Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

Copper-Catalyzed Decarboxylative C(sp^2)-C(sp^3) Coupling Reactions *via* Radical Mechanism

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General Information:

All reactions were carried out under an argon atmosphere condition. CuO was purchased from Strem Corp. with high purity (99.995%), various cinnamic acids and various benzylic hydrocarbons were purchased from Aldrich, Acros or Alfa. Column chromatography was generally performed on silica gel (100-200 mesh) and reactions were monitored by thin layer chromatography (TLC) using UV light (254 nm) to visualize the course of the reactions. The ^{1}H (300 MHz or 400 MHz) and ^{13}C NMR (75 MHz or 100 MHz) data were recorded on Varian 300 M or 400 M spectrometers using $CDCl_3$ as solvent. The chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. ^{1}H NMR spectra was recorded with tetramethylsilane (δ = 0.00 ppm) as internal reference; ^{13}C NMR spectra was recorded with $CDCl_3$ (δ = 77.500 ppm) as internal reference. ESI-MS and HRMS were performed by the State-authorized Analytical Center in Soochow University.

General procedure for copper-catalyzed decarboxylative C-H functionalization coupling between cinnamic acid and benzylic hydrocarbon:

To a Schlenk tube equipped with a magnetic stir bar were added under argon, CuO (0.03 mmol) and cinnamic acid (0.3 mmol). Under argon, benzylic hydrocarbon (2.0 mL) and DTBP (di-*tert*-butyl peroxide, 0.6 mmol) were added. The resulting reaction mixture was kept stirring at the required temperature for 24 h. After required reaction time, the mixture was cooled down to room temperature. Evaporation of the solvent followed by purification by flash chromatography (petroleum ether or petroleum ether/ethyl acetate) afforded the corresponding product.

Copy of Certificate of Analysis of CuO:

STREM CHEMICALS, INC.

CERTIFICATE OF ANALYSIS

29-0590 Copper (II) oxide (99.995%-Cu)PURATREM

Lot Number:	18604800
%Cu:	79.70000
Color and Form:	BLACK PWDR.
Metals purity:	<50PPM

Element	ppm/%	Element	ppm/%
Pb (ppm):	<1	Sn (ppm):	<1

Quality Control Manager

September 17, 2010

Corporate Headquarters:
7 Mulliken Way
Newburyport, MA 01950-4098 U.S.A.
Tel: (978) 499-1600
Fax: (978) 465-3104

Strem Chemicals, Inc.
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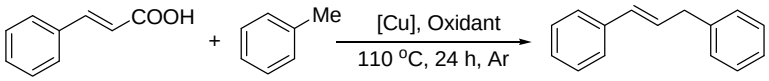
European Headquarters:
15, rue de l' Atome
67800 BISCHHEIM (France)
Tel: (33) 03 88 62 52 60
Fax: (33) 03 88 62 26 81

Experimental data:

Table S1: Optimization of reaction conditions.[a]



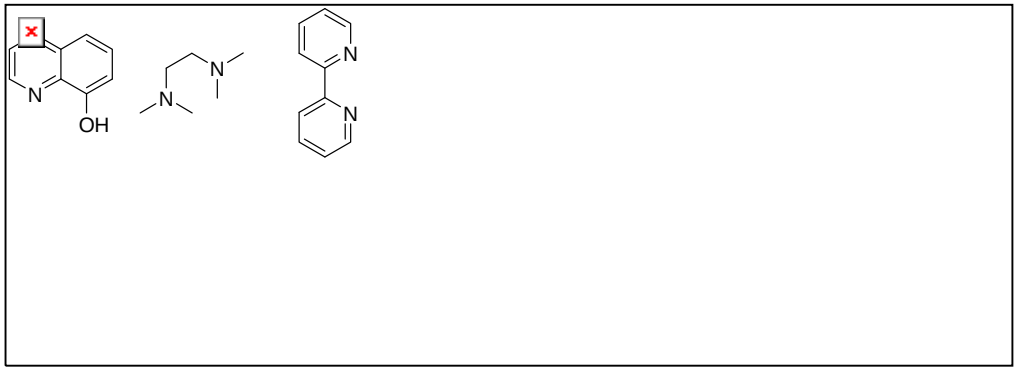
1a **2a** **3a**

Entry	Copper cat.	Oxidant	Solvent	Yield [%][b]
1	CuBr	TBHP	Toluene	18
2	CuBr	DTBP	Toluene	44
3	CuI	DTBP	Toluene	53
4	CuCl	DTBP	Toluene	55
5	Cu_2O	DTBP	Toluene	35
6	$CuCl_2$	DTBP	Toluene	26
7	$CuBr_2$	DTBP	Toluene	52
8	$Cu(OAc)_2$	DTBP	Toluene	43
9	$Cu(acac)_2$	DTBP	Toluene	55
10	$Cu(OTf)_2$	DTBP	Toluene	NR
11	$CuSO_4$	DTBP	Toluene	34
12	CuO	DTBP	Toluene	60
13	Cu powder	DTBP	Toluene	35
14[c]	CuO	DTBP	Toluene	38
15[d]	CuO	DTBP	Toluene	Trace
16[e]	CuO	DTBP	Toluene	Trace
17[f]	CuO	DTBP	DMF	Trace
18[f]	CuO	DTBP	DMSO	Trace
19[f]	CuO	DTBP	Dioxane	Trace
20[f]	CuO	DTBP	NMP	Trace
21[f]	CuO	DTBP	chlorobenzene	Trace
22	CuO	H_2O_2	Toluene	NR
23	CuO	BQ	Toluene	NR
24	CuO	DCP	Toluene	38
25	CuO(10%)	AIBN	Toluene	NR
26	CuO(100%)	AIBN	Toluene	NR
27[g]	CuO	DTBP	Toluene	61
28	CuO	DTBP(400%)	Toluene	56
29	CuO	DTBP(100%)	Toluene	56
30	CuO	DTBP(50%)	Toluene	33
31	CuO(30%)	DTBP	Toluene	59
32	CuO(10%)	DTBP	Toluene	70
33	CuO(5%)	DTBP	Toluene	53
34[h]	CuO(10%)	DTBP	Toluene	**76**
35	–	DTBP	Toluene	20

36	CuO	–	Toluene	NR

[a] Catalytic conditions: Cinnamic acid (0.3 mmol), solvent (2 mL), copper catalyst (20mol%), oxidant (2 equiv), 110 °C, 24 h, Ar. [b] Isolated yield based on cinnamic acid. [c] 130 °C, air. [d] 90 °C, Ar. [e] 70 °C, Ar. [f] toluene (3 mmol). [g] 48 h. [h] CuO was purchased from Strem Corp. with high purity (99.995%)

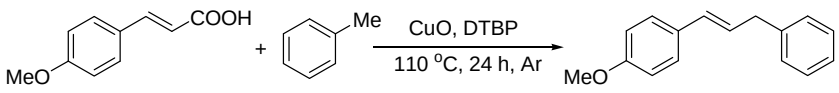
Scheme S1 The evaluated ligand in this study



Entry	Copper cat. [20 mol%]	Oxidant	Ligand	Yield [%][b]
1	CuO	DTBP	**L1**	19
2	CuO	DTBP	**L2**	18
3	CuO	DTBP	**L3**	12
4	CuO	DTBP	**L4**	52
5	CuO	DTBP	**L5**	36
6	CuO	DTBP	**L6**	52
7	CuO	DTBP	**L7**	60
8	CuO	DTBP	**L8**	19
9	CuO	DTBP	**L9**	54
10	CuO	DTBP	**L10**	48
11	CuO	DTBP	**L11**	36
12	CuO	DTBP	**L12**	38
13	CuO	DTBP	**L13**	47
14	CuO	DTBP	**L14**	50

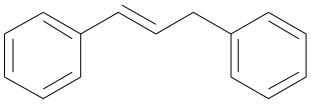
[a] Catalytic conditions: Cinnamic acid (**1a**) (0.3 mmol), toluene (**2a**) (2 mL), copper catalyst (20 mol%), ligand (2 equiv),oxidant (2 equiv), 110 °C, 24 h, Ar. [b] Isolated yield based on cinnamic acid.

Scheme S2 Effect of Radical Inhibitors



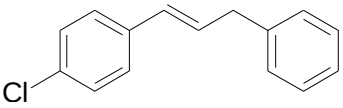
Isolated yield
No Scavenger = 77%
TEMPO< 1%
AIBN< 1%
BQ< 1%

Characterization of the corresponding products:



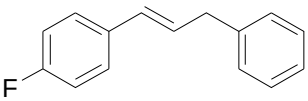
(E)-1-1, 3-diphenylpropene (3a)

^{1}H NMR (300 MHz, $CDCl_3$) (δ, ppm) 7.40–7.28 (m, 6H), 7.26–7.15 (m, 3H), 6.46 (d, J = 15.9 Hz, 1H), 6.40–6.30 (m, 1H), 3.55 (d, J = 6.2 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 140.6, 137.9, 131.5, 129.7, 129.2, 129.1, 129.0, 127. 6, 126.6, 126.6, 39.8; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{15}H_{13}$, 193.1017; found, 193.0963.



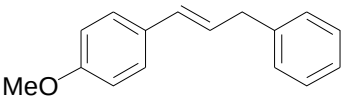
(*E*)-1-(4-Chlorophenyl)-3-phenylpropene (3b)

^{1}H NMR (300 MHz, $CDCl_3$) (δ, ppm) 7.35–7.28 (m, 2H), 7.23 (d, J = 10.2 Hz, 6H), 6.44–6.26 (m, 2H), 3.53 (d, J = 5.6 Hz,2H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 140.0, 136.1, 132.8, 130.2, 123.0, 128.8, 128.8, 128.7, 127.5, 126.4, 39.5; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{15}H_{12}Cl$, 227.0627; found, 227.0630.



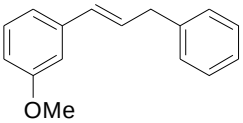
(*E*)-1-(4-Fluorophenyl)-3-phenylpropene (3c)

^{1}H NMR (300 MHz, $CDCl_3$) (δ, ppm) ^{1}H NMR (300 MHz, $CDCl_3$) (δ, ppm) 7.33 – 7.27 (m, 4H), 7.23 (d, J = 7.0 Hz, 3H), 6.96 (t, J = 8.7 Hz, 2H), 6.40 (d, J = 15.8 Hz, 1H), 6.26 (dt, J = 15.7, 6.7 Hz, 1H), 3.52 (d, J = 6.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 162.0 (d, ^1J = 244.6 Hz), 140.0, 133.6 (d, ^4J = 3.1 Hz), 129.8, 129.0, 128.6, 128.5, 127.5 (d, ^3J = 7.8 Hz), 126.2, 115.3 (d, ^2J = 21.4 Hz), 39.3;HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{15}H_{12}F$, 211.0923; found, 211.0845.



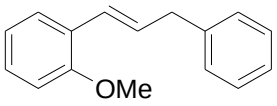
(*E*)-1-(4-Methoxyphenyl)-3-phenylpropene (3d)

^{1}H NMR (300 MHz, $CDCl_3$) (δ, ppm) 7.78–7.23 (m, 7H), 6.95 (d, J = 8.3 Hz, 2H), 6.52 (d, J = 15.8 Hz, 1H), 6.43–6.22 (m, 1H), 3.91 (s, 3H), 3.64 (d, J = 6.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 159.3, 140.9, 130.9, 130.8, 129.1, 128.9, 127.7, 127.5, 126.6, 114.4, 55.7, 39.8; MS ESI (m/z): $[M+H]^+$ calcd for $C_{16}H_{17}O$, 225.1; found, 225.1.



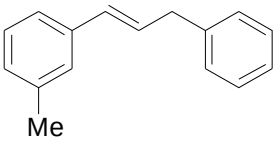
(*E*)-1-(3-Methoxyphenyl)-3-phenylpropene (3e)

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.31 (t, J = 7.3 Hz, 2H), 7.25–7.18 (m, 4H), 6.95 (d, J = 7.6 Hz, 1H), 6.89 (s, 1H), 6.76 (d, J = 8.1 Hz, 1H), 6.43 (d, J = 15.9 Hz, 1H), 6.39–6.28 (m, 1H), 3.79 (s, 3H), 3.54 (d, J = 6.3 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 159.8, 140.1, 138.9, 131.0, 129.6, 129.5, 128.71, 128.5, 126.2, 118.8, 112.9, 111.4, 55.2, 39.3; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{16}H_{15}O$, 223.1123; found, 223.1008.



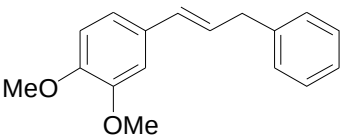
(*E*)-1-(2-Methoxyphenyl)-3-phenylpropene (3f)

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.41 (d, J = 7.6 Hz, 1H), 7.3–7.26 (m, 2H), 7.25–7.13 (m, 4H), 6.92–6.84 (m, 2H), 6.82 (d, J = 16.8 Hz, 1H), 6.39–6.29 (m, 1H), 3.83 (s, 3H), 3.57 (d, J = 7.1 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 156.5, 140.7, 129.9, 128.7, 128.5, 128.2, 126.7, 126.6, 126.2, 125.9, 120.7, 110.9, 55.6, 40.0; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{16}H_{15}O$, 223.1123; found, 223.0844.



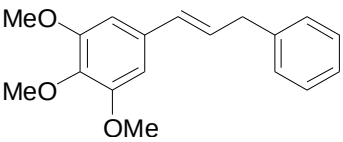
(*E*)-1-(3-Methylphenyl)-3-phenylpropene (3g)

^{1}H NMR (300 MHz, $CDCl_3$) (δ, ppm) 7.39–7.27 (m, 2H), 7.26–7.20 (m, 3H), 7.19–7.13 (m, 2H), 7.01 (d, J = 4.9 Hz, 1H), 6.43 (d, J = 16.0 Hz, 1H), 6.38–6.25 (m, 1H), 3.53 (d, J = 6.1 Hz, 2H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 140.4, 138.2, 137.6, 131.3, 129.2, 128.8, 128.6, 128.5, 128.0, 127.0, 126.3, 123.4, 39.5, 21.5; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{16}H_{15}$, 207.1174; found, 207.1146.



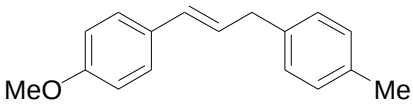
(*E*)-1-(3,4-Dimethoxyphenyl)-3-phenylpropene (3h)

^{1}H NMR (300 MHz, $CDCl_3$) (δ, ppm) 7.53–7.05 (m, 5H), 6.89 (d, J = 10.5 Hz, 2H), 6.79 (d, J = 8.1 Hz, 1H), 6.40 (d, J = 15.8 Hz, 1H), 6.29–6.07 (m, 1H), 3.87 (s, 3H), 3.53 (d, J = 6.6 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 149.4, 148.9, 140.8, 131.1, 129.2, 128.9, 127.8, 126.6, 119.6, 111.6, 109.0, 56.4, 56.2, 39.8; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{17}H_{17}O_2$, 253.1228; found, 253.1016.



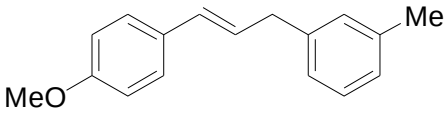
(*E*)-1-(3,4,5-Trimethoxyphenyl)-3-phenylpropene (3i)

^{1}H NMR (300 MHz, $CDCl_3$) (δ, ppm) 7.37–7.19 (m, 5H), 6.56 (t, J = 8.4 Hz, 2H), 6.38 (d, J = 15.8 Hz, 1H), 6.33–6.20 (m, 1H), 3.85 (s, 6H), 3.83 (s, 3H) 3.54 (d, J = 6.3 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) (δ, ppm) 153.4, 140.2, 137.6, 133.4, 131.0, 129.0, 128.8, 128.6, 126.4, 103.3, 61.0, 56.2, 39.4; MS ESI (m/z): $[M+H]^+$ calcd for $C_{18}H_{21}O_3$, 285.2; found, 285.2 .



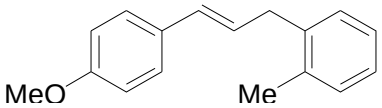
(*E*)-1-(4-Methoxyphenyl)-3-(4-methylphenyl)-propene (4b)

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.26 (t, J = 11.6 Hz, 2H), 7.12 (m, 4H), 6.82 (d, J = 8.4 Hz, 2H), 6.38 (d, J = 15.7 Hz, 1H), 6.31–6.10 (m, 1H), 3.78 (s, 3H), 3.48 (d, J = 6.7 Hz, 2H), 2.32 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 158.8, 137.4, 135.6, 130.4, 130.2, 129.2, 128.5, 127.4, 127.2, 113.9, 55.3, 38.9, 21.0; MS ESI (m/z): $[M+H]^+$ calcd for $C_{17}H_{19}O$, 239.1; found, 239.1.



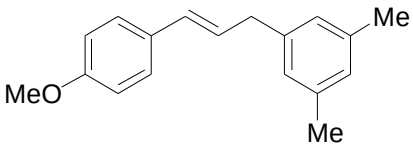
(*E*)-1-(4-Methoxyphenyl)-3-(3-methylphenyl)-propene (4c)

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.29 (d, J = 8.0 Hz, 2H), 7.24–7.12 (m, 2H), 7.04 (d, J = 7.4 Hz, 2H), 6.83 (d, J = 7.9 Hz, 2H), 6.40 (d, J = 15.9 Hz, 1H), 6.26–6.15 (m, 1H), 3.79 (s, 3H), 3.49 (d, J = 6.6 Hz, 2H), 2.33 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 158.8, 140.4, 138.1, 130.3, 130.3, 129.4, 128.5, 128.4, 127.2, 126.8, 125.7, 113.9, 55.3, 39.3, 21.4; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{17}H_{17}O$, 237.1279; found, 237.1266.



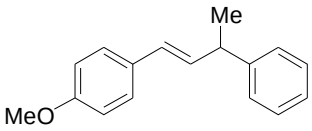
(*E*)-3-(2-Methylphenyl)-1-(4-methoxyphenyl) -propene (4d)

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.27–7.15 (m, 7H), 6.81 (d, J = 8.4 Hz, 2H), 6.30 (d, J = 15.9 Hz, 1H), 6.26–6.01 (m, 1H), 3.77 (s, 3H), 3.49 (d, J = 6.0 Hz, 2H), 2.32 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 158.9, 138.6, 136.4, 130.4, 130.3, 130.3, 130.0, 129.3, 127.2, 126.4, 126.1, 114.0, 55.3, 36.9, 19.5; MS ESI (m/z): $[M+H]^+$ calcd for $C_{17}H_{19}O$, 239.1; found, 239.1.



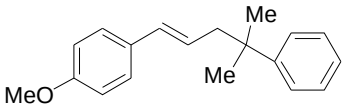
(*E*)-1-(4-Methoxyphenyl)-3-(3,5-dimethylphenyl)-propene (4e)

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.29 (d, J = 8.7 Hz, 2H), 7.06–6.73 (m, 4H), 6.39 (d, J = 15.7 Hz, 1H), 6.31–6.06 (m, 1H), 3.78 (s, 3H), 3.44 (d, J = 6.9 Hz, 2H), 2.29 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 158.8, 140.4, 138.0, 130.4, 130.2, 127.7, 127.3, 127.2, 126.4, 113.9, 55.3, 39.3, 21.3; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{18}H_{19}O$, 251.1436; found, 251.1258.



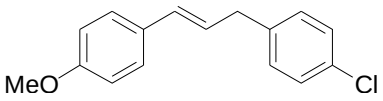
(*E*)-1-(4-Methoxyphenyl)-3-phenylbutene (4f)

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.37–7.30 (m, 7H), 7.27–7.23 (m,1H), 6.87 (d, J = 8.7 Hz, 2H), 6.40 (d, J = 15.9 Hz, 1H), 6.31–6.26 (m, 1H), 3.82 (s, 3H), 3.67–3.62 (m, 1H), 1.49 (d, J = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 158.8, 145.9, 133.1, 130.4, 128.5, 127.9, 127.3, 127.2, 126.2, 113.9, 55.3, 42.6, 21.4; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{17}H_{17}O$, 237.1279; found, 237.0856.



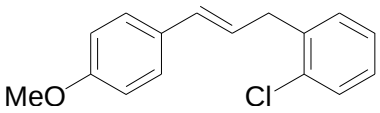
(*E*)-1-(4-Methoxyphenyl)-3-methyl-3-phenylbutene (4g)

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.28 (s, 1H), 7.25 (s, 2H), 7.23–7.17 (m, 2H), 7.12 (d, J = 7.3 Hz, 2H), 6.84 (d, J = 8.3 Hz, 2H), 6.18–6.07 (m, 2H), 3.80 (s, 3H), 2.66 (s, 2H), 1.09 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 158.6, 138.8, 138.1, 130.8, 130.6, 127.6, 127.1, 125.9, 125.2, 113.9, 55.3, 49.7, 37.2, 27.0; ESI (m/z): $[M-H]^+$ calcd for $C_{19}H_{21}O$, 265.1592; found, 265.1494.



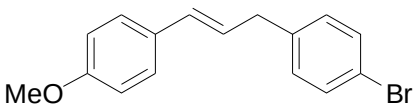
(*E*)-1-(4-Methoxyphenyl)-3-(4-chlorophenyl)-propene (4h)

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.27 (t, J = 7.8 Hz, 4H), 7.15 (d, J = 8.3 Hz, 2H), 6.83 (d, J = 8.7 Hz, 2H), 6.37 (d, J = 15.8 Hz, 1H), 6.24–6.08 (m, 1H), 3.79 (s, 3H), 3.48 (d, J = 6.7 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 158.9, 138.9, 131.8, 130.8, 130.0, 128.7, 128.5, 127.2, 126.4, 114.0, 55.3, 38.6; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{16}H_{14}ClO$, 257.0733; found, 257.0698.



(*E*)-3-(2-Chlorophenyl)-1-(4-methoxyphenyl)-propene (4i)

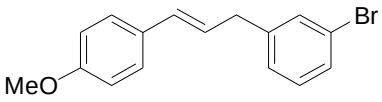
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.36 (d, J = 7.4 Hz, 1H), 7.33–7.25 (m, 3H), 7.23–7.11 (m, 2H), 6.83 (d, J = 8.5 Hz, 2H), 6.39 (d, J = 15.8 Hz, 1H), 6.26–6.11 (m, 1H), 3.79 (s, 3H), 3.63 (d, J = 6.6 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 158.9, 138.1, 134.0, 131.1, 130.4, 130.2, 129.4, 127.6, 127.3, 126.9, 125.0, 113.9, 55.3, 36.8; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{16}H_{14}ClO$, 257.0733; found, 257.0740.



(*E*)-3-(4-Bromophenyl)-1-(4-methoxyphenyl)-propene (4j)

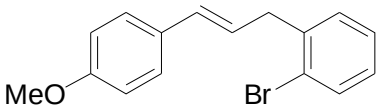
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.41 (d, J = 8.1 Hz, 2H), 7.28 (d, J = 8.3 Hz, 2H), 7.11 (d, J = 7.9 Hz, 2H), 6.83 (d, J = 8.5 Hz, 2H), 6.38 (d, J = 15.7 Hz, 1H), 6.21–6.10 (m, 1H), 3.79 (s, 3H),

3.46 (d, J = 6.8 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 158.9, 139.4, 131.5, 130.9, 130.4, 130.0, 127.2, 126.2, 119.9, 113.9, 55.3, 38.7; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{16}H_{16}BrO$, 303.0385; found, 257.0203.



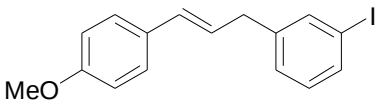
(*E*)-3-(3-Bromophenyl)-1-(4-methoxyphenyl)-propene (4k)

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.36–7.32 (m, 2H), 7.29 (d, J = 6.9 Hz, 2H), 7.16 (s, 2H), 6.84 (d, J = 8.4 Hz, 2H), 6.39 (d, J = 15.7 Hz, 1H), 6.22–6.09 (m, 1H), 3.79 (s, 3H), 3.48 (d, J = 6.3 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 159.0, 142.8, 131.6, 131.1, 130.0, 129.2, 127.3, 125.9, 122.5, 113.9, 55.3, 38.9; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{16}H_{16}BrO$, 303.0385; found, 303.0212.



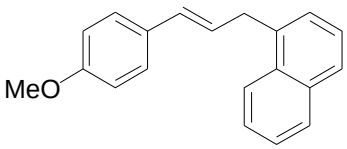
(*E*)-3-(2-Bromophenyl)-1-(4-methoxyphenyl)-propene (4l)

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.55 (d, J = 7.9 Hz, 1H), 7.29 (d, J = 8.4 Hz, 2H), 7.27–7.22 (m, 2H), 7.08 (t, J = 7.4 Hz, 1H), 6.83 (d, J = 8.4 Hz, 2H), 6.40 (d, J = 15.7 Hz, 1H), 6.25–6.11 (m, 1H), 3.79 (s, 3H), 3.64 (d, J = 6.8 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 158.9, 139.8, 132.8, 131.1, 130.5, 130.2, 127.8, 127.5, 127.3, 125.1, 124.6, 113.9, 55.3, 39.4; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{16}H_{14}BrO$, 301.0228; found, 301.0211.



(*E*)-3-(3-Iodophenyl)-1-(4-methoxyphenyl)-propene (4m)

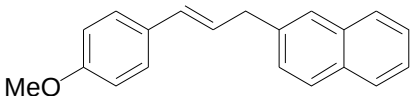
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.59 (s, 1H), 7.55 (d, J = 7.6 Hz, 1H), 7.29 (d, J = 8.4 Hz, 2H), 7.20 (d, J = 7.9 Hz, 1H), 7.03 (t, J = 7.7 Hz, 1H), 6.84 (d, J = 8.3 Hz, 2H), 6.40 (d, J = 15.6 Hz, 1H), 6.20–6.06 (m, 1H), 3.80 (s, 3H), 3.46 (d, J = 6.7 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 159.0, 142.9, 137.6, 135.2, 131.1, 130.2, 130.0, 127.9, 127.3, 126.0, 114.0, 94.6, 55.3, 38.8; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{16}H_{14}IO$, 349.0089; found, 349.0005.



(*E*)-1-(4-Methoxyphenyl)-3-α-naphthalenylpropene (4n)

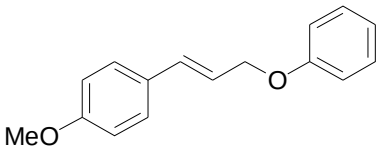
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 8.08 (d, J = 7.8 Hz, 1H), 7.85 (d, J = 7.9 Hz, 1H), 7.74 (d, J = 7.6 Hz, 1H), 7.52–7.45 (m, 2H), 7.43–7.37 (m, 2H), 7.25 (d, J = 8.4 Hz, 2H), 6.80 (d, J = 8.6 Hz, 2H), 6.41 (d, J = 16.0 Hz, 1H), 6.37–6.30(m, 1H), 3.96 (d, J = 5.8 Hz, 2H), 3.76 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 158.9, 136.6, 133.9, 132.1, 130.7, 130.3, 128.7, 127.2, 127.0,

126.7, 126.4, 126.0, 125.7, 125.6, 124.1, 113.9, 55.3, 36.4; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{20}H_{17}O$, 273.1279; found 273.1273.



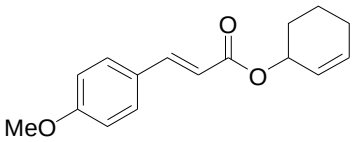
(*E*)-1-(4-Methoxyphenyl)-3-β-naphthalenylpropene (4o)

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.79 (t, J = 8.8 Hz, 3H), 7.66 (s, 1H), 7.48–7.39 (m, 2H), 7.37 (d, J = 8.4 Hz, 1H), 7.30 (d, J = 8.0 Hz, 2H), 6.83 (d, J = 8.0 Hz, 2H), 6.44 (d, J = 15.8 Hz, 1H), 6.34–6.22 (m, 1H), 3.78 (s, 3H), 3.67 (d, J = 6.6 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 158.9, 138.0, 133.7, 132.2, 130.7, 130.3, 128.0, 127.7, 127.5, 127.3, 126.9, 126.7, 126.0, 125.3, 114.0, 55.3, 39.5; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{20}H_{17}O$, 273.1279; found 273.1270.



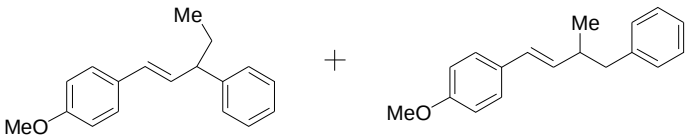
(*E*)-1-(4-Methoxyphenyl)-3-phenoxylpropene (4p)

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.34 (d, J = 8.1 Hz, 2H), 7.29 (t, J = 7.9 Hz, 2H), 6.96 (d, J = 8.1 Hz, 2H), 6.85 (d, J = 8.4 Hz, 2H), 6.67 (d, J = 15.9 Hz, 1H), 6.32–6.25 (m, 1H), 4.66 (d, J = 5.4 Hz, 2H), 3.80 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 159.4, 158.6, 132.8, 129.4, 129.2, 127.8, 122.1, 120.8, 114.8, 114.0, 68.8, 55.3, 30.9; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{16}H_{15}O_2$, 239.1072; found, 239.1101.



(*E*)-3-(4-Methoxy-phenyl)-acrylic acid cyclohex-2-enyl ester (4q)

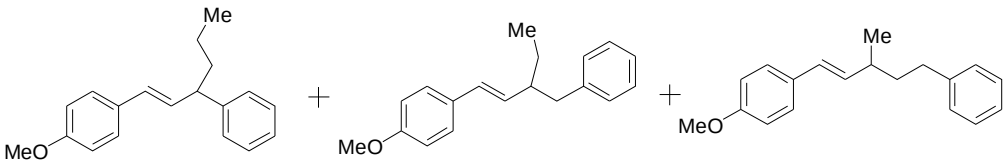
^{1}H NMR (300 MHz, $cdcl_3$) (δ, ppm) 7.64 (d, J = 16.0 Hz, 1H), 7.46 (d, J = 8.8 Hz, 2H), 6.88 (d, J = 8.7 Hz, 2H), 6.31 (d, J = 16.0 Hz, 1H), 5.97 (dt, J = 9.0, 3.3 Hz, 1H), 5.80-5.76 (m, 1H), 5.44 – 5.35 (m, 1H), 3.81 (s, 3H), 2.09-2.05 (m, 1H), 2.01 – 1.86 (m, 2H), 1.85-1.76 (m, 2H), 1.73 – 1.64 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 167.3, 161.7, 144.5, 133.0, 130.0, 127.6, 126.3, 116.4, 114.6, 68.3, 55.7, 28.8, 25.3, 19.3; MS ESI (m/z): $[M+Na]^+$ calcd for $C_{16}H_{18}O_3Na$ 281.1; found 281.1.



(*E*)-1-(4-Methoxyphenyl)-3-phenylpentene; (*E*)-1-(4-Methoxyphenyl)-3-benzylbutene.

Both isomers are reported together (ratio: 3.1:1 as determined by NMR).

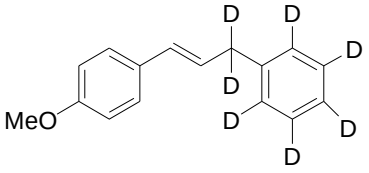
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.33–7.27 (m, 4H), 7.24 (t, J = 7.1 Hz, 4H), 7.21–7.16 (m, 2H), 6.82 (d, J = 8.7 Hz, 3H), 6.34 (d, J = 15.8 Hz, 1H), 6.28 (d, J = 15.8 Hz, 0.3H), 6.23–6.16 (m, 1H), 6.07–6.01 (m, 0.3H), 3.79 (s, 1H), 3.78 (s, 3H), 3.30–3.25 (m, 1H), 2.80–2.73 (m, 0.3H), 2.65–2.53 (m, 1H), 1.95–1.71 (m, 2H), 1.07 (d, J = 6.2 Hz, 1H), 0.90 (t, J = 7.3 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm) 158.8, 158.7, 144.8, 140.7, 133.9, 132.1, 130.7, 130.5, 129.3, 128.8, 128. 5, 128.1, 127.7, 127.6, 127.2, 127.1, 126.1, 125.8, 113.9, 55.3, 51.0, 43.8, 38.8, 28.9, 19.9, 12.3; HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{18}H_{19}O$, 251.1436; found, 251.1423.



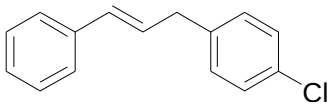
(*E*)-1-(4-Methoxyphenyl)-3-phenylhexene; (*E*)-1-(4-Methoxyphenyl)-3-benzylpentene; (*E*)-1-(4-Methoxyphenyl)-3-(beta-phenethyl)-butene.

The mixture of three isomers is reported together (ratio: 3.2:1:1.4 as determined by NMR).

^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.35 (d, J = 7.2 Hz, 2H), 7.33–7.28 (m, 7H), 7.23 (t, J = 7.3 Hz, 3H), 6.87 (dd, J = 12.3, 8.9 Hz, 4H), 6.36 (dd, J = 15.9, 6.9 Hz, 2H), 6.22 (dd, J = 15.7, 8.0 Hz, 1.5H), 6.01 (dd, J = 15.8, 8.0 Hz, 0.5H), 5.91 (dd, J = 15.8, 8.5 Hz, 0.15H), 3.83 (s, 2H), 3.81 (s, 4H), 3.43 (q, J = 7.5 Hz, 1H), 2.74 (d, J = 7.0 Hz, 0.6H), 2.67 (dd, J = 18.3, 7.8 Hz, 0.9H), 2.39–2.29 (m, 0.6H), 2.30–2.21 (m, 0.1H), 1.86–1.74 (m, 3.3H), 1.75–1.66 (m, 1H), 1.42–1.26 (m, 3H), 1.14 (d, J = 6.7 Hz, 1.2H), 0.95 (t, J = 7.4 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 158.7, 158.7, 144.9, 142.7, 140.7, 134.2, 132.3, 132.2, 130.6, 130.4, 129.3, 128.6, 128.4, 128.3, 128.0, 127.9, 127.6, 127.2, 127.0, 126.1, 125.7, 125.6, 55.2, 48.9, 46.6, 42.0, 38.9, 38.2, 36.9, 33.7, 27.3, 20.9, 20.8, 14.0, 11.8. HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{19}H_{21}O$, 265.1592; found, 265.1590.



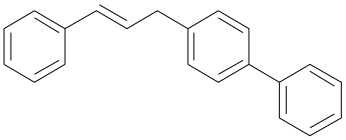
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.29 (d, J = 8.3 Hz, 2H), 7.25 (s, 1H), 6.83 (d, J = 8.1 Hz, 2H), 6.40 (d, J = 15.7 Hz, 1H), 6.20 (d, J = 15.8 Hz, 1H), 3.79 (s, 3H);



(*E*)-3-(4-Chlorophenyl)-1-phenyl-propene (6)

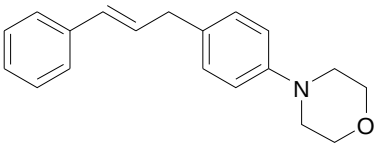
^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.34 (d, J = 7.5 Hz, 2H), 7.27 (dd, J = 7.7, 5.9 Hz, 4H), 7.21 (d, J = 5.9 Hz, 1H), 7.15 (d, J = 8.3 Hz, 2H), 6.43 (d, J = 15.8 Hz, 1H), 6.29 (dt, J = 15.7, 6.7 Hz, 1H), 3.49 (d, J = 6.7 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) (δ, ppm) 139.0, 137.7, 132.4, 131.9,

130.5, 129.0, 127.7, 126.6, 39.07. HRMS ESI (m/z): $[M-H]^+$ calcd for $C_{15}H_{12}Cl$, 227.0628; found, 227.0621.



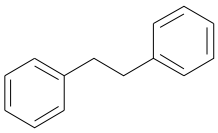
(*E*)-1-(4-Phenyl)-3-(4-phenylphenyl)-propene (7)

^1H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.57 (d, J = 7.6 Hz, 2H), 7.53 (d, J = 7.9 Hz, 2H), 7.41 (t, J = 7.5 Hz, 2H), 7.36 (d, J = 7.6 Hz, 2H), 7.33–7.25 (m, 5H), 7.19 (t, J = 7.3 Hz, 1H), 6.48 (d, J = 15.8 Hz, 1H), 6.42–6.31 (m, 1H), 3.57 (d, J = 6.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 141.4, 139.7, 139.6, 137.9, 131.7, 129.6, 129.5, 129.20, 129.0, 127.7, 127.6, 127.6, 127.5, 126.6, 39.44. MS ESI (m/z): $[M+H]^+$ calcd for $C_{21}H_{19}$ 271.1; found 271.1.



(*E*)-4-[4-(3-Phenyl-allyl)-phenyl]-morpholine (8)

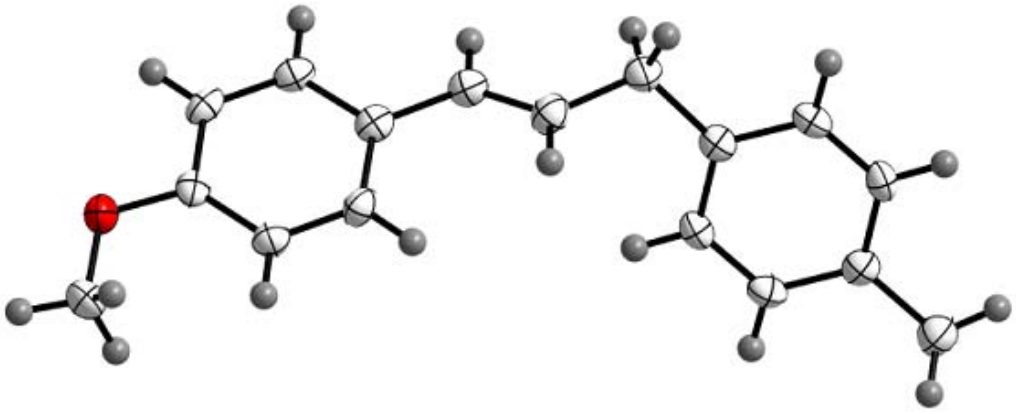
^1H NMR (300 MHz, DMSO) (δ, ppm) 7.43–7.34 (m, 2H), 7.29 (t, J = 7.4 Hz, 2H), 7.19 (t, J = 7.2 Hz, 1H), 7.11 (d, J = 8.5 Hz, 2H), 6.87 (d, J = 8.6 Hz, 2H), 6.48–6.31 (m, 2H), 3.77–3.68 (t, J = 4.8 Hz, 4H), 3.42 (d, J = 5.2 Hz, 2H), 3.08–2.98 (t, J = 4.8 Hz, 4H); ^{13}C NMR (75 MHz, DMSO) (δ, ppm) 149.5, 137.1, 130.6, 130.0, 129.0, 128.5, 127.0, 126.7, 125.9, 115.4, 66.1, 48.8, 38.7. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_{19}H_{22}NO$, 280.1701; found, 280.1691.



1,2-diphenylethane.

^1H NMR (400 MHz, $CDCl_3$) (δ, ppm) 7.28–7.23 (m, 4H), 7.16 (t, J = 7.0 Hz, 6H), 2.90 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm) 142.1, 128.8, 128.7, 126.3, 38.3. MS ESI (m/z): $[M+H]^+$ calcd for $C_{14}H_{15}$ 183.1; found 183.1.

X-ray crystallographic data for compound 4b:



X-ray of 4b

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C C 0.0033 0.0016 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
O O 0.0106 0.0060 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
H H 0.0000 0.0000 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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'x, y, z'
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'-x+1/2, -y, z+1/2'
'-x, y+1/2, -z+1/2'
'x+1/2, -y+1/2, -z'
'-x, -y, -z'
'x-1/2, y, -z-1/2'
'x, -y-1/2, z-1/2'
'-x-1/2, y-1/2, z'

_cell_length_a 14.4230(19)
_cell_length_b 6.1054(8)
_cell_length_c 30.487(4)
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_cell_angle_beta 90.00
_cell_angle_gamma 90.00
_cell_volume 2684.6(6)
_cell_formula_units_Z 8
_cell_measurement_temperature 296(2)
_cell_measurement_reflns_used 2403
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_exptl_crystal_density_diffrn 1.179
_exptl_crystal_density_method 'not measured'
_exptl_crystal_F_000 1024
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_diffrn_reflns_limit_l_min           -34
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_reflns_number_gt                    1859
_reflns_threshold_expression       >2sigma(I)

