

Supporting information

Palladium Catalyzed Desulfurative Coupling of Allyl Sulfides with Organoboronic Acids

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I. General Information

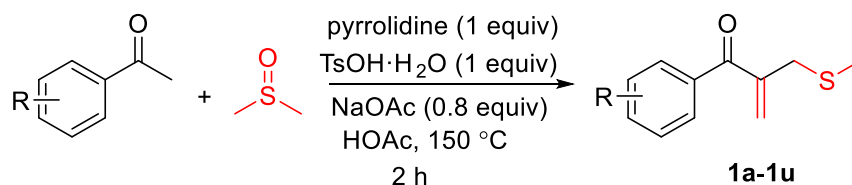
Unless otherwise noted, all chemicals were purchased from Energy Chemical, J & K Scientific, Adamas-beta and used without further purification. Column chromatography purifications were performed using 200–300 mesh silica gel. Commercial grade solvents and reagents were used without further purification. Experiments involving moisture and/or air sensitive components were performed in oven-dried glassware under a positive pressure of nitrogen using freshly distilled solvents. Analytical thin layer chromatography (TLC) was performed using Merck 60 F254 pre-coated silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualized using UV radiation (254 nm) on Spectroline Model ENF-24061/F 254 nm. Further visualization was possible by staining with basic solution of potassium permanganate or acidic solution of ceric molybdate. Flash chromatography was performed using 200-300 mesh silica gel with the indicated solvent system. Columns were typically packed as slurry and equilibrated with the appropriate solvent system prior to use. High resolution mass spectral analysis (HRMS) was performed on an Thermo Scientific Q Exactive Instruments and was identified by Q Exactive-Orbitrap MS with an electrospray ionization (ESI) source. The melting point were collected on a WRS-3 melting point apparatus from Shanghai INESA Physico-Optical Instrument Co.,Ltd.. ¹H

NMR and ^{13}C NMR experiments were performed with a BRUKER AVANCE III HD 600 MHz and 151 MHz NMR spectrometer, respectively (Bruker, Billerica, MA). Chemical shifts for ^1H NMR spectra are reported as δ in units of parts per million (ppm) downfield from SiMe_4 (δ 0.0) and relative to the signal of chloroform-*d* (δ 7.2600, singlet). Multiplicities were given as: s (singlet); d (doublet); t (triplet); q (quartet); dd (doublets of doublet); dt (doublets of triplet); or m (multiplets). The number of protons (n) for a given resonance is indicated by nH. Coupling constants are reported as a *J* value in Hz. Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as δ in units of parts per million (ppm) downfield from SiMe_4 (δ 0.0) and relative to the signal of chloroform-*d* (δ 77.0, triplet).

II. Experimental Sections

2.1 Preparation of allylic sulfides

The allylic sulfides **1a** – **1u**, **1a-d**₇ were prepared according to our previously reported synthetic procedure¹.

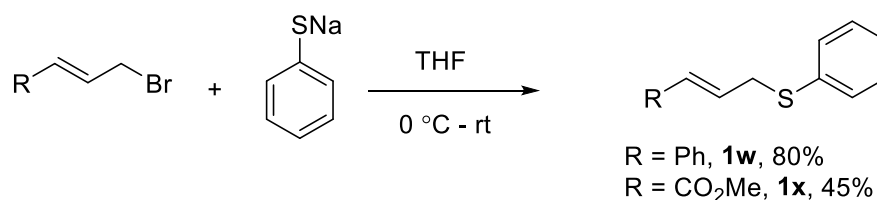


A 10 mL reaction tube equipped with a magnetic stirring bar was added with substitute acetophenone (1 mmol, 1.0 equiv), DMSO (1 mL),

TsOH • H₂O (1 mmol, 1.0 equiv), pyrrolidine (1 mmol, 1.0 equiv), NaOAc (0.8 mmol, 0.8 equiv) in 4 mL HOAc. The tube was stirred at 150 °C for 2-8 h monitored by TLC until the starting material was completely consumed. The reaction mixture was cooled to room temperature, followed by the addition of water and ethyl acetate. The organic layer was separated and the aqueous layer was extracted with ethyl acetate (3×20 mL). The combined organic layer was washed with brine and dried over anhydrous Na₂SO₄, filtrated and concentrated *in vacuo*, the residue was purified through column chromatography on silica gel to give the desired products.

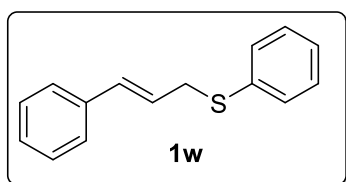
In addition, the compounds **4** and **6** were also prepared from the reported literature.¹

Sulfides **1v** are commercially available from Energy Chemical. Sulfides **1w**² and **1x**³ was synthesized according the reported methods⁴.

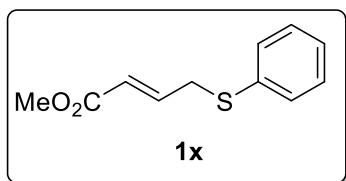


To a mixture of a flame-dried flask equipped with a stir bar, PhSNa (0.66 g, 5 mol) and THF (10 mL), substituted allylbromide (4 mmol) was added dropwise while stirring at 0 °C under water-ice bath. After addition, the mixture was warmed to room temperature and then stirred for 5 h. The

reaction was quenched by addition of 30 mL saturated NH_4Cl solution, then extracted with ethyl acetate (30 mL \times 2), the extracted organic mixture was dried over Na_2SO_4 and concentrated under vacuum. The crude product was purified by silica gel column chromatography (hexanes) to afford the corresponding product.



This compound was prepared by the general procedure described above and was obtained as colorless oil (0.72 g, 80 %). Rf (10:1 PE/EtOAc) 0.7; ^1H NMR (600 MHz, CDCl_3) δ 7.39 (d, $J = 7.9$ Hz, 2H), 7.32 – 7.27(m, 6H), 7.23 – 7.19(m, 2H), 6.43 (d, $J = 15.7$ Hz, 1H), 6.28 – 6.23 (m, 1H), 3.72 (d, $J = 7.1$ Hz, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 136.72, 135.80, 132.76, 130.26, 128.83, 128.50, 127.55, 126.40, 126.31, 125.04, 37.12.



This compound was prepared by the general procedure described above and was obtained as colorless oil (375 mg, 45%). Rf (PE:EtOAc = 10:1): 0.4; ^1H NMR (600 MHz, CDCl_3) δ 7.32 (d, $J = 6.9$ Hz, 2H), 7.27 (t, $J = 6.8$ Hz, 2H), 7.21 – 7.20 (m, 1H), 6.95 – 6.91 (m, 1H), 5.80 (d, $J = 15.4$ Hz, 1H), 3.69 (s, 3H), 3.60 (d, $J = 6.4$ Hz, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 166.3, 143.1, 134.5, 130.5, 129.0, 126.9, 122.8, 51.5, 35.6.

2.2 General procedure for the palladium-catalyzed cross-coupling reaction of allylic sulfides with organoboronic acids



A 10 mL reaction tube equipped with a magnetic stirring bar was added allylic sulfides (0.1 mmol), PdCl₂ (0.01 mmol, 10 mol%), Ag₂CO₃ (0.2 mmol, 2 equiv), organoboronic acids (0.25 mmol, 2.5 equiv), in DMA (1 mL) at 90 °C in oil bath for 4 h. The reaction mixture was cooled to room temperature, followed by the addition of water and ethyl acetate. The organic layer was separated and the aqueous layer was extracted with ethyl acetate (3 × 10 mL). The combined organic layer was washed with brine and dried over anhydrous Na₂SO₄, filtrated and concentrated in *vacuo*, the residue was purified through column chromatography on silica gel to give the desired products.

Detailed procedure for the synthesis of **3a** on a 1 mmol scale

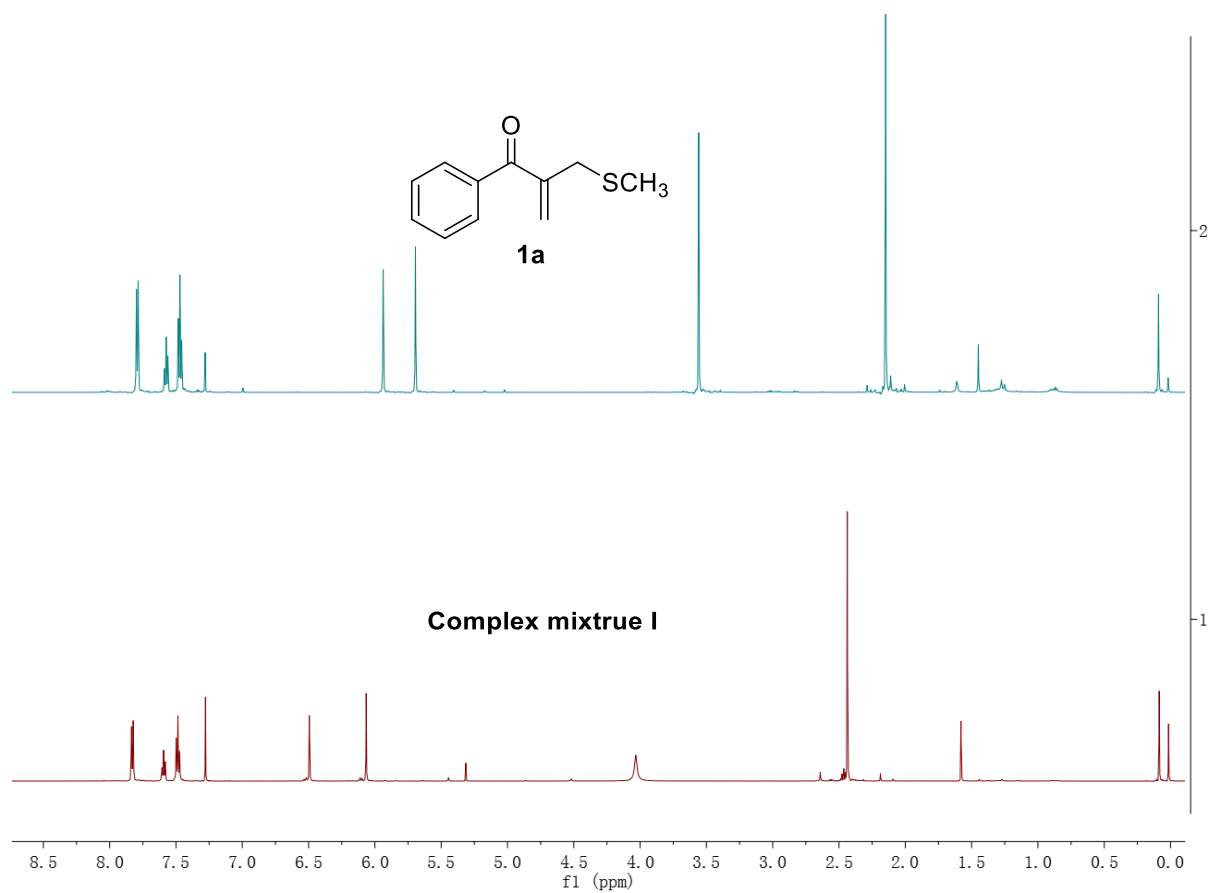
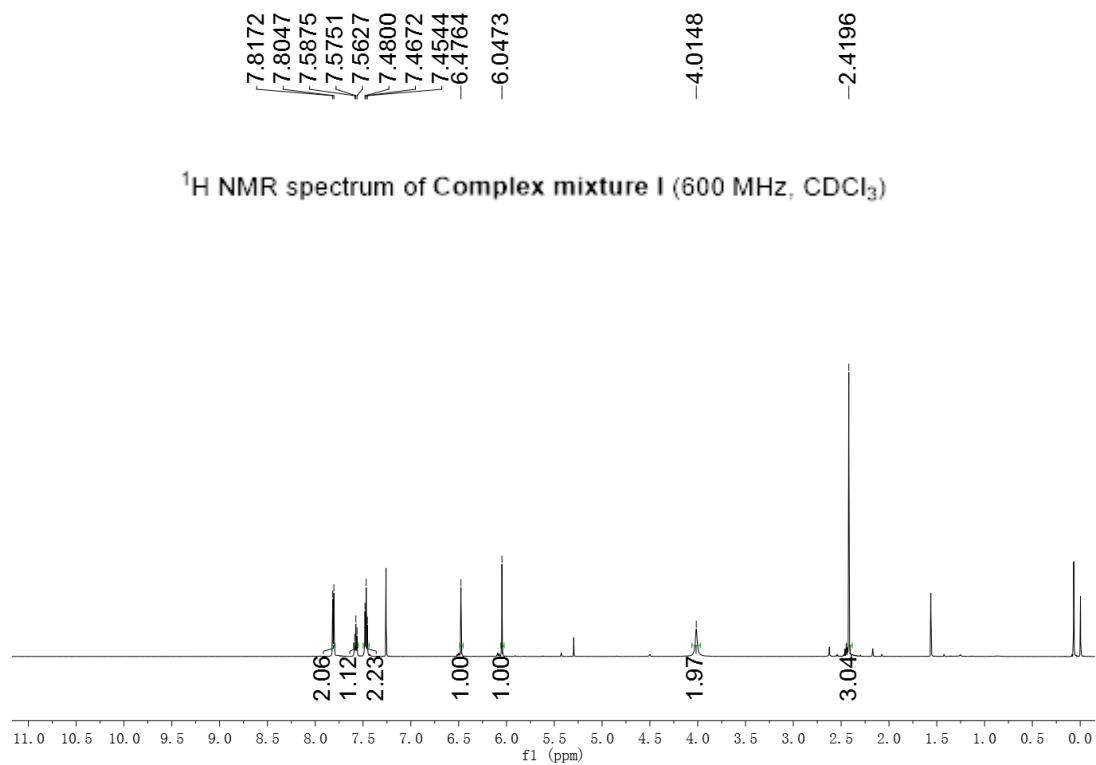
A 10 mL reaction tube equipped with a magnetic stirring bar was added allylic sulfide **1a** (1.0 mmol), PdCl₂ (0.1 mmol, 10 mol%), Ag₂CO₃ (2 mmol, 2 equiv) and phenyl boronic acid (2.5 mmol, 2.5 equiv), in DMA (4 mL) at 90 °C in oil bath for 4 h. The reaction mixture was cooled to room temperature, followed by the addition of water and ethyl acetate. The organic layer was separated and the aqueous layer was extracted with ethyl acetate (3 × 30 mL). The combined organic layer was washed with

brine and dried over anhydrous Na_2SO_4 , filtrated and concentrated in *vacuo*, the residue was purified through column chromatography on silica gel to give the desired products **3a** in 74% (0.164 g) yields.

2.3 Instant monitoring of the reaction of **1a** with stoichiometric PdCl_2 in CDCl_3



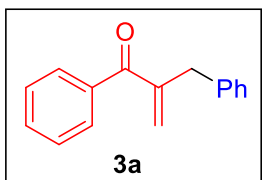
A 10 mL reaction tube equipped with a magnetic stirring bar was added allylic sulfides (0.1 mmol), PdCl_2 (0.1 mmol, 1equiv), Ag_2CO_3 (0.05 mmol, 0.5 equiv) in CDCl_3 (0.5 mL) at $90\text{ }^\circ\text{C}$ for 1 h, the reaction mixture (**Complex mixture I**) was measured directly by ^1H NMR experiment. After that, organoboronic acids (0.2 mmol, 2 equiv) and DMA (1 mL) were added to the reaction mixture stirred at $90\text{ }^\circ\text{C}$ for 1 h. The reaction mixture was cooled to room temperature, followed by the addition of water and ethyl acetate. The organic layer was separated and the aqueous layer was extracted with ethyl acetate ($3 \times 10\text{ mL}$). The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4 , filtrated and concentrated in *vacuo*, the residue was purified through column chromatography on silica gel to give the desired products **3a** in 34% yield.



¹H NMR spectrum between **1a** and **Complex mixture I**

2.4 Characterization data for the products

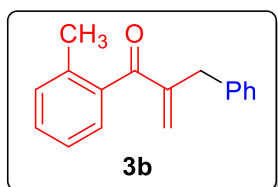
2-benzyl-1-phenylprop-2-en-1-one (3a)⁵



This compound was prepared by the general procedure described above and was obtained as yellow oil (17.8 mg, 80%). Rf (PE:EtOAc = 10:1): 0.61; ¹H NMR (600

MHz, CDCl₃) δ 7.72 (d, *J* = 7.4 Hz, 2H), 7.52 (t, *J* = 7.4 Hz, 1H), 7.42 (t, *J* = 7.7 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.27 (d, *J* = 6.3 Hz, 2H), 7.22 (t, *J* = 7.2 Hz, 1H), 5.76 (s, 1H), 5.69 (s, 1H), 3.81 (s, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 197.6, 147.6, 138.7, 137.7, 132.2, 129.5, 129.2, 128.5, 128.1, 126.9, 126.3, 38.3; HRMS (ESI) *m/z* calculated for C₁₆H₁₅O [M+H]⁺: 223.1117, found 223.1116.

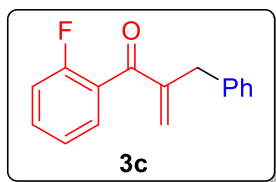
2-benzyl-1-(*o*-tolyl)prop-2-en-1-one (3b)



This compound was prepared by the general procedure described above and was obtained as yellow oil (17.2 mg, 73%). Rf (PE:EtOAc = 10:1):

0.65; ¹H NMR (600 MHz, CDCl₃) δ 7.33 (dt, *J* = 7.2, 3.4 Hz, 3H), 7.28 (d, *J* = 6.8 Hz, 2H), 7.26 – 7.18 (m, 4H), 5.82 (s, 1H), 5.69 (s, 1H), 3.81 (s, 2H), 2.28 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 199.8, 149.1, 138.9, 138.7, 136.3, 130.7, 130.0, 129.8, 129.2, 128.5, 128.0, 126.3, 125.0, 37.0, 19.6; HRMS (ESI) *m/z* calculated for C₁₇H₁₇O [M+H]⁺: 237.1274, found 237.1272.

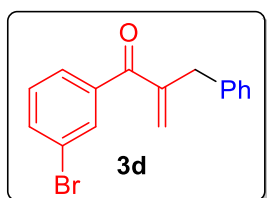
2-benzyl-1-(2-fluorophenyl)prop-2-en-1-one (3c)



This compound was prepared by the general procedure described above and was obtained as yellow oil (18.2 mg, 76%). Rf (PE:EtOAc = 10:1):

0.58; ^1H NMR (600 MHz, CDCl_3) δ 7.45 (dd, $J = 13.5, 7.3$ Hz, 1H), 7.40 (t, $J = 7.1$ Hz, 1H), 7.33 (t, $J = 7.5$ Hz, 2H), 7.27 – 7.22 (m, 3H), 7.19 (t, $J = 7.5$ Hz, 1H), 7.11 (t, $J = 9.1$ Hz, 1H), 5.82 (s, 1H), 5.80 (s, 1H), 3.80 (s, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 194.6, 159.8 (d, $J = 251.7$ Hz), 148.6, 138.6, 132.5 (d, $J = 8.4$ Hz), 130.2 (d, $J = 3.3$ Hz), 129.8 (d, $J = 2.2$ Hz), 129.2, 128.4, 127.2 (d, $J = 15.2$ Hz), 126.3, 124.0 (d, $J = 3.9$ Hz), 116.1 (d, $J = 21.8$ Hz), 36.9; HRMS (ESI) m/z calculated for $\text{C}_{16}\text{H}_{14}\text{FO}$ $[\text{M}+\text{H}]^+$: 241.1023, found 241.1020.

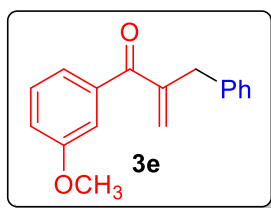
2-benzyl-1-(3-bromophenyl)prop-2-en-1-one (3d)



This compound was prepared by the general procedure described above and was obtained as yellow oil (25.0 mg, 83%). Rf (PE:EtOAc = 10:1):

0.57; ^1H NMR (600 MHz, CDCl_3) δ 7.81 (t, $J = 1.7$ Hz, 1H), 7.63 – 7.59 (m, 2H), 7.30 – 7.27 (m 3H), 7.24 – 7.19 (m, 3H), 5.78 (s, 1H), 5.68 (s, 1H), 3.77 (s, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 196.0, 147.3, 139.5, 138.4, 135.0, 132.2, 129.8, 129.1, 128.6, 128.0, 127.7, 126.5, 122.4, 38.1; HRMS (ESI) m/z calculated for $\text{C}_{16}\text{H}_{14}\text{BrO}$ $[\text{M}+\text{H}]^+$: 301.0223, found 301.0220.

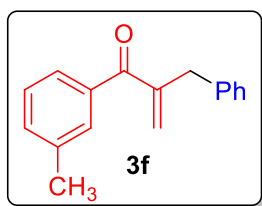
2-benzyl-1-(3-methoxyphenyl)prop-2-en-1-one (3e)



This compound was prepared by the general procedure described above and was obtained as yellow oil (13.0 mg, 52%). Rf (PE:EtOAc = 10:1):

0.49; $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.31 (dd, $J = 15.8, 7.8$ Hz, 4H), 7.26 – 7.25 (m, 3H), 7.21 (t, $J = 7.2$ Hz, 1H), 7.07 (dd, $J = 8.0, 1.5$ Hz, 1H), 5.75 (s, 1H), 5.70 (s, 1H), 3.83 (s, 3H), 3.80 (s, 2H); $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 197.4, 159.4, 147.6, 139.0, 138.6, 129.1, 129.1, 128.5, 126.8, 126.3, 122.2, 118.6, 113.9, 55.4, 38.4; HRMS (ESI) m/z calculated for $\text{C}_{17}\text{H}_{17}\text{O}_2$ $[\text{M}+\text{H}]^+$: 253.1223, found 253.1221.

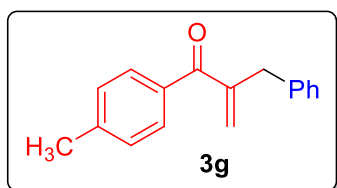
2-benzyl-1-(*m*-tolyl)prop-2-en-1-one (3f)⁶



This compound was prepared by the general procedure described above and was obtained as yellow oil (18.7 mg, 79%). Rf (PE:EtOAc = 10:1):

0.58; $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.53 (s, 1H), 7.49 (d, $J = 7.5$ Hz, 1H), 7.30 (dq, $J = 14.8, 7.4$ Hz, 4H), 7.26 – 7.25 (m, 2H), 7.21 (t, $J = 7.2$ Hz, 1H), 5.73 (s, 1H), 5.68 (s, 1H), 3.79 (s, 2H), 2.38 (s, 3H); $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 197.9, 147.7, 138.7, 138.0, 137.7, 133.0, 129.9, 129.2, 128.5, 128.0, 126.9, 126.8, 126.3, 38.3, 21.3; HRMS (ESI) m/z calculated for $\text{C}_{17}\text{H}_{17}\text{O}$ $[\text{M}+\text{H}]^+$: 237.1274, found 237.1272.

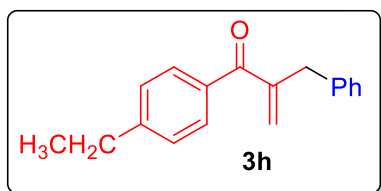
2-benzyl-1-(*p*-tolyl)prop-2-en-1-one (3g)



This compound was prepared by the general procedure described above and was obtained as

yellow oil (18.2 mg, 77%). Rf (PE:EtOAc = 10:1): 0.59; ^1H NMR (600 MHz, CDCl_3) δ 7.65 (d, $J = 8.0$ Hz, 2H), 7.30 (t, $J = 7.5$ Hz, 2H), 7.25 (d, $J = 7.6$ Hz, 2H), 7.21 (t, $J = 8.4$ Hz, 3H), 5.71 (s, 1H), 5.65 (s, 1H), 3.80 (s, 2H), 2.40 (s, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 197.4, 147.7, 143.0, 138.7, 134.9, 129.7, 129.2, 128.9, 128.5, 126.3, 126.0, 38.5, 21.6; HRMS (ESI) m/z calculated for $\text{C}_{17}\text{H}_{17}\text{O}$ $[\text{M}+\text{H}]^+$: 237.1274, found 237.1272.

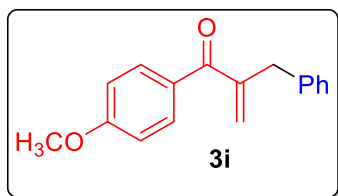
2-benzyl-1-(4-ethylphenyl)prop-2-en-1-one (3h)



This compound was prepared by the general procedure described above and was obtained as yellow oil (21.1 mg, 84%). Rf (PE:EtOAc

= 10:1): 0.63; ^1H NMR (600 MHz, CDCl_3) δ 7.67 (d, $J = 8.0$ Hz, 2H), 7.29 (t, $J = 7.5$ Hz, 2H), 7.26 – 7.23 (m, 4H), 7.20 (t, $J = 7.2$ Hz, 1H), 5.71 (s, 1H), 5.66 (s, 1H), 3.80 (s, 2H), 2.69 (q, $J = 7.6$ Hz, 2H), 1.25 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 197.4, 149.2, 147.7, 138.7, 135.1, 129.8, 129.2, 128.5, 127.7, 126.3, 126.1, 38.5, 28.9, 15.2; HRMS (ESI) m/z calculated for $\text{C}_{18}\text{H}_{19}\text{O}$ $[\text{M}+\text{H}]^+$: 251.1430, found 251.1429.

2-benzyl-1-(4-methoxyphenyl)prop-2-en-1-one (3i)

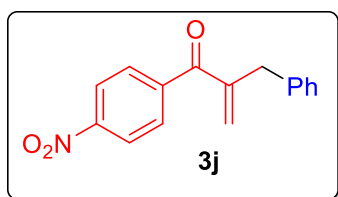


This compound was prepared by the general procedure described above and was obtained as colorless oil (14.9 mg, 59%). Rf (PE:EtOAc =

10:1): 0.38; ^1H NMR (600 MHz, CDCl_3) δ 7.78 (d, $J = 8.6$ Hz, 2H), 7.31 (t, $J = 7.5$ Hz, 2H), 7.27 (t, $J = 5.6$ Hz, 2H), 7.22 (t, $J = 7.2$ Hz, 1H), 6.92

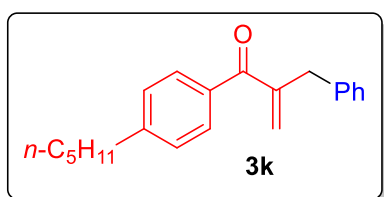
(d, $J = 8.7$ Hz, 2H), 5.68 (s, 1H), 5.62 (s, 1H), 3.87 (s, 3H), 3.81 (s, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 196.4, 163.1, 147.7, 138.7, 131.9, 130.1, 129.1, 128.5, 126.3, 124.7, 113.4, 55.4, 38.8; HRMS (ESI) m/z calculated for $\text{C}_{17}\text{H}_{17}\text{O}_2$ $[\text{M}+\text{H}]^+$: 253.1223, found 253.1222.

2-benzyl-1-(4-nitrophenyl)prop-2-en-1-one (3j)



This compound was prepared by the general procedure described above and was obtained as yellow oil (17.5 mg, 65%). Rf (PE:EtOAc = 10:1): 0.37; ^1H NMR (600 MHz, CDCl_3) δ 8.27 (d, $J = 8.5$ Hz, 2H), 7.82 (d, $J = 8.5$ Hz, 2H), 7.32 (t, $J = 7.5$ Hz, 2H), 7.27 – 7.22 (m, 3H), 5.90 (s, 1H), 5.70 (s, 1H), 3.81 (s, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 195.7, 149.7, 147.4, 143.1, 138.1, 130.1, 129.1, 128.9, 128.7, 126.6, 123.4, 37.9; HRMS (ESI) m/z calculated for $\text{C}_{16}\text{H}_{13}\text{NO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$: 289.0709, found 289.0716.

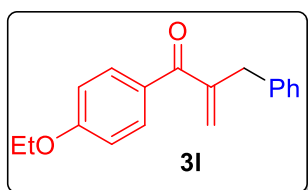
2-benzyl-1-(4-pentylphenyl)prop-2-en-1-one (3k)



This compound was prepared by the general procedure described above and was obtained as yellow oil (20.4 mg, 70%). Rf (PE:EtOAc = 10:1): 0.70; ^1H NMR (600 MHz, CDCl_3) δ 7.66 (d, $J = 8.1$ Hz, 2H), 7.29 (t, $J = 7.5$ Hz, 2H), 7.26 – 7.25 (m, 2H), 7.22 – 7.19 (m, 3H), 5.71 (s, 1H), 5.66 (s, 1H), 3.80 (s, 2H), 2.64 (t, $J = 7.7$ Hz, 2H), 1.65 – 1.60 (m, 2H), 1.37 – 1.30 (m, 4H), 0.89 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (151 MHz,

CDCl₃) δ 197.4, 148.0, 147.7, 138.7, 135.1, 129.7, 129.2, 128.5, 128.2, 126.3, 126.1, 38.5, 35.9, 31.4, 30.8, 22.5, 14.0; HRMS (ESI) m/z calculated for C₂₁H₂₅O [M+H]⁺: 293.1900, found 293.1898.

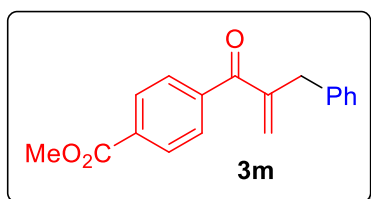
2-benzyl-1-(4-ethoxyphenyl)prop-2-en-1-one (3l)



This compound was prepared by the general procedure described above and was obtained as yellow oil (19.7 mg, 74%). R_f (PE:EtOAc = 10:1):

0.45; ¹H NMR (600 MHz, CDCl₃) δ 7.77 (d, J = 8.8 Hz, 2H), 7.31 (t, J = 7.5 Hz, 2H), 7.27 (t, J = 6.0 Hz, 2H), 7.22 (t, J = 7.2 Hz, 1H), 6.90 (d, J = 8.8 Hz, 2H), 5.67 (s, 1H), 5.61 (s, 1H), 4.10 (q, J = 7.0 Hz, 2H), 3.81 (s, 2H), 1.45 (t, J = 7.0 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 196.5, 162.6, 147.7, 138.7, 132.0, 129.9, 129.1, 128.5, 126.3, 124.7, 113.9, 63.7, 38.8, 14.7; HRMS (ESI) m/z calculated for C₁₈H₁₉O₂ [M+H]⁺: 267.1380, found 267.1378.

methyl 4-(2-benzylacryloyl)benzoate (3m)

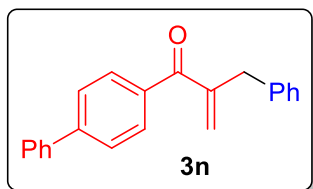


This compound was prepared by the general procedure described above and was obtained as white solid (17.4 mg, 62%). R_f (PE:EtOAc

= 10:1): 0.30; m.p. 77.1-78.9 °C; ¹H NMR (600 MHz, CDCl₃) δ 8.08 (d, J = 8.1 Hz, 2H), 7.73 (d, J = 8.1 Hz, 2H), 7.31 (t, J = 7.4 Hz, 2H), 7.26 – 7.21 (m, 3H), 5.83 (s, 1H), 5.69 (s, 1H), 3.94 (s, 3H), 3.80 (s, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 196.9, 166.3, 147.5, 141.5, 138.4, 133.0,

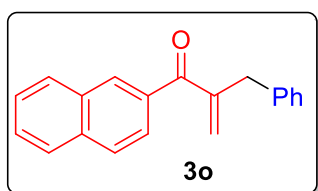
129.4, 129.2, 129.1, 128.6, 128.2, 126.5, 52.4, 38.0; HRMS (ESI) m/z calculated for $C_{18}H_{17}O_3$ $[M+H]^+$: 281.1172, found 281.1171.

