Supporting Information

n-BuLi/$LiCH_2CN$–mediated one-carbon homologation of aryl epoxides into conjugated allyl alcohols

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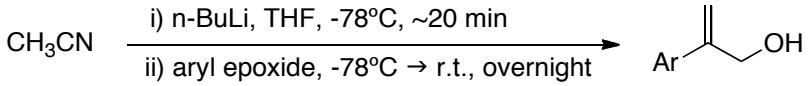
Materials and Methods

All experiments were performed in flame-dried glassware fitted with rubber septa under an argon atmosphere. Tetrahydrofuran (THF) was distilled over sodium/benzophenone ketyl. All aryl epoxides except styrene oxide and 2-methyl-3-phenyloxirane were prepared in accordance with literature procedure.[1] Unless otherwise noted, reagents were obtained from commercial sources and used as received. ^{1}H Nuclear Magnetic Resonance (NMR) spectra were recorded at 300 or 500 MHz. Data are presented as follows: chemical shift (in ppm on the δ scale relative to δH 7.26 for the residual protons in $CDCl_3$), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constant (*J*/Hz), integration. Coupling constants were taken directly from the spectra and are uncorrected. ^{13}C NMR spectra were recorded at 75 or 125 MHz, and all chemical shift values are reported in ppm on the δ scale, with an internal reference of δC 77.0 for $CDCl_3$. Analytical TLC was performed on silica gel plates using UV light and/or potassium permanganate stain followed by heating. Flash column chromatography was performed on silica gel 60A (32-63D).

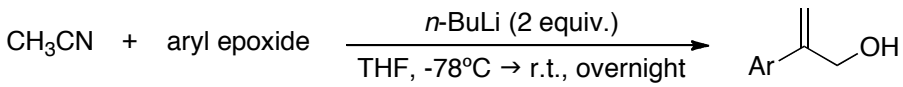
General Experimental Procedure

An original step-wise method for Table 1



Into a flame-dried 10 mL round-bottomed flask was added dry THF (3.0 mL) under an argon atmosphere. After cooling to –78ºC (acetone/dry-ice bath), *n*-BuLi (1.64 mL, 2.5 M in hexane, 4.1 mmol) and dry CH_3CN (110 μL, 2.1 mmol) were slowly added respectively. After stirring for 15–20 min, aryl epoxide (2.0 mmol) was then added. The reaction mixture was gradually warmed up to room temperature overnight and quenched with saturated $NaHCO_3$ (3 mL). After the phase separation, the aqueous layer was extracted with Et_2O (x2). The combined organics were washed with brine, dried over $MgSO_4$, and concentrated under reduced pressure. The crude product was purified by SiO_2 column chromatography (Hex/EtOAc = 7/3). **Caution:** due to the generation of cyanide ion during the course of the reaction, all operations, including work-up, should be carried out in a fume hood and the cyanide-containing waste should be properly handled and disposed.

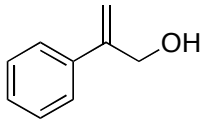
A modified, concise method for Schemes 6 and 7



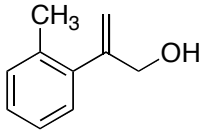
Into a flame-dried 10 mL round-bottomed flask were added aryl epoxide (2.0 mmol), dry CH_3CN (110 μL, 2.1 mmol), and dry THF (3.0 mL) under an argon atmosphere. After

cooling to –78ºC (acetone/dry-ice bath), *n*-BuLi (1.64 mL, 2.5 M in hexane, 4.1 mmol) was added dropwise. The reaction mixture was then gradually warmed up to room temperature overnight and quenched with saturated $NaHCO_3$ (3 mL). After the phase separation, the aqueous layer was extracted with Et_2O (x2). The combined organics were washed with brine, dried over $MgSO_4$, and concentrated under reduced pressure. The crude product was purified by SiO_2 column chromatography (Hex/EtOAc = 7/3 for **2a** and **2b**, and Hex/EtOAc = 9/1 for **2m**). **Caution:** due to the generation of cyanide ion during the course of the reaction, all operations, including work-up, should be carried out in a fume hood and the cyanide-containing waste should be properly handled and disposed.

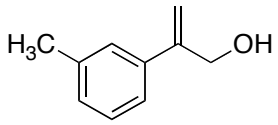
Spectral data



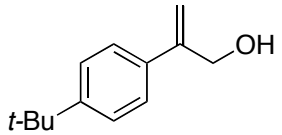
2-phenylprop-2-en-1-ol (2a): Column chromatography yielded **2a** as a colorless liquid (215 mg, 80%). R_f 0.30 (Hex/EtOAc = 7/3); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47–7.44 (m, 2H), 7.39–7.29 (m, 3H), 5.48 (s, 1H), 5.36 (d, J = 1.2 Hz, 1H), 4.56 (d, J = 6.0 Hz, 2H), 1.55 (t, J = 6.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.2. 138.5, 128.5, 127.9, 126.0, 112.5, 64.9; HRMS (TOF MS ES$^+$) calcd for $C_9H_{11}O$ 135.0810 $[M + H]^+$, found 135.0781. The spectral data matched those reported previously.[2]



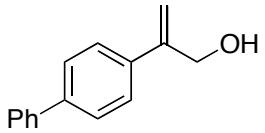
2-(*o*-tolyl)prop-2-en-1-ol (2b): Column chromatography yielded **2b** as a colorless liquid (240 mg, 81%). R_f 0.37 (Hex/EtOAc = 7/3); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30–7.10 (m, 4H), 5.49 (d, J = 1.5 Hz, 1H), 5.06 (d, J = 1.2 Hz, 1H), 4.31 (d, J = 6.3 Hz, 2H), 2.32 (s, 3H), 1.75 (t, J = 6.3 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.0, 139.6, 135.4, 130.2, 128.7, 127.5, 125.6, 113.2, 66.1, 19.7; HRMS (TOF MS ES$^+$) calcd for $C_{10}H_{13}O$ 149.0966 $[M + H]^+$, found 149.0957.



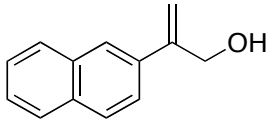
2-(*m*-tolyl)prop-2-en-1-ol (2c): Column chromatography yielded **2c** as a colorless liquid (213 mg, 72%). R_f 0.32 (Hex/EtOAc = 7/3); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28–7.23 (m, 3H), 7.15–7.11 (m, 1H), 5.45 (s, 1H), 5.33 (d, J = 1.0 Hz, 1H), 4.54 (d, J = 5.5 Hz, 2H), 2.37 (s, 3H), 1.64 (t, J = 5.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 147.4, 138.5, 138.1, 128.7, 128.4, 126.8, 123.2, 112.4, 65.1, 21.5; HRMS (TOF MS ES$^+$) calcd for $C_{10}H_{13}O$ 149.0966 $[M + H]^+$, found 149.0940.



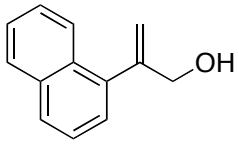
2-(4-(tert-butyl)phenyl)prop-2-en-1-ol (2d): Column chromatography yielded **2d** as a colorless liquid (194 mg, 51%). R_f 0.36 (Hex/EtOAc = 7/3); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40–7.36 (m, 4H), 5.46 (s, 1H), 5.31 (d, J = 1.2 Hz, 1H), 4.55 (d, J = 6.0 Hz, 2H), 1.54 (t, J = 6.0 Hz, 1H), 1.33 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.0, 147.0, 135.5, 125.7, 125.4, 111.9, 65.1, 34.5, 31.3. The spectral data matched those reported previously.[3]



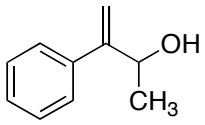
2-([1,1'-biphenyl]-4-yl)prop-2-en-1-ol (2e): Column chromatography yielded **2e** as a yellow solid (269 mg, 64%). R_f 0.24 (Hex/EtOAc = 7/3); **1**H NMR (500 MHz, $CDCl_3$) δ 7.65–7.30 (m, 9H), 5.54 (s, 1H), 5.39 (d, J = 1.0 Hz, 1H), 4.60 (d, J = 6.5 Hz, 2H), 1.56 (t, J = 6.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 146.8, 140.8, 140.6, 137.3, 128.8, 127.4, 127.2, 127.0, 126.5, 112.7, 65.1. The spectral data matched those reported previously.[3]



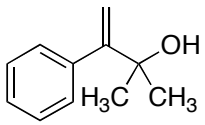
2-(naphthalen-2-yl)prop-2-en-1-ol (2f): Column chromatography yielded **2f** as a white solid (261 mg, 71%). R_f 0.28 (Hex/EtOAc = 7/3); **1**H NMR (300MHz, $CDCl_3$) δ 7.90–7.80 (m, 4H), 7.64–7.60 (m, 1H), 7.51–7.45 (m, 2H), 5.63 (s, 1H), 5.47 (d, J = 0.9 Hz, 1H), 4.68 (d, J = 5.1 Hz, 2H), 1.55 (t, J = 5.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.1, 135.7, 133.4, 133.0, 128.2, 128.1, 127.6, 126.3, 126.1, 124.8, 124.3, 113.2, 65.2. The spectral data matched those reported previously.[4]



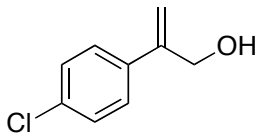
2-(naphthalen-1-yl)prop-2-en-1-ol (2g): Column chromatography yielded **2g** as a colorless liquid (284 mg, 77%). R_f 0.30 (Hex/EtOAc = 7/3); ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.05–8.01 (m, 1H), 7.89–7.79 (m, 2H), 7.52–7.31 (m, 4H), 5.71 (d, J = 1.5 Hz, 1H), 5.27 (d, J = 1.5 Hz, 1H), 4.48 (d, J = 6.0 Hz, 2H), 1.67 (t, J = 6.3 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 147.8, 137.9, 133.7, 131.5, 128.3, 127.8, 126.1, 125.8, 125.7, 125.4, 125.2, 114.8, 66.7. The spectral data matched those reported previously.[5]



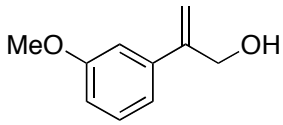
3-phenylbut-3-en-2-ol (2h): Column chromatography yielded **2h** as a colorless liquid (193 mg, 65%). R_f 0.36 (Hex/EtOAc = 7/3); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40–7.30 (m, 5 H), 5.37 (s, 1 H), 5.28 (s, 1H), 4.85–4.78 (m, 1H), 1.68 (d, J = 3.5 Hz, 1H), 1.33 (d, J = 6.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.1, 139.9, 128.4, 127.6, 126.8, 111.6, 69.5, 22.6. The spectral data matched those reported previously.[6]



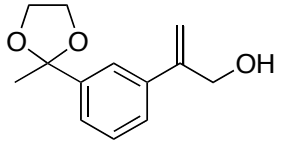
2-methyl-3-phenylbut-3-en-2-ol (2i): Column chromatography yielded **2i** as a white solid (211 mg, 65%). R_f 0.42 (Hex/EtOAc = 7/3); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33–7.27 (m, 5H), 5.43 (d, J = 1.2 Hz, 1H), 4.97 (d, J = 0.9 Hz, 1H), 1.56 (s, 1H), 1.42 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.1, 141.5, 128.8, 127.8, 127.0, 112.5, 73.0, 29.7; HRMS (TOF MS ES$^-$) calcd for $C_{11}H_{13}O$ 161.0966 $[M - H]^-$, found 161.0945.



2-(4-chlorophenyl)prop-2-en-1-ol (2j): Column chromatography yielded **2j** as a light yellow liquid (148 mg, 44%). R_f 0.25 (Hex/EtOAc = 7/3); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41–7.30 (m, 4H), 5.47 (d, J = 0.9, 1H), 5.37 (d, J = 0.9, 1H), 4.52 (d, J = 4.8 Hz, 2H), 1.55 (t, J = 5.4 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 146.2, 136.9, 133.8, 128.7, 127.4, 113.3, 65.0. The spectral data matched those reported previously.[2]

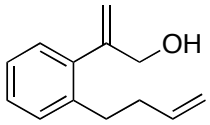


2-(3-methoxyphenyl)prop-2-en-1-ol (2k): Column chromatography yielded **2k** as a colorless liquid (197 mg, 60%). R_f 0.24 (Hex/EtOAc = 7/3); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31–7.25 (m, 1H), 7.06–6.98 (m, 2H), 6.88–6.84 (m, 1H), 5.47 (d, J = 0.9 Hz, 1H), 5.36 (d, J = 1.2 Hz, 1H), 4.53 (d, J = 6.0 Hz, 2H), 3.83 (s, 3H), 1.60 (t, J = 6.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.7, 147.2, 140.0, 129.5, 118.6, 113.2, 112.8, 112.1, 65.1, 55.2; HRMS (TOF MS ES$^-$) calcd for $C_{10}H_{11}O_2$ 163.0759 $[M - H]^-$, found 163.0788.

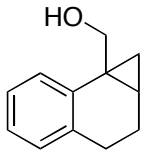


2-(3-(2-methyl-1,3-dioxolan-2-yl)phenyl)prop-2-en-1-ol (2l): Column chromatography yielded **2l** as a colorless liquid (287 mg, 65%). R_f 0.17 (Hex/EtOAc = 7/3); ^{1}H NMR

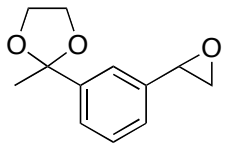
(500 MHz, $CDCl_3$) δ 7.57 (s, 1H), 7.45–7.31 (m, 3H), 5.50 (s, 1H), 5.37 (s, 1H), 4.56 (d, J = 6.0 Hz, 2H), 4.05 (t, J = 7.0 Hz, 2H), 3.79 (t, J = 7.0 Hz, 2H), 1.67 (s, 3H), 1.55 (t, J = 6.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.3, 143.7, 138.5, 128.4, 125.6, 124.9, 122.9, 112.8, 108.8, 65.0, 64.5, 27.7; HRMS (TOF MS ES$^+$) calcd for $C_{13}H_{17}O_3$ 221.1178 $[M + H]^+$, found 221.1151.



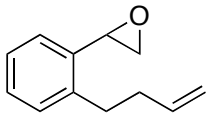
2-(2-(but-3-en-1-yl)phenyl)prop-2-en-1-ol (2m): Column chromatography yielded **2m** as a light orange liquid (149 mg, 40%). R_f 0.31 (Hex/EtOAc = 9/1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26–7.09 (m, 4H), 5.86 (ddt, J = 16.8, 10.2, 6.6 Hz, 1H), 5.49 (d, J = 0.9 Hz, 1H), 5.10–4.95 (m, 3H), 4.31 (s, 2H), 2.72 (t, 7.8 Hz, 2H), 2.34 (apparent q, J = 7.5 Hz, 2H), 1.72 (bs,1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 148.7, 139.4, 139.3, 138.1, 129.2, 129.0, 127.6, 125.7, 114.8, 113.2, 66.6, 35.7, 32.3; HRMS (TOF MS ES$^+$) calcd for $C_{13}H_{17}O$ 189.1279 $[M + H]^+$, found 189.1260.



cyclopropane (9): The initial column chromatography (Hex/EtOAc = 9/1) separated **2m** and **9**, but **9** was still impure. The second column chromatography ($CHCl_3$/EtOAc = 3/1) yielded pure **9** as a white solide (94 mg, 27%). R_f 0.21 (Hex/EtOAc = 9/1), R_f 0.64 ($CHCl_3$/EtOAc = 3/1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.59 (d, J = 7.8 Hz, 1H), 7.22–7.14 (m, 1H), 7.11–7.00 (m, 2H), 4.07 (d, J = 11.7 Hz, 1H), 3.57 (d, J = 11.7 Hz, 1H), 2.63 (ddd, J = 15.9, 5.1, 2.4 Hz, 1H), 2.47 (ddd, J = 15.9, 12.6, 6.0 Hz, 1H), 2.15–1.98 (m, 2H), 1.86–1.72 (m, 1H), 1.52–1.43 (m, 1H), 0.95 (apparent t, J = 5.1 Hz, 1H), 0.79 (dd, J = 8.4, 5.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.2, 135.0, 128.8, 126.3, 126.0, 125.0, 69.0, 26.4, 24.3, 21.5, 19.7, 13.1; HRMS (TOF MS ES$^+$) calcd for $C_{12}H_{15}O$ 175.1123 $[M + H]^+$, found 175.1114.



epoxide (entry 11 in table 1): The epoxide was prepared from 3-(2-methyl-1,3-dioxolan-2-yl)benzaldehyde[7] and was obtained as a colorless liquid. R_f 0.21 (Hex/EtOAc = 99:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45–7.41 (m, 2H), 7.36–7.29 (m, 1H), 7.22–7.18 (m, 1H), 4.08–4.00 (m, 2H), 3.89–3.85 (m, 1H), 3.81–3.74 (m, 2H), 3.15 (dd, J = 5.4, 4.2 Hz, 1H), 2.83 (dd, J = 5.4, 2.7 Hz, 1H), 1.65 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.9, 137.7, 128.5, 125.2, 124.9, 122.7, 108.7, 64.5, 64.4, 52.4, 51.1, 27.6; HRMS (TOF MS ES$^+$) calcd for $C_{12}H_{15}O_3$ 207.1021 $[M + H]^+$, found 207.0993.



epoxide (8): The epoxide was prepared from 2-(but-3-en-1-yl)benzaldehyde[8] and was obtained as a colorless liquid. R_f 0.64 (toluene); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30–7.19 (m, 4H), 5.92 (ddt, J = 17.1, 10.2, 6.6 Hz, 1H), 5.14–5.02 (m, 2H), 4.07 (dd, J = 3.9, 2.7 Hz, 1H), 3.17 (dd, J = 5.7, 4.2 Hz, 1H), 2.92–2.85 (m, 2H), 2.72 (dd, J = 5.7, 2.7 Hz, 1H), 2.48–2.38 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.0, 137.6, 135.4, 128.9, 127.6, 126.3, 124.1, 115.1, 50.3, 50.0, 35.0, 32.0; HRMS (TOF MS ES$^+$) calcd for $C_{12}H_{15}O$ 175.1123 $[M + H]^+$, found 175.1096.

References:

[1] (a) Nielsen, D. K.; Doyle, A. G. *Angew. Chem. Int. Ed.* **2011,** *50,* 6056. (b) Ebner, C.; Muller, C. A.; Markert, C.; Pfaltz, A. *J. Am. Chem. Soc.* **2011,** *133,* 4710.

[2] Sun, X.; Frimpong, K.; Tan, K. L. *J. Am. Chem. Soc.* **2010,** *132,* 11841.

[3] Hollingworth, C.; Hazari, A.; Hopkinson, M. N.; Tredwell, M.; Benedetto, E.; Huiban, M.; Gee, A. D.; Brown, J. M.; Gouverneur, V. *Angew. Chem. Int. Ed.* **2011,** *50,* 2613.

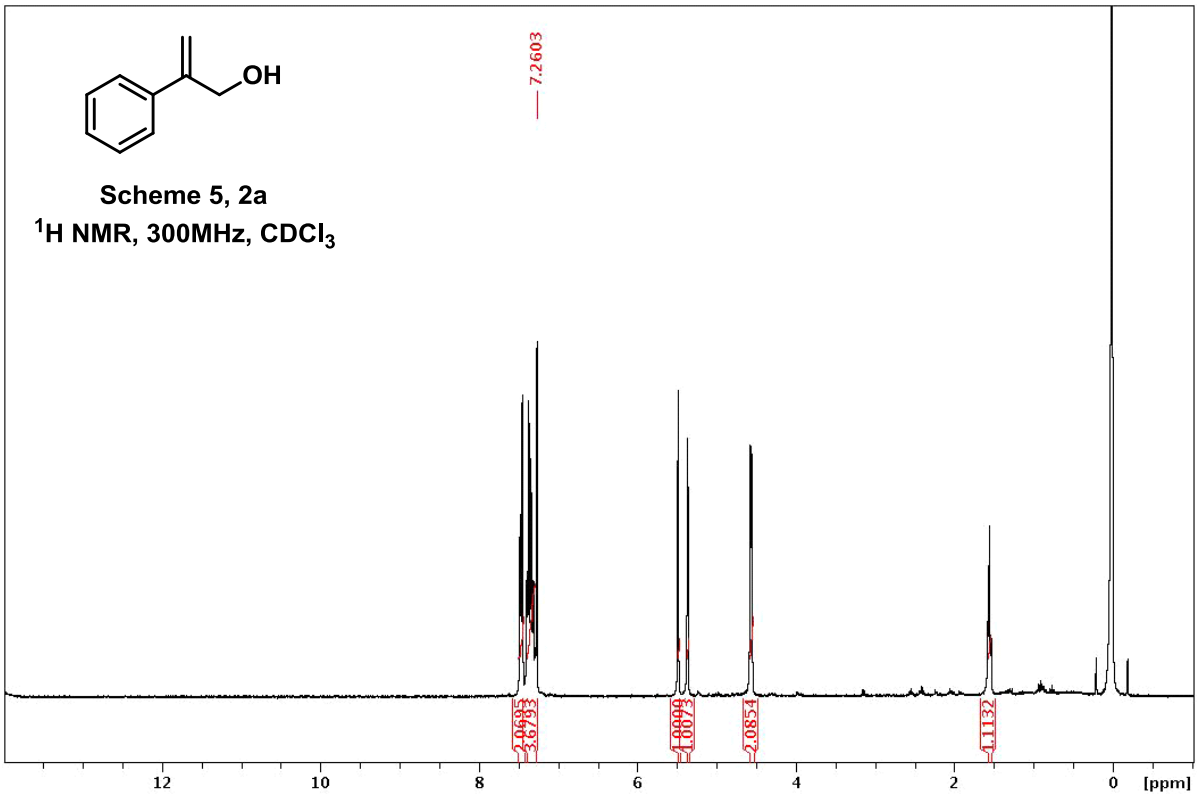
[4] Mo, L.; Xu, L.; Ruan, J.; Liu, S.; Xiao, J. *Chem. Commun.* **2006,** 3591.

[5] Piel, I.; Steinmetz, M.; Hirano, K.; Frohlich, R.; Grimme, S.; Glorius, F. *Angew. Chem. Int. Ed.* **2011,** *50,* 4983.

[6] Gao, G.-Y.; Jones, J. E.; Vyas, R.; Harden, J. D.; Zhang, X. P. *J. Org. Chem.* **2006,** *71,* 6655.

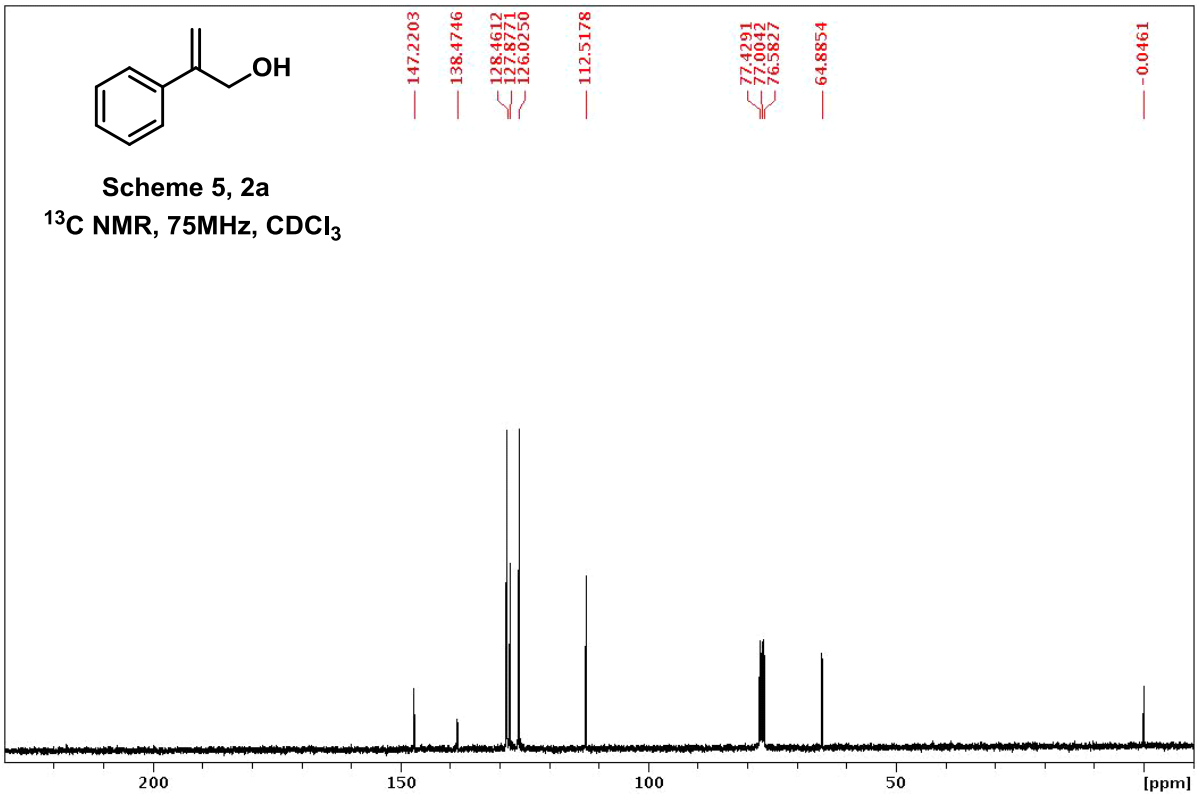
[7] (a) Suh, Y.-G.; Kim, N.-J.; Koo, B.-W.; Lee, K.-O.; Moon, S.-H.; Shi, D.-H.; Jung, J.-W.; Paek, S.-M.; Chang, D.-J.; Li, F.; Kang, H.-J.; Le, T. V. T.; Chae, Y. N.; Shin, C. Y.; Kim, M.-K.; Lim, J. I.; Ryu, J.-S.; Park, H.-J. *J. Med. Chem.* **2008,** *51,* 6318. (b) Chun, J.; Yin, Y. I.; Yang, G.; Tarassishin, L.; Li, Y.-M. *J. Org. Chem.* **2004,** *69,* 7344.

[8] Watson, I. D. G.; Ritter, S.; Toste, F. D. *J. Am. Chem. Soc.* **2009,** *131,* 2058.



