Supporting information for

Re_2O_7 Catalyzed Dienone-Phenol Rearrangement

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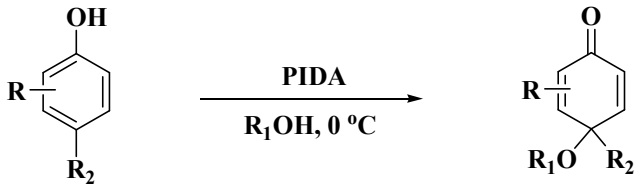
1. Experimental part

General Information. Unless otherwise specified, all reagents purchased from commercial suppliers were used as received. Non-aqueous reactions were conducted under an inert atmosphere of argon in flame-dried glassware. Anhydrous solvents were treated as followed: tetrahydrofuran, toluene and diethyl ether were distilled from benzophenone ketyl under nitrogen atmosphere, dimethylformamide was distilled over calcium hydride under reduced pressure, dichloromethane was distilled from calcium hydride under nitrogen atmosphere. Thin layer chromatography was conducted on Merck 60 F254 pre-coated silica gel plates. Column chromatography was carried out by normal silica gel (40-60 μm, 230-400 mesh, Silicycle P60). NMR data including ^{1}H NMR or ^{13}C NMR spectra were recorded on Mercury 300 and MR 400. ^{1}H NMR Chemical shifts were reported in ppm from the solvent resonance as the internal standard ($CDCl_3$:7.27 ppm). ^{13}C NMR chemical shifts were reported in ppm relative to the solvent ($CDCl_3$:77 ppm). Infrared spectra were performed on a Nicolet 380FT-IR and are reported in terms of frequency of absorption (cm^{-1}). Mass spectra were measured on a Shimadzu LCMS-2010EV mass spectrometer (EI). High resolution mass spectra were obtained from IonSpec 4.7 Tesla FTMS mass spectrometer (MALDI) and Bruker APEXIII 7.0 TESLA FTMS (EI).

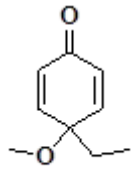
General procedure for the syntheses of substrates

Substrates **1aa-1af**, **1ca-aci** and **2g-2i** are synthesized according to related literature[1].



A solution of phenols (10 mmol, 1.0 equiv) in R_1OH (10 mL) is cooled to 0 °C and PIDA (12 mmol, 1.2 equiv) is added portionwise in 0.5 h and the reaction mixture is stirred for 2 h at 0 °C. The reaction mixture is poured into water and is extracted with ethyl acetate. The organic layers are combined, washed with cold 4 M NaOH and brine successively, dried with Na_2SO_4 and concentrated, and then purified by flash chromatography to afford target products.

Note：These substrates are unstable, even at -20 °C for a few days. Thus, it is advisable that using the substrate for the next step as soon as possible.

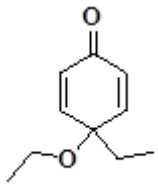


1aa

[1] a) S. Large, N. Roques, B. R. Langlois, *J. Org. Chem.* **2000**, *65*, 8848-8856. b) T. Dohi, T. Uchiyama, D. Yamashita, N. Washimi, Y. Kita, *Tetrahedron Letters*. **2011**, *52*, 2212-2215.

Following the general procedure, **1aa** was obtained as yellow solid (1.14 g, 75%), and it has been reported in the literature[2].

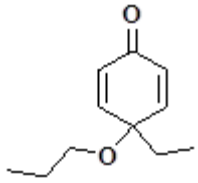
R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 2970, 2941, 2823, 1738, 1670, 1379, 1232, 1078 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.73-6.70 (m, 2H), 6.40-6.36 (m, 2H), 3.22 (s, 3H), 1.77 (q, *J* = 7.5 Hz, 2H), 0.84 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.5, 151.0, 131.7, 76.4, 53.2, 32.3, 7.8. HRMS-EI calcd. for $C_9H_{12}O_2$ (M^+) 152.0837. Found: $C_9H_{12}O_2$ 152.0838.



1ab

Following the general procedure, **1ab** was obtained as yellow oil (1.06 g, 64%).

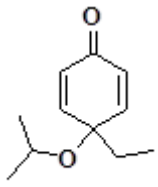
R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 2976, 2938, 2843, 1738, 1671, 1369, 1202, 1028 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.75-6.72 (m, 2H), 6.35-6.32 (m, 2H), 3.38 (q, *J* = 7.0 Hz, 2H), 1.77 (q, *J* = 7.5 Hz, 2H), 1.16 (t, *J* = 7.0 Hz, 3H), 0.82 (t, *J* = 7.5 Hz, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 185.7, 151.6, 131.1, 76.1, 61.0, 32.4, 16.0, 7.8. HRMS-EI calcd. for $C_{10}H_{14}O_2$ (M^+) 166.0994. Found: $C_{10}H_{14}O_2$ 166.0993.



1ac

Following the general procedure, **1ac** was obtained as yellow viscous solid (930 mg, 52%).

R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 2960, 2951, 2813, 1738, 1671, 1359, 1232, 1088 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.74-6.72 (m, 2H), 6.36-6.32 (m, 2H), 3.28 (t, *J* = 6.5 Hz, 2H), 1.77 (q, *J* = 7.5 Hz, 2H), 1.58-1.52 (m, 2H), 0.90 (t, *J* = 6.5 Hz, 3H), 0.84 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.7, 151.8, 131.1, 75.9, 67.1, 32.4, 23.6, 10.5, 7.8. HRMS-EI calcd. for $C_{11}H_{16}O_2$ (M^+) 180.1150. Found: $C_{11}H_{16}O_2$ 180.1152.

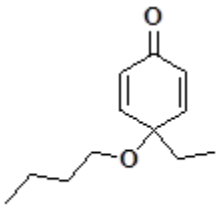


1ad

Following the general procedure, **1ad** was obtained as yellow oil (690 mg, 38%).

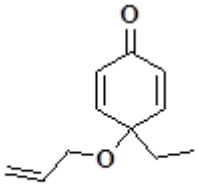
R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 2976, 2940, 2833, 1738, 1670, 1369, 1212, 1068 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.79-6.75 (m, 2H), 6.32-6.29 (m, 2H), 3.57 (hept, *J* = 6.0 Hz, 1H), 1.73 (q, *J* = 7.5 Hz, 2H), 1.11 (d, *J* = 6.0 Hz, 6H), 0.82 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.9, 151.9, 130.4, 76.3, 68.1, 32.7, 24.8, 7.8. HRMS-EI calcd. for $C_{11}H_{16}O_2$ (M^+) 180.1150. Found: $C_{11}H_{16}O_2$ 180.1151.

[2] M. P. Capparelli, R. E. DeSchepper, J. S.; Swenton, *J. Org. Chem.* **1987**, *52*, 4953-4961.



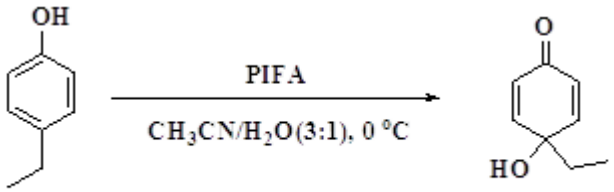
1ae

Following the general procedure, **1ae** was obtained as yellow oil (790 mg, 40%).
R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 2978, 2938, 2830, 1738, 1670, 1379, 1202, 1058 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.75-6.71 (m, 2H), 6.35-6.313 (m, 2H), 3.31 (t, *J* = 6.5 Hz, 2H), 1.75 (q, *J* = 7.5 Hz, 2H), 1.50 (tt, *J* = 12.0, 6.5 Hz, 2H), 1.36-1.33 (m, 2H), 0.89 (t, *J* = 7.5 Hz, 3H), 0.83 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.7, 151.8, 131.1, 75.8, 65.2, 32.5, 32.4, 19.2, 13.8, 7.8. HRMS-EI calcd. for $C_{12}H_{18}O_2$ (M^+) 194.1307. Found: $C_{12}H_{18}O_2$ 194.1303.



1af

Following the general procedure, **1af** was obtained as yellow oil (660 mg, 37%).
R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 2970, 2945, 2843, 1738, 1673, 1359, 1202, 1078 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.75-6.72 (m, 2H), 6.35-6.32 (m, 2H), 5.89-5.82 (m, 1H), 5.24 (dq, *J* = 17.0, 1.5 Hz, 1H), 5.13 (dq, *J* = 17.0, 1.5 Hz, 1H), 3.85 (dt, *J* = 5.5, 1.5 Hz, 2H), 1.80 (q, *J* = 7.5 Hz, 2H), 0.83 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.5, 151.0, 134.8, 131.3, 116.7, 76.2, 66.5, 32.3, 7.8. HRMS-EI calcd. for $C_{11}H_{14}O_2$ (M^+) 178.0994. Found: $C_{11}H_{14}O_2$ 178.0996.



1ag

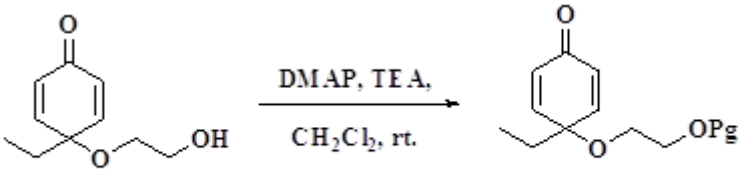
Substrate **1ag** is synthesized according to related literature[3].

A solution of 4-Ethylphenol (10 mmol, 1.0 equiv) in acetonitrile-water (3: 1; 40 mL) is cooled to 0 °C and PIFA (12 mmol, 1.2 equiv) is added portionwise in 0.5 h and the reaction mixture is stirred for 2 h at 0 °C. The reaction mixture is poured into water and is extracted with ethyl acetate. The organic layers are combined, washed with cold 4 M NaOH and brine successively, dried with Na_2SO_4 and concentrated, and then purified by flash chromatography (petroleum ether / ethyl acetate = 4:1, V/V) to afford the target product as yellow solid (810 mg, 58%).

R*f* = 0.5 (petroleum ether / ethyl acetate = 1:1, V/V); IR (film): 3385, 2970, 2941, 2853, 1738, 1672, 1359, 1232, 1088 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.81-6.78 (m, 2H), 6.17-6.13 (m, 2H), 2.92 (s,

[3] M. Alexande, M. Lee, T. J. K. Richard, *J. Chem. Soc. Perkin Trans,* **1994**, *1*, 2047-2048.

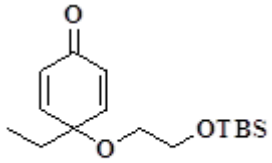
1H), 1.78 (q, *J* = 7.5 Hz, 2H), 0.84 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 186.0, 151.6, 128.3, 70.4, 32.7, 7.9. HRMS-EI calcd. for $C_8H_{10}O_2$ (M^+) 138.0681. Found: $C_8H_{10}O_2$ 138.0679.



1b-S 1b

Substrates **1ba-1bf** were synthesized from **1b-S** using common methods for protection of hydroxyl. And **1b-S** was obtained following the general procedure from 4-ethylphenol (70 mmol) as yellow oil (5.6 g, 44%).[4]

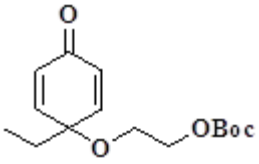
R*f* = 0.5 (petroleum ether / ethyl acetate = 1:2, V/V); IR (film): 3450, 2970, 2937, 1671, 1630, 1098, 1067 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.81-6.71 (m, 2H), 6.36-6.26 (m, 2H), 3.69 (t, *J* = 5.0 Hz, 2H), 3.44 (t, *J* = 5.0 Hz, 2H), 2.13 (s, 1H), 1.79 (q, *J* = 7.5 Hz, 2H), 0.83 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.4, 150.8, 131.4, 76.1, 66.5, 62.1, 32.2, 7.8. HRMS-EI calcd. for $C_{10}H_{14}O_3$ (M^+) 182.0943. Found: $C_{10}H_{14}O_3$ 182.0942.



1ba

1ba was obtained from **1b-S** (2.5 mmol) as colorless oil (695 mg, 94%) .

R*f* = 0.5 (petroleum ether / ethyl acetate = 10:1, V/V); IR (film): 2955, 2930, 2858, 1672, 1254, 1087 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.75-6.72 (m, 2H), 6.35-6.32 (m, 2H), 3.70 (t, *J* = 5.0 Hz, 2H), 3.39 (t, *J* = 5.0 Hz, 2H), 1.77 (q, *J* = 7.5 Hz, 2H), 0.89 (s, 9H), 0.83 (t, *J* = 7.5 Hz, 3H), 0.06 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.6, 151.3, 131.2, 76.1, 67.0, 62.8, 32.3, 25.9, 18.4, 7.8, -5.2. HRMS-EI calcd. for $C_{16}H_{28}O_3Si$ (M^+) 296.1808. Found: $C_{16}H_{28}O_3Si$ 296.1810.

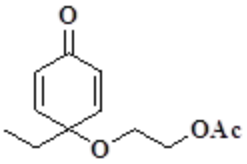


1bb

1bb was obtained from **1b-S** (2.5 mmol) as colorless oil (670 mg, 95%) .

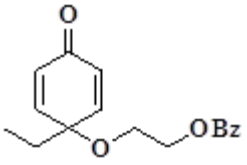
R*f* = 0.5 (petroleum ether / ethyl acetate = 8:1, V/V); IR (film): 2972, 2936, 2843, 1738, 1640, 1355, 1234, 1067 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.74-6.71 (m, 2H), 6.35-6.32 (m, 2H), 4.14 (t, *J* = 5.0 Hz, 2H), 3.52 (t, *J* = 5.0 Hz, 2H), 1.79 (q, *J* = 7.5 Hz, 2H), 1.47 (s, 9H), 0.82 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.3, 153.4, 150.6, 131.5, 82.2, 76.3, 66.1, 63.4, 32.2, 27.7, 7.8. HRMS-EI calcd. for $C_{15}H_{22}O_5$ (M^+) 282.1467. Found: $C_{15}H_{22}O_5$ 282.1465.

[4] Q. Gu, Z.-Q. C. Zheng, S.-L. You, *J. Am. Chem. So.c*, **2010**, *132*,4 056 – 4057.



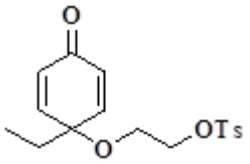
1bd

1bd was obtained from **1b-S** (2.5 mmol) as colorless oil (520 mg, 93%) .
R*f* = 0.5 (petroleum ether / ethyl acetate = 7:1, V/V); IR (film): 2971, 2939, 2838, 1741, 1633, 1377, 1235, 1097 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72-6.69 (m, 2H), 6.34-6.31 (m, 2H), 4.13 (t, *J* = 5.0 Hz, 2H), 3.50 (t, *J* = 5.0 Hz, 2H), 2.04 (s, 3H), 1.76 (q, *J* = 7.5 Hz, 2H), 0.80 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.3, 170.8, 150.6, 131.5, 76.3, 63.7, 63.4, 32.2, 20.9, 7.7. HRMS-EI calcd. for $C_{12}H_{16}O_4$ (M^+) 166.0994. Found: $C_{12}H_{16}O_4$ 166.0998.



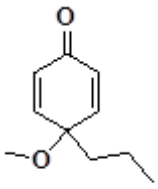
1be

1be was obtained from **1b-S** (2.5 mmol) as colorless oil (635 mg, 89%) .
R*f* = 0.5 (petroleum ether / ethyl acetate = 5:1, V/V); IR (film): 2974, 2930, 2858, 1737, 1630, 1350, 1225, 1047 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05 (d, *J* = 7.0 Hz, 2H), 7.58 (t, *J* = 7.0 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 2H), 6.76-6.74 (m, 2H), 6.37-6.35 (m, 2H), 4.42 (t, *J* = 5.0 Hz, 2H), 3.67 (t, *J* = 5.0 Hz, 2H), 1.80 (q, *J* = 7.5 Hz, 2H), 0.84 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.3, 166.4, 150.6, 133.1, 131.5, 130.0, 129.6, 128.4, 76.3, 64.1, 63.5, 32.3, 7.8. HRMS-EI calcd. for $C_{17}H_{18}O_4$ (M^+) 286.1205. Found: $C_{17}H_{18}O_4$ 286.1206.



1bf

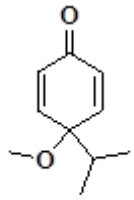
1bf was obtained from **1b-S** (2.5 mmol) as colorless oil (770 mg, 92%) .
R*f* = 0.5 (petroleum ether / ethyl acetate = 5:2, V/V); IR (film): 2955, 2930, 2855, 1670, 1357, 1177, 1096 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 6.64-6.61 (m, 2H), 6.30-6.28 (m, 2H), 4.09 (t, *J* = 5.0 Hz, 2H), 3.47 (t, *J* = 5.0 Hz, 2H), 2.43 (s, 3H), 1.68 (q, *J* = 7.5 Hz, 2H), 0.77 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.1, 150.2, 144.9, 133.1, 131.5, 129.8, 127.9, 76.2, 69.3, 63.0, 32.1, 21.6, 7.7. HRMS-EI calcd. for $C_{17}H_{20}O_5S$ (M^+) 336.1031. Found: $C_{17}H_{20}O_5S$ 336.1030.



1ca

Following the general procedure, **1ca** was obtained as viscous yellow solid (740 mg, 44%).

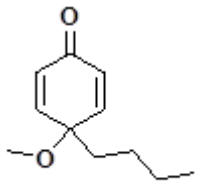
R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 2973, 2942, 2830, 1738, 1671, 1359, 1222, 1069 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.73-6.70 (m, 2H), 6.35-6.32 (m, 2H), 3.19 (s, 3H), 1.70-1.66 (m, 2H), 1.29-1.22 (m, 2H), 0.87 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.5, 151.2, 131.4, 75.8, 53.0, 41.6, 16.9, 14.2. HRMS-EI calcd. for $C_{10}H_{14}O_2$ (M^+) 166.0994. Found: $C_{10}H_{14}O_2$ 166.0998.



1cb

Following the general procedure, **1cb** was obtained as yellow oil (400 mg, 24%, 78% purity by ^{1}H NMR).[2]

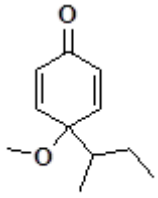
R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 2978, 2945, 2832, 1738, 1670, 1359, 1232, 1038 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.71-6.69 (m, 2H), 6.39-6.37 (m, 2H), 3.18 (s, 3H), 1.97-1.94 (m, 1H), 1.11 (d, *J* = 6.8 Hz, 3H), 0.91 (d, *J* = 7.0 Hz, 3H). HRMS-EI calcd. for $C_{10}H_{14}O_2$ (M^+) 166.0994. Found: $C_{10}H_{14}O_2$ 166. 1000.



1cc

Following the general procedure, **1cc** was obtained as yellow solid (630 mg, 35%), and it has been reported in the literature[5].

R*f* = 0.5 (petroleum ether / ethyl acetate = 7:1, V/V); IR (film): 2979, 2938, 2823, 1738, 1670, 1379, 1202, 1048 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.73-6.69 (m, 2H), 6.35-6.32 (m, 2H), 3.19 (s, 3H), 1.71-1.68 (m, 2H), 1.27-1.17 (m, 4H), 0.84 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.5, 151.2, 131.4, 75.8, 53.0, 39.2, 25.6, 22.9, 13.8. HRMS-EI calcd. for $C_{11}H_{16}O_2$ (M^+) 180.1150. Found: $C_{11}H_{16}O_2$ 180.1149.



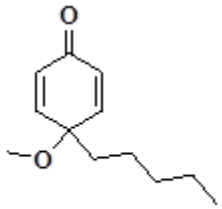
1cd

Following the general procedure, **1cd** was obtained as yellow oil (470 mg, 26%).

R*f* = 0.5 (petroleum ether / ethyl acetate = 7:1, V/V); IR (film): 2974, 2943, 2823, 1738, 1671, 1370, 1222, 1069 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.71-6.68 (m, 2H), 6.41-6.37 (m, 2H), 3.20 (s, 3H), 1.79-1.76 (m, 2H), 0.92-0.88 (m, 7H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.7, 150.5, 150.0, 132.4,

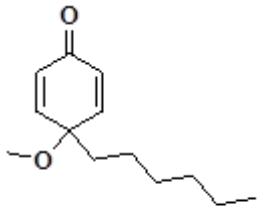
[5] A. J. Stern, J. S. Swenton, *J. Org. Chem.* **1988**, *53*, 2465-2468.

132.0, 78.5, 52.9, 43.6, 23.8, 13.4, 12.5. HRMS-EI calcd. for $C_{11}H_{16}O_2$ (M^+) 180.1150. Found: $C_{11}H_{16}O_2$ 180.1152.



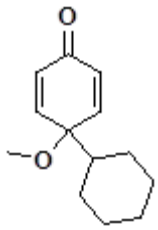
1ce

Following the general procedure, **1ce** was obtained as viscous yellow solid (540 mg, 28%). R*f* = 0.5 (petroleum ether / ethyl acetate = 8:1, V/V); IR (film): 2978, 2949, 2823, 1738, 1670, 1370, 1232, 1058 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.74-6.71 (m, 2H), 6.36-6.33 (m, 2H), 3.20 (s, 3H), 1.71-1.68 (m, 2H), 1.27-1.21 (m, 6H), 0.85 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.5, 151.2, 131.4, 75.9, 53.0, 39.4, 31.9, 23.1, 22.4, 13.9. HRMS-EI calcd. for $C_{12}H_{18}O_2$ (M^+) 194.1307. Found: $C_{12}H_{18}O_2$ 194.1306.



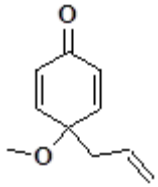
1cf

Following the general procedure, **1cf** was obtained as viscous yellow solid (440 mg, 21%). R*f* = 0.5 (petroleum ether / ethyl acetate = 8:1, V/V); IR (film): 2976, 2946, 2822, 1738, 1670, 1360, 1242, 1038 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.74-6.71 (m, 2H), 6.37-6.34 (m, 2H), 3.20 (s, 3H), 1.72-1.69 (m, 2H), 1.28-1.21 (m, 8H), 0.85 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.6, 151.3, 131.4, 75.9, 53.1, 39.5, 31.6, 29.4, 23.4, 22.5, 14.0. HRMS-EI calcd. for $C_{13}H_{20}O_2$ (M^+) 208.1463. Found: $C_{13}H_{20}O_2$ 208.1464.



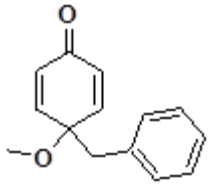
1cg

Following the general procedure, **1cg** was obtained as viscous yellow solid (450 mg, 22%). R*f* = 0.5 (petroleum ether / ethyl acetate = 8:1, V/V); IR (film): 2976, 2969, 2820, 1738, 1670, 1372, 1212, 1038 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.74-6.71 (m, 2H), 6.41-6.36 (m, 2H), 3.20 (s, 3H), 1.88-1.85 (m, 2H), 1.77-1.73 (m, 2H), 1.68-1.61 (m, 2H), 1.24-1.16 (m, 2H), 1.12-1.06 (m, 1H), 0.96-0.88 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.8, 150.6, 132.0, 78.1, 52.8, 46.7, 27.4, 26.4, 26.4. HRMS-EI calcd. for $C_{13}H_{18}O_2$ (M^+) 206.1307. Found: $C_{13}H_{18}O_2$ 206.1304.



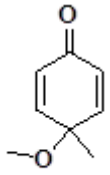
1ch

Following the general procedure, **1ch** was obtained as yellow oil (590 mg, 36%).
R*f* = 0.5 (petroleum ether / ethyl acetate = 2:1, V/V); IR (film): 2973, 2959, 2813, 1738, 1670, 1330, 1222, 1078 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.74-6.71 (m, 2H), 6.35-6.32 (m, 2H), 5.10-5.08 (m, 1H), 5.08-5.03 (m, 2H), 3.20 (s, 3H), 2.45 (dt, *J* = 7.0, 1.0 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.3, 150.6, 131.5, 131.0, 119.5, 75.2, 53.2, 43.9. HRMS-EI calcd. for $C_{10}H_{12}O_2$ (M^+) 164.0837. Found: $C_{10}H_{12}O_2$ 164.0839.



1ci

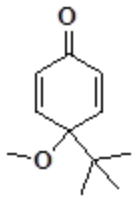
Following the general procedure, **1ci** was obtained as yellow oil (780 mg, 36%).[6]
R*f* = 0.5 (petroleum ether / ethyl acetate = 4:1, V/V); IR (film): 2978, 2949, 1738, 1670, 1360, 1235, 1048 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27-21 (m, 3H), 7.17-7.15 (m, 2H), 6.75-6.71 (m, 2H), 6.31-6.28 (m, 2H), 3.21 (s, 3H), 3.00 (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.1, 150.7, 134.8, 131.3, 130.7, 128.0, 127.0, 75.6, 53.3, 46.3. HRMS-EI calcd. for $C_{14}H_{14}O_2$ (M^+) 214. 0994. Found: $C_{14}H_{14}O_2$ 214. 0998.



1d

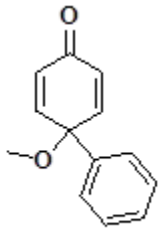
Following the general procedure, **1d** was obtained as yellow solid (1.1 g, 80%), and it has been reported in the literature[7].
R*f* = 0.5 (petroleum ether / ethyl acetate = 5:1, V/V); IR (film): 2976, 2932, 2834, 1738, 1670, 1350, 1242, 1020 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.77-6.74 (m, 2H), 6.32-6.28 (m, 2H), 3.19 (s, 3H), 1.42 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.2, 151.8, 130.5, 72.6, 53.3, 26.3. HRMS-EI calcd. for $C_8H_{10}O_2$ (M^+) 138.0681. Found: 138.0680.

[6] A. Pelter, S.M. A. Elgendy, *J. Chem. Soc., Perkin Trans.* 1, **1993**, *16*, 1891-1896.
[7] S. Large, N. Roques, B. R. Langlois; *J. Org. Chem.* **2000**, *65*, 8848-8856.



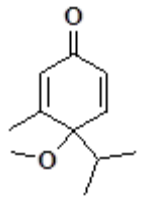
1e

Following the general procedure, **1e** was obtained as yellow oil (470 mg, 26%).[8].
R*f* = 0.5 (petroleum ether / ethyl acetate = 8:1, V/V); IR (film): 2970, 2936, 2835, 1738, 1670, 1320, 1245, 1025 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.89-6.85 (m, 2H), 6.41-6.37 (m, 2H), 3.18 (s, 3H), 0.99 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.0, 150.6, 132.3, 79.7, 53.2, 39.3, 25.6. HRMS-EI calcd. for $C_{11}H_{16}O_2$ (M^+) 180.1150. Found: $C_{11}H_{16}O_2$ 180.1149.



1f

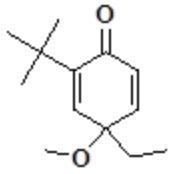
Following the general procedure, **1f** was obtained as slightly yellow solid (1.0 g, 50%), and it has been reported in the literature[8].
R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 2970, 2936, 2835, 1738, 1670, 1320, 1245, 1025 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.447-7.45 (m, 2H), 7.37-7.29 (m, 3H), 6.82-6.79 (m, 2H), 6.42-6.39 (m, 2H), 3.43 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.5, 150.5, 138.3, 130.1, 128.8, 128.3, 125.7, 76.6, 52.9. HRMS-EI calcd. for $C_{13}H_{12}O_2$ (M^+) 200.0837. Found: $C_{13}H_{12}O_2$ 200.0839.



1g

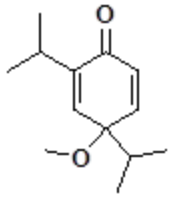
Following the general procedure, **1g** was obtained as viscous yellow solid (540 mg, 30%).
R*f* = 0.5 (petroleum ether / ethyl acetate = 5:1, V/V); IR (film): 2976, 2948, 2825, 1738, 1670, 1370, 1242, 1052 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.76 (d, *J* = 10.0 Hz, 1H), 6.41 (dd, *J* = 10.0, 2.0 Hz, 1H), 6.24 (dq, *J* = 3.0, 1.0 Hz, 1H), 3.08 (s, 3H), 210-2.07(m, 1H), 1.90 (d, *J* = 1.5 Hz, 3H), 1.09 (d, *J* = 7.0 Hz, 3H), 0.64 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.5, 160.6, 148.3, 132.8, 130.7, 80.8, 52.6, 35.1, 17.7, 17.0, 16.6. HRMS-EI calcd. for $C_{11}H_{16}O_2$ (M^+) 180.1150. Found: $C_{11}H_{16}O_2$ 180.1152.

[8] A. S. Mitchell, R. A. Russell, *Tetrahedron*. **1997**, *53*, 4387-4410.



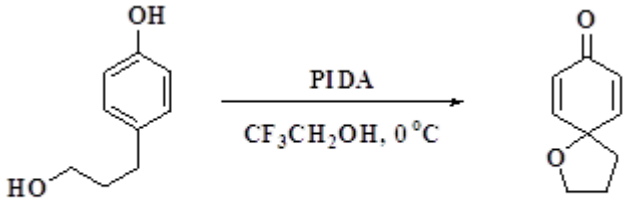
1h

Following the general procedure, **1h** was obtained as yellow oil (910 mg, 44%).
R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 2978, 2943, 2815, 1738, 1670, 1378, 1232, 1022 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.558-6.55 (m, 1H), 6.43-6.42 (m, 1H), 6.27-6.24 (m, 1H), 3.16 (s, 3H), 1.72 (qd, *J* = 7.5, 2.0 Hz, 2H), 1.22 (s, 9H), 0.76 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.7, 149.4, 148.2, 144.1, 133.4, 76.7, 52.7, 34.8, 32.5, 29.3, 7.9. HRMS-EI calcd. for $C_{13}H_{20}O_2$ (M^+) 208.1463. Found: 208.1460.



1i

Following the general procedure, **1i** was obtained as yellow oil (600 mg, 29%).
R*f* = 0.5 (petroleum ether / ethyl acetate = 3:1, V/V); IR (film): 2979, 2938, 2835, 1738, 1670, 1360, 1249, 1022 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.64 (dd, *J* = 10.0, 3.0 Hz, 1H), 6.39 (dd, *J* = 10.0, 3.0 Hz, 1H), 6.37 (d, *J* = 10.2, 1H), 3.14 (s, 3H), 3.04-2.98 (m, 1H), 1.95-1.93 (m, 1H), 1.07 (d, *J* = 7.0 Hz, 6H), 0.88 (d, *J* = 7.0 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.4, 148.9, 148.7, 142.2, 132.7, 78.4, 52.7, 36.7, 26.3, 22.1, 21.7, 17.1. HRMS-EI calcd. for $C_{13}H_{20}O_2$ (M^+) 208.1463. Found: 208.1465.



1j

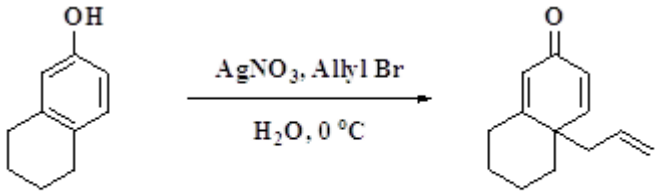
Substrate **1j** is synthesized according to related literature[9].

A solution of 4-(3-hydroxypropyl)phenol (10 mmol, 1.0 equiv) in CF_3CH_2OH (10 mL) is cooled to 0 °C and PIDA (12 mmol, 1.2 equiv) is added portionwise in 0.5 h and the reaction mixture is stirred for 2 h at 0 °C. The reaction mixture is poured into water and is extracted with ethyl acetate. The organic layers are combined, washed with cold 4 M NaOH and brine successively, dried with Na_2SO_4 and concentrated, and then purified by flash chromatography (petroleum ether / ethyl acetate=5:1, V/V) to afford the target product as yellow solid (650 mg, 43%).

R*f* = 0.5 (petroleum ether / ethyl acetate = 2:1, V/V); IR (film): 2978, 2938, 2815, 1738, 1670, 1368, 1245, 1032 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.79-6.76 (m, 2H), 6.11-6.08 (m, 2H), 4.04 (t, *J* = 7.0 Hz, 2H), 2.16-2.10 (m, 2H), 2.04-2.01 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.6, 149.8, 127.2,

[9] K. Ohkata, Y. Tamura, B. B. Shetuni, R.Takagi, W. Miyanaga, S. Kojima, L. A. Paquette, *J. Am. Chem. Soc*. **2004**, *126*, 16783-16792.

77.4, 69.3, 37.0, 26.8. HRMS-EI calcd. for $C_9H_{10}O_2$ (M^+) 150.0681. Found: 150.0679.



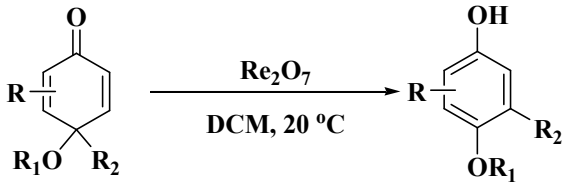
1k

Substrate **1k** is synthesized according to related literature[10].

A mixture of 5,6,7,8-tetrahydro-2-naphthol (10 mmol, 1.0 equiv) and allylbromide (10 mmol, 1.0 equiv) in H_2O (10 mL) is cooled to 0 °C and silver nitrate (12 mmol, 1.2 equiv) is added portionwise in 0.5 h and the reaction mixture is stirred for 1 h at 0 °C. The solid formed in the reaction mixture is filtered and washed with ethyl acetate. The filtrate is poured into water and is extracted with ethyl acetate. The organic layers are combined, washed with cold 4 M NaOH and brine successively, dried with Na_2SO_4 and concentrated, and then purified by flash chromatography (petroleum ether / ethyl acetate = 5:1, V/V) to afford the target product as colorless oil (330 mg, 18%).

R*f* = 0.5 (petroleum ether / ethyl acetate = 2:1, V/V); IR (film): 2976, 2948, 2825, 1738, 1670, 1527, 892 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.70 (d, *J* = 10.0 Hz, 1H), 6.26 (dd, *J* = 10.0, 1.8 Hz, 1H), 6.15 (d, *J* = 1.5 Hz, 1H), 5.3-5.369 (m, 1H), 5.02-4.96 (m, 2H), 2.71 (dd, *J* = 14.0, 7.5 Hz, 1H), 2.38-2.35 (m, 2H), 2.26 (dd, *J* = 14.0, 7.5 Hz, 1H), 2.04-2.01 (m, 1H), 1.95-1.91 (m, 1H), 1.79-1.73 (m, 1H), 1.72-1.71 (m, 1H), 1.38-1.32 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 187.2, 165.9, 155.6, 131.9, 128.5, 126.0, 118.1, 44.8, 39.7, 37.1, 32.7, 27.9, 20.7. HRMS-EI calcd. for $C_{13}H_{16}O$ (M^+) 188.1201. Found: 188.1200.

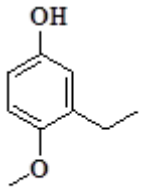
General procedure for the syntheses of products



To a mixture of Re_2O_7 (0.1 equiv) in CH_2Cl_2 (0.5 mL / 0.1 mmol) under N_2 at 20 °C is added a solution of substrates (1.0 equiv) in CH_2Cl_2 (0.5 mL / mmol), and the reaction mixture is stirred at 20 °C until TLC showed that the substrates are consumed completely (1-4 h). The target products were purified by preparative TLC using a solvent system of petroleum ether and ethyl acetate (See R*f* of target products).

2D-NMR experiments of the product **2cb** are used as an example to confirm the structures of the products.

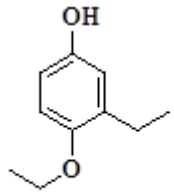
[10] U. Widmer, J. Zsindely, H.-J. Hansen and H. Schmid, *Helv. Chim. Acta*, **1973**, *56*, 75-105.



2aa

Following the general procedure, **2aa** was obtained from **1aa** (0.1 mmol, 15 mg) as slightly brown viscous oil (14 mg, 93%).

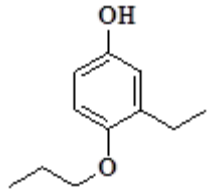
R*f* = 0.5 (petroleum ether / ethyl acetate = 5:1, V/V); IR (film): 3393, 2976, 2948, 2825, 1502, 1470, 1242, 917 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (d, *J* = 8.5 Hz, 1H), 6.68 (d, *J* = 3.0 Hz, 1H), 6.63 (dd, *J* = 8.5, 3.0 Hz, 1H), 4.71 (s, 1H), 3.79 (s, 3H), 2.60 (q, *J* = 7.5 Hz, 2H), 1.18 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.6, 149.2, 134.2, 116.3, 112.5, 111.6, 56.1, 23.1, 14.1. HRMS-EI calcd. for $C_9H_{12}O_2$ (M^+) 152.0837. Found: 152.0834.



2ab

Following the general procedure, **2ab** was obtained from **1ab** (0.1 mmol, 17 mg) as slightly brown viscous oil (13 mg, 78%).

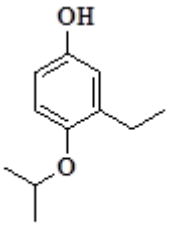
R*f* = 0.5 (petroleum ether / ethyl acetate = 5:1, V/V); IR (film): 3343, 2975, 2958, 2828, 1512, 1474, 1243, 832 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (d, *J* = 8.5 Hz, 1H), 6.68 (d, *J* = 3.0 Hz, 1H), 6.61 (dd, *J* = 8.5, 3.0 Hz, 1H), 5.09 (s, 1H), 3.98 (q, *J* = 7.0 Hz, 2H), 2.61 (q, *J* = 7.5 Hz, 2H), 1.40 (t, *J* = 7.0 Hz, 3H), 1.19 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.9, 149.3, 134.6, 116.2, 113.2, 112.7, 64.7, 23.2, 15.1, 14.1. HRMS-EI calcd. for $C_{10}H_{14}O_2$ (M^+) 166.0994. Found: $C_{10}H_{14}O_2$ 166.0990.



2ac

Following the general procedure, **2ac** was obtained from **1ac** (0.1 mmol, 18 mg) as slightly brown viscous oil (16.5 mg, 92%).

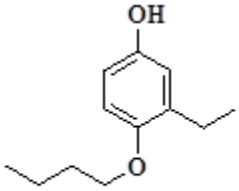
R*f* = 0.5 (petroleum ether / ethyl acetate = 5:1, V/V); IR (film): 3373, 2978, 2938, 2815, 1512, 1472, 1249, 919 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.71 (d, *J* = 8.5 Hz, 1H), 6.68 (d, *J* = 3.0 Hz, 1H), 6.61 (dd, *J* = 8.5, 3.0 Hz, 1H), 4.93 (s, 1H), 3.88 (t, *J* = 7.0 Hz, 2H), 2.62 (q, *J* = 7.5 Hz, 2H), 1.82-1.80 (m, 2H), 1.19 (t, *J* = 7.5 Hz, 3H), 1.06 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.0, 149.1, 134.5, 116.2, 112.8, 112.5, 70.5, 23.2, 22.9, 14.1, 10.7. HRMS-EI calcd. for $C_{11}H_{16}O_2$ (M^+) 180.1150. Found: $C_{11}H_{16}O_2$ 180.1151.



2ad

Following the general procedure, **2ad** was obtained from **1ad** (0.1 mmol, 18 mg) as slightly brown viscous oil (14.5 mg, 81%).

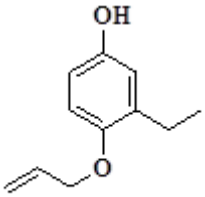
R*f* = 0.5 (petroleum ether / ethyl acetate = 5:1, V/V); IR (film): 3323, 2970, 2941, 2823, 1512, 1480, 1232, 862 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.74 (d, *J* = 8.5 Hz, 1H), 6.67 (d, *J* = 3.0 Hz, 1H), 6.60 (dd, *J* = 8.5, 3.0 Hz, 1H), 4.84 (s, 1H), 4.40-4.34 (m, 1H), 2.59 (q, *J* = 7.5 Hz, 2H), 1.31 (d, *J* = 6.0 Hz, 6H), 1.17 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.6, 149.4, 135.8, 116.1, 115.6, 112.6, 71.4, 23.2, 22.3, 14.1. HRMS-EI calcd. for $C_{11}H_{16}O_2$ (M^+) 180.1150. Found: $C_{11}H_{16}O_2$ 180.1152.



2ae

Following the general procedure, **2ae** was obtained from **1ae** (0.1 mmol, 19 mg) as slightly brown viscous oil (17 mg, 89%).

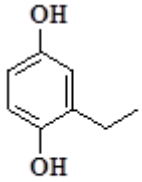
R*f* = 0.5 (petroleum ether / ethyl acetate = 5:1, V/V); IR (film): 3363, 2979, 2942, 2824, 1504, 1430, 1212, 809 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (d, *J* = 8.5 Hz, 1H), 6.68 (d, *J* = 3.0 Hz, 1H), 6.61 (dd, *J* = 8.5, 3.0 Hz, 1H), 4.99 (s, 1H), 3.92 (t, *J* = 6.5 Hz, 2H), 2.61 (q, *J* = 7.5 Hz, 2H), 1.79-1.77 (m, 2H), 1.54-1.50 (m, 2H), 1.19 (t, *J* = 7.5 Hz, 3H), 0.99 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.1, 149.1, 134.4, 116.2, 112.7, 112.5, 68.6, 31.6, 23.2, 19.4, 14.1, 13.2. HRMS-EI calcd. for $C_{12}H_{18}O_2$ (M^+) 194.1307. Found: $C_{12}H_{18}O_2$ 194.1304.



2af

Following the general procedure, **2af** was obtained from **1af** (0.1 mmol, 18 mg) as slightly brown viscous oil (4.6 mg, 26%) after 8 h with **1f** recovered (11 mg, 60%).

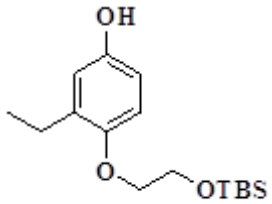
R*f* = 0.5 (petroleum ether / ethyl acetate = 2:1, V/V); IR (film): 3399, 2966, 2938, 2820, 1522, 1477, 1222, 823 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (d, *J* = 8.5 Hz, 1H), 6.68 (d, *J* = 3.0 Hz, 1H), 6.60 (dd, *J* = 8.5, 3.0 Hz, 1H), 6.10-6.02 (m, 1H), 5.42 (dq, *J* = 17.0, 1.5 Hz, 1H), 5.26 (dq, *J* = 17.0, 1.5 Hz, 1H), 4.51 (s, 1H), 4.49 (dt, *J* = 5.0, 1.5 Hz, 2H), 2.64 (q, *J* = 7.5 Hz, 2H), 1.20 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.6, 149.4, 134.6, 133.9, 116.7, 116.2, 113.1, 112.4, 69.7, 23.2, 14.1. HRMS-EI calcd. for $C_{11}H_{14}O_2$ (M^+) 178.0994. Found: $C_{11}H_{14}O_2$ 178.0993.



2ag

Following the general procedure, **2ag** was obtained from **1ag** (0.1 mmol, 14 mg) as slightly brown viscous oil (11 mg, 78%).[11]

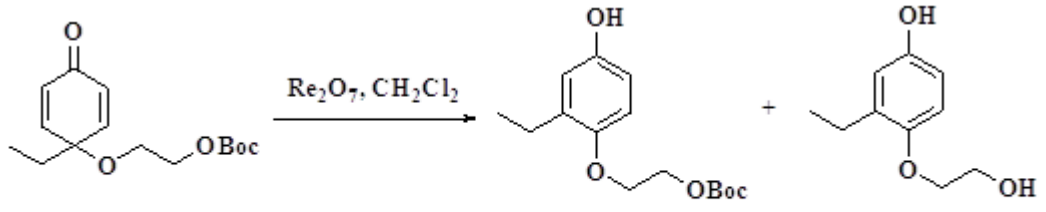
R*f* = 0.5 (petroleum ether / ethyl acetate = 3:2, V/V); IR (film): 3343, 2973, 2918, 2822, 1512, 1471, 1222, 844 cm^{-1}; ^{1}H NMR (400 MHz, CD_3OD) δ 6.56 (d, *J* = 8.5 Hz, 1H), 6.53 (d, *J* = 3.0 Hz, 1H), 6.42 (dd, *J* = 8.5, 3.0 Hz, 1H), 4.87 (s, 2H), 2.52 (q, *J* = 7.5 Hz, 2H), 1.14 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, CD_3OD) δ 149.7, 147.5, 131.4, 115.4, 115.2, 112.4, 22.9, 13.3. HRMS-EI calcd. for $C_8H_{10}O_2$ (M^+) 138.0681. Found: $C_8H_{10}O_2$ 138.0680.



2ba

Following the general procedure, **2ba** was obtained from **1ba** (0.2 mmol, 60 mg) as slightly brown viscous oil (20 mg, 33%).

R*f* = 0.5 (petroleum ether / ethyl acetate = 7:1, V/V); IR (film): 3389, 2957, 2930, 2858, 1502, 1277, 1215, 1120, 835 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (d, *J* = 8.5 Hz, 1H), 6.67 (d, *J* = 3.0 Hz, 1H), 6.60 (dd, *J* = 8.5, 3.0 Hz, 1H), 4.45 (s, 1H), 3.98-3.96 (m, 4H), 2.62 (q, *J* = 7.5 Hz, 2H), 1.18 (t, *J* = 7.5 Hz, 3H), 0.92 (s, 9H), 0.11 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.0, 149.4, 134.6, 116.2, 113.1, 112.5, 70.5, 62.2, 25.9, 23.2, 18.4, 14.2, -5.3. HRMS-EI calcd. for $C_{16}H_{28}O_3Si$ (M^+) 296.1808. Found: $C_{16}H_{28}O_3Si$ 296.1809.



1bb **2bb** **2bc**

23% **52%**

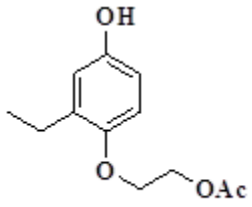
Following the general procedure, from **1bb** (0.2 mmol, 56 mg), **2bb** (12 mg, 23%) and **2bc** (29 mg, 52%) were obtained as slightly brown viscous oil.

For **2bb**: R*f* = 0.5 (petroleum ether / ethyl acetate = 7:1, V/V); IR (film): 3343, 2973, 2918, 2822, 1740, 1512, 1471, 1222, 894 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.67 (d, *J* = 8.5 Hz, 1H), 6.63 (d, *J* = 3.0 Hz, 1H), 6.55 (dd, *J* = 8.2, 3.0 Hz, 1H), 4.65 (s, 1H), 3.98 (t, *J* = 5.5 Hz, 2H), 3.68 (t, *J* = 5.5 Hz, 2H), 2.58 (q, *J* = 7.5 Hz, 2H), 1.22 (s, 9H), 1.16 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.9, 149.4,

[11] Z. Florjanczyk,W. Kuran,S. Pasynkiewicz,G. Kwas, *J. Organomet. Chem.* **1976**, *112*, 21-28.

134.5, 116.2, 113.0, 112.5, 73.3, 68.8, 60.7, 27.5, 23.3, 14.1. HRMS-EI calcd. for $C_{15}H_{22}O_5$ (M^+) 282.1467. Found: $C_{15}H_{22}O_5$ 282.1466.

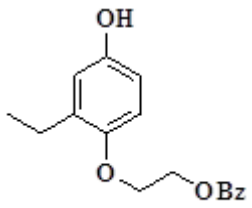
For **2bc**, R*f* = 0.5 (petroleum ether / ethyl acetate = 2:1, V/V); IR (film): 3343, 2973, 2918, 2822, 1514, 1471, 1222, 874 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.69 (d, *J* = 8.5 Hz, 1H), 6.66 (d, *J* = 3.0 Hz, 1H), 6.58 (dd, *J* = 8.5, 3.0 Hz, 1H), 5.12 (s, 1H), 4.00 (t, *J* = 5.5 Hz, 2H), 3.94 (t, *J* = 5.5 Hz, 2H), 2.58 (q, *J* = 7.5 Hz, 2H), 2.20 (s, 1H), 1.16 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.4, 149.9, 134.5, 116.3, 113.3, 112.7, 70.3, 61.8, 23.1, 14.1. HRMS-EI calcd. for $C_{10}H_{14}O_3$ (M^+) 182.0943. Found: $C_{10}H_{14}O_3$ 182.0943.



2bd

Following the general procedure, **2bd** was obtained from **1bd** (0.25 mmol, 56 mg) as slightly brown viscous oil (45 mg, 80%).

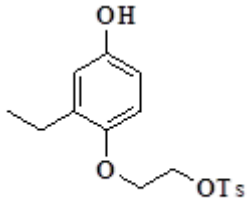
R*f* = 0.5 (petroleum ether / ethyl acetate = 5:2, V/V); IR (film): 3413, 2966, 2918, 2822, 1741, 1503, 1450, 1211, 1052 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.70 (d, *J* = 8.5 Hz, 1H), 6.68 (d, *J* = 3.0 Hz, 1H), 6.61 (dd, *J* = 8.5, 3.0 Hz, 1H), 5.41 (s, 1H), 4.42 (t, *J* = 5.5 Hz, 2H), 4.11 (t, *J* = 5.5 Hz, 2H), 2.59 (q, *J* = 7.5 Hz, 2H), 2.11 (s, 3H), 1.17 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.5, 150.3, 150.0, 134.8, 116.4, 113.4, 112.6, 67.1, 63.2, 23.2, 20.9, 14.1. HRMS-EI calcd. for $C_{12}H_{16}O_4$ (M^+) 224.1049. Found: $C_{12}H_{16}O_4$ 224.1049.



