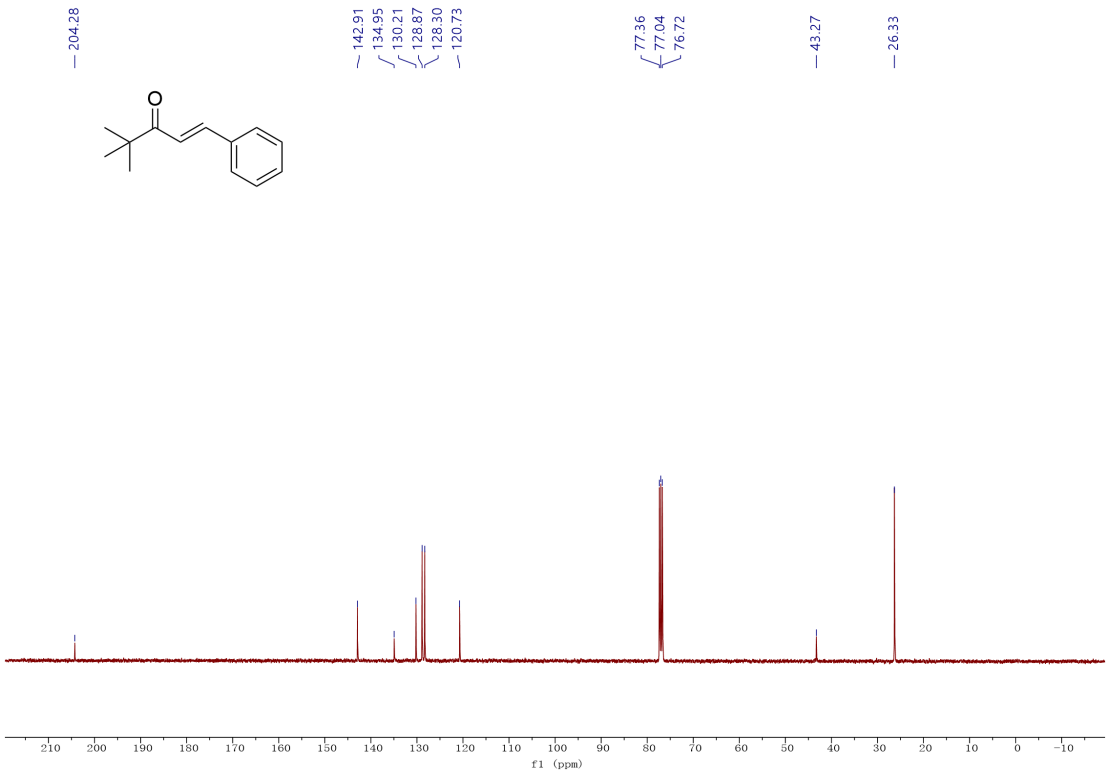


^{13}C NMR ($CDCl_3$) spectrum for **2w**

(*E*)-benzylidenepinacolone (2x)

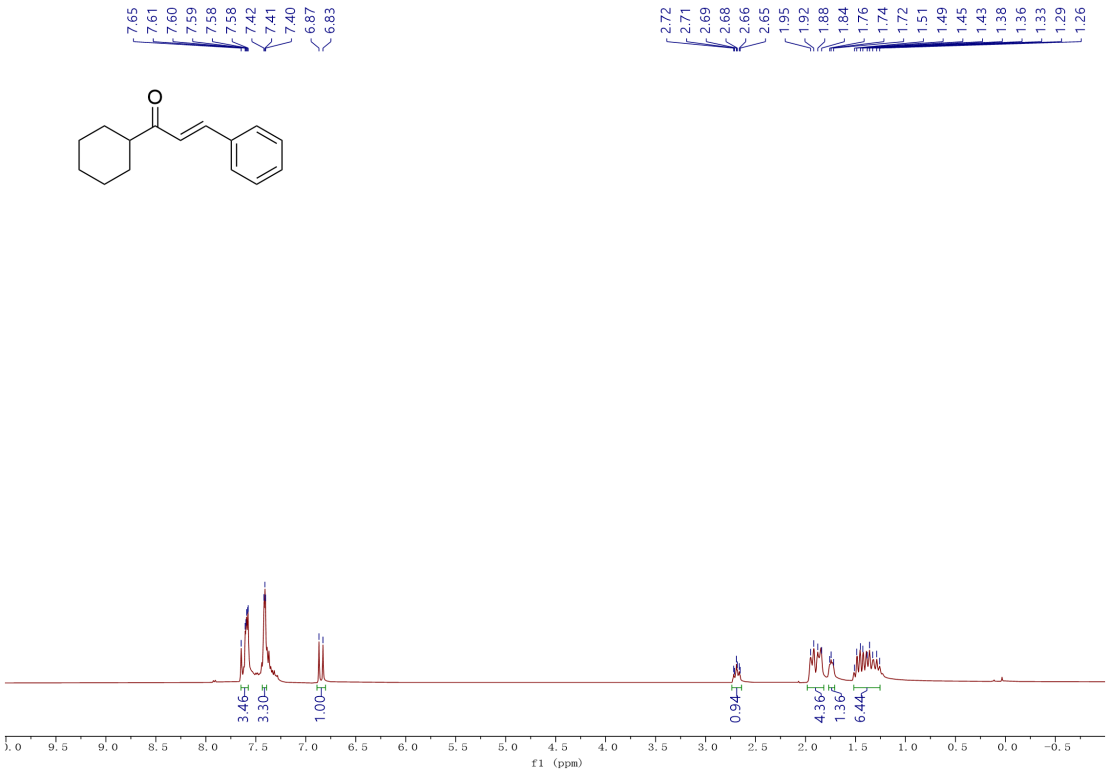


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2x**



^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2x**

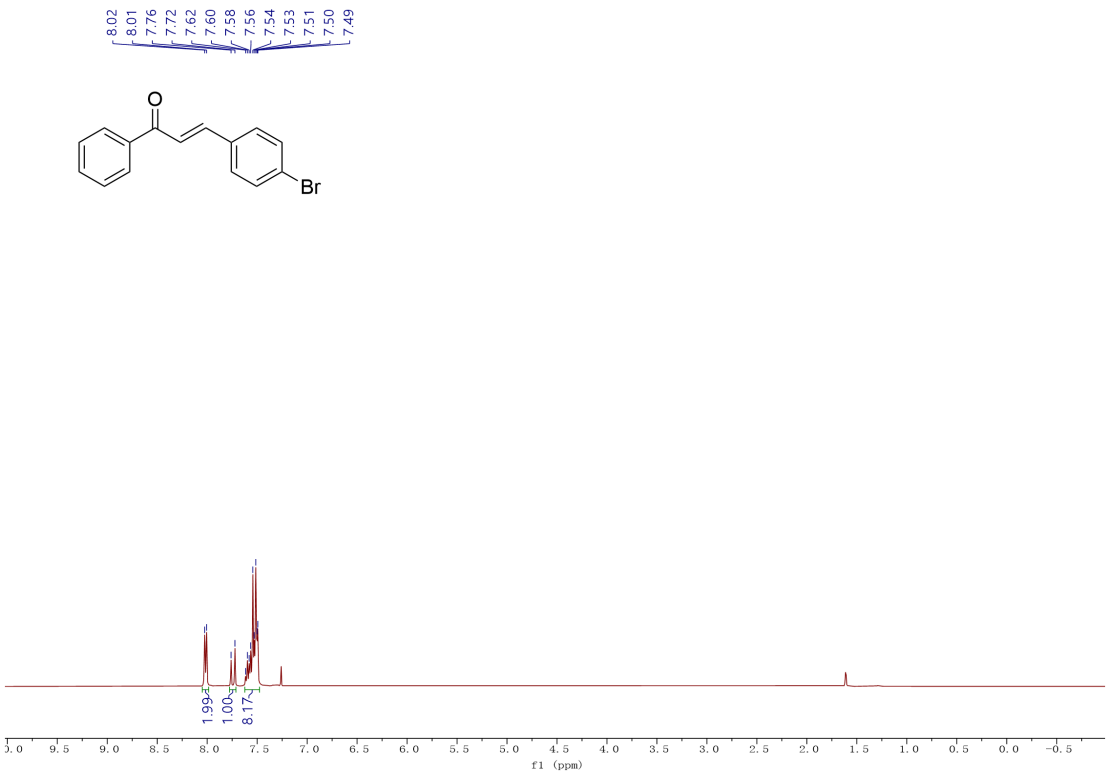
(*E*)-1-cyclohexyl-3-phenylprop-2-en-1-one (2y)



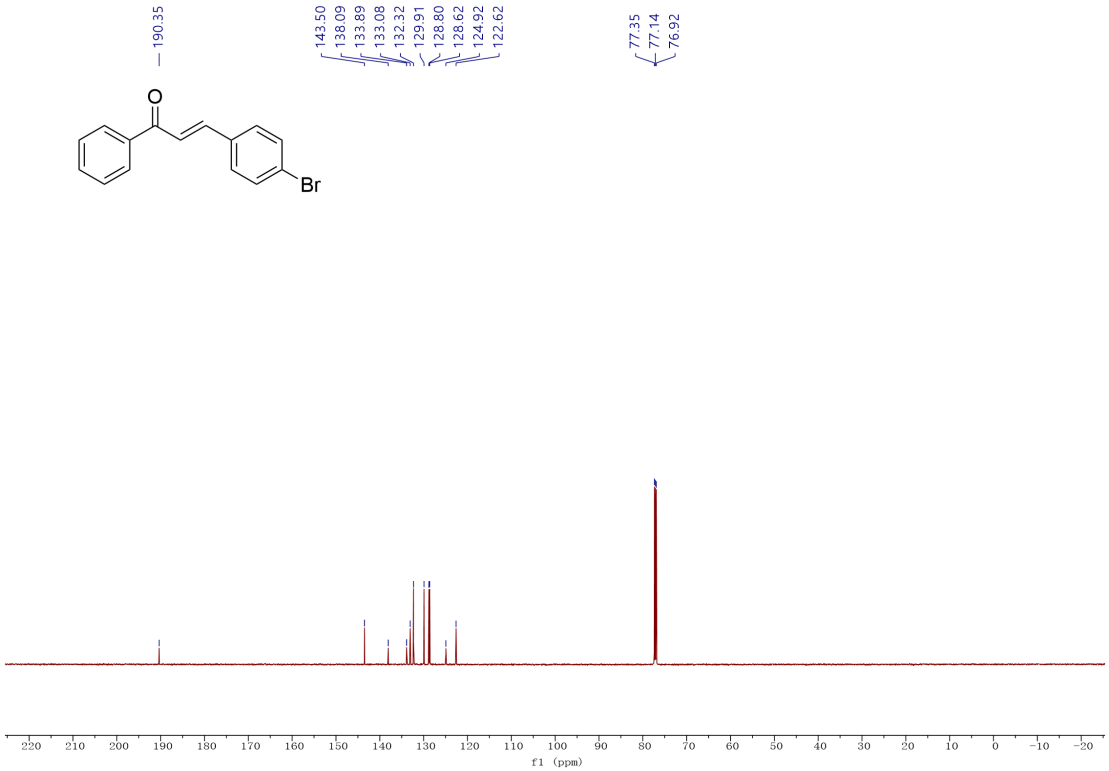
^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2y**

