Electronic Supplementary Material (ESI) for Green Chemistry.

Supporting Information

A Metal-free Transformation of Alkynes to Carbonyls Directed by Remote OH Group

Dao-Qian Chen, Chun-Huan Guo, Heng-Rui Zhang, Dong-Po Jin, Xue-Song Li, Pin Gao, Xin-Xing Wu, Xue-Yuan Liu* and Yong-Min Liang

State key Laboratory of Applied Organic Chemistry, Lanzhou University,

Lanzhou 730000, P. R. China.

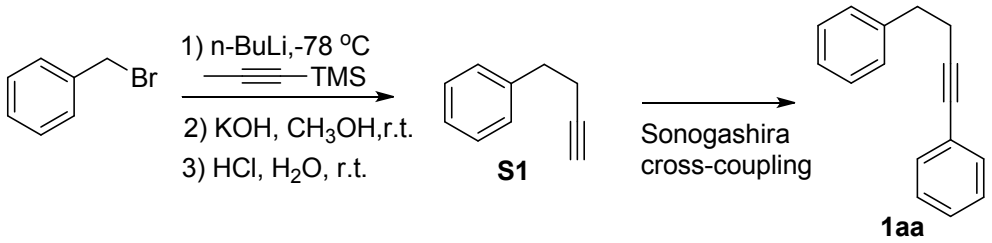
Table of Contents

1. General remarks

Column chromatography was carried out on silica gel and analytical TLC was performed with silica gel GF254 plates. NMR spectra were recorded on a Bruker advance Ⅲ 400 spectrometer in $CDCl_3$ at 400 MHz (^1H NMR), 100 MHz (^{13}C NMR). IR spectra were recorded on a FT-IR spectrometer and only major peaks are reported in cm^{-1}. Data collections for crystal structure were performed at room temperature (295 K). Melting points were determined on a microscopic apparatus and were uncorrected. High-resolution mass spectral analysis (HRMS) data were measured on a Bruker Apex II. All products were further characterized by high resolution mass spectra (HRMS). Copies of their ^1H NMR, ^{13}C NMR are provided.

Unless otherwise noted, solvents obtained from commercial suppliers were used without further purification. Homopropargylic alcohols were prepared according to literature procedures[1].

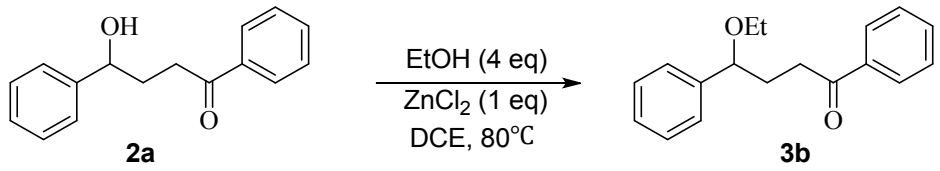
2. General procedure for the synthesis of substrates:
A: 1aa were prepared in the method:



Under a argon atmosphere, to the solution of 0.67g trimethyl(prop-1-yn-1-yl)silane in anhydrous THF (50 mL) in a 100 mL round-bottom flask was added n-BuLi (2.5M in Hexane, 1.1 eq, 5.5mmol, 2.2ml） at -78℃. Then the reaction was stirred for 2 hours at the same temperature. To the solution, benzyl bromide (0.59 mL, 5 mmol) in the 10 mL THF was then injected. After addition, the reaction was moved to room temperature for further 1 hour then quenched with 30 mL sat. NaCl (aq). The aqueous layer was extracted with ether and the extracts were combined with the above organic layer. The combined solution was dried over Na_2SO_4. After evaporation of the solvent, the crude residue was directly dissolved in CH_3OH (40ml). KOH (336mg, 6mmol) was then added the solution, which was stirred overnight. The reaction was neutralized with 8 mL HCl (1N) and was extracted with ether（30 mL*3） and the extracts were combined with the above organic layer. The combined solution was dried over Na_2SO_4. After evaporation of the solvent, the residue was purified by column chromatography (silica gel, appropriate mixture of *n*-hexane/ethyl acetate= 50/1) to afford **S1**(80%).

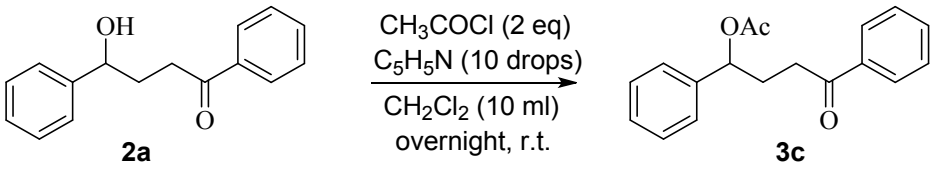
The preparation of **1aa** from **S1** is a simple procedure[1].

B: 3b are prepared in the method:



2a (48mg, 0.2 mmol), $ZnCl_2$ (27.2 mg, 0.2 mmol) and EtOH (0.8 mmol) were successively added to a solution of anhydrous DCE (2.0 mL). After the reactor was sealed, the reaction mixture was stirred at 80 °C for 2 hour. The organic phases were extracted with ethyl acetate (3 * 5 mL) and the combined extracts were dried (Na_2SO_4). After evaporation of the solvent, the residue was purified by column chromatography on silica gel to give **3b** (43 mg, 80 %).

C:3aa[2], 3a and 3c were prepared in the method:

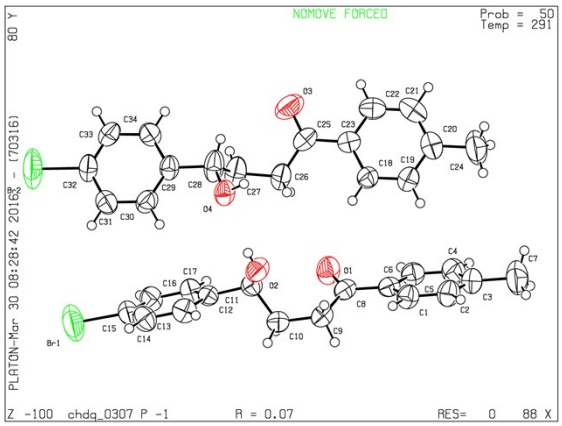


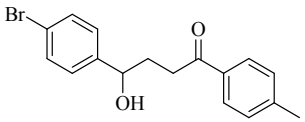
CH_3COCl (2 eq, 1.5 mmol) was added dropwise to a solution of **2a** (0.75 mmol, 168 mg) in 10 ml anhydrous CH_2Cl_2. After pyridine (10 drops) was added successively, the reactor was stirred at r.t. overnight. The reaction mixture was quenched with 2 mL HCl (1N), and washed by water then extracted with CH_2Cl_2 (2* 20 mL). The organic phases were combined, and washed by sat. NaCl (aq.) then dried over sodium sulfate. The mixture was concentrated in vacuo and then purified by column chromatography on silica gel to afford **3c** (65%).

3. General experimental procedure

General procedure for synthesis of 4-hydroxy-1,4-diphenylbutan-1-one from homopropargylic alcohol: homopropargylic alcohol **1a** (44.4 mg, 0.2 mmol), HOAc (1mL) and EtOH (1 mL) were added to a reactor, and the reactor was flushed with O_2 and sealed. The reaction mixture was stirring at 60 °C for 5.5 hour. The organic phases were extracted with ethyl acetate (3 * 5 mL) and the combined extracts were dried (Na_2SO_4). After evaporation of the solvent, the mixture was concentrated in vacuo and then purified by column chromatography (*n*-hexane/ethyl acetate= 20/1~10/1) on silica gel to afford **2a** (85%).

4. Crystallographic data of 2p





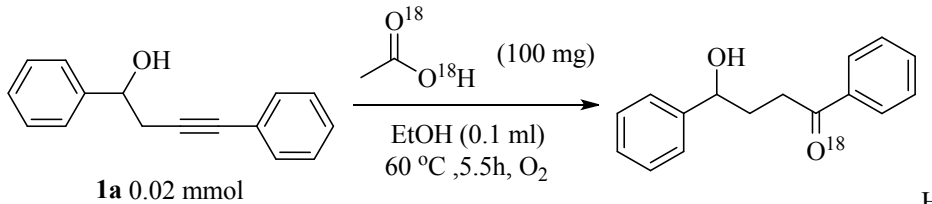
2p

Bond precision: C-C = 0.0071 A Wavelength=0.71000
Cell: a=9.5598(6) b=9.6393(7) c=18.0624(12)
alpha=75.207(6) beta=81.884(6) gamma=72.871(6)
Temperature: 291 K

	Calculated	Reported
Volume	1533.97(19)	1533.97(18)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C17 H17 Br O2	C17 H17 Br O2
Sum formula	C17 H17 Br O2	C17 H17 Br O2
Mr	333.21	333.22
Dx,g cm-3	1.443	1.443
Z	4	4
Mu (mm-1)	2.676	2.678
F000	680.0	680.0
F000’ 678.77		
h,k,lmax	11,11,21	11,11,21
Nref	5791	5612
Tmin,Tmax	0.552,0.570	0.786,1.000
Tmin’ 0.501		

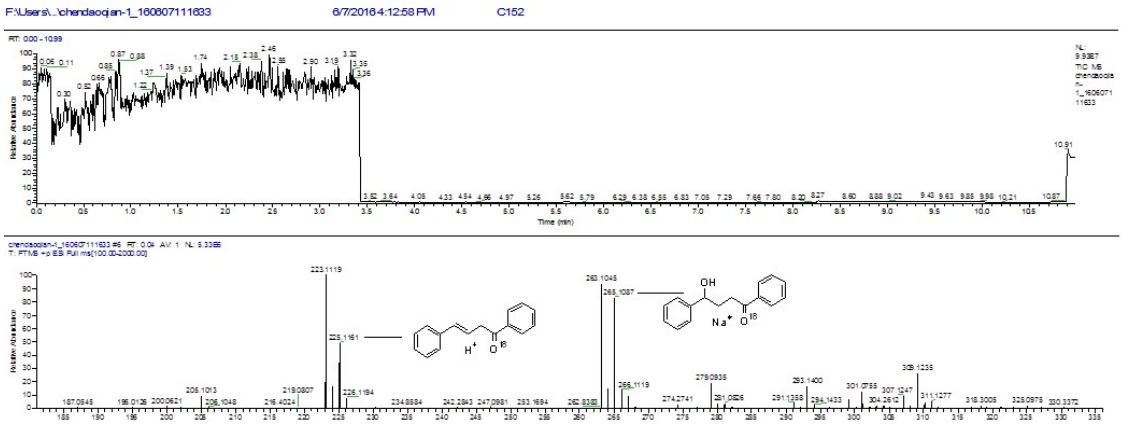
Correction method= # Reported T Limits: Tmin=0.786 Tmax=1.000
AbsCorr = MULTI-SCAN
Data completeness= 0.969 Theta(max)= 25.610
R(reflections)= 0.0673(3864) wR2(reflections)= 0.1845(5612)
S = 1.050 Npar= 368

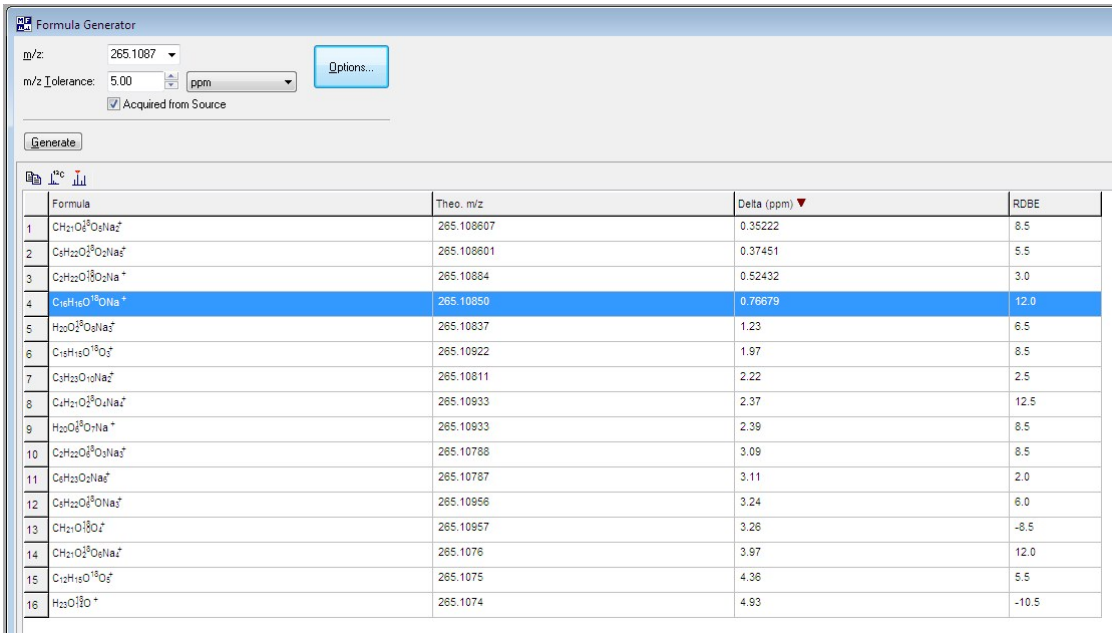
5. Reaction with ^{18}O labeled acetic acid



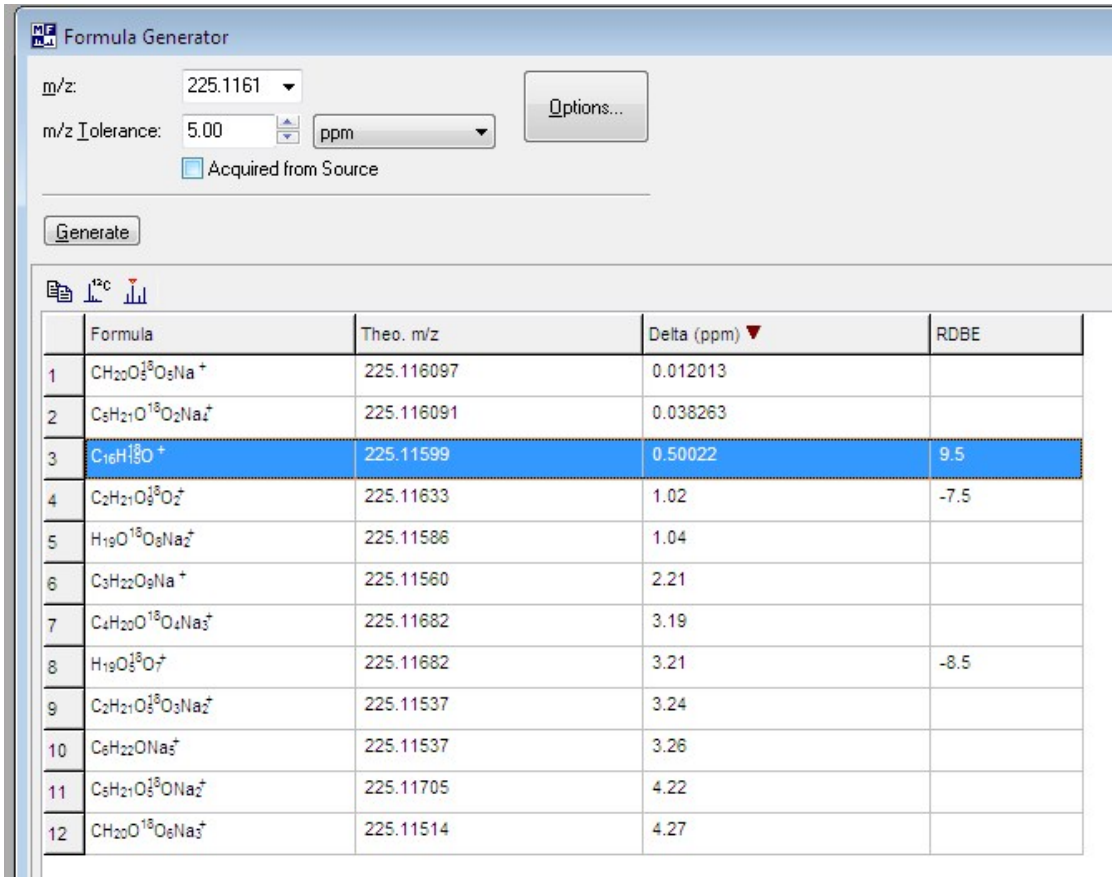
1a 0.02 mmol

Homopropargylic alcohol **1a** (4.44 mg, 0.02 mmol), HOAc (100 mg, from Aldrich, 95 atom % 18O) and EtOH (0.1 mL) were added to a reactor, and the reactor was flushed with O_2 and sealed. The reaction mixture was stirring at 60 °C for 5.5 hour.



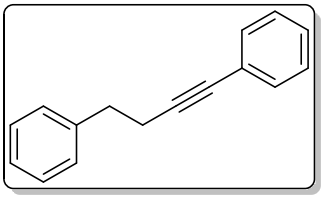


	Formula	Theo. m/z	Delta (ppm)	RDBE
1	$CH_{21}O_6^{18}O_3Na_2^+$	265.108607	0.35222	8.5
2	$C_9H_{22}O_4^{18}O_2Na_2^+$	265.108601	0.37451	5.5
3	$C_{23}H_{22}O_2^{18}O_2Na^+$	265.10884	0.52432	3.0
4	$C_{16}H_{16}O^{18}ONa^+$	265.10850	0.76679	12.0
5	$H_{20}O_2^{18}O_8Na_2^+$	265.10837	1.23	6.5
6	$C_{15}H_{15}O^{18}O_2^+$	265.10922	1.97	8.5
7	$C_3H_{23}O_{10}Na_2^+$	265.10811	2.22	2.5
8	$C_4H_{21}O_4^{18}O_4Na_2^+$	265.10933	2.37	12.5
9	$H_{20}O_6^{18}O_7Na^+$	265.10933	2.39	8.5
10	$C_2H_{22}O_4^{18}O_3Na_2^+$	265.10788	3.09	8.5
11	$C_6H_{23}O_2Na_6^+$	265.10787	3.11	2.0
12	$C_8H_{22}O_4^{18}ONa_2^+$	265.10956	3.24	6.0
13	$CH_{21}O_1^{18}O_4^+$	265.10957	3.26	-8.5
14	$CH_{21}O_2^{18}O_6Na_2^+$	265.1076	3.97	12.0
15	$C_{12}H_{15}O^{18}O_2^+$	265.1075	4.36	5.5
16	$H_{23}O_1^{18}O^+$	265.1074	4.93	-10.5

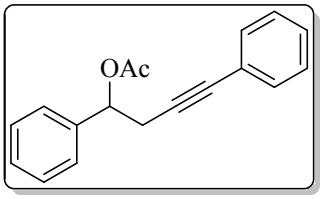


	Formula	Theo. m/z	Delta (ppm) ▼	RDBE
1	$CH_{20}O_3^{18}O_5Na^+$	225.116097	0.012013	
2	$C_5H_{21}O^{18}O_2Na_4^+$	225.116091	0.038263	
3	$C_{16}H_{19}^{18}O^+$	225.11599	0.50022	9.5
4	$C_2H_{21}O_9^{18}O_2^+$	225.11633	1.02	-7.5
5	$H_{19}O^{18}O_8Na_2^+$	225.11586	1.04	
6	$C_3H_{22}O_9Na^+$	225.11560	2.21	
7	$C_4H_{20}O^{18}O_4Na_3^+$	225.11682	3.19	
8	$H_{19}O_5^{18}O_7^+$	225.11682	3.21	-8.5
9	$C_2H_{21}O_5^{18}O_3Na_2^+$	225.11537	3.24	
10	$C_6H_{22}ONa_5^+$	225.11537	3.26	
11	$C_5H_{21}O_5^{18}ONa_2^+$	225.11705	4.22	
12	$CH_{20}O^{18}O_6Na_3^+$	225.11514	4.27	

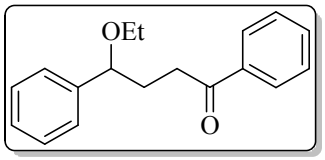
6. Characterization data of 1aa, 3a-3c, 2a-2t



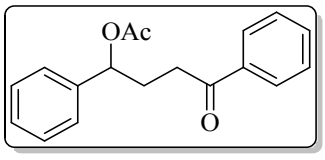
but-1-yne-1,4-diyldibenzene (1aa), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.68 (t, J = 7.6 Hz, 2H), 2.91 (t, J = 7.6 Hz, 2H), 7.19-7.23 (m, 1H), 7.23-7.32 (m, 7H), 7.35-7.38 (m, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 140.7, 131.5, 128.5, 128.4, 127.6, 123.8, 89.5, 81.3, 35.2, 21.6.



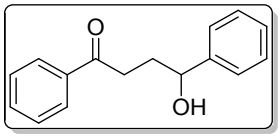
1,4-diphenylbut-3-yn-1-yl acetate (3a), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.12 (s, 3H), 2.95 (dd, J_1 = 6.8 Hz, J_2 = 16.8 Hz, 2H), 5.98 (t, J = 6.8 Hz, 1H), 7.25-7.26 (m, 3H), 7.31-7.33 (m, 3H), 7.35-7.38 (m, 2H), 7.43 (d, J = 6.8 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.0, 139.2, 131.5, 128.4, 128.3, 128.2, 127.8, 126.5, 123.3, 85.0, 82.8, 73.8, 27.5, 21.1.



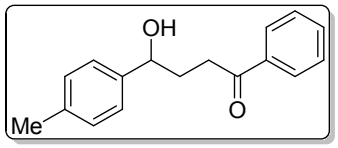
4-ethoxy-1,4-diphenylbutan-1-one (3b), ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.52 (t, J = 7.2 Hz, 3H), 2.08-2.21 (m, 2H), 3.00-3.13 (m, 2H), 3.26-3.34 (m, 1H), 3.36-3.43 (m, 1H), 4.35 (t, J = 7.2 Hz, 1H), 7.24-7.28 (m, 1H), 7.33-7.36 (m, 4H), 7.43 (t, J = 7.6 Hz, 2H), 7.53 (t, J = 7.6 Hz, 1H), 7.94 (d, J = 7.6 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.0, 142.6, 137.0, 132.8, 128.5, 128.3, 128.0, 127.4, 126.4, 80.8, 64.1, 34.6, 32.6, 15.3.



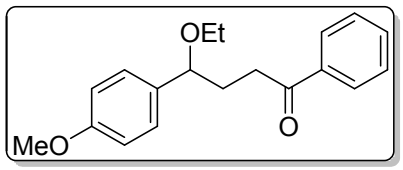
4-oxo-1,4-diphenylbutyl acetate (3c), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.05 (s, 3H), 2.21-2.40 (m, 2H), 2.91-3.05 (m, 2H), 5.86 (dd, J_1= 7.6 Hz, J_2= 6.0 Hz, 1H), 7.28-7.31 (m, 1H), 7.32-7.37 (m, 4H), 7.41-7.45 (m, 2H), 7.52-7.55 (m, 1H), 7.88-7.91 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 198.8, 170.2, 140.1, 136.7, 133.0, 128.5, 128.5, 128.0, 127.9, 126.3, 75.2, 34.3, 30.5, 21.1; IR (thin film, cm^{-1}) 2952.9, 2930.6, 1970.5, 1730.3, 1679.1, 1597.4, 1449.3, 1428.6, 1374.5, 1244.1, 1035.2, 946.1, 889.1, 767.8, 739.5, 709.1, 688.8, 546.2; HRMS (ESI) m/z calcd for Chemical Formula: $C_{18}H_{18}O_3$ $[M+Na]^+$: 305.1148, found: 305.1146.



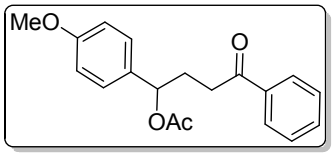
4-hydroxy-1,4-diphenylbutan-1-one (2a), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.16-2.22 (m, 2H), 2.54-2.59 (s, 1H), 3.10 (t, J = 7.2 Hz, 2H), 4.82 (dd, J_1= 7.2 Hz, J_1= 6.8 Hz, 1H), 7.26 (d, J = 6.8 Hz, 1H), 7.32-7.39 (m, 4H), 7.42 (m, 2H), 7.54 (d, J = 7.2 Hz, 1H), 7.93 (d, J = 7.2 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.5, 144.3, 136.8, 133.1, 128.5, 128.5, 128.5, 128.5, 128.1, 127.5, 125.7, 73.6, 34.8, 33.1; IR (thin film, cm^{-1}) 3413.6, 3027.4, 2948.8, 1966.7, 1667.5, **1**448.1, 1370.2, 1323.7, 1271.8, 1005.1, 1015.2, 735.8, 700.2, 687.4, 547.9 cm^{-1}; HRMS (ESI) m/z calcd for $C_{16}H_{16}O_2$ $[M+Na]^+$: 263.1043, found: 263.1040.



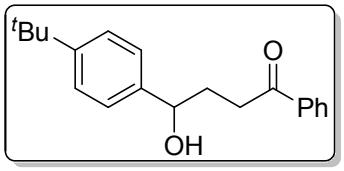
4-hydroxy-1-phenyl-4-(p-tolyl)butan-1-one (2b),^{1}H NMR (400 MHz, $CDCl_3$) δ 2.14-2.12 (m, 2H), 2.33 (s, 3H), 2.52 (s, 1H), 3.08 (t, J = 6.8 Hz, 2H), 4.77 (t, J = 6.4 Hz, 1H), 7.14 (d, J = 8.0 Hz, 2H), 7.25 (d, J = 8.0 Hz, 2H), 7.43 (t, J = 7.6 Hz, 2H), 7.52-7.54 (m, 1H), 7.92 (d, J = 1.6 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.6, 141.4, 137.2, 136.9, 133.1, 129.2, 128.6, 128.1, 125.8, 73.5, 34.8, 33.1, 21.1; IR (thin film, cm^{-1}) 3407.8, 3056.6, 2929.3, 1904.8, 1681.2, 1597.3, 1514.2, 1448.7, 1374.0, 1264.7, 1029.4, 818.5, 749.6, 690.1, 545.9 cm^{-1}; HRMS (ESI) m/z calcd for $C_{17}H_{18}O_2$ $[M+Na]^+$: 277.1199, found: 277.1201.



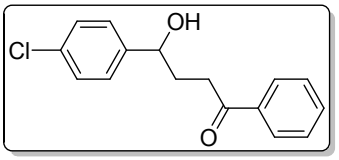
4-ethoxy-4-(4-methoxyphenyl)-1-phenylbutan-1-one (2c`), ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.14 (t, J = 7.2 Hz, 3H), 2.07-2.20 (m, 2H), 3.04 (t, J = 7.2 Hz, 2H), 3.26-3.39 (m, 2H), 3.80 (s, 3H), 4.28-4.31 (m, 1H), 6.80 (t, J = 6.8 Hz, 2H), 7.24 (d, J = 8.4 Hz, 2H), 7.44 (t, J = 7.6 Hz, 2H), 7.53 (d, J = 7.6 Hz, 1H), 7.92-7.94 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.1, 159.0, 137.1, 134.6, 132.8, 128.5, 128.0, 127.6, 113.8, 80.4, 63.9, 55.2, 34.7, 32.6, 15.3; IR (thin film, cm^{-1}) 3430.4, 2981.7, 2955.4, 1680.9, 1612.4, 1512.3, 1449.7, 1266.1, 1174.6, 1085.7, 1033.6, 837.5, 816.8, 733.8, 683.1, 563.6 cm^{-1}; HRMS (ESI) m/z calcd for $C_{19}H_{22}O_3$ $[M+Na]^+$: 321.1461, found: 321.1467.



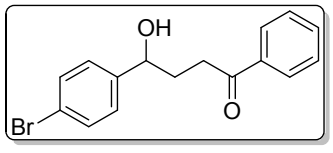
1-(4-methoxyphenyl)-4-oxo-4-phenylbutyl acetate (2c``), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.03 (s, 3H), 2.20-2.40 (m, 2H), 2.88-3.03 (m, 2H), 3.79 (s, 3H), 5.81 (t, J = 6.8 Hz , 1H), 6.88 (d, J = 8.4 Hz, 2H), 7.30 (d, J = 8.4 Hz, 2H), 7.42-7.46 (m, 2H), 7.54 (t, J = 7.6 Hz, 1H), 7.89 (d, J = 1.6 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 199.0, 170.3, 159.4, 136.8, 133.1, 132.2, 128.6, 128.0, 128.0, 114.0, 75.1, 55.3, 34.6, 30.5, 21.2; IR (thin film, cm^{-1}) 2957.9, 2930.3, 1733.5, 1684.5, 1611.8, 1514.3, 1448.6, 1371.2, 1238.7, 1176.8, 1029.1, 831.7, 752.4, 691.2, 551.7; HRMS (ESI) m/z calcd for $C_{19}H_{20}O_4$ $[M+Na]^+$: 335.1254, found: 335.1252.



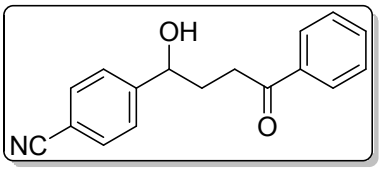
4-(4-(tert-butyl)phenyl)-4-hydroxy-1-phenylbutan-1-one (2d), ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.31 (s, 9H), 2.17-2.22 (m, 2H), 2.45 (s, 1H), 3.11 (t, J = 7.2 Hz, 2H), 4.77-4,81 (m, 1H), 7.30 (d, J = 8.4 Hz, 2H), 7.37 (d, J = 8.4 Hz, 4H), 7.44 (t, J = 9.6 Hz, 2H), 7.52-7.56 (m, 1H), 7.93-7.95 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.5, 150.5, 141.3, 136.9, 133.0, 128.5, 128.1, 125.5, 125.4, 73.4, 34.8, 34.5, 33.0, 31.3; IR (thin film, cm^{-1}) 3433.0, 3057.7, 2961.0, 1685.0, 1597.7, 1448.8, 1267.4, 1180.9, 1070.0, 1016.1, 833.9, 740.9, 690.4, 576.9; HRMS (ESI) m/z calcd for $C_{20}H_{24}O_2$ $[M+Na]^+$: 319.1669, found: 319.1667.



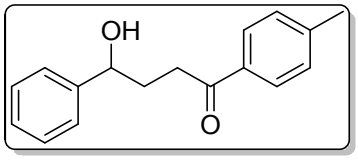
4-(4-chlorophenyl)-4-hydroxy-1-phenylbutan-1-one (2e), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.11-2.18 (m, 2H), 2.89 (s, 1H), 3.06-3.11 (m, 2H), 4.77-4.81 (m, 1H), 7.29 （s, 4H）,7.44 (t, J = 7.6 Hz, 2H), 7.55 (t, J = 7.6 Hz, 1H), 7.91-7.93 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.5, 142.4, 136.7, 133.2, 133.1, 128.6, 128.5, 128.0, 127.1, 72.8, 34.6, 33.0; IR (thin film, cm^{-1}) 3492.9, 2922.1, 1909.7, 1681.5, 1593.1, 1489.7, 1446.7, 1408.1, 1363.5, 1213.2, 1075.0, 829.5, 755.4, 687.6, 536.7 cm^{-1}; HRMS (ESI) m/z calcd for $C_{16}H_{15}ClO_2$ $[M+Na]^+$: 297.0663, found: 297.0650.



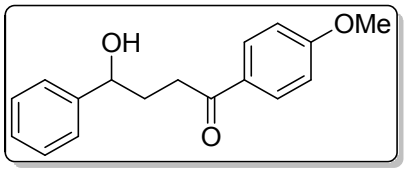
4-(4-bromophenyl)-4-hydroxy-1-phenylbutan-1-one (2f), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.10-2.22 (m, 2H), 2.84 (s, 1H), 3.03-3.16 (m, 2H), 4.77-4.80 (m, 1H), 7.24 (d, J = 8.4 Hz, 2H), 7.45 (d, J = 8.0 Hz, 3H), 7.56 (t, J = 7.2 Hz, 2H), 7.93 (d, J = 7.2 Hz, 2H); **^{13}C** NMR (100 MHz, $CDCl_3$) δ 200.6, 143.5, 136.7, 133.3, 131.6, 128.6, 128.1, 127.5, 121.3 72.9, 34.6, 33.0; IR (thin film, cm^{-1}) 3448.1, 3057.8, 2910.0, 1681.9, 1592.9, 1483.9, 1447.2, 1403.7, 1362.8, 1210.1, 1071.3, 824.0, 754.1, 686.5, 539.2 cm^{-1}; HRMS (ESI) m/z calcd for $C_{16}H_{15}BrO_2$ $[M+Na]^+$: 341.0148, 343.0127, found: 341.0144, 343.0123.



4-(1-hydroxy-4-oxo-4-phenylbutyl)benzonitrile (2g), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.08-2.25 (m, 2H), 3.08-3.20 (m, 2H), 4.88-4.91 (m, 1H), 7.44-7.50 (m, 4H), 7.55-7.63 (m, 3H), 7.92 (d, J = 1.2 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.6, 150.0, 136.6, 133.4, 132.3, 128.7, 128.1, 126.4, 118.8, 111.1, 72.7, 34.5, 33.0; IR (thin film, cm^{-1}) 3479.6, 3083.8, 2879.4, 2225.3, 1978.0, 1670.5, 1595.9, 1446.6, 1405.8, 1370.1, 1212.7, 749.7, 736.9, 689.5, 565.3 cm^{-1}; HRMS (ESI) m/z calcd for $C_{17}H_{15}NO_2$ $[M+Na]^+$: 288.0995, found: 288.0992.

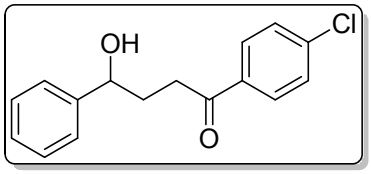


4-hydroxy-4-phenyl-1-(p-tolyl)butan-1-one (2h), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.14-2.20 (m, 2H), 2.39 (s, 3H), 2.73 (s, 1H), 3.06 (t, J = 6.8 Hz, 2H), 4.80 (t, J = 6.4 Hz, 1H), 7.22-7.27 (m, 3H), 7.31-7.37 (m, 4H), 7.83 (d, J = 8.4 Hz, 2H) **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.3, 144.4, 143.9, 134.3, 129.2, 128.4, 128.2, 127.5, 125.7, 73.6, 34.6, 33.1, 21.6; IR (thin film, cm^{-1}) 3445.4, 3032.6, 2923.7, 2365.9, 1719.3, 1680.7, 1608.0, 1451.2, 1408.6, 1269.9, 1179.4, 737.4, 701.1, 544.9 cm^{-1}; HRMS (ESI) m/z calcd for $C_{17}H_{18}O_2$ $[M+Na]^+$: 277.1199, found: 277.1196.

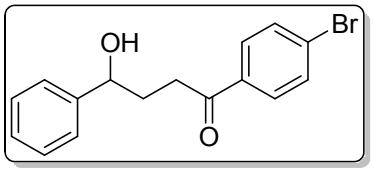


4-hydroxy-1-(4-methoxyphenyl)-4-phenylbutan-1-one (2i), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.14-2.20 (m, 2H), 2.87 (s, 1H), 3.04 (t, J = 6.8 Hz, 2H), 3.84 (s, 3H), 4.79-4.82 (m, 1H), 6.90 (d, J = 8.4 Hz, 2H), 7.24-7.27 (m, 1H), 7.31-7.38 (m, 4H), 7.89-7.92 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 199.2, 163.5, 144.4, 130.4, 129.9, 128.4, 127.4, 125.7, 113.7, 73.6, 55.4, 34.4, 33.2; IR

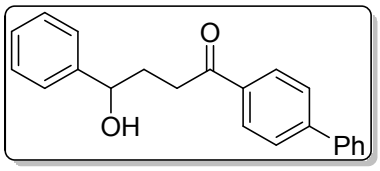
(thin film, cm^{-1}) 3425.8, 3383.2, 2956.4, 1916.4, 1674.1, 1602.6, 1509.2, 1455.4, 1416.7, 1332.9, 1263.5, 812.7, 753.9, 701.3, 542.8 cm^{-1}; HRMS (ESI) m/z calcd for $C_{17}H_{18}O_3$ $[M+Na]^+$: 293.1148, found: 293.1150.



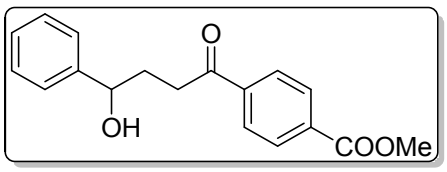
1-(4-chlorophenyl)-4-hydroxy-4-phenylbutan-1-one (2j), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.15-2.21 (m, 2H), 2.41 (s, 1H), 3.06 (t, J = 7.2 Hz, 2H), 4.81 (dd, J_1 =7.2 Hz, J_2 = 7.2 Hz, 1H), 7.25-7.29 (m, 1H), 7.32-7.37 (m, 4H), 7.39-7.42 (m, 2H), 7.85-7.88 (m, 2H); 199.2, 144.2, 139.5, 135.2, 129.5, 128.9, 128.5, 127.6, 125.7, 125.7, 73.5, 34.7, 33.0; IR (thin film, cm^{-1}) 3432.8, 3064.2, 2926.7, 1722.5, 1683.9, 1592.5, 1489.5, 1400.7, 1270.2, 1092.6, 1014.2, 759.8, 699.8, 526.2 cm^{-1}; HRMS (ESI) m/z calcd for $C_{16}H_{15}ClO_2$ [M+Na]+: 297.0653, found: 297.0649.



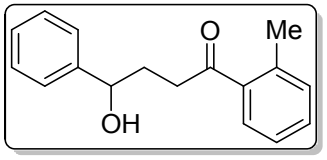
1-(4-bromophenyl)-4-hydroxy-4-phenylbutan-1-one (2k), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.13-2.23 (m, 2H), 2.46-2.50 (m, 1H), 3.06 (t, J = 7.2 Hz, 2H), 4.79-4.82 (m, 1H), 7.32-7.36 (d, 5H), 7.57 (d, J = 8.8 Hz, 2H), 7.90 (d, J = 8.4 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 199.4, 144.2, 135.5, 131.9, 129.6, 128.5, 128.2, 127.6, 125.7, 73.5, 34.6, 32.9; IR (thin film, cm^{-1}) 3355.1, 3027.2, 2922.7, 1950.8, 1684.1, 1584.6, 1491.9, 1484.3, 1451.6, 1396.1, 1071.2, 796.1, 759.6, 746.6, 549.3 cm^{-1}; HRMS (ESI) m/z calcd for $C_{16}H_{15}BrO_2$ $[M+Na]^+$: 341.0148, 343.0127, found: 341.0151, 343.0129.



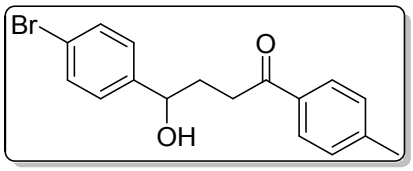
1-([1,1'-biphenyl]-4-yl)-4-hydroxy-4-phenylbutan-1-one (2l), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.15-2.28 (m, 2H), 2.62 (s, 1H), 3.13 (t, J = 7.2 Hz, 2H), 4.83 (t, J = 6.0 Hz, 2H), 7.25-7.29 (m, 1H), 7.33-7.40 (m, 5H), 7.40-7.47 (m, 2H), 7.60-7.66 (m, 4H), 8.00 (d, J = 8.4 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.1, 145.7, 144.4, 139.8, 135.5, 128.9, 128.7, 128.5, 128.2, 127.5, 127.2, 125.7, 73.6, 34.8, 33.1; IR (thin film, cm^{-1}) 3333.5, 3027.4, 2929.9, 1949.7, 1675.1, 1604.8, 1453.9, 1446.2, 1401.4, 1371.7, 1006.8, 816.5, 734.7, 699.1, 551.8 cm^{-1};HRMS (ESI) m/z calcd for $C_{22}H_{20}O_2$ [M+Na]+:339.1356, found: 339.1352.



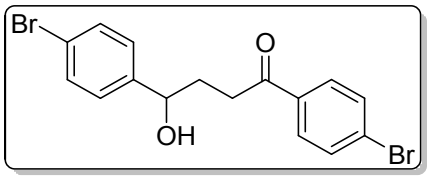
Methyl 4-(4-hydroxy-4-phenylbutanoyl)benzoate (2m), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.15-2.24 (m, 2H), 2.52 (s, 1H), 3.12 (d, J = 6.8 Hz, 2H), 3.93 (s, 3H), 4.82 (t, J = 6.4 Hz, 1H), 7.27-7.29 (m, 2H), 7.32-7.39 (m, 4H), 7.96 (d, J = 8.4 Hz, 2H), 8.09 (d, J = 8.0 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 204.6, 144.3, 138.0, 137.9, 131.9, 131.3, 128.5, 128.4 127.6, 125.7, 125.6, 73.6, 37.7, 33.2, 21.3; IR (thin film, cm^{-1}) 3517.5, 2924.7, 2859.2, 1959.7, 1705.6, 1688.6, 1571.3, 1454.3, 1437.6, 1406.5, 1288.4, 865.0,765.1, 752.8, 548.8 cm^{-1}; HRMS (ESI) m/z calcd for $C_{18}H_{18}O_4$ $[M+Na]^+$:321.1097, found: 321.1094.



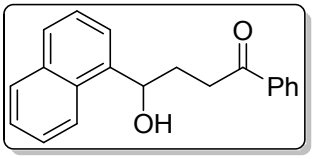
4-hydroxy-4-phenyl-1-(o-tolyl)butan-1-one (2n), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.15-2.20 (m, 2H), 2,45 (s, 1H), 2,48(s, 3H), 3.02 (t, J = 7.2 Hz, 2H), 4.82 (t, J = 6.4 Hz, 1H),7.21-7.24 (m, 2H), 7.27-7.29 (m, 1H), 7.33-7.38 (m, 5H), 7.59-7.61 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.6, 150.0, 136.6, 133.4, 132.3, 128.7, 128.1, 126.4, 118.8, 111.1, 72.7, 34.5, 33.0; IR (thin film, cm^{-1}) 3433.2, 3027.7, 2960.1, 2926.9, 1682.9, 1600.7, 1453.8, 1287.4, 1257.1, 1056.5, 920.9, 755.2, 701.5, 654.9, 546.7 cm^{-1}; HRMS (ESI) m/z calcd for $C_{17}H_{18}O_2$ $[M+Na]^+$: 277.1199, found: 277.1201.



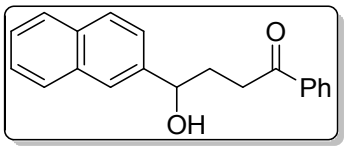
4-(4-bromophenyl)-4-hydroxy-1-(p-tolyl)butan-1-one (2p), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.07-2.21 (m, 2H), 2.40 (s, 3H), 2.89 (s, 1H), 3.05-3.09 (m, 2H), 4.77-4.80 (m, 1H), 7.40 (d, J = 8.4 Hz, 4H), 7.45 (d, J = 8.4 Hz, 2H), 7.83 (d, J = 8.0 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.3, 144.1, 143.5, 134.2, 131.5, 129.3, 128.2, 127.5, 121.2, 72.9, 34.5, 33.0, 21.6; IR (thin film, cm^{-1}) 3313.6, 2917.0, 1680.5,1606.5, 1487.3, 1402.9, 1240.2, 1183.3, 1068.3, 1055.9, 1008.0, 826.7, 814.6, 782.2, 535.7 cm^{-1}; HRMS (ESI) m/z calcd for $C_{17}H_{17}BrO_2$ $[M+Na]^+$: 355.0304, 357.0284, found: 355.0308, 357.0287.



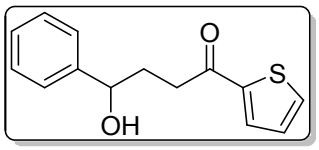
1,4-bis(4-bromophenyl)-4-hydroxybutan-1-one (2q), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.07-2.22 (m, 2H), 2.58 (d, J = 2.4 Hz, 1H), 3.03-3.08 (m, 2H), 4.77-4.80 (m, 1H), 7.24 (d, J = 8.4 Hz, 2H), 7.46 (d, J = 8.4 Hz, 2H), 7.59 (t, J = 8.4 Hz, 2H), 7.79 (d, J = 7.2 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 199.3, 143.3, 135.4, 131.9, 131.6, 129.6, 128.4, 127.4, 121.3, 72.8, 34.5, 32.8; IR (thin film, cm^{-1}) 3472.1, 2924.9, 1907.3, 1685.4, 1583.2, 1483.9, 1398.3, 1366.9, 1210.7, 1070.5, 1008.2, 828.4, 795.6, 755.9, 534.6 cm^{-1}; HRMS (ESI) m/z calcd for $C_{16}H_{14}Br_2O_2$ $[M+Na]^+$: 418.9253, 420.9232, 422.9212, found: 418.9251, 420.9231, 422.9210.



4-hydroxy-4-(naphthalen-1-yl)-1-phenylbutan-1-one (2r), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.14-2.23 (m, 1H), 2.39-2.46 (m, 1H), 2.79 (s, 1H), 3.05-3.28 (m, 2H), 5.56-5.58 (m, 1H), 7.38-7.52 (m, 6H), 7.66 (d, J = 7.2 Hz, 1H), 7.74 (d, J = 8.4 Hz, 1H), 7.84 (d, J = 7.6 Hz, 1H), 7.92 (d, J = 7.6 Hz, 2H), 8.15 (d, J = 8.0 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.8, 140.2, 136.8, 133.7, 133.1, 130.2, 128.8, 128.5, 128.0, 127.9, 126.1, 125.5, 125.4, 123.2, 122.6, 70.0, 34.8, 32.1; IR (thin film, cm^{-1}) 3471.5, 3057.5, 2923.4, 1953.5, 1723.8, 1671.1, 1446.2, 1407.0, 1370.1, 1203.6, 1086.9, 776.6, 734.3, 681.8, 569.8 cm^{-1}; HRMS (ESI) m/z calcd for $C_{20}H_{18}O_2$ $[M+Na]^+$: 313.1199, found: 313.1195.



4-hydroxy-4-(naphthalen-2-yl)-1-phenylbutan-1-one (2s), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.18-2.30 (m, 2H), 2.90 (s, 1H), 3.06-3.09 (m, 2H), 4.92-4.95 (m, 1H), 7.37-7.52 (m, 6H), 7.78-7.80 (m, 4H), 7.88-7.90 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.8, 140.2, 136.8, 133.7, 133.1, 130.2, 128.8, 128.5, 128.0, 127.9, 126.1, 125.5, 125.4, 123.2, 122.6, 70.0, 34.8, 32.1; IR (thin film, cm^{-1}) 3449.8, 3054.9, 2917.1, 1673.3, 1596.0, 1447.8, 1368.8, 1327.1, 1266.7, 1203.9, 1071.8, 824.6, 741.6, 687.8, 486.2 cm^{-1}; HRMS (ESI) m/z calcd for $C_{20}H_{18}O_2$ $[M+Na]^+$: 313.1199, found:313.1194.



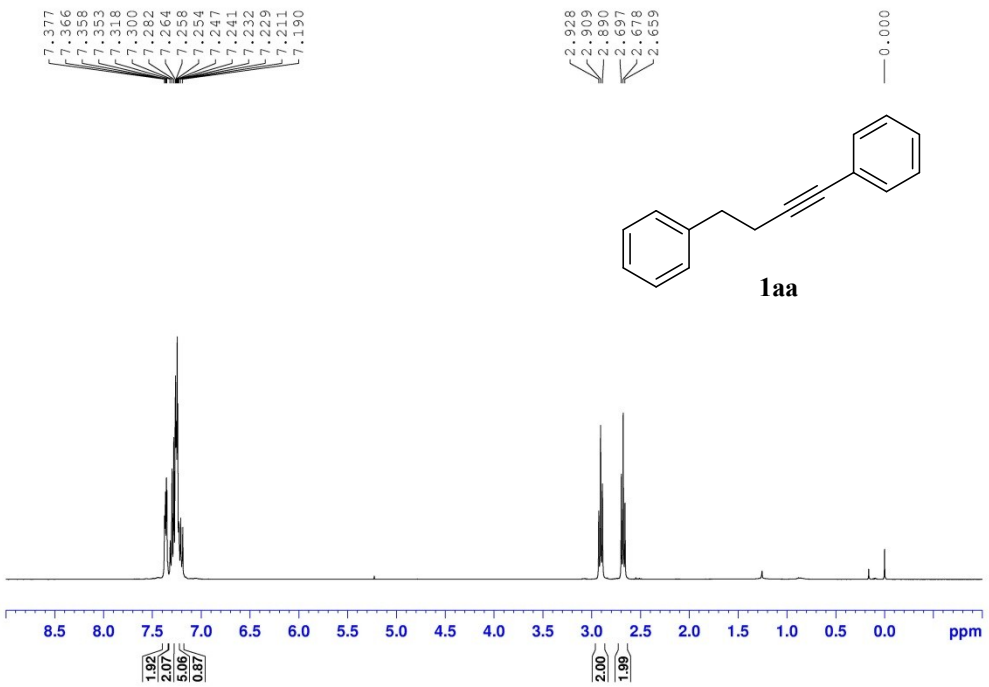
4-hydroxy-4-phenyl-1-(thiophen-2-yl)butan-1-one (2t), ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.14-2.21 (m, 2H), 2.70 (s, 1H), 3.02-3.05(m, 2H), 4.78-4.81 (m, 1H), 7.09-7.11 (m, 1H), 7.25-7.28 (m, 1H), 7.31-7.37 (m, 4H), 7.60-7.62 (m, 1H), 7.67-7.68 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.4, 144.2, 144.0, 133.6, 132.6, 128.4, 128.1, 127.5, 125.7, 73.4, 35.4, 33.3; IR (thin film, cm^{-1}) 3504.6, 3383.0, 3085.4, 2920.6, 1719.9, 1645.1, 1513.8, 1453.8, 1414.6, 1358.8, 1063.0, 770.0, 751.0, 703.1, 544.3 cm^{-1}; HRMS (ESI) m/z calcd for $C_{14}H_{14}O_2S$ $[M+Na]^+$: 269.0607, found: 269.0613.

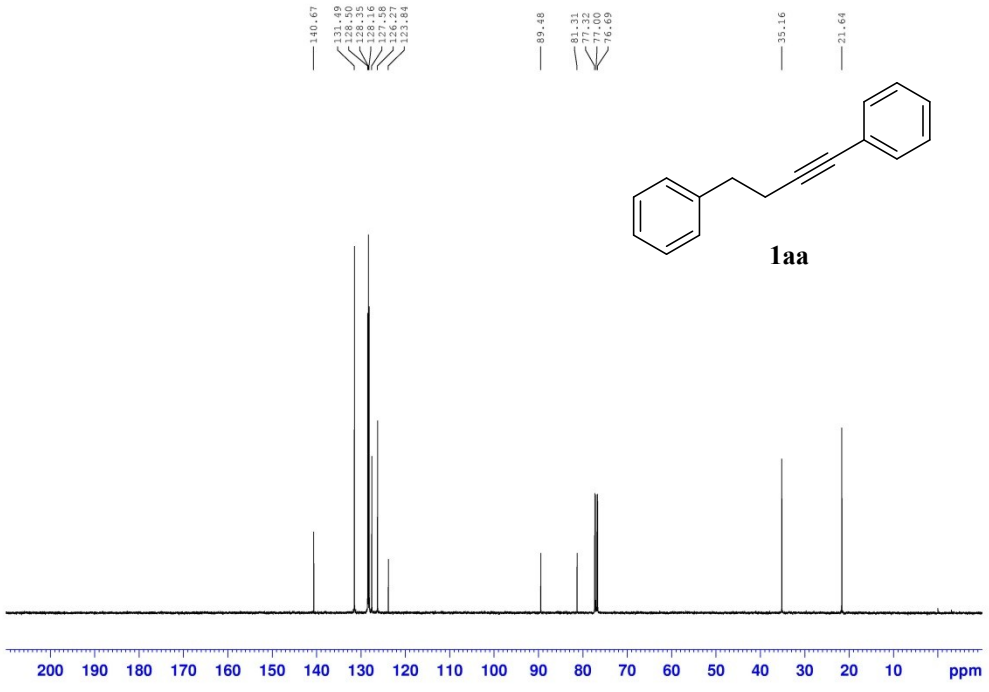
7. References

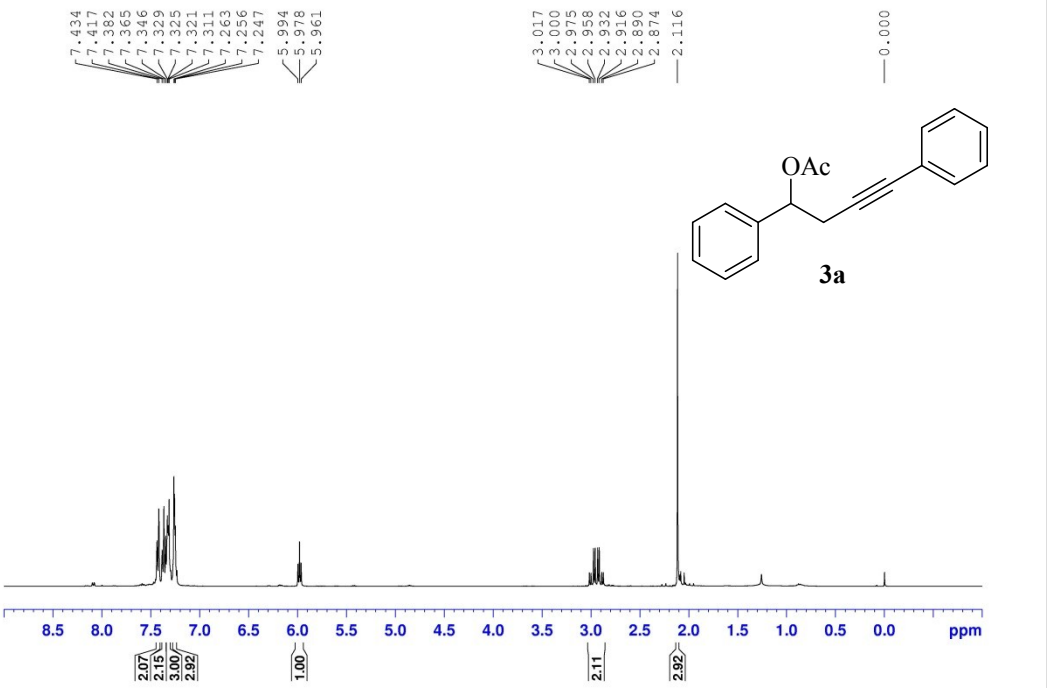
[1] a) D.-Q. Chen, P. Gao, P.-X. Zhou, X.-R. Song, Y.-F. Qiu, X.-Y. Liu and Y.-M. Liang, *Chem. Commun.*, 2015, **51**, 6637; b) P. Gao, Y.-W. Shen, R. Fang, X.-H. Hao, Z.-H. Qiu, F. Yang, X.-B. Yan, Q. Wang, X.-J. Gong, X.-Y. Liu and Y.-M. Liang, *Angew. Chem., Int. Ed.*, 2014, **53**, 7629.

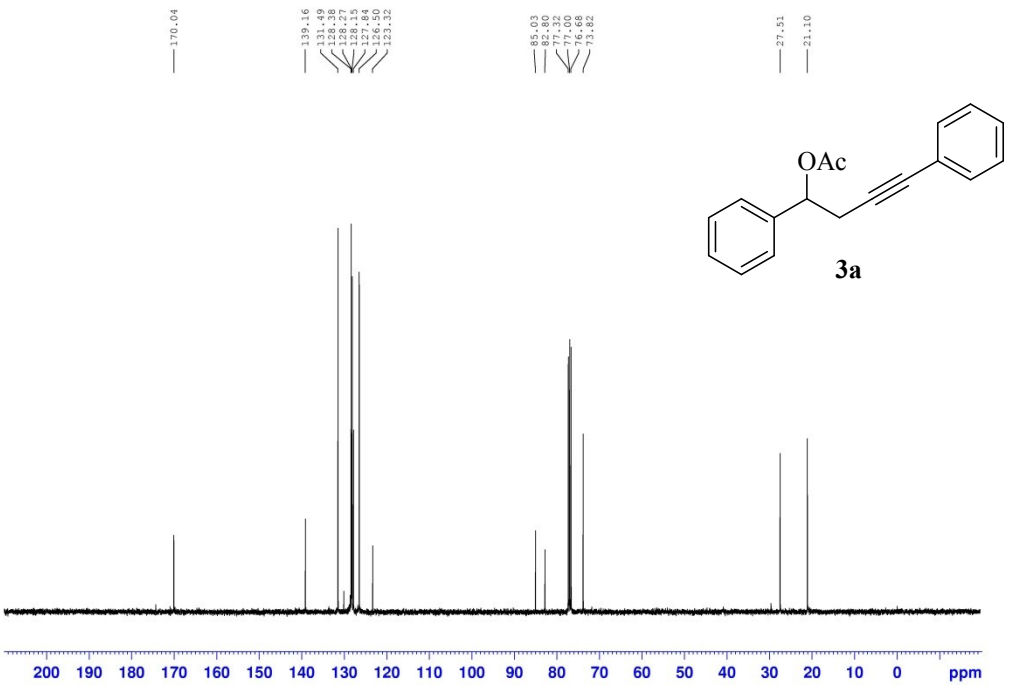
[2] E. Shirakawa, Y. Yasuhara and T. Hayashi, *Chem. Lett.*, 2006, **35**, 768.

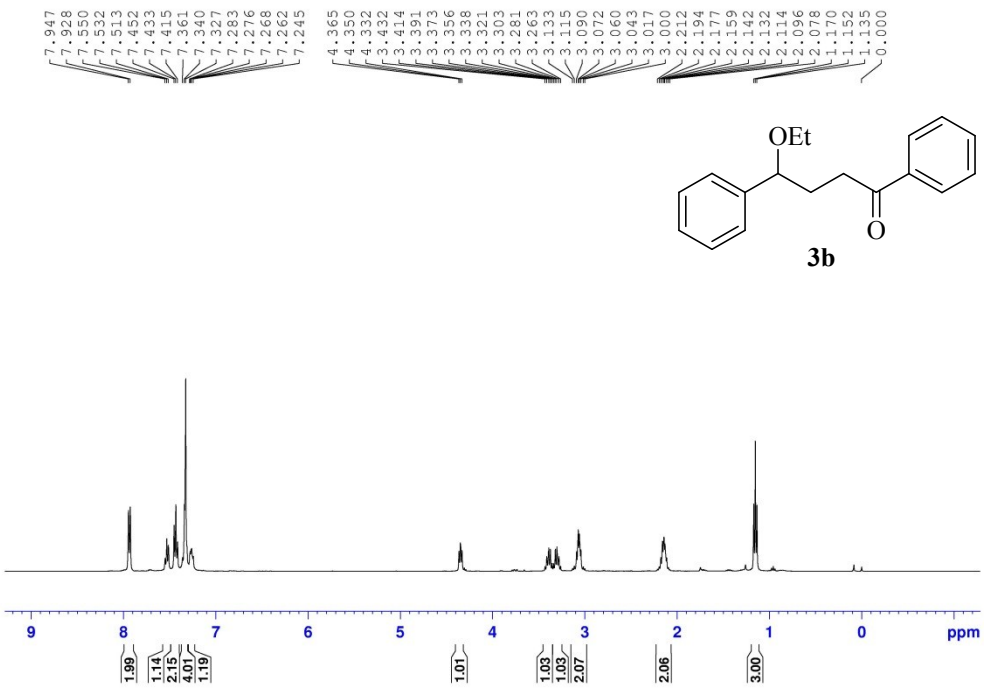
8. ^{1}H NMR and ^{13}C NMR spectra of 1aa, 3a-3c, 2a-2t

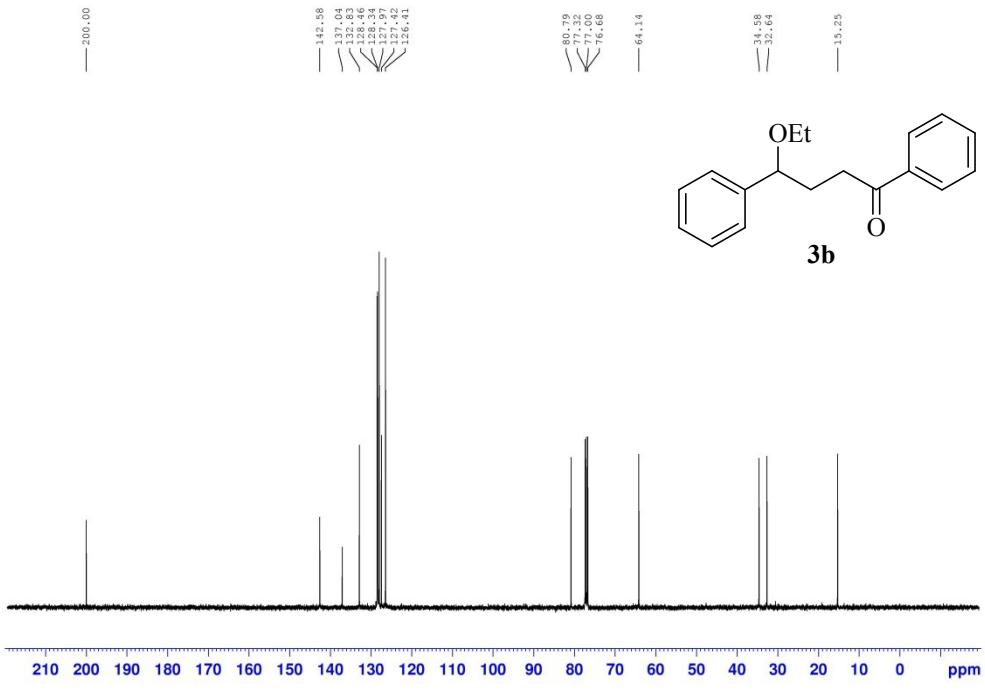


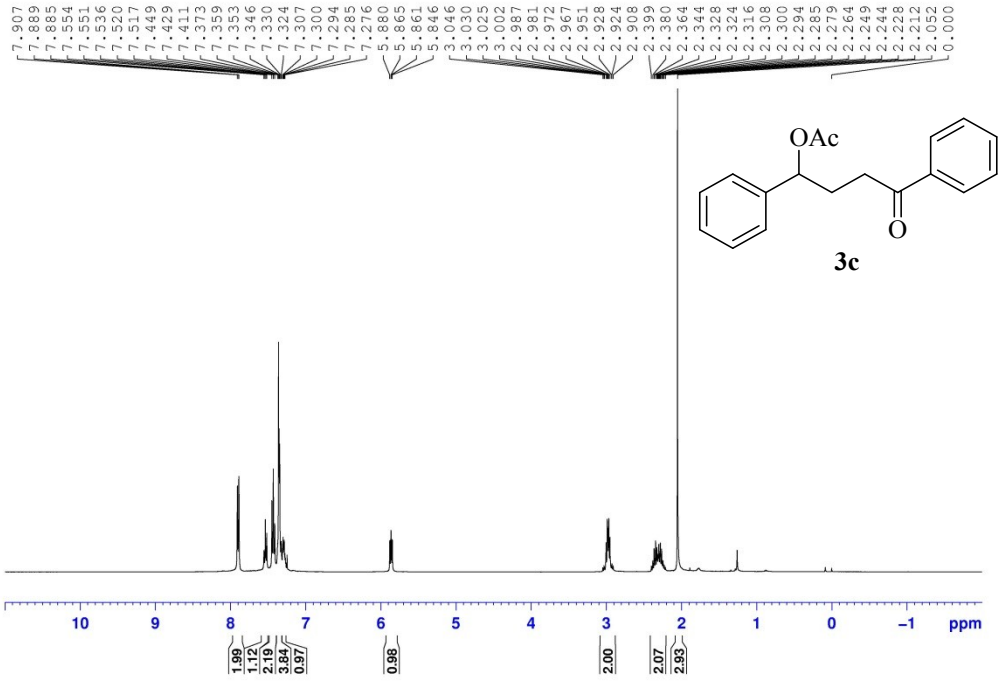


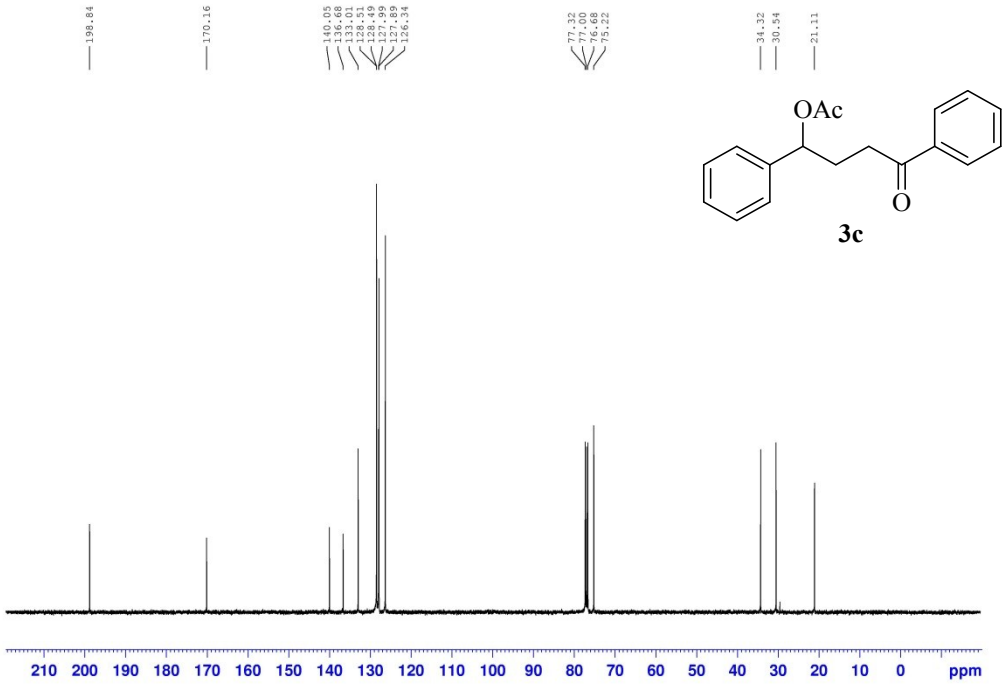

7.434
7.417
7.382
7.365
7.346
7.329
7.325
7.321
7.311
7.263
7.256
7.247
5.994
5.978
5.961
3.017
3.000
2.975
2.958
2.932
2.916
2.890
2.874
2.116
0.000
OAc
3a
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.07
2.15
3.00
2.92
1.00
2.11
2.92

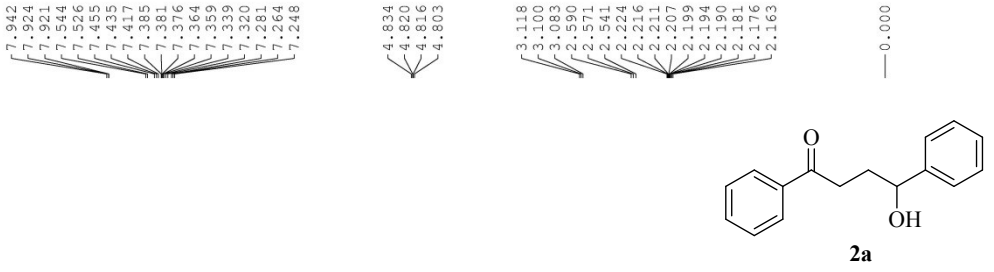

170.04
139.16
131.49
128.58
128.37
128.15
127.84
126.50
123.32
85.03
82.80
77.32
77.00
76.68
73.82
27.51
21.10
OAc
3a
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.947
7.928
7.550
7.532
7.513
7.452
7.433
7.415
7.361
7.340
7.327
7.283
7.276
7.268
7.262
7.245
4.365
4.350
4.332
3.432
3.414
3.391
3.373
3.356
3.338
3.321
3.303
3.281
3.263
3.133
3.115
3.090
3.072
3.060
3.043
3.017
3.000
2.212
2.194
2.177
2.159
2.142
2.132
2.114
2.096
2.078
1.170
1.152
1.135
0.000
OEt
O
3b
9
8
7
6
5
4
3
2
1
0
ppm
1.99
1.14
2.15
4.01
1.19
1.01
1.03
1.03
2.07
2.06
3.00

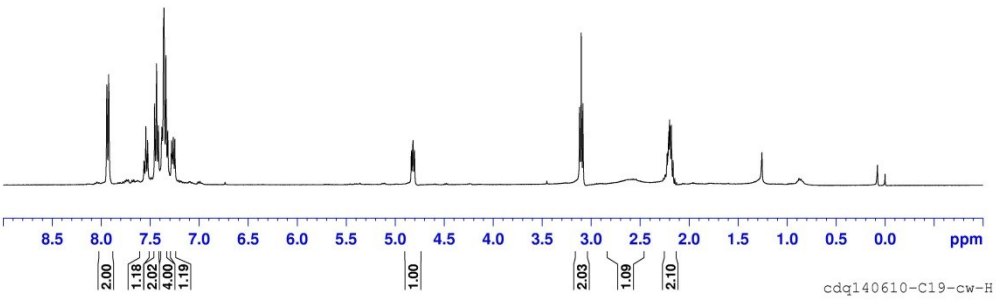

200.00
142.58
137.04
132.83
128.46
128.34
128.07
127.42
126.41
80.79
77.32
77.00
76.68
64.14
34.58
32.64
15.25
OEt
O
3b
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

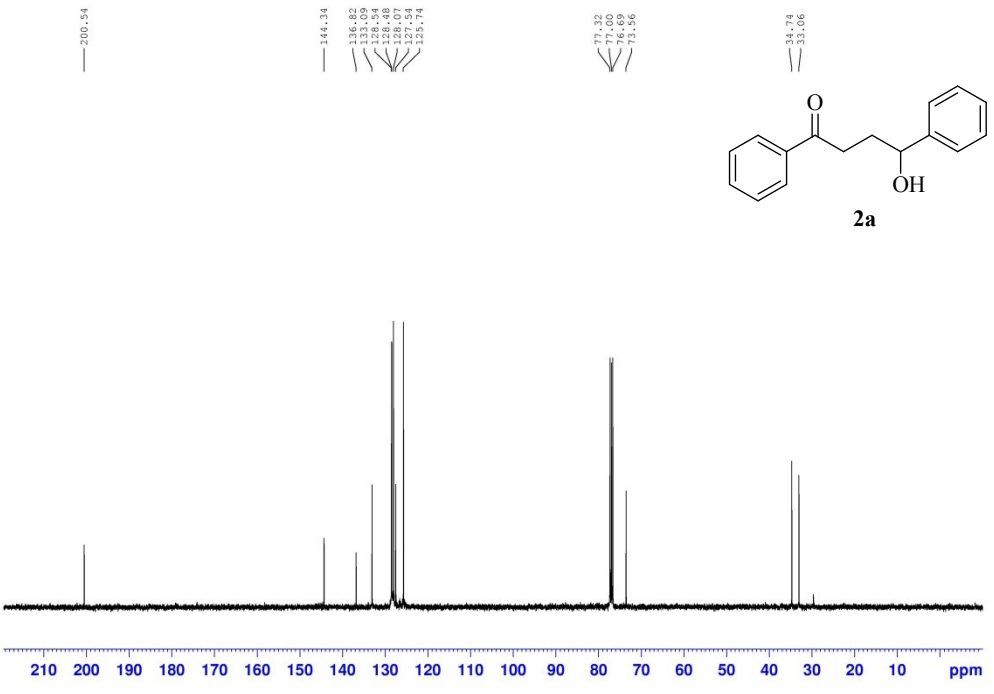

7.907
7.889
7.885
7.554
7.551
7.536
7.520
7.517
7.449
7.429
7.411
7.373
7.359
7.353
7.346
7.330
7.324
7.307
7.300
7.294
7.285
7.276
5.880
5.865
5.861
5.846
3.046
3.030
3.025
3.002
2.987
2.981
2.972
2.967
2.951
2.928
2.924
2.908
2.399
2.380
2.364
2.344
2.328
2.324
2.316
2.308
2.300
2.294
2.285
2.279
2.264
2.249
2.244
2.228
2.212
2.052
0.000
OAc
O
3c
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
1.99
1.12
2.19
3.84
0.97
0.98
2.00
2.07
2.93


198.84
170.16
140.05
136.68
133.01
128.31
128.09
127.99
127.89
126.34
77.32
77.00
76.68
75.22
34.32
30.54
21.11
OAc
O
3c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

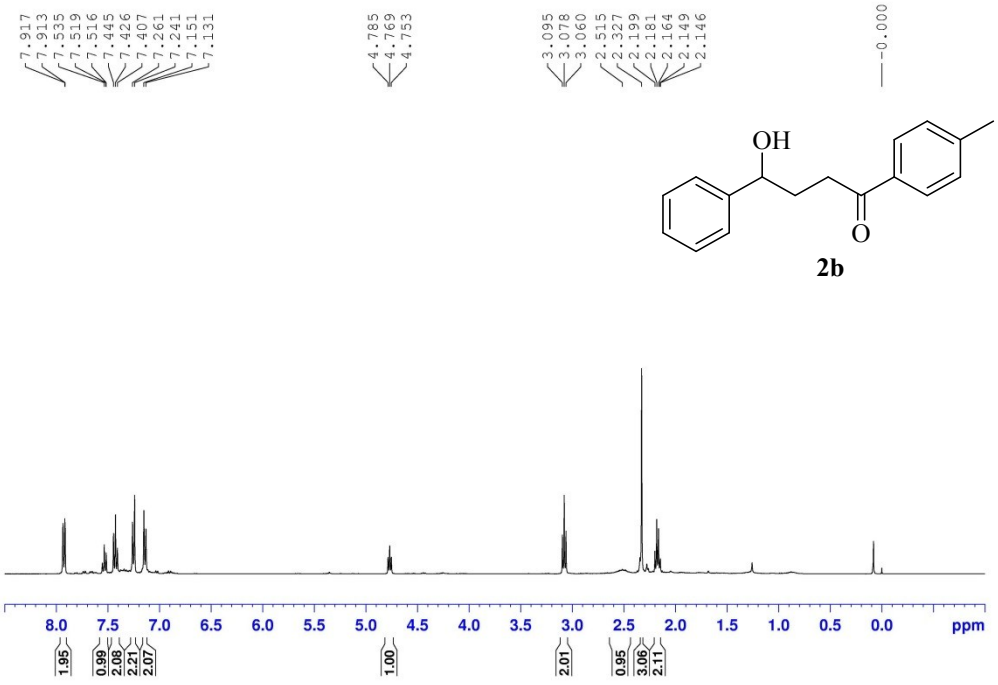

7.942
7.924
7.921
7.544
7.526
7.455
7.435
7.417
7.385
7.381
7.376
7.364
7.359
7.339
7.320
7.281
7.264
7.248
4.834
4.820
4.816
4.803
3.118
3.100
3.083
2.590
2.571
2.541
2.224
2.216
2.211
2.207
2.199
2.194
2.190
2.181
2.176
2.163
0.000
O
OH

2a

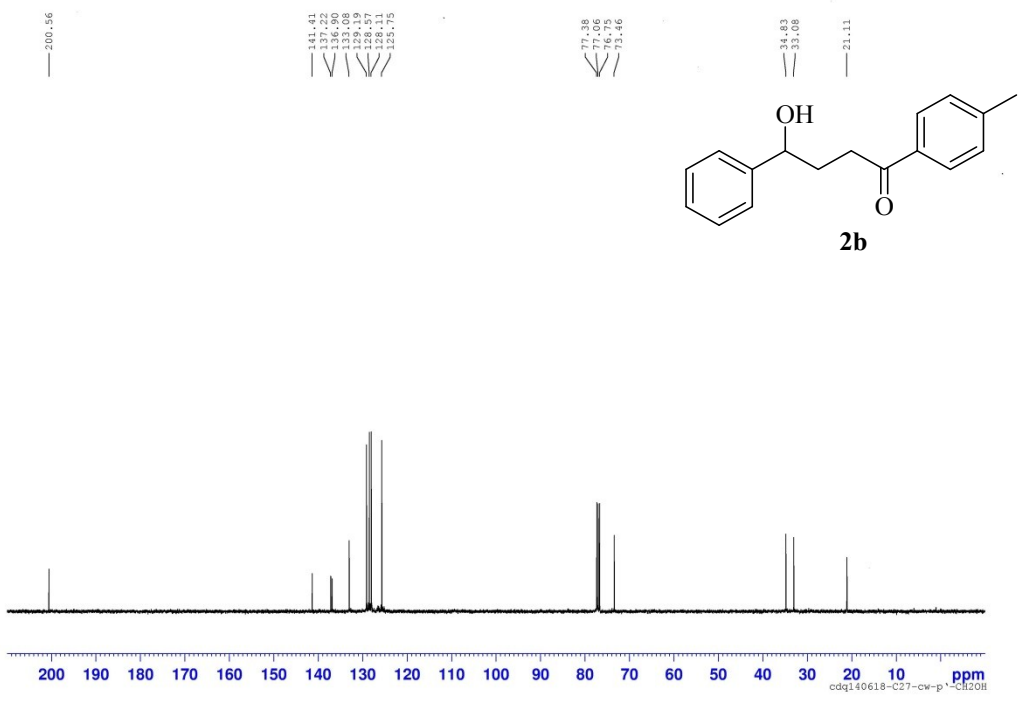

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
1.18
2.02
4.00
1.19
1.00
2.03
1.09
2.10
cdq140610-C19-cw-H


200.54
144.34
136.82
133.09
128.54
128.48
128.07
127.54
125.74
77.32
77.00
76.69
73.56
34.74
33.06
O
OH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

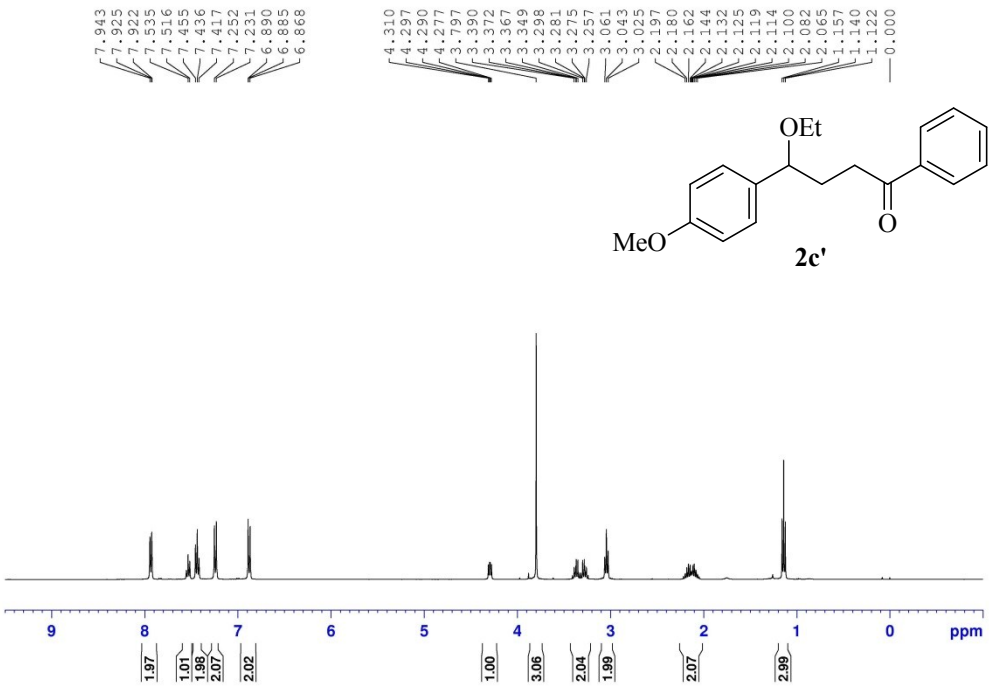
2a

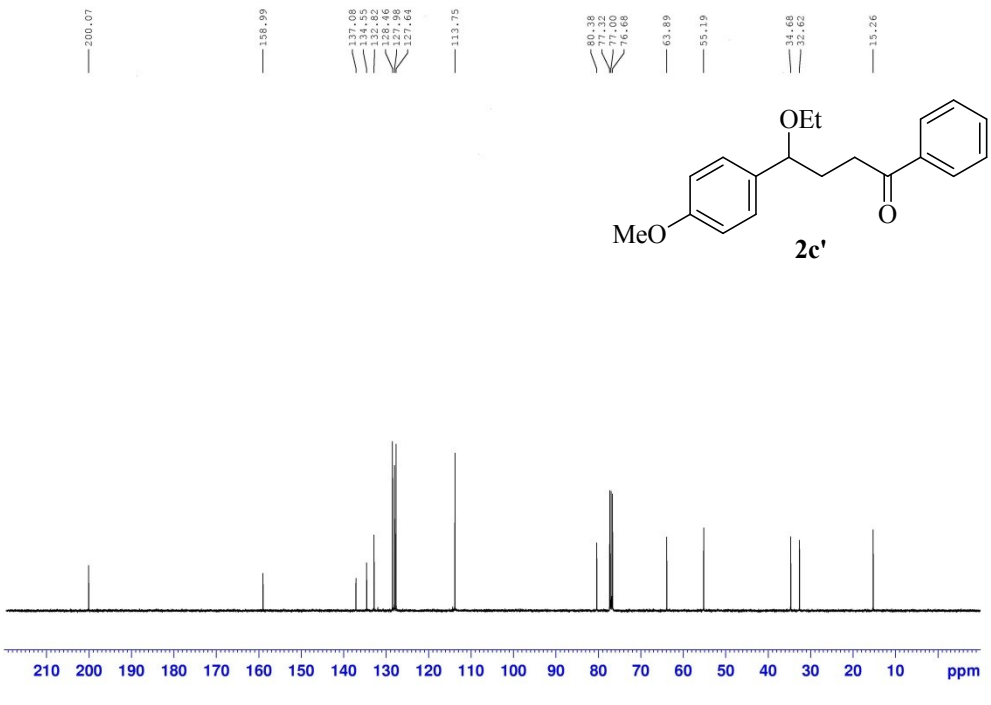

7.917
7.913
7.535
7.519
7.516
7.445
7.426
7.407
7.261
7.241
7.151
7.131
4.785
4.769
4.753
3.095
3.078
3.060
2.515
2.327
2.199
2.181
2.164
2.149
2.146
0.000
OH
O
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.95
0.99
2.08
2.21
2.07
1.00
2.01
0.95
3.06
2.11

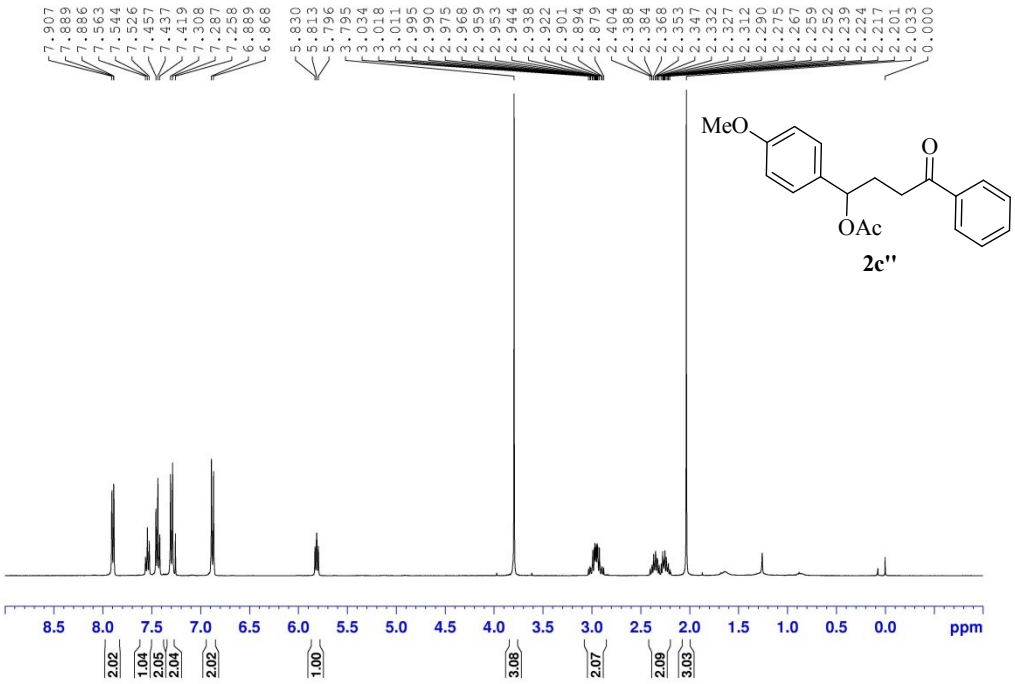
2b


200.56
141.41
137.22
136.90
133.08
129.19
128.57
128.11
125.75
77.38
77.06
76.75
73.46
34.83
33.08
21.11
OH
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
cdq140618-C27-cw-p1-CH2OH

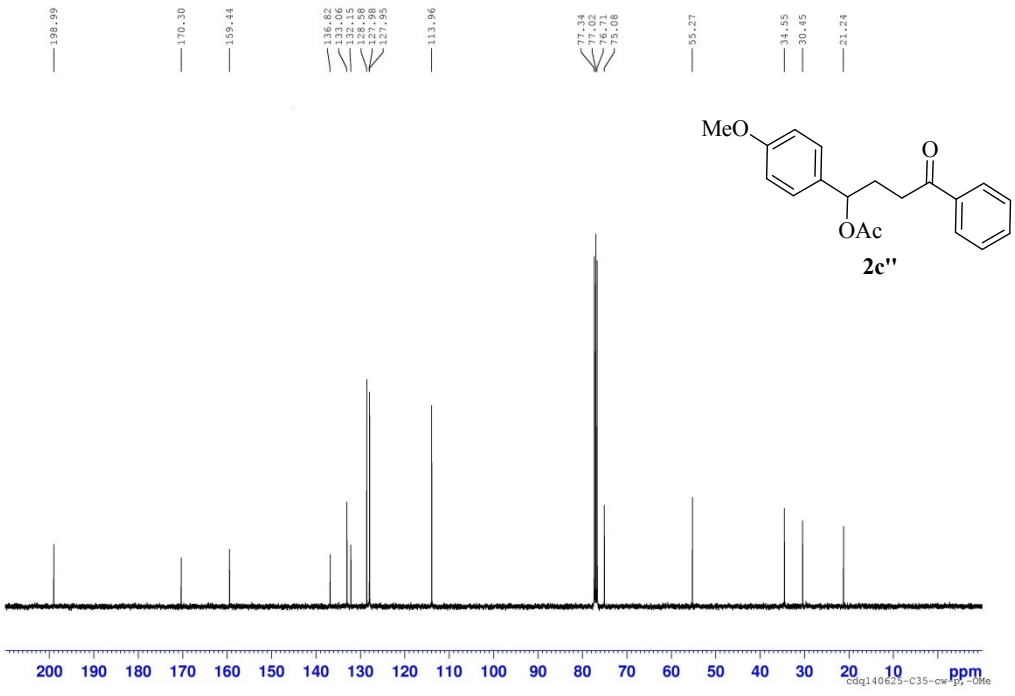
2b


7.943
7.925
7.922
7.535
7.516
7.455
7.436
7.417
7.252
7.231
6.890
6.885
6.868
4.310
4.297
4.290
4.277
3.797
3.390
3.372
3.367
3.349
3.298
3.281
3.275
3.257
3.061
3.043
3.025
2.197
2.180
2.162
2.144
2.132
2.125
2.119
2.114
2.100
2.082
2.065
1.157
1.140
1.122
0.000
OEt
O
MeO
2c'
9
8
7
6
5
4
3
2
1
0
ppm
1.97
1.01
1.98
2.07
2.02
1.00
3.06
2.04
1.99
2.07
2.99

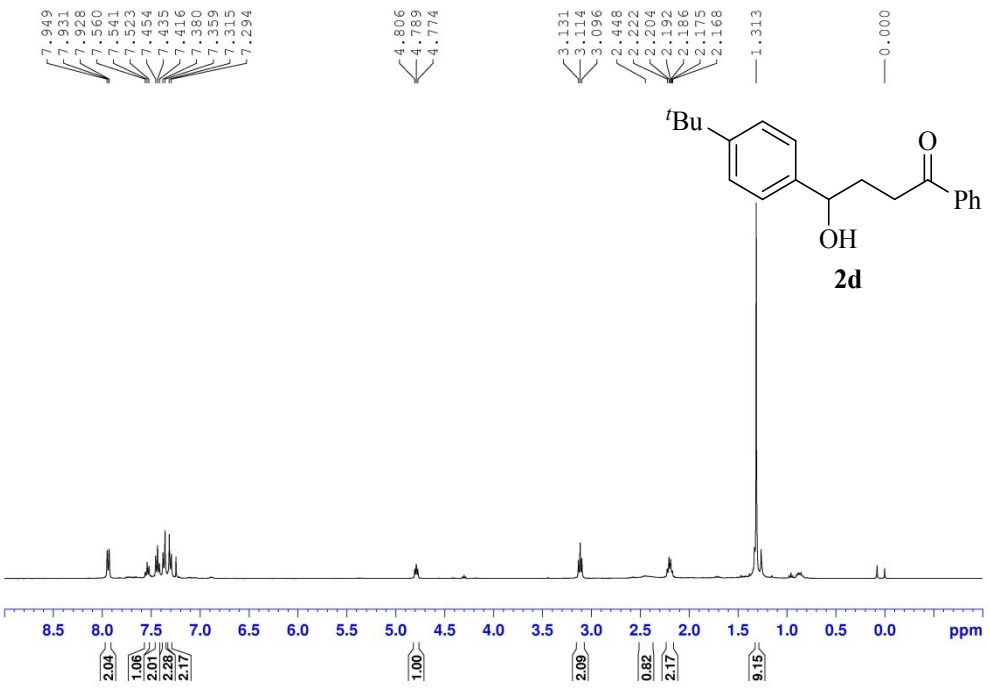

200.07
158.99
137.08
134.55
132.82
128.46
127.98
127.64
113.75
80.38
77.32
77.00
76.68
63.89
55.19
34.68
32.62
15.26
OEt
O
MeO
2c'
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

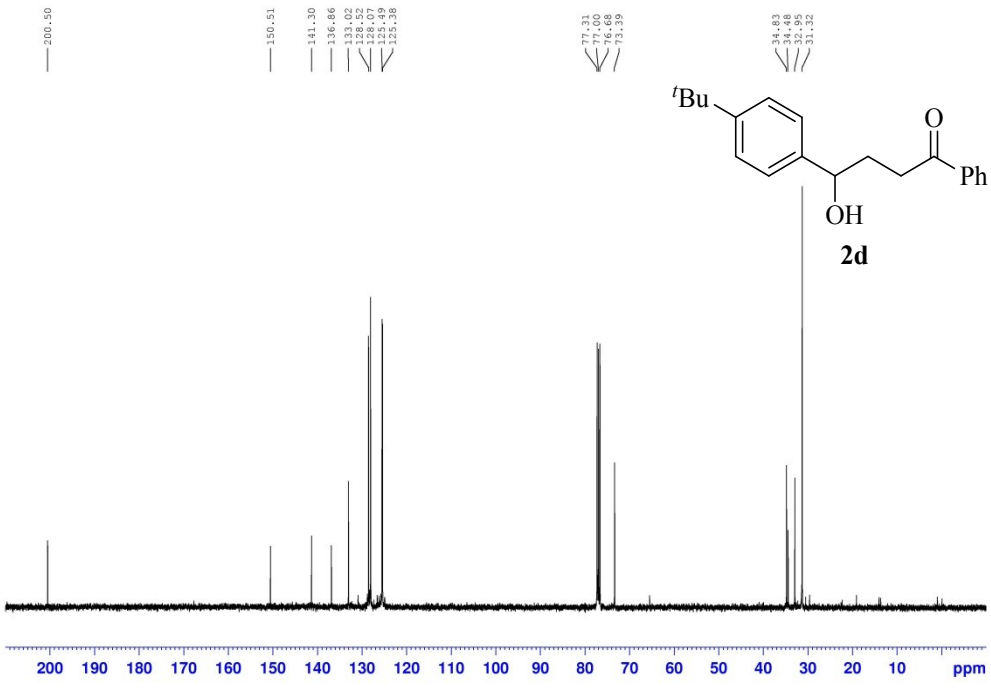

7.907
7.889
7.886
7.563
7.544
7.526
7.457
7.437
7.419
7.308
7.287
7.258
6.889
6.868
5.830
5.813
5.796
3.795
3.034
3.018
3.011
2.995
2.990
2.975
2.968
2.959
2.953
2.944
2.938
2.922
2.901
2.894
2.879
2.404
2.388
2.384
2.368
2.353
2.347
2.332
2.327
2.312
2.290
2.275
2.267
2.259
2.252
2.239
2.224
2.217
2.201
2.033
0.000
MeO
O
OAc
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.02
1.04
2.05
2.04
2.02
1.00
3.08
2.07
2.09
3.03

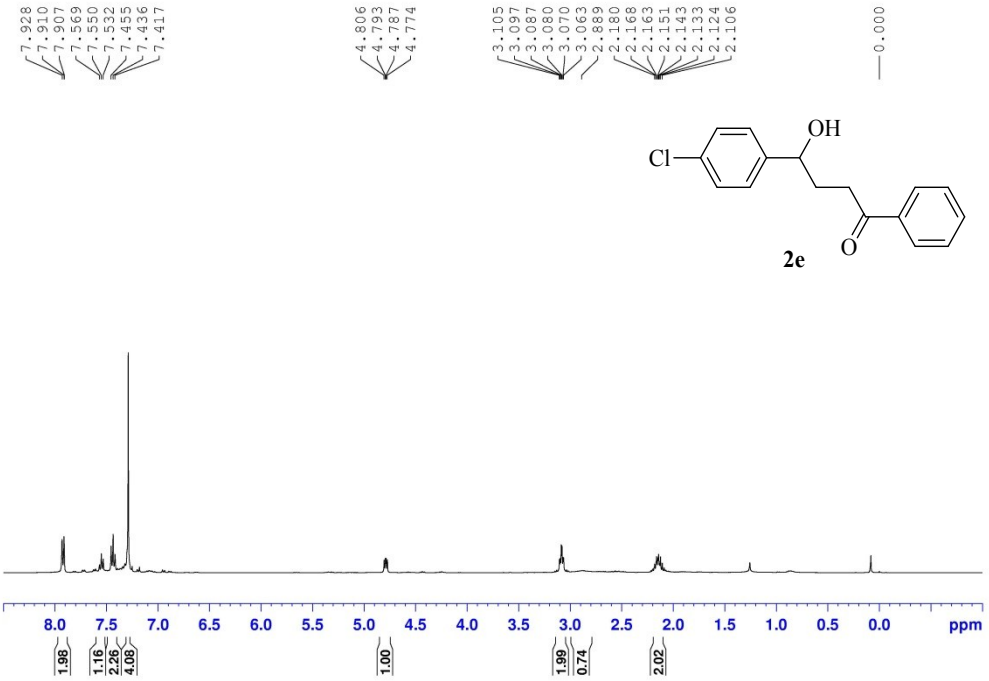
2c''


198.99
170.30
159.44
136.82
133.06
132.15
128.58
128.08
127.95
113.96
77.34
77.02
76.71
75.08
55.27
34.55
30.45
21.24
MeO
O
OAc
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
cdq140625-C35-cw-Py-OMe

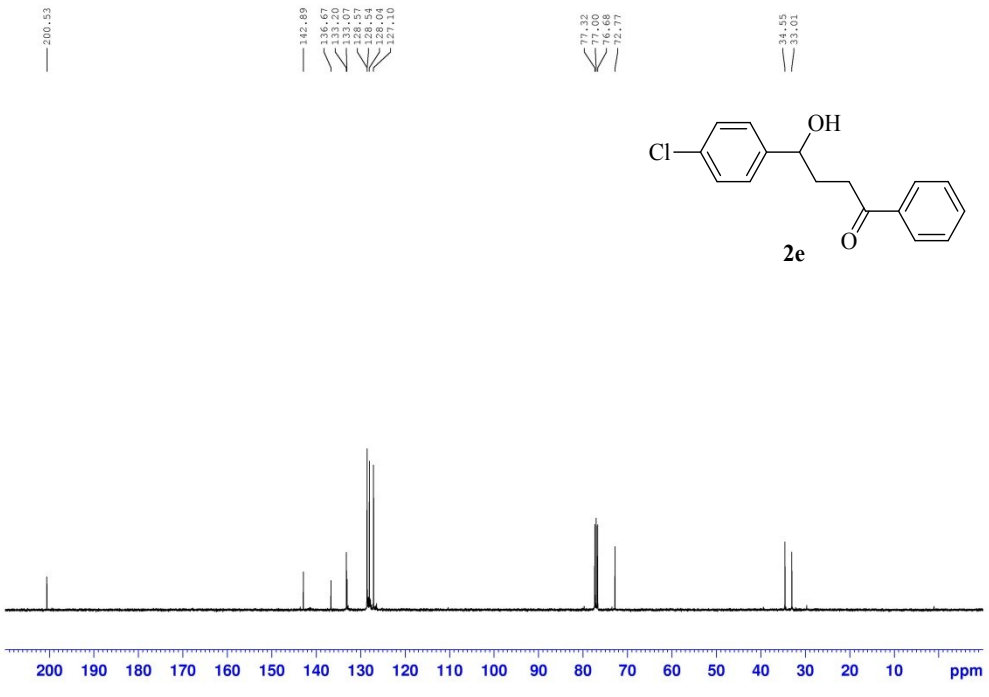
2c''


7.949
7.931
7.928
7.560
7.541
7.523
7.454
7.435
7.416
7.380
7.359
7.315
7.294
4.806
4.789
4.774
3.131
3.114
3.096
2.448
2.222
2.204
2.192
2.186
2.175
2.168
1.313
0.000
tBu
O
Ph
OH
2d
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.04
1.06
2.01
2.28
2.17
1.00
2.09
0.82
2.17
9.15

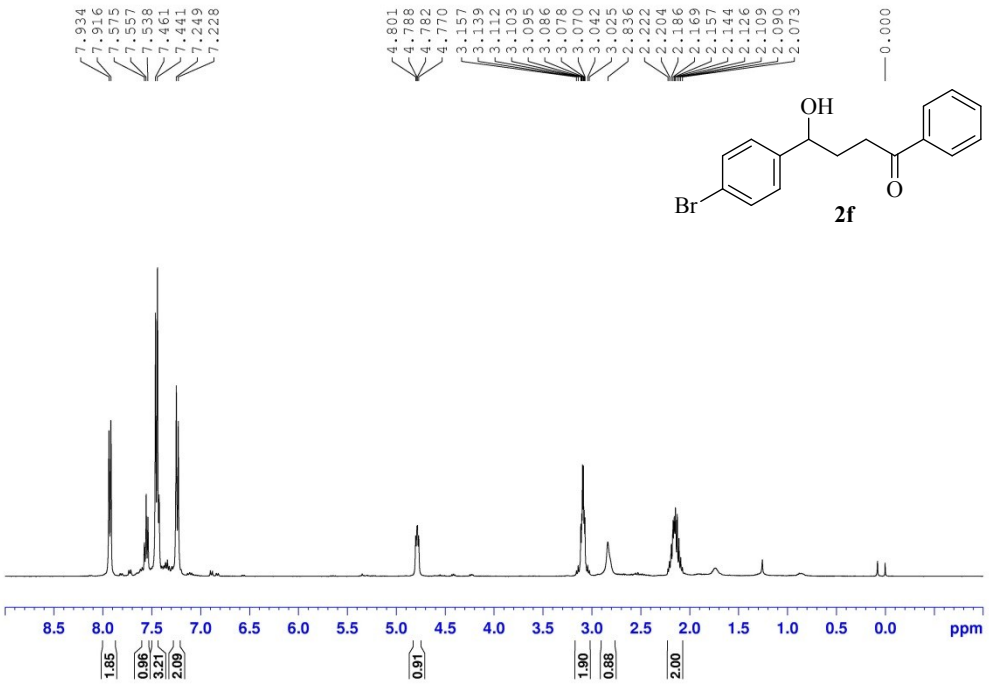

200.50
150.51
141.30
136.86
133.02
128.52
128.07
125.49
125.38
77.31
77.00
76.68
73.39
34.83
34.48
32.95
31.32
tBu
O
Ph
OH
2d
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

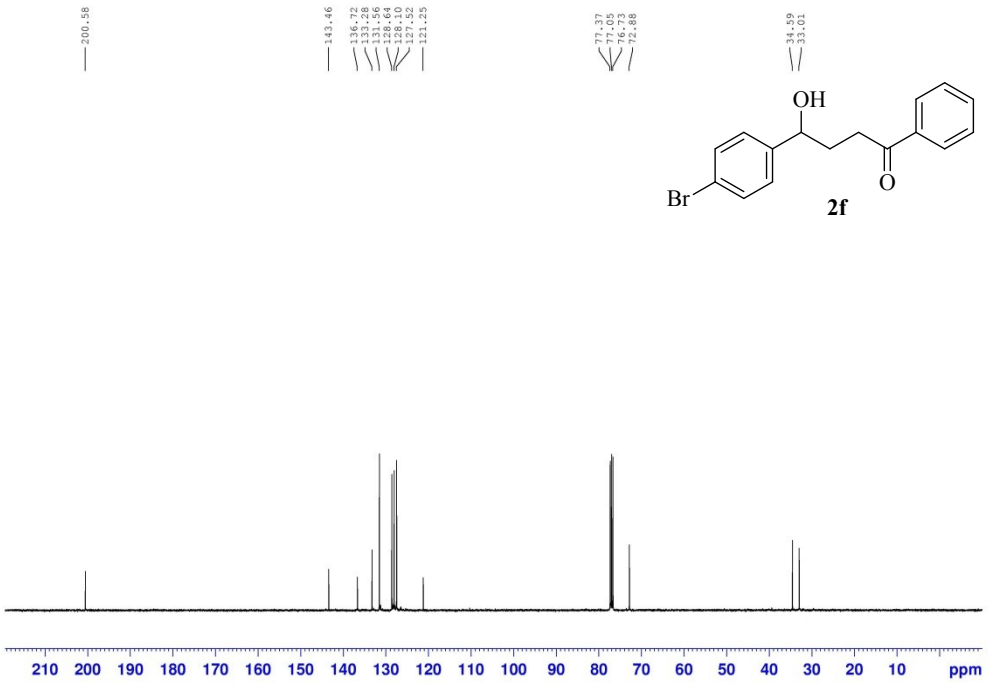

7.928
7.910
7.907
7.569
7.550
7.532
7.455
7.436
7.417
4.806
4.793
4.787
4.774
3.105
3.097
3.087
3.080
3.070
3.063
2.889
2.180
2.168
2.163
2.151
2.143
2.133
2.124
2.106
0.000
Cl
OH
O
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.98
1.16
2.26
4.08
1.00
1.99
0.74
2.02

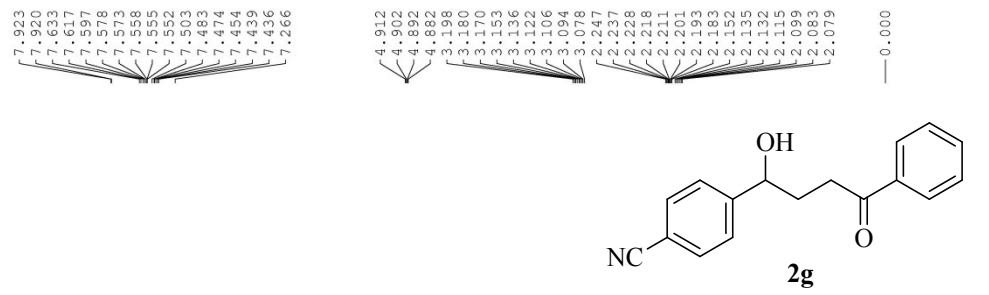
2e


200.53
142.89
136.67
133.20
133.07
128.57
128.54
128.04
127.10
77.32
77.00
76.68
72.77
34.55
33.01
Cl
OH
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

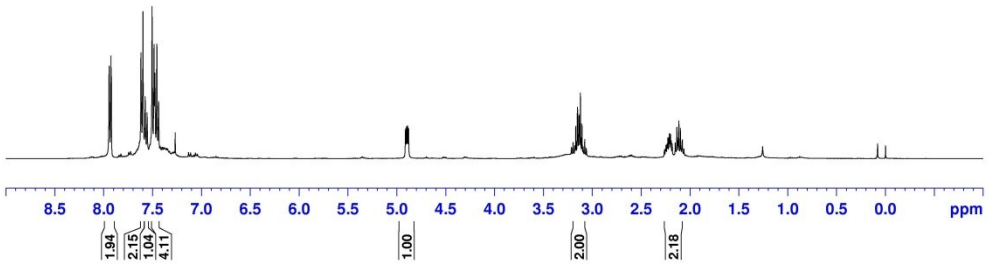
2e

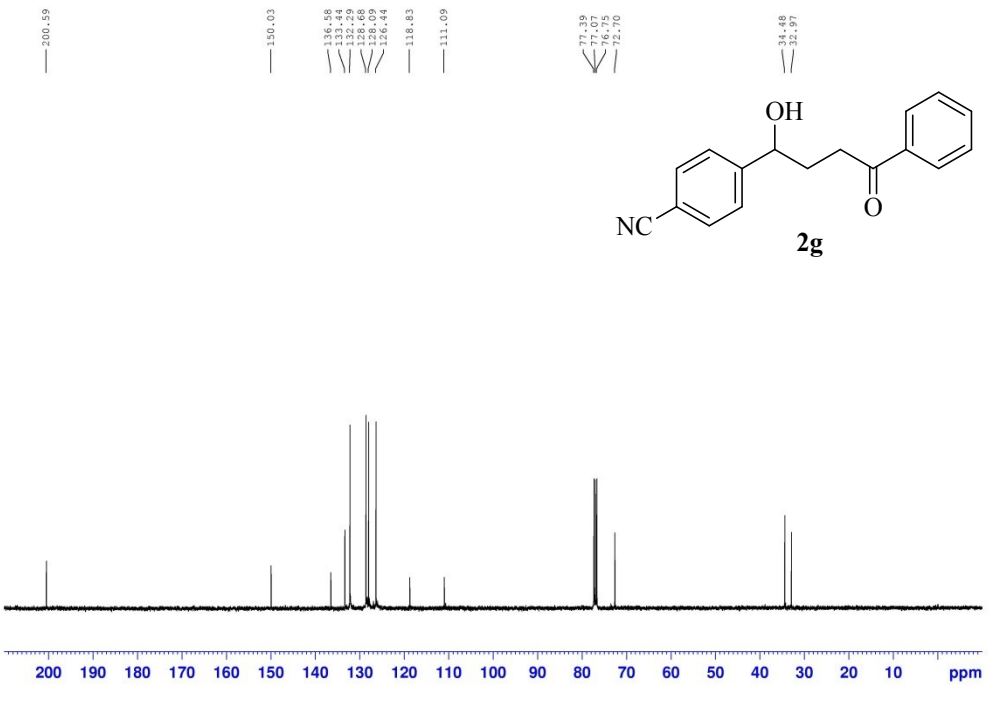

7.934
7.916
7.575
7.557
7.538
7.461
7.441
7.249
7.228
4.801
4.788
4.782
4.770
3.157
3.139
3.112
3.103
3.095
3.086
3.078
3.070
3.042
3.025
2.836
2.222
2.204
2.186
2.169
2.157
2.144
2.126
2.109
2.090
2.073
0.000
OH
Br
O
2f
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.85
0.96
3.21
2.09
0.91
1.90
0.86
2.00


200.58
143.46
136.72
133.28
131.56
129.64
128.10
127.52
121.25
77.37
77.05
76.73
72.88
34.59
33.01
OH
Br
O
2f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

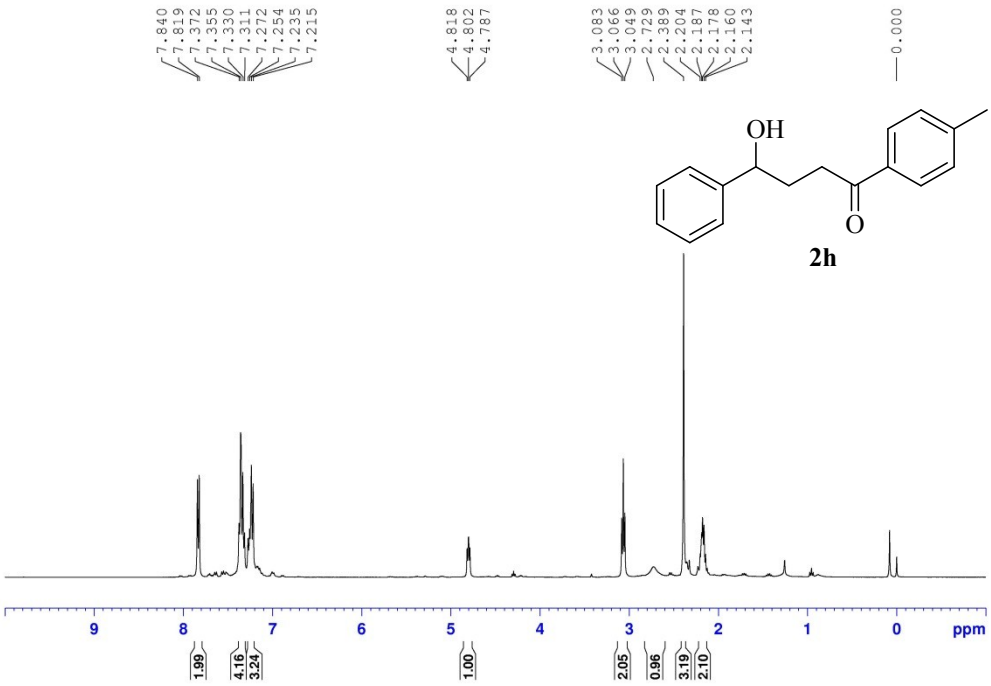

7.923
7.920
7.633
7.617
7.597
7.578
7.573
7.558
7.555
7.552
7.503
7.483
7.474
7.454
7.439
7.436
7.266
4.912
4.902
4.892
4.882
3.198
3.180
3.170
3.153
3.136
3.122
3.106
3.094
3.078
2.247
2.237
2.228
2.218
2.211
2.201
2.193
2.183
2.152
2.135
2.132
2.115
2.099
2.083
2.079
0.000
OH
NC
O

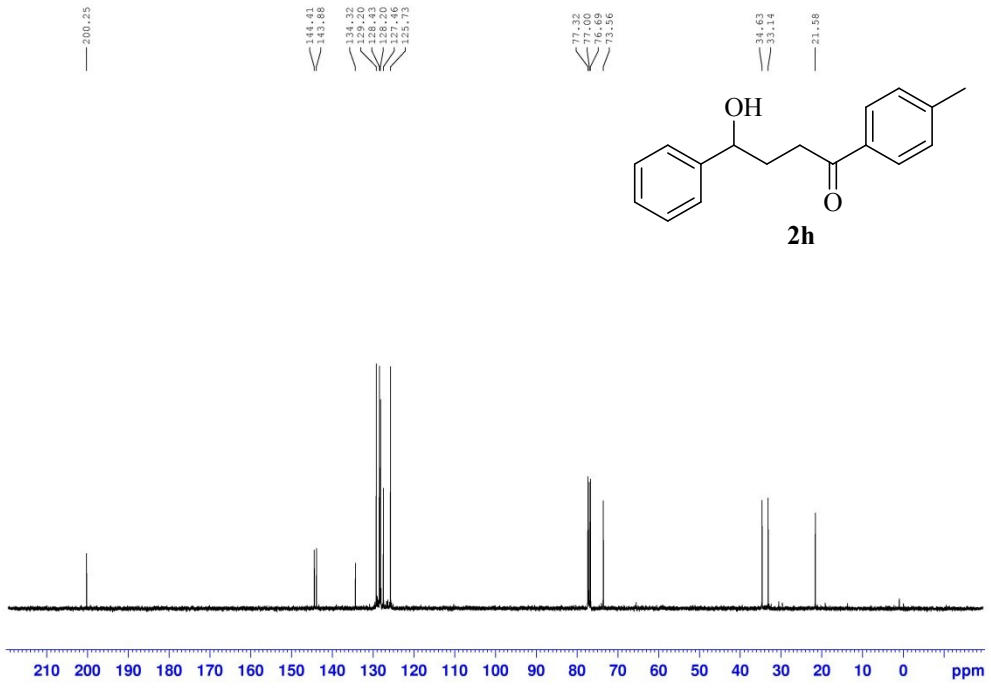
2g

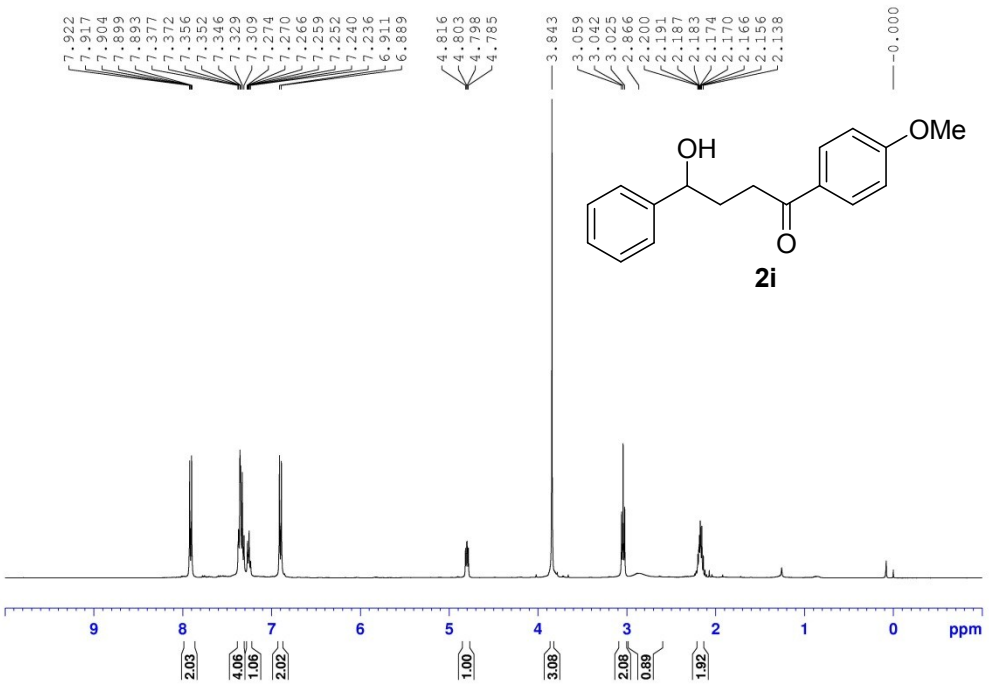

8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.94
2.15
1.04
4.11
1.00
2.00
2.18

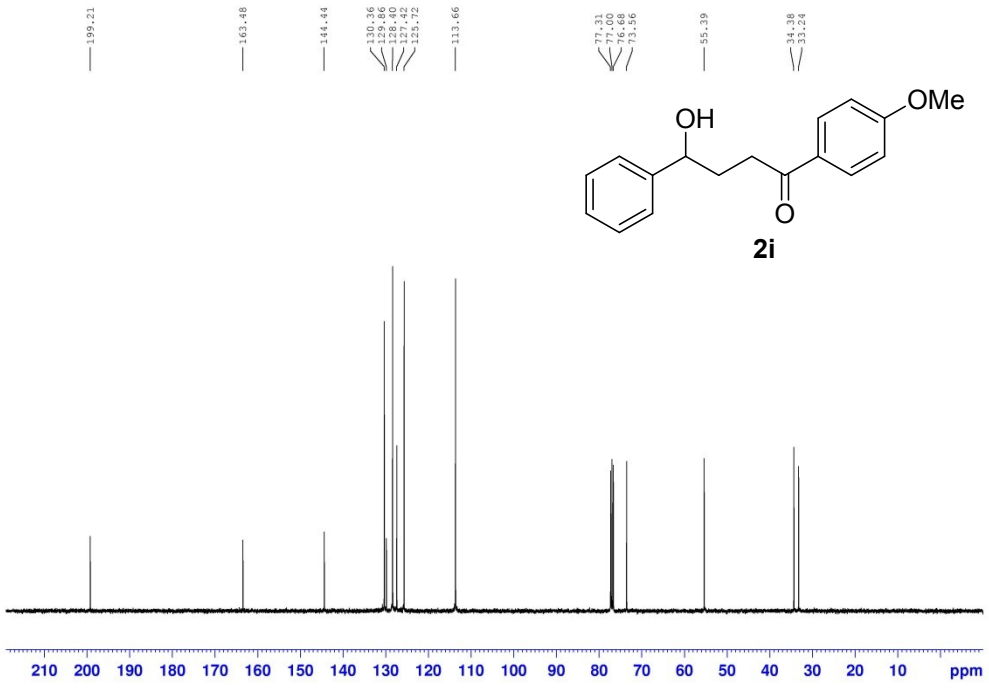

200.59
150.03
136.58
133.44
132.29
128.68
128.09
126.44
118.83
111.09
77.39
77.07
76.75
72.70
34.48
32.97
OH
NC
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

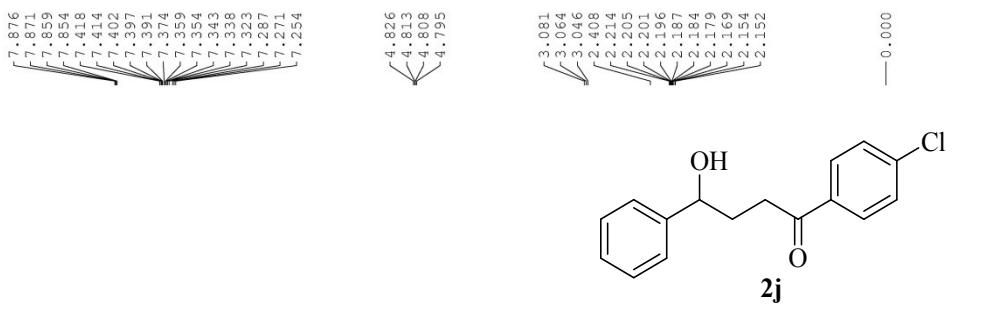
2g


7.840
7.819
7.372
7.355
7.330
7.311
7.272
7.254
7.235
7.215
4.818
4.802
4.787
3.083
3.066
3.049
2.729
2.389
2.204
2.187
2.178
2.160
2.143
0.000
OH
O
2h
9
8
7
6
5
4
3
2
1
0
ppm
1.99
4.16
3.24
1.00
2.05
0.96
3.19
2.10

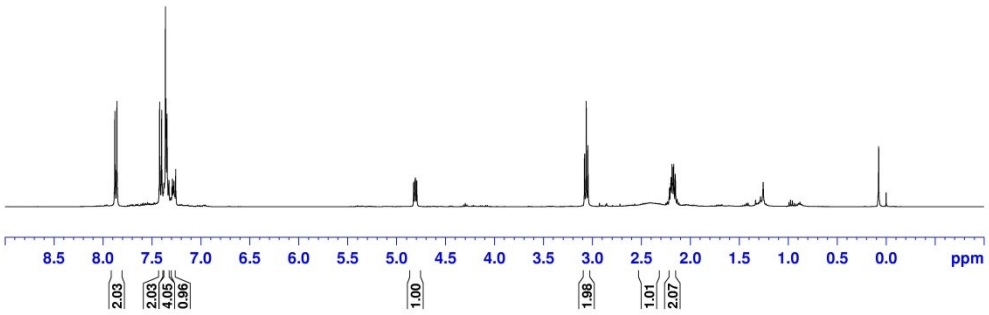

200.25
144.41
143.88
134.32
129.20
128.63
128.20
127.46
125.73
77.32
77.00
76.69
73.56
34.63
33.14
21.58
OH
O
2h
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

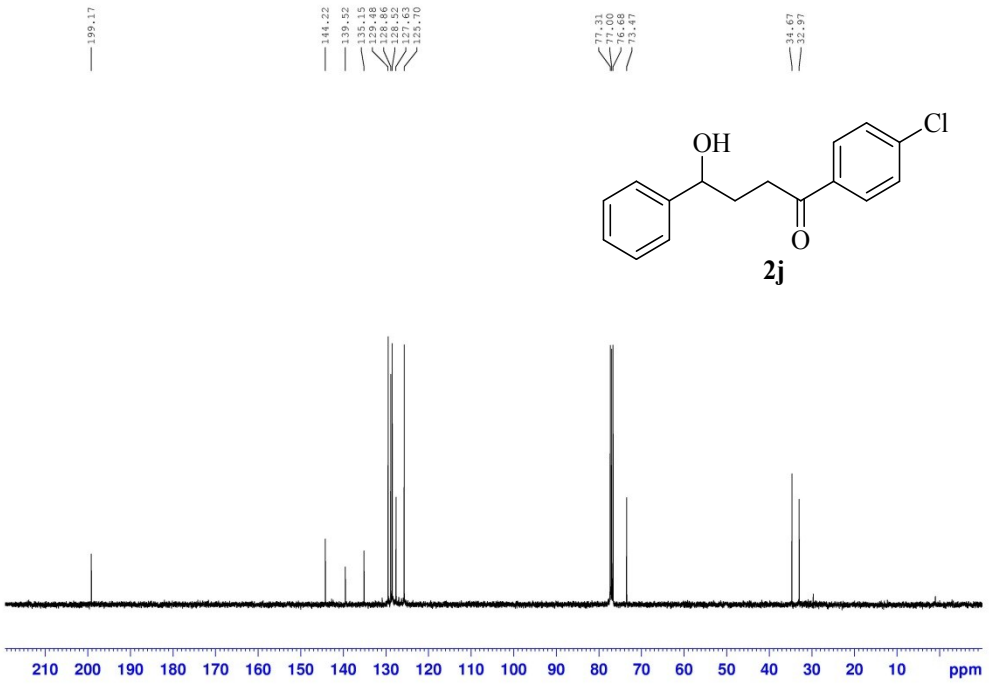

7.922
7.917
7.904
7.899
7.893
7.377
7.372
7.356
7.352
7.346
7.329
7.309
7.274
7.270
7.266
7.259
7.252
7.240
7.236
6.911
6.889
4.816
4.803
4.798
4.785
3.843
3.059
3.042
3.025
2.866
2.200
2.191
2.187
2.183
2.174
2.170
2.166
2.156
2.138
0.000
OH
OMe
O
2i
9
8
7
6
5
4
3
2
1
0
ppm
2.03
4.06
1.06
2.02
1.00
3.08
2.08
0.89
1.92


199.21
163.48
144.44
130.36
129.86
128.40
127.42
125.72
113.66
77.31
77.00
76.68
73.56
55.39
34.38
33.24
OH
OMe
O
2i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

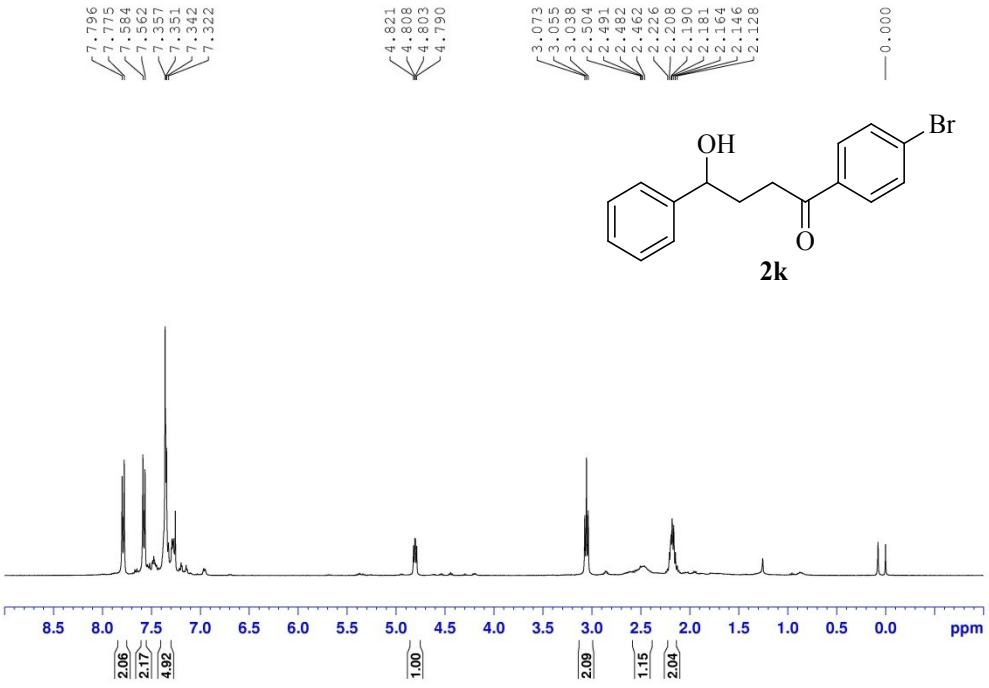

7.876
7.871
7.859
7.854
7.418
7.414
7.402
7.397
7.391
7.374
7.359
7.354
7.343
7.338
7.323
7.287
7.271
7.254
4.826
4.813
4.808
4.795
3.081
3.064
3.046
2.408
2.214
2.205
2.201
2.196
2.187
2.184
2.179
2.169
2.154
2.152
0.000
OH
Cl
O

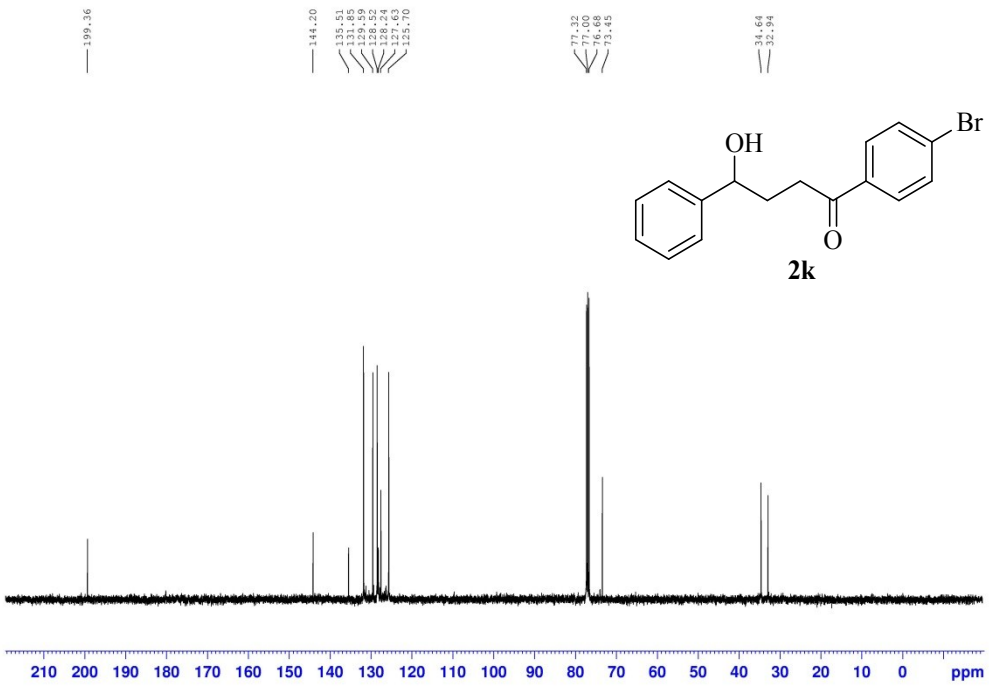
2j

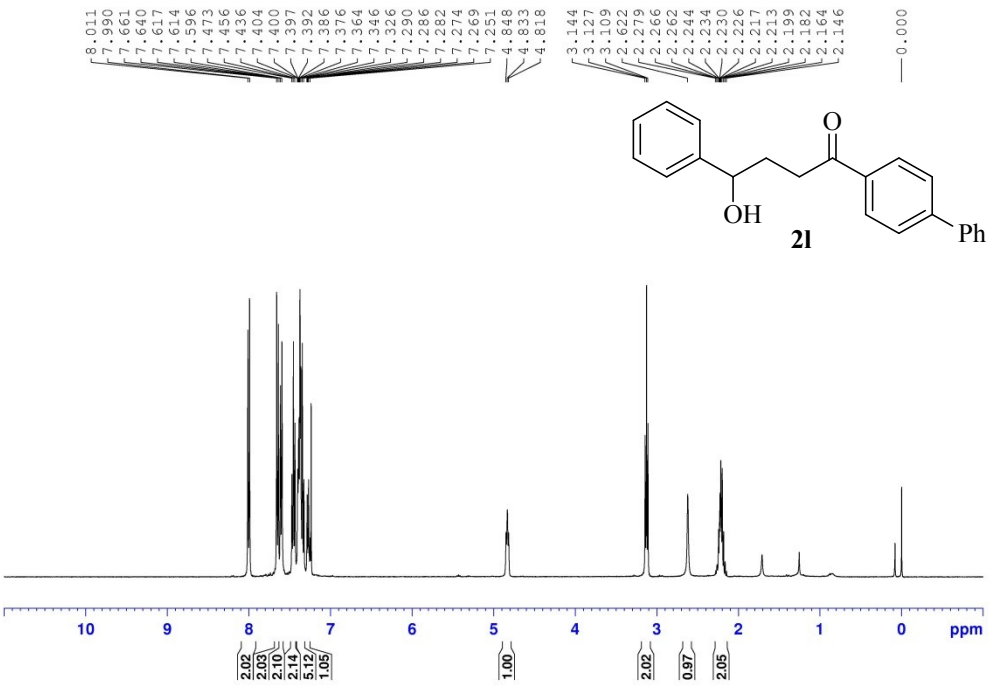

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.03
2.03
4.05
0.96
1.00
1.98
1.01
2.07


199.17
144.22
139.52
135.15
129.88
128.86
128.52
127.63
125.70
77.31
77.00
76.68
73.47
34.67
32.97
OH
Cl
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

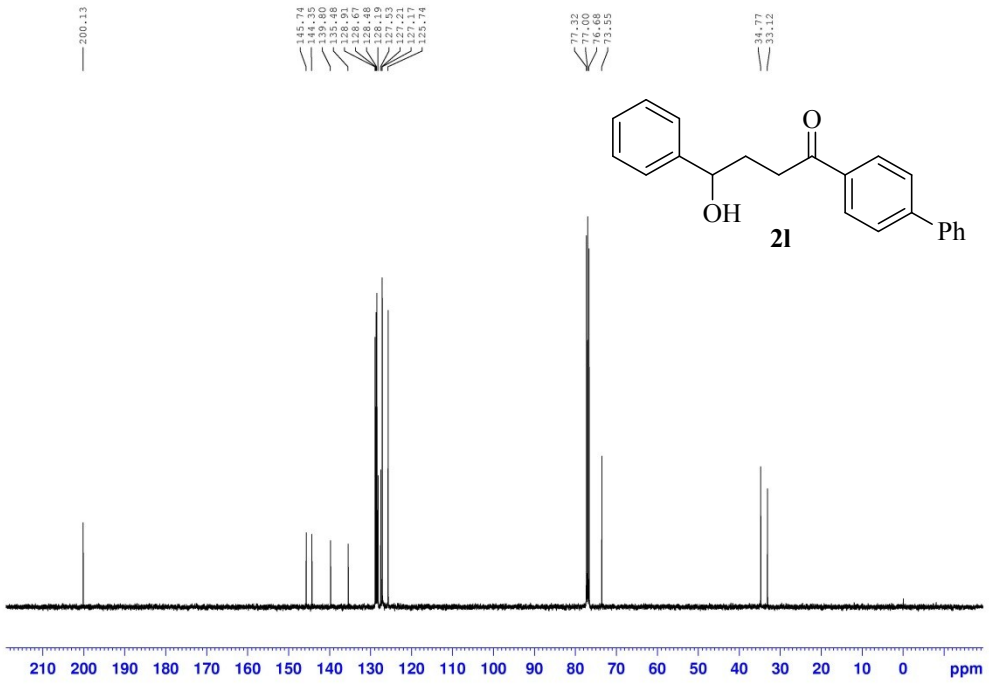
2j


7.796
7.775
7.584
7.562
7.357
7.351
7.342
7.322
4.821
4.808
4.803
4.790
3.073
3.055
3.038
2.504
2.491
2.482
2.462
2.226
2.208
2.190
2.181
2.164
2.146
2.128
0.000
OH
Br
O
2k
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.06
2.17
4.92
1.00
2.09
1.15
2.04

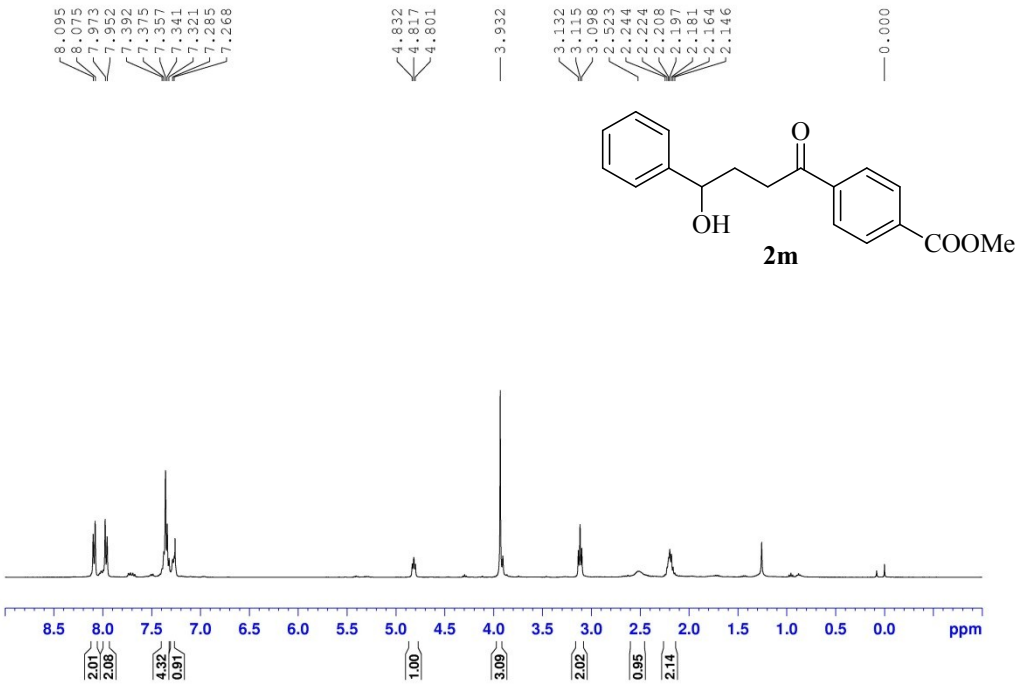

199.36
144.20
135.51
131.85
129.59
128.52
128.24
127.63
125.70
77.32
77.00
76.68
73.45
34.64
32.94
OH
Br
O
2k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

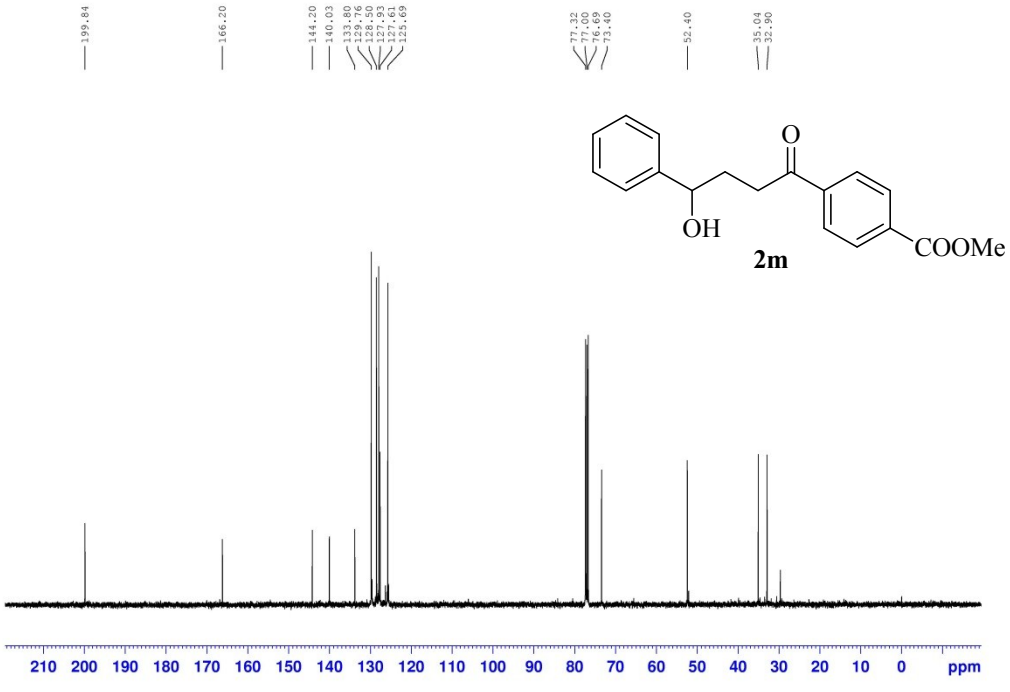

8.011
7.990
7.661
7.640
7.617
7.614
7.596
7.473
7.456
7.436
7.404
7.400
7.397
7.392
7.386
7.376
7.364
7.346
7.326
7.290
7.286
7.282
7.274
7.269
7.251
4.848
4.833
4.818
3.144
3.127
3.109
2.622
2.279
2.266
2.262
2.244
2.234
2.230
2.226
2.217
2.213
2.199
2.182
2.164
2.146
0.000
O
OH
Ph
10
9
8
7
6
5
4
3
2
1
0
ppm
2.02
2.03
2.10
2.14
5.12
1.05
1.00
2.02
0.97
2.05

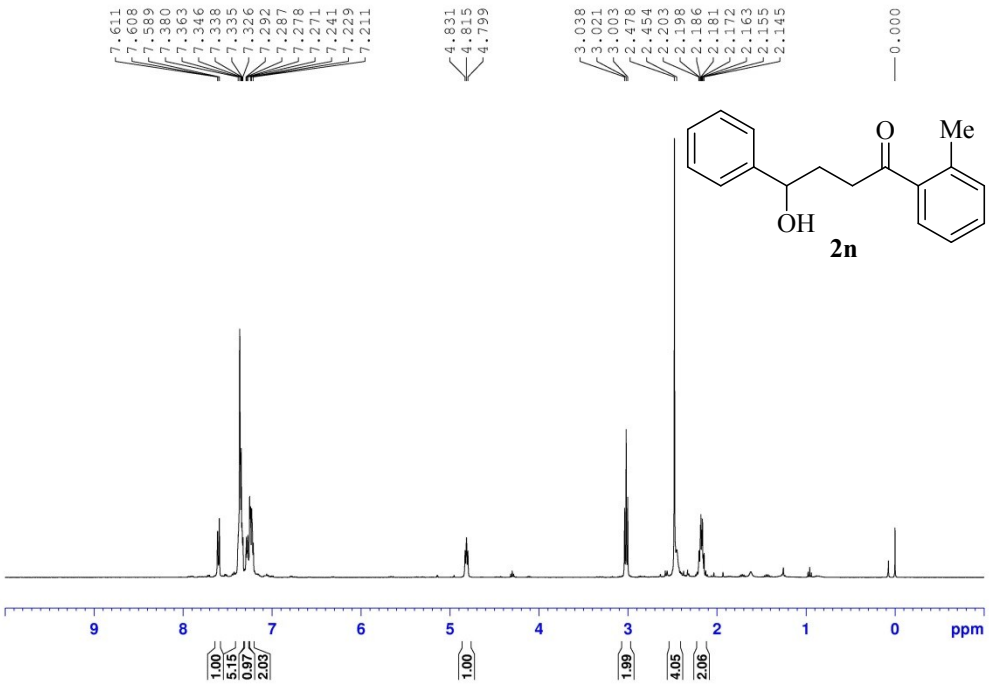
2l

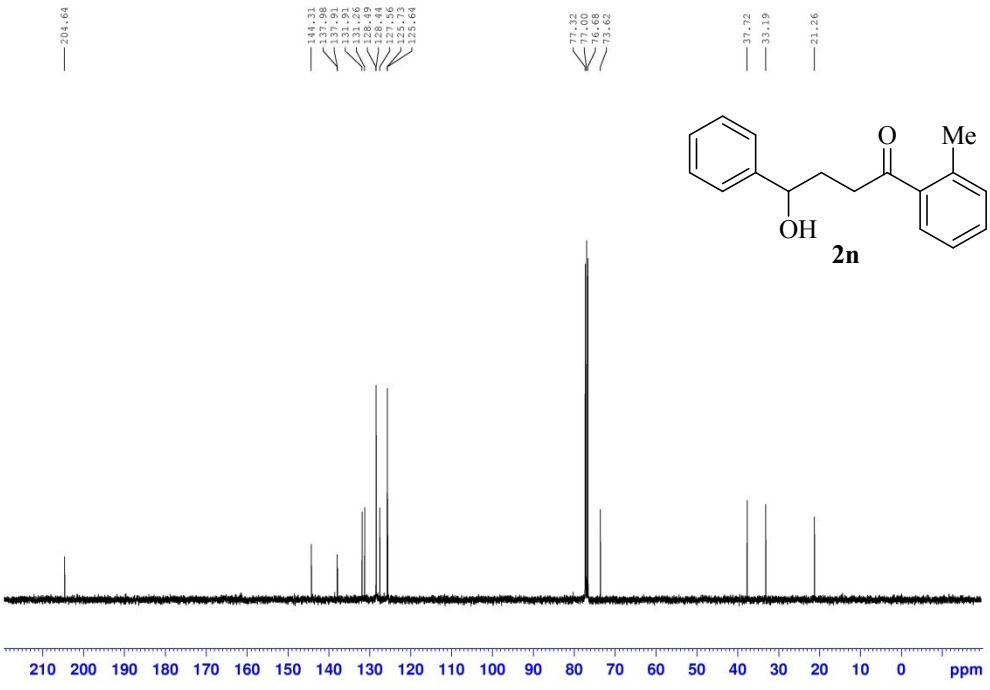

200.13
145.74
144.35
139.80
135.48
128.71
128.67
128.48
128.19
127.53
127.21
127.17
125.74
77.32
77.00
76.68
73.55
34.77
33.12
O
OH
Ph
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

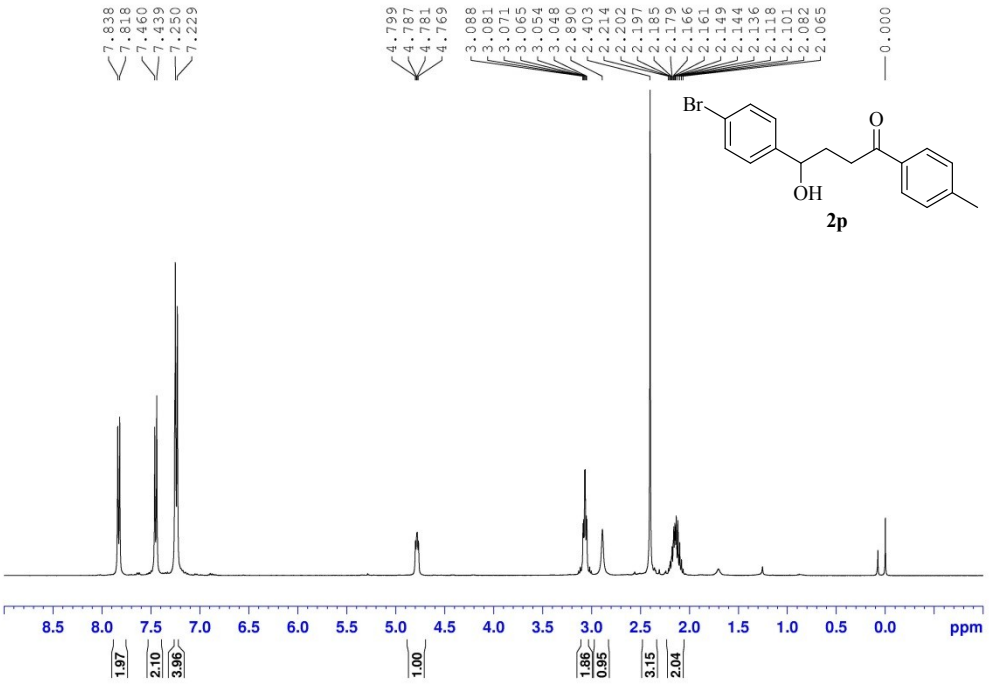
2l


8.095
8.075
7.973
7.952
7.392
7.375
7.357
7.341
7.321
7.285
7.268
4.832
4.817
4.801
3.932
3.132
3.115
3.098
2.523
2.244
2.224
2.208
2.197
2.181
2.164
2.146
0.000
O
OH
COOMe
2m
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.01
2.08
4.32
0.91
1.00
3.09
2.02
0.95
2.14

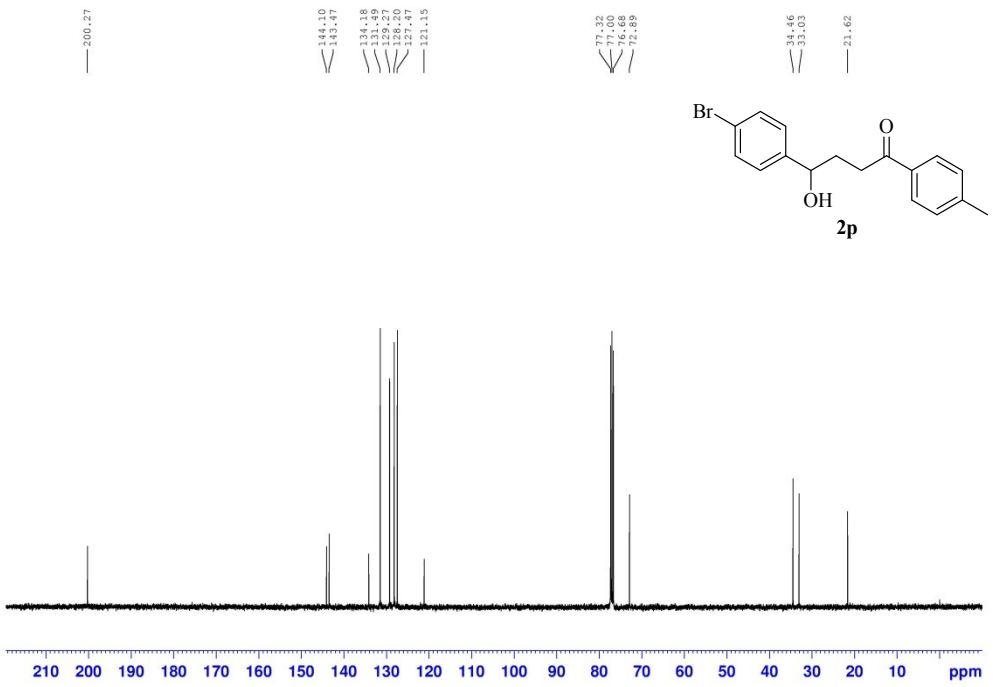

199.84
166.20
144.20
140.03
133.80
129.76
128.50
127.93
127.61
125.69
77.32
77.00
76.68
73.40
52.40
35.04
32.90
O
OH
COOMe
2m
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.611
7.608
7.589
7.380
7.363
7.346
7.338
7.335
7.326
7.292
7.287
7.278
7.271
7.241
7.229
7.211
4.831
4.815
4.799
3.038
3.021
3.003
2.478
2.454
2.203
2.198
2.186
2.181
2.172
2.163
2.155
2.145
0.000
O
Me
OH
2n
9
8
7
6
5
4
3
2
1
0
ppm
1.00
5.15
0.97
2.03
1.00
1.99
4.05
2.06

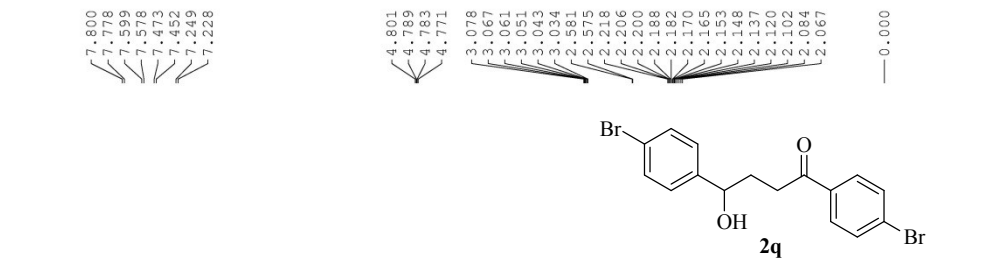

204.64
144.31
137.98
137.91
131.91
131.06
128.49
128.44
127.56
125.73
125.64
77.32
77.00
76.68
73.62
37.72
33.19
21.26
O
Me
OH
2n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.838
7.818
7.460
7.439
7.250
7.229
4.799
4.787
4.781
4.769
3.088
3.081
3.071
3.065
3.054
3.048
2.890
2.403
2.214
2.202
2.197
2.185
2.179
2.166
2.161
2.149
2.144
2.136
2.118
2.101
2.082
2.065
0.000
Br
O
OH
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.97
2.10
3.96
1.00
1.86
0.95
3.15
2.04

2p

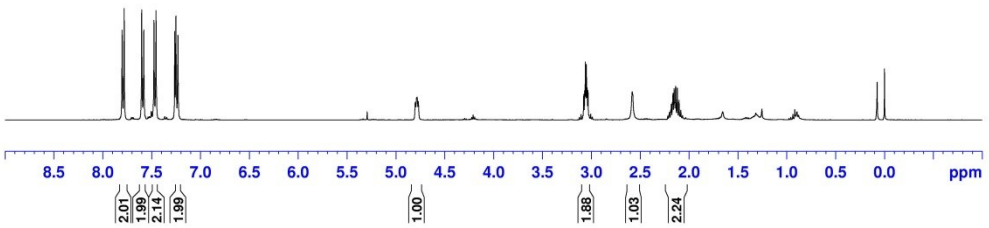

200.27
144.10
143.47
134.18
131.99
130.27
128.20
127.47
121.15
77.32
77.00
76.68
72.89
34.46
33.03
21.62
Br
O
OH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

2p

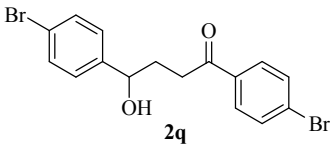

7.800
7.778
7.599
7.578
7.473
7.452
7.249
7.228
4.801
4.789
4.783
4.771
3.078
3.067
3.061
3.051
3.043
3.034
2.581
2.575
2.218
2.206
2.200
2.188
2.182
2.170
2.165
2.153
2.148
2.137
2.120
2.102
2.084
2.067
0.000

Br
O
OH
Br

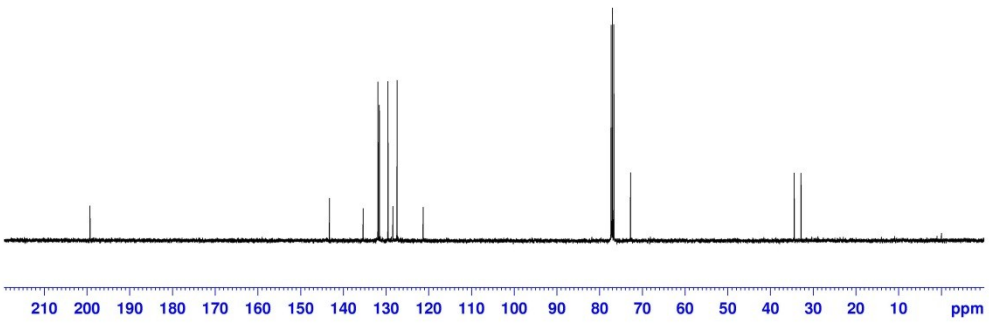
2q

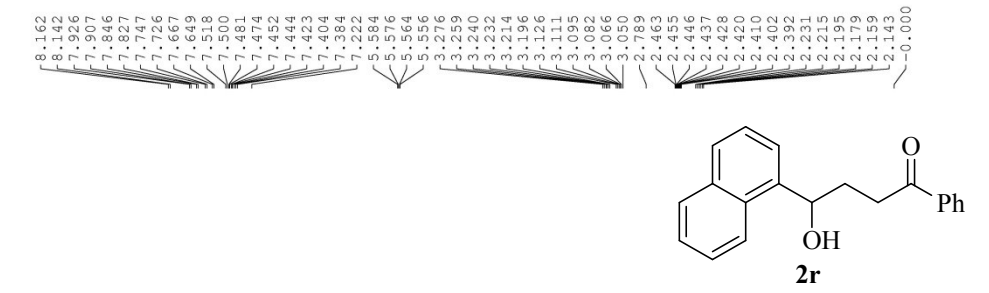

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.01
1.99
2.14
1.99
1.00
1.88
1.03
2.24


199.33
143.26
135.39
131.91
131.58
129.57
128.22
127.43
121.33
77.32
77.00
76.68
72.77
34.47
32.84

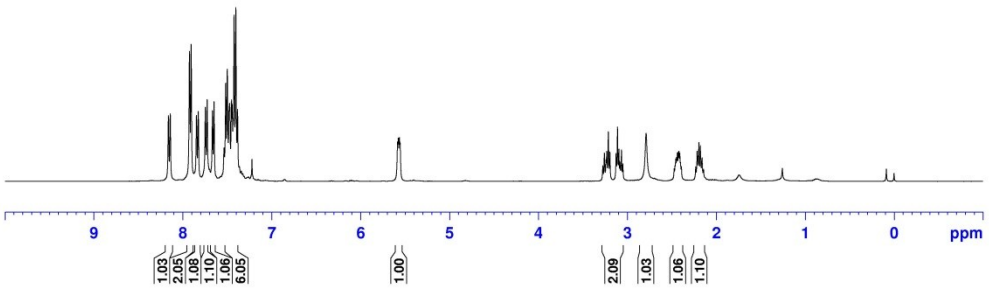

Br
O
OH
Br

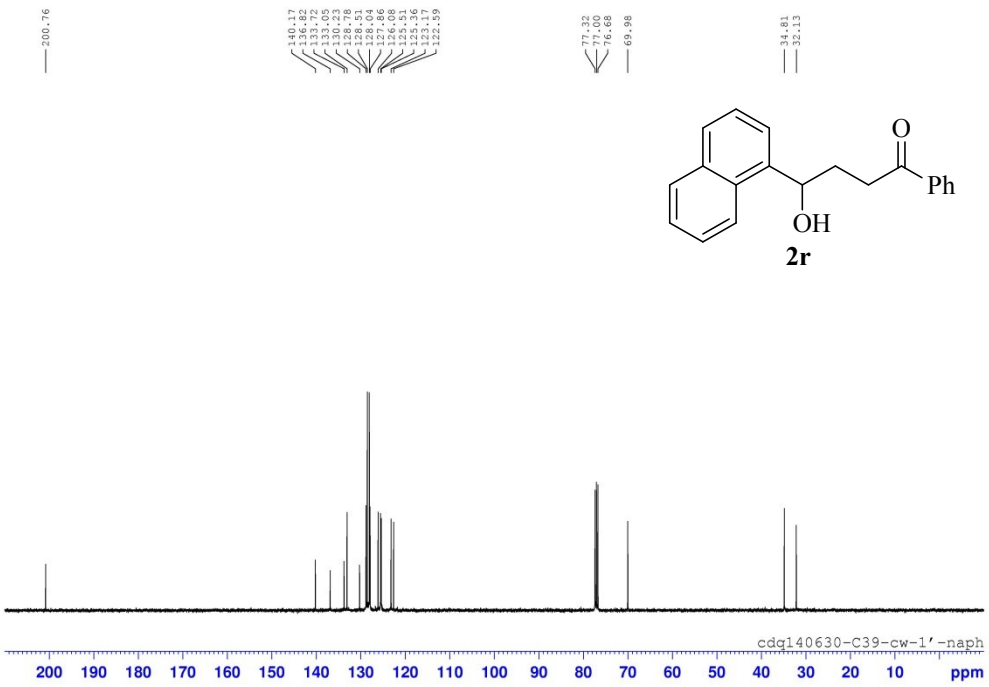
2q


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

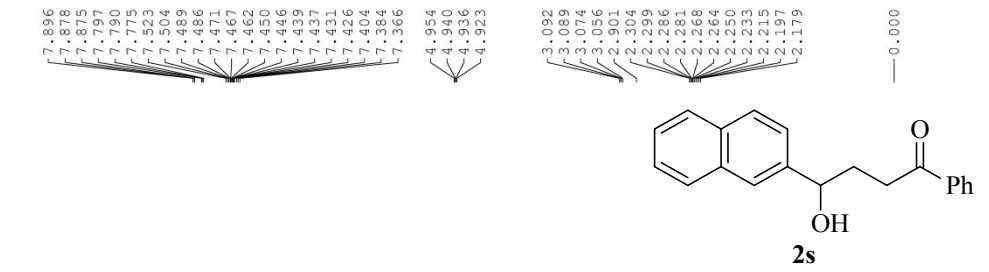

8.162
8.142
7.926
7.907
7.846
7.827
7.747
7.726
7.667
7.649
7.518
7.500
7.481
7.474
7.452
7.444
7.423
7.404
7.384
7.222
5.584
5.576
5.564
5.556
3.276
3.259
3.240
3.232
3.214
3.196
3.126
3.111
3.095
3.082
3.066
3.050
2.789
2.463
2.455
2.446
2.437
2.428
2.420
2.410
2.402
2.392
2.231
2.215
2.195
2.179
2.159
2.143
-0.000
O
Ph
OH

2r

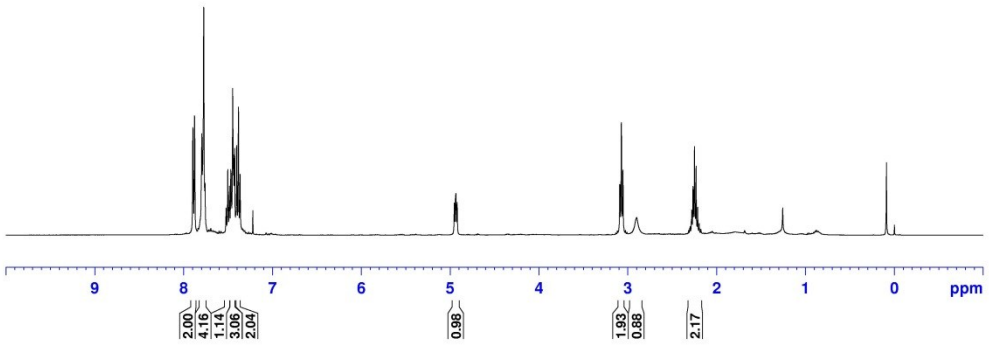

9
8
7
6
5
4
3
2
1
0
ppm
1.03
2.05
1.08
1.10
1.06
6.05
1.00
2.09
1.03
1.06
1.10

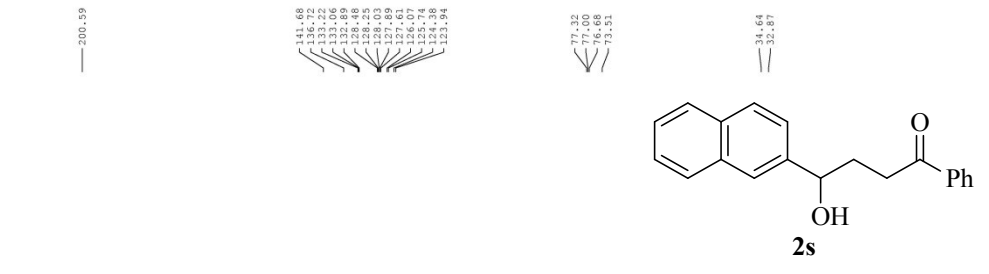

200.76
140.17
136.22
133.72
133.05
130.23
128.78
128.51
128.04
127.86
126.08
125.91
125.56
123.17
122.59
77.32
77.00
76.68
69.98
34.81
32.13
O
Ph
OH
cdq140630-C39-cw-1'-naph
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

2r

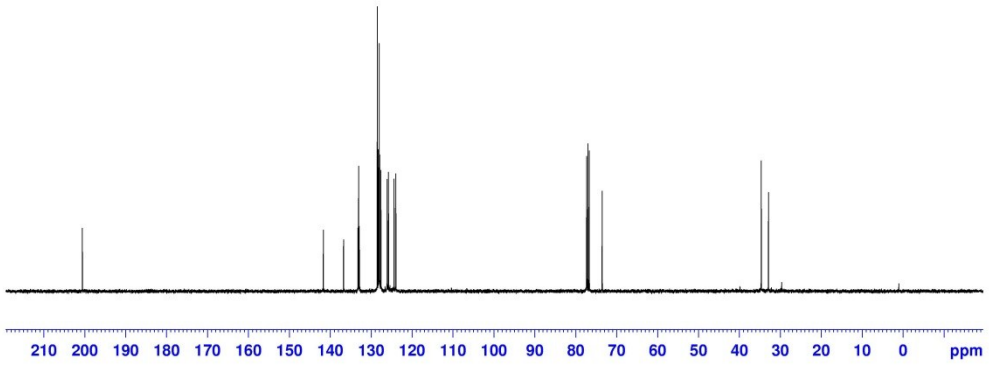

7.896
7.878
7.875
7.797
7.790
7.775
7.523
7.504
7.489
7.486
7.471
7.467
7.462
7.450
7.446
7.439
7.437
7.431
7.426
7.404
7.384
7.366
4.954
4.940
4.936
4.923
3.092
3.089
3.074
3.056
2.901
2.304
2.299
2.286
2.281
2.268
2.264
2.250
2.233
2.215
2.197
2.179
0.000
O
Ph
OH

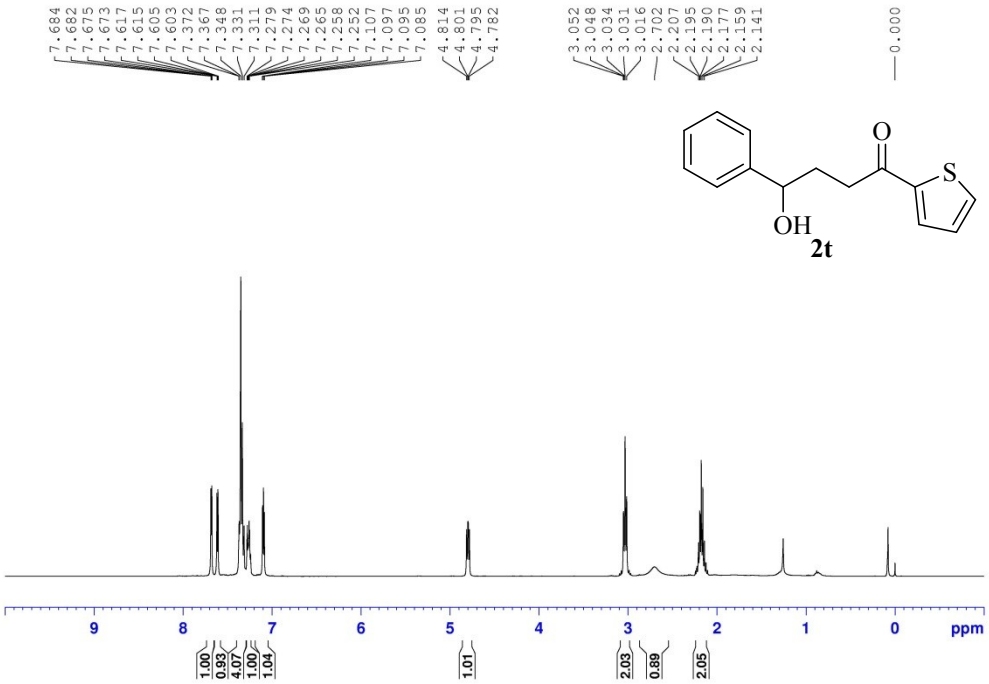
2s

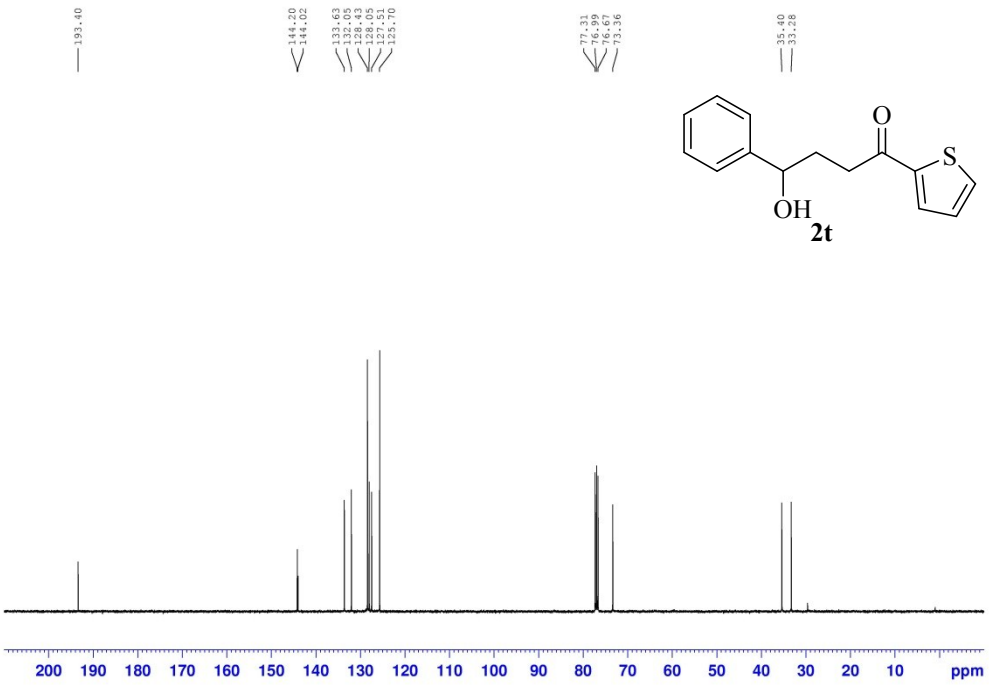

9
8
7
6
5
4
3
2
1
0
ppm
2.00
4.16
1.14
3.06
2.04
0.98
1.93
0.88
2.17


200.59
141.68
136.72
133.22
133.06
128.68
128.48
128.25
128.03
127.69
127.61
126.07
125.74
124.38
123.94
77.32
77.00
76.68
75.51
34.64
32.87
O
Ph
OH

2s


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.684
7.682
7.675
7.673
7.617
7.615
7.605
7.603
7.372
7.367
7.348
7.331
7.311
7.279
7.274
7.269
7.265
7.258
7.252
7.107
7.097
7.095
7.085
4.814
4.801
4.795
4.782
3.052
3.048
3.034
3.031
3.016
2.702
2.207
2.195
2.190
2.177
2.159
2.141
0.000
O
S
OH
2t
9
8
7
6
5
4
3
2
1
0
ppm
1.00
0.93
4.07
1.00
1.04
1.01
2.03
0.89
2.05


193.40
144.20
144.02
133.63
132.05
128.43
128.05
127.51
125.70
77.31
76.99
76.67
73.36
35.40
33.28
O
S
OH
2t
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm