Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Tandem arylation and regioselective allylic etherification of 2,3-allenol via a Pd/B cooperative catalysis

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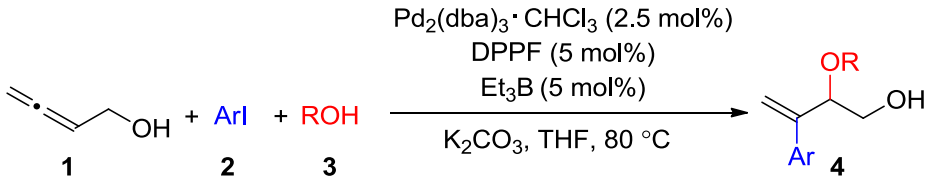
General experimental details

Analytical thin-layer chromatography (TLC) was carried out using 0.2-mm commercial silica gel plates (Yantai Jiangyou Silica Gel Development Co., Ltd., silica gel HSGF 254). Reactions were monitored by thin-layer chromatography (TLC) on silica plates (F-254) and visualized under UV light. Preparative column chromatography employing silica gel (Qingdao Shenghai Fine Silica Gel Chemical Co., Ltd., 200-300 mesh) was performed according to the method of Still. High-resolution mass spectra (HRMS) were performed at Instrumental Analysis Center of Shanghai Jiao Tong University using ESI method. Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded with a Bruker AVANCE III HD 500 (500 MHz) spectrometer. Chemical shifts are reported in delta (δ) units, parts per million (ppm) downfield from trimethylsilane or ppm relative to the center of the singlet at 7.26 ppm for deuteriochloroform. Coupling constants are reported in Hertz (Hz). Carbon-13 nuclear magnetic resonance (^{13}C NMR) spectra were recorded with a Bruker AVANCE III HD 500 (125 MHz) spectrometer. Chemical shifts are reported in delta (δ) units, ppm relative to the center of the triplet at 77.0 ppm for deuteriochloroform. ^{13}C NMR spectra were routinely run with broadband decoupling. Buta-2,3-dien-1-ol were synthesized according to reported procedures, all characterization data are in accordance with literature.[1] Unless otherwise noted, all reagents and solvents were obtained from commercial sources and used without further purification.

General procedure and product characterization

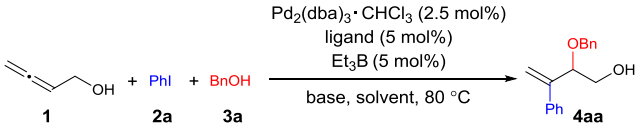
General procedures for the tandem arylation and allylic etherification of 2,3-allenol



Buta-2,3-dien-1-ol **(1)** (14.02 mg, 0.2 mmol, 1.0 equiv.), aryl iodides **2** (0.24 mmol, 1.2 equiv.), alcohols **3** (0.24 mmol, 1.2 equiv.) and BEt_3 (10 μL, 0.01 mmol, 5 mol%) were consecutively added to a sealed tube charged with a mixture of K_2CO_3 (55 mg, 0.4 mmol, 2.0 equiv.), $Pd_2(dba)_3 \cdot CHCl_3$ (5.2 mg, 0.005 mmol, 2.5 mol%), and DPPF (5.54 mg, 0.01 mmol, 5 mol%) under an atmosphere of nitrogen. THF (2 mL) were added sequentially. The reaction mixture was stirred at 80 ℃ for 12 h. After cooling to room temperature, the solvent was removed in vacuum. The residue was purified by flash column chromatography on silica gel to afford products **4**.

Condition optimizations

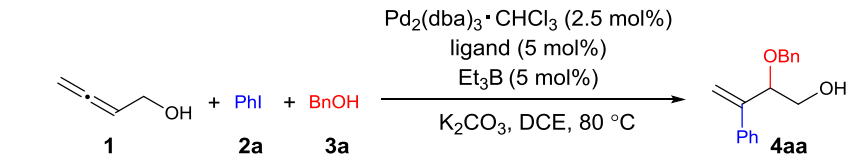
Table S1 Condition optimizations[a]



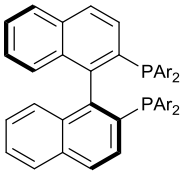
entry	ligand	solvent	base	yield(%)[b]
1	PPh_3	DCE	K_2CO_3	46
2	PCy_3	DCE	K_2CO_3	43
3	P(2-furanyl)$_3$	DCE	K_2CO_3	40
4	$P(C_6F_5)_3$	DCE	K_2CO_3	NR
5	SPhos	DCE	K_2CO_3	41
6	XPhos	DCE	K_2CO_3	39
7	DPPM	DCE	K_2CO_3	29
8	DPPE	DCE	K_2CO_3	25
9	DPPP	DCE	K_2CO_3	20
10	DPPB	DCE	K_2CO_3	30
11	DPPF	DCE	K_2CO_3	64
12	DPEphos	DCE	K_2CO_3	48
13	BINAP	DCE	K_2CO_3	42
14	DPPF	DCE	Cs_2CO_3	48
15	DPPF	DCE	K_3PO_4	35
16	DPPF	DCE	KF	trace
17	DPPF	DCE	KOH	trace
18	DPPF	DCE	Et_3N	trace
19	DPPF	benzene	K_2CO_3	70
20	DPPF	toluene	K_2CO_3	47
21	DPPF	THF	K_2CO_3	75
22	DPPF	MTBE	K_2CO_3	68
23	DPPF	dioxane	K_2CO_3	trace
24	DPPF	CH_3CN	K_2CO_3	35
25[c]	DPPF	THF	K_2CO_3	NR
26[d]	DPPF	THF	K_2CO_3	38

[a] Reaction conditions: $Pd_2(dba)_3$•$CHCl_3$ (2.5 mol%), ligand (10 mol% for mono-phosphines, 5 mol% for bidentate ligands), BEt_3 (5mol%), base (0.4 mmol), **1** (0.2 mmol), **2a** (0.24 mmol), **3a** (0.24 mmol), solvent (2.0 mL), at 80 °C for 12 h. [b] Yields are of isolated materials. [c] The reaction was carried out without BEt_3 under otherwise identical conditions. [d] the reaction was carried out with bromobenzene instead of iodobenzene. DCE = 1,2-Dichloroethane; THF = tetrahydrofuran; MTBE = methyl *tert*-butyl ether.

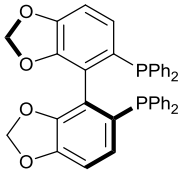
Table S2 Tandem arylation and allylic etherification with chiral ligands[a]



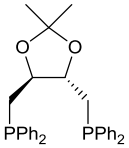
1 **2a** **3a** **4aa**



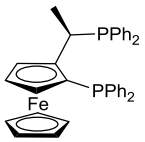
BINAP, 6% ee
Tol-BINAP, 3% ee
Xyl-BINAP, 7% ee



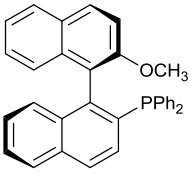
3% ee



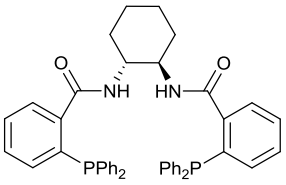
0% ee



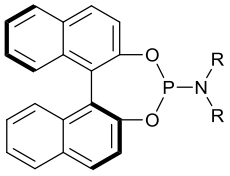
5% ee



14% ee



NR

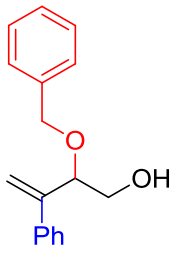


L1, R = *i*Pr, NR
L2, R = (*S*)-1-phenylethyl, NR
L3, R = (*R*)-1-phenylethyl, NR

[a] Reaction conditions: $Pd_2(dba)_3$•$CHCl_3$ (2.5 mol%), ligand (10 mol% for mono-phosphines, 5 mol% for bidentate ligands), BEt_3 (5mol%), K_2CO_3 (0.4 mmol), **1** (0.2 mmol), **2a** (0.24 mmol), **3a** (0.24 mmol), DCE (2.0 mL), at 80 °C for 12 h. The enantiomeric excesses were determined by HPLC using a chiral stationary phase.

Characterization of products 4

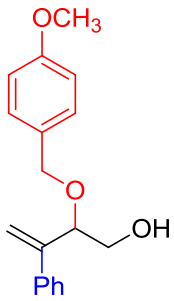
2-(benzyloxy)-3-phenylbut-3-en-1-ol (4aa)



4aa

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52–7.29 (m, 10H), 5.55 (d, *J* = 1.2 Hz, 1H), 5.47 (s, 1H), 4.78 (d, *J* = 11.4 Hz, 1H), 4.48 (dd, *J* = 11.4, 6.2 Hz, 2H), 3.64–3.56 (m, 2H), 2.17 (d, *J* = 3.7 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.86, 139.26, 137.93, 128.52, 128.26, 127.96, 127.89, 127.86, 126.57, 115.69, 82.24, 70.78, 65.54; HRMS (ESI-MS): Calcd. for $C_{17}H_{18}O_2$ (M + Na): 277.1204, Found: 277.1203.

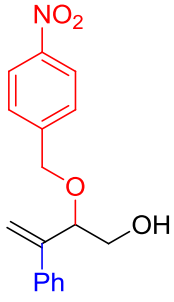
2-((4-methoxybenzyl)oxy)-3-phenylbut-3-en-1-ol (4ab)



4ab

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43–7.26 (m, 7H), 6.91 (d, *J* = 8.6 Hz, 2H), 5.54 (d, *J* = 1.2 Hz, 1H), 5.46 (s, 1H), 4.70 (d, *J* = 11.1 Hz, 1H), 4.45 (dd, *J* = 7.5, 4.0 Hz, 1H), 4.41 (d, *J* = 11.1 Hz, 1H), 3.81 (s, 3H), 3.64–3.47 (m, 2H), 2.18 (dd, *J* = 8.9, 4.2 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.35, 145.00, 139.33, 130.04, 129.64, 128.46, 127.88, 126.59, 115.64, 113.94, 81.92, 70.49, 65.56, 55.29; HRMS (ESI-MS): Calcd. for $C_{18}H_{20}O_3$ (M + Na): 307.1310, Found: 307.1317.

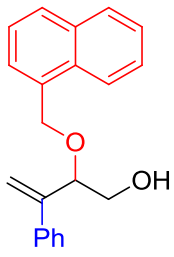
2-((4-nitrobenzyl)oxy)-3-phenylbut-3-en-1-ol (4ac)



4ac

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.23 (d, *J* = 8.8 Hz, 2H), 7.54 (d, *J* = 8.8 Hz, 2H), 7.44–7.30 (m, 4H), 5.57 (d, *J* = 1.2 Hz, 1H), 5.45 (s, 1H), 4.86 (d, *J* = 12.9 Hz, 1H), 4.63 (d, *J* = 12.9 Hz, 1H), 4.52–4.47 (m, 1H), 3.67 (t, *J* = 6.4 Hz, 2H), 2.07 (dd, *J* =12.9, 6.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 147.43, 145.56, 144.61, 138.92, 128.60, 128.13, 127.88, 126.53, 123.72, 116.01, 83.08, 69.55, 65.52; HRMS (ESI-MS): Calcd. for $C_{17}H_{17}NO_4$ (M + Na): 322.1055, Found: 322.1057.

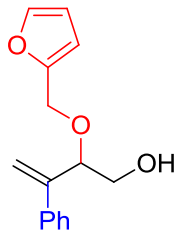
2-(naphthalen-1-ylmethoxy)-3-phenylbut-3-en-1-ol (4ad)



4ad

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93–7.73 (m, 4H), 7.58–7.27 (m, 8H), 5.58 (d, *J* = 1.2 Hz, 1H), 5.51 (s, 1H), 4.93 (d, *J* = 11.7 Hz, 1H), 4.65 (d, *J* = 11.7 Hz, 1H), 4.54–4.51 (m, 1H), 3.72–3.46 (m, 2H), 2.20 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.92, 139.28, 135.39, 133.25, 133.01, 128.49, 128.34, 127.92, 127.86, 127.70, 126.75, 126.60, 126.18, 125.99, 125.85, 115.79, 82.22, 70.90, 65.57; HRMS (ESI-MS): Calcd. for $C_{21}H_{20}O_2$ (M + Na): 327.1361, Found: 327.1366.

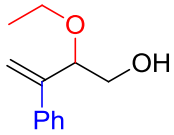
2-(furan-2-ylmethoxy)-3-phenylbut-3-en-1-ol (4ae)



4ae

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52–7.26 (m, 6H), 6.37–6.34 (m, 2H), 5.53 (s, 1H), 5.46 (s, 1H), 4.69 (d, *J* = 12.7 Hz, 1H), 4.56–4.36 (m, 2H), 3.58–3.50 (m, 2H), 2.24 (d, *J* = 8.7 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.51, 144.61, 143.02, 139.25, 128.47, 127.91, 126.58, 115.65, 110.33, 109.60, 81.83, 65.45, 62.68; HRMS (ESI-MS): Calcd. for $C_{15}H_{16}O_3$ (M + Na): 267.0997, Found: 267.0992.

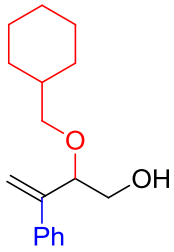
2-ethoxy-3-phenylbut-3-en-1-ol (4af)



4af

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40–7.27 (m, 5H), 5.46 (d, *J* = 1.4 Hz, 1H), 5.37 (d, *J* = 1.1 Hz, 1H), 4.34 (ddd, *J* = 7.8, 3.8, 0.8 Hz, 1H), 3.78–3.72 (m, 1H), 3.61–3.37 (m, 3H), 2.30 (d, *J* = 6.6 Hz, 1H), 1.27 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.37, 139.46, 128.40, 127.78, 126.55, 115.10, 82.75, 65.49, 64.50, 15.33; HRMS (ESI-MS): Calcd. for $C_{12}H_{16}O_2$ (M + Na): 215.1048, Found: 215.1044.

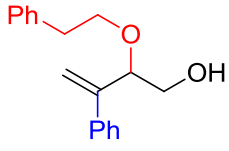
2-(cyclohexylmethoxy)-3-phenylbut-3-en-1-ol (4ag)



4ag

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49–7.27 (m, 5H), 5.46 (d, *J* = 1.4 Hz, 1H), 5.36 (s, 1H), 4.30 (dd, *J* = 8.0, 3.2 Hz, 1H), 3.60–3.48 (m, 3H), 3.23 (dd, *J* = 9.0, 6.1 Hz, 1H), 2.24 (dd, *J* = 9.7, 3.2 Hz, 1H), 1.89–1.58 (m, 5H), 1.35–1.10 (m, 4H), 0.99 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.32, 139.47, 128.39, 127.76, 126.55, 115.15, 82.98, 75.01, 65.63, 38.25, 30.19, 30.12, 26.56, 25.87, 25.83; HRMS (ESI-MS): Calcd. for $C_{17}H_{24}O_2$ (M + Na): 283.1674, Found: 283.1667.

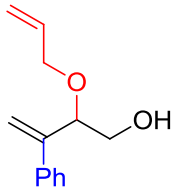
2-phenethoxy-3-phenylbut-3-en-1-ol (4ah)



4ah

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44–7.09 (m, 10H), 5.42 (d, *J* = 1.3 Hz, 1H), 5.28 (s, 1H), 4.31 (dd, *J* = 8.2, 3.4 Hz, 1H), 3.93 (ddd, *J* = 6.8, 6.8, 9.3 Hz, 1H), 3.64 (ddd, J = 7.0, 7.0, 9.3 Hz, 1H), 3.55–3.45 (m, 2H), 3.00–2.90 (m, 2H), 2.10 (d, *J* = 9.2 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.07, 139.29, 138.87, 128.90, 128.39, 127.78, 126.53, 126.31, 115.34, 83.20, 69.89, 65.50, 36.45; HRMS (ESI-MS): Calcd. for $C_{18}H_{20}O_2$ (M + Na): 291.1361, Found: 291.1355.

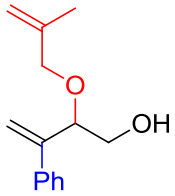
2-(allyloxy)-3-phenylbut-3-en-1-ol (4ai)



4ai

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40–7.28 (m, 5H), 6.02–5.94 (m, 1H), 5.48 (d, *J* = 1.2 Hz, 1H), 5.39 (s, 1H), 5.33 (dd, *J* = 17.2, 1.6 Hz, 1H), 5.23 (dd, *J* = 10.4, 1.2 Hz, 1H), 4.42 (dd, *J* = 7.9, 3.4 Hz, 1H), 4.23 (dd, *J* = 11.3, 3.8 Hz, 1H), 4.01 (dd, *J* = 12.6, 6.2 Hz, 1H), 3.61–3.52 (m, 2H), 2.18 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.99, 139.34, 134.58, 128.40, 127.80, 126.54, 117.29, 115.42, 82.07, 69.75, 65.42; HRMS (ESI-MS): Calcd. for $C_{13}H_{16}O_2$ (M + Na): 227.1048, Found: 227.1051.

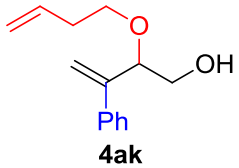
2-((2-methylallyl)oxy)-3-phenylbut-3-en-1-ol (4aj)



4aj

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42–7.27 (m, 5H), 5.49 (d, *J* = 1.4 Hz, 1H), 5.40 (dd, *J* = 1.2, 1.1 Hz, 1H), 5.04 (s, 1H), 4.95 (s, 1H), 4.40 (ddd, *J* = 7.5, 3.9, 0.7 Hz, 1H), 4.12 (d, *J* = 12.4 Hz, 1H), 3.92 (d, *J* = 12.3 Hz, 1H), 3.65–3.46 (m, 2H), 2.15 (dd, *J* = 9.3, 3.8 Hz, 1H), 1.80 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.94, 142.03, 139.40, 128.44, 127.22, 126.60, 115.53, 112.68, 81.68, 72.58, 65.53, 19.74; HRMS (ESI-MS): Calcd. for $C_{14}H_{18}O_2$ (M + Na): 241.1204, Found: 241.1209.

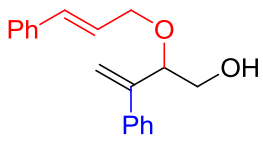
2-(but-3-en-1-yloxy)-3-phenylbut-3-en-1-ol (4ak)



4ak

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50–7.26 (m, 5H), 5.95–5.78 (m, 1H), 5.47 (d, *J* = 1.3 Hz, 1H), 5.37 (s, 1H), 5.21–5.04 (m, 2H), 4.34 (dd, *J* = 8.2, 3.4 Hz, 1H), 3.79–3.74 (m, 1H), 3.60–3.47 (m, 3H), 2.41 (td, *J* = 6.7, 1.2 Hz, 2H), 2.28 (dd, *J* = 9.3, 2.2 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.14, 139.35, 135.26, 128.43, 127.83, 126.55, 116.71, 115.25, 83.00, 68.33, 65.61, 34.32; HRMS (ESI-MS): Calcd. for $C_{14}H_{18}O_2$ (M + Na): 241.1204, Found: 241.1209.

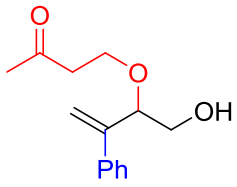
2-(cinnamyloxy)-3-phenylbut-3-en-1-ol (4al)



4al

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46–7.27 (m, 10H), 6.64 (d, *J* = 15.9 Hz, 1H), 6.35 (ddd, *J* = 15.9, 6.5, 5.7 Hz, 1H), 5.52 (d, *J* = 1.3 Hz, 1H), 5.44 (s, 1H), 4.48 (ddd, *J* = 7.5, 4.0, 0.7 Hz, 1H), 4.39 (ddd, *J* = 11.8, 6.4, 1.5 Hz, 1H), 4.18 (ddd, *J* = 11.8, 7.2, 1.3 Hz, 1H), 3.67–3.52 (m, 2H), 2.19 (dd, *J* = 9.1, 3.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 144.95, 139.31, 136.52, 132.75, 128.56, 128.48, 127.89, 127.79, 126.58, 126.49, 125.76, 115.60, 82.05, 69.48, 65.52; HRMS (ESI-MS): Calcd. for $C_{19}H_{20}O_2$ (M + Na): 303.1361, Found: 303.1355.

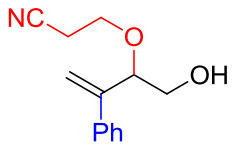
4-((1-hydroxy-3-phenylbut-3-en-2-yl)oxy)butan-2-one (4am)



4am

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78–7.04 (m, 5H), 5.46 (s, 1H), 5.35 (s, 1H), 4.36 (dd, *J* = 8.2, 3.1 Hz, 1H), 3.94–3.90 (m, 1H), 3.75 (ddd, *J* = 9.2, 7.5, 5.1 Hz, 1H), 3.60–3.56 (m, 1H), 3.53–3.42 (m, 1H), 2.89–2.68 (m, 2H), 2.62 (dd, *J* = 9.6, 3.2 Hz, 1H), 2.21 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 207.28, 145.07, 139.26, 128.41, 127.82, 126.50, 115.00, 83.36, 65.44, 64.08, 43.59, 30.37; HRMS (ESI-MS): Calcd. for $C_{14}H_{18}O_3$ (M + Na): 257.1154, Found: 257.1151.

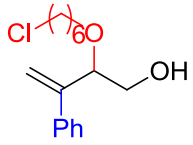
3-((1-hydroxy-3-phenylbut-3-en-2-yl)oxy)propanenitrile (4an)



4an

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52–7.29 (m, 5H), 5.53 (d, *J* = 0.7 Hz, 1H), 5.42 (s, 1H), 4.41 (dd, *J* = 7.2, 4.0 Hz, 1H), 3.91 (dt, *J* = 9.4, 6.3 Hz, 1H), 3.68 (dt, *J* = 9.4, 6.3 Hz, 1H), 3.63–3.57 (m, 2H), 2.74–2.57 (m, 2H), 2.26 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.29, 138.76, 128.56, 128.09, 126.42, 117.81, 115.78, 83.79, 65.49, 63.58, 19.08; HRMS (ESI-MS): Calcd. for $C_{13}H_{15}NO_2$ (M + Na): 240.1000, Found: 240.0996.

2-((6-chlorohexyl)oxy)-3-phenylbut-3-en-1-ol (4ao)

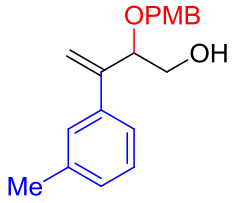


4ao

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47–7.28 (m, 5H), 5.47 (d, *J* = 1.4 Hz, 1H), 5.36 (s, 1H), 4.32 (dd, *J* = 8.1, 3.5 Hz, 1H), 3.80–3.18 (m, 6H), 2.19 (dd, *J* = 9.5, 3.3 Hz, 1H), 1.86–1.73 (m, 2H), 1.73–1.55 (m, 2H), 1.54–1.35 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.31, 139.38, 128.41,

127.81, 126.54, 115.19, 82.94, 69.00, 65.56, 45.01, 32.51, 30.28, 26.68, 25.55; HRMS (ESI-MS): Calcd. for $C_{16}H_{23}ClO_2$ (M + Na): 305.1284, Found: 305.1282.

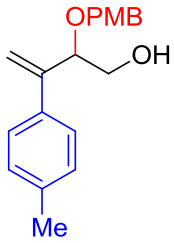
2-((4-methoxybenzyl)oxy)-3-(m-tolyl)but-3-en-1-ol (4cb)



4cb

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35–7.05 (m, 6H), 6.90 (d, *J* = 8.5 Hz, 2H), 5.52 (s, 1H), 5.44 (s, 1H), 4.70 (d, *J* = 11.1 Hz, 1H), 4.50–4.33 (m, 2H), 3.80 (s, 3H), 3.60–3.52 (m, 2H), 2.35 (s, 3H), 2.26 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.35, 145.09, 139.35, 138.04, 130.10, 129.64, 128.63, 128.35, 127.29, 123.65, 115.29, 113.93, 81.90, 70.49, 65.62, 55.29, 21.46; HRMS (ESI-MS): Calcd. for $C_{19}H_{22}O_3$ (M + Na): 321.1467, Found: 321.1464.

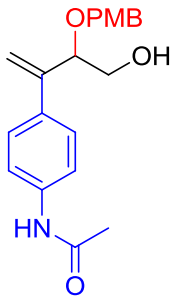
2-((4-methoxybenzyl)oxy)-3-(p-tolyl)but-3-en-1-ol (4db)



4db

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33–7.26 (m, 4H), 7.14 (d, *J* = 7.9 Hz, 2H), 6.97–6.81 (m, 2H), 5.52 (d, *J* = 1.4 Hz, 1H), 5.42 (s, 1H), 4.70 (d, *J* = 11.1 Hz, 1H), 4.47–4.42 (m, 1H), 4.40 (d, *J* = 11.1 Hz, 1H), 3.82 (s, 3H), 3.59–3.55 (m, 2H), 2.34 (s, 3H), 2.14 (d, *J* = 4.8 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.35, 144.74, 137.73, 136.39, 130.12, 129.63, 129.15, 126.42, 114.86, 113.94, 81.95, 70.46, 65.61, 55.29, 21.10; HRMS (ESI-MS): Calcd. for $C_{19}H_{22}O_3$ (M + Na): 321.1467, Found: 321.1460.

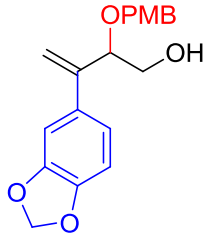
N-(4-(4-hydroxy-3-((4-methoxybenzyl)oxy)but-1-en-2-yl)phenyl)acetamide (4eb)



4eb

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.47 (d, *J* = 8.6 Hz, 2H), 7.35 (d, *J* = 8.6 Hz, 2H), 7.28 (d, *J* = 8.6 Hz, 2H), 6.89 (d, *J* = 8.6 Hz, 2H), 5.51 (s, 1H), 5.41 (s, 1H), 4.67 (d, *J* = 11.1 Hz, 1H), 4.43–4.38 (m, 2H), 3.80 (s, 3H), 3.62–3.52 (m, 2H), 2.40 (s, 1H), 2.15 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.42, 159.34, 144.14, 137.62, 135.10, 129.99, 129.63, 127.15, 119.74, 115.21, 113.93, 81.85, 70.44, 65.47, 55.27, 24.53; HRMS (ESI-MS): Calcd. for $C_{20}H_{23}NO_4$ (M + H): 342.1705, Found: 342.1700.

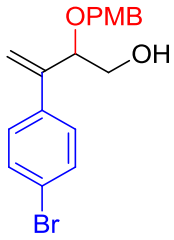
3-(benzo[d][1,3]dioxol-5-yl)-2-((4-methoxybenzyl)oxy)but-3-en-1-ol (4fb)



4fb

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.5 Hz, 2H), 6.93–6.85 (m, 4H), 6.76 (d, *J* = 8.1 Hz, 1H), 5.94 (s, 2H), 5.45 (d, *J* = 1.2 Hz, 1H), 5.37 (s, 1H), 4.67 (d, *J* = 11.1 Hz, 1H), 4.42–4.27 (m, 2H), 3.80 (s, 3H), 3.59–3.43 (m, 2H), 2.28 (d, *J* = 5.3 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.31, 147.69, 147.28, 144.43, 133.41, 129.58, 128.54, 120.07, 114.81, 113.88, 108.13, 107.20, 101.05, 82.09, 70.39, 65.43, 55.22; HRMS (ESI-MS): Calcd. for $C_{19}H_{20}O_5$ (M + Na): 351.1208, Found: 351.1203.

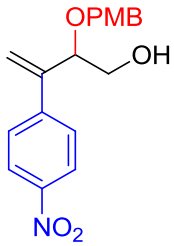
3-(4-bromophenyl)-2-((4-methoxybenzyl)oxy)but-3-en-1-ol (4gb)



4gb

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50–7.42 (m, 2H), 7.30–7.27 (m, 4H), 6.95–6.84 (m, 2H), 5.55 (d, *J* = 1.1 Hz, 1H), 5.47 (s, 1H), 4.68 (d, *J* = 11.0 Hz, 1H), 4.42–4.24 (m, 2H), 3.82 (s, 3H), 3.58–3.53 (m, 2H), 2.13 (dd, *J* = 6.6, 6.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.38, 143.94, 138.09, 131.57, 129.78, 129.66, 128.28, 121.97, 116.48, 113.95, 81.80, 70.51, 65.37, 55.29; HRMS (ESI-MS): Calcd. for $C_{18}H_{19}BrO_3$ (M + Na): 385.0415, Found: 385.0423.

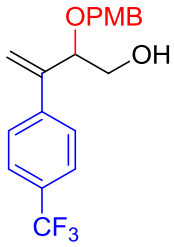
2-((4-methoxybenzyl)oxy)-3-(4-nitrophenyl)but-3-en-1-ol (4hb)



4hb

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.21–8.17 (m, 2H), 7.61–7.54 (m, 2H), 7.30–7.26 (m, 2H), 6.94–6.87 (m, 2H), 5.68 (d, *J* = 0.5 Hz, 1H), 5.63 (s, 1H), 4.68 (d, *J* = 11.1 Hz, 1H), 4.46–4.30 (m, 2H), 3.82 (s, 3H), 3.57 (d, *J* = 6.8 Hz, 2H), 2.15 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.51, 147.34, 145.78, 143.65, 129.64, 129.48, 127.57, 123.71, 119.34, 114.02, 81.66, 70.70, 65.17, 55.30; HRMS (ESI-MS): Calcd. for $C_{18}H_{19}NO_5$ (M + Na): 352.1161, Found: 352.1164.

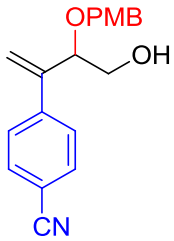
2-((4-methoxybenzyl)oxy)-3-(4-(trifluoromethyl)phenyl)but-3-en-1-ol (4ib)



4ib

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.2 Hz, 2H), 7.51 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 8.5 Hz, 2H), 6.91 (d, *J* = 8.5 Hz, 2H), 5.60 (s, 1H), 5.55 (s, 1H), 4.69 (d, *J* = 11.1 Hz, 1H), 4.49–4.37 (m, 2H), 3.82 (s, 3H), 3.56 (t, *J* = 5.7 Hz, 2H), 2.13 (d, *J* = 5.8 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.47, 144.15, 142.87, 129.72, 129.64, 127.05, 125.42, 125.39, 117.81, 114.00, 81.82, 70.62, 65.29, 55.30; HRMS (ESI-MS): Calcd. for $C_{19}H_{19}F_3O_3$ (M + Na): 375.1184, Found: 375.1181.

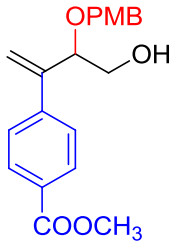
4-(4-hydroxy-3-((4-methoxybenzyl)oxy)but-1-en-2-yl)benzonitrile (4jb)



4jb

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 8.6 Hz, 2H), 6.90 (d, *J* = 8.6 Hz, 2H), 5.63 (s, 1H), 5.58 (s, 1H), 4.67 (d, *J* = 11.1 Hz, 1H), 4.48–4.35 (m, 2H), 3.82 (s, 3H), 3.58–3.51 (m, 2H), 2.17 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.47, 143.87, 143.81, 132.24, 129.61, 129.53, 127.39, 118.73, 118.64, 113.99, 111.51, 81.62, 70.64, 65.19, 55.28; HRMS (ESI-MS): Calcd. for $C_{19}H_{19}NO_3$ (M + Na): 332.1263, Found: 332.1254.

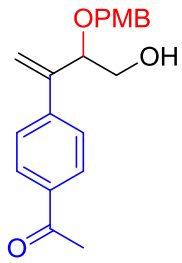
methyl 4-(4-hydroxy-3-((4-methoxybenzyl)oxy)but-1-en-2-yl)benzoate (4kb)



4kb

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03–7.96 (m, 2H), 7.49–7.45 (m, 2H), 7.31–7.26 (m, 2H), 6.95–6.83 (m, 2H), 5.63 (d, *J* = 1.0 Hz, 1H), 5.54 (s, 1H), 4.69 (d, *J* = 11.1 Hz, 1H), 4.47–4.38 (m, 2H), 3.92 (s, 3H), 3.82 (s, 3H), 3.56 (d, *J* = 6.1 Hz, 2H), 2.20 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.74, 159.41, 144.38, 143.81, 129.78, 129.74, 129.63, 129.46, 117.48, 113.96, 81.71, 70.58, 65.39, 55.27, 52.13; HRMS (ESI-MS): Calcd. for $C_{20}H_{22}O_5$ (M + Na): 365.1365, Found: 365.1360.

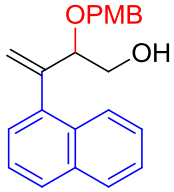
1-(4-(4-hydroxy-3-((4-methoxybenzyl)oxy)but-1-en-2-yl)phenyl)ethanone (4lb)



4lb

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98–7.81 (m, 2H), 7.54–7.45 (m, 2H), 7.29 (d, *J* = 8.6 Hz, 2H), 6.95–6.85 (m, 2H), 5.64 (d, *J* = 1.0 Hz, 1H), 5.56 (s, 1H), 4.69 (d, *J* = 11.1 Hz, 1H), 4.47–4.41 (m, 2H), 3.82 (s, 3H), 3.57 (t, *J* = 5.7 Hz, 2H), 2.61 (s, 3H), 2.16 (dd, *J* = 6.7, 6.1 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.57, 159.44, 144.32, 143.97, 136.38, 129.76, 129.64, 128.55, 126.82, 117.68, 113.98, 81.69, 70.61, 65.40, 55.29, 26.61; HRMS (ESI-MS): Calcd. for $C_{20}H_{22}O_4$(M + H): 327.1596, Found: 327.1597.

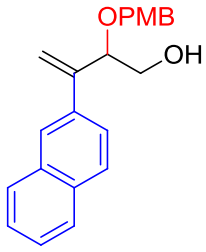
2-((4-methoxybenzyl)oxy)-3-(naphthalen-1-yl)but-3-en-1-ol (4mb)



4mb

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05–7.99 (m, 1H), 7.86–7.84 (m, 1H), 7.79 (d, *J* = 8.2 Hz, 1H), 7.52–7.41 (m, 3H), 7.37 (d, *J* = 8.5 Hz, 2H), 7.29 (d, *J* = 7.0 Hz, 1H), 6.93 (d, *J* = 8.6 Hz, 2H), 5.85 (s, 1H), 5.41 (d, *J* = 1.7 Hz, 1H), 4.92 (d, *J* = 11.0 Hz, 1H), 4.60 (d, *J* = 11.0 Hz, 1H), 4.36 (dd, *J* = 7.1, 2.9 Hz, 1H), 3.82 (s, 3H), 3.61–3.33 (m, 2H), 2.09 (d, *J* = 4.2 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.42, 144.11, 137.82, 133.75, 131.55, 130.10, 129.58, 128.41, 127.84, 126.08, 125.85, 125.80, 125.20, 125.11, 117.77, 114.00, 83.08, 71.06, 64.62, 55.30; HRMS (ESI-MS): Calcd. for $C_{22}H_{22}O_3$ (M + Na): 357.1467, Found: 357.1469.

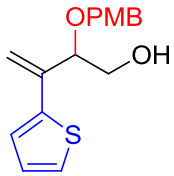
2-((4-methoxybenzyl)oxy)-3-(naphthalen-2-yl)but-3-en-1-ol (4nb)



4nb

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88–7.76 (m, 4H), 7.56 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.51–7.42 (m, 2H), 7.35–7.26 (m, 2H), 6.95–6.79 (m, 2H), 5.69 (d, *J* = 1.1 Hz, 1H), 5.56 (s, 1H), 4.77 (d, *J* = 6.0 Hz, 1H), 4.59 (dd, *J* = 7.3, 3.4 Hz, 1H), 4.46 (d, *J* = 11.1 Hz, 1H), 3.82 (s, 3H), 3.67–3.59 (m, 2H), 2.23 (dd, *J* = 8.8, 3.9 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.38, 144.79, 136.53, 133.23, 132.89, 130.04, 129.68, 128.61, 128.15, 128.09, 127.53, 126.31, 126.12, 125.23, 124.83, 116.07, 113.96, 113.92, 81.88, 70.56, 65.68, 55.28; HRMS (ESI-MS): Calcd. for $C_{22}H_{22}O_3$ (M + Na): 357.1467, Found: 357.1474.

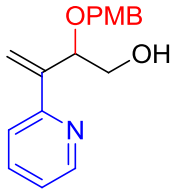
2-((4-methoxybenzyl)oxy)-3-(thiophen-2-yl)but-3-en-1-ol (4ob)



4ob

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30–7.26 (m, 2H), 7.22 (dd, *J* = 5.1, 1.0 Hz, 1H), 7.17 (dd, *J* = 3.6, 1.0 Hz, 1H), 7.00 (dd, *J* = 10.3, 6.7 Hz, 1H), 6.92–6.85 (m, 2H), 5.66 (s, 1H), 5.35 (s, 1H), 4.66 (d, *J* = 11.1 Hz, 1H), 4.46–4.31 (m, 2H), 3.81 (s, 3H), 3.72–3.62 (m, 2H), 2.16 (dd, *J* = 12.9, 5.3 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.38, 141.65, 138.41, 129.83, 129.74, 127.42, 124.91, 124.09, 114.05, 113.91, 82.00, 70.53, 65.68, 55.28; HRMS (ESI-MS): Calcd. for $C_{16}H_{18}SO_3$ (M + Na): 313.0874, Found: 313.0876.

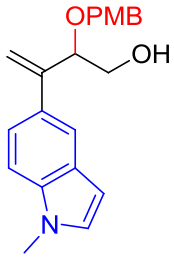
2-((4-methoxybenzyl)oxy)-3-(pyridin-2-yl)but-3-en-1-ol (4pb)



4pb

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.55 (d, *J* = 4.8 Hz, 1H), 7.69 (td, *J* = 7.7, 1.8 Hz, 1H), 7.57 (d, *J* = 8.0 Hz, 1H), 7.27–7.18 (m, 3H), 6.87 (d, *J* = 8.6 Hz, 2H), 5.88 (s, 1H), 5.71 (s, 1H), 4.75 (t, *J* = 5.4 Hz, 1H), 4.59 (d, *J* = 11.2 Hz, 1H), 4.38 (d, *J* = 11.2 Hz, 1H), 3.83–3.77 (m, 4H), 3.73 (dd, *J* = 11.2, 6.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.23, 157.15, 148.36, 146.21, 136.83, 130.25, 129.44, 122.45, 121.23, 117.62, 113.82, 80.09, 70.68, 66.24, 55.25; HRMS (ESI-MS): Calcd. for $C_{17}H_{20}NO_3$ (M + H): 286.1443, Found: 286.1444.

2-((4-methoxybenzyl)oxy)-3-(1-methyl-1H-indol-5-yl)but-3-en-1-ol (4qb)

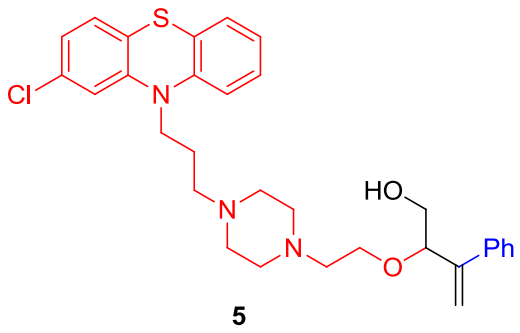


4qb

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 (s, 1H), 7.38–7.27 (m, 4H), 7.06 (d, *J* = 3.1 Hz, 1H), 6.93–6.77 (m, 2H), 6.47 (d, *J* = 3.1 Hz, 1H), 5.54 (d, *J* = 1.5 Hz, 1H), 5.43 (s, 1H), 4.76 (d, *J* = 11.1 Hz, 1H), 4.56 (dd, *J* = 7.8, 3.6 Hz, 1H), 4.45 (d, *J* = 11.1 Hz, 1H), 3.83 (s, 3H), 3.80 (s, 3H), 3.68–3.53 (m, 2H), 2.16 (dd, *J* = 8.9, 3.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.29, 145.62, 136.36, 130.72, 130.29, 129.68, 129.47, 128.39, 120.63, 118.79, 113.89, 109.14, 101.24, 82.34, 70.43, 65.77, 55.29, 32.91; HRMS (ESI-MS): Calcd. for $C_{21}H_{23}O_3$ (M + Na): 360.1576, Found: 360.1573.

Characterization of product 5

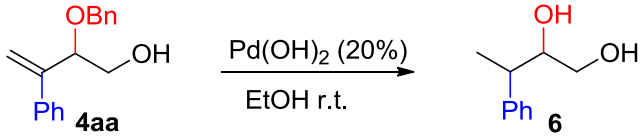
2-(2-(4-(3-(2-chloro-10H-phenothiazin-10-yl)propyl)piperazin-1-yl)ethoxy)-3-phenylbut-3-en-1-ol (5)



5

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44–7.26 (m, 5H), 7.18–7.06 (m, 2H), 7.01 (d, *J* = 8.2 Hz, 1H), 6.97–6.75 (m, 4H), 5.40 (d, *J* = 7.4 Hz, 2H), 4.37 (dd, *J* = 8.7, 2.4 Hz, 1H), 3.94–3.88 (m, 2H), 3.57–3.47 (m, 3H), 2.86–2.36 (m, 12H), 2.01–1.89 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 146.43, 145.74, 144.43, 139.43, 133.14, 128.38, 127.83, 127.73, 127.46, 127.36, 126.46, 124.69, 123.42, 122.83, 122.17, 115.76, 115.72, 114.27, 83.03, 65.88, 65.29, 57.32, 55.25, 52.93, 52.81, 45.27, 24.17; HRMS (ESI-MS): Calcd. for $C_{31}H_{36}ClN_3O_2S$ (M + H): 550.2295, Found: 550.2302.

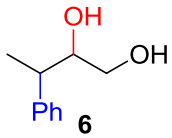
Procedure for reduction of 4aa to diol 6



To a 5 mL reaction vial containing a stirring bar, **4aa** (0.4 mmol) in EtOH (1.0 mL) was added. Then, $Pd(OH)_2$ (11.23 mg, 20 mol%) was added to the reaction mixture. The reaction mixture was vigorously stirred in the presence of hydrogen gas at room temperature for 18 hours. The precipitate was filtered out with celite and washed with ethanol, and the filtrate was removed in vacuum. The residue was purified by flash column chromatography on silica gel to afford product **6** (30.5 mg, 92%).

Characterization of diol 6

3-phenylbutane-1,2-diol (6)



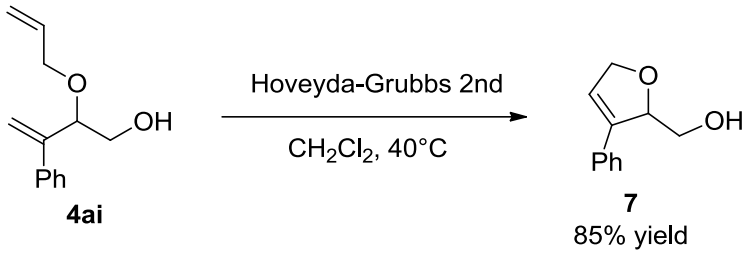
6

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.21 (m, 10H), 3.87–3.73 (m, 3H), 3.66–3.54 (m, 1H), 3.48 (dd, *J* = 11.2, 3.1 Hz, 1H), 3.38 (dd, *J* = 11.2, 7.6 Hz, 1H), 2.92–2.85 (m, 1H), 2.87–2.77 (m, 1H), 2.42 (s, 1H), 2.20 (s, 1H), 2.00 (s, 1H), 1.74 (s, 1H), 1.39 (d, *J* = 7.0 Hz, 3H), 1.30 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.59, 143.04, 128.74, 128.60, 127.95, 127.53, 126.91, 126.65, 76.67, 76.29, 65.07, 64.57, 42.92, 42.80, 17.82, 17.31; HRMS (ESI-MS): Calcd. for $C_{10}H_{14}O_2$ (M + Na): 189.0891, Found: 189.0890.

Procedure for ring-closing metathesis of 4ai to cyclic ethers 7

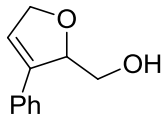
Under nitrogen atmosphere, **4ai** (40.86 mg, 0.2 mmol), Hoveyda-Grubbs 2nd catalyst (6.26 mg, 5 mol%), and CH_2Cl_2 (2.0 mL) were added into a Schlenk flask in order and the mixture was refluxed at 40℃ for overnight. After cooling to room temperature, the reaction mixture was concentrated under reduce pressure and the resulting residue was chromatographed on silica gel (EA/PE = 1/10) to give the

desired product **7** as a tan oil (30.0 mg, 85%).



Characterization of cyclic ethers 7

(3-phenyl-2,5-dihydrofuran-2-yl)methanol (7)



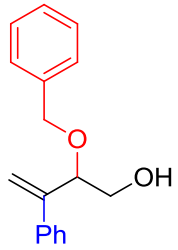
7

Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45–7.27 (m, 5H), 6.23 (d, *J* = 1.8 Hz, 1H), 5.42–5.33 (m, 1H), 4.88–4.81 (m, 2H), 3.99–3.80 (m, 1H), 3.75–3.57 (m, 1H), 1.93 (dd, *J* = 12.7, 6.3 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 138.57, 132.64, 128.74, 128.11, 126.24, 123.39, 86.50, 75.54, 64.14; HRMS (ESI-MS): Calcd. for $C_{11}H_{12}O_2$ (M + Na): 199.0735, Found: 199.0729.

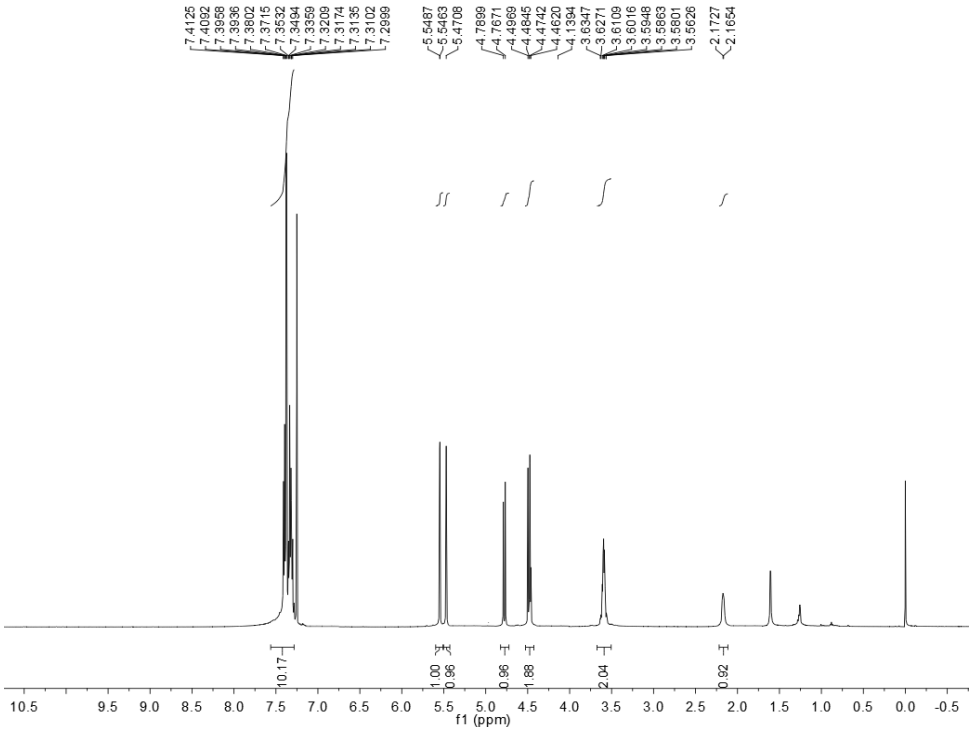
Reference

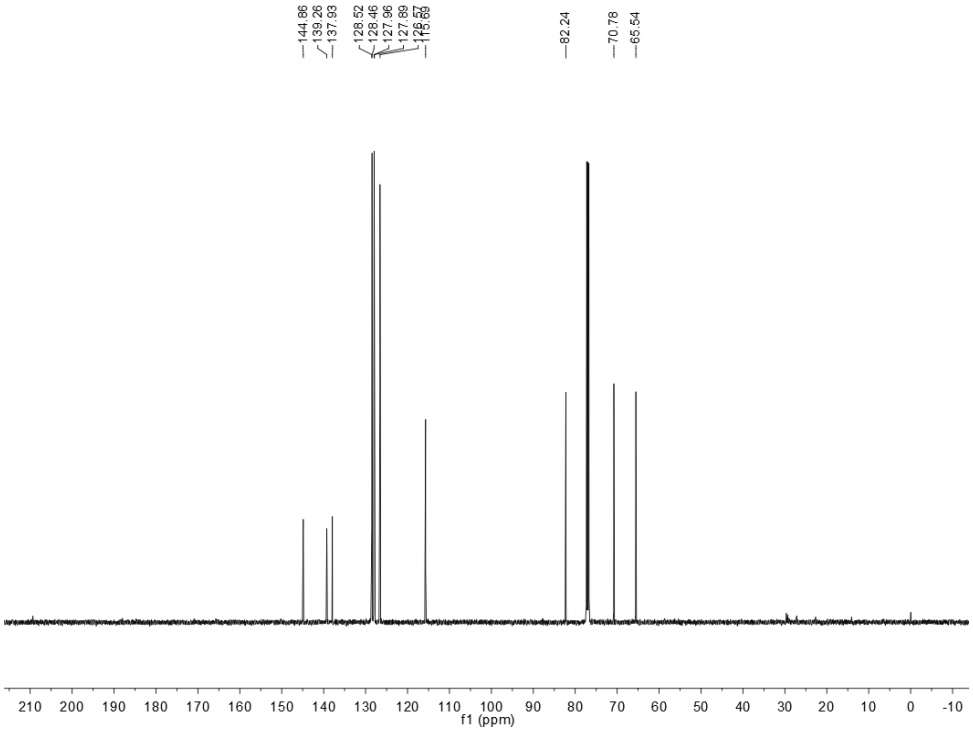
1. F. Lehrich, H. Hopf, J. Grunenberg, *Eur. J. Org. Chem*. 2011, **14**, 2705.

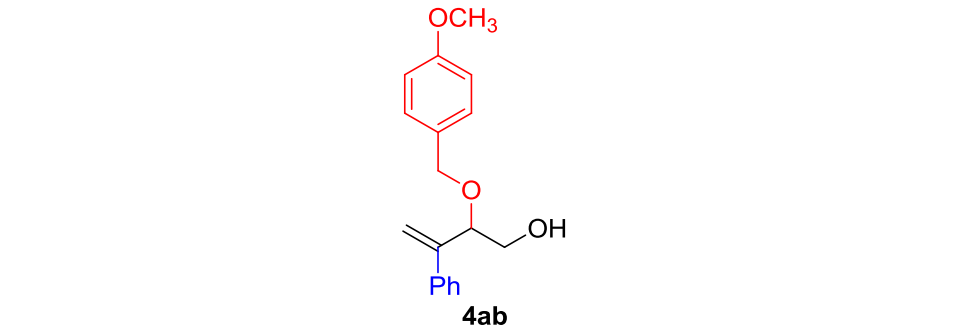
Copies of ^{1}H and ^{13}C NMR spectra



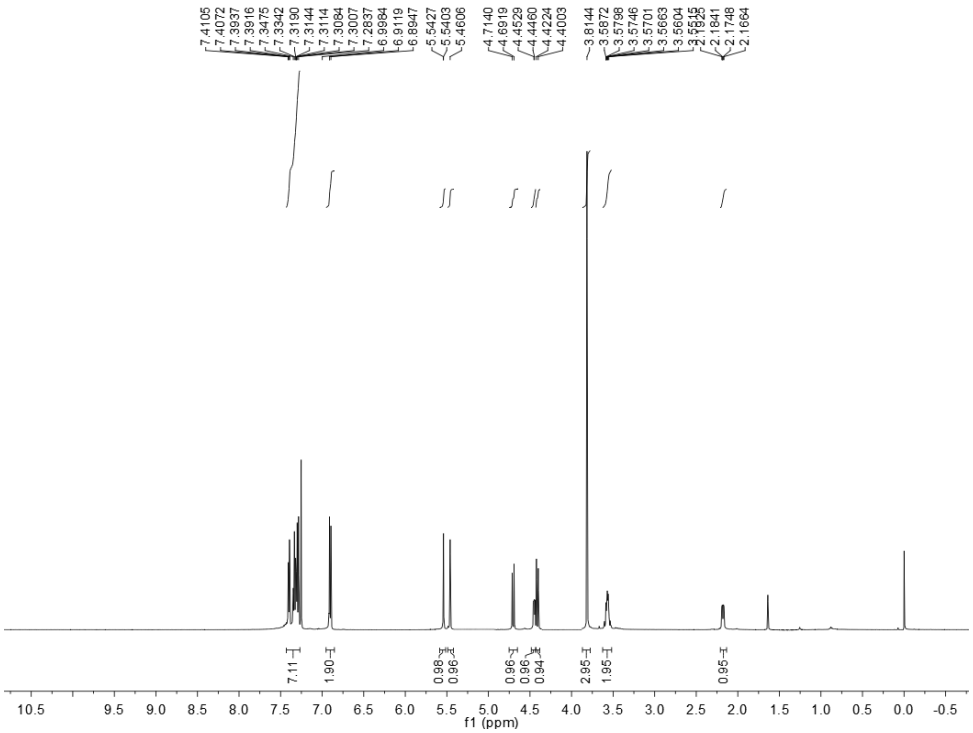
4aa






OCH3
O
OH
Ph

4ab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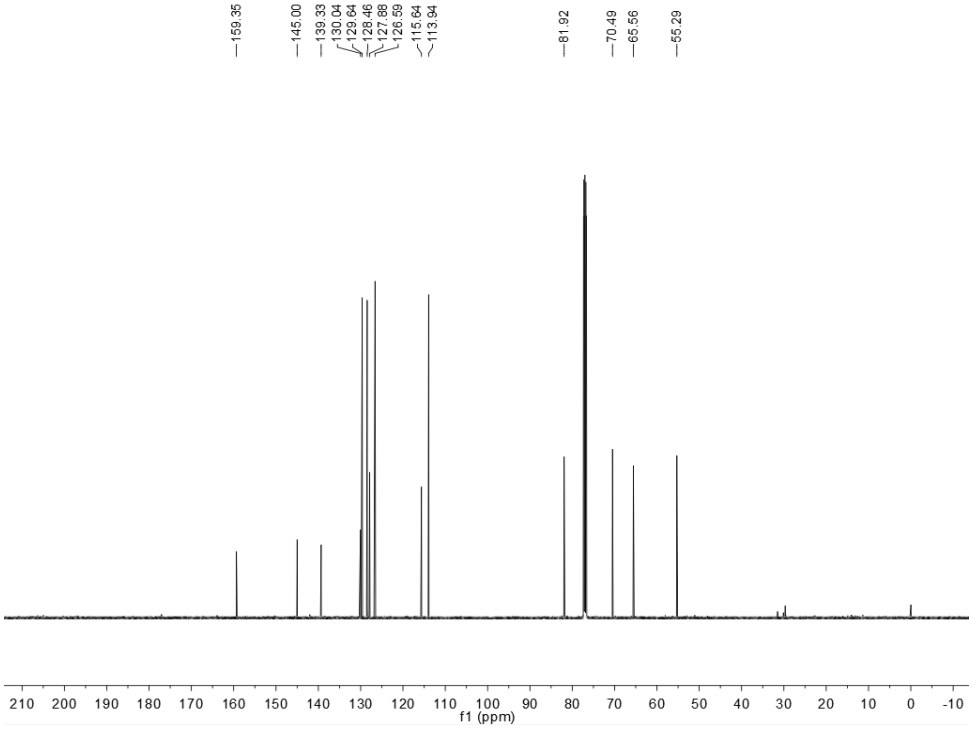
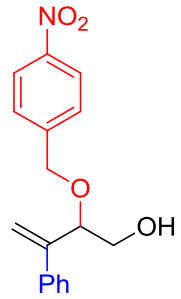
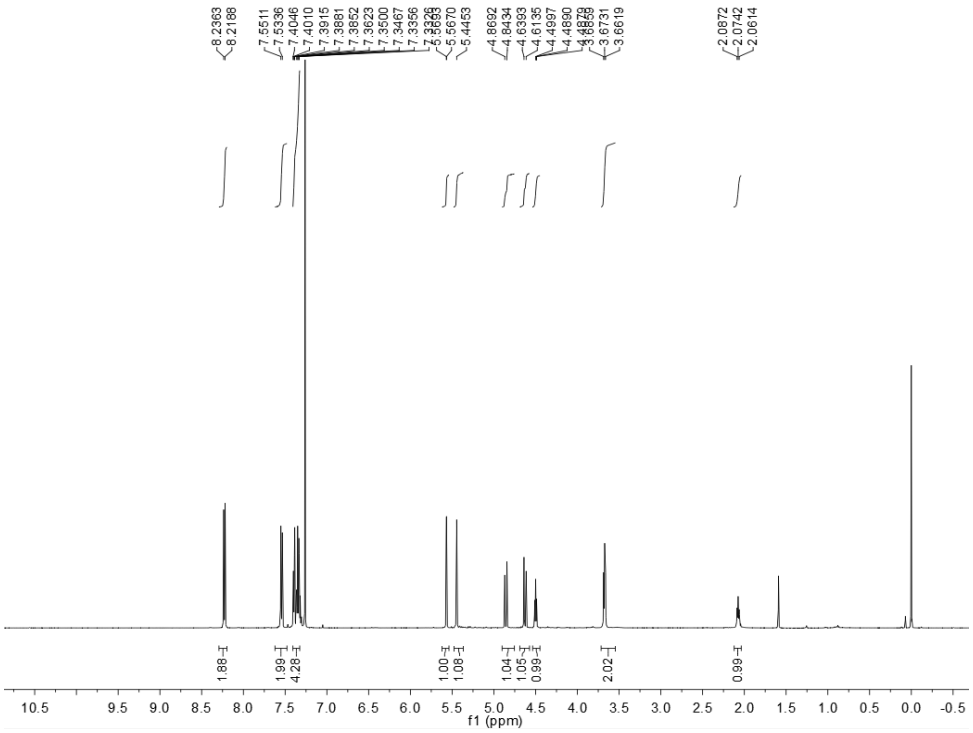
7.3475
7.3342
7.3190
7.3144
7.3114
7.3084
7.3007
7.2837
6.9984
6.9119
6.8947
5.5427
5.5403
5.4606
4.7140
4.6919
4.4529
4.4460
4.4224
4.4003
3.8144
3.5872
3.5798
3.5746
3.5701
3.5663
3.5604
2.1925
2.1841
2.1748
2.1664
7.11
1.90
0.98
0.96
0.96
0.96
0.94
2.95
1.95
0.95
f1 (ppm)


159.35
145.00
139.33
130.04
129.64
128.46
127.88
126.59
115.64
113.94
81.92
70.49
65.56
55.29
f1 (ppm)


NO2
O
OH
Ph

4ac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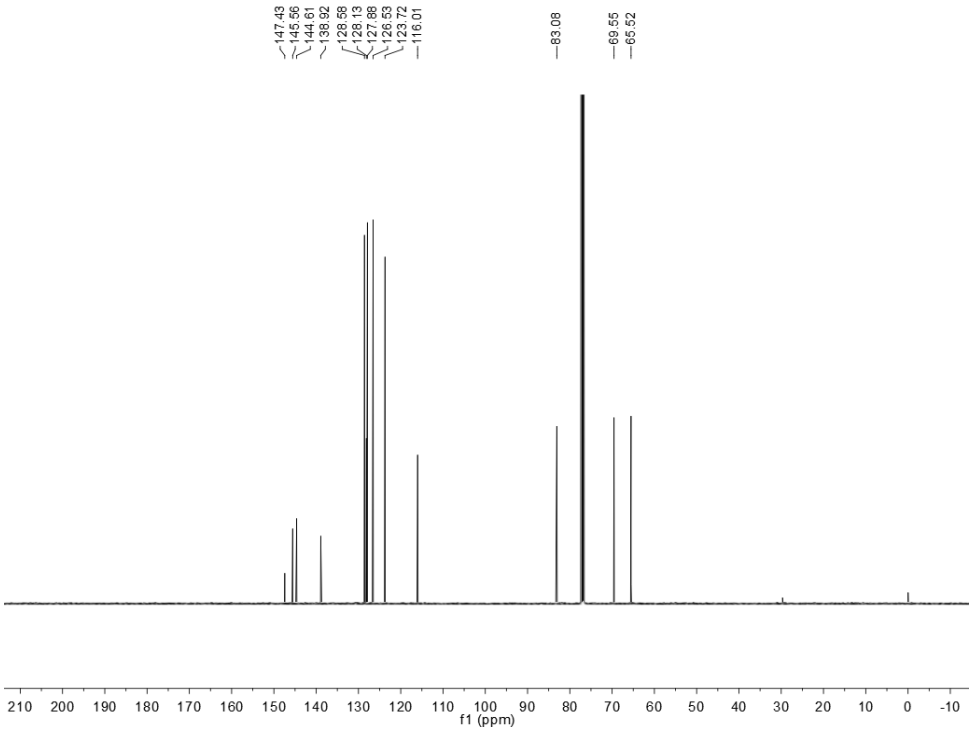
7.5336
7.4046
7.4010
7.3915
7.3881
7.3852
7.3623
7.3500
7.3467
7.3356
5.5693
5.5670
5.4453
4.8692
4.8434
4.6393
4.6135
4.4997
4.4890
3.6731
3.6619
2.0872
2.0742
2.0614
1.88
1.99
4.28
1.00
1.08
1.04
1.05
0.99
2.02
0.99
f1 (ppm)


147.43
145.56
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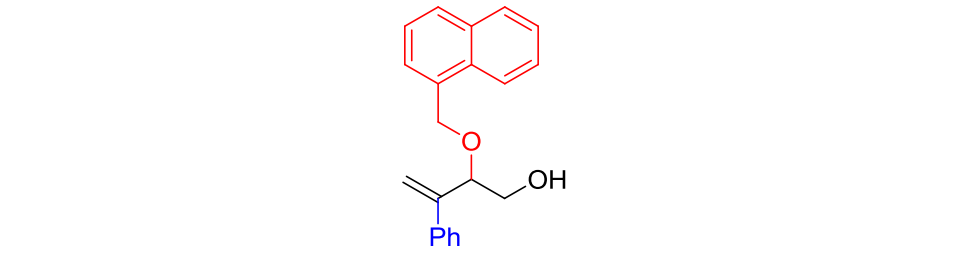
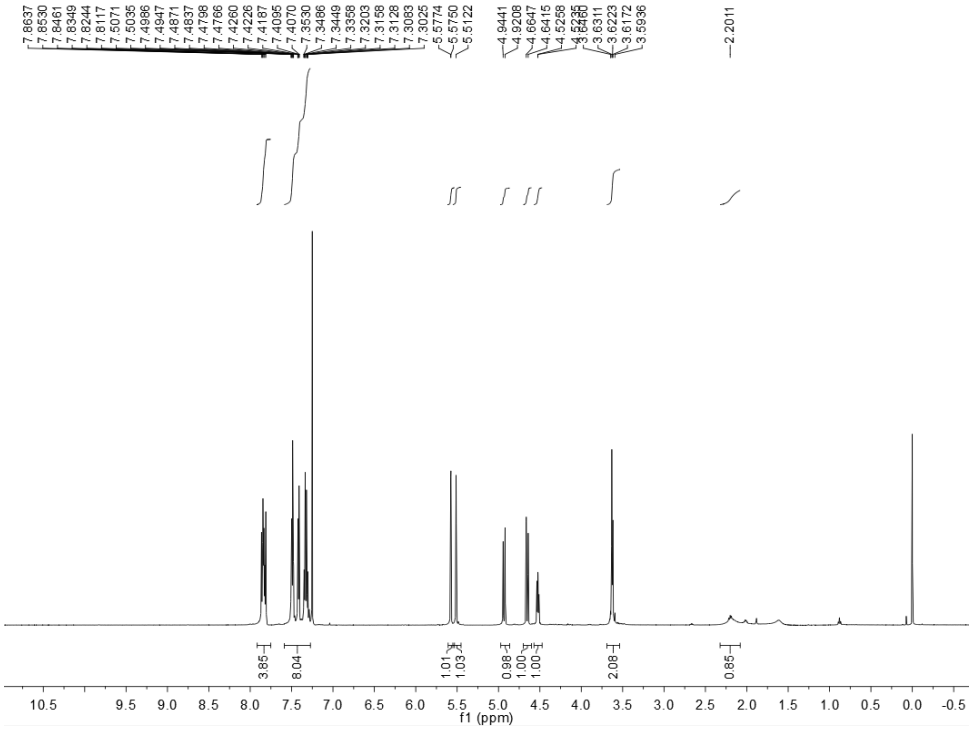
128.13
127.88
126.53
123.72
116.01
83.08
69.55
65.52
f1 (ppm)


O
OH
Ph

4ad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7.8349
7.8244
7.8117
7.5071
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7.4871
7.4837
7.4798
7.4766
7.4260
7.4226
7.4187
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7.4070
7.3530
7.3486
7.3449
7.3358
7.3203
7.3158
7.3128
7.3083
7.3025
5.5774
5.5750
5.5122
4.9441
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3.6172
3.5936
2.2011
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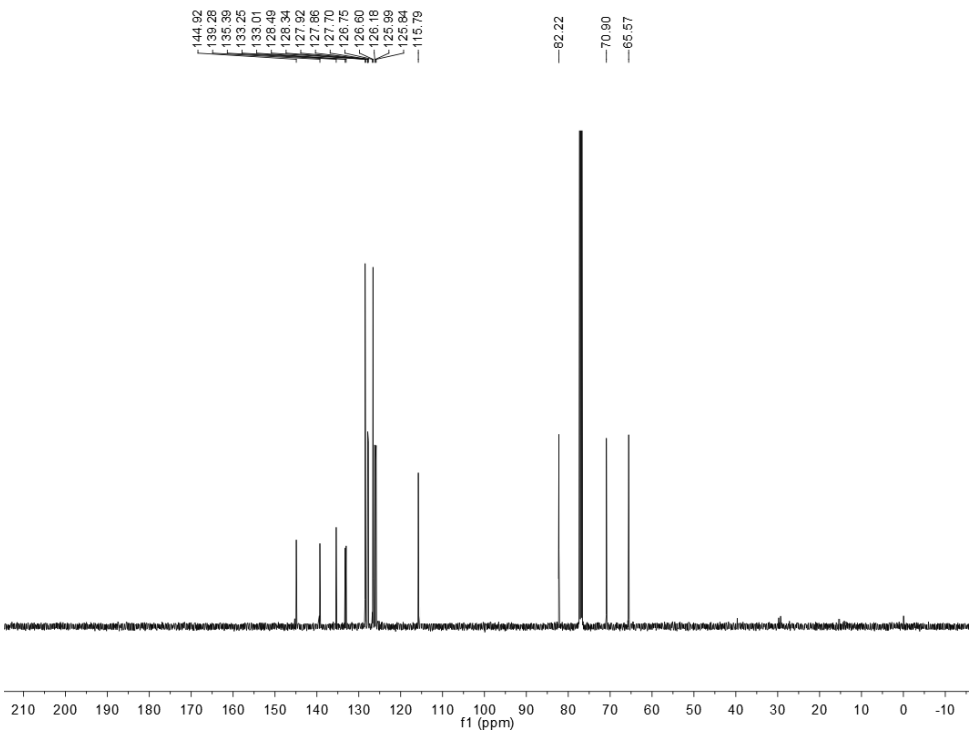
8.04
1.01
1.03
0.98
1.00
1.00
2.08
0.85
f1 (ppm)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139.28
135.39
133.25
133.01
128.49
128.34
127.92
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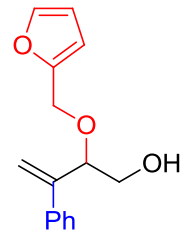
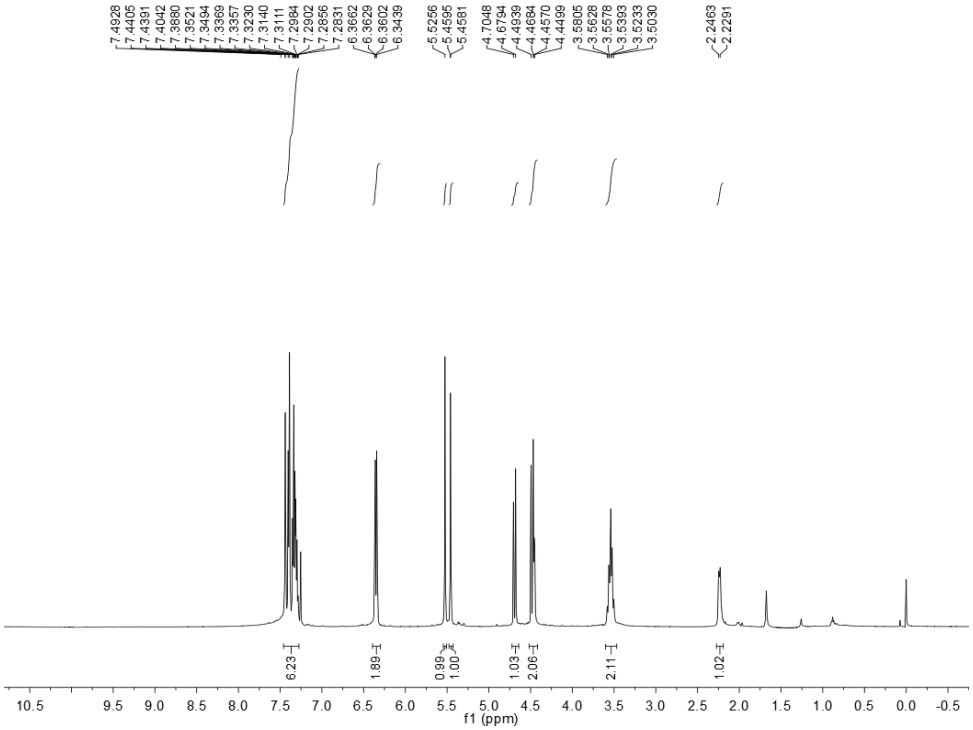
127.70
126.75
126.60
126.18
125.99
125.84
115.79
82.22
70.90
65.57
f1 (ppm)


O
O
OH
Ph

4ae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7.3357
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7.2984
7.2902
7.2856
7.2831
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3.5805
3.5628
3.5578
3.5393
3.5233
3.5030
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0.99
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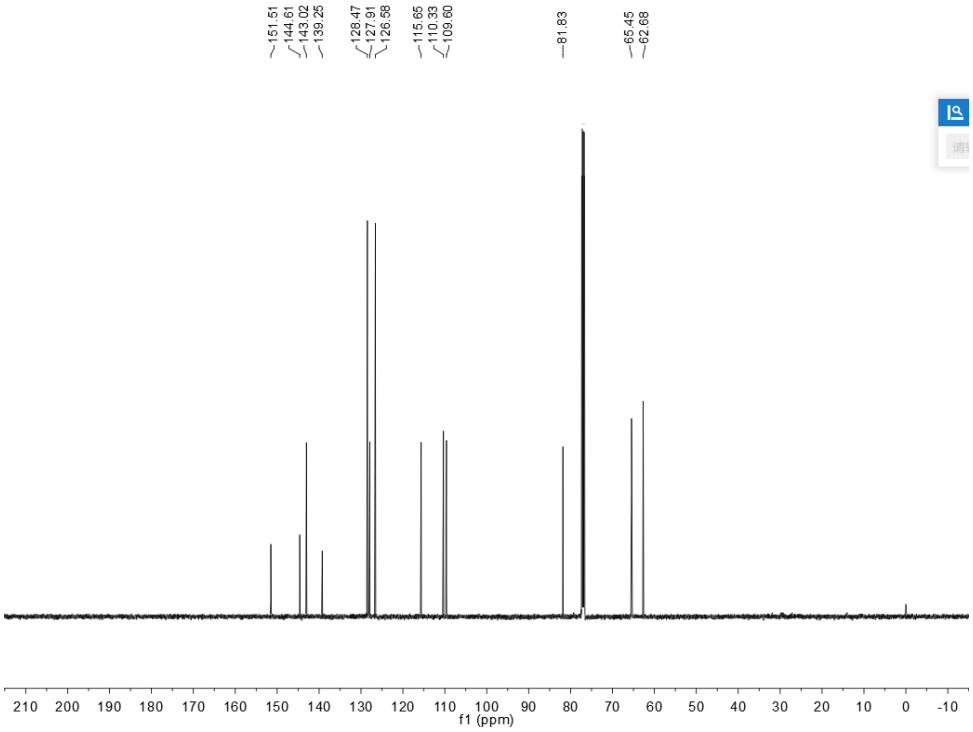
2.06
2.11
1.02
f1 (ppm)


151.51
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127.91
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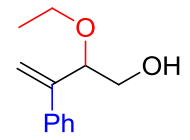
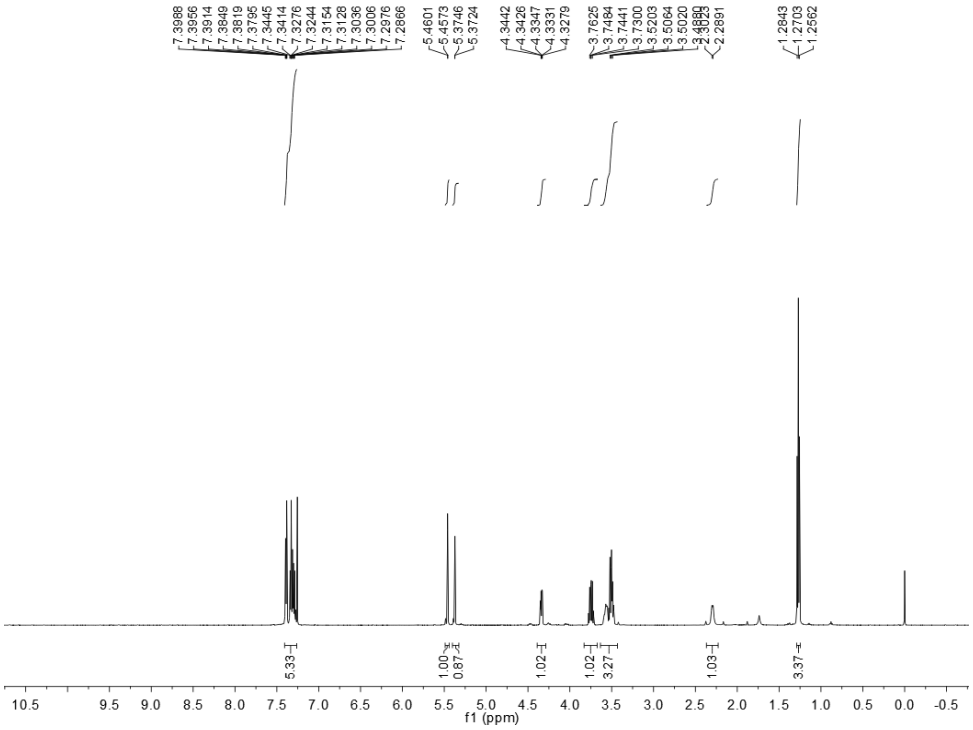
115.65
110.33
109.60
81.83
65.45
62.68
f1 (ppm)


O
OH
Ph

4af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7.3445
7.3414
7.3276
7.3244
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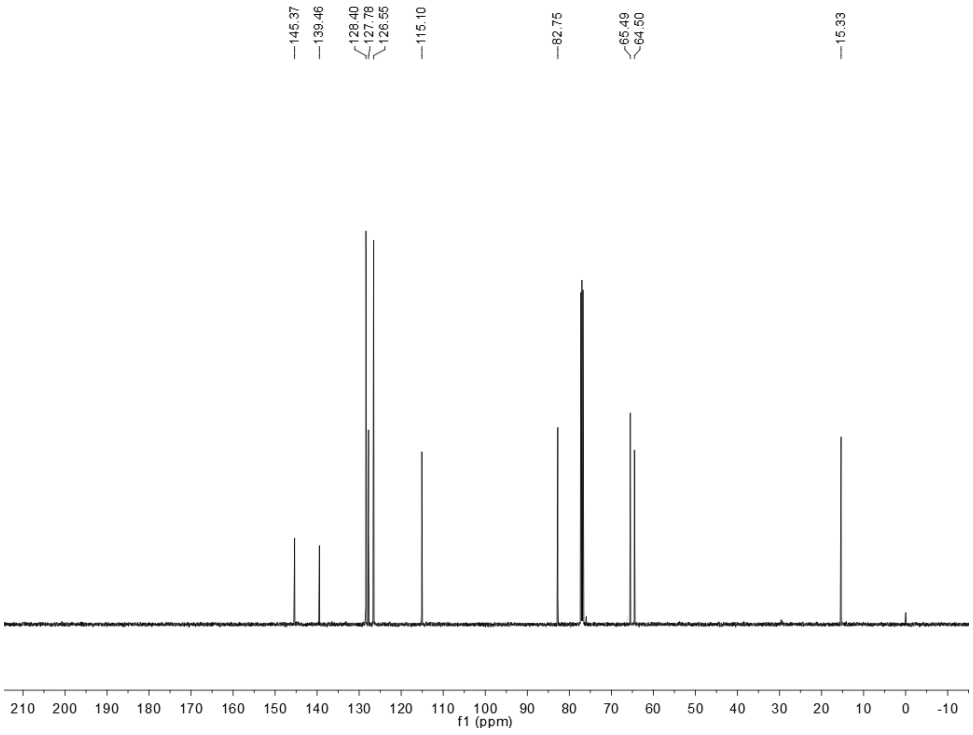
7.3128
7.3036
7.3006
7.2976
7.2866
5.4601
5.4573
5.3746
5.3724
4.3442
4.3426
4.3347
4.3331
4.3279
3.7625
3.7484
3.7441
3.7300
3.5203
3.5064
3.5020
2.2891
1.2843
1.2703
1.2562
5.33
1.00
0.87
1.02
1.02
3.27
1.03
3.37
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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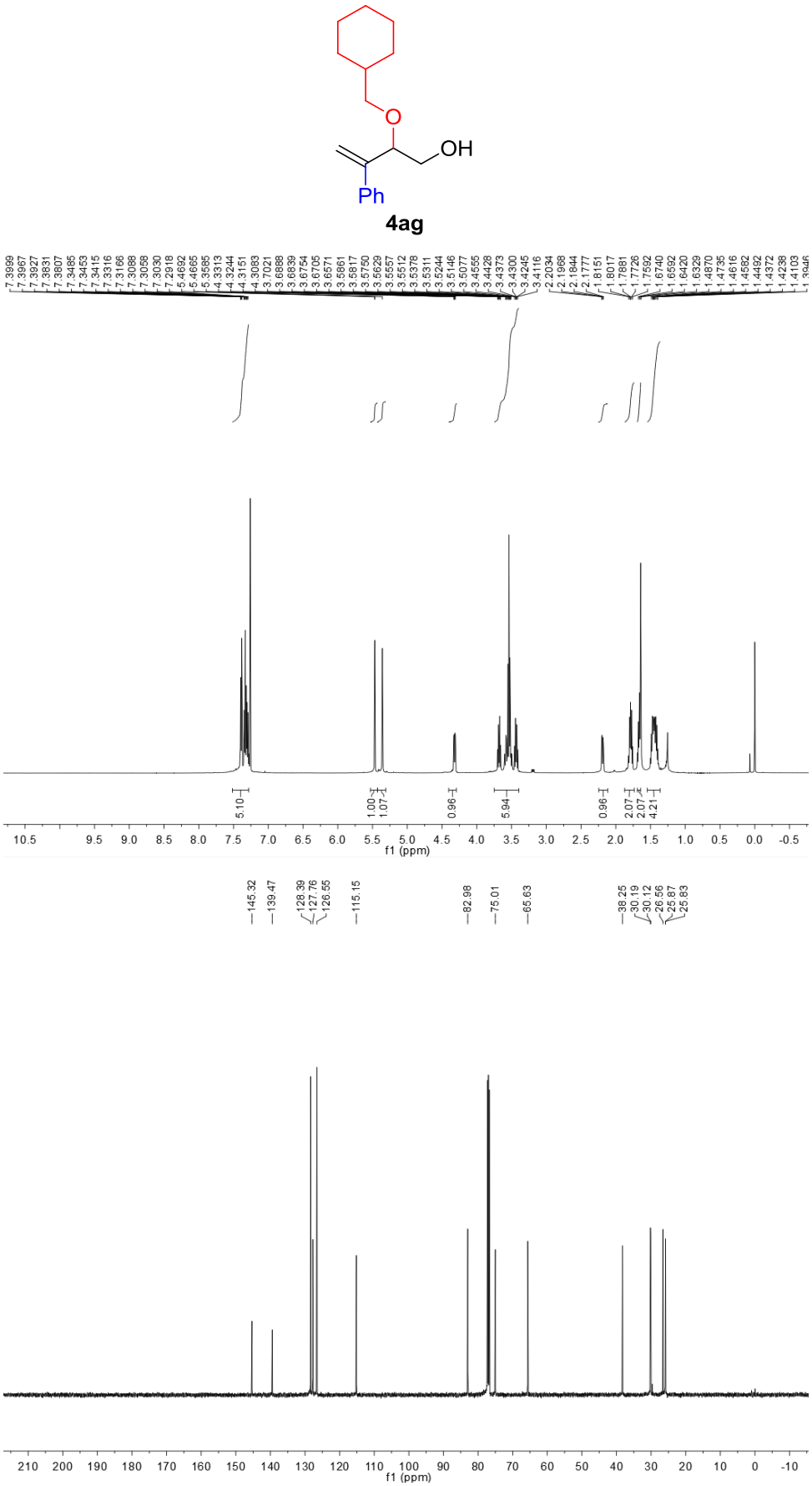
139.46
128.40
127.78
126.55
115.10
82.75
65.49
64.50
15.33
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


O
OH
Ph
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7.3453
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5.4665
5.3585
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4.3151
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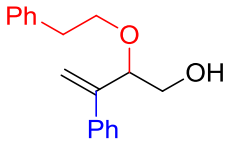
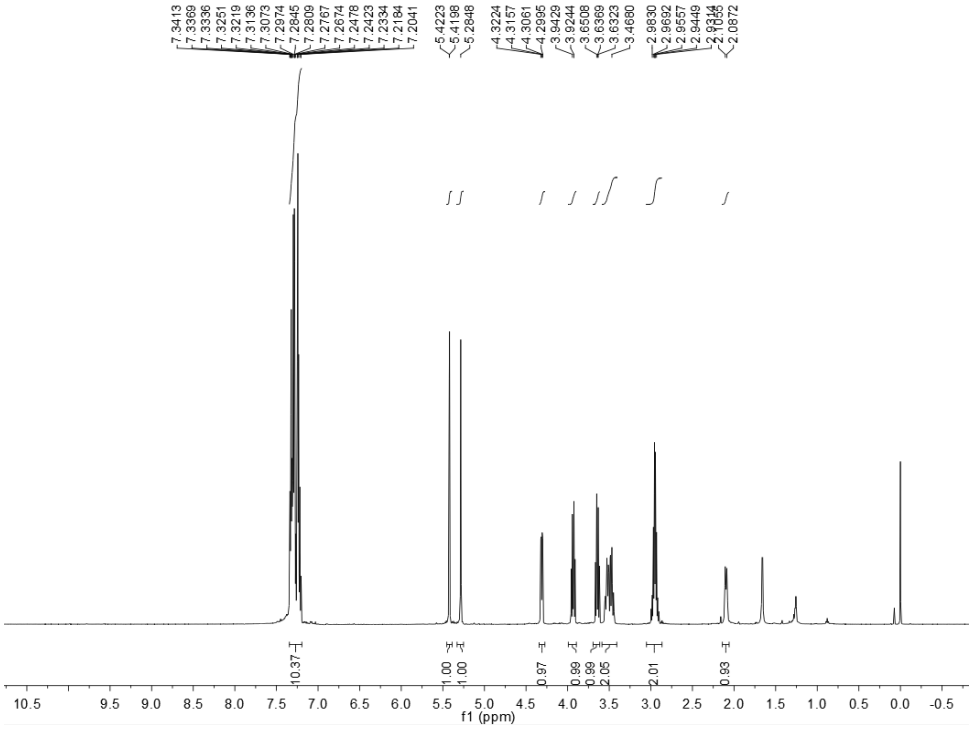
3.4116
2.2034
2.1968
2.1844
2.1777
2.1151
1.8017
1.7881
1.7726
1.7592
1.6740
1.6592
1.6420
1.6329
1.4870
1.4735
1.4616
1.4582
1.4492
1.4372
1.4238
1.4103
5.10
1.00
1.07
0.96
5.94
0.96
2.07
2.07
4.21
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
145.32
139.47
128.39
127.76
126.55
115.15
82.98
75.01
65.63
38.25
30.19
30.12
26.56
25.87
25.83
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

4ag


Ph
O
OH
Ph

4ah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7.2674
7.2478
7.2423
7.2334
7.2184
7.2041
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5.4198
5.2848
4.3224
4.3157
4.3061
4.2995
3.9429
3.9244
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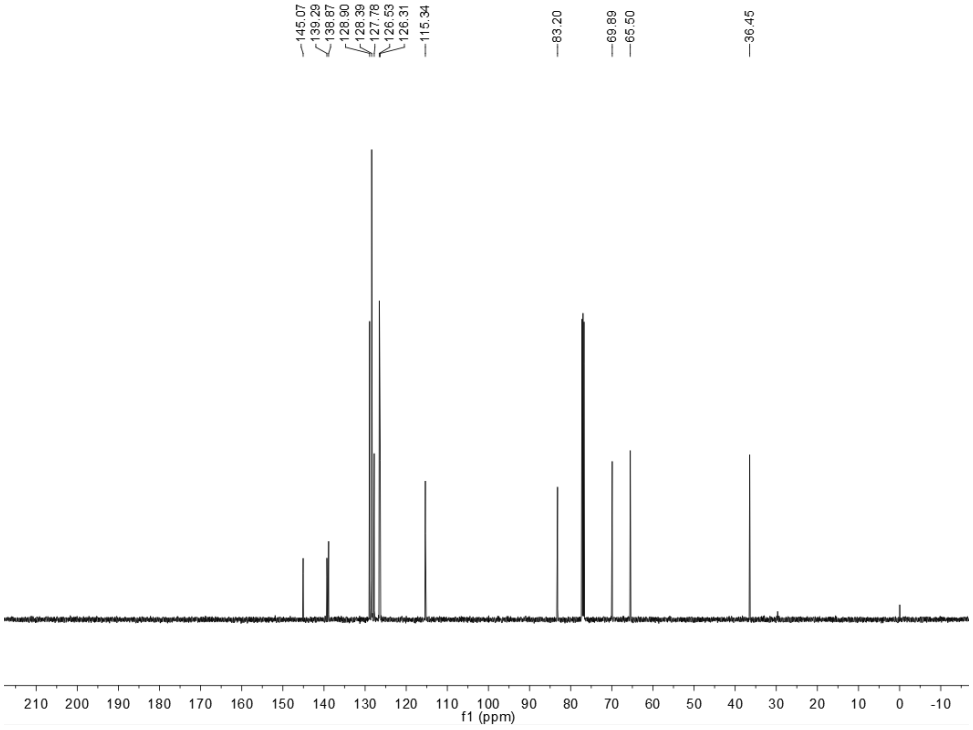
3.6369
3.6323
3.4660
2.9830
2.9692
2.9557
2.9449
2.9314
2.1058
2.0872
10.37
1.00
1.00
0.97
0.99
0.99
2.05
2.01
0.93
f1 (ppm)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139.29
138.87
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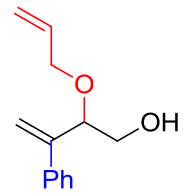
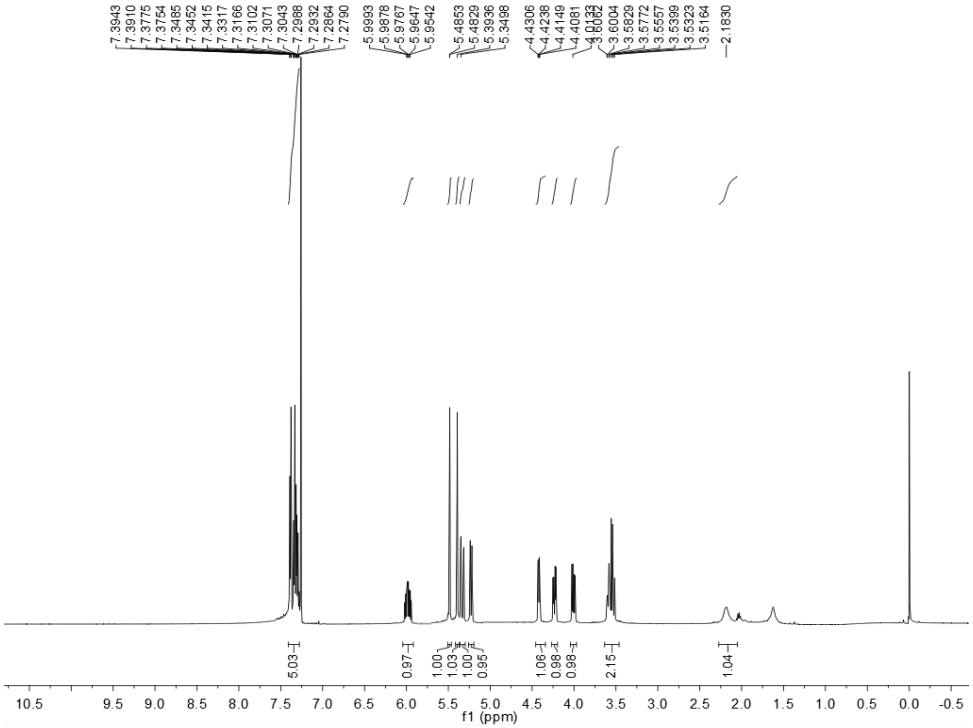
128.39
127.78
126.53
126.31
115.34
83.20
69.89
65.50
36.45
f1 (ppm)


O
OH
Ph

4ai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7.3071
7.3043
7.2988
7.2932
7.2864
7.2790
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5.9767
5.9647
5.9542
5.4853
5.4829
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4.4306
4.4238
4.4149
4.4081
3.6004
3.5929
3.5772
3.5557
3.5399
3.5323
3.5164
2.1830
5.03
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1.03
1.00
0.95
1.06
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0.98
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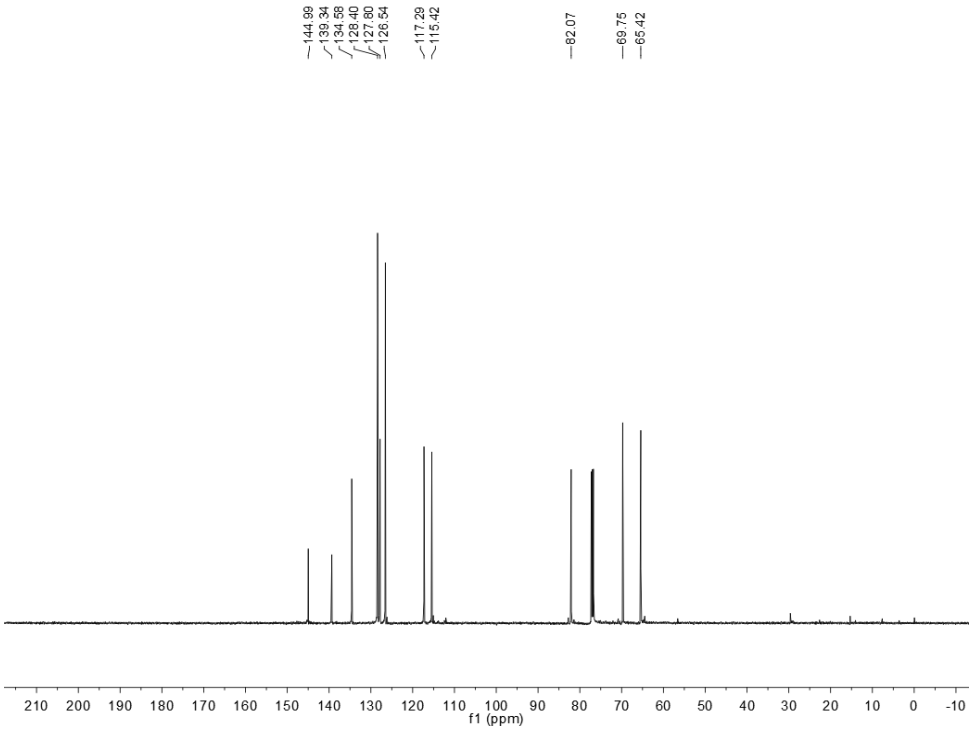
1.04
f1 (ppm)


144.99
139.34
134.58
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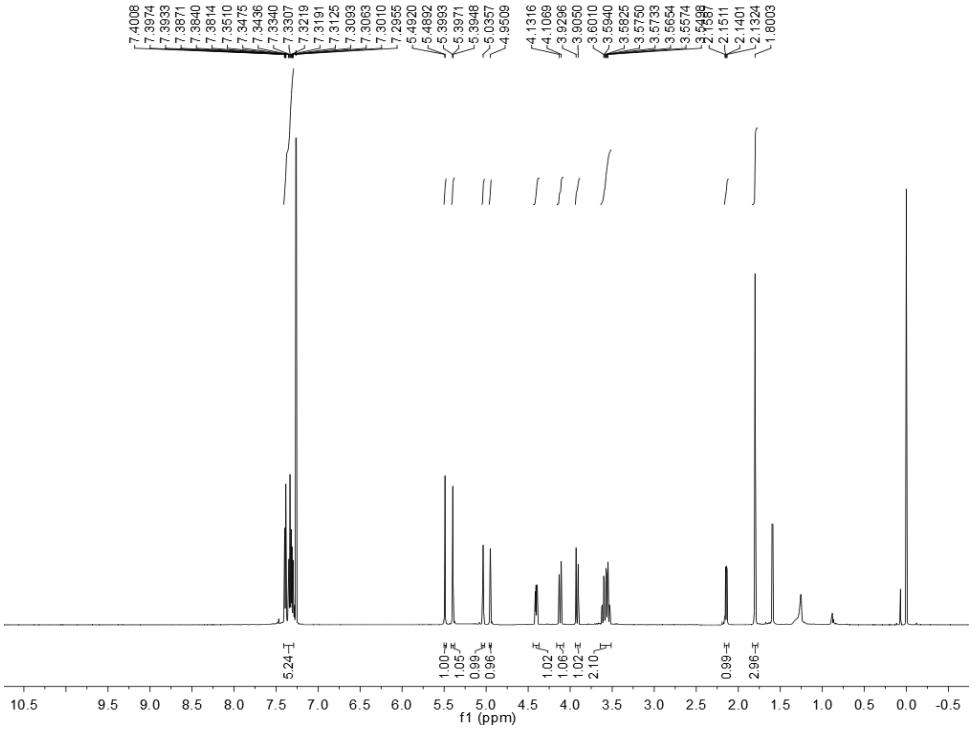
117.29
115.42
82.07
69.75
65.42
f1 (ppm)


O
OH
Ph

4aj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7.3307
7.3219
7.3191
7.3125
7.3093
7.3063
7.3010
7.2955
5.4920
5.4892
5.3993
5.3971
5.3948
5.0357
4.9509
4.1316
4.1069
3.9296
3.9050
3.6010
3.5940
3.5825
3.5750
3.5733
3.5654
3.5574
2.1511
2.1401
2.1324
1.8003
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0.99
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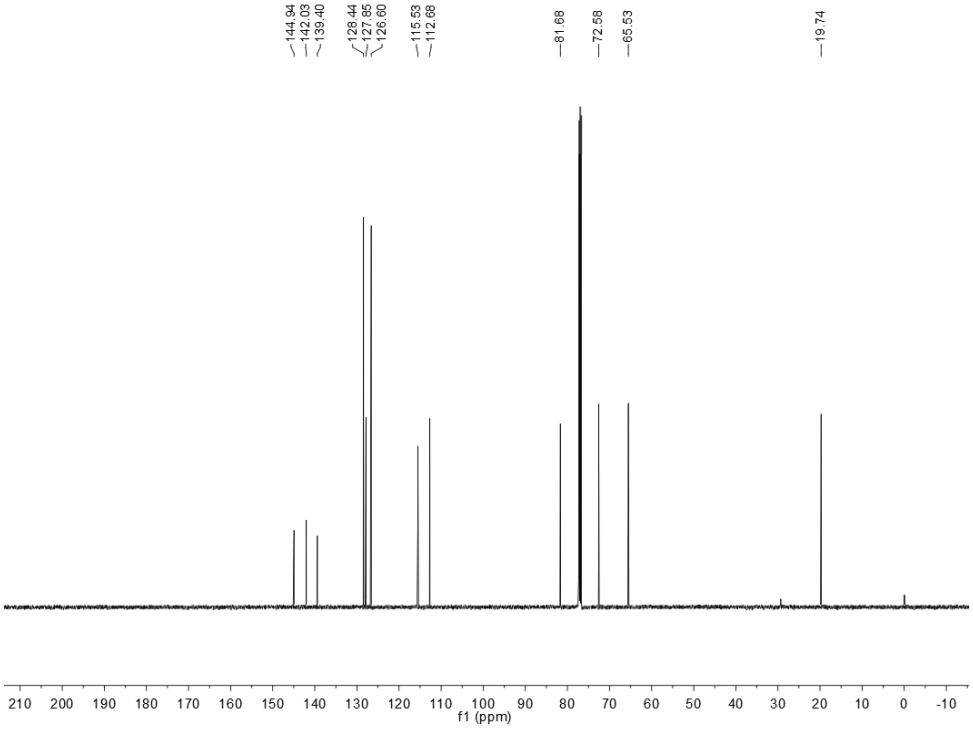
1.02
1.06
1.02
2.10
0.99
2.96
f1 (ppm)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139.40
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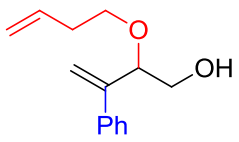
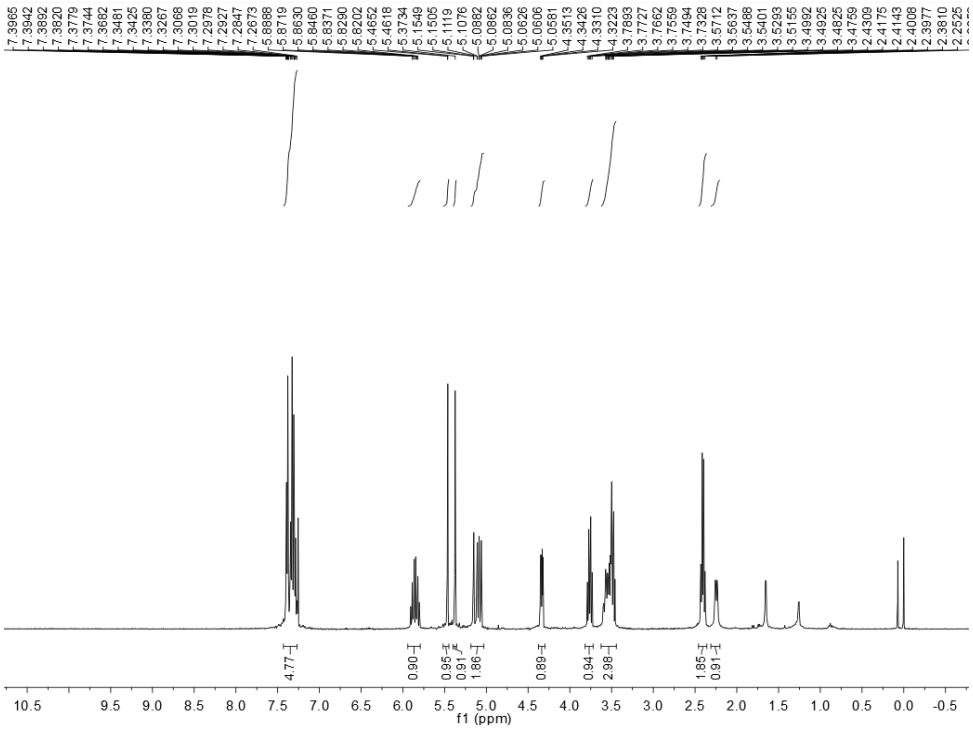
127.85
126.60
115.53
112.68
81.68
72.58
65.53
19.74
f1 (ppm)


O
OH
Ph

4ak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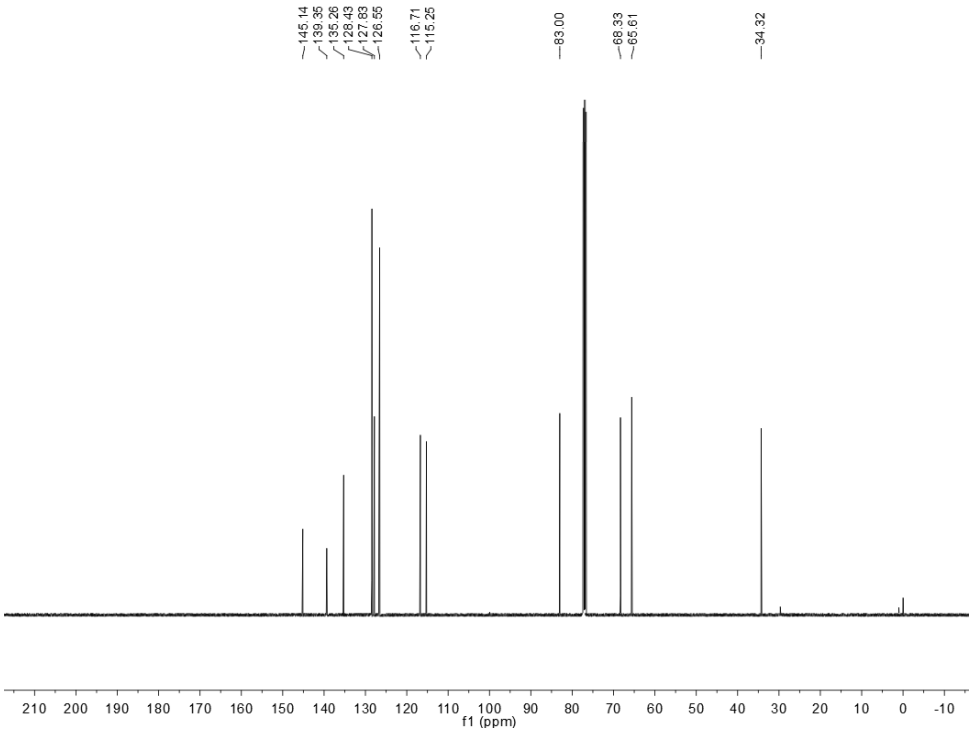
0.89
0.94
2.98
1.85
0.91
f1 (ppm)


145.14
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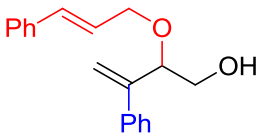
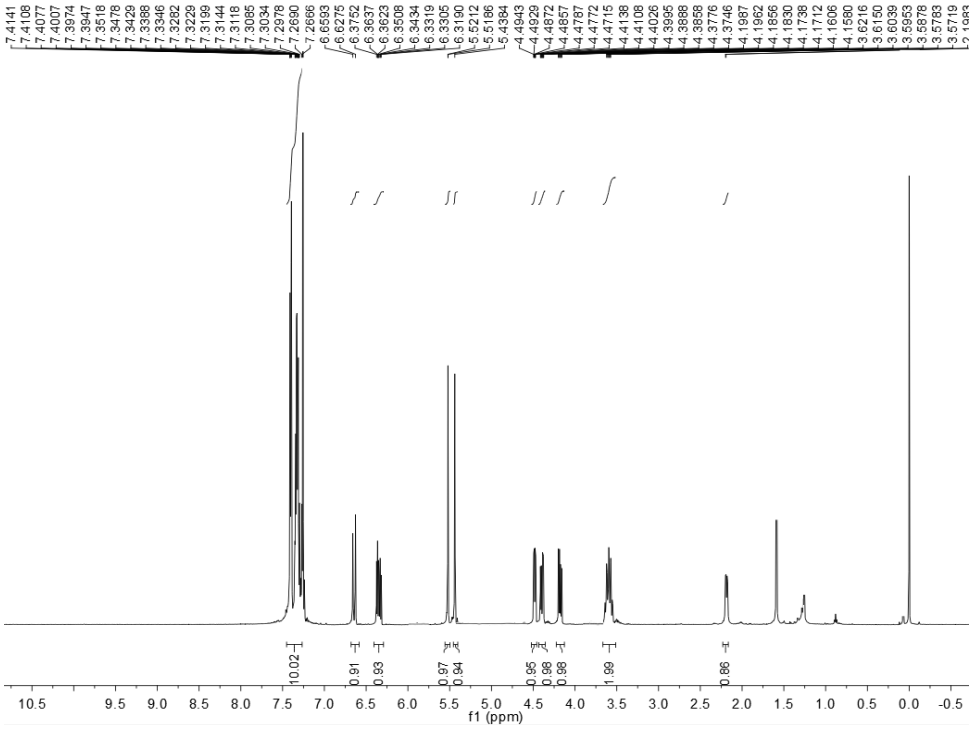
135.26
128.43
127.83
126.55
116.71
115.25
83.00
68.33
65.61
34.32
f1 (ppm)


Ph
O
OH
Ph

4al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0.97
0.94
0.95
0.98
0.98
1.99
0.86
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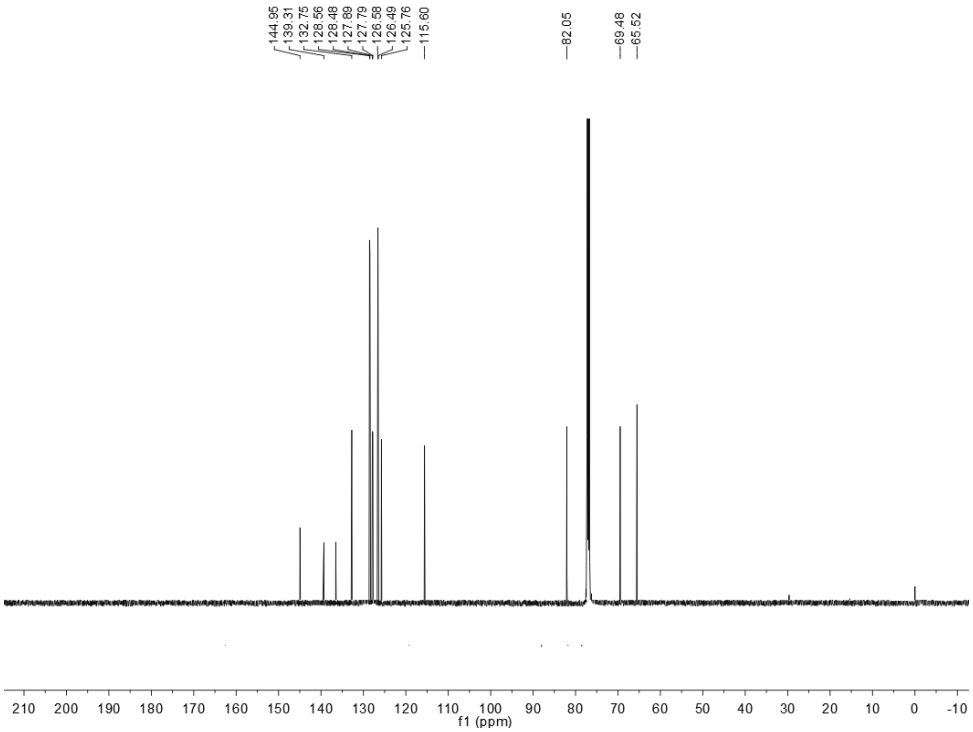
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132.75
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128.48
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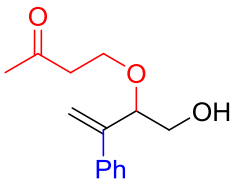
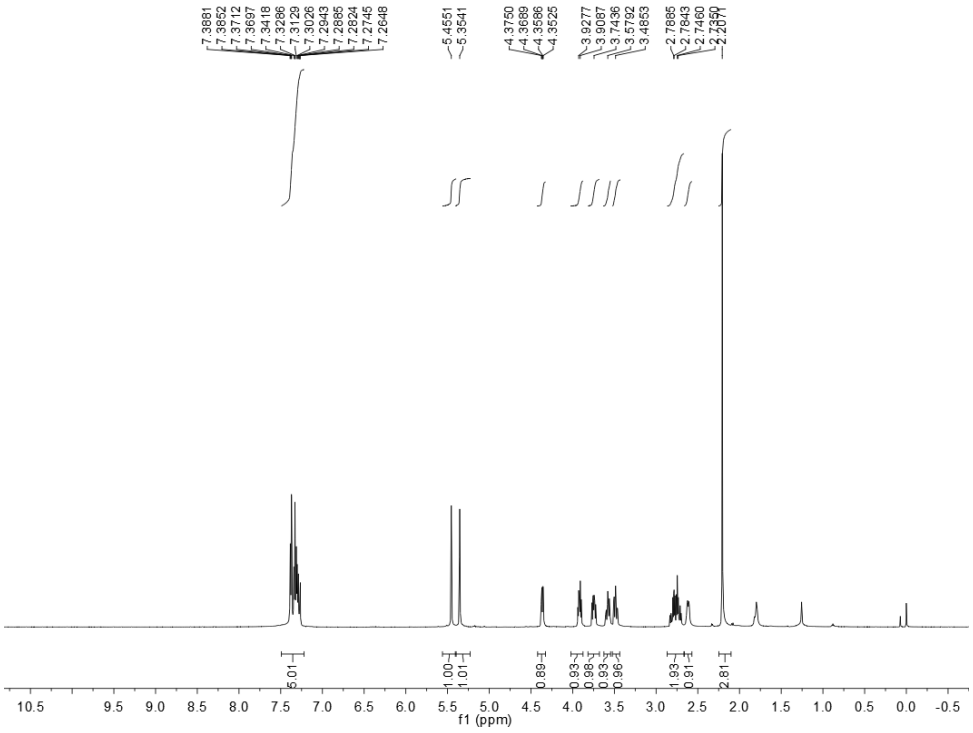
127.79
126.68
126.49
125.76
115.60
82.05
69.48
65.52
f1 (ppm)


O
O
OH
Ph

4am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7.2885
7.2824
7.2745
7.2648
5.4551
5.3541
4.3750
4.3689
4.3586
4.3525
3.9277
3.9087
3.7496
3.5792
3.4853
2.7885
2.7643
2.7460
2.2570
2.2370
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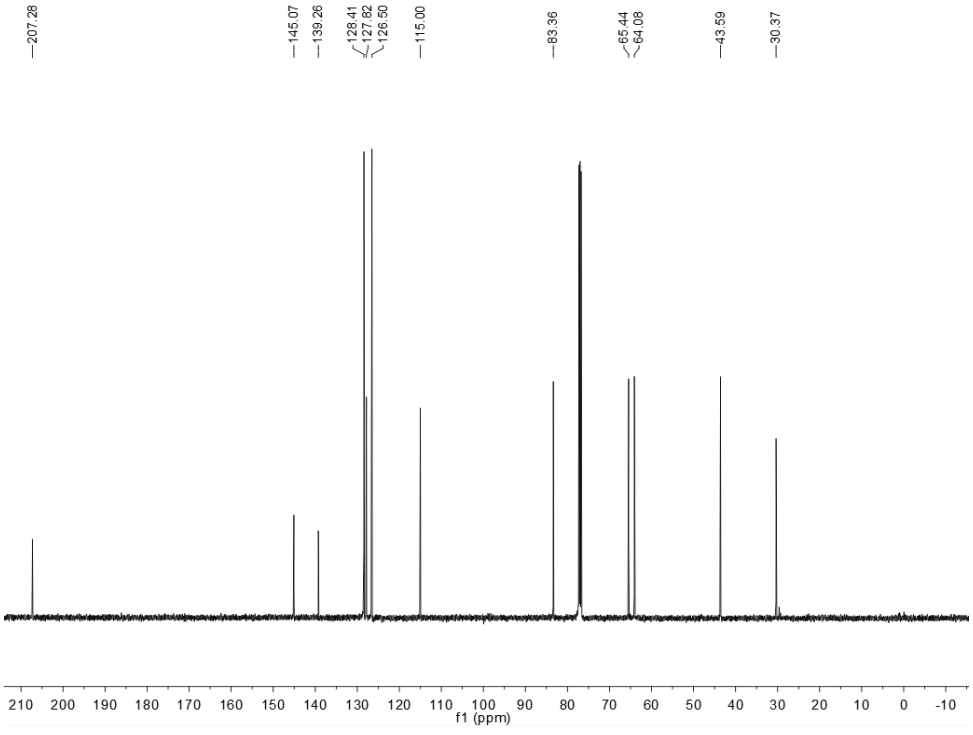
1.00
1.01
0.89
0.93
0.98
0.93
0.96
1.93
0.91
2.81
f1 (ppm)


207.28
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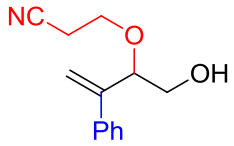
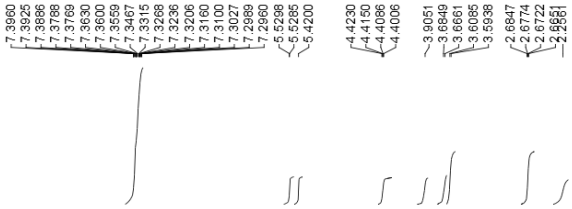
126.50
115.00
83.36
65.44
64.08
43.59
30.37
f1 (ppm)


NC
O
OH
Ph

4an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7.3236
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7.3160
7.3100
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7.2989
7.2960
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5.5285
5.4200
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4.4150
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4.4006
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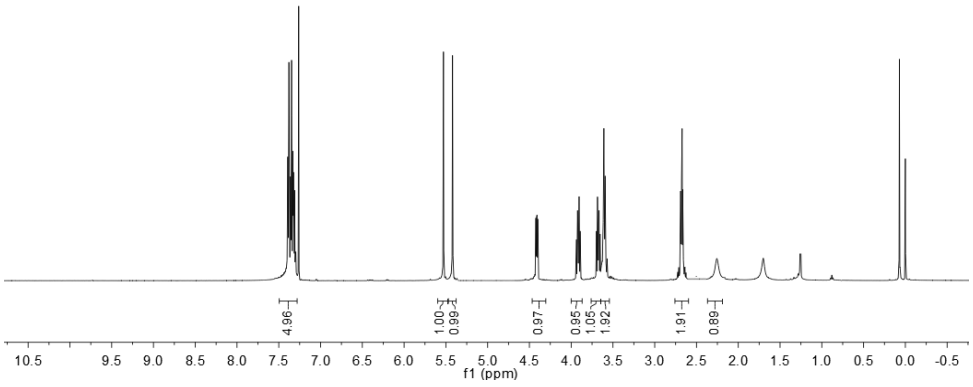
2.6774
2.6722
2.6551
2.6361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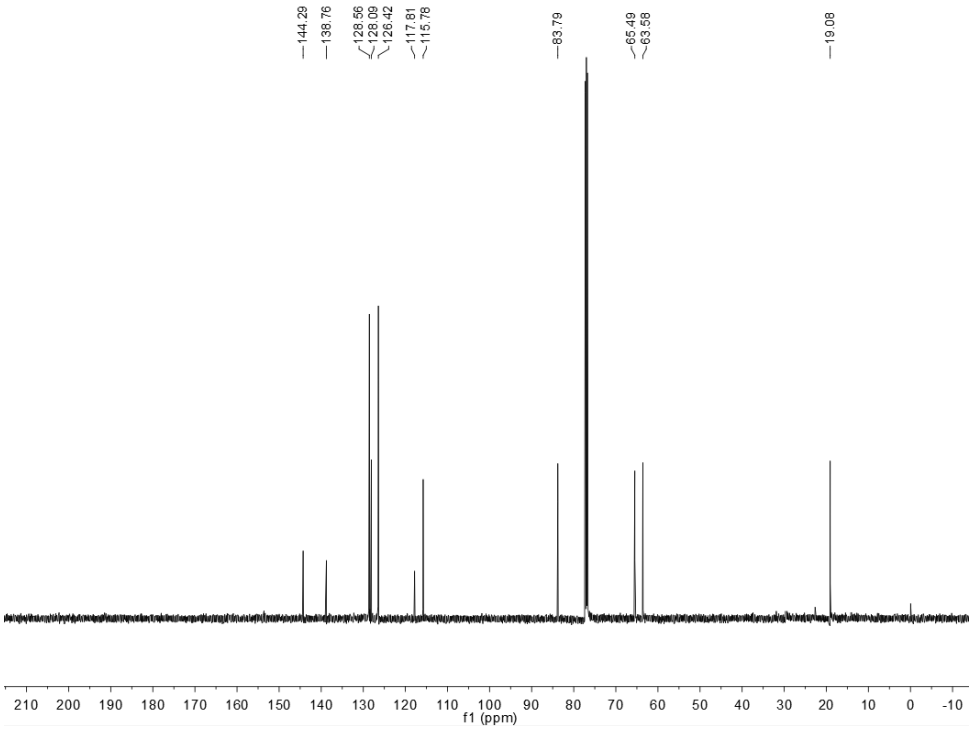
0.99
0.97
0.95
1.05
1.92
1.91
0.89
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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128.09
126.42
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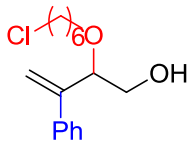
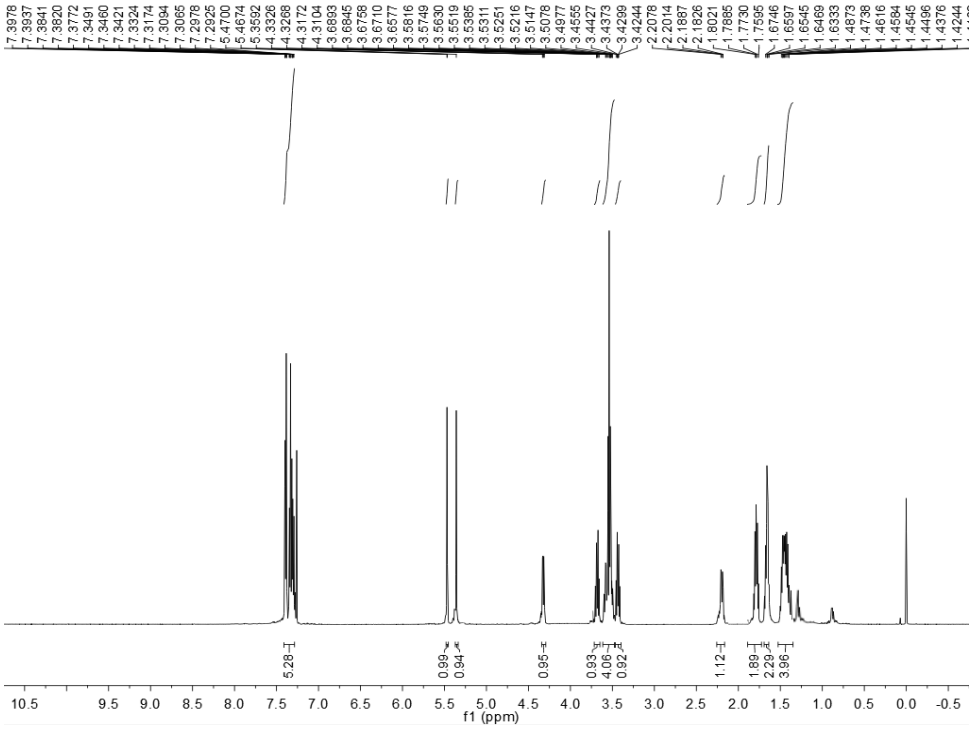
65.49
63.58
19.08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Cl
6
O
OH
Ph

4ao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7.3065
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0.95
0.93
4.06
0.92
1.12
1.89
2.29
3.96
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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3.0
2.5
2.0
1.5
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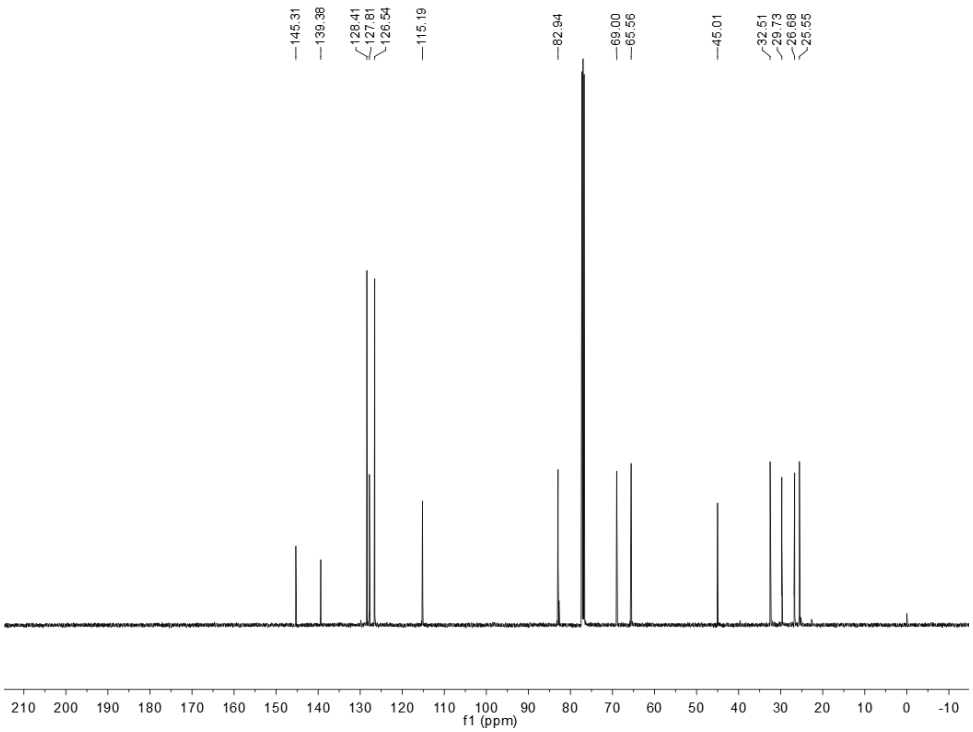
0.5
0.0
-0.5
f1 (ppm)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69.00
65.56
45.01
32.51
29.73
26.68
25.55
210
200
190
180
170
160
150
140
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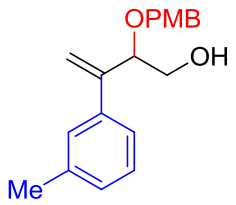
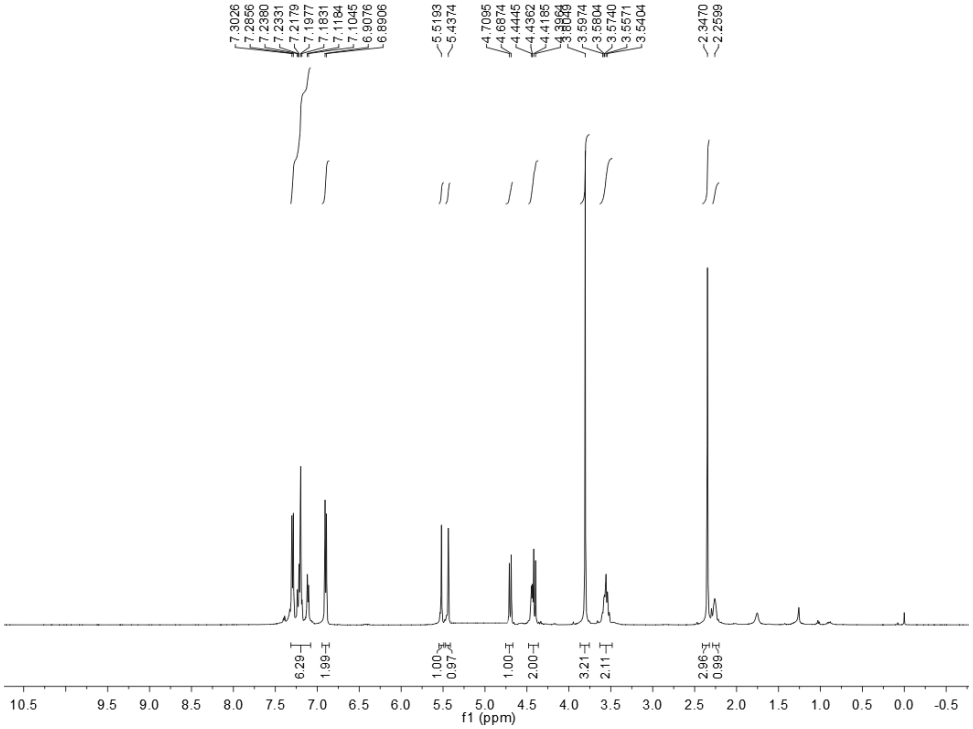
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


OPMB
OH
Me

4cb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6.8906
5.5193
5.4374
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3.5804
3.5740
3.5571
3.5404
2.3470
2.2599
6.29
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1.00
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1.00
2.00
3.21
2.11
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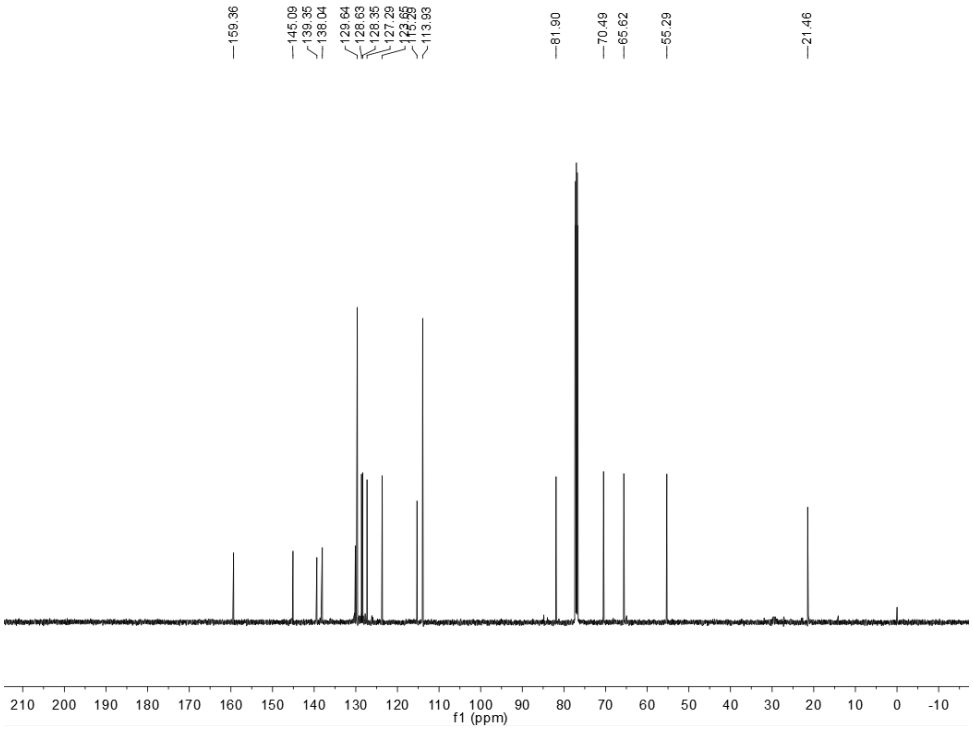
0.99
f1 (ppm)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129.64
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128.35
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123.65
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113.93
81.90
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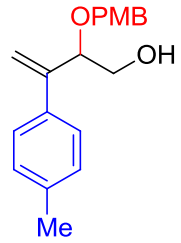
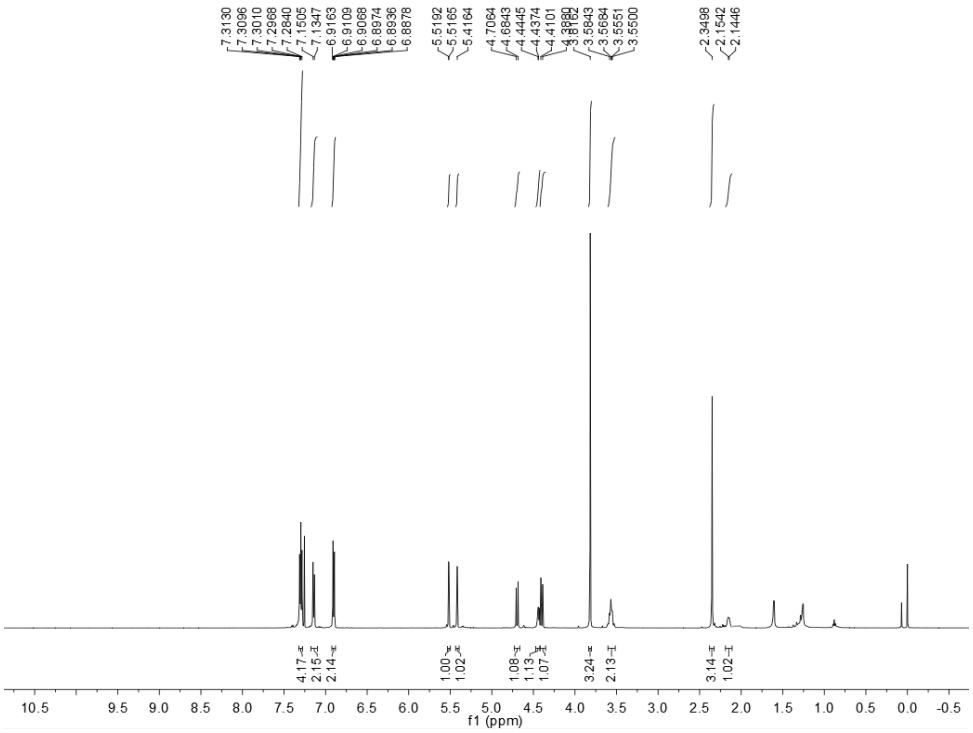
65.62
55.29
21.46
f1 (ppm)


OPMB
OH
Me

4db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4.17
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1.07
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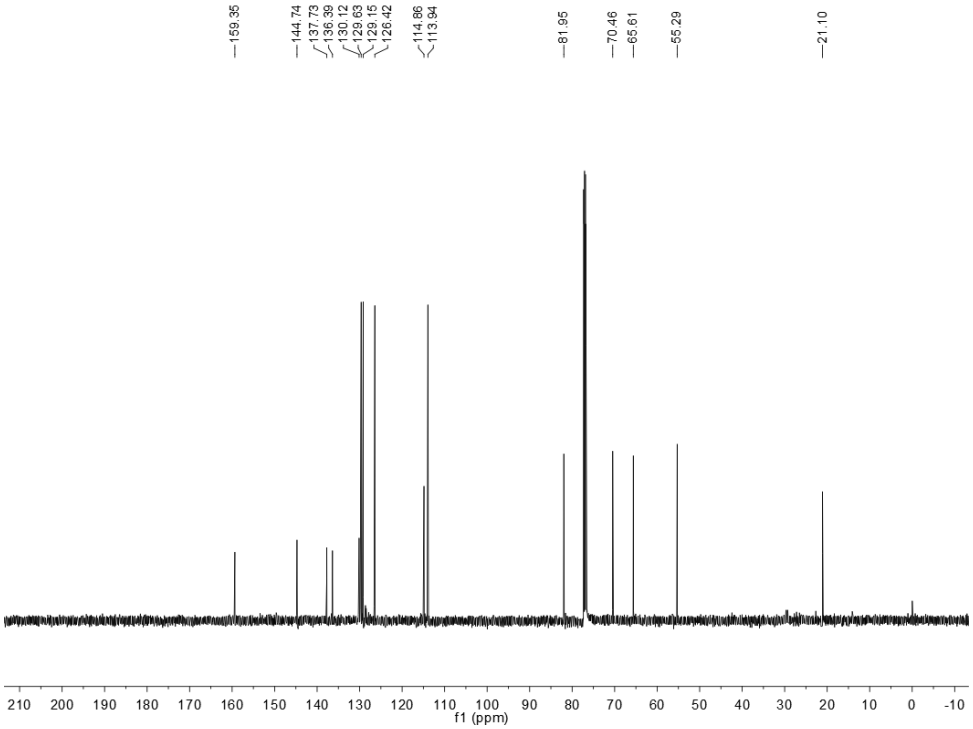
2.13
3.14
1.02
f1 (ppm)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144.74
137.73
136.39
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129.63
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113.94
81.95
70.46
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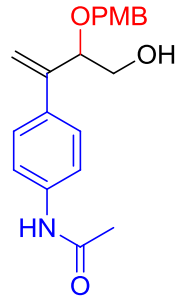
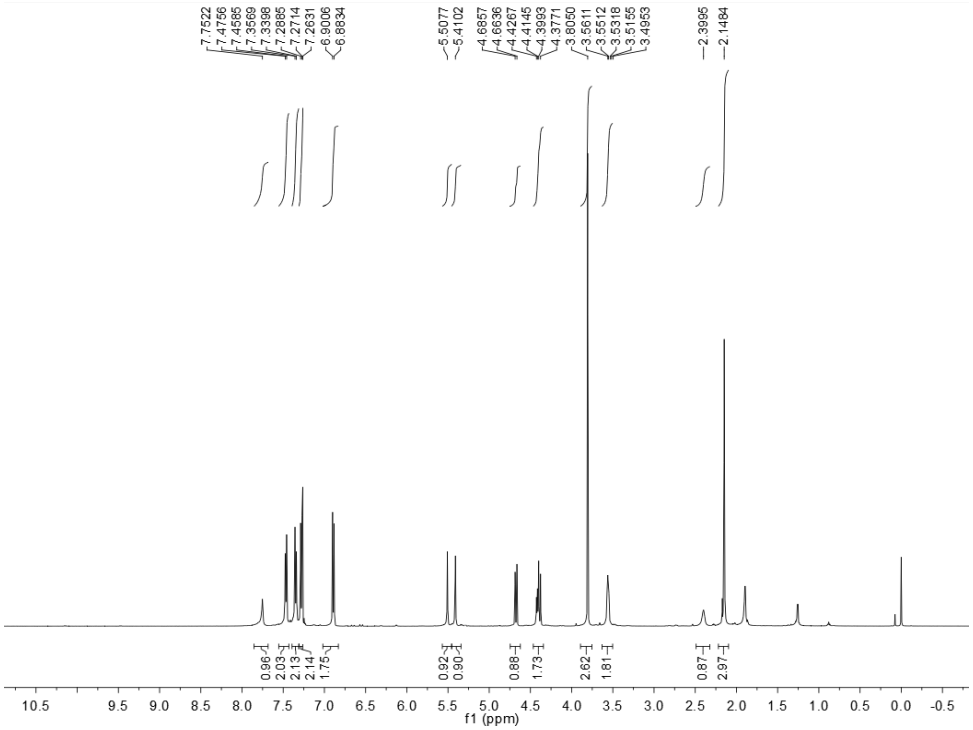
55.29
21.10
f1 (ppm)


OPMB
OH
HN
O

4eb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2.1484
0.96
2.03
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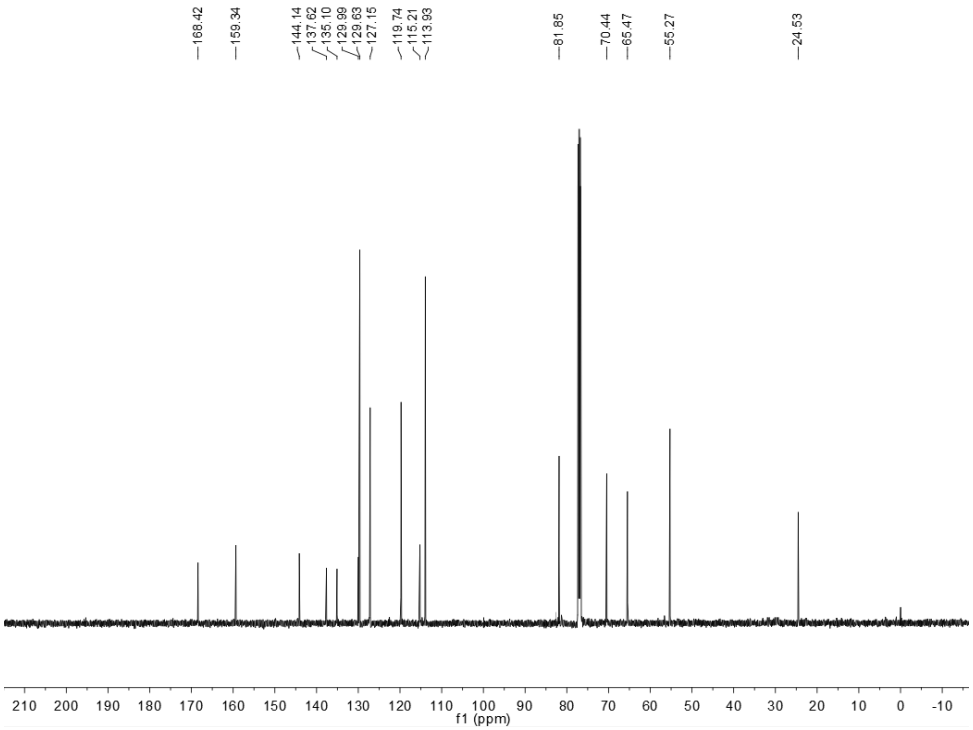
f1 (ppm)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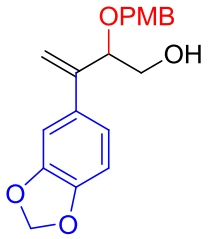
24.53
f1 (ppm)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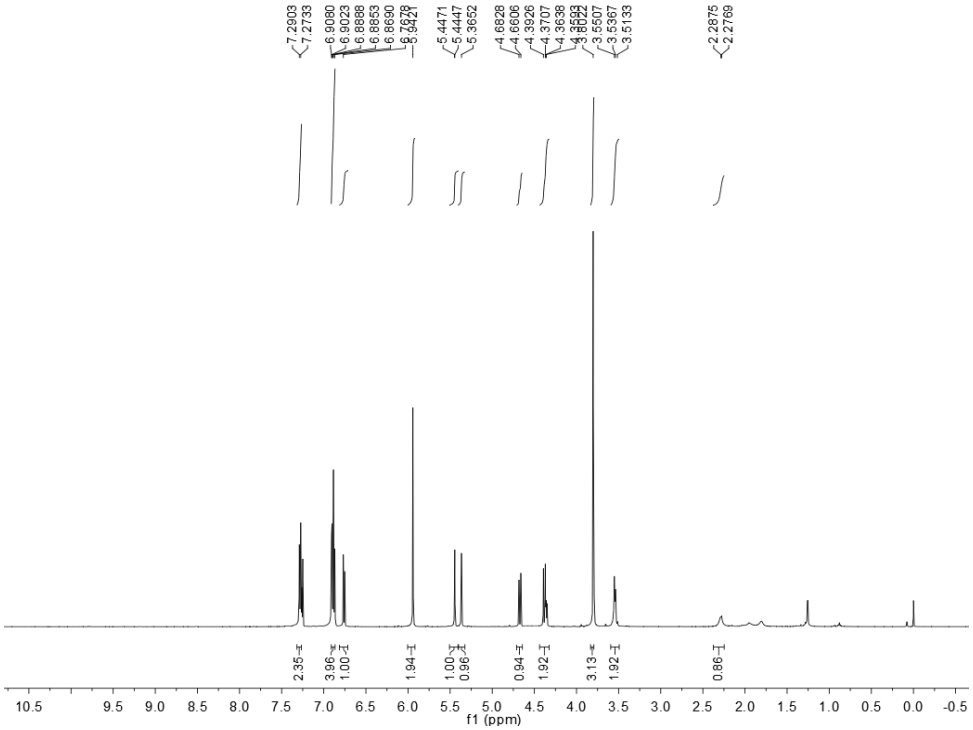
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3.5507
3.5367
3.5133
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2.2769
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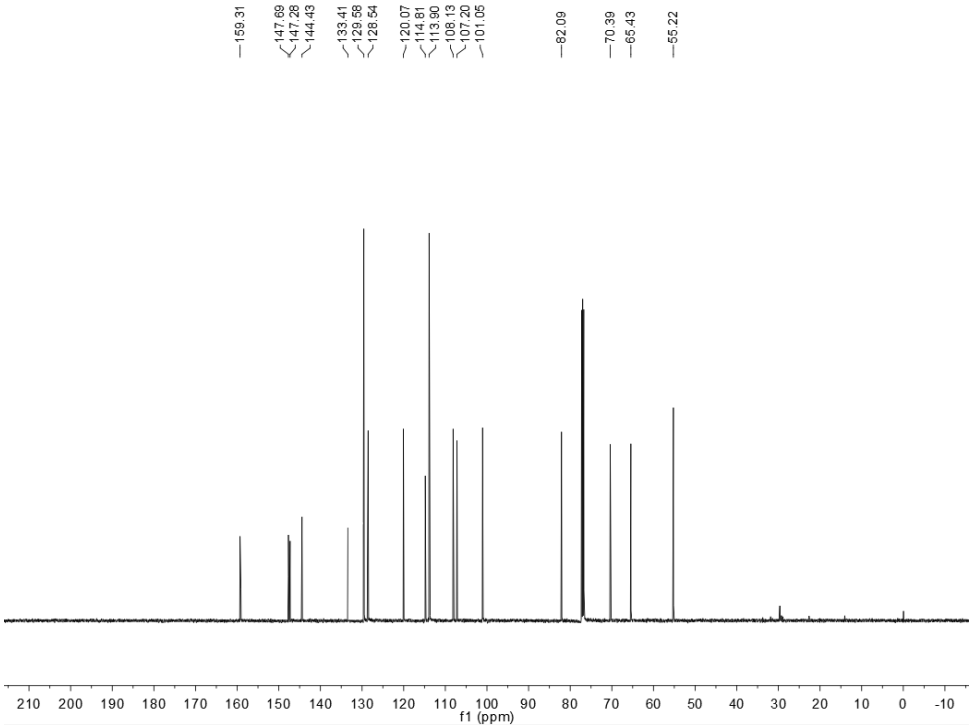
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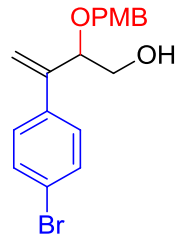
55.22
f1 (ppm)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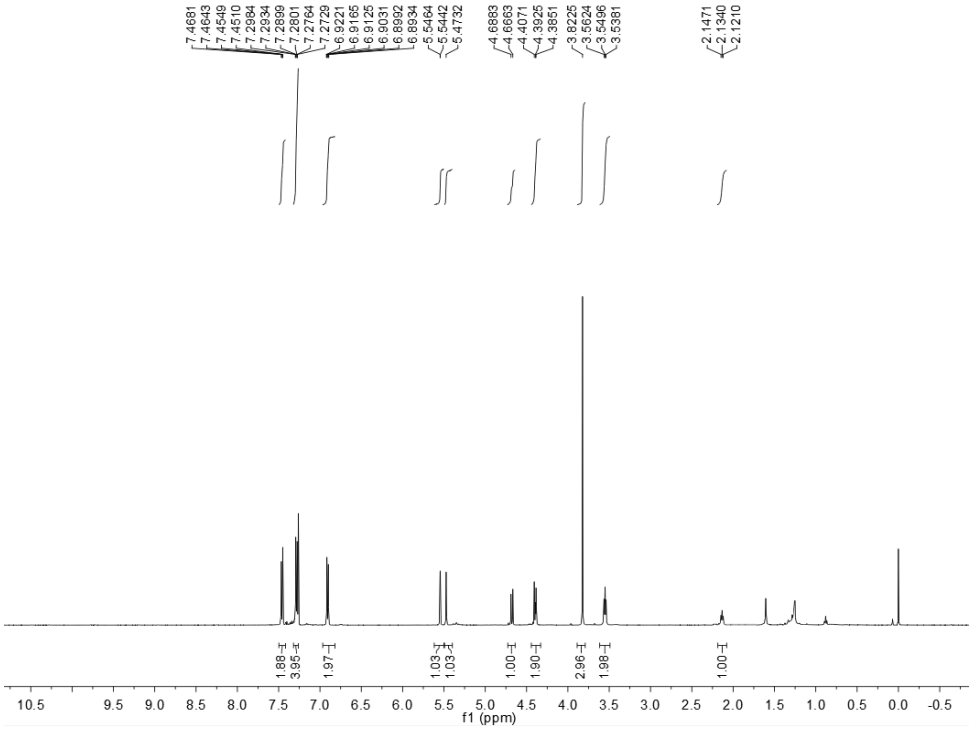
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2.1340
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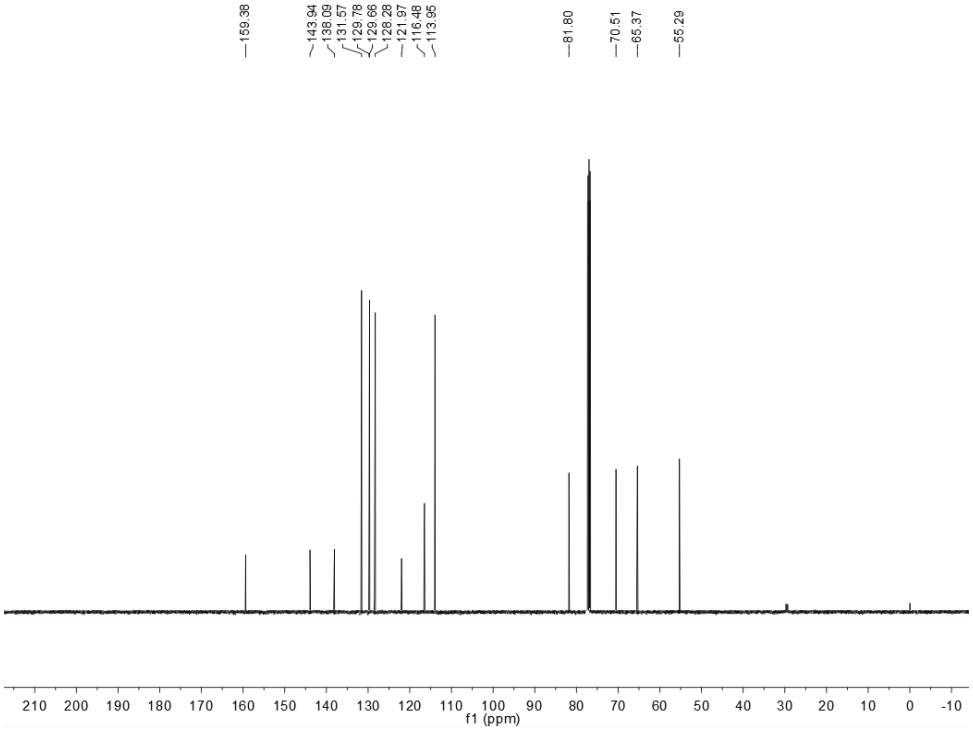
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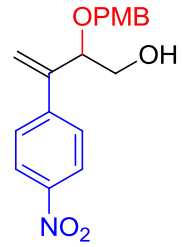
55.29
f1 (ppm)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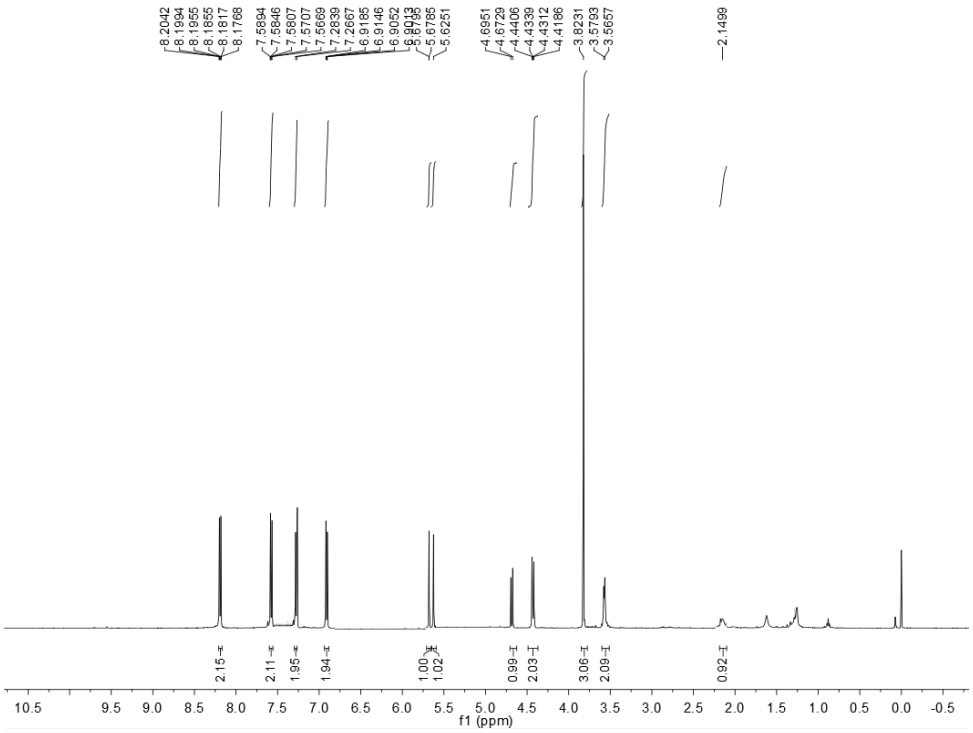
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6.9146
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5.6785
5.6251
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4.4312
4.4186
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3.5657
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2.03
3.06
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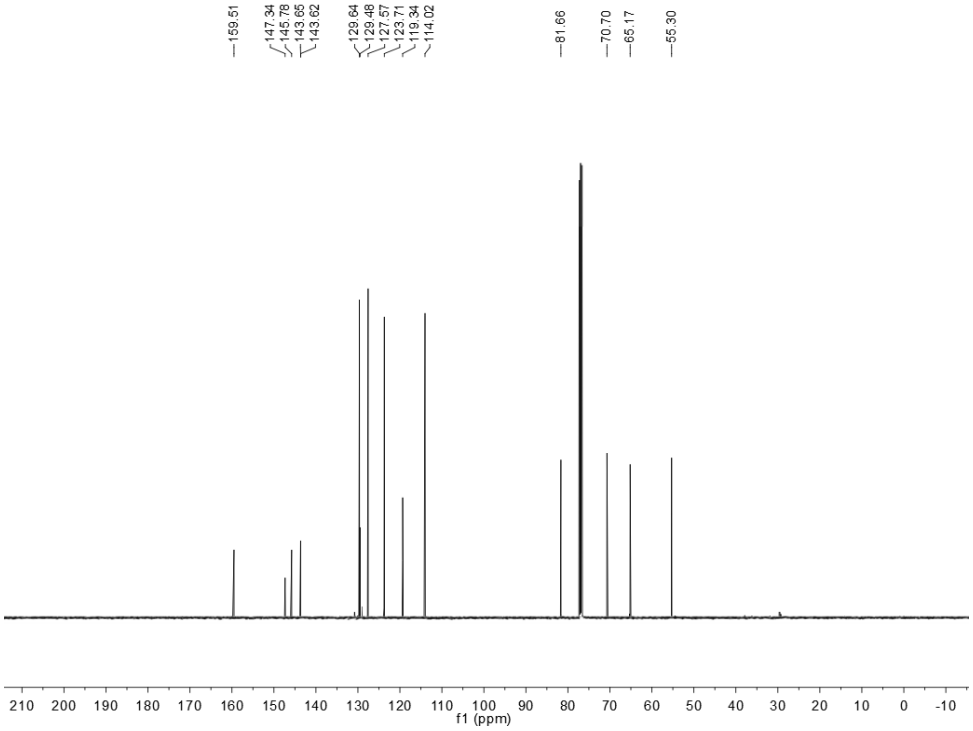
0.92
f1 (ppm)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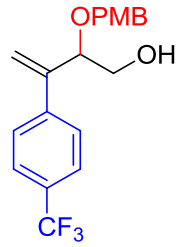
55.30
f1 (ppm)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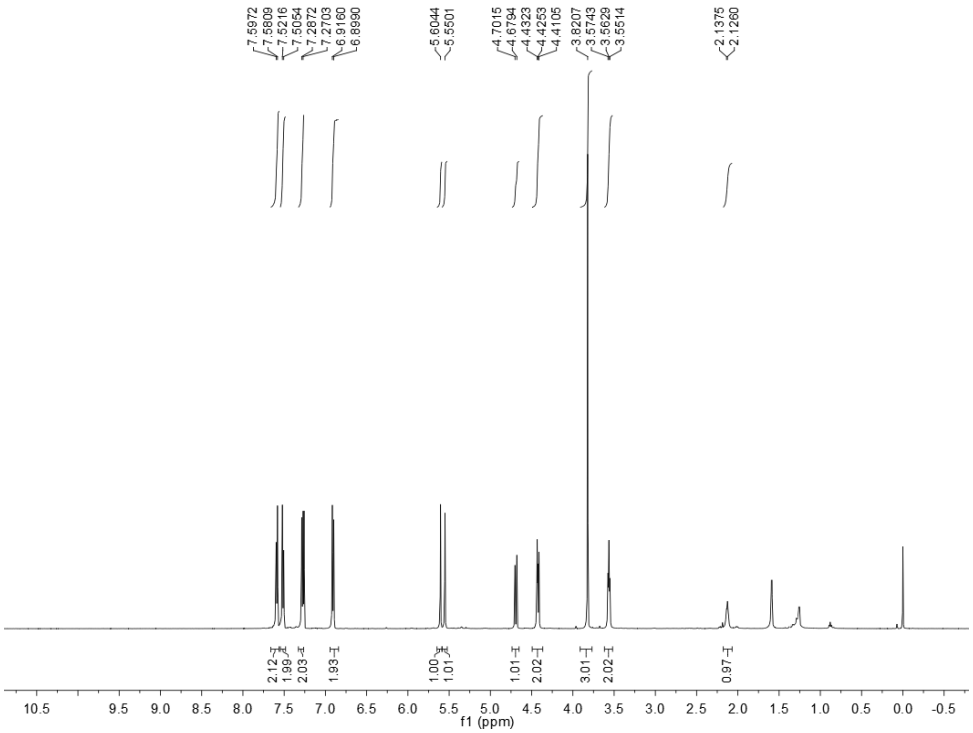
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3.5514
2.1375
2.1260
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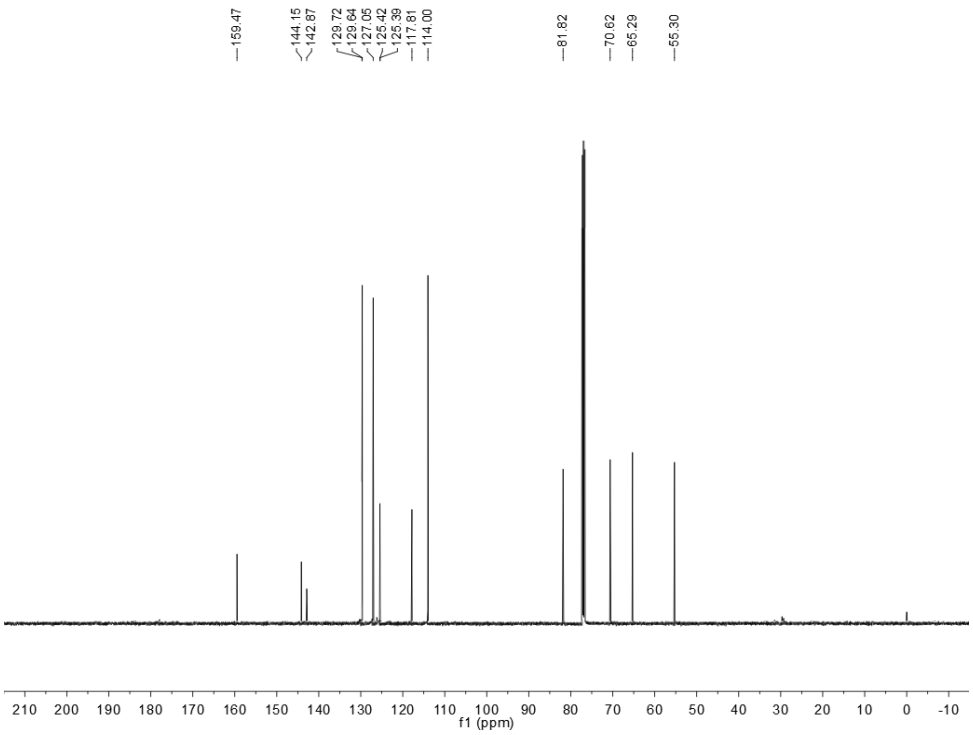
1.01
2.02
3.01
2.02
0.97
f1 (ppm)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114.00
81.82
70.62
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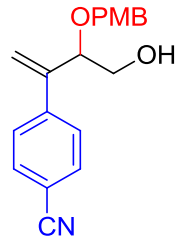
55.30
f1 (ppm)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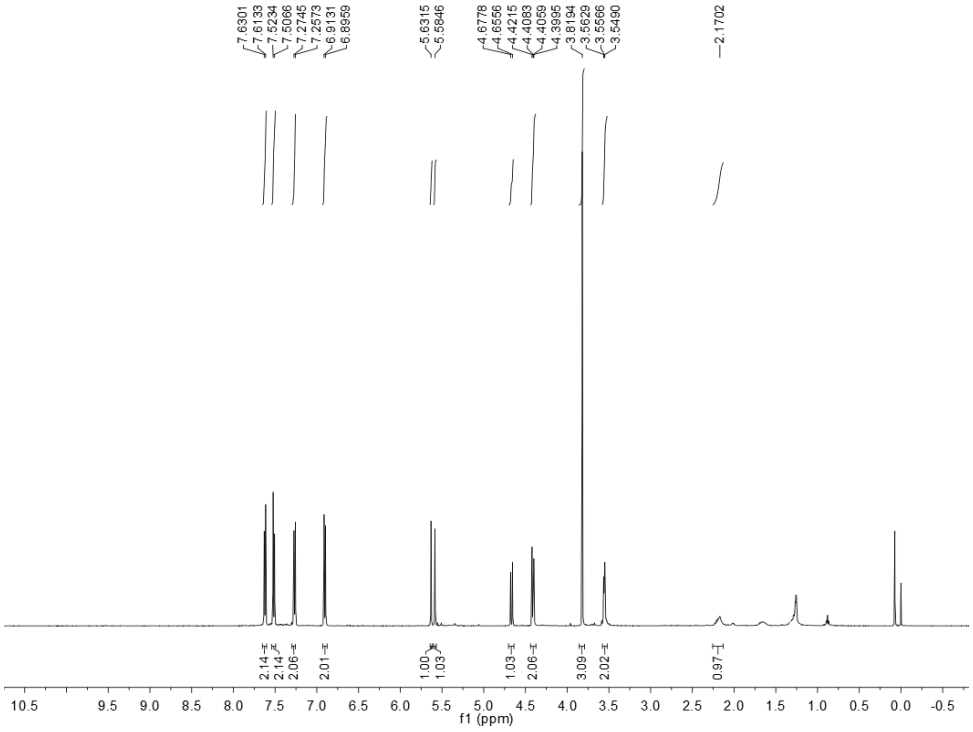
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2.1702
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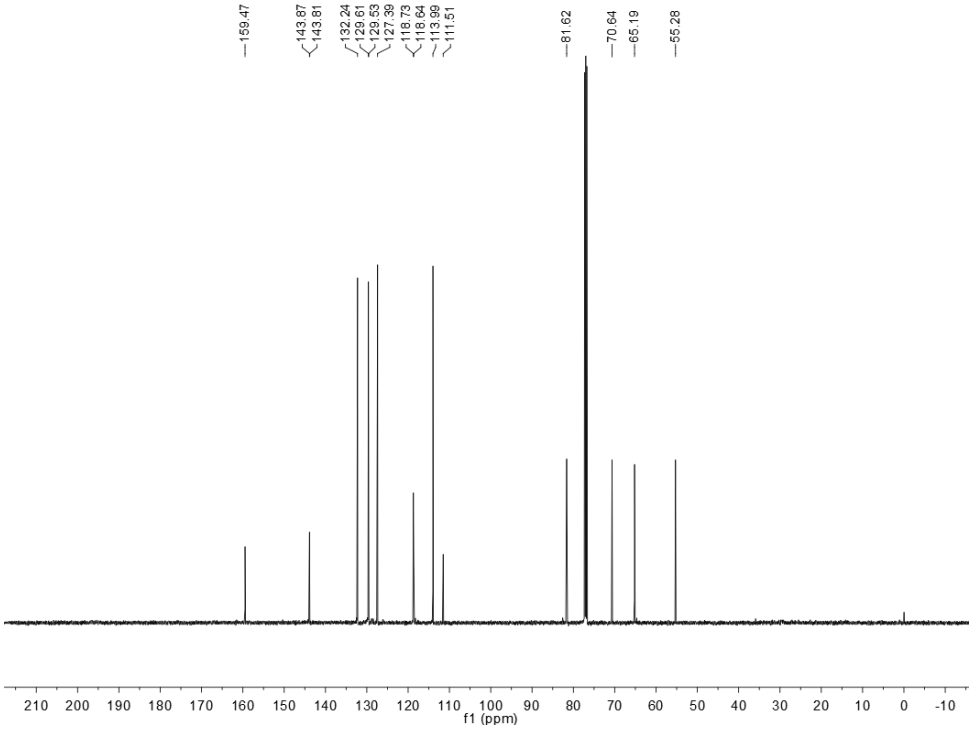
1.03
1.03
2.06
3.09
2.02
0.97
f1 (ppm)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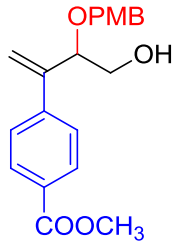
f1 (ppm)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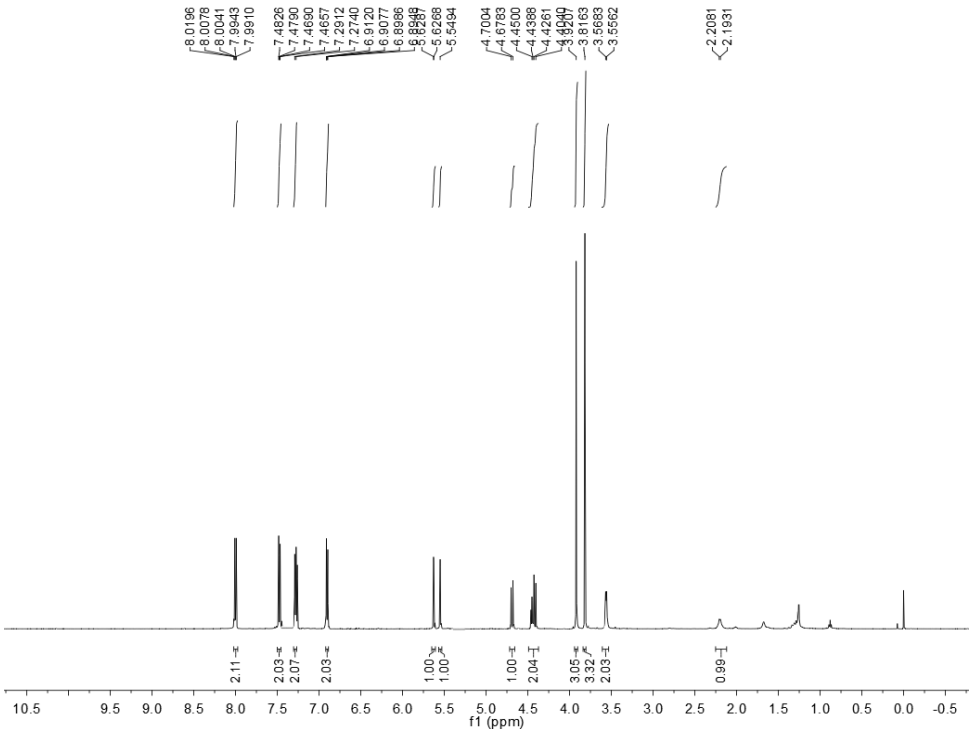
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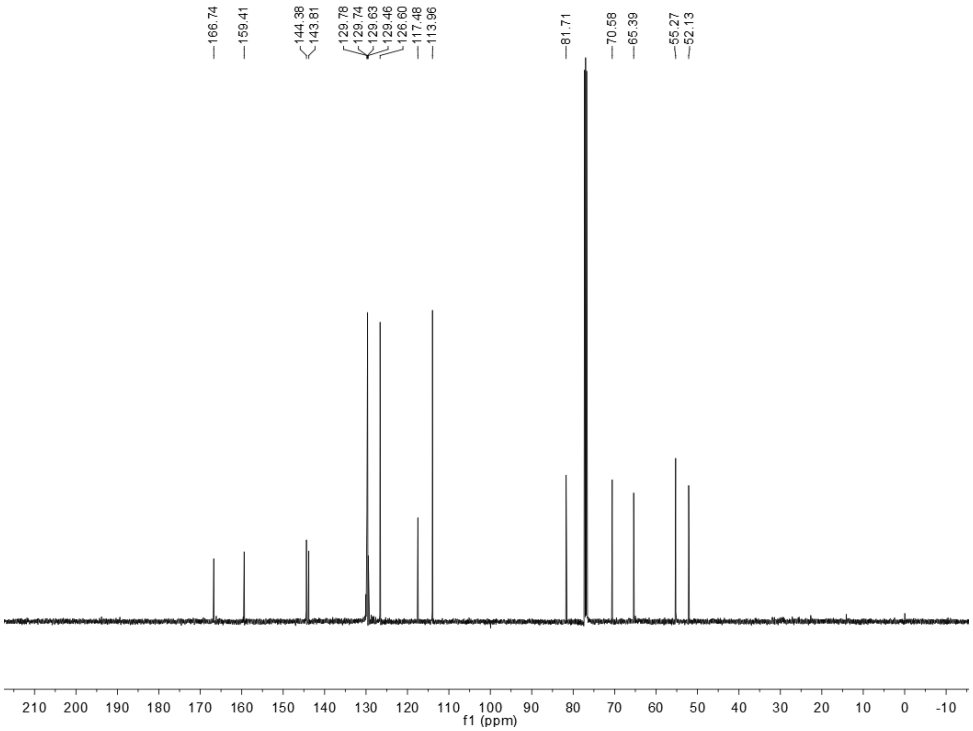
0.99
f1 (ppm)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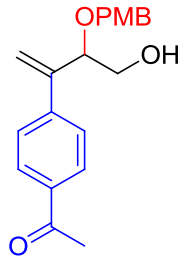
f1 (ppm)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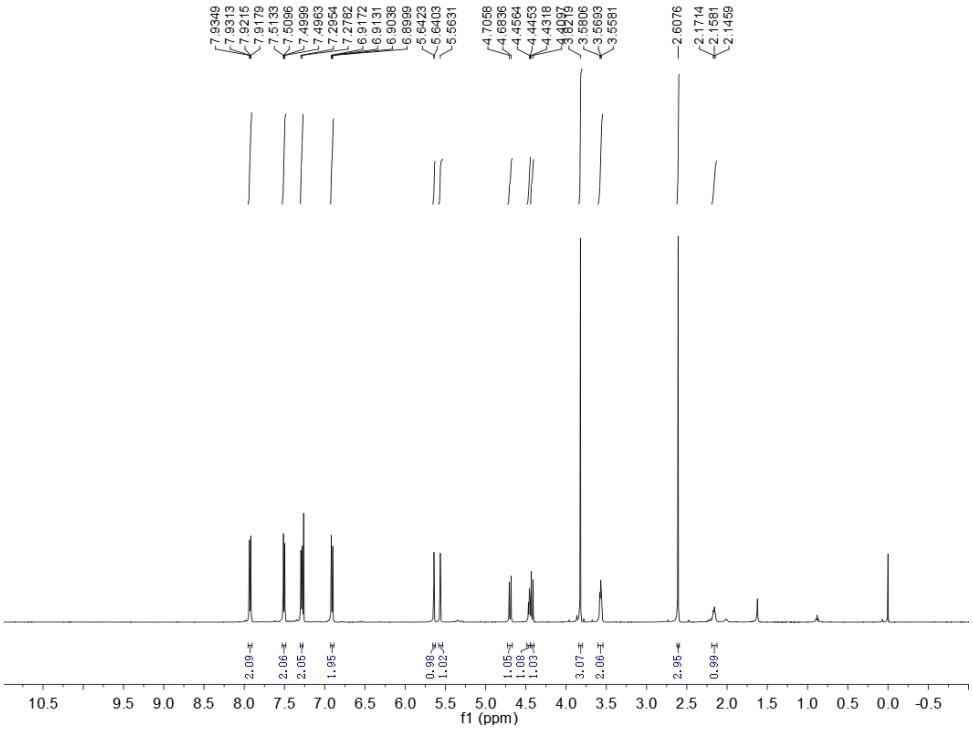
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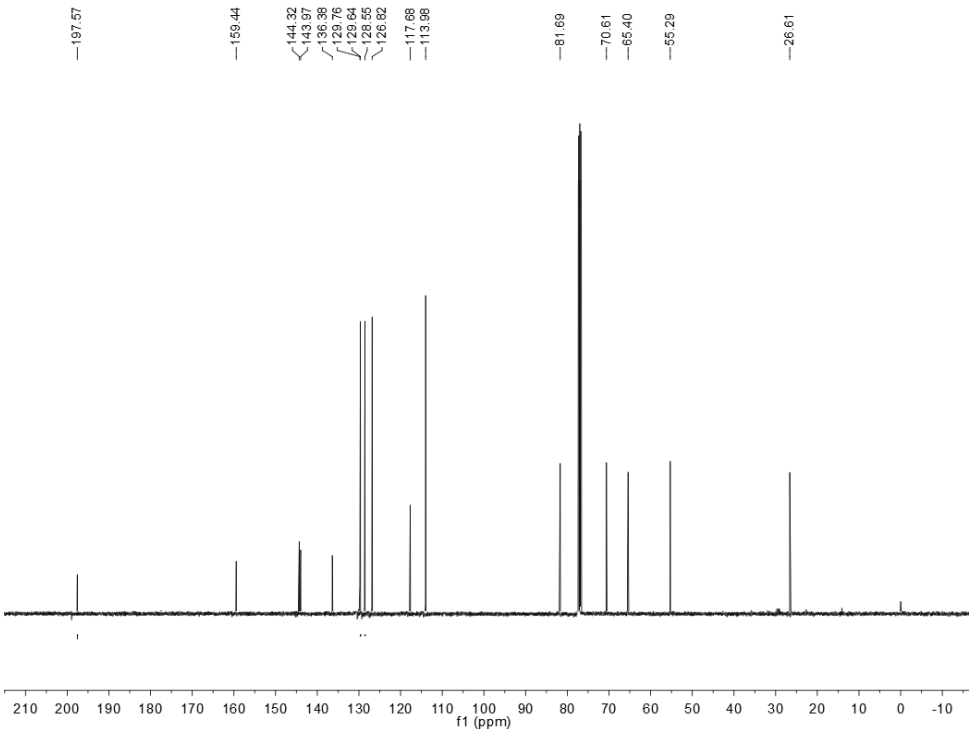
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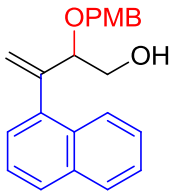
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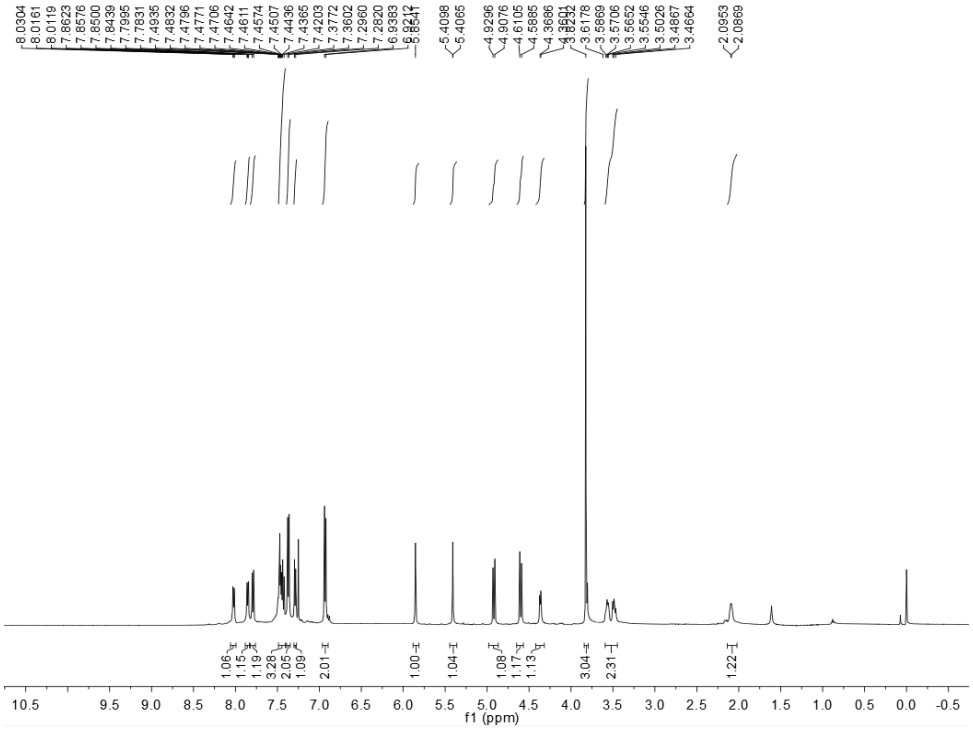
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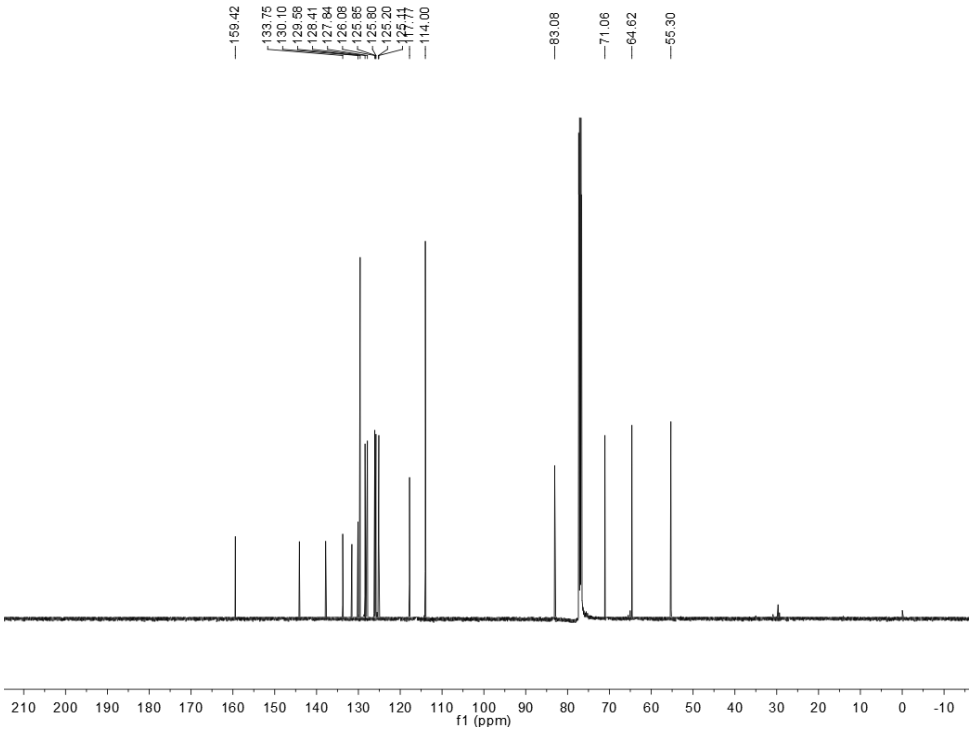
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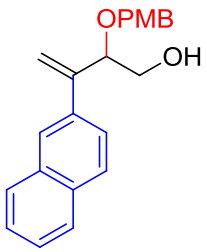
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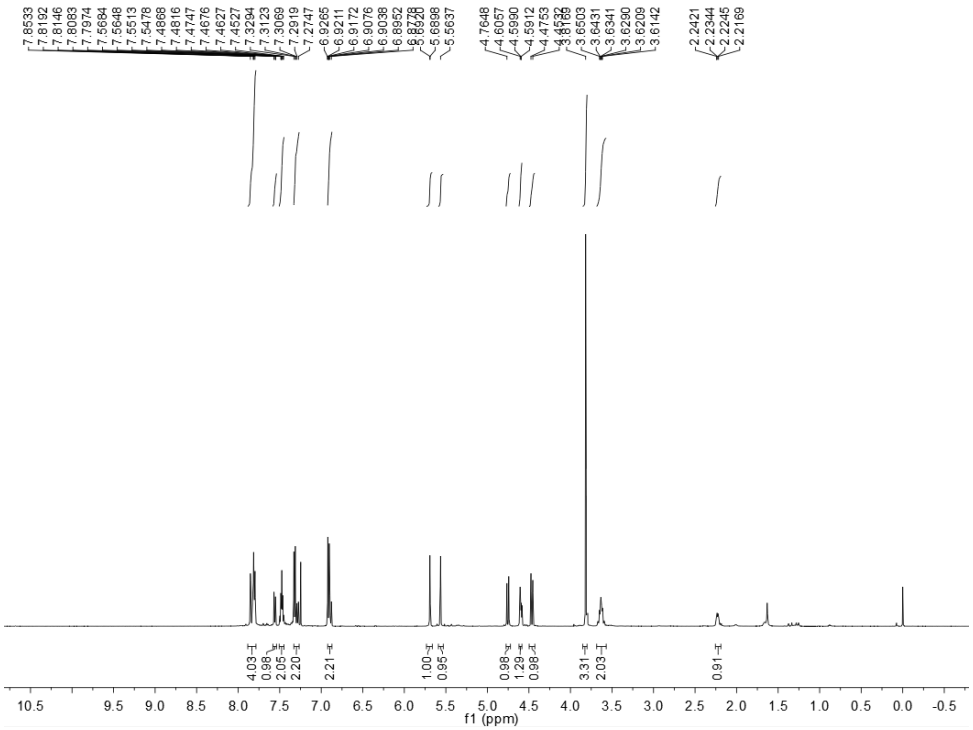
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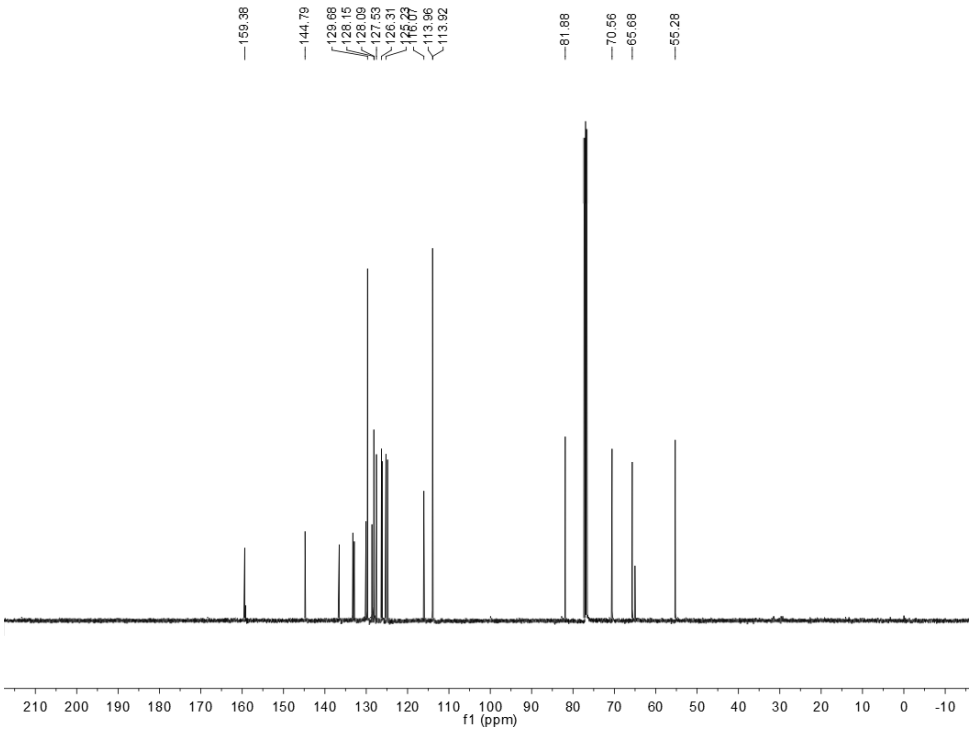
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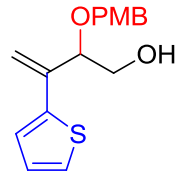
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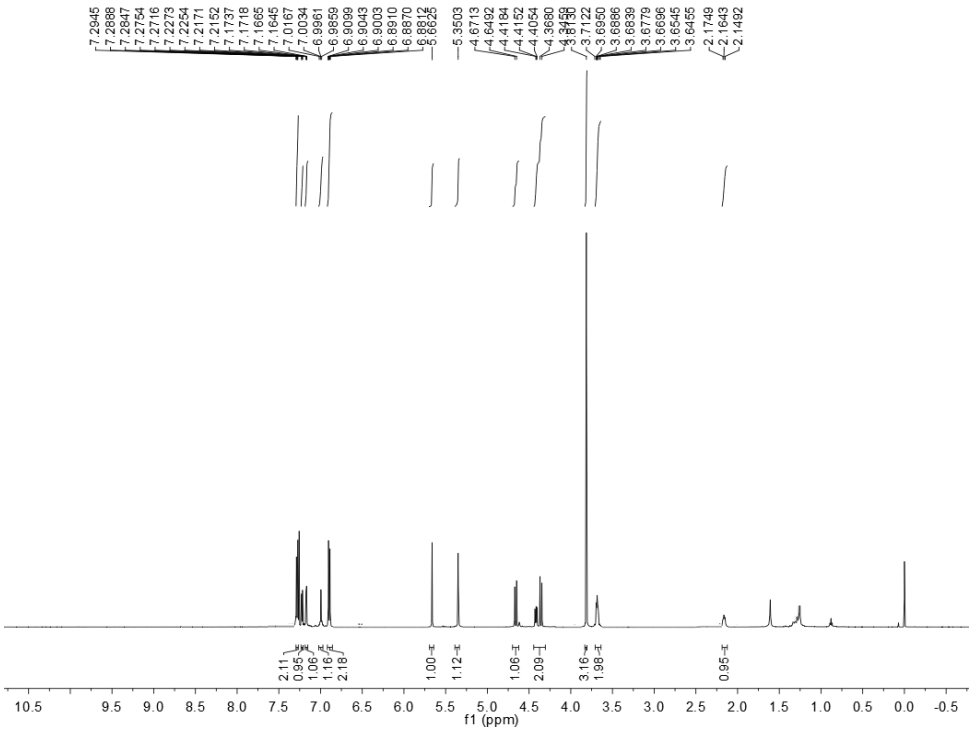
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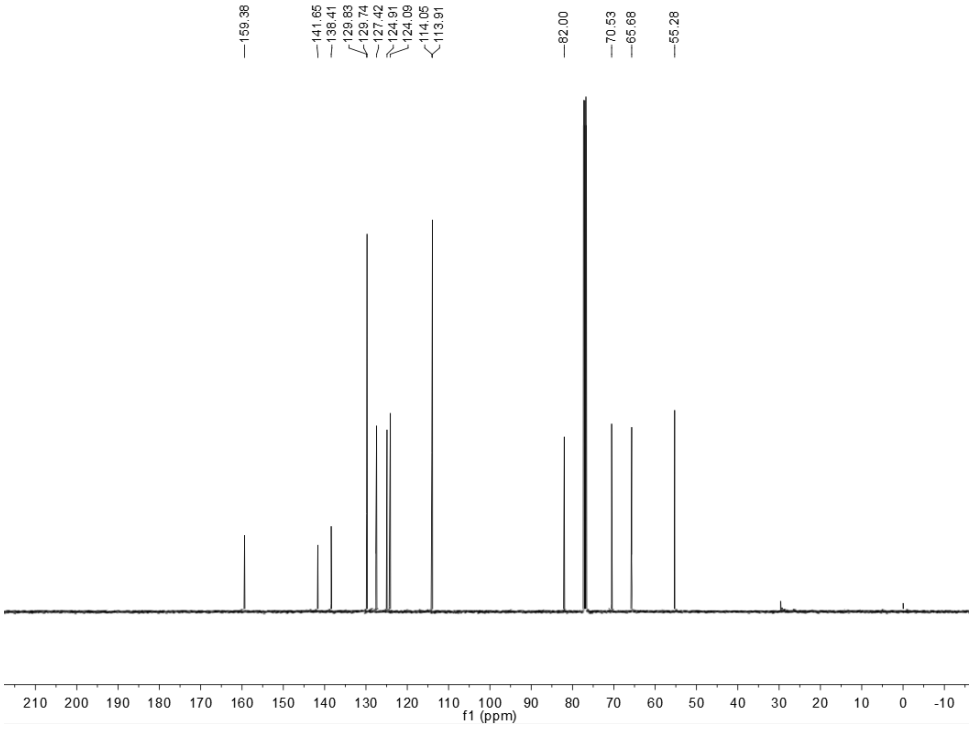
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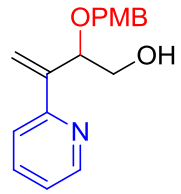
f1 (ppm)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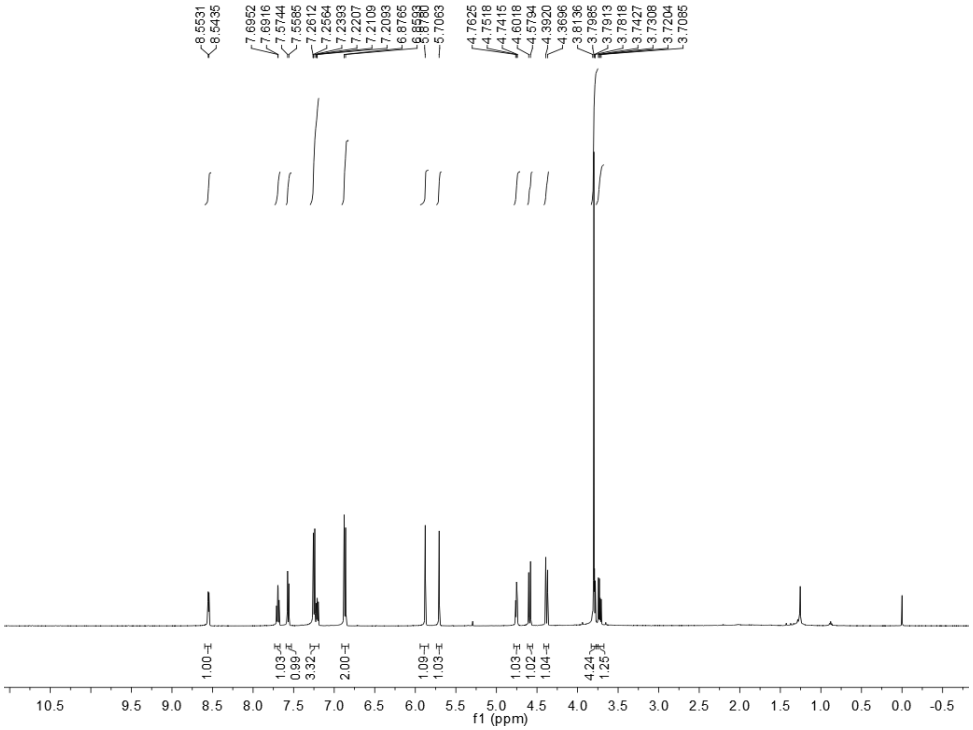
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7.2109
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5.7063
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4.7518
4.7415
4.6018
4.5794
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3.7985
3.7913
3.7818
3.7427
3.7308
3.7204
3.7085
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1.03
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2.00
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1.03
1.03
1.02
1.04
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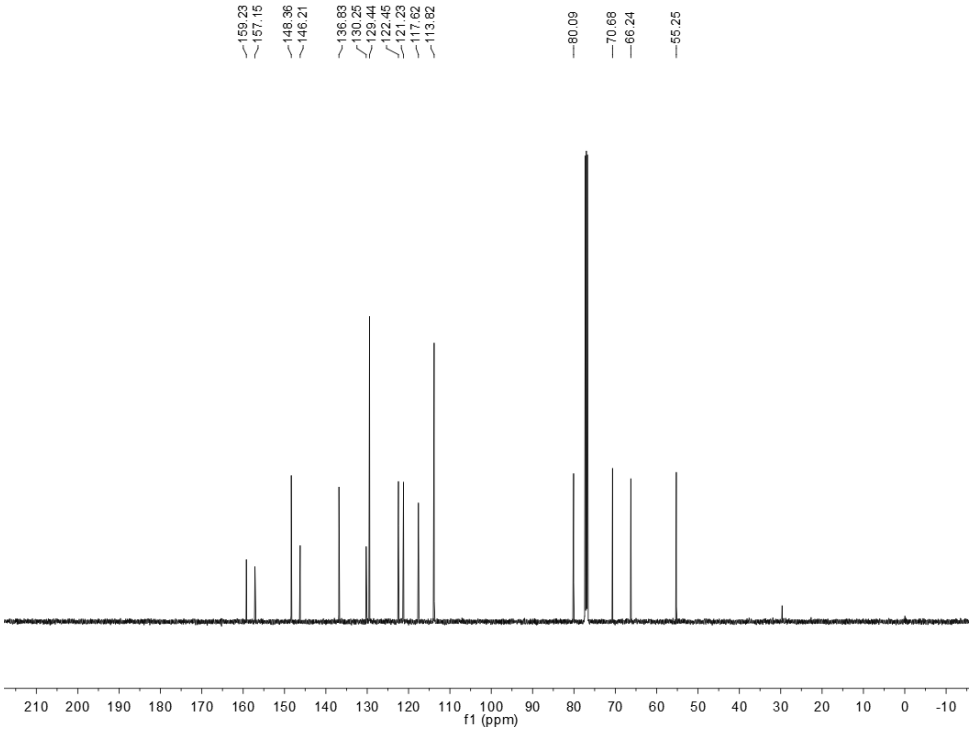
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113.82
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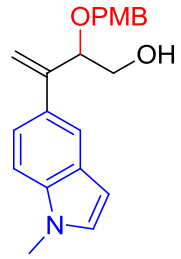
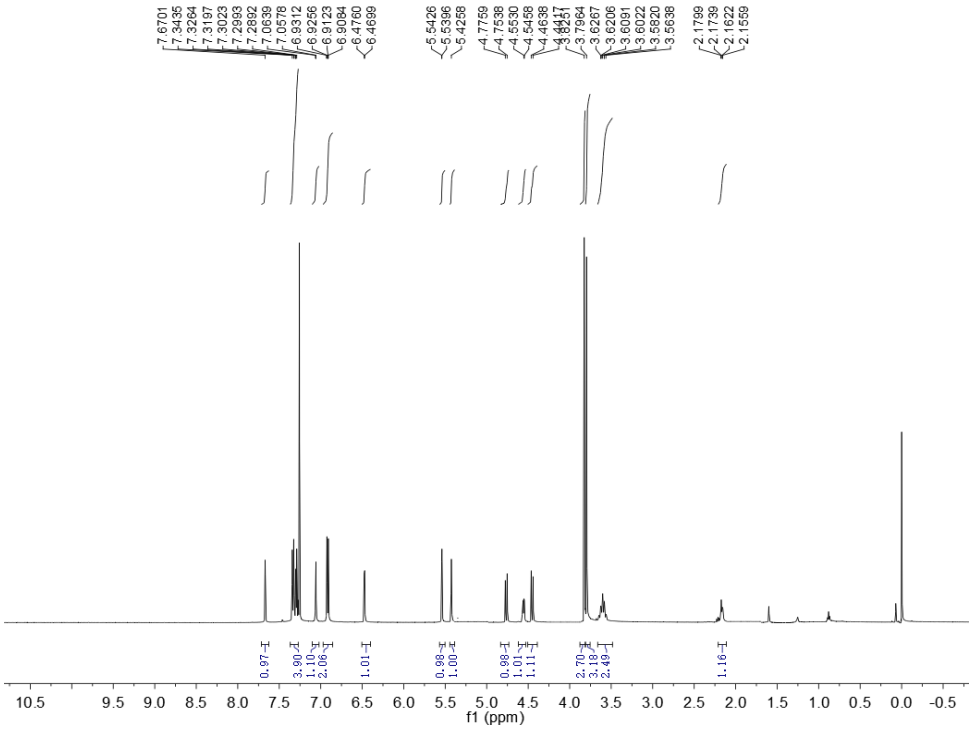
55.25
f1 (ppm)


OPMB
OH
N

4qb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6.9256
6.9123
6.9084
6.4760
6.4699
5.5426
5.5396
5.4258
4.7759
4.7538
4.5530
4.5458
4.4638
4.4417
3.8251
3.7964
3.6267
3.6206
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3.6022
3.5820
3.5638
2.1799
2.1739
2.1622
2.1559
0.97
3.90
1.10
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0.98
1.00
0.98
1.01
1.11
2.70
3.18
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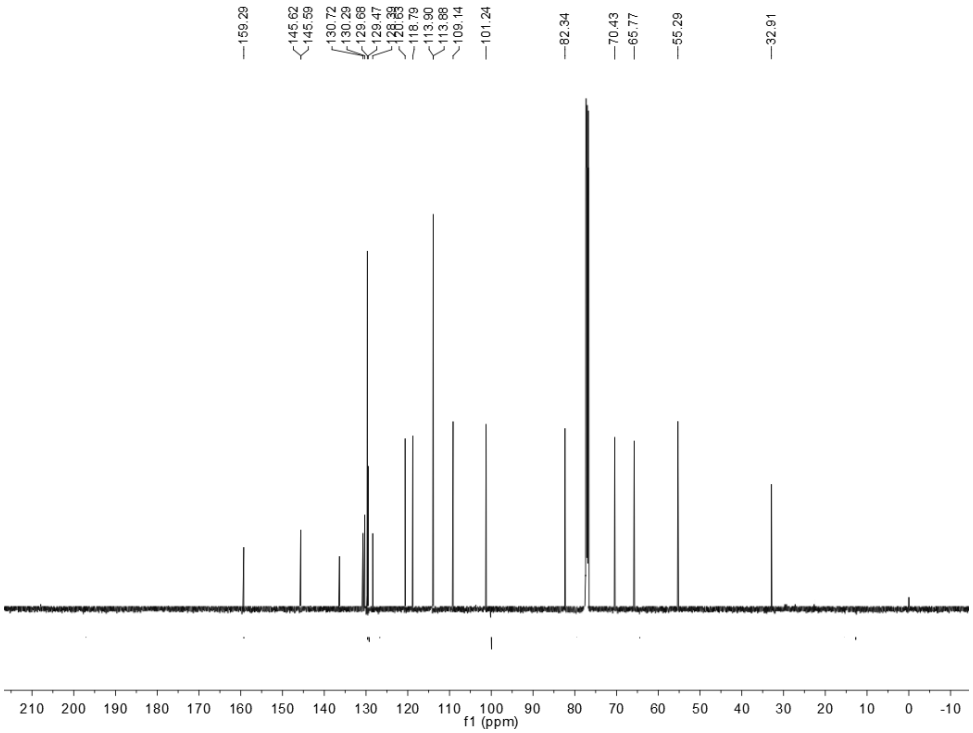
f1 (ppm)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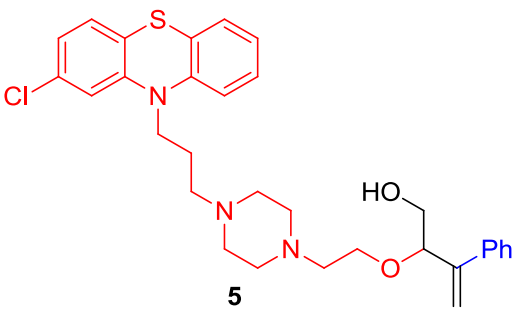
55.29
32.91
f1 (ppm)


S
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HO
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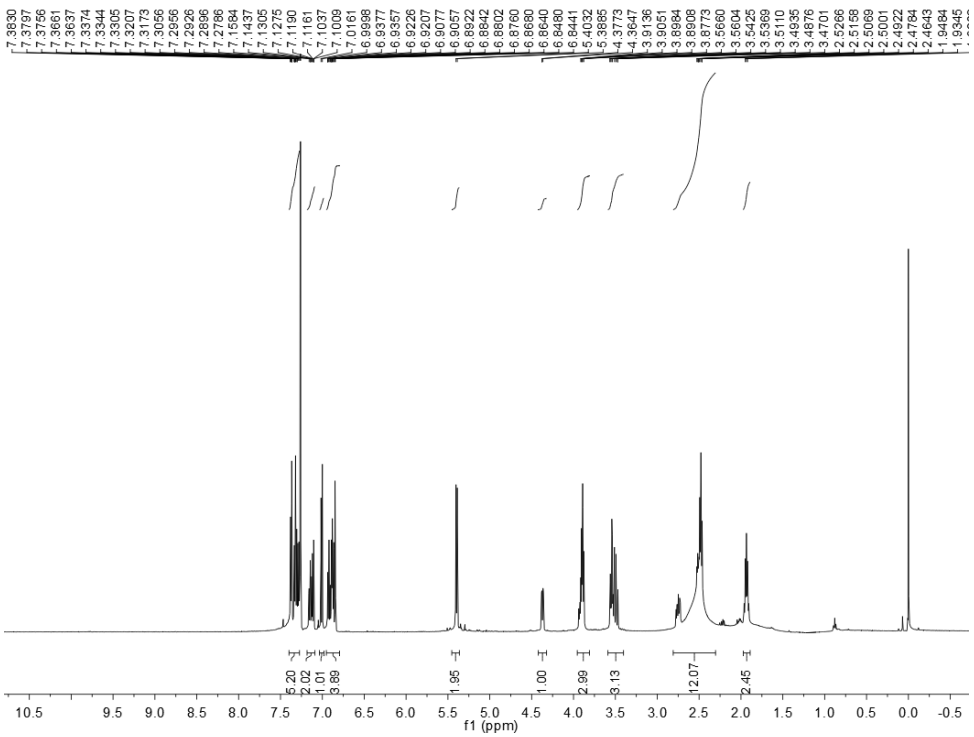
N
O
Ph

5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9.0
8.5
8.0
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6.5
6.0
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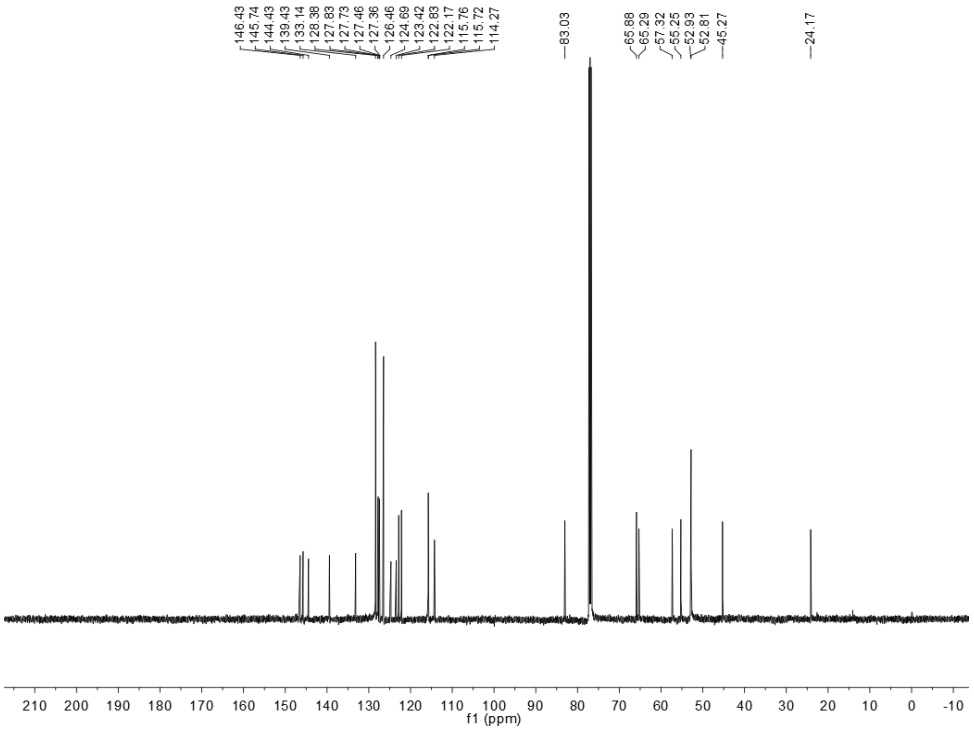
f1 (ppm)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122.17
115.76
115.72
114.27
83.03
65.88
65.29
57.32
55.25
52.93
52.81
45.27
24.17
210
200
190
180
170
160
150
140
130
120
110
100
90
80
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60
50
40
30
20
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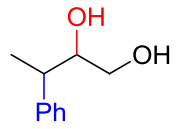
0
-10
f1 (ppm)



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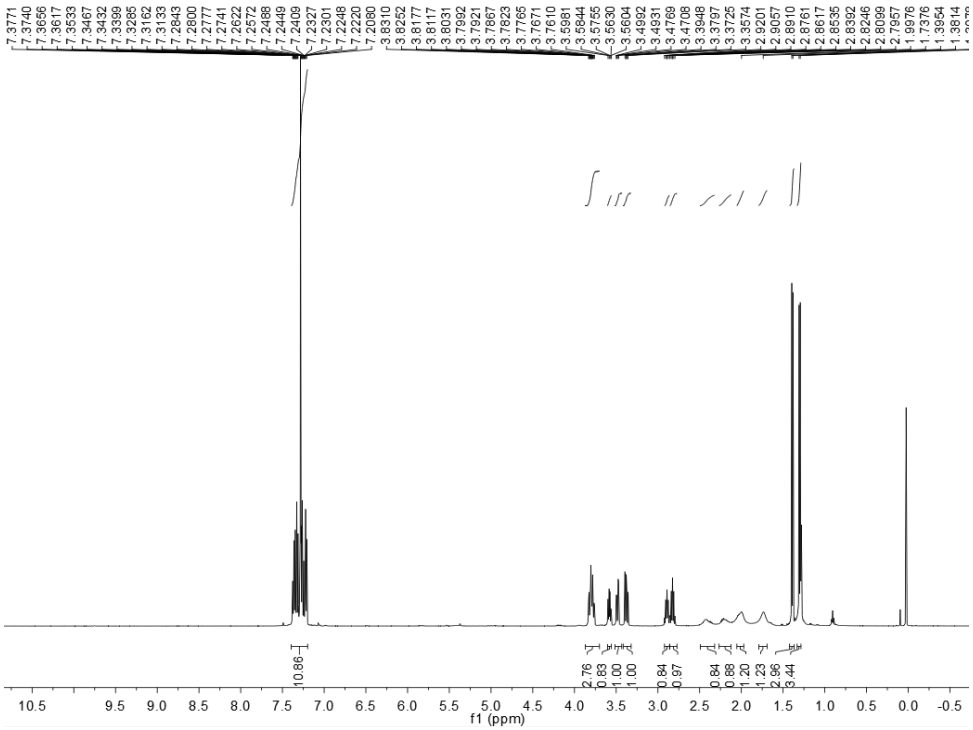
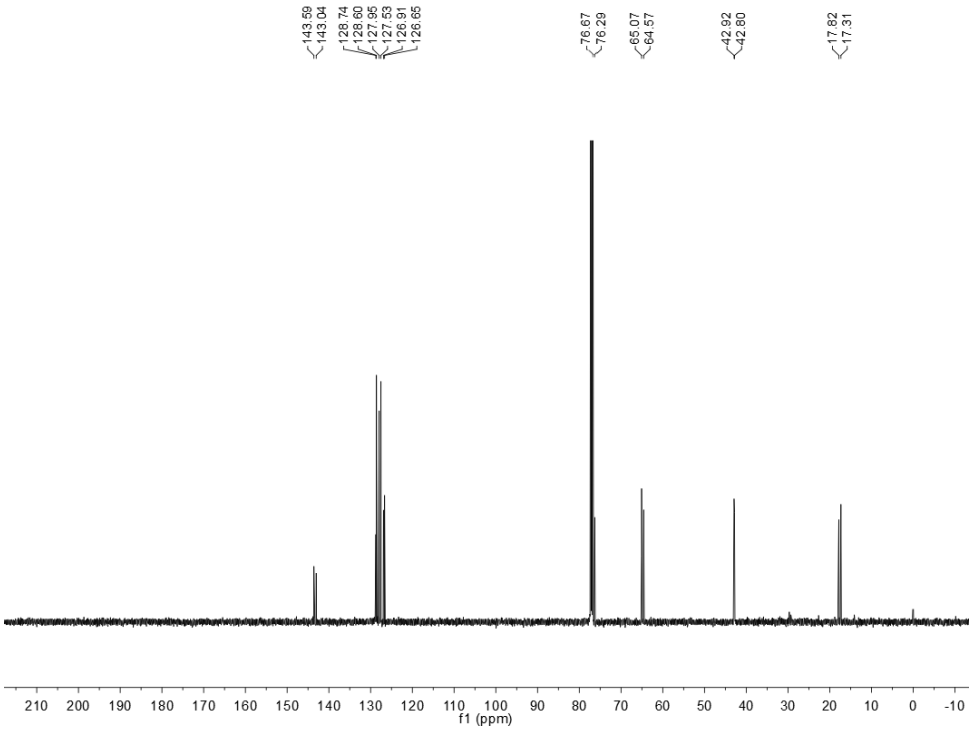
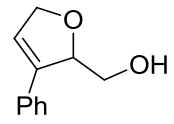
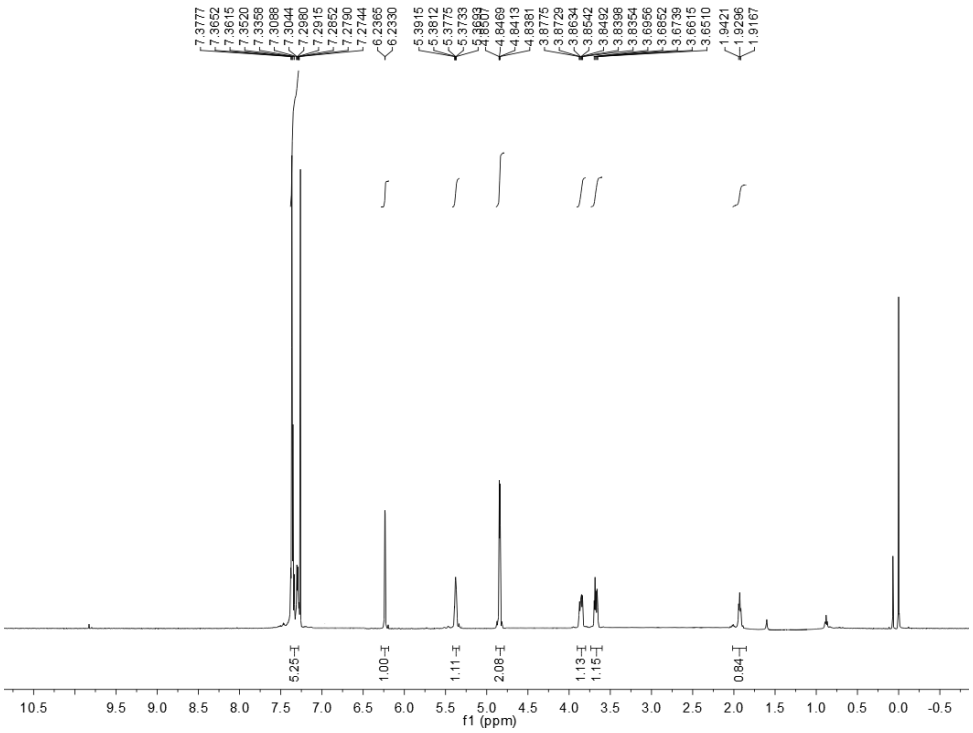





O
OH
Ph

7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3.6615
3.6510
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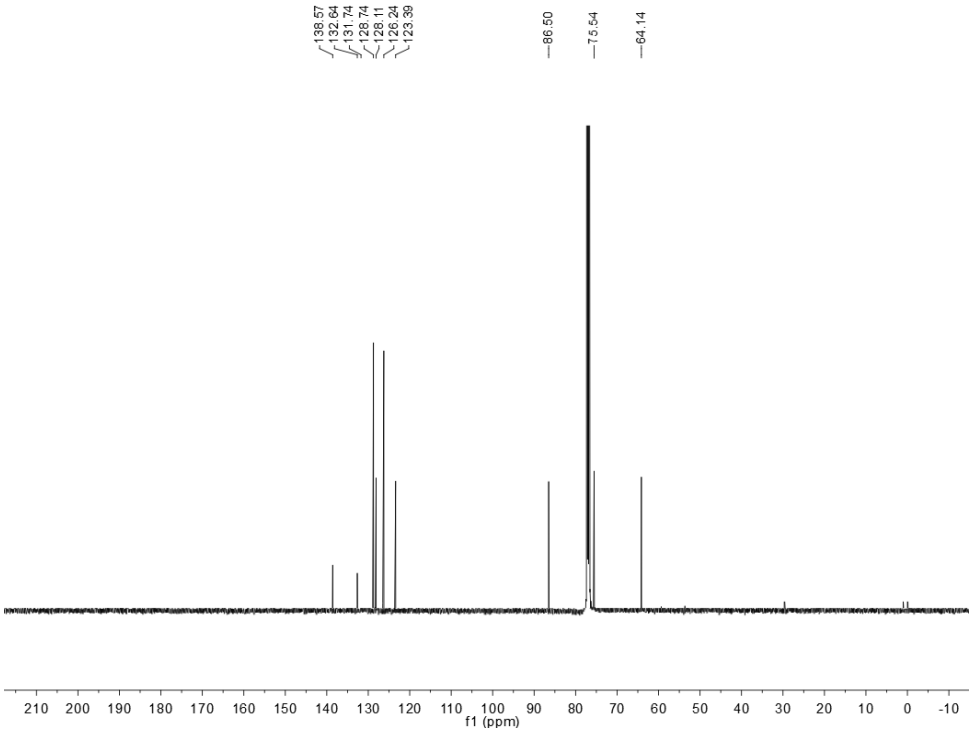
f1 (ppm)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126.24
123.39
86.50
75.54
64.14
f1 (ppm)