Electronic Supplementary Information (ESI)

Palladium-Catalyzed Sonogashira Coupling of Amides: Access to Ynones via C-N Bond Cleavage

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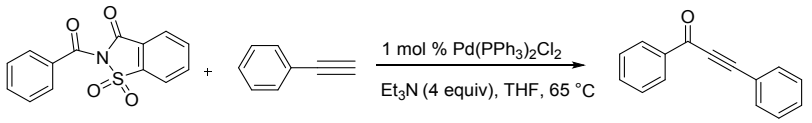
General experimental details and materials

The reactions were conducted in sealed tube under the protection of a nitrogen atmosphere. All reactants reported in the manuscript are commercially available and have been prepared by the method reported previously. N-Methoxy-*N*-Methylbenzamide **4b** was purchased at the China Suppliers. Other amides were prepared by standard methods.[2-5] *N*-Acylsaccharins were prepared by the general methods. All solvents were purchased at the China suppliers and used without any purification. Triethylamine was purified by distillation with calcium hydride. Flash chromatography was performed using 200-300 mesh silica gel. All ynone products are known compounds. ^{1}H and ^{13}C and ^{19}F NMR data were recorded with Bruker Advance III (500 MHz) and Varian (400 MHz) spectrometers in $CDCl_3$ with tetramethylsiliane as an internal standard.

Table 1 Optimation of the reaction conditions[a]



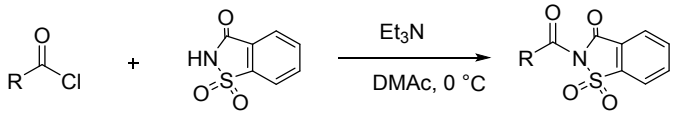
1a 2a 3aa

Entry	Catalyst	Ligand	Base	Solvent	yield[b] (%)
1	$Pd(PPh_3)_2Cl_2$	ND	K_3PO_4	toluene	61
2	$Pd(OAc)_2$	PPh_3	K_3PO_4	toluene	48
3	$Pd(PPh_3)_4$	ND	K_3PO_4	toluene	45
4	$Pd(OAc)_2$	ND	K_3PO_4	toluene	ND
5	$Pd(OAc)_2$	PCy_3	K_3PO_4	toluene	ND
6[c]	$Pd(OAc)_2$	dppf	K_3PO_4	toluene	ND
7	$Pd(PPh_3)_2Cl_2$	ND	K_3PO_4	THF	75
8	$Pd(PPh_3)_2Cl_2$	ND	K_3PO_4	CH_3CN	62
9	$Pd(PPh_3)_2Cl_2$	ND	K_3PO_4	dioxane	57
10	$Pd(PPh_3)_2Cl_2$	ND	ND	THF	ND
11	$Pd(PPh_3)_2Cl_2$	ND	K_2CO_3	THF	trace
12	$Pd(PPh_3)_2Cl_2$	ND	Cs_2CO_3	THF	trace
13	$Pd(PPh_3)_2Cl_2$	ND	Et_3N	THF	95
14	$Pd(PPh_3)_2Cl_2$	ND	*i*-Pr_2NEt	THF	78
15	$Pd(PPh_3)_2Cl_2$	ND	Pyridine	THF	ND
16	$Pd(PPh_3)_2Cl_2$	ND	DBU	THF	ND
17	$Pd(PPh_3)_2Cl_2$	ND	DABCO	THF	ND
18[d]	$Pd(PPh_3)_2Cl_2$	ND	Et_3N	THF	92
19[e]	$Pd(PPh_3)Cl_2$	ND	Et_3N	THF	42

[a] DBU = 1,8-diazabicyclo[5.4.0]undec-7-ene. DABCO = triethylenediamine. dppf = 1,1`-Bis(disphenylphosphino)ferrocene. PCy_3 = tri(cyclohexyl)phosphine. Reaction conditions (unless otherwise noted): 1a (0.5 mmol), 2b (0.75 mmol), catalyst (5 mol %), base (4 equiv, 2 mmol), ligand (10 mol %), solvent (3 mL). The mixture stirred and heated at 65 °C for 24 h. [b]Isolated yield. [c]dppf (5 mol %). [d]catalyst (1 mol %). [e]catalyst (0.5 mol %).

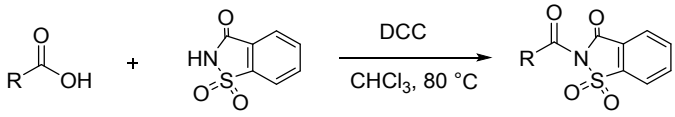
General procedure for N-acylsaccharin synthesis 1a-1p:

General procedure A:



An oven-dried round-bottomed flask (25 mL) equipped with a stir bar was charged with Et_3N (10 mmol, 1.0 equiv), DMAc (8 mL) and saccharin (10 mmol, 1.0 equiv) were slowly added into the mixture. Acyl chloride (1.0 equiv) was added dropwise to the reaction mixture with vigorous stirring at 0 ℃, and the temperature was maintained at 0 ℃ for 1h. After the indicated time, the reaction mixture was poured into 80 mL of water and extracted with ethyl acetate (3 × 20 mL). The organic layer was combined and washed with brine (3 × 30 mL), dried, and concentrated. The obtained residue was purified by column chromatography using petroleum ether/ethyl acetate (3/1) as eluent afforded *N*-benzoylsaccharin (2.10 g, 73.1%).

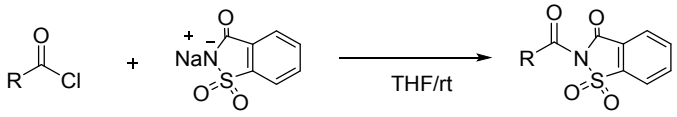
General procedure B:



An oven-dried round-bottomed flask (100 mL) equipped with a stir bar was charged with $CHCl_3$ (50 mL), saccharin (10 mmol, 1.0 equiv), benzoic acid (1.0equiv). DCC (1.0 equiv) was added to the reaction mixture with vigorous stirring at 80^{o}C, and the temperature was maintained at 80 ℃ for 12h. After cooling to room temperature, the mixture was filtrated through a short pad of silica gel, then the silica gel was washed with $CHCl_3$ (3 × 20 mL) and the organic phases were combined. After the solvent was removed, the crude product was purified by silica gel column chromatography using

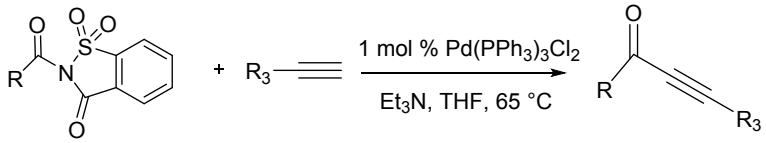
petroleum ether/ethyl acetate (3/1) as eluent afforded *N*-benzoylsaccharin (1.46 g, 51.2%).

General procedure C:



An oven-dried round-bottomed flask (100 mL) equipped with a stir bar was charged with THF (30 mL), saccharin sodium salt (10 mmol, 1.0 equiv), acetic chloride (1.0 equiv). The reaction mixture was vigorous stirring at room temperature for 24h. After the solvent was removed, the crude product was purified by silica gel column chromatography using petroleum ether/ethyl acetate (3/1) as eluent afforded *N*-acetylsaccharin (1.93 g, 85.7%).

General Procedures for the synthesis of products 3aa-3an, 3ba-3pa:



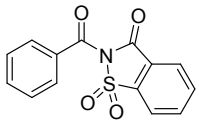
In an oven-dried Teflon septum screw-capped tube (15 mL), add N-acylsacchrin **1a-1p** (0.5 mmol), $Pd(PPh_3)_2Cl_2$ (3.5 mg, 0.005 mmol). The tube was then charged with nitrogen. Super dry tetrahydrofuran (3 ml) was injected into tube by springe. Et_3N (0.3 mL, 2 mmol) and the alkyne reagent **2a-2n** (0.75 mmol) was subsequently injected into the reaction tube. The reaction was monitored by TLC. The reaction was then heated to 65 ℃ and stirred for 24 h. After cooling to room temperature, the solvents were removed, the crude product was purified by silica gel column chromatography using petroleum ether/ethyl acetate as eluent.

Evaluating different amides Scheme 2

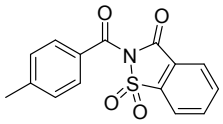
According to the general procedure, different amide **4a-4f** (0.5 mmol) was reacted with $Pd(PPh_3)_2Cl_2$ (3.5 mg, 0.005 mmol), Et_3N (0.3 mL, 2 mmol) and Phenylacetylene **2a** (0.75 mmol) in Super dry tetrahydrofuran (3 ml) for 24 h at 65 ℃. The reaction was monitored by TLC. The reaction was completely unsuccessful. After cooling to room temperature, the solvents were removed, the crude product was purified by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent. The none of ynone products were obtained under optimized condition.

Detail description of for starting materials and products

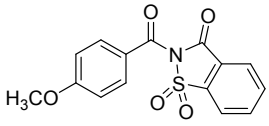
Characterization Data of *N*-Acylsaccharins



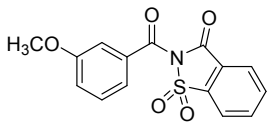
***N*-Benzoylsaccharin (1a).[1]** Following general procedure A, **1a** was isolated as a white solid (2.10 g, 73.1%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.13 – 8.09 (m, 1H), 7.99 (dd, *J* = 4.7, 0.9, 2H), 7.93 – 7.87 (m, 1H), 7.79 – 7.70 (m, 2H), 7.66 – 7.61 (m, 1H), 7.52 – 7.46 (m, 2H).



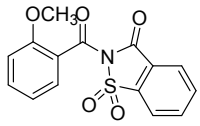
***N*-(4-methylbenzoyl)saccharin (1b).** Following general procedure A, **1b** was isolated as a white solid (2.14 g, 71.2%). ^{1}H NMR (400 MHz, $CDCl_3$)δ = 8.14 – 8.11 (m, 1H), 8.01 – 7.96 (m, 2H), 7.90 (ddd, *J* = 7.8, 5.1, 3.4, 1H), 7.83 – 7.79 (m, 2H), 6.98 – 6.95 (m, 2H), 3.89 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.39, 157.42, 138.38, 136.47, 134.94, 133.89, 132.33, 129.53, 128.42, 126.38, 125.43, 121.24, 21.56. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{15}H_{11}NO_4S$ 301.0409; Found 301.0417.



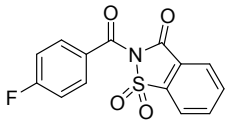
***N*-(4-methoxybenzoyl)saccharin (1c).** Following general procedure A, **1c** was isolated as a white solid (2.64 g, 83.3%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.15 – 8.11 (m, 1H), 8.00 – 7.95 (m, 2H), 7.90 (ddd, *J* = 7.8, 5.1, 3.4, 1H), 7.84 – 7.79 (m, 2H), 6.99 – 6.95 (m, 2H), 3.89 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 164.67, 157.92, 138.47, 136.24, 134.82, 132.74, 126.27, 125.79, 124.37, 121.19, 113.92, 109.99, 55.64. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{15}H_{11}NO_5S$ 317.0358; Found 317.0361.



***N*-(3-methoxybenzoyl)saccharin (1d).** Following general procedure A, **1d** was isolated as a white solid (2.58 g, 81.4%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.13 (d, *J* = 7.6, 1H), 8.05 – 7.99 (m, 2H), 7.94 – 7.90 (m, 1H), 7.40 (t, *J* = 7.9, 1H), 7.36 – 7.28 (m, 2H), 7.19 (dd, *J* = 8.1, 2.6, 1H), 3.86 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$)δ = 166.18, 159.49, 157.46, 138.41, 136.41, 134.90, 133.53, 129.47, 126.39, 121.93, 121.25, 121.09, 120.25, 114.22, 55.54. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{15}H_{11}NO_5S$ 317.0358; Found 317.0363.

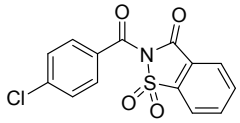


***N*-(2-methoxybenzoyl)saccharin (1e).** Following general procedure B, **1e** was isolated as a white solid (1.57 g, 49.6%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.07 (d, *J* = 7.9, 1H), 8.03 – 7.94 (m, 2H), 7.91 – 7.87 (m, 1H), 7.60 – 7.47 (m, 2H), 7.08 (t, *J* = 7.5, 1H), 6.93 (d, *J* = 8.8, 1H), 3.71 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 164.83, 157.19, 156.63, 138.45, 136.28, 134.78, 133.88, 130.04, 126.03, 125.51, 122.96, 121.27, 120.95, 111.09, 55.85. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{15}H_{11}NO_5S$ 317.0358; Found 317.0360.

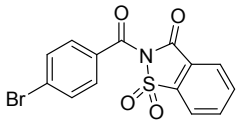


***N*-(4-fluorobenzoyl)saccharin (1f).** Following general procedure A, **1f** was

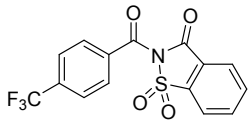
isolated as a white solid (2.25 g, 73.8%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.14 (d, J = 7.6, 1H), 8.03 – 7.98 (m, 2H), 7.95 – 7.91 (m, 1H), 7.81 (ddd, J = 8.0, 5.1, 2.5, 2H), 7.22 – 7.14 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.16, 157.52, 138.35(d, J^1 = 258.2), 136.52, 135.02, 132.54 (d, J^3 = 9.2, 2H), 128.51 (d, J^4 = 2.1), 126.42, 125.40, 121.28, 115.95, 115.73(d, J^2 = 23.2). ^{19}F NMR (376 MHz, $CDCl_3$) δ = -102.62. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{14}H_8FNO_4S$ 305.0158; Found 305.0160.



***N*-(4-chlorobenzoyl)saccharin (1g).** Following general procedure A, **1g** was isolated as a white solid (2.19 g, 68.3%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.13 (d, J = 7.6, 1H), 8.00 (t, J = 3.6, 2H), 7.95 – 7.90 (m, 1H), 7.73 – 7.68 (m, 2H), 7.50 – 7.45 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.39, 157.41, 140.51, 138.38, 136.54, 134.98, 130.98, 130.66, 128.85, 126.45, 125.34, 121.29. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{14}H_8ClNO_4S$ 321.0201; Found 321.0200.

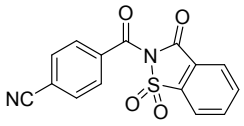


***N*-(4-bromobenzoyl)saccharin (1h).** Following general procedure A, **1h** was isolated as a white solid (2.65 g, 72.5%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (dd, J = 7.6, 0.7 Hz, 1H), 7.95 (d, J = 3.8 Hz, 2H), 7.87 (dd, J = 8.1, 4.1 Hz, 1H), 7.60 – 7.54 (m, 4H). ^{13}C NMR (101 MHz, CDCl3) δ 165.61, 157.50, 136.53, 134.97, 131.82, 130.98, 126.46, 121.29, 77.31, 76.99, 76.68. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{14}H_8ClNO_4S$ 364.9357; Found 364.9362.

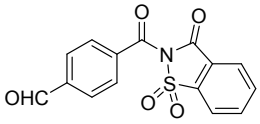


***N*-(4-(trifluoromethyl)benzoyl)saccharin (1i).** Following general procedure A, **1i** was isolated as a white solid (1.62 g, 45.6%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.13 (d, J = 7.6, 1H), 8.03 (d, J = 3.9, 2H), 7.95 (dd, J = 7.6, 4.3,1H), 7.84 (d, J = 8.2, 2H),

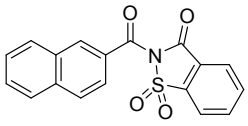
7.76 (d, J = 8.2, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.46, 157.19, 138.34(q, J^2 = 37.1), 136.73, 135.63, 135.10(q, J^3 = 3.8), 130.55, 129.55, 126.53, 125.46, 125.42, 125.10, 121.36(q, J^1 = 274.2). ^{19}F NMR (376 MHz, $CDCl_3$) δ = -63.23. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{15}H_8F_3NO_4S$ 355.0126; Found 355.0121.



***N*-(4-cyanobenzoyl)saccharin (1j).** Following general procedure A, **1j** was isolated as a white solid (1.68 g, 53.8%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.13 (d, J = 7.6, 1H), 8.05 – 8.01 (m, 2H), 7.97 – 7.93 (m, 1H), 7.79 (d, J = 7.6, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.06, 157.11, 138.34, 136.84, 136.19, 135.16, 132.13, 129.54, 126.58, 124.94, 121.40, 117.57, 116.77. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{15}H_8N_2O_4S$ 312.0205; Found 313.0212.

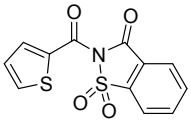


N-(4-formylbenzoyl)saccharin **(1k).** Following general procedure A, **1k** was isolated as a white solid (1.94 g, 61.5%). ^{1}H NMR (400 MHz, $CDCl_3$)δ = 10.12 (s, 1H), 8.12 (d, J = 7.6, 1H), 8.04 – 7.99 (m, 4H), 7.94 (dt, J = 8.1, 4.2, 1H), 7.86 (d, J = 8.3, 2H). ^{13}C NMR (100 MHz, $CDCl_3$)δ = 191.18, 165.66, 157.16, 139.18, 138.39, 137.31, 136.72, 135.08, 129.64, 129.41, 126.54, 125.10, 121.36. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{14}H_8ClNO_4S$ 295.0409; Found 295.0309.

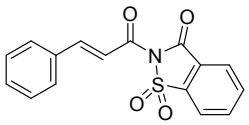


***N*-(2-naphthoyl)saccharin (1l).** Following general procedure A, **1l** was isolated as a white solid (2.24 g, 66.5%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.36 (s, 1H), 8.14 (d, J = 7.7, 1H), 8.01 (t, J = 7.5, 2H), 7.98 – 7.94 (m, 1H), 7.92 (t, J = 8.2, 3H), 7.77 (dd, J = 8.6, 1.8, 1H), 7.66 – 7.62 (m, 1H), 7.59 – 7.55 (m, 1H). ^{13}C NMR (101 MHz, cdcl$_3$) δ 166.38, 157.59, 138.50, 136.39, 135.83, 134.89, 132.10, 131.88, 129.59, 129.57,

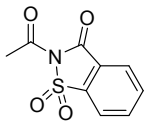
129.12, 128.30, 127.91, 127.13, 126.42, 125.60, 124.69, 121.26. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{18}H_{11}NO_4S$ 337.0740; Found 337.0743.



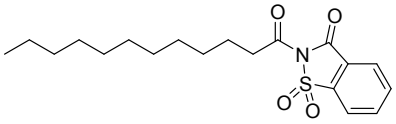
***N*-(thiophene-2-carbonyl)saccharin (1m).** Following general procedure A, **1m** was isolated as a white solid (2.03 g, 69.3%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.18 (dd, *J* = 9.4, 3.7, 1H), 8.01 – 7.90 (m, 4H), 7.84 (d, *J* = 5.0, 1H), 7.20 – 7.16 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 158.87, 158.03, 138.47, 137.20, 136.61, 135.79, 135.18, 128.43, 126.56, 125.88, 125.86, 121.48. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{12}H_7NO_4S_2$ 292.9817; Found 292.9808.



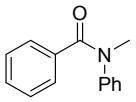
***N*-cinnamoylsaccharin (1n).** Following general procedure A, **1n** was isolated as a white solid (1.98 g, 63.3%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.19 (d, *J* = 7.6, 1H), 8.08 (d, *J* = 15.7, 1H), 8.01 – 7.96 (m, 2H), 7.95 – 7.90 (m, 1H), 7.72 (d, *J* = 15.6, 1H), 7.67 (dd, *J* = 7.5, 2.0, 2H), 7.47 – 7.41 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 163.26, 157.82, 149.68, 138.31, 136.42, 134.86, 134.01, 131.53, 129.05, 129.00, 126.27, 125.20, 121.20, 117.03. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{16}H_{11}NO_4S$ 313.0402; Found 313.0399.



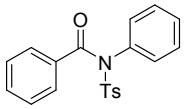
***N*-acetylsaccharin (1o).** Following general procedure C, **1o** was isolated as a white solid (1.93 g, 85.7%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.16 (d, *J* = 7.6, 1H), 8.01 – 7.95 (m, 2H), 7.92 (ddd, *J* = 7.8, 5.9, 2.7, 1H), 2.70 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.10, 157.61, 138.17, 136.50, 134.90, 126.28, 125.03, 121.24, 26.04. HRMS (EI) m/z: $[M]^+$ Calcd for $C_9H_7NO_4S$ 225.0096; Found 225.0099.



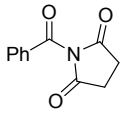
***N*-dodecanoylsaccharin (1p).** Following general procedure C, **1p** was isolated as a white solid (3.01 g, 82.3%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.14 (d, *J* = 7.6, 1H), 8.00 – 7.94 (m, 2H), 7.90 (ddd, *J*=7.7, 5.9, 2.7, 1H), 3.02 (t, *J*=7.4, 2H), 1.74 (dd, *J* = 15.0, 7.5, 2H), 1.42 – 1.24 (m, 16H), 0.87 (t, *J* = 6.8, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 171.40, 157.59, 138.19, 136.41, 134.87, 126.21, 125.07, 121.19, 38.11, 33.84, 31.88, 29.58, 29.40, 29.30, 29.28, 28.88, 24.68, 23.49, 22.66, 14.10. HRMS (EI) m/z: $[M]^+$ Calcd for $C_{19}H_{27}NO_4S$ 365.1999; Found 365.1996.



***N*-Methyl-*N*-phenylbenzamide (4a).** Following general procedure of literature, **4a** was was purified by column chromatography (Petroleum ether/EtOAc = 3/1) to afford a white solid . ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (dd, *J* = 5.2, 3.3 Hz, 2H), 7.05 – 6.99 (m, 3H), 6.99 – 6.91 (m, 3H), 6.89 – 6.84 (m, 2H), 3.32 (s, 3H). Spectral data match those previously reported[2].

