Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Phase-Transfer-Catalyzed Cyclization Reaction of Nucleophilic Addition to Electron-Deficient 1,3-Conjugated Enynes for Synthesis of Functionalized 4*H*-Pyran

Jie Hu,[a] Lei Liu,[a] Shangdong Yang,[a,*] and Yong-Min Liang[a,*]

[a] State Key Laboratory of Applied Organic Chemistry, Lanzhou University, Lanzhou 730000, P.R. China.
Fax: +86-931-8912582; Tel: +86-931-8912593; e-mail: yangshd@lzu.edu.cn ; liangym@lzu.edu.cn,

Table of Contents

General Remarks:

Column chromatography was carried out on silica gel. ^{1}H NMR spectra were recorded on 400 MHz in $CDCl_3$ and ^{13}C NMR spectra were recorded on 100 MHz in $CDCl_3$ using TMS as internal standard. IR spectra were recorded on a FT-IR spectrometer and only major peaks are reported in cm^{-1}. All new compounds were further characterized by element analysis; copies of their ^{1}H NMR and ^{13}C NMR spectra are provided. Commercially available reagents and solvents were used without further purification.

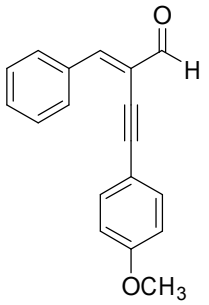
Starting Materials:

The preparation of the PTC 1 and PTC 2 were described in previous report,[1] the starting materials **1a**, **1b** and **1g** were prepared according to the previous literature[2-5] and the synthesis of the remaining substrates were similar.

(1) K. Friedrich and H. Henning, *Chem. Ber*. 92, 2756 (**1959**).
(2) M. Lautens, M. L. Maddess, E. L. O. Sauer, S. G. Ouellet, *Org Lett.* **2002**, *4*, 83.
(3) M. Lautens, M. L. Maddess, *Org Lett.* **2004**, *6*, 1883.
(4) T. Yao, X. Zhang, R. C. Larock, *J. Am. Chem. Soc.* **2004**, *126*, 11164.
(5) X. Yu, B. Du, K. Wang, J. Zhang, *Org Lett.* **2010**, *12*, 1876.

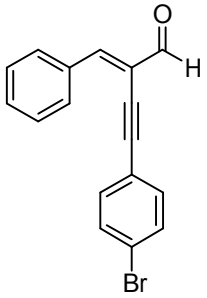
Typical procedure for the preparation of 2

To a solution of **1** (0.20 mmol) in 2.0 mL of CH_2Cl_2 was added Cs_2CO_3 (130.3 mg, 0.40 mmol) in the reaction vessel. The mixture was allowed to stir at room temperature for 1 minute and $TBAF{\cdot}3H_2O$ (3.15 mg, 5 mol %) was added. The vessel was sealed and the resulting mixture was then heated at 60 ^{o}C. When the reaction was considered complete as determined by TLC analysis, the reaction was allowed to cool to room temperature and quenched with a saturated aqueous solution of ammonium chloride, and the mixture was extracted with EtOAc. The combined organic extracts were washed with water and saturated brine. The organic layers were dried over Na_2SO_4, filtered. Solvents were evaporated under reduced pressure. The residue was purified by chromatography on silica gel to afford **2.**

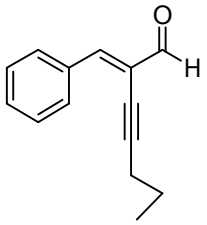


(*E*)-2-benzylidene-4-(4-methoxyphenyl)but-3-ynal (**1c**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.62 (s, 1H), 8.14-8.11 (m, 2H), 7.54-7.45 (m, 6H), 6.91-6.89 (d, J = 8.4 Hz, 2H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.1, 160.2, 150.3, 134.2, 133.3, 131.4, 130.5, 128.7, 122.9, 114.6, 114.1, 101.2, 82.2, 55.3. IR (neat, cm^{-1}): 3367, 2198, 1692, 1508, 1250, 1027, 832, 758. Anal. Calcd for $C_{18}H_{14}O_2$: C 82.42; H 5.38. Found: C

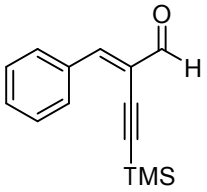
82.49; H 5.42.



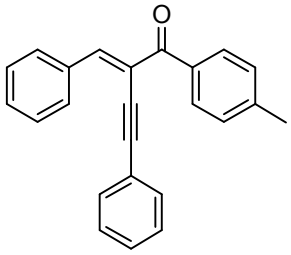
(*E*)-2-benzylidene-4-(4-bromophenyl)but-3-ynal (**1d**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12-8.09 (m, 2H), 7.55-7.43 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.7, 151.7, 134.0, 133.2, 131.8, 131.7, 130.6, 128.8, 123.5, 122.4, 121.4, 99.7, 84.2. IR (neat, cm^{-1}): 3371, 2204, 1692, 1484, 1137, 1069, 823, 756. Anal. Calcd for $C_{17}H_{11}BrO$: C 65.62; H 3.56. Found: C 65.68; H 3.46.



(*E*)-2-benzylidenehept-3-ynal (**1e**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.53 (s, 1H), 8.09-8.07 (m, 2H), 7.43-7.39 (m, 4H), 2.53-2.49 (t, J = 7.2 Hz, 2H), 1.71-1.65 (t, J = 7.2, 14.4 Hz, 2H), 1.09-1.05 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.4, 150.7, 134.0, 131.1, 128.4, 123.3, 103.1, 74.4, 21.7, 21.7, 13.4. IR (neat, cm^{-1}): 3366, 2964, 1692, 1598, 1176, 758, 689. Anal. Calcd for $C_{14}H_{14}O$: C 84.81; H 7.12. Found: C 84.78; H 7.23.

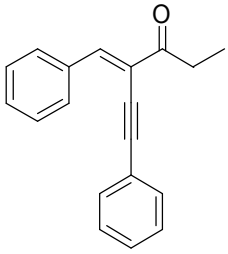


(*E*)-2-benzylidene-4-(trimethylsilyl)but-3-ynal (**1f**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.52 (s, 1H), 8.12-8.10 (m, 2H), 7.48 (s, 1H), 7.44-7.42 (m, 3H), -0.30 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.5, 152.1, 133.7, 131.5, 130.5, 128.4, 122.2, 107.5, 98.4, -0.59. IR (neat, cm^{-1}): 3367, 2960, 1692, 1596, 1158, 891, 759, 689. Anal. Calcd for $C_{14}H_{16}OSi$: C 73.63; H 7.06. Found: C 73.69; H 7.15.

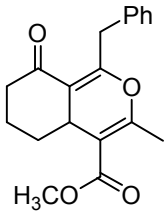


(*E*)-2-benzylidene-4-phenyl-1-*p*-tolylbut-3-yn-1-one (**1h**). ^{1}H NMR (400 MHz,

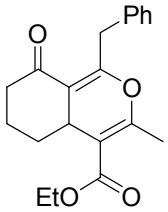
$CDCl_3$) δ 8.09-8.07 (d, *J* = 6.8 Hz, 2H), 7.94-7.92 (d, *J* = 8.4 Hz, 2H), 7.57 (s, 1H), 7.42-7.37 (m, 5H), 7.29-7.23 (m, 5H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.7, 144.5, 143.3, 134.8, 134.2, 131.2, 130.3, 130.1, 130.0, 129.8, 128.7, 128.4, 128.3, 122.8, 121.0, 100.6, 87.2, 21.5. IR (neat, cm^{-1}): 3058, 2921, 1659, 1603, 1264, 1179, 757, 689. Anal. Calcd for $C_{24}H_{18}O$: C 89.41; H 5.63. Found: C 89.50; H 5.53.



(*E*)-4-benzylidene-6-phenylhex-5-yn-3-one (**1i**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10-8.08 (dd, *J* = 2.0 Hz, 7.2 Hz, 2H), 7.85 (s, 1H), 7.56-7.54 (m, 2H), 7.44-7.39 (m, 6H), 3.08-3.02 (dd, *J* = 7.2 Hz, 14.4 Hz, 2H), 1.22-1.19 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.9, 142.6, 134.6, 131.3, 130.7, 130.6, 128.9, 128.6, 128.5, 122.8, 119.6, 99.0, 86.7, 33.7, 8.2. IR (neat, cm^{-1}): 3376, 2934, 1696, 1141, 755, 689. Anal. Calcd for $C_{19}H_{16}O$: C 87.66; H 6.19. Found: C 87.58; H 6.10.

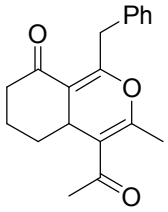


Methyl 1-benzyl-5,6,7,8-tetrahydro-3-methyl-8-oxo-4a*H*-isochromene-4-carboxylate (**2aa**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.21 (m, 5H), 3.87-3.83 (d, *J* = 14.0 Hz, 1H), 3.75-3.68 (m, 4H), 3.49-3.45 (dd, *J* = 4.0, 12.0 Hz, 1H), 2.56-2.51 (m, 1H), 2.49-2.35 (m, 1H), 2.17-2.11 (m, 4H), 1.91-1.84 (m, 2H), 1.61-1.51 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.4, 167.5, 157.2, 153.3, 137.2, 128.9, 128.3, 126.5, 113.7, 106.5, 51.3, 40.9, 35.9, 33.3, 31.7, 21.3, 18.5. IR (neat, cm^{-1}): 3298, 2928, 1708, 763, 700. Anal. Calcd for $C_{19}H_{20}O_4$: C 73.06; H 6.45. Found: C 73.15; H 6.36.

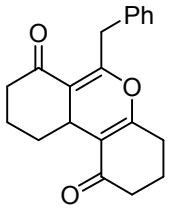


Ethyl 1-benzyl-5,6,7,8-tetrahydro-3-methyl-8-oxo-4a*H*-isochromene-4-carboxylate (**2ab**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.21 (m, 5H), 4.25-4.16 (m, 2H), 3.86-3.83 (d, *J* = 14.0 Hz, 1H), 3.72-3.69 (d, *J* = 14.0 Hz, 1H), 3.50-3.47 (m, 1H), 2.56-2.49 (m, 2H), 2.44-1.90 (m, 4H), 1.88-1.85 (m, 2H), 1.62-1.55 (m, 1H), 1.30-1.26 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.4, 167.1, 156.8, 153.5, 137.3, 129.0, 128.3, 126.5, 113.7, 106.8, 60.2, 41.0, 36.0, 33.3, 31.7, 21.3, 18.5, 14.2. IR (neat, cm^{-1}): 3394, 2938, 1740, 1681, 1163, 736, 702. Anal. Calcd for $C_{20}H_{22}O_4$: C 73.60; H

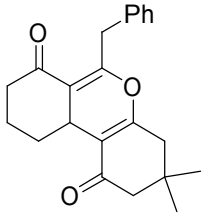
6.79. Found: C 73.69; H 6.69.



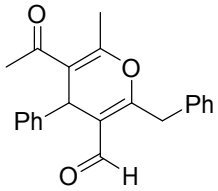
4-Acetyl-1-benzyl-4a,5,6,7-tetrahydro-3-methylisochromen-8-one (**2ac**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.21 (m, 5H), 3.88-3.84 (d, *J* = 14.0 Hz, 1H), 3.70-3.67 (d, *J* = 14.0 Hz, 1H), 3.56-3.53 (m, 1H), 2.58-2.37 (m, 2H), 2.24 (s, 3H), 1.99 (s, 3H), 1.95-1.86 (m, 3H), 1.64-1.58 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.9, 199.9, 153.8, 153.3, 137.1, 128.9, 128.3, 126.5, 115.7, 113.4, 41.0, 36.0, 33.8, 32.1, 29.2, 21.5, 18.3. IR (neat, cm^{-1}): 3384, 2931, 1717, 1678, 1176, 1083, 701. Anal. Calcd for $C_{19}H_{20}O_3$: C 77.00; H 6.80. Found: C 77.09; H 6.77.



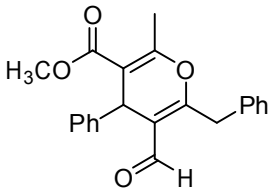
6-Benzyl-3,4,8,9,10,10a-hexahydro-2*H*-benzo[*c*]chromene-1,7-dione (**2ad**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.26 (m, 5H), 3.86-3.82 (d, *J* = 14.0 Hz, 1H), 3.74-3.70 (d, *J* = 14.0 Hz, 1H), 3.42-3.38 (m, 1H), 2.59-2.30 (m, 7H), 1.97-1.90 (m, 4H), 1.48-1.42 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.8, 197.6, 165.2, 152.8, 137.0, 129.0, 128.4, 126.6, 115.7, 114.1, 41.3, 37.1, 35.9, 31.0, 30.7, 27.1, 21.7, 20.1. IR (neat, cm^{-1}): 3306, 2947, 1699, 1657, 1170, 1133, 724. Anal. Calcd for $C_{20}H_{20}O_3$: C 77.90; H 6.54. Found: C 77.79; H 6.46.



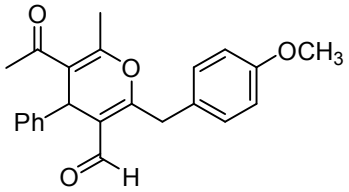
6-Benzyl-3,4,8,9,10,10a-hexahydro-3,3-dimethyl-2*H*-benzo[*c*]chromene-1,7-dione (**2ae**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.20 (m, 5H), 3.86 -3.82 (d, *J* = 14.0 Hz, 1H), 3.74-3.70 (d, *J* = 14.0 Hz, 1H), 3.43-3.39 (m, 1H), 2.59-2.52 (m, 2H), 2.43-2.37 (m, 1H), 2.36-2.18 (m, 4H), 1.96-1.88 (m, 2H), 1.48-1.43 (m, 1H), 1.01 (s, 3H), 1.00 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.8, 197.5, 163.4, 152.4, 137.1, 128.9, 128.3, 126.6, 115.6, 112.8, 51.0, 41.2, 40.7, 35.9, 31.7, 30.9, 30.5, 28.5, 27.9, 21.6. IR (neat, cm^{-1}): 3305, 2958, 1700, 1387, 1162, 913, 728. Anal. Calcd for $C_{22}H_{24}O_3$: C 78.54; H 7.19. Found: C 78.61; H 7.25.



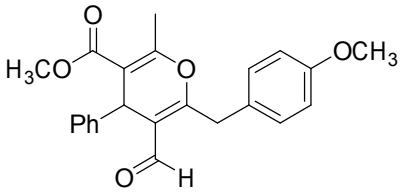
5-Acety-2-benzyl-6-methyl-4-phenyl-4*H*-pyran-3-carbaldehyde (**2ba**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.0, 7.34-7.22 (m, 10H), 4.86 (s, 1H), 4.05-4.02 (d, J = 15.2 Hz, 1H), 3.93-3.90 (d, J = 15.2 Hz, 1H), 2.34 (s, 3H), 2.15 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.9, 187.8, 164.4, 157.3, 143.3, 135.4, 128.8, 128.6, 128.4, 128.3, 127.3, 127.1, 118.8, 115.8, 35.9, 34.8, 29.6, 18.7. IR (neat, cm^{-1}): 3313, 3029, 1663, 1160, 949, 702. Anal. Calcd for $C_{22}H_{20}O_3$: C 79.50; H 6.06. Found: C 79.60; H 6.15.



Methyl 6-benzyl-5-formyl-2-methyl-4-phenyl-4*H*-pyran-3-carboxylate (**2bb**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98, 7.34-7.13 (m, 10H), 4.85 (s, 1H), 4.03-3.99 (d, J = 14.8 Hz, 1H), 3.95-3.91 (d, J = 14.8 Hz, 1H), 3.59 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.8, 166.5, 164.3, 144.1, 135.5, 128.7, 128.4, 128.1, 128.1, 127.2, 126.7, 118.4, 108.8, 51.4, 35.2, 34.7, 18.3. IR (neat, cm^{-1}): 3427, 1714, 1699, 1161, 1087, 700. Anal. Calcd for $C_{22}H_{20}O_4$: C 75.84; H 5.79. Found: C 75.80; H 5.71.

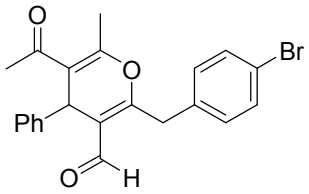


2-(4-Methoxybenzyl)-5-acetyl-6-methyl-4-phenyl-4*H*-pyran-3-carbaldehyde (**2ca**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.99 (s, 1H), 7.26-7.19 (m, 4H), 7.18-7.15 (dd, J = 4.0, 8.4 Hz, 1H), 7.13-7.11 (d, J = 8.8 Hz, 2H), 6.86-6.84 (d, J = 8.8 Hz, 2H), 4.84 (s, 1H), 3.97-3.93 (d, J = 14.8 Hz, 1H), 3.85-3.81 (d, J = 14.8 Hz, 1H), 3.79 (s, 3H), 2.32 (s, 3H), 2.13 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.9, 187.8, 164.8, 158.8, 157.2, 143.4, 129.4, 128.6, 128.3, 127.3, 127.0, 118.6, 115.8, 114.3, 55.2, 35.9, 34.0, 29.6, 18.7. IR (neat, cm^{-1}): 3420, 2928, 1660, 1249, 1158, 700. Anal. Calcd for $C_{23}H_{22}O_4$: C 76.22; H 6.12. Found: C 76.28; H 6.18.

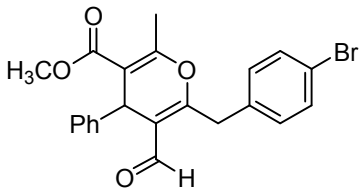


Methyl 6-(4-methoxybenzyl)-5-formyl-2-methyl-4-phenyl-4*H*-pyran-3-carboxylate (**2cb**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 1H), 7.24-7.22 (d, J = 6.0 Hz, 4H), 7.15-7.14 (d, J = 8.4 Hz, 3H), 6.86-6.84 (d, J = 8.4 Hz, 2H), 4.83 (s, 1H), 3.97-3.93 (m, 2H), 3.78 (s, 3H), 3.61 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.8,

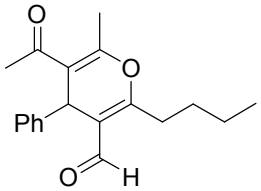
166.6, 164.8, 158.8, 158.6, 144.2, 129.4, 128.2, 128.1, 127.4, 126.6, 118.2, 114.2, 108.8, 55.2, 51.4, 35.2, 33.9, 18.4. IR (neat, cm^{-1}): 3410, 2951, 1714, 1669, 1246, 1158, 1036, 732. Anal. Calcd for $C_{23}H_{22}O_5$: C 73.00; H 5.86. Found: C 73.07; H 5.79.



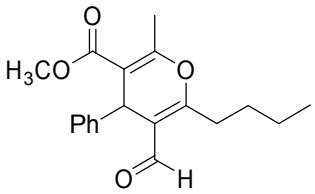
2-(4-Bromobenzyl)-5-acetyl-6-methyl-4-phenyl-4*H*-pyran-3-carbaldehyde (**2da**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (s, 1H), 7.46-7.43 (d, J = 8.4 Hz, 2H), 7.27-7.24 (m, 5H), 7.09-7.07 (d, J = 8.0 Hz, 2H), 4.84 (s, 1H), 3.97-3.94 (d, J = 15.2 Hz, 1H), 3.88-3.84 (d, J = 15.2 Hz, 1H), 2.31 (s, 1H), 2.13 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.7, 187.6, 163.6, 157.1, 143.2, 134.4, 131.9, 130.1, 128.6, 128.2, 127.2, 121.3, 118.9, 115.8, 35.9, 34.2, 29.6, 18.7. IR (neat, cm^{-1}): 3407, 2925, 1661, 1160, 1020, 800. Anal. Calcd for $C_{22}H_{19}BrO_3$: C 64.25; H 4.66. Found: C 64.28; H 4.58.



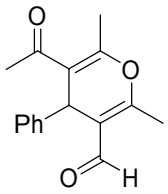
Methyl 6-(4-bromobenzyl)-5-formyl-2-methyl-4-phenyl-4*H*-pyran-3-carboxylate (**2db**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.95 (s, 1H), 7.47-7.44 (d, J = 8.4 Hz, 2H), 7.25-7.21 (m, 5H), 7.12-7.10 (d, J = 8.4 Hz, 2H), 4.84 (s, 1H), 3.99-3.89 (m ,2H), 3.62 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.7, 166.5, 163.5, 158.5, 144.0, 134.5, 131.9, 130.2, 128.2, 128.1, 126.8, 121.3, 118.7, 108.9, 51.5, 35.4, 34.2, 18.4. IR (neat, cm^{-1}): 3430, 2949, 1714, 1670, 1161, 1080, 733. Anal. Calcd for $C_{22}H_{19}BrO_4$: C 61.84; H 4.48. Found: C 64.75; H 4.41.



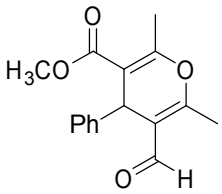
5-Acetyl-2-butyl-6-methyl-4-phenyl-4*H*-pyran-3-carbaldehyde (**2ea**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.83 (s, 1H), 7.26-7.17 (m, 5H), 4.80 (s, 1H), 2.69-2.58 (m, 2H), 2.39 (s, 3H), 2.14 (s, 3H), 1.66-1.60 (m, 2H), 1.39-1.34 (m, 2H), 0.94-0.91 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.0, 187.8, 167.1, 157.2, 143.6, 128.6, 128.2, 126.9, 118.3, 115.8, 35.8, 29.9, 29.6, 28.6, 22.1, 18.8, 13.7. IR (neat, cm^{-1}): 3407, 2929, 1661, 1176, 1030, 747. Anal. Calcd for $C_{19}H_{22}O_3$: C 76.48; H 7.43. Found: C 76.59; H 7.49.



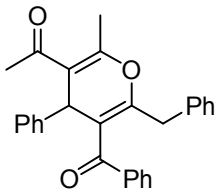
Methyl 6-butyl-5-formyl-2-methyl-4-phenyl-4*H*-pyran-3-carboxylate (**2eb**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.83 (s, 1H), 7.26-7.13 (m, 5H), 4.79 (s, 1H), 3.63 (s, 3H), 2.70-2.65 (m, 2H), 2.42 (s, 3H), 1.70-1.63 (m, 2H), 1.43-1.36 (m, 2H), 0.96-0.92 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.8, 167.1, 166.8, 158.5, 144.4, 128.1, 128.1, 126.6, 118.9, 51.5, 35.1, 29.9, 28.5, 22.1, 18.4, 13.7. IR (neat, cm^{-1}): 3399, 2956, 1715, 1669, 1175, 1039, 698. Anal. Calcd for $C_{19}H_{22}O_3$: C 76.48; H 7.43. Found: C 76.58; H 7.36.



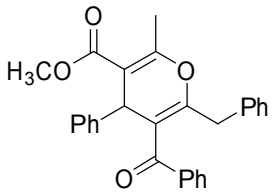
5-Acetyl-2,6-dimethyl-4-phenyl-4*H*-pyran-3-carbaldehyde (**2fa**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.83 (s, 1H), 7.27-7.16 (m, 5H), 4.79 (s, 1H), 2.39 (s, 3H), 2.29 (s, 3H), 2.13 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.1, 188.1, 163.3, 157.1, 143.5, 128.6, 128.3, 127.0, 118.3, 115.8, 35.8, 29.6, 18.9, 15.4. IR (neat, cm^{-1}): 3433, 2925, 1660, 1194, 1022, 700. Anal. Calcd for $C_{16}H_{16}O_3$: C 74.98; H 6.29. Found: C 74.88; H 6.37.



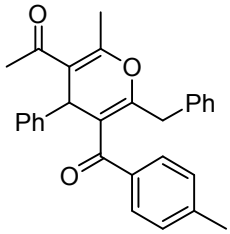
Methyl 5-formyl-2,6-dimethyl-4-phenyl-4*H*-pyran-3-carboxylate (**2fb**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.82 (s, 1H), 7.26-7.12 (m, 5H), 4.79 (s, 1H), 3.62 (s, 3H), 2.42 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.1, 166.7, 163.3, 158.3, 144.3, 128.1, 126.6, 117.9, 108.9, 51.4, 35.1, 18.5, 15.3. IR (neat, cm^{-1}): 3416, 2925, 1714, 1670, 1192, 1021, 699. Anal. Calcd for $C_{16}H_{16}O_4$: C 70.57; H 5.92. Found: C 70.68; H 5.84.



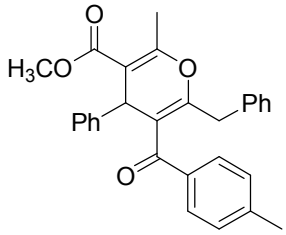
5-Acety-2-benzyl-6-methyl-4-phenyl-4*H*-pyran-3-benzaldehyde (**2ga**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56-7.46 (m, 3H), 7.34-7.30 (t, J = 7.6 Hz, 2H), 7.24-7.05 (m, 10 H), 4.92 (s, 1H), 3.42-3.29 (m, 2H), 2.30 (s, 3H), 2.06 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.9, 197.2, 158.2, 150.6, 143.3, 138.4, 136.4, 132.9, 128.9, 128.7, 128.6, 128.4, 127.7, 127.2, 126.7, 117.2, 113.8, 41.9, 37.1, 29.6, 19.1. IR (neat, cm^{-1}): 3397. 2961, 1693, 1598, 1209, 1026, 699. Anal. Calcd for $C_{28}H_{24}O_3$: C 82.33; H 5.92. Found: C 82.27; H 5.88.



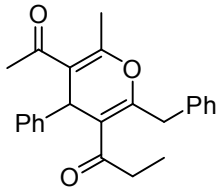
Methyl 6-benzyl-5-benzoyl-2-methyl-4-phenyl-4*H*-pyran-3-carboxylate (**2gb**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60-7.58 (d, J = 8.4 Hz, 2H), 7.48-7.46 (d, J = 7.6 Hz, 1H), 7.36-7.32 (m, 2H), 7.25-7.07 (m, 10H), 4.86 (s, 1H), 3.55 (s, 3H), 3.49-3.38 (m, 2H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.9, 167.1, 159.7, 151.1, 143.9, 138.1, 136.6, 132.8, 128.8, 128.6, 128.5, 128.4, 128.3, 127.7, 126.8, 126.6, 117.1, 106.5, 51.3, 41.1, 37.0, 18.7. IR (neat, cm^{-1}): 3400, 3028, 1714, 1164, 1088, 698. Anal. Calcd for $C_{23}H_{24}O_4$: C 79.22; H 5.70. Found: C 79.16; H 5.66.



5-Acety-2-benzyl-6-methyl-4-phenyl-4*H*-pyran-3-(4-methybenzaldehyde) (**2ha**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.45 (d, J = 8.0 Hz, 2H), 7.25-7.18 (m, 6H), 7.15-7.05 (m, 6H), 4.91 (s, 1H), 3.43-3.28 (m, 2H), 2.35 (s, 3H), 2.29 (s, 3H), 2.06 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.9, 196.8, 158.3, 149.9, 143.9, 143.3, 136.5, 135.7, 129.3, 129.0, 128.8, 128.6, 128.3, 127.7, 127.1, 126.6, 117.2, 113.7, 41.9, 37.1, 29.6, 21.6, 19.1. IR (neat, cm^{-1}): 3434, 2924, 1602, 1209, 1171, 1133, 737. Anal. Calcd for $C_{29}H_{26}O_3$: C 82.44; H 6.20. Found: C 82.38; H 6.14.

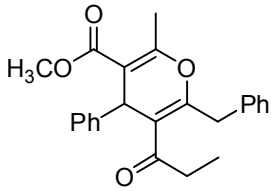


Methyl 6-benzyl-5-(4-methybenzoyl)-2-methyl-4-phenyl-4*H*-pyran-3-carboxylate (**2hb**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53-7.51 (d, J = 8.0 Hz, 2H), 7.24-7.08 (m, 12H), 4.85 (s, 1H), 3.55 (s, 3H), 3.49-3.36 (m, 2H), 2.35 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.6, 167.2, 159.8, 150.4, 143.9, 143.8, 136.7, 135.5, 129.2, 129.1, 128.7, 128.4, 128.3, 127.7, 126.9, 126.6, 117.2, 106.4, 51.2, 41.2, 37.0, 21.6, 18.7. IR (neat, cm^{-1}): 3409, 2981, 1685, 1179, 1044, 758. Anal. Calcd for $C_{29}H_{26}O_4$: C 79.43; H 5.98. Found: C 79.49; H 5.91.

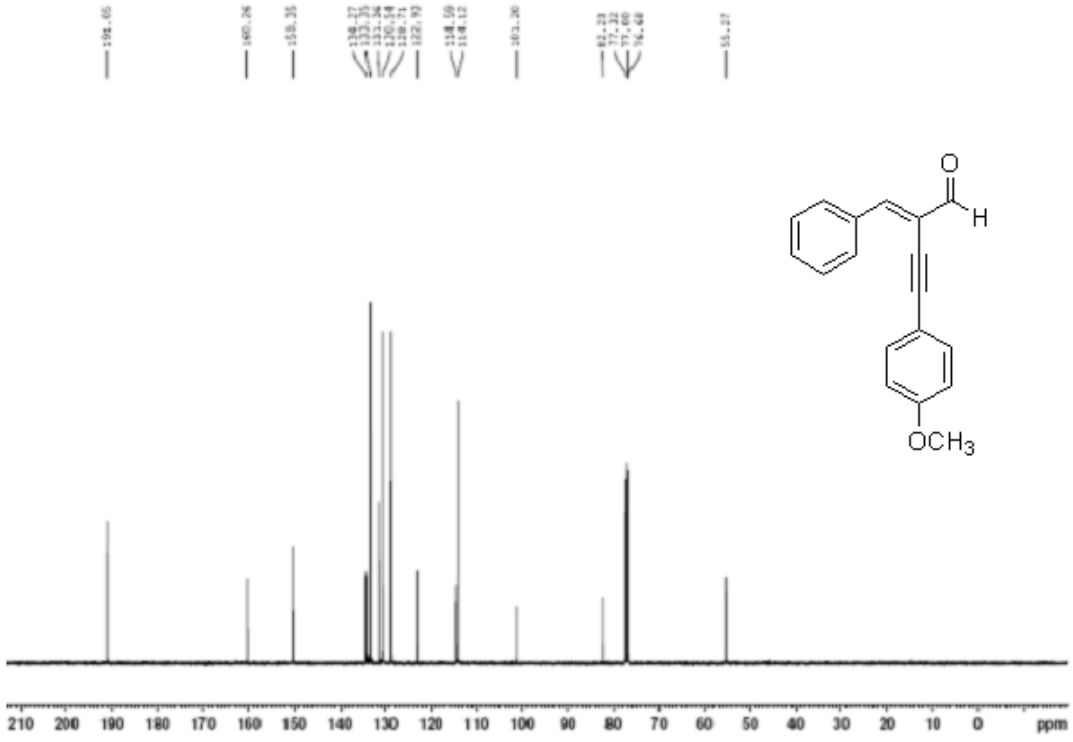


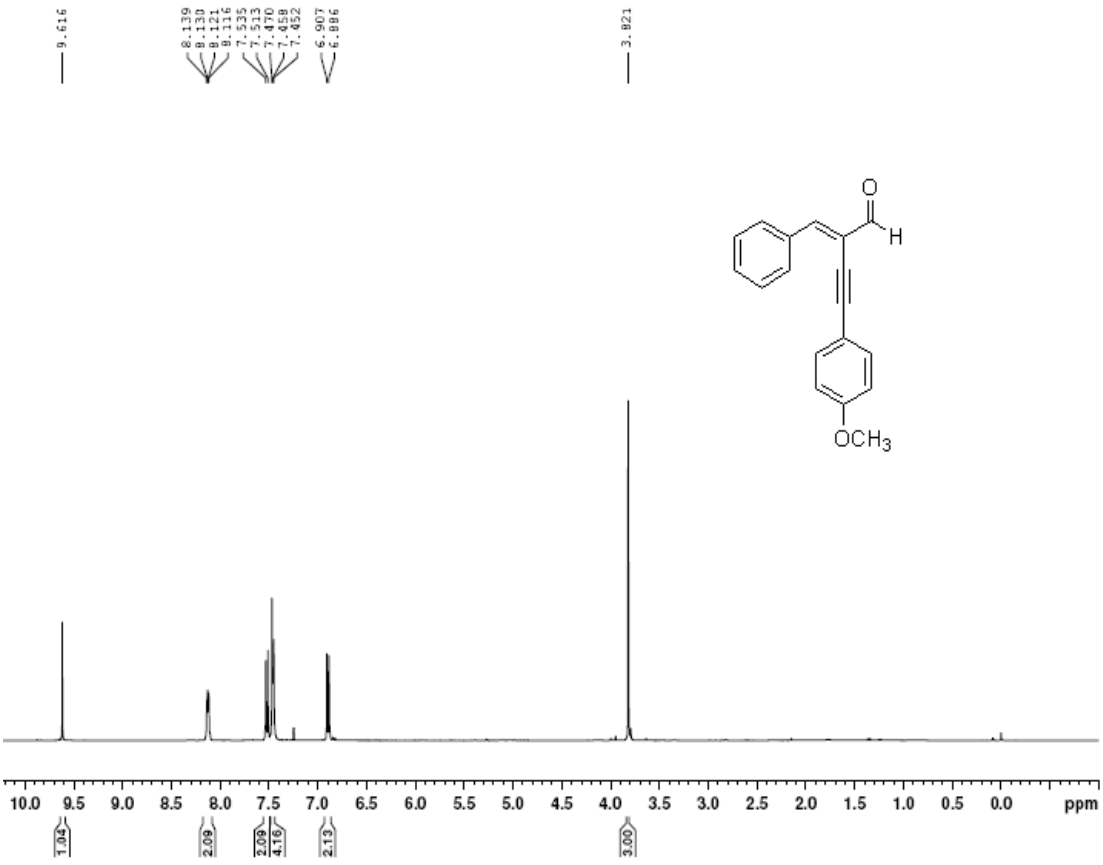
5-Acety-2-benzyl-6-methyl-4-phenyl-4*H*-pyran-3-propionaldehyde (**2ia**). ^{1}H NMR

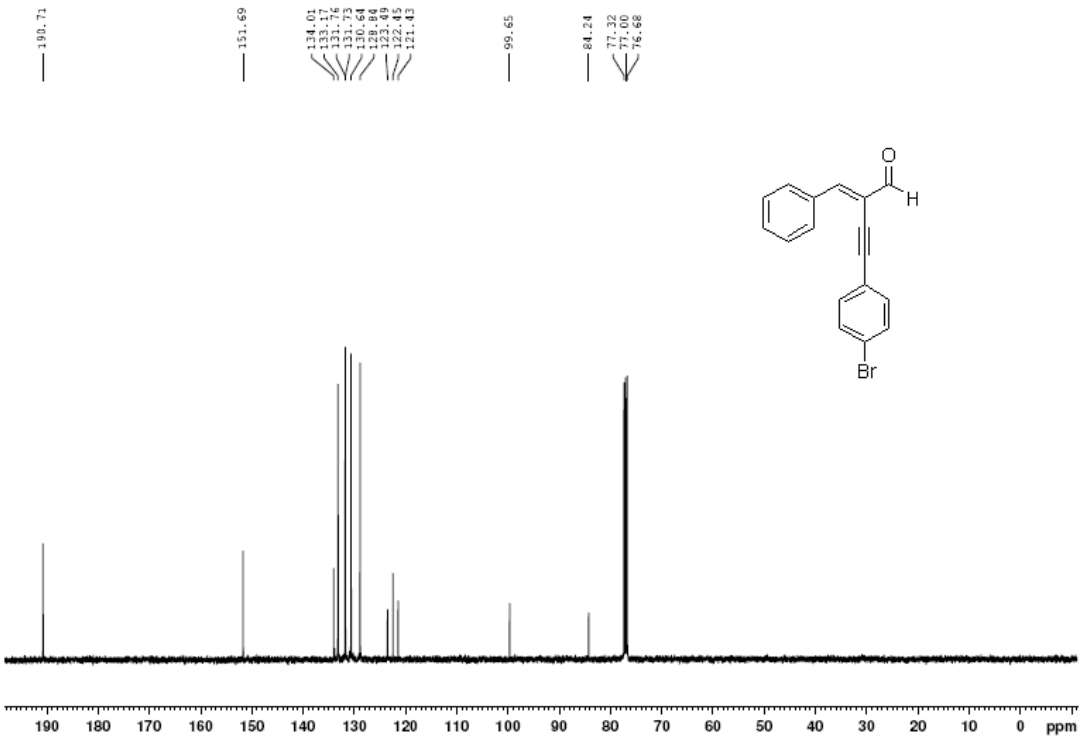
(400 MHz, $CDCl_3$) δ 7.31-7.18 (m, 10H), 4.85 (s, 1H), 4.03-3.92 (m, 2H), 2.60-2.53 (m, 2H), 2.45-2.39 (m, 3H), 2.22-2.21 (d, J = 7.2 Hz, 3H), 0.98-0.94 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.1, 198.0, 157.1, 156.0, 144.1, 137.0, 128.9, 128.8, 128.7, 128.5, 128.0, 127.1, 126.7, 117.2, 39.2, 37.1, 34.8, 30.6, 19.4, 7.9. IR (neat, cm^{-1}): 3432, 2927, 1689, 1597, 1183, 1027, 701. Anal. Calcd for $C_{24}H_{24}O_3$: C 79.97; H 6.71. Found: C 79.90; H 6.80.

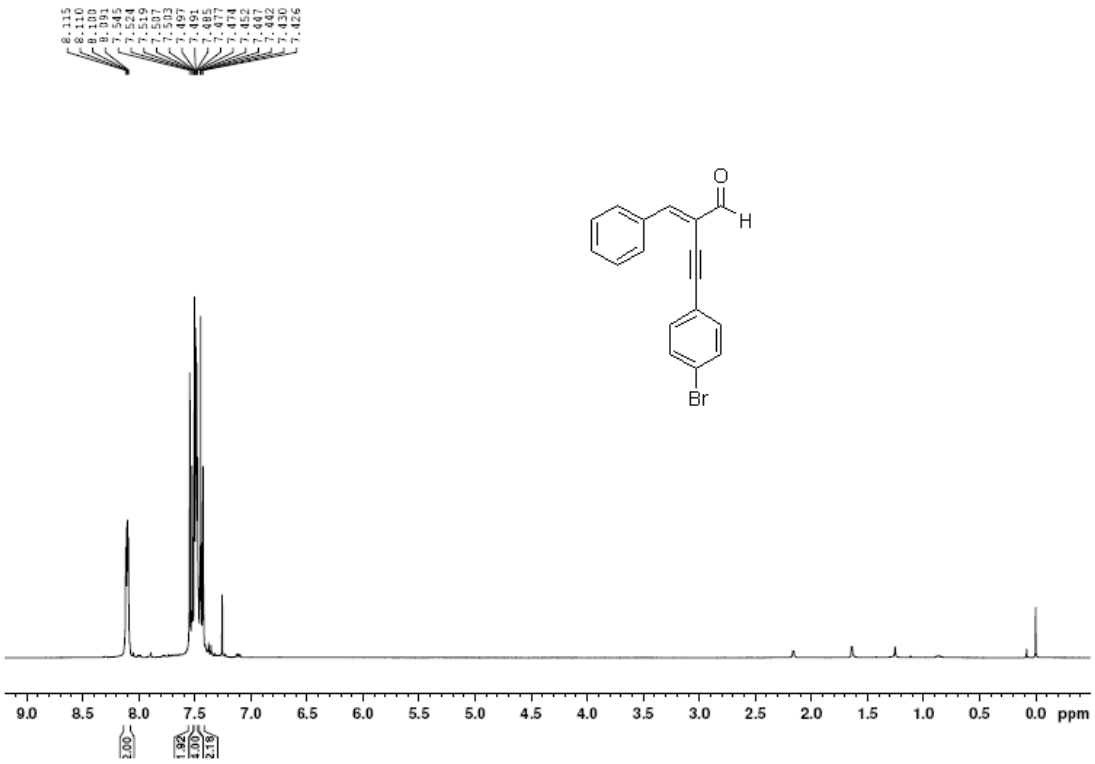


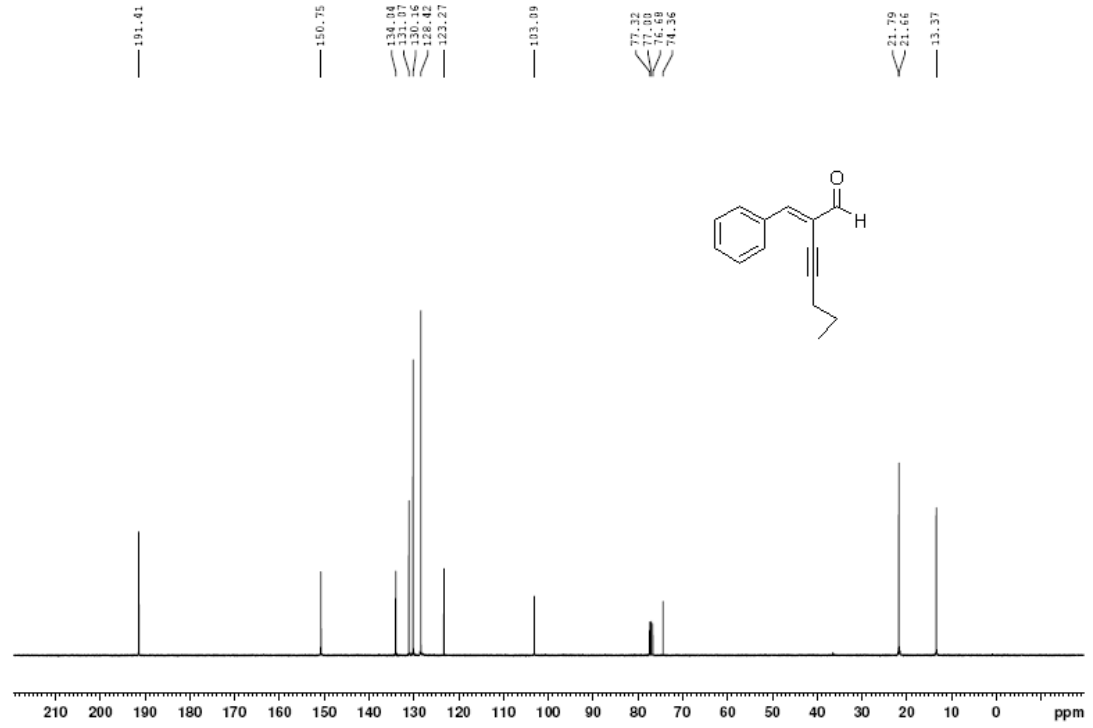
Methyl 6-benzyl-5-propionyl-2-methyl-4-phenyl-4*H*-pyran-3-carboxylate (**2ib**). ^1H NMR (400 MHz, $CDCl_3$) δ 7.36-7.17 (m, 10H), 4.81 (s, 1H), 4.07-3.98 (m, 2H), 3.67 (s, 3H), 2.65-2.55 (m, 1H), 2.42-2.32 (m, 1H), 2.25 (s, 3H), 0.97-0.94 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.9, 167.1, 158.7, 156.9, 144.4, 137.3, 128.9, 128.6, 128.4, 128.1, 127.0, 126.6, 116.2, 108.3, 51.4, 38.9, 37.0, 34.4, 18.8, 7.85. IR (neat, cm^{-1}): 3406, 2942, 1697, 1603, 1186, 1076, 701. Anal. Calcd for $C_{24}H_{24}O_4$: C 76.57; H 6.43. Found: C 76.66; H 6.50.

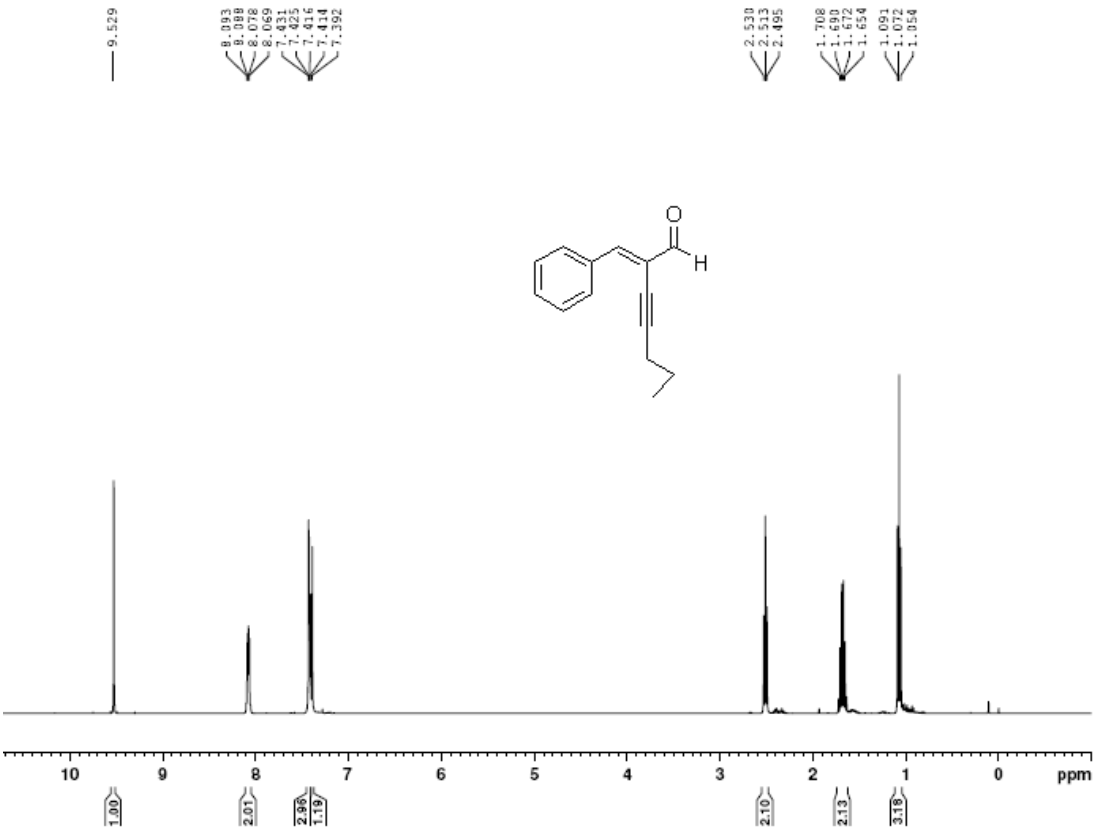

191.65
160.26
158.35
138.27
133.35
132.54
129.84
129.71
122.93
114.59
114.12
103.20
82.23
77.32
77.00
76.68
55.27
O
H
OCH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

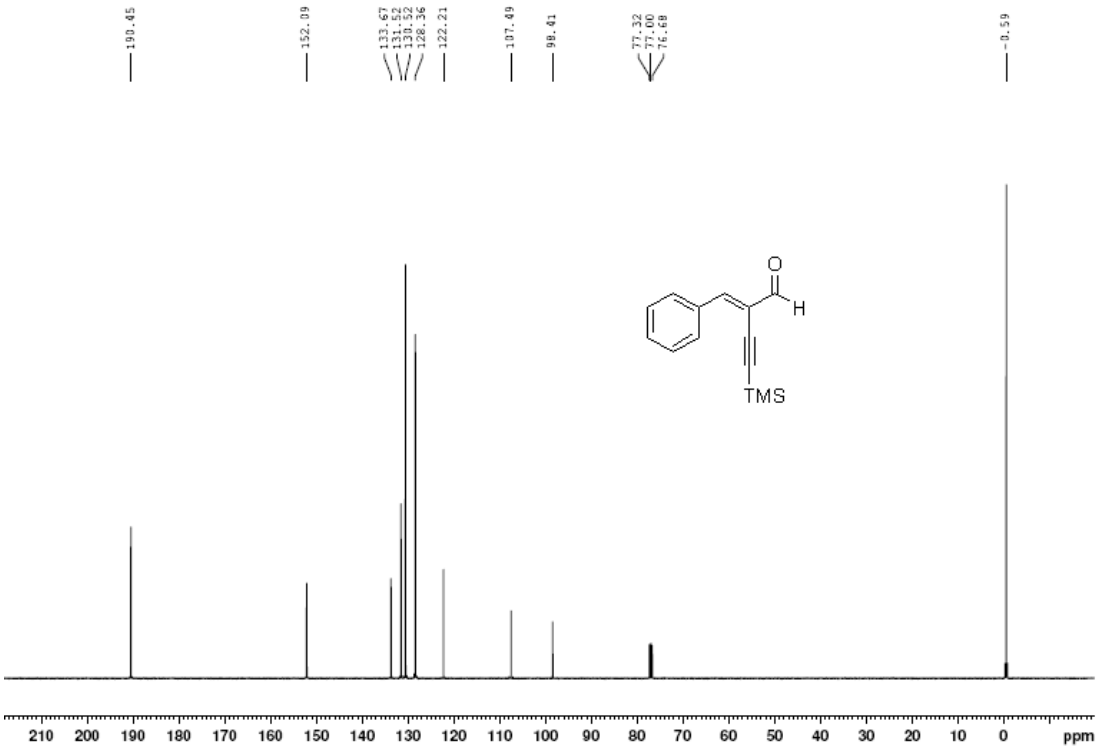

9.616
8.139
8.130
8.121
8.116
7.535
7.513
7.470
7.459
7.452
6.907
6.886
3.821
O
H
OCH3
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.04
2.09
2.09
4.16
2.13
3.00

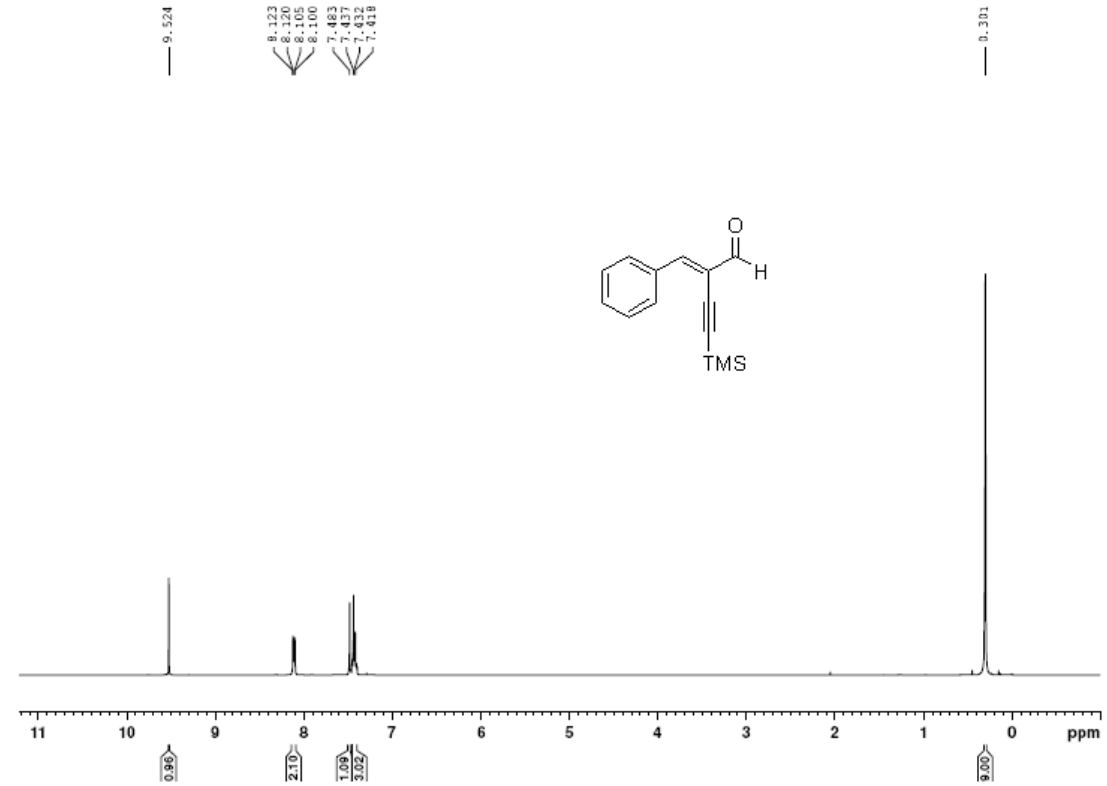

190.71
151.69
134.01
133.17
131.76
131.73
130.64
129.84
129.49
122.45
121.43
99.65
84.24
77.32
77.00
76.68
O
H
Br
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

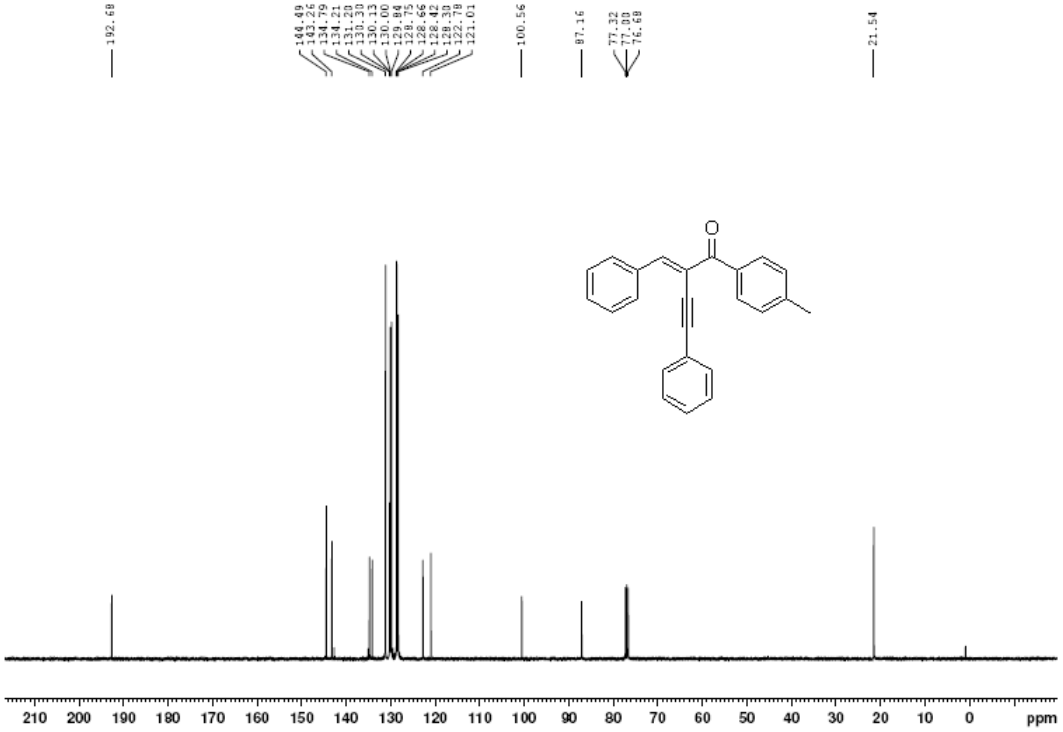

8.115
8.110
8.103
8.101
7.545
7.524
7.519
7.507
7.503
7.497
7.491
7.485
7.477
7.474
7.452
7.447
7.442
7.430
7.426
O
H
Br
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
1.92
1.00
2.18

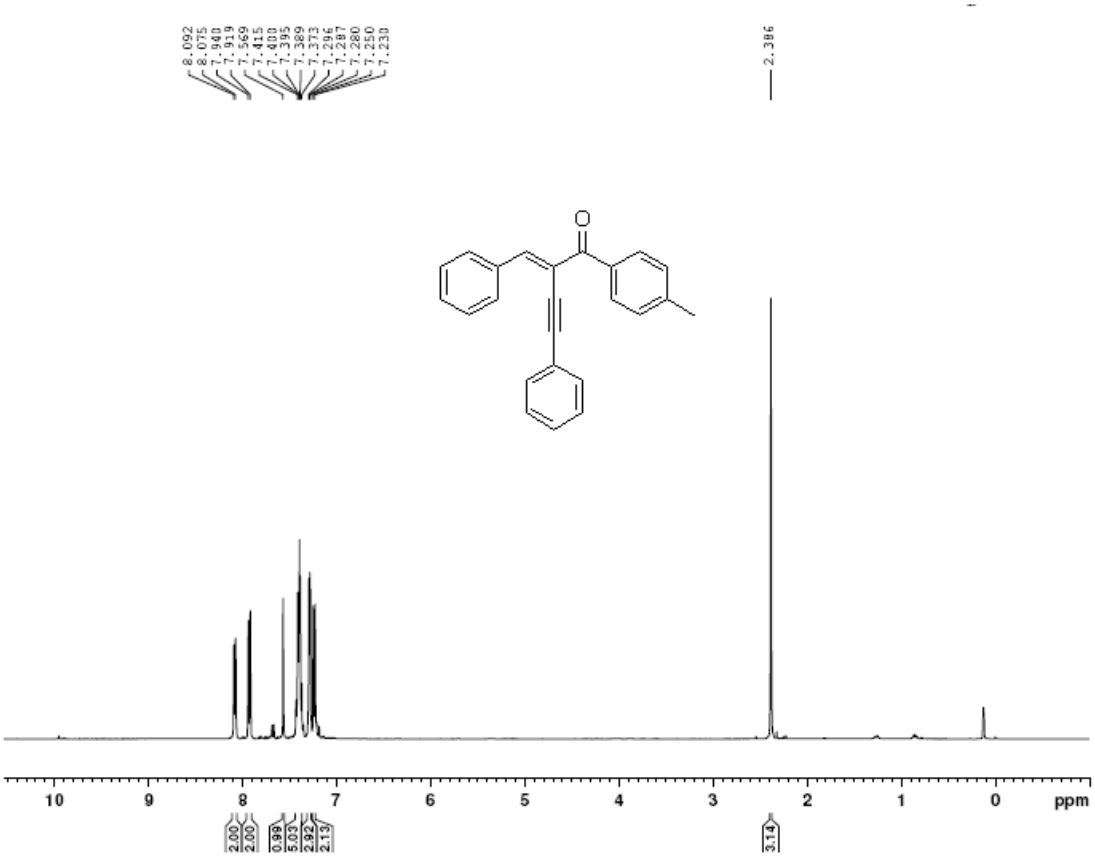

191.41
150.75
134.04
131.07
130.16
128.42
123.27
103.09
77.32
77.00
76.68
74.36
21.79
21.66
13.37
O
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

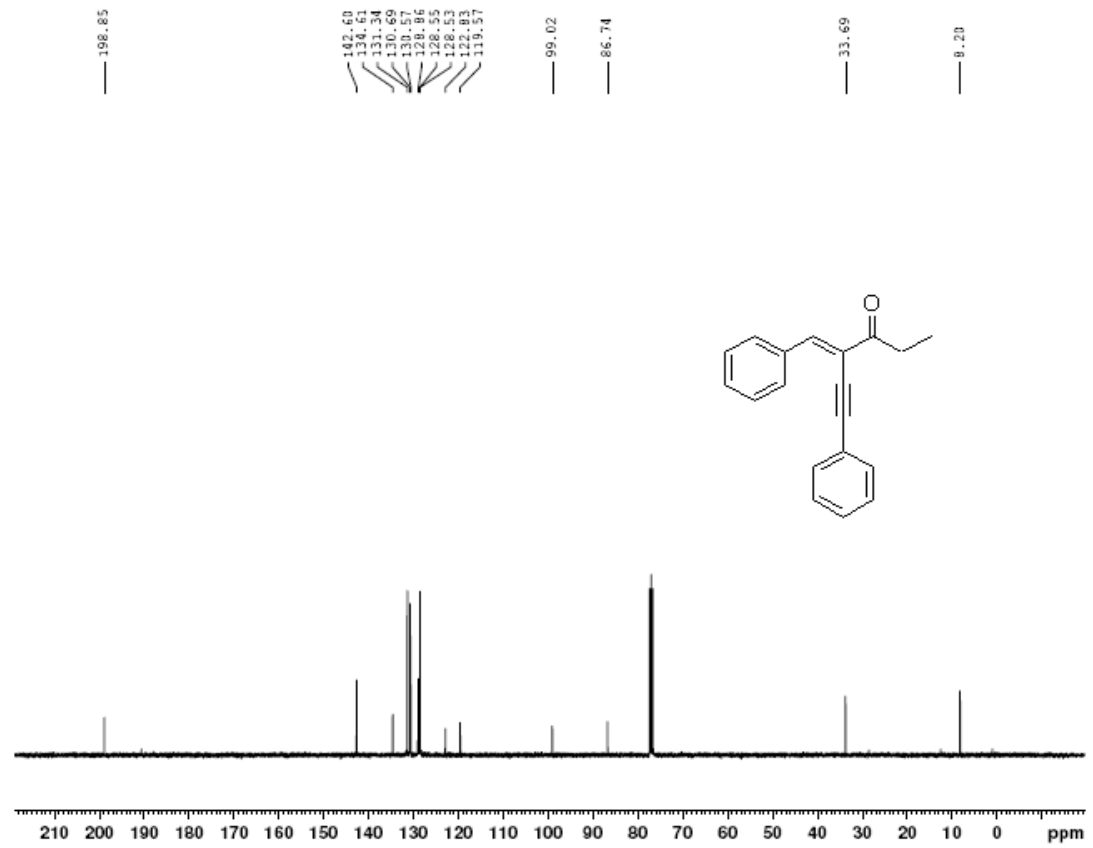

9.529
8.093
8.089
8.078
8.069
7.431
7.425
7.416
7.414
7.392
2.530
2.513
2.495
1.708
1.690
1.672
1.654
1.091
1.072
1.054
O
H
10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
2.01
2.96
1.19
2.10
2.13
3.16

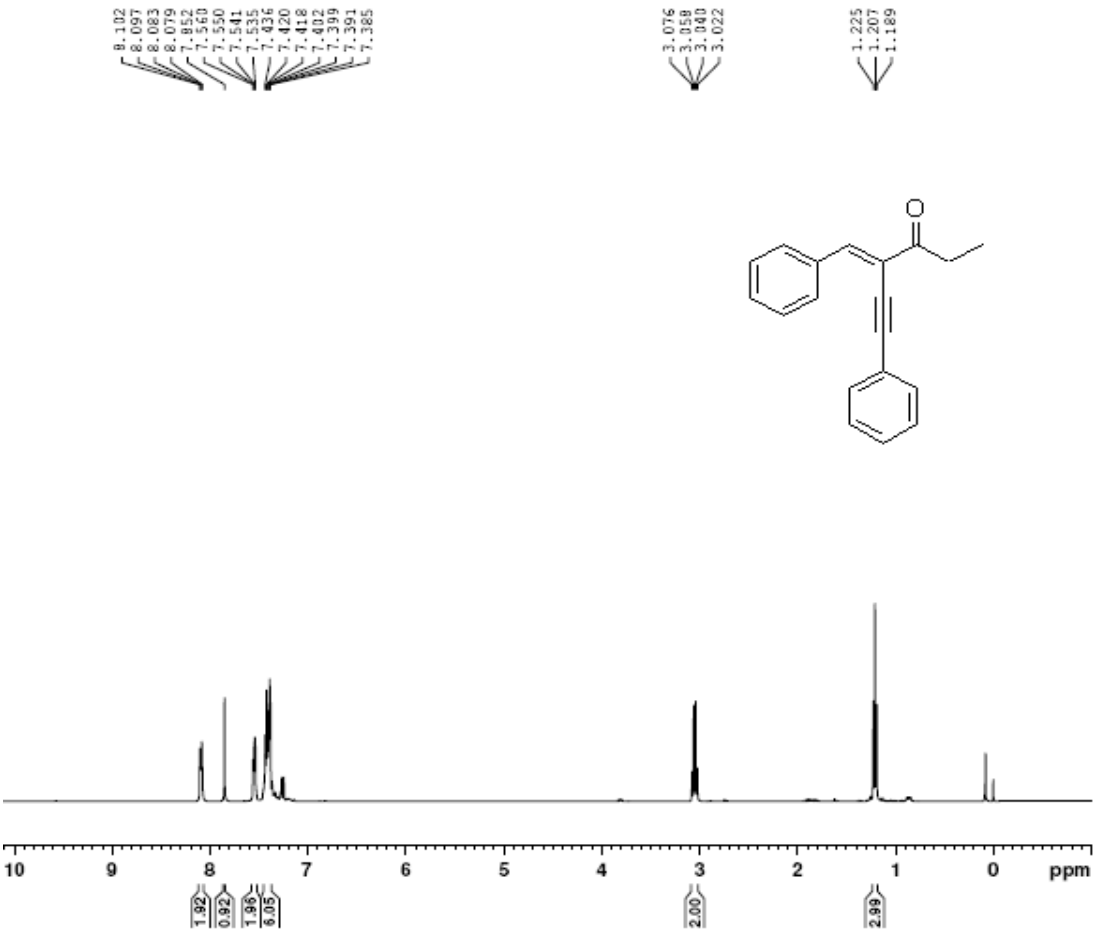

190.45
152.09
133.67
131.52
130.52
128.36
122.21
107.49
98.41
77.32
77.00
76.68
-0.59
O
H
TMS
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

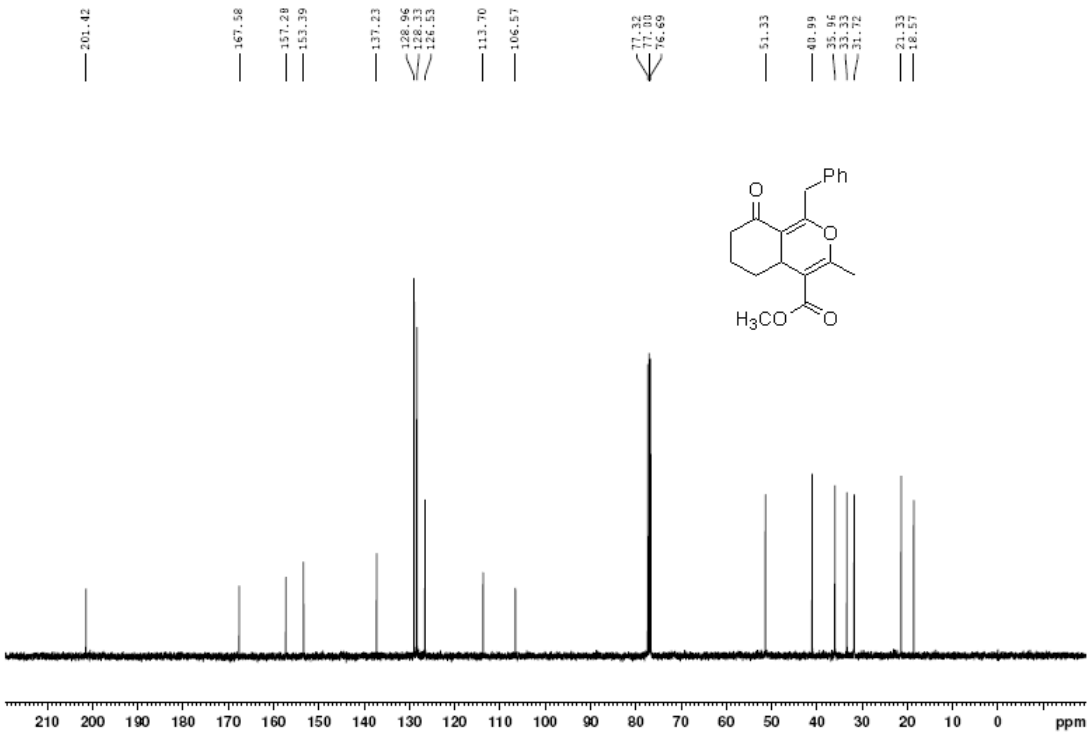

9.524
8.123
8.120
8.105
8.100
7.483
7.437
7.432
7.418
0.301
O
H
TMS
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.96
2.10
1.09
3.02
9.00

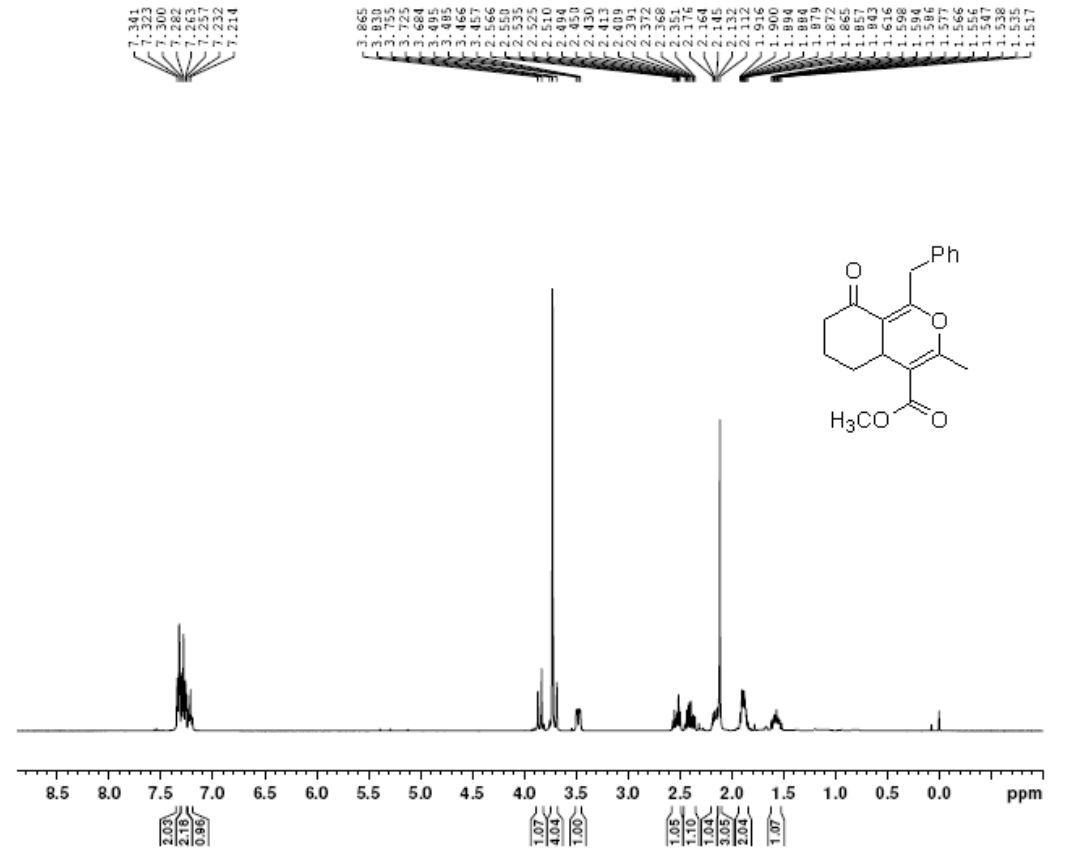

192.68
144.49
143.26
134.79
134.21
131.30
131.03
130.13
130.00
129.84
128.75
128.66
128.42
128.30
122.78
121.01
100.56
87.16
77.32
77.00
76.68
21.54
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

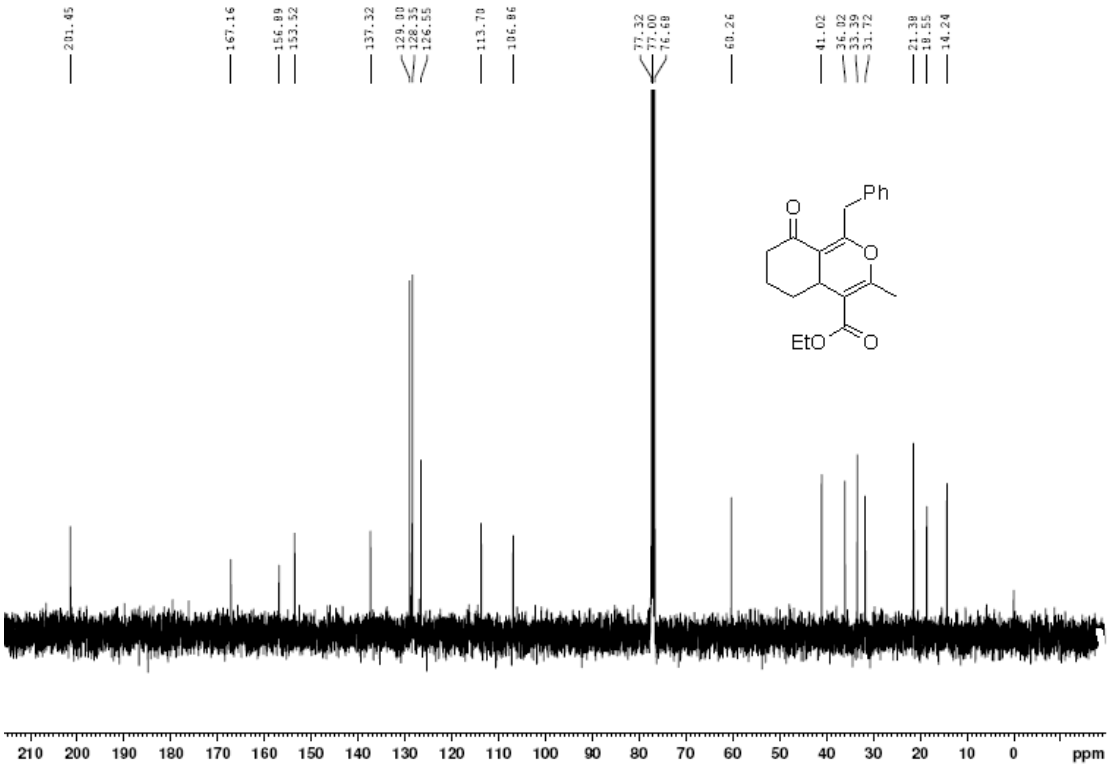

8.092
8.075
7.940
7.919
7.569
7.415
7.400
7.395
7.389
7.373
7.296
7.287
7.280
7.250
7.230
2.386
O
10 9 8 7 6 5 4 3 2 1 0 ppm
2.00
2.00
0.99
5.03
2.92
2.13
3.14

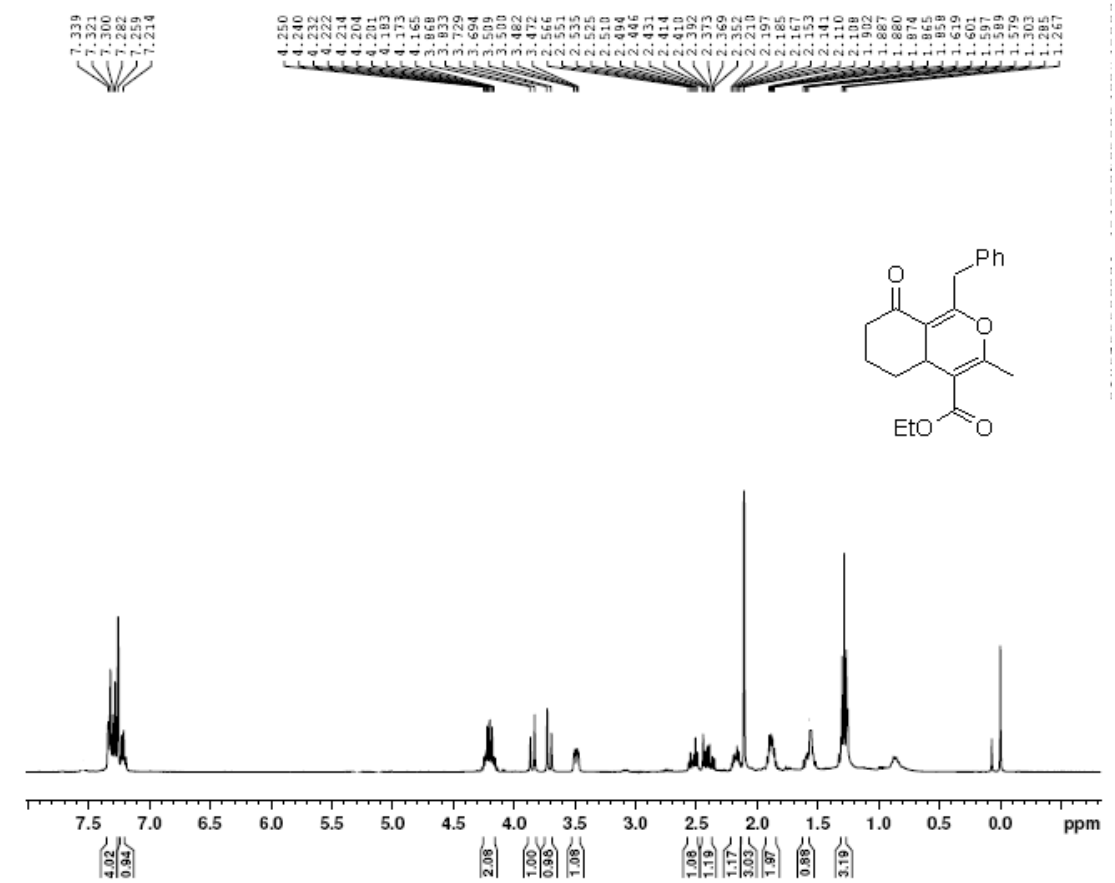

198.85
142.60
134.61
131.34
130.69
130.57
128.86
128.55
128.53
122.83
119.57
99.02
86.74
33.69
8.28
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

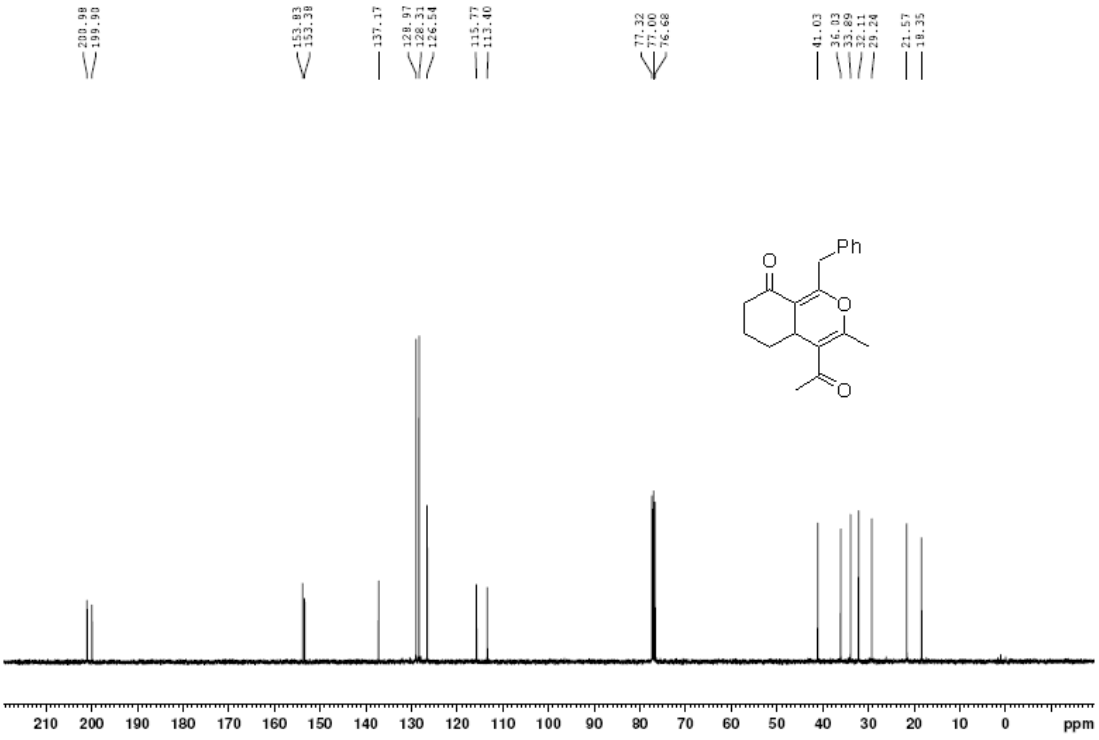

8.102
8.097
8.083
8.079
7.852
7.560
7.550
7.541
7.535
7.436
7.420
7.418
7.402
7.399
7.391
7.385
3.076
3.058
3.040
3.022
1.225
1.207
1.189
O
10 9 8 7 6 5 4 3 2 1 0 ppm
1.92
0.92
1.96
6.05
2.00
2.99

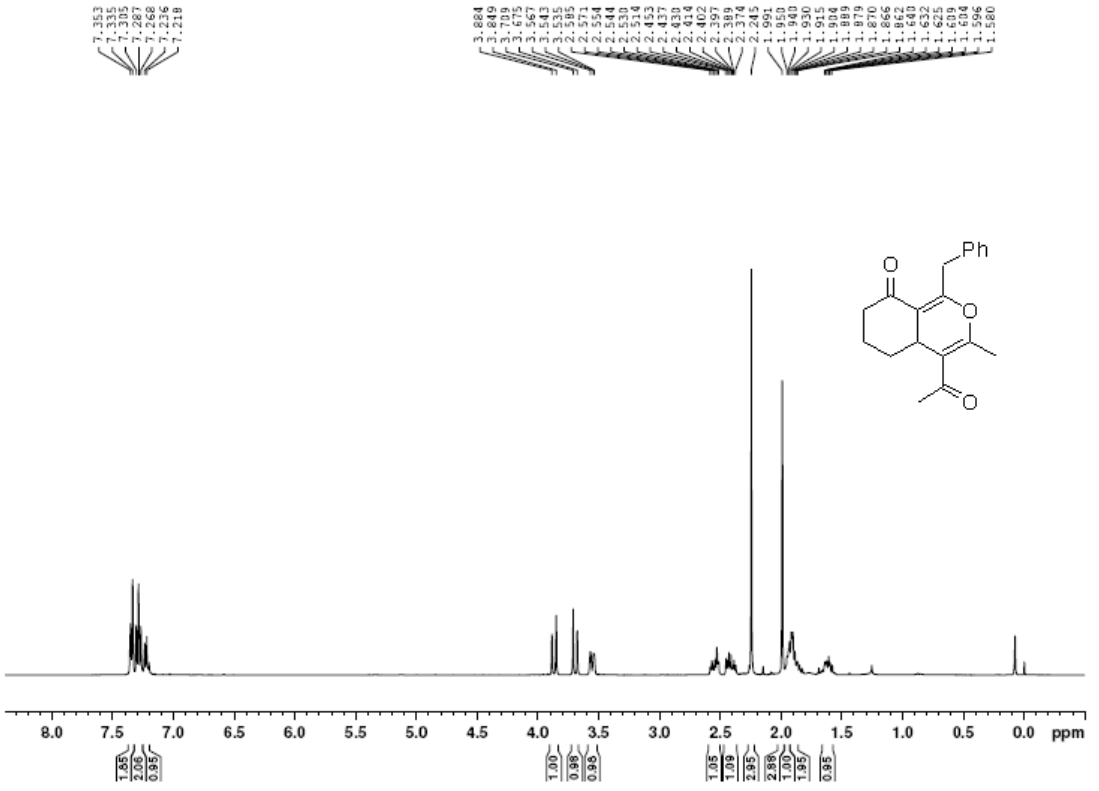

201.42
167.58
157.28
153.39
137.23
128.96
128.33
126.53
113.70
106.57
77.32
77.00
76.69
51.33
40.99
35.96
33.33
31.72
21.33
18.57
Ph
O
O
H3CO
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

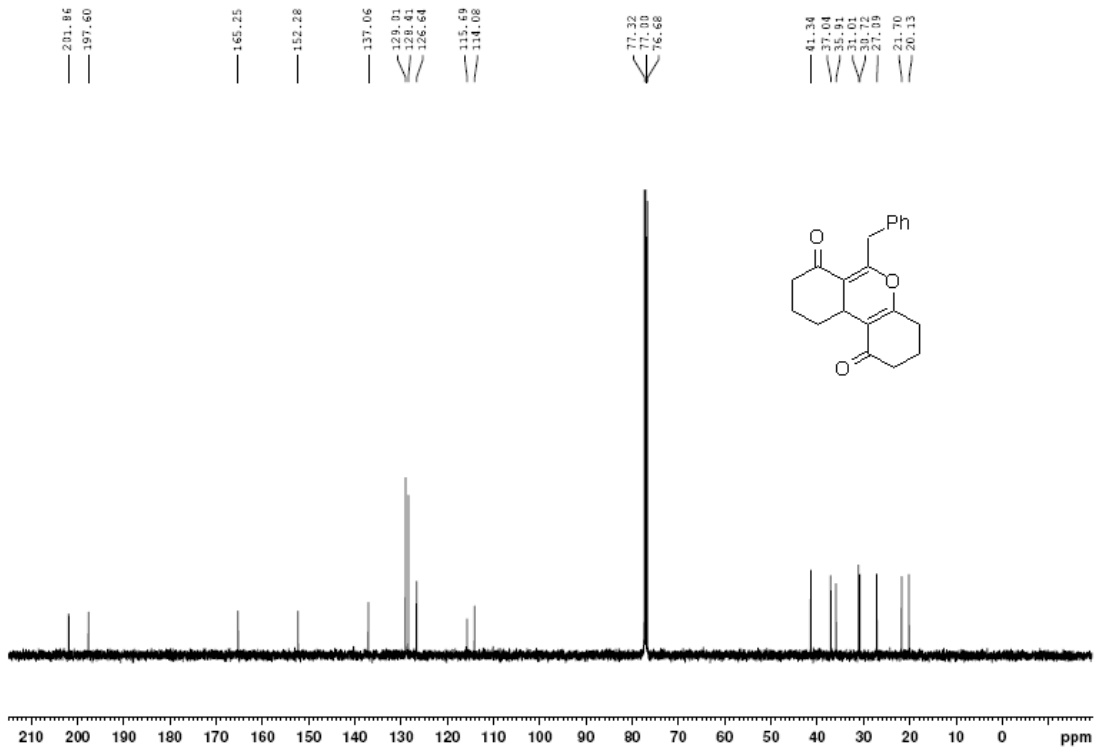

7.341
7.323
7.300
7.282
7.263
7.257
7.232
7.214
3.865
3.830
3.755
3.725
3.684
3.495
3.485
3.466
3.457
2.566
2.550
2.535
2.525
2.510
2.494
2.450
2.430
2.413
2.409
2.391
2.372
2.368
2.351
2.176
2.164
2.145
2.132
2.112
1.916
1.900
1.894
1.884
1.879
1.872
1.865
1.857
1.843
1.616
1.598
1.594
1.586
1.577
1.566
1.556
1.547
1.538
1.535
1.517
Ph
O
O
H3CO
O
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
2.18
0.96
1.07
4.04
1.00
1.05
1.10
1.04
3.05
2.04
1.07

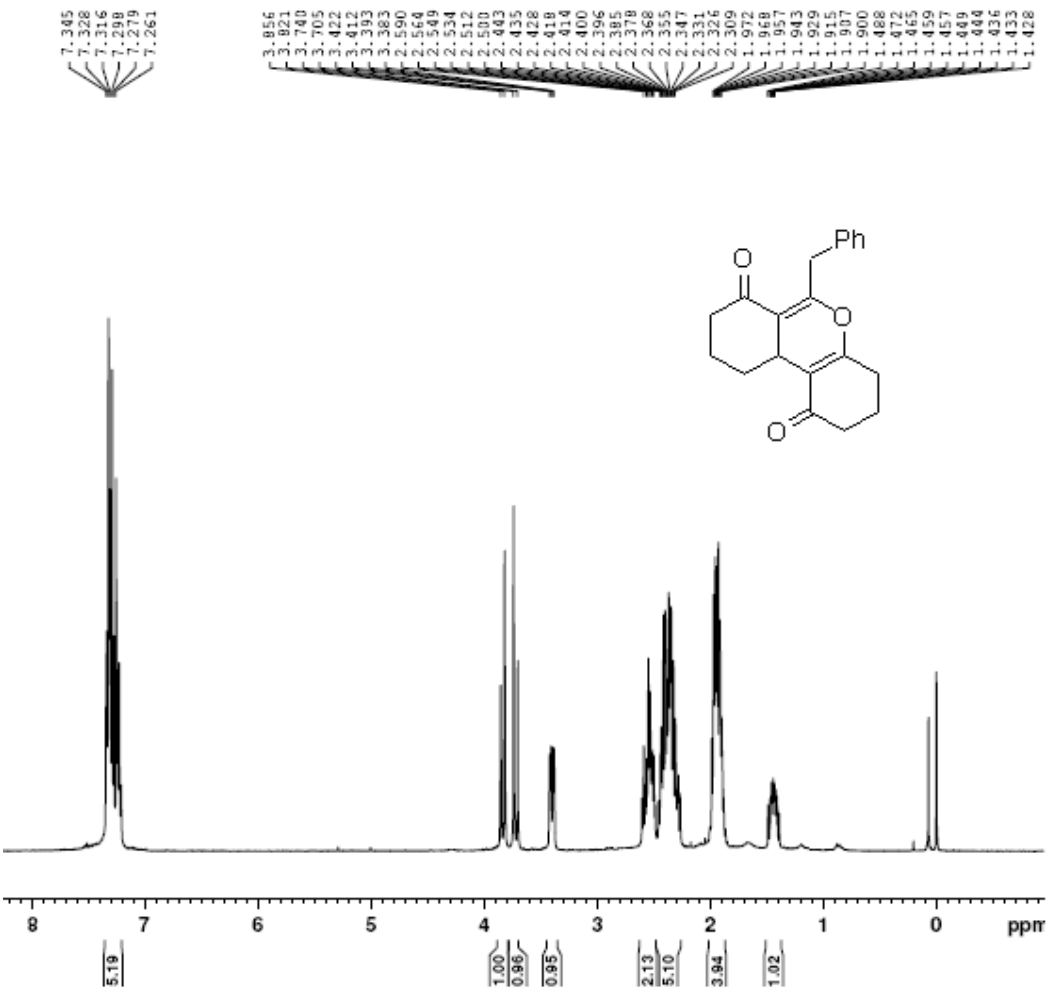

201.45
167.16
156.89
153.52
137.32
129.00
128.35
126.55
113.78
106.86
77.32
77.00
76.68
60.26
41.02
36.02
33.39
31.72
21.38
18.55
14.24
Ph
O
O
EtO
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

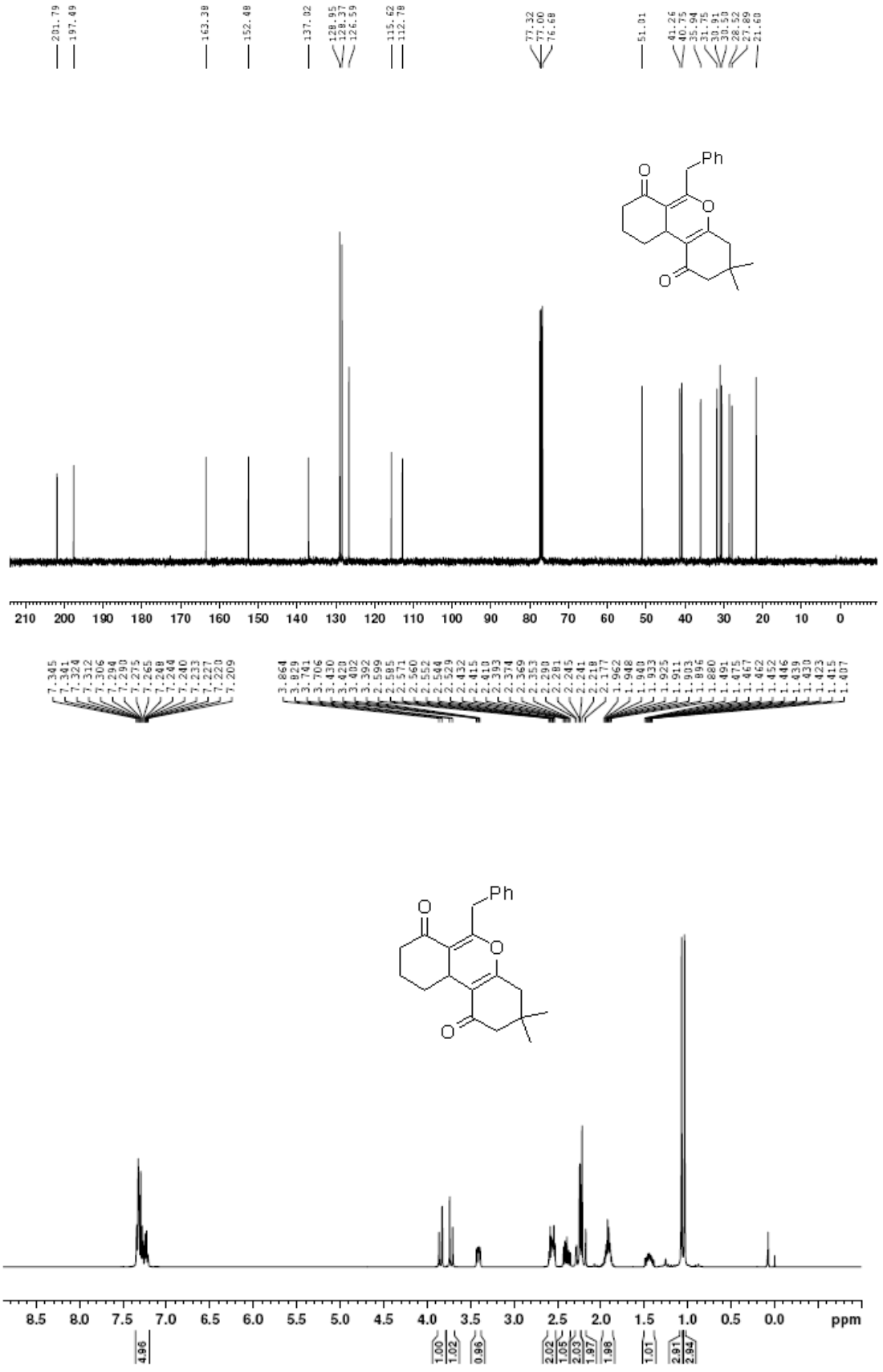

7.339
7.321
7.300
7.282
7.259
7.214
4.250
4.240
4.232
4.222
4.214
4.204
4.201
4.183
4.173
4.165
3.868
3.833
3.729
3.694
3.509
3.500
3.482
3.472
2.566
2.551
2.535
2.525
2.510
2.494
2.466
2.431
2.414
2.410
2.392
2.373
2.369
2.352
2.210
2.197
2.185
2.167
2.153
2.141
2.110
2.108
1.902
1.887
1.880
1.874
1.865
1.858
1.619
1.601
1.597
1.589
1.579
1.303
1.285
1.267
Ph
O
O
EtO
O
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
4.02
0.94
2.08
1.00
0.96
1.08
1.08
1.19
1.17
3.03
1.97
0.88
3.19

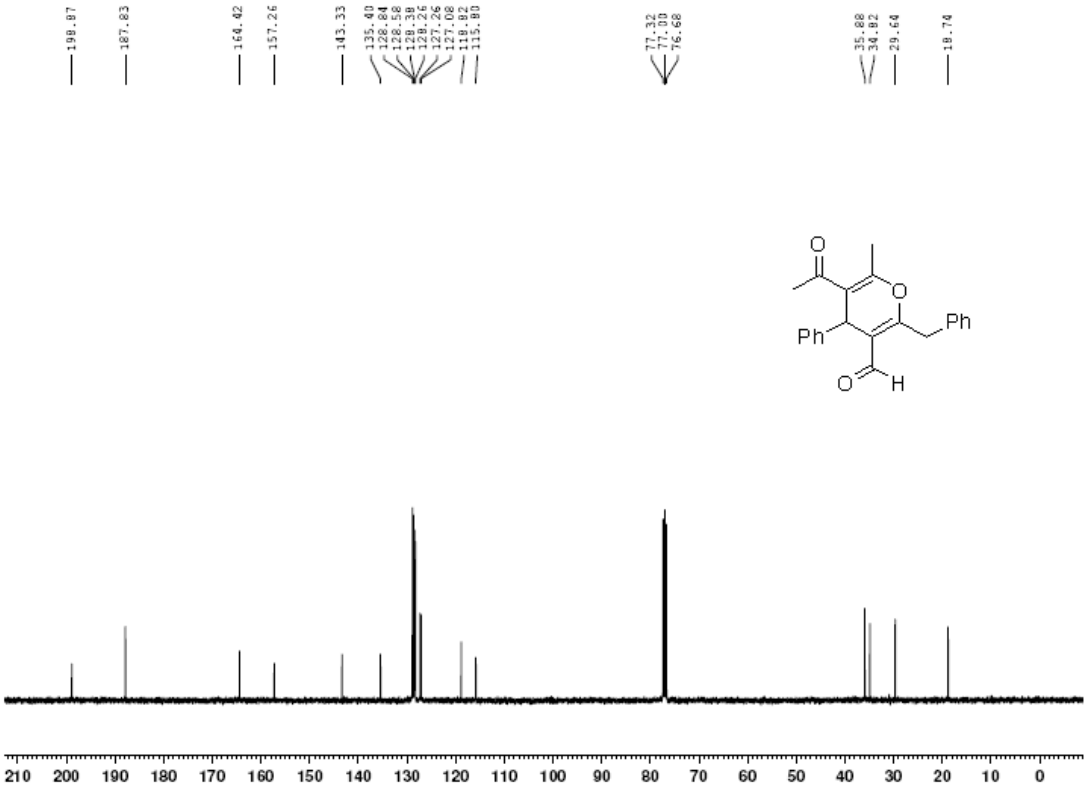

209.98
199.99
153.83
153.38
137.17
128.97
128.71
126.54
115.77
113.40
77.32
77.00
76.68
41.03
36.03
33.89
32.11
29.24
21.57
18.35
Ph
O
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

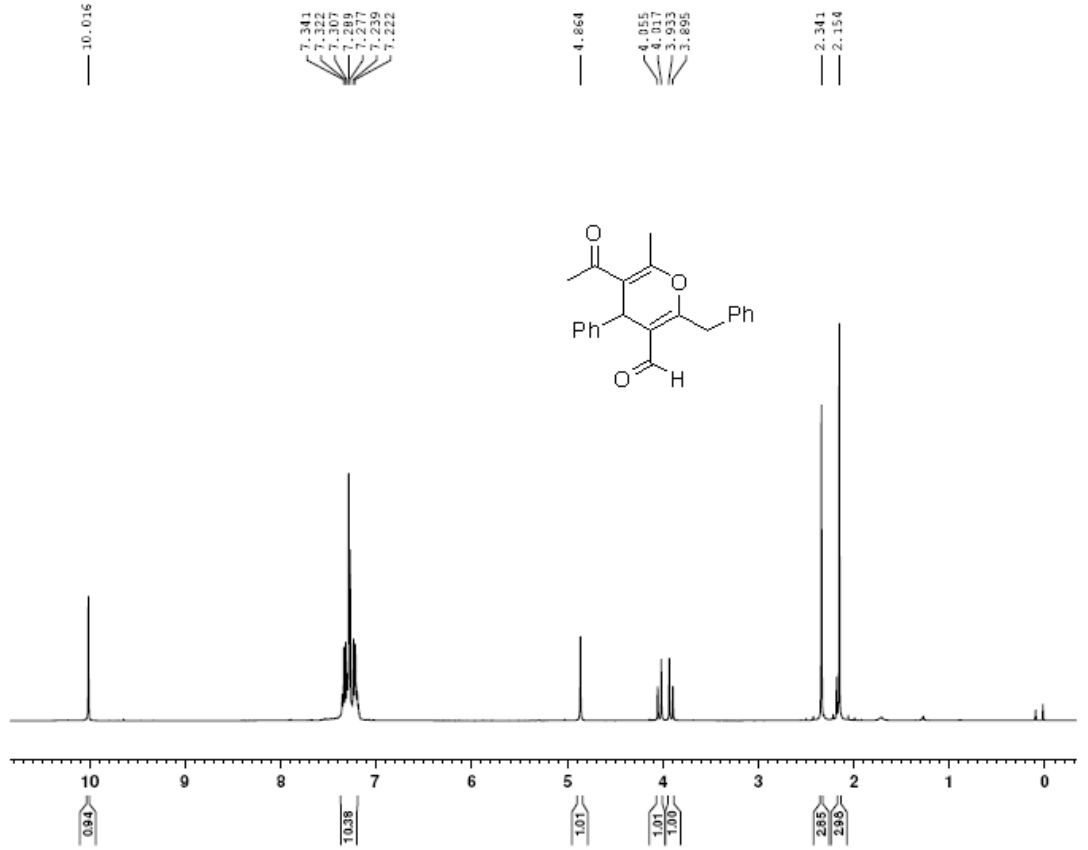

7.353
7.335
7.305
7.287
7.268
7.236
7.218
3.884
3.849
3.709
3.675
3.667
3.643
3.535
2.595
2.571
2.554
2.544
2.530
2.514
2.453
2.437
2.430
2.414
2.402
2.397
2.379
2.374
2.245
1.991
1.950
1.940
1.930
1.915
1.904
1.889
1.879
1.870
1.866
1.862
1.640
1.632
1.625
1.619
1.604
1.596
1.580
Ph
O
O
O
8.0 7.5 7.0 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
1.05
2.06
0.95
1.00
0.98
0.95
1.05
1.09
2.95
2.98
1.00
1.95
0.95

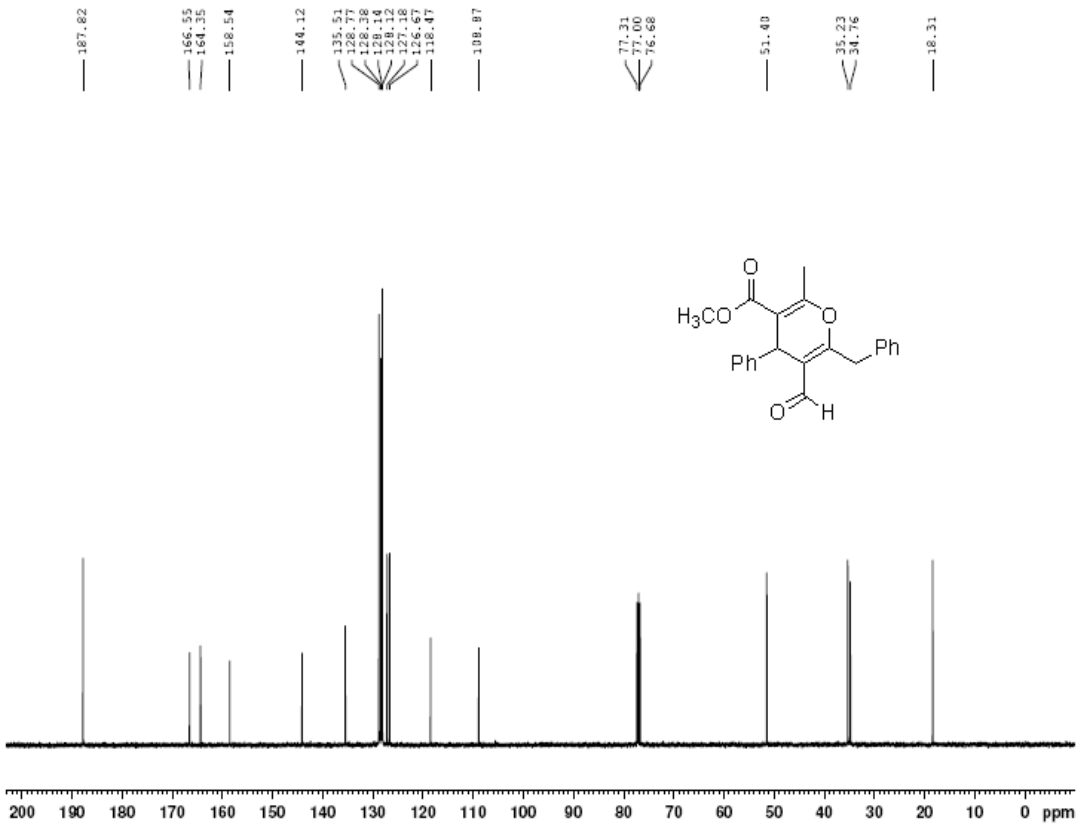

201.86
197.60
165.25
152.28
137.06
129.01
128.41
126.64
115.69
114.08
77.32
77.00
76.68
41.34
37.04
35.01
31.01
30.72
27.09
21.70
20.13
Ph
O
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

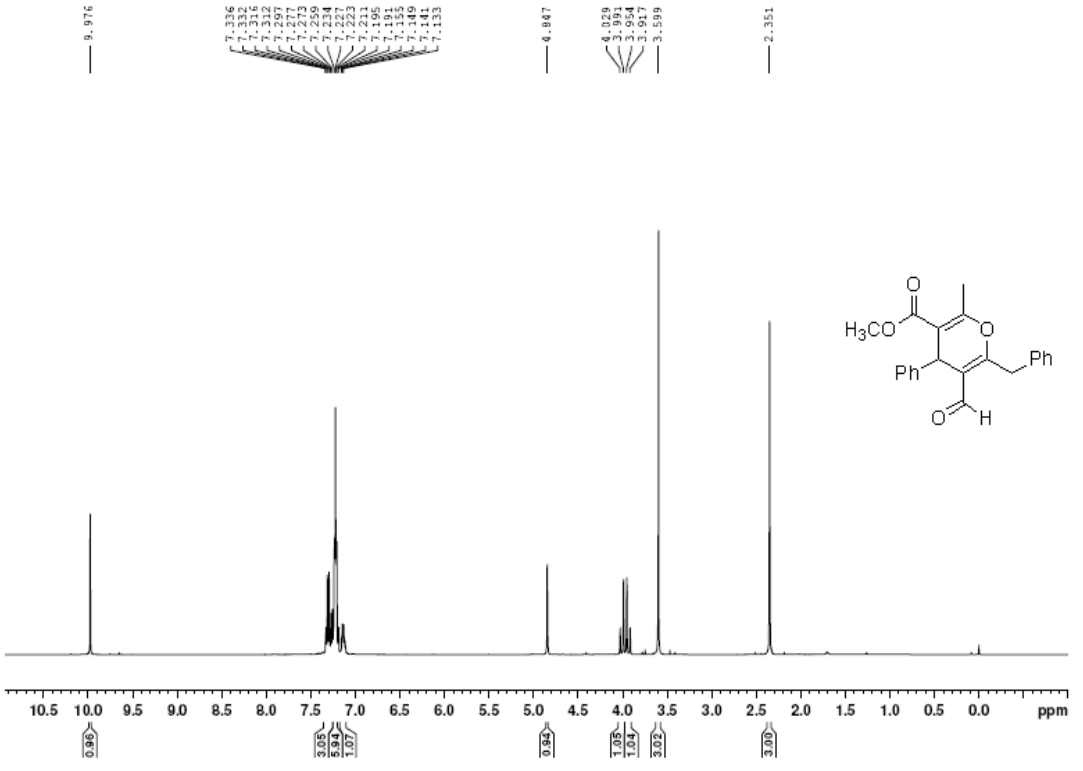

7.345
7.328
7.316
7.298
7.279
7.261
3.856
3.821
3.740
3.705
3.422
3.412
3.393
3.383
2.590
2.564
2.549
2.534
2.512
2.500
2.443
2.435
2.428
2.418
2.414
2.400
2.396
2.385
2.378
2.368
2.355
2.347
2.331
2.326
2.309
1.972
1.968
1.957
1.943
1.929
1.915
1.907
1.900
1.488
1.472
1.465
1.459
1.457
1.449
1.444
1.436
1.433
1.428
Ph
O
O
O
8 7 6 5 4 3 2 1 0 ppm
5.19
1.00
0.96
0.95
2.13
5.10
3.94
1.02

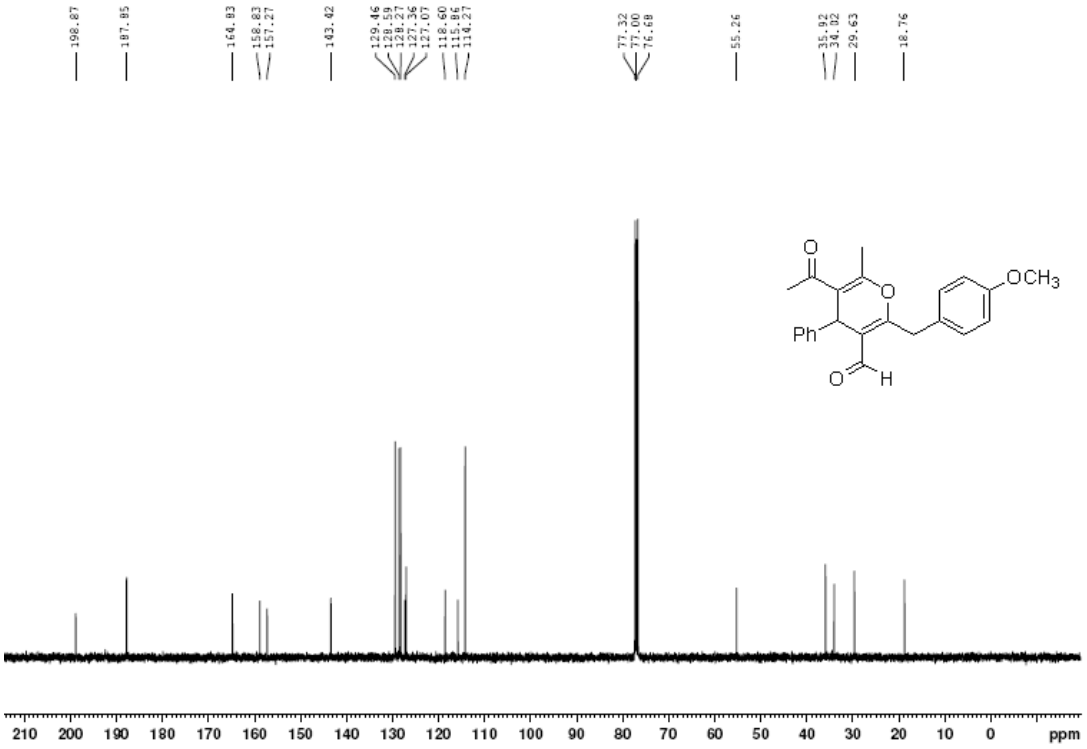

201.79
197.49
163.38
152.48
137.02
129.05
128.37
126.59
115.62
112.78
77.32
77.00
76.68
51.01
41.26
40.75
35.94
31.75
30.91
30.51
28.52
27.89
21.60
Ph
O
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
7.345
7.341
7.324
7.312
7.306
7.294
7.290
7.275
7.265
7.248
7.244
7.240
7.233
7.227
7.220
7.209
3.864
3.829
3.741
3.706
3.430
3.420
3.402
3.392
2.599
2.585
2.571
2.560
2.552
2.544
2.529
2.432
2.415
2.410
2.393
2.374
2.369
2.353
2.290
2.281
2.245
2.241
2.218
2.177
1.962
1.948
1.940
1.933
1.925
1.911
1.903
1.896
1.880
1.491
1.475
1.467
1.462
1.452
1.446
1.439
1.430
1.423
1.415
1.407
Ph
O
O
O
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
4.96
1.00
1.02
0.96
2.02
1.05
2.03
1.97
1.98
1.01
2.91
2.94

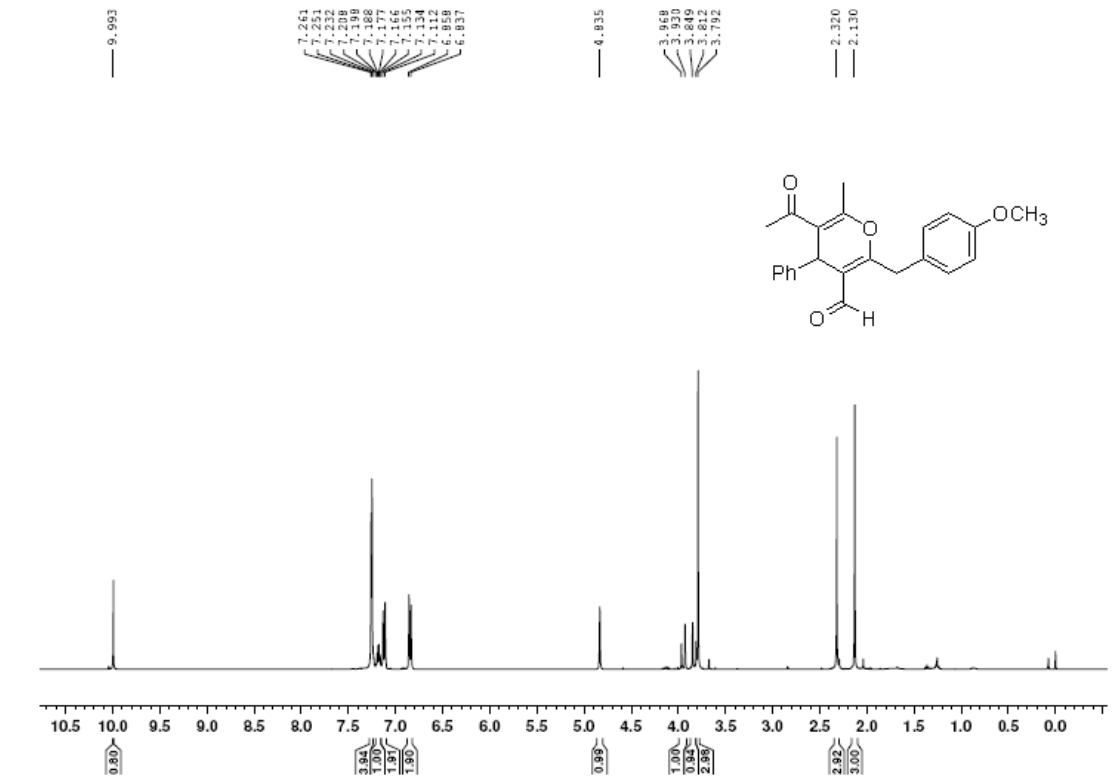

198.87
187.83
164.42
157.26
143.33
135.40
128.84
128.58
128.38
128.26
127.26
127.08
118.92
115.80
77.32
77.00
76.68
35.88
34.82
29.64
18.74
O
O
Ph
Ph
O
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

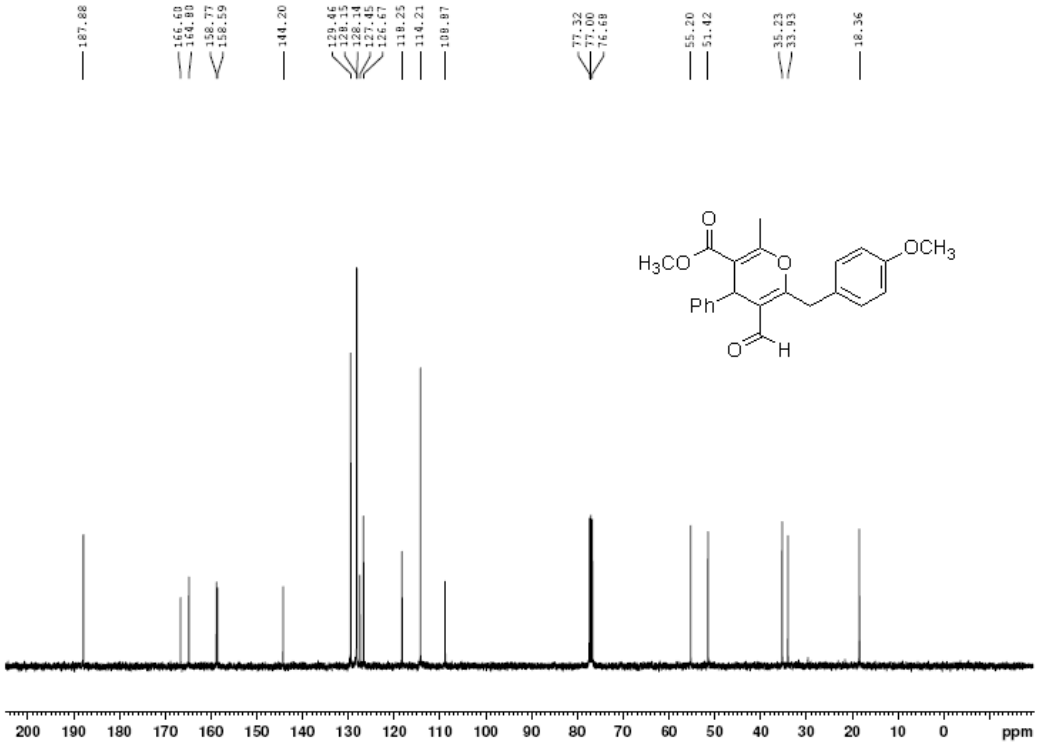

10.016
7.341
7.322
7.307
7.289
7.277
7.239
7.222
4.864
4.055
4.017
3.933
3.895
2.341
2.154
O
O
Ph
Ph
O
H
10 9 8 7 6 5 4 3 2 1 0
0.94
1.38
1.01
1.01
1.00
2.85
2.98

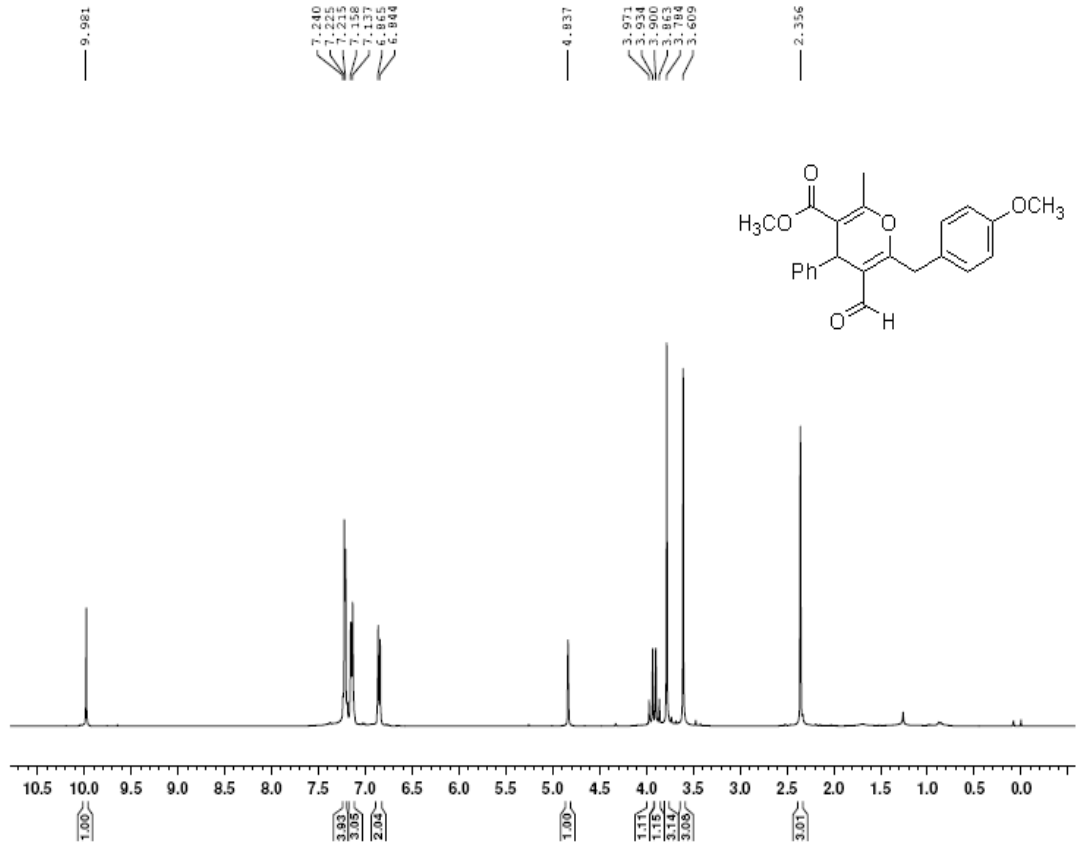

187.82
166.55
164.35
158.54
144.12
135.51
128.77
128.38
128.14
128.12
127.18
126.67
118.47
108.87
77.31
77.00
76.68
51.40
35.23
34.76
18.31
O
H3CO
O
Ph
Ph
O
H
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

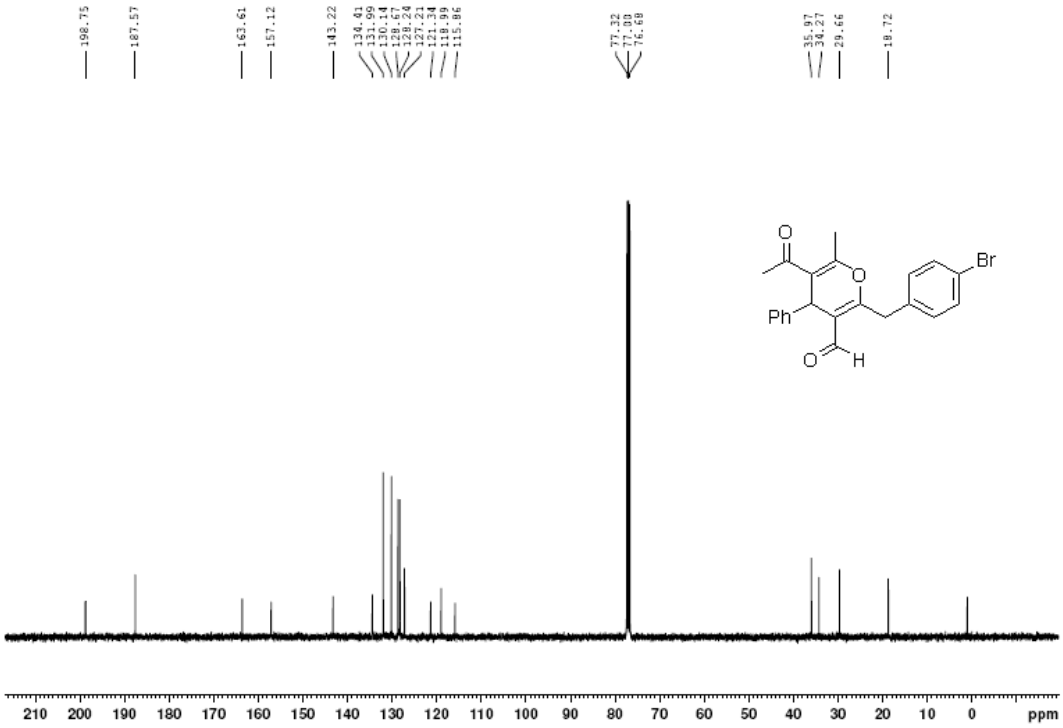

9.976
7.336
7.332
7.315
7.312
7.297
7.277
7.273
7.259
7.234
7.227
7.211
7.195
7.191
7.155
7.149
7.141
7.133
4.847
4.039
3.991
3.954
3.917
3.599
2.351
O
H3CO
O
Ph
Ph
O
H
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.90
3.05
5.94
1.07
0.94
1.08
1.04
3.02
3.00

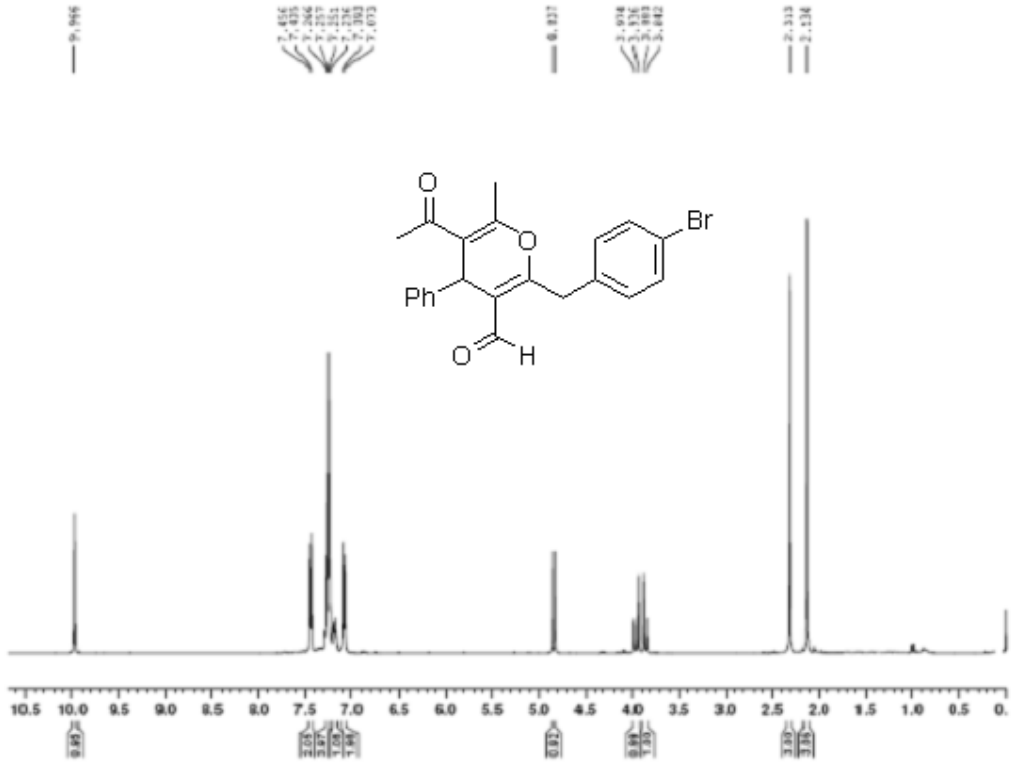

198.87
187.85
164.83
158.83
157.27
143.42
129.46
128.59
128.27
127.36
127.07
118.60
115.86
114.27
77.32
77.00
76.68
55.26
35.92
34.02
29.63
18.76
O
O
OCH3
Ph
O
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

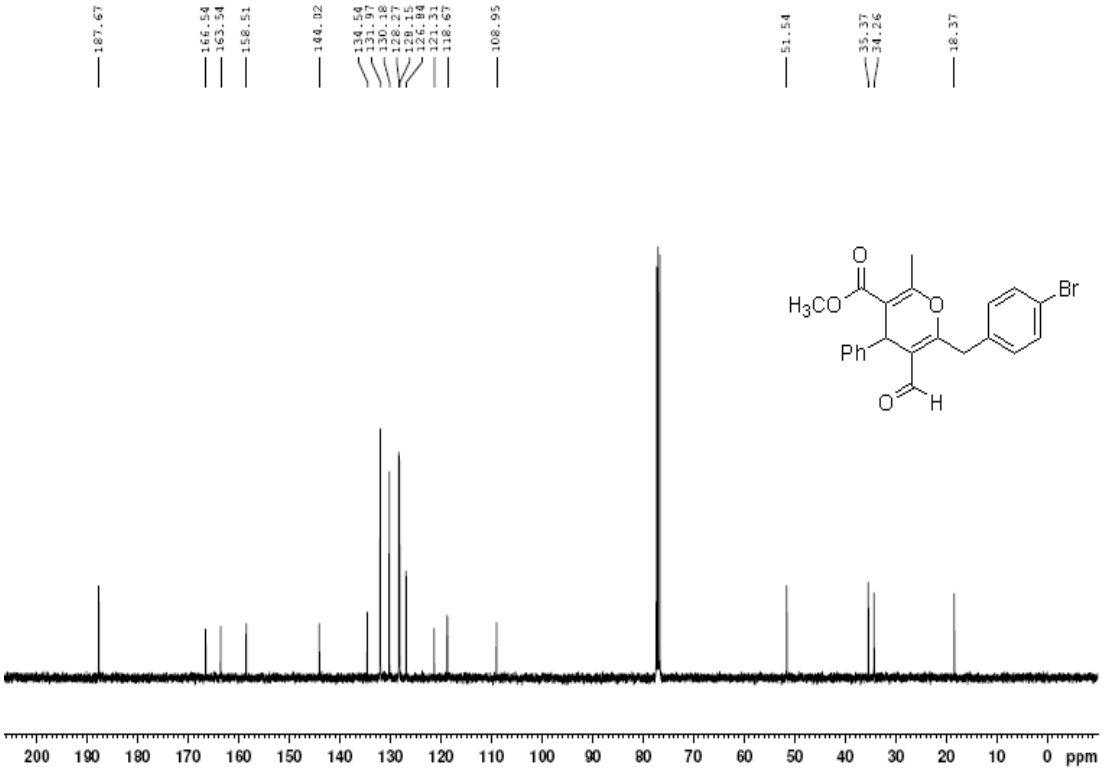

9.993
7.261
7.251
7.232
7.208
7.198
7.188
7.177
7.166
7.155
7.134
7.112
6.858
6.837
4.835
3.968
3.930
3.849
3.812
3.792
2.320
2.130
O
O
OCH3
Ph
O
H
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
0.80
3.94
1.00
1.97
1.90
0.98
1.00
0.94
2.98
2.92
3.00

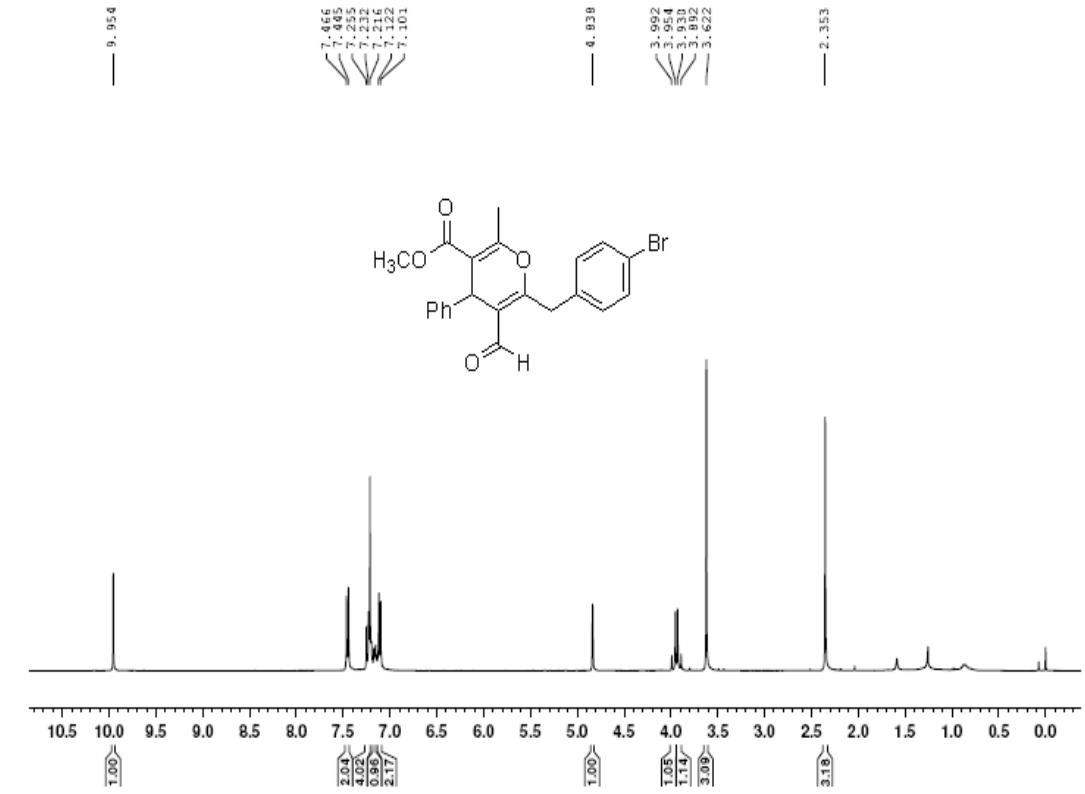

187.88
166.69
164.80
158.77
158.59
144.20
129.46
129.15
128.14
127.45
126.67
119.25
114.21
108.87
77.32
77.00
76.68
55.20
51.42
35.23
33.93
18.36
O
H3CO
O
OCH3
Ph
O
H
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

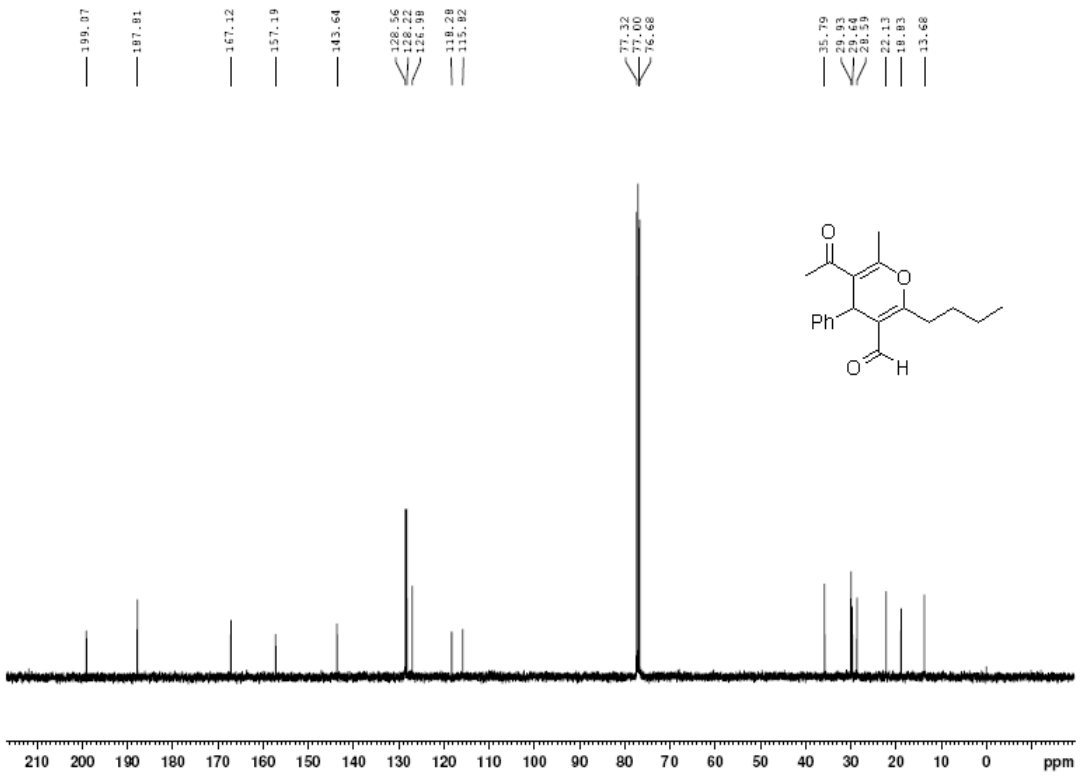

9.981
7.240
7.225
7.215
7.158
7.137
6.865
6.844
4.837
3.971
3.934
3.900
3.863
3.784
3.609
2.356
O
H3CO
O
OCH3
Ph
O
H
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
1.00
3.93
3.05
2.04
1.00
1.11
1.16
3.14
3.06
3.01

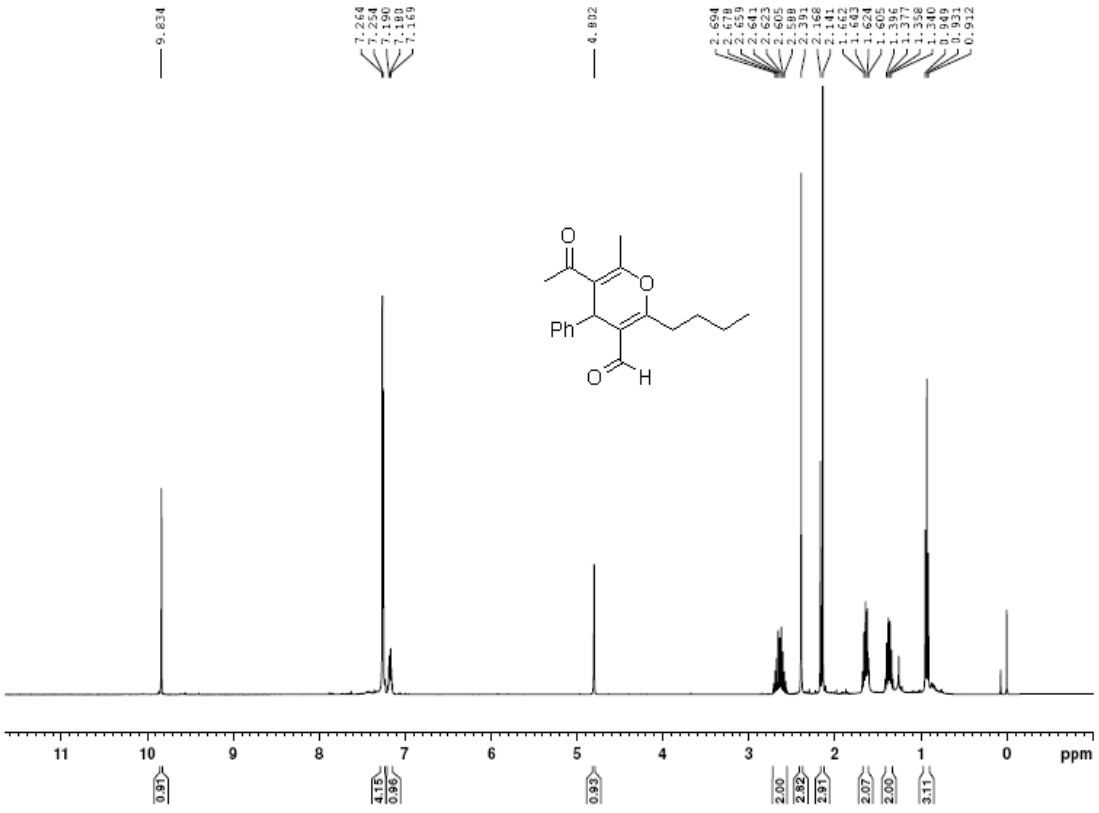

198.75
187.57
163.61
157.12
143.22
134.41
131.99
130.14
129.67
129.24
127.21
121.34
118.99
115.86
77.32
77.00
76.68
35.97
34.27
29.66
18.72
O
O
Br
Ph
O
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

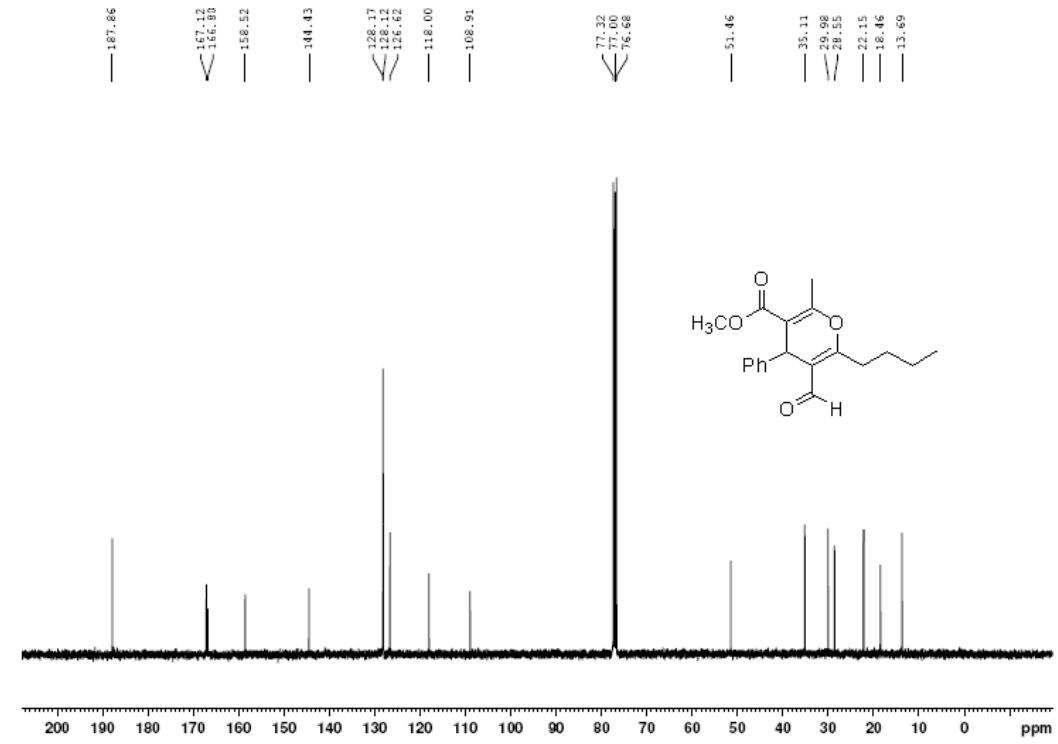

9.966
7.456
7.435
7.266
7.257
7.251
7.241
7.230
7.073
6.837
2.974
2.136
2.003
1.842
2.313
2.134
O
O
Br
Ph
O
H
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.

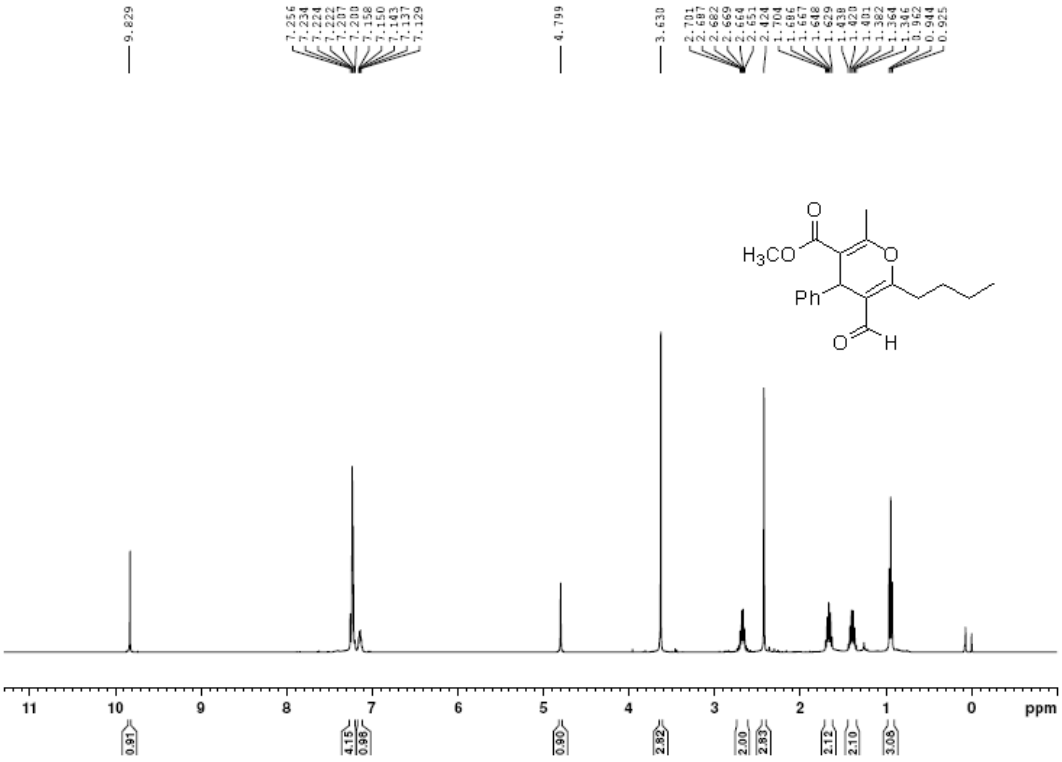

187.67
166.54
163.54
158.51
144.02
134.54
131.97
130.18
128.27
128.15
121.31
118.67
108.95
51.54
35.37
34.26
18.37
O
H3CO
O
Br
Ph
O
H
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

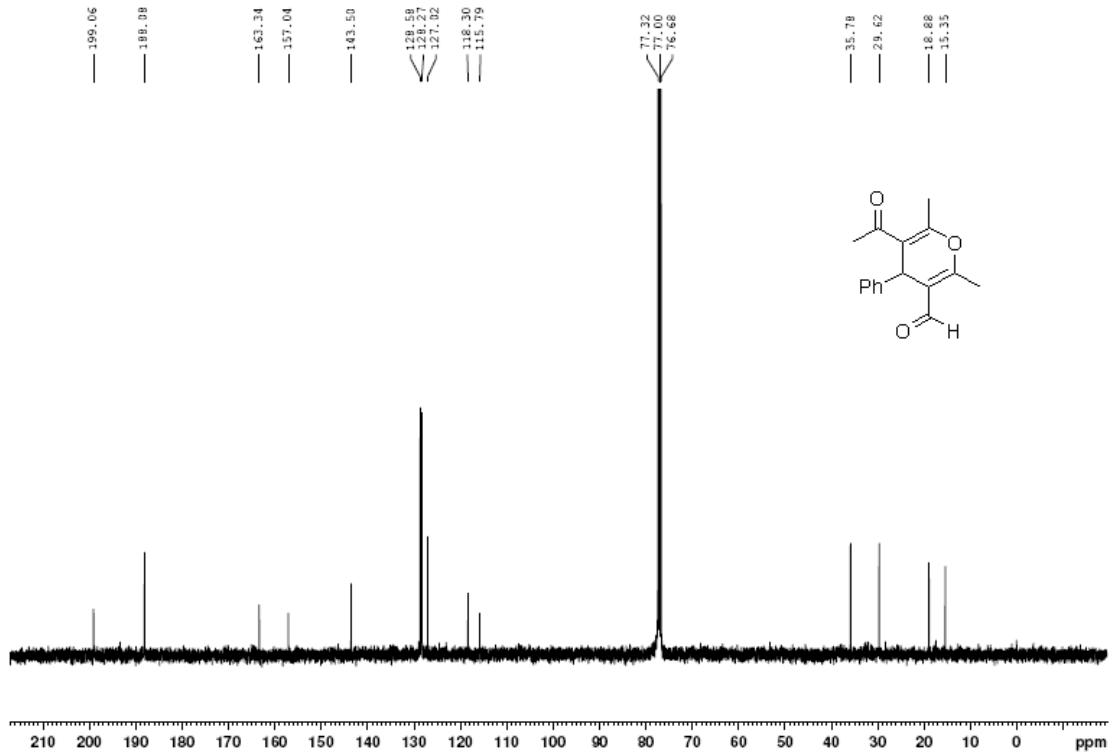

9.954
7.466
7.445
7.435
7.232
7.216
7.122
7.101
4.838
3.992
3.954
3.930
3.892
3.622
2.353
O
H3CO
O
Br
Ph
O
H
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
1.00
2.04
4.02
0.96
2.17
1.00
1.05
1.14
3.09
3.18

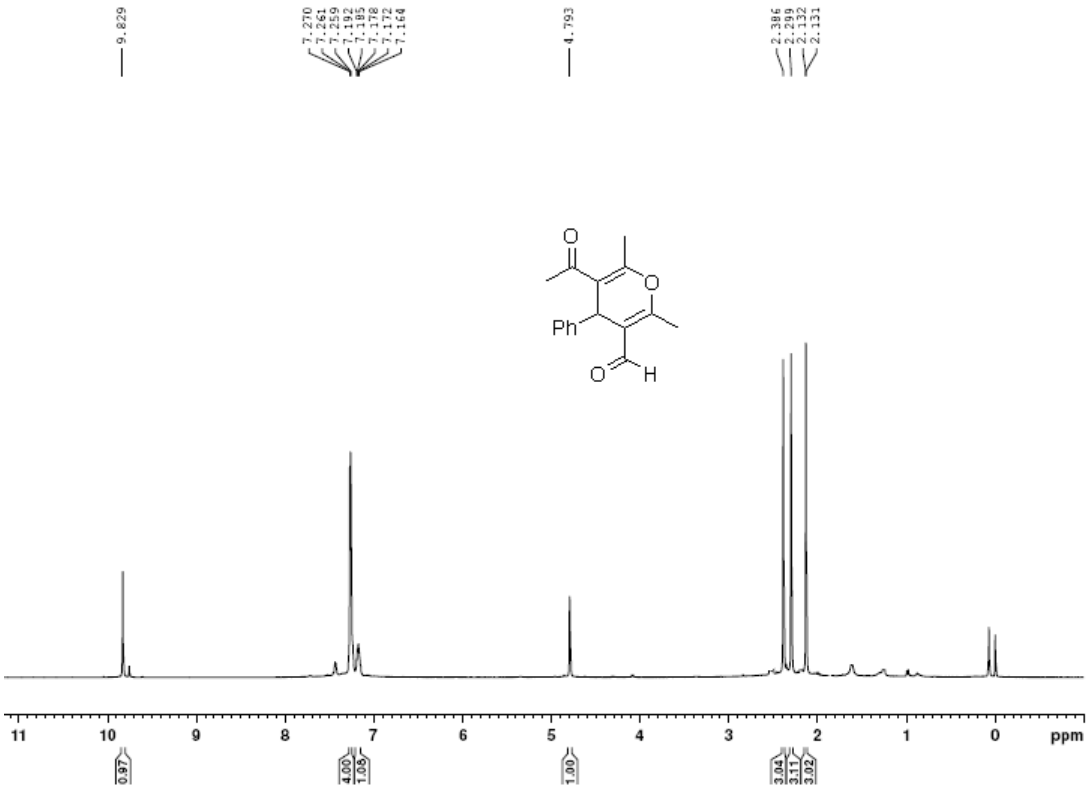

199.07
187.81
167.12
157.19
143.64
128.56
128.22
126.98
118.28
115.82
77.32
77.00
76.68
35.79
29.93
29.64
28.59
22.13
19.83
13.68
O
O
Ph
O
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

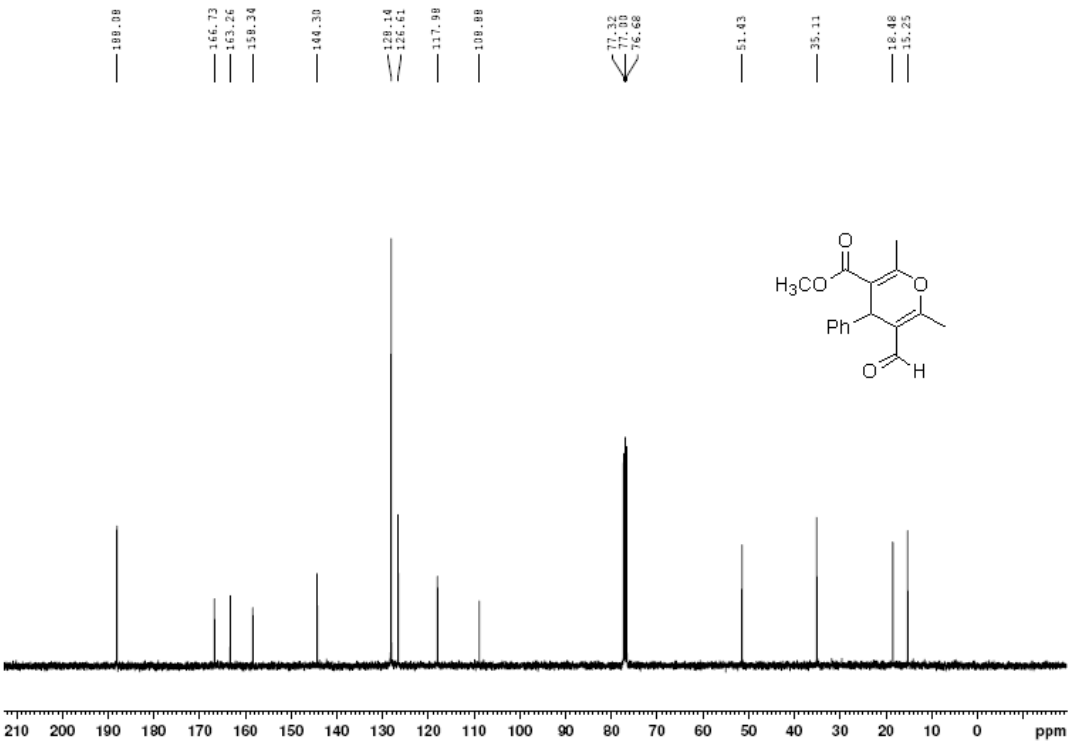

9.834
7.264
7.254
7.190
7.180
7.169
4.802
2.694
2.678
2.659
2.641
2.623
2.605
2.588
2.391
2.168
2.141
1.662
1.643
1.624
1.605
1.396
1.377
1.358
1.340
0.949
0.931
0.912
O
O
Ph
O
H
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.91
4.15
0.96
0.93
2.00
2.82
2.91
2.07
2.00
3.11

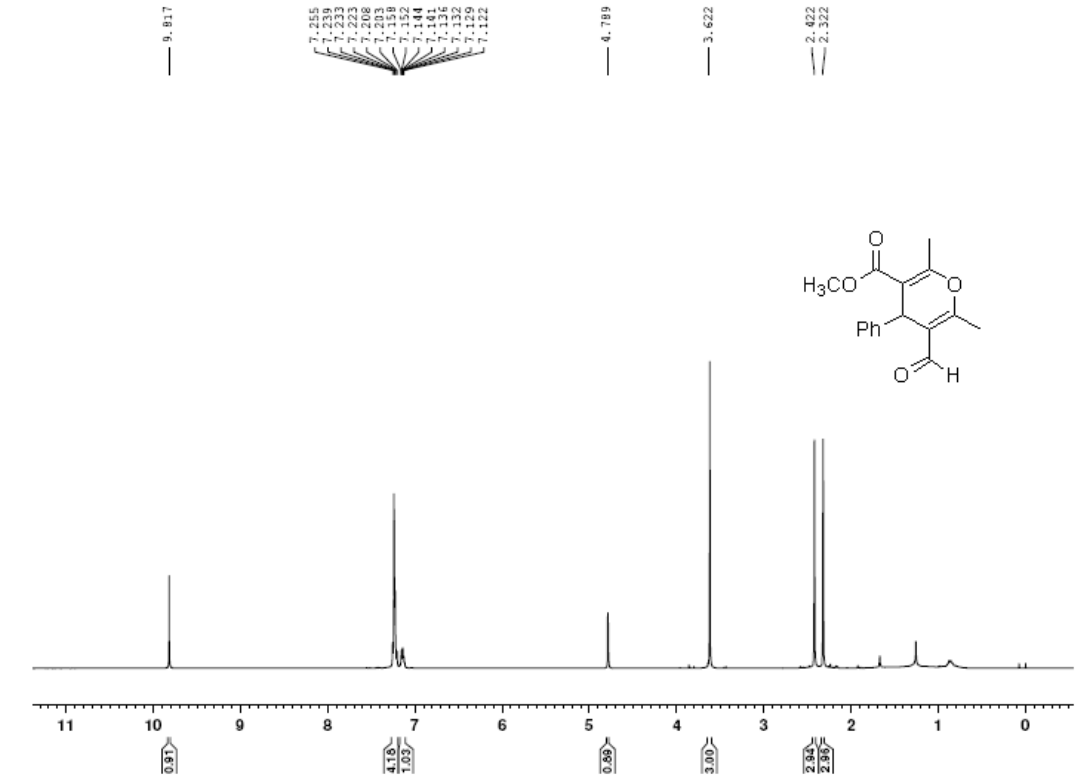

187.86
167.12
166.80
158.52
144.43
128.17
128.12
126.62
118.00
108.91
77.32
77.00
76.68
51.46
35.11
29.98
28.55
22.15
18.46
13.69
H3CO
O
Ph
O
H
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

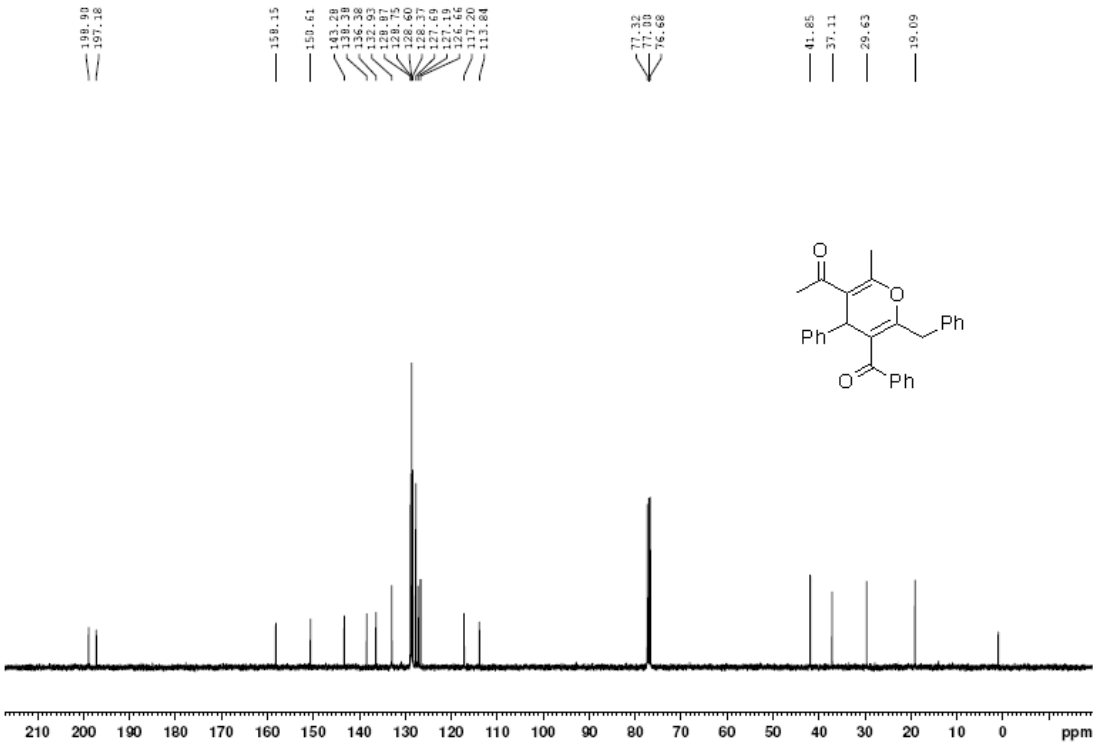

9.829
7.256
7.234
7.224
7.222
7.207
7.200
7.158
7.150
7.143
7.137
7.129
4.799
3.630
2.701
2.687
2.682
2.669
2.664
2.651
2.424
1.704
1.686
1.667
1.648
1.629
1.438
1.429
1.401
1.382
1.364
1.346
0.962
0.944
0.925
H3CO
O
Ph
O
H
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.91
4.15
0.98
0.90
2.82
2.00
2.83
2.12
2.10
3.06

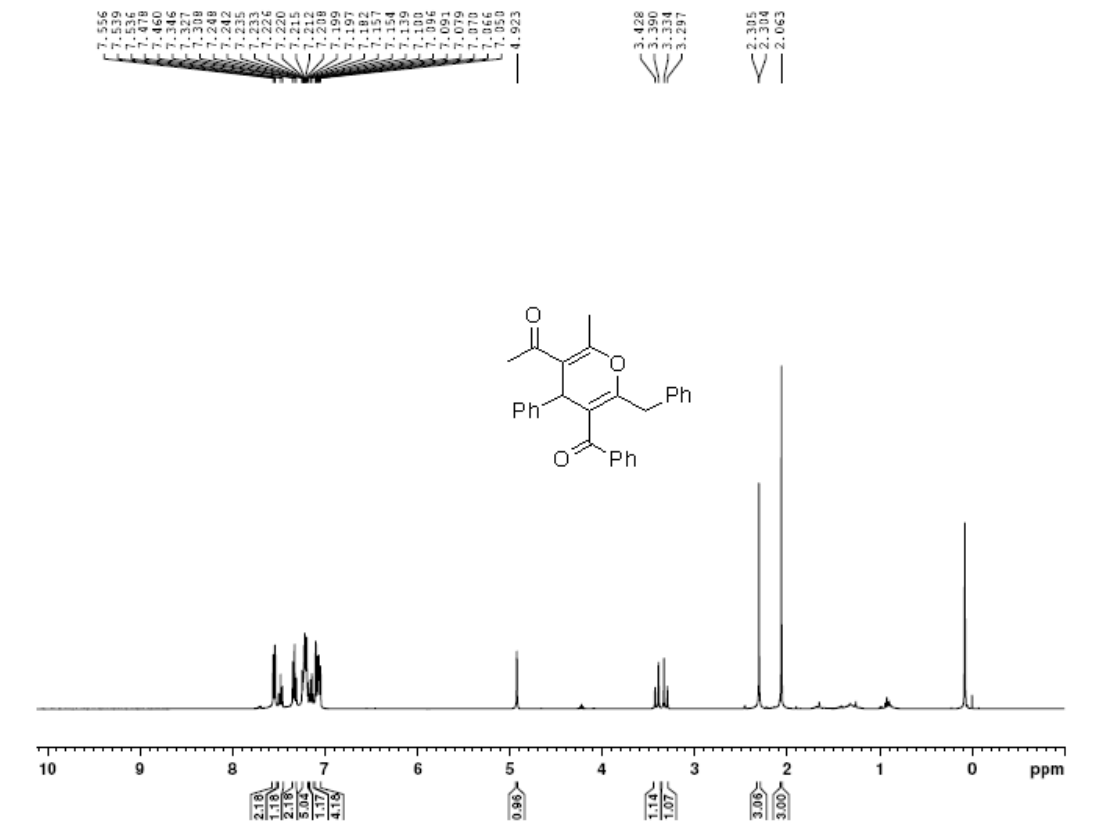

199.06
188.08
163.34
157.04
143.50
129.59
129.27
127.02
118.30
115.79
77.32
77.00
76.68
35.78
29.62
18.88
15.35
O
O
Ph
O
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

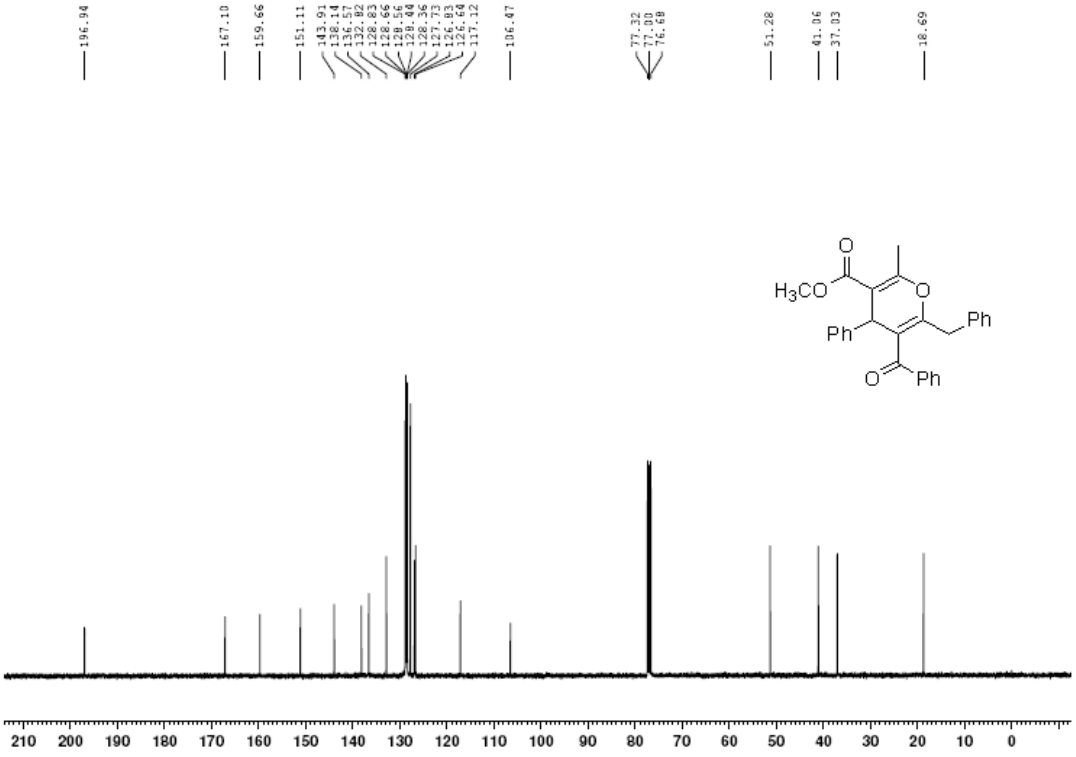

9.829
7.270
7.261
7.259
7.192
7.185
7.178
7.172
7.164
4.793
2.386
2.299
2.132
2.131
O
O
Ph
O
H
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.97
4.00
1.08
1.00
3.04
3.11
3.02

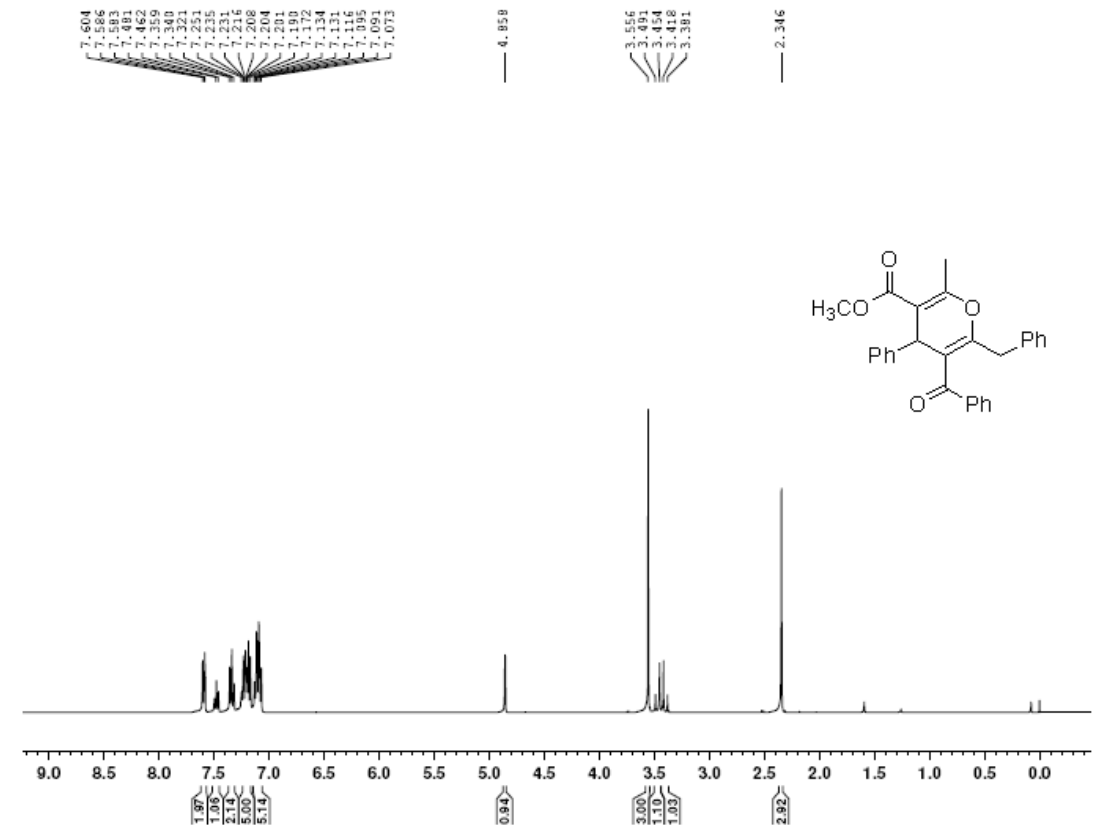

189.08
166.73
163.26
158.34
144.30
129.14
126.61
117.98
108.88
77.32
77.03
76.68
51.43
35.11
18.48
15.25
O
H3CO
O
Ph
O
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

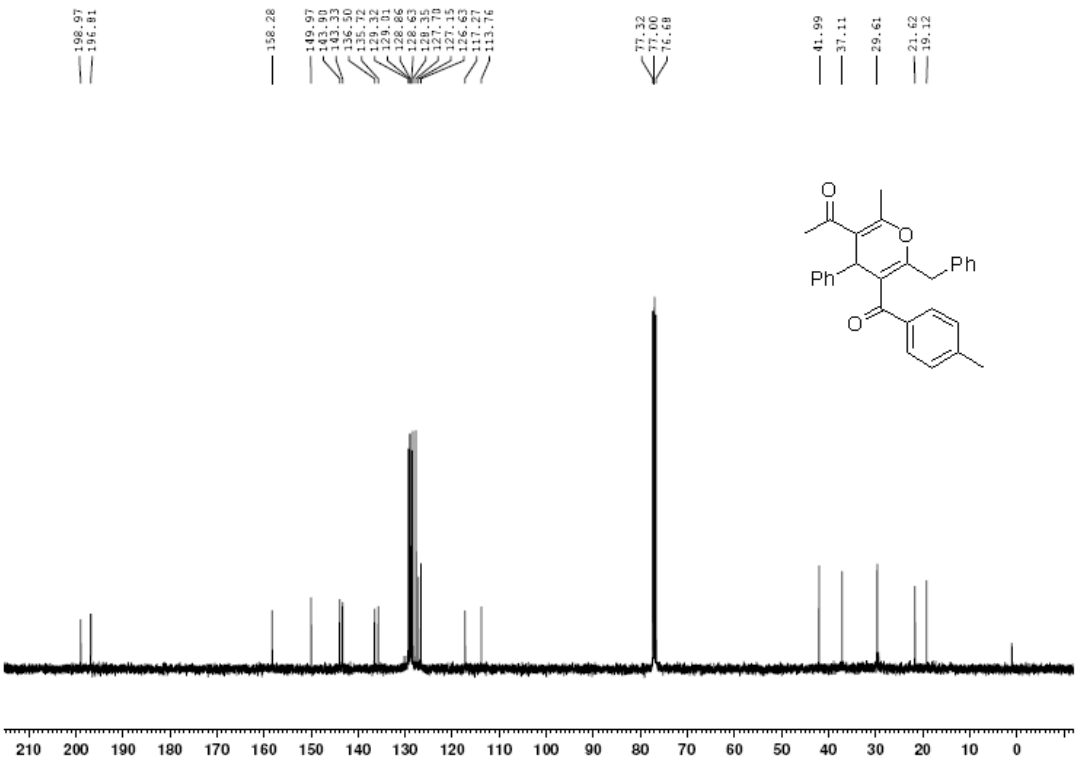

9.817
7.255
7.239
7.233
7.223
7.208
7.203
7.159
7.152
7.144
7.141
7.136
7.132
7.129
7.122
4.789
3.622
2.422
2.322
O
H3CO
O
Ph
O
H
11 10 9 8 7 6 5 4 3 2 1 0
0.91
4.18
1.03
0.89
3.00
2.94
2.96

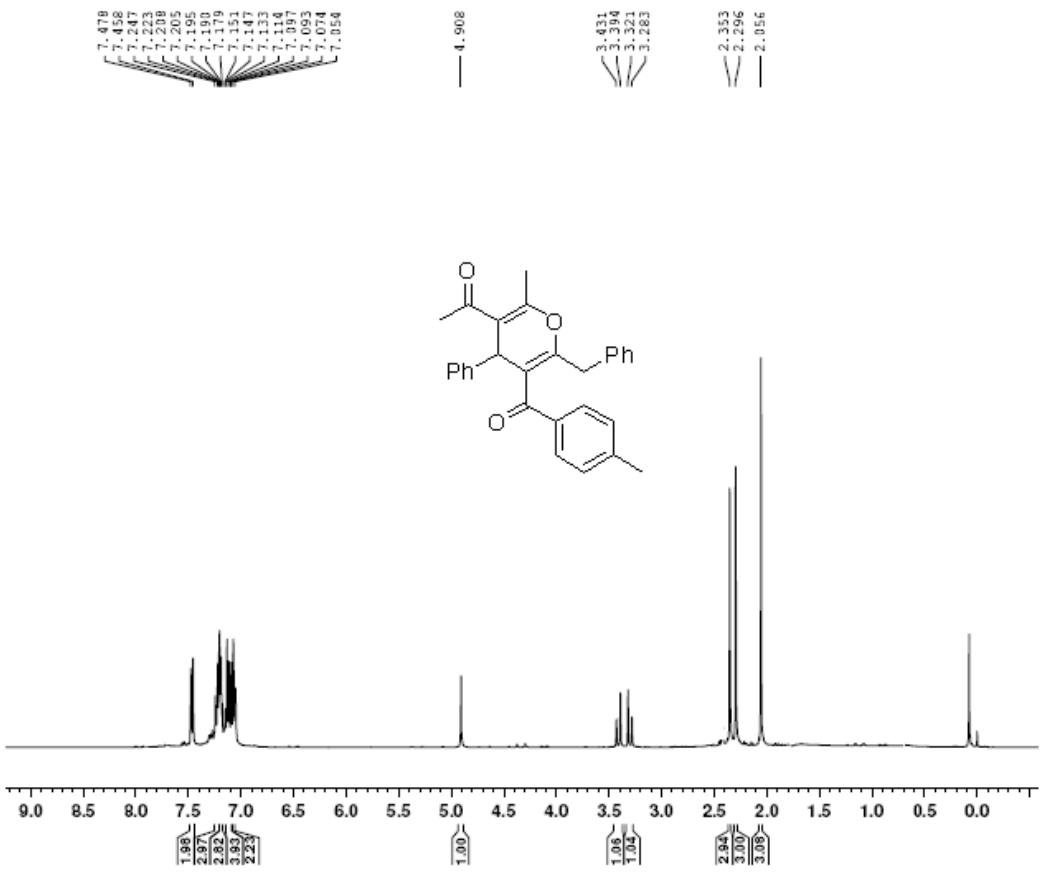

198.90
197.18
158.15
150.61
143.28
139.38
136.38
132.93
128.87
128.75
128.60
128.37
127.69
127.19
126.45
117.20
113.84
77.32
77.00
76.68
41.85
37.11
29.63
19.09
O
O
Ph
Ph
O
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

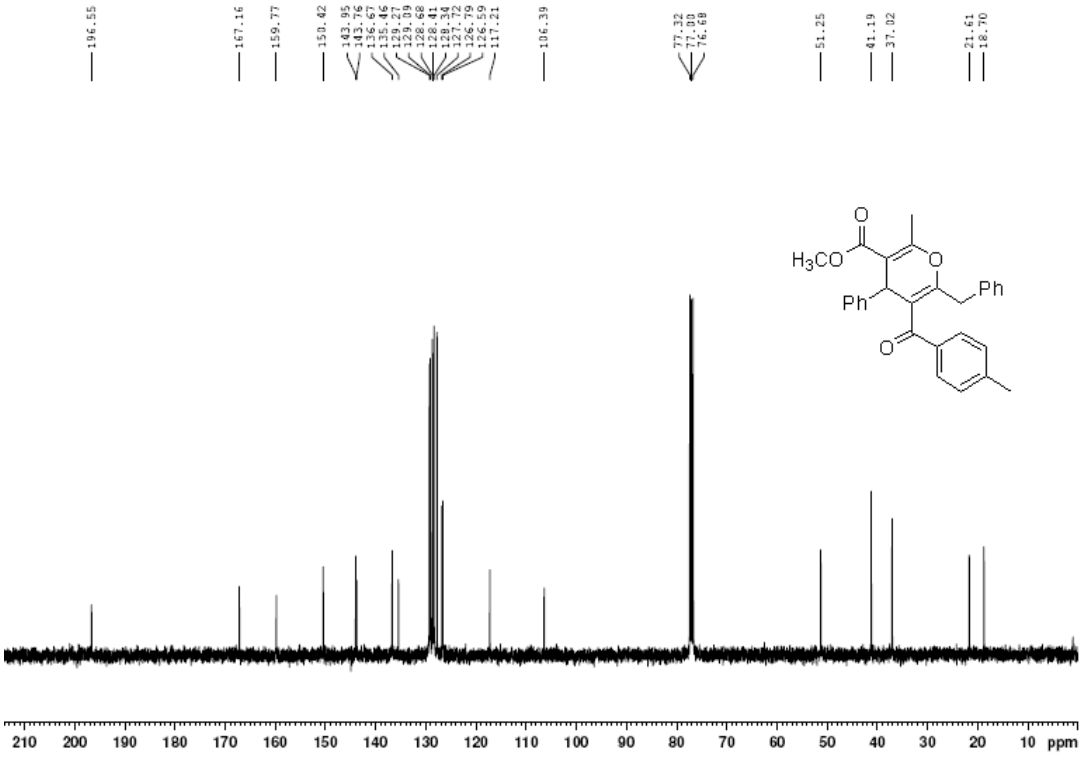

7.556
7.539
7.536
7.478
7.460
7.346
7.327
7.308
7.248
7.242
7.235
7.233
7.226
7.220
7.215
7.212
7.208
7.199
7.197
7.182
7.157
7.154
7.139
7.100
7.096
7.091
7.079
7.070
7.066
7.050
4.923
3.428
3.390
3.334
3.297
2.305
2.304
2.063
O
O
Ph
Ph
O
Ph
10 9 8 7 6 5 4 3 2 1 0 ppm
2.18
1.18
2.18
5.04
1.17
4.18
0.96
1.14
1.07
3.06
3.00

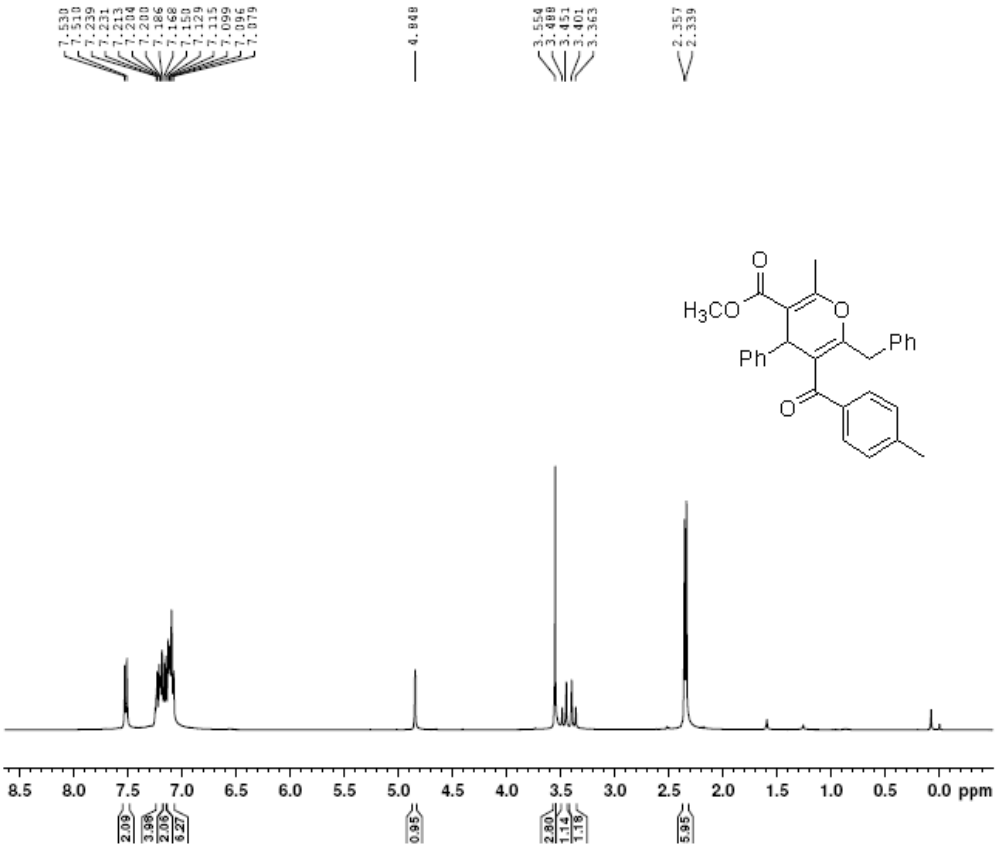

196.94
167.10
159.66
151.11
143.91
138.14
136.57
132.82
128.83
128.66
128.56
128.44
128.36
127.73
127.03
126.83
126.68
117.12
106.47
77.32
77.00
76.68
51.28
41.06
37.03
18.69
O
H3CO
O
Ph
Ph
O
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

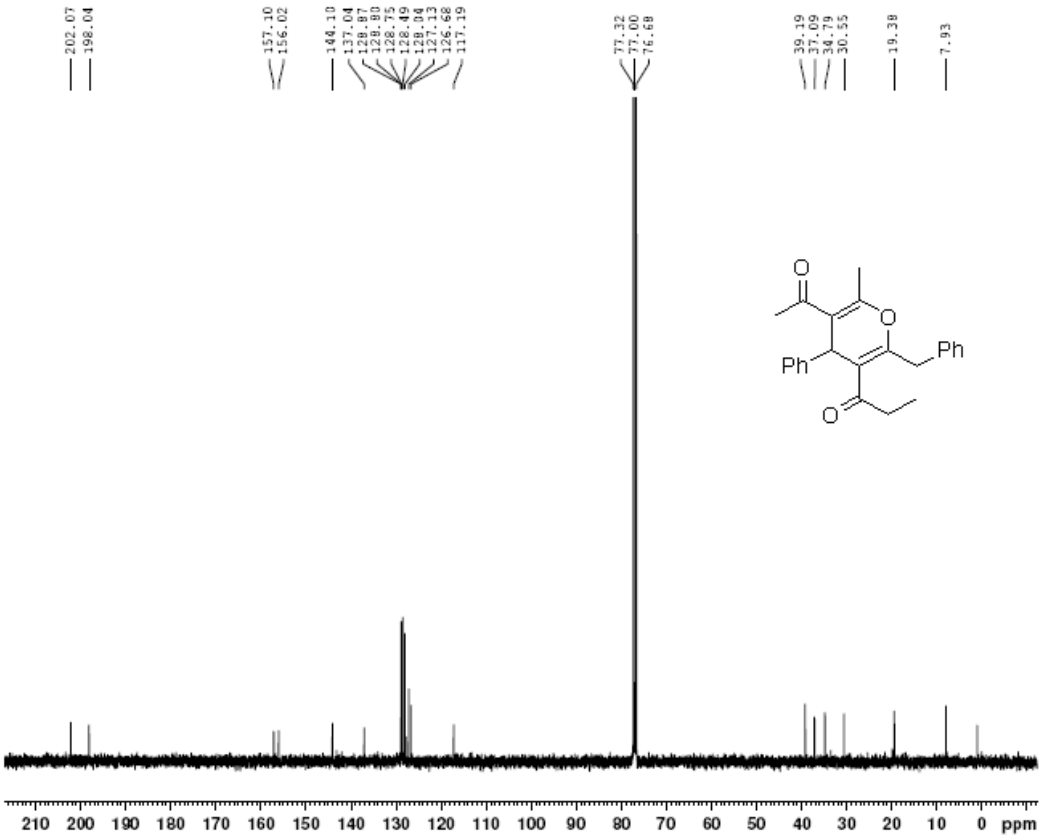

7.604
7.586
7.583
7.481
7.462
7.359
7.340
7.321
7.235
7.231
7.216
7.208
7.204
7.201
7.180
7.172
7.134
7.131
7.116
7.095
7.091
7.073
4.859
3.556
3.491
3.464
3.418
3.381
2.346
O
H3CO
O
Ph
Ph
O
Ph
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
1.97
1.06
2.14
5.00
5.14
0.94
3.00
1.10
1.03
2.92

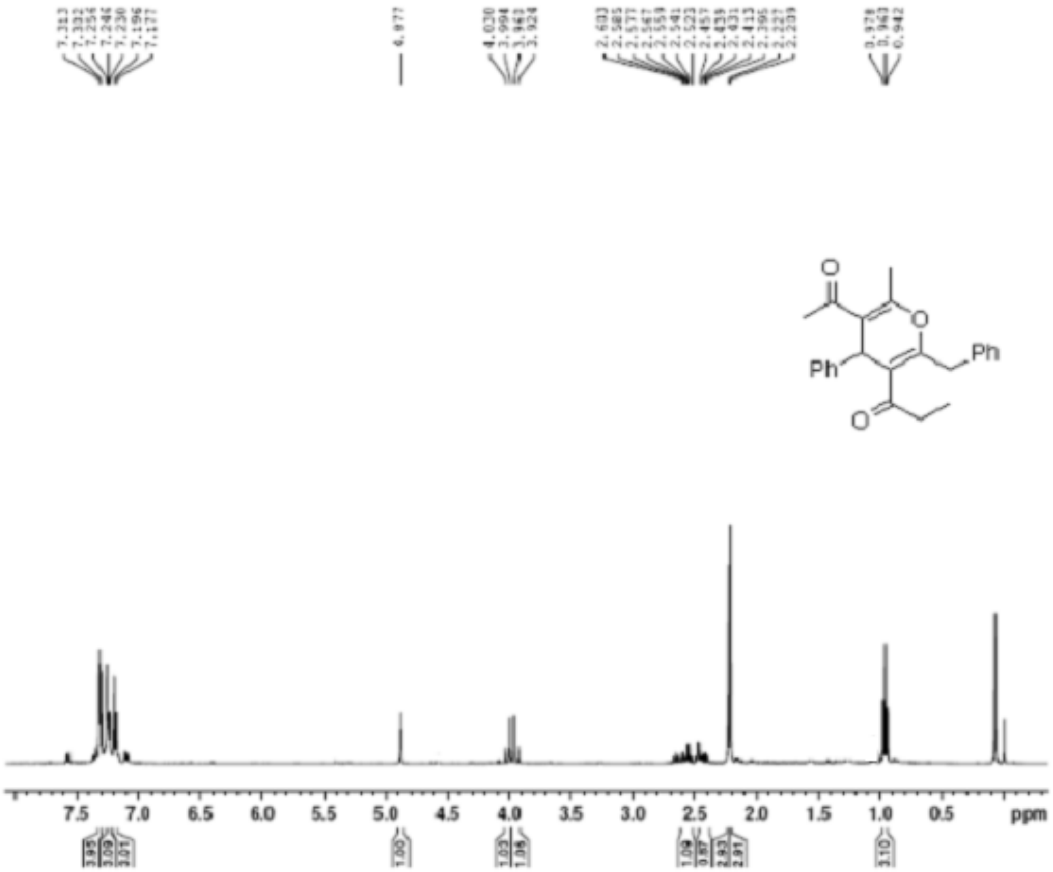

198.97
196.81
158.28
149.97
143.90
143.33
136.50
135.72
129.31
128.86
128.63
128.35
127.70
127.15
126.63
117.27
113.76
77.32
77.00
76.68
41.99
37.11
29.61
21.62
19.12
O
O
Ph
Ph
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

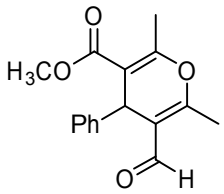

7.478
7.458
7.247
7.223
7.208
7.205
7.196
7.190
7.179
7.151
7.147
7.133
7.114
7.097
7.093
7.074
7.054
4.908
3.431
3.394
3.321
3.283
2.353
2.296
2.056
O
O
Ph
Ph
O
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
1.98
2.97
2.83
3.93
2.23
1.00
1.06
1.04
2.94
3.00
3.08


196.55
167.16
159.77
150.42
143.95
143.76
136.67
135.46
129.27
129.09
128.68
128.41
128.34
127.72
126.79
126.59
117.21
106.39
77.32
77.00
76.68
51.25
41.19
37.02
21.61
18.70
O
H3CO
O
Ph
Ph
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.530
7.510
7.239
7.231
7.213
7.204
7.200
7.186
7.168
7.150
7.129
7.115
7.099
7.096
7.079
4.848
3.554
3.488
3.451
3.401
3.363
2.357
2.339
O
H3CO
O
Ph
Ph
O
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
2.09
3.98
2.06
6.27
0.95
2.80
1.14
1.18
5.95


202.07
198.04
157.10
156.02
144.10
137.04
129.87
129.80
128.75
128.49
128.04
127.13
126.68
117.19
77.32
77.00
76.68
39.19
37.09
34.79
30.55
19.38
7.93
O
O
Ph
Ph
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.353
7.332
7.254
7.244
7.230
7.196
7.177
6.977
4.030
3.984
3.963
3.924
2.603
2.585
2.577
2.567
2.559
2.541
2.523
2.457
2.439
2.431
2.413
2.396
2.221
2.239
3.978
3.963
6.942
O
O
Ph
Ph
O
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 ppm



CCDC 805342

2fb