Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

for

Chemoselective Catalytic Reduction of Conjugate α,β-Unsaturated Ketones to Saturated Ketones via Hydroboration /Protodeboronation strategy

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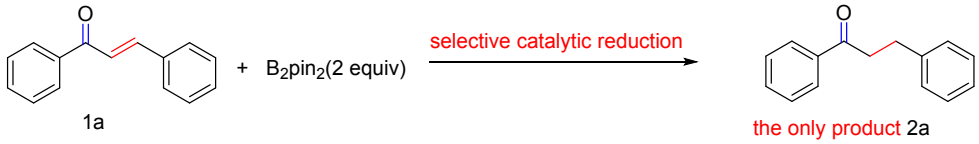
General information

All experiments were conducted with a sealed pressure vessel. Flash column chromatography was performed over silica gel (200-300 mesh). ^{1}H NMR spectra were recorded on a Bruker AVIII-500M spectrometers, Chemical shifts (in ppm) were referenced to $CDCl_3$ (δ = 7.26 ppm) as an internal standard. ^{13}C NMR spectra were obtained by using the same NMR spectrometers and were calibrated with $CDCl_3$ (δ = 77.0 ppm). Unless otherwise noted, materials obtained from commercial suppliers were used without further purification.

Optimization of the reaction conditions

Optimization of the reaction conditions.[a]

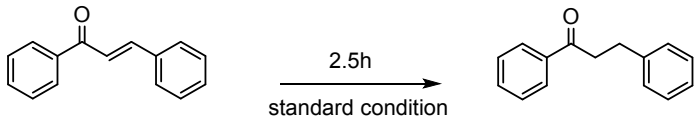


Entry	Catalyst (10 mol %)	base (1.5 equiv)	B_2pin2 (2 equiv)	temp (^{o}C)	solvent (mL)	Time(22 h)	atm	yield[b]
1	CuBr	Cs_2CO_3		90	Toulene (4) THF (1)		O_2	65 %
2	CuBr	Cs_2CO_3		90	Toulene (4) THF (1)		air	86 %[c]
3	CuBr	Cs_2CO_3		90	Toulene (4) THF (1)		N_2	88 %
4	$CuBr_2$	Cs_2CO_3		90	Toulene (4) THF (1)		air	71 %
5	CuI	Cs_2CO_3		90	Toulene (4) THF (1)		air	85 %
6	CuCl	Cs_2CO_3		90	Toulene (4) THF (1)		air	75 %
7	$Cu(OAc)_2$	Cs_2CO_3		90	Toulene (4) THF (1)		air	79 %
8	CuO	Cs_2CO_3		90	Toulene (4) THF (1)		air	trace
9	$Cu(OTf)_2$	Cs_2CO_3		90	Toulene (4) THF (1)		air	83 %
10	$CuCl_2$	Cs_2CO_3		90	Toulene (4) THF (1)		air	68 %
11	CuBr	Cs_2CO_3		90	Toulene (5)		air	38 %
12	CuBr	Cs_2CO_3		90	THF (5)		air	trace
13	CuBr	Cs_2CO_3		90	DMF(5)		air	0 %
14	CuBr	Cs_2CO_3		90	Dioxane(5)		air	41 %
15	CuBr	Cs_2CO_3		90	H_2O(5)		air	24 %
16	$PdCl_2$	Cs_2CO_3		90	Toulene (4) THF (1)		air	48 %
17	$FeCl_2$	Cs_2CO_3		90	Toulene (4) THF (1)		air	trace
18	CuBr	K_2CO_3		90	Toulene (4) THF (1)		air	78 %
19	CuBr	KOAc		90	Toulene (4) THF (1)		air	0 %
20	CuBr	nBuOK		90	Toulene (4) THF (1)		air	31 %
21	CuBr	pyridine		90	Toulene (4) THF (1)		air	0%
22	CuBr	Et_3N		90	Toulene (4) THF (1)		air	0 %

23	CuBr	Cs_2CO_3		80	Toulene (4) THF (1)	1 h	air	73 %
24	CuBr	Cs_2CO_3		70	Toulene (4) THF (1)	1 h	air	70 %
25	CuBr	Cs_2CO_3		90	Toulene (4) THF (1)	1 h	air	88 %
26	-	Cs_2CO_3		90	Toulene (4) THF (1)	1 h	air	0 %
27	CuBr	-		90	Toulene (4) THF (1)	1 h	air	0 %
28	CuBr	Cs_2CO_3	-	90	Toulene (4) THF (1)	1 h	air	0 %

Reaction Conditions: a = chalcone (1a, 0.25 mmol), B_2pin_2 (2a, 2 equiv), Cu salt (10 mol%), base (1.5 equiv), solvent (5 mL), 22 h, temp., corresponding atmosphere. b = GC yield. c = Isolated yield.

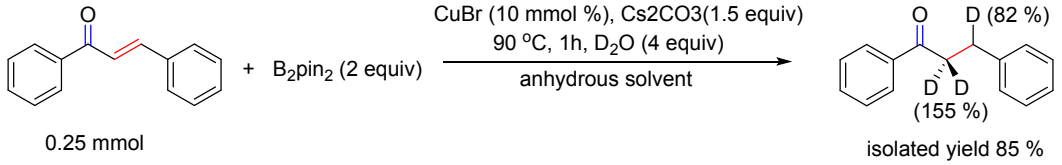
Prepative scale



5 mmol 0.888 g 86 %

A sealed pressure vessel was charged with chalcone (**1a**) (1040 mg, 5 mmol), B_2pin_2 (2560mg, 10 mmol), CuBr (72 mg, 0.5 mmol), Cs_2CO_3 (2420 mg, 7.5mmol), and THF (20 mL), Toluene (100 mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 2.5 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 888.5 mg of 1,3-diphenylpropan-1-one (**2a**) in 86% isolated yield as a white solid.

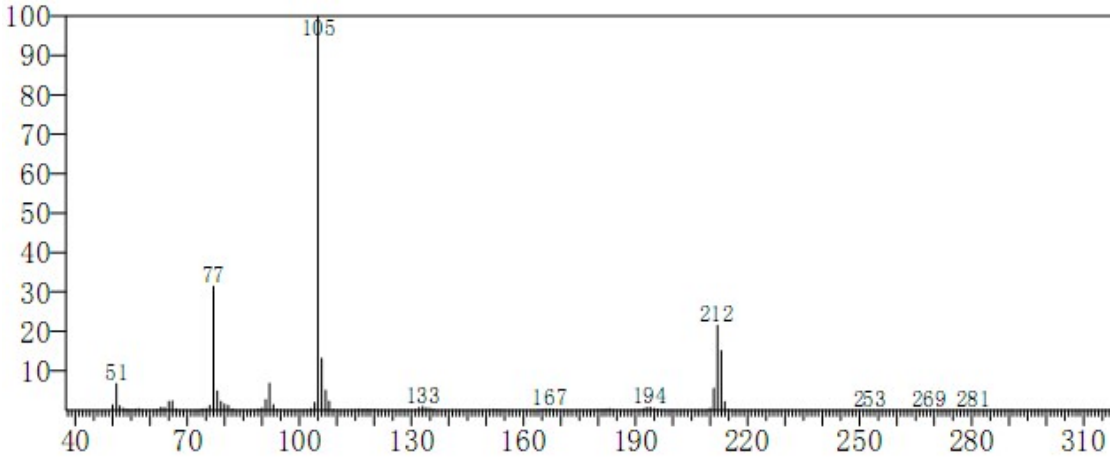
Labeling Experiments



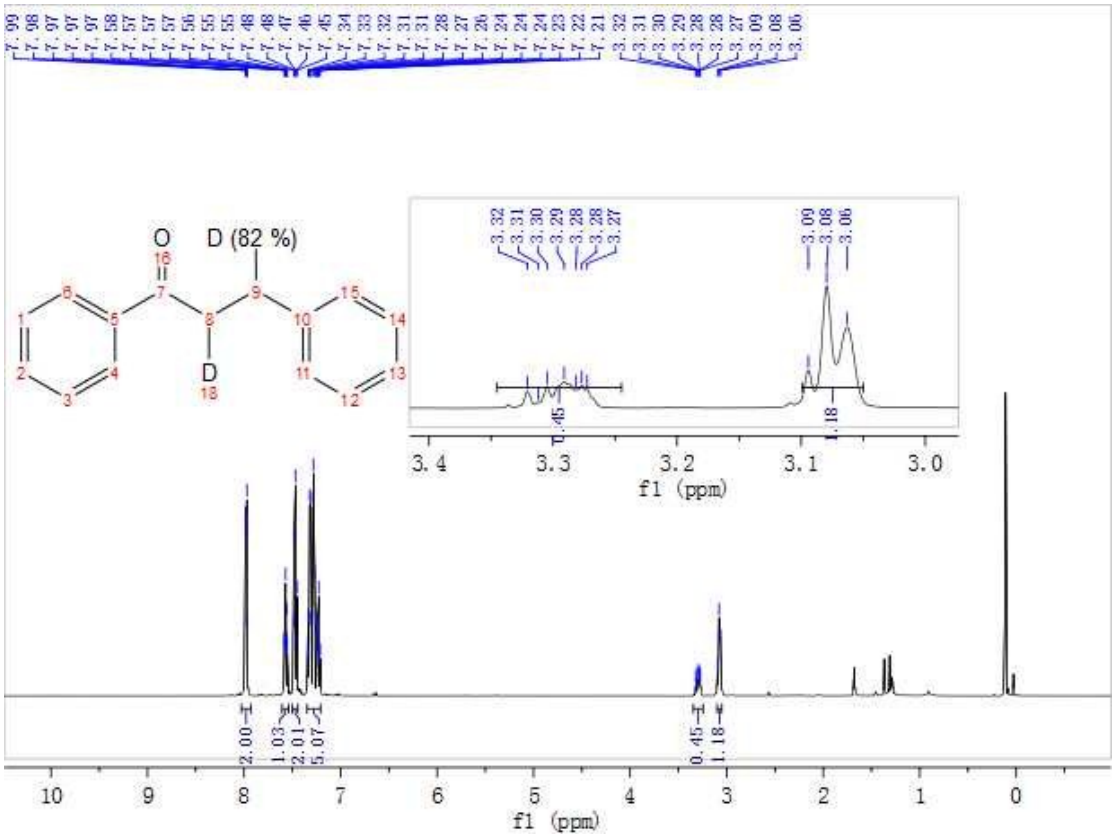
0.25 mmol isolated yield 85 %

A sealed pressure vessel was charged with chalcone (**1a**) (52.0 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.75mmol), and anhydrous THF (1 mL), anhydrous Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 1 hour. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 44.6 mg of the labeled 1,3-diphenylpropan-1-one (**2**) in 85% isolated yield as a white solid.

GC-MS Spectra

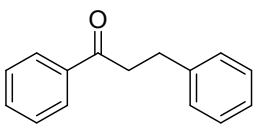


H NMR Spectra



Characterization data for products

1,3-diphenylpropan-1-one (CAS: 1083-30-3)[1]



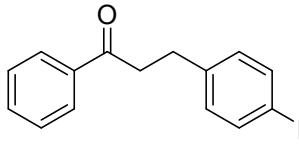
2a

A sealed pressure vessel was charged with chalcone (**1a**) (52.0 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 ^oC under air monitored by TLC and GC for

1 hour. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 47 mg of 1,3-diphenylpropan-1-one (**2a**) in 88% isolated yield as a white solid. mp = 70.1 – 71.9 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 – 7.97 (m, 2H), 7.59 – 7.54 (m, 1H), 7.50 – 7.43 (m, 2H), 7.35 – 7.26 (m, 4H), 7.25 – 7.21 (t, 1H), 3.32 (t, *J* = 7.6Hz, 2H), 3.08 (t, *J* = 7.6Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 199.13 (s), 141.22 (s), 136.78 (s), 132.99 (s), 128.54 (s), 128.46(s), 128.36 (s), 127.97 (s), 126.07 (s), 40.38 (s), 30.06 (s).

3-(4-fluorophenyl)-1-phenylpropan-1-one (CAS: 41865-46-7)[1]

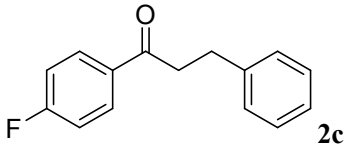


2b

A sealed pressure vessel was charged with (E)-3-(4-fluorophenyl)-1-phenylprop-2-en-1-one (**1b**) (56.5 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 22 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 54.8 mg of 3-(4-fluorophenyl)-1-phenylpropan-1-one (**2b**) in 96% isolated yield as a white solid. mp = 61.2 – 62.1 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97 – 7.95 (m, 2H), 7.59 – 7.53 (m, 1H), 7.50 – 7.40 (m, 2H), 7.25 – 7.17 (m, 2H), 7.02 – 6.94 (m, 2H), 3.28 (t, *J* = 7.6 Hz, 2H), 3.05 (t, *J* = 7.6 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 198.94 (s), 161.33 (d, *J* = 243.8 Hz), 136.83 (d, *J* = 3.2 Hz), 136.73 (s), 133.07 (s), 129.78 (d, *J* = 7.8 Hz), 128.57 (s), 127.95 (s), 115.18 (d, *J* = 21.2 Hz), 40.35 (s), 29.19 (s).

1-(4-fluorophenyl)-3-phenylpropan-1-one (CAS: 41938-64-1)[1]

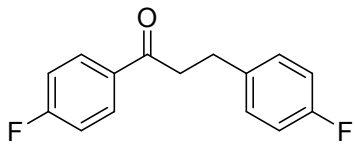


2c

A sealed pressure vessel was charged with (E)-1-(4-fluorophenyl)-3-phenylprop-2-en-1-one (**1c**) (56.5 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 22 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 46.2 mg of 1-(4-fluorophenyl)-3-phenylpropan-1-one (**2c**) in 81% isolated yield as a light yellow liquid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 - 7.97 (m, 2H), 7.33 – 7.21 (m, 5H), 7.14 – 7.11 (m, 2H), 3.28 (t, *J* = 7.6Hz, 2H), 3.07 (t, *J* = 7.6Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.54 (s), 165.66 (d, *J* = 254.6 Hz), 141.09 (s), 133.24 (d, *J* = 3.0 Hz), 130.60 (d, *J* = 9.3 Hz), 128.51 (s), 128.36 (s), 126.15 (s), 115.63 (d, *J* = 21.8 Hz), 40.32 (s), 30.06 (s).

1,3-bis(4-fluorophenyl)propan-1-one (CAS: 104147-29-7)[2]

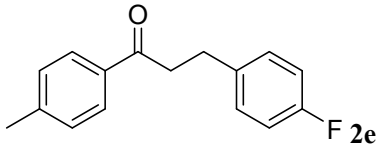


2d

A sealed pressure vessel was charged with (E)-1,3-bis(4-fluorophenyl)prop-2-en-1-one (**1d**) (61.0 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 22 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 55.4 mg of 1,3-bis(4-fluorophenyl)propan-1-one (**2d**) in 90% isolated yield as a white solid. mp = 40.1 – 40.3 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99- 7.96 (m, 2H), 7.21 – 7.18 (m, 2H), 7.13 – 7.10 (m, 2H), 6.99 – 6.95 (m, 2H), 3.25 (t, *J* = 7.6Hz, 2H), 3.04 (t, *J* = 7.6Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.31 (s), 165.69 (d, *J* = 254.8 Hz), 161.36 (d, *J* = 243.9 Hz), 136.69 (d, *J* = 3.2 Hz), 133.18 (d, *J* = 3.0 Hz), 130.58 (d, *J* = 9.3 Hz), 129.77 (d, *J* = 7.8 Hz), 115.65 (d, *J* = 21.9 Hz), 115.21 (d, *J* = 21.2 Hz), 40.28 (s), 29.17 (s).

3-(4-fluorophenyl)-1-(p-tolyl)propan-1-one (CAS: 898767-89-0)[3]

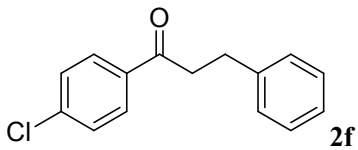


2e

A sealed pressure vessel was charged with (E)-3-(4-fluorophenyl)-1-(p-tolyl)prop-2-en-1-one (**1e**) (61.0 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 22 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 44.5 mg of 3-(4-fluorophenyl)-1-(p-tolyl)propan-1-one (**2e**) in 73% isolated yield as a white solid. mp = 69.2-70.3 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88 – 7.86 (m, 1H), 7.27 – 7.20 (m, 4H), 7.00 - 6.97 (m, 2H), 3.26 (t, *J* = 7.6 Hz, 2H), 3.05 (t, *J* = 7.6 Hz, 2H), 2.42 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 198.62 (s), 161.31 (d, *J* = 243.7 Hz), 143.86 (s), 136.93 (d, *J* = 3.2 Hz), 134.27 (s), 129.76 (d, *J* = 7.8 Hz), 129.24 (s), 128.08 (s), 115.15 (d, *J* = 21.1 Hz), 40.23 (s), 29.29 (s), 21.57 (s).

1-(4-chlorophenyl)-3-phenylpropan-1-one (CAS: 5739-37-7)[1]



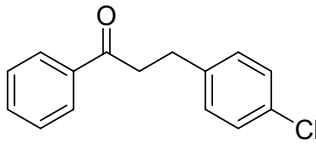
2f

A sealed pressure vessel was charged with (E)-1-(4-chlorophenyl)-3-phenylprop-2-en-1-one (**1f**) (60.5 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 8 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator

and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 55.5 mg of 1-(4-chlorophenyl)-3-phenylpropan-1-one (**2f**) in 91% isolated yield as a white solid. mp = 78.1 – 78.4 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 – 7.78 (m, 2H), 7.34 – 7.32 (m, 2H), 7.23 – 7.20 (m, 2H), 7.17 – 7.11 (m, 3H), 3.18 (t, *J* = 7.7 Hz, 1H), 2.97 (t, *J* = 7.7 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.89 (s), 141.00 (s), 139.43 (s), 135.09 (s), 129.40 (s), 128.86 (s), 128.51 (s), 128.35 (s), 126.17 (s), 40.37 (s), 29.99 (s).

3-(4-chlorophenyl)-1-phenylpropan-1-one (CAS: 5739-39-9)[1]



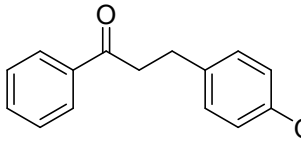
2g

A sealed pressure vessel was charged with (E)-3-(4-chlorophenyl)-1-phenylprop-2-en-1-one (**1g**) (60.5 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 1 hour. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 47.6 mg of 3-(4-chlorophenyl)-1-phenylpropan-1-one (**2g**) in 78% isolated yield as a white solid. mp = 58.3 – 58.8 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89 – 7.81 (m, 2H), 7.50 – 7.43 (m, 1H), 7.40 – 7.32 (m, 2H), 7.16 (dt, *J* = 7.1, 2.2 Hz, 2H), 7.12 – 7.05 (m, 2H), 3.19 (t, *J* = 7.5 Hz, 2H), 2.95 (t, *J* = 7.5 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 198.76 (s), 139.68 (s), 136.67 (s), 133.11 (s), 131.79 (s), 129.77 (s), 128.58 (s), 128.53 (s), 127.95 (s), 40.07 (s), 29.31 (s).