2be

Following the general procedure, **2be** was obtained from **1be** (0.1 mmol, 57 mg) as slightly brown viscous oil (44 mg, 77%).

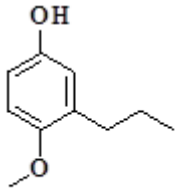
R*f* = 0.5 (petroleum ether / ethyl acetate = 3:1, V/V); IR (film): 3343, 2973, 2918, 2822, 1738, 1512, 1471, 1222, 924 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 7.0 Hz, 2H), 7.55 (t, *J* = 7.0 Hz, 1H), 7.42 (t, *J* = 7.5 Hz, 2H), 6.72 (d, *J* = 8.0 Hz, 1H), 6.66 (d, *J* = 3.0 Hz, 1H), 6.60 (dd, *J* = 8.5, 3.0 Hz, 1H), 4.95 (s, 1H), 4.65 (t, *J* = 5.5 Hz, 2H), 4.23 (t, *J* = 5.5 Hz, 2H), 2.58 (q, *J* = 7.5 Hz, 2H), 1.13 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.7, 150.4, 149.9, 134.9, 133.1, 129.9, 129.7, 128.4, 116.4, 113.3, 112.6, 67.2, 63.6, 23.2, 14.2. HRMS-EI calcd. for $C_{17}H_{18}O_4$ (M^+) 286.1205. Found: $C_{17}H_{18}O_4$ 286.1205.



2bf

Following the general procedure, **2bf** was obtained from **1bf** (0.2 mmol, 67 mg) as slightly brown viscous oil (45 mg, 65%).

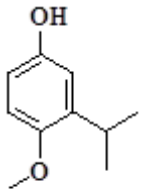
R*f* = 0.5 (petroleum ether / ethyl acetate = 2:3, V/V); IR (film): 3380, 2970, 2935, 2853, 1738, 1501, 1449, 1355, 1216, 1175, 926 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 6.65 (d, *J* = 8.5 Hz, 1H), 6.57 (d, *J* = 3.0 Hz, 1H), 6.55 (dd, *J* = 8.5, 3.0 Hz, 1H), 4.98 (s, 1H), 4.34 (t, *J* = 5.5 Hz, 2H), 4.08 (t, *J* = 5.5 Hz, 2H), 2.48 (q, *J* = 7.5 Hz, 2H), 2.45 (s, 3H). 1.10 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.0, 149.9, 145.0, 134.7, 132.8, 129.9, 127.9, 116.3, 113.1, 112.5, 68.5, 66.5, 23.0, 21.6, 14.1. HRMS-EI calcd. for $C_{17}H_{20}O_5S$ (M^+) 336.1031. Found: $C_{17}H_{20}O_5S$ 336.1031.



2ca

Following the general procedure, **2ca** was obtained from **1ca** (0.1 mmol, 17 mg) as slightly brown viscous oil (15.5 mg, 91%).[12]

R*f* = 0.5 (petroleum ether / ethyl acetate = 5:1, V/V); IR (film): 3341, 2967, 2932, 2845, 1502, 1452, 1215, 917 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (d, *J* = 8.5 Hz, 1H), 6.66 (d, *J* = 3.0 Hz, 1H), 6.63 (dd, *J* = 8.5, 3.0 Hz, 1H), 4.57 (s, 1H), 3.78 (s, 3H), 2.54 (t, *J* = 7.0 Hz, 2H), 1.60 (m, 2H), 0.95 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.8, 149.0, 132.6, 117.1, 112.5, 111.6, 56.1, 32.2, 22.9, 14.0. HRMS-EI calcd. for $C_{10}H_{14}O_2$ (M^+) 166.0994. Found: $C_{10}H_{14}O_2$ 166.0995.



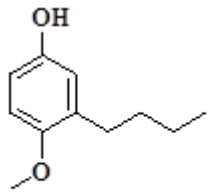
2cb

Following the general procedure, **2cb** was obtained from **1cb** (0.1 mmol, 17 mg) as slightly brown viscous oil (14.5 mg, 86%).

R*f* = 0.5 (petroleum ether / ethyl acetate = 5:1, V/V); IR (film): 3391, 2972, 2944, 2865, 1517, 1410, 1252, 872 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.74 (d, *J* = 8.5 Hz, 1H), 6.74 (d, *J* = 3.0 Hz, 1H), 6.62 (dd, *J* = 8.5, 3.0 Hz, 1H), 4.50 (s, 1H), 3.79 (s, 3H), 3.33-3.25 (m, 1H), 1.20 (d, *J* = 7.0 Hz, 6H). ^{13}C

[12] F.-T. Hong, K.-S. Lee, Y.-F. Tsai, C.-C. Liao, *J. Chin. Chem. Soc.,***1998**, *45*,1-12

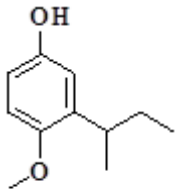
NMR (126 MHz, $CDCl_3$) δ 151.0, 149.4, 138.7, 113.5, 112.3, 111.8, 56.2, 26.7, 22.7. HRMS-EI calcd. for $C_{10}H_{14}O_2$ (M^+) 166.0994. Found: $C_{10}H_{14}O_2$ 166.0998.



2cc

Following the general procedure, **2cc** was obtained from **1cc** (0.1 mmol, 18 mg) as slightly brown viscous oil (16 mg, 90%).

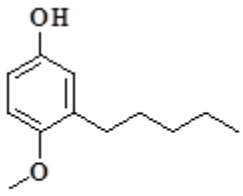
R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 3383, 2970, 2947, 2825, 1506, 1450, 1222, 827 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (d, *J* = 8.5 Hz, 1H), 6.66 (d, *J* = 3.0 Hz, 1H), 6.62 (dd, *J* = 8.5, 3.0 Hz, 1H), 4.60 (s, 1H), 3.78 (s, 3H), 2.58-2.55 (m, 2H), 1.57-1.54 (m, 2H), 1.39-1.35 (m, 2H), 0.93 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.8, 149.1, 132.9, 117.0, 112.5, 111.6, 56.1, 32.0, 29.8, 22.6, 14.0. HRMS-EI calcd. for $C_{11}H_{16}O_2$ (M^+) 180.1150. Found: $C_{11}H_{16}O_2$ 180.1149.



2cd

Following the general procedure, **2cd** was obtained from **1cd** (0.1 mmol, 18 mg) as slightly brown viscous oil (13 mg, 71%).

R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 3389, 2966, 2958, 2835, 1512, 1480, 1252, 832 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.74 (d, *J* = 8.5 Hz, 1H), 6.68 (d, *J* = 3.0 Hz, 1H), 6.62 (dd, *J* = 8.5, 3.0 Hz, 1H), 4.62 (s, 1H), 3.77 (s, 3H), 3.09-3.04 (m, 1H), 1.61-1.51 (m, 2H), 1.17 (d, *J* = 7.0 Hz, 3H), 0.85 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.4, 149.4, 137.7, 114.1, 112.3, 112.0, 56.3, 33.5, 29.8, 20.5, 12.1. HRMS-EI calcd. for $C_{11}H_{16}O_2$ (M^+) 180.1150. Found: $C_{11}H_{16}O_2$ 180.1150.

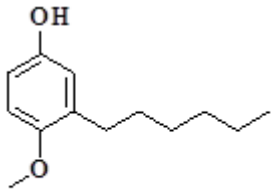


2ce

Following the general procedure, **2ce** was obtained from **1ce** (0.1 mmol, 19.5 mg) as slightly brown viscous oil (17.5 mg, 90%).

R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 3373, 2977, 2958, 2845, 1507, 1474, 1232, 852 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (d, *J* = 8.5 Hz, 1H), 6.67 (d, *J* = 3.0 Hz, 1H), 6.63 (dd, *J* = 8.5, 3.0 Hz, 1H), 5.06 (s, 1H), 3.78 (s, 3H), 2.58-2.54 (m, 2H), 1.58-1.55 (m, 2H), 1.36-1.32

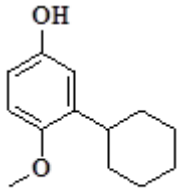
(m, 4H), 0.91 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.7, 149.1, 132.9, 117.0, 112.6, 111.7, 56.1, 31.7, 30.0, 29.5, 22.6, 14.1. HRMS-EI calcd. for $C_{12}H_{18}O_2$ (M^+) 194.1307. Found: $C_{12}H_{18}O_2$ 194.1308.



2cf

Following the general procedure, **2cf** was obtained from **1cf** (0.1 mmol, 21 mg) as slightly brown viscous oil (19 mg, 91%).

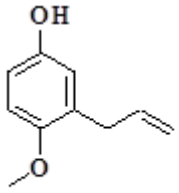
R*f* = 0.5 (petroleum ether / ethyl acetate = 6:1, V/V); IR (film): 3395, 2966, 2938, 2835, 1512, 1475, 1232, 832 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (d, J = 8.5 Hz, 1H), 6.67 (d, J = 3.0 Hz, 1H), 6.63 (dd, J = 8.5, 3.0 Hz, 1H), 4.98 (s, 1H), 3.78 (s, 3H), 2.58-2.55 (m, 2H), 1.58-1.55 (m, 2H), 1.37-1.30 (m, 6H), 0.90 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.7, 149.1, 132.9, 117.0, 112.6, 111.7, 56.1, 31.8, 30.1, 29.8, 29.3, 22.7, 14.1. HRMS-EI calcd. for $C_{13}H_{20}O_2$ (M^+) 208.1463. Found: $C_{13}H_{20}O_2$ 208.1461.



2cg

Following the general procedure, **2cg** was obtained from **1cg** (0.1 mmol, 21 mg) as slightly brown viscous oil (17.5 mg, 84%).

R*f* = 0.5 (petroleum ether / ethyl acetate = 4:1, V/V); IR (film): 3383, 2976, 2938, 2815, 1502, 1473, 1222, 847 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.74 (d, J = 8.5 Hz, 1H), 6.72 (d, J = 3.0 Hz, 1H), 6.63 (dd, J = 8.5, 3.0 Hz, 1H), 5.05 (s, 1H), 3.79 (s, 3H), 2.93-2.90 (m, 1H), 1.83-1.74 (m, 5H), 1.44-1.39 (m, 2H), 1.35-1.23 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.0, 149.4, 138.0, 114.2, 112.4, 112.0, 56.3, 36.8, 33.2, 27.0, 26.4. HRMS-EI calcd. for $C_{13}H_{18}O_2$ (M^+) 206.1307. Found: $C_{13}H_{18}O_2$ 206.1306.

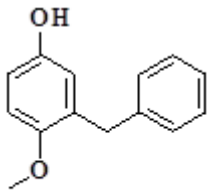


2ch

Following the general procedure, **2ch** was obtained from **1ch** (0.1 mmol, 16.5 mg) as slightly brown viscous oil (14.5 mg, 88%).

R*f* = 0.5 (petroleum ether / ethyl acetate = 2:1, V/V); IR (film): 3343, 2976, 2938, 2826, 1506, 1478, 1222, 912 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.76 (d, J = 8.5 Hz, 1H), 6.70 (d, J = 3.0 Hz, 1H), 6.68 (dd, J = 8.5, 3.0 Hz, 1H), 6.04-5.99 (m, 1H), 5.18-5.15 (m, 2H), 4.83 (s, 1H), 3.77 (s, 3H), 3.40-3.39

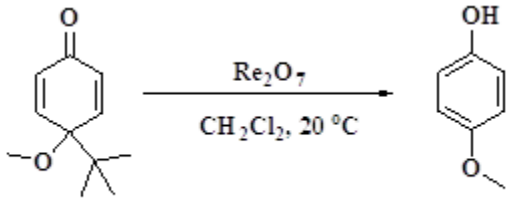
(m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.7, 148.0, 136.2, 126.6, 116.5, 116.5, 116.0, 112.6, 55.8, 35.3. HRMS-EI calcd. for $C_{10}H_{12}O_2$ (M^+) 164.0837. Found: $C_{10}H_{12}O_2$ 164.0838.



2ci

Following the general procedure, **2ci** was obtained from **1ci** (0.1 mmol, 21.5 mg) as slightly brown viscous oil (16.5 mg, 77%).

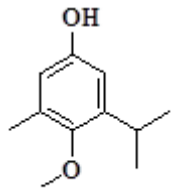
R*f* = 0.5 (petroleum ether / ethyl acetate = 4:1, V/V); IR (film): 3323, 2975, 2938, 2815, 1506, 1430, 1247, 847 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32-7.29 (m, 2H), 7.27-7.22 (m, 3H), 6.76 (d, *J* = 8.5 Hz, 1H), 6.66 (dd, *J* = 8.5, 3.0 Hz, 1H), 6.56 (d, *J* = 3.0 Hz, 1H), 4.82 (s, 1H), 3.95 (s, 2H), 3.79 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.6, 149.2, 140.7, 131.1, 129.1, 128.4, 125.9, 117.6, 113.3, 111.9, 56.2, 35.8. HRMS-EI calcd. for $C_{14}H_{14}O_2$ (M^+) 214.0994. Found: $C_{14}H_{14}O_2$ 214.0999.



1e **2e**

Following the general procedure, **2e** was obtained from **1e** (0.1 mmol, 12.5 mg) as slightly brown viscous oil (11.5 mg, 92%).[13]

R*f* = 0.5 (petroleum ether / ethyl acetate = 4:1, V/V); IR (film): 3485, 2970, 2926, 2841, 1516, 1472, 1245, 1022 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.81-6.77 (m, 4H), 5.20 (s, 1H), 3.77 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.6, 149.5, 116.1, 114.9, 55.9. HRMS-EI calcd. for $C_7H_8O_2$ (M^+) 124.0524. Found: $C_7H_8O_2$ 124.0527.

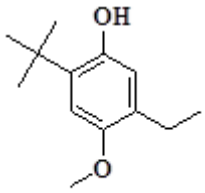


2g

Following the general procedure, **2g** was obtained from **1g** (0.1 mmol, 18 mg) as slightly brown viscous oil (15 mg, 85%).

R*f* = 0.5 (petroleum ether / ethyl acetate = 5:1, V/V); IR (film): 3353, 2970, 2958, 2823, 1502, 1474, 1232, 807 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.55 (d, *J* = 3.0Hz, 1H), 6.49 (d, *J* = 3.0 Hz, 1H), 5.50 (s, 1H), 3.71 (s, 3H), 3.33-3.25 (m, 1H), 2.25 (s, 3H), 1.18 (d, *J* = 7.0 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.8, 149.2, 142.9, 132.0, 115.1, 110.7, 61.1, 26.5, 23.8, 16.4. HRMS-EI calcd. for $C_{11}H_{16}O_2$ (M^+) 180.1150. Found: $C_{11}H_{16}O_2$ 180.1153.

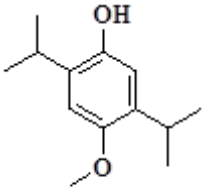
[13] G. A. Molander, L. N.Cavalcanti, *J. Org. Chem.* **2011**, *76*, 623-630.



2h

Following the general procedure, **2h** was obtained from **1h** (0.1 mmol, 21 mg) as slightly brown viscous oil (15.5 mg, 74%).

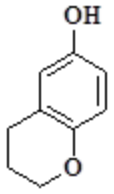
R*f* = 0.5 (petroleum ether / ethyl acetate = 5:1, V/V); IR (film): 3353, 2979, 2958, 2835, 1512, 1476, 1222, 858 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.84 (s, 1H), 6.51 (s, 1H), 4.65 (s, 1H), 3.83 (s, 3H), 2.59 (q, *J* = 7.5 Hz, 2H), 1.45 (s, 9H), 1.20 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.0, 147.9, 133.8, 131.1, 117.4, 110.7, 56.5, 34.6, 29.7, 22.4, 14.1. HRMS-EI calcd. for $C_{13}H_{20}O_2$ (M^+) 208.1463. Found: $C_{13}H_{20}O_2$ 208.1462.



2i

Following the general procedure, **2i** was obtained from **1i** (0.1 mmol, 21 mg) as slightly brown viscous oil (15 mg, 72%).

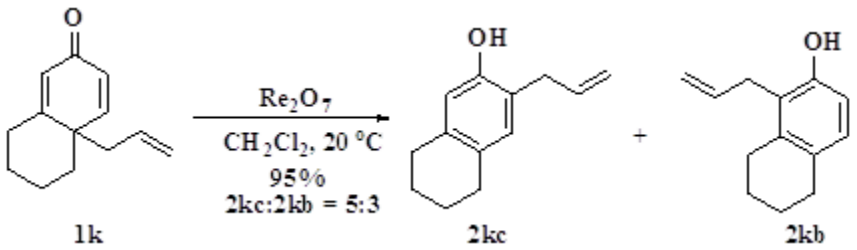
R*f* = 0.5 (petroleum ether / ethyl acetate = 3:1, V/V); IR (film): 3405, 2975, 2928, 2845, 1506, 1473, 1248, 917 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.74 (s, 1H), 6.64 (s, 1H), 4.71 (s, 1H), 3.83 (s, 3H), 3.30-3.26 (m, 1H), 3.24-3.19 (m, 1H), 1.28 (d, *J* = 7.0 Hz, 6H), 1.19 (d, *J* = 7.0 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.1, 146.5, 135.6, 131.8, 113.6, 109.7, 56.5, 27.2, 26.4, 22.8, 22.7. HRMS-EI calcd. for $C_{13}H_{20}O_2$ (M^+) 208.1463. Found: $C_{13}H_{20}O_2$ 208.1460.



2j

Following the general procedure, **2j** was obtained from **1j** (0.1 mmol, 15 mg) as slightly brown viscous oil (13.5 mg, 90%).[9]

R*f* = 0.5 (petroleum ether / ethyl acetate = 3:1, V/V); IR (film): 3373, 2970, 2947, 2823, 1517, 1420, 1241, 907 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.68 (d, *J* = 8.5 Hz, 1H), 6.59 (dd, *J* = 8.5, 3.0 Hz, 1H), 6.54 (d, *J* = 3.0 Hz, 1H), 5.51 (s, 1H), 4.14 (t, *J* = 6.5 Hz, 2H), 2.72 (t, *J* = 6.5 Hz, 2H), 2.00-1.95 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.9, 148.7, 123.2, 117.3, 116.0, 114.4, 66.4, 24.9, 22.4. HRMS-EI calcd. for $C_9H_{10}O_2$ (M^+) 150.0681. Found: $C_9H_{10}O_2$ 150.0683.



Following the general procedure, **2k** was obtained from **1k** (0.1 mmol, 19 mg) as an inseparable mixture of **2kc** and **2kb** (**2kc : 2kb** = 5:3) as slightly brown viscous oil (18 mg, 95%). And their structures were confirmed by NOESY.[10]

R*f* = 0.5 (petroleum ether / ethyl acetate = 3:1, V/V); IR (film): 3343, 2970, 2943, 2815, 1508, 1479, 782 cm^{-1};

2xb: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.89 (d, *J* = 8.0 Hz, 1H), 6.65 (d, *J* = 8.0 Hz, 1H), 6.02-5.97 (m, 1H), 5.11-5.05 (m, 2H), 4.80 (s, 1H), 3.45-3.43 (m, 2H), 2.77-2.73 (m, 4H), 1.84 -1.76 (m, 4H).^{13}C NMR (126 MHz, $CDCl_3$) δ 151.7, 136.6, 135.7, 129.9, 128.1, 123.3, 115.3, 113.2, 30.0, 29.7, 26.6, 23.4, 22.9.

2xc: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.84 (s, 1H), 6.56 (s, 1H), 6.08-6.01 (m, 1H), 5.23-5.16 (m, 2H), 4.86 (s, 1H), 3.40-3.39 (m, 2H), 2.74-2.70 (m, 4H), 1.84-1.76 (m, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.7, 136.9, 136.6, 130.8, 129.4, 122.8, 116.2, 115.9, 34.9, 29.1, 28.5, 23.5, 23.2.

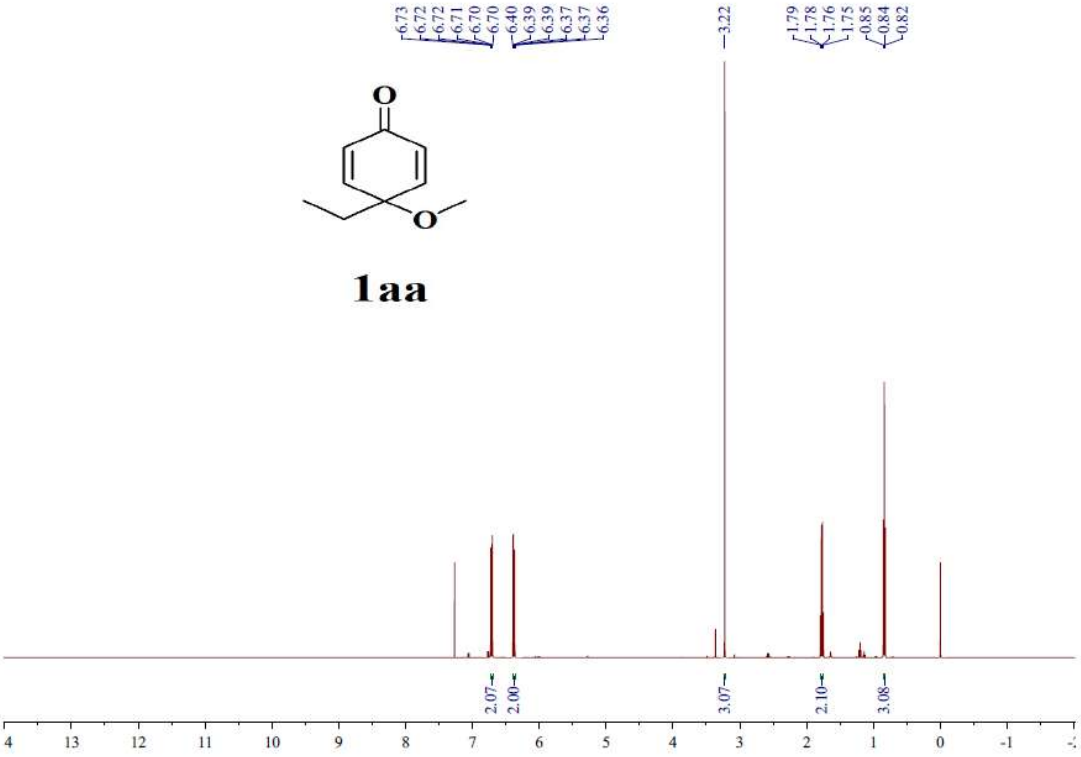
HRMS-EI calcd. for $C_{13}H_{16}O$ (M^+) 188.1201. Found: $C_{13}H_{16}O$ 188.1200.

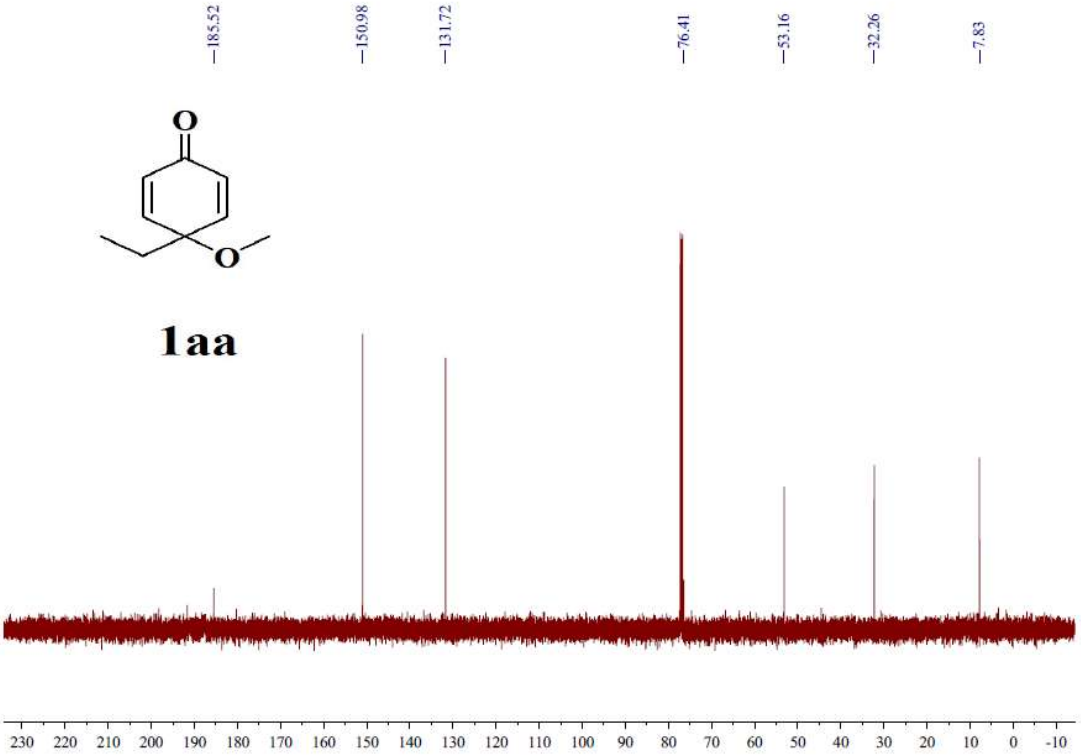
Gram scale syntheses of 2g and 2h

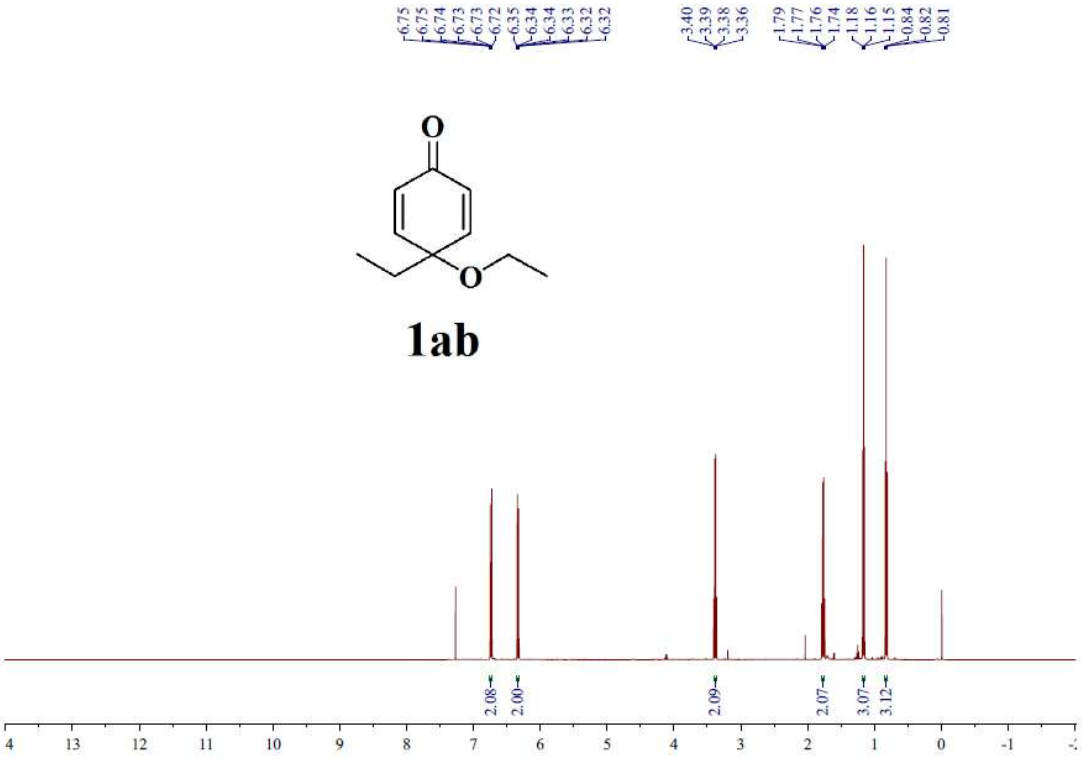
To a mixture of Re_2O_7 (145 mg, 5% mmol) and CH_2Cl_2 (30 mL) under Ar at 25 °C, a solution of substrate **1g** (1.08 g, 6 mmol, 1.0 equiv) in CH_2Cl_2 (30 mL) was added, and the mixture was stirred at 25 °C for 2 h. TLC (petroleum ether / ethyl acetate = 5:1, V/V) showed no starting material remained. The reaction mixture was concentrated, and then purified by flash chromatography (petroleum ether / ethyl acetate = 20:1, V/V) to afford **2g** (880 mg, 82%) as brown solid (mp. 119-122 °C).

To a mixture of Re_2O_7 (120 mg, 5% mmol) and CH_2Cl_2 (25 mL) under Ar at 25 °C, a solution of substrate **1h** (1.04 g, 5 mmol, 1.0 equiv) in CH_2Cl_2 (25 mL) was added, and the mixture was stirred at 25 °C for 2 h. TLC (petroleum ether / ethyl acetate = 5:1, V/V) showed no starting material remained. The reaction mixture was concentrated, and then purified by flash chromatography (petroleum ether / ethyl acetate = 20:1, V/V) to afford **2h** (860 mg, 83%) as brown solid (mp. 123-125 °C).

