Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Rhodium-Catalyzed Chemo- and Regioselective Decarboxylative Addition of β-Ketoacids to Alkynes

Changkun Li, Christian P. Grugel and Bernhard Breit*

Institut für Organische Chemie, Albert-Ludwigs-Universität Freiburg, Albertstraße 21, 79104, Freiburg, Germany

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Table of Contents

General

Column Chromatography was accomplished using MACHEREY-NAGEL silica gel 60® (230-400 mesh). TLC (Thin Layer Chromatography) was performed on aluminum plates pre-coated with silica gel (MERCK 60 F_{254}), which were visualized by UV fluorescence (λ_{max} = 254 nm) and/or by staining with 1% w/v $KMnO_4$ in 0.5 M aqueous K_2CO_3. Melting points were determined on a BÜCHI Dr. Tottoli apparatus and are listed uncorrected. NMR (Nuclear Magnetic Resonance) spectra were acquired on a BRUKER Avance 400 spectrometer (400 MHz and 100.6 MHz for ^1H and ^{13}C respectively). All ^1H NMR spectra are reported in parts per million (ppm) downfield of TMS and were measured relative to the signals at 7.26 ppm ($CHCl_3$). All ^{13}C NMR spectra were reported in ppm relative to residual $CHCl_3$ (77.16 ppm). Data for ^1H NMR are described as following: chemical shift (δ in ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad signal), coupling constant (Hz), integration. Data for ^{13}C NMR spectra are described in terms of chemical shift (δ in ppm). HRMS (High resolution mass spectra) were obtained on a FINNIGAN MAT 8200 instrument (CI/NH_3: 110 eV; EI: 70 eV). MS-CI (Chemical ionization mass spectrometry) was performed on a TSQ 700 or MAT 95XL mass spectrometer from Thermo Fisher Scientific Inc. at an ionization energy of 110 eV and a source temperature of 200 °C. Ammonia or isobutene were used as reactant gases. Chiral HPLC was performed on a MERCK HITACHI HPLC apparatus (pump: L-7100, UV detector: D-7400, oven: L-7360; column: OD-3, DAICEL).

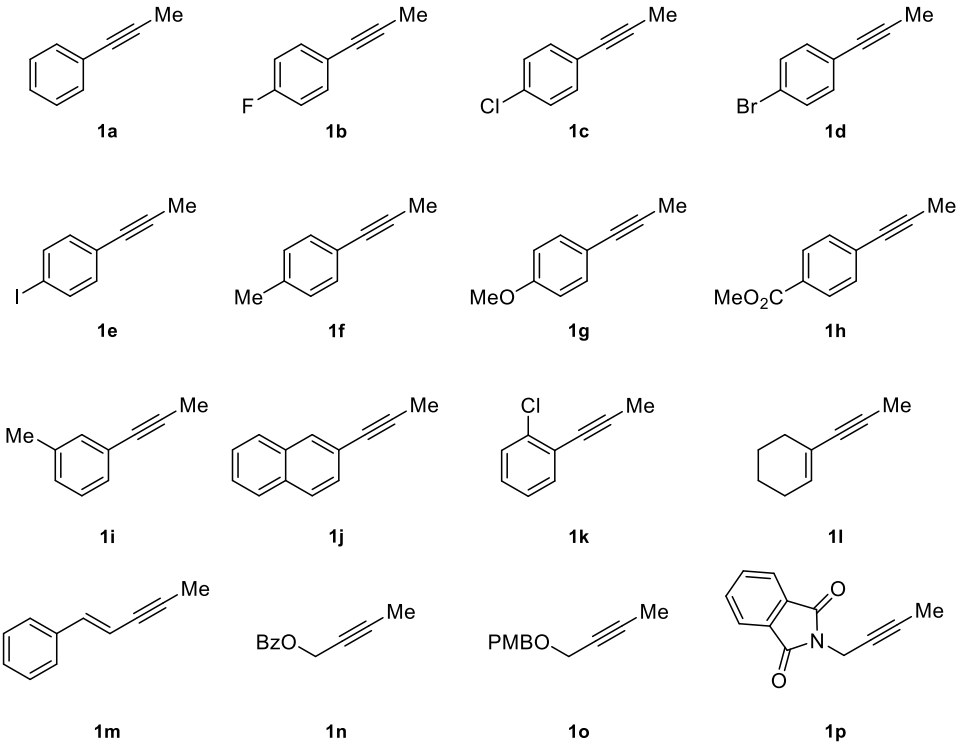
Materials

Solvents: 1,2-Dichloroethane (DCE) and dichloromethane were freshly distilled over CaH_2 and degassed by three Freeze-Pump-Thaw cycles prior to use. Toluene was refluxed over sodium and benzoquinone and distilled prior to use.
Solvents employed for work-up and column chromatography were purchased in technical grade quality and distilled by rotary evaporation before use. The ligands were purchased from Sigma-Aldrich, ABCR, Alfa Aesar and used without further purification. $[Rh(COD)Cl]_2$ was purchased from Sigma-Aldrich.

Preparation of substrates

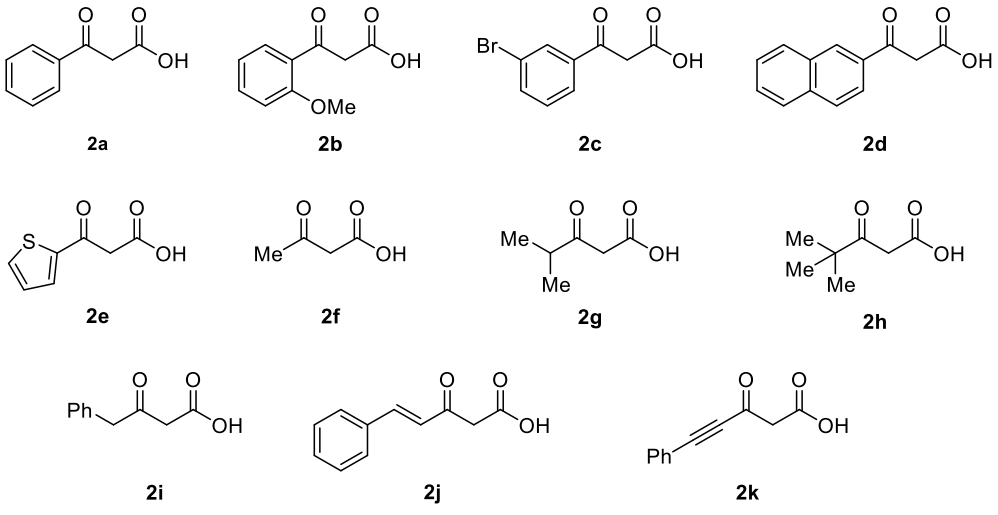
A. Preparation of alkynes

1a was purchased from Alfa-Aesar and used without further purification. **1b**[1], **1c**[1], **1d**[2], **1e**[3], **1h**[4], **1j**[5], **1k**[1] were prepared by Sonogashira coupling with in situ generated 1-propyne following a method developed by Suffert *et al.*.[6] **1f**[1], **1g**[4], **1i**[7], **1l**[8] were prepared by methylation of the corresponding terminal alkynes. **1m**[9] was prepared by a Corey-Fuchs reaction starting from cinnamaldehyde. **1n**[10], **1o**[11] and **1p**[12] were prepared in analogy to literature procedures.

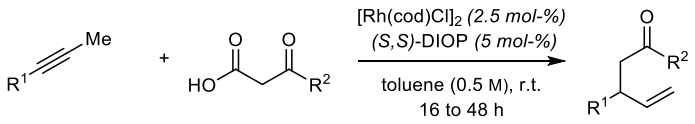


B. Preparation of β-Ketoacids

2a-**2k** were either prepared from the corresponding β-ketoesters[13] or by acylation of TMS-protected malonic acid[14] in analogy to literature procedures.[15]

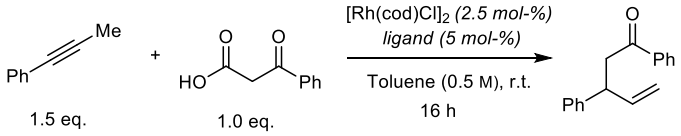


General procedure for rhodium-catalyzed coupling of β-ketoacids and internal alkynes



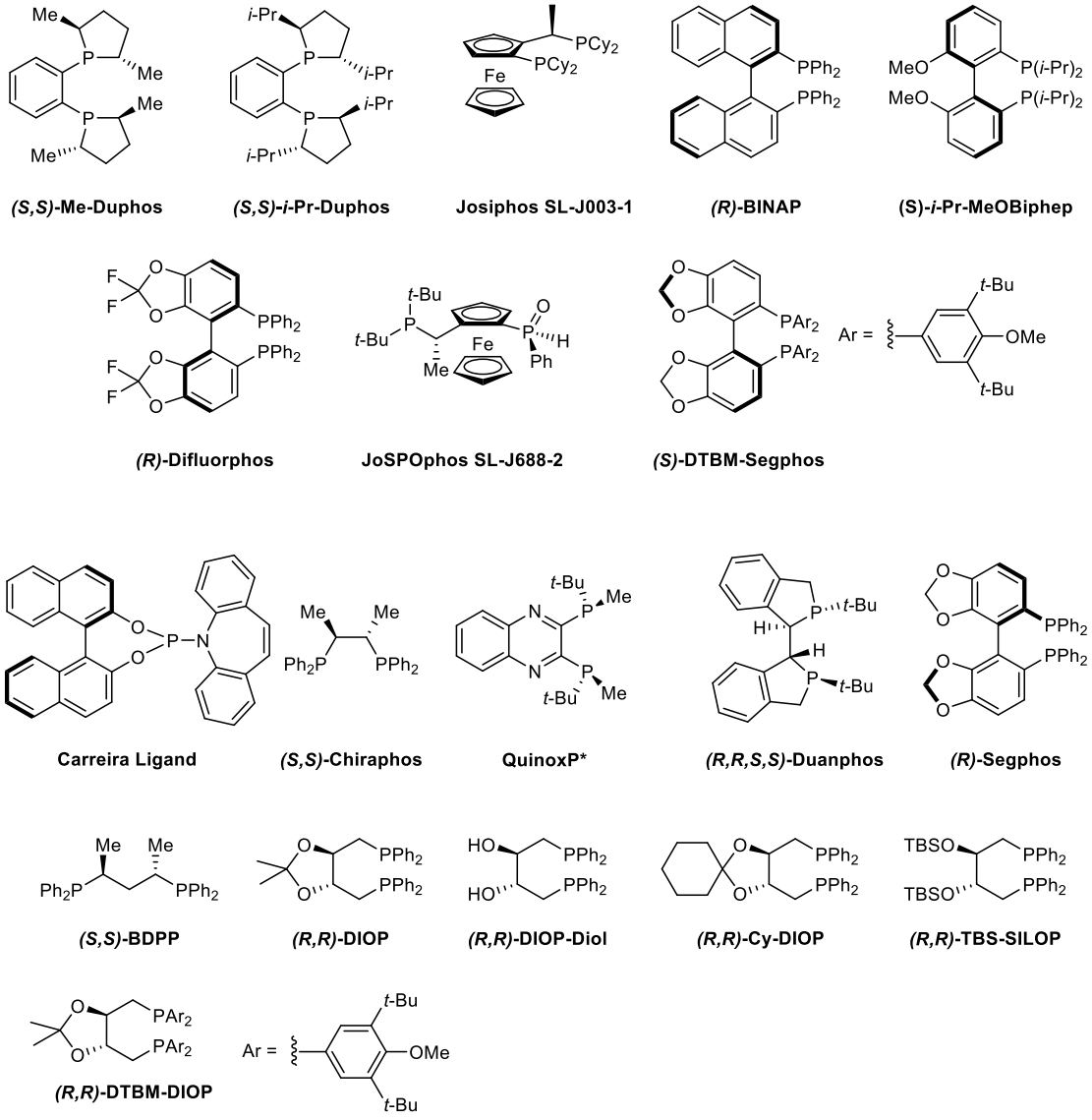
A flame-dried sealed Schlenk tube was charged with $[Rh(cod)Cl]_2$ (6.16 mg, 0.0125 mmol, 2.5 mol-%), *(S,S)*-DIOP (12.46 mg, 0.025 mmol, 5.0 mol-%) and the β -ketoacid (0.75 mmol, 1.5 eq.). The tube was evacuated and backfilled with argon for three times. 1 ml of freshly distilled toluene and the internal alkyne (0.5 mmol, 1.0 eq.) were added by syringe while maintaining an argon counterflow. The tube was sealed with a screw-cap and the resulting suspension was stirred at r.t. until all solid was dissolved (16 to 48 h). The solvent was subsequently removed under reduced pressure and the resulting residue was purified by flash column chromatography on silica gel.

Screening of chiral ligands

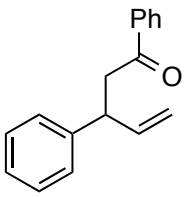


ligand	solvent	isolated yield	*ee*
(S,S)-Me-Duphos	Toluene	no conversion	n.d.
Josiphos SL-003-1	Toluene	no conversion	n.d.
(R)-BINAP	Toluene	< 5 %[a]	n.d.
(S)-DTBM-Segphos	Toluene	< 5 %[a]	n.d.
(S)-*i*-Pr-MeOBiphep	Toluene	< 5 %[a]	n.d.
(R)-Difluorophos	Toluene	< 5 %[a]	n.d.
(S,S)-*i*-Pr-Duphos	Toluene	< 5 %[a]	n.d.
JoSPOphos SL-J688-2	Toluene	< 5 %[a]	n.d.
Carreira Ligand	Toluene	< 5 %[a]	n.d.
(S,S)-Chiraphos	Toluene	< 5 %[a]	n.d.
(R,R)-QuinoxP*	Toluene	< 5 %[a]	n.d.
(R,R,S,S)-Duanphos	Toluene	14 %	54 %
(R)-Segphos	Toluene	12 %	8 %
(S,S)-BDPP	Toluene	36 %	25 %
(R,R)-DIOP	Toluene	79 %	10 %
(R,R)-DIOP-Diol	Toluene	< 5 %[a]	n.d.
(R,R)-Cy-DIOP	Toluene	79 %	11 %
***(R,R)*-DTBM-DIOP**	**Toluene**	**25 %**	**64 %**
(R,R)-TBS-SILOP	Toluene	33 %	7 %

[a]NMR-yield



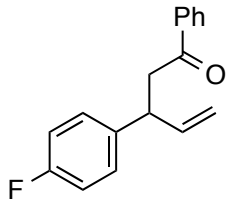
Analytical Data



1-phenyl-3-phenylpent-4-en-1-one (3aa)

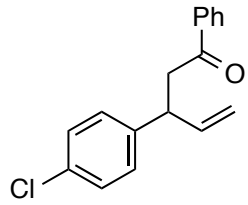
Preparation according to the general procedure. Flash column chromatography (PE : toluene = 1:1) furnished the title compound (98.0 mg, 0.420 mmol, 83 %) as colorless oil. $\mathbf{R_f}$ (PE : EtOAc = 10:1) = 0.53; **^{1}H-NMR (400 MHz, $CDCl_3$)**: 8.01 – 7.85 (m, 2H), 7.59 – 7.51 (m, 1H), 7.49 – 7.41 (m, 2H), 7.35 – 7.16 (m, 5H), 6.06 (ddd, *J* = 16.5, 10.4, 6.8 Hz, 1H), 5.08 (ddd, *J* = 11.4, 1.4 Hz, 1H), 5.04 (ddd, *J* = 17.1, 1.4 Hz, 1.4 Hz, 1H), 4.23 – 4.07 (m, 1H), 3.44 (dd, *J* = 16.6, 7.6 Hz, 1H), 3.37 (dd, *J* = 16.6, 6.6 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: 198.35, 143.30, 140.82, 137.33, 133.12, 128.70, 128.18, 127.84, 126.67, 114.84, 44.70, 44.20 ppm; **HRMS** (pos. APCI): calculated for $C_{17}H_{17}O$ $[M+H]^+$ 237.12739, found 237.12753. HPLC (CHIRALCEL® OD-3, n-heptane/^{i}PrOH = 98 : 2, 0.5 mL/min, 230 nm) t_R = 9.79 min (major), t_R = 10.63 min (minor), 64% ee.

3-(4-fluorophenyl)-1-phenylpent-4-en-1-one (3ba)



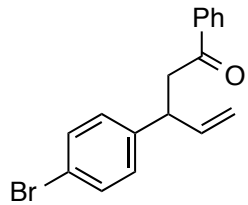
Preparation according to the general procedure. Flash column chromatography (PE : Et_2O = 60:1 → 50:1) furnished the title compound (93.2 mg, 0.37 mmol, 74 %) as colorless oil. $\mathbf{R}_f$ (PE :EtOAc = 10:1) = 0.56; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 7.97 – 7.83 (m, 2H), 7.62 – 7.51 (m, 1H), 7.49 – 7.40 (m, 2H), 7.24 – 7.19 (m, 2H), 7.05 – 6.92 (m, 2H), 6.03 (ddd, *J* = 20.0, 10.3, 6.6 Hz, 1H), 5.08 (ddd, *J* = 10.3, 1.3 Hz, 1H), 5.02 (ddd, *J* = 17.2, 1.4 Hz, 1H), 4.19 – 4.08 (m, 1H), 3.41 (dd, *J* = 16.7, 7.2 Hz, 1H), 3.34 (dd, *J* = 16.6, 7.1 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 198.17 (s), 161.68 (d, *J* = 244.7 Hz), 140.75 (s), 138.89 (d, *J* = 3.2 Hz), 137.24 (s), 133.22 (s), 129.33 (d, *J* = 7.9 Hz), 128.74 (s), 128.16 (s), 115.46 (d, *J* = 21.2 Hz), 114.93 (s), 44.20 (s), 43.88 (s) ppm; **^{19}F-NMR (282 MHz, $CDCl_3$):** = -116.60 (m_c) ppm. The analytical data are in accordance with the literature.[16]

3-(4-chlorophenyl)-1-phenylpent-4-en-1-one (3ca)



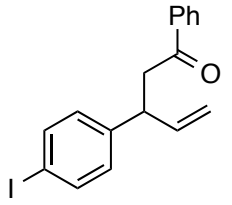
Preparation according to the general procedure. Flash column chromatography (PE:EtOAc = 40:1) furnished the title compound (124.6 mg, 0.46 mmol, 92 %) as colorless oil. $\mathbf{R}_f$ (PE : EtOAc = 10:1) = 0.55; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 7.98 – 7.84 (m, 2H), 7.61 – 7.52 (m, 1H), 7.50 – 7.41 (m, 2H), 7.30 – 7.23 (m, 2H), 7.21 – 7.16 (m, 2H), 6.02 (ddd, *J* = 17.1, 10.3, 6.6 Hz, 1H), 5.09 (ddd, *J* = 10.3, 1.3, 1.3 Hz, 1H), 5.03 (ddd, *J* = 17.1, 1.4, 1.4 Hz, 1H), 4.19 – 4.07 (m, 1H), 3.42 (dd, *J* = 16.8, 7.1 Hz, 1H), 3.34 (dd, *J* = 16.7, 7.1 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 197.97, 141.72, 140.43, 137.17, 133.26, 132.4, 129.27, 128.81, 128.75, 128.15, 115.18, 43.99, 43.96 ppm; **HRMS** (pos. ESI): calculated for $C_{17}H_{16}OCl$ $[M+H]^+$ 271.08842, found 271.08847.

3-(4-bromophenyl)-1-phenylpent-4-en-1-one (3da)



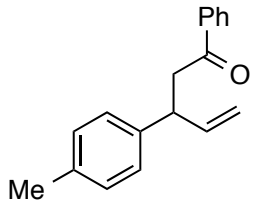
Preparation according to the general procedure. Flash column chromatography (PE:toluene = 1:2) furnished the title compound (114 mg, 0.36 mmol, 73 %) as colorless oil. $\mathbf{R}_f$ (PE : EtOAc = 10:1) = 0.51; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 7.96 – 7.86 (m, 2H), 7.60 – 7.51 (m, 1H), 7.49 – 7.38 (m, 4H), 7.19 – 7.08 (m, 2H), 6.02 (ddd, *J* = 17.1, 10.3, 6.6 Hz, 1H), 5.09 (ddd, *J* = 10.3, 1.3 Hz, 1H), 5.04 (ddd, *J* = 17.2, 1.4 Hz, 1H), 4.16 – 4.06 (m, 1H), 3.42 (dd, *J* = 16.8, 7.1 Hz, 1H), 3.34 (dd, *J* = 16.8, 7.1 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 197.92, 142.25, 140.33, 137.14, 133.25, 131.75, 129.66, 128.74, 128.13, 120.45, 115.22, 44.00, 43.91. **HRMS** (pos. APCI): calculated for $C_{17}H_{19}ONBr$ $[M+NH_4]^+$ 332.06445, found 332.06458.

3-(4-iodophenyl)-1-phenylpent-4-en-1-one (3ea)



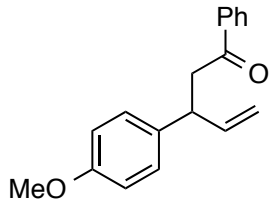
Preparation according to the general procedure. Flash column chromatography (PE : Toluene = 1:1) furnished the title compound (41 mg, 0.11 mmol, 21 %) as colorless oil. $\mathbf{R}_f$ (PE : EtOAc = 10:1) = 0.48; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 7.96 – 7.87 (m, 2H), 7.65 – 7.59 (m, 2H), 7.58 – 7.52 (m, 1H), 7.49 – 7.41 (m, 2H), 7.06 – 6.98 (m, 2H), 6.01 (ddd, *J* = 17.1, 10.3, 6.7 Hz, 1H), 5.09 (ddd, *J* = 10.3, 1.2 Hz, 1H), 5.03 (ddd, *J* = 17.2, 1.2 Hz, 1H), 4.10 (q, *J* = 7.0 Hz, 1H), 3.41 (dd, *J* = 16.8, 7.1 Hz, 1H), 3.34 (dd, *J* = 16.8, 7.1 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 197.91, 142.98, 140.30, 137.74, 137.15, 133.27, 129.99, 128.75, 128.15, 115.26, 91.88, 44.11, 43.88 ppm; **HRMS** (pos. APCI): calculated for $C_{17}H_{19}ONI$ $[M+NH_4]^+$ 380.05058, found 380.05066.

1-phenyl-3-(p-tolyl)pent-4-en-1-one (3fa)



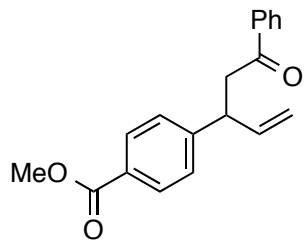
Preparation according to the general procedure. Flash column chromatography (PE : toluene = 1:1) furnished the title compound (99.2 mg, 0.396 mmol, 79 %) as colorless oil. $\mathbf{R_f}$ (PE : EtOAc = 10:1) = 0.55; **^{1}H-NMR (400 MHz, CDCl$_3$)**: δ = 8.12 – 7.79 (m, 2H), 7.63 – 7.51 (m, 1H), 7.50 – 7.40 (m, 2H), 7.20 – 7.08 (m, 4H), 6.04 (ddd, *J* = 17.1, 10.4 Hz, 6.8 Hz, 1H), 5.06 (ddd, *J* = 10.4, 1.4, 1.4 Hz, 1H), 5.03 (ddd, *J* =17.1, 1.4, 1.4 Hz, 1H), 4.17 – 4.01 (m, 1 H, C*H*), 3.43 (dd, *J* = 16.5, 7.7 Hz, 1H), 3.35 (dd, *J* = 16.5, 6.6 Hz, 1H), 2.32 (s, 3H) ppm; **^{13}C-NMR (101 MHz, CDCl$_3$)**: δ = 198.44, 141.02, 140.28, 137.35, 136.18, 133.08, 129.39, 128.68, 128.19, 127.67, 114.61, 44.33, 44.26, 21.11 ppm. **HRMS** (pos. ESI): calculated for $C_{10}H_{11}$ $[M\text{-}C_8H_7O]^+$ 131.08553, found 131.08562.

3-(4-methoxyphenyl)-1-phenylpent-4-en-1-one (3ga)



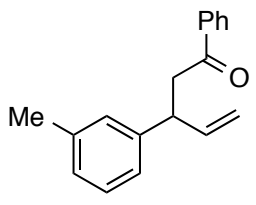
Preparation according to the general procedure. Flash column chromatography (PE:toluene = 1:1 → 1:2) furnished the title compound (98.1 mg, 0.37 mmol, 74 %) as colorless oil. $\mathbf{R_f}$ (PE : EtOAc = 10:1) = 0.45; **^{1}H-NMR (400 MHz, CDCl$_3$)**: δ = 7.98 – 7.86 (m, 2H), 7.58 – 7.50 (m, 1H), 7.47 – 7.40 (m, 2H), 7.21 – 7.12 (m, 2H), 6.87 – 6.75 (m, 2H), 6.04 (ddd, *J* = 17.1, 10.3, 6.7 Hz, 1H), 5.06 (ddd, *J* = 10.5, 1.4, 1.4 Hz, 1H), 5.02 (ddd, *J* = 17.2, 1.4, 1.4 Hz, 1H), 4.17 – 4.03 (m, 1H), 3.78 (s, 3H), 3.41 (dd, *J* = 16.5, 7.5 Hz, 1H), 3.34 (dd, *J* = 16.5, 6.8Hz, 1H) ppm; **^{13}C-NMR (101 MHz, CDCl$_3$)**: δ = 198.50, 158.35, 141.14, 137.35, 135.31, 133.07, 128.77, 128.67, 128.17, 114.46, 114.11, 55.35, 44.32, 43.89 ppm; **HRMS** (pos. ESI): calculated for $C_{18}H_{18}O_2Na$ $[M+Na]^+$ 289.11990, found 289.12009.

methyl 4-(5-oxo-5-phenylpent-1-en-3-yl)benzoate (3ha)



Preparation according to the general procedure. Flash column chromatography (PE:EtOAc = 20:1) furnished the title compound (138 mg, 0.47 mmol, 94 %) as colorless oil. $\mathbf{R_f}$ (PE : EtOAc = 10:1) = 0.26; **^{1}H-NMR (400 MHz, CDCl$_3$)**: δ = 8.00 – 7.95 (m, 2H), 7.95 – 7.89 (m, 2H), 7.59 – 7.51 (m, 1H), 7.48 – 7.41 (m, 2H), 7.37 – 7.31 (m, 2H), 6.04 (ddd, *J* = 17.1, 10.3, 6.7 Hz, 1H), 5.11 (ddd, *J* = 10.3, 1.2 Hz, 1H), 5.08 – 5.02 (m, 1H), 4.21 (q, *J* = 7.0 Hz, 1H), 3.89 (s, 3H), 3.46 (dd, *J* = 16.8, 7.2 Hz, 1H), 3.39 (dd, *J* = 16.8, 7.0 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, CDCl$_3$)**: δ = 197.82, 167.03, 148.60, 140.07, 137.13, 133.28, 130.05, 128.75, 128.67, 128.14, 127.95, 115.52, 52.11, 44.58, 43.83 ppm. **HRMS** (pos. ESI): calculated for $C_{19}H_{18}O_3Na$ $[M+Na]^+$ 317.11482, found 317.11484.