3-(4-methoxyphenyl)-1-phenylpropan-1-one (CAS: 1669-49-4)[1]

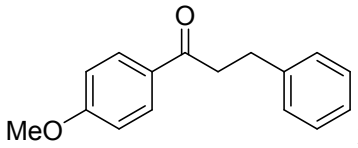


2h

A sealed pressure vessel was charged with (E)-3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one (**1h**) (59.5 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 22 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:10) to give 37.8 mg of 3-(4-methoxyphenyl)-1-phenylpropan-1-one (**2h**) in 63% isolated yield as a white solid. mp = 59.3 – 60.1 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 – 7.90 (m, 2H), 7.59 – 7.52 (m, 1H), 7.50 – 7.40 (m, 2H), 7.18 (d, *J* = 8.6 Hz, 2H), 6.88 – 6.80 (m, 2H), 3.79 (s, 3H), 3.28 (t, *J* = 7.5 Hz, 2H), 3.02 (t, *J* = 7.5 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 199.34 (s), 157.94 (s), 136.85 (s), 133.26 (s), 132.99 (s), 129.31 (s), 128.55 (s), 127.99 (s), 113.89 (s), 55.23 (s), 40.67 (s), 29.24 (s).

1-(4-methoxyphenyl)-3-phenylpropan-1-one (CAS: 5739-38-8)[1]

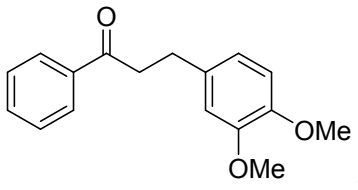


2i

A sealed pressure vessel was charged with (E)-1-(4-methoxyphenyl)-3-phenylprop-2-en-1-one (**1i**) (59.5 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 22 hour. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:10) to give 43.2 mg of 1-(4-methoxyphenyl)-3-phenylpropan-1-one (**2i**) in 72% isolated yield as a white solid. mp = 95.7 – 98.1 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 – 7.92 (m, 2H), 7.34 – 7.18 (m, 5H), 6.96 – 6.90 (m, 2H), 3.87 (s, 3H), 3.26 (t, *J* = 7.5 Hz, 2H), 3.07 (t, *J* = 7.5 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.74 (s), 163.38 (s), 141.41 (s), 130.24 (s), 129.89 (s), 128.44(s) , 128.37(s), 126.02 (s), 113.66 (s), 55.40 (s), 40.06 (s), 30.27 (s).

3-(3,4-dimethoxyphenyl)-1-phenylpropan-1-one (CAS: 7468-58-8)[4]

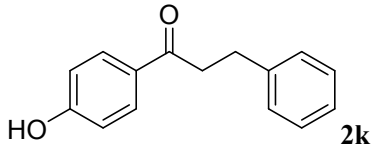


2j

A sealed pressure vessel was charged with (E)-3-(3,4-dimethoxyphenyl)-1-phenylprop-2-en-1-one (**1j**) (67.0 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 22 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:3) to give 44.6 mg of 3-(3,4-dimethoxyphenyl)-1-phenylpropan-1-one (**2j**) in 66% isolated yield as a white solid. mp = 68.5 - 69.2 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 – 7.91 (m, 2H), 7.59 – 7.51 (m, 1H), 7.44 (dd, *J* = 10.6, 4.7 Hz, 2H), 6.84 – 6.73 (m, 3H), 3.86 (s, 3H), 3.85 (s, 3H), 3.28 (t, *J* = 7.6 Hz, 2H), 3.02 (t, *J* = 7.6 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 199.30 (s), 148.82 (s), 147.31 (s), 136.82 (s), 133.82 (s), 132.98 (s), 128.53 (s), 127.95 (s), 120.10 (s), 111.76 (s), 111.26 (s), 55.86(s), 55.76 (s), 40.62 (s), 29.74 (s).

1-(4-hydroxyphenyl)-3-phenylpropan-1-one (CAS: 36941-00-1)[5]



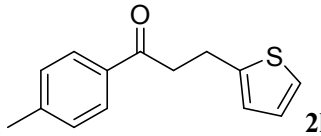
2k

A sealed pressure vessel was charged with (E)-1-(4-hydroxyphenyl)-3-phenylprop-2-en-1-one (**1k**) (56.0 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 22 hours. Upon completion of the reaction, the solvents were

cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:5) to give 25.5 mg of 1-(4-hydroxyphenyl)-3-phenylpropan-1-one (**2k**) in 45% isolated yield as a white solid. mp = 73.6 – 74.5 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 – 7.86 (m, 2H), 7.31 – 7.19 (m, 5H), 6.92 – 6.84 (m, 2H), 6.63 (s, 1H), 3.26 (t, *J* = 7.6 Hz, 2H), 3.06 (t, *J* = 7.6 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 198.61 (s), 160.48 (s), 141.24 (s), 130.72 (s), 129.75 (s), 128.50(s), 128.38 (s), 126.11 (s), 115.41 (s), 40.10 (s), 30.41 (s).

3-(thiophen-2-yl)-1-(p-tolyl)propan-1-one (CAS: 654674-60-9)[6]

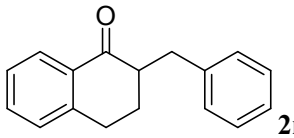


2l

A sealed pressure vessel was charged with (E)-3-(thiophen-2-yl)-1-(p-tolyl)prop-2-en-1-one (**1l**) (57.0 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 8 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 49.5 mg of 3-(thiophen-2-yl)-1-(p-tolyl)propan-1-one (**2l**)in 86% isolated yield as a white solid. mp = 37.5 – 38.1 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 – 7.79 (m, 2H), 7.27 – 7.24 (m, 2H), 7.13 (dd, *J* = 5.1, 1.2 Hz, 1H), 6.92 (dd, *J* = 5.1, 3.4 Hz, 1H), 6.90 – 6.80 (m, 1H), 3.36 – 3.33 (m, 2H), 3.30 – 3.27 (m, 2H), 2.41 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 198.21 (s), 143.97(s), 143.93 (s), 134.23 (s), 129.27 (s), 128.12 (s), 126.80 (s), 124.59 (s), 123.30 (s), 40.39 (s), 24.25 (s), 21.62 (s).

2-benzyl-3,4-dihydronaphthalen-1(2H)-one (CAS: 27019-08-5)[7]

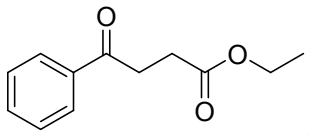


2m

A sealed pressure vessel was charged with (E)-2-benzylidene-3,4-dihydronaphthalen-1(2H)-one (**1m**) (58.5 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 8 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 46 mg of 2-benzyl-3,4-dihydronaphthalen-1(2H)-one (**2m**) in 78% isolated yield as a white solid. mp = 53.4 – 54.1 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (dd, *J* = 7.9, 1.1 Hz, 1H), 7.49 – 7.45 (m, 1H), 7.35 – 7.29 (m, 3H), 7.26 – 7.21 (m, 4H), 3.51 (dd, *J* = 13.7, 4.1 Hz, 1H), 2.98 – 2.89 (m, 2H), 2.79- 2.73 (m, 1H), 2.66 (dd, *J* = 13.7, 9.6 Hz, 1H), 2.15 – 2.09 (m, 1H), 1.84 – 1.76 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 199.31 (s), 143.97 (s), 139.98 (s), 133.21 (s), 132.40 (s), 129.20 (s), 128.66 (s), 128.34 (s), 127.48 (s), 126.56 (s), 126.07 (s), 49.39 (s), 35.62 (s), 28.57 (s), 27.61 (s).

ethyl 4-oxo-4-phenylbutanoate (CAS: 6270-17-3)[9]

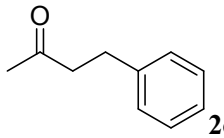


2n

A sealed pressure vessel was charged with ethyl (E)-4-oxo-4-phenylbut-2-enoate (**1n**) (51.0 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 1 hour. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 38.1 mg of ethyl 4-oxo-4-phenylbutanoate (**2n**) in 74% isolated yield as a white solid. mp = 165.1 – 166.1 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 – 7.89 (m, 2H), 7.54 (dd, *J* = 10.7, 4.1 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 4.14 (q, *J* = 7.1 Hz, 2H), 3.29 (t, *J* = 6.6 Hz, 2H), 2.73 (t, *J* = 6.6 Hz, 2H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 198.08 (s), 172.84 (s), 136.52 (s), 133.14 (s), 128.54 (s), 127.97 (s), 60.59 (s), 33.33 (s), 28.24 (s), 14.14 (s).

4-phenylbutan-2-one (CAS: 2550-26-7)[2]



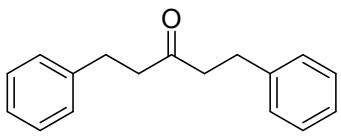
2o

A sealed pressure vessel was charged with (E)-4-phenylbut-3-en-2-one (**1o**) (36.5 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 22 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 30.7 mg of 4-phenylbutan-2-one (**2o**) in 83% isolated yield as a colorless liquid.

A sealed pressure vessel was charged with 4-phenylbut-3-yn-2-one (**1r**) (36 mg, 0.25 mmol), B_2pin_2 (254mg, 1mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (242 mg, 0.75mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 22 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 26 mg of 4-phenylbutan-2-one (**2o**) in 71% isolated yield as a colorless liquid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (dd, *J* = 8.2, 6.8 Hz, 2H), 7.22 – 7.16 (m, 3H), 2.90 (t, *J* = 7.6 Hz, 2H), 2.76 (t, *J* = 7.6 Hz, 2H), 2.14 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 207.91 (s), 140.93 (s), 128.44 (s), 128.23 (s), 126.06 (s), 45.12 (s), 30.03 (s), 29.68 (s).

1,5-diphenylpentan-3-one (CAS: 5396-91-8)[10]

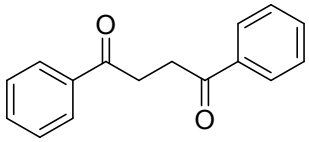


2p

A sealed pressure vessel was charged with (1E,4E)-1,5-diphenylpenta-1,4-dien-3-one (**1p**) (58.5 mg, 0.25 mmol), B_2pin_2 (254mg, 1mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (242 mg, 0.75mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 22 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 37.5 mg of 1,5-diphenylpentan-3-one (**2p**) in 63% isolated yield as a colorless liquid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (dd, *J* = 10.3, 4.6 Hz, 4H), 7.22 – 7.16 (m, 6H), 2.91 (t, *J* = 7.6 Hz, 4H), 2.73 (t, *J* = 7.6 Hz, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 209.07 (s), 140.95 (s), 128.45 (s), 128.26 (s), 126.06 (s), 44.46 (s), 29.68 (s).

1,4-diphenylbutane-1,4-dione (CAS: 495-71-6)[8]

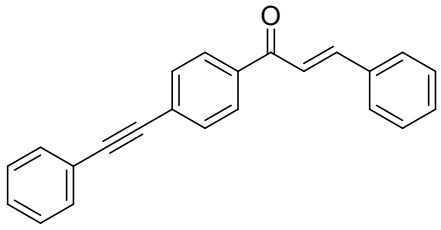


2q

A sealed pressure vessel was charged with (E)-1,4-diphenylbut-2-ene-1,4-dione (**1q**) (52.0 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 1 hour. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 44 mg of 1,4-diphenylbutane-1,4-dione (**2q**) in 74% isolated yield as a white solid. mp = 144.1 – 145 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10 – 7.98 (m, 4H), 7.61 – 7.53 (m, 2H), 7.52 – 7.43 (m, 4H), 3.47 (s, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 198.63 (s), 136.71 (s), 133.11 (s), 128.55 (s), 128.07 (s), 32.54 (s).

(E)-3-phenyl-1-(4-(phenylethynyl)phenyl)prop-2-en-1-one (new compound)



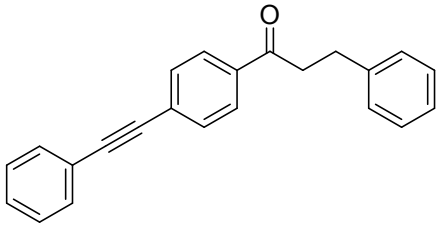
1s

(E)-3-phenyl-1-(4-(phenylethynyl)phenyl)prop-2-en-1-one was prepared from Boumendje's work[11] with a little change. A sealed pressure vessel was charged with 1-(4-(phenylethynyl)phenyl)ethan-1-one (440mg, 2mmol), benzaldehyde(212mg, 2mmol), NaOH(8mg, 0.2mmol), MeOH(10 mL), The resulting solution was stirred at room temperature for overnight. Upon completion of the reaction, the solvents were filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to get the product (E)-3-phenyl-1-(4-(phenylethynyl)phenyl)prop-2-en-1-one (**1s**) as a brown solid. mp = 132.3 – 133.6 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05 – 8.00 (m, 2H), 7.84 (d, *J* = 15.7 Hz, 1H), 7.70 – 7.63 (m, 4H), 7.59 – 7.52 (m, 3H), 7.44 – 7.36 (m, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 189.42 (s), 145.02 (s),

137.27 (s), 134.74 (s), 131.70 (s), 130.60 (s), 128.92 (s), 128.74 (s), 128.53 – 128.29 (m), 127.84 (s), 122.63 (s), 121.63 (s), 92.66 (s), 88.71 (s). HRMS m/z (EI) calcd for $(M+H)^+$, 308.1201 ,found 309.1274.

3-phenyl-1-(4-(phenylethynyl)phenyl)propan-1-one (new compound)

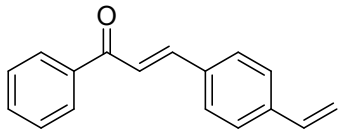


2s

A sealed pressure vessel was charged with (E)-3-phenyl-1-(4-(phenylethynyl)phenyl)prop-2-en-1-one (**1s**) (77.0 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 ^oC under air monitored by TLC and GC for 3 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 47.2 mg of 3-phenyl-1-(4-(phenylethynyl)phenyl)propan-1-one (**2s**) in 61% isolated yield as a white solid. mp = 115.4 – 116. 7 ^oC

^1H NMR (500 MHz, $CDCl_3$) δ 7.98 – 7.90 (m, 2H), 7.62 – 7.54 (m, 4H), 7.39 – 7.35 (m, 3H), 7.31 (dd, J = 10.2, 4.6 Hz, 2H), 7.28 – 7.25 (m, 2H), 7.22 (dd, J = 10.1, 4.3 Hz, 1H), 3.31 (t, J = 7.6 Hz, 2H), 3. 08 (t, J = 7.6 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 198.40 (s), 141.14 (s), 135.87 (s), 131.72, 131.70 (s), 128.79 (s), 128.53(s) , 128.42 (s), 128.40(s), 128.13 (s), 127.98(s), 126.16 (s), 122.62 (s), 92.69 (s), 88.58 (s), 40.49 (s), 30.08 (s). HRMS m/z (EI) calcd for $(M+H)^+$, 311.1436 ,found 311.1430.

(E)-1-phenyl-3-(4-vinylphenyl)prop-2-en-1-one (CAS: 25917-08-2)

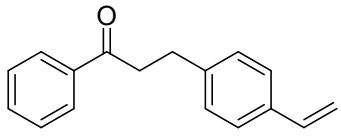


1t

(E)-1-phenyl-3-(4-vinylphenyl)prop-2-en-1-one was prepared from Brown's[12] work with a little change. A sealed pressure vessel was charged with a solution of potassium vinyltrifluoroborate (134 mg, 1.00 mmol), PdCl2 (3.5 mg, 0.02 mmol), PPh3 (16 mg, 0.06 mmol), Cs2CO3(978 mg, 3.00 mmol), and (E)-3-(4-bromophenyl)-1-phenylprop-2-en-1-one (286 mg, 1.00 mmol) in THF/H2O (9:1) (2 mL) was heated at 85 °C under an N2 atmosphere in a sealed tube. The reaction mixture was stirred at 85 °C for 22 h, then cooled to room temperature and diluted with H2O (3 mL) followed by extraction with CH2Cl2 (10 mL × 3). The solvent was removed in vacuo, and the crude product was purified by silica gel chromatography (eluting with 20:1 n-pentane ether) to yield (E)-1-phenyl-3-(4-vinylphenyl)prop-2-en-1-one as a white solid (187 mg, 0.8 mmol, 80%)

^1H NMR (400 MHz, $CDCl_3$) δ 8.09 – 8.01 (m, 2H), 7.82 (d, J = 15.7 Hz, 1H), 7.69 – 7.43 (m, 8H), 6.76 (dd, J = 17.6, 10.9 Hz, 1H), 5.86 (d, J = 17.6 Hz, 1H), 5.37 (d, J = 11.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.51 (s), 144.36 (s), 139.82 (s), 138.32 (s), 136.15 (s), 134.38 (s), 132.74 (s), 128.76 (s) , 128.63 (s), 128.50 (s), 126.77 (s), 121.85 (s), 115.45 (s).

1-phenyl-3-(4-vinylphenyl)propan-1-one (CAS: 93318-87-7)

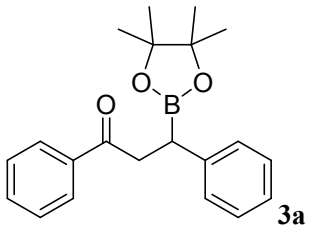


2t

(E)-1-phenyl-3-(4-vinylphenyl)prop-2-en-1-one (**1t**) (58.5 mg, 0.25 mmol), B_2pin_2 (127mg, 0.5mmol), CuBr (3.6 mg, 0.025 mmol), Cs_2CO_3 (121 mg, 0.375mmol), and THF (1 mL), Toluene(5mL). The resulting solution was stirred at 90 °C under air monitored by TLC and GC for 8 hours. Upon completion of the reaction, the solvents were cooled to room temperature, filtered through a pad of celite, then removed via rotary evaporator and the residue was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether=1:30) to give 41.8 mg of 1-phenyl-3-(4-vinylphenyl)propan-1-one (**2t**) in 72% isolated yield as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 – 7.89 (m, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.38 (d, *J* = 8.1 Hz, 2H), 7.25 (d, *J* = 8.1 Hz, 2H), 6.73 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.74 (dd, *J* = 17.6, 0.5 Hz, 1H), 5.24 (d, *J* = 11.3 Hz, 1H), 3.32 (t, *J* = 7.6 Hz, 2H), 3.10 (t, *J* = 7.6 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.14 (s), 141.01 (s), 136.92 (s), 136.60 (s), 135.66 (s), 133.06 (s), 128.61 (s), 128.05 (s), 126.39 (s), 113.22 (s), 40.31 (s), 29.87 (s).