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_refine_special_details
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Refinement of F^2^ against ALL reflections. The weighted R-factor wR and
goodness of fit S are based on F^2^, conventional R-factors R are based
on F, with F set to zero for negative F^2^. The threshold expression of
F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement. R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type               full
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```
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_atom_sites_solution_secondary       difmap
_atom_sites_solution_hydrogens       geom
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_refine_ls_extinction_method         none
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O1 O 0.87548(7) 0.11833(17) 0.74364(3) 0.0325(3) Uani 1 1 d . . .
C4 C 0.88757(9) 0.3750(2) 0.87046(5) 0.0266(3) Uani 1 1 d . . .
C10 C 0.86685(9) 0.3901(2) 1.03205(5) 0.0256(3) Uani 1 1 d . . .
C2 C 0.83323(10) 0.0902(2) 0.82103(5) 0.0292(3) Uani 1 1 d . . .
H2 H 0.8002 -0.0388 0.8167 0.035 Uiso 1 1 calc R . .
C3 C 0.83953(10) 0.1810(3) 0.86245(5) 0.0297(4) Uani 1 1 d . . .
H3 H 0.8107 0.1102 0.8858 0.036 Uiso 1 1 calc R . .
C1 C 0.87665(9) 0.1929(2) 0.78607(5) 0.0255(3) Uani 1 1 d . . .
C13 C 0.89115(9) 0.1258(2) 1.10709(5) 0.0270(3) Uani 1 1 d . . .
```

C14 C 0.85071(9) 0.3312(2) 1.10992(5) 0.0281(3) Uani 1 1 d . . .
H14 H 0.8315 0.3829 1.1371 0.034 Uiso 1 1 calc R . .
C6 C 0.92560(9) 0.3852(2) 0.79303(5) 0.0280(4) Uani 1 1 d . . .
H6 H 0.9549 0.4545 0.7697 0.034 Uiso 1 1 calc R . .
C9 C 0.85553(11) 0.5398(3) 0.99281(5) 0.0327(4) Uani 1 1 d . . .
H9A H 0.8013 0.6306 0.9975 0.039 Uiso 1 1 calc R . .
H9B H 0.9089 0.6363 0.9915 0.039 Uiso 1 1 calc R . .
C12 C 0.91897(10) 0.0547(2) 1.06600(5) 0.0292(4) Uani 1 1 d . . .
H12 H 0.9464 -0.0824 1.0632 0.035 Uiso 1 1 calc R . .
C15 C 0.83827(9) 0.4609(2) 1.07327(5) 0.0274(3) Uani 1 1 d . . .
H15 H 0.8104 0.5975 1.0762 0.033 Uiso 1 1 calc R . .
C7 C 0.89550(10) 0.4743(3) 0.91418(5) 0.0289(3) Uani 1 1 d . . .
H7 H 0.9407 0.5816 0.9175 0.035 Uiso 1 1 calc R . .
C11 C 0.90677(10) 0.1835(2) 1.02907(5) 0.0281(3) Uani 1 1 d . . .
H11 H 0.9256 0.1310 1.0019 0.034 Uiso 1 1 calc R . .
C8 C 0.84563(10) 0.4285(3) 0.94940(5) 0.0320(4) Uani 1 1 d . . .
H8 H 0.8011 0.3189 0.9470 0.038 Uiso 1 1 calc R . .
C5 C 0.93081(9) 0.4738(2) 0.83452(5) 0.0280(4) Uani 1 1 d . . .
H5 H 0.9640 0.6027 0.8387 0.034 Uiso 1 1 calc R . .
C16 C 0.90298(11) -0.0159(3) 1.14725(5) 0.0373(4) Uani 1 1 d . . .
H16A H 0.8756 0.0557 1.1721 0.056 Uiso 1 1 calc R . .
H16B H 0.9678 -0.0392 1.1526 0.056 Uiso 1 1 calc R . .
H16C H 0.8730 -0.1544 1.1426 0.056 Uiso 1 1 calc R . .
C17 C 0.82714(12) -0.0799(3) 0.73542(5) 0.0405(4) Uani 1 1 d . . .
H17A H 0.8520 -0.1940 0.7536 0.061 Uiso 1 1 calc R . .
H17B H 0.8342 -0.1196 0.7051 0.061 Uiso 1 1 calc R . .
H17C H 0.7626 -0.0602 0.7419 0.061 Uiso 1 1 calc R . .

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C4 0.0212(7) 0.0289(8) 0.0296(8) 0.0028(7) -0.0022(6) 0.0006(6)
C10 0.0219(7) 0.0271(8) 0.0279(8) -0.0016(6) -0.0021(6) -0.0004(6)
C2 0.0290(8) 0.0273(8) 0.0311(8) 0.0010(7) 0.0016(6) -0.0043(7)
C3 0.0287(8) 0.0322(8) 0.0282(8) 0.0053(6) 0.0033(6) -0.0058(7)
C1 0.0233(7) 0.0287(8) 0.0246(8) 0.0012(6) 0.0008(6) 0.0060(6)
C13 0.0224(7) 0.0294(8) 0.0292(8) 0.0009(7) -0.0022(6) -0.0043(6)
C14 0.0282(8) 0.0330(8) 0.0231(7) -0.0061(6) 0.0003(6) -0.0016(6)

C6 0.0235(7) 0.0314(8) 0.0290(8) 0.0093(7) 0.0026(6) -0.0005(6)
C9 0.0374(9) 0.0305(8) 0.0303(8) 0.0005(7) -0.0016(6) 0.0049(7)
C12 0.0287(7) 0.0250(7) 0.0341(8) -0.0024(7) 0.0011(6) 0.0032(6)
C15 0.0252(7) 0.0240(7) 0.0330(8) -0.0062(6) -0.0010(6) 0.0018(6)
C7 0.0260(7) 0.0291(8) 0.0317(8) 0.0024(7) -0.0041(6) -0.0021(6)
C11 0.0287(7) 0.0297(8) 0.0257(7) -0.0057(7) 0.0023(6) 0.0014(7)
C8 0.0308(8) 0.0352(9) 0.0299(8) 0.0035(7) -0.0040(6) -0.0029(7)
C5 0.0226(7) 0.0277(8) 0.0339(8) 0.0047(7) -0.0020(6) -0.0033(6)
C16 0.0399(9) 0.0373(9) 0.0346(9) 0.0047(8) -0.0012(7) -0.0014(8)
C17 0.0569(11) 0.0317(9) 0.0330(9) -0.0062(7) 0.0027(7) -0.0050(8)

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;
All esds (except the esd in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell esds are taken
into account individually in the estimation of esds in distances, angles
and torsion angles; correlations between esds in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell esds is used for estimating esds involving l.s. planes.
;

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O1 C17 1.4189(19) . ?
C4 C3 1.394(2) . ?
C4 C5 1.397(2) . ?
C4 C7 1.469(2) . ?
C10 C11 1.390(2) . ?
C10 C15 1.391(2) . ?
C10 C9 1.514(2) . ?
C2 C3 1.382(2) . ?
C2 C1 1.386(2) . ?
C1 C6 1.386(2) . ?
C13 C12 1.385(2) . ?
C13 C14 1.386(2) . ?
C13 C16 1.509(2) . ?
C14 C15 1.382(2) . ?
C6 C5 1.378(2) . ?
C9 C8 1.495(2) . ?

C12 C11 1.385(2) . ?
C7 C8 1.322(2) . ?

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C1 O1 C17 117.12(12) . . ?
C3 C4 C5 116.82(14) . . ?
C3 C4 C7 123.26(13) . . ?
C5 C4 C7 119.91(13) . . ?
C11 C10 C15 117.63(14) . . ?
C11 C10 C9 122.75(13) . . ?
C15 C10 C9 119.61(13) . . ?
C3 C2 C1 119.41(14) . . ?
C2 C3 C4 122.26(14) . . ?
O1 C1 C6 115.58(12) . . ?
O1 C1 C2 124.70(13) . . ?
C6 C1 C2 119.72(13) . . ?
C12 C13 C14 117.51(14) . . ?
C12 C13 C16 121.42(14) . . ?
C14 C13 C16 121.07(13) . . ?
C15 C14 C13 121.54(14) . . ?
C5 C6 C1 120.03(13) . . ?
C8 C9 C10 115.80(13) . . ?
C11 C12 C13 121.37(14) . . ?
C14 C15 C10 120.93(13) . . ?
C8 C7 C4 127.40(14) . . ?
C12 C11 C10 121.00(13) . . ?
C7 C8 C9 124.82(15) . . ?
C6 C5 C4 121.75(14) . . ?

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_diffrn_reflns_theta_full 25.00
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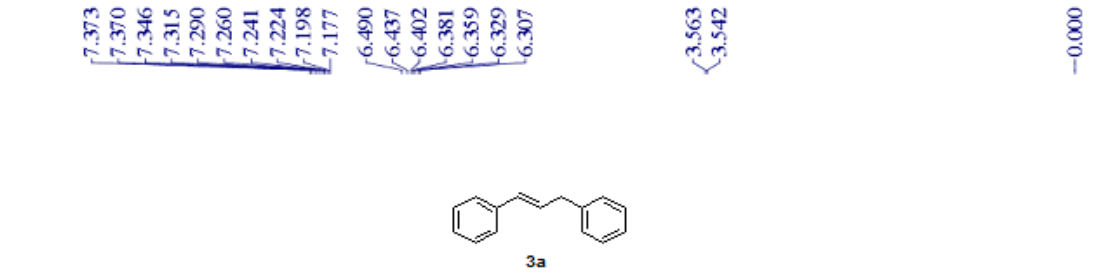
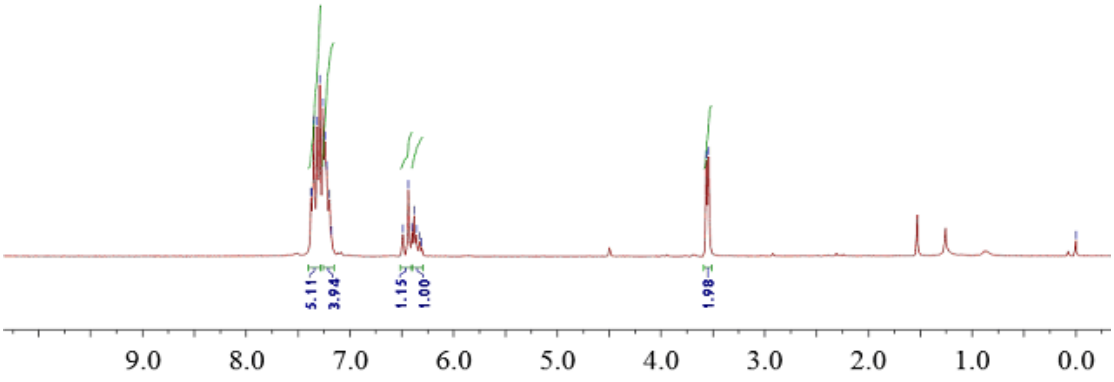
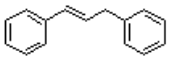
Copy of HRMS and NMR Spectra for desired products:

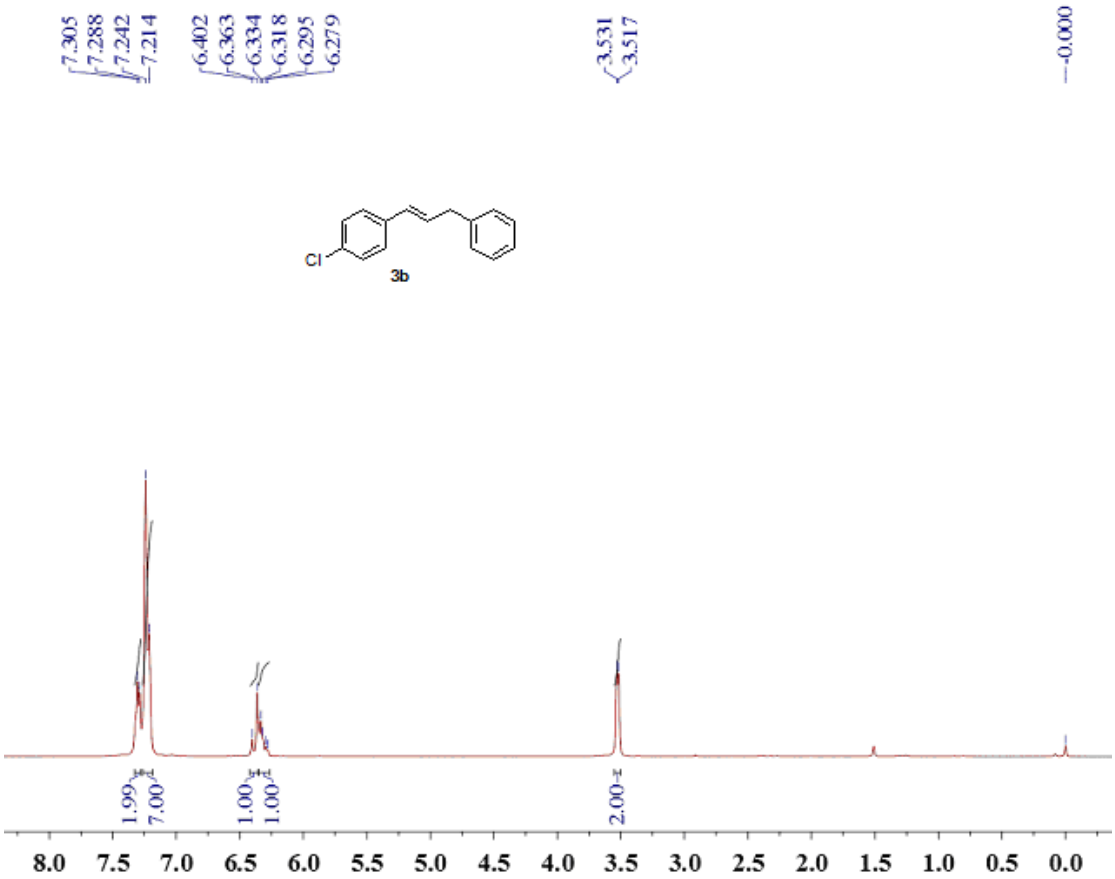


3a





3a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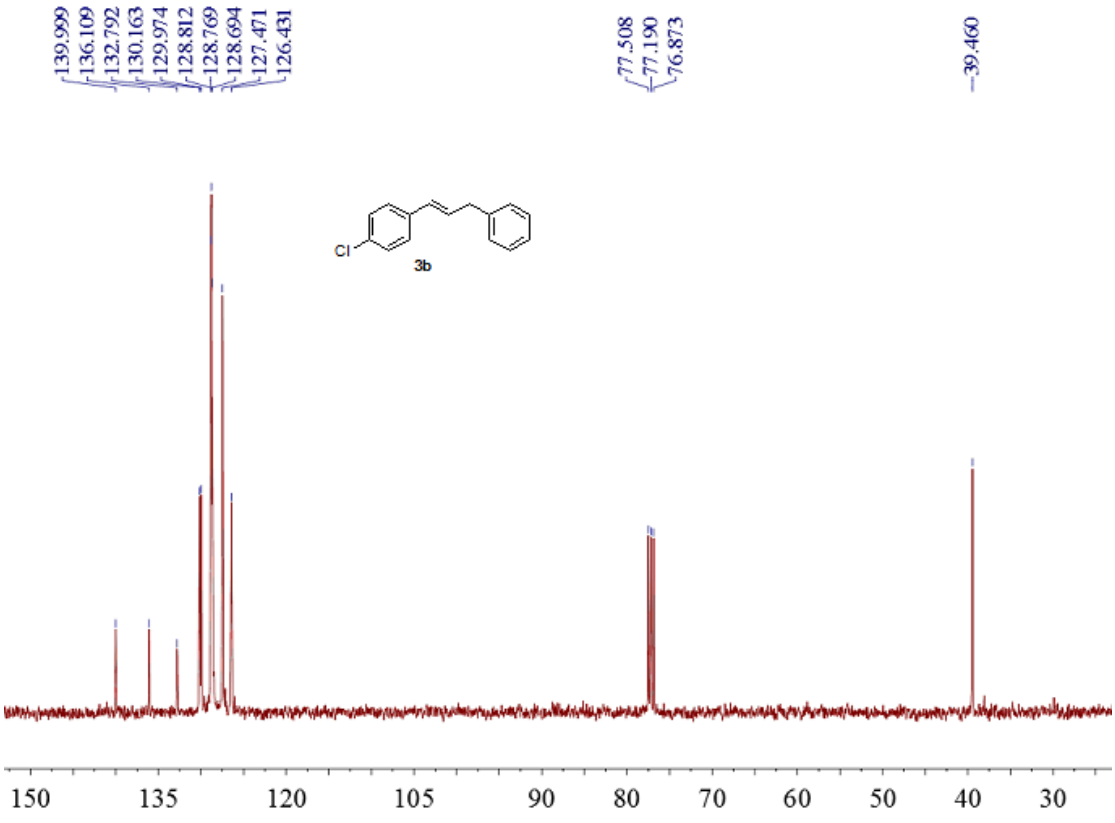
6.334
6.318
6.295
6.279
3.531
3.517
-0.000
Cl
3b
1.99
7.00
1.00
1.00
2.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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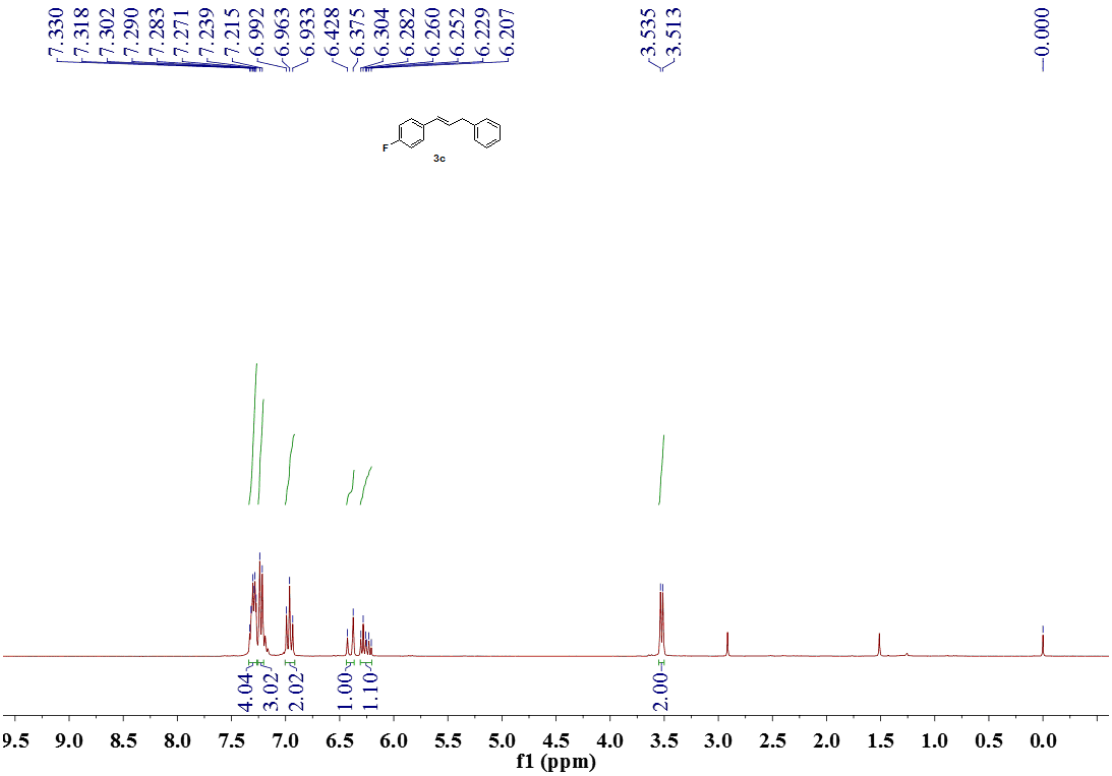
76.873
39.460
Cl
3b
150 135 120 105 90 80 70 60 50 40 30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7.271
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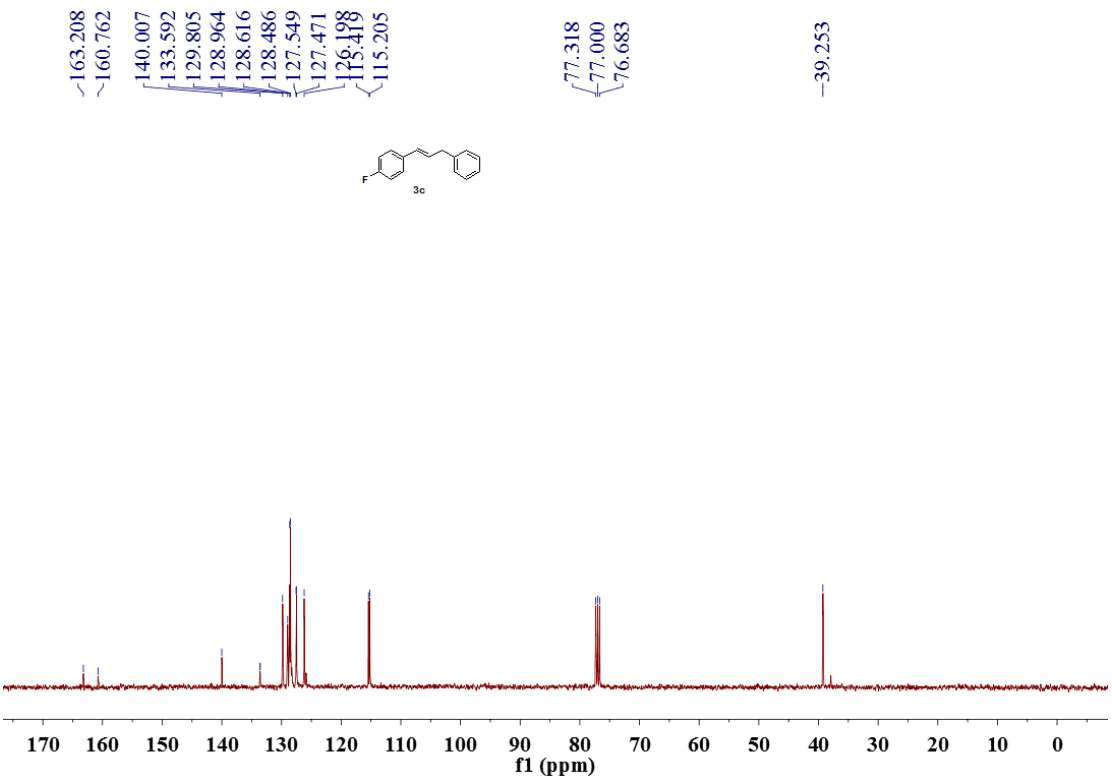
6.963
6.933
6.428
6.375
6.304
6.282
6.260
6.252
6.229
6.207
3.535
3.513
0.000
3c
4.04
3.02
2.02
1.00
1.10
2.00
f1 (ppm)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128.616
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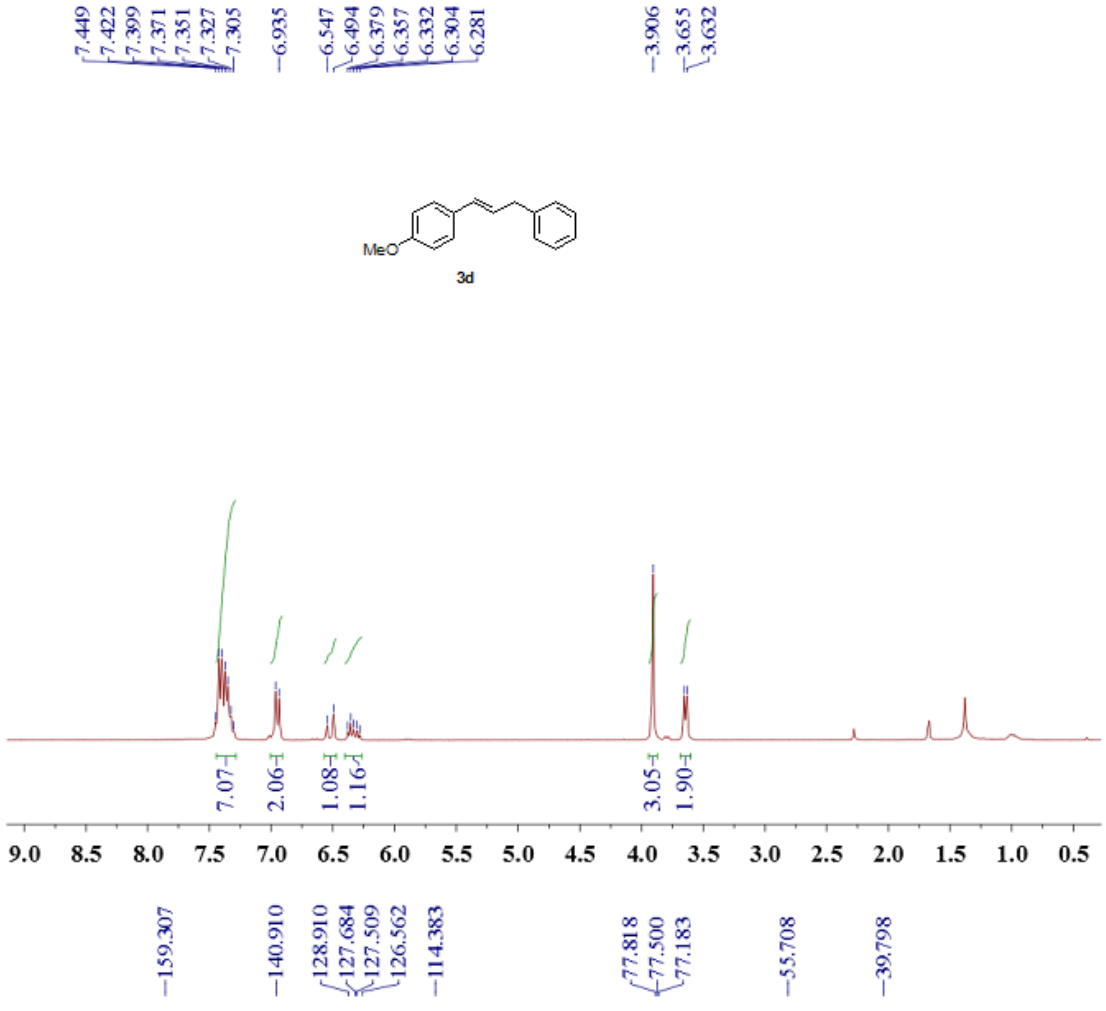
115.205
77.318
77.000
76.683
39.253
3c
f1 (ppm)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6.494
6.379
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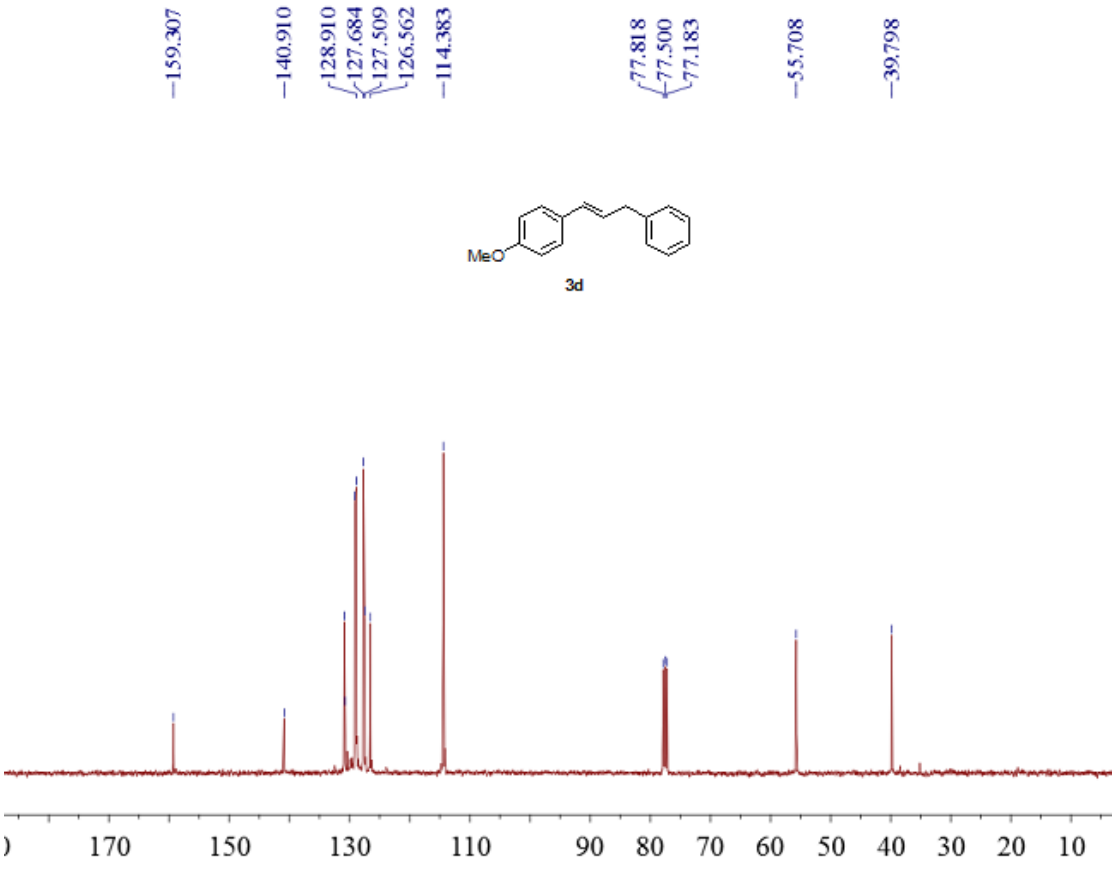
6.332
6.304
6.281
3.906
3.655
3.632
MeO
3d
7.07
2.06
1.08
1.16
3.05
1.90
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5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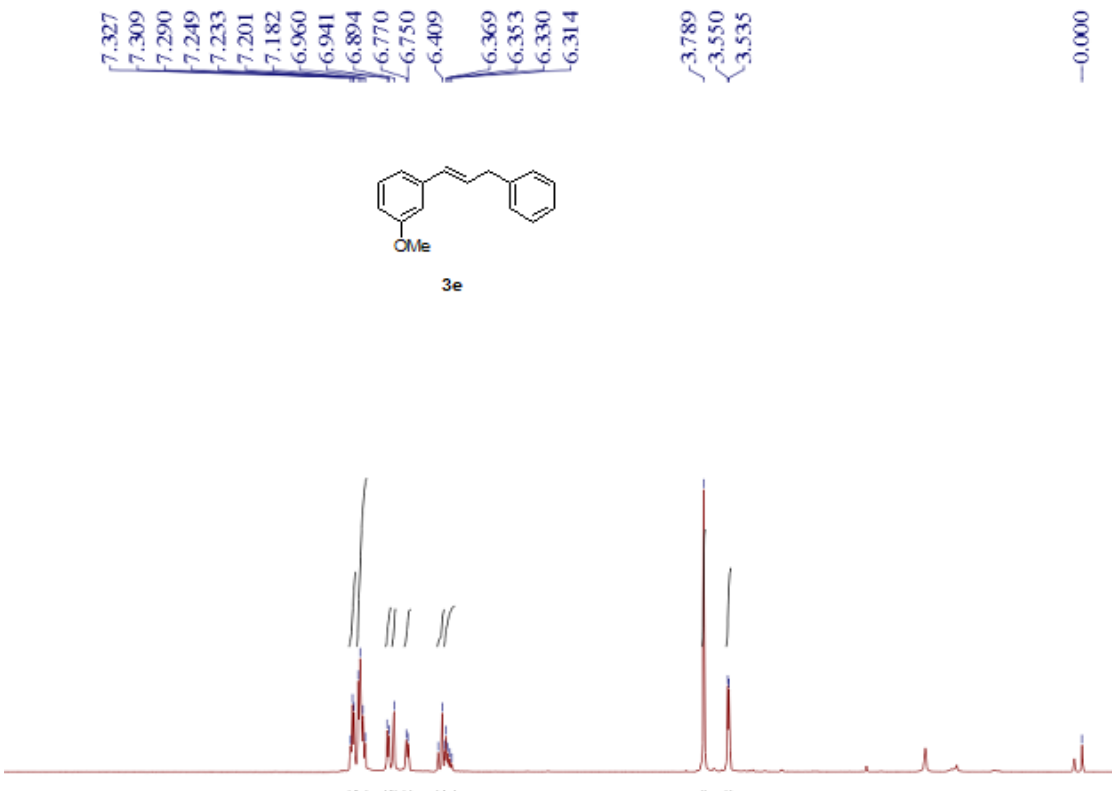
39.798
MeO
3d
170 150 130 110 90 80 70 60 50 40 30 20 10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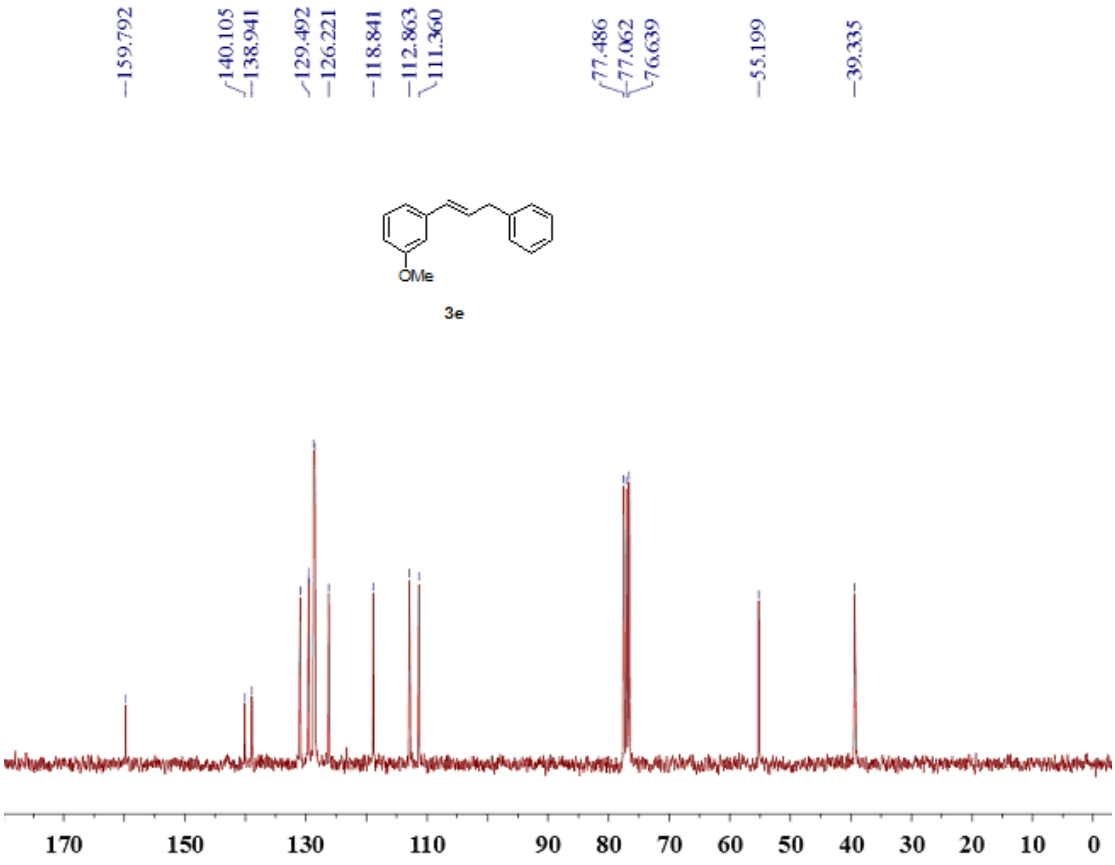
6.894
6.770
6.750
6.409
6.369
6.353
6.330
6.314
3.789
3.550
3.535
0.000
OMe
3e
2.00
4.49
1.05
1.02
1.00
1.00
1.08
3.14
2.11
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0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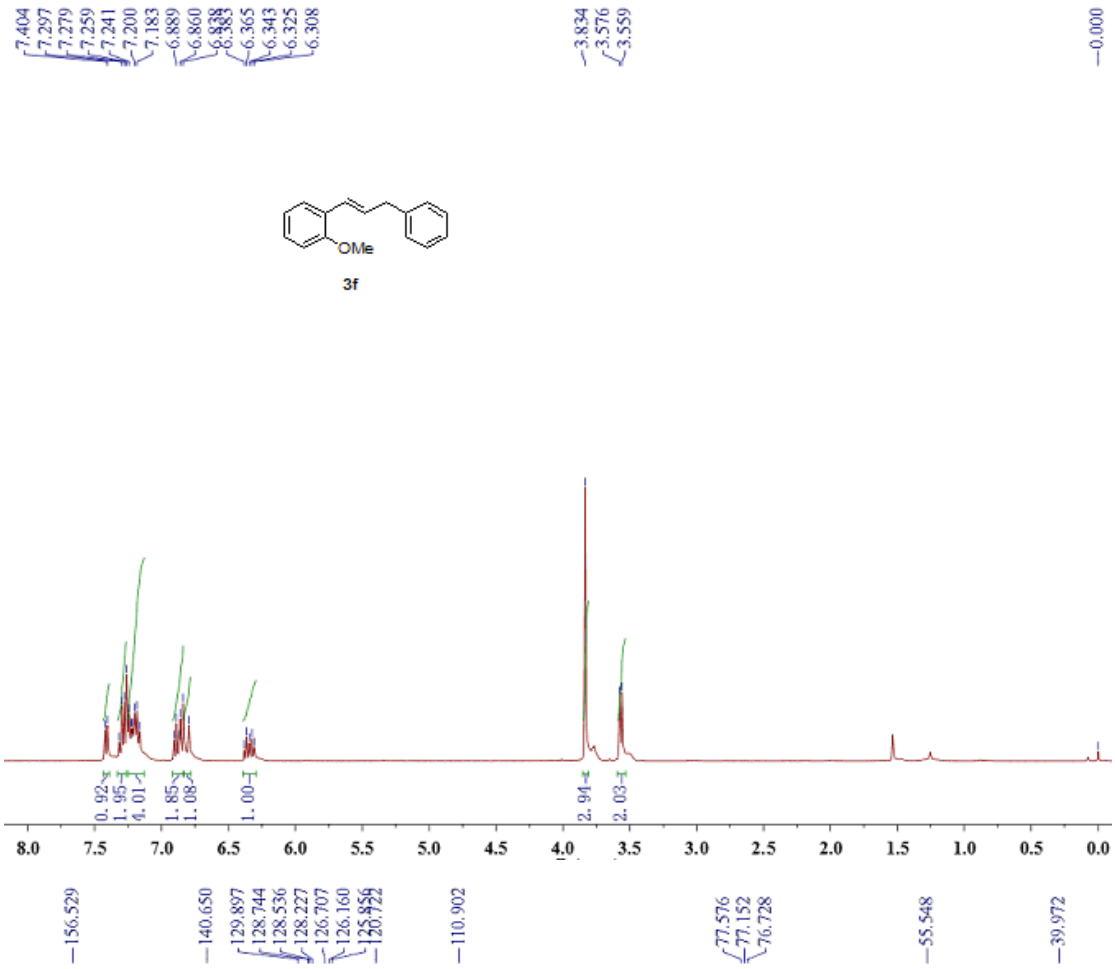
OMe
3e
170
150
130
110
90
80
70
60
50
40
30
20
10
0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6.308
3.834
3.576
3.559
0.000
OMe
3f
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1.95
4.01
1.85
1.08
1.00
2.94
2.03
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
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140.650
129.897
128.744
128.536
128.227
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126.160
110.902
77.576
77.152
76.728
55.548
39.972
OMe
3f
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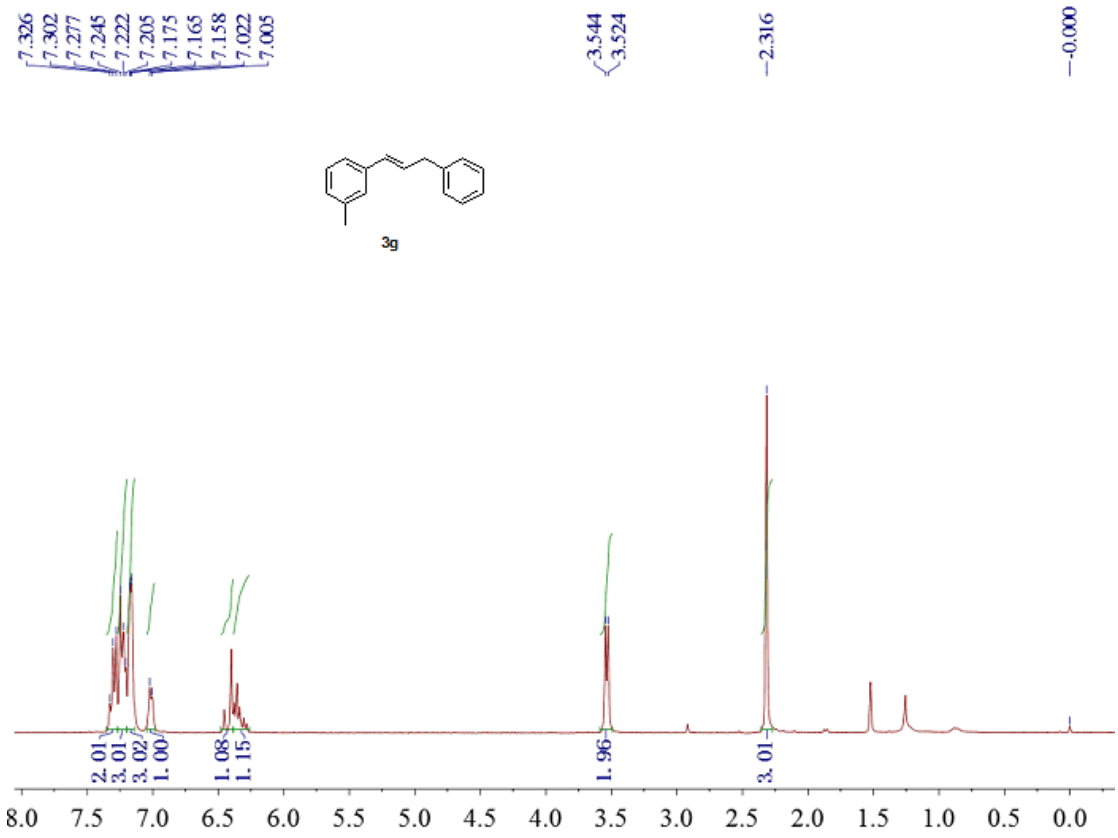
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7.165
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7.022
7.005
3.544
3.524
2.316
0.000
3g
2.01
3.01
3.02
1.00
1.08
1.15
1.96
3.01
8.0
7.5
7.0
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6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


140.372
138.147
137.547
131.261
129.149
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123.419
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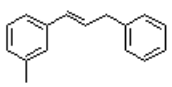
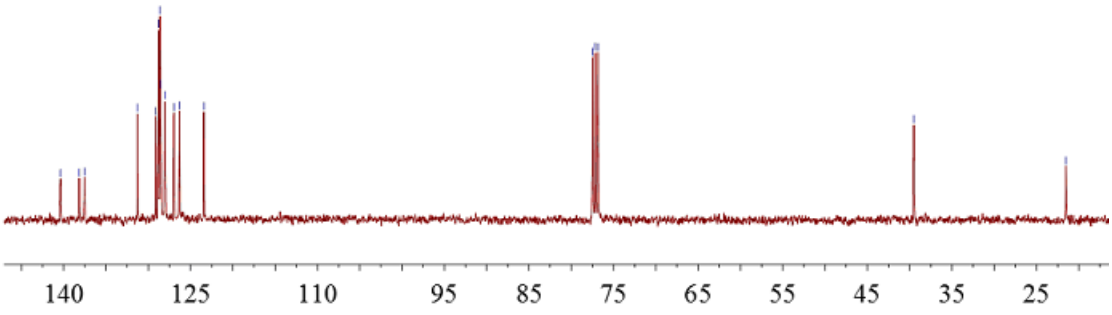
21.526



3g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125
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95
85
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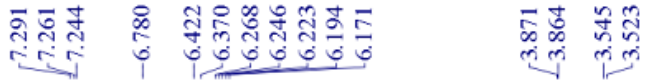
35
25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7.244
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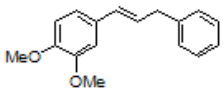
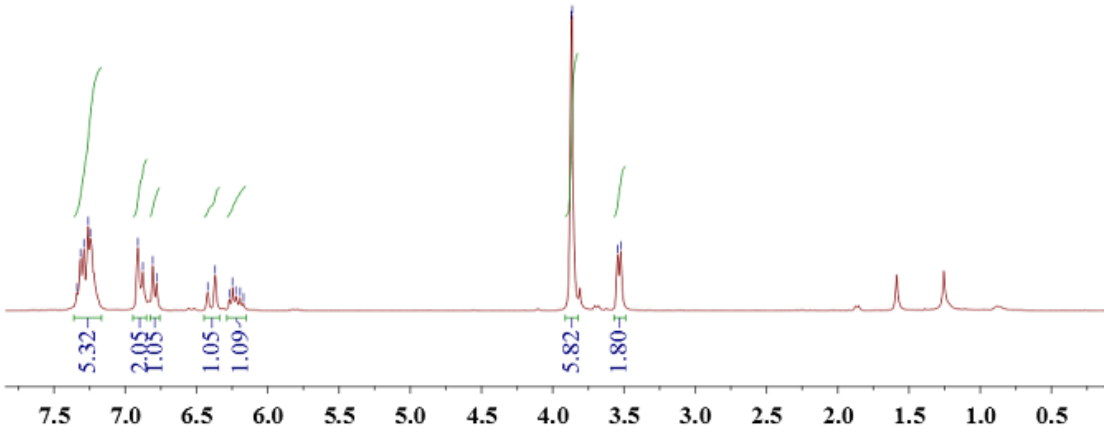
6.246
6.223
6.194
6.171
3.871
3.864
3.545
3.523


MeO
OMe

3h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1.05
1.05
1.09
5.82
1.80
7.5
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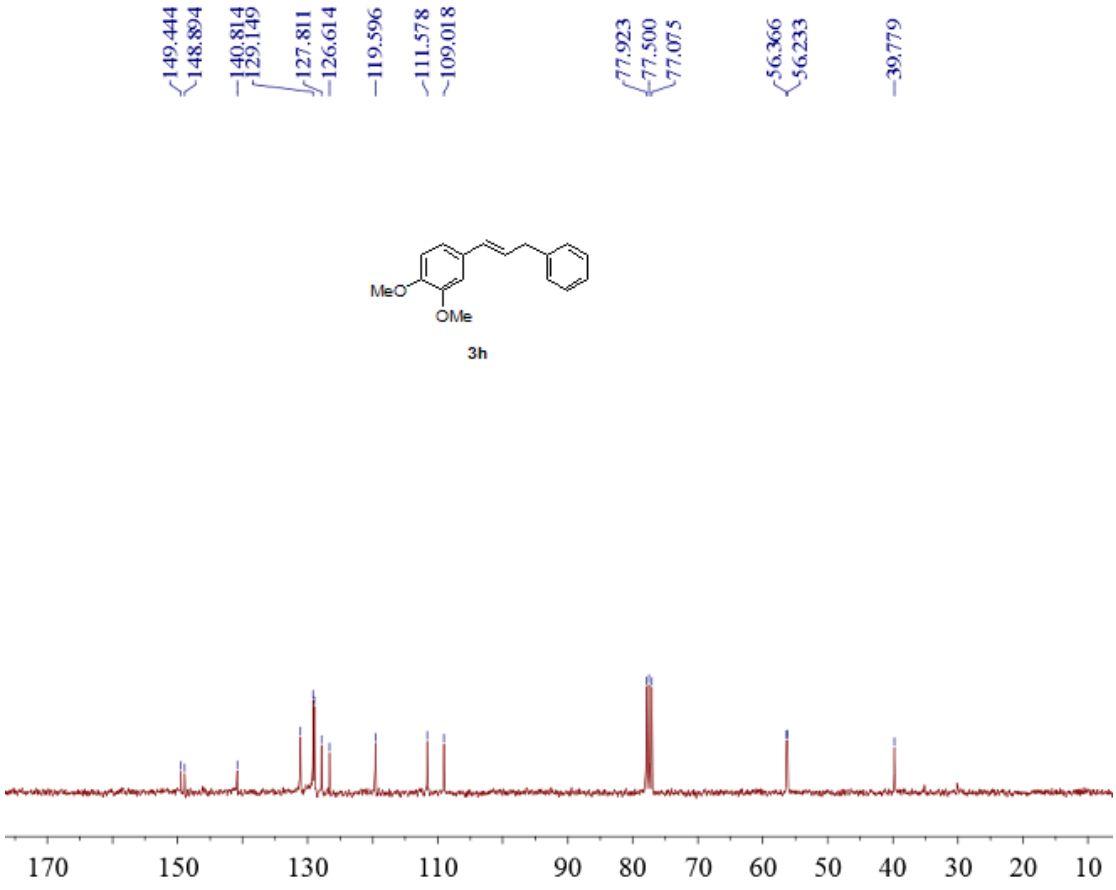
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5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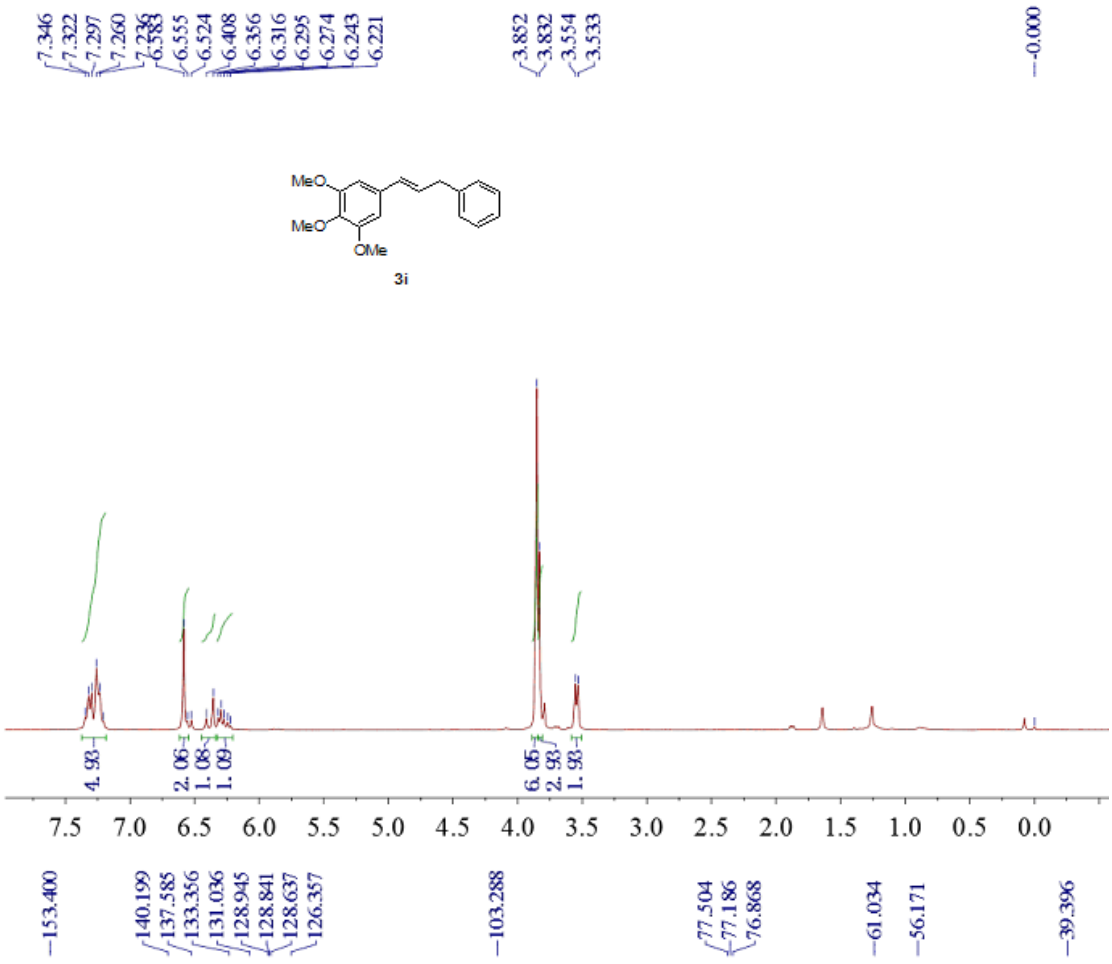
148.894
140.814
129.149
127.811
126.614
119.596
111.578
109.018
77.923
77.500
77.075
56.366
56.233
39.779
MeO
OMe
3h
170
150
130
110
90
80
70
60
50
40
30
20
10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7.322
7.297
7.260
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6.524
6.408
6.356
6.316
6.295
6.274
6.243
6.221
3.852
3.832
3.554
3.533
0.000
MeO
MeO
OMe
3i
4.93
2.06
1.08
1.09
6.05
2.93
1.93
7.5
7.0
6.5
6.0
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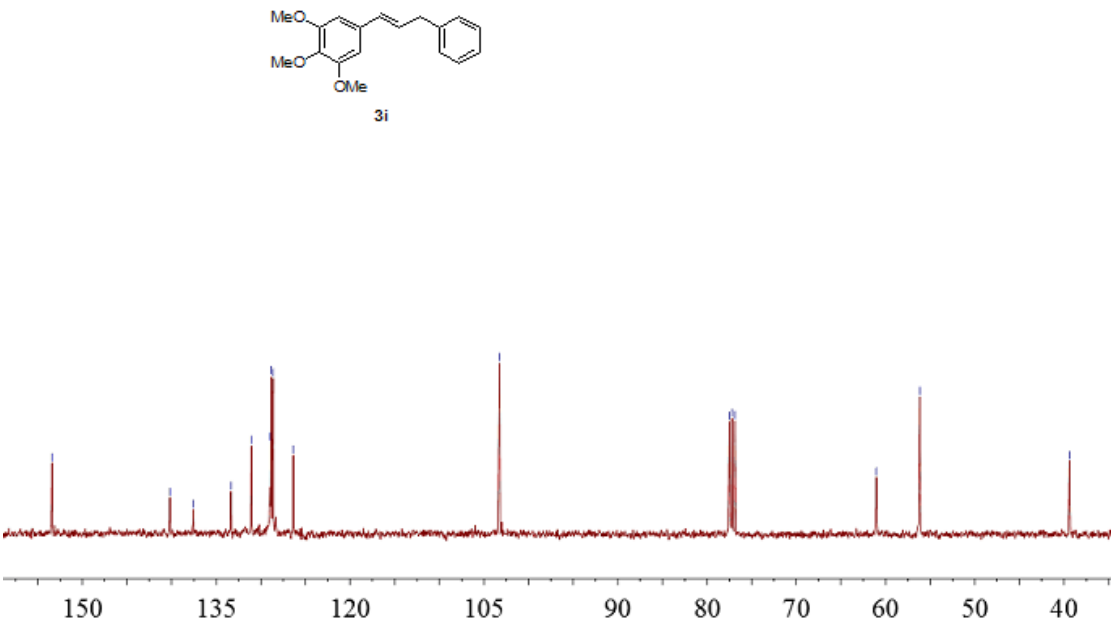
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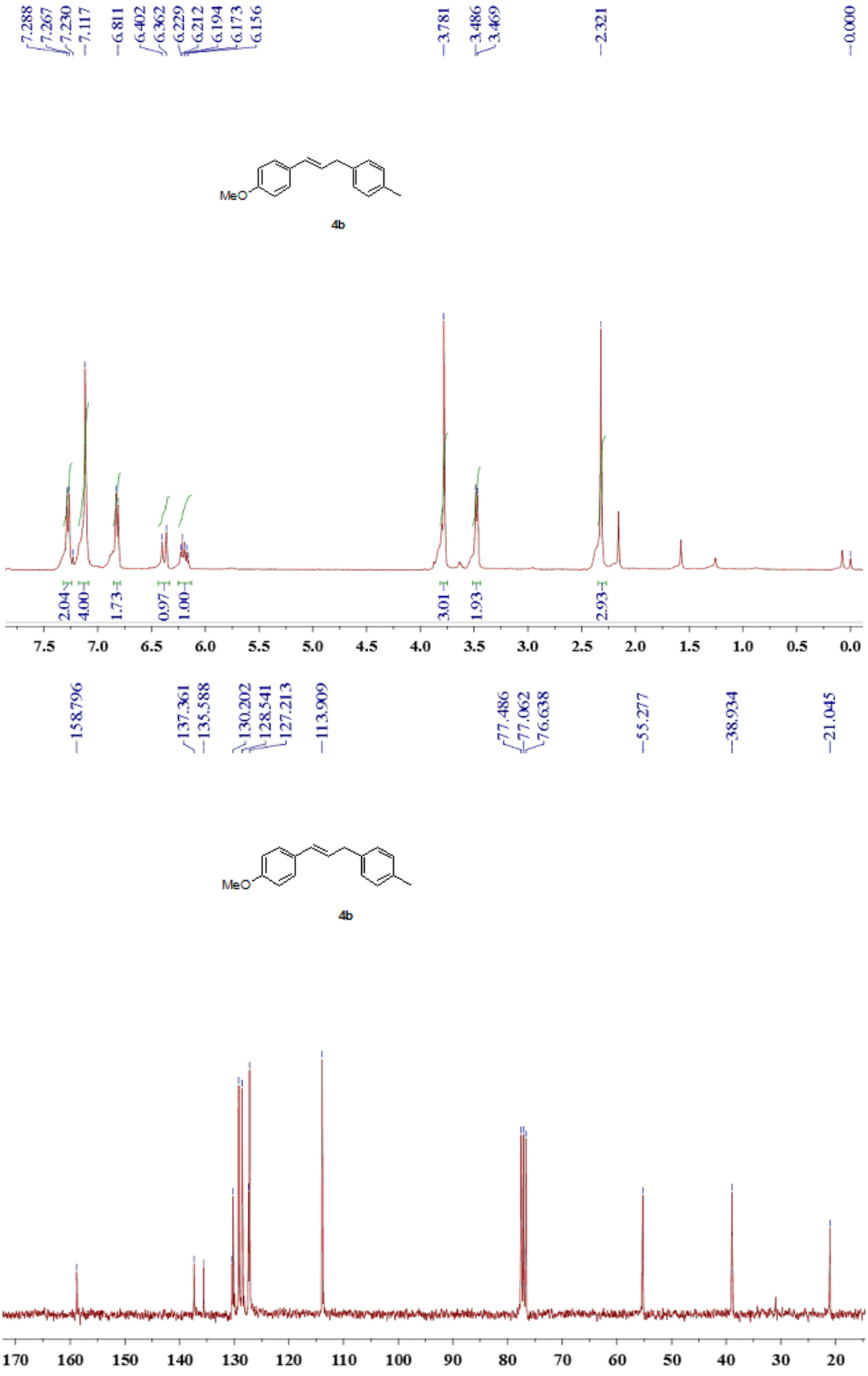
128.945
128.841
128.637
126.357
103.288
77.504
77.186
76.868
61.034
56.171
39.396
MeO
MeO
OMe
3i
150
135
120
105
90
80
70
60
50
40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7.267
7.230
7.117
6.811
6.402
6.362
6.229
6.212
6.194
6.173
6.156
3.781
3.486
3.469
2.321
0.000
MeO
4b
2.04
4.00
1.73
0.97
1.00
3.01
1.93
2.93
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
158.796
137.361
135.588
130.202
128.541
127.213
113.909
77.486
77.062
76.638
55.277
38.934
21.045
MeO
4b
170
160
150
140
130
120
110
100
90
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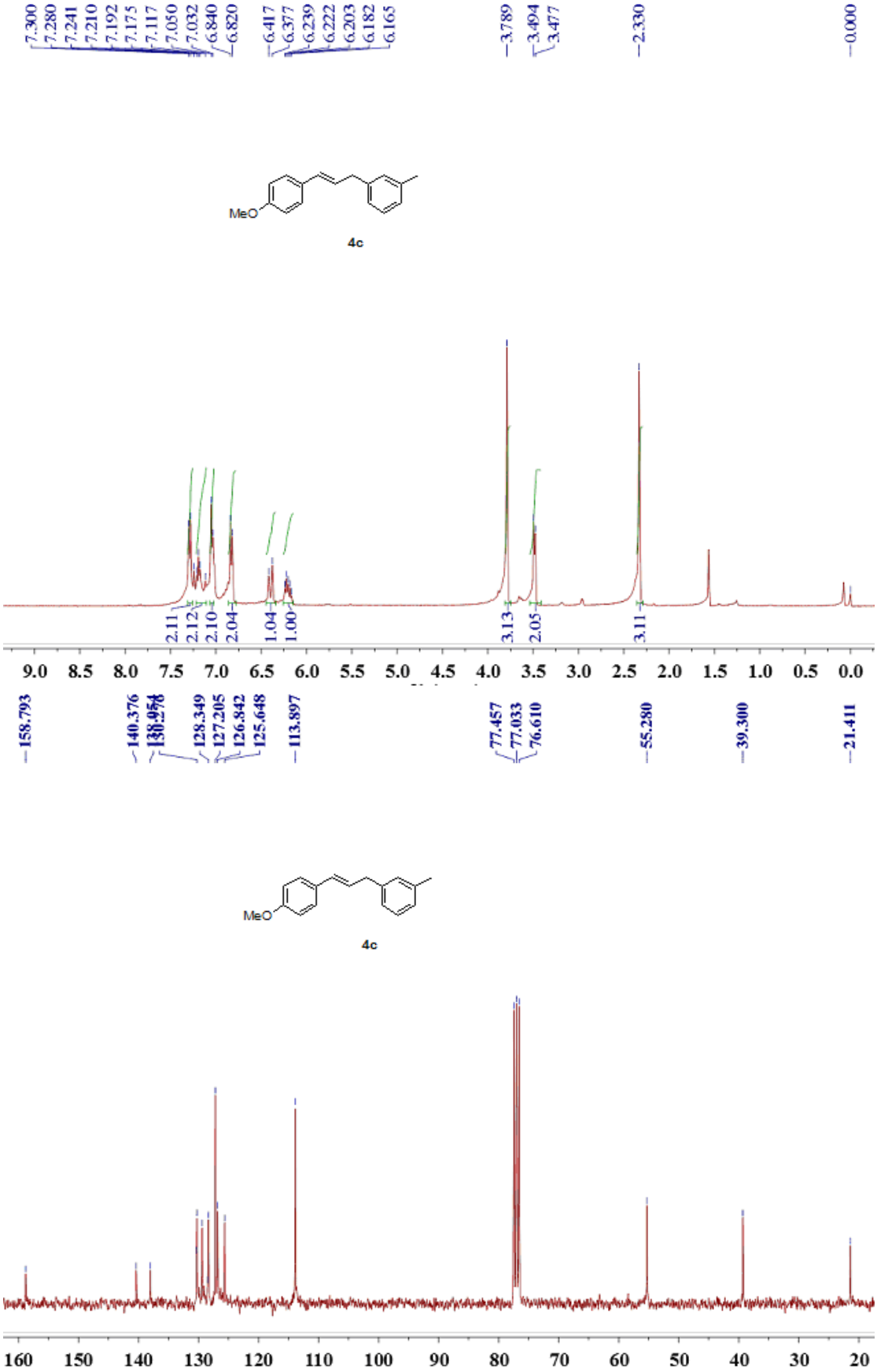
50
40
30
20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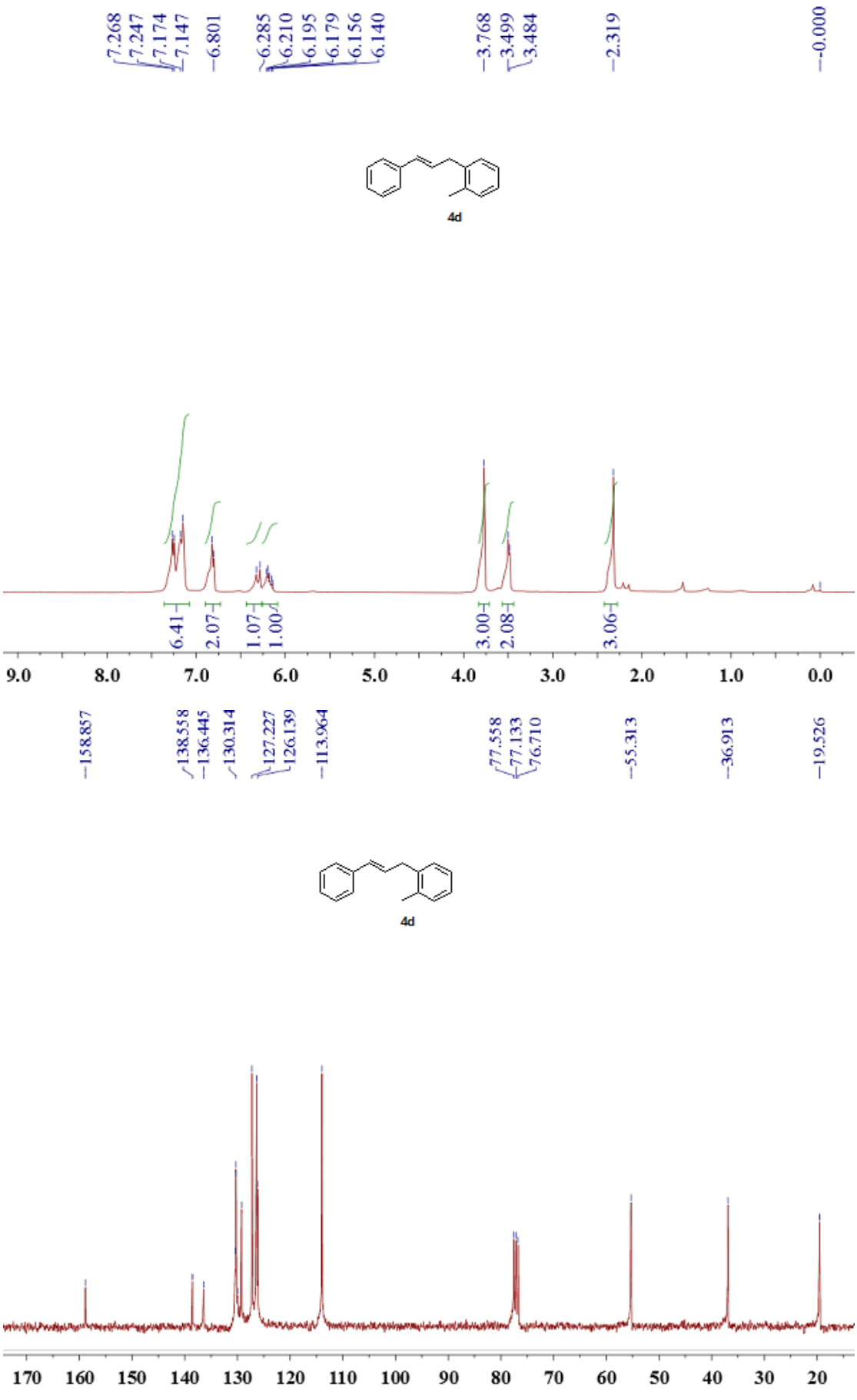
7.280
7.241
7.210
7.192
7.175
7.117
7.050
7.032
6.840
6.820
6.417
6.377
6.239
6.222
6.203
6.182
6.165
3.789
3.494
3.477
2.330
0.000
MeO
4c
2.11
2.12
2.10
2.04
1.04
1.00
3.13
2.05
3.11
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
158.793
140.376
128.349
127.205
126.842
125.648
113.897
77.457
77.033
76.610
55.280
39.300
21.411
MeO
4c
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20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7.247
7.174
7.147
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6.285
6.210
6.195
6.179
6.156
6.140
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3.499
3.484
2.319
0.000
4d
6.41
2.07
1.07
1.00
3.00
2.08
3.06
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
158.857
138.558
136.445
130.314
127.227
126.139
113.964
77.558
77.133
76.710
55.313
36.913
19.526
4d
170
160
150
140
130
120
110
100
90
80
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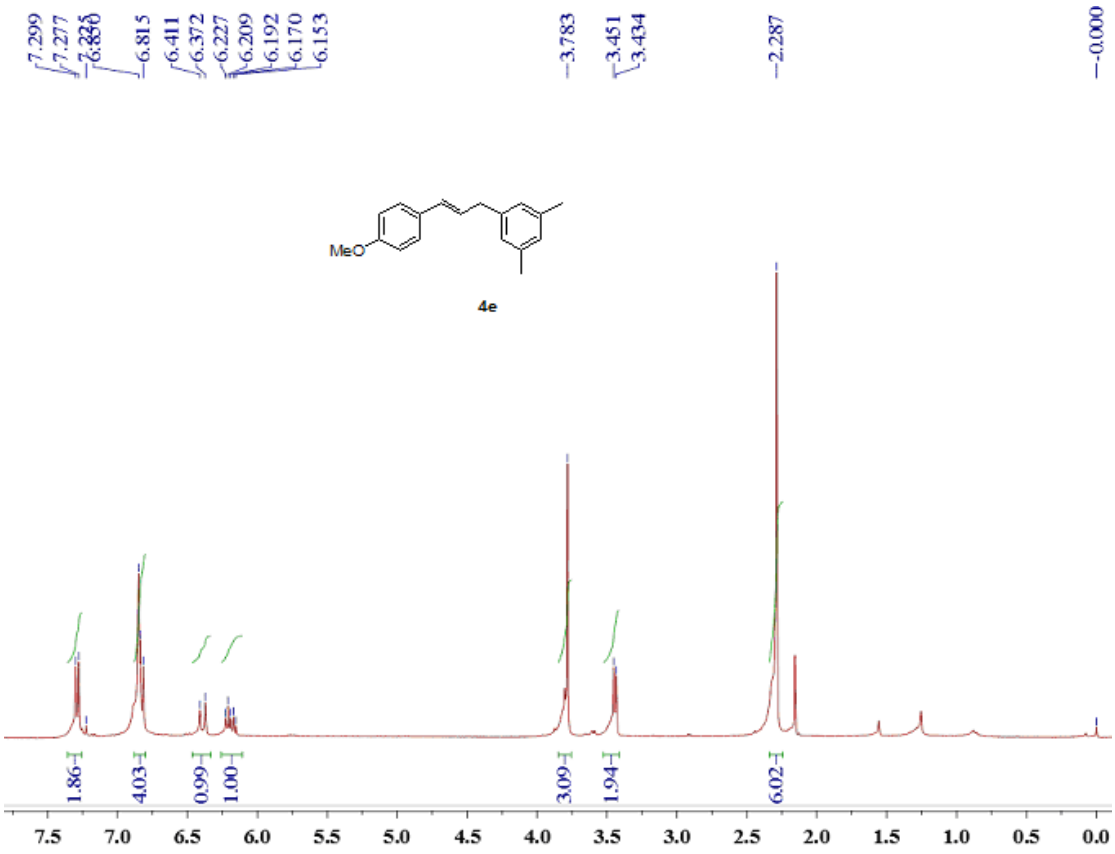
60
50
40
30
20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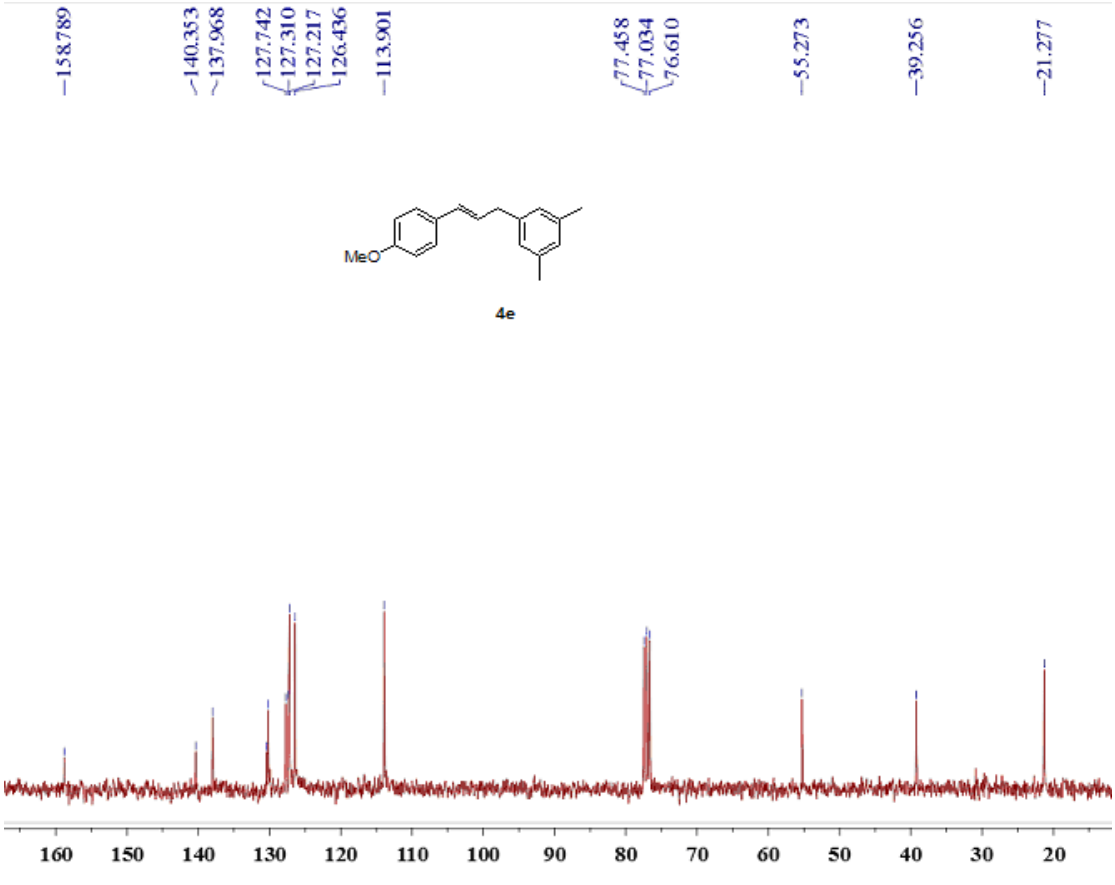
6.411
6.372
6.227
6.209
6.192
6.170
6.153
3.783
3.451
3.434
2.287
0.000
MeO
4e
1.86
4.03
0.99
1.00
3.09
1.94
6.02
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0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137.968
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127.310
127.217
126.436
113.901
77.458
77.034
76.610
55.273
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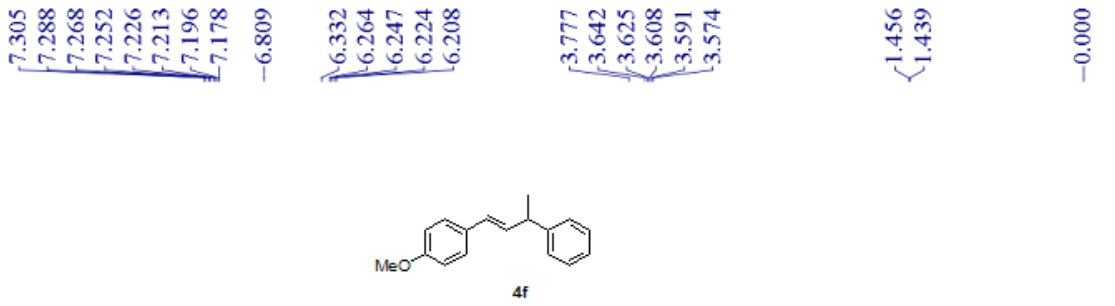
21.277
MeO
4e
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20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7.252
7.226
7.213
7.196
7.178
6.809
6.332
6.264
6.247
6.224
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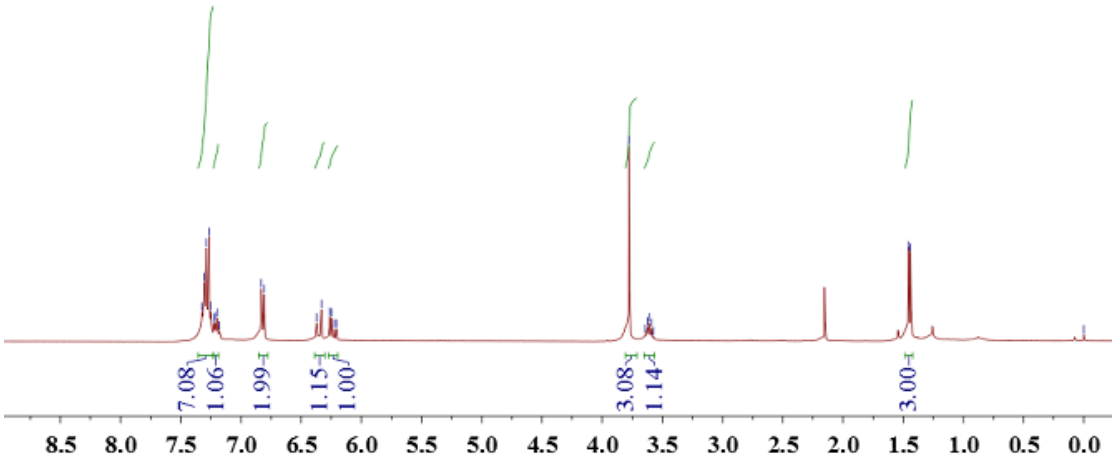
3.642
3.625
3.608
3.591
3.574
1.456
1.439
0.000

MeO

4f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1.06
1.99
1.15
1.00
3.08
1.14
3.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


158.802
145.901
133.139
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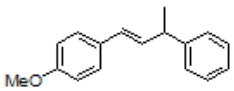
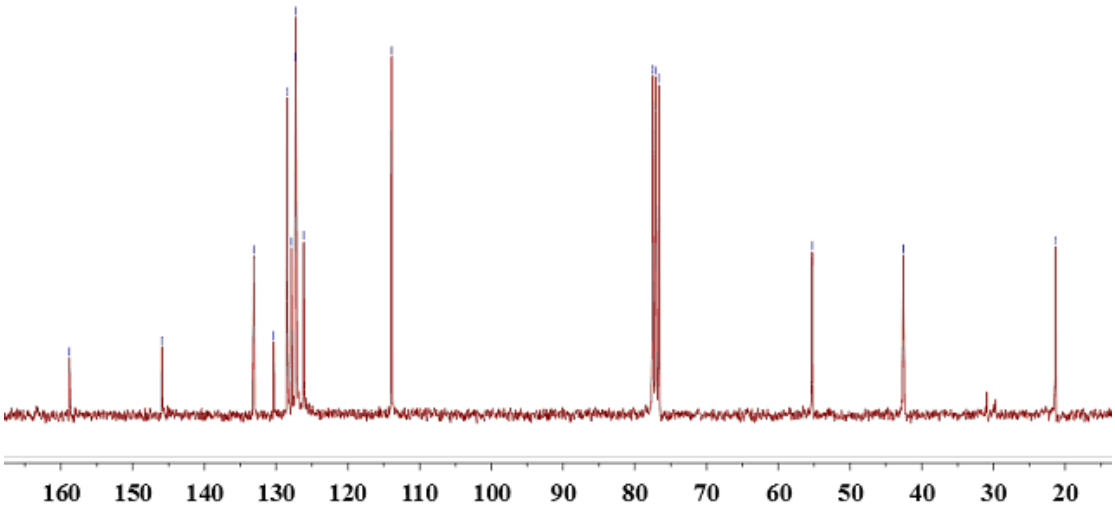
21.351


MeO

4f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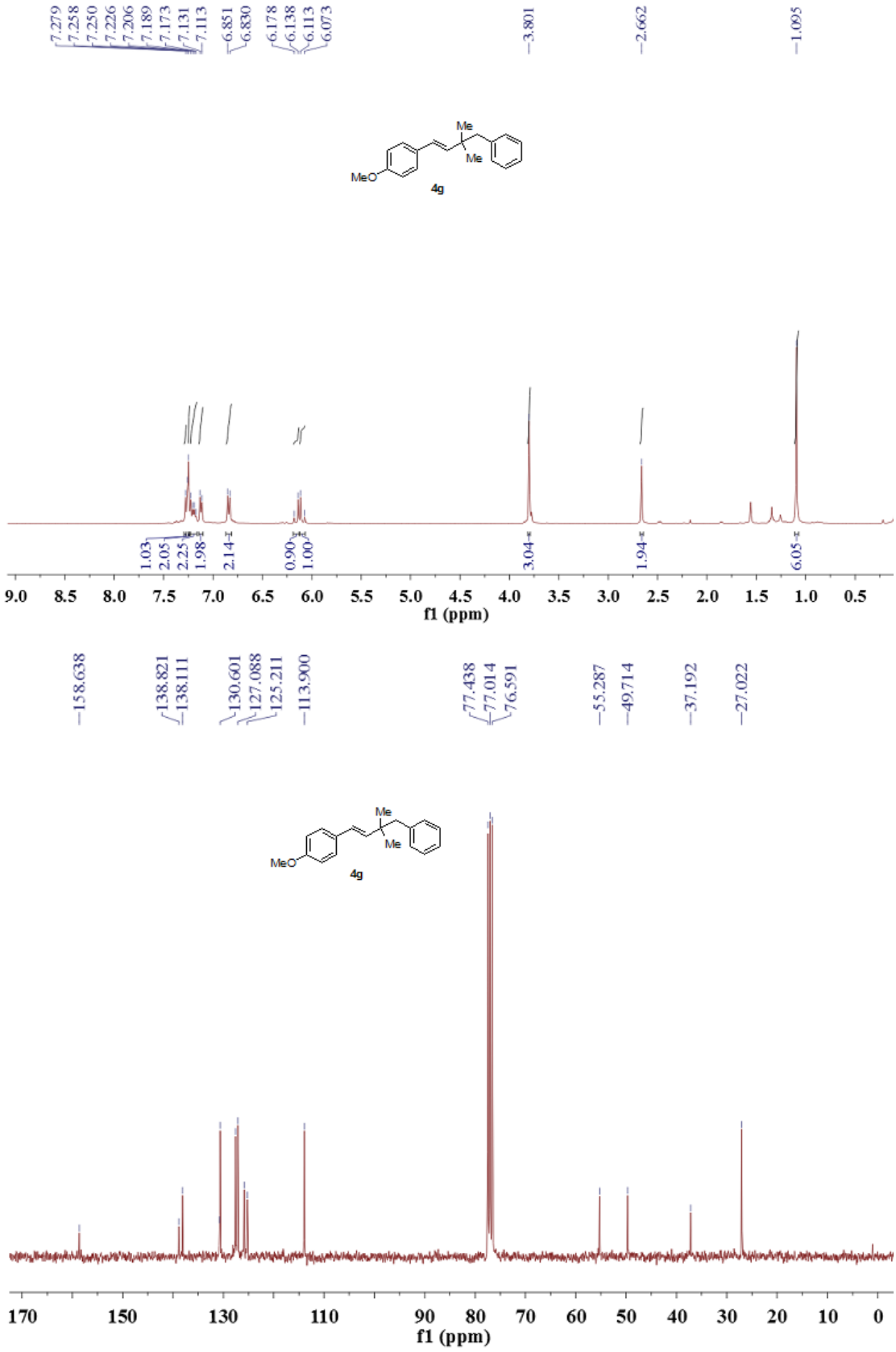
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7.173
7.131
7.113
6.851
6.830
6.178
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6.113
6.073
3.801
2.662
1.095
Me
Me
MeO
4g
1.03
2.05
2.25
1.98
2.14
0.90
1.00
3.04
1.94
6.05
f1 (ppm)
158.638
138.821
138.111
130.601
127.088
125.211
113.900
77.438
77.014
76.591
55.287
49.714
37.192
27.022
f1 (ppm)


7.287
7.268
7.248
7.164
7.143
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6.393
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6.190
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6.156
6.134
6.117
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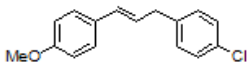
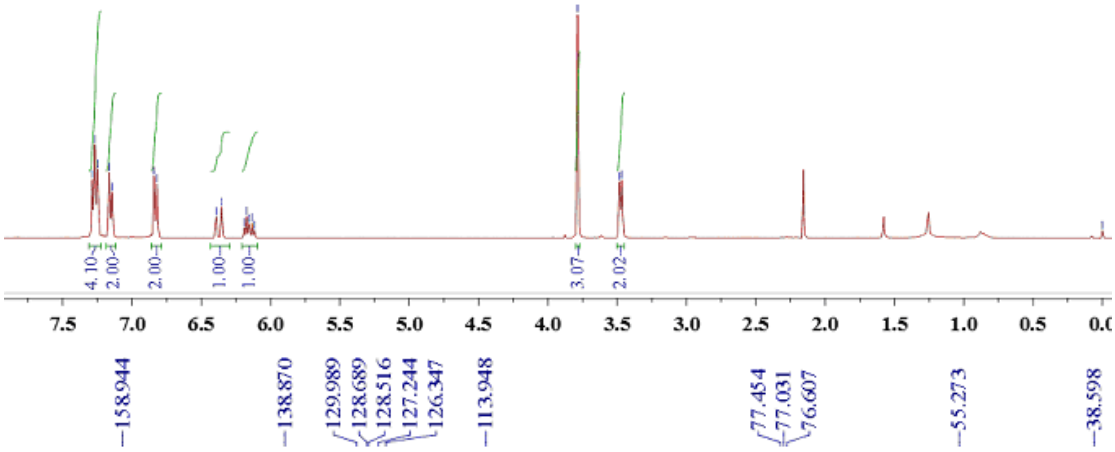
0.000


MeO
Cl

4h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7.5
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6.0
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5.0
4.5
4.0
3.5
3.0
2.5
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1.0
0.5
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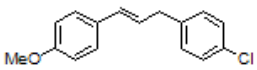
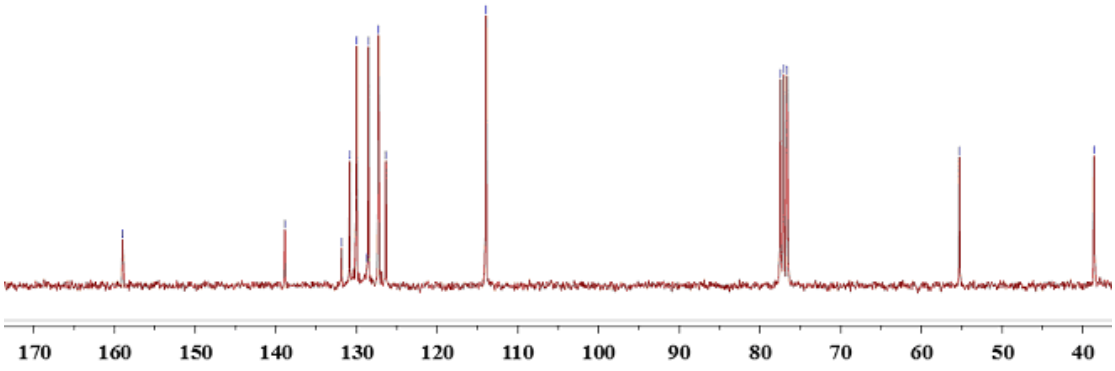
38.598


MeO
Cl

4h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160
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110
100
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80
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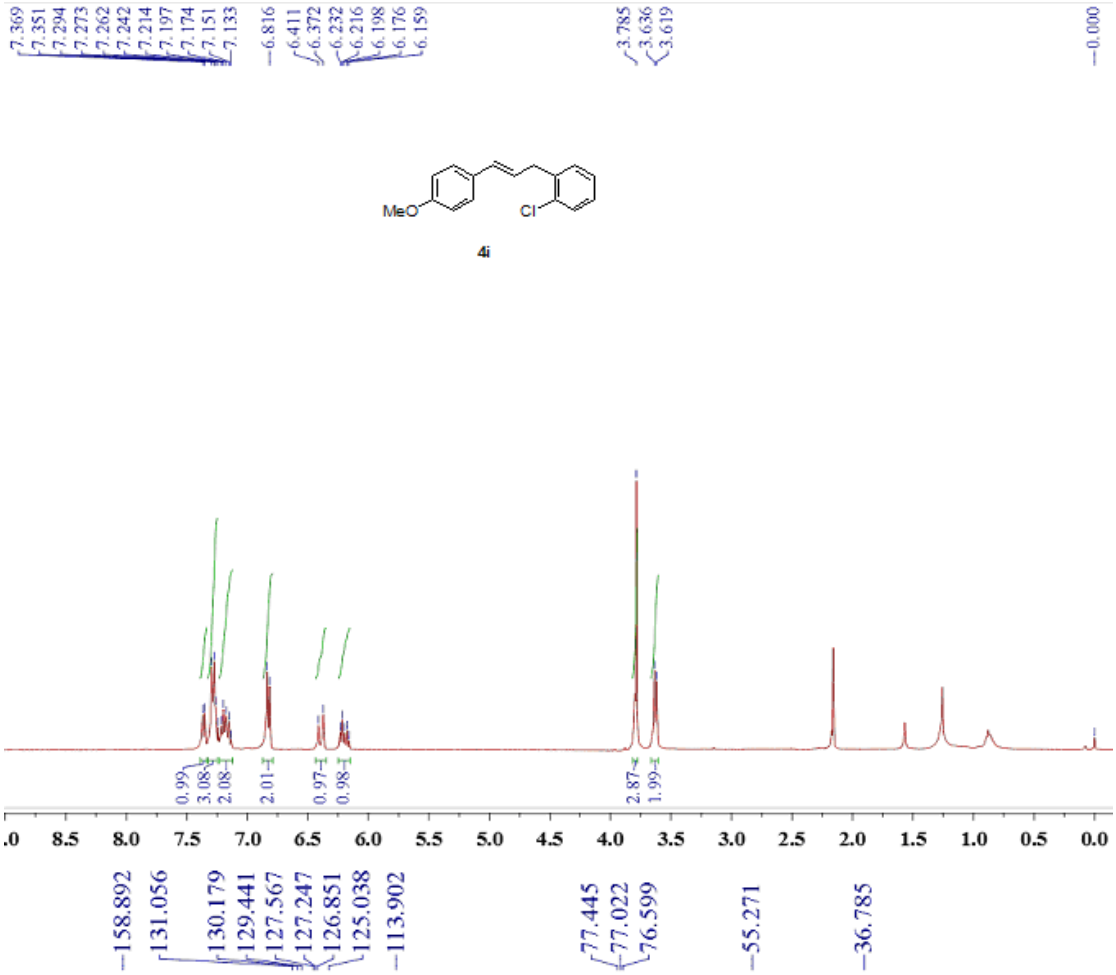
50
40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7.151
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6.411
6.372
6.232
6.216
6.198
6.176
6.159
3.785
3.636
3.619
0.000
MeO
Cl
4i
0.99
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2.08
2.01
0.97
0.98
2.87
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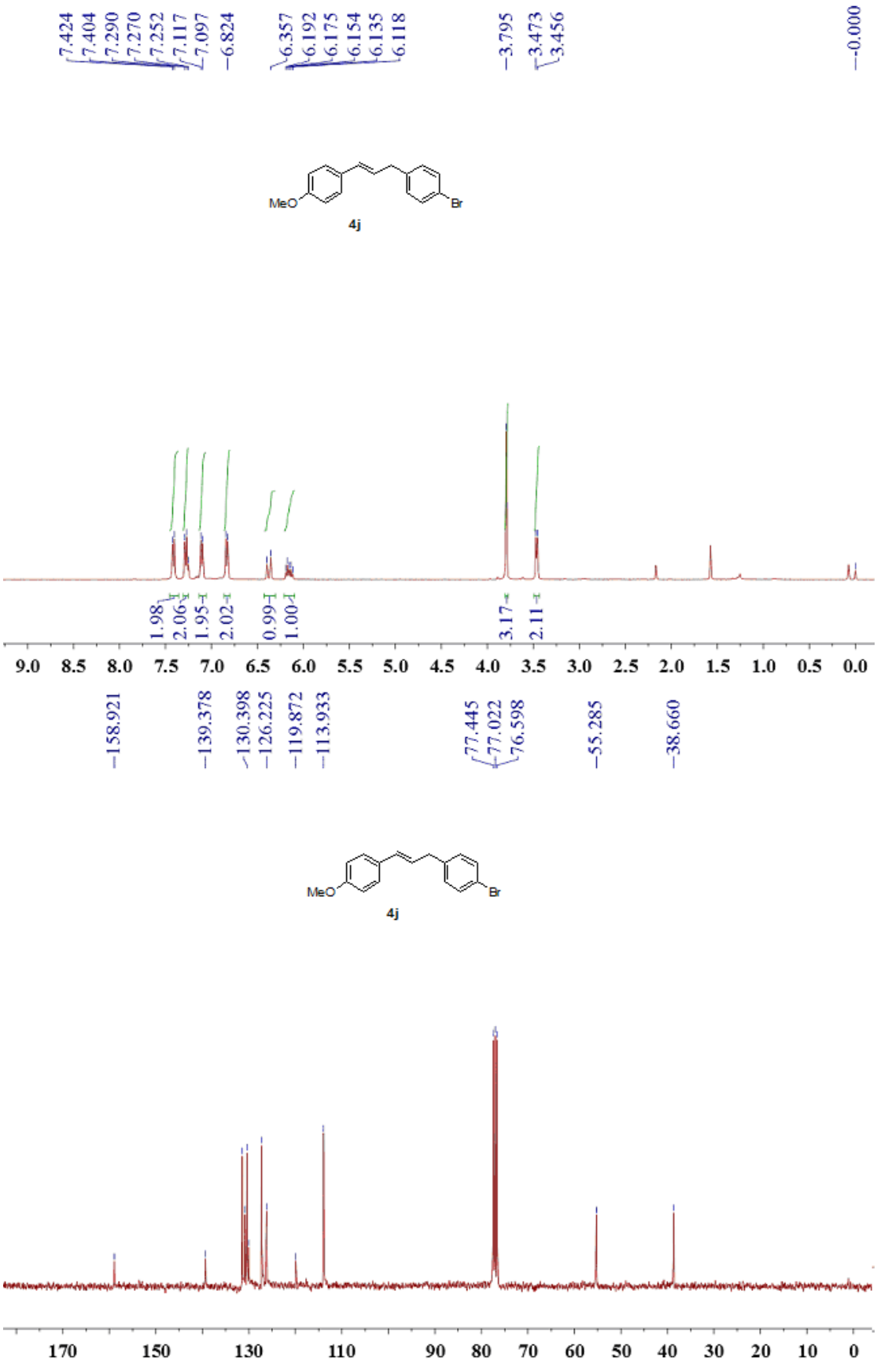
MeO
Cl
4i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6.118
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3.473
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0.000
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Br
4j
1.98
2.06
1.95
2.02
0.99