^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2y**

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

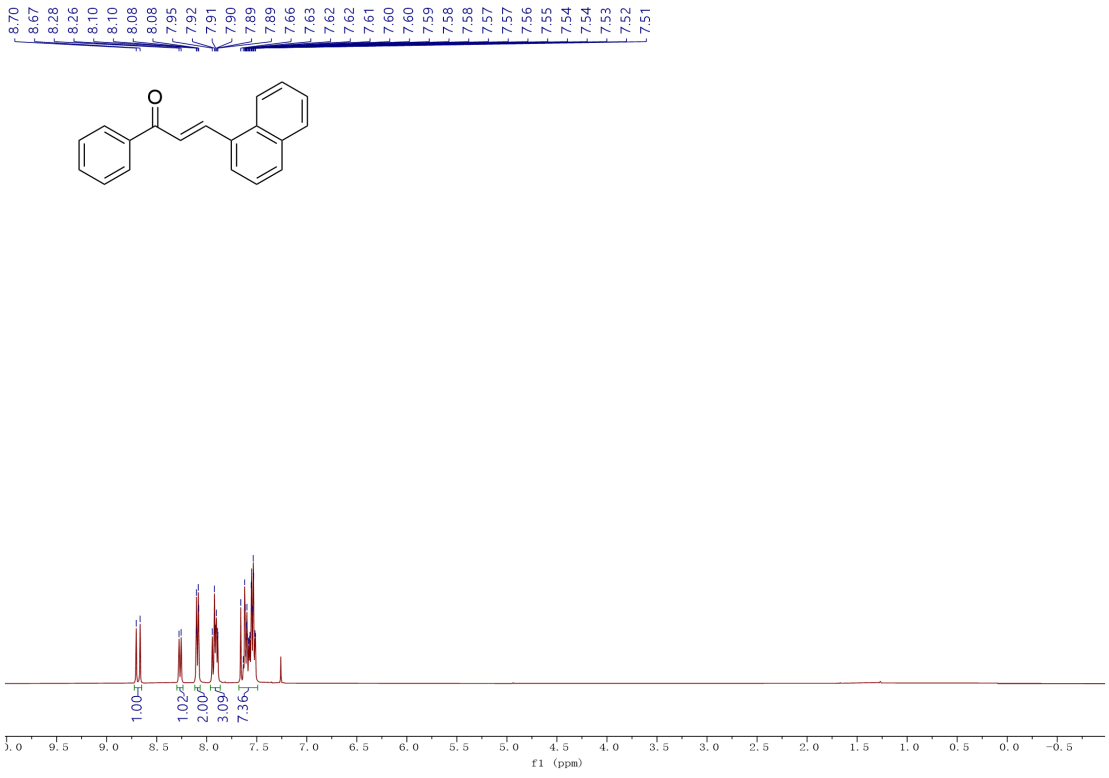


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2z**

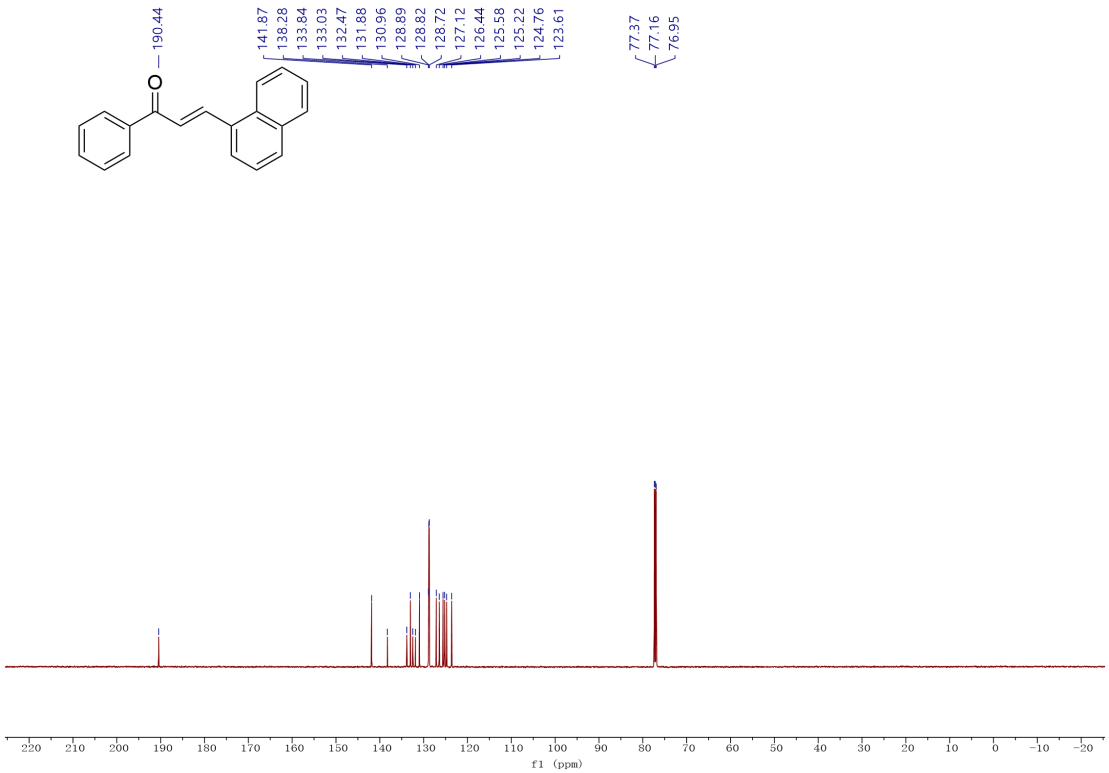


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2z**

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

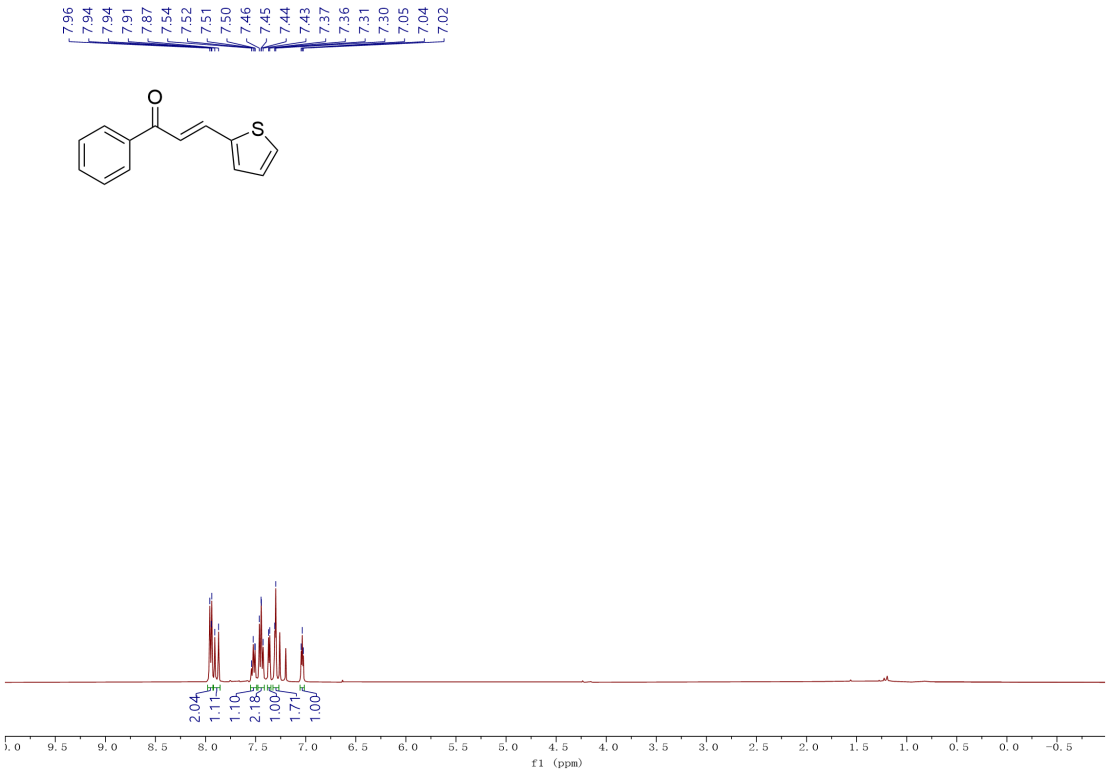


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2aa**

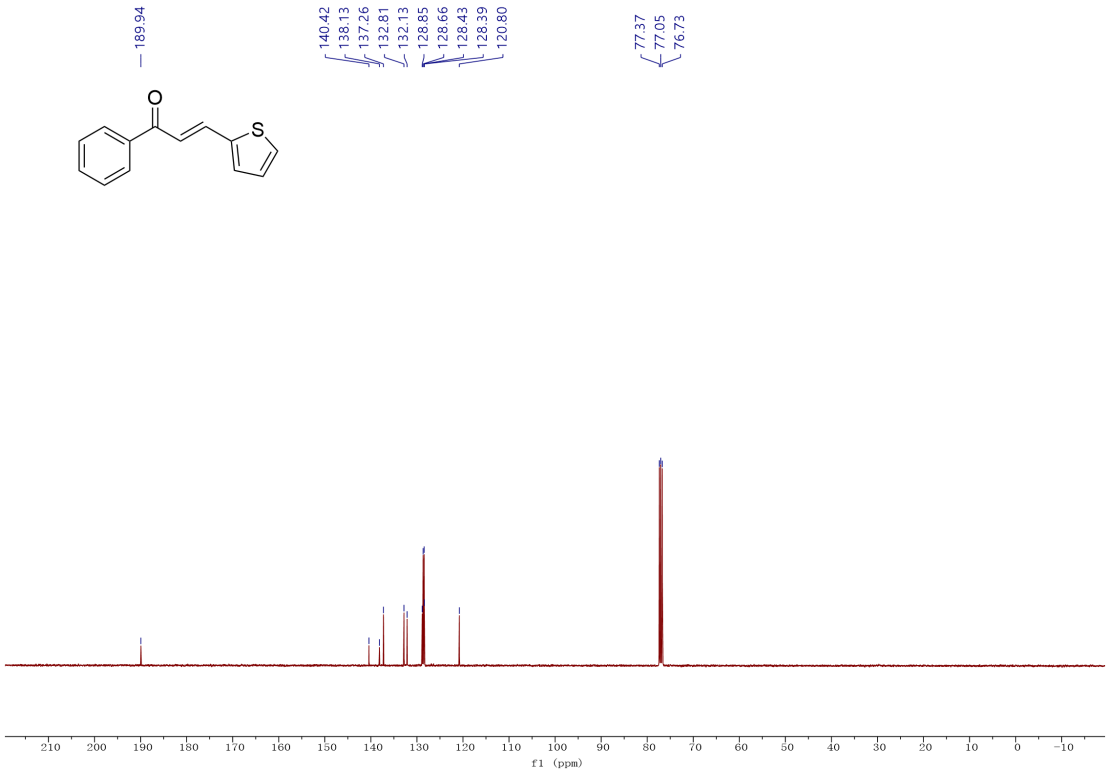


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2aa**

(*E*)-1-phenyl-3-(thiophen-2-yl)-2-propen-1-one (2ab)

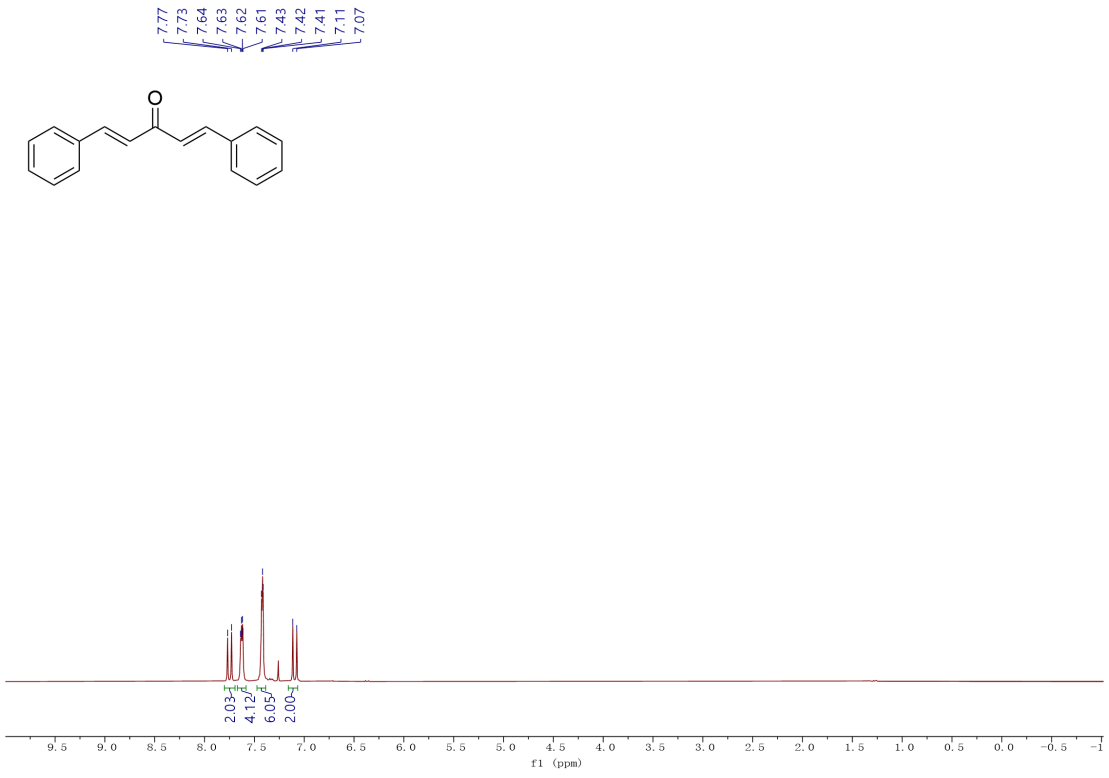


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2ab**

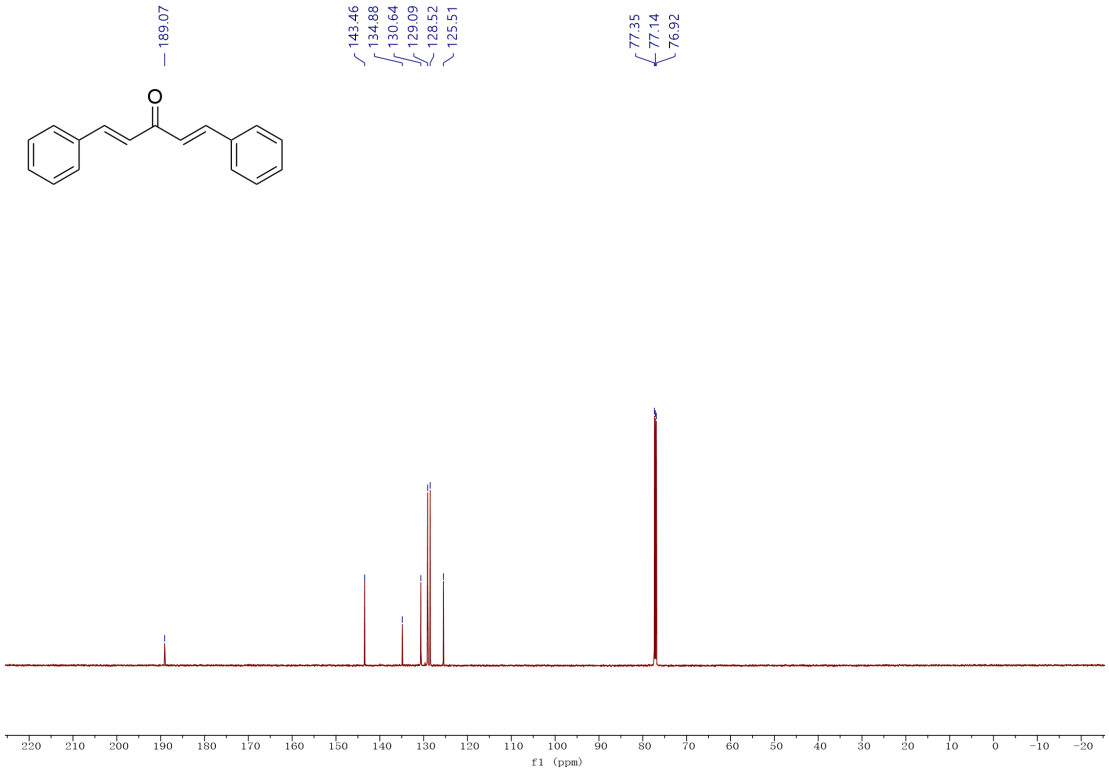


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2ab**

Dibenzylideneacetone (2ac)

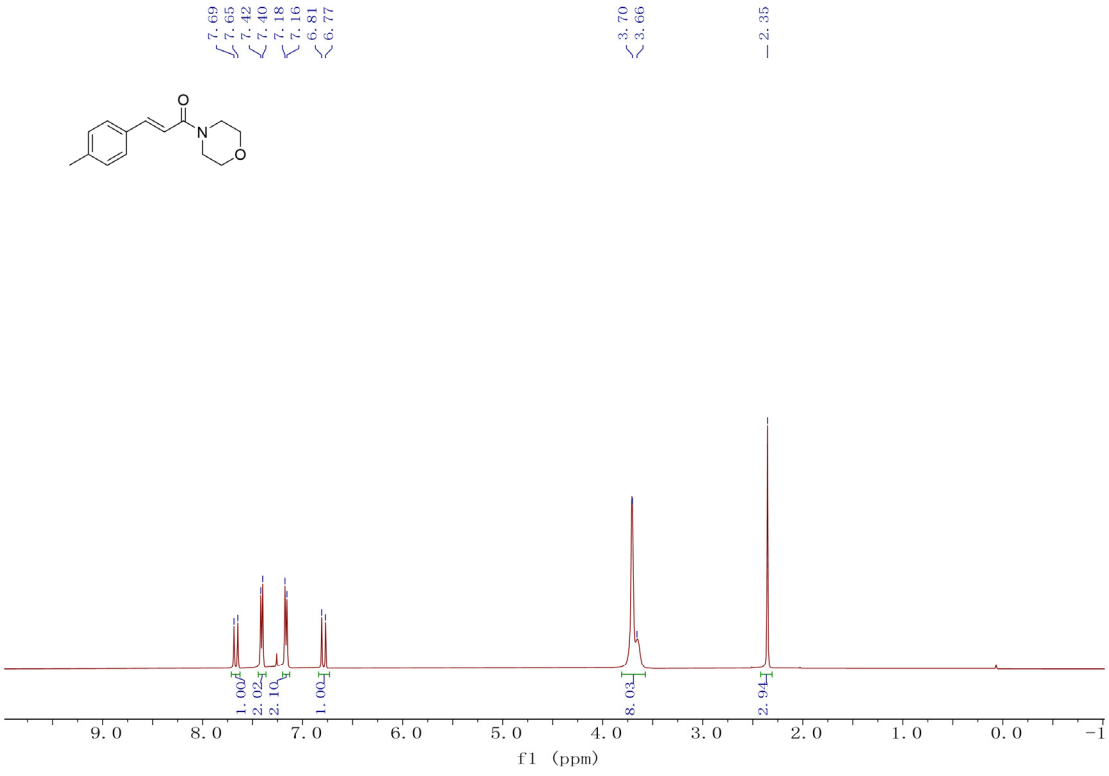


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2ac**

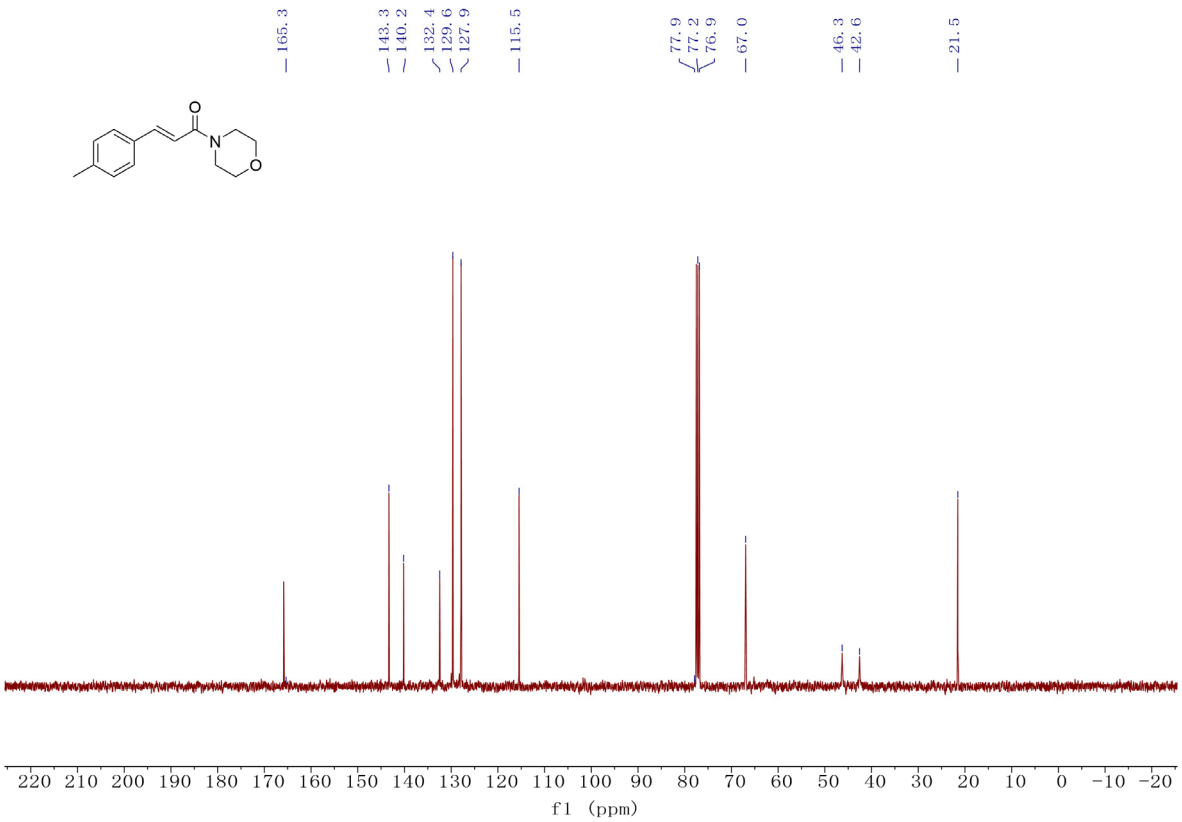


^{13}C NMR (400 MHz, $CDCl_3$) spectrum for **2ac**

(*E*)-1-morpholino-3-(p-tolyl)prop-2-en-1-one (2ad)