Copies of NMR spectra of synthesized compounds



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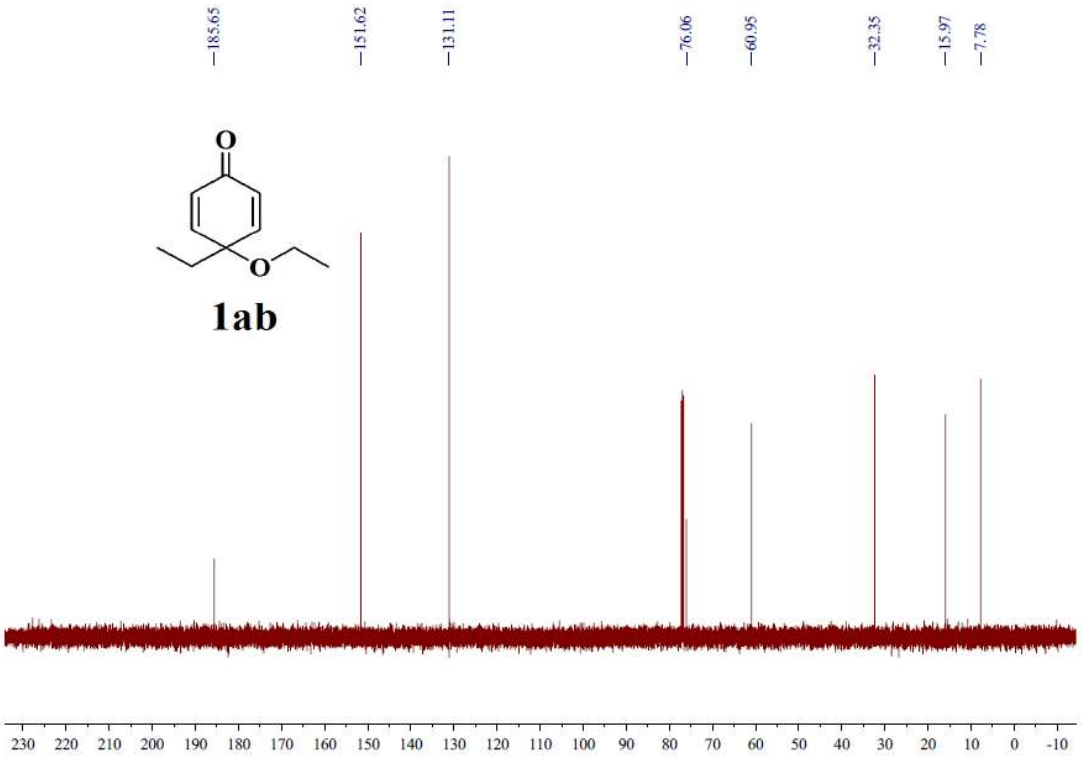
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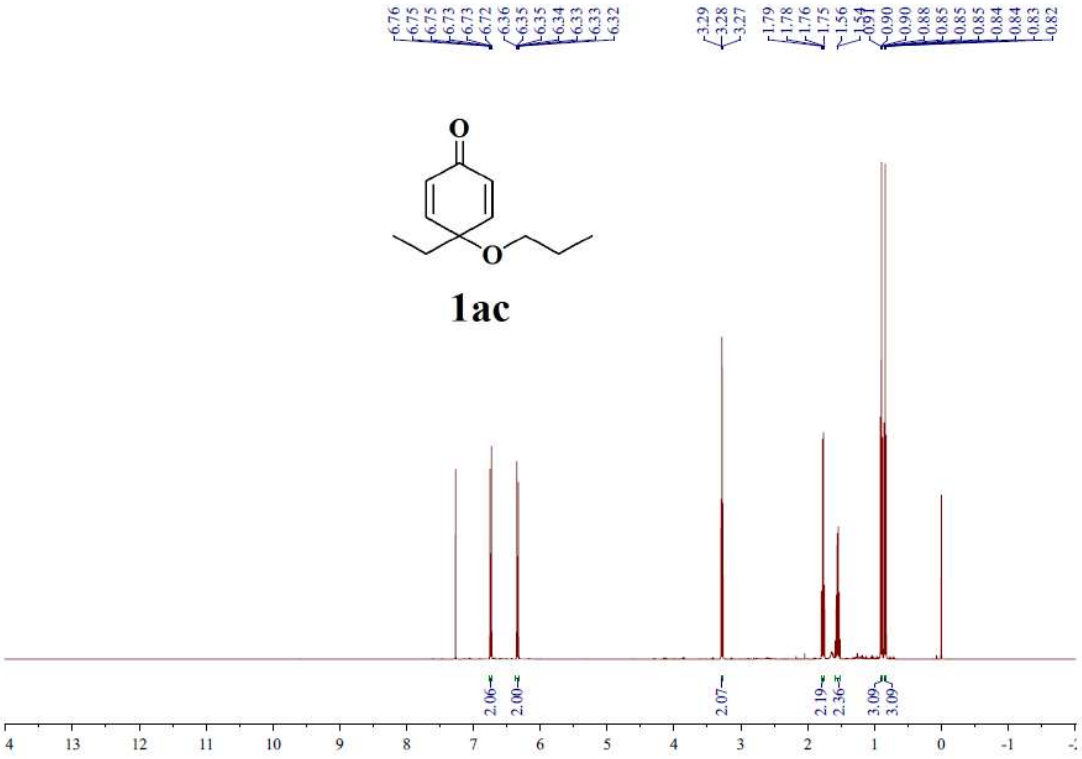
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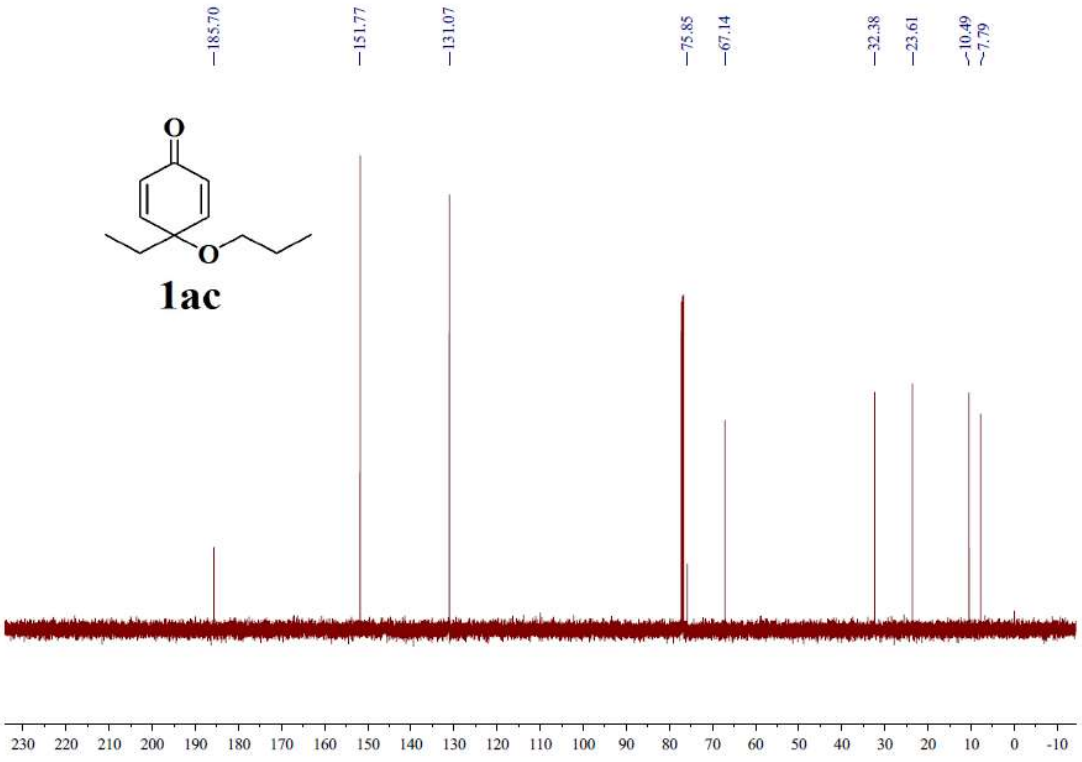
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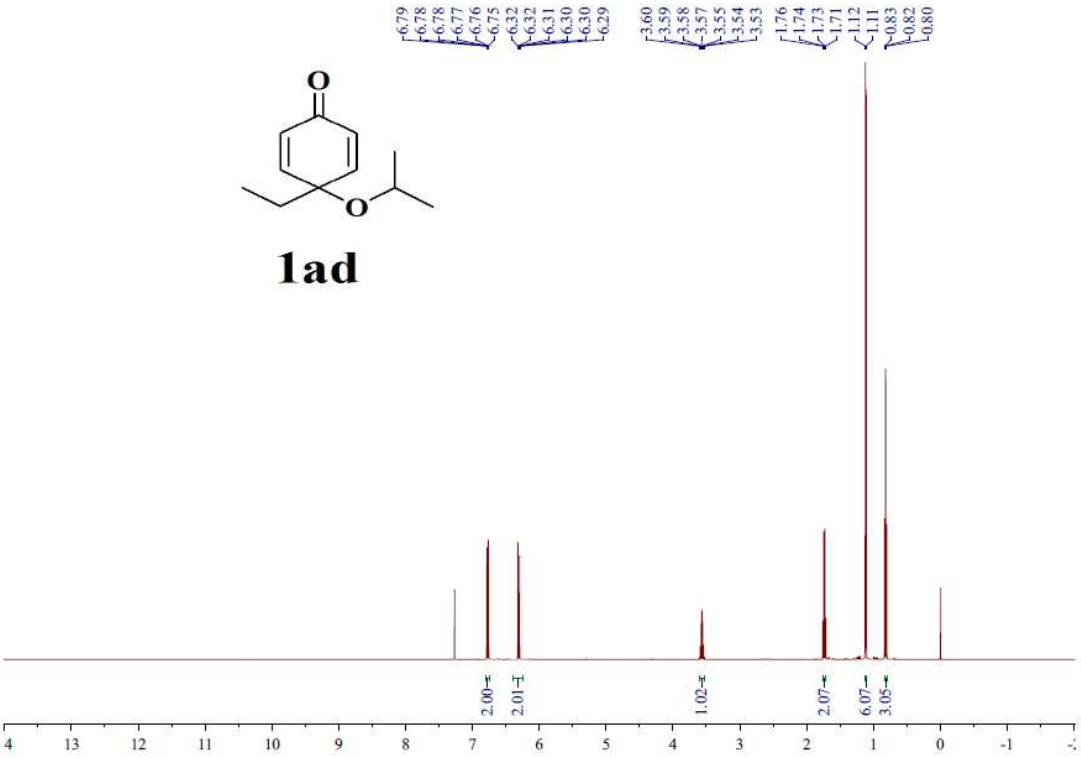
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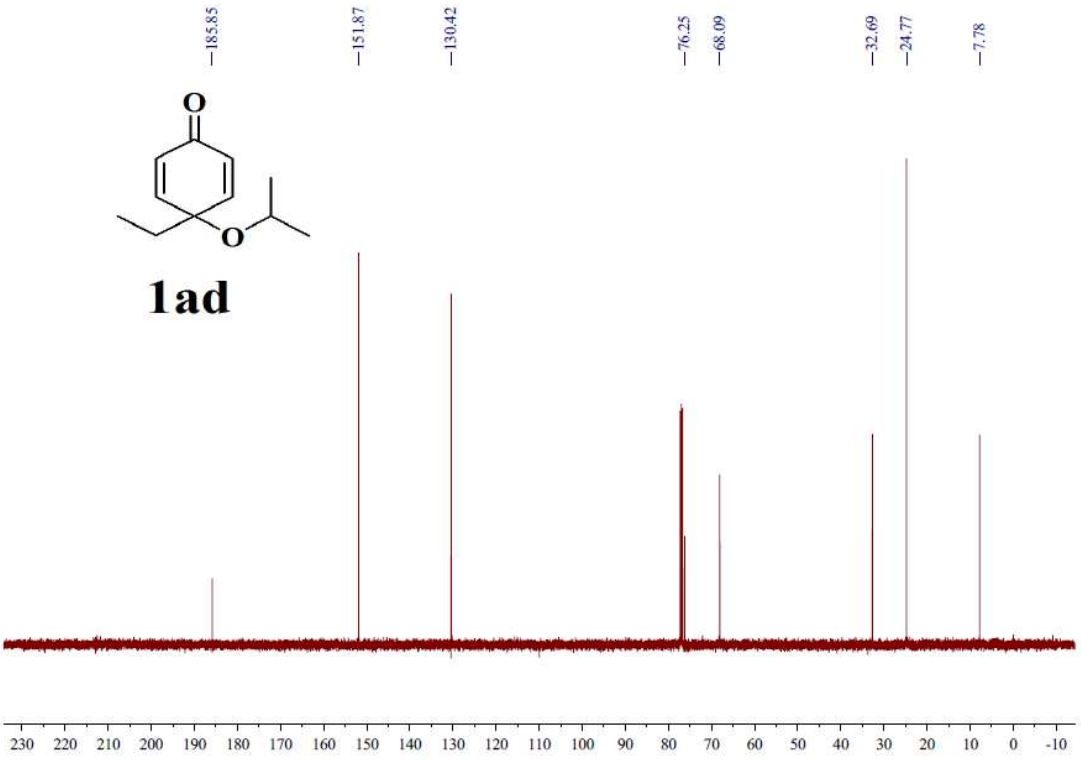
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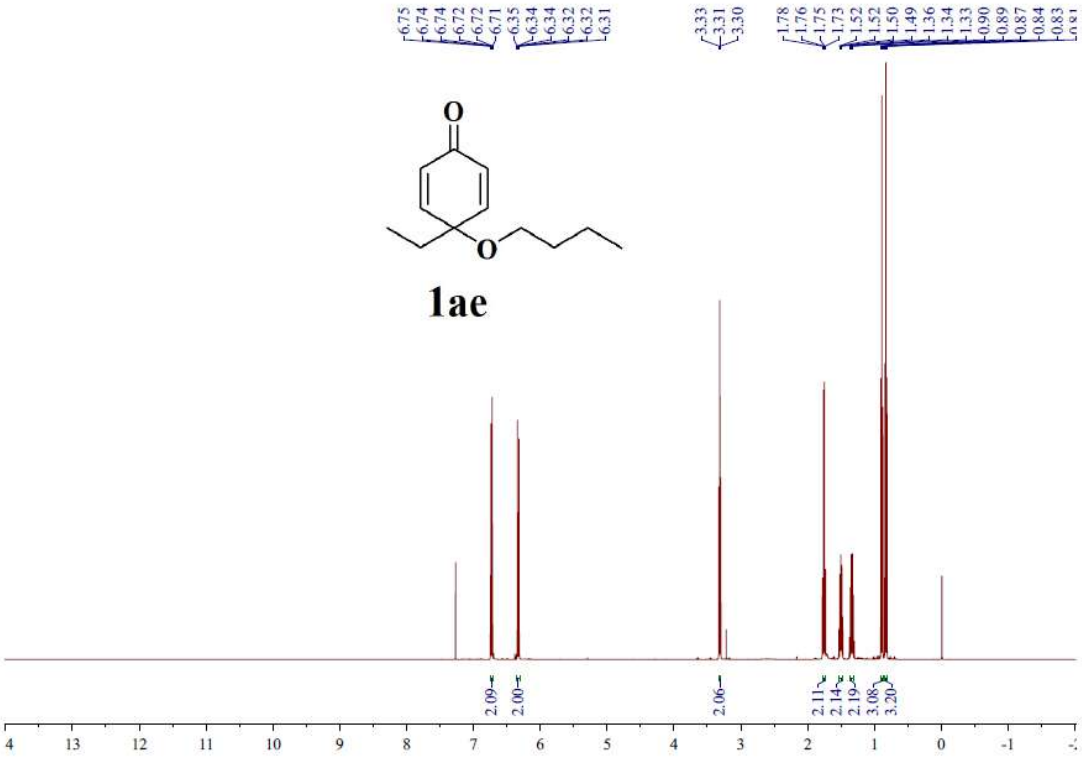
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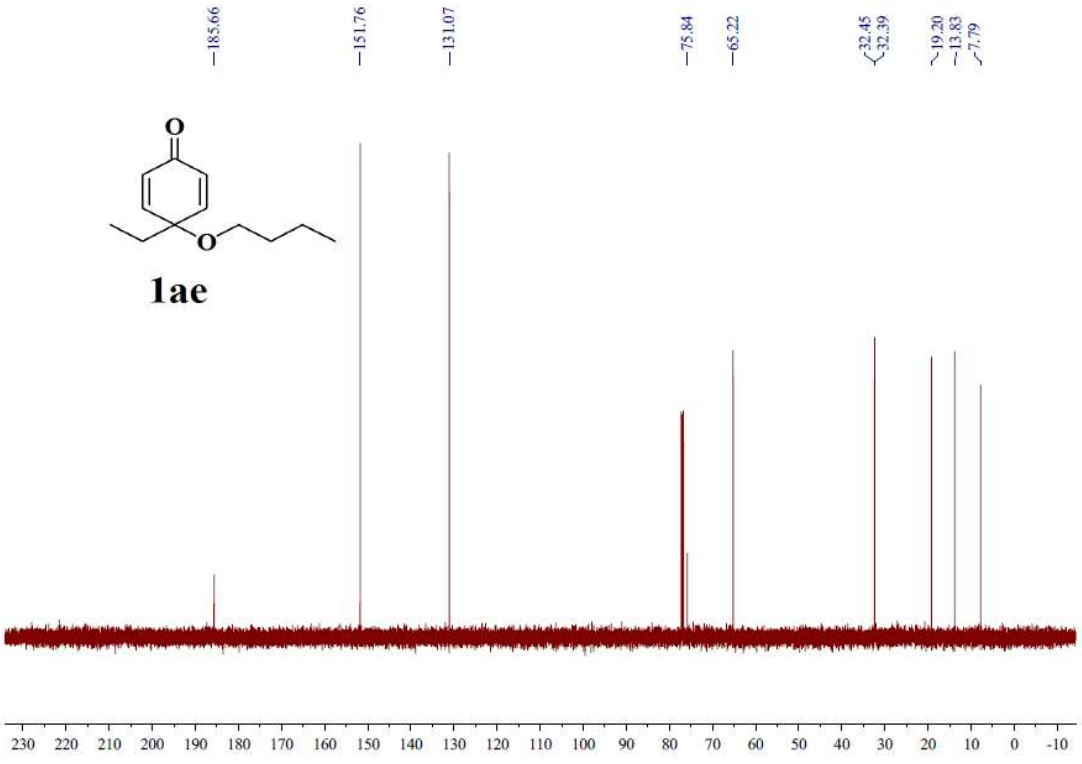
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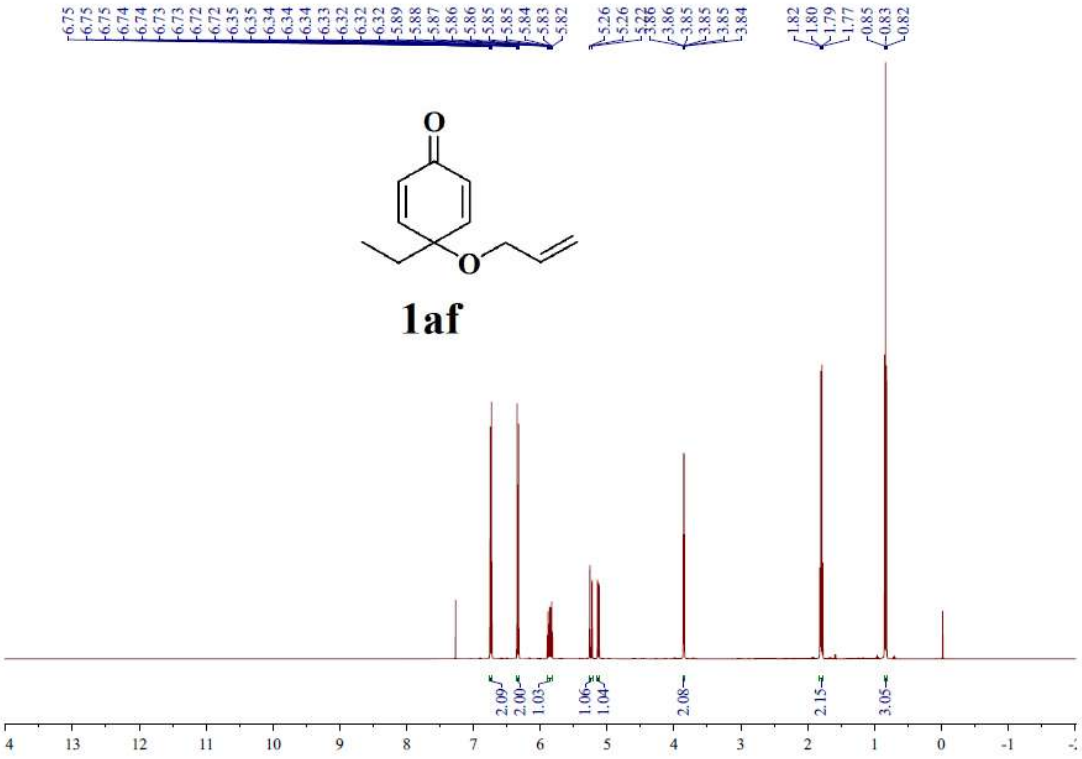
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5.85
5.85
5.84
5.83
5.82
5.26
5.26
5.22
3.86
3.86
3.85
3.85
3.85
3.84
1.82
1.80
1.79
1.77
0.85
0.83
0.82
O
O
1af
2.09
2.00
1.03
1.06
1.04
2.08
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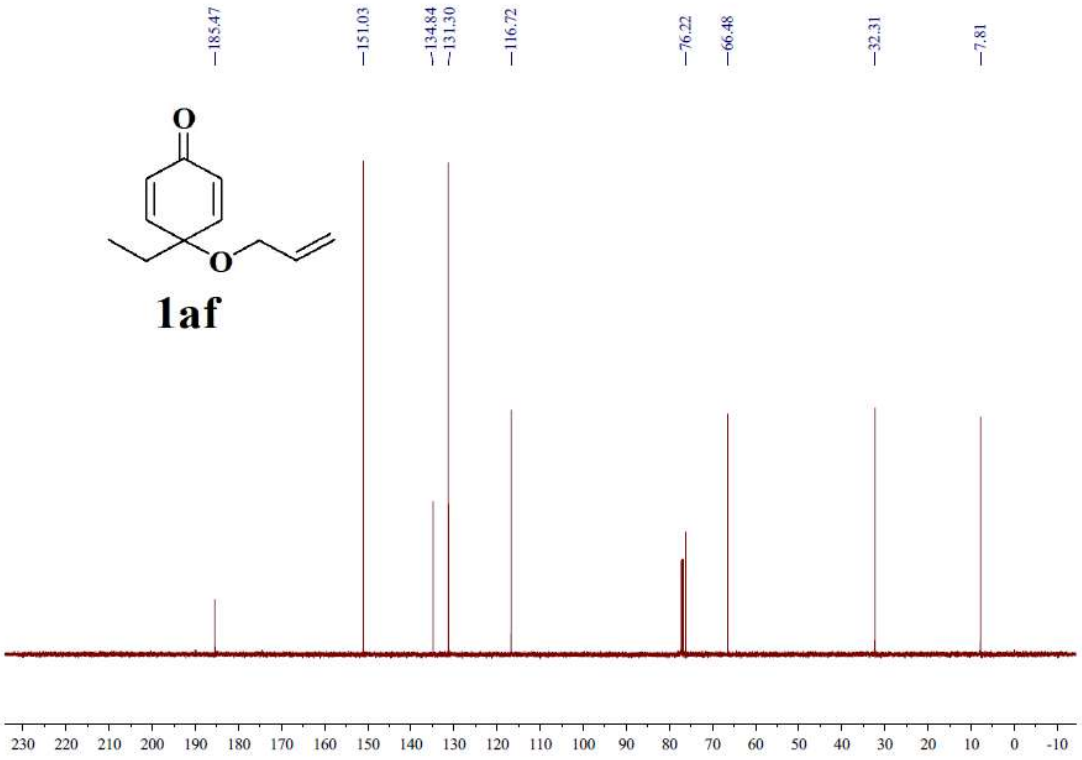
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200
190
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0
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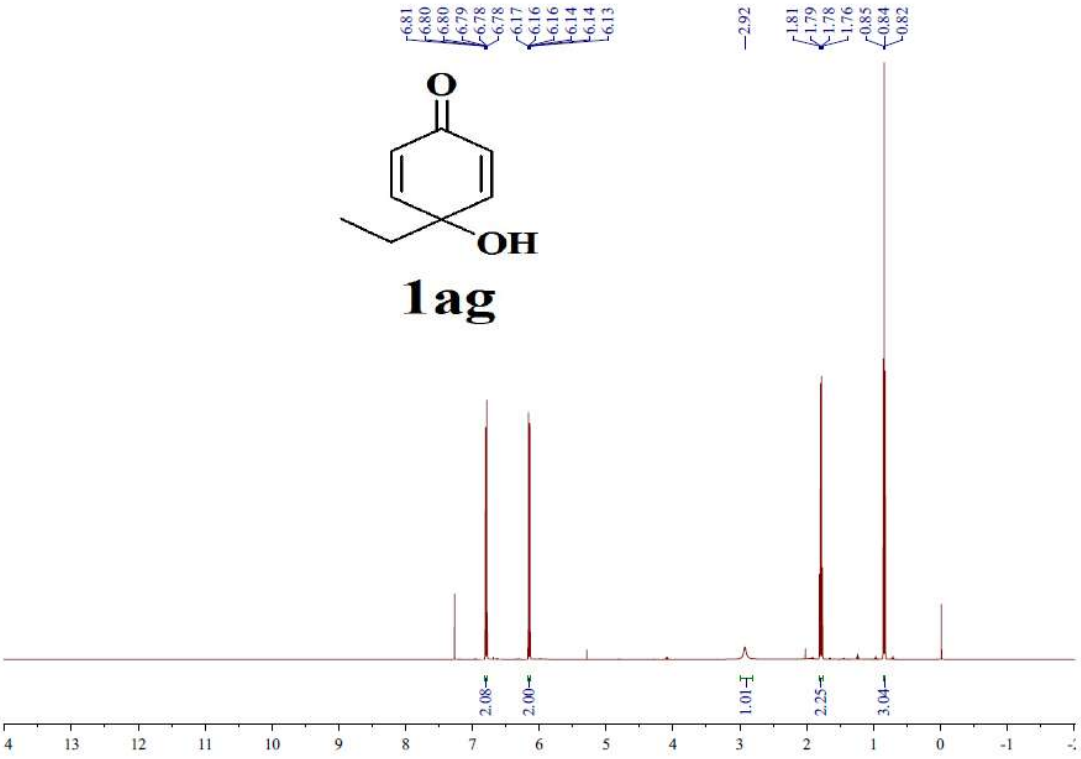
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6.14
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1.79
1.78
1.76
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0.84
0.82
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OH
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2.08
2.00
1.01
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4
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2
1
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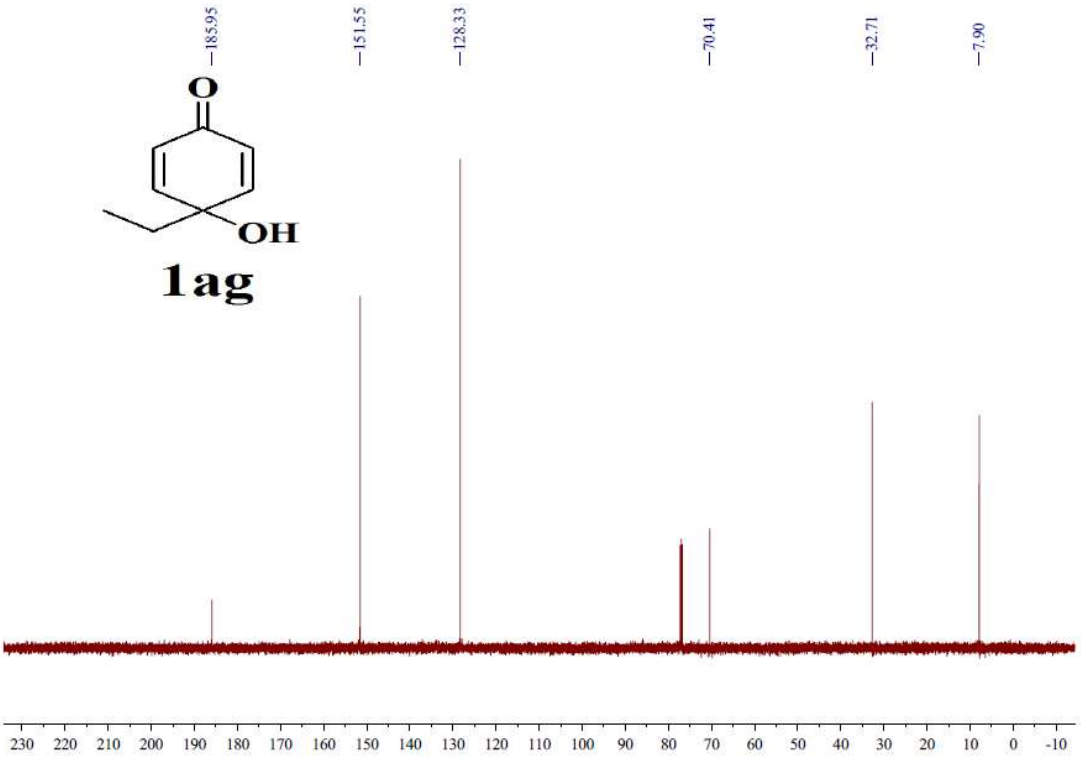
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200
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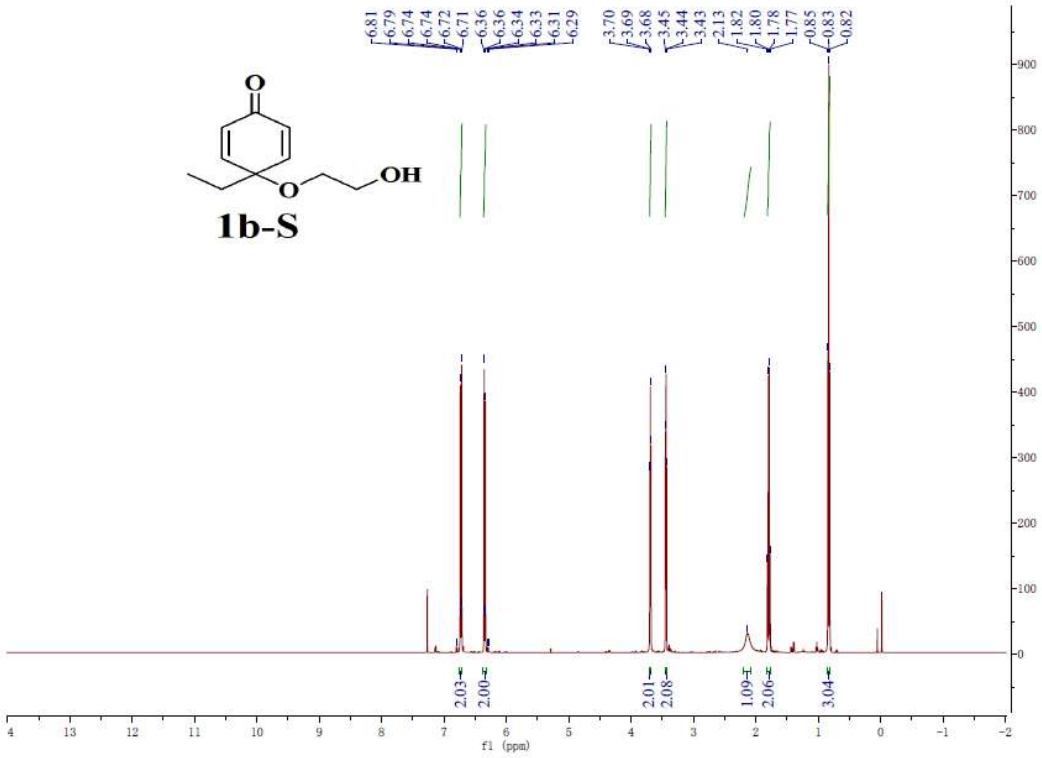
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6.31
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3.70
3.69
3.68
3.45
3.44
3.43
2.13
1.82
1.80
1.78
1.77
0.85
0.83
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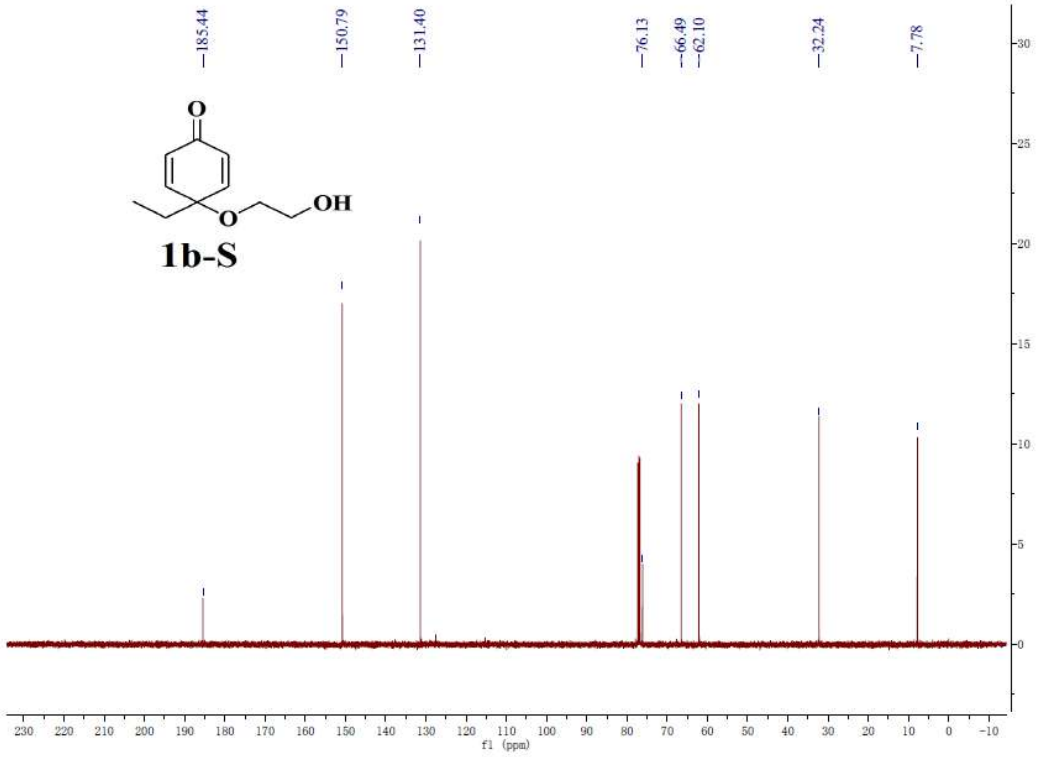
O
1b-S
2.03
2.00
2.01
2.08
1.09
2.06
3.04
f1 (ppm)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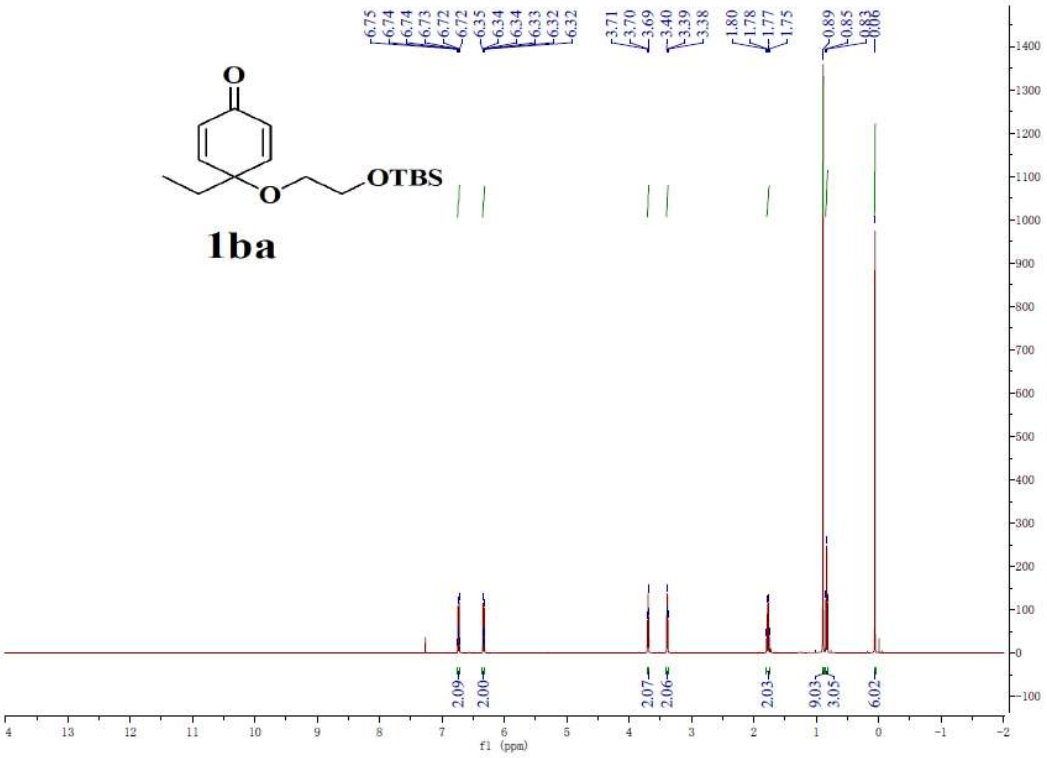
OH
O
1b-S
f1 (ppm)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6.32
6.32
3.71
3.70
3.69
3.40
3.39
3.38
1.80
1.78
1.77
1.75
0.89
0.85
0.83
0.06
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O
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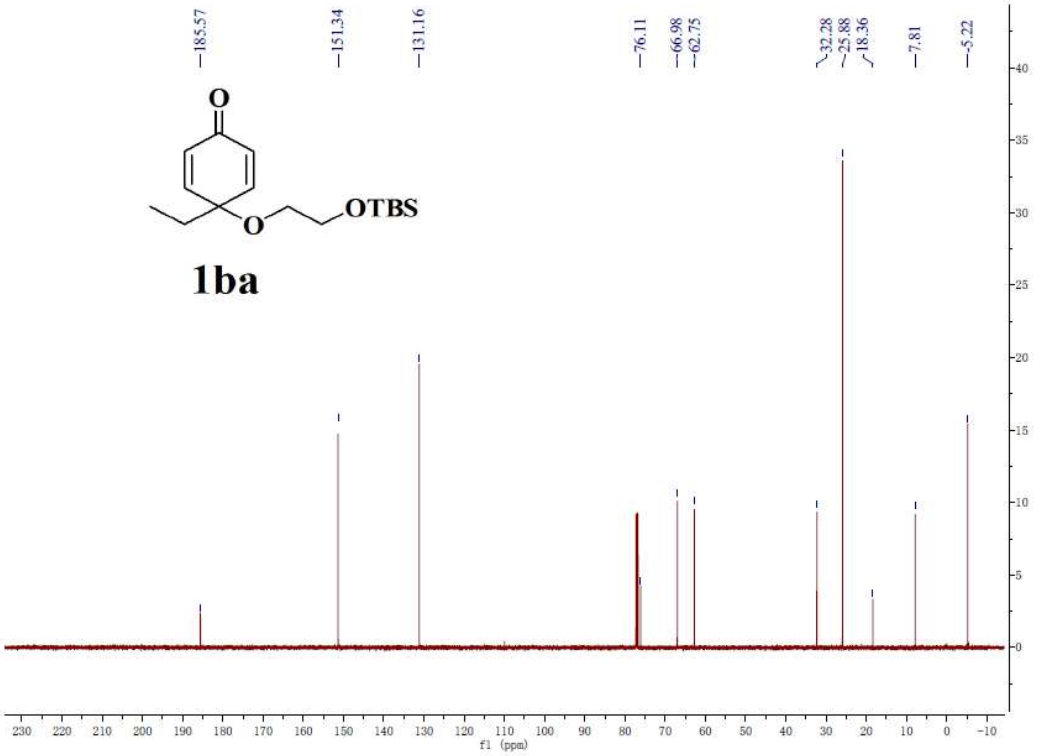
2.09
2.00
2.07
2.06
2.03
9.03
3.05
6.02