1,3-diphenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (CAS: 199999-46-7)[13]



3a

1,3-diphenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (**4**) was prepared from Taku's work.[12] white solid. mp = 74.5 – 75.4 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 – 7.95 (m, 2H), 7.54 (dd, *J* = 10.5, 4.3 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.35 – 7.26 (m, 4H), 7.21 – 7.12 (m, 1H), 3.57 (dd, *J* = 18.3, 10.9 Hz, 1H), 3.43 (dd, *J* = 18.3, 5.0 Hz, 1H), 2.81 (dd, *J* = 10.9, 5.0 Hz, 1H), 1.26 (s, 6H), 1.18 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 199.63 (s), 141.90 (s), 136.73 (s), 132.88 (s), 128.47 (s), 128.44 (s), 128.34 (s) , 128.00 (s), 125.55 (s), 83.33 (s), 43.23 (s), 24.53 (s), 24.49 (s).

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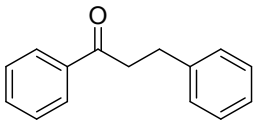
NMR Spectra

1,3-diphenylpropan-1-one (CAS: 1083-30-3)[1]



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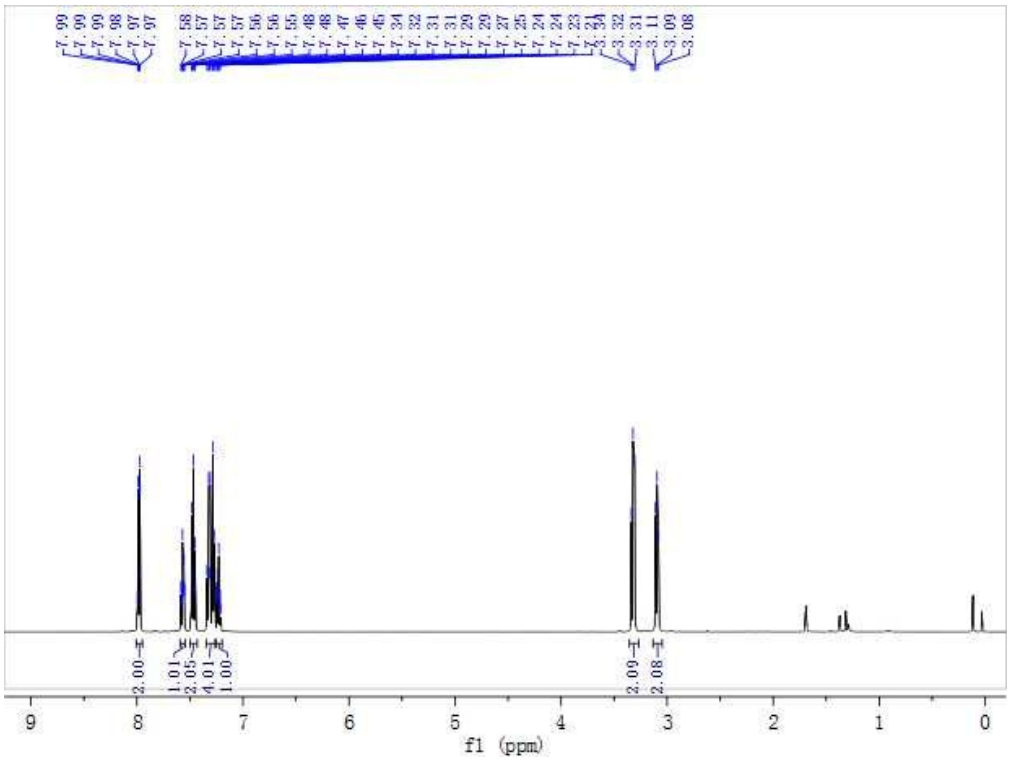

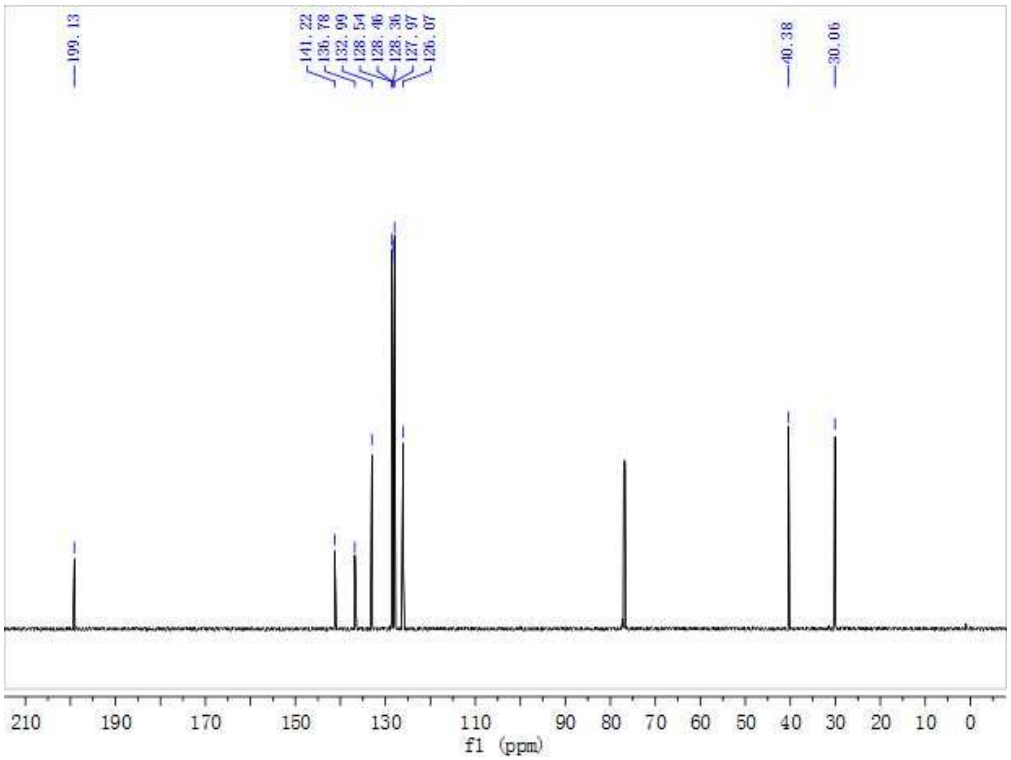

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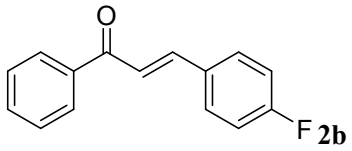

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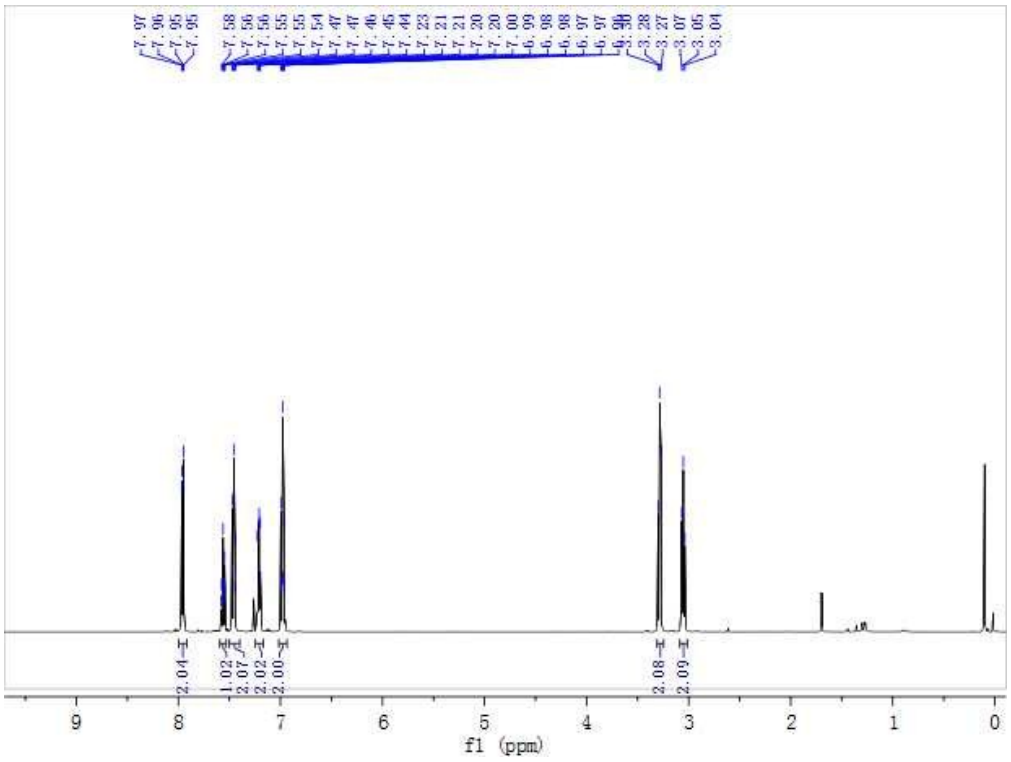

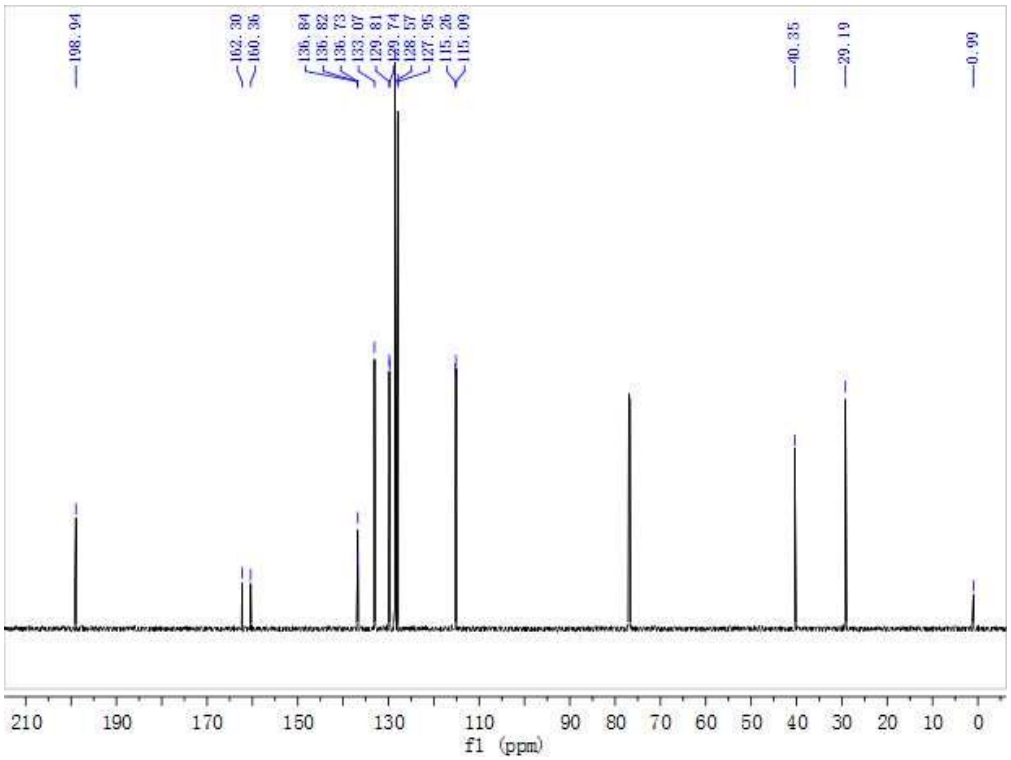

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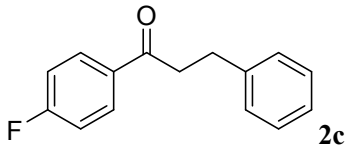

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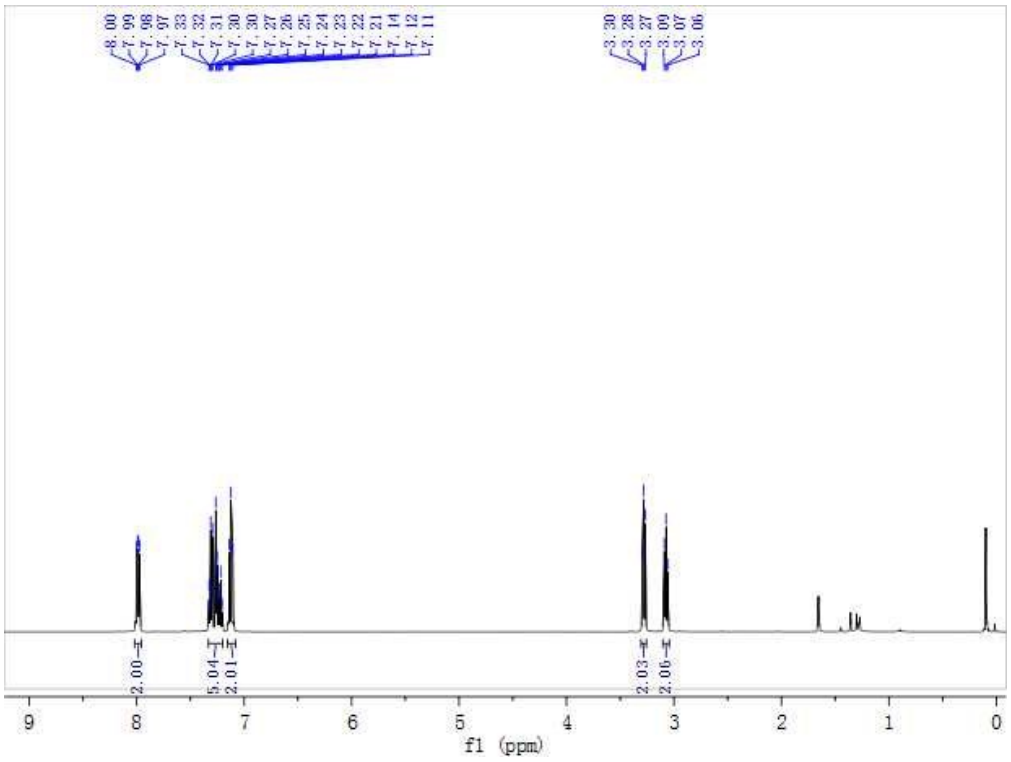

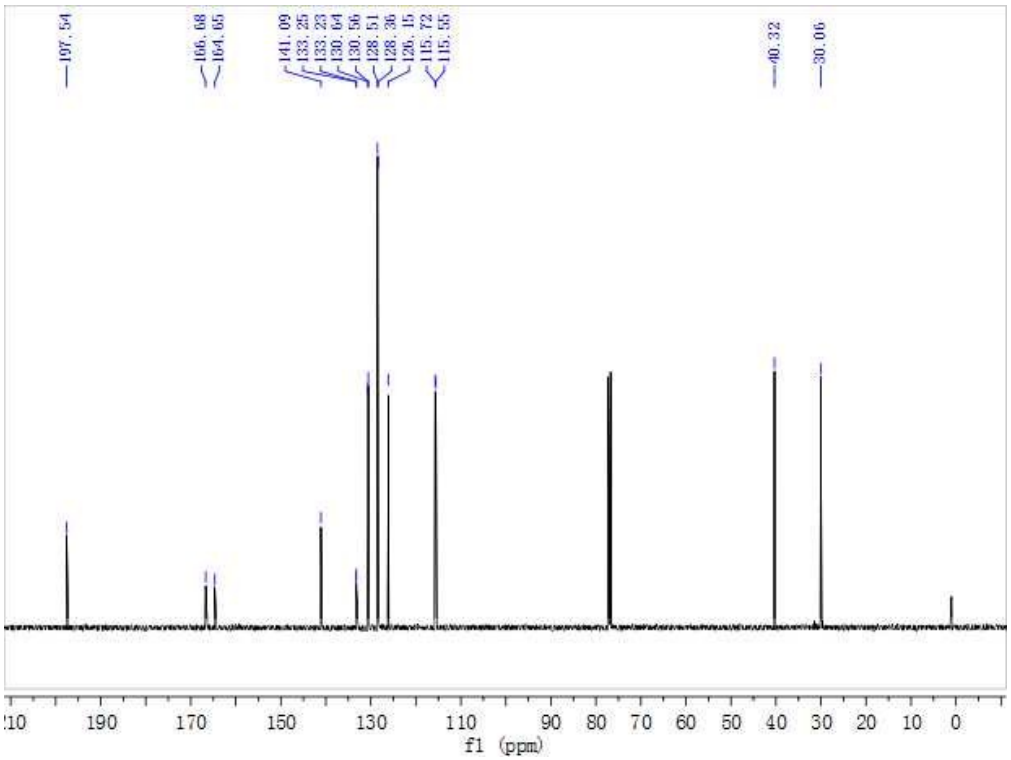

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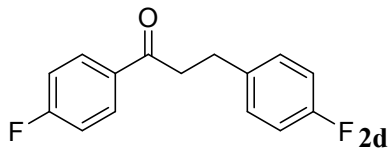

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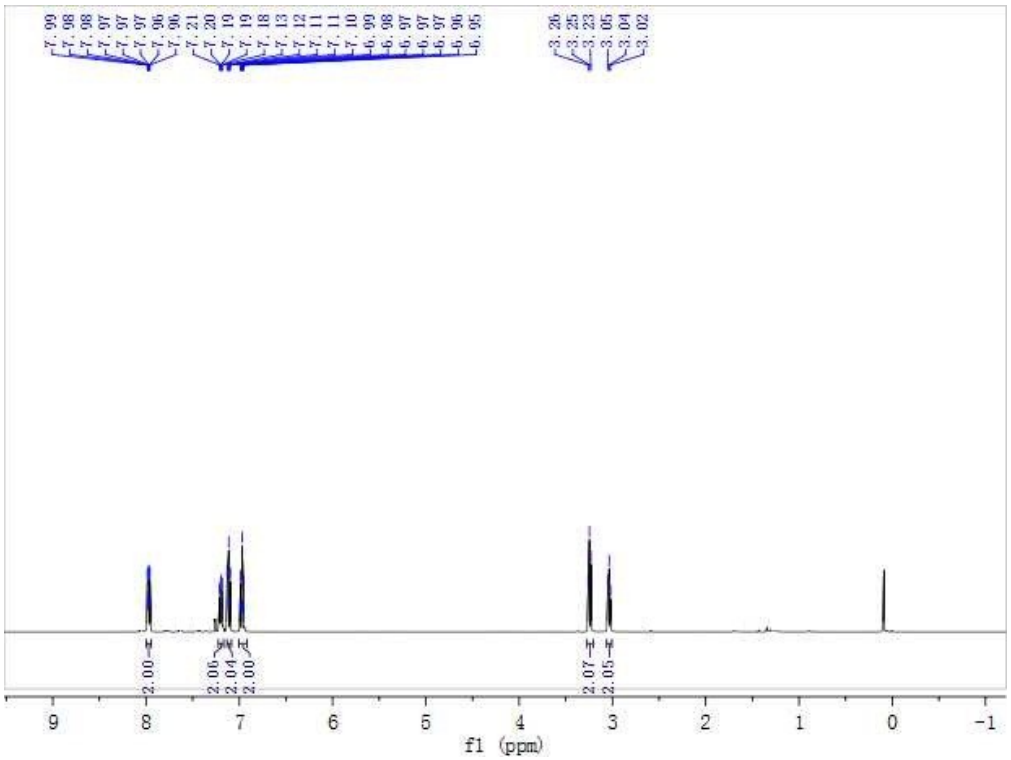