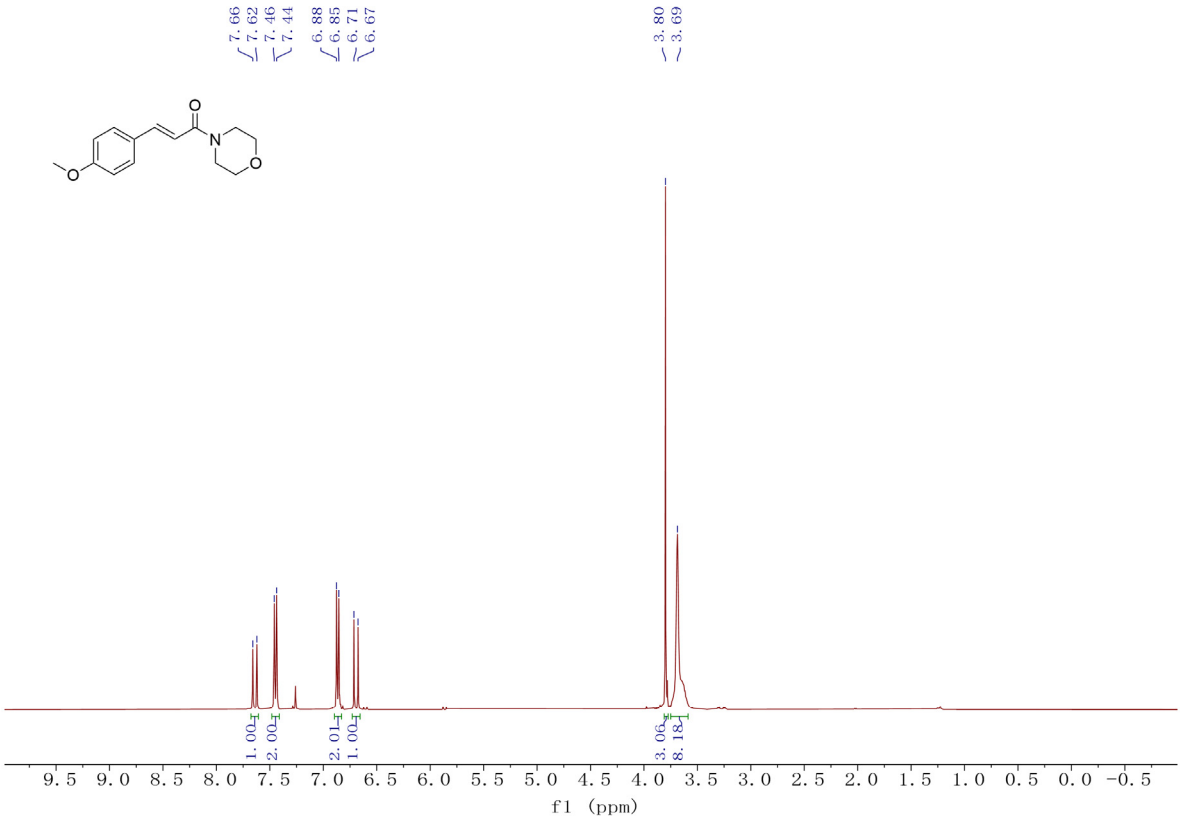


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2ad**

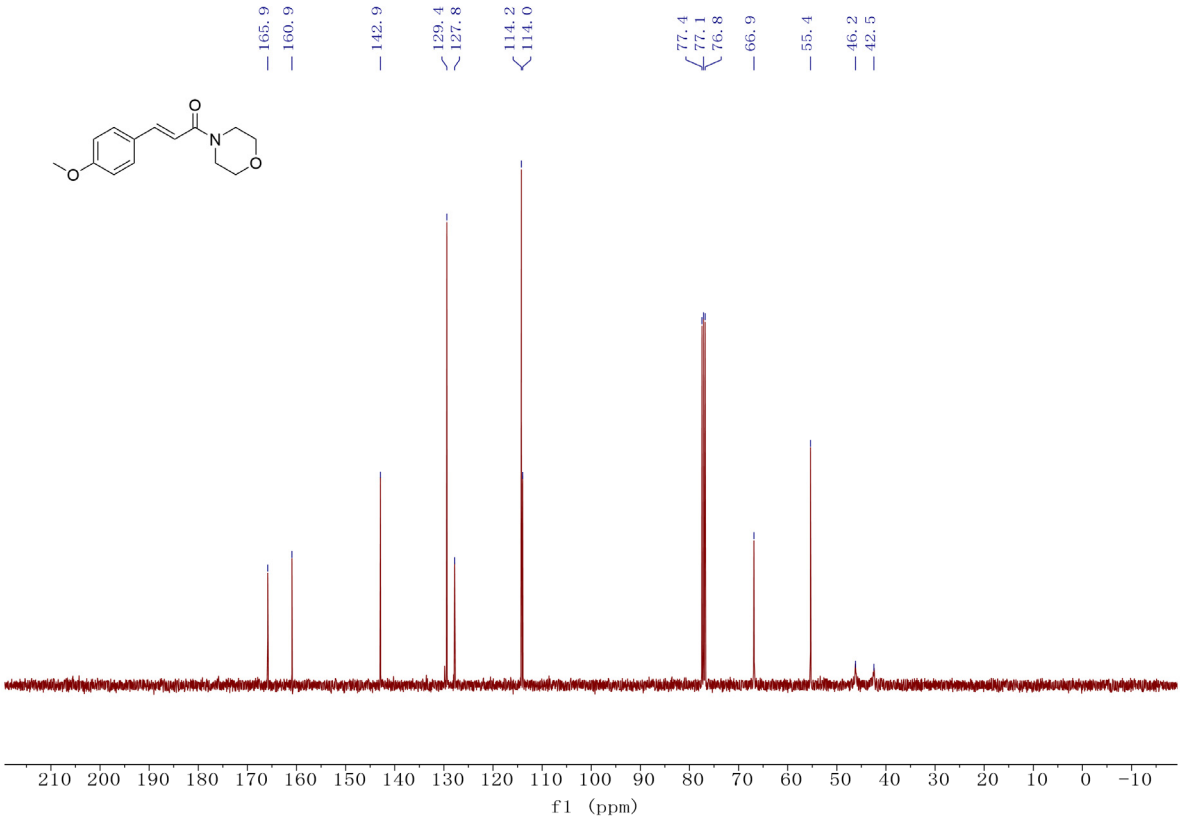


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2ad**

(*E*)-3-(4-methoxyphenyl)-1-morpholinoprop-2-en-1-one (2ae)

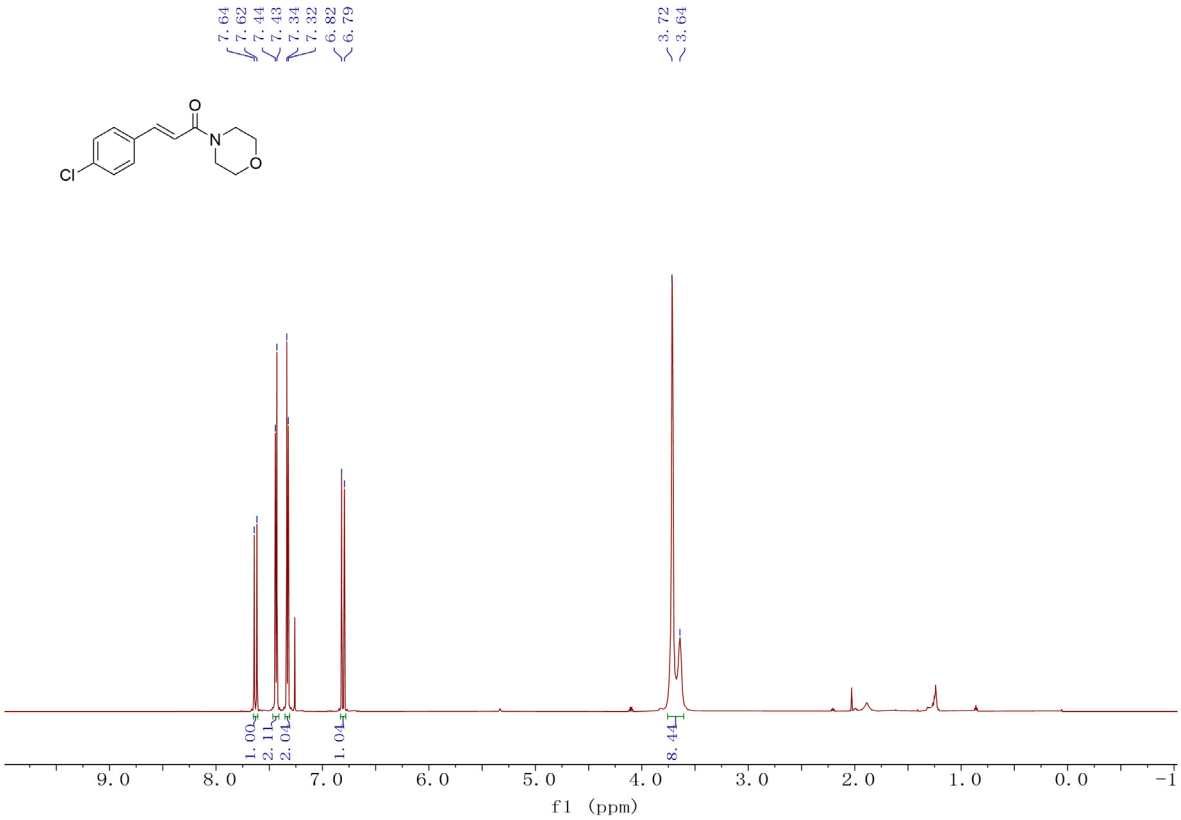


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2ae**

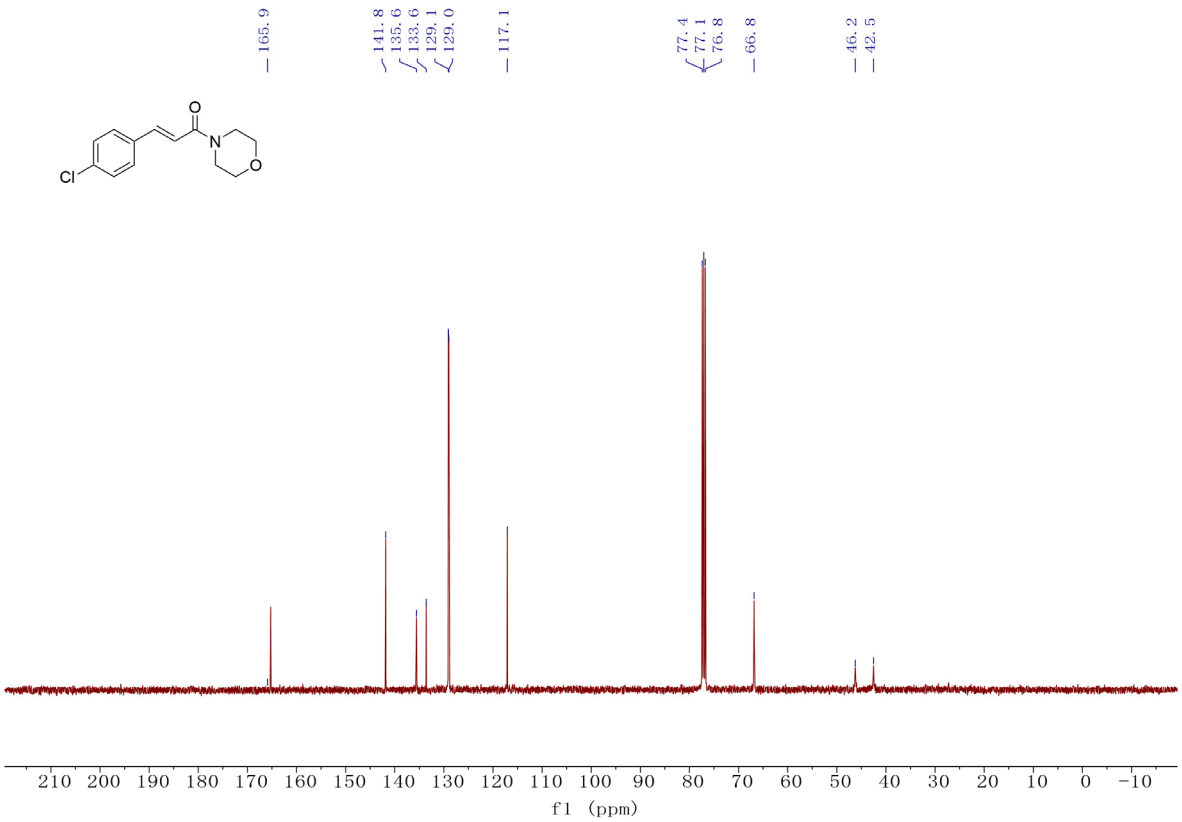


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2ae**

(*E*)-3-(4-chlorophenyl)-1-morpholinoprop-2-en-1-one (2af)

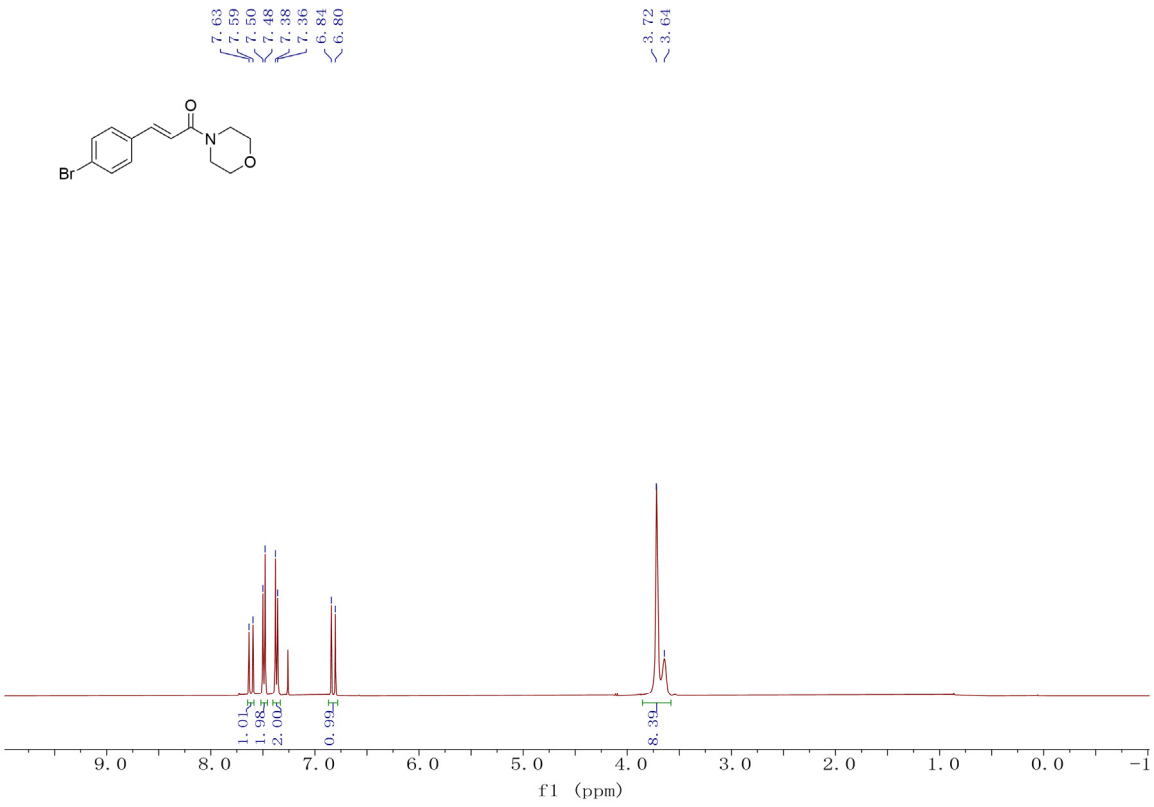


^{1}H NMR (600 MHz, $CDCl_3$) spectrum for **2af**

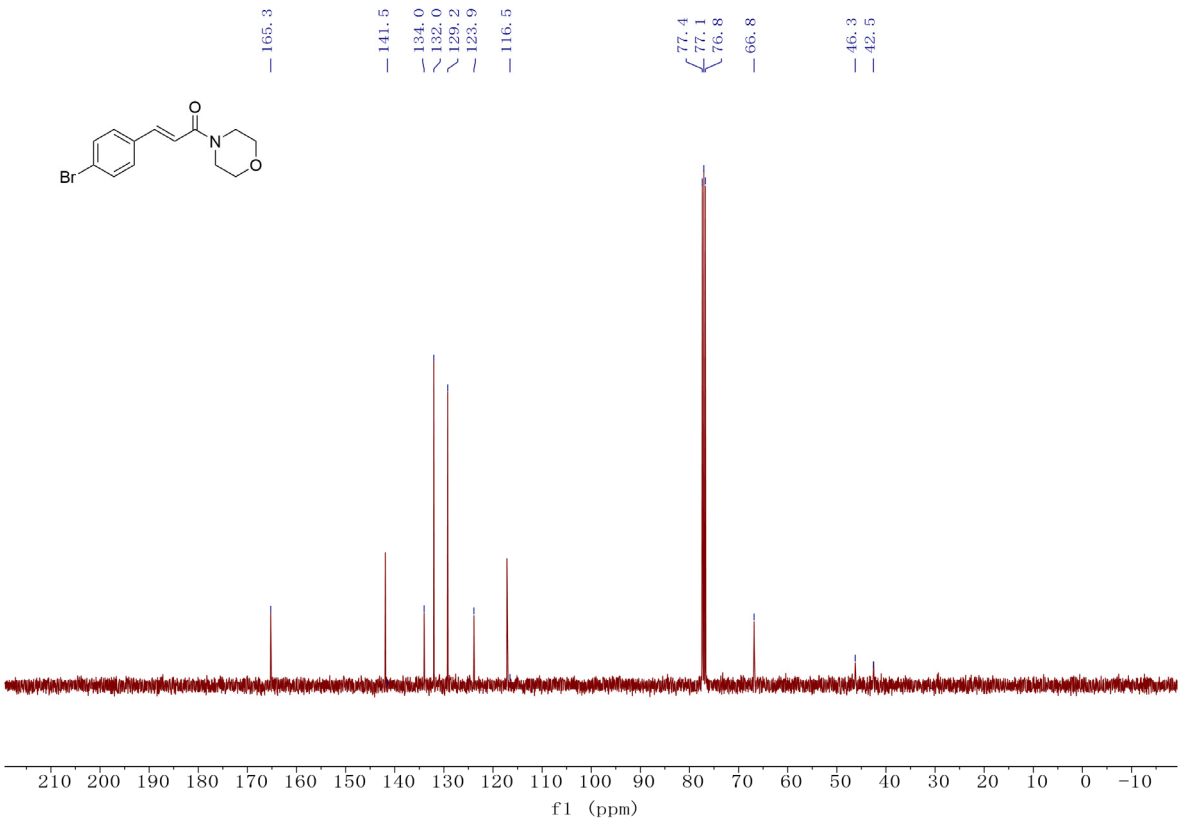


^{13}C NMR (151 MHz, $CDCl_3$) spectrum for **2af**

4-[(2E)-3-(4-bromophenyl)prop-2-enoyl]morpholine (2ag)