Scheme 5, 2a

^{1}H NMR, 300MHz, $CDCl_3$



Scheme 5, 2a

^{13}C NMR, 75MHz, $CDCl_3$

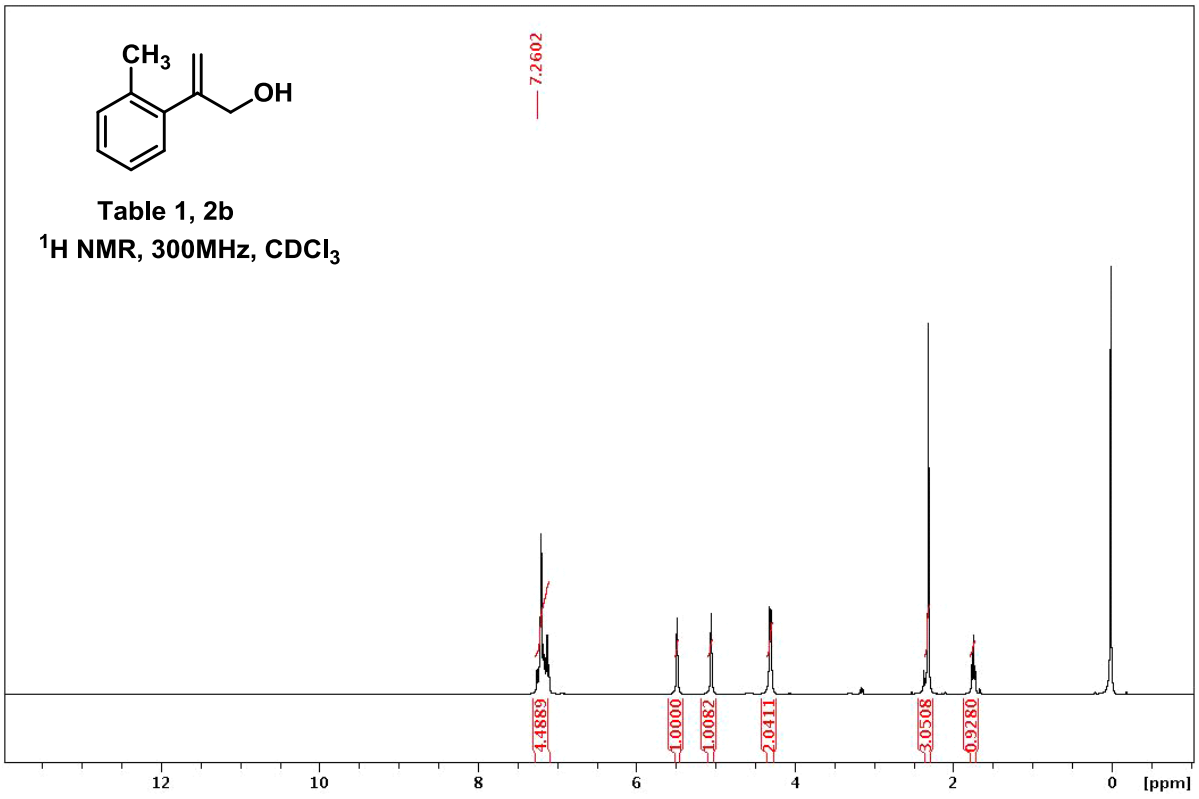


Table 1, 2b

^{1}H NMR, 300MHz, $CDCl_3$

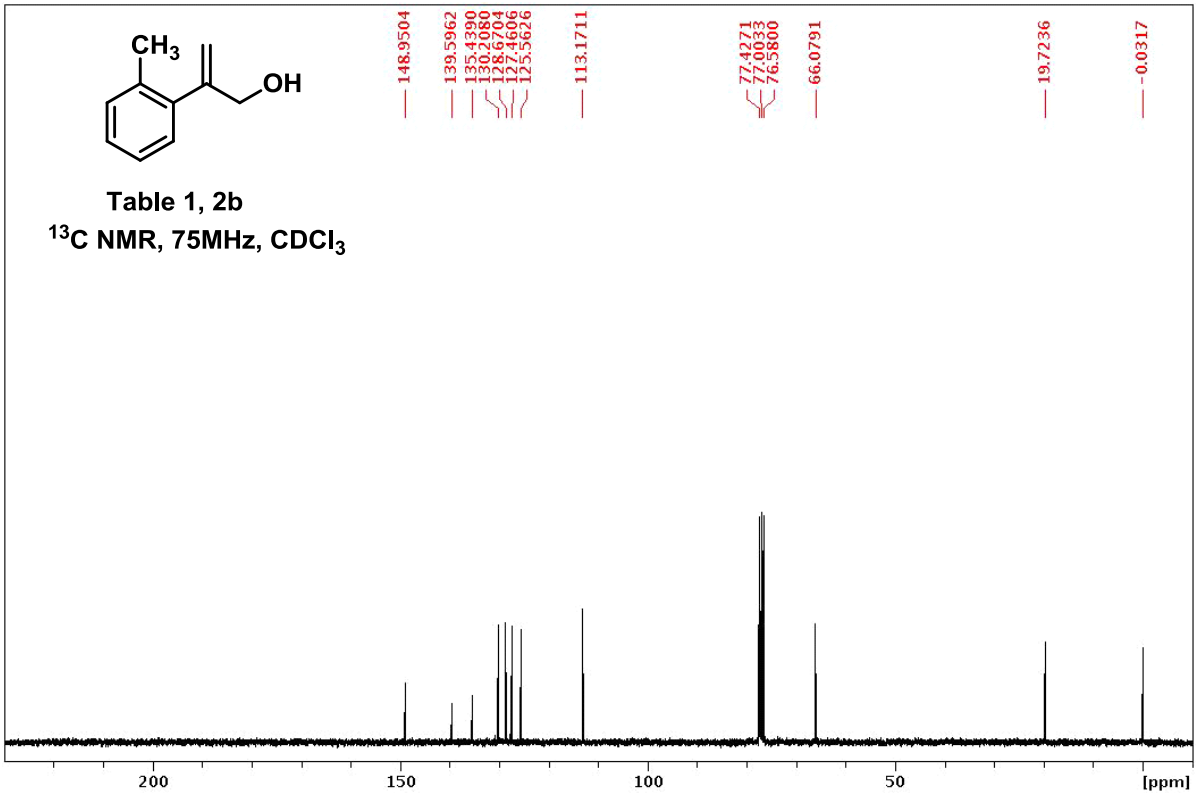
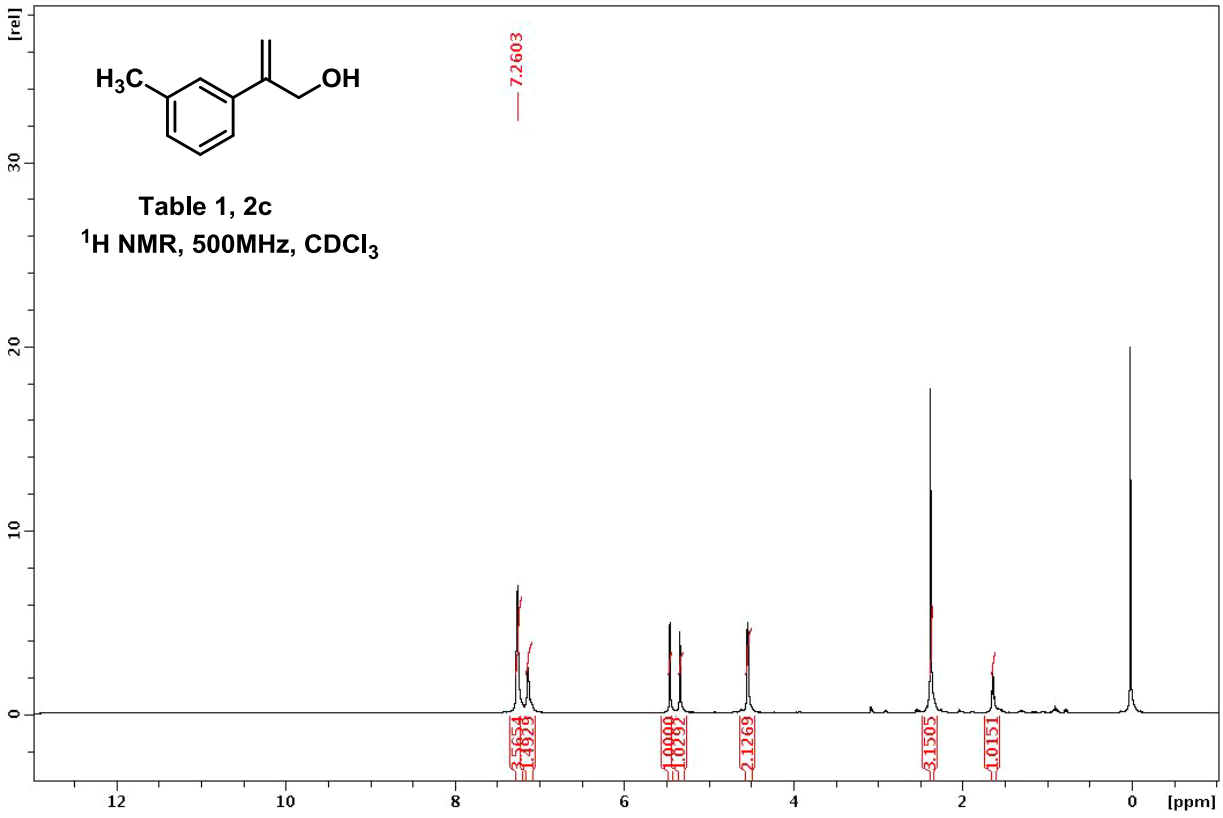
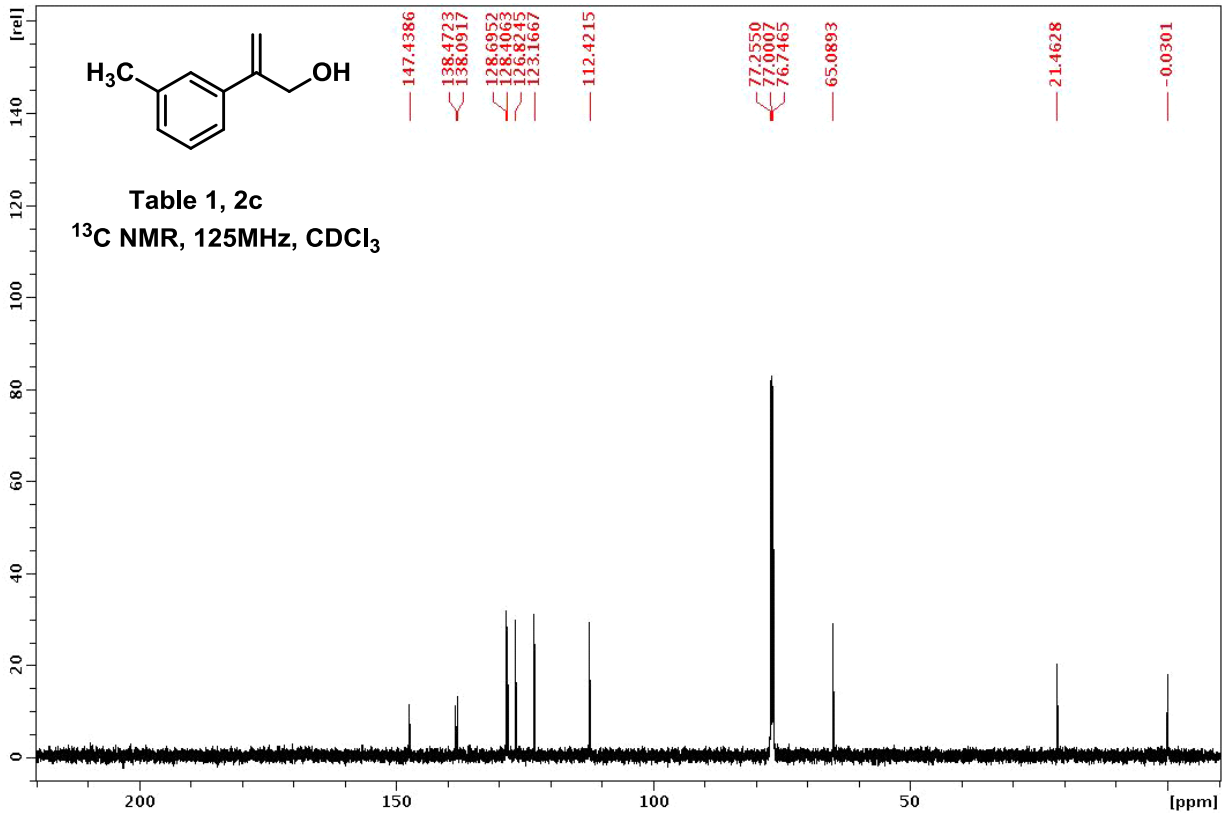


Table 1, 2b

^{13}C NMR, 75MHz, $CDCl_3$


H3C
OH
7.2603
Table 1, 2c
1H NMR, 500MHz, CDCl3
[rel]
[ppm]