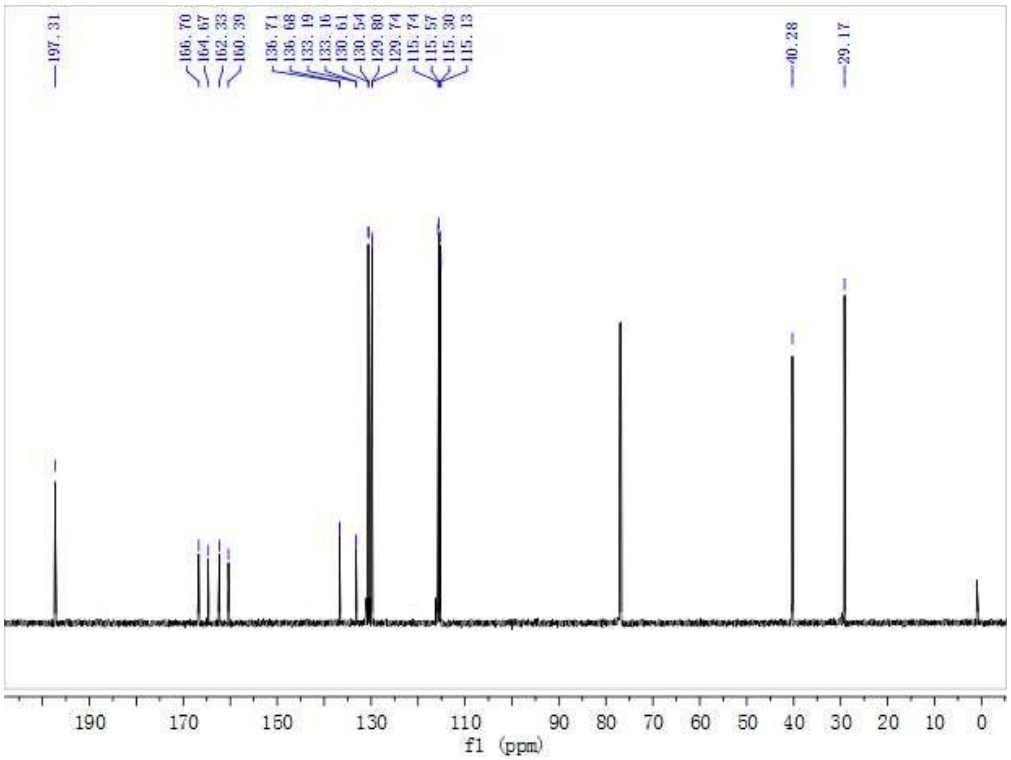

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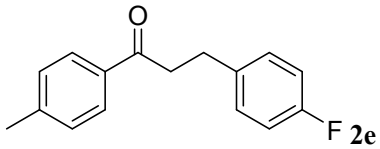

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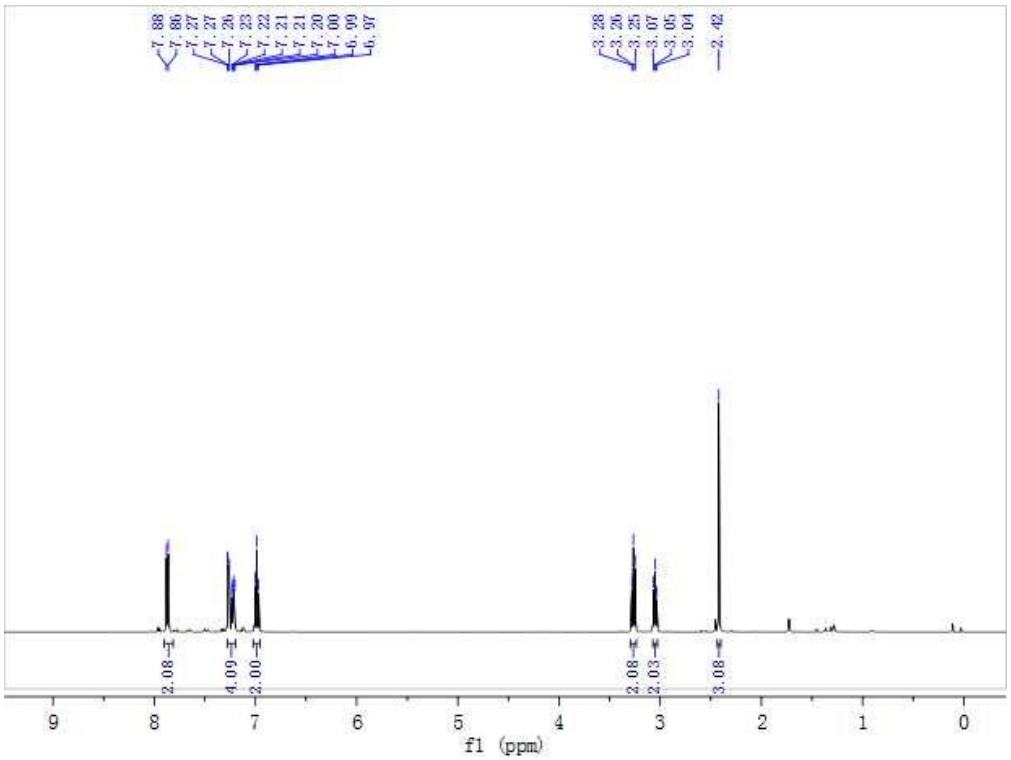

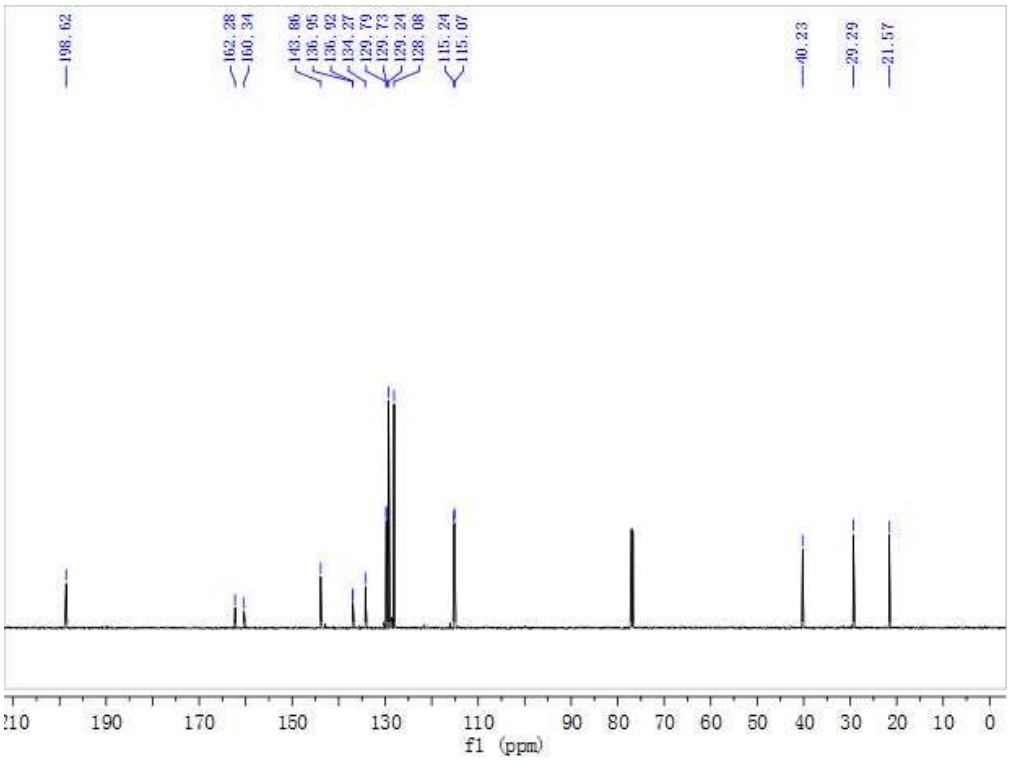

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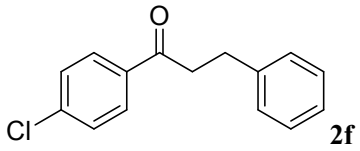

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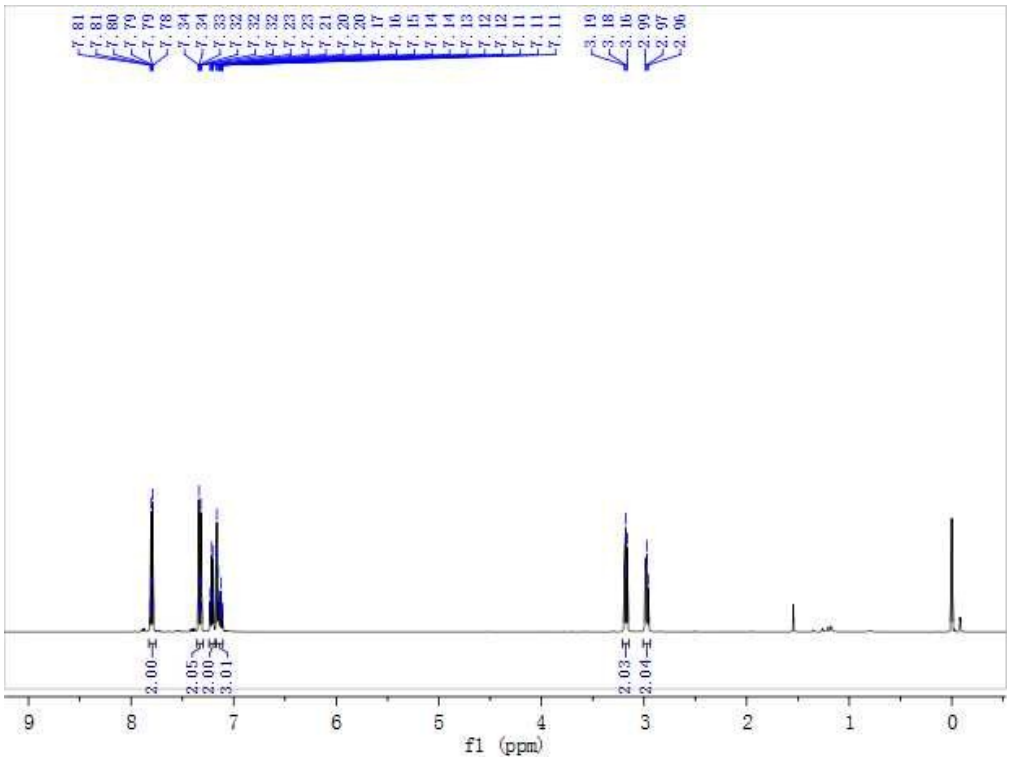

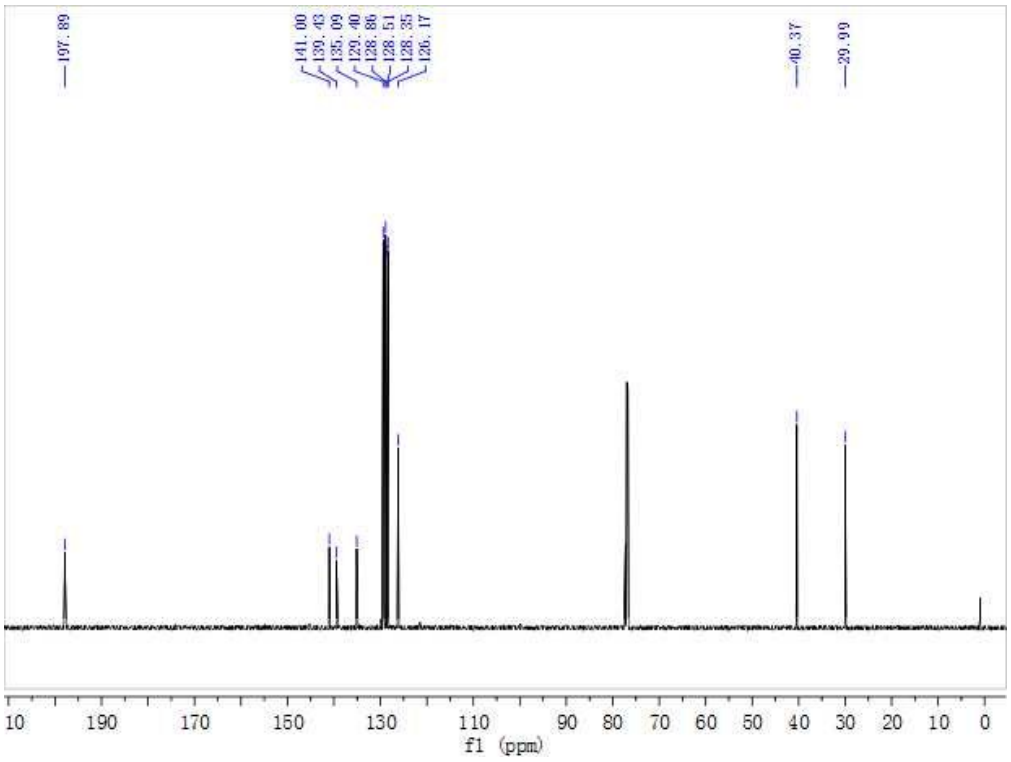

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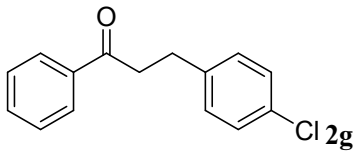

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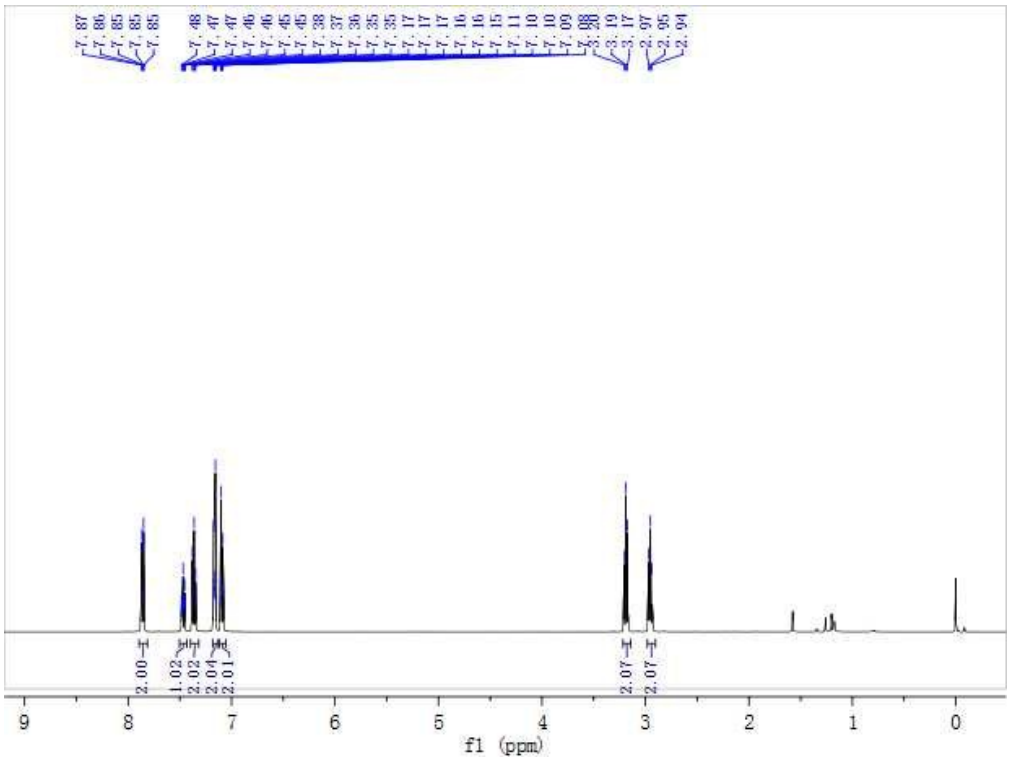

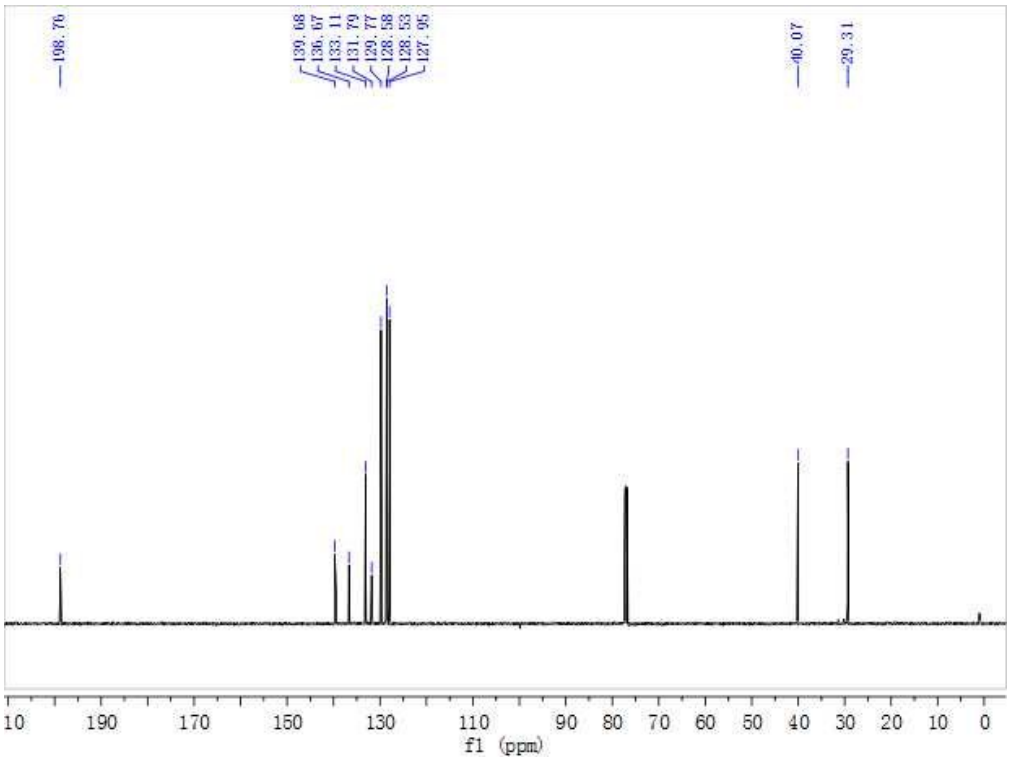

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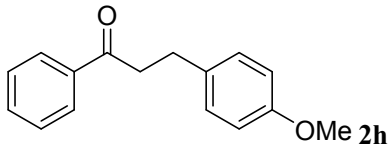