f1 (ppm)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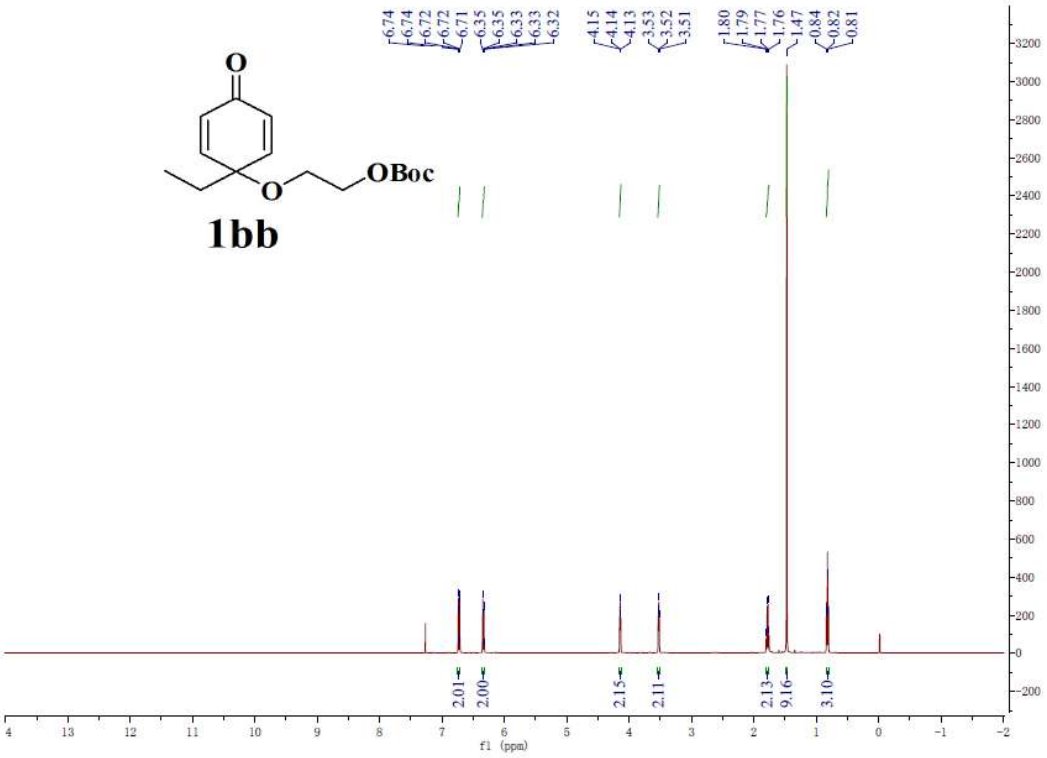
18.36
7.81
-5.22
O
O
OTBS
1ba
f1 (ppm)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6.72
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6.35
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6.33
6.33
6.32
4.15
4.14
4.13
3.53
3.52
3.51
1.80
1.79
1.77
1.76
1.47
0.84
0.82
0.81
O
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OBoc
1bb
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2.00
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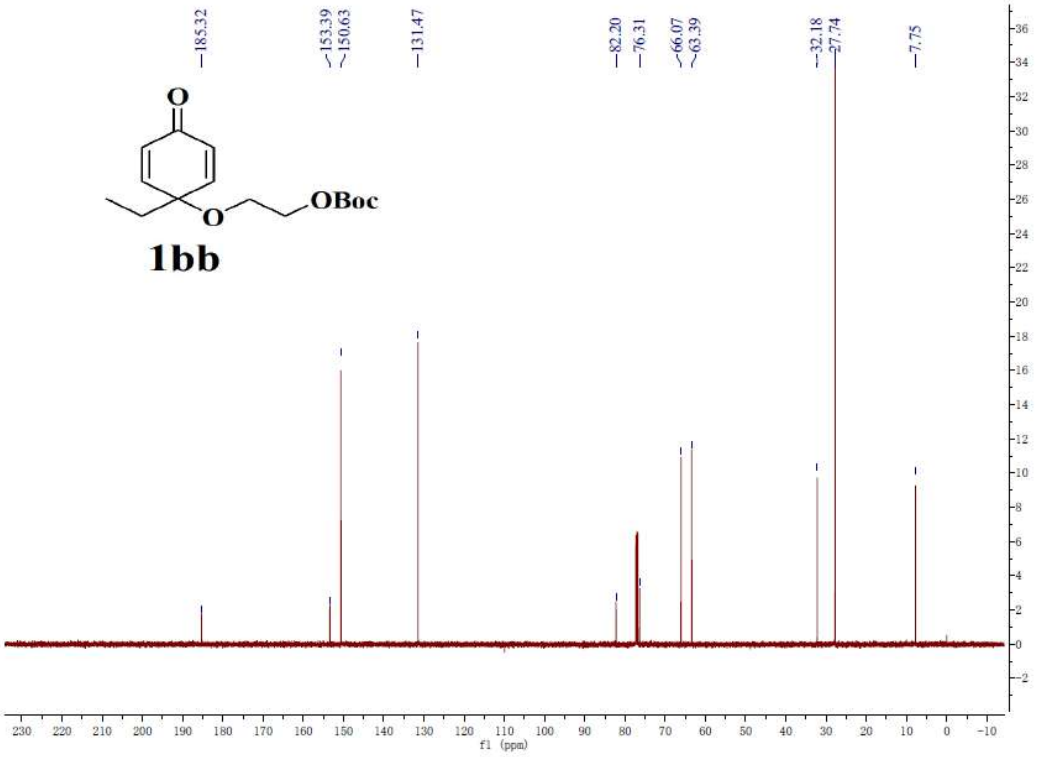
2.11
2.13
9.16
3.10
f1 (ppm)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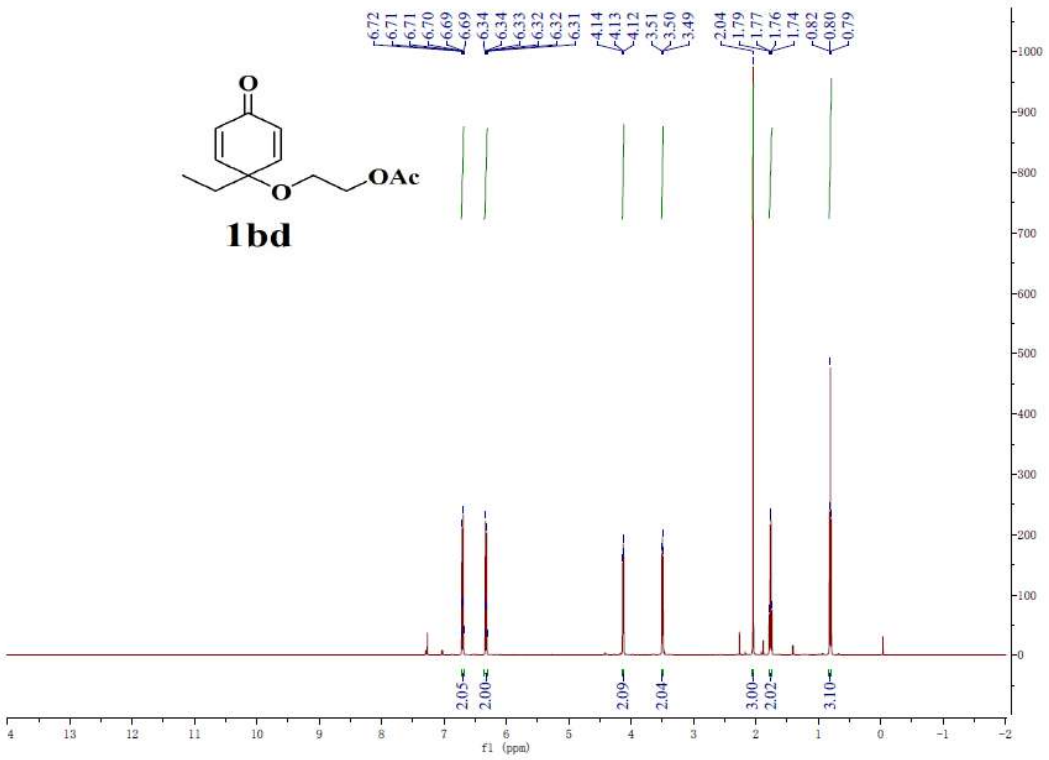
27.74
7.75
O
O
OBoc
1bb
f1 (ppm)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6.69
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6.34
6.33
6.32
6.32
6.31
4.14
4.13
4.12
3.51
3.50
3.49
2.04
1.79
1.77
1.76
1.74
0.82
0.80
0.79
O
O
OAc
1bd
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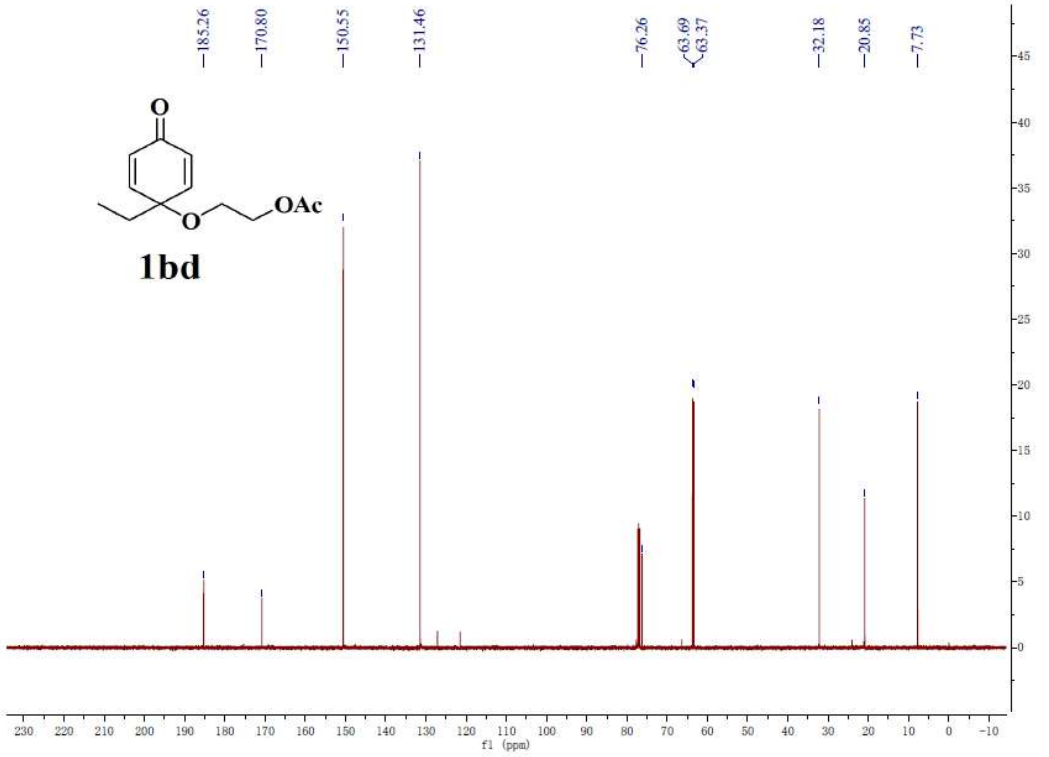
2.00
2.09
2.04
3.00
2.02
3.10
f1 (ppm)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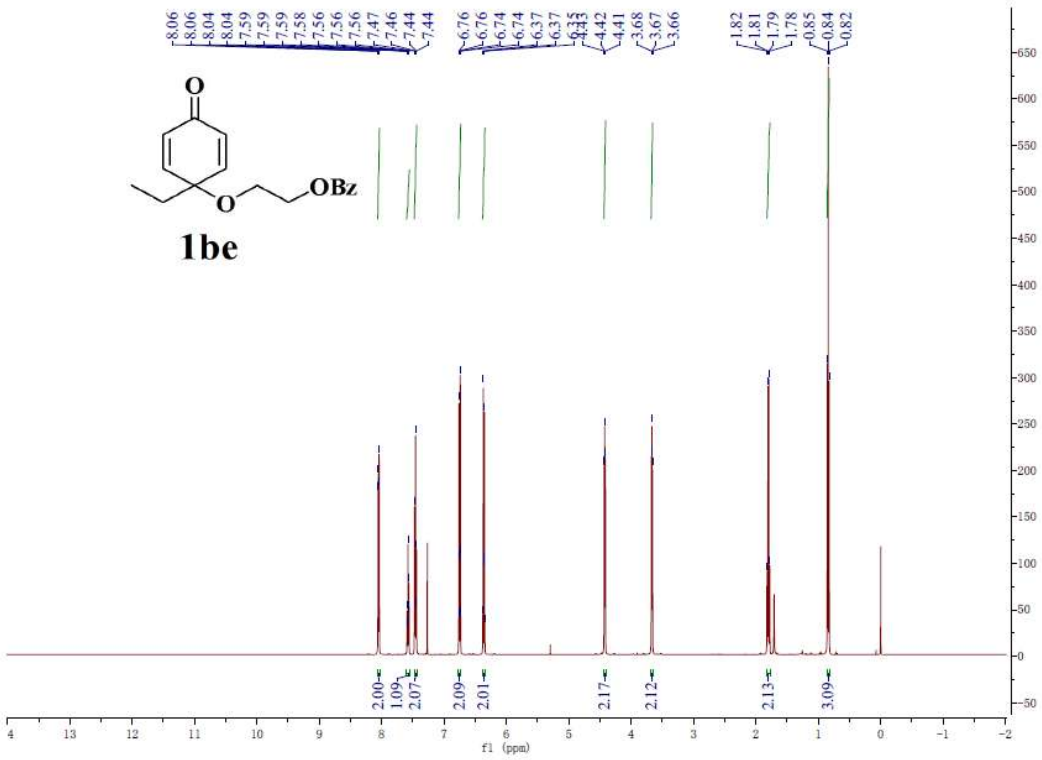
O
OAc
1bd
f1 (ppm)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8.04
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7.59
7.59
7.58
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7.56
7.56
7.47
7.46
7.44
7.44
6.76
6.76
6.74
6.74
6.37
6.37
4.42
4.41
3.68
3.67
3.66
1.82
1.81
1.79
1.78
0.85
0.84
0.82
O
OBz
O
1be
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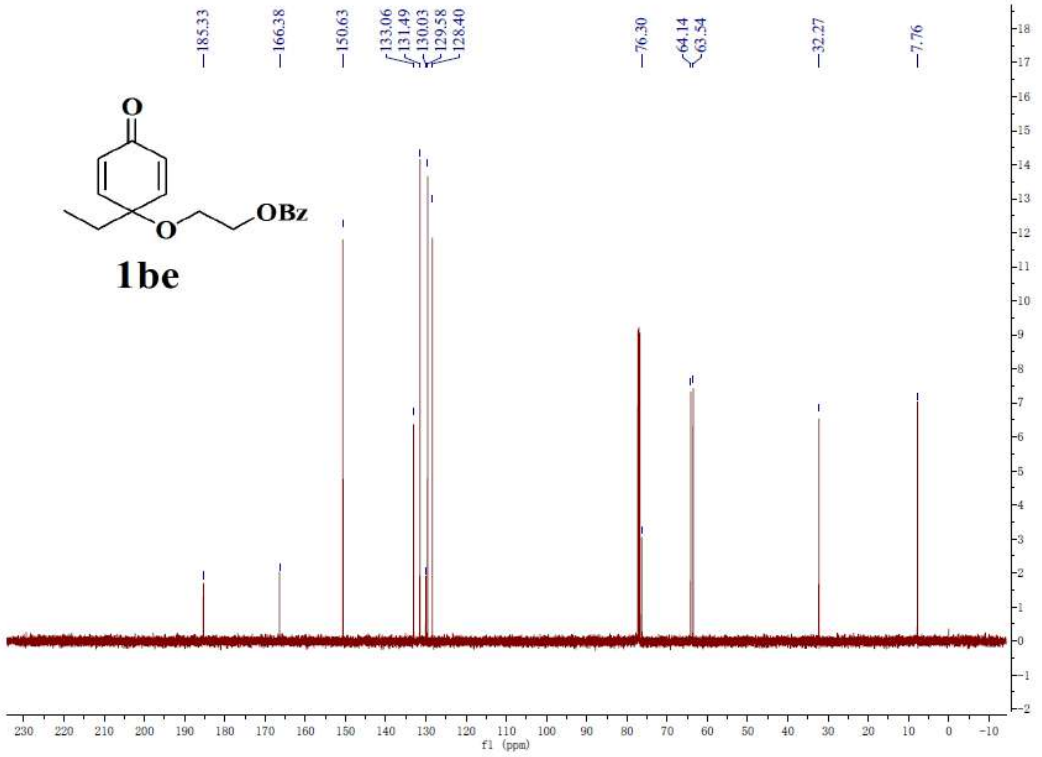
1.09
2.07
2.09
2.01
2.17
2.12
2.13
3.09
f1 (ppm)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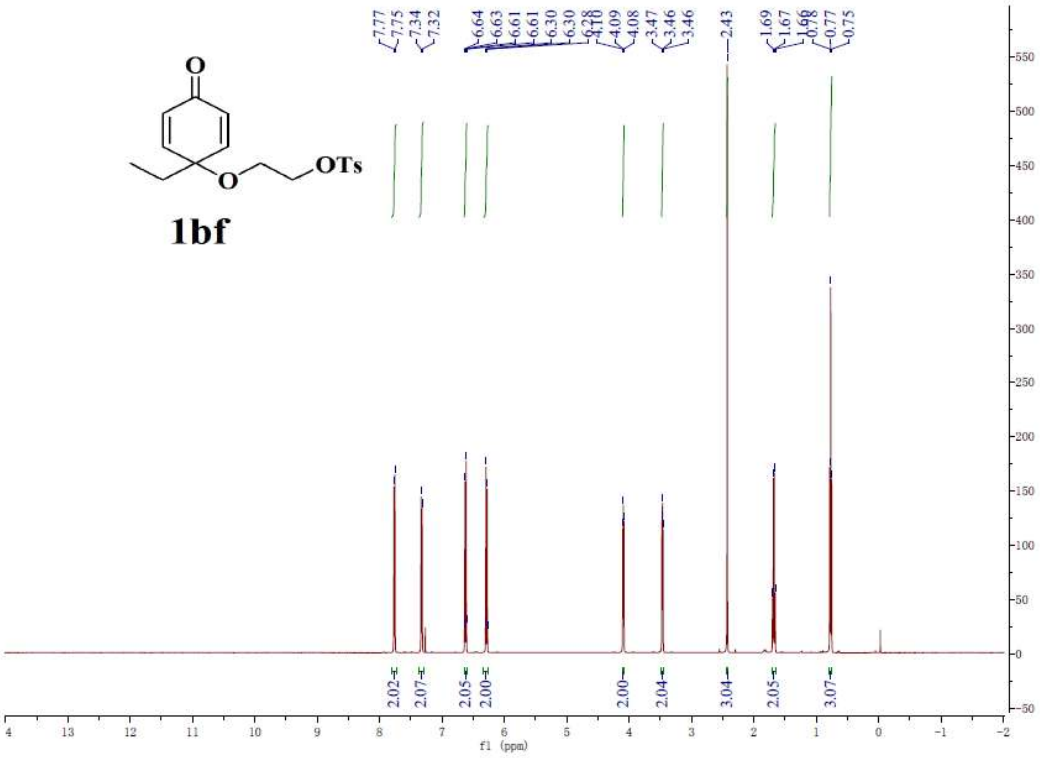
32.27
7.76
O
OBz
O
1be
f1 (ppm)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4.08
3.47
3.46
3.46
2.43
1.69
1.67
0.78
0.77
0.75
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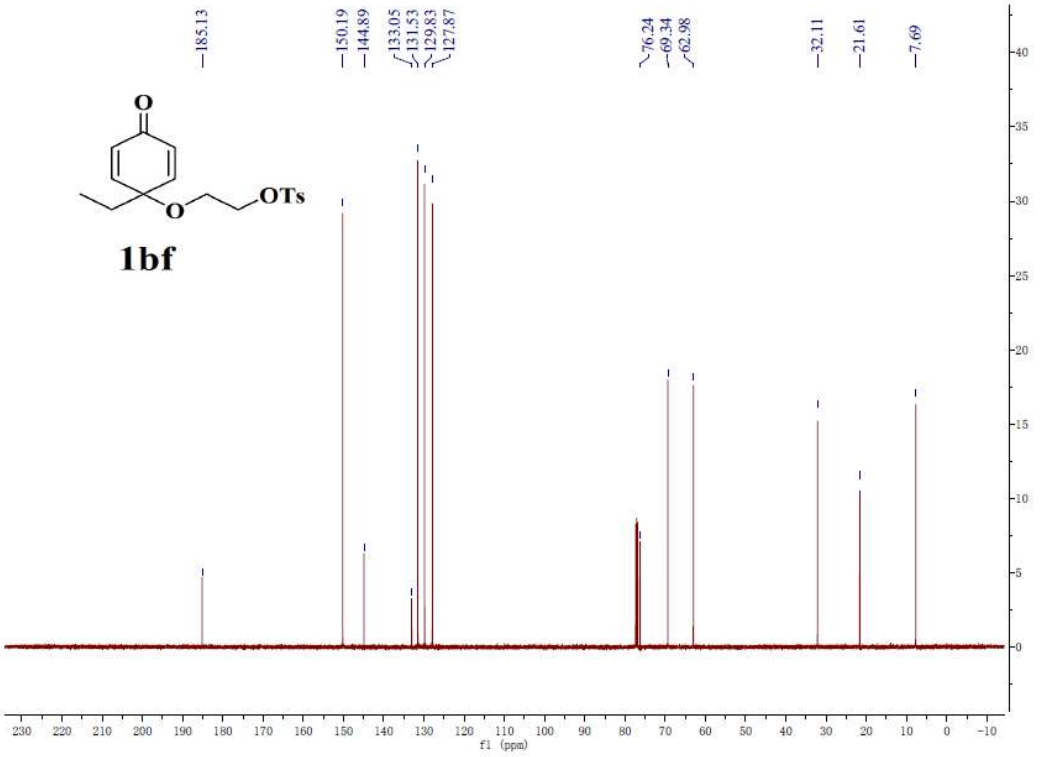
2.05
2.00
2.00
2.04
3.04
2.05
3.07
f1 (ppm)
OTs
1bf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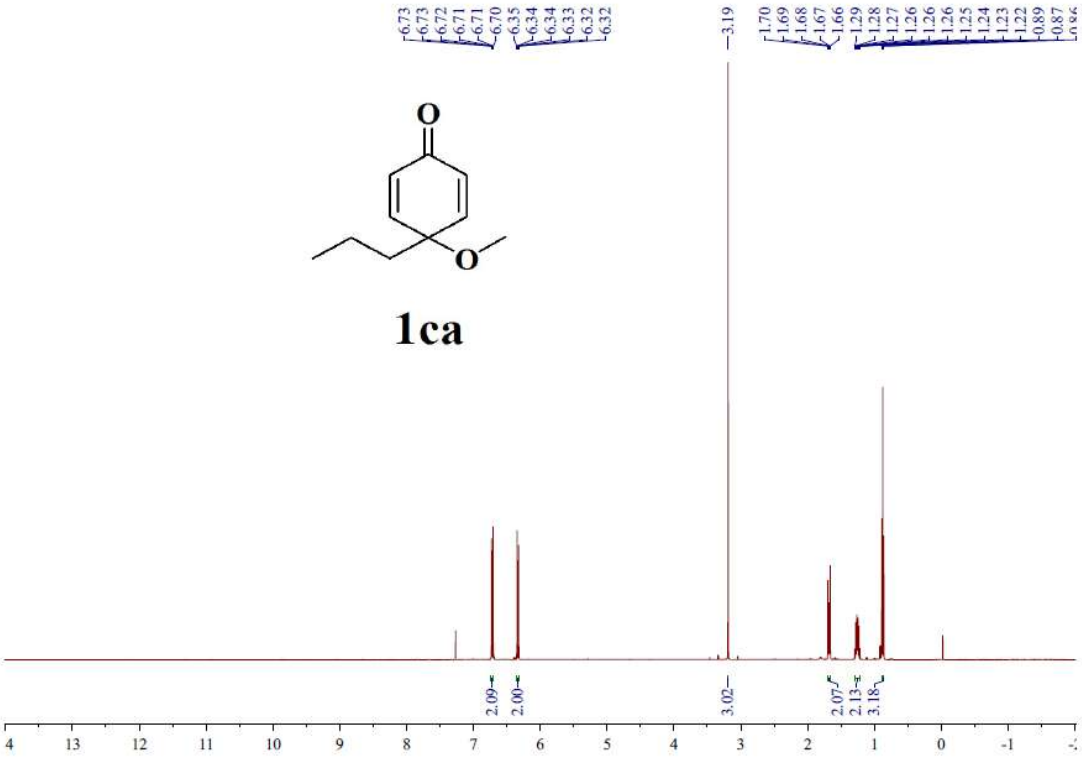
f1 (ppm)
OTs
1bf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6.32
6.32
3.19
1.70
1.69
1.68
1.67
1.66
1.29
1.28
1.27
1.26
1.26
1.26
1.25
1.24
1.23
1.22
0.89
0.87
O
O
1ca
2.09
2.00
3.02
2.07
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4
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5
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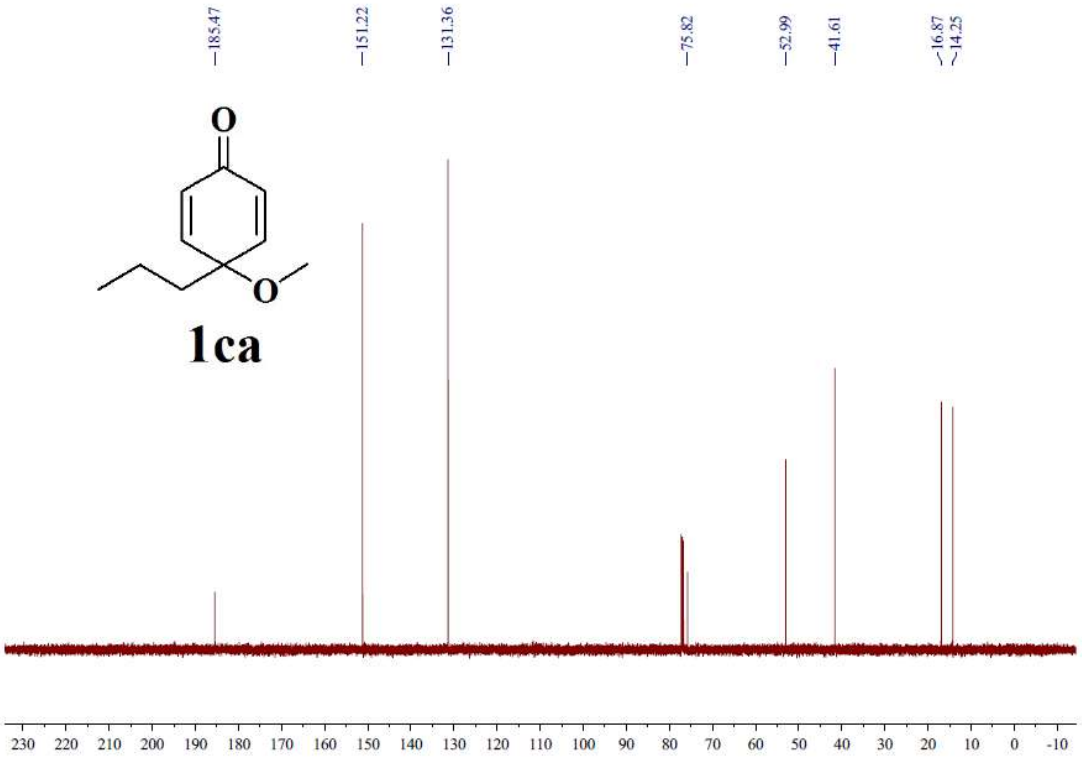
0
-1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O
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200
190
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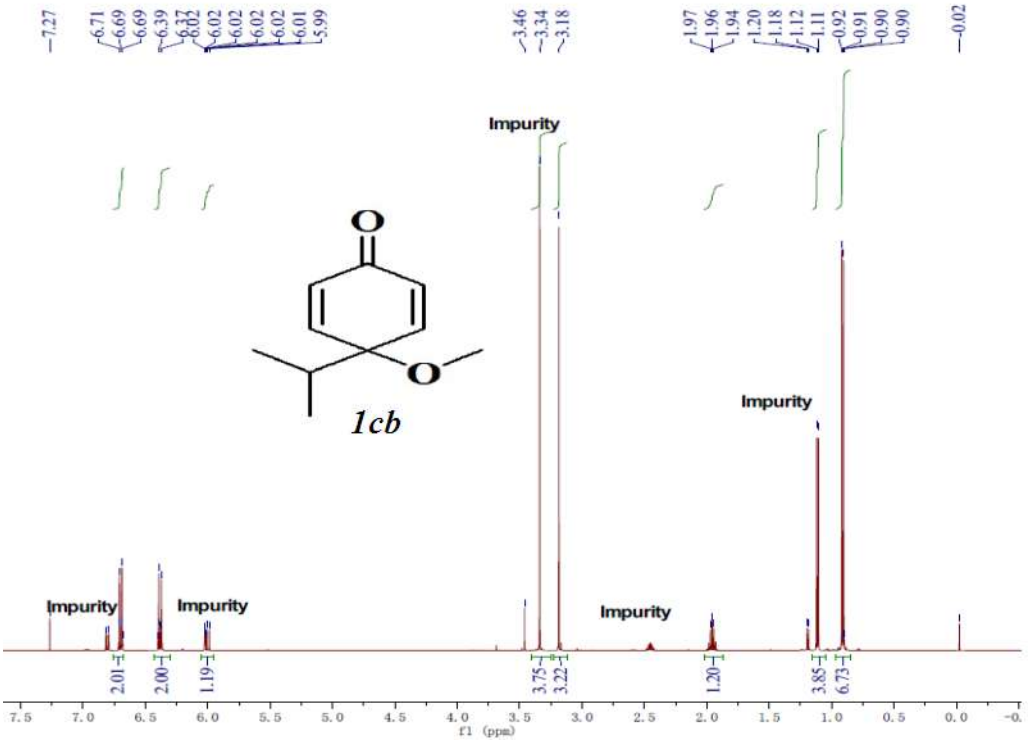
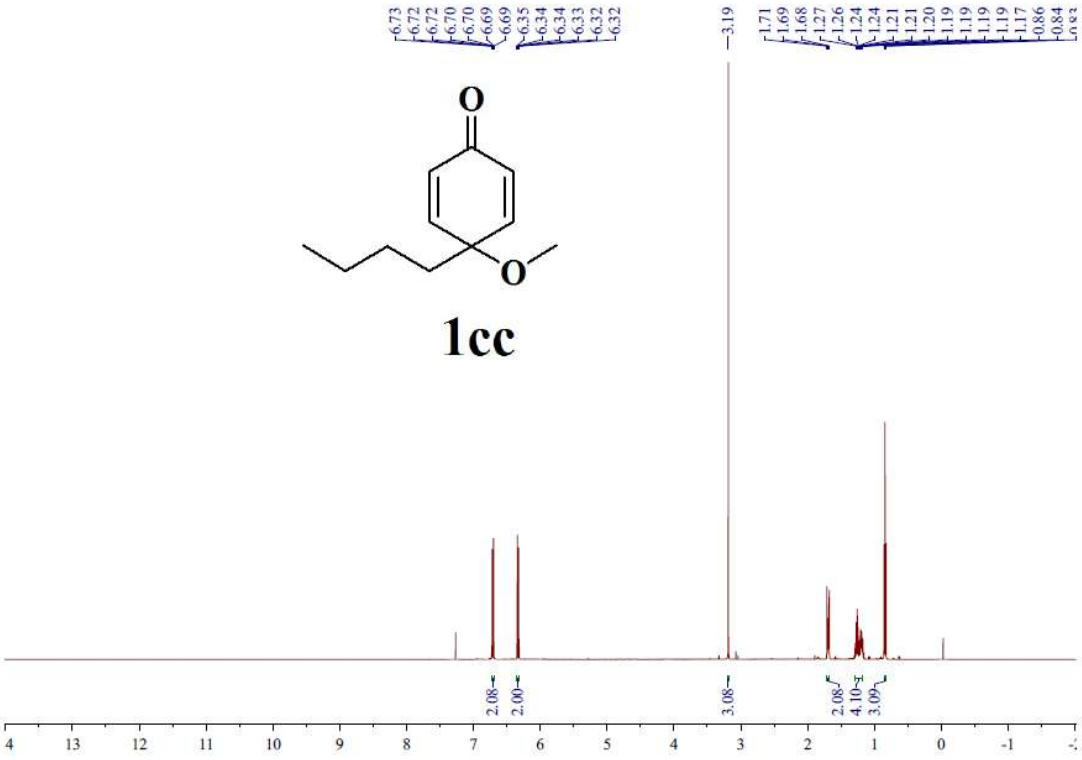
0
-10