1-([1,1'-biphenyl]-4-yl)-2-benzylprop-2-en-1-one (3n)



This compound was prepared by the general procedure described above and was obtained as white solid (26.2 mg, 88%). R_f (PE:EtOAc = 10:1): 0.49; m.p. 87.0-87.7 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.82 (d, J = 8.2 Hz, 2H), 7.63 (dd, J = 16.0, 7.9 Hz, 4H), 7.47 (t, J = 7.6 Hz, 2H), 7.40 (t, J = 7.3 Hz, 1H), 7.33 – 7.28 (m, 4H), 7.23 (t, J = 7.1 Hz, 1H), 5.78 (s, 1H), 5.74 (s, 1H), 3.84 (s, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 197.2, 147.7, 145.0, 140.0, 138.7, 136.3, 130.1, 129.2, 128.9, 128.5, 128.1, 127.2, 126.9, 126.5, 126.4, 38.5; HRMS (ESI) m/z calculated for $C_{22}H_{19}O$ $[M+H]^+$: 299.1430, found 299.1428.

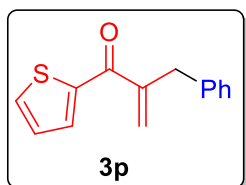
2-benzyl-1-(naphthalen-2-yl)prop-2-en-1-one (3o)



This compound was prepared by the general procedure described above and was obtained as pale yellow oil (22.4 mg, 82%). R_f (PE:EtOAc = 10:1): 0.51; ¹H NMR (600 MHz, CDCl₃) δ 8.22 (s, 1H), 7.91 (d, J = 8.1 Hz, 1H), 7.88 (dd, J = 8.2, 5.2 Hz, 2H), 7.85 (dd, J = 8.5, 1.5 Hz, 1H), 7.60 – 7.57 (m, 1H), 7.55 – 7.53 (m, 1H), 7.34 – 7.31 (m, 4H), 7.23 (ddd, J = 8.5, 6.1, 2.1 Hz, 1H), 5.80 (s, 1H), 5.76 (s, 1H), 3.87 (s, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 197.6, 147.8, 138.7, 135.2, 134.9, 132.1,

131.0, 129.3, 129.2, 128.5, 128.2, 128.1, 127.7, 126.7, 126.7, 126.4, 125.4, 38.5; HRMS (ESI) m/z calculated for $C_{20}H_{17}O$ $[M+H]^+$: 273.1274, found 273.1272.

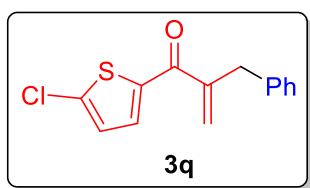
2-benzyl-1-(thiophen-2-yl)prop-2-en-1-one (3p)



This compound was prepared by the general procedure described above and was obtained as white solid (20.5 mg, 90%). R_f (PE:EtOAc = 10:1): 0.49; m.p.

63.2-65.4 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.65 (ddd, J = 4.8, 4.3, 1.1 Hz, 2H), 7.30 (t, J = 7.5 Hz, 2H), 7.26 (t, J = 7.9 Hz, 2H), 7.22 (t, J = 7.3 Hz, 1H), 7.11 (dd, J = 4.9, 3.8 Hz, 1H), 5.90 (s, 1H), 5.65 (s, 1H), 3.80 (s, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 189.1, 147.9, 143.4, 138.3, 134.0, 133.9, 129.1, 128.5, 127.8, 126.4, 124.2, 38.6; HRMS (ESI) m/z calculated for $C_{14}H_{13}OS$ $[M+H]^+$: 229.0682, found 229.0681.

2-benzyl-1-(5-chlorothiophen-2-yl)prop-2-en-1-one (3q)

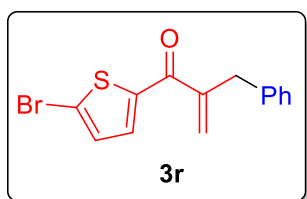


This compound was prepared by the general procedure described above and was obtained as white solid (14.9 mg, 57%). R_f (PE:EtOAc = 10:1):

0.67; m.p. 63.9-65.8 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.41 (d, J = 4.0 Hz, 1H), 7.29 (t, J = 7.5 Hz, 2H), 7.22 – 7.20 (m, 3H), 6.92 (d, J = 4.0 Hz, 1H), 5.83 (s, 1H), 5.63 (s, 1H), 3.75 (s, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 188.1, 147.2, 142.0, 139.9, 138.1, 133.4, 129.1, 128.5, 127.3, 126.5, 124.1, 38.6; HRMS (ESI) m/z calculated for $C_{14}H_{12}ClOS$ $[M+H]^+$:

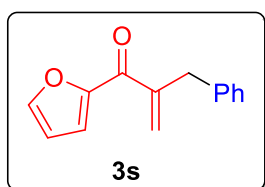
263.0292, found 263.0290.

2-benzyl-1-(5-bromothiophen-2-yl)prop-2-en-1-one (3r)



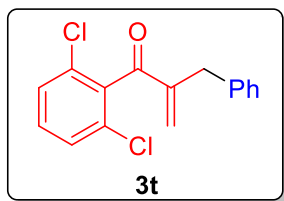
This compound was prepared by the general procedure described above and was obtained as yellow solid (15.9 mg, 52%). Rf (PE:EtOAc = 10:1): 0.54; m.p. 87.4-87.9 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.36 (d, *J* = 4.0 Hz, 1H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.22 – 7.19 (m, 3H), 7.06 (d, *J* = 4.0 Hz, 1H), 5.83 (s, 1H), 5.63 (s, 1H), 3.75 (s, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 187.9, 147.3, 144.8, 138.1, 134.0, 131.0, 129.1, 128.5, 126.5, 124.2, 123.0, 38.6; HRMS (ESI) *m/z* calculated for C₁₄H₁₂BrOS [M+H]⁺: 306.9787, found 306.9786.

2-benzyl-1-(furan-2-yl)prop-2-en-1-one (3s)



This compound was prepared by the general procedure described above and was obtained as yellow oil (15.3 mg, 72%). Rf (PE:EtOAc = 10:1): 0.35; ¹H NMR (600 MHz, CDCl₃) δ 7.62 (s, 1H), 7.28 (dd, *J* = 14.4, 7.0 Hz, 2H), 7.21 (dd, *J* = 16.4, 7.6 Hz, 3H), 7.12 (d, *J* = 3.5 Hz, 1H), 6.51 – 6.50 (m, 1H), 6.05 (s, 1H), 5.65 (s, 1H), 3.77 (s, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 183.5, 152.0, 147.3, 147.0, 138.4, 129.2, 128.5, 126.4, 125.0, 119.9, 111.9, 38.2; HRMS (ESI) *m/z* calculated for C₁₄H₁₃O₂ [M+H]⁺: 213.0910, found 213.0909.

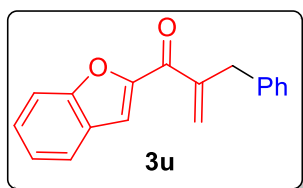
2-benzyl-1-(2,6-dichlorophenyl)prop-2-en-1-one (3t)



This compound was prepared by the general procedure described above and was obtained as colorless oil (17.1 mg, 59%). Rf (PE:EtOAc = 10:1):

0.52; ^1H NMR (600 MHz, CDCl_3) δ 7.32 (d, $J = 10.4$ Hz, 4H), 7.25 – 7.23 (m, 3H), 7.21 (s, 1H), 5.86 (s, 1H), 5.72 (s, 1H), 3.76 (s, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 194.9, 147.9, 139.9, 138.3, 132.7, 131.8, 131.1, 130.8, 129.4, 129.3, 128.5, 128.5, 126.5, 36.3; HRMS (ESI) m/z calculated for $\text{C}_{16}\text{H}_{12}\text{Cl}_2\text{ONa}$ $[\text{M}+\text{Na}]^+$: 313.0157, found 313.0162.

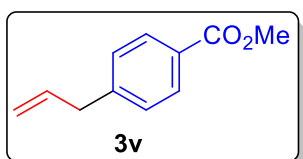
1-(benzofuran-2-yl)-2-benzylprop-2-en-1-one (3u)



This compound was prepared by the general procedure described above and was obtained as white solid (11.6 mg, 44%). Rf (PE:EtOAc = 10:1):

0.55; m.p. 94.5-96.1 °C; ^1H NMR (600 MHz, CDCl_3) δ 7.69 (d, $J = 7.9$ Hz, 1H), 7.59 (d, $J = 8.4$ Hz, 1H), 7.47 (t, $J = 7.8$ Hz, 1H), 7.43 (s, 1H), 7.32 – 7.28 (m, 3H), 7.25 (d, $J = 7.5$ Hz, 2H), 7.21 (t, $J = 7.2$ Hz, 1H), 6.18 (s, 1H), 5.77 (s, 1H), 3.83 (s, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 185.3, 156.0, 152.0, 147.5, 138.3, 129.2, 128.5, 128.3, 126.8, 126.5, 125.8, 123.9, 123.2, 115.8, 112.5, 38.3; HRMS (ESI) m/z calculated for $\text{C}_{18}\text{H}_{15}\text{O}_2$ $[\text{M}+\text{H}]^+$: 263.1067, found 263.1065.

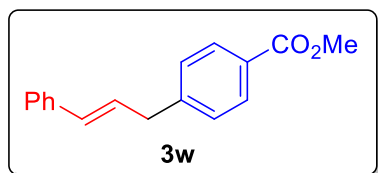
methyl 4-allylbenzoate (3v)⁷



This compound was prepared by the general procedure described above in 0.2 mmol scale and

was obtained as colorless oil (17.5 mg, 50%). Rf (PE:EtOAc = 10:1): 0.7; ^1H NMR (600 MHz, CDCl_3) δ 7.96 (d, $J = 7.6$ Hz, 2H), 7.25 (d, $J = 7.3$ Hz, 2H), 5.98 – 5.92 (m, 1H), 5.11 – 5.08 (m, 2H), 3.90 (s, 3H), 3.43 (d, $J = 6.6$ Hz, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 167.1, 145.5, 136.4, 129.7, 128.6, 128.1, 116.6, 52.0, 40.1; HRMS (ESI) m/z calculated for $\text{C}_{11}\text{H}_{13}\text{O}_2$ $[\text{M}+\text{H}]^+$: 177.0910, found 177.0914.

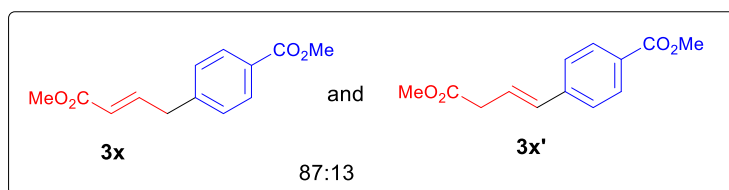
methyl 4-cinnamylbenzoate (**3w**)⁸



This compound was prepared by the general procedure described above and was obtained as colorless oil (14.1 mg, 56%). Rf

(PE:EtOAc = 10:1): 0.45; ^1H NMR (600 MHz, CDCl_3) δ 7.99 (d, $J = 8.2$ Hz, 2H), 7.36 (d, $J = 7.3$ Hz, 2H), 7.32 – 7.29 (m, 4H), 7.22 (t, $J = 7.3$ Hz, 1H), 6.47 (d, $J = 15.8$ Hz, 1H), 6.34 (dt, $J = 15.7, 6.9$ Hz, 1H), 3.91 (s, 3H), 3.60 (d, $J = 6.8$ Hz, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 167.1, 145.6, 137.2, 131.8, 129.8, 128.7, 128.5, 128.2, 128.0, 127.3, 126.1, 52.0, 39.3; HRMS (ESI) m/z calculated for $\text{C}_{17}\text{H}_{16}\text{O}_2\text{K}$ $[\text{M}+\text{K}]^+$: 291.0782, found 291.0781.

methyl (E)-4-(4-methoxy-4-oxobut-2-en-1-yl)benzoate (**3x**)⁹

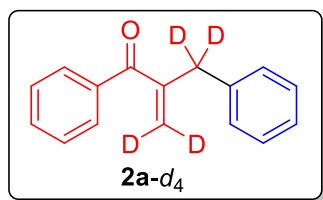


This compound was prepared by the general procedure

described above and was obtained as colorless oil (14.9 mg) of

inseparable mixture (**3x/3x'** = 87:13) in 64% yield. R_f (PE:EtOAc = 5:1): 0.40; ¹H and ¹³C NMR are described for the **3x** isomer: ¹H NMR (600 MHz, CDCl₃) δ 7.99 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.2 Hz, 2H), 7.09 (dt, *J* = 15.6, 6.8 Hz, 1H), 5.83 (d, *J* = 15.6 Hz, 1H), 3.91 (s, 3H), 3.73 (s, 3H), 3.58 (d, *J* = 6.7 Hz, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 166.9, 166.7, 146.3, 143.0, 130.0, 128.8, 128.7, 122.6, 52.1, 51.6, 38.3; HRMS (ESI) *m/z* calculated for C₁₃H₁₃O₄ [M-H]⁻: 233.0808, found 233.0818.

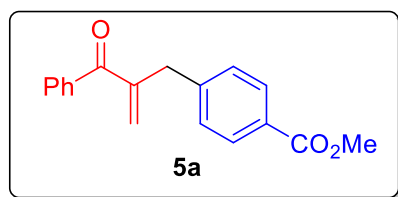
1-phenyl-2-(phenylmethyl-d₂)prop-2-en-1-one-3,3-d₂ (**2a-d₄**)



This compound was prepared by the general procedure described above and was obtained as yellow oil (42.2 mg, 75%). R_f (PE:EtOAc = 15:1):

0.49; ¹H NMR (600 MHz, CDCl₃) δ 7.73 (d, *J* = 7.9 Hz, 2H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.32 (t, *J* = 7.6 Hz, 2H), 7.29 (d, *J* = 7.3 Hz, 2H), 7.23 (t, *J* = 7.0 Hz, 1H); ¹³C NMR (151 MHz, CDCl₃) δ 197.6, 147.3, 138.6, 137.6, 132.1, 129.4, 129.1, 128.5, 128.1, 126.3, 29.6; HRMS (ESI) *m/z* calculated for C₁₆H₁₁D₄O [M+H]⁺: 227.1368, found 227.1368.

methyl 4-(2-benzoylallyl)benzoate (**5a**)

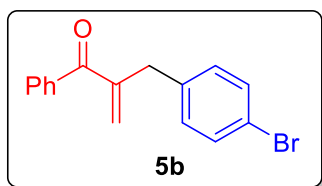


This compound was prepared by the general procedure described above and was obtained as yellow oil (25.2 mg, 90%). R_f (PE:EtOAc

= 10:1): 0.38; ¹H NMR (600 MHz, CDCl₃) δ 7.97 (d, *J* = 8.2 Hz, 2H),

7.69 (d, $J = 7.4$ Hz, 2H), 7.52 (t, $J = 7.4$ Hz, 1H), 7.41 (t, $J = 7.7$ Hz, 2H), 7.33 (d, $J = 8.1$ Hz, 2H), 5.80 (s, 1H), 5.73 (s, 1H), 3.89 (s, 3H), 3.85 (s, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 197.2, 167.0, 146.8, 144.2, 137.5, 132.3, 129.9, 129.4, 129.1, 128.4, 128.2, 127.5, 52.0, 38.4; HRMS (ESI) m/z calculated for $\text{C}_{18}\text{H}_{16}\text{O}_3\text{Na}$ $[\text{M}+\text{Na}]^+$: 303.0992, found 303.0987.

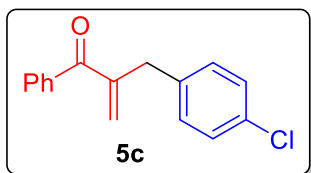
2-(4-bromobenzyl)-1-phenylprop-2-en-1-one (5b)¹⁰



This compound was prepared by the general procedure described above and was obtained as yellow oil (22.0 mg, 73%). Rf (PE:EtOAc = 10:1):

0.60; ^1H NMR (600 MHz, CDCl_3) δ 7.69 (d, $J = 7.9$ Hz, 2H), 7.53 (t, $J = 7.4$ Hz, 1H), 7.42 (dd, $J = 7.5, 6.0$ Hz, 4H), 7.14 (d, $J = 8.2$ Hz, 2H), 5.78 (s, 1H), 5.71 (s, 1H), 3.75 (s, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 197.3, 147.0, 137.7, 137.5, 132.3, 131.6, 130.9, 129.4, 128.2, 127.3, 120.3, 37.8; HRMS (ESI) m/z calculated for $\text{C}_{16}\text{H}_{14}\text{BrO}$ $[\text{M}+\text{H}]^+$: 301.0223, found 301.0221.

2-(4-chlorobenzyl)-1-phenylprop-2-en-1-one (5c)

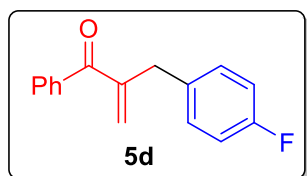


This compound was prepared by the general procedure described above and was obtained as yellow oil (20.3 mg, 79%). Rf (PE:EtOAc = 20:1):

0.32; ^1H NMR (600 MHz, CDCl_3) δ 7.70 (d, $J = 7.2$ Hz, 2H), 7.53 (t, $J = 7.3$ Hz, 1H), 7.42 (t, $J = 7.3$ Hz, 2H), 7.27 (d, $J = 7.2$ Hz, 2H), 7.20 (d, $J = 7.6$ Hz, 2H), 5.79 (s, 1H), 5.71 (s, 1H), 3.77 (s, 2H); ^{13}C NMR (151

MHz, CDCl₃) δ 197.4, 147.1, 137.5, 137.1, 132.3, 132.2, 130.5, 129.4, 128.6, 128.2, 127.3, 37.7; HRMS (ESI) m/z calculated for C₁₆H₁₄ClO [M+H]⁺: 257.0728, found 257.0727.

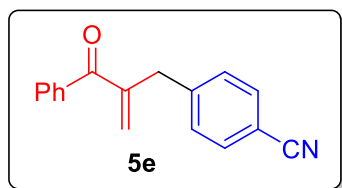
2-(4-fluorobenzyl)-1-phenylprop-2-en-1-one (5d)¹¹



This compound was prepared by the general procedure described above and was obtained as yellow oil (17.2 mg, 71%). R_f (PE:EtOAc = 20:1):

0.33; ¹H NMR (600 MHz, CDCl₃) δ 7.71 (d, J = 7.8 Hz, 2H), 7.53 (t, J = 7.4 Hz, 1H), 7.42 (t, J = 7.1 Hz, 2H), 7.23 (t, J = 6.1 Hz, 2H), 6.99 (t, J = 8.0 Hz, 2H), 5.78 (s, 1H), 5.70 (s, 1H), 3.78 (s, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 197.5, 161.5 (d, J = 244.3 Hz), 147.3, 137.5, 134.2 (d, J = 3.1 Hz), 132.3, 130.5 (d, J = 7.9 Hz), 129.4, 128.1, 127.1, 115.2 (d, J = 21.3 Hz), 37.5; HRMS (ESI) m/z calculated for C₁₆H₁₂FO [M-H]⁻: 239.0867, found 239.0867.

4-(2-benzoylallyl)benzonitrile (5e)

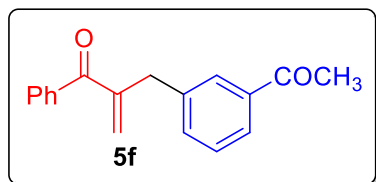


This compound was prepared by the general procedure described above and was obtained as colorless oil (10.9 mg, 44%). R_f (PE:EtOAc =

10:1): 0.33; ¹H NMR (600 MHz, CDCl₃) δ 7.68 (d, J = 8.0 Hz, 2H), 7.59 (d, J = 8.1 Hz, 2H), 7.54 (t, J = 7.3 Hz, 1H), 7.43 (t, J = 7.7 Hz, 2H), 7.38 (d, J = 8.1 Hz, 2H), 5.85 (s, 1H), 5.78 (s, 1H), 3.85 (s, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 196.9, 146.1, 144.5, 137.3, 132.5, 132.3, 129.8,

129.4, 128.3, 128.2, 118.9, 110.4, 38.5; HRMS (ESI) m/z calculated for $C_{17}H_{14}NO$ $[M+H]^+$: 248.1070, found 248.1070.

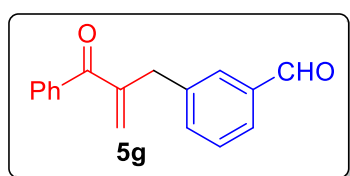
2-(3-acetylbenzyl)-1-phenylprop-2-en-1-one (5f)



This compound was prepared by the general procedure described above and was obtained as yellow oil (18.1 mg, 68%). Rf (PE:EtOAc

= 10:1): 0.25; 1H NMR (600 MHz, $CDCl_3$) δ 7.87 (s, 1H), 7.80 (d, $J = 7.7$ Hz, 1H), 7.70 (d, $J = 7.5$ Hz, 2H), 7.52 (t, $J = 7.4$ Hz, 1H), 7.48 (d, $J = 7.6$ Hz, 1H), 7.40 (dt, $J = 12.3, 7.7$ Hz, 3H), 5.81 (s, 1H), 5.74 (s, 1H), 3.86 (s, 2H), 2.59 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 198.1, 197.3, 146.9, 139.3, 137.5, 137.4, 134.0, 132.3, 129.4, 128.8, 128.8, 128.2, 127.6, 126.6, 38.2, 26.7; HRMS (ESI) m/z calculated for $C_{18}H_{17}O_2$ $[M+H]^+$: 265.1223, found 265.1222.

3-(2-benzoylallyl)benzaldehyde (5g)

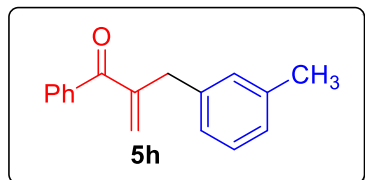


This compound was prepared by the general procedure described above and was obtained as yellow oil (13.1 mg, 52%). Rf (PE:EtOAc =

10:1): 0.33; 1H NMR (600 MHz, $CDCl_3$) δ 10.00 (s, 1H), 7.79 (s, 1H), 7.73 (d, $J = 7.5$ Hz, 1H), 7.71 – 7.69 (m, 2H), 7.54 (dd, $J = 18.2, 7.5$ Hz, 2H), 7.47 (t, $J = 7.6$ Hz, 1H), 7.42 (t, $J = 7.7$ Hz, 2H), 5.83 (s, 1H), 5.76 (s, 1H), 3.88 (s, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.2, 192.3, 146.7, 140.0, 137.5, 136.7, 135.4, 132.3, 130.0, 129.4, 129.2, 128.3, 128.1,

127.8, 38.1; HRMS (ESI) m/z calculated for $C_{17}H_{15}O_2$ $[M+H]^+$: 251.1067, found 251.1065.

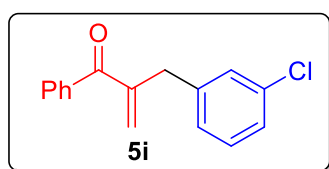
2-(3-methylbenzyl)-1-phenylprop-2-en-1-one (5h)



This compound was prepared by the general procedure described above and was obtained as yellow oil (14.6 mg, 62%). Rf (PE:EtOAc =

10:1): 0.65; 1H NMR (600 MHz, $CDCl_3$) δ 7.72 (d, $J = 7.6$ Hz, 2H), 7.52 (t, $J = 7.4$ Hz, 1H), 7.42 (t, $J = 7.7$ Hz, 2H), 7.19 (t, $J = 7.5$ Hz, 1H), 7.08 – 7.02 (m, 3H), 5.76 (s, 1H), 5.68 (s, 1H), 3.77 (s, 2H), 2.33 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.7, 147.7, 138.6, 138.1, 137.7, 132.1, 130.0, 129.5, 128.4, 128.1, 127.1, 127.0, 126.1, 38.2, 21.4; HRMS (ESI) m/z calculated for $C_{17}H_{17}O$ $[M+H]^+$: 237.1274, found 237.1272.

2-(3-chlorobenzyl)-1-phenylprop-2-en-1-one (5i)¹¹

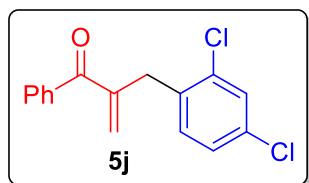


This compound was prepared by the general procedure described above and was obtained as yellow oil (18.7 mg, 73%). Rf (PE:EtOAc =

20:1): 0.32; 1H NMR (600 MHz, $CDCl_3$) δ 7.71 (d, $J = 7.4$ Hz, 2H), 7.53 (t, $J = 7.2$ Hz, 1H), 7.42 (t, $J = 7.1$ Hz, 2H), 7.27 – 7.25 (m, 1H), 7.24 – 7.21 (m, 1H), 7.20 – 7.18 (m, 1H), 7.15 (d, $J = 7.1$ Hz, 1H), 5.80 (s, 1H), 5.73 (s, 1H), 3.77 (s, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.3, 146.8, 140.7, 137.5, 134.2, 132.3, 129.7, 129.4, 129.2, 128.2, 127.7, 127.3, 126.6, 37.9; HRMS (ESI) m/z calculated for $C_{16}H_{14}ClO$ $[M+H]^+$:

257.0728, found 257.0726.

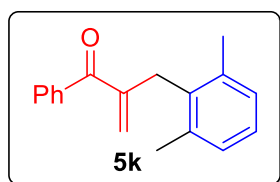
2-(2,4-dichlorobenzyl)-1-phenylprop-2-en-1-one (5j)



This compound was prepared by the general procedure described above and was obtained as yellow oil (14.5 mg, 50%). R_f (PE:EtOAc = 20:1):

0.34; ¹H NMR (600 MHz, CDCl₃) δ 7.74 (d, *J* = 7.7 Hz, 2H), 7.54 (t, *J* = 7.1 Hz, 1H), 7.44 – 7.41 (m, 3H), 7.25 (d, *J* = 9.2 Hz, 1H), 7.20 (d, *J* = 8.2 Hz, 1H), 5.74 (s, 1H), 5.67 (s, 1H), 3.88 (s, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 197.3, 145.1, 137.4, 135.1, 135.0, 133.0, 132.4, 129.5, 129.4, 128.2, 127.7, 127.2, 35.3; HRMS (ESI) *m/z* calculated for C₁₆H₁₃Cl₂O [M+H]⁺: 291.0338, found 291.0337.

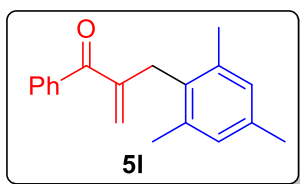
2-(2,6-dimethylbenzyl)-1-phenylprop-2-en-1-one (5k)



This compound was prepared by the general procedure described above and was obtained as colorless oil in (7.2 mg, 29%). R_f (PE:EtOAc =

10:1): 0.68; ¹H NMR (600 MHz, CDCl₃) δ 7.78 (d, *J* = 7.3 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.11 – 7.06 (m, 3H), 5.60 (s, 1H), 5.26 (s, 1H), 3.79 (s, 2H), 2.30 (s, 6H); ¹³C NMR (151 MHz, CDCl₃) δ 198.3, 145.4, 137.8, 137.2, 135.2, 132.3, 129.5, 128.2, 128.1, 126.5, 125.7, 31.4, 19.9; HRMS (ESI) *m/z* calculated for C₁₈H₁₉O [M+H]⁺: 251.1430, found 251.1428.

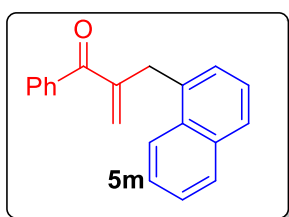
1-phenyl-2-(2,4,6-trimethylbenzyl)prop-2-en-1-one (5l)



This compound was prepared by the general procedure described above and was obtained as yellow oil (10.8 mg, 41%). Rf (PE:EtOAc =10:1):

0.68; $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.77 (d, $J = 8.0$ Hz, 2H), 7.55 (dd, $J = 10.9, 3.8$ Hz, 1H), 7.45 (t, $J = 7.6$ Hz, 2H), 6.90 (s, 2H), 5.59 (s, 1H), 5.28 (s, 1H), 3.75 (s, 2H), 2.29 (s, 3H), 2.26 (s, 6H); $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 198.4, 145.7, 137.8, 137.0, 135.9, 132.2, 132.0, 129.5, 128.9, 128.2, 125.7, 31.1, 20.9, 19.8; HRMS (ESI) m/z calculated for $\text{C}_{19}\text{H}_{21}\text{O}$ $[\text{M}+\text{H}]^+$: 265.1587, found 265.1586.

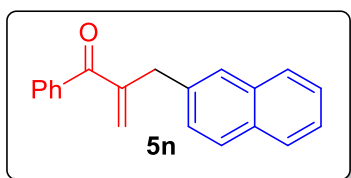
2-(naphthalen-1-ylmethyl)-1-phenylprop-2-en-1-one (5m)



This compound was prepared by the general procedure described above and was obtained as yellow oil (14.1 mg, 52%). Rf (PE:EtOAc = 10:1):

0.58; $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 8.00 (d, $J = 8.2$ Hz, 1H), 7.88 (d, $J = 7.9$ Hz, 1H), 7.78 (t, $J = 7.4$ Hz, 3H), 7.55 – 7.48 (m, 3H), 7.46 – 7.42 (m, 4H), 5.69 (s, 1H), 5.50 (s, 1H), 4.26 (s, 2H); $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 197.8, 147.0, 137.6, 134.8, 134.0, 132.3, 132.0, 129.6, 128.7, 128.2, 127.9, 127.7, 127.4, 126.1, 125.6, 125.6, 124.3, 35.0; HRMS (ESI) m/z calculated for $\text{C}_{22}\text{H}_{19}\text{O}$ $[\text{M}+\text{H}]^+$ $\text{C}_{20}\text{H}_{17}\text{O}$ $[\text{M}+\text{H}]^+$: 273.1274, found 273.1273.

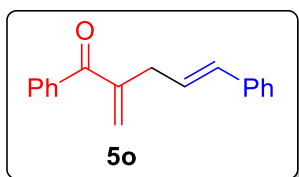
2-(naphthalen-2-ylmethyl)-1-phenylprop-2-en-1-one (5n)¹¹



This compound was prepared by the general procedure described above and was obtained as white solid (18.1 mg, 66%). R_f (PE:EtOAc =

20:1): 0.3; m.p. 78.0-78.7 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.81 (t, *J* = 7.9 Hz, 3H), 7.75 – 7.73 (m, 3H), 7.52 (t, *J* = 7.3 Hz, 1H), 7.48 – 7.44 (m, 2H), 7.43 – 7.40 (m, 3H), 5.81 (s, 1H), 5.73 (s, 1H), 3.98 (s, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 197.6, 147.5, 137.6, 136.2, 133.6, 132.2, 132.2, 129.5, 128.2, 128.1, 127.7, 127.6, 127.6, 127.3, 126.0, 125.4, 38.4; HRMS (ESI) *m/z* calculated for C₂₀H₁₇O [M+H]⁺: 273.1274, found 273.1273.