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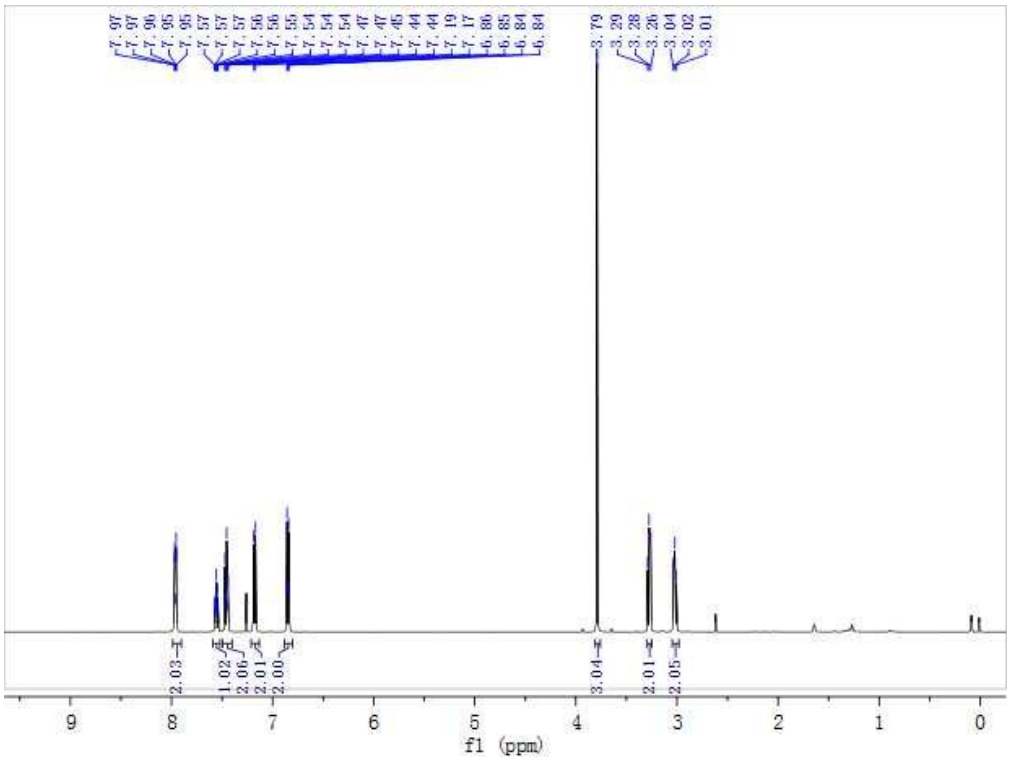

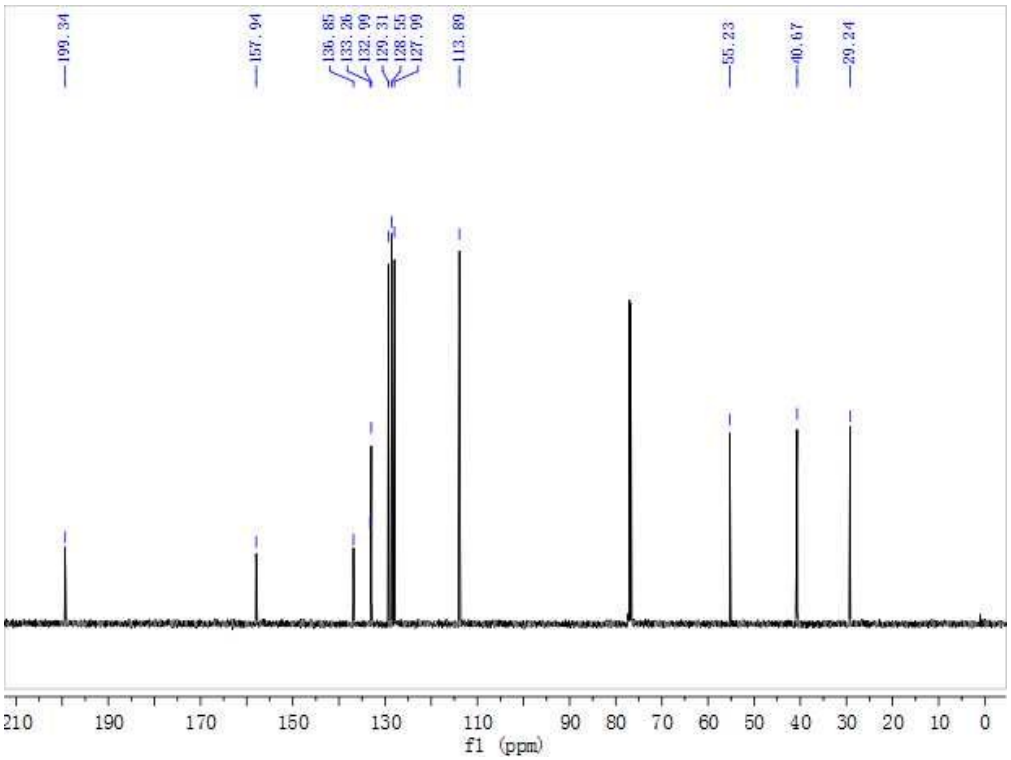

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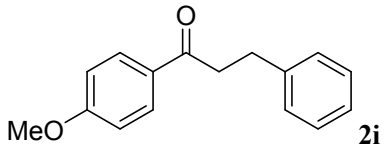

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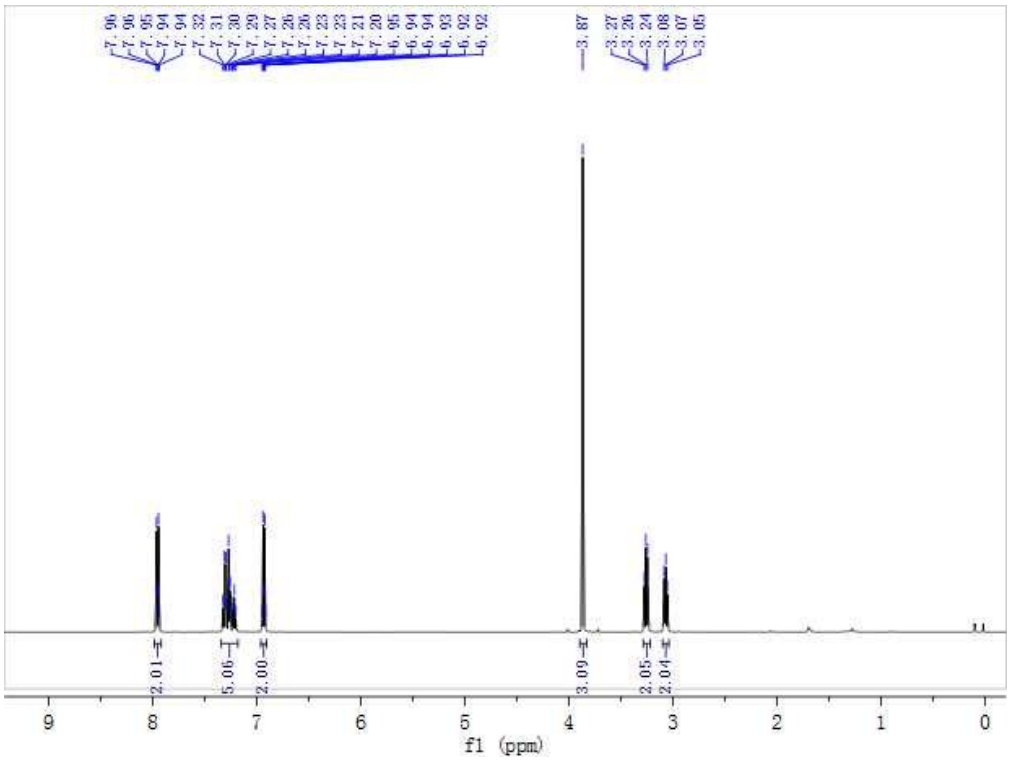

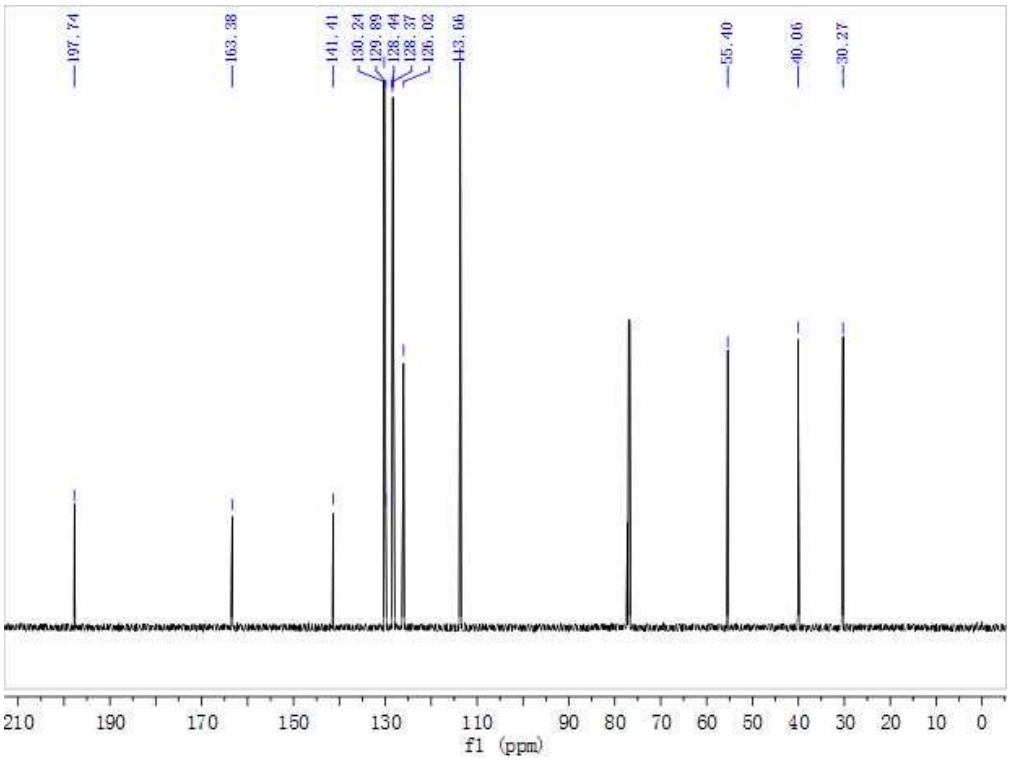

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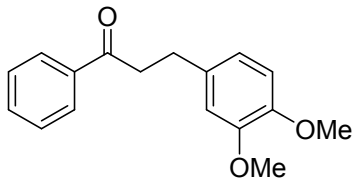

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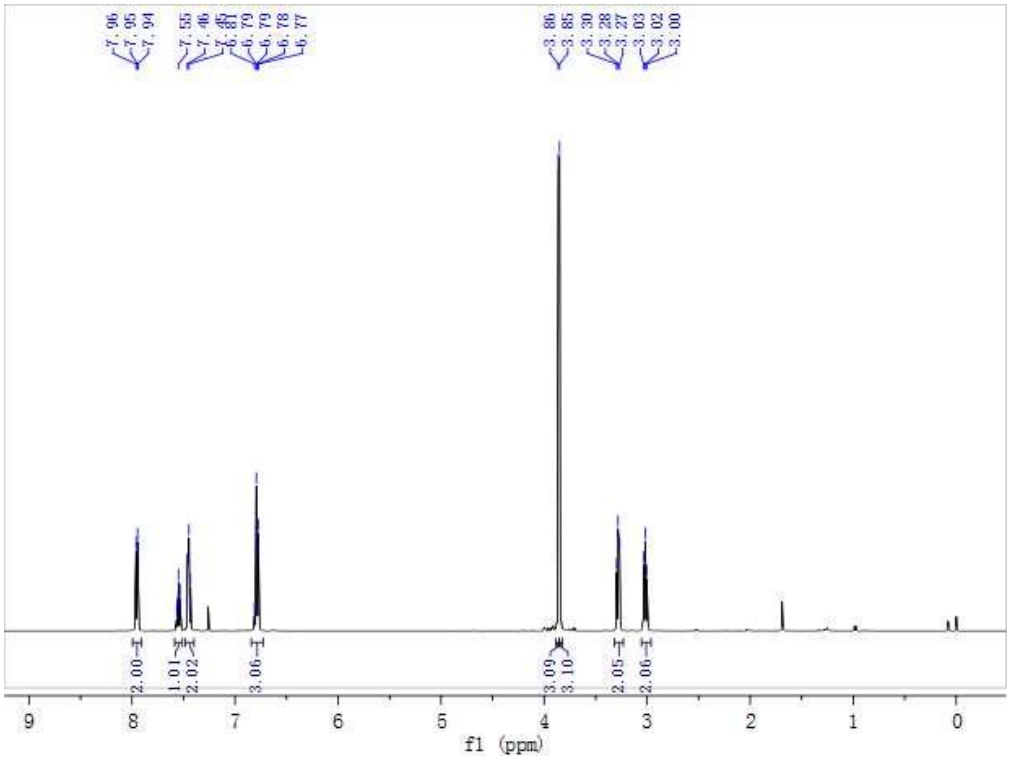

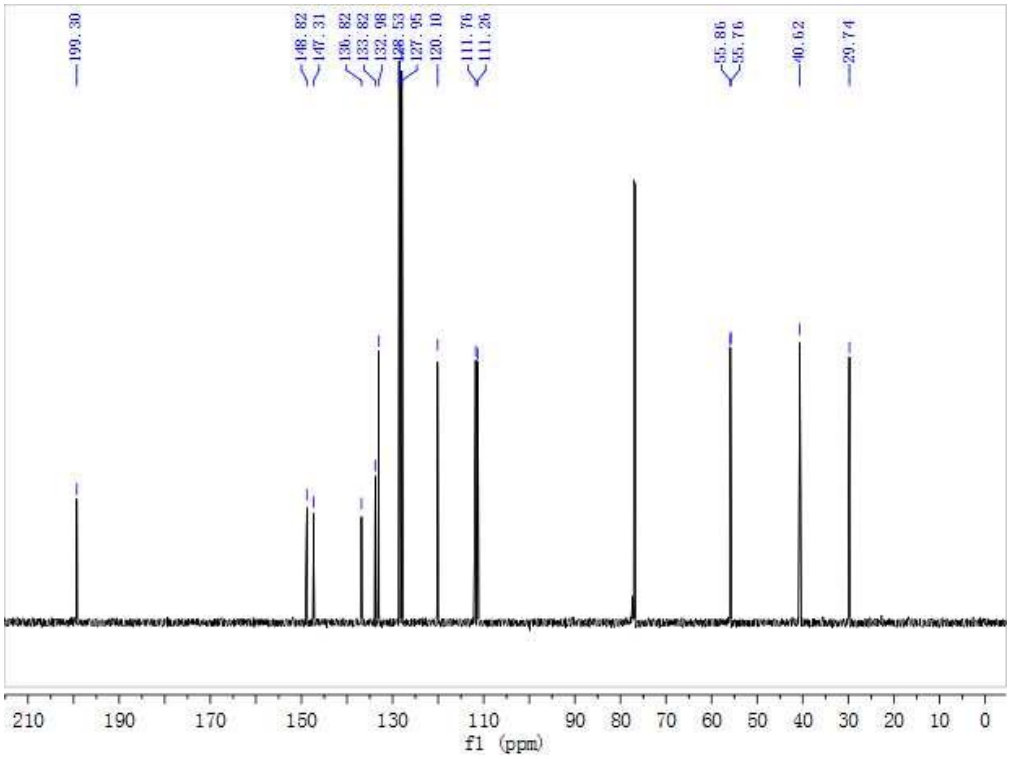

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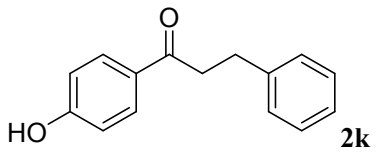

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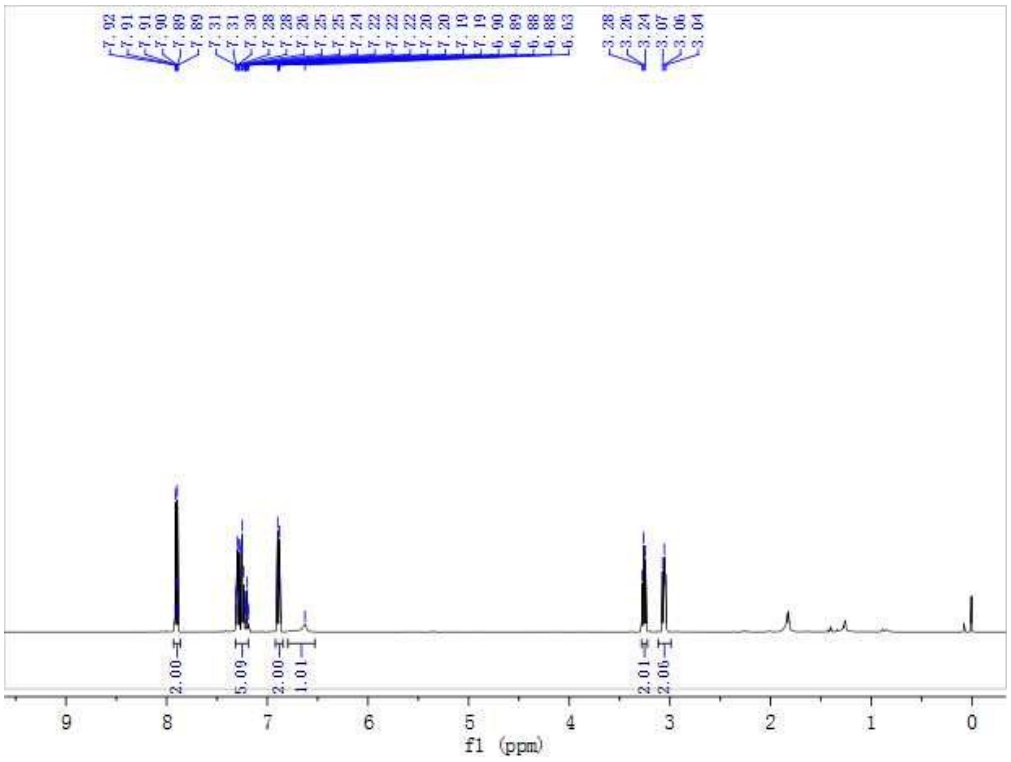