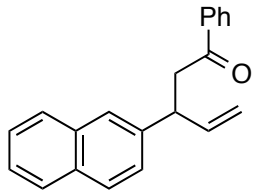
1-phenyl-3-(m-tolyl)pent-4-en-1-one (3ia)



Preparation according to the general procedure. Flash column chromatography (PE : toluene = 1:1) furnished the title compound (110 mg, 0.44 mmol, 89 %) as colorless oil. $\mathbf{R_f}$ (PE : EtOAc = 10:1) = 0.60; **^{1}H-NMR (400 MHz, CDCl$_3$)**: δ = 7.98 – 7.91 (m, 2H), 7.59 – 7.52 (m, 1H), 7.49 – 7.42 (m, 2 H), 7.23 – 7.17 (m, 1 H), 7.12 – 7.00 (m, 3 H), 6.05 (ddd, *J* = 17.2, 10.4, 6.8 Hz, 1H), 5.07 (ddd, *J* = 10.4, 1.3, 1.3 Hz, 1H), 5.04 (ddd, *J* =17.2, 1.4, 1.4, 1H), 4.15 – 4.08 (m, 1H), 3.44 (dd, *J* = 16.6, 7.8 Hz, 1H), 3.36 (dd, *J* = 16.6, 6.4Hz, 1H), 2.34 (s, 3H) ppm; **^{13}C-NMR (101 MHz, CDCl$_3$)**: δ = 198.40 , 143.27, 140.88, 138.26, 137.35, 133.08, 128.67, 128.61, 128.58, 128.18, 127.42, 124.75, 114.70, 44.67, 44.21, 21.56 ppm; **HRMS** (pos. ESI): calculated for $C_{18}H_{18}ONa$ $[M+Na]^+$

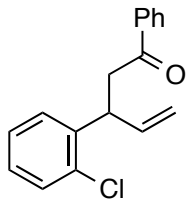
273.12499, found 273.12509.

3-(2-naphthyl)-1-phenylpent-4-en-1-one (3ja)



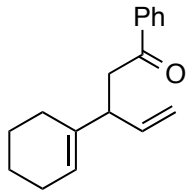
Preparation according to the general procedure. Flash column chromatography (PE : Toluene = 1:1) furnished the title compound (120.8 mg, 0.42 mmol, 85 %) as colorless oil. $\mathbf{R_f}$ (PE : EtOAc = 10:1) = 0.51; **^{1}H-NMR (400 MHz, CDCl$_3$)**: δ = 8.00 – 7.93 (m, 2H), 7.84 – 7.76 (m, 3H), 7.73 – 7.67 (m, 1H), 7.62 – 7.51 (m, 1H), 7.50 – 7.40 (m, 5H), 6.15 (ddd, *J* = 17.1, 10.4, 6.7 Hz, 1H), 5.13 (ddd, *J* = 9.7, 1.3 Hz, 2H), 5.10 (ddd, *J* = 16.5, 1.3 Hz, 1H), 4.34 (q, *J* = 6.8 Hz, 1H), 3.55 (dd, *J* = 16.7, 7.5 Hz, 1H), 3.48 (dd, *J* = 16.7, 6.7 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, CDCl$_3$)**: δ = 198.25, 140.73, 137.30, 133.71, 133.14, 132.49, 128.70, 128.36, 128.19, 127.81, 127.70, 126.44, 126.19, 126.12, 125.63, 115.12, 44.72, 44.11. **HRMS** (pos. ESI): calculated for $C_{21}H_{18}ONa$ $[M+Na]^+$ 309.12499, found 309.12524.

3-(2-chlorophenyl)-1-phenylpent-4-en-1-one (3ka)



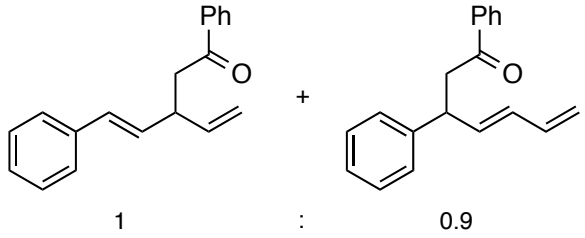
Preparation according to the general procedure together with the addition of $PhCO_2H$ (61.1 mg, 0.5 mmol, 1.0 eq.). Flash column chromatography (PE : Toluene = 1:1 → 1:2) furnished the title compound (90.2 mg, 0.33 mmol, 67 %) as colorless oil. $\mathbf{R_f}$ (PE : EtOAc = 10:1) = 0.48; **^{1}H-NMR (400 MHz, CDCl$_3$)**: δ = 8.00 – 7.90 (m, 2H), 7.59 – 7.52 (m, 1H), 7.49 – 7.43 (m, 2H), 7.40 – 7.36 (m, 1H), 7.29 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.25 – 7.20 (m, 1H), 7.18 – 7.12 (m, 1H), 6.06 (ddd, *J* = 17.1, 10.1, 6.5 Hz, 1H), 5.12 (ddd, *J* = 10.4, 1.3 Hz, 1H), 5.06 – 4.99 (m, 1H), 4.65 (ddddd, *J* = 7.9, 6.3, 6.3, 1.4, 1.4 Hz, 1H), 3.46 (dd, *J* = 16.8, 8.1 Hz, 1H), 3.39 (dd, *J* = 16.8, 6.1 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, CDCl$_3$)**: δ = 197.80, 140.70, 138.99, 137.11, 133.92, 133.19, 130.06, 128.81, 128.72, 128.21, 127.84, 127.09, 115.75, 43.11, 40.94 ppm; **HRMS** (pos. ESI): calculated for $C_{17}H_{15}OClNa$ $[M+Na]^+$ 293.07036, found 293.07047.

3-(cyclohex-1-en-1-yl)-1-phenylpent-4-en-1-one (3la)



Preparation according to the general procedure. Flash column chromatography (PE:toluene = 1:1) furnished the title compound (85.6 mg, 0.35 mmol, 73 %) as colorless oil. $\mathbf{R_f}$ (PE: EtOAc = 10:1) = 0.65; **^{1}H-NMR (400 MHz, CDCl$_3$)**: δ = 8.01 – 7.87 (m, 2H), 7.60 – 7.50 (m, 1H), 7.49 – 7.41 (m, 2H), 5.86 – 5.71 (m, 1H), 5.54 – 5.42 (m, 1H), 5.03 – 4.96 (m, 2H), 3.39 – 3.27 (m, 1H), 3.16 (dd, *J* = 15.7, 6.4 Hz, 1H), 3.09 (dd, *J* = 15.7, 8.0 Hz, 1H), 2.09 – 1.86 (m, 4H), 1.71 – 1.39 (m, 4H) ppm; **^{13}C-NMR (101 MHz, CDCl$_3$)**: δ = 199.37, 140.25, 138.49, 137.61, 132.95, 128.67, 128.22, 122.41, 114.58, 46.83, 41.63, 26.76, 25.41, 23.09, 22.62 ppm; **HRMS** (pos. APCI): calculated for $C_{17}H_{21}O$ $[M+H]^+$ 241.15869, found 241.15880.

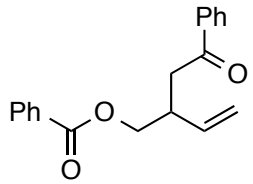
(*E*)-1,5-diphenyl-3-vinylpent-4-en-1-one (3ma)



Preparation according to the general procedure. Flash column chromatography (PE : Toluene = 1:1) furnished an unseparable mixture of the title compound together with its isomer **iso-3ma** (91.3 mg, 0.35 mmol, 71 %) as colorless oil. . $\mathbf{R_f}$ (PE : EtOAc = 10:1) = 0.55; **^{1}H-NMR (3ma, 400 MHz, CDCl$_3$)**: δ = 8.00 – 7.90 (m, 2H), 7.58 (t, *J* = 7.3 Hz, 1H), 7.50 – 7.47 (m, 2H), 7.36 – 7.26 (m, 4H), 7.24 – 7.16 (m, 1H), 6.45 (d, *J* = 16.0 Hz, 1H), 6.22 (dd, *J* = 15.9, 7.3 Hz, 1H),

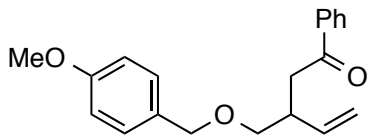
5.94 (ddd, *J* = 17.2, 10.3, 6.9 Hz, 1H), 5.18 – 5.06 (m, 2H), 3.70 (pent, *J* = 7.0 Hz, 1H), 3.21 (d, *J* = 7.1 Hz, 2H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 198.46, 139.81, 137.40, 133.13, 131.45, 130.57, 128.74, 128.59, 128.23, 127.35, 126.32, 115.30, 43.42, 42.28 ppm. The analytical data are in accordance with the literature.[17]

2-(2-oxo-2-phenylethyl)but-3-en-1-yl benzoate (3na)



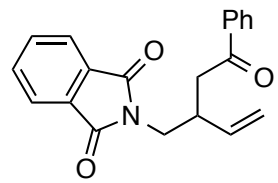
Preparation according to the general procedure. Flash column chromatography (*n*-pentane : Et_2O = 50:1) furnished the title compound (86.4 mg, 0.29 mmol, 58 %) as colorless oil. **R_f** (PE: EtOAc = 10:1) = 0.30; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 8.05 – 7.90 (m, 4H), 7.60 – 7.51 (m, 2H), 7.47 – 7.36 (m, 4H), 5.89 (ddd, *J* = 17.3, 10.4, 7.5 Hz, 1H), 5.21 (ddd, *J* = 17.3, 1.3 Hz, 1H), 5.14 (ddd, *J* = 10.5, 1.2 Hz, 1H), 4.43 (dd, *J* = 10.9, 6.0 Hz, 1H), 4.36 (dd, *J* = 10.9, 6.4 Hz, 1H), 3.42 – 3.31 (m, 1H), 3.24 (dd, *J* = 16.7, 6.1 Hz, 1H), 3.14 (dd, *J* = 16.7, 7.3 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 198.20, 166.50, 137.59, 137.17, 133.27, 133.08, 130.27, 129.70, 128.75, 128.48, 128.22, 116.95, 67.10, 40.21, 38.65 ppm; **HRMS** (pos. APCI): calculated for $C_{19}H_{22}O_3N$ $[M+NH_4]^+$ 312.15942, found 312.15952.

3-(((4-methoxybenzyl)oxy)methyl)-1-phenylpent-4-en-1-one (3oa)



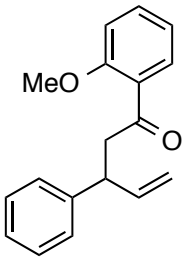
Preparation according to the general procedure. Flash column chromatography (PE:EtOAc = 50:1 → 30:1) furnished the title compound (119.8 mg, 0.39 mmol, 77 %) as colorless oil. **R_f** (PE: EtOAc = 10:1) = 0.34; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 7.99 – 7.90 (m, 2H), 7.59 – 7.50 (m, 1H), 7.49 – 7.40 (m, 2H), 7.23 – 7.17 (m, 2H), 6.89 – 6.81 (m, 2H), 5.84 (ddd, *J* = 17.4, 10.4, 7.4 Hz, 1H), 5.13 – 5.04 (m, 2H), 4.43 (s, 2H), 3.80 (s, 3H), 3.53 (dd, *J* = 9.3, 5.4 Hz, 1H), 3.45 (dd, *J* = 9.3, 6.7 Hz, 1H), 3.25 (dd, *J* = 16.1, 5.8 Hz, 1H), 3.18 – 3.09 (m, 1H), 3.02 – 2.92 (m, 1H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 199.25, 159.28, 138.78, 137.48, 132.99, 130.58, 129.30, 128.63, 128.25, 115.88, 113.87, 72.80, 72.65, 55.38, 40.38, 39.64 ppm; **HRMS** (pos. APCI): calculated for $C_{20}H_{26}O_3N$ $[M+NH_4]^+$ 328.19072, found 328.19089.

2-(2-(2-oxo-2-phenylethyl)but-3-en-1-yl)isoindoline-1,3-dione (3pa)



Preparation according to the general procedure. Flash column chromatography (PE:EtOAc = 10:1 → 5:1) furnished the title compound (87.7 mg, 0.28 mmol, 55%) as colorless oil. **R_f** (PE: EtOAc = 5:1) = 0.24; **m.p.** 68–72 °C; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 7.93 – 7.86 (m, 2H), 7.85 – 7.78 (m, 2H), 7.73 – 7.65 (m, 2H), 7.58 – 7.50 (m, 1H), 7.47 – 7.39 (m, 2H), 5.77 (ddd, *J* = 17.2, 10.3, 8.3 Hz, 1H), 5.06 (ddd, *J* = 17.2, 1.2 Hz, 1H), 5.01 (ddd, *J* = 10.3, 1.4, 0.7 Hz, 1H), 3.82 (dd, *J* = 15.4, 7.3 Hz, 1H), 3.79 (dd, *J* = 15.4, 7.3 Hz, 1H), 3.43 – 3.30 (m, 1H), 3.11 (d, *J* = 6.7 Hz, 2H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 198.00, 168.48, 138.12, 137.13, 134.01, 133.15, 132.11, 128.68, 128.17, 123.37, 117.30, 41.83, 41.31, 39.03 ppm; **HRMS** (pos. APCI): calculated for $C_{20}H_{18}O_3N$ $[M+H]^+$ 320.12812, found 320.12823.

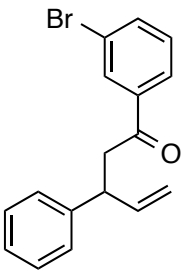
1-(2-methoxyphenyl)-3-phenylpent-4-en-1-one (3ab)



Preparation according to the general procedure. Flash column chromatography (PE : EtOAc = 40:1) furnished the title compound (103.6 mg, 0.39 mmol, 77 %) as colorless oil. **R_f** (PE : EtOAc = 20:1) = 0.31; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 7.58 – 7.52 (m, 1H), 7.47 – 7.39 (m, 1H), 7.34 – 7.16 (m, 5H), 7.01 – 6.93 (m, 2H), 6.03 (dddd, *J* = 17.1, 10.3,

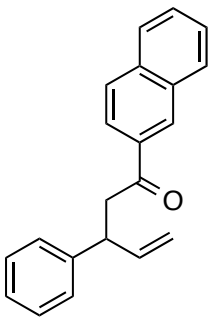
6.8, 0.8 Hz, 1H), 5.08 – 4.98 (m, 2H), 4.08 (q, *J* = 7.1 Hz, 1H), 3.89 (s, 3H), 3.50 – 3.44 (m, 1H), 3.41 (ddd, *J* = 7.9, 4.3, 0.9 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 200.95, 158.31, 143.55, 141.16, 133.31, 130.40, 128.86, 128.51, 127.85, 126.40, 120.80, 114.46, 111.51, 55.58, 49.29, 44.99 ppm. The analytical data are in accordance with the literature.[18]

1-(3-bromophenyl)-3-phenylpent-4-en-1-one (3ac)



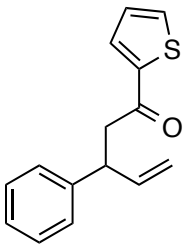
Preparation according to the general procedure. Flash column chromatography (PE : Toluene = 1:1) furnished the title compound (105.4 mg, 0.34 mmol, 67 %) as colorless oil. **R_f** (PE : EtOAc = 10:1) = 0.61; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 8.11 – 7.98 (m, 1H), 7.83 (ddd, *J* = 7.8, 1.7, 1.1 Hz, 1H), 7.66 (ddd, *J* = 8.0, 2.0, 1.0 Hz, 1H), 7.41 – 7.12 (m, 6H), 6.04 (ddd, *J* = 17.1, 10.3, 6.8 Hz, 1H), 5.08 (ddd, *J* = 10.3, 1.3 Hz, 1H), 5.04 (ddd, *J* = 17.1, 1.4 Hz, 1H), 4.17 – 4.06 (m, 1H), 3.40 (dd, *J* = 16.7, 7.5 Hz, 1H), 3.33 (dd, *J* = 16.7, 6.7 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 196.94, 142.98, 140.54, 139.00, 135.97, 131.26, 130.27, 128.74, 127.79, 126.77, 126.65, 123.08, 114.99, 44.60, 44.24 ppm; **HRMS** (pos. ESI): calculated for $C_{17}H_{15}OBrNa$ $[M+Na]^+$ 337.01985, found 337.01996.

1-(naphthalen-2-yl)-3-phenylpent-4-en-1-one (3ad)



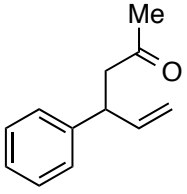
Preparation according to the general procedure. Flash column chromatography (PE : Et_2O = 40:1) furnished the title compound (107.2 mg, 0.37 mmol, 75 %) as colorless oil. **R_f** (PE : EtOAc = 10:1) = 0.54; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 8.45 – 8.41 (m, 1H), 8.00 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.97 – 7.93 (m, 1H), 7.90 – 7.84 (m, 2H), 7.63 – 7.52 (m, 2H), 7.37 – 7.28 (m, 4H), 7.24 – 7.17 (m, 1H), 6.10 (ddd, *J* = 17.1, 10.4, 6.8 Hz, 1H), 5.10 (ddd, *J* = 10.4, 1.3 Hz, 1H), 5.07 (ddd, *J* = 17.1, 1.4 Hz, 1H), 4.26 – 4.14 (m, 1H), 3.58 (dd, *J* = 16.4, 7.7 Hz, 1H), 3.50 (dd, *J* = 16.4, 6.6 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: 198.17, 162.89, 160.46, 140.75, 138.90, 138.87, 137.24, 133.22, 129.37, 129.29, 128.74, 128.16, 115.56, 115.35, 114.93, 44.20, 43.88 ppm. The analytical data are in accordance with the literature.[16]

3-phenyl-1-(thiophen-2-yl)pent-4-en-1-one (3ae)



Preparation according to the general procedure. Flash column chromatography (PE : Et_2O = 80:1 → 65:1) furnished the title compound (105.3 mg, 0.44 mmol, 87 %) as colorless oil. **R_f** (PE : EtOAc = 10:1) = 0.43; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 7.69 (dd, *J* = 3.8, 1.1 Hz, 1H), 7.61 (dd, *J* = 5.0, 1.1 Hz, 1H), 7.33 – 7.26 (m, 3H), 7.26 – 7.24 (m, 1H), 7.23 – 7.18 (m, 1H), 7.10 (dd, *J* = 5.0, 3.8 Hz, 1H), 6.05 (ddd, *J* = 17.1, 10.4, 6.8 Hz, 1H), 5.08 (ddd, *J* = 10.4, 1.3 Hz, 1H), 5.06 (ddd, *J* = 17.1, 1.4 Hz, 1H), 4.19 – 4.08 (m, 1H), 3.36 (dd, *J* = 15.9, 7.8 Hz, 1H), 3.28 (dd, *J* = 15.9, 6.7 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 191.17, 144.69, 143.03, 140.53, 133.74, 131.89, 128.72, 128.15, 127.81, 126.74, 115.05, 45.06, 45.01 ppm. The analytical data are in accordance with the literature.[17]

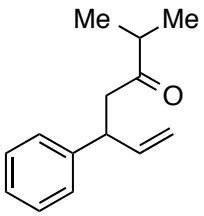
4-phenylhex-5-en-2-one (3af)



Preparation according to the general procedure. Flash column chromatography (PE : Et_2O = 80:1 → 65:1) furnished the title compound (66.3 mg, 0.38 mmol, 76 %) as colorless oil. **R_f** (PE : EtOAc = 10:1) = 0.48; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 7.47 – 7.04 (m, 5H), 5.97 (ddd, *J* = 17.1, 10.3, 6.8 Hz, 1H), 5.06 (ddd, *J* = 10.3, 1.3 Hz, 1H), 5.02 (ddd, *J* = 17.1, 1.3 Hz, 1H), 3.96 – 3.86 (m, 1H), 2.92 – 2.78 (m, 2H), 2.09 (s, 3H) ppm; **^{13}C-**

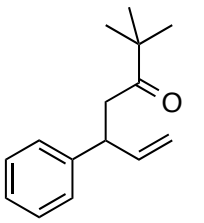
NMR (101 MHz, $CDCl_3$): δ = 207.01, 142.93, 140.69, 128.74, 128.72, 127.71, 126.72, 114.72, 49.16, 44.70, 30.73 ppm; **HRMS** (pos. ESI): calculated for $C_{18}H_{12}ON$ $[M+NH_4]^+$ 192.13829, found 192.13826.

2-methyl-5-phenylhept-6-en-3-one (3ag)



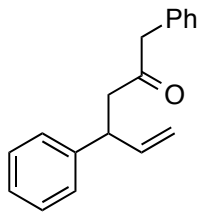
Preparation according to the general procedure. Flash column chromatography (PE : Toluene = 1:1) furnished the title compound (67.3 mg, 0.33 mmol, 69 %) as colorless oil. $\mathbf{R_f}$ (PE : EtOAc = 10:1) = 0.65; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 7.32 – 7.26 (m, 2H), 7.23 – 7.16 (m, 3H), 5.98 (ddd, *J* = 17.1, 10.4, 6.8 Hz, 1H), 5.05 (ddd, *J* = 10.4, 1.3 Hz, 1H), 5.01 (ddd, *J* = 17.1, 1.4 Hz, 1H), 4.01 – 3.91 (m, 1H), 2.90 (dd, *J* = 16.3, 7.2 Hz, 1H), 2.84 (dd, *J* = 16.4, 7.0 Hz, 1H), 2.50 (hept, *J* = 6.9 Hz, 1H), 1.04 (d, *J* = 6.9 Hz, 3H), 0.98 (d, *J* = 6.9 Hz, 3H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 212.60, 143.29, 140.92, 128.65, 127.80, 126.62, 114.62, 46.10, 44.45, 41.47, 18.05, 17.94 ppm. The analytical data are in accordance with the literature.[17]

2,2-dimethyl-5-phenylhept-6-en-3-one (3ah)



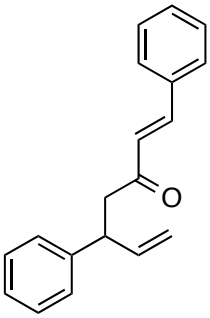
Preparation according to the general procedure. Flash column chromatography (PE : Toluene = 1:1) furnished the title compound (83.1 mg, 0.38 mmol, 77 %) as colorless oil. $\mathbf{R_f}$ (PE : EtOAc = 20:1) = 0.33; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 7.33 – 7.24 (m, 2H), 7.24 – 7.16 (m, 3H), 5.99 (ddd, *J* = 17.1, 10.4, 6.8 Hz, 1H), 5.05 (ddd, *J* = 10.5, 1.4 Hz, 1H), 5.02 (ddd, *J* = 17.2, 1.4 Hz, 1H), 4.00 (q, *J* = 7.0 Hz, 1H), 2.93 (dd, *J* = 17.1, 7.2 Hz, 1H), 2.89 (dd, *J* = 17.1, 7.0 Hz, 1H), 1.06 (d, *J* = 2.1 Hz, 9H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 213.34, 143.50, 141.06, 128.56, 127.89, 126.51, 114.53, 44.12, 42.45, 26.22 ppm; **HRMS:** calculated for $C_{15}H_{24}ON$ $[M+NH_4]^+$ 234.18524, found 234.18527.

1,4-diphenylhex-5-en-2-one (3ai)



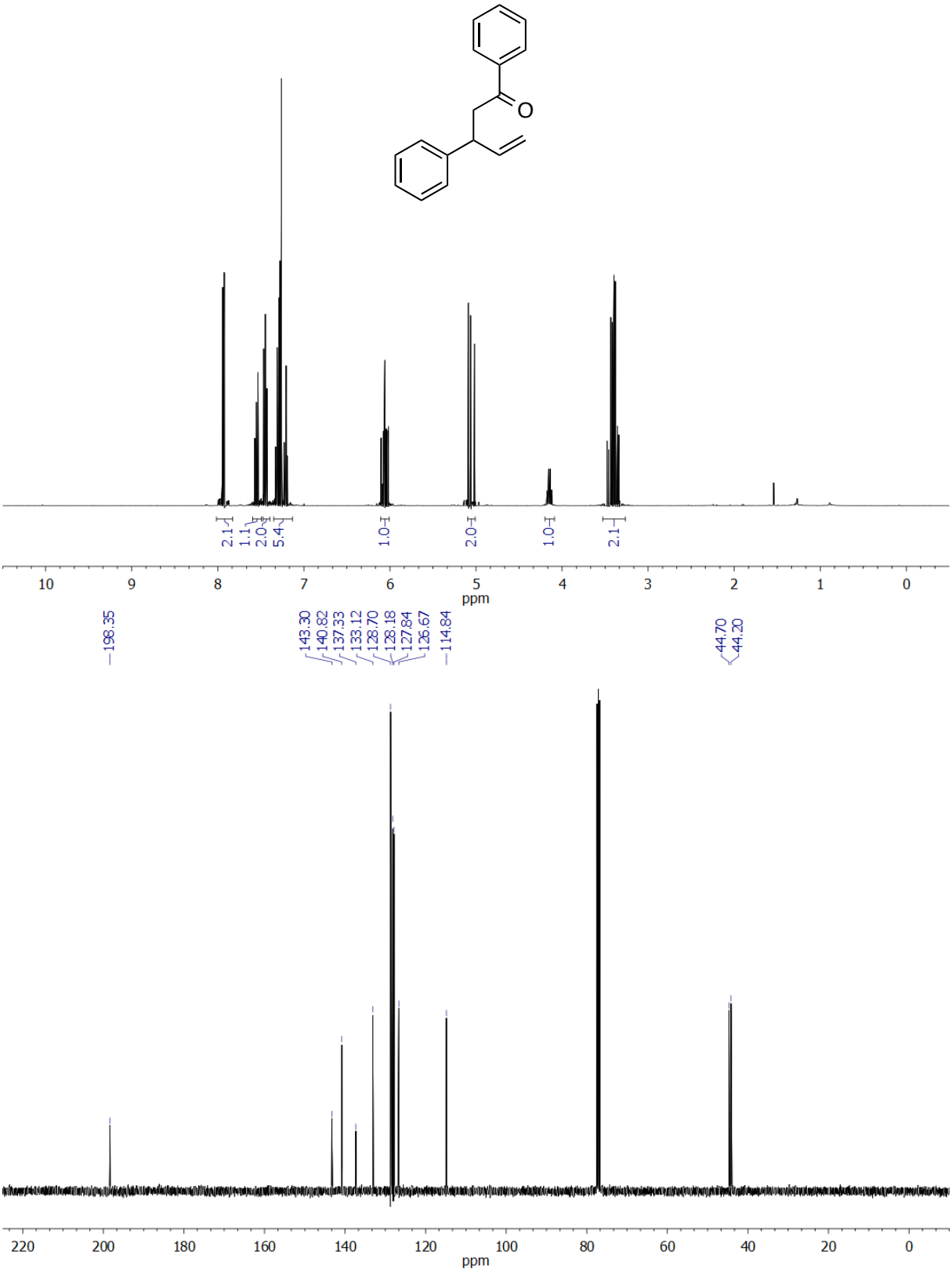
Preparation according to the general procedure. Flash column chromatography (PE : Et_2O = 40:1) furnished the title compound (85.5 mg, 0.36 mmol, 72 %) as colorless oil. $\mathbf{R_f}$ (PE : EtOAc = 15:1) = 0.28; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 7.33 – 7.17 (m, 6H), 7.17 – 7.12 (m, 2H), 7.12 – 7.07 (m, 2H), 5.93 (ddd, *J* = 17.8, 10.3, 6.8 Hz, 1H), 5.03 (ddd, *J* = 10.3, 1.3 Hz, 1H), 4.97 (ddd, *J* = 17.2, 1.4 Hz, 1H), 3.96 – 3.83 (m, 1H), 3.60 (s, 2H), 2.89 (dd, *J* = 16.3, 7.4 Hz, 1H), 2.84 (dd, *J* = 16.3, 7.3 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 206.35, 142.85, 140.60, 133.96, 129.56, 128.80, 128.69, 127.75, 127.12, 126.68, 114.73, 50.98, 47.34, 44.62 ppm. **HRMS** (pos. APCI): calculated for $C_{18}H_{22}ON$ $[M+NH_4]^+$ 268.16959, found 268.16959.

(*E*)-1,5-diphenylhepta-1,6-dien-3-one (3aj)

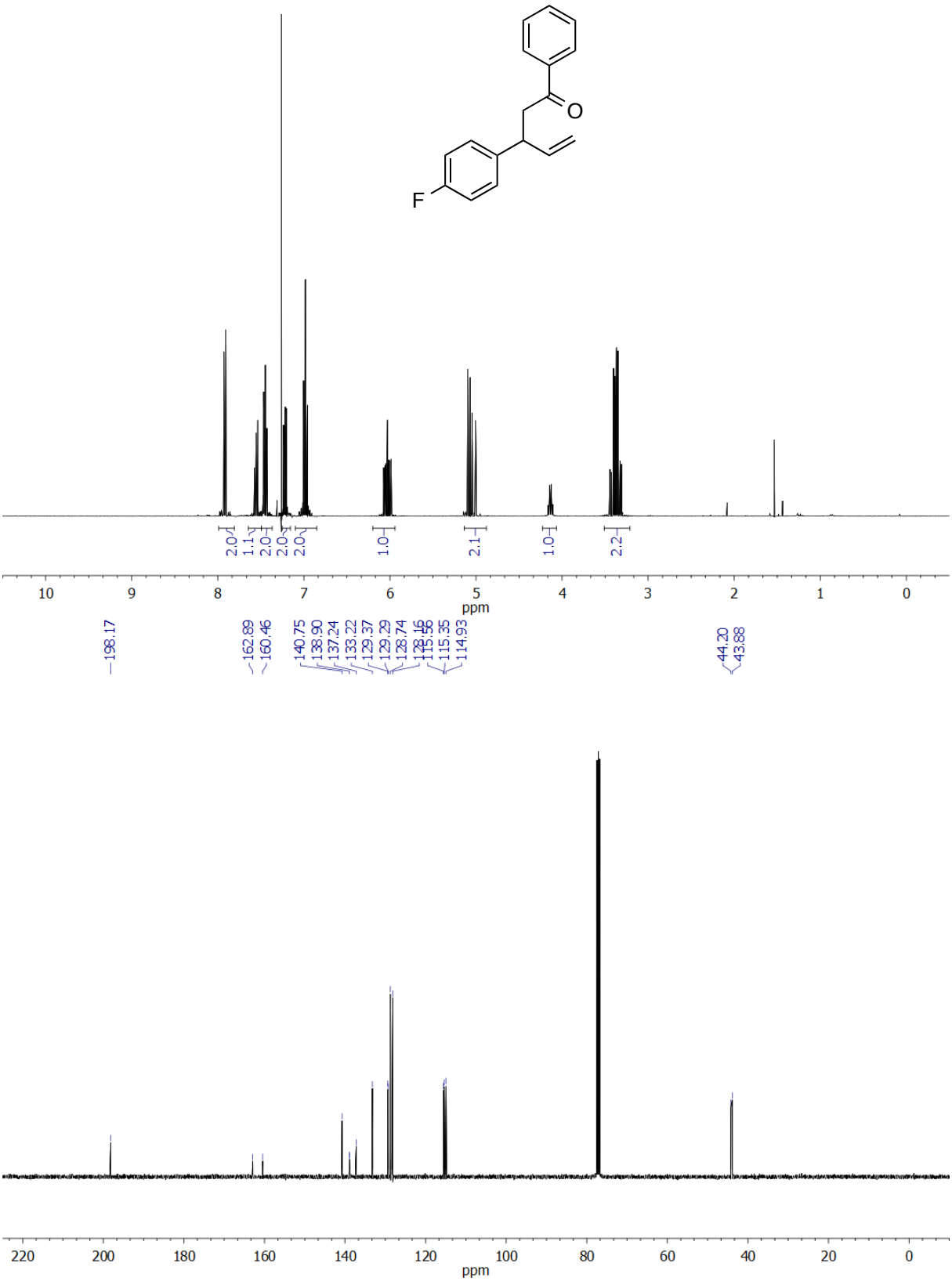


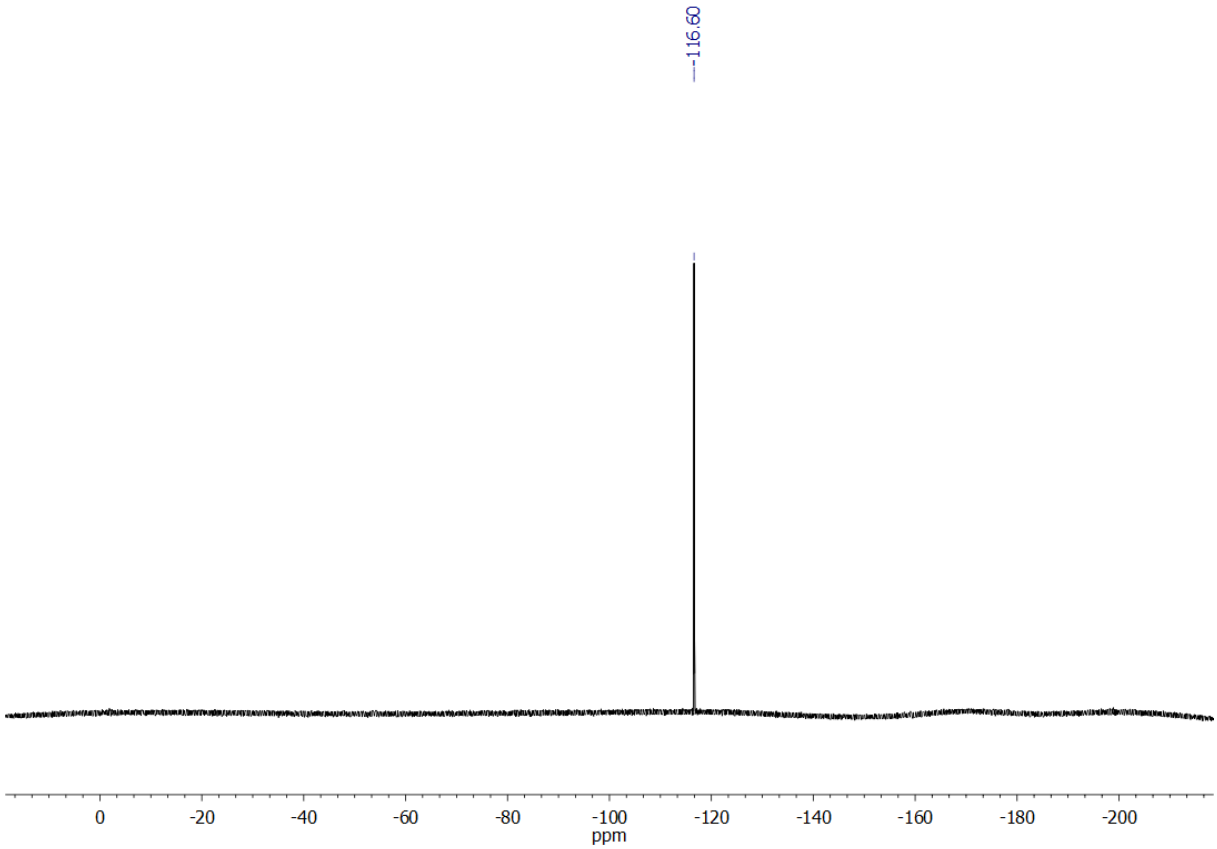
Preparation according to the general procedure. Flash column chromatography (PE : Toluene = 1:1) furnished the title compound (110.3 mg, 0.42 mmol, 84 %) as colorless oil. $\mathbf{R_f}$ (PE : EtOAc = 20:1) = 0.41; **^{1}H-NMR (400 MHz, $CDCl_3$)**: δ = 7.56 – 7.47 (m, 1H), 7.43 – 7.35 (m, 1H), 7.35 – 7.24 (m, 1H), 7.24 – 7.18 (m, 1H), 6.71 (d, *J* = 16.2 Hz, 1H), 6.04 (ddd, *J* = 17.1, 10.4, 6.8 Hz, 1H), 5.09 (ddd, *J* = 10.4, 1.3 Hz, 1H), 5.06 (ddd, *J* = 17.1, 1.4 Hz, 1H), 4.11 – 4.00 (m, 1H), 3.14 (dd, *J* = 15.9, 7.7 Hz, 1H), 3.06 (dd, *J* = 15.9, 6.9 Hz, 1H) ppm; **^{13}C-NMR (101 MHz, $CDCl_3$)**: δ = 198.31, 143.15, 142.82, 140.75, 134.61, 130.58, 129.03, 128.70, 128.39, 127.81, 126.69, 126.49, 114.82, 46.48, 44.89 ppm. The analytical data are in accordance with the literature.[19]

NMR spectra
1-phenyl-3-phenylpent-4-en-1-one (3aa)

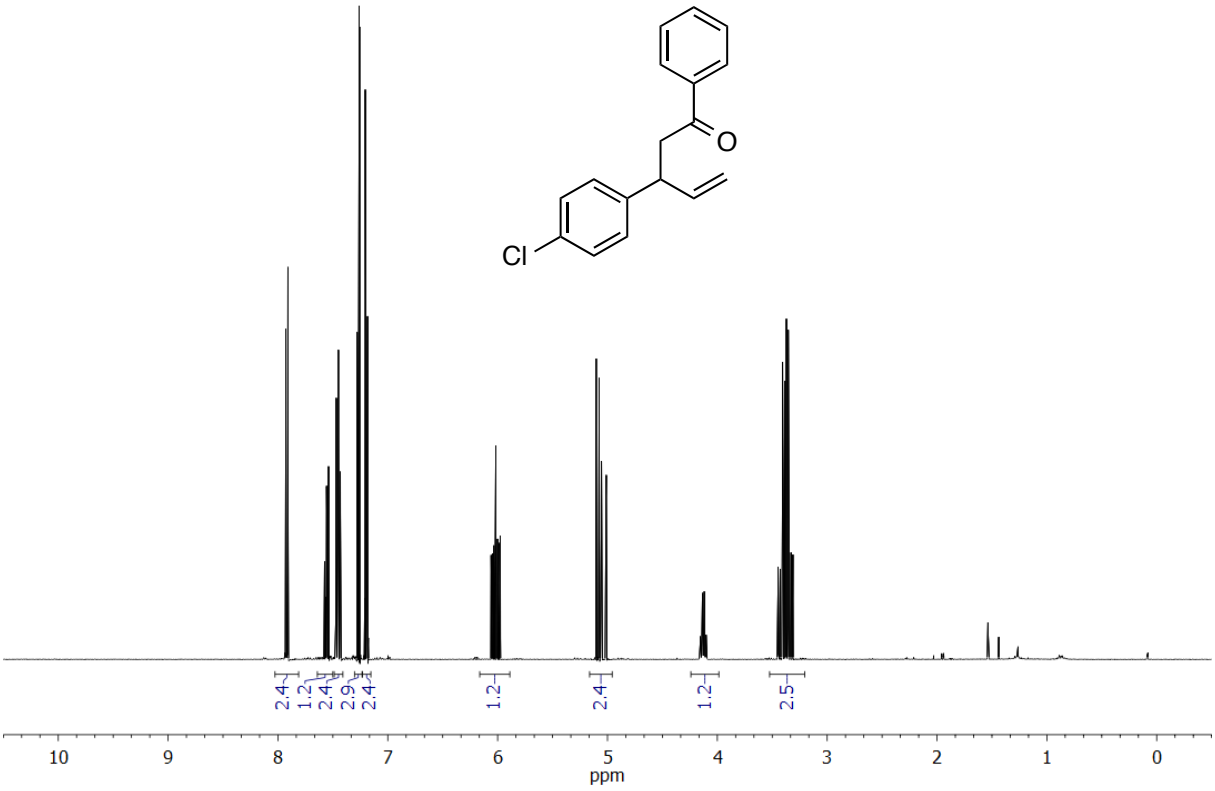


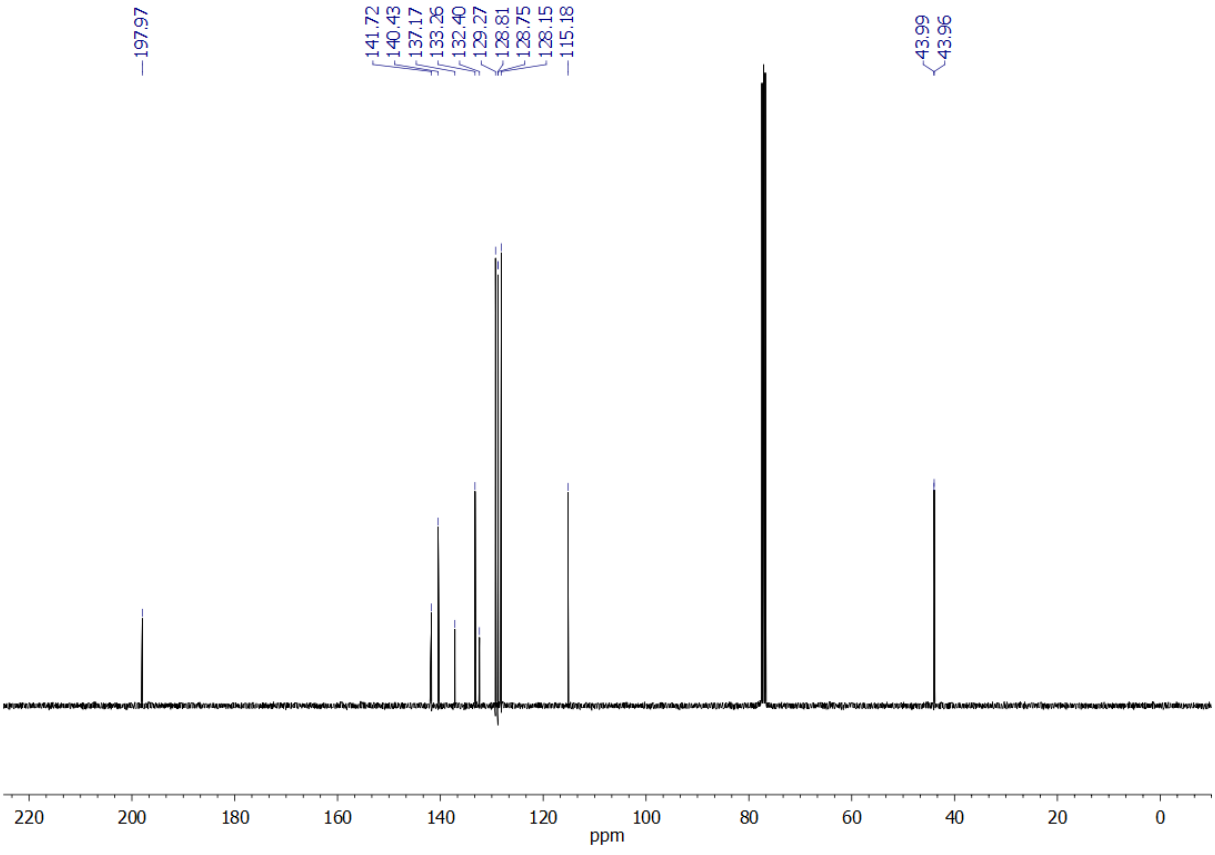
3-(4-fluorophenyl)-1-phenylpent-4-en-1-one (3ba)



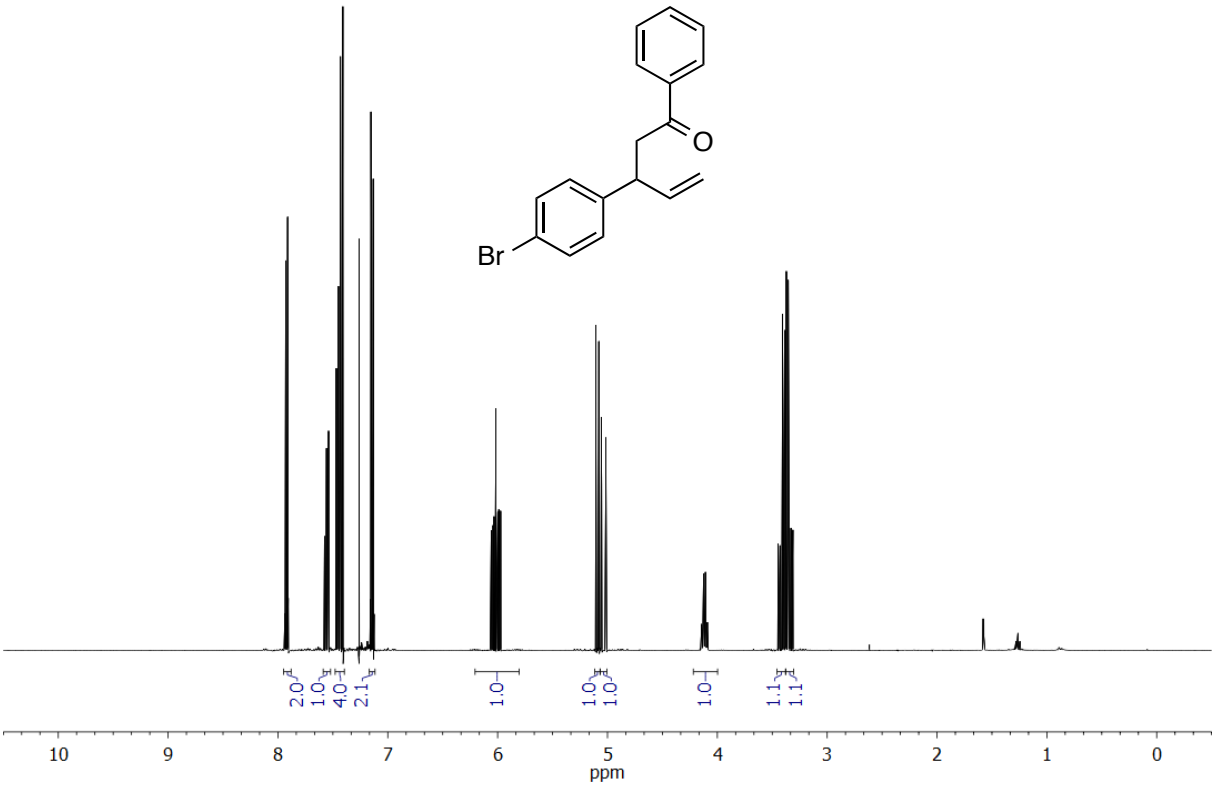

-116.60
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm

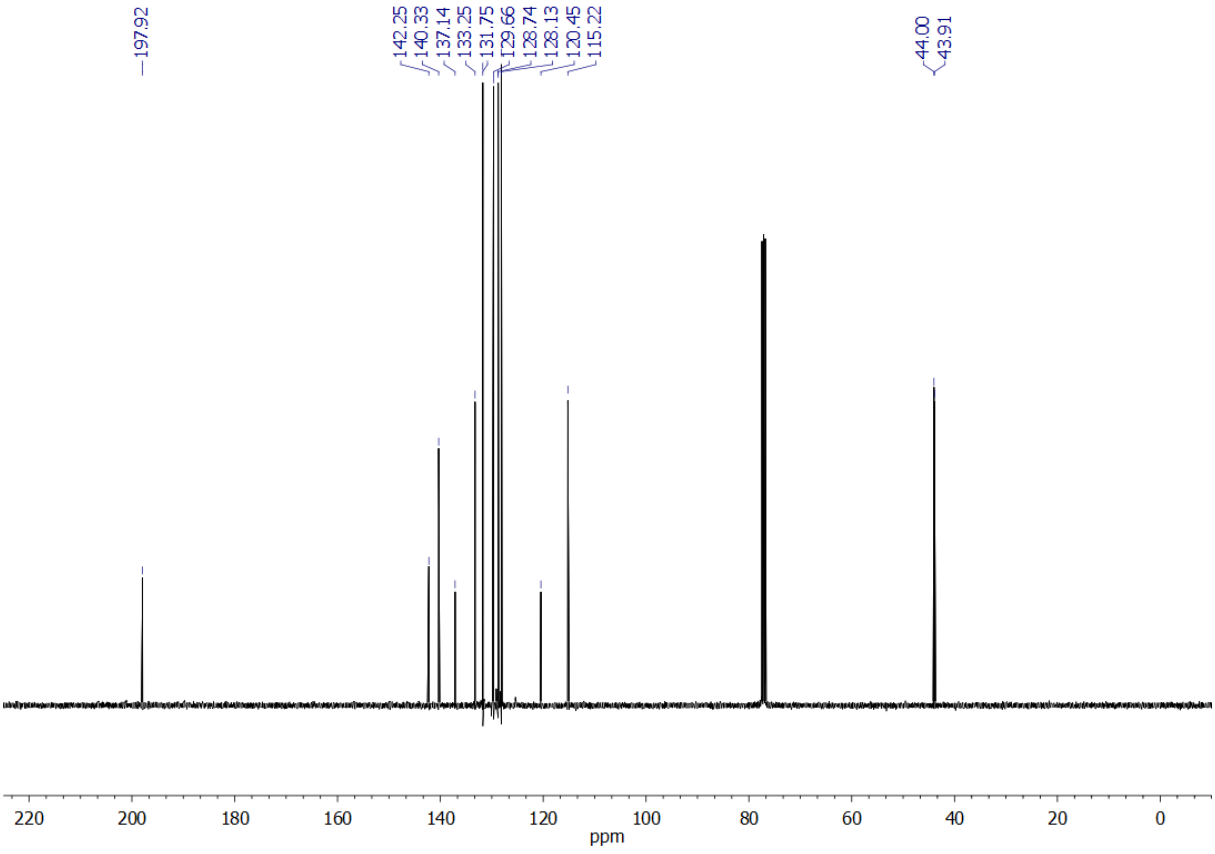
3-(4-chlorophenyl)-1-phenylpent-4-en-1-one (3ca)



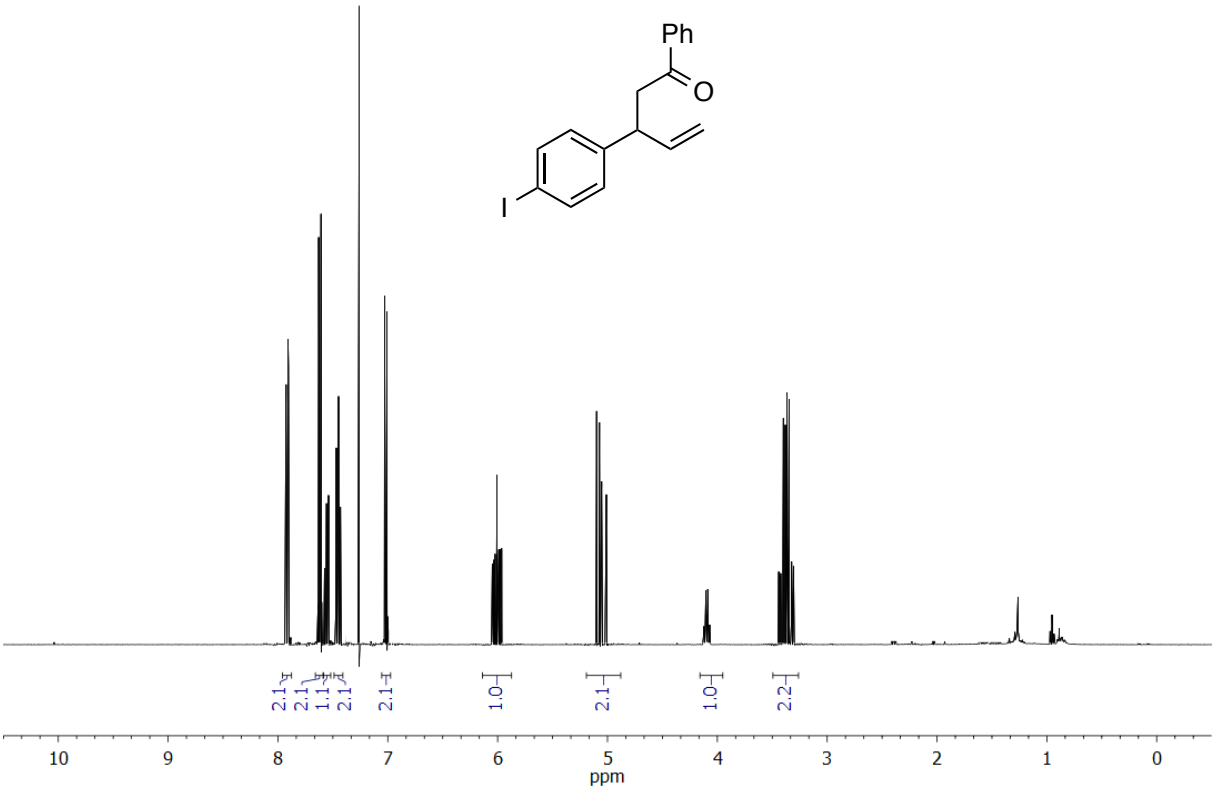


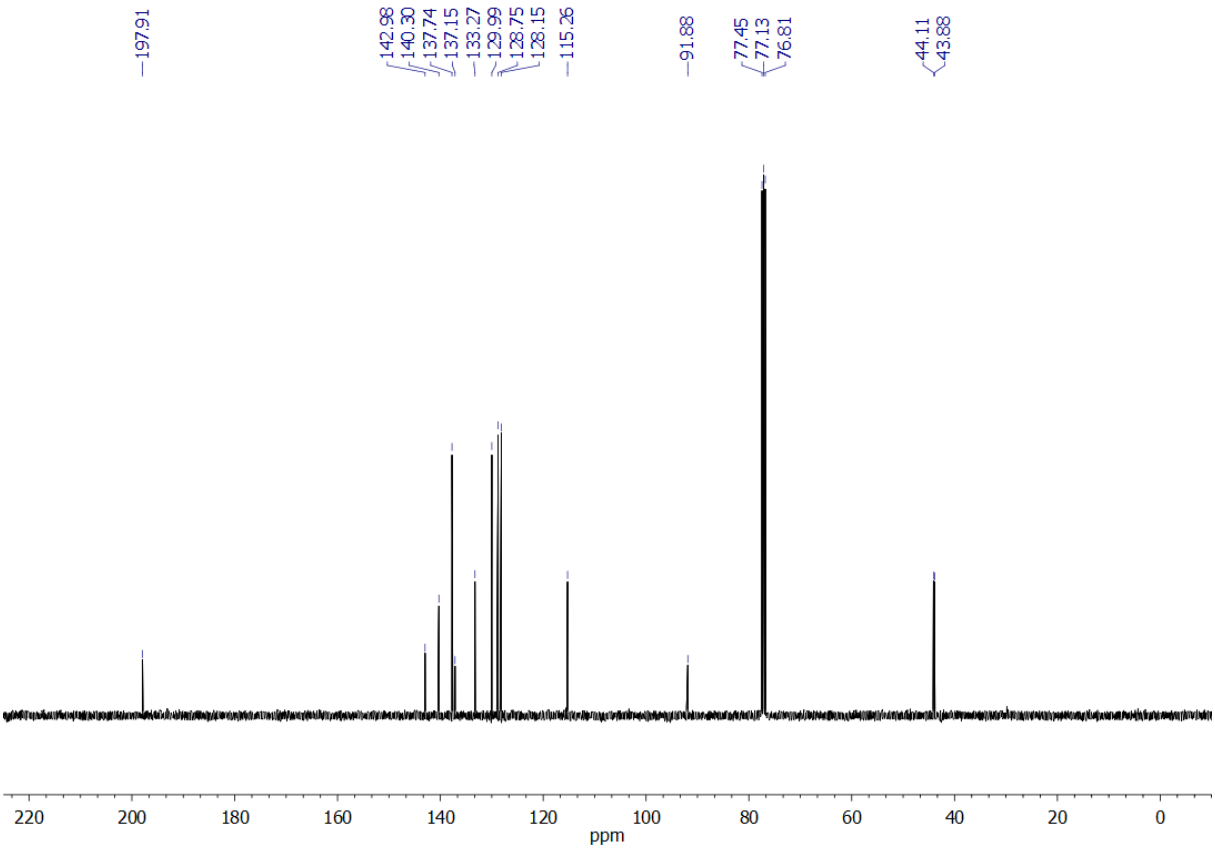
3-(4-bromophenyl)-1-phenylpent-4-en-1-one (3da)



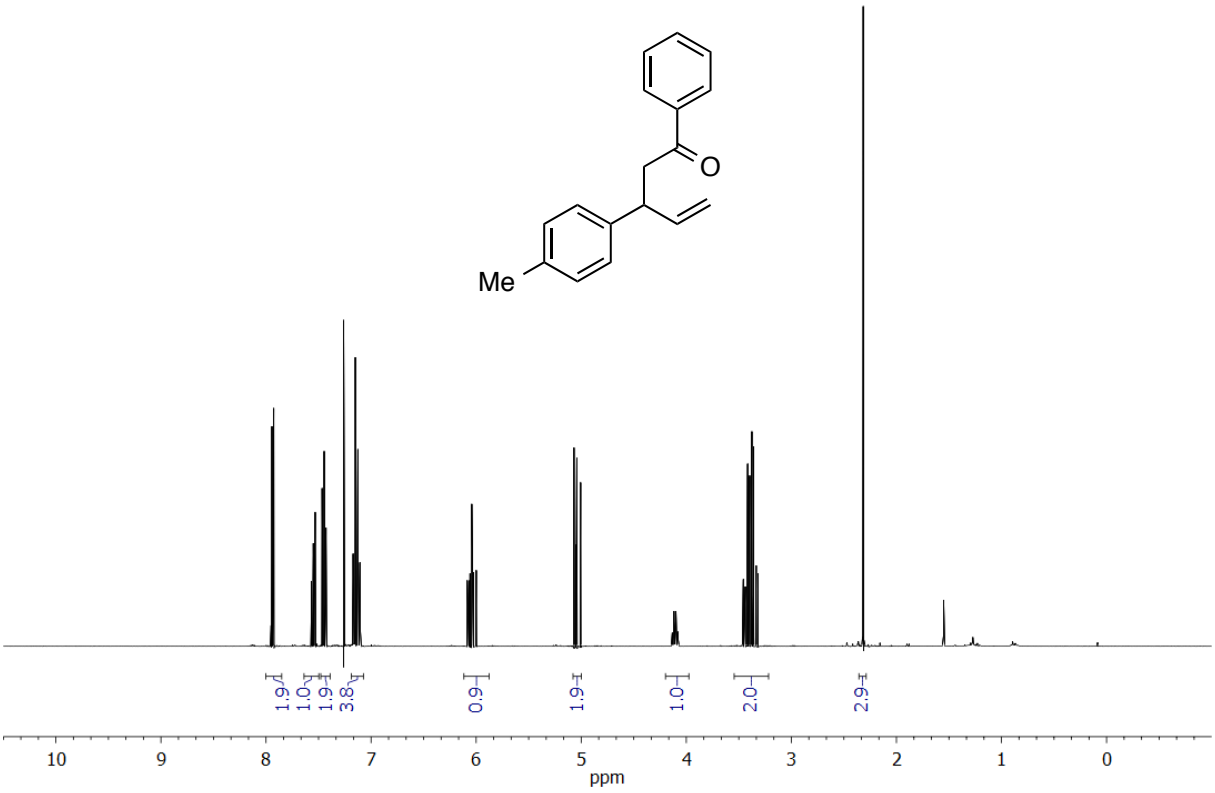


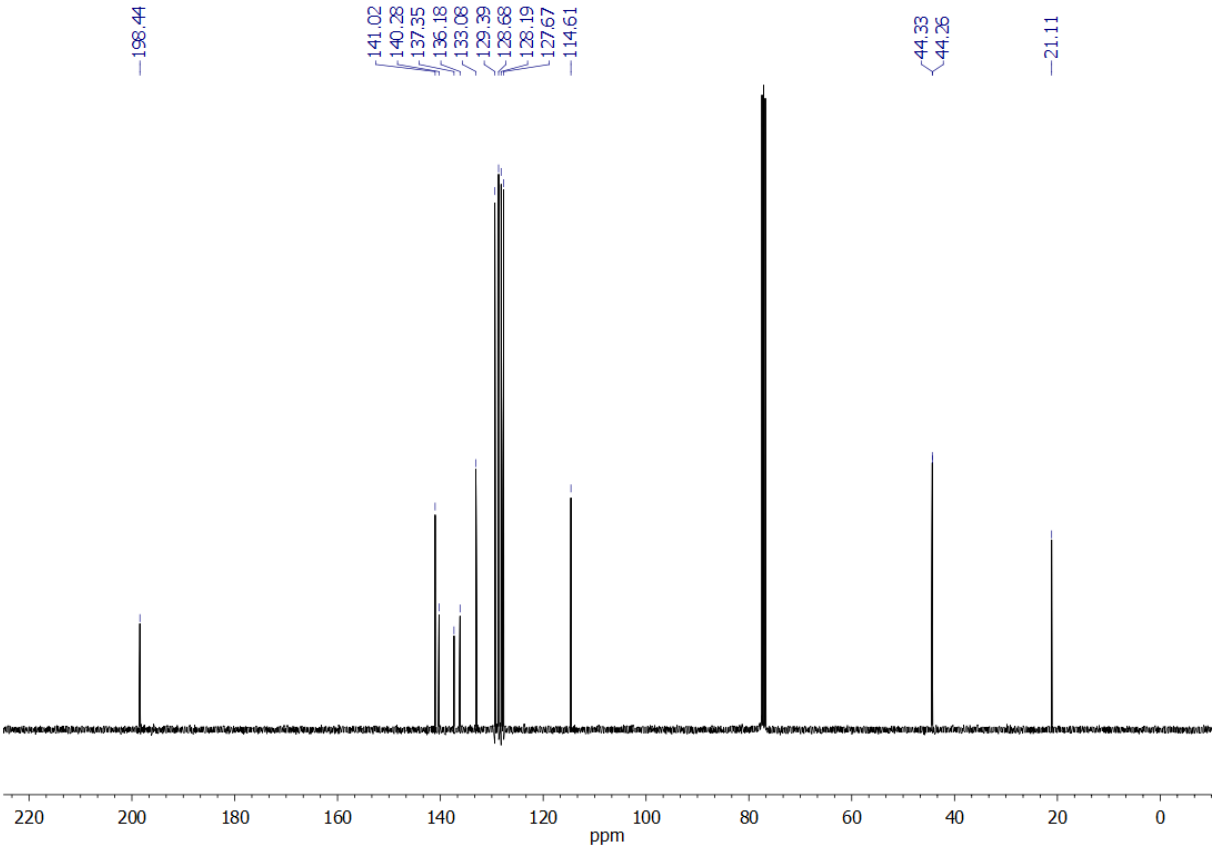
3-(4-iodophenyl)-1-phenylpent-4-en-1-one (3ea)



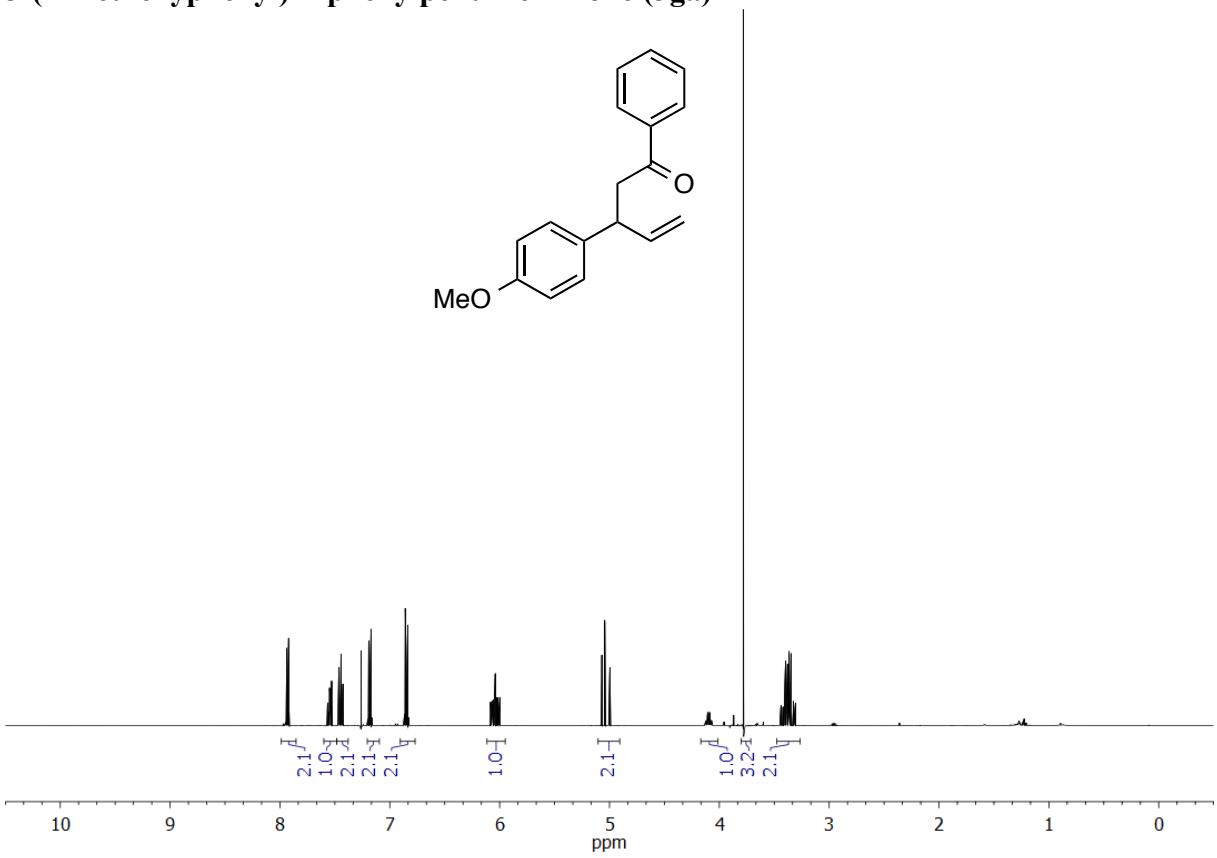


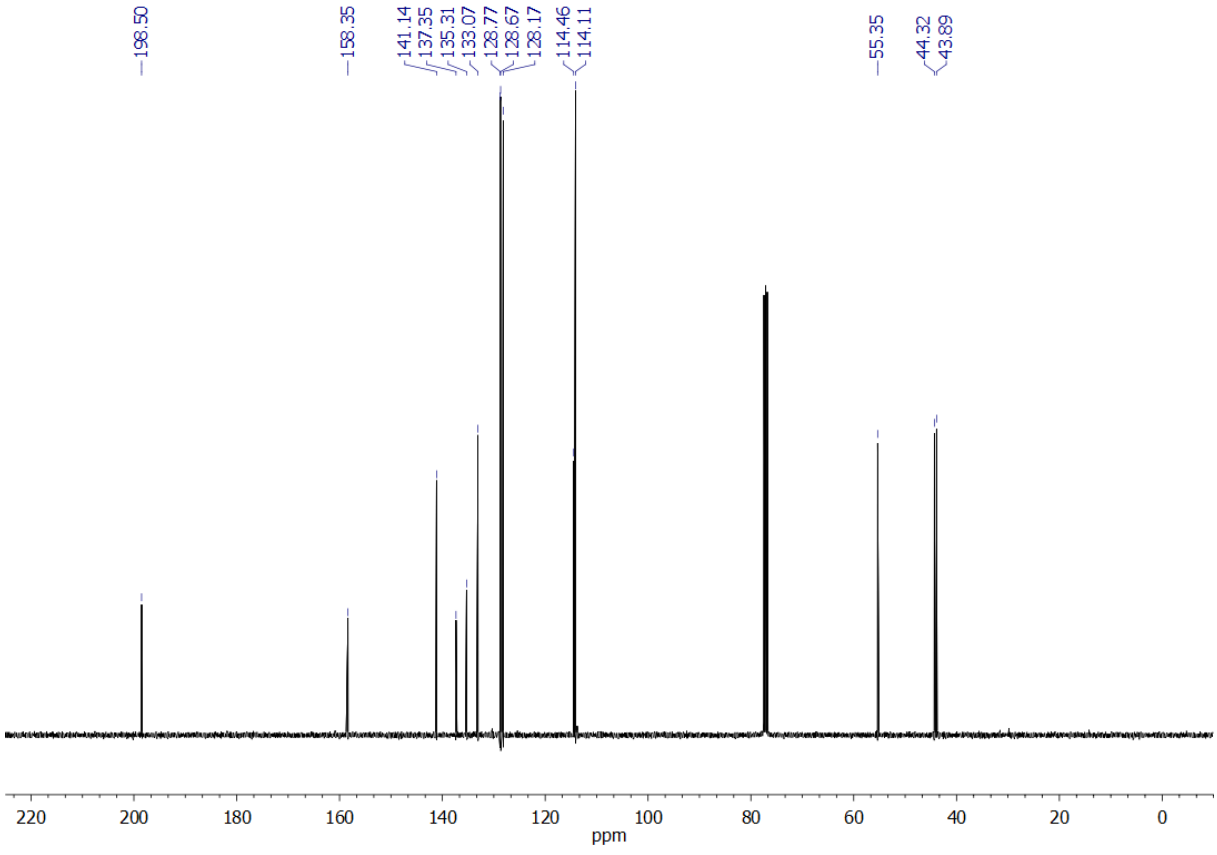
1-phenyl-3-(p-tolyl)pent-4-en-1-one (3fa)



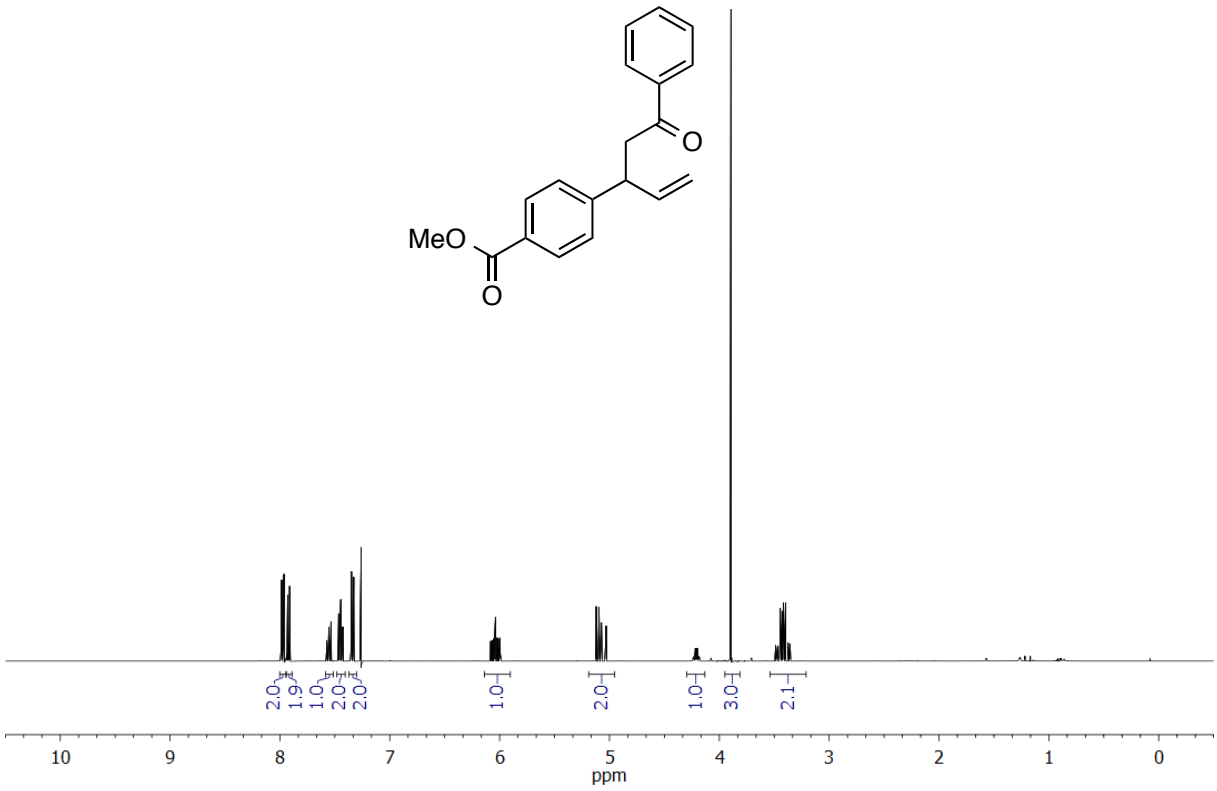


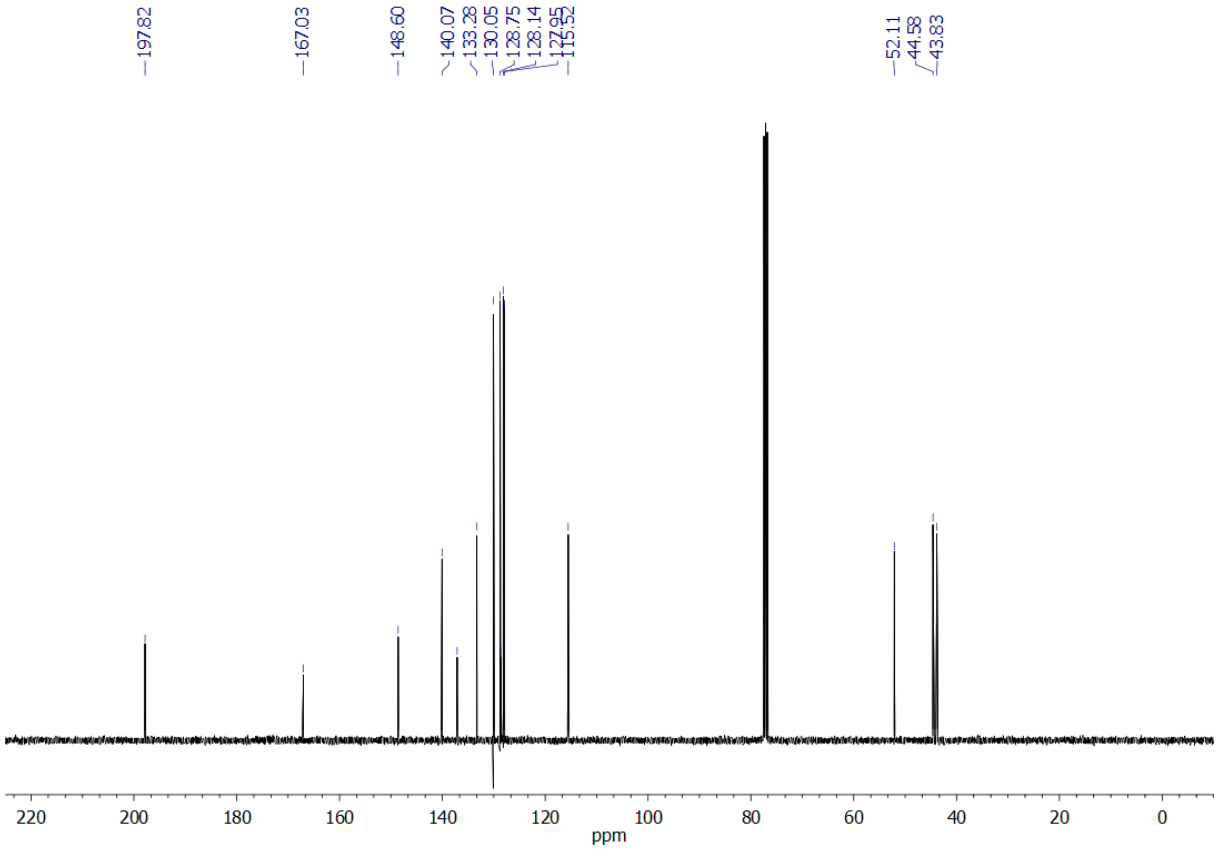
3-(4-methoxyphenyl)-1-phenylpent-4-en-1-one (3ga)



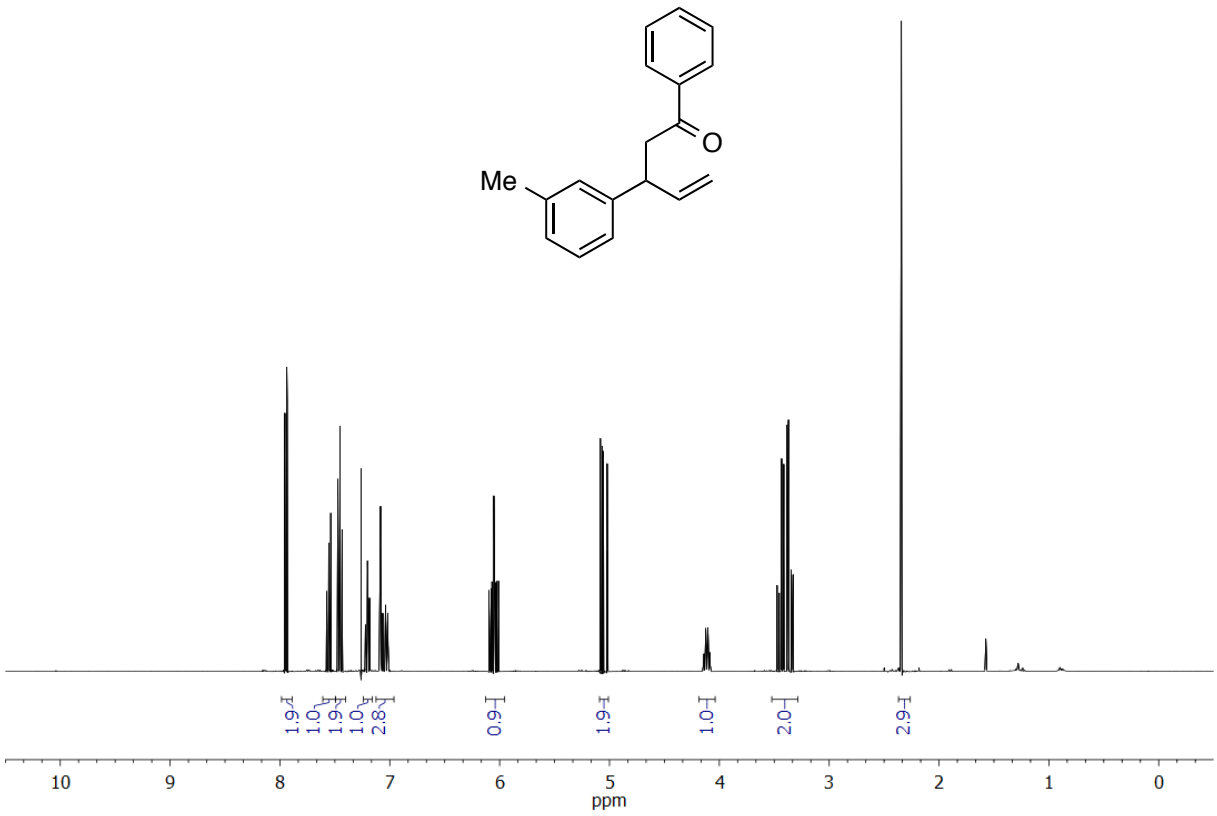


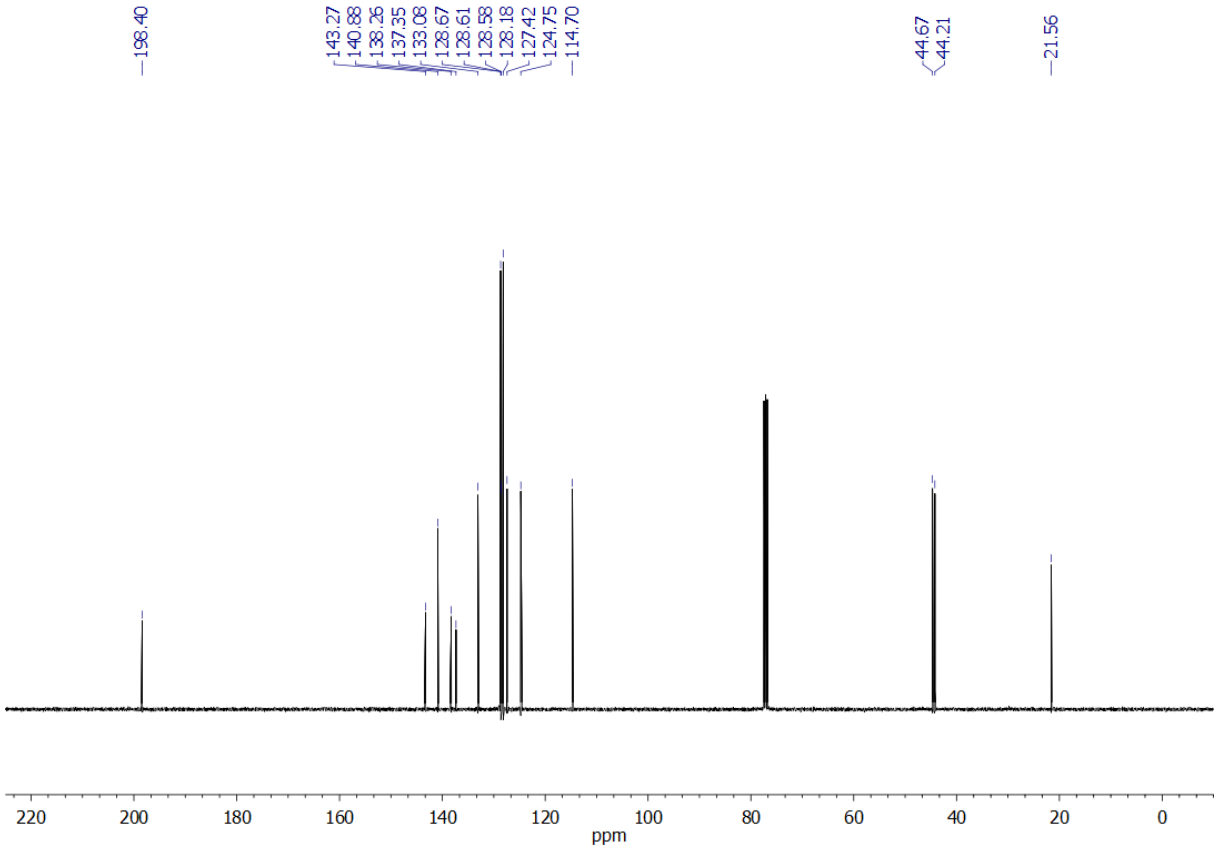
methyl 4-(5-oxo-5-phenylpent-1-en-3-yl)benzoate (3ha)