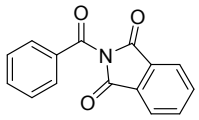


***N*-phenyl-*N*-tosyl benzamide (4c).** Following general procedure of literature, **4c** was was purified by column chromatography (Petroleum ether/EtOAc = 2/1) to afford a white solid (3.05 g, 87%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.89 – 7.78 (m, 2H), 7.47 – 7.39 (m, 2H), 7.34 – 7.24 (m, 6H), 7.19 – 7.12 (m, 4H), 2.44 (s, 3H). Spectral data match those previously reported[3].

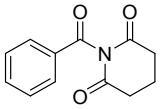


1-Benzoylpyrrolidine-2,5-dione (4d). Following general procedure of literature, **4d** was was purified by column chromatography (Petroleum ether/EtOAc = 3/1) to

afford a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 – 7.71 (m, 2H), 7.57 (t, *J* = 7.5 Hz, 1H), 7.40 (t, *J* = 7.8 Hz, 2H), 2.82 (s, 4H). Spectral data match those previously reported[4].

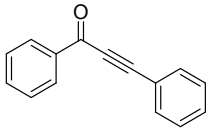


2-Benzoylisoindoline-1,3-dione (4e). Following general procedure of the literature, **4e** was purified by column chromatography (Petroleum ether/EtOAc = 3/1) to afford a white solid (2.10 g, 83.2%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.98 (dd, *J* = 4.6, 2.0, 1H), 7.89 – 7.84 (m, 4H), 7.76 (dd, *J* = 5.4, 3.2, 1H), 7.70 – 7.62 (m, 1H), 7.53 – 7.44 (m, 2H). Spectral data match those previously reported[5].



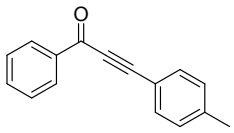
1-Benzoylpiperidine-2,6-dione (4f). Following general procedure of the literature, **4f** was was purified by column chromatography (Petroleum ether/EtOAc = 3/1) to afford a white solid (2.18 g, 75.1%). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.90 – 7.81 (m, 2H), 7.66 – 7.61 (m, 1H), 7.51 – 7.45 (m, 2H), 2.80 – 2.74 (m, 4H), 2.15 (ddd, *J* = 13.1, 6.5, 2.7, 2H). Spectral data match those previously reported[4].

Detail descriptions for products

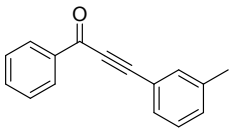


1,3-Diphenylprop-2-yn-1-one (3aa): In an oven-dried Teflon septum screw-capped tube (15 mL), N-benzoylsaccharin **1a** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene **2a** (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product 3aa (94.9 mg, 92%) as a yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$)

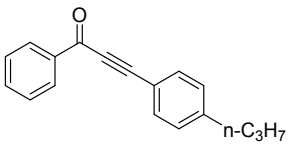
δ 8.18 – 8.14 (m, 2H), 7.63 – 7.60 (m, 2H), 7.58 – 7.54 (m, 1H), 7.47 – 7.43 (m, 2H), 7.43 – 7.39 (m, 1H), 7.35 (ddd, *J* = 6.7, 4.5, 1.2 Hz, 2H). Spectral data match those previously reported[6].



1-Phenyl-3-(p-tolyl)prop-2-yn-1-one (3ab): In an oven-dried Teflon septum screw-capped tube (15 mL), N-benzoylsaccharin **1a** (0.5 mmol, 1.0 equiv) was reacted with 4-ethynyltoluene **2b** (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3ab** (89.2 mg, 81%) as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 – 8.13 (m, 2H), 7.58 – 7.50 (m, 3H), 7.45 (dd, *J* = 10.6, 4.7 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 2.34 (s, 3H). Spectral data match those previously reported[6].

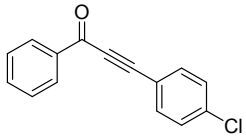


1-Phenyl-3-(m-tolyl)prop-2-yn-1-one (3ac): In an oven-dried Teflon septum screw-capped tube (15 mL), N-benzoylsaccharin **1a** (0.5 mmol, 1.0 equiv) was reacted with 3-ethynyltoluene **3c** (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3ac** (67.2 mg, 61%) as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 – 8.20 (m, 2H), 7.64 – 7.60 (m, 1H), 7.54 – 7.48 (m, 4H), 7.32 – 7.28 (m, 2H), 2.38 (s, 3H). Spectral data match those previously reported[6].

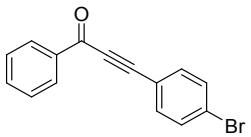


1-phenyl-3-(4-propylphenyl)prop-2-yn-1-one (3ad): In an oven-dried Teflon

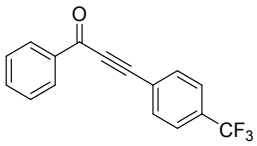
septum screw-capped tube (15 mL), N-benzoylsaccharin **1a** (0.5 mmol, 1.0 equiv) was reacted with 1-ethynyl-4-propylbenzene **2d** (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (20/1) as eluent afforded the product **3ad** (109.3 mg, 88%) as a pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$)δ 8.22 (dt, *J* = 8.5, 1.6 Hz, 2H), 7.63 – 7.58 (m, 3H), 7.53 – 7.49 (m, 2H), 7.23 (d, *J* = 8.2 Hz, 2H), 2.65 – 2.60 (m, 2H), 1.68 – 1.63 (m, 2H), 0.95 (dd, *J* = 9.8, 4.9 Hz, 3H). Spectral data match those previously reported[7].



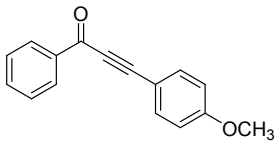
1-Phenyl-3-(p-chlorophenyl)prop-2-yn-1-one (3ae): In an oven-dried Teflon septum screw-capped tube (15mL), N-benzoylsaccharin **1a** (0.5 mmol, 1.0 equiv) was reacted with 1-chloro-4-ethynylbenzene **2e** (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3ae** (103.5 mg, 86%) as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 – 8.18 (m, 2H), 7.65 – 7.60 (m, 3H), 7.52 (t, *J* = 7.7 Hz, 2H), 7.42 – 7.40 (m, 2H). Spectral data match those previously reported[6].



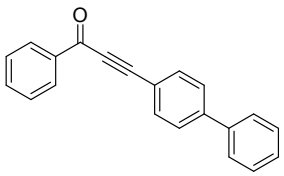
1-Phenyl-3-(p-bomophenyl)prop-2-yn-1-one (3af): In an oven-dried Teflon septum screw-capped tube (15mL), N-benzoylsaccharin **1a** (0.5 mmol, 1.0 equiv) was reacted with 1-bromo-4-ethynylbenzene **2f** (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3af** (112.6 mg, 79%) as a pale yellow solid pale. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 – 8.18 (m, 2H), 7.65 (t, *J* = 7.4 Hz, 1H), 7.59 – 7.52 (m, 6H). Spectral data match those previously reported[6].



1-Phenyl-3-(4-trifluoromethylphenyl)prop-2-yn-1-one (3ag): In an oven-dried Teflon septum screw-capped tube (15mL), N-benzoylsaccharin **1a** (0.5 mmol,1.0 equiv) was reacted with 1-ethynyl-4-(trifluoromethyl)benzene **2g** (0.75 mmol, 1.5equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3ag** (93.2 mg, 68%) as a pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.24 – 8.19 (m, 2H), 7.79 (d, *J* = 8.0 Hz, 2H), 7.70 – 7.65 (m, 3H), 7.54 (t, *J* = 7.7 Hz, 2H). ^{19}F NMR (471 MHz, $CDCl_3$) δ -63.13. Spectral data match those previously reported[6].

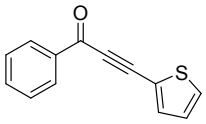


1-Phenyl-3-(4-methoxylphenyl)prop-2-yn-1-one (3ah): In an oven-dried Teflon septum screw-capped tube (15mL), N-benzoylsaccharin **1a** (0.5 mmol, 1.0 equiv) was reacted with 1-ethynyl-4-(trifluoromethyl)benzene **2h** (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (20/1) as eluent afforded the product **3ah** (94.5 mg, 80%) as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (dd, *J* = 8.2, 1.1 Hz, 2H), 7.68 – 7.61 (m, 3H), 7.52 (t, *J* = 7.6 Hz, 2H), 6.96 – 6.92 (m, 2H), 3.87 (s, 3H). Spectral data match those previously reported[6].

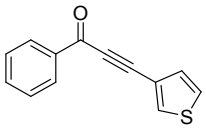


1-Phenyl-3-(4-diphenyl)prop-2-yn-1-one (3ai): In an oven-dried Teflon septum screw-capped tube (15mL), N-benzoylsaccharin **1a** (0.5 mmol, 1.0 equiv) was reacted

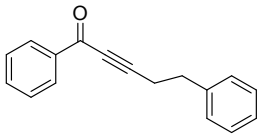
with 4-ethynyl-1,1'-biphenyl **2i** (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3ai** (120.0 mg, 85%) as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 – 8.19 (m, 2H), 7.77 – 7.74 (m, 2H), 7.67 – 7.60 (m, 6H), 7.52 (t, *J* = 7.6 Hz, 2H), 7.47 (dd, *J* = 10.1, 4.8 Hz, 2H), 7.41 – 7.37 (m, 1H). Spectral data match those previously reported[8].



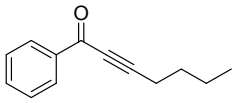
1-Phenyl-3-(thiophen-2-yl)prop-2-yn-1-one (3aj): In an oven-dried Teflon septum screw-capped tube (15mL), N-benzoylsaccharin **1a** (0.5 mmol, 1.0 equiv) was reacted with 2-ethynylthiophene **2j** (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (20/1) as eluent afforded the product **3aj** (73.2 mg, 69%) as a pale yellow solid. ^{1}H NMR (400 MHz,$CDCl_3$) δ 8.21 – 8.16 (m, 2H), 7.62 (d, *J* = 7.3 Hz, 1H), 7.59 (d, *J* = 3.7 Hz, 1H), 7.53 (dd, *J* = 10.6, 4.4 Hz, 3H), 7.11 (dd, *J* = 5.1, 3.8 Hz, 1H). Spectral data match those previously reported[6].



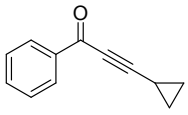
1-Phenyl-3-(thiophen-3-yl)prop-2-yn-1-one (3ak): In an oven-dried Teflon septum screw-capped tube (15mL), N-benzoylsaccharin **1a** (0.5 mmol, 1.0 equiv) was reacted with 3-ethynylthiophene **2k** (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (20/1) as eluent afforded the product **3ak** (94.5 mg, 89%) as a brown liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (dd, *J* = 5.3, 3.2 Hz, 2H), 7.86 (dd, *J* = 2.9, 1.0 Hz, 1H), 7.66 – 7.61 (m, 1H), 7.52 (dd, *J* = 10.3, 5.0 Hz, 2H), 7.39 (dd, *J* = 5.0, 2.9 Hz, 1H), 7.35 – 7.32 (m, 1H). Spectral data match those previously reported[9].



1,5-Diphenylpent-2-yn-1-one (3al): In an oven-dried Teflon septum screw-capped tube (15mL), N-benzoylsaccharin **1a** (0.5 mmol, 1.0 equiv) was reacted with but-3-yn-1-ylbenzene 2l (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (20/1) as eluent afforded the product **3al** (94.9 mg, 81%) as a pale yellow liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 (d, *J* = 7.4 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.42 (t, *J* = 7.8 Hz, 2H), 7.36 – 7.32 (m, 2H), 7.27 (d, *J* = 7.9 Hz, 2H), 2.99 (t, *J* = 7.3 Hz, 2H), 2.82 (t, *J* = 7.3 Hz, 2H). Spectral data match those previously reported[10].

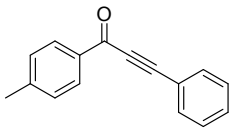


1-Phenyl-3-hept-2-yn-1-one (3am): In an oven-dried Teflon septum screw-capped tube (15mL), N-benzoylsaccharin **1a** (0.5 mmol, 1.0 equiv) was reacted with hex-1-yne **2m** (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3am** (54.0 mg, 58%) as a yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 7.7 Hz, 2H), 7.59 (t, *J* = 7.3 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 2.50 (t, *J* = 7.1 Hz, 2H), 1.69 – 1.63 (m, 2H), 1.50 (dd, *J* = 14.9, 7.4 Hz, 2H), 0.96 (t, *J* = 7.3 Hz, 3H). Spectral data match those previously reported[11].

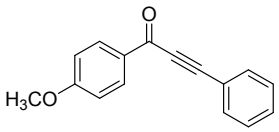


3-Cyclopropyl-1-phenylprop-2-yn-1-one (3an): In an oven-dried Teflon septum screw-capped tube (15mL), N-benzoylsaccharin **1a** (0.5 mmol, 1.0 equiv) was reacted with ethynylcyclopropane **2n** (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3an** (54.0 mg, 58%) as a colorless liquid. ^{1}H

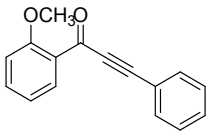
NMR (500 MHz, $CDCl_3$) δ 8.13 – 8.07 (m, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 2H), 1.54 (tt, *J* = 7.8, 5.3 Hz, 1H), 1.06 – 1.00 (m, 4H). Spectral data match those previously reported[6].



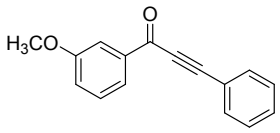
1-(4-p-tolyl)-3-phenylprop-2-yn-1-one (3ba):[8] In an oven-dried Teflon septum screw-capped tube (15mL), N-4-methylbenzoyl saccharin **1b** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3ah** (72.7 mg, 66%) as a yellow oil. ^1H NMR (500 MHz, CDCl3) δ 8.11 (d, *J* = 8.1 Hz, 2H), 7.68 – 7.65 (m, 2H), 7.46 (t, *J* = 7.4 Hz, 1H), 7.40 (t, *J* = 7.4 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 2.43 (s, 3H). Spectral data match those previously reported[12].



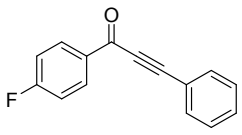
1-(4-methoxyl)-3-phenylprop-2-yn-1-one (3ca):[2] In an oven-dried Teflon septum screw-capped tube (15mL), N-4-methoxybenzoyl saccharin **1c** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (20/1) as eluent afforded the product **3ca** (81.5 mg, 69%) as a pale yellow solid. ^1H NMR (500 MHz, $CDCl_3$) δ 8.25 – 8.14 (m, 2H), 7.71 – 7.65 (m, 2H), 7.49 – 7.45 (m, 1H), 7.42 (t, *J* = 7.4 Hz, 2H), 6.99 (d, *J* = 8.9 Hz, 2H), 3.90 (s, 3H). Spectral data match those previously reported[6].



1-(2-Methoxyphenyl)-3-(p-tolyl)prop-2-yn-1-one(3da):[2] In an oven-dried Teflon septum screw-capped tube (15mL), N-2-methoxybenzoyl saccharin **1d** (0.5mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (20/1) as eluent afforded the product **3da** (57.9 mg, 49%) as a pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (dd, *J* = 7.8, 1.6 Hz, 2H), 7.51 – 7.48 (m, 1H), 7.33 – 7.29 (m, 4H), 6.93 (dd, *J* = 10.9, 4.1 Hz, 1H), 6.89 (d, *J* = 8.4 Hz, 1H), 3.90 (s, 3H). Spectral data match those previously reported[6].

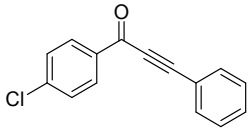


1-(3-methoxylphenyl)-3-phenylprop-2-yn-1-one (3ea): In an oven-dried Teflon septum screw-capped tube (15 mL), N-3-methoxybenzoyl saccharin **1e** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (20/1) as eluent afforded the product **3da** (73.2 mg, 62%) as a pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88 – 7.85 (m, 1H), 7.71 – 7.67 (m, 3H), 7.48 (t, *J* = 7.5 Hz, 1H), 7.42 (dd, *J* = 7.7, 2.4 Hz, 3H), 7.19 – 7.16 (m, 1H), 3.88 (s, 3H). Spectral data match those previously reported[13].

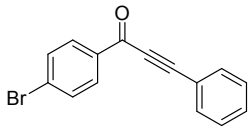


1-(4-fluorophenyl)-3-phenylprop-2-yn-1-one (3fa): In an oven-dried Teflon septum screw-capped tube (15 mL), N-4-fluorobenzoylsaccharin **1f** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate

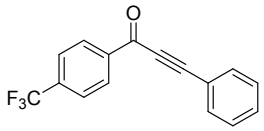
(40/1) as eluent afforded the product **3ca** (76.2 mg, 68%) as a pale yellow solid pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 – 8.20 (m, 2H), 7.72 – 7.68 (m, 2H), 7.51 (ddd, *J* = 6.2, 3.6, 1.1 Hz, 1H), 7.47 – 7.42 (m, 2H), 7.23 – 7.18 (m, 2H). ^{19}F NMR (376 MHz, $cdcl_3$) δ -102.98, -102.99, -103.00, -103.02, -103.02. Spectral data match those previously reported[6].



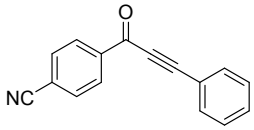
1-(4-Chlorophenyl)-3-(p-tolyl)prop-2-yn-1-one (3ga): In an oven-dried Teflon septum screw-capped tube (15 mL), N-4-chlorobenzoylsaccharin **1g** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3ga** (103.5 mg, 86%) as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 – 8.20 (m, 2H), 7.72 – 7.68 (m, 2H), 7.51 (ddd, *J* = 6.2, 3.6, 1.1 Hz, 1H), 7.47 – 7.42 (m, 2H), 7.23 – 7.18 (m, 2H). Spectral data match those previously reported[6].



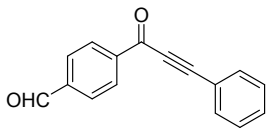
1-(4-bromophenyl)-3-phenylprop-2-yn-1-one (3ha): In an oven-dried Teflon septum screw-capped tube (15mL), 4-bromobenzoylsaccharin **1h** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3ha** (87.0mg, 61%) as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 7.8 Hz, 2H), 7.67 (t, *J* = 7.5 Hz, 4H), 7.49 (d, *J* = 7.6 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 2H). Spectral data match those previously reported[6].



3-phenyl-1-(4-(trifluoromethyl)phenyl)prop-2-yn-1-one (3ia): In an oven-dried Teflon septum screw-capped tube (15 mL), N-4-(trifluoromethyl)benzoyl saccharin **1i** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3ia** (87.8 mg, 64%) as a pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.32 (d, *J* = 8.0 Hz, 2H), 7.78 (d, *J* = 8.1 Hz, 2H), 7.69 (d, *J* = 7.9 Hz, 2H), 7.51 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 2H). ^{19}F NMR (471 MHz, CDCl3) δ -63.11. Spectral data match those previously reported[6].

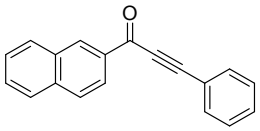


1-(4-cyanophenyl)-3-phenylprop-2-yn-1-one (3ja): In an oven-dried Teflon septum screw-capped tube (15mL), N-4-cyanobenzoylsaccharin **1j** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3ja** (72.8 mg, 63%) as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 – 8.31 (m, 2H), 7.87 – 7.82 (m, 2H), 7.75 – 7.70 (m, 2H), 7.55 (ddd, *J* = 7.5, 3.9, 1.3 Hz, 1H), 7.50 – 7.45 (m, 2H). Spectral data match those previously reported[14].

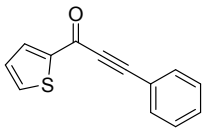


1-(4-formalphenyl)-3-phenylprop-2-yn-1-one (3ka): In an oven-dried Teflon septum screw-capped tube (15 mL), N-4-formylbenzoylsaccharin **1k** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl

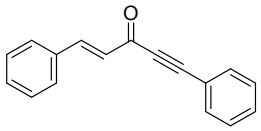
acetate (10/1) as eluent afforded the product **3ka** (59.7 mg, 51%) as a pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.15 (s, 1H), 8.37 (d, *J* = 8.2 Hz, 2H), 8.03 (d, *J* = 8.2 Hz, 2H), 7.71 (d, *J* = 7.2 Hz, 2H), 7.52 (t, *J* = 7.5 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 2H). Spectral data match those previously reported[15].



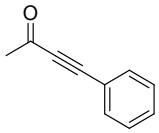
1-(Naphthalen-2-yl)-3-phenylprop-2-yn-1-one (3la): In an oven-dried Teflon septum screw-capped tube (15 mL), N-2-naphthoylsaccharin **1l** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3la** (97.7 mg, 76%) as a pale yellow solid. 1H NMR (500 MHz, $CDCl_3$) δ 8.75 (s, 1H), 8.19 (dd, *J* = 8.6, 1.6 Hz, 1H), 7.99 (d, *J* = 8.1 Hz, 1H), 7.87 (dd, *J* = 12.0, 8.4 Hz, 2H), 7.73 – 7.69 (m, 2H), 7.62 – 7.58 (m, 1H), 7.55 (dd, *J* = 11.0, 3.9 Hz, 1H), 7.49 – 7.45 (m, 1H), 7.42 (t, *J* = 7.3 Hz, 2H). Spectral data match those previously reported[6].



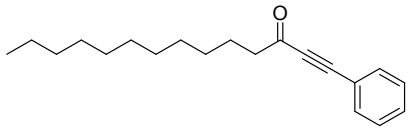
1-(Thiophen-2-yl)-3-(p-tolyl)prop-2-yn-1-one (3ma): In an oven-dried Teflon septum screw-capped tube (15mL), N-thiophene-2-carbonyl saccharin **1m** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3ma** (65.8 mg, 62%) as a pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (dd, *J* = 3.8, 1.0 Hz, 1H), 7.64 (dd, *J* = 4.9, 1.0 Hz, 1H), 7.59 – 7.56 (m, 2H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 2H), 7.10 (dd, *J* = 4.7, 4.0 Hz, 1H). Spectral data match those previously reported[6].



1-pentene-5-phenyl-4-yne-3-one (3na): In an oven-dried Teflon septum screw-capped tube (15 mL), N-cinnamoylsaccharin **1n** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (40/1) as eluent afforded the product **3na** (113.8 mg, 98%) as a pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90 (d, *J* = 16.1 Hz, 1H), 7.66 – 7.61 (m, 2H), 7.58 (dd, *J* = 6.6, 2.8 Hz, 2H), 7.46 (dd, *J* = 8.6, 6.2 Hz, 1H), 7.43 – 7.38 (m, 5H), 6.85 (d, *J* = 16.1 Hz, 1H). Spectral data match those previously reported[16].



1-methyl-3-phenylprop-2-yne-1-one (3oa): In an oven-dried Teflon septum screw-capped tube (15 mL), N-acetylsaccharin **1o** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (50/1) as eluent afforded the product **3oa** (34.6 mg, 48%) as a yellow liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 – 7.55 (m, 2H), 7.45 (d, *J* = 7.5 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 3H), 2.46 (s, 3H). Spectral data match those previously reported[17].



1-undecyl-3-phenylprop-2-yne-1-one (3pa): In an oven-dried Teflon septum screw-capped tube (15 mL), N-dodecanoylsaccharin **1p** (0.5 mmol, 1.0 equiv) was reacted with phenylacetylene (0.75 mmol, 1.5 equiv) using standard procedures followed by silica gel column chromatography using petroleum ether/ethyl acetate (50/1)as eluent afforded the product **3pa** (86.6 mg, 58%) as a yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 – 7.51 (m, 2H), 7.48 – 7.42 (m, 1H), 7.41 – 7.35 (m, 2H), 2.66 (t, *J* =

7.4 Hz, 2H), 1.78 – 1.69 (m, 2H), 1.25 (s, 16H), 0.87 (t, J = 6.8 Hz, 3H). Spectral data match those previously reported[17].

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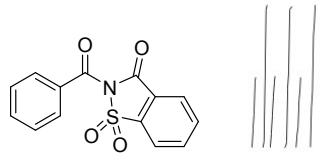
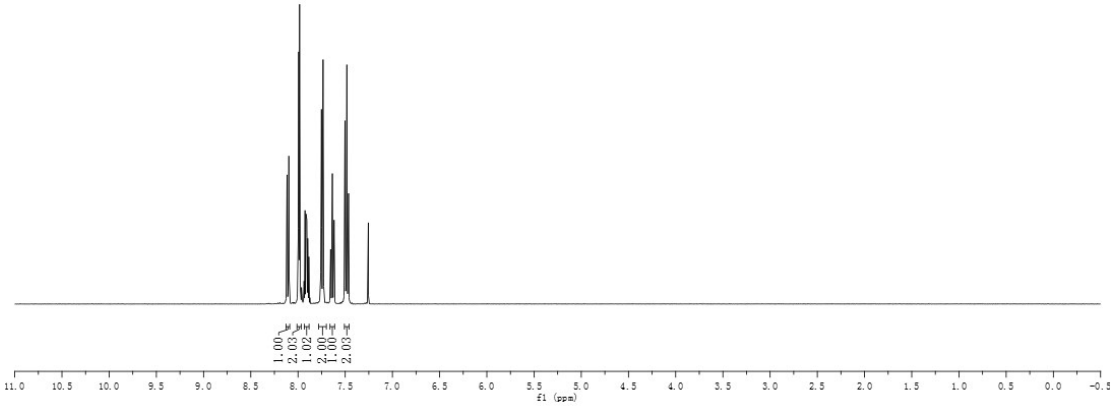
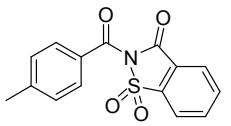
Copies of ^{1}H NMR, ^{19}F NMR and ^{13}C NMR of Starting Materials



^{1}H NMR (400 MHz, $CDCl_3$)

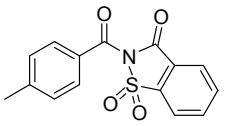
1a





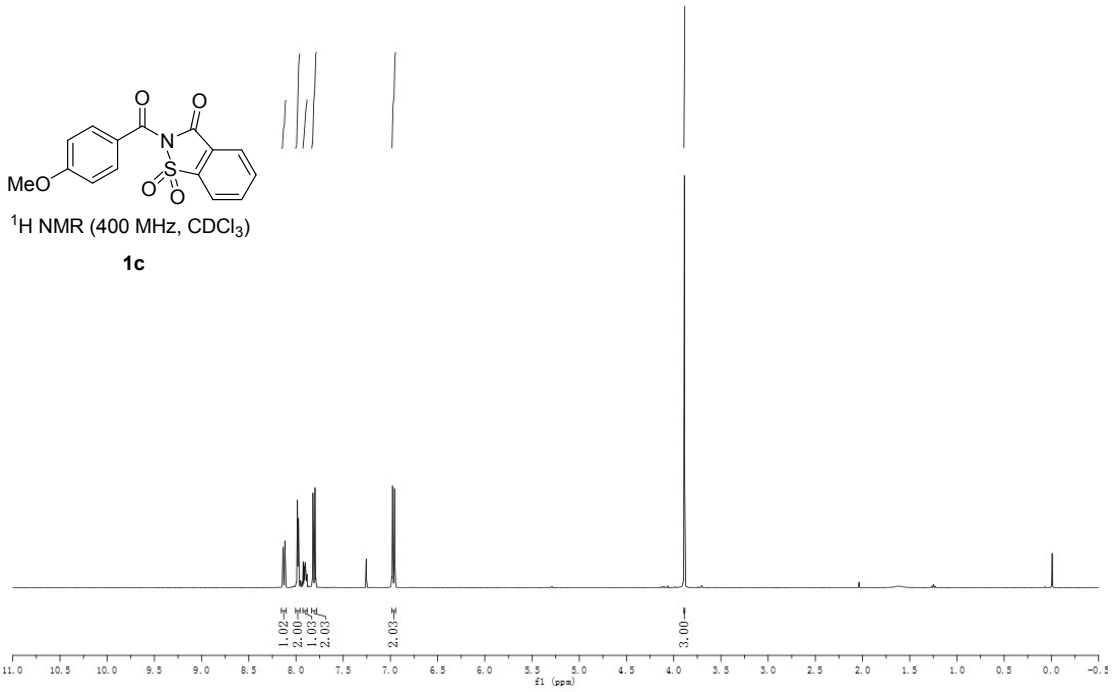
^{1}H NMR (400 MHz, $CDCl_3$)

1b



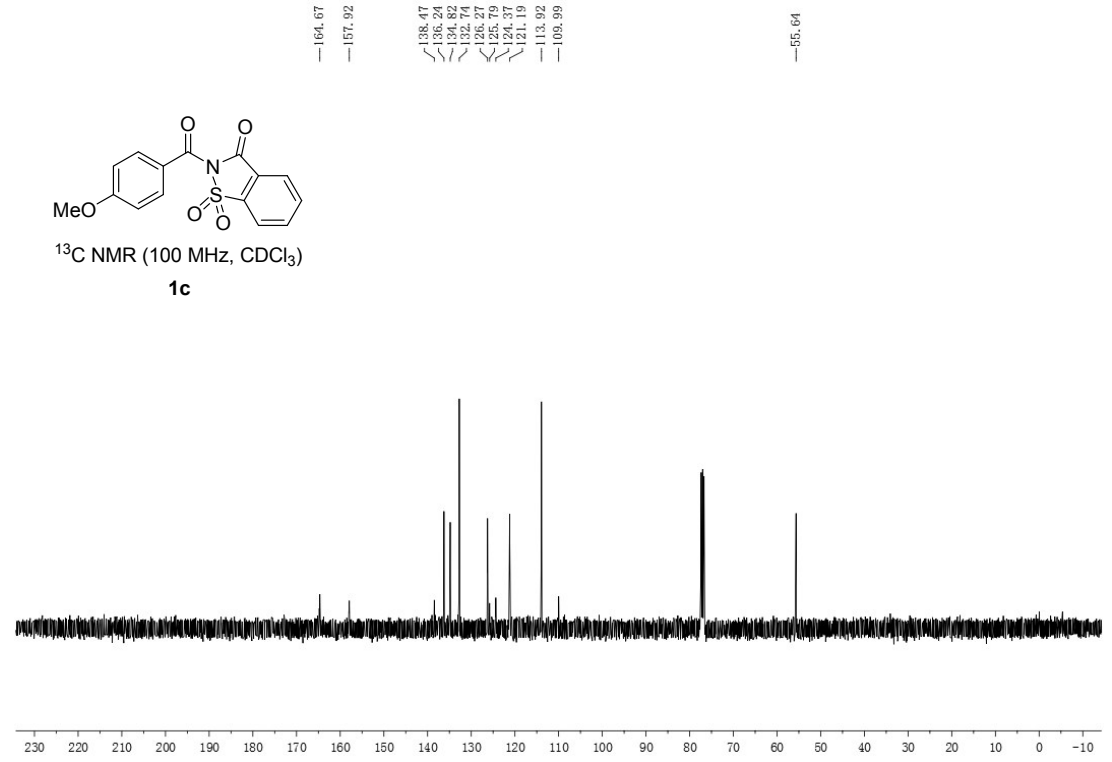
^{13}C NMR (100 MHz, $CDCl_3$)

1b



^{1}H NMR (400 MHz, $CDCl_3$)

1c



^{13}C NMR (100 MHz, $CDCl_3$)

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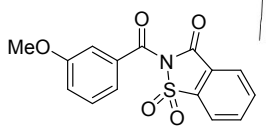

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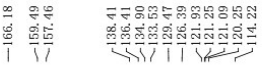
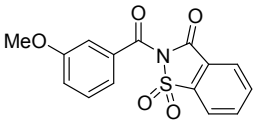
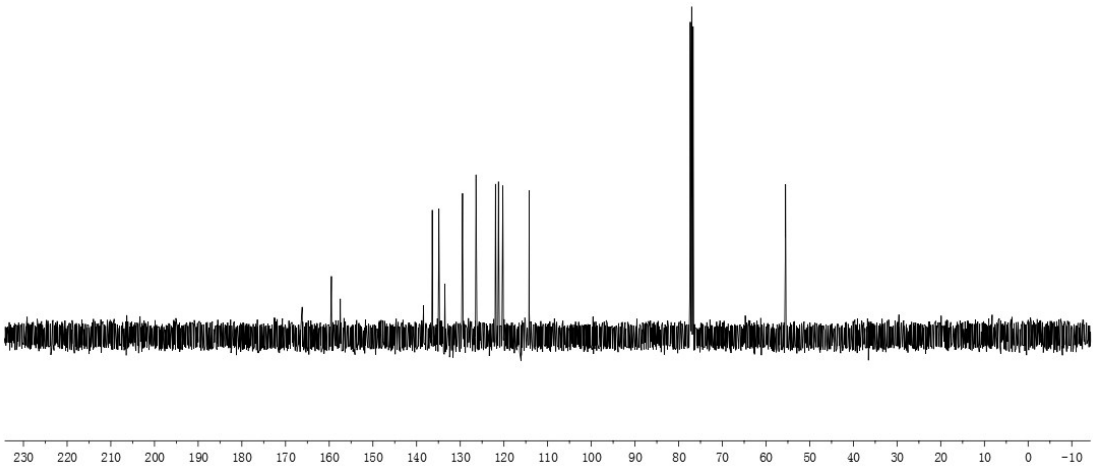
1d

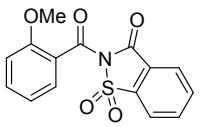




^{13}C NMR (100 MHz, $CDCl_3$)

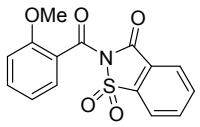
1d





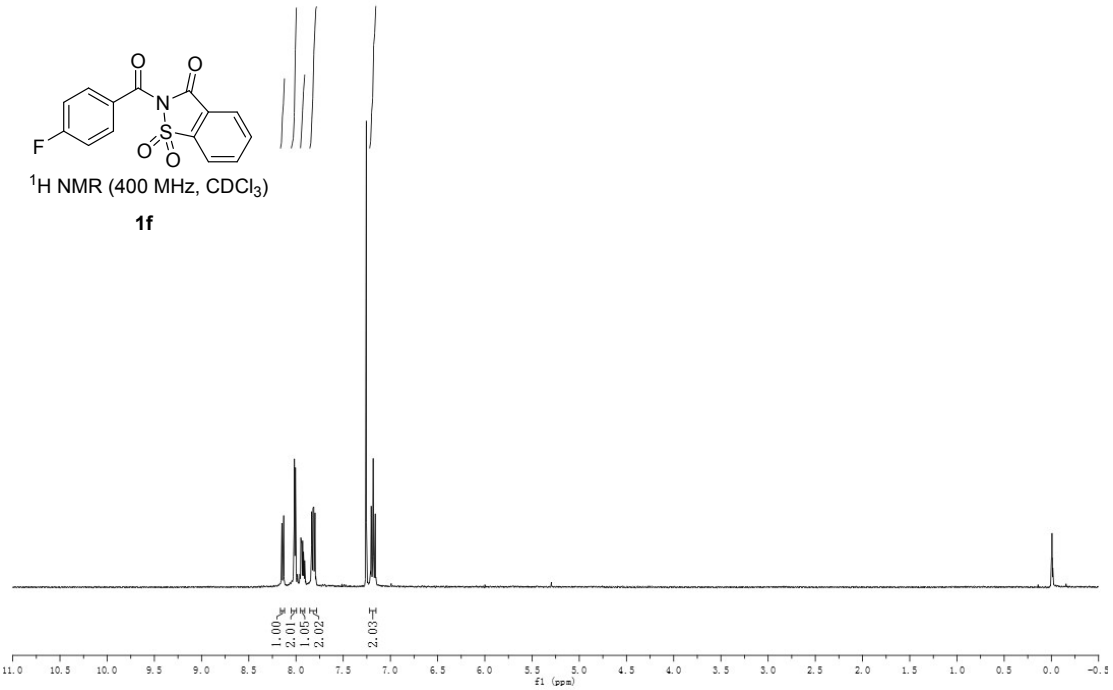
^{1}H NMR (400 MHz, $CDCl_3$)

1e



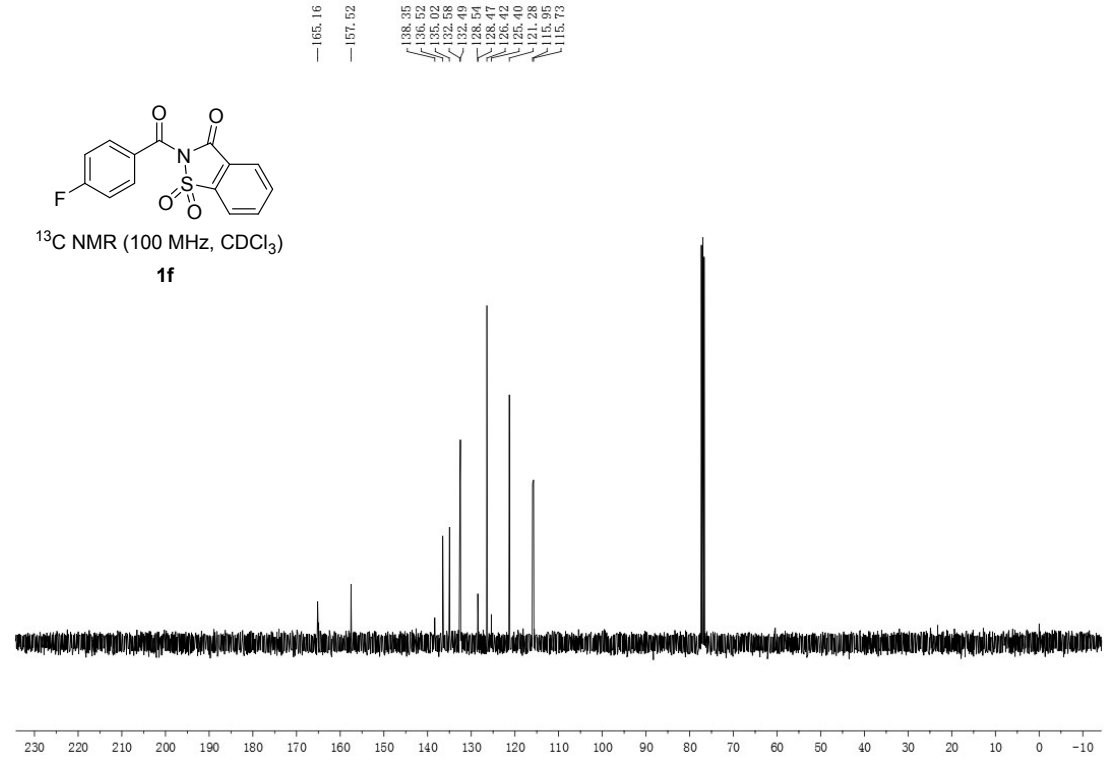
^{1}H NMR (400 MHz, $CDCl_3$)

1e



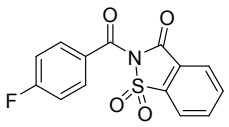
^{1}H NMR (400 MHz, $CDCl_3$)

1f



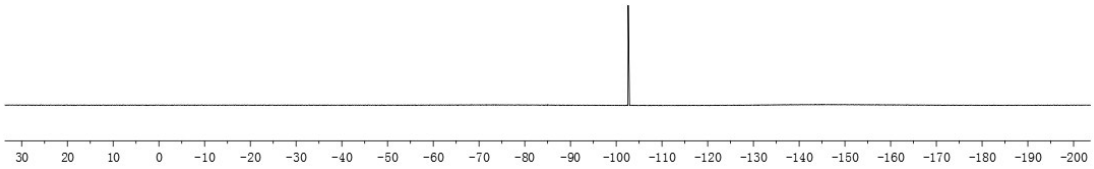
^{13}C NMR (100 MHz, $CDCl_3$)

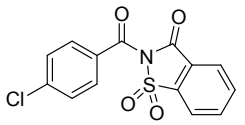
1f



^{19}F NMR (376 MHz, $CDCl_3$)

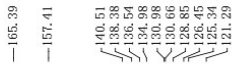
1f

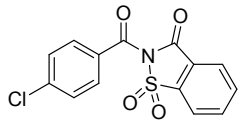




^{1}H NMR (400 MHz, $CDCl_3$)

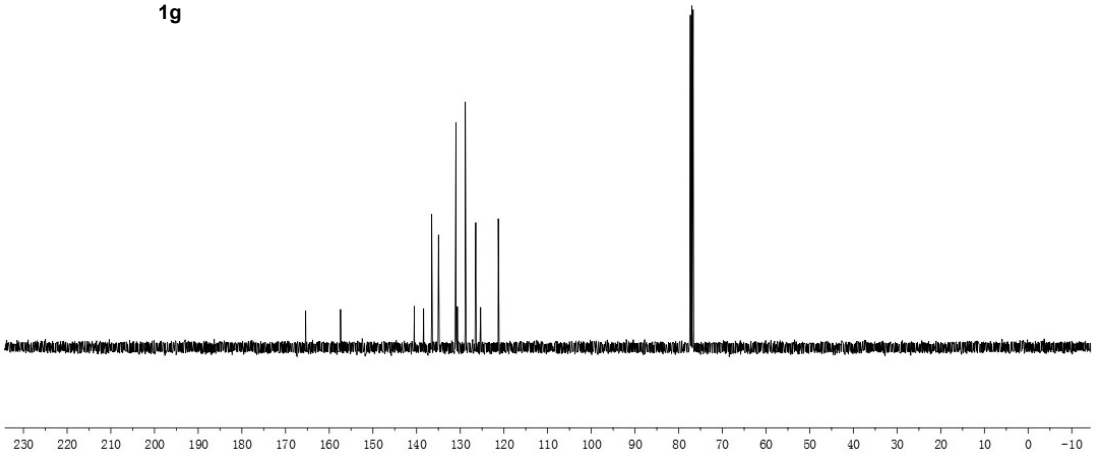
1g

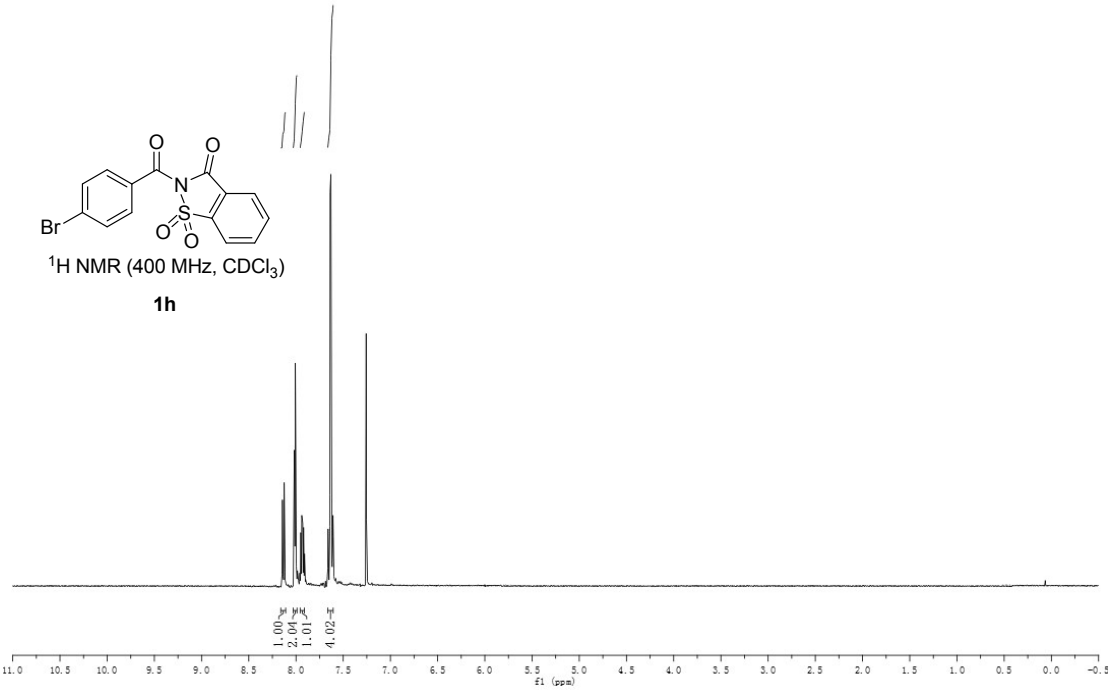




^{13}C NMR (100 MHz, $CDCl_3$)

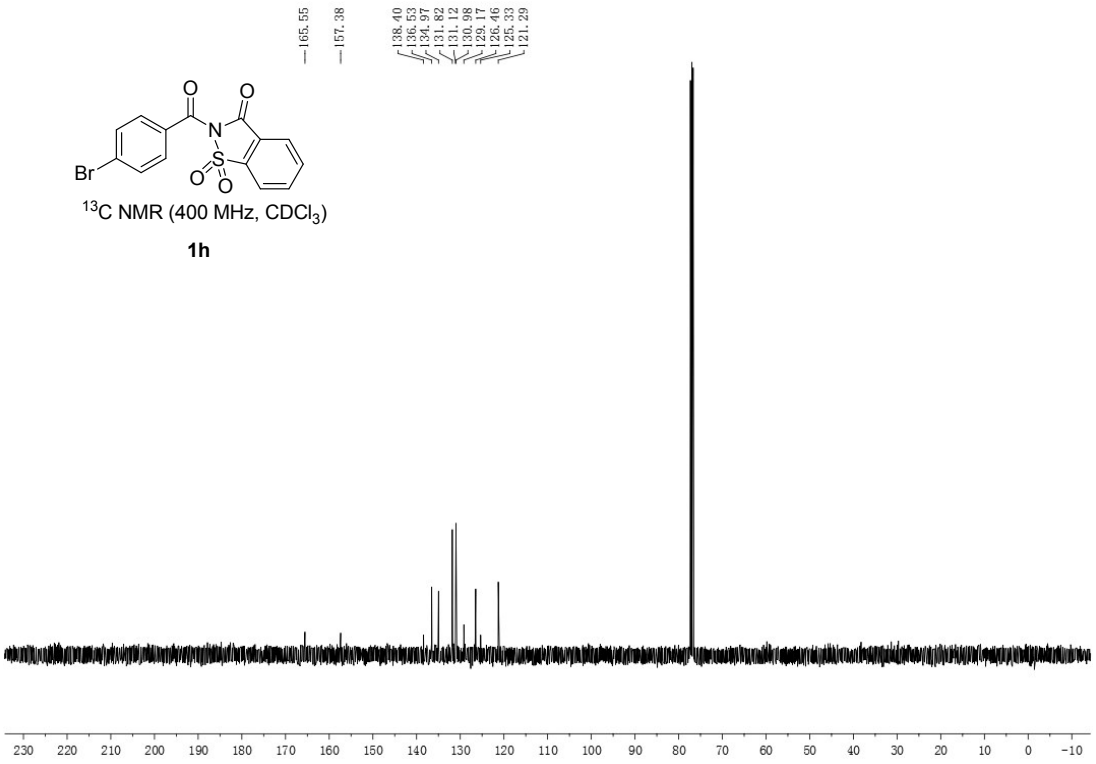
1g





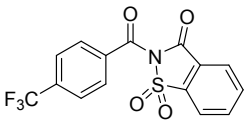
^{1}H NMR (400 MHz, $CDCl_3$)

1h



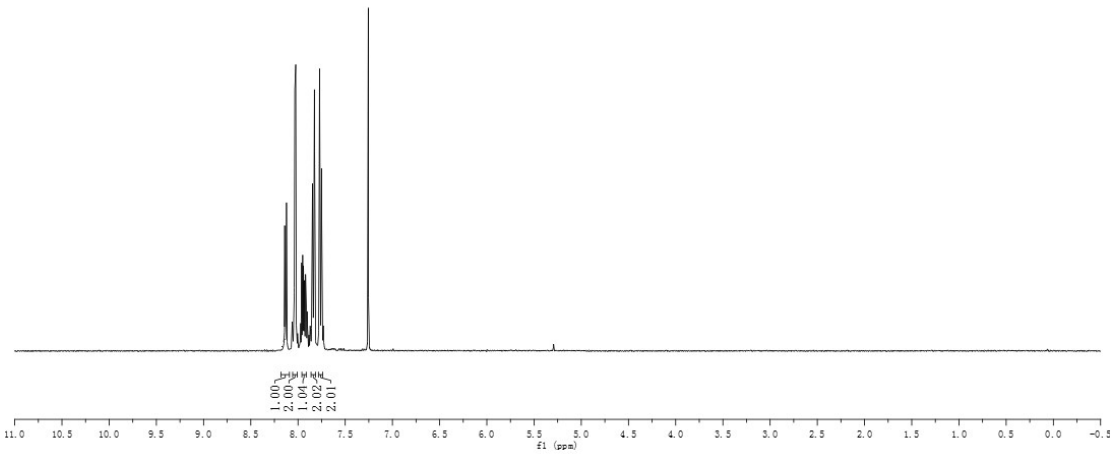
^{13}C NMR (400 MHz, $CDCl_3$)

1h



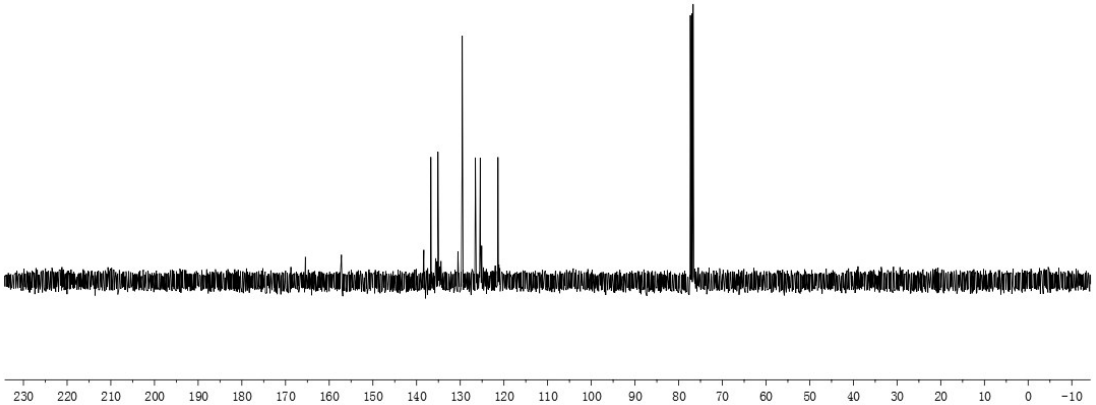
^{1}H NMR (400 MHz, $CDCl_3$)

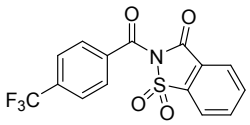
1i



^{13}C NMR (100 MHz, $CDCl_3$)

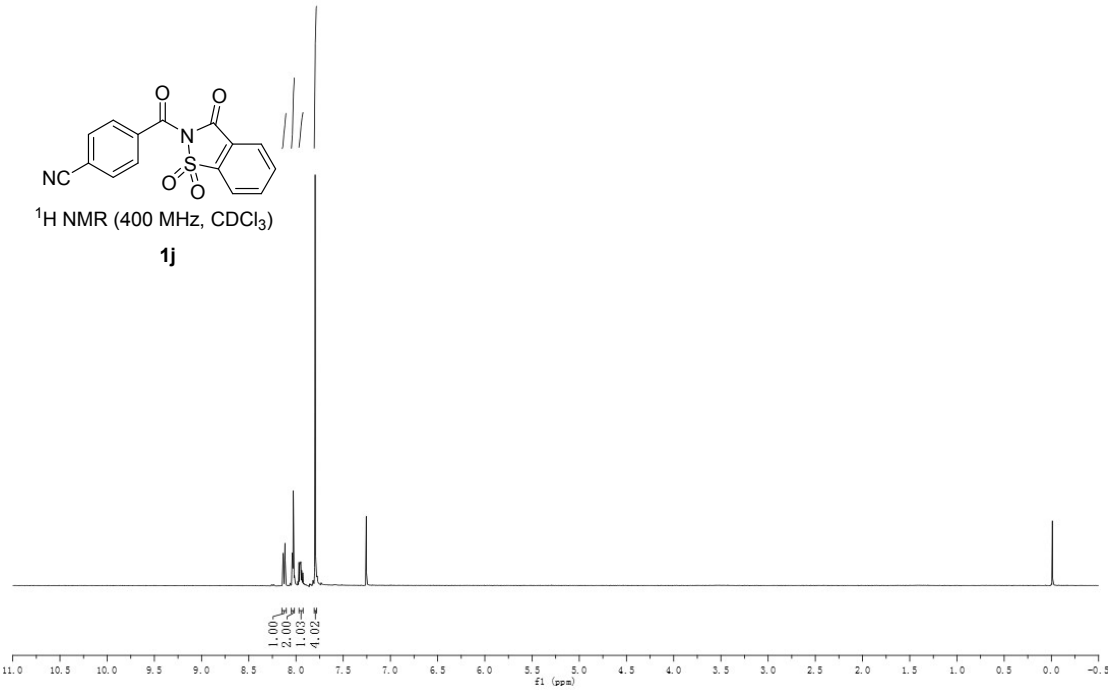
1i





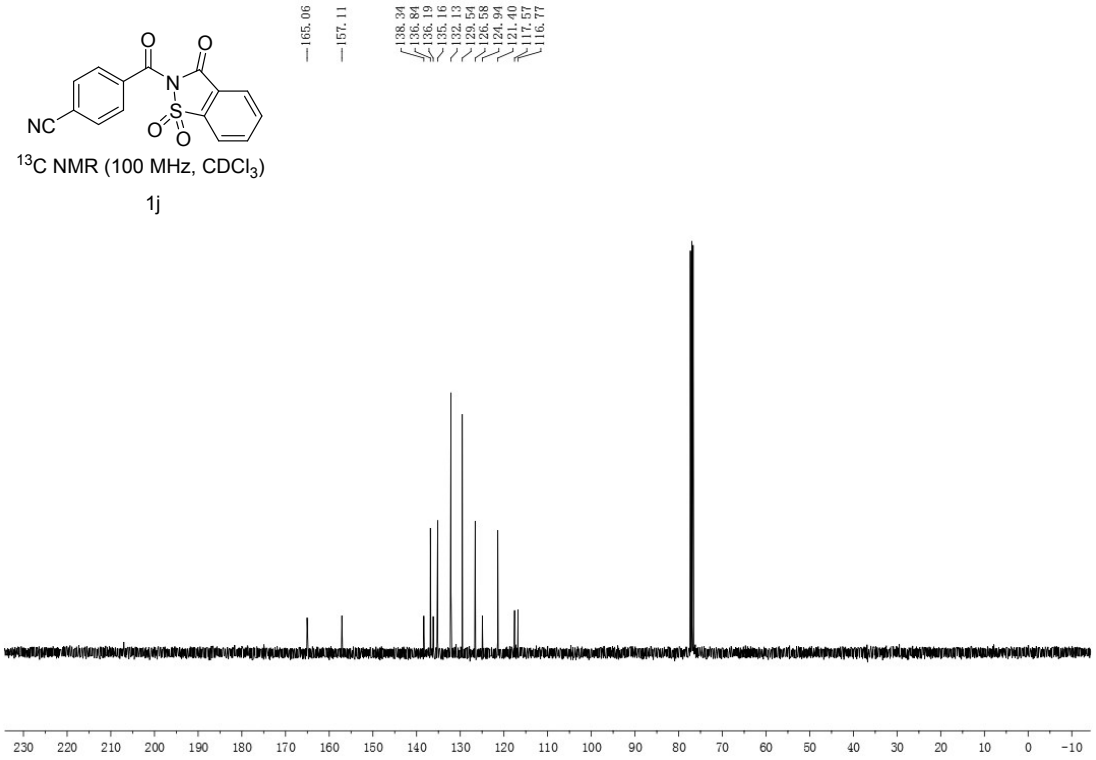
^{19}F NMR (376 MHz, $CDCl_3$)

1i



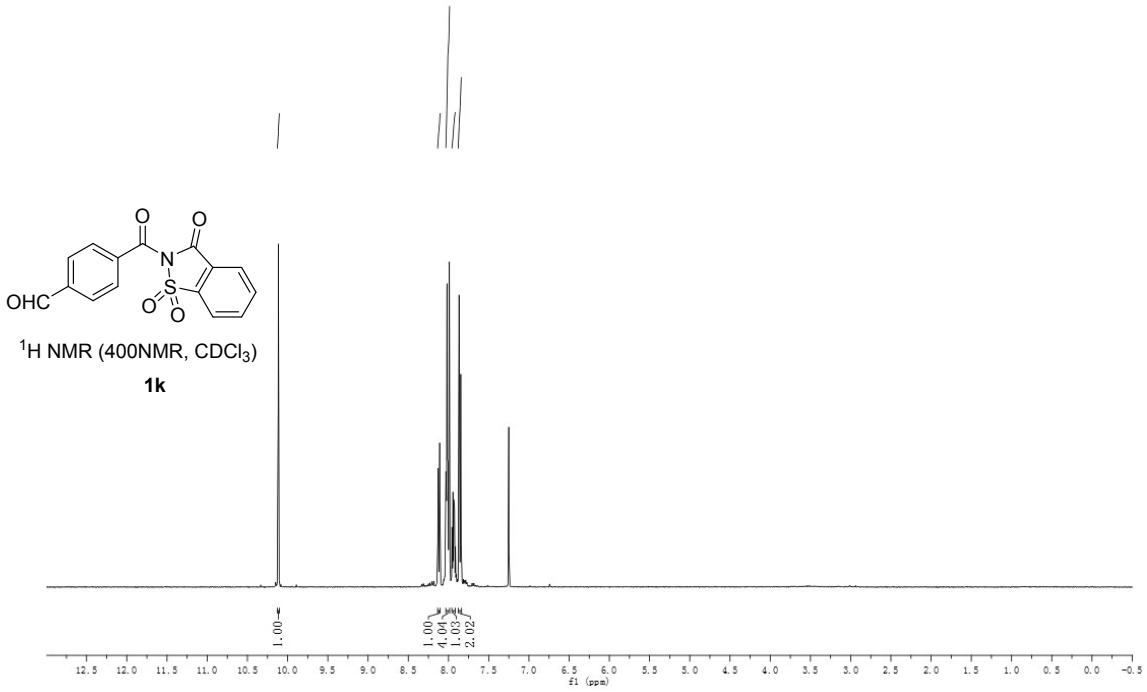
^{1}H NMR (400 MHz, $CDCl_3$)

1j



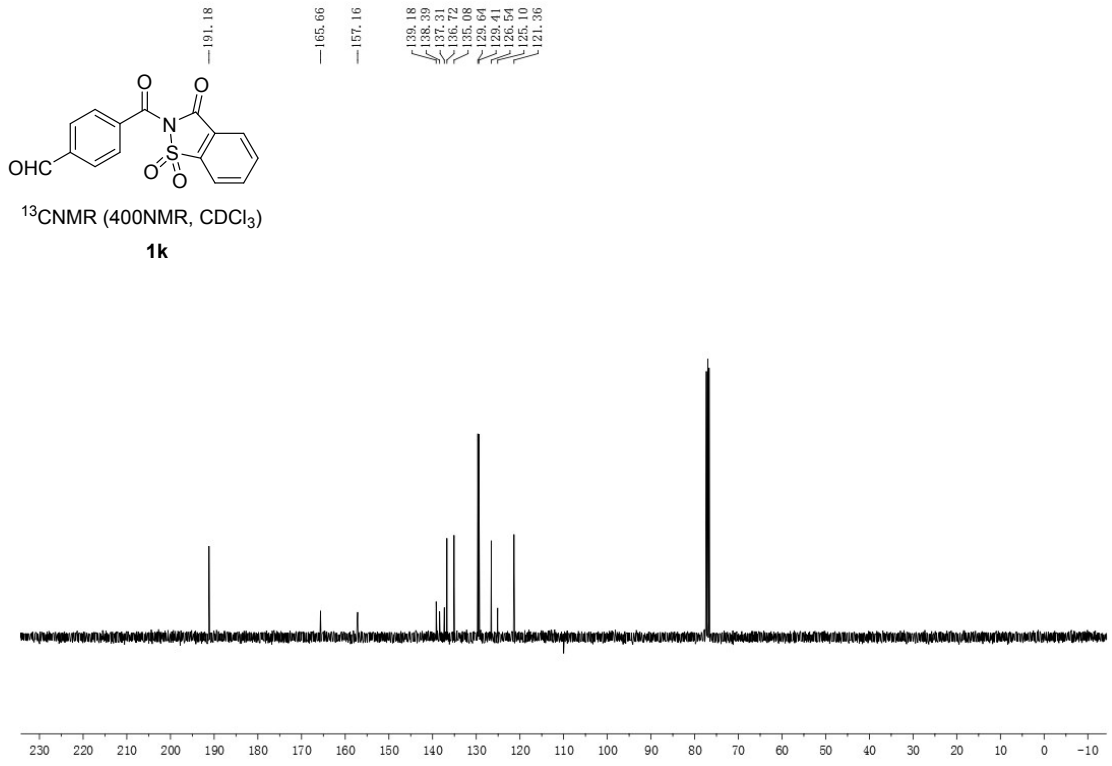
^{13}C NMR (100 MHz, $CDCl_3$)

1j



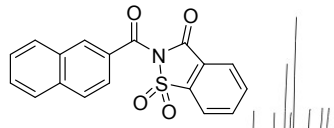
^{1}H NMR (400NMR, $CDCl_3$)

1k



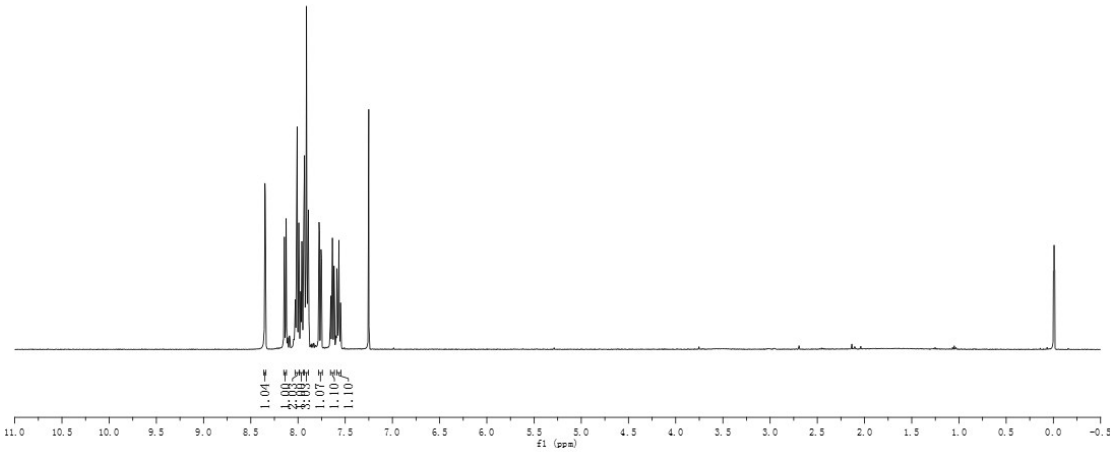
^{13}CNMR (400NMR, $CDCl_3$)

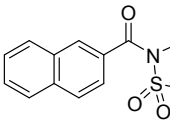
1k



^{1}H NMR (400 MHz, $CDCl_3$)

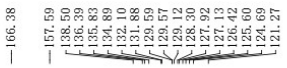
1l

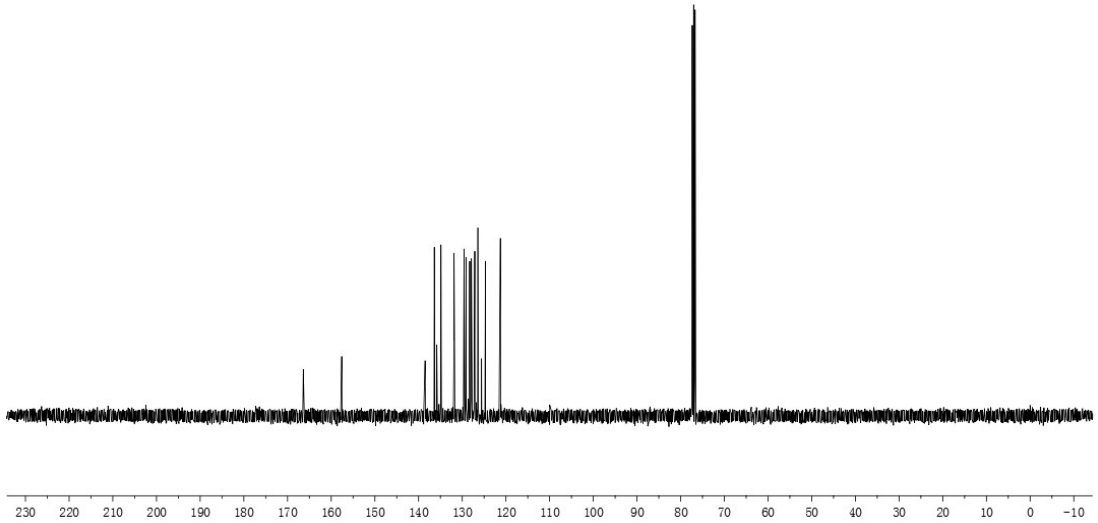


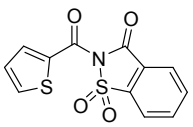


^{13}C NMR (100 MHz, $CDCl_3$)

1l

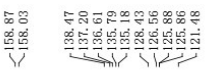


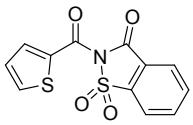




^{1}H NMR (400 MHz, $CDCl_3$)

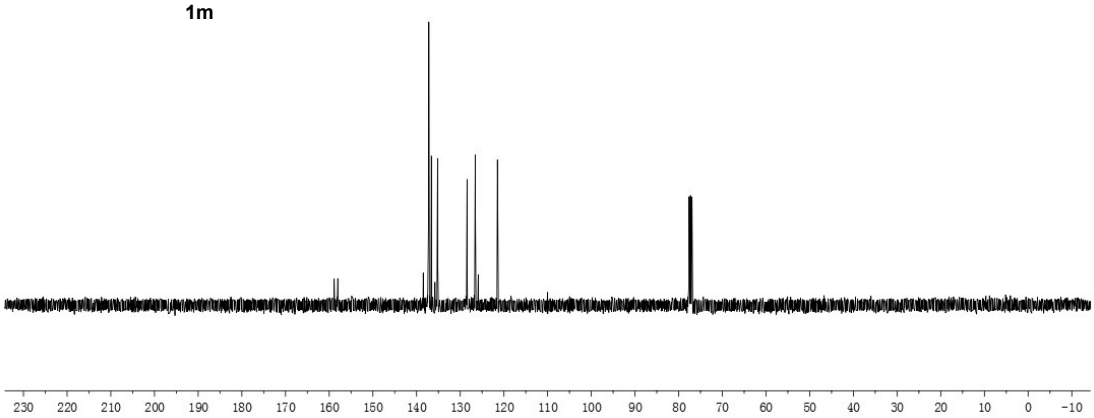
1m

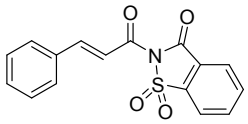




^{13}C NMR (100 MHz, $CDCl_3$)

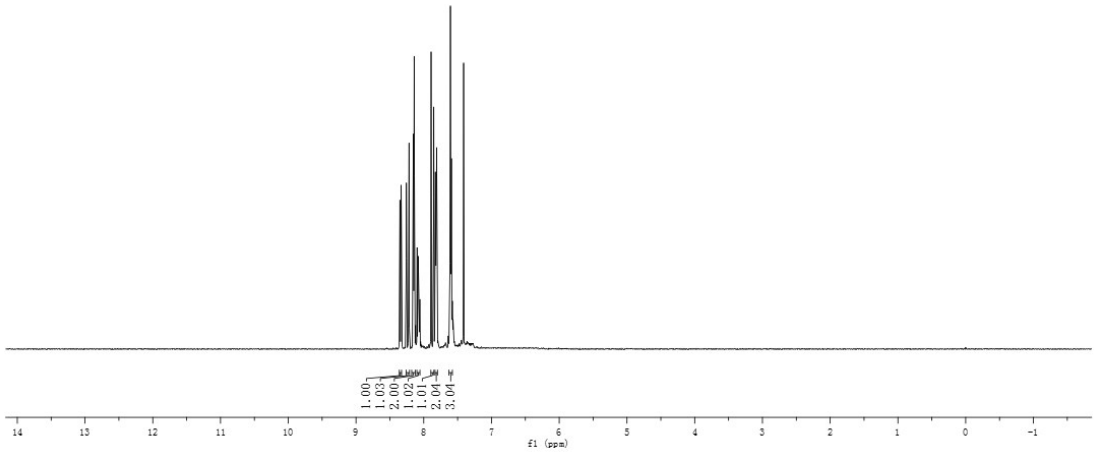
1m

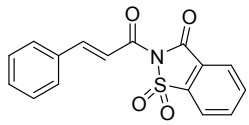




^{1}H NMR (400 MHz, $CDCl_3$)

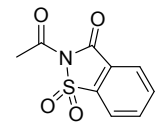
1n





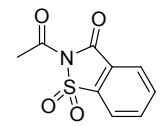
^{13}C NMR (100 MHz, $CDCl_3$)

1n



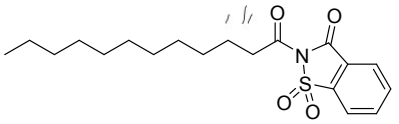
^{1}H NMR (400 MHz, $CDCl_3$)

1o



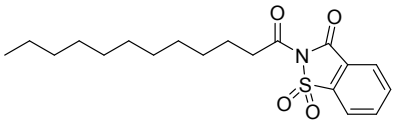
^{13}C NMR (100 MHz, $CDCl_3$)

1o



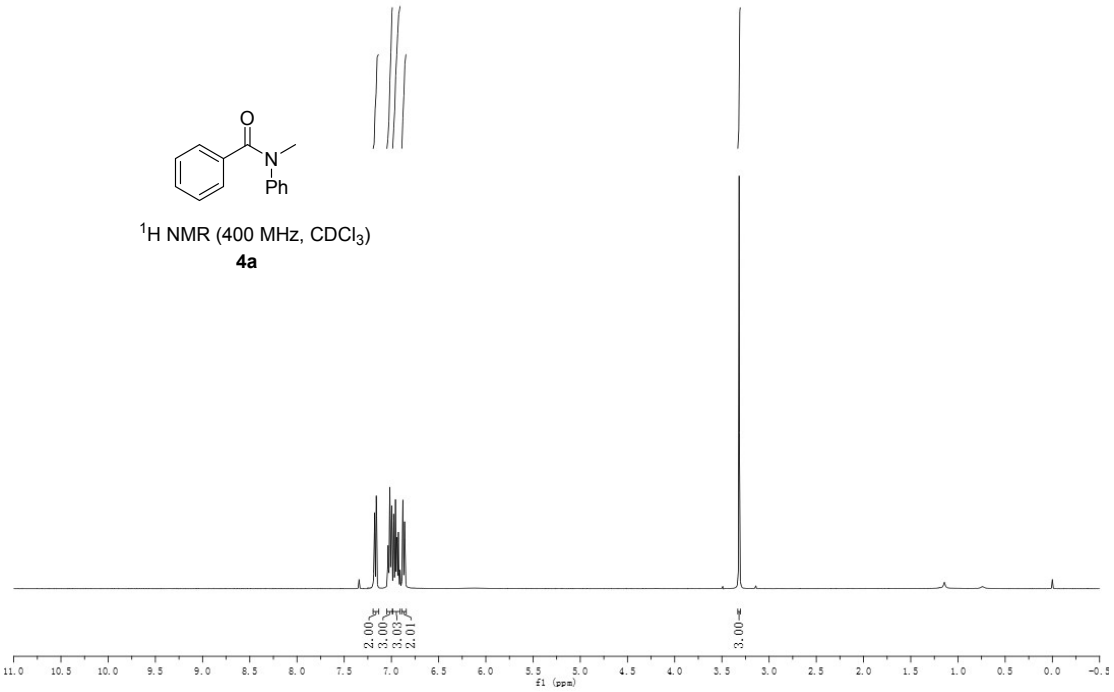
^{1}H NMR (400 MHz, $CDCl_3$)

1p



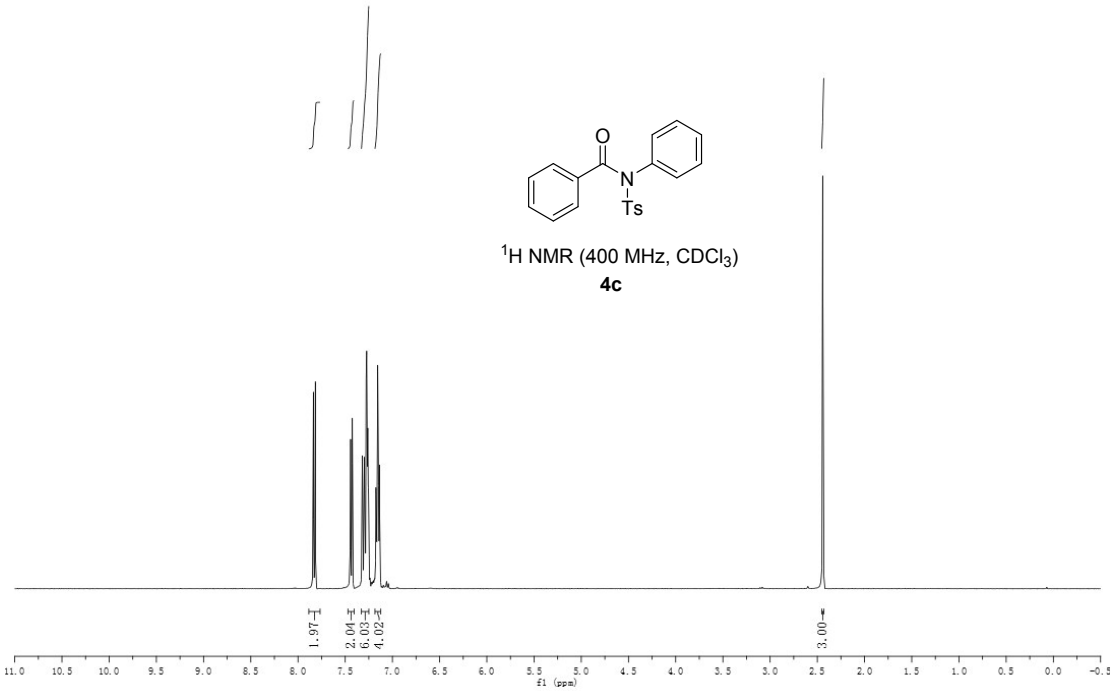
^{13}C NMR (100 MHz, $CDCl_3$)

1p



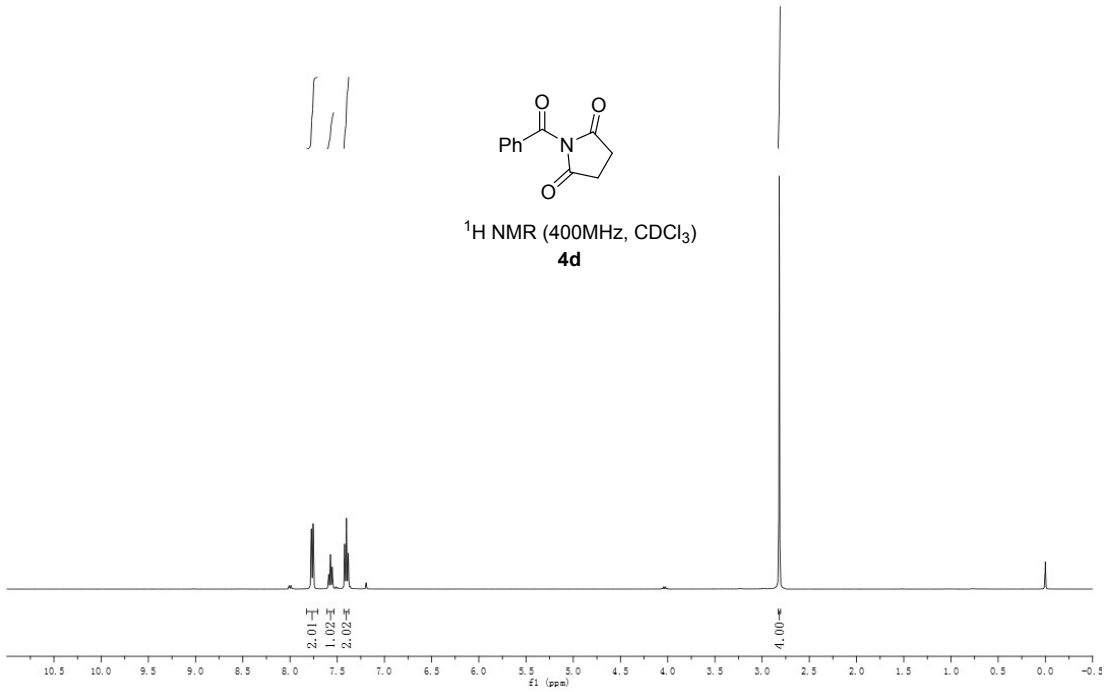
^{1}H NMR (400 MHz, $CDCl_3$)

4a



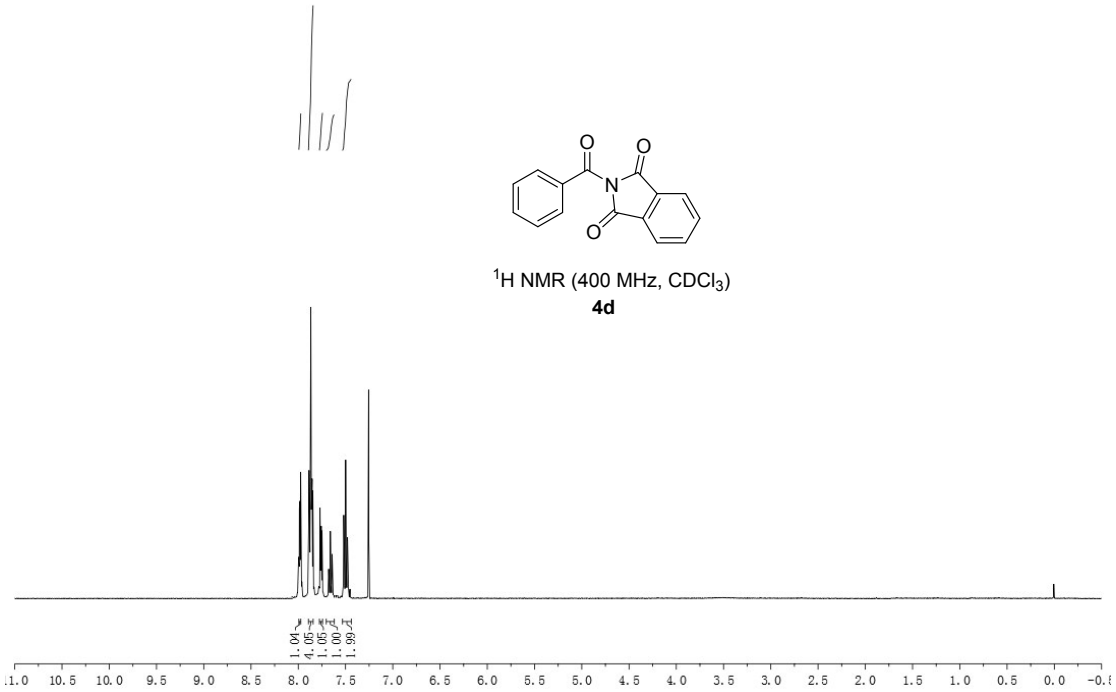
^{1}H NMR (400 MHz, $CDCl_3$)

4c



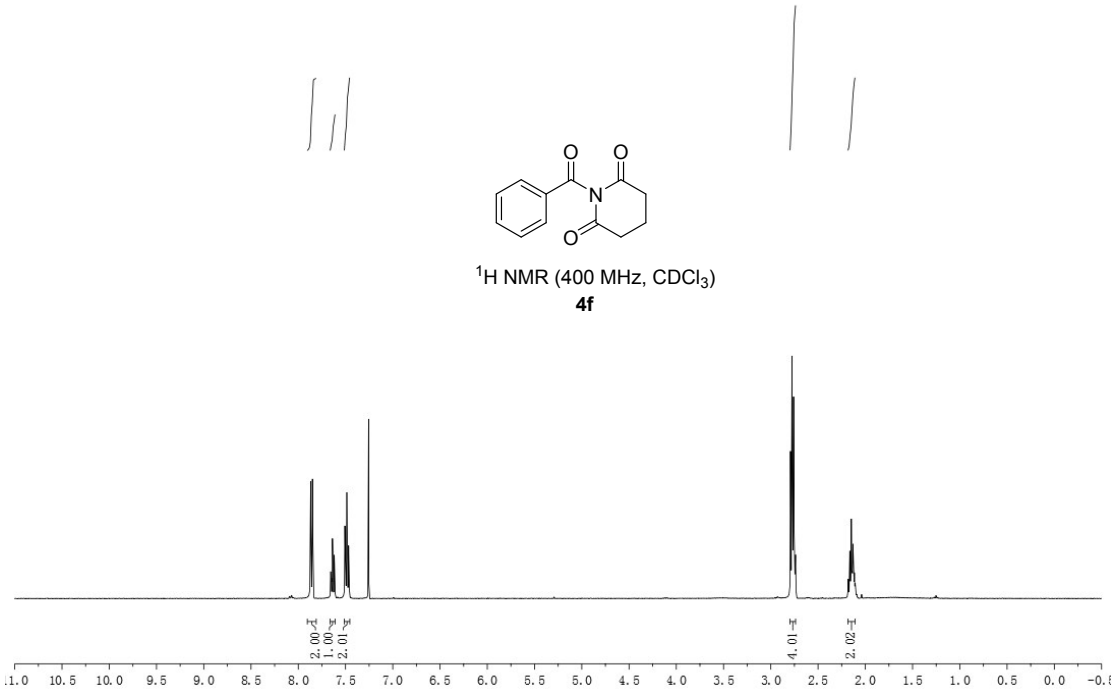
^{1}H NMR (400MHz, $CDCl_3$)

4d



^{1}H NMR (400 MHz, $CDCl_3$)

4d

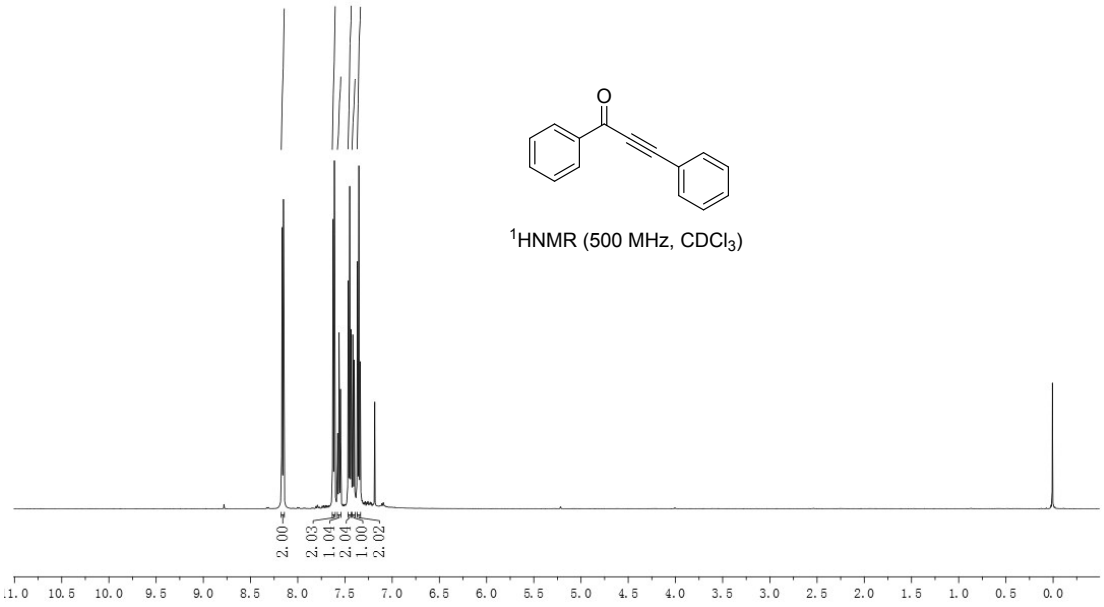


^{1}H NMR (400 MHz, $CDCl_3$)