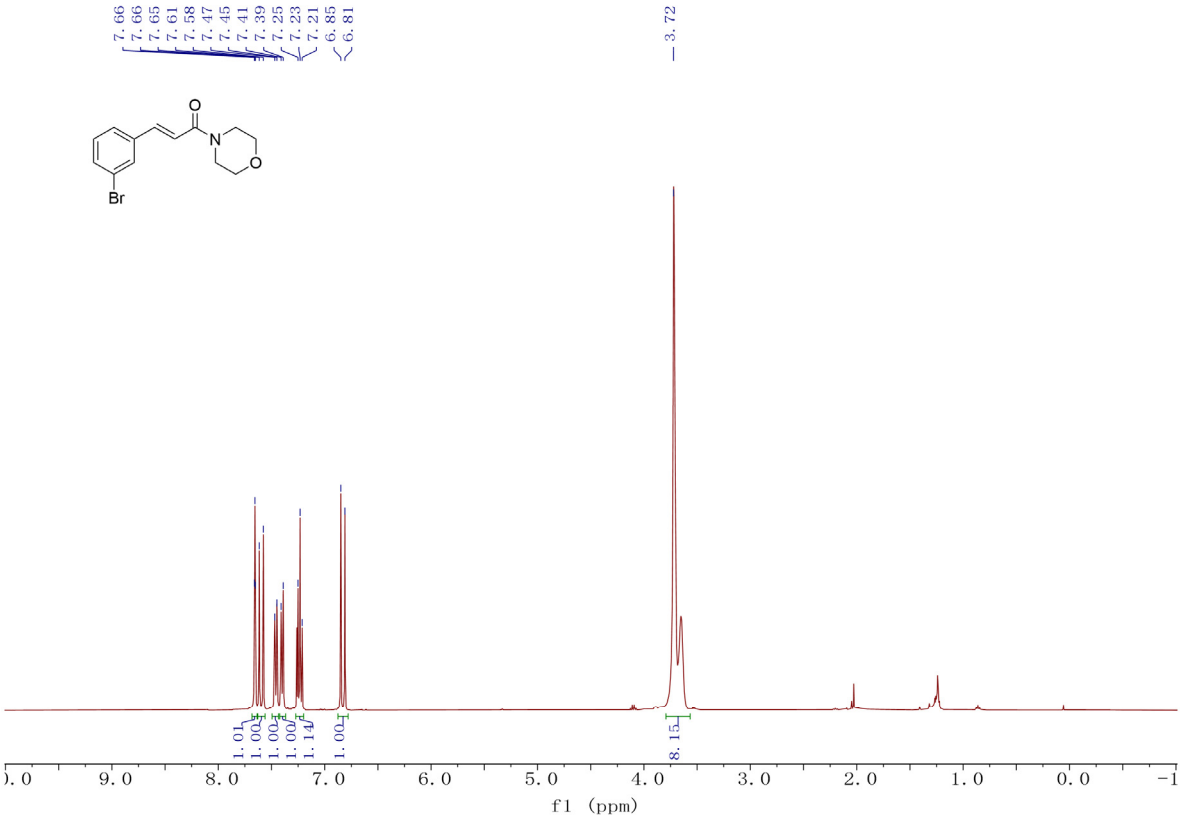


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2ag**

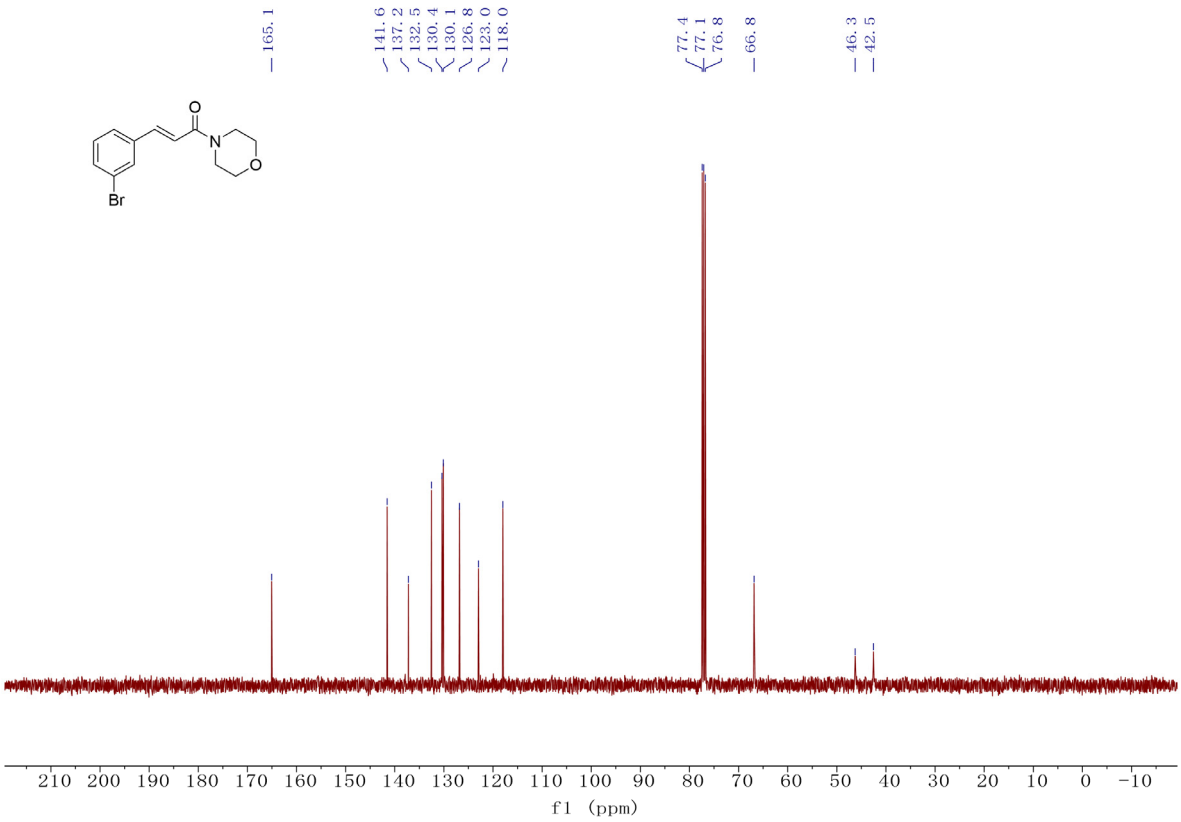


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2ag**

3-(3-bromophenyl)-1-morpholinoprop-2-en-1-one (2ah)

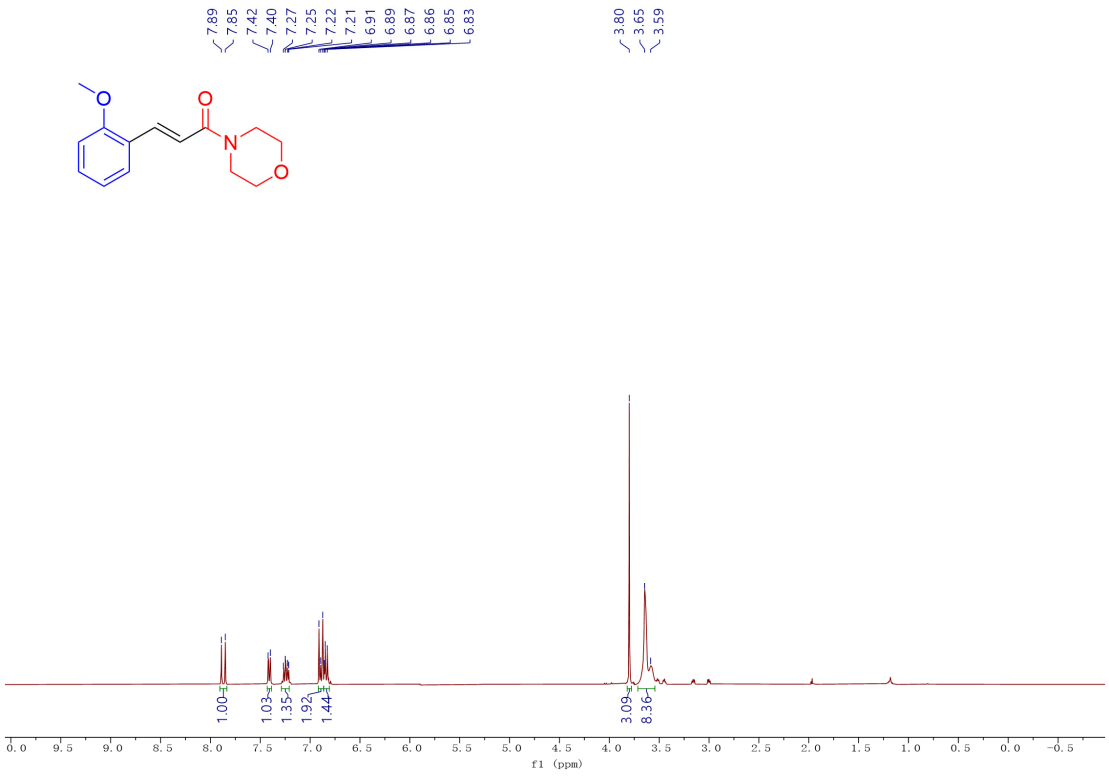


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2ah**

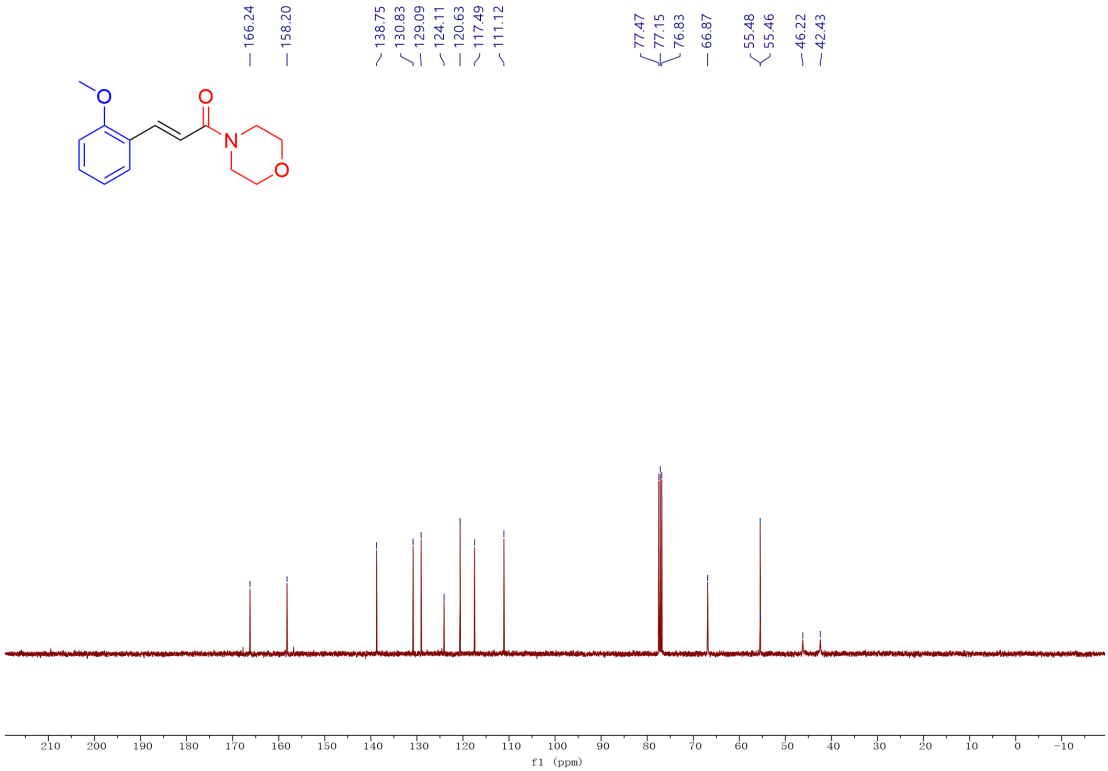


^{13}C NMR ($CDCl_3$) spectrum for **2ah**

(*E*)-3-(2-methoxyphenyl)-1-morpholin-4-ylprop-2-en-1-one (2ai)

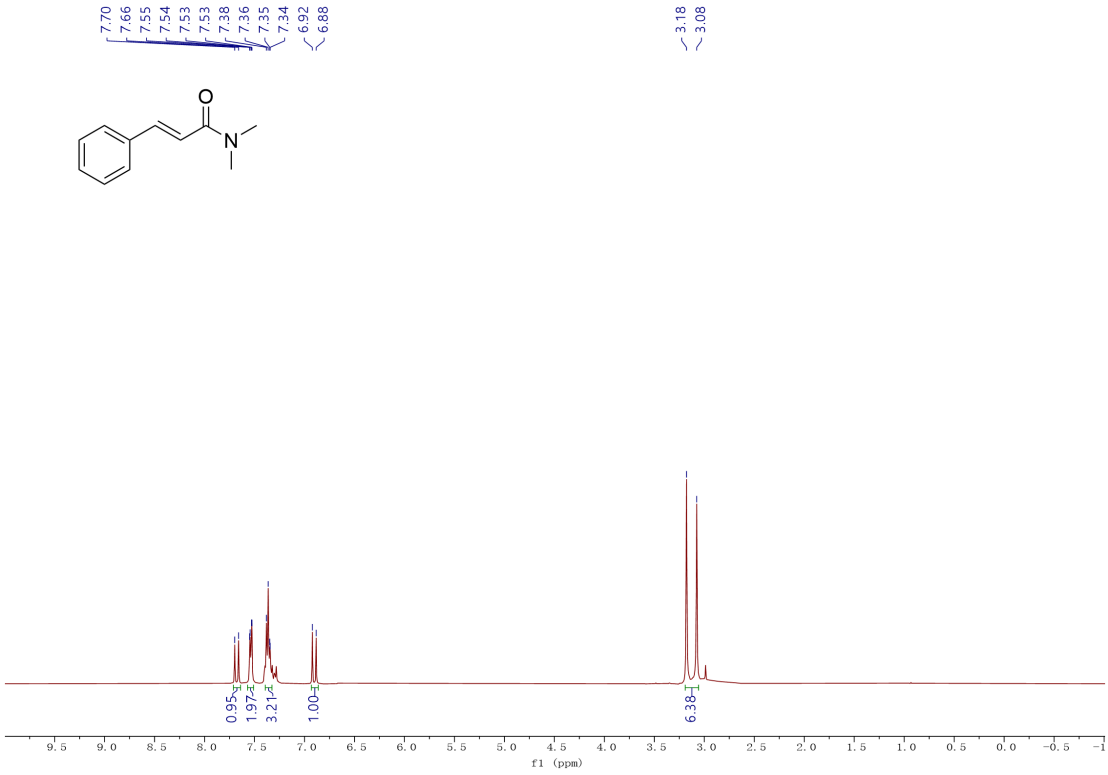


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2ai**

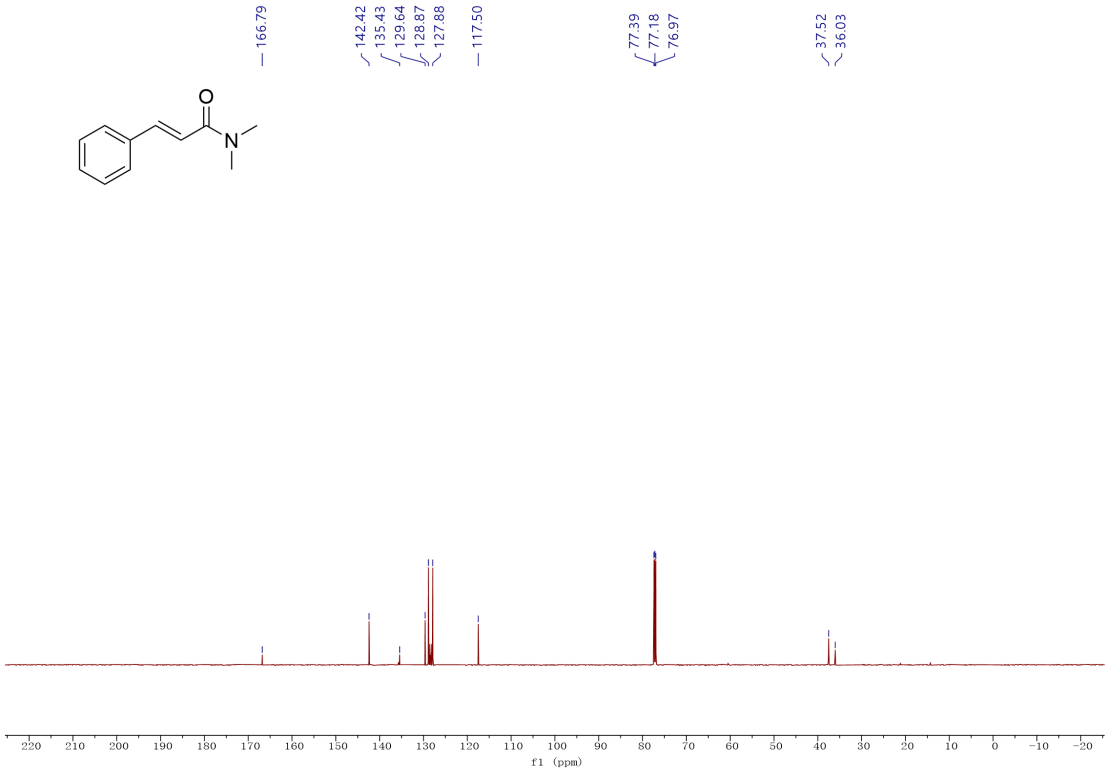


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2ai**

(E)-N,N-dimethylcinnamamide (2aj)