Impurity
Impurity
Impurity
Impurity
Impurity
1cb
f1 (ppm)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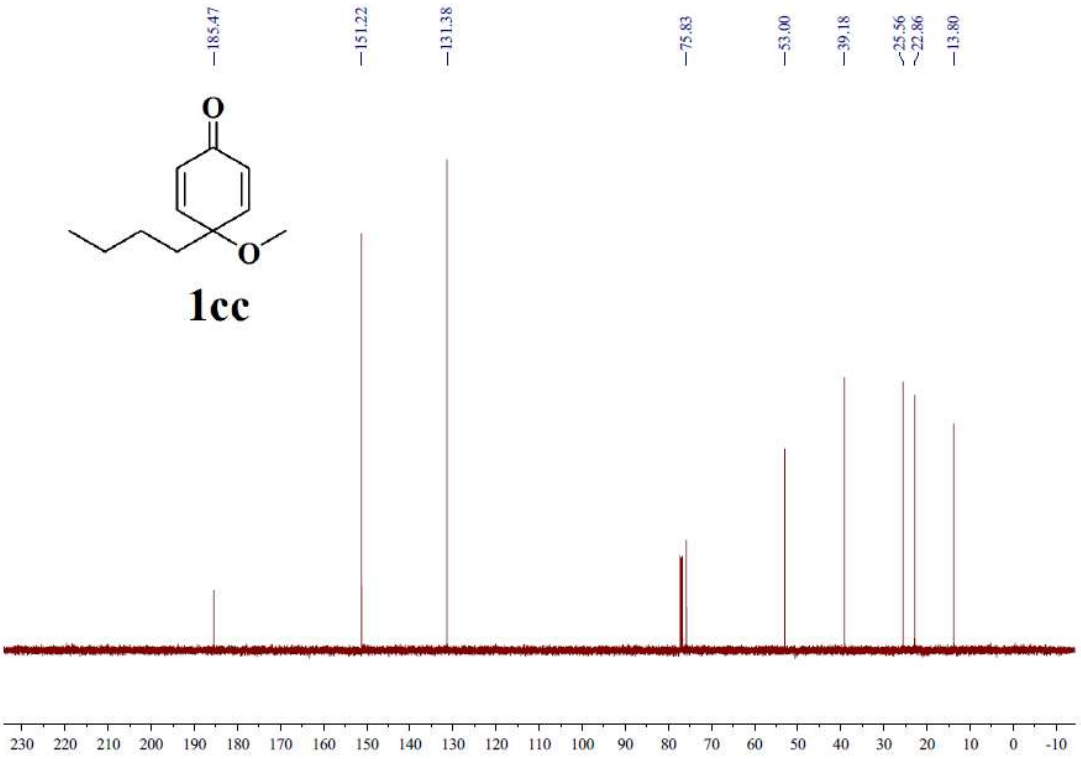
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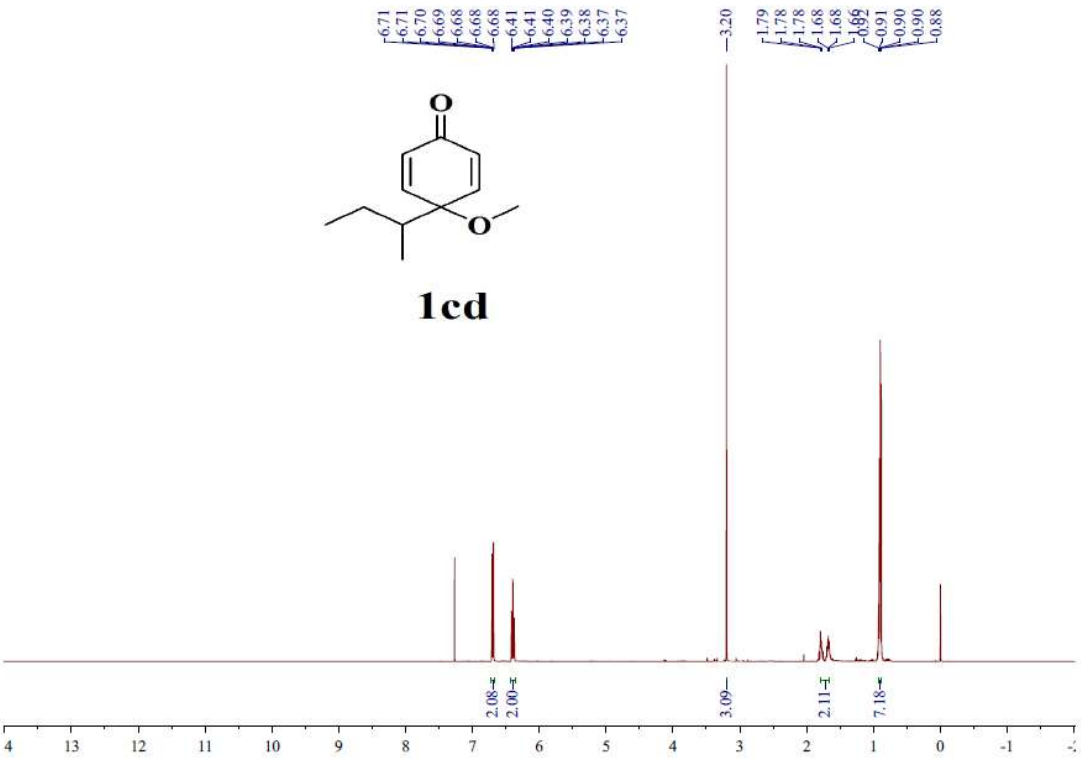
O
1cc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6.38
6.37
6.37
3.20
1.79
1.78
1.78
1.68
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0.90
0.88
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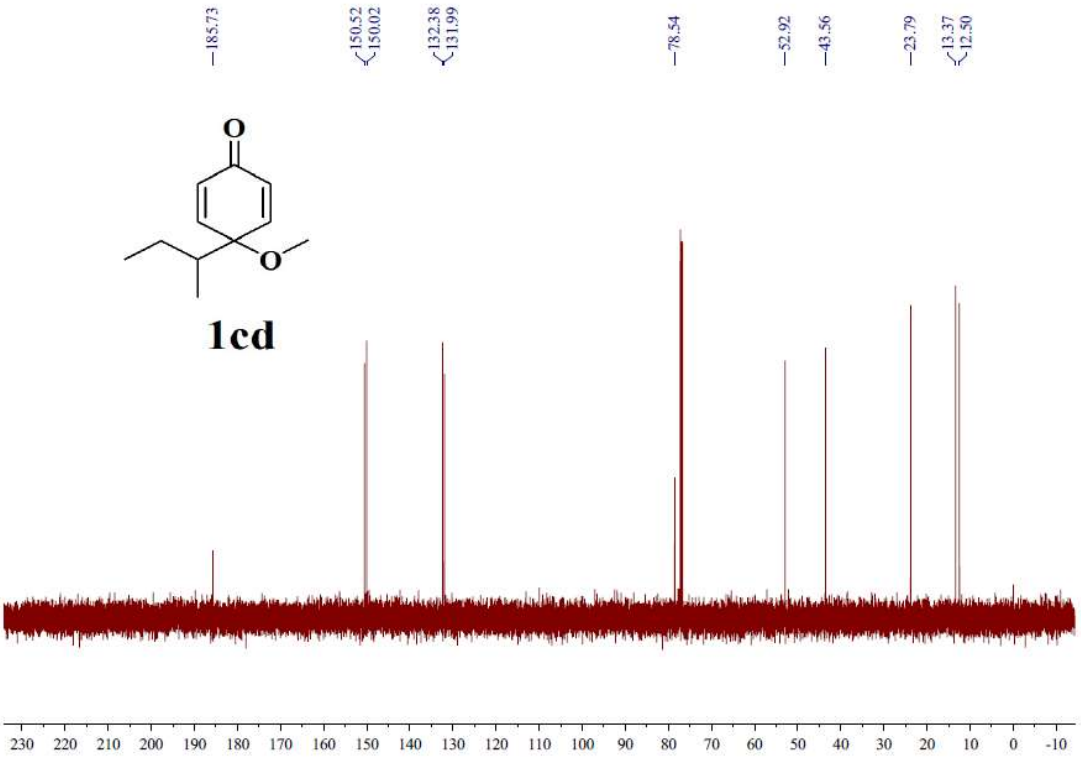
3.09
2.11
7.18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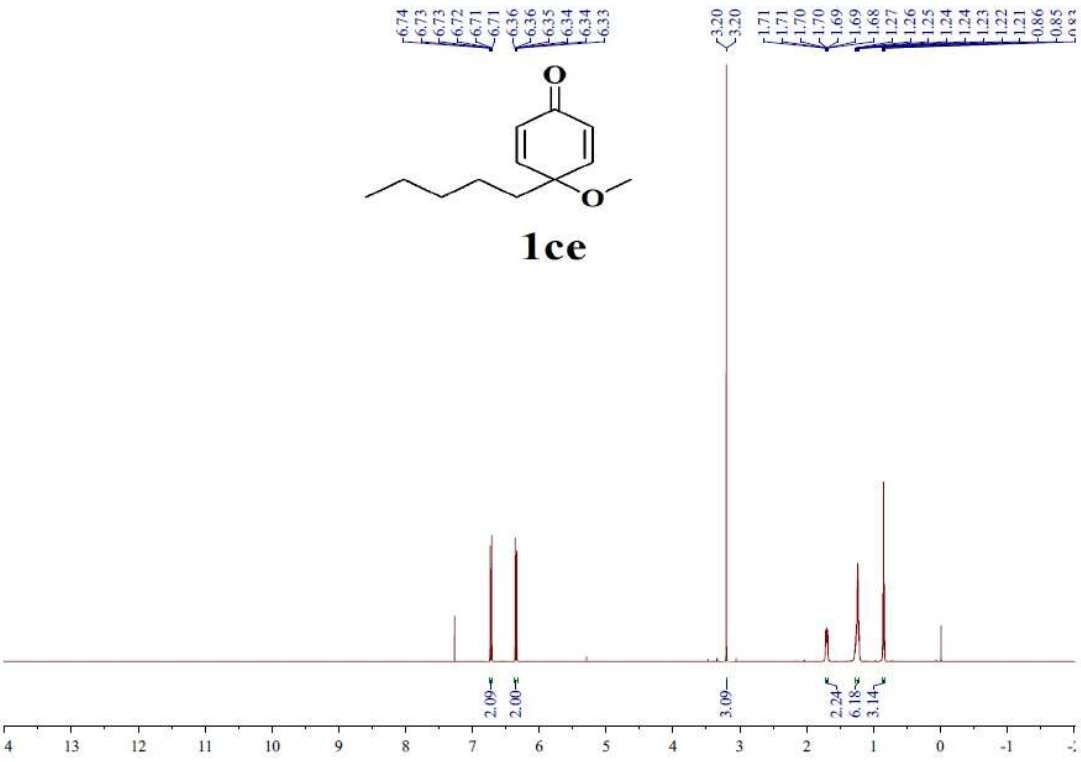
O
1cd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6.33
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3.20
1.71
1.71
1.70
1.70
1.69
1.69
1.68
1.27
1.26
1.25
1.24
1.24
1.23
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2.00
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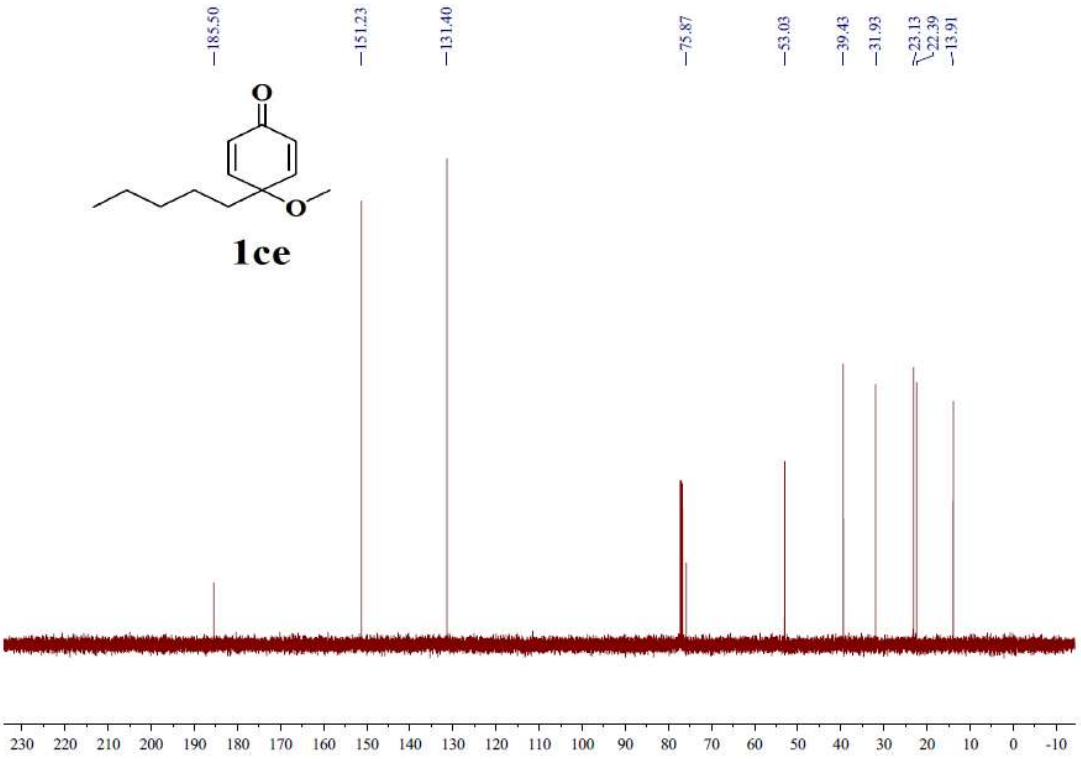
3.14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22.39
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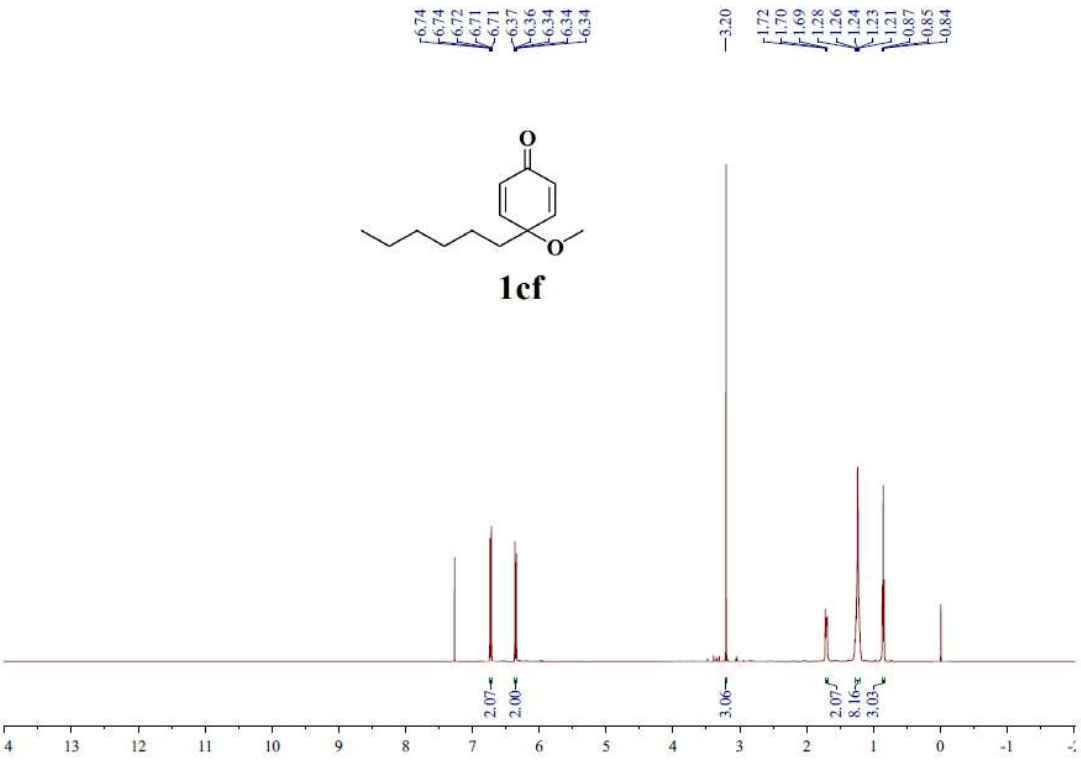
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1.70
1.69
1.28
1.26
1.24
1.23
1.21
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0.85
0.84
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3.06
2.07
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3.03
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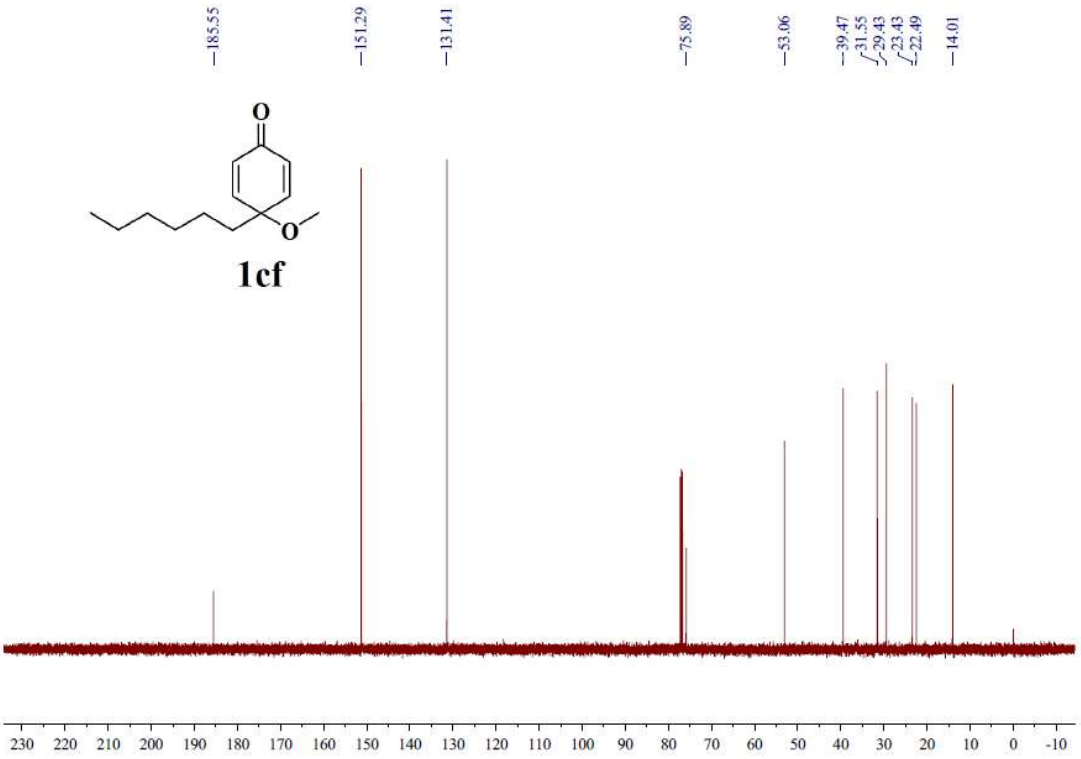
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O
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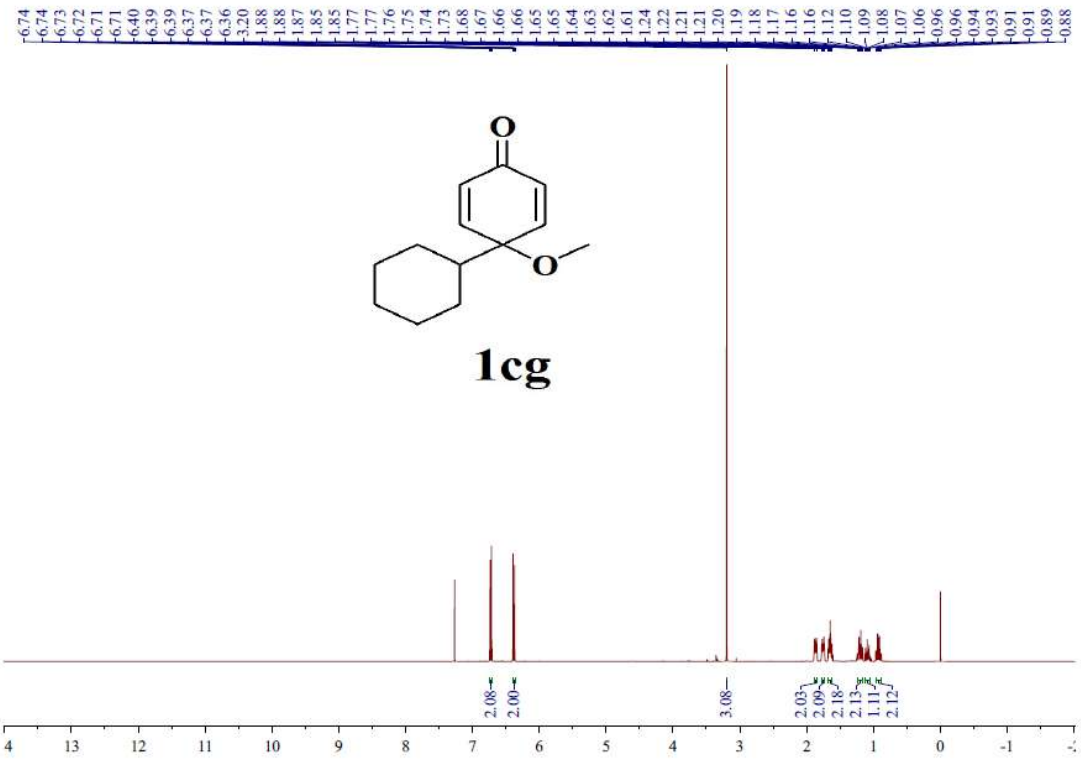
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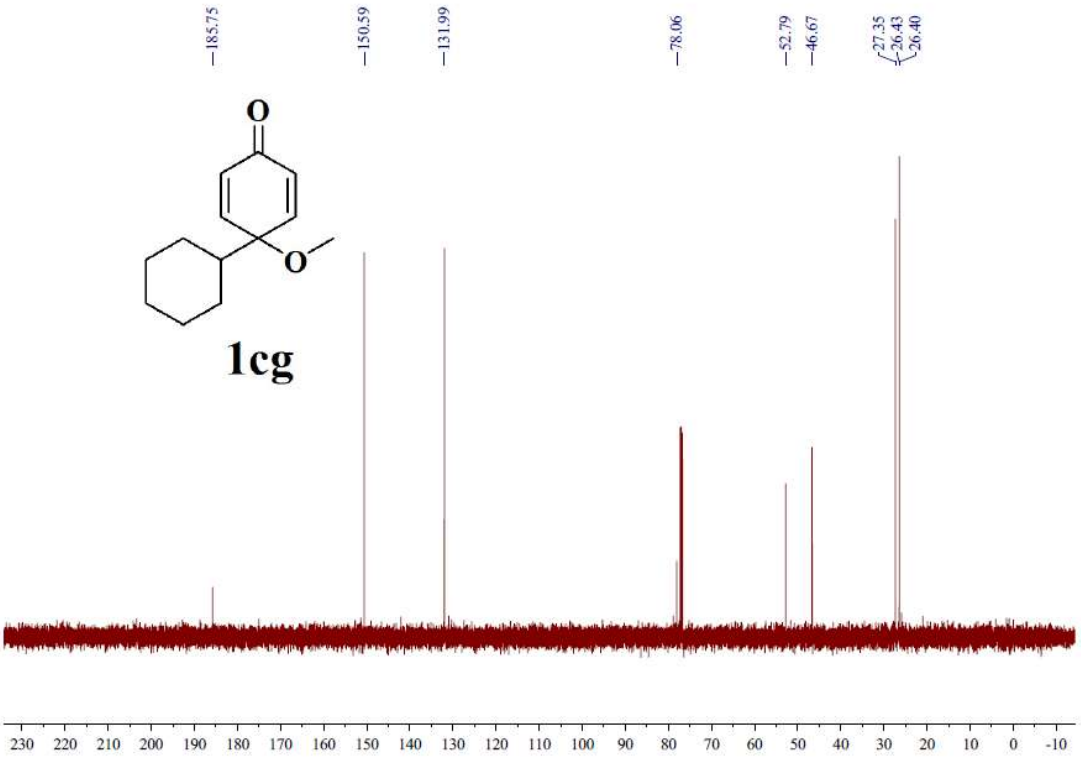
1.11
2.12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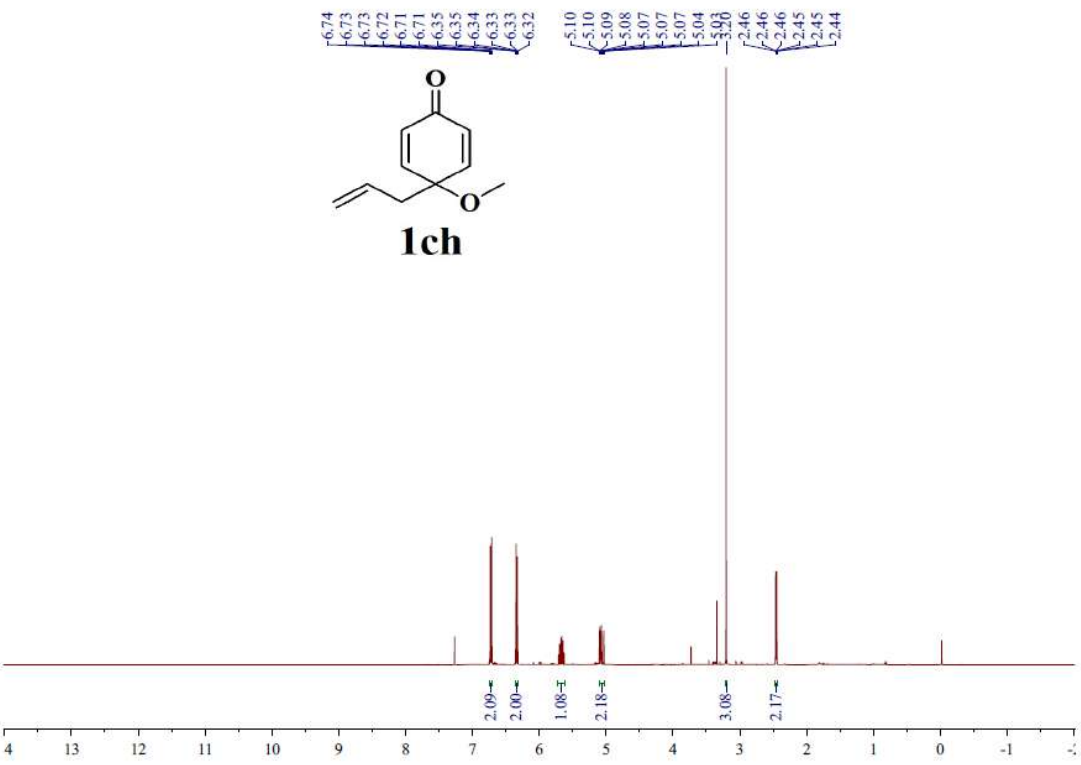
1cg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5.10
5.10
5.09
5.08
5.07
5.07
5.07
5.04
3.20
2.46
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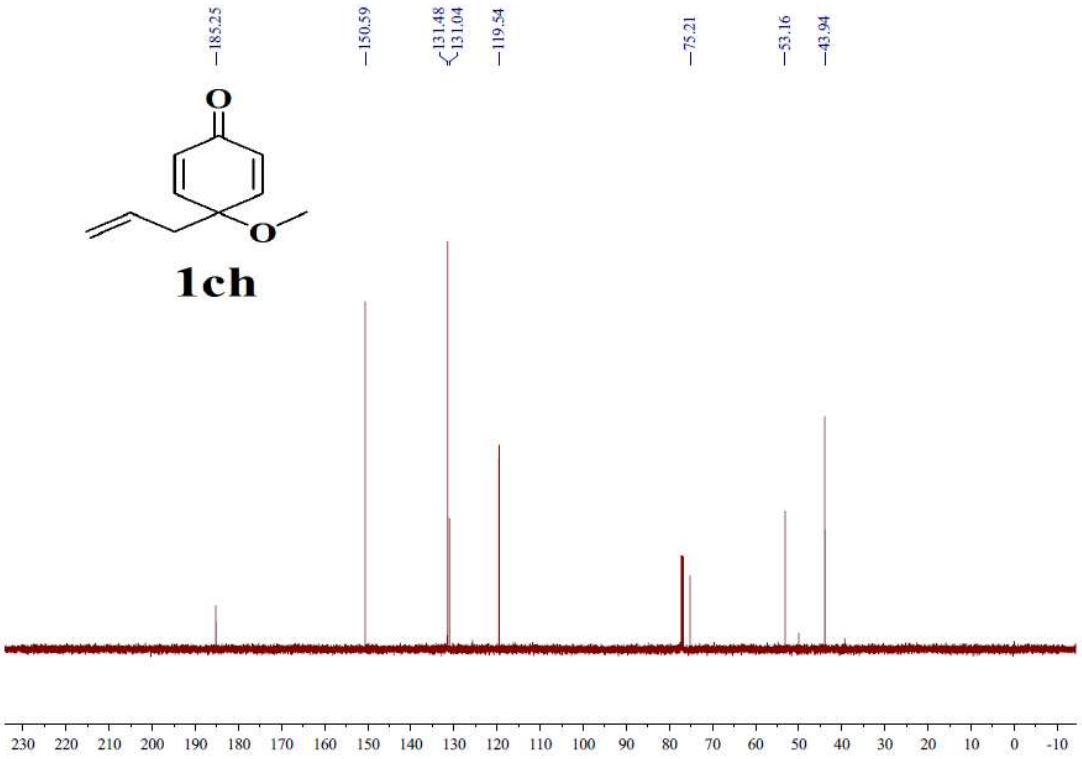
3.08
2.17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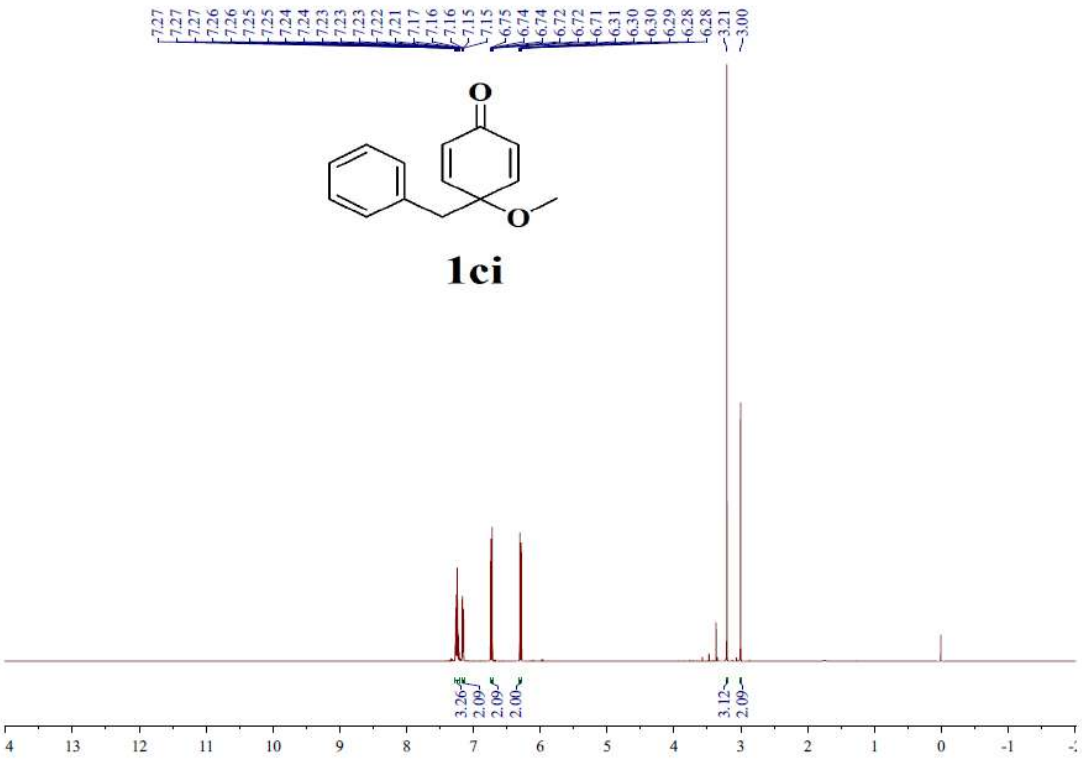
O
1ch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6.72
6.71
6.31
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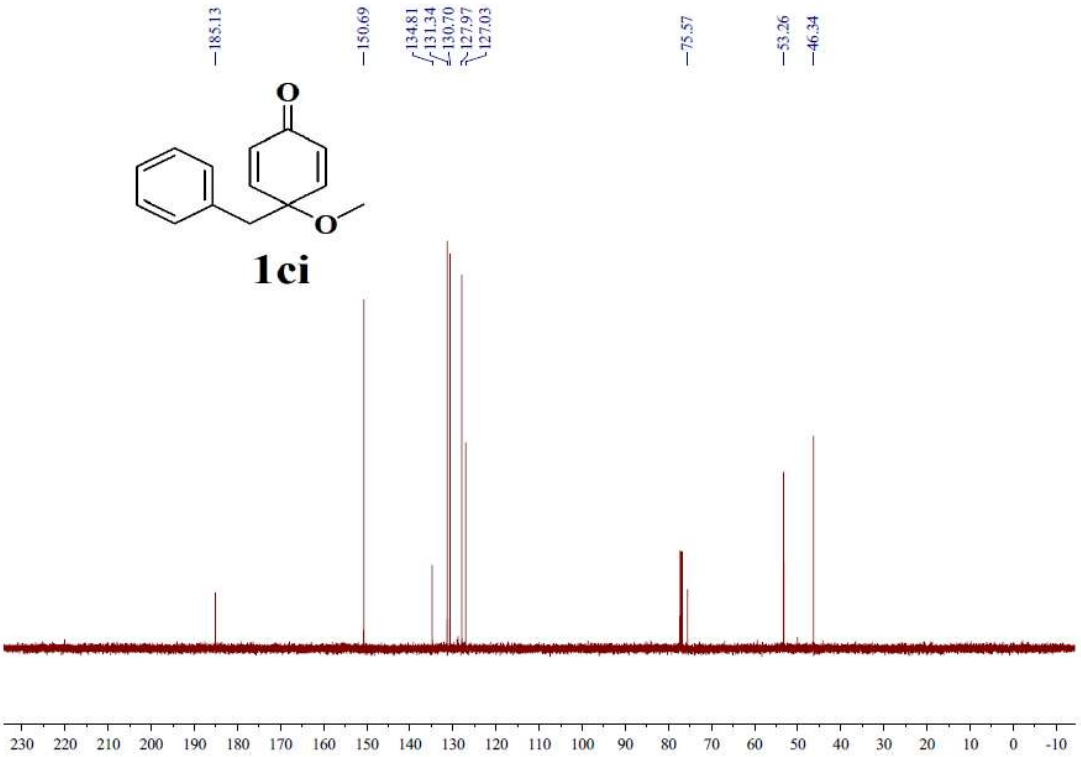
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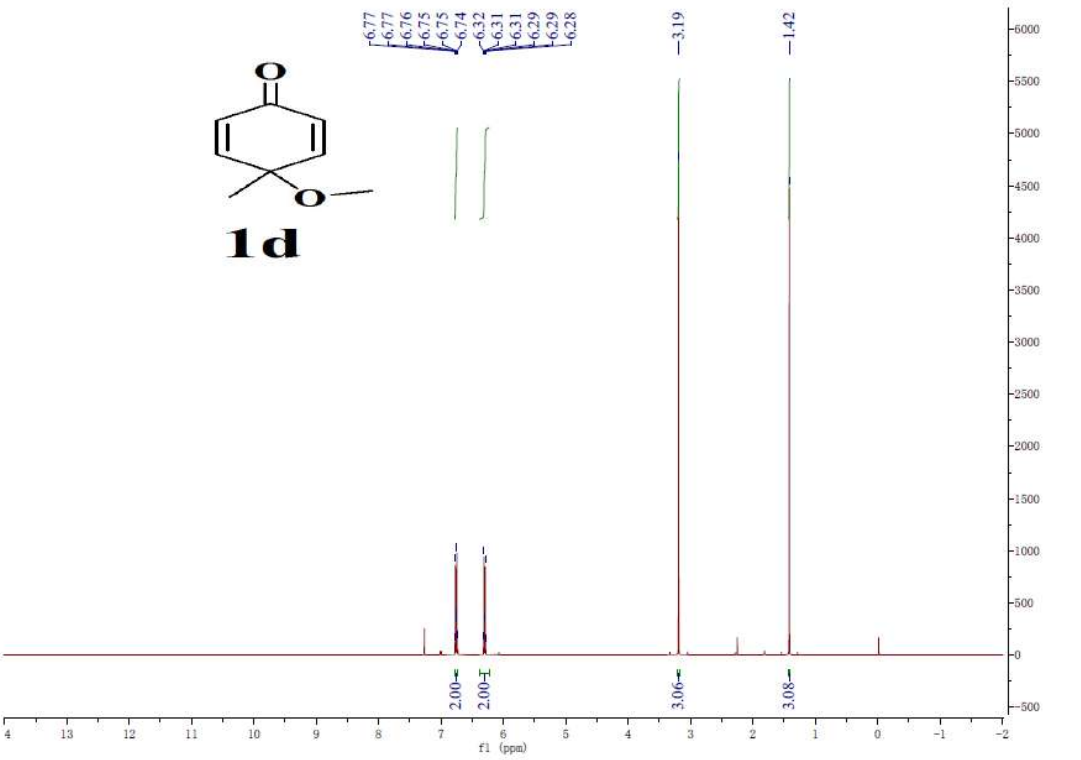
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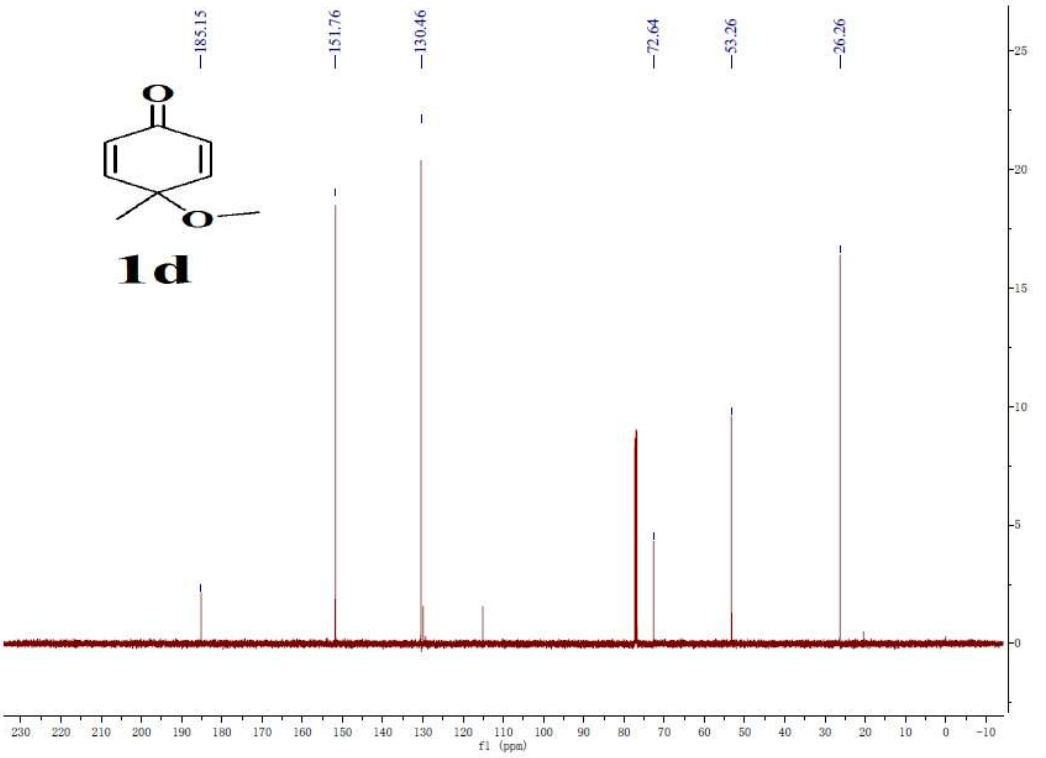
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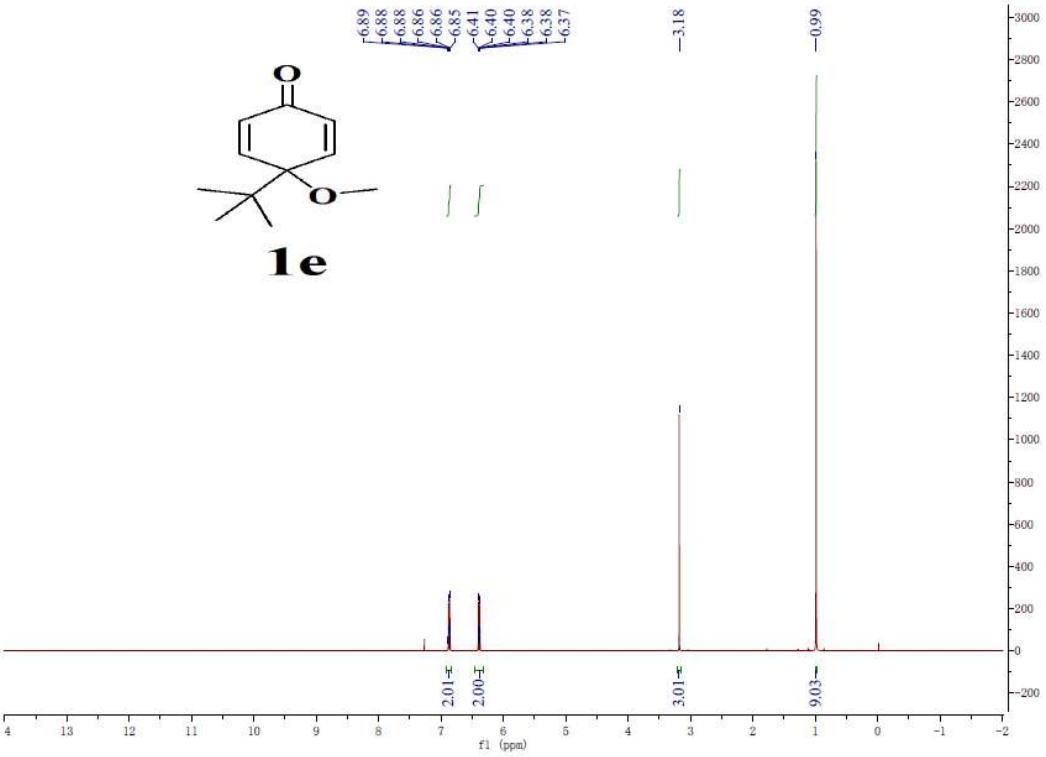
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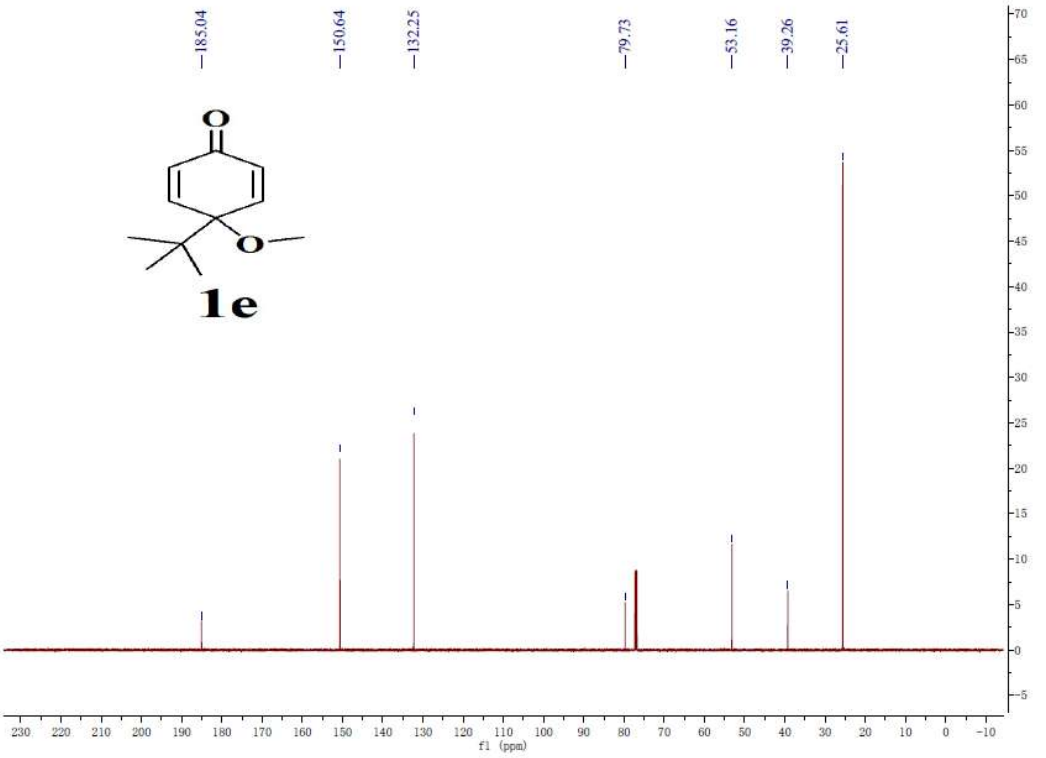
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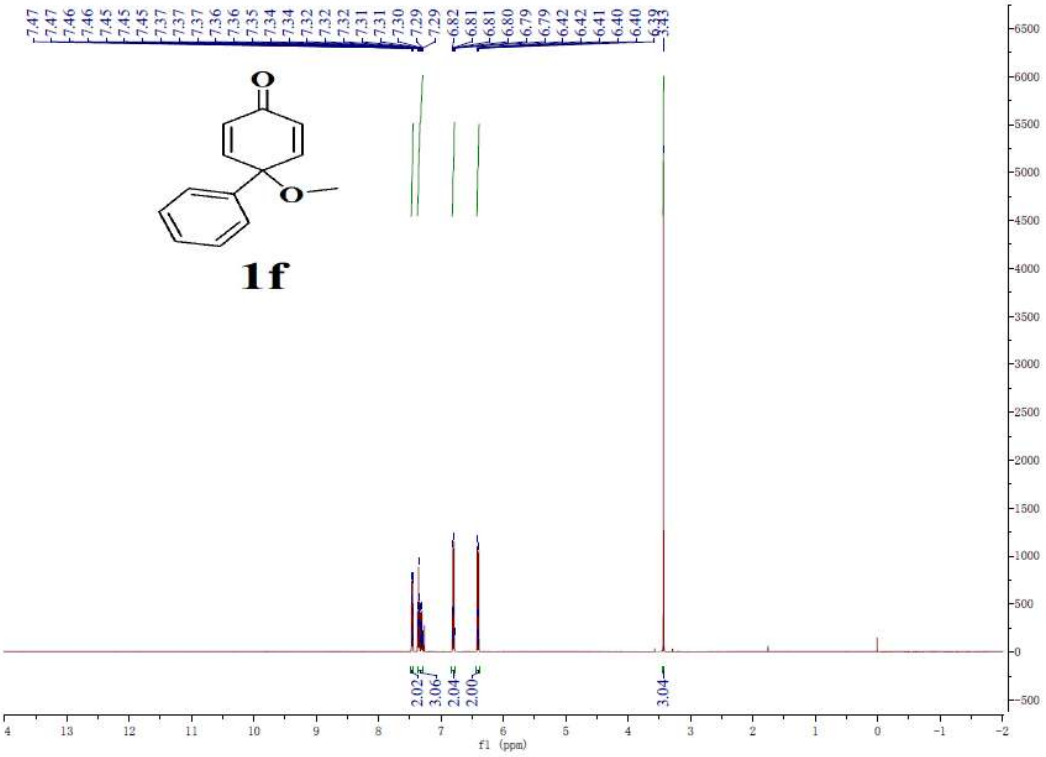
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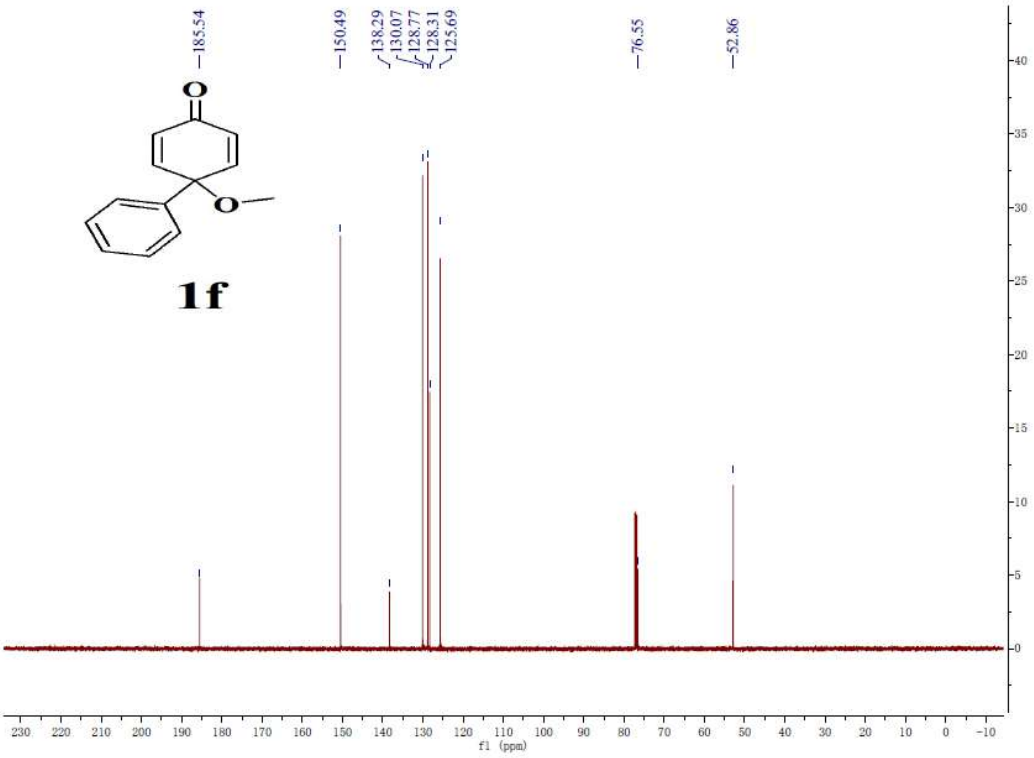
f1 (ppm)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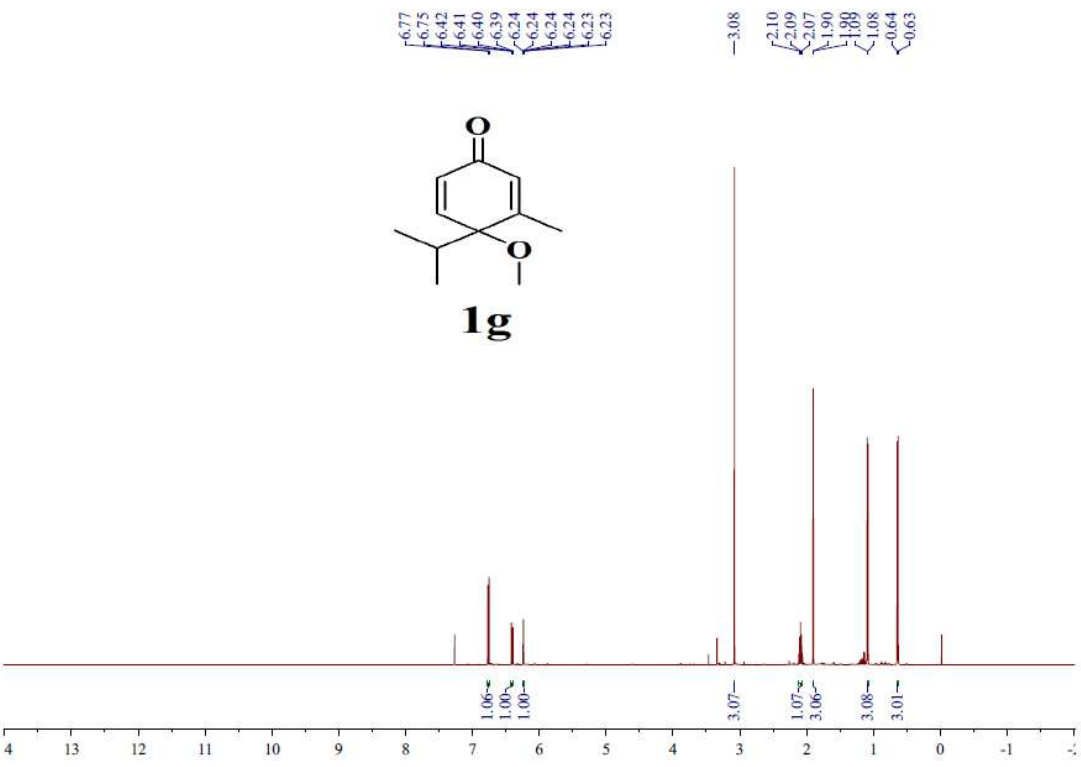
1f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2.09
2.07
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1.07
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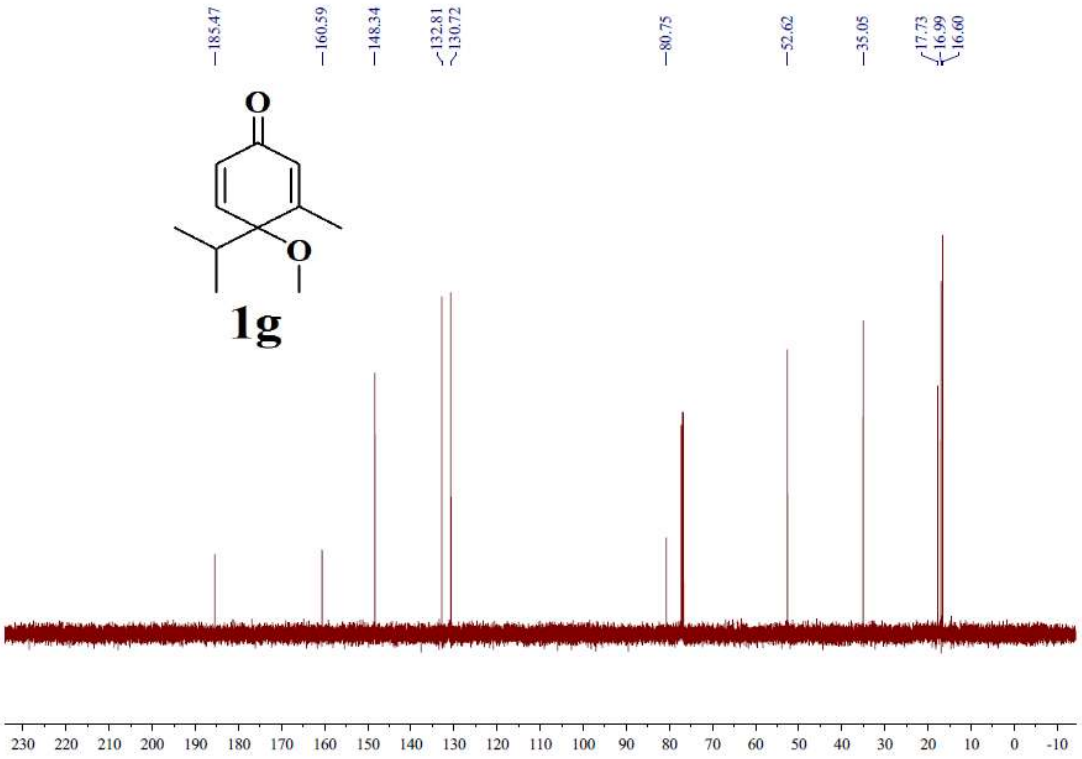
3.08
3.01