1.00
3.17
2.11
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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Br
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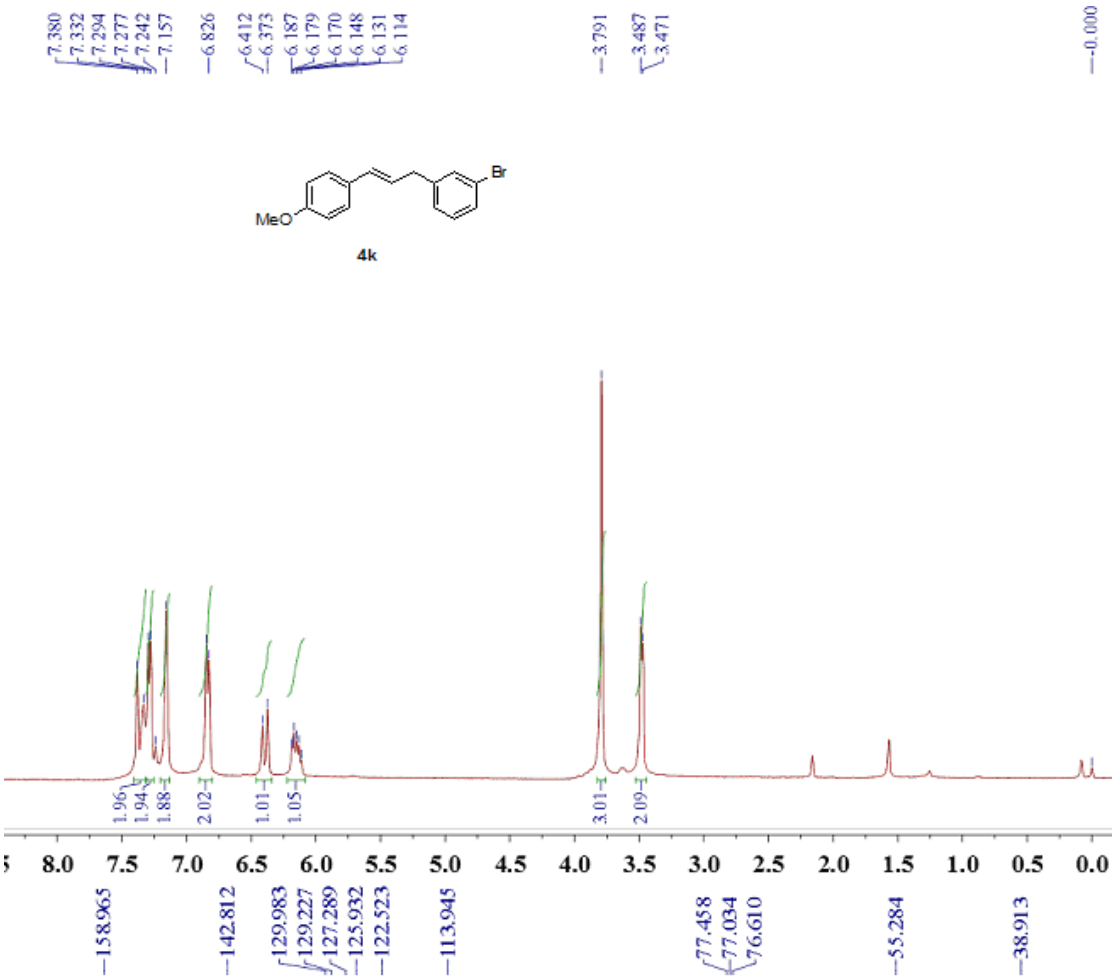
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6.170
6.148
6.131
6.114
3.791
3.487
3.471
0.000
Br
MeO
4k
1.96
1.94
1.88
2.02
1.01
1.05
3.01
2.09
8.0
7.5
7.0
6.5
6.0
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1.0
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0.0

158.965
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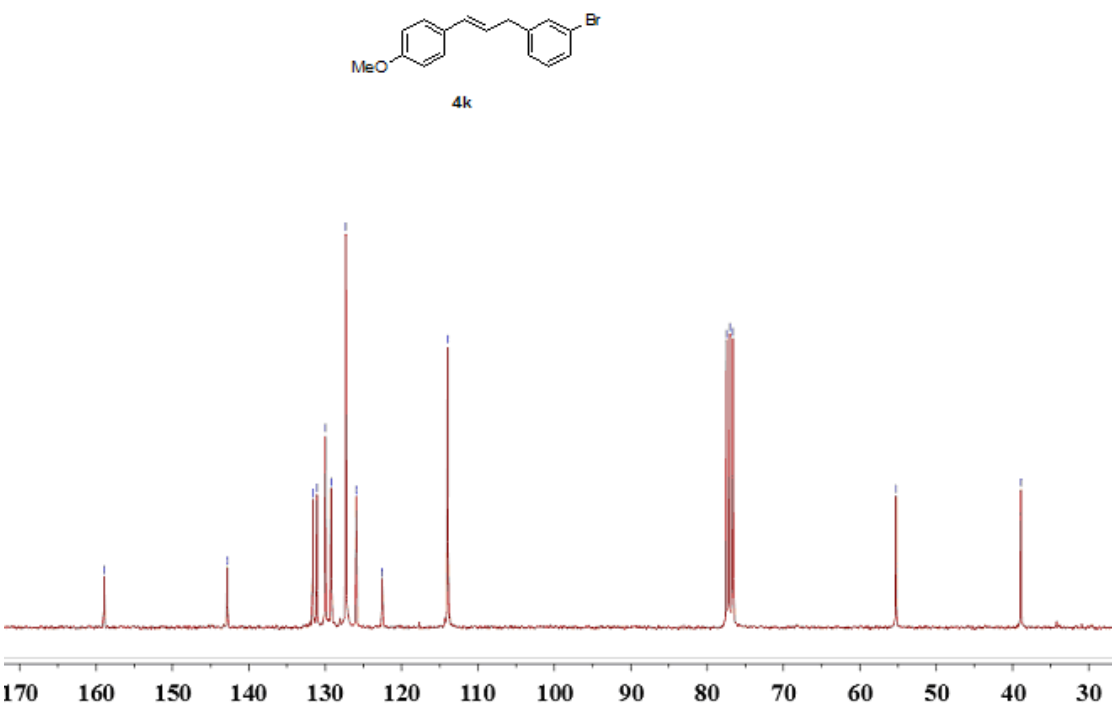
113.945
77.458
77.034
76.610
55.284
38.913


Br
MeO
4k
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160
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130
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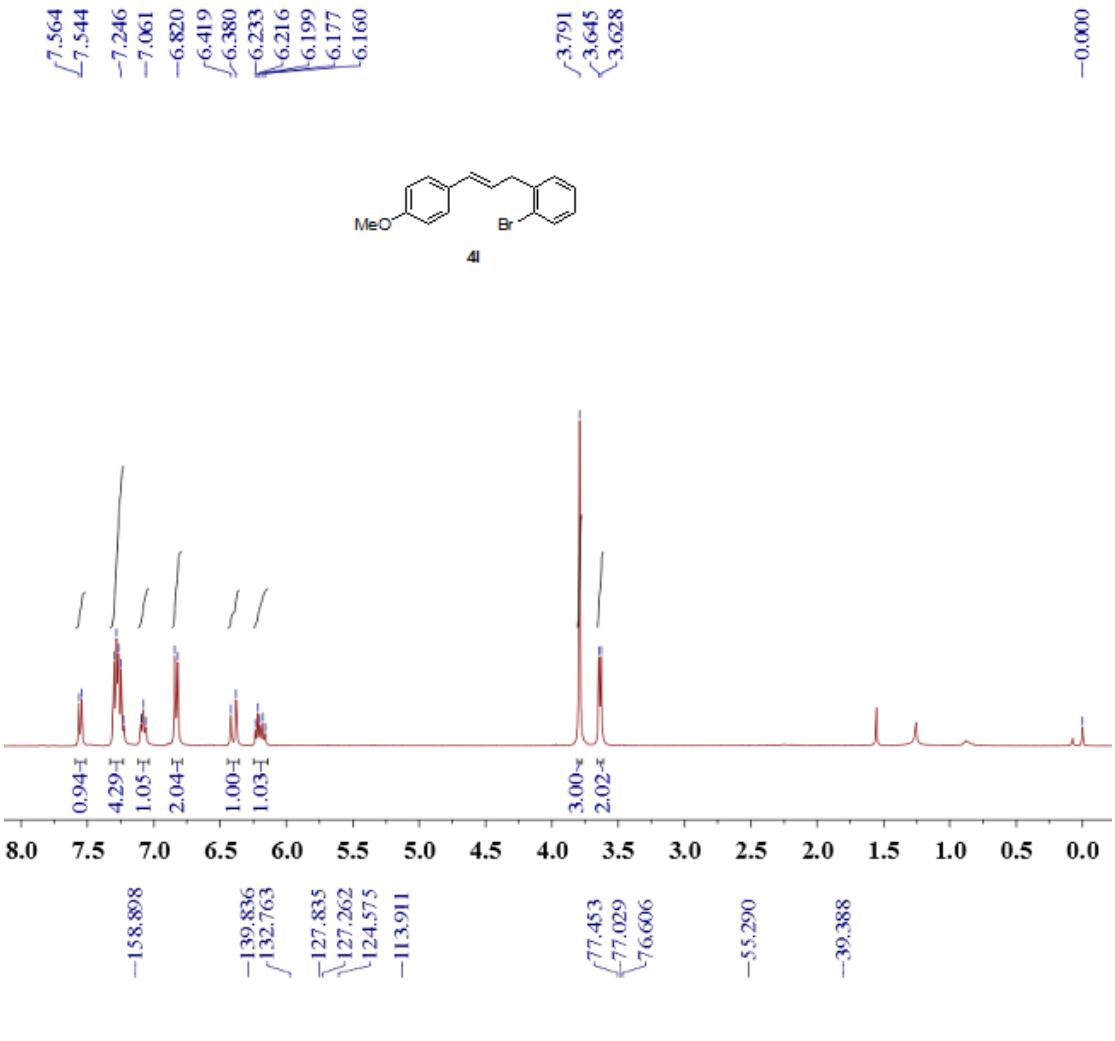
40
30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3.645
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0.000
MeO
Br
4l
0.94
4.29
1.05
2.04
1.00
1.03
3.00
2.02
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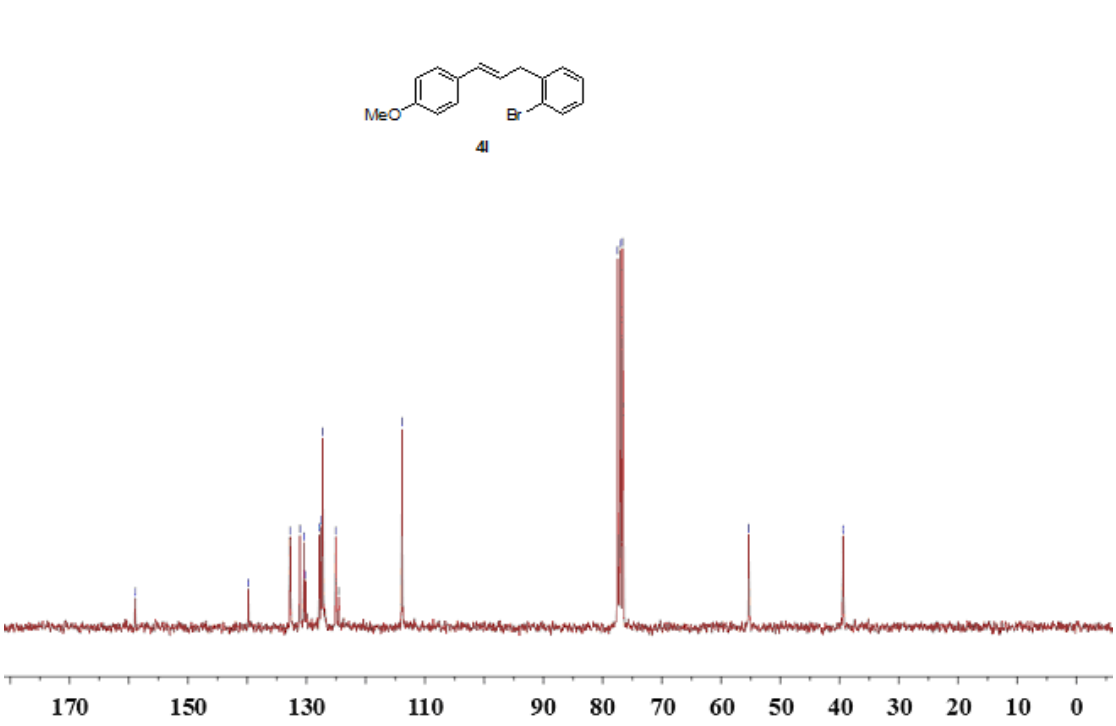
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132.763
127.835
127.262
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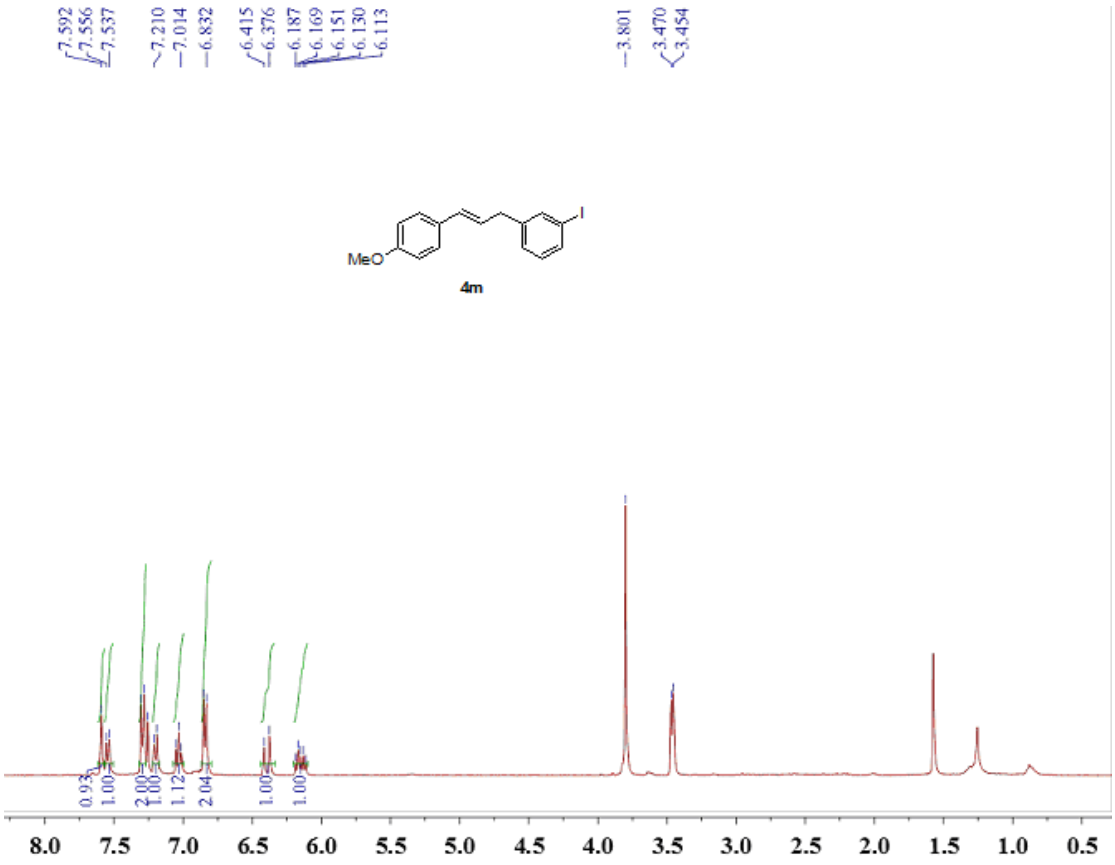
77.029
76.606
55.290
39.388
MeO
Br
4l
170 150 130 110 90 80 70 60 50 40 30 20 10 0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7.556
7.537
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6.832
6.415
6.376
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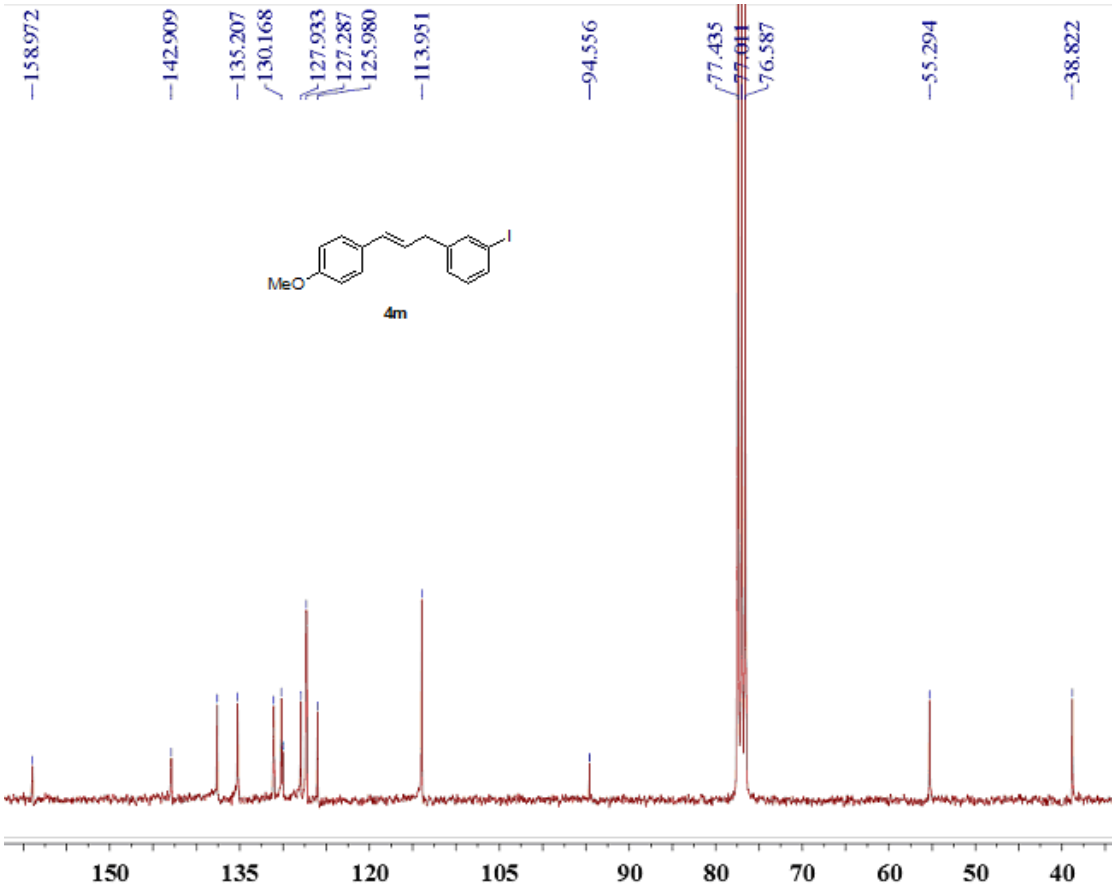
6.169
6.151
6.130
6.113
3.801
3.470
3.454
MeO
I
4m
0.93
1.00
2.00
1.08
1.12
2.04
1.00
1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5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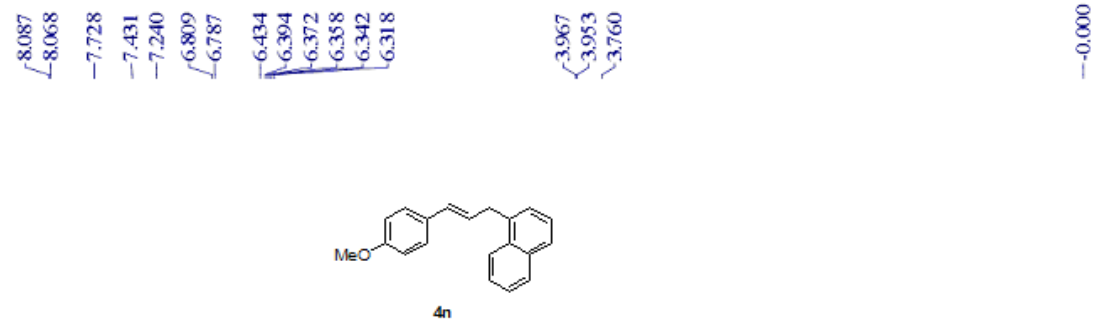
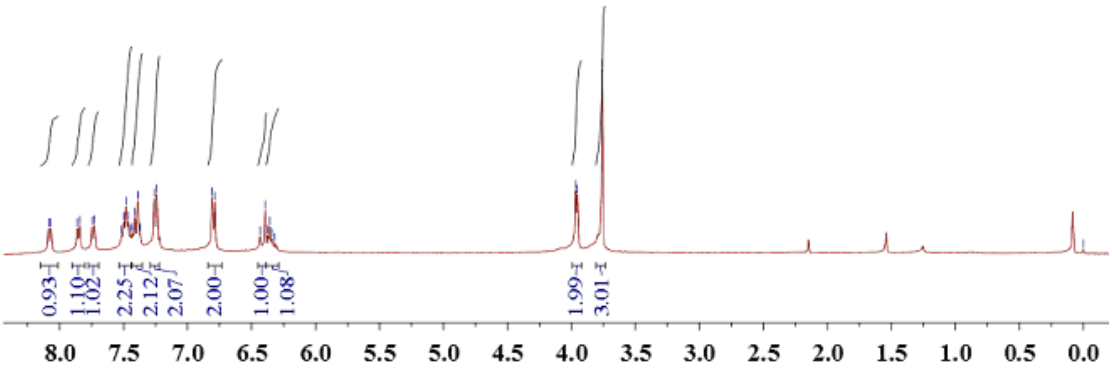
142.909
135.207
130.168
127.933
127.287
125.980
113.951
94.556
77.435
77.011
76.587
55.294
38.822
MeO
I
4m
150
135
120
105
90
80
70
60
50
40


8.087
8.068
7.728
7.431
7.240
6.809
6.787
6.434
6.394
6.372
6.358
6.342
6.318
3.967
3.953
3.760
0.000
MeO

4n


0.93
1.10
1.02
2.25
2.12
2.07
2.00
1.00
1.08
1.99
3.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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4.0
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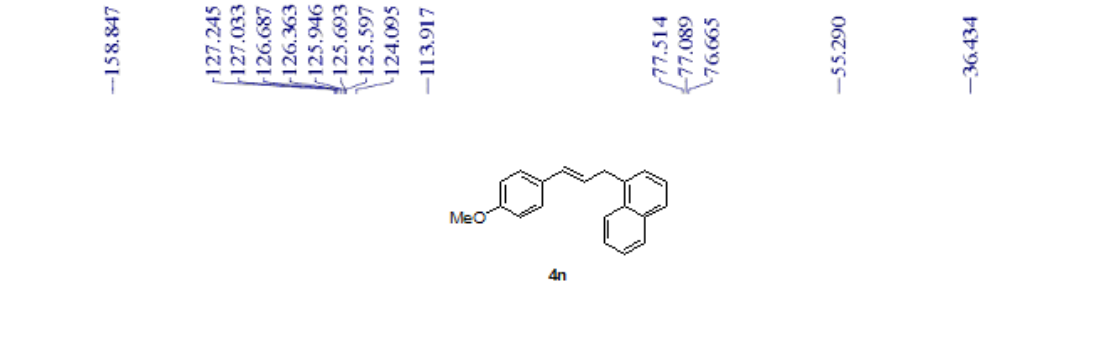
3.0
2.5
2.0
1.5
1.0
0.5
0.0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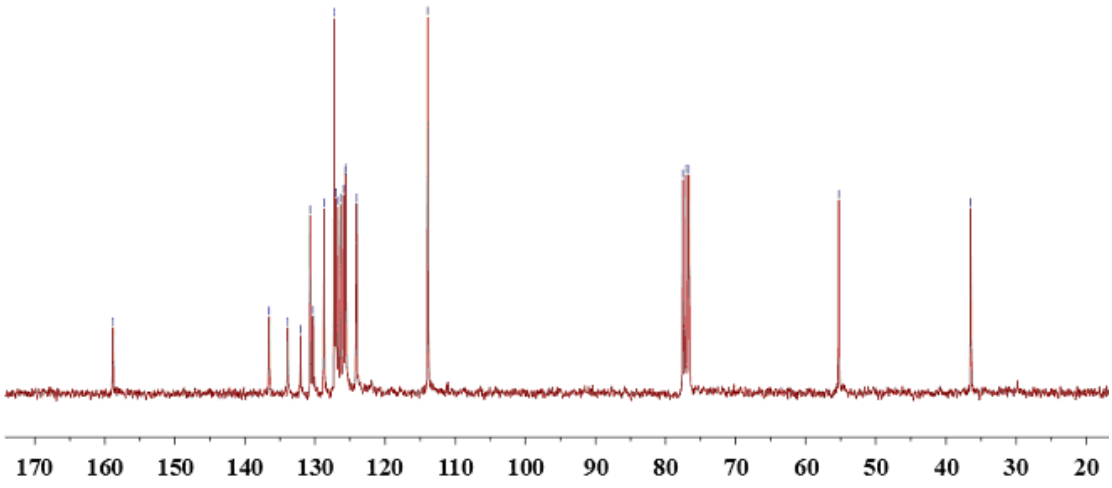
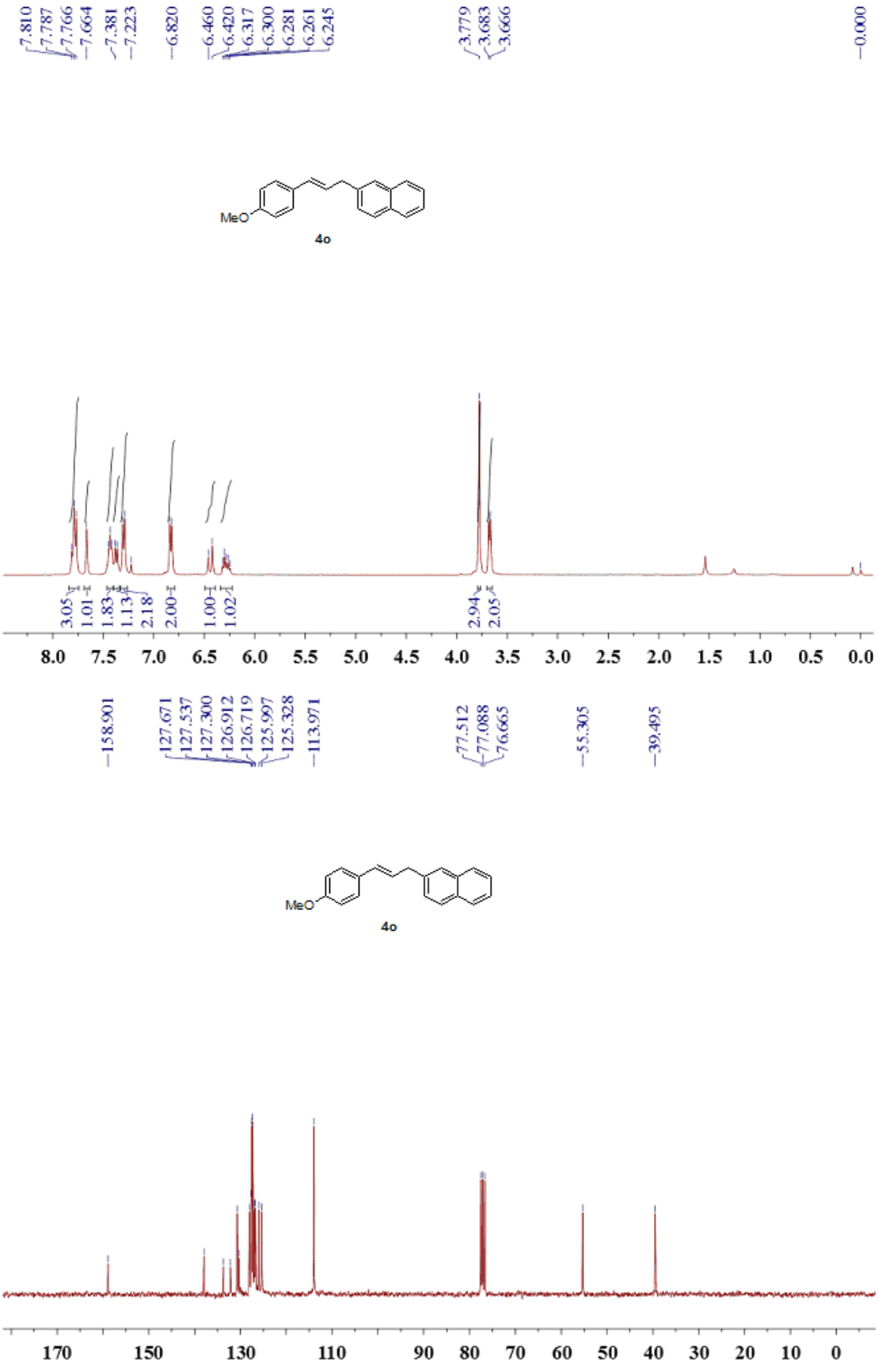
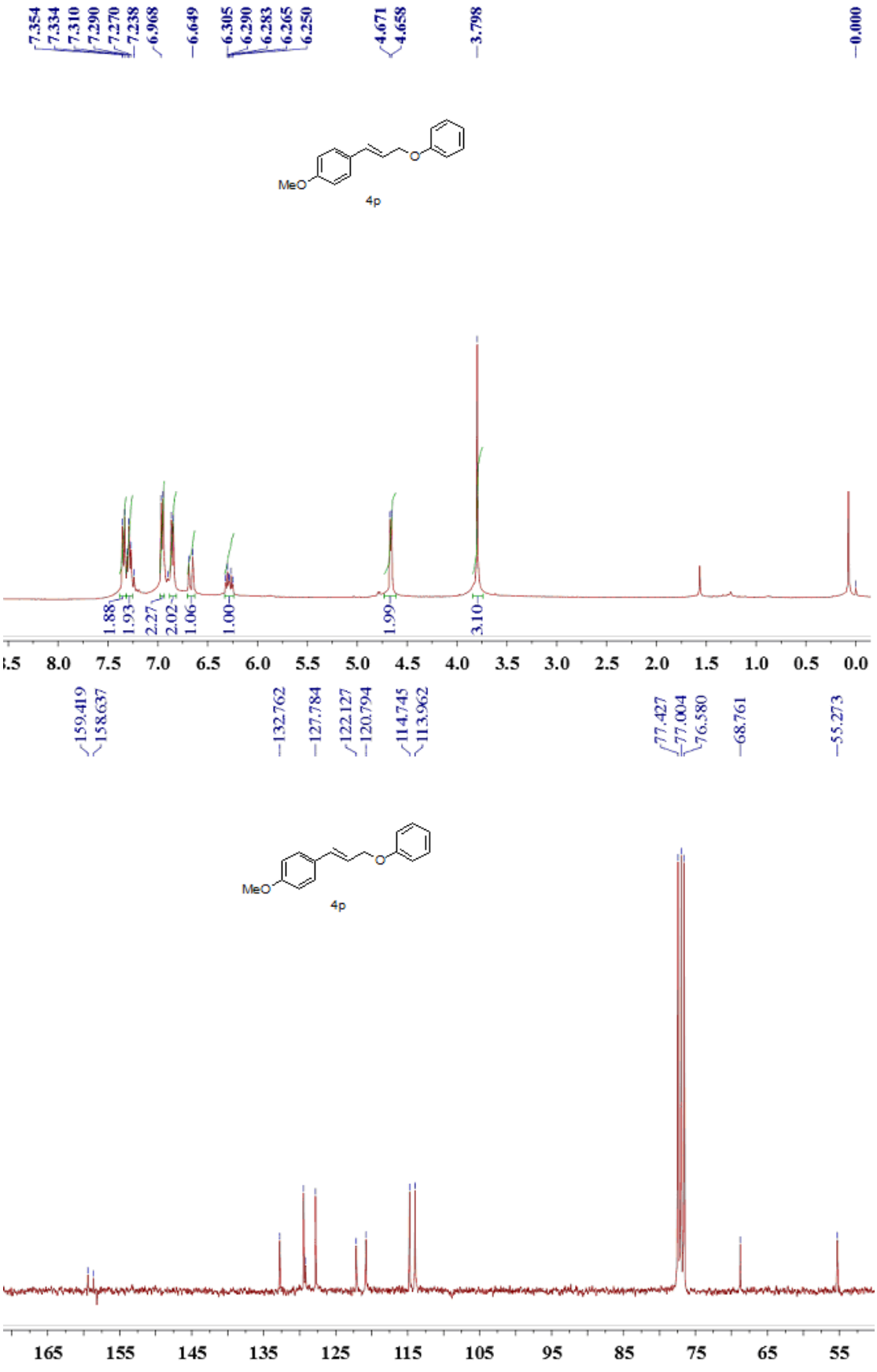
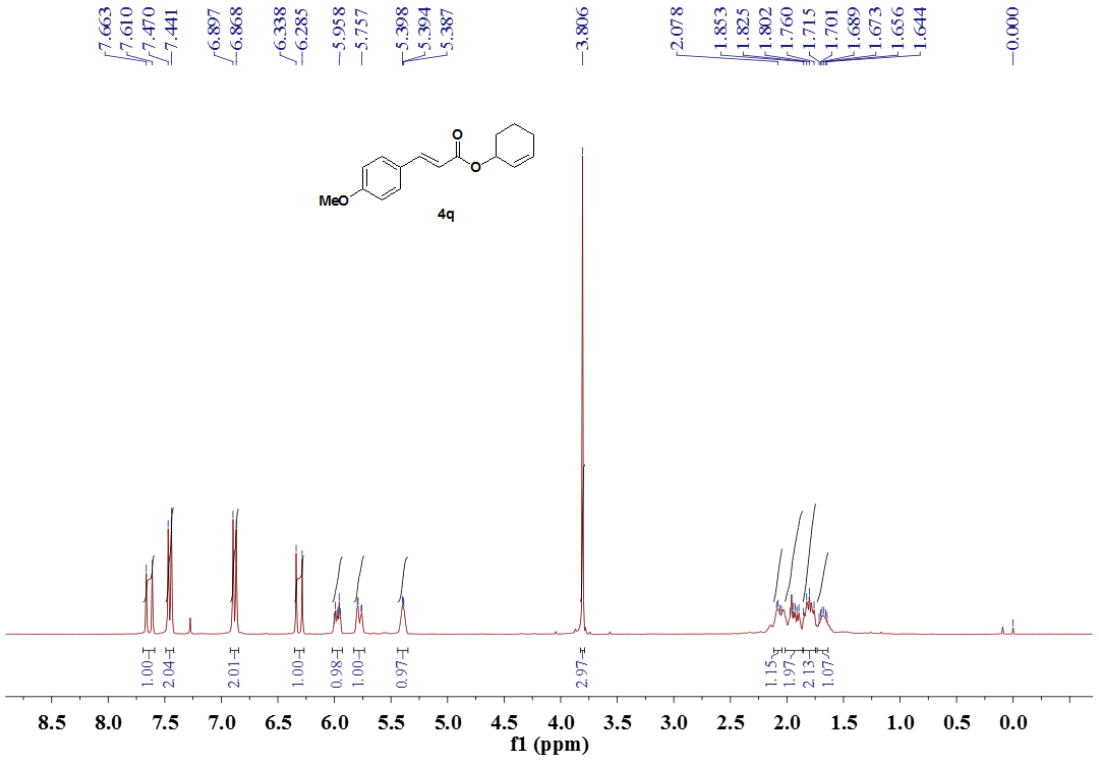
127.245
127.033
126.687
126.363
125.946
125.693
125.597
124.095
113.917
77.514
77.089
76.665
55.290
36.434
MeO

4n


170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20


7.810
7.787
7.766
7.664
7.381
7.223
6.820
6.460
6.420
6.317
6.300
6.281
6.261
6.245
3.779
3.683
3.666
0.000
MeO
4o
3.05
1.01
1.83
1.13
2.18
2.00
1.00
1.02
2.94
2.05
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
158.901
127.671
127.537
127.300
126.912
126.719
125.997
125.328
113.971
77.512
77.088
76.665
55.305
39.495
MeO
4o
170 150 130 110 90 80 70 60 50 40 30 20 10 0


7.354
7.334
7.310
7.290
7.270
7.238
6.968
6.649
6.305
6.290
6.283
6.265
6.250
4.671
4.658
3.798
0.000
MeO
4p
1.88
1.93
2.27
2.02
1.06
1.00
1.99
3.10
159.419
158.637
132.762
127.784
122.127
120.794
114.745
113.962
77.427
77.004
76.580
68.761
55.273
MeO
4p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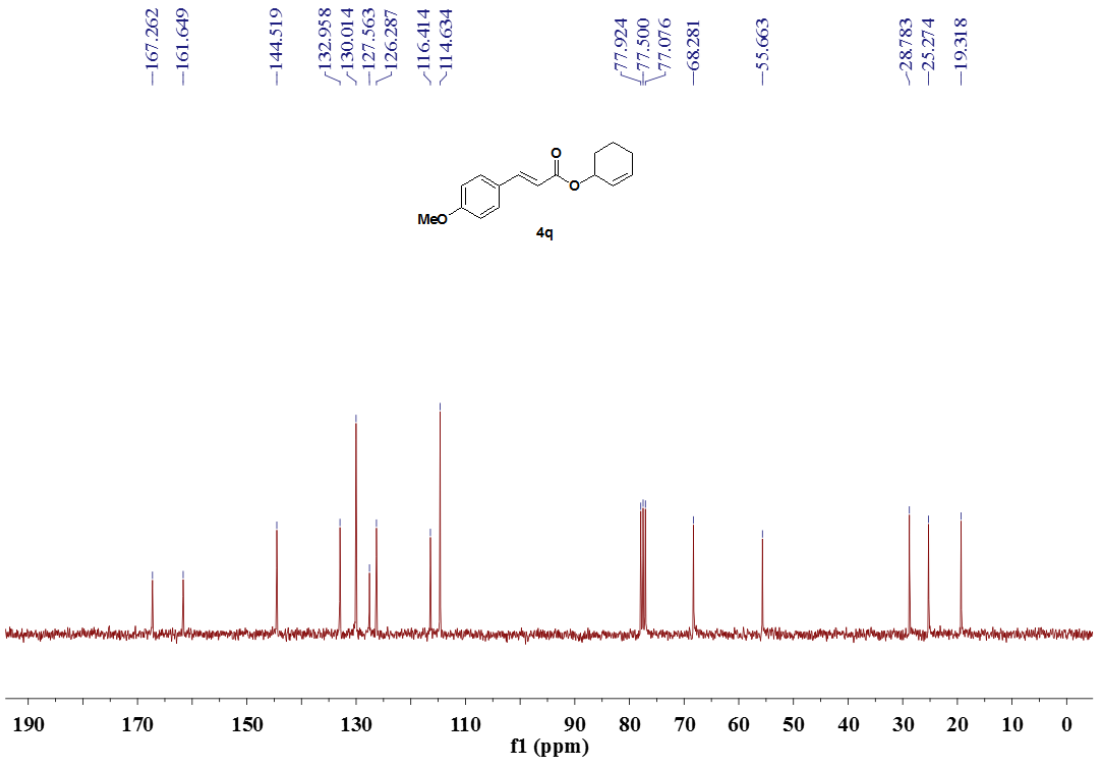
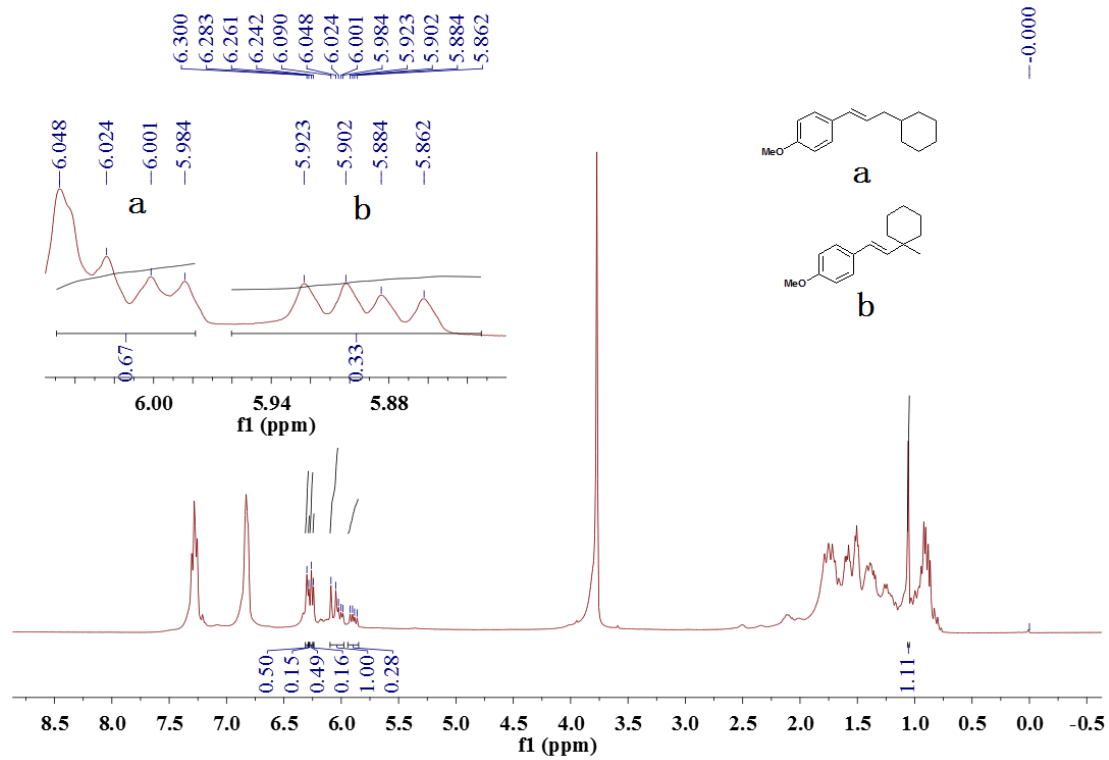
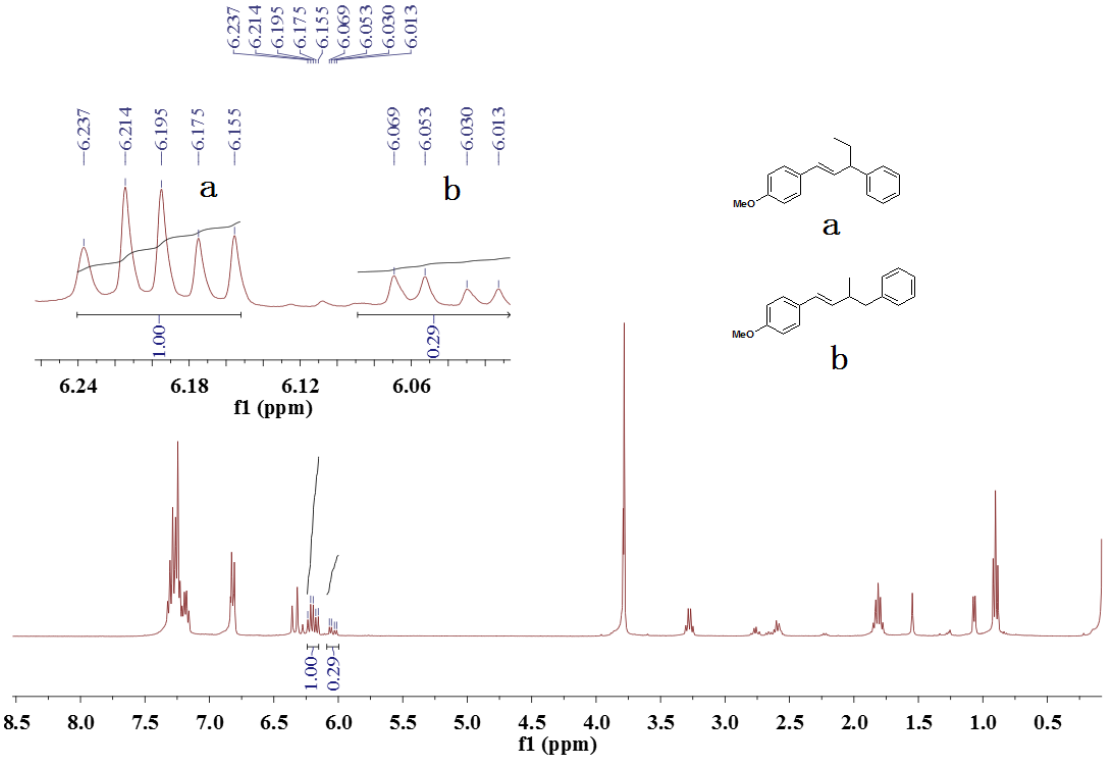
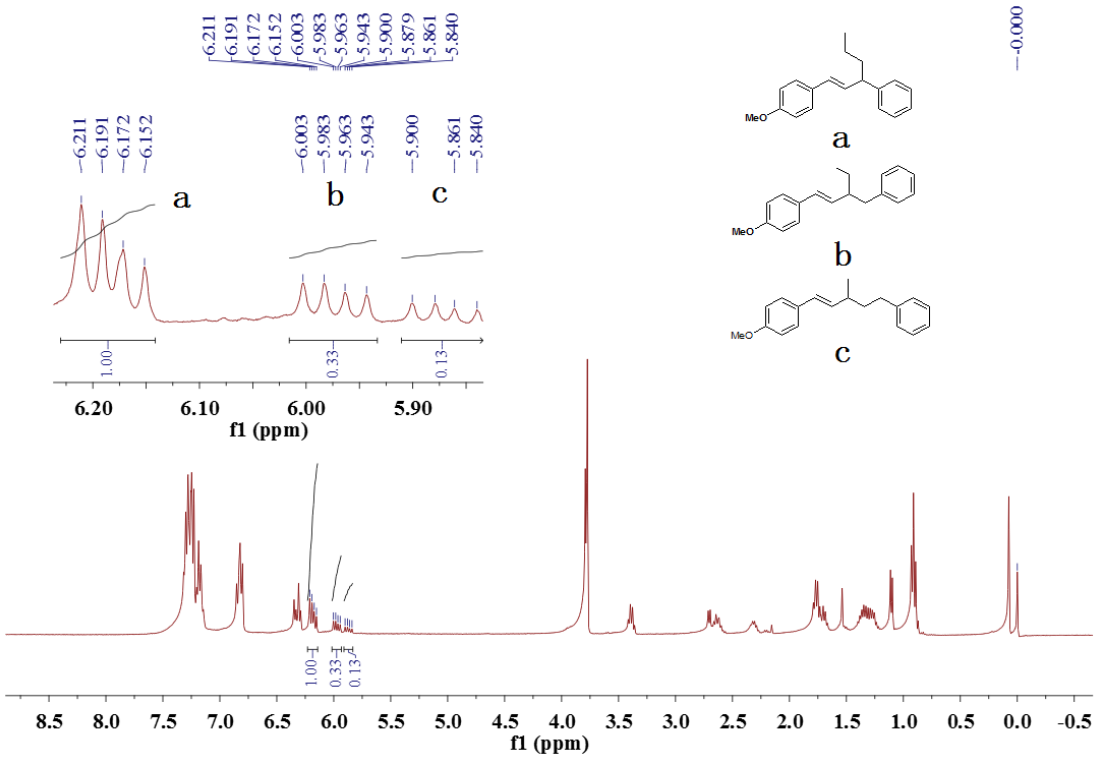
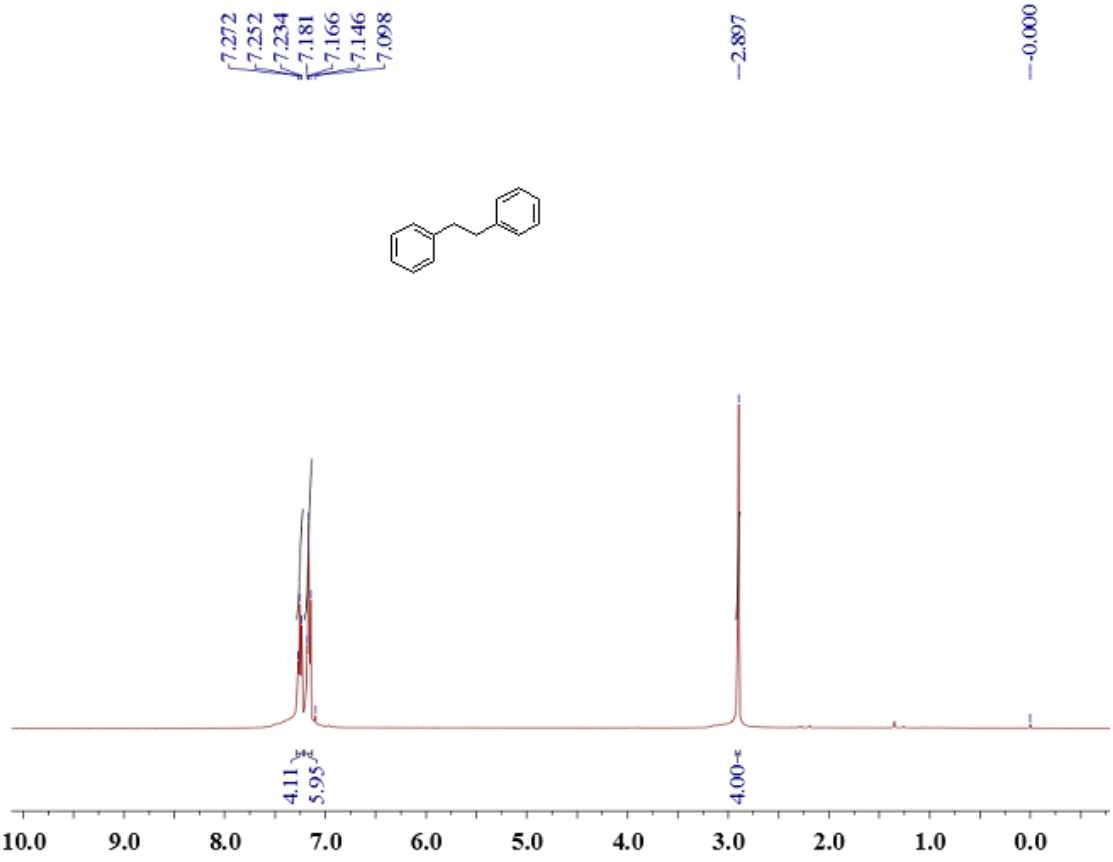
7.610
7.470
7.441
6.897
6.868
6.338
6.285
5.958
5.757
5.398
5.394
5.387
3.806
2.078