2.1269
3.1505
1.0151


H3C
OH
147.4386
138.4723
138.0917
128.6952
128.4063
126.8245
123.1667
112.4215
77.2550
77.0007
76.7465
65.0893
21.4628
-0.0301
Table 1, 2c
13C NMR, 125MHz, CDCl3
[rel]
[ppm]

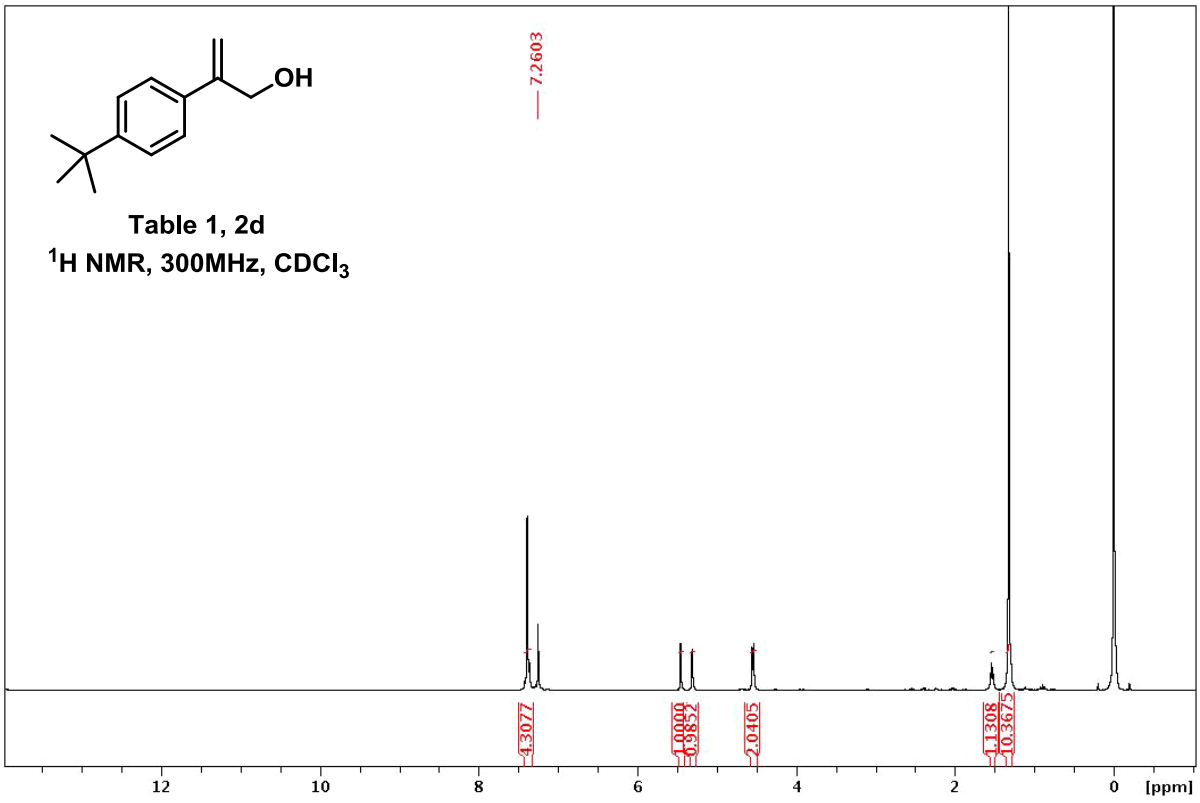


Table 1, 2d

^{1}H NMR, 300MHz, $CDCl_3$

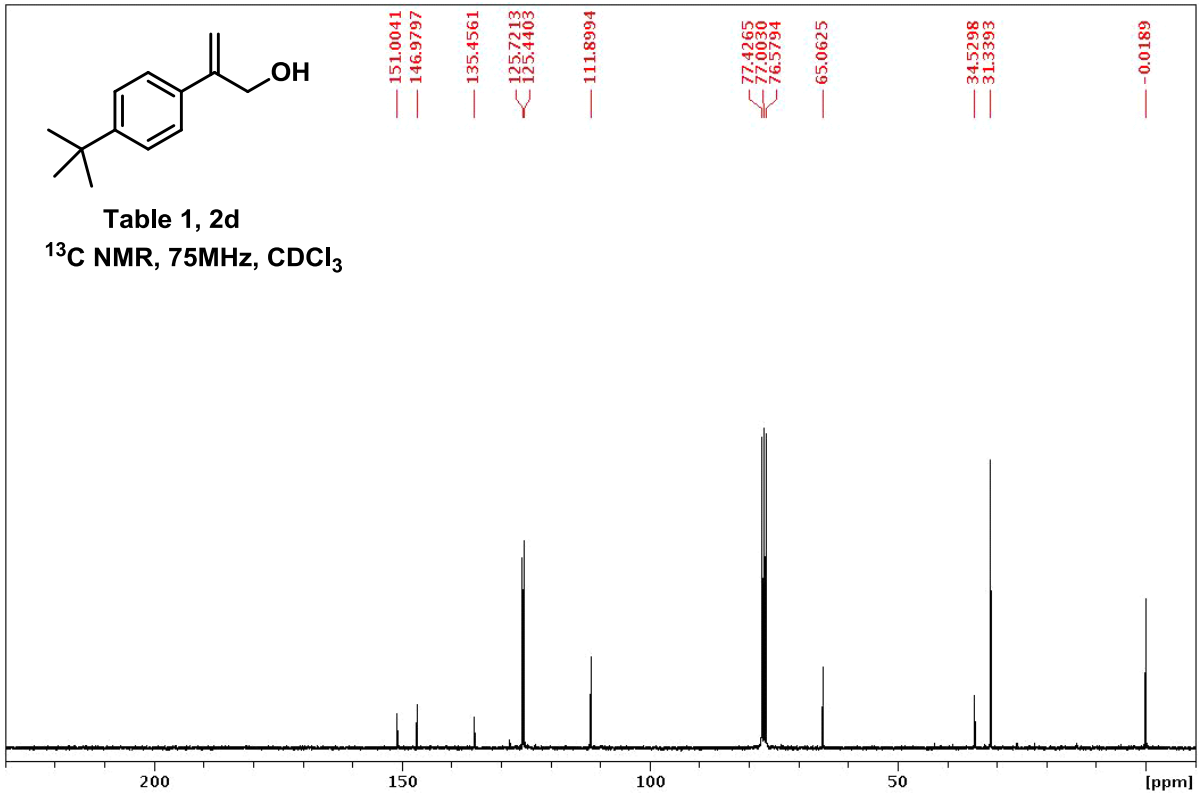
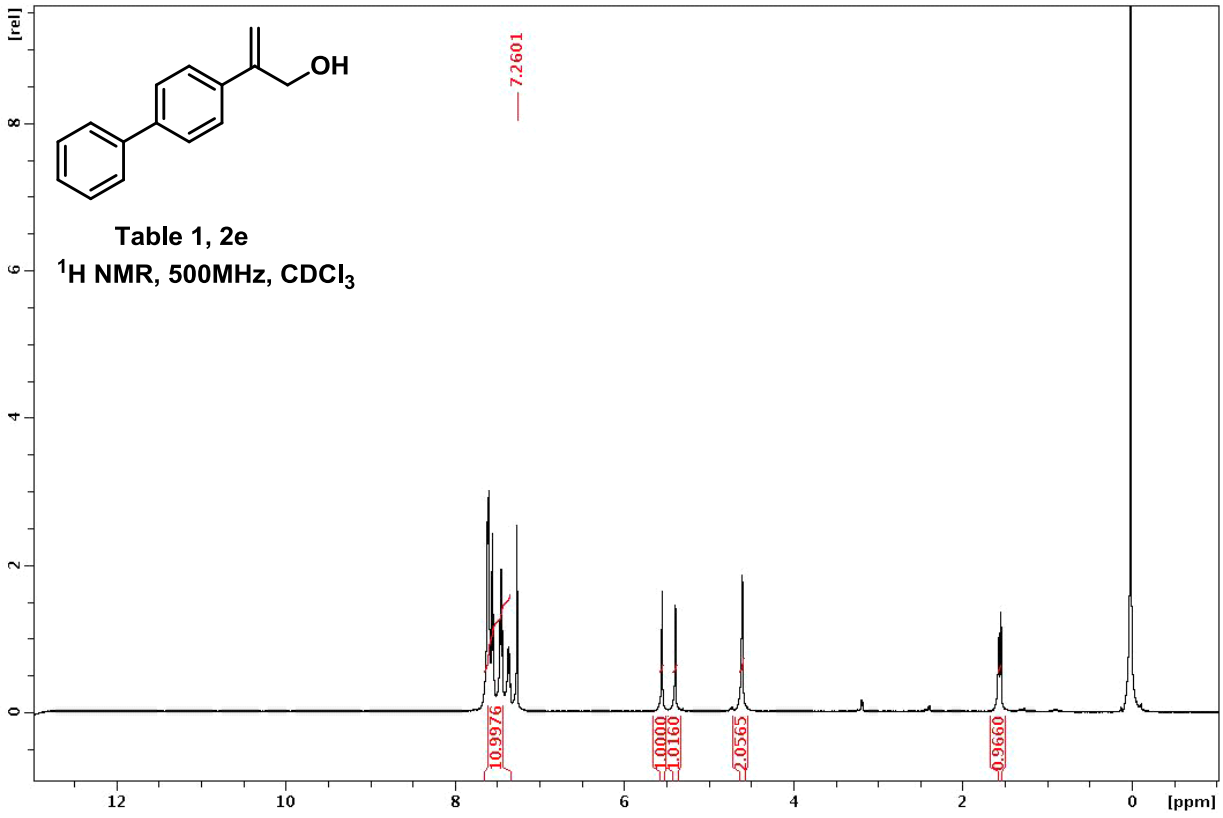
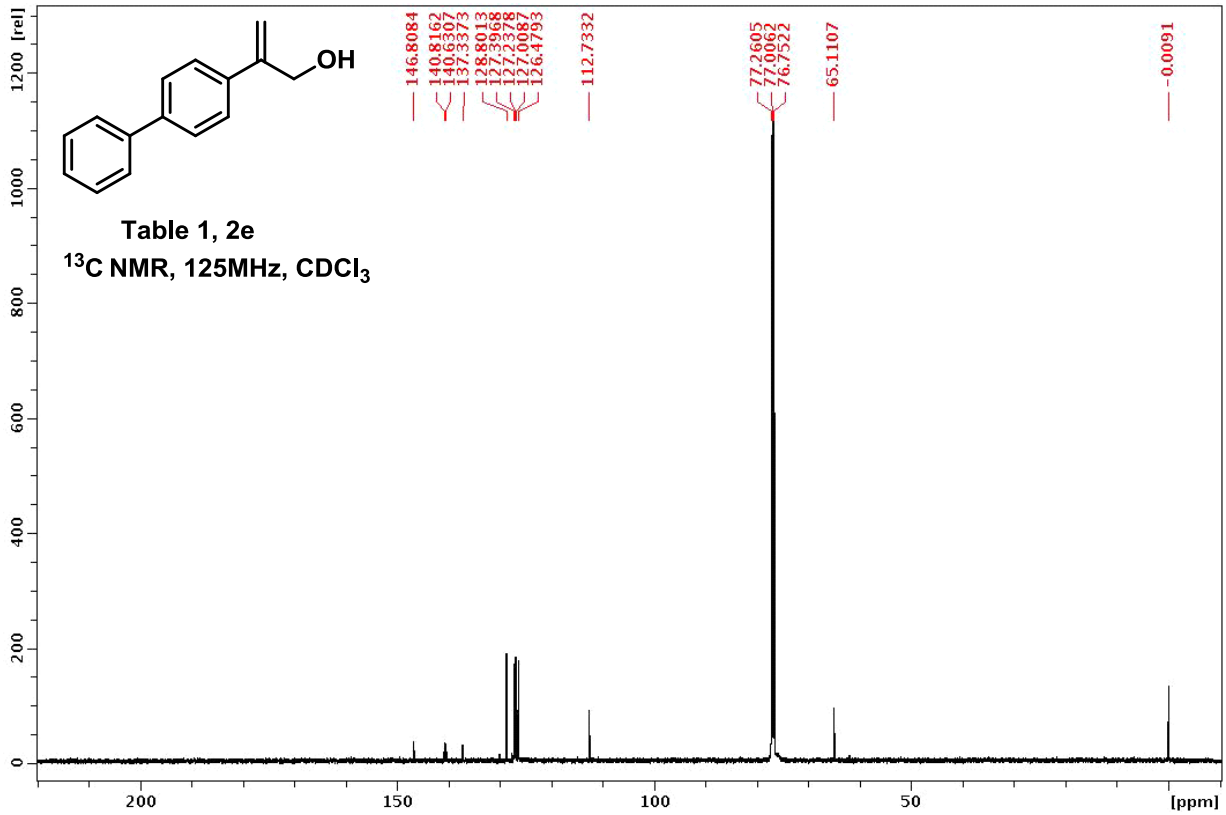


Table 1, 2d

^{13}C NMR, 75MHz, $CDCl_3$


OH
7.2601
Table 1, 2e
^{1}H NMR, 500MHz, $CDCl_3$
[rel]
[ppm]
10.9976
1.0000
1.0160
2.0565
0.9660


OH
146.8084
140.8162
140.6307
137.3373
128.8013
127.3968
127.2378
127.0087
126.4793
112.7332
77.2605
77.0962
76.7322
65.1107
-0.0091
Table 1, 2e
^{13}C NMR, 125MHz, $CDCl_3$
[rel]
[ppm]

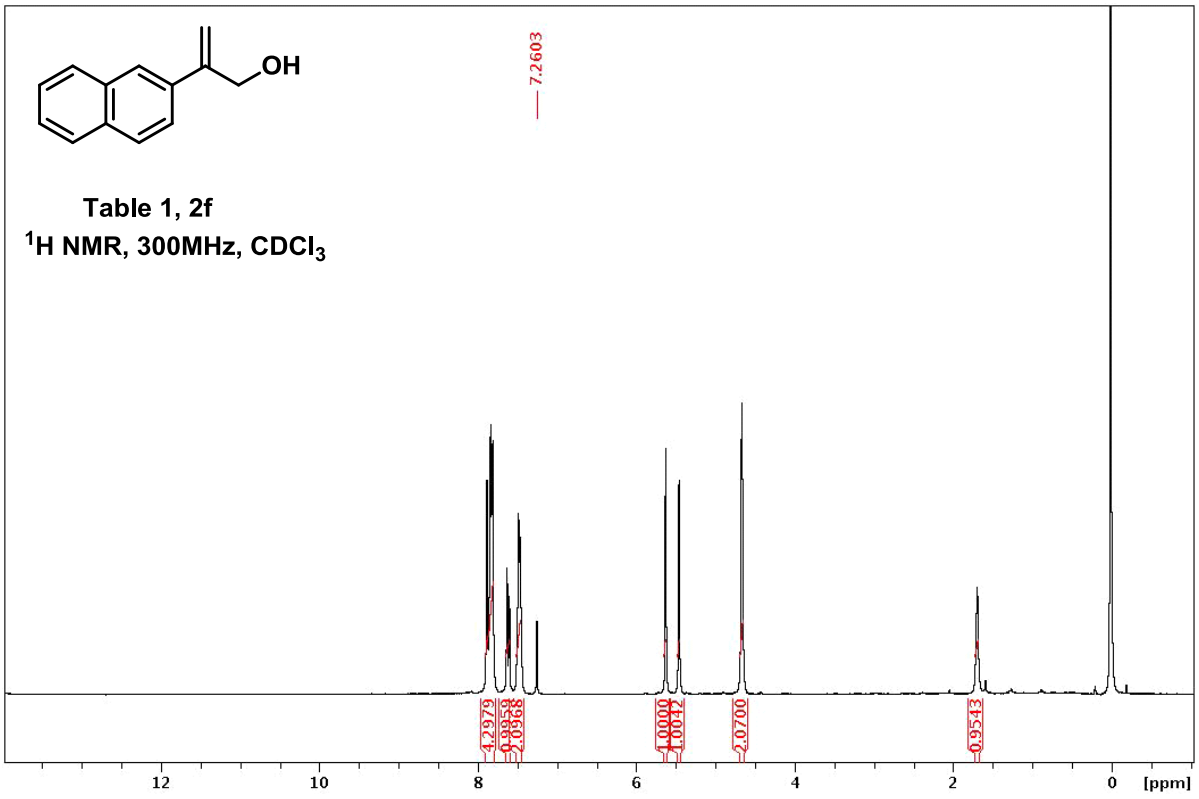


Table 1, 2f

^{1}H NMR, 300MHz, $CDCl_3$

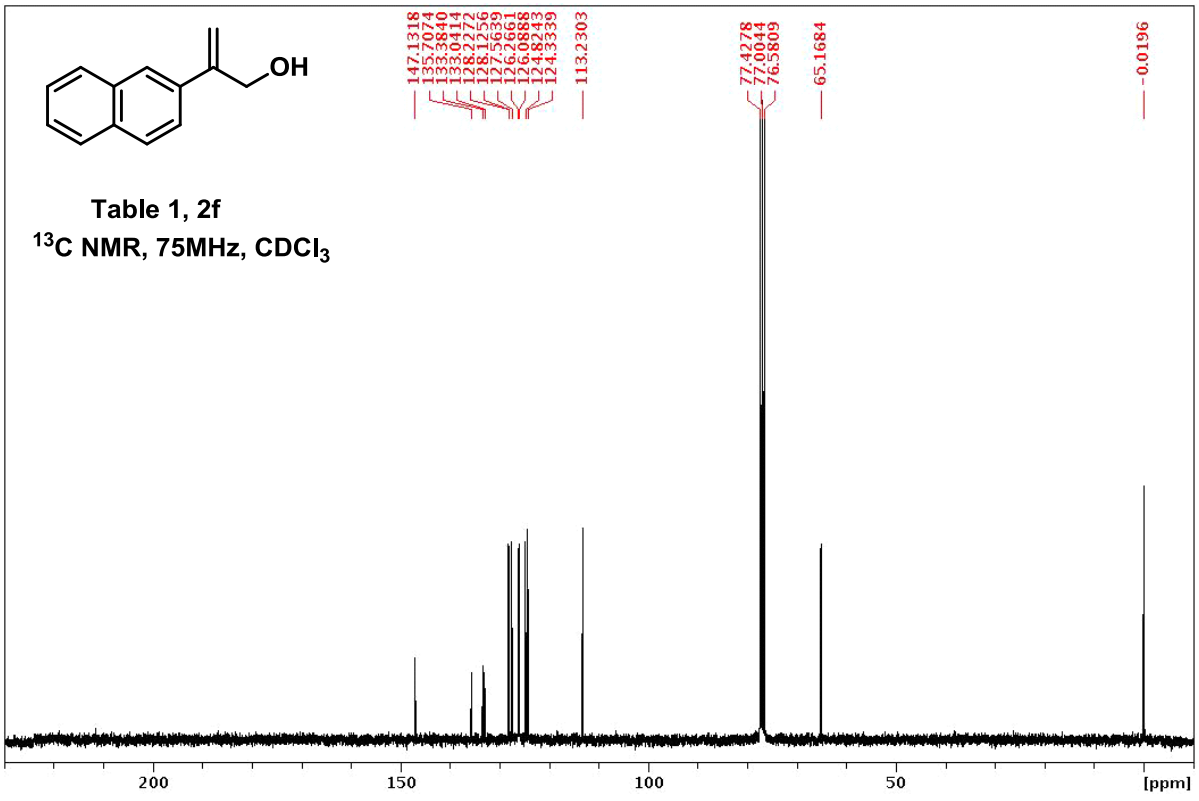


Table 1, 2f

^{13}C NMR, 75MHz, $CDCl_3$

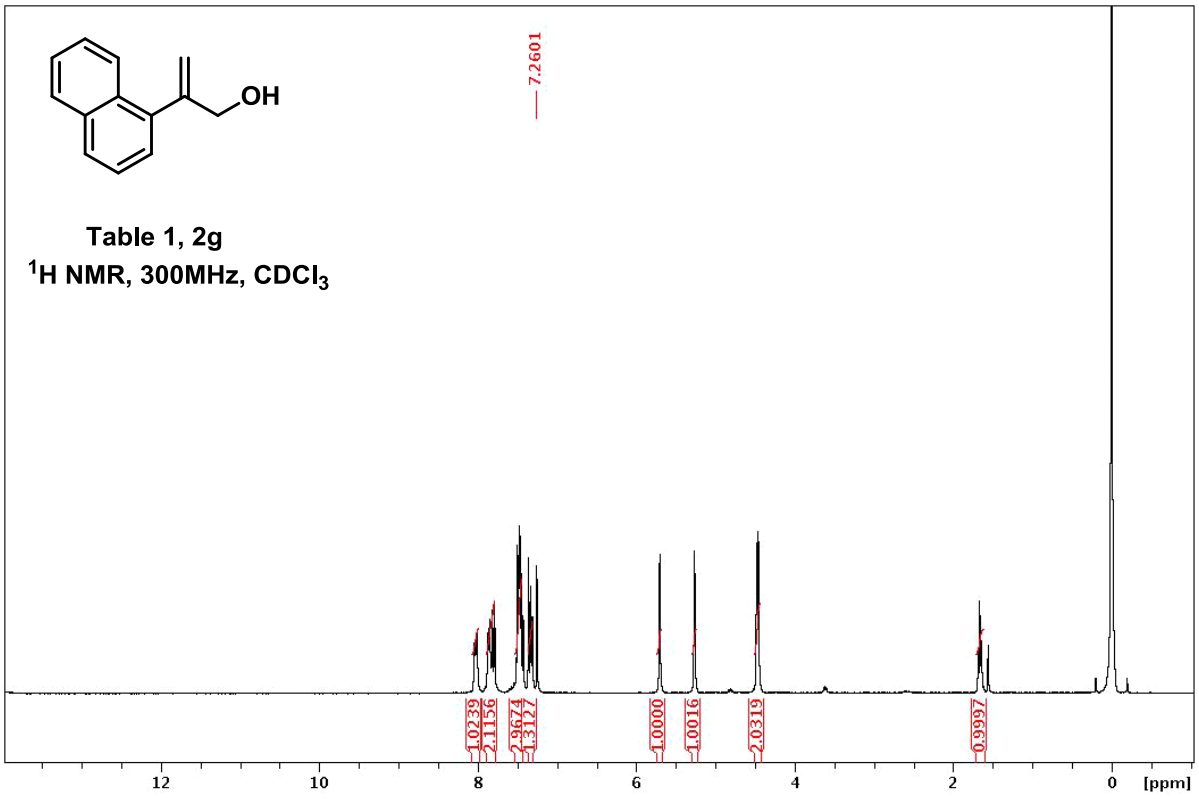


Table 1, 2g
^{1}H NMR, 300MHz, $CDCl_3$

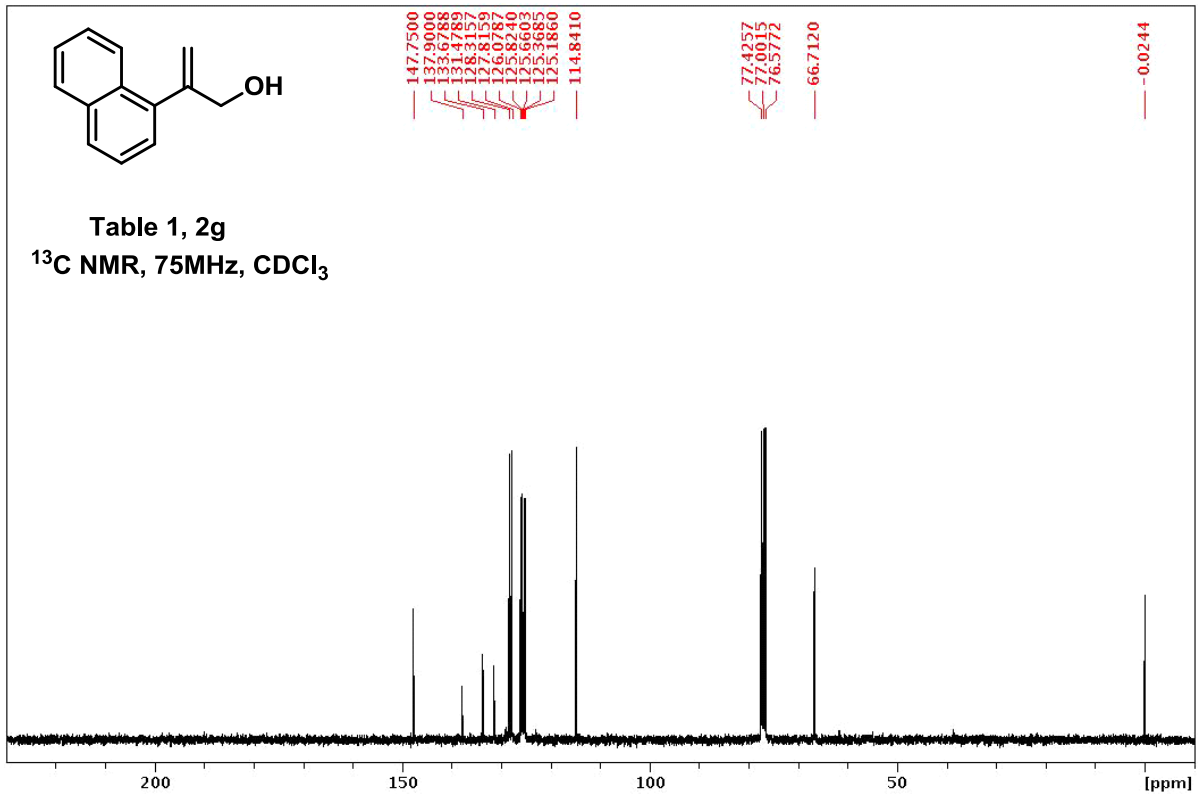
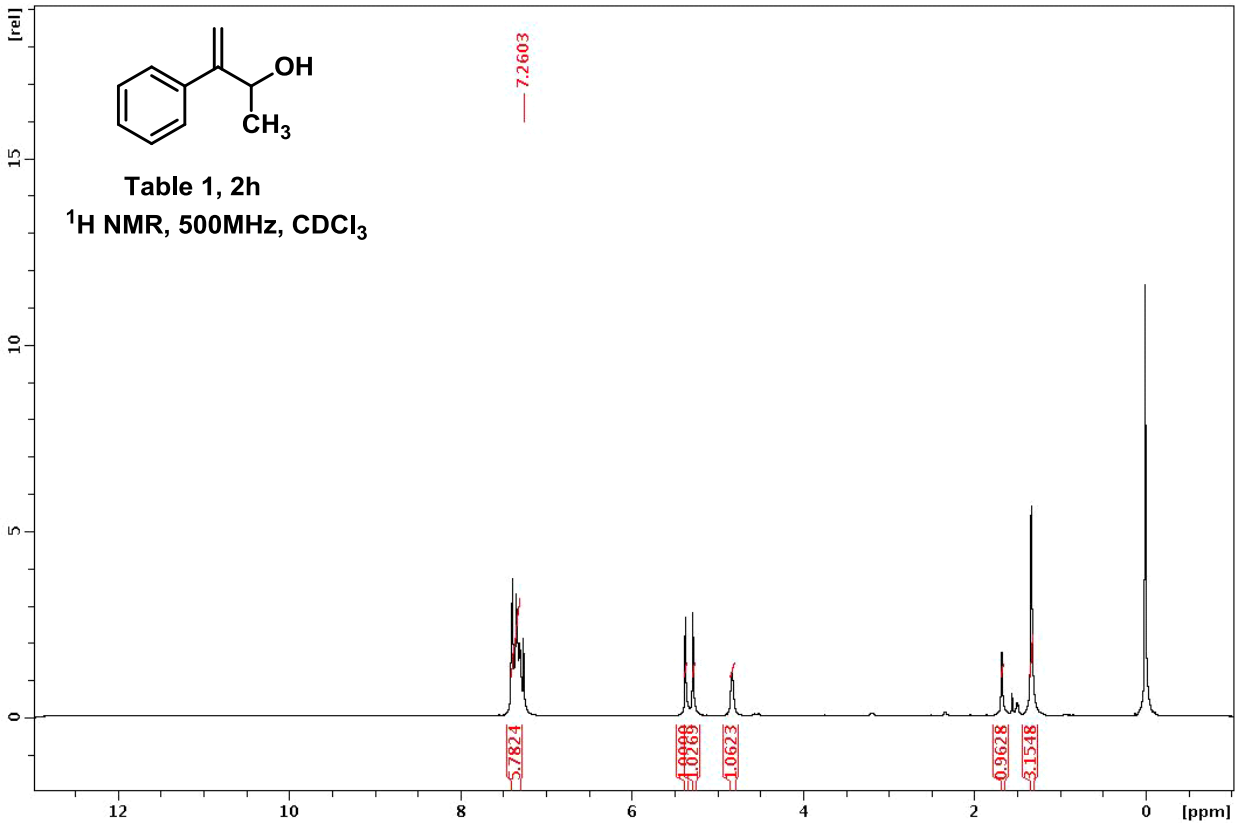
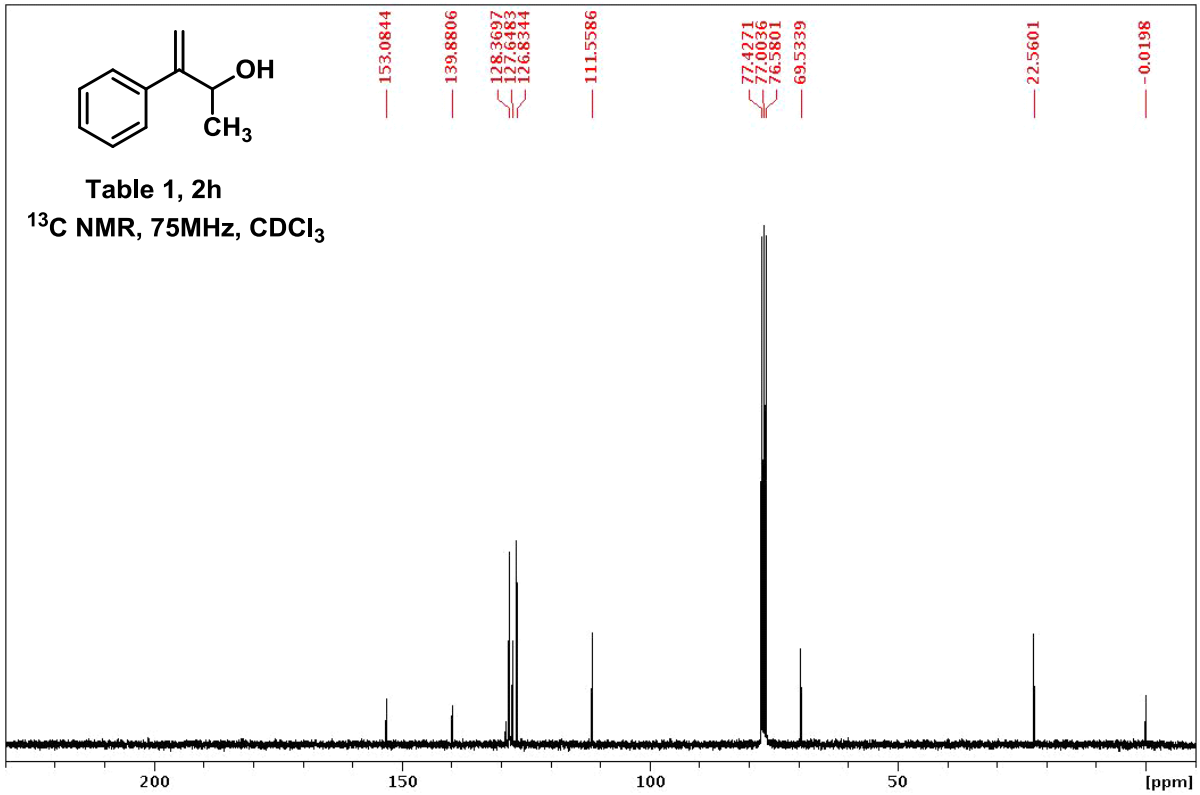


Table 1, 2g
^{13}C NMR, 75MHz, $CDCl_3$


OH
CH3
Table 1, 2h
1H NMR, 500MHz, CDCl3
7.2603
[rel]
15
10
5
0
5.7824
1.0000
1.0265
1.0623
0.9628
3.1548
12
10
8
6
4
2
0
[ppm]


OH
CH3
Table 1, 2h
13C NMR, 75MHz, CDCl3
153.0844
139.8806
128.3697
127.6483
126.8344
111.5586
77.4271
77.0636
76.5801
69.5339
22.5601
-0.0198
200
150
100
50
[ppm]

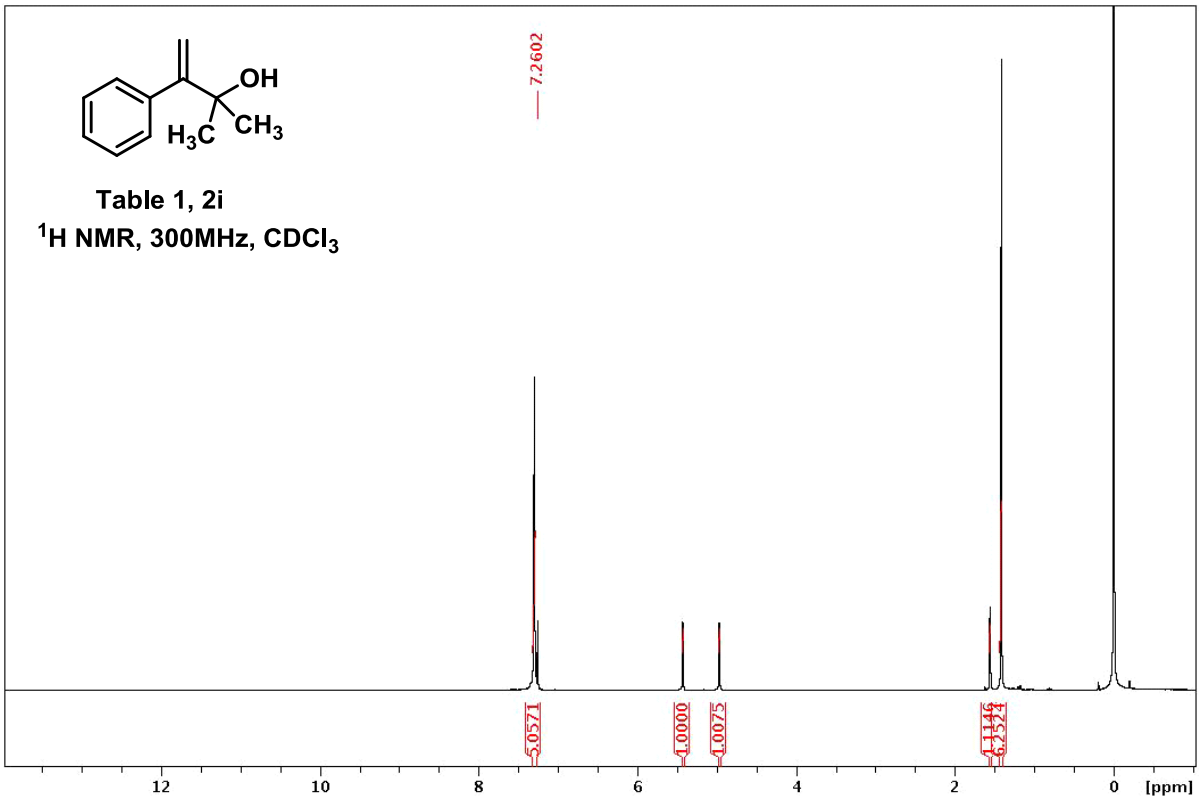


Table 1, 2i

^{1}H NMR, 300MHz, $CDCl_3$

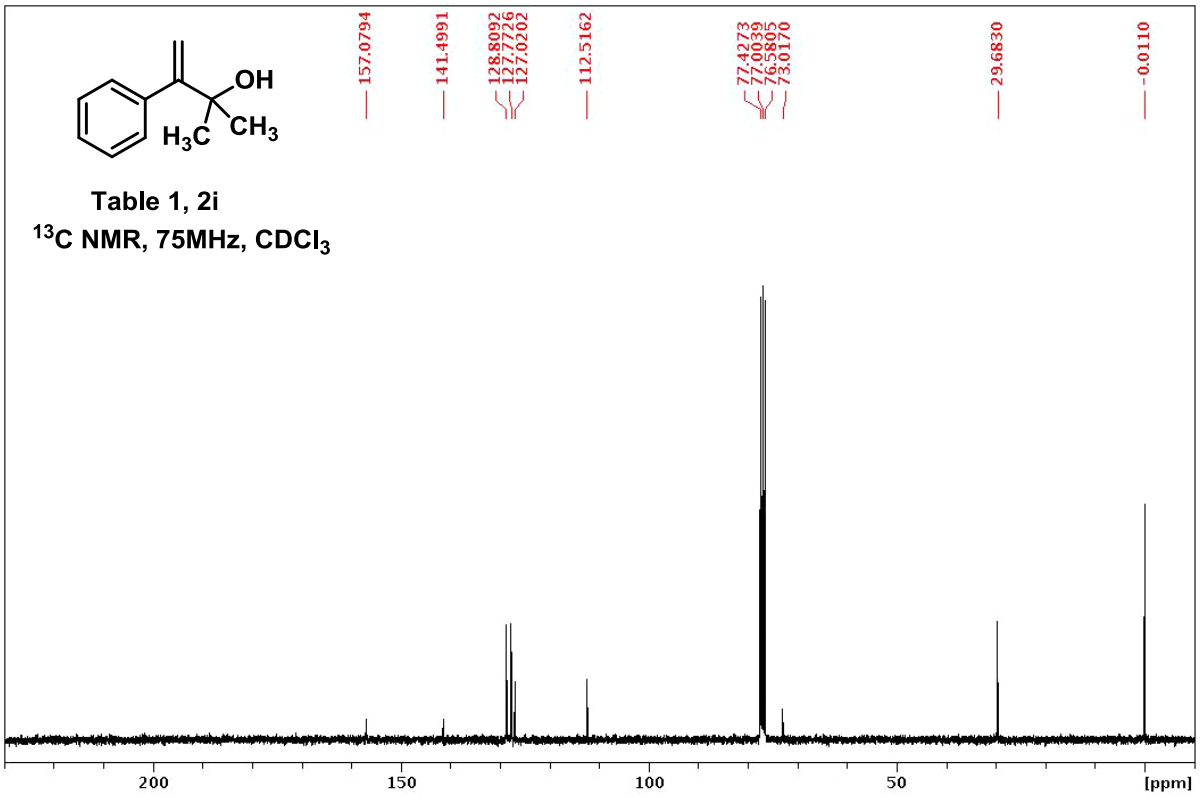


Table 1, 2i

^{13}C NMR, 75MHz, $CDCl_3$

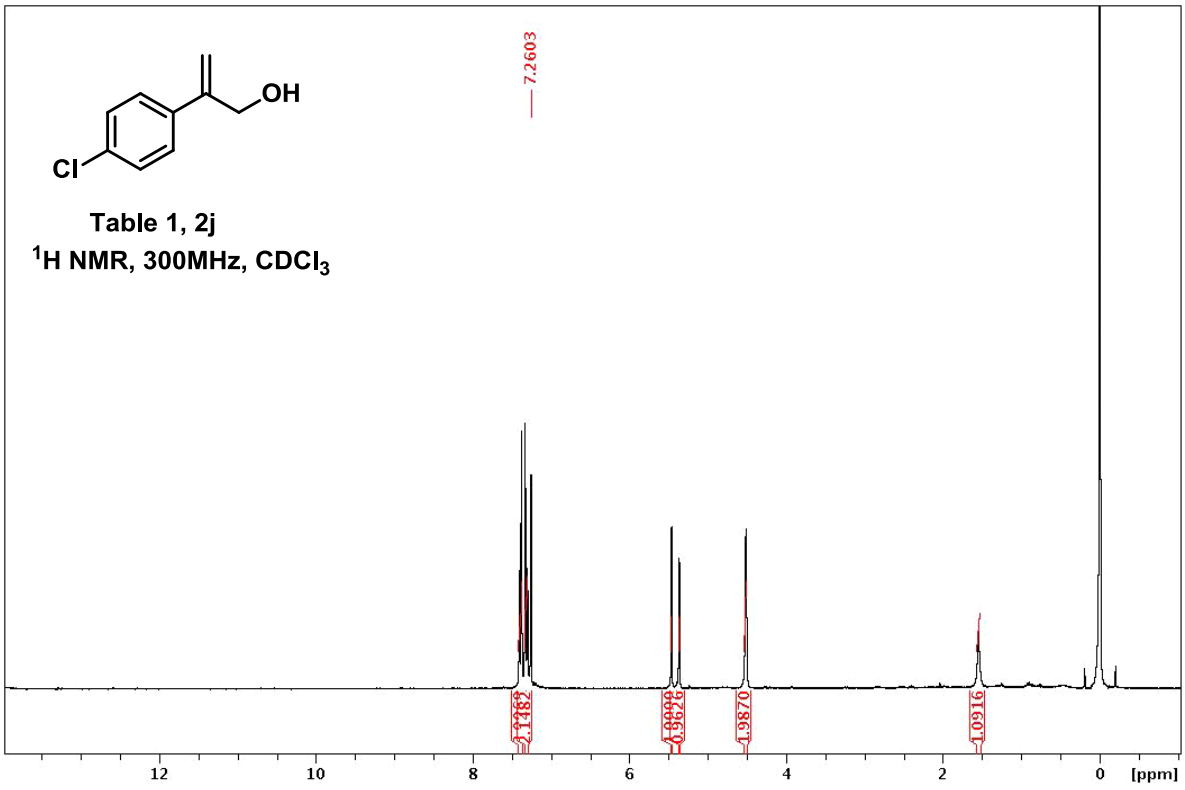


Table 1, 2j

^{1}H NMR, 300MHz, $CDCl_3$

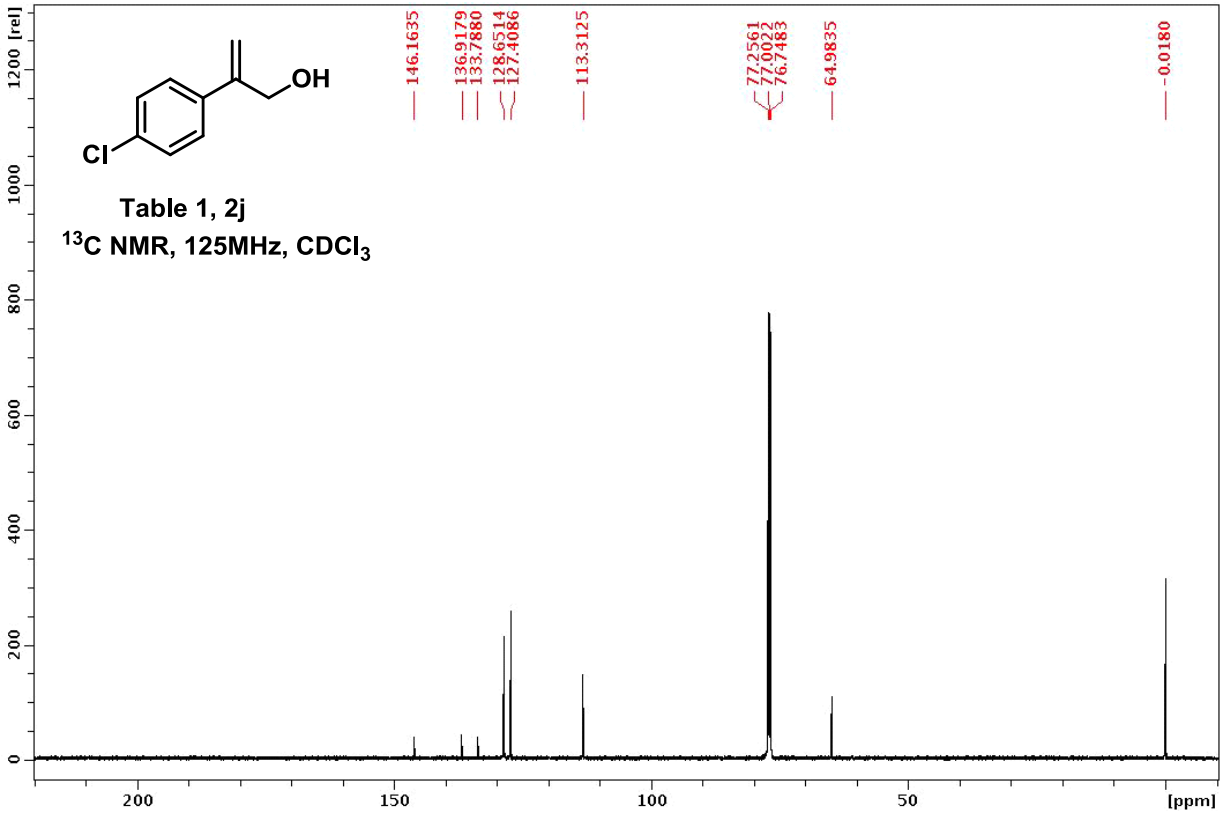


Table 1, 2j

^{13}C NMR, 125MHz, $CDCl_3$

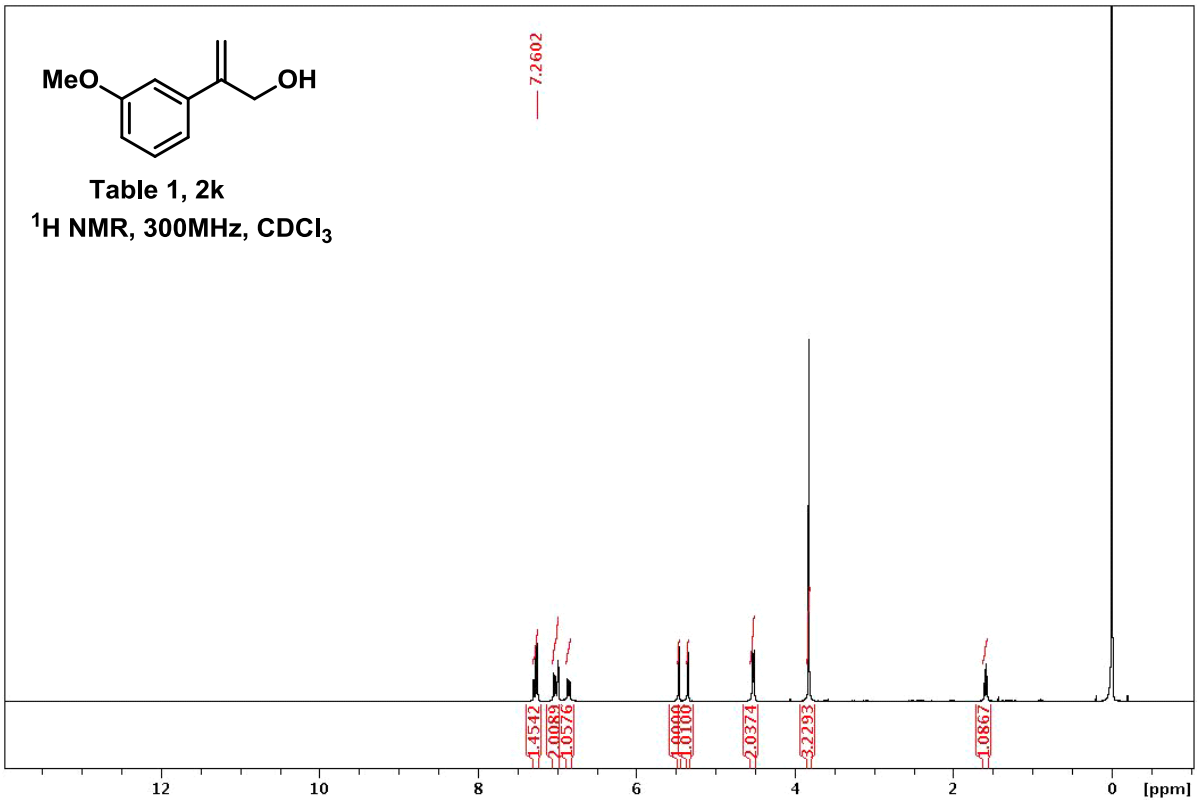


Table 1, 2k

^{1}H NMR, 300MHz, $CDCl_3$

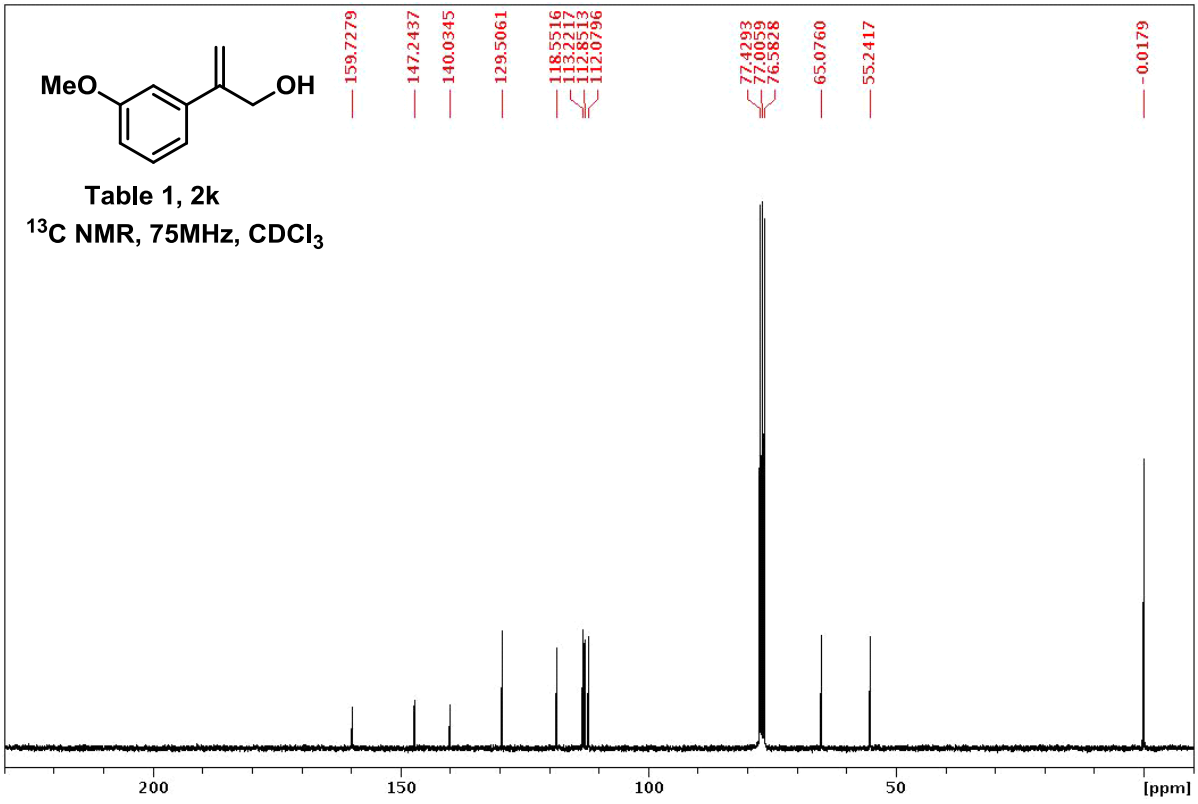


Table 1, 2k

^{13}C NMR, 75MHz, $CDCl_3$

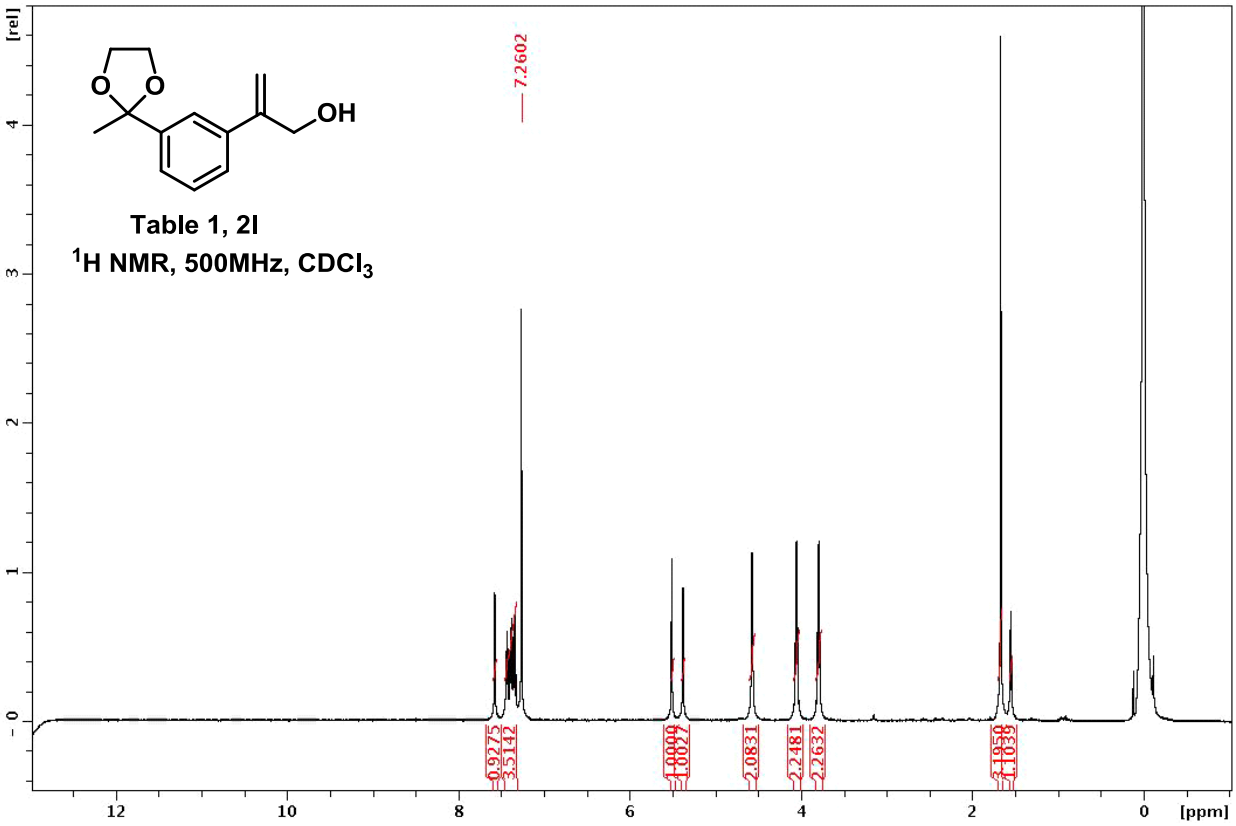


Table 1, 2l
^{1}H NMR, 500MHz, CDCl$_3$
7.2602
OH
[rel]
[ppm]

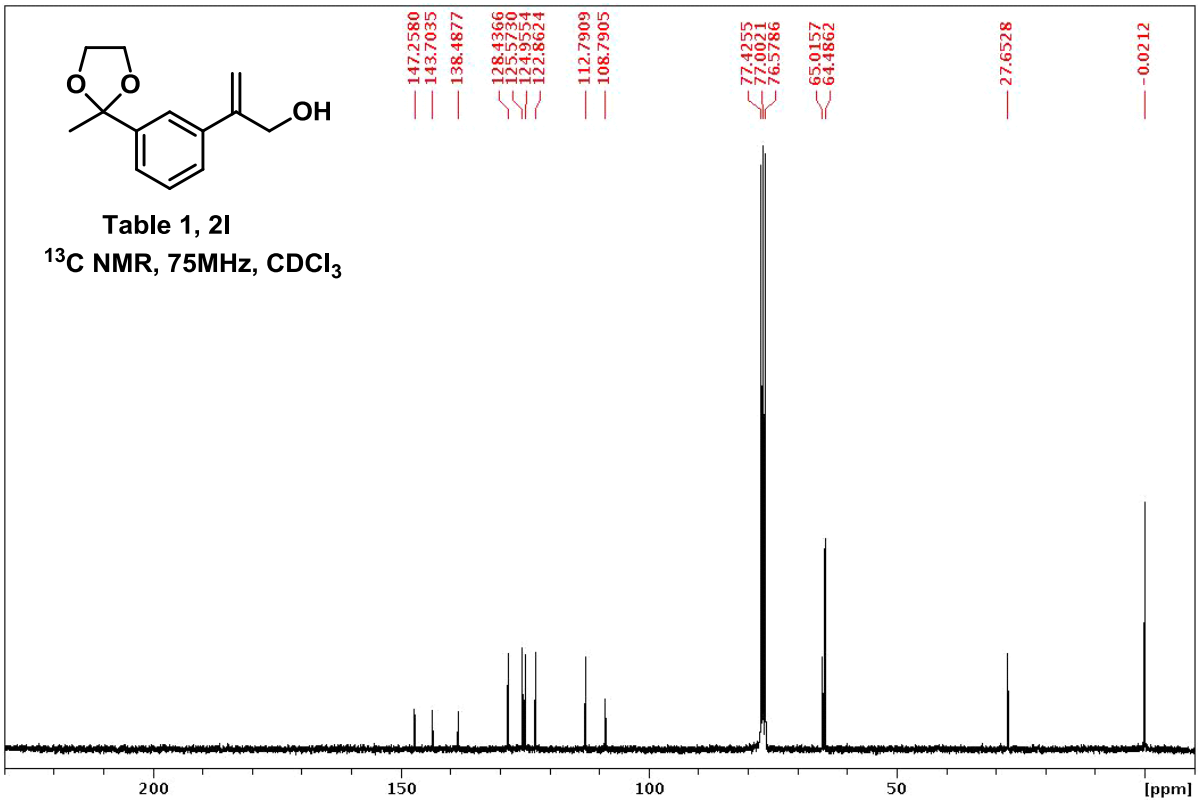
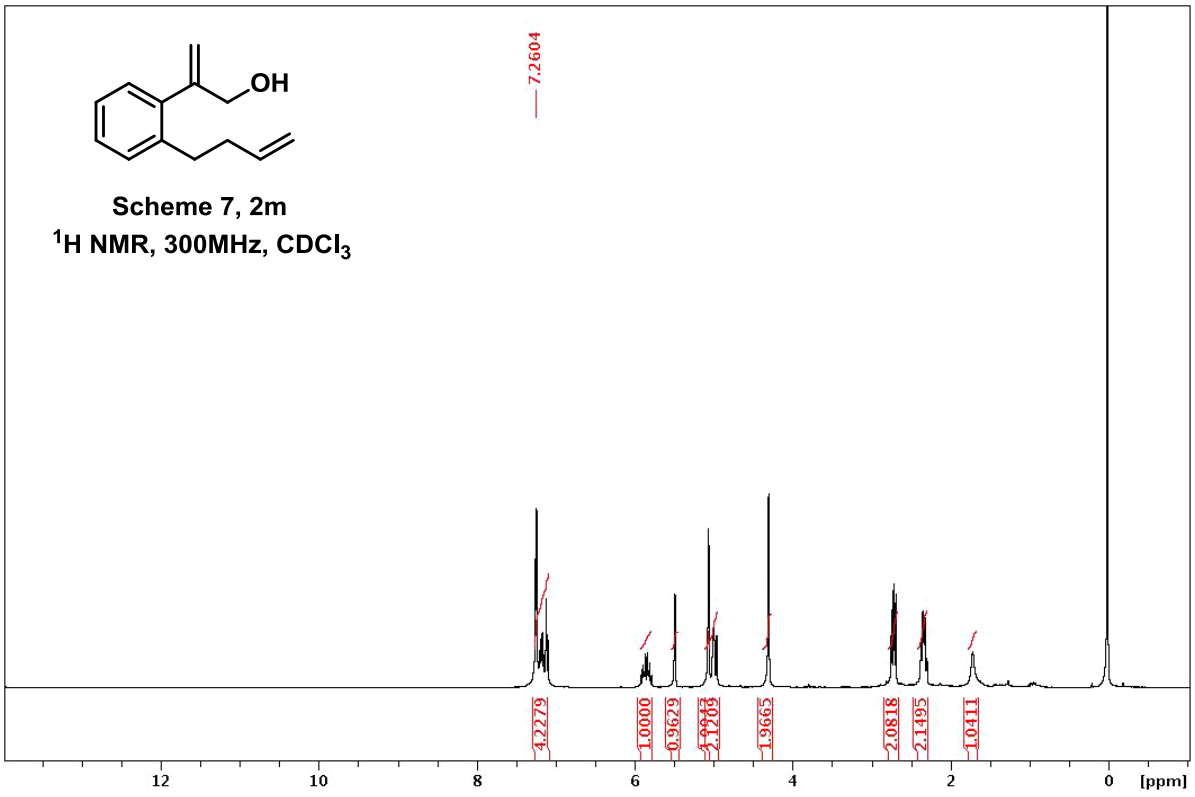
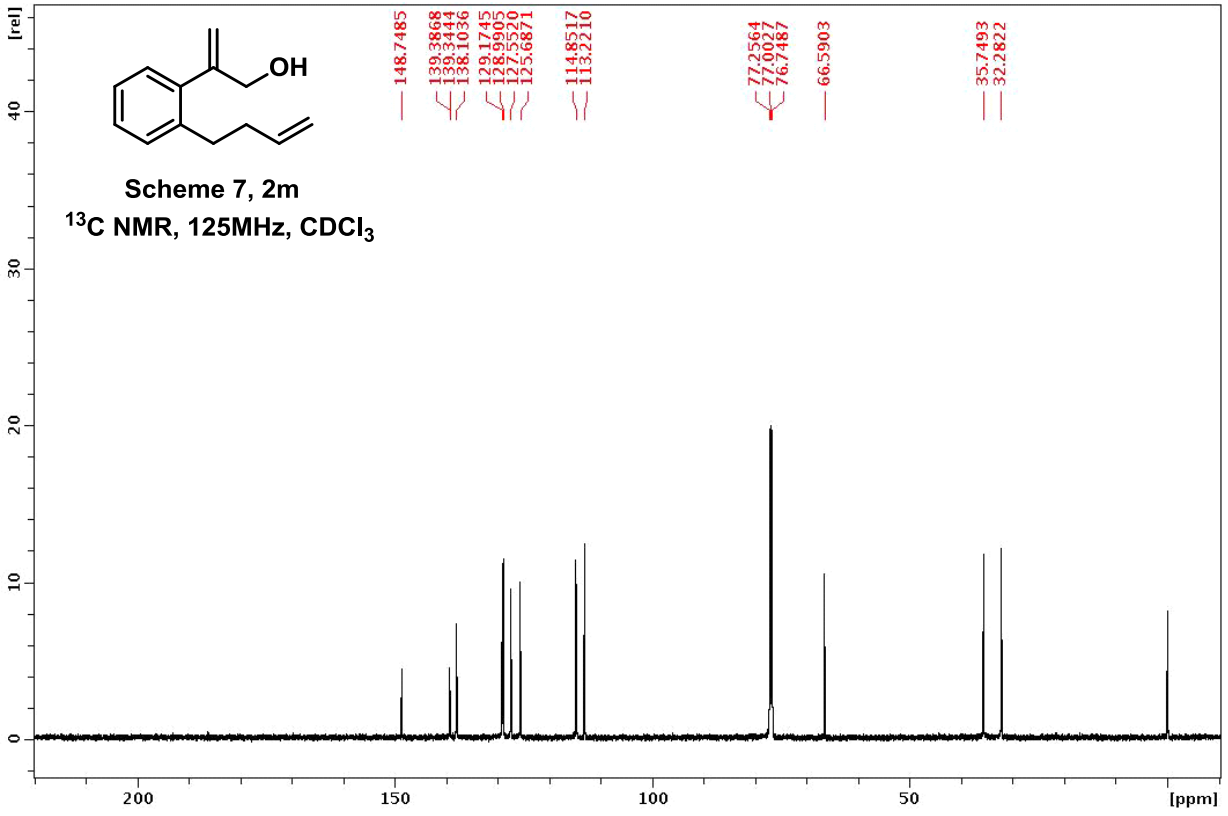


Table 1, 2l
^{13}C NMR, 75MHz, CDCl$_3$
147.2580
143.7035
138.4877
128.4366
125.3730
123.3554
122.8624
112.7909
108.7905
77.4255
77.0021
76.5786
65.0157
64.4862
27.6528
-0.0212
OH
[ppm]



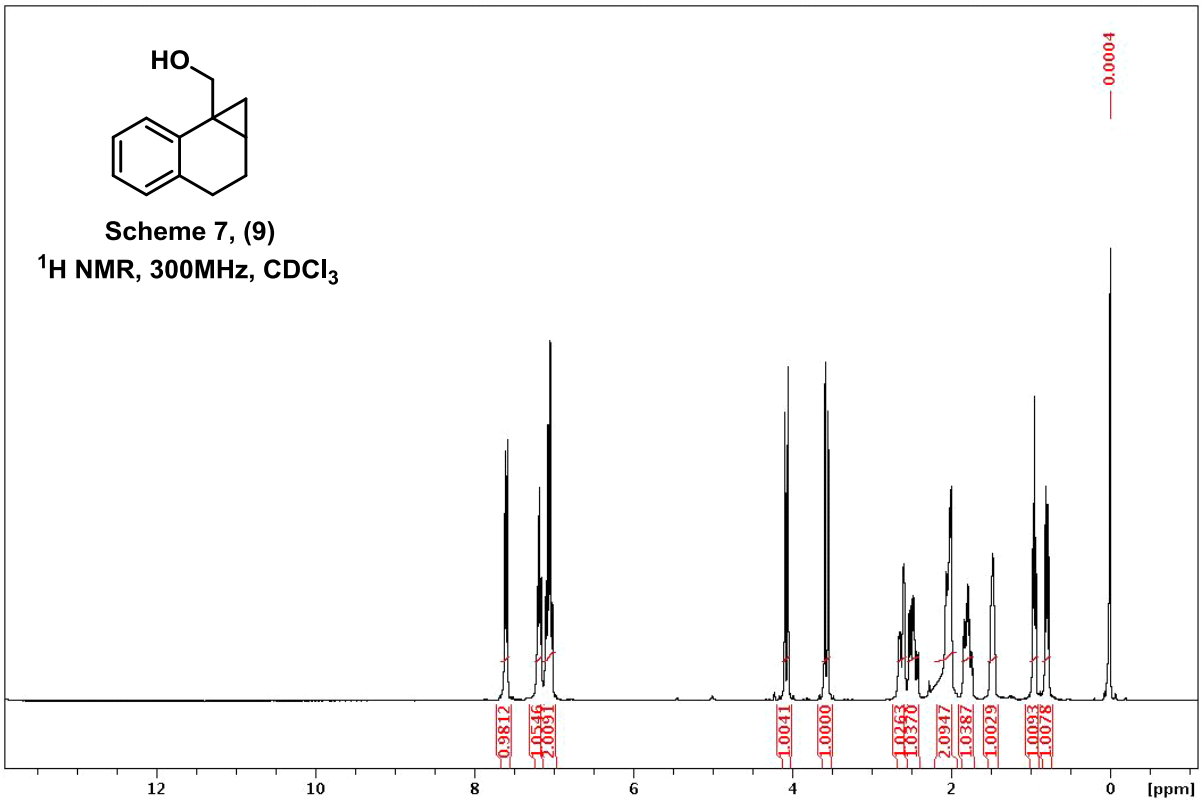
Scheme 7, 2m

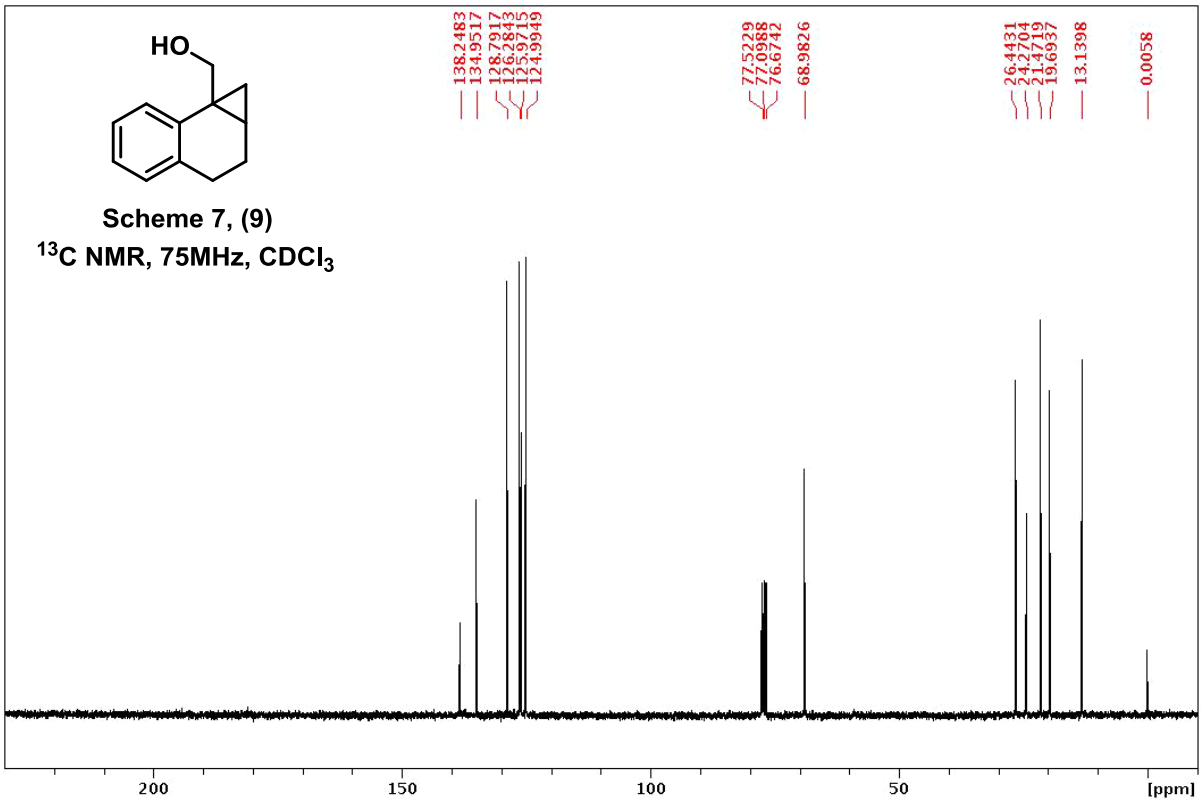
^{1}H NMR, 300MHz, $CDCl_3$

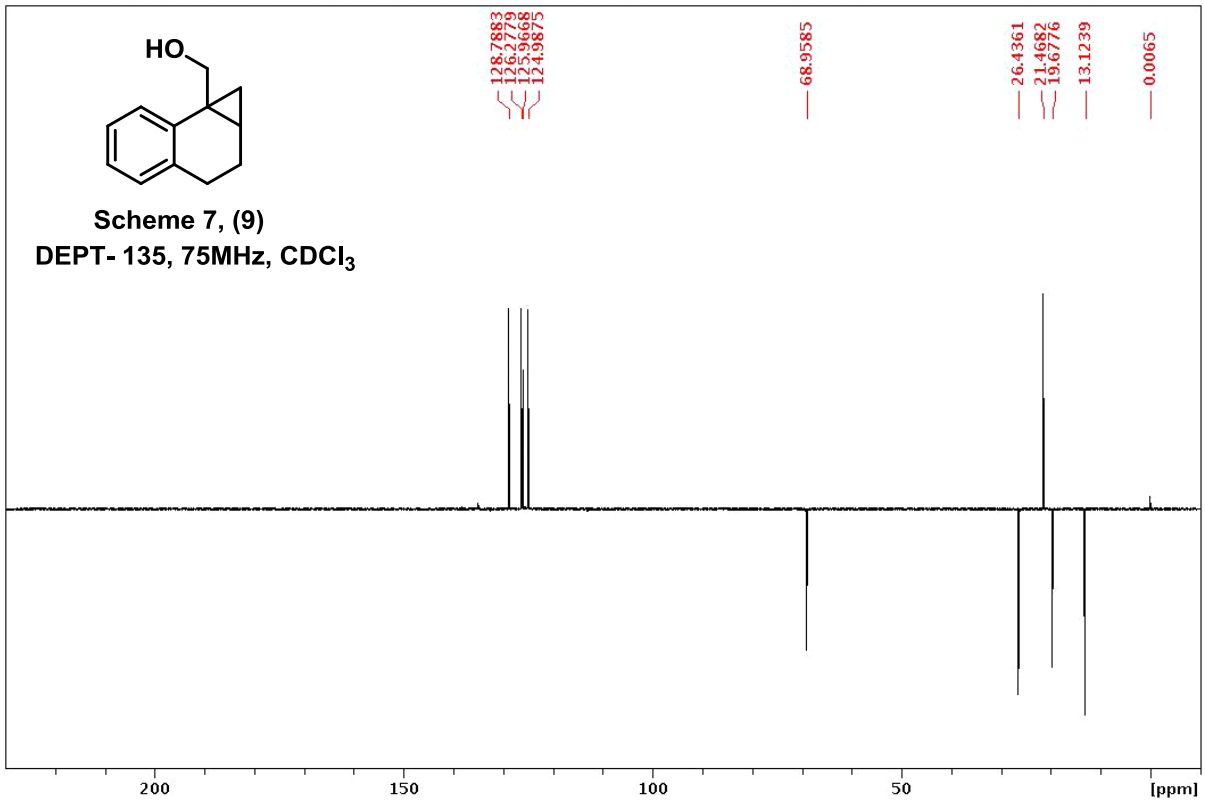


Scheme 7, 2m

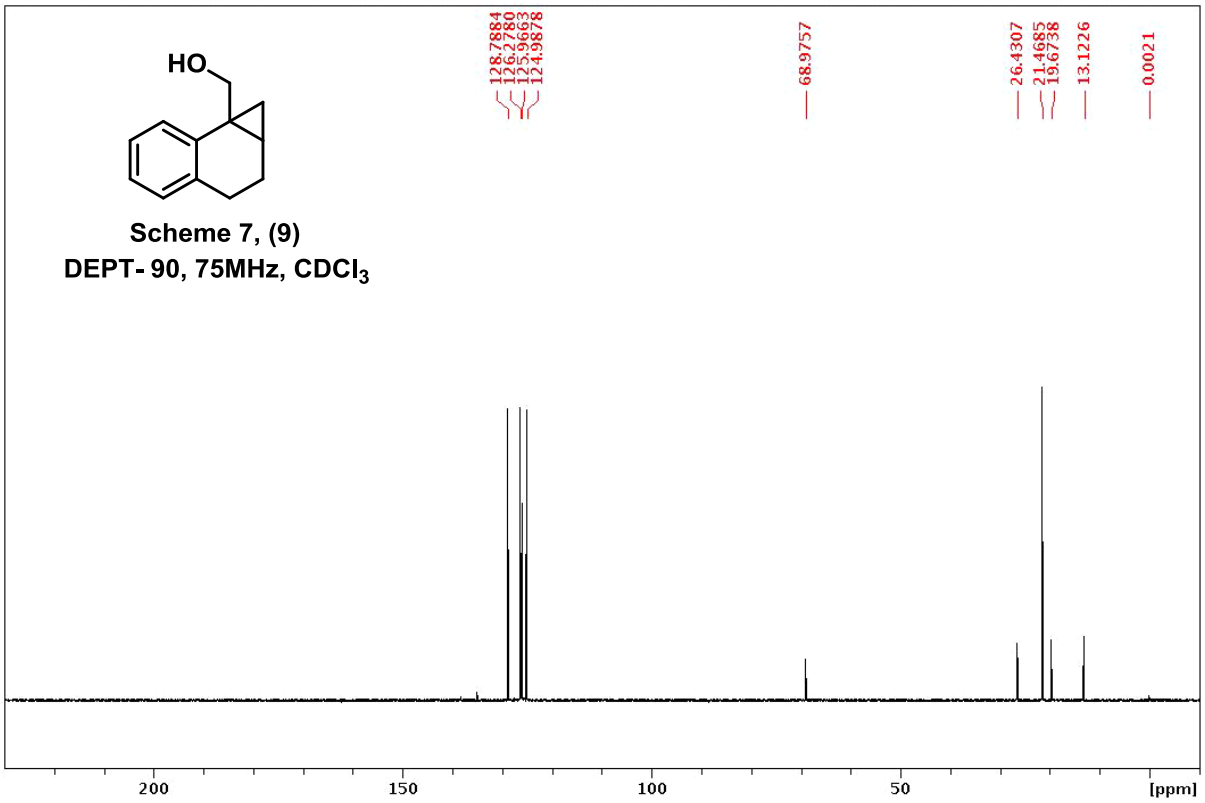
^{13}C NMR, 125MHz, $CDCl_3$


HO
Scheme 7, (9)
^{1}H NMR, 300MHz, CDCl$_3$
0.0004
0.9812
1.0546
2.0091
1.0041
1.0000
1.0263
1.0370
2.0947
1.0387
1.0029
1.0093
1.0078
12
10
8
6
4
2
0
[ppm]

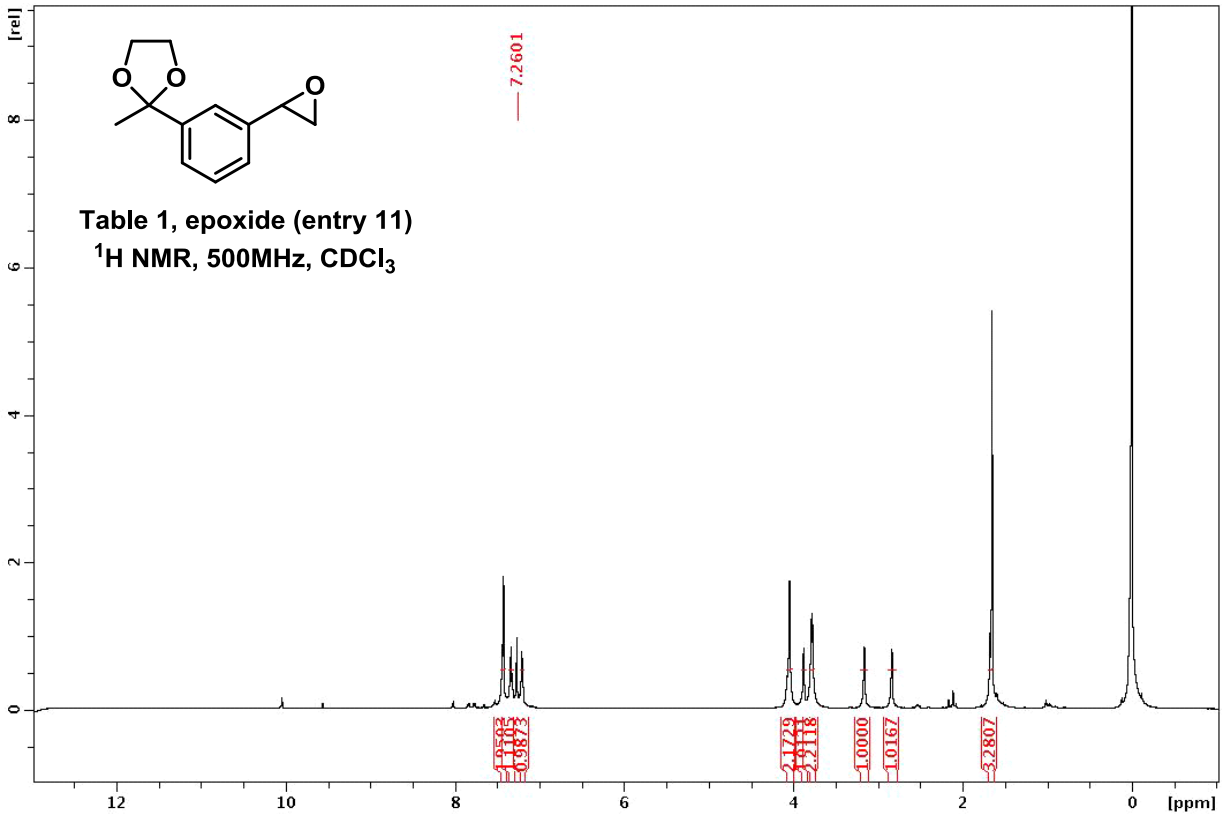

HO
Scheme 7, (9)
^{13}C NMR, 75MHz, CDCl$_3$
138.2483
134.9517
128.7917
126.2843
125.9715
124.9949
77.5229
77.0988
76.6742
68.9826
26.4431
24.2704
21.4719
19.6937
13.1398
0.0058
200
150
100
50
[ppm]

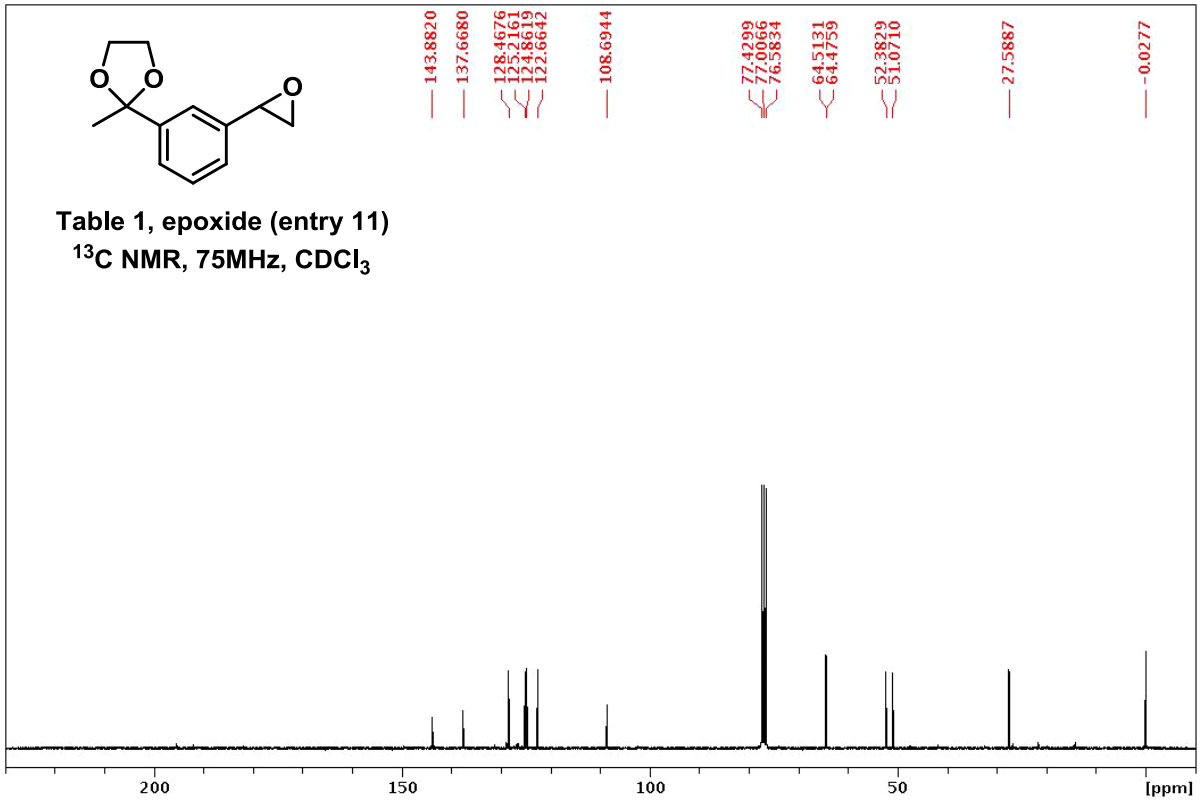


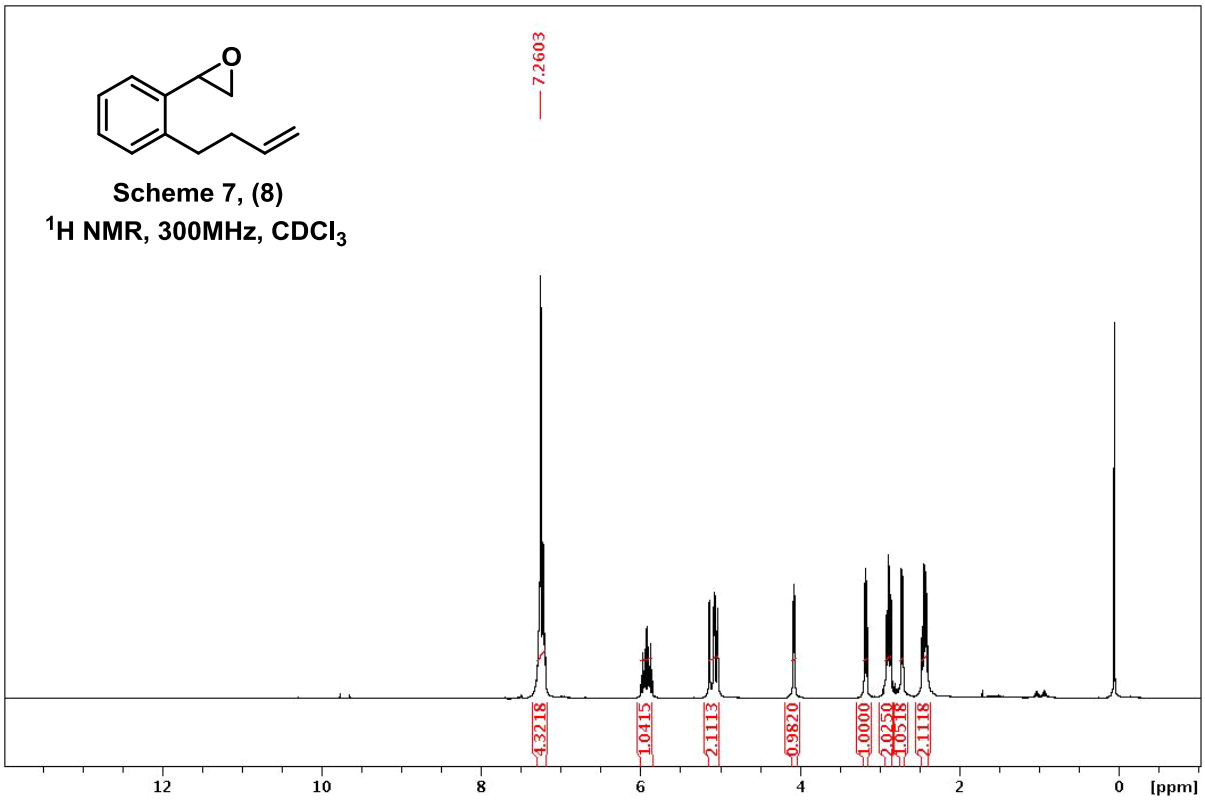
Scheme 7, (9)
DEPT- 135, 75MHz, $CDCl_3$

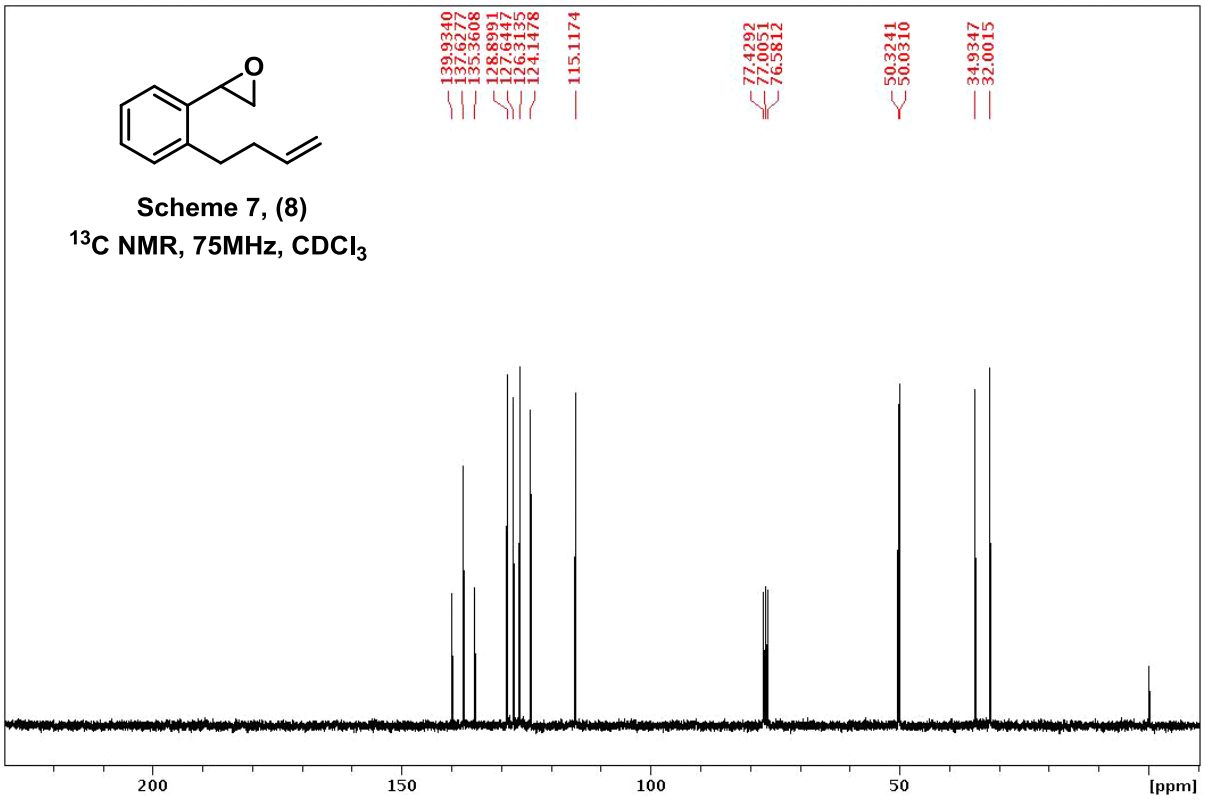


Scheme 7, (9)
DEPT- 90, 75MHz, $CDCl_3$


7.2601
Table 1, epoxide (entry 11)
1H NMR, 500MHz, CDCl3
[rel]
[ppm]
1.0000
1.0167
3.2807


143.8820
137.6680
128.4676
125.2161
124.8619
122.6642
108.6944
77.4299
77.0066
76.5834
64.5131
64.4759
52.3829
51.0710
27.5887
-0.0277
Table 1, epoxide (entry 11)
13C NMR, 75MHz, CDCl3
[ppm]


Scheme 7, (8)
1H NMR, 300MHz, CDCl3
7.2603
4.3218
1.0415
2.1113
0.9820
1.0000
2.0250
1.0518
2.1118
12
10
8
6
4
2
0
[ppm]


Scheme 7, (8)
13C NMR, 75MHz, CDCl3
139.9340
137.6277
135.3608
128.8991
127.6447
126.3135
124.1478
115.1174
77.4292
77.0051
76.5812
50.3241
50.0310
34.9347
32.0015
200
150
100
50
[ppm]