1g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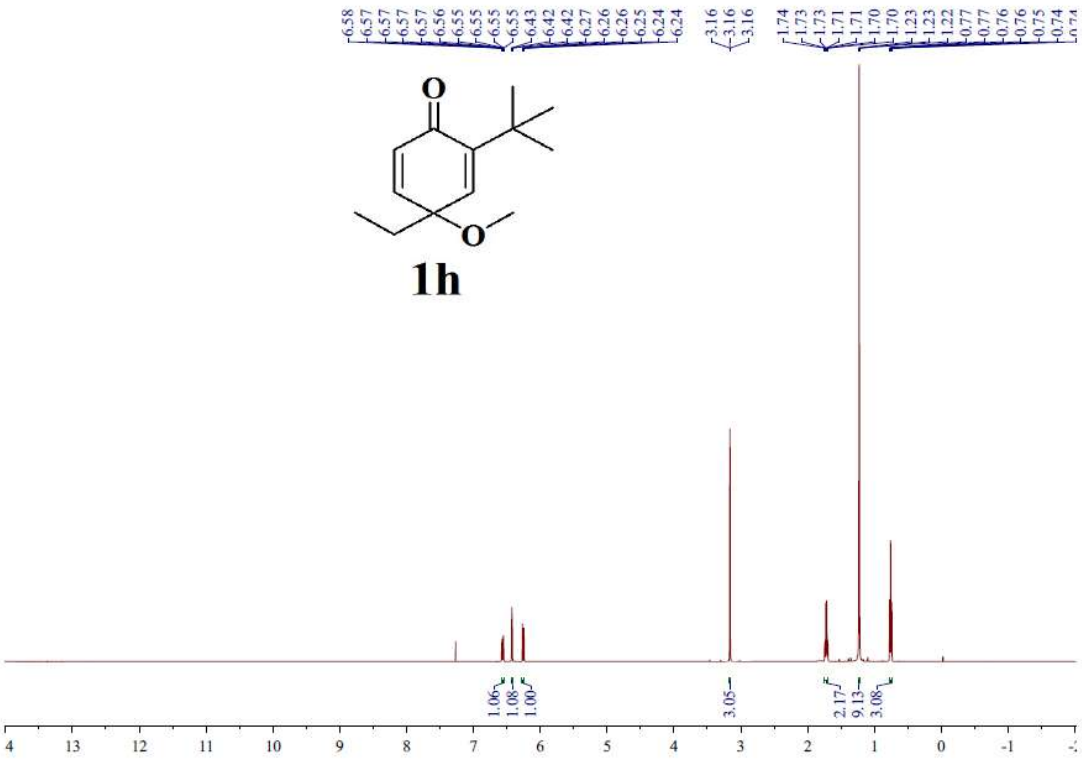
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3.16
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1.73
1.71
1.71
1.70
1.70
1.23
1.23
1.22
0.77
0.77
0.76
0.76
0.75
0.74
O
O
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1.00
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2.17
9.13
3.08
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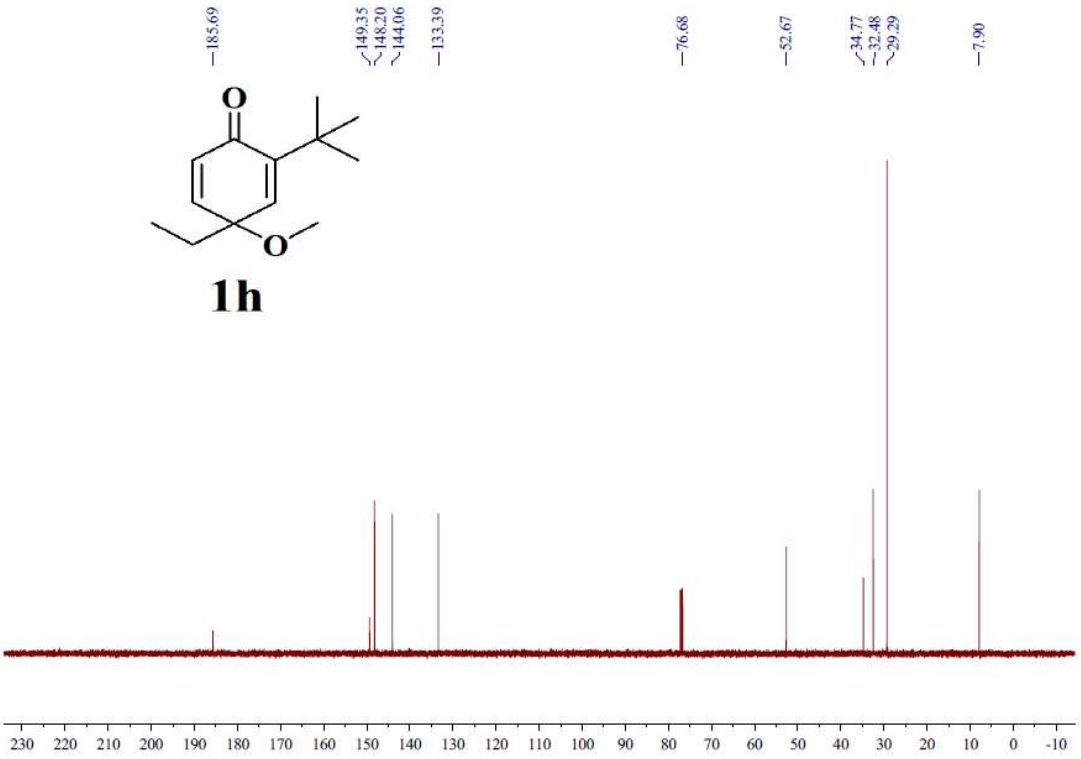
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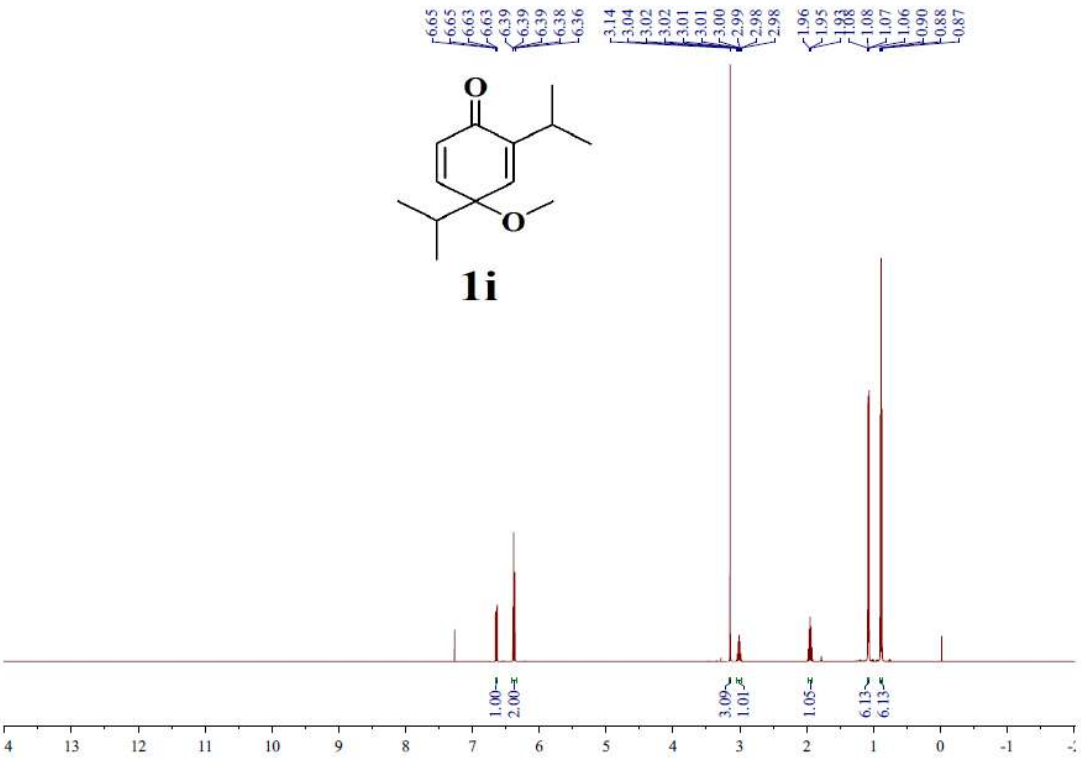
O
O
1h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3.01
3.00
2.99
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2.98
1.96
1.95
1.08
1.08
1.07
1.06
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O
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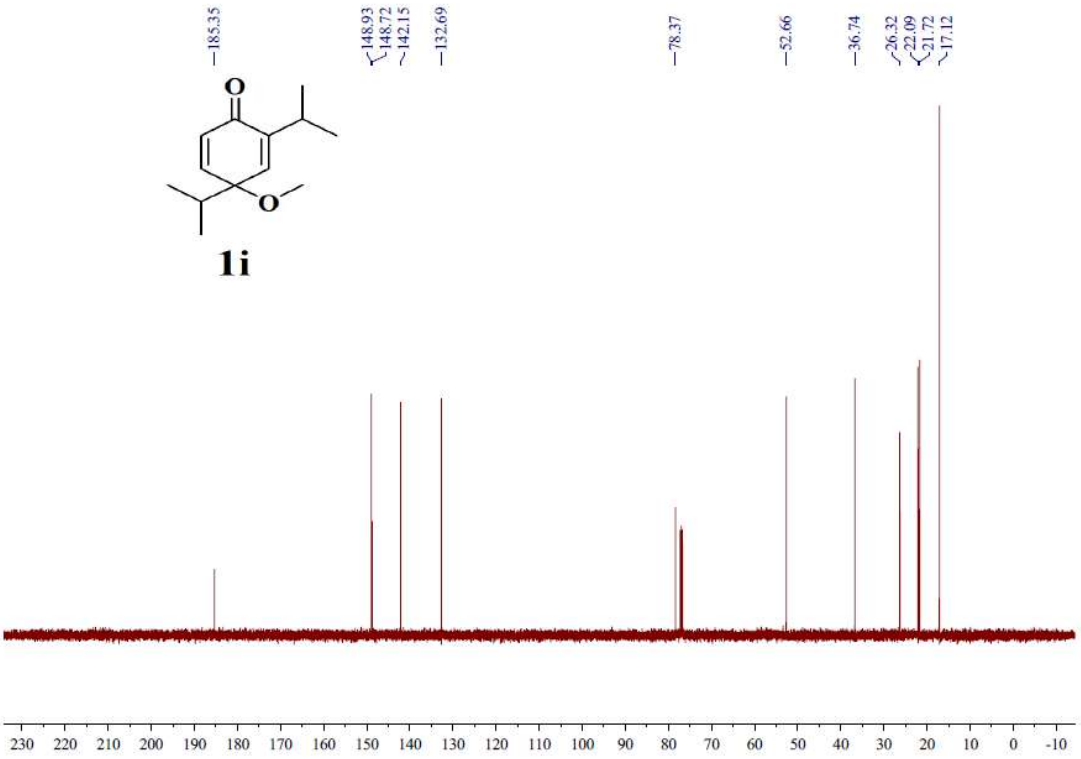
1.05
6.13
6.13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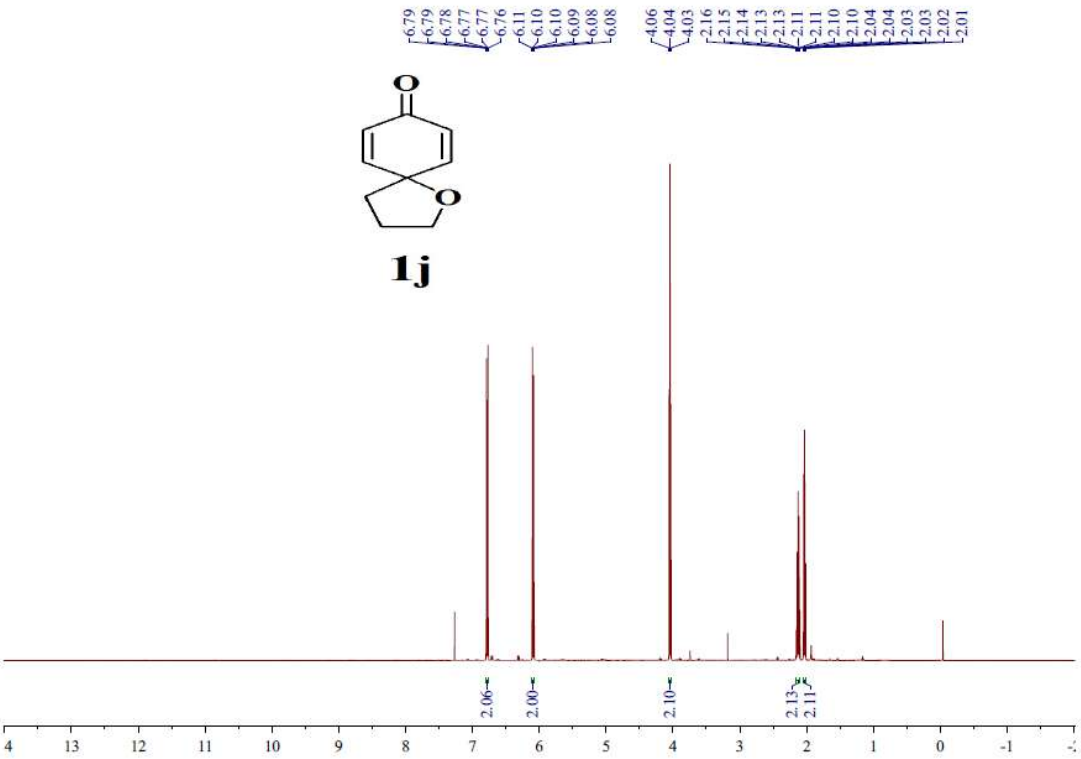
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4.04
4.03
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2.14
2.13
2.13
2.11
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2.10
2.10
2.04
2.04
2.03
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2.11
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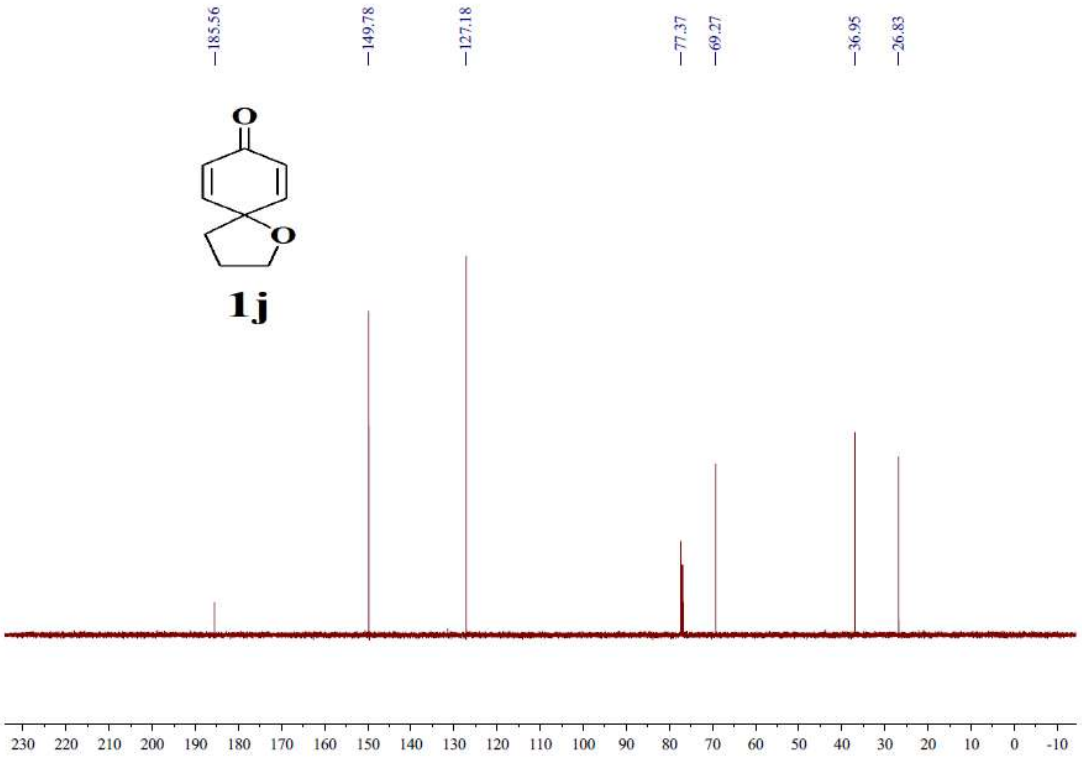
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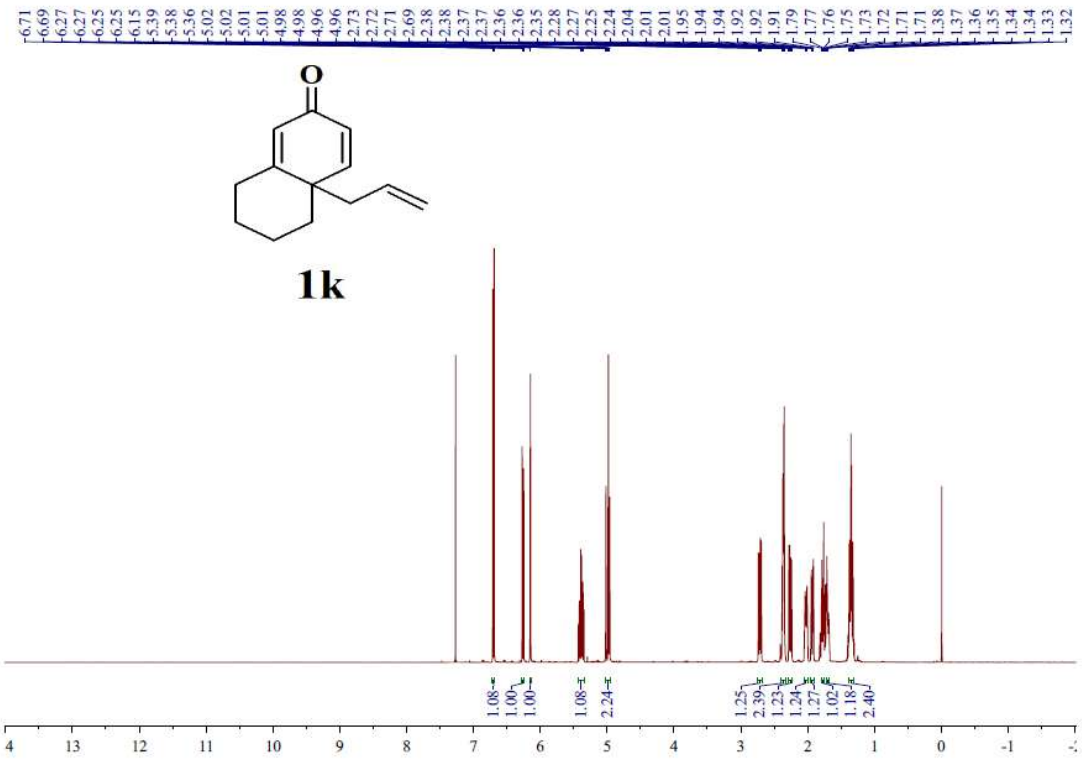
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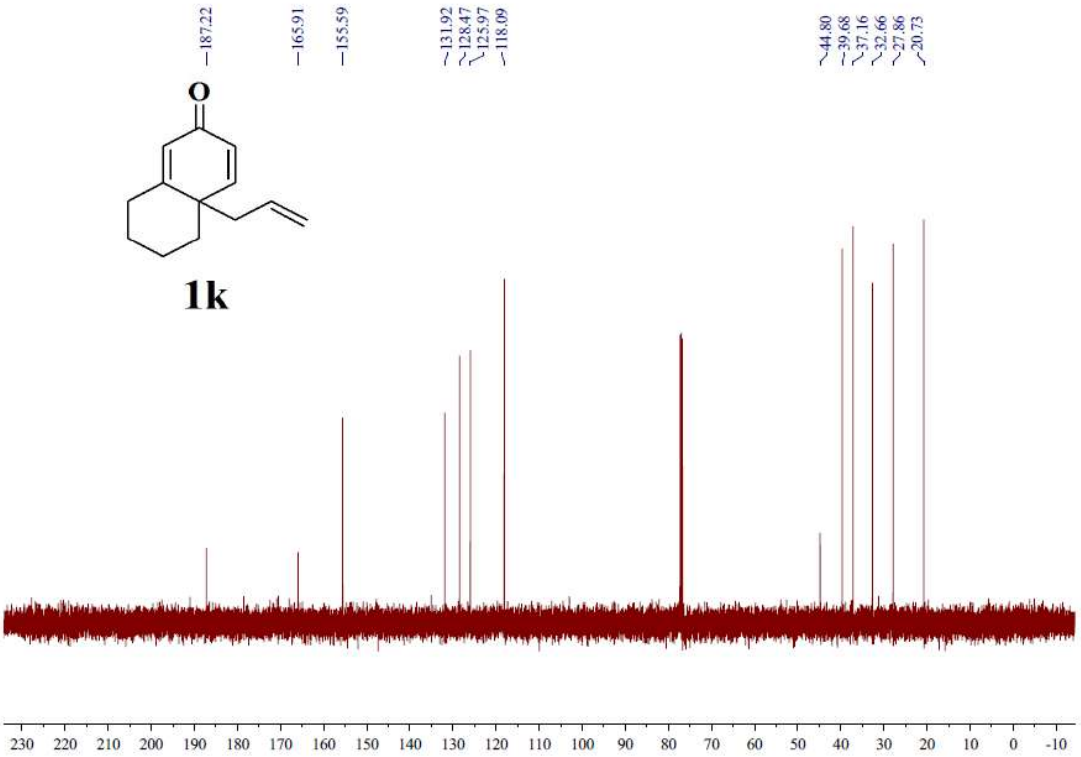
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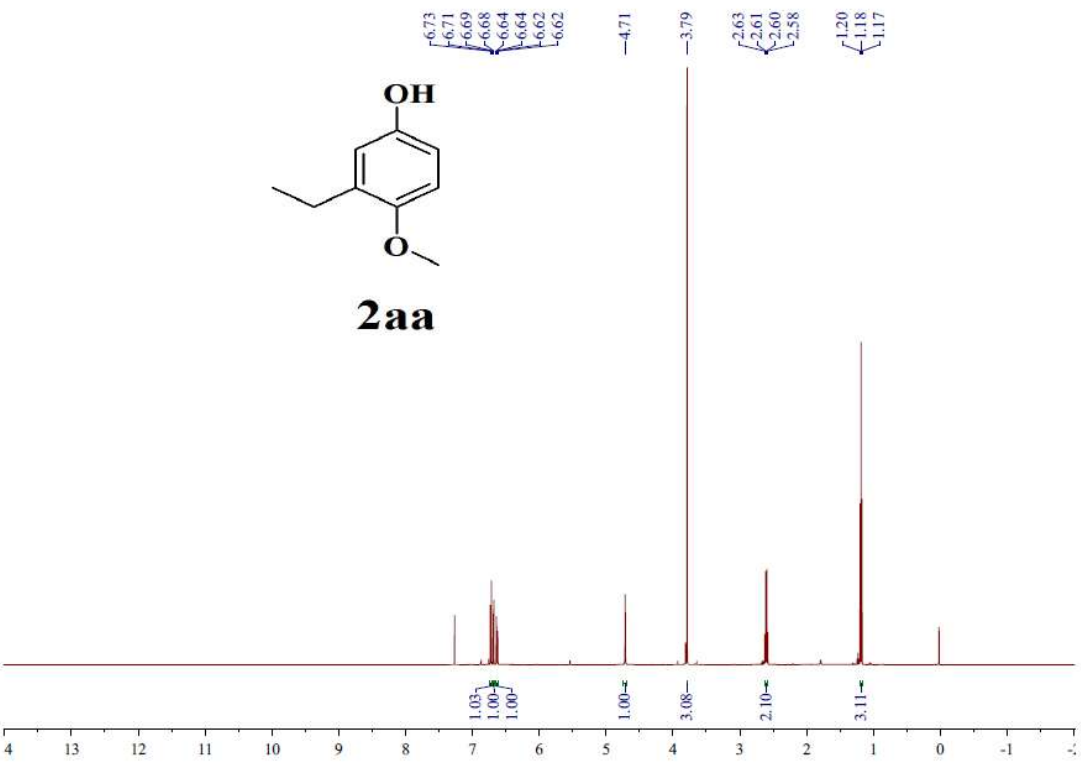
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1.17
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1.00
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3.11
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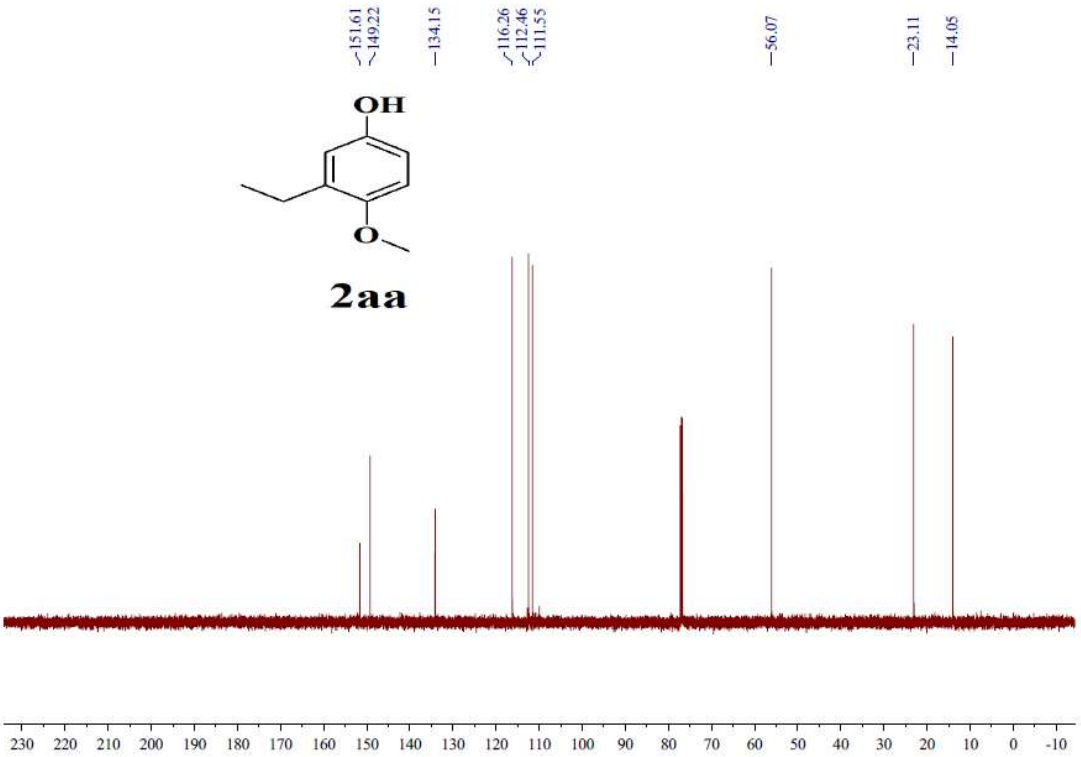
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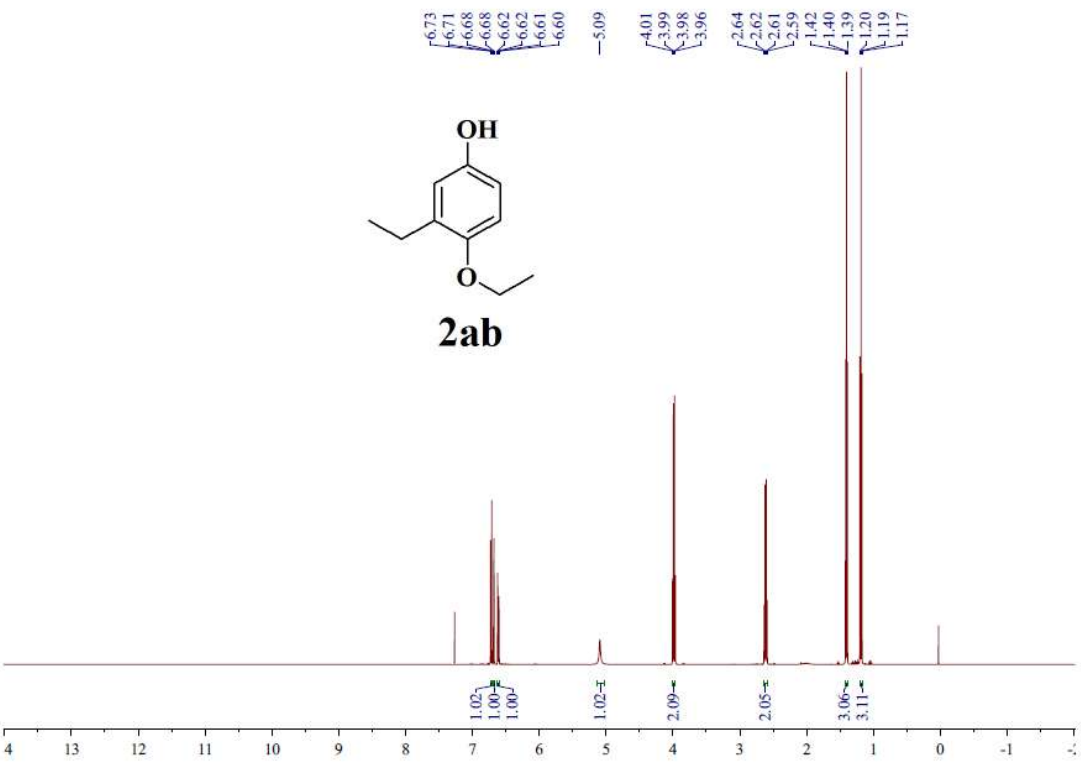
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1.17
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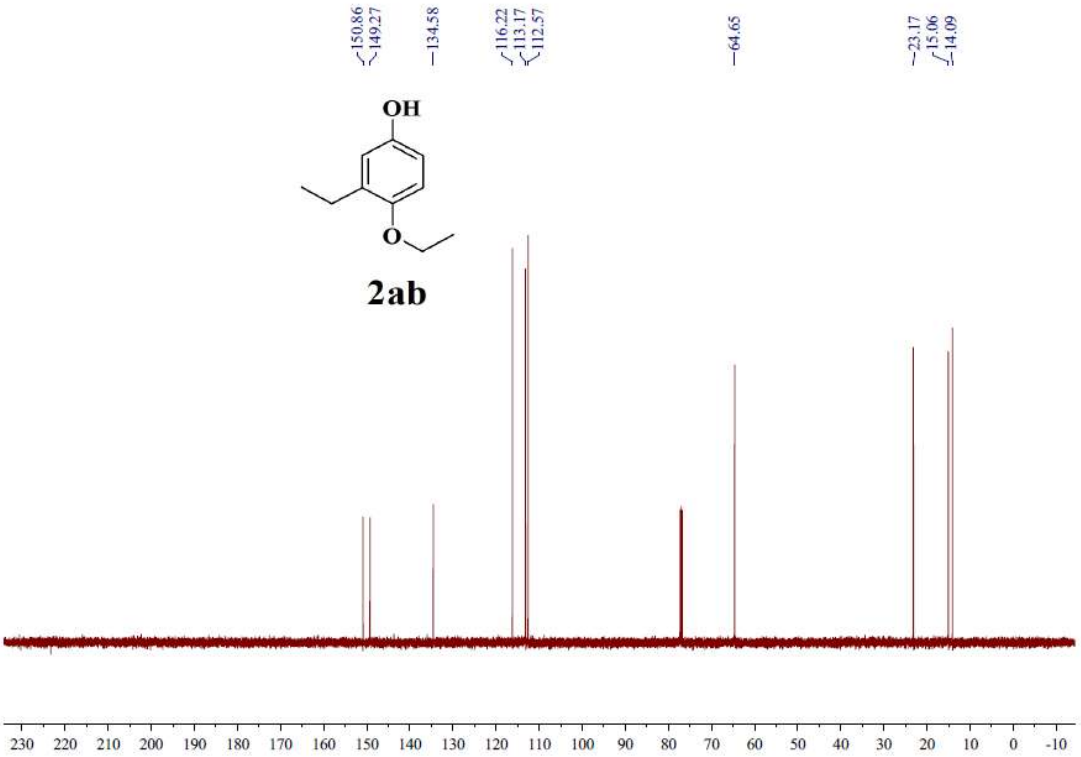
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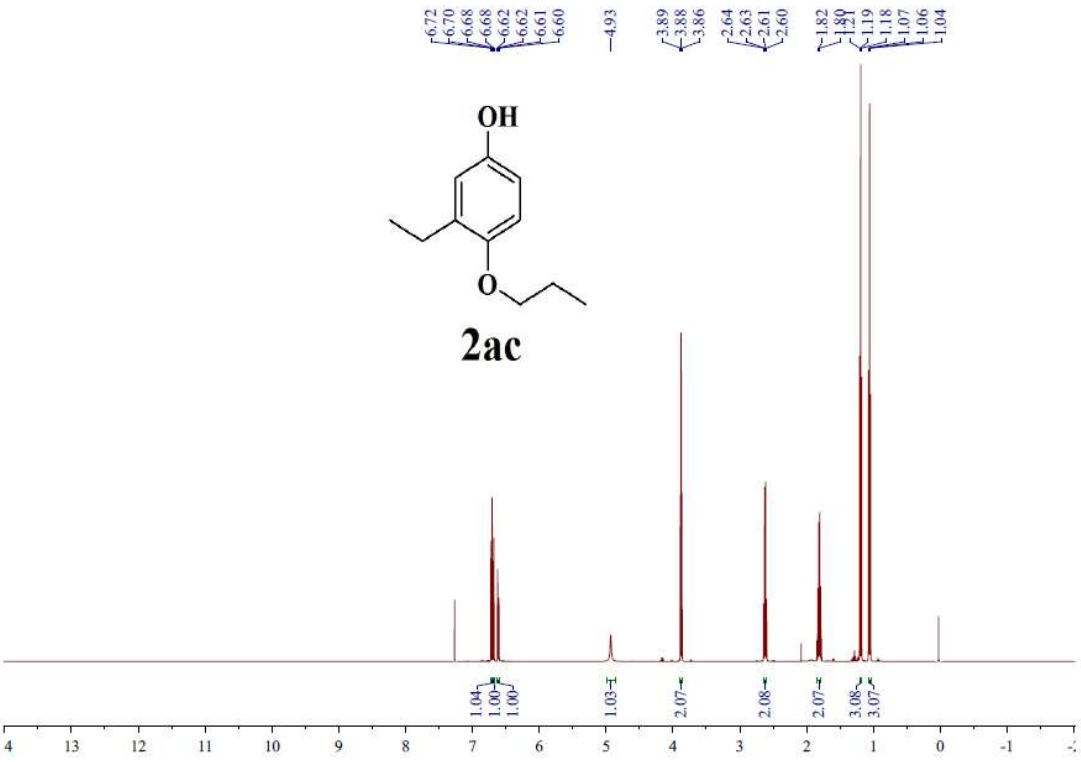
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1.18
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1.06
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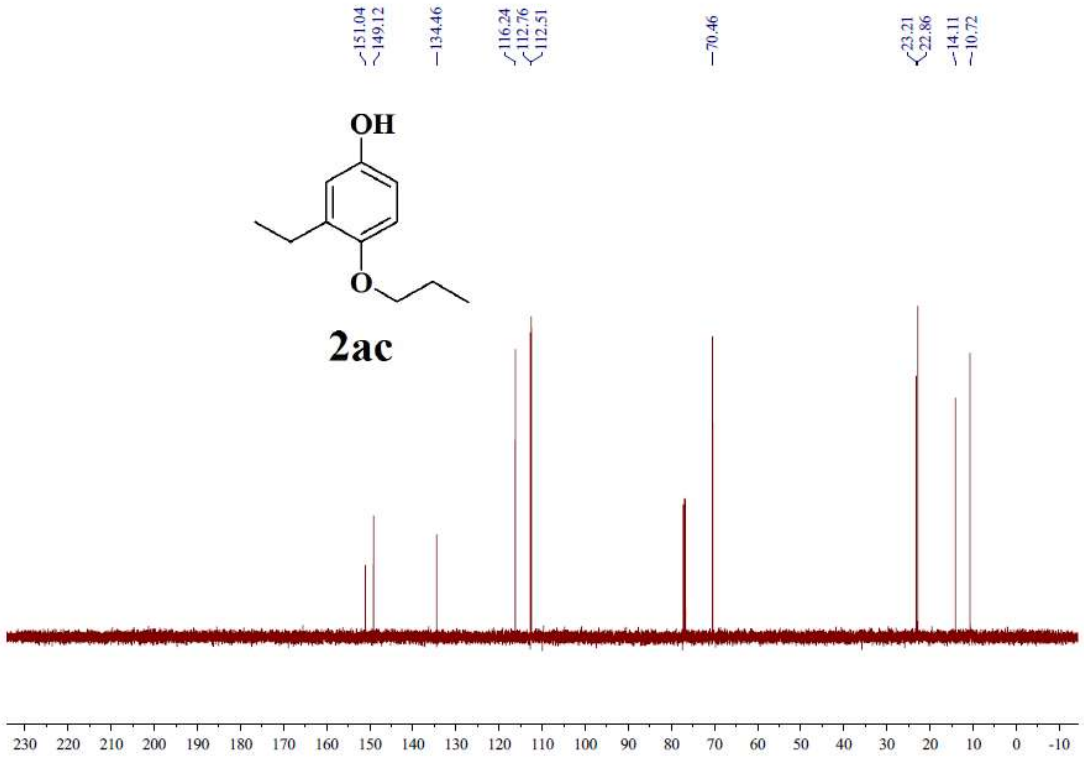
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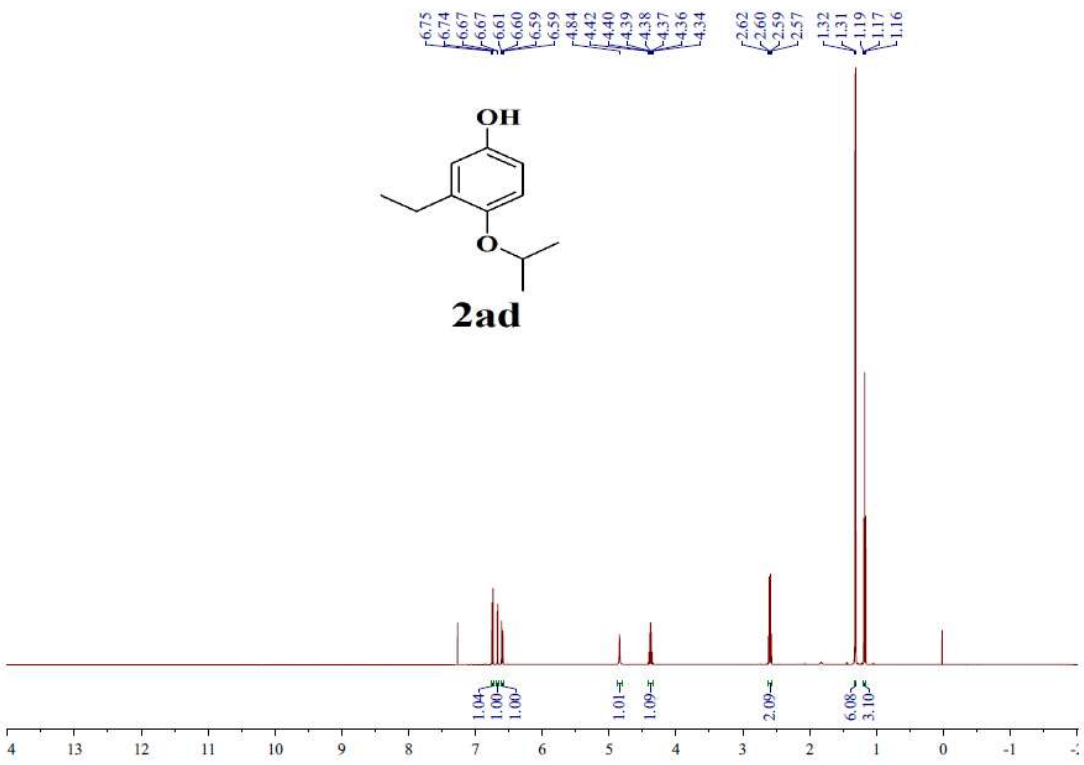
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2.57
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1.31
1.19
1.17
1.16
OH
O
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1.01
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2.09
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3.10
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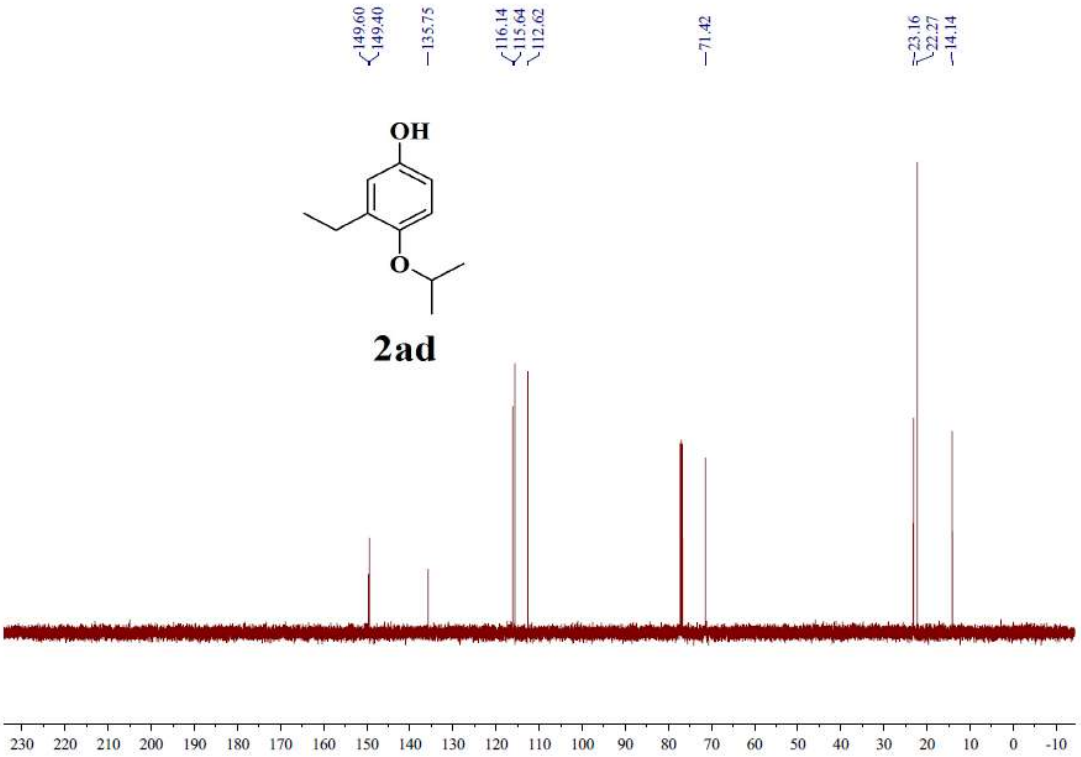
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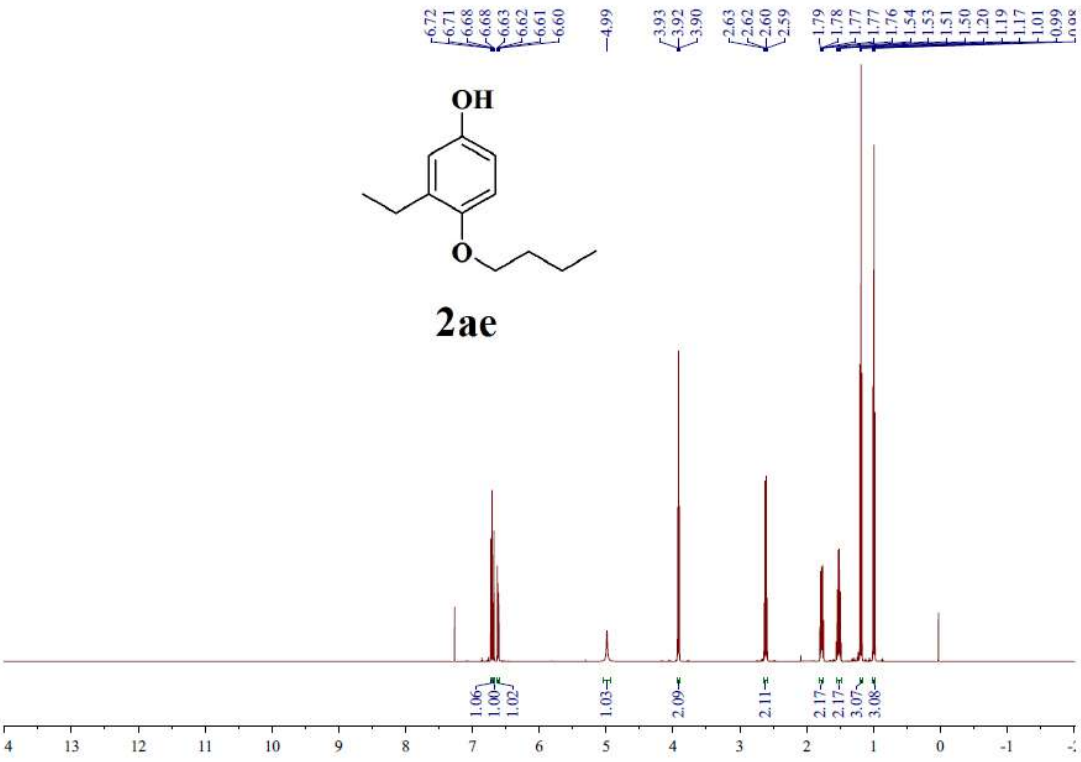
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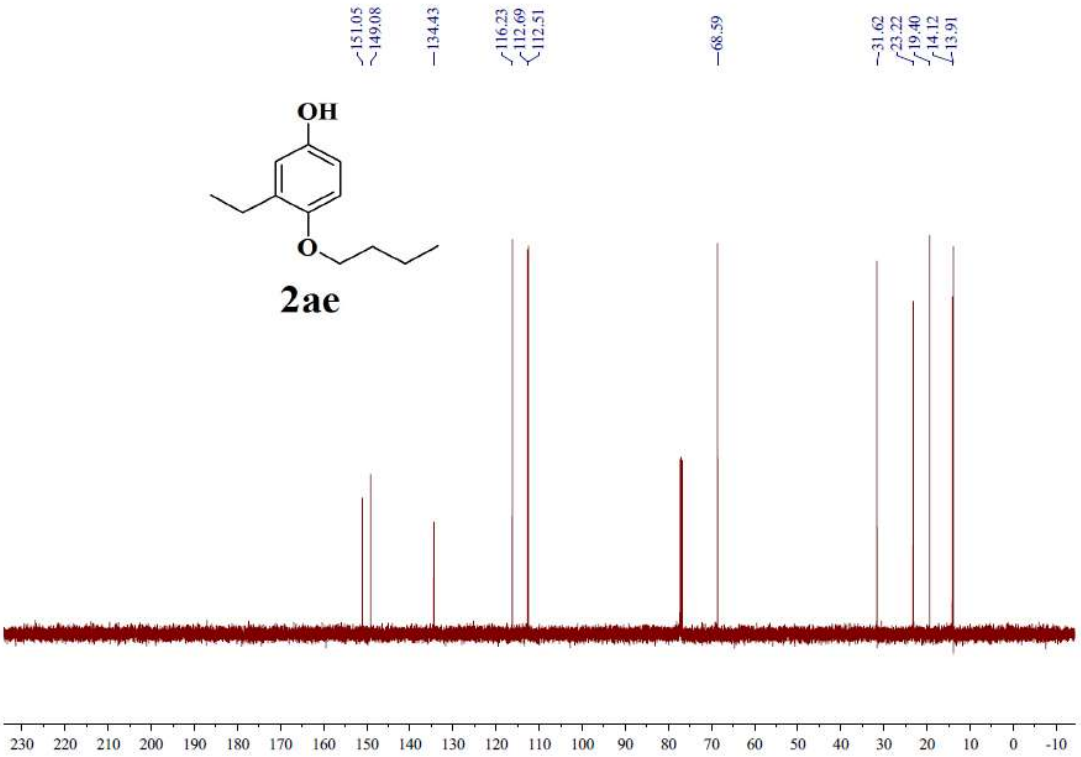
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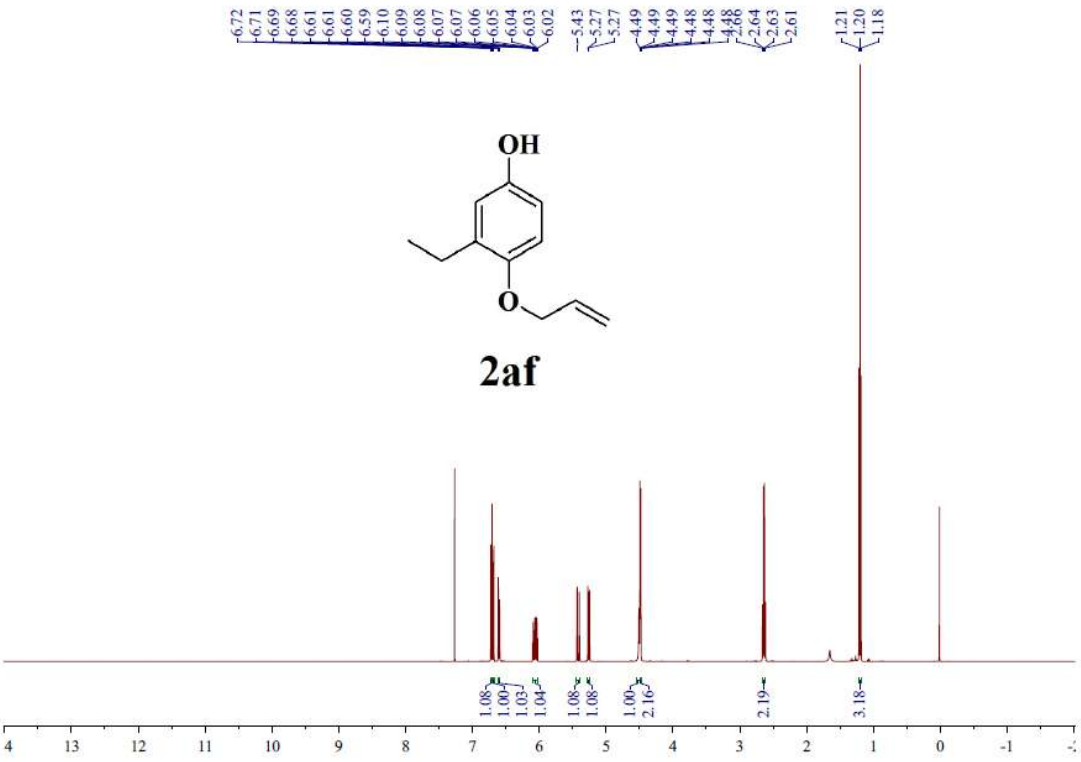
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2af
1.08
1.00
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1.08
1.08
1.00
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2.19
3.18
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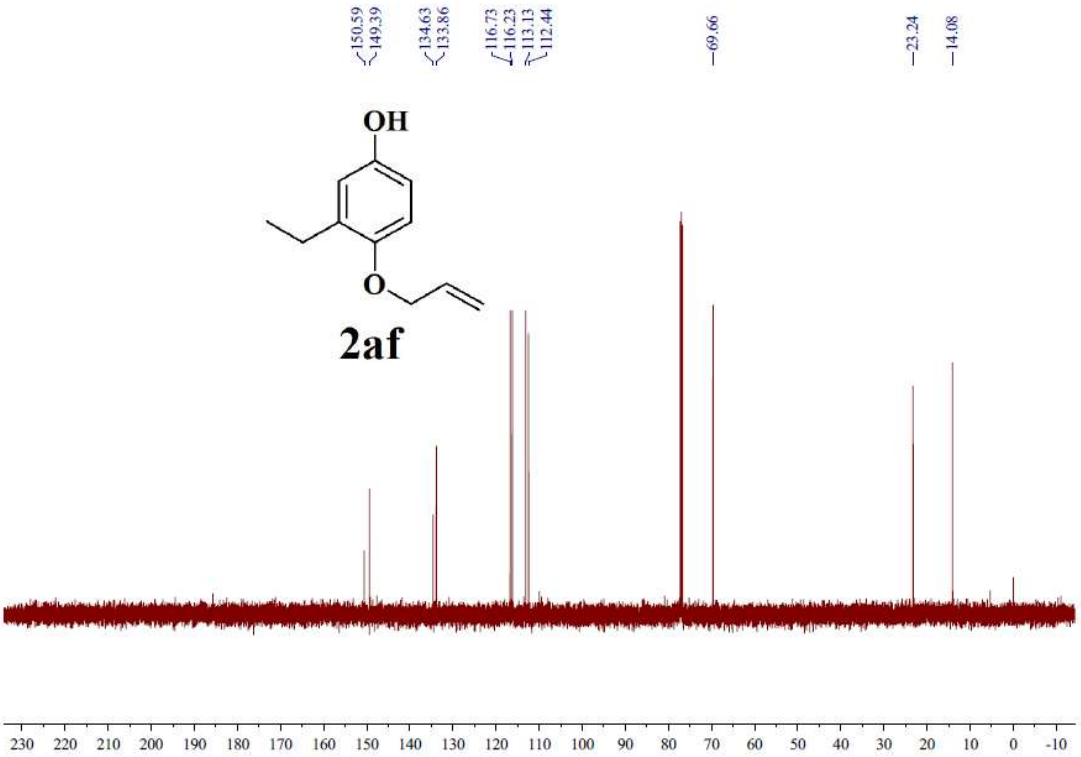
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2af
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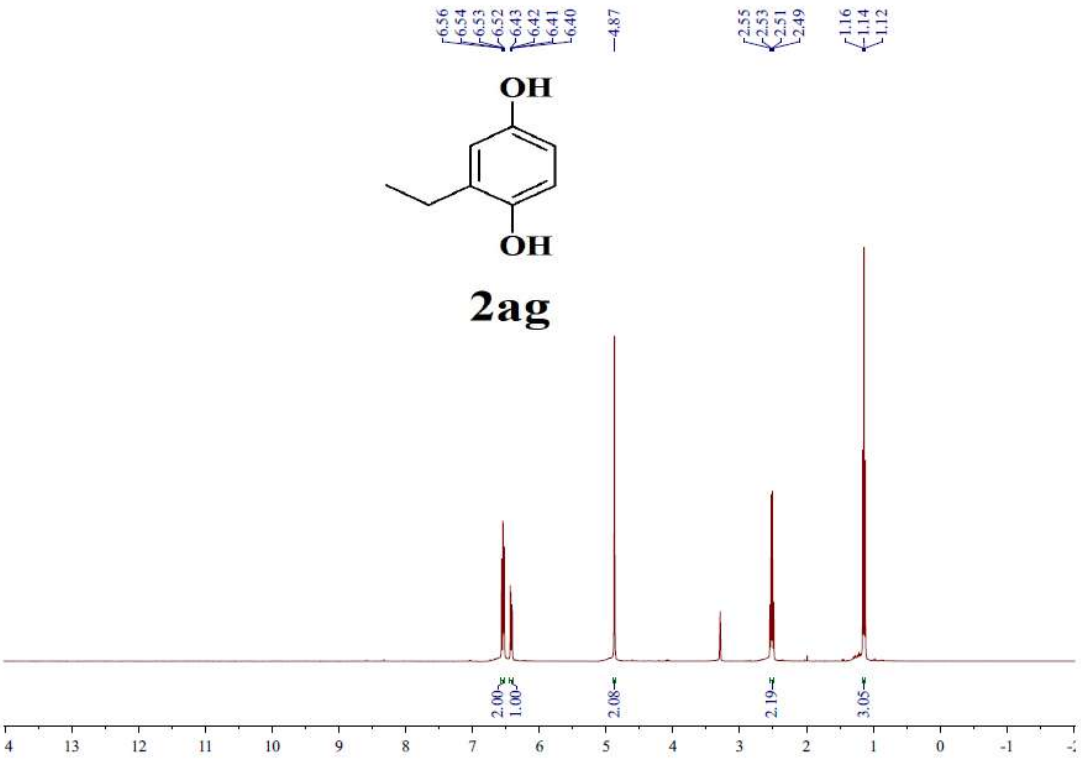
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3.05
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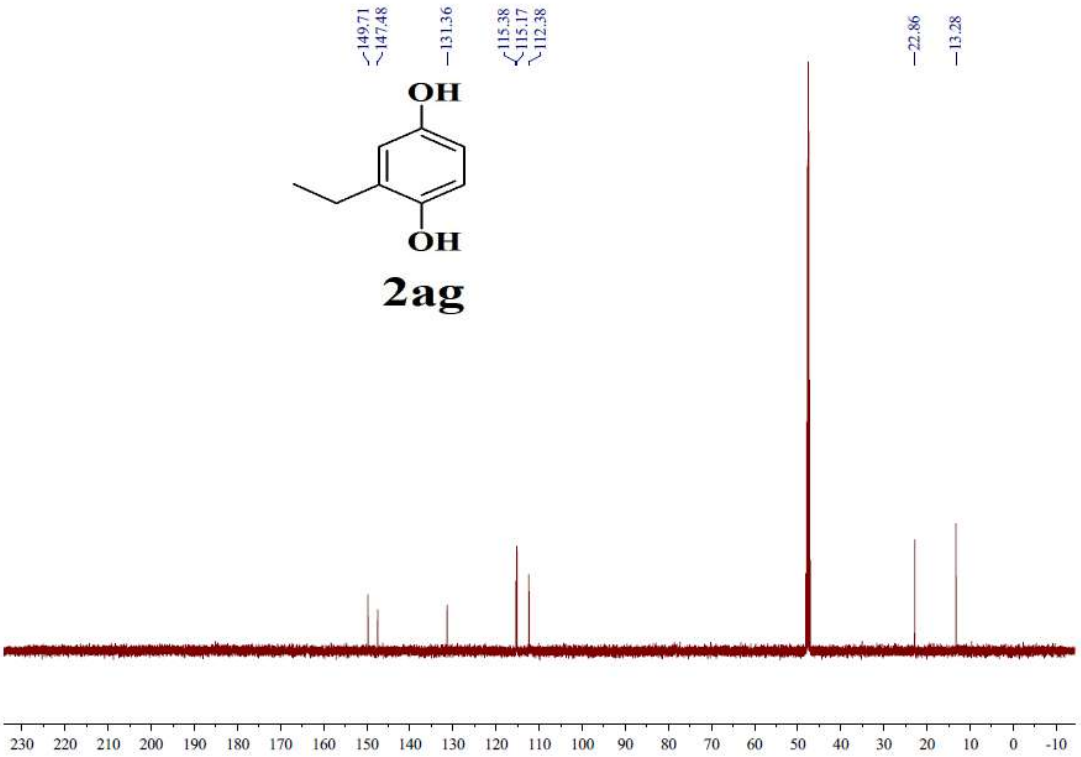
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OH
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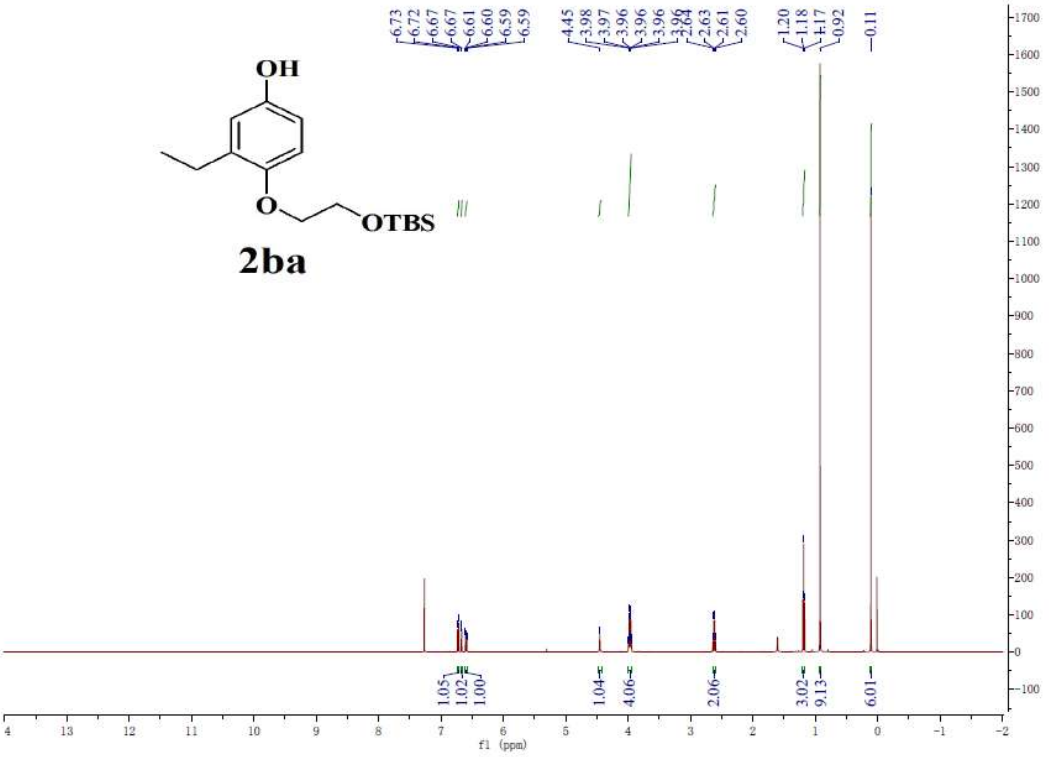
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3.96
3.96
3.96
2.64
2.63
2.61
2.60
1.20
1.18
1.17
0.92
0.11
OH
O
OTBS