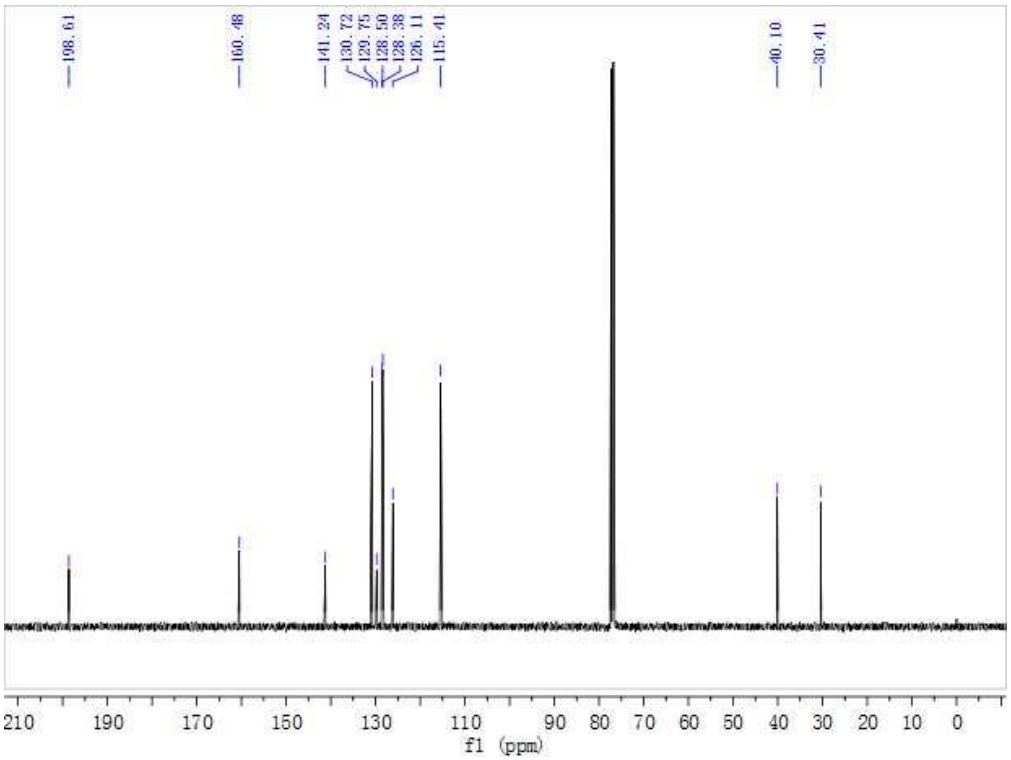

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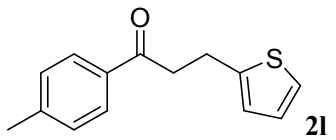

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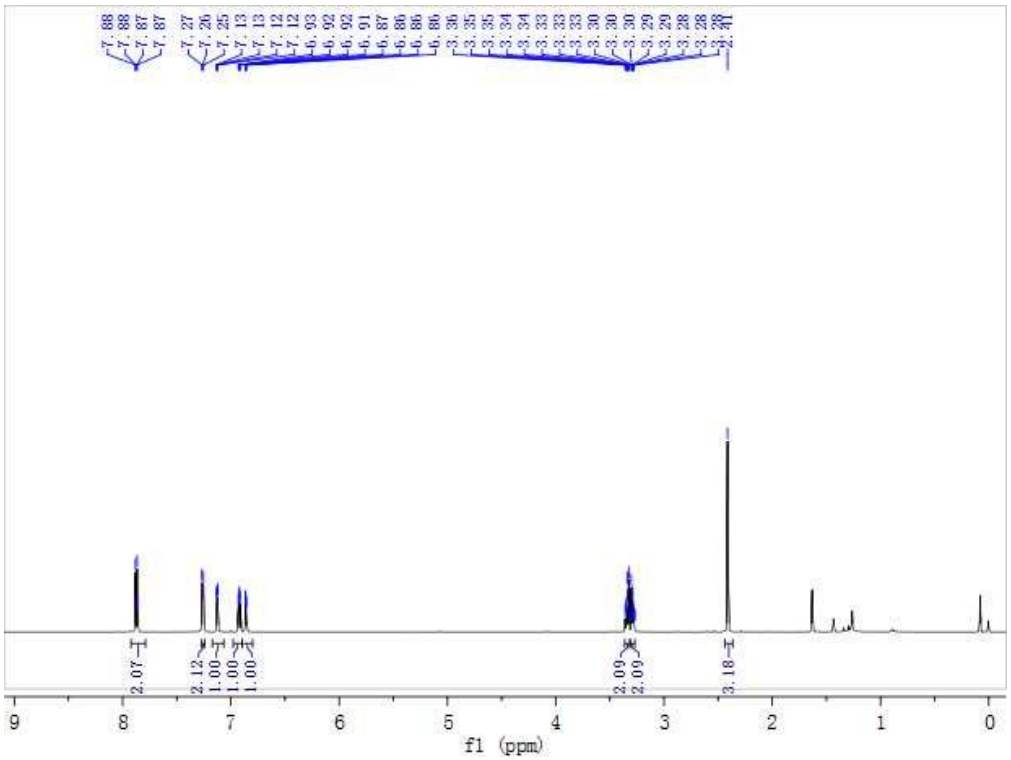

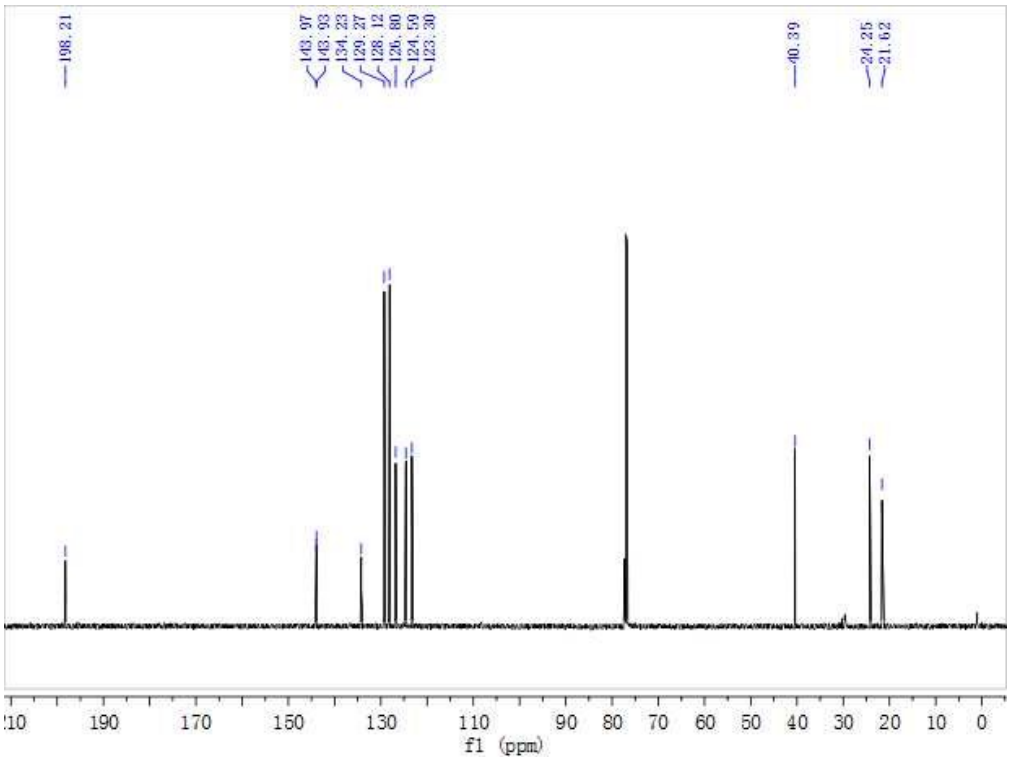

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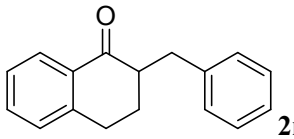

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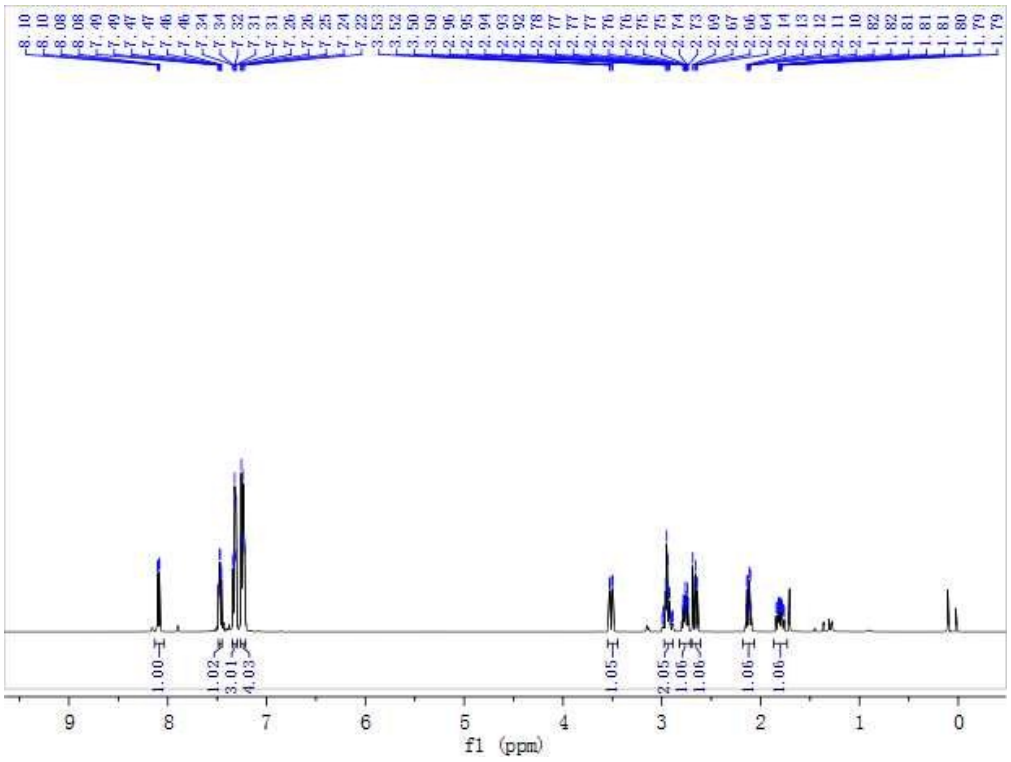
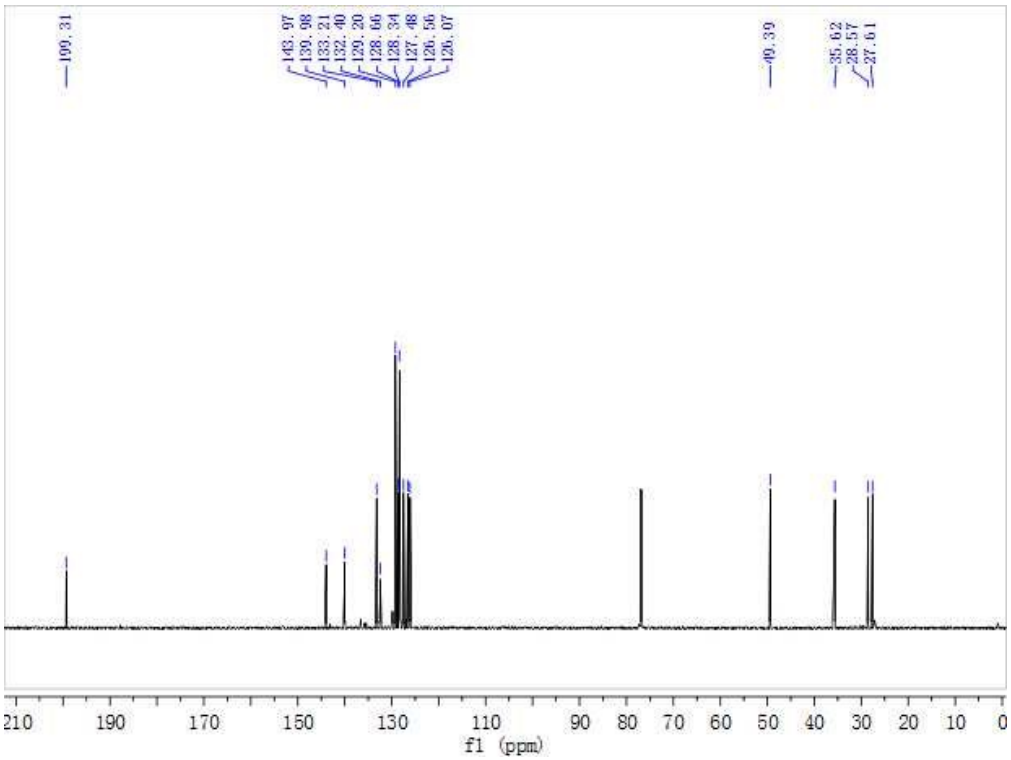
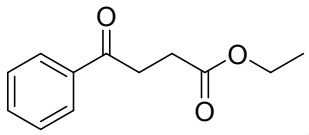




ethyl 4-oxo-4-phenylbutanoate (CAS: 6270-17-3)[9]



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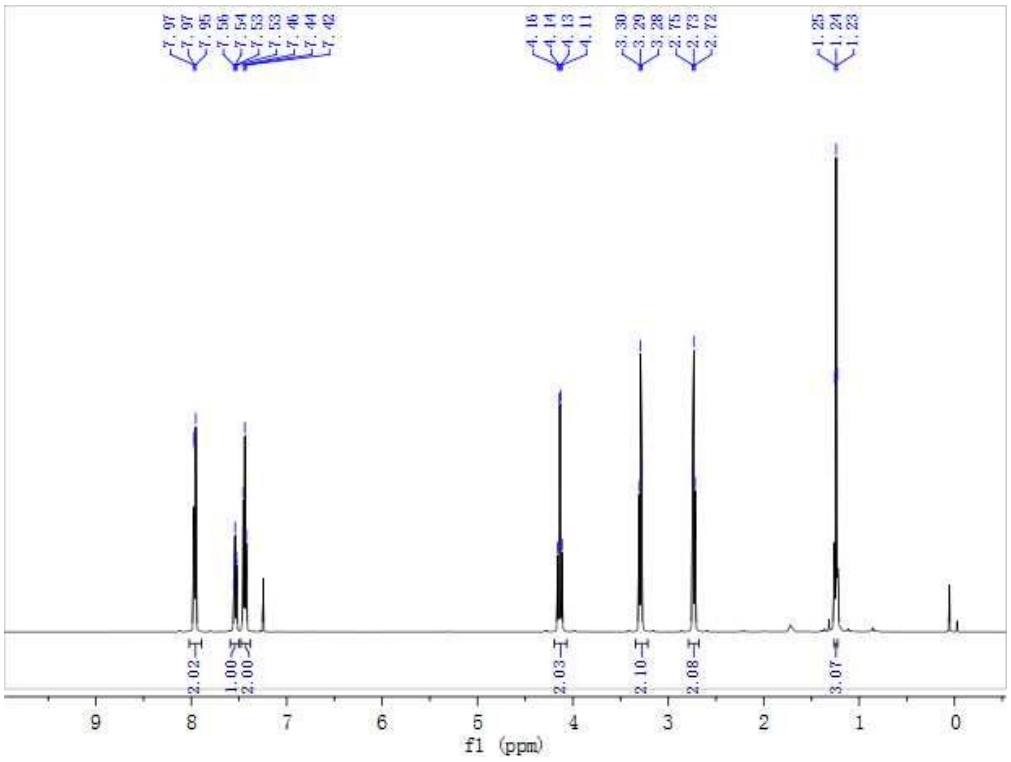
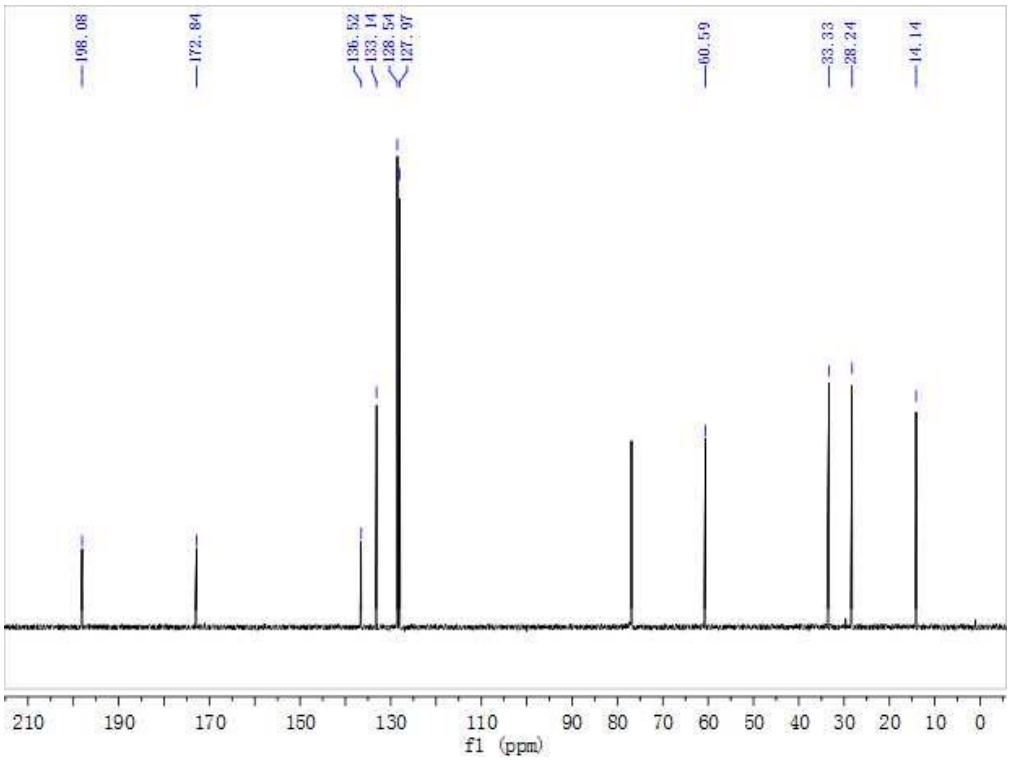
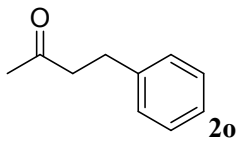




4-phenylbutan-2-one (CAS: 2550-26-7)[2]



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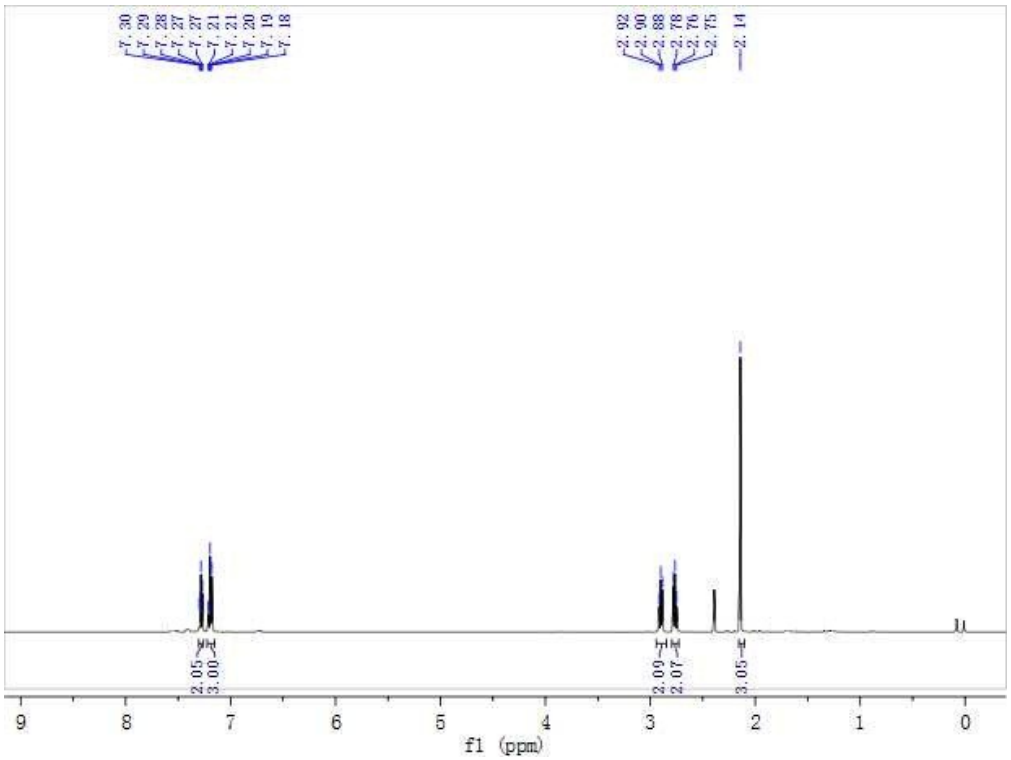

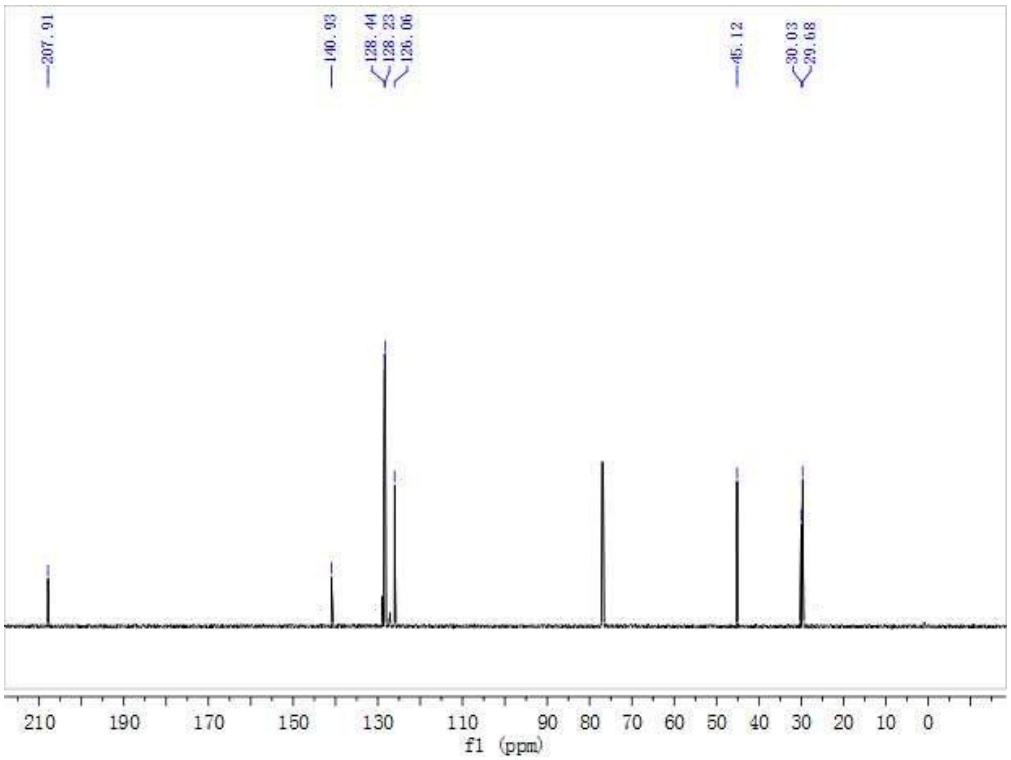

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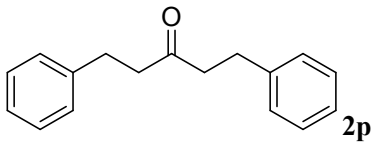

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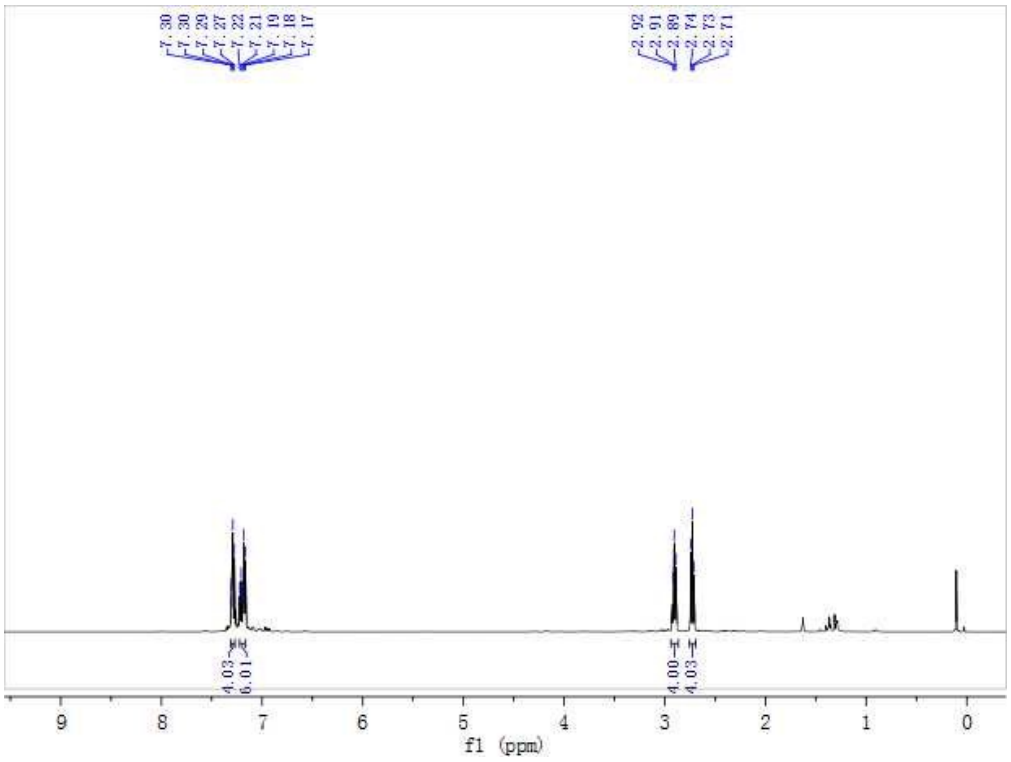
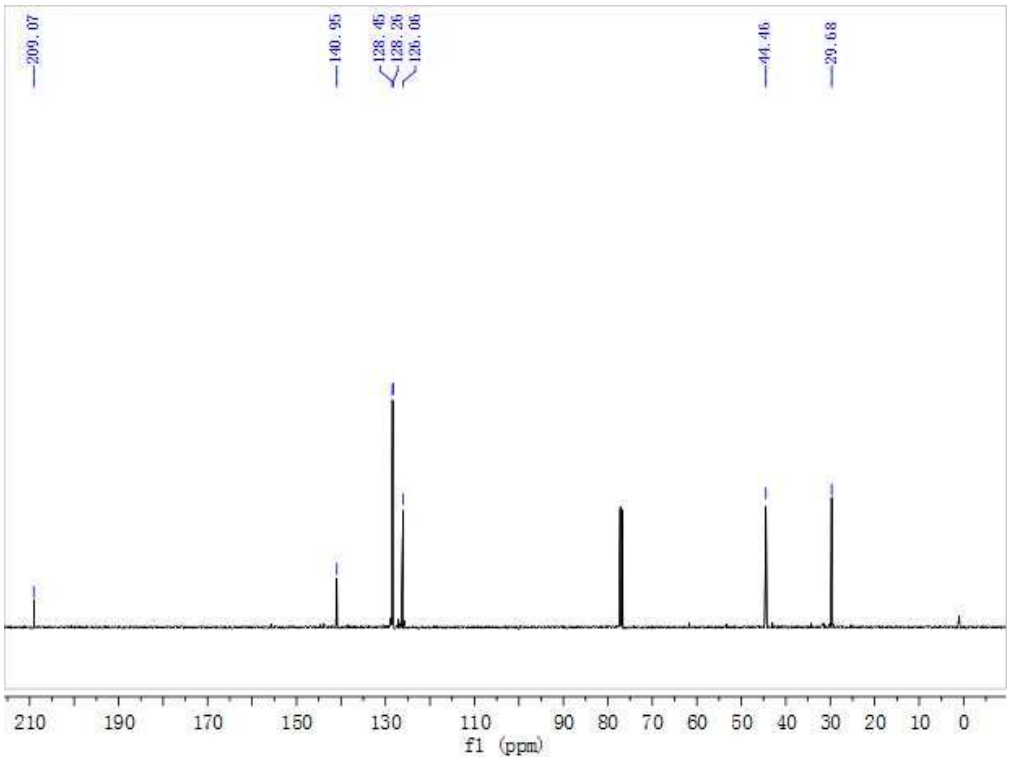
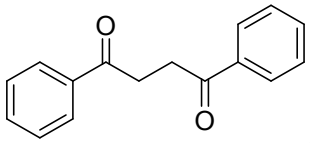




1,4-diphenylbutane-1,4-dione (CAS: 495-71-6)[8]



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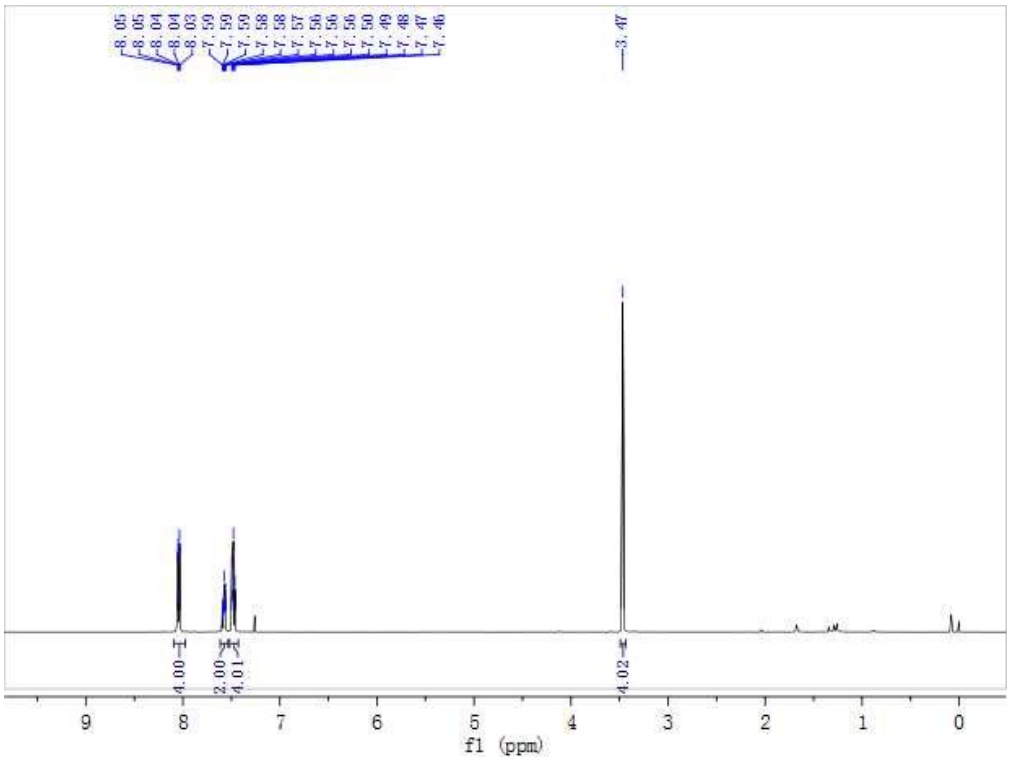

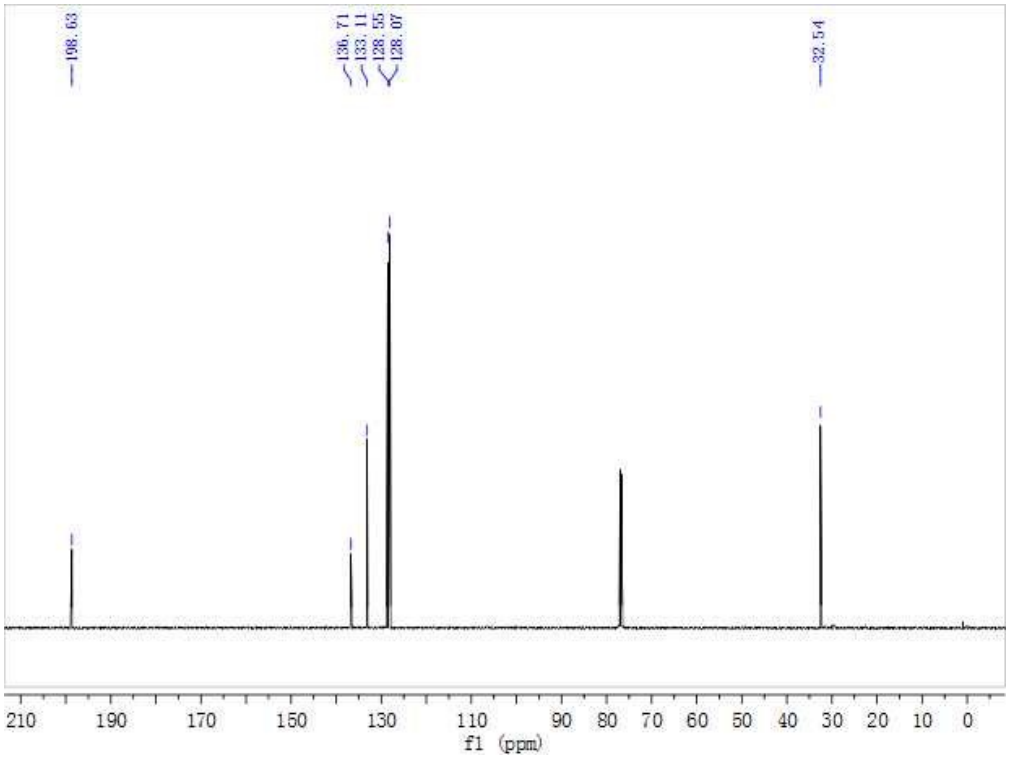

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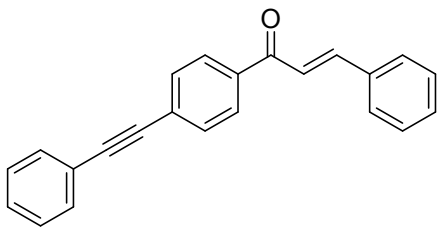

