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Supporting Information

A Photoredox Nozaki-Hiyama Reaction Catalytic in Chromium

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Synthetic Procedures and Characterization

General Method

All commercial chemicals and dry solvents were purchased from Sigma Aldrich, Alfa Aesar or TCI Chemicals. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Inova 400 NMR instrument with a 5 mm probe. All chemical shifts are referenced using deuterated solvent signals. GC-MS spectra were obtained by EI ionization at 70 eV on a Hewlett-Packard 5971 with GC injection; they are reported as: *m/z* (rel. intensity).

Flash chromatography purifications were carried out using VWR or Merck silica gel (40-63 µm particle size). Thin-layer chromatography was performed on Merck 60 F254 plates.

Aldehydes **1b-f**,[1] **1k**[1] and **1l**[2] were prepared according to literature procedures. Cinnamyl bromide was prepared starting from cinnamyl alcohol following literature procedures.[3] Photocatalysts **3CzBn** and **3DPAFIPN** were preparing according to literature procedures.[4]

Reaction mixtures were irradiated with Kessil® PR160L@456 nm.[5]

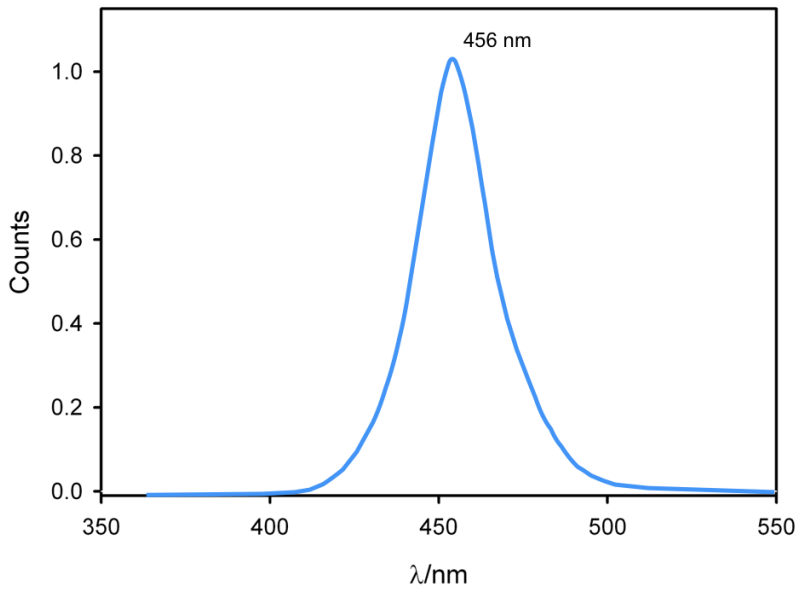
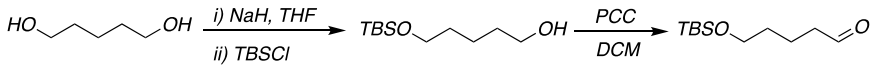


Figure S1. Emission profile of Kessil® PR160L 456 nm, the LED used for the irradiation of the reaction mixtures.

Synthesis of substrates

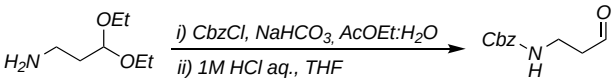
*Synthesis of aldehyde **1n***



To a suspension of NaH (60% in mineral oil, 320 mg, 5 mmol, 1 equiv.) in anhydrous THF (25 mL) at 0°C, 1,5-pentandiol (0.52 mL, 5 mmol) was slowly added. The reaction mixture was warmed at room temperature and stirred for 45 minutes. TBSCl (750 mg, 5 mmol, 1 equiv.) was slowly added at 0°C and the reaction mixture was stirred at room temperature for 45 minutes. A saturated aqueous solution of K_2CO_3 (20 mL) and diethyl ether (20 mL) were added, the layers were separated, and the aqueous phase was extracted with Et_2O (3 x 15 mL). The combined organic phases were dried over Na_2SO_4 and concentrated to obtain a yellow oil. The crude was further purified by flash column chromatography (cHex/EtOAc 15/85) to obtain desired product as yellow oil (95% yield, 1.035 g, 4.75 mmol).

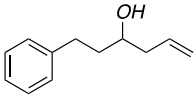
To a solution of monoprotected diol (1.035 g, 4.75 mmol) in DCM (40 ml), pyridinium chlorochromate (PCC, 1.537 g, 7.13 mmol, 1.5 equiv.) was added. After consumption of the starting alcohol (TLC), ethanol (20 mL) was added, and the reaction mixture was stirred for 1 hour. Celite was added and the suspension was filtered through a Celite pad. The solvent was removed under reduced pressure to obtain aldehyde **1n** (72% yield, 0.739 g, 3.42 mmol) as yellowish oil. The product presented a good purity and was used in the next step without any further purification. Spectroscopic data are in agreement with those reported in literature.[6]

*Synthesis of aldehyde **1o***

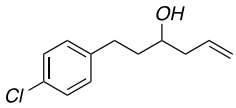


To a solution of 3,3-diethoxy-1-aminopropane (250 µL, 1.54 mmol) in EtOAc (3 mL) were added $NaHCO_3$ (650 mg, 7.7 mmol, 5 equiv.), water (3mL) and benzyl chloroformate (331 µL, 2.3 mmol, 1.5 equiv.). The resulting mixture was allowed to stir overnight at room temperature and checked by GC-MS and TLC (cHex/EtOAc 6/4) to complete conversion. After that, the layers were separated, and the aqueous phase was extracted with EtOAc (3 x 5 mL), dried over Na_2SO_4 and concentrated. The crude was dissolved in THF (0.5 mL), treated with 1M HCl (250 µL, 12 mol%) at 0°C and stirred overnight at room temperature. After the reaction mixture was diluted with Et_2O (10 mL) and washed with a saturated solution of $NaHCO_3$ (5 mL), the combined organic phase was dried over Na_2SO_4 and concentrated to obtain a yellowish oil. The crude was further purified by flash column chromatography (cHex/EtOAc 7/3) to obtain **1o** as yellow solid, 51% yield (0.79 mmol, 164 mg). Spectroscopic data are in agreement with those reported in literature.[6]

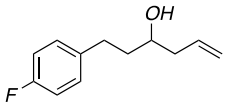
Characterization of final products



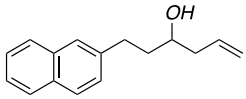
3a: pale yellow oil, 80% (0.16 mmol, 28 mg). The general procedure was applied using previously distilled **1a** (0.2 mmol, 26 mg, 24 µL), and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The title compound was isolated by flash column chromatography (100% DCM). Spectroscopic data are in agreement with those reported in literature.[7]



3b: pale yellow oil, 70% (0.14 mmol, 30 mg). The general procedure was applied using **1b** (0.2 mmol, 34 mg) and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The reaction time was extended to 48h. The title compound was isolated by flash column chromatography (100% DCM). Spectroscopic data are in agreement with those reported in literature.[8]

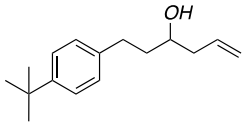


3c: pale yellow oil, 49% (0.1 mmol, 19 mg). The general procedure was applied using **1c** (0.2 mmol, 30 mg), and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The title compound was isolated by flash column chromatography (100% DCM). Spectroscopic data are in agreement with those reported in literature.[8] ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm): 7.23–7.08 (m, 2H), 7.05–6.91 (m, 2H), 5.90–5.74 (m, 1H), 5.22–5.07 (m, 2H), 3.74–3.61 (m, 1H), 2.83–2.72 (m, 1H), 2.73–2.61 (m, 1H), 2.37–2.28 (m, 1H), 2.24–2.12 (m, 1H), 1.80–1.71 (m, 2H), 1.62 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ(ppm): 161.40 (d, *J* = 243.2 Hz), 137.77 (d, *J* = 3.2 Hz), 134.64, 129.87 (d, *J* = 7.7 Hz, 2C), 118.58, 115.24 (d, *J* = 21.1 Hz, 2C), 69.88, 42.25, 38.68, 31.35. ^{19}F NMR (376.5 MHz, $CDCl_3$) δ(ppm): -116.62 (m).

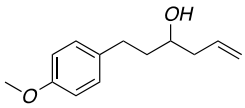


3d: pale yellow oil, 53% (0.10 mmol, 24 mg). The general procedure was applied using **1d** (0.2 mmol, 37 mg), and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The title compound was isolated by flash column chromatography (100% DCM). ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm): 7.86–7.72 (m, 3H), 7.65 (s, 1H), 7.44 (pd, *J* = 6.9, 1.4 Hz, 2H), 7.36 (dd, *J* = 8.4, 1.4 Hz, 1H), 5.83 (dddd, *J* = 14.5, 9.5, 7.9, 6.6 Hz, 1H), 5.24–5.09 (m, 2H), 3.79–3.62 (m, 1H), 3.04–2.94 (m, 1H), 2.92–2.82 (m, 1H), 2.42–2.28 (m, 1H), 2.28–2.14 (m, 1H), 1.97–1.80 (m, 2H), 1.65 (bs, 1H). ^{13}C NMR (100 MHz, $CDCl_3$)

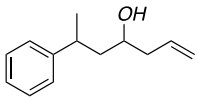
δ(ppm): 139.7, 134.7, 133.8, 132.1, 128.1, 127.7, 127.54, 127.46, 126.6, 126.1, 125.3, 118.5, 70.0, 42.3, 38.5, 32.3.



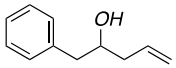
3e: pale yellow oil, 45% (0.09 mmol, 21 mg). The general procedure was applied using **1e** (0.2 mmol, 26 mg, 37 µL), and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The title compound was isolated by flash column chromatography (100% DCM). 1H NMR (400 MHz, CDCl3) δ 7.32 (d, J = 8.3 Hz, 2H), 7.16 (d, J = 8.1 Hz, 2H), 5.92–5.76 (m, 1H), 5.22–5.11 (m, 2H), 3.74–3.66 (m, 1H), 2.86–2.74 (m, 1H), 2.72–2.62 (m, 1H), 2.41–2.29 (m, 1H), 2.26–2.13 (m, 1H), 1.85–1.75 (m, 2H), 1.65 (s, 1H), 1.32 (s, 9H). 13C NMR (100 MHz, CDCl3) δ 148.8, 139.1, 134.8, 128.2 (2C), 125.4 (2C), 118.4, 70.2, 42.2, 38.6, 34.5, 31.6, 31.5 (3C).



3f: pale yellow oil, 58% (0.12 mmol, 24 mg). The general procedure was applied using **1q** (0.2 mmol, 33 mg) and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The reaction time was extended to 48h. The title compound was isolated by flash column chromatography (100% DCM). Spectroscopic data are in agreement with those reported in literature.[9]

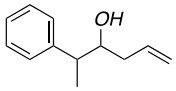


3g: pale yellow oil, 57% (0.11 mmol, 22 mg) as 1:1 *syn*:*anti* mixture of diastereoisomers. The general procedure was applied using **1g** (0.2 mmol, 29 mg), and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The title compound was isolated by flash column chromatography (100% DCM). Peak assignment wass attributed in analogy with the reported literature.[10] ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm): 7.35–7.12 (m, 5H), 5.89–5.64 (m, 1H), 5.19–5.02 (m, 2H), 3.74–3.62 (m, 1H, *anti*), 3.44–3.32 (m, 1H, *syn*), 3.07–2.99 (m, 1H, *syn*), 2.97–2.90 (m, 1H, *anti*), 2.39–1.99 (m, 2H), 1.86–1.62 (m, 3H), 1.28 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ(ppm): 147.6 and 146.8, 134.8, 128.7 and 128.6 (2C), 127.3 and 127.0 (2C), 123.3 and 123.2, 118.4 and 118.3, 69.0 and 68.5, 45.5 and 45.4, 42.7 and 42.3, 36.7 and 36.6, 23.4 and 22.0.

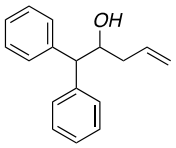


3h: pale yellow oil, 54% (0.11 mmol, 17 mg). The general procedure was applied using previously distilled **1h** (0.2 mmol, 24 mg, 22 µL), and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The reaction time

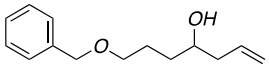
was extended to 48h. The title compound was isolated by flash column chromatography (100% DCM). Spectroscopic data are in agreement with those reported in literature.[9]



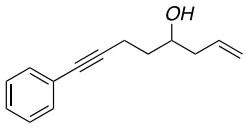
3i: yellow oil, 75% (0.15 mmol, 26 mg) as 2:1 *syn*:*anti* mixture of diastereoisomers. The general procedure was applied using **1i** (0.2 mmol, 26 µL) freshly distilled at 52°C/3 mbar and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The title compound was isolated by flash column chromatography (100% DCM). Spectroscopic data are in agreement with those reported in literature.[11]



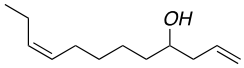
3j: yellow oil, 85% (0.17 mmol, 40 mg). The general procedure was applied using **1j** (0.2 mmol, 39 mg) and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The title compound was isolated by flash column chromatography (100% DCM). Spectroscopic data are in agreement with those reported in literature.[12]



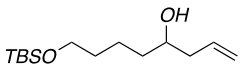
3k: yellow oil, 61% (0.12 mmol, 27 mg). The general procedure was applied using **1k** (0.2 mmol, 36 mg) and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The reaction time was extended to 48h. The title compound was isolated by flash column chromatography (100% DCM). Spectroscopic data are in agreement with those reported in literature.[7]



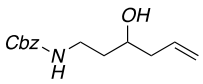
3l: pale yellow oil, 37% (0.07 mmol, 15 mg). The general procedure was applied using **1l** (0.2 mmol, 32 mg), and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The title compound was isolated by flash column chromatography (100% DCM). ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm): 7.44–7.35 (m, 2H), 7.31–7.25 (m, 3H), 5.86 (dddd, *J* = 11.1, 9.3, 7.7, 6.6 Hz, 1H), 5.22–5.12 (m, 2H), 3.88 (tt, *J* = 8.3, 4.2 Hz, 1H), 2.57 (t, *J* = 7.1 Hz, 2H), 2.36 (dddt, *J* = 7.6, 5.8, 4.5, 1.3 Hz, 1H), 2.28–2.18 (m, 1H), 1.90–1.65 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ(ppm): 134.6, 131.7 (2C), 128.3 (2C), 127.8, 123.9, 118.5, 89.7, 81.2, 69.9, 42.0, 35.6, 16.2.



