Meyer-Schuster Rearrangement of Propargylic Alcohols Mediated by Phosphorus-Based Brønsted Acid Catalysts

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Supporting Information

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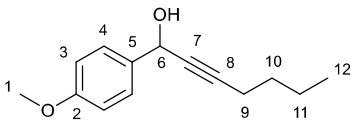
1. General Considerations

All reactions were carried out in air using technical solvents without any particular precautions to exclude moisture or oxygen, unless stated otherwise. Commercially available reagents were used as received without further purification. Column chromatography and TLC were performed on silica gel (Kieselgel 60), using UV light and a phosphomolybdic acid dip to visualize the products. Melting point ranges were determined on an Electrothermal Gallenkamp apparatus and are uncorrected. Infrared spectra were recorded in reciprocal centimetres (cm^{-1}) using a FT-IR ATR spectrometer. ^{1}H, ^{13}C and ^{31}P NMR spectra were recorded in $CDCl_3$ on Bruker AVANCE 400 spectrometers (^{1}H: 400 MHz, ^{13}C: 101MHz, ^{31}P: 162 MHz) at 23 °C. The chemical shifts, δ, are given in ppm relatively to tetramethylsilane (0.00 ppm), $CDCl_3$ (77.0 ppm), H_3PO_4 (0.00 ppm). Multiplicity is given as br, s, d, t, q and m for broad, singlet, doublet, triplet, quartet and multiplet. Assignments of some ^{1}H and ^{13}C NMR signals rely on COSY, HSQC, HMBC, and/or DEPT experiments. High resolution mass spectra were recorded on either a Micromass Autospec Premier, Micromass LCT Premier or a VG Platform II spectrometer using ESI techniques at the Mass Spectrometry Service of Imperial College London.

2. Preparation of Propargylic Alcohols

General procedure for propargylic alcohols bearing internal alkynes: *n*-Butyllithium (2.5 M solution in hexane, 1.3 equiv.) was added dropwise to a stirred solution of the chosen alkyne (1.3–1.5 equiv.) in anhydrous THF (1 M) at -78°C under N_2 atmosphere. After 30 min, the chosen aldehyde or ketone (1.0 equiv.) was added and the resulting solution was stirred for 5 min at 0 °C and 16 h at room temperature. The solution mixture was then quenched with aqueous saturated NH_4Cl and extracted with diethyl ether. The combined organic phases were washed with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was then purified by column chromatography to give the pure propargylic alcohol **1**.

1-(4-Methoxyphenyl)hept-2-yn-1-ol (1a)

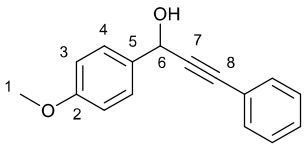


Following the general procedure from anisaldehyde (7.20 mL, 60.0 mmol) and 1-hexyne (9.00 mL, 78.0 mmol), the title compound was isolated by column chromatography (petroleum ether/EtOAc = 90:10) as a colourless oil (12.70 g, 98%). Spectroscopic data for the title compound is in accordance with the literature.[1]

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.47 (d, J = 8.5 Hz, 2H, H^{Ar}), 6.90 (d, J = 8.5 Hz, 2H, H^{Ar}), 5.40 (br d, J = 6.0 Hz, 1H, H^{6}), 3.81 (s, 3H, H^{1}), 2.33 (td, J = 7.0; 2.0 Hz, 2H, H^{9}), 2.03 (br d, J = 6.0 Hz, 1H, OH), 1.58–1.52 (m, 2H, H^{10}), 1.49–1.40 (m, 2 H, H^{11}), 0.92 (t, J = 7.0 Hz, 2 H, H^{12}); $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 101 MHz): δ 160.0 (C^{2}), 134.1 (C^{5}), 128.6 (CH^{Ar}), 114.2 (CH^{Ar}), 88.1 (C^{8}), 80.6 (C^{7}), 65.0 (C^{6}), 55.9 (C^{1}), 31.2 (C^{9}), 22.4 (C^{10}), 19.2 (C^{11}), 14.2 (C^{12}).

1-(4-Methoxyphenyl)-3-phenylprop-2-yn-1-ol (1b)

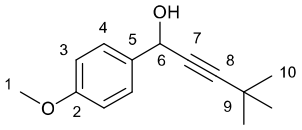
Following the general procedure from anisaldehyde (9.70 mL, 80.0 mmol) and ethynylbenzene (13.20 mL, 120.0 mmol), the title compound was isolated by trituration with Et_2O as a white



solid (11.00 g, 58%). Spectroscopic data for the title compound is in accordance with the literature.[2]

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.54 (d, J = 8.5 Hz, 2H, H^{Ar}), 7.48–7.46 (m, 2H, H^{Ar}), 7.33–7.31 (m, 3H, H^{Ar}), 6.93 (d, J = 8.5 Hz, 2H, H^{Ar}), 5.65 (d, J = 6.0 Hz, 1H, H^{6}), 3.82 (s, 3H, H^{1}), 2.20 (d, J = 6.0 Hz, 1H, OH); $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 101 MHz): δ 159.8 (C^{Ar}), 133.1 (C^{Ar}), 131.8 (C^{Ar}), 128.7 (CH^{Ar}), 128.4 (CH^{Ar}), 128.3 (CH^{Ar}), 122.6 (CH^{Ar}), 114.1 (CH^{Ar}), 89.0 (C^{7}), 86.6 (C^{8}), 64.9 (C^{6}), 55.5 (C^{1}).

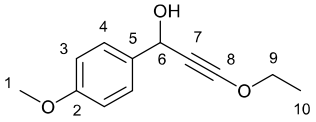
1-(4-Methoxyphenyl)-4,4-dimethylpent-2-yn-1-ol (1c)



Following the general procedure from anisaldehyde (1.20 mL, 10.0 mmol) and ethynylbenzene (1.85 mL, 15.0 mmol), the title compound was isolated by column chromatography (petroleum ether/EtOAc = 90:10) as a yellow oil (1.52 g, 53%). Spectroscopic data for the title compound is in accordance with the literature.[3]

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.42 (d, J = 8.5 Hz, 2H, H^{Ar}), 6.85 (d, J = 8.5 Hz, 2H, H^{Ar}), 5.35 (s, 1H, H^{6}), 3.75 (s, 3H, H^{1}), 1.24 (s, 9H, H^{10}); $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 101 MHz): δ 159.4 (C^{Ar}), 133.8 (C^{Ar}), 128.1 (CH^{Ar}), 113.8 (CH^{Ar}), 95.5 (C^{8}), 78.7 (C^{7}), 64.2 (C^{6}), 55.3 (C^{1}), 31.0 (C^{10}), 27.5 (C^{9}).

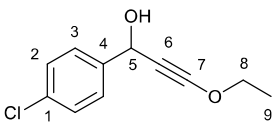
3-Ethoxy-1-(4-methoxyphenyl)prop-2-yn-1-ol (1d)



Following the general procedure from anisaldehyde (3.65 mL, 30.0 mmol) and 1-ethoxyacetylene (4.35 mL, 45.0 mmol), the title compound was isolated by column chromatography (petroleum ether/EtOAc = 80:20) as a brown oil (3.74 g, 61%).

R_f = 0.06 (petroleum ether/EtOAc = 4:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.46 (d, J = 8.5 Hz, 2H, H^{Ar}), 6.89 (d, J = 8.5 Hz, 2H, H^{Ar}), 5.46 (d, J = 6.0 Hz, 1H, H^{6}), 4.15 (q, J = 7.0 Hz, 2H, H^{9}), 3.81 (s, 3H, H^{1}), 1.96 (d, J = 6.0 Hz, 1H, OH), 1.39 (t, J = 7.0 Hz, 3H, H^{10}); $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 101 MHz): δ 159.2 (C^{Ar}), 134.4 (C^{Ar}), 127.8 (CH^{Ar}), 113.6 (CH^{Ar}), 95.1 (C^{8}), 74.7 (C^{9}), 63.9 (C^{6}), 55.2 (C^{1}), 38.9 (C^{7}), 14.3 (C^{10}). IR: ν_{max} (cm^{-1}) 3381 (br s, OH), 2952, 2931, 2872, 2258 (s, C≡C), 1606, 1505, 1461, 1386, 1226, 1170, 1028, 991, 883; HRMS (ESI) *m/z*: Calcd for $C_{12}H_{15}O_3$ $[M+H]^+$ 207.1021; Found 207.1028.

1-(4-Chlorophenyl)-3-ethoxyprop-2-yn-1-ol (1e)

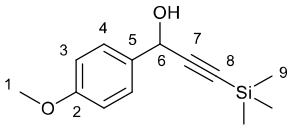


Following the general procedure from 4-chlorobenzaldehyde (0.70 g, 5.0 mmol) and 1-ethoxyacetylene (0.75 mL, 7.5 mmol), the title compound was isolated by column chromatography (petroleum ether/EtOAc = 80:20) as a yellow oil (0.13 g, 12%).

R_f = 0.08 (petroleum ether/EtOAc = 4:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.47 (d, J = 8.5 Hz, 2H, H^{Ar}), 7.33 (d, J = 8.5 Hz, 2H, H^{Ar}), 5.47 (d, J = 6.0 Hz, 1H, H^{5}), 4.14 (q, J = 7.0 Hz, 2H, H^{8}), 2.01 (d, J = 6.0 Hz, 1H, OH), 1.39 (t, J = 7.0 Hz, 3H, H^{9}); $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 101 MHz): δ 140.6 (C^{Ar}), 133.8 (C^{Ar}), 128.6 (CH^{Ar}), 128.0 (CH^{Ar}), 95.7 (C^{7}), 75.0 (C^{8}), 64.0 (C^{5}), 38.6 (C^{6}), 14.5 (C^{9}). IR: ν_{max} (cm^{-1}) 3354 (br s, OH), 2978, 2896, 2258 (s, C≡C), 1718, 1487,

1386, 1230, 1088, 991, 834, 879, 764, 462; HRMS (ESI) *m/z*: Calcd for $C_{11}H_{12}ClO_2$ $[M+H]^+$ 210.0448; Found 210.0450.

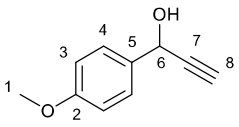
1-(4-Methoxyphenyl)-3-(trimethylsilyl)prop-2-yn-1-ol (1f)



Following the general procedure from anisaldehyde (1.20 mL, 10.0 mmol) and ethynyltrimethylsilane (2.10 mL, 15.0 mmol), the title compound was isolated by column chromatography (petroleum ether/EtOAc = 80:20) as a yellow oil (2.32 g, 99%). Spectroscopic data for the title compound is in accordance with the literature.[4]

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.46 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 6.90 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 5.40 (d, *J* = 6.0 Hz, 1H, H^6), 3.81 (s, 3H, H^1), 0.20 (s, 9H, H^9); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 159.6 (C^{Ar}), 132.8 (C^{Ar}), 128.2 (CH^{Ar}), 113.9 (CH^{Ar}), 105.4 (C^8), 91.2 (C^7), 64.5 (C^6), 55.3 (C^1), 0.00 (C^9).

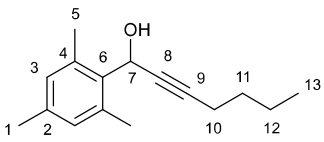
1-(4-Methoxyphenyl)prop-2-yn-1-ol (1g)



Ethynylmagnesium bromide (0.5 M solution in THF, 36 mL, 1.2 equiv.) was added dropwise to a stirred solution of anisaldehyde (1.80 mL, 15.0 mmol) in anhydrous THF (15 mL) at 0 °C under N_2 atmosphere. After 2 h, the reaction mixture was warmed to room temperature and then quenched with saturated aqueous NH_4Cl and extracted with diethyl ether. The combined organic phases were washed with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was then purified by column chromatography (petroleum ether/EtOAc = 90:10) to give title compound as a yellow oil (1.61 g, 66%). Spectroscopic data for the title compound is in accordance with the literature.[5]

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.48 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 6.91 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 5.42 (dd, *J* = 6.0; 2.0 Hz, 1H, H^6), 3.81 (s, 3H, H^1), 2.66 (d, *J* = 2.0 Hz, 1H, H^8), 2.13 (br d, *J* = 6.0 Hz, 1H, OH); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 159.8 (C^{Ar}), 132.4 (C^{Ar}), 128.0 (CH^{Ar}), 114.0 (CH^{Ar}), 83.7 (C^7), 74.5 (C^8), 64.0 (C^6), 55.3 (C^1).

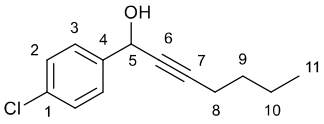
1-Mesitylhept-2-yn-1-ol (1h)



Following the general procedure from mesityl aldehyde (11.80 mL, 80.0 mmol) and 1-hexyne (14.00 mL, 120.0 mmol), the title compound was isolated by column chromatography (petroleum ether/EtOAc = 90:10) as a pale yellow oil (12.97 g, 70%). Spectroscopic data for the title compound is in accordance with the literature.[6]

^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.84 (s, 2H, H^{Ar}), 5.89–5.87 (m, 1H, H^7), 2.49 (s, 6H, H^5), 2.25 (s, 3H, H^1), 2.23–2.19 (td, *J* = 7.0; 2.0 Hz, 2H, H^{10}), 1.89 (d, *J* = 4.0 Hz, 1H, OH), 1.52–1.45 (m, 2H, H^{11}), 1.43–1.34 (m, 2H, H^{12}), 0.89 (t, 3H, *J* = 7.0 Hz, H^{13}); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 137.3 (C^{Ar}), 136.3 (C^{Ar}), 134.2 (C^{Ar}), 129.9 (CH^{Ar}), 86.3 (C^9), 79.7 (C^8), 60.4 (C^7), 30.7 (C^{10}), 22.0 (C^{11}), 20.8 (C^5), 20.2 (C^1), 18.6 (C^{12}), 13.6 (C^{13}).

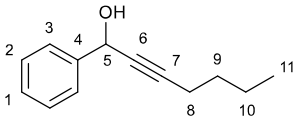
1-(4-Chlorophenyl)hept-2-yn-1-ol (1i)



Following the general procedure from 4-chlorobenzaldehyde (31.00 mL, 78.0 mmol) and 1-hexyne (10.00 mL, 90.0 mmol), the title compound was isolated by column chromatography (petroleum ether/EtOAc = 90:10) as an orange oil (7.18 g, 54%). Spectroscopic data for the title compound is in accordance with the literature.[7]

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.47 (d, J = 8.5 Hz, 2H, H^{Ar}) , 7.34 (d, J = 8.5 Hz, 2H, H^{Ar}), 5.42 (d, J = 6.0 Hz, H^5), 2.27 (td, J = 7.0; 2.0 Hz, H^8), 2.17 (br s, 1H, OH), 1.56–1.49 (m, 2H, H^9), 1.46–1.37 (m, 2H, H^{10}), 0.92 (t, 3H, J = 7 Hz, H^{11}); ^{13}C{^{1}H} NMR ($CDCl_3$, 101 MHz): δ 139.9 (C^1), 134.1 (C^4), 128.8 (C^2), 128.2 (C^3), 88.2 (C^7), 79.7 (C^6), 64.3 (C^5), 30.7 (C^8), 22.1 (C^9), 18.6 (C^{10}), 13.7 (C^{11}).

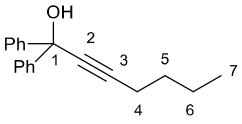
1-Phenylhept-2-yn-1-ol (1j)



Following the general procedure from benzaldehyde (4.00 mL, 40.0 mmol) and 1-hexyne (6.90 mL, 60.0 mmol), the title compound was isolated by column chromatography (petroleum ether/EtOAc = 90:10) as a yellow oil (6.50 g, 86%). Spectroscopic data for the title compound is in accordance with the literature.[7]

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.55–7.53 (m, 2H, H^{Ar}), 7.40–7.36 (m, 2H, H^{Ar}), 7.33–7.31(m, 1H, H^{Ar}), 5.45 (d, J = 6.0 Hz, 1H, H^5), 2.28 (td, J = 7.0; 2.0 Hz, 2H, H^8), 2.05 (d, J = 6.0 Hz, 1H, OH), 1.52–1.49 (m, 2H, H^9), 1.47–1.38 (m, 2H, H^{10}), 0.91 (t, J = 7.5 Hz, 3H, H^{11}); ^{13}C{^{1}H} NMR ($CDCl_3$, 101 MHz): δ 141.4 (C^4), 128.5 (CH^{Ar}), 128.2 (CH^{Ar}), 126.7 (CH^{Ar}), 87.7 (C^7), 80.1 (C^6), 64.9 (C^5), 30.7 (C^8), 22.0 (C^9), 18.6 (C^{10}), 13.6 (C^{11}).

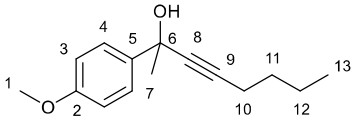
1,1-Diphenylhept-2-yn-1-ol (1k)



Following the general procedure from benzophenone (14.50 g, 80.0 mmol) and 1-hexyne (14.00 mL, 120.0 mmol), the title compound was isolated by column (petroleum ether/ EtOAc = 90:10) as a colourless oil (10.48 g, 75%). Spectroscopic data for the title compound is in accordance with the literature.[8]

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.60 (d, J = 7.5 Hz, 4H, H^{Ar}), 7.32–7.29 (m, 4H, H^{Ar}), 7.25–7.22 (m, 2H, H^{Ar}), 2.69 (br s, 1H, OH), 2.33 (t, J = 7.0 Hz, 2H, H^4), 1.61–1.51 (m, 2H, H^5), 1.49–1.40 (m, 2H, H^6), 0.92 (t, J = 7.5 Hz, 3H, H^7); ^{13}C{^{1}H} NMR ($CDCl_3$, 101 MHz): δ 145.6 (C^{Ar}), 128.2 (C^{Ar}), 127.6 (CH^{Ar}), 126.1 (CH^{Ar}), 88.4 (C^2), 83.1 (C^3), 74.6 (C^1), 30.8 (C^4), 22.2 (C^5), 18.7 (C^6), 13.7 (C^7).

2-(4-Methoxyphenyl)oct-3-yn-2-ol (1l)



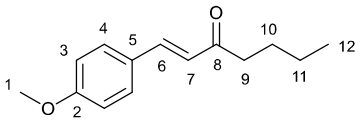
Following the general procedure from 4-methoxyacetophenone (6.00 g, 40.0 mmol) and 1-hexyne (6.90 mL, 60.0 mmol), the title compound was isolated by column chromatography (petroleum ether/EtOAc = 90:10) as a colourless oil (8.14 g, 88%). Spectroscopic data for the title compound is in accordance with the literature.[9]

R_f = 0.30 (petroleum ether/EtOAc = 4:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.57 (d, *J* = 9.0 Hz, 2H, H^{Ar}), 6.87 (d, *J* = 9.0 Hz, 2H, H^{Ar}), 3.81 (s, 3H, H^1), 2.27 (t, *J* = 7.0 Hz, 2H, H^{10}), 2.20 (br s, 1H, OH), 1.73 (s, 3H, H^7), 1.57–1.50 (m, 2H, H^{11}), 1.48–1.39 (m, 2H, H^{12}), 0.92 (t, *J* = 7.5 Hz, 3H, H^{13}); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 158.9 (C^{Ar}), 138.5 (C^{Ar}), 126.2 (CH^{Ar}), 113.4 (CH^{Ar}), 85.4 (C^9), 83.9 (C^8), 69.6 (C^6), 55.2 (C^1), 33.4 (C^7), 30.7 (C^{10}), 21.9 (C^{11}), 18.3 (C^{12}), 13.5 (C^{13}); IR: ν_{max} (cm^{-1}) 3413 (br s, OH), 2956, 2931, 2872, 2836, 2238 (C≡C), 1610, 1584, 1507, 1483, 1363, 1327, 1300, 1245, 1175, 1089, 1031, 913, 831, 779, 729; HRMS (ESI) *m/z*: Calcd for $C_{15}H_{19}O$ $[M+H]^+$ 215.1436; Found 215.1431.

3. Meyer-Schuster Rearrangement of Propargylic Alcohols

General Procedure: The chosen propargylic alcohol (1.0 mmol) was added to a solution of aq. $(OH)P(O)H_2$ (50 wt% aq. solution, 5–10 mol%) in technical toluene (1.0 mL). The reaction mixture was stirred at 90–110 °C on a heating block for 18 h, before being cooled to room temperature. The reaction mixture was quenched with a saturated aqueous solution of $NaHCO_3$ and then extracted with EtOAc. The combined organic layers were washed with brine, dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure. The obtained residue was then purified by column chromatography (reaction crude was dry loaded onto stationary phase) if necessary.

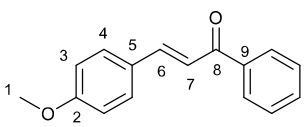
(*E*)-1-(4-Methoxyphenyl)hept-1-en-3-one (2a)



Following the general procedure at 90 °C from **1a** (0.218 g, 1.0 mmol) with aq. $(OH)P(O)H_2$ (11 μL, 10 mol%), the title compound was obtained after work-up as a yellow oil (0.207 g, 95%). Spectroscopic data for this compound is in accordance with the literature.[10]

R_f = 0.62 (petroleum ether/EtOAc, 9:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.52–7.47 (m, 3H, H^{Ar} and H^7), 6.89 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 6.62 (d, *J* = 16.0 Hz, 1H, H^6), 3.82 (s, 3H, H^1), 2.63 (t, *J* = 7.5 Hz, 2H, H^9), 1.68–1.61 (m, 2H, H^{10}), 1.41–1.33 (m, 2H, H^{11}), 0.93 (t, *J* = 7.5 Hz, 3H, H^{12}); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 200.6 (C^8), 161.5 (C^2), 142.1 (C^7), 129.9 (CH^{Ar}), 127.2 (C^5), 124.1 (C^6), 114.4 (CH^{Ar}), 55.4 (C^1), 40.6 (C^9), 26.6 (C^{10}), 22.5 (C^{11}), 13.9 (C^{12}).

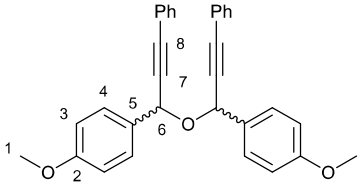
(*E*)-3-(4-Methoxyphenyl)-1-phenylprop-2-en-1-one (2b)



Following the general procedure at 90 °C from **1b** (0.238 g, 1.0 mmol) with aq. $(OH)P(O)H_2$ (11 μL, 10 mol%), the title compound was isolated by column chromatography (petroleum ether/EtOAc, 100:1→90:10 gradient) as a yellow solid (0.193 g, 81%). Spectroscopic data for this compound is in accordance with the literature.[11]

R_f = 0.51 (petroleum ether/EtOAc, 9:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.06–8.01 (m, 2H, H^{Ar}), 7.84 (d, *J* = 15.6 Hz, 1H, H^6), 7.66–7.61 (m, 2H, H^{Ar}), 7.61–7.57 (m, 1H, H^{Ar}), 7.56–7.49 (m, 2H, H^{Ar}), 7.46 (d, *J* = 15.6 Hz, 1H, H^7), 6.99–6.94 (m, 2H, H^{Ar}), 3.89 (s, 3H, H^1); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 194.0 (C^8), 160.3 (C^{Ar}), 140.2 (C^7), 137.7 (C^{Ar}), 132.9 (CH^{Ar}), 131.6 (CH^{Ar}), 128.8 (CH^{Ar}), 128.5 (CH^{Ar}), 127.8 (C^{Ar}), 123.8 (C^6), 113.6 (CH^{Ar}), 55.2 (C^1).

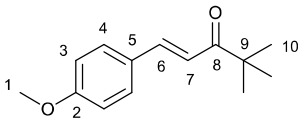
4,4'-[Oxybis(3-phenylprop-2-yne-1,1-diyl)]bis(methoxybenzene) (3b)



Following the general procedure at 60 °C from **1b** (0.119 g, 0.5 mmol) with aq. $(OH)P(O)H_2$ (5.5 μL, 10 mol%), the title compound was isolated by column chromatography (petroleum ether/EtOAc, 100:1→80:20 gradient) as a 58:42 mixture of diastereomers as a brown oil (0.145 g, 64%).

R_f = 0.13 (petroleum ether/EtOAc, 9:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.57 (d, *J* = 8.5 Hz, 4H, H^{Ar}), 7.53–7.45 (m, 12H, H^{Ar}), 7.33–7.28 (m, 12H^{maj}, H^{Ar}), 6.91 (t, *J* = 7.5 Hz, 8H^{maj}, H^{Ar}), 5.85 (s, 2H^{maj}, H^6), 5.49 (s, 2H^{min}, H^6), 3.80 (s, 6H^{min}, H^1), 3.79 (s, 6H^{maj}, H^1); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 160.0 (C^{Ar}),159.8 (C^{Ar}), 132.0 (CH^{Ar}), 131.9 (CH^{Ar}), 130.9 (C^{Ar}), 130.7 (C^{Ar}), 129.5 (CH^{Ar}), 129.3 (CH^{Ar}), 128.6 (CH^{Ar}), 128.5 (CH^{Ar}), 128.4 (CH^{Ar}), 128.3 (CH^{Ar}), 122.8 (C^{Ar}), 122.7 (C^{Ar}), 114.1 (CH^{Ar}), 113.9 (CH^{Ar}), 87.9 (C^8), 87.6 (C^8), 87.5 (C^7), 87.2 (C^7), 69.8 (C^6), 69.1 (C^6), 55.4 (C^1); IR: ν_{max} (cm^{-1}) 2996, 2929, 2832, 2221 (w, C≡C), 1606, 1505, 1438, 1300, 1244, 1170, 1028, 827, 752, 689, 570, 525; HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{32}H_{27}O_3$ 459.1952; Found 459.1960.

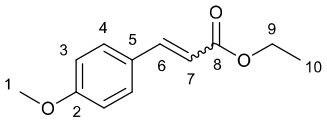
(*E*)-1-(4-Methoxyphenyl)-4,4-dimethylpent-1-en-3-one (2c)



Following the general procedure at 90 °C from **1c** (0.109 g, 0.5 mmol) with aq. $(OH)P(O)H_2$ (5.5 μL, 10 mol%), the title compound was obtained after work-up as an orange oil (0.108 g, 99%). Spectroscopic data for this compound is in accordance with the literature.[12]

R_f = 0.23 (petroleum ether/EtOAc, 9:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.65 (d, *J* = 15.5 Hz, 1H, H^7), 7.52 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 7.01 (d, *J* = 15.5 Hz, 1H, H^6), 6.90 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 3.83 (s, 3H, H^1), 1.22 (s, 9H, H^{10}); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 204.3 (C^8), 161.4 (C^2), 142.7 (C^7), 130.0 (CH^{Ar}), 127.7 (C^5), 118.5 (C^6), 114.4 (CH^{Ar}), 55.4 (C^1), 43.2 (C^9), 26.5 (C^{10}).

Ethyl 3-(4-methoxyphenyl)acrylate (2d)

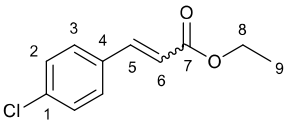


Following the general procedure at 90 °C from **1d** (0.104 g, 0.5 mmol) with aq. $(OH)P(O)H_2$ (5.5 μL, 10 mol%), the title compound was obtained after work-up as a 1:1 mixture of stereoisomers as a yellow oil (0.094 g, 91%). Spectroscopic data for this compound is in accordance with the literature.[13]

R_f = 0.26 (petroleum ether/EtOAc, 9:1); ***E*-isomer**: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.63 (d, *J* = 16.0 Hz, 1H, H^7), 7.45 (d, *J* = 8.0 Hz, 2H, H^{Ar}), 6.87 (t, *J* = 7.0 Hz, 2H, H^{Ar}), 6.30 (d, *J* = 16.0 Hz, 1H, H^6), 4.24 (q, *J* = 7.0 Hz, 2H, H^9), 3.80 or 3.79 (s, 3H, H^1), 1.32 (t, *J* = 7.0 Hz, 3H, H^{10}); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 167.3 (C^8), 161.3 (C^2), 144.2 (C^7), 129.7 (CH^{Ar}), 127.2 (C^5), 115.7 (C^6), 114.3 (CH^{Ar}), 60.3 (C^9), 55.3 (C^1), 14.2 (C^{10}); ***Z*-isomer**: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.68 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 6.87 (t, *J* = 7.0 Hz, 2H, H^{Ar}), 6.82 (d, *J* = 12.5 Hz, 1H, H^7), 5.82 (d, *J* = 12.5 Hz, 1H, H^6), 4.18 (q, *J* = 7.0 Hz, 2H, H^9), 3.80 or 3.79 (s, 3H, H^1), 1.26 (t, *J* = 7.0 Hz, 3H, H^{10}); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 166.4 (C^8), 160.4

(C^2), 143.1 (C^7), 132.2 (CH^{Ar}), 127.4 (C^5), 117.2 (C^6), 113.4 (CH^{Ar}), 60.1 (C^9), 55.2 (C^1), 14.4 (C^{10}).

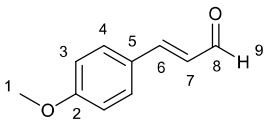
Ethyl 3-(4-chlorophenyl)acrylate (2e)



Following the general procedure at 90 °C from **1e** (0.105 g, 0.5 mmol) with aq. $(OH)P(O)H_2$ (5.5 μL, 10 mol%), the title compound was isolated by column chromatography (petroleum ether/EtOAc, 100:1→90:10 gradient) as a 68:32 mixture of stereoisomers as a yellow oil (0.054 g, 51%). Spectroscopic data for this compound is in accordance with the literature.[14]

R_f = 0.33 (petroleum ether/EtOAc, 9:1); ***E*-isomer**: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.62 (d, *J* = 16.0 Hz, 1H, H^6), 7.42 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 7.34 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 6.40 (d, *J* = 16.0 Hz, 1H, H^5), 4.26 (q, *J* = 7.0 Hz, 2H, H^8), 1.33 (t, *J* = 7.0 Hz, 3H, H^9); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 166.7 (C^7), 143.1 (C^6), 136.1 (C^1), 133.0 (C^4), 129.2 (CH^{Ar}), 129.2 (CH^{Ar}), 118.9 (C^5), 60.6 (C^8), 14.4 (C^9); ***Z*-isomer**: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.54 (d, *J* = 8.5 Hz, 1H, H^{Ar}), 7.31 (d, *J* = 8.5 Hz, 1H, H^{Ar}), 6.87 (d, *J* = 12.5 Hz, 1H, H^6), 5.96 (d, *J* = 12.5 Hz, 1H, H^5), 4.17 (q, *J* = 7.0 Hz, 2H, H^8),1.25 (t, *J* = 7.0 Hz, 3H, H^9); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 165.9 (C^7), 141.9 (C^6), 134.9 (C^1), 133.3 (C^4), 131.2 (CH^{Ar}), 128.2 (CH^{Ar}), 120.4 (C^5), 60.4 (C^8), 14.2 (C^9).

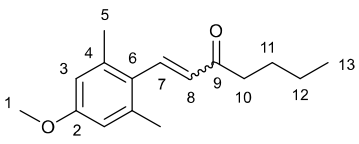
(*E*)-3-(4-Methoxyphenyl)acrylaldehyde (2g)



Following the general procedure at 90 °C from **1g** (0.162 g, 1.0 mmol) with aq. $(OH)P(O)H_2$ (5.5 μL, 5 mol%), the title compound was isolated by column chromatography (petroleum ether/EtOAc, 90:10→80:20 gradient) as a yellow solid (0.055 g, 34%). Spectroscopic data for this compound is in accordance with the literature.[15]

Mp = 58–60 °C; R_f = 0.06 (petroleum ether/EtOAc, 9:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 9.65 (d, *J* = 7.5 Hz, 1H, H^9), 7.52 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 7.42 (d, *J* = 16.0 Hz, 1H, H^7), 6.94 (d, *J* = 8.5 Hz, 1H, H^{Ar}), 6.61 (dd, *J* = 16.0; 7.5 Hz, 1H, H^6), 3.86 (s, 3H, H^1); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 193.6 (C^8), 162.2 (C^2), 152.6 (C^7), 130.3 (CH^{Ar}), 126.8 (C^5), 126.5 (C^6), 114.6 (CH^{Ar}), 55.4 (C^1).

1-Mesitylhept-1-en-3-one (2h)

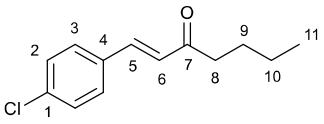


Following the general procedure at 90 °C from **1h** (0.230 g, 1.0 mmol) with aq. $(OH)P(O)H_2$ (11 μL, 10 mol%), the title compound was obtained after work-up as a 53:47 mixture of stereoisomers as a yellow oil (0.214 g, 93%).

R_f = 0.70 (petroleum ether/EtOAc, 4:1); ^{1}H NMR ($CDCl_3$, 400 MHz): (*E*-isomer) δ 7.70 (d, *J* = 16.5 Hz, 1H, H^7), 6.89 (s, 2H, H^3), 6.35 (d, *J* = 16.5 Hz, 1H, H^8), 2.65 (t, *J* = 7.5 Hz, 1H, H^{10}), 2.32 (s, 6H, H^5) 2.28 (s, 3H, H^1) 1.71–1.63 (m, 2H, H^{11}) 1.44–1.34 (m, 2H, H^{12}) , 0.94 (t, *J* = 7.5 Hz, H^{13}); (*Z*-isomer) δ 6.93 (d, *J* = 12.5 Hz, 1H, H^7), 6.86 (s, 2H, H^3), 6.29 (d, *J* = 12.5 Hz, 1H, H^8), 2.17 (t, *J* = 7.5 Hz, 2H, H^{10}), 2.27 (s, 3H, H^1), 2.15 (s, 6H, H^5), 1.44–1.34 (m, 2H, H^{11}), 1.17–1.08 (m, 2H, H^{12}), 0.76 (t, *J* = 7.5 Hz, 3H, H^{13}); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz):

(*E*- & *Z*-isomers) δ 202.4 (C^9), 200.8 (C^9), 140.9 (C^7), 140.6 (C^7), 138.4 (C^2), 137.2 (C^2), 136.9 (C^4), 134.7 (C^4), 133.0 (C^6), 131.5 (C^8), 131.3 (C^8), 131.2 (C^6), 129.3 (C^3), 128.3 (C^3), 41.9 (C^{10}), 40.8 (C^{10}), 26.7 (C^{11}), 25.9 (C^{11}), 22.5 (C^{12}), 22.3 (C^{12}), 21.2 (C^5), 21.2 (C^1), 21.1 (C^1), 20.3 (C^5), 14.0 (C^{13}), 13.8 (C^{13}); IR: ν_{max} (cm^{-1}) 2952, 2926, 2866, 1666 (w, C=C), 1606, 1457, 1375, 1177, 1058, 984, 849, 745; HRMS (ESI) m/z: Calcd for $C_{16}H_{23}O$ $[M+H]^+$ 231.1743; Found 231.1739.

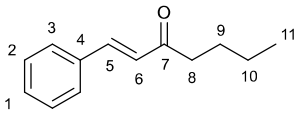
(*E*)-1-(4-Chlorophenyl)hept-1-en-3-one (2i)



Following the general procedure at 110 °C from **1i** (0.111 g, 0.5 mmol) with aq. $(OH)P(O)H_2$ (5.5 μL, 10 mol%), the title compound was obtained after work-up as a yellow oil (0.064 g, 58%). Spectroscopic data for this compound is in accordance with the literature.[10]

R_f = 0.33 (petroleum ether/EtOAc, 9:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.50–7.47 (m, 3H, H^{Ar} and H^6), 7.36 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 6.71 (d, *J* = 16.0 Hz, 1H, H^5), 2.65 (t, *J* = 7.5 Hz, 1H, H^8), 1.70–1.62 (m, 2H, H^9), 1.42–1.33 (m, 2H, H^{10}), 0.94 (t, *J* = 7.5 Hz, 3H, H^{11}); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 200.3 (C^7), 140.7 (C^6), 136.2 (C^1), 133.1 (C^4), 129.4 (CH^{Ar}), 129.2 (CH^{Ar}), 126.6 (C^5), 40.9 (C^8), 26.4 (C^9), 22.5 (C^{10}), 13.9 (C^{11}).

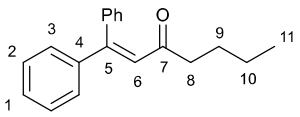
(*E*)-1-Phenylhept-1-en-3-one (2j)



Following the general procedure at 110 °C from **1j** (0.094 g, 0.5 mmol) with aq. $(OH)P(O)H_2$ (2.3 μL, 5 mol%), the title compound was isolated by column chromatography (petroleum ether/EtOAc, 100:1→90:10 gradient) as a yellow oil (0.065 g, 69%). Spectroscopic data for this compound is in accordance with the literature.[10]

R_f = 0.33 (petroleum ether/EtOAc, 9:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.57–7.52 (m, 3H, H^6 and H^{Ar}), 7.38–7.37 (m, 3H, H^{Ar}), 6.73 (d, *J* = 12.0 Hz, 1H, H^5), 2.65 (t, *J* = 7.5 Hz, 2H, H^8), 1.70–1.62 (m, 2H, H^9), 1.42–1.33 (m, 2H, H^{10}), 0.95 (t, *J* = 7.5 Hz, 3H, H^{11}); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 200.6 (C^7), 142.3 (C^6), 134.6 (C^4), 130.4 (C^1), 128.9 (CH^{Ar}), 128.2 (CH^{Ar}), 126.3 (C^5), 40.7 (C^8), 26.5 (C^9), 22.5 (C^{10}), 13.9 (C^{11}).

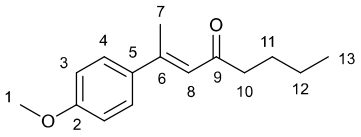
1,1-Diphenylhept-1-en-3-one (2k)



Following the general procedure from **1k** (0.26 g, 1.0 mmol) with aq. $(OH)P(O)H_2$ (11 μL, 10 mol%) the title compound was isolated by column chromatography (petroleum ether/EtOAc, 85:15) as a light yellow oil (0.22 g, 85%). Spectroscopic data for this compound is in accordance with the literature.[8]

R_f = 0.71 (petroleum ether/EtOAc, 9:1); ^{1}H NMR ($CDCl_3$, 400 MHz): d 7.39–7.27 (m, 8H, H^{Ar}), 7.20–7.18 (m, 2H, H^{Ar}), 6.57 (s, H, H^6), 2.22 (t, *J* = 7.5 Hz, 2H, H^8), 1.51–1.43 (m, 2H, H^9), 1.22–1.13 (m, 2H, H^{10}), 0.80 (t, *J* = 7.5 Hz, 3H, H^{11}); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 202.6 (C^7), 153.1 (C^5), 141.2 (C^{Ar}), 139.2 (C^{Ar}), 129.6 (CH^{Ar}), 129.4 (CH^{Ar}), 128.6 (CH^{Ar}), 128.5 (CH^{Ar}), 128.4 (CH^{Ar}), 126.8 (C^6), 43.0 (C^8), 26.5 (C^9), 22.3 (C^{10}), 13.8 (C^{11}).

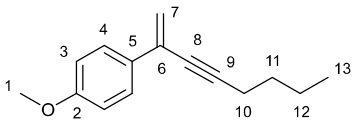
(*E*)-2-(4-Methoxyphenyl)oct-2-en-4-one (2l)



Following the general procedure from **1l** (0.52 g, 2.0 mmol) and 4-cyanoaniline (0.35 g, 3.0 mmol) with $(HO)P(OEt)_2$ (26.00 µL, 10 mol%) at 110 °C, the title compound was isolated as a light brown solid by column chromatography (petroleum ether/EtOAc, 100:0→95:5) (0.30 g, 65%). Spectroscopic data for this compound is in accordance with the literature.[16]

R_f = 0.56 (petroleum ether/EtOAc, 4:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.46 (d, *J* = 9.0 Hz, 2H, H^{Ar}), 6.90 (d, *J* = 9.0 Hz, 2H, H^{Ar}), 6.48 (q, *J* = 1.0 Hz, 1H, H^8), 3.83 (s, 3H, H^1), 2.54–2.50 (m, 5H, H^7 and H^{10}), 1.66–1.59 (m, 2H, H^{11}), 1.40–1.31 (m, 2H, H^{12}), 0.92 (t, *J* = 7.5 Hz, 3H, H^{13}); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 201.7 (C^9), 160.6 (C^2), 153.1 (C^6), 134.9 (C^5), 127.9 (C^3), 122.8 (C^8), 114.0 (C^4), 55.5 (C^1), 44.8 (C^{10}), 26.7 (C^{11}), 22.6 (C^{12}), 18.2 (C^7), 14.0 (C^{13}).

