Iridium-catalyzed regioselective decarboxylative allylation of *β*-ketoacids: efficient construction of *γ*, *δ*-unsaturated ketones

Shu-Jie Chen, Guo-Ping Lu and Chun Cai*

School of Chemical Engineering, Nanjing University of Science and Technology, Nanjing 210094, People's Republic of China

c.cai@mail.njust.edu.cn

Supporting information

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General information

All air-sensitive manipulations were carried out under the inert gas atmosphere using standard Schlenk techniques. Glassware was oven or flame dried immediately prior to use. 1,2-Dichloroethane (DME) was distilled from P_2O_5 under nitrogen and kept with 4A molecular sieves. ^{1}H NMR, ^{19}F NMR, and ^{13}C NMR spectra were recorded on an AVANCE 500 Bruker spectrometer operating at 500 MHz, 470 MHz, and 125 MHz in $CDCl_3$, respectively, and chemical shifts were reported in ppm relative to the center of the singlet at 7.26 ppm for $CDCl_3$ or downfield from internal tetramethylsilane). GC/MS were performed on an ISQ Trace 1300 (electrospray ionization: EI). GC analysis were performed on an Agilent 7890A instrument (Column: Agilent 19091J-413: 30 m × 320 μm × 0.25 μm, carrier gas: N_2, FID detector. Elemental analyses were performed on a Yanagimoto MT3CHN recorder. Preparative high performance liquid chromatography was performed on the column XDB-C18 (9.6 mm × 250 mm) with methanol/water as eluent.

Monosubstituted allylic alcohols were prepared by the reaction of the corresponding aldehyde with vinyl magnesium bromide.[1] Allylic alcohols **2n**[2] and **2p** were prepared by a 1,2-reduction of the corresponding *α*,*β*-unsaturated ketones. *β*-Ketoacids **1g**-**1h** were prepared from the corresponding aryl ketones.[3] *β*-Ketoacids **1i**-**1l** were prepared from the commercially available β-ketoesters.[4]

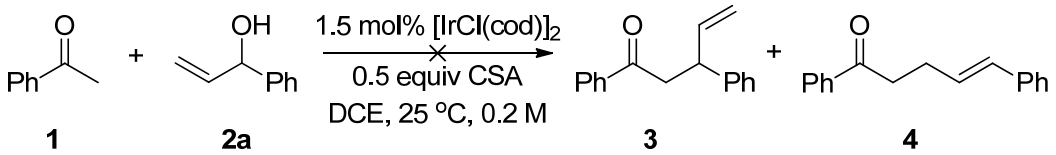
General procedure for the preparation of allylic alcohols 2n and 2p.

In an oven-dried round-bottom flask, a solution of the α,β-unsaturated ketone (2 mmol, 1 equiv) in 10 ml THF/MeOH (v/v=1/1) was stirred for 5 minutes at 0 ^{o}C. Then $NaBH_4$ (2.59 g, 2 mmol, 1.0 equiv) was added in one portion and the reaction mixture was maintained at this temperature for 1 h (monitored by TLC). Then the reaction was quenched with several mLs of 0.1 M HCl until no further hydrogen evolution was observed. The solution was then made basic using $NaHCO_3$ and extracted with diethyl ether (15 ml ×3). The combined organic phase was washed by a saturated solution of NaCl, dried over anhydrous $MgSO_4$, and evaporated to leave the crude product which was purified by column chromatography over silica gel.

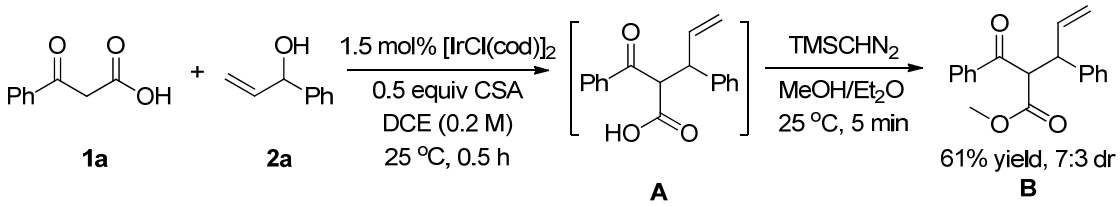
General procedure for the decarboxylative allylation of β-ketoacids with allylic alcohols.

A 10 ml schlenk tube was charged with $[Ir(cod)Cl]_2$ (3.0 mg, 0.0045 mmol, 0.015 equiv), (±)-10-Camphorsulfonic Acid (CSA) (35 mg, 0.15 mmol, 0.5 equiv) and β-ketoacids (0.39 mmol, 1.3 equiv). The tube was flushed with nitrogen for three times. Then 1.5 ml of dry 1,2-dichloroethane (DCE) and allylic alcohol (0.3 mmol, 1.0 equiv) were added by syringe and the reaction was stirred at 25 ^{o}C for 10 hours. After the indicated time, the solvent was removed by vacuum and the residue was analyzed by ^{1}H NMR spectroscopy to determine the ratio of branched to linear regioisomers. The solvent was then removed again, and the residue was purified by flash column chromatography on silica gel with pentane and Et_2O as elute.

Controlled experiment and the procedure for capture of intermediate.[5]

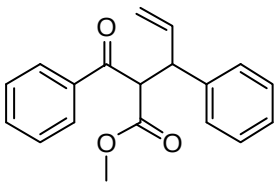


A 10 ml schlenk tube was charged with $[Ir(cod)Cl]_2$ (3.0 mg, 0.0045 mmol, 0.015 equiv), (±)-10-Camphorsulfonic Acid (CSA) (35 mg, 0.15 mmol, 0.5 equiv) and acetophenone **1** (0.39 mmol, 1.3 equiv). The tube was flushed with nitrogen for three times. Then 1.5 ml of dry 1,2-dichloroethane (DCE) and allylic alcohol **2a** (0.3 mmol, 1.0 equiv) were added by syringe and the reaction was stirred at 25 °C for 10 hours. The GC-MS of the crude mixture showed no conversion of **1a** and no product **3** or **4** was observed.



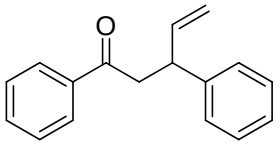
A 10 ml schlenk tube was charged with $[Ir(cod)Cl]_2$ (3.0 mg, 0.0045 mmol, 0.015 equiv), (±)-10-Camphorsulfonic Acid (CSA) (35 mg, 0.15 mmol, 0.5 equiv) and benzoylaceticacid **1a** (0.39 mmol, 1.3 equiv). The tube was flushed with nitrogen for three times. Then 1.5 ml of dry 1,2-dichloroethane (DCE) and allylic alcohol **2a** (0.3 mmol, 1.0 equiv) were added by syringe and the reaction was stirred at 25 °C for 0.5 h. Then methanol/ ether (1 ml/1 mL) and trimethylsilyldiazomethane solution (2.0 M in hexane, 0.9 mmol, 3 equiv) were added and the mixture was stirred at room temperature for 5 minutes. The reaction mixture was concentrated in vacuo and purified by flash column chromatography on silica gel with pentane/Et_2O as elute to get 1,3-diphenylpent-4-en-1-one **3a** (8.5 mg, 12 %) and methyl 2-benzoyl-3-phenylpent-4-enoate **B** (53.8 mg, 61 %, dr = 7 :3).

Caution: When adding the $TMSCHN_2$, the nitrogen is generated dramatically. Perform this process in an open system.



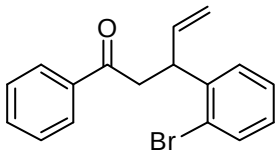
Methyl 2-benzoyl-3-phenylpent-4-enoate (**B**, one isomer, separated by preparative HPLC with acetonitrile/water as elute). White solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05 – 8.00 (m, 2H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 2H), 7.28 – 7.22 (m, 4H), 7.19 – 7.15 (m, 1H), 5.88 – 5.79 (m, 1H), 4.93 (d, *J* = 7.4 Hz, 1H), 4.90 (d, *J* = 0.8 Hz, 1H), 4.86 (d, *J* = 11.1 Hz, 1H), 4.38 (dd, *J* = 11.0, 7.3 Hz, 1H), 3.33 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 193.01, 168.04, 140.15, 138.07, 136.80, 133.74, 128.85, 128.79, 128.59, 128.33, 127.12, 116.50, 59.09, 52.42, 49.40. GCMS (EI) m/z: 294 (M^+).

Analytical data for the products



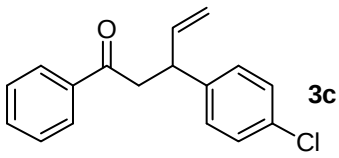
3a

1,3-Diphenylpent-4-en-1-one (**3a**).[5] Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 – 7.89 (m, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.27 (d, *J* = 7.7 Hz, 2H), 7.21 (t, *J* = 7.1 Hz, 1H), 6.06 (ddd, *J* = 17.1, 10.3, 6.8 Hz, 1H), 5.06 (dd, *J* = 18.6, 13.7 Hz, 2H), 4.15 (q, *J* = 6.9 Hz, 1H), 3.41 (qd, *J* = 16.6, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.39, 143.27, 140.78, 137.25, 133.15, 128.71, 128.18, 127.83, 126.67, 114.84, 44.64, 44.14.



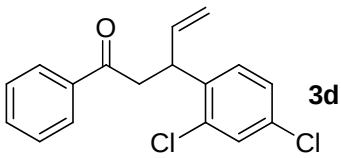
3b

3-(2-Bromophenyl)-1-phenylpent-4-en-1-one (**3b**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 – 7.91 (m, 2H), 7.57 (ddd, *J* = 8.6, 3.1, 2.0 Hz, 2H), 7.46 (dd, *J* = 10.6, 4.8 Hz, 2H), 7.34 – 7.22 (m, 2H), 7.17 – 7.02 (m, 1H), 6.04 (ddd, *J* = 17.0, 10.4, 6.4 Hz, 1H), 5.08 (ddt, *J* = 58.1, 17.2, 1.2 Hz, 2H), 4.65 (ddd, *J* = 9.9, 6.3, 1.3 Hz, 1H), 3.42 (qd, *J* = 16.9, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.71, 142.30, 138.98, 136.98, 133.37, 133.24, 128.82, 128.73, 128.22, 127.74, 124.68, 115.79, 43.22. GCMS (EI) m/z: 314 (M^+); Anal. Calcd for $C_{17}H_{15}BrO$: C, 64.78; H, 4.80. Found: C, 64.99; H, 4.66%.



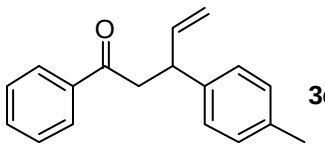
3c

3-(4-Chlorophenyl)-1-phenylpent-4-en-1-one (**3c**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96 – 7.88 (m, 2H), 7.56 (dd, *J* = 10.5, 4.3 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.30 – 7.24 (m, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 6.01 (ddd, *J* = 17.1, 10.3, 6.6 Hz, 1H), 5.13 – 4.96 (m, 2H), 4.12 (q, *J* = 6.9 Hz, 1H), 3.38 (qd, *J* = 16.8, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.00, 141.69, 140.40, 137.08, 133.29, 132.36, 129.27, 128.79, 128.76, 128.14, 115.18, 77.39, 77.14, 76.88, 43.91. GCMS (EI) m/z: 270 (M^+); Anal. Calcd for $C_{17}H_{15}ClO$: C, 75.41; H, 5.58. Found: C, 75.14; H, 5.69%.



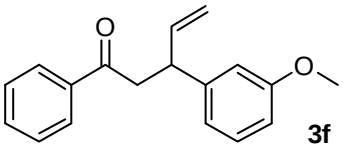
3d

3-(2,4-Dichlorophenyl)-1-phenylpent-4-en-1-one (**3d**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97 (dt, *J* = 8.5, 1.5 Hz, 2H), 7.67 – 7.52 (m, 1H), 7.51 – 7.44 (m, 2H), 7.43 – 7.38 (m, 1H), 7.26 – 7.18 (m, 2H), 6.02 (ddd, *J* = 17.0, 10.4, 6.4 Hz, 1H), 5.09 (ddt, *J* = 61.1, 17.2, 1.2 Hz, 2H), 4.62 (dtt, *J* = 7.7, 6.3, 1.4 Hz, 1H), 3.43 (qd, *J* = 17.0, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.47, 139.27, 138.56, 136.87, 134.61, 133.36, 132.84, 129.77, 129.62, 128.78, 128.17, 127.37, 116.06, 42.82, 40.35. GCMS (EI) m/z: 304 (M^+); Anal. Calcd for $C_{17}H_{14}Cl_2O$: C, 66.90; H, 4.62. Found: C, 67.20; H, 4.43%.



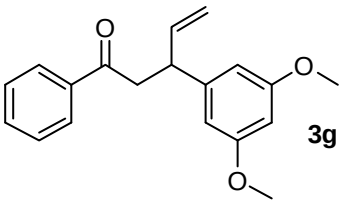
3e

1-Phenyl-3-(p-tolyl)pent-4-en-1-one (**3e**).[6] Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97 – 7.89 (m, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.13 (dd, *J* = 20.5, 8.1 Hz, 4H), 6.03 (ddd, *J* = 17.1, 10.3, 6.8 Hz, 1H), 5.21 – 4.90 (m, 2H), 4.10 (q, *J* = 6.9 Hz, 1H), 3.38 (ddd, *J* = 23.1, 16.6, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.49, 140.98, 140.25, 137.27, 136.20, 133.13, 129.40, 128.69, 128.20, 127.67, 114.62, 44.27, 44.20, 21.12.



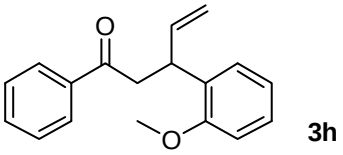
3f

3-(3-Methoxyphenyl)-1-phenylpent-4-en-1-one (**3f**).[6] Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.57 – 7.53 (m, 1H), 7.47 – 7.43 (m, 2H), 7.23 (t, *J* = 7.9 Hz, 1H), 6.87 (t, *J* = 5.7 Hz, 1H), 6.83 – 6.79 (m, 1H), 6.78 – 6.73 (m, 1H), 6.03 (ddd, *J* = 17.1, 10.3, 6.8 Hz, 1H), 5.06 (ddt, *J* = 18.1, 17.1, 1.3 Hz, 2H), 4.12 (q, *J* = 6.7 Hz, 1H), 3.79 (s, 3H), 3.40 (ddd, *J* = 23.0, 16.6, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.34, 159.87, 144.97, 140.58, 137.27, 133.14, 129.67, 128.69, 128.17, 120.14, 114.90, 113.82, 111.81, 55.28, 44.67, 44.09.



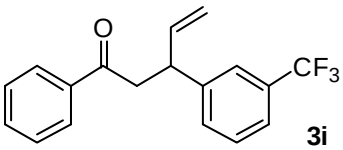
3g

3-(3,5-Dimethoxyphenyl)-1-phenylpent-4-en-1-one (**3g**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 (dd, *J* = 5.1, 3.4 Hz, 2H), 7.61 – 7.50 (m, 1H), 7.45 (dd, *J* = 10.6, 4.8 Hz, 2H), 6.42 (d, *J* = 2.2 Hz, 2H), 6.32 (t, *J* = 2.2 Hz, 1H), 6.02 (ddd, *J* = 17.1, 10.4, 6.8 Hz, 1H), 5.10 – 5.01 (m, 2H), 4.08 (dd, *J* = 14.2, 6.7 Hz, 1H), 3.77 (s, 6H), 3.38 (ddd, *J* = 22.9, 16.7, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.33, 160.99, 145.82, 140.37, 137.23, 133.17, 128.71, 128.19, 114.99, 105.98, 98.40, 55.39, 44.84, 44.00. GCMS (EI) m/z: 296 (M^+); Anal. Calcd for $C_{19}H_{20}O_3$: C, 77.00; H, 6.80. Found: C, 77.25; H, 7.06%.



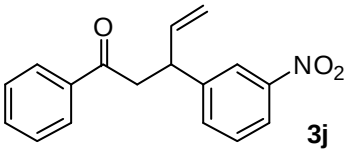
3h

3-(2-Methoxyphenyl)-1-phenylpent-4-en-1-one (**3h**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 7.7 Hz, 2H), 7.56 (dd, *J* = 13.7, 6.8 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.21 (t, *J* = 7.8 Hz, 2H), 6.93 (t, *J* = 7.5 Hz, 1H), 6.87 (d, *J* = 8.0 Hz, 1H), 6.12 (ddd, *J* = 17.2, 10.3, 6.9 Hz, 1H), 5.05 (dd, *J* = 22.4, 13.8 Hz, 2H), 4.48 (q, *J* = 7.0 Hz, 1H), 3.83 (s, 3H), 3.44 – 3.34 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.91, 156.93, 139.91, 137.33, 132.93, 131.65, 128.58, 128.43, 128.25, 127.69, 120.76, 114.88, 110.90, 55.44, 43.30, 39.11. GCMS (EI) m/z: 266 (M^+); Anal. Calcd for $C_{18}H_{18}O_2$: C, 81.17; H, 6.81. Found: C, 81.09; H, 7.14%.



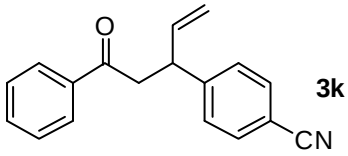
3i

1-Phenyl-3-(3-(trifluoromethyl)phenyl)pent-4-en-1-one (**3i**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96 – 7.90 (m, 2H), 7.58 – 7.54 (m, 1H), 7.51 (s, 1H), 7.48 – 7.40 (m, 5H), 6.04 (ddd, *J* = 17.1, 10.3, 6.7 Hz, 1H), 5.09 (ddt, *J* = 36.4, 17.2, 1.2 Hz, 2H), 4.22 (q, *J* = 6.9 Hz, 1H), 3.43 (ddd, *J* = 52.2, 16.9, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.79, 144.22, 139.96, 137.02, 133.34, 131.48, 130.96 (q, *J* = 31.5 Hz). 129.11, 128.77, 128.42, 128.14, 124.58, 124.25(q, *J* = 270.1 Hz), 123.58, 115.64, 44.27, 43.83. ^{19}F NMR (470 MHz, $CDCl_3$) δ -62.50. GCMS (EI) m/z: 304 (M^+); Anal. Calcd for $C_{18}H_{15}F_3O$: C, 71.04; H, 4.97. Found: C, 71.15; H, 4.68%.



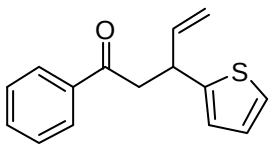
3j

3-(3-Nitrophenyl)-1-phenylpent-4-en-1-one (**3j**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.14 (t, *J* = 1.9 Hz, 1H), 8.07 (ddd, *J* = 8.2, 2.1, 0.9 Hz, 1H), 7.98 – 7.88 (m, 2H), 7.63 (d, *J* = 7.7 Hz, 1H), 7.60 – 7.55 (m, 1H), 7.47 (dt, *J* = 7.6, 6.0 Hz, 3H), 6.04 (ddd, *J* = 17.1, 10.3, 6.7 Hz, 1H), 5.13 (dd, *J* = 35.1, 13.7 Hz, 2H), 4.28 (q, *J* = 6.9 Hz, 1H), 3.47 (qd, *J* = 17.1, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.40, 148.55, 145.36, 139.57, 136.81, 134.57, 133.49, 129.55, 128.82, 128.12, 122.70, 121.84, 116.11, 44.03, 43.63. GCMS (EI) m/z: 281 (M^+); Anal. Calcd for $C_{17}H_{15}NO_3$: C, 72.58; H, 5.37. Found: C, 72.43; H, 5.44%.



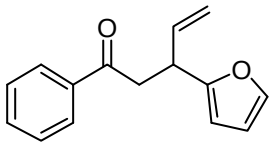
3k

4-(5-Oxo-5-phenylpent-1-en-3-yl)benzonitrile (**3k**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 (dt, *J* = 8.5, 1.5 Hz, 2H), 7.61 – 7.54 (m, 3H), 7.50 – 7.43 (m, 2H), 7.41 – 7.34 (m, 2H), 6.01 (ddd, *J* = 17.1, 10.3, 6.7 Hz, 1H), 5.10 (ddt, *J* = 40.7, 17.2, 1.2 Hz, 2H), 4.21 (q, *J* = 6.9 Hz, 1H), 3.43 (qd, *J* = 17.1, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.44, 148.78, 139.51, 136.84, 133.49, 132.51, 128.81, 128.11, 118.98, 116.01, 110.57, 44.47, 43.53. GCMS (EI) m/z: 261 (M^+); Anal. Calcd for $C_{18}H_{15}NO$: C, 82.73; H, 5.79. Found: C, 83.04; H, 5.62%.



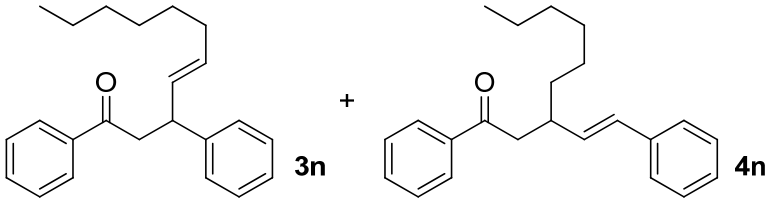
3l

1-Phenyl-3-(thiophen-2-yl)pent-4-en-1-one (**3l**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 – 7.90 (m, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 2H), 7.16 (dd, *J* = 5.1, 1.1 Hz, 1H), 6.96 – 6.84 (m, 2H), 6.04 (ddd, *J* = 17.2, 10.2, 7.2 Hz, 1H), 5.12 (dd, *J* = 13.5, 12.9 Hz, 2H), 4.44 (q, *J* = 6.9 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.82, 146.94, 140.16, 137.12, 133.29, 128.75, 128.21, 126.90, 124.15, 123.76, 115.50, 45.08, 39.99. GCMS (EI) m/z: 242 (M^+); Anal. Calcd for $C_{15}H_{14}OS$: C, 74.34; H, 5.82. Found: C, 74.56; H, 5.98%.



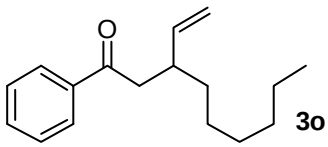
3m

3-(Furan-2-yl)-1-phenylpent-4-en-1-one (**3m**).[6] Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 – 7.92 (m, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.49 – 7.43 (m, 2H), 7.32 (d, *J* = 1.1 Hz, 1H), 6.28 (dd, *J* = 3.1, 1.9 Hz, 1H), 6.07 (d, *J* = 3.2 Hz, 1H), 6.03 – 5.92 (m, 1H), 5.18 – 5.03 (m, 2H), 4.23 (q, *J* = 7.0 Hz, 1H), 3.41 (ddd, *J* = 24.4, 16.7, 7.0 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.88, 156.03, 141.52, 137.88, 137.10, 133.22, 128.71, 128.20, 116.22, 110.29, 105.50, 42.00, 38.58.



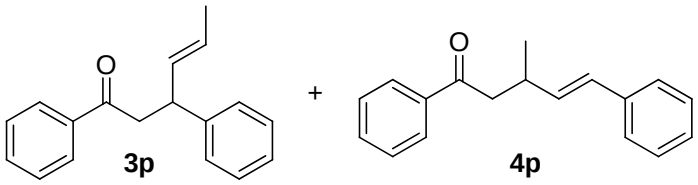
3n **4n**

(*E*)-1,3-Diphenylundec-4-en-1-one (**3n**) and (*E*)-1-Phenyl-3-styrylnonan-1-one (**4n**). Mixture of **3n** and **4n**, the ratio is 75:25. Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 – 7.85 (m, 3H), 7.54 (dd, *J* = 9.6, 5.2 Hz, 1H), 7.49 – 7.39 (m, 3H), 7.35 – 7.22 (m, 6H), 7.22 – 7.14 (m, 1H), 6.38 (d, *J* = 15.8 Hz, 1H), 6.07 (dd, *J* = 15.8, 8.7 Hz, 1H), 5.62 (dd, *J* = 15.3, 7.2 Hz, 1H), 5.48 – 5.38 (m, 1H), 4.07 (q, *J* = 7.1 Hz, 1H), 3.36 (qd, *J* = 16.2, 7.2 Hz, 2H), 3.10 – 3.03 (m, 1H), 2.96 – 2.88 (m, 1H), 1.96 (q, *J* = 7.0 Hz, 2H), 1.40 – 1.08 (m, 13H), 0.88 – 0.83 (m, 4H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 199.49, 198.76, 144.25, 137.46, 133.73, 132.99, 132.25, 131.29, 130.21, 128.62, 128.20, 127.68, 127.10, 126.42, 126.20, 44.93, 44.50, 44.06, 39.26, 35.25, 32.58, 31.89, 31.76, 29.79, 29.36, 28.85, 27.37, 22.68, 14.15. GCMS (EI) m/z: 320 (M^+).



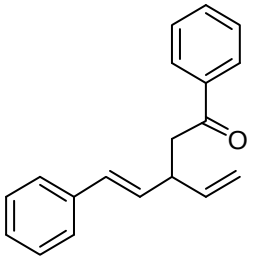
3o

1-Phenyl-3-vinylnonan-1-one (**3o**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.3 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 5.68 (ddd, *J* = 17.3, 10.2, 8.3 Hz, 1H), 4.98 (dd, *J* = 13.7, 7.2 Hz, 2H), 2.97 (d, *J* = 6.8 Hz, 2H), 2.80 – 2.67 (m, 1H), 1.30 – 1.23 (m, 8H), 0.87 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 199.67, 141.75, 137.53, 132.97, 128.65, 128.22, 114.76, 44.06, 39.88, 34.84, 31.89, 29.37, 27.15, 22.72, 14.17. GCMS (EI) m/z: 244 (M^+); Anal. Calcd for $C_{17}H_{24}O$: C, 83.55; H, 9.90. Found: C, 83.24; H, 9.97%.