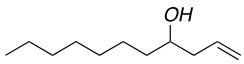
3m: pale yellow oil, 72% (0.14 mmol, 26 mg). The general procedure was applied using **1m** (0.2 mmol, 28 mg, 33 µL), and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The reaction time was extended to 48h. The title compound was isolated by flash column chromatography (100% DCM). Spectroscopic data are in agreement with those reported in literature.[7]



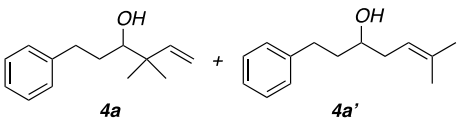
3n: yellow oil, 71% (0.14 mmol, 37 mg). The general procedure was applied using **1n** (0.2 mmol, 43 mg) and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL). The title compound was isolated by flash column chromatography (100% DCM). Spectroscopic data are in agreement with those reported in literature.[13]



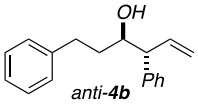
3o: yellow oil, 31% (0.06 mmol, 15 mg). The general procedure was applied using **1o** (0.2 mmol, 41 mg) and **2a** (.6 mmol, 3 equiv., 73 mg, 52 µL). The title compound was isolated by flash column chromatography (cyclohexane/ethyl acetate). Spectroscopic data are in agreement with those reported in literature.[11]



3p: brown oil, 67% (0.13 mmol, 23 mg). The general procedure was applied using **1p** (0.2 mmol, 32 µL) freshly distilled at 59°C/21 mbar and **2a** (0.6 mmol, 3 equiv., 73 mg, 52 µL) and performed in duplicate. The title compound was isolated by flash column chromatography (100% DCM). Spectroscopic data are in agreement with those reported in literature.[11]



4a-4a': brown oil, 82% (0.16 mmol, 33 mg) as a mixture of regioisomers **4a:4a'** >20:1. The general procedure was applied using **1a** (0.2 mmol, 26 mg, 24 µL) and **2b** (95%, 0.6 mmol, 3 equiv., 72 µL). Spectroscopic data are in agreement with those reported in literature.[14]

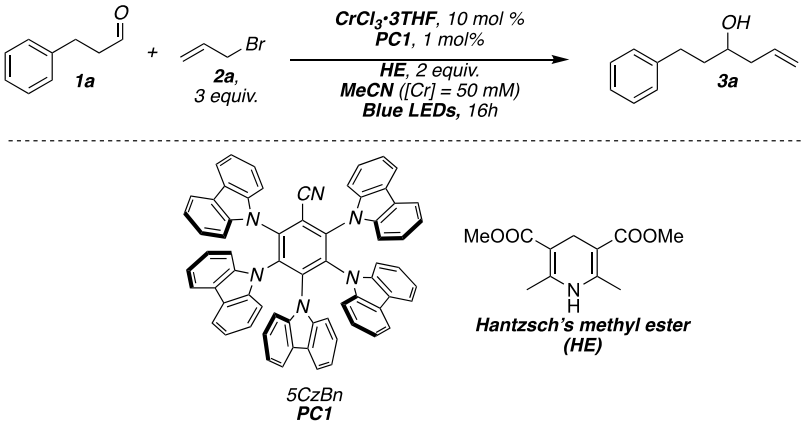


anti-**4b**

anti-**4b**: yellowish oil, 35% (0.07 mmol, 18 mg). The general procedure was applied using **1a** (0.2 mmol, 26 mg, 24 µL) and **2c** (0.6 mmol, 3 equiv., 118 mg, 89 µL). Spectroscopic data are in agreement with those reported in literature.[14]

Screening of reaction conditions

Table S1



Entry[a]	Deviation from standard conditions	Yields, %[b]
1	None	>99(80)[c]
2	$Cr(acac)_3$ instead of $CrCl_3$•3THF	no reaction
3	$CrBr_3$•$6H_2O$ instead of $CrCl_3$•3THF	traces
4	$Al(OTf)_3$ instead of $CrCl_3$•3THF	no reaction
5[d]	2–Naphthaldehyde instead of **1a**	32[c]
6[e]	1,3-Dimethyl-2-phenyl-2,3-dihydro-1H-benzimidazole (BIH) 2 equiv. instead of **HE**	no reaction
7[e]	$ZrCp_2Cl_2$ 2 equiv. instead of **HE**	22
8[e]	1-(trimethylsilyl)collidinium chloride 1.1 equiv. instead of **HE**	no reaction
9[e]	No $CrCl_3$•3THF	21[c]
10	THF instead of MeCN	48

[a] Reactions performed on 0.1 mmol scale. [b] Determined by ^{1}H-NMR analysis. [c] Reaction performed on 0.2 mmol scale; in parenthesis yield after chromatographic purification. [d] By ^{1}H-NMR analysis of the reaction crude the formation of pinacol coupling product was observed as major outcome (*meso*: *d/l* 1:1). [e] Reaction performed employing 2–naphthaldehyde instead of **1a**.

Photophysical Studies

Among all the reagents present in the reaction mixture, only $CrCl_3(THF)_3$ and Hantzsch ester are able to quench **PC1** emissive excited state in degassed conditions at room temperature, as reported below.

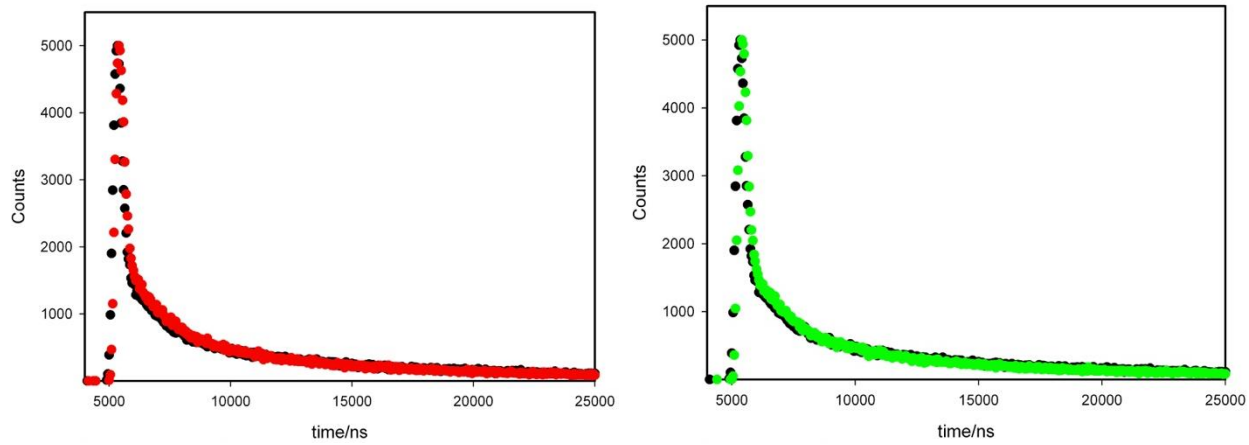


Figure S2. Emission intensity decays of **PC1** ($4x10^{-5}$ M) in acetonitrile solution in absence (left and right, black dots) and in presence of allyl bromide 0.15 M (left, red dots) or hydrocinnamaldehyde 0.05 M (right, green dots), upon excitation at 405 nm.

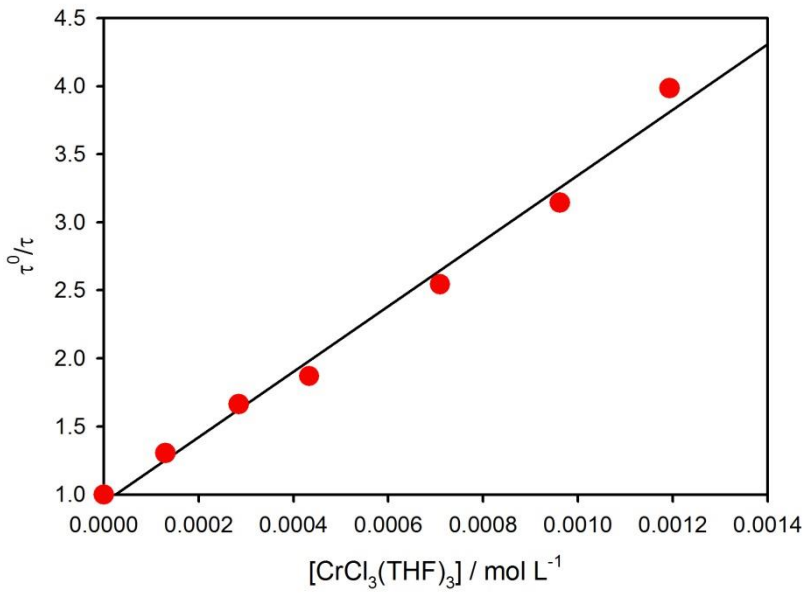


Figure S3. Lifetime of **PC1** emissive excited state in degassed acetonitrile solution in the absence (τ^0) and in the presence (τ) of increasing amount of $CrCl_3(THF)_3$. The slope represents the Stern-Volmer constant, i.e. the product of the quenching constant and τ^0.

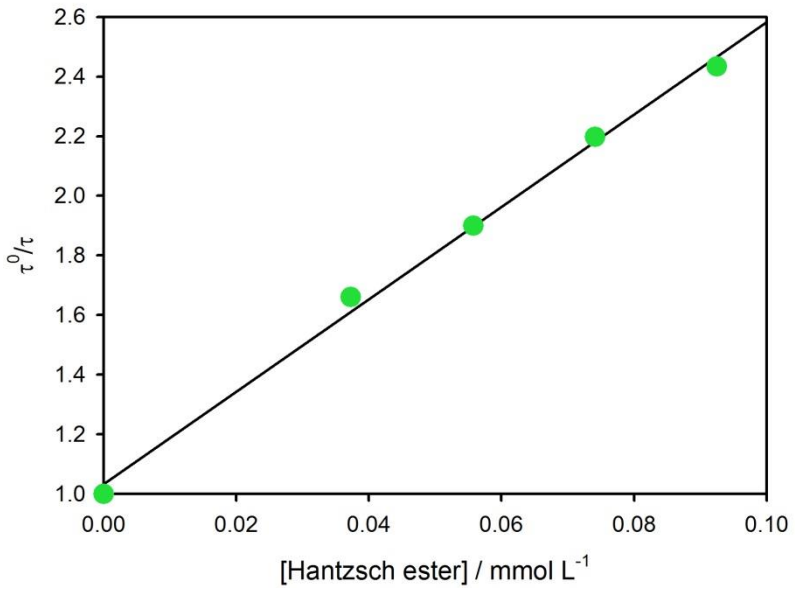


Figure S4. Lifetime of **PC1** fluorescent excited state in degassed acetonitrile solution in the absence (τ^0) and in the presence (τ) of increasing amount of Hantzsch ester. The slope represents the Stern-Volmer constant, i.e. the product of the quenching constant and τ^0.

The Stern-Volmer plots show a linear correlation between the ratio τ^0/τ and the quencher concentration, as expected for a dynamic quenching process according to the Stern-Volmer equation:

(1) $\tau^0/\tau = 1 + K_{SV}[Q] = 1 + k_q\, \tau^0[Q]$

where τ^0 and τ are the lifetimes in the absence and in the presence of the quencher Q, respectively, K_{SV} is the Stern-Volmer constant and k_q is the quenching constant.

The analysis of the plots reported above yields the following quenching constants:

(2) $k_q = 3.5 \times 10^8\ M^{-1}s^{-1}$ for $CrCl_3(THF)_3$

(3) $k_q = 3.4 \times 10^9\ M^{-1}s^{-1}$ for Hantzsch ester

Quenching efficiency of species η_q^i was calculated according to the following formula:

$$\eta_q^i = \frac{k_q^i \cdot [Q]^i}{k_{nr} + k_r + \sum_0^n k_q^n \cdot [Q]^n} \cdot 100$$

Quencher	k_q (M^{-1} s^{-1})	[Q] (M)	η_q^i (%)
Allyl bromide	not measurable	0.15	/
Hydrocinnamaldehyde	not measurable	0.05	/
$CrCl_3(THF)_3$	3.5 x 10^8	0.005	0.6
Hantzsch ester	3.4 x 10^9	0.1	99.4

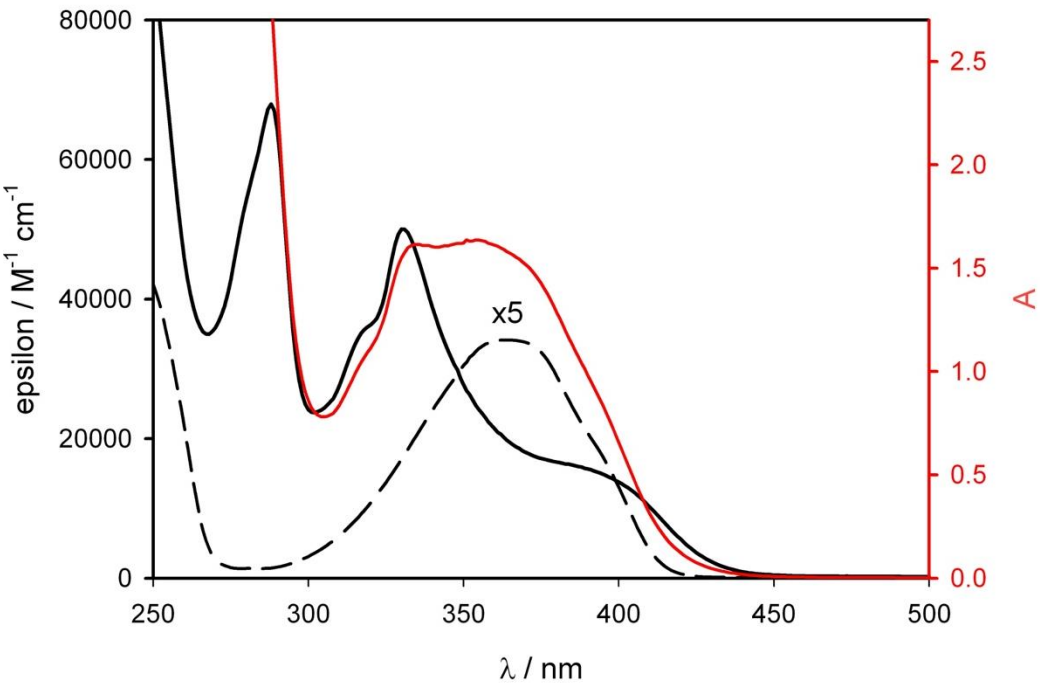
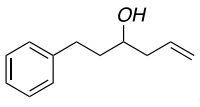


Figure S5. Absorption spectrum of **PC1** in acetonitrile (black solid line), Hantzsch ester in acetonitrile (black dashed line) and reaction mixture containing hydrocinnamaldehyde 0.05 M, allylbromide 0.15 M, Hantzsch ester 0.10 M, **PC1** 2.5 mM (5%) and $CrCl_3(THF)_3$ 5 mM (10%) in acetonitrile after 24 hours irradiation (red solid line). It was necessary to dilute the reaction mixture 50 times in order to reach the proper concentration range for the absorption.

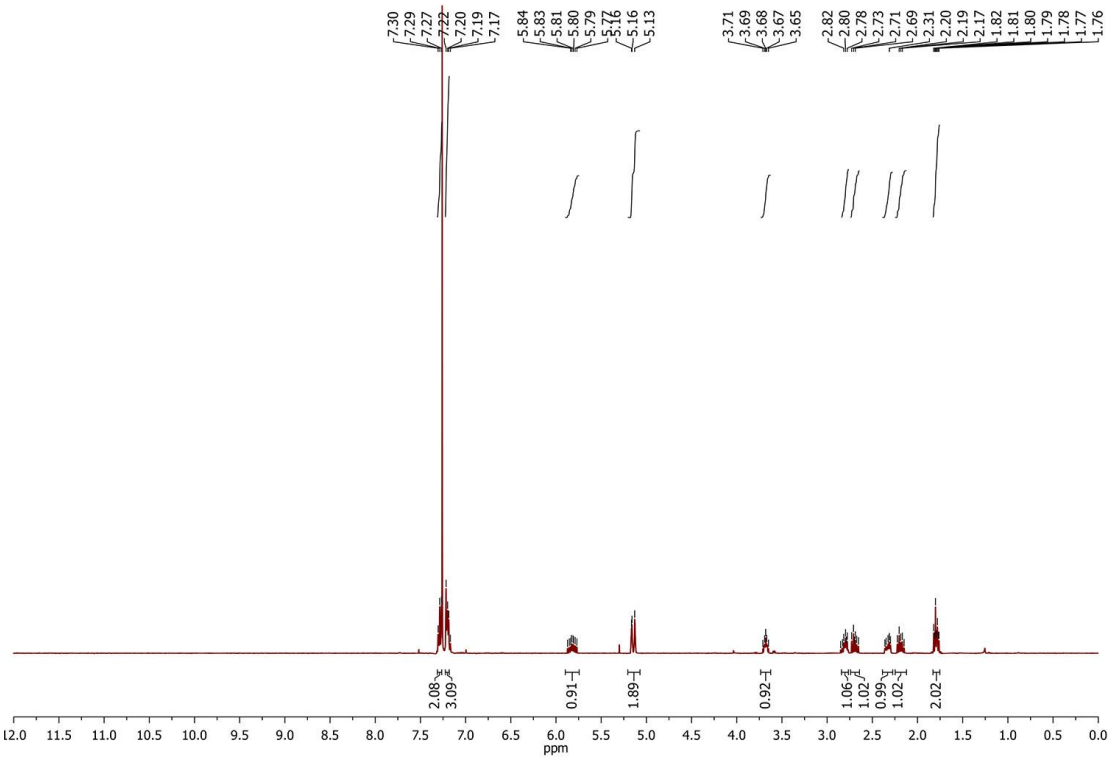
In order to estimate the amount of photosensitizer at the end of the irradiation, the absorption spectrum of Hantzsch ester has been mathematical subtracted from the reaction mixture one (it is the only species in the reaction mixture that absorbs in the same spectral region of the **PC1**); when the spectrum so obtained has the typical feature of **PC1** one, we were able to estimate the concentration of the photosensitizer at the end of the irradiation, i.e. 75% of the initial amount can be recovered.

Copies of NMR Spectra

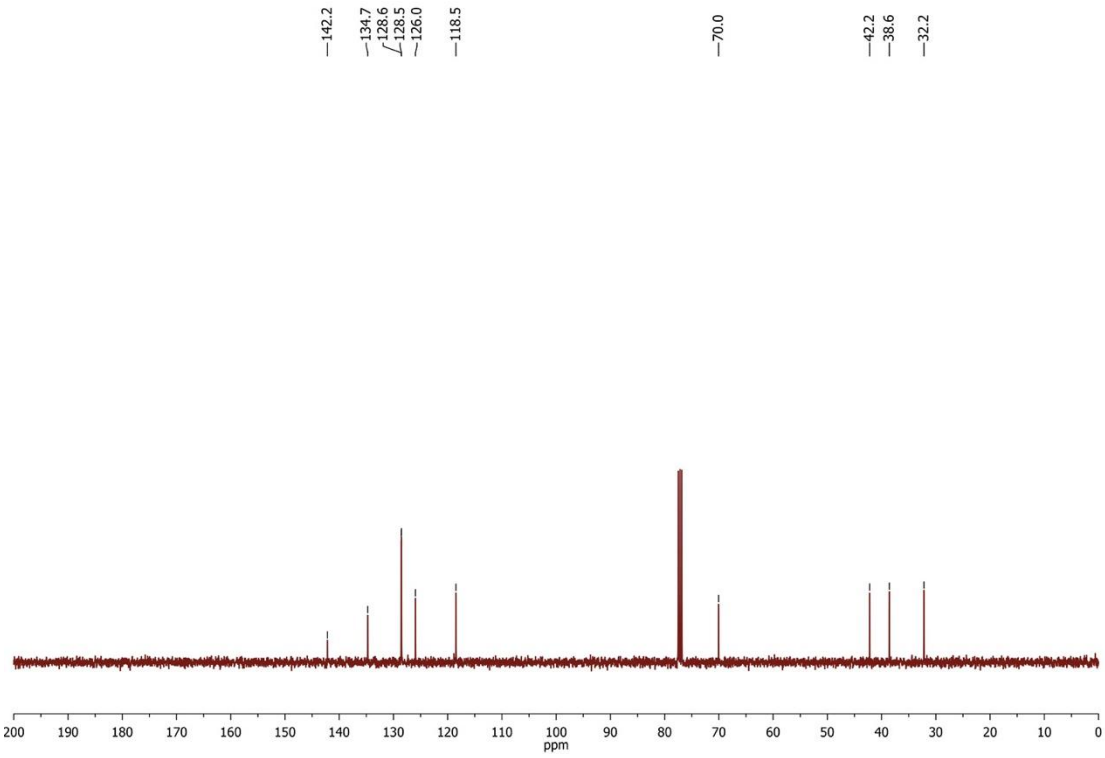


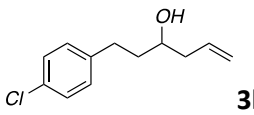
3a

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



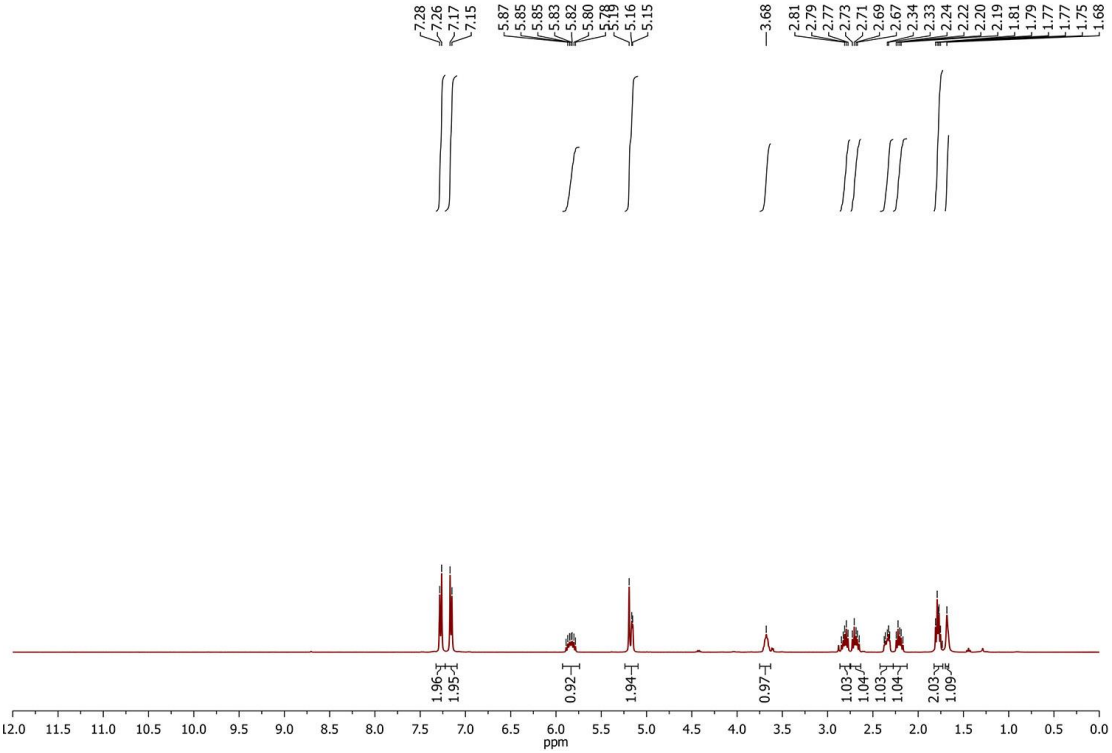
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)



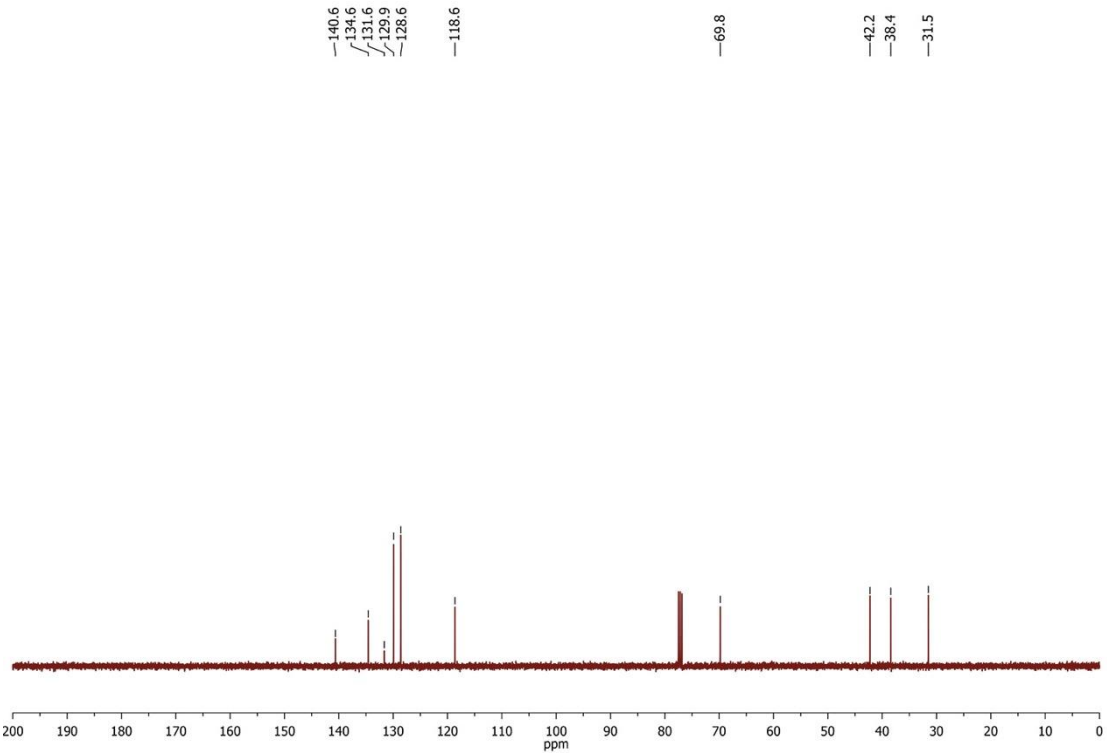


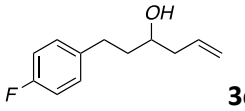
3b

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



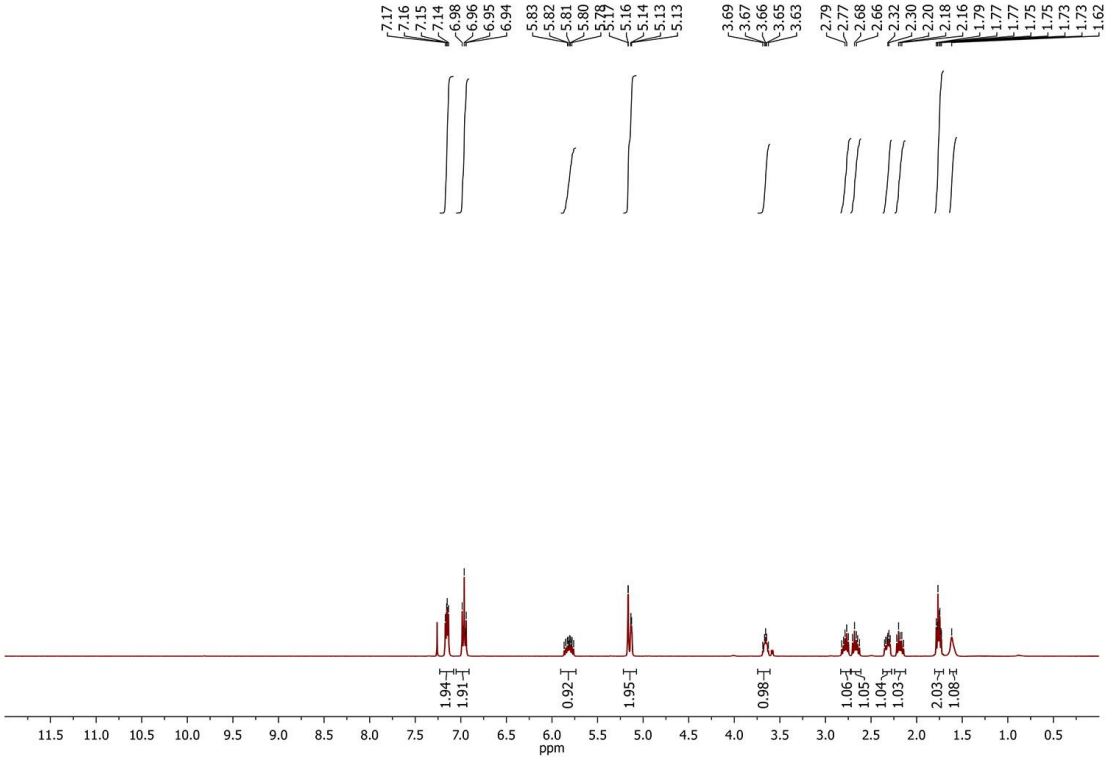
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)



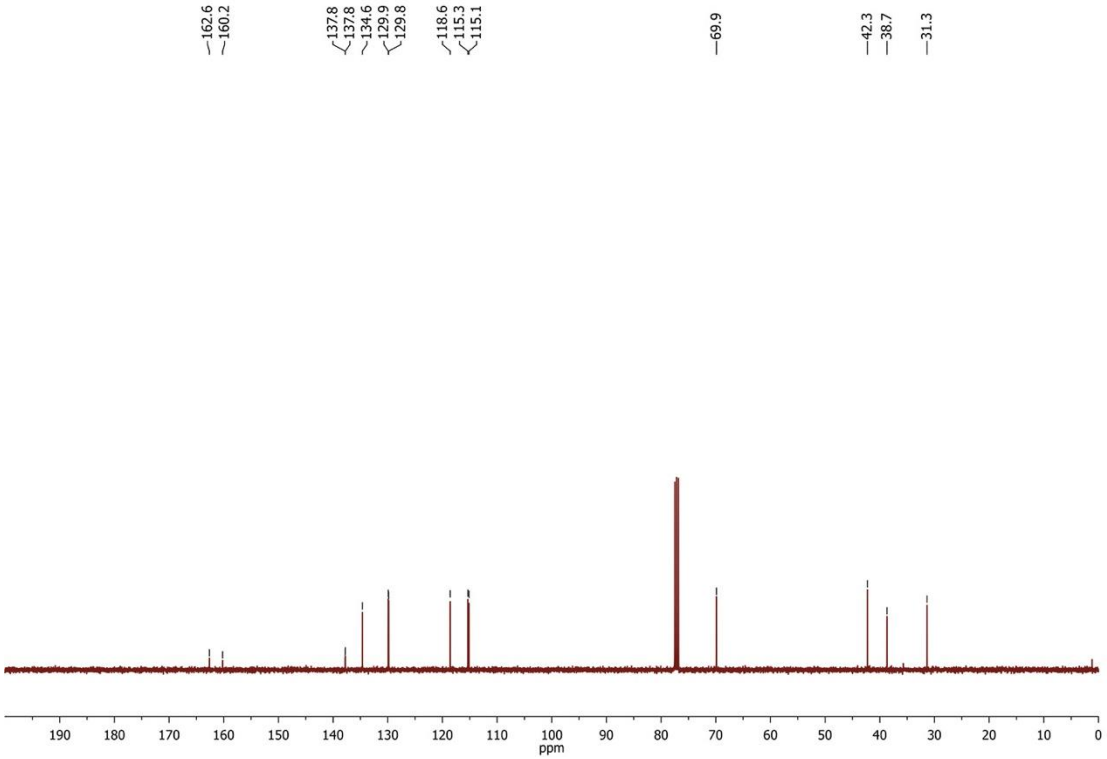


3c

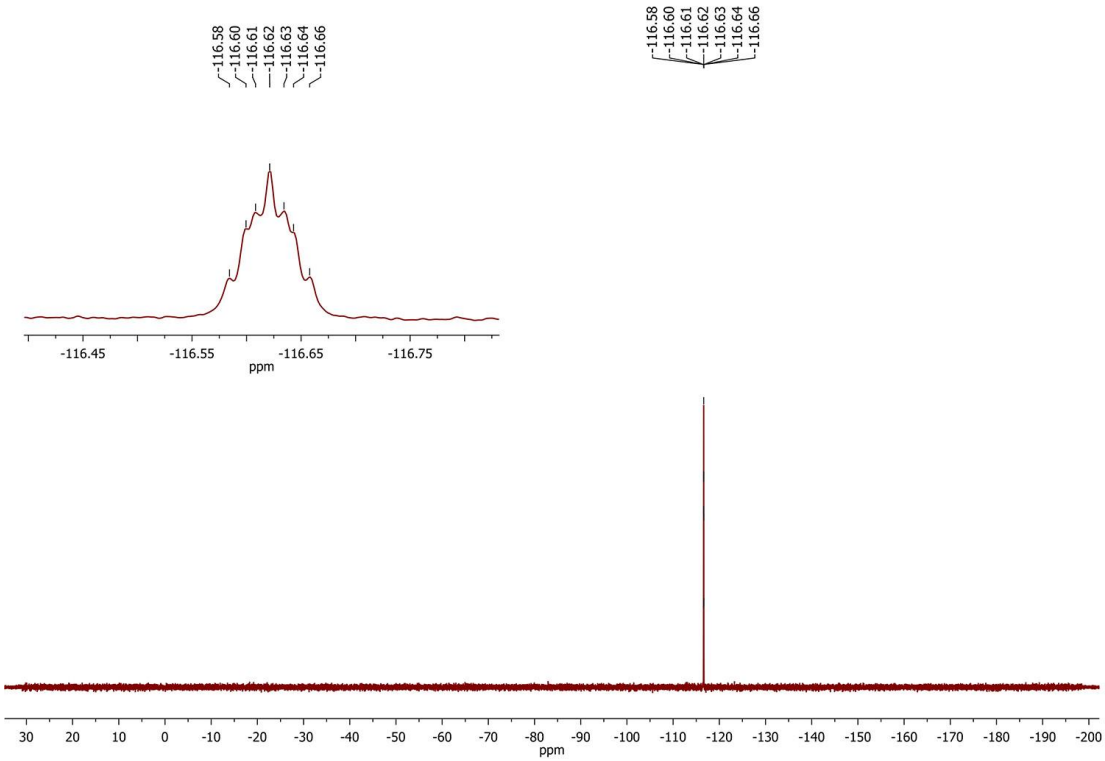
^{1}H NMR (400 MHz, $CDCl_3$)

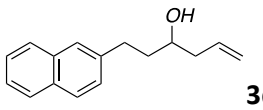


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)



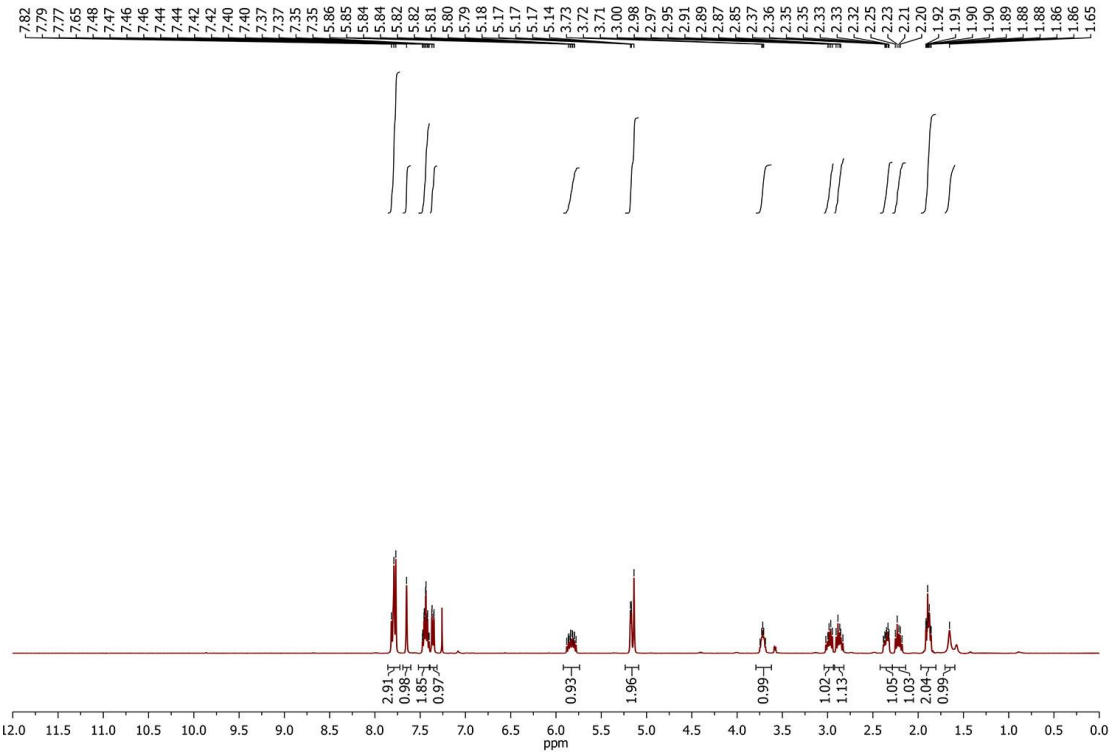
^{19}F NMR (376.5 MHz, $CDCl_3$)



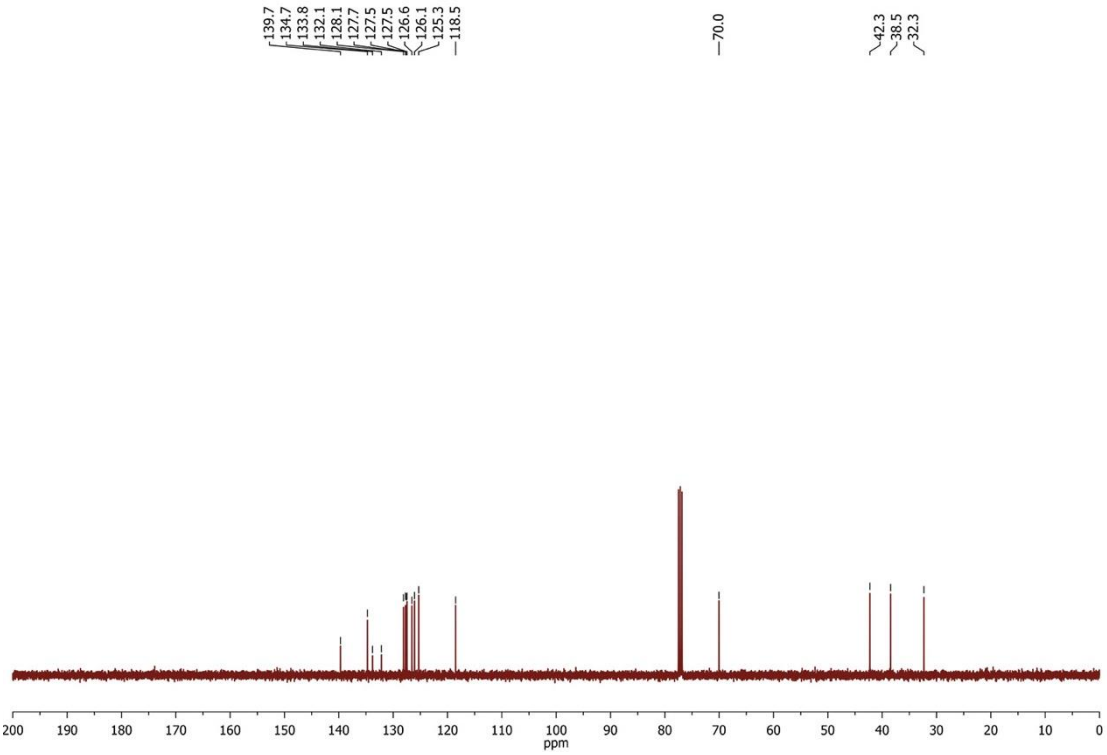


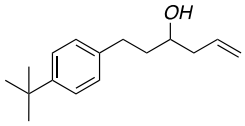
3d

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



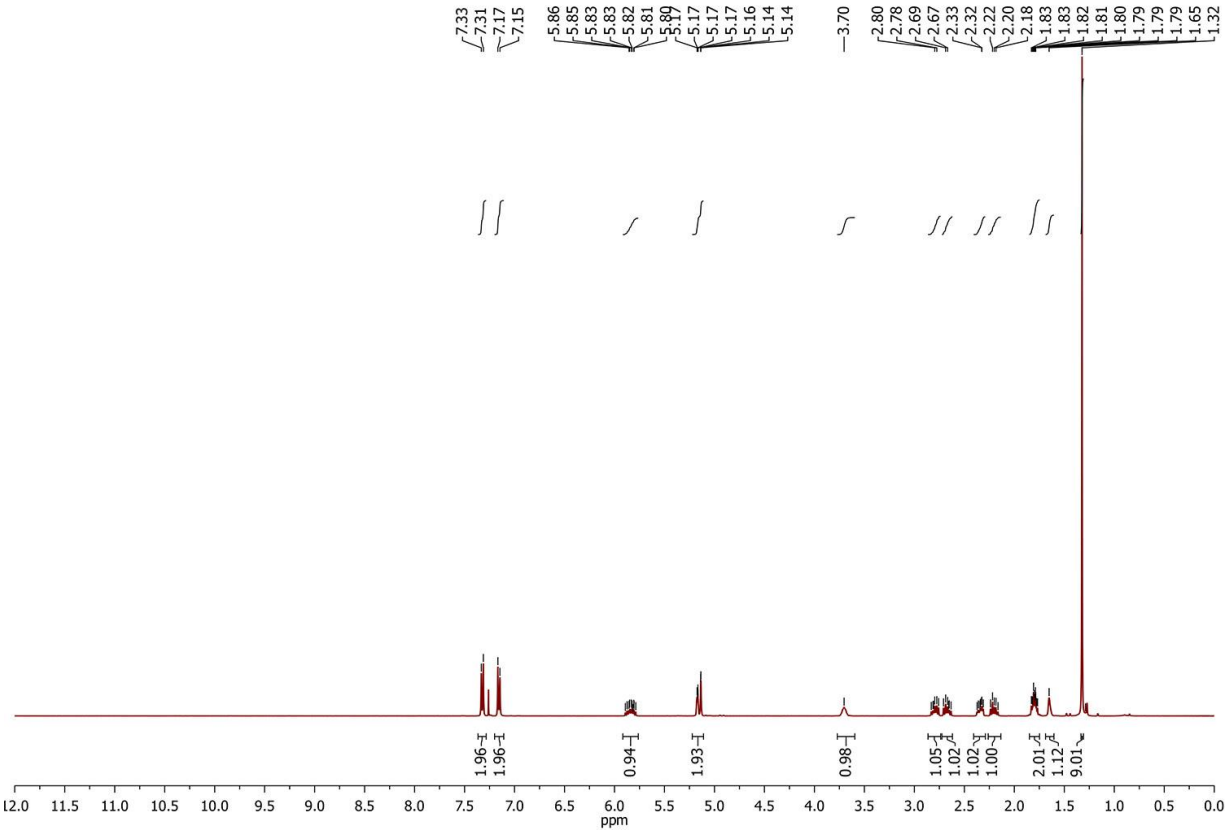
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)



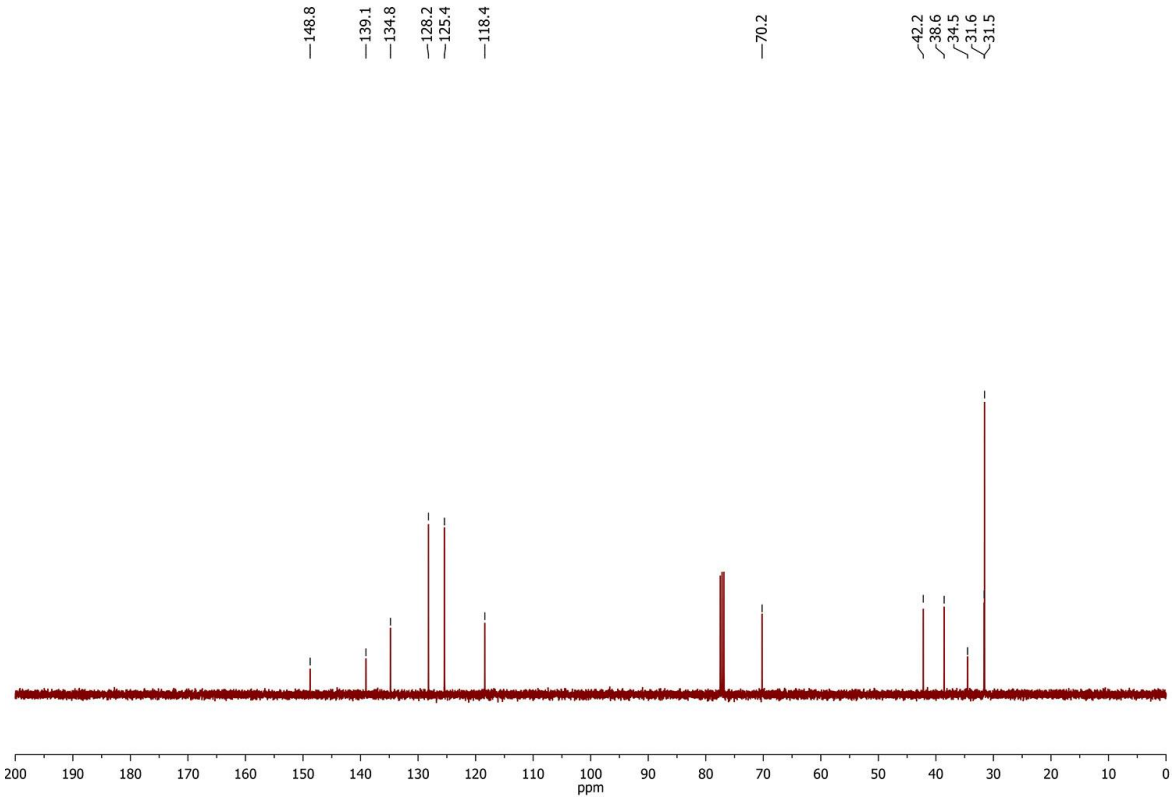


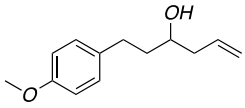
3e

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



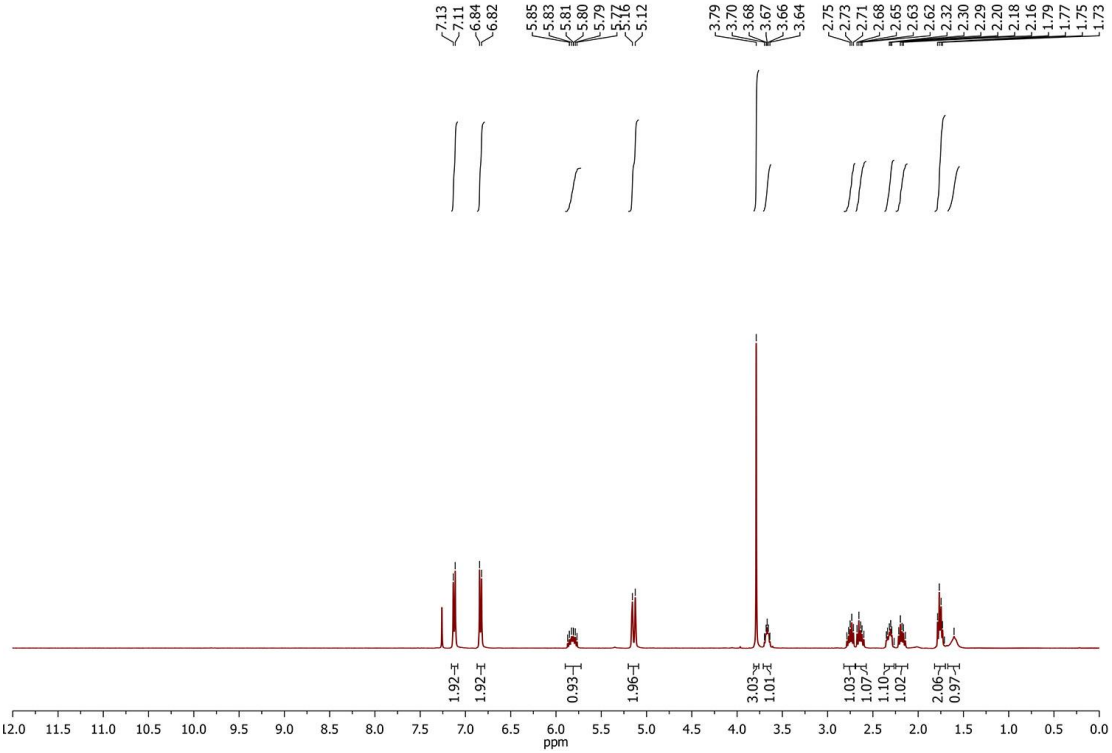
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)





3f

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



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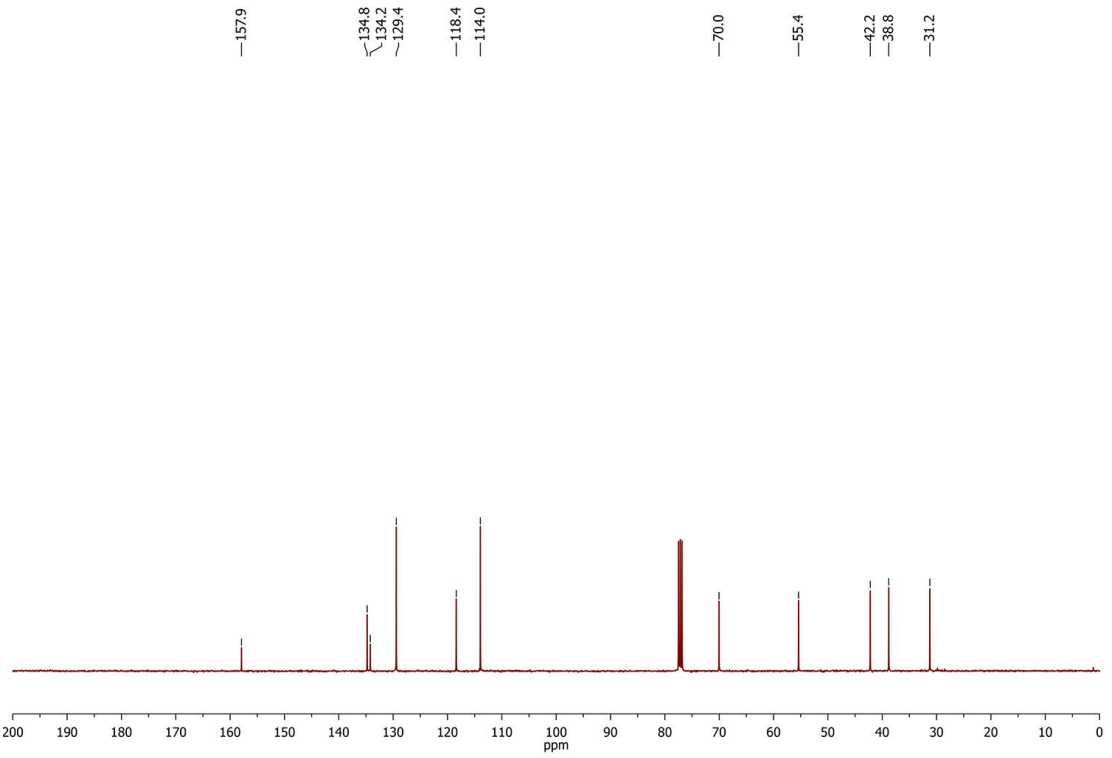
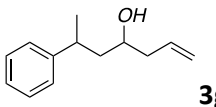
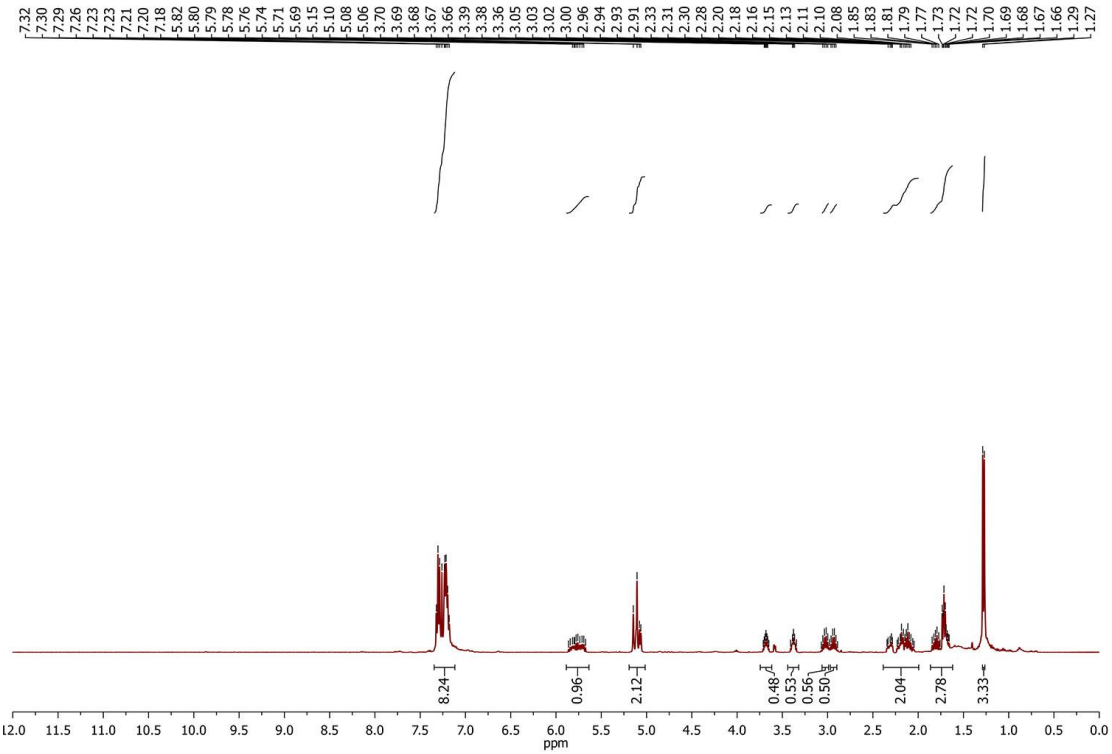


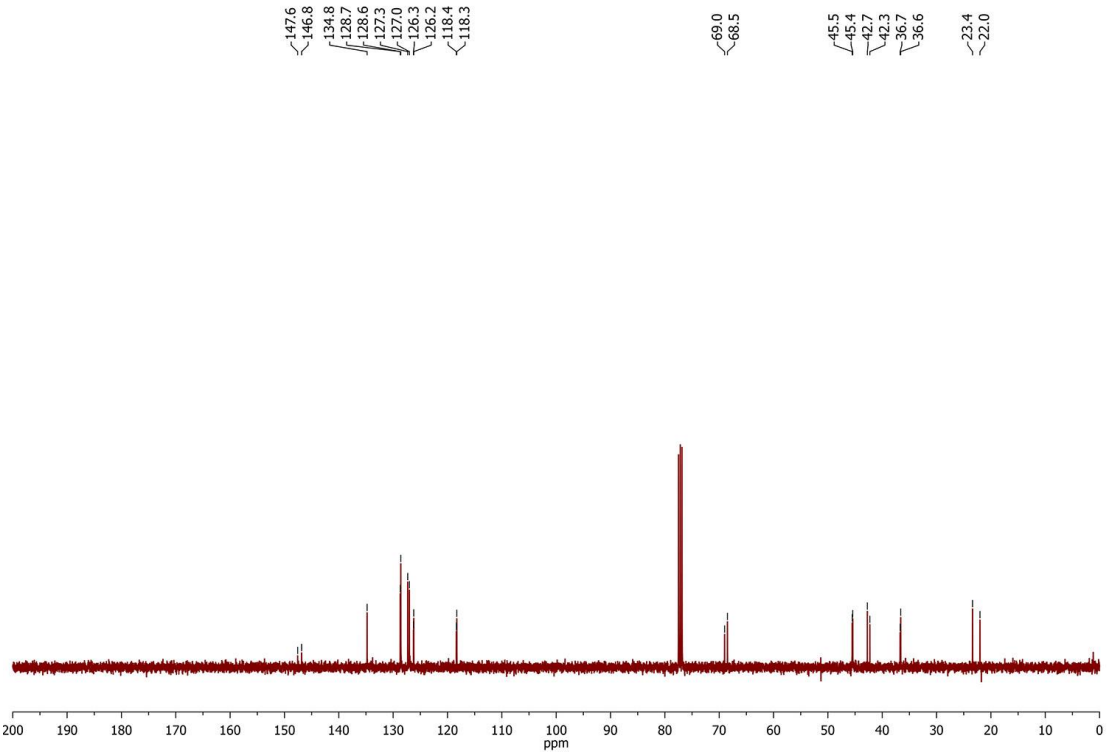


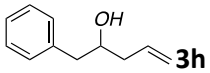
3g

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



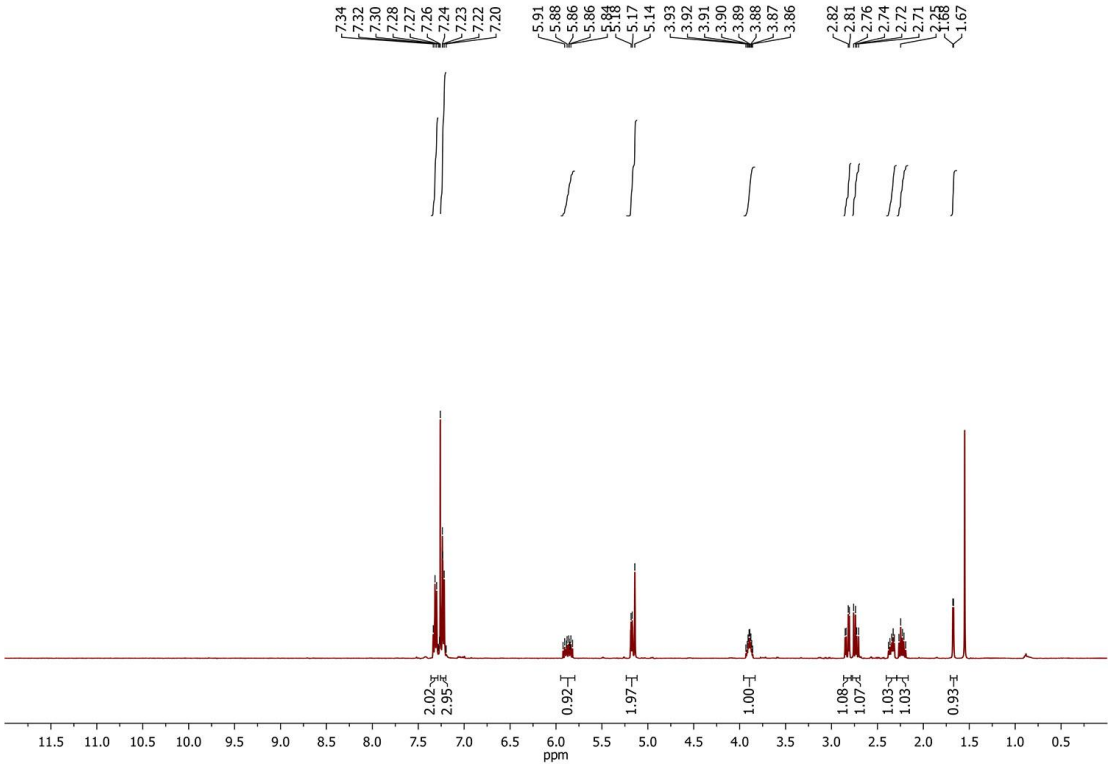
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)



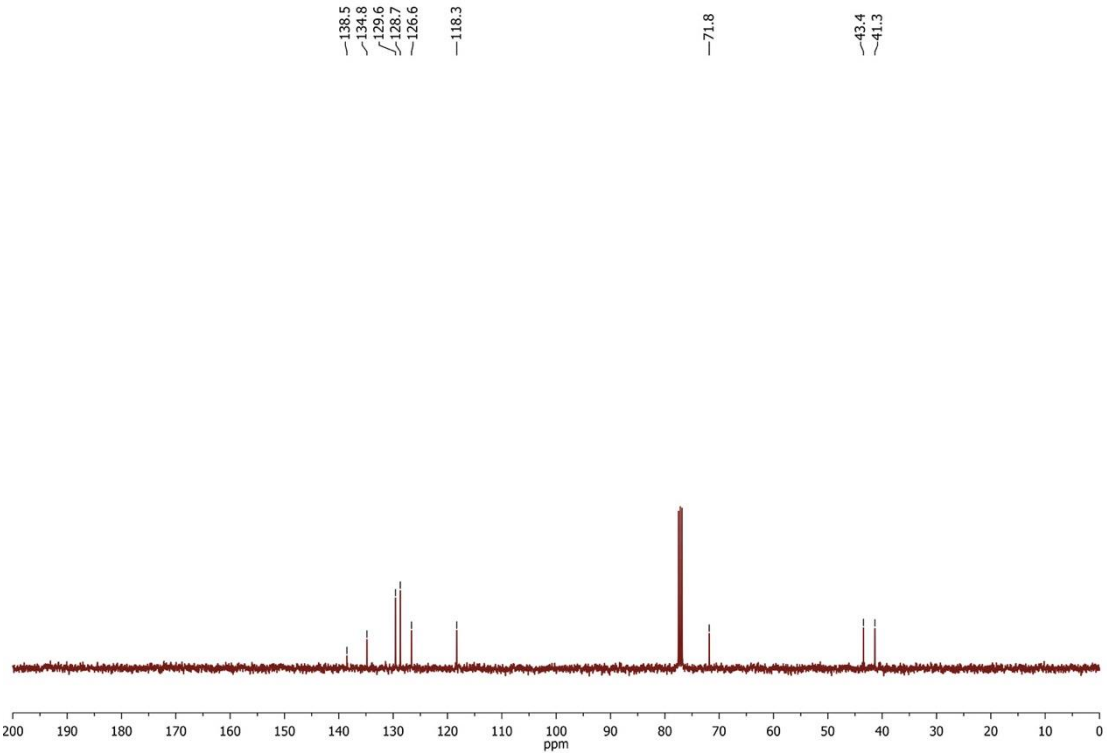


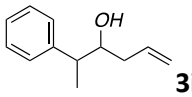
3h

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



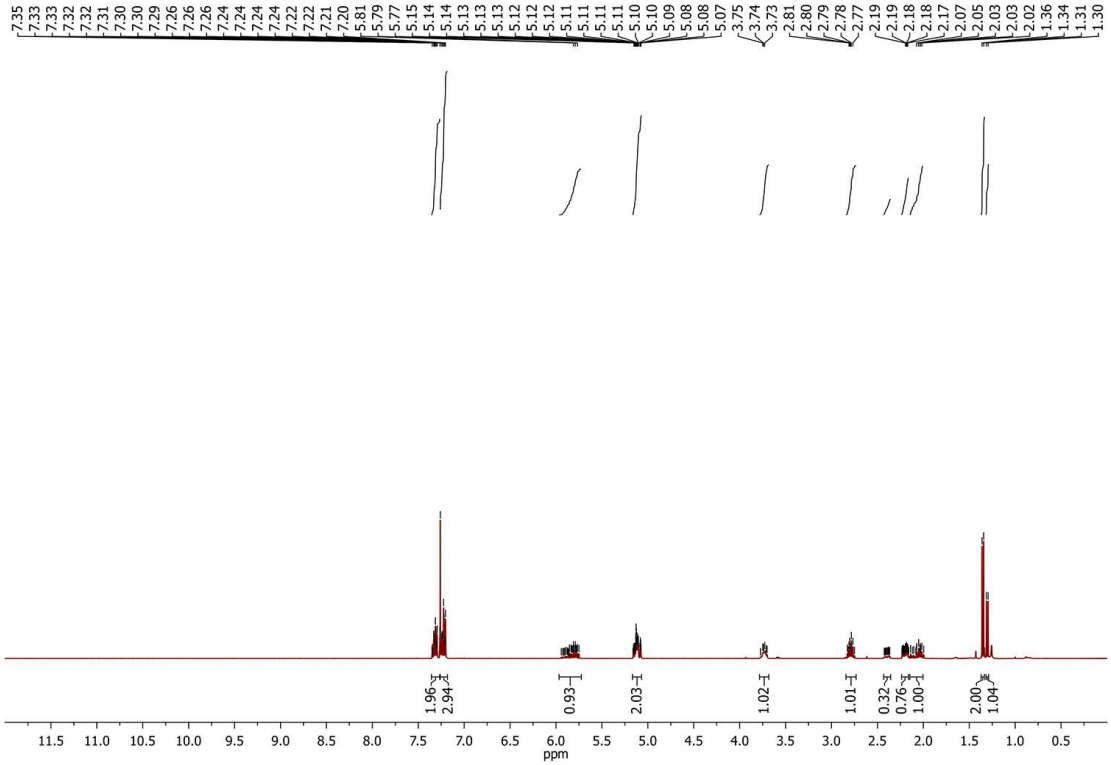
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)



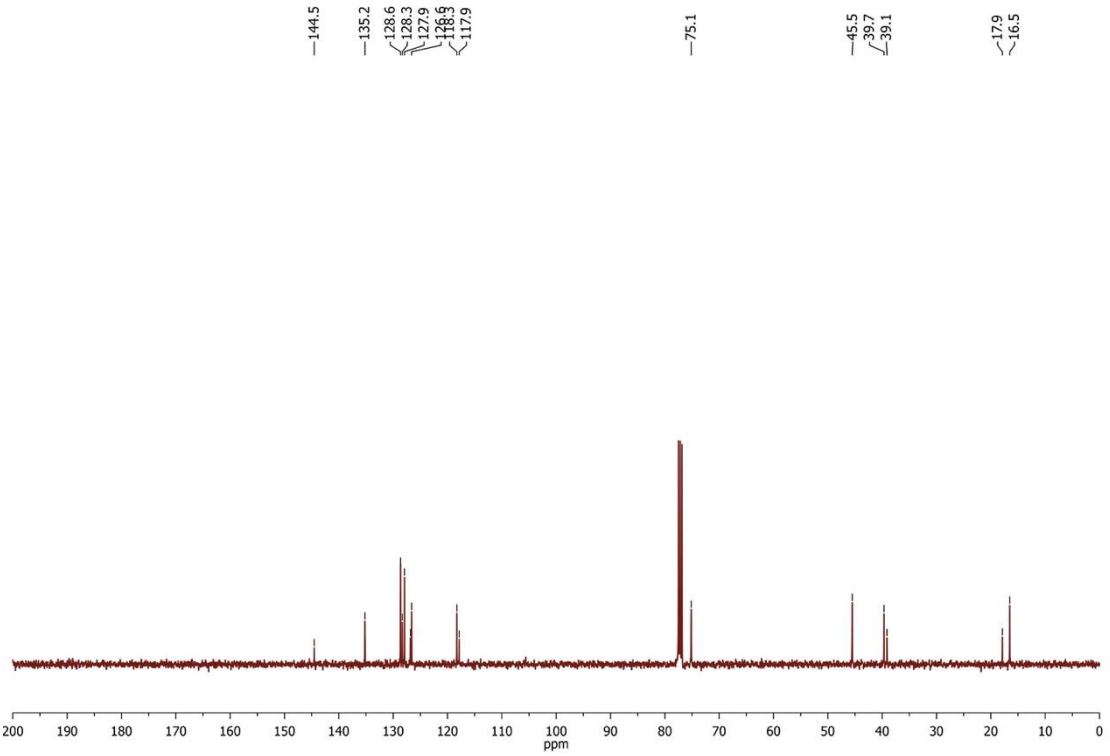


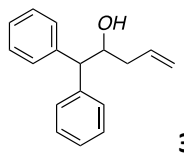
3i

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



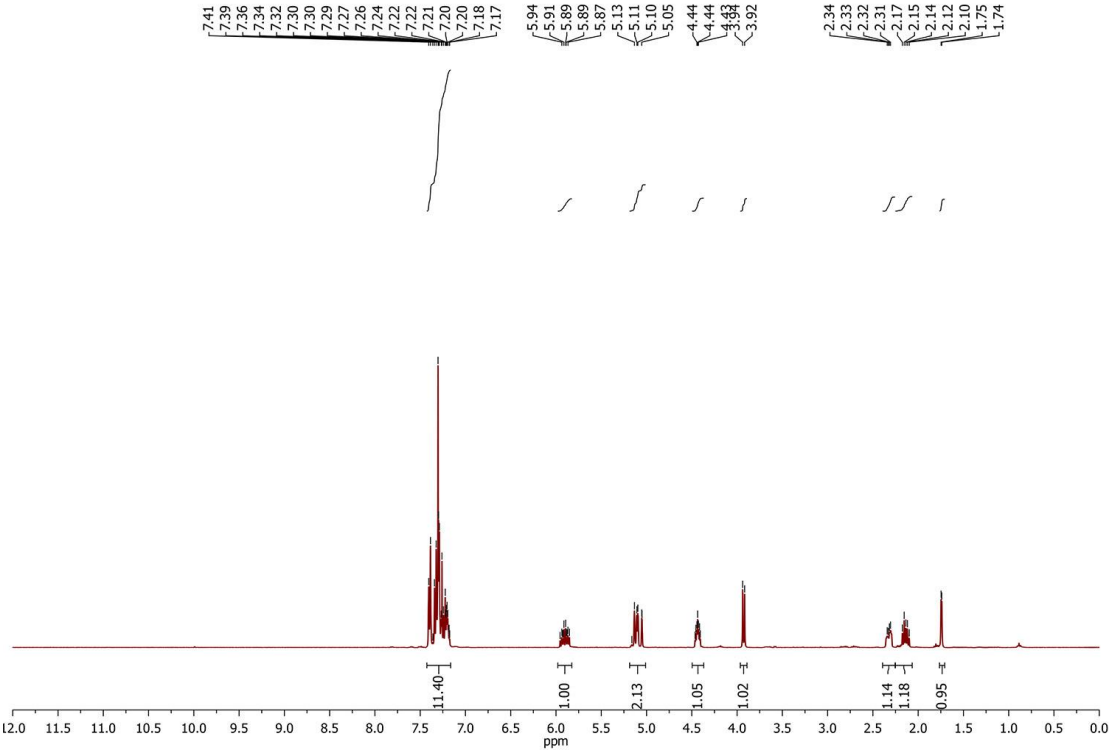
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)