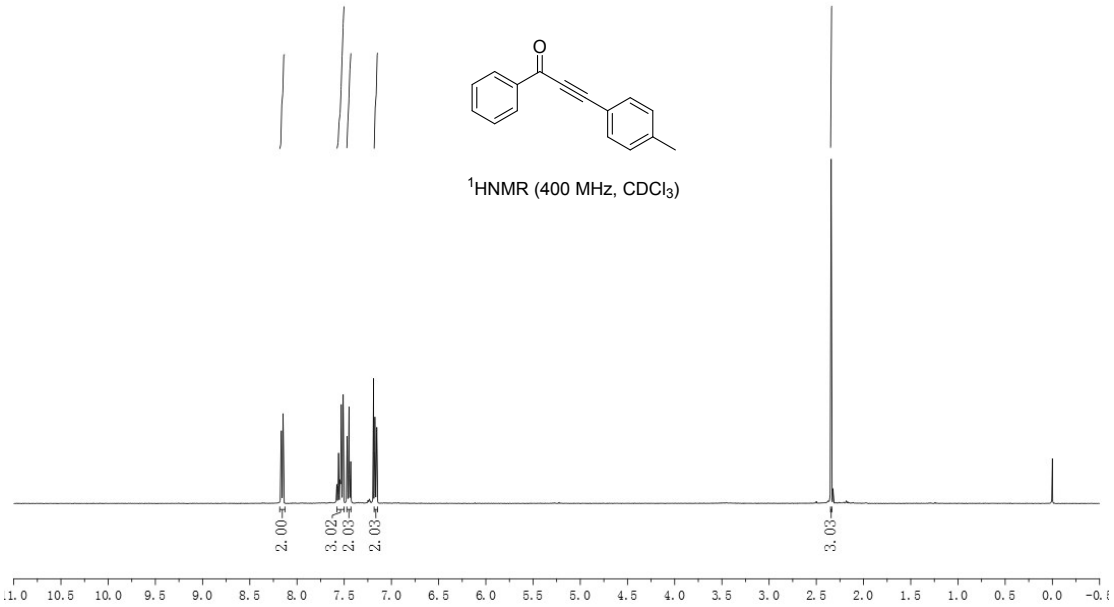
4f

Copies of ^{1}H NMR, ^{19}F NMR of Products

3aa

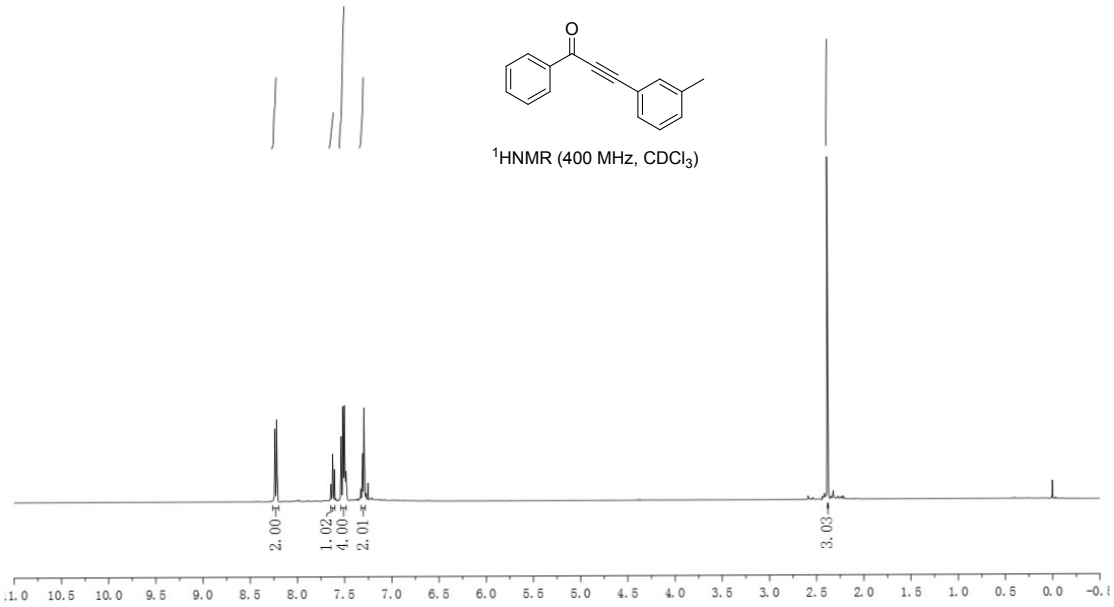


3ab



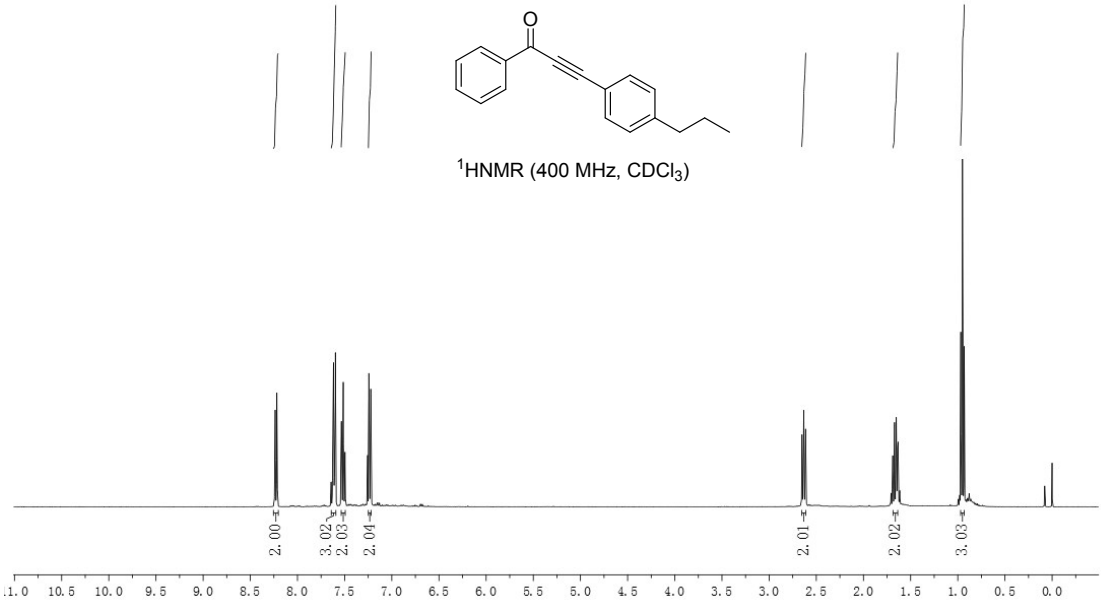
^{1}HNMR (400 MHz, $CDCl_3$)

3ac

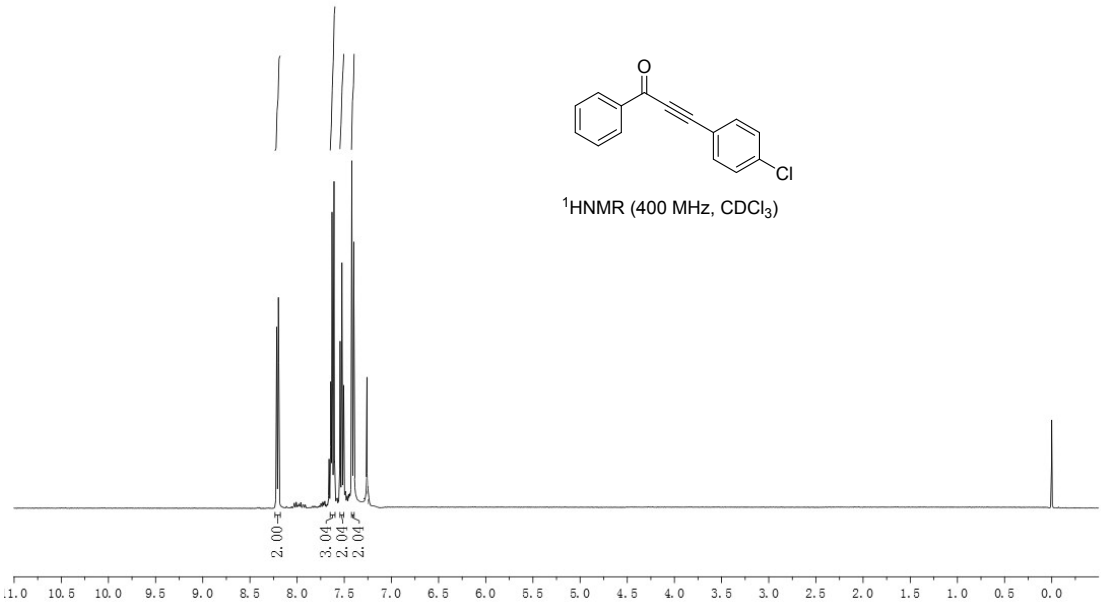


^{1}HNMR (400 MHz, $CDCl_3$)

3ad

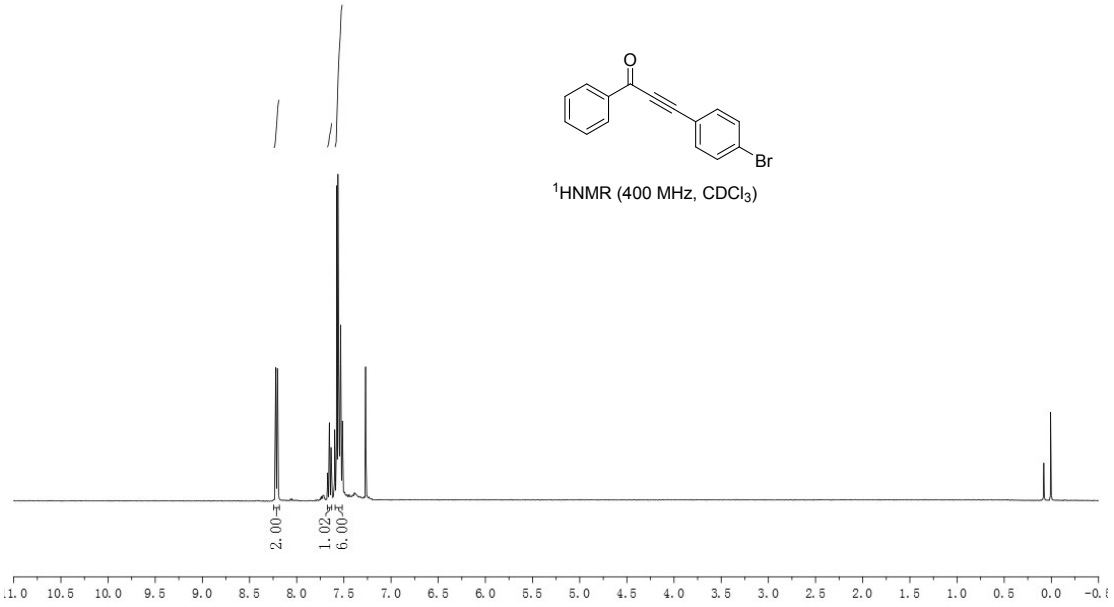

O
1HNMR (400 MHz, CDCl3)
2.00
3.02
2.03
2.04
2.01
2.02
3.03
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

3ae



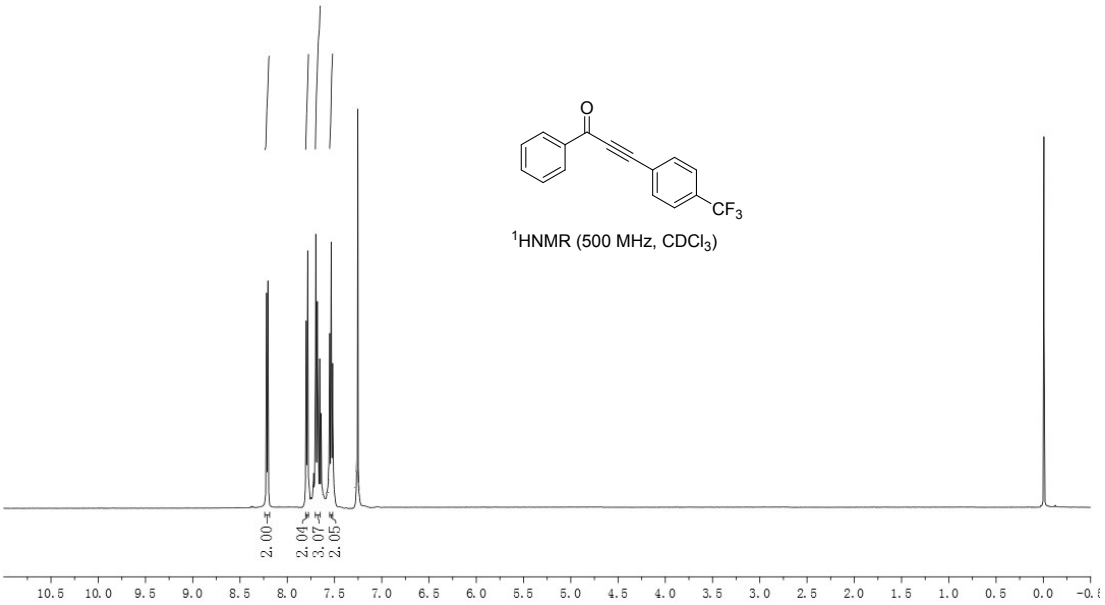
^{1}HNMR (400 MHz, $CDCl_3$)

3af

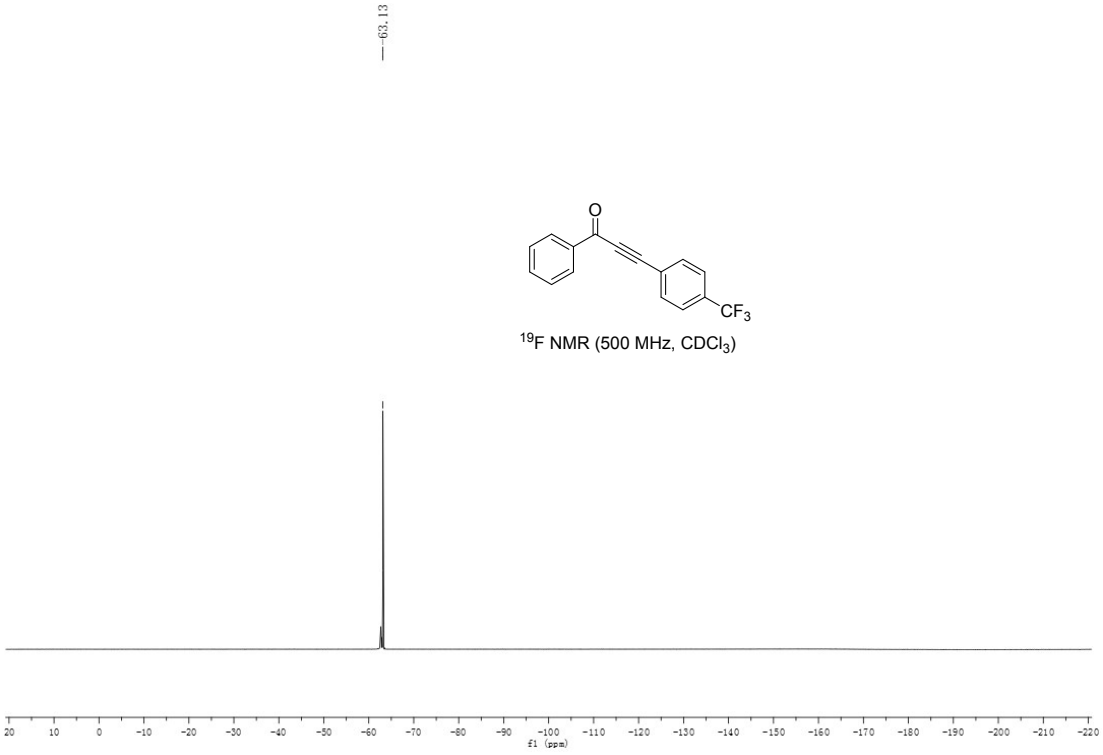


^{1}HNMR (400 MHz, $CDCl_3$)

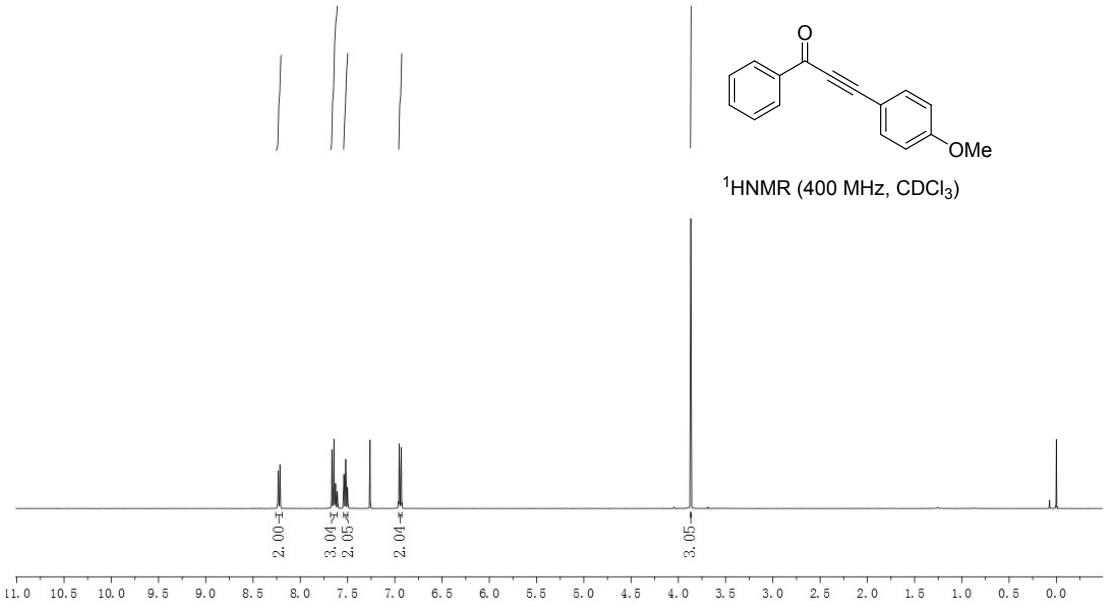
3ag


O
CF3
1HNMR (500 MHz, CDCl3)
2.00
2.04
3.07
2.05
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

3ag

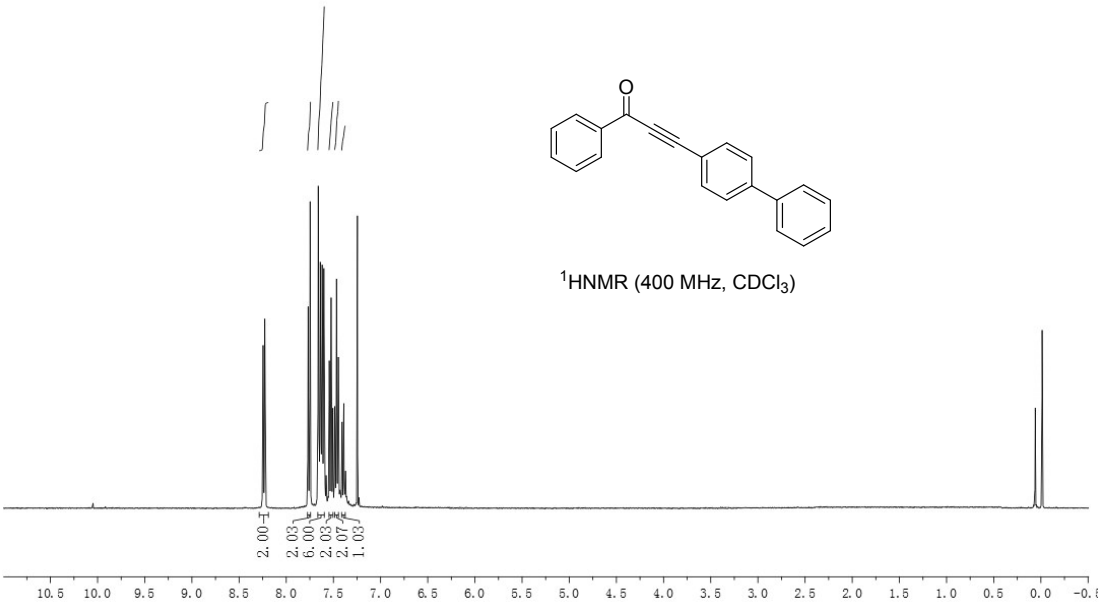

-63.13
O
CF3
19F NMR (500 MHz, CDCl3)
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

3ah



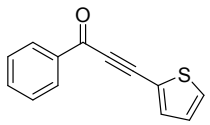
^{1}HNMR (400 MHz, $CDCl_3$)

3ai

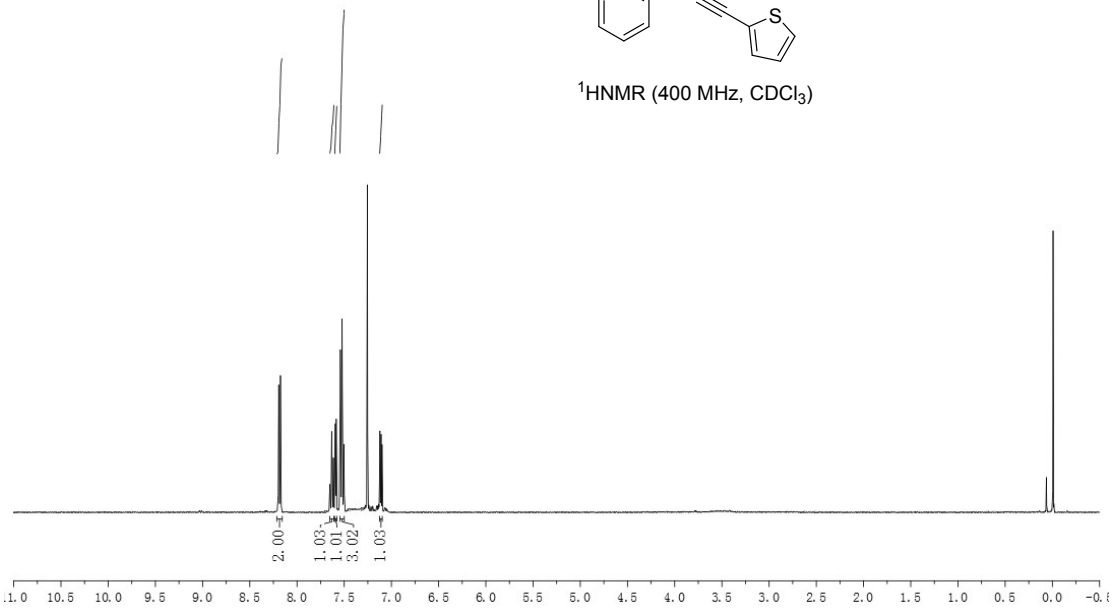


^{1}HNMR (400 MHz, $CDCl_3$)