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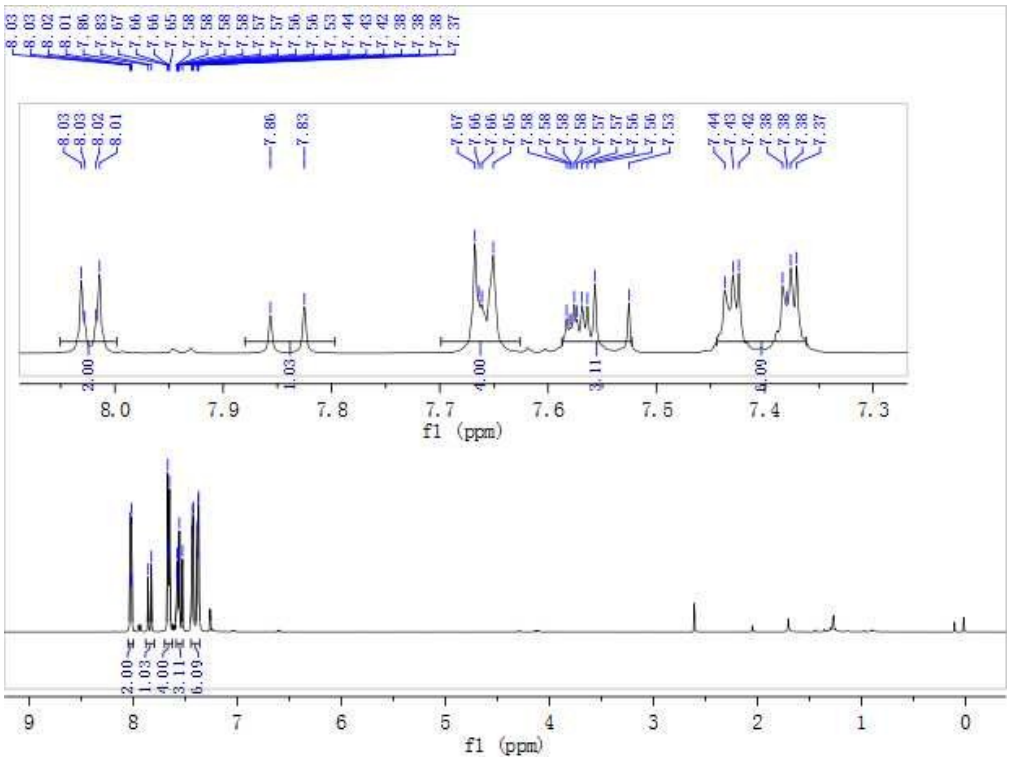
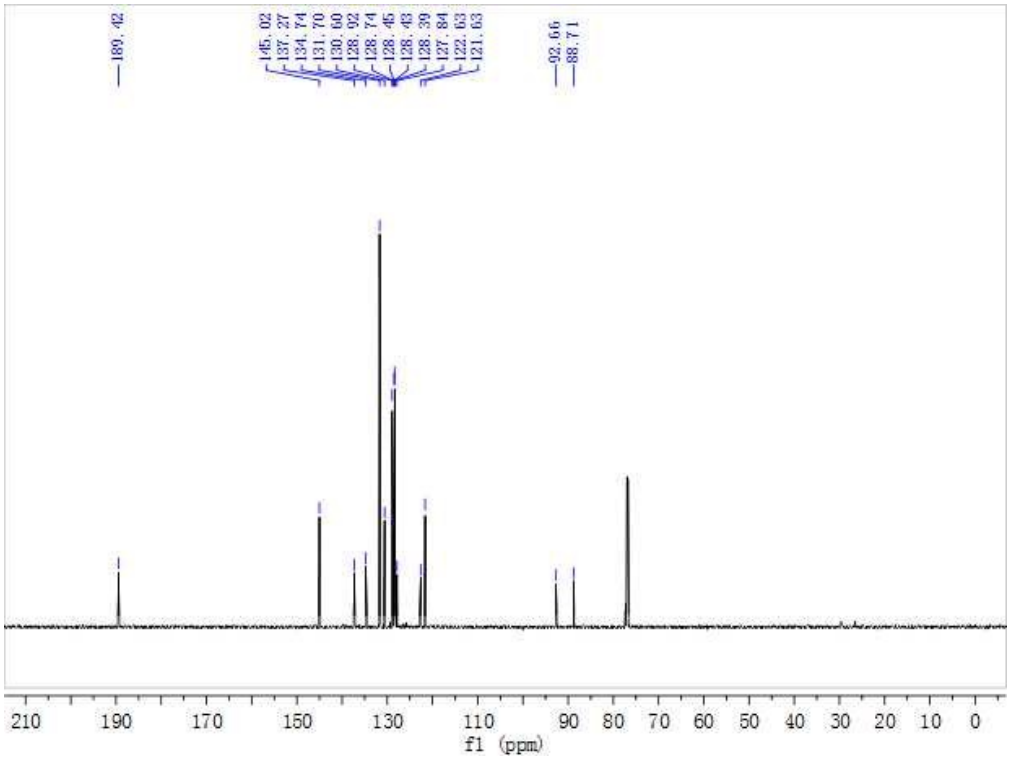
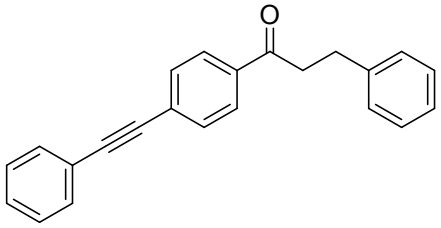
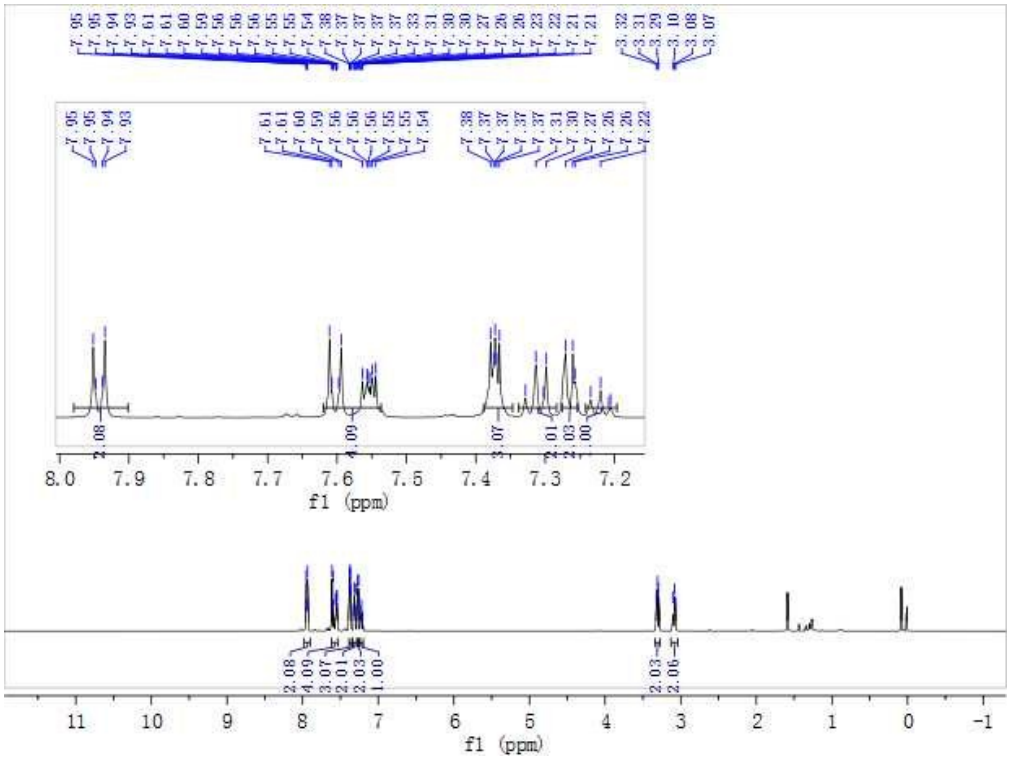


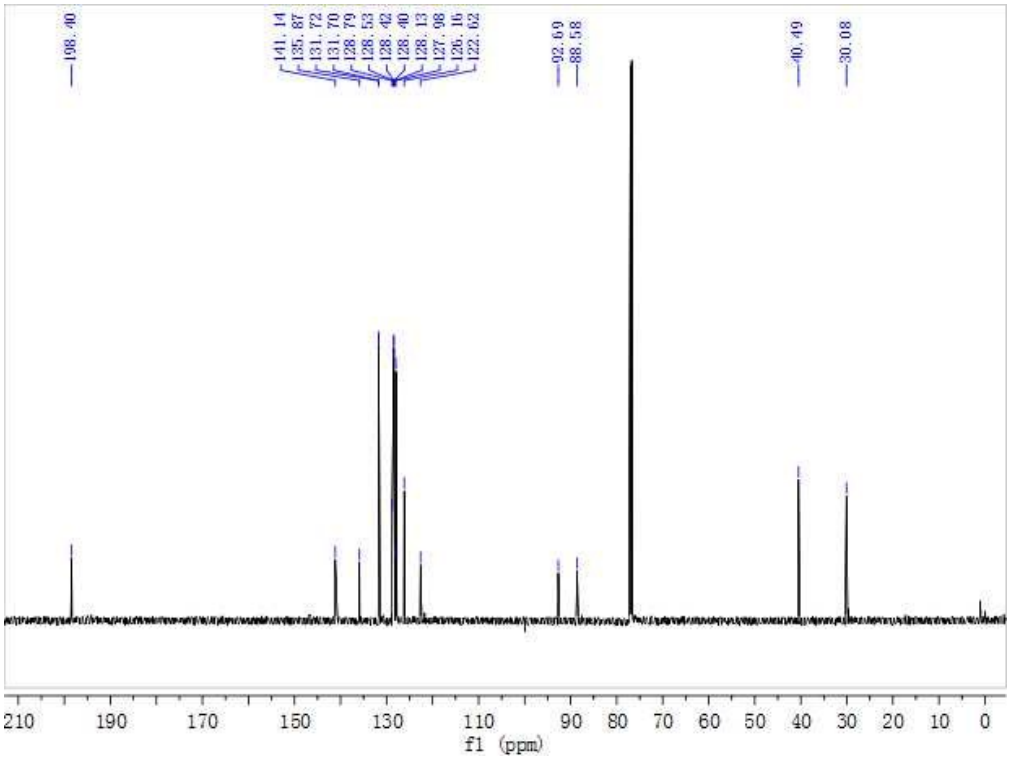


3-phenyl-1-(4-(phenylethynyl)phenyl)propan-1-one (new compound)

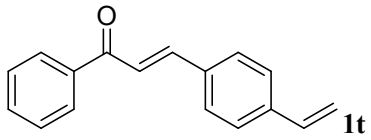


2s

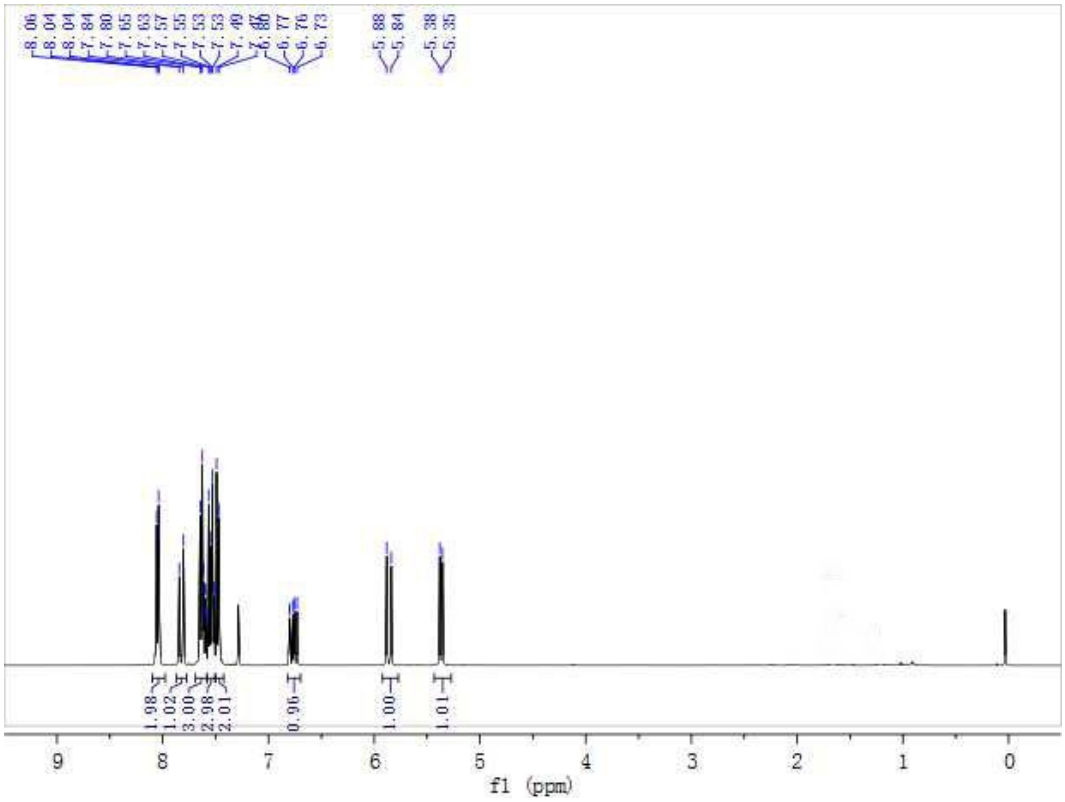


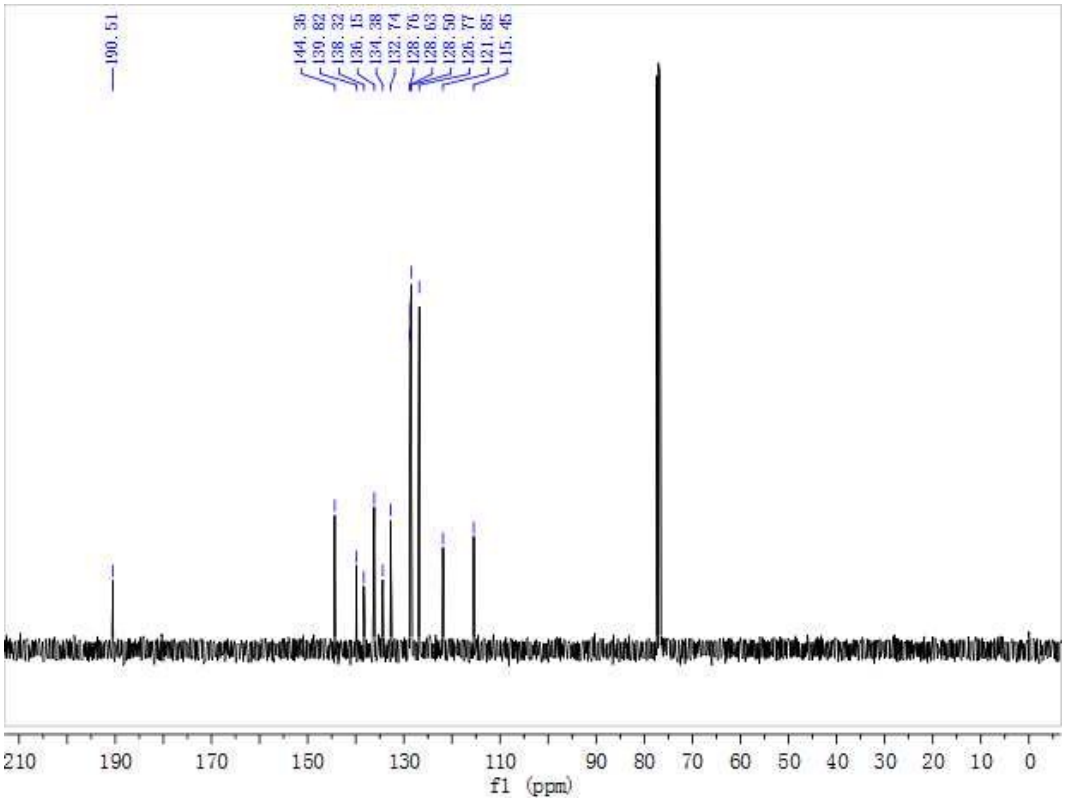


(E)-1-phenyl-3-(4-vinylphenyl)prop-2-en-1-one (CAS: 52506-31-7)

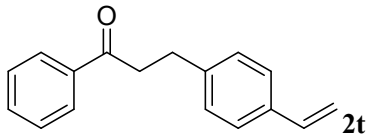


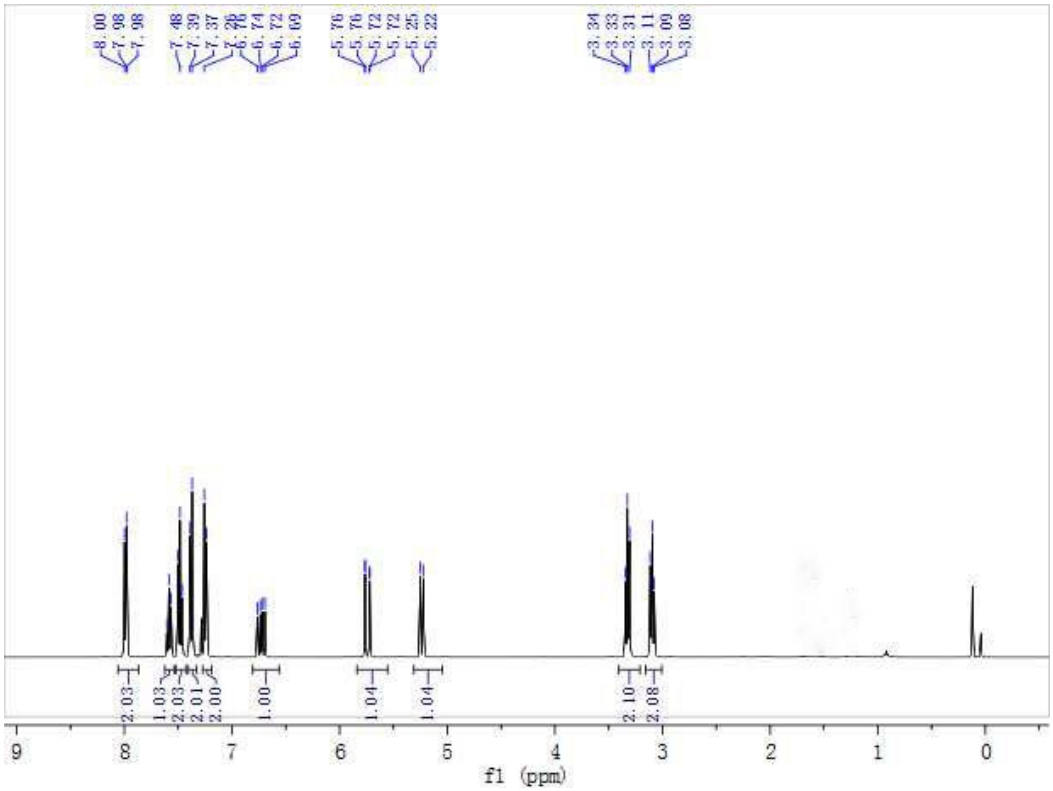
1t

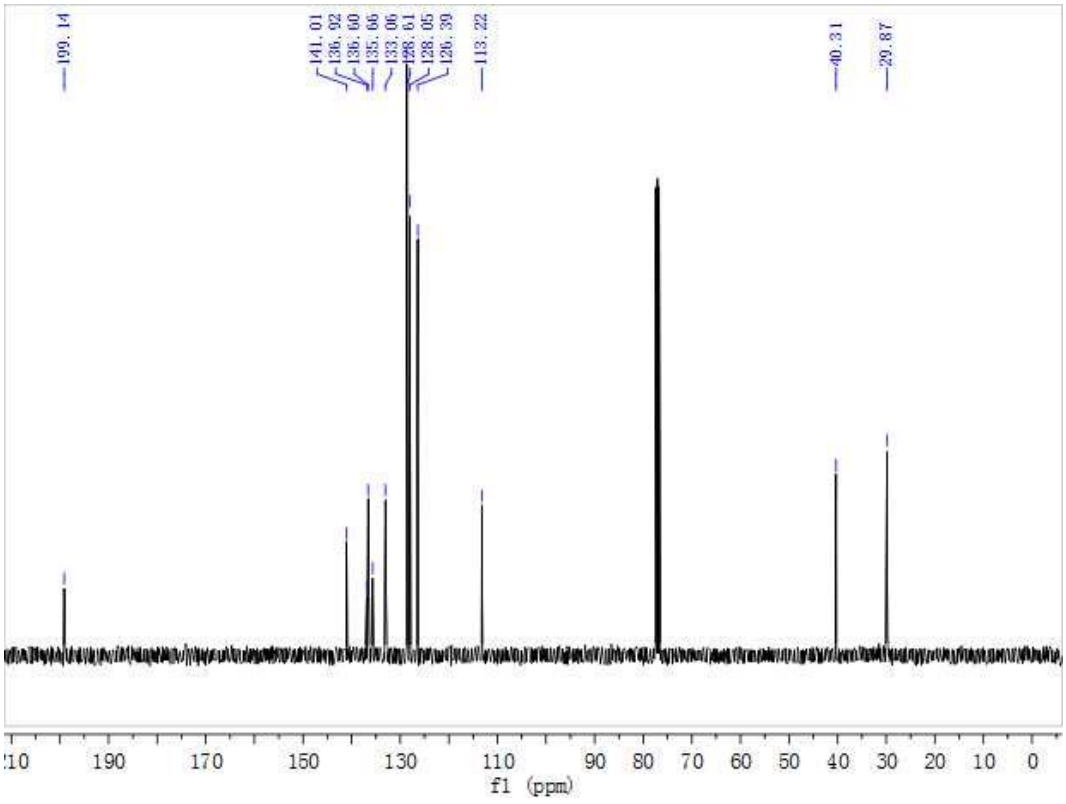




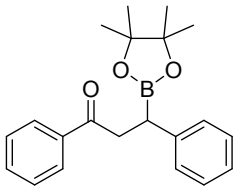
1-phenyl-3-(4-vinylphenyl)propan-1-one (CAS: 93318-87-7)







1,3-diphenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propan-1-one (CAS: 199999-46-7)[12]



3a