3j

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



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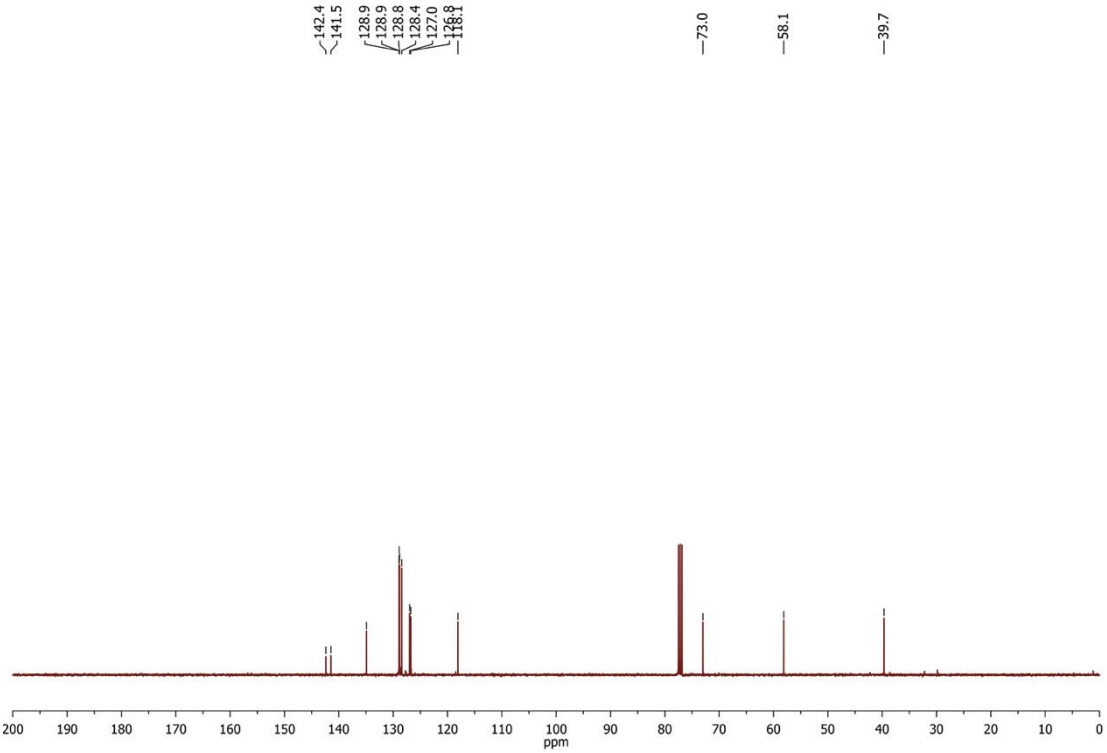

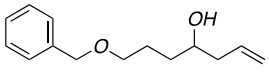

3k

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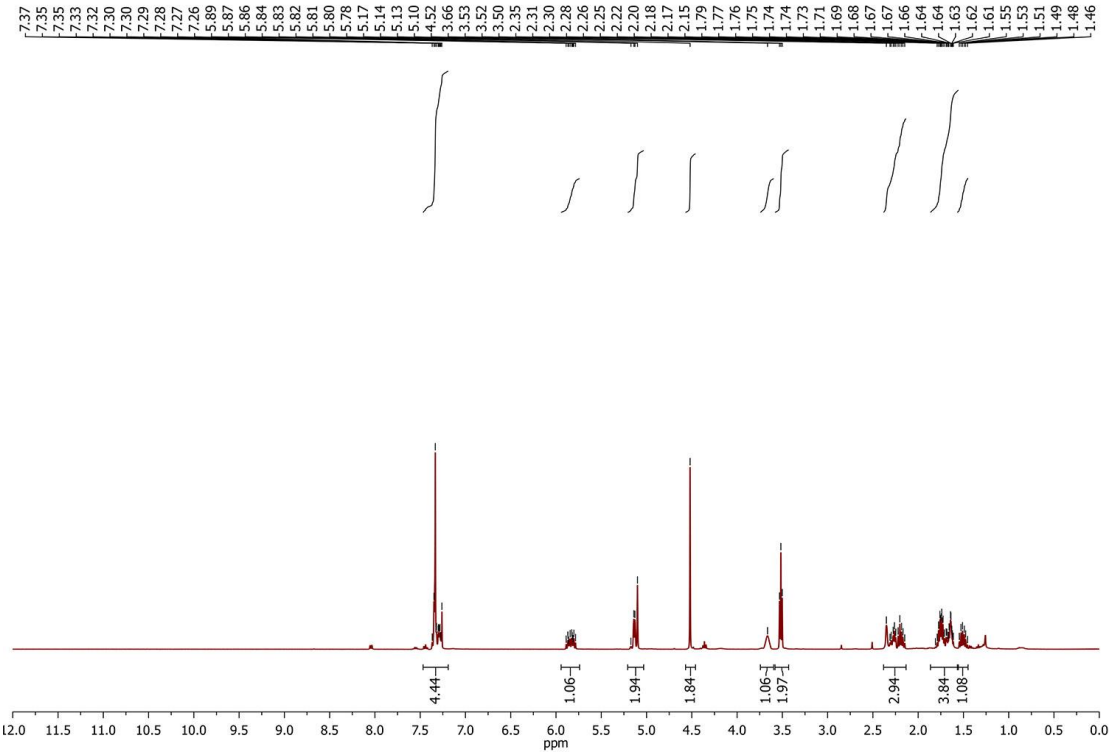
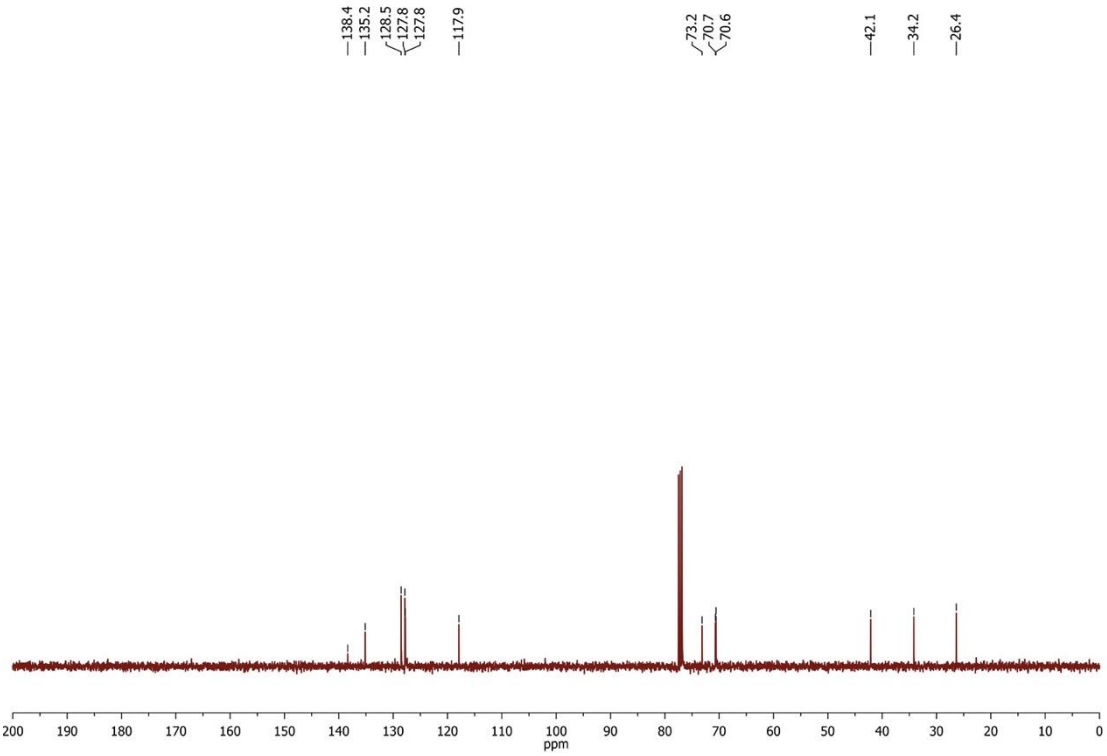
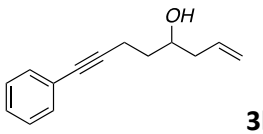


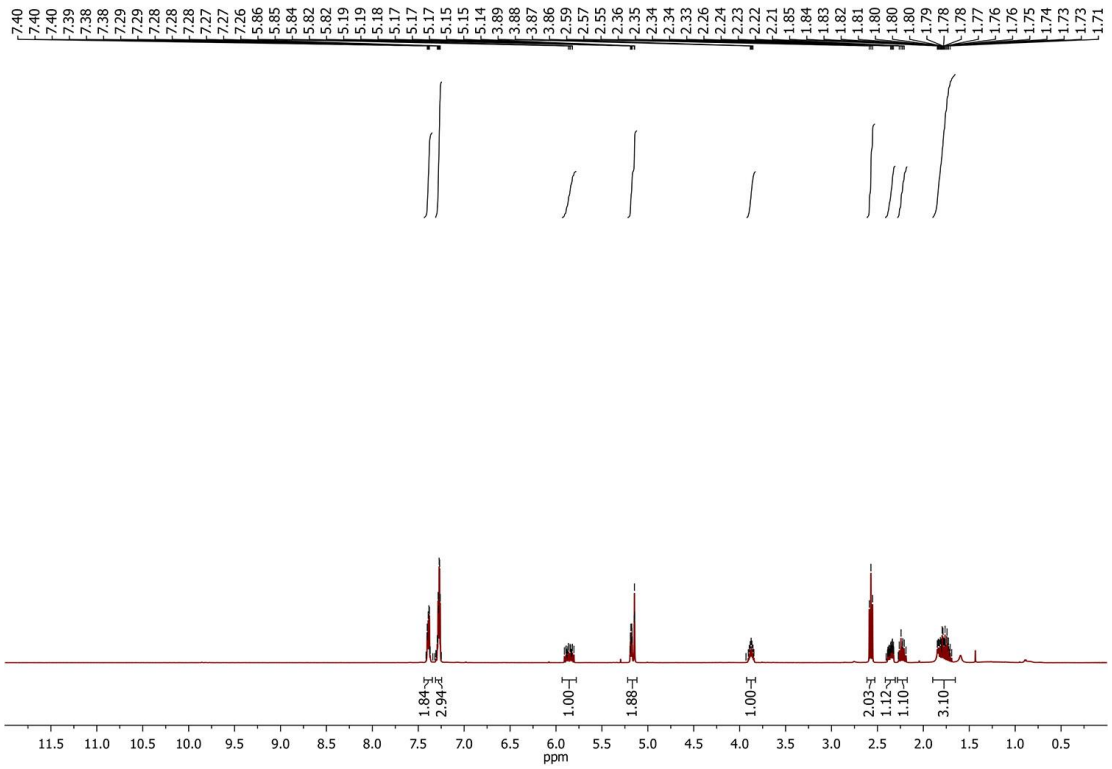
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)



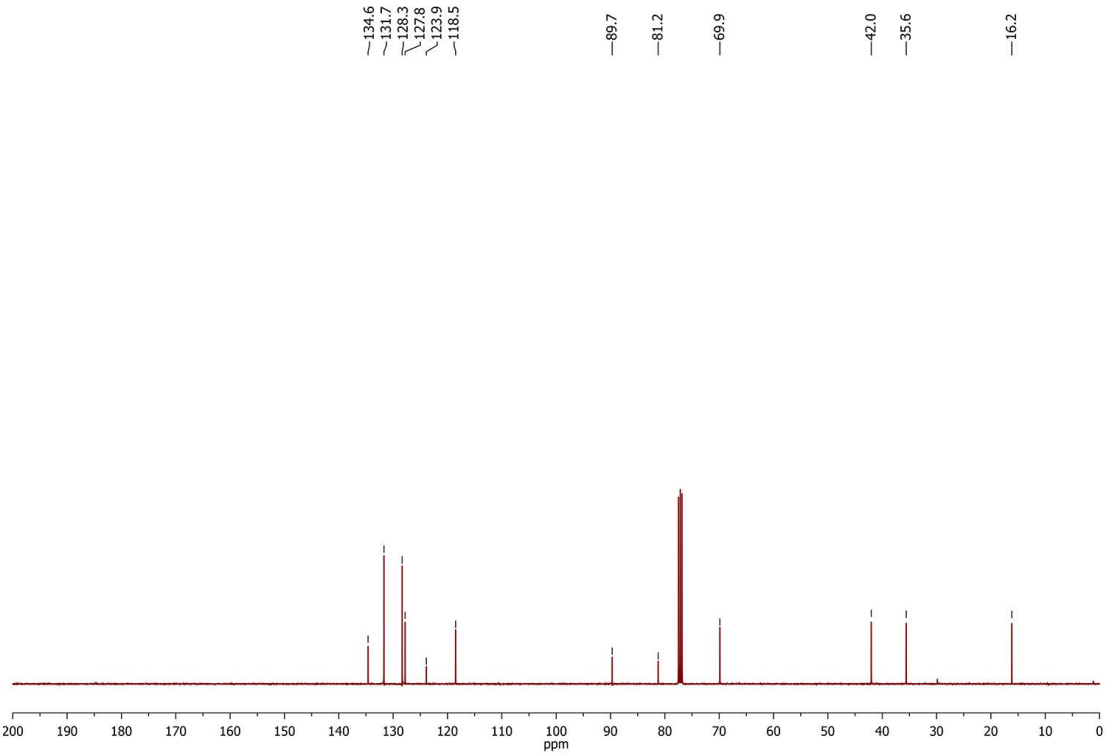


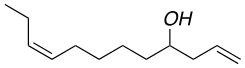
3l

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



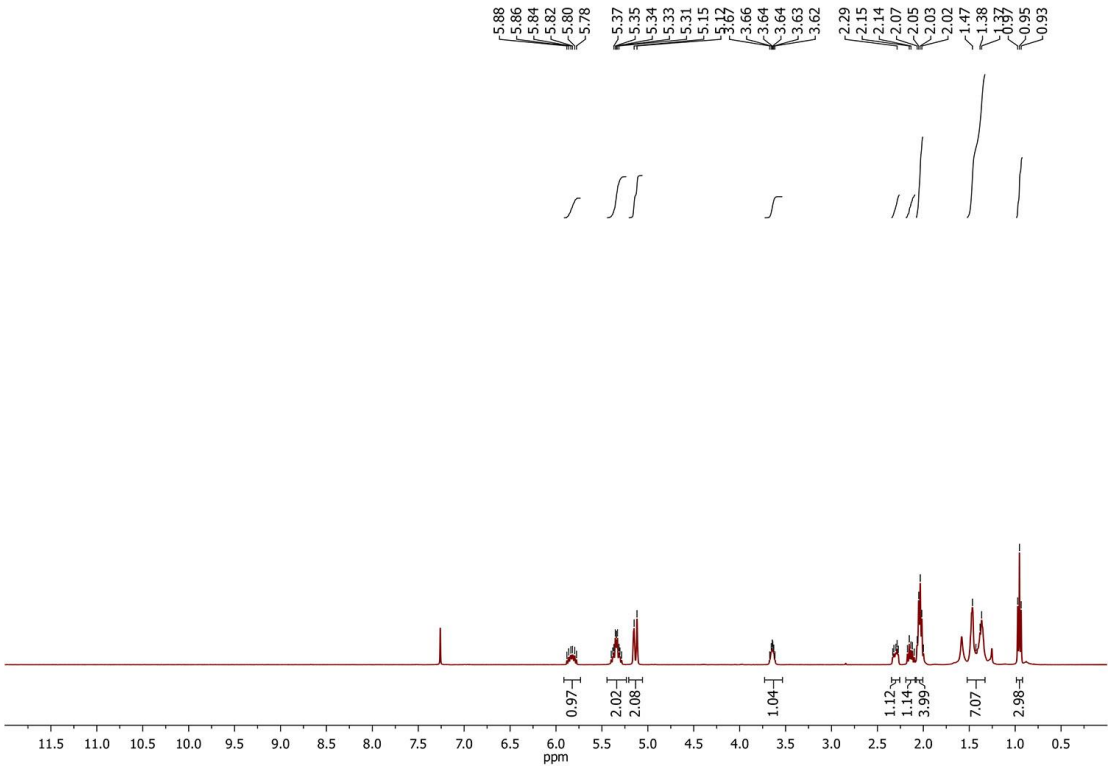
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)