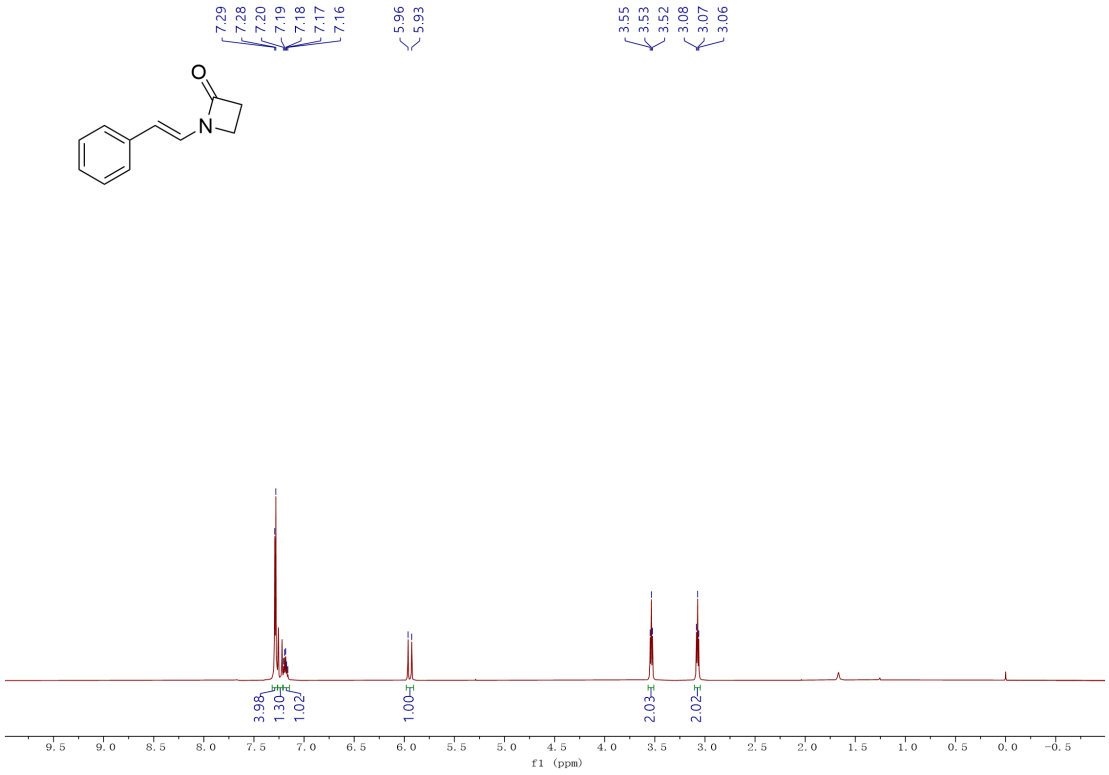


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2aj**

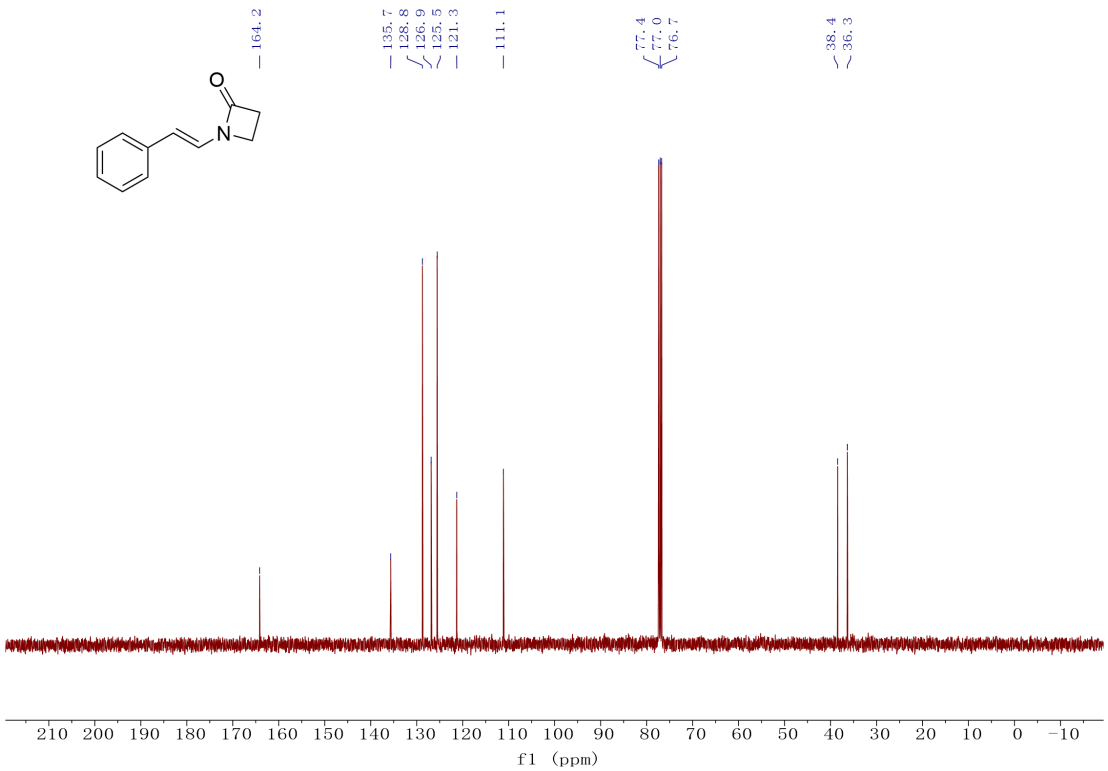


^{13}C NMR (400 MHz, $CDCl_3$) spectrum for **2aj**

2-(phenyl)ethen-1-ylazetidinone (2ak)

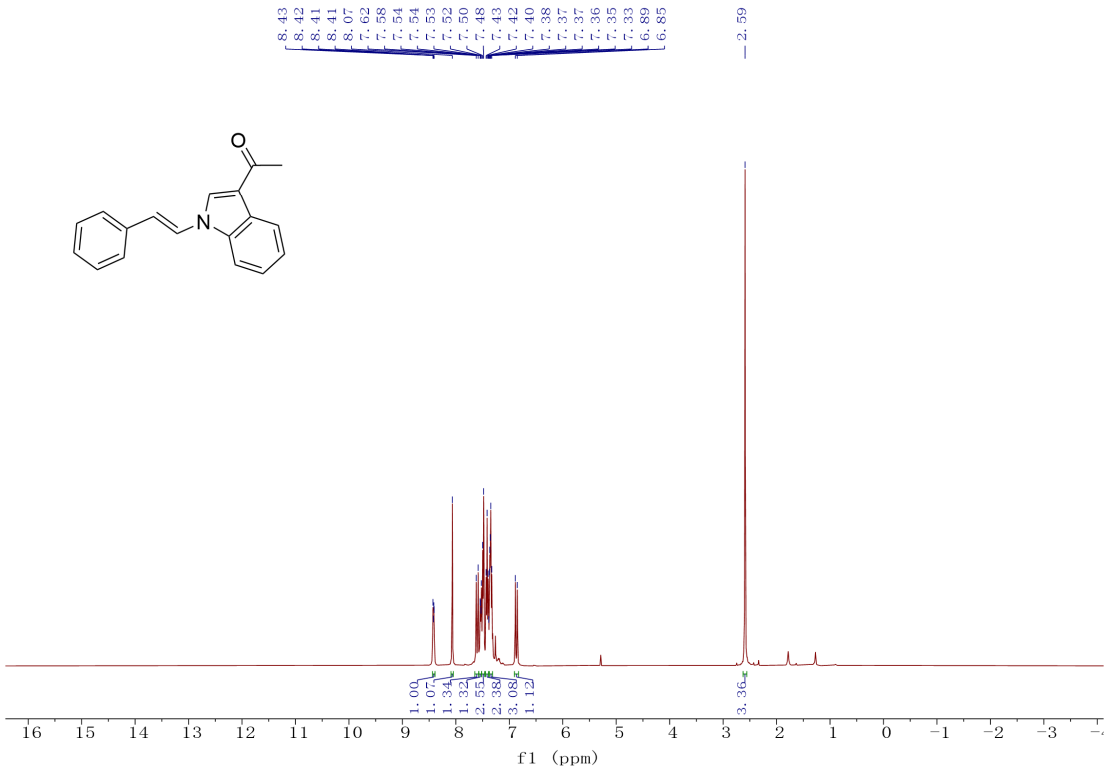


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2ak**

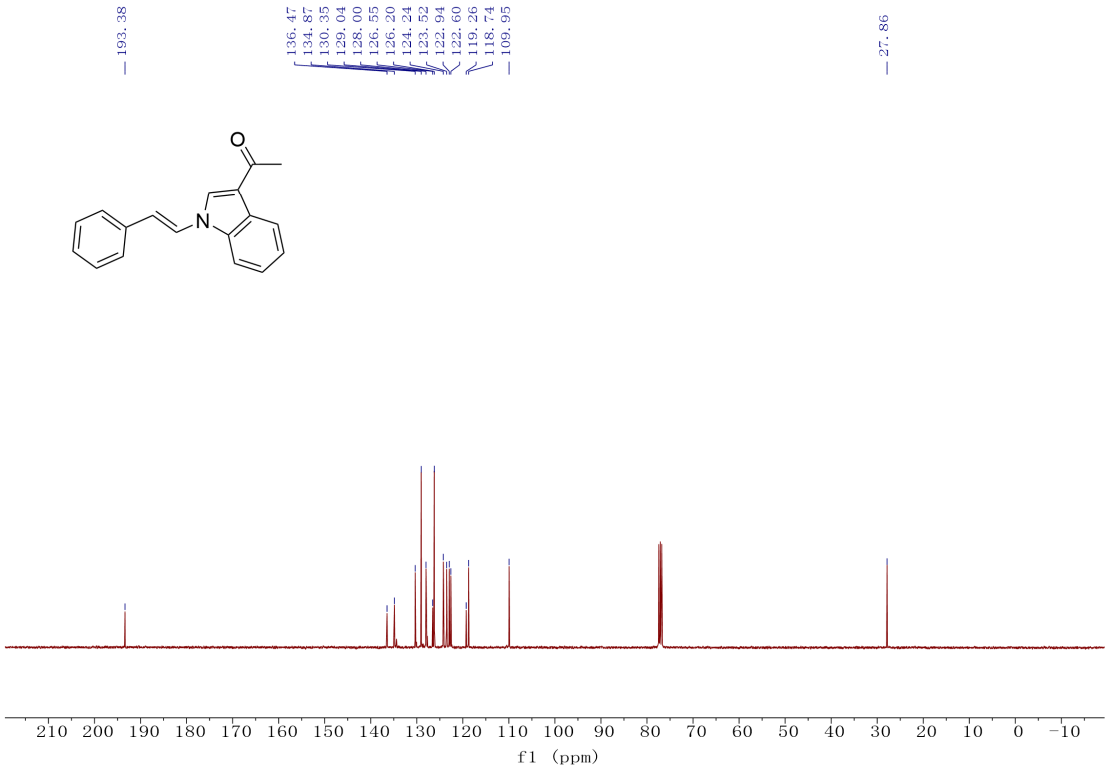


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2ak**

(*E*)-1-(1-styryl-1H-indol-3-yl)ethan-1-one (2al)

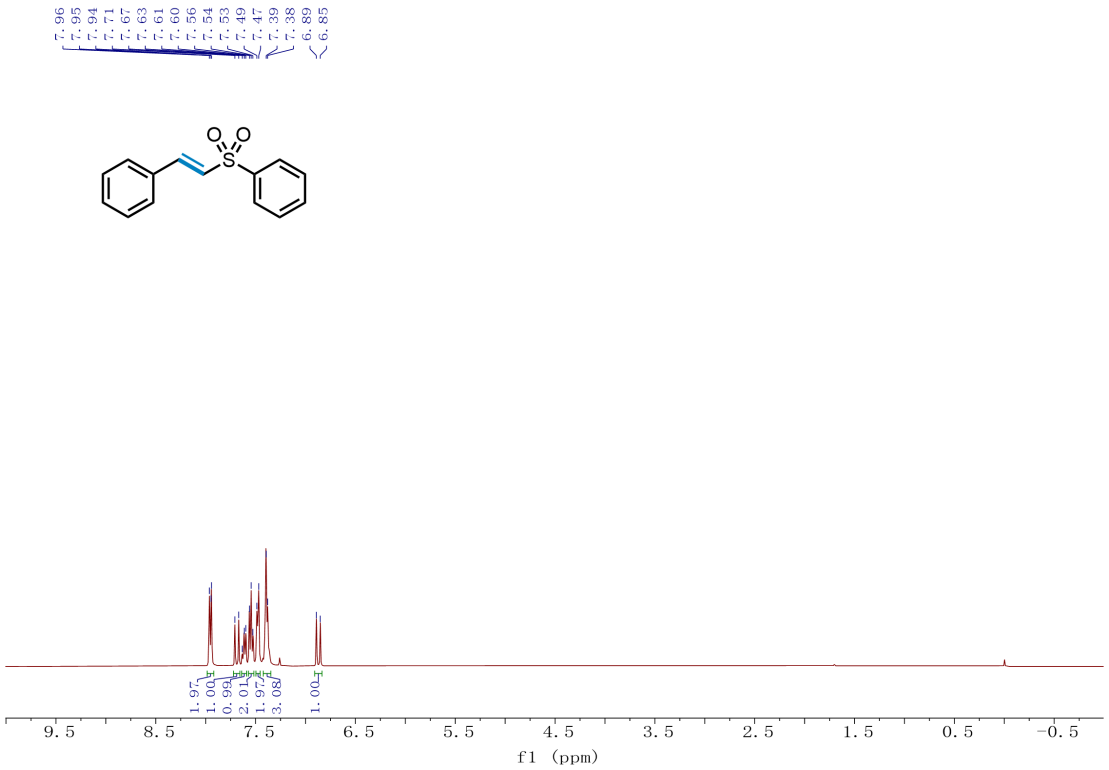


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2al**

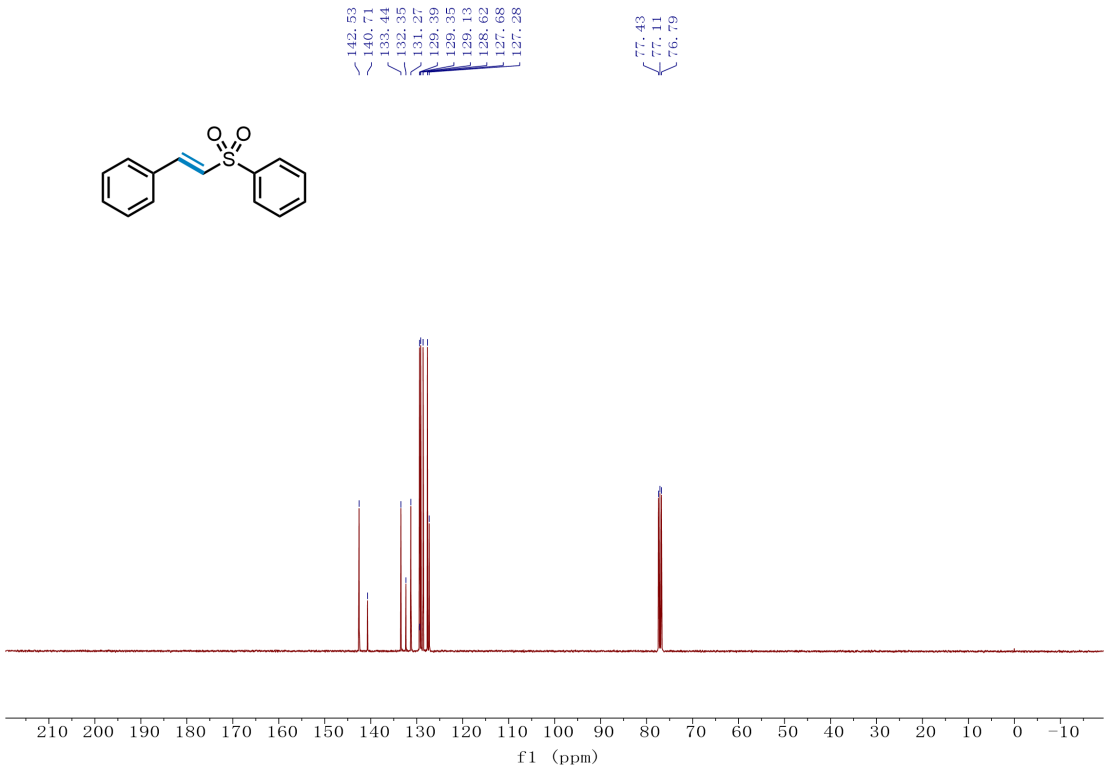


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2al**

phenyl styryl sulfone (2am)

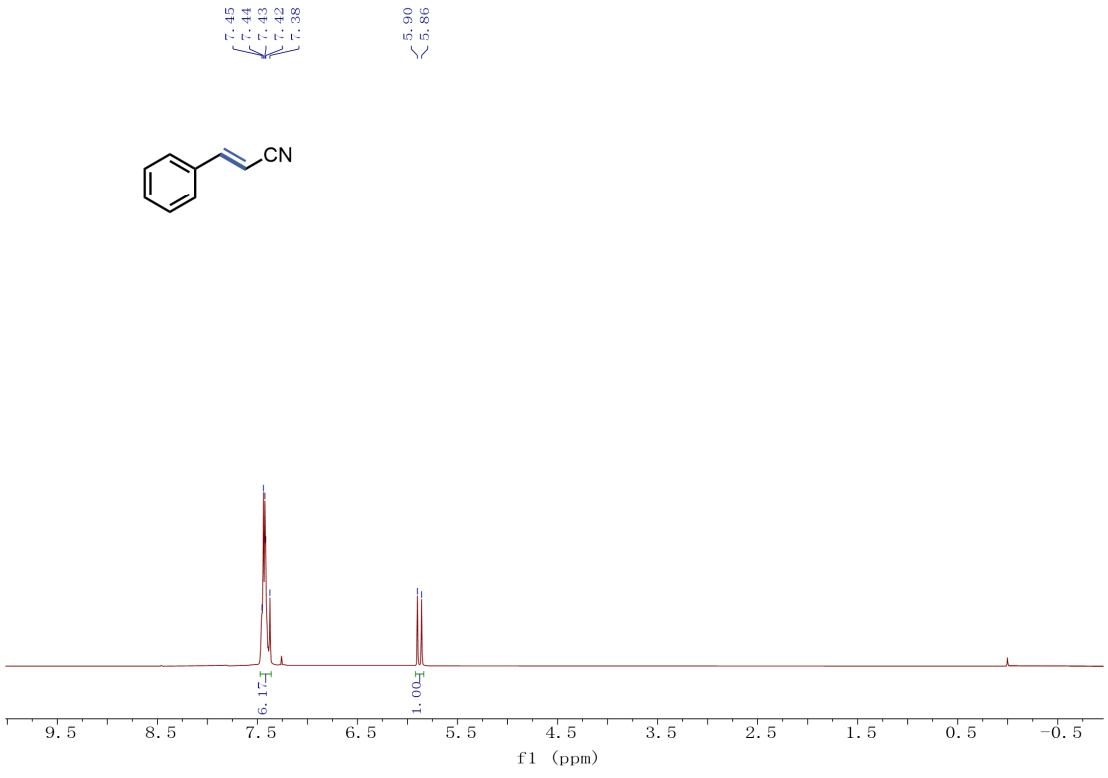


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2am**

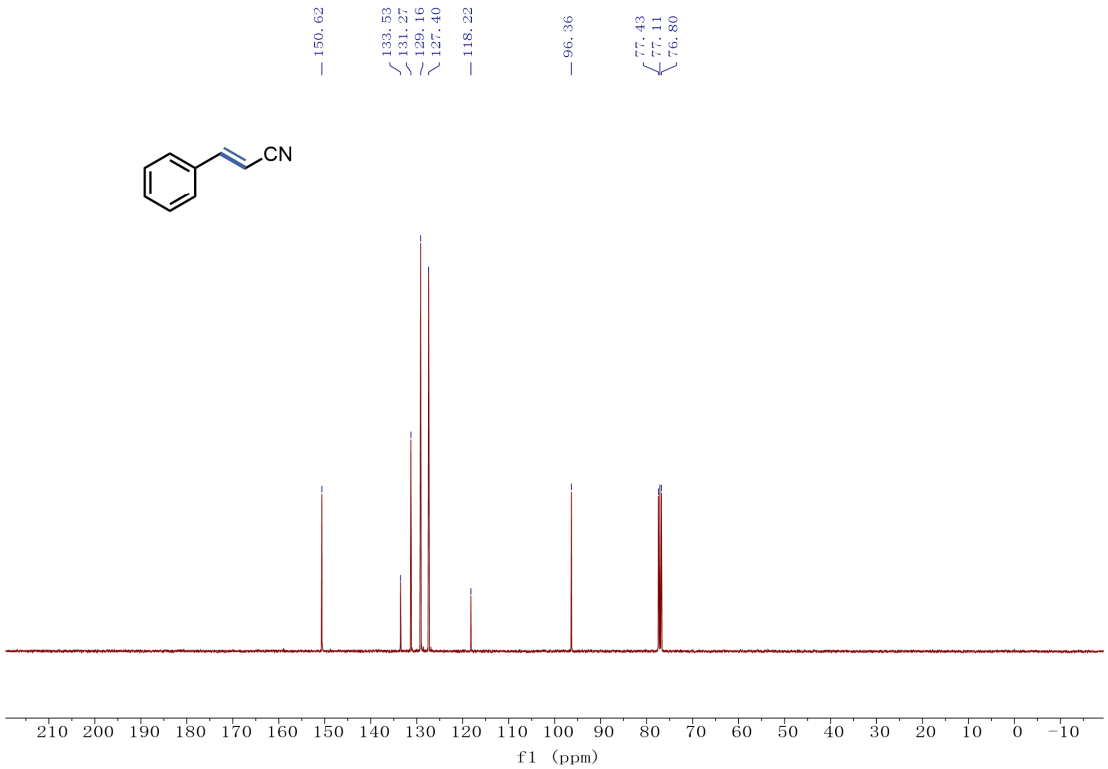


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2am**

cinnamic nitrile (2an)

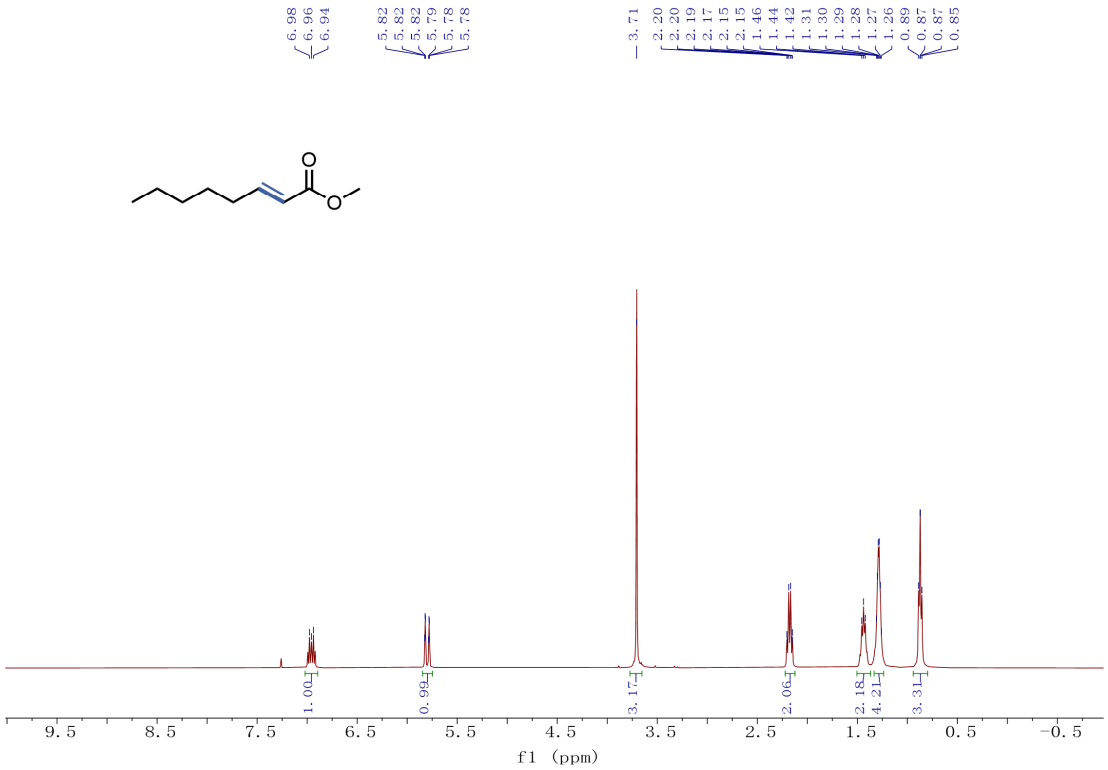


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2an**

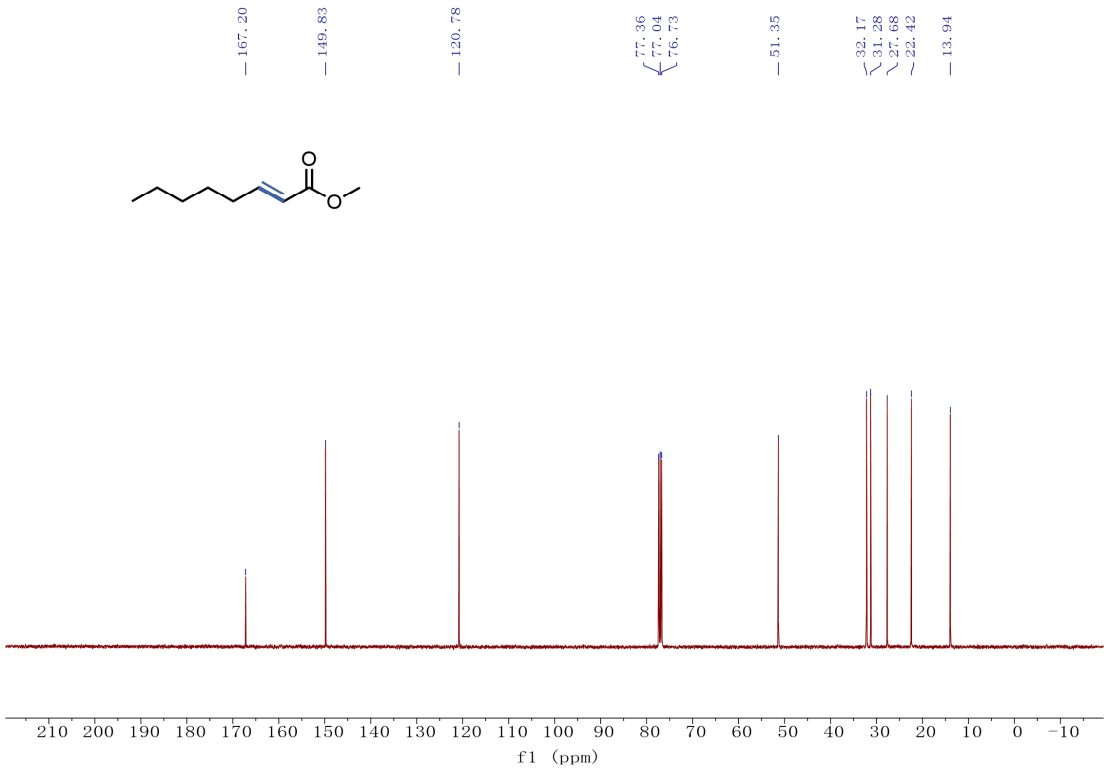


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2an**

Methyl (E)-oct-2-enoate (2ao)

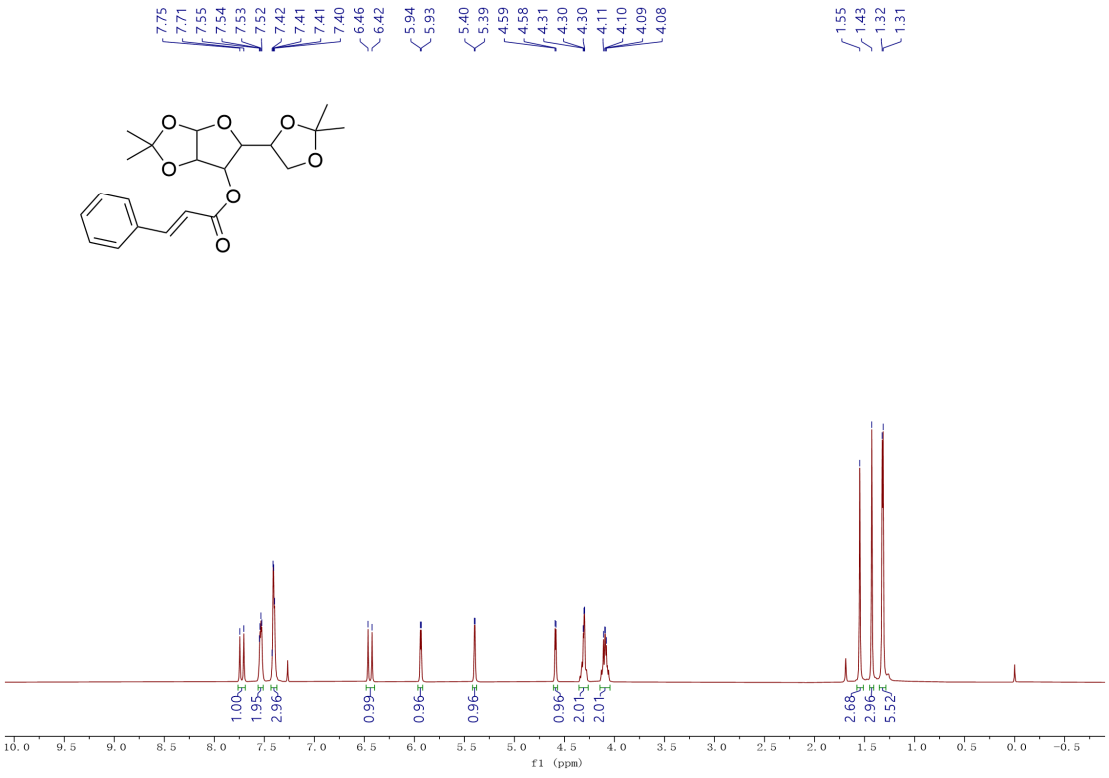


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2ao**

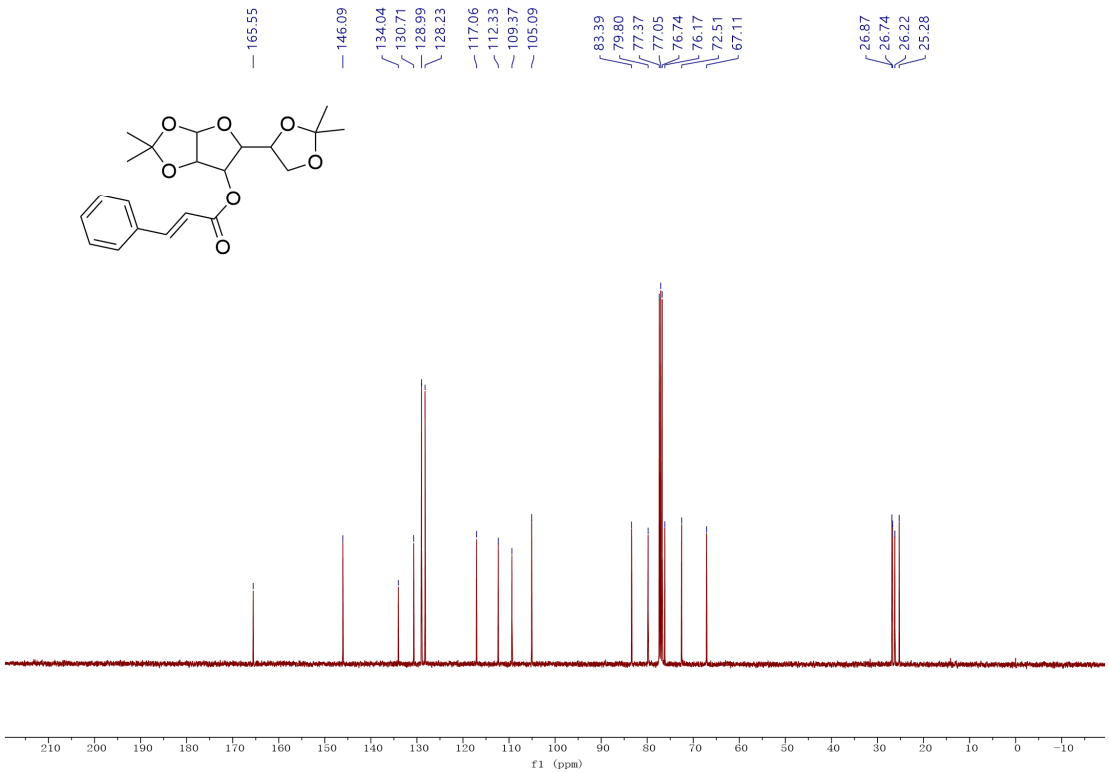


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2ao**

5-(2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-6-yl cinnamate (2ar)