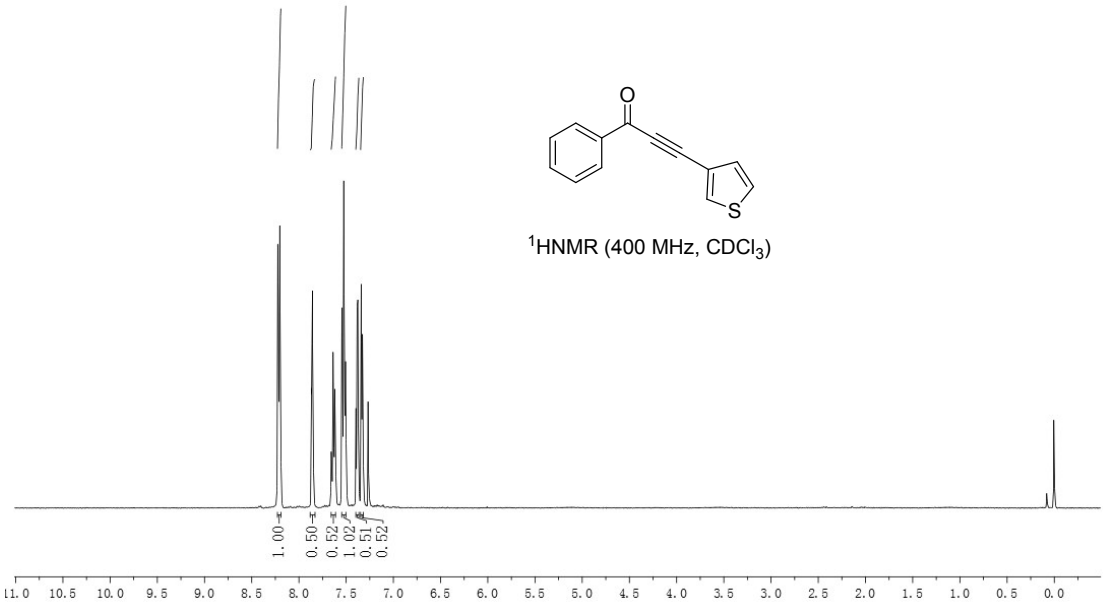
3aj



^{1}HNMR (400 MHz, $CDCl_3$)

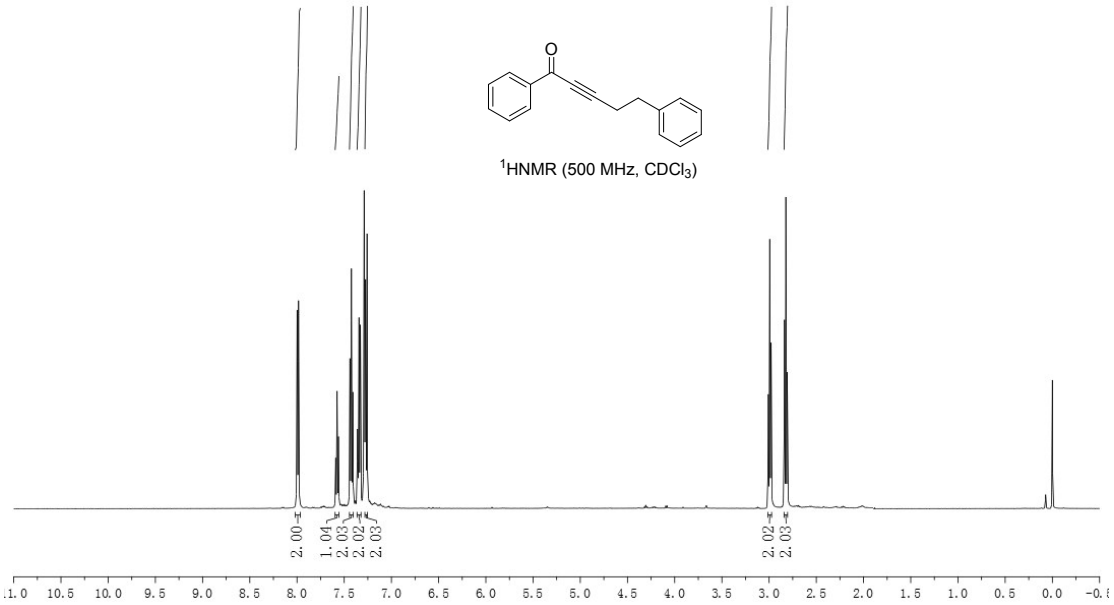


3ak

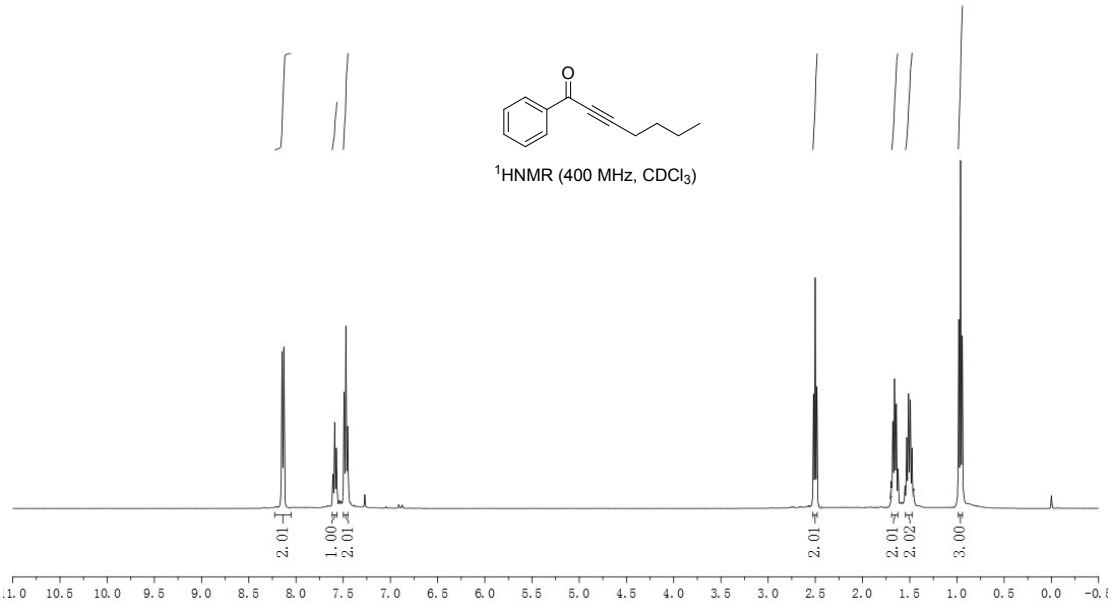


^{1}HNMR (400 MHz, $CDCl_3$)

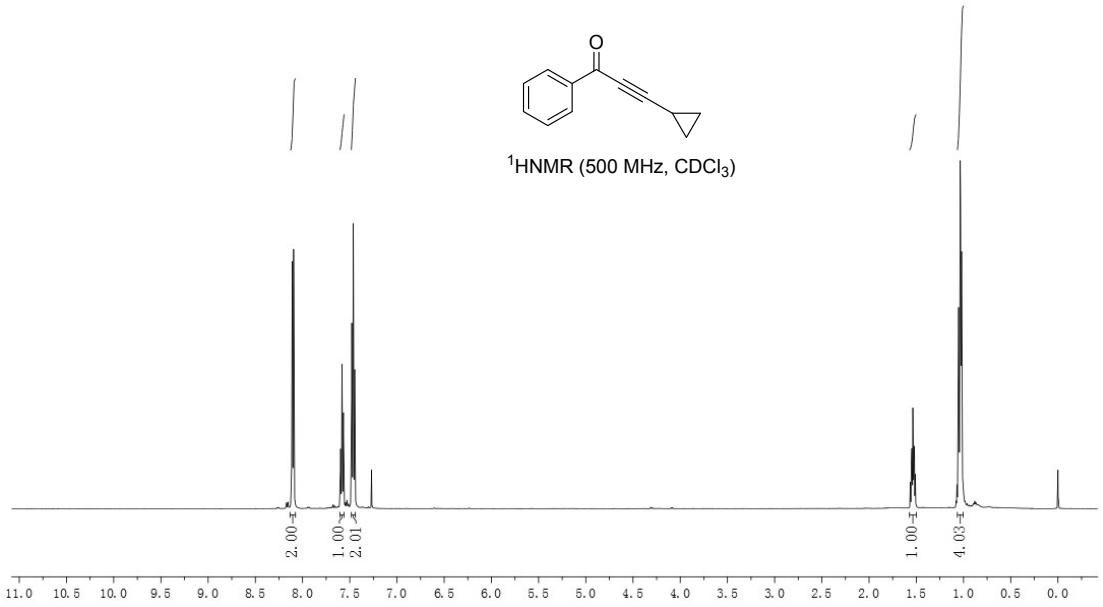
3al



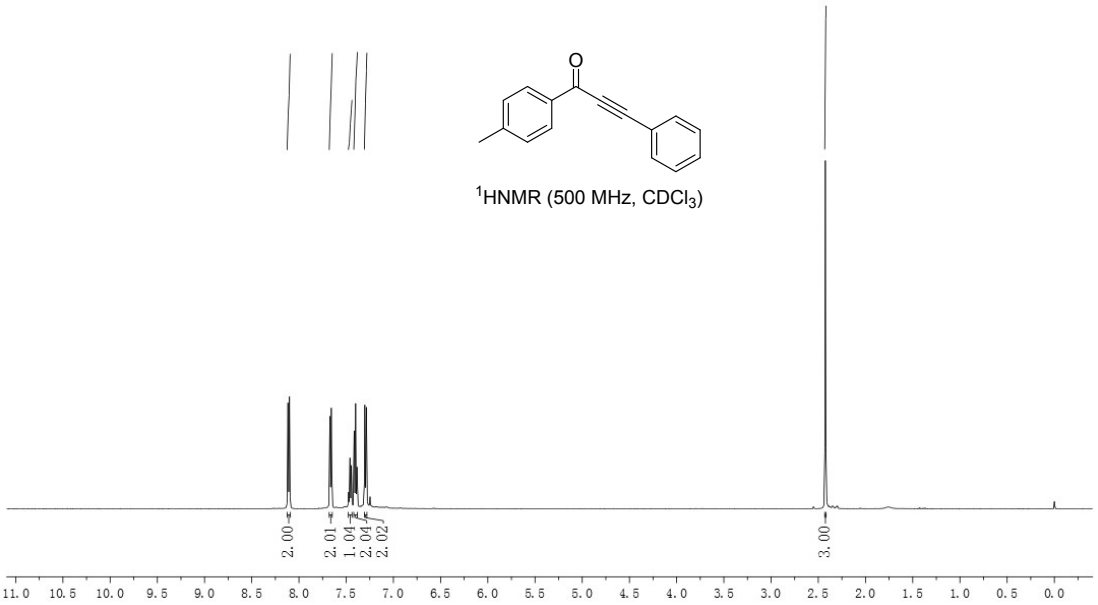
3am



3an

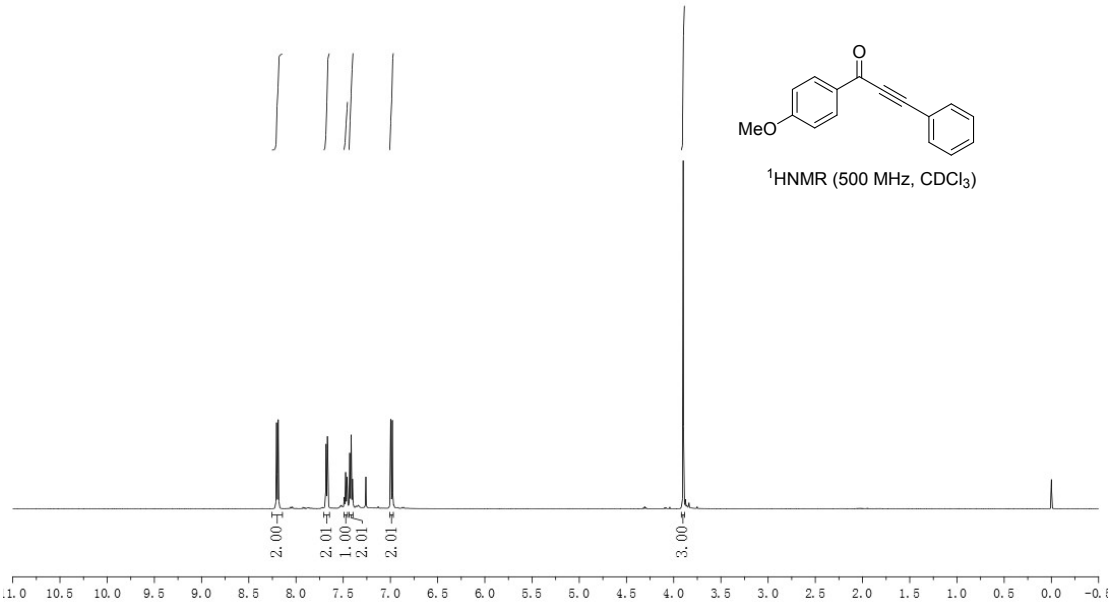

O
^{1}HNMR (500 MHz, $CDCl_3$)
2.00
1.00
2.01
1.00
4.03
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

3ba

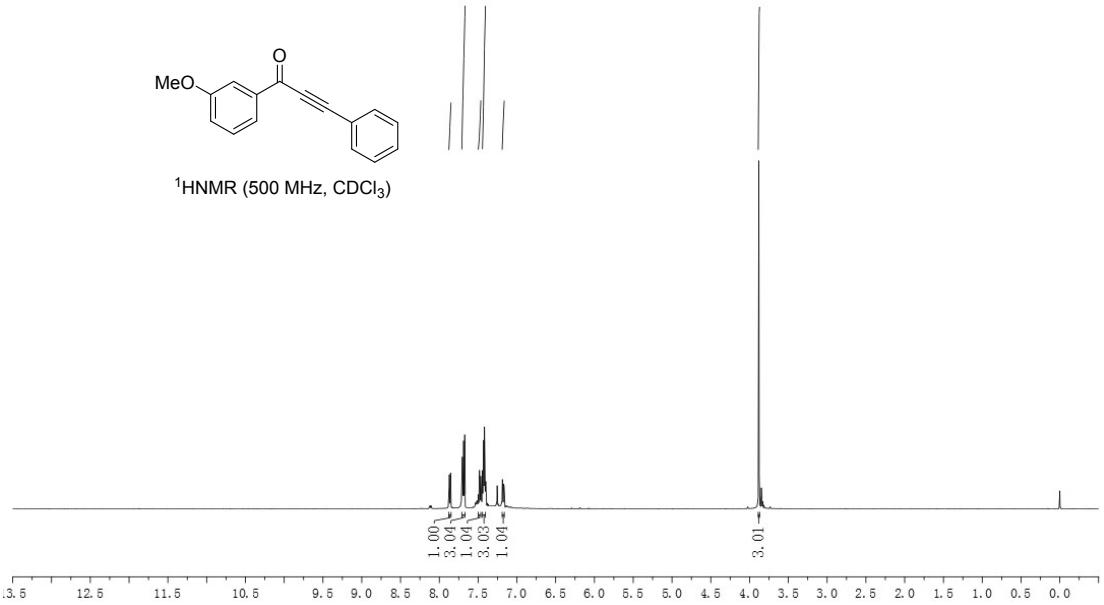


^{1}HNMR (500 MHz, $CDCl_3$)

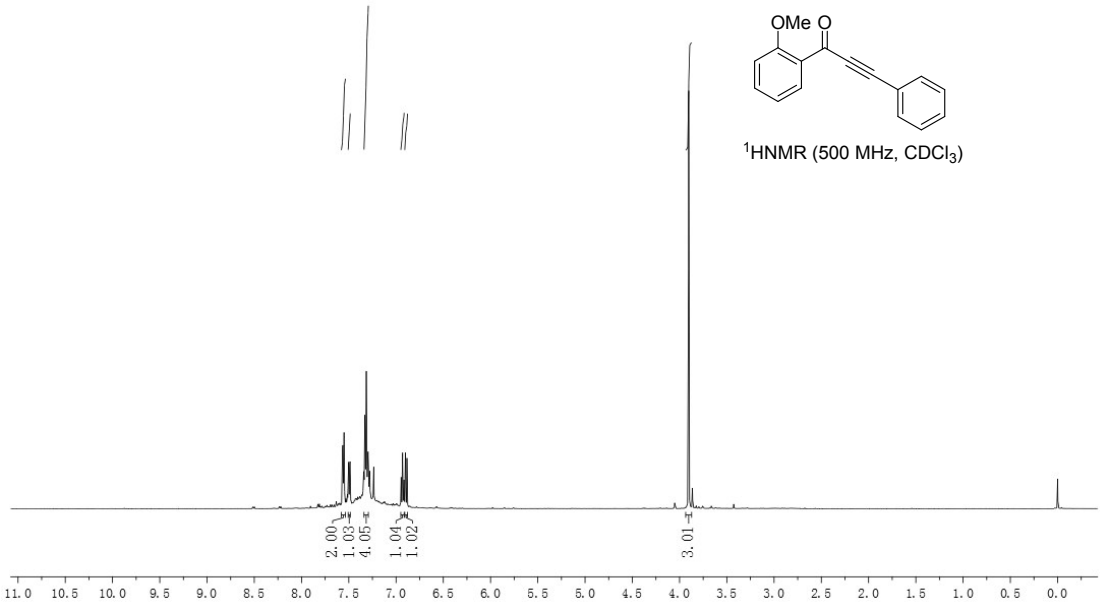
3ca


O
MeO
1HNMR (500 MHz, CDCl3)
2.00
2.01
1.00
2.01
2.01
3.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

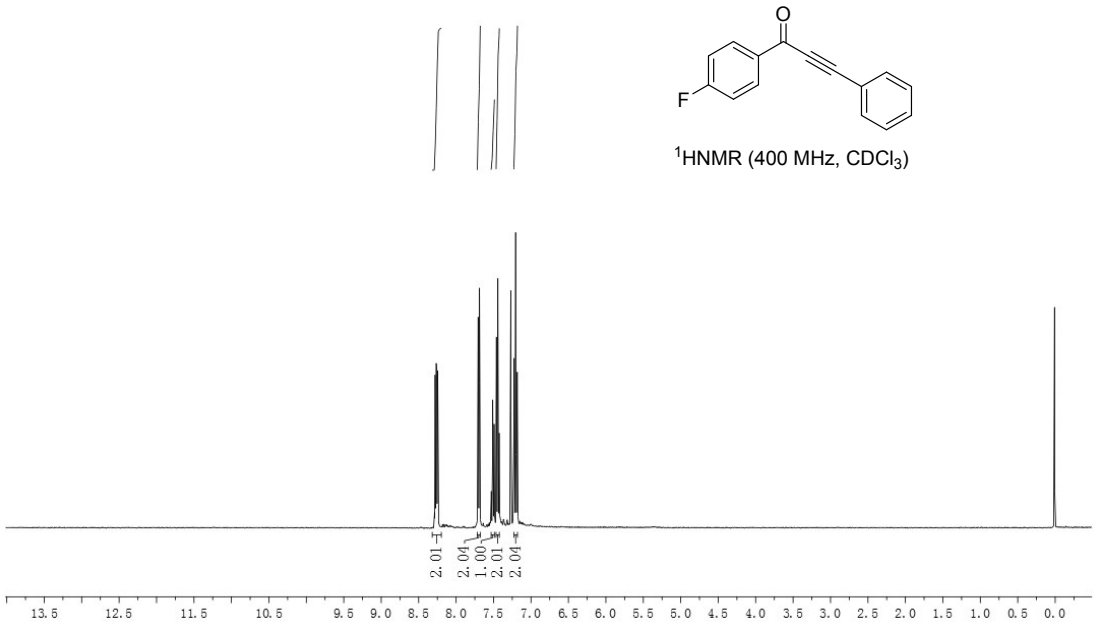
3da



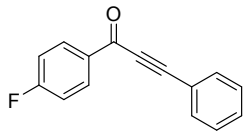
3ea


OMe O
^{1}HNMR (500 MHz, $CDCl_3$)
2.00
1.03
4.05
1.04
1.02
3.01
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

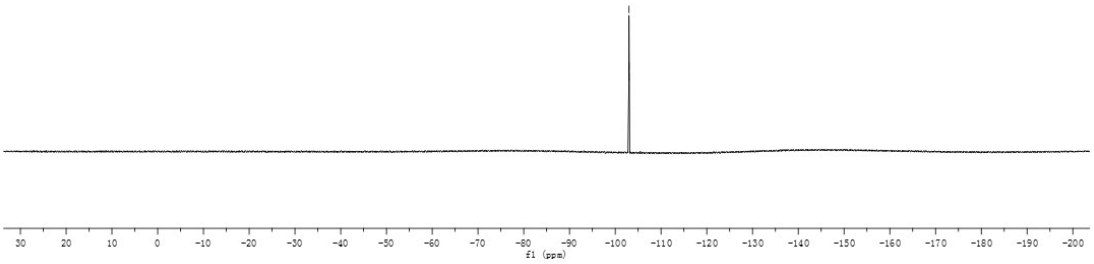
3fa


O
F
^{1}HNMR (400 MHz, $CDCl_3$)
2.01
2.04
1.00
2.01
2.04
13.5 12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

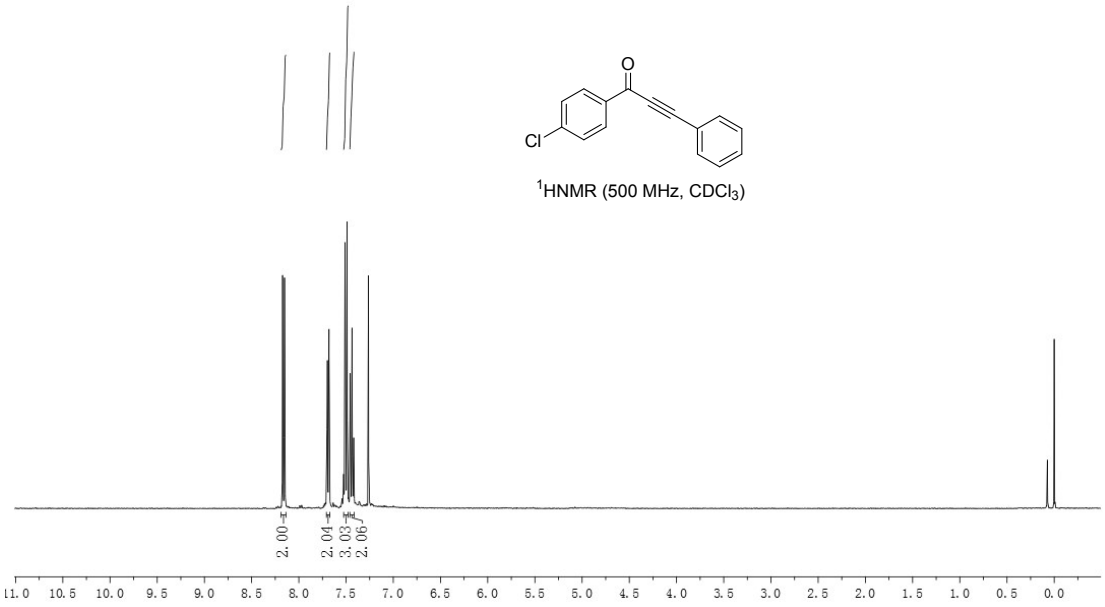
3fa



^{19}F NMR (400 MHz, $CDCl_3$)

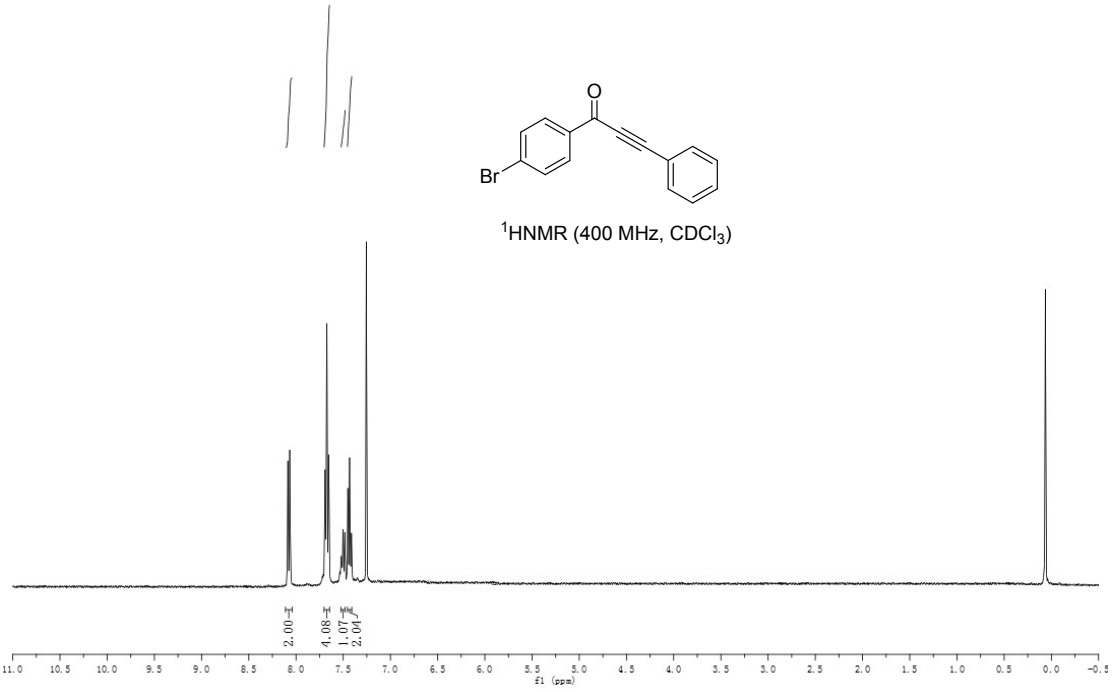


3ga



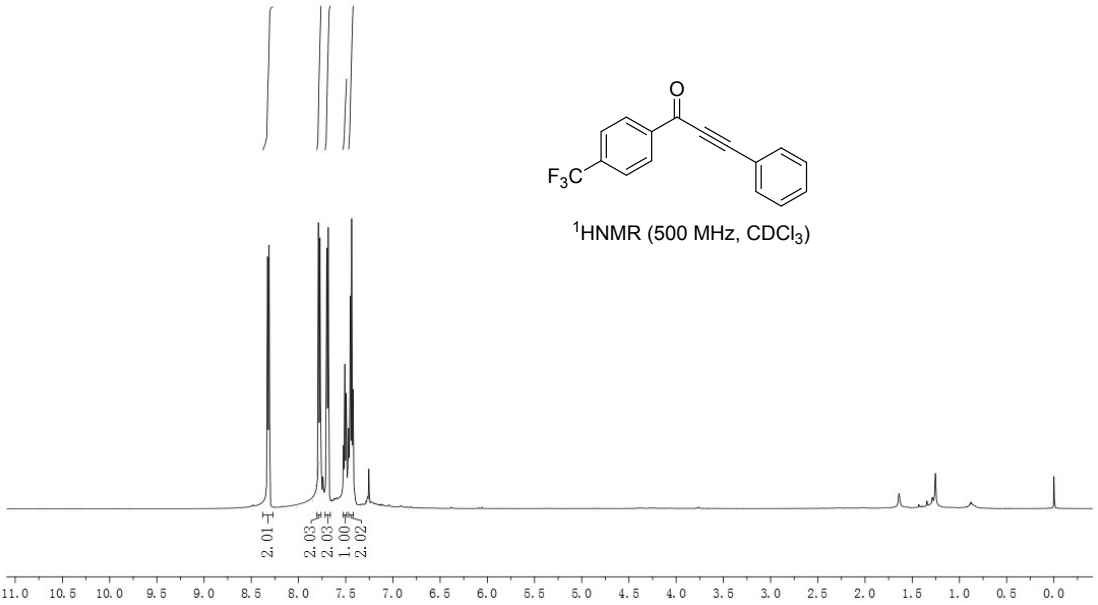
^{1}HNMR (500 MHz, $CDCl_3$)

3ha



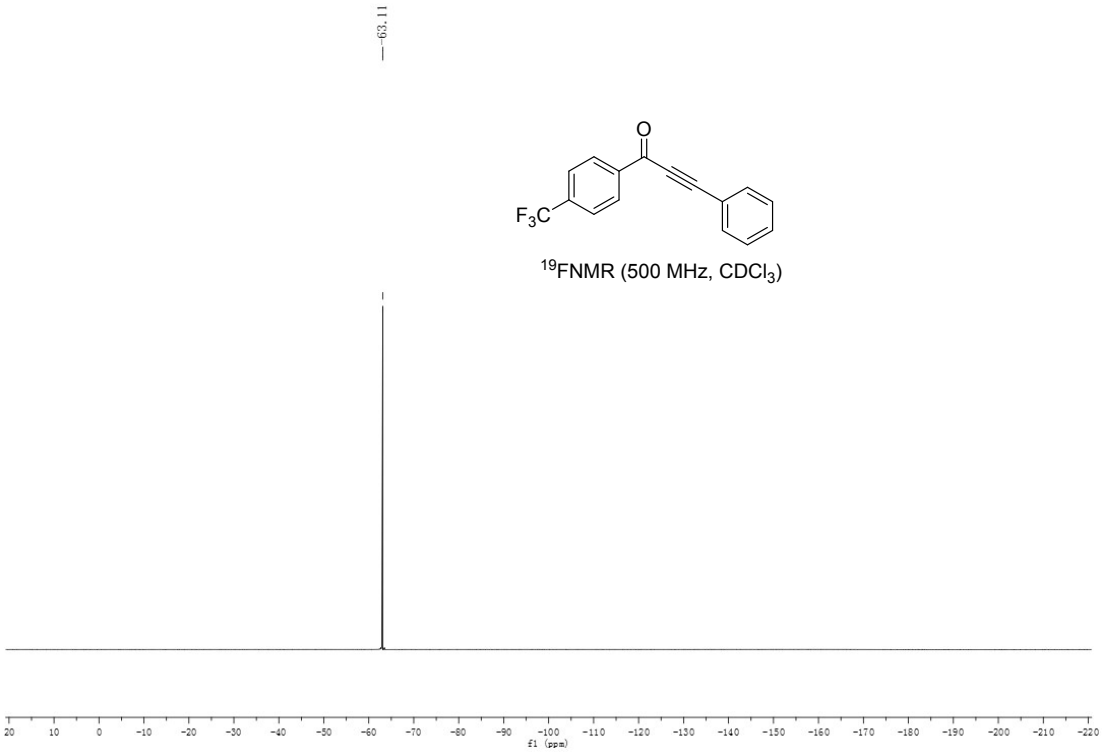
^{1}HNMR (400 MHz, $CDCl_3$)

3ia



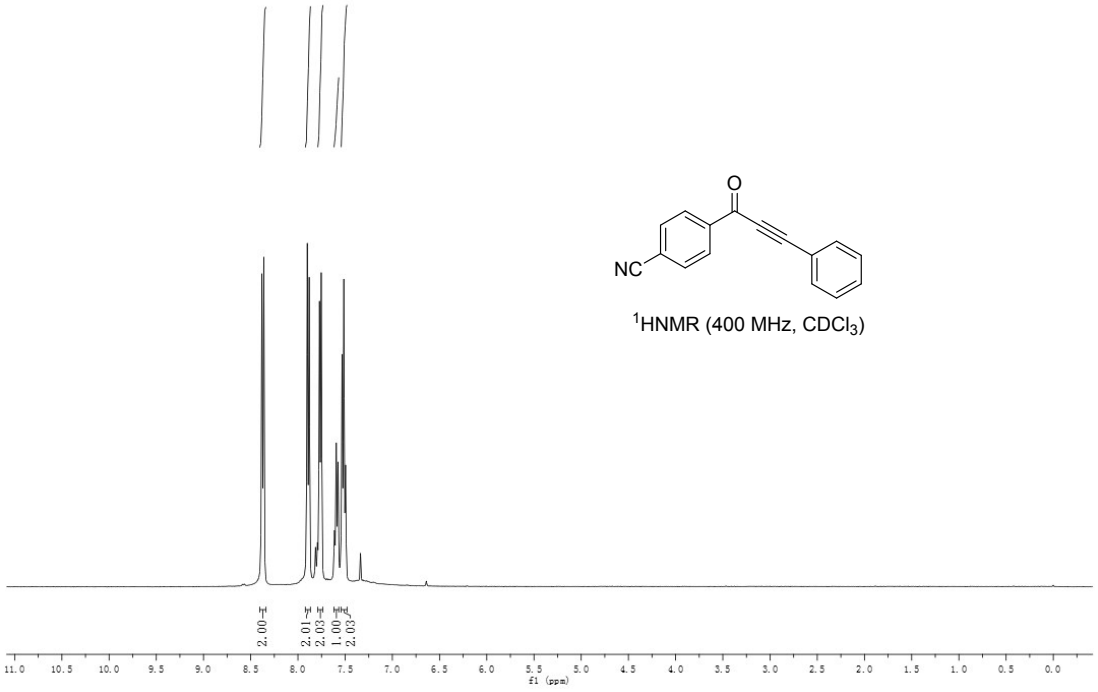
^{1}HNMR (500 MHz, $CDCl_3$)

3ia



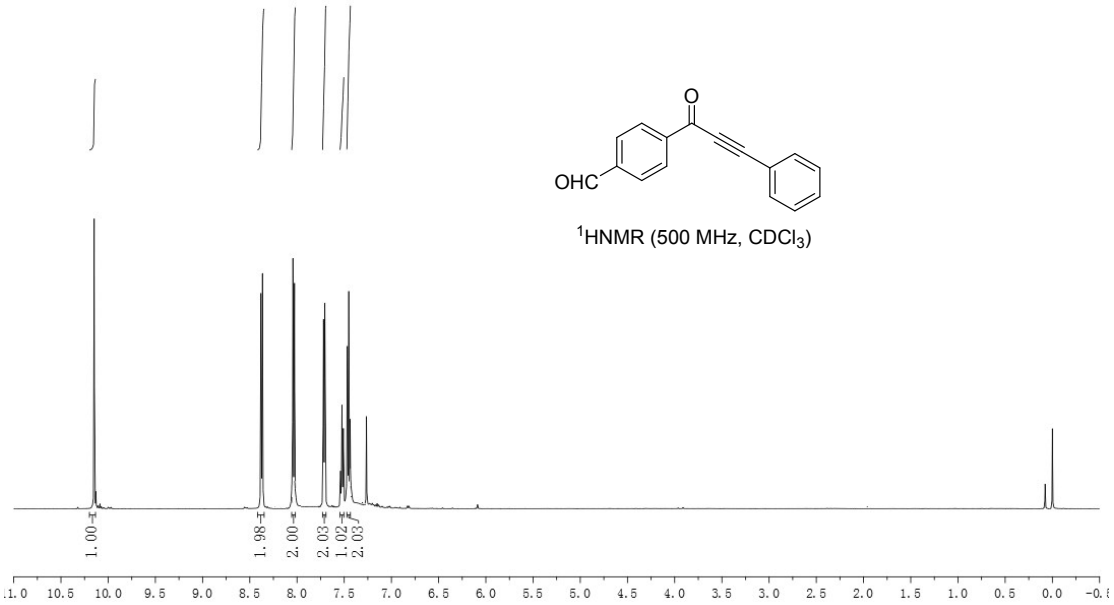
^{19}FNMR (500 MHz, $CDCl_3$)

3ja



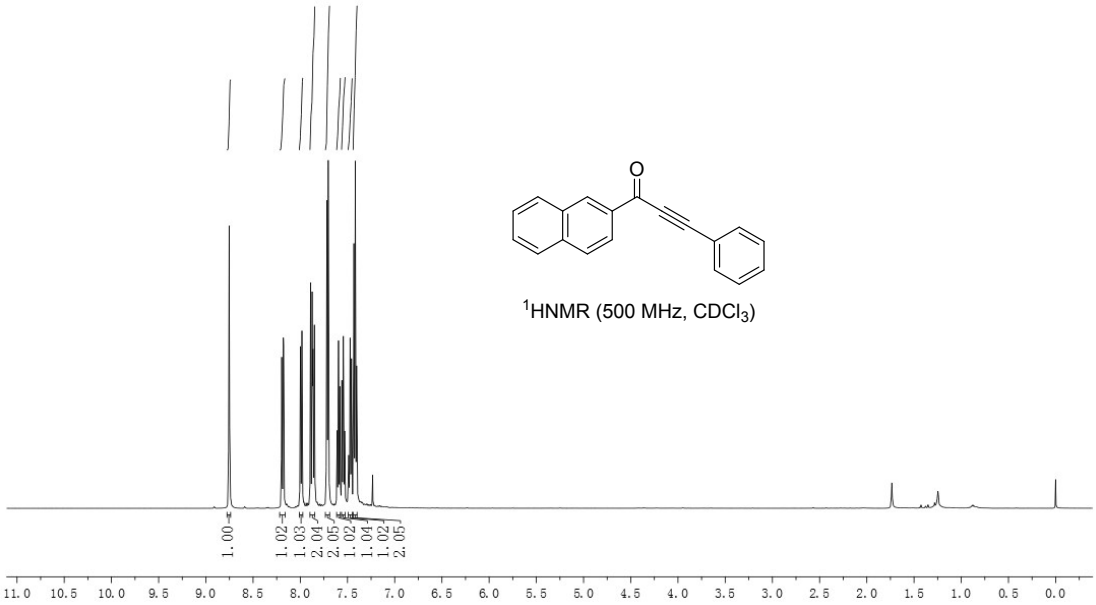
^{1}HNMR (400 MHz, $CDCl_3$)

3ka



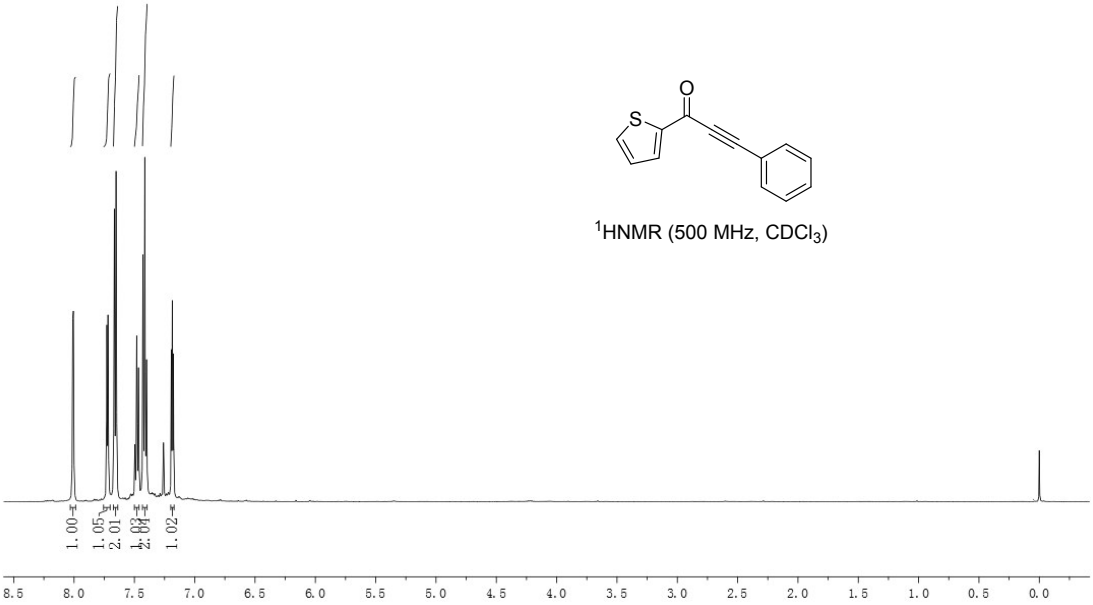
^{1}HNMR (500 MHz, $CDCl_3$)

3la



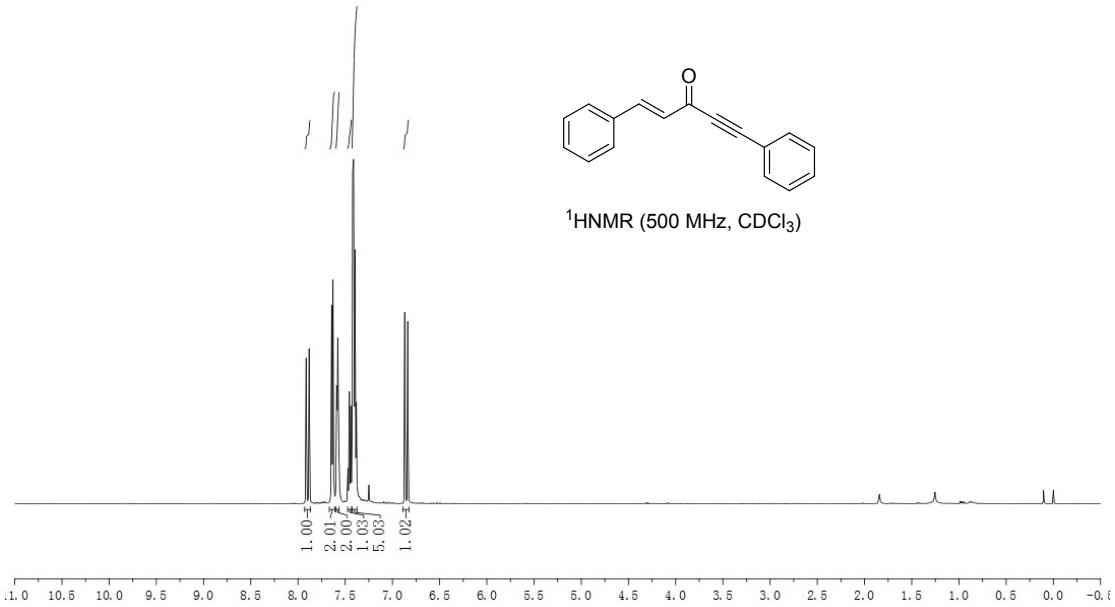
^{1}HNMR (500 MHz, $CDCl_3$)

3ma



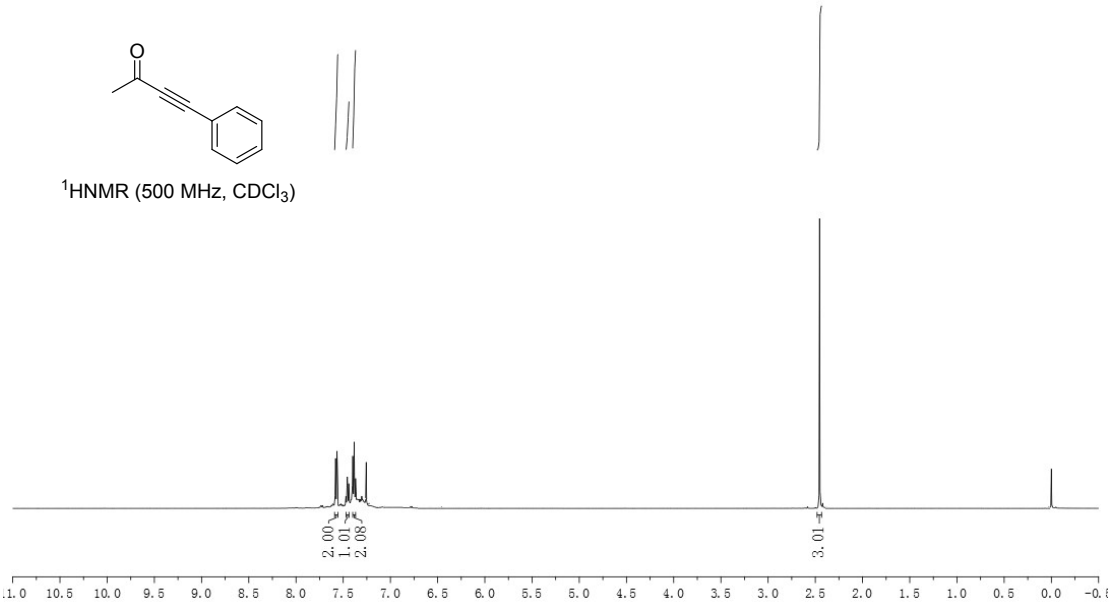
^{1}HNMR (500 MHz, $CDCl_3$)

3na



^{1}HNMR (500 MHz, $CDCl_3$)

3oa



^{1}HNMR (500 MHz, $CDCl_3$)

3pa