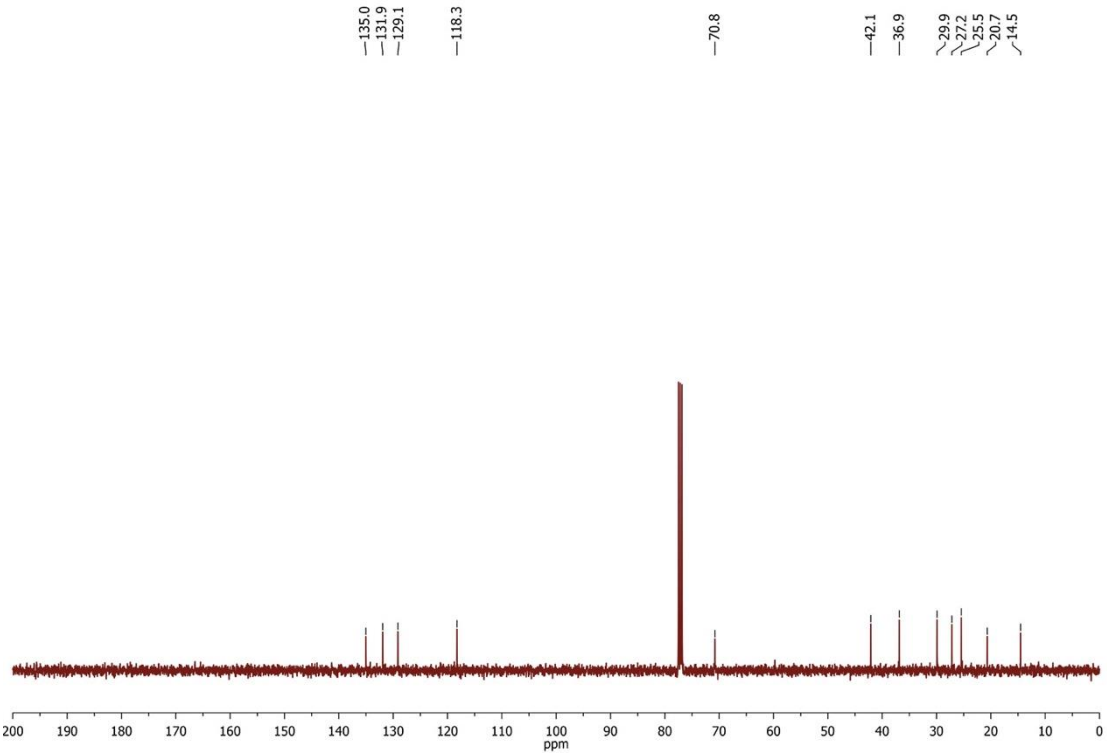


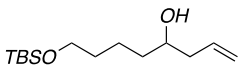
3m

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



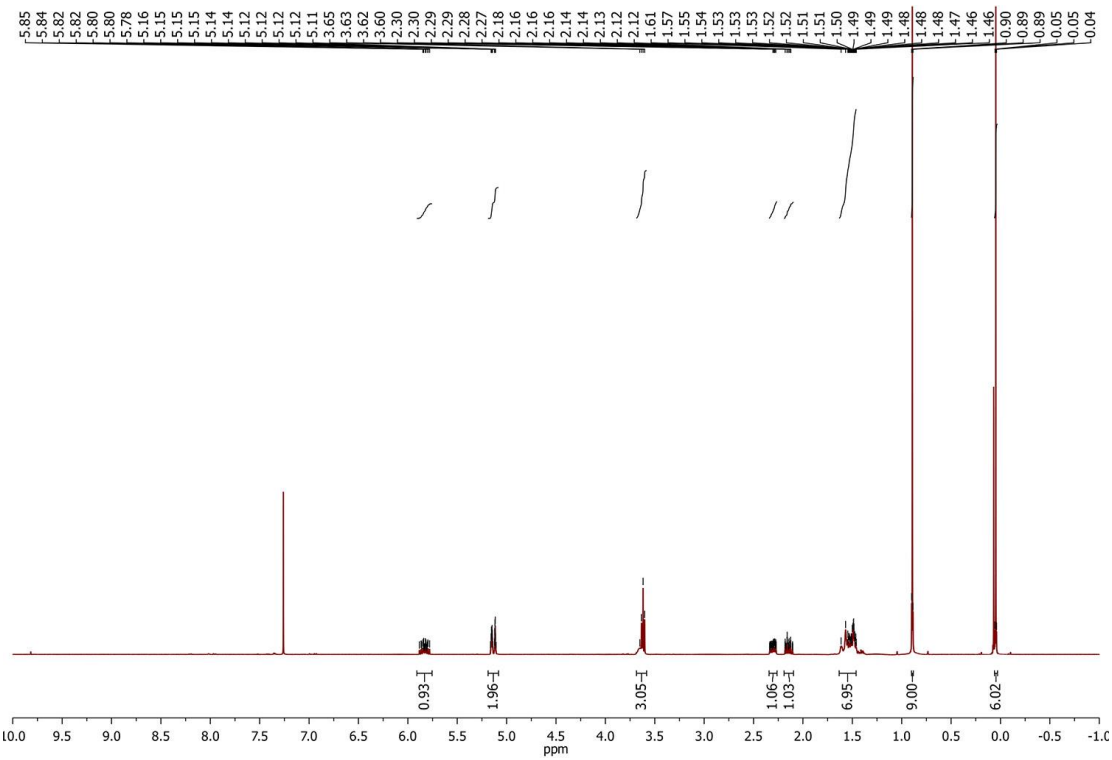
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)



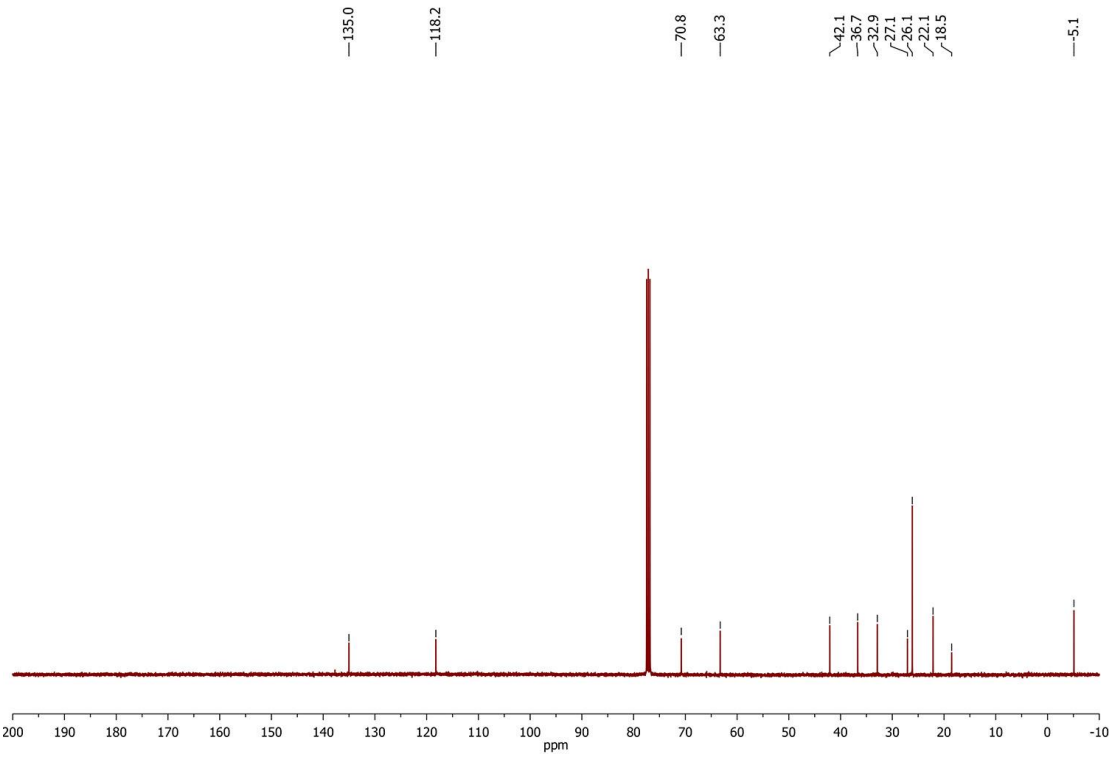


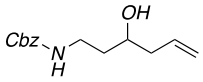
3n

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



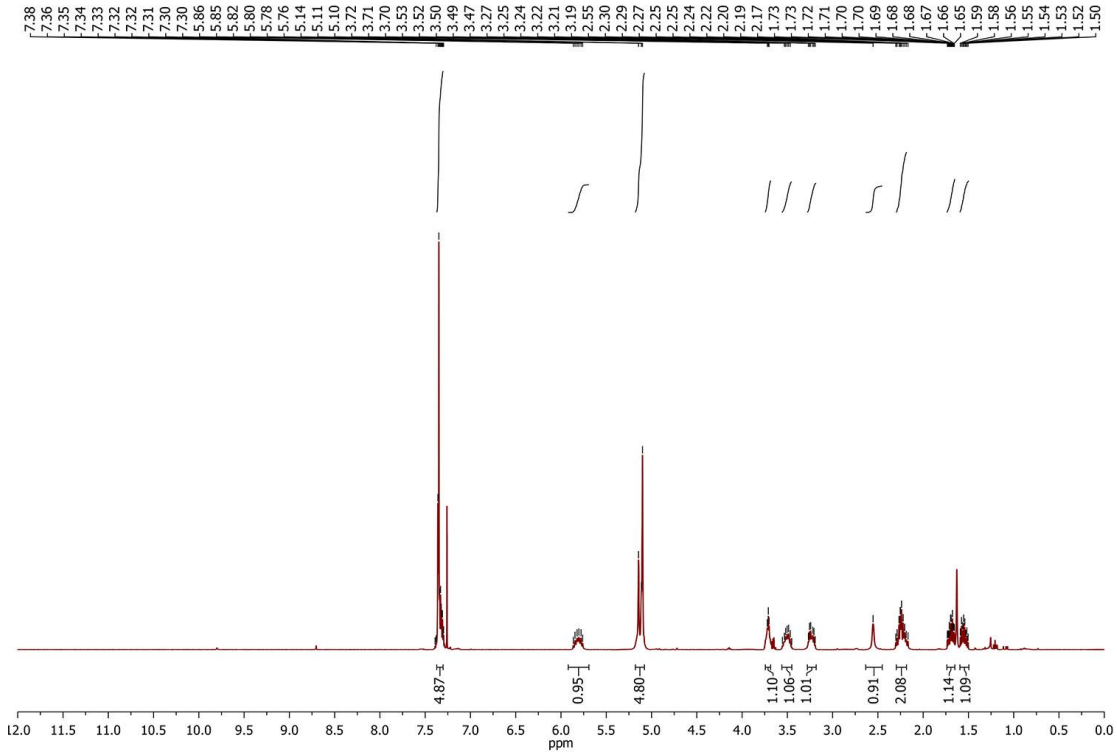
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)



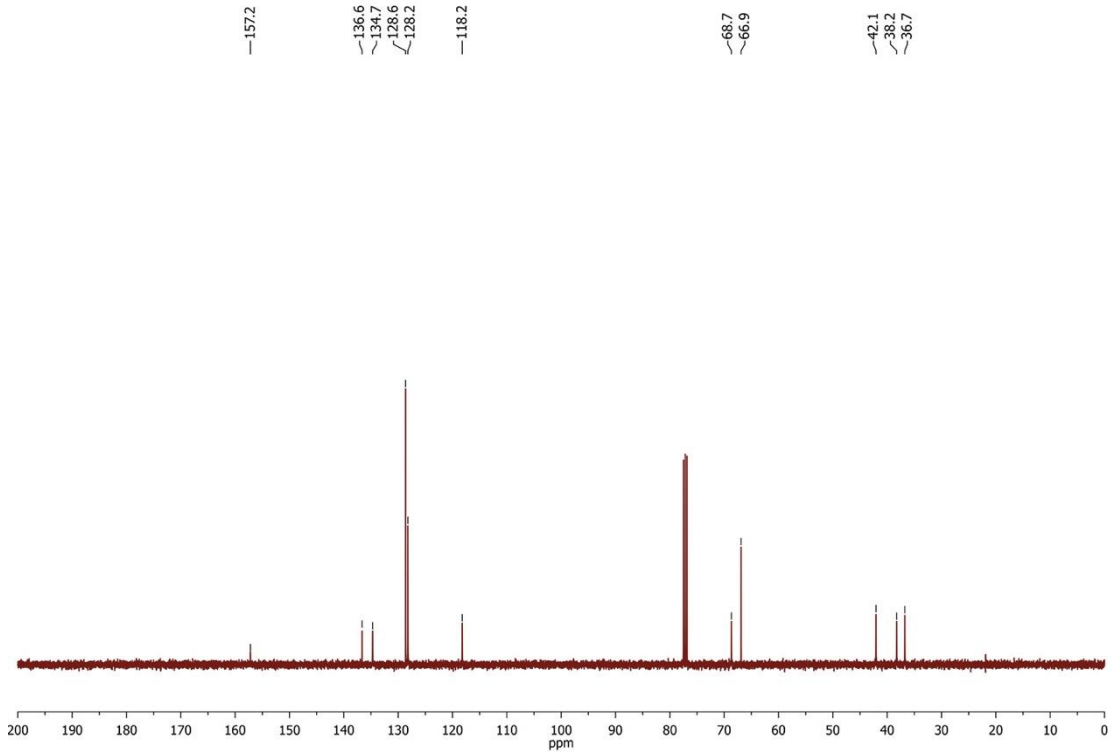


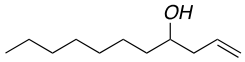
3o

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



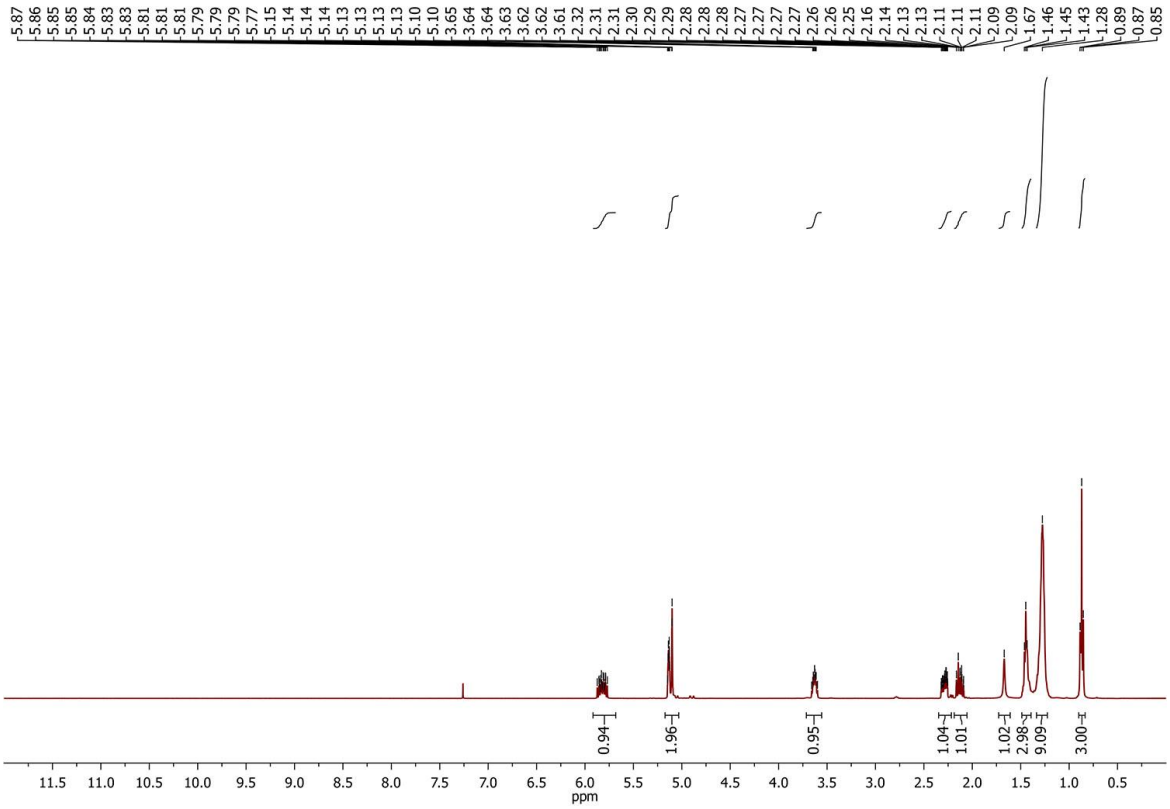
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)