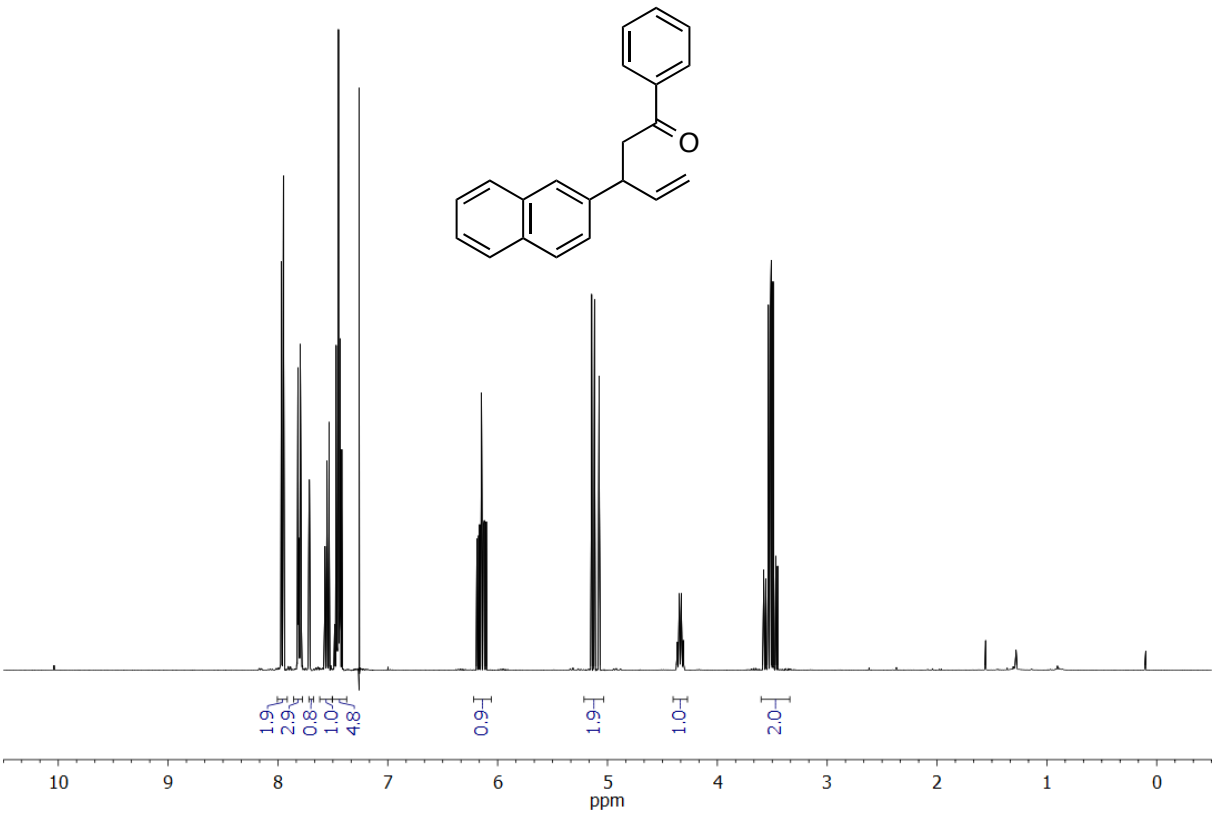


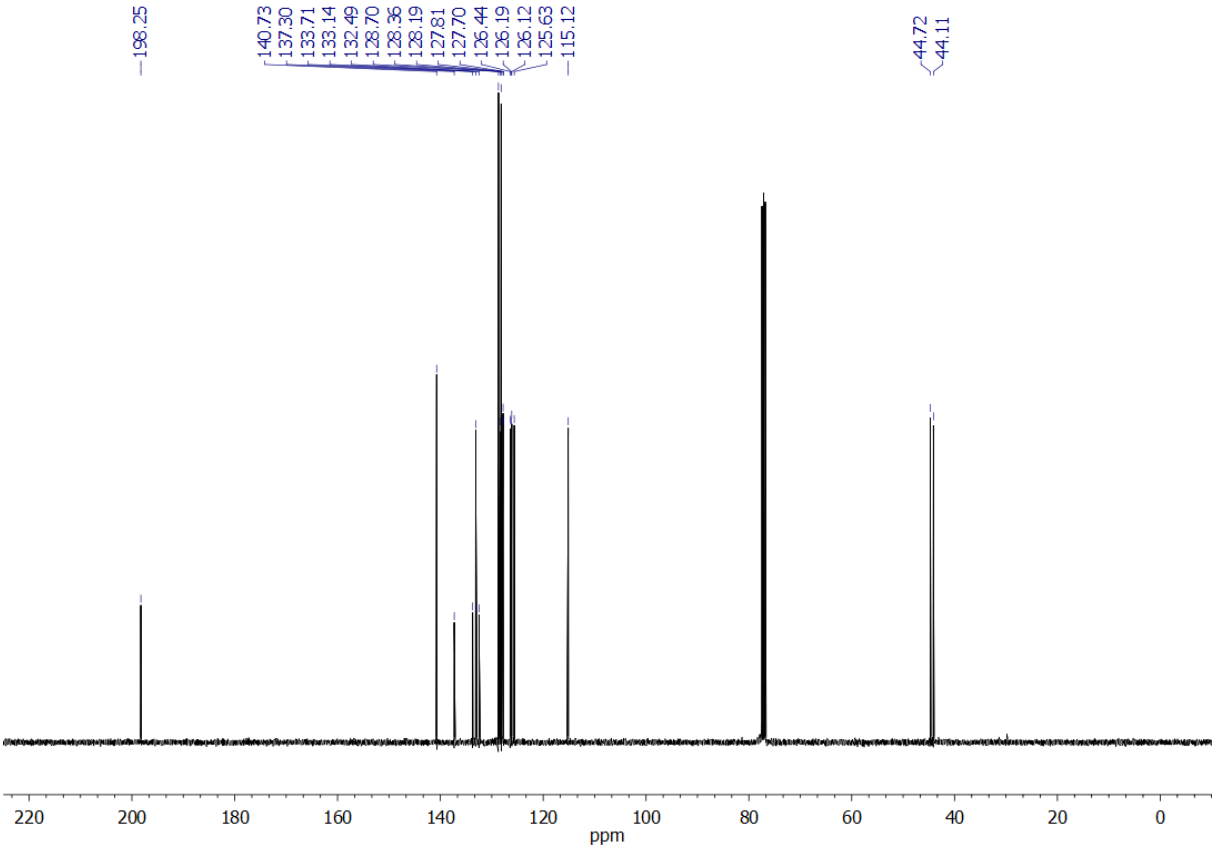
1-phenyl-3-(m-tolyl)pent-4-en-1-one (3ia)



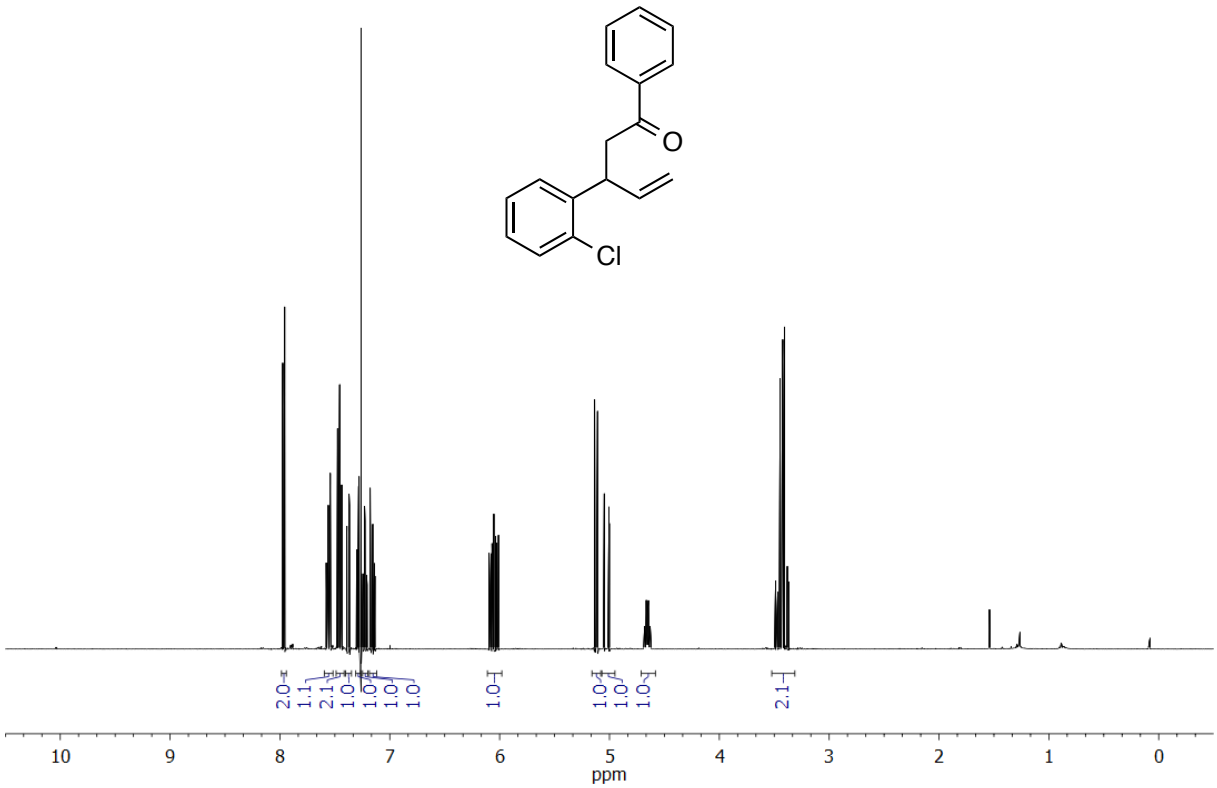


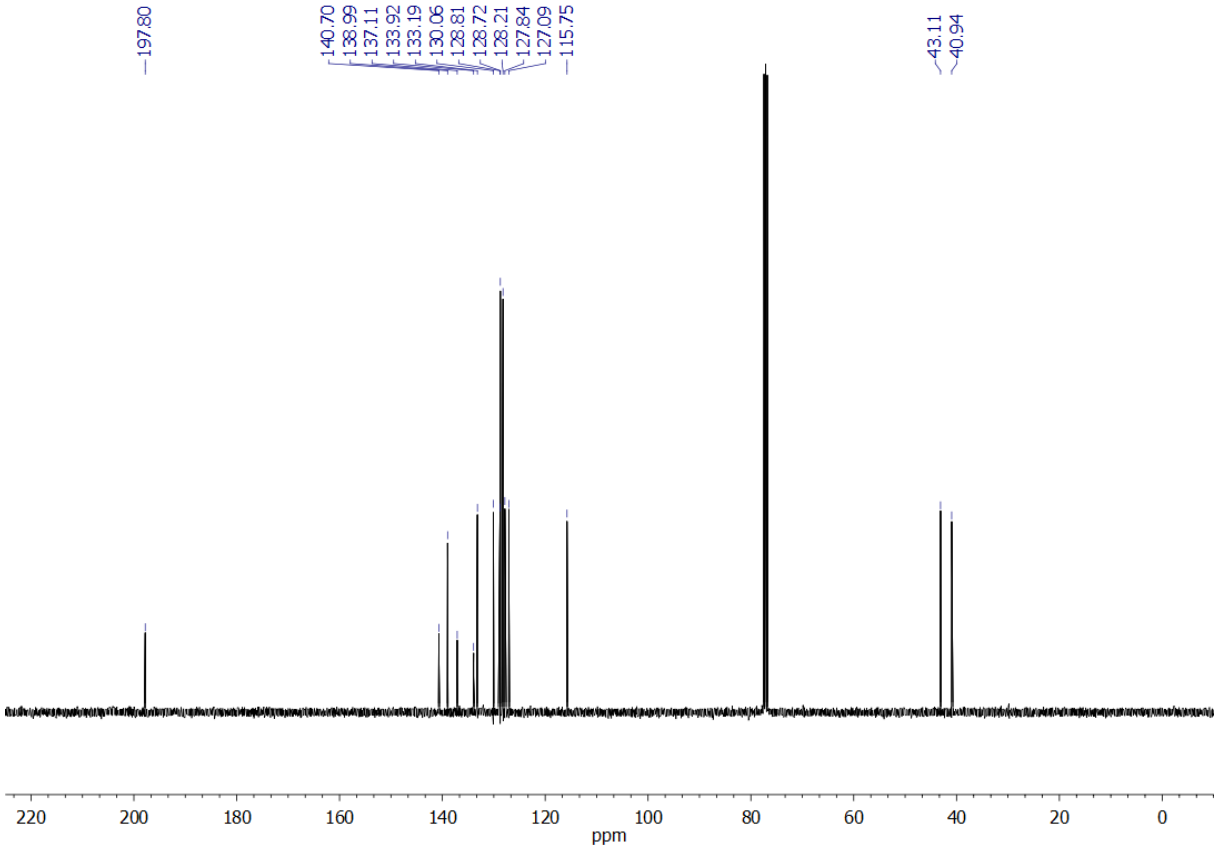
3-(2-naphthyl)-1-phenylpent-4-en-1-one (3ja)



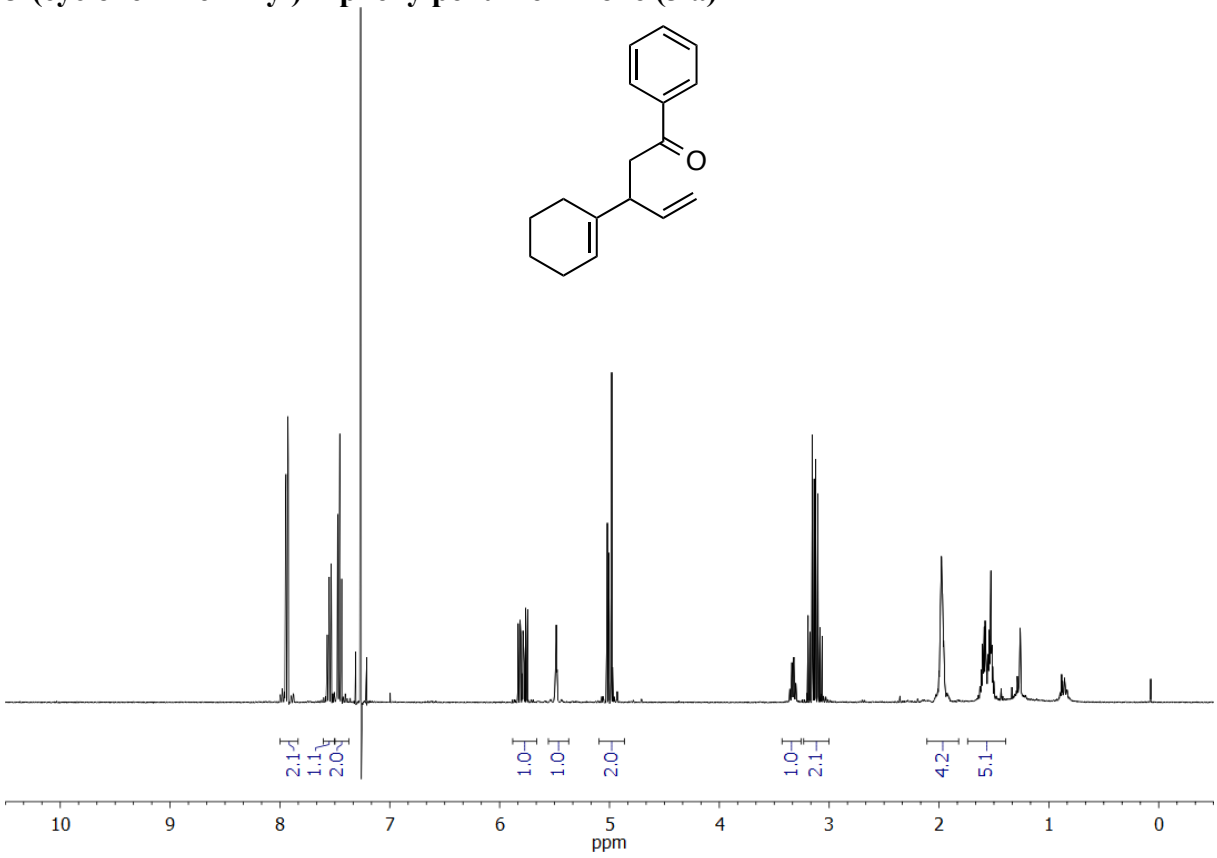


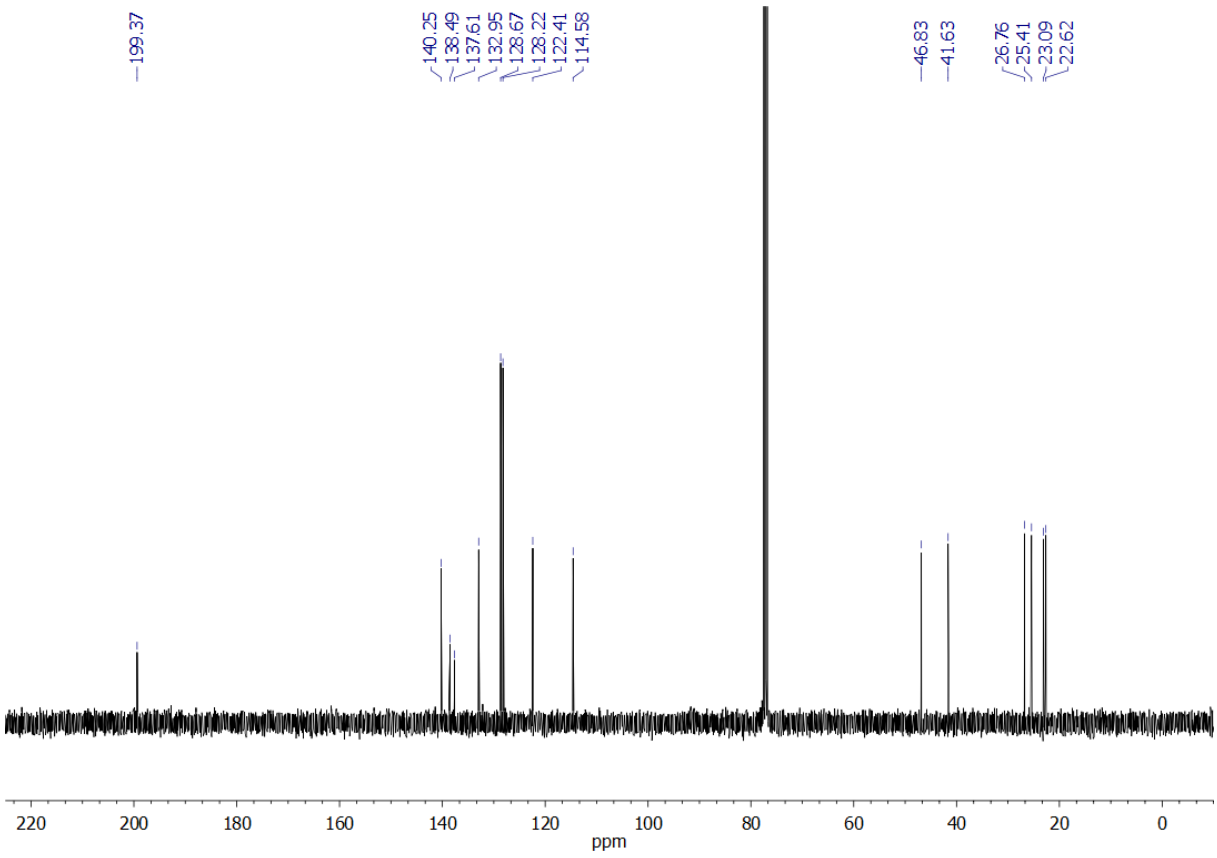
3-(2-chlorophenyl)-1-phenylpent-4-en-1-one (3ka)



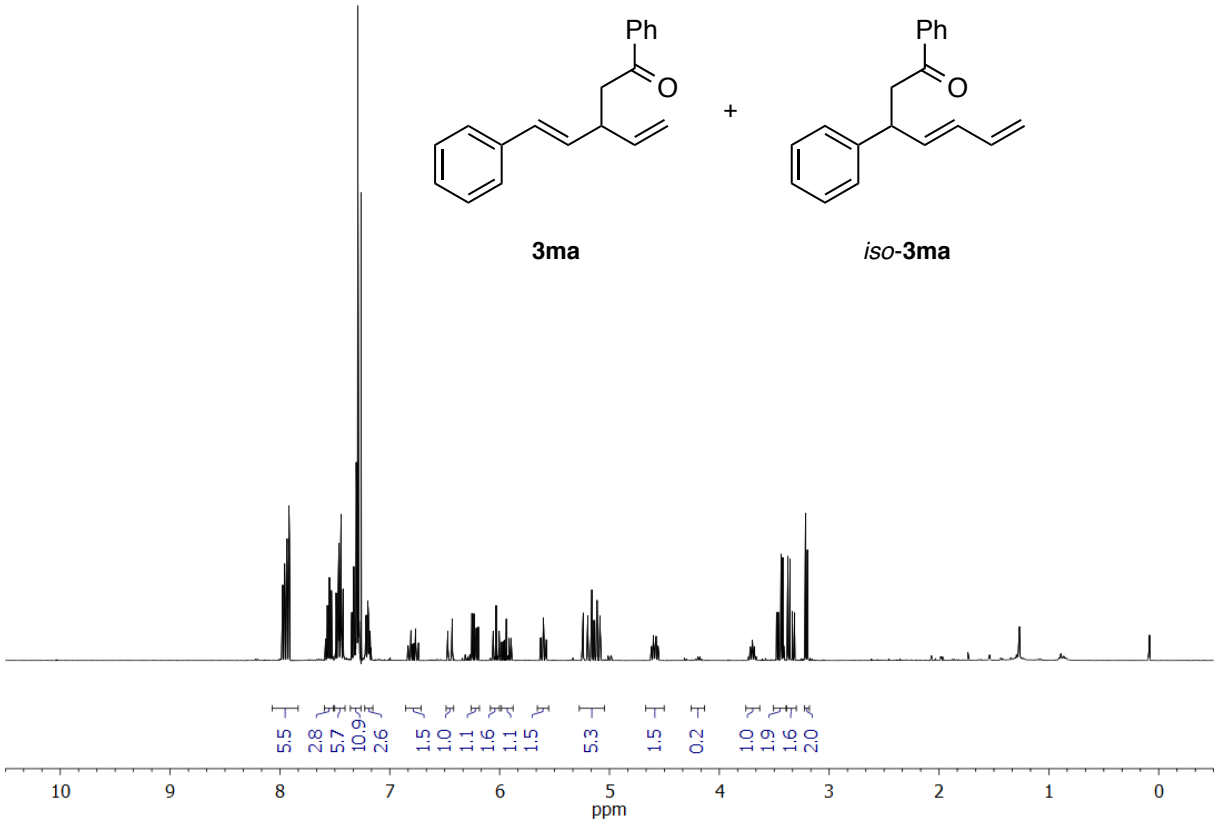


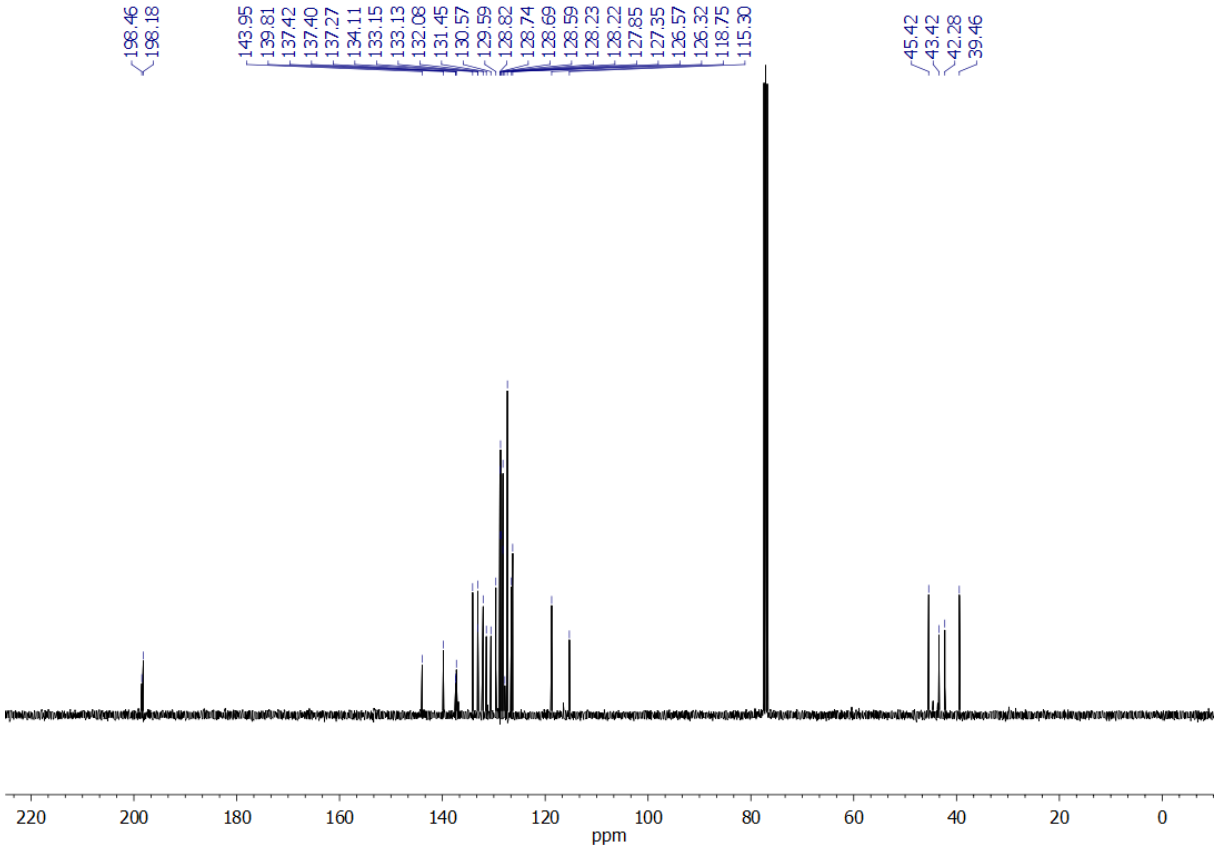
3-(cyclohex-1-en-1-yl)-1-phenylpent-4-en-1-one (3la)