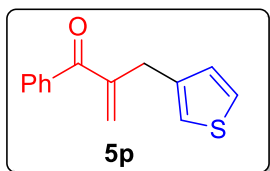
(E)-2-methylene-1,5-diphenylpent-4-en-1-one (5o)¹²



This compound was prepared by the general procedure described above and was obtained as colorless oil (11.6 mg, 47%). R_f (PE:EtOAc =

50:1): 0.24; ¹H NMR (400 MHz, CDCl₃) δ 7.79 – 7.77 (m, 2H), 7.57 – 7.53 (m, 1H), 7.47 – 7.43 (m, 2H), 7.39 – 7.37 (m, 2H), 7.33 – 7.29 (m, 2H), 7.24 – 7.20 (m, 1H), 6.52 (d, *J* = 15.8 Hz, 1H), 6.34 – 6.27 (m, 1H), 5.95 (s, 1H), 5.72 (s, 1H), 3.40 – 3.38 (m, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 197.7, 146.5, 137.6, 137.3, 132.4, 132.2, 129.5, 128.5, 128.2, 127.2, 126.8, 126.5, 126.1, 35.4; HRMS (ESI) *m/z* calculated for C₁₈H₁₇O [M+H]⁺: 249.1274, found 249.1276.

1-phenyl-2-(thiophen-3-ylmethyl)prop-2-en-1-one (5p)



This compound was prepared by the general procedure described above and was obtained as yellow oil (10.6 mg, 46%). R_f (PE:EtOAc = 50:1):

0.24; ¹H NMR (600 MHz, CDCl₃) δ 7.73 – 7.72 (m, 2H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 1H), 7.22 – 7.26 (m, 1H), 7.06 – 7.04 (m, 1H), 6.99 – 6.98 (m, 1H), 5.80 (s, 1H), 5.69 (s, 1H), 3.83 (s, 1H); ¹³C NMR (151 MHz, CDCl₃) δ 197.6, 147.0, 138.8, 137.6, 132.2, 129.5, 128.5, 128.2, 126.8, 125.7, 121.9, 32.8; HRMS (ESI) *m/z* calculated for C₁₄H₁₃OS [M+H]⁺: 229.0682, found 229.0683.

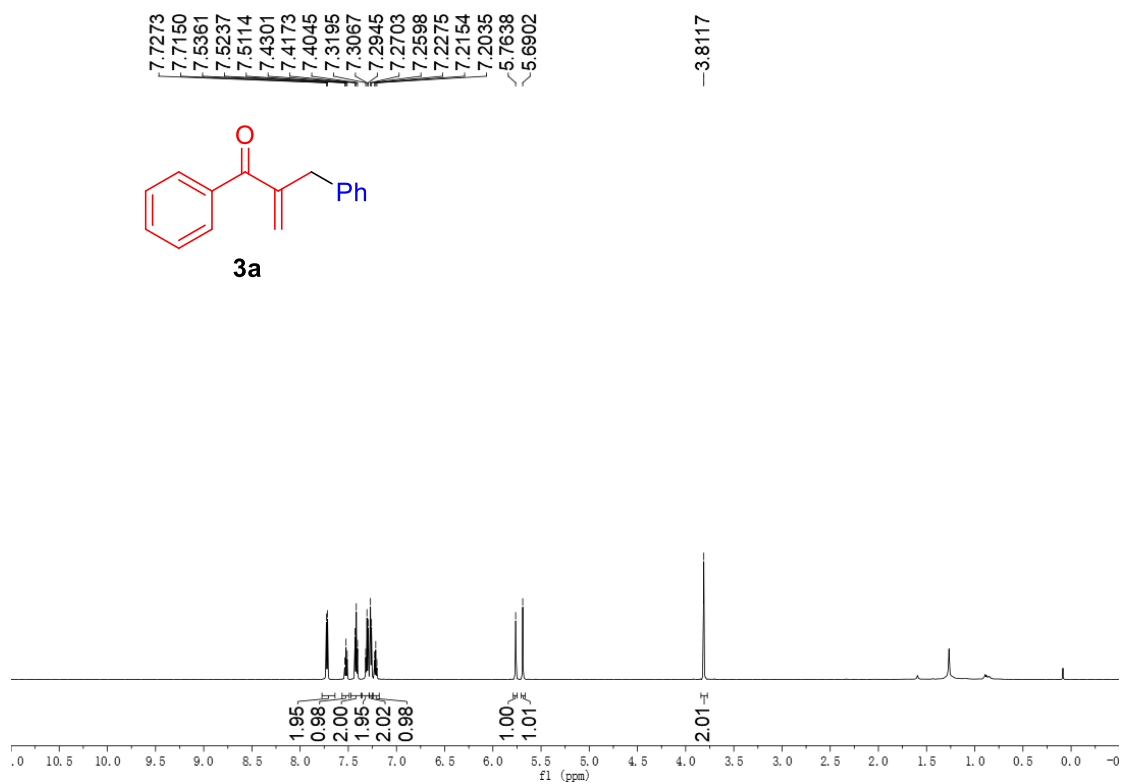
III. Reference:

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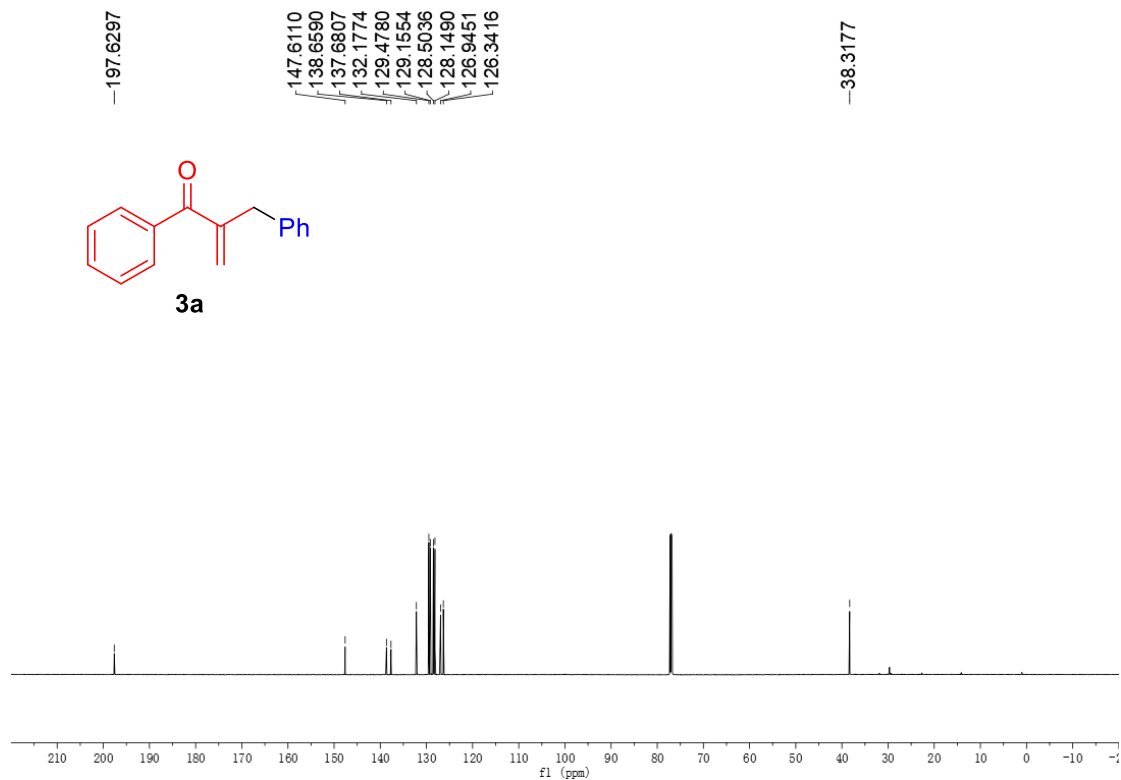
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IV. Copy of ^1H NMR, ^{13}C NMR Spectra

^1H NMR spectrum of **3a** (600 MHz, CDCl_3)



^{13}C NMR spectrum of **3a** (151 MHz, CDCl_3)

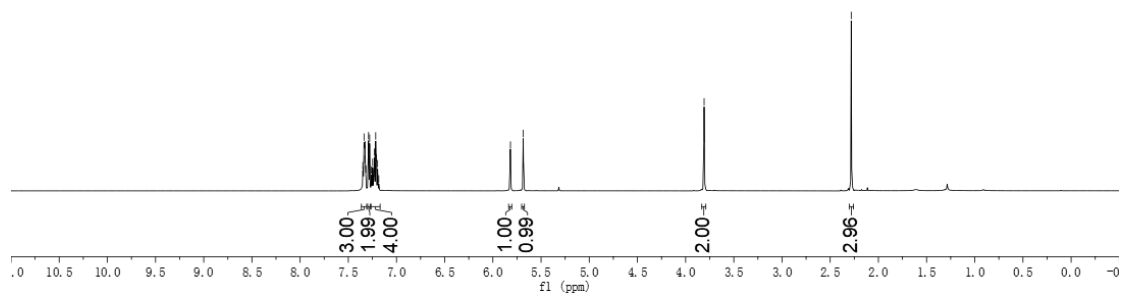
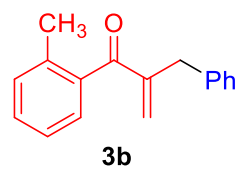


¹H NMR spectrum of **3b** (600 MHz, CDCl₃)

7.3496
7.3452
7.3370
7.3298
7.3246
7.3178
7.2900
7.2786
7.2595
7.2474
7.2351
7.2288
7.2157
7.2084
7.1962
7.1839
5.8176
5.6850

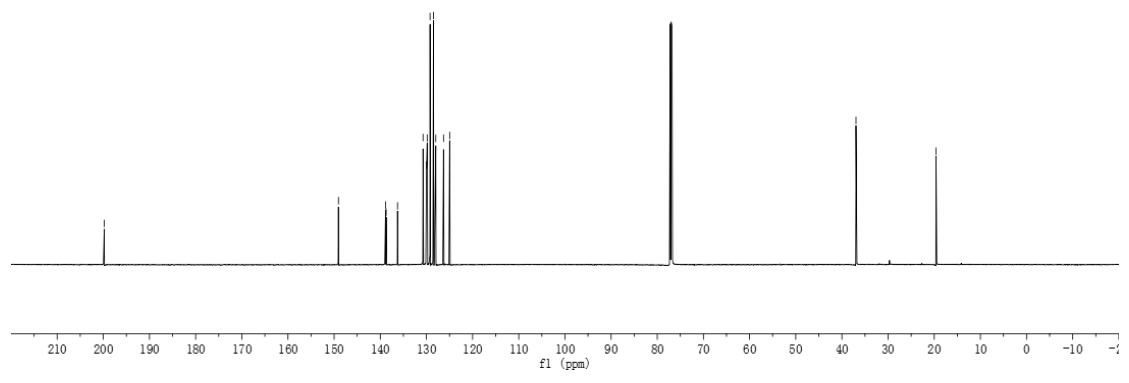
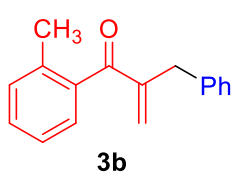
-3.8082

-2.2817



¹³C NMR spectrum of **3b** (151 MHz, CDCl₃)

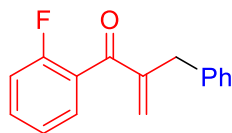
199.7953
149.0628
138.8572
138.7291
136.2634
130.7362
129.9948
129.8335
129.2135
128.4813
127.9786
126.3032
124.9714
36.9492
19.6051



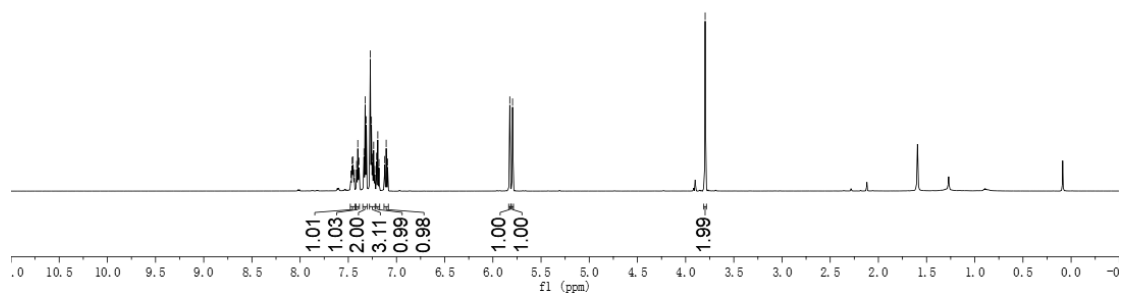
¹H NMR spectrum of **3c** (600 MHz, CDCl₃)

7.4722
7.4602
7.4499
7.4376
7.4136
7.4018
7.3900
7.3378
7.3252
7.3130
7.2717
7.2598
7.2489
7.2368
7.2246
7.2065
7.1940
7.1815
7.1223
7.1071
7.0920
5.8246
5.7959

-3.7974



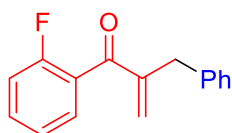
3c



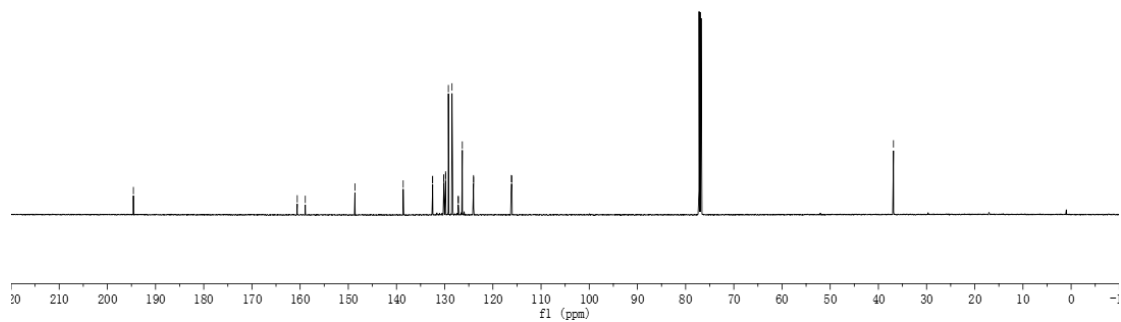
¹³C NMR spectrum of **3c** (151 MHz, CDCl₃)

-194.6019
160.5921
158.9243
148.6329
138.6076
132.5431
132.4884
130.1967
130.1771
129.8241
129.8131
129.2357
128.4778
127.2281
127.1280
126.3373
124.0150
123.9914
116.1789
116.0351

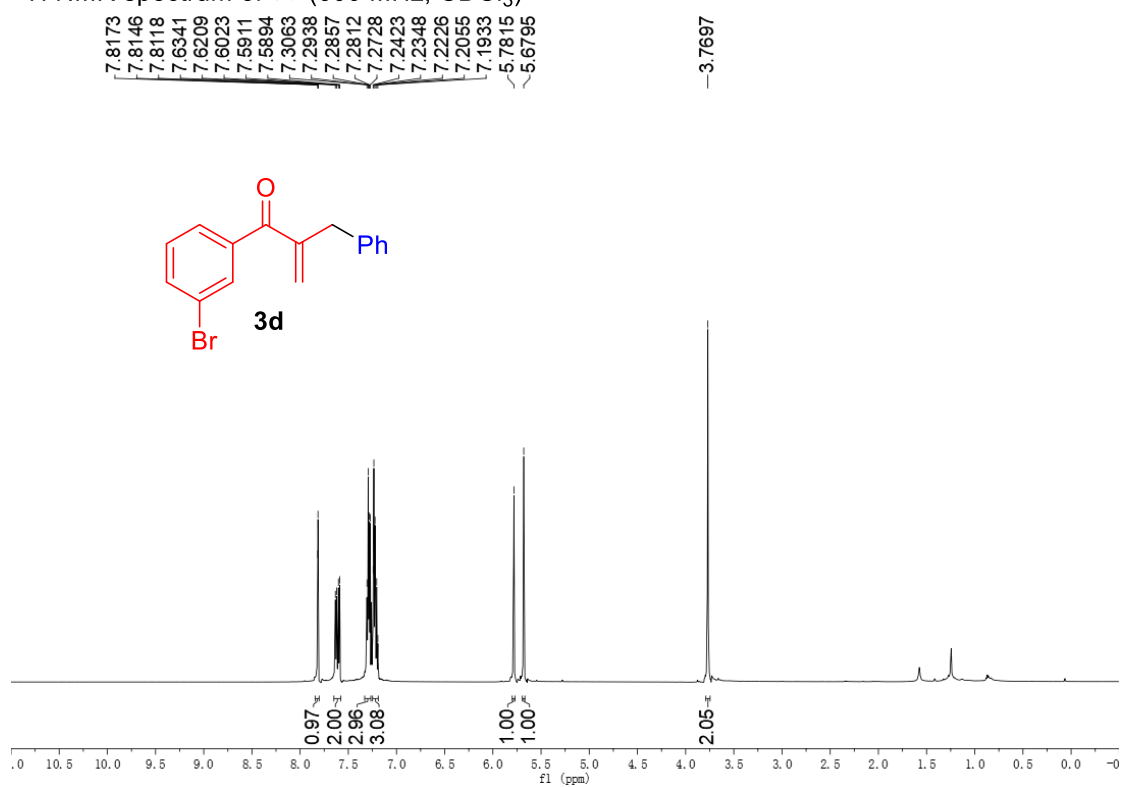
-36.8651



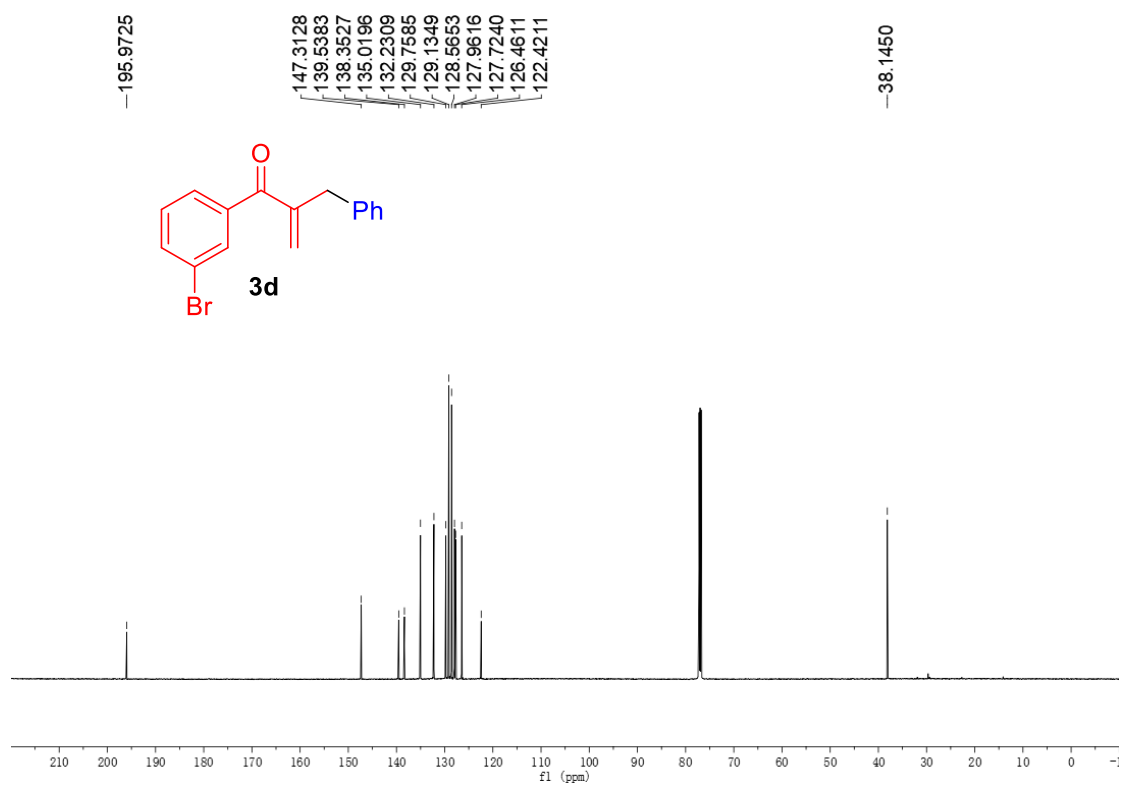
3c



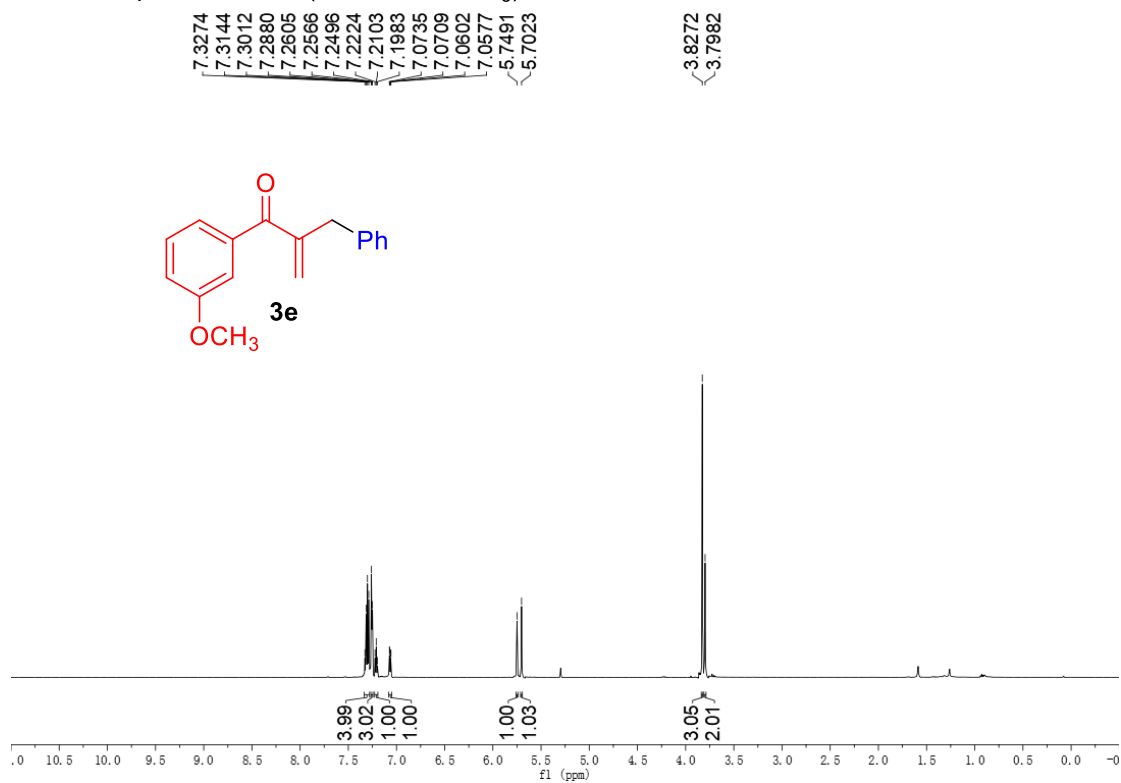
¹H NMR spectrum of **3d** (600 MHz, CDCl₃)



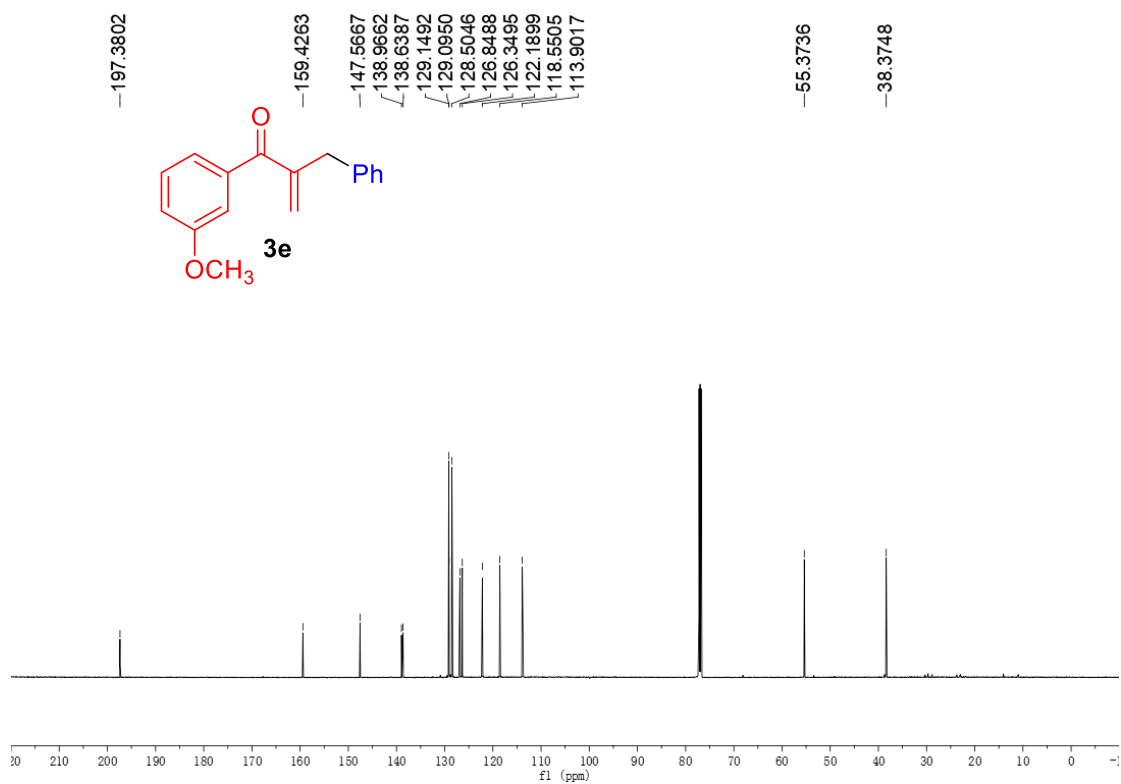
¹³C NMR spectrum of **3d** (151 MHz, CDCl₃)



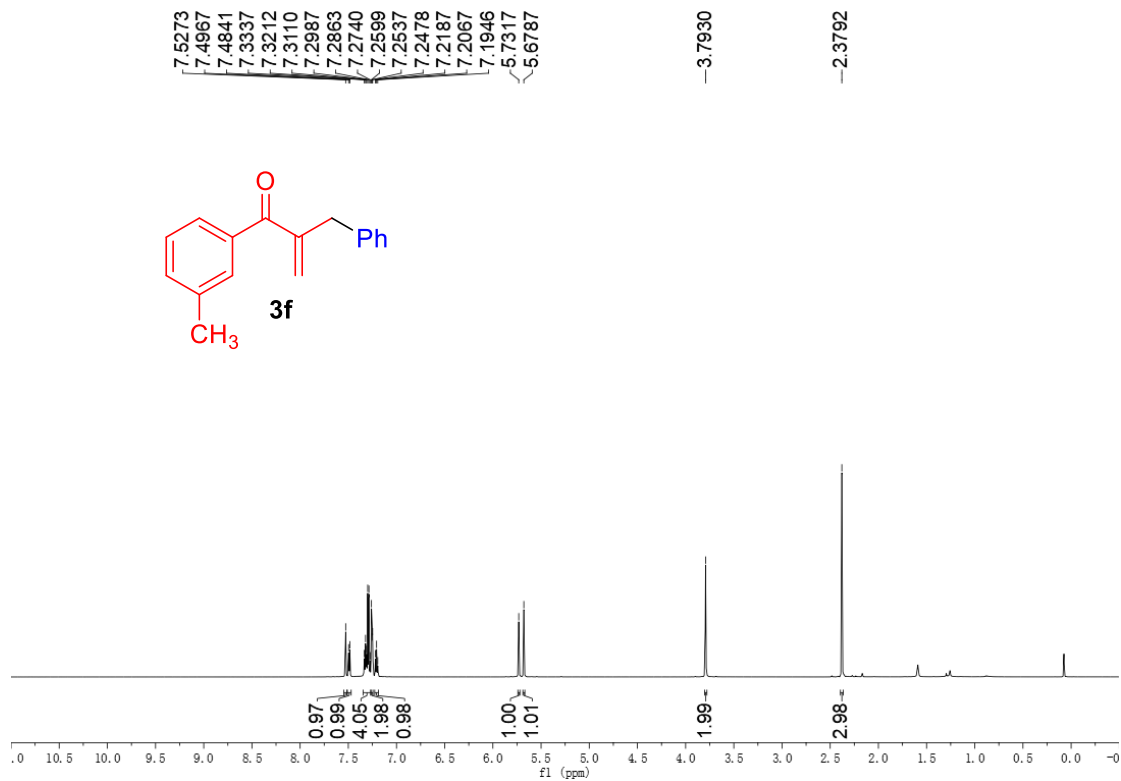
¹H NMR spectrum of **3e** (600 MHz, CDCl₃)



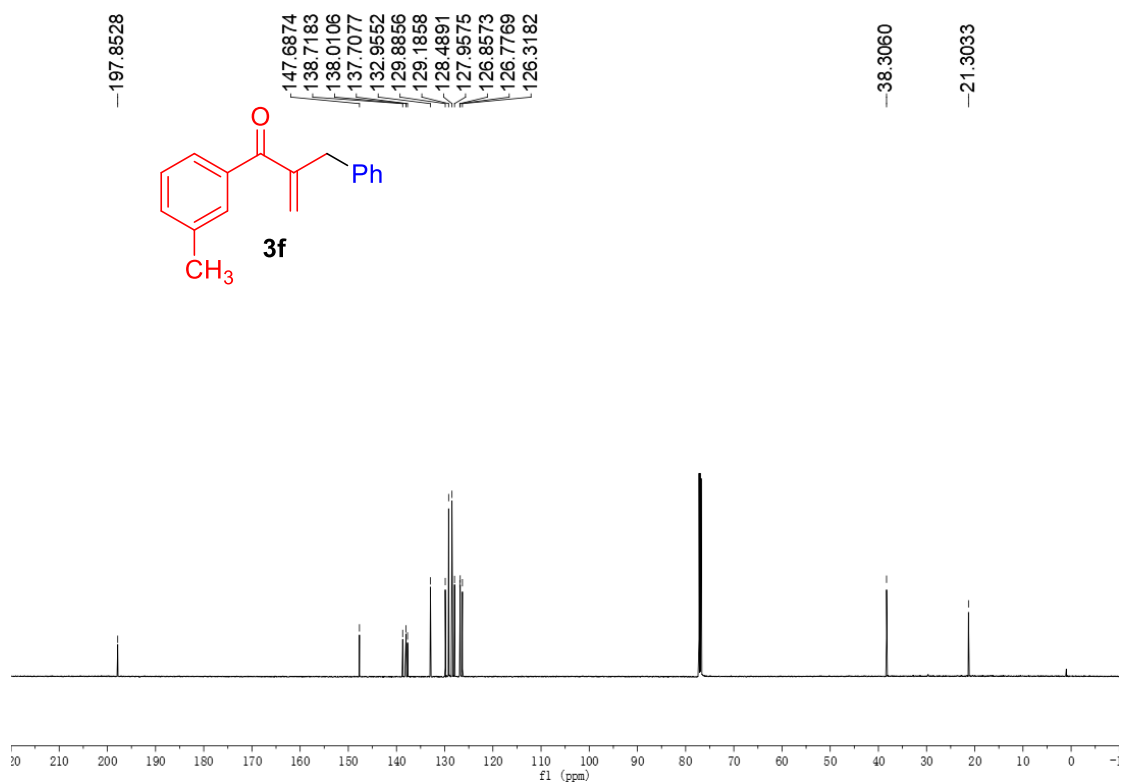
¹³C NMR spectrum of **3e** (151 MHz, CDCl₃)