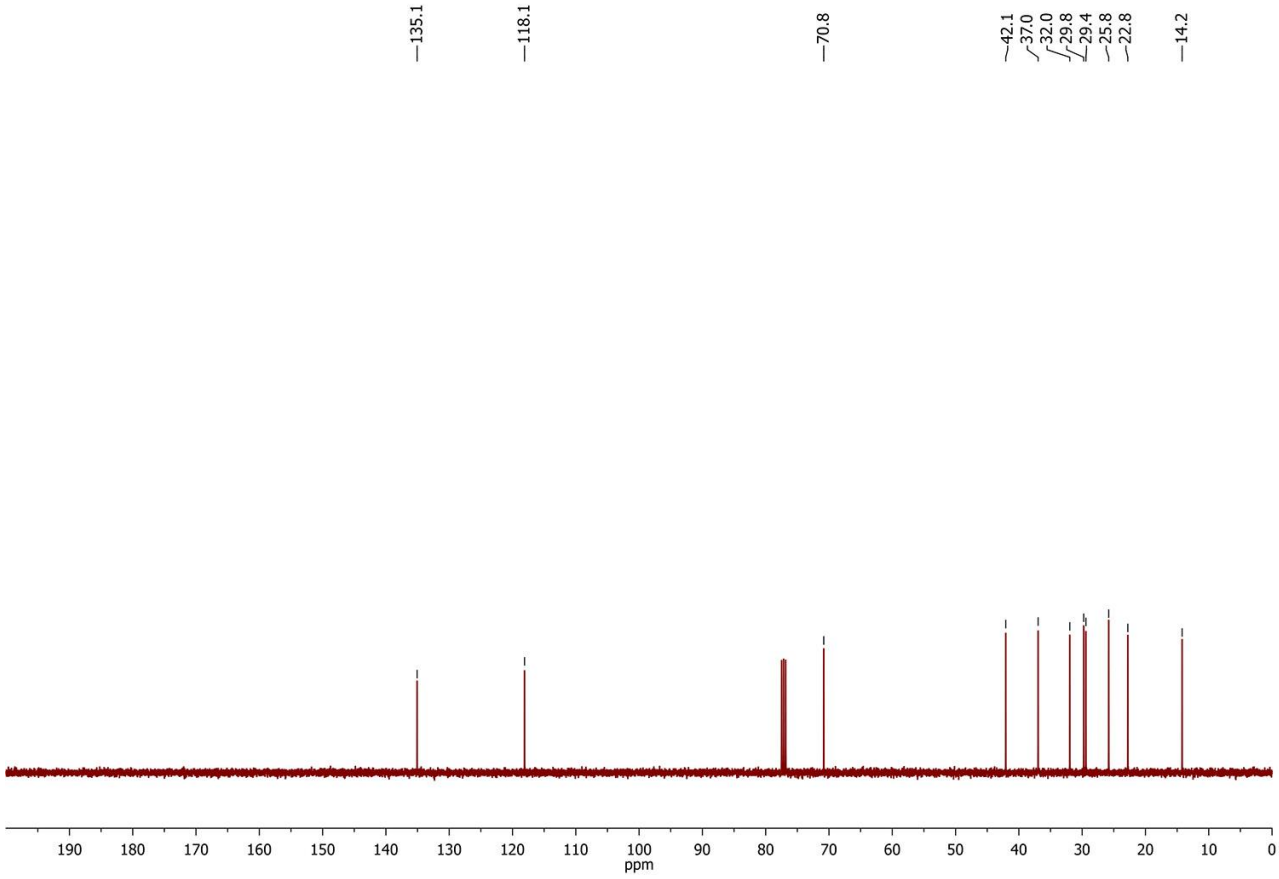


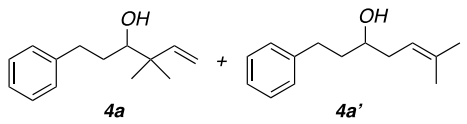
3p

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

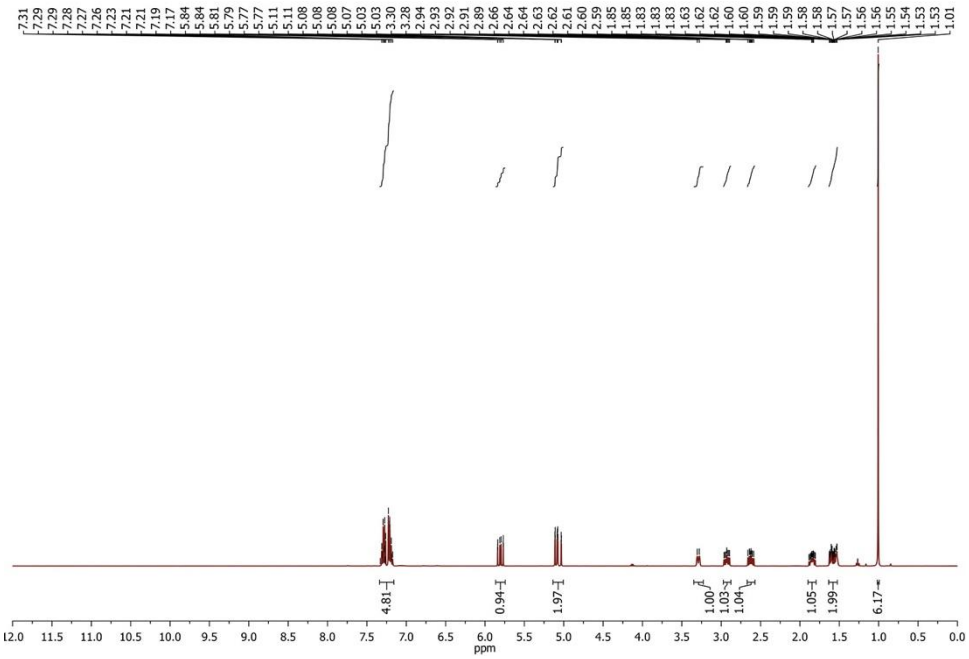


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)

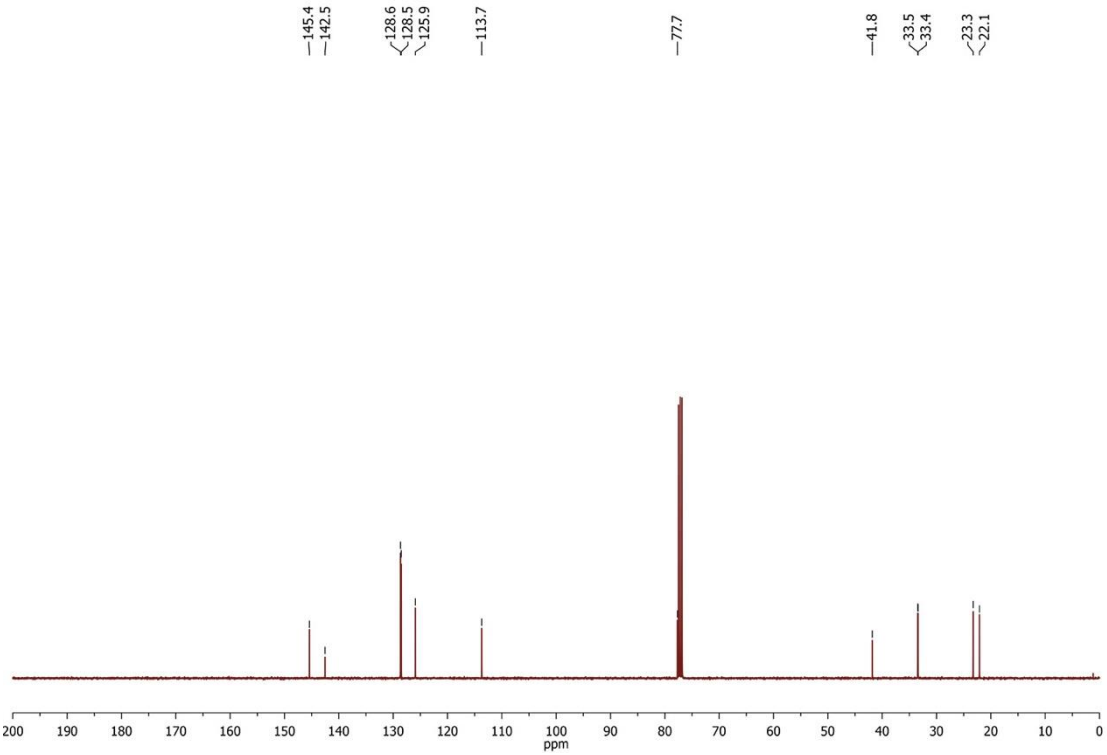


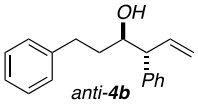


^{1}H NMR (400 MHz, $CDCl_3$)

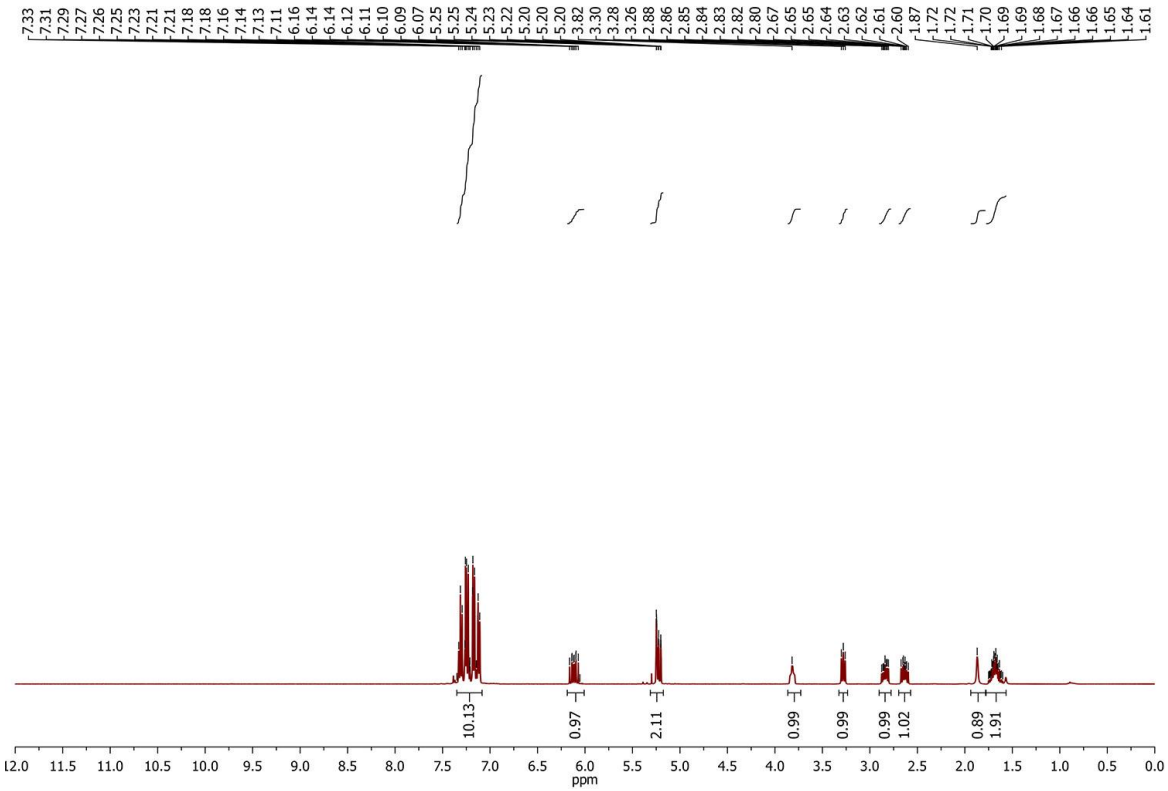


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)

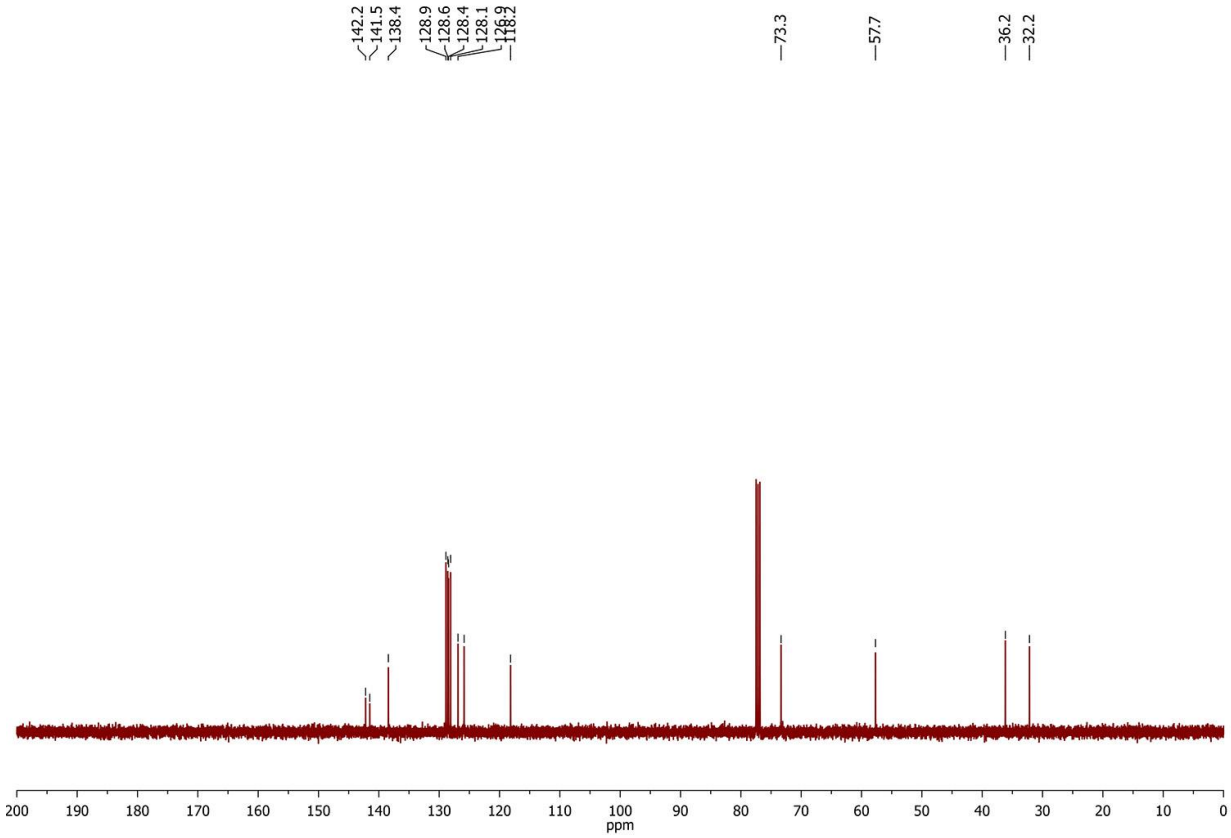




^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)



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