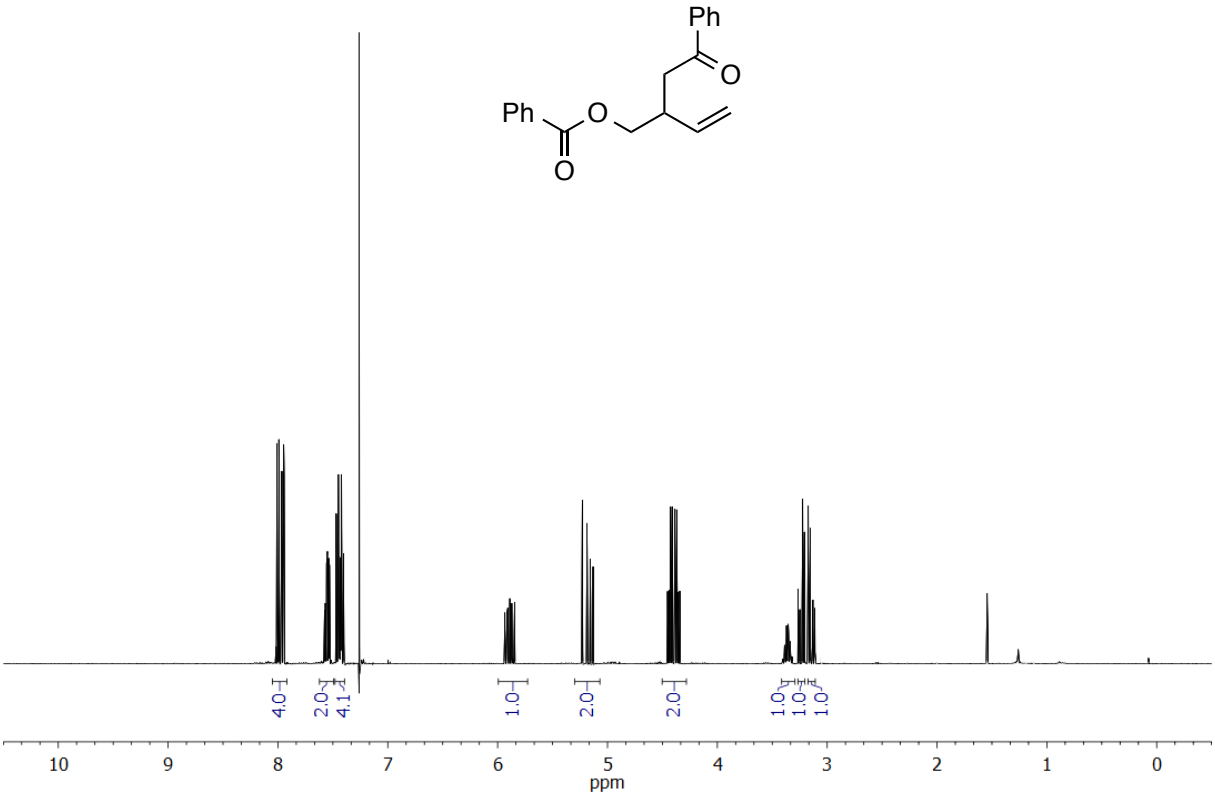


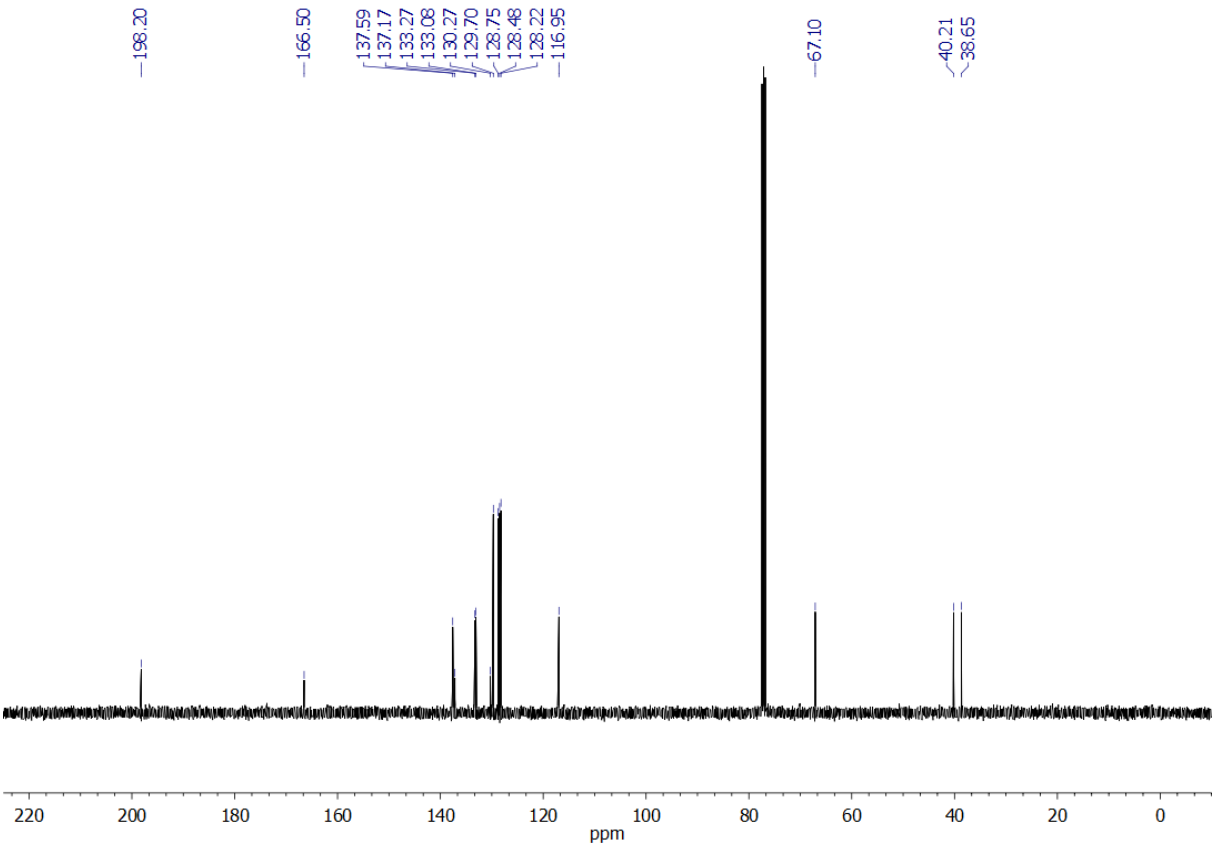
(*E*)-1,5-diphenyl-3-vinylpent-4-en-1-one (3ma)



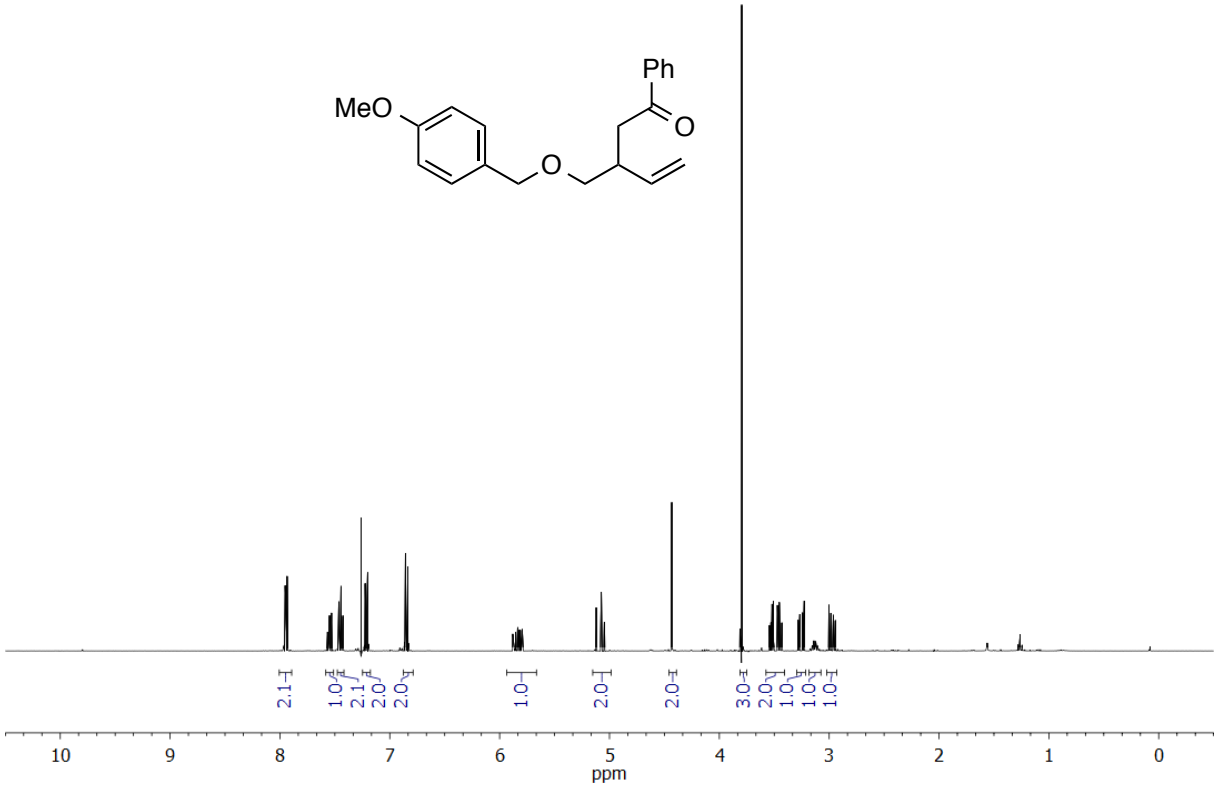


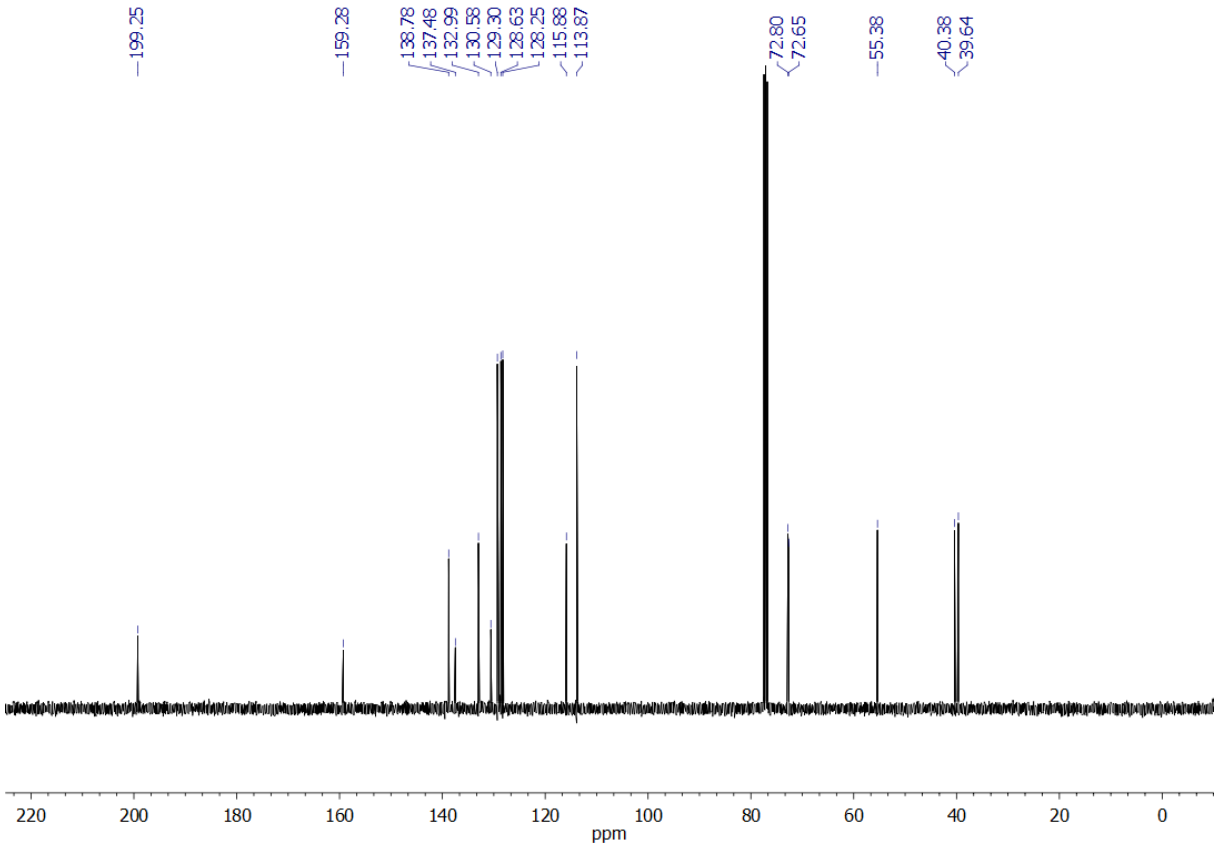
2-(2-oxo-2-phenylethyl)but-3-en-1-yl benzoate (3na)



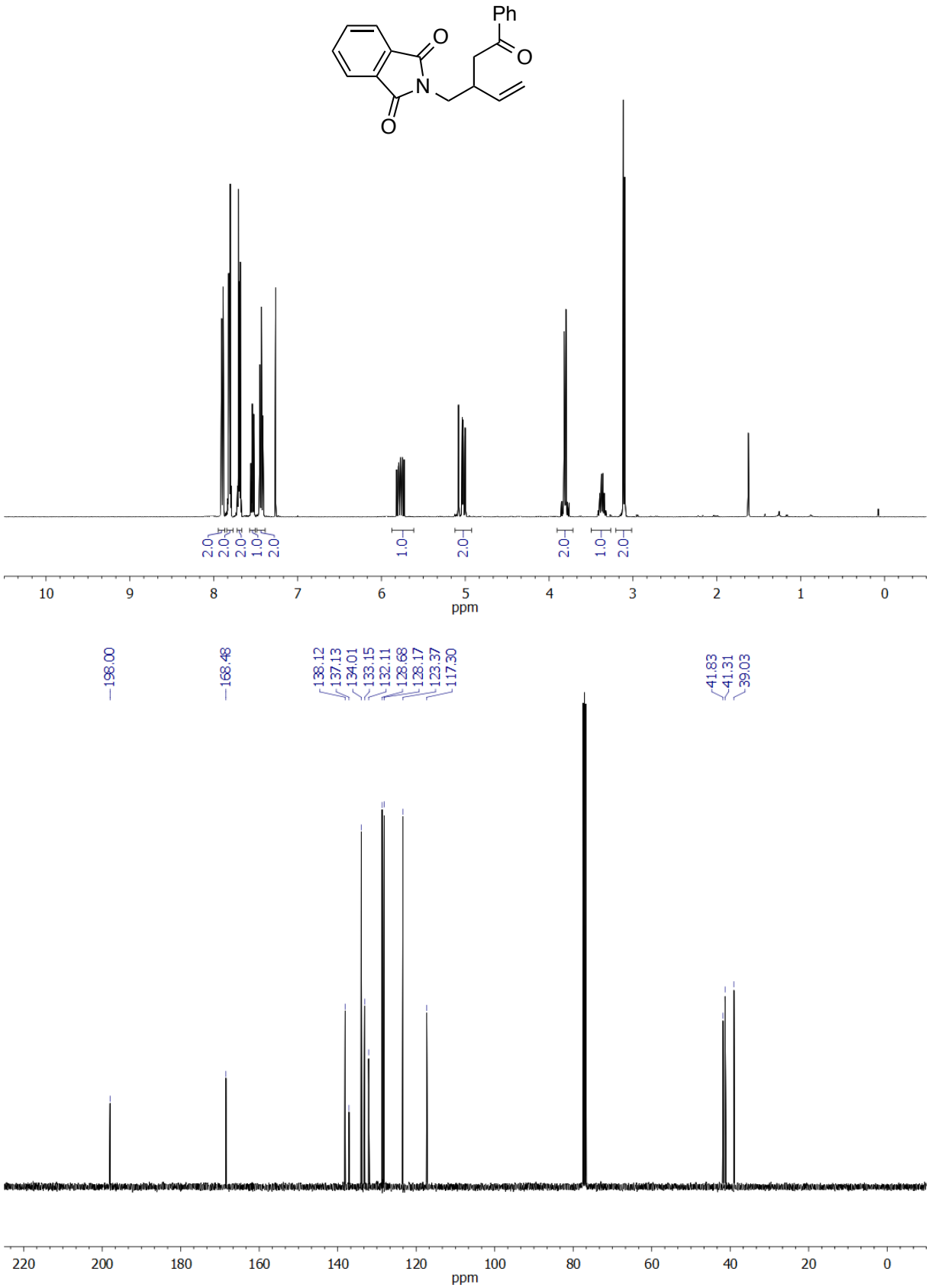


3-(((4-methoxybenzyl)oxy)methyl)-1-phenylpent-4-en-1-one (3oa)

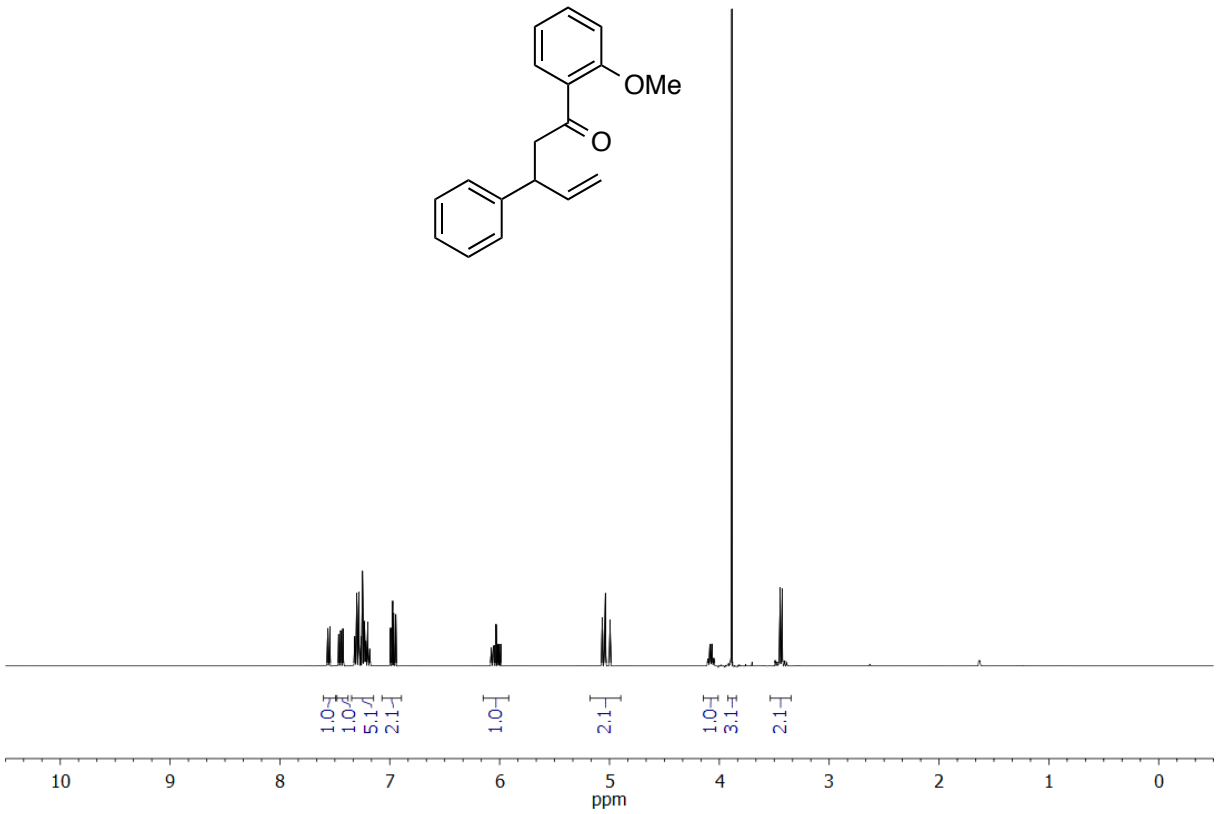


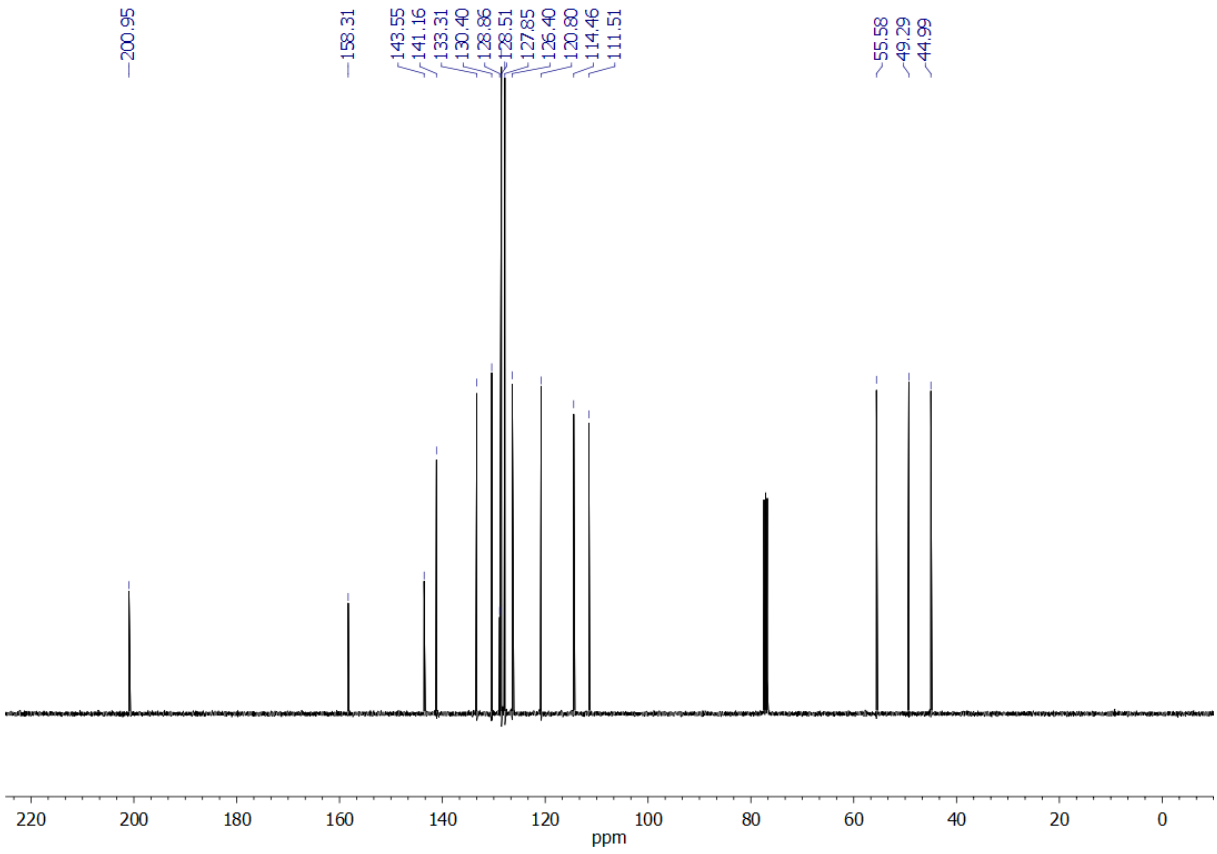


2-(2-(2-oxo-2-phenylethyl)but-3-en-1-yl)isoindoline-1,3-dione (3pa)

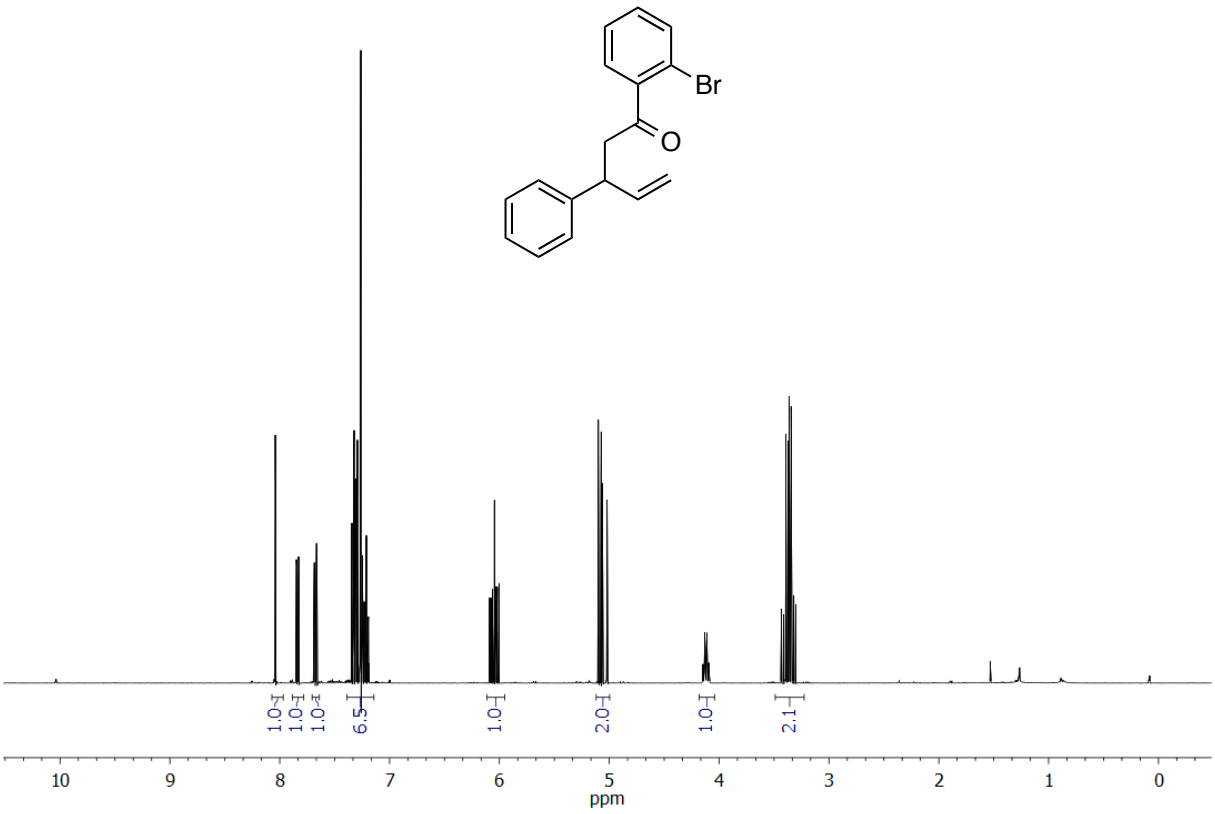


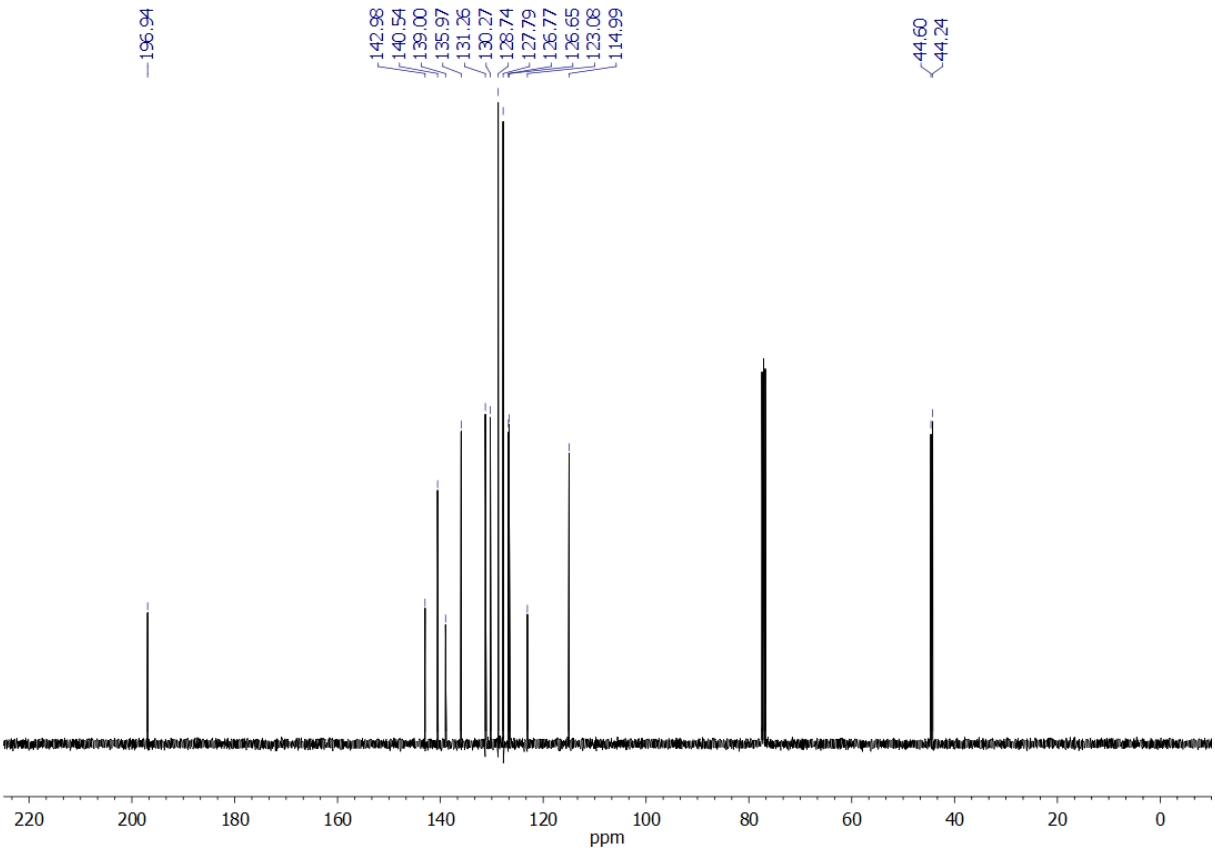
1-(2-methoxyphenyl)-3-phenylpent-4-en-1-one (3ab)



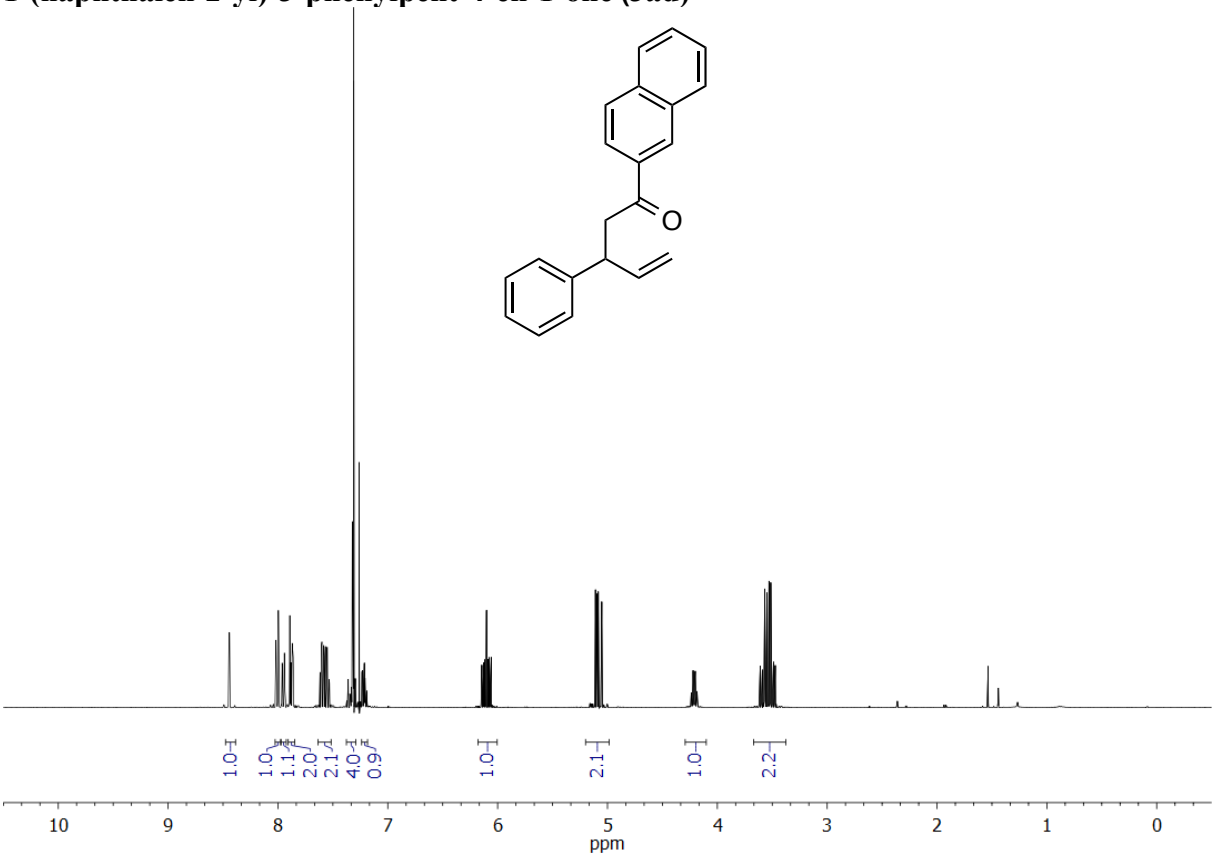


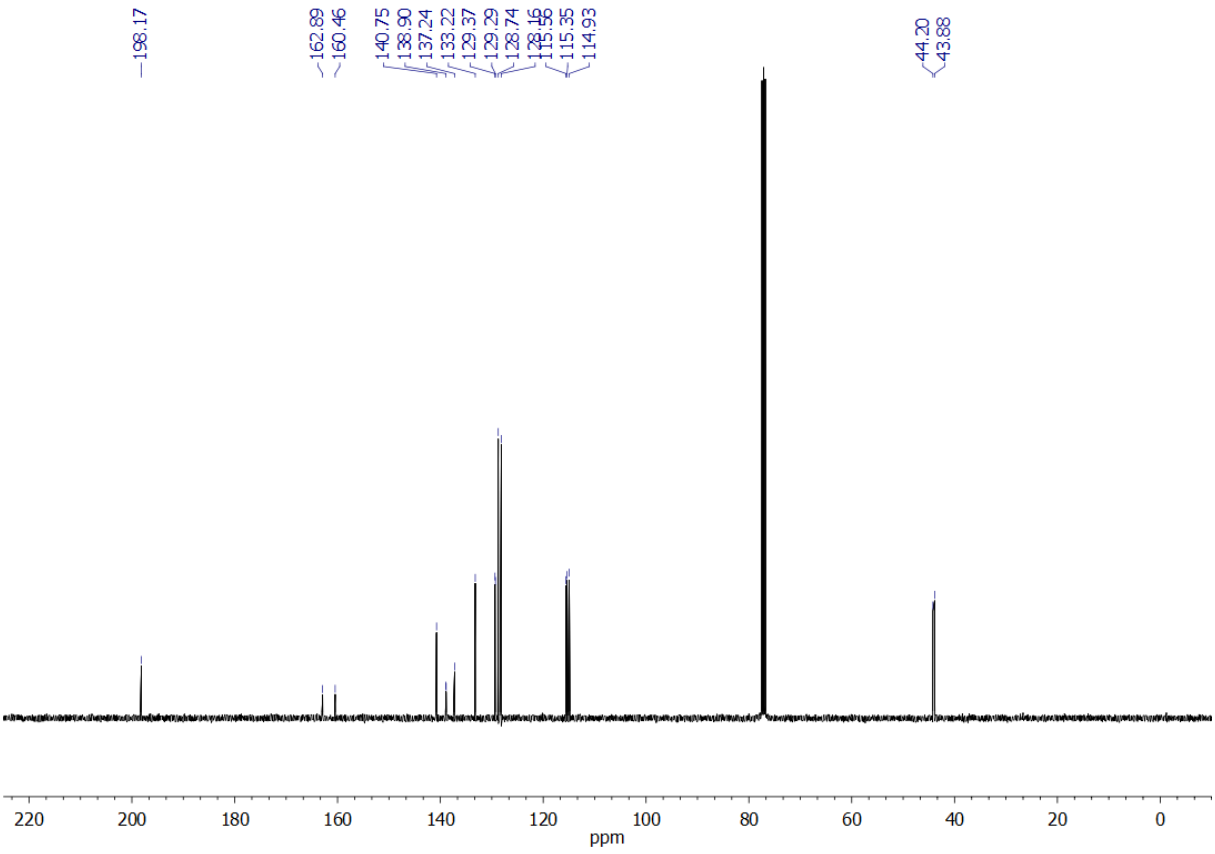
1-(3-bromophenyl)-3-phenylpent-4-en-1-one (3ac)



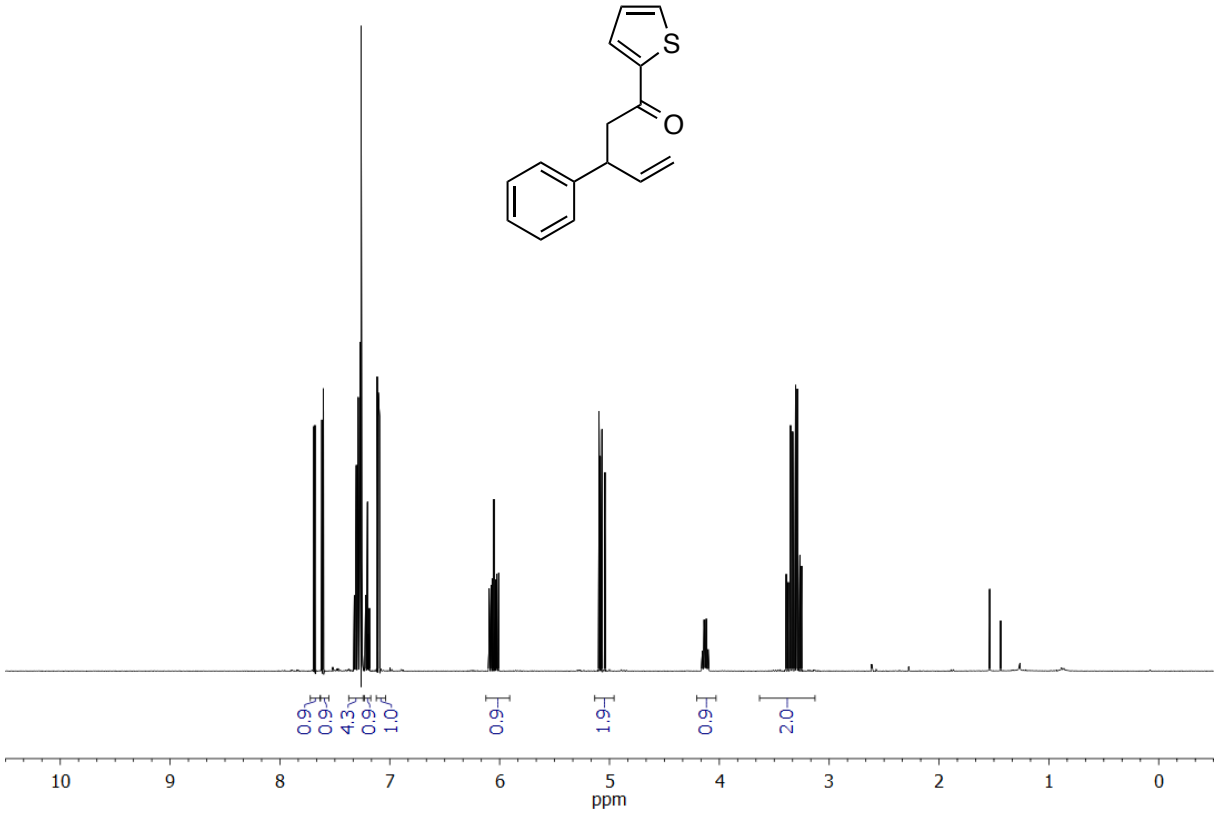


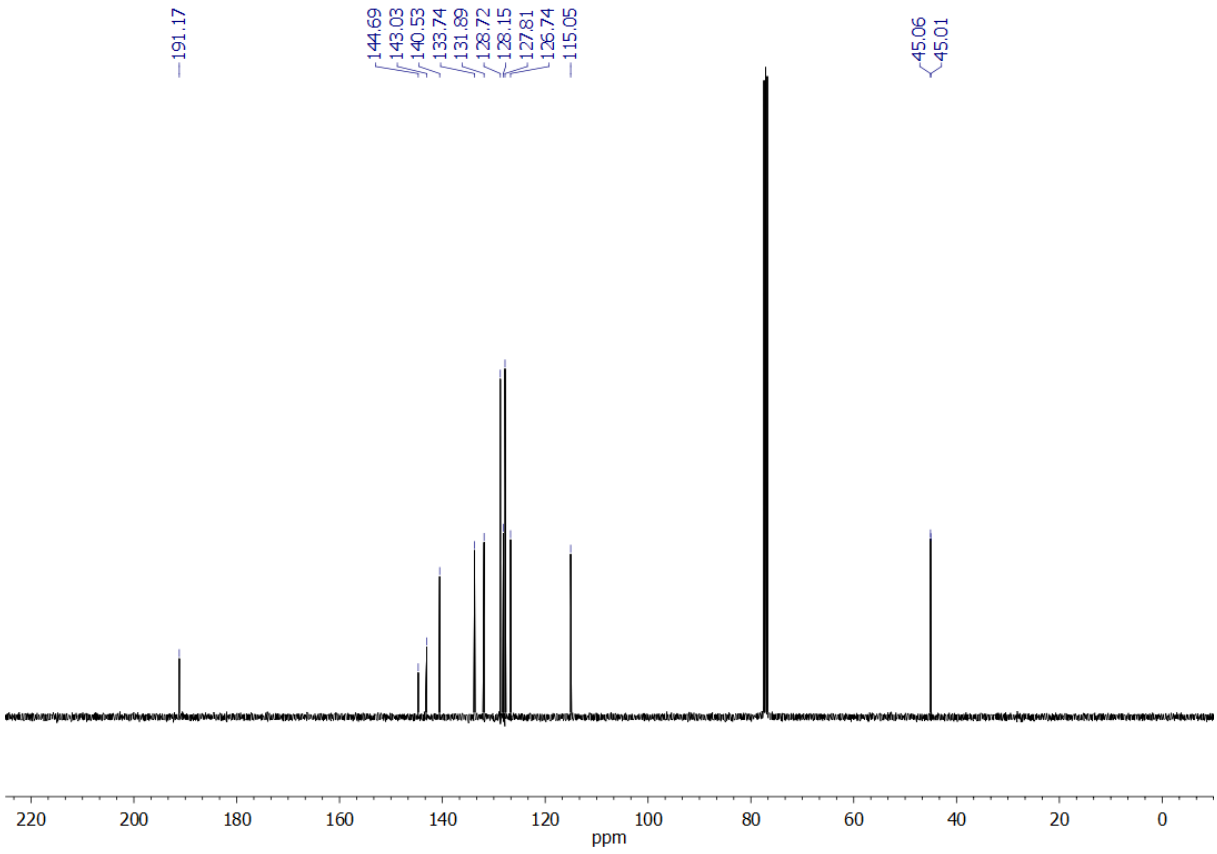
1-(naphthalen-2-yl)-3-phenylpent-4-en-1-one (3ad)



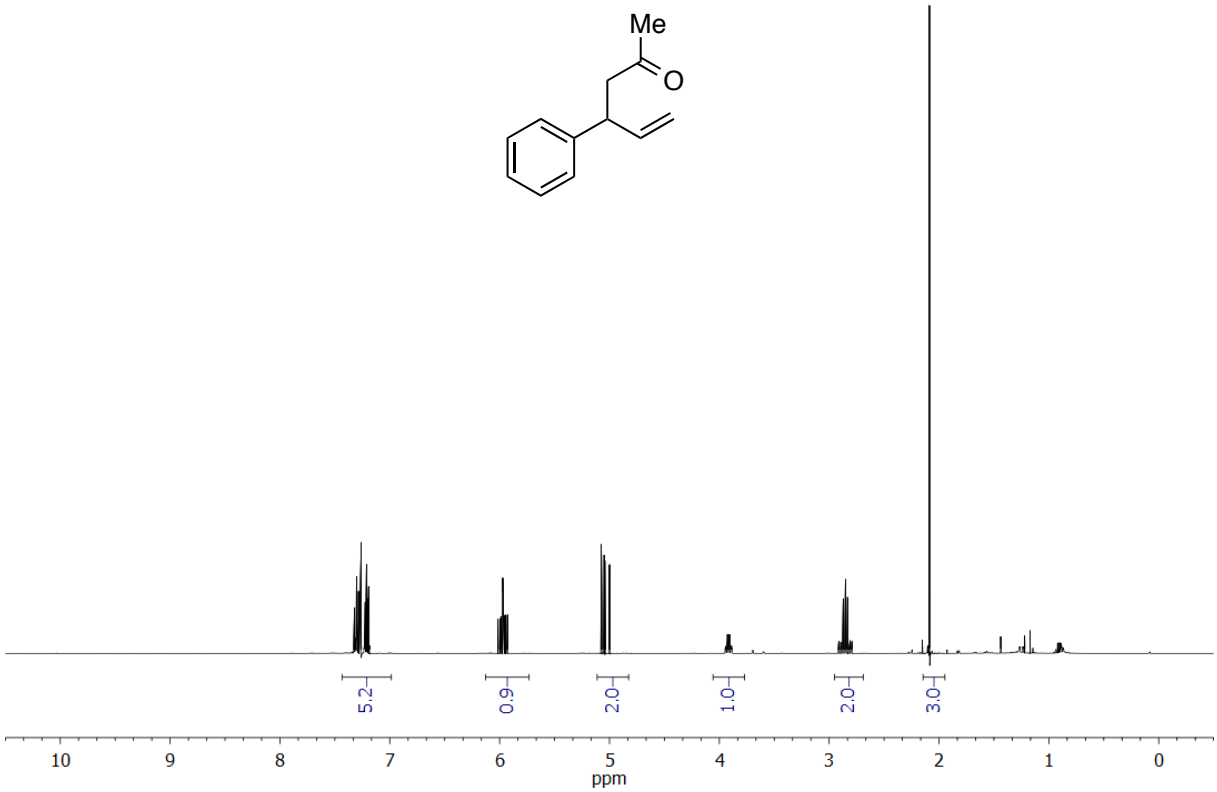


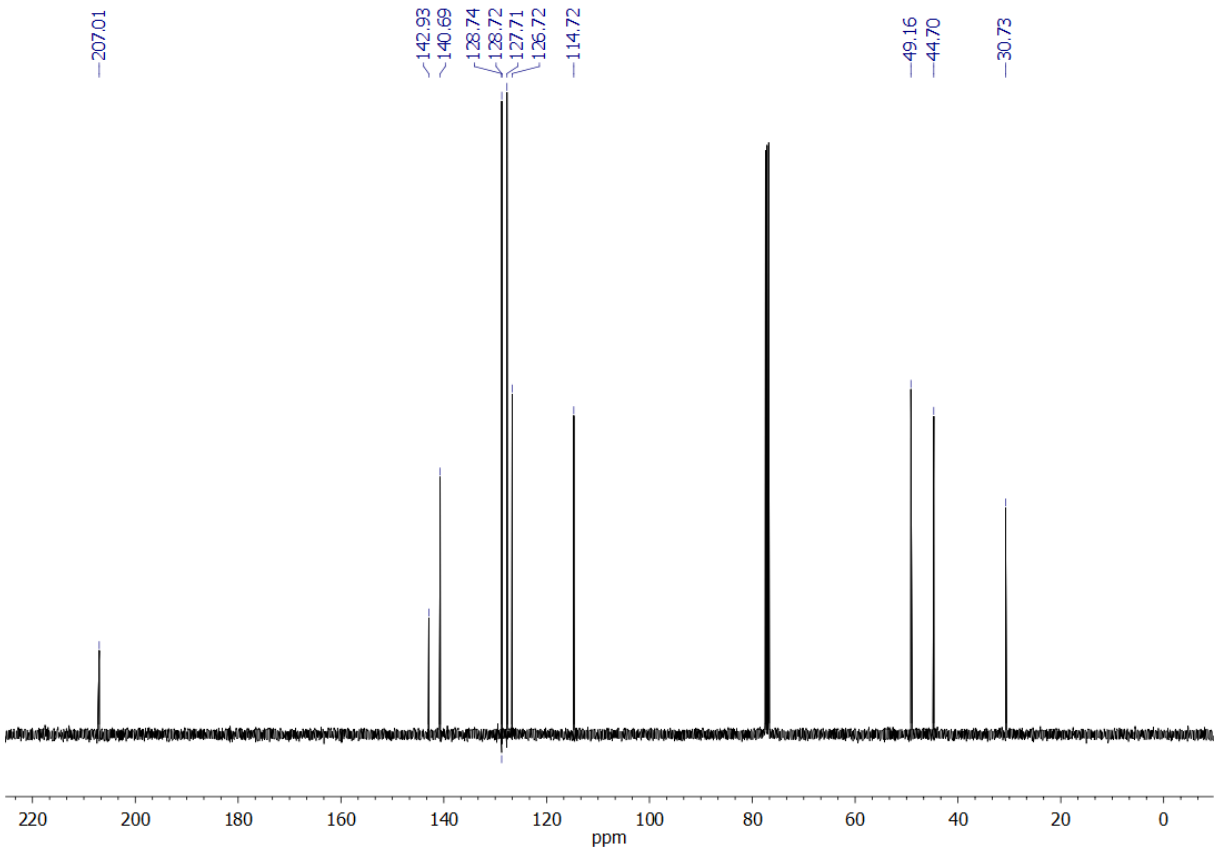
3-phenyl-1-(thiophen-2-yl)pent-4-en-1-one (3ae)



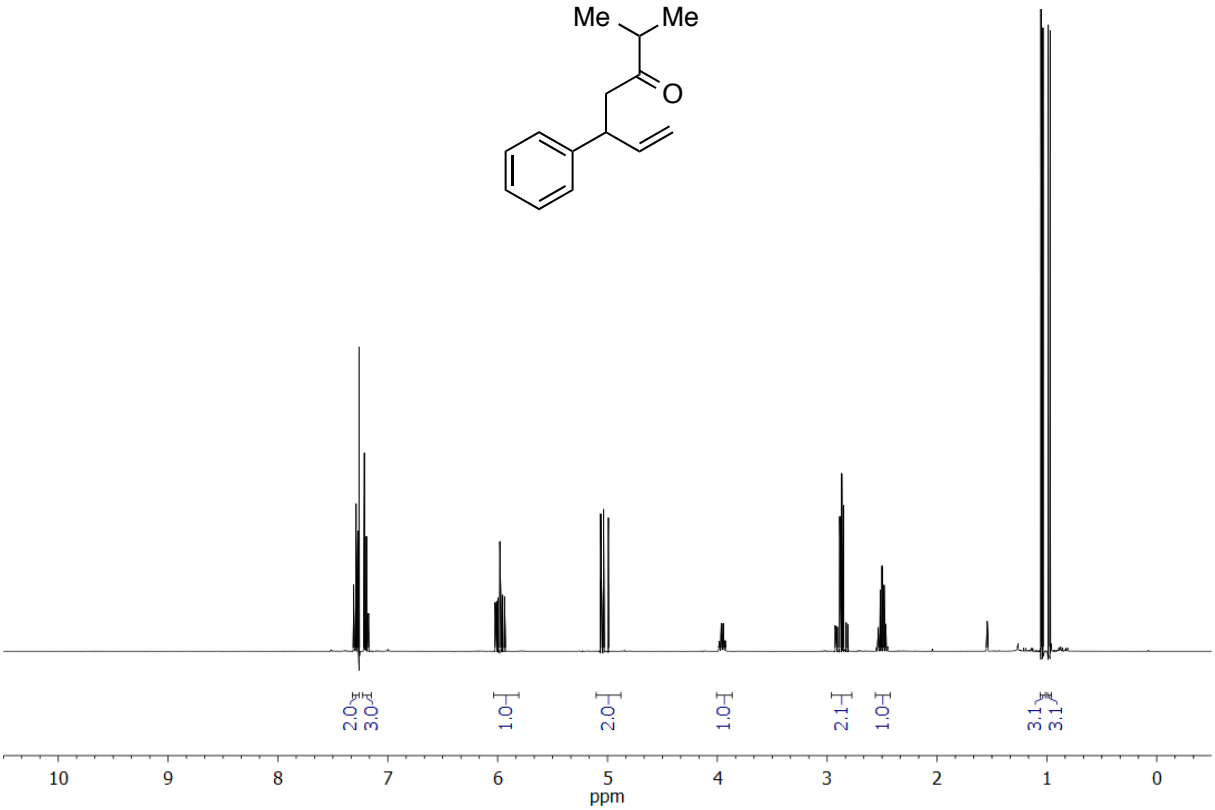


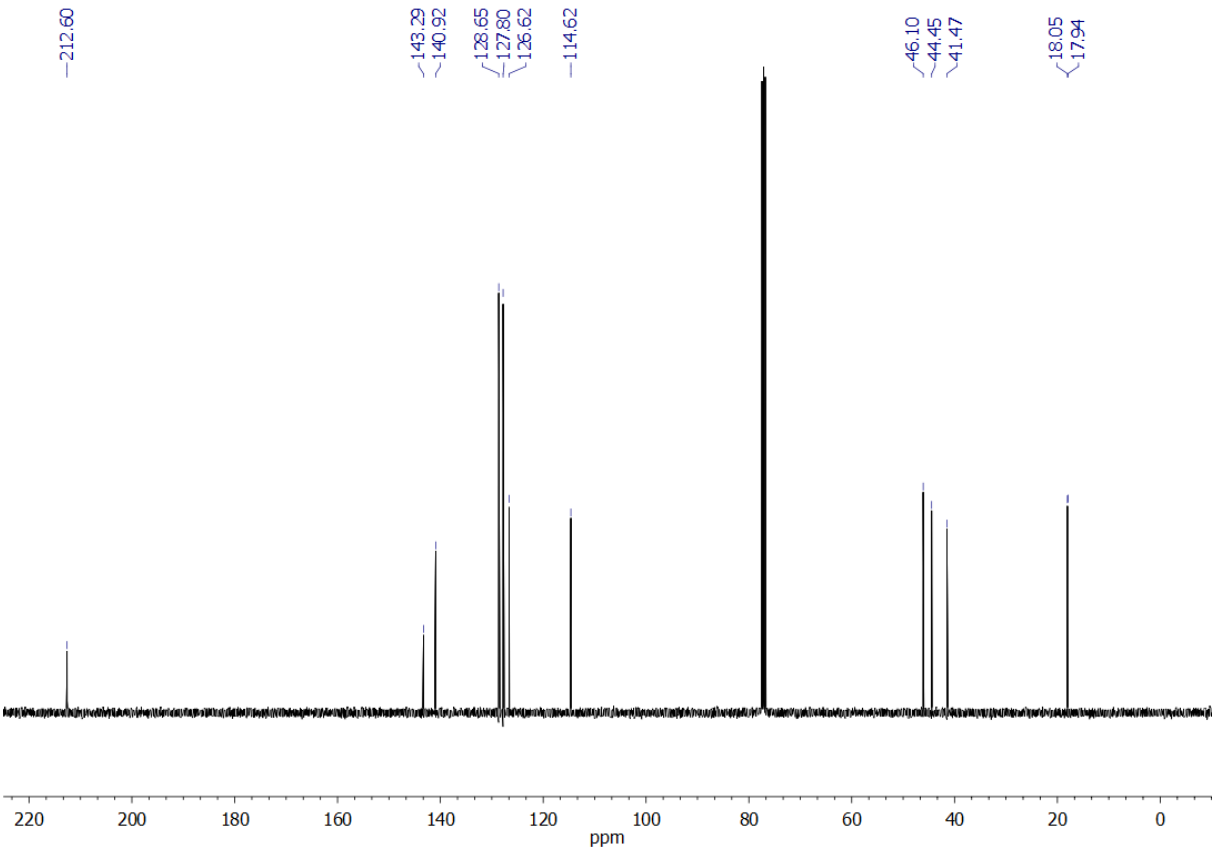
4-phenylhex-5-en-2-one (3af)



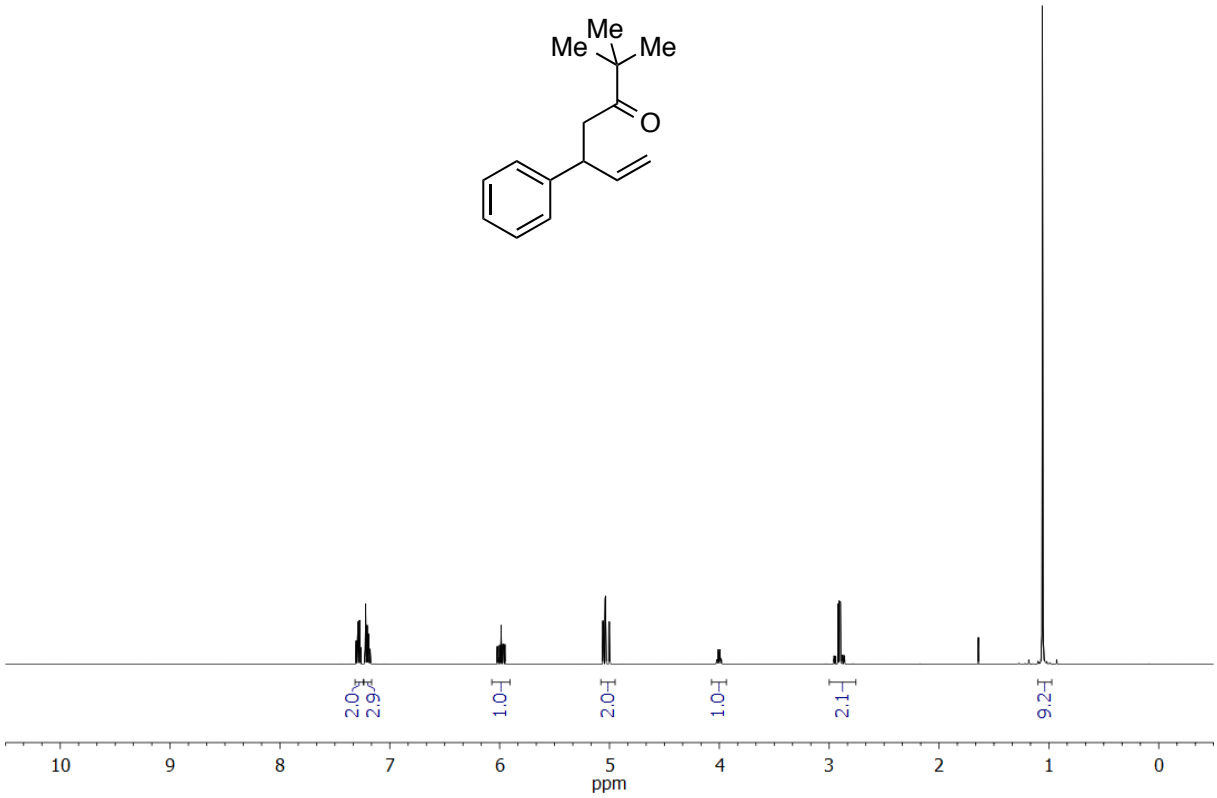


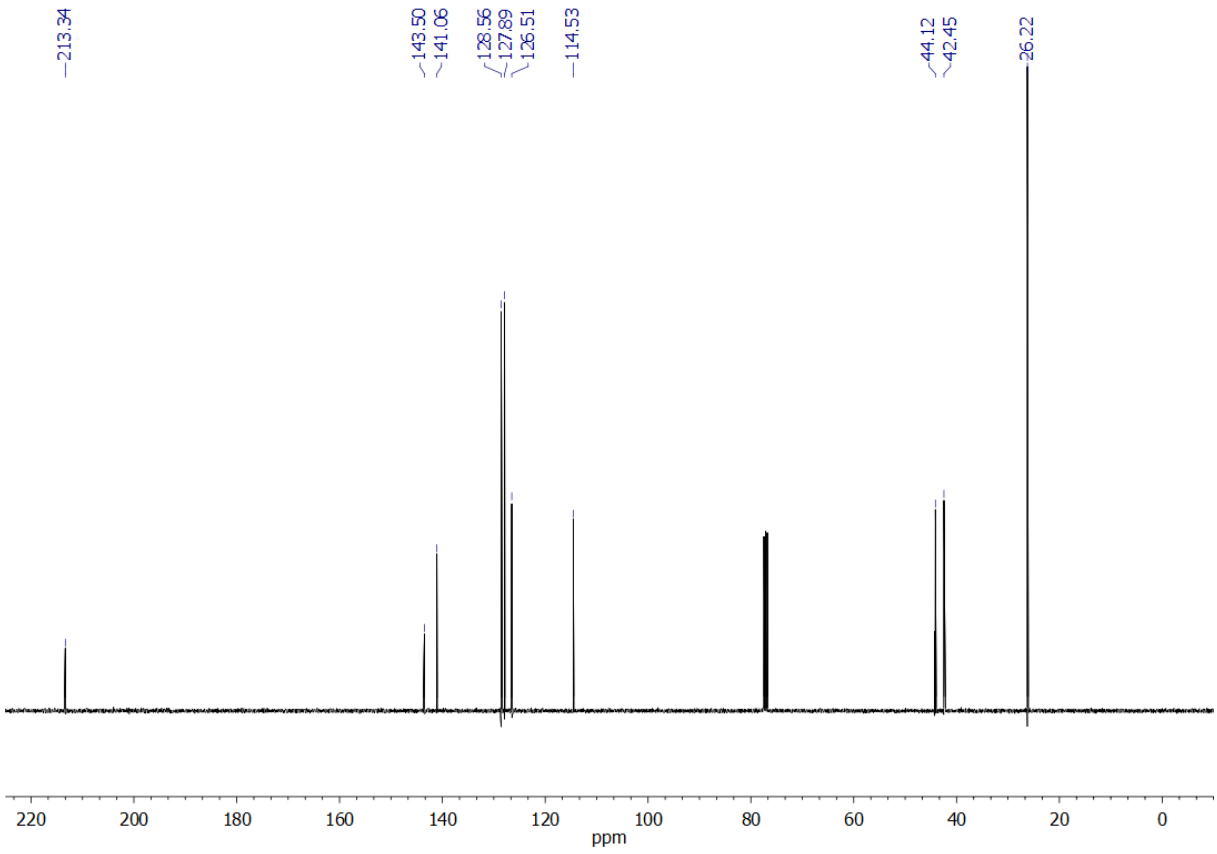
2-methyl-5-phenylhept-6-en-3-one (3ag)



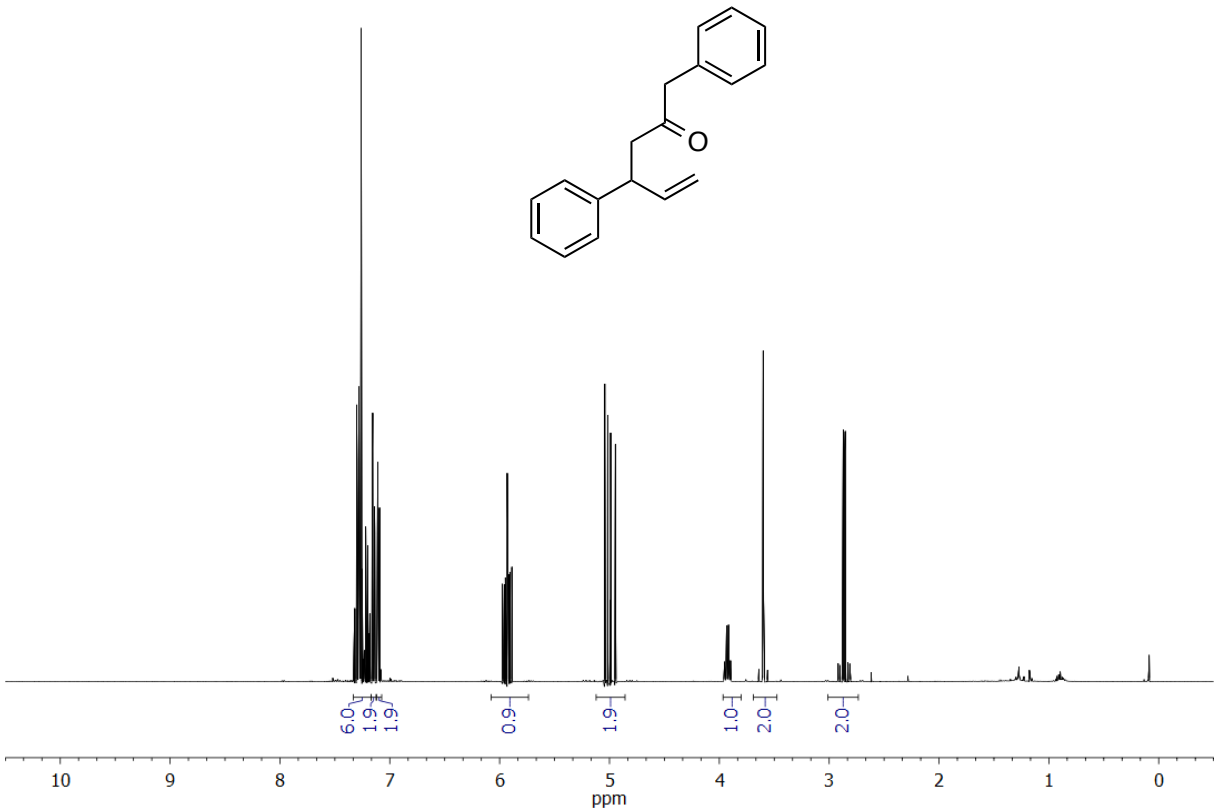


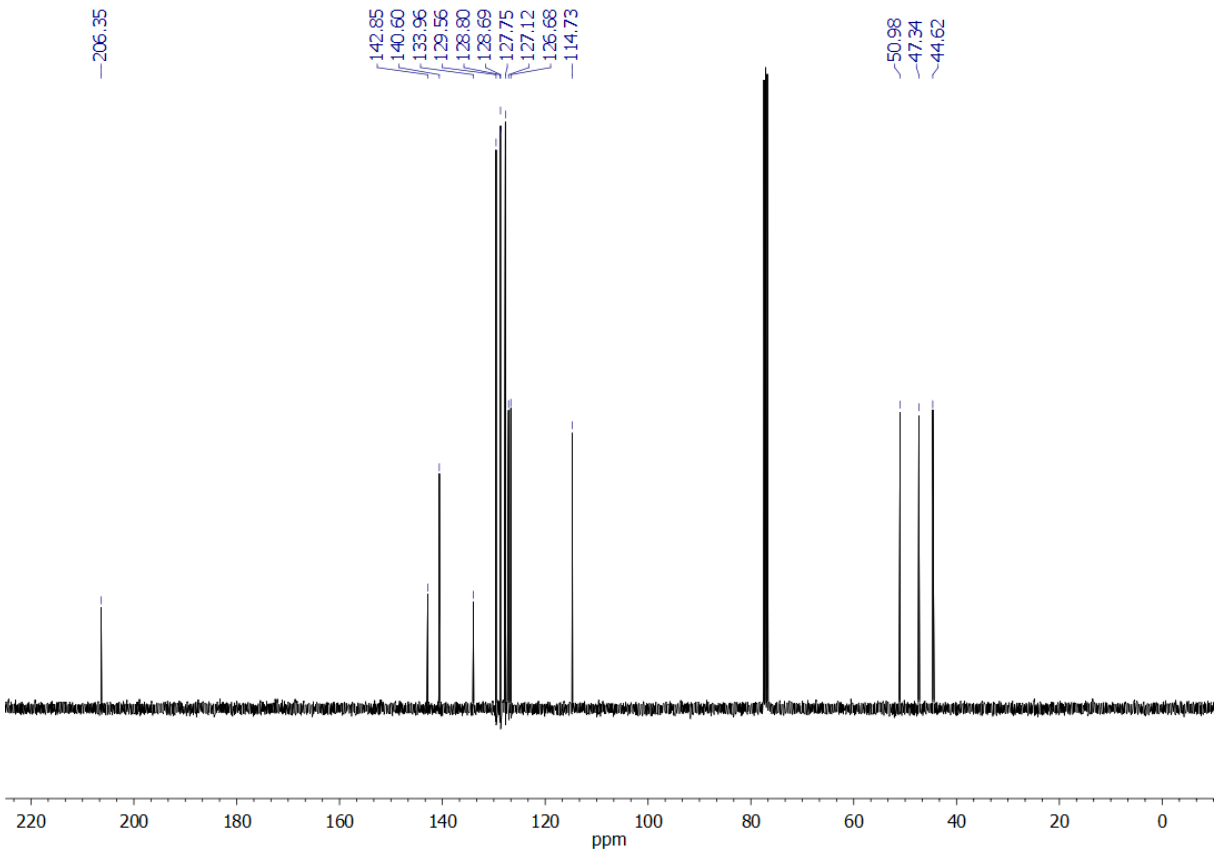
2,2-dimethyl-5-phenylhept-6-en-3-one (3ah)



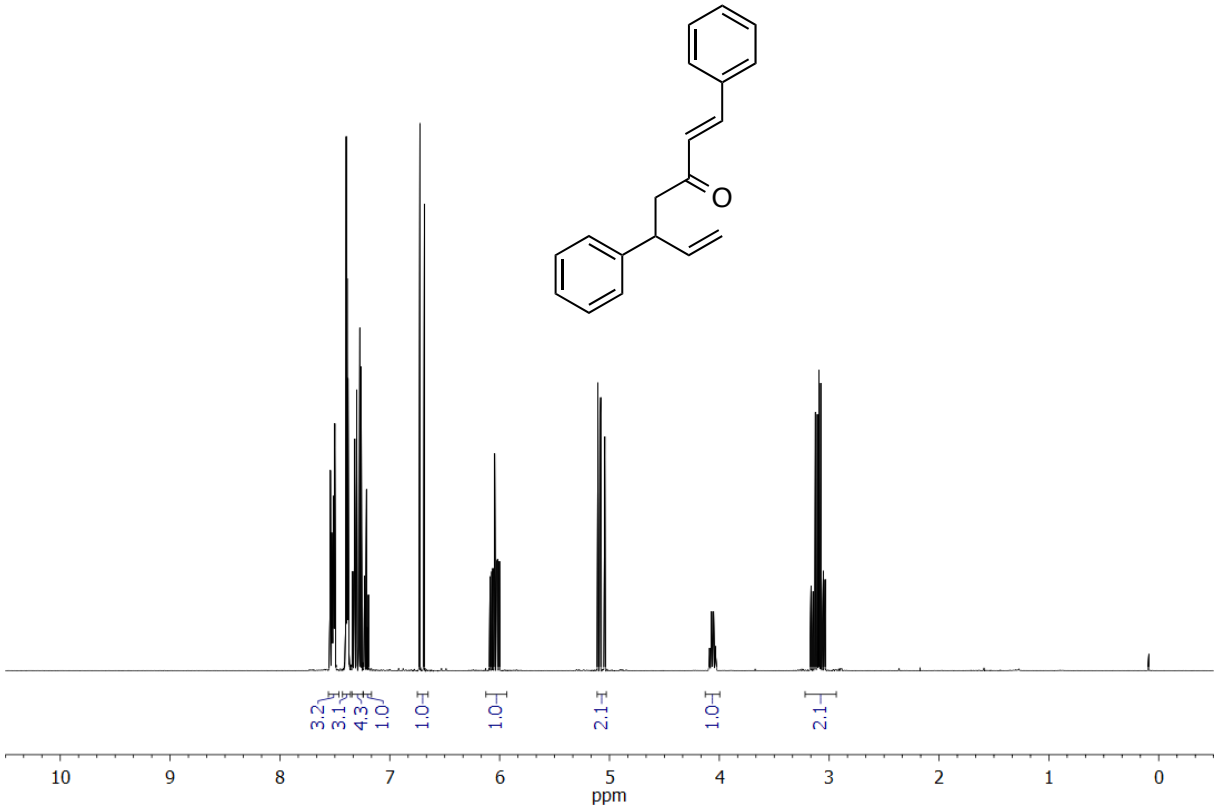


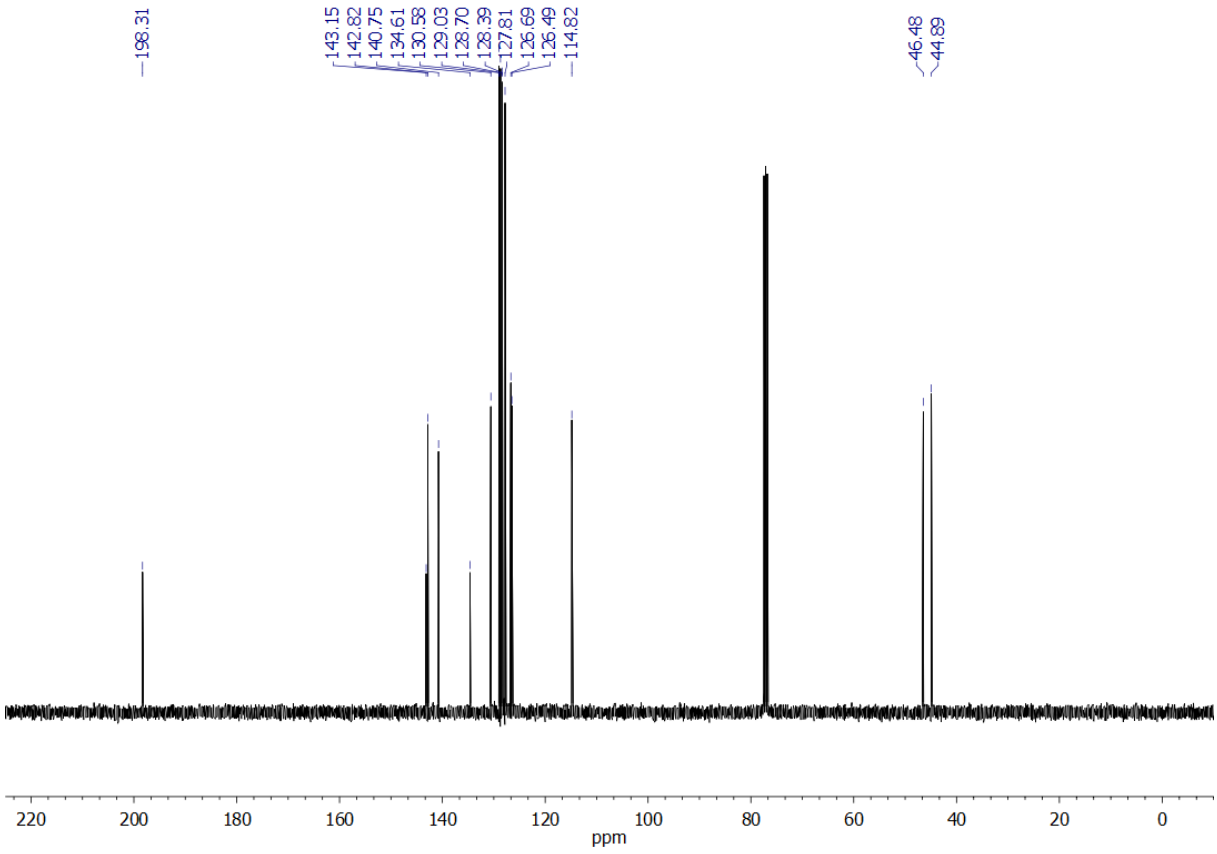
1,4-diphenylhex-5-en-2-one (3ai)





(*E*)-1,5-diphenylhepta-1,6-dien-3-one (3aj)





Chromatogram for the racemic catalysis

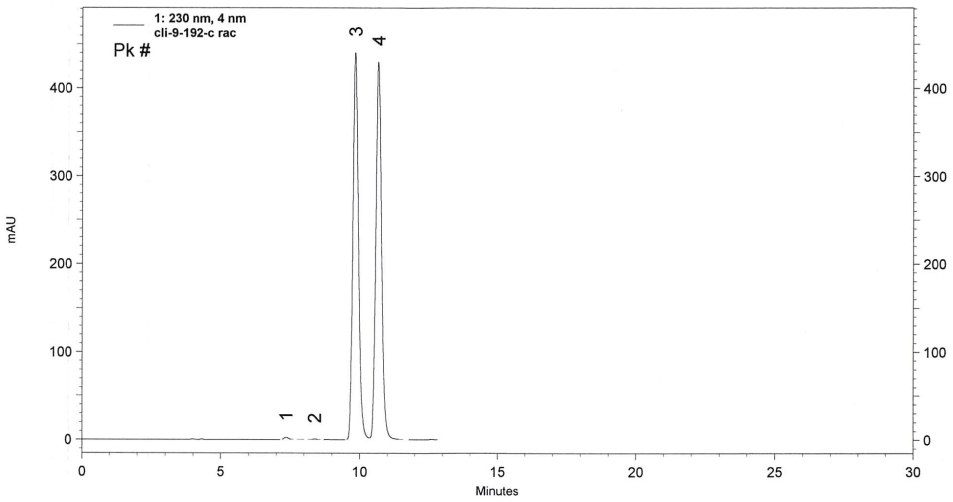
Albert-Ludwigs-Universität Freiburg

Institut für Organische Chemie und Biochemie

cli-9-192-c rac In Hoffnung das die Probe nicht so dreckig wird

E:\EZDatenOC\Li\Method\OD-3, Hept_IPA, 98_2, 0,5ml, 22°C.met

Vial: 191 Injection Volume (µl): 2
Run Time: 01.02.2016 11:22:32 Analysis Time: 01.02.2016 11:35:13



Spectrum Max Plot Results

Pk #	Retention Time	Area Percent	Lambda Max
1	3,980	0,249	202
2	4,373	1,558	201
3	7,340	0,113	201
4	9,833	48,828	205
5	10,660	49,252	205

DAD-241 nm Results

Pk #	Retention Time	Area Percent	Lambda Max
1	7,340	0,127	201
2	8,387	0,085	201
3	9,833	49,742	205
4	10,660	50,047	205

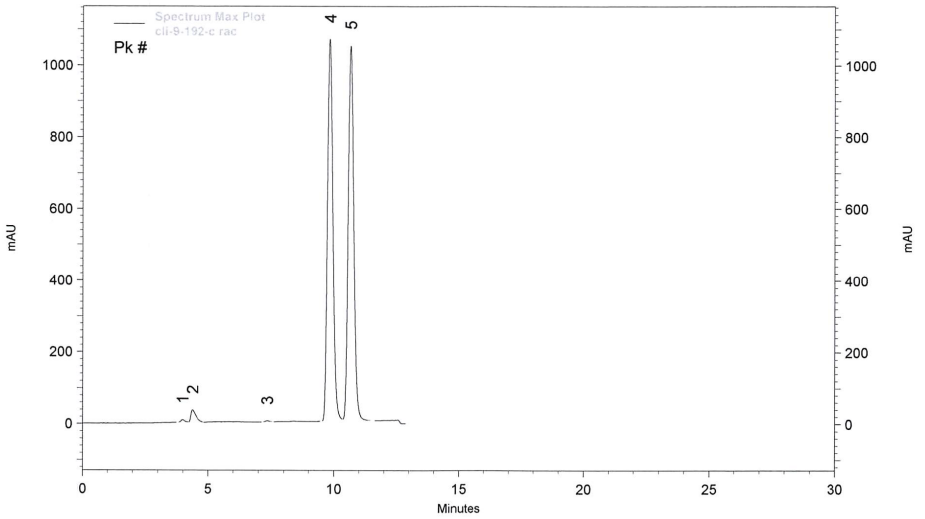
1: 230 nm, 4 nm Results

Pk #	Retention Time	Area Percent	Lambda Max
1	7,340	0,259	201

2	8,380	0,131	201
3	9,833	49,669	205
4	10,660	49,941	205

2: 210 nm, 4 nm Results

Pk #	Retention Time	Area Percent	Lambda Max
1	3,973	0,259	202
2	4,360	0,770	201
3	9,833	49,353	205
4	10,660	49,618	205



Chromatogram for *(R,R)*-DTBM-DIOP catalysis

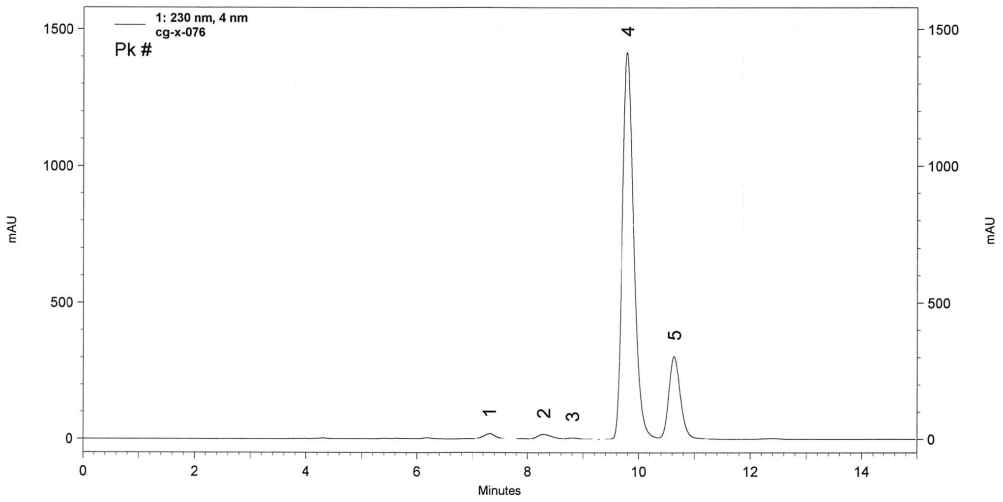
Albert-Ludwigs-Universität Freiburg

Institut für Organische Chemie und Biochemie

cg-x-076 In Hoffnung das die Probe nicht so dreckig wird

E:\EZDatenOC\Li\Method\OD-3, Hept_IPA, 98_2, 0,5ml, 22°C.met

Vial: 195 Injection Volume (µl): 2
Run Time: 01.02.2016 12:45:55 Analysis Time: 01.02.2016 12:57:09



Spectrum Max Plot Results

Pk #	Retention Time	Area Percent	Lambda Max
1	4,353	2,133	202
2	7,307	0,571	203
3	8,260	1,211	204
4	9,800	72,754	209
5	10,627	23,331	205

DAD-241 nm Results

Pk #	Retention Time	Area Percent	Lambda Max
1	7,300	0,511	203
2	8,260	1,140	204
3	9,787	80,674	209
4	10,633	17,675	205

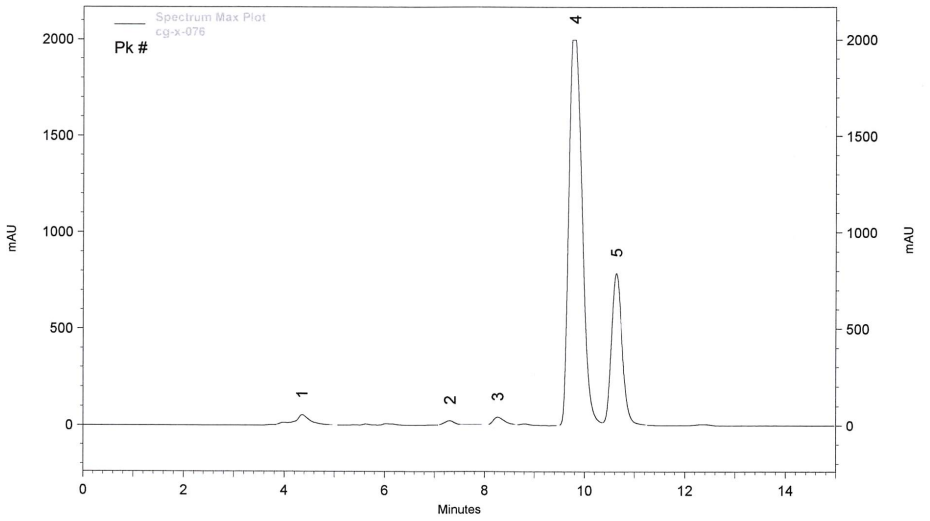
1: 230 nm, 4 nm Results

Pk #	Retention Time	Area Percent	Lambda Max
1	7,307	0,975	203

2	8,287	1,136	204
3	8,807	0,215	203
4	9,787	80,464	209
5	10,633	17,209	205

2: 210 nm, 4 nm Results

Pk #	Retention Time	Area Percent	Lambda Max
1	4,340	1,107	202
2	6,160	0,310	221
3	7,307	0,414	203
4	8,287	0,581	204
5	9,787	78,943	209
6	10,633	18,646	205



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