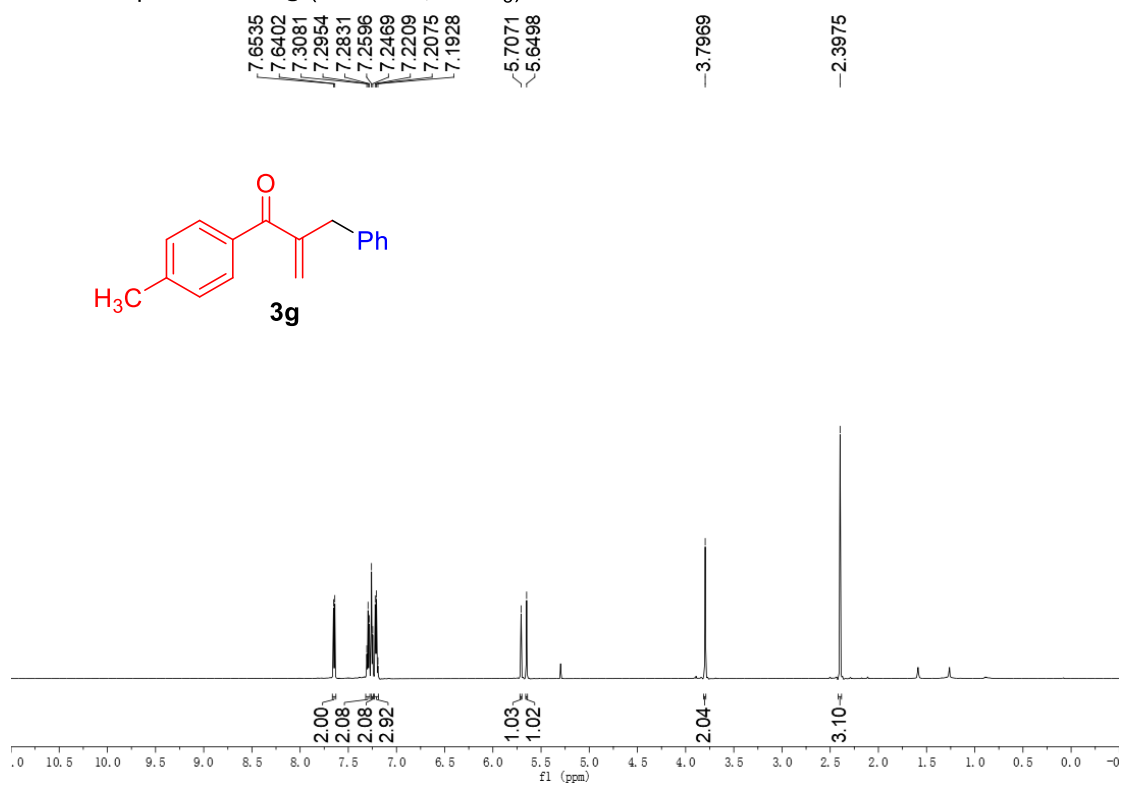
¹H NMR spectrum of **3f** (600 MHz, CDCl₃)



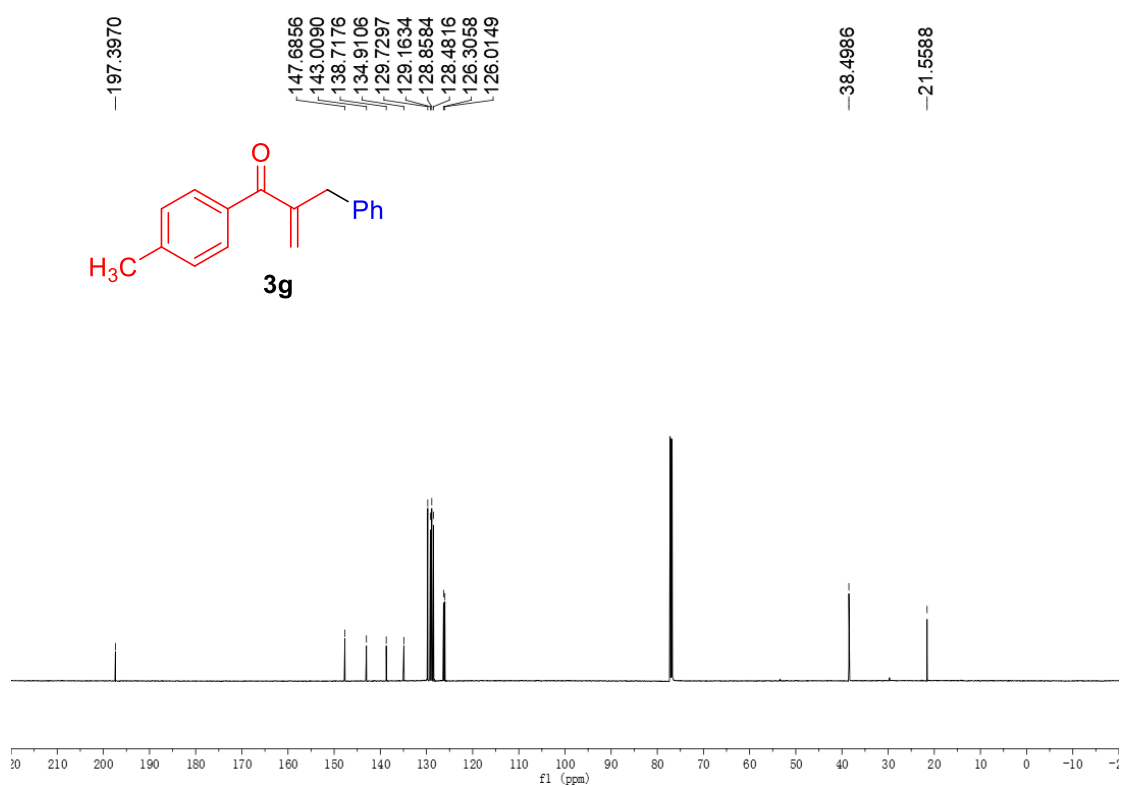
¹³C NMR spectrum of **3f** (151 MHz, CDCl₃)



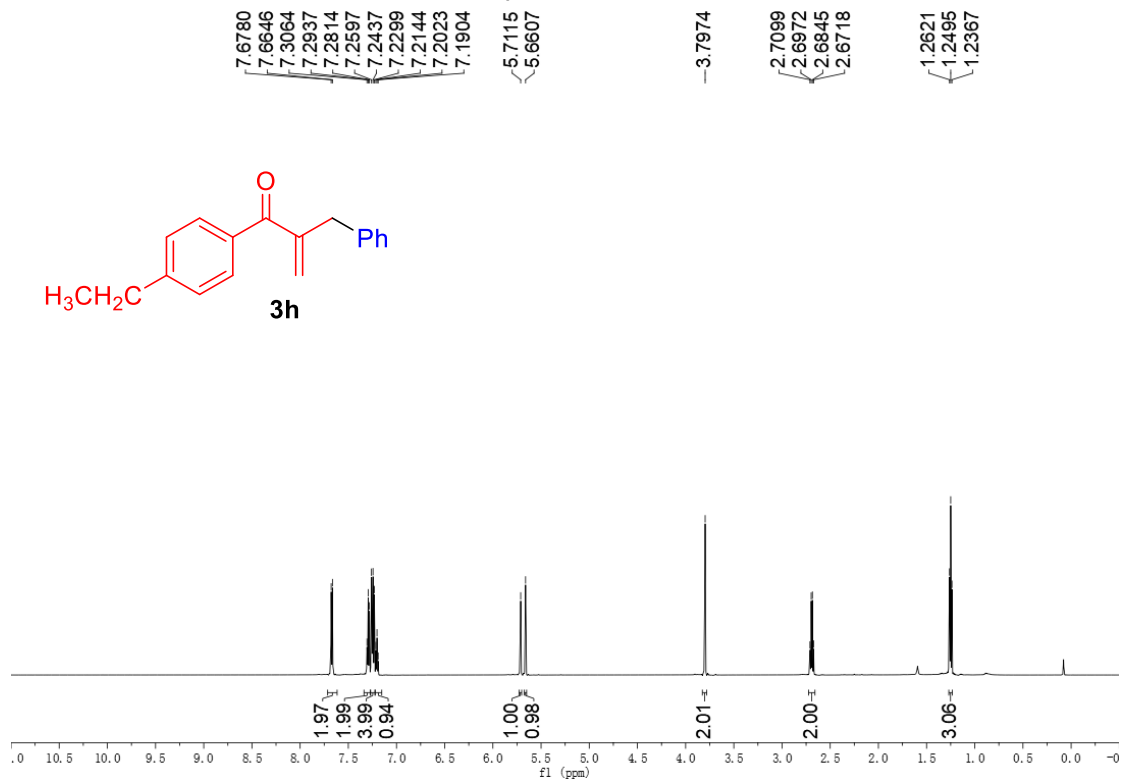
¹H NMR spectrum of **3g** (600 MHz, CDCl₃)



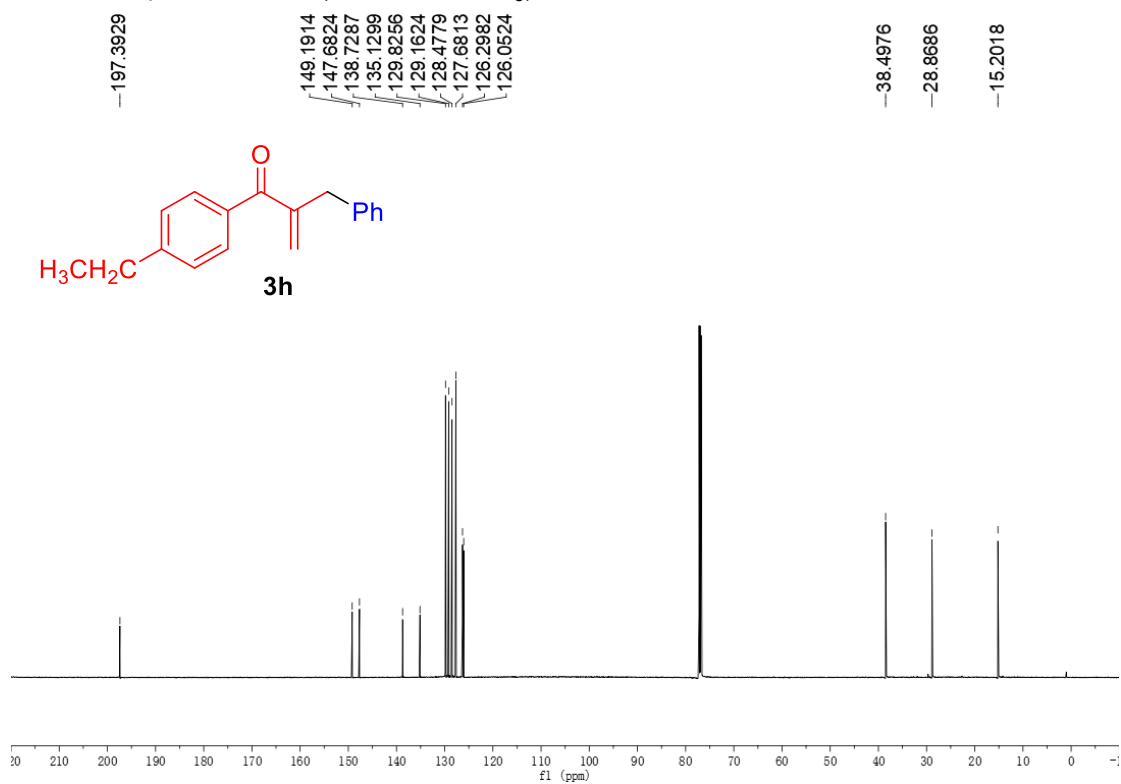
¹³C NMR spectrum of **3g** (151 MHz, CDCl₃)



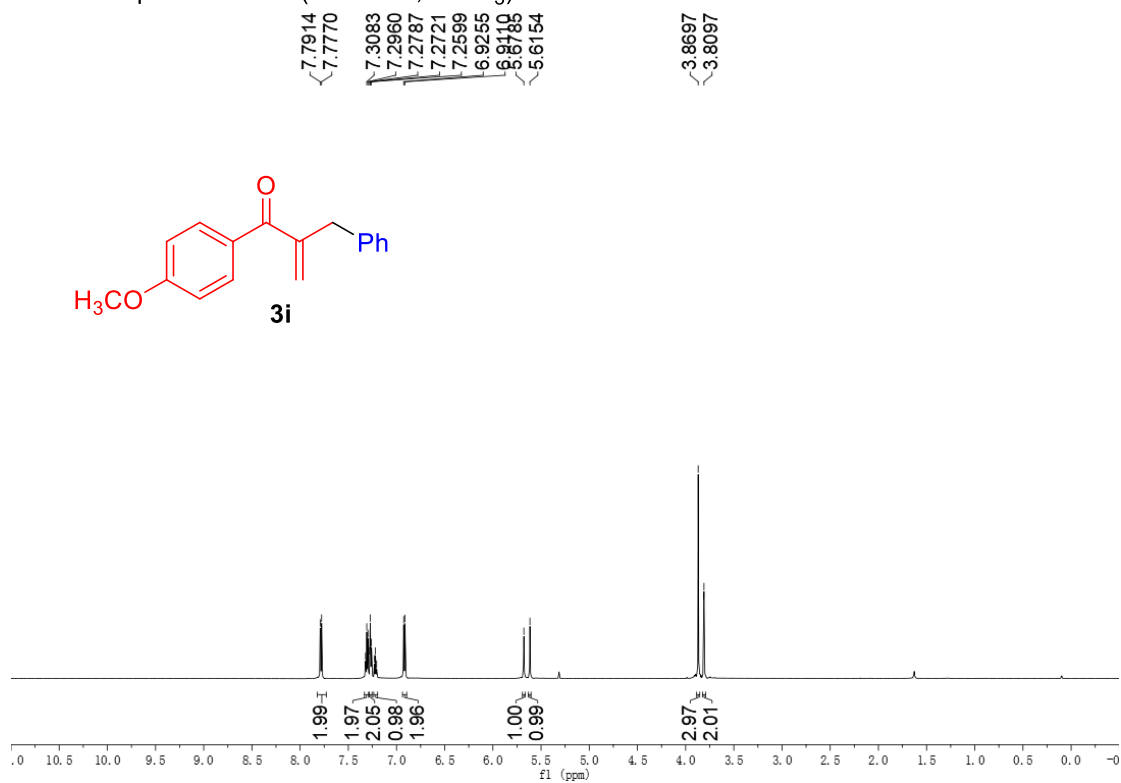
¹H NMR spectrum of **3h** (600 MHz, CDCl₃)



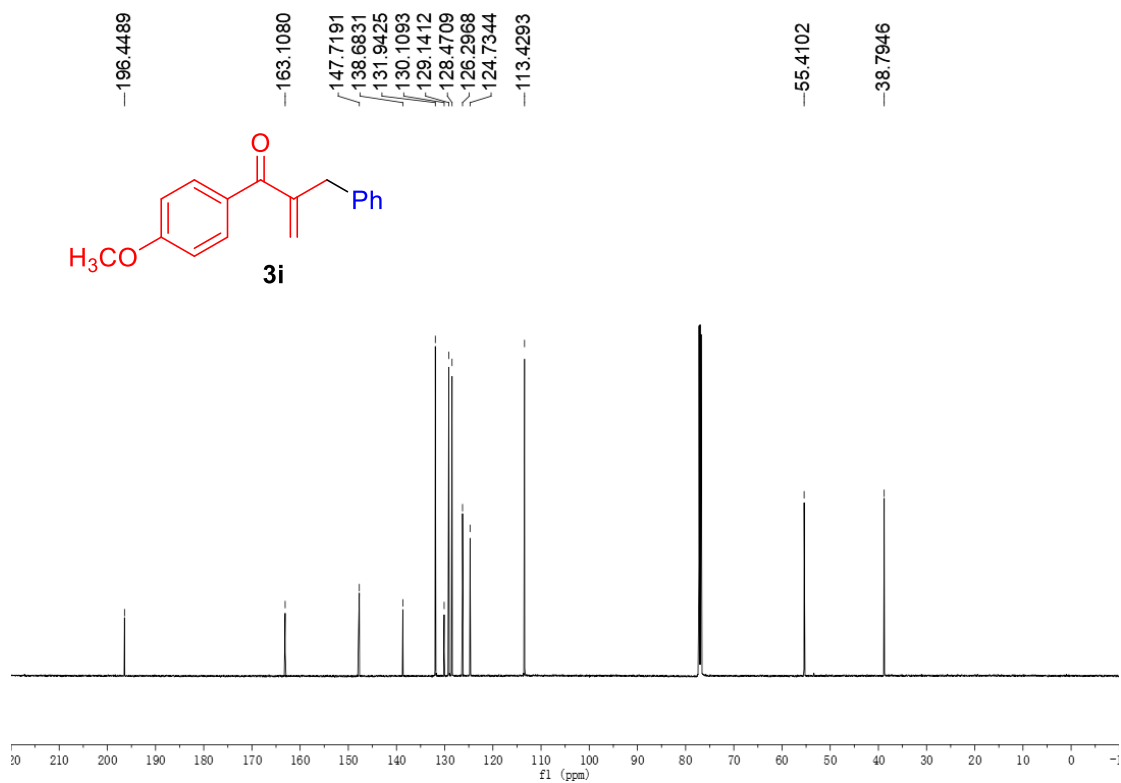
¹³C NMR spectrum of **3h** (151 MHz, CDCl₃)



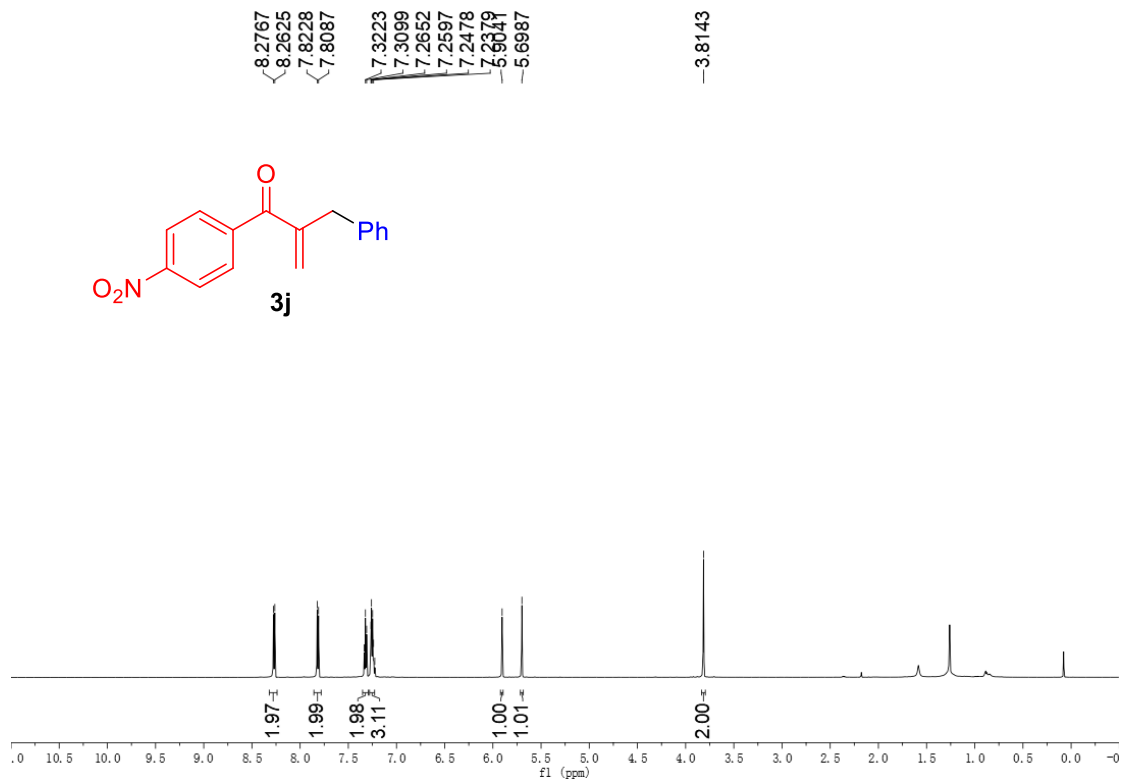
¹H NMR spectrum of **3i** (600 MHz, CDCl₃)



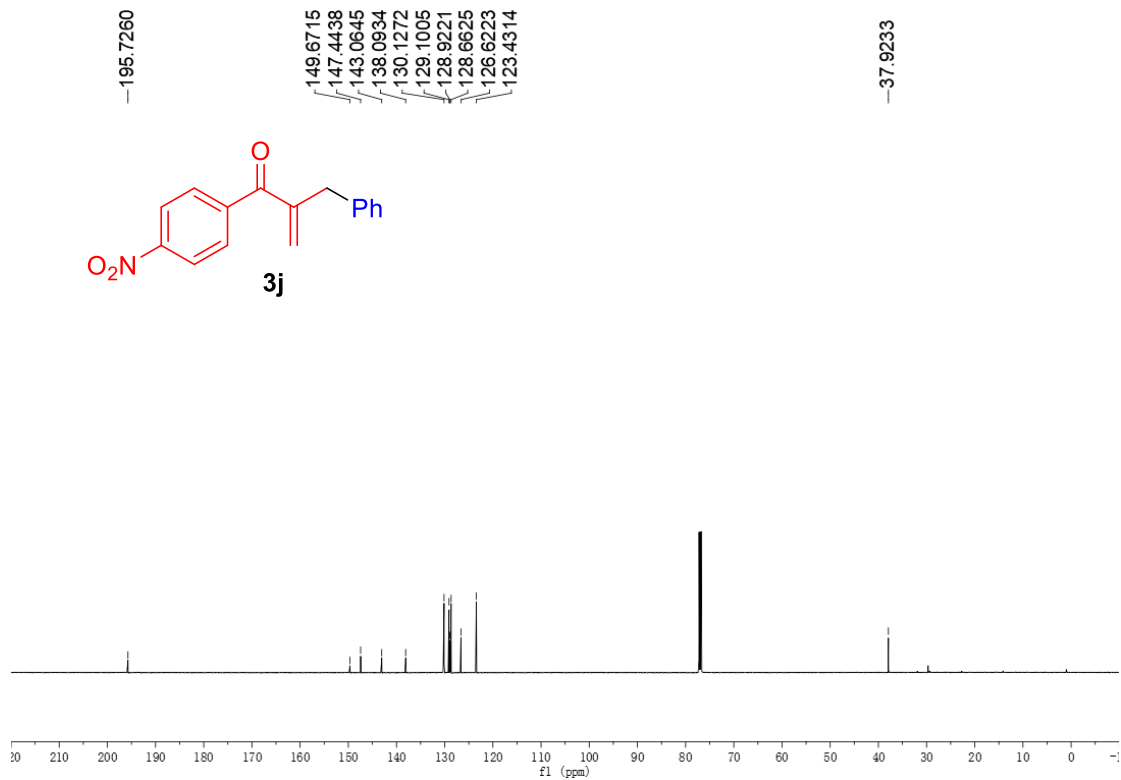
¹³C NMR spectrum of **3i** (151 MHz, CDCl₃)



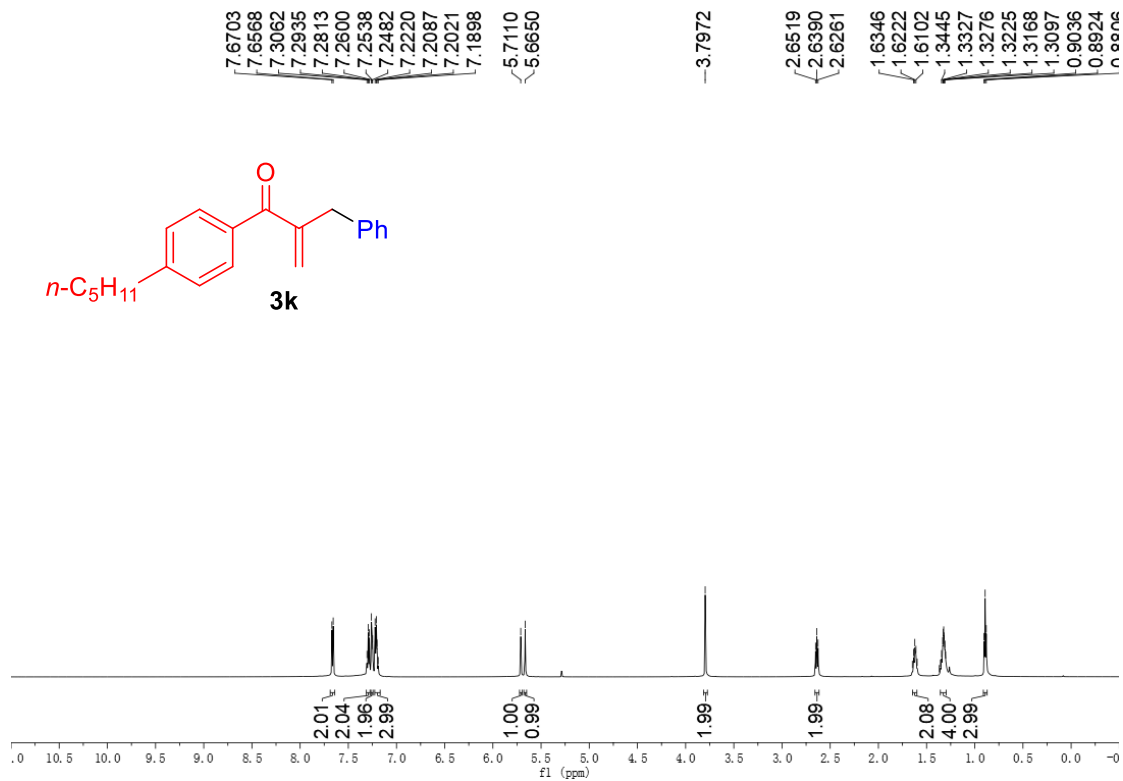
^1H NMR spectrum of **3j** (600 MHz, CDCl_3)



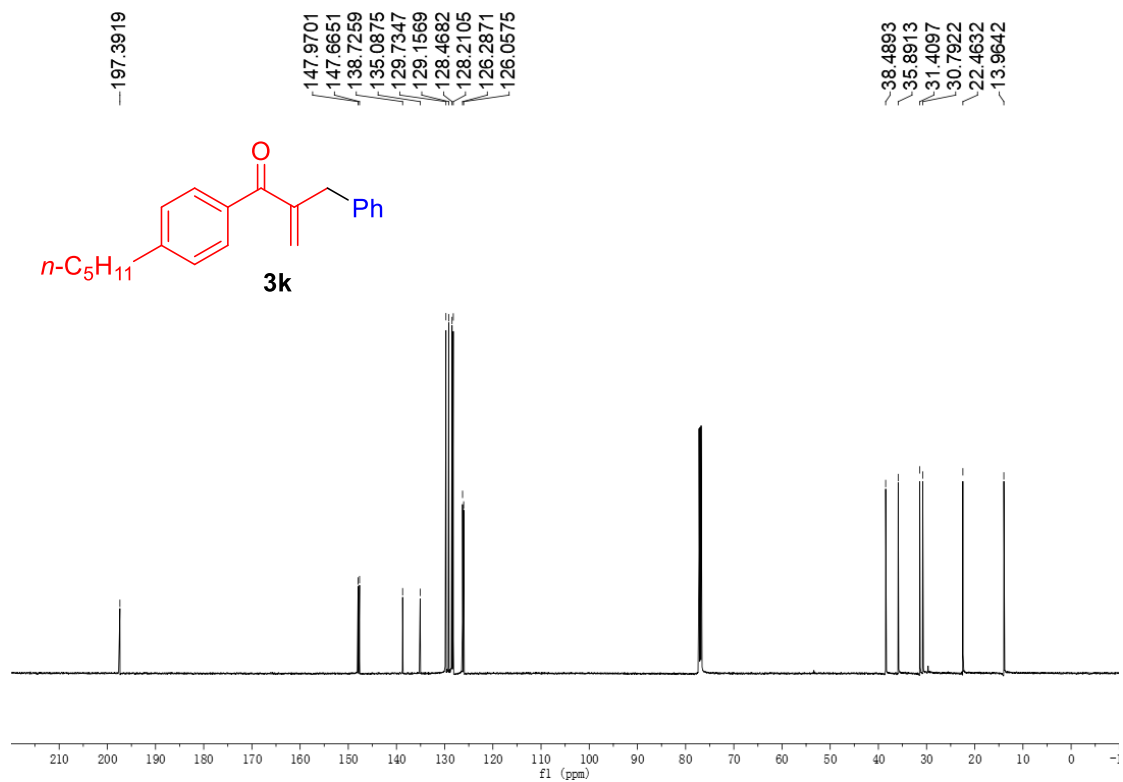
^{13}C NMR spectrum of **3j** (151 MHz, CDCl_3)



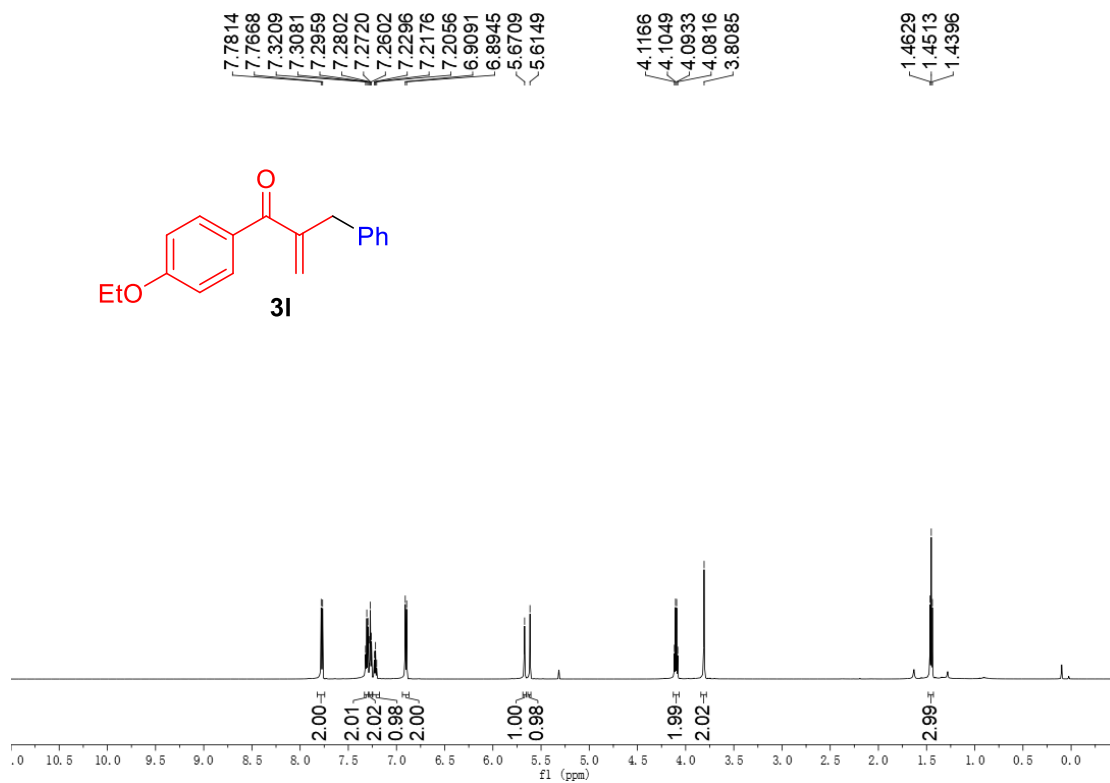
¹H NMR spectrum of **3k** (600 MHz, CDCl₃)