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1.02
1.00
1.04
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3.02
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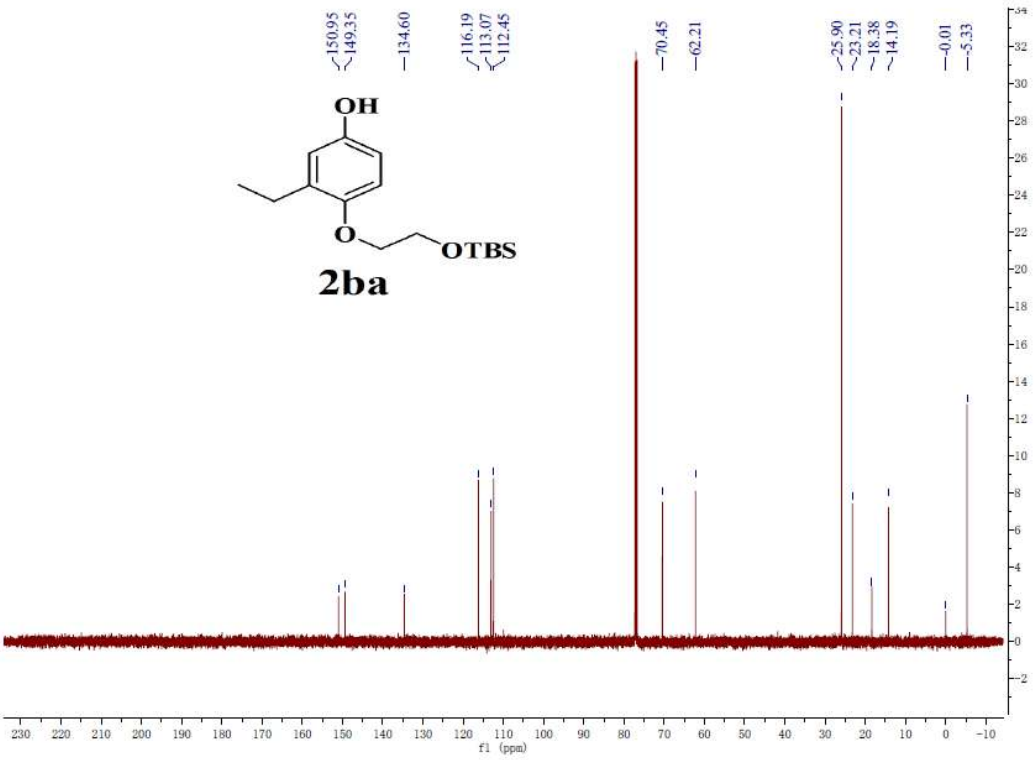
6.01
f1 (ppm)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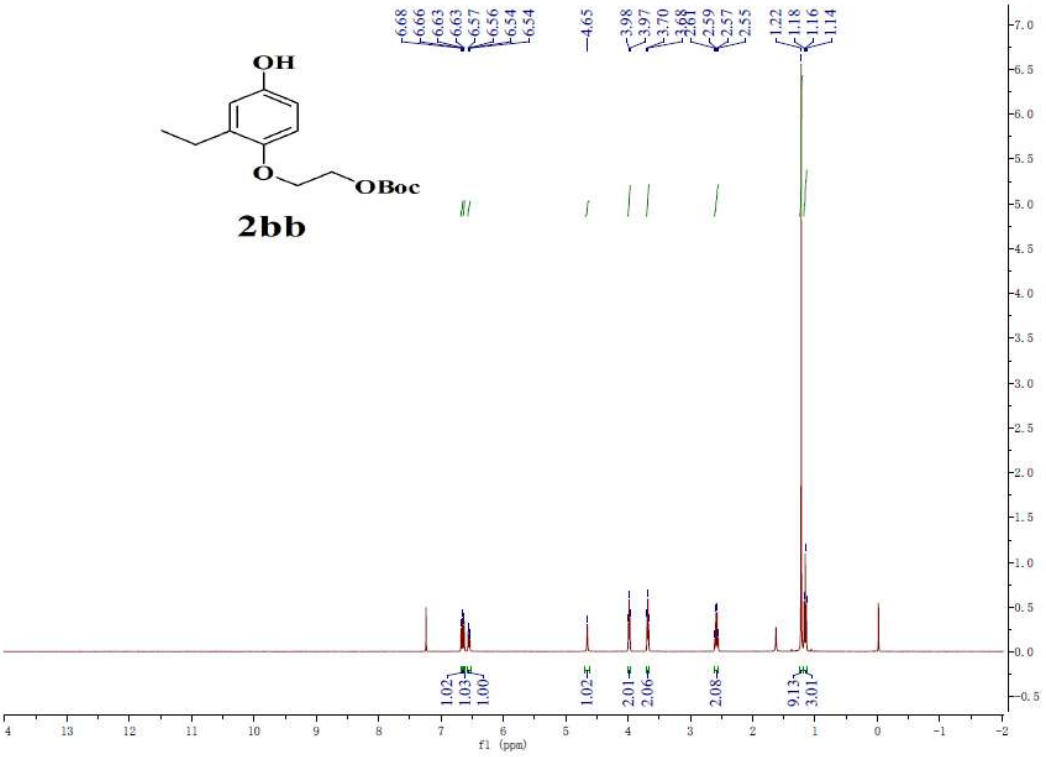
23.21
18.38
14.19
-0.01
-5.33
OH
O
OTBS
2ba
f1 (ppm)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6.63
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6.54
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3.98
3.97
3.70
3.68
2.61
2.59
2.57
2.55
1.22
1.18
1.16
1.14
OH
O
OBoc
2bb
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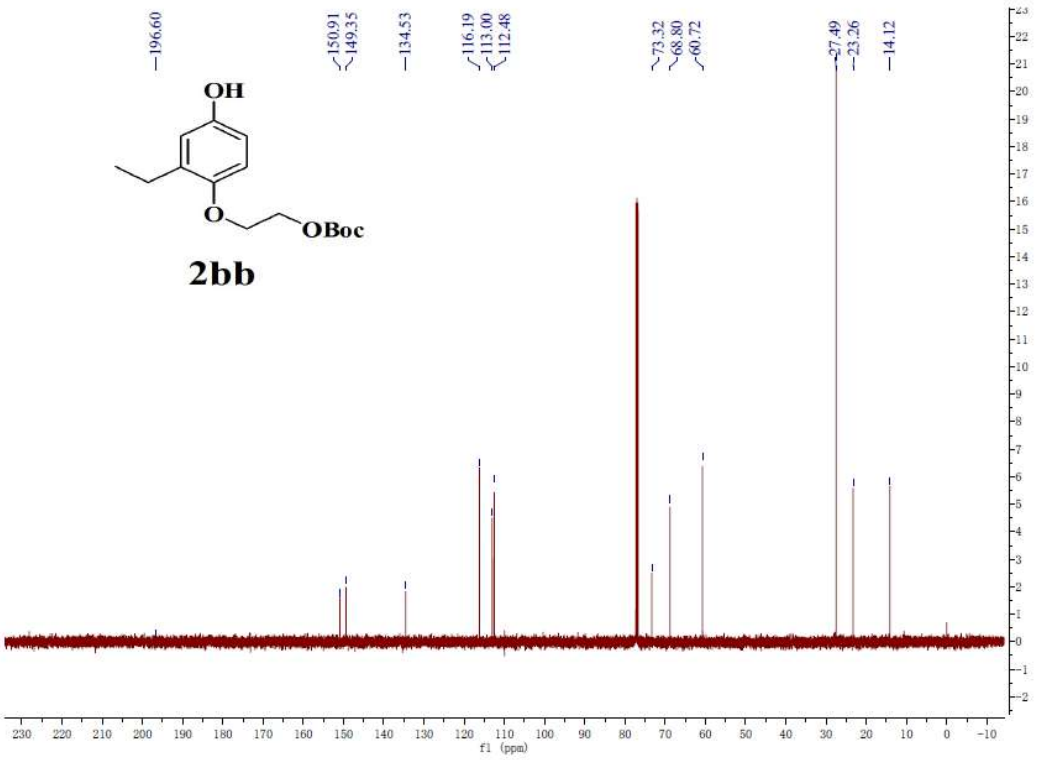
1.03
1.00
1.02
2.01
2.06
2.08
9.13
3.01
f1 (ppm)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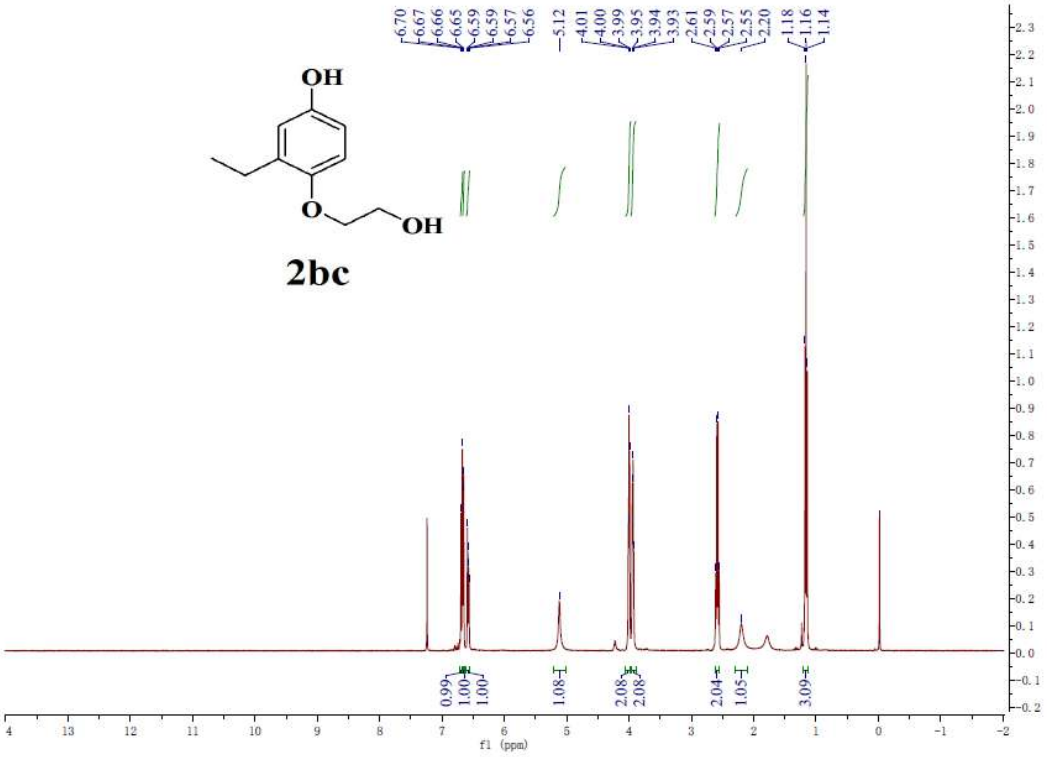
27.49
23.26
14.12
OH
O
OBoc
2bb
f1 (ppm)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6.57
6.56
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4.01
4.00
3.99
3.95
3.94
3.93
2.61
2.59
2.57
2.55
2.20
1.18
1.16
1.14
OH
O
OH
2bc
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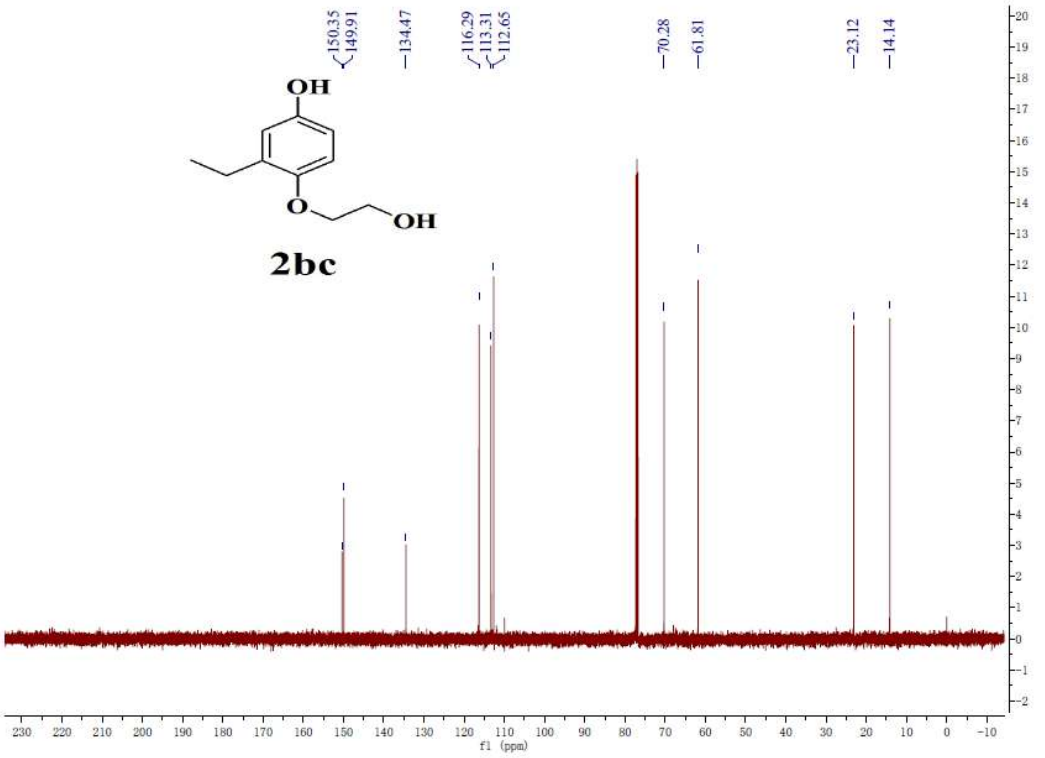
1.00
1.00
1.08
2.08
2.08
2.04
1.05
3.09
f1 (ppm)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112.65
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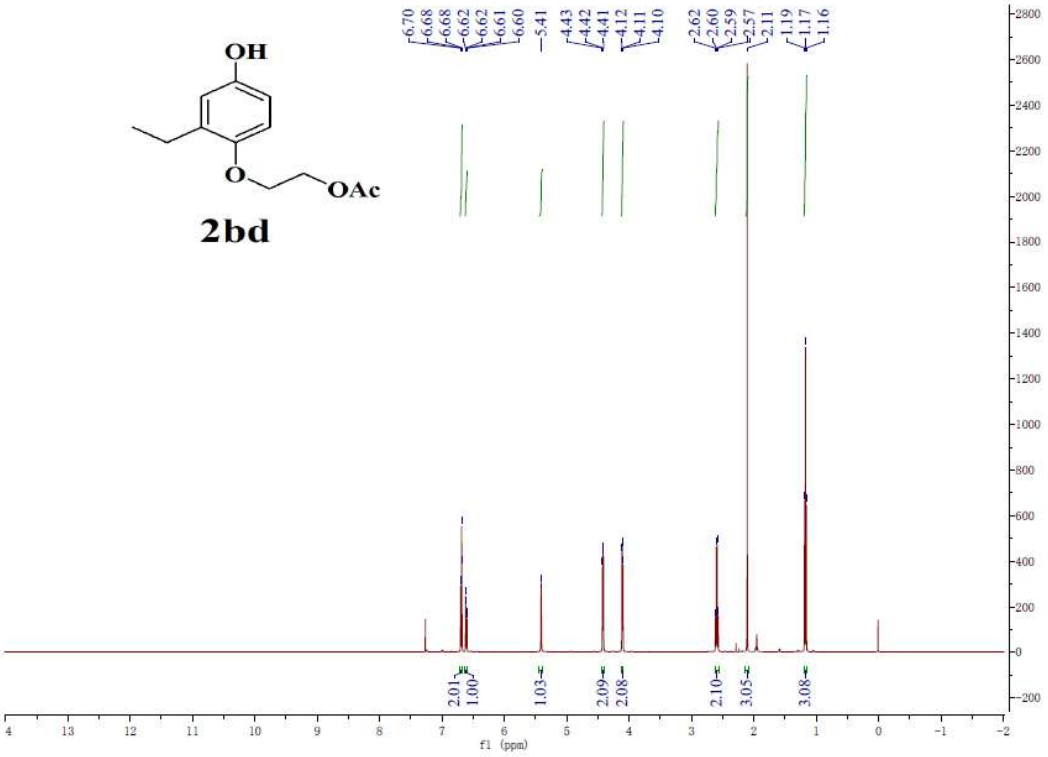
61.81
23.12
14.14
OH
O
OH
2bc
f1 (ppm)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6.61
6.60
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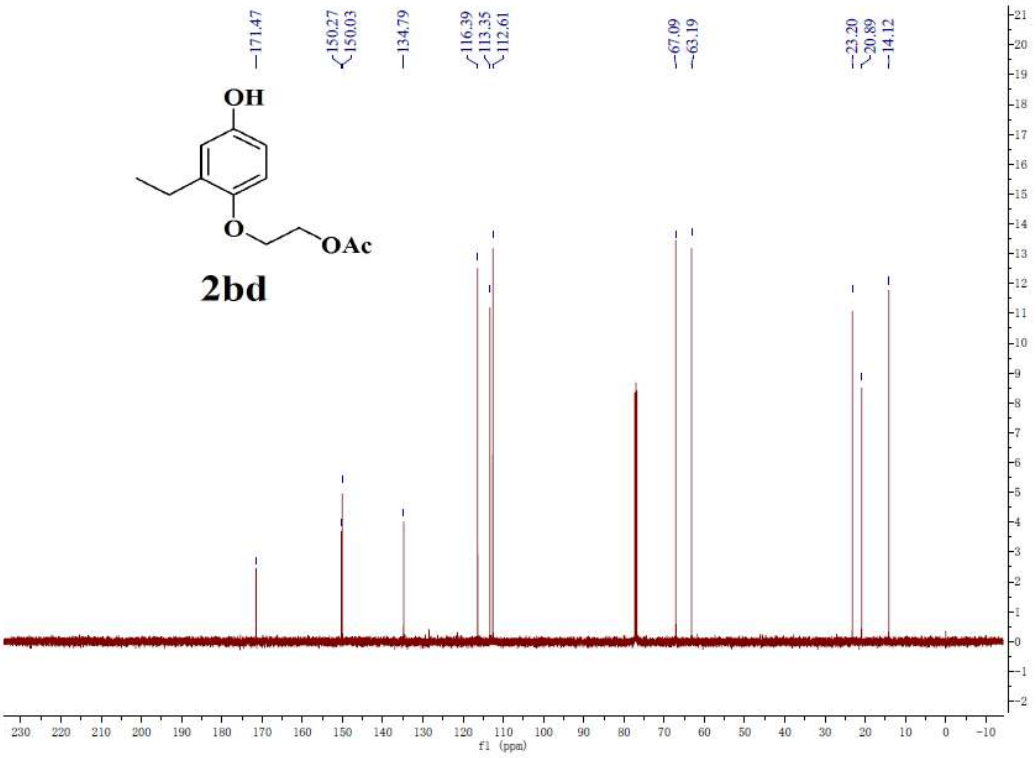
4.42
4.41
4.12
4.11
4.10
2.62
2.60
2.59
2.57
2.11
1.19
1.17
1.16
OH
O
OAc
2bd
2.01
1.00
1.03
2.09
2.08
2.10
3.05
3.08
f1 (ppm)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67.09
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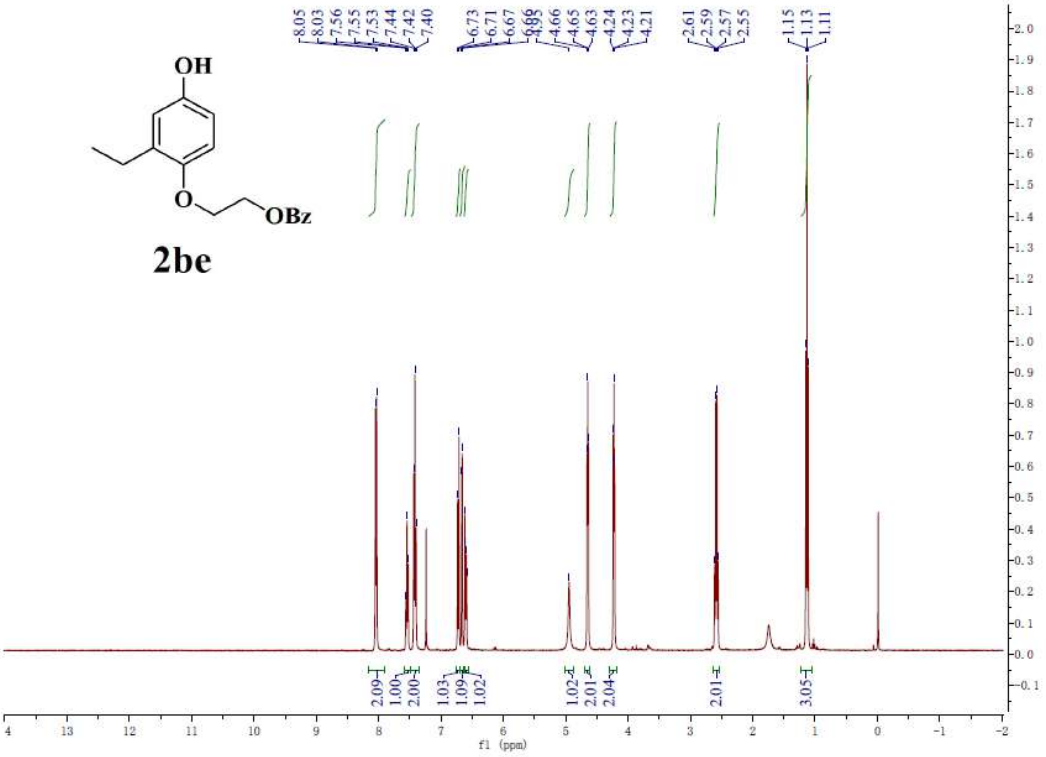
23.20
20.89
14.12
OH
O
OAc
2bd
f1 (ppm)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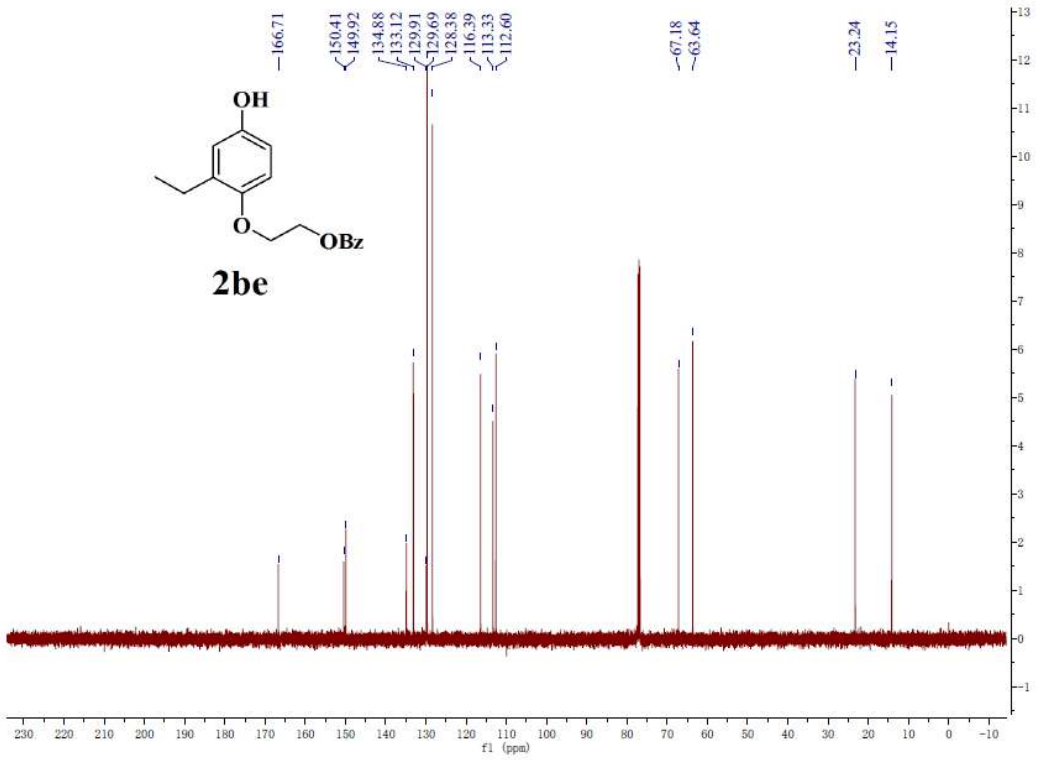
6.71
6.67
4.66
4.65
4.63
4.24
4.23
4.21
2.61
2.59
2.57
2.55
1.15
1.13
1.11
OH
O
OBz
2be
2.09
1.00
2.00
1.03
1.09
1.02
1.02
2.01
2.04
2.01
3.05
f1 (ppm)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149.92
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129.91
129.69
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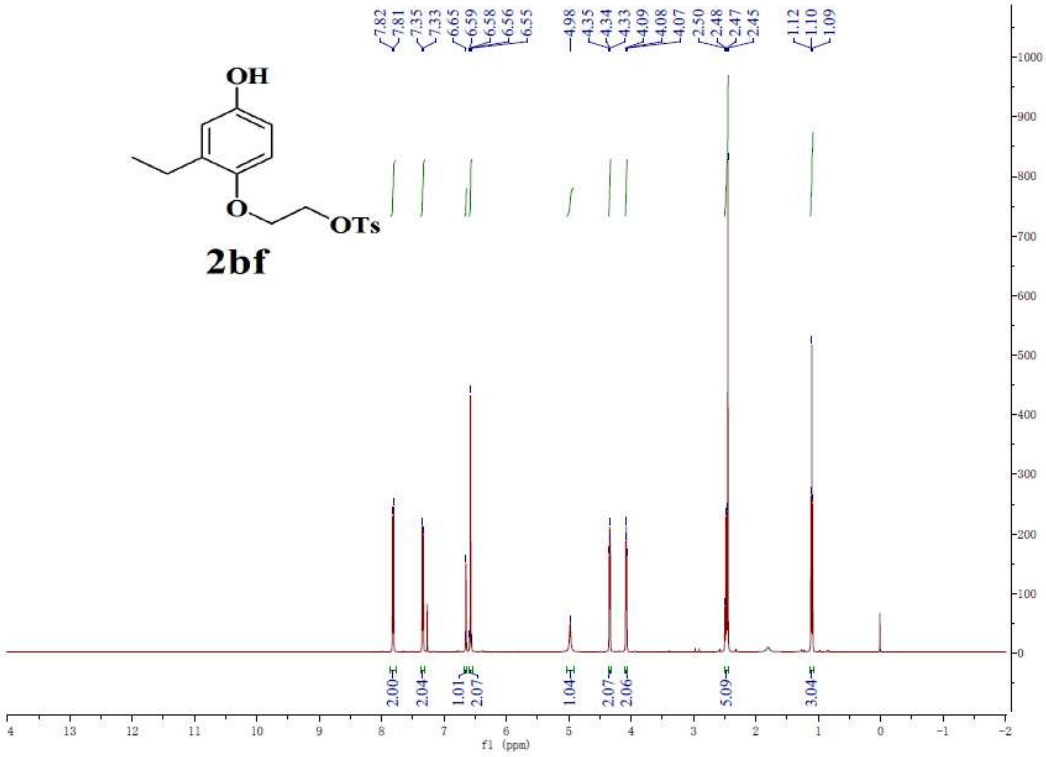
113.33
112.60
67.18
63.64
23.24
14.15
OH
O
OBz
2be
f1 (ppm)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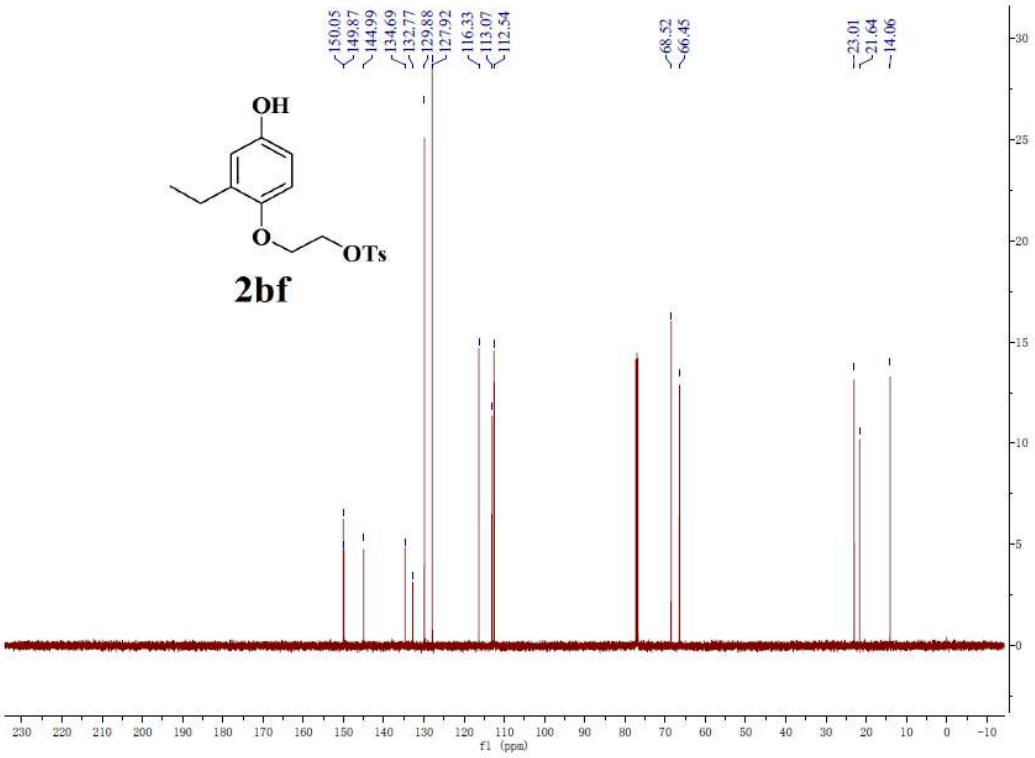
6.56
6.55
4.98
4.35
4.34
4.33
4.09
4.08
4.07
2.50
2.48
2.47
2.45
1.12
1.10
1.09
OH
O
OTs
2bf
2.00
2.04
1.01
2.07
1.04
2.07
2.06
5.09
3.04
f1 (ppm)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129.88
127.92
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113.07
112.54
68.52
66.45
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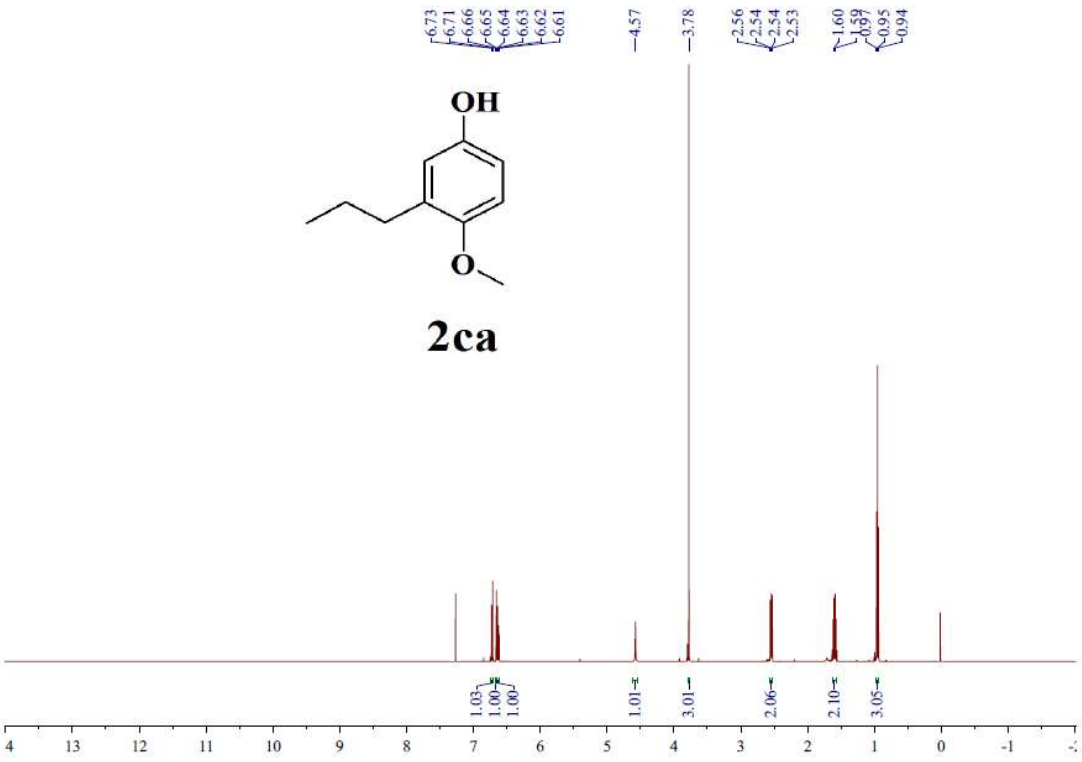
14.06
OH
O
OTs
2bf
f1 (ppm)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6.66
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6.64
6.63
6.62
6.61
4.57
3.78
2.56
2.54
2.54
2.53
1.60
0.97
0.95
0.94
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O
2ca
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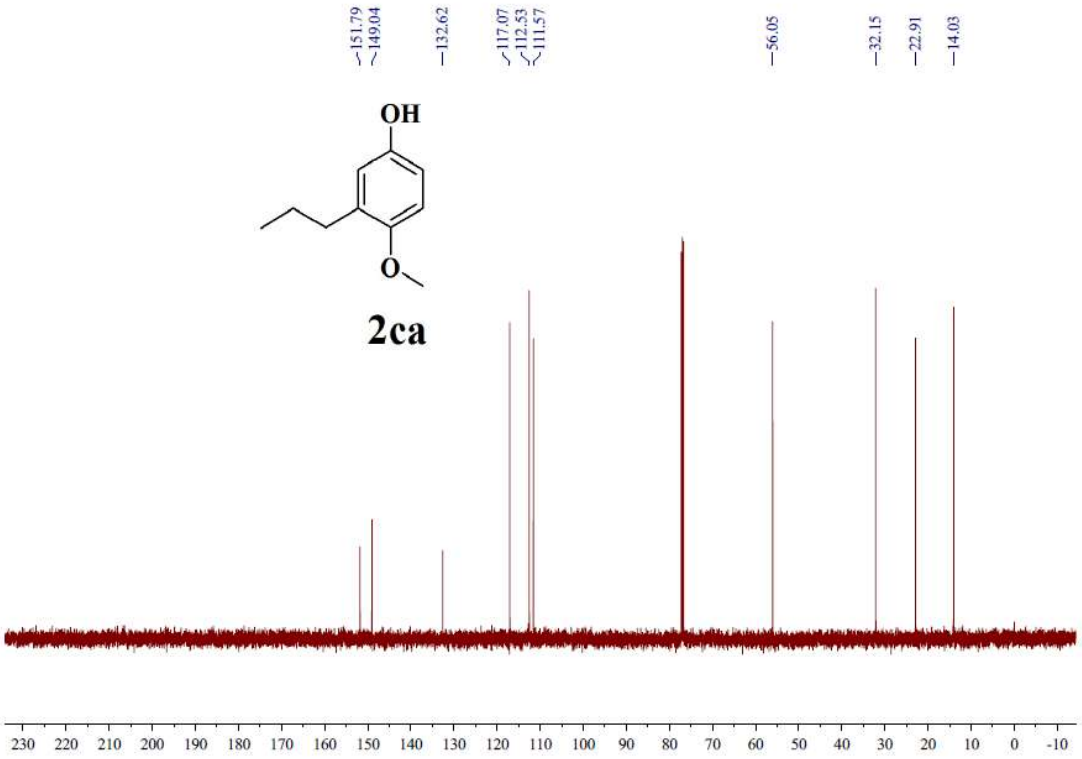
1.00
1.00
1.01
3.01
2.06
2.10
3.05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132.62
117.07
112.53
111.57
56.05
32.15
22.91
14.03
OH
O
2ca
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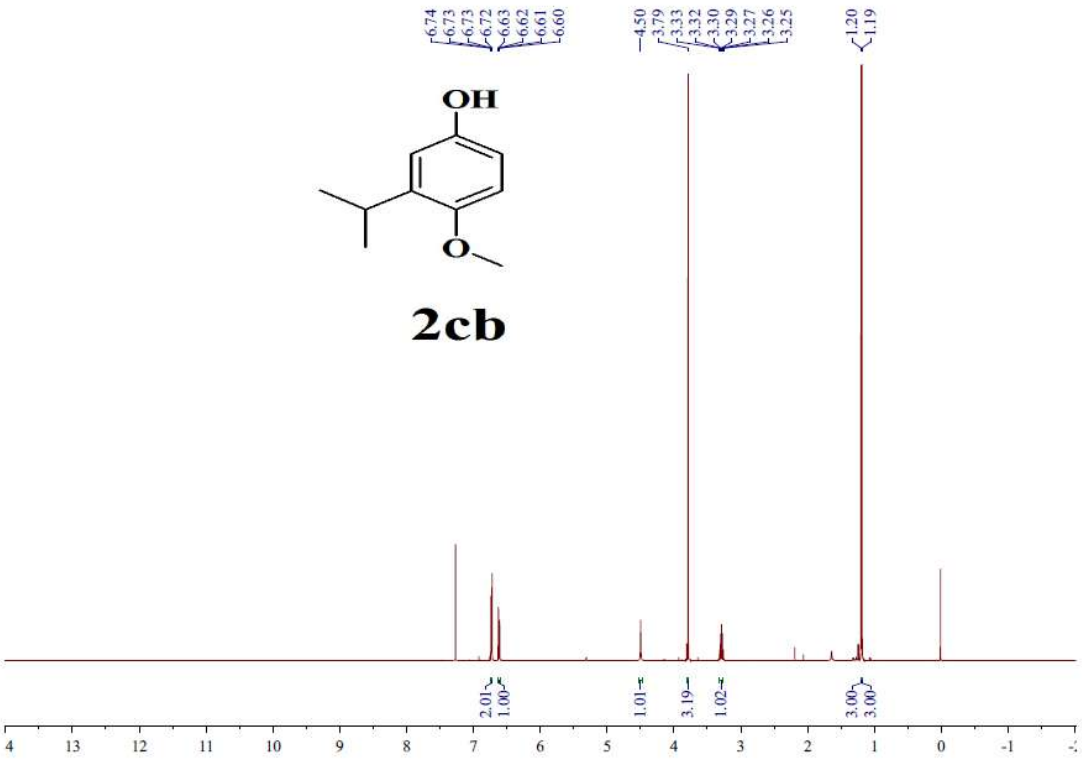
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6.73
6.73
6.72
6.63
6.62
6.61
6.60
4.50
3.79
3.33
3.32
3.30
3.29
3.27
3.26
3.25
1.20
1.19
OH
O
2cb
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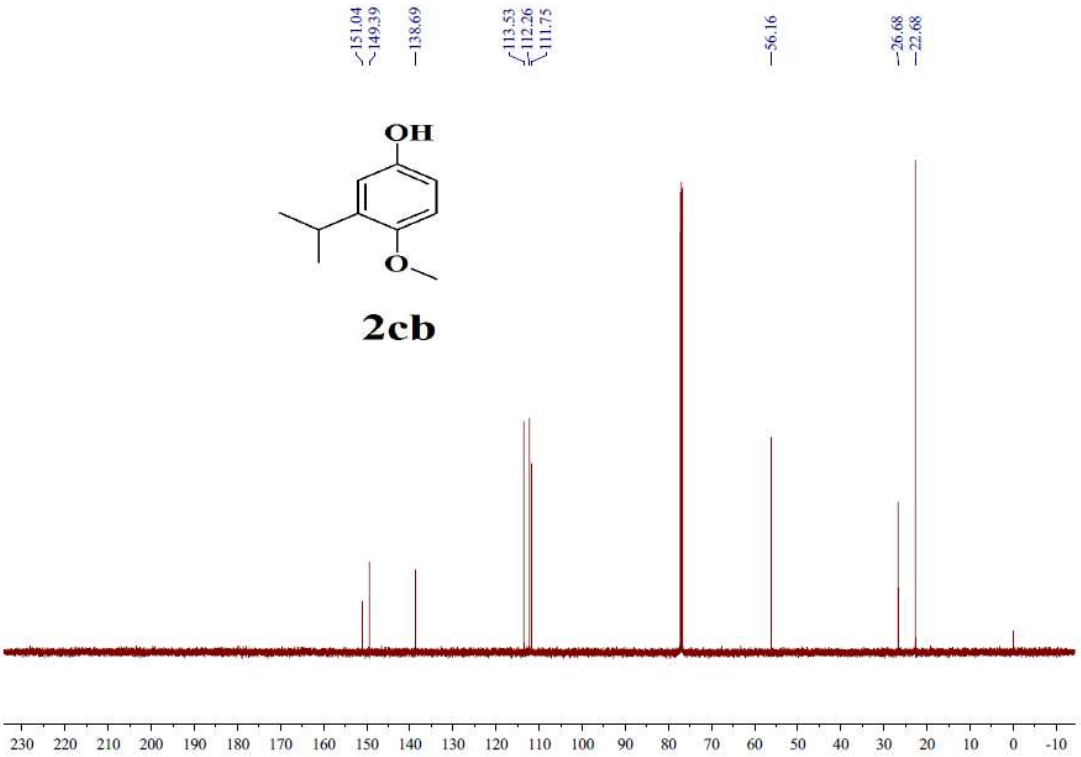
1.00
1.01
3.19
1.02
3.00
3.00
4 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1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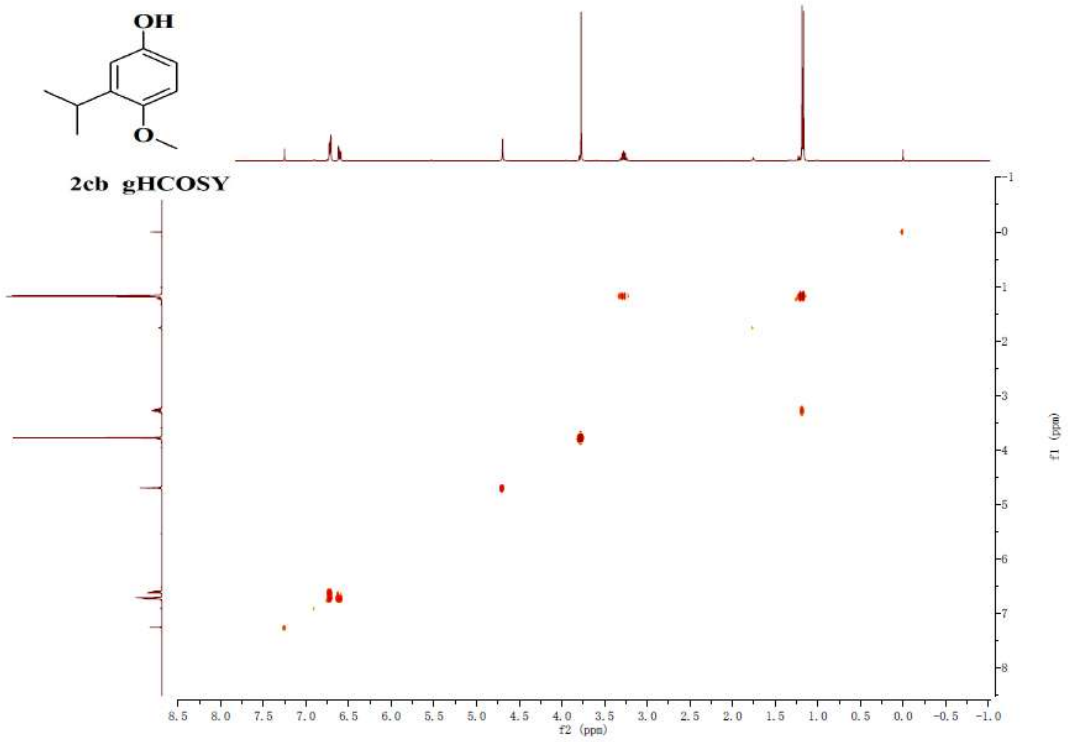
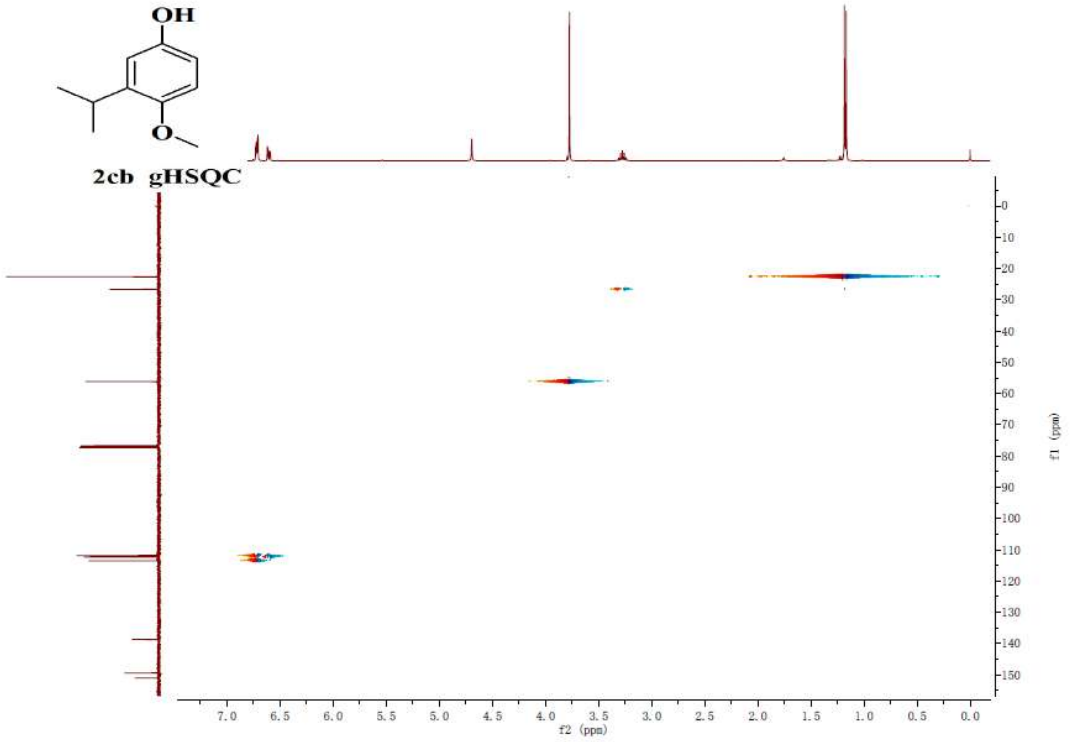
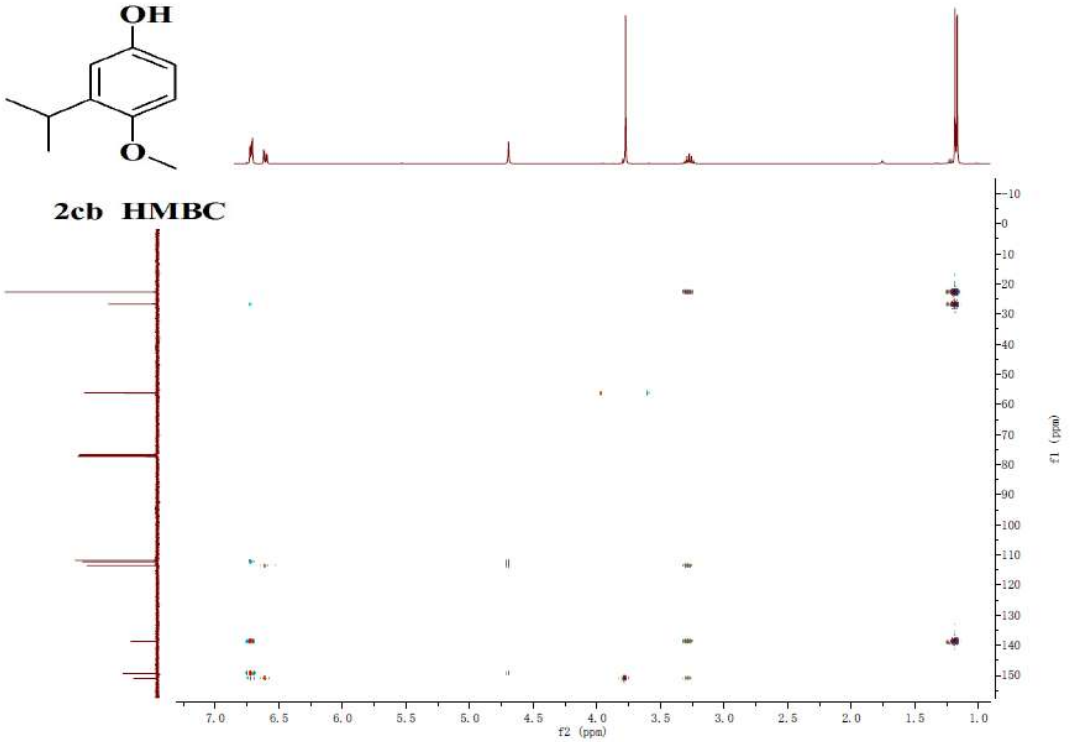
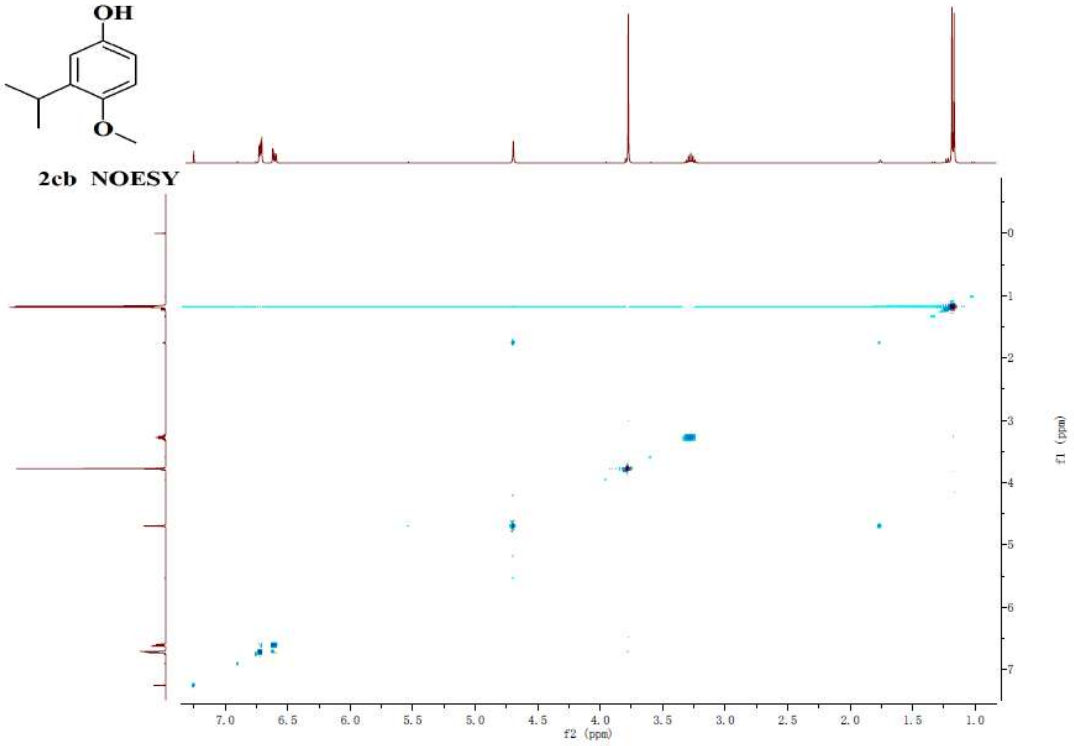
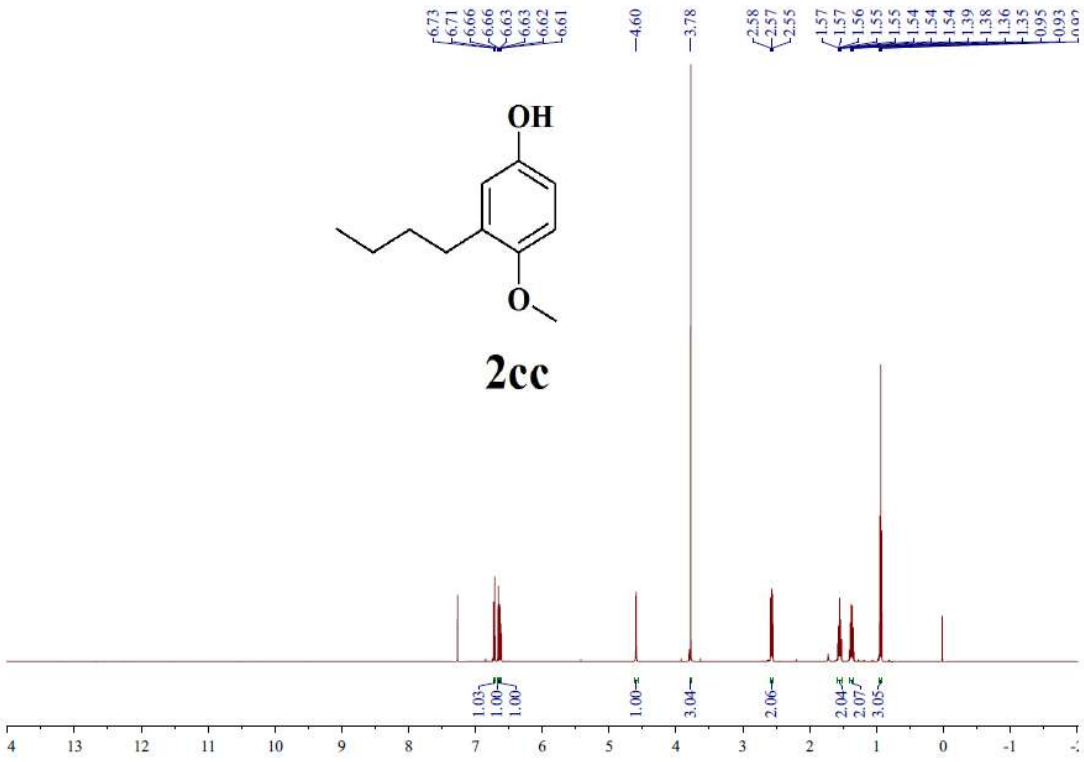
111.75
56.16
26.68
22.68
OH
O
2cb