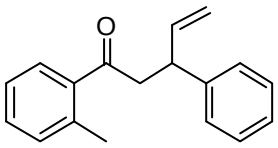
3p + **4p**

(*E*)-1,3-Diphenylhex-4-en-1-one (**3p**) and (*E*)-3-Methyl-1,5-diphenylpent-4-en-1-one (**4p**).[4] Mixture of **3p** and **4p**, the ratio is 60:40. Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 (dd, *J* = 19.9, 8.0 Hz, 4H), 7.55 (dt, *J* = 7.8, 4.0 Hz, 2H), 7.51 – 7.41 (m, 4H), 7.37 – 7.23 (m, 8H), 7.19 (t, *J* = 7.0 Hz, 2H), 6.42 (d, *J* = 15.9 Hz, 1H), 6.23 (dd, *J* = 15.9, 6.7 Hz, 1H), 5.65 (dd, *J* = 15.2, 7.2 Hz, 1H), 5.51 – 5.41 (m, 1H), 4.07 (q, *J* = 7.1 Hz, 1H), 3.37 (qd, *J* = 16.4, 7.1 Hz, 2H), 3.16 – 3.05 (m, 1H), 3.00 (dd, *J* = 14.4, 5.8 Hz, 1H), 1.64 (d, *J* = 6.4 Hz, 3H), 1.20 (d, *J* = 6.0 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 199.33, 198.67, 144.20, 137.59, 137.36, 135.03, 133.66, 133.06, 128.66, 128.19, 127.69, 127.16, 126.47, 126.20, 125.64, 45.69, 44.86, 43.97, 33.25, 20.37, 18.04. GCMS (EI) m/z: 250 (M^+).



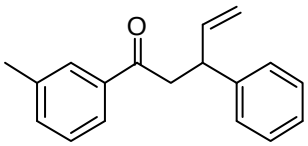
3q

(*E*)-1,5-Diphenyl-3-vinylpent-4-en-1-one (**3q**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 – 7.93 (m, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.7 Hz, 2H), 7.33 (d, *J* = 7.4 Hz, 2H), 7.28 (t, *J* = 7.6 Hz, 2H), 7.20 (t, *J* = 7.2 Hz, 1H), 6.45 (d, *J* = 15.9 Hz, 1H), 6.22 (dd, *J* = 15.9, 7.4 Hz, 1H), 5.93 (ddd, *J* = 17.2, 10.3, 6.9 Hz, 1H), 5.11 (dd, *J* = 18.8, 13.8 Hz, 2H), 3.69 (p, *J* = 6.9 Hz, 1H), 3.21 (d, *J* = 7.0 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.49, 139.77, 137.32, 133.18, 131.40, 130.53, 128.73, 128.58, 128.23, 127.35, 126.30, 115.32, 43.36, 42.24. GCMS (EI) m/z: 262 (M^+); Anal. Calcd for $C_{19}H_{18}O$: C, 86.99; H, 6.92. Found: C, 86.64; H, 7.09%.



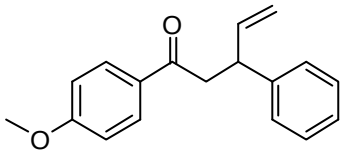
5a

3-Phenyl-1-(o-tolyl)pent-4-en-1-one (**5a**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.6 Hz, 1H), 7.35 (t, *J* = 7.5 Hz, 1H), 7.29 (t, *J* = 7.6 Hz, 2H), 7.25 – 7.18 (m, 5H), 6.02 (ddd, *J* = 17.1, 10.3, 6.8 Hz, 1H), 5.05 (dd, *J* = 18.3, 13.8 Hz, 2H), 4.07 (q, *J* = 7.1 Hz, 1H), 3.32 (qd, *J* = 16.2, 7.4 Hz, 2H), 2.34 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 202.84, 143.01, 140.80, 138.52, 138.05, 131.96, 131.20, 128.68, 128.15, 127.84, 126.68, 125.67, 114.76, 47.19, 45.12, 20.91. GCMS (EI) m/z: 250 (M^+); Anal. Calcd for $C_{18}H_{18}O$: C, 86.36; H, 7.25. Found: C, 86.49; H, 7.01%.



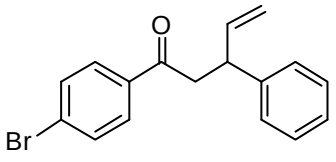
5b

3-Phenyl-1-(m-tolyl)pent-4-en-1-one (**5b**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.0 Hz, 2H), 7.29 (d, *J* = 5.3 Hz, 1H), 7.26 – 7.19 (m, 5H), 7.16 – 7.12 (m, 1H), 5.98 (ddd, *J* = 17.1, 10.3, 6.7 Hz, 1H), 5.04 – 4.94 (m, 2H), 4.08 (q, *J* = 6.7 Hz, 1H), 3.33 (ddd, *J* = 23.1, 16.6, 7.1 Hz, 2H), 2.33 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.56, 143.34, 140.81, 138.49, 137.30, 133.89, 128.69, 128.55, 127.84, 126.63, 125.39, 114.80, 44.63, 44.19, 21.45. GCMS (EI) m/z: 250 (M^+); Anal. Calcd for $C_{18}H_{18}O$: C, 86.36; H, 7.25. Found: C, 86.54; H, 7.18%.



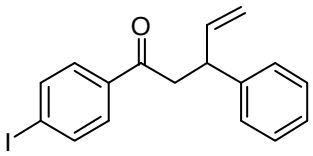
5c

1-(4-Methoxyphenyl)-3-phenylpent-4-en-1-one (**5c**).[6] Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 – 7.83 (m, 2H), 7.32 – 7.25 (m, 4H), 7.23 – 7.17 (m, 1H), 6.99 – 6.84 (m, 2H), 6.05 (ddd, *J* = 17.1, 10.3, 6.8 Hz, 1H), 5.05 (ddt, *J* = 20.8, 17.2, 1.3 Hz, 2H), 4.14 (q, *J* = 6.7 Hz, 1H), 3.85 (s, 3H), 3.35 (ddd, *J* = 22.9, 16.4, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.92, 163.56, 143.43, 140.93, 130.48, 130.36, 128.68, 127.85, 126.62, 114.75, 113.83, 55.57, 44.81, 43.77.



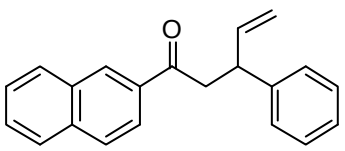
5d

1-(4-Bromophenyl)-3-phenylpent-4-en-1-one (**5d**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 – 7.75 (m, 2H), 7.62 – 7.55 (m, 2H), 7.30 (dd, *J* = 10.4, 4.6 Hz, 2H), 7.25 (d, *J* = 7.0 Hz, 2H), 7.23 – 7.19 (m, 1H), 6.04 (ddd, *J* = 17.1, 10.3, 6.8 Hz, 1H), 5.05 (ddt, *J* = 27.8, 17.2, 1.2 Hz, 2H), 4.11 (q, *J* = 6.9 Hz, 1H), 3.36 (qd, *J* = 16.6, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.40, 143.02, 140.58, 135.93, 132.01, 129.71, 128.75, 128.34, 127.78, 126.77, 114.96, 44.67, 44.08. GCMS (EI) m/z: 314 (M^+); Anal. Calcd for $C_{17}H_{15}BrO$: C, 64.78; H, 4.80. Found: C, 65.09; H, 4.55%.



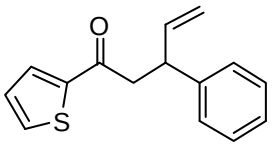
5e

1-(4-Iodophenyl)-3-phenylpent-4-en-1-one (**5e**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.5 Hz, 2H), 7.56 (d, *J* = 8.5 Hz, 2H), 7.23 (t, *J* = 7.5 Hz, 2H), 7.17 (d, *J* = 7.1 Hz, 2H), 7.14 (t, *J* = 7.2 Hz, 1H), 5.96 (ddd, *J* = 17.1, 10.3, 6.8 Hz, 1H), 4.98 (dd, *J* = 24.9, 13.7 Hz, 2H), 4.19 – 4.17 (m, 1H), 4.03 (q, *J* = 6.9 Hz, 1H), 3.28 (qd, *J* = 16.6, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.70, 143.02, 140.58, 138.02, 136.48, 129.58, 128.74, 127.77, 126.75, 114.94, 101.08, 44.67, 44.03. GCMS (EI) m/z: 362 (M^+); Anal. Calcd for $C_{17}H_{15}IO$: C, 56.37; H, 4.17. Found: C, 56.26; H, 3.98%.



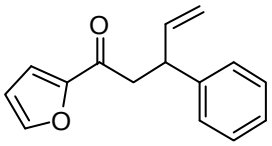
5f

1-(Naphthalen-2-yl)-3-phenylpent-4-en-1-one (**5f**).[6] ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.45 (s, 1H), 8.01 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.95 (d, *J* = 8.0 Hz, 1H), 7.88 (dd, *J* = 8.2, 5.5 Hz, 2H), 7.58 (dtd, *J* = 16.1, 6.9, 1.2 Hz, 2H), 7.34 – 7.29 (m, 4H), 7.22 (ddd, *J* = 6.6, 5.7, 2.6 Hz, 1H), 6.10 (ddd, *J* = 17.1, 10.4, 6.8 Hz, 1H), 5.09 (ddt, *J* = 16.5, 15.3, 1.2 Hz, 2H), 3.55 (ddd, *J* = 23.0, 16.5, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.34, 143.33, 140.81, 135.68, 134.59, 132.62, 129.82, 129.67, 128.74, 128.58, 127.88, 126.89, 126.71, 124.02, 114.91, 44.82, 44.22.



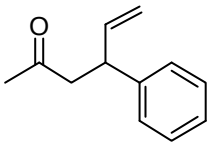
5g

3-Phenyl-1-(thiophen-2-yl)pent-4-en-1-one (**5g**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (dd, *J* = 3.8, 1.0 Hz, 1H), 7.61 (dd, *J* = 4.9, 1.0 Hz, 1H), 7.34 – 7.25 (m, 4H), 7.24 – 7.19 (m, 1H), 7.10 (dd, *J* = 4.9, 3.9 Hz, 1H), 6.05 (ddd, *J* = 17.1, 10.4, 6.8 Hz, 1H), 5.14 – 4.99 (m, 2H), 4.13 (q, *J* = 6.9 Hz, 1H), 3.33 (ddd, *J* = 22.6, 15.9, 7.3 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.26, 144.64, 143.00, 140.50, 133.85, 131.98, 128.74, 128.21, 127.82, 126.76, 115.06, 44.96. GCMS (EI) m/z: 242 (M^+); Anal. Calcd for $C_{15}H_{14}OS$: C, 74.34; H, 5.82. Found: C, 74.62; H, 5.95%.



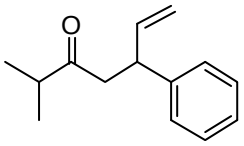
5h

1-(Furan-2-yl)-3-phenylpent-4-en-1-one (**5h**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (s, 1H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.25 (d, *J* = 6.6 Hz, 2H), 7.20 (t, *J* = 7.1 Hz, 1H), 7.15 (d, *J* = 3.6 Hz, 1H), 6.50 (dd, *J* = 3.4, 1.4 Hz, 1H), 6.03 (ddd, *J* = 17.1, 10.4, 6.9 Hz, 1H), 5.11 – 4.97 (m, 2H), 4.10 (dd, *J* = 15.1, 7.5 Hz, 1H), 3.25 (ddd, *J* = 22.6, 15.9, 7.4 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 187.61, 153.07, 146.41, 142.97, 140.51, 128.67, 127.78, 126.69, 117.17, 114.95, 112.34, 44.66, 43.96. GCMS (EI) m/z: 226 (M^+); Anal. Calcd for $C_{15}H_{14}O_2$: C, 79.62; H, 6.24. Found: C, 79.36; H, 6.17%.



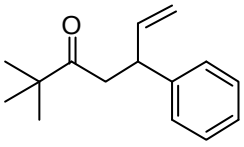
5i

4-Phenylhex-5-en-2-one (**5i**).[5] Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 – 7.28 (m, 2H), 7.21 (dd, *J* = 10.0, 4.4 Hz, 3H), 5.97 (ddd, *J* = 17.1, 10.3, 6.8 Hz, 1H), 5.10 – 4.96 (m, 2H), 3.91 (q, *J* = 7.1 Hz, 1H), 2.85 (qd, *J* = 16.2, 7.3 Hz, 2H), 2.09 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 207.14, 142.89, 140.66, 128.72, 127.70, 126.72, 114.72, 49.11, 44.66, 30.75.



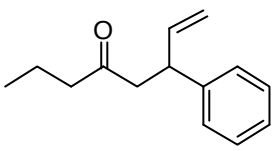
5j

2-Methyl-5-phenylhept-6-en-3-one (**5j**).[7] Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.26 (m, 2H), 7.23 – 7.16 (m, 3H), 5.98 (ddt, *J* = 12.5, 10.3, 6.3 Hz, 1H), 5.20 – 4.88 (m, 2H), 3.95 (dd, *J* = 12.5, 6.0 Hz, 1H), 3.07 – 2.74 (m, 2H), 2.50 (dq, *J* = 13.7, 6.8 Hz, 1H), 1.04 (t, *J* = 6.3 Hz, 3H), 0.97 (t, *J* = 6.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 212.70, 143.24, 140.88, 128.64, 127.78, 126.61, 114.61, 46.05, 44.40, 41.45, 18.02, 17.91.



5k

2,2-Dimethyl-5-phenylhept-6-en-3-one (**5k**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.5 Hz, 2H), 7.23 – 7.16 (m, 3H), 5.97 (ddd, *J* = 17.1, 10.3, 6.8 Hz, 1H), 5.11 – 4.91 (m, 2H), 3.99 (q, *J* = 7.0 Hz, 1H), 2.95 – 2.84 (m, 2H), 1.05 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 213.49, 143.48, 141.03, 128.57, 127.89, 126.52, 114.55, 44.23, 44.09, 42.42, 26.20. GCMS (EI) m/z: 216 (M^+); Anal. Calcd for $C_{15}H_{20}O$: C, 83.28; H, 9.32. Found: C, 83.13; H, 9.46%.



5l

6-Phenyloct-7-en-4-one (**5l**). Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (dd, *J* = 10.6, 4.5 Hz, 2H), 7.20 (ddd, *J* = 7.1, 3.3, 2.4 Hz, 3H), 5.97 (ddd, *J* = 17.1, 10.3, 6.8 Hz, 1H), 5.03 (ddt, *J* = 22.1, 17.1, 1.3 Hz, 2H), 3.93 (q, *J* = 7.1 Hz, 1H), 2.82 (qd, *J* = 16.1, 7.3 Hz, 2H), 2.31 (qt, *J* = 16.8, 7.3 Hz, 2H), 1.59 – 1.47 (m, 2H), 0.84 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 209.32, 143.07, 140.80, 128.68, 127.74, 126.65, 114.65, 48.22, 45.64, 44.61, 17.09, 13.75. GCMS (EI) m/z: 202 (M^+); Anal. Calcd for $C_{14}H_{18}O$: C, 83.12; H, 8.97. Found: C, 83.35; H, 8.81%.

References

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(b) Y. Jiang, X. Chen, Y. Zheng, Z. Xue, C. Shu, W. Yuan and X. Zhang, *Angew. Chem., Int. Ed.,* 2011, **50**, 7304.

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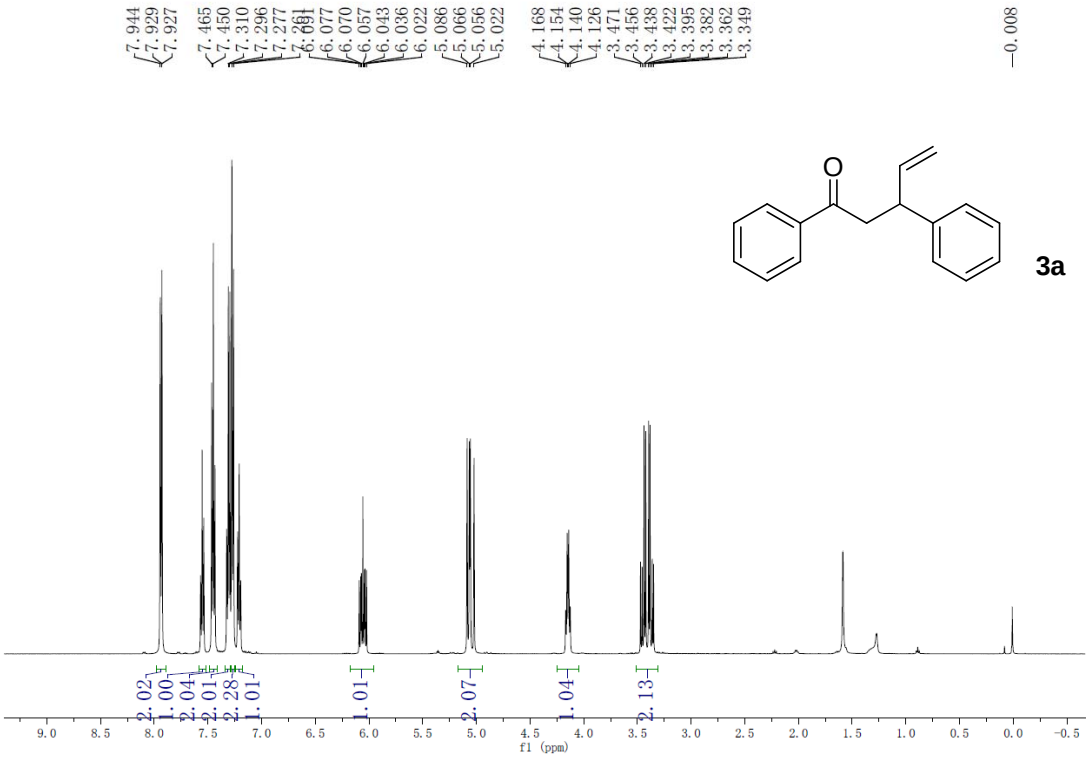
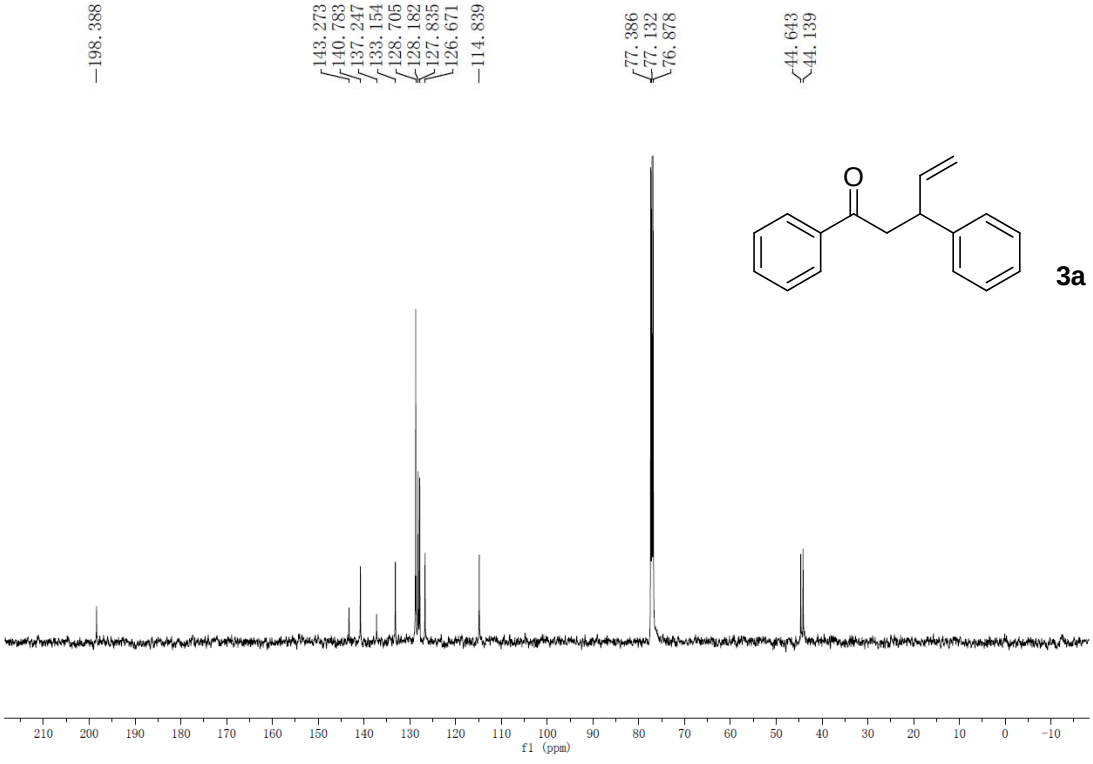
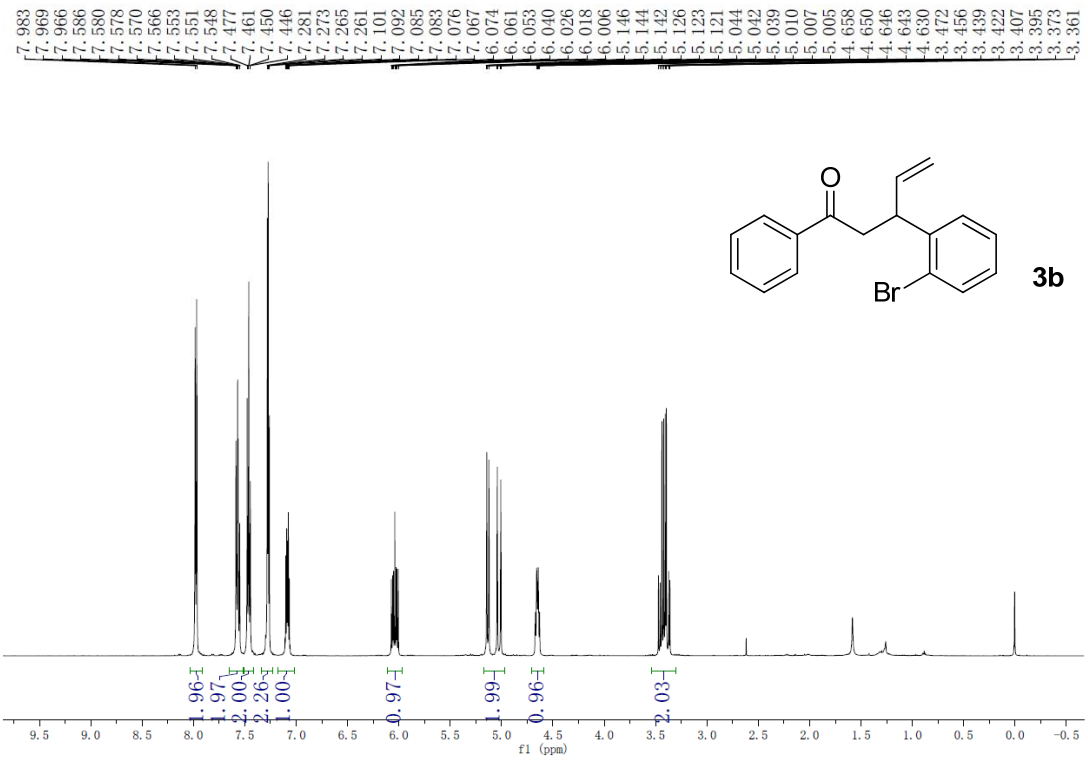
7 D. J. Weix and J. F. Hartwig, *J. Am. Chem. Soc.,* 2007, **129**, 7720.

Copies of ^{1}H and ^{13}C NMR spectra



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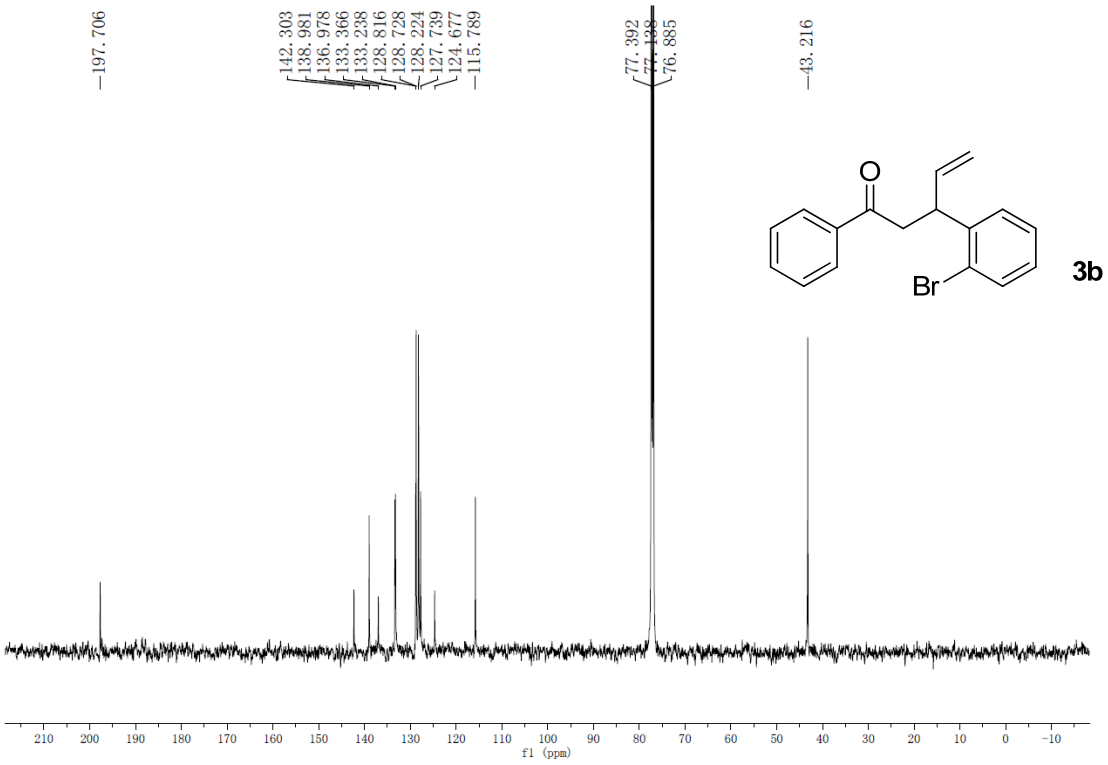
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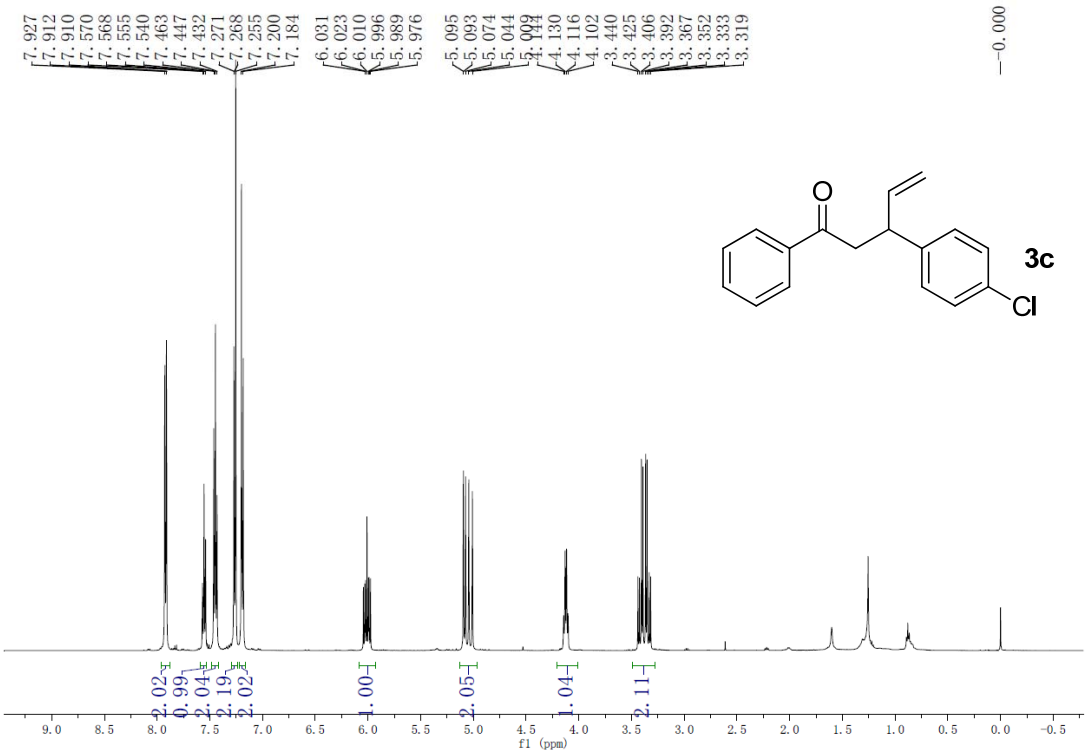
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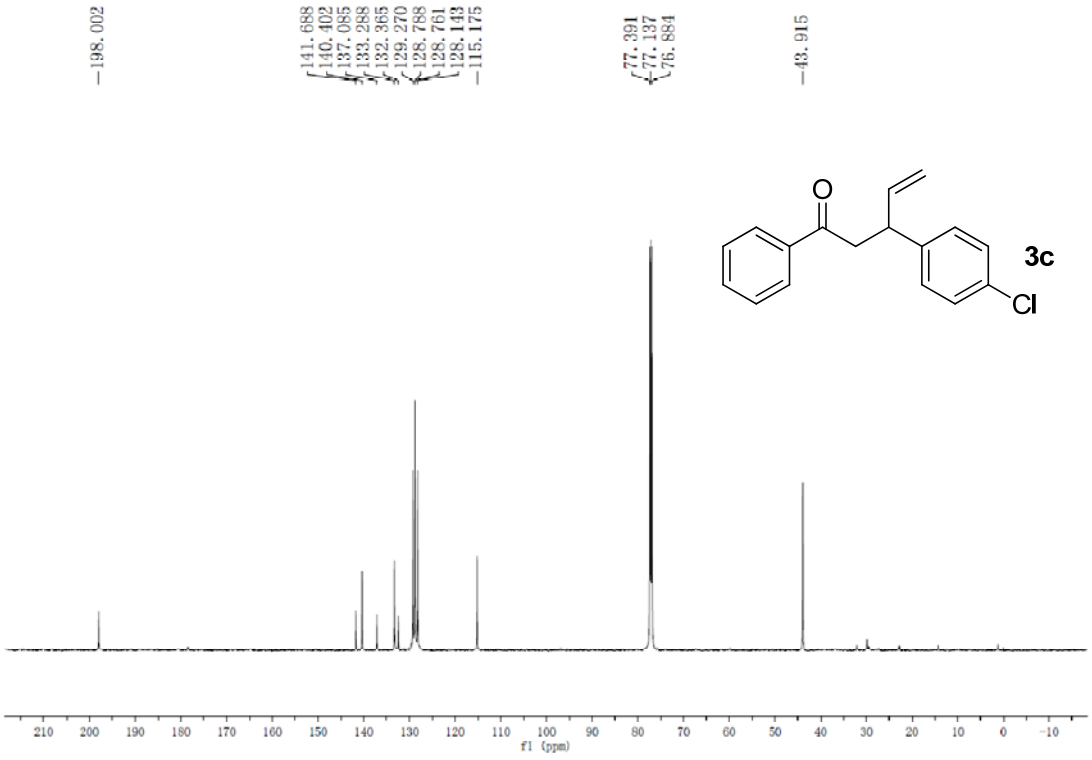
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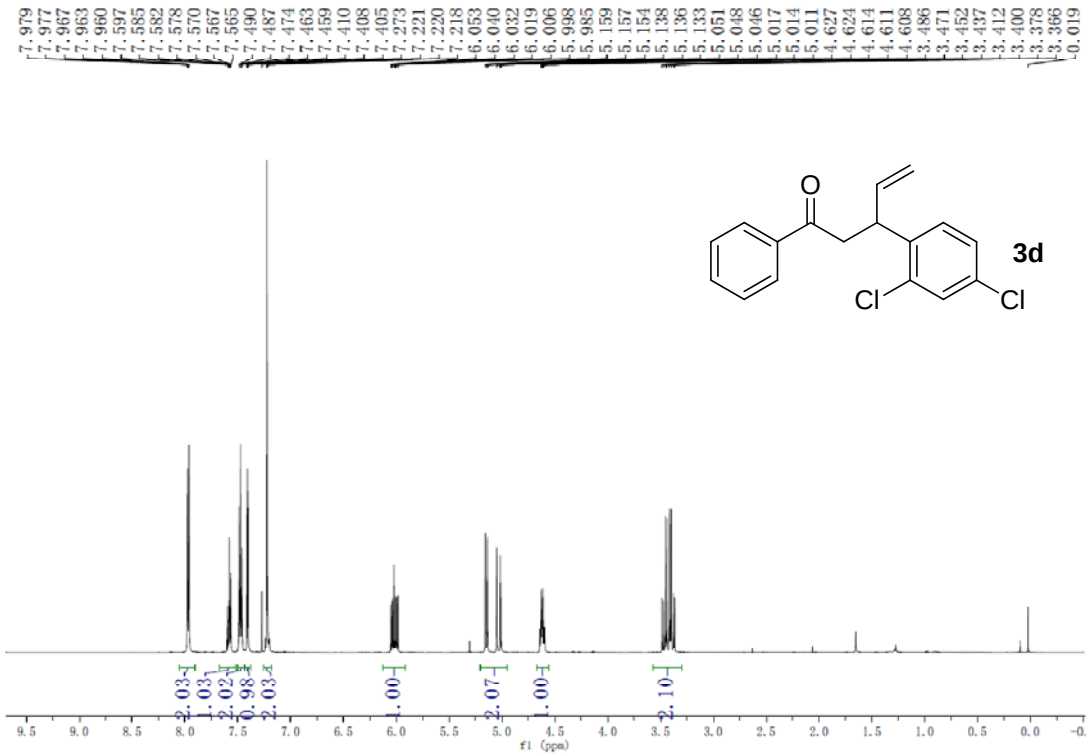
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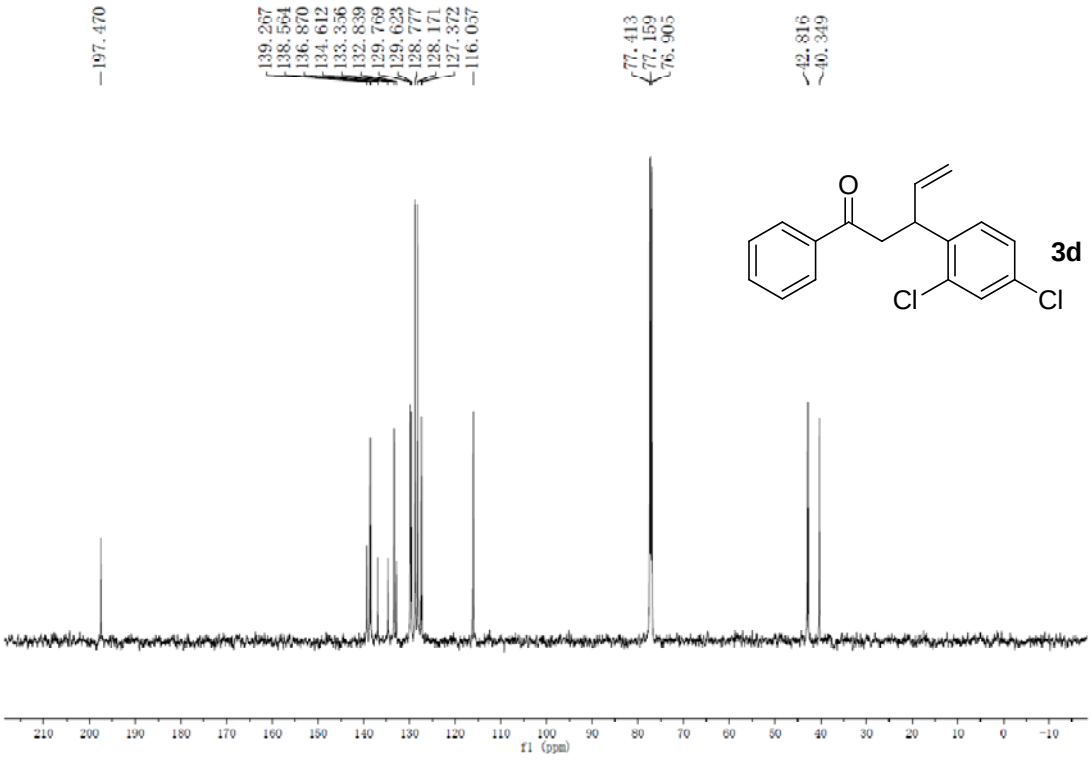
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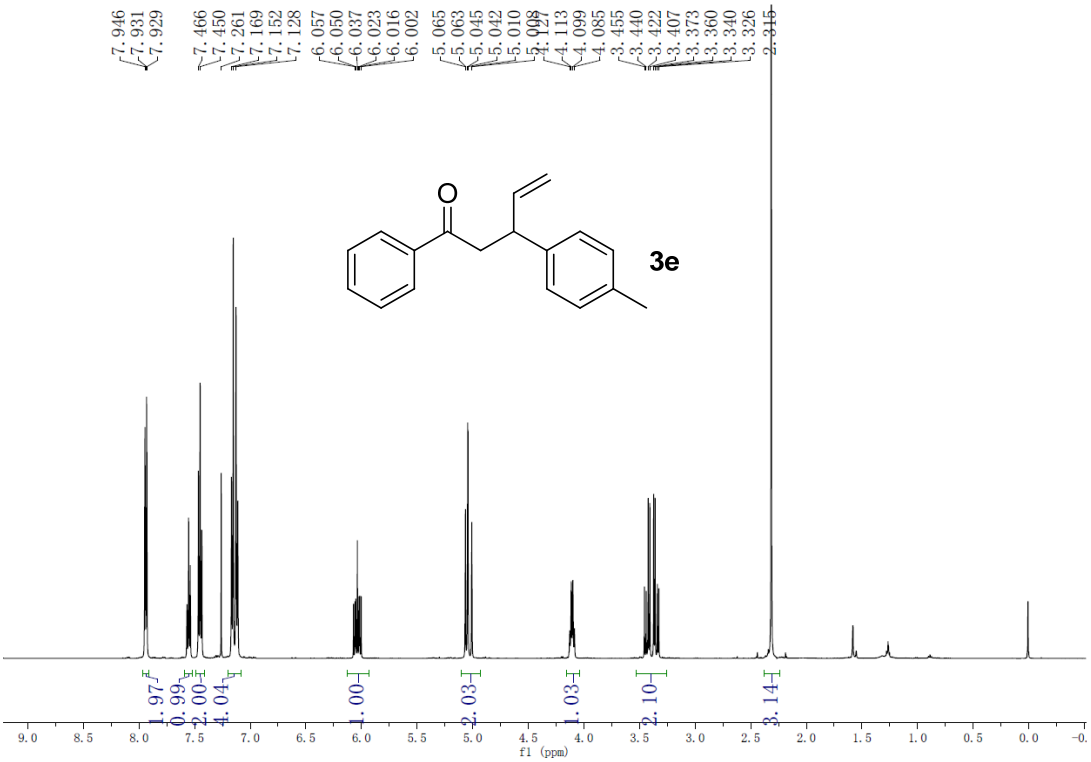
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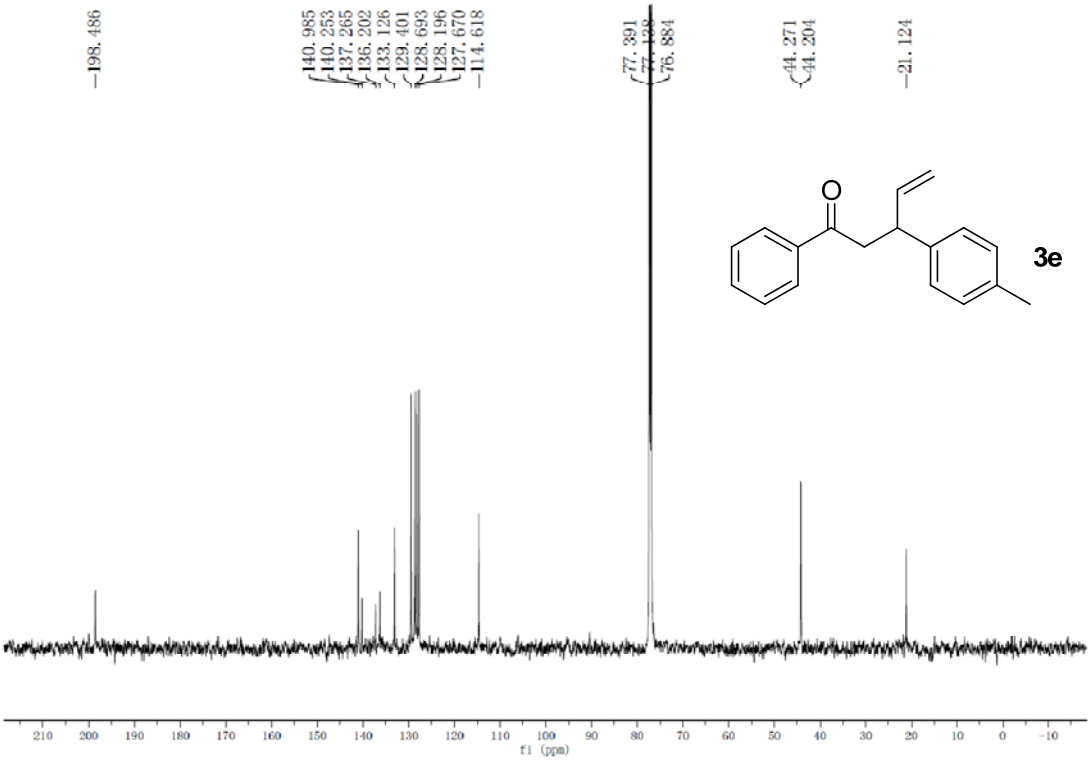
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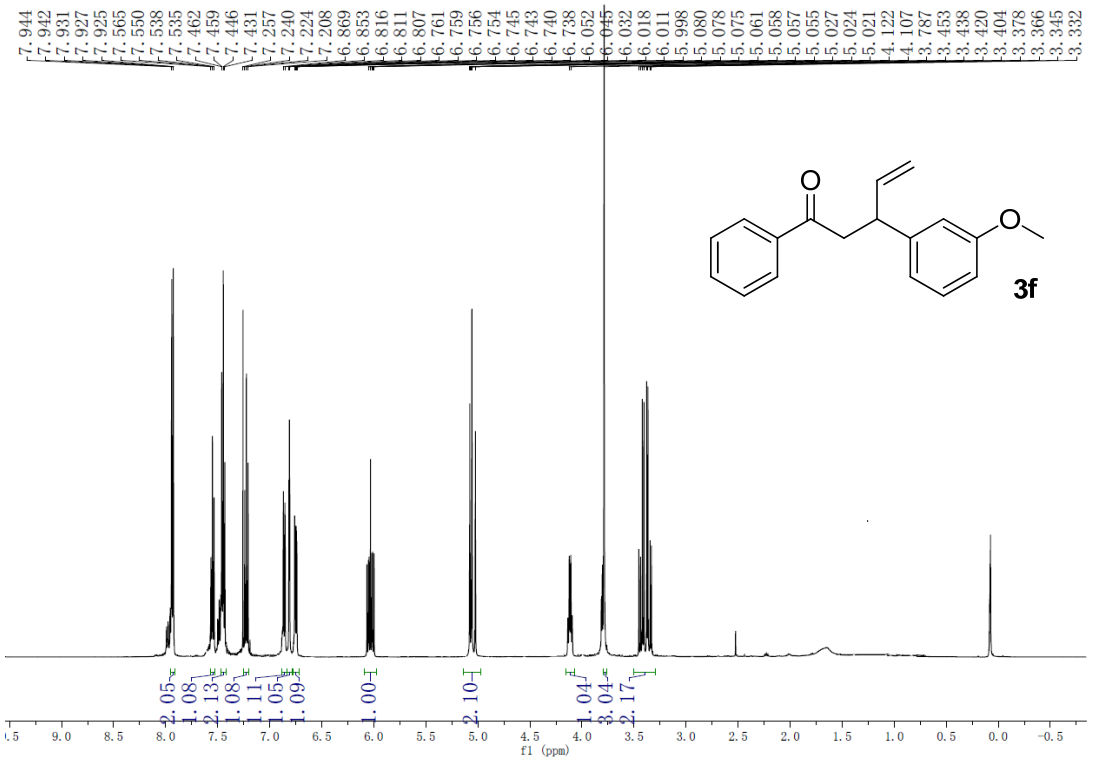
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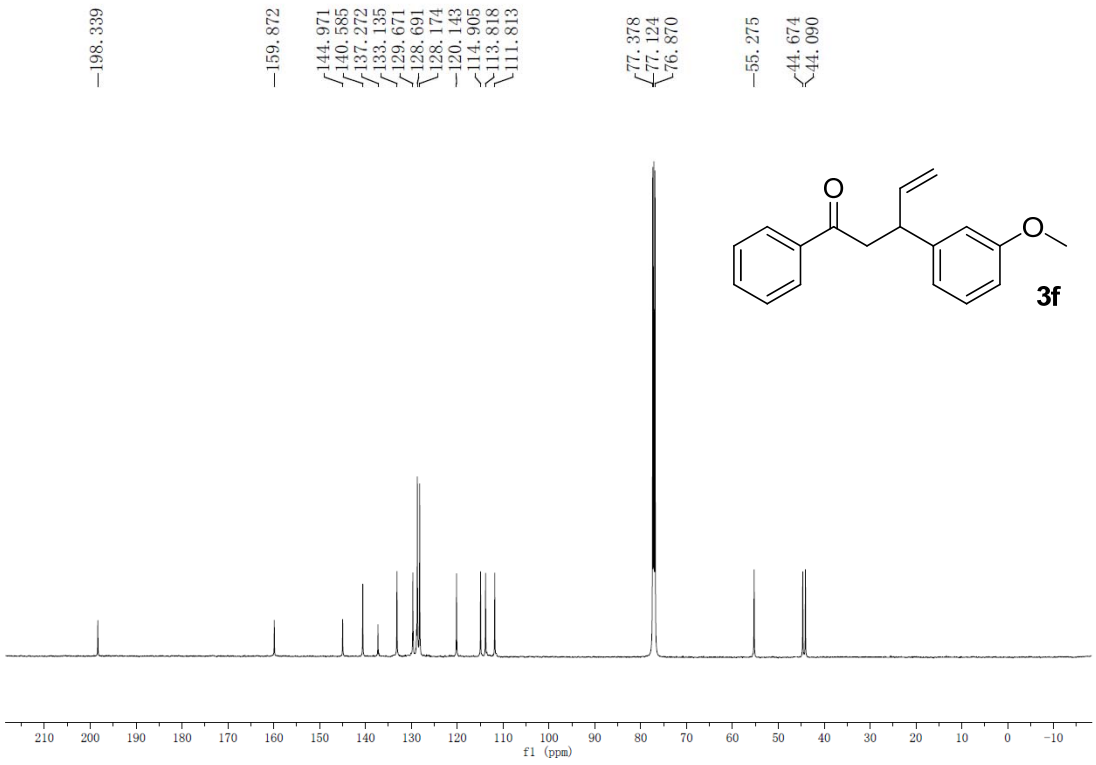
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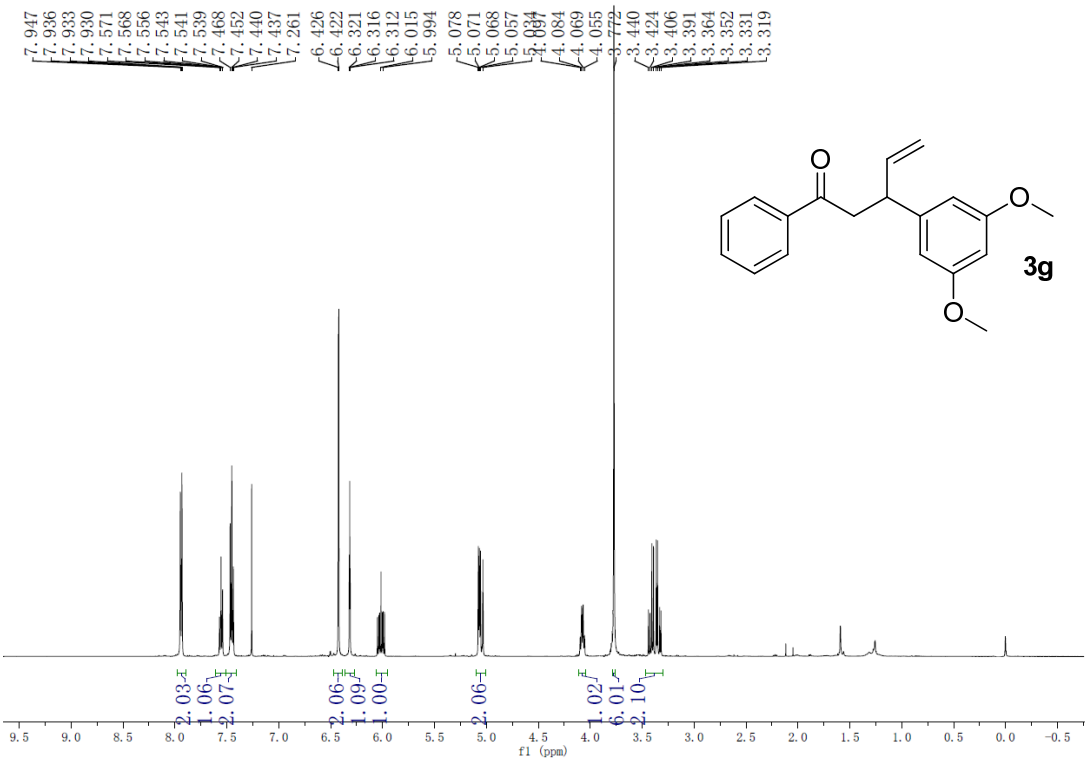
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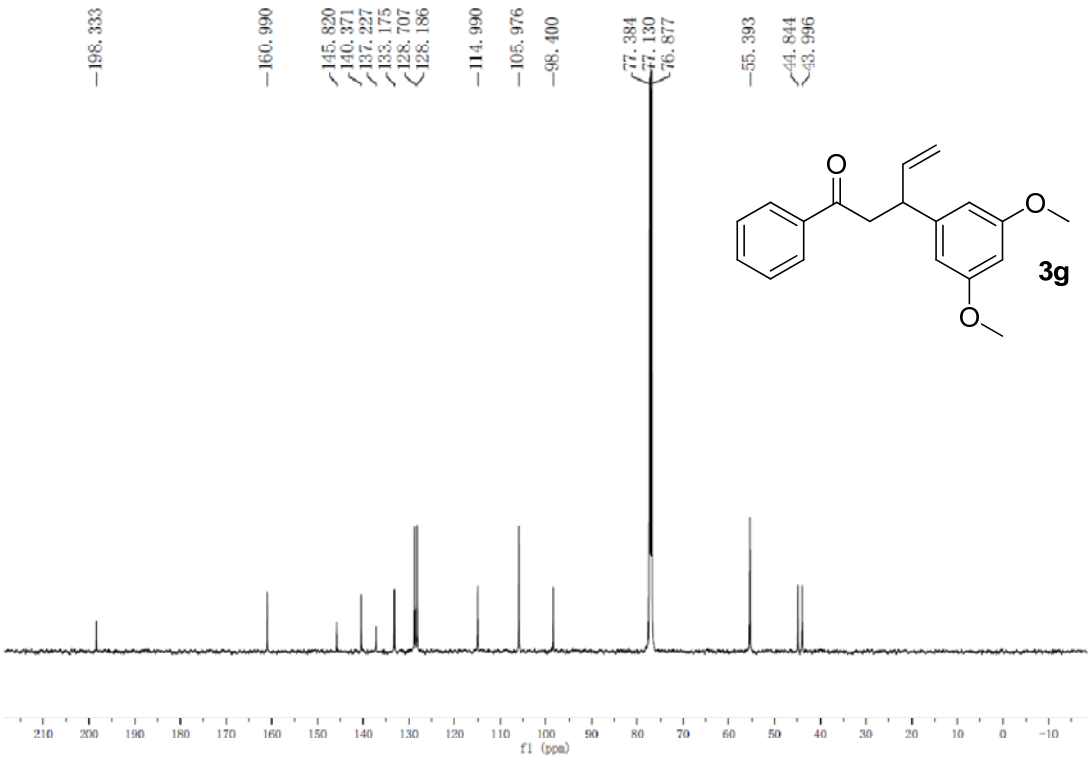
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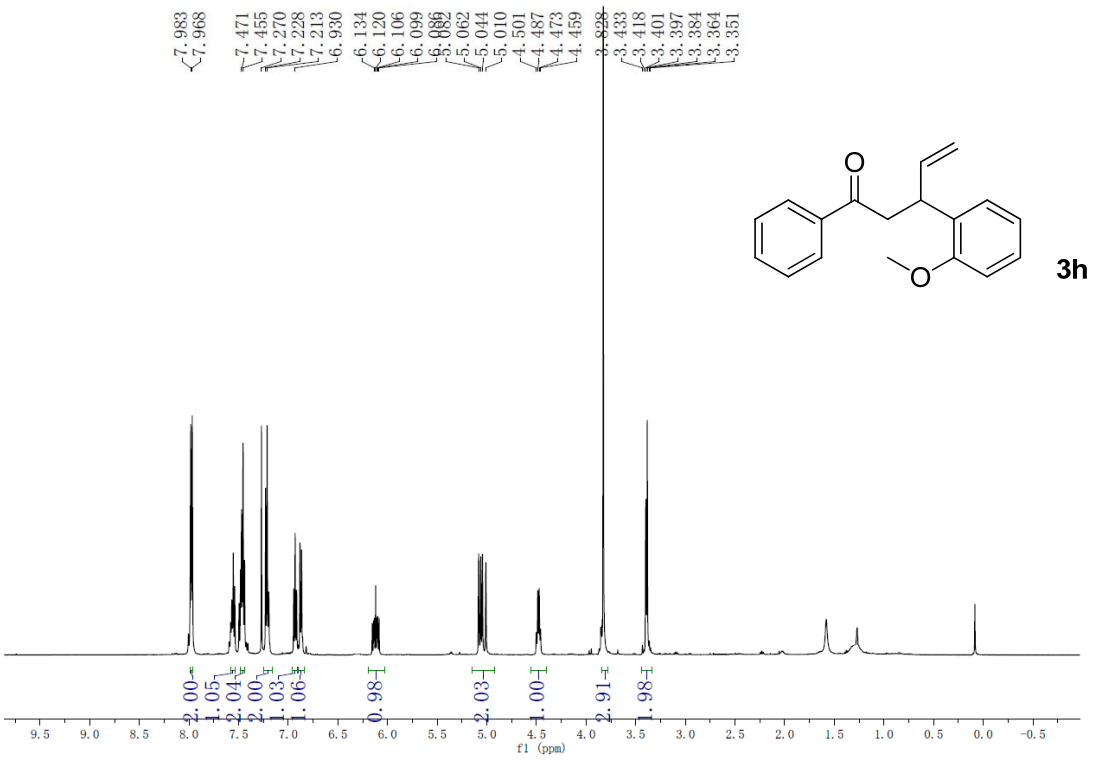
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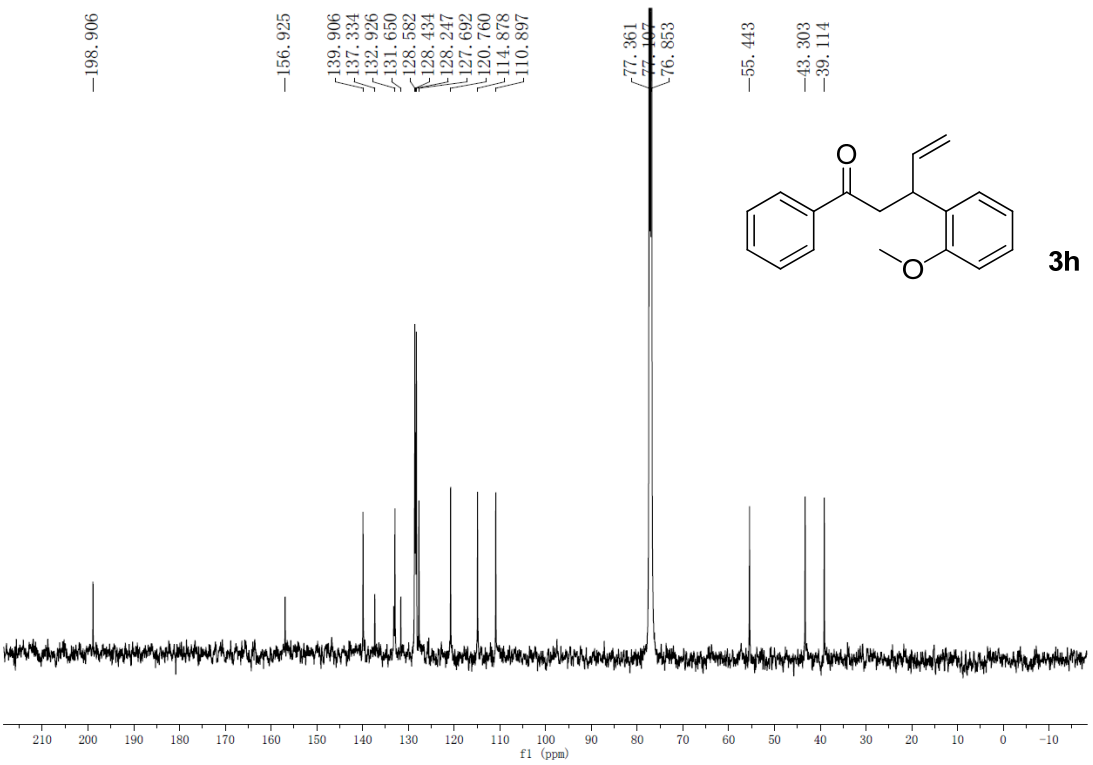
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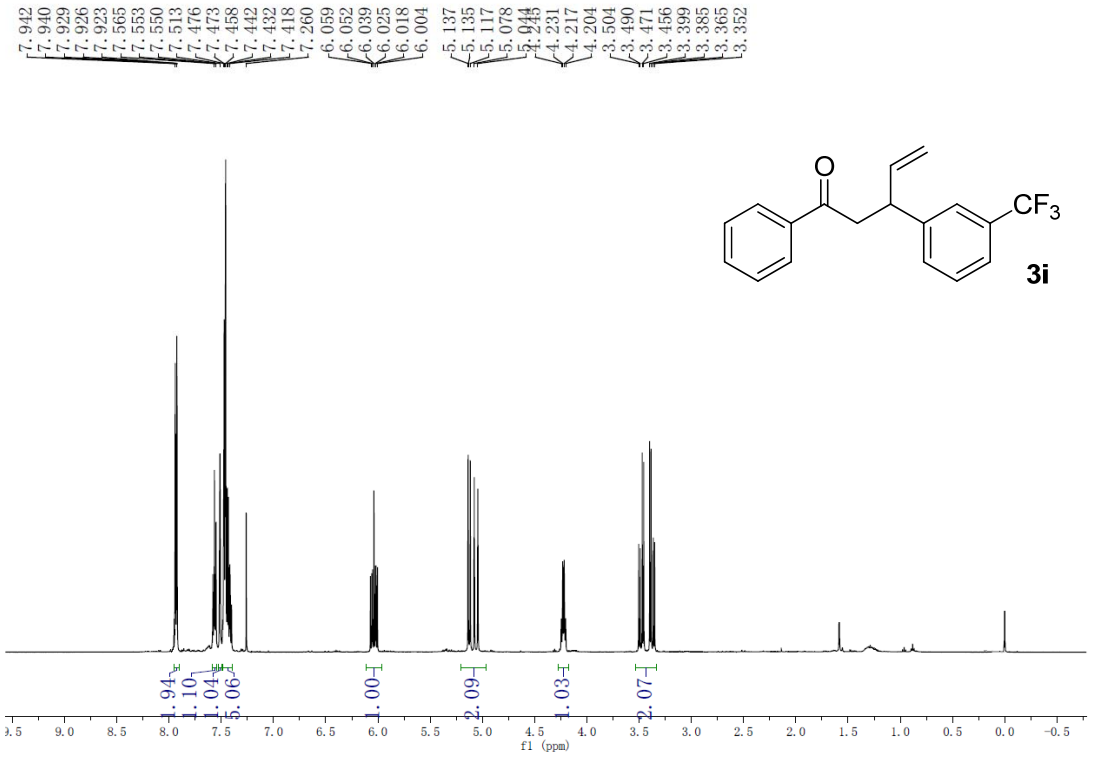
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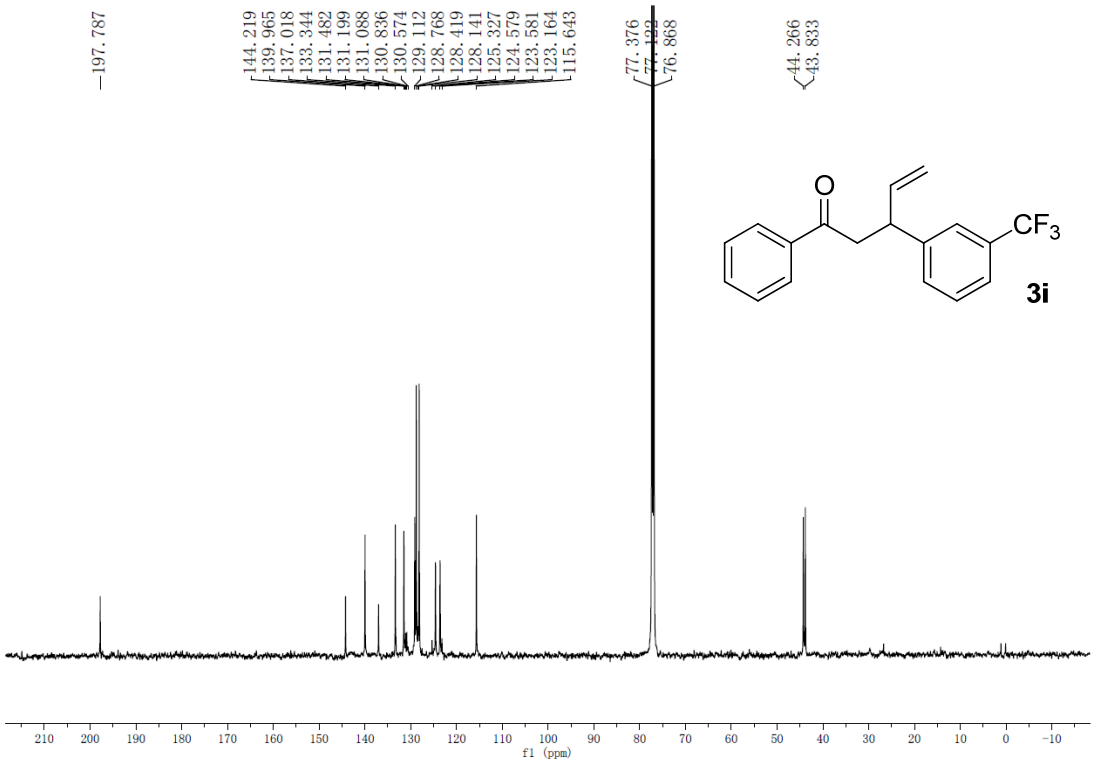
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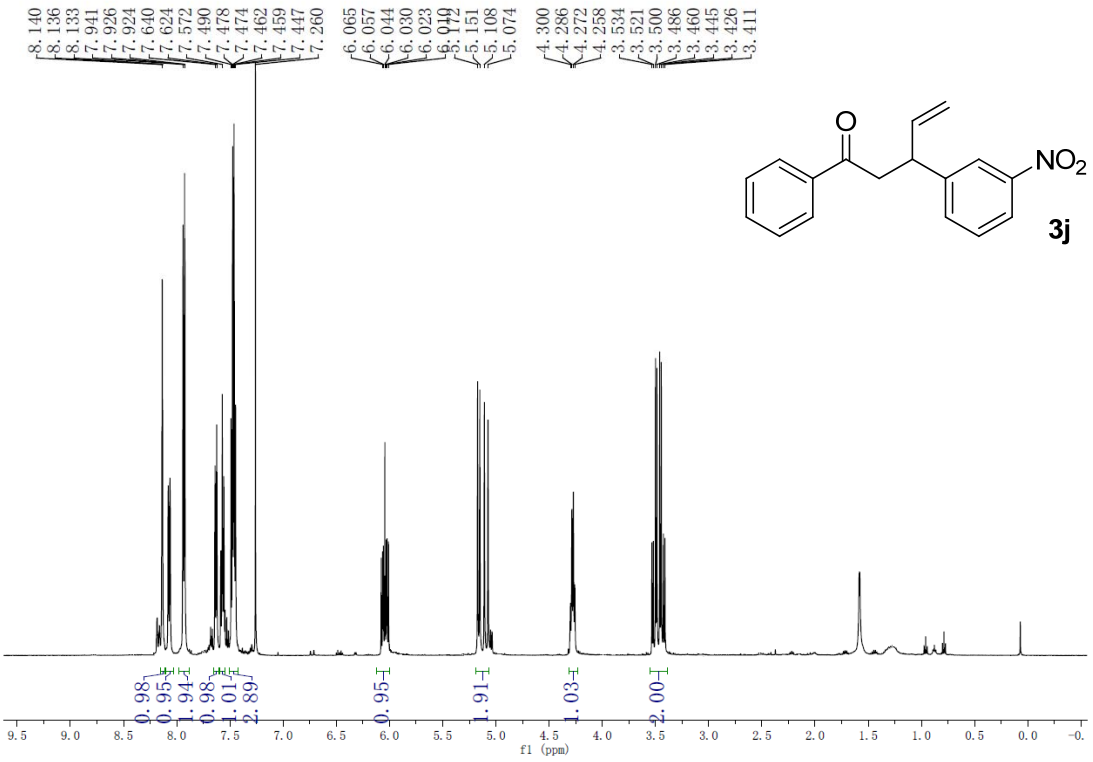
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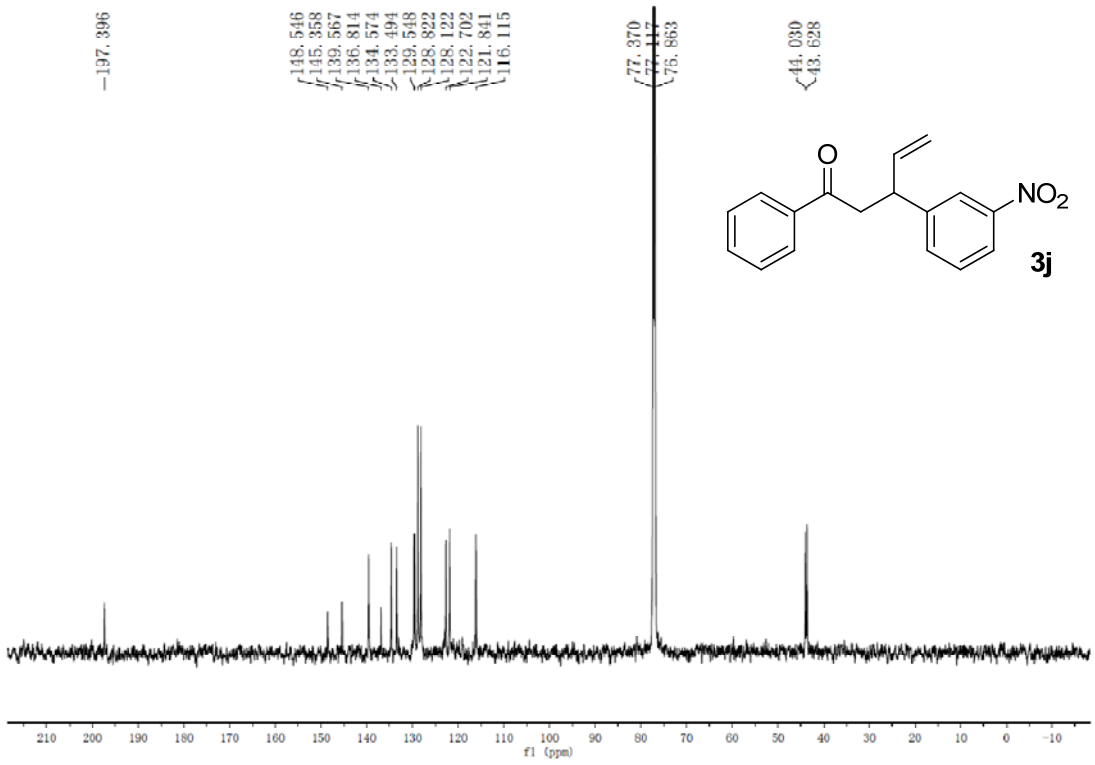
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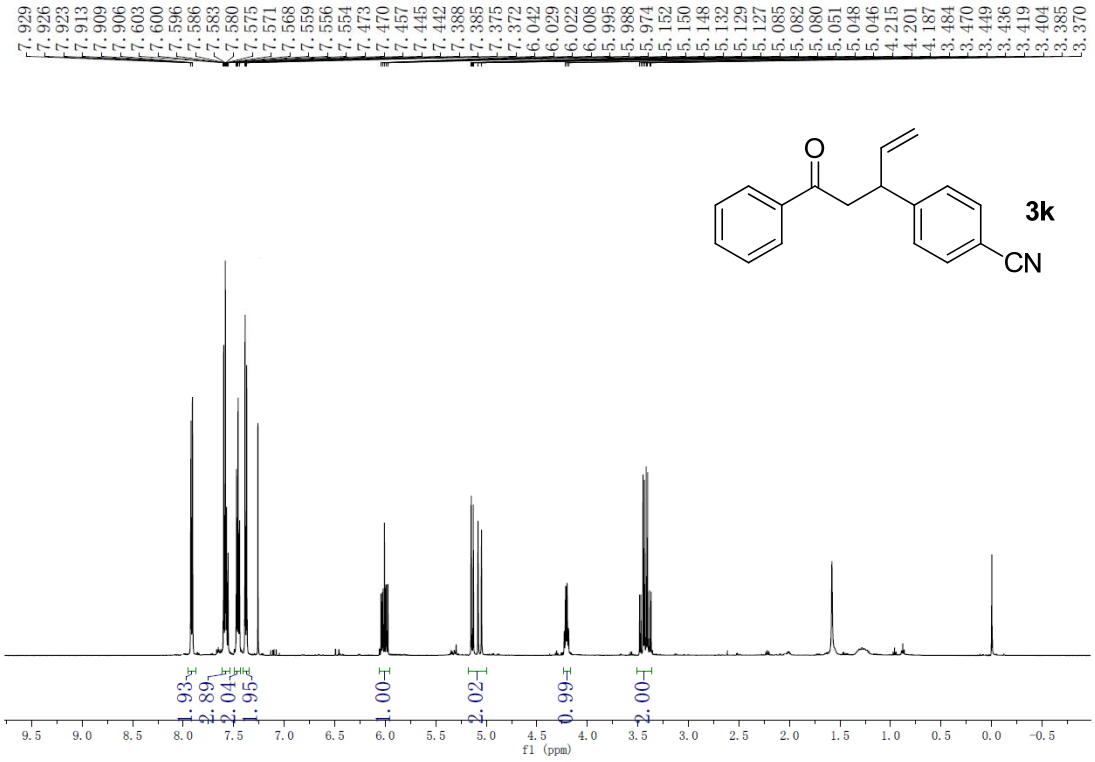
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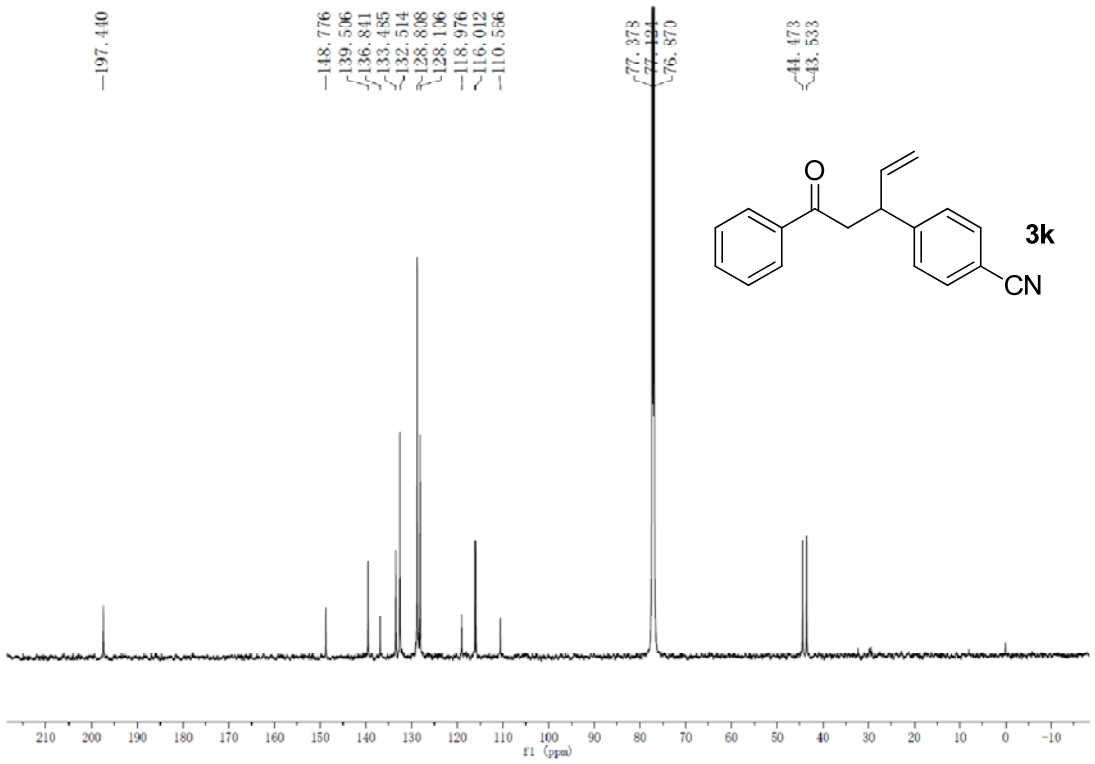
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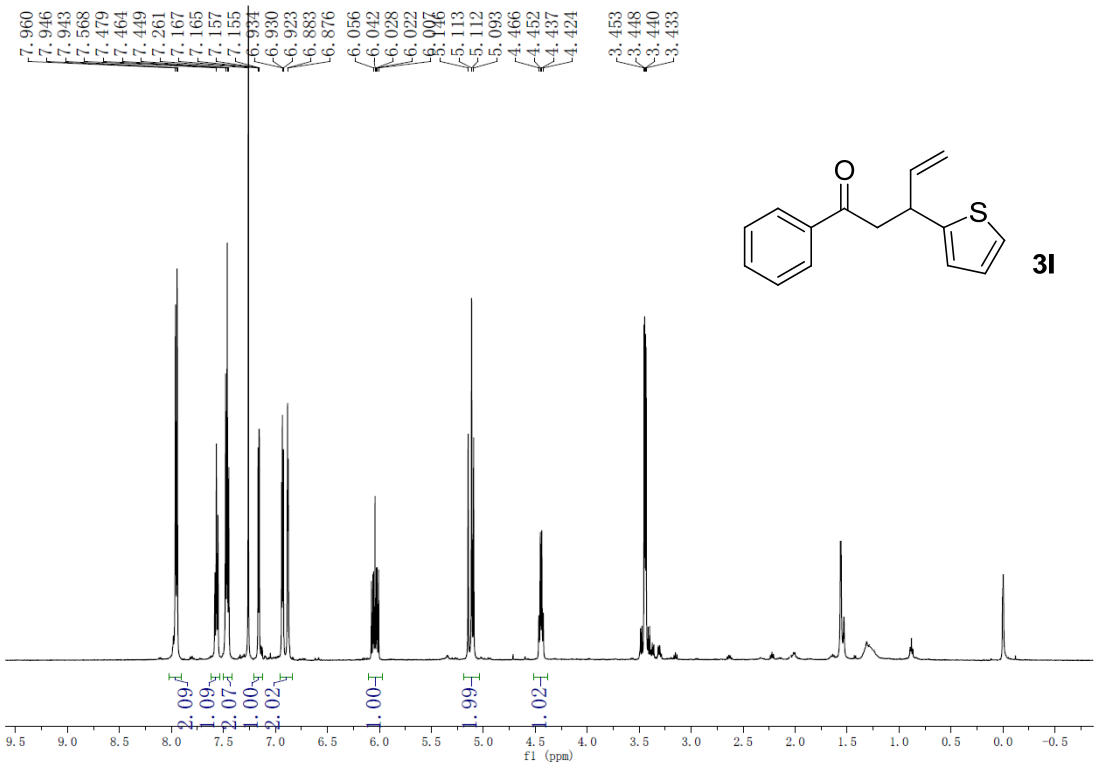
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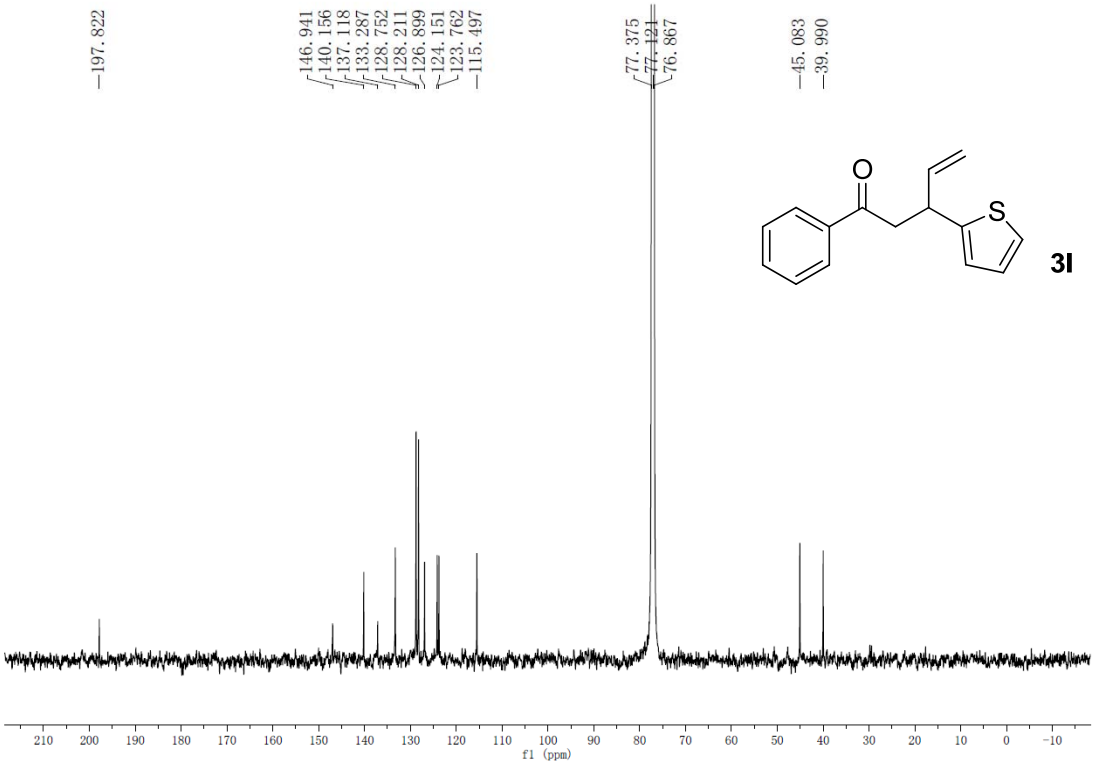
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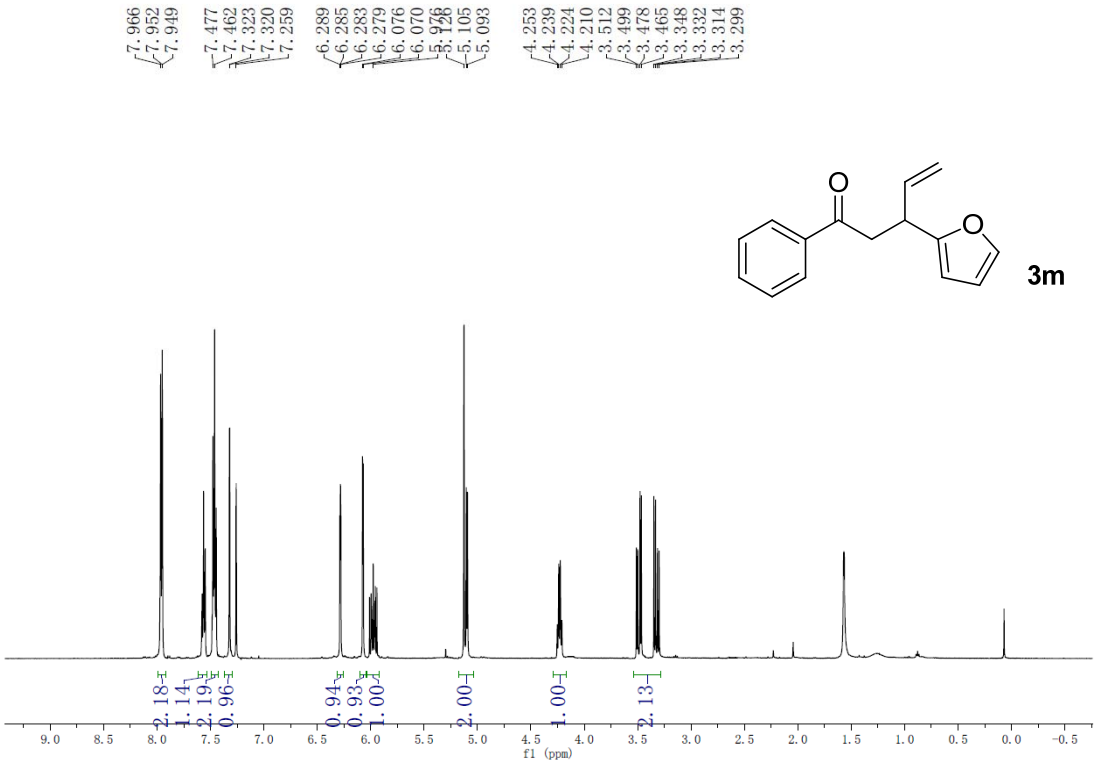
3l
f1 (ppm)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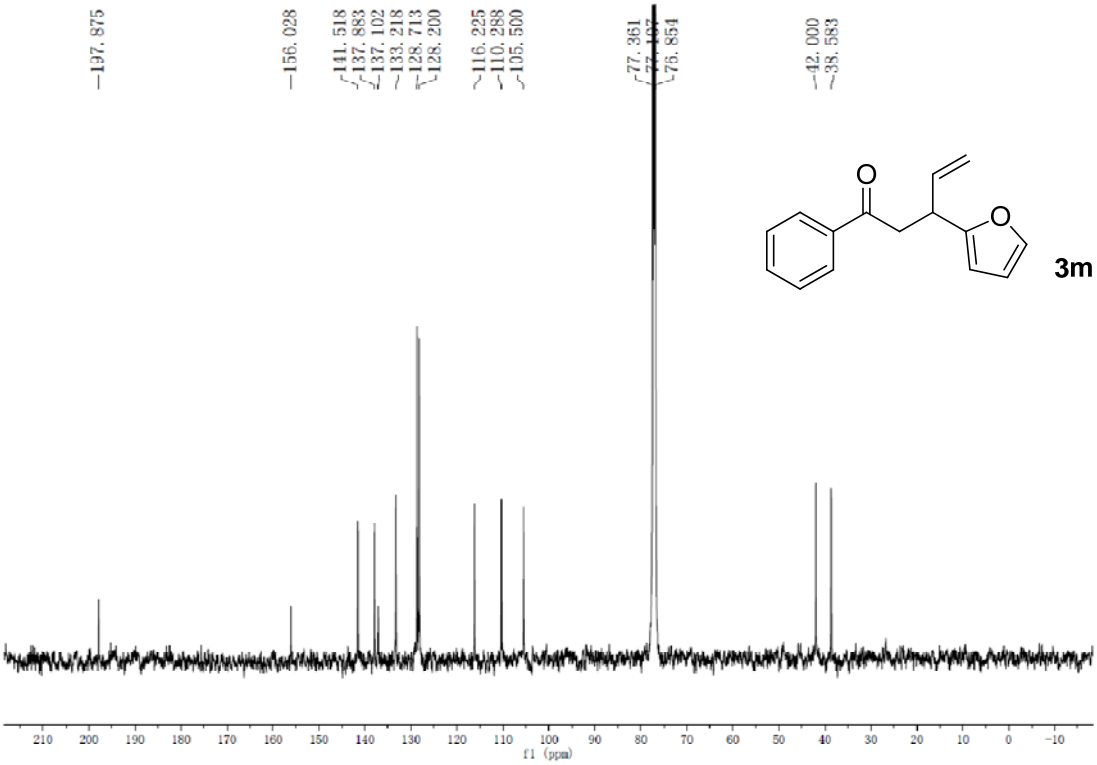
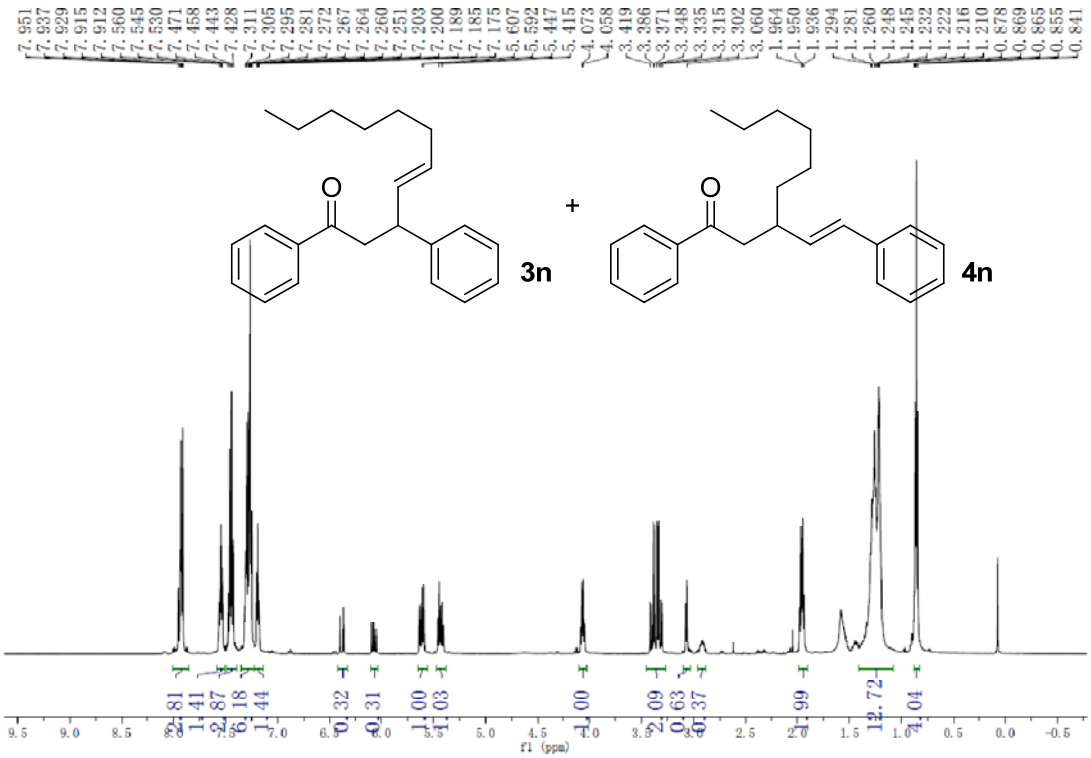
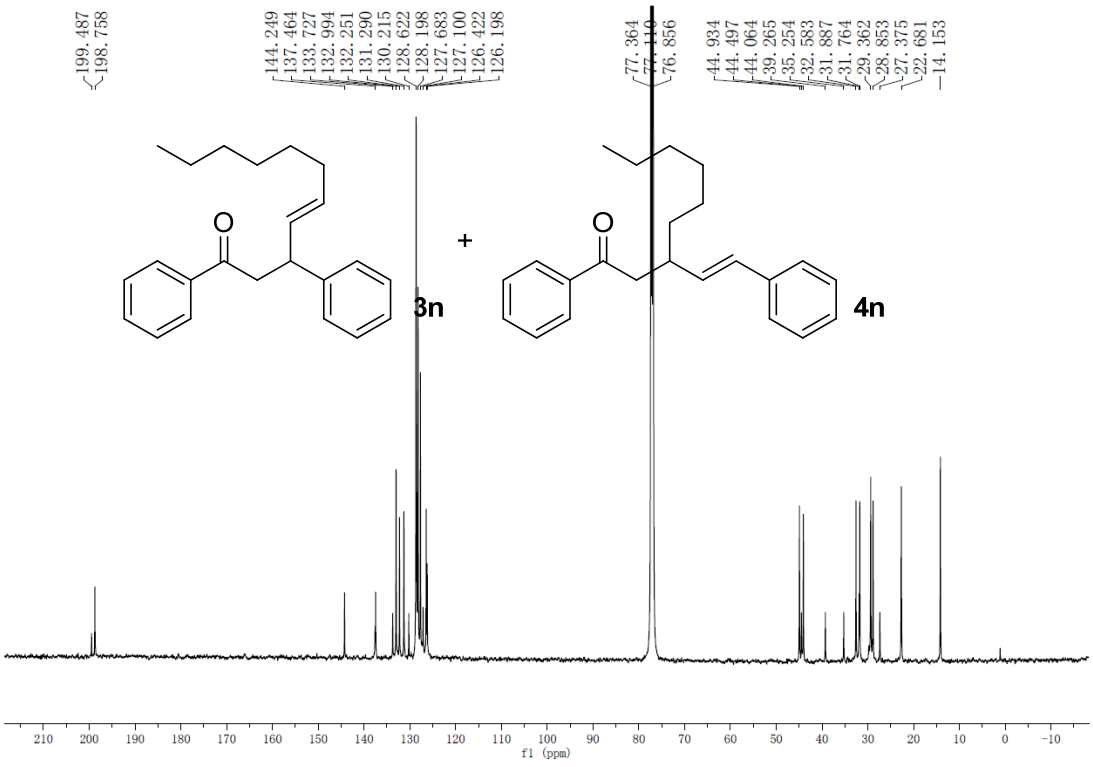
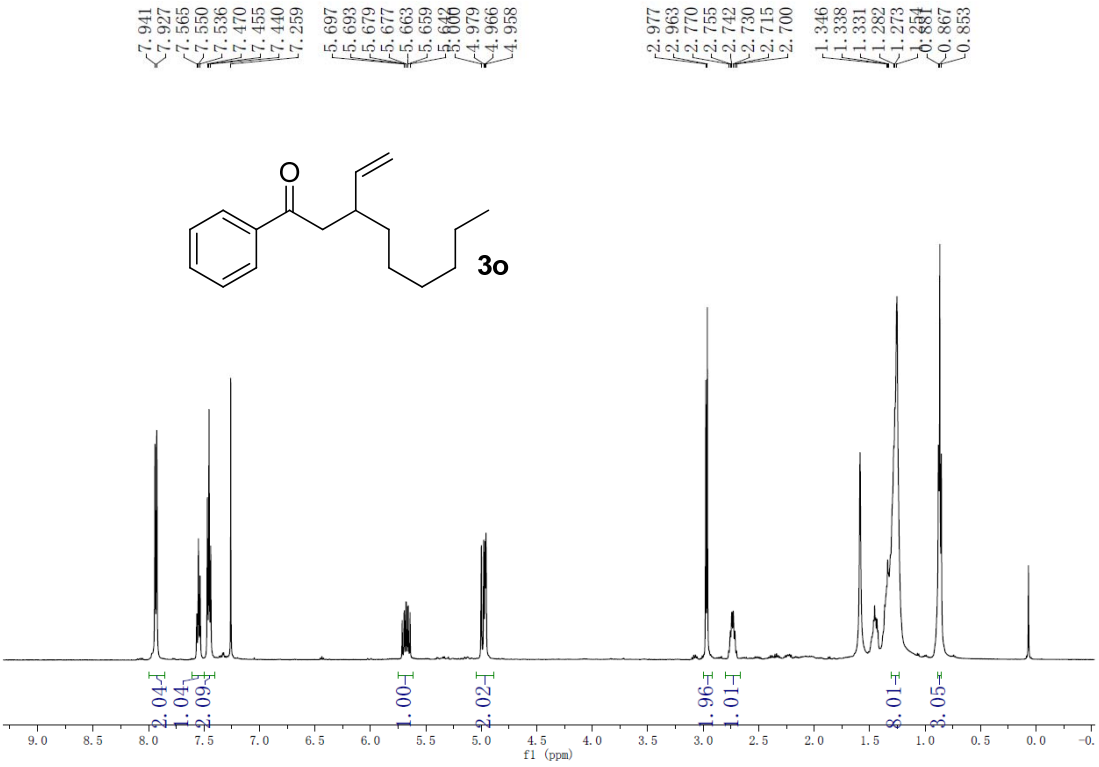
7.949
7.477
7.462
7.323
7.320
7.259
6.289
6.285
6.283
6.279
6.076
6.070
5.105
5.093
4.253
4.239
4.224
4.210
3.512
3.499
3.478
3.465
3.348
3.332
3.314
3.299
O
O
3m
2.18
1.14
2.19
0.96
0.94
0.93
1.00
2.00
1.00
2.13
f1 (ppm)


197.875
156.028
141.518
137.883
137.102
133.218
128.713
128.200
116.225
110.288
105.500
77.361
76.854
42.000
38.583
O
O
3m
f1 (ppm)


3n
4n
f1 (ppm)


3n
4n
f1 (ppm)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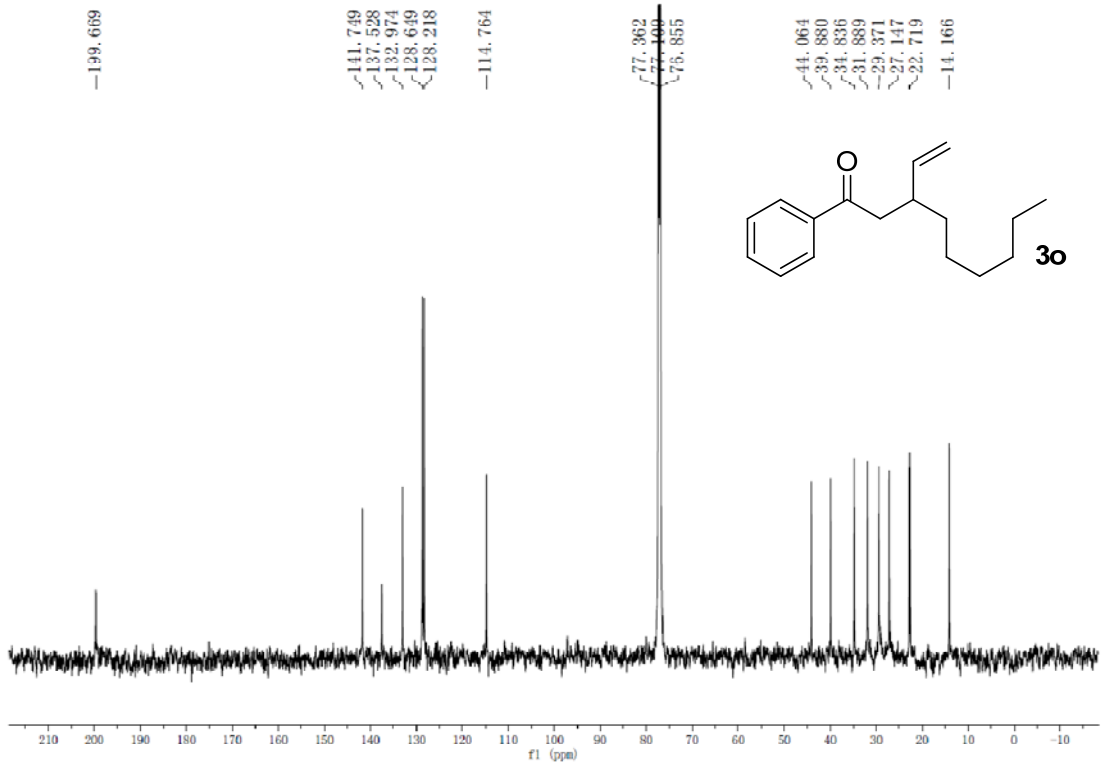
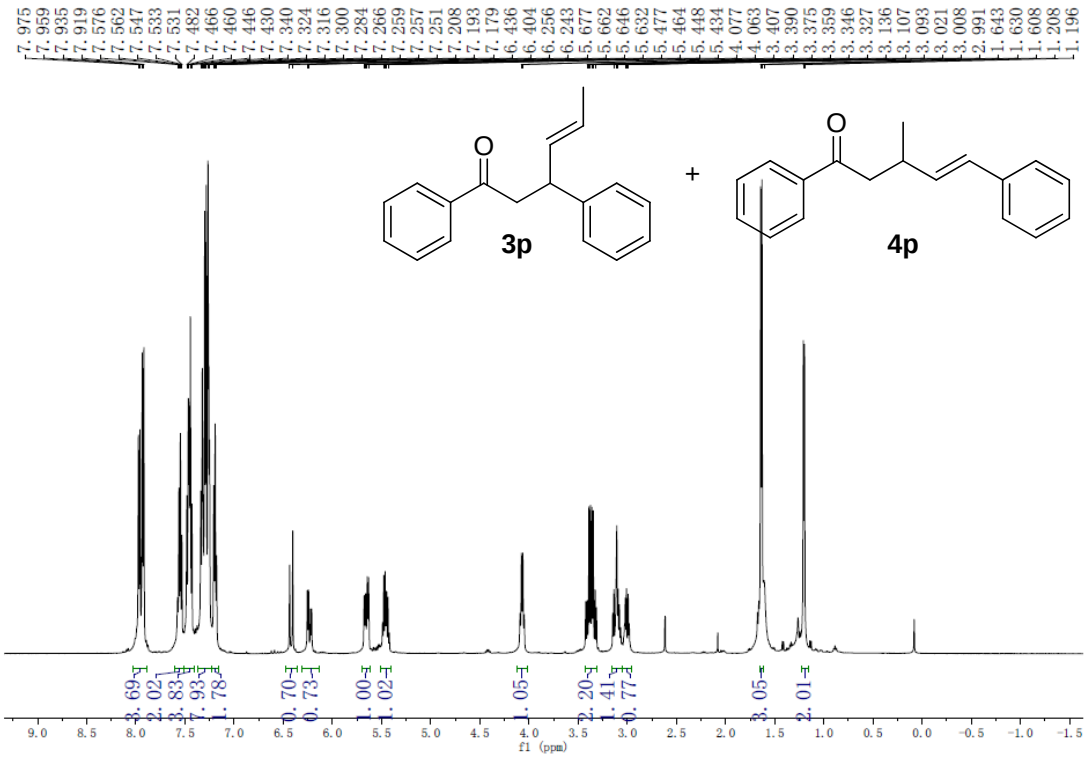
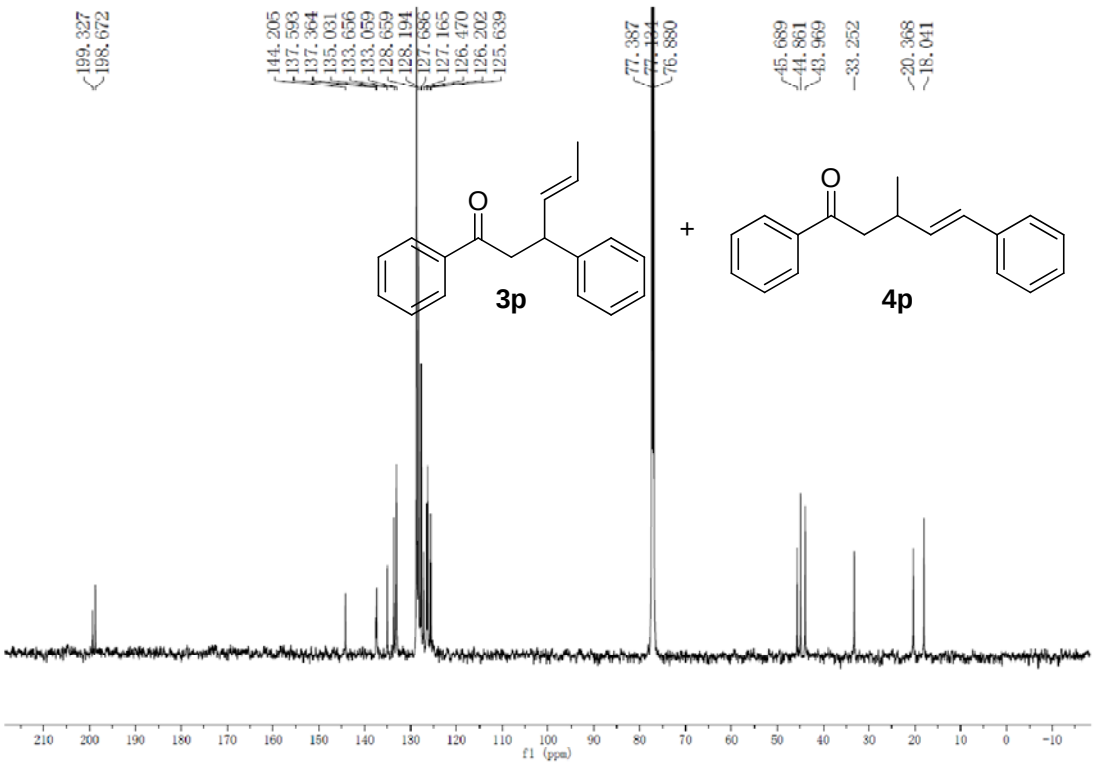
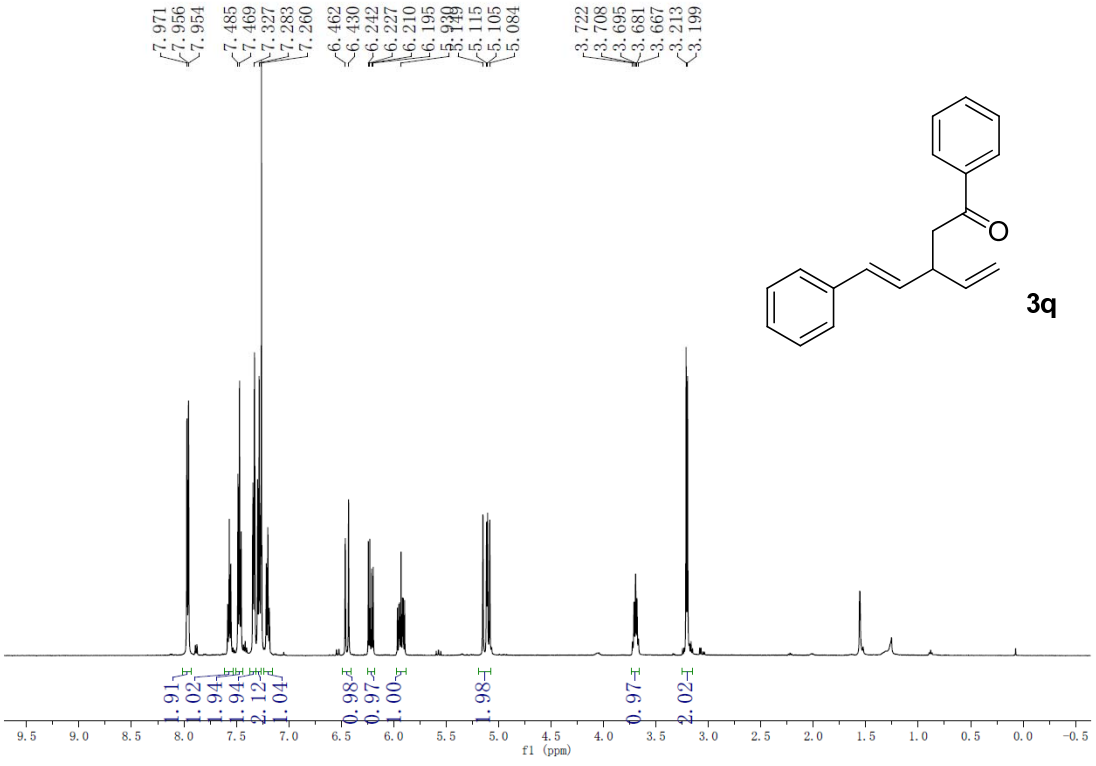
7.565
7.550
7.536
7.470
7.455
7.440
7.259
5.697
5.693
5.679
5.677
5.663
5.659
5.642
5.638
4.979
4.966
4.958
2.977
2.963
2.770
2.755
2.742
2.730
2.715
2.700
1.346
1.338
1.331
1.282
1.273
1.254
0.881
0.867
0.853
O
3o
2.04
1.04
2.09
1.00
2.02
1.96
1.01
8.01
3.05
f1 (ppm)


199.669
141.749
137.528
132.974
128.649
128.218
114.764
77.362
77.100
76.855
44.064
39.880
34.836
31.889
29.371
27.147
22.719
14.166
O
3o
f1 (ppm)


3p
4p
f1 (ppm)


3p
4p
f1 (ppm)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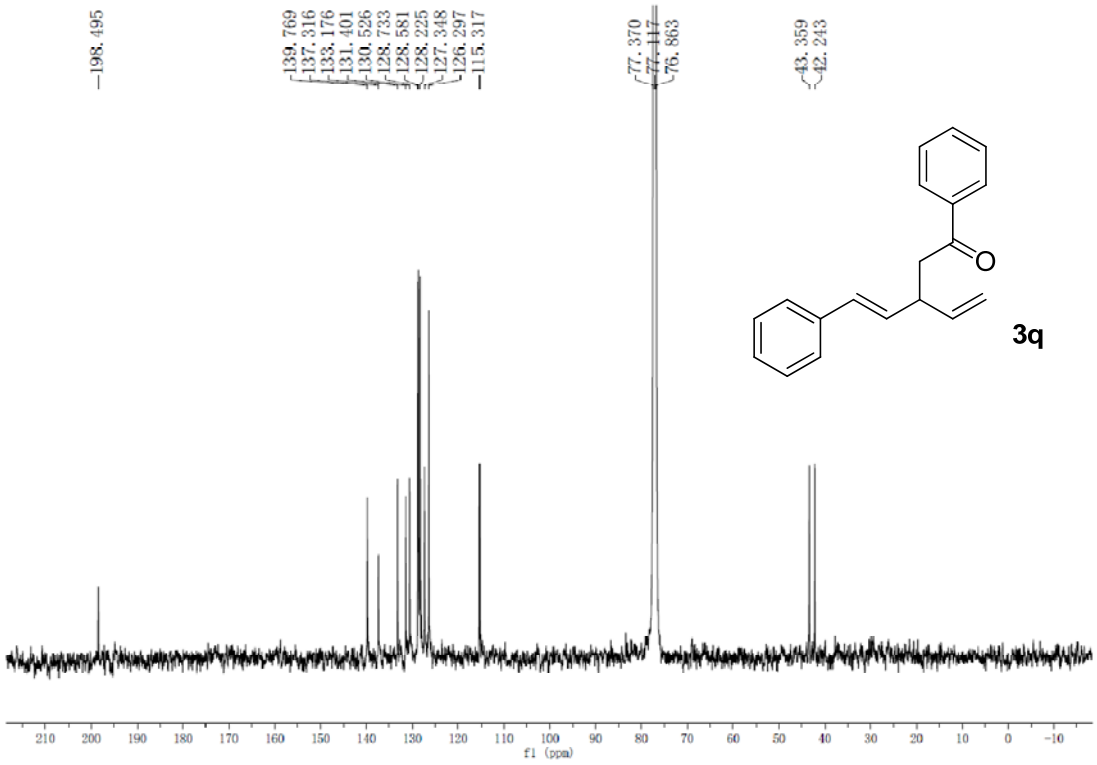
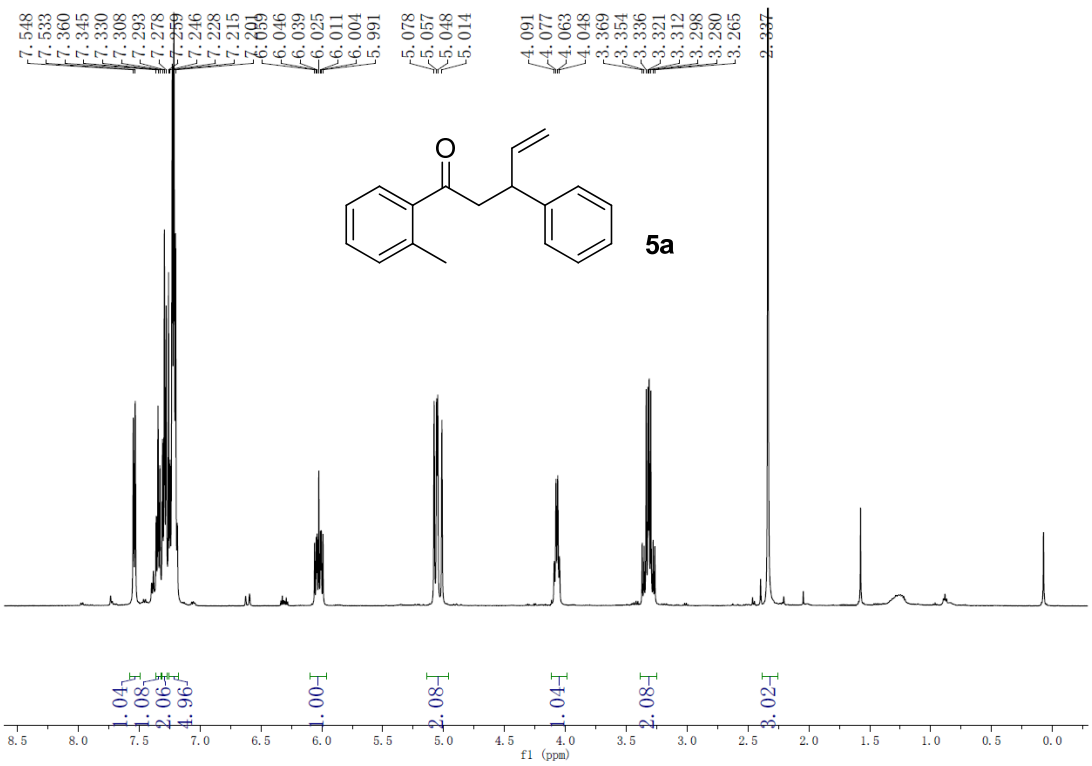
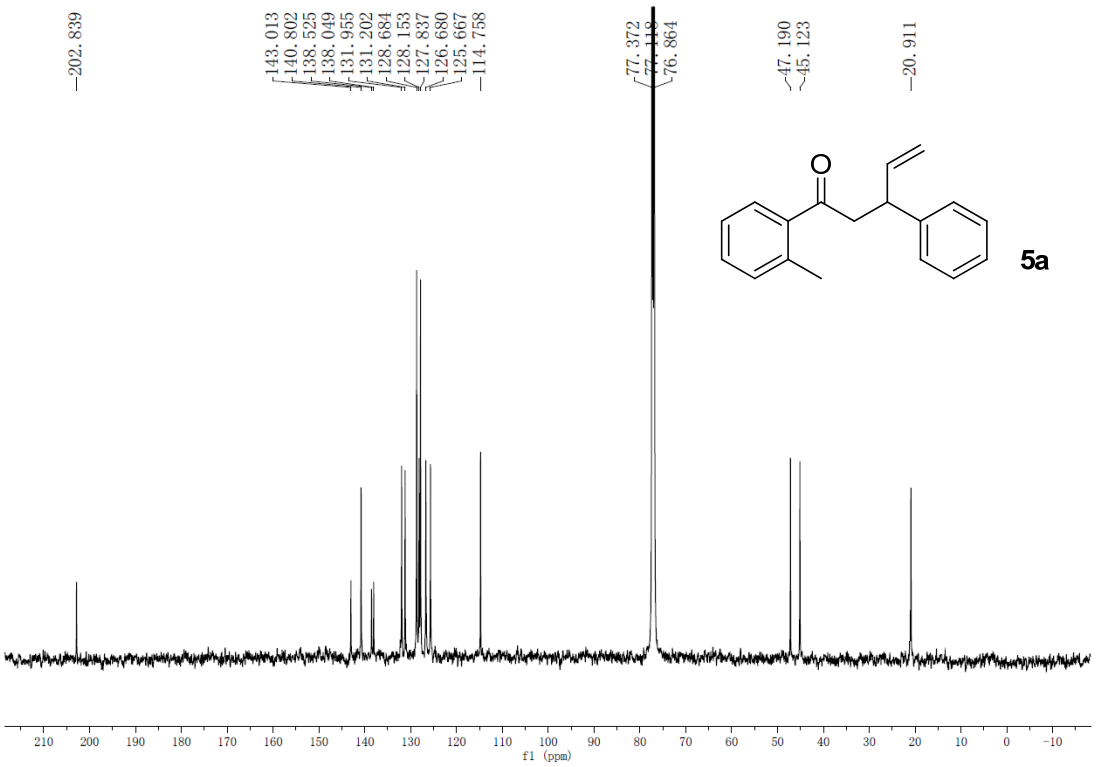
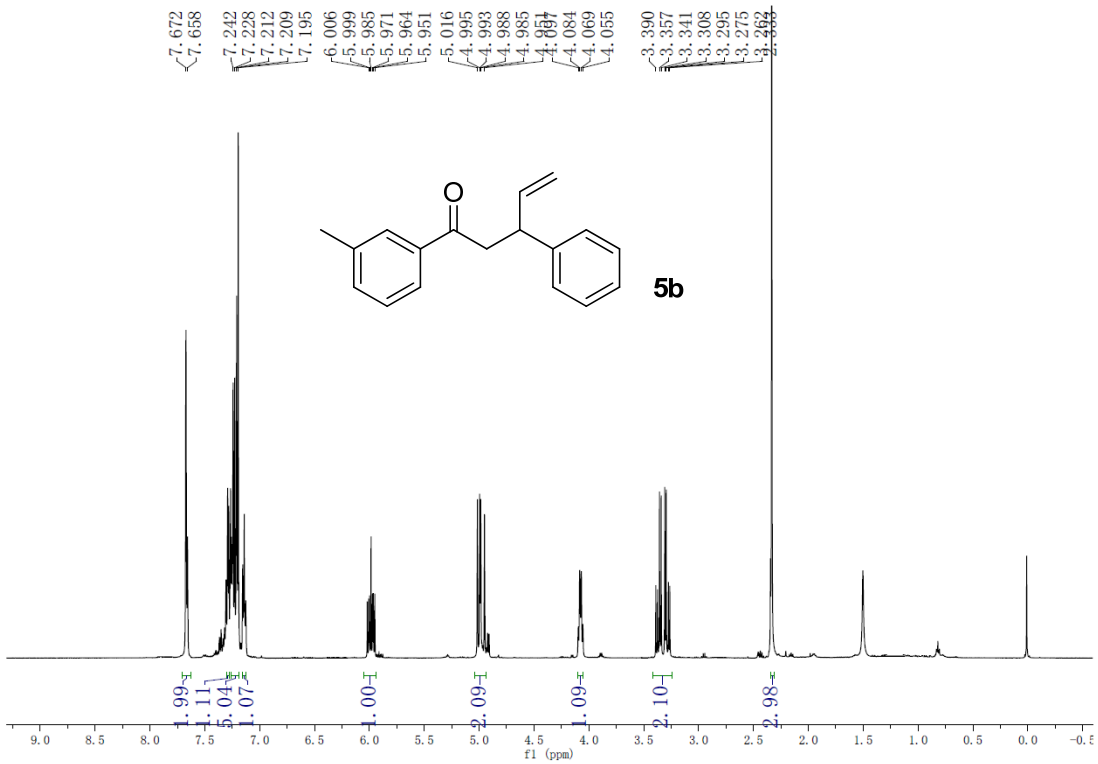
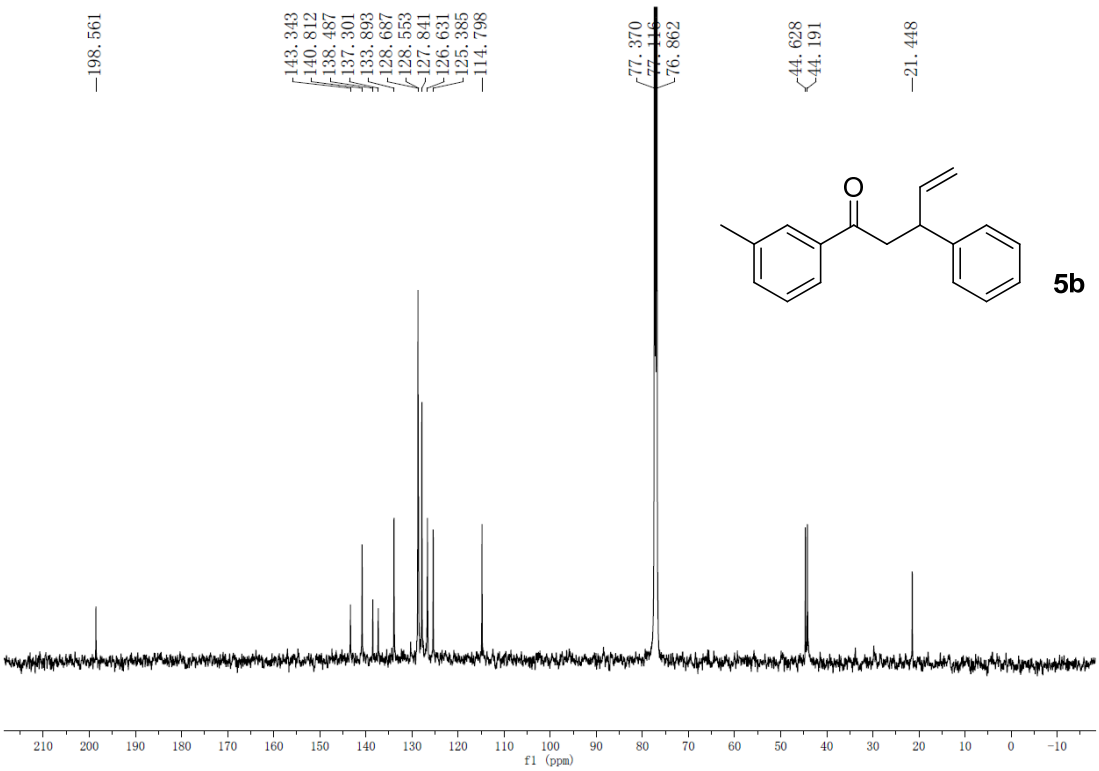
7.327
7.283
7.260
6.462
6.430
6.242
6.227
6.210
6.195
5.115
5.105
5.084
3.722
3.708
3.695
3.681
3.667
3.213
3.199
1.91
1.02
1.94
1.94
2.12
1.04
0.98
0.97
1.00
1.98
0.97
2.02
f1 (ppm)
O
3q


198.495
139.769
137.316
133.176
131.401
130.526
128.733
128.581
128.225
127.348
126.297
115.317
77.370
77.117
76.863
43.359
42.243
f1 (ppm)
O
3q


7.548
7.533
7.360
7.345
7.330
7.308
7.293
7.278
7.259
7.246
7.228
7.215
6.046
6.039
6.025
6.011
6.004
5.991
5.078
5.057
5.048
5.014
4.091
4.077
4.063
4.048
3.369
3.354
3.336
3.321
3.312
3.298
3.280
3.265
2.337
O
5a
1.04
1.08
2.06
4.96
1.00
2.08
1.04
2.08
3.02
f1 (ppm)


202.839
143.013
140.802
138.525
138.049
131.955
131.202
128.684
128.153
127.837
126.680
125.667
114.758
77.372
77.118
76.864
47.190
45.123
20.911
O
5a
f1 (ppm)


7.672
7.658
7.242
7.228
7.212
7.209
7.195
6.006
5.999
5.985
5.971
5.964
5.951
5.016
4.995
4.993
4.988
4.985
4.084
4.069
4.055
3.390
3.357
3.341
3.308
3.295
3.275
2.383
O
5b
1.99
1.11
5.04
1.07
1.00
2.09
1.09
2.10
2.98
f1 (ppm)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138.487
137.301
133.893
128.687
128.553
127.841
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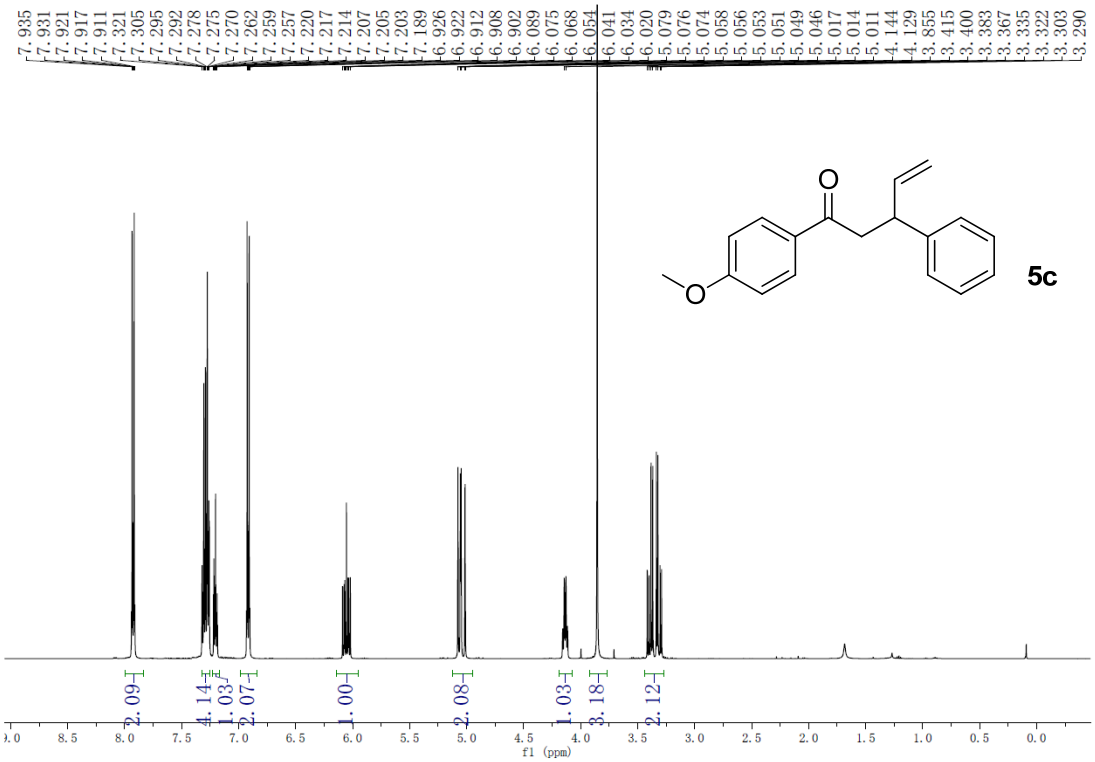
114.798
77.370
77.116
76.862
44.628
44.191
21.448
O
5b
f1 (ppm)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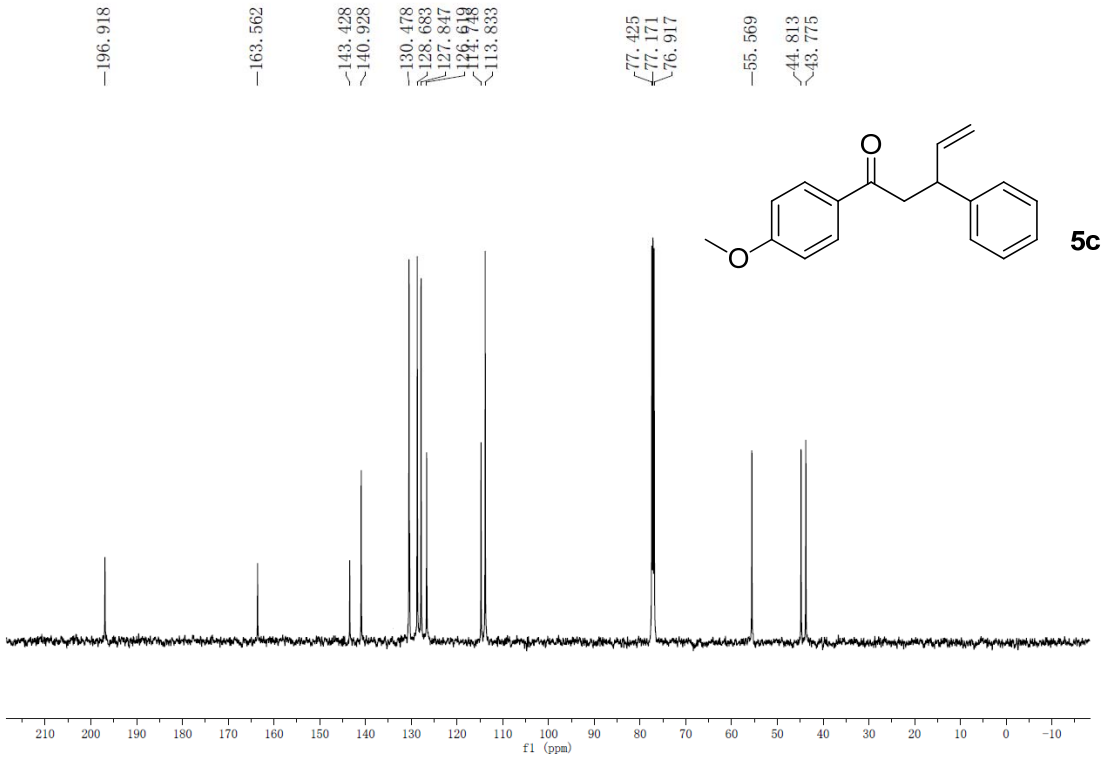
7.921
7.917
7.911
7.321
7.305
7.295
7.292
7.278
7.275
7.270
7.262
7.259
7.257
7.220
7.217
7.214
7.207
7.205
7.203
7.189
6.926
6.922
6.912
6.908
6.902
6.089
6.075
6.068
6.054
6.041
6.034
6.020
5.079
5.076
5.074
5.058
5.056
5.053
5.051
5.049
5.046
5.017
5.014
5.011
4.144
4.129
3.855
3.415
3.400
3.383
3.367
3.335
3.322
3.303
3.290
2.09
4.14
1.03
2.07
1.00
2.08
1.03
3.18
2.12
f1 (ppm)
5c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130.478
128.683
127.847
126.619
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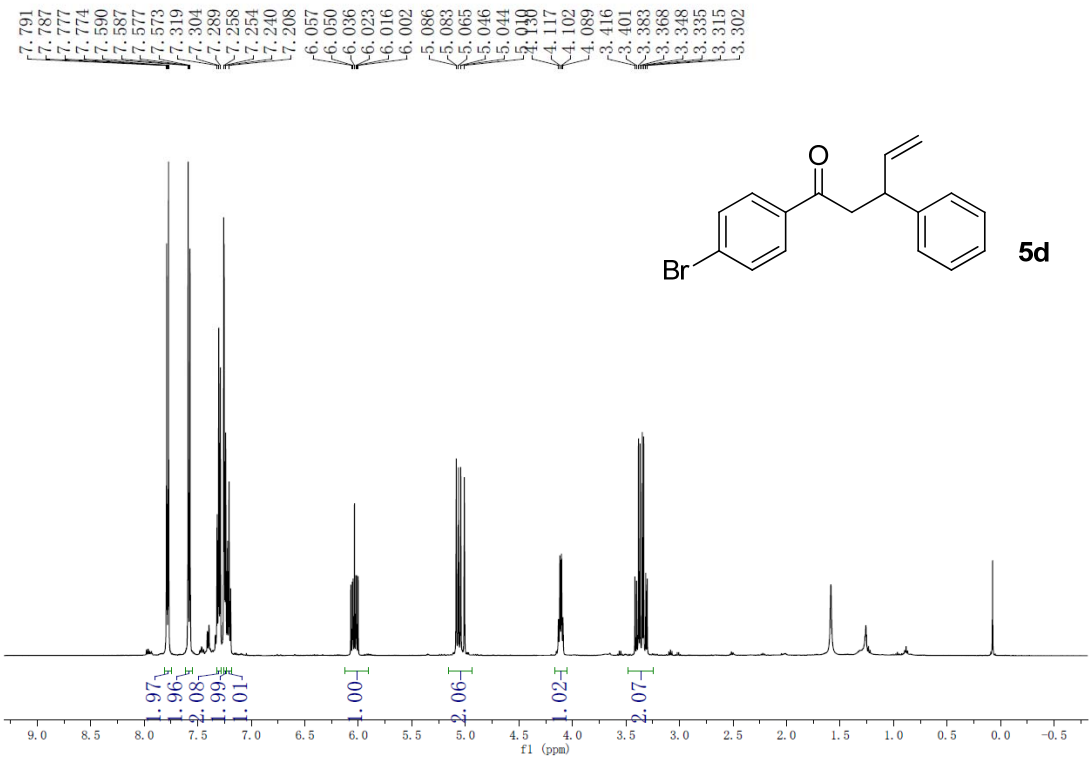
113.833
77.425
77.171
76.917
55.569
44.813
43.775
f1 (ppm)
5c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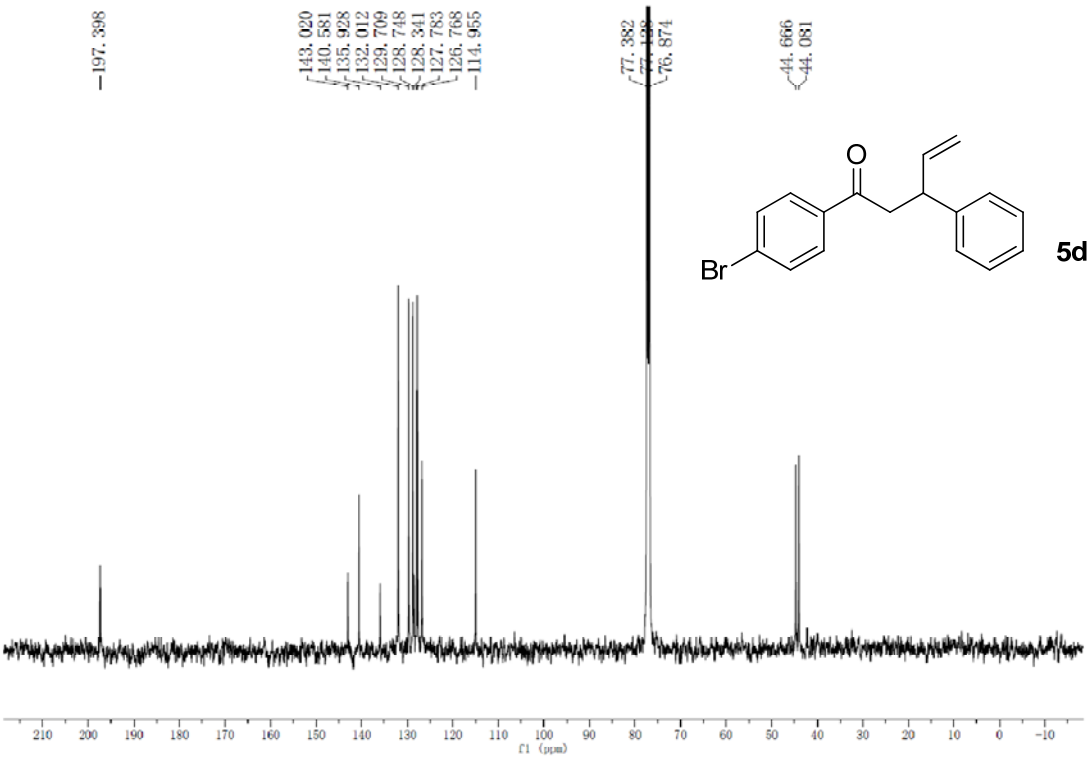
7.587
7.577
7.573
7.319
7.304
7.289
7.258
7.254
7.240
7.208
6.057
6.050
6.036
6.023
6.016
6.002
5.086
5.083
5.065
5.046
5.044
4.117
4.102
4.089
3.416
3.401
3.383
3.368
3.348
3.335
3.315
3.302
O
Br
5d
1.97
1.96
2.08
1.99
1.01
1.00
2.06
1.02
2.07
f1 (ppm)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132.012
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126.768
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77.382
76.874
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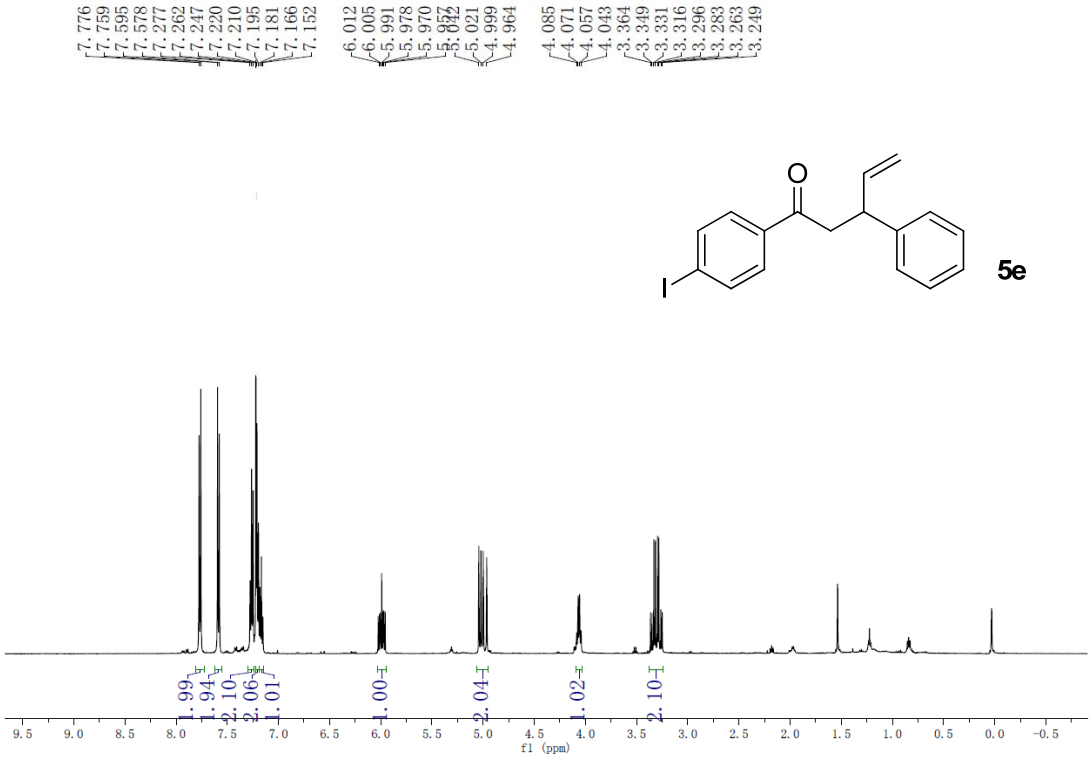
44.081
O
Br
5d
f1 (ppm)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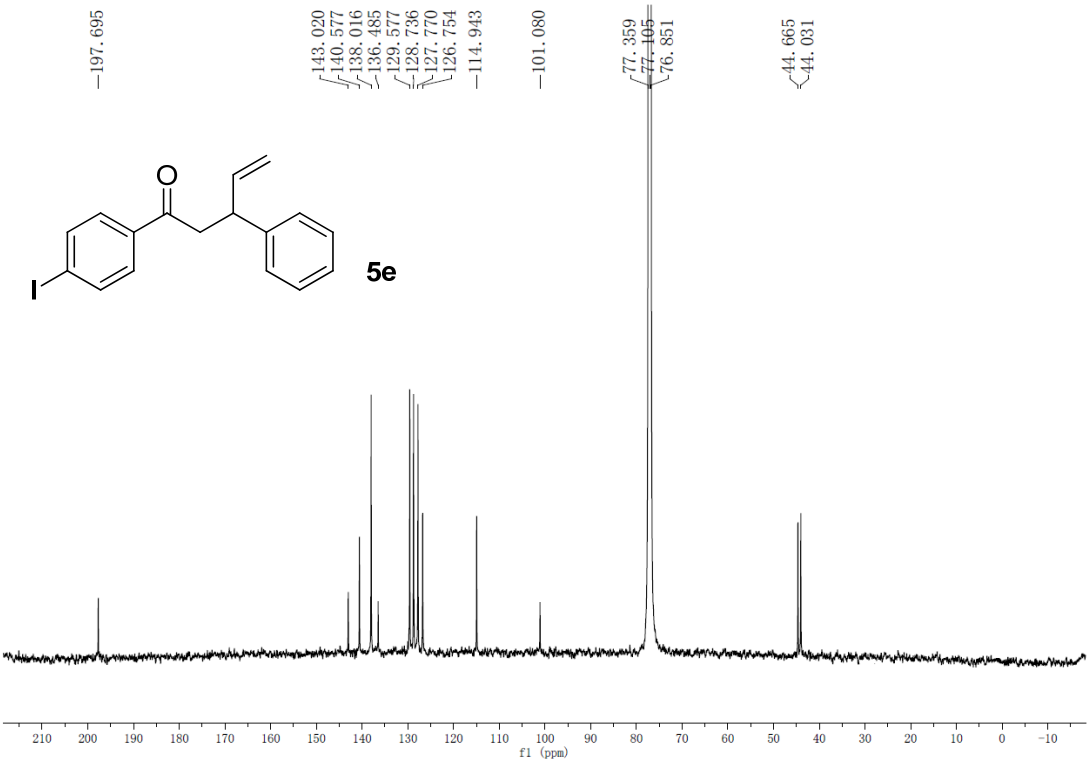
7.247
7.220
7.210
7.195
7.181
7.166
7.152
6.012
6.005
5.991
5.978
5.970
5.957
5.042
5.021
4.999
4.964
4.085
4.071
4.057
4.043
3.364
3.349
3.331
3.316
3.296
3.283
3.263
3.249
O
I
5e
1.99
1.94
2.10
2.06
1.01
1.00
2.04
1.02
2.10
f1 (ppm)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101.080
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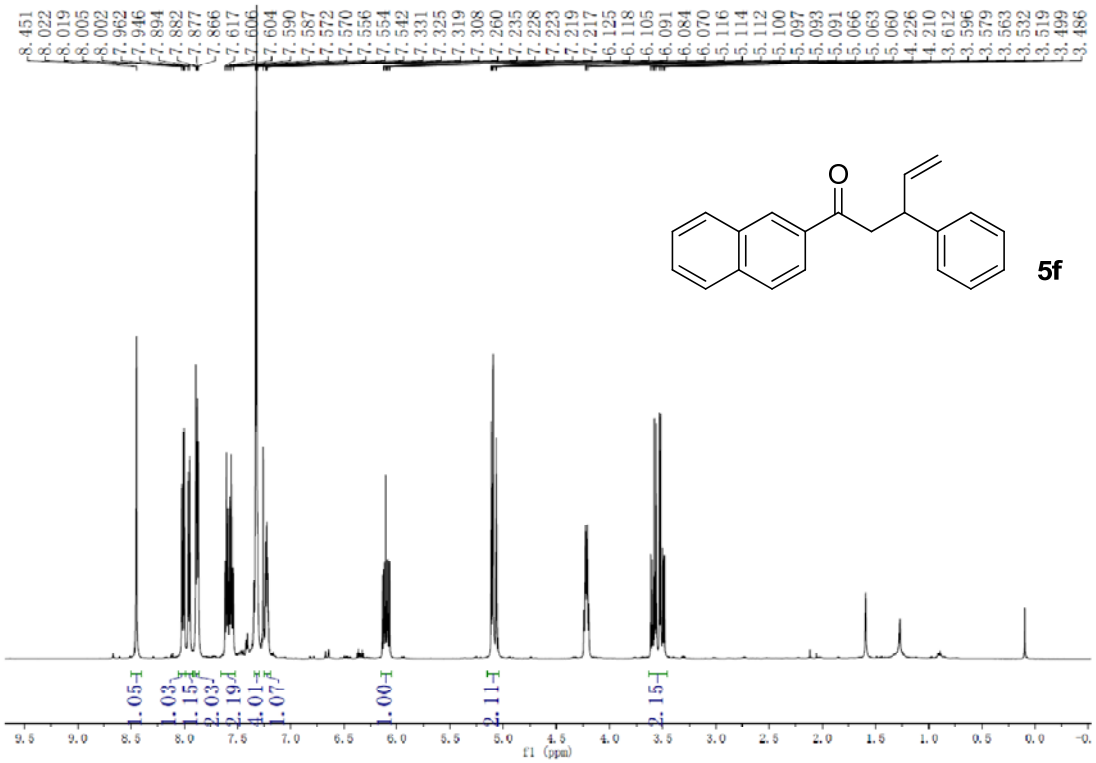
77.105
76.851
44.665
44.031
O
I
5e
f1 (ppm)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7.894
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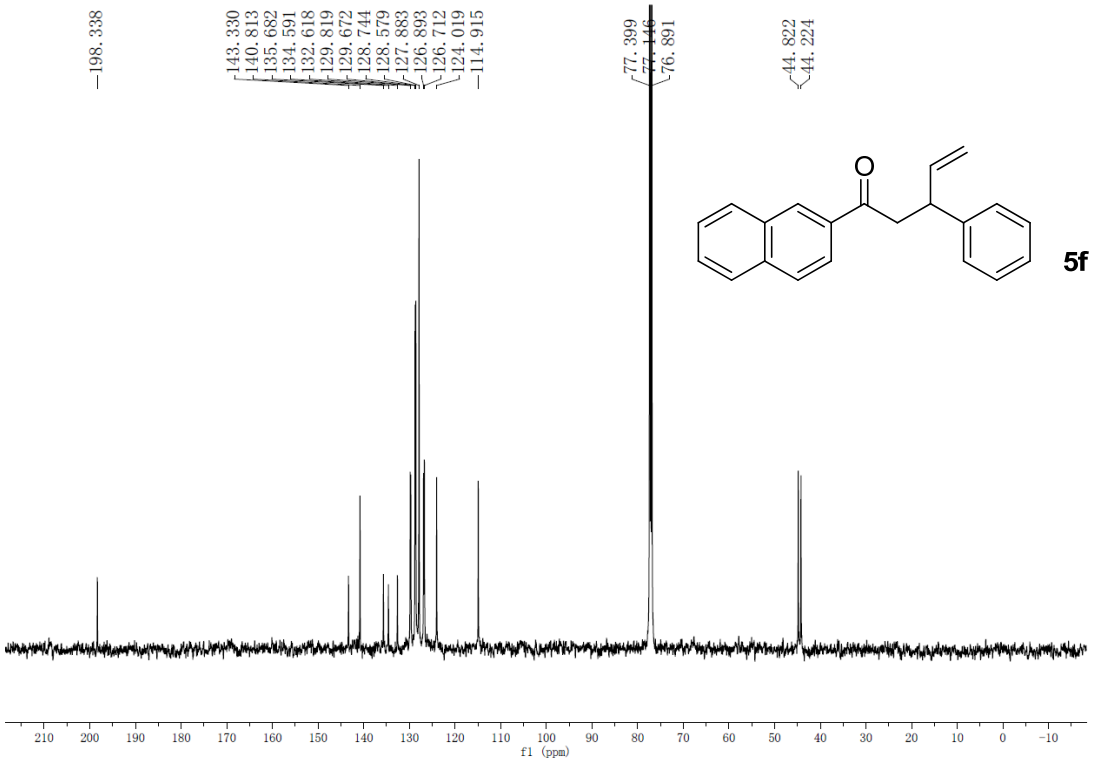
7.606
7.604
7.590
7.587
7.572
7.570
7.556
7.554
7.542
7.331
7.325
7.319
7.308
7.260
7.235
7.228
7.223
7.219
7.217
6.125
6.118
6.105
6.091
6.084
6.070
5.116
5.114
5.112
5.100
5.097
5.093
5.091
5.066
5.063
5.060
4.226
4.210
3.612
3.596
3.579
3.563
3.532
3.519
3.499
3.486
O
5f
1.05
1.03
1.15
2.03
2.19
4.01
1.07
1.00
2.11
2.15
f1 (ppm)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129.672
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128.579
127.883
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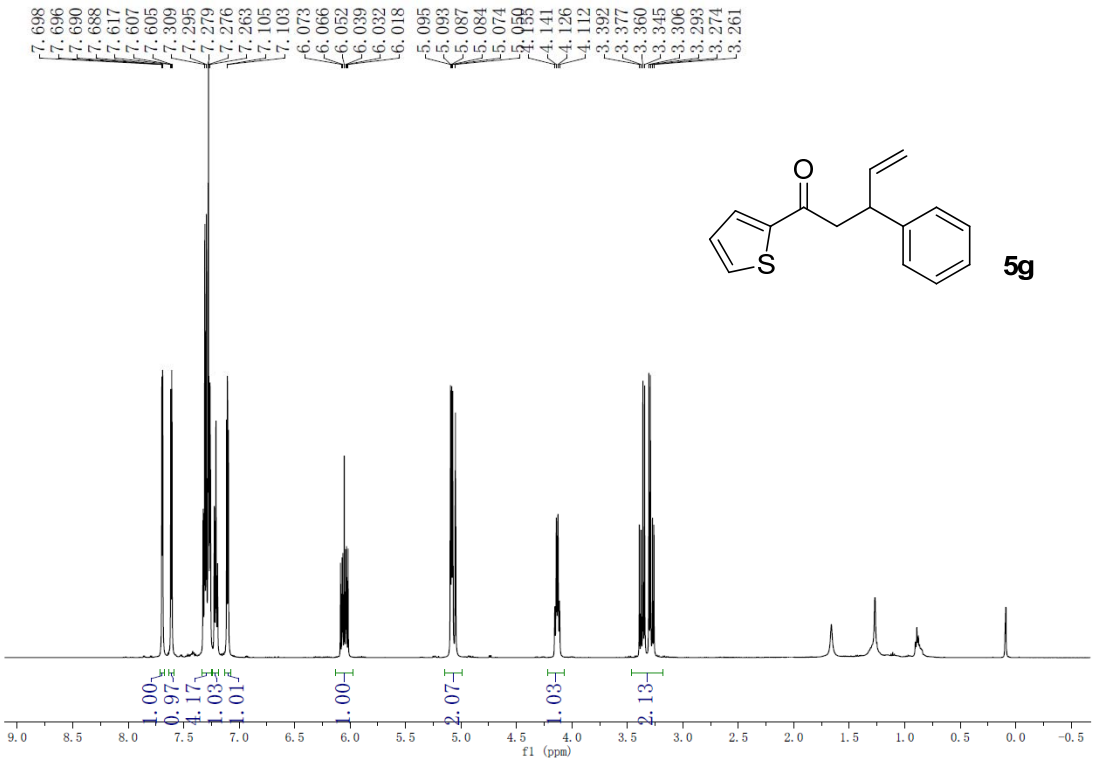
126.712
124.019
114.915
77.399
77.146
76.891
44.822
44.224
O
5f
f1 (ppm)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7.309
7.295
7.279
7.276
7.263
7.105
7.103
6.073
6.066
6.052
6.039
6.032
6.018
5.095
5.093
5.087
5.084
5.074
4.141
4.126
4.112
3.392
3.377
3.360
3.345
3.306
3.293
3.274
3.261
O
S
5g
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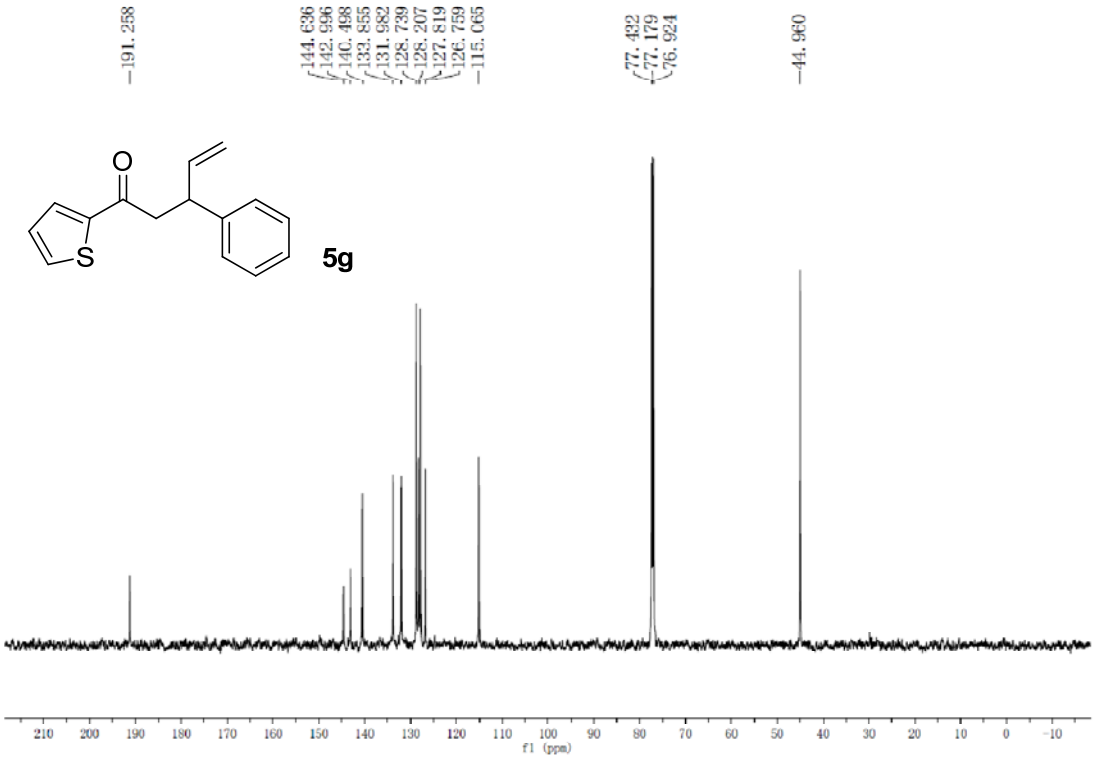
1.01
1.00
2.07
1.03
2.13
f1 (ppm)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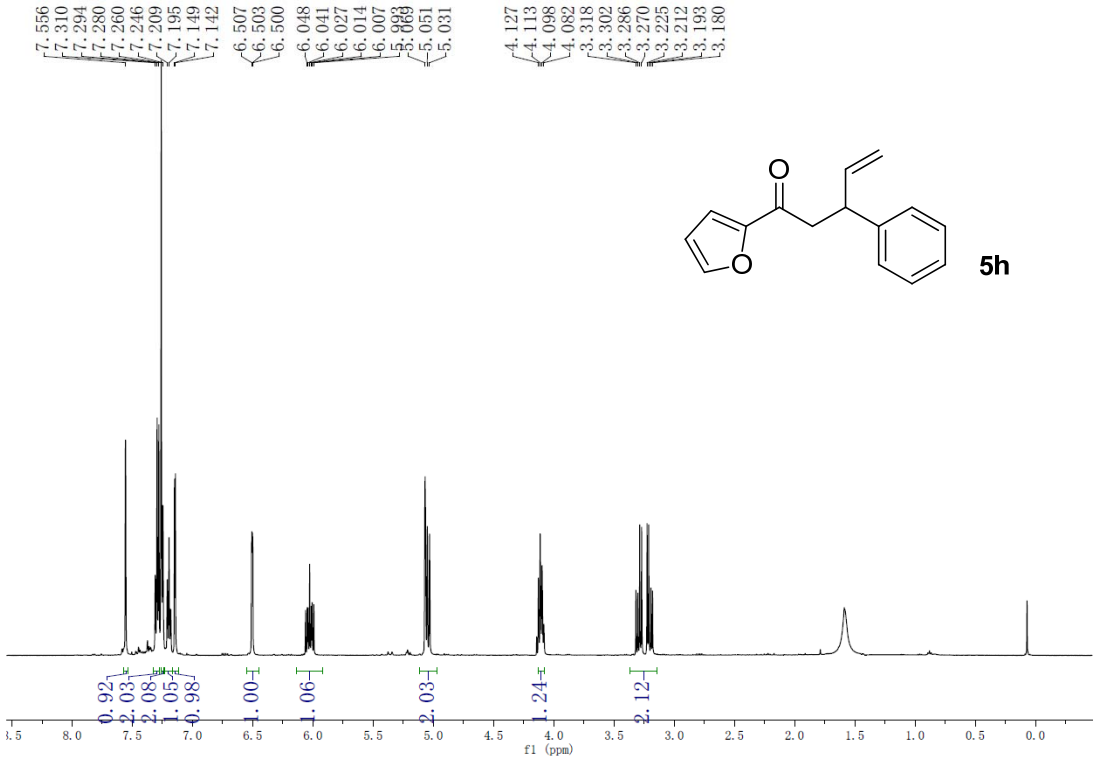
76.924
44.960
O
S
5g
f1 (ppm)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7.142
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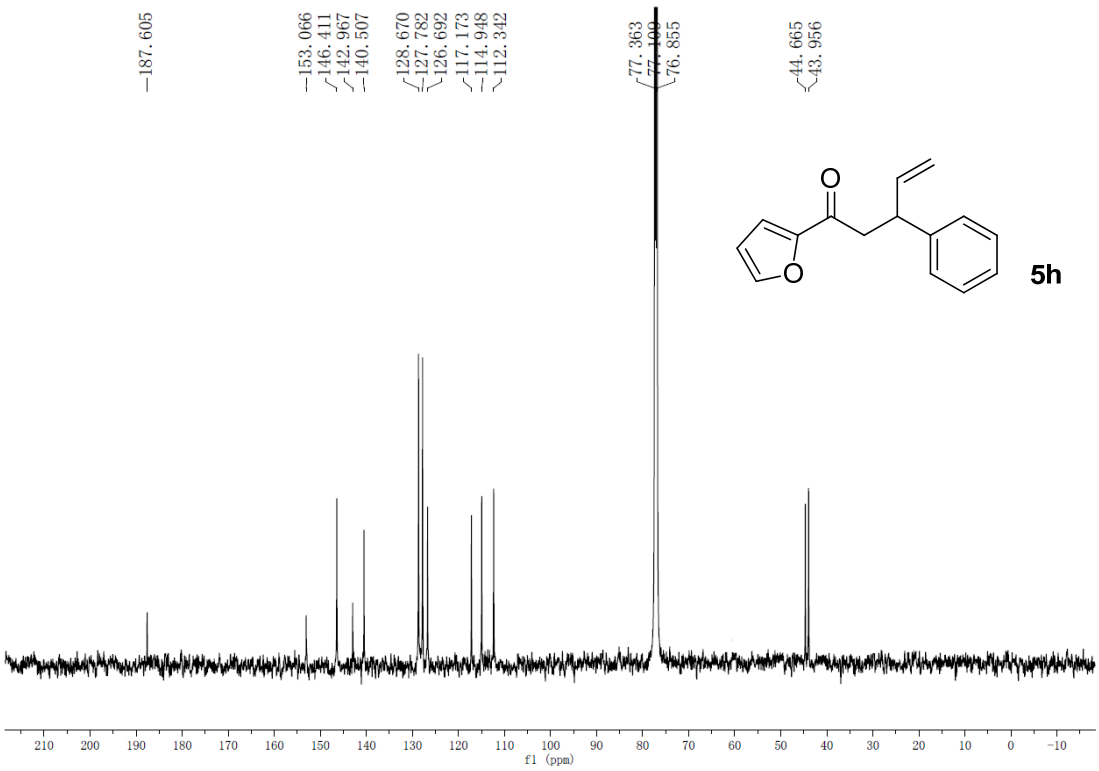
6.503
6.500
6.048
6.041
6.027
6.014
6.007
5.069
5.051
5.031
4.127
4.113
4.098
4.082
3.318
3.302
3.286
3.270
3.225
3.212
3.193
3.180
0.92
2.03
2.08
1.05
0.98
1.00
1.06
2.03
1.24
2.12
f1 (ppm)
5h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128.670
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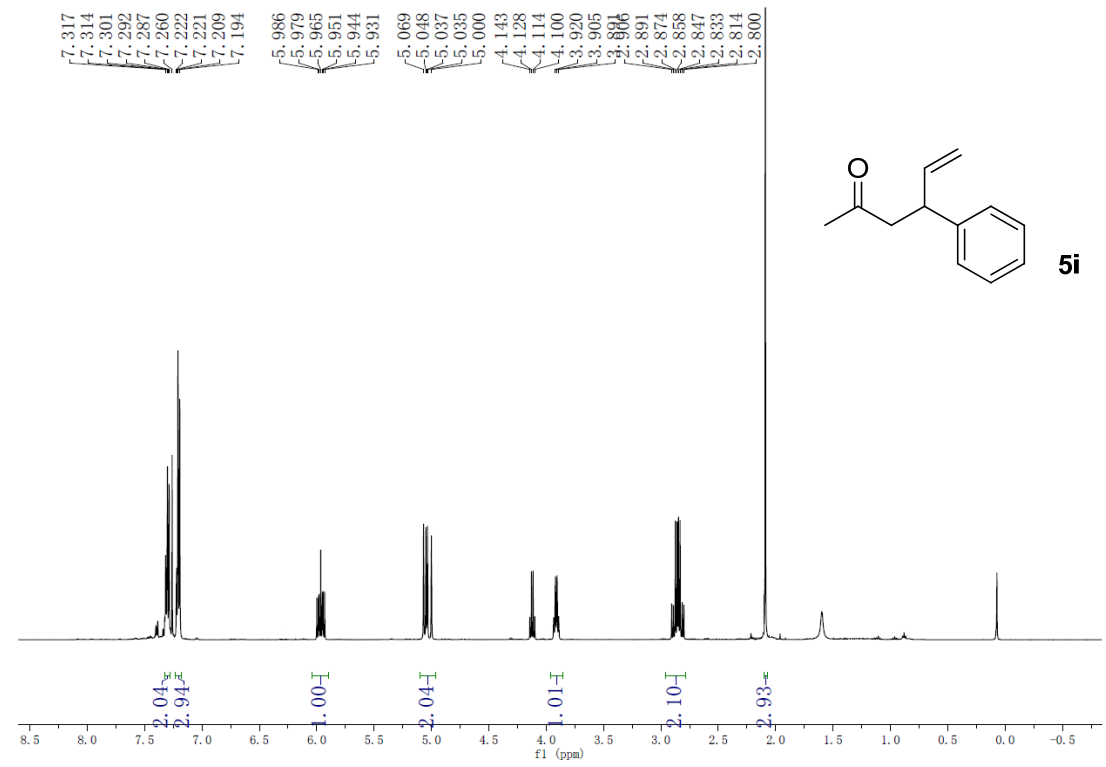
77.109
76.855
44.665
43.956
f1 (ppm)
5h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7.292
7.287
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7.221
7.209
7.194
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5.944
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5.069
5.048
5.037
5.035
5.000
4.143
4.128
4.114
4.100
3.920
3.905
2.891
2.874
2.858
2.847
2.833
2.814
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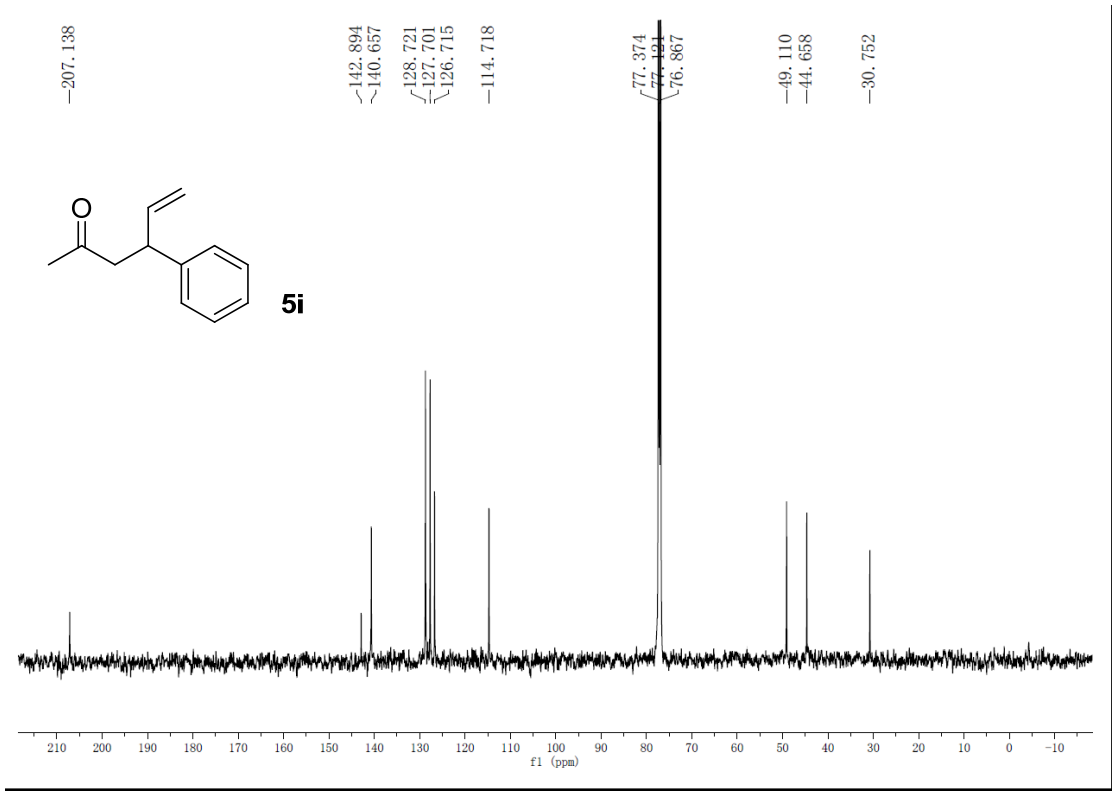
2.04
2.94
1.00
2.04
1.01
2.10
2.93
f1 (ppm)
5i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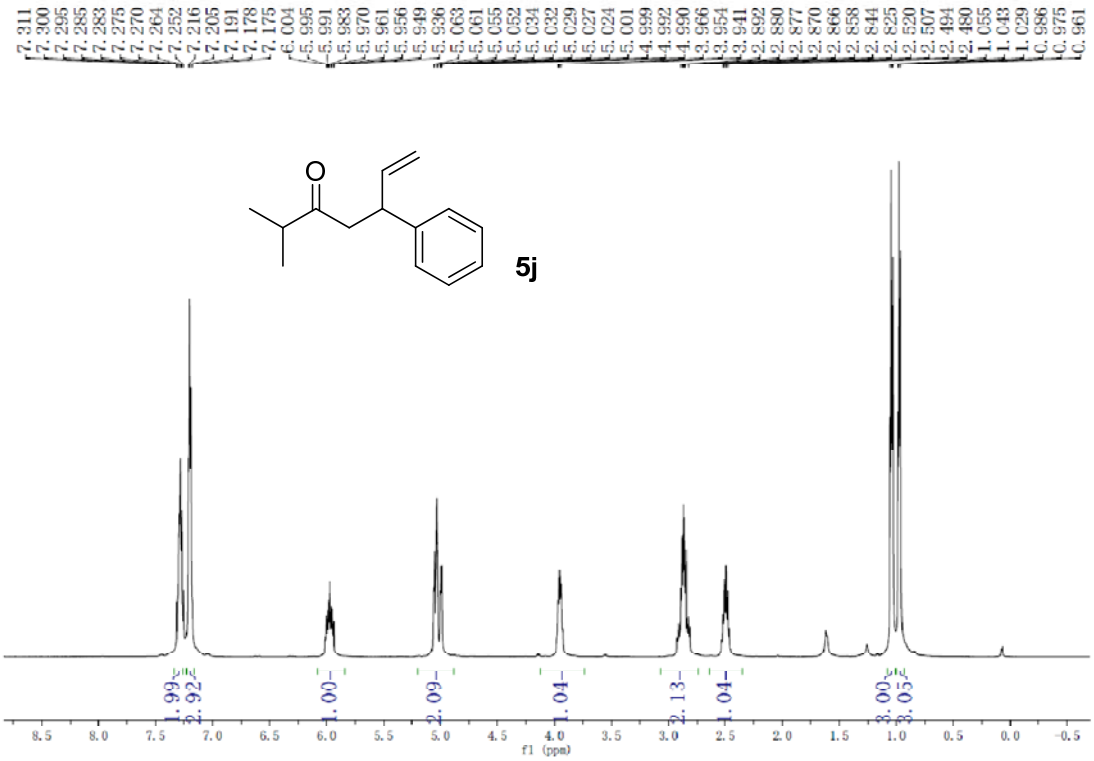
f1 (ppm)
5i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7.178
7.175
6.004
5.995
5.991
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5.032
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5.027
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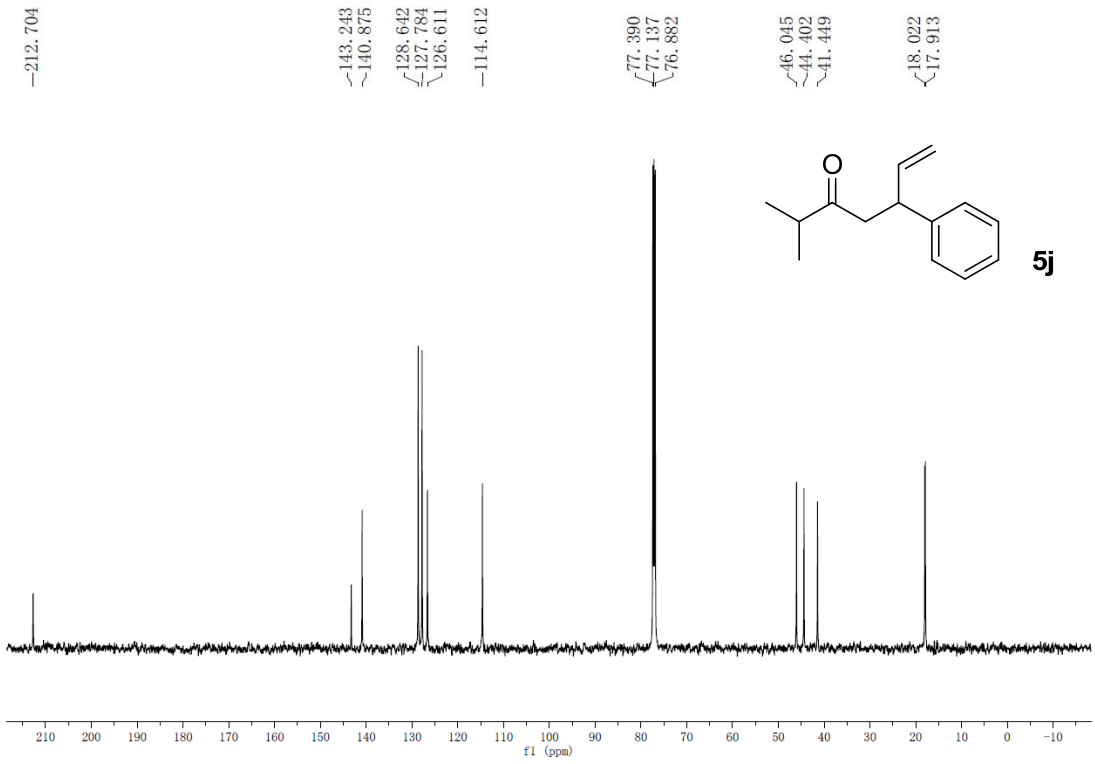
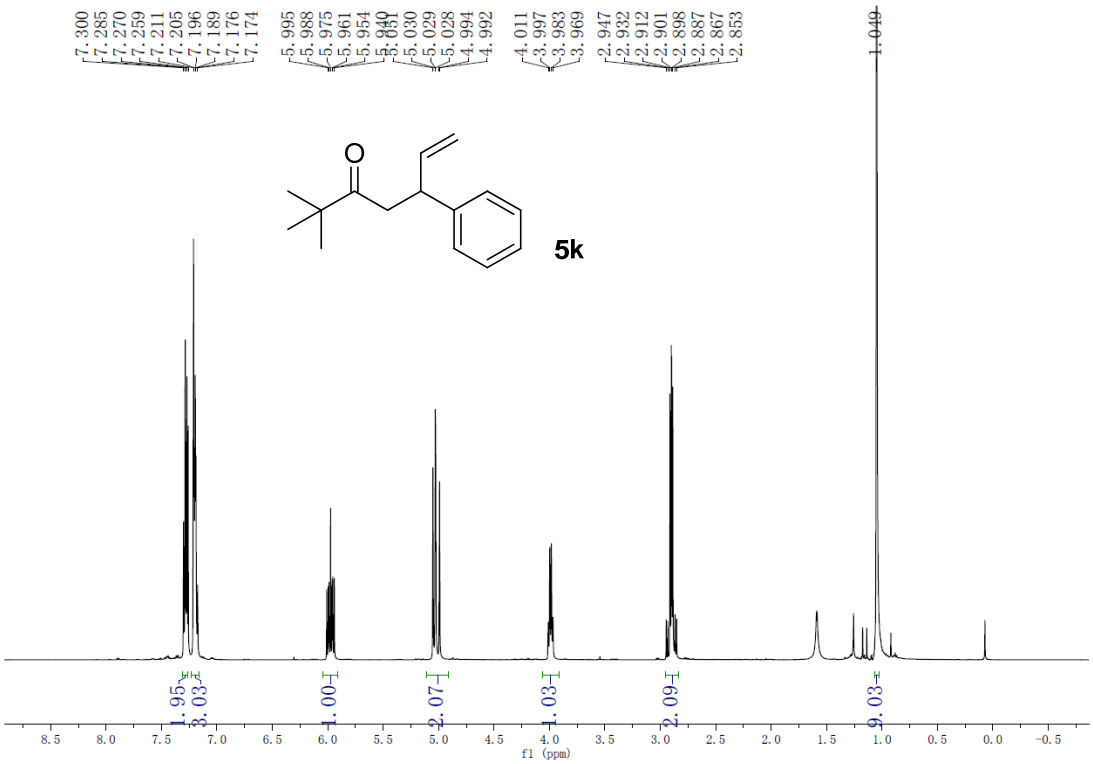
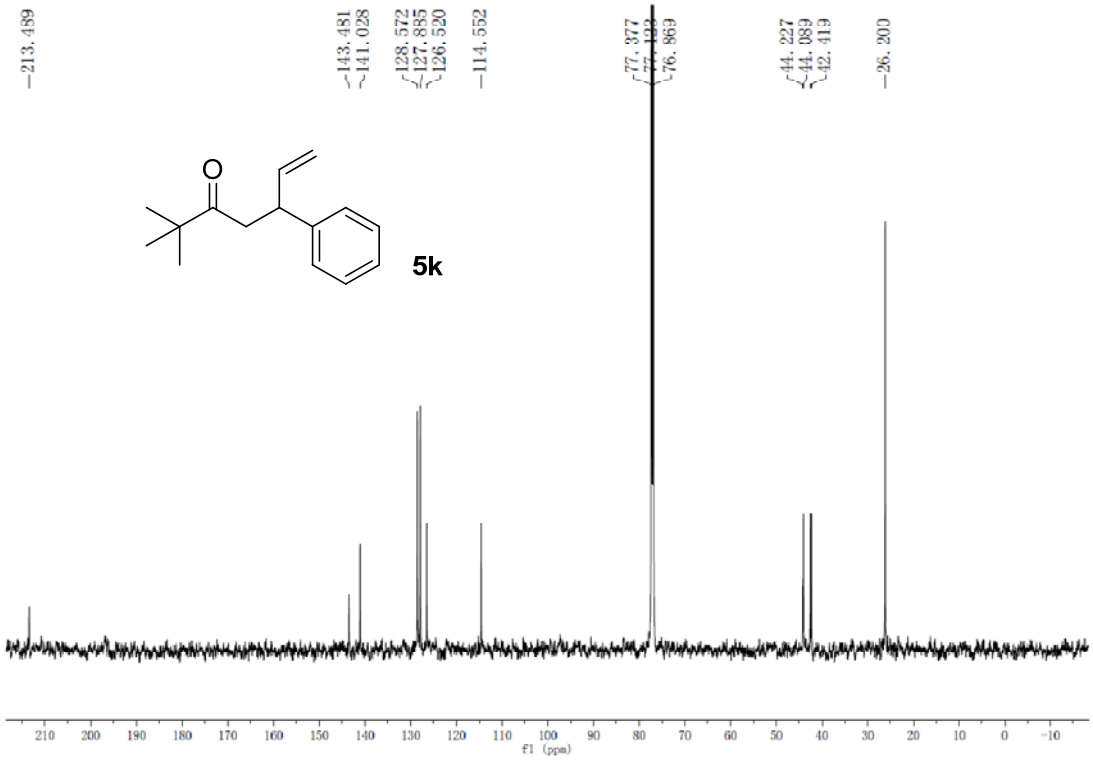
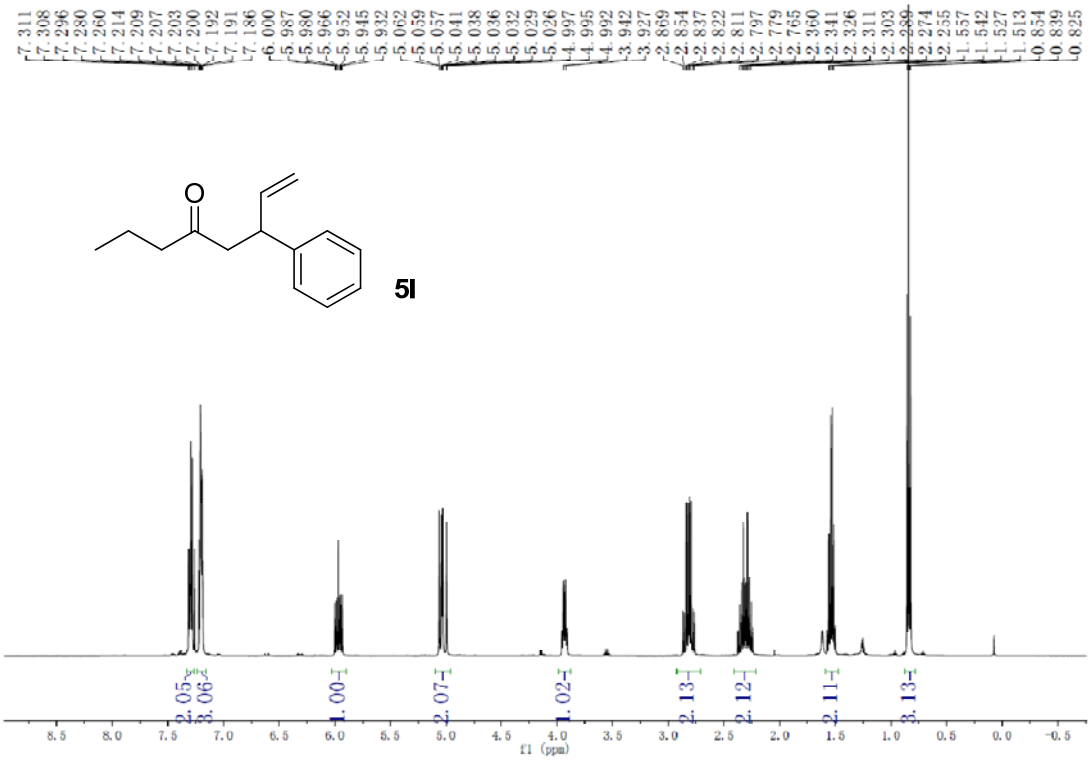
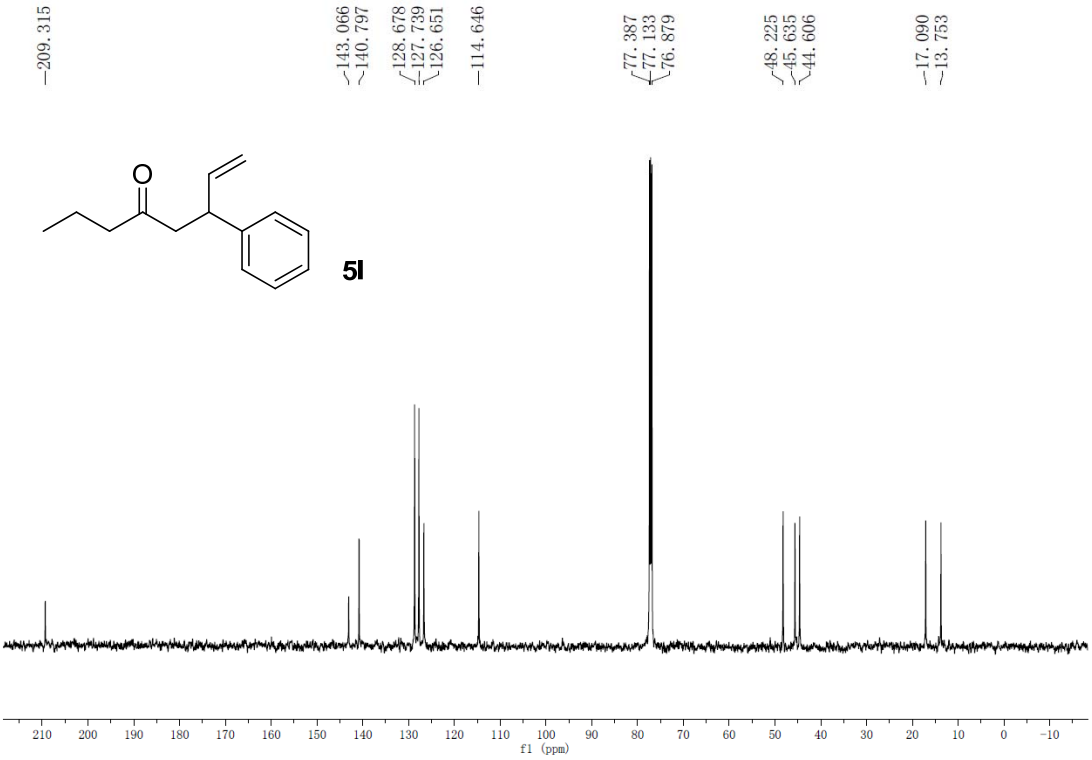
2.880
2.877
2.870
2.866
2.858
2.844
2.825
2.520
2.507
2.494
2.480
1.055
1.043
1.029
0.986
0.975
0.961
O
5j
1.99
2.92
1.00
2.09
1.04
2.13
1.04
3.00
3.05
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


212.704
143.243
140.875
128.642
127.784
126.611
114.612
77.390
77.137
76.882
46.045
44.402
41.449
18.022
17.913
O
5j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.300
7.285
7.270
7.259
7.211
7.205
7.196
7.189
7.176
7.174
5.995
5.988
5.975
5.961
5.954
5.030
5.029
5.028
4.994
4.992
4.011
3.997
3.983
3.969
2.947
2.932
2.912
2.901
2.898
2.887
2.867
2.853
1.049
O
5k
1.95
3.03
1.00
2.07
1.03
2.09
9.03
f1 (ppm)


213.489
143.481
141.028
128.572
127.885
126.520
114.552
77.377
77.193
76.869
44.227
44.089
42.419
26.200
O
5k
f1 (ppm)


7.311
7.308
7.296
7.280
7.260
7.214
7.209
7.207
7.203
7.200
7.192
7.191
7.186
6.000
5.987
5.980
5.966
5.952
5.945
5.932
5.062
5.059
5.057
5.041
5.038
5.036
5.032
5.029
5.026
4.997
4.995
4.992
3.942
3.927
2.869
2.854
2.837
2.822
2.811
2.797
2.779
2.765
2.360
2.341
2.326
2.311
2.303
2.289
2.274
2.255
1.557
1.542
1.527
1.513
0.854
0.839
0.825
O
5l
2.05
3.06
1.00
2.07
1.02
2.13
2.12
2.11
3.13
f1 (ppm)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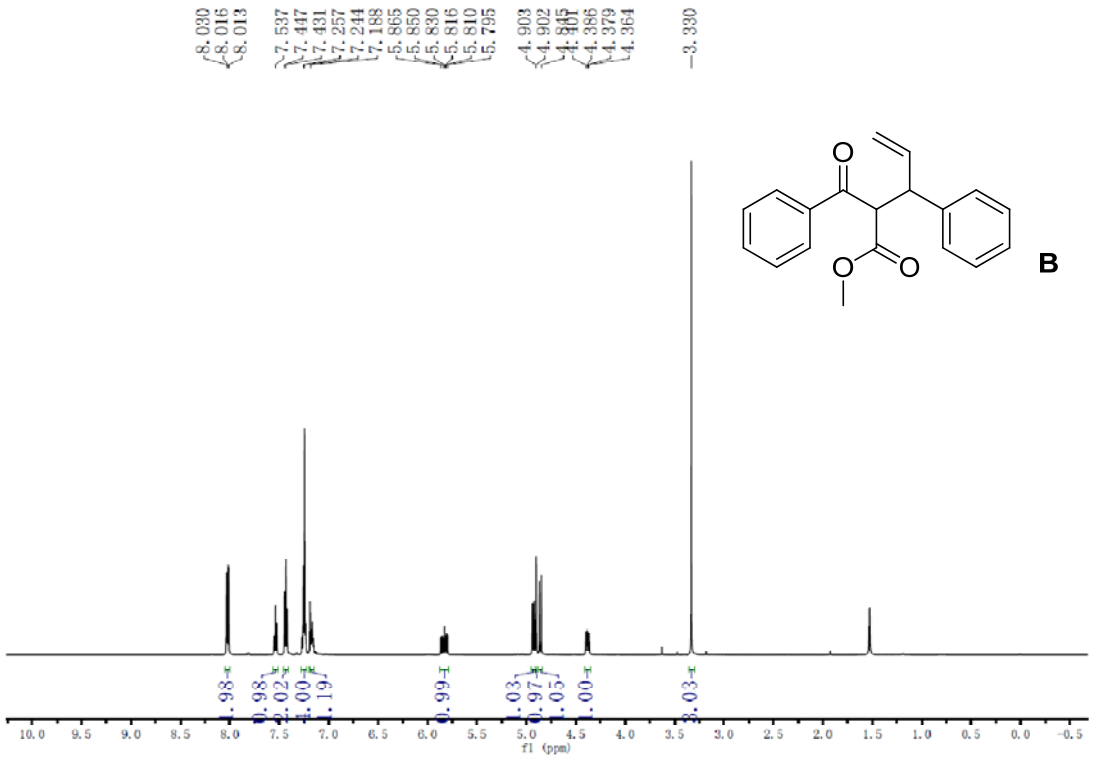
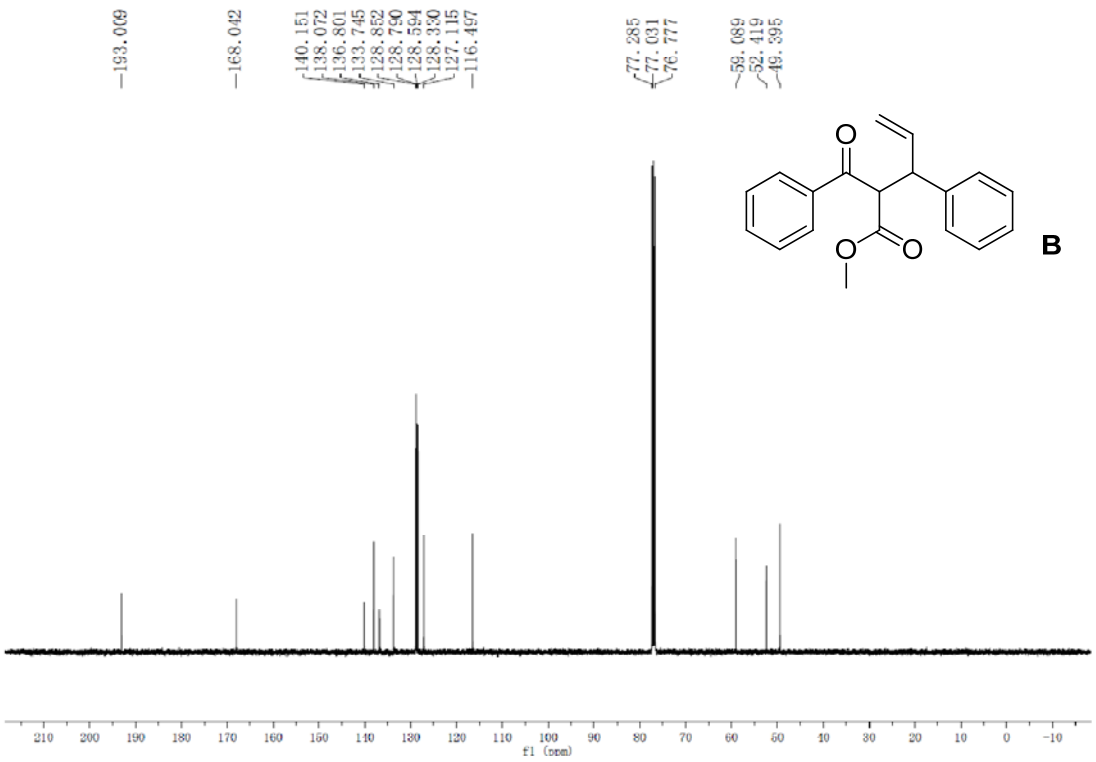
143.066
140.797
128.678
127.739
126.651
114.646
77.387
77.133
76.879
48.225
45.635
44.606
17.090
13.753
O
5l
f1 (ppm)


8.030
8.016
8.013
7.537
7.447
7.431
7.257
7.244
7.188
5.865
5.850
5.830
5.816
5.810
5.795
4.903
4.902
4.401
4.386
4.379
4.364
3.330
O
O
O
B
0.98
0.98
2.02
1.00
1.19
0.99
1.03
0.97
1.05
1.00
3.03
f1 (ppm)


193.009
168.042
140.151
138.072
136.801
133.745
128.852
128.790
128.594
128.330
127.115
116.497
77.285
77.031
76.777
59.089
52.419
49.595
O
O
O
B
f1 (ppm)