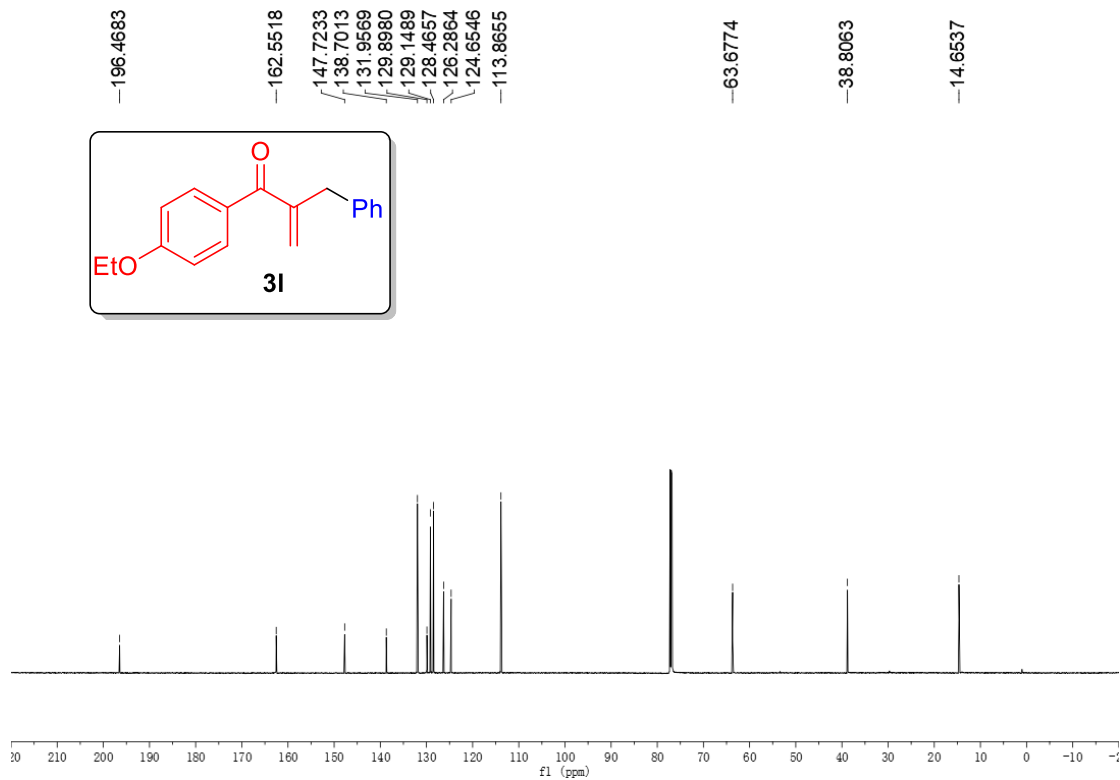
¹³C NMR spectrum of **3k** (151 MHz, CDCl₃)



¹H NMR spectrum of **3I** (600 MHz, CDCl₃)

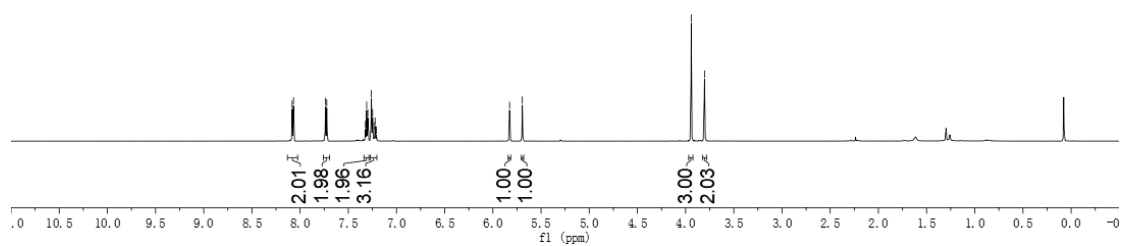
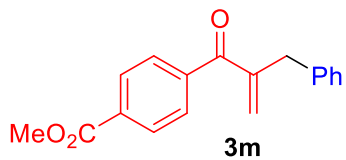


¹³C NMR spectrum of **3I** (151 MHz, CDCl₃)



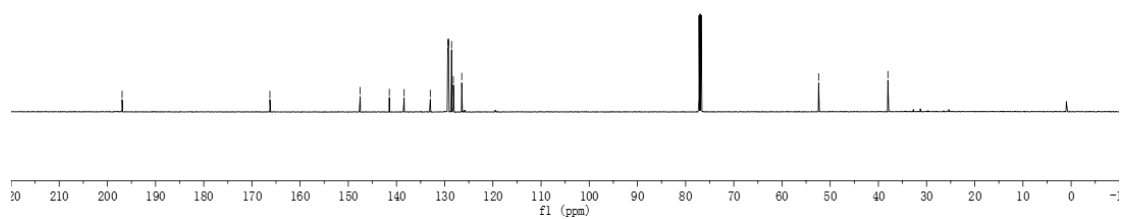
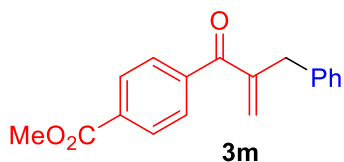
¹H NMR spectrum of **3m** (600 MHz, CDCl₃)

8.0827
8.0693
7.7370
7.7235
7.3222
7.3096
7.2974
7.2605
7.2473
7.2333
7.2213
7.2092
5.8267
5.6944
3.9417
3.8041

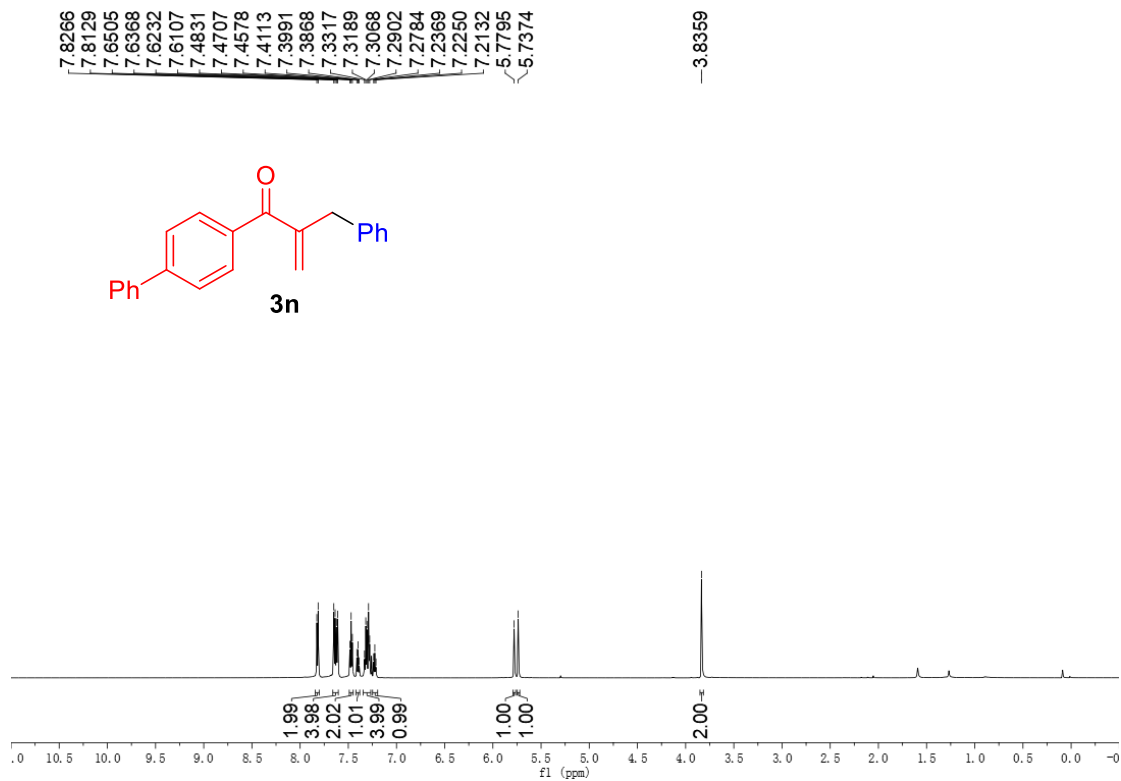


¹³C NMR spectrum of **3m** (151 MHz, CDCl₃)

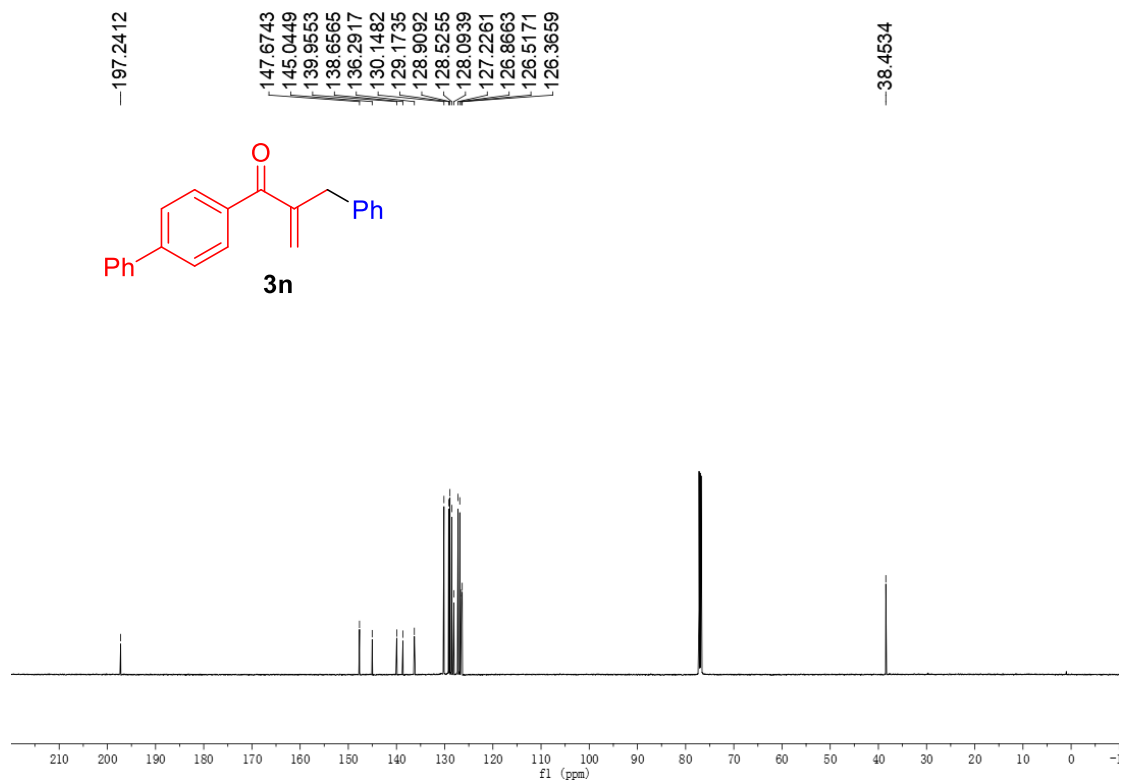
196.9442
166.2766
147.5312
141.4923
138.4141
132.9871
129.3805
129.1970
129.1383
128.5704
128.1968
126.4624
52.4007
38.0298



¹H NMR spectrum of **3n** (600 MHz, CDCl₃)



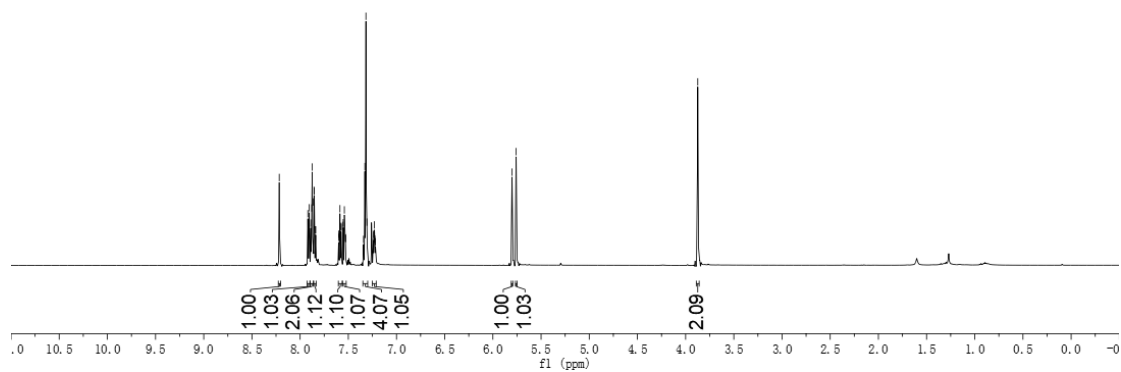
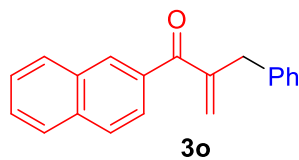
¹³C NMR spectrum of **3n** (151 MHz, CDCl₃)



¹H NMR spectrum of **3o** (600 MHz, CDCl₃)

8.2165
7.9199
7.9065
7.8886
7.8795
7.8744
7.8664
7.8554
7.8529
7.8412
7.8387
7.5885
7.5888
7.5753
7.5733
7.5544
7.5526
7.5410
7.3282
7.3172
7.3064
5.6288
5.6014
5.7585

—3.8740

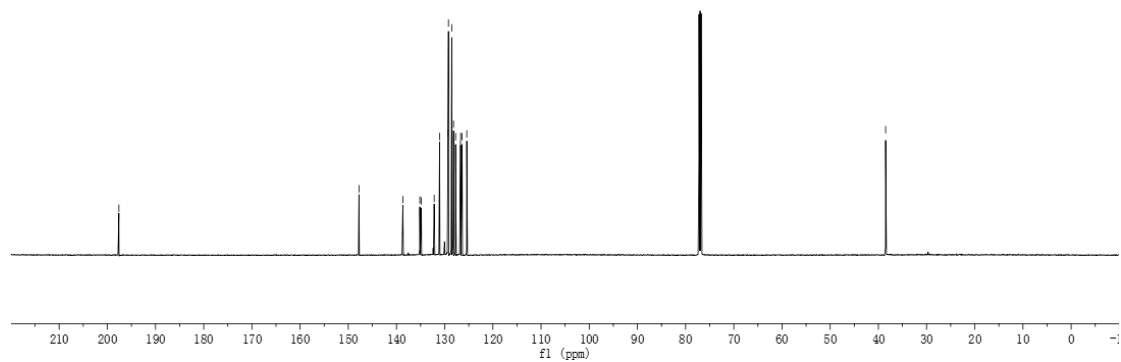
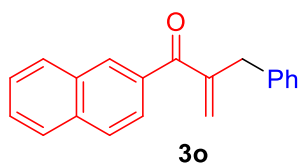


¹³C NMR spectrum of **3o** (151 MHz, CDCl₃)

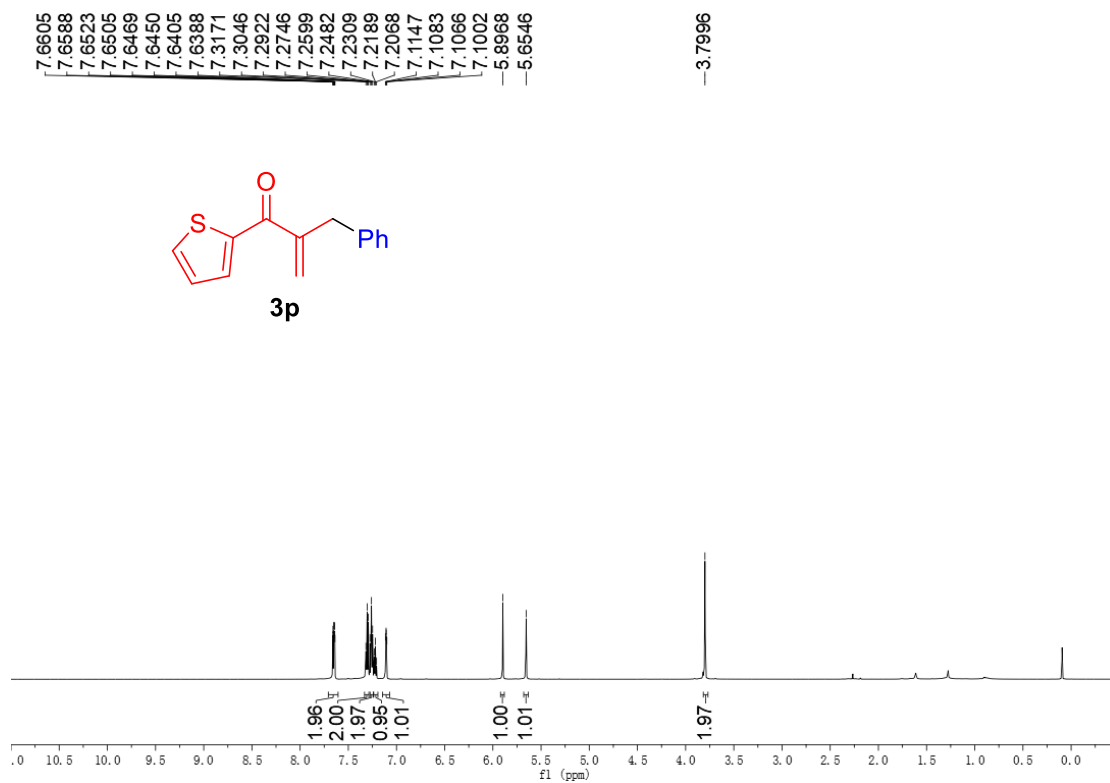
—197.6036

147.7826
138.6747
135.1583
134.8506
132.1814
131.0496
129.3297
129.2116
128.5377
128.1565
128.1363
127.7297
126.7153
126.6806
126.3836
125.4126

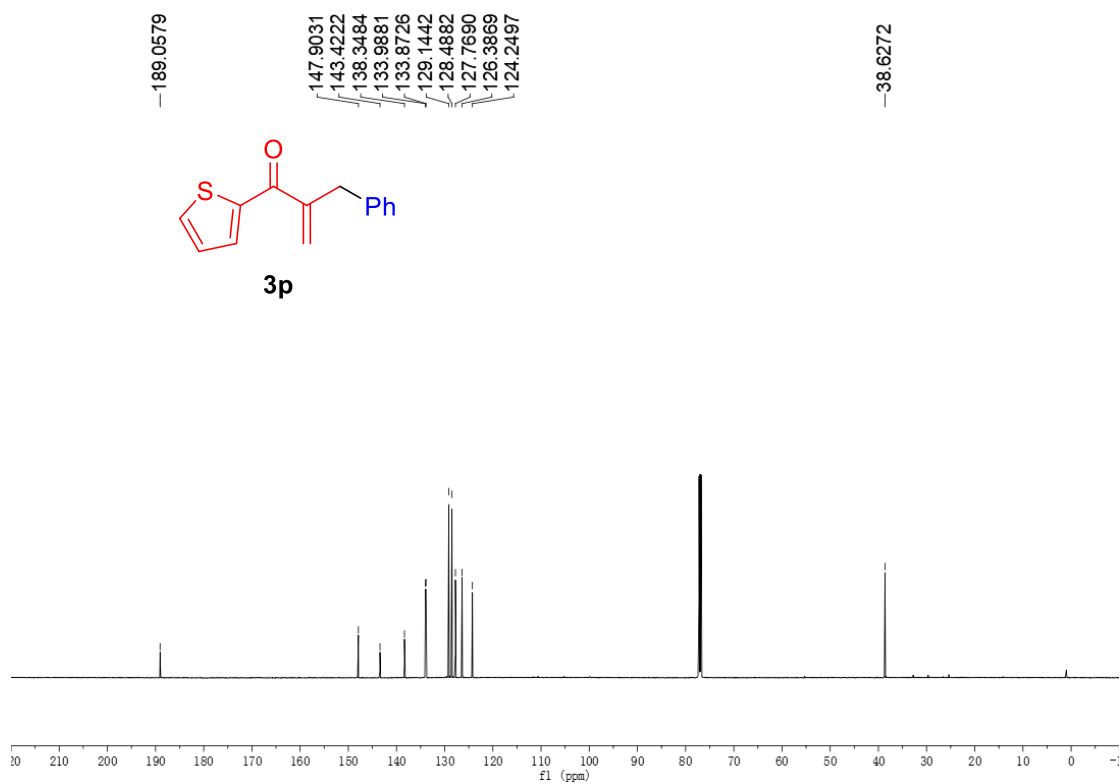
—38.4969



¹H NMR spectrum of **3p** (600 MHz, CDCl₃)



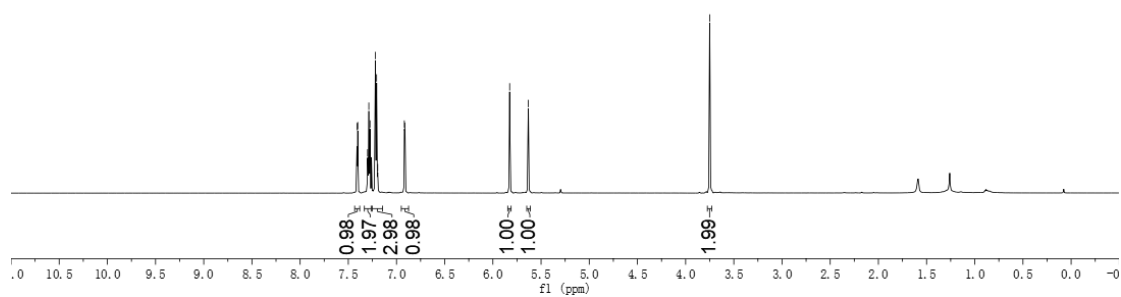
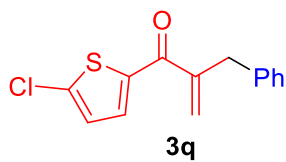
¹³C NMR spectrum of **3p** (151 MHz, CDCl₃)



^1H NMR spectrum of **3q** (600 MHz, CDCl_3)

7.4084
7.4017
7.2999
7.2871
7.2749
7.2186
7.2065
7.1951
6.9186
6.9119
5.8259
5.6321

3.7502

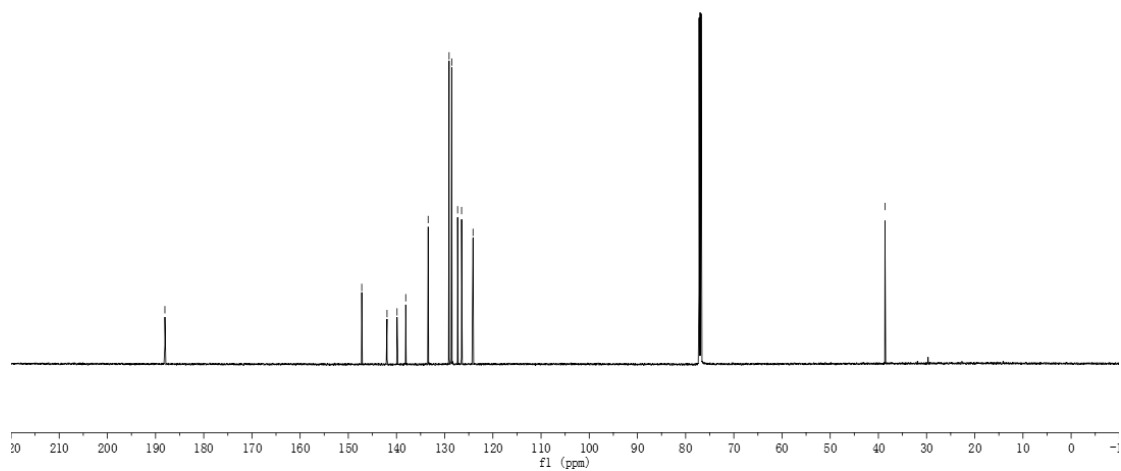
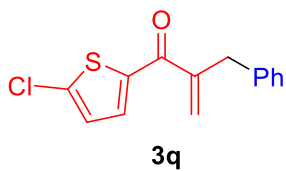


^{13}C NMR spectrum of **3q** (151 MHz, CDCl_3)

188.0618

147.2108
141.9651
139.8828
138.0834
133.4308
129.0830
128.5409
127.3180
126.4902
124.1219

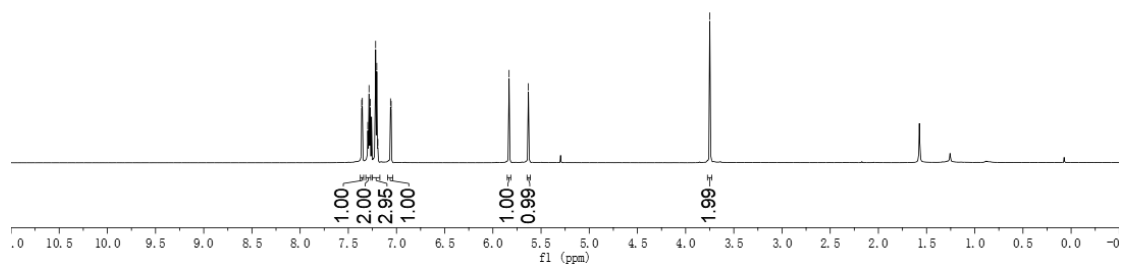
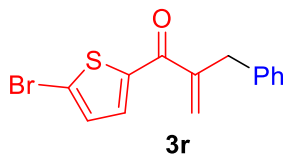
38.6367



¹H NMR spectrum of **3r** (600 MHz, CDCl₃)

7.3612
7.3545
7.2978
7.2850
7.2728
7.2165
7.2046
7.1934
7.0620
7.0554
-5.8319
-5.6314

-3.7493

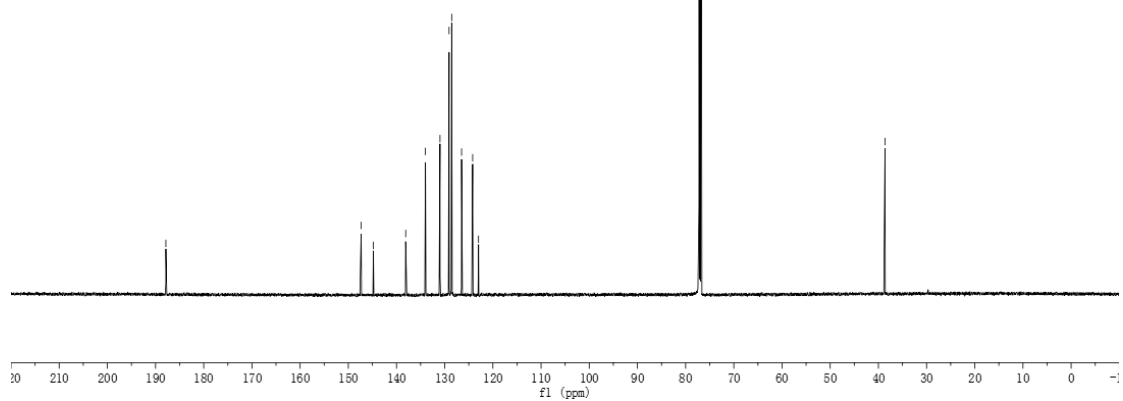
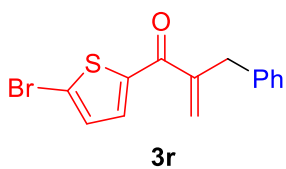


¹³C NMR spectrum of **3r** (151 MHz, CDCl₃)

-187.8568

147.3319
144.7944
138.0926
133.9961
130.9724
129.0871
128.5466
126.4943
124.1936
122.9786

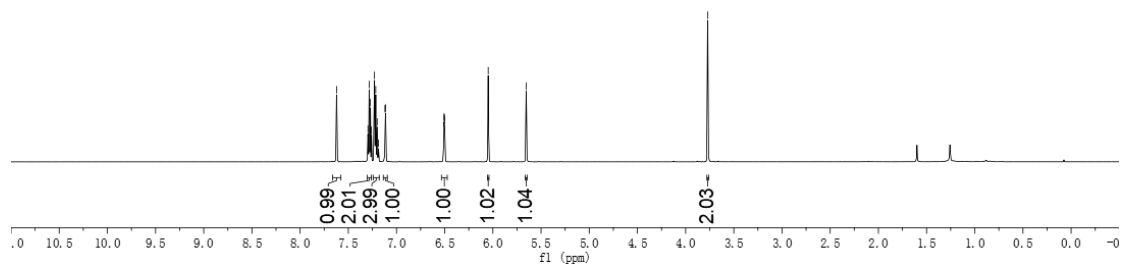
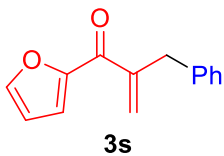
-38.6233



¹H NMR spectrum of **3s** (600 MHz, CDCl₃)

7.6217
7.2959
7.2833
7.2709
7.2602
7.2283
7.2151
7.2004
7.1883
7.1199
7.1141
6.5066
6.5035
6.5023
6.0485
-5.6539

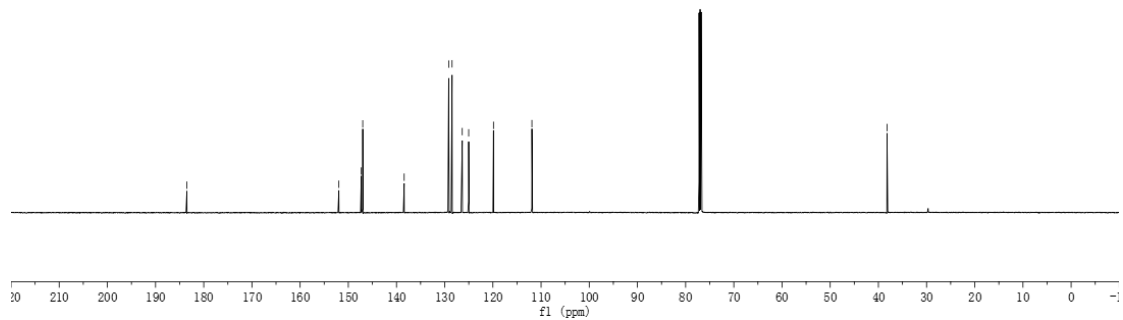
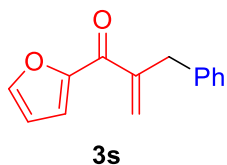
-3.7713



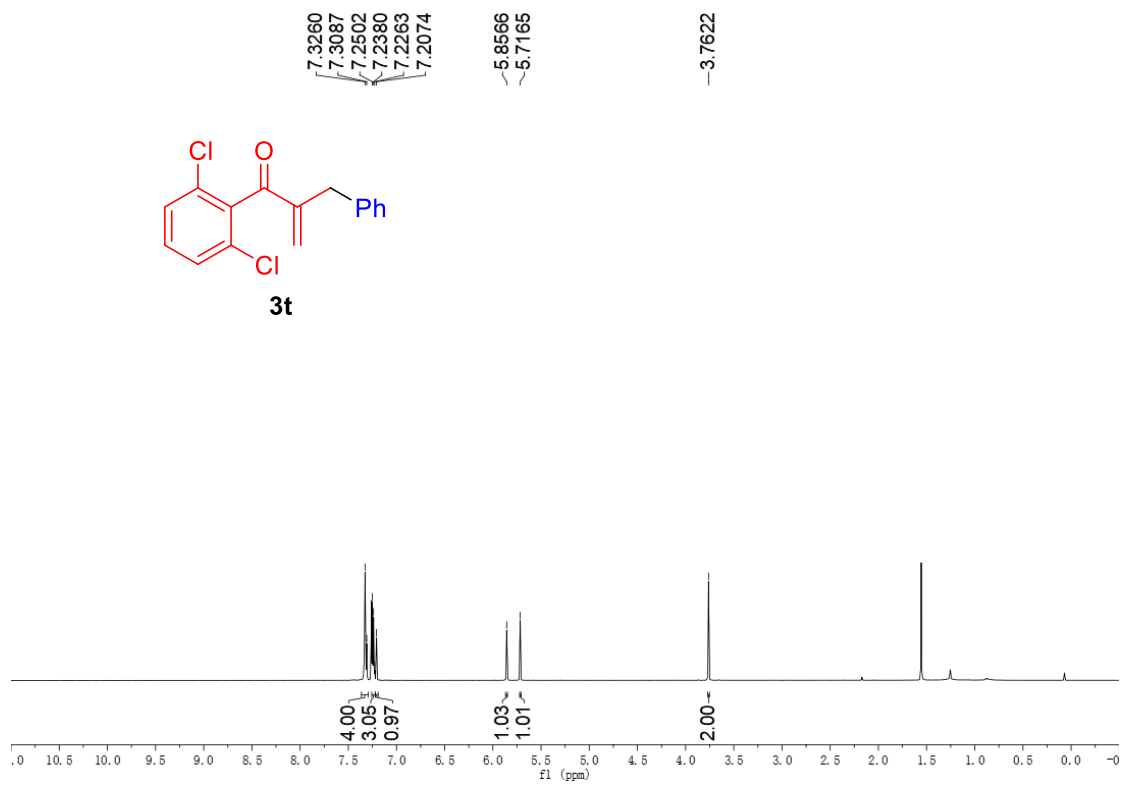
¹³C NMR spectrum of **3s** (151 MHz, CDCl₃)

183.5013
151.9639
147.3133
147.0058
138.4056
129.1618
128.4661
126.3551
125.0216
119.8730
111.9002

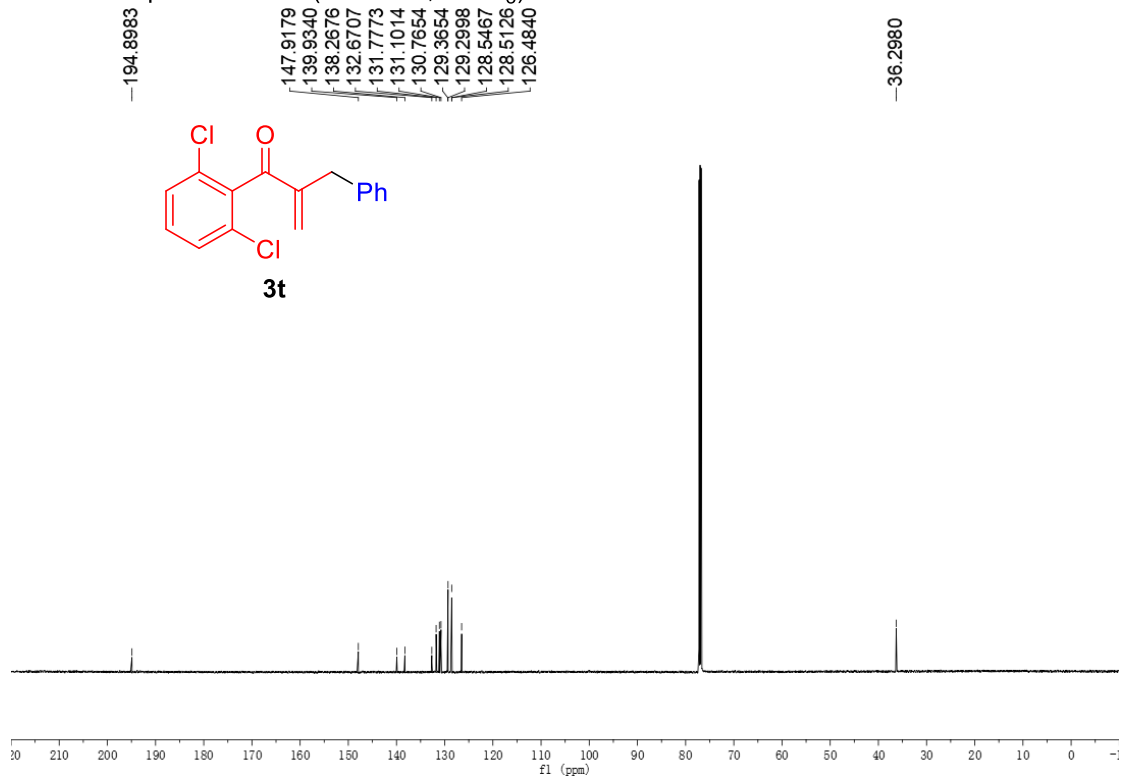
-38.2068



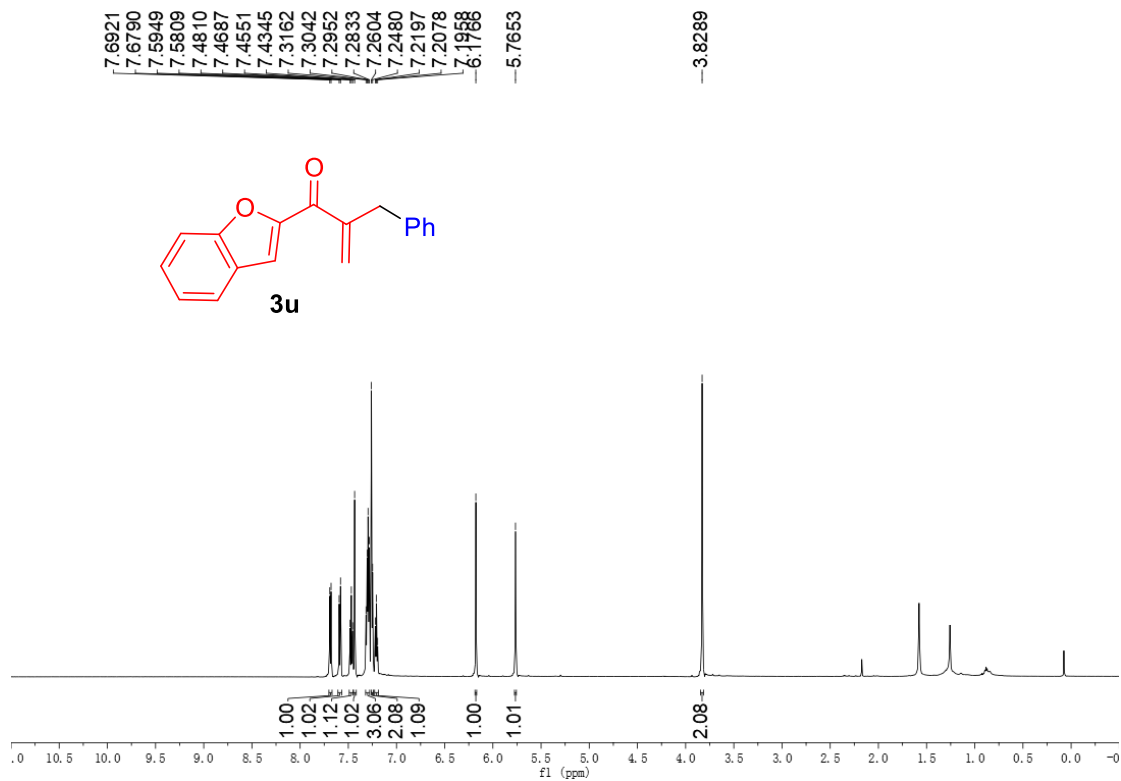
¹H NMR spectrum of **3t** (600 MHz, CDCl₃)



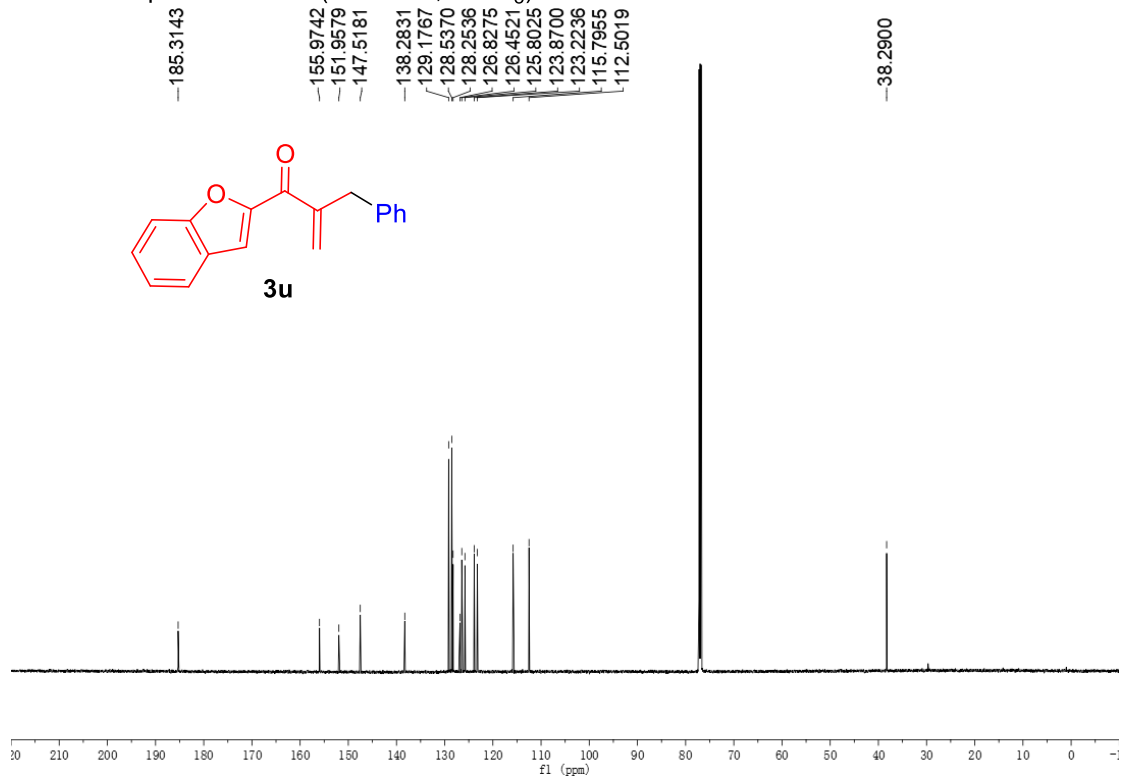
¹³C NMR spectrum of **3t** (151 MHz, CDCl₃)



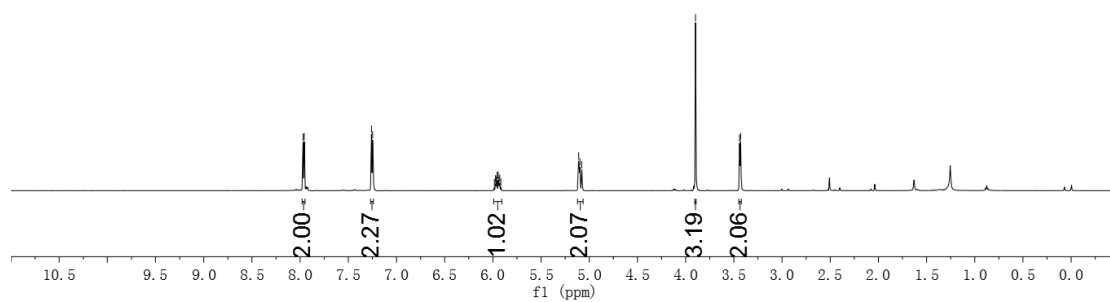
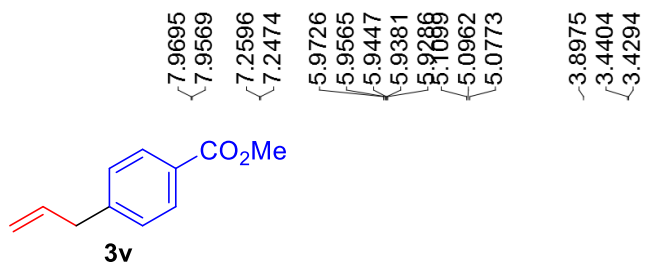
¹H NMR spectrum of **3u** (600 MHz, CDCl₃)



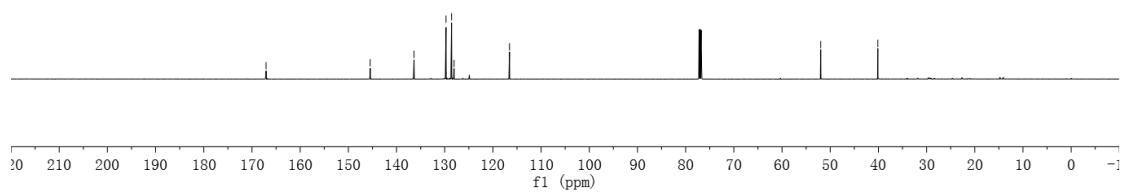
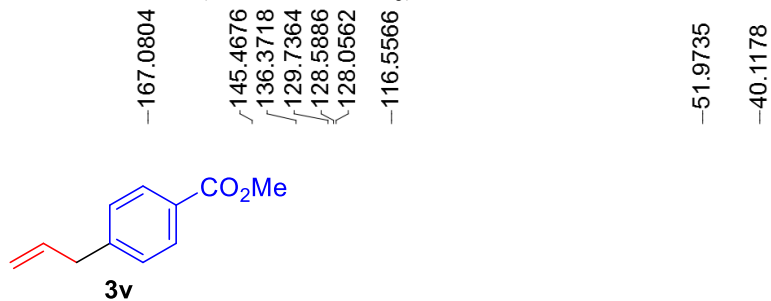
¹³C NMR spectrum of **3u** (151 MHz, CDCl₃)



^1H NMR spectrum of **3v** (600 MHz, CDCl_3)

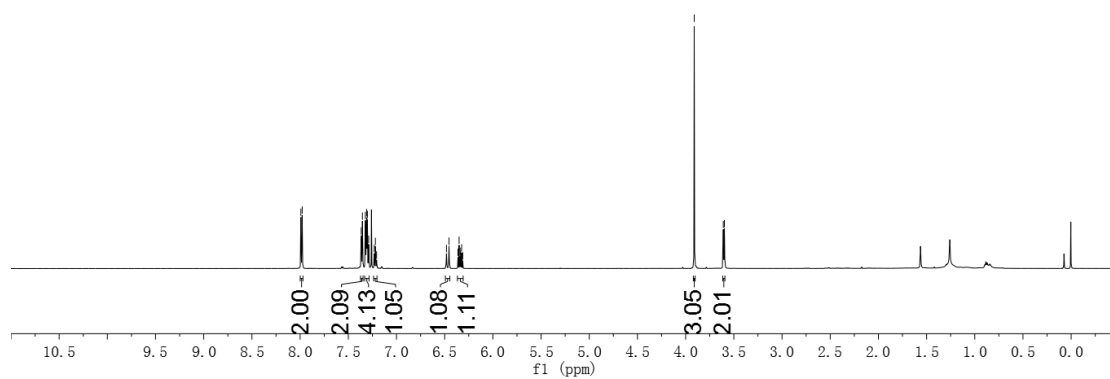
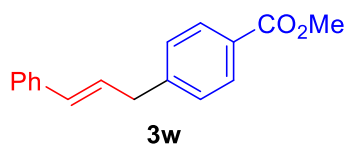


^{13}C NMR spectrum of **3v** (151 MHz, CDCl_3)



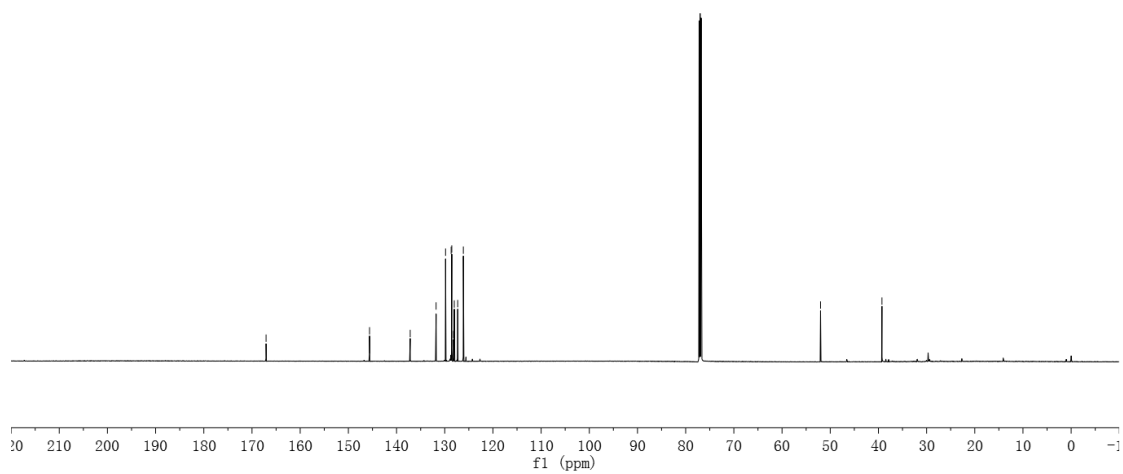
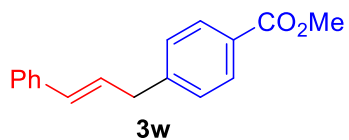
¹H NMR spectrum of **3w** (600 MHz, CDCl₃)

7.9922
7.9785
7.3230
7.3094
6.4818
6.4553
6.3621
6.3507
6.3392
6.3359
6.3244
6.3130
3.9109
3.6091
3.5978

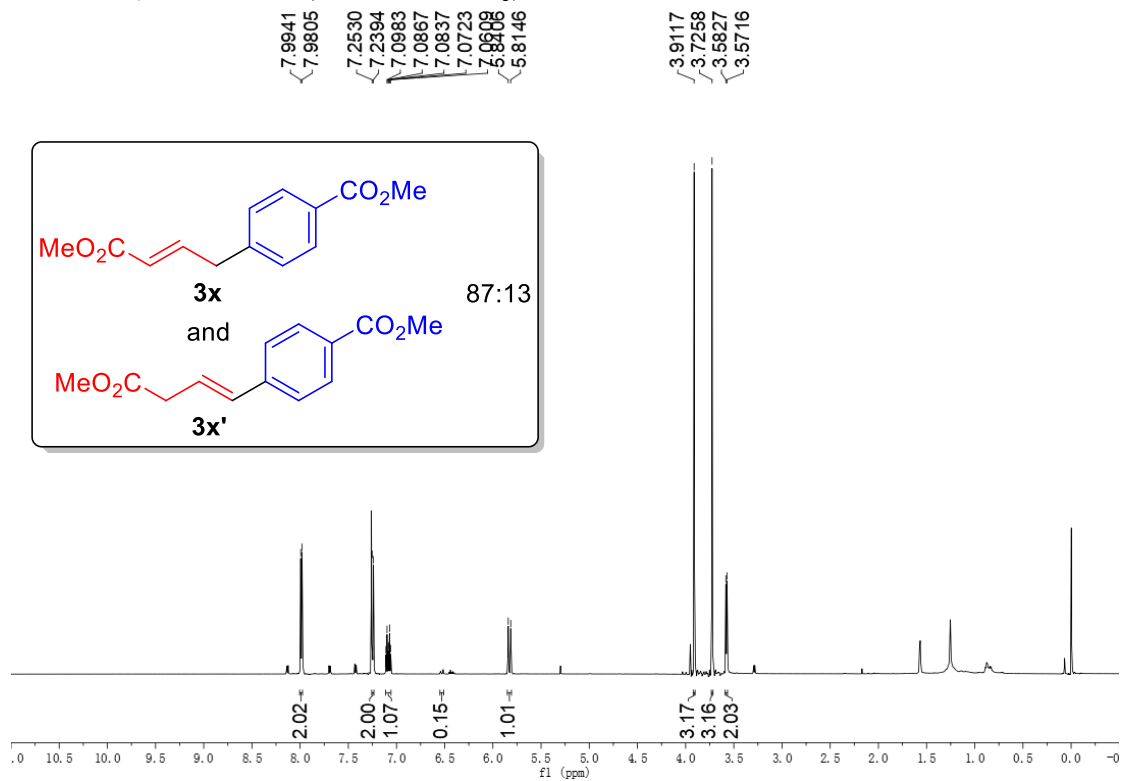


¹³C NMR spectrum of **3w** (151 MHz, CDCl₃)

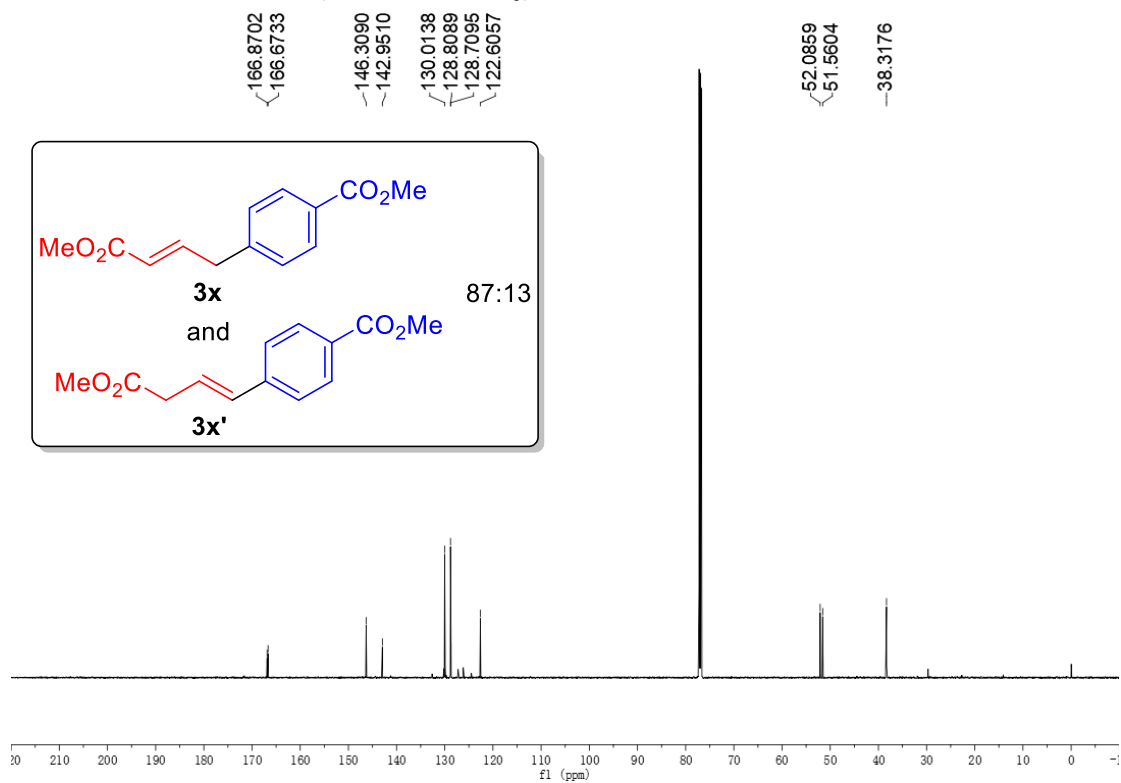
167.0761
145.6064
137.1685
131.7958
129.8296
128.6772
128.5405
128.1850
128.0207
127.3092
126.1406
-52.0138
-39.2654



¹H NMR spectrum of **3x** (600 MHz, CDCl₃)

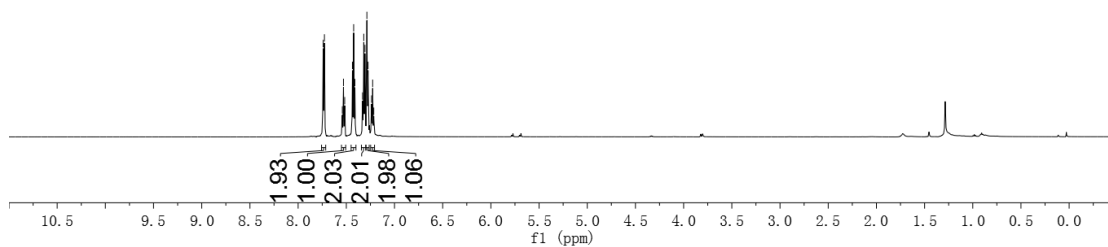
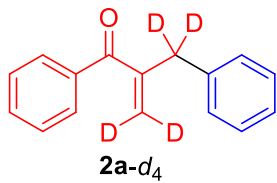


¹³C NMR spectrum of **3x** (151 MHz, CDCl₃)



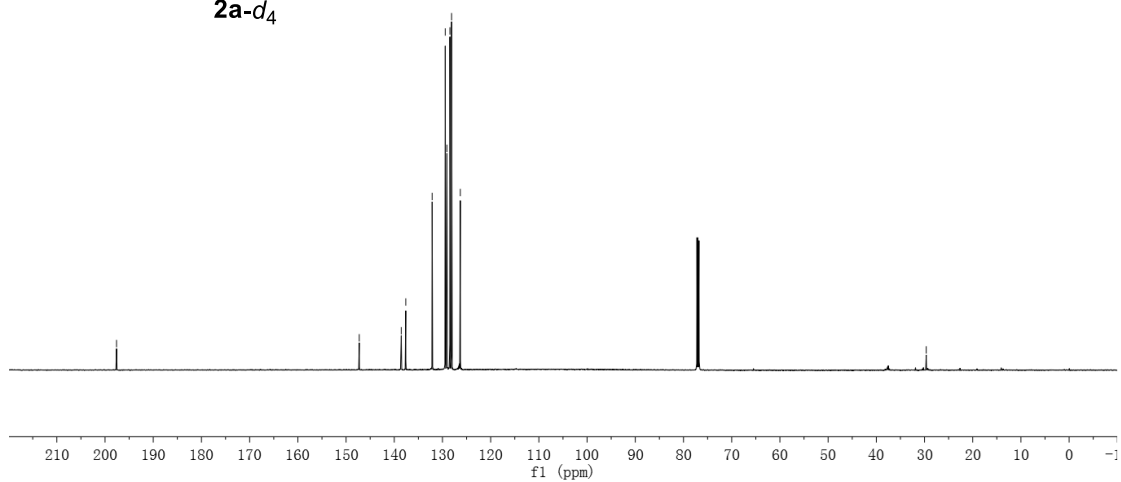
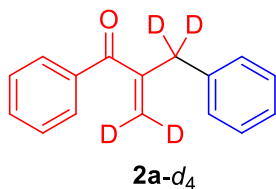
¹H NMR spectrum of **2a-d₄** (600 MHz, CDCl₃)

7.7403
7.7272
7.5413
7.5296
7.5167
7.4366
7.4239
7.4112
7.3316
7.3188
7.3064
7.2865
7.2743
7.2384
7.2267
7.2150



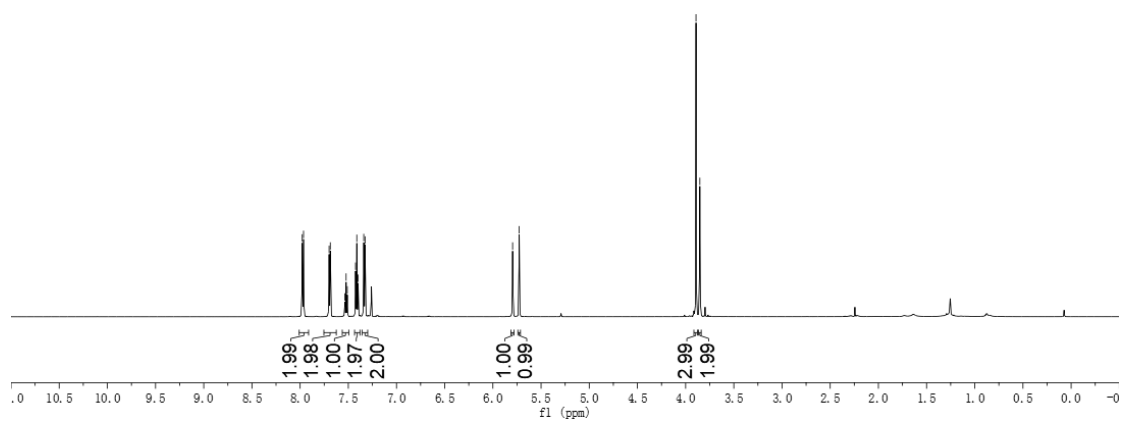
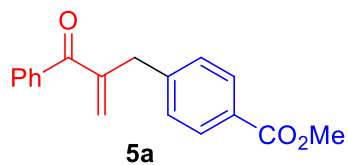
¹³C NMR spectrum of **2a-d₄** (151 MHz, CDCl₃)

197.6159
147.2640
138.5523
137.6161
132.1441
129.4285
129.0777
128.4584
128.1107
126.2982
29.6455



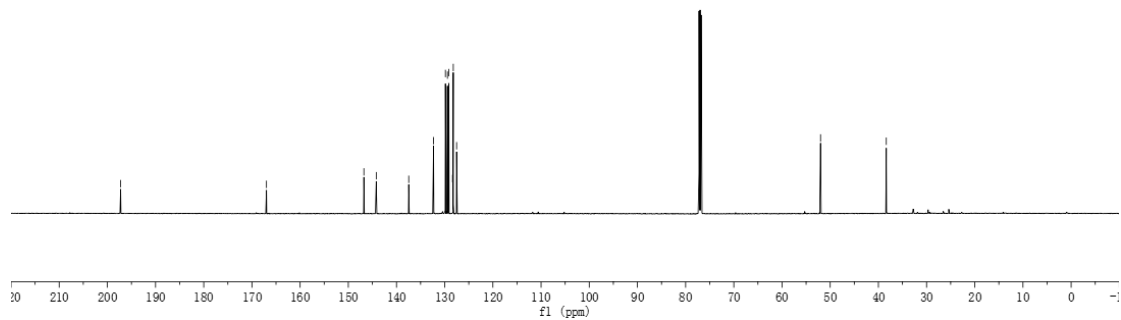
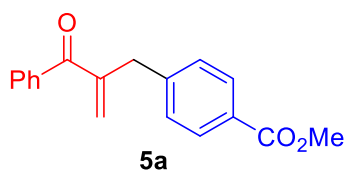
¹H NMR spectrum of **5a** (600 MHz, CDCl₃)

7.9782
7.9646
7.6992
7.6869
7.5352
7.5228
7.5105
7.4249
7.4121
7.3993
7.3398
7.3262
5.7953
5.7266
3.8922
3.8524

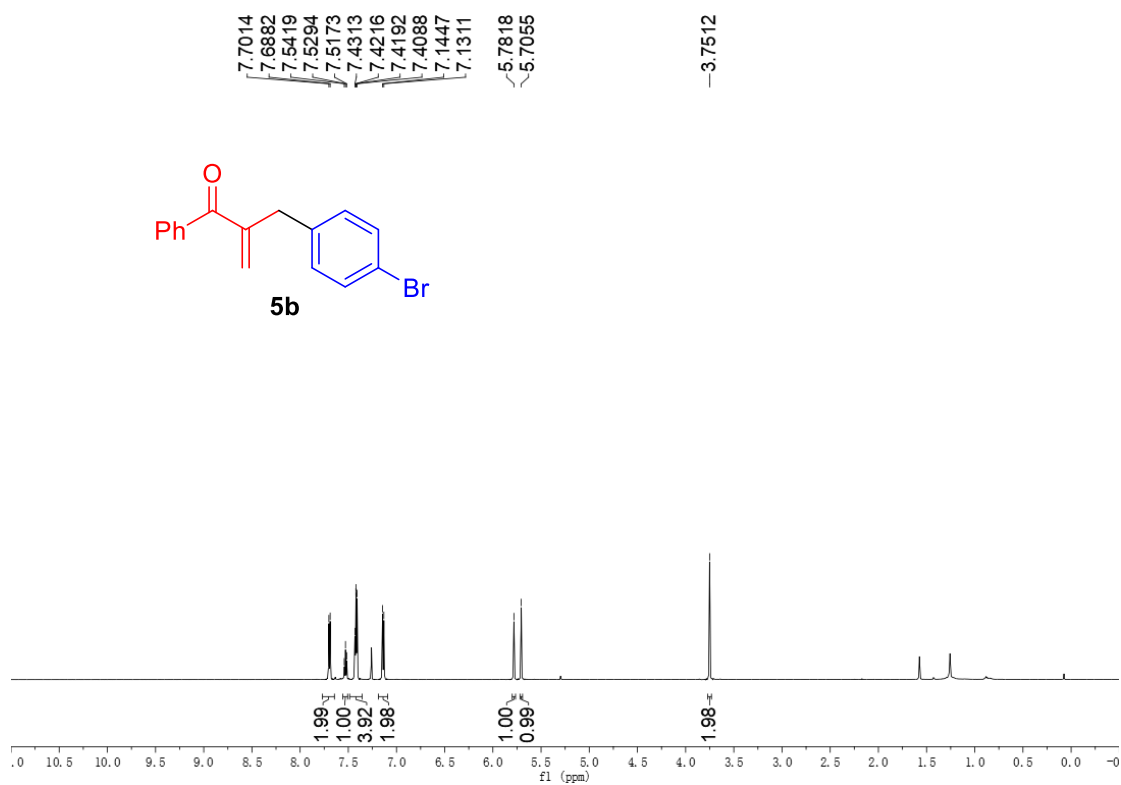


¹³C NMR spectrum of **5a** (151 MHz, CDCl₃)

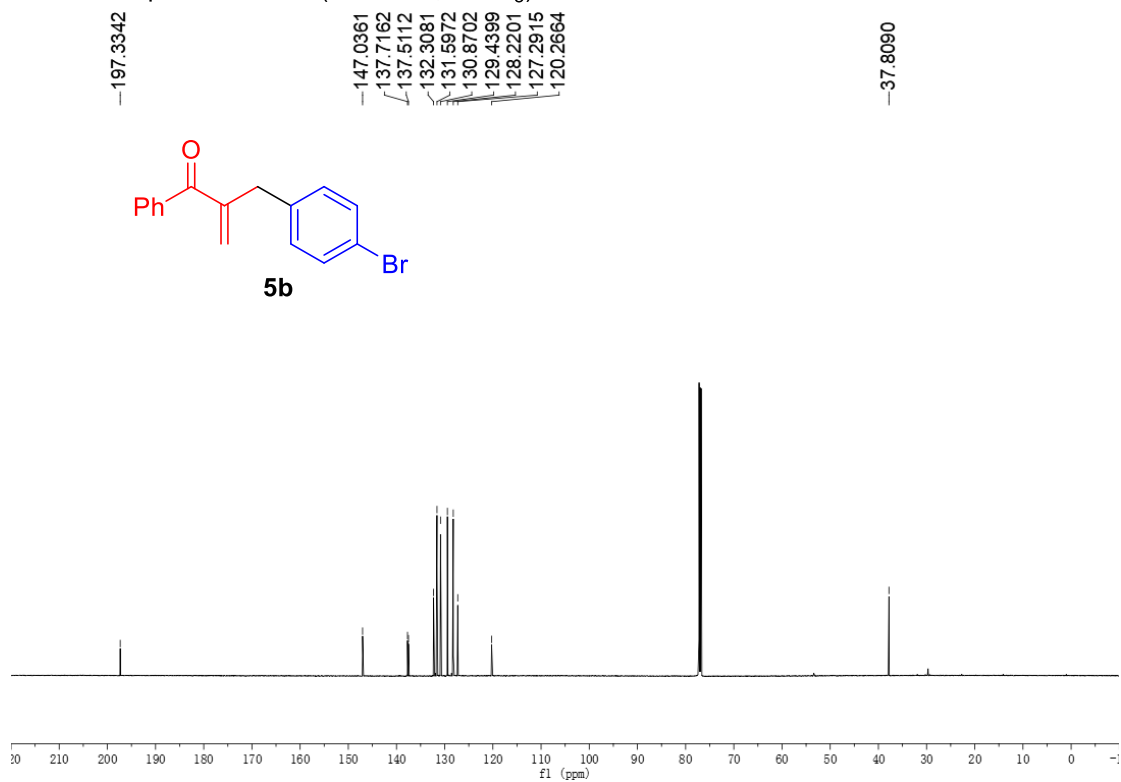
197.2451
166.9812
146.7670
144.1973
137.4613
132.3129
129.8526
129.4286
129.1383
128.3685
128.2175
127.5259
52.0029
38.3603



¹H NMR spectrum of **5b** (600 MHz, CDCl₃)

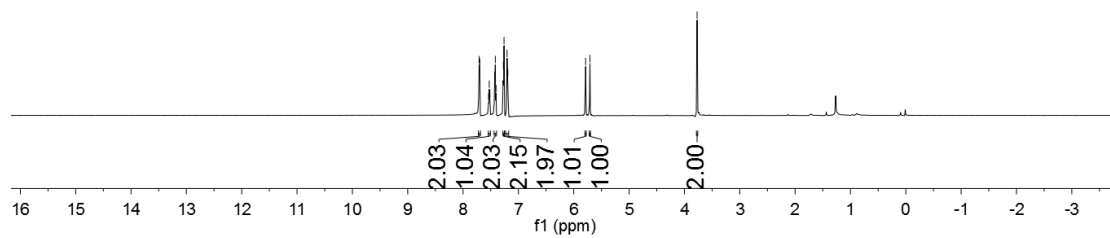
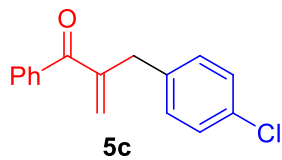


¹³C NMR spectrum of **5b** (151 MHz, CDCl₃)



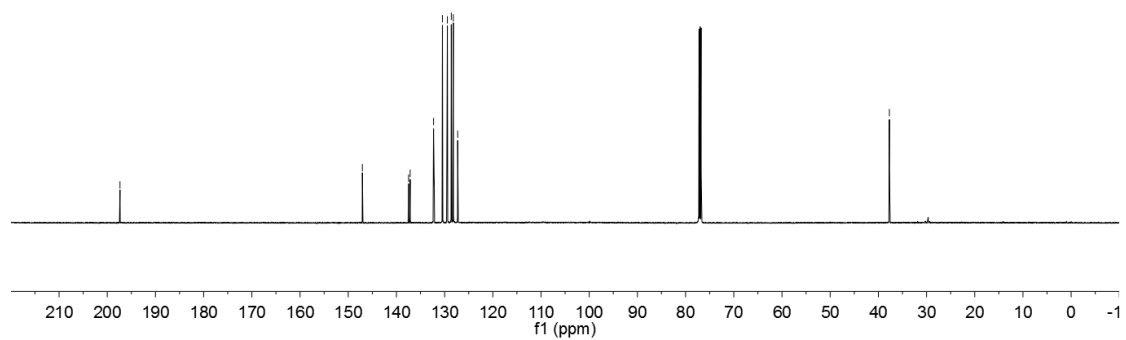
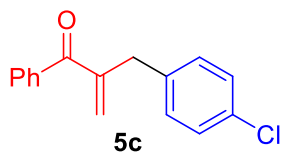
¹H NMR spectrum of **5c** (600 MHz, CDCl₃)

7.7097
7.6977
7.5389
7.5270
7.5146
7.4295
7.4176
7.4053
7.2716
7.2596
7.2039
7.1913
5.7855
5.7074
-3.7713



¹³C NMR spectrum of **5c** (151 MHz, CDCl₃)

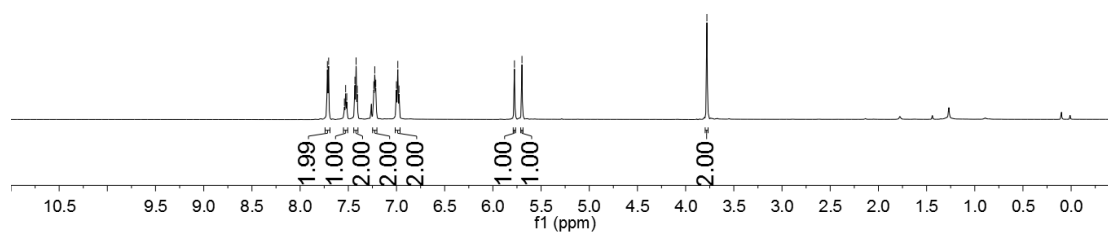
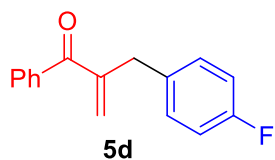
197.3594
147.0684
137.4812
137.1495
132.2988
132.1527
130.4565
129.4272
128.6171
128.2003
127.2894
37.7182



¹H NMR spectrum of **5d** (600 MHz, CDCl₃)

7.7171
7.7041
7.5407
7.5280
7.5162
7.4312
7.4192
7.4074
7.2353
7.2257
7.2151
7.0006
6.9863
6.9741
5.7762
5.6980

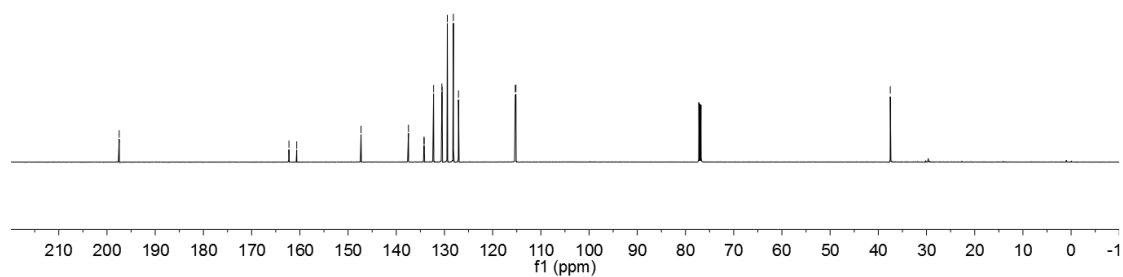
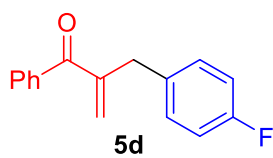
-3.7794



¹³C NMR spectrum of **5d** (151 MHz, CDCl₃)

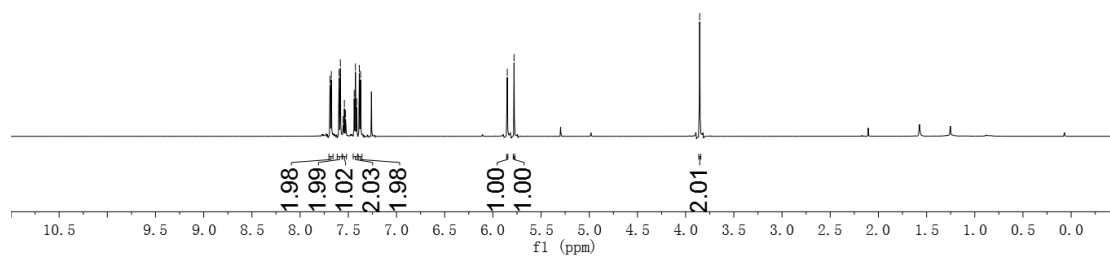
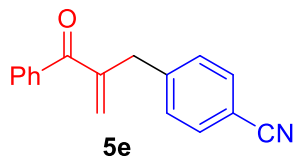
197.4732
162.2629
160.6448
132.2561
130.5299
130.4775
129.3977
128.1493
127.0889
115.3137
115.1729

-37.5117



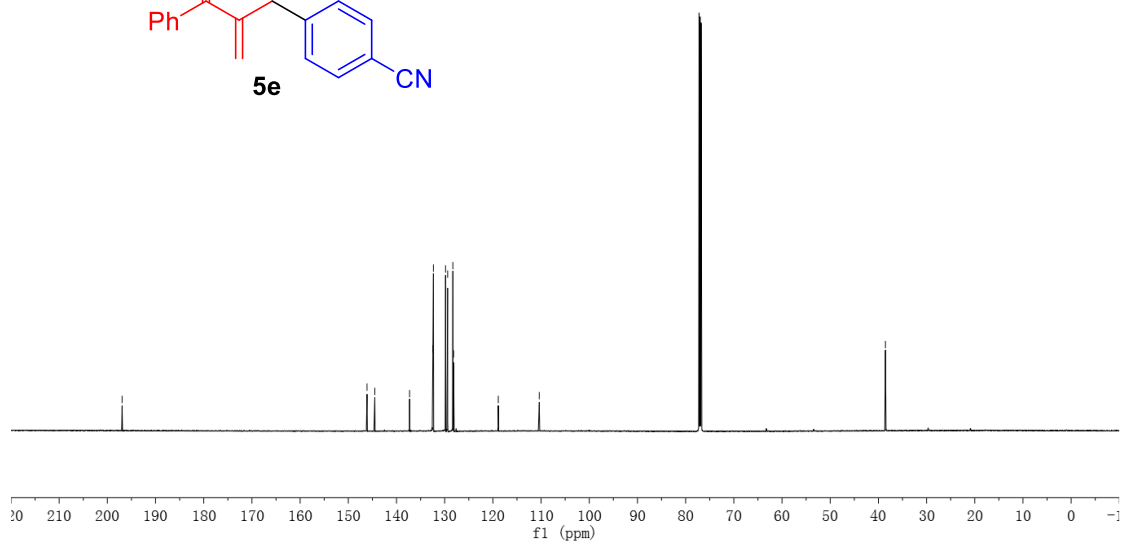
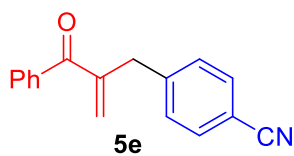
¹H NMR spectrum of **5e** (600 MHz, CDCl₃)

7.6896
7.6763
7.5957
7.5822
7.5530
7.5408
7.5285
7.4389
7.4262
7.4134
7.3858
7.3723
5.8508
5.7803
3.8542

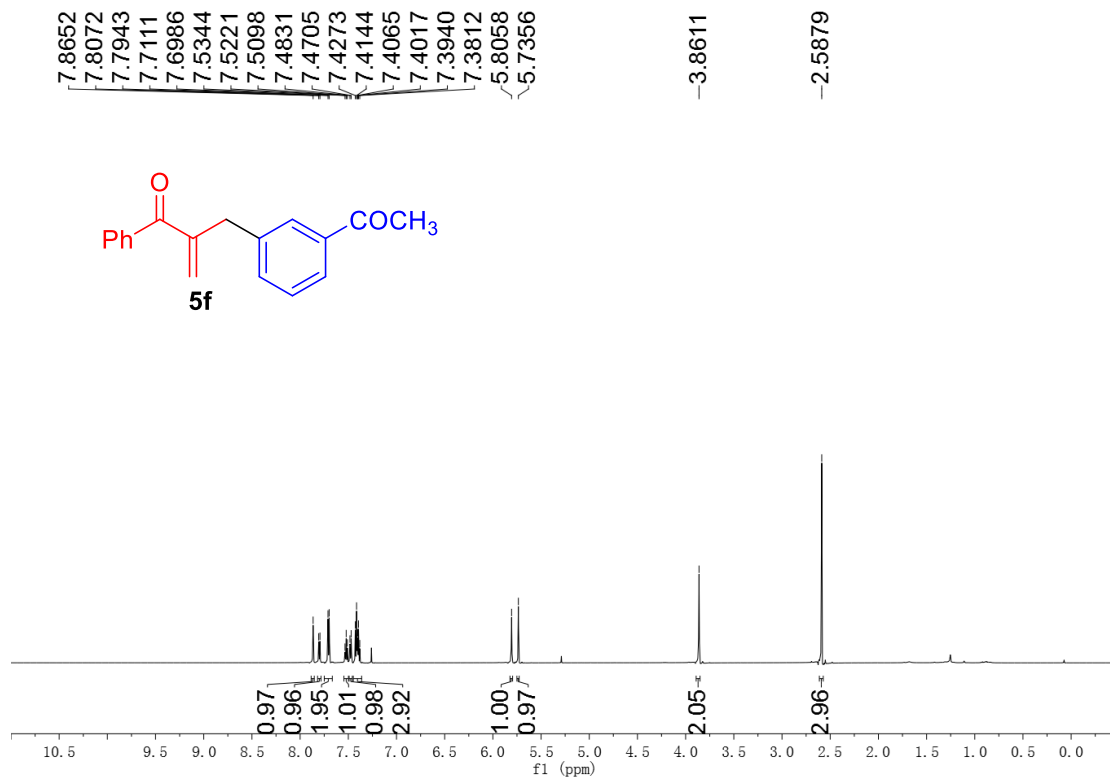


¹³C NMR spectrum of **5e** (151 MHz, CDCl₃)

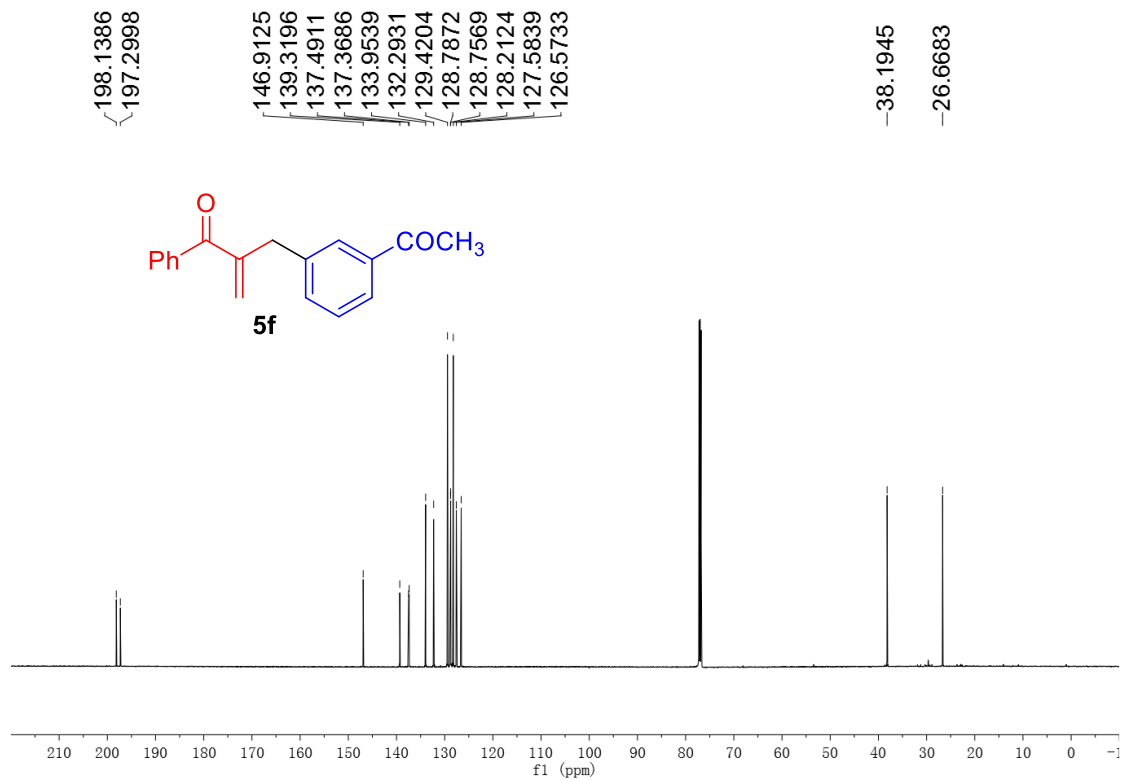
196.9402
146.1181
144.4998
137.2829
132.4612
132.3416
129.8202
129.3900
128.3000
128.1591
118.8795
110.3756
38.5368



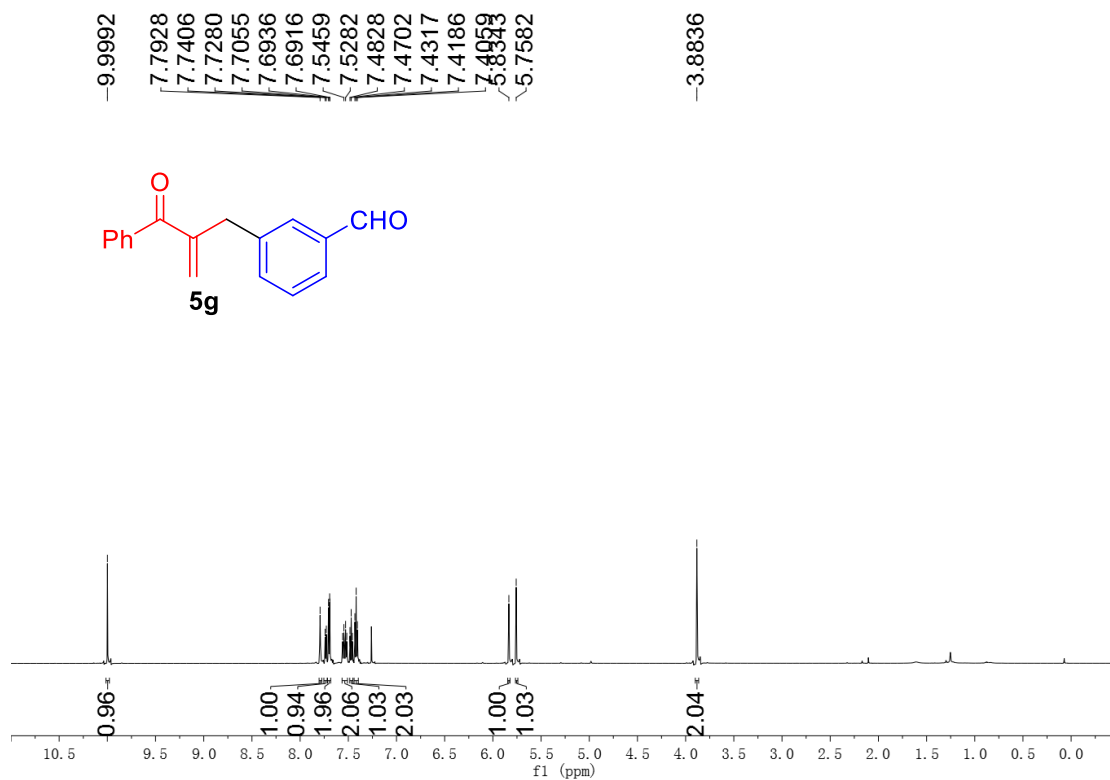
¹H NMR spectrum of **5f** (600 MHz, CDCl₃)



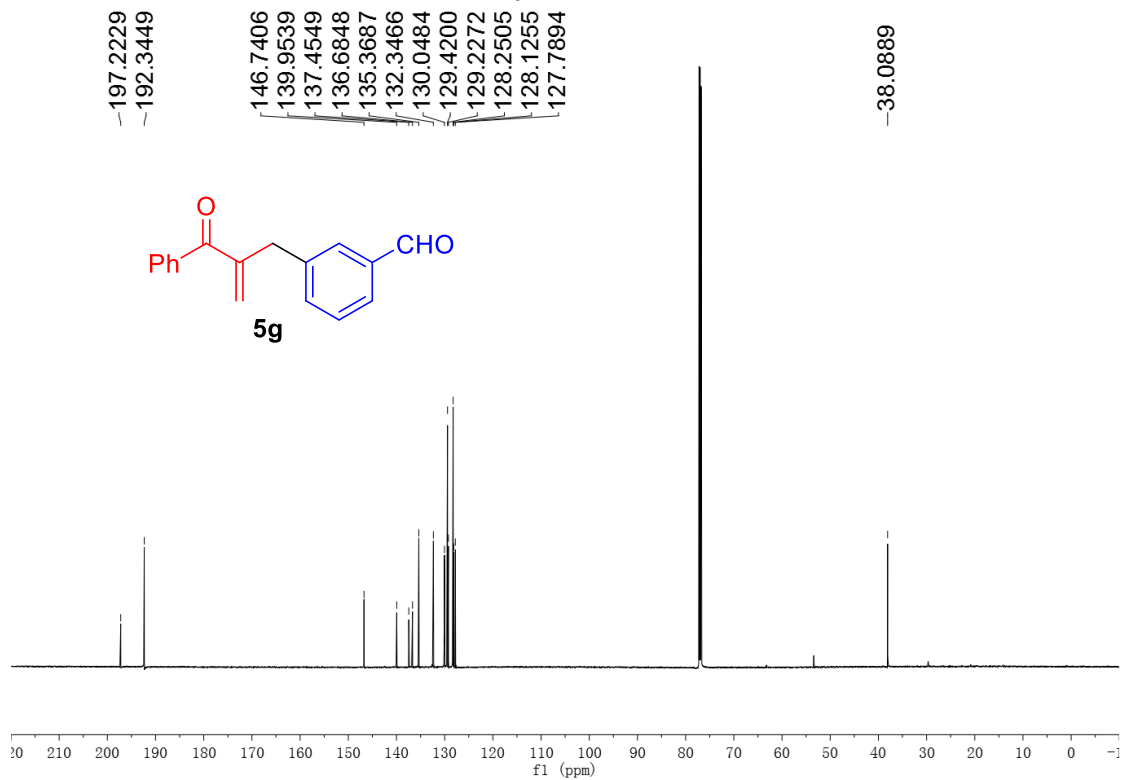
¹³C NMR spectrum of **5f** (151 MHz, CDCl₃)



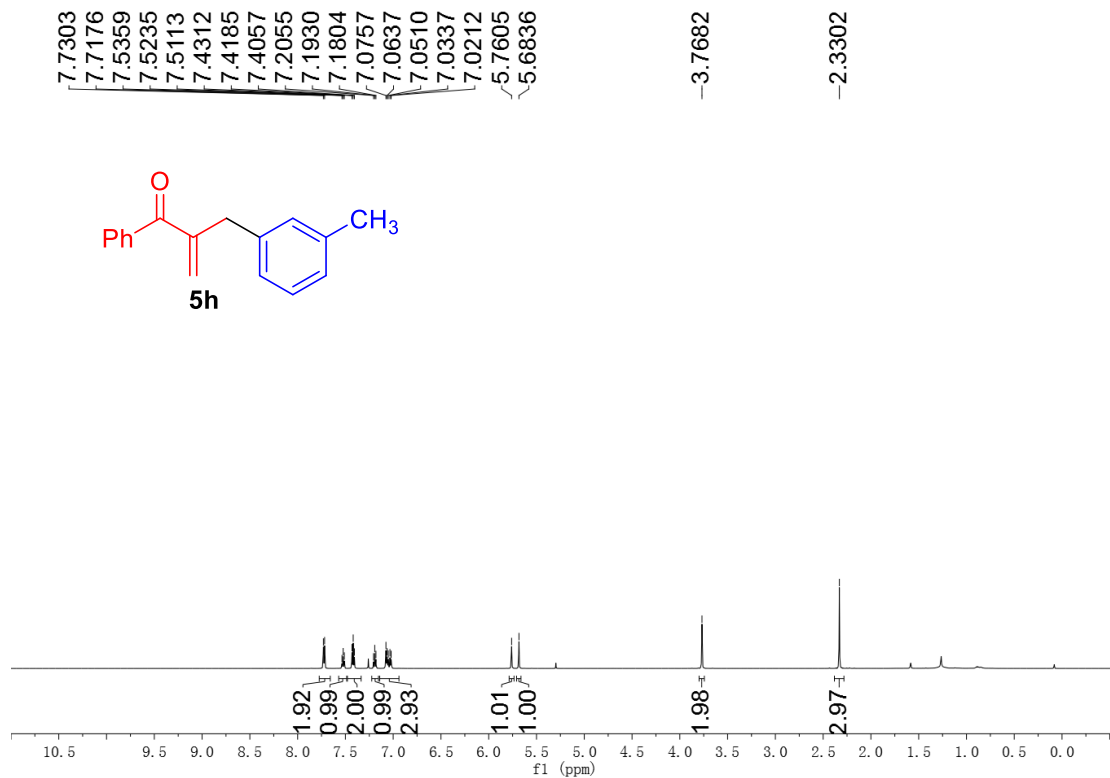
¹H NMR spectrum of **5g** (600 MHz, CDCl₃)



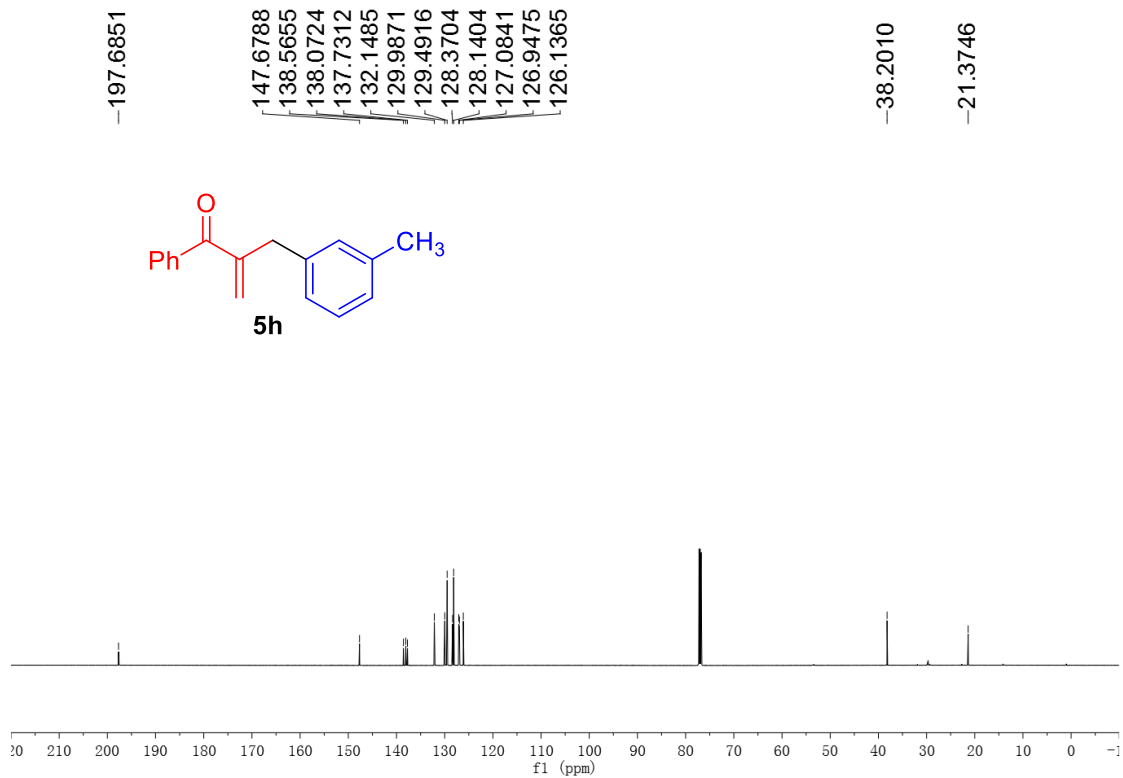
¹³C NMR spectrum of **5g** (151 MHz, CDCl₃)



¹H NMR spectrum of **5h** (600 MHz, CDCl₃)



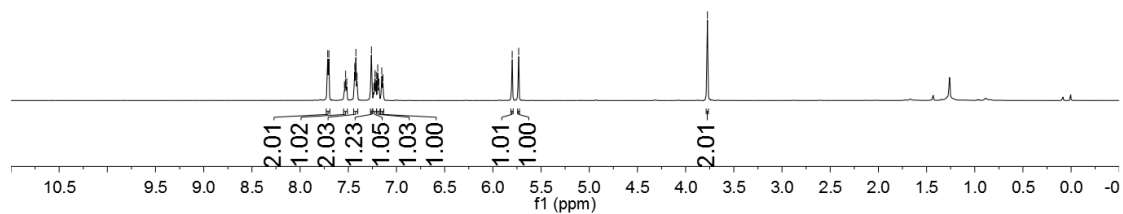
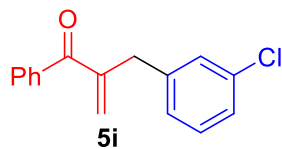
¹³C NMR spectrum of **5h** (151 MHz, CDCl₃)



¹H NMR spectrum of **5i** (600 MHz, CDCl₃)

7.7137
7.7013
7.5403
7.5283
7.5162
7.4331
7.4214
7.4096
7.2604
7.2352
7.2229
7.2102
7.1947
7.1819
7.1527
7.1408
5.7997
5.7323

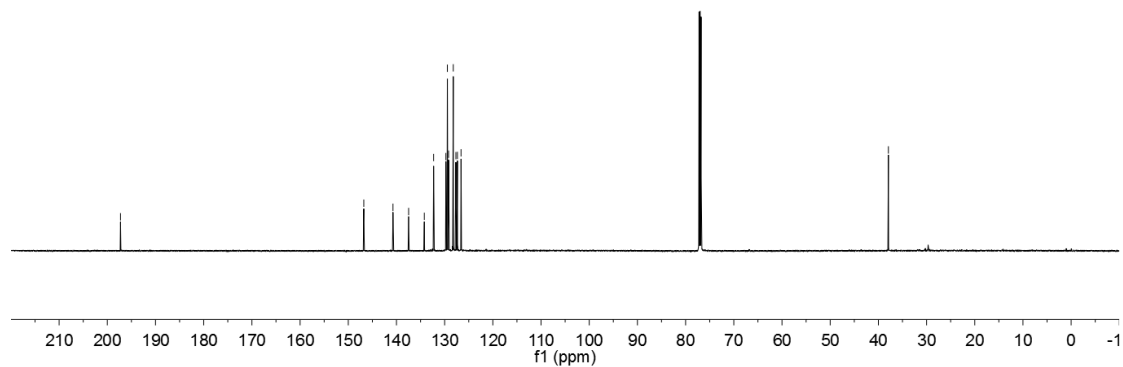
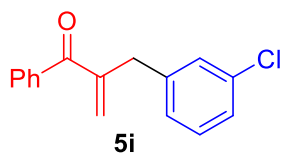
-3.7738



¹³C NMR spectrum of **5i** (151 MHz, CDCl₃)

197.2621
146.7787
140.7254
137.4759
134.2214
132.2934
129.7456
129.4392
129.1856
128.2152
127.6706
127.3499
126.5993

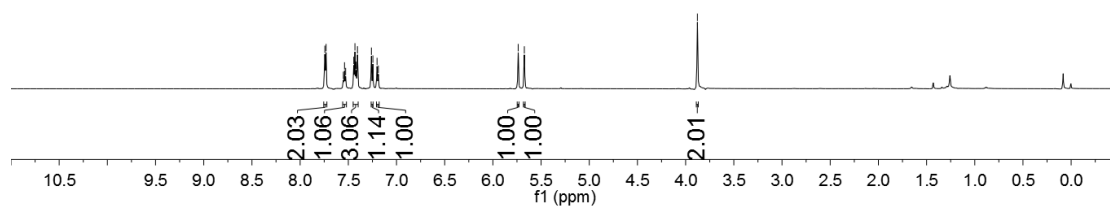
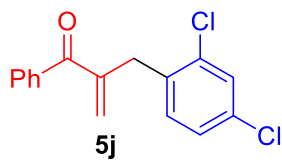
-37.9235



¹H NMR spectrum of **5j** (600 MHz, CDCl₃)

7.7435
7.7307
7.5513
7.5394
7.5277
7.4434
7.4313
7.4188
7.4051
7.2604
7.2451
7.2019
7.1883
5.7373
5.6736

3.8779

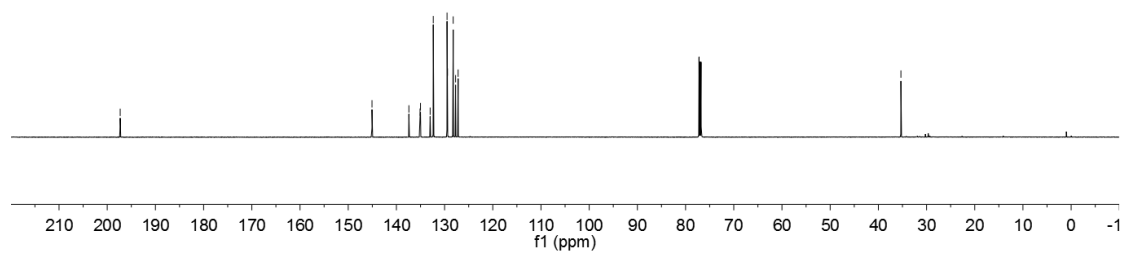
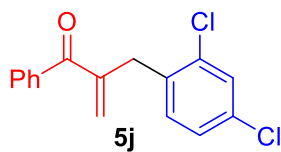


¹³C NMR spectrum of **5j** (151 MHz, CDCl₃)

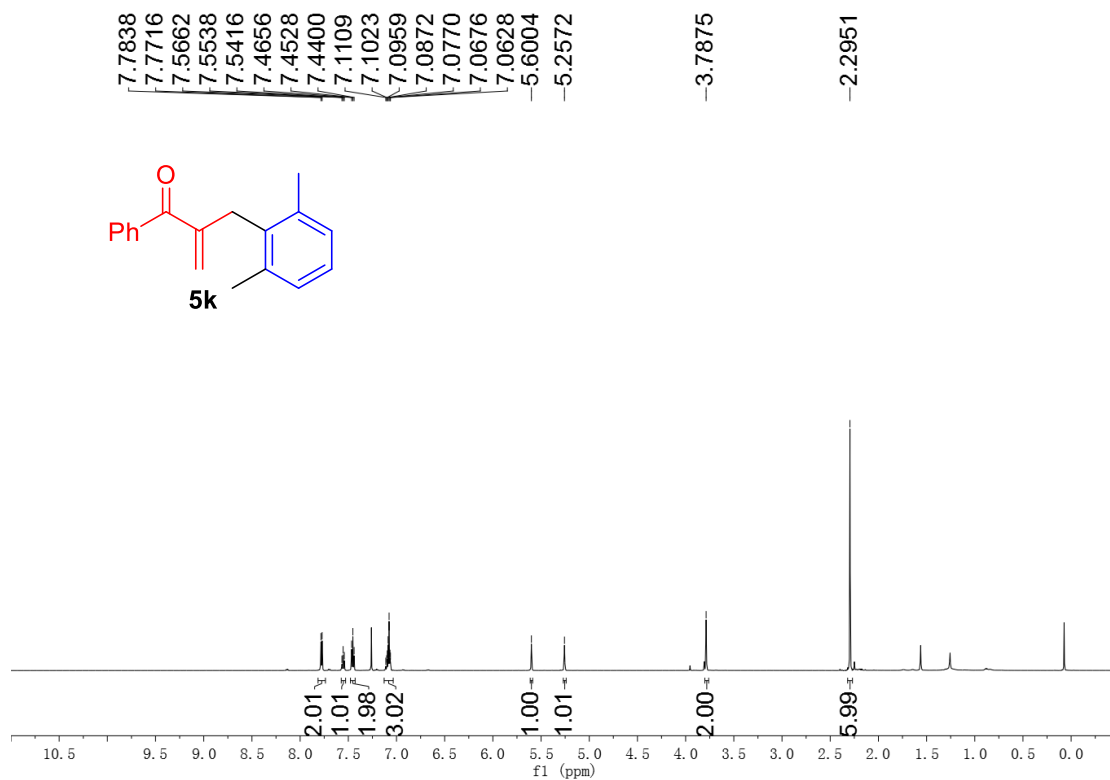
197.3103

145.0639
137.4087
135.1002
135.0037
132.9999
132.3524
129.4945
129.3982
128.2176
127.7201
127.1945

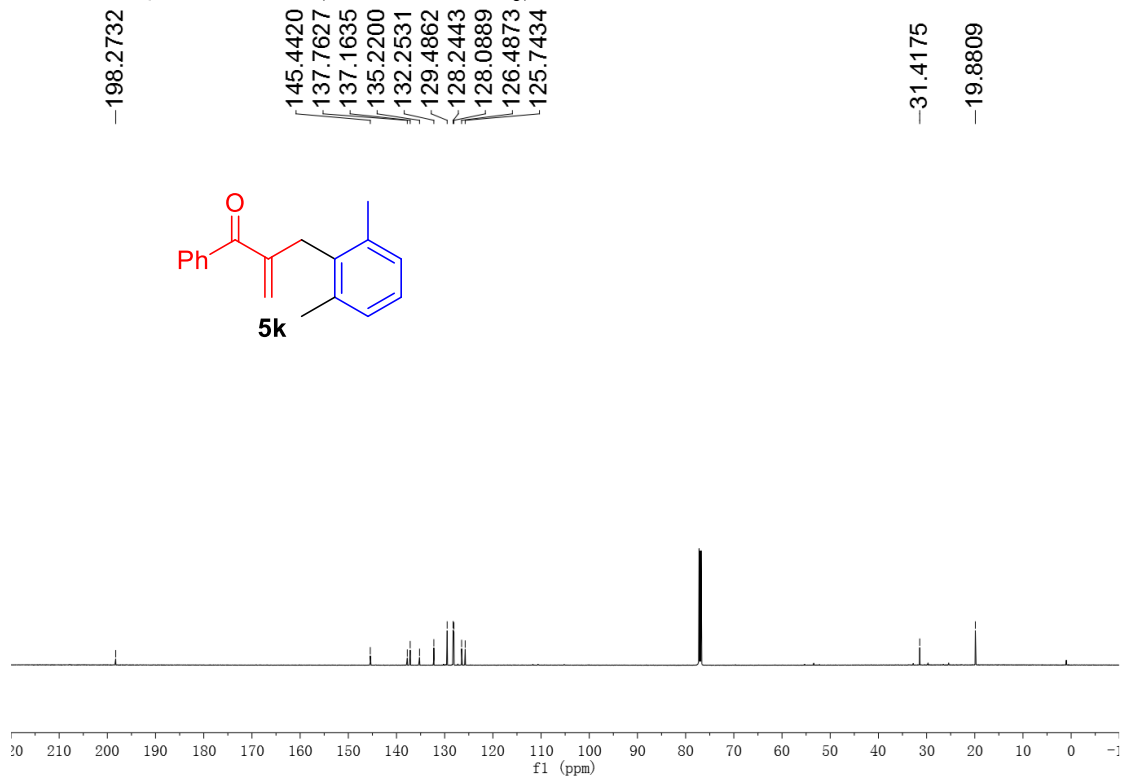
35.3191



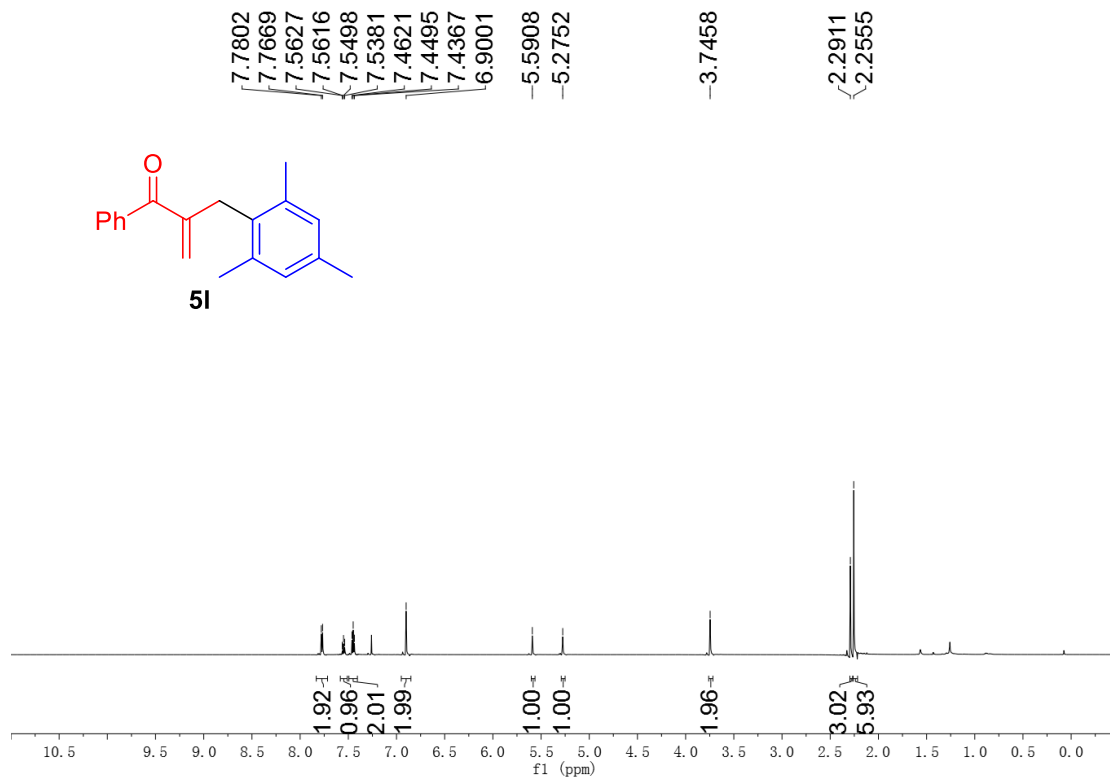
¹H NMR spectrum of **5k** (600 MHz, CDCl₃)



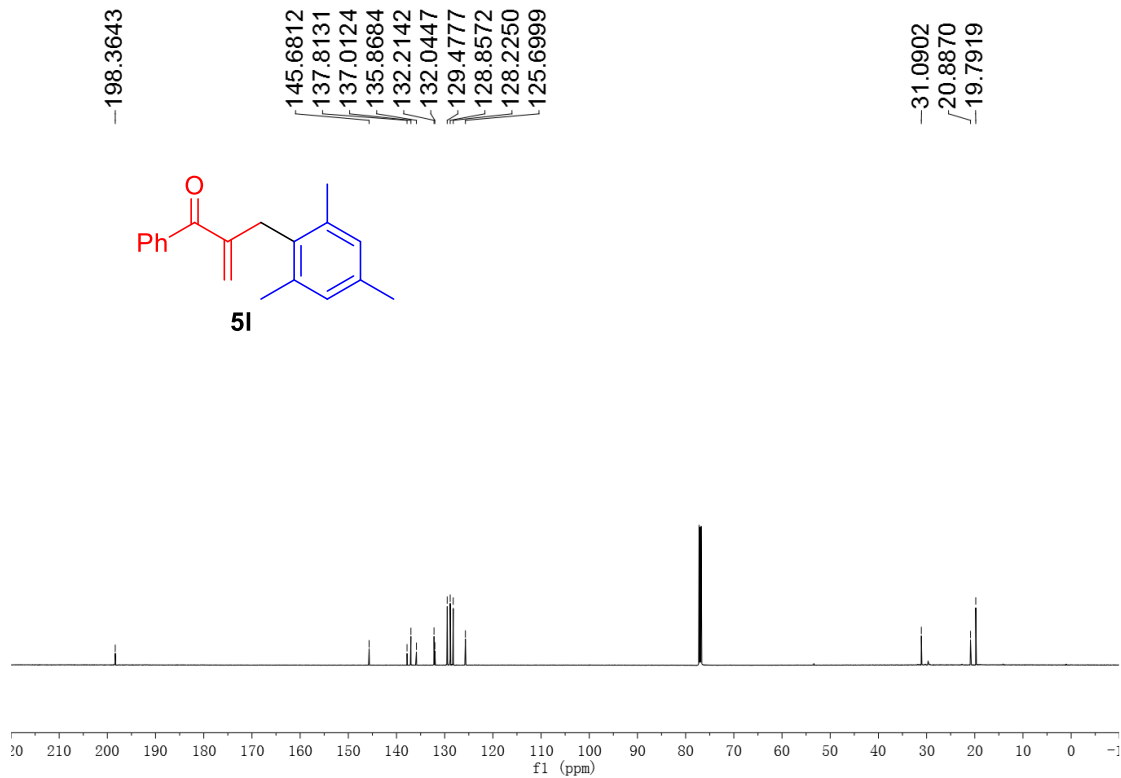
¹³C NMR spectrum of **5k** (151 MHz, CDCl₃)



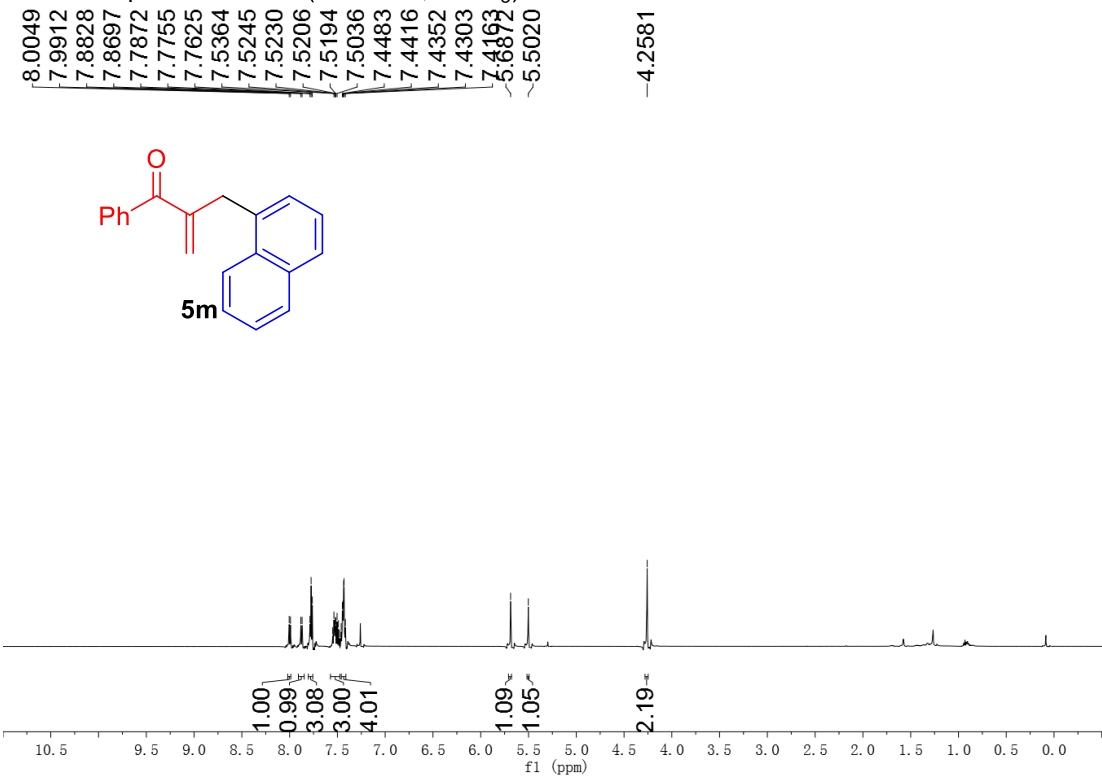
¹H NMR spectrum of **5I** (600 MHz, CDCl₃)



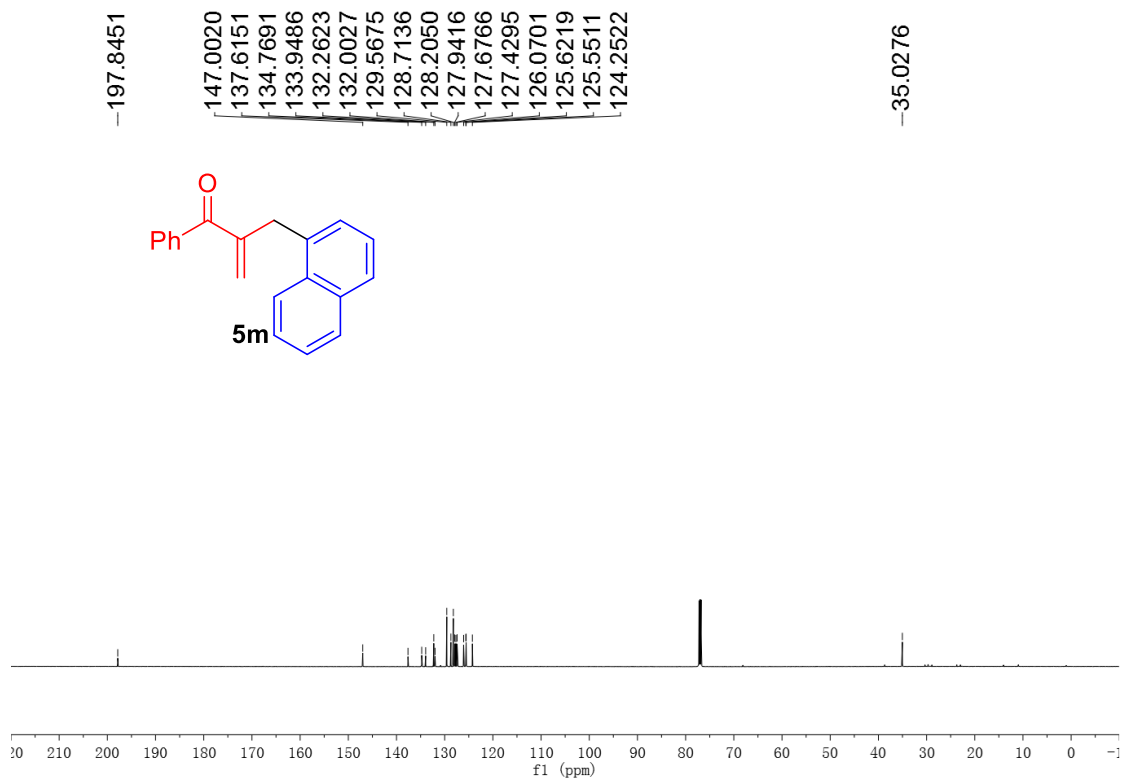
¹³C NMR spectrum of **5I** (151 MHz, CDCl₃)



¹H NMR spectrum of **5m** (600 MHz, CDCl₃)

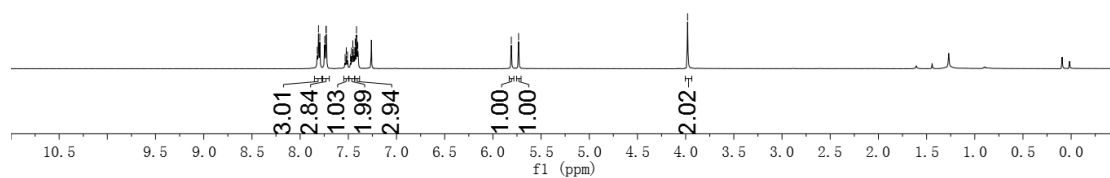
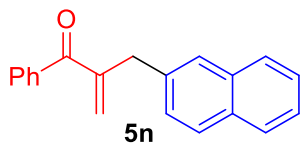


¹³C NMR spectrum of **5m** (151 MHz, CDCl₃)



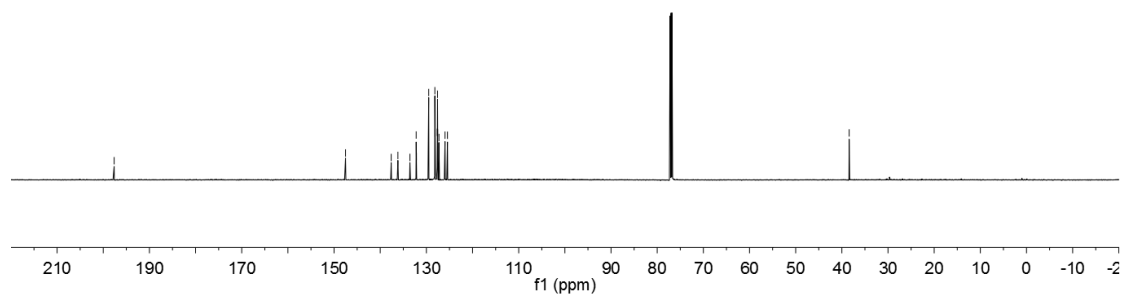
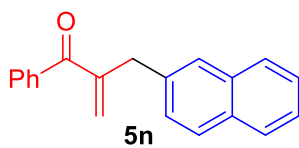
¹H NMR spectrum of **5n** (600 MHz, CDCl₃)

7.8219
7.8092
7.7957
7.7450
7.7313
7.7296
7.5333
7.5212
7.5089
7.4774
7.4663
7.4534
7.4399
7.4282
7.4150
7.4019
5.8089
5.7331
-3.9798



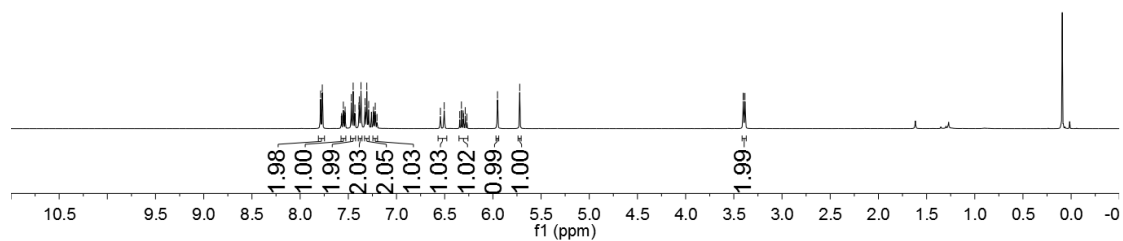
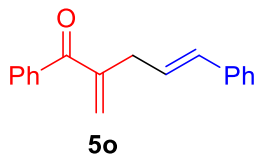
¹³C NMR spectrum of **5n** (151 MHz, CDCl₃)

197.6398
147.5164
137.6232
136.1696
133.5673
132.2145
132.1510
129.4909
128.1648
128.1247
127.6591
127.5858
127.5519
127.2737
125.9646
125.4367
-38.4175



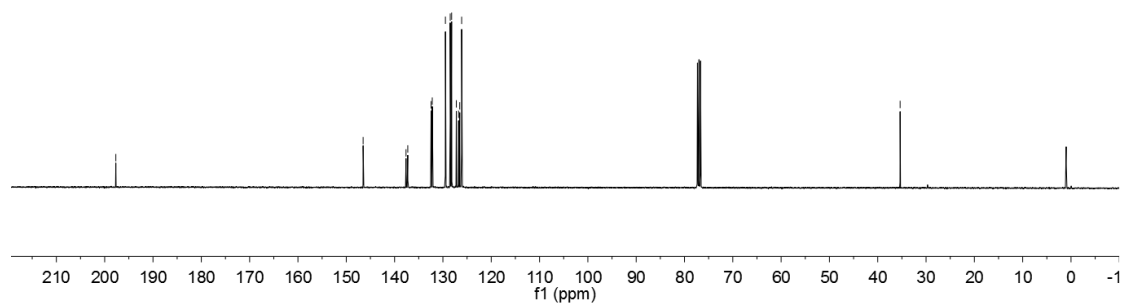
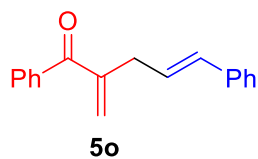
¹H NMR spectrum of **5o** (400 MHz, CDCl₃)

7.7876
7.7746
7.7700
7.7664
7.5700
7.5516
7.5468
7.5362
7.5330
7.5299
7.4692
7.4498
7.4353
7.4315
7.3899
7.3865
7.3684
7.3263
7.3216
7.3080
7.2886
7.2414
7.2385
7.2354
7.2254
7.2204
7.2023
6.5445
6.5049
6.3439
6.3263
6.3087
6.3046
6.2869
6.2692
5.9521
5.7215
3.4021
3.4006
3.3846



¹³C NMR spectrum of **5o** (101 MHz, CDCl₃)

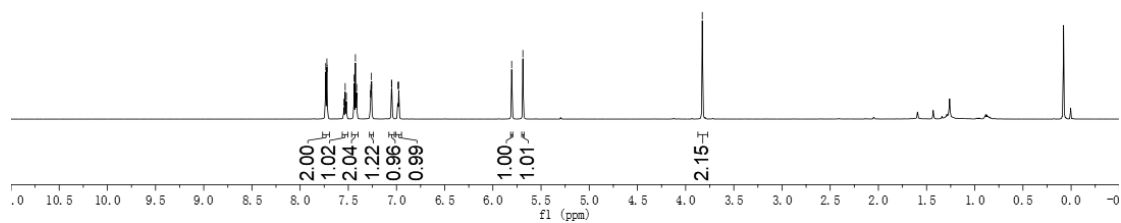
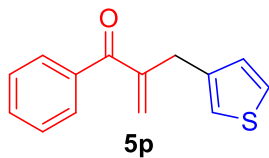
197.6915
146.5132
137.6334
137.2699
132.4420
132.2009
129.4788
128.4850
128.1942
127.2220
126.7597
126.5145
126.1182
35.3708



¹H NMR spectrum of **5p** (600 MHz, CDCl₃)

7.7342
7.7223
7.7206
7.5447
7.5324
7.5200
7.4391
7.4262
7.4134
7.2704
7.2653
7.2601
7.2577
7.0516
7.0500
7.0487
6.9850
6.9769
5.8048
5.6888

—3.8270



¹³C NMR spectrum of **5p** (151 MHz, CDCl₃)

—197.5642
147.0068
138.7770
137.6322
132.2264
129.4675
128.4919
128.1831
126.7857
125.6632
121.9283

—32.8396