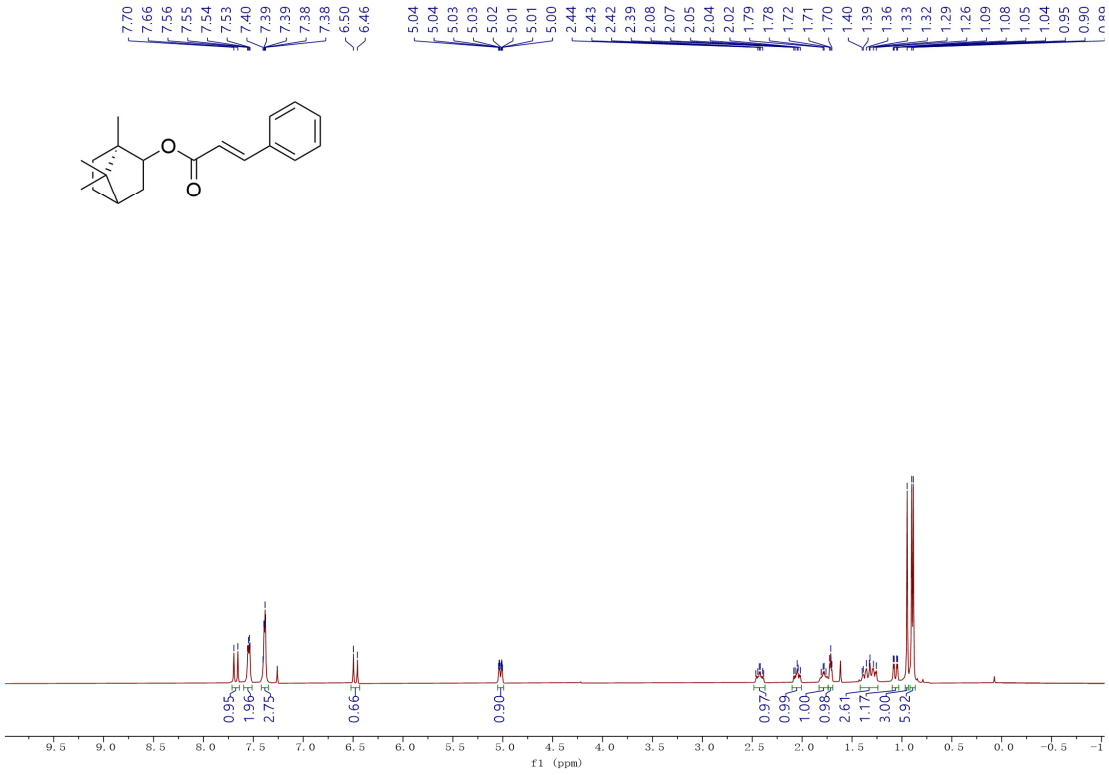


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2ar**

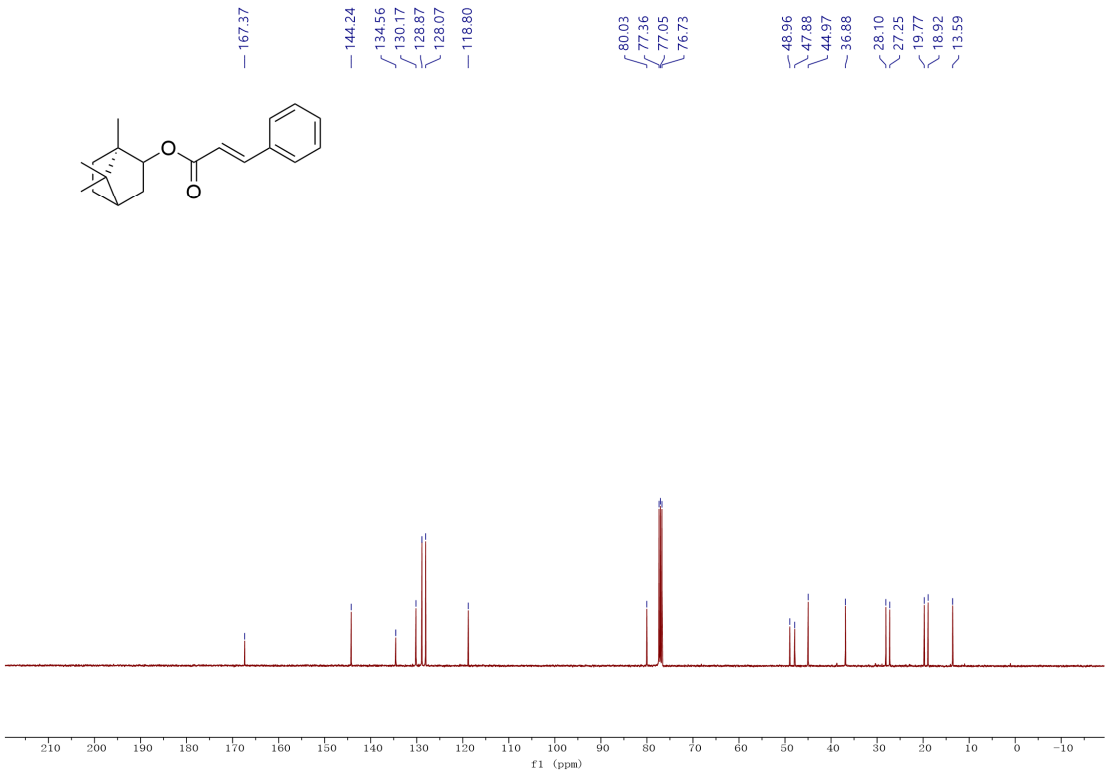


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2ar**

(1R)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl cinnamate (2as)

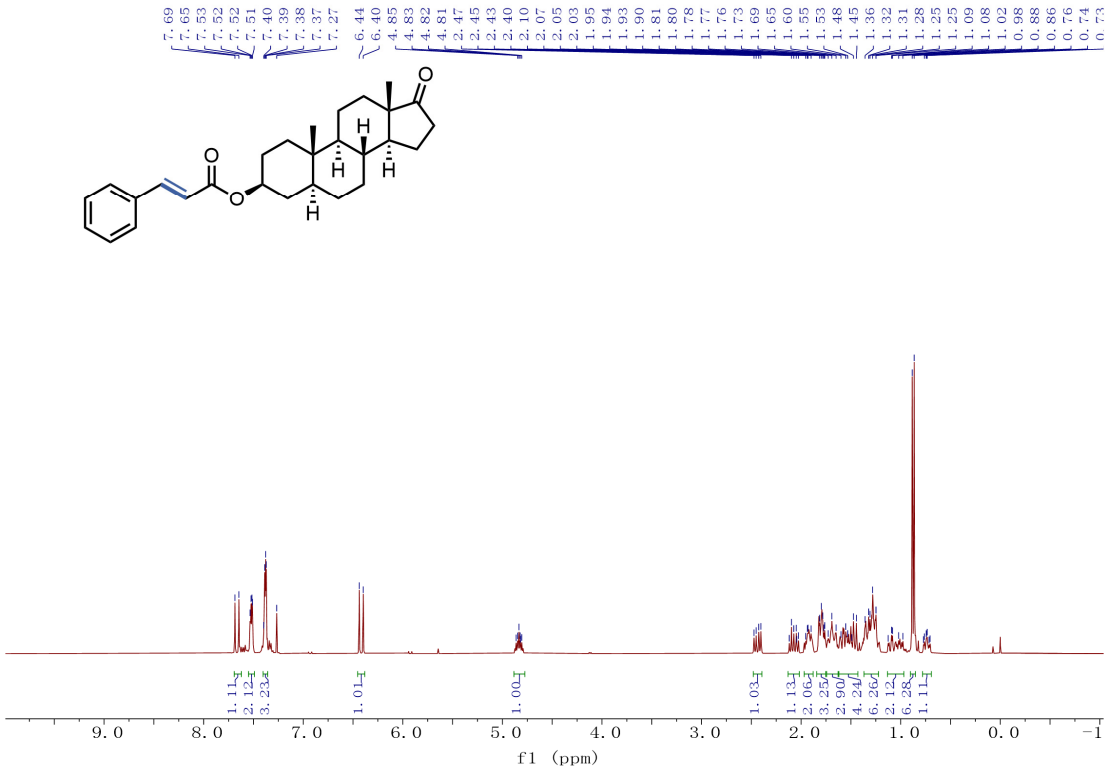


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2as**

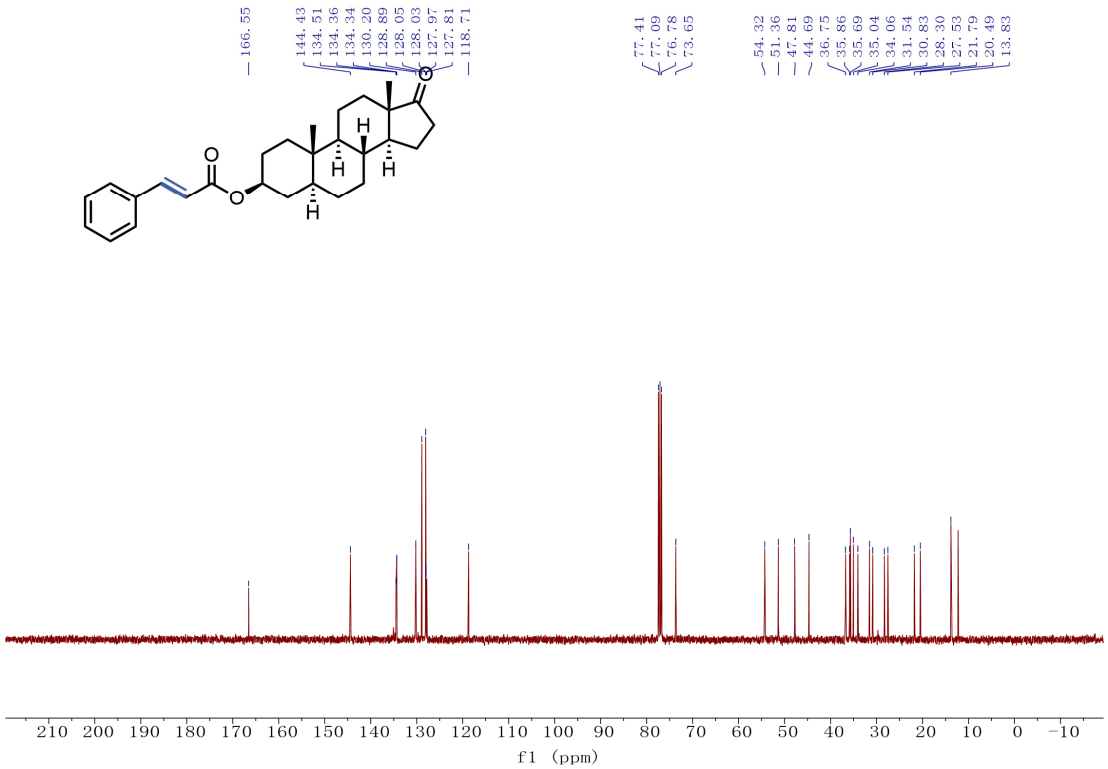


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2as**

(3S,5S,8R,9S,10S,13S,14S)-10,13-dimethyl-17-oxohexadecahydro-1H-cyclopenta[a]phenanthren-3-yl cinnamate (2at)

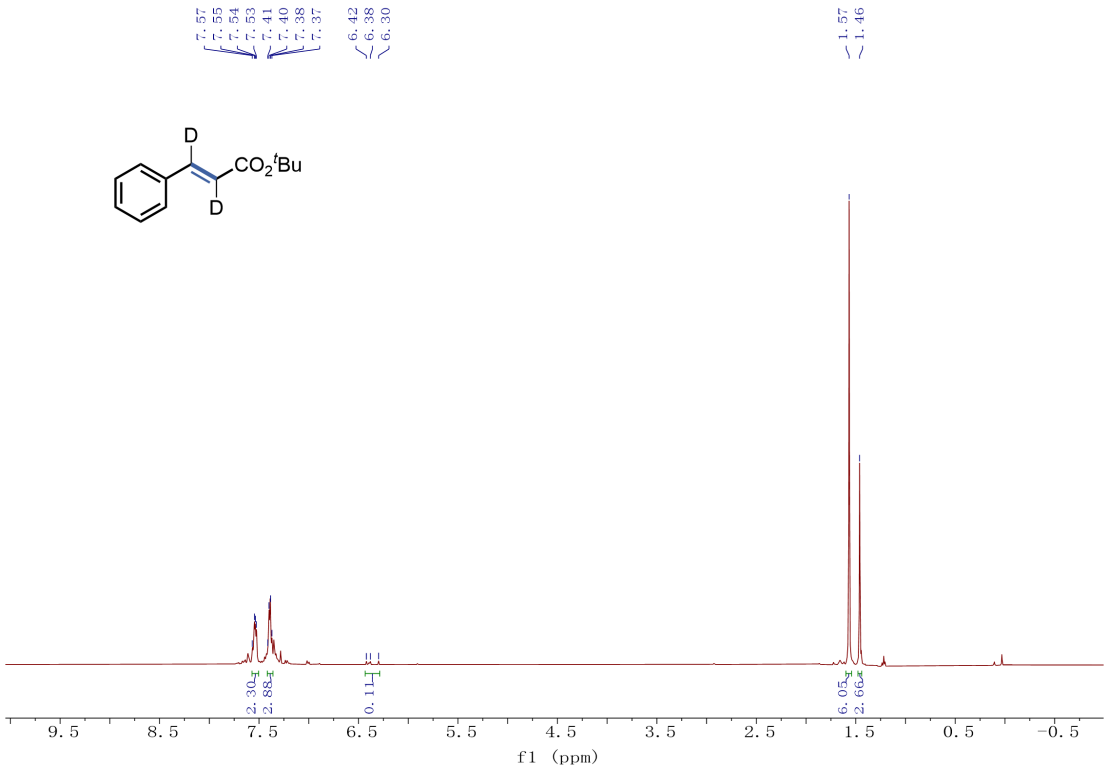


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2at**

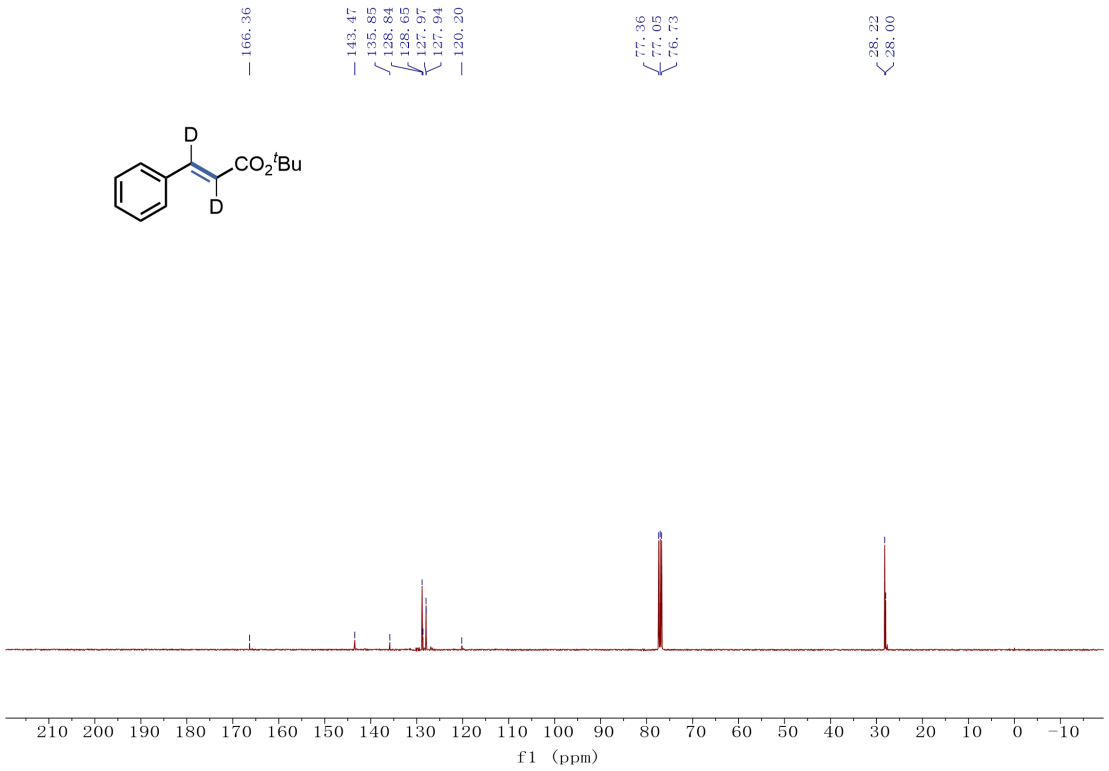


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2at**

tert-butyl cinnamate-2,3-*d*$_2$ (2c-*d*$_2$)

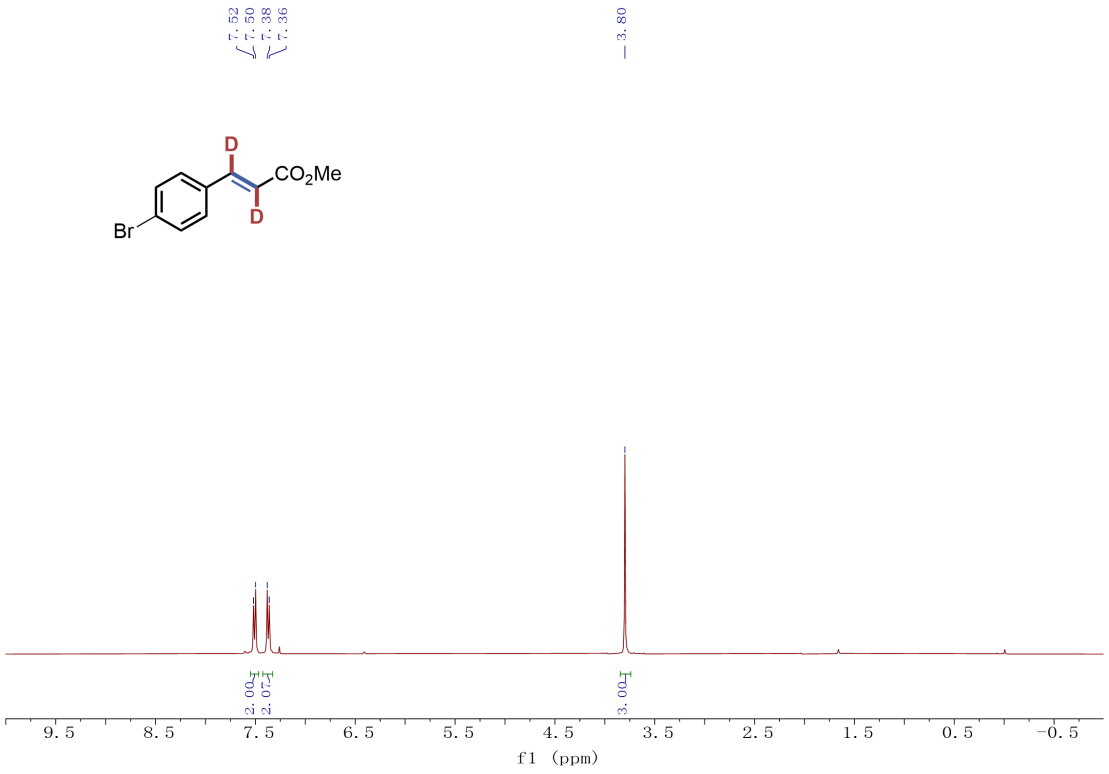


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2c-*d*$_2$**

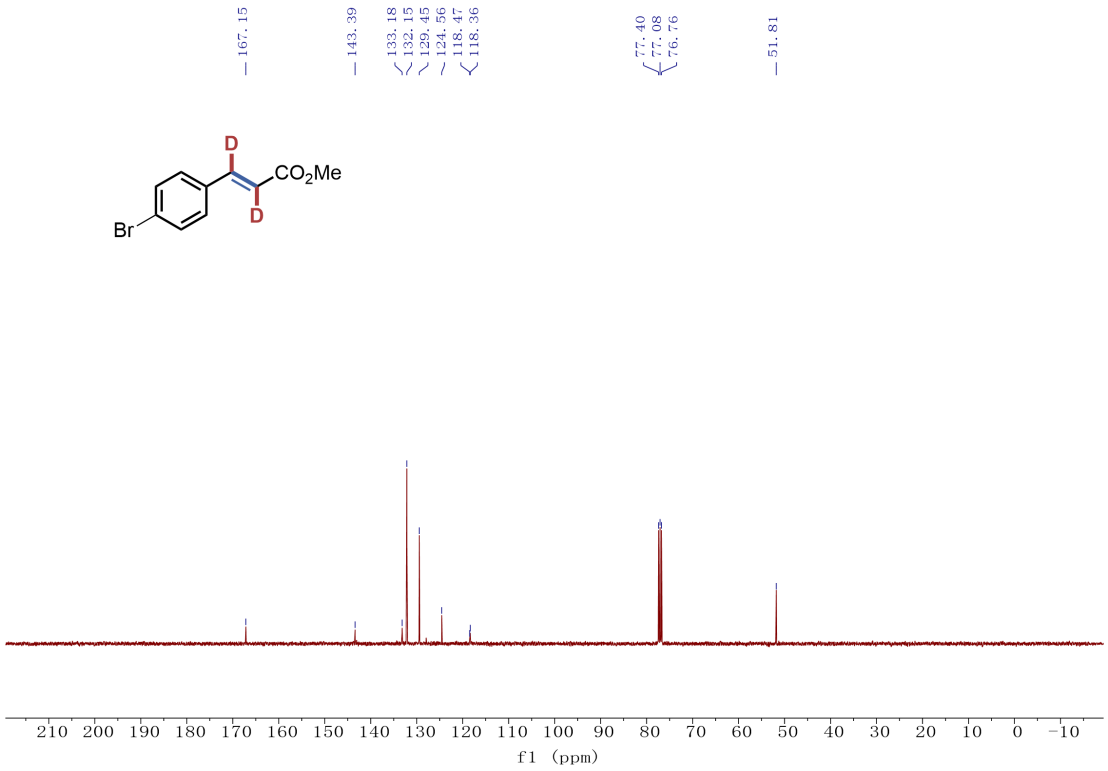


^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2c-*d*$_2$**

methyl (*E*)-3-(4-bromophenyl)acrylate-d$_2$ (2h-*d*$_2$)

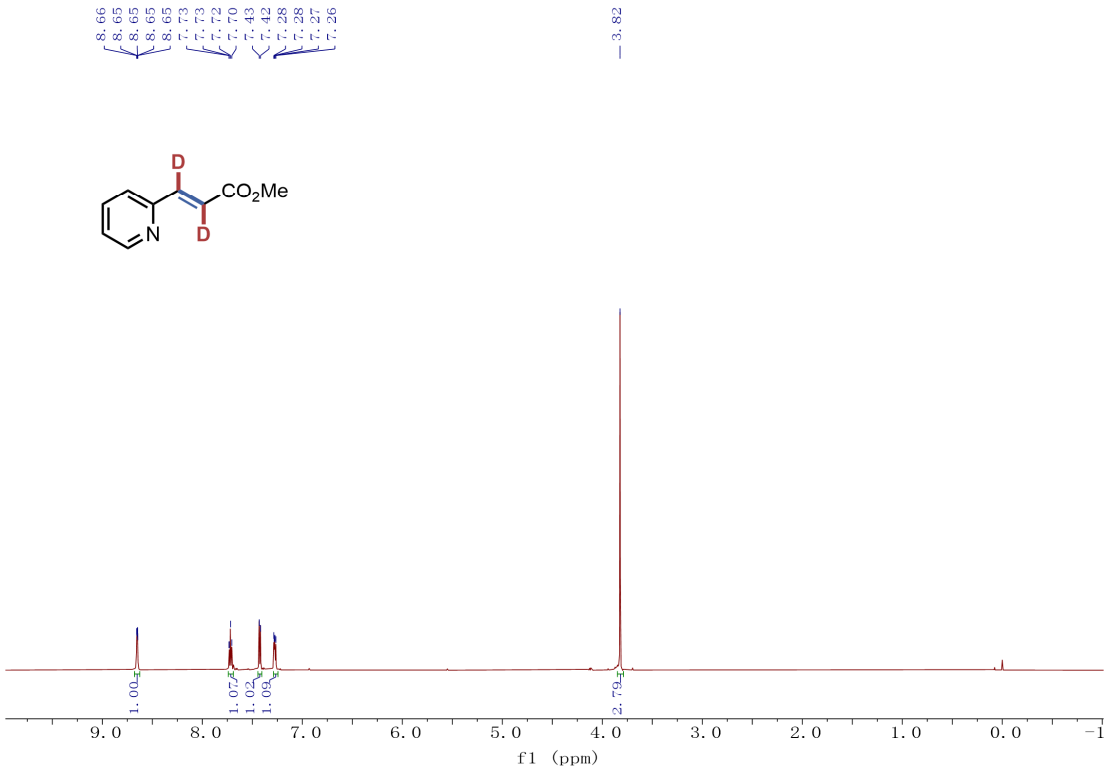


^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2h-*d*$_2$**

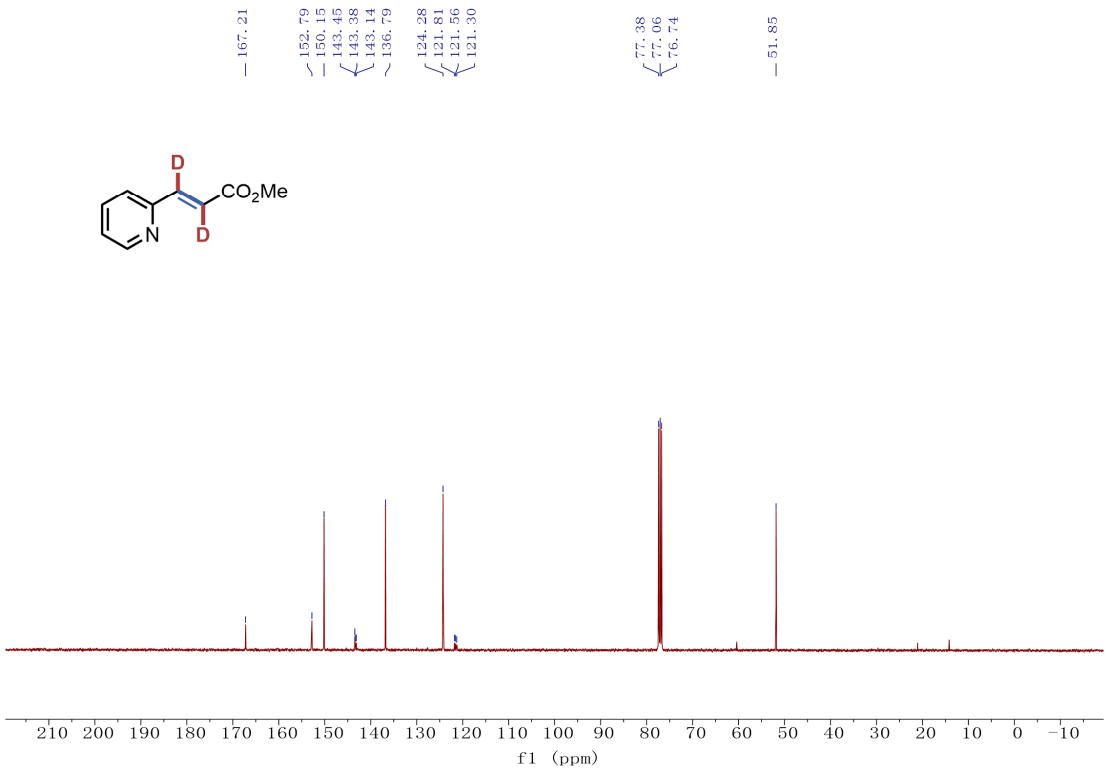


^{13}C NMR ($CDCl_3$, 101 MHz) spectrum for **2h-*d*$_2$**

methyl (*E*)-3-(pyridin-2-yl)acrylate-d$_2$ (2q-*d*$_2$)

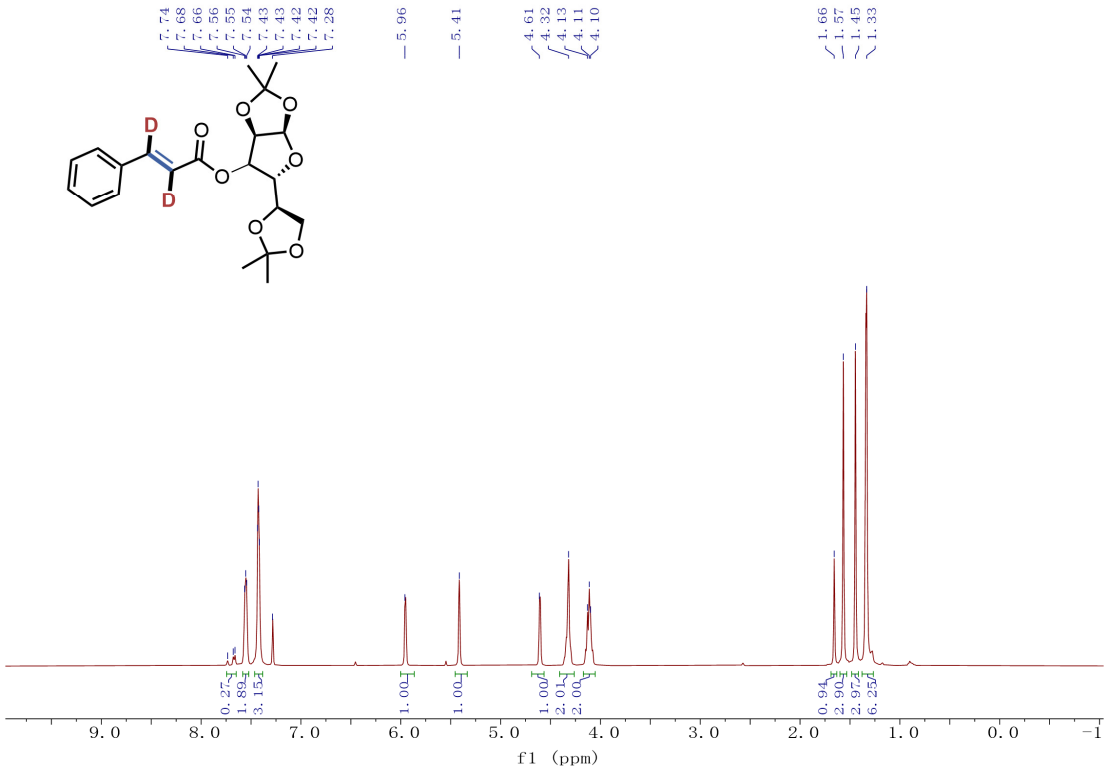


^{1}H NMR ($CDCl_3$, 600 MHz) spectrum for **2q-*d*$_2$**

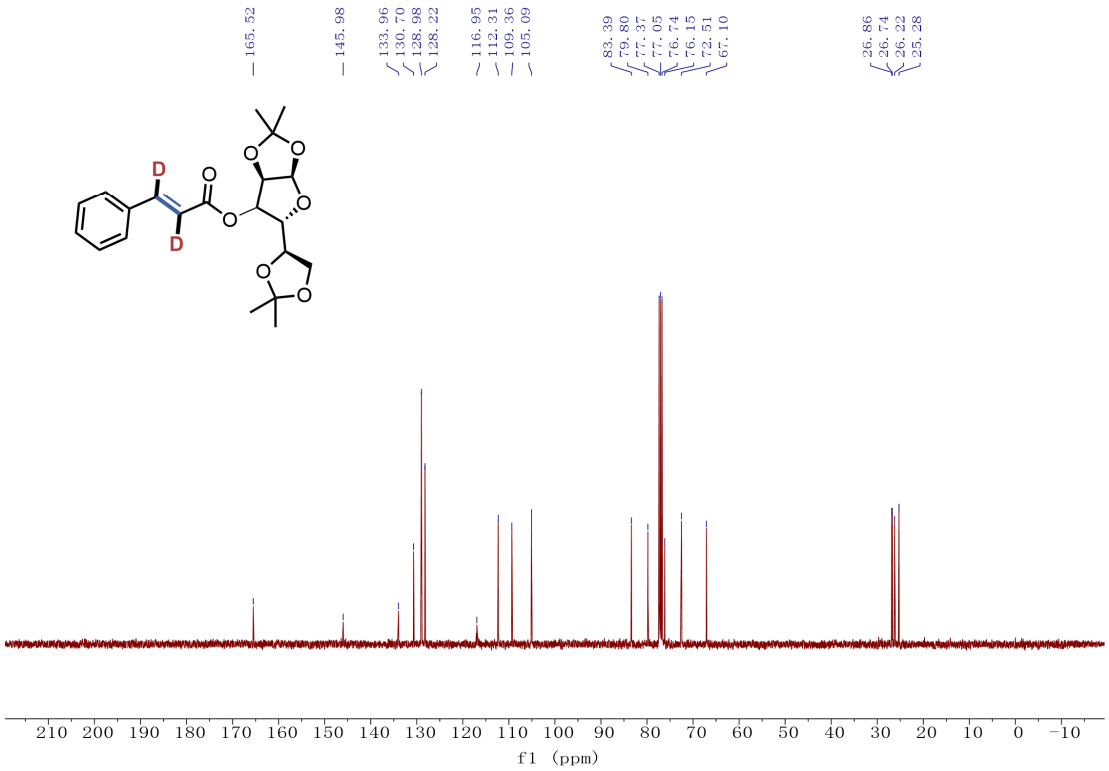


^{13}C NMR (151 MHz, $CDCl_3$) spectrum for **2q-*d*$_2$**

(3aR,5R,6aR)-5-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro [2,3-d][1,3]dioxol-6-yl cinnamate-2,3-d$_2$ (2ar-*d*$_2$)



^{1}H NMR (400 MHz, $CDCl_3$) spectrum for **2ar-*d*$_2$**



^{13}C NMR (101 MHz, $CDCl_3$) spectrum for **2ar-d_2**