1-Methoxy-4-(oct-1-en-3-yn-2-yl)benzene (6l)



Following the general procedure at 50 °C from **1l** (0.116 g, 0.5 mmol) with aq. $(OH)P(O)H_2$ (5.5 µL, 10 mol%), the title compound was isolated as a yellow oil by column chromatography (petroleum ether/EtOAc, 99:1→90:10 gradient) followed by preparative thin layer chromatography (petroleum ether/EtOAc, 95:5) (0.015 g, 7%).

R_f = 0.56 (petroleum ether/EtOAc, 9:1); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.59 (d, *J* = 9.0 Hz, 2H, H^{Ar}), 6.86 (d, *J* = 9.0 Hz, 2H, H^{Ar}), 5.73 (d, *J* = 1.0 Hz, 1H, H^7), 5.47 (s, 1H, H^7), 3.81 (s, 3H, H^1), 2.41 (t, *J* = 7.0 Hz, 2H, H^{10}), 1.63–1.56 (m, 2H, H^{11}), 1.52–1.44 (m, 2H, H^{12}), 0.94 (t, *J* = 7.5 Hz, 3H, H^{13}); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 159.7 (C^2), 130.6 (C^6), 130.4 (C^5), 127.4 (CH^{Ar}), 117.5 (C^7), 113.7 (CH^{Ar}), 91.8 (C^9), 80.0 (C^8), 55.4 (C^1), 31.0 (C^{10}), 22.2 (C^{11}), 19.2 (C^{12}), 13.7 (C^{13}); IR: ν_{max} (cm^{-1}) 2952, 2929, 2866, 2236 (C≡C), 1714, 1681, 1636 (C=C), 1595, 1505, 1461, 1244, 1174, 1028, 831, 725; HRMS (ESI) m/z: $[M-H]^-$ Calcd for $C_{15}H_{17}O$ 213.1279; Found 213.1284.

4. ^{31}P NMR Analysis Before and After Catalysis

The ^{31}P and ^{31}P{^{1}H} NMR spectra of commercially available (OH)P(O)H$_2$ (50 wt% aqueous solution) were recorded in $CDCl_3$ and toluene-d8 (Figure S1).

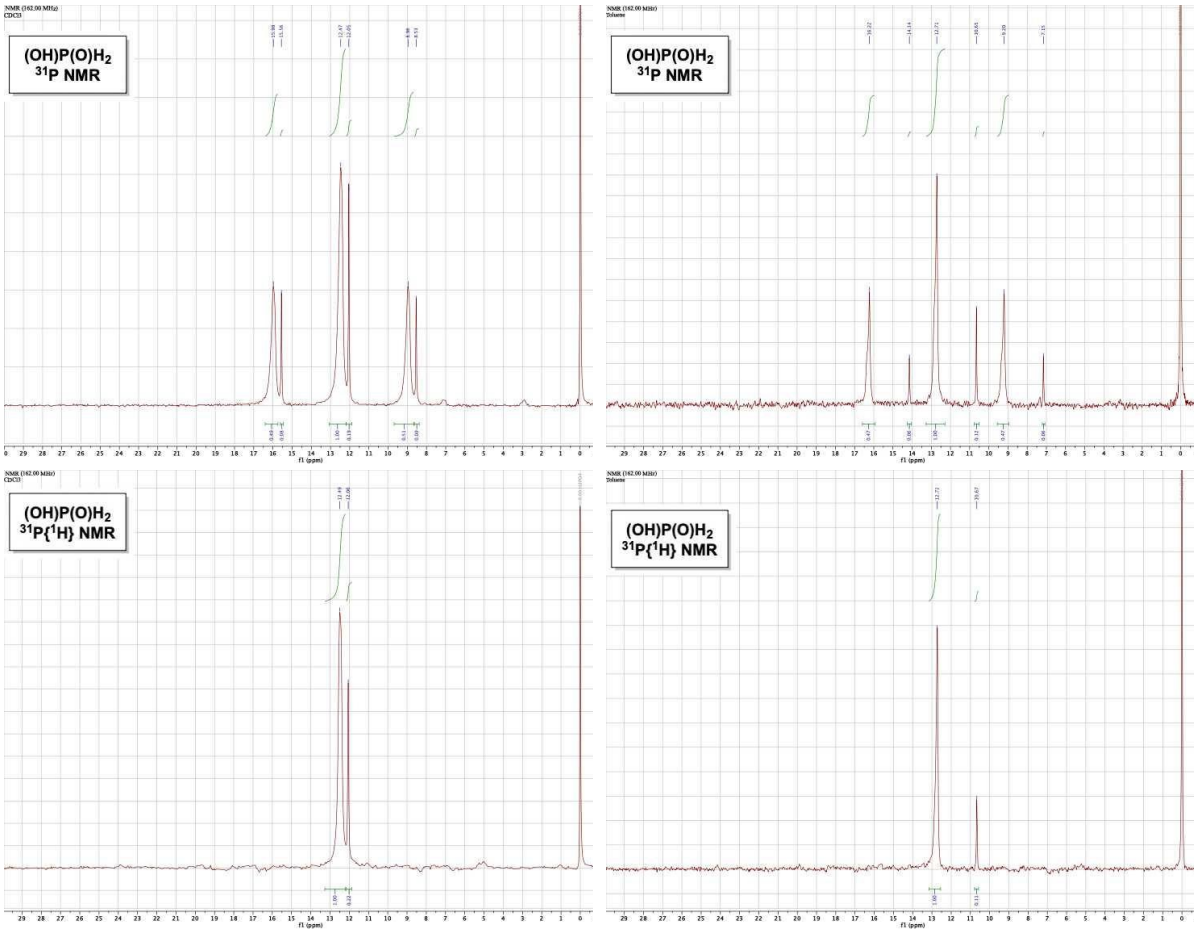


Figure S1. NMR spectra for (OH)P(O)H$_2$ in $CDCl_3$ (left) and toluene-d8 (right).

Propargylic alcohol **1a** (0.218 g, 1.0 mmol) was added to a solution of aq. (OH)P(O)H$_2$ (50 wt% aq. solution, 11 µL, 10 mol%) in technical toluene (1.0 mL). The reaction mixture was stirred at 90–110 °C on a heating block for 18 h, before being cooled to room temperature. The reaction mixture was concentrated under reduced pressure without any work-up (the water bath of the rotatory evaporator was kept at room temperature) and a sample of the obtained residue was dissolved in $CDCl_3$ (Figure S2).

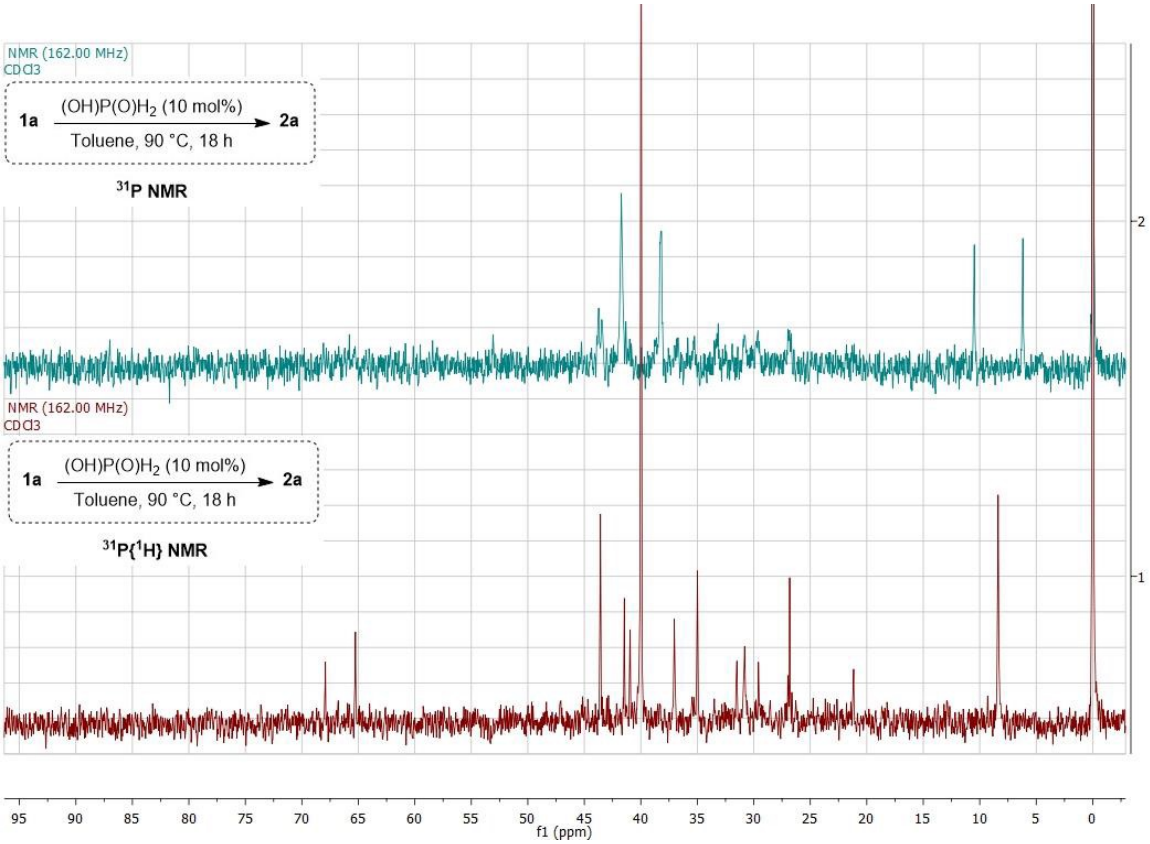


Figure S2. ^{31}P NMR analysis of a crude mixture

5. References

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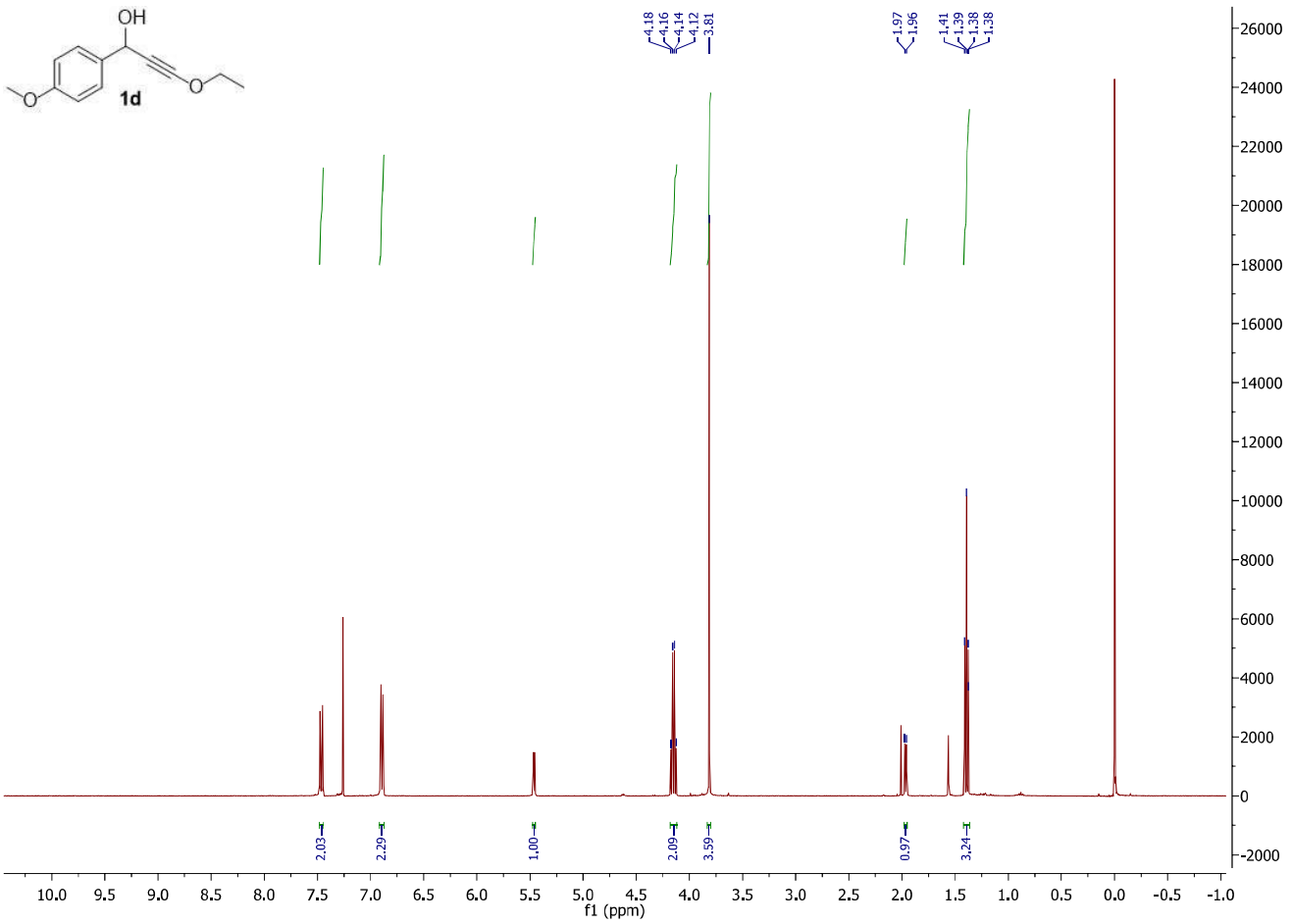
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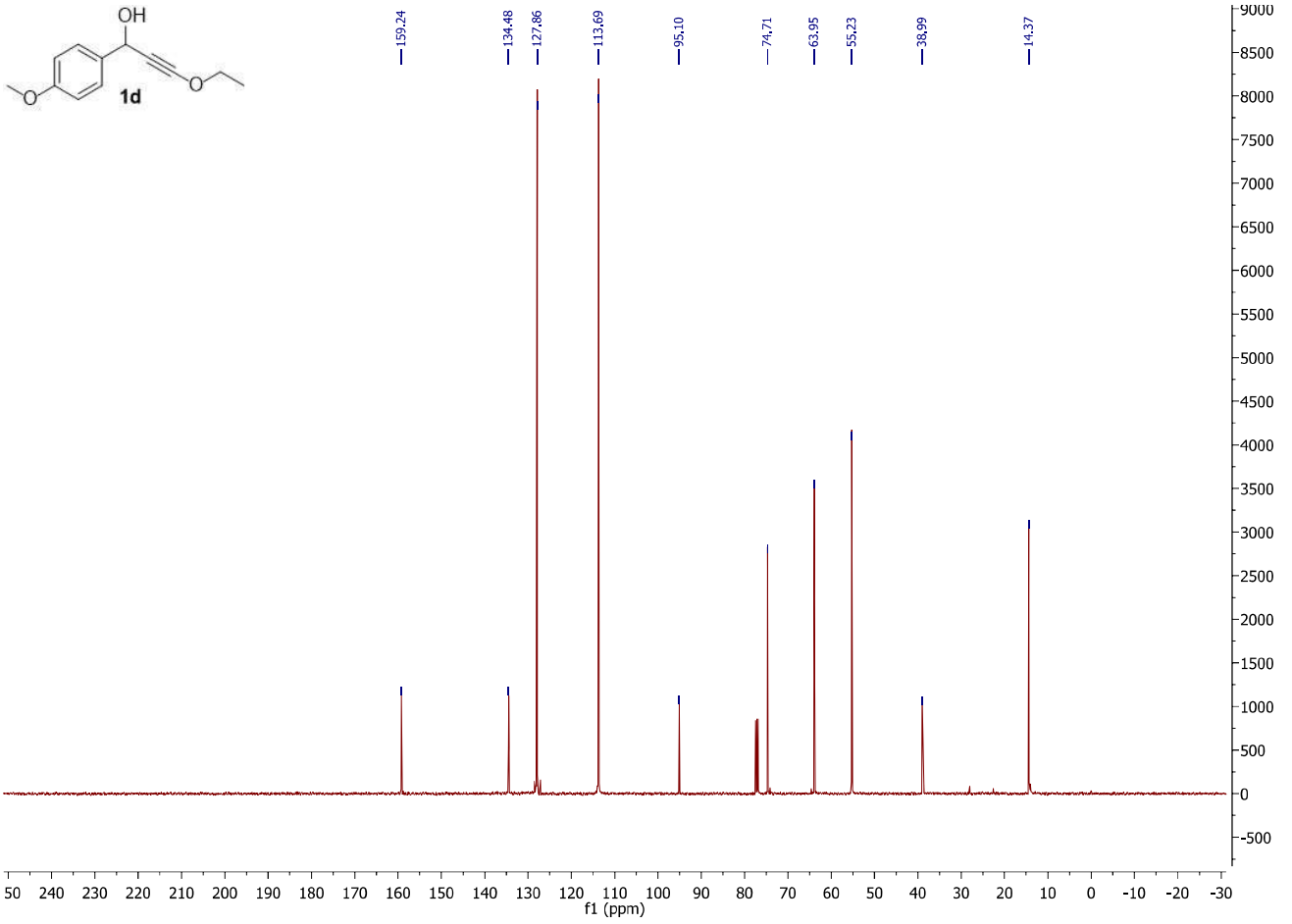
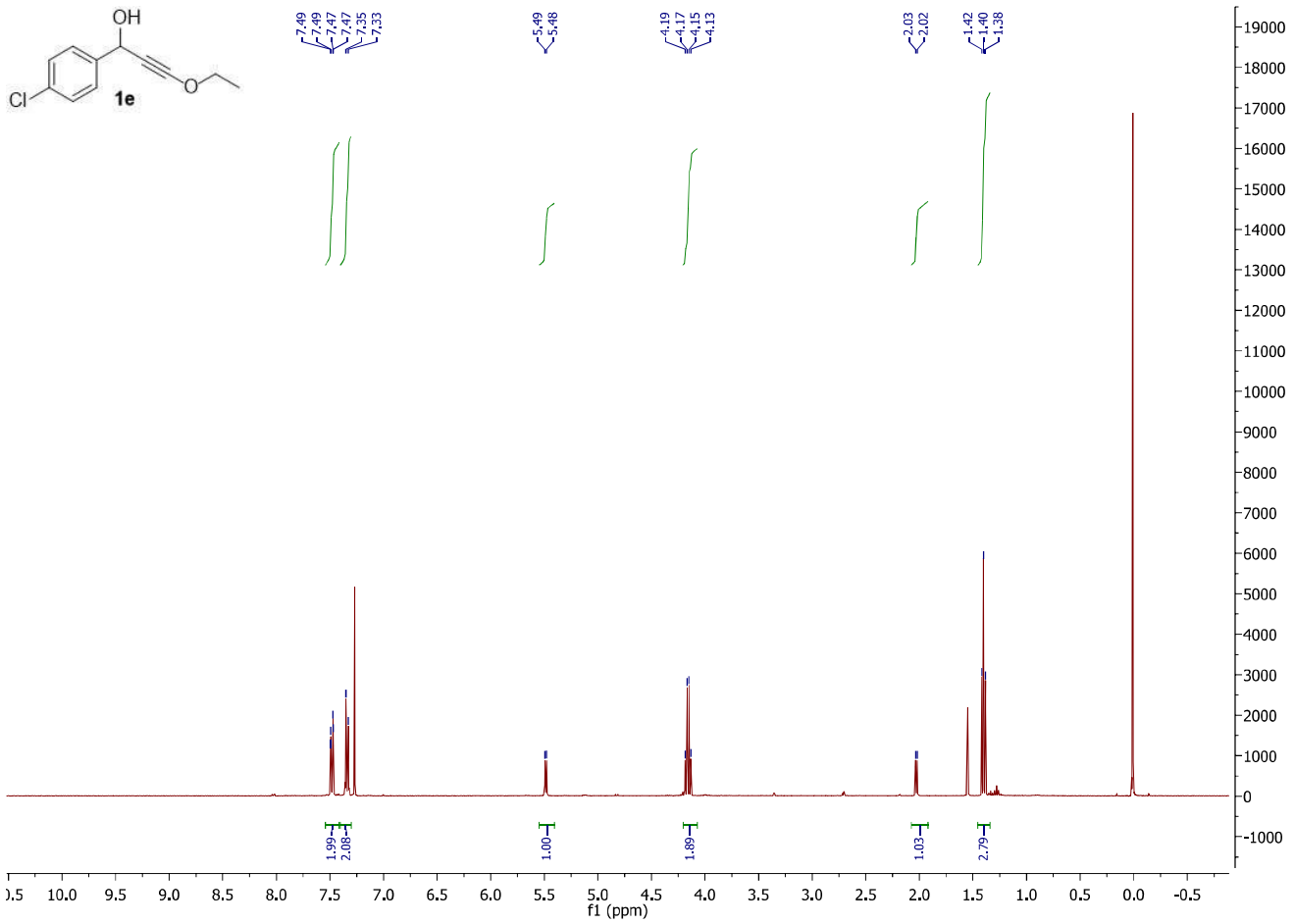
O
O
1d
4.18
4.16
4.14
4.12
3.81
1.97
1.96
1.41
1.39
1.38
1.38
2.03
2.29
1.00
2.09
3.59
0.97
3.24
f1 (ppm)


OH
O
O
1d
159.24
134.48
127.86
113.69
95.10
74.71
63.95
55.23
38.99
14.37
f1 (ppm)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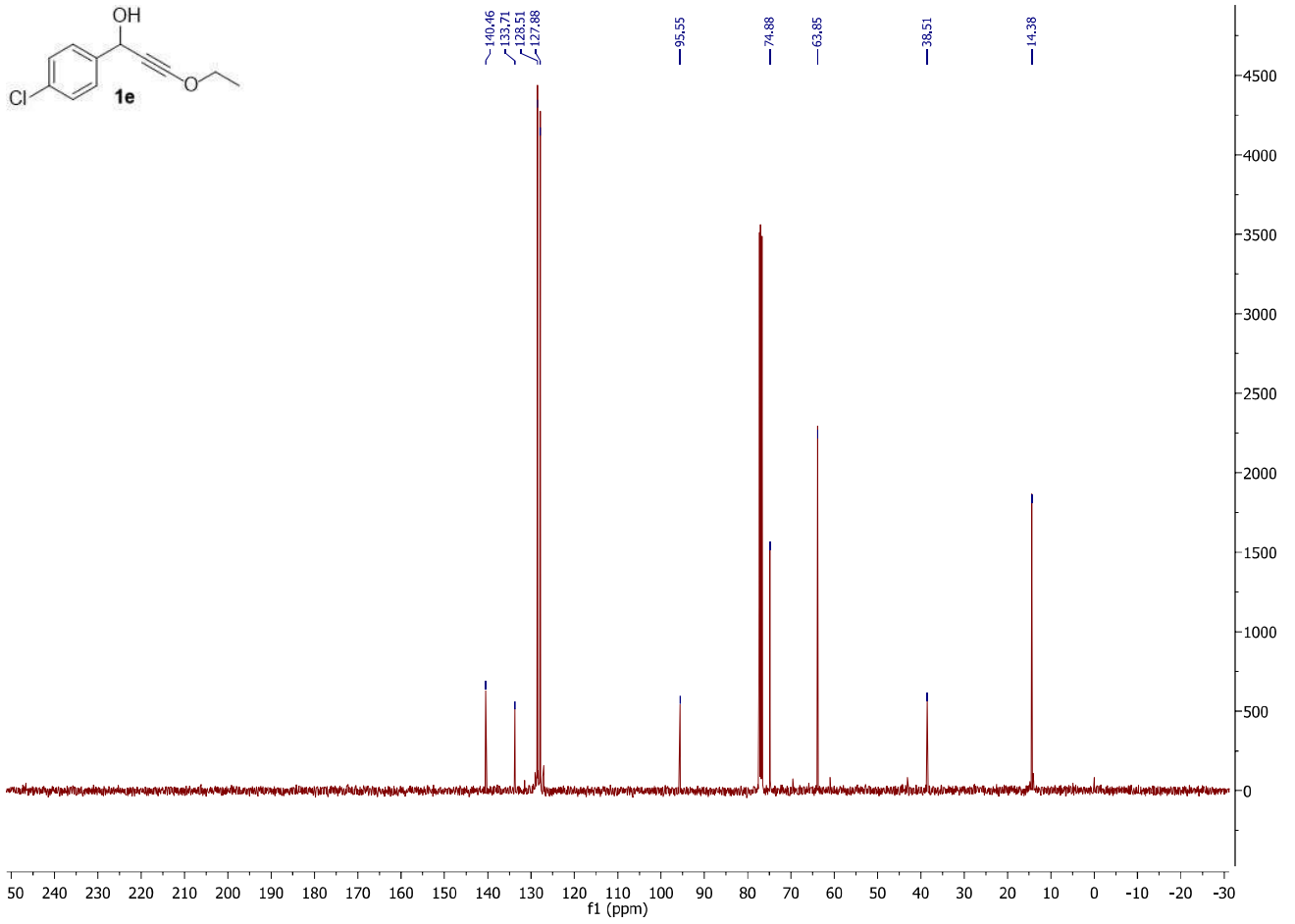
Cl
O
1e
7.49
7.49
7.47
7.47
7.35
7.33
5.49
5.48
4.19
4.17
4.15
4.13
2.03
2.02
1.42
1.40
1.38
1.99
2.08
1.00
1.89
1.03
2.79
f1 (ppm)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Cl
O
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95.55
74.88
63.85
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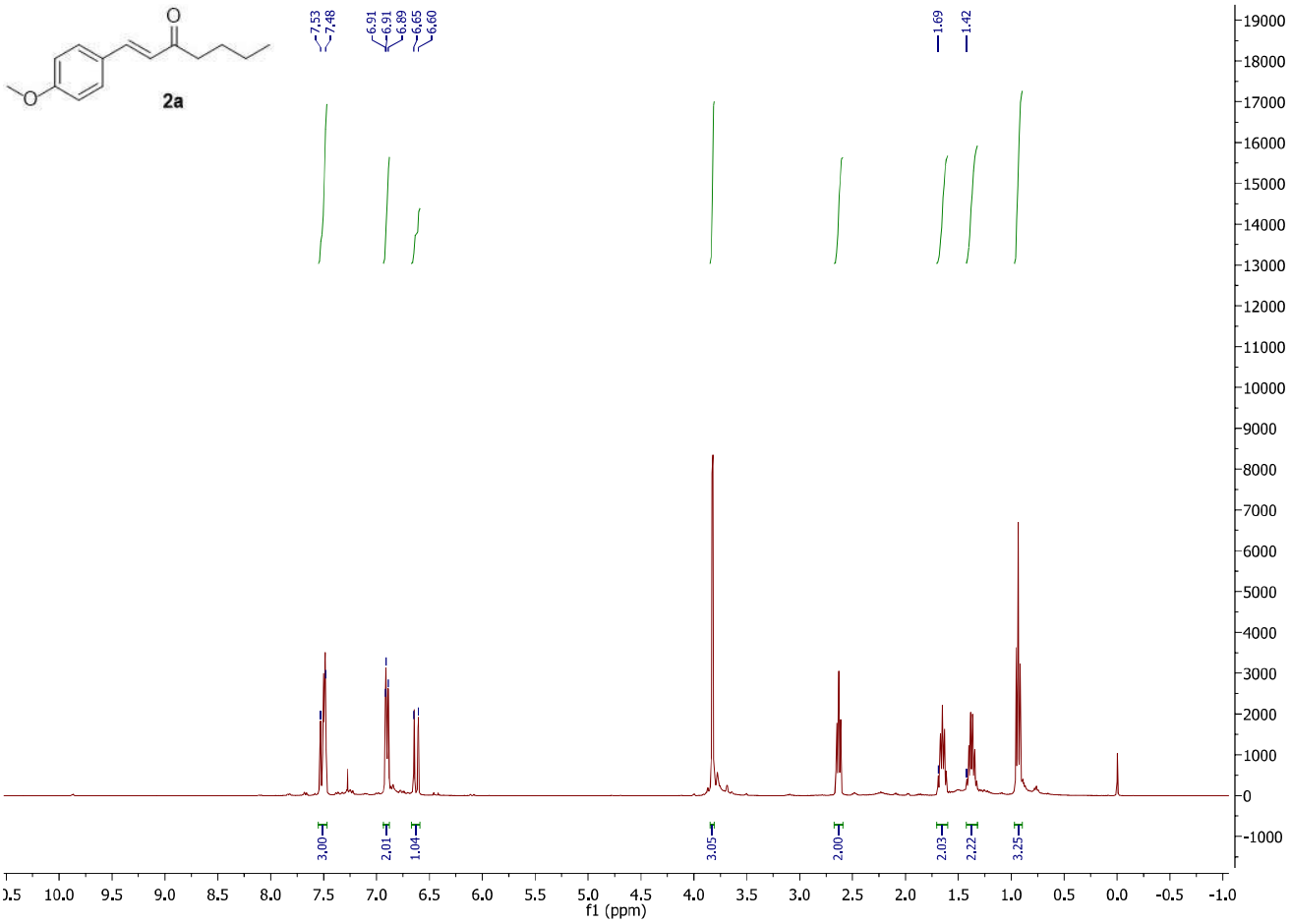
f1 (ppm)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7.53
7.48
6.91
6.91
6.89
6.65
6.60
1.69
1.42
3.00
2.01
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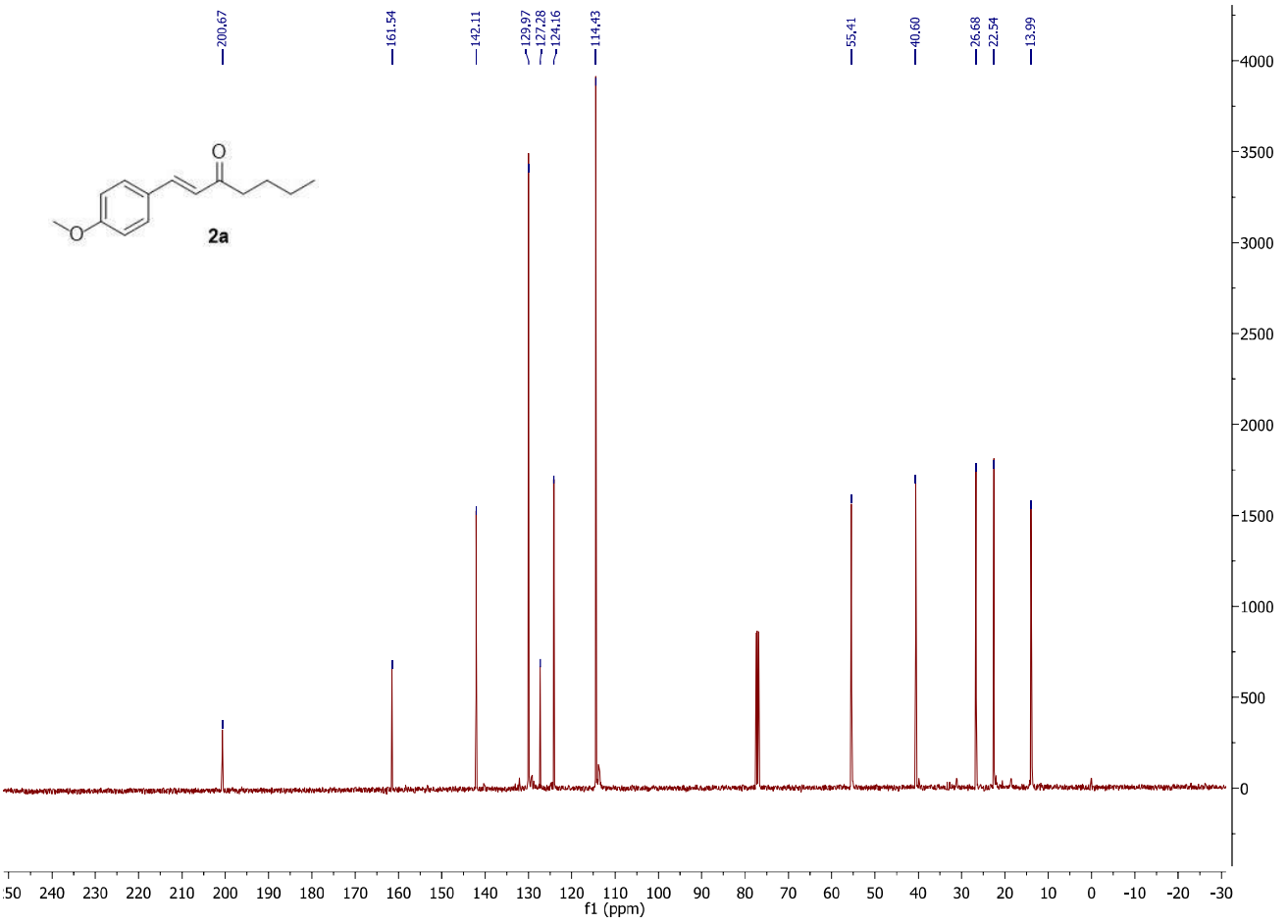
2.00
2.03
2.22
3.25
f1 (ppm)


2a
200.67
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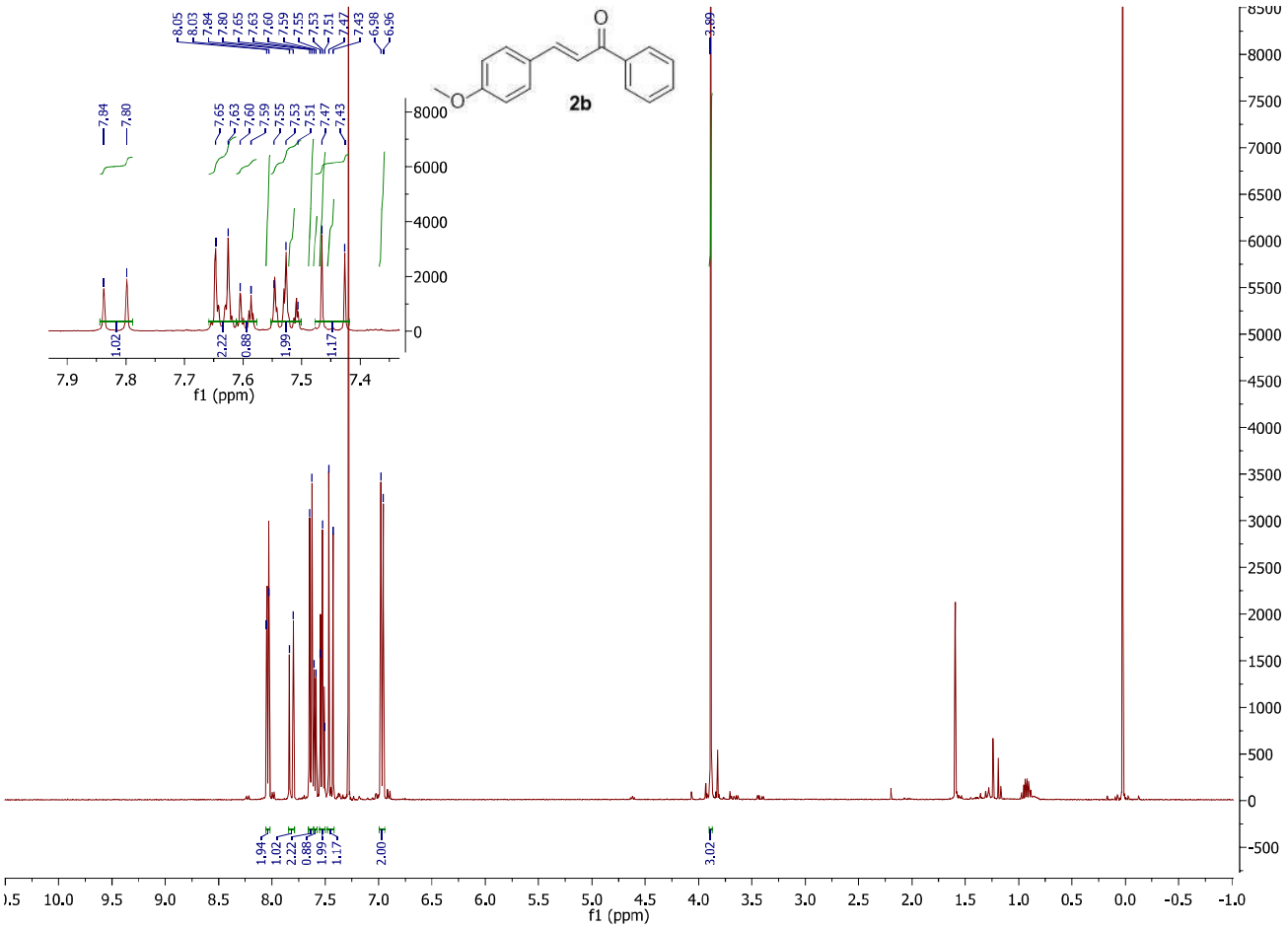
142.11
129.97
127.28
124.16
114.43
55.41
40.60
26.68
22.54
13.99
f1 (ppm)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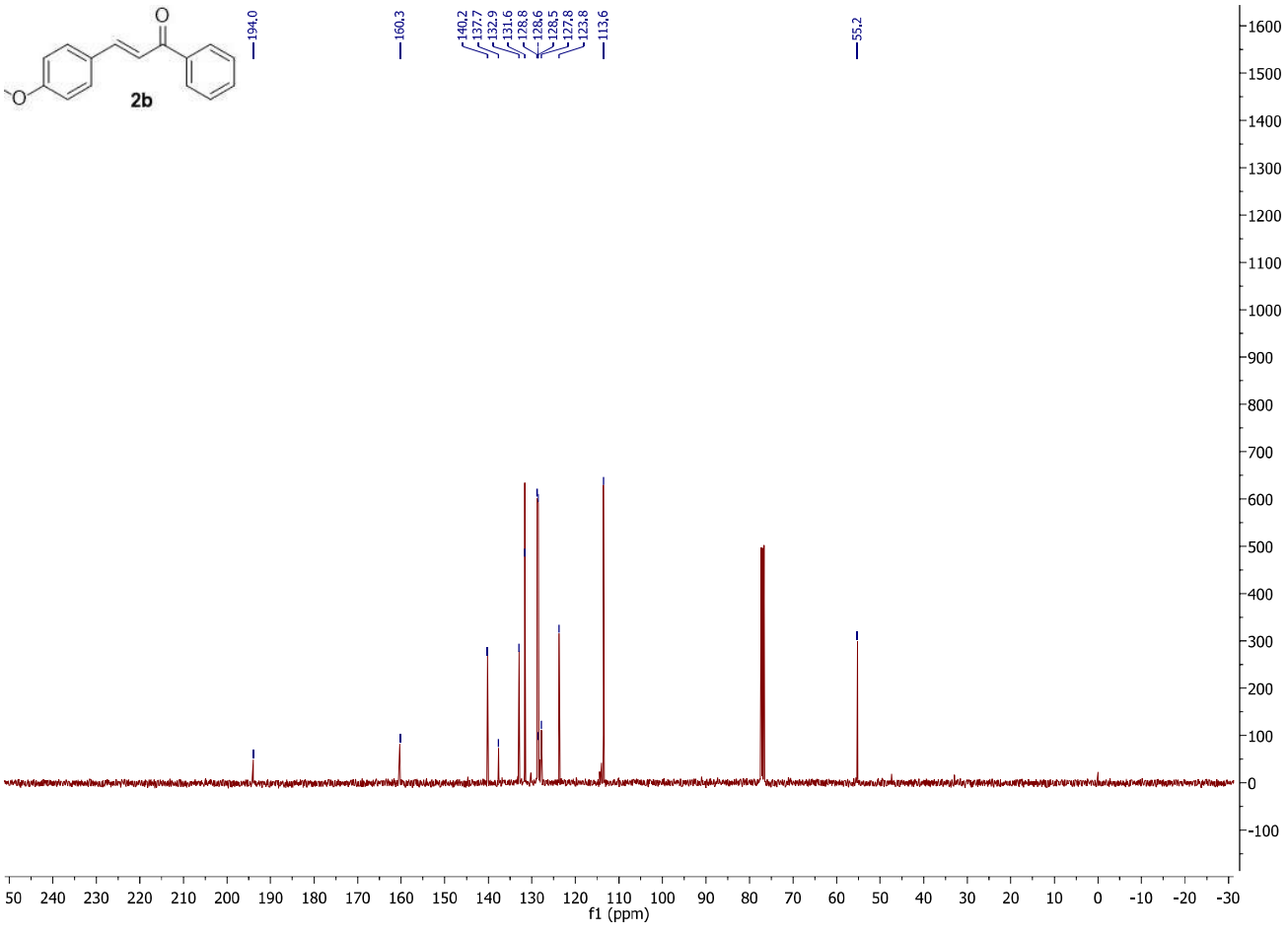
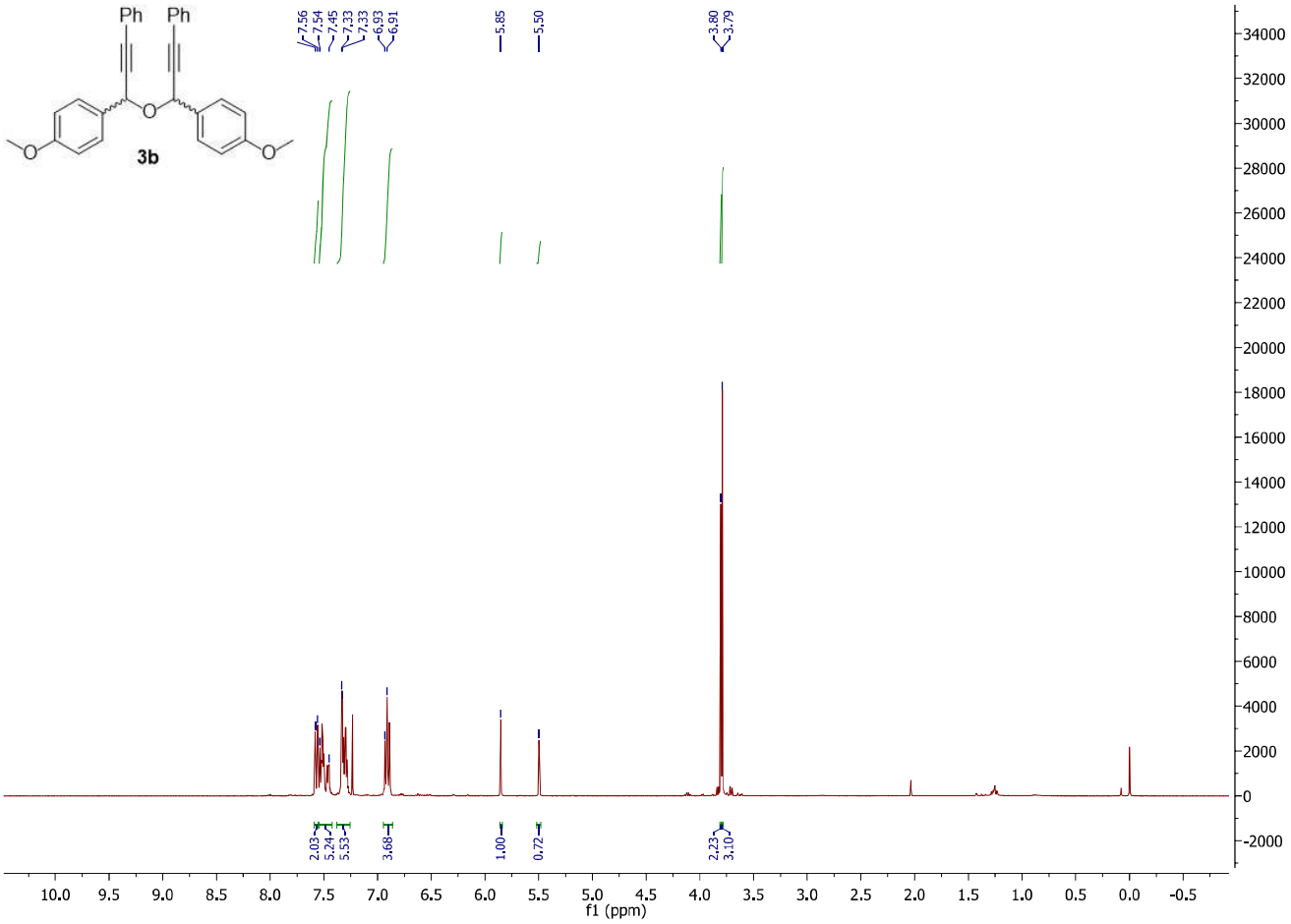
f1 (ppm)


2b
f1 (ppm)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Ph
O
O
O
3b
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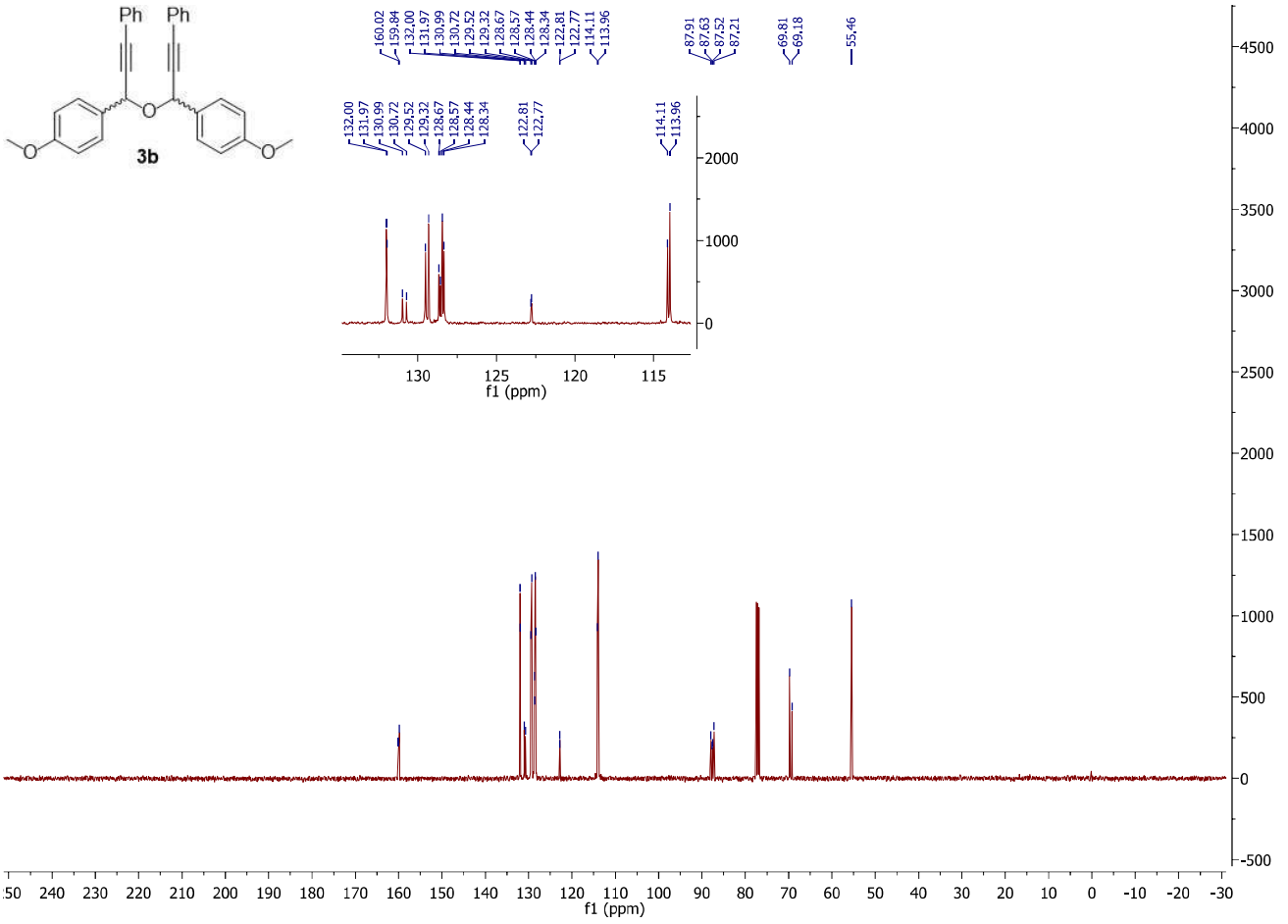
7.54
7.45
7.33
7.33
6.93
6.91
5.85
5.50
3.80
3.79
2.03
5.24
5.53
3.68
1.00
0.72
2.23
3.10
f1 (ppm)


Ph
Ph
O
O
O
3b
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159.84
132.00
131.97
130.99
130.72
129.52
129.32
128.67
128.57
128.44
128.34
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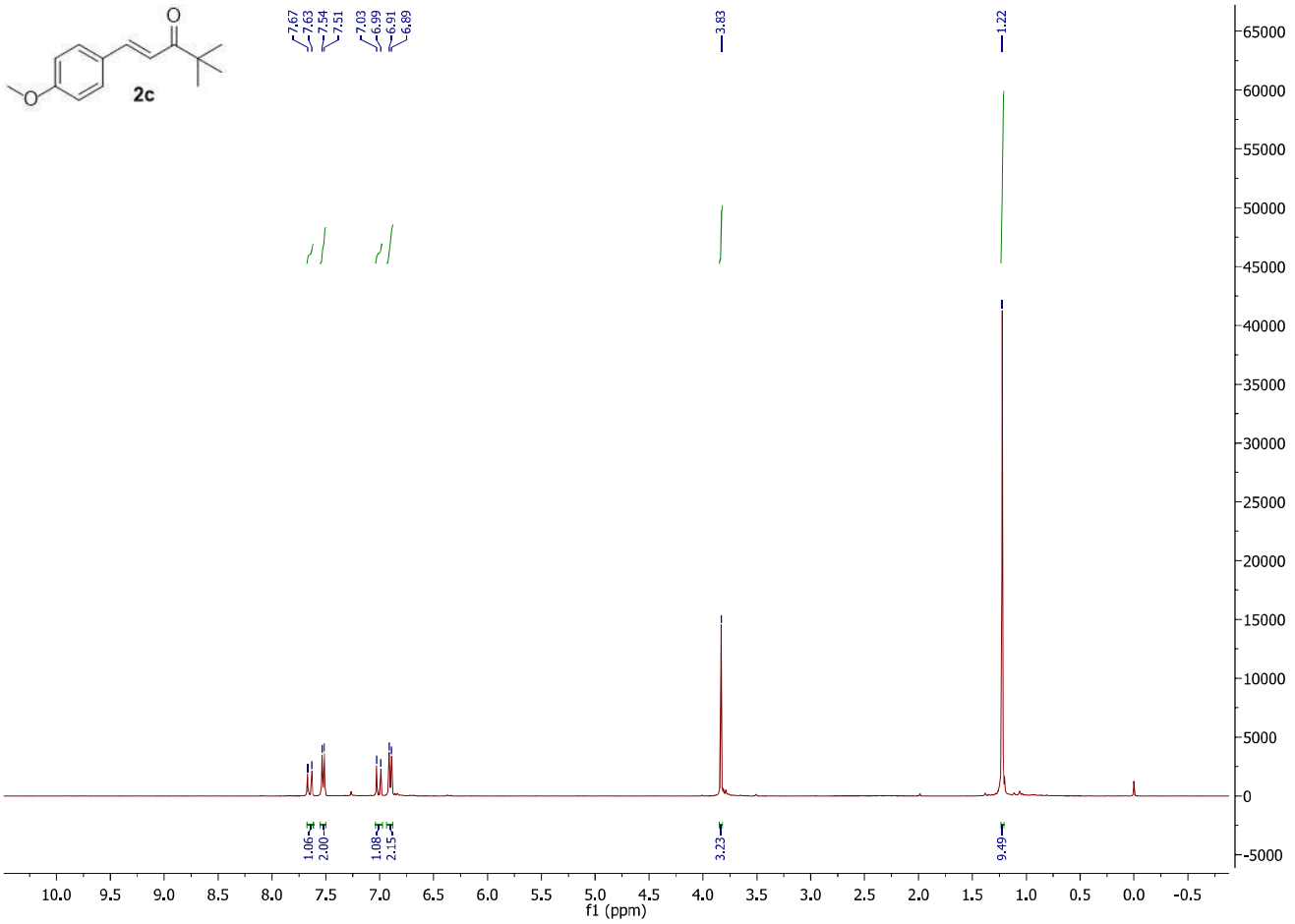
87.63
87.52
87.21
69.81
69.18
55.46
f1 (ppm)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7.54
7.51
7.03
6.99
6.91
6.89
3.83
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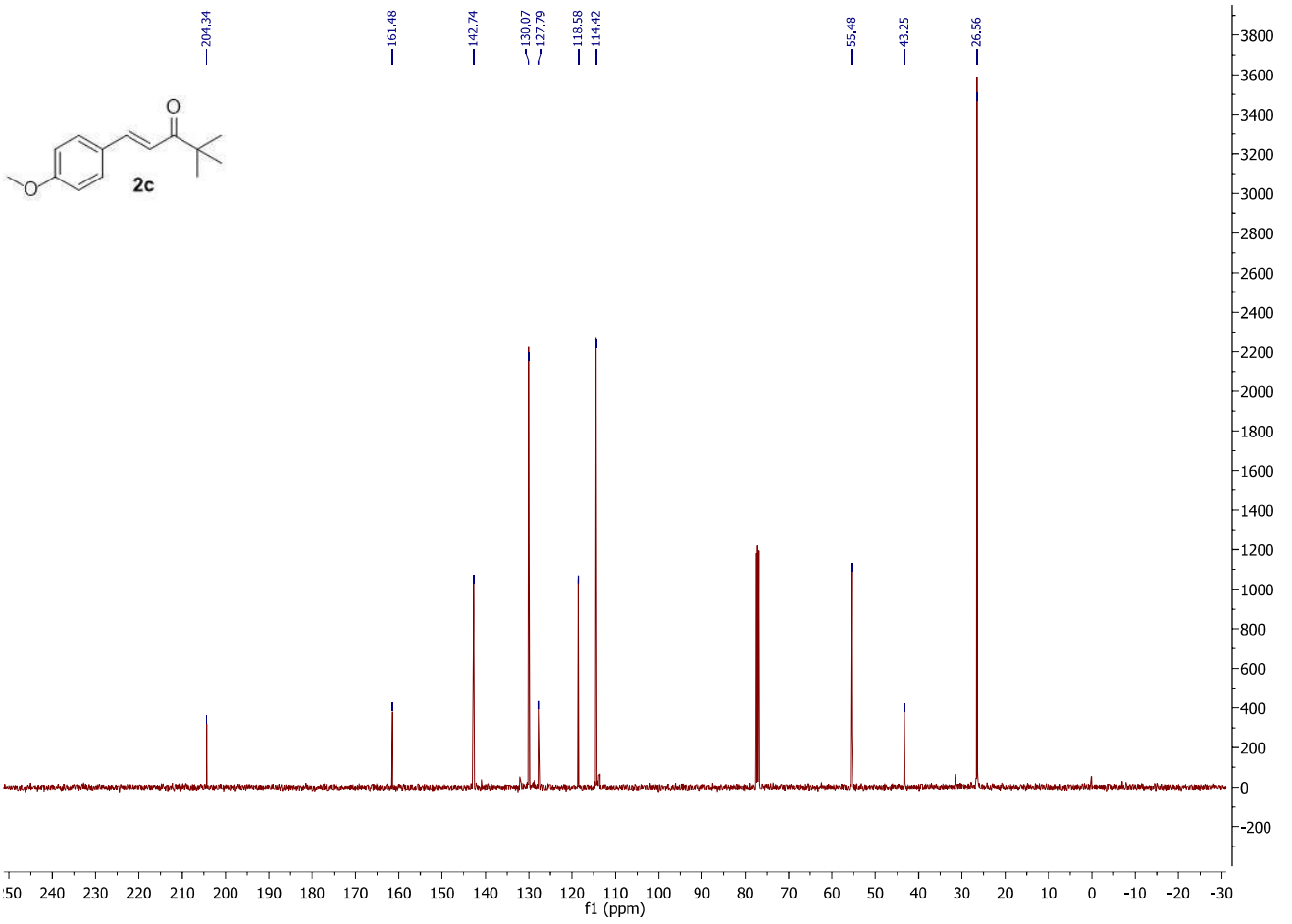
1.06
2.00
1.08
2.15
3.23
9.49
f1 (ppm)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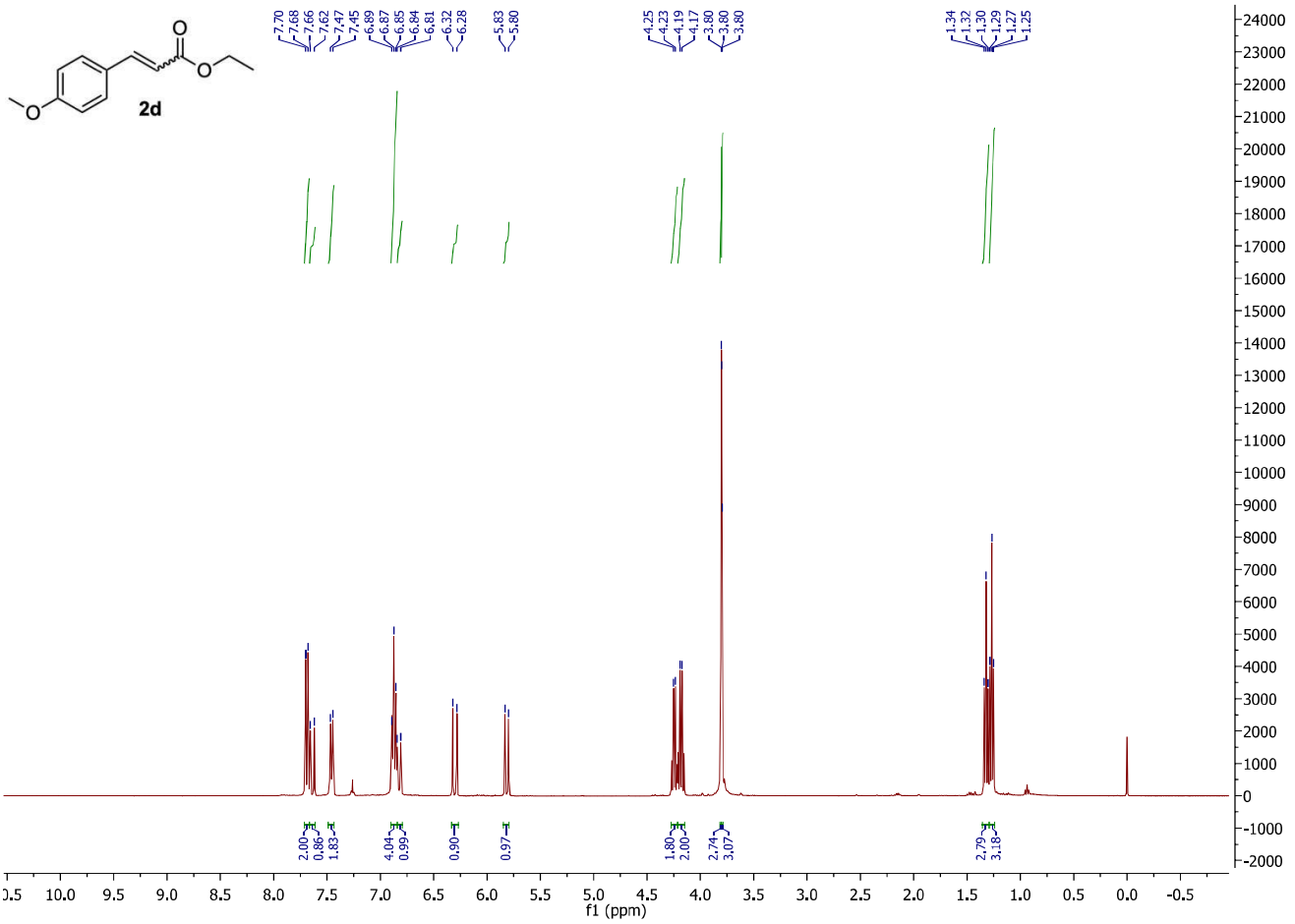
114.42
55.48
43.25
26.56
f1 (ppm)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7.68
7.66
7.62
7.47
7.45
6.89
6.87
6.85
6.84
6.81
6.32
6.28
5.83
5.80
4.25
4.23
4.19
4.17
3.80
3.80
3.80
1.34
1.32
1.30
1.29
1.27
1.25
2.00
0.86
1.83
4.04
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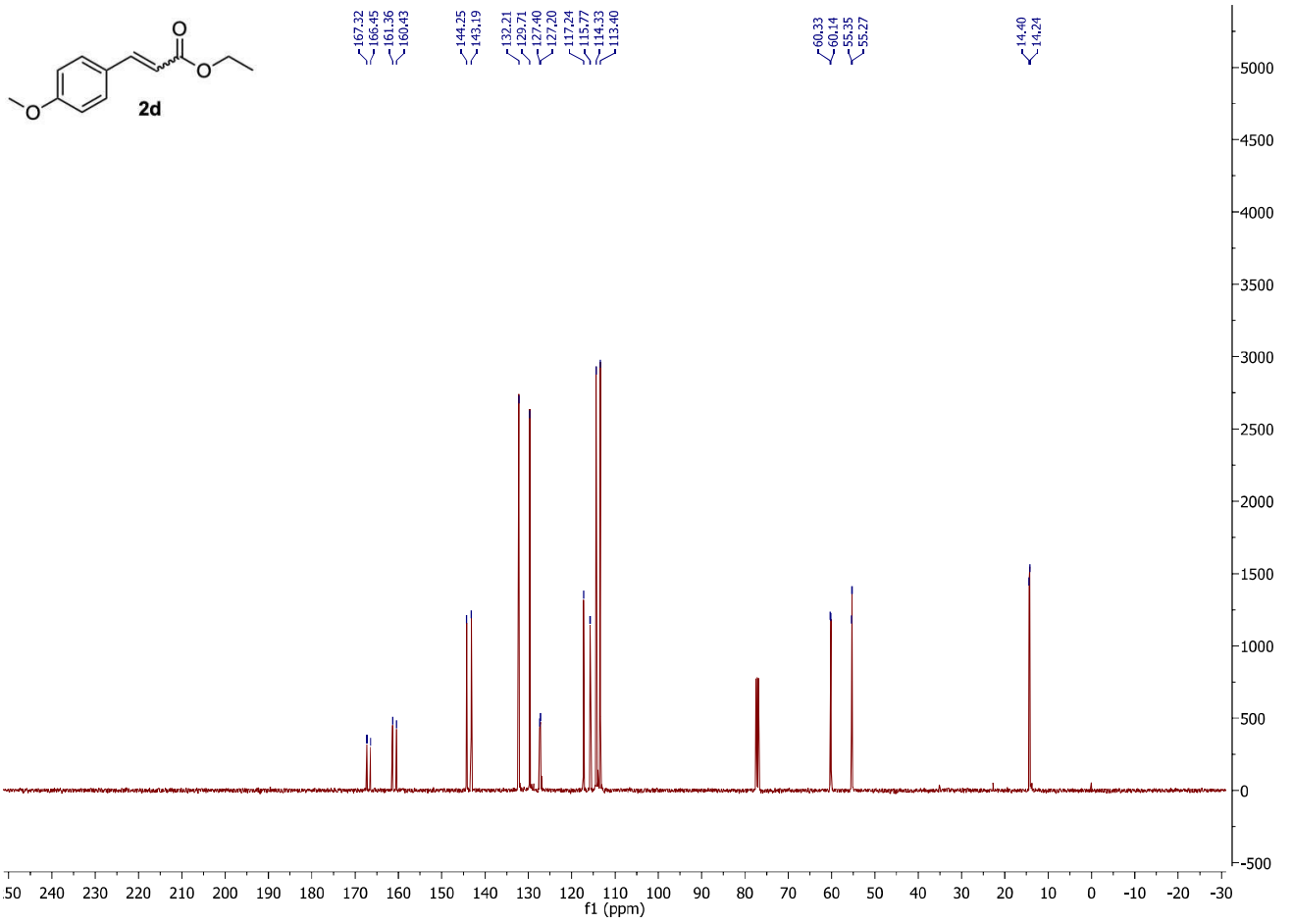
0.90
0.97
1.80
2.00
2.74
3.07
2.79
3.18
f1 (ppm)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127.40
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60.33
60.14
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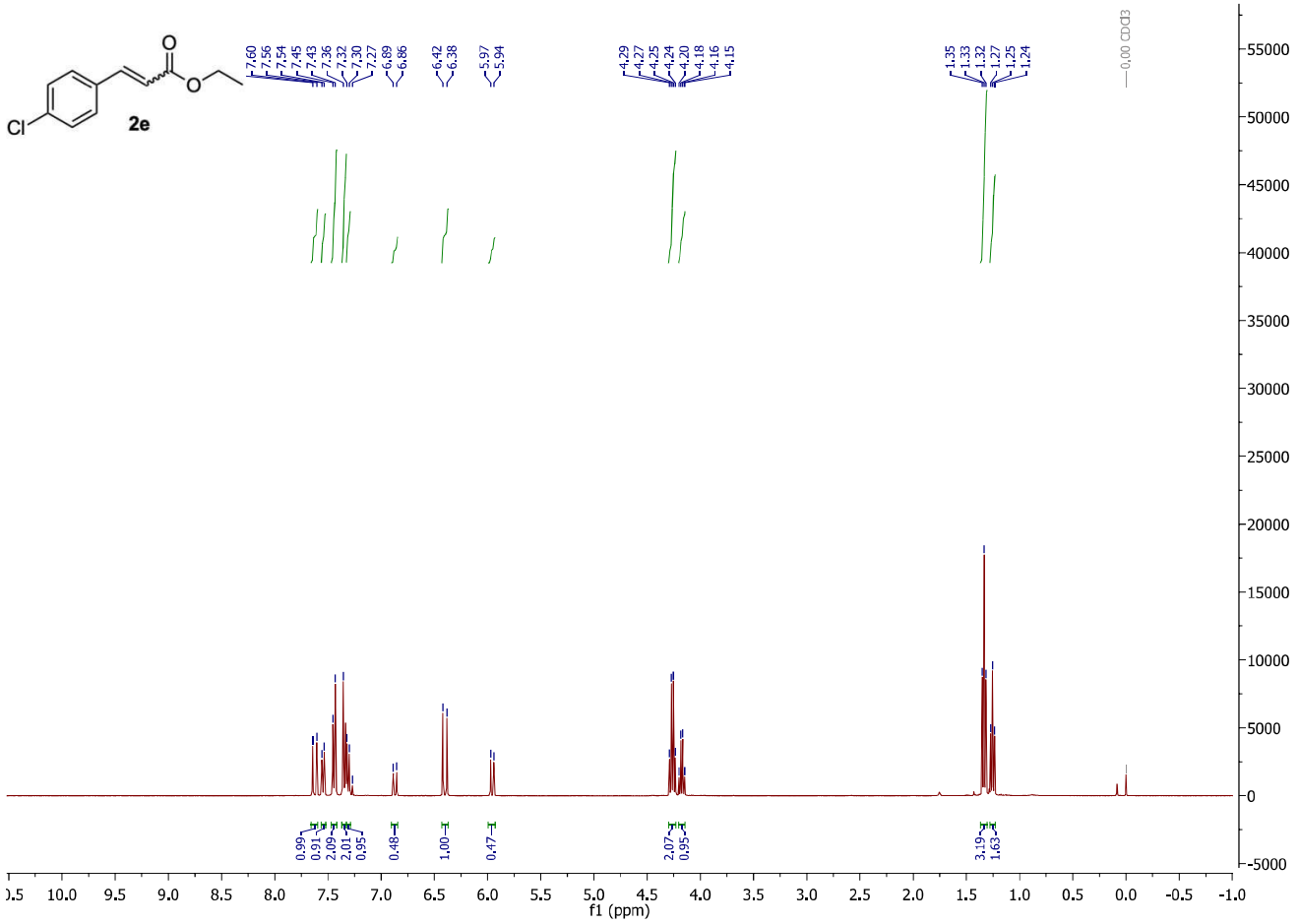
14.24
f1 (ppm)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O
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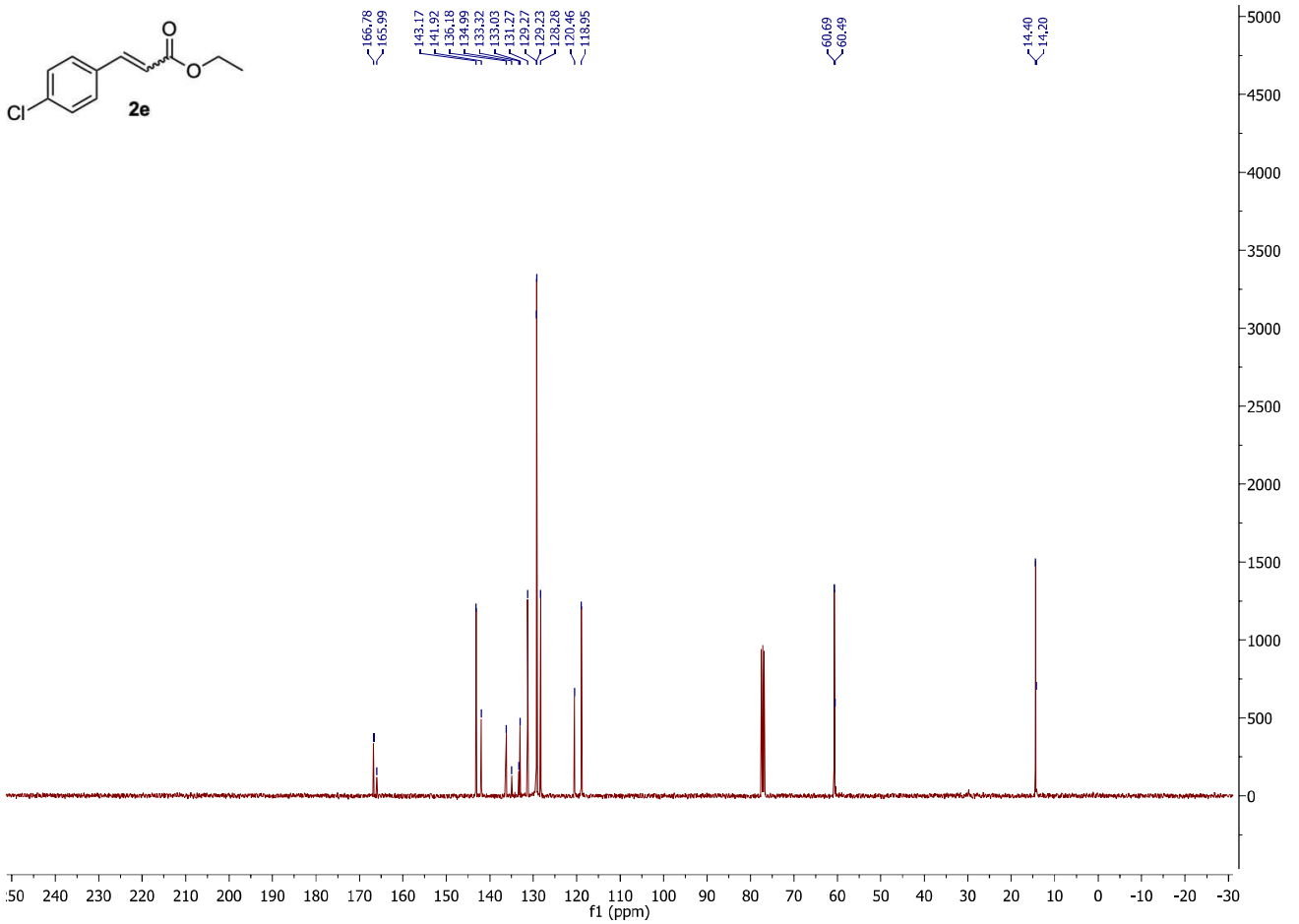
7.56
7.54
7.45
7.43
7.36
7.32
7.30
7.27
6.89
6.86
6.42
6.38
5.97
5.94
4.29
4.27
4.25
4.24
4.20
4.18
4.16
4.15
1.35
1.33
1.32
1.27
1.25
1.24
0.00 CDCl3
0.99
0.91
2.09
2.01
0.95
0.48
1.00
0.47
2.07
0.95
3.19
1.63
f1 (ppm)


2e
Cl
O
O
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143.17
141.92
136.18
134.99
133.32
133.03
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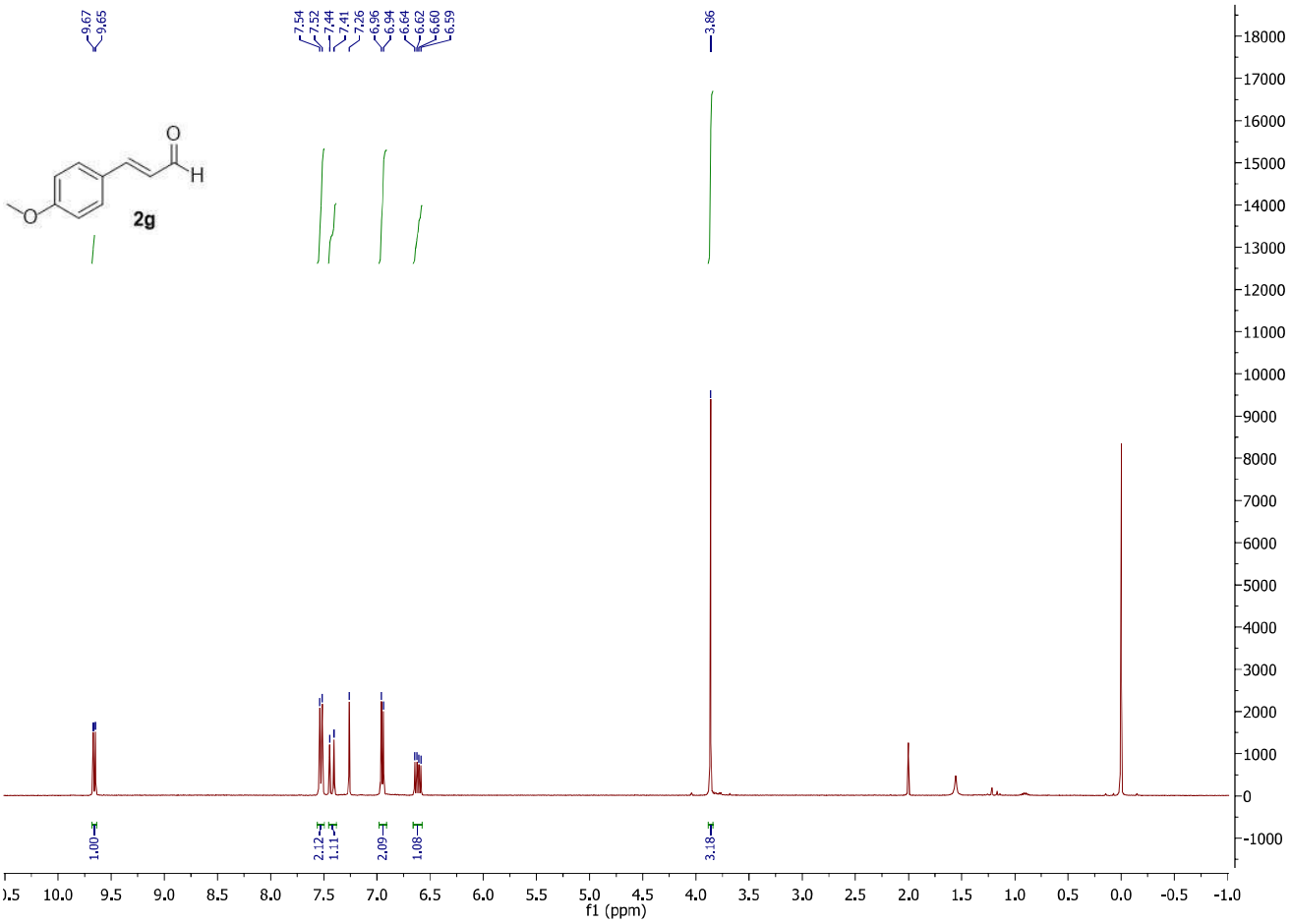
129.23
128.28
120.46
118.95
60.69
60.49
14.40
14.20
f1 (ppm)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7.26
6.96
6.94
6.64
6.62
6.60
6.59
3.86
O
H
O
2g
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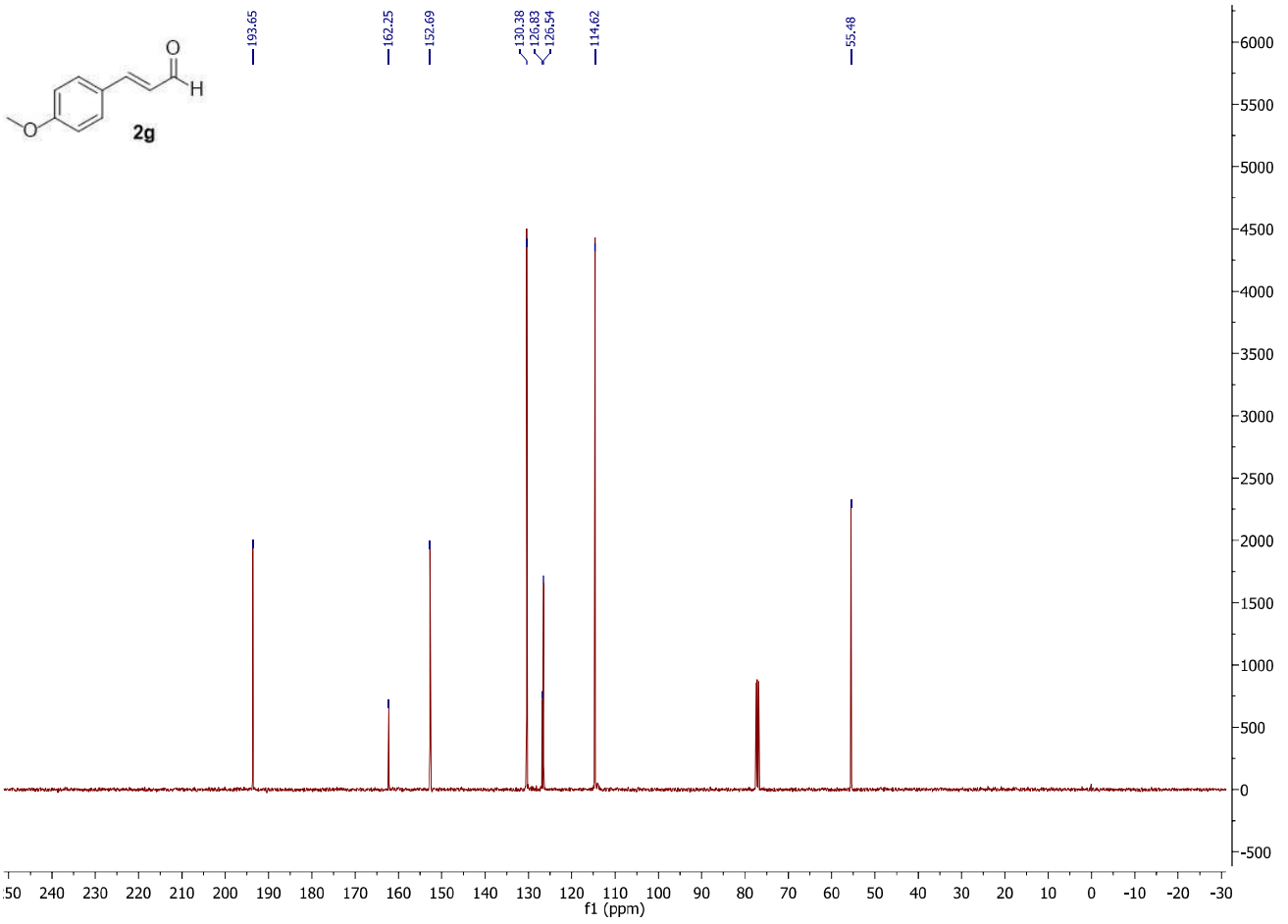
2.12
1.11
2.09
1.08
3.18
f1 (ppm)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114.62
55.48
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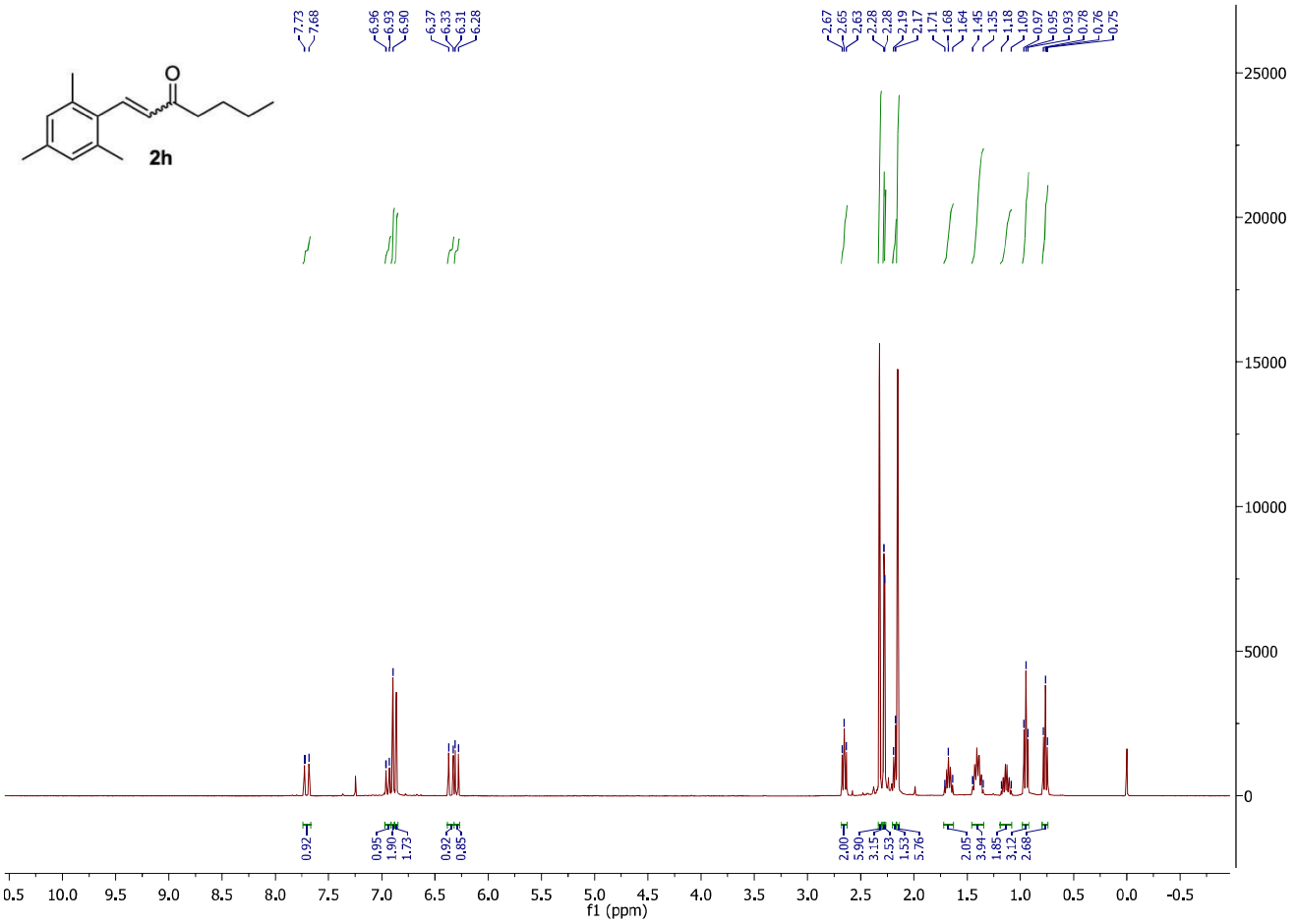
H
O
2g
f1 (ppm)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6.90
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6.28
2.67
2.65
2.63
2.28
2.28
2.19
2.17
1.71
1.68
1.64
1.45
1.35
1.18
1.09
0.97
0.95
0.93
0.78
0.76
0.75
0.92
0.95
1.90
1.73
0.92
0.85
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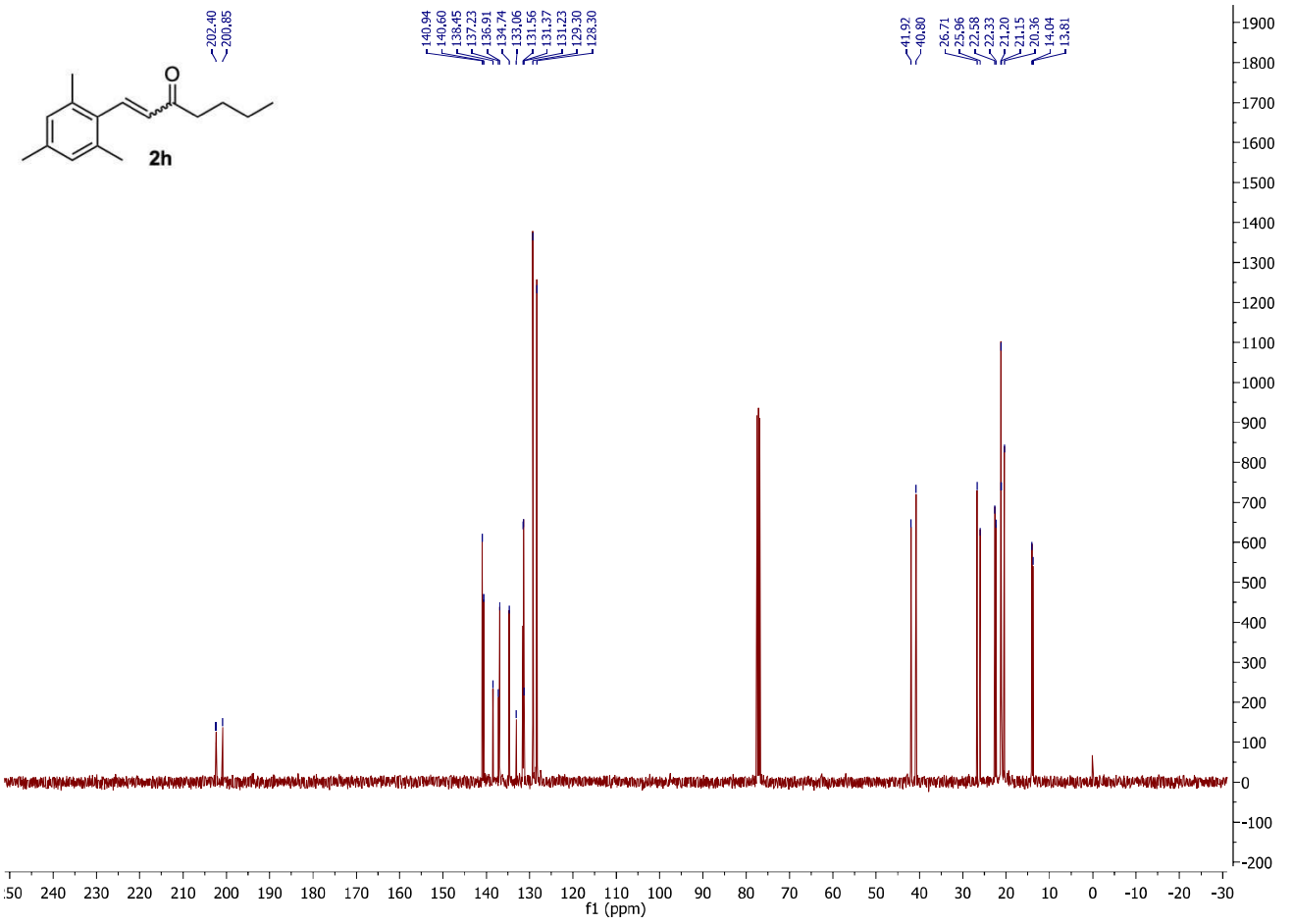
1.85
3.12
2.68
f1 (ppm)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200.85
140.94
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133.06
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131.37
131.23
129.30
128.30
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25.96
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22.33
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21.15
20.36
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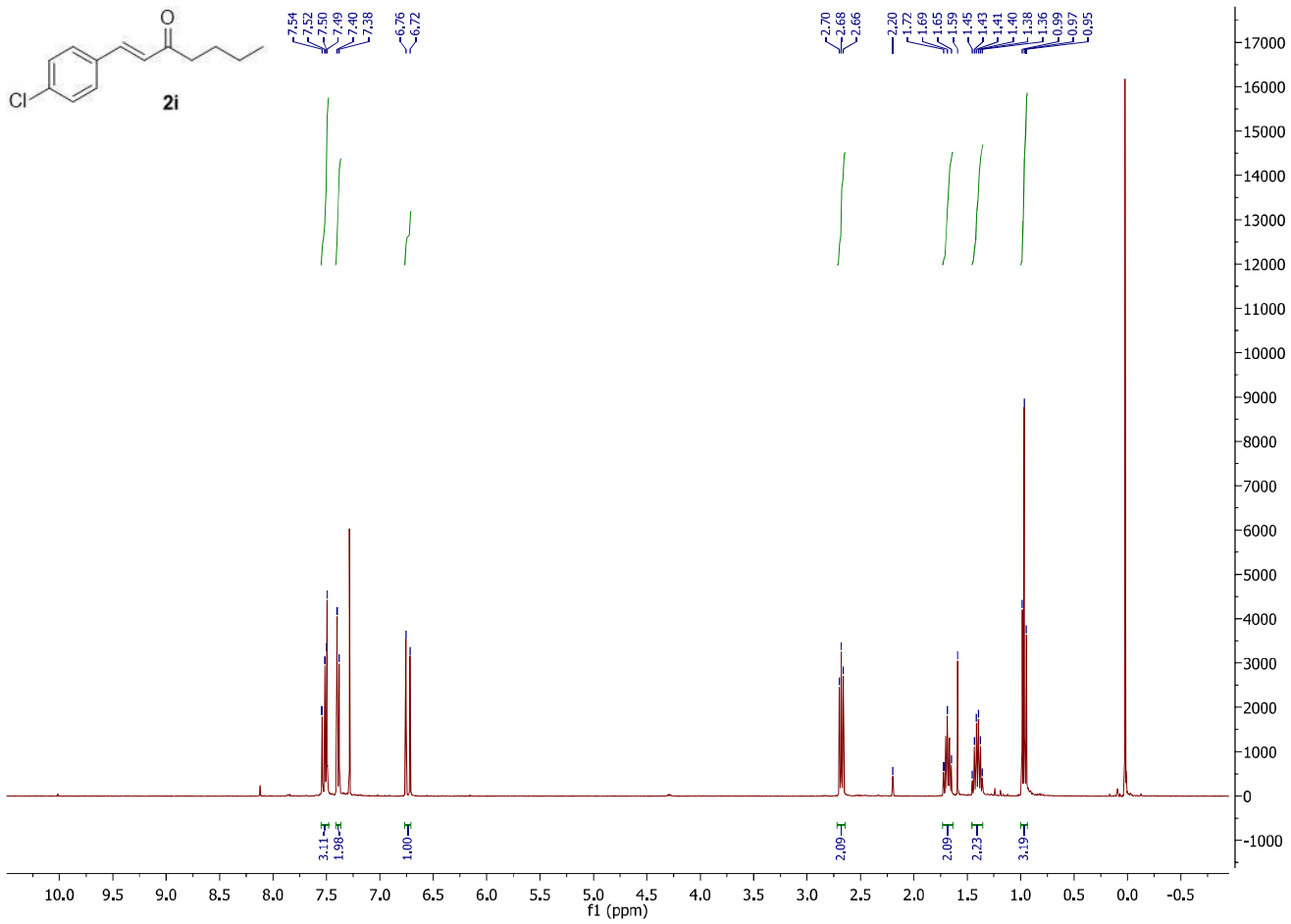
f1 (ppm)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2.68
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2.20
1.72
1.69
1.65
1.59
1.45
1.43
1.41
1.40
1.38
1.36
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0.95
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2.09
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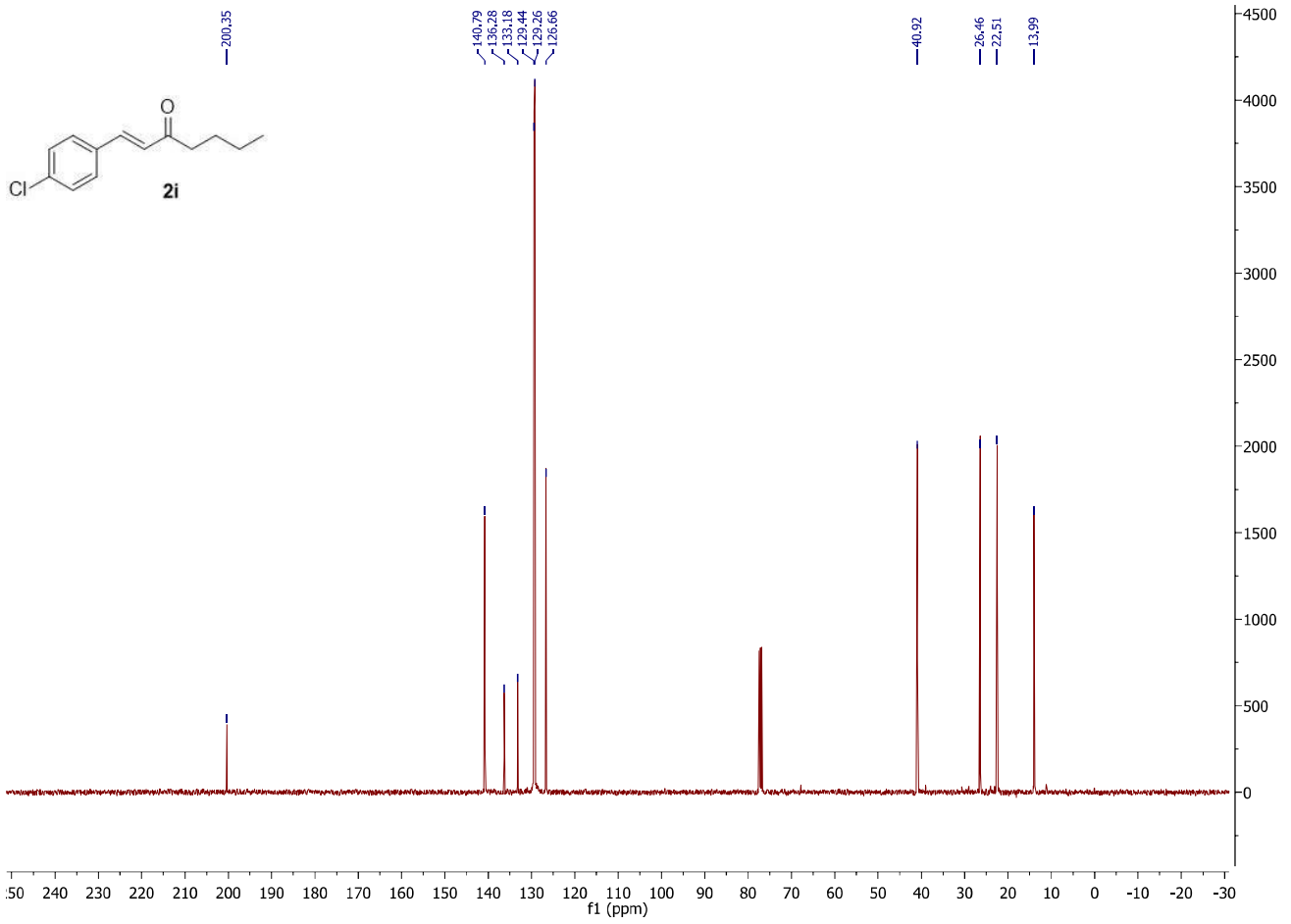
3.19
f1 (ppm)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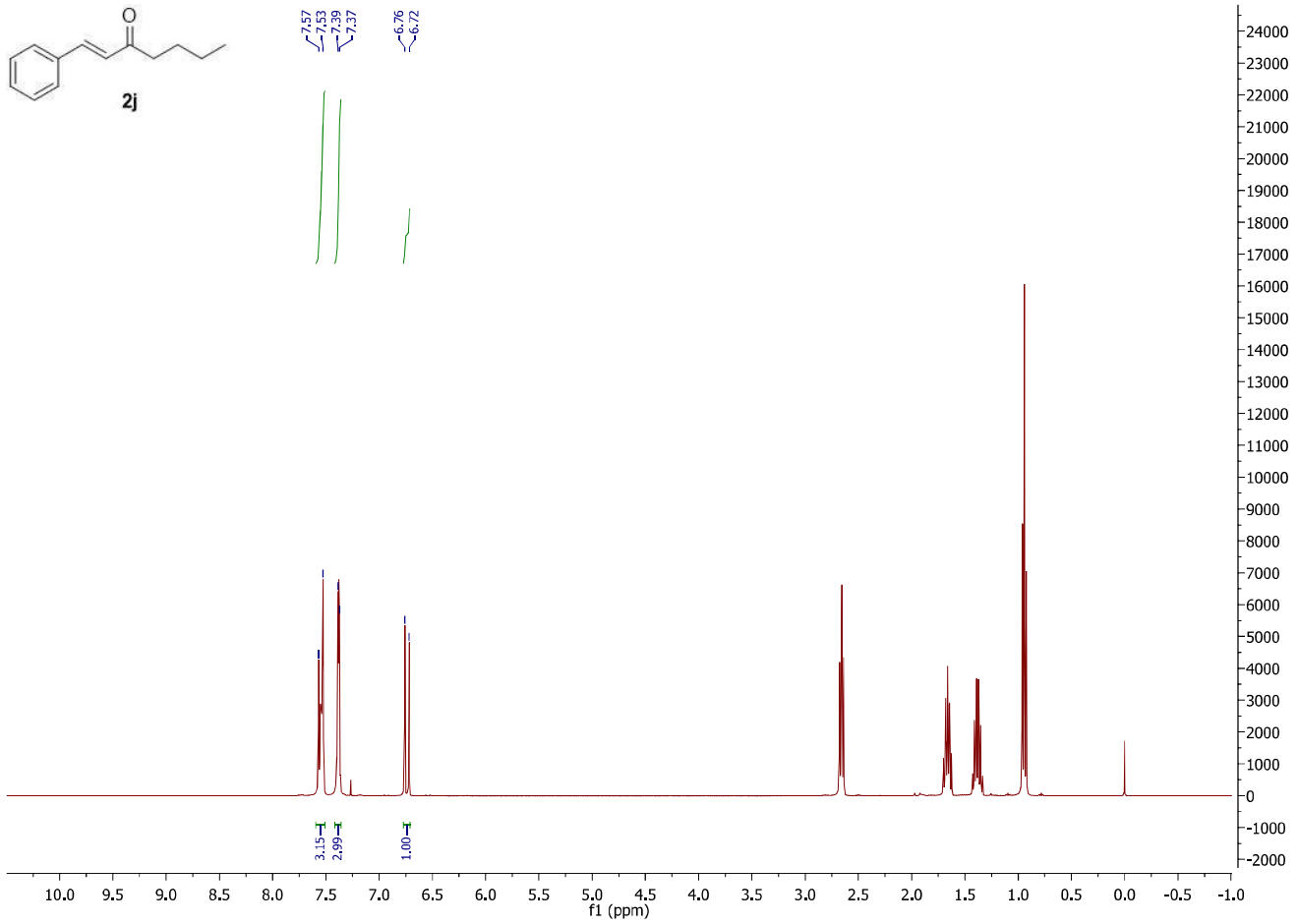
f1 (ppm)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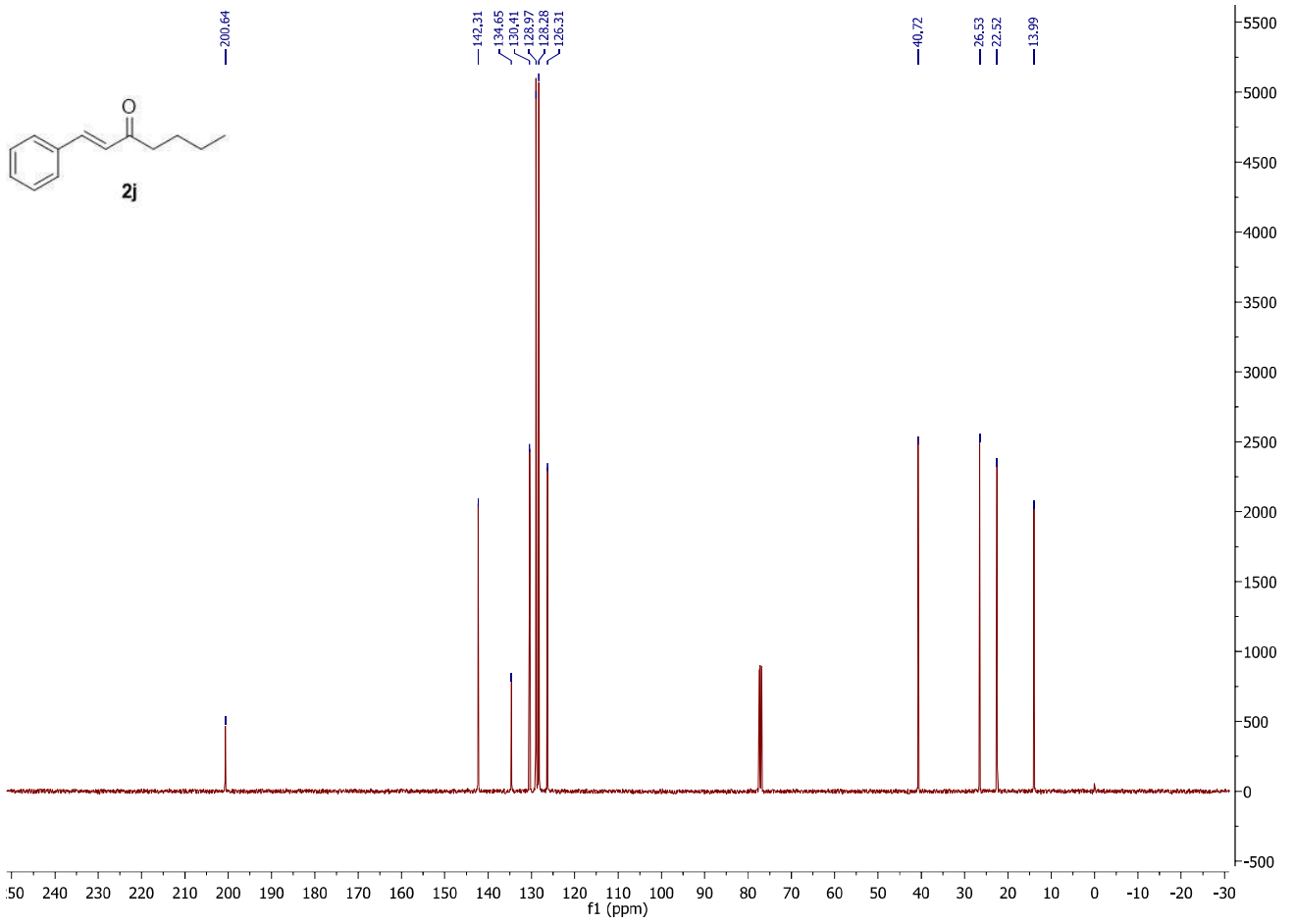
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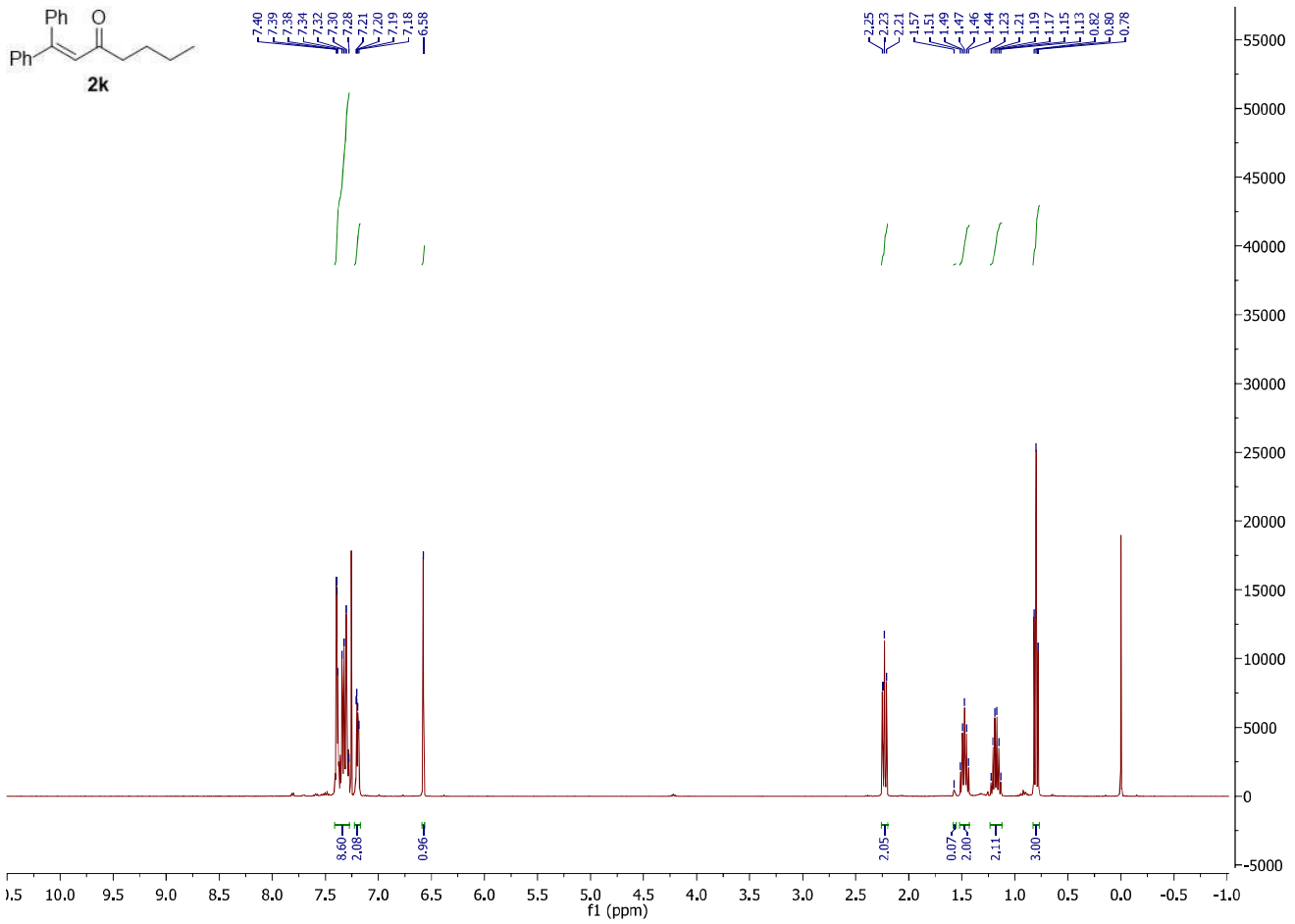
128.28
126.31
40.72
26.53
22.52
13.99
f1 (ppm)


Ph
O
Ph
2k
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7.38
7.34
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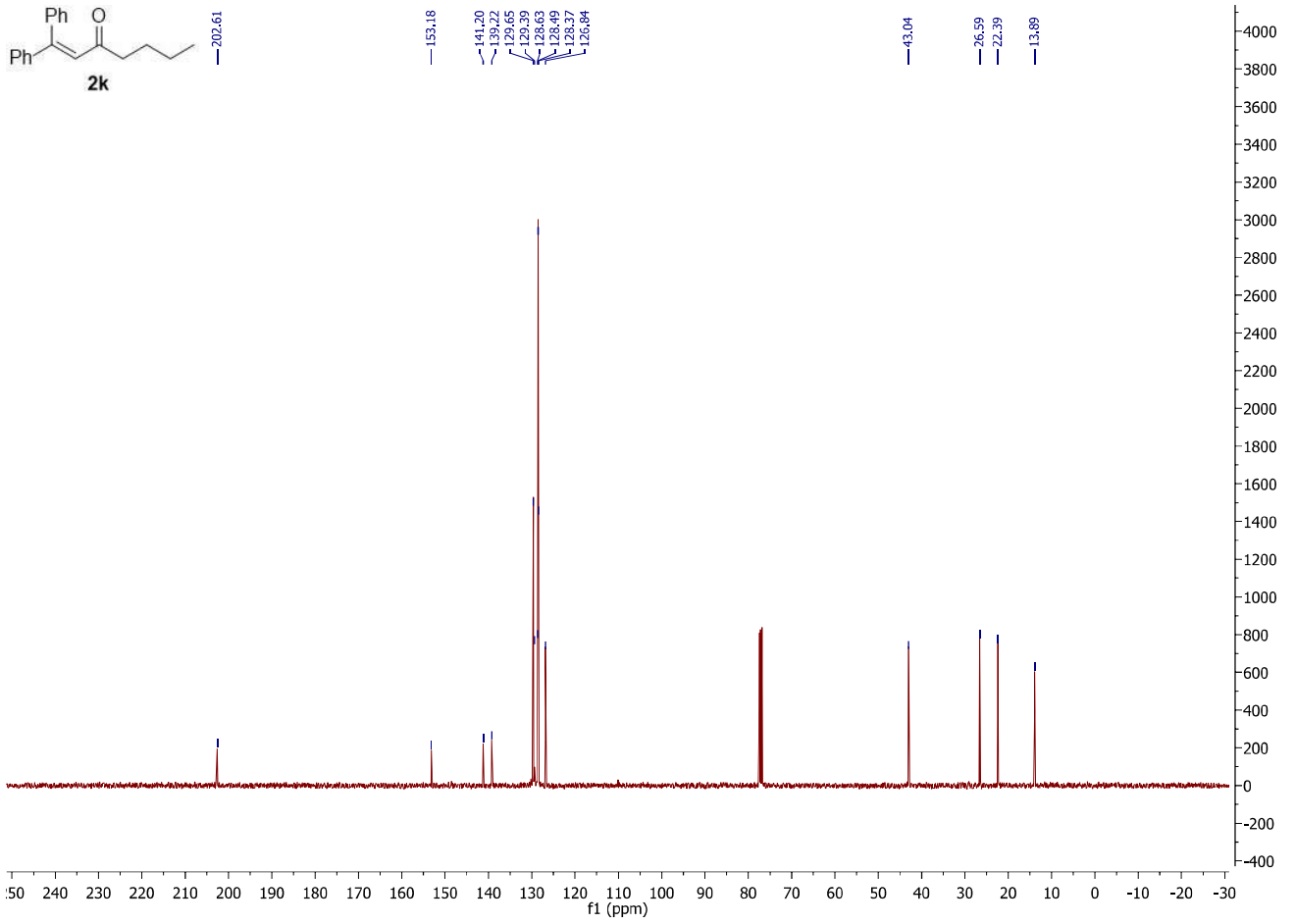
7.30
7.28
7.21
7.20
7.19
7.18
6.58
2.25
2.23
2.21
1.57
1.51
1.49
1.47
1.46
1.44
1.23
1.21
1.19
1.17
1.15
1.13
0.82
0.80
0.78
8.60
2.08
0.96
2.05
0.07
2.00
2.11
3.00
f1 (ppm)


Ph
O
Ph
2k
202.61
153.18
141.20
139.22
129.65
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128.63
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128.37
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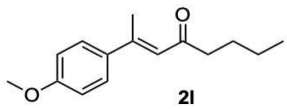
43.04
26.59
22.39
13.89
f1 (ppm)


O
O

2l

7.5
7.5
6.9
6.9
6.5
6.5
6.5
6.5
3.8
2.5
2.5
2.5
2.5
2.5
1.6
1.6
1.6
1.6
1.6
1.4
1.4
1.4
1.4
1.3
0.9
0.9
0.9
1.99
2.07
1.00
3.08
4.90
2.36
2.35
3.16
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.5
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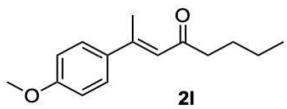
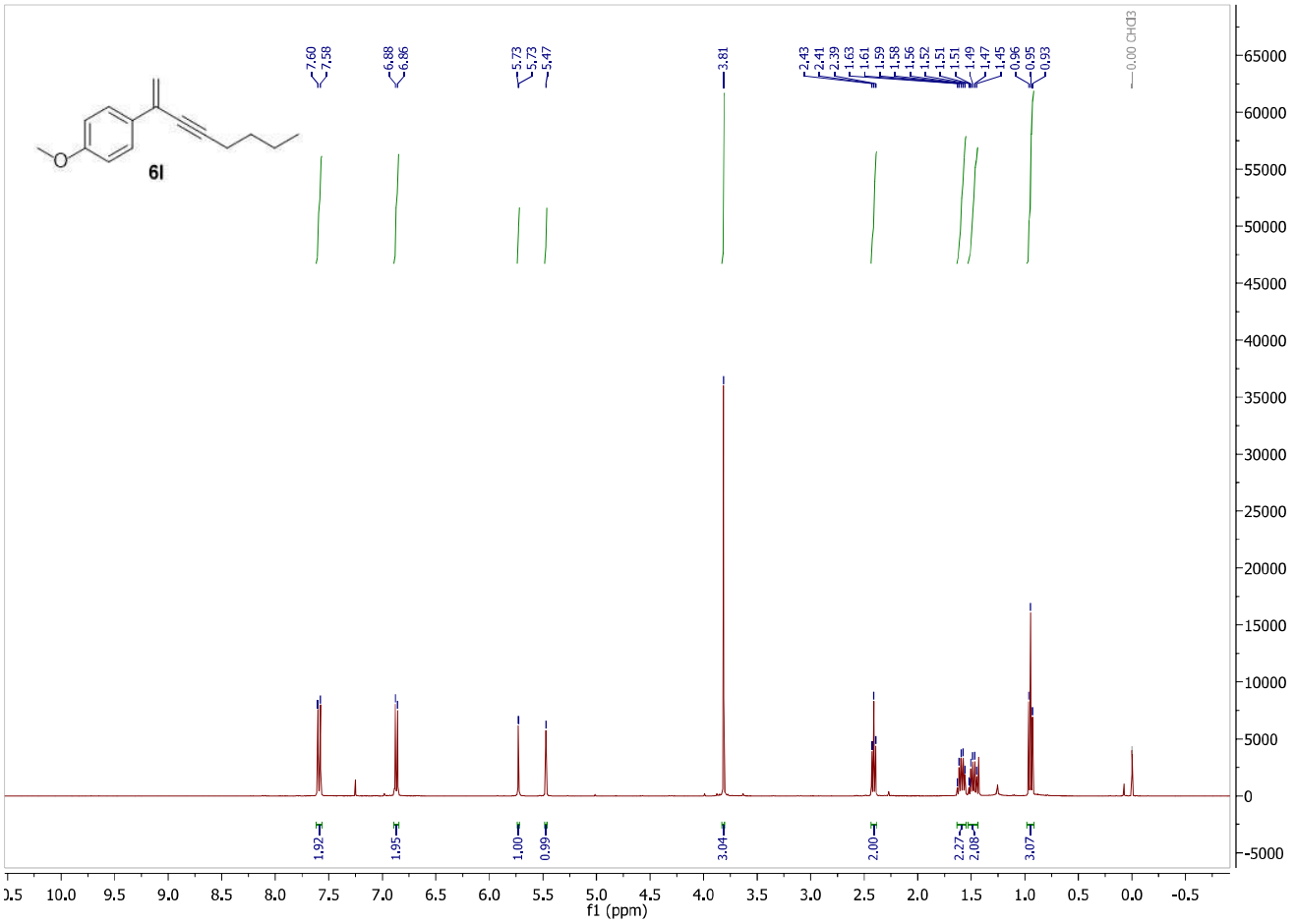
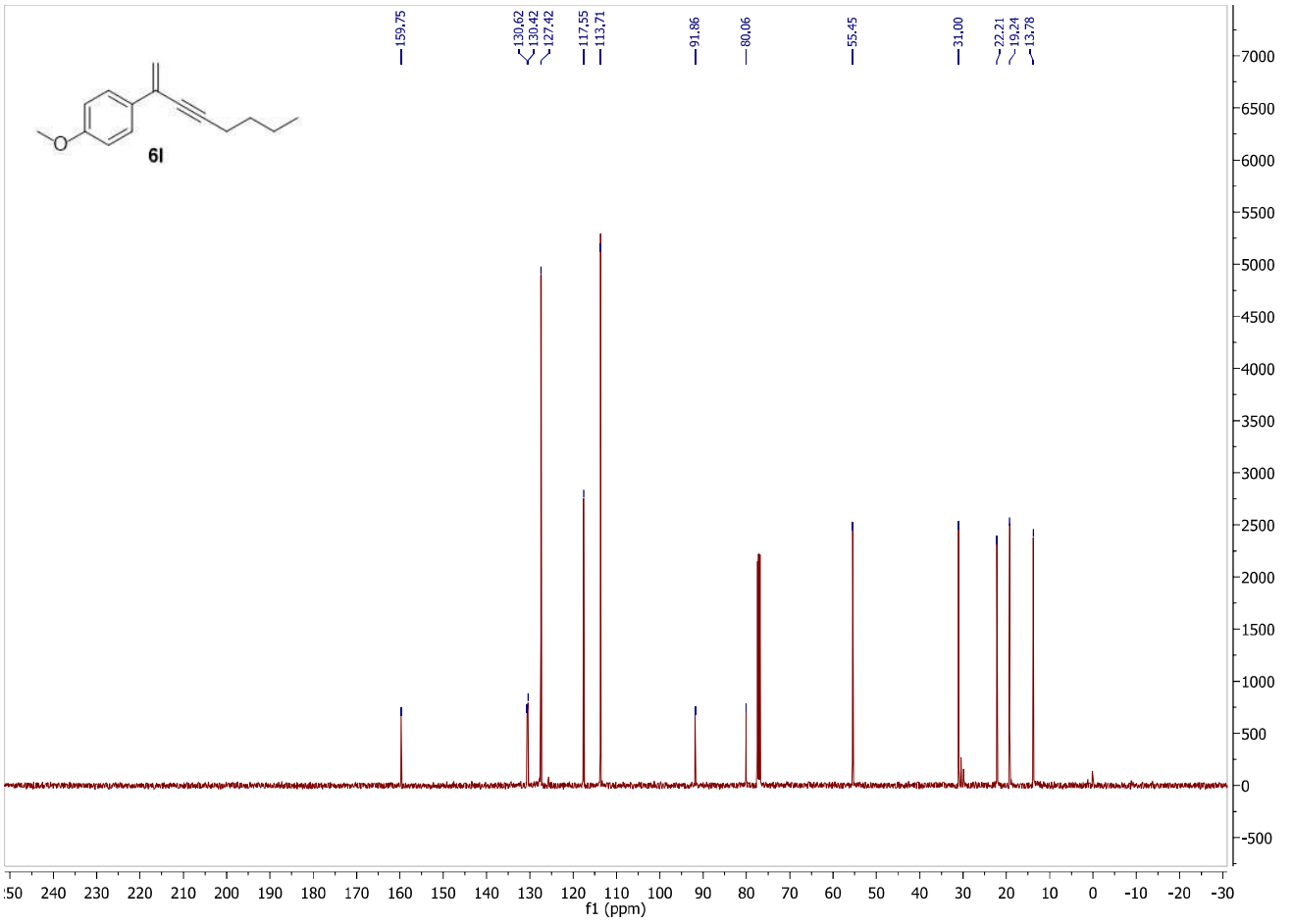
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


O
O

2l

160.7
153.1
134.9
128.0
122.8
114.1
55.5
44.8
26.8
22.6
18.2
14.1
250
240
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
f1 (ppm)


6l
7.60
7.58
6.88
6.86
5.73
5.73
5.47
3.81
2.43
2.41
2.39
1.63
1.61
1.59
1.58
1.56
1.52
1.51
1.51
1.49
1.47
1.45
0.96
0.95
0.93
0.00 CHCl3
1.92
1.95
1.00
0.99
3.04
2.00
2.27
2.08
3.07
f1 (ppm)


6l
159.75
130.62
130.42
127.42
117.55
113.71
91.86
80.06
55.45
31.00
22.21
19.24
13.78
f1 (ppm)