1.853
1.825
1.802
1.760
1.715
1.701
1.689
1.673
1.656
1.644
0.000
MeO
O
O
4q
1.00
2.04
2.01
1.00
0.98
1.00
0.97
2.97
1.15
1.97
2.13
1.07
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

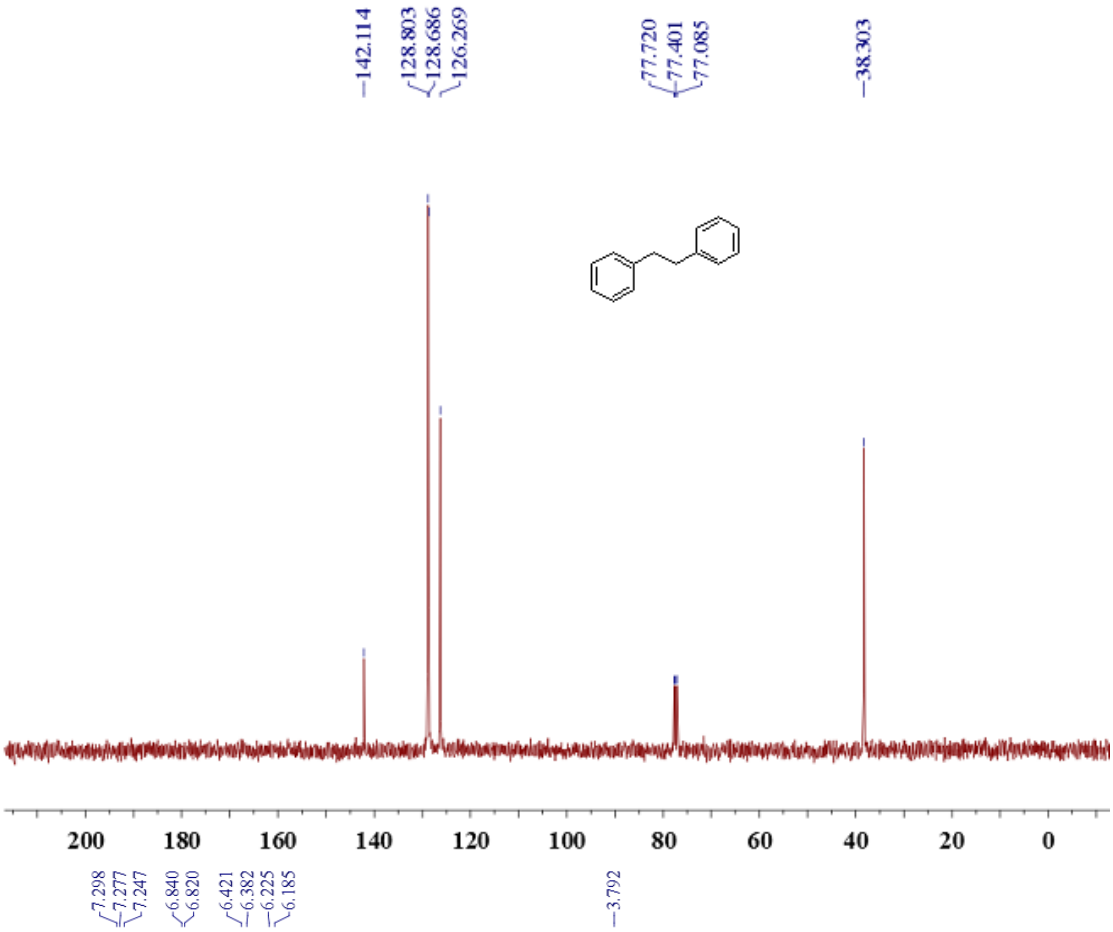

167.262
161.649
144.519
132.958
130.014
127.563
126.287
116.414
114.634
77.924
77.500
77.076
68.281
55.663
28.783
25.274
19.318
MeO
O
O
4q
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


6.300
6.283
6.261
6.242
6.090
6.048
6.024
6.001
5.984
5.923
5.902
5.884
5.862
-0.000
6.048
6.024
6.001
5.984
5.923
5.902
5.884
5.862
a
b
MeO
a
MeO
b
0.67
0.33
6.00
5.94
5.88
f1 (ppm)
0.50
0.15
0.49
0.16
1.00
0.28
1.11
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


6.237
6.214
6.195
6.175
6.155
6.069
6.053
6.030
6.013
6.237
6.214
6.195
6.175
6.155
6.069
6.053
6.030
6.013
a
b
MeO
a
MeO
b
1.00
0.29
6.24
6.18
6.12
6.06
f1 (ppm)
1.00
0.29
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


6.211
6.191
6.172
6.152
6.003
5.983
5.963
5.943
5.900
5.879
5.861
5.840
-0.000
a
b
c
MeO
1.00
0.33
0.13
6.20
6.10
6.00
5.90
f1 (ppm)
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


7.272
7.252
7.234
7.181
7.166
7.146
7.098
2.897
-0.000
4.11
5.95
4.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0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128.803
128.686
126.269
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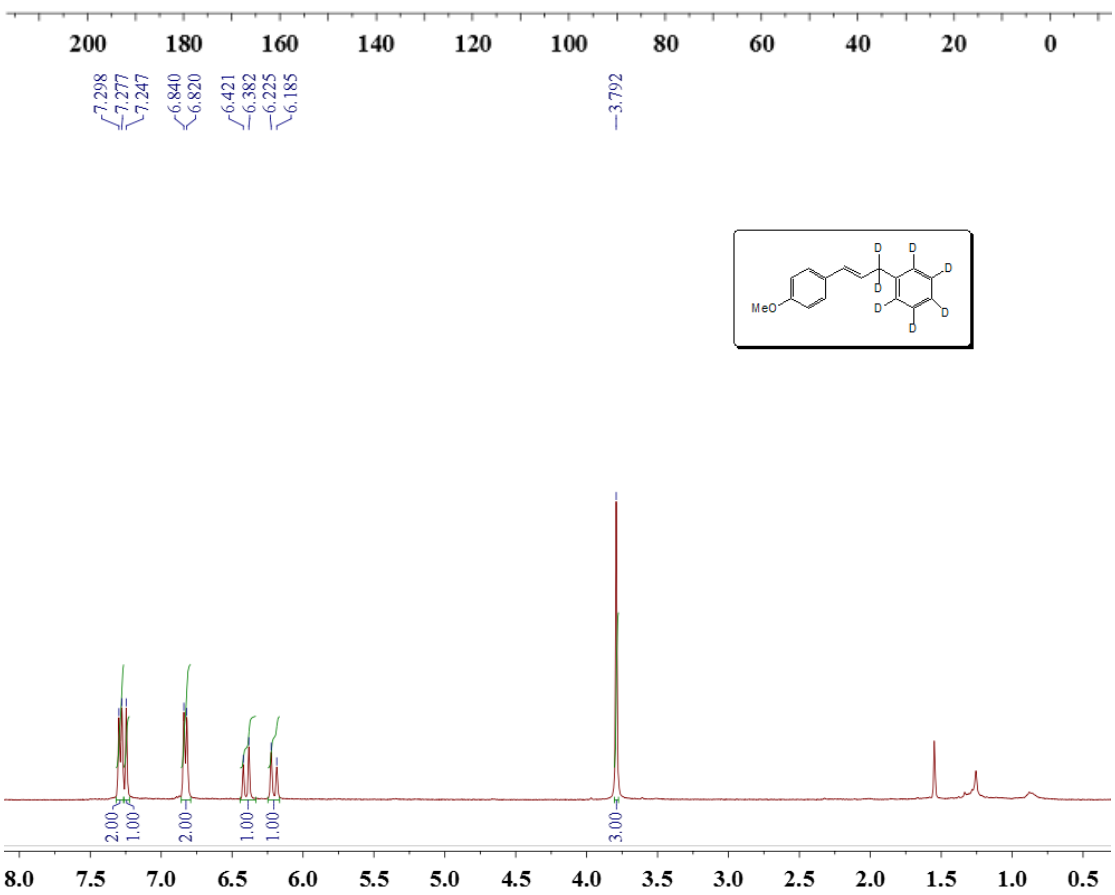
77.401
77.085
38.303
200
180
160
140
120
100
80
60
40
20
0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7.247
6.840
6.820
6.421
6.382
6.225
6.185
3.792
MeO
D
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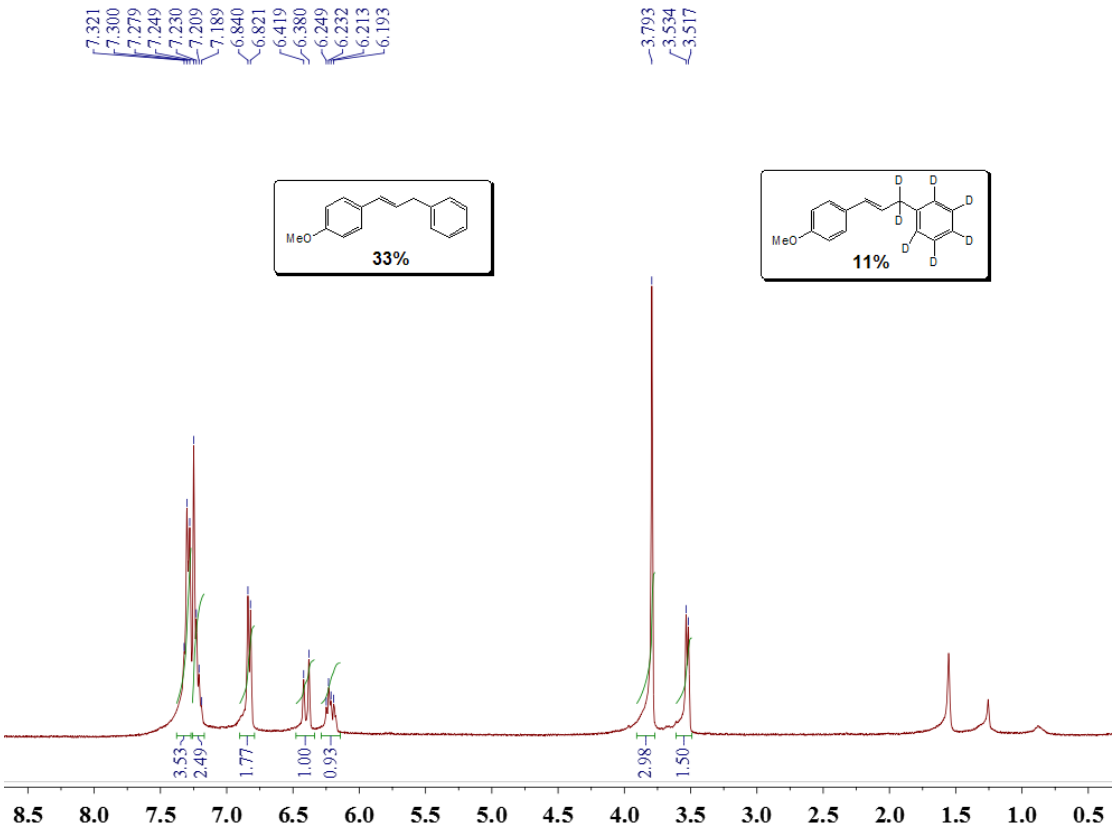
1.00
2.00
1.00
1.00
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5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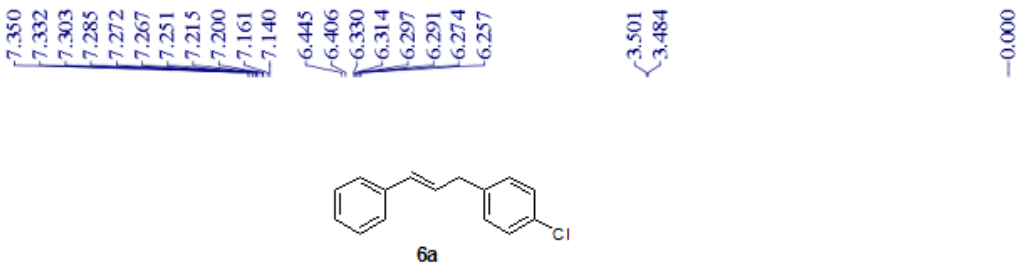
7.209
7.189
6.840
6.821
6.419
6.380
6.249
6.232
6.213
6.193
3.793
3.534
3.517
MeO
33%
MeO
11%
3.53
2.49
1.77
1.00
0.93
2.98
1.50
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5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7.332
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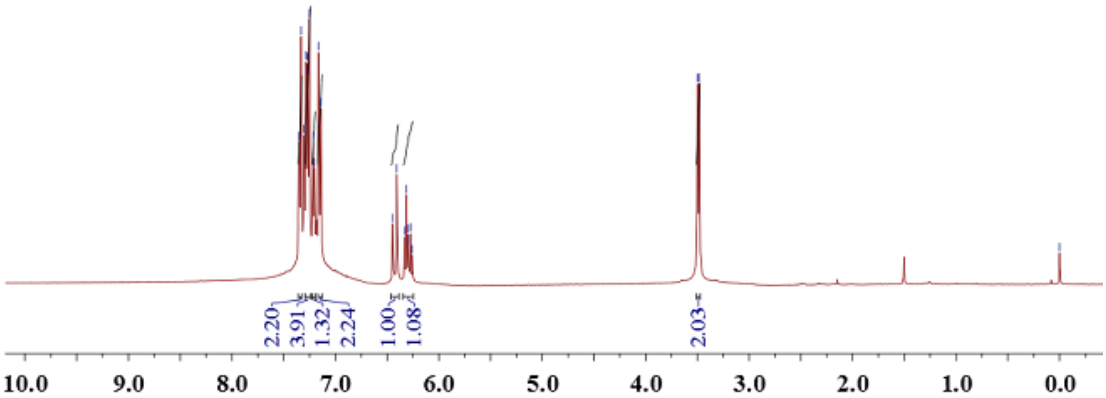
7.285
7.272
7.267
7.251
7.215
7.200
7.161
7.140
6.445
6.406
6.330
6.314
6.297
6.291
6.274
6.257
3.501
3.484
0.000

Cl

6a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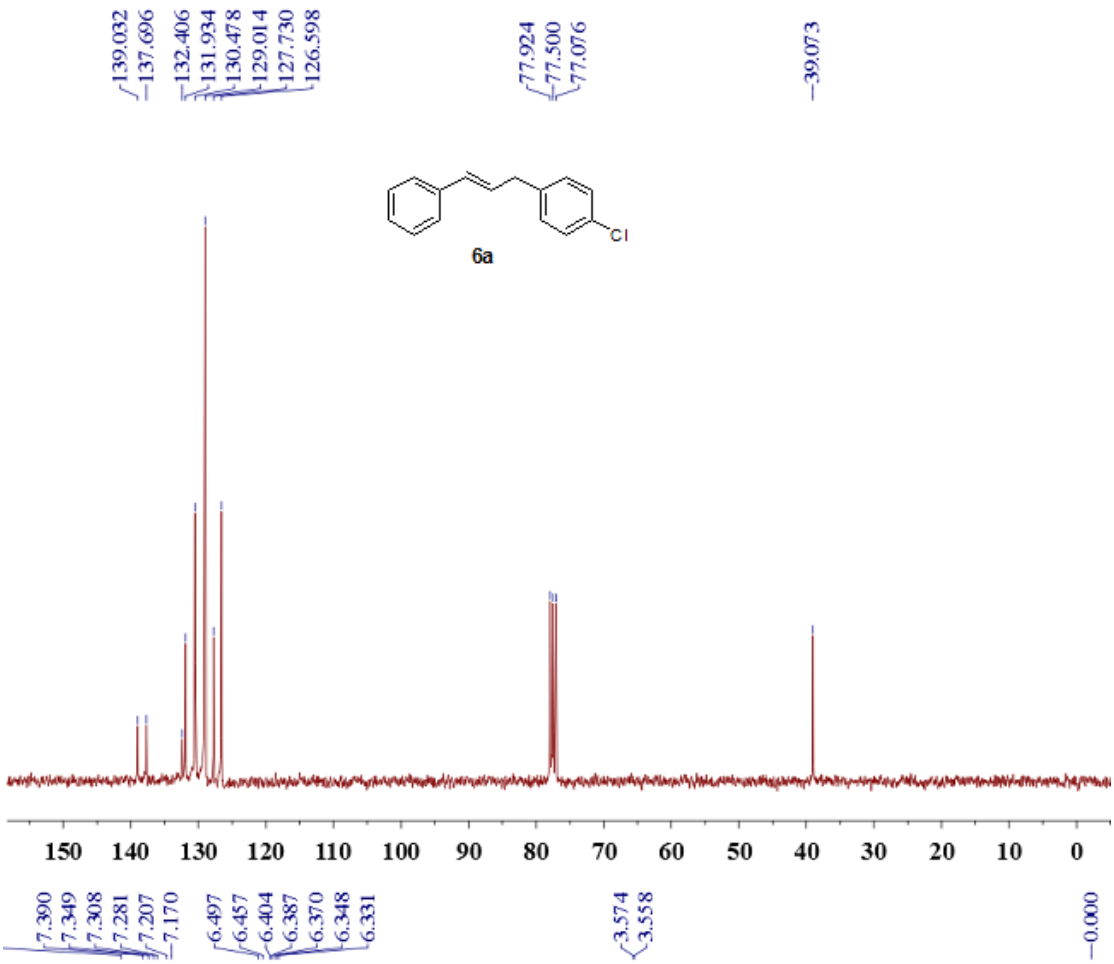
3.91
1.32
2.24
1.00
1.08
2.03
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0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137.696
132.406
131.934
130.478
129.014
127.730
126.598
77.924
77.500
77.076
39.073
Cl
6a
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
7.390
7.349
7.308
7.281
7.207
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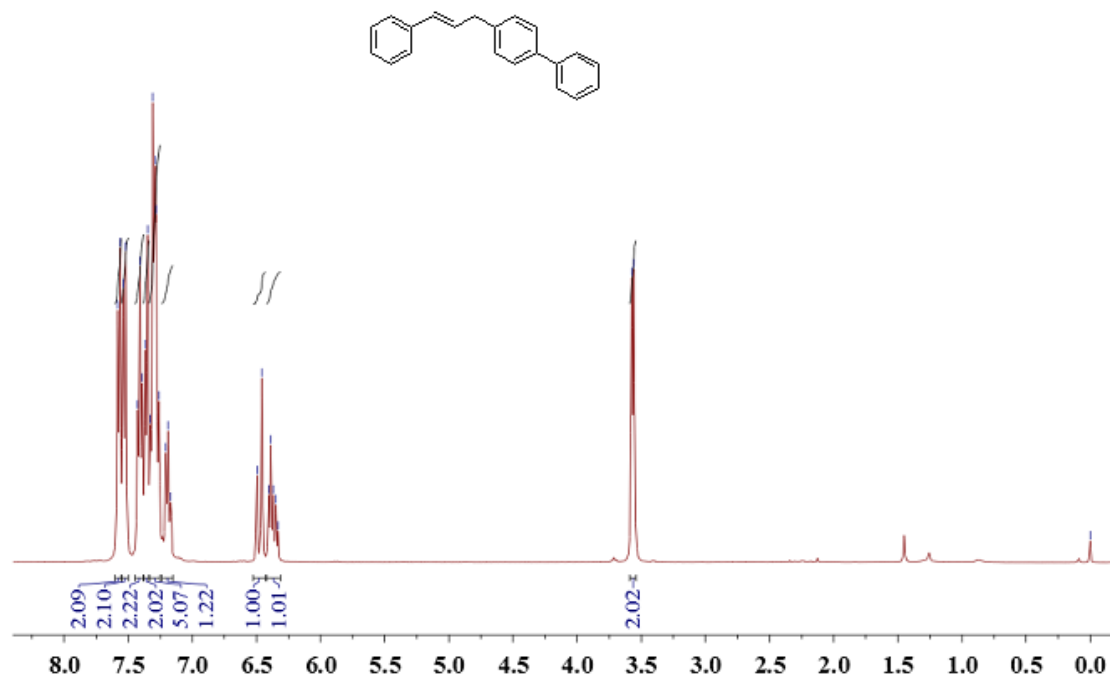
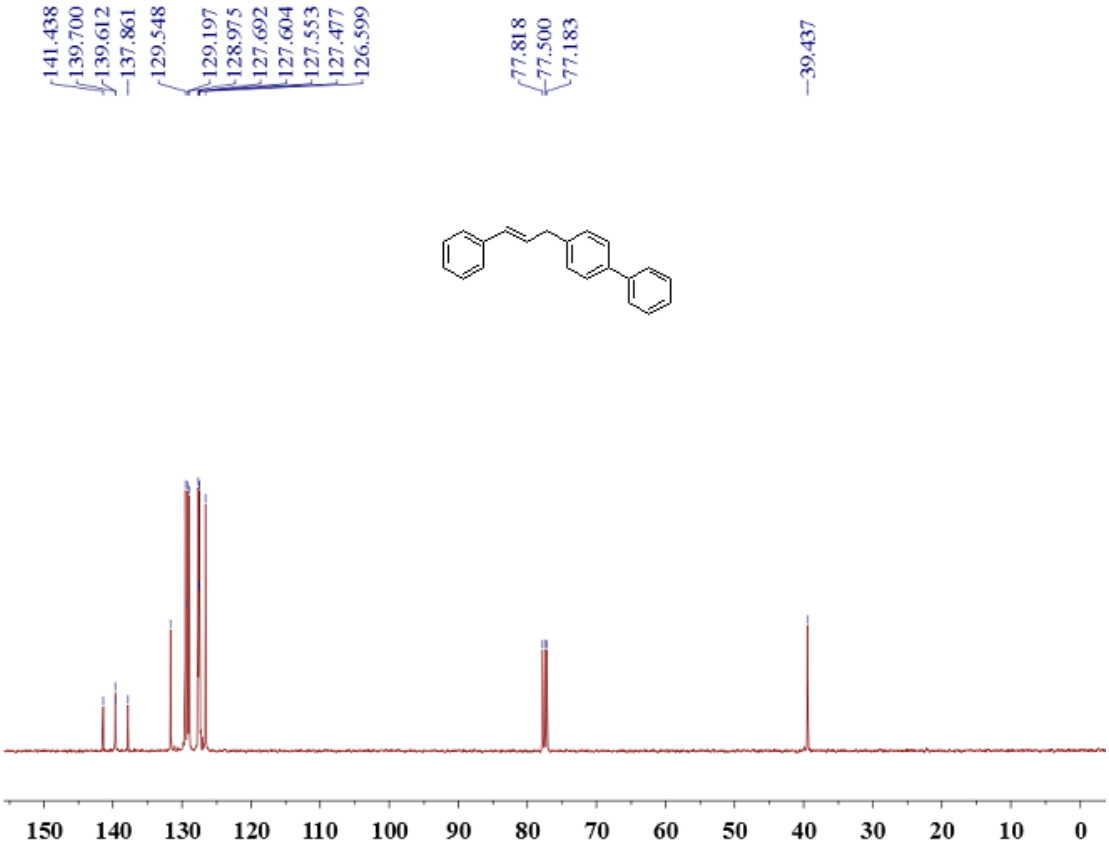
6.497
6.457
6.404
6.387
6.370
6.348
6.331
3.574
3.558
0.000


2.09
2.10
2.22
2.02
5.07
1.22
1.00
1.01
2.02
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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137.861
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129.197
128.975
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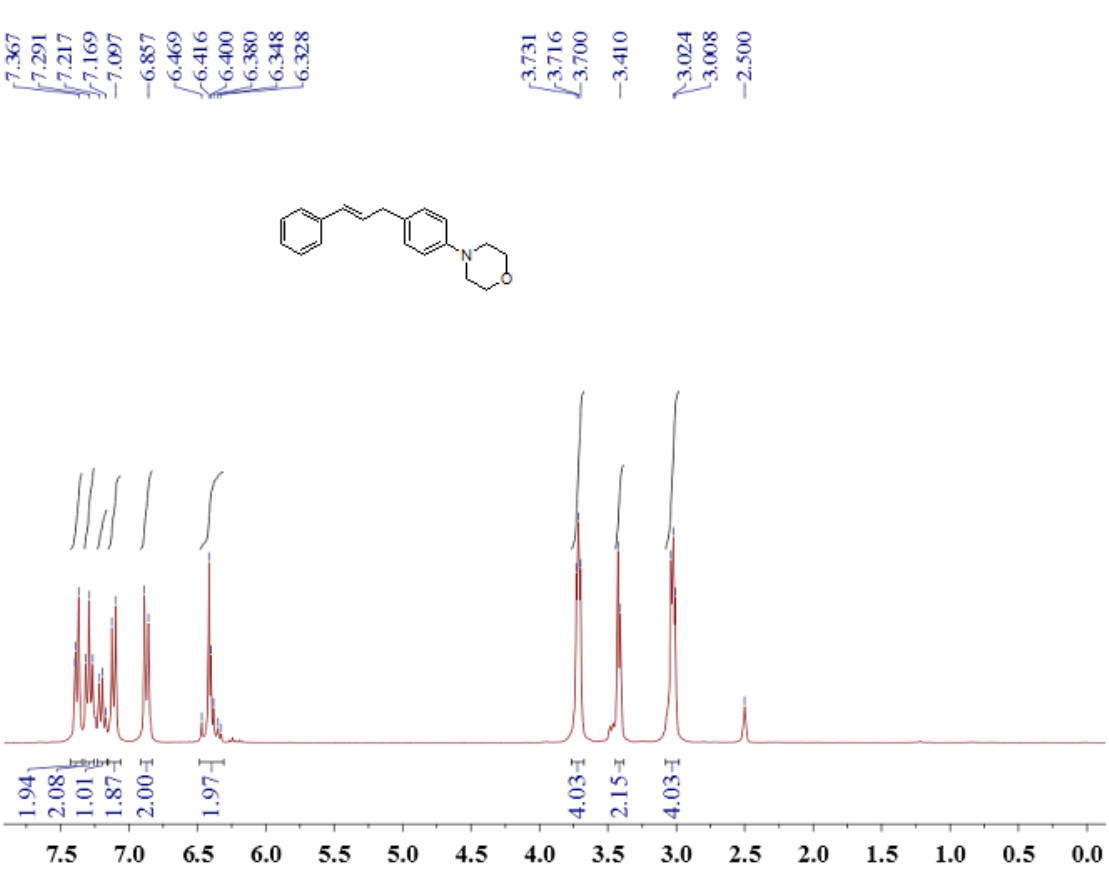
127.477
126.599
77.818
77.500
77.183
39.437
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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7.291
7.217
7.169
7.097
6.857
6.469
6.416
6.400
6.380
6.348
6.328
3.731
3.716
3.700
3.410
3.024
3.008
2.500
1.94
2.08
1.01
1.87
2.00
1.97
4.03
2.15
4.03
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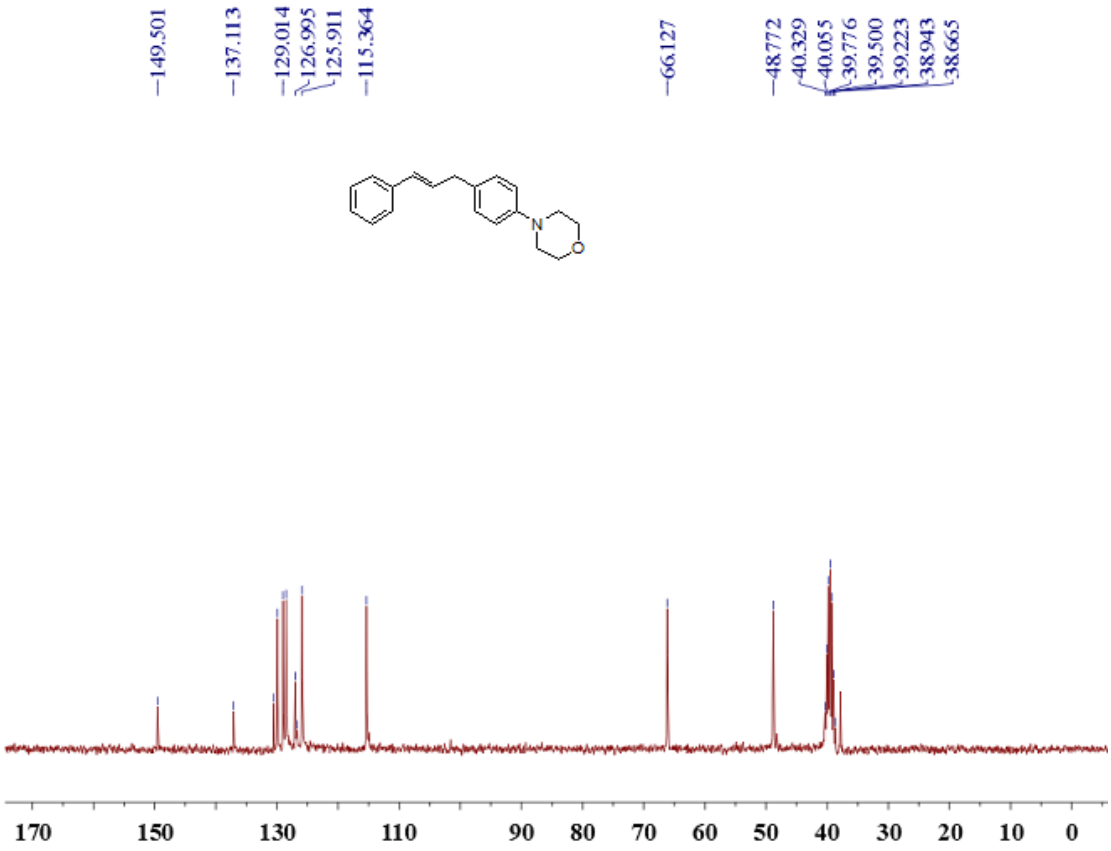
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115.364
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39.223
38.943
38.665
N
O
170
150
130
110
90
80
70
60
50
40
30
20
10
0