OH
O
2cb gHCOSY
f2 (ppm)
f1 (ppm)


OH
O
2cb gHSQC
f2 (ppm)
f1 (ppm)


OH
O
2cb HMBC
f2 (ppm)
f1 (ppm)


OH
O
2cb NOESY
f2 (ppm)
f1 (ppm)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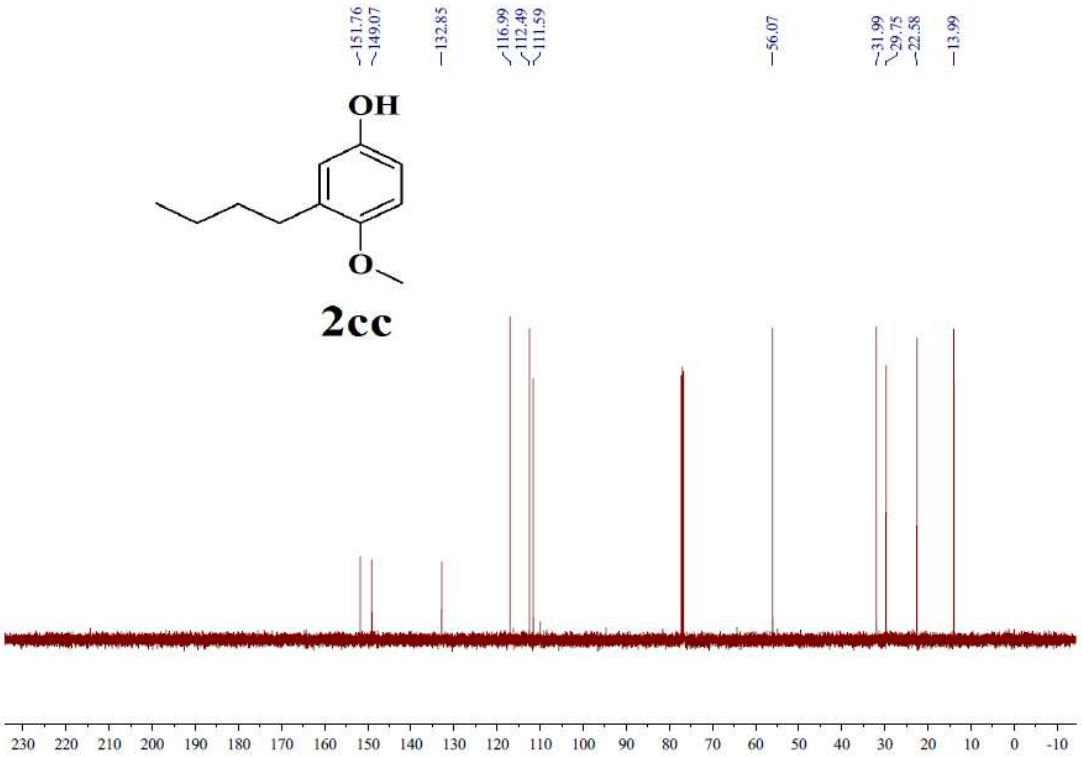
O
2cc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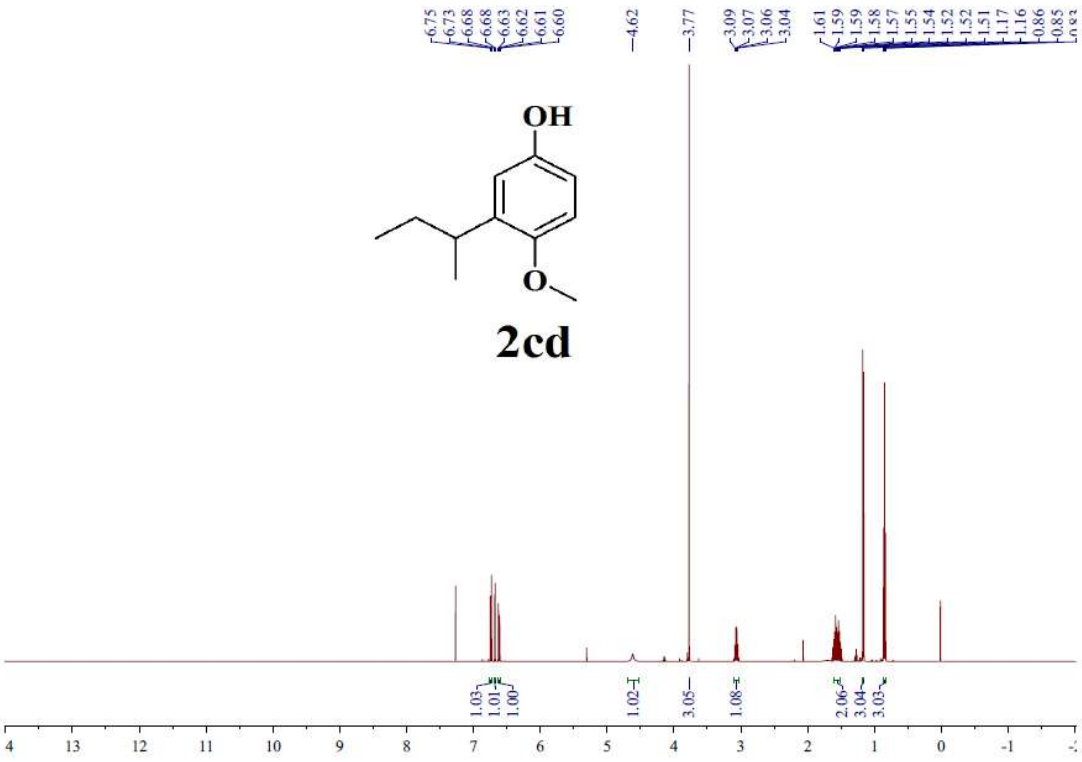
13.99
OH
O
2cc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6.60
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3.06
3.04
1.61
1.59
1.59
1.58
1.57
1.55
1.54
1.52
1.52
1.51
1.17
1.16
0.86
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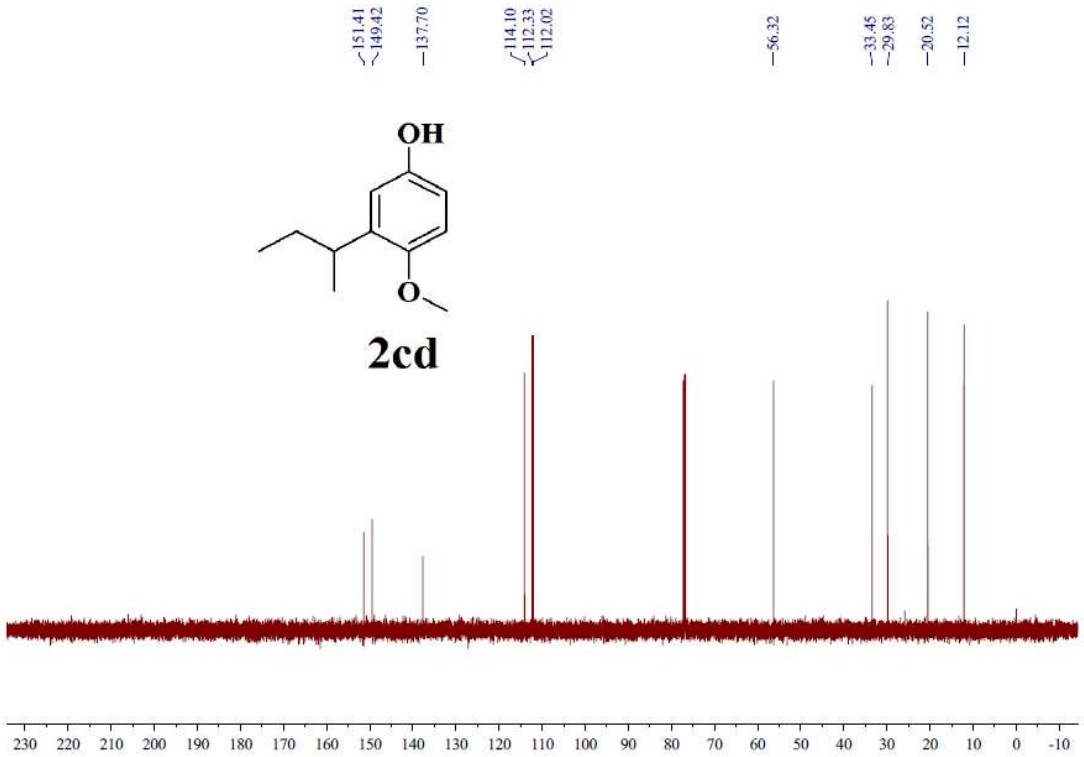
3.04
3.03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20.52
12.12
OH
O
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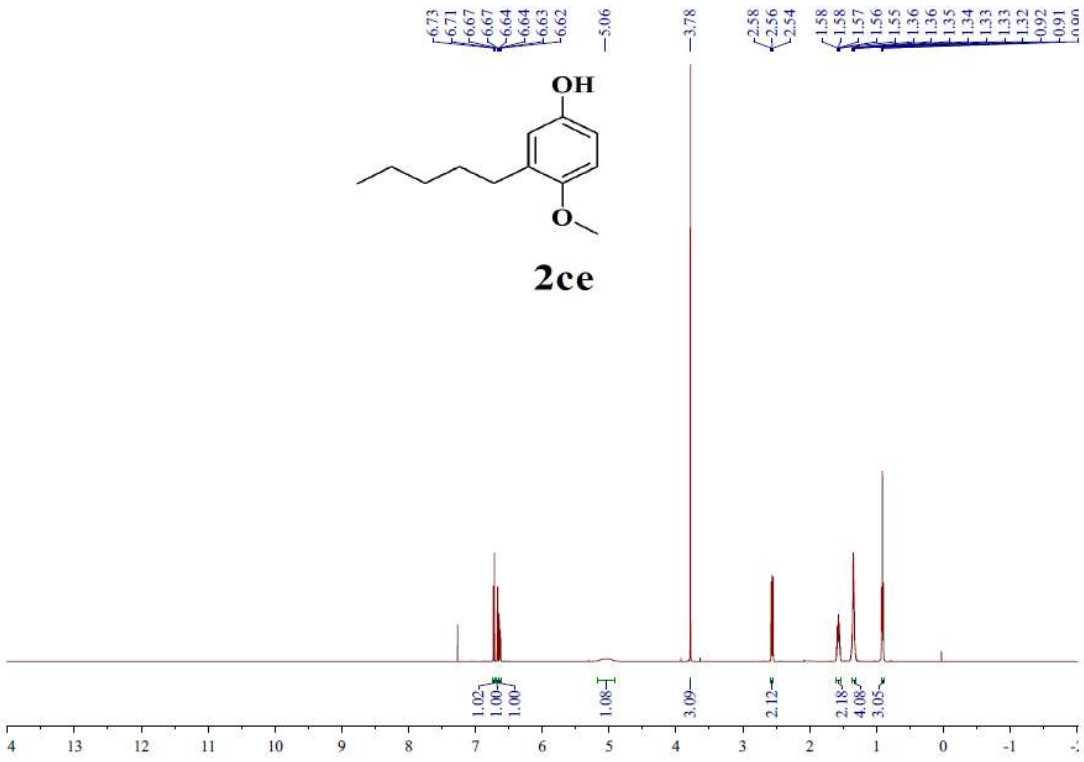
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6.63
6.62
5.06
3.78
2.58
2.56
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1.58
1.57
1.56
1.55
1.36
1.36
1.35
1.34
1.33
1.33
1.32
0.92
0.91
OH
O
2ce
1.02
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1.08
3.09
2.12
2.18
4.08
3.05
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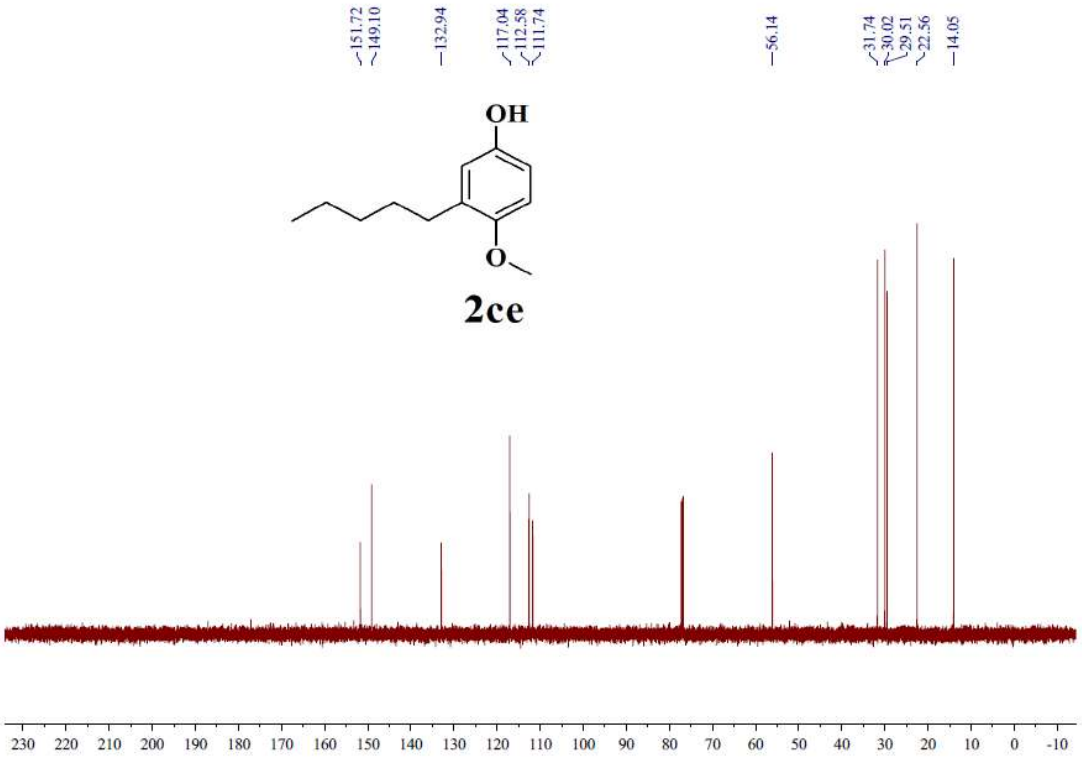
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O
2ce
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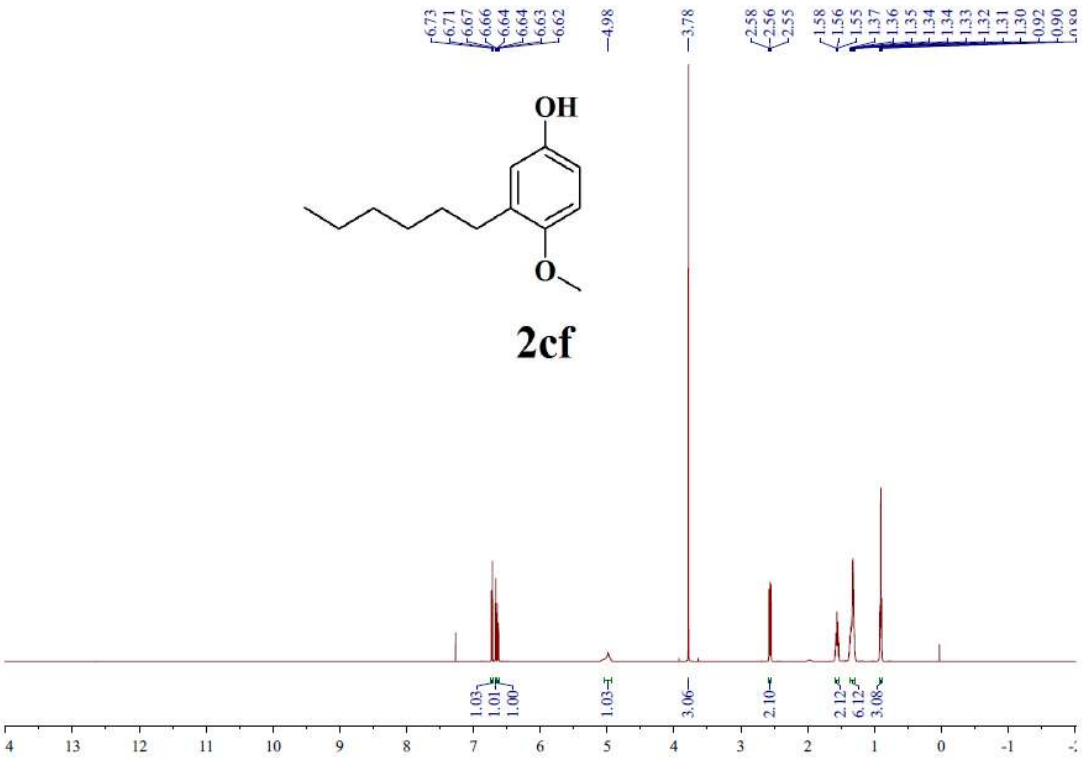
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1.56
1.55
1.37
1.36
1.35
1.34
1.34
1.33
1.32
1.31
1.30
0.92
0.90
OH
O
2cf
1.03
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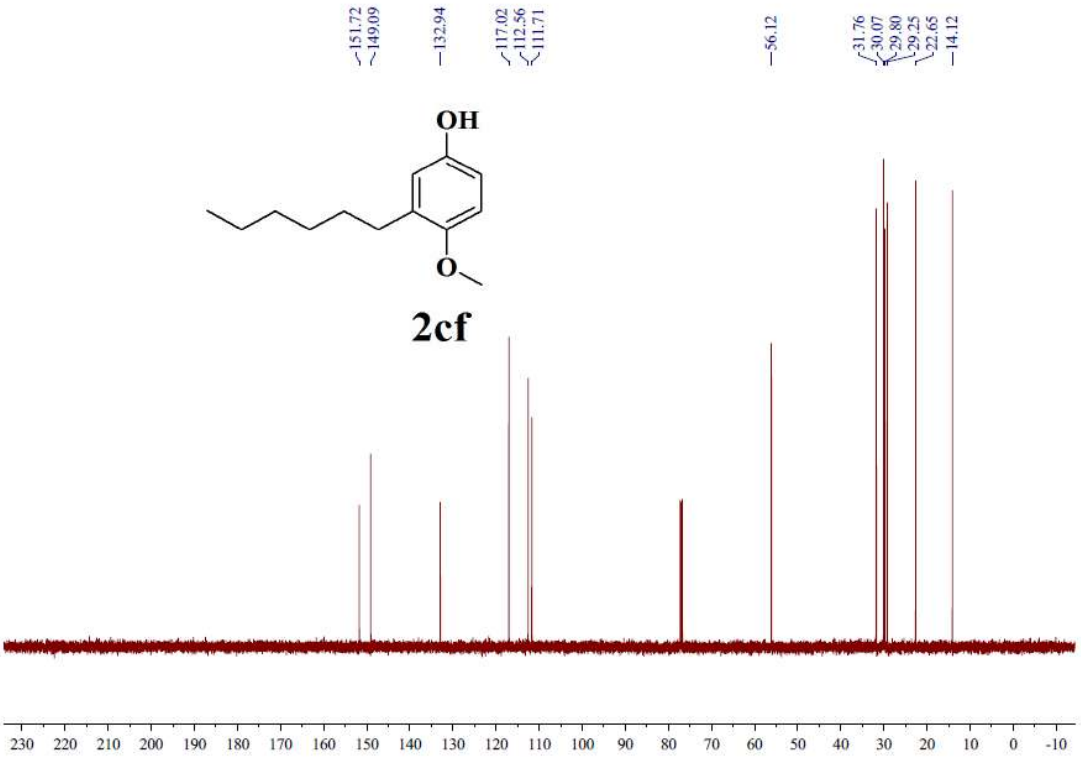
1.03
3.06
2.10
2.12
6.12
3.08
4 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1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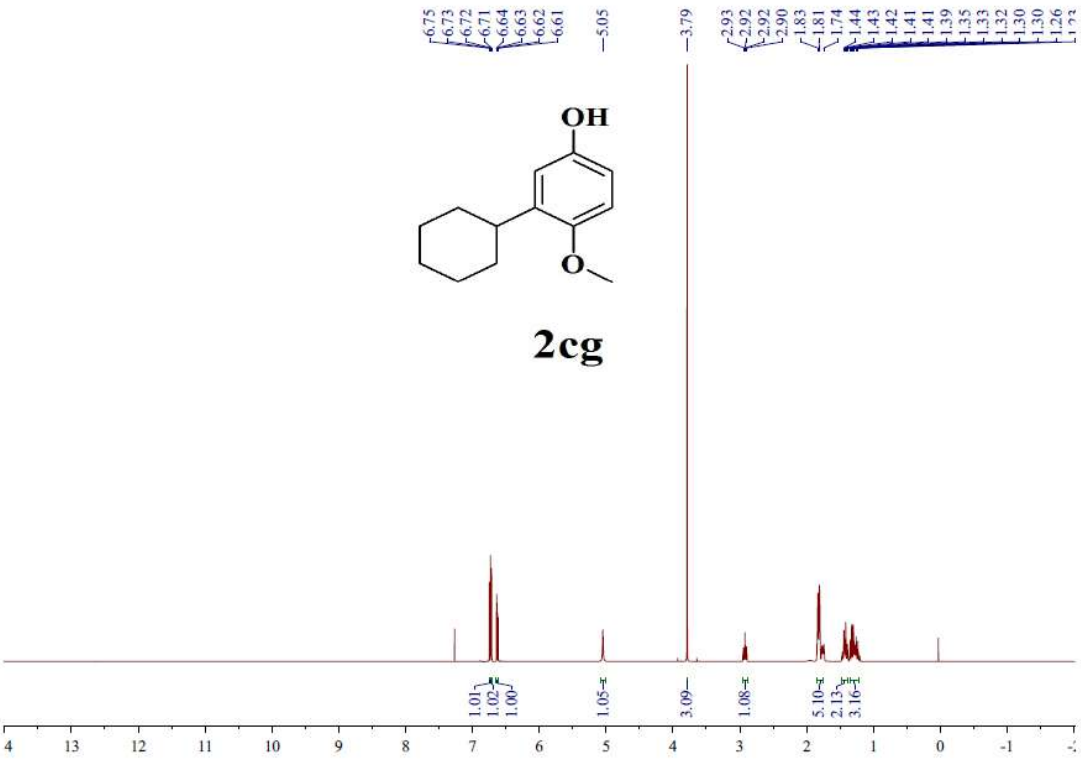
O
2cf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1.74
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1.43
1.42
1.41
1.41
1.39
1.35
1.33
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1.30
1.30
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1.08
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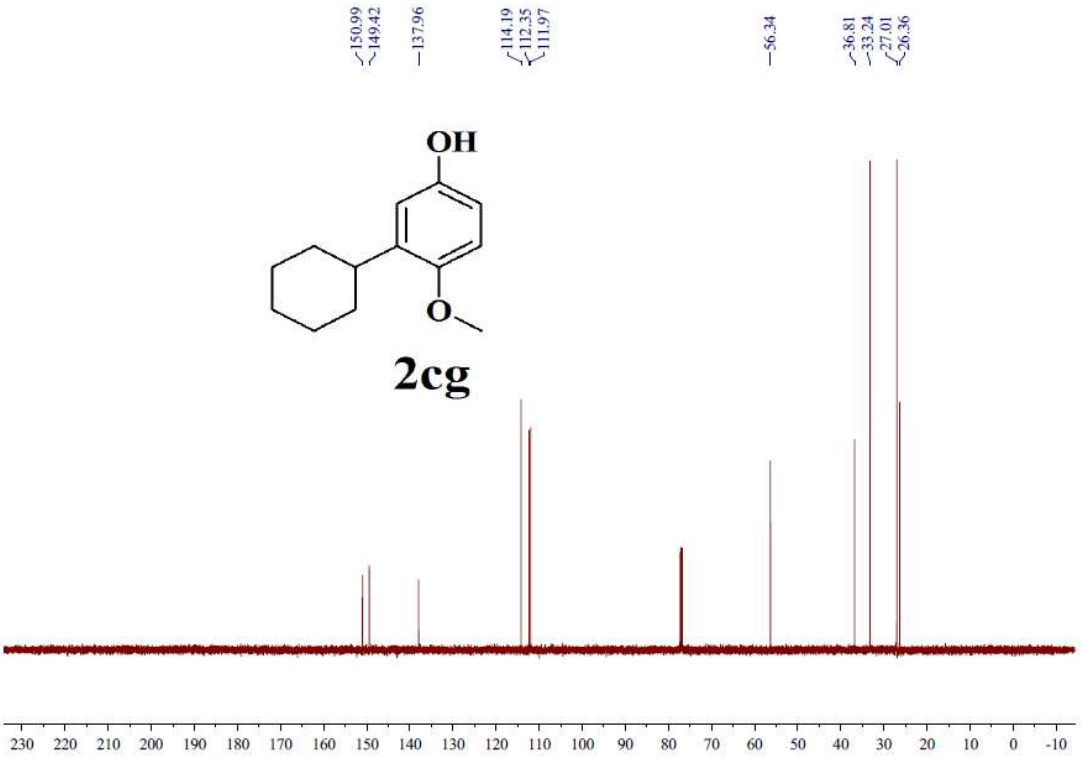
3.16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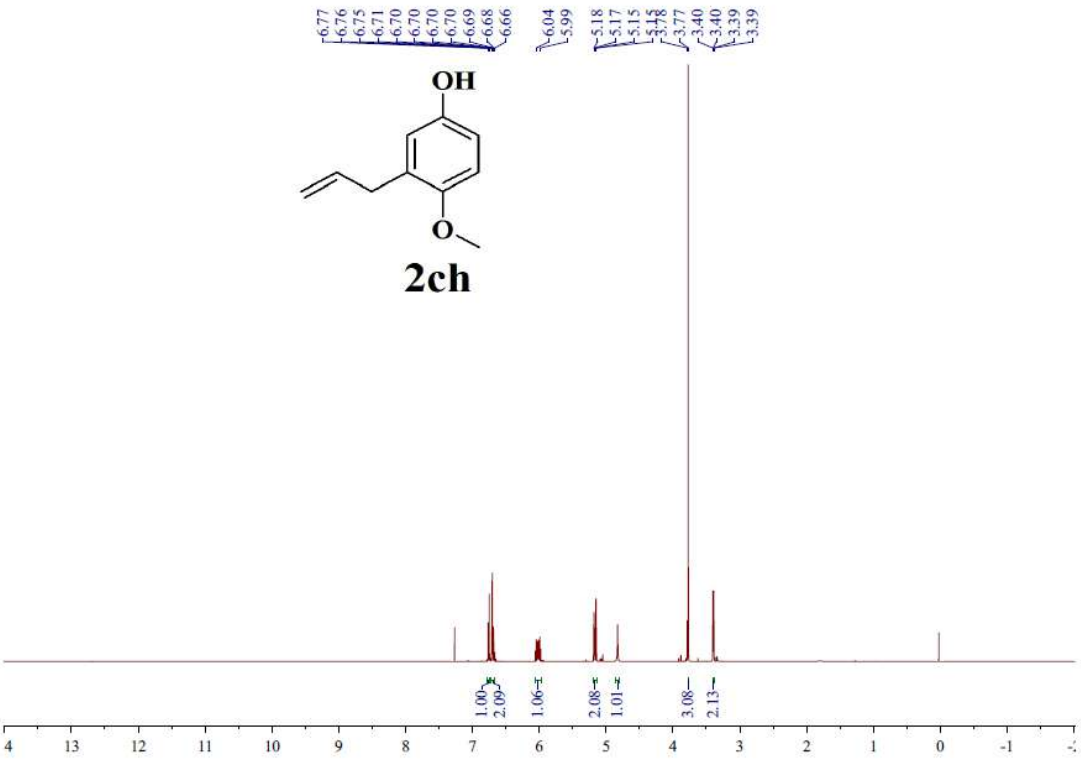
27.01
26.36
OH
O
2cg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5.18
5.17
5.15
5.15
3.78
3.77
3.40
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1.06
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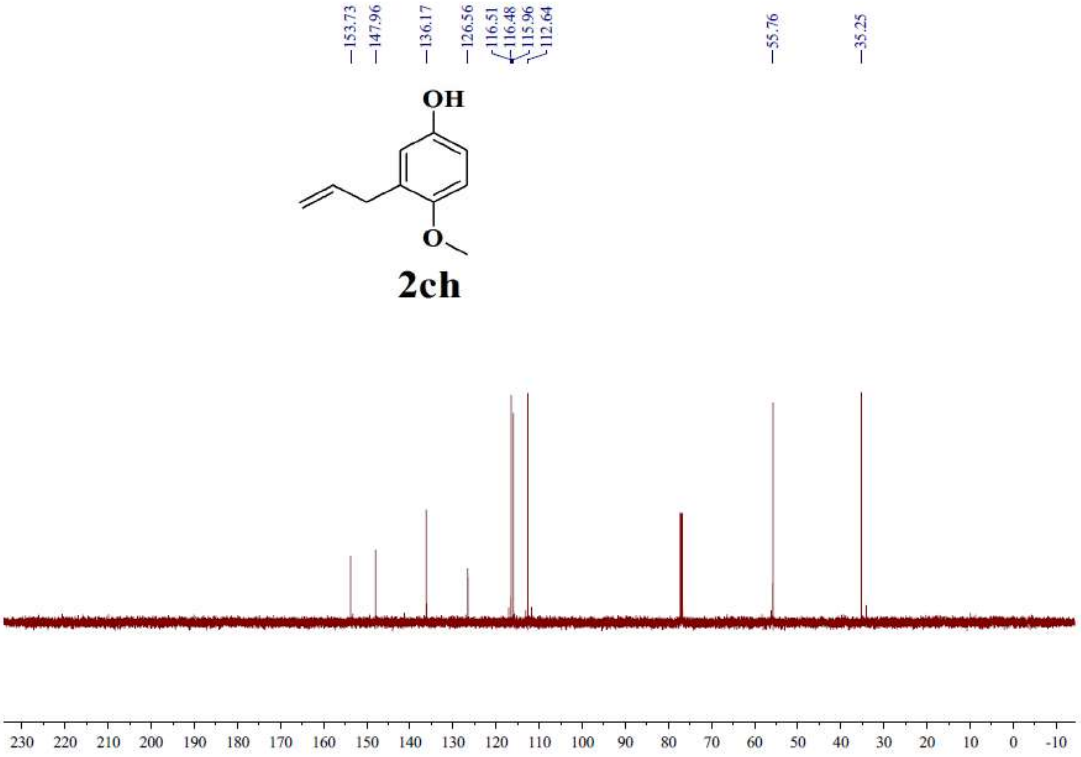
1.01
3.08
2.13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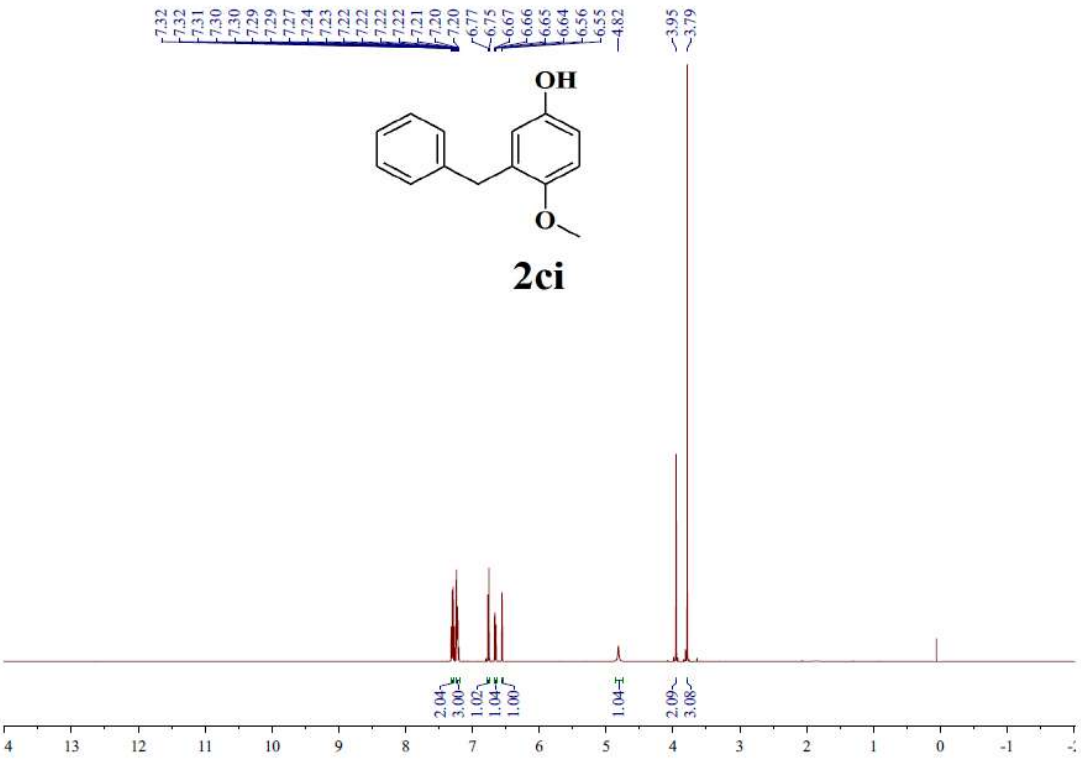
O
2ch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6.75
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6.66
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6.64
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6.55
4.82
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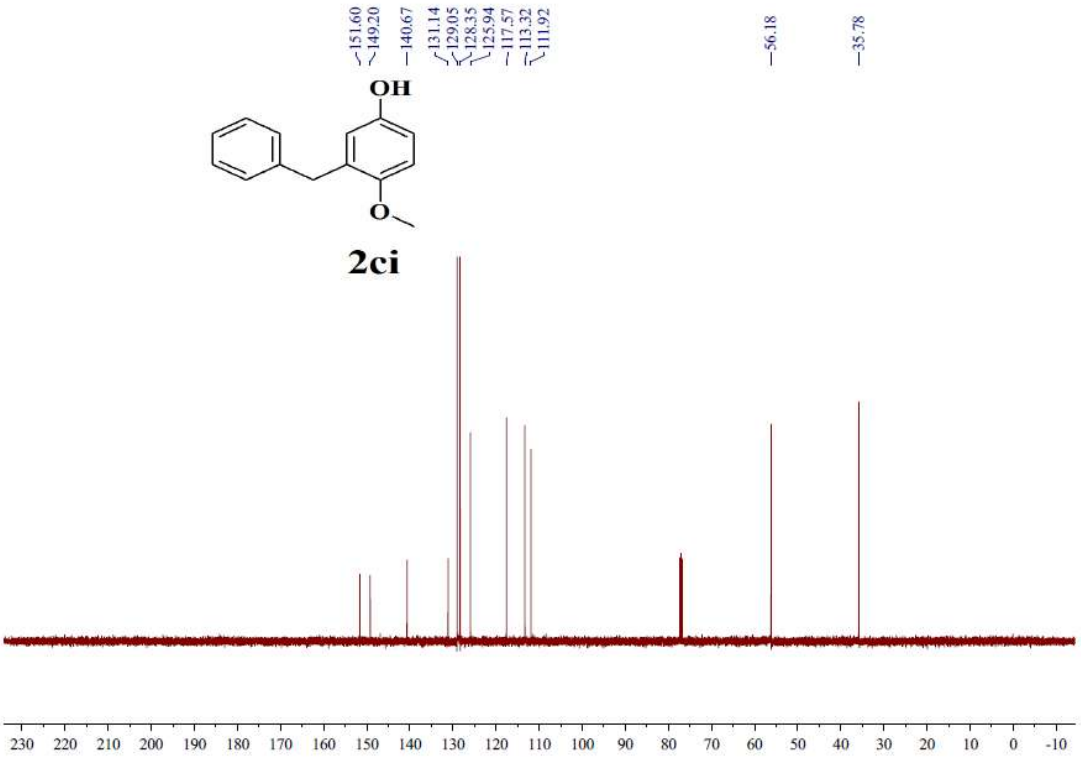
1.04
1.00
1.04
2.09
3.08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128.35
125.94
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113.32
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56.18
35.78
OH
O
2ci
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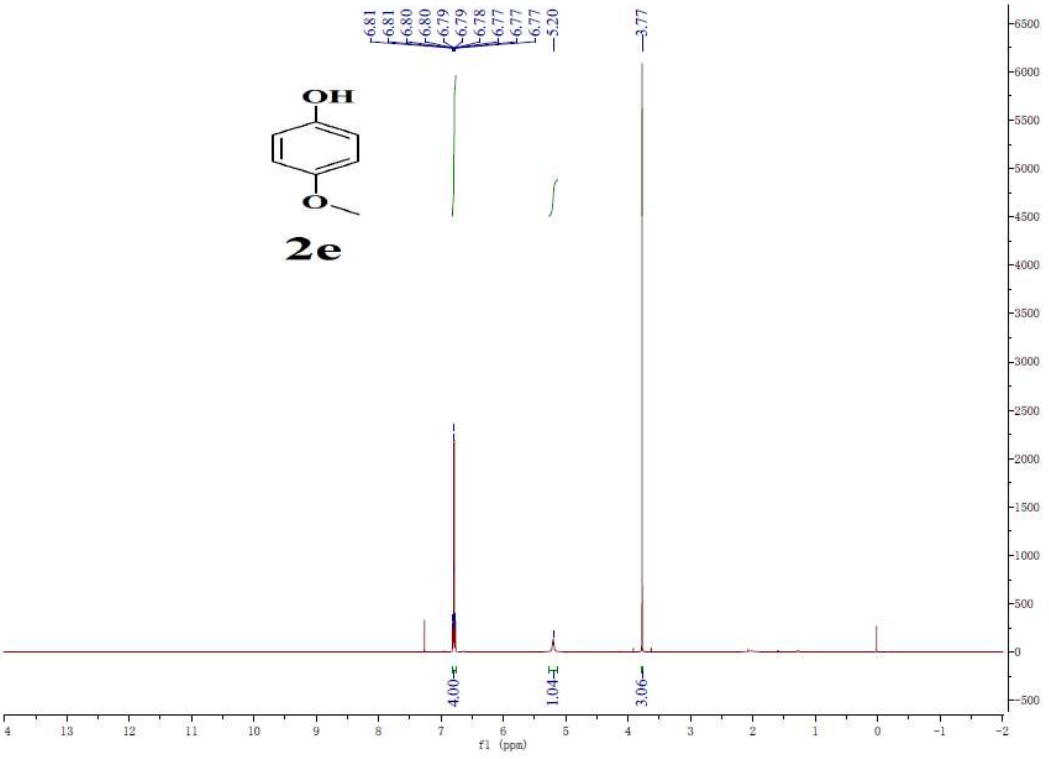
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6.79
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5.20
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OH
O
2e
4.00
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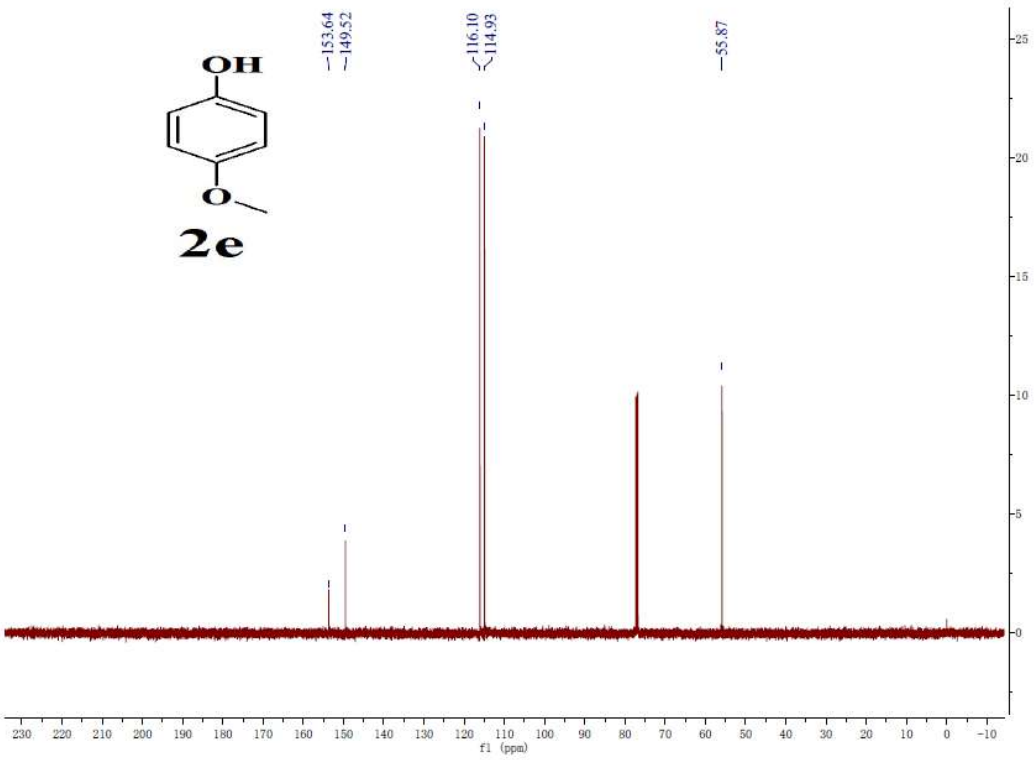
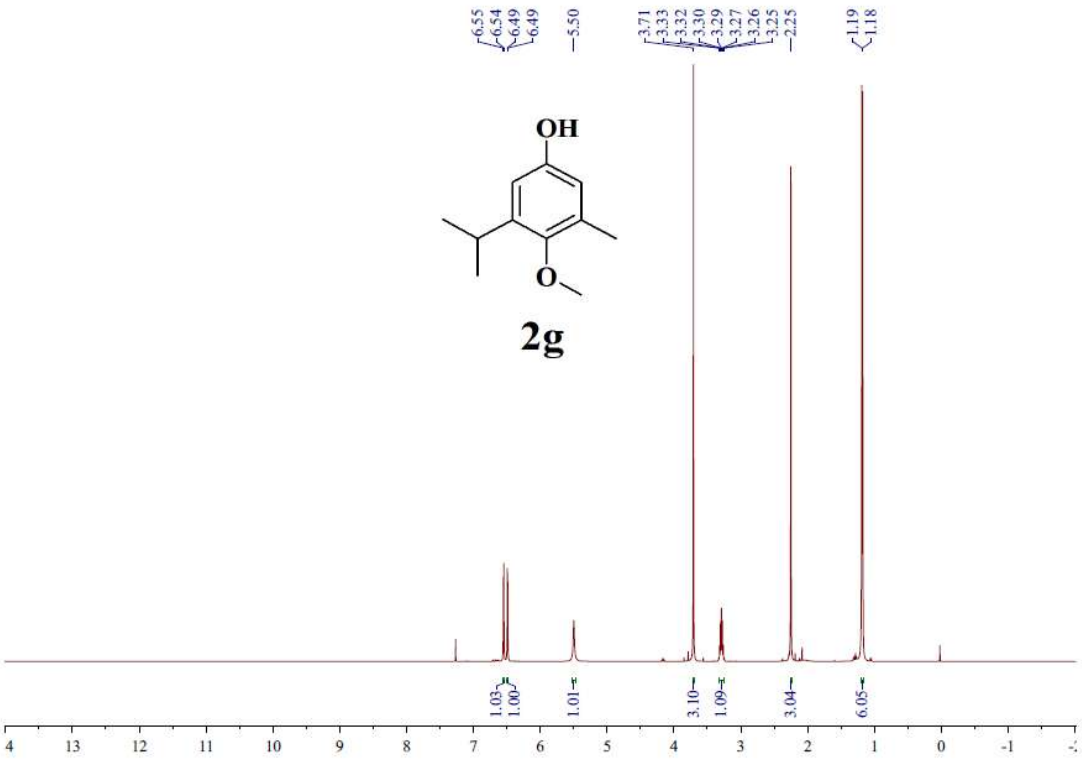
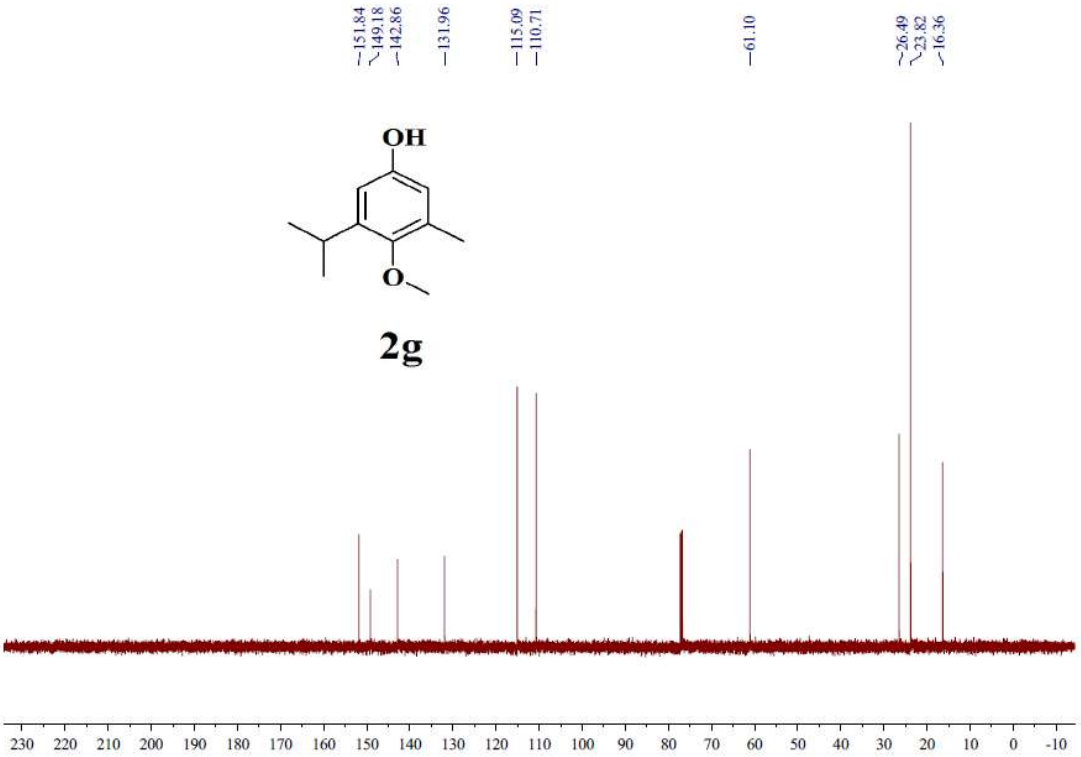
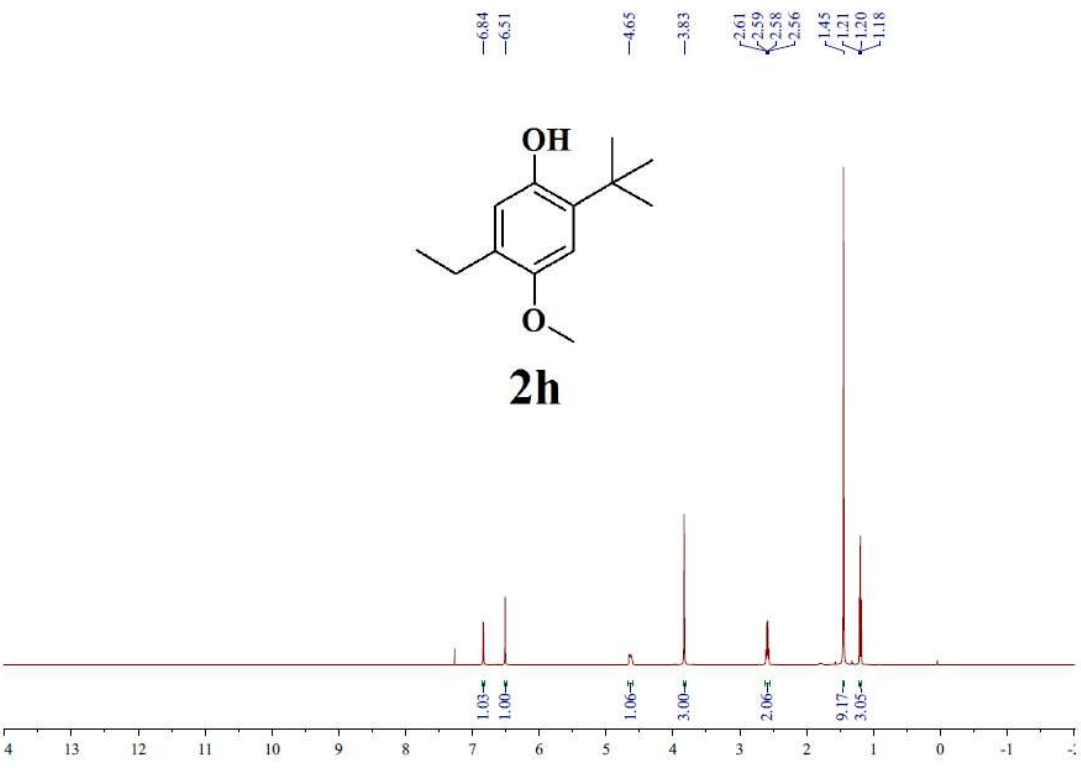
3.06
f1 (ppm)


OH
O
2e
153.64
149.52
116.10
114.93
55.87
f1 (ppm)

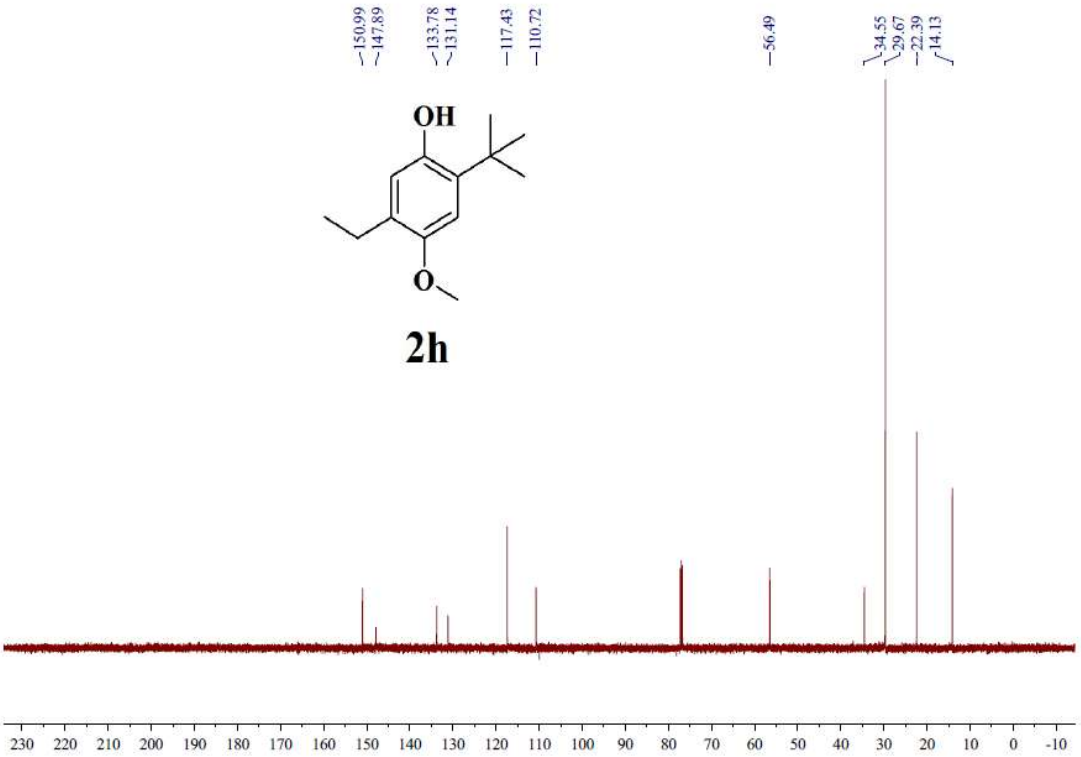

OH
O
2g
6.55
6.54
6.49
6.49
5.50
3.71
3.33
3.32
3.30
3.29
3.27
3.26
3.25
2.25
1.19
1.18
1.03
1.00
1.01
3.10
1.09
3.04
6.05

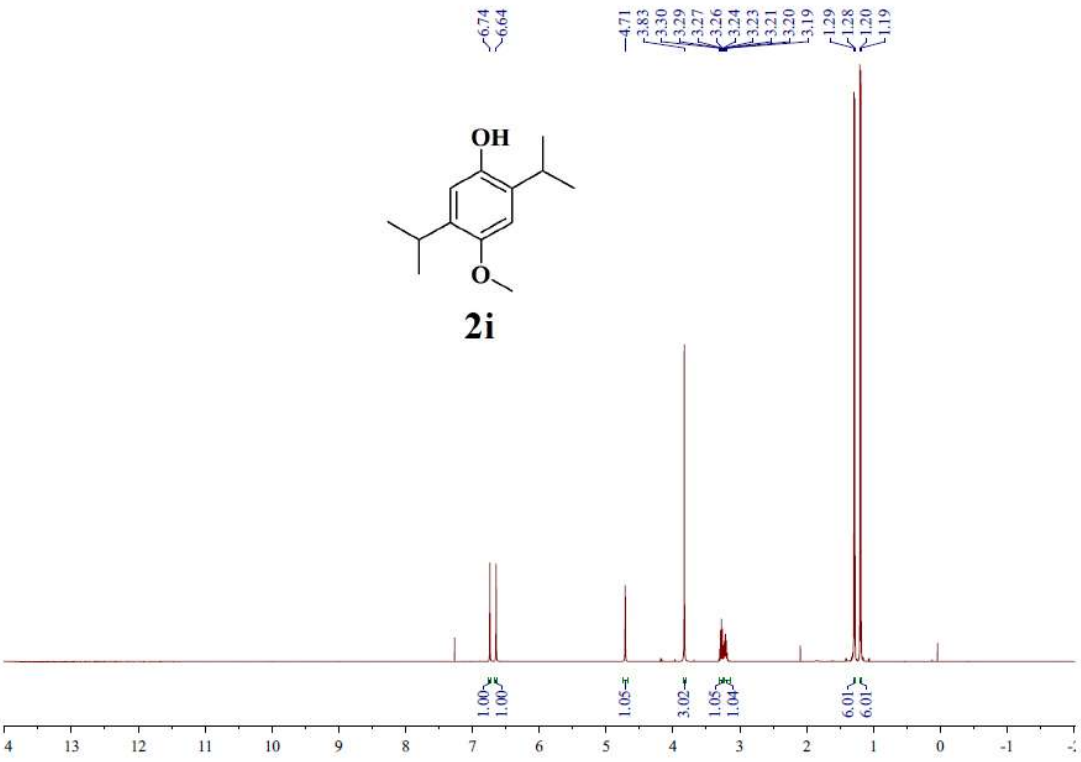

151.84
149.18
142.86
131.96
115.09
110.71
61.10
26.49
23.82
16.36
OH
O
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

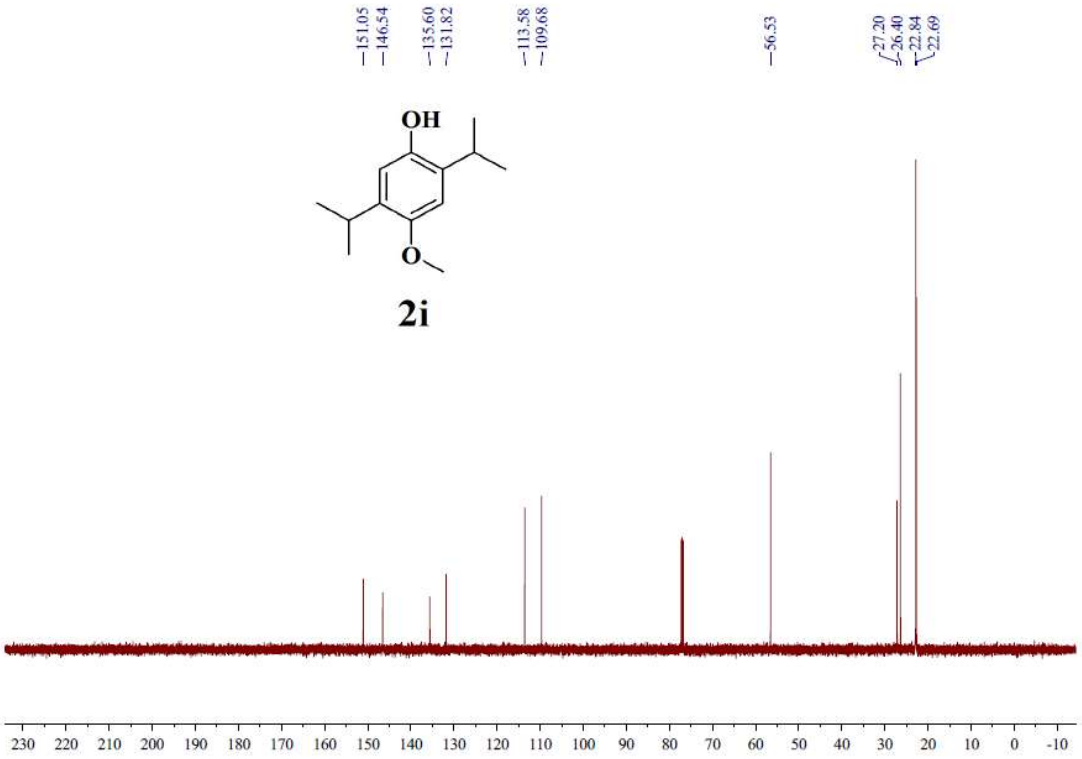
2g

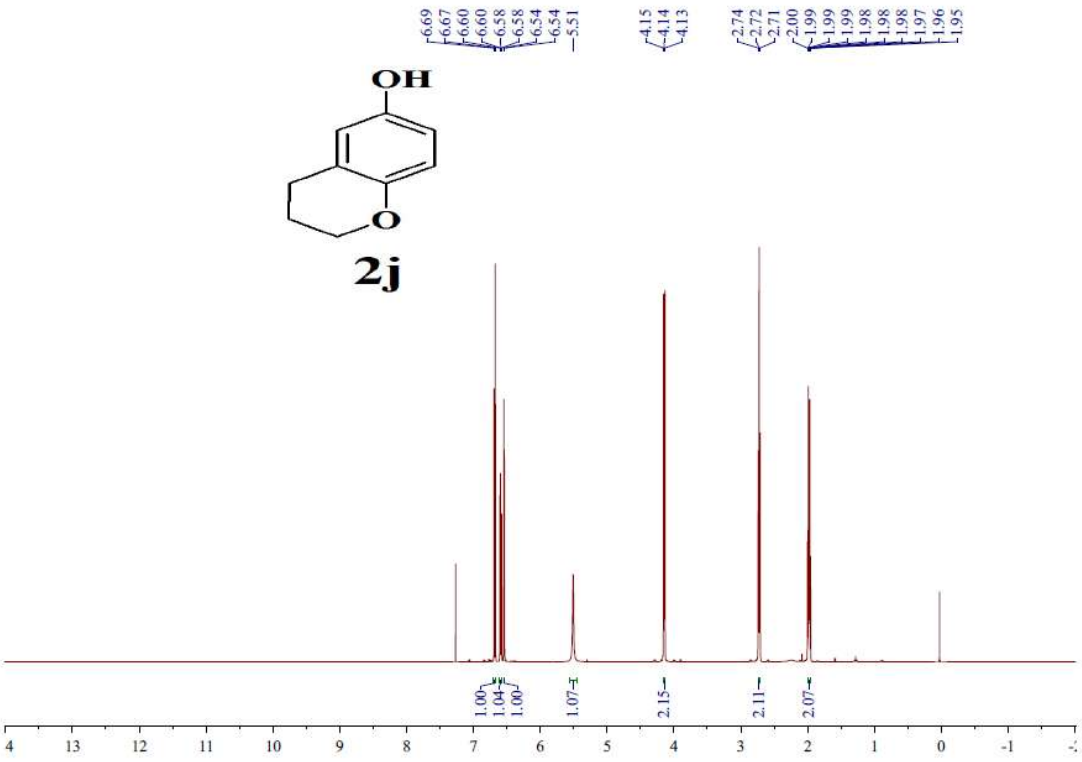

6.84
6.51
4.65
3.83
2.61
2.59
2.58
2.56
1.45
1.21
1.20
1.18
OH
O
1.03
1.00
1.06
3.00
2.06
9.17
3.05
4 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1

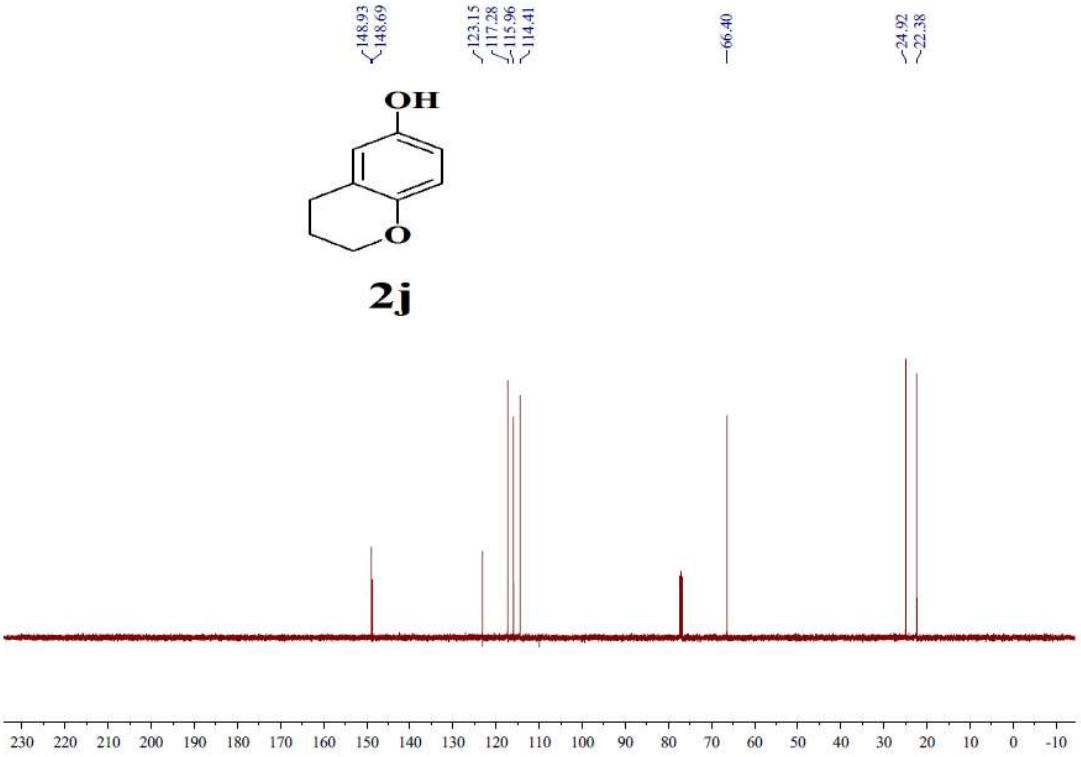
2h

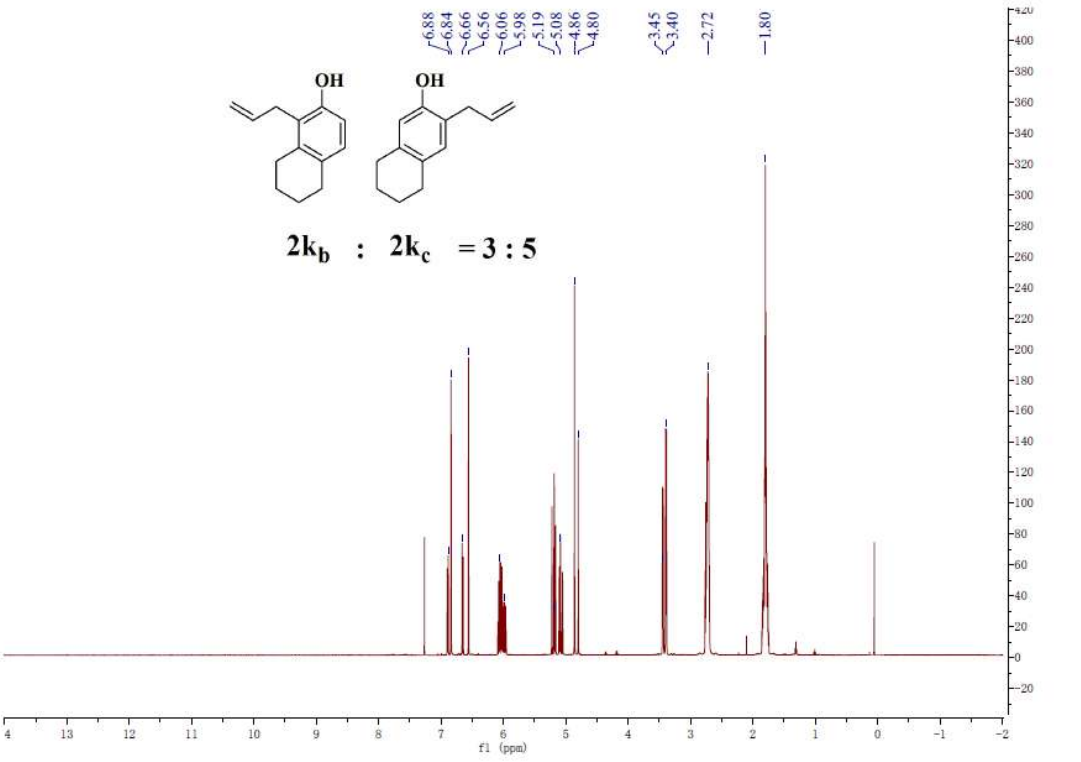

150.99
147.89
133.78
131.14
117.43
110.72
56.49
34.55
29.67
22.39
14.13
OH
O
2h
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

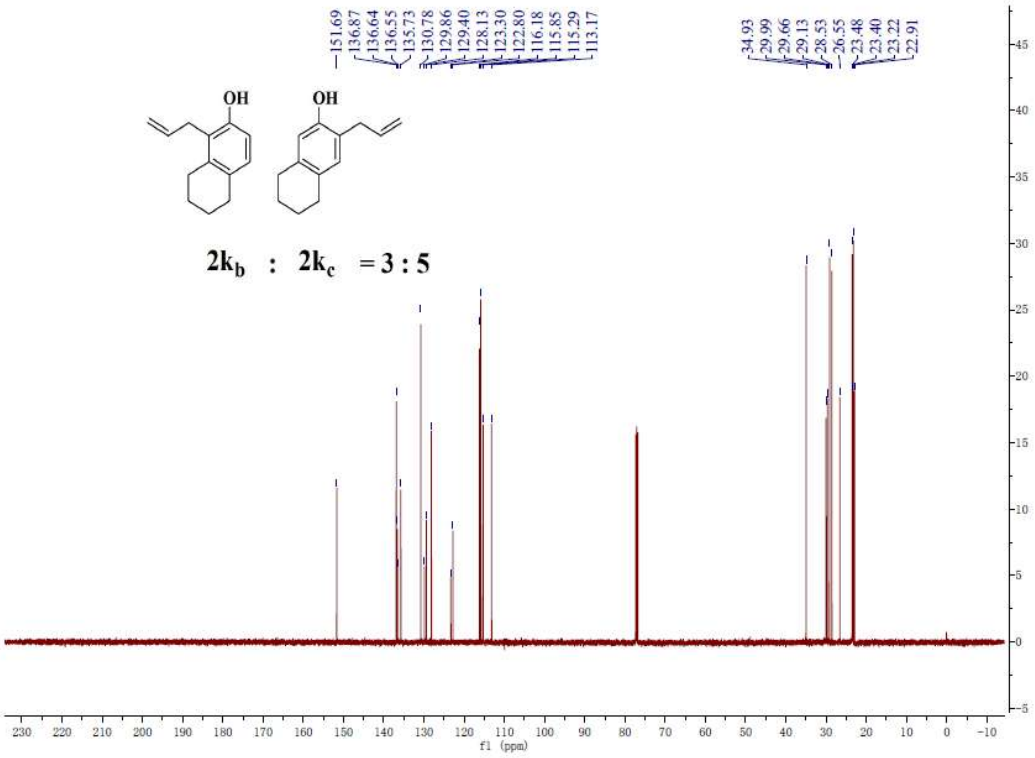

6.74
6.64
4.71
3.83
3.30
3.29
3.27
3.26
3.24
3.23
3.21
3.20
3.19
1.29
1.28
1.20
1.19
OH
O
2i
1.00
1.00
1.05
3.02
1.05
1.04
6.01
6.01
4 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1


151.05
146.54
135.60
131.82
113.58
109.68
56.53
27.20
26.40
22.84
22.69
OH
O
2i

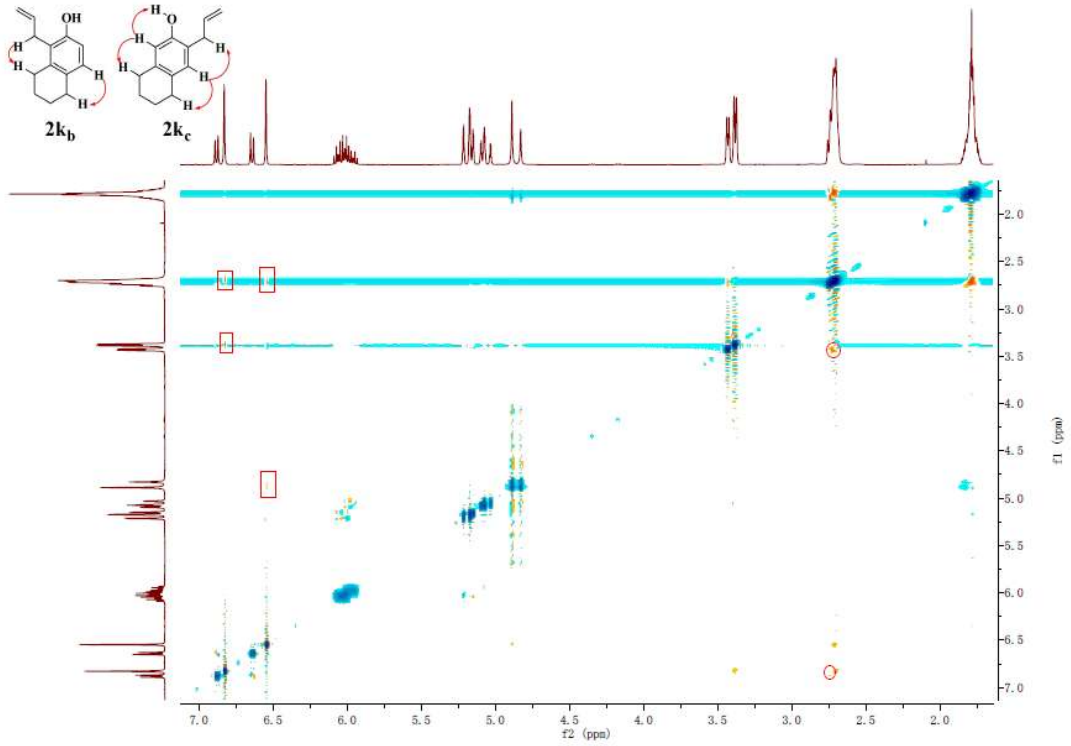

6.69
6.67
6.60
6.60
6.58
6.58
6.54
6.54
5.51
4.15
4.14
4.13
2.74
2.72
2.71
2.00
1.99
1.99
1.99
1.98
1.98
1.98
1.97
1.96
1.95
OH
O
2j
1.00
1.04
1.00
1.07
2.15
2.11
2.07


148.93
148.69
123.15
117.28
115.96
114.41
66.40
24.92
22.38
OH
O
2j


6.88
6.84
6.66
6.56
6.06
5.98
5.19
5.08
4.86
4.80
3.45
3.40
2.72
1.80
OH
OH
2k_b : 2k_c = 3 : 5
f1 (ppm)



NOE Spectrum:



HMQC Spectrum: