Supporting Information

$Sc(OTf)_3$-Catalyzed Cyclization of α-Allylated 1,3-Dicarbonyls: an Efficient Access to 2,2-Disubstituted 2,3-Dihydrofuran Derivatives

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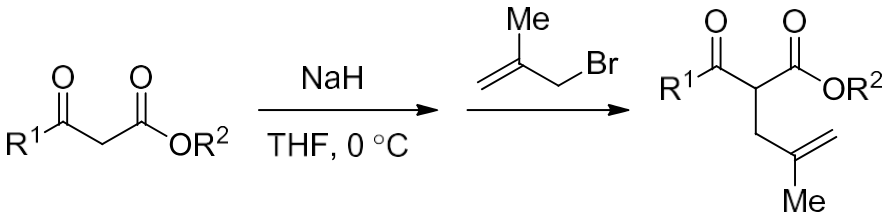
1- General information

All reactions were conducted in clean glassware with magnetic stirring. Commercially available reagents were used as received without further purification. Solvents were treated prior to use according to the standard methods. For chromatographic purification, technical-grade solvents were used. Purified compounds were further dried on high vacuum. NMR-spectra were measured in the given solvent at RT on *Bruker Ascend^{TM} 500M* (500.1 MHz, ^{1}H; 125.8 MHz, ^{13}C) instrument operating at the denoted spectrometer frequency given in mega Hertz (MHz) for the specified nucleus. Chemical shifts are given in parts per million (ppm) relative to tetramethylsilane (TMS) as an external standard for ^{1}H- and ^{13}C-NMR spectra and calibrated against the solvent residual peak. Multiplicities are reported as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, or as combination of them. Coupling constants *J* are given in Hertz (Hz). High resolution mass spectra were obtained with a Micromass GCT-TOF mass spectrometer.

2- Synthesis of α-allylated 1,3-dicarbonyl compounds

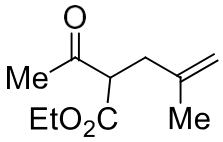


To a mixture of NaH (10 mmol, 60% dispersion in mineral oil) in dry THF (40 mL) in ice-water bath was added β-ketoester (10 mmol) dropwise, and the resulting mixture was stirred for about 20 minutes. In the same temperature, 3-bromo-2-methylpropene (10 mmol) was added dropwise, and then the reaction was warmed to room temperature for 6-12 hours. The reaction was quenched with sat. NH_4Cl solution (10 mL) and water (10 mL) sequentially. The subsequent mixture was extracted with ethyl ether (20 mL X 3). The organic phase was washed with brine, dried over Na_2SO_4, and filtered. The filtrate was condensed to the oily liquid which was purified by column chromatography on silica gel (200-300 mesh, eluent: PE/EA = 100/1 to 20/1) to provide the pure product **5**.

3- General procedure for synthesis of 2,3-dihydrofurans

To a solution of the α-allylated 1,3-dicarbonyl compound **5** (0.2 mmol) in 1,2-dichloroethane (2 mL) was added $Sc(OTf)_3$ (10 mol%) at rt. The resulting mixture was heated to 60 ˚C, generally for 1.5 h. After completion, the mixture was concentrated and purified by column chromatography on silica gel (200-300 mesh, eluent: PE/EA = 20/1) to provide the desired 2,3-dihydrofuran product **6**.

4- Characterization for all compounds



5a

Compound **5a** was obtained as colourless oil in 65% yield.

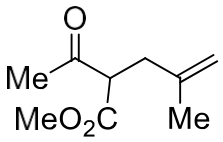
^{1}H NMR (500 MHz, $CDCl_3$): δ 4.78 (s, 1H), 4.69 (s, 1H), 4.18 (q, *J* = 7.2 Hz, 2H), 3.66 (dd, *J* = 7.7, 7.6 Hz, 1H), 2.89 (dd, *J* = 15.2, 8.1 Hz, 1H), 2.54 (dd, *J* = 15.2, 7.2 Hz, 1H), 2.23 (s, 3H), 1.73 (s, 3H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 202.6, 169.4, 141.8, 112.2, 61.4, 58.2, 35.8, 28.8, 22.4, 14.1.

IR (KBr, cm^{-1}): 1746, 1712, 1653.

HRMS (ESI) calcd for $C_{10}H_{17}O_3$ $(M+H)^+$: 185.1172; Found: 185.1175.

(Ref: B. M. Šmit and R. Z. Pavlović, *Tetrahedron*, 2015, **71**, 1101.)



5b

Compound **5b** was obtained as colourless oil in 70% yield.

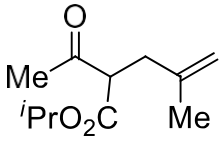
^{1}H NMR (500 MHz, $CDCl_3$): δ 4.78 (s, 1H), 4.68 (s, 1H), 3.72 (s, 3H), 3.68 (dd, *J* = 7.7, 7.6 Hz, 1H), 2.58 (dd, *J* = 15.1, 8.1 Hz, 1H), 2.54 (dd, *J* = 15.1, 7.2 Hz, 1H), 2.23 (s, 3H), 1.73 (s, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 202.5, 169.9, 141.8, 112.3, 58.1, 52.4, 35.8, 28.8, 22.4.

IR (KBr, cm^{-1}): 1740, 1710, 1637, 1442, 1357.

HRMS (ESI) calcd for $C_9H_{15}O_3$ $(M+H)^+$: 171.1016; Found: 171.1020.

(Ref: C. M. R. Volla, S. R. Dubbaka and P. Vogel, *Tetrahedron*, 2009, **65**, 504.)



5c

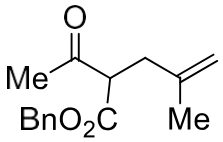
Compound **5c** was obtained as colourless oil in 60% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 5.04 (sept, *J* = 6.3 Hz, 1H), 4.77 (s, 1H), 4.68 (s, 1H), 3.61 (dd, *J* = 7.8, 7.5 Hz, 1H), 2.57 (dd, *J* = 15.2, 8.4 Hz, 1H), 2.52 (dd, *J* = 15.2, 7.1 Hz, 1H), 2.22 (s, 3H), 1.73 (s, 3H), 1.23 (d, *J* = 6.3 Hz, 6H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 202.6, 168.9, 141.9, 112.1, 69.0, 58.4, 35.7, 28.7, 22.4, 21.6, 21.5.

IR (KBr, cm^{-1}): 1738, 1719, 1376, 1107.

HRMS (ESI) calcd for $C_{11}H_{19}O_3$ $(M+H)^+$: 199.1329; Found: 199.1323.



5d

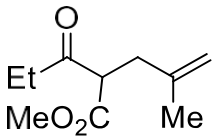
Compound **5d** was obtained as colourless oil in 58% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.39-7.31 (m, 5H), 5.16 (s, 2H), 4.76 (s, 1H), 4.67 (s, 1H), 3.72 (dd, *J* = 7.8, 7.5 Hz, 1H), 2.60 (dd, *J* = 15.2, 8.2 Hz, 1H), 2.56 (dd, *J* = 15.2, 7.1 Hz, 1H), 2.19 (s, 3H), 1.72 (s, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 202.3, 169.3, 141.7, 135.2, 128.6, 128.5, 128.3, 112.3, 67.2, 58.2, 35.8, 28.8, 22.4.

IR (KBr, cm^{-1}): 1754, 1718, 1656, 1462.

HRMS (ESI) calcd for $C_{15}H_{19}O_3$ $(M+H)^+$: 247.1329; Found: 247.1330.



5e

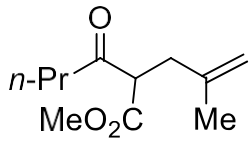
Compound **5e** was obtained as colourless oil in 62% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 4.77 (s, 1H), 4.67 (s, 1H), 3.71 (s, 3H), 3.73-3.67 (m, 1H), 2.65-2.45 (m, 4H), 1.72 (s, 3H), 1.05 (t, *J* = Hz, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 205.2, 170.0, 141.9, 112.2, 57.1, 52.4, 35.9, 35.3, 22.4, 7.5.

IR (KBr, cm^{-1}): 1717, 1652, 919.

HRMS (ESI) calcd for $C_{10}H_{17}O_3$ $(M+H)^+$: 185.1172; Found: 185.1175.



5f

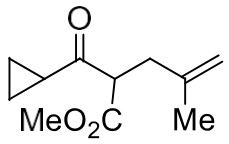
Compound **5f** was obtained as colourless oil in 65% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 4.77 (s, 1H), 4.67 (s, 1H), 3.71 (s, 3H), 3.74-3.66 (m, 1H), 2.60-2.42 (m, 4H), 1.72 (s, 3H), 1.64-1.55 (m, 2H), 0.89 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 204.6, 170.0, 141.9, 112.2, 57.4, 52.4, 43.8, 35.8, 22.4, 16.8, 13.5.

IR (KBr, cm^{-1}): 1701, 1673.

HRMS (ESI) calcd for $C_{11}H_{19}O_3$ $(M+H)^+$: 199.1329; Found: 199.1330.



5g

Compound **5g** was obtained as colourless oil in 68% yield.

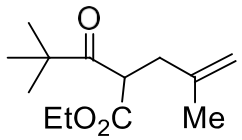
^{1}H NMR (500 MHz, $CDCl_3$): δ 4.79-4.77 (m, 1H), 4.72-4.69 (m, 1H), 3.84 (dd, *J* = 8.0, 7.1 Hz, 1H), 3.73 (s, 3H), 2.66-2.56 (m, 2H), 2.13-2.05 (m, 1H), 1.74 (s, 3H), 1.10-1.02 (m, 2H), 0.96-0.91 (m, 2H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 204.7, 170.0, 141.9, 112.1, 58.3, 52.4, 35.8, 22.5, 19.7, 11.9, 11.7.

IR (KBr, cm^{-1}): 1740, 1700, 1633.

HRMS (ESI) calcd for $C_{11}H_{17}O_3$ $(M+H)^+$: 197.1172; Found: 197.1172.

(Ref: A. Faulkner, J. S. Scott and J. F. Bower, *J. Am. Chem. Soc*., 2015, **137**, 7224.)



5h

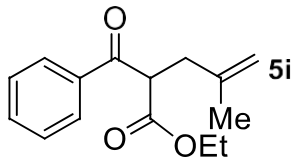
Compound **5h** was obtained as colourless oil in 46% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 4.75 (s, 1H), 4.69 (s, 1H), 4.20-4.06 (m, 3H), 2.59 (dd, *J* = 14.6, 8.1 Hz, 1H), 2.40 (dd, *J* = 14.6, 6.3 Hz, 1H), 1.72 (s, 3H), 1.21 (t, *J* = 7.2 Hz, 3H), 1.15 (s, 9H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 209.2, 169.2, 142.0, 112.5, 61.2, 51.2, 45.3, 37.4, 26.1, 22.5, 14.0.

IR (KBr, cm^{-1}): 1743, 1707, 1478, 1198.

HRMS (ESI) calcd for $C_{13}H_{23}O_3$ $(M+H)^+$: 227.1642; Found: 227.1647.



Compound **5i** was obtained as colourless oil in 75% yield.

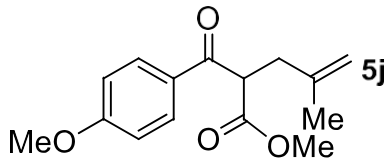
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00 (dd, *J* = 8.6, 1.4 Hz, 2H), 7.58 (tt, *J* = 7.4, 1.2 Hz, 1H), 7.47 (dd, *J* = 8.6, 7.5 Hz, 2H), 4.77 (s, 1H), 4.71 (s, 1H), 4.54 (dd, *J* = 8.0, 6.7 Hz, 1H), 4.13 (dq, *J* = 7.2, 1.3 Hz, 2H), 2.75 (dd, *J* = 15.2, 8.0 Hz, 1H), 2.69 (dd, *J* = 15.2, 6.6 Hz, 1H), 1.76 (s, 3H), 1.16 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 194.5, 169.4, 142.0, 136.1, 133.4, 128.7, 128.6, 112.1, 61.4, 52.7, 36.4, 22.6, 13.9.

IR (KBr, cm^{-1}): 1743, 1691, 1598, 1452.

HRMS (ESI) calcd for $C_{15}H_{19}O_3$ $(M+H)^+$: 247.1329; Found: 247.1328.

(Ref: R. Queignec, B. Kirschleger, F. Lambert and M. Aboutaj, S*ynth. Commun.*, 1988, **18**, 1213.)



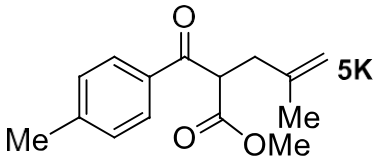
Compound **5j** was obtained as colourless oil in 79% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00 (d, *J* = 9.0 Hz, 2H), 6.95 (d, *J* = 9.0 Hz, 2H), 4.76 (s, 1H), 4.70 (s, 1H), 4.53 (dd, *J* = 7.9, 6.7 Hz, 1H), 3.87 (s, 3H), 3.67 (s, 3H), 2.75 (dd, *J* = 15.2, 8.0 Hz, 1H), 2.67 (dd, *J* = 15.2, 6.6 Hz, 1H), 1.75 (s, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 192.8, 170.2, 163.9, 142.2, 131.0, 129.1, 114.0, 112.0, 55.5, 52.5, 52.2, 36.6, 22.7.

IR (KBr, cm^{-1}): 1741, 1681, 1633.

HRMS (ESI) calcd for $C_{15}H_{19}O_4$ $(M+H)^+$: 263.1278; Found: 263.1285.



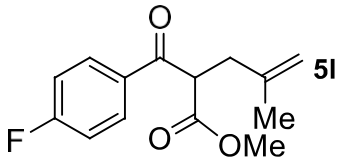
Compound **5k** was obtained as colourless oil in 55% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.91 (d, *J* = 8.2 Hz, 2H), 7.28 (d, *J* = 8.2 Hz, 2H), 4.77 (s, 1H), 4.70 (s, 1H), 4.56 (dd, *J* = 7.8, 6.8 Hz, 1H), 3.67 (s, 3H), 2.75 (dd, *J* = 15.2, 8.0 Hz, 1H), 2.68 (dd, *J* = 15.2, 6.7 Hz, 1H), 2.42 (s, 3H), 1.76 (s, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 194.1, 170.1, 144.6, 142.1, 133.6, 129.5, 128.8, 112.1, 52.5, 52.4, 36.6, 22.7, 21.7.

IR (KBr, cm^{-1}): 1737, 1683.

HRMS (ESI) calcd for $C_{15}H_{19}O_3$ $(M+H)^+$: 247.1329; Found: 247.1325.



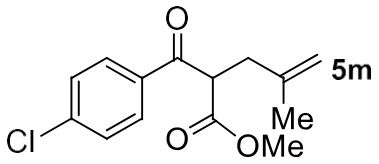
Compound **5l** was obtained as colourless oil in 63% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.04 (dd, *J* = 8.9, 5.4 Hz, 2H), 7.14 (dd, *J* = 8.7, 8.6 Hz, 2H), 4.76 (s, 1H), 4.68 (s, 1H), 4.53 (dd, *J* = 7.7, 7.0 Hz, 1H), 3.67 (s, 3H), 2.75 (dd, *J* = 15.2, 7.8 Hz, 1H), 2.68 (dd, *J* = 15.2, 6.8 Hz, 1H), 1.74 (s, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 192.8, 169.7, 166.0 (d, *J* = 256.2 Hz), 141.9, 132.5 (d, *J* = 3.1 Hz), 131.3 (d, *J* = 9.6 Hz), 115.9 (d, *J* = 22.1 Hz), 112.2, 52.5, 52.4, 36.4, 22.5.

IR (KBr, cm^{-1}): 1748, 1684, 1599, 1510, 1438.

HRMS (ESI) calcd for $C_{14}H_{16}FO_3$ $(M+H)^+$: 251.1078; Found: 251.1083.



Compound **5m** was obtained as colourless oil in 68% yield.

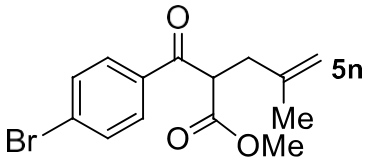
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.95 (d, *J* = 8.7 Hz, 2H), 7.45 (d, *J* = 8.7 Hz, 2H), 4.77 (s, 1H), 4.68 (s, 1H), 4.52 (dd, *J* = 7.6, 7.1 Hz, 1H), 3.67 (s, 3H), 2.75 (dd, *J* = 15.2, 7.8 Hz, 1H), 2.69 (dd, *J* = 15.2, 6.9 Hz, 1H), 1.75 (s, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 193.2, 169.6, 141.8, 140.1, 134.4, 130.0, 129.1, 112.3, 52.6, 52.5, 36.4, 22.6.

IR (KBr, cm^{-1}): 1743, 1691, 1592, 1440.

HRMS (ESI) calcd for $C_{14}H_{16}ClO_3$ $(M+H)^+$: 267.0782; Found: 267.0782.

(Ref: S. Chowdhury, S. Koley, T. Chanda and M. S. Singh, *Tetrahedron Lett.*, 2015, **56**, 5553.)



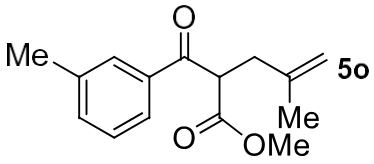
Compound **5n** was obtained as colourless oil in 77% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.86 (ddd, *J* = 8.7, 2.4, 1.9 Hz, 2H), 7.62 (ddd, *J* = 8.7, 2.4, 1.9 Hz, 2H), 4.77 (s, 1H), 4.68 (s, 1H), 4.51 (dd, *J* = 7.5, 7.2 Hz, 1H), 3.67 (s, 3H), 2.74 (dd, *J* = 15.2, 7.7 Hz, 1H), 2.68 (dd, *J* = 15.2, 7.0 Hz, 1H), 1.74 (s, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 193.5, 169.7, 141.8, 134.8, 132.1, 130.1, 128.9, 112.3, 52.6, 52.5, 36.4, 22.6.

IR (KBr, cm^{-1}): 1744, 1689, 1591.

HRMS (ESI) calcd for $C_{14}H_{16}BrO_3$ $(M+H)^+$: 311.0277; Found: 311.0277.



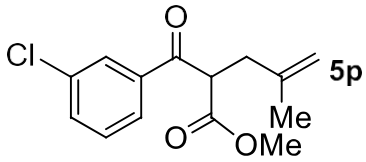
Compound **5o** was obtained as colourless oil in 83% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.81 (s, 1H), 7.80 (d, *J* = 8.5 Hz, 1H), 7.40 (d, *J* = 7.5 Hz, 1H), 7.37 (dd, *J* = 7.5, 7.4 Hz, 1H), 4.77 (s, 1H), 4.71 (s, 1H), 4.57 (dd, *J* = 7.7, 6.9 Hz, 1H), 3.68 (s, 3H), 2.75 (dd, *J* = 15.1, 7.9 Hz, 1H), 2.69 (dd, *J* = 15.1, 6.7 Hz, 1H), 2.42 (s, 3H), 1.76 (s, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 194.7, 170.0, 142.1, 138.7, 136.1, 134.4, 129.1, 128.6, 125.9, 112.2, 52.5, 36.6 (2C), 22.6, 21.4.

IR (KBr, cm^{-1}): 1743, 1686, 1651, 1433, 1152.

HRMS (ESI) calcd for $C_{15}H_{19}O_3$ $(M+H)^+$: 247.1329; Found: 247.1332.



5p

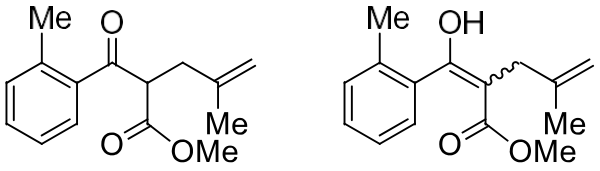
Compound **5p** was obtained as colourless oil in 64% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.96 (dd, *J* = 1.8, 1.8 Hz, 1H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.55 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.42 (dd, *J* = 7.9, 7.9 Hz, 1H), 4.77 (s, 1H), 4.69 (s, 1H), 4.52 (dd, *J* = 7.4, 7.3 Hz, 1H), 3.68 (s, 3H), 2.75 (dd, *J* = 15.1, 7.7 Hz, 1H), 2.69 (dd, *J* = 15.1, 7.0 Hz, 1H), 1.75 (s, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 193.3, 169.6, 141.8, 137.7, 135.2, 133.5, 130.1, 128.6, 126.6, 112.4, 52.62, 52.60, 36.4, 22.6.

IR (KBr, cm^{-1}): 1743, 1695, 1574, 1229.

HRMS (ESI) calcd for $C_{14}H_{16}ClO_3$ $(M+H)^+$: 267.0782; Found: 267.0788.



5q (2:1) **5q'**

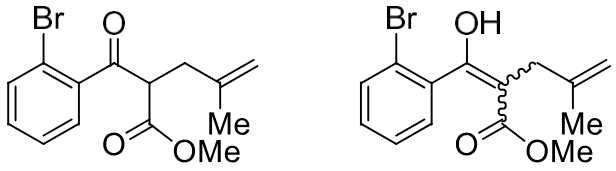
Compound **5q** and **5q'** was obtained as colourless oil in 49% yield.

^{1}H NMR (500 MHz, $CDCl_3$): **5q**/**5q'**, δ 12.90 (s, 0.5H), 7.70 (dd, *J* = 7.8, 0.9 Hz, 1H), 7.38 (ddd, *J* = 7.5, 7.5, 1.3 Hz, 1H), 7.30-7.23 (m, 2.5Hs), 7.21 (dd, *J* = 7.7, 1.5 Hz, 0.5+0.5 H), 7.16 (dd, *J* = 7.6, 7.1 Hz, 0.5H), 4.77 (s, 1H), 4.71 (s, 1H), 4.69-4.66 (m, 0.5H), 4.54-4.51 (m, 0.5H), 4.47 (dd, *J* = 7.5, 7.3 Hz, 1H), 3.80 (s, 1.5H), 3.67 (s, 3H), 2.72-2.64 (m, 3Hs), 2.47 (s, 3H), 2.32 (s, 1.5H), 1.74 (s, 3H), 1.58 (s, 1.5H).

^{13}C NMR (125.8 MHz, $CDCl_3$): **5q**/**5q'**, δ 198.0, 174.0, 172.6, 170.0, 144.5, 141.9, 138.9, 136.9, 135.7, 134.3, 132.0, 131.6, 130.2, 129.1, 128.3, 127.7, 125.6, 125.2, 112.2, 109.5, 99.9, 54.9, 52.3, 51.6, 36.5, 34.1, 22.8, 22.4, 20.9, 19.2.

IR (KBr, cm^{-1}): 3400, 1748, 1693, 1441, 1262.

HRMS (ESI) calcd for $C_{15}H_{19}O_3$ $(M+H)^+$: 247.1329; Found: 247.1329.



5r (1.5: 1) **5r'**

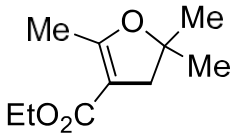
Compound **5r** and **5r'** was obtained as colourless oil in 58% yield.

^{1}H NMR (500 MHz, $CDCl_3$): **5r**/**5r'**, δ 12.82 (s, 0.67H), 7.64-7.59 (m, 1.67Hs), 7.43 (dd, *J* = 7.6, 1.8 Hz, 0.67H), 7.38 (ddd, *J* = 7.6, 7.4, 1.2 Hz, 0.67H), 7.35-7.28 (m, 2.67Hs), 7.27-7.22 (m, 1H), 4.79 (s, 0.67H), 4.74 (s, 0.67H), 4.66 (dd, *J* = 1.5, 1.5 Hz, 1H), 4.54-4.51 (m, 1H), 4.48 (dd, *J* = 9.0, 5.9 Hz, 0.67H), 3.82 (s, 3H), 3.67 (s, 2H), 2.95-2.75 (m, 1H), 2.73 (dd, *J* = 15.0, 9.1 Hz, 1H), 2.65 (dd, *J* = 15.0, 5.9 Hz, 1H), 2.60-2.40 (m, 0.67H), 1.75 (s, 2H), 1.58 (s, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): **5r**/**5r'**, δ 197.9, 173.8, 170.4, 169.3, 144.2, 141.6, 140.2, 135.8, 133.9, 132.9, 132.0, 130.6, 129.7, 128.9, 127.4, 127.0, 121.6, 119.2, 112.6, 109.9, 100.7, 56.4, 52.4, 51.9, 36.1, 34.1, 22.8, 22.5.

IR (KBr, cm^{-1}): 3400, 1743, 1707, 1652, 1443, 1254.

HRMS (ESI) calcd for $C_{14}H_{16}BrO_3$ $(M+H)^+$: 311.0277; Found: 311.0280.



6a

Compound **6a** was obtained as colourless oil.

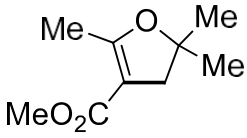
^{1}H NMR (500 MHz, $CDCl_3$): δ 4.15 (q, *J* = 7.1 Hz, 2H), 2.67 (q, *J* = 1.6 Hz, 2H), 2.16 (t, *J* = 1.6 Hz, 3H), 1.38 (s, 6H), 1.27 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 166.7, 166.6, 101.0, 86.0, 59.3, 42.6, 28.3, 14.5, 14.4.

IR (KBr, cm^{-1}): 1700, 1652, 912.

HRMS (ESI) calcd for $C_{10}H_{17}O_3$ $(M+H)^+$: 185.1172; Found: 185.1175.

(Ref: T. Sakai, K. Miyata, S. Tsuboi and M. Utaka, *Bull. Chem. Soc. Jpn.*, 1989, **62**, 4072.)



6b

Compound **6b** was obtained as colourless oil.

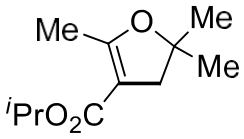
^{1}H NMR (500 MHz, $CDCl_3$): δ 3.69 (s, 3H), 2.67 (q, *J* = 1.6 Hz, 2H), 2.16 (t, *J* = 1.6 Hz, 3H), 1.37 (s, 6H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 167.0, 166.9, 100.7, 86.1, 50.7, 42.6, 28.2, 14.4.

IR (KBr, cm^{-1}): 1688, 1668, 910.

HRMS (ESI) calcd for $C_9H_{15}O_3$ $(M+H)^+$: 171.1016; Found: 171.1021.

(Ref: V. Roland, K. Norbert De, C. Dirk, B. Laurent De and S. Niceas, *Tetrahedron*, 1982, **38**, 3649.)



6c

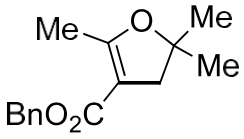
Compound **6c** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 5.04 (sept, *J* = 6.3 Hz, 1H), 2.66 (q, *J* = 1.5 Hz, 2H), 2.16 (t, *J* = 1.5 Hz, 3H), 1.37 (s, 6H), 1.25 (d, *J* = 6.3 Hz, 6H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 166.3, 166.2, 101.4, 85.8, 66.4, 42.7, 28.3, 22.2, 14.5.

IR (KBr, cm^{-1}): 1743, 1691, 1646, 1276, 1263, 1142, 1110, 767.

HRMS (ESI) calcd for $C_{11}H_{19}O_3$ $(M+H)^+$: 199.1329; Found: 199.1330.



6d

Compound **6d** was obtained as colourless oil.

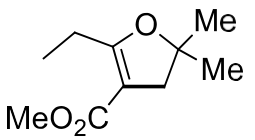
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40-7.34 (m, 4H), 7.34-7.27 (m, 1H), 5.16 (s, 2H), 2.71 (q, *J* = 1.5 Hz, 2H), 2.18 (t, *J* = 1.5 Hz, 3H), 1.38 (s, 6H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 167.4, 166.2, 136.8, 128.5, 127.9, 127.8, 100.7, 86.3, 65.2, 42.5, 28.3, 14.5.

IR (KBr, cm^{-1}): 1716, 1633, 1273, 1264.

HRMS (ESI) calcd for $C_{15}H_{19}O_3$ $(M+H)^+$: 247.1329; Found: 247.1335.

(Ref: S. Kitagaki, D. Shibata and C. Mukai, *Tetrahedron Lett.*, 2007, **48**, 1735.)



6e

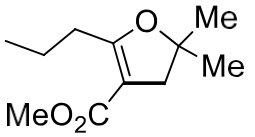
Compound **6e** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 3.69 (s, 3H), 2.66 (s, 2H), 2.61 (q, *J* = 7.6 Hz, 2H), 1.37 (s, 6H), 1.10 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 171.9, 166.8, 99.6, 85.9, 50.7, 42.6, 28.1, 21.4, 11.1.

IR (KBr, cm^{-1}): 1637, 1278, 1266, 1079, 762, 753.

HRMS (ESI) calcd for $C_{10}H_{17}O_3$ $(M+H)^+$: 185.1172; Found: 185.1173.



6f

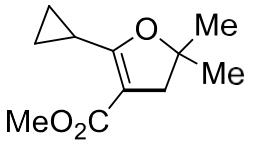
Compound **6f** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 3.69 (s, 3H), 2.67 (s, 2H), 2.59 (t, *J* = 7.5 Hz, 2H), 1.60-1.54 (m, 2H), 1.37 (s, 6H), 0.94 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 170.7, 166.8, 100.4, 85.9, 50.6, 42.6, 29.7, 28.2, 20.2, 13.7.

IR (KBr, cm^{-1}): 1653, 1643, 1278, 1262, 917.

HRMS (ESI) calcd for $C_{11}H_{19}O_3$ $(M+H)^+$: 199.1329; Found: 199.1329.



6g

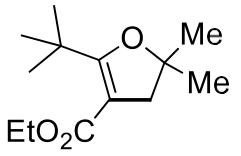
Compound **6g** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 3.71 (s, 3H), 2.73-2.67 (m, 1H), 2.67 (s, 2H), 1.30 (s, 6H), 0.96-0.91 (m, 2H), 0.85-0.80 (m, 2H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 170.7, 167.4, 100.0, 85.8, 50.6, 42.9, 27.9, 9.2, 7.2.

IR (KBr, cm^{-1}): 1749, 1630, 1413, 1385, 1098.

HRMS (ESI) calcd for $C_{11}H_{17}O_3$ $(M+H)^+$: 197.1172; Found: 197.1172.



6h

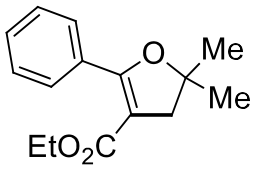
Compound **6h** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 4.11 (q, *J* = 7.2 Hz, 2H), 2.70 (s, 2H), 1.33 (s, 6H), 1.27 (s, 9H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 176.2, 165.8, 98.6, 84.0, 59.2, 44.6, 27.9, 27.5, 26.4, 14.4.

IR (KBr, cm^{-1}): 1735, 1644, 1264, 1276, 767, 751.

HRMS (ESI) calcd for $C_{13}H_{23}O_3$ $(M+H)^+$: 227.1642; Found: 227.1646.



6i

Compound **6i** was obtained as colourless oil.

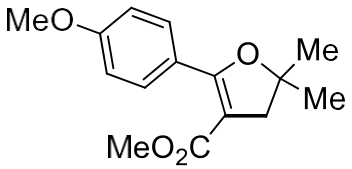
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.74 (dd, *J* = 7.6, 1.5 Hz, 2H), 7.43-7.33 (m, 3H), 4.12 (q, *J* = 7.2 Hz, 2H), 2.92 (s, 2H), 1.49 (s, 6H), 1.20 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 165.7, 164.0, 130.6, 130.1, 129.2, 127.5, 101.6, 85.5, 59.6, 44.3, 28.2, 14.2.

IR (KBr, cm^{-1}): 1642, 1628, 1492, 1262.

HRMS (ESI) calcd for $C_{15}H_{19}O_3$ $(M+H)^+$: 247.1329; Found: 247.1334.

(Ref: T. Sakai, K. Miyata, S. Tsuboi and M. Utaka, *Bull. Chem. Soc. Jpn.*, 1989, **62**, 4072.)



6j

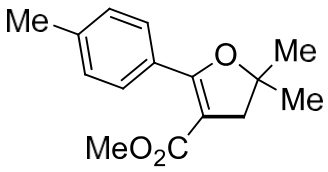
Compound **6j** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.78 (dt, *J* = 9.0, 2.8 Hz, 2H), 6.89 (dt, *J* = 9.0, 2.8 Hz, 2H), 3.83 (s, 3H), 3.67 (s, 3H), 2.90 (s, 2H), 1.47 (s, 6H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 166.2, 164.2, 161.1, 131.0, 122.8, 113.0, 99.9, 85.1, 55.3, 50.8, 44.3, 28.1.

IR (KBr, cm^{-1}): 1702, 1688, 1609, 1512, 1369, 1254.

HRMS (ESI) calcd for $C_{15}H_{19}O_4$ $(M+H)^+$: 263.1278; Found: 263.1274.



6k

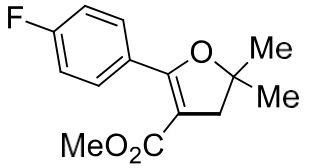
Compound **6k** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.66 (d, *J* = 8.2 Hz, 2H), 7.18 (d, *J* = 8.2 Hz, 2H), 3.66 (s, 3H), 2.91 (s, 2H), 2.37 (s, 3H), 1.48 (s, 6H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 166.1, 164.6, 140.4, 129.1, 128.3, 127.6, 100.6, 85.4, 50.8, 44.2, 28.1, 21.5.

IR (KBr, cm^{-1}): 1713, 1629.

HRMS (ESI) calcd for $C_{15}H_{19}O_3$ $(M+H)^+$: 247.1329; Found: 247.1329.



6l

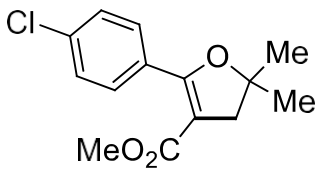
Compound **6l** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.84-7.77 (m, 2H), 7.10-7.03 (m, 2H), 3.66 (s, 3H), 2.91 (s, 2H), 1.48 (s, 6H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 166.0, 163.7 (d, *J* = 250.5 Hz), 163.2, 131.5 (d, *J* = 8.5 Hz), 126.5 (d, *J* = 3.1 Hz), 114.7 (d, *J* = 21.7 Hz), 101.1, 85.6, 50.9, 44.2, 28.1.

IR (KBr, cm^{-1}): 1663, 1633, 1504, 1273, 1084.

HRMS (ESI) calcd for $C_{14}H_{16}FO_3$ $(M+H)^+$: 251.1078; Found: 251.1078.



6m

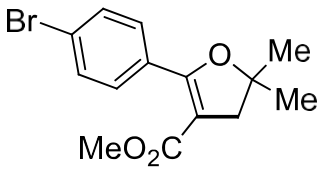
Compound **6m** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.73 (d, *J* = 8.6 Hz, 2H), 7.35 (d, *J* = 8.6 Hz, 2H), 3.66 (s, 3H), 2.91 (s, 2H), 1.48 (s, 6H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 165.9, 163.0, 136.2, 130.7, 128.8, 127.9, 101.7, 85.8, 51.0, 44.2, 28.1.

IR (KBr, cm^{-1}): 1703, 1693, 1375.

HRMS (ESI) calcd for $C_{14}H_{16}ClO_3$ $(M+H)^+$: 267.0782; Found: 267.0786.



6n

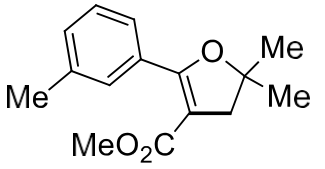
Compound **6n** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.66 (ddd, *J* = 8.6, 2.4, 1.9 Hz, 2H), 7.51 (ddd, *J* = 8.6, 2.3, 1.9 Hz, 2H), 3.66 (s, 3H), 2.91 (s, 2H), 1.48 (s, 6H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 165.9, 163.0, 130.9, 130.8, 129.3, 124.6, 101.8, 85.8, 51.0, 44.2, 28.1.

IR (KBr, cm^{-1}): 1705, 1663, 1632.

HRMS (ESI) calcd for $C_{14}H_{16}BrO_3$ $(M+H)^+$: 311.0277; Found: 311.0281.



6o

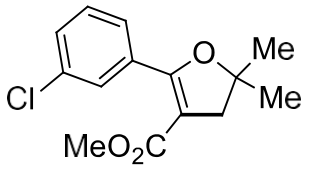
Compound **6o** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.55 (d, *J* = 7.6 Hz, 1H), 7.52 (s, 1H), 7.29-7.24 (m, 1H), 7.21 (d, *J* = 7.4 Hz, 1H), 3.65 (d, *J* = 0.6 Hz, 3H), 2.91 (s, 2H), 2.37 (s, 3H), 1.49 (s, 6H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 166.1, 164.6, 137.3, 131.0, 130.4, 129.5, 127.5, 126.5, 101.2, 85.6, 50.9, 44.2, 28.1, 21.4.

IR (KBr, cm^{-1}): 1707, 1688, 1629, 1598, 1269, 1128.

HRMS (ESI) calcd for $C_{15}H_{19}O_3$ $(M+H)^+$: 247.1329; Found: 247.1329.



6p

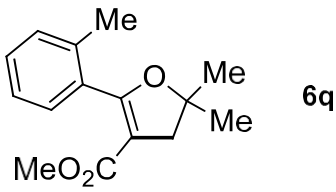
Compound **6p** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.74 (dd, *J* = 1.7, 1.6 Hz, 1H), 7.69 (d, *J* = 7.7 Hz, 1H), 7.39-7.35 (m, 1H), 7.31 (dd, *J* = 7.9, 7.8 Hz, 1H), 3.67 (s, 3H), 2.92 (s, 2H), 1.49 (s, 6H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 165.7, 162.4, 133.6, 132.2, 130.2, 129.2, 128.9, 127.5, 102.3, 85.9, 51.0, 44.2, 28.1.

IR (KBr, cm^{-1}): 1705, 1626, 1595, 1566, 1262.

HRMS (ESI) calcd for $C_{14}H_{16}ClO_3$ $(M+H)^+$: 267.0782; Found: 267.0780.



6q

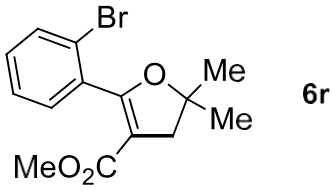
Compound **6q** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.31-7.26 (m, 2H), 7.23-7.17 (m, 2H), 3.55 (s, 3H), 2.91 (s, 2H), 2.32 (s, 3H), 1.51 (s, 6H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 165.8, 165.6, 136.6, 131.0, 129.9, 129.4, 129.2, 125.2, 103.1, 86.8, 50.8, 43.1, 28.3, 19.4.

IR (KBr, cm^{-1}): 1693, 1646, 1364, 1280.

HRMS (ESI) calcd for $C_{15}H_{19}O_3$ $(M+H)^+$: 247.1329; Found: 247.1334.



6r

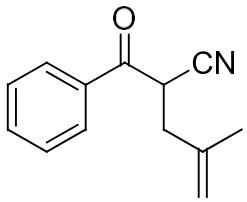
Compound **6r** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.59 (d, *J* = 8.1 Hz, 1H), 7.35-7.31 (m, 2H), 7.26-7.22 (m, 1H), 3.55 (s, 3H), 2.91 (s, 2H), 1.54 (s, 6H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 165.5, 163.5, 133.0, 132.7, 130.7 (2C), 126.9, 122.3, 104.3, 87.8, 50.9, 43.0, 28.3.

IR (KBr, cm^{-1}): 1695, 1651.

HRMS (ESI) calcd for $C_{14}H_{16}BrO_3$ $(M+H)^+$: 311.0277; Found: 311.0277.



7

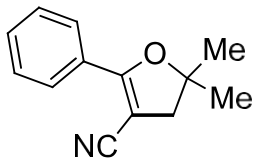
Compound **7** was obtained as colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 (dd, *J* = 8.6, 1.3 Hz, 2H), 7.66 (ddd, *J* = 7.5, 7.5, 1.2 Hz, 1H), 7.54 (dd, *J* = 8.1, 7.6 Hz, 2H), 4.97 (s, 1H), 4.89 (s, 1H), 4.48 (dd, *J* = 9.2, 5.7 Hz, 1H), 2.74 (dd, *J* = 14.8, 5.7 Hz, 1H), 2.68 (dd, *J* = 14.8, 9.1 Hz, 1H), 1.84 (s, 3H).

^{13}C NMR (125.8 MHz, $CDCl_3$): δ 190.2, 139.6, 134.5, 134.0, 129.1, 128.8, 117.0, 114.6, 38.4, 37.3, 22.3.

IR (KBr, cm^{-1}): 1696, 1658, 1455, 1273, 1260, 753.

HRMS (ESI) calcd for $C_{13}H_{14}NO$ $(M+H)^+$: 200.1070; Found: 200.1073.



8

Compound **8** was obtained as colourless oil.

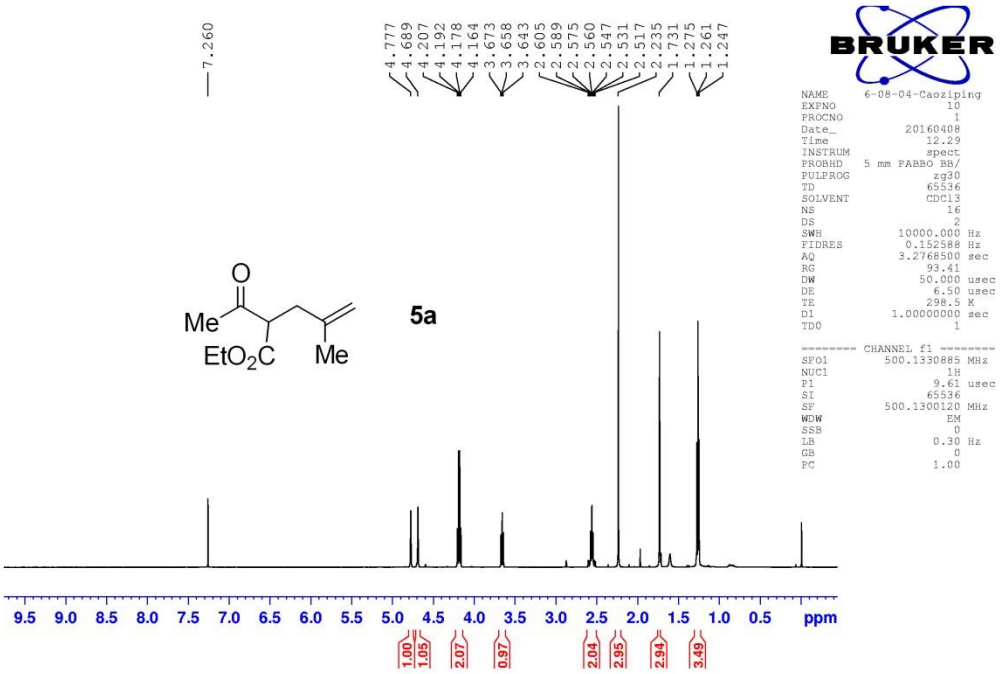
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.93 (dd, *J* = 8.2, 1.4 Hz, 2H), 7.48-7.40 (m, 3H), 2.88 (s, 2H), 1.51 (s, 6H).

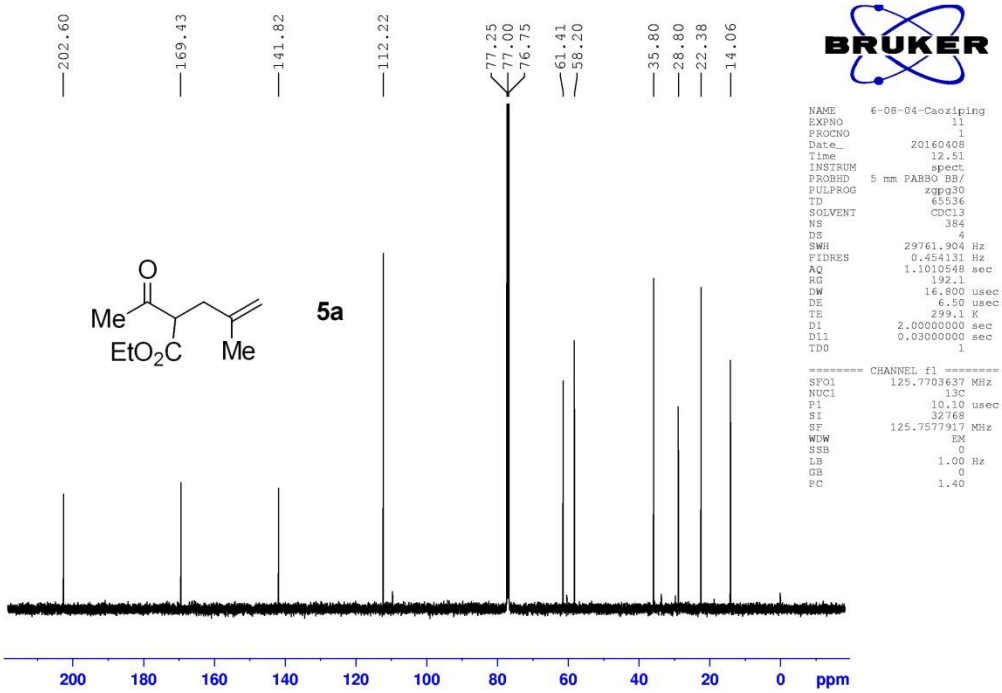
^{13}C NMR (125.8 MHz, $CDCl_3$): δ 165.8, 131.1, 128.6 (2C), 127.1, 118.2, 87.6, 78.2, 44.3, 28.0.

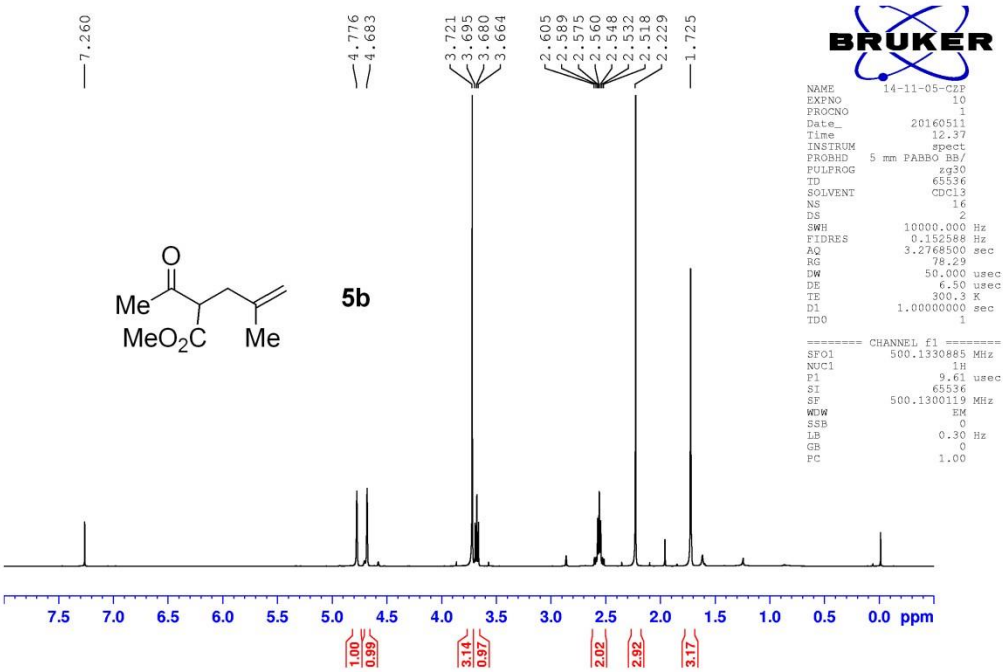
IR (KBr, cm^{-1}): 1648, 1630, 1422, 1098.

HRMS (ESI) calcd for $C_{13}H_{14}NO$ $(M+H)^+$: 200.1070; Found: 200.1070.

5- Copies of NMR spectra

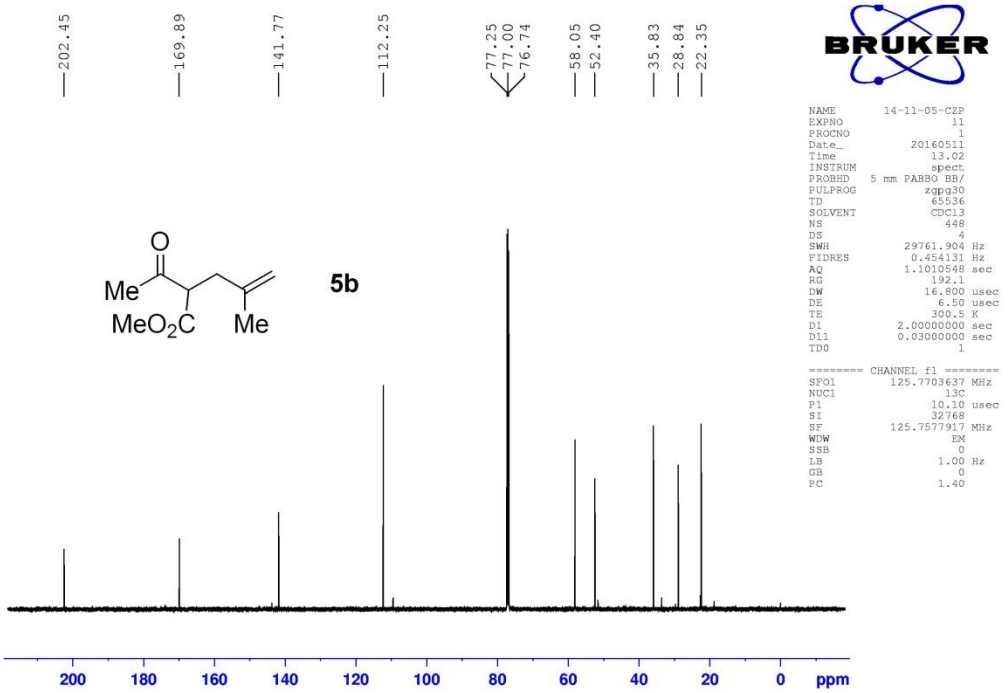






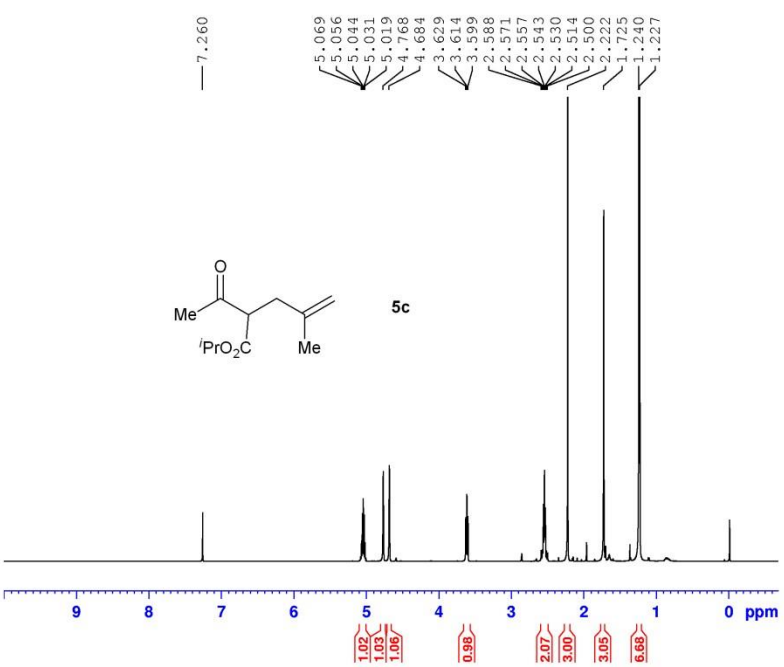

```
NAME            14-11-05-CZP
EXPNO                     10
PROCNO                     1
Date_               20160511
Time                   12.37
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               10000.000 Hz
FIDRES                0.152588 Hz
AQ                3.2768500 sec
RG                    78.29
DW                   50.000 usec
DE                    6.50 usec
TE                   300.3 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             500.1330885 MHz
NUC1                       1H
P1                       9.61 usec
SI                      65536
SF               500.1300119 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```




```
NAME            14-11-05-CZP
EXPNO                     11
PROCNO                     1
Date_               20160511
Time                   13.02
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      448
DS                        4
SWH               29761.904 Hz
FIDRES                0.454131 Hz
AQ                1.1010548 sec
RG                    192.1
DW                   16.800 usec
DE                    6.50 usec
TE                   300.5 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             125.7703637 MHz
NUC1                       13C
P1                      10.10 usec
SI                      32768
SF               125.7577917 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```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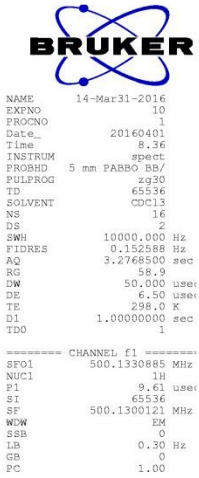
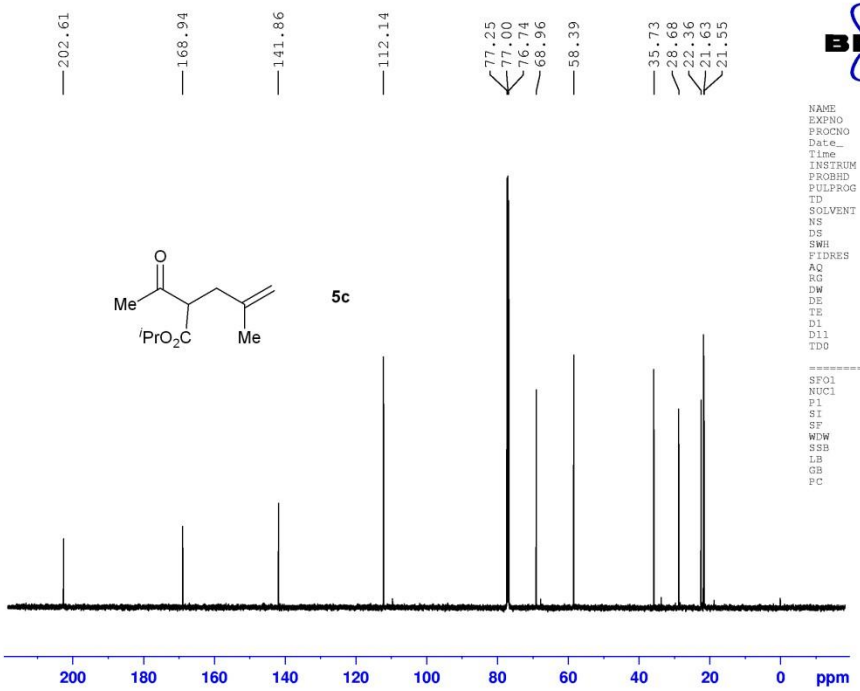
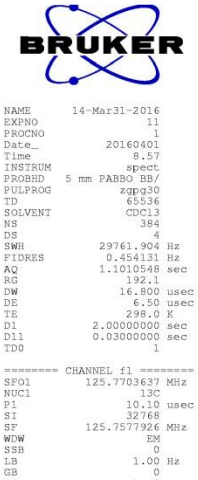
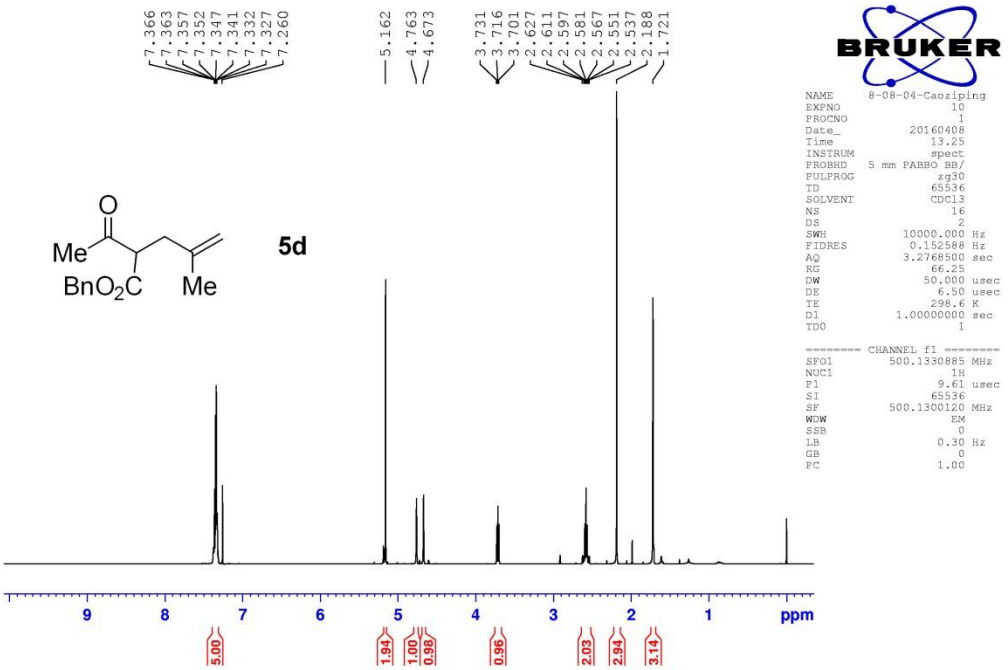
5.069
5.056
5.044
5.031
5.019
4.768
4.684
3.629
3.614
3.599
2.588
2.571
2.557
2.543
2.530
2.514
2.500
2.222
1.725
1.240
1.227
O
Me
iPrO2C
Me
5c
9
8
7
6
5
4
3
2
1
0 ppm
1.02
1.03
1.06
0.98
2.07
3.00
3.05
6.68


BRUKER
NAME 14-Mar31-2016
EXPNO 10
PROCNO 1
Date_ 20160401
Time 8.36
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 58.9
DW 50.000 use
DE 6.50 use
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 use
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


202.61
168.94
141.86
112.14
77.25
77.00
76.74
68.96
58.39
35.73
28.68
22.36
21.63
21.55
O
Me
iPrO2C
Me
5c
200
180
160
140
120
100
80
60
40
20
0 ppm


BRUKER
NAME 14-Mar31-2016
EXPNO 11
PROCNO 1
Date_ 20160401
Time 8.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 384
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577926 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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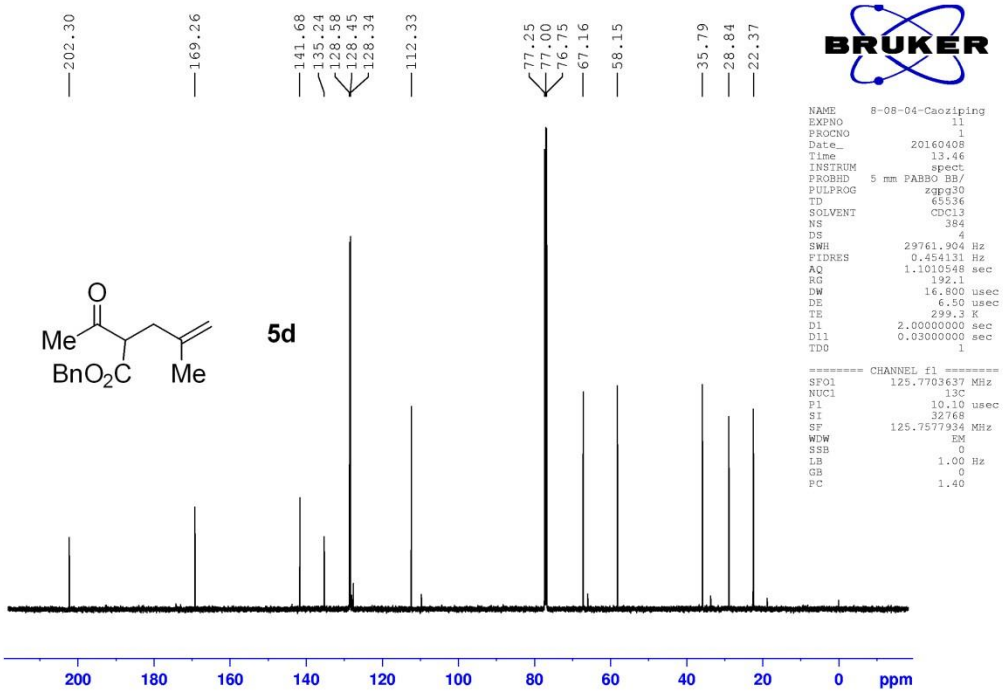
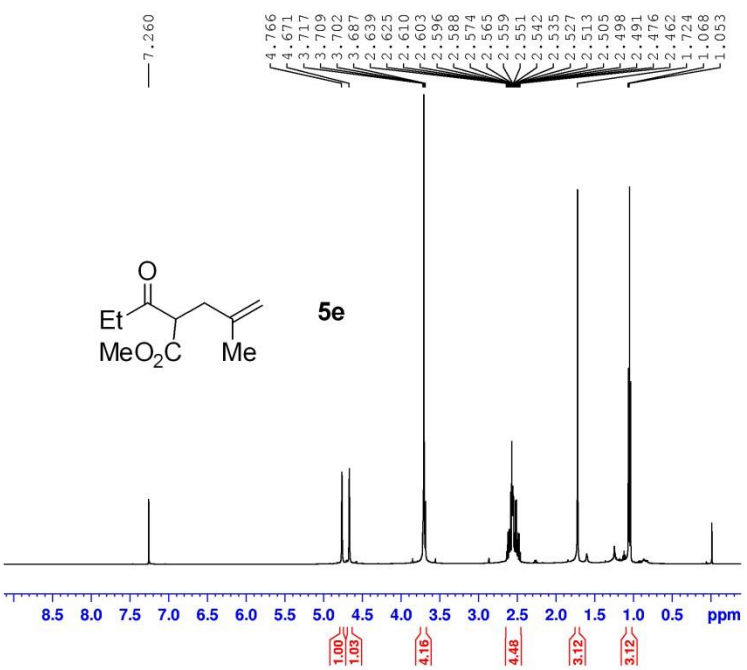
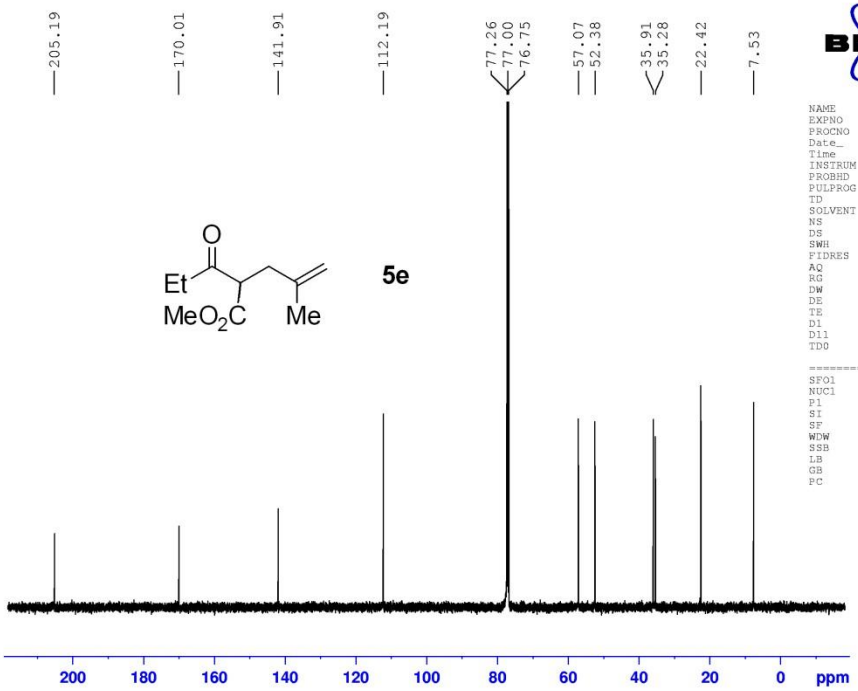
7.363
7.357
7.352
7.347
7.341
7.332
7.327
7.260
5.162
4.763
4.673
3.731
3.716
3.701
2.627
2.611
2.597
2.581
2.567
2.551
2.537
2.188
1.721
5d
Me
BnO2C
Me
O
BRUKER
NAME 8-08-04-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160408
Time 13.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 66.25
DW 50.000 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
5.00
1.94
1.00
0.98
0.96
2.03
2.94
3.14


202.30
169.26
141.68
135.24
128.58
128.45
128.34
112.33
77.25
77.00
76.75
67.16
58.15
35.79
28.84
22.37
5d
Me
BnO2C
Me
O
BRUKER
NAME 8-08-04-Caoziping
EXPNO 11
PROCNO 1
Date_ 20160408
Time 13.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 384
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577934 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm



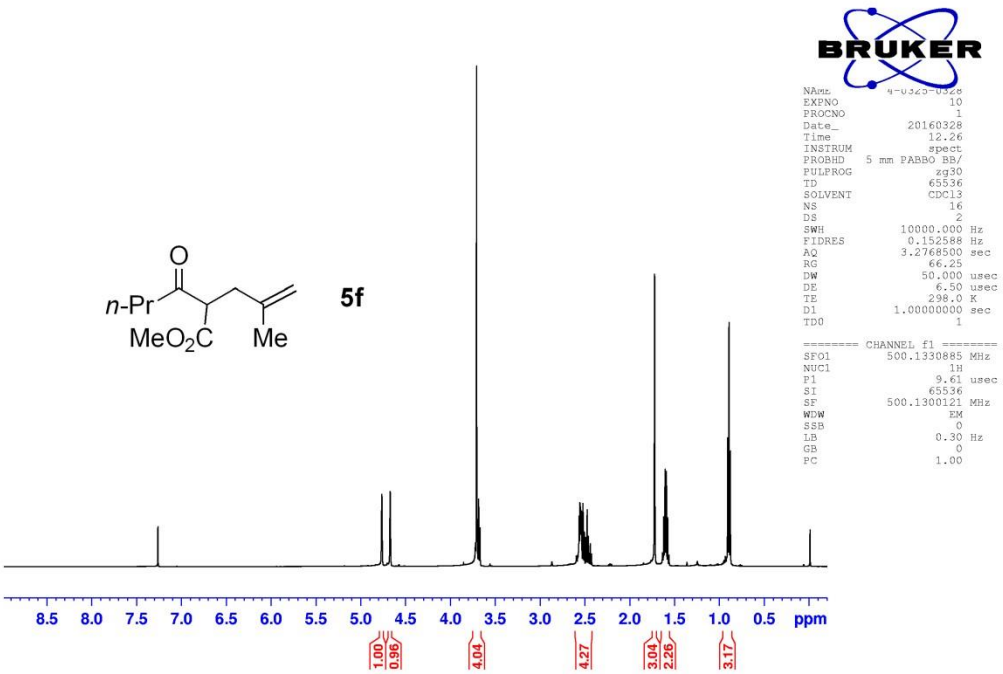
```
NAME          17-23-03-Caoziping
EXPNO                         10
PROCNO                         1
Date_                   20160323
Time                        9.51
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                   10000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2768500 sec
RG                        66.25
DW                       50.000 usec
DE                        6.50 usec
TE                       297.4 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                500.1330885 MHz
NUC1                        1H
P1                        9.90 usec
SI                       65536
SF                  500.1300119 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```



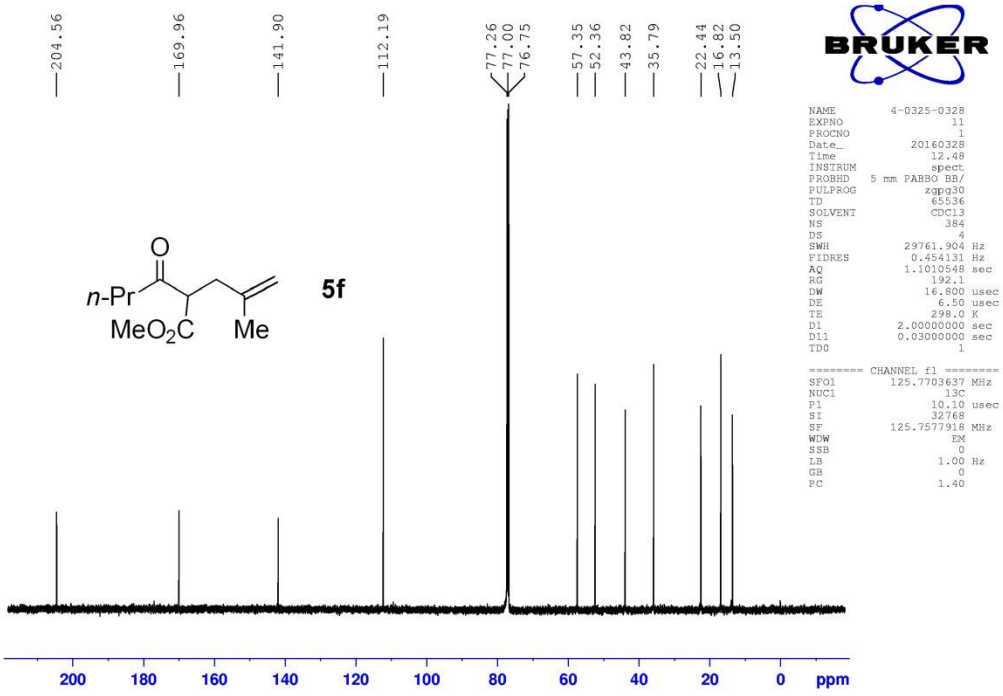
```
NAME          17-23-03-Caoziping
EXPNO                         11
PROCNO                         1
Date_                   20160323
Time                       10.05
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          256
DS                            4
SWH                   29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010548 sec
RG                        192.1
DW                       16.800 usec
DE                        6.50 usec
TE                       298.5 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                125.7703637 MHz
NUC1                        13C
P1                       10.00 usec
SI                       32768
SF                  125.7577919 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```



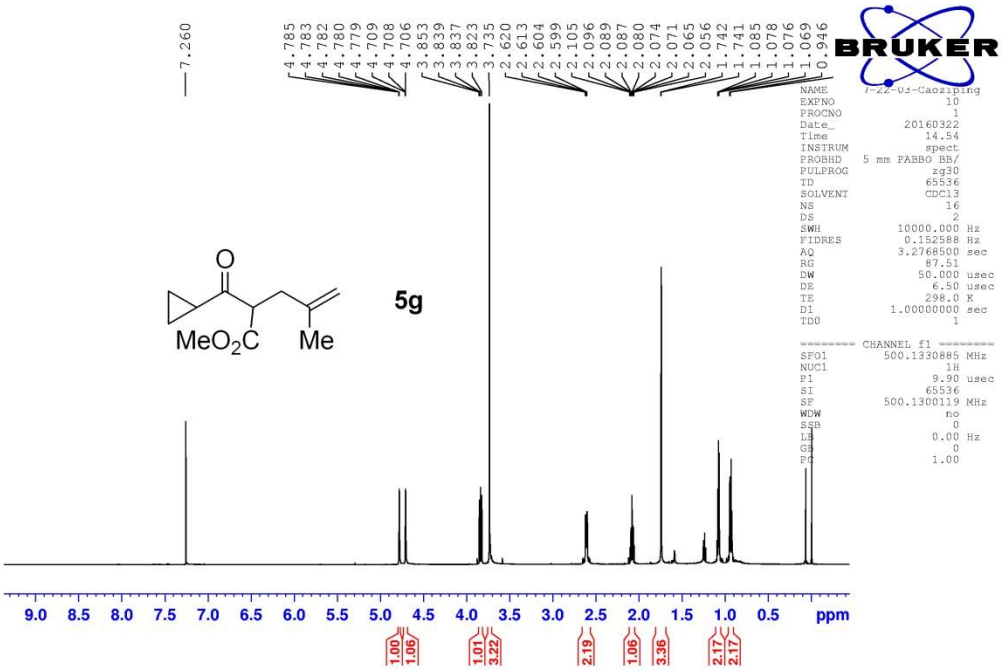

```
NAme                4-0325-0328
EXPNO                        10
PROCNO                        1
Date_                  20160328
Time                      12.26
INSTRUM                   spect
PROBHD   5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                10000.000 Hz
FIDRES                 0.152588 Hz
AQ                 3.2768500 sec
RG                       66.25
DW                      50.000 usec
DE                        6.50 usec
TE                      298.0 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               500.1330885 MHz
NUC1                        1H
P1                        9.61 usec
SI                       65536
SF                 500.1300121 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```




```
NAME                4-0325-0328
EXPNO                        11
PROCNO                        1
Date_                  20160328
Time                      12.48
INSTRUM                   spect
PROBHD   5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         384
DS                           4
SWH                29761.904 Hz
FIDRES                 0.454131 Hz
AQ                 1.1010548 sec
RG                       192.1
DW                      16.800 usec
DE                        6.50 usec
TE                      298.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               125.7703637 MHz
NUC1                        13C
P1                       10.10 usec
SI                       32768
SF                 125.7577918 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```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4.782
4.780
4.779
4.709
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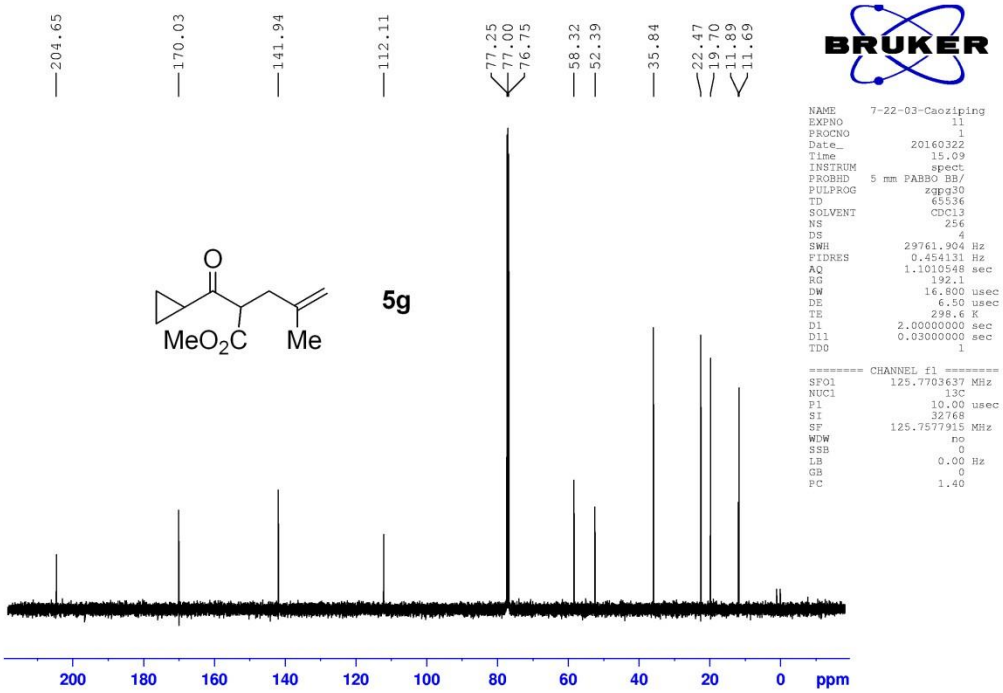
4.706
3.853
3.839
3.837
3.823
3.735
2.620
2.613
2.604
2.599
2.105
2.096
2.089
2.087
2.080
2.074
2.071
2.065
2.056
1.742
1.741
1.085
1.078
1.076
1.069
0.946
BRUKER
NAME 7-22-03-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160322
Time 14.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 87.51
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.90 usec
SI 65536
SF 500.1300119 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
O
MeO2C
Me
5g
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 ppm
1.00
1.06
1.01
3.22
2.19
1.06
3.36
2.17
2.17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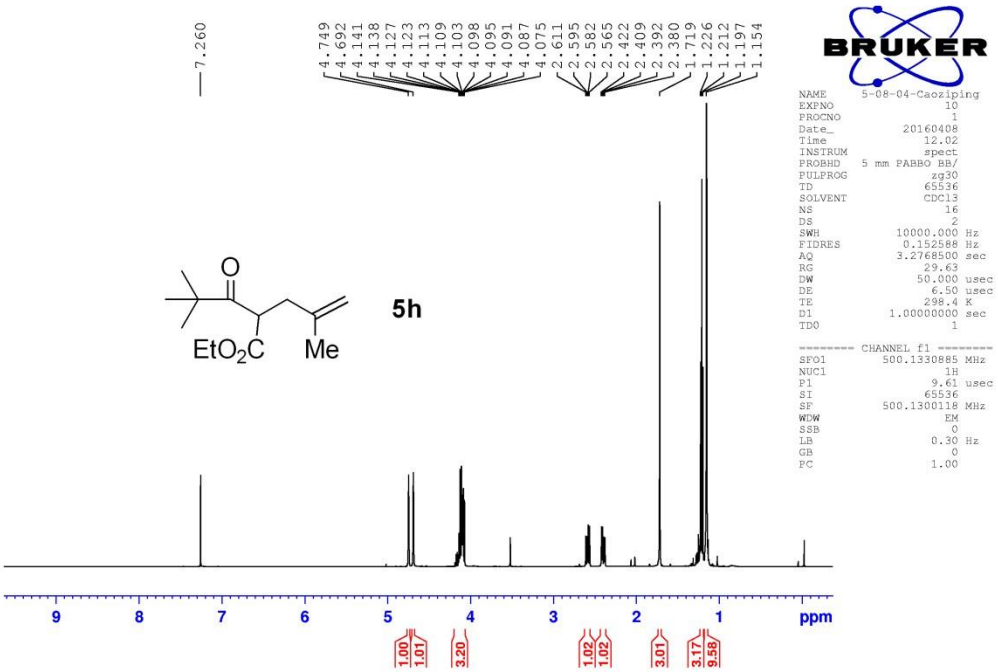
112.11
77.25
77.00
76.75
58.32
52.39
35.84
22.47
19.70
11.89
11.69
BRUKER
NAME 7-22-03-Caoziping
EXPNO 11
PROCNO 1
Date_ 20160322
Time 15.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 125.7577915 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
O
MeO2C
Me
5g
200 180 160 140 120 100 80 60 40 20 0 ppm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4.087
4.075
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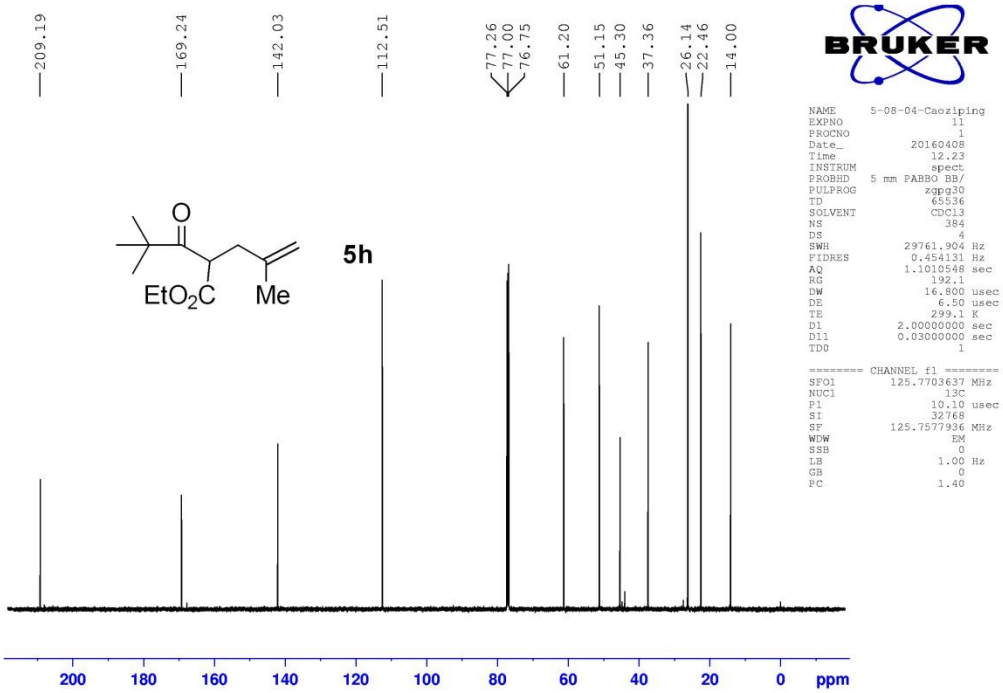
2.595
2.582
2.565
2.422
2.409
2.392
2.380
1.719
1.226
1.212
1.197
1.154
O
EtO2C
Me
5h
BRUKER
NAME 5-08-04-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160408
Time 12.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 29.63
DW 50.000 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
3.20
1.02
1.02
3.01
3.17
9.58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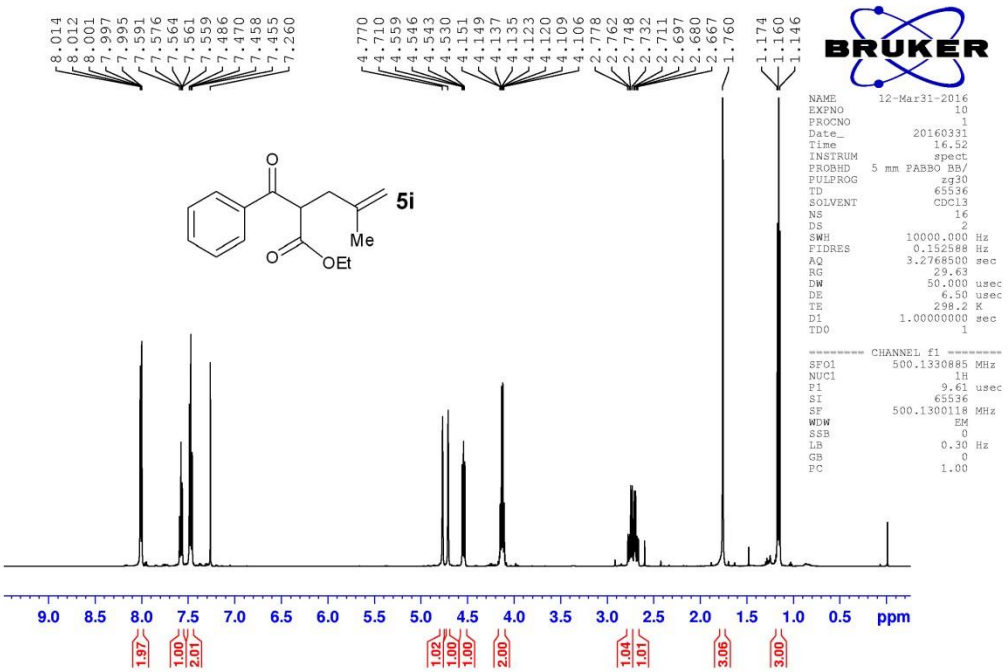
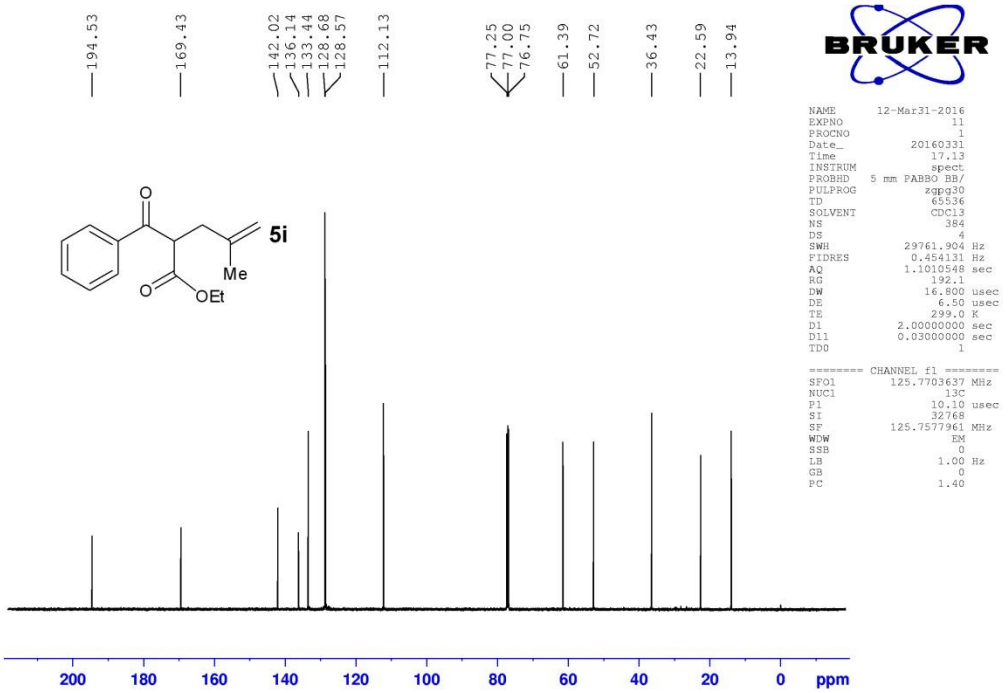
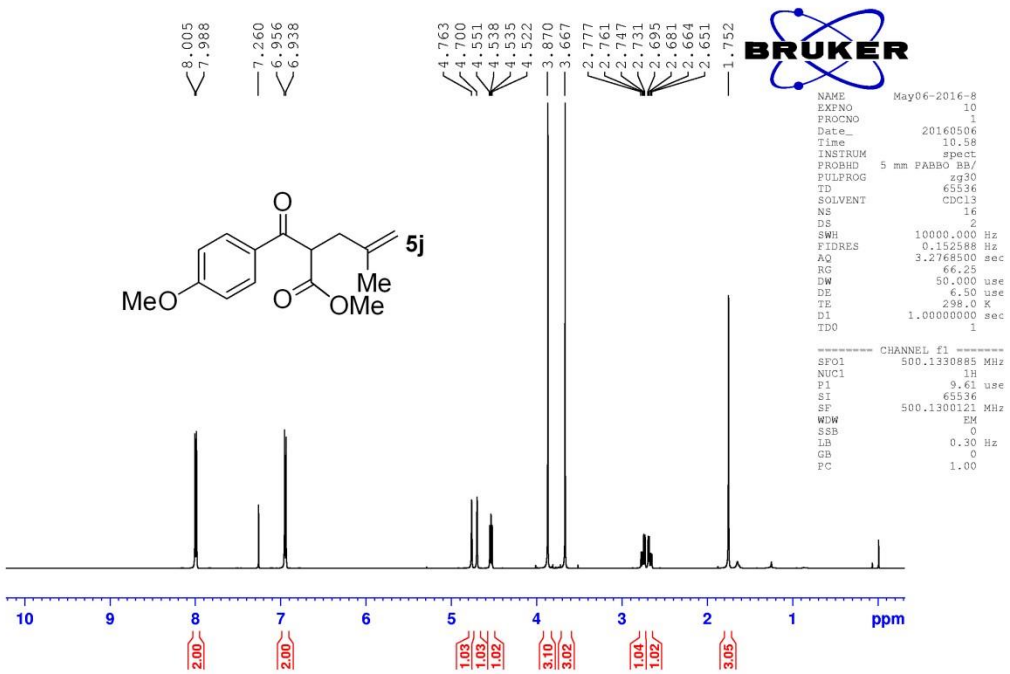
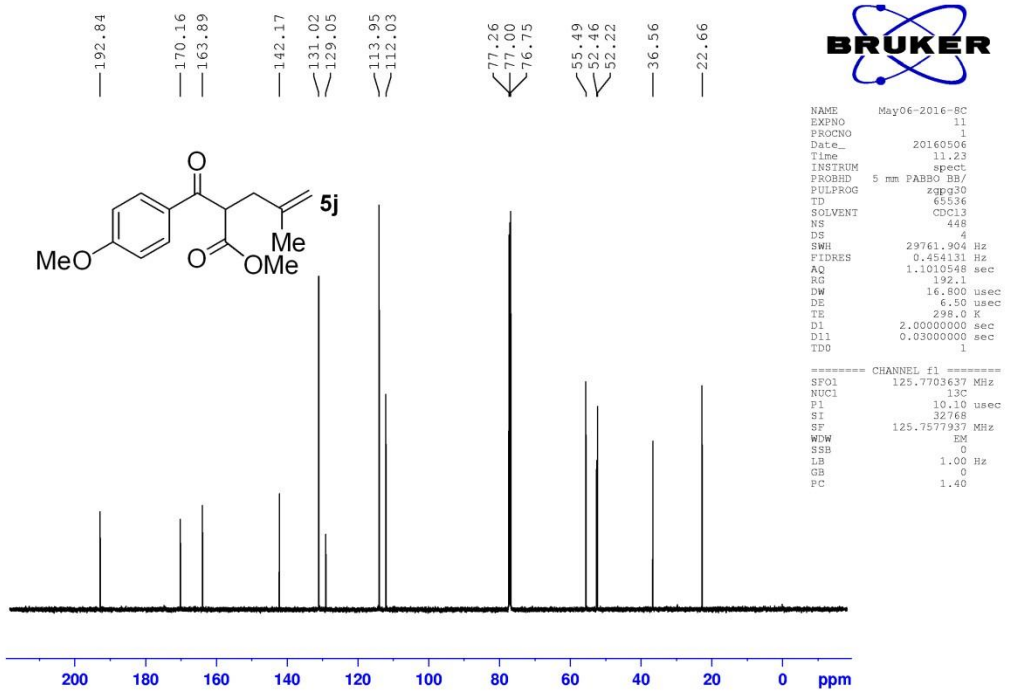
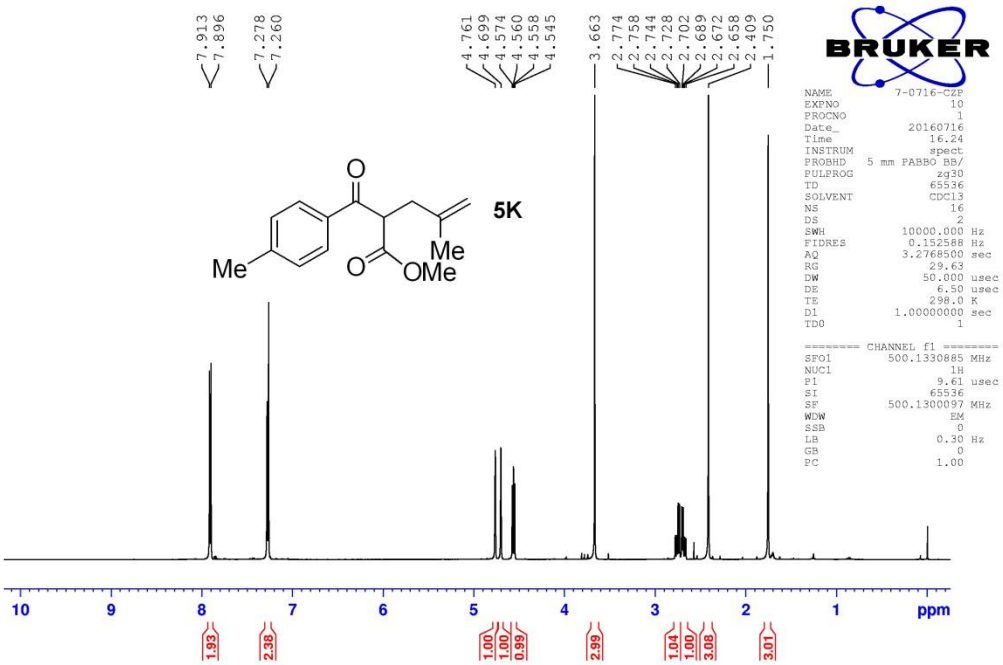
112.51
77.26
77.00
76.75
61.20
51.15
45.30
37.36
26.14
22.46
14.00
O
EtO2C
Me
5h
BRUKER
NAME 5-08-04-Caoziping
EXPNO 11
PROCNO 1
Date_ 20160408
Time 12.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 384
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 299.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577936 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

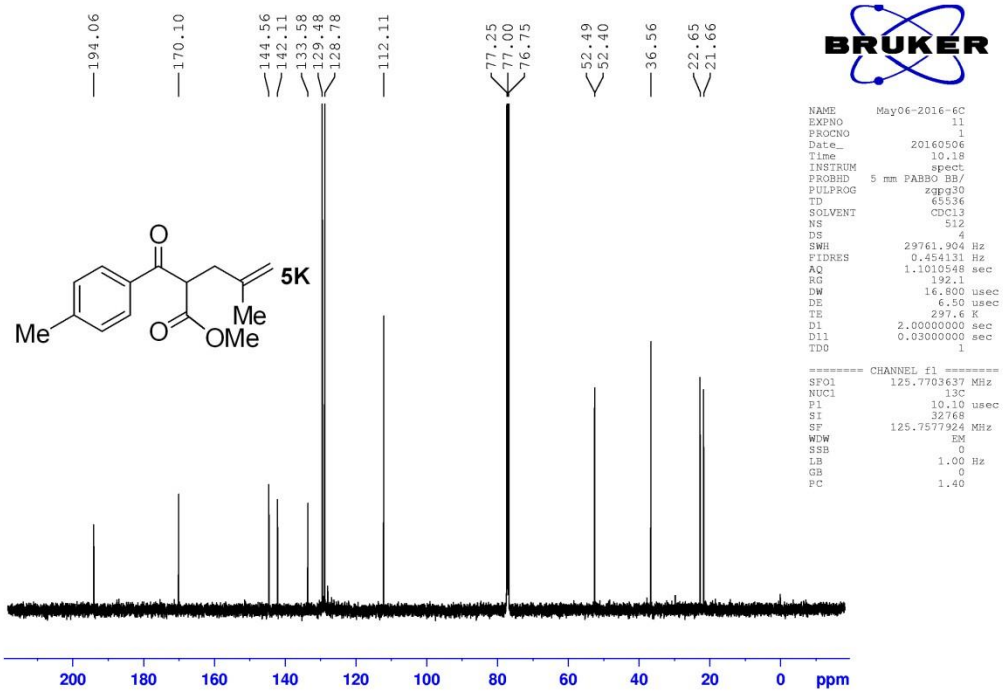

8.014
8.012
8.001
7.997
7.995
7.591
7.576
7.564
7.561
7.559
7.486
7.470
7.458
7.455
7.260
4.770
4.710
4.559
4.546
4.543
4.530
4.151
4.149
4.137
4.135
4.123
4.120
4.109
4.106
2.778
2.762
2.748
2.732
2.711
2.697
2.680
2.667
1.760
1.174
1.160
1.146
5i
Me
OEt
BRUKER
NAME 12-Mar31-2016
EXPNO 10
PROCNO 1
Date_ 20160331
Time 16.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 29.63
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
1.97 1.00 2.01 1.02 1.00 1.00 2.00 1.04 1.01 3.06 3.00

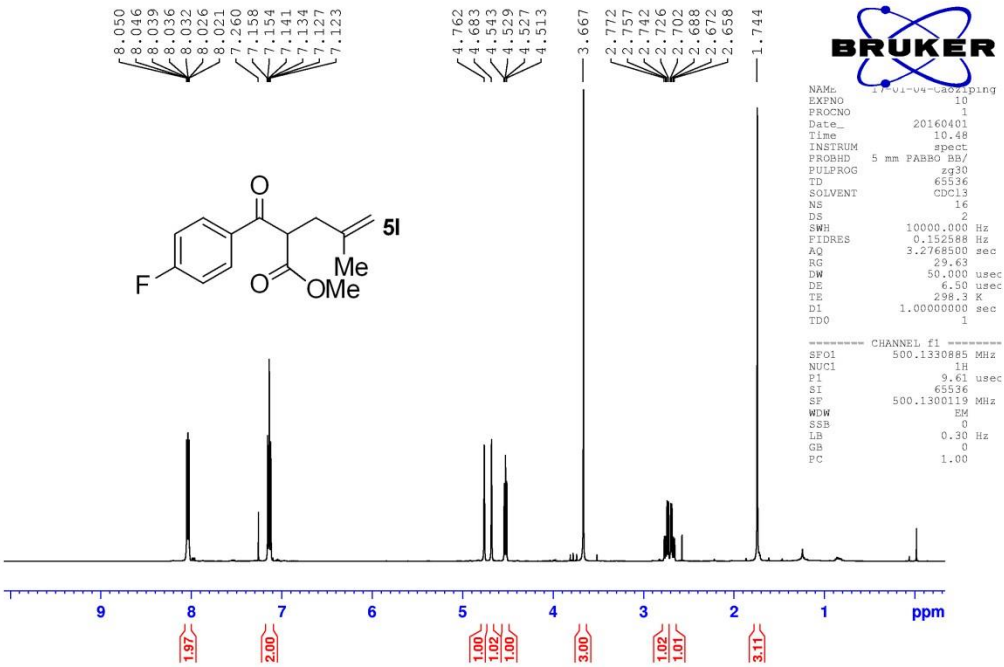

194.53
169.43
142.02
136.14
133.44
128.68
128.57
112.13
77.25
77.00
76.75
61.39
52.72
36.43
22.59
13.94
5i
Me
OEt
BRUKER
NAME 12-Mar31-2016
EXPNO 11
PROCNO 1
Date_ 20160331
Time 17.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 384
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577961 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm


8.005
7.988
7.260
6.956
6.938
4.763
4.700
4.551
4.538
4.535
4.522
3.870
3.667
2.777
2.761
2.747
2.731
2.695
2.681
2.664
2.651
1.752
5j
MeO
O
OMe
Me
NAME May06-2016-8
EXPNO 10
PROCNO 1
Date_ 20160506
Time 10.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 66.25
DW 50.000 use
DE 6.50 use
TE 298.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 use
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 ppm
2.00 2.00 1.03 1.03 1.02 3.10 3.02 1.04 1.02 3.05

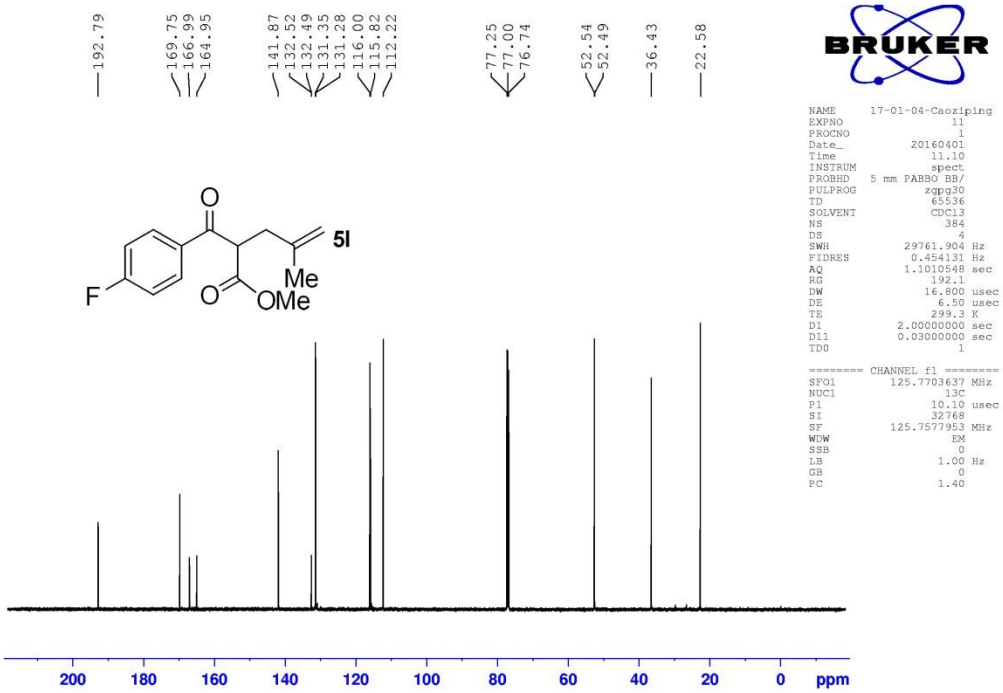

192.84
170.16
163.89
142.17
131.02
129.05
113.95
112.03
77.26
77.00
76.75
55.49
52.46
52.22
36.56
22.66
5j
MeO
O
OMe
Me
NAME May06-2016-8C
EXPNO 11
PROCNO 1
Date_ 20160506
Time 11.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 448
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577937 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm


7.913
7.896
7.278
7.260
4.761
4.699
4.574
4.560
4.558
4.545
3.663
2.774
2.758
2.744
2.728
2.702
2.689
2.672
2.658
2.409
1.750
5K
Me
O
OMe
Me
BRUKER
NAME 7-0716-CZP
EXPNO 10
PROCNO 1
Date_ 20160716
Time 16.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 29.63
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.93
2.38
1.00
1.00
0.99
2.99
1.04
1.00
3.08
3.01

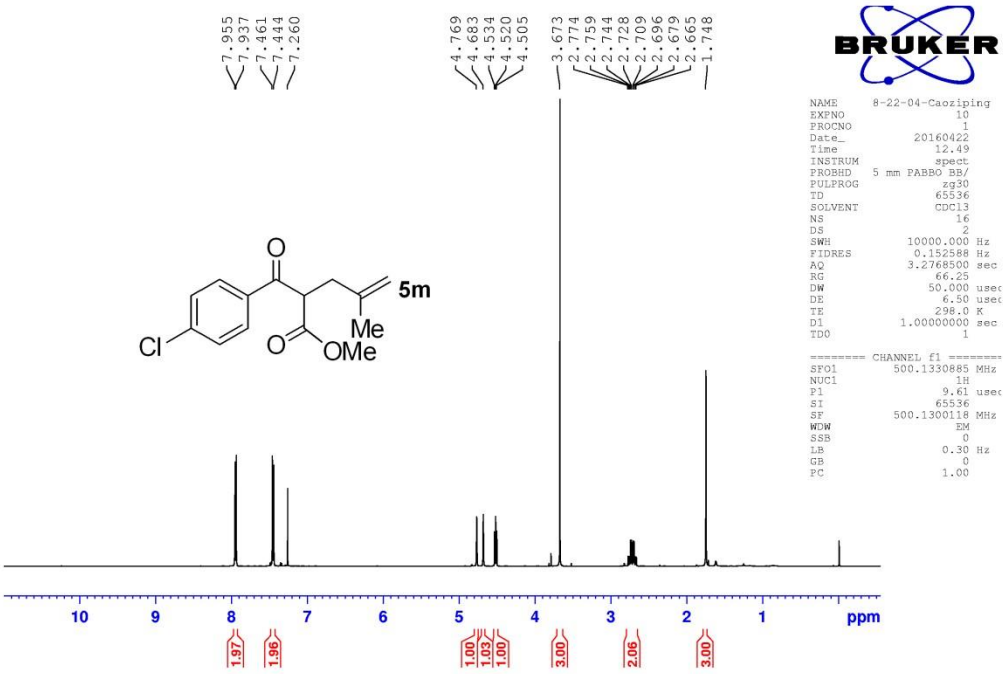

194.06
170.10
144.56
142.11
133.58
129.48
128.78
112.11
77.25
77.00
76.75
52.49
52.40
36.56
22.65
21.66
5K
Me
O
OMe
Me
BRUKER
NAME May06-2016-6C
EXPNO 11
PROCNO 1
Date_ 20160506
Time 10.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577924 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

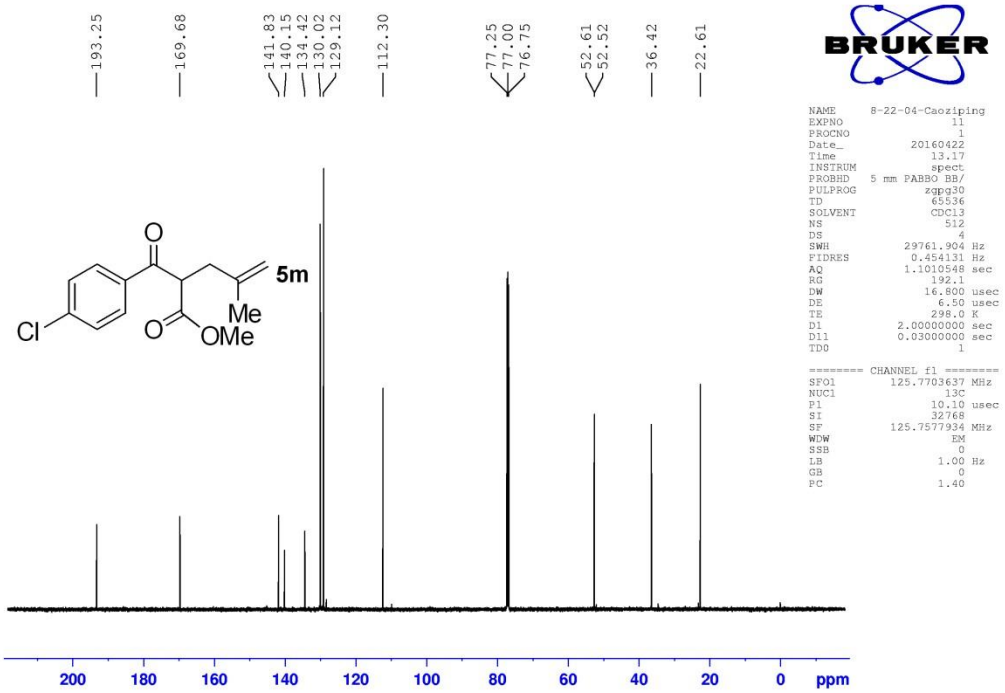


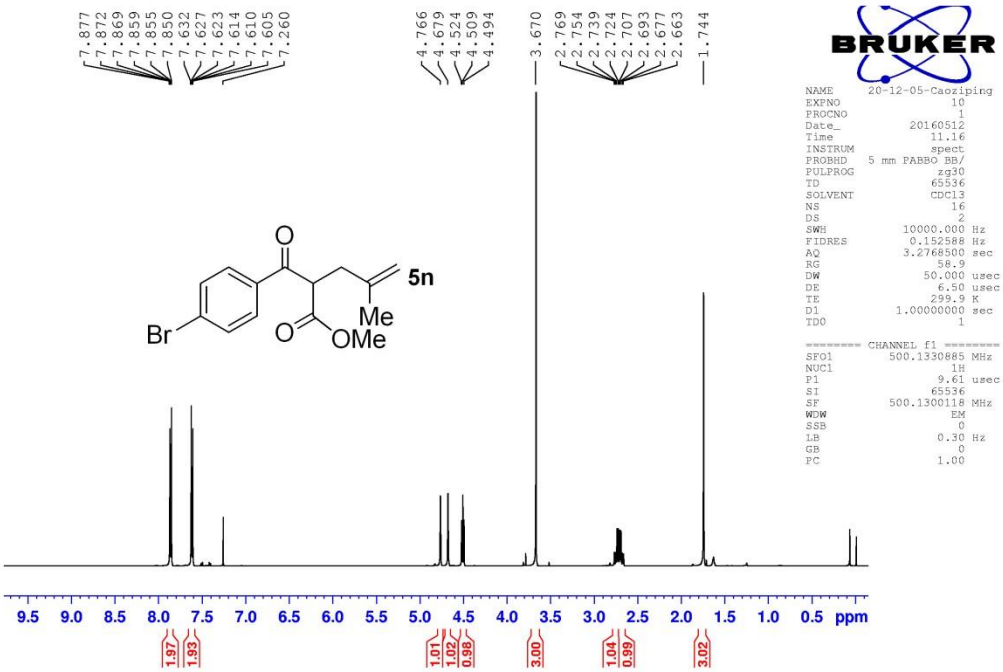

```
NAME        17-01-04-Caoziping
EXPNO                       10
PROCNO                       1
Date_                 20160401
Time                     10.48
INSTRUM                  spect
PROBHD   5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                 10000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2768500 sec
RG                        29.63
DW                     50.000 usec
DE                      6.50 usec
TE                     298.3 K
D1                    1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1                500.1330885 MHz
NUC1                        1H
P1                       9.61 usec
SI                      65536
SF                  500.1300119 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

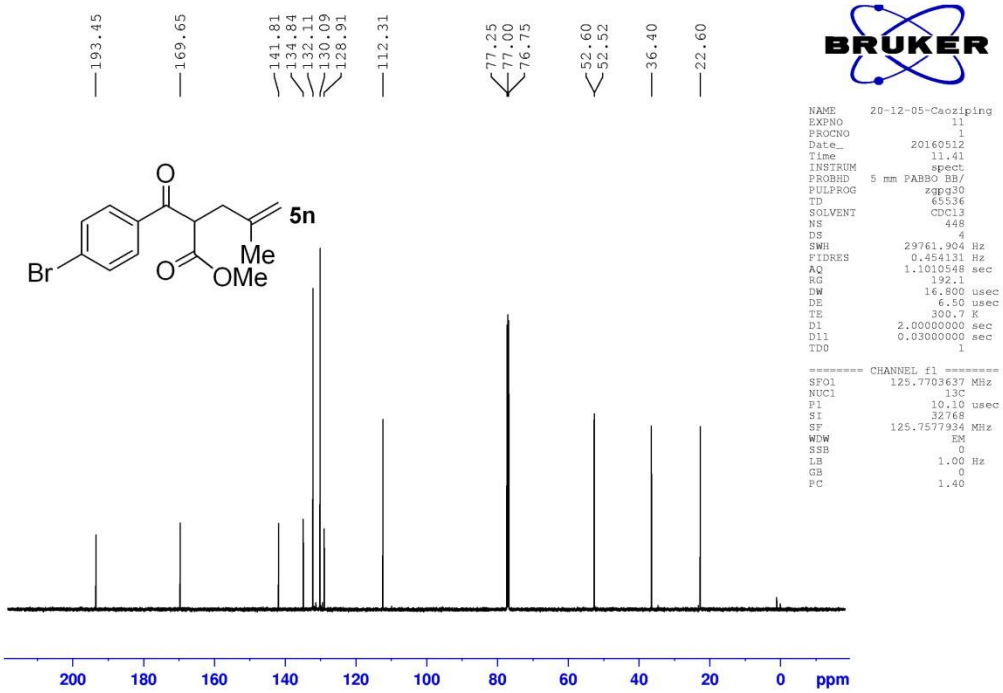


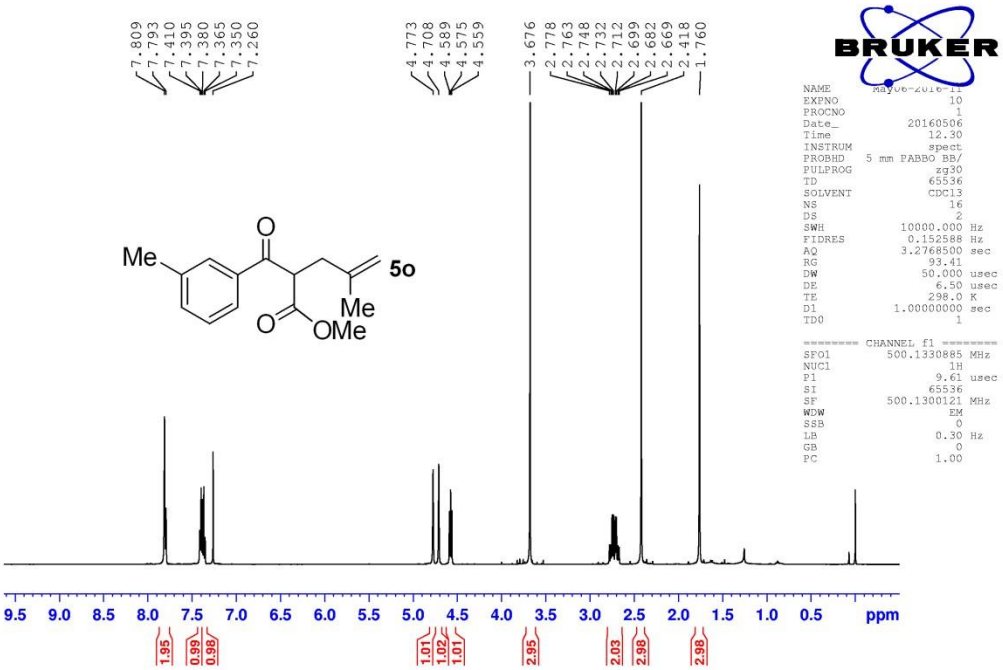

```
NAME        17-01-04-Caoziping
EXPNO                       11
PROCNO                       1
Date_                 20160401
Time                     11.10
INSTRUM                  spect
PROBHD   5 mm PABBO BB/
PULPROG                 zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        384
DS                          4
SWH                 29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010548 sec
RG                        192.1
DW                     16.800 usec
DE                      6.50 usec
TE                     299.3 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1                125.7703637 MHz
NUC1                        13C
P1                      10.10 usec
SI                      32768
SF                  125.7577953 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

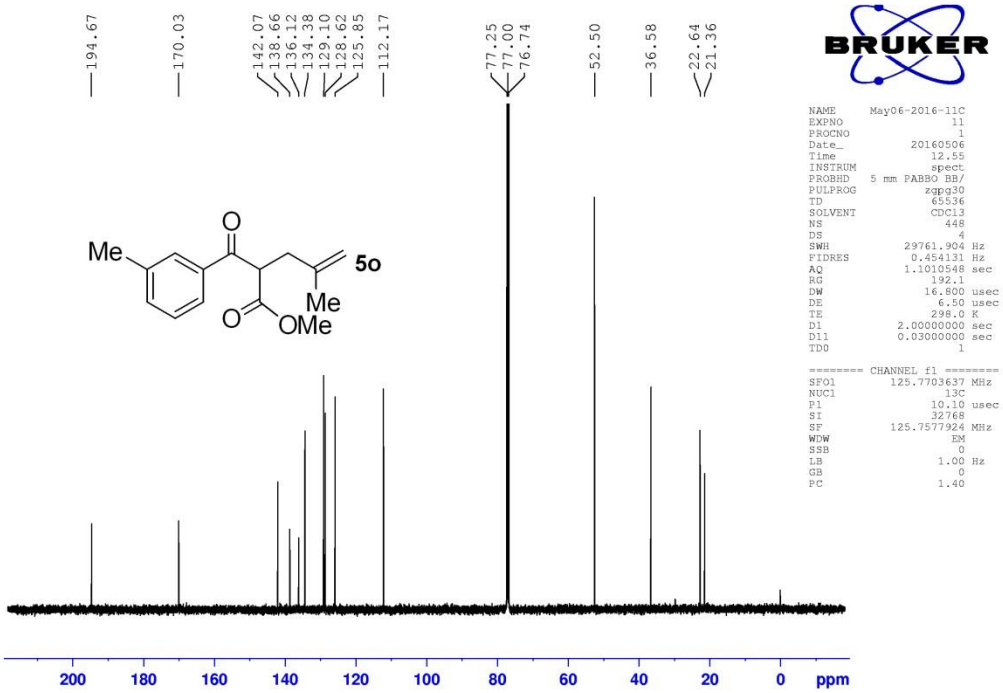

7.955
7.937
7.461
7.444
7.260
4.769
4.683
4.534
4.520
4.505
3.673
2.774
2.759
2.744
2.728
2.709
2.696
2.679
2.665
1.748
5m
Cl
O
Me
OMe
NAME 8-22-04-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160422
Time 12.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 66.25
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.97
1.96
1.00
1.03
1.00
3.00
2.06
3.00

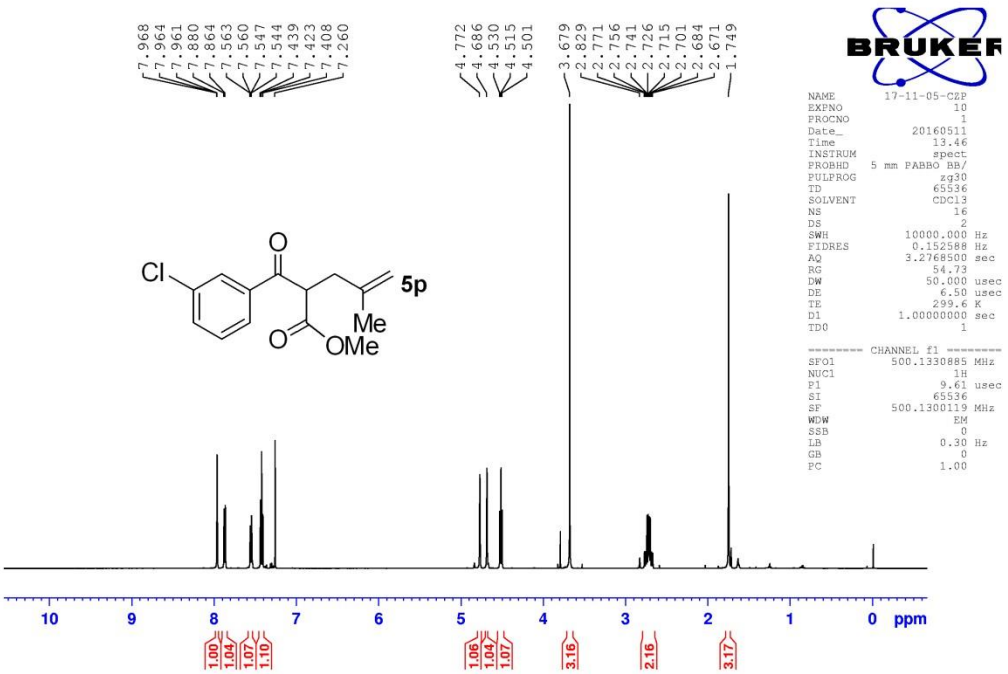

193.25
169.68
141.83
140.15
134.42
130.02
129.12
112.30
77.25
77.00
76.75
52.61
52.52
36.42
22.61
5m
Cl
O
Me
OMe
NAME 8-22-04-Caoziping
EXPNO 11
PROCNO 1
Date_ 20160422
Time 13.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577934 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

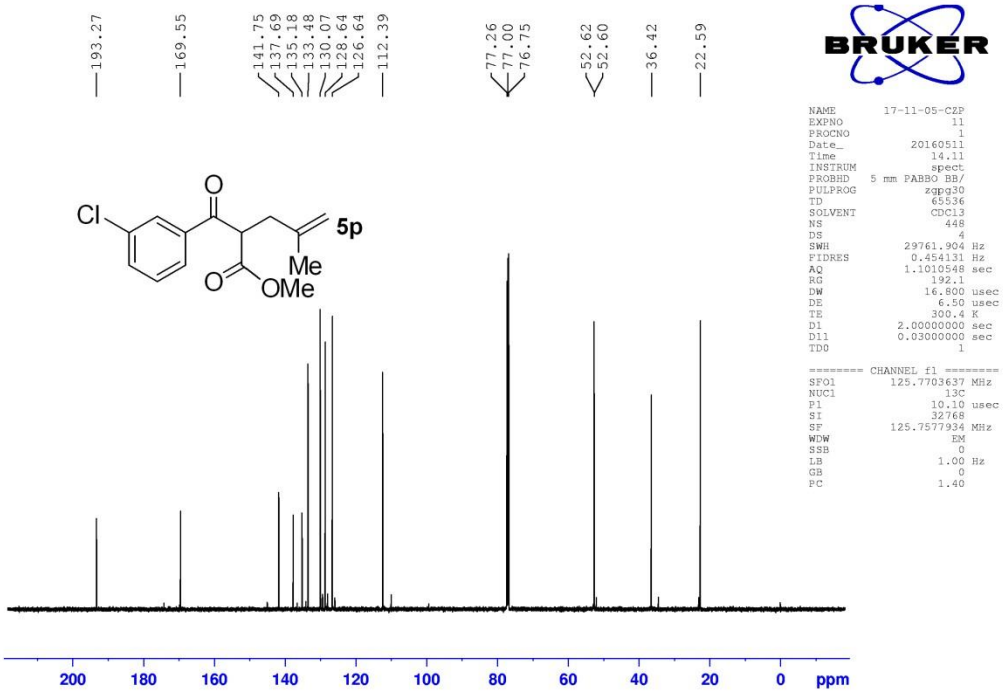

7.877
7.872
7.869
7.859
7.855
7.850
7.632
7.627
7.623
7.614
7.610
7.605
7.260
4.766
4.679
4.524
4.509
4.494
3.670
2.769
2.754
2.739
2.724
2.707
2.693
2.677
2.663
1.744
BRUKER
NAME 20-12-05-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160512
Time 11.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 58.9
DW 50.000 usec
DE 6.50 usec
TE 299.9 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
Br
O
Me
OMe
5n
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 ppm
1.97
1.93
1.01
1.02
0.98
3.00
1.04
0.99
3.02

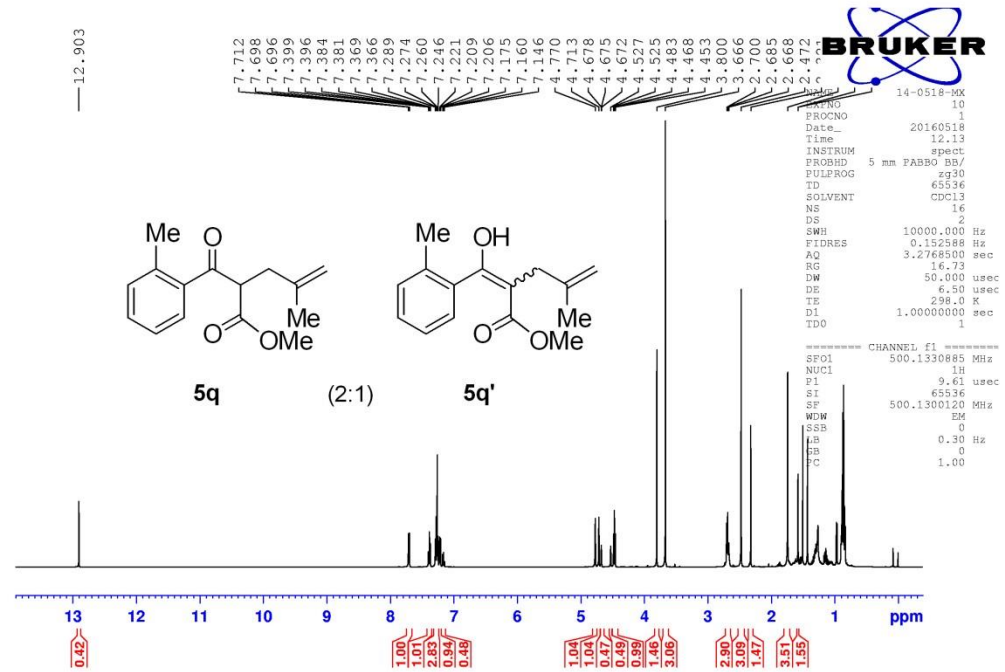

193.45
169.65
141.81
134.84
132.11
130.09
128.91
112.31
77.25
77.00
76.75
52.60
52.52
36.40
22.60
BRUKER
NAME 20-12-05-Caoziping
EXPNO 11
PROCNO 1
Date_ 20160512
Time 11.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 448
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 300.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577934 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
Br
O
Me
OMe
5n
200 180 160 140 120 100 80 60 40 20 0 ppm

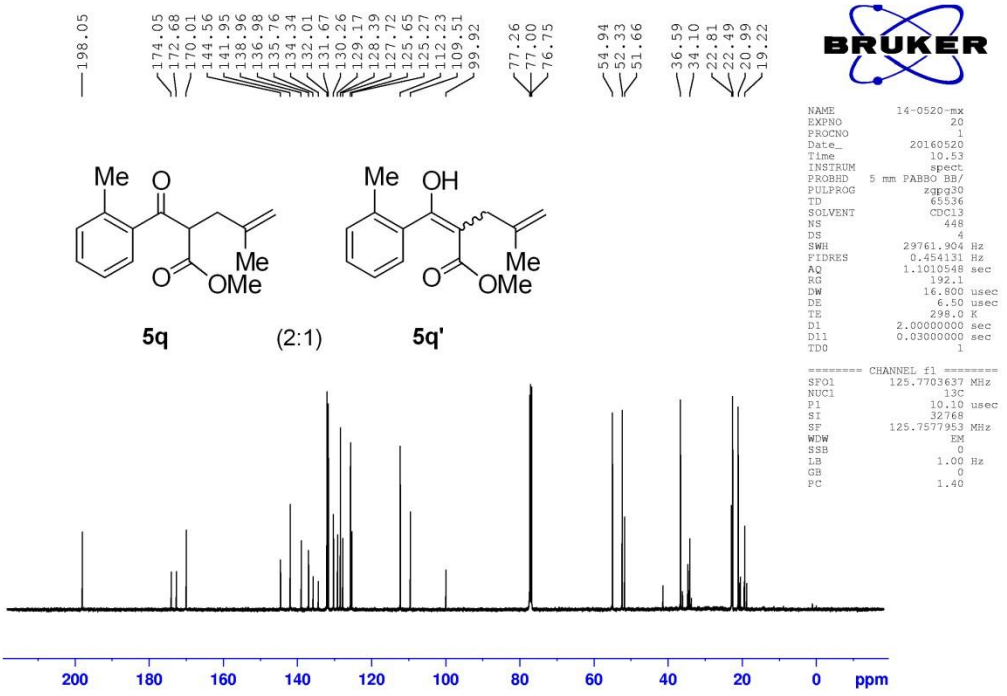

7.809
7.793
7.410
7.395
7.380
7.365
7.350
7.260
4.773
4.708
4.589
4.575
4.559
3.676
2.778
2.763
2.748
2.732
2.712
2.699
2.682
2.669
2.418
1.760
Me
O
5o
Me
O
OMe
BRUKER
NAME May06-2016-11
EXPNO 10
PROCNO 1
Date_ 20160506
Time 12.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 93.41
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
1.95
0.99
0.98
1.01
1.02
1.01
2.95
2.03
2.98
2.98

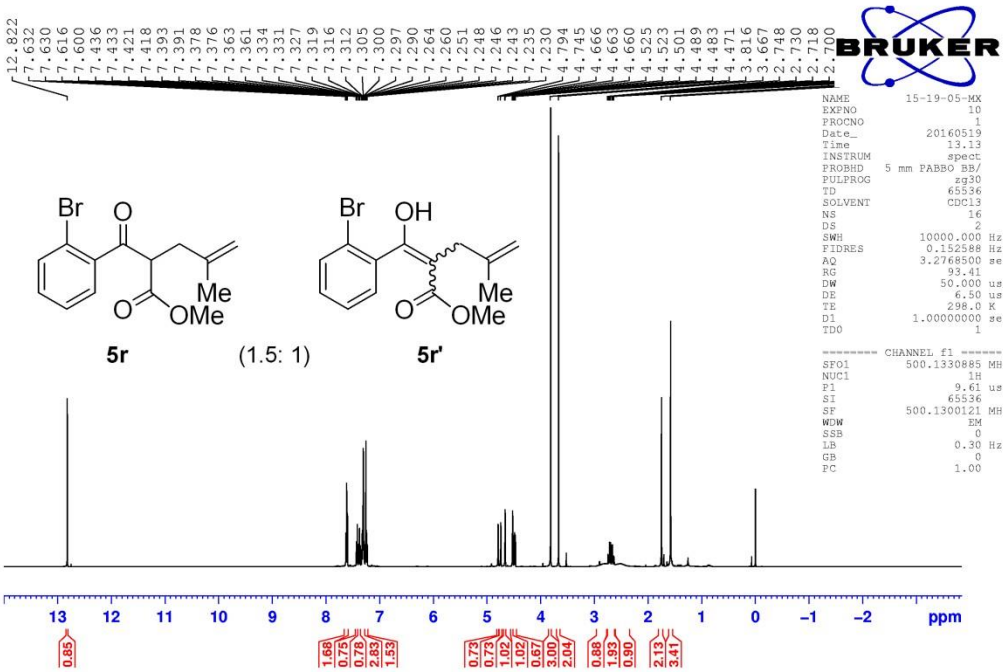

194.67
170.03
142.07
138.66
136.12
134.38
129.10
128.62
125.85
112.17
77.25
77.00
76.74
52.50
36.58
22.64
21.36
Me
O
5o
Me
O
OMe
BRUKER
NAME May06-2016-11C
EXPNO 11
PROCNO 1
Date_ 20160506
Time 12.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 448
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577924 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

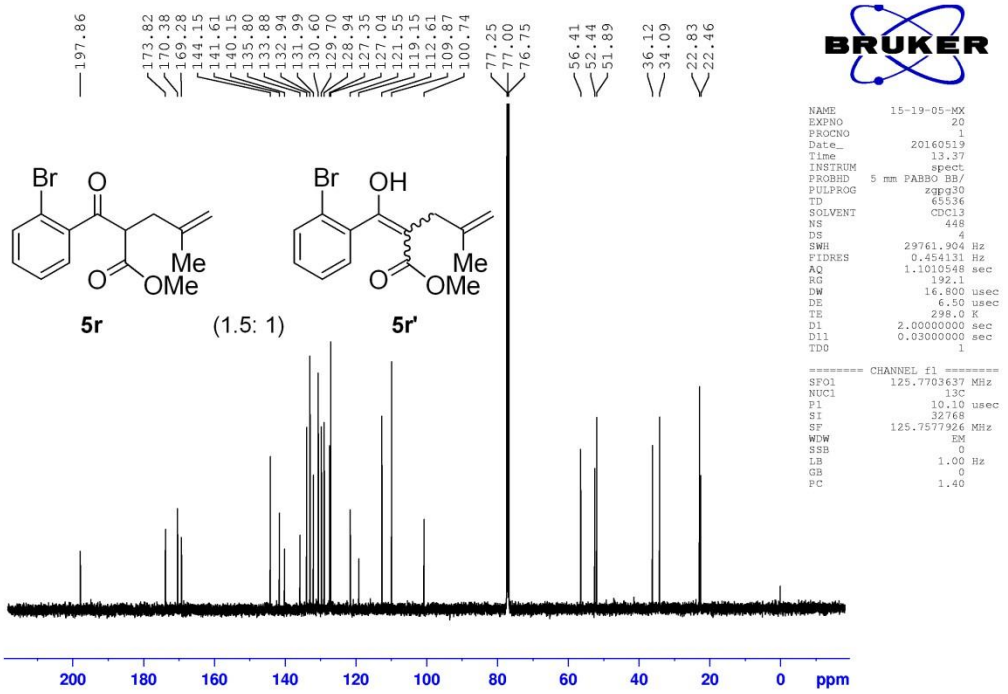

7.968
7.964
7.961
7.880
7.864
7.563
7.560
7.547
7.544
7.439
7.423
7.408
7.260
4.772
4.686
4.530
4.515
4.501
3.679
2.829
2.771
2.756
2.741
2.726
2.715
2.701
2.684
2.671
1.749
Cl
O
O
Me
OMe
5p
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.04
1.07
1.10
1.06
1.04
1.07
3.16
2.16
3.17
BRUKER
NAME 17-11-05-CZP
EXPNO 10
PROCNO 1
Date_ 20160511
Time 13.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 54.73
DW 50.000 usec
DE 6.50 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300119 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

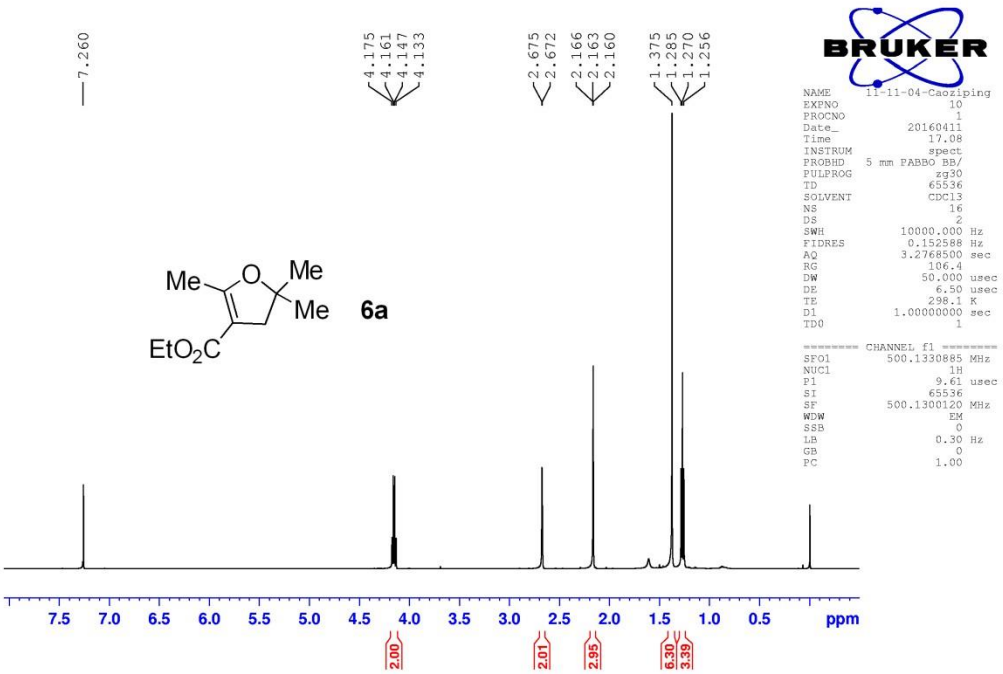

193.27
169.55
141.75
137.69
135.18
133.48
130.07
128.64
126.64
112.39
77.26
77.00
76.75
52.62
52.60
36.42
22.59
Cl
O
O
Me
OMe
5p
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME 17-11-05-CZP
EXPNO 11
PROCNO 1
Date_ 20160511
Time 14.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 448
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577934 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


12.903
7.712
7.698
7.696
7.399
7.396
7.384
7.381
7.369
7.366
7.289
7.274
7.260
7.246
7.221
7.209
7.206
7.175
7.160
7.146
4.770
4.713
4.678
4.675
4.672
4.527
4.525
4.483
4.468
4.453
3.800
3.666
3.700
2.685
2.668
2.472
BRUKER
NAME 14-0518-MX
EXPNO 10
PROCNO 1
Date_ 20160518
Time 12.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 16.73
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
O
Me
O
OMe
Me
OH
Me
O
OMe
5q
(2:1)
5q'
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.42
1.00
1.01
2.83
0.94
0.48
1.04
1.04
0.47
0.49
0.99
1.46
3.06
2.90
3.09
1.47
3.51
1.55

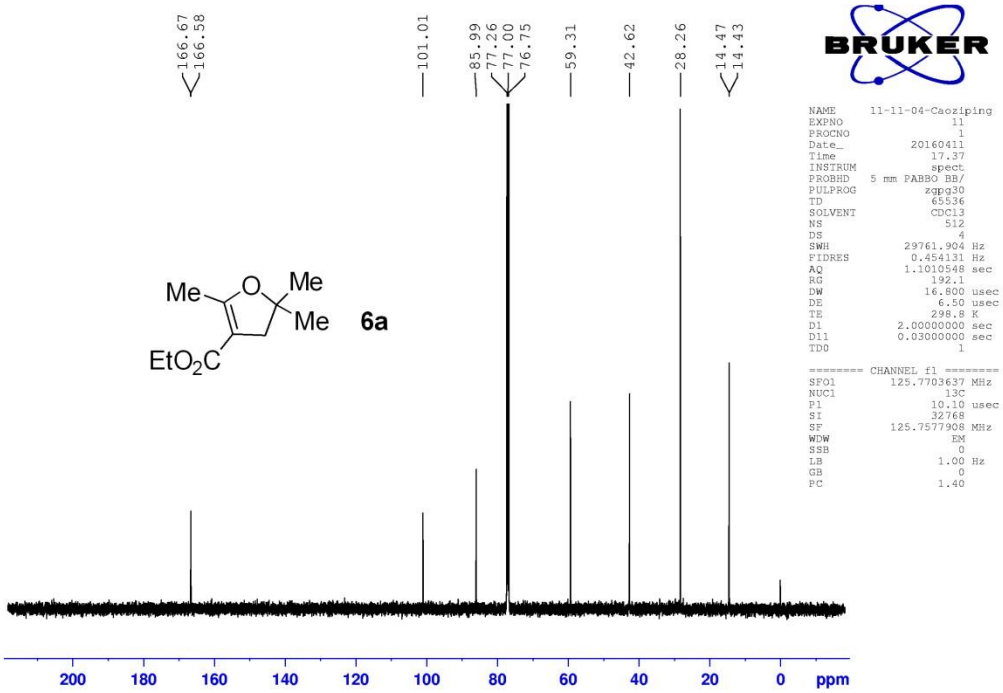

198.05
174.05
172.68
170.01
144.56
141.95
138.96
136.98
135.76
134.34
132.01
131.67
130.26
129.17
128.39
127.72
125.65
125.27
112.23
109.51
99.92
77.26
77.00
76.75
54.94
52.33
51.66
36.59
34.10
22.81
22.49
20.99
19.22
BRUKER
NAME 14-0520-mx
EXPNO 20
PROCNO 1
Date_ 20160520
Time 10.53
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 448
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577953 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
O
Me
O
OMe
Me
OH
Me
O
OMe
5q
(2:1)
5q'
200
180
160
140
120
100
80
60
40
20
0
ppm


12.822
7.632
7.630
7.616
7.600
7.436
7.433
7.421
7.418
7.393
7.391
7.378
7.376
7.363
7.361
7.334
7.331
7.327
7.319
7.316
7.312
7.305
7.300
7.297
7.290
7.264
7.260
7.251
7.248
7.246
7.243
7.235
7.230
4.794
4.745
4.666
4.663
4.660
4.525
4.523
4.501
4.489
4.483
4.471
3.816
3.667
2.748
2.730
2.718
2.700
BRUKER
NAME 15-19-05-MX
EXPNO 10
PROCNO 1
Date_ 20160519
Time 13.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 se
RG 93.41
DW 50.000 us
DE 6.50 us
TE 298.0 K
D1 1.00000000 se
TD0 1
CHANNEL f1
SFO1 500.1330885 MH
NUC1 1H
P1 9.61 us
SI 65536
SF 500.1300121 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Br
O
Me
O
OMe
5r
(1.5: 1)
Br
OH
Me
O
OMe
5r'
13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 ppm
0.85
1.68 0.75 0.78 2.83 1.53
0.73 0.73 1.02 1.02 0.67 3.00 2.04
0.88 1.93 0.90
2.13 3.41

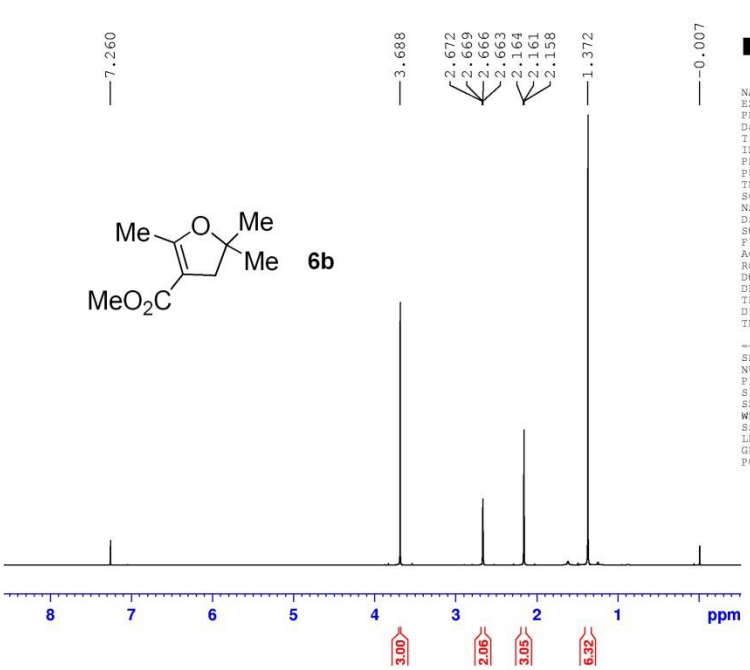

197.86
173.82
170.38
169.28
144.15
141.61
140.15
135.80
133.88
132.94
131.99
130.60
129.70
128.94
127.35
127.04
121.55
119.15
112.61
109.87
100.74
77.25
77.00
76.75
56.41
52.44
51.89
36.12
34.09
22.83
22.46
BRUKER
NAME 15-19-05-MX
EXPNO 20
PROCNO 1
Date_ 20160519
Time 13.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 448
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577926 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Br
O
Me
O
OMe
5r
(1.5: 1)
Br
OH
Me
O
OMe
5r'
200 180 160 140 120 100 80 60 40 20 0 ppm



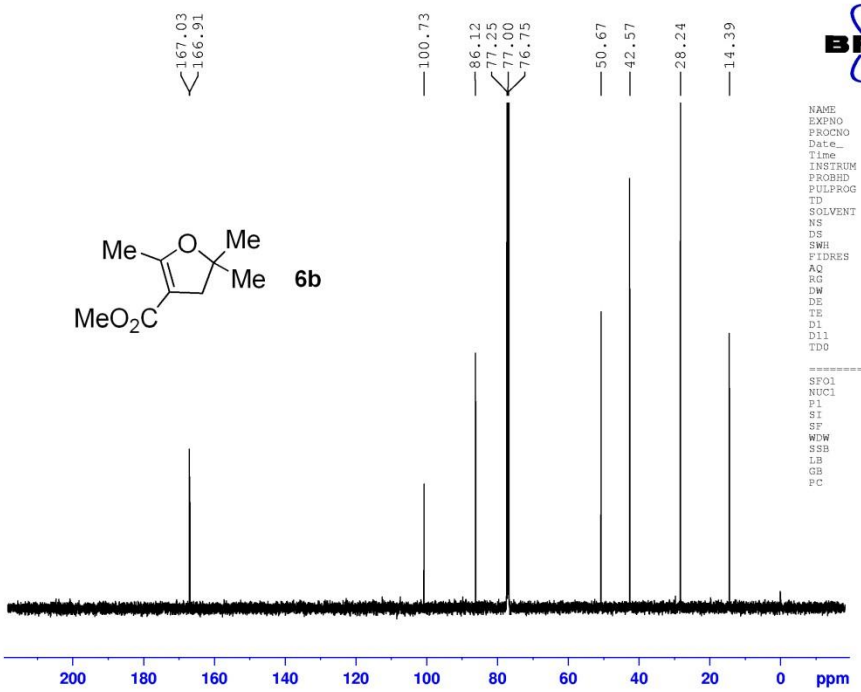

```
NAME          11-11-04-Caoziping
EXPNO                         10
PROCNO                         1
Date_                   20160411
Time                       17.08
INSTRUM                    spect
PROBHD   5 mm PABBO BB/
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                 10000.000 Hz
FIDRES                  0.152588 Hz
AQ                    3.2768500 sec
RG                        106.4
DW                       50.000 usec
DE                        6.50 usec
TE                       298.1 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1               500.1330885 MHz
NUC1                         1H
P1                         9.61 usec
SI                        65536
SF                 500.1300120 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```



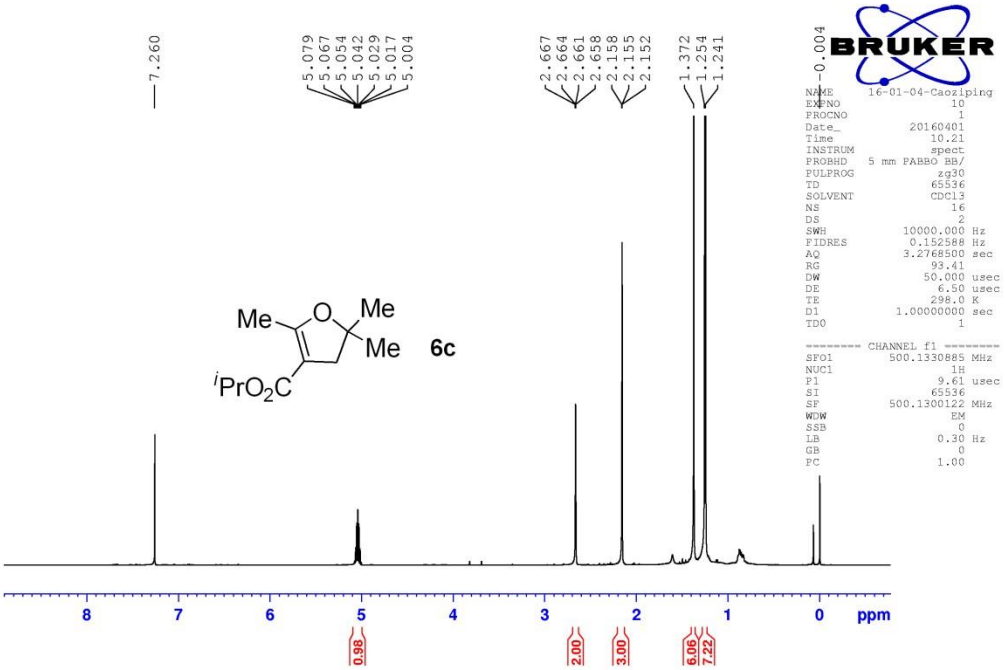

```
NAME          11-11-04-Caoziping
EXPNO                         11
PROCNO                         1
Date_                   20160411
Time                       17.37
INSTRUM                    spect
PROBHD   5 mm PABBO BB/
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                           512
DS                             4
SWH                 29761.904 Hz
FIDRES                  0.454131 Hz
AQ                    1.1010548 sec
RG                        192.1
DW                       16.800 usec
DE                        6.50 usec
TE                       298.8 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1               125.7703637 MHz
NUC1                         13C
P1                        10.10 usec
SI                        32768
SF                 125.7577908 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```

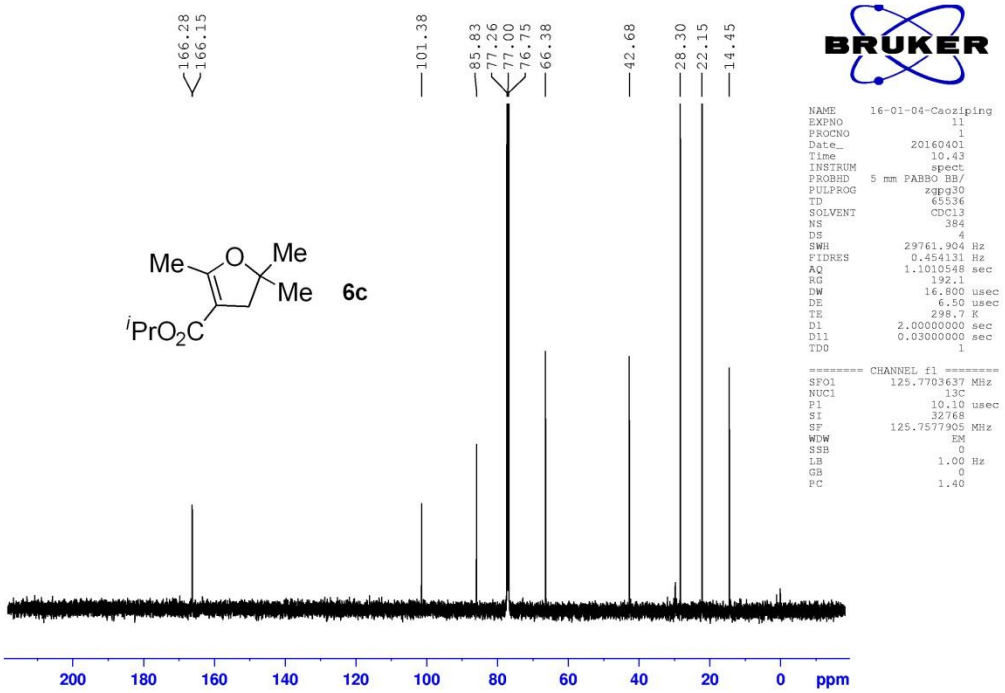


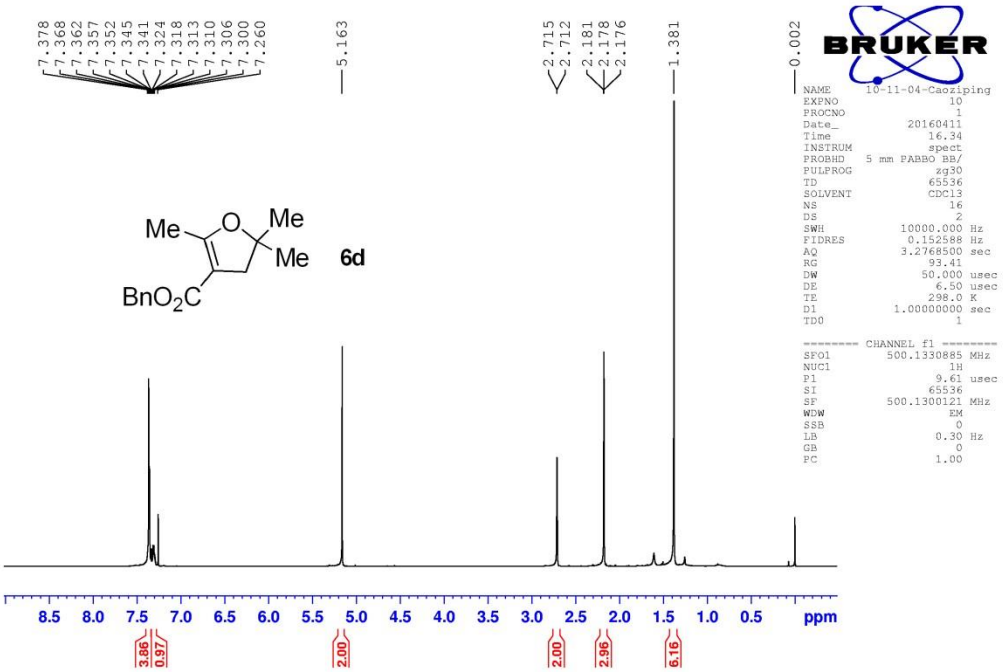

```
NAME          16-11-05-CZP
EXPNO                   10
PROCNO                   1
Date_             20160511
Time                 13.15
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10000.000 Hz
FIDRES            0.152588 Hz
AQ             3.2768500 sec
RG                  93.41
DW                50.000 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                   9.61 usec
SI                  65536
SF           500.1300119 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```




```
NAME          16-11-05-CZP
EXPNO                   11
PROCNO                   1
Date_             20160511
Time                 13.40
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    448
DS                      4
SWH           29761.904 Hz
FIDRES            0.454131 Hz
AQ             1.1010548 sec
RG                 192.1
DW                16.800 usec
DE                 6.50 usec
TE                300.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                   13C
P1                  10.10 usec
SI                  32768
SF           125.7577904 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

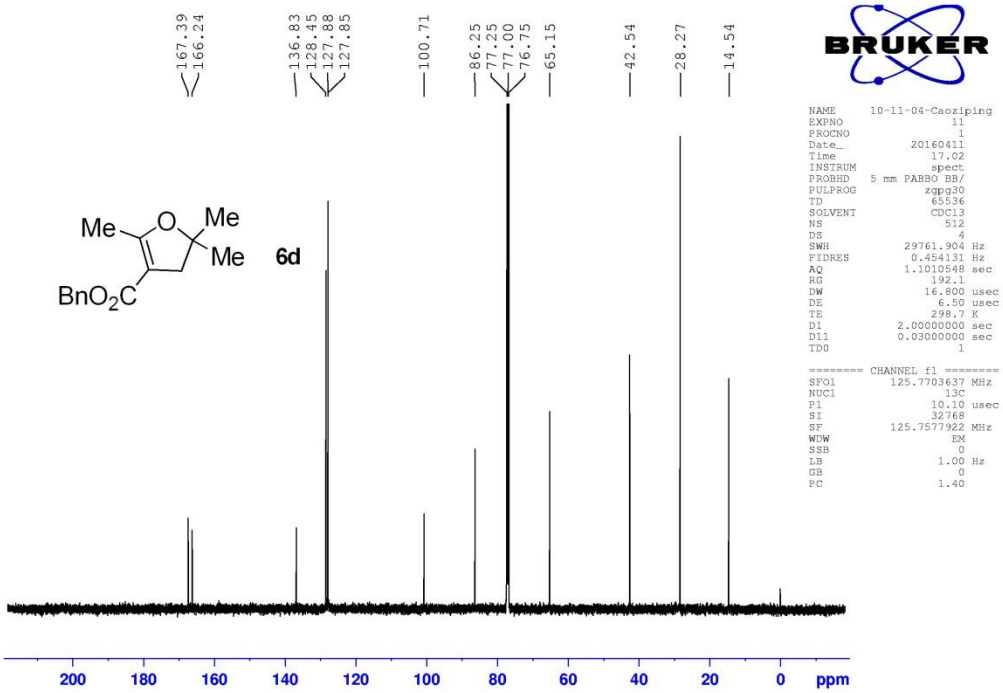

7.260
5.079
5.067
5.054
5.042
5.029
5.017
5.004
2.667
2.664
2.661
2.658
2.158
2.155
2.152
1.372
1.254
1.241
-0.004
Me
O
Me
Me
6c
iPrO2C
8
7
6
5
4
3
2
1
0
ppm
0.98
2.00
3.00
6.06
7.22
BRUKER
NAME 16-01-04-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160401
Time 10.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 93.41
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


166.28
166.15
101.38
85.83
77.26
77.00
76.75
66.38
42.68
28.30
22.15
14.45
Me
O
Me
Me
6c
iPrO2C
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME 16-01-04-Caoziping
EXPNO 11
PROCNO 1
Date_ 20160401
Time 10.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 384
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577905 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



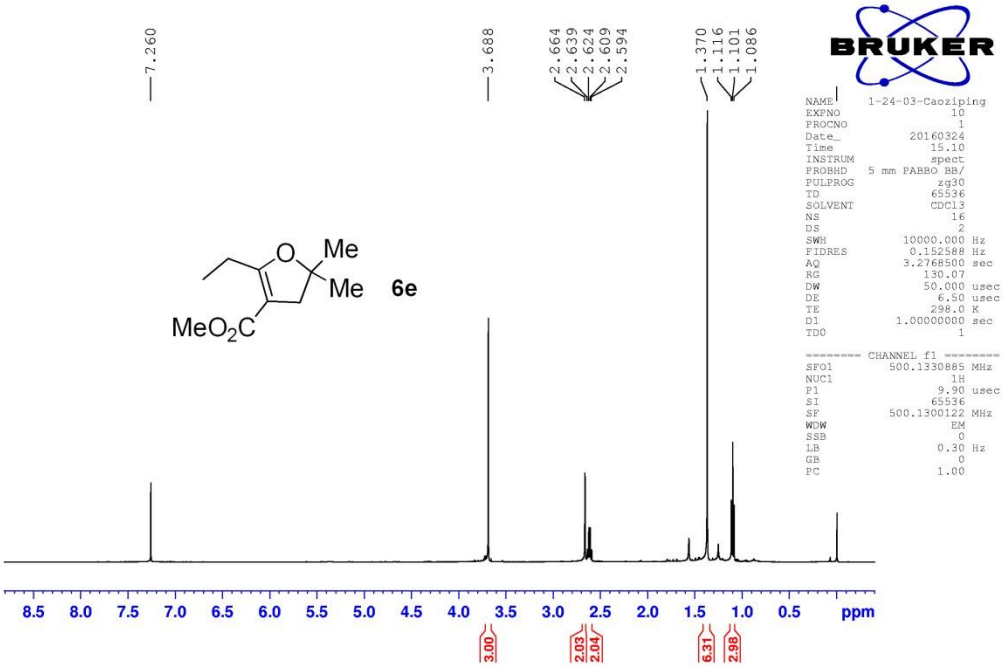
BRUKER

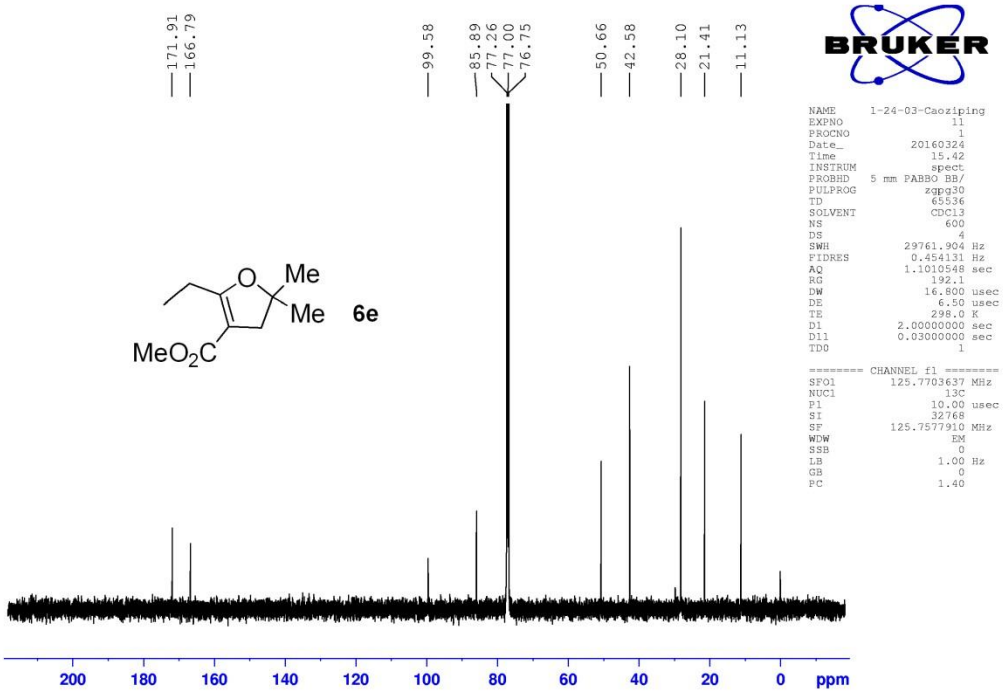
```
NAME          10-11-04-Caoziping
EXPNO                         10
PROCNO                         1
Date_                   20160411
Time                       16.34
INSTRUM                    spect
PROBHD   5 mm PABBO BB/
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                   10000.000 Hz
FIDRES                    0.152588 Hz
AQ                    3.2768500 sec
RG                         93.41
DW                        50.000 usec
DE                         6.50 usec
TE                        298.0 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                500.1330885 MHz
NUC1                        1H
P1                         9.61 usec
SI                        65536
SF                  500.1300121 MHz
WDW                        EM
SSB                         0
LB                         0.30 Hz
GB                          0
PC                         1.00
```

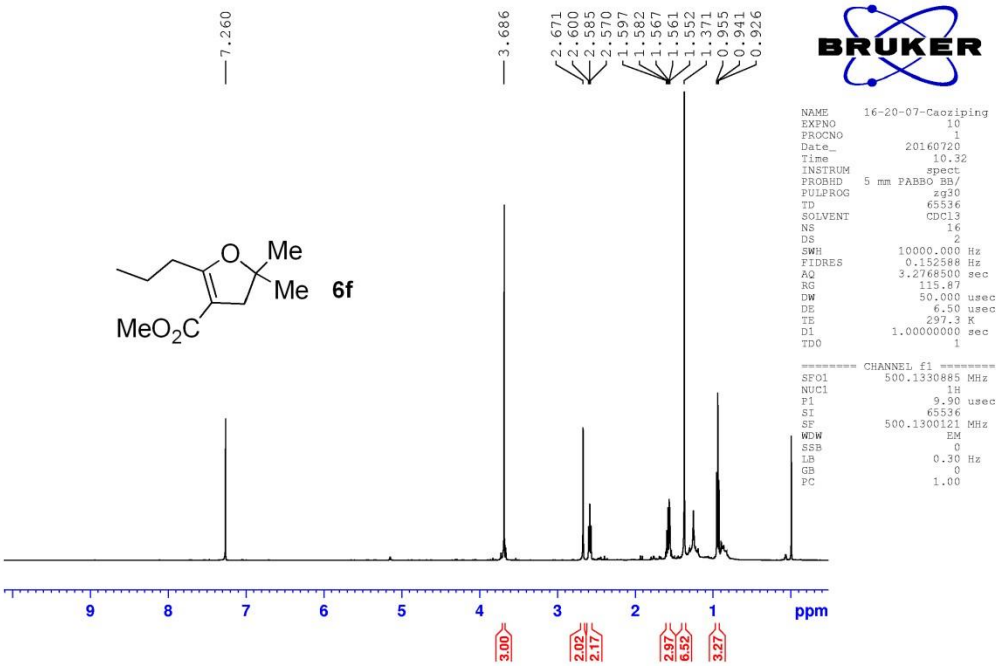


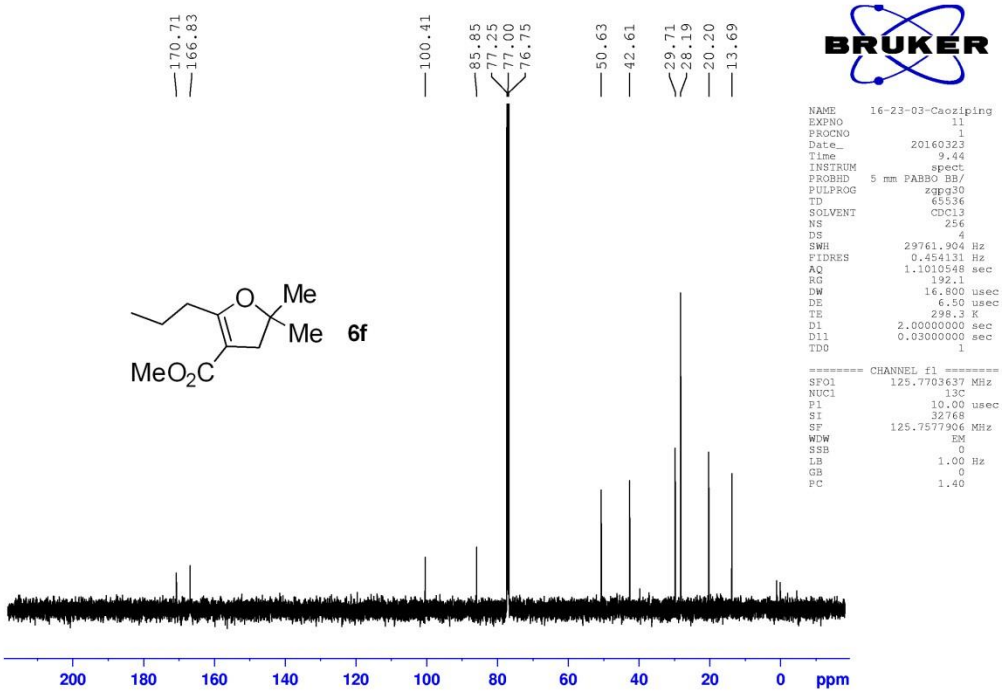
BRUKER

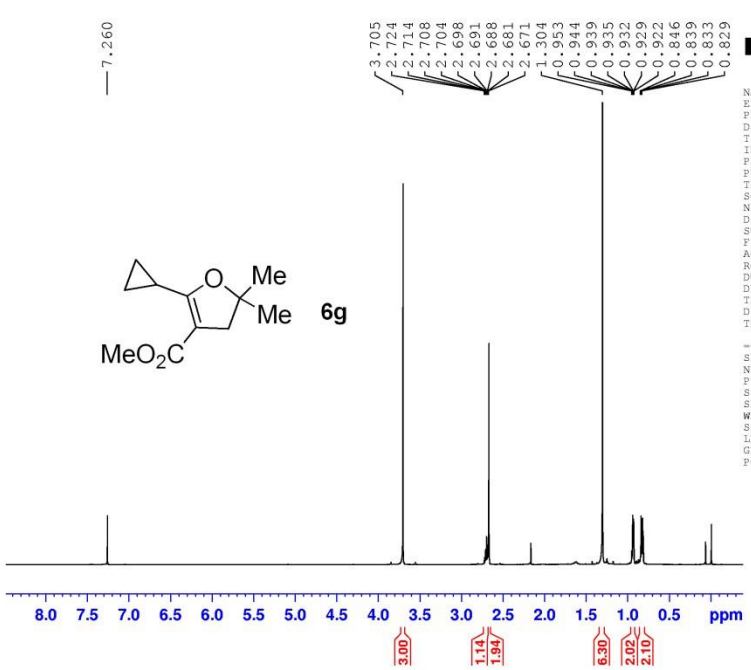
```
NAME          10-11-04-Caoziping
EXPNO                         11
PROCNO                         1
Date_                   20160411
Time                       17.02
INSTRUM                    spect
PROBHD   5 mm PABBO BB/
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                           512
DS                             4
SWH                   29761.904 Hz
FIDRES                    0.454131 Hz
AQ                    1.1010548 sec
RG                         192.1
DW                        16.800 usec
DE                         6.50 usec
TE                        298.7 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                125.7703637 MHz
NUC1                        13C
P1                        10.10 usec
SI                        32768
SF                  125.7577922 MHz
WDW                        EM
SSB                         0
LB                         1.00 Hz
GB                          0
PC                         1.40
```


7.260
3.688
2.664
2.639
2.624
2.609
2.594
1.370
1.116
1.101
1.086
Me
Me
O
MeO2C
6e
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 ppm
3.00
2.03
2.04
6.31
2.98
BRUKER
NAME 1-24-03-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160324
Time 15.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 130.07
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.90 usec
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

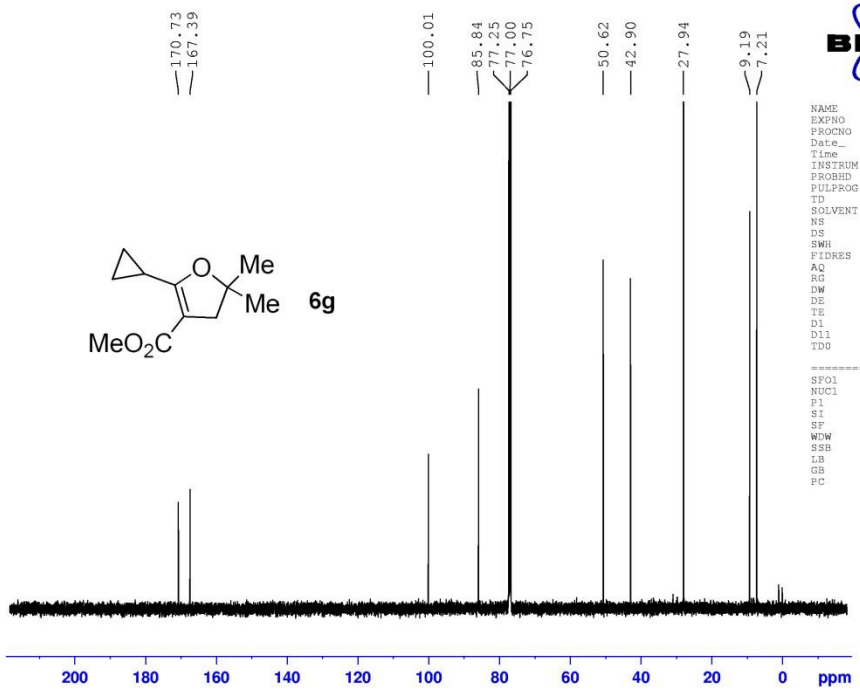

171.91
166.79
99.58
85.89
77.26
77.00
76.75
50.66
42.58
28.10
21.41
11.13
Me
Me
O
MeO2C
6e
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
NAME 1-24-03-Caoziping
EXPNO 11
PROCNO 1
Date_ 20160324
Time 15.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 125.7577910 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.260
3.686
2.671
2.600
2.585
2.570
1.597
1.582
1.567
1.561
1.552
1.371
0.955
0.941
0.926
Me
Me
6f
MeO2C
9
8
7
6
5
4
3
2
1
ppm
3.00
2.02
2.17
2.97
6.52
3.27
BRUKER
NAME 16-20-07-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160720
Time 10.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 115.87
DW 50.000 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.90 usec
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

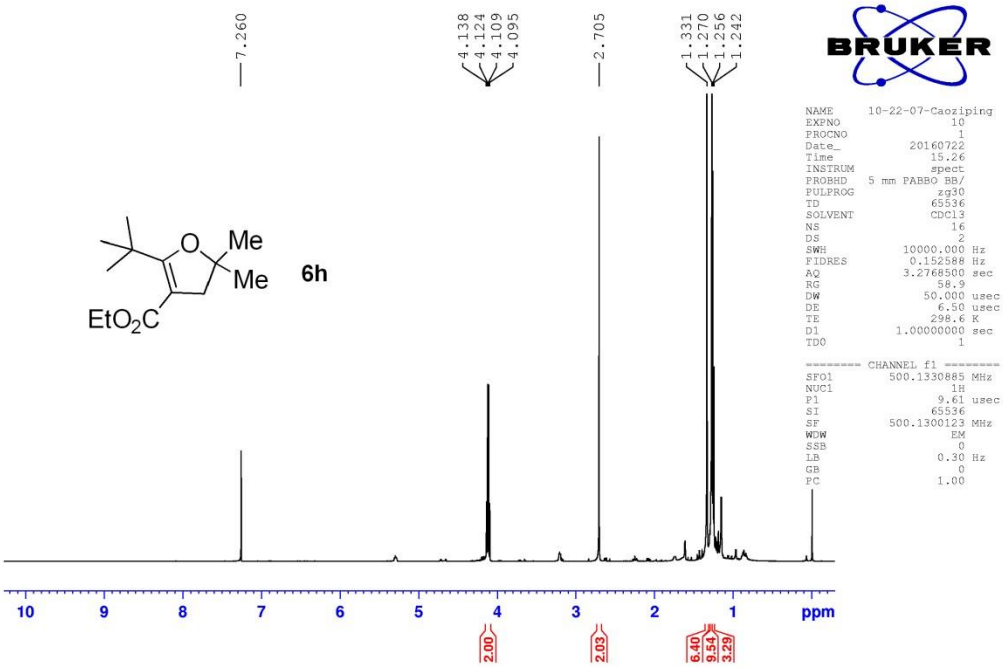

170.71
166.83
100.41
85.85
77.25
77.00
76.75
50.63
42.61
29.71
28.19
20.20
13.69
Me
Me
6f
MeO2C
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME 16-23-03-Caoziping
EXPNO 11
PROCNO 1
Date_ 20160323
Time 9.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 125.7577906 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

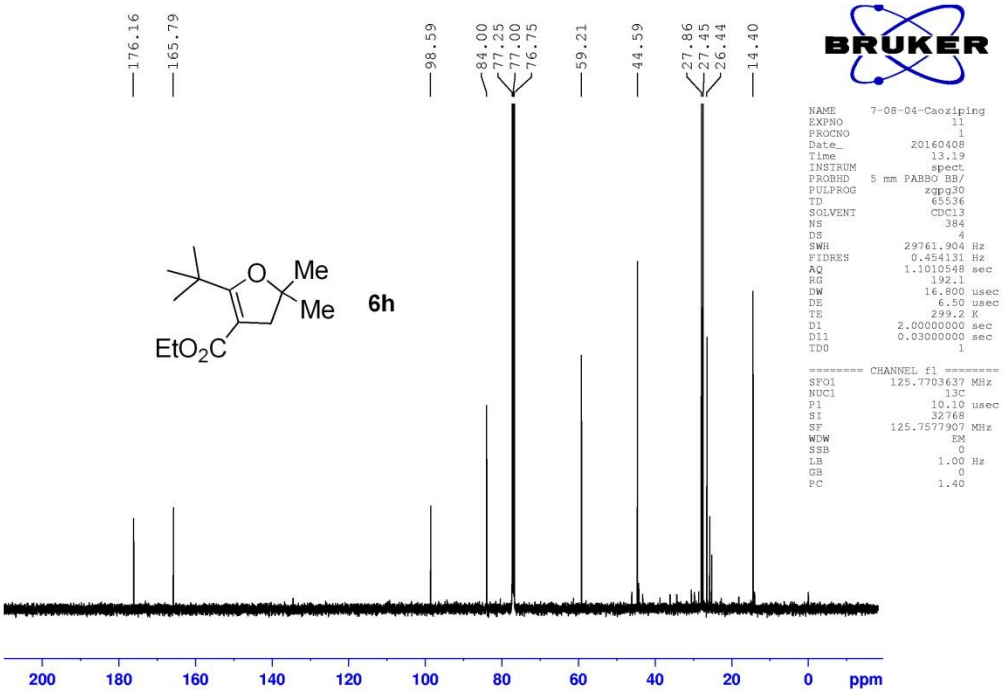


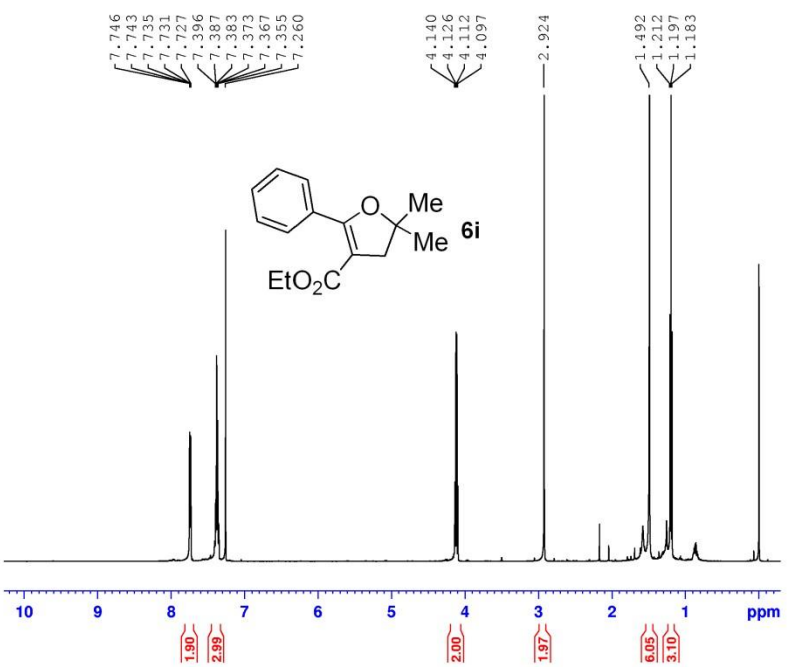

```
NAME          17-15-04-Caoziping
EXPNO                         10
PROCNO                         1
Date_                   20160415
Time                       12.57
INSTRUM                    spect
PROBHD     5 mm PABBO BB/
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                   10000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2768500 sec
RG                        93.41
DW                       50.000 usec
DE                         6.50 usec
TE                       298.0 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               500.1330885 MHz
NUC1                         1H
P1                         9.61 usec
SI                        65536
SF                 500.1300123 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```




```
NAME          17-15-04-Caoziping
EXPNO                         11
PROCNO                         1
Date_                   20160415
Time                       13.26
INSTRUM                    spect
PROBHD     5 mm PABBO BB/
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          512
DS                            4
SWH                   29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010548 sec
RG                        192.1
DW                       16.800 usec
DE                         6.50 usec
TE                       298.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               125.7703637 MHz
NUC1                         13C
P1                        10.10 usec
SI                        32768
SF                 125.7577917 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```

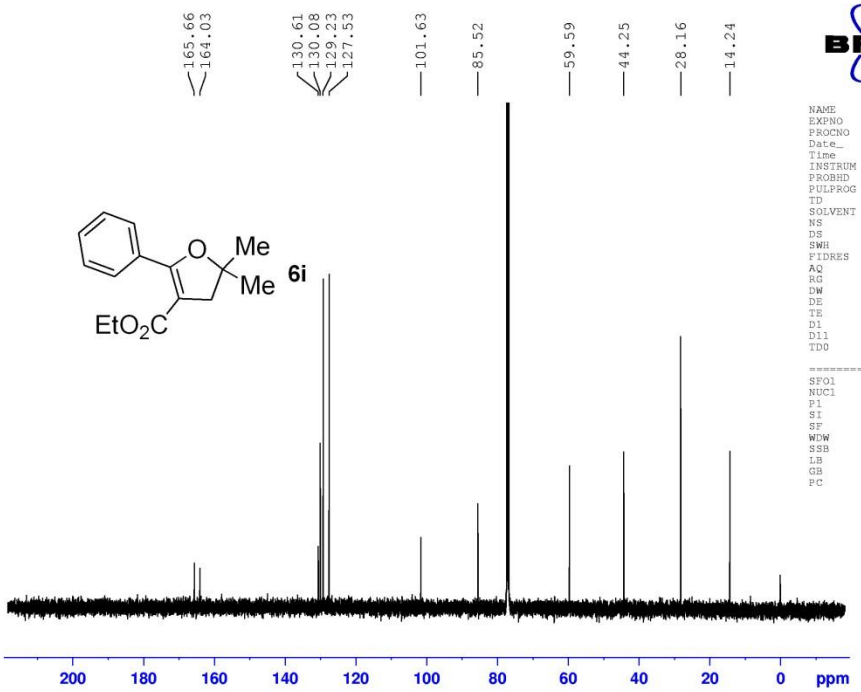

7.260
4.138
4.124
4.109
4.095
2.705
1.331
1.270
1.256
1.242
Me
Me
O
EtO2C
6h
NAME 10-22-07-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160722
Time 15.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 58.9
DW 50.000 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 ppm
2.00
2.03
6.40
9.54
3.29


176.16
165.79
98.59
84.00
77.25
77.00
76.75
59.21
44.59
27.86
27.45
26.44
14.40
Me
Me
O
EtO2C
6h
NAME 7-08-04-Caoziping
EXPNO 11
PROCNO 1
Date_ 20160408
Time 13.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 384
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 299.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577907 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



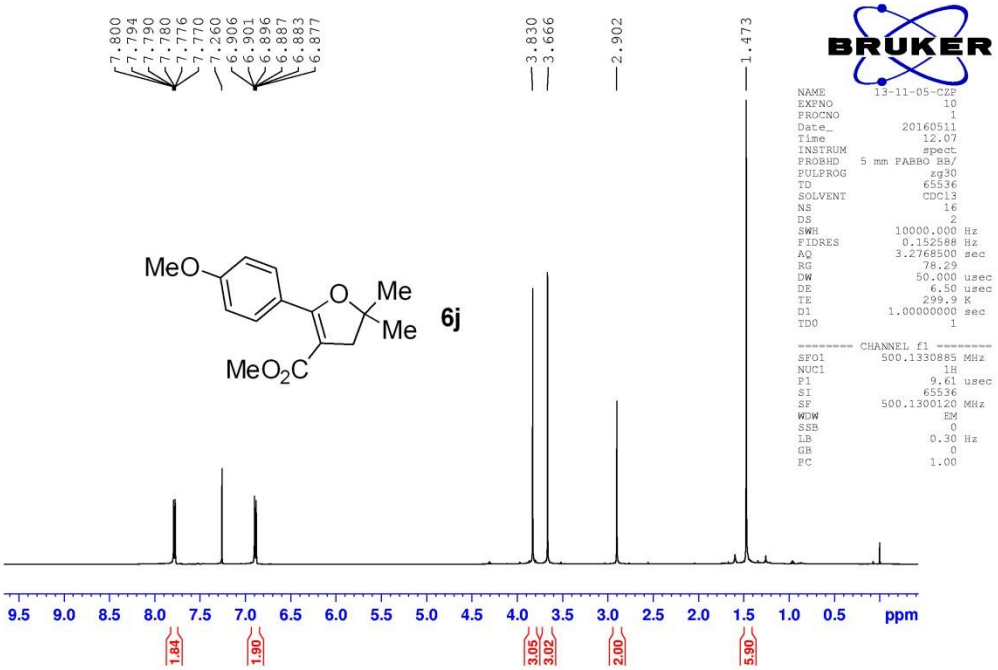
BRUKER

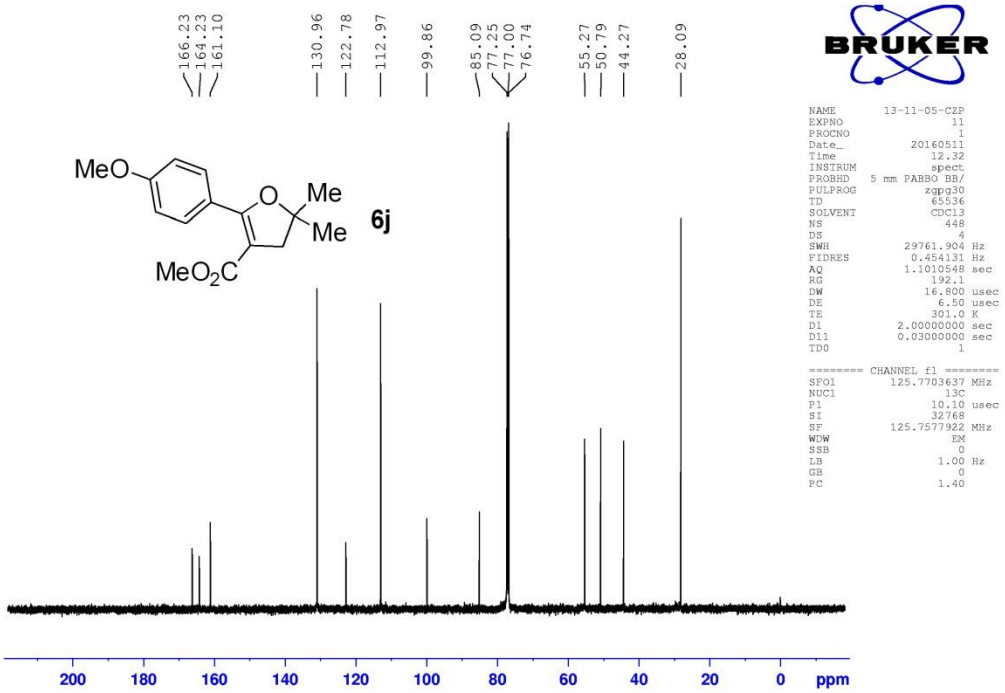
NAME	liangg
EXPNO	10
PROCNO	1
Date_	20160331
Time	16.18
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	130.07
DW	50.000 use
DE	6.50 use
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	9.61 use
SI	65536
SF	500.1300123 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

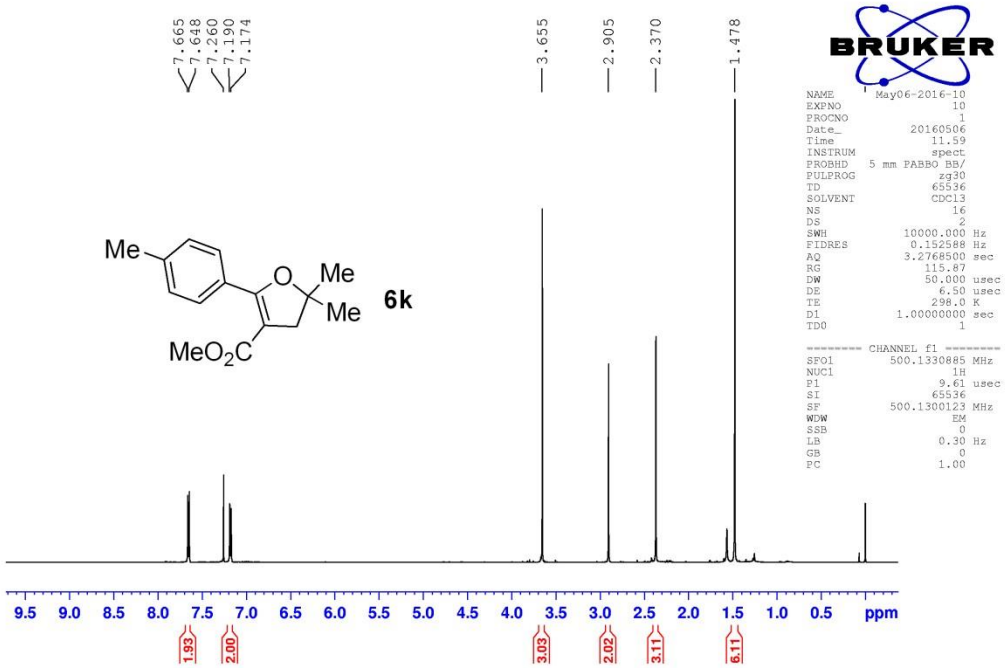


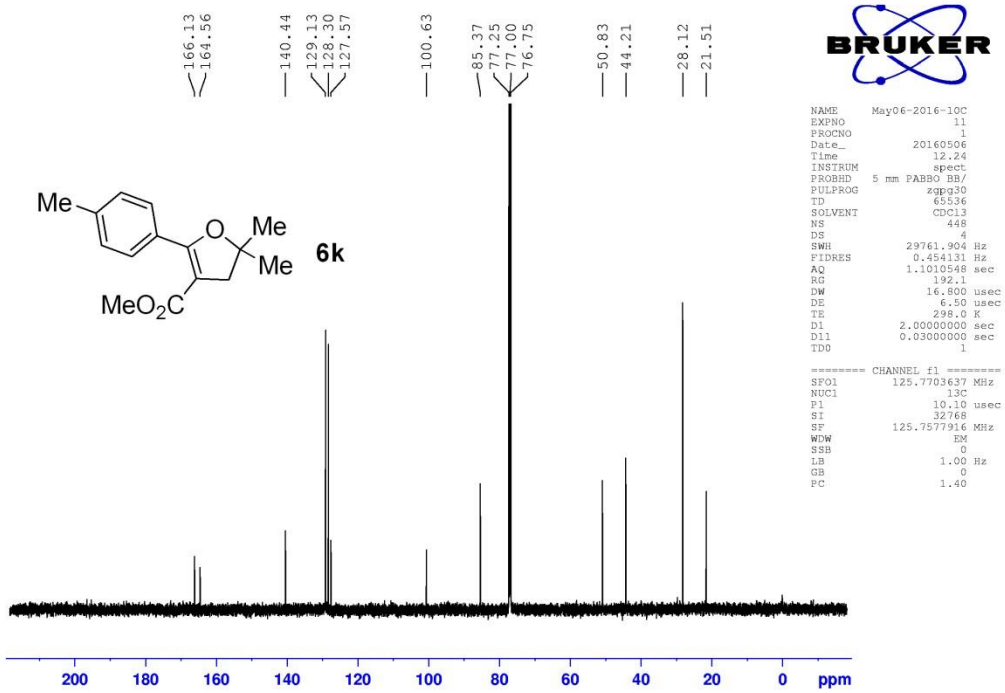
BRUKER

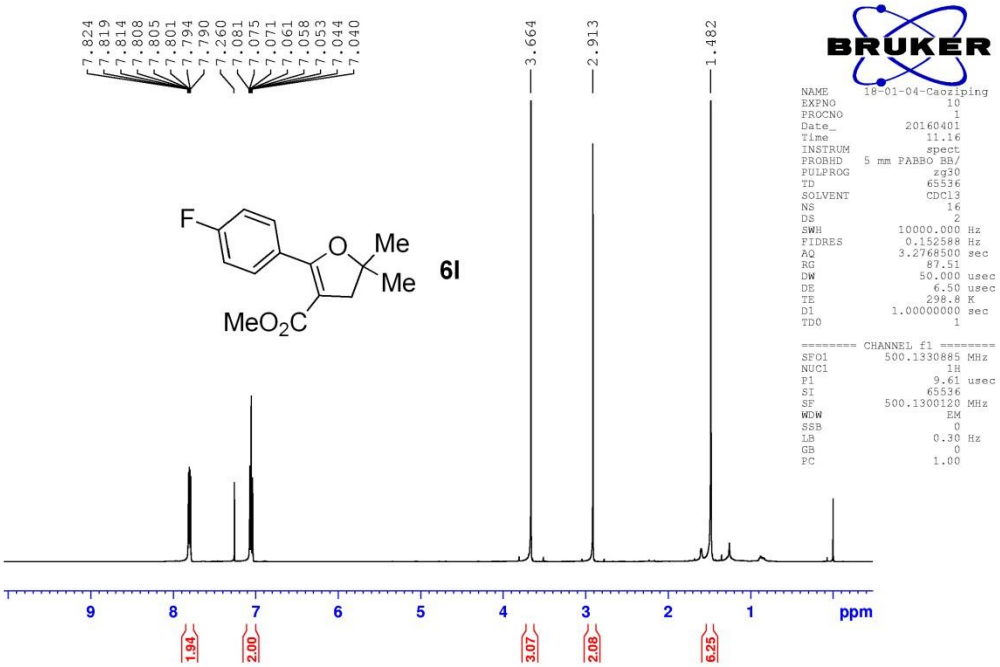
NAME	liangg
EXPNO	20
PROCNO	1
Date_	20160331
Time	16.46
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	192.1
DW	16.800 usec
DE	6.50 usec
TE	298.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	125.7703637 MHz
NUC1	13C
P1	10.10 usec
SI	32768
SF	125.7577908 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

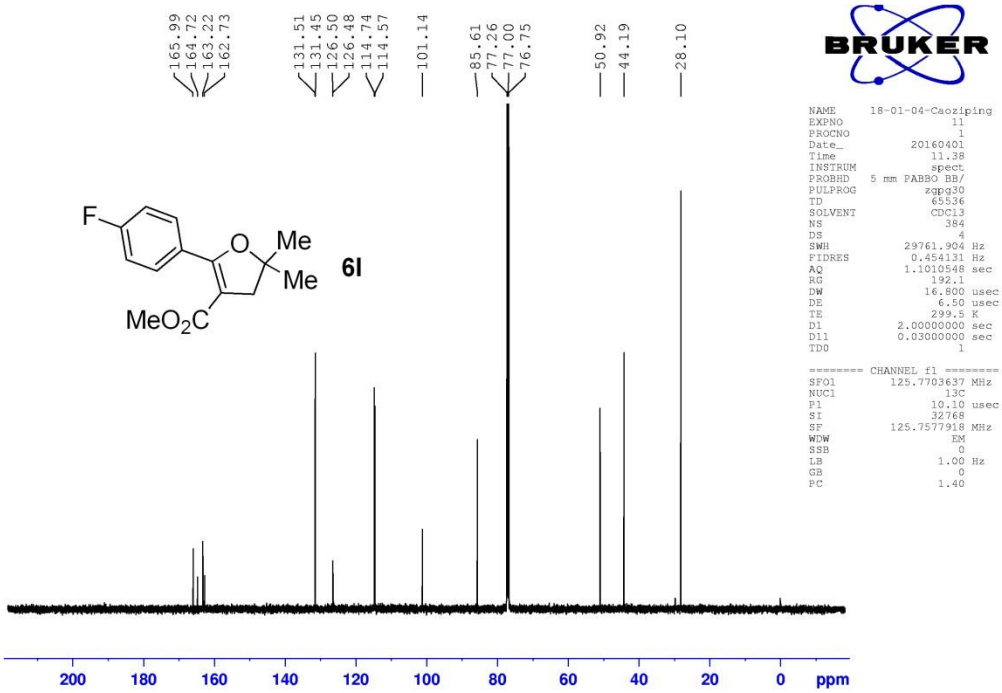

7.800
7.794
7.790
7.780
7.776
7.770
7.260
6.906
6.901
6.896
6.887
6.883
6.877
3.830
3.666
2.902
1.473
MeO
Me
Me
MeO2C
6j
BRUKER
NAME 13-11-05-CZP
EXPNO 10
PROCNO 1
Date_ 20160511
Time 12.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 78.29
DW 50.000 usec
DE 6.50 usec
TE 299.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
1.84
1.90
3.05
3.02
2.00
5.90

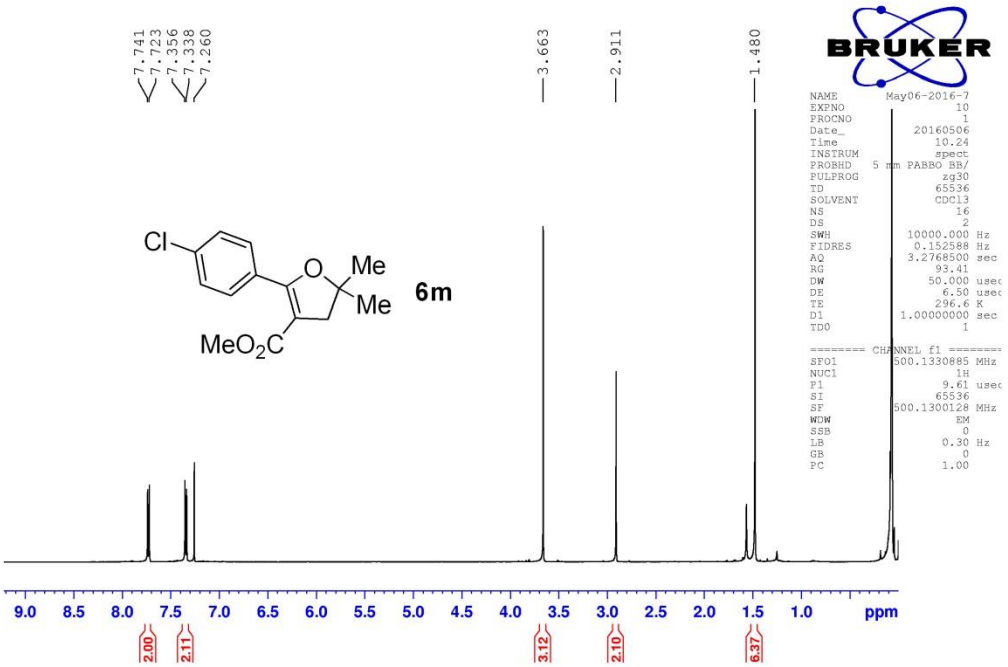

166.23
164.23
161.10
130.96
122.78
112.97
99.86
85.09
77.25
77.00
76.74
55.27
50.79
44.27
28.09
MeO
Me
Me
MeO2C
6j
BRUKER
NAME 13-11-05-CZP
EXPNO 11
PROCNO 1
Date_ 20160511
Time 12.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 448
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 301.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577922 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

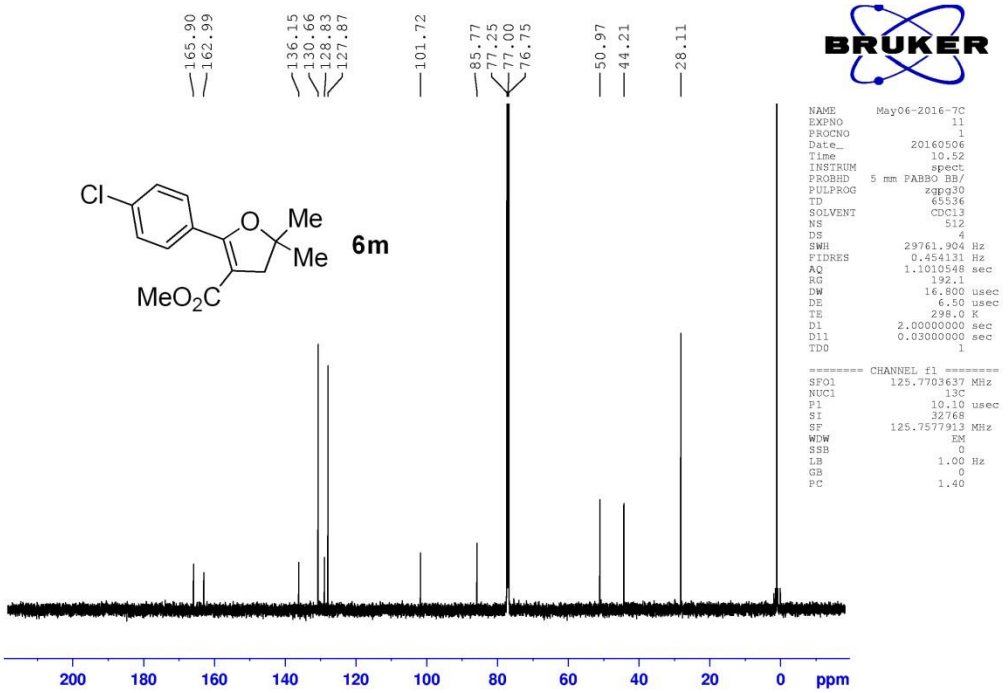

7.665
7.648
7.260
7.190
7.174
3.655
2.905
2.370
1.478
Me
O
Me
Me
MeO2C
6k
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.93
2.00
3.03
2.02
3.11
6.11

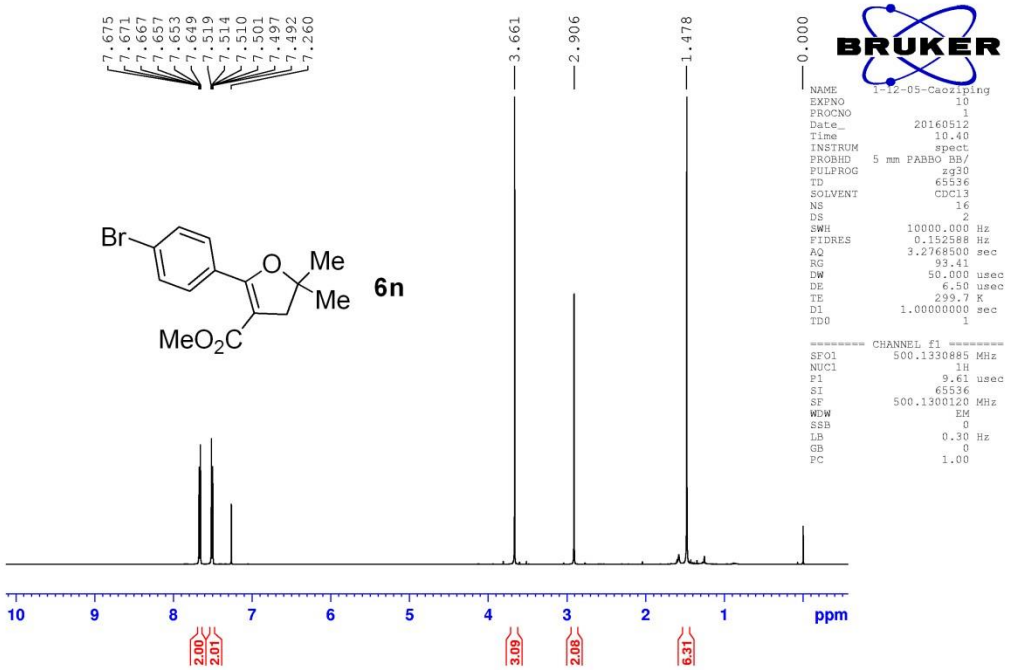

166.13
164.56
140.44
129.13
128.30
127.57
100.63
85.37
77.25
77.00
76.75
50.83
44.21
28.12
21.51
Me
O
Me
Me
MeO2C
6k
200 180 160 140 120 100 80 60 40 20 0 ppm

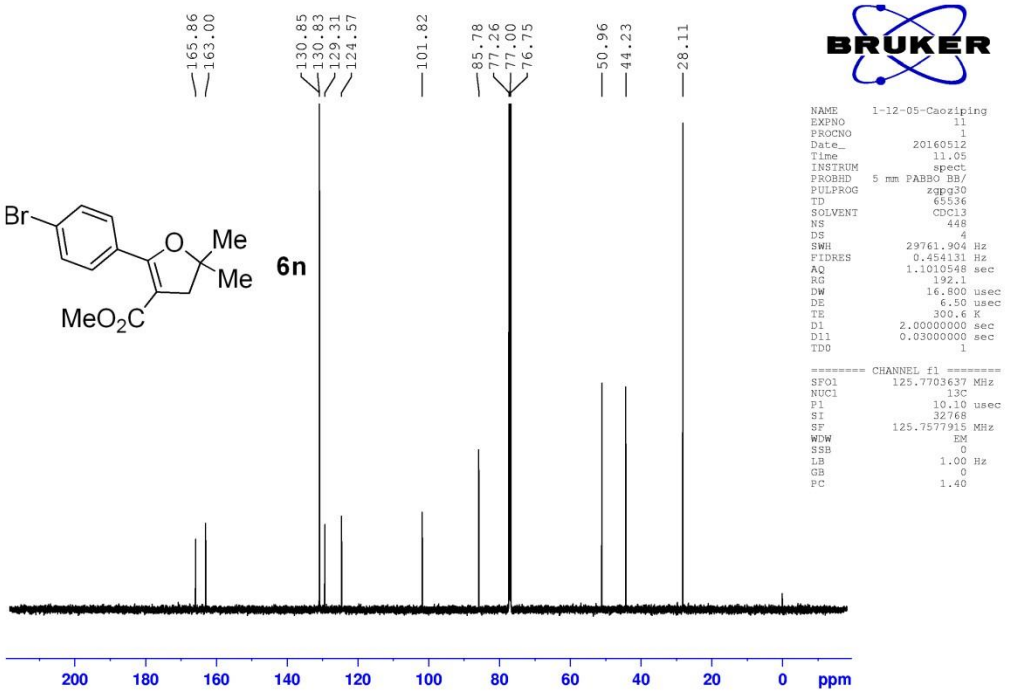

7.824
7.819
7.814
7.808
7.805
7.801
7.794
7.790
7.260
7.081
7.075
7.071
7.061
7.058
7.053
7.044
7.040
3.664
2.913
1.482
F
O
Me
Me
MeO2C
6l
NAME 18-01-04-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160401
Time 11.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 87.51
DW 50.000 usec
DE 6.50 usec
TE 298.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.94
2.00
3.07
2.08
6.25

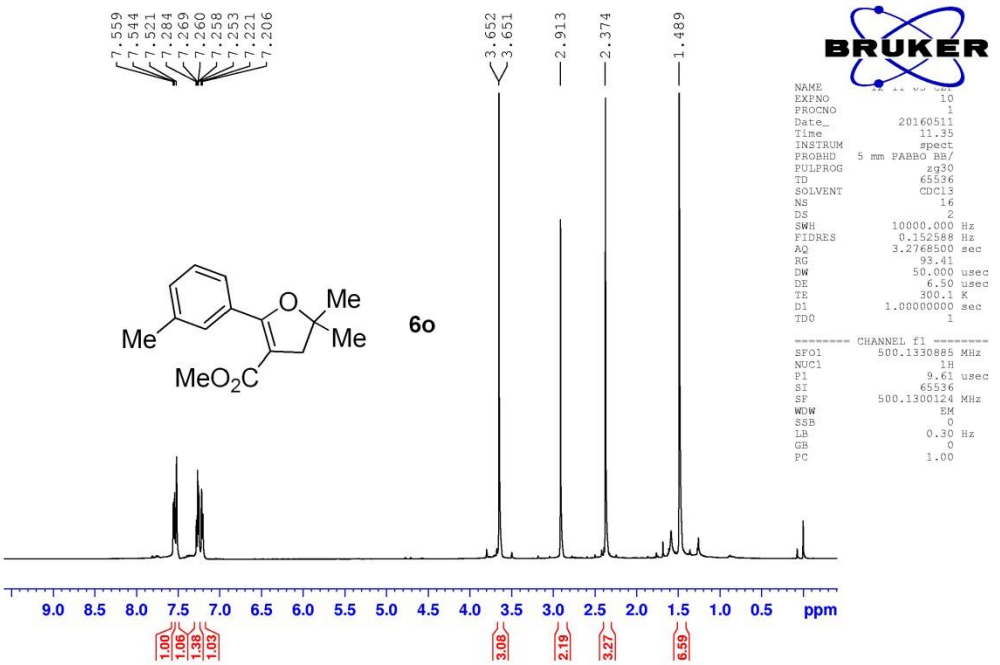

165.99
164.72
163.22
162.73
131.51
131.45
126.50
126.48
114.74
114.57
101.14
85.61
77.26
77.00
76.75
50.92
44.19
28.10
F
O
Me
Me
MeO2C
6l
NAME 18-01-04-Caoziping
EXPNO 11
PROCNO 1
Date_ 20160401
Time 11.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 384
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 299.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577918 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

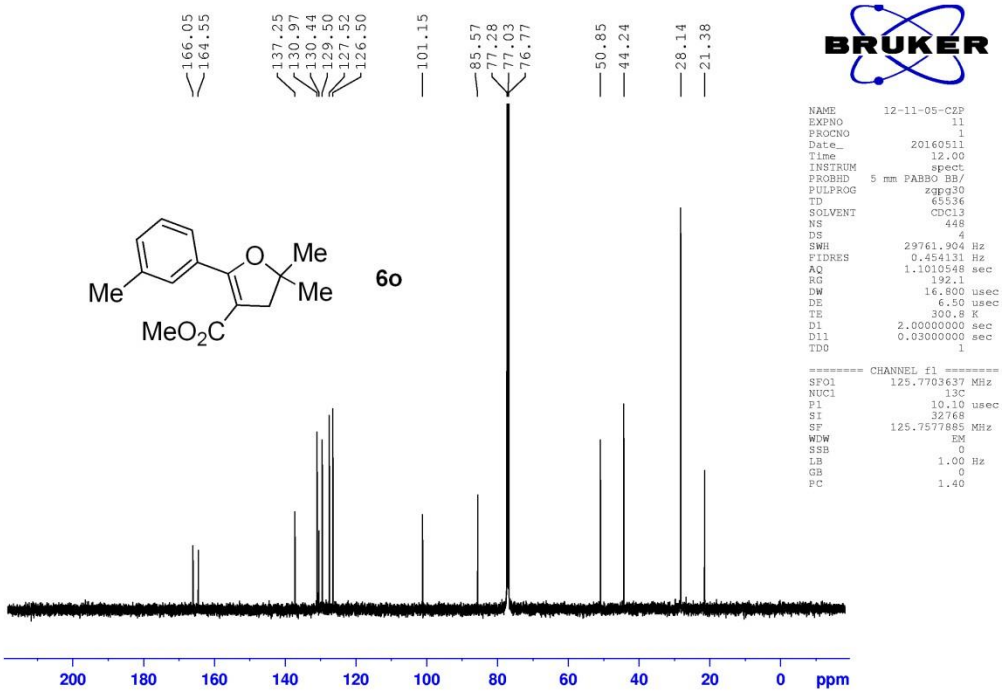

7.741
7.723
7.356
7.338
7.260
3.663
2.911
1.480
Cl
O
Me
Me
MeO2C
6m
BRUKER
NAME May06-2016-7
EXPNO 10
PROCNO 1
Date_ 20160506
Time 10.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 93.41
DW 50.000 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
2.00
2.11
3.12
2.10
6.37

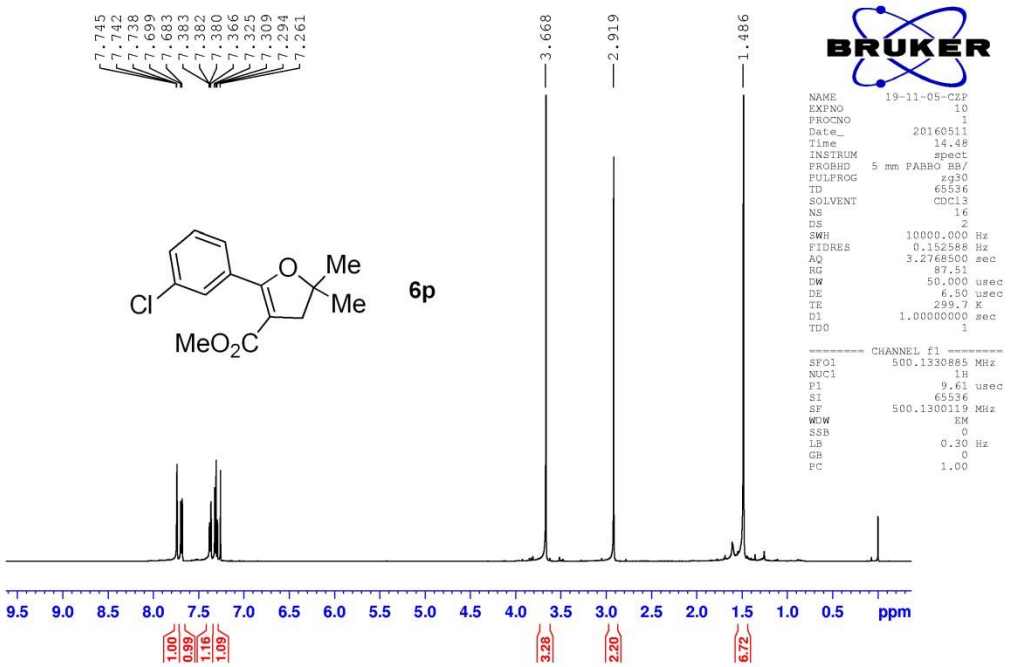

165.90
162.99
136.15
130.66
128.83
127.87
101.72
85.77
77.25
77.00
76.75
50.97
44.21
28.11
Cl
O
Me
Me
MeO2C
6m
BRUKER
NAME May06-2016-7C
EXPNO 11
PROCNO 1
Date_ 20160506
Time 10.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577913 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

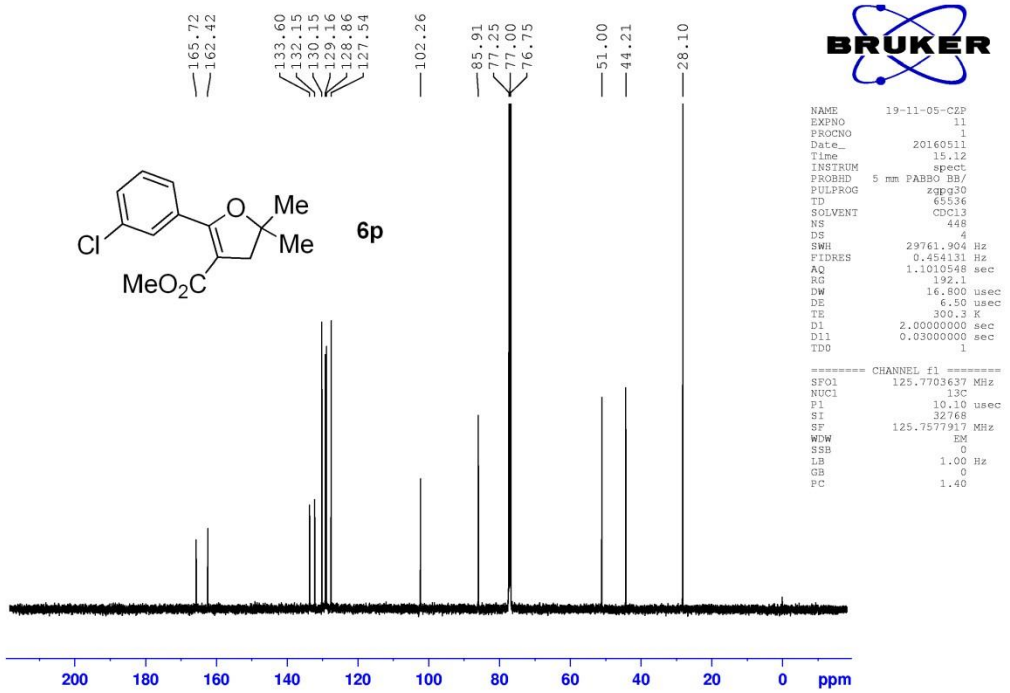

7.675
7.671
7.667
7.657
7.653
7.649
7.519
7.514
7.510
7.501
7.497
7.492
7.260
3.661
2.906
1.478
0.000
Br
O
Me
Me
MeO2C
6n
10
9
8
7
6
5
4
3
2
1
ppm
2.00
2.01
3.09
2.08
6.31
BRUKER
NAME 1-12-05-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160512
Time 10.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 93.41
DW 50.000 usec
DE 6.50 usec
TE 299.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

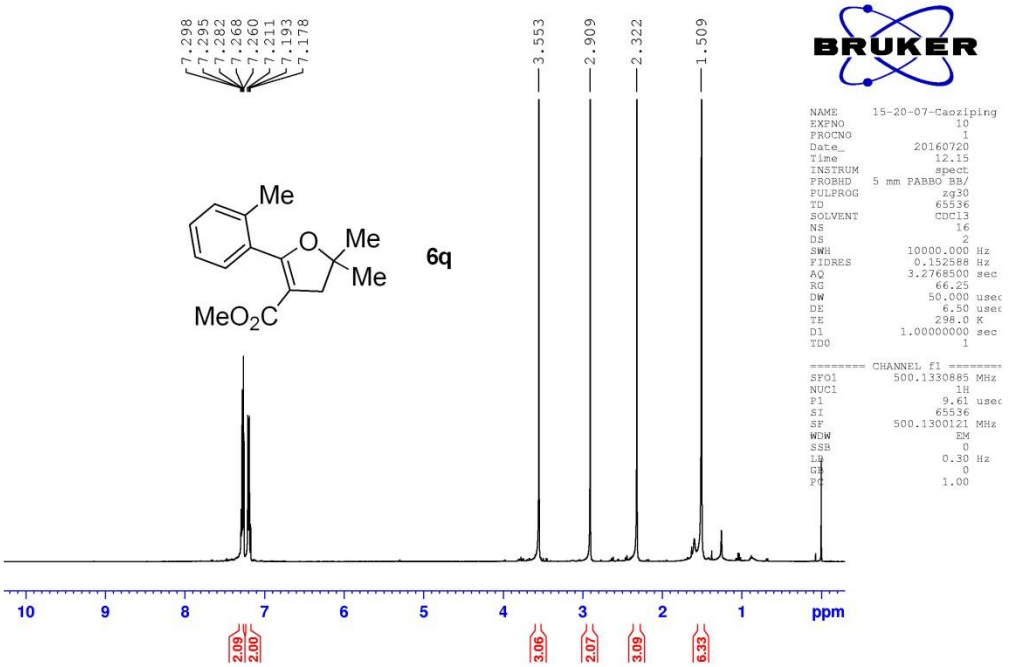

165.86
163.00
130.85
130.83
129.31
124.57
101.82
85.78
77.26
77.00
76.75
50.96
44.23
28.11
Br
O
Me
Me
MeO2C
6n
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME 1-12-05-Caoziping
EXPNO 11
PROCNO 1
Date_ 20160512
Time 11.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 448
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 300.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577915 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

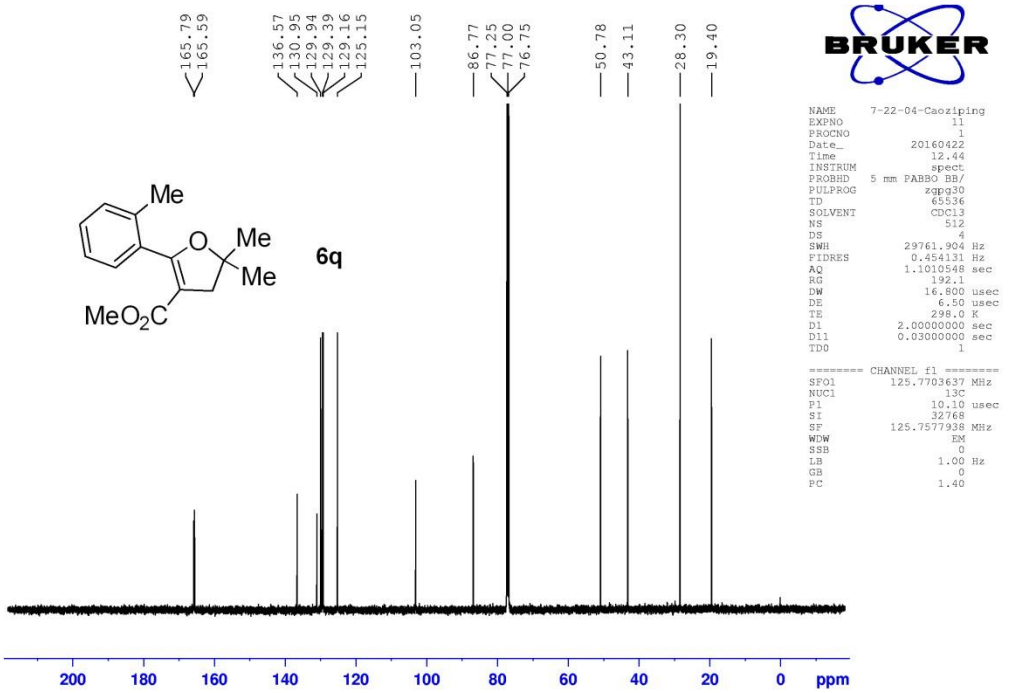

7.559
7.544
7.521
7.284
7.269
7.260
7.258
7.253
7.221
7.206
3.652
3.651
2.913
2.374
1.489
Me
O
Me
Me
MeO2C
6o
BRUKER
NAME
EXPNO 10
PROCNO 1
Date_ 20160511
Time 11.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 93.41
DW 50.000 usec
DE 6.50 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300124 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
1.00
1.06
1.38
1.03
3.08
2.19
3.27
6.59

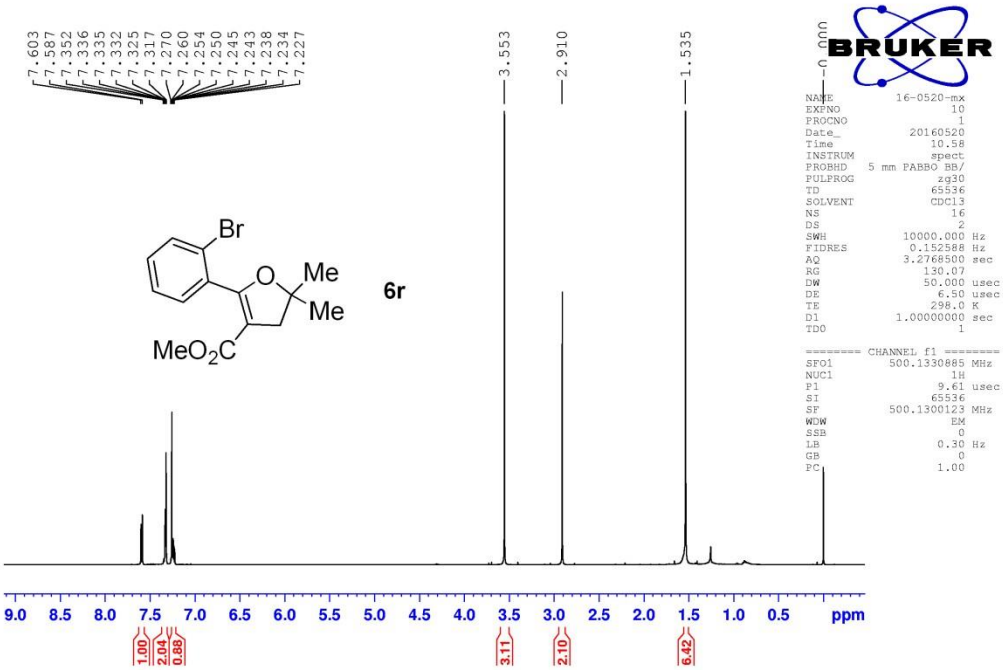

166.05
164.55
137.25
130.97
130.44
129.50
127.52
126.50
101.15
85.57
77.28
77.03
76.77
50.85
44.24
28.14
21.38
Me
O
Me
Me
MeO2C
6o
BRUKER
NAME 12-11-05-CZP
EXPNO 11
PROCNO 1
Date_ 20160511
Time 12.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 448
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 300.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

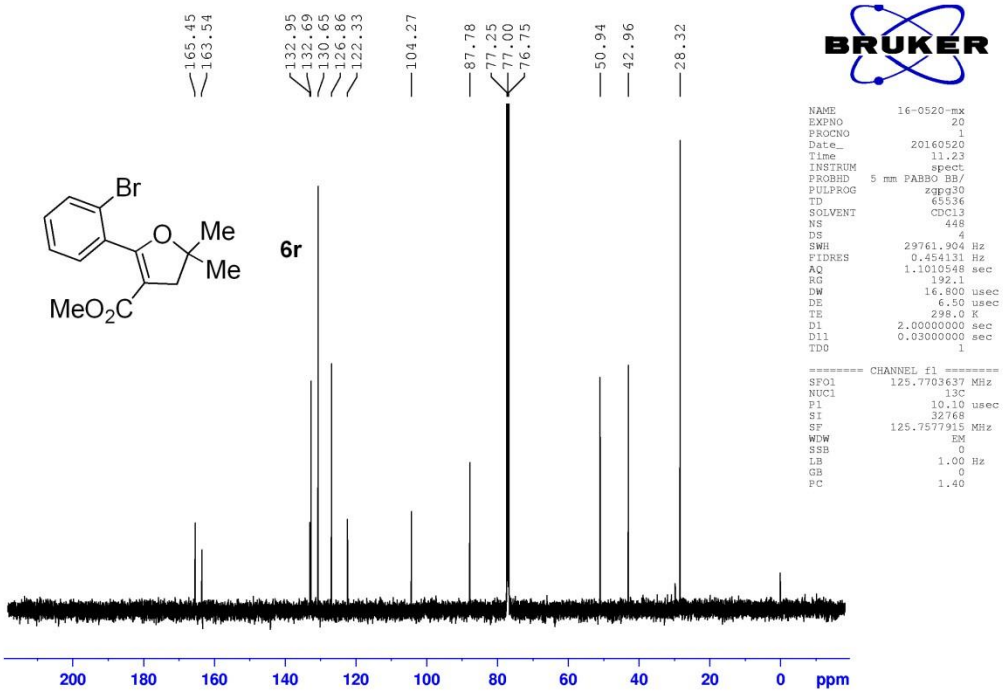

7.745
7.742
7.738
7.699
7.683
7.383
7.382
7.380
7.366
7.325
7.309
7.294
7.261
3.668
2.919
1.486
Cl
O
Me
Me
MeO2C
6p
BRUKER
NAME 19-11-05-CZP
EXPNO 10
PROCNO 1
Date_ 20160511
Time 14.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 87.51
DW 50.000 usec
DE 6.50 usec
TE 299.7 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300119 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
1.00
0.99
1.16
1.09
3.28
2.20
6.72

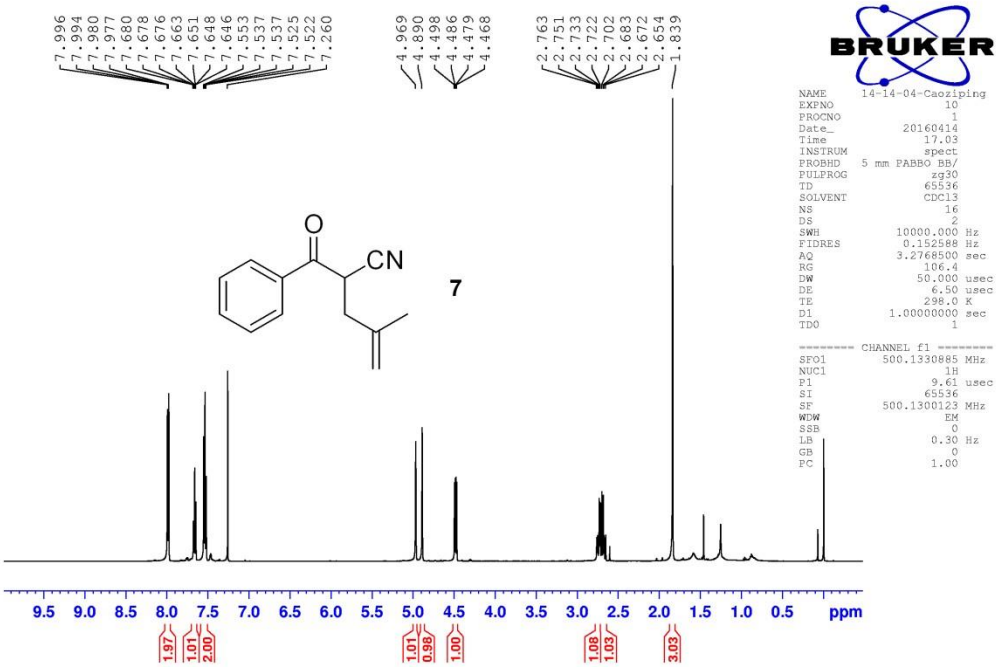

165.72
162.42
133.60
132.15
130.15
129.16
128.86
127.54
102.26
85.91
77.25
77.00
76.75
51.00
44.21
28.10
Cl
O
Me
Me
MeO2C
6p
BRUKER
NAME 19-11-05-CZP
EXPNO 11
PROCNO 1
Date_ 20160511
Time 15.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 448
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 300.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577917 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

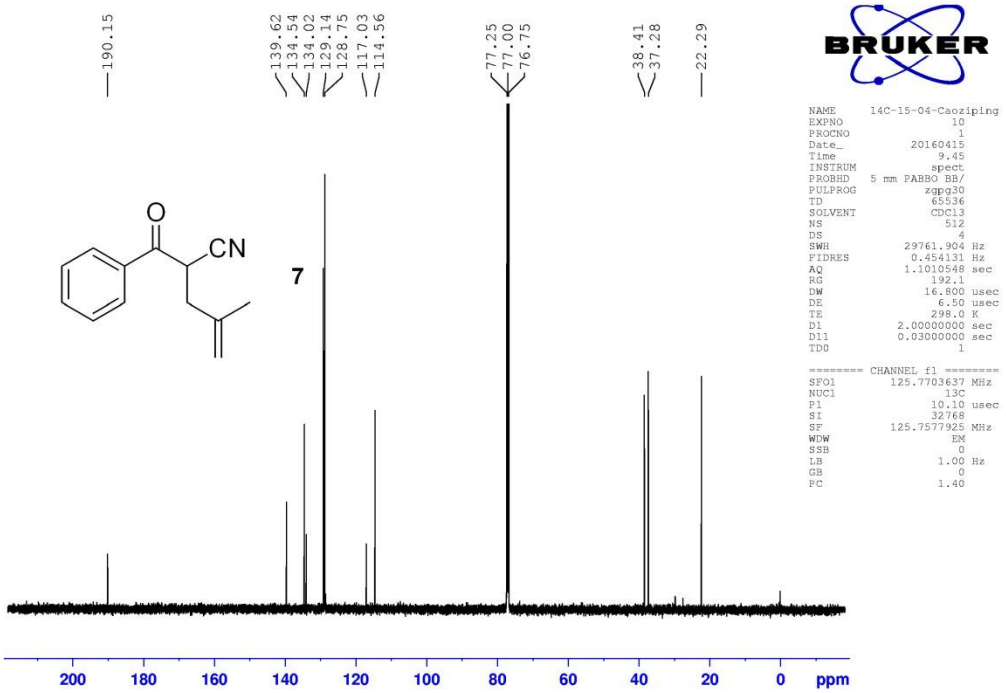

7.298
7.295
7.282
7.268
7.260
7.211
7.193
7.178
3.553
2.909
2.322
1.509
Me
Me
Me
O
MeO2C
6q
10
9
8
7
6
5
4
3
2
1
ppm
2.09
2.00
3.06
2.07
3.09
6.33
BRUKER
NAME 15-20-07-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160720
Time 12.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 66.25
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

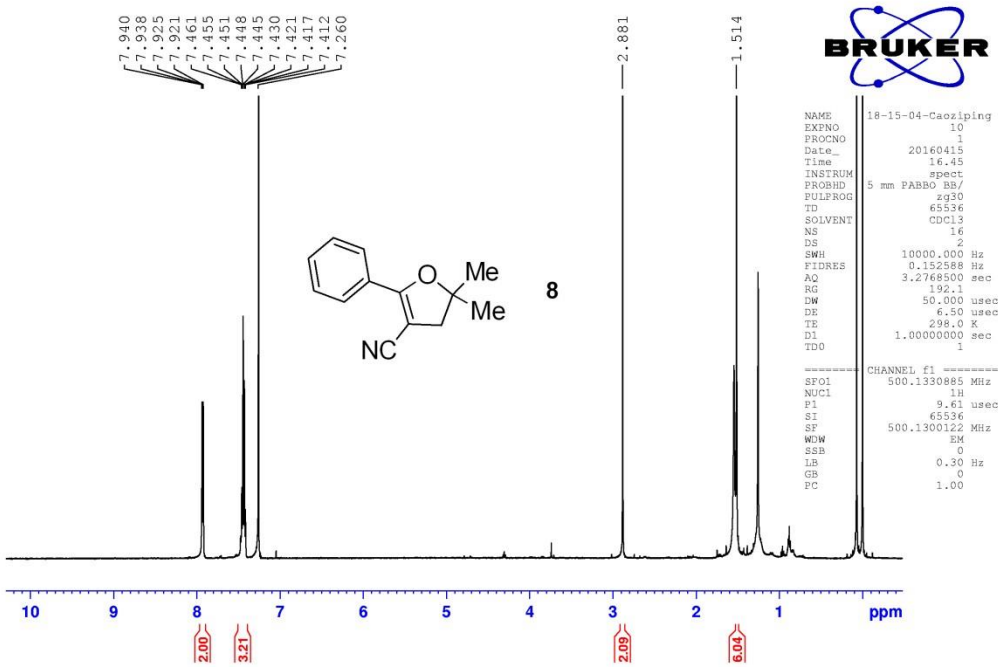

165.79
165.59
136.57
130.95
129.94
129.39
129.16
125.15
103.05
86.77
77.25
77.00
76.75
50.78
43.11
28.30
19.40
Me
Me
Me
O
MeO2C
6q
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME 7-22-04-Caoziping
EXPNO 11
PROCNO 1
Date_ 20160422
Time 12.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577938 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

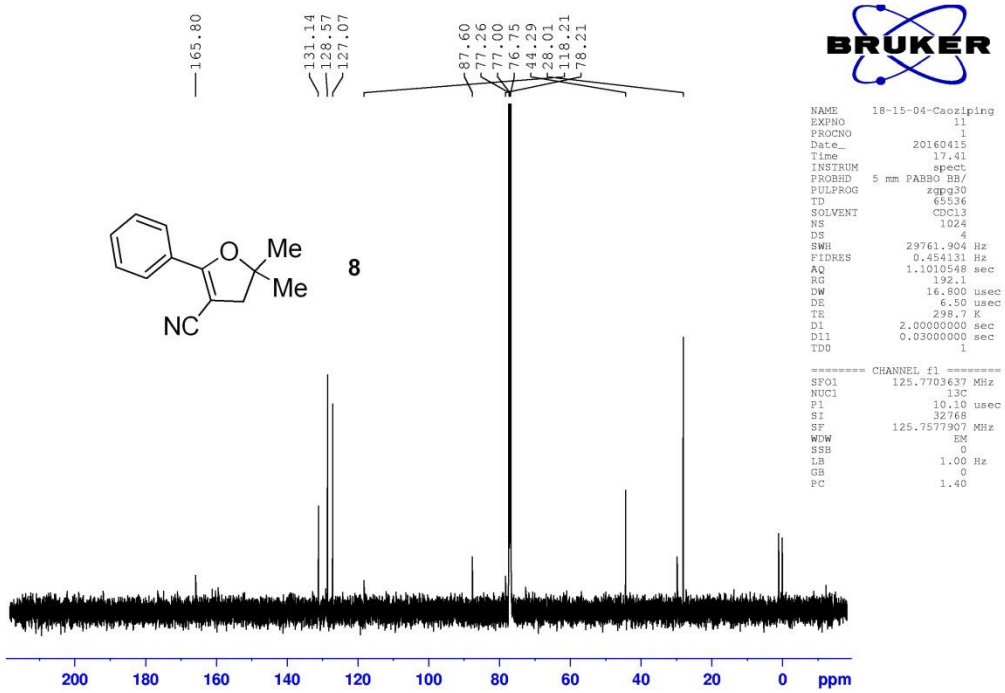

7.603
7.587
7.352
7.336
7.335
7.332
7.325
7.317
7.270
7.260
7.254
7.250
7.245
7.243
7.238
7.234
7.227
3.553
2.910
1.535
Br
O
Me
Me
MeO2C
6r
BRUKER
NAME 16-0520-mx
EXPNO 10
PROCNO 1
Date_ 20160520
Time 10.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 130.07
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
1.00
2.04
0.88
3.11
2.10
6.42


165.45
163.54
132.95
132.69
130.65
126.86
122.33
104.27
87.78
77.25
77.00
76.75
50.94
42.96
28.32
Br
O
Me
Me
MeO2C
6r
BRUKER
NAME 16-0520-mx
EXPNO 20
PROCNO 1
Date_ 20160520
Time 11.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 448
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577915 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm


7.996
7.994
7.980
7.977
7.680
7.678
7.676
7.663
7.651
7.648
7.646
7.553
7.537
7.537
7.525
7.522
7.260
4.969
4.890
4.498
4.486
4.479
4.468
2.763
2.751
2.733
2.722
2.702
2.683
2.672
2.654
1.839
O
CN
7
BRUKER
NAME 14-14-04-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160414
Time 17.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 106.4
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
1.97
1.01
2.00
1.01
0.98
1.00
1.08
1.03
3.03


190.15
139.62
134.54
134.02
129.14
128.75
117.03
114.56
77.25
77.00
76.75
38.41
37.28
22.29
O
CN
7
BRUKER
NAME 14C-15-04-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160415
Time 9.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577925 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm


7.940
7.938
7.925
7.921
7.461
7.455
7.451
7.448
7.445
7.430
7.421
7.417
7.412
7.260
2.881
1.514
BRUKER
NAME 18-15-04-Caoziping
EXPNO 10
PROCNO 1
Date_ 20160415
Time 16.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 192.1
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.61 usec
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
Me
Me
NC
8
10
9
8
7
6
5
4
3
2
1
ppm
2.00
3.21
2.09
6.04


165.80
131.14
128.57
127.07
87.60
77.26
77.00
76.75
44.29
28.01
118.21
78.21
BRUKER
NAME 18-15-04-Caoziping
EXPNO 11
PROCNO 1
Date_ 20160415
Time 17.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.10 usec
SI 32768
SF 125.7577907 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
Me
Me
NC
8
200
180
160
140
120
100
80
60
40
20
0
ppm