SUPPORTING INFORMATION

Nickel-Catalyzed Ring-Opening of α-Hydroxylcyclobutenones with Remarkable Ligand Effect

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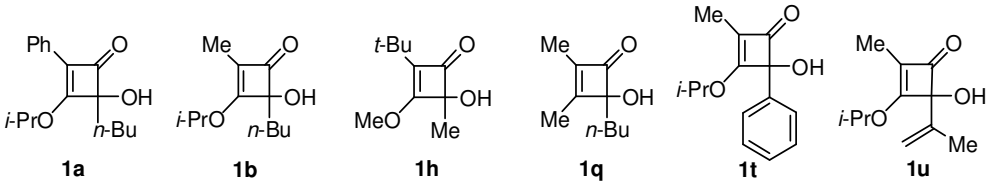
1. General Information

Column chromatography was carried out on 200-300 Mesh silica gel. And thin layer chromatography (TLC) was performed on silica gel GF254 plates. High resolution Mass spectra (HRMS) were recorded in ESI mode using a TOF analyzer. ^{1}H NMR spectra were recorded on 400 MHz in $CDCl_3$ and ^{13}C NMR spectra were recorded on 100 MHz in $CDCl_3$ using TMS as internal standard. All new products were further characterized by HRMS; copies of their ^{1}H NMR and ^{13}C NMR spectra are provided. Unless otherwise stated, all reagents and solvents were purchased from commercial suppliers and used without further purification. Data for ^{1}H NMR are reported as follows: chemical shift (ppm), multiplicity (s = singlet; d = doublet; t = triplet; q = quartet; hept = heptet; m = multiplet), coupling constants, *J*, in (Hz), and integration.

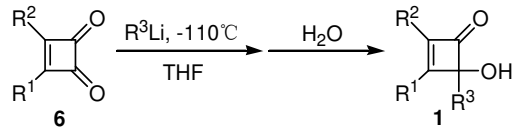
2. Preparation of α-Hydroxylcyclobutenones

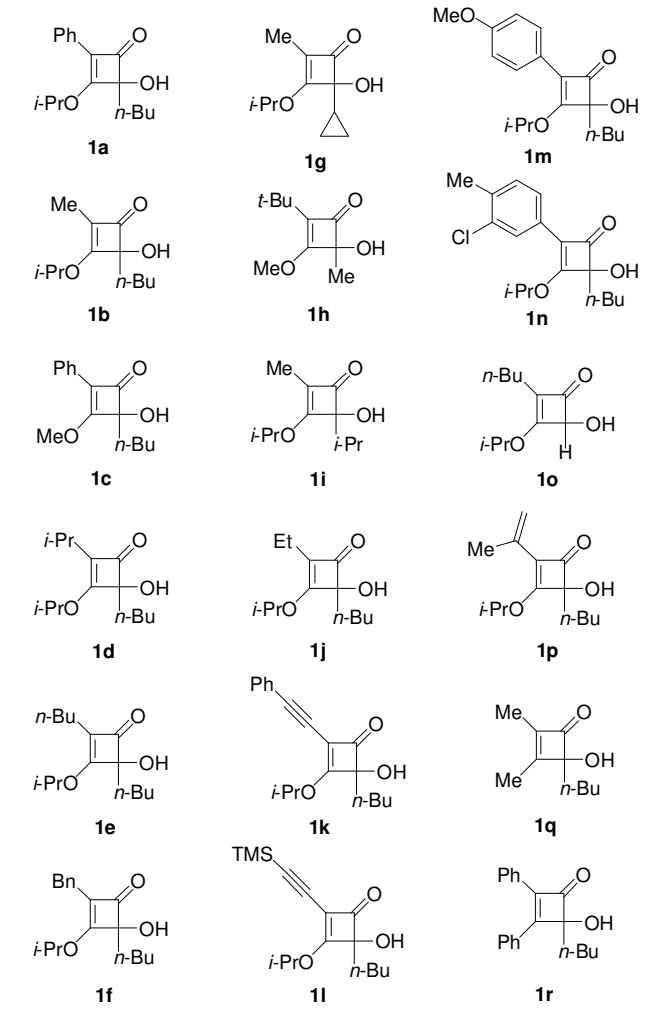


1a **1b** **1h** **1q** **1t** **1u**

α-hydroxycyclobutenones of **1a**, **1b**, **1h**, **1q**, **1t** and **1u** were prepared according to previous report. (Song, P.; Li, Q.; Wang, C.; Wu, W.; Mao, X.; Wang, J.; Hu, X. *Adv. Synth. Catal.* **2016**, *358*, 1208-1212.)

Preparation of other α-Hydroxylcyclobutenones





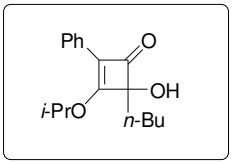
General procedure:[1]

A solution of R^3Li (1 mmol, 2.5M in THF) was added to the solution of cyclobutenediones **6** (0.5 M in THF, 1.1mmol) at -110℃ under N_2. The system was stirred for 5 minutes, and then quenched with saturated ammonium chloride solution. The aqueous phase was extracted with EtOAc three times. The combined organic layer was washed with saturated ammonium chloride solution, saturated brine and dried over Na_2SO_4. The solvent was removed under reduced pressure. The crude residue was purified by silica gel chromatography using petroleum ether/ethyl acetate as eluent to afford **1**.

Reference:

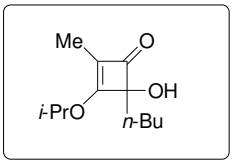
(1) Tomooka, C. S.; Liu, H.; Moore, H. W. *J. Org. Chem.* **1996**, *61*, 6009-6012.

4-Butyl-4-hydroxy-3-isopropoxy-2-phenyl-cyclobut-2-enone (1a)



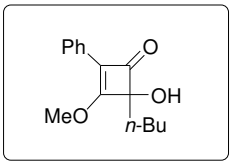
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* =7.4 Hz, 2H), 7.29 (t, *J* =7.4 Hz, 2H), 7.21 (t, *J* = 7.4 Hz, 1H), 5.31 (s, 1H), 5.13 (hept, *J* = 6.0 Hz, 2H), 2.19-2.12 (m, 1H), 1.93-1.87 (m, 1H), 1.53 (d, *J* = 6.0 Hz, 3H), 1.46 (d, *J* = 6.0 Hz, 3H), 1.34-1.30 (m, 4H), 0.86 (t, *J* = 6.6 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.9, 182.1, 128.8, 128.2, 127.5, 126.7, 123.1, 93.0, 78.5, 33.6, 27.4, 23.1, 23.0, 22.7, 13.8. **HRMS** Calcd (ESI) m/z for $C_{17}H_{22}NaO_3^+$: $[M+Na]^+$ 297.1461, Found: 297.1467. **IR** (neat) ν_{max} 3238, 2957, 2926, 1735, 1619, 1337, 759, 698.

4-Butyl-4-hydroxy-3-isopropoxy-2-methyl-cyclobut-2-enone (1b)



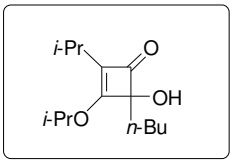
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.84 (hept, *J* = 4.0 Hz, 1H), 3.12 (s, 1H), 1.90-1.83 (m, 1H), 1.80-1.75 (m, 1H), 1.70 (s, 3H), 1.43 (d, *J* = 4.0 Hz, 6H), 1.35-1.26 (m, 4H), 0.89 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.8, 182.9, 120.8, 91.2, 76.4, 32.2, 27.0, 22.7, 22.6, 22.4, 13.8, 6.6. **HRMS** Calcd (ESI) m/z for $C_{12}H_{20}NaO_3^+$: $[M+Na]^+$ 235.1305, Found: 235.1303. **IR** (neat) ν_{max} 3308, 2954, 2931, 1745, 1605, 1313.

4-Butyl-4-hydroxy-3-methoxy-2-phenyl-cyclobut-2-enone (1c)



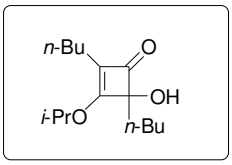
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.4 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 1H). 7.26-7.23 (m, 1H), 7.28-7.20 (m, 1H), 4.59 (s, 1H), 4.26 (s, 3H), 2.18-2.11 (m, 1H), 1.94-1.86 (m, 1H), 1.34-1.23 (m, 4H), 0.87 (t, *J* = 6.4 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.5, 182.2, 128.4, 128.4, 127.9, 126.9, 124.1, 92.9, 59.7, 33.4, 27.5, 22.7, 13.8. **HRMS** Calcd (ESI) m/z for $C_{15}H_{18}NaO_3^+$: $[M+Na]^+$ 269.1148, Found: 269.1145. **IR** (neat) ν_{max} 3235, 2960, 2923, 1738, 1616, 1337, 761, 698.

4-Butyl-4-hydroxy-3-isopropoxy-2-isopropyl-cyclobut-2-enone (1d)



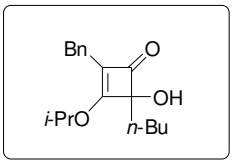
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.01 (s, 1H), 4.77 (hept, *J* = 6.1 Hz, 1H), 2.39-2.29 (m, *J* = 6.2 Hz, 1H), 1.89-1.81 (m, 1H), 1.69-1.62 (m, 1H), 1.30 (d, *J* = 6.1 Hz, 3H), 1.27 (d, *J* = 6.1 Hz, 3H), 1.24-1.17 (m, 2H), 1.15-1.07 (m, 2H), 1.03(d, *J* = 6.2 Hz, 3H), 1.01 (d, *J* = 6.2 Hz, 3H), 0.76 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.9, 181.7, 131.1, 91.3, 76.5, 32.6, 27.2, 23.7, 22.6, 22.5, 22.5, 20.2, 13.6. **HRMS** Calcd (ESI) m/z for $C_{14}H_{24}NaO_3^+$: $[M+Na]^+$ 263.1618, Found: 263.1616. **IR** (neat) ν_{max} 3367, 2963, 2931, 1743, 1607, 1338.

2,4-Dibutyl-4-hydroxy-3-isopropoxy-cyclobut-2-enone (1e)



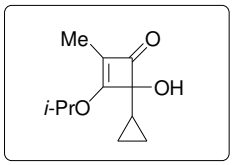
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.82 (hept, *J* = 6.6 Hz, 1H), 4.29 (s, 1H), 2.11-2.02 (m, 2H), 1.96-1.88 (m, 1H), 1.81-1.74 (m, 1H), 1.51-1.46 (m, 2H), 1.43(d, *J* = 6.6 Hz, 3H), 1.41 (d, *J* = 6.6 Hz, 3H), 1.35-1.23 (m, 6H), 0.92-0.87 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.5, 182.5, 126.1, 91.4, 76.5, 32.5, 29.5, 27.2, 22.7, 22.6, 22.5, 22.0, 13.8, 13.6. **HRMS** Calcd (ESI) m/z for $C_{15}H_{26}NaO_3^+$: $[M+Na]^+$ 277.1774, Found: 277.1772. **IR** (neat) ν_{max} 3270, 2969, 2932, 1746, 1602, 1317.

2-Benzyl-4-butyl-4-hydroxy-3-isopropoxy-cyclobut-2-enone (1f)



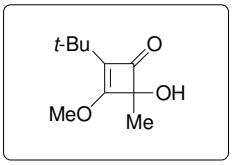
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28-7.16 (m, 5H), 4.77 (hept, *J* = 6.0 Hz, 1H), 4.16 (s, 1H), 3.48-3.37 (m, 2H), 1.96-1.90 (m, 1H), 1.83-1.75 (m, 1H), 1.34 (d, *J* = 6.0 Hz, 6H), 1.30-1.18 (m, 4H), 0.84 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.0, 183.2, 137.9, 128.4, 128.2, 126.2, 123.8, 91.6, 77.2, 32.6, 27.9, 27.2, 22.7, 22.6, 22.5, 13.8. **HRMS** Calcd (ESI) m/z for $C_{18}H_{24}NaO_3^+$: $[M+Na]^+$ 311.1618, Found: 311.1620. **IR** (neat) ν_{max} 3229, 2957, 1742, 1613, 1333, 756, 699.

4-Cyclopropyl-4-hydroxy-3-isopropoxy-2-methyl-cyclobut-2-enone (1g)



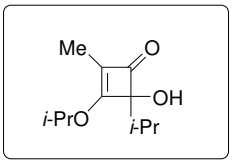
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.87 (hept, *J* = 6.2 Hz, 1H), 3.86 (s, 1H), 1.67 (s, 3H), 1.44 (d, *J* = 6.2 Hz, 6H), 1.28-1.19 (m, 1H), 0.67-0.61 (m, 1H), 0.59-0.52 (m, 2H), 0.46-0.51 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.9, 182.5, 121.1, 90.7, 76.6, 22.5, 13.9, 6.5, 2.6, 2.4. **HRMS** Calcd (ESI) m/z for $C_{11}H_{16}NaO_3^+$: $[M+Na]^+$ 219.0992, Found: 219.0990. **IR** (neat) ν_{max} 3370, 2982, 2931, 1753, 1610, 1315.

2-tert-Butyl-4-hydroxy-3-methoxy-4-methyl-cyclobut-2-enone (1h)



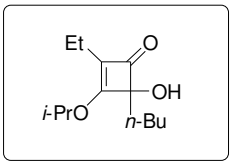
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.15 (s, 3H), 1.62 (s, 3H), 1.15 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.5, 183.6, 133.4, 87.7, 77.4, 77.1, 76.8, 58.9, 30.7, 28.0, 20.6. **HRMS** Calcd (ESI) m/z for $C_{10}H_{16}NaO_3^+$: $[M+Na]^+$ 207.0992, Found: 207.0995. **IR** (neat) ν_{max} 3272, 2963, 2934, 1745, 1613, 1349.

4-Hydroxy-3-isopropoxy-4-isopropyl-2-methyl-cyclobut-2-enone (1i)



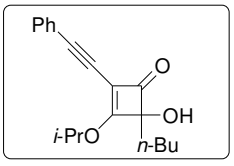
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.78 (hept, *J* = 2.8 Hz, 1H), 2.84 (s, 1H), 2.14-2.03 (m, *J* = 7.0 Hz, 1H), 1.73 (s, 3H), 1.44 (d, *J* = 2.8 Hz, 3H), 1.43 (d, *J* = 2.8 Hz, 3H), 1.04 (d, *J* = 7.0 Hz, 3H), 1.02 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.8, 181.8, 120.9, 93.9, 76.2, 31.4, 22.7, 22.3, 18.0, 17.9, 7.0. **HRMS** Calcd (ESI) m/z for $C_{11}H_{18}NaO_3^+$: $[M+Na]^+$ 221.1148, Found: 221.1156. **IR** (neat) ν_{max} 3278, 2982, 2955, 1747, 1605, 1313.

4-Butyl-2-ethyl-4-hydroxy-3-isopropoxy-cyclobut-2-enone (1j)



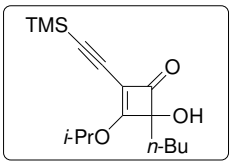
^{1}H NMR (400 MHz, $CDCl_3$) δ ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.83 (hept, *J* = 6.0 Hz, 1H), 3.91 (s, 1H), 2.16-2.05 (m, 2H), 1.95-1.87 (m, 1H), 1.81-1.74 (m, 1H), 1.43 (d, *J* = 6.0 Hz, 2H), 1.42 (d, *J* = 6.0 Hz, 2H), 1.36-1.19 (m, 4H), 1.10 (t, *J* = 7.6 Hz, 3H), 0.89 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.5, 182.2, 127.0, 91.3, 76.6, 32.4, 27.1, 22.7, 22.6, 22.4, 15.7, 13.8, 12.0. **HRMS** Calcd (ESI) m/z for $C_{13}H_{22}NaO_3^+$: $[M+Na]^+$ 249.1461, Found: 249.1468. **IR** (neat) ν_{max} 3372, 2966, 2932, 1749, 1336.

4-Butyl-4-hydroxy-3-isopropoxy-2-phenylethynyl-cyclobut-2-enone (1k)



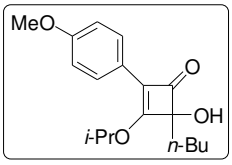
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45-7.42 (m, 2H), 7.34-7.31 (m, 3H), 5.30 (hept, *J* = 2.6 Hz, 1H), 3.52 (s, 1H), 1.94-1.81 (m, 2H), 1.54 (d, *J* = 2.6 Hz, 3H), 1.53 (d, *J* = 2.6 Hz, 3H), 1.39-1.32 (m, 4H), 0.90 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 184.5, 131.6, 128.8, 128.3, 122.2, 106.3, 92.7, 92.0, 79.0, 76.1, 32.1, 26.6, 22.8, 22.4, 22.1, 13.8. **HRMS** Calcd (ESI) m/z for $C_{19}H_{22}NaO_3^+$: $[M+Na]^+$ 321.1461, Found: 321.1454. **IR** (neat) ν_{max} 3299, 2969, 2928, 1748, 1606, 1332.

4-Butyl-4-hydroxy-3-isopropoxy-2-trimethylsilanylethynyl-cyclobut-2-enone (1l)



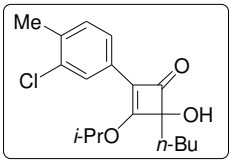
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.19 (hept, *J* = 6.2 Hz, 1H), 2.01 (s, 1H), 1.82-1.72 (m, 2H), 1.46 (d, *J* = 6.2 Hz, 6H), 1.35-1.26 (m, 4H), 0.86 (t, *J* = 6.7 Hz, 3H), 0.16 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 190.2, 184.9, 106.5, 99.2, 91.8, 91.1, 78.9, 60.4, 32.0, 26.5, 22.7, 22.4, 22.1, 13.8, -0.4. **HRMS** Calcd (ESI) m/z for $C_{16}H_{26}NaO_3Si^+$: $[M+Na]^+$ 317.1543, Found: 317.1538. **IR** (neat) ν_{max} 3293, 2971, 2931, 1753, 1596, 1315.

4-Butyl-4-hydroxy-3-isopropoxy-2-(4-methoxy-phenyl)-cyclobut-2-enone (1m)



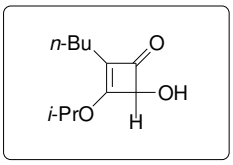
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.9 Hz, 2H), 6.81 (d, *J* = 8.9 Hz, 2H), 5.08 (hept, *J* = 6.1 Hz, 1H), 4.84 (s, 1H), 3.78 (s, 3H), 2.15-2.08 (m, 1H), 1.91-1.84 (m, 1H), 1.51 (d, *J* = 6.1 Hz, 3H), 1.46 (d, *J* = 6.1 Hz, 3H), 1.38-1.26 (m, 4H), 0.86 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.6, 180.6, 158.9, 128.2, 123.2, 121.6, 113.6, 93.0, 78.1, 55.1, 33.6, 27.4, 26.8, 23.1, 23.0, 22.7, 13.8. **HRMS** Calcd (ESI) m/z for $C_{18}H_{24}NaO_4^+$: $[M+Na]^+$ 327.1567, Found: 327.1560. **IR** (neat) ν_{max} 3233, 2963, 2926, 1735, 1603, 1303, 846.

4-Butyl-2-(3-chloro-4-methyl-phenyl)-4-hydroxy-3-isopropoxy-cyclobut-2-enone (1n)



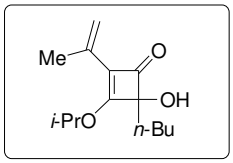
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (s, 1H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.10(d, *J* = 8.0 Hz, 1H), 5.12 (hept, *J* = 8.0 Hz, 1H), 4.58 (s, 1H), 2.33 (s, 3H), 2.14-2.04 (m, 1H), 1.91-1.84 (m, 1H), 1.56 (d, *J* = 8.0 Hz, 3H), 1.50 (d, *J* = 8.0 Hz, 3H), 1.34-1.23 (m, 4H), 0.87 (t, *J* = 8.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.5, 182.3, 135.4, 134.2, 130.7, 127.8, 126.9, 124.9, 121.9, 93.2, 78.9, 33.7, 27.4, 23.2, 23.0, 22.8, 20.0, 13.8. **HRMS** Calcd (ESI) m/z for $C_{18}H_{23}ClNaO_3^+$: $[M+Na]^+$ 345.1228, Found: 345.1221. **IR** (neat) ν_{max} 3248, 2957, 2927, 1736, 1594, 1338 900, 824.

2-Butyl-4-hydroxy-3-isopropoxy-cyclobut-2-enone (1o)



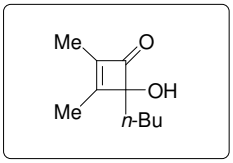
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.21 (s, 1H), 4.93 (hept, *J* = 6.0 Hz, 1H), 2.05-2.01 (t, *J* = 7.5 Hz, 2H), 1.52-1.46 (m, 2H), 1.44 (d, *J* = 6.0 Hz, 3H), 1.40 (d, *J* = 6.0 Hz, 3H), 1.35-1.26 (m, 2H), 0.96-0.84 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.2, 182.0, 127.1, 81.2, 77.3, 28.8, 23.1, 22.4, 21.4, 13.6. **HRMS** Calcd (ESI) m/z for $C_{11}H_{18}NaO_3^+$: $[M+Na]^+$ 221.1148, Found: 221.1145. **IR** (neat) ν_{max} 3343, 2959, 2931, 1749, 1613, 1341.

4-Butyl-4-hydroxy-2-isopropenyl-3-isopropoxy-cyclobut-2-enone (1p)



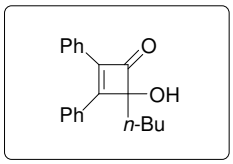
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.42 (s, 1H), 5.01-4.95 (m, 3H), 2.05-1.98 (m, 1H), 1.86 (s, 3H), 1.82-1.75 (m, 1H), 1.41 (d, *J* = 6.2 Hz, 3H), 1.37 (d, *J* = 6.2 Hz, 3H), 1.33-1.19 (m, 4H), 0.85 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.1, 181.8, 132.6, 124.9, 116.1, 92.4, 77.9, 33.5, 27.3, 22.9, 22.8, 22.7, 20.3, 13.8. **HRMS** Calcd (ESI) m/z for $C_{14}H_{22}NaO_3^+$: $[M+Na]^+$ 261.1461, Found: 261.1464. **IR** (neat) ν_{max} 3289, 2961, 2931, 1736, 1634, 1336.

4-Butyl-4-hydroxy-2,3-dimethyl-cyclobut-2-enone (1q)



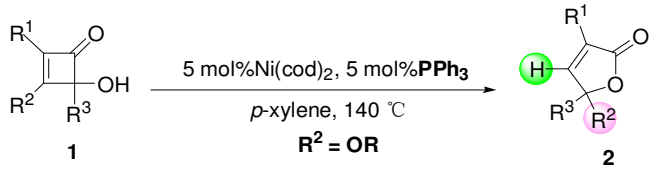
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.00 (s, 1H), 2.37-2.34 (m, 1H), 2.11 (s, 3H), 1.85-1.76 (m, 1H), 1.69 (s, 3H), 1.32-1.27 (m, 2H), 1.25-1.21 (m, 2H), 0.88 (t, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 197.5, 179.6, 147.3, 93.5, 32.3, 26.9, 22.8, 13.7, 10.5, 7.0. **HRMS** Calcd (ESI) m/z for $C_{10}H_{16}NaO_2^+$: $[M+Na]^+$ 191.1043, Found: 191.1045. **IR** (neat) ν_{max} 3396, 2959, 2929, 1752, 1637, 1309.

4-Butyl-4-hydroxy-2,3-diphenyl-cyclobut-2-enone (1r)



^{1}H NMR (400 MHz, $CDCl_3$) δ 9.50 (s, 1H), 7.71-7.69 (m, 2H), 7.45-7.35 (m, 4H), 7.31-7,21 (m, 3H), 2.49-2.41 (m, 1H), 2.23-2.16 (m, 1H), 1.35-1.21 (m, 4H), 0.84 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.2, 146.9, 146.6, 140.8, 131.3, 130.1, 129.0, 128.9, 128.4, 127.1, 126.6, 65.5, 31.7, 28.2, 23.1, 13.9. **HRMS** Calcd (ESI) m/z for $C_{20}H_{20}NaO_2^+$: $[M+Na]^+$ 315.1356, Found: 315.1346. **IR** (neat) ν_{max} 3189, 2955, 2924, 1729, 1641, 1345, 740, 695.

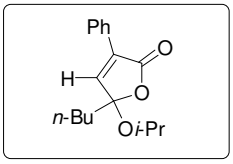
3. Nickel-Catalyzed Ring-Opening of α-Hydroxylcyclobutenones with PPh_3 as the Ligand



General procedure:

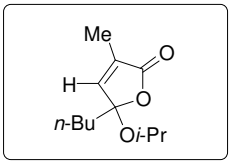
A solution of $Ni(cod)_2$ (0.025 mmol), α-hydroxylcyclotenone (**1**, 0.5 mmol), and PPh_3 (0.025 mmol) in *p*-xylene (2.5 mL) was stirred at 140 °C under N_2. After the complete consumption of **1**, solvent was removed under reduced pressure. Then the crude product was further purified by silica gel column chromatography using petroleum ether/ethyl acetate as eluent. The compound 2-furanone **2** was afforded.

5-Butyl-5-isopropoxy-3-phenyl-5H-furan-2-one (2a)



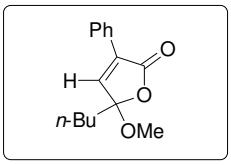
Yield: 85%; yellow solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91-7,89 (m, 2H), 7.46-7.44 (m, 3H), 3.79 (hept, *J* = 6.2 Hz, 1H), 1.99-1.95 (m, 2H), 1.44-1.31 (m, 4H), 1.22 (d, *J* = 6.2 Hz, 3H), 1.15 (d, *J* = 6.2 Hz, 3H), 0.91 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.3, 145.0, 134.3, 129.8, 128.9, 128.7, 127.4, 108.4, 66.8, 38.0, 25.5, 24.0, 23.8, 22.6, 13.9. **HRMS** Calcd (ESI) m/z for $C_{17}H_{22}NaO_3^+$: $[M+Na]^+$ 297.1461, Found: 297.1463. **IR** (neat) ν_{max} 3081, 2960, 2865, 1760, 1678, 1453, 1378, 1324, 1171, 1108, 750, 691.

5-Butyl-5-isopropoxy-3-methyl-5H-furan-2-one (2b)



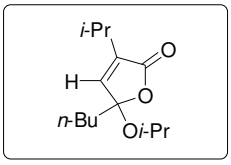
Yield: 70%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.73 (s, 1H), 3.66 (hept, *J* = 6.2 Hz, 1H), 1.95 (s, 3H), 1.85-1.81 (m, 2H), 1.39-1.28 (m, 4H), 1.15 (m, *J* = 8.0 Hz, 3H), 1.09 (m, *J* = 6.2 Hz, 3H), 0.98-0.80 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 175.5, 172.2, 96.2, 77.9, 73.3, 31.7, 26.4, 22.6, 22.3, 13.8, 8.6. **HRMS** Calcd (ESI) m/z for $C_{12}H_{20}NaO_3^+$: $[M+Na]^+$ 235.1305, Found: 235.1299. **IR** (neat) ν_{max} 3081, 2961, 2870, 1768, 1662, 1461, 1375, 1318, 1174, 1138.

5-Butyl-5-methoxy-3-phenyl-5H-furan-2-one (2c)



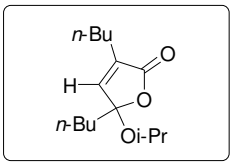
Yield: 91%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82-7.80 (m, 2H), 7.36-7.34 (m, 3H), 7.16 (s, 1H), 3.19 (s, 3H), 1.91-1.88 (m, 2H), 1.36-1.24 (m, 4H), 0.82 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.1, 144.6, 134.8, 129.8, 128.7, 128.7, 127.4, 108.3, 51.1, 37.1, 25.4, 22.6, 13.8. **HRMS** Calcd (ESI) m/z for $C_{15}H_{18}NaO_3^+$: $[M+Na]^+$ 269.1148, Found: 269.1160. **IR** (neat) ν_{max} 3074, 2961, 2861, 1751, 1631, 1455, 1378, 1304, 1171, 1114, 751, 697.

5-Butyl-5-isopropoxy-3-isopropyl-5H-furan-2-one (2d)



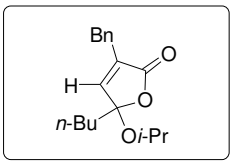
Yield: 82%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.65 (s, 1H), 3.63 (hept, *J* = 4.9 Hz, 1H), 2.70 (q, *J* = 6.2 Hz, 1H), 1.90-1.80 (m, 2H), 1.34-1.25 (m, 4H), 1.19 (d, *J* = 4.9 Hz, 3H), 1.18 (d, *J* = 4.9 Hz, 3H), 1.15 (d, *J* = 6.2 Hz, 3H), 1.09 (d, *J* = 6.2 Hz, 3H), 0.88 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.8, 143.9, 143.5, 109.0, 66.3, 37.8, 25.5, 25.4, 23.9, 23.7, 22.6, 21.1, 21.1, 13.9. **HRMS** Calcd (ESI) m/z for $C_{14}H_{24}NaO_3^+$: $[M+Na]^+$ 263.1618, Found: 263.1617. **IR** (neat) ν_{max} 3082, 2961, 2870, 1761, 1655, 1461, 1375, 1318, 1174, 1141.

3,5-Dibutyl-5-isopropoxy-5H-furan-2-one (2e)



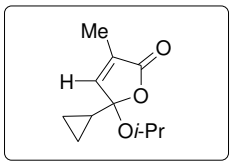
Yield: 80%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.69 (s, 1H), 3.64 (hept, *J* = 6.1 Hz, 1H), 2.31 (t, *J* = 7.6 Hz, 2H), 1.89-1.82 (m, *J* = 6.2 Hz, 2H), 1.58-1.51 (m, 2H), 1.42-1.34 (m, 2H), 1.31-1.25 (m, 4H), 1.15 (d, *J* = 6.1 Hz, 3H), 1.09 (d, *J* = 6.1 Hz, 3H), 0.93 (t, *J* = 7.6 Hz, 3H), 0.88 (t, *J* = 6.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.4, 145.4, 137.9, 109.4, 66.4, 37.9, 29.5, 25.5, 24.8, 24.0, 23.7, 22.6, 22.3, 13.9, 13.7. **HRMS** Calcd (ESI) m/z for $C_{15}H_{26}NaO_3^+$: $[M+Na]^+$ 277.1774, Found: 277.1772. **IR** (neat) ν_{max} 3080, 2961, 2868 1764, 1657, 1461, 1378, 1318, 1171, 1138.

3-Benzyl-5-butyl-5-isopropoxy-5H-furan-2-one (2f)



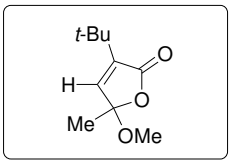
Yield: 80%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.35-7.21 (m, 5H), 6.53 (s, 1H), 3.70-3.57 (m, 3H), 1.85-1.79 (m, 2H), 1.30-1.26 (m, 4H), 1.14 (d, *J* = 6.2 Hz, 3H), 1.03 (d, *J* = 6.2 Hz, 3H), 0.86 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.9, 146.8, 137.4, 137.0, 128.8, 128.7, 126.8, 109.5, 66.4, 37.7, 31.5, 25.4, 23.9, 23.55, 22.5, 13.8. **HRMS** Calcd (ESI) m/z for $C_{18}H_{24}NaO_3^+$: $[M+Na]^+$ 311.1618, Found: 311.1614. **IR** (neat) ν_{max} 3068, 2961, 2868, 1764, 1663, 1458, 1375, 1317, 1171, 1134, 741, 697.

5-Cyclopropyl-5-isopropoxy-3-methyl-5H-furan-2-one (2g)



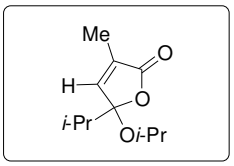
Yield: 52%; colorless liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.76 (s, 1H), 3.83 (hept, *J* = 6.1 Hz, 1H), 1.94 (s, 3H), 1.17 (d, *J* = 6.1 Hz, 3H), 1.11 (d, *J* = 6.1 Hz, 3H), 0.68-0.63 (m, 1H), 0.62-0.55 (m, 1H), 0.52-0.45 (m, 1H), 0.40-0.34 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.9, 146.5, 132.3, 108.0, 66.8, 23.9, 23.6, 16.8, 10.4, 2.4, 1.1. **HRMS** Calcd (ESI) m/z for $C_{11}H_{16}NaO_3^+$: $[M+Na]^+$ 219.0992, Found: 219.0996. **IR** (neat) ν_{max} 3088, 2973, 2874, 1766, 1631, 1451, 1378, 1308, 1185, 1131.

3-tert-Butyl-5-methoxy-5-methyl-5H-furan-2-one (2h)



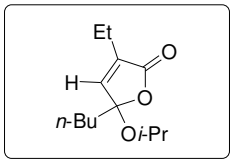
Yield: 78%; colorless liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.63 (s, 1H), 3.18 (s, 3H), 1.59 (s, 3H), 1.25 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.2, 145.9, 143.9, 105.6, 50.7, 31.7, 28.1, 23.7. **HRMS** Calcd (ESI) m/z for $C_{10}H_{16}NaO_3^+$: $[M+Na]^+$ 207.0992, Found: 207.0997. **IR** (neat) ν_{max} 3080, 2957, 2870, 1760, 1644, 1454, 1367, 1294, 1174, 1125.

5-Isopropoxy-5-isopropyl-3-methyl-5H-furan-2-one (2i)



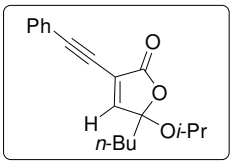
Yield: 61%; colorless liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.75 (s, 1H), 3.62 (hept, *J* = 6.1 Hz, 1H), 2.06 (hept, *J* = 6.9Hz, 1H), 1.97 (s, 3H), 1.15 (d, *J* = 6.1 Hz, 3H), 1.08 (d, *J* = 6.1 Hz, 3H), 0.98 (d, *J* = 6.9Hz, 3H), 0.93 (d, *J* = 6.9Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.8, 145.1, 133.9, 111.6, 66.3, 35.9, 23.9, 23.5, 17.1, 16.6, 10.5. **HRMS** Calcd (ESI) m/z for $C_{11}H_{18}NaO_3^+$: $[M+Na]^+$ 221.1148, Found: 221.1154. **IR** (neat) ν_{max} 3081, 2974, 2881, 1764, 1664, 1464, 1378, 1338, 1174, 1121.

5-Butyl-3-ethyl-5-isopropoxy-5H-furan-2-one (2j)



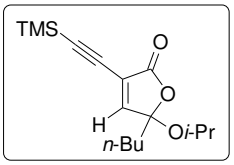
Yield: 63%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.71 (s, 1H), 4.12 (q, *J* = 7.1 Hz, 1H), 3.67 (hept, *J* = 7.5 Hz, 1H), 2.34 (q, *J* = 7.4 Hz, 2H), 1.87-1.83 (m, 2H), 1.33-1.31 (m, 4H), 1.20-1.15 (m, 6H), 1.10 (d, *J* = 7.5 Hz, 2H), 0.89 (t, *J* = 5.8 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.2, 144.7, 139.3, 109.4, 66.4, 60.3, 37.8, 25.5, 23.9, 23.7, 22.6, 21.0, 18.5, 14.1, 13.8, 11.7. **HRMS** Calcd (ESI) m/z for $C_{13}H_{22}NaO_3^+$: $[M+Na]^+$ 249.1461, Found: 249.1462. **IR** (neat) ν_{max} 3081, 2964, 2871, 1764, 1680, 1465, 1378, 1321, 1172, 1138.

5-Butyl-5-methoxy-3-phenylethynyl-5H-furan-2-one (2k)



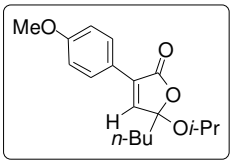
Yield: 59%; red liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.55-7.53 (m, 2H), 7.44-7.30 (m, 3H), 7.19 (s, 1H), 3.73 (hept, *J* = 6.1 Hz, 1H), 1.92-1.88 (m, 2H), 1.43-1.32 (m, 4H), 1.18 (d, *J* = 6.1 Hz, 3H), 1.13 (d, *J* = 6.1 Hz, 3H), 0.89 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.4, 152.0, 132.0, 129.5, 128.4, 121.3, 120.8, 110.1, 97.6, 78.0, 67.3, 37.9, 25.4, 23.9, 23.7, 22.5, 13.8. **HRMS** Calcd (ESI) m/z for $C_{19}H_{22}NaO_3^+$: $[M+Na]^+$ 321.1461, Found: 321.1456. **IR** (neat) ν_{max} 3084, 2961, 2867, 2220, 1774, 1631, 1460, 1380, 1324, 1174, 1140, 758, 691.

5-Butyl-5-isopropoxy-3-trimethylsilanylethynyl-5H-furan-2-one (2l)



Yield: 45%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.20 (s, 1H), 3.72 (hept, *J* = 6.2 Hz, 1H), 1.92-1.88 (m, 2H), 1.36-1.19 (m, 4H), 1.20 (d, *J* = 6.2 Hz, 3H), 1.16 (d, *J* = 6.2 Hz, 3H), 0.90 (t, *J* = 7.0Hz, 3H), 0.26 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.2, 153.3, 120.8, 109.9, 104.9, 92.6, 67.3, 37.8, 25.3, 23.9, 23.7, 22.5, 13.8, -0.5. **HRMS** Calcd (ESI) m/z for $C_{16}H_{26}NaO_3Si^+$: $[M+Na]^+$ 317.1543, Found: 317.1536. **IR** (neat) ν_{max} 3087, 2964, 2870, 2164, 1782, 1621, 1461, 1381, 1321, 1168, 1138.

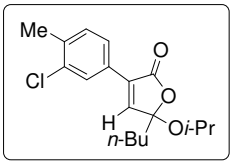
5-Butyl-5-isopropoxy-3-(4-methoxy-phenyl)-5H-furan-2-one (2m)



Yield: 84%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.9 Hz, 2H), 6.95 (d, *J* = 8.9 Hz, 2H), 3.84 (s, 3H), 3.76 (hept, *J* = 6.2 Hz, 1H), 1.94 (m, 2H), 1.42-1.33 (m, 4H), 1.19 (d, *J* = 6.2 Hz, 3H), 1.13 (d, *J* = 6.2 Hz, 3H), 0.90 (t, *J* = 7.1

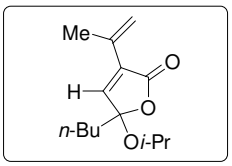
Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.6, 160.7, 142.5, 133.6, 128.8, 121.4, 114.0, 108.4, 66.6, 55.3, 38.0, 25.5, 24.0, 23.8, 22.6, 13.8. **HRMS** Calcd (ESI) m/z for $C_{18}H_{24}NaO_4^+$: $[M+Na]^+$ 327.1567, Found: 327.1557. **IR** (neat) ν_{max} 3080, 2961, 2867, 1758, 1604, 1461, 1375, 1324, 1174, 1141, 838.

5-Butyl-3-(3-chloro-4-methyl-phenyl)-5-isopropoxy-5H-furan-2-one (2n)



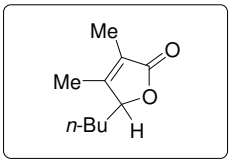
Yield: 88%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87 (s, 1H), 7.71 (s, 1H), 7.28 (s, 1H), 3.75 (hept, *J* = 6.1 Hz, 1H), 2.40 (s, 3H), 1.97 (s, 3H), 1.36-1.29 (m, 4H), 1.20 (d, *J* = 6.1 Hz, 3H), 1.13 (d, *J* = 6.1 Hz, 3H), 0.90 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.0, 145.1, 137.9, 134.8, 133.0, 131.1, 128.0, 127.7, 125.5, 108.5, 66.9, 37.9, 25.5, 24.0, 23.8, 22.6, 20.0, 13.9. **HRMS** Calcd (ESI) m/z for $C_{18}H_{23}ClNaO_3^+$: $[M+Na]^+$ 345.1228, Found: 345.1220. **IR** (neat) ν_{max} 3081, 2961, 2871, 1758, 1604, 1461, 1378, 1321, 1198, 1138, 955, 822.

5-Butyl-3-isopropenyl-5-isopropoxy-5H-furan-2-one (2p)



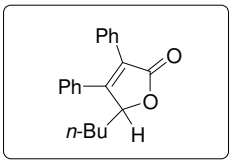
Yield: 73%; colorless liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.82 (s, 1H), 6.47-6.23 (m, 1H), 5.55-5.28 (m, 1H), 3.67 (hept, *J* = 6.2 Hz, 1H), 2.02-1.94 (m, 3H), 1.91-1.83 (m, 2H), 1.39-1.27 (m, 4H), 1.16 (d, *J* = 6.2 Hz, 3H), 1.10 (d, *J* = 6.2 Hz, 3H), 0.88 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.8, 144.7, 134.3, 132.4, 120.4, 107.7, 66.6, 37.9, 25.5, 24.0, 23.8, 22.6, 21.5, 13.9. **HRMS** Calcd (ESI) m/z for $C_{14}H_{22}NaO_3^+$: $[M+Na]^+$ 261.1461, Found: 261.1463. **IR** (neat) ν_{max} 3080, 2961, 2870, 1764, 1604, 1457, 1378, 1317, 1171, 1140.

5-Butyl-3,4-dimethyl-5H-furan-2-one (3q)



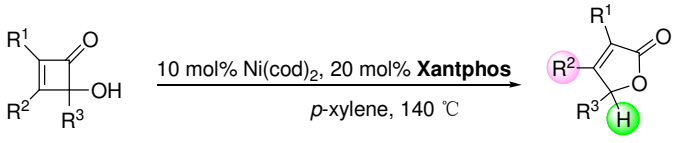
Yield: 75%; colorless liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.74-4.73 (m, 1H), 1.95 (s, 3H), 1.91-1.87 (m, 1H), 1.81 (s, 3H), 1.50-1.35 (m, 5H), 0.90 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.7, 159.3, 123.2, 83.2, 31.7, 26.4, 22.3, 13.8, 11.9, 8.3. **HRMS** Calcd (ESI) m/z for $C_{10}H_{16}NaO_2^+$: $[M+Na]^+$ 191.1043, Found: 191.1044. **IR** (neat) ν_{max} 2954, 2868, 1754, 1680, 1441, 1384, 1328, 1117, 1091.

5-Butyl-3,4-diphenyl-5H-furan-2-one (3r)



Yield: 68%; yellow solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.42-7.22 (m, 10H), 5.47-5.44 (dd, *J* = 7.0, 2.8 Hz, 1H), 1.89-1.82 (m, 1H), 1.55-1.21 (m, 5H), 0.84 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.6, 160.5, 131.2, 129.9, 129.9, 129.2, 128.9, 128.5, 128.4, 128.0, 126.5, 81.6, 32.5, 26.5, 22.2, 13.8. **HRMS** Calcd (ESI) m/z for $C_{20}H_{20}NaO_2^+$: $[M+Na]^+$ 315.1356, Found: 315.1345. **IR** (neat): 3058, 2958, 2865, 1751, 1644, 1447, 1378, 1334, 1110, 1080, 774, 694.

4. Nickel-Catalyzed Ring-Opening of α-Hydroxylcyclobutenones with Xantphos as the Ligand

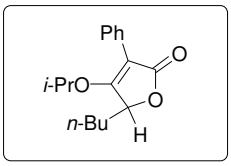


1 **3**

General procedure:

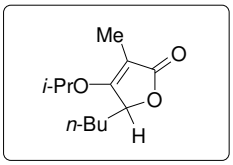
A solution of $Ni(cod)_2$ (0.05 mmol), α-hydroxylcyclotenone (**1**, 0.5 mmol), and Xantphos (0.1 mmol) in *p*-xylene (2.5 mL) was stirred at 140 °C under N_2. After the complete consumption of **1**, solvent was removed under reduced pressure. Then the crude product was further purified by silica gel column chromatography using petroleum ether/ethyl acetate as eluent. The compound 2-furanones **3** was afforded.

5-Butyl-4-isopropoxy-3-phenyl-5H-furan-2-one (3a)



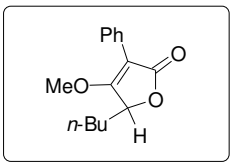
Yield: 80%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.4 Hz, 2H), 7.38 (t, *J* = 7.4 Hz, 2H), 7.32 (t, *J* = 7.4 Hz, 1H), 4.80-4.78 (dd, *J* = 7.6, 2.9 Hz, 1H), 4.60 (hept, *J* = 6.0 Hz, 1H), 2.03-1.95 (m, 1H), 1.70-1.61 (m, 1H), 1.54-1.44 (m, 2H), 1.43-1.34 (m, 2H), 1.22 (d, *J* = 6.0 Hz, 3H), 1.18(d, *J* = 6.0 Hz, 3H), 0.99-0.84 (m, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.2, 173.1, 130.2, 129.4, 128.2, 127.9, 104.1, 77.5, 74.5, 32.2, 26.5, 22.4, 22.3, 22.1, 13.9. **HRMS** Calcd (ESI) m/z for $C_{17}H_{22}NaO_3^+$: $[M+Na]^+$ 297.1461, Found: 297.1467. **IR** (neat) ν_{max} 2958, 2868, 1751, 1651, 1458, 1384, 1318, 1174, 1098, 755, 698.

5-Butyl-4-isopropoxy-3-methyl-5H-furan-2-one (3b)



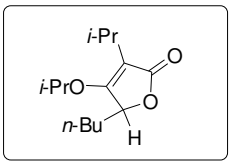
Yield: 60%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.85(hept, *J* = 6.1 Hz, 1H), 4.71 (dd, *J* = 12.1, 6.0 Hz, 1H), 4.58 (dd, *J* = 7.6, 3.4 Hz, 1H), 1.91 (s, 3H), 1.53-1.45 (m, 2H), 1.41-1.30 (m, 10H), 0.90 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.9, 171.6, 103.5, 77.6, 73.5, 31.8, 26.3, 22.5, 22.3, 16.7, 14.0, 13.8. **HRMS** Calcd (ESI) m/z for $C_{12}H_{20}NaO_3^+$: $[M+Na]^+$ 235.1305, Found: 235.1312. **IR** (neat) ν_{max} 2957, 2870, 1754, 1661, 1460, 1387, 1304, 1214, 1084.

5-Butyl-4-methoxy-3-phenyl-5H-furan-2-one (3c)



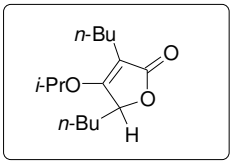
Yield: 84%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.48 (d, *J* = 7.0 Hz, 2H), 7.38 (t, *J* = 7.0 Hz, 2H), 7.35-7.30 (t, *J* = 7.0 Hz, 1H). 4.84 (dd, *J* = 7.6, 3.3 Hz, 1H), 3.79 (s, 3H), 2.05-1.98 (m, 1H), 1.72-1.63 (m, 1H), 1.53-1.45 (m, 2H), 1.42-1.34 (m, 2H), 0.94 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.5, 172.8, 129.9, 128.1, 128.0, 104.1, 77.3, 60.1, 31.8, 26.3, 22.4, 13.8. HRMS Calcd (ESI) m/z for $C_{15}H_{18}NaO_3^+$: $[M+Na]^+$ 269.1148, Found: 269.1145. **IR** (neat) ν_{max} 2958, 2864, 1741, 1654, 1458, 1368, 1328, 1174, 1041, 751, 698.

5-Butyl-4-isopropoxy-3-isopropyl-5H-furan-2-one (3d)



Yield: 81%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.67 (dd, *J* = 7.5, 3.1 Hz, 1H), 4.49 (hept, *J* = 6.1 Hz, 1H), 2.77 (hept, *J* = 5.1 Hz, 1H), 1.90-1.83 (m, 1H), 1.54-1.39 (m, 2H), 1.37 (d, *J* = 6.1 Hz, 2H), 1.33-1.31 (m, 2H), 1.29 (d, *J* = 6.1 Hz, 3H), 1.21 (d, *J* = 5.1 Hz, 3H), 1.19 (d, *J* = 5.1 Hz, 3H), 0.89 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.4, 171.9, 111.1, 76.4, 74.0, 32.3, 26.3, 23.8, 22.6, 22.6, 22.34 20.4, 20.3, 13.8. HRMS Calcd (ESI) m/z for $C_{14}H_{24}NaO_3^+$: $[M+Na]^+$ 263.1618, Found: 263.1617. **IR** (neat) ν_{max} 2963, 2874, 1747, 1661, 1461, 1381, 1301,1101, 1024.

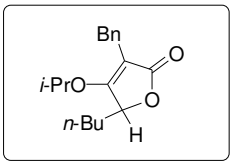
3,5-Dibutyl-4-isopropoxy-5H-furan-2-one (3e)



Yield: 80%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.68 (hept, *J* = 6.1 Hz, 1H),

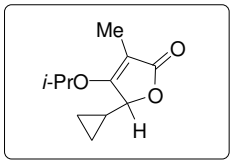
4.57 (dd, *J* = 7.5, 3.2 Hz, 1H), 2.26-2.22 (m, 2H), 1.90-1.81 (m, 1H), 1.53-1.37 (m, 5H), 1.37-1.28 (m, 10H), 0.89 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 175.0, 172.0, 102.6, 77.6, 73.4, 31.9, 31.5, 26.3, 23.0, 22.5, 22.5, 22.3, 13.8, 13.8. **HRMS** Calcd (ESI) m/z for $C_{15}H_{26}NaO_3^+$: $[M+Na]^+$ 277.1774, Found: 277.1772. **IR** (neat) ν_{max} 2958, 2867, 1755, 1655, 1460, 1378, 1295, 1098, 1045.

3-Benzyl-5-butyl-4-isopropoxy-5H-furan-2-one (3f)



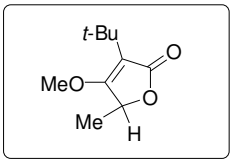
Yield: 70%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.29-7.26 (m, 2H), 7.19-7.17 (m, 3H), 4.70-4.64 (m, 2H), 3.67 (s, 2H), 1.93-1.88 (m, 1H), 1.61-1.52 (m, 1H), 1.45-1.31 (m, 4H), 1.27 (d, *J* = 8.0 Hz, 3H), 1.21 (d, *J* = 8.0 Hz, 3H), 0.90 (t, *J* = 8.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 175.0, 173.5, 139.1, 128.5, 127.8, 126.3, 100.8, 77.9, 74.1, 32.0, 28.8, 26.4, 22.6, 22.5, 22.4, 13.9. **HRMS** Calcd (ESI) m/z for $C_{18}H_{24}NaO_3^+$: $[M+Na]^+$ 311.1618, Found: 311.1617. **IR** (neat) ν_{max} 3031, 2958, 2867, 1751, 1651, 1457, 1378, 1315, 1108, 1041, 749, 703.

5-Cyclopropyl-4-isopropoxy-3-methyl-5H-furan-2-one (3g)



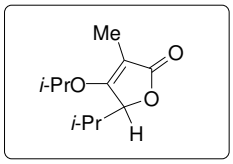
Yield: 42%; colorless liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.88 (hept, *J* = 6.1 Hz, 1H), 4.22 (d, *J* = 6.4 Hz, 1H), 1.91 (s, 3H), 1.38 (d, *J* = 6.1 Hz, 3H), 1.36 (d, *J* = 6.1 Hz, 3H), 1.04-0.99 (m, 1H), 0.64-0.61 (m, 1H), 0.56-0.48 (m, 2H), 0.41-0.36 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 175.3, 172.4, 96.4, 79.6, 73.4, 22.7, 22.6, 12.6, 8.6, 1.9, 0.8. **HRMS** Calcd (ESI) m/z for $C_{11}H_{16}NaO_3^+$: $[M+Na]^+$ 219.0992, Found: 219.0995. **IR** (neat) ν_{max} 2974, 2860, 1754, 1661, 1457, 1388, 1307, 1097, 1034.

3-tert-Butyl-4-methoxy-5-methyl-5H-furan-2-one (3h)



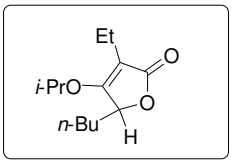
Yield: 75%; colorless liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.89 (q, *J* = 6.5 Hz, 1H), 3.83 (s, 3H), 1.50 (d, *J* = 6.5 Hz, 3H), 1.25 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.9, 172.3, 112.4, 71.0, 57.6, 31.4, 28.8, 19.1. **HRMS** Calcd (ESI) m/z for $C_{10}H_{16}NaO_3^+$: $[M+Na]^+$ 207.0992, Found: 207.1000. **IR** (neat) ν_{max} 2958, 2867, 1744, 1647, 1458, 1355, 1305, 1088, 1038.

4-Isopropoxy-5-isopropyl-3-methyl-5H-furan-2-one (3i)



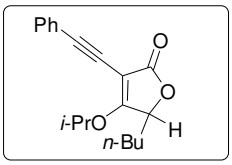
Yield: 53%; colorless liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.85 (hept, *J* = 6.1 Hz, 1H), 4.45 (d, *J* = 1.0 Hz, 1H), 2.13-2.05 (m, 1H), 1.90 (s, 3H), 1.34 (d, *J* = 6.1 Hz, 3H), 1.32 (d, *J* = 6.1 Hz, 3H), 1.06 (d, *J* = 7.0 Hz, 3H), 0.75 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 175.8, 171.2, 96.6, 81.8, 73.3, 29.6, 22.7, 22.6, 18.9, 14.2, 8.7. **HRMS** Calcd (ESI) m/z for $C_{11}H_{18}NaO_3^+$: $[M+Na]^+$ 221.1148, Found: 221.1153. **IR** (neat) ν_{max} 2971, 2877, 1754, 1662, 1461, 1388, 1308, 1138, 1111.

5-Butyl-3-ethyl-4-isopropoxy-5H-furan-2-one (3j)



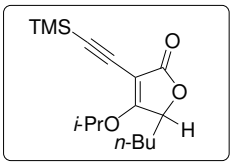
Yield: 55%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.71 (hept, *J* = 6.1 Hz, 1H), 4.58 (dd, *J* = 7.6, 3.4 Hz, 1H), 2.31 (q, *J* = 7.5 Hz, 2H), 1.92-1.83 (m, 1H), 1.56-1.47 (m, 2H), 1.43-1.31 (m, 10H), 1.11 (t, *J* = 7.5 Hz, 3H), 0.90 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.9, 171.6, 103.5, 77.6, 73.5, 31.8, 26.3, 22.5, 22.3, 16.7, 14.0, 13.8. **HRMS** Calcd (ESI) m/z for $C_{13}H_{22}NaO_3^+$: $[M+Na]^+$ 249.1461, Found: 249.1464. **IR** (neat) ν_{max} 2963, 2870, 1751, 1654, 1461, 1381, 1298, 1094, 1051.

5-Butyl-4-methoxy-3-phenylethynyl-5H-furan-2-one (3k)



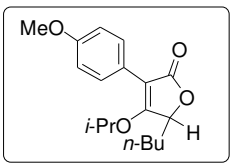
Yield: 51%; red liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.47-7.45 (m, 2H), 7.33-7.31 (m, 3H), 5.62 (hept, *J* = 2.5 Hz, 1H), 4.70 (dd, *J* = 7.6, 3.7 Hz, 1H), 1.95-1.87 (m, 1H), 1.66-1.54 (m, 2H), 1.47 (d, *J* = 2.5 Hz, 3H), 1.46 (d, *J* = 2.5 Hz, 3H), 1.43-1.34 (m, 3H), 0.91 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.3, 171.3, 131.4, 128.6, 128.3, 122.6, 93.8, 86.6, 78.7, 76.1, 31.6, 29.7, 26.1, 22.6, 22.3, 13.8. **HRMS** Calcd (ESI) m/z for $C_{19}H_{22}NaO_3^+$: $[M+Na]^+$ 321.1461, Found: 321.1455. **IR** (neat) ν_{max} 2957, 2864, 2218, 1760, 1637, 1457, 1397, 1328, 1104, 1031, 756, 687.

5-Butyl-4-isopropoxy-3-trimethylsilanylethynyl-5H-furan-2-one (3l)



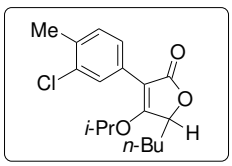
Yield: 43%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.57 (hept, *J* = 3.7 Hz, 1H), 4.64 (dd, *J* = 7.6, 3.6 Hz, 1H), 1.91-1.82 (m, 1H), 1.63-1.53 (m, 2H), 1.41 (d, *J* = 3.7 Hz, 3H), 1.40 (d, *J* = 3.7 Hz, 3H), 1.37-1.29 (m, 3H), 0.89 (t, *J* = 7.1 Hz, 3H), 0.20 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 178.2, 171.2, 100.2, 93.6, 86.8, 78.5, 76.0, 53.4, 31.5, 26.1, 22.6, 22.6, 22.3, 13.8, -0.26. **HRMS** Calcd (ESI) m/z for $C_{16}H_{26}NaO_3Si^+$: $[M+Na]^+$ 317.1543, Found: 317.1534. **IR** (neat) ν_{max} 2958, 2861, 2158, 1768, 1628, 1461, 1381, 1318, 1101, 1031.

5-Butyl-4-isopropoxy-3-(4-methoxy-phenyl)-5H-furan-2-one (3m)



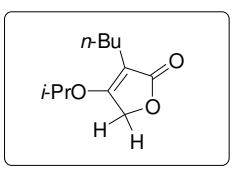
Yield: 69%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.8 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 4.77 (dd, *J* = 7.8, 3.3 Hz, 1H), 4.61 (hept, *J* = 6.1 Hz, 1H), 3.82 (s, 3H), 2.03-1.92 (m, 1H), 1.71-1.56 (m, 2H), 1.52-1.44 (m, 2H), 1.42-1.34 (m, 2H), 1.23 (d, *J* = 6.1 Hz, 3H), 1.19 (d, *J* = 6.1 Hz, 3H), 0.93 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.4, 172.6, 159.1, 130.5, 122.3, 113.7, 103.9, 77.5, 74.2, 55.2, 32.2, 26.5, 22.4, 22.3, 22.2, 13.9. **HRMS** Calcd (ESI) m/z for $C_{18}H_{24}NaO_4^+$: $[M+Na]^+$ 327.1567, Found: 327.1560. **IR** (neat) ν_{max} 2957, 2867, 1747, 1650, 1460, 1380, 1320, 1171, 1097, 1034, 834.

5-Butyl-3-(3-chloro-4-methyl-phenyl)-4-isopropoxy-5H-furan-2-one (3n)



Yield:76%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 (s, 1H), 7.39 (d, *J* = 7.9 Hz, 1H), 7.24 (d, *J* = 7.9 Hz, 1H), 4.80 (dd, *J* = 7.8, 3.3 Hz, 1H), 4.60 (hept, *J* = 6.1 Hz, 1H), 2.38 (s, 3H), 2.03-1.94 (m, 1H), 1.69-1.60 (m, 1H), 1.53-1.44 (m, 2H), 1.42-1.35 (m, 2H), 1.28 (d, *J* = 6.1 Hz, 3H), 1.22 (d, *J* = 6.1 Hz, 3H), 0.93 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.6, 172.6, 135.8, 134.2, 130.7, 129.5, 129.2, 127.3, 103.1, 77.4, 74.9, 32.2, 26.4, 22.4, 22.3, 22.2, 19.9, 13.8. **HRMS** Calcd (ESI) m/z for $C_{18}H_{23}ClNaO_3^+$: $[M+Na]^+$ 345.1228, Found: 345.1223. **IR** (neat) ν_{max} 2958, 2868, 1751, 1648, 1378, 1325, 1174, 1094, 1049, 906, 827.

3-Butyl-4-isopropoxy-5H-furan-2-one (3o)

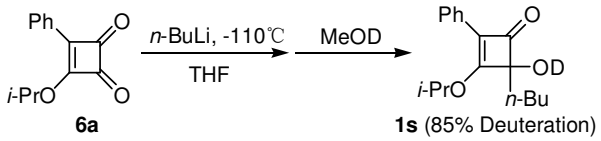


Yield: 64%; yellow liquid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.66 (s, 2H), 4.45 (hept, *J* = 6.1 Hz, 1H), 2.20 (t, *J* = 7.6 Hz, 2H), 1.47 (m, 2H), 1.37 (d, *J* = 6.1 Hz, 3H), 1.34-1.23 (m, 2H), 0.91 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 175.1, 171.1, 103.9, 73.8, 65.6, 30.1, 22.7, 22.3, 21.7, 13.7. **HRMS** Calcd (ESI) m/z for $C_{11}H_{18}NaO_3^+$: $[M+Na]^+$ 221.1148, Found: 221.1142. **IR** (neat) ν_{max} 2961, 2867, 1751, 1665, 1388,

1334, 1105, 1049, 1005.

5. Deuterated Experiments: Nickel-Catalyzed Ring-Opening of α-Hydroxylcyclobutenone 1s with PPh_3 or Xantphos as the Ligand

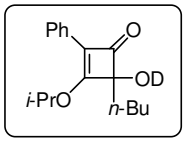
5.1 Preparation of deuterated α-Hydroxylcyclobutenone 1s:



6a **1s** (85% Deuteration)

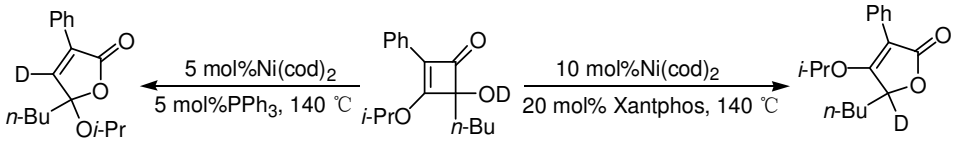
A solution of *n*-BuLi (1 mmol, 2.5M in THF) was added to the solution of cyclobutenedione **6a** (0.5 M in THF, 1.1mmol) at -110℃ under N_2. The system was stirred for 5 minutes, and then quenched with CD_3OD. The combined organic layer was removed under reduced pressure. The crude residue of **1s** (85% deuteration) was afforded.

1s (85% deuteration)



^{1}H NMR (400 MHz, CD_3COCD_3) δ 7.74-7.72 (m, 2H), 7.39-7.35 (m, 2H), 7.29-7.25 (m, 1H), 5.35 (s, 1H, 15% integration), 5.17 (hept, *J* = 6.1 Hz, 1H), 2.06-2.04 (m, 1H), 1.96-1.89 (m, 1H), 1.51 (d, *J* = 6.1 Hz, 6H), 1.40-1.35 (m, 4H), 0.89 (t, *J* = 7.1 Hz, 3H). **HRMS** Calcd (ESI) m/z for $C_{17}H_{21}DNaO_3^+$: $[M+Na]^+$ 298.1528, Found: 298.1534.

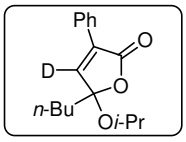
5.2 Nickel-Catalyzed Ring-Opening of 1s with PPh_3 or Xantphos as the Ligand:



2s **1s** (85% Deuteration) **3s**

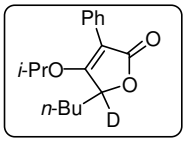
73% (44% Deuteration)	Solvent: *p*-xylene	64% (42% Deuteration)
75% (88% Deuteration)	Solvent: CD_3OD:*p*-xylene (1:10)	66% (89% Deuteration)

2s (44% deuteration, obtained from the reaction of **1s** (85% deuteration) in *p*-xylene using PPh_3 as the ligand)



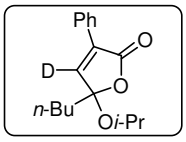
Yield: 73%; **^{1}H NMR** (400 MHz, CD_3OD) δ 7.82-7.80 (m, 2H), 7.57 (s, 1H, 56% integration), 7.42-7.27 (m, 3H), 3.64 (hept, *J* = 6.2 Hz, 1H), 1.84-1.80 (m, 2H), 1.30-1.24 (m, 4H), 1.05 (d, *J* = 6.2 Hz, 3H), 1.03 (d, *J* = 6.2 Hz, 3H), 0.80 (t, *J* = 7.1 Hz, 3H). **HRMS** Calcd (ESI) m/z for $C_{17}H_{21}DNaO_3^+$: $[M+Na]^+$ 298.1528, Found: 298.1531.

3s (42% deuteration, obtained from the reaction of **1s** (85% deuteration) in *p*-xylene using Xantphos as the ligand)



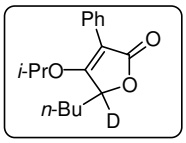
Yield: 64%; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.4 Hz, 2H), 7.38 (t, *J* = 7.4 Hz, 2H), 7.32 (t, *J* = 7.4 Hz, 1H), 4.80-4.78 (dd, *J* = 7.6, 2.9 Hz, 1H, 58% integration), 4.60 (hept, *J* = 6.0 Hz, 1H), 2.03-1.95 (m, 1H), 1.70-1.61 (m, 1H), 1.54-1.44 (m, 2H), 1.43-1.34 (m, 2H), 1.22 (d, *J* = 6.0 Hz, 3H), 1.18(d, *J* = 6.0 Hz, 3H), 0.99-0.84 (m, *J* = 7.1 Hz, 3H). **HRMS** Calcd (ESI) m/z for $C_{17}H_{21}DNaO_3^+$: $[M+Na]^+$ 298.1528, Found: 298.1533.

2s (88% deuteration, obtained from the reaction of **1s** (85% deuteration) in CD_3OD:*p*-xylene=1:10 using PPh_3 as the ligand)



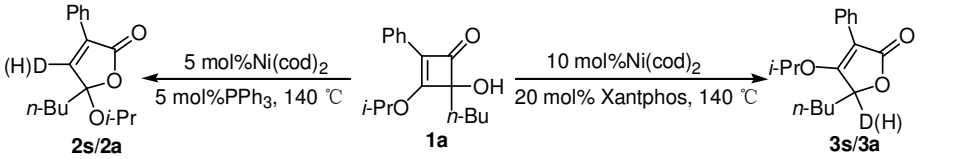
Yield: 75%; **^{1}H NMR** (400 MHz, CD_3OD) δ 7.82-7.80 (m, 2H), 7.55 (s, 1H, 12% integration), 7.42-7.27 (m, 3H), 3.64 (hept, *J* = 6.2 Hz, 1H), 1.84-1.80 (m, 2H), 1.30-1.24 (m, 4H), 1.05 (d, *J* = 6.2 Hz, 3H), 1.03 (d, *J* = 6.2 Hz, 3H), 0.80 (t, *J* = 7.1 Hz, 3H). **HRMS** Calcd (ESI) m/z for $C_{17}H_{21}DNaO_3^+$: $[M+Na]^+$ 298.1528, Found: 298.1532.

3s (89% deuteration, obtained from the reaction of **1s** (85% deuteration) in CD_3OD:*p*-xylene=1:10 using Xantphos as the ligand)



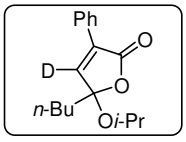
Yield: 66%; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.4 Hz, 2H), 7.38 (t, *J* = 7.4 Hz, 2H), 7.32 (t, *J* = 7.4 Hz, 1H), 4.80-4.78 (dd, *J* = 7.6, 2.9 Hz, 1H, 11% integration), 4.60 (hept, *J* = 6.0 Hz, 1H), 2.03-1.95 (m, 1H), 1.70-1.61 (m, 1H), 1.54-1.44 (m, 2H), 1.43-1.34 (m, 2H), 1.22 (d, *J* = 6.0 Hz, 3H), 1.18(d, *J* = 6.0 Hz, 3H), 0.99-0.84 (m, *J* = 7.1 Hz, 3H). **HRMS** Calcd (ESI) m/z for $C_{17}H_{21}DNaO_3^+$: $[M+Na]^+$ 298.1528, Found: 298.1531.

5.3 Related Control Experiments:



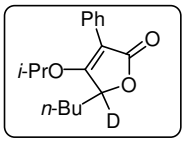
75% (**2s**, 36% Deuteration)	Solvent: CD_3OD:*p*-xylene (1:10)	67% (**3s**, 38% Deuteration)
83% (**2a**)	Solvent: *p*-xylene-d_{10}	77% (**3a**)

2s (36% deuteration, obtained from the reaction of **1a** in CD_3OD:*p*-xylene=1:10 using PPh_3 as the ligand)



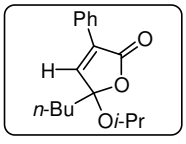
Yield: 75%; **^{1}H NMR** (400 MHz, CD_3OD) δ 7.82-7.80 (m, 2H), 7.57 (s, 1H, 64% integration), 7.31-7.30 (m, 3H), 3.64 (hept, *J* = 6.2 Hz, 1H), 1.84-1.80 (m, 2H), 1.30-1.24 (m, 4H), 1.05 (d, *J* = 6.2 Hz, 3H), 1.03 (d, *J* = 6.2 Hz, 3H), 0.80 (t, *J* = 7.1 Hz, 3H). **HRMS** Calcd (ESI) m/z for $C_{17}H_{21}DNaO_3^+$: $[M+Na]^+$ 298.1528, Found: 298.1533.

3s (38% deuteration, obtained from the reaction of **1a** in CD_3OD:*p*-xylene=1:10 using Xantphos as the ligand)



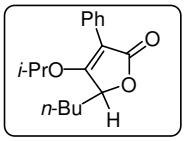
Yield: 67%; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.4 Hz, 2H), 7.38 (t, *J* = 7.4 Hz, 2H), 7.32 (t, *J* = 7.4 Hz, 1H), 4.80-4.78 (dd, *J* = 7.6, 2.9 Hz, 1H, 62% integration), 4.60 (hept, *J* = 6.0 Hz, 1H), 2.03-1.95 (m, 1H), 1.70-1.61 (m, 1H), 1.54-1.44 (m, 2H), 1.43-1.34 (m, 2H), 1.22 (d, *J* = 6.0 Hz, 3H), 1.18(d, *J* = 6.0 Hz, 3H), 0.99-0.84 (m, *J* = 7.1 Hz, 3H). **HRMS** Calcd (ESI) m/z for $C_{17}H_{21}DNaO_3^+$: $[M+Na]^+$ 298.1528, Found: 298.1532.

2a (obtained from the reaction of **1a** in *p*-xylene-d_{10} using PPh_3 as the ligand)



Yield: 83%; **^{1}H NMR** (400 MHz, CD_3OD) δ 7.82-7.80 (m, 2H), 7.57 (s, 1H), 7.31-7.30 (m, 3H), 3.64 (hept, *J* = 6.2 Hz, 1H), 1.84-1.80 (m, 2H), 1.30-1.24 (m, 4H), 1.05 (d, *J* = 6.2 Hz, 3H), 1.03 (d, *J* = 6.2 Hz, 3H), 0.80 (t, *J* = 7.1 Hz, 3H). **HRMS** Calcd (ESI) m/z for $C_{17}H_{22}NaO_3^+$: $[M+Na]^+$ 297.1461, Found: 297.1467.

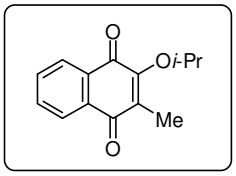
3a (obtained from the reaction of **1a** in *p*-xylene-d_{10} using Xantphos as the ligand)



Yield: 77%; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.4 Hz, 2H), 7.38 (t, *J* = 7.4 Hz, 2H), 7.32 (t, *J* = 7.4 Hz, 1H), 4.80-4.78 (dd, *J* = 7.6, 2.9 Hz, 1H), 4.60 (hept, *J* = 6.0 Hz, 1H), 2.03-1.95 (m, 1H), 1.70-1.61 (m, 1H), 1.54-1.44 (m, 2H), 1.43-1.34 (m, 2H), 1.22 (d, *J* = 6.0 Hz, 3H), 1.18(d, *J* = 6.0 Hz, 3H), 0.99-0.84 (m, *J* = 7.1 Hz, 3H). **HRMS** Calcd (ESI) m/z for $C_{17}H_{22}NaO_3^+$: $[M+Na]^+$ 297.1461, Found: 297.1467.

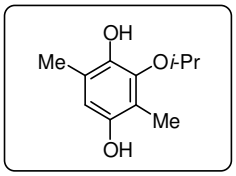
6. Nickel-Catalyzed Ring-Opening of α-Hydroxylcyclobutenones Bearing an Phenyl or Propenyl group as the R^3 Substituent with PPh_3 or Xantphos as the Ligand

2-Isopropoxy-3-methyl-[1,4]naphthoquinone (4)



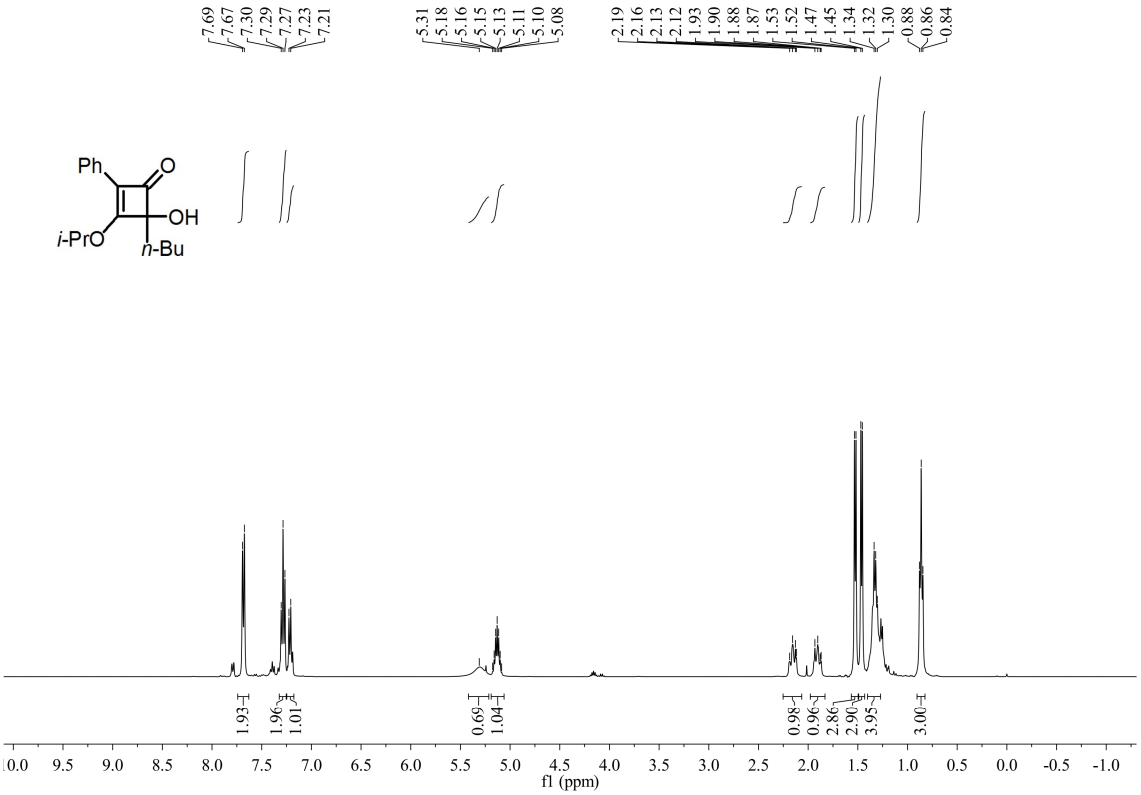
Yellow solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.10-8.05 (m, 2H), 7.72-7.70 (m, 2H), 5.02 (hept, *J* = 6.1 Hz, 1H), 2.13 (s, 2H), 1.39 (d, *J* = 6.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 185.8, 181.4, 156.7, 133.6, 133.5, 133.1, 132.3, 131.5, 126.1, 76.2, 23.0, 9.7. **HRMS** Calcd (ESI) m/z for $C_{14}H_{14}NaO_3^+$: $[M+Na]^+$ 253.0841, Found: 253.0839.

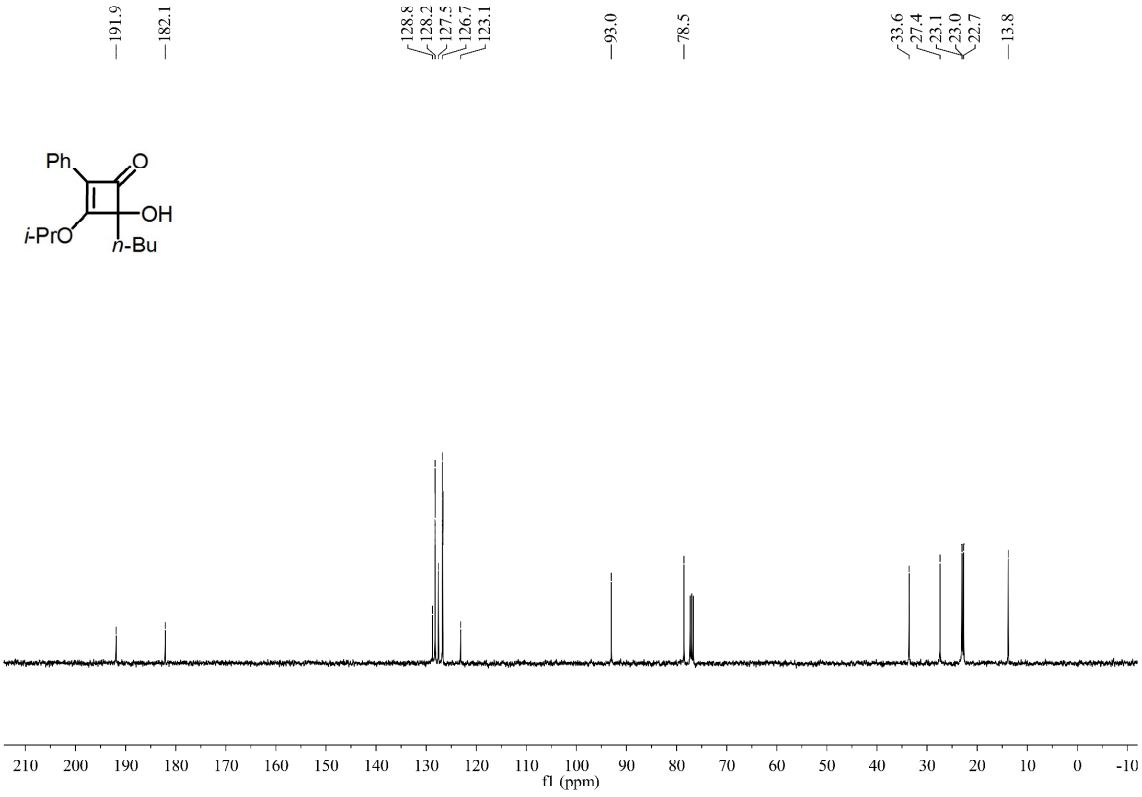
3-Isopropoxy-2,5-dimethyl-benzene-1,4-diol (5)

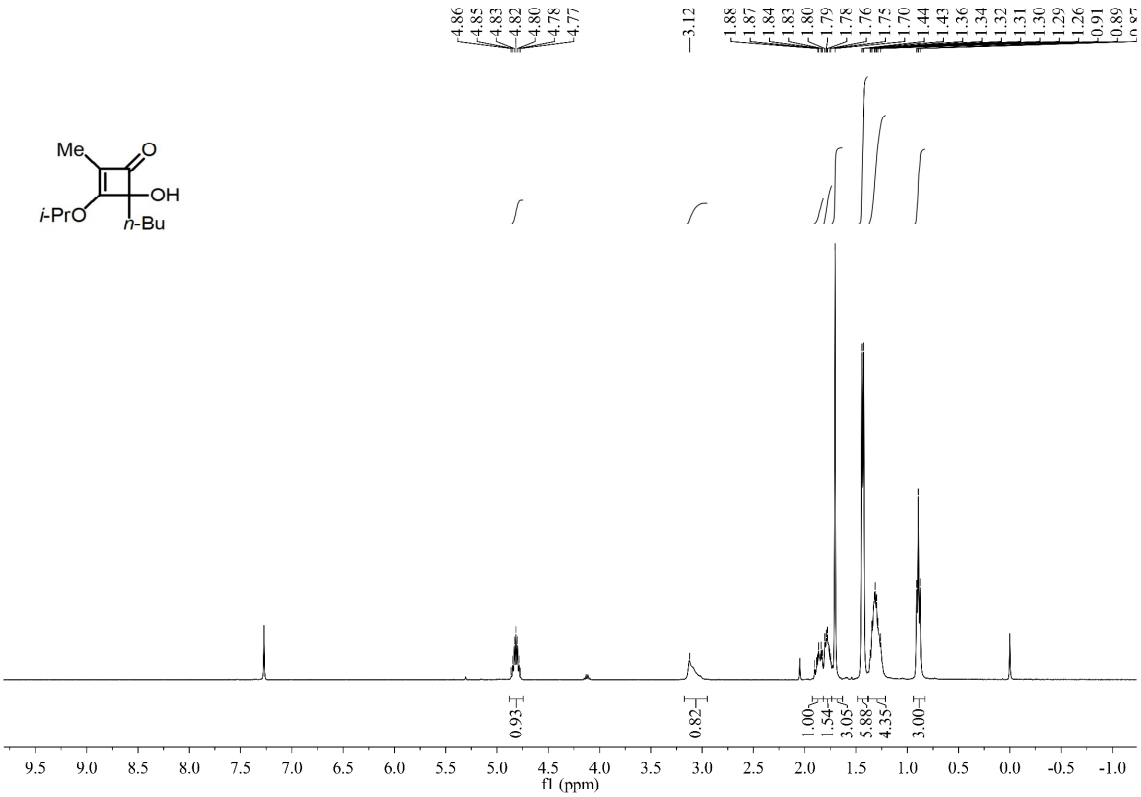


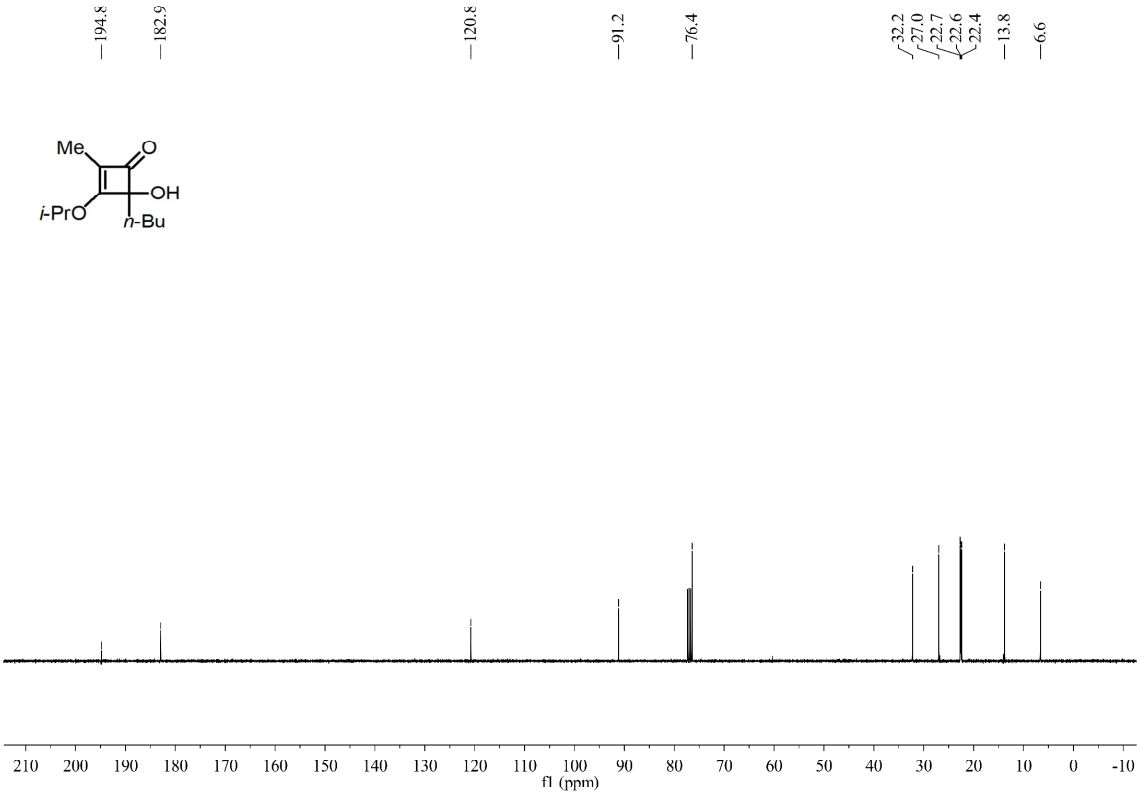
Yellow solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.35 (s, 1H), 5.38 (s, 1H), 4.72 (s, 1H), 4.16 (hept, *J* = 5.9 Hz, 1H), 2.17 (s, 3H), 2.13 (s, 3H), 1.32 (d, *J* = 5.9 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 146.8, 143.0, 141.6, 121.2, 114.9, 112.2, 76.1, 22.4, 15.5, 9.9. **HRMS** Calcd (ESI) m/z for $C_{11}H_{16}NaO_3^+$: $[M+Na]^+$ 219.0997, Found: 219.0995.

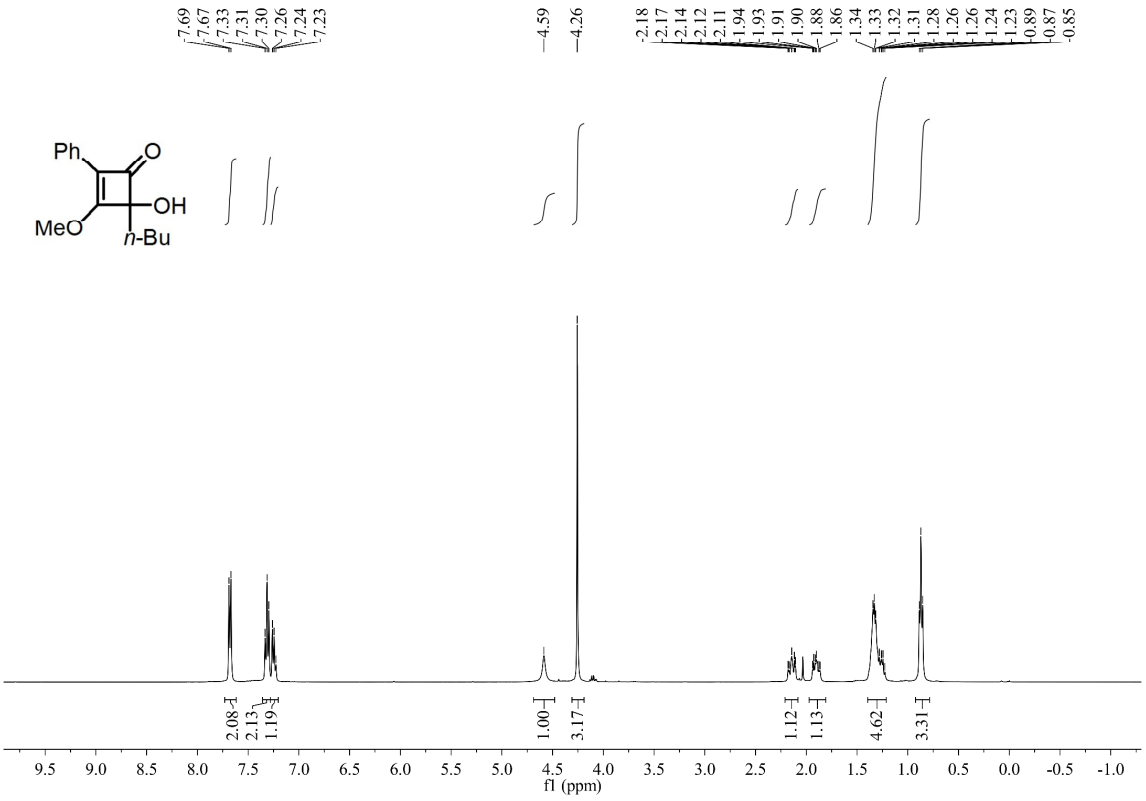
7. NMR Spectra of Compounds

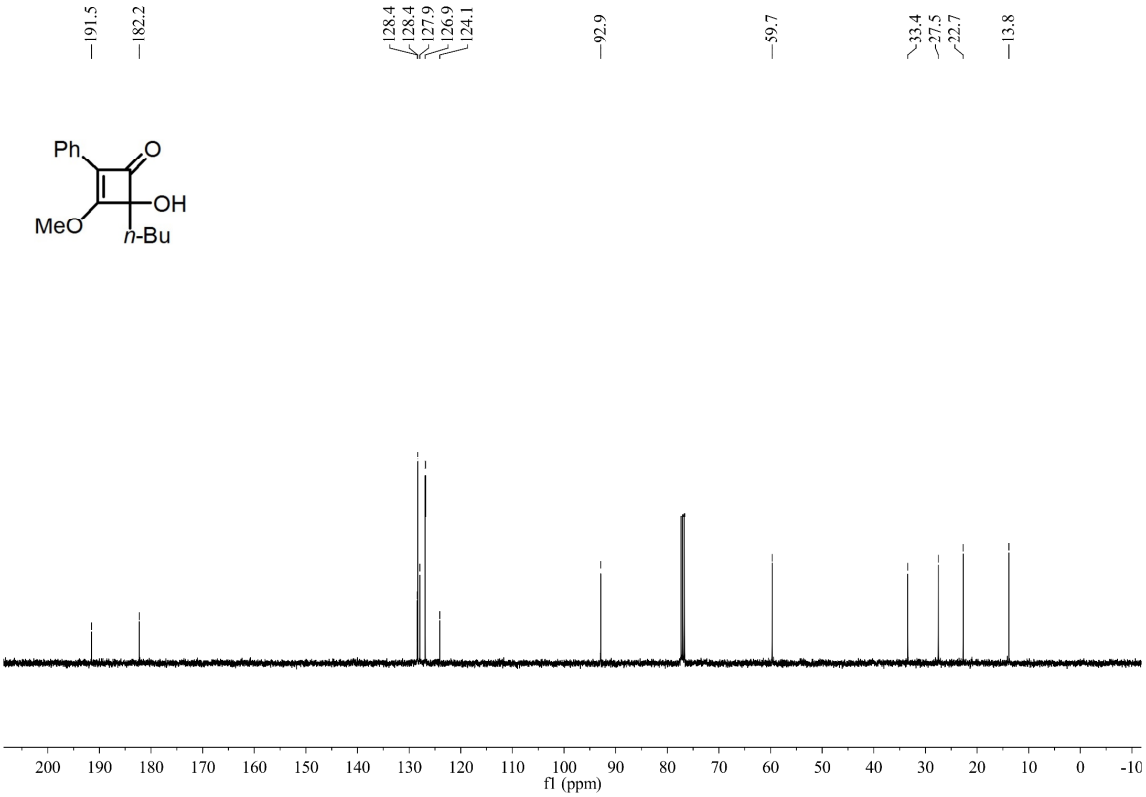


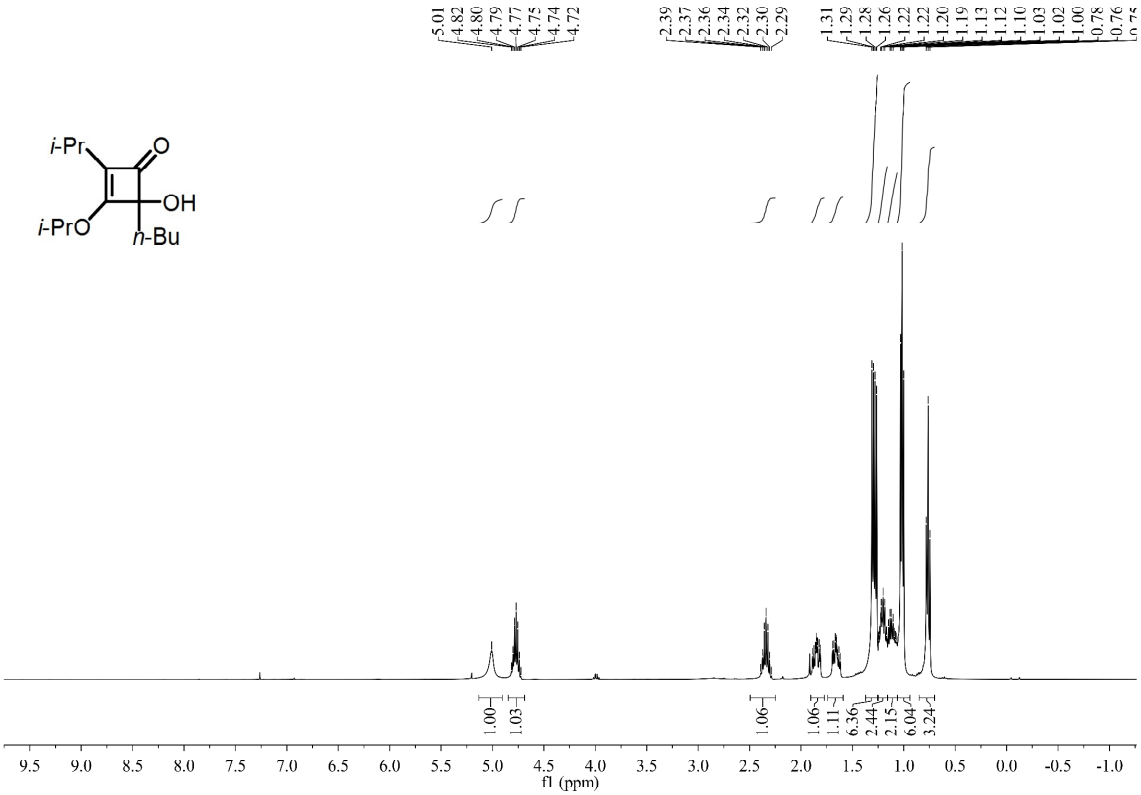


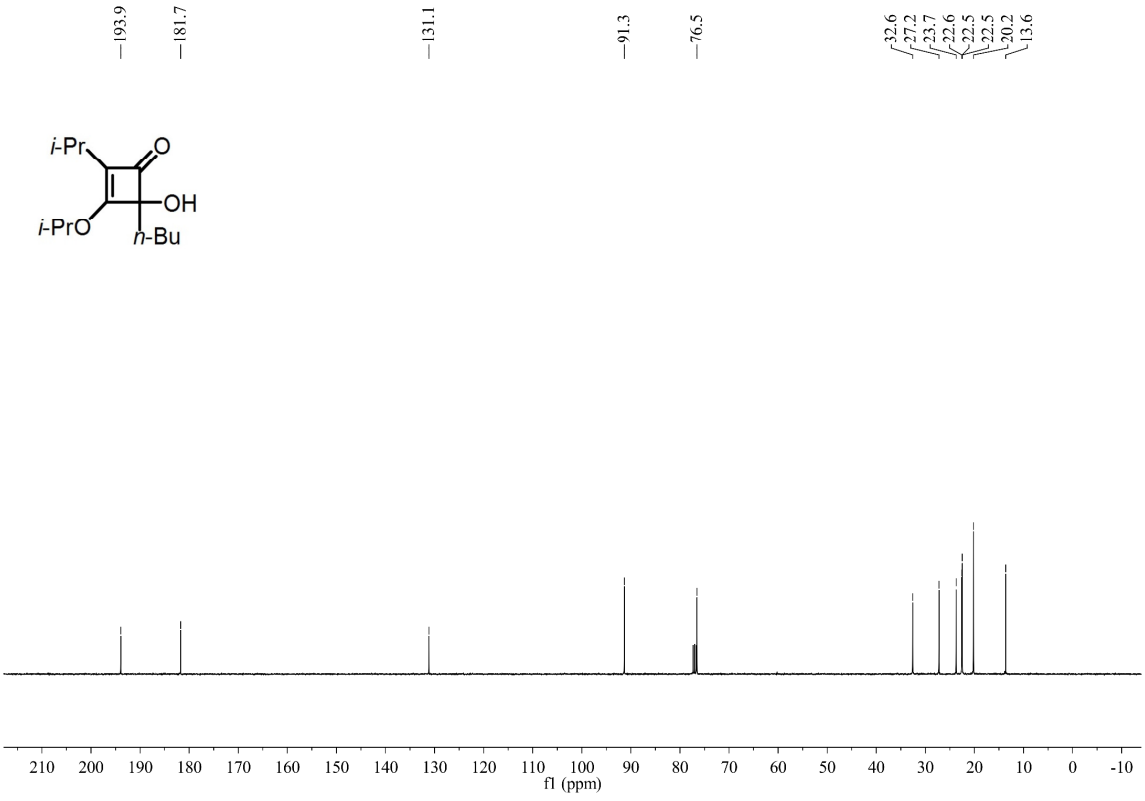

Me
O
OH
i-PrO
n-Bu
4.86
4.85
4.83
4.82
4.80
4.78
4.77
3.12
1.88
1.87
1.84
1.83
1.80
1.79
1.78
1.76
1.75
1.70
1.44
1.43
1.36
1.34
1.32
1.31
1.30
1.29
1.26
0.91
0.93
0.82
1.00
1.54
3.05
5.88
4.35
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

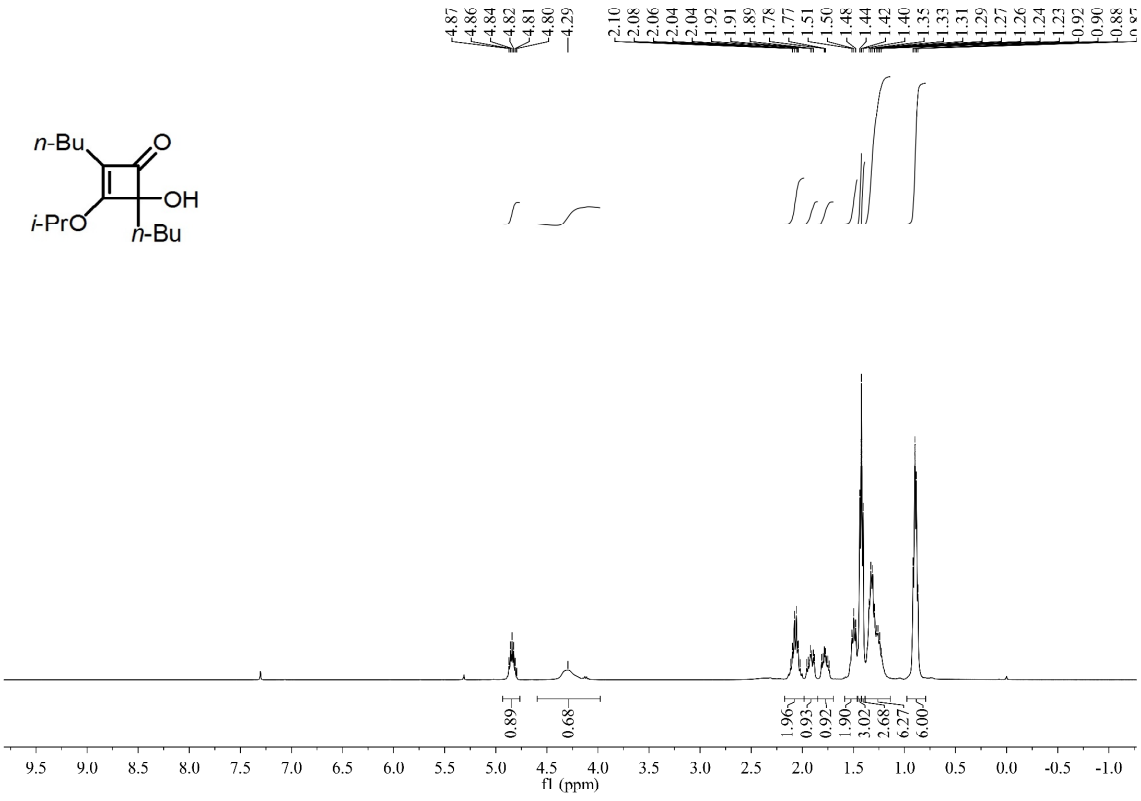

194.8
182.9
120.8
91.2
76.4
32.2
27.0
22.7
22.6
22.4
13.8
6.6
Me
O
OH
i-PrO
n-Bu
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

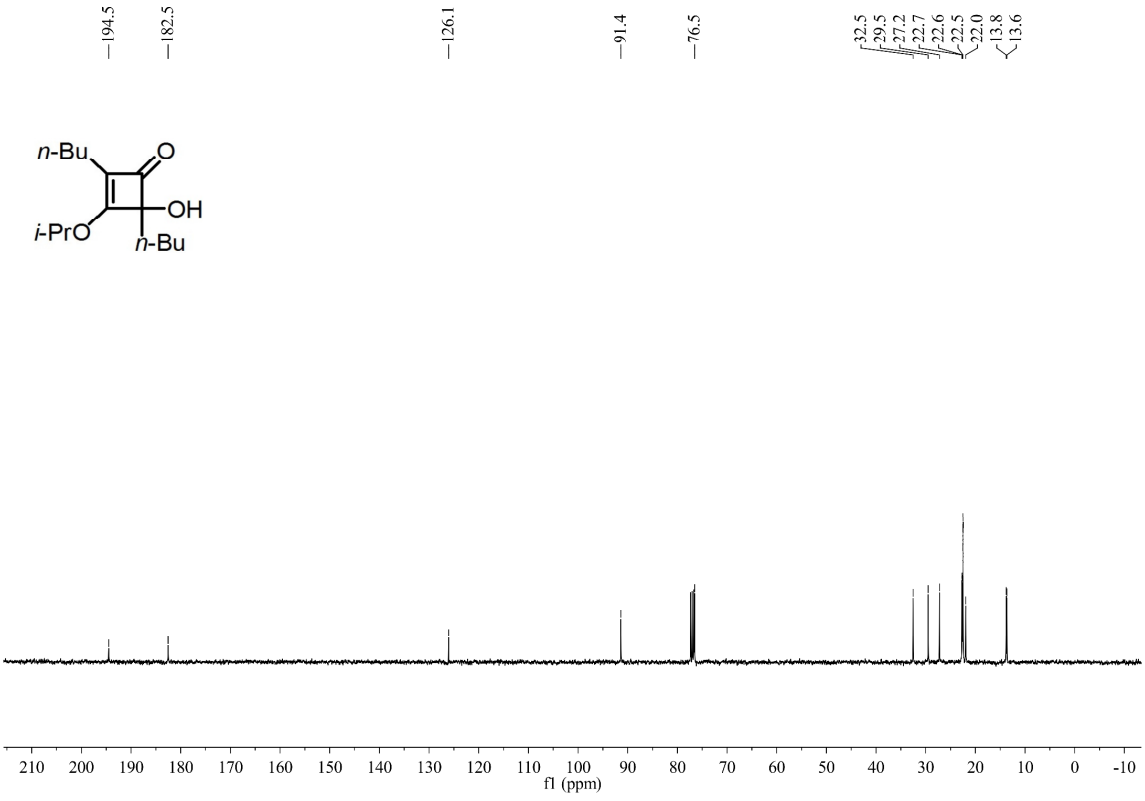

7.69
7.67
7.33
7.31
7.30
7.26
7.24
7.23
4.59
4.26
2.18
2.17
2.14
2.12
2.11
1.94
1.93
1.91
1.90
1.88
1.86
1.34
1.33
1.32
1.31
1.28
1.26
1.26
1.24
1.23
0.89
0.87
0.85
Ph
O
OH
MeO
n-Bu
2.08
2.13
1.19
1.00
3.17
1.12
1.13
4.62
3.31
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

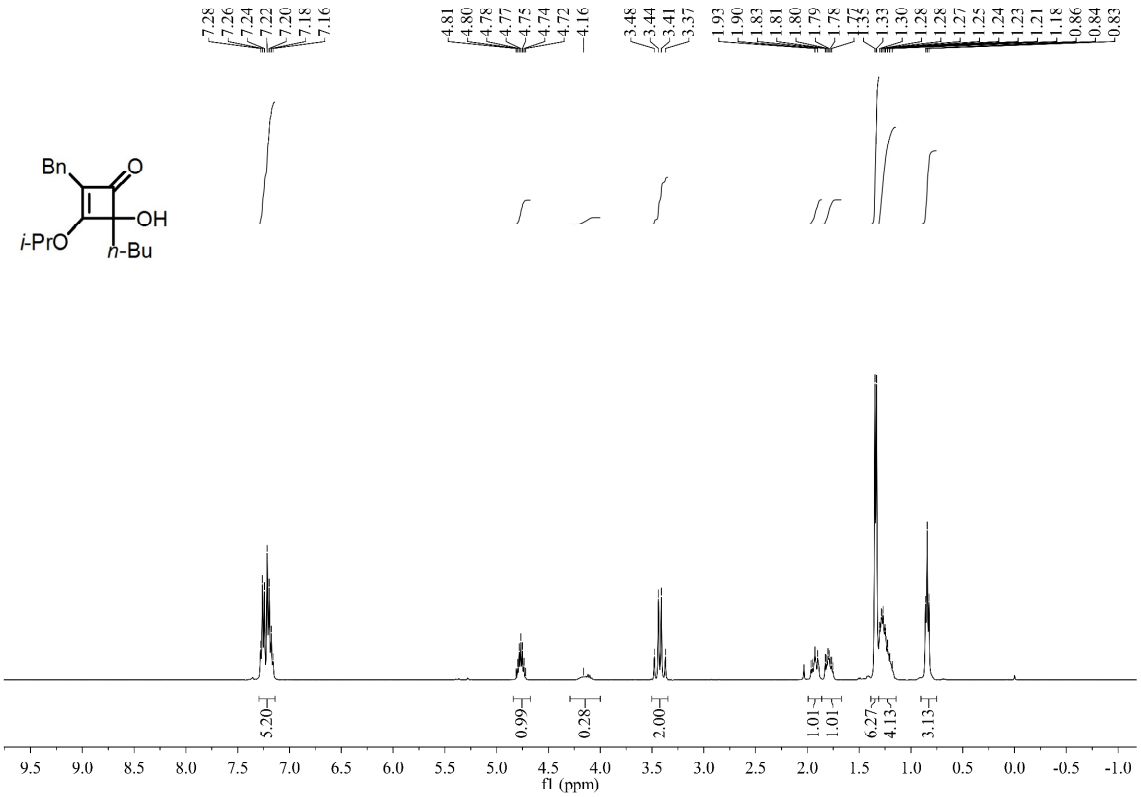

191.5
182.2
128.4
128.4
127.9
126.9
124.1
92.9
59.7
33.4
27.5
22.7
13.8
Ph
O
OH
MeO
n-Bu
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

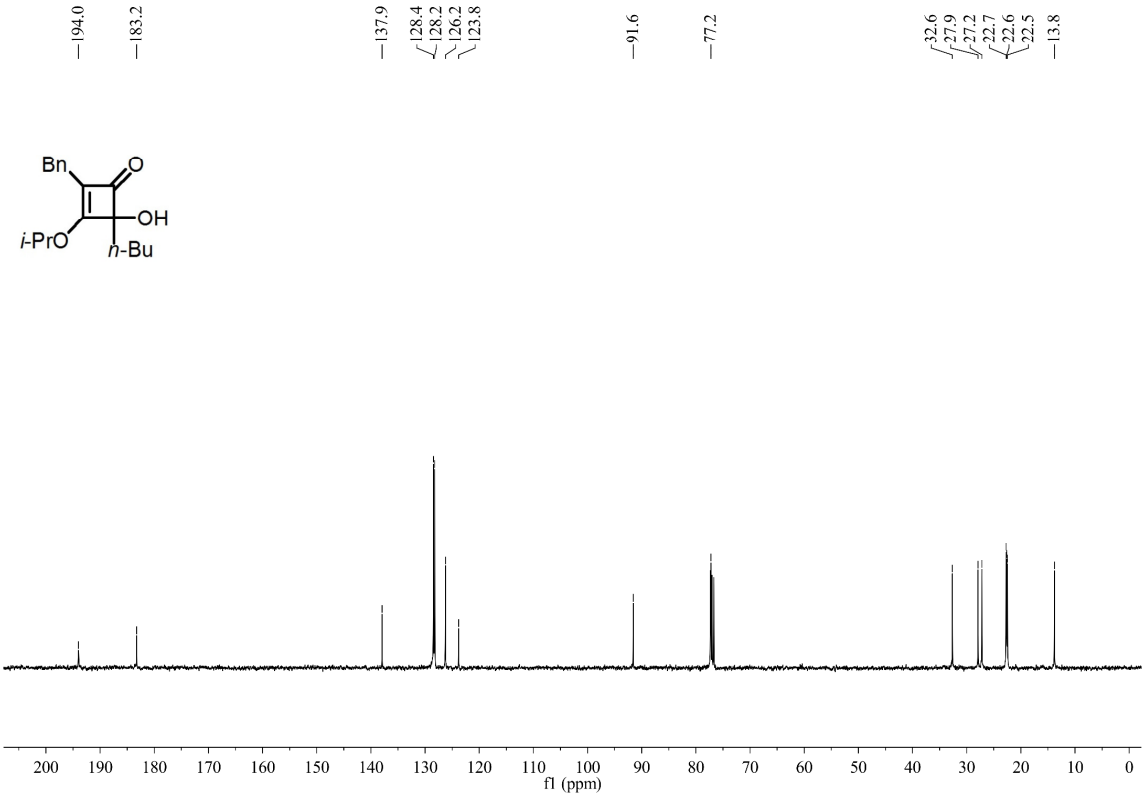

i-Pr
O
OH
i-PrO
n-Bu
5.01
4.82
4.80
4.79
4.77
4.75
4.74
4.72
2.39
2.37
2.36
2.34
2.32
2.30
2.29
1.31
1.29
1.28
1.26
1.22
1.22
1.20
1.19
1.13
1.12
1.10
1.03
1.02
1.00
0.78
0.76
1.00
1.03
1.06
1.06
1.11
6.36
2.44
2.15
6.04
3.24
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

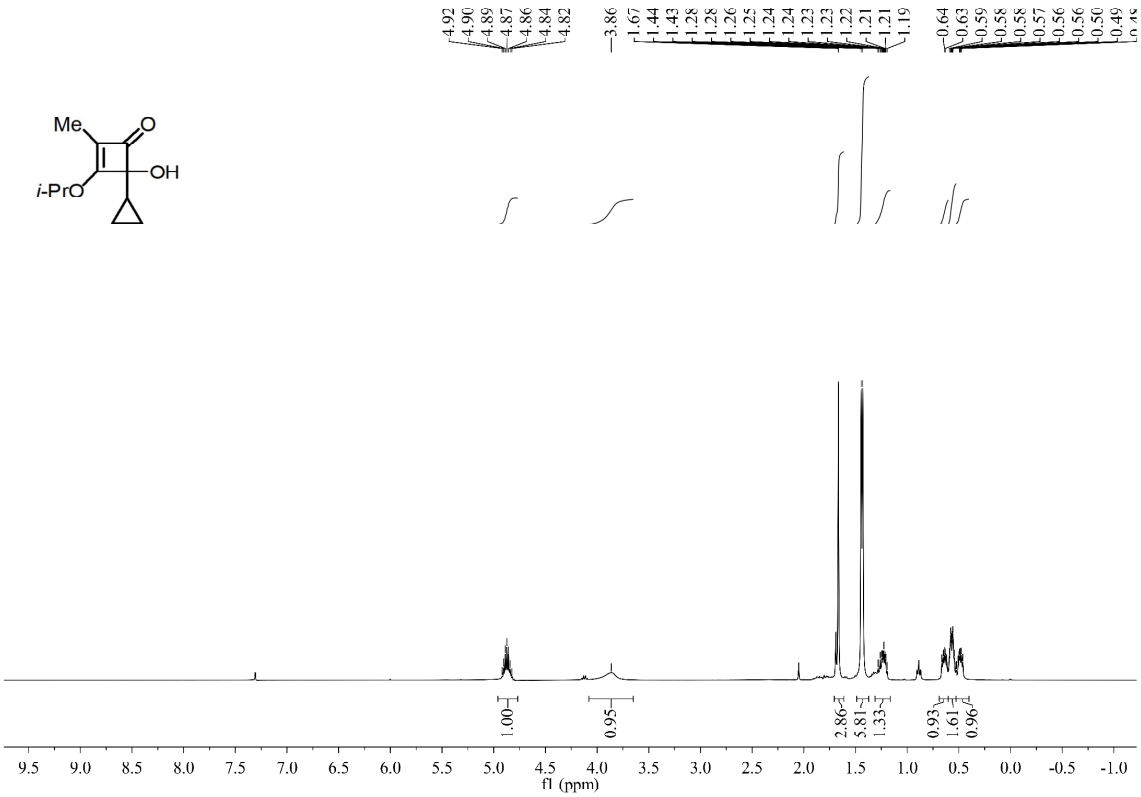

193.9
181.7
131.1
91.3
76.5
32.6
27.2
23.7
22.6
22.5
22.5
20.2
13.6
i-Pr
O
OH
i-PrO
n-Bu
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

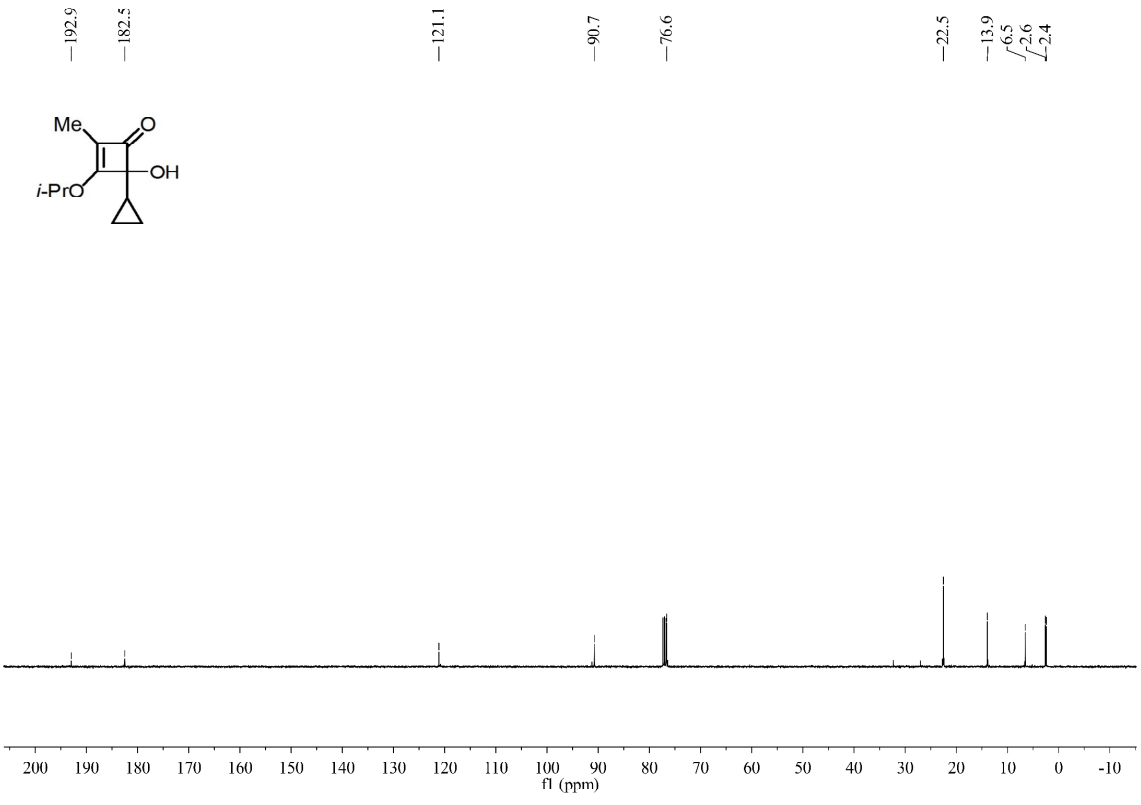

n-Bu
O
OH
i-PrO
n-Bu
4.87
4.86
4.84
4.82
4.81
4.80
4.29
2.10
2.08
2.06
2.04
2.04
1.92
1.91
1.89
1.78
1.77
1.51
1.50
1.48
1.44
1.42
1.40
1.35
1.33
1.31
1.29
1.27
1.26
1.24
1.23
0.92
0.90
0.88
0.89
0.68
1.96
0.93
0.92
1.90
3.02
2.68
6.27
6.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

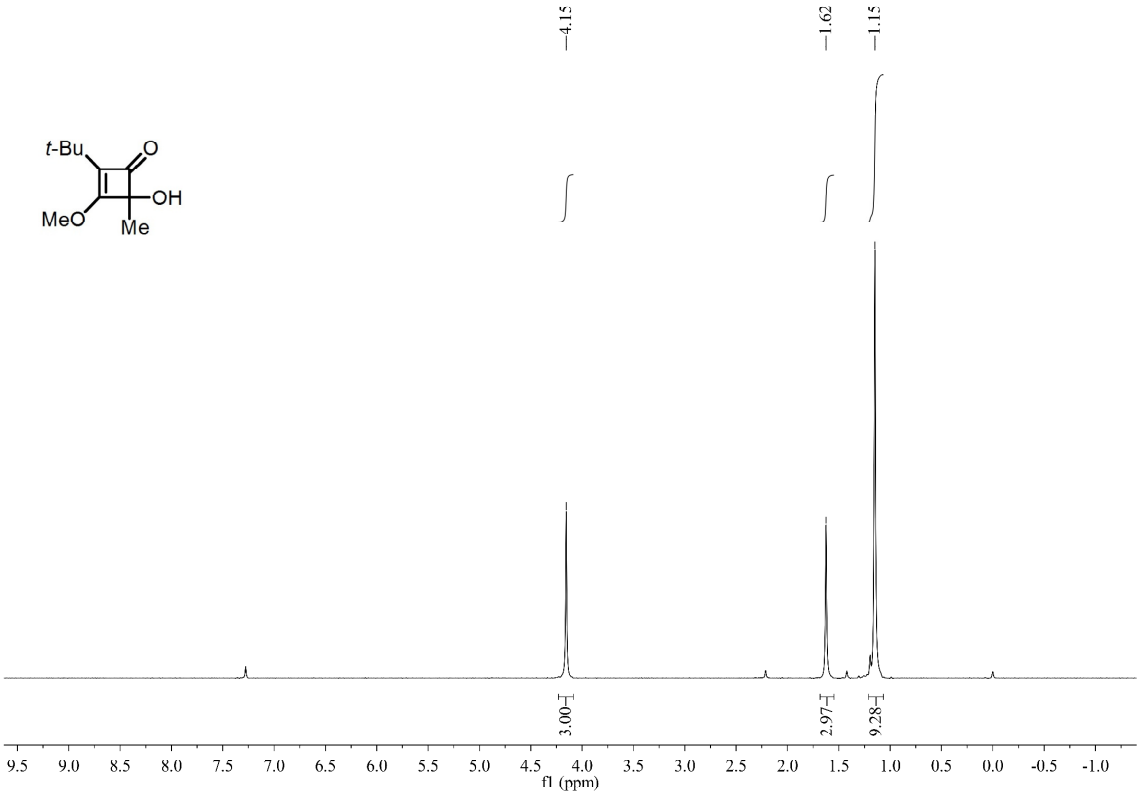

194.5
182.5
126.1
91.4
76.5
32.5
29.5
27.2
22.7
22.6
22.5
22.0
13.8
13.6
n-Bu
O
OH
i-PrO
n-Bu
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

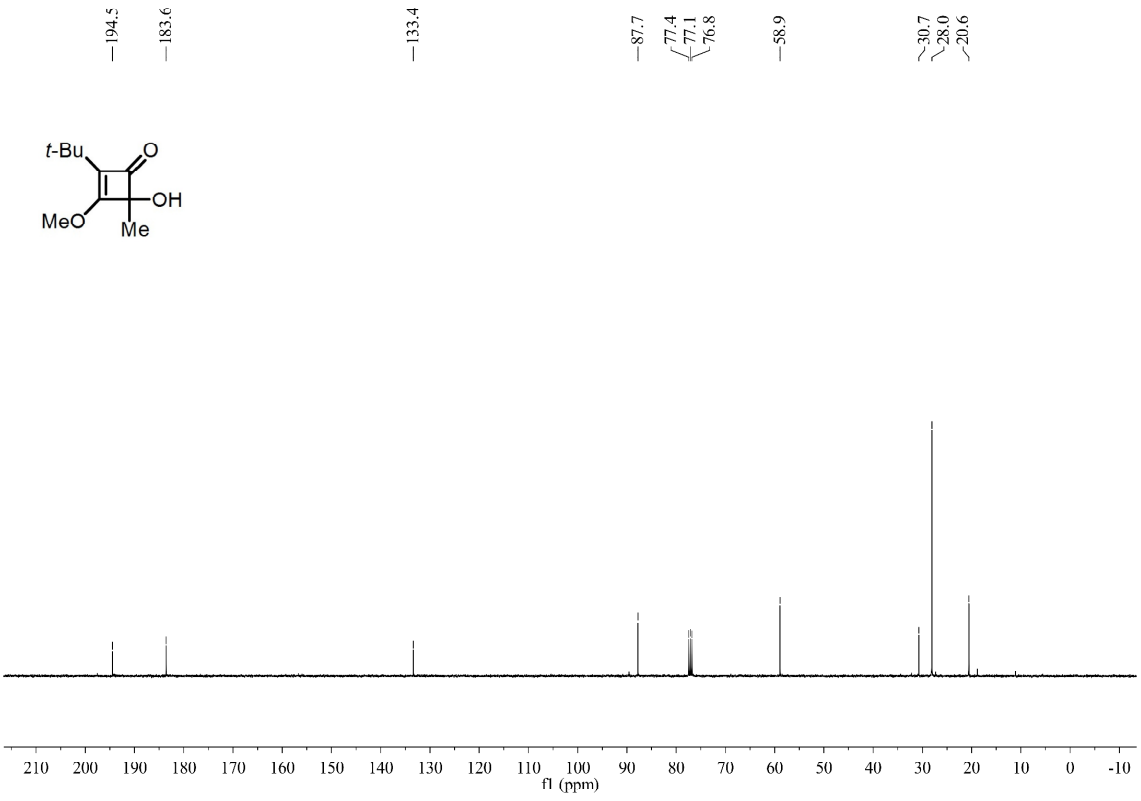

Bn
O
OH
i-PrO
n-Bu
7.28
7.26
7.24
7.22
7.20
7.18
7.16
4.81
4.80
4.78
4.77
4.75
4.74
4.72
4.16
3.48
3.44
3.41
3.37
1.93
1.90
1.83
1.81
1.80
1.79
1.78
1.77
1.33
1.30
1.28
1.28
1.27
1.25
1.24
1.23
1.21
1.18
0.86
0.84
0.83
5.20
0.99
0.28
2.00
1.01
1.01
6.27
4.13
3.13
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

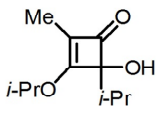

194.0
183.2
137.9
128.4
128.2
126.2
123.8
91.6
77.2
32.6
27.9
27.2
22.7
22.6
22.5
13.8
Bn
O
OH
i-PrO
n-Bu
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Me
O
OH
i-PrO
4.92
4.90
4.89
4.87
4.86
4.84
4.82
3.86
1.67
1.44
1.43
1.28
1.26
1.25
1.24
1.24
1.23
1.23
1.22
1.21
1.21
1.19
0.64
0.63
0.59
0.58
0.58
0.57
0.56
0.56
0.50
0.49
1.00
0.95
2.86
5.81
1.33
0.93
1.61
0.96
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

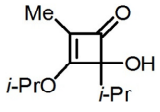

192.9
182.5
121.1
90.7
76.6
22.5
13.9
6.5
2.6
2.4
Me
O
OH
i-PrO
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


t-Bu
O
OH
MeO
Me
4.15
1.62
1.15
3.00
2.97
9.28
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

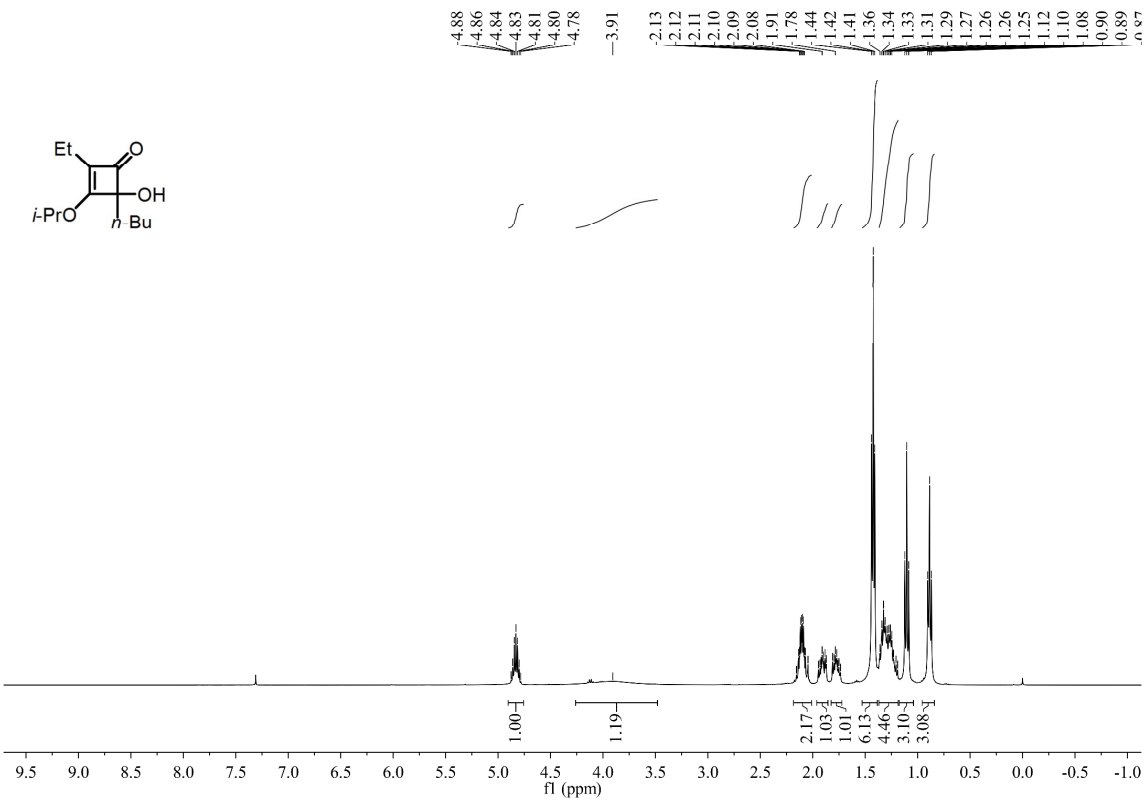

194.5
183.6
133.4
87.7
77.4
77.1
76.8
58.9
30.7
28.0
20.6
t-Bu
O
OH
MeO
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

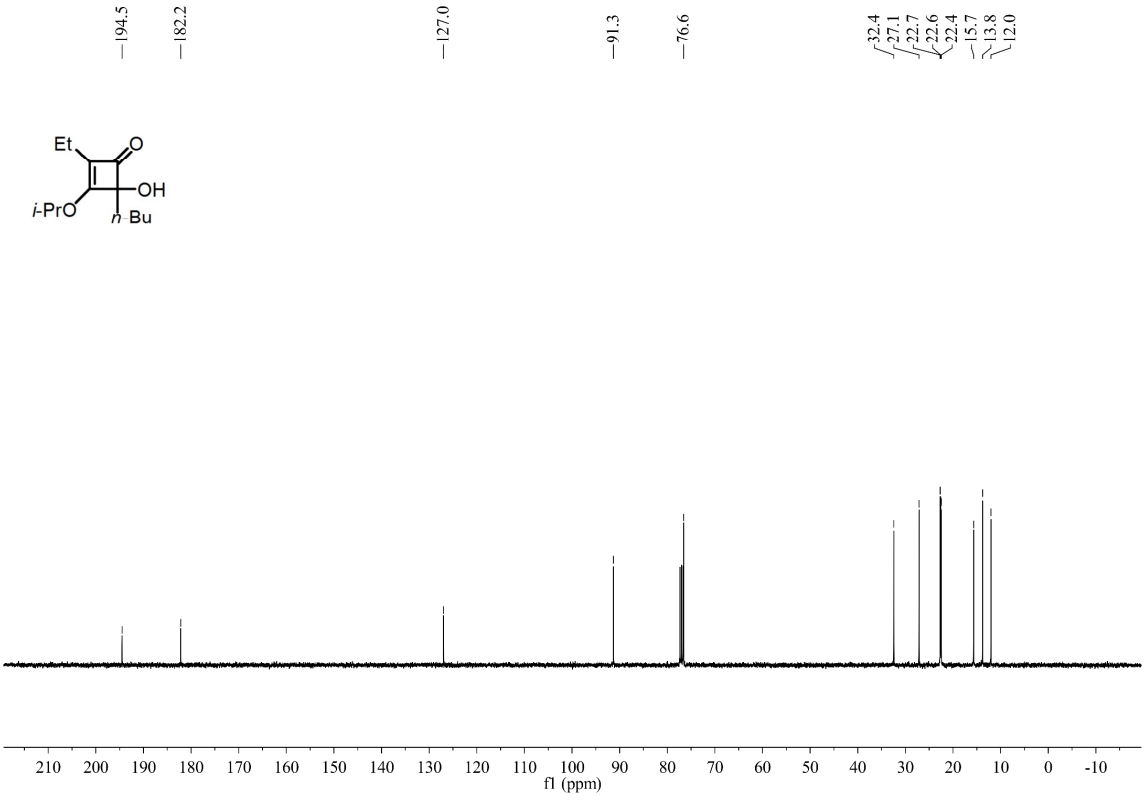

Me
O
OH
i-PrO
i-Pr

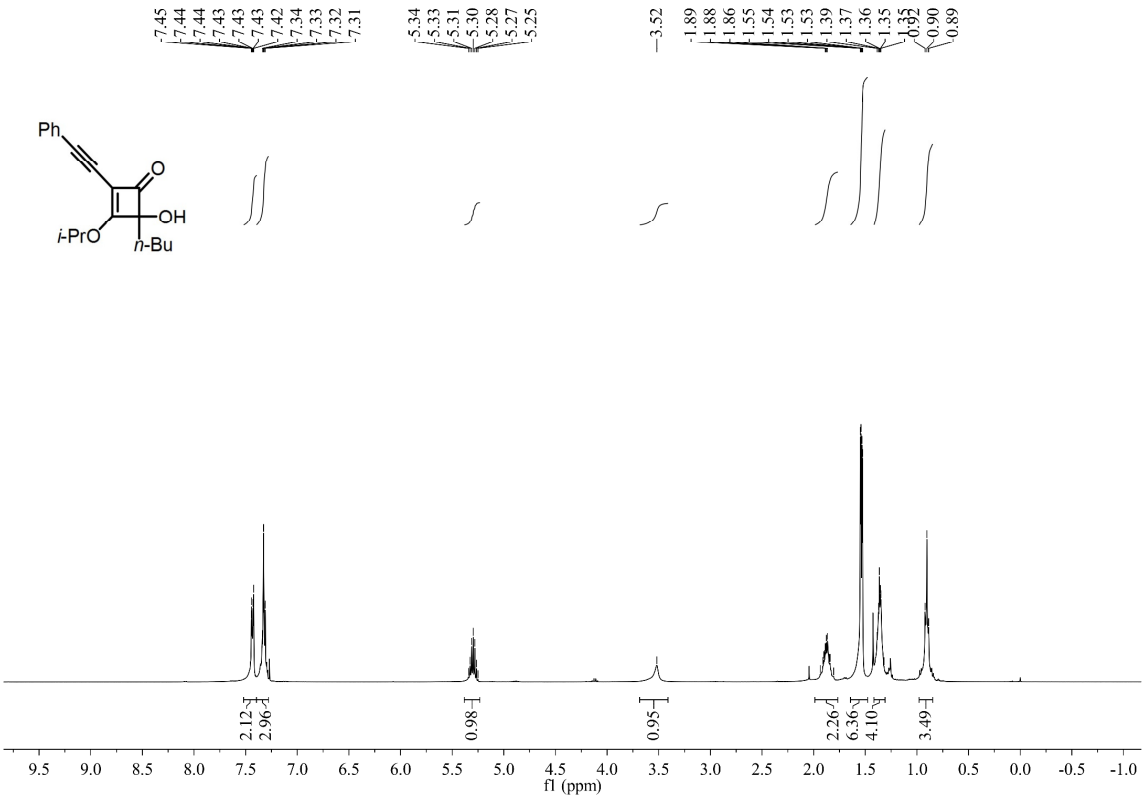
4.83
4.81
4.80
4.78
4.77
4.75
4.74
2.84
2.10
2.08
2.07
1.73
1.45
1.44
1.43
1.03
1.03
1.01
1.00
1.01
1.11
3.36
6.09
6.07
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

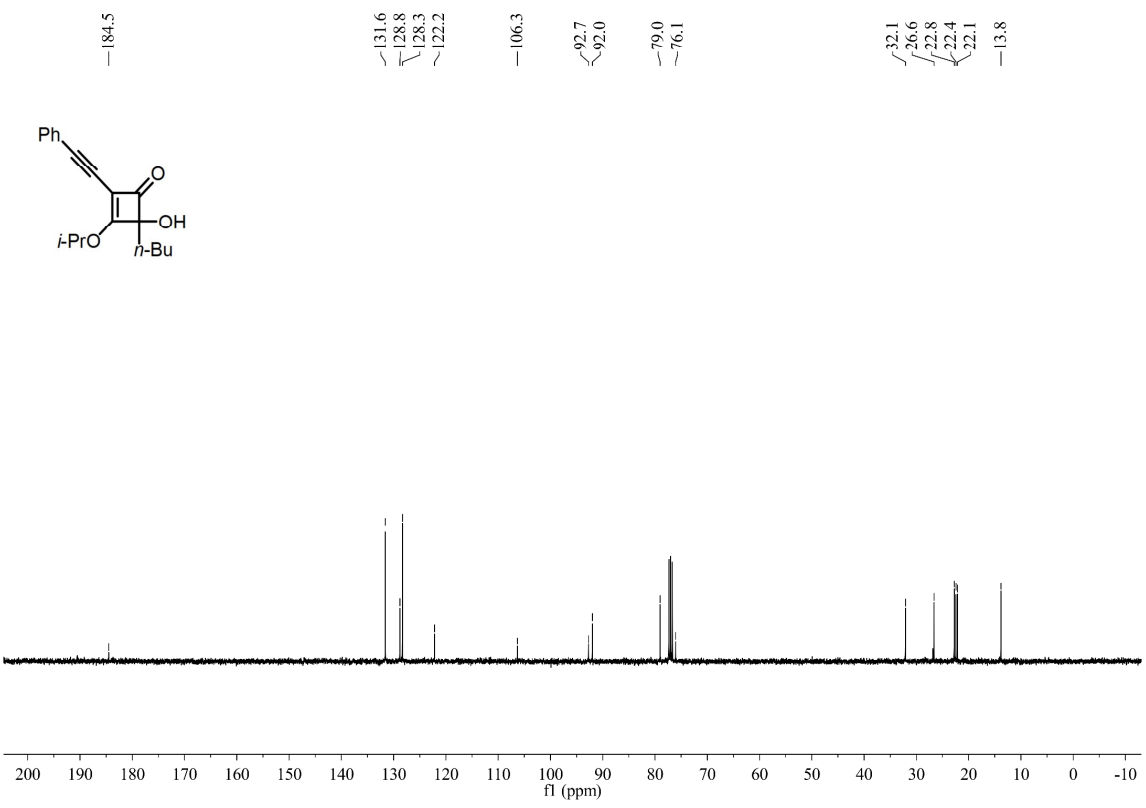

Me
O
OH
i-PrO
i-Pr

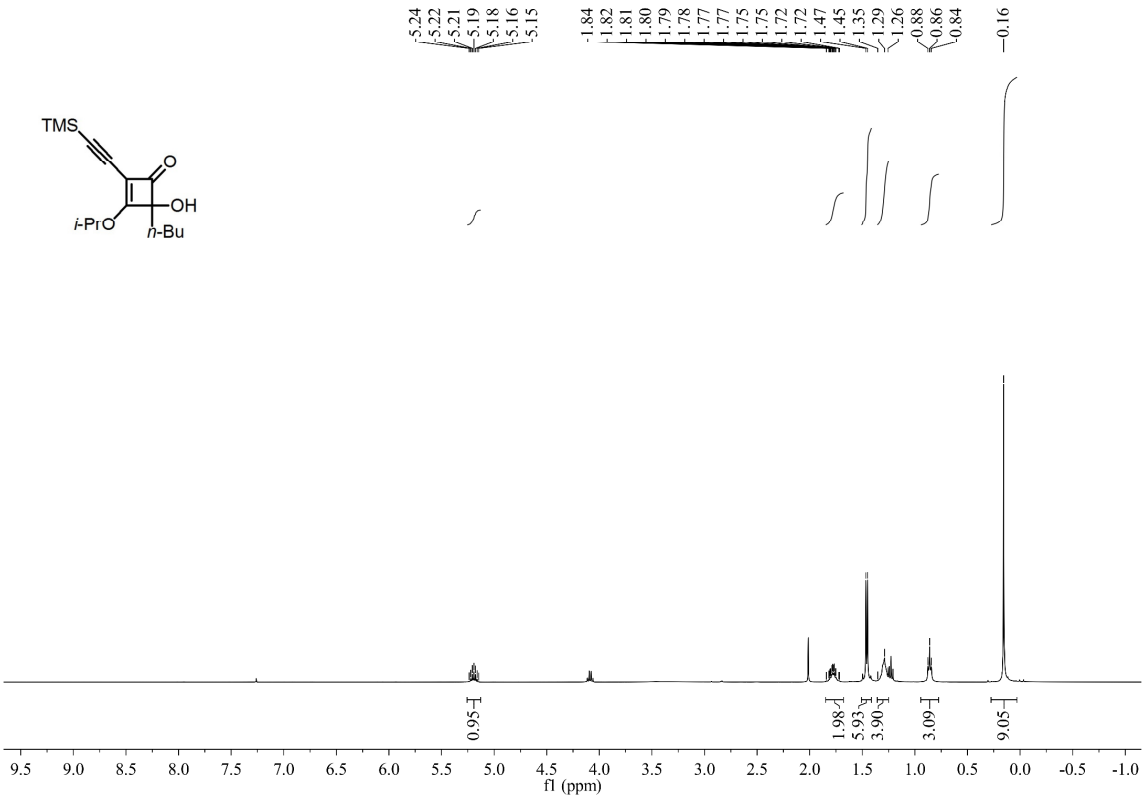
193.8
181.8
120.9
93.9
76.2
31.4
22.7
22.3
18.0
17.9
7.0
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

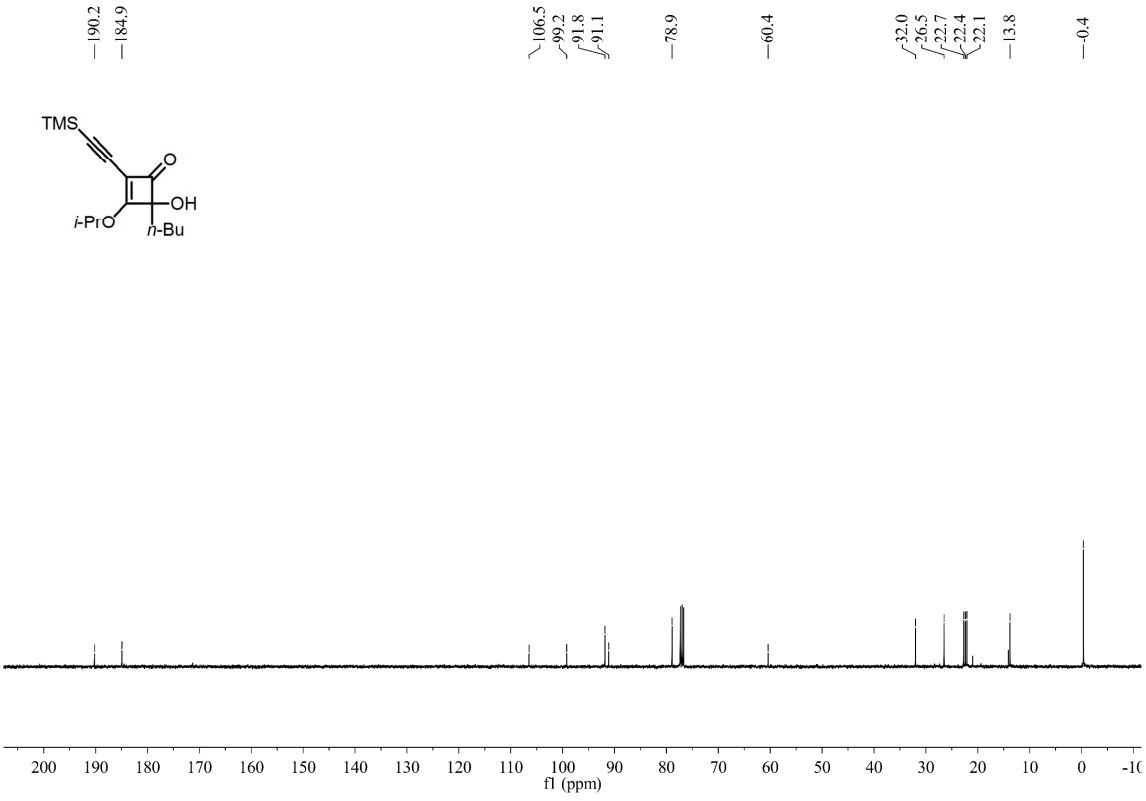

Et
O
OH
i-PrO
n Bu
4.88
4.86
4.84
4.83
4.81
4.80
4.78
3.91
2.13
2.12
2.11
2.10
2.09
2.08
1.91
1.78
1.44
1.42
1.41
1.36
1.34
1.33
1.31
1.29
1.27
1.26
1.26
1.25
1.12
1.10
1.08
0.90
0.89
1.00
1.19
2.17
1.03
1.01
6.13
4.46
3.10
3.08
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

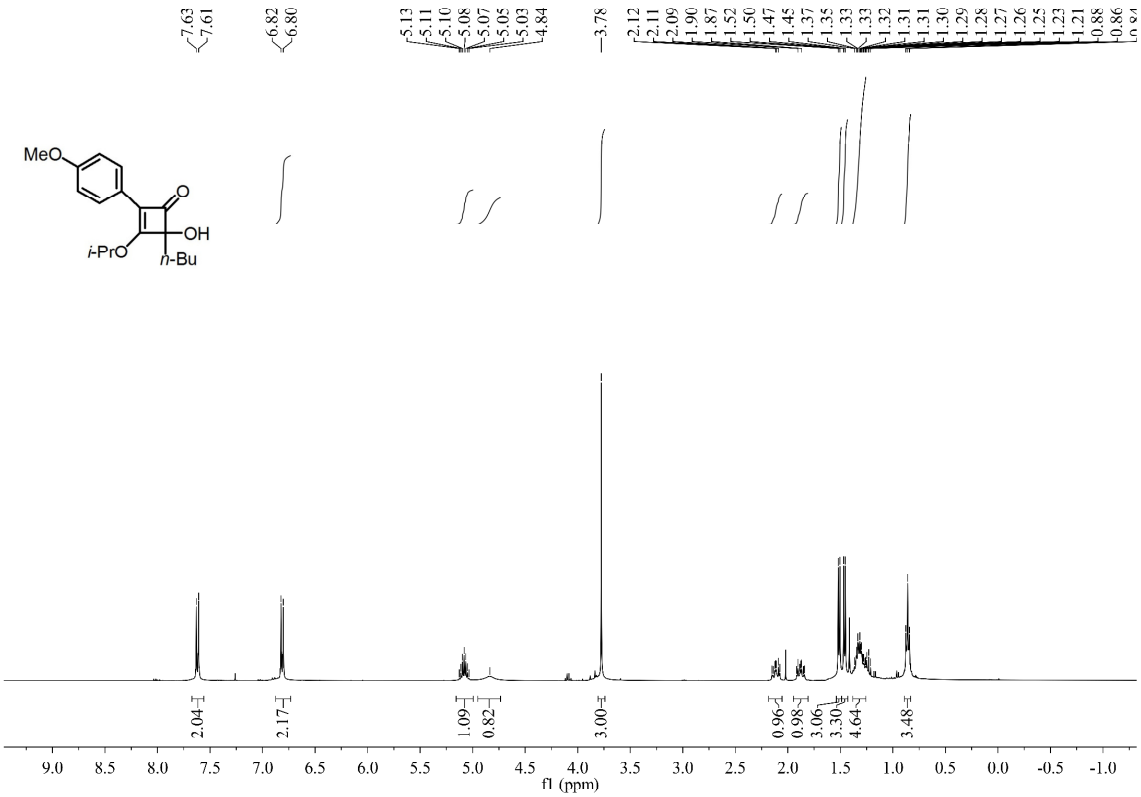

194.5
182.2
127.0
91.3
76.6
32.4
27.1
22.7
22.6
22.4
15.7
13.8
12.0
Et
O
OH
i-PrO
n Bu
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

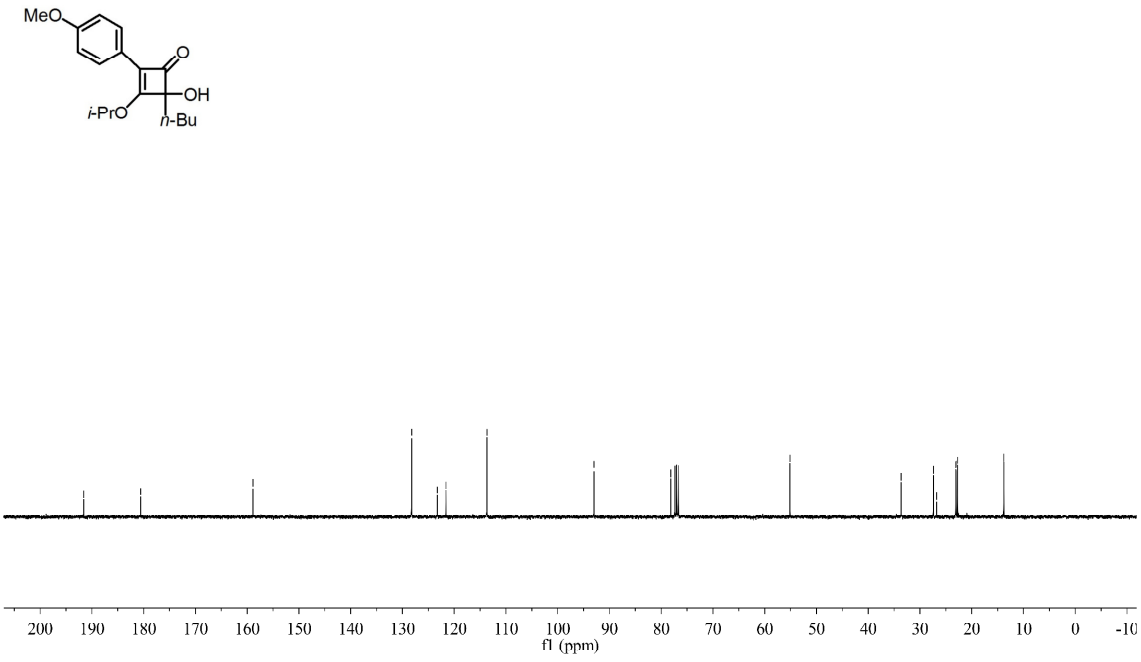

7.45
7.44
7.44
7.43
7.43
7.43
7.42
7.34
7.33
7.32
7.31
5.34
5.33
5.31
5.30
5.28
5.27
5.25
3.52
1.89
1.88
1.86
1.55
1.54
1.53
1.53
1.39
1.37
1.36
1.35
0.92
0.90
0.89
Ph
O
OH
i-PrO
n-Bu
2.12
2.96
0.98
0.95
2.26
6.36
4.10
3.49
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

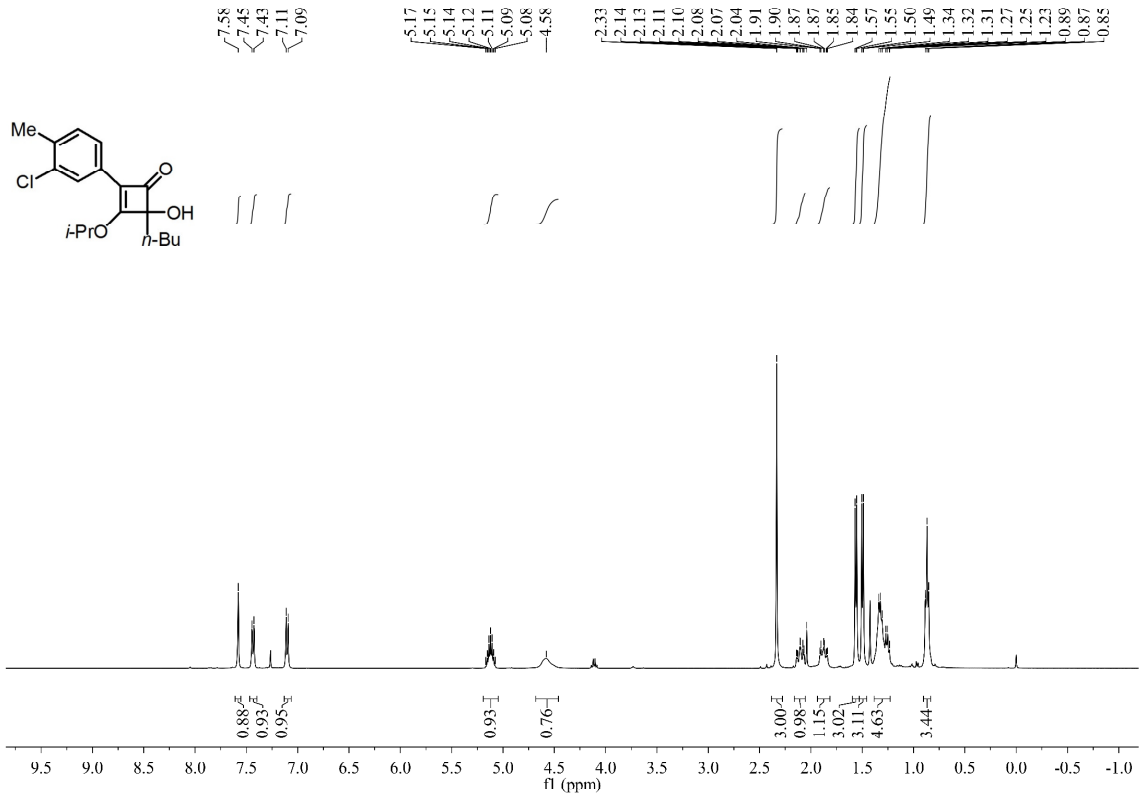

184.5
131.6
128.8
128.3
122.2
106.3
92.7
92.0
79.0
76.1
32.1
26.6
22.8
22.4
22.1
13.8
Ph
O
OH
i-PrO
n-Bu
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

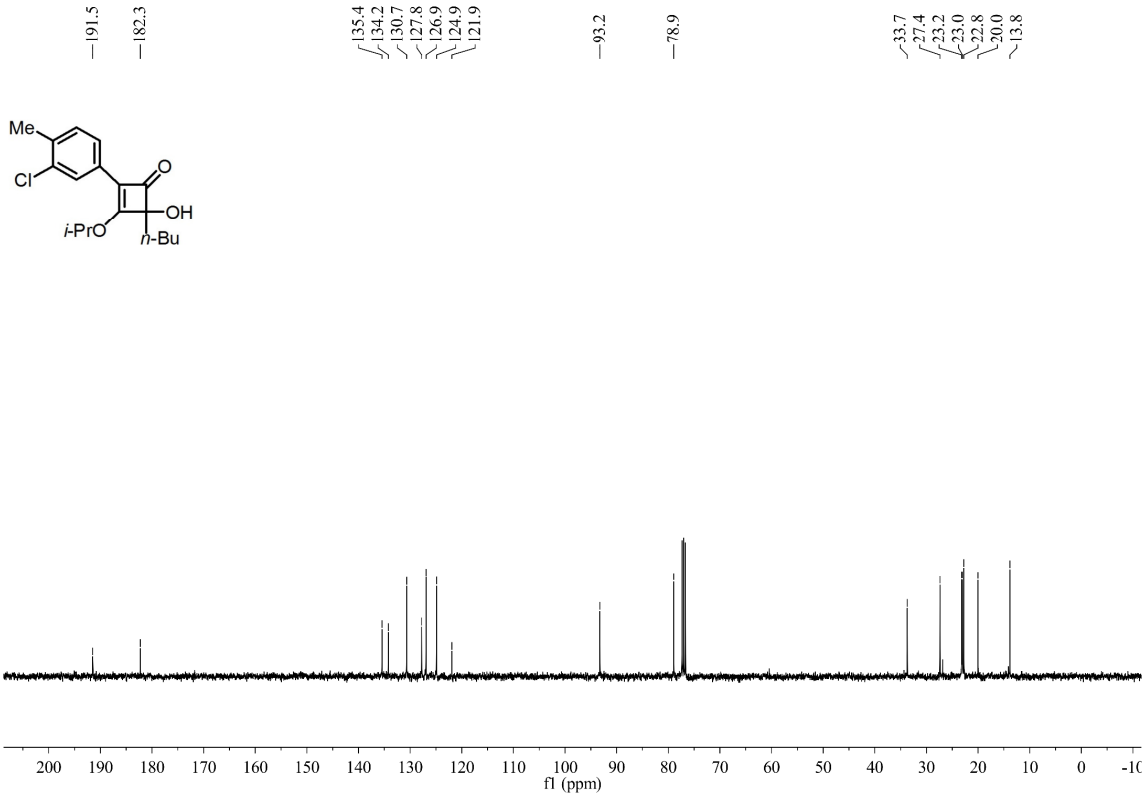

5.24
5.22
5.21
5.19
5.18
5.16
5.15
1.84
1.82
1.81
1.80
1.79
1.78
1.77
1.77
1.75
1.75
1.72
1.72
1.47
1.45
1.35
1.29
1.26
0.88
0.86
0.84
0.16
TMS
O
OH
i-PrO
n-Bu
0.95
1.98
5.93
3.90
3.09
9.05
fl (ppm)

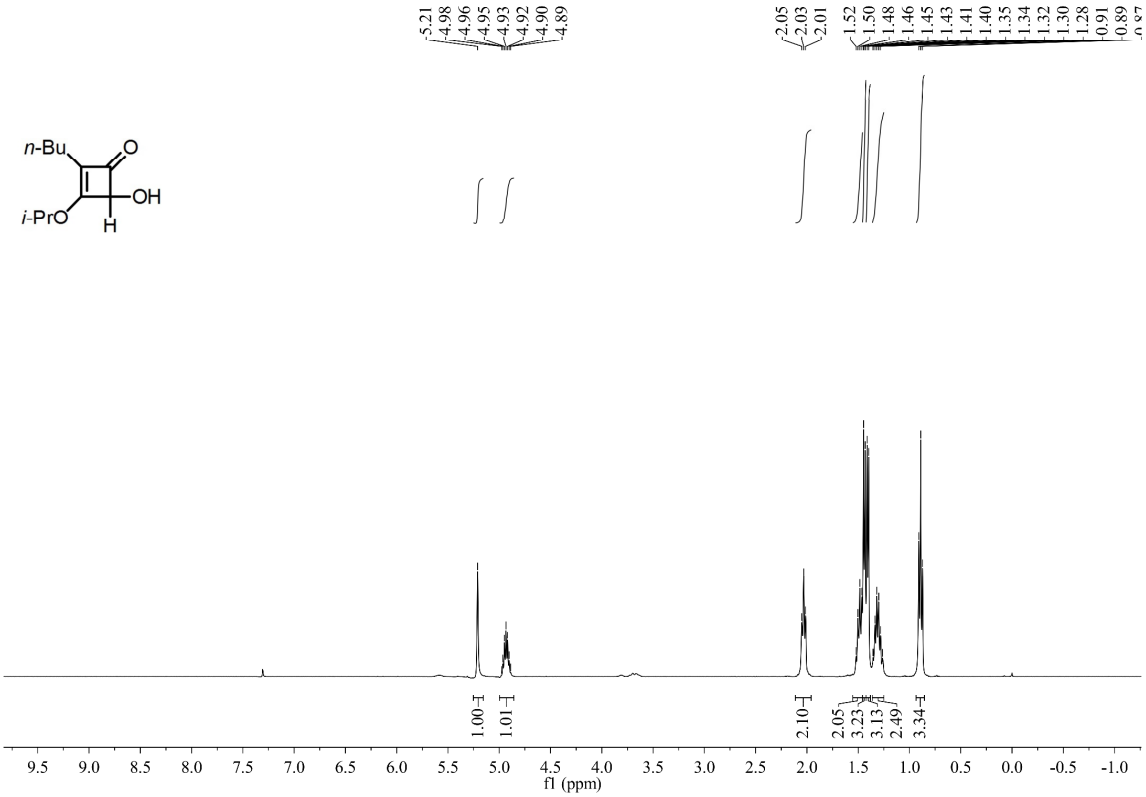

190.2
184.9
106.5
99.2
91.8
91.1
78.9
60.4
32.0
26.5
22.7
22.4
22.1
13.8
0.4
TMS
O
OH
i-PrO
n-Bu
fl (ppm)

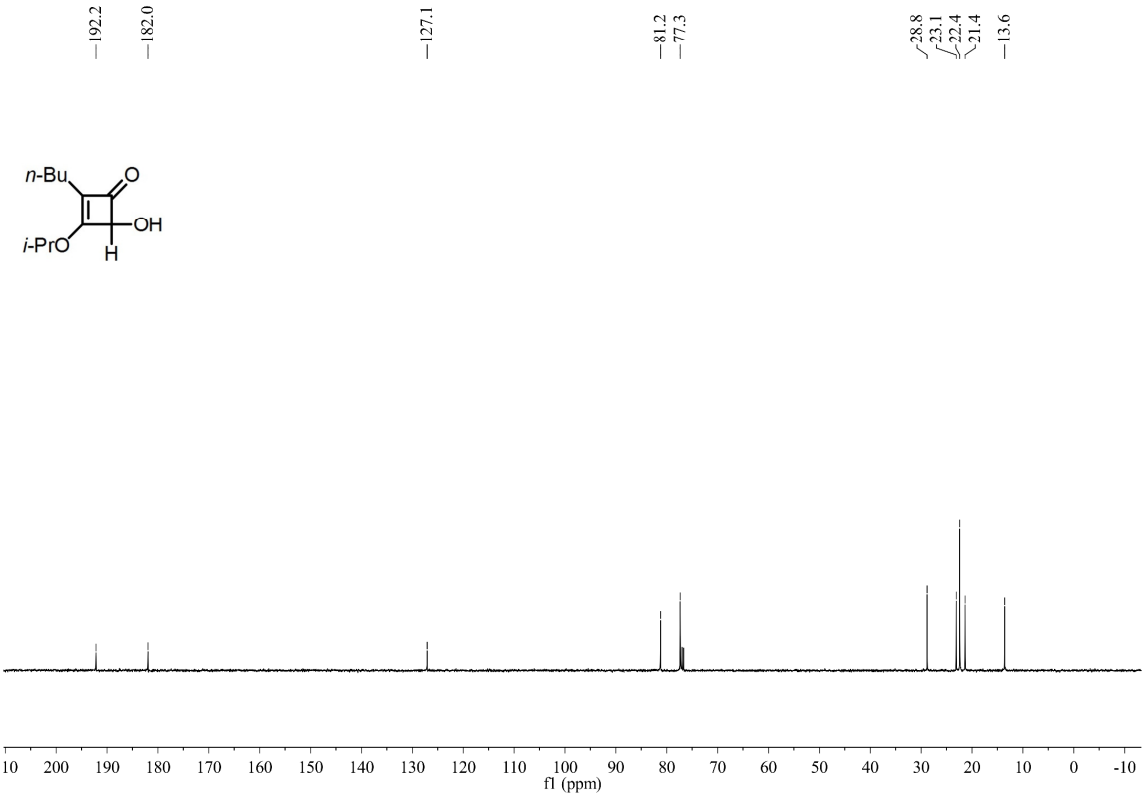

MeO
O
OH
i-PrO
n-Bu
f1 (ppm)

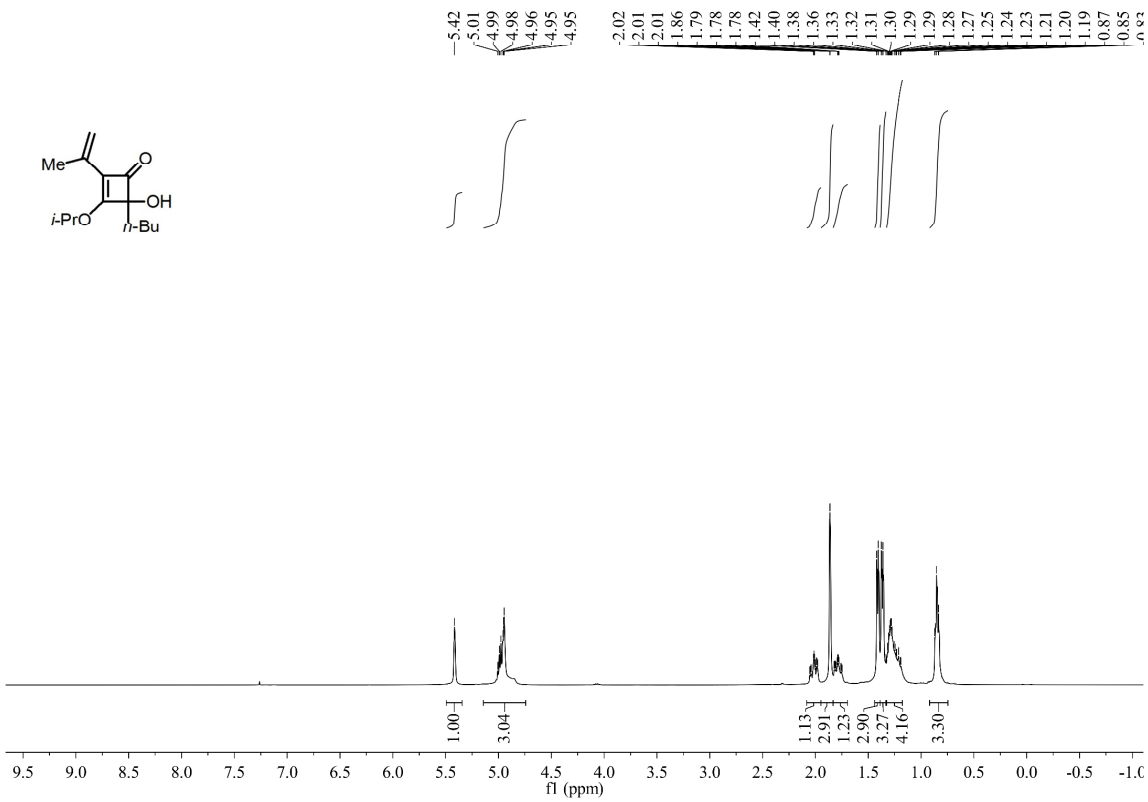

MeO
O
OH
i-PrO
n-Bu
f1 (ppm)


Me
Cl
O
OH
i-PrO
n-Bu
7.58
7.45
7.43
7.11
7.09
5.17
5.15
5.14
5.12
5.11
5.09
5.08
4.58
2.33
2.14
2.13
2.11
2.10
2.08
2.07
2.04
1.91
1.90
1.87
1.87
1.85
1.84
1.57
1.55
1.50
1.49
1.34
1.32
1.31
1.27
1.25
1.23
0.89
0.87
0.85
0.88
0.93
0.95
0.93
0.76
3.00
0.98
1.15
3.02
3.11
4.63
3.44
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


191.5
182.3
135.4
134.2
130.7
127.8
126.9
124.9
121.9
93.2
78.9
33.7
27.4
23.2
23.0
22.8
20.0
13.8
Me
Cl
O
OH
i-PrO
n-Bu
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


5.21
4.98
4.96
4.95
4.93
4.92
4.90
4.89
2.05
2.03
2.01
1.52
1.50
1.48
1.46
1.45
1.43
1.41
1.40
1.35
1.34
1.32
1.30
1.28
0.91
0.89
n-Bu
O
OH
i-PrO
H
1.00
1.01
2.10
2.05
3.23
3.13
2.49
3.34
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


192.2
182.0
127.1
81.2
77.3
28.8
23.1
22.4
21.4
13.6
n-Bu
O
OH
i-PrO
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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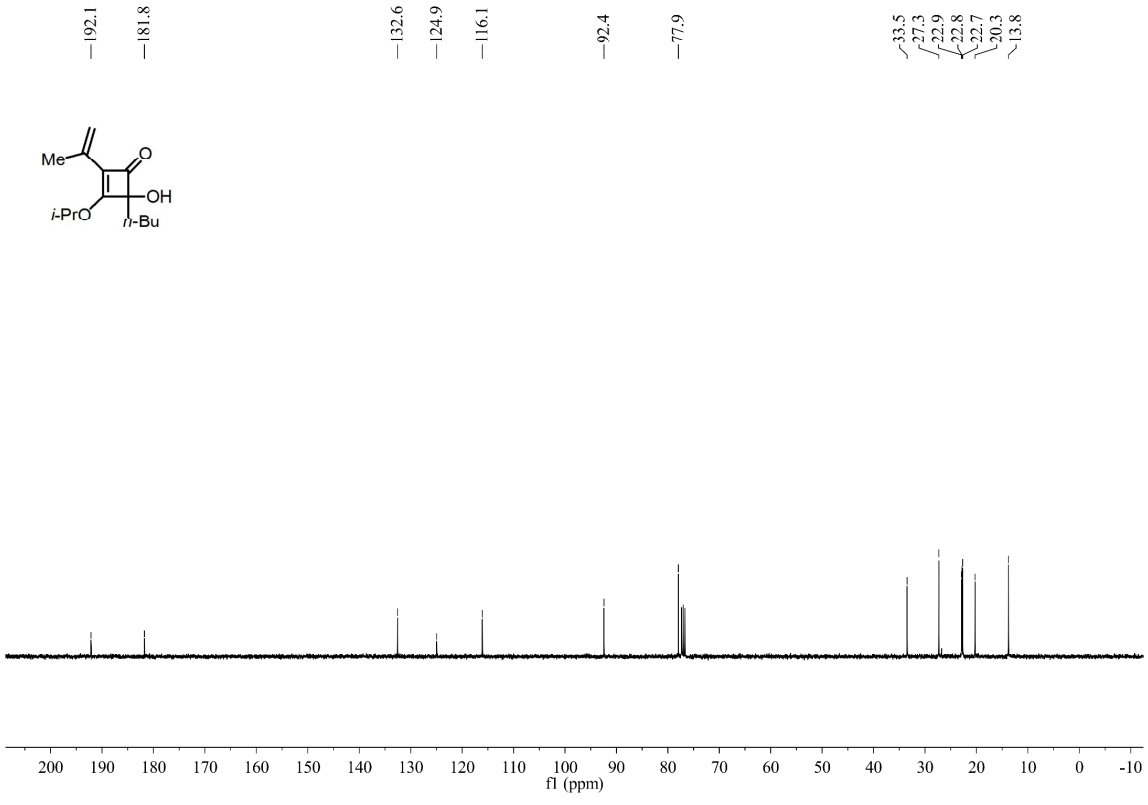
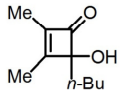
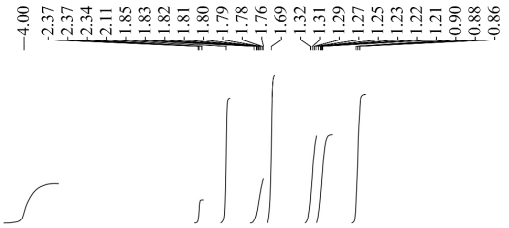
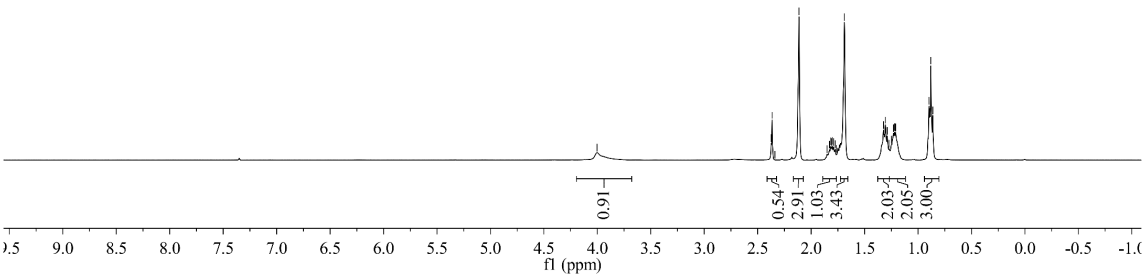
4.98
4.96
4.95
4.95
2.02
2.01
2.01
1.86
1.79
1.78
1.78
1.42
1.40
1.38
1.36
1.33
1.32
1.31
1.30
1.29
1.29
1.28
1.27
1.25
1.24
1.23
1.21
1.20
1.19
0.87
0.85
Me
O
OH
i-PrO
n-Bu
1.00
3.04
1.13
2.91
1.23
2.90
3.27
4.16
3.30
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

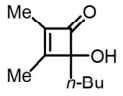

192.1
181.8
132.6
124.9
116.1
92.4
77.9
33.5
27.3
22.9
22.8
22.7
20.3
13.8
Me
O
OH
i-PrO
n-Bu
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

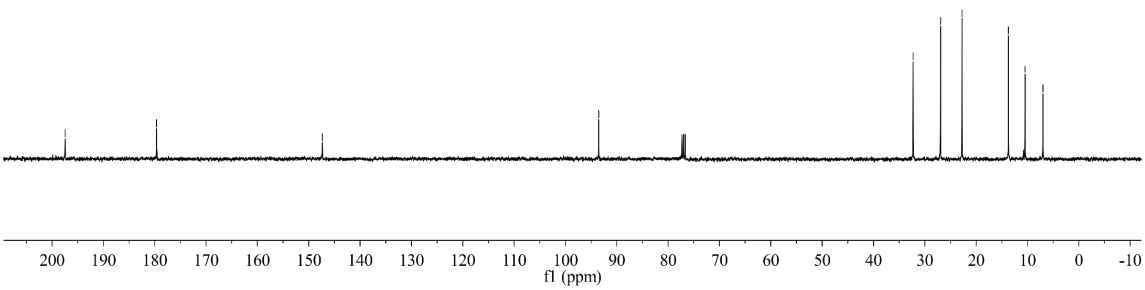

Me
O
OH
Me
n-Bu

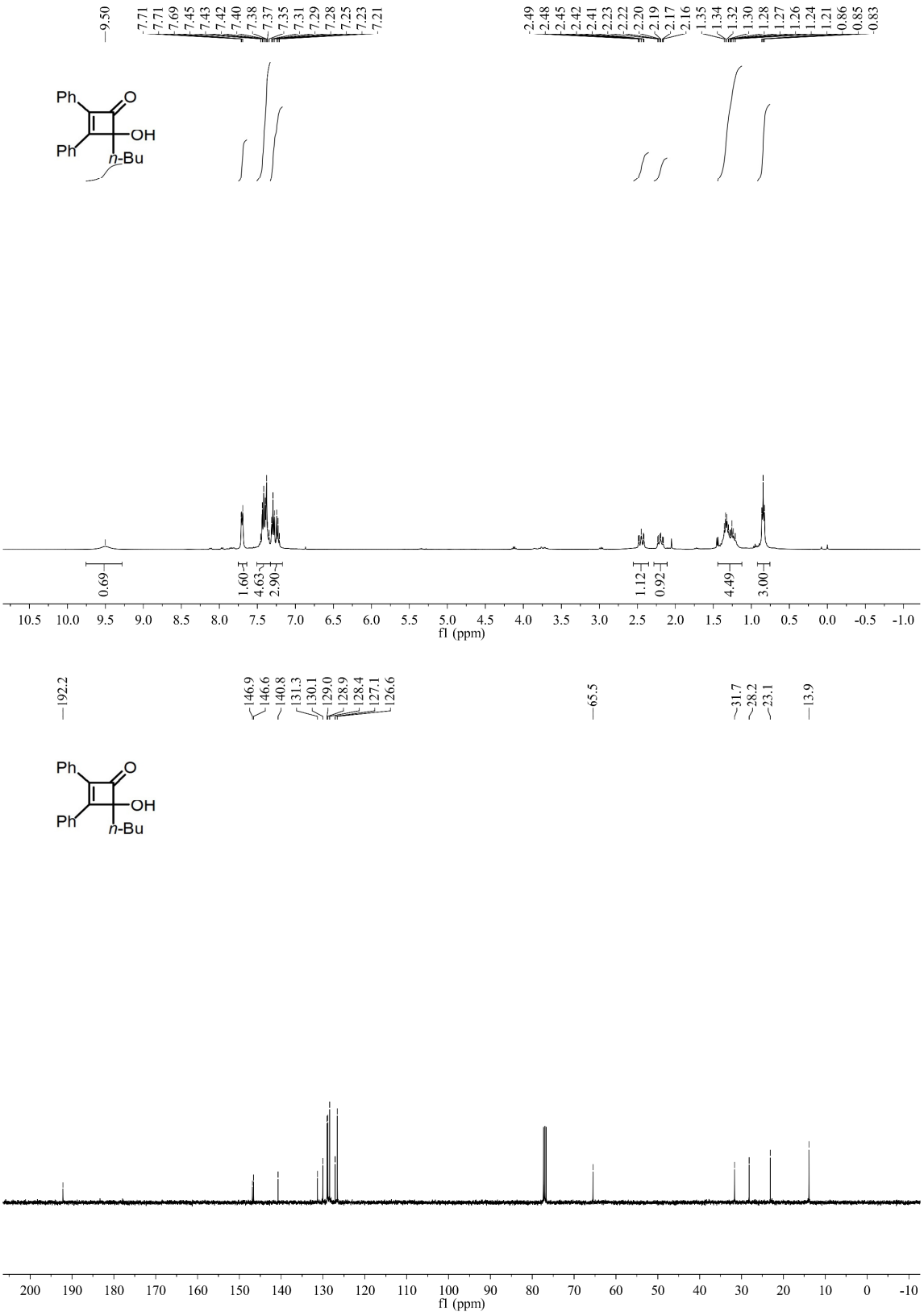

4.00
2.37
2.37
2.34
2.11
1.85
1.83
1.82
1.81
1.80
1.79
1.78
1.76
1.69
1.32
1.31
1.29
1.27
1.25
1.23
1.22
1.21
0.90
0.88
0.86

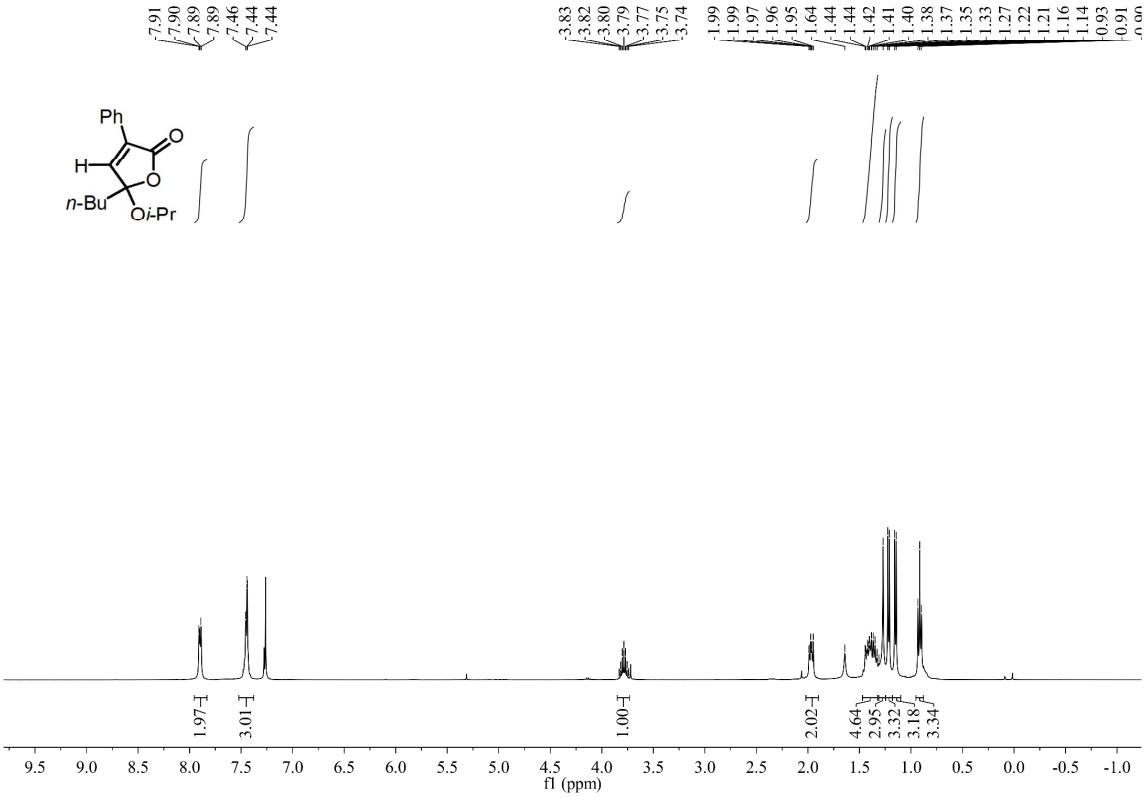

0.91
0.54
2.91
1.03
3.43
2.03
2.05
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

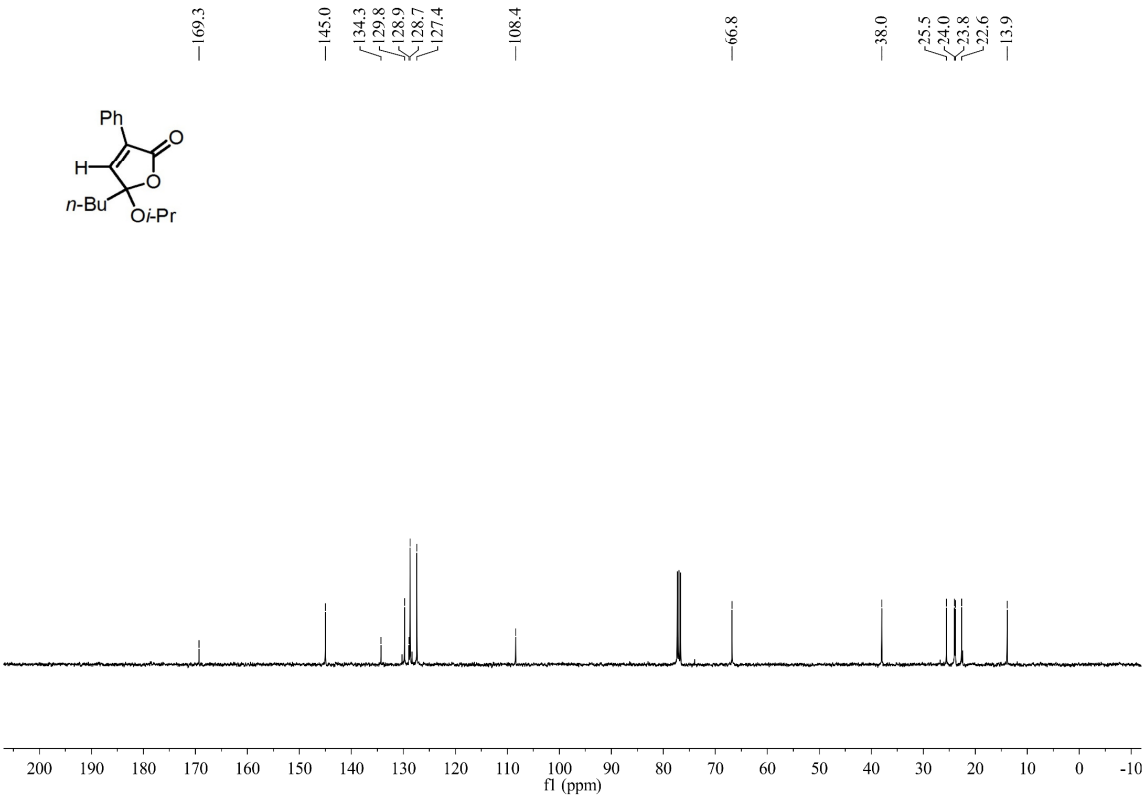

197.5
179.6
147.3
93.5
32.3
26.9
22.8
13.7
10.5
7.0

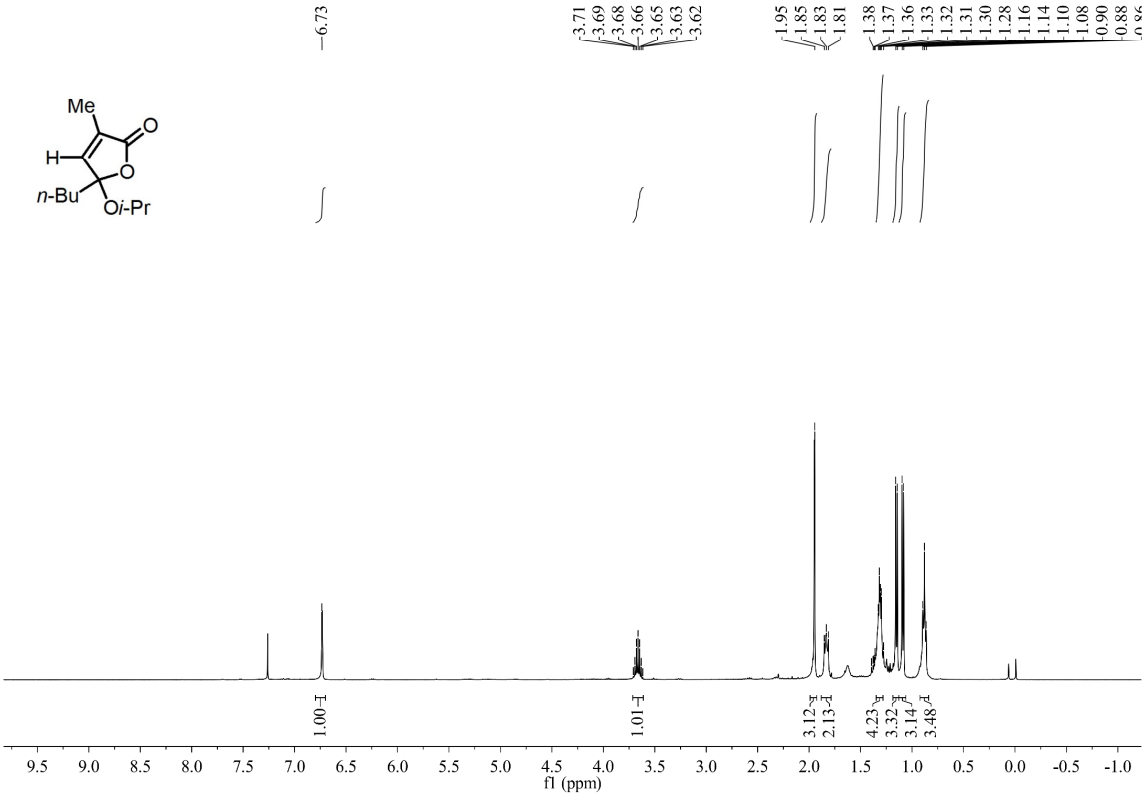

Me
O
OH
Me
n-Bu

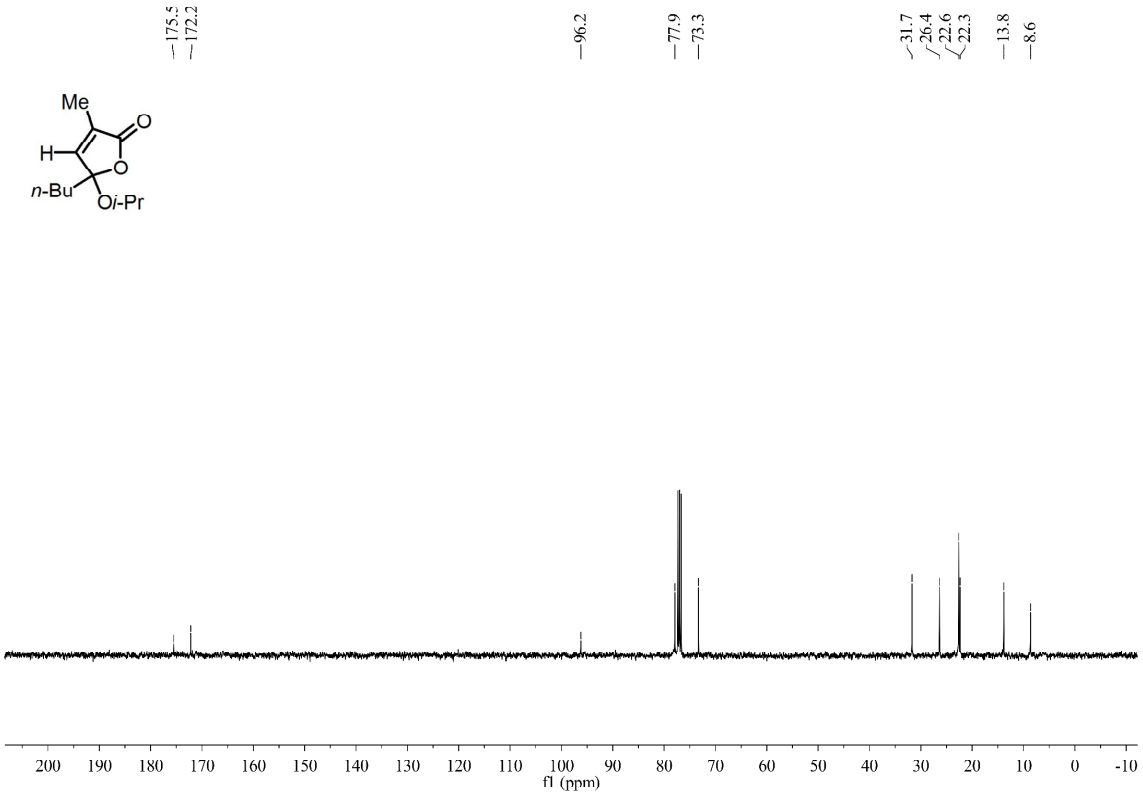

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

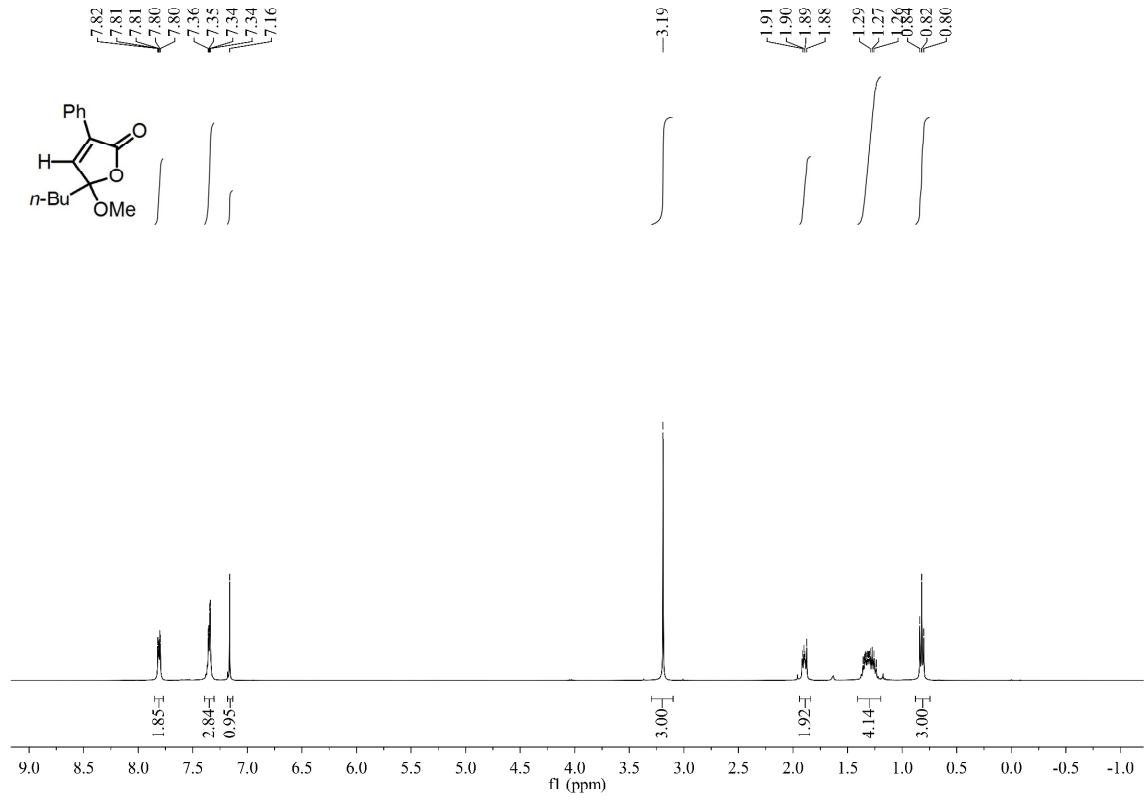

9.50
7.71
7.71
7.69
7.45
7.43
7.42
7.40
7.38
7.37
7.35
7.31
7.29
7.28
7.25
7.23
7.21
2.49
2.48
2.45
2.42
2.41
2.23
2.22
2.20
2.19
2.17
2.16
1.35
1.34
1.32
1.30
1.28
1.27
1.26
1.24
1.21
0.86
0.85
0.83
Ph
O
OH
Ph
n-Bu
0.69
1.60
4.63
2.90
1.12
0.92
4.49
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)
192.2
146.9
146.6
140.8
131.3
130.1
129.0
128.9
128.4
127.1
126.6
65.5
31.7
28.2
23.1
13.9
Ph
O
OH
Ph
n-Bu
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

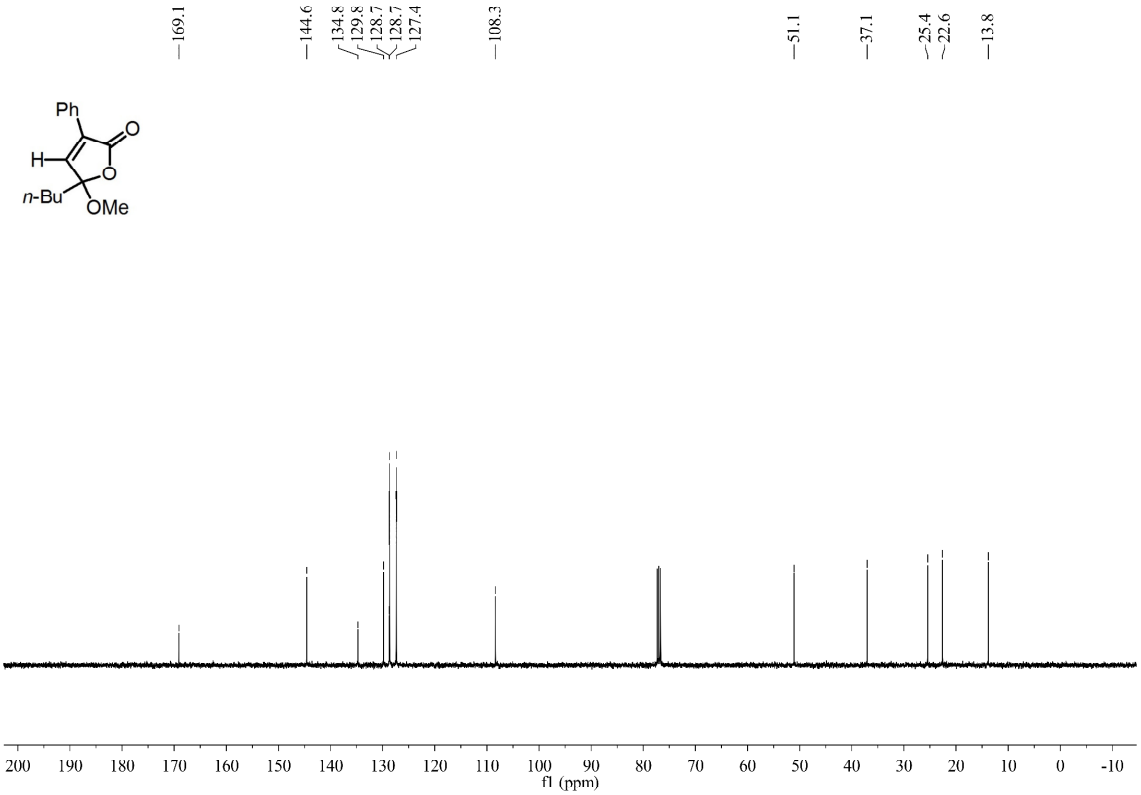

7.91
7.90
7.89
7.89
7.46
7.44
7.44
3.83
3.82
3.80
3.79
3.77
3.75
3.74
1.99
1.99
1.97
1.96
1.95
1.64
1.44
1.44
1.42
1.41
1.40
1.38
1.37
1.35
1.33
1.27
1.22
1.21
1.16
1.14
0.93
0.91
Ph
H
O
O
n-Bu
Oi-Pr
1.97
3.01
1.00
2.02
4.64
2.95
3.32
3.18
3.34
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

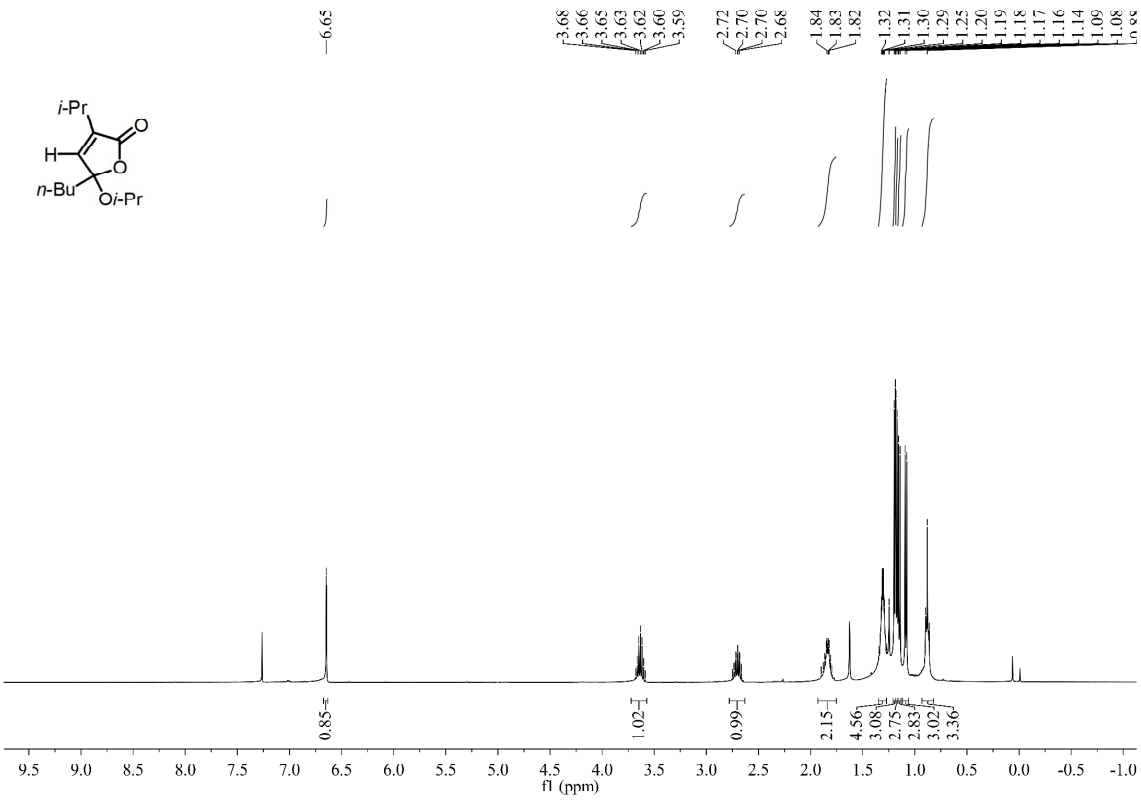

169.3
145.0
134.3
129.8
128.9
128.7
127.4
108.4
66.8
38.0
25.5
24.0
23.8
22.6
13.9
Ph
H
O
O
n-Bu
Oi-Pr
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

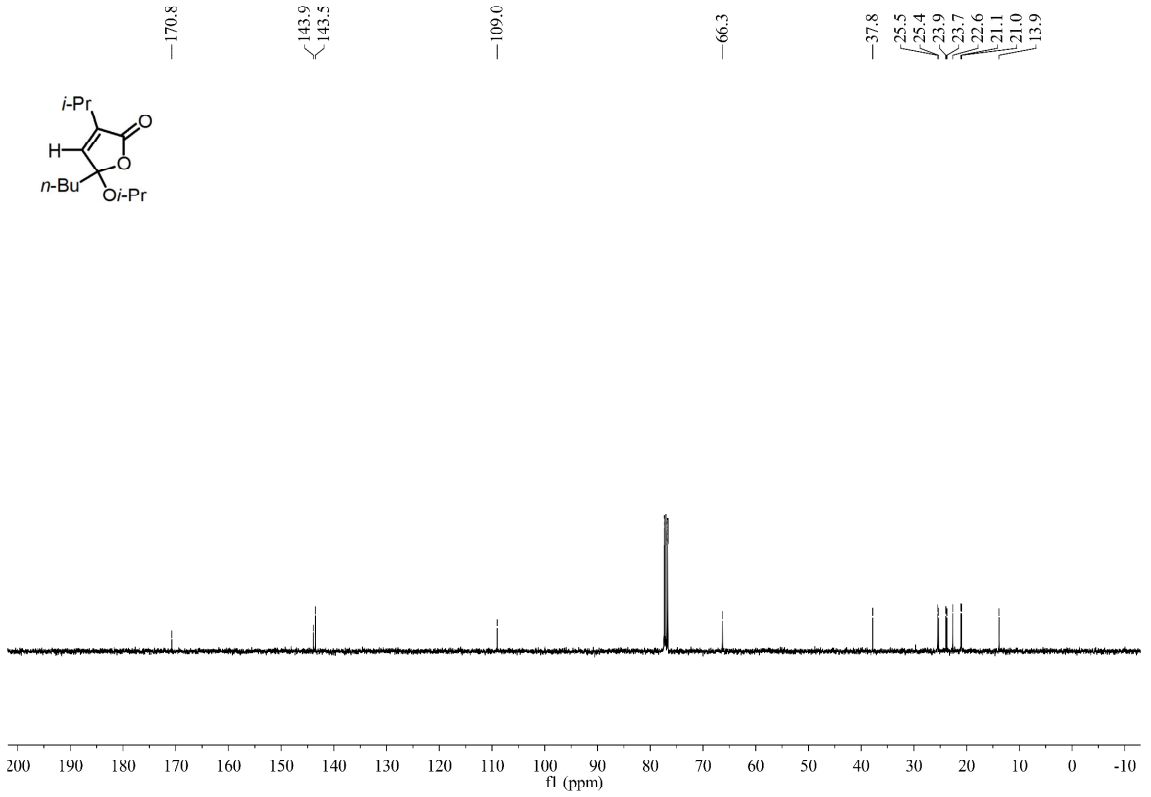

Me
H
n-Bu
O
O
Oi-Pr
6.73
3.71
3.69
3.68
3.66
3.65
3.63
3.62
1.95
1.85
1.83
1.81
1.38
1.37
1.36
1.33
1.32
1.31
1.30
1.28
1.16
1.14
1.10
1.08
0.90
0.88
1.00
1.01
3.12
2.13
4.23
3.32
3.14
3.48
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

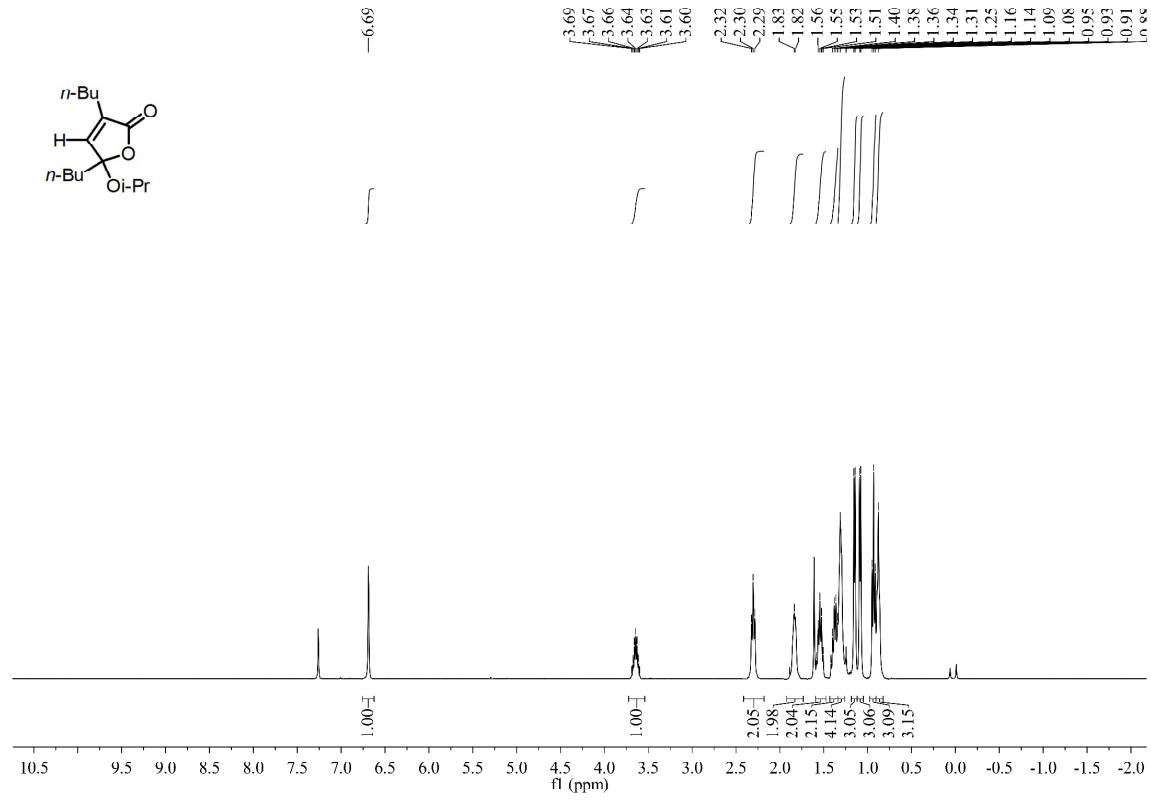

175.5
172.2
96.2
77.9
73.3
31.7
26.4
22.6
22.3
13.8
8.6
Me
O
H
O
n-Bu
Oi-Pr
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

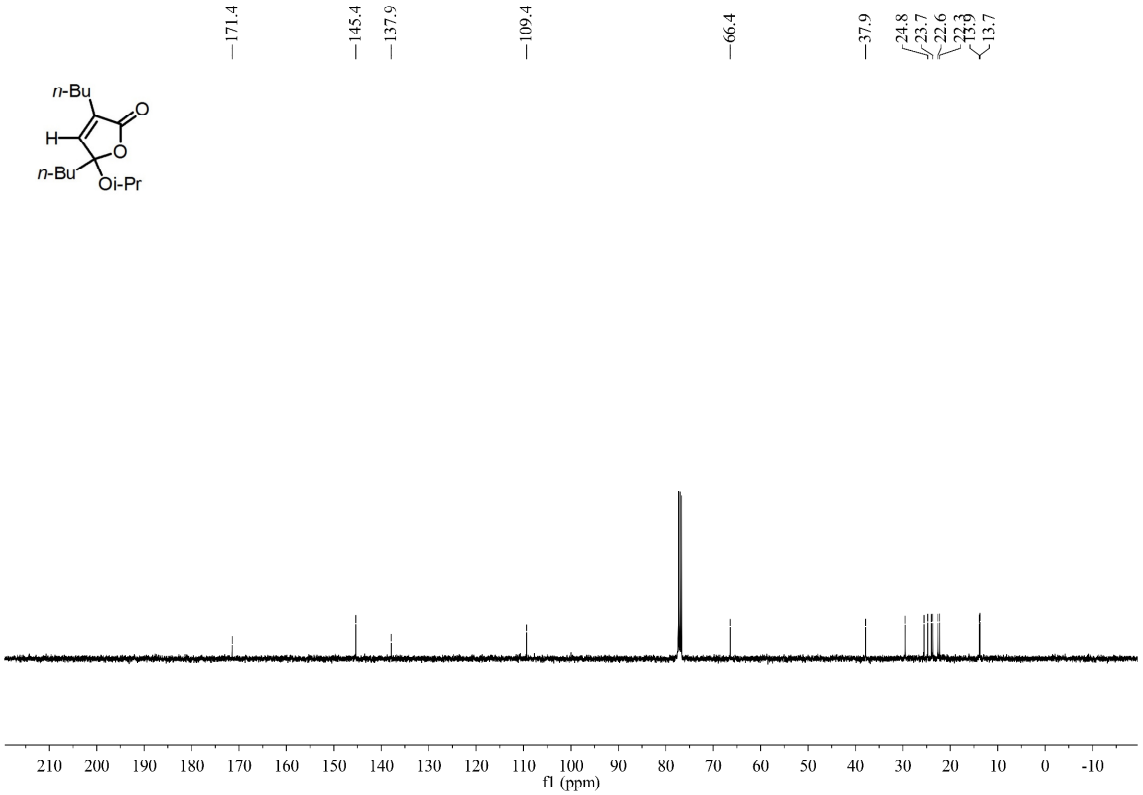

7.82
7.81
7.81
7.80
7.80
7.36
7.35
7.34
7.34
7.16
3.19
1.91
1.90
1.89
1.88
1.29
1.27
0.84
0.82
0.80
Ph
O
H
O
n-Bu
OMe
1.85
2.84
0.95
3.00
1.92
4.14
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

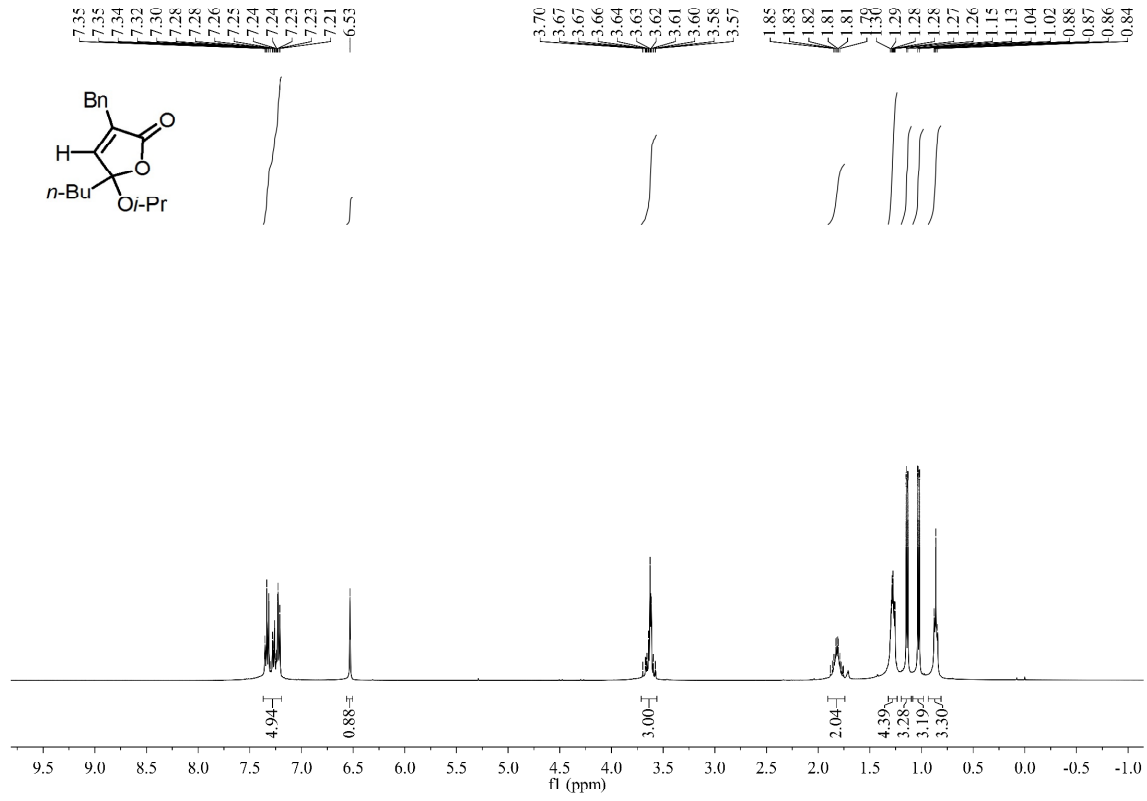

169.1
144.6
134.8
129.8
128.7
128.7
127.4
108.3
51.1
37.1
25.4
22.6
13.8
Ph
H
O
O
n-Bu
OMe
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

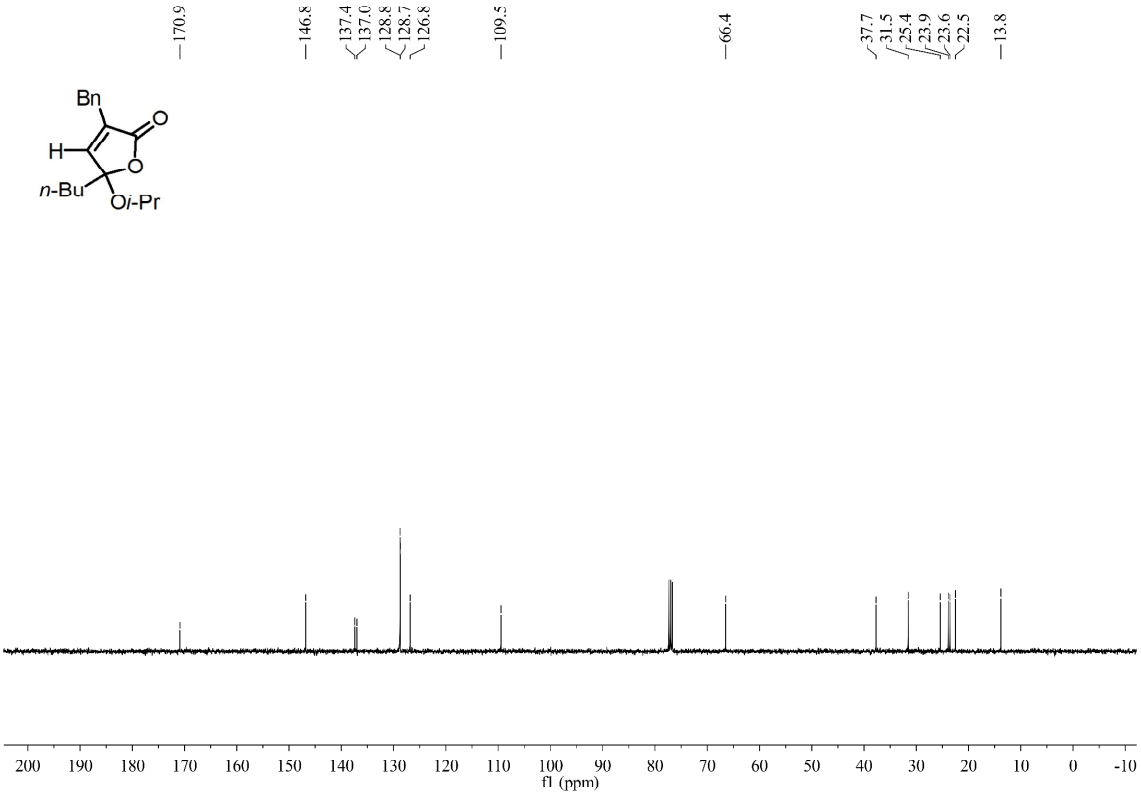

6.65
3.68
3.66
3.65
3.63
3.62
3.60
3.59
2.72
2.70
2.70
2.68
1.84
1.83
1.82
1.32
1.31
1.30
1.29
1.25
1.20
1.19
1.18
1.17
1.16
1.14
1.09
1.08
i-Pr
H
O
O
n-Bu
Oi-Pr
0.85
1.02
0.99
2.15
4.56
3.08
2.75
2.83
3.02
3.36
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

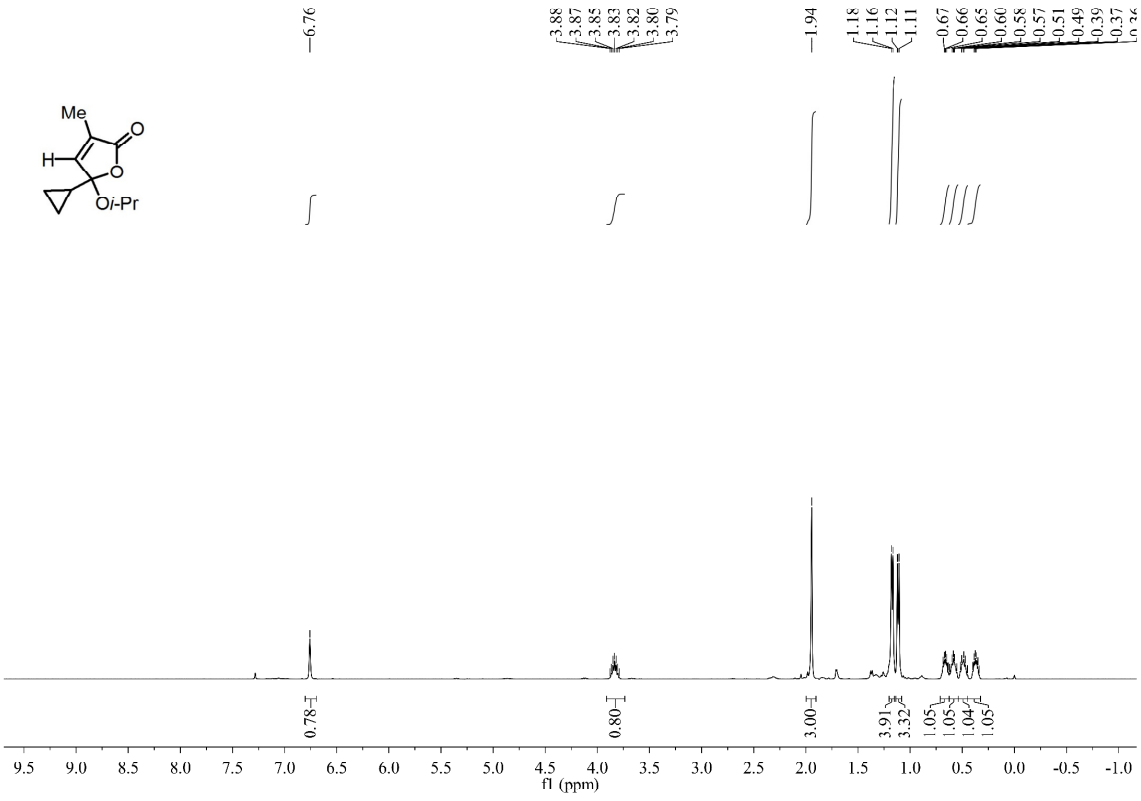

170.8
143.9
143.5
109.0
66.3
37.8
25.5
25.4
23.9
23.7
22.6
21.1
21.0
13.9
i-Pr
H
n-Bu
Oi-Pr
f1 (ppm)

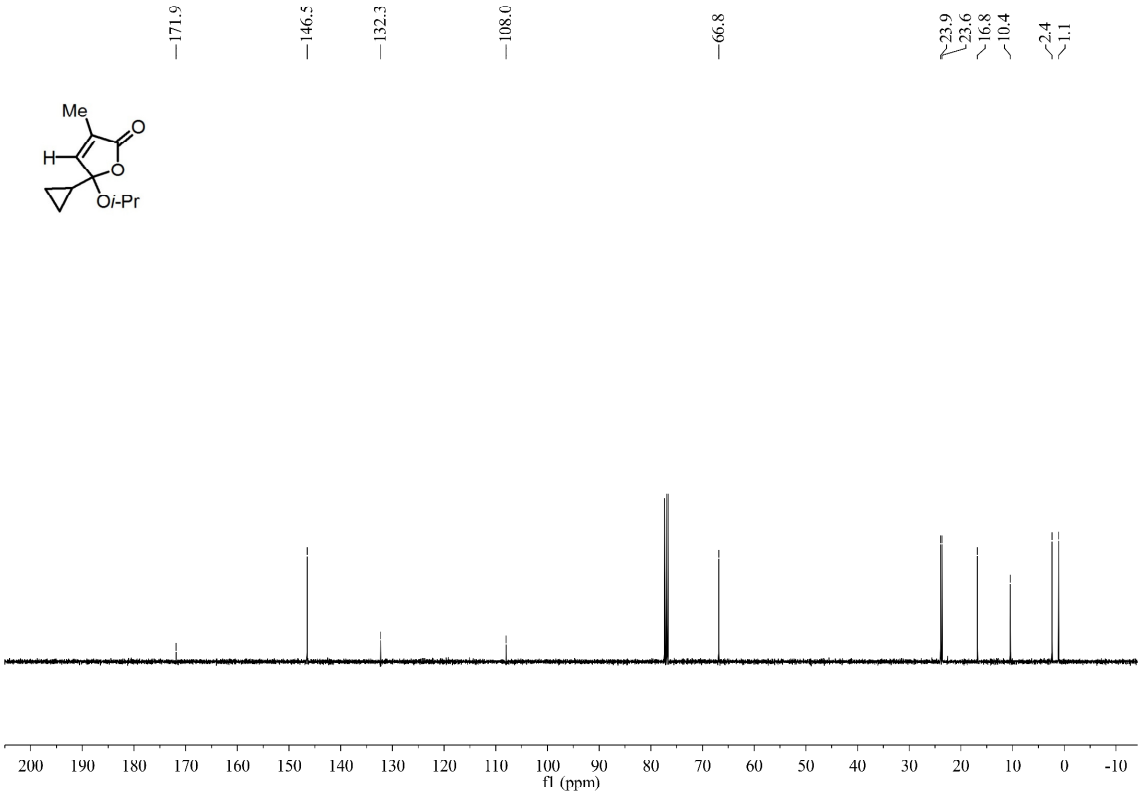

6.69
3.69
3.67
3.66
3.64
3.63
3.61
3.60
2.32
2.30
2.29
1.83
1.82
1.56
1.55
1.53
1.51
1.40
1.38
1.36
1.34
1.31
1.25
1.16
1.14
1.09
1.08
0.95
0.93
0.91
n-Bu
H
n-Bu
Oi-Pr
1.00
1.00
2.05
1.98
2.04
2.15
4.14
3.05
3.06
3.09
3.15
f1 (ppm)

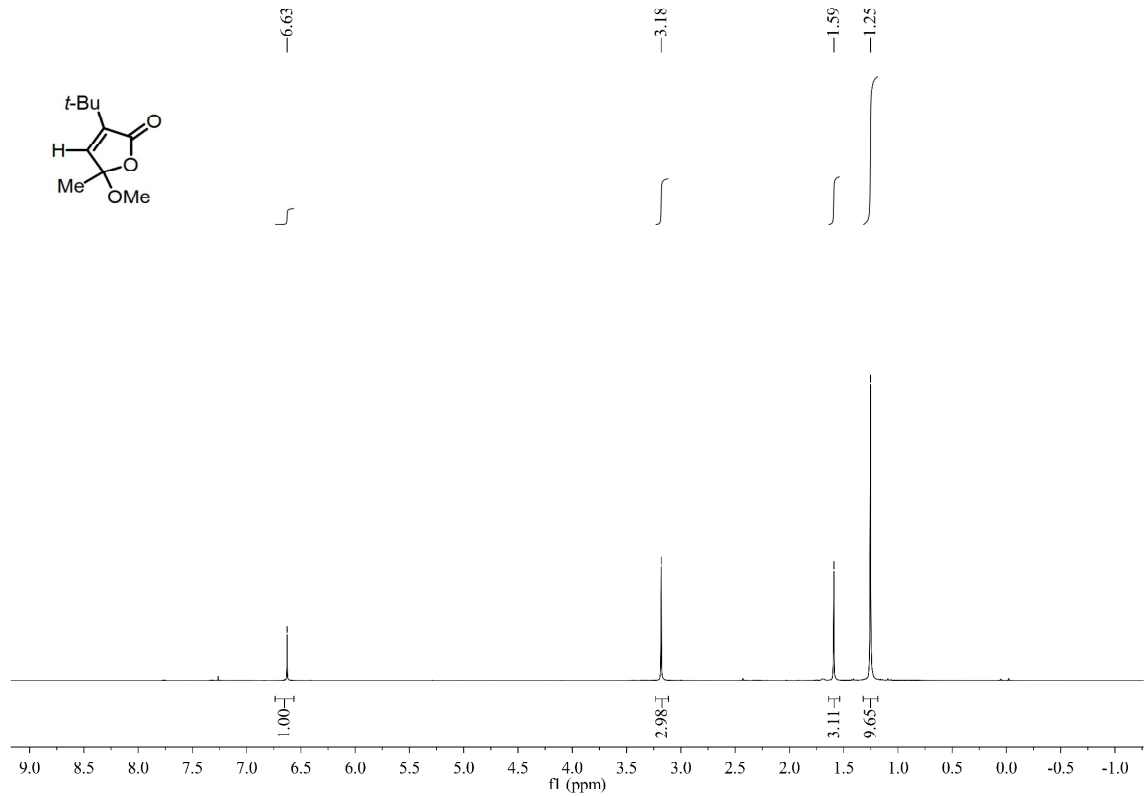

171.4
145.4
137.9
109.4
66.4
37.9
24.8
23.7
22.6
22.3
13.9
13.7
n-Bu
H
n-Bu
Oi-Pr
O
O
f1 (ppm)

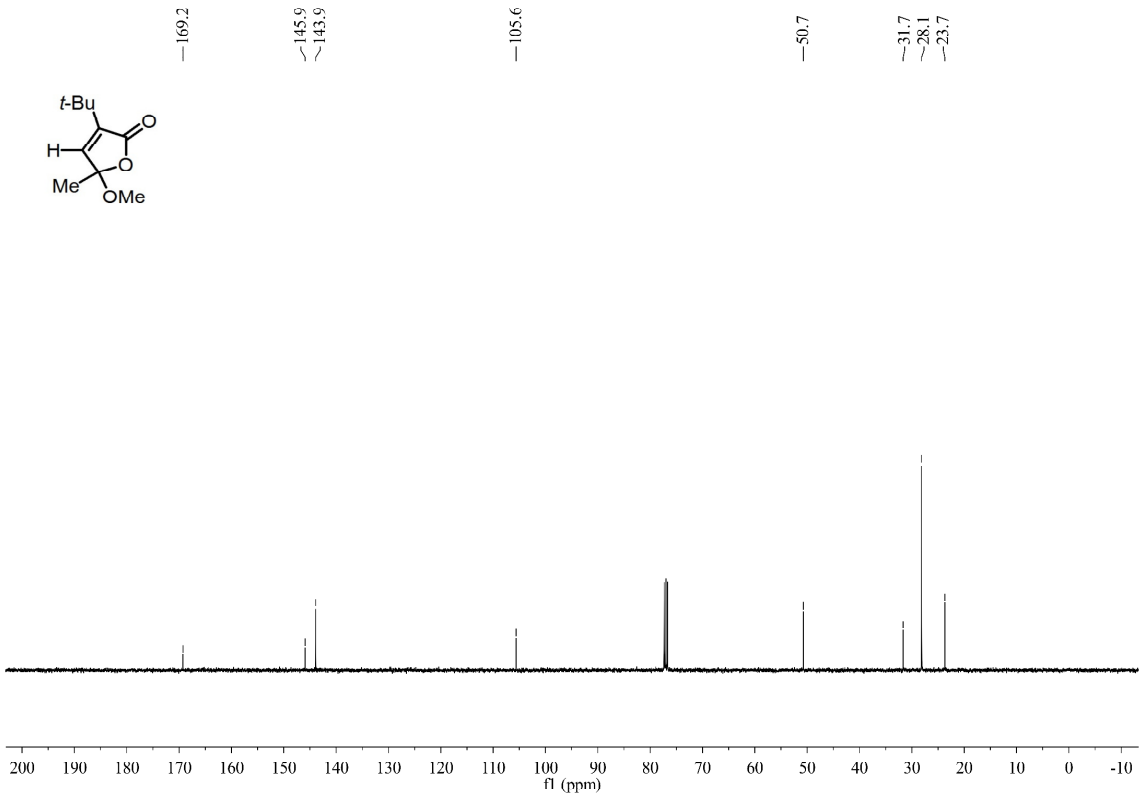

7.35
7.35
7.34
7.32
7.30
7.28
7.28
7.26
7.25
7.24
7.24
7.23
7.23
7.21
6.53
3.70
3.67
3.67
3.66
3.64
3.63
3.62
3.61
3.60
3.58
3.57
1.85
1.83
1.82
1.81
1.81
1.79
1.30
1.29
1.28
1.28
1.27
1.26
1.15
1.13
1.04
1.02
0.88
0.87
0.86
0.84
Bn
H
n-Bu
Oi-Pr
O
O
4.94
0.88
3.00
2.04
4.39
3.28
3.19
3.30
f1 (ppm)

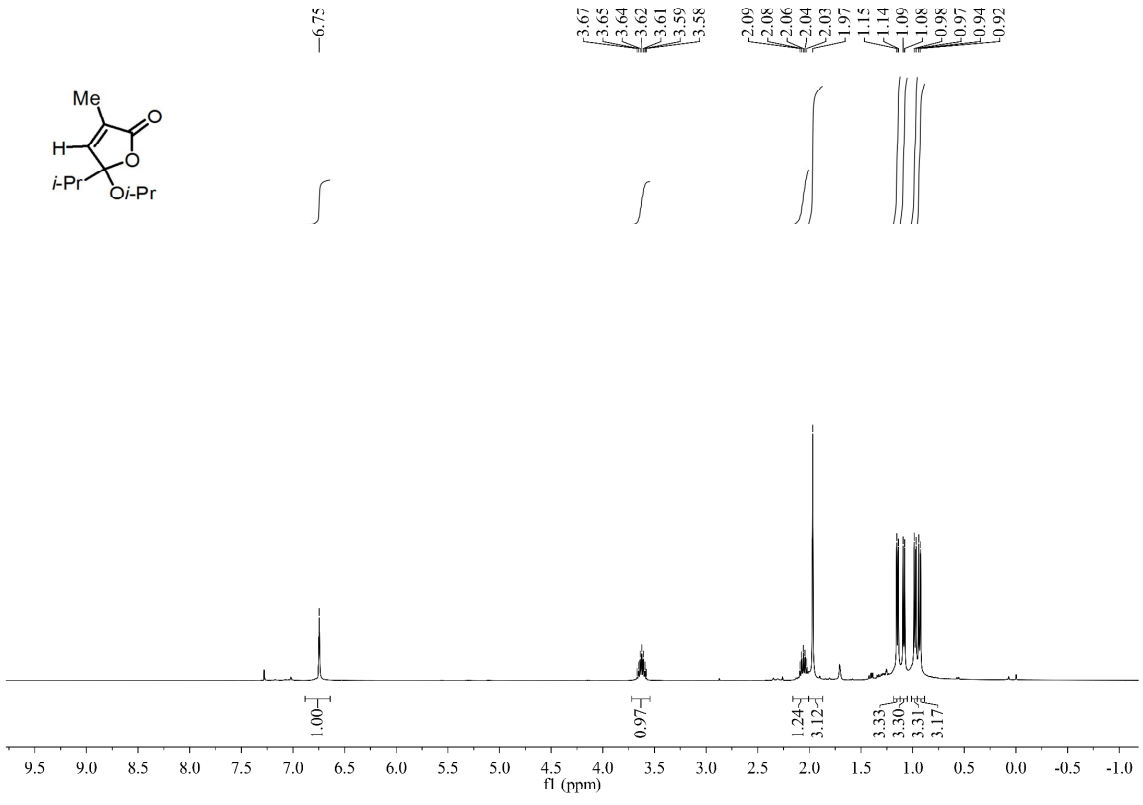

Bn
H
n-Bu
Oi-Pr
O
O
170.9
146.8
137.4
137.0
128.8
128.7
126.8
109.5
66.4
37.7
31.5
25.4
23.9
23.6
22.5
13.8
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Me
H
Oi-Pr
O
O
6.76
3.88
3.87
3.85
3.83
3.82
3.80
3.79
1.94
1.18
1.16
1.12
1.11
0.67
0.66
0.65
0.60
0.58
0.57
0.51
0.49
0.39
0.37
0.78
0.80
3.00
3.91
3.32
1.05
1.05
1.04
1.05
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


171.9
146.5
132.3
108.0
66.8
23.9
23.6
16.8
10.4
2.4
1.1
Me
H
O
O
Oi-Pr
f1 (ppm)


6.63
3.18
1.59
1.25
t-Bu
H
O
O
Me
OMe
1.00
2.98
3.11
9.65
f1 (ppm)


169.2
145.9
143.9
105.6
50.7
31.7
28.1
23.7
t-Bu
H
Me
OMe
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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3.67
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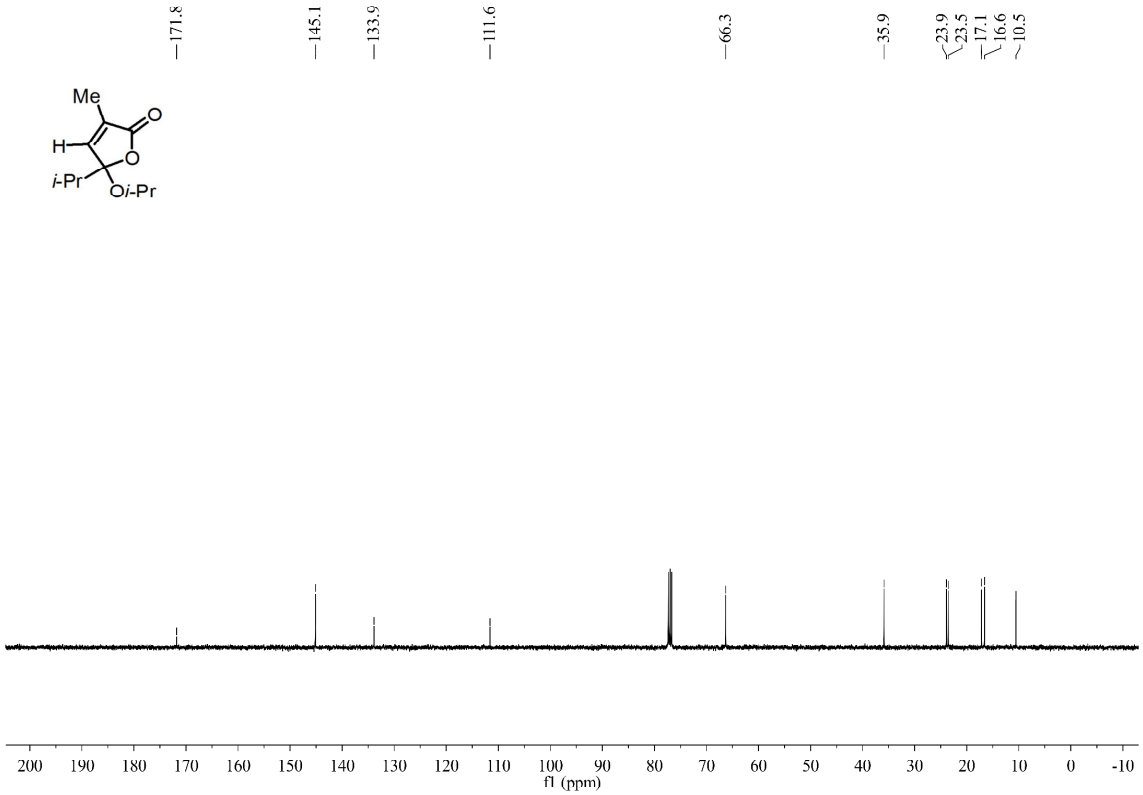
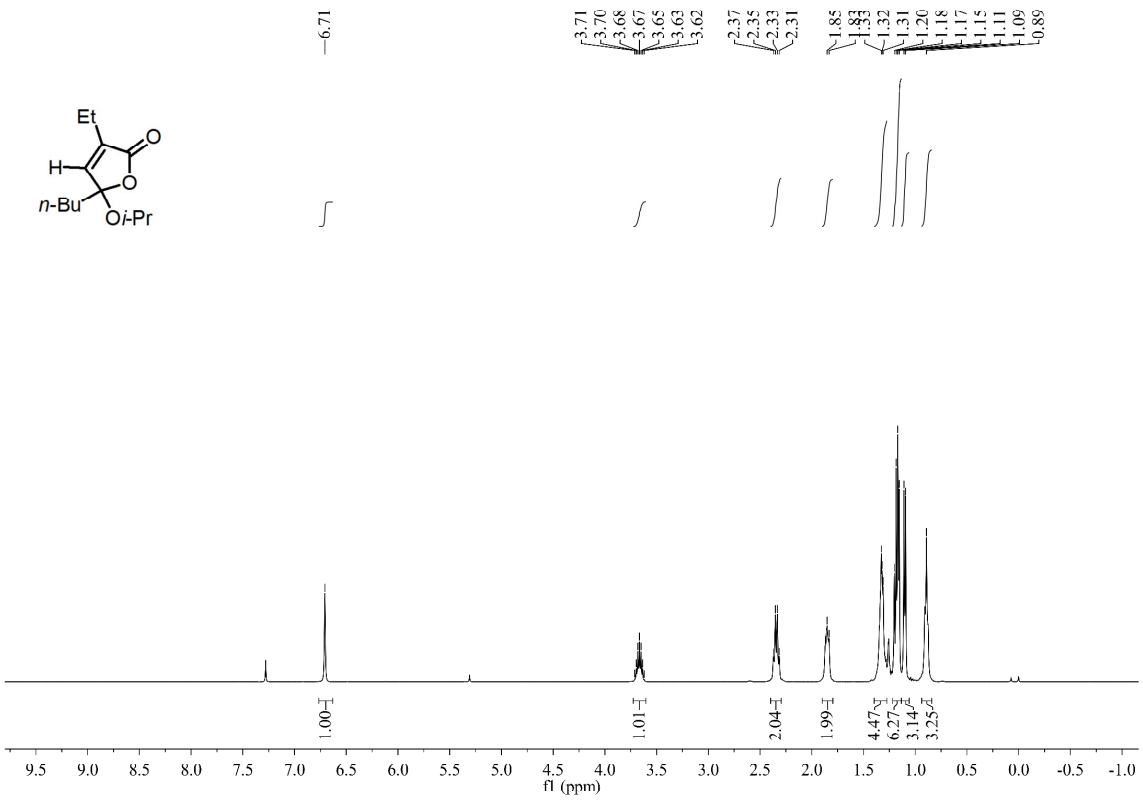
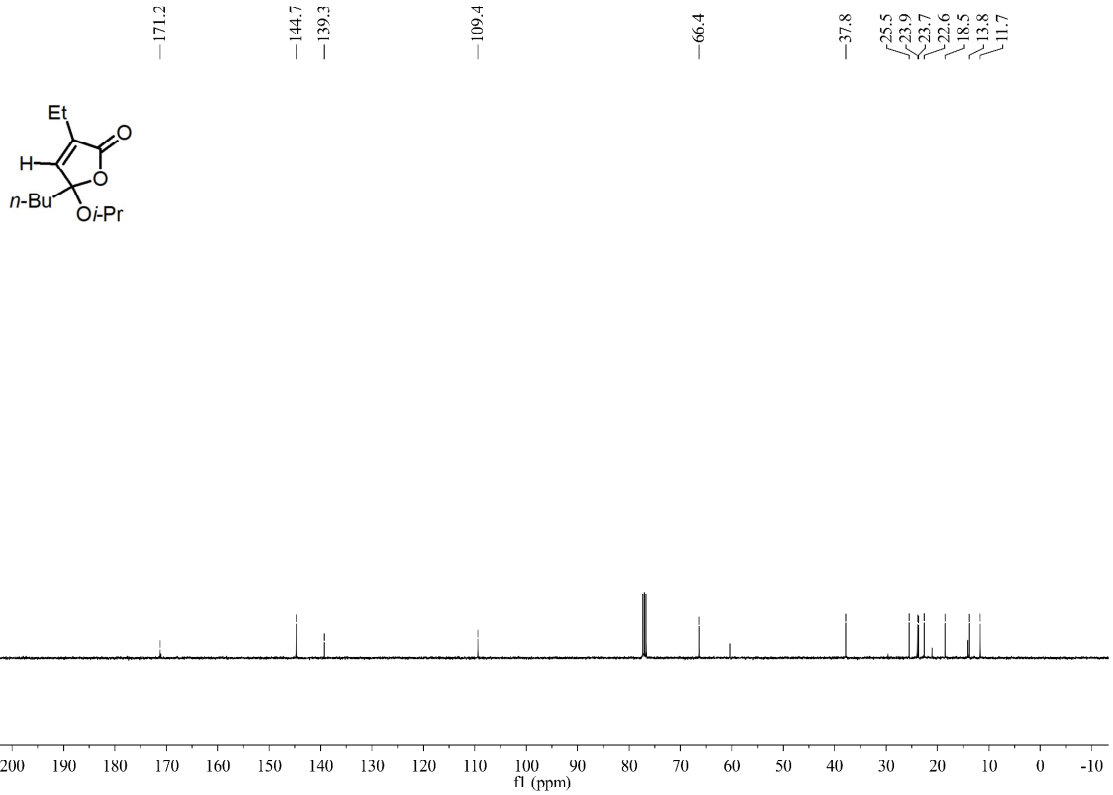
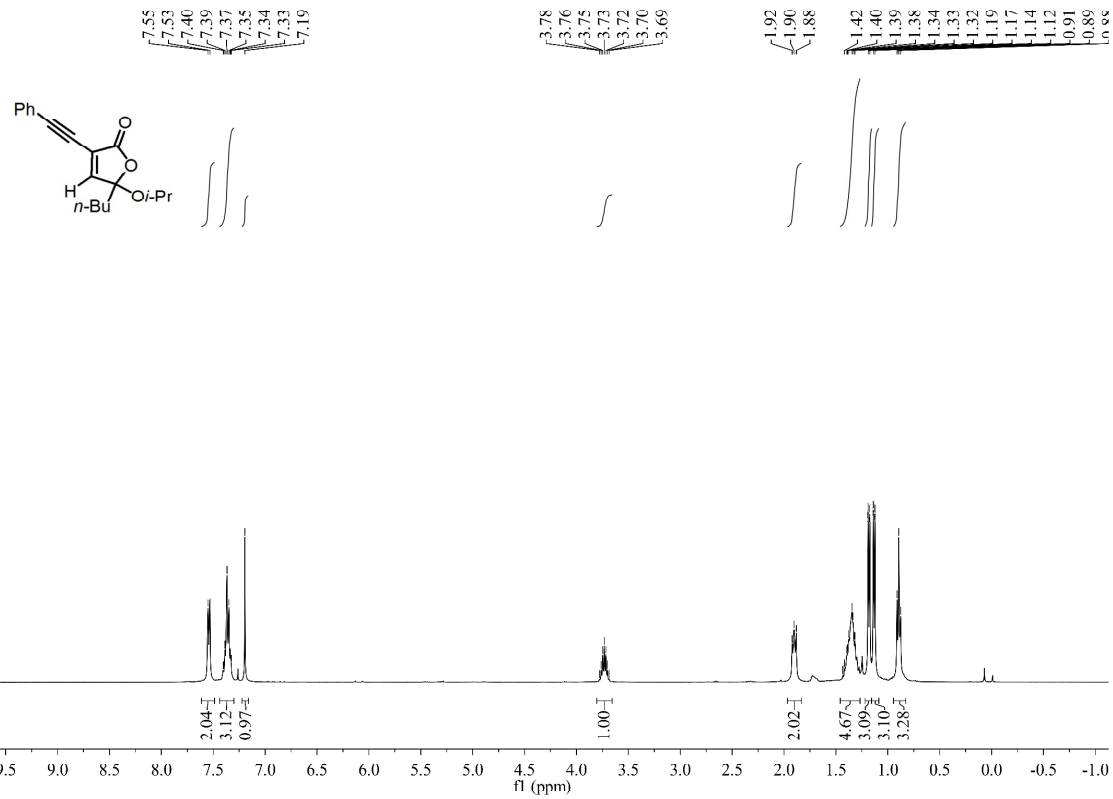
3.64
3.62
3.61
3.59
3.58
2.09
2.08
2.06
2.04
2.03
1.97
1.15
1.14
1.09
1.08
0.98
0.97
0.94
0.92
Me
H
i-Pr
Oi-Pr
O
1.00
0.97
1.24
3.12
3.33
3.30
3.31
3.17
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


171.8
145.1
133.9
111.6
66.3
35.9
23.9
23.5
17.1
16.6
10.5
Me
H
O
O
i-Pr
Oi-Pr
fl (ppm)


6.71
3.71
3.70
3.68
3.67
3.65
3.63
3.62
2.37
2.35
2.33
2.31
1.85
1.83
1.33
1.32
1.31
1.20
1.18
1.17
1.15
1.11
1.09
0.89
Et
H
O
O
n-Bu
Oi-Pr
1.00
1.01
2.04
1.99
4.47
6.27
3.14
3.25
fl (ppm)


171.2
144.7
139.3
109.4
66.4
37.8
25.5
23.9
23.7
22.6
18.5
13.8
11.7
Et
H
n-Bu
Oi-Pr
f1 (ppm)


7.55
7.53
7.40
7.39
7.37
7.35
7.34
7.33
7.19
3.78
3.76
3.75
3.73
3.72
3.70
3.69
1.92
1.90
1.88
1.42
1.40
1.39
1.38
1.34
1.33
1.32
1.19
1.17
1.14
1.12
0.91
0.89
Ph
H
n-Bu
Oi-Pr
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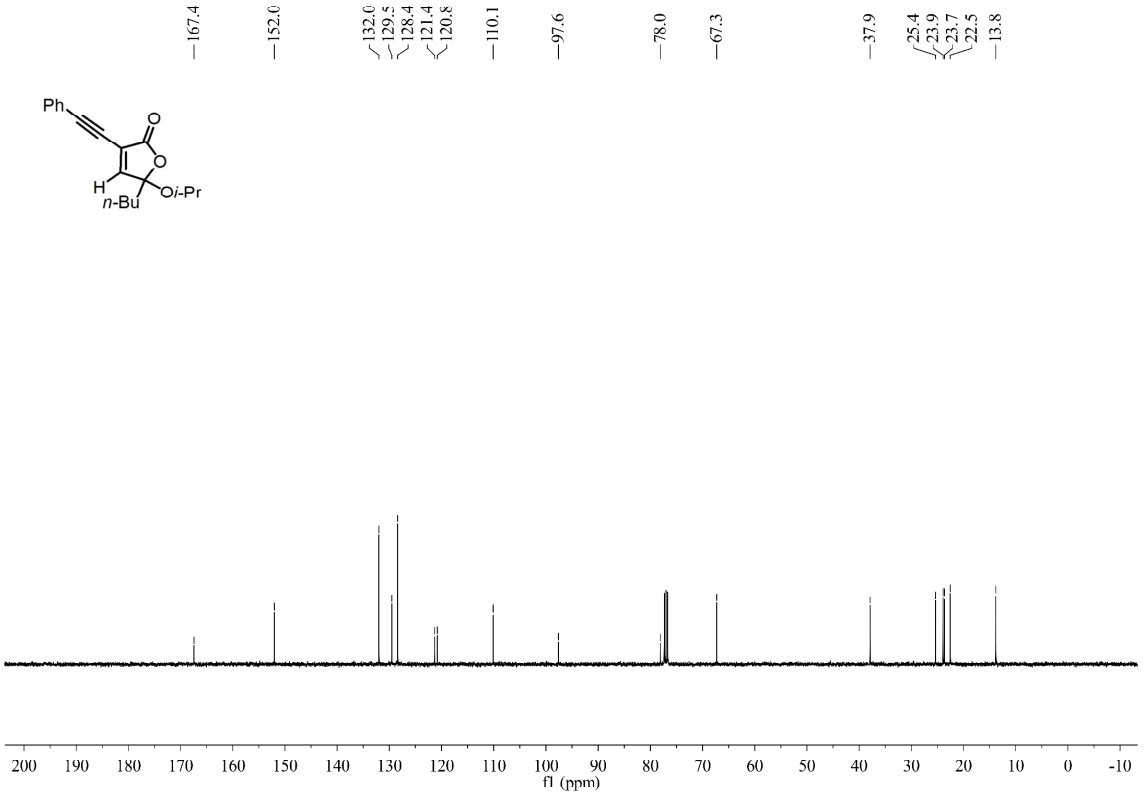
3.12
0.97
1.00
2.02
4.67
3.09
3.10
3.28
f1 (ppm)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132.0
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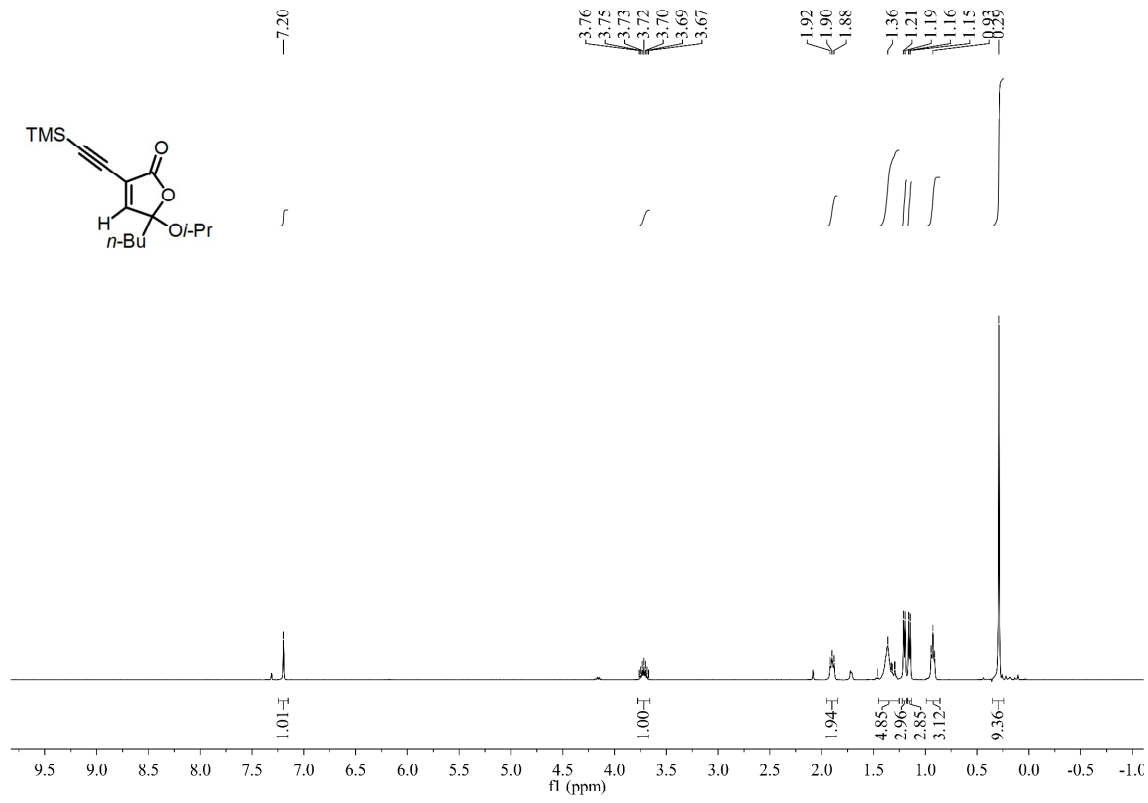
128.4
121.4
120.8
110.1
97.6
78.0
67.3
37.9
25.4
23.9
23.7
22.5
13.8
Ph
O
O
H
n-Bu
Oi-Pr
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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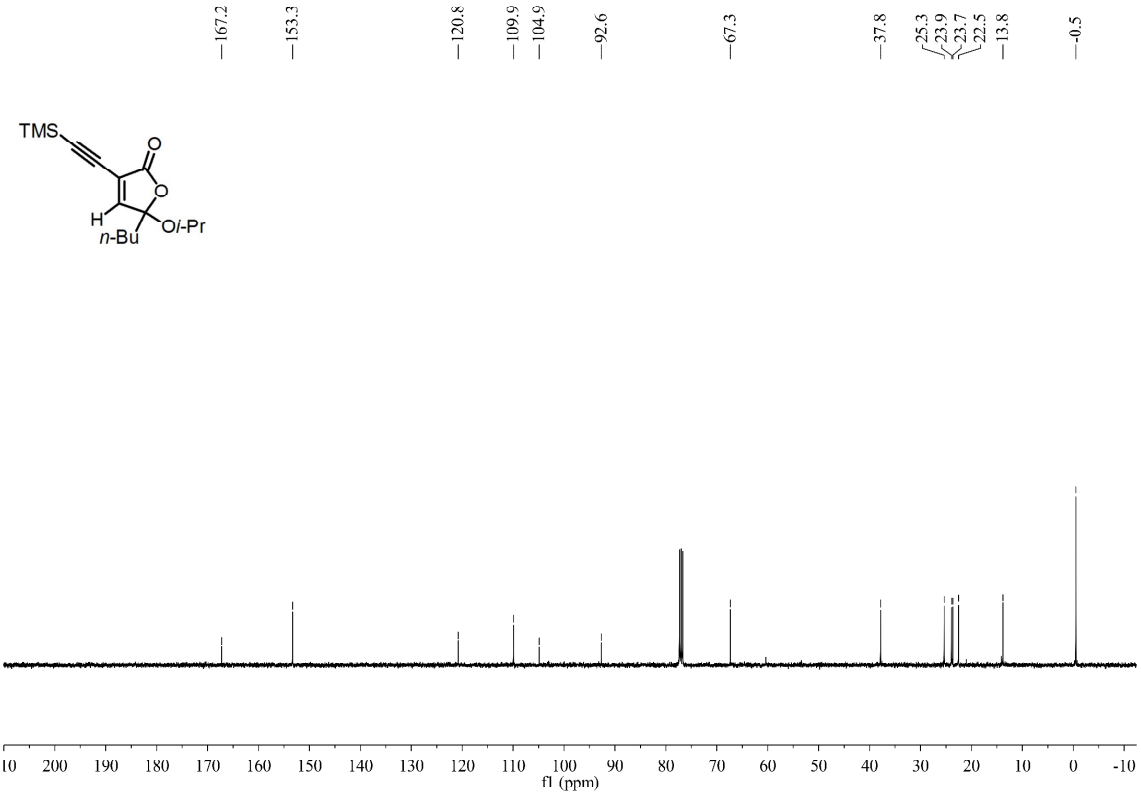
3.76
3.75
3.73
3.72
3.70
3.69
3.67
1.92
1.90
1.88
1.36
1.21
1.19
1.16
1.15
0.93
0.29
TMS
O
O
H
n-Bu
Oi-Pr
1.01
1.00
1.94
4.85
2.96
2.85
3.12
9.36
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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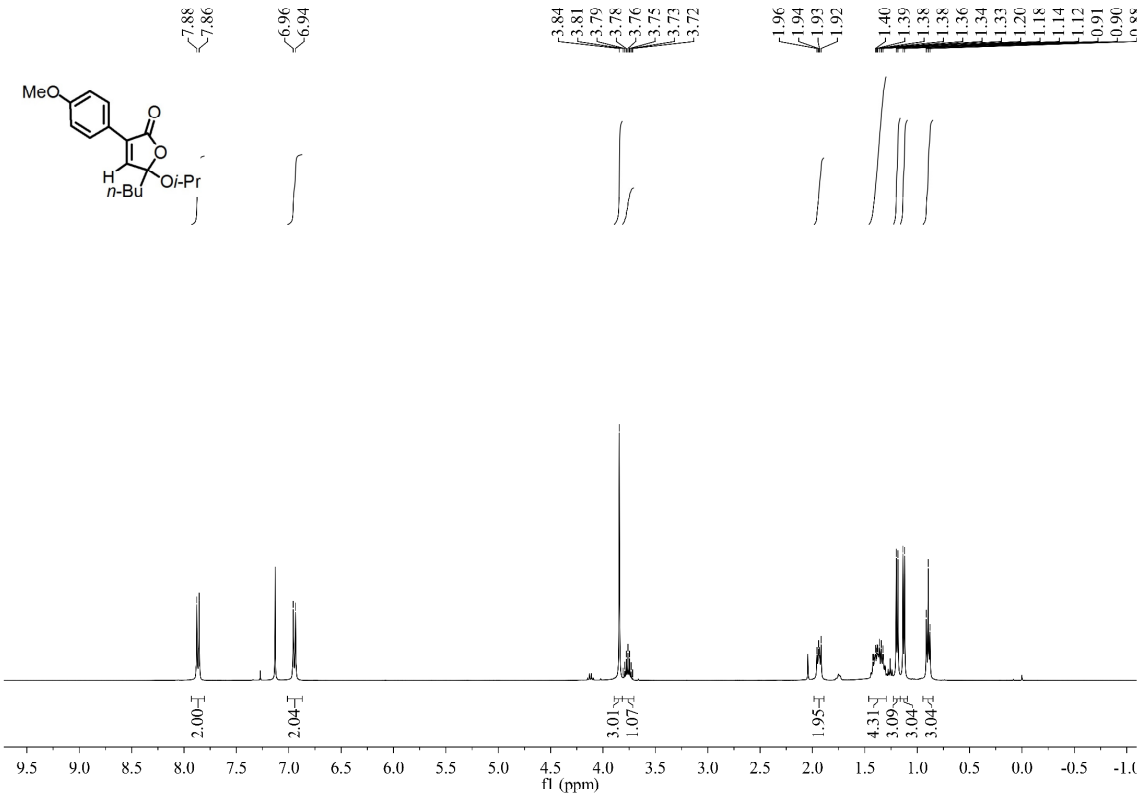
153.3
120.8
109.9
104.9
92.6
67.3
37.8
25.3
23.9
23.7
22.5
13.8
-0.5
TMS
O
O
H
n-Bu
Oi-Pr
f1 (ppm)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3.81
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3.78
3.76
3.75
3.73
3.72
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1.94
1.93
1.92
1.40
1.39
1.38
1.36
1.34
1.33
1.20
1.18
1.14
1.12
0.91
MeO
O
O
H
n-Bu
Oi-Pr
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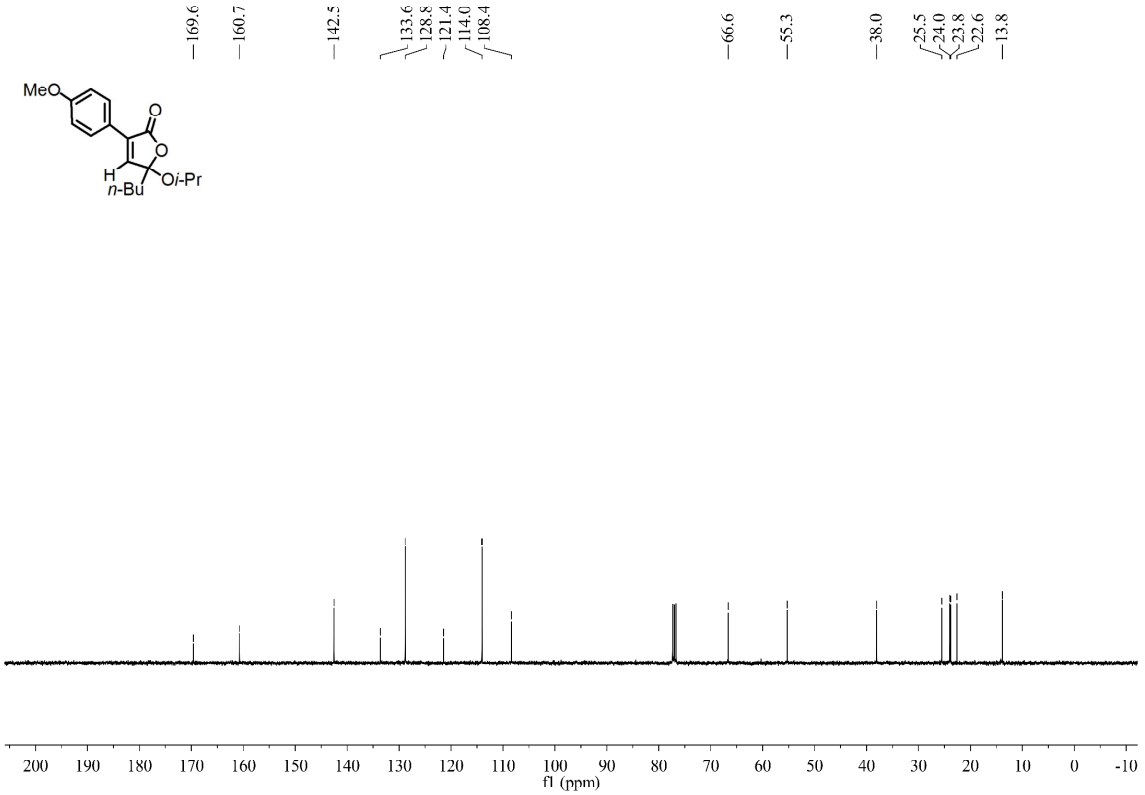
1.07
1.95
4.31
3.09
3.04
3.04
f1 (ppm)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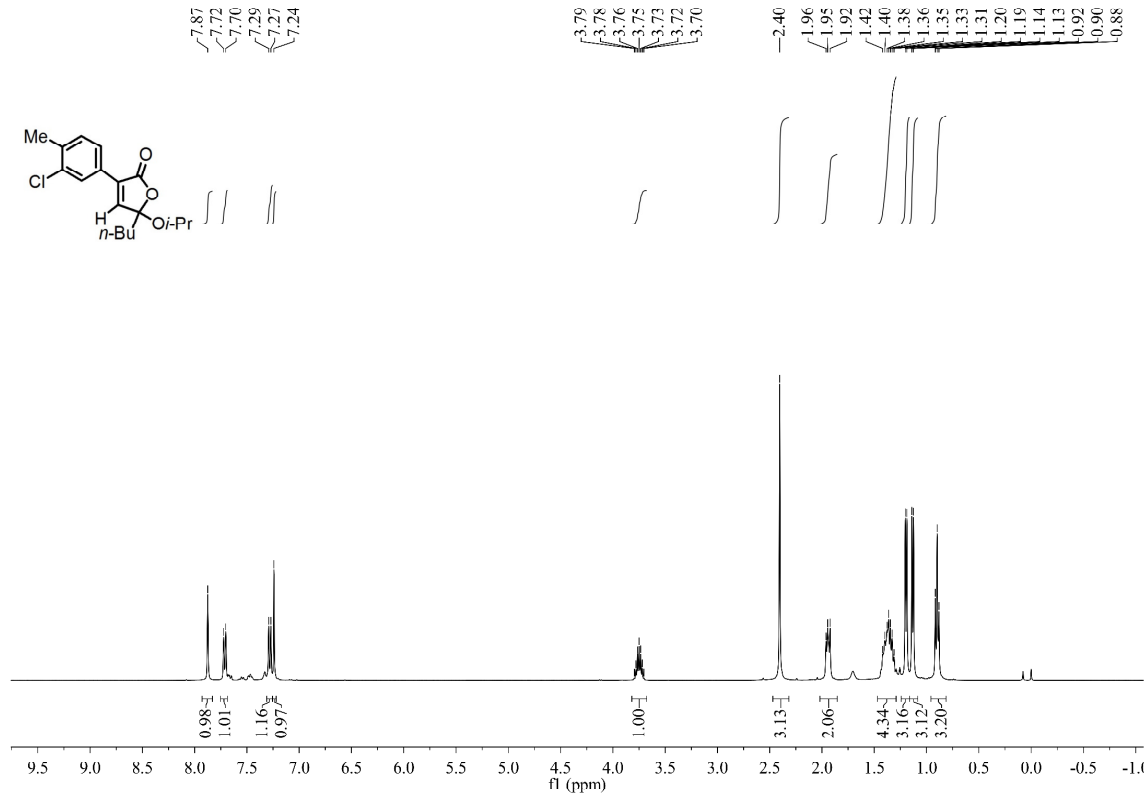
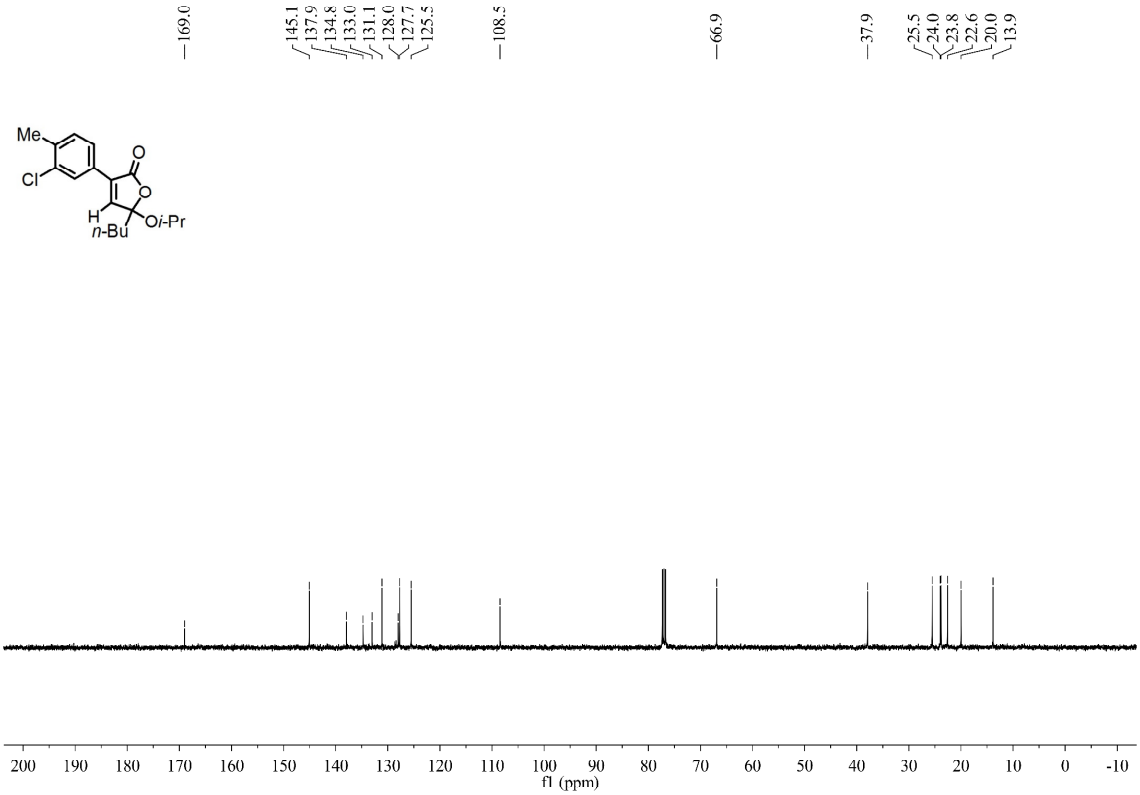
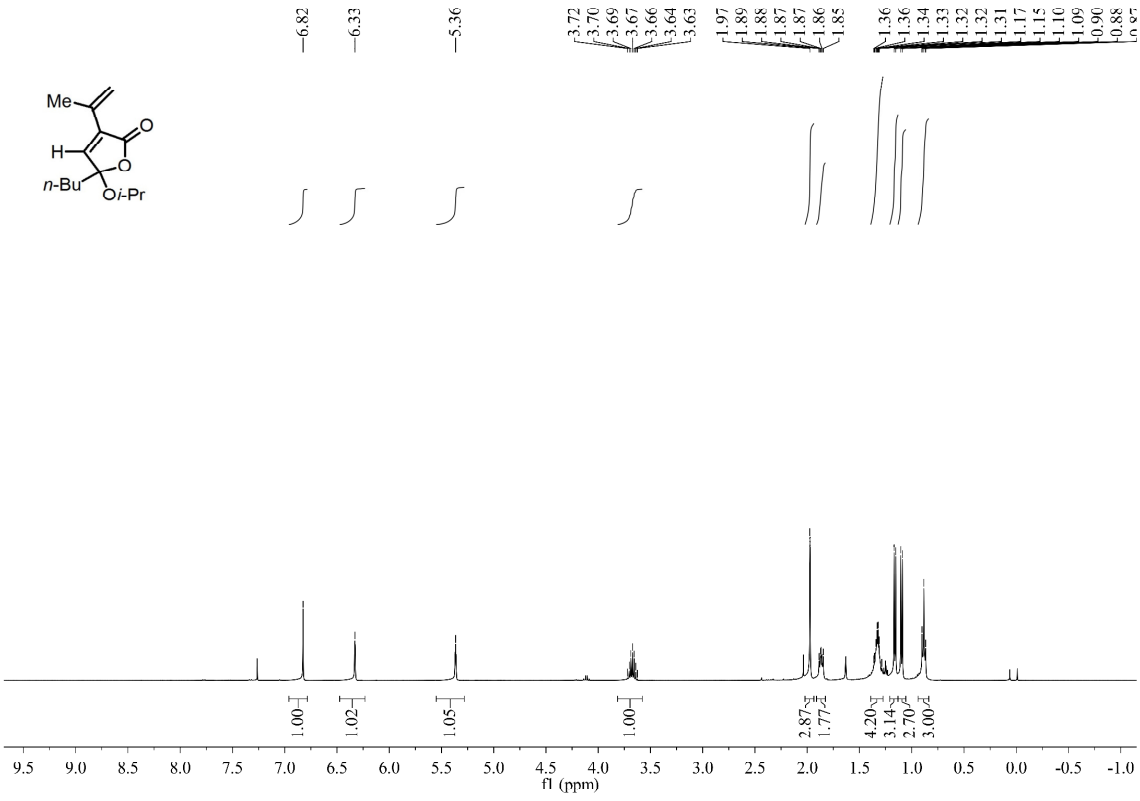
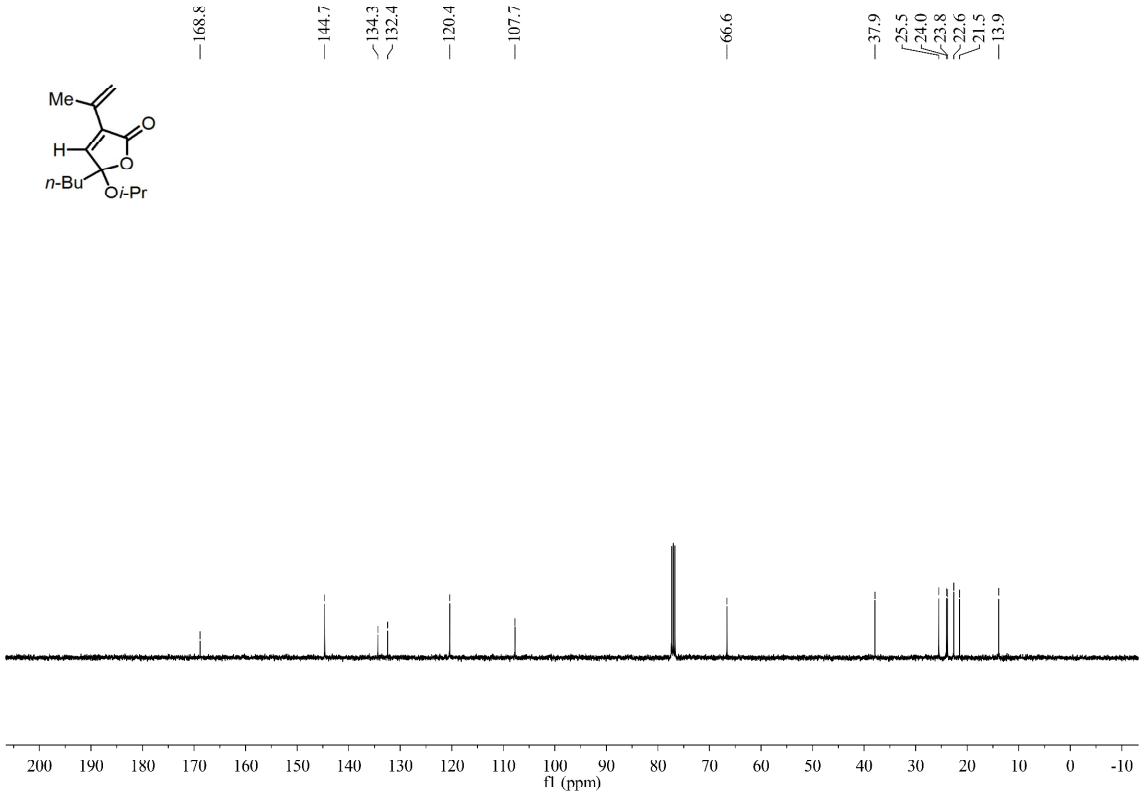
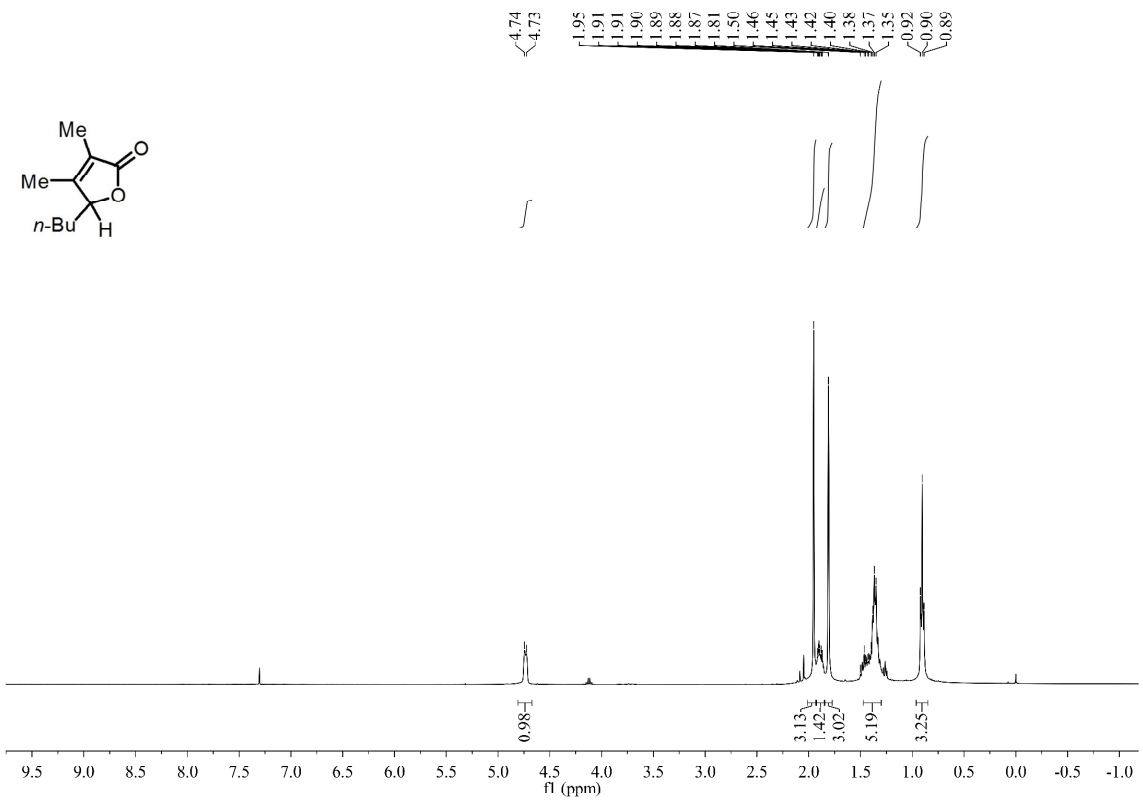
142.5
133.6
128.8
121.4
114.0
108.4
66.6
55.3
38.0
25.5
24.0
23.8
22.6
13.8
MeO
O
O
H
n-Bu
Oi-Pr
f1 (ppm)

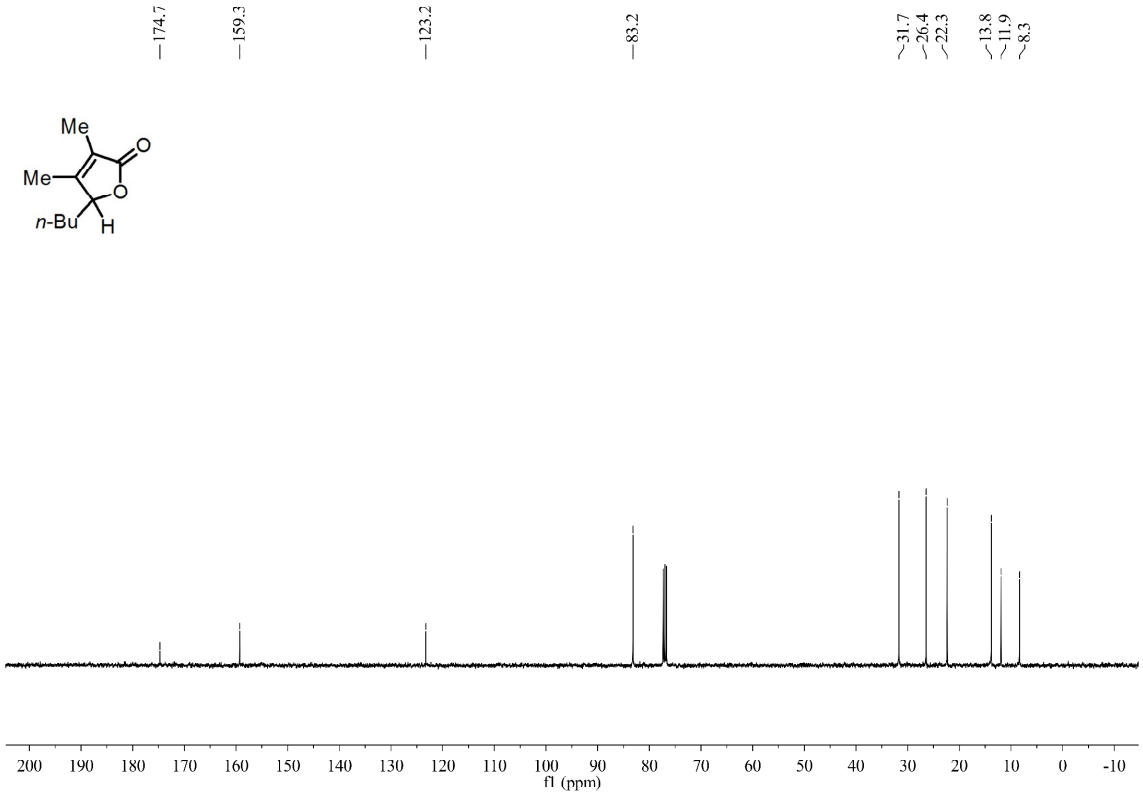

7.87
7.72
7.70
7.29
7.27
7.24
3.79
3.78
3.76
3.75
3.73
3.72
3.70
2.40
1.96
1.95
1.92
1.42
1.40
1.38
1.36
1.35
1.33
1.31
1.20
1.19
1.14
1.13
0.92
0.90
0.88
Me
Cl
O
O
H
n-Bu
Oi-Pr
0.98
1.01
1.16
0.97
1.00
3.13
2.06
4.34
3.16
3.12
3.20
f1 (ppm)

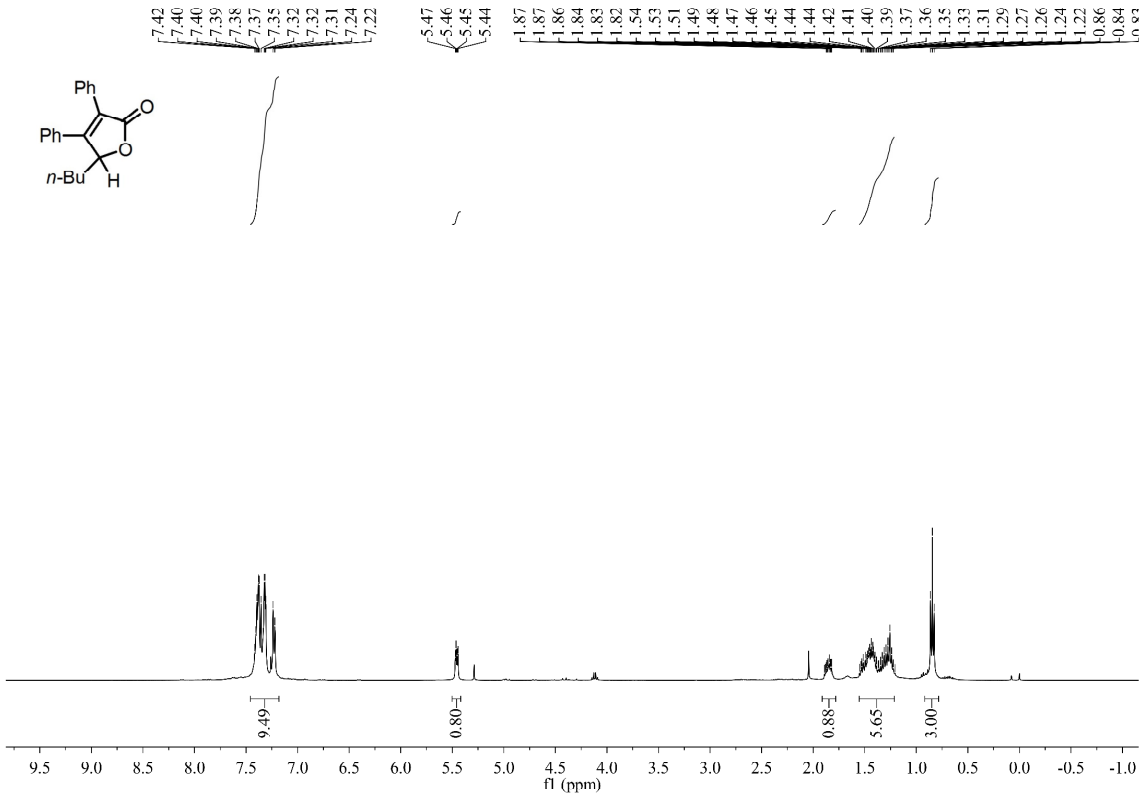

169.0
145.1
137.9
134.8
133.0
131.1
128.0
127.7
125.5
108.5
66.9
37.9
25.5
24.0
23.8
22.6
20.0
13.9
Me
Cl
O
O
H
n-Bu
Oi-Pr
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


6.82
6.33
5.36
3.72
3.70
3.69
3.67
3.66
3.64
3.63
1.97
1.89
1.88
1.87
1.87
1.86
1.85
1.36
1.36
1.34
1.33
1.32
1.32
1.31
1.17
1.15
1.10
1.09
0.90
0.88
Me
H
O
O
n-Bu
Oi-Pr
1.00
1.02
1.05
1.00
2.87
1.77
4.20
3.14
2.70
3.00
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 0.0 -0.5 -1.0
f1 (ppm)


168.8
144.7
134.3
132.4
120.4
107.7
66.6
37.9
25.5
24.0
23.8
22.6
21.5
13.9
Me
H
O
O
n-Bu
Oi-Pr
f1 (ppm)


4.74
4.73
1.95
1.91
1.91
1.90
1.89
1.88
1.87
1.81
1.50
1.46
1.45
1.43
1.42
1.40
1.38
1.37
1.35
0.92
0.90
0.89
Me
Me
O
O
n-Bu
H
0.98
3.13
1.42
3.02
5.19
3.25
f1 (ppm)


174.7
159.3
123.2
83.2
31.7
26.4
22.3
13.8
11.9
8.3
Me
Me
O
O
n-Bu
H
f1 (ppm)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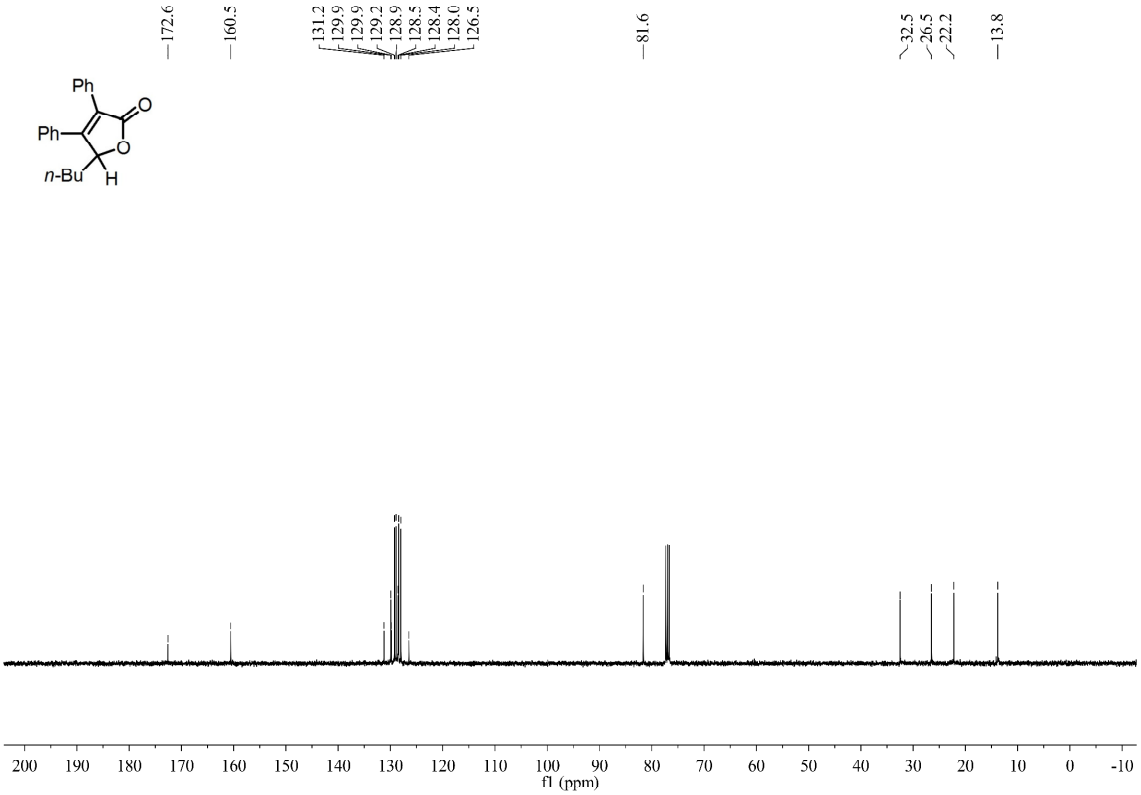
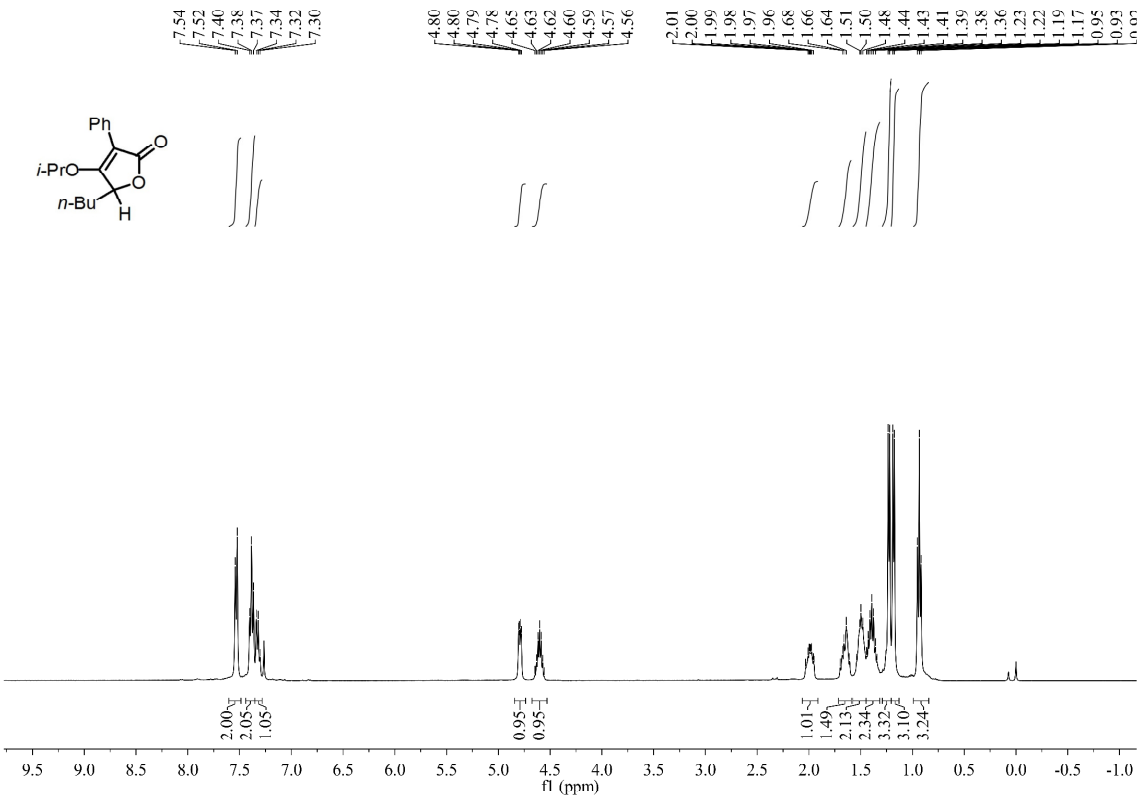
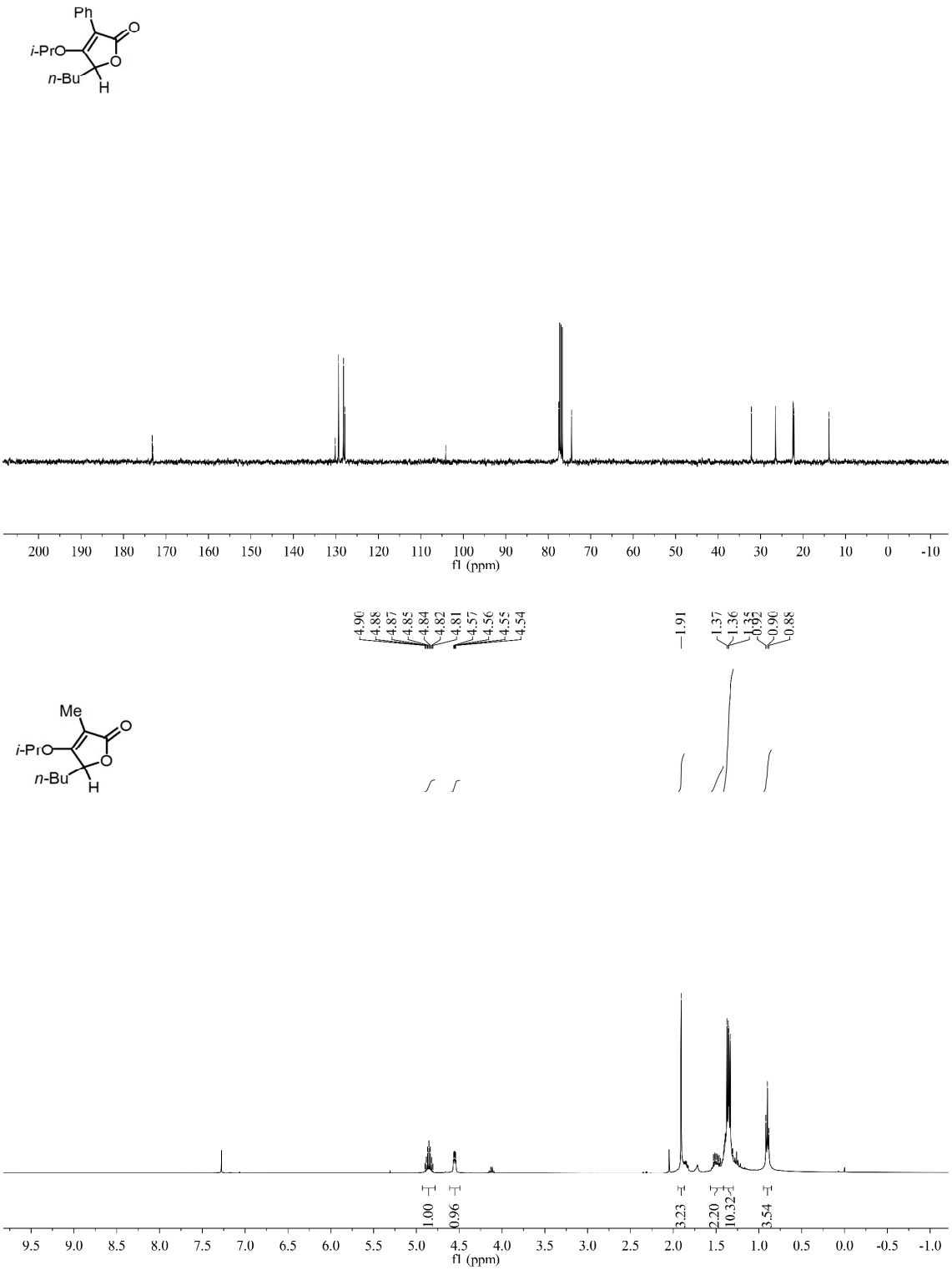
7.40
7.40
7.39
7.38
7.37
7.35
7.32
7.32
7.31
7.24
7.22
5.47
5.46
5.45
5.44
1.87
1.87
1.86
1.84
1.83
1.82
1.54
1.53
1.51
1.49
1.48
1.47
1.46
1.45
1.44
1.44
1.42
1.41
1.40
1.39
1.37
1.36
1.35
1.33
1.31
1.29
1.27
1.26
1.24
1.22
0.86
0.84
Ph
Ph
O
O
n-Bu
H
9.49
0.80
0.88
5.65
3.00
f1 (ppm)


Ph
Ph
O
O
n-Bu
H
172.6
160.5
131.2
129.9
129.9
129.2
128.9
128.5
128.4
128.0
126.5
81.6
32.5
26.5
22.2
13.8
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Ph
i-PrO
O
O
n-Bu
H
7.54
7.52
7.40
7.38
7.37
7.34
7.32
7.30
4.80
4.80
4.79
4.78
4.65
4.63
4.62
4.60
4.59
4.57
4.56
2.01
2.00
1.99
1.98
1.97
1.96
1.68
1.66
1.64
1.51
1.50
1.48
1.44
1.43
1.41
1.39
1.38
1.36
1.23
1.22
1.19
1.17
0.95
0.93
2.00
2.05
1.05
0.95
0.95
1.01
1.49
2.13
2.34
3.32
3.10
3.24
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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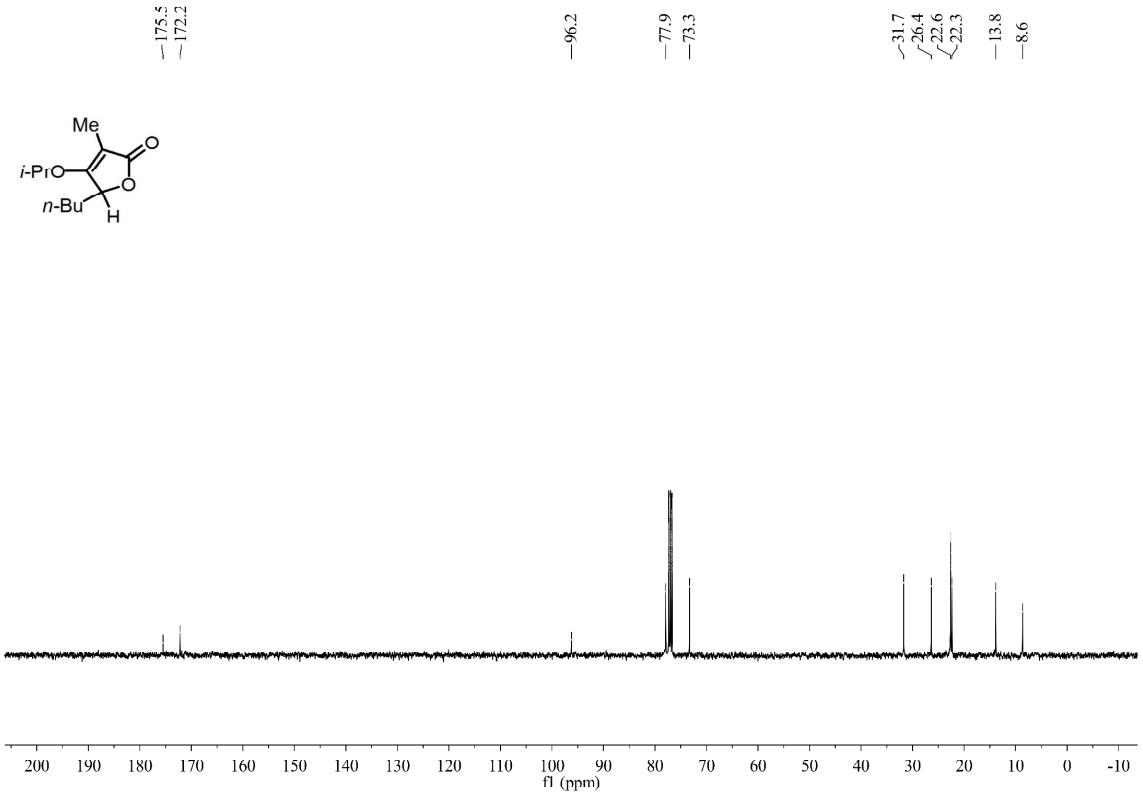
130.2
129.4
128.2
127.9
104.1
77.5
74.5
32.2
26.5
22.4
22.3
22.1
13.9
Ph
i-PrO
n-Bu
H
O
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
4.90
4.88
4.87
4.85
4.84
4.82
4.81
4.57
4.56
4.55
4.54
1.91
1.37
1.36
1.35
0.92
0.90
0.88
Me
i-PrO
n-Bu
H
O
O
1.00
0.96
3.23
2.20
10.32
3.54
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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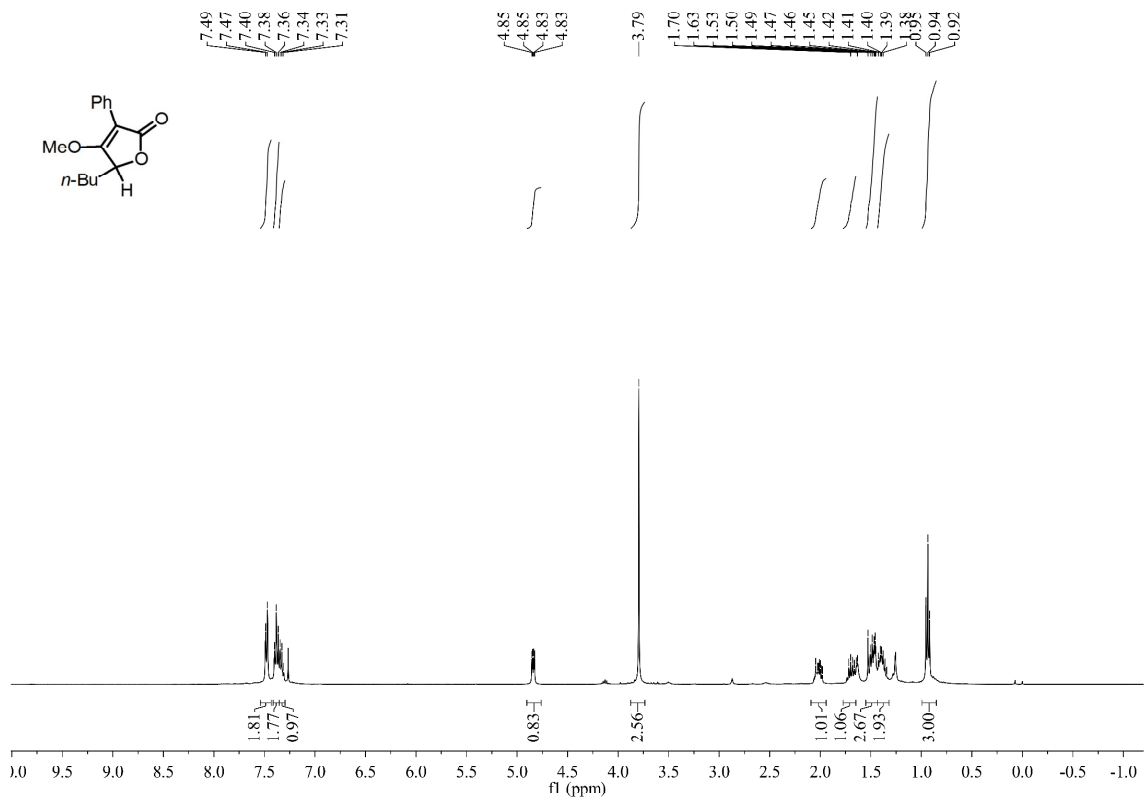
172.2
96.2
77.9
73.3
31.7
26.4
22.6
22.3
13.8
8.6
Me
i-PrO
n-Bu
H
O
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.34
7.33
7.31
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4.85
4.83
4.83
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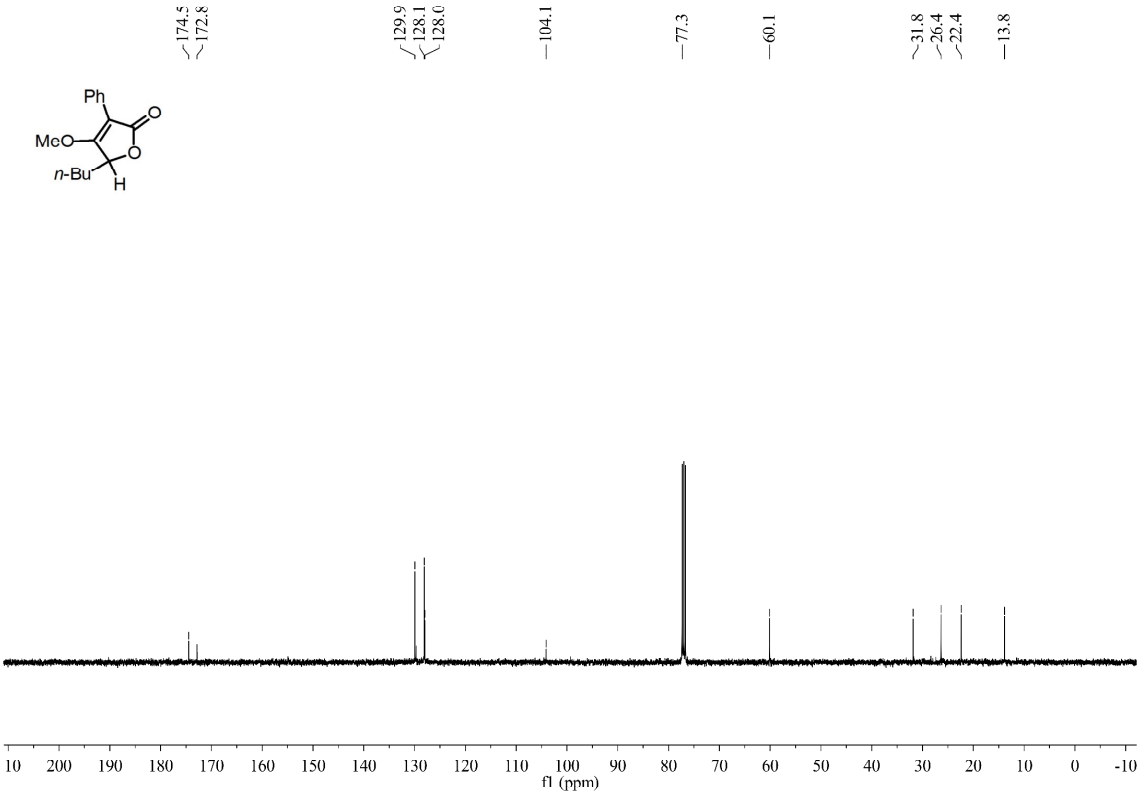
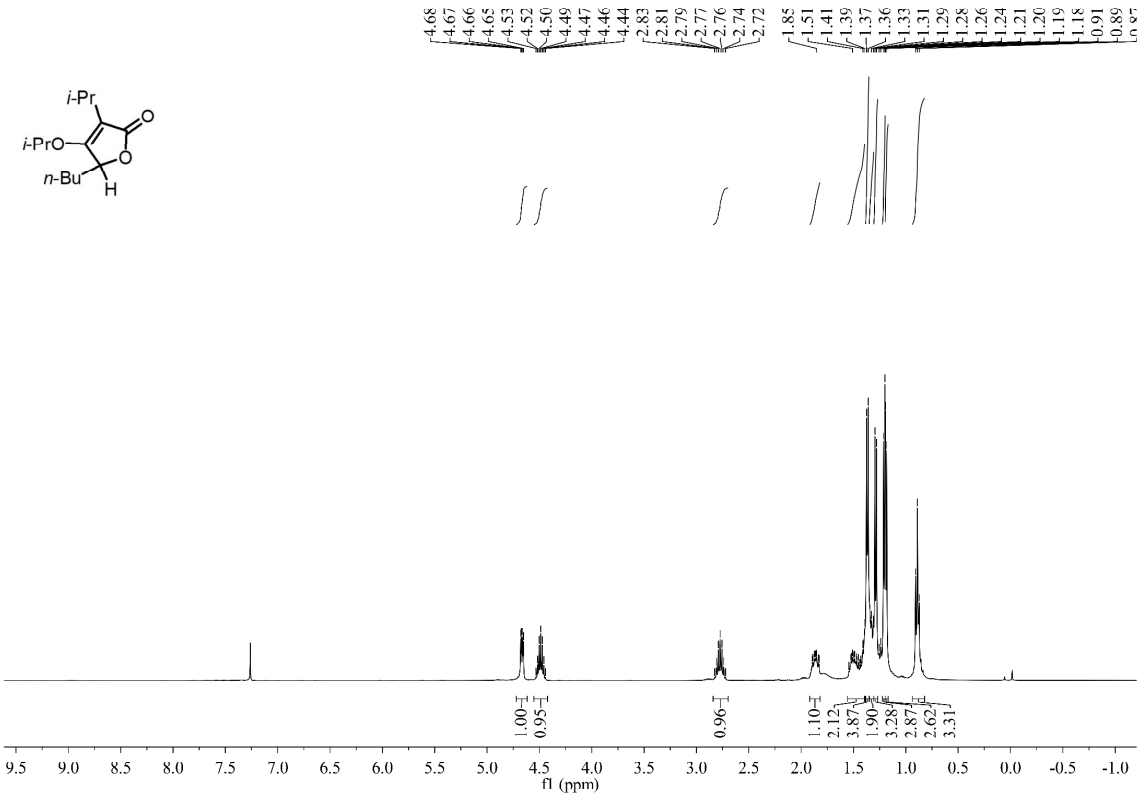
1.70
1.63
1.53
1.50
1.49
1.47
1.46
1.45
1.42
1.41
1.40
1.39
0.94
0.92
Ph
MeO
n-Bu
H
O
O
1.81
1.77
0.97
0.83
2.56
1.01
1.06
2.67
1.93
3.00
f1 (ppm)


174.5
172.8
129.9
128.1
128.0
104.1
77.3
60.1
31.8
26.4
22.4
13.8
Ph
MeO
n-Bu
H
O
f1 (ppm)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4.67
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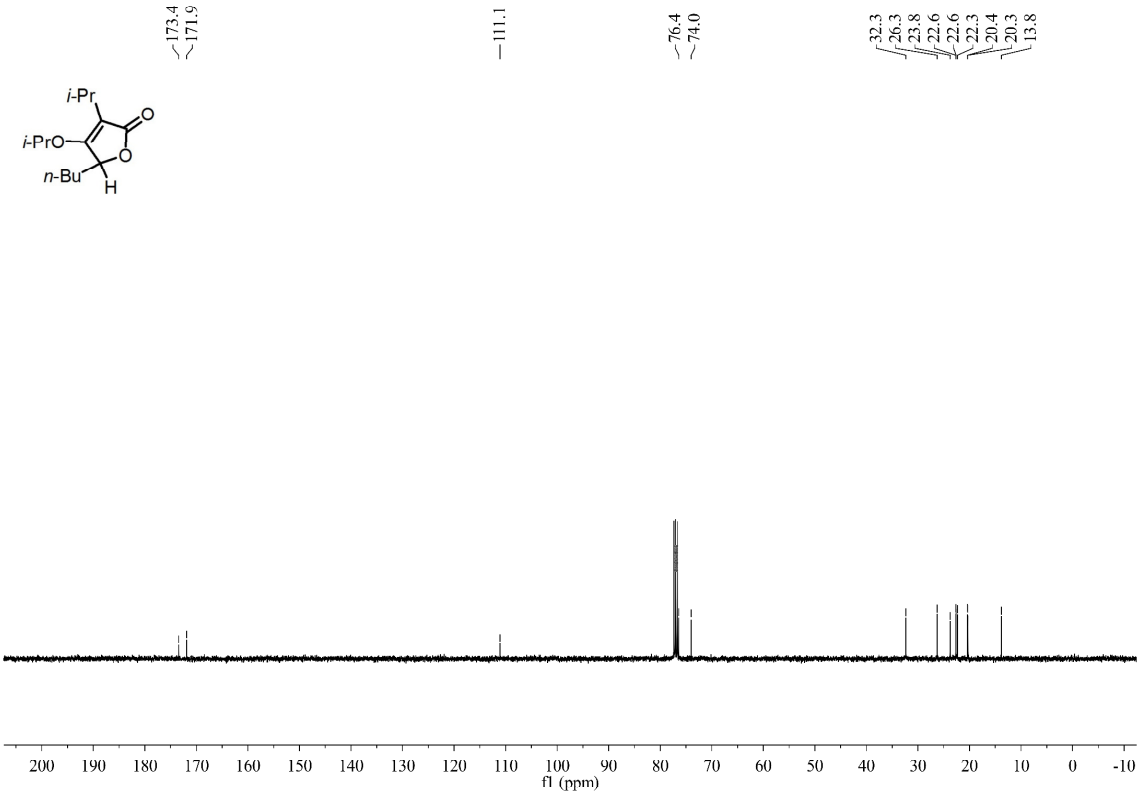
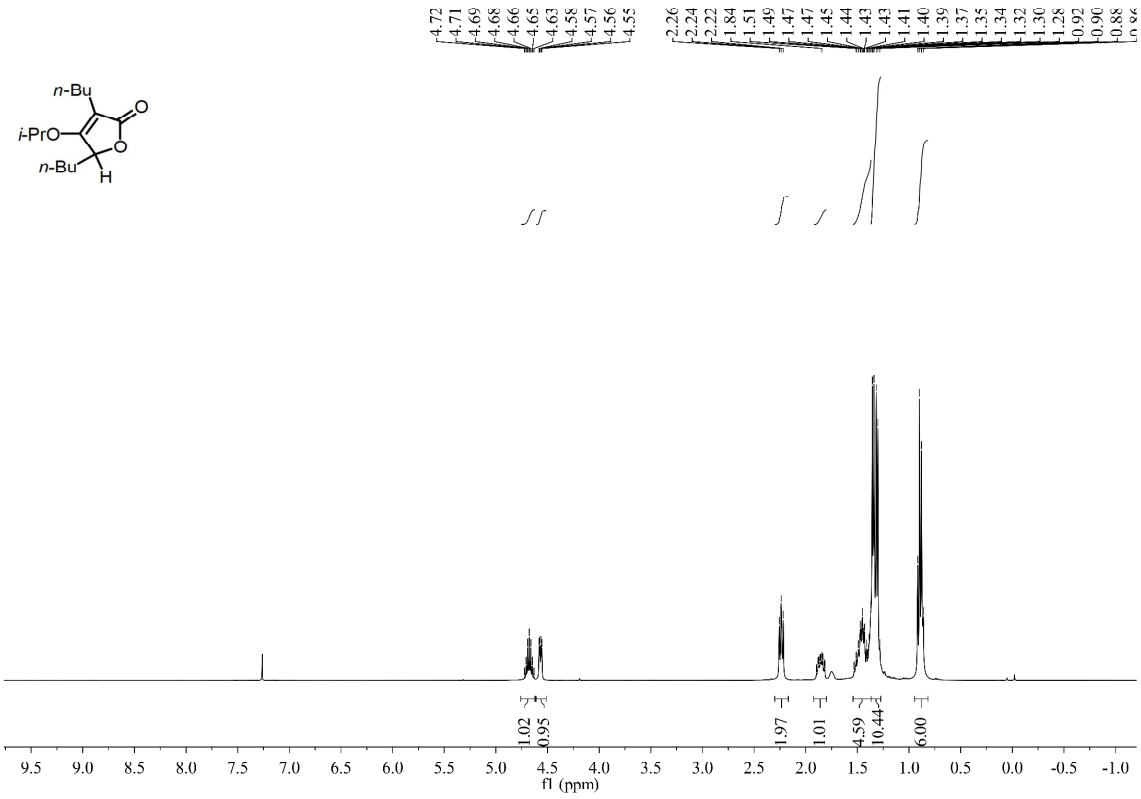
4.53
4.52
4.50
4.49
4.47
4.46
4.44
2.83
2.81
2.79
2.77
2.76
2.74
2.72
1.85
1.51
1.41
1.39
1.37
1.36
1.33
1.31
1.29
1.28
1.26
1.24
1.21
1.20
1.19
1.18
0.91
0.89
i-Pr
i-PrO
n-Bu
H
O
1.00
0.95
0.96
1.10
2.12
3.87
1.90
3.28
2.87
2.62
3.31
f1 (ppm)


173.4
171.9
111.1
76.4
74.0
32.3
26.3
23.8
22.6
22.6
22.3
20.4
20.3
13.8
i-Pr
i-PrO
n-Bu
H
O
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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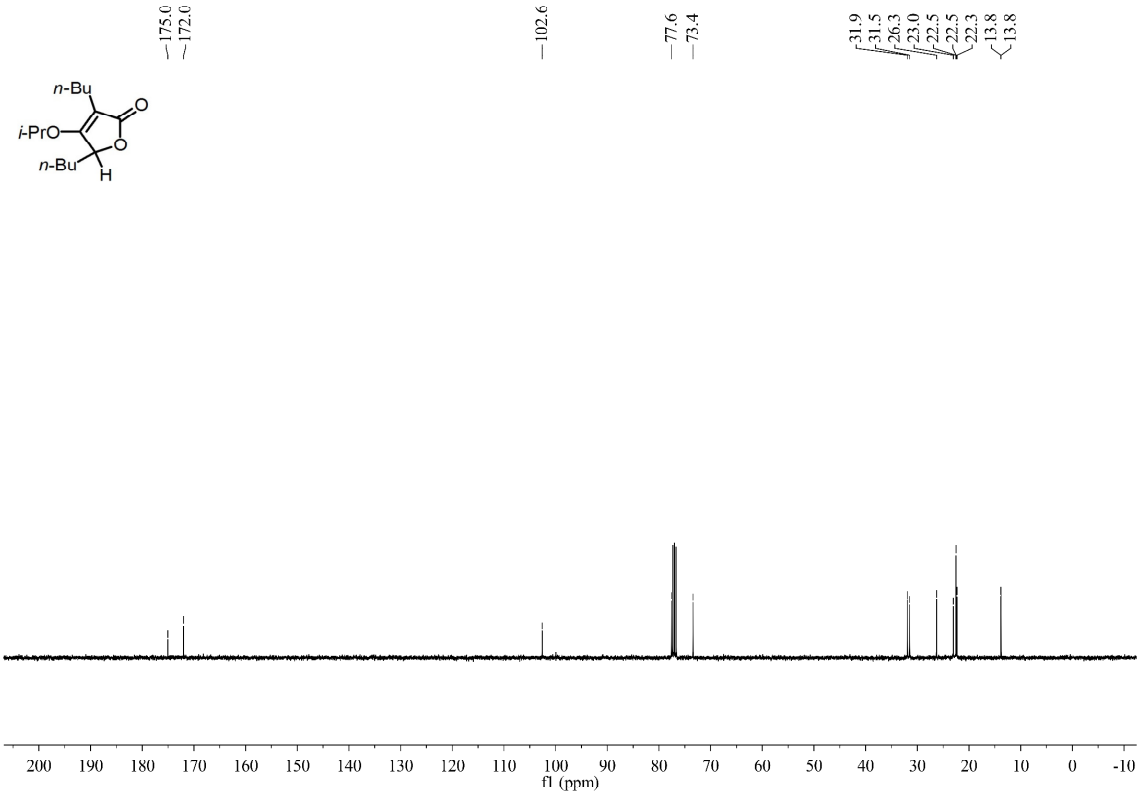
4.65
4.63
4.58
4.57
4.56
4.55
2.26
2.24
2.22
1.84
1.51
1.49
1.47
1.47
1.45
1.44
1.43
1.43
1.41
1.40
1.39
1.37
1.35
1.34
1.32
1.30
1.28
0.92
0.90
n-Bu
i-PrO
n-Bu
H
O
O
1.02
0.95
1.97
1.01
4.59
10.44
6.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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172.0
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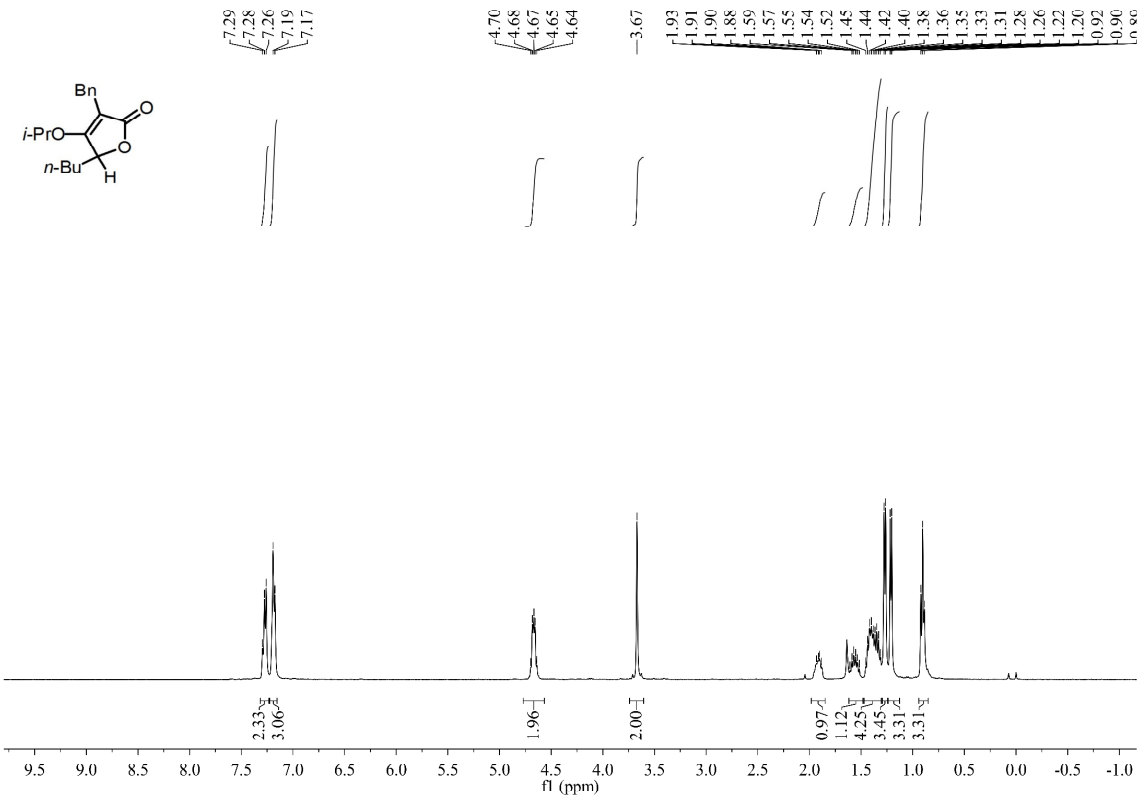
77.6
73.4
31.9
31.5
26.3
23.0
22.5
22.5
22.3
13.8
13.8
n-Bu
i-PrO
n-Bu
H
O
O
fl (ppm)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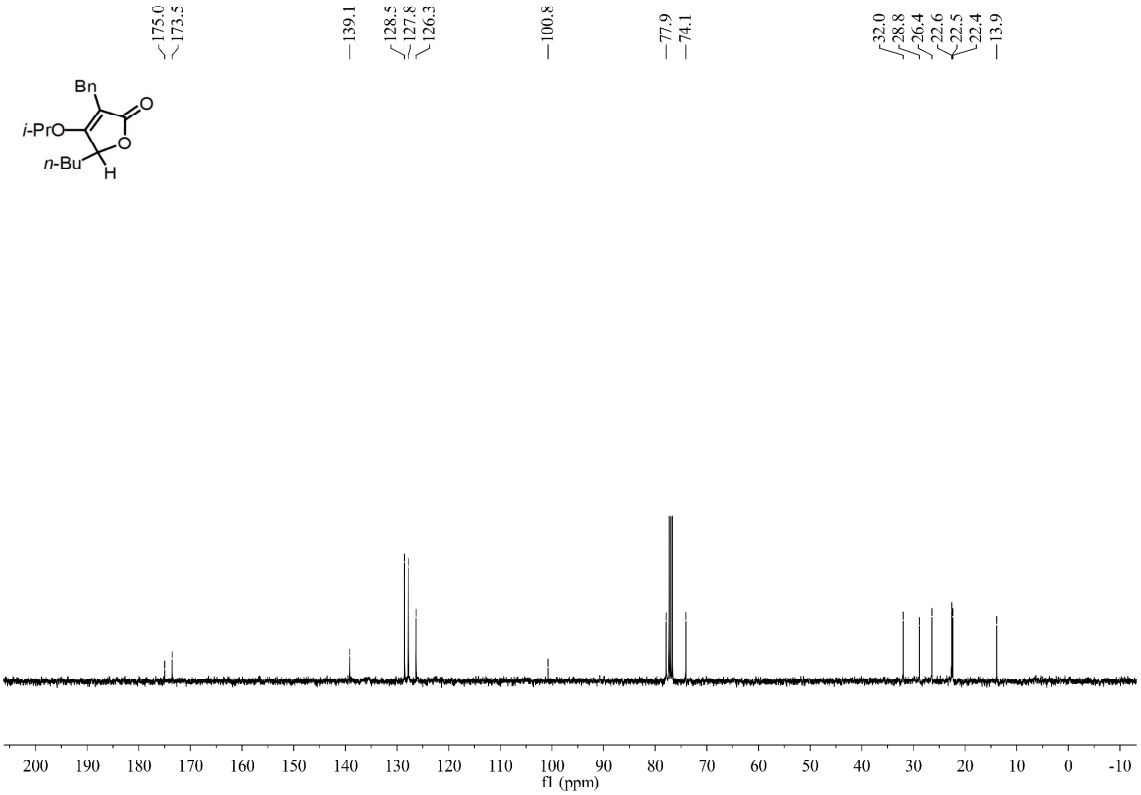
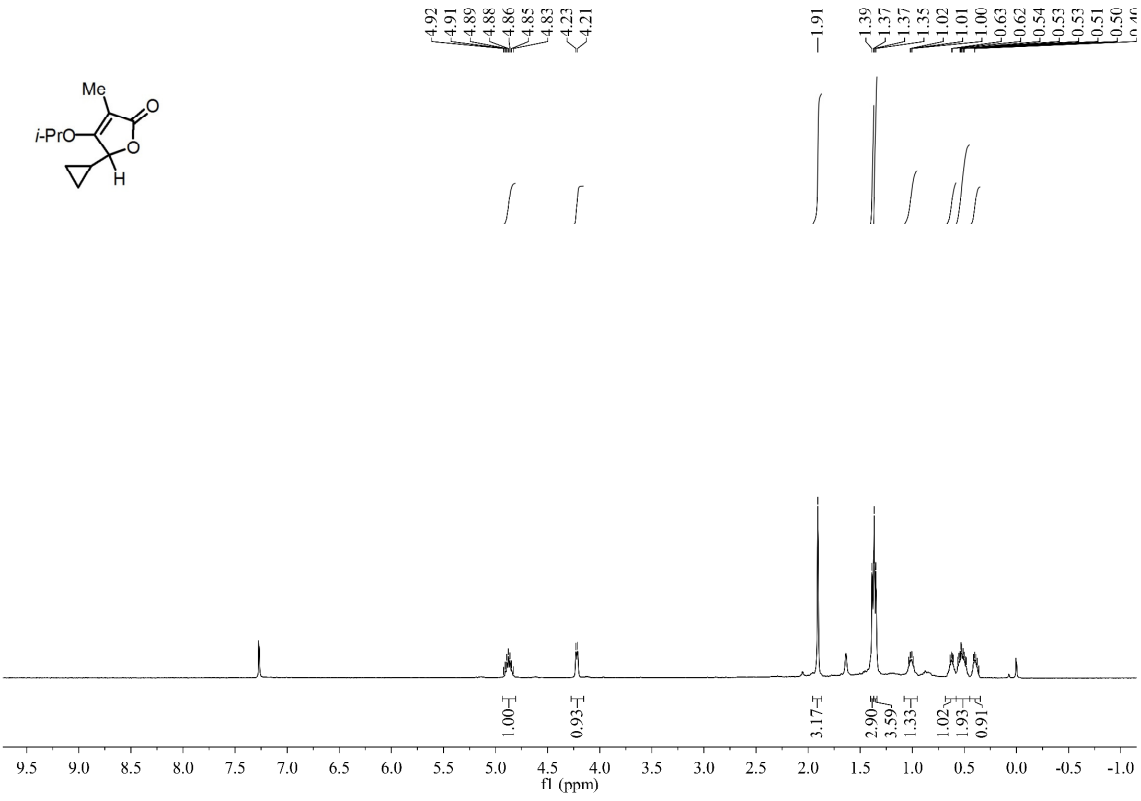
4.70
4.68
4.67
4.65
4.64
3.67
1.93
1.91
1.90
1.88
1.59
1.57
1.55
1.54
1.52
1.45
1.44
1.42
1.40
1.38
1.36
1.35
1.33
1.31
1.28
1.26
1.22
1.20
0.92
0.90
Bn
i-PrO
n-Bu
H
O
O
2.33
3.06
1.96
2.00
0.97
1.12
4.25
3.45
3.31
3.31
fl (ppm)


175.0
173.5
139.1
128.5
127.8
126.3
100.8
77.9
74.1
32.0
28.8
26.4
22.6
22.5
22.4
13.9
Bn
i-PrO
n-Bu
H
O
O
fl (ppm)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4.88
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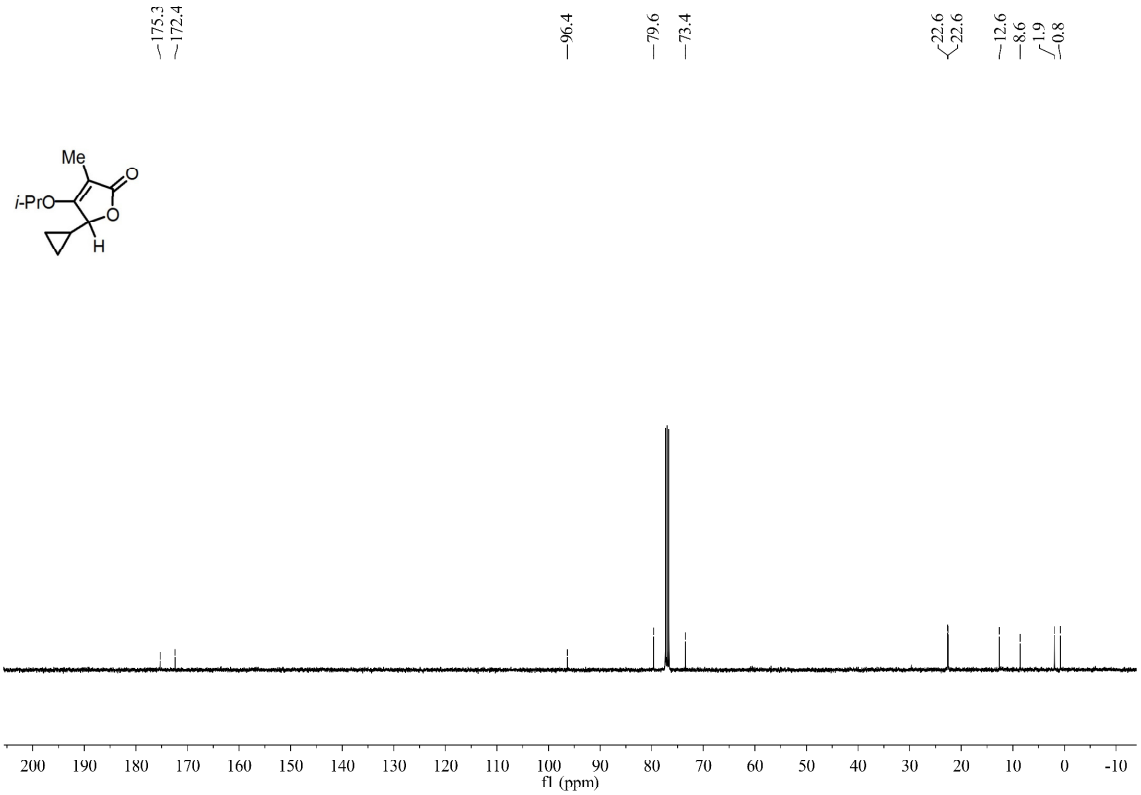
4.85
4.83
4.23
4.21
1.91
1.39
1.37
1.37
1.35
1.02
1.01
1.00
0.63
0.62
0.54
0.53
0.53
0.51
Me
i-PrO
H
O
O
1.00
0.93
3.17
2.90
3.59
1.33
1.02
1.93
0.91
fl (ppm)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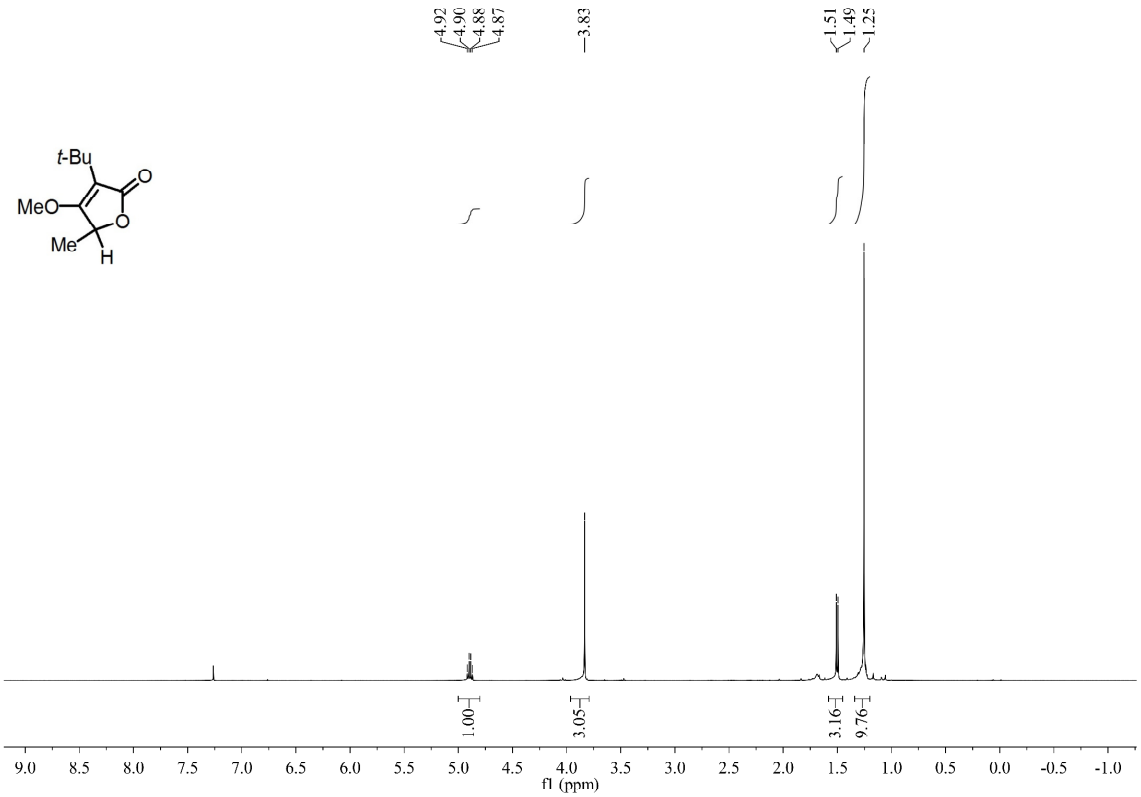
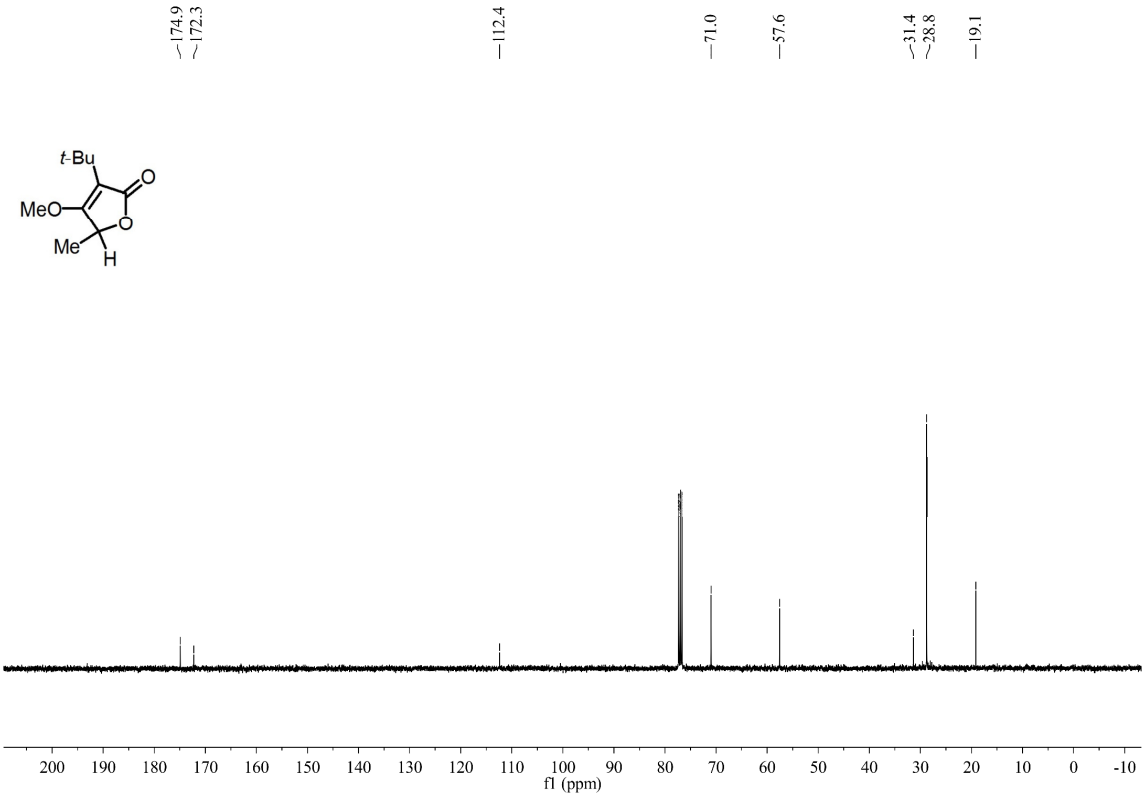
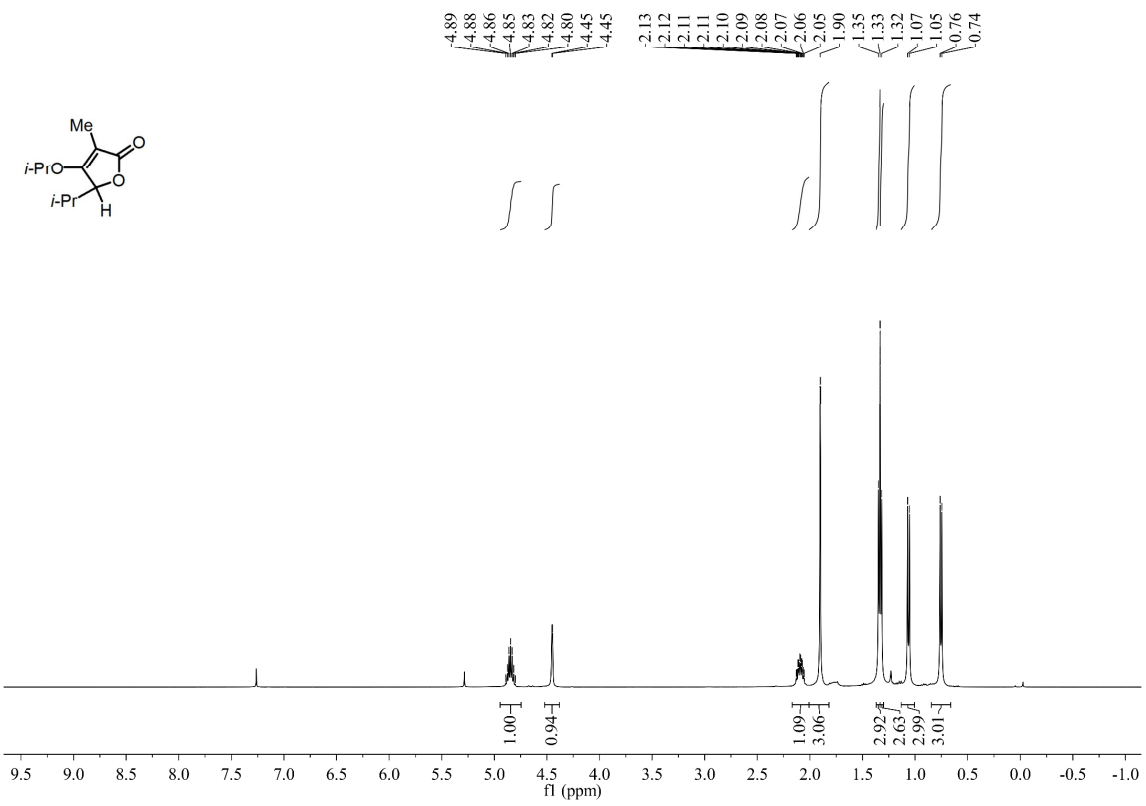
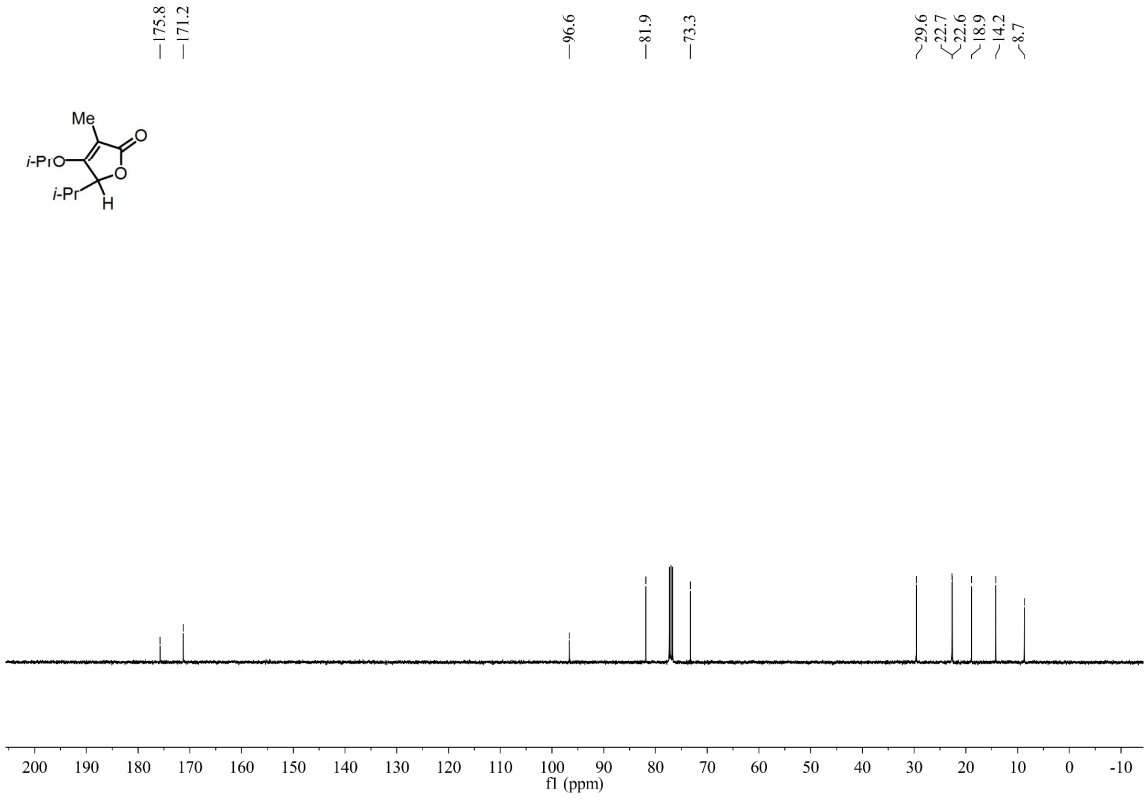
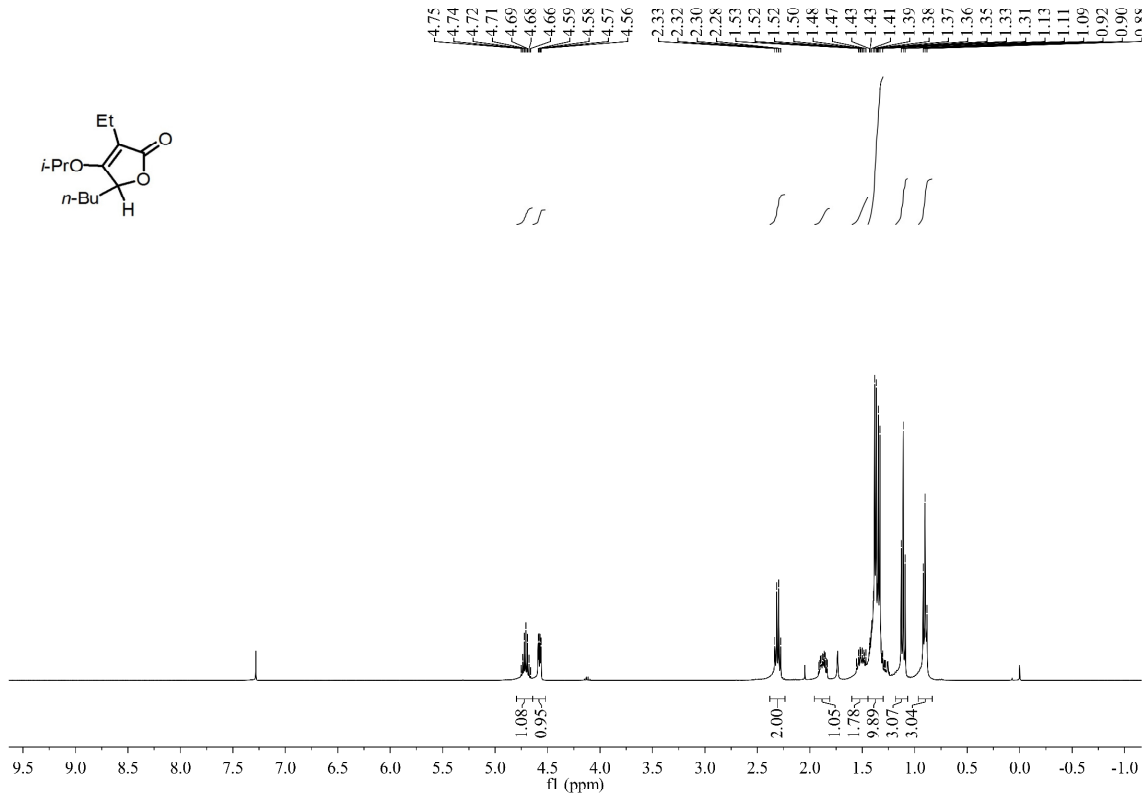
172.4
96.4
79.6
73.4
22.6
22.6
12.6
8.6
1.9
0.8
Me
i-PrO
O
O
H
f1 (ppm)

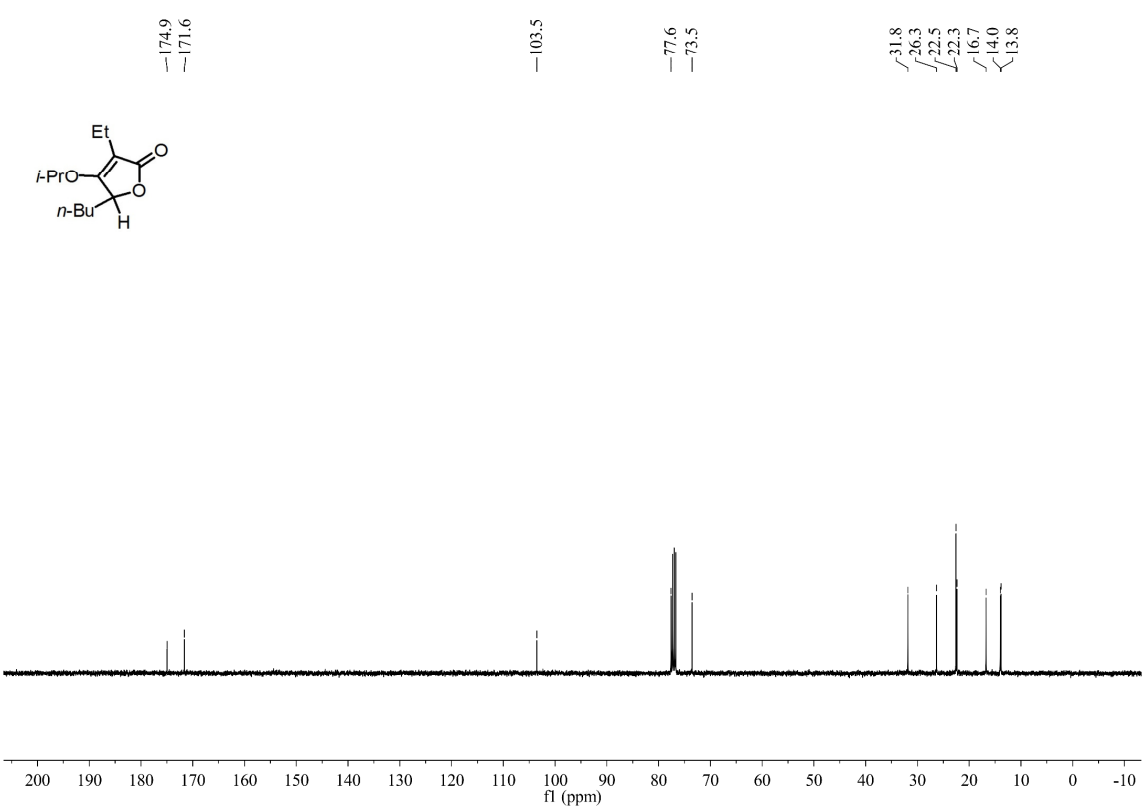

4.92
4.90
4.88
4.87
3.83
1.51
1.49
1.25
t-Bu
MeO
Me
O
O
H
1.00
3.05
3.16
9.76
f1 (ppm)

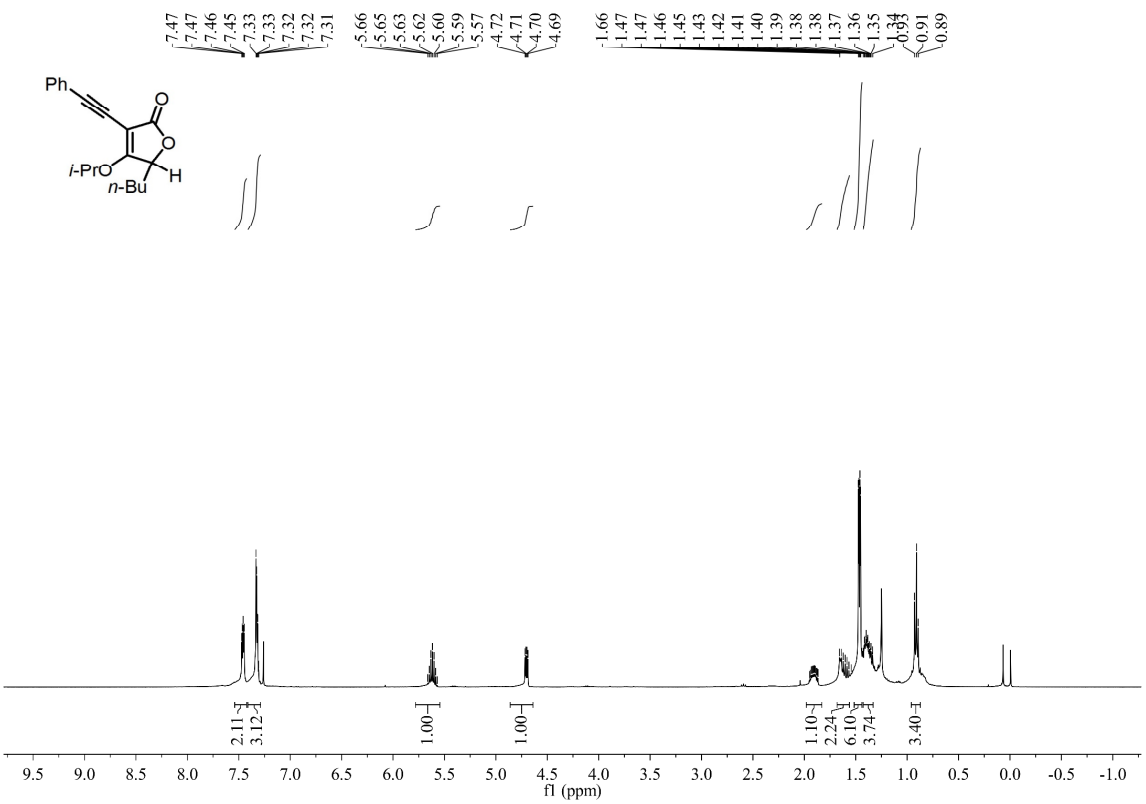

174.9
172.3
112.4
71.0
57.6
31.4
28.8
19.1
t-Bu
MeO
Me
H
O
O
fl (ppm)

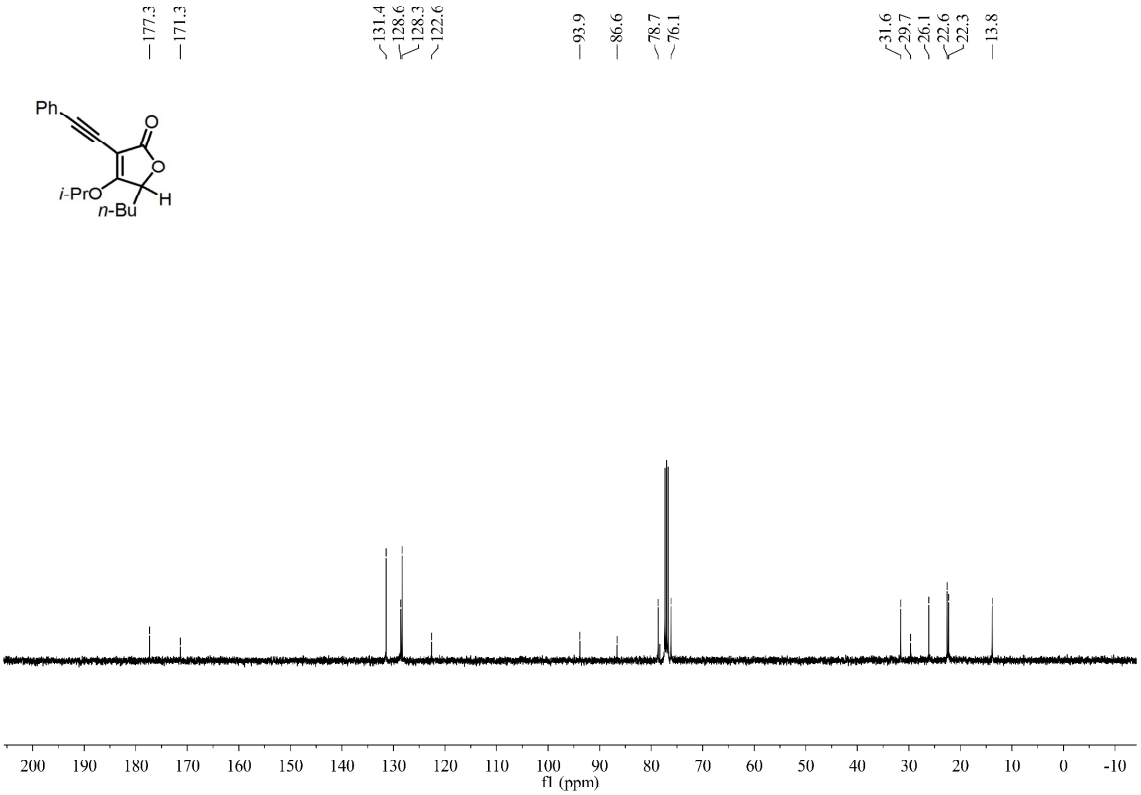

4.89
4.88
4.86
4.85
4.83
4.82
4.80
4.45
4.45
2.13
2.12
2.11
2.11
2.10
2.09
2.08
2.07
2.06
2.05
1.90
1.35
1.33
1.32
1.07
1.05
0.76
0.74
Me
i-PrO
i-Pr
H
O
O
1.00
0.94
1.09
3.06
2.92
2.63
2.99
3.01
fl (ppm)

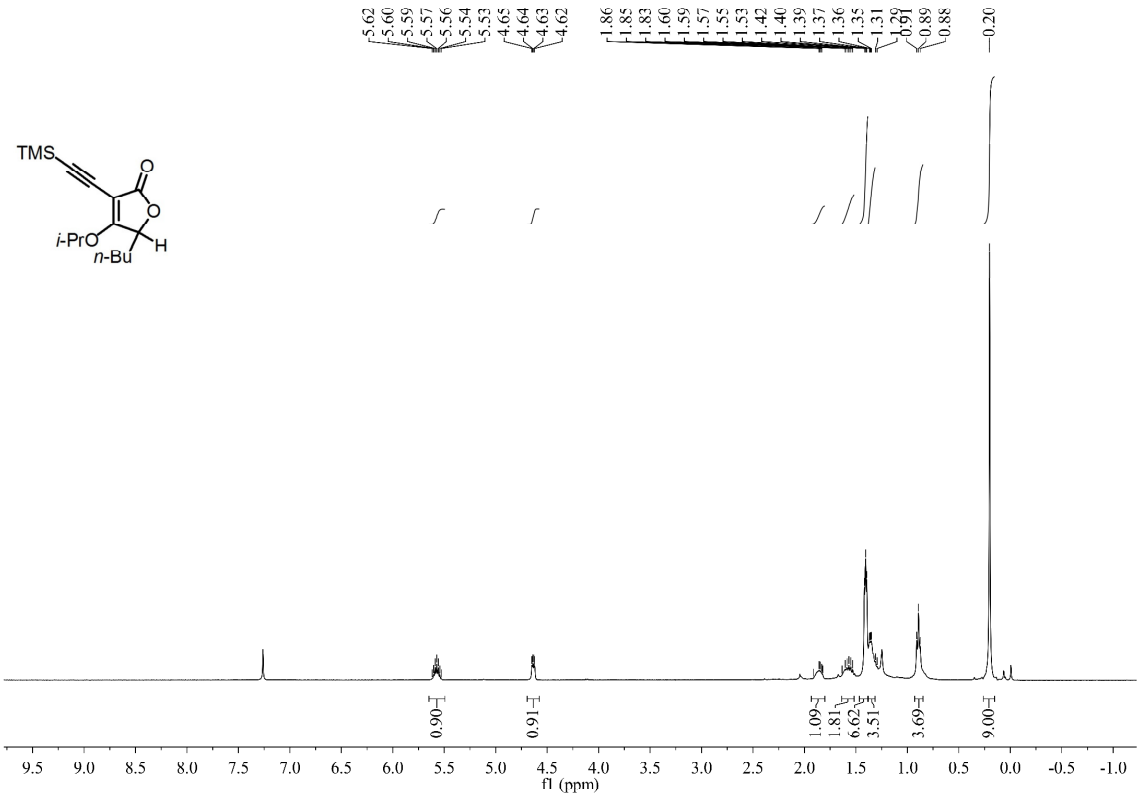

175.8
171.2
96.6
81.9
73.3
29.6
22.7
22.6
18.9
14.2
8.7
Me
i-PrO
i-Pr
H
O
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

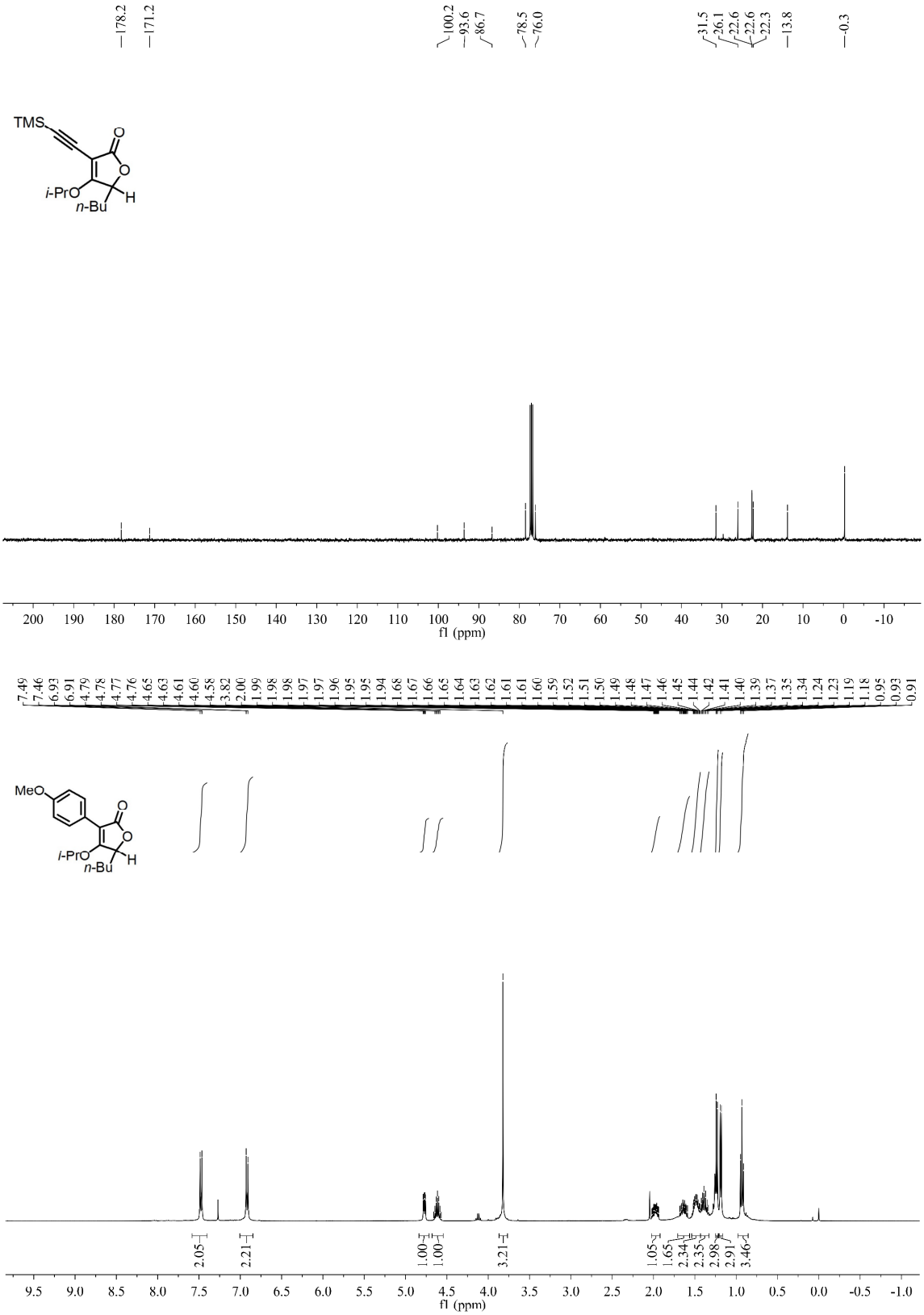

Et
i-PrO
n-Bu
H
O
O
1.08
0.95
2.00
1.05
1.78
9.89
3.07
3.04
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

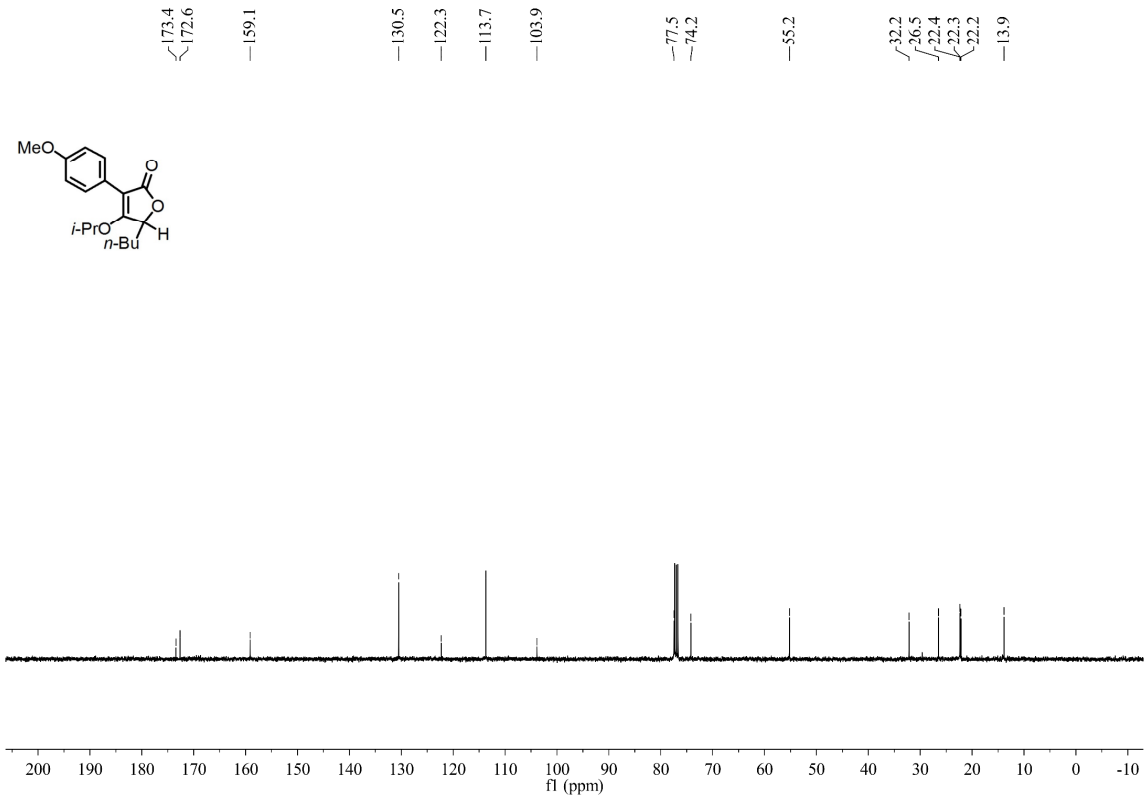

174.9
171.6
103.5
77.6
73.5
31.8
26.3
22.5
22.3
16.7
14.0
13.8
Et
O
i-PrO
O
n-Bu
H
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

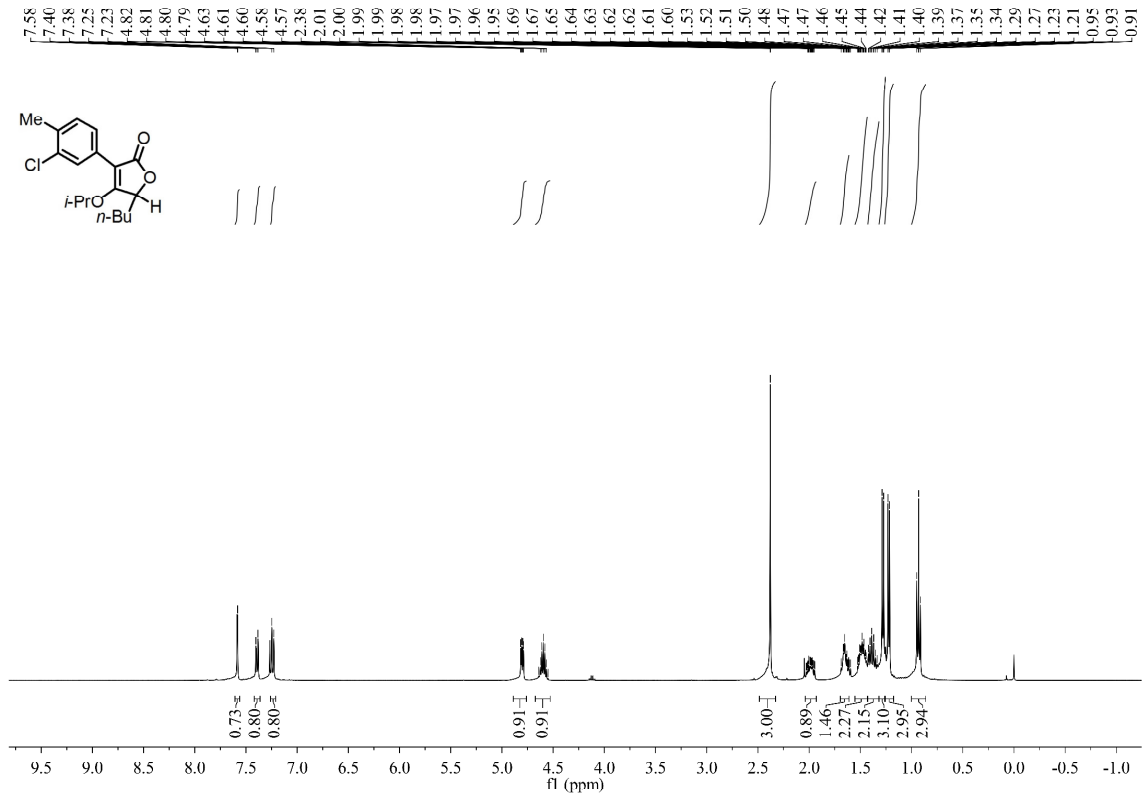

7.47
7.47
7.46
7.45
7.33
7.33
7.32
7.32
7.31
5.66
5.65
5.63
5.62
5.60
5.59
5.57
4.72
4.71
4.70
4.69
1.66
1.47
1.47
1.46
1.45
1.43
1.42
1.41
1.40
1.39
1.38
1.38
1.37
1.36
1.35
0.93
0.91
0.89
Ph
O
O
i-PrO
H
n-Bu
2.11
3.12
1.00
1.00
1.10
2.24
6.10
3.74
3.40
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

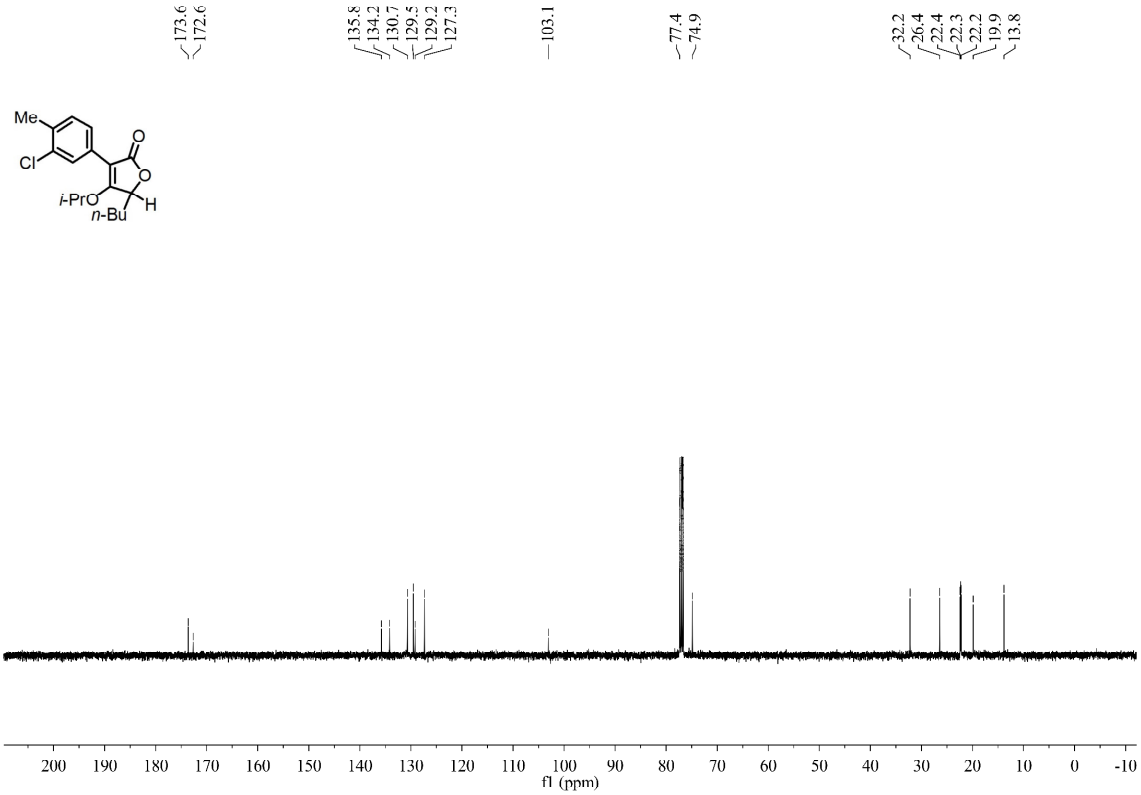

177.3
171.3
131.4
128.6
128.3
122.6
93.9
86.6
78.7
76.1
31.6
29.7
26.1
22.6
22.3
13.8
Ph
O
O
i-PrO
H
n-Bu
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

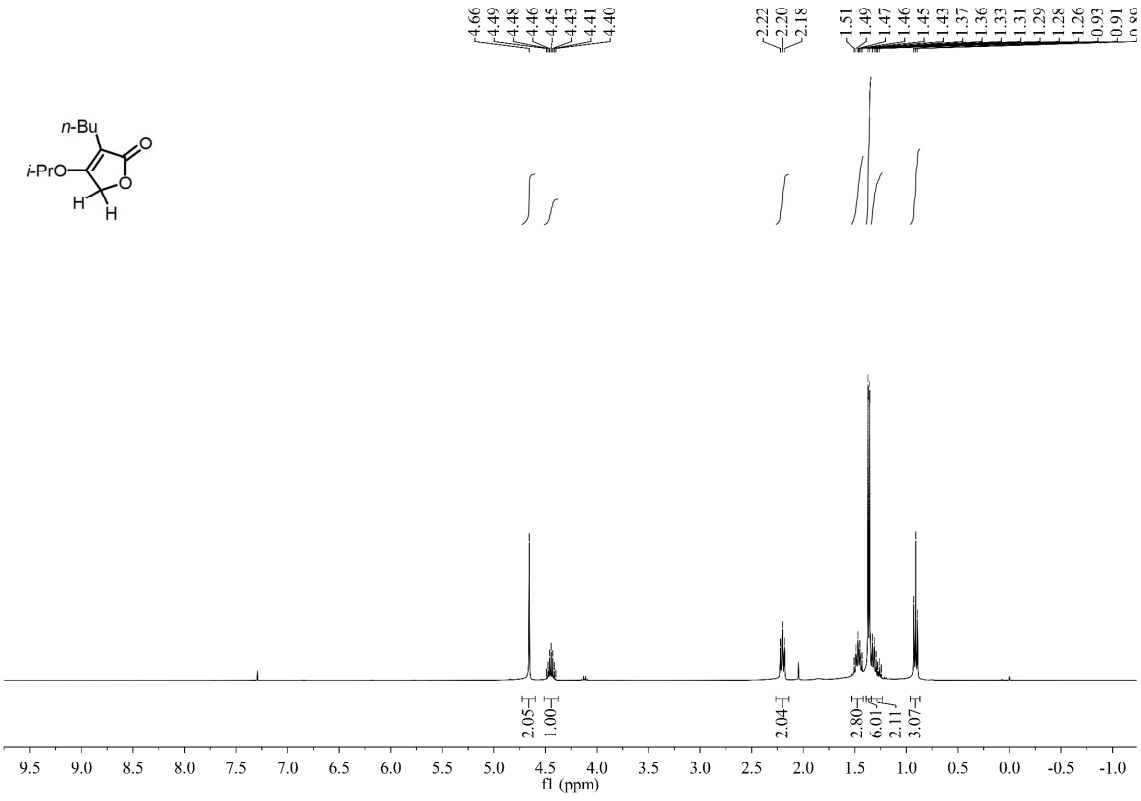

5.62
5.60
5.59
5.57
5.56
5.54
5.53
4.65
4.64
4.63
4.62
1.86
1.85
1.83
1.60
1.59
1.57
1.55
1.53
1.42
1.40
1.39
1.37
1.36
1.35
1.31
1.29
0.91
0.89
0.88
0.20
TMS
O
O
i-PrO
H
n-Bu
0.90
0.91
1.09
1.81
6.62
3.51
3.69
9.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

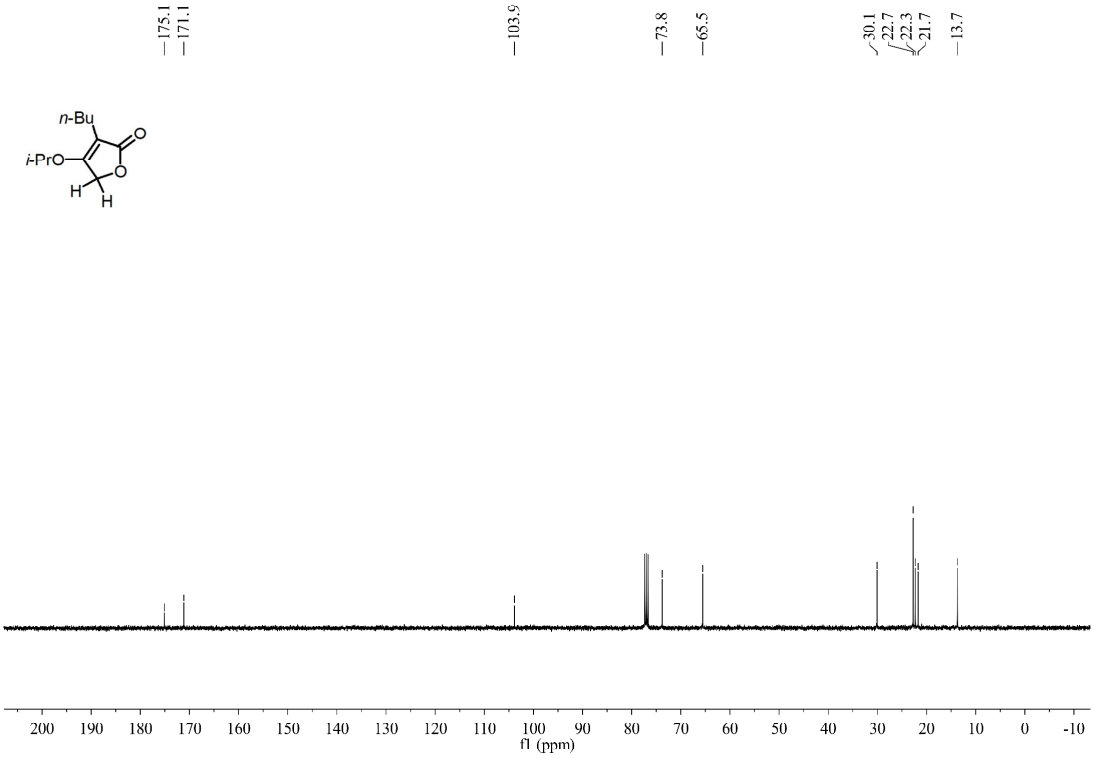

178.2
171.2
100.2
93.6
86.7
78.5
76.0
31.5
26.1
22.6
22.6
22.3
13.8
-0.3
TMS
O
O
i-PrO
H
n-Bu
fl (ppm)
7.49
7.46
6.93
6.91
4.79
4.78
4.77
4.76
4.65
4.63
4.61
4.60
4.58
3.82
2.00
1.99
1.98
1.98
1.97
1.97
1.96
1.95
1.95
1.94
1.68
1.67
1.66
1.65
1.64
1.63
1.62
1.61
1.61
1.60
1.59
1.52
1.51
1.50
1.49
1.48
1.47
1.46
1.45
1.44
1.42
1.41
1.40
1.39
1.37
1.35
1.34
1.24
1.23
1.19
1.18
0.95
0.93
0.91
MeO
O
O
i-PrO
H
n-Bu
2.05
2.21
1.00
1.00
3.21
1.05
1.65
2.34
2.35
2.98
2.91
3.46
fl (ppm)


173.4
172.6
159.1
130.5
122.3
113.7
103.9
77.5
74.2
55.2
32.2
26.5
22.4
22.3
22.2
13.9
MeO
O
O
i-PrO
n-Bu
H
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.58
7.40
7.38
7.25
7.23
4.82
4.81
4.80
4.79
4.63
4.61
4.60
4.58
4.57
2.38
2.01
2.00
1.99
1.99
1.98
1.98
1.97
1.97
1.96
1.95
1.69
1.67
1.65
1.64
1.63
1.62
1.62
1.61
1.60
1.53
1.52
1.51
1.50
1.48
1.47
1.47
1.46
1.45
1.44
1.42
1.41
1.40
1.39
1.37
1.35
1.34
1.29
1.27
1.23
1.21
0.95
0.93
0.91
Me
Cl
O
O
i-PrO
n-Bu
H
0.73
0.80
0.80
0.91
0.91
3.00
0.89
1.46
2.27
2.15
3.10
2.95
2.94
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


173.6
172.6
135.8
134.2
130.7
129.5
129.2
127.3
103.1
77.4
74.9
32.2
26.4
22.4
22.3
22.2
19.9
13.8
Me
Cl
O
O
i-PrO
H
n-Bu
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

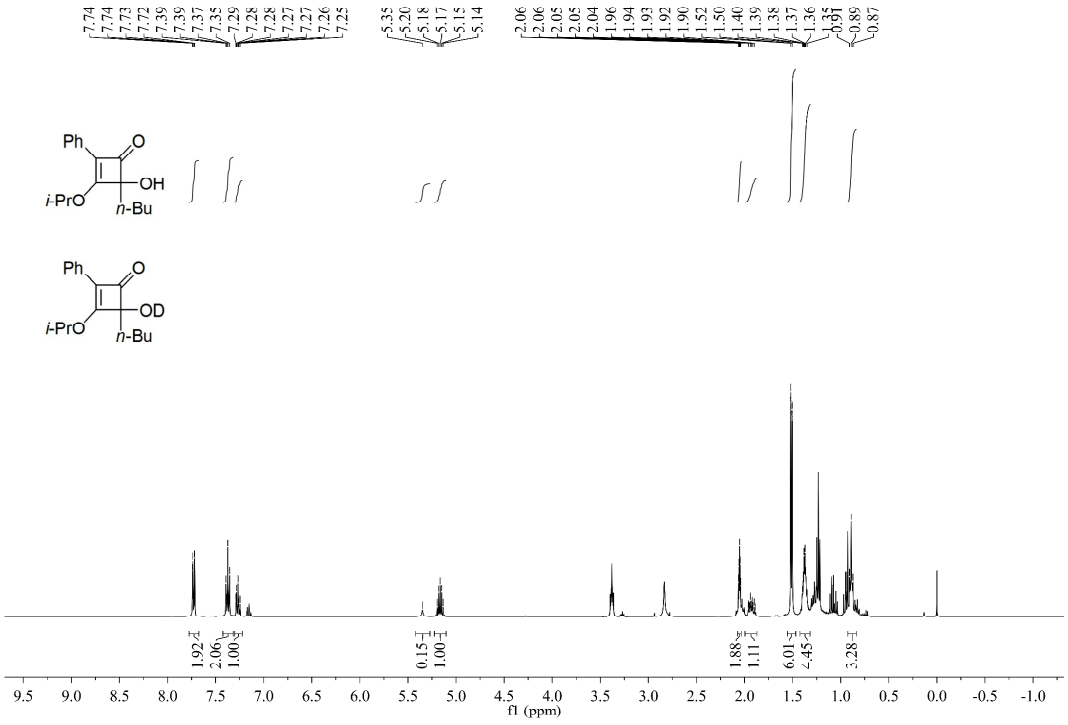

4.66
4.49
4.48
4.46
4.45
4.43
4.41
4.40
2.22
2.20
2.18
1.51
1.49
1.47
1.46
1.45
1.43
1.37
1.36
1.33
1.31
1.29
1.28
1.26
0.93
0.91
n-Bu
i-PrO
O
O
H
H
2.05
1.00
2.04
2.80
6.01
2.11
3.07
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)



8. ^{1}H NMR Spectrum from Deuterated Experiments.

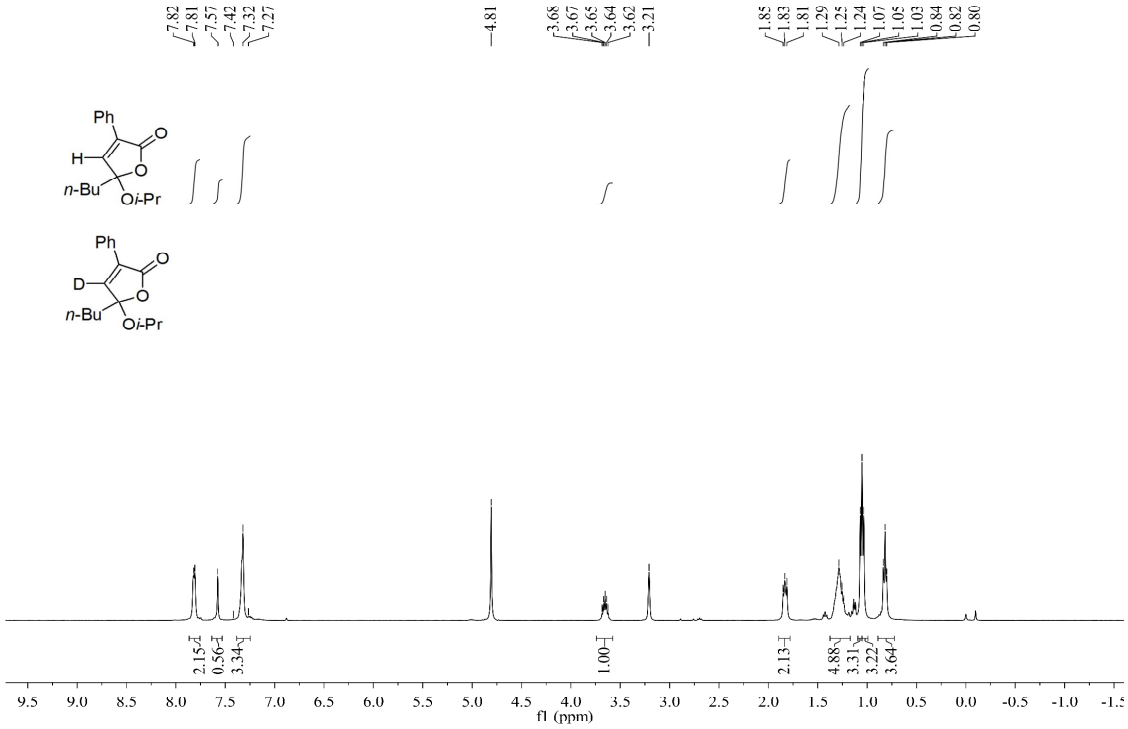
5.1 Preparation of α-Hydroxylcyclobutenone 1s:

^{1}H NMR Spectrum of **1s** (85% deuteration, 400 MHz, CD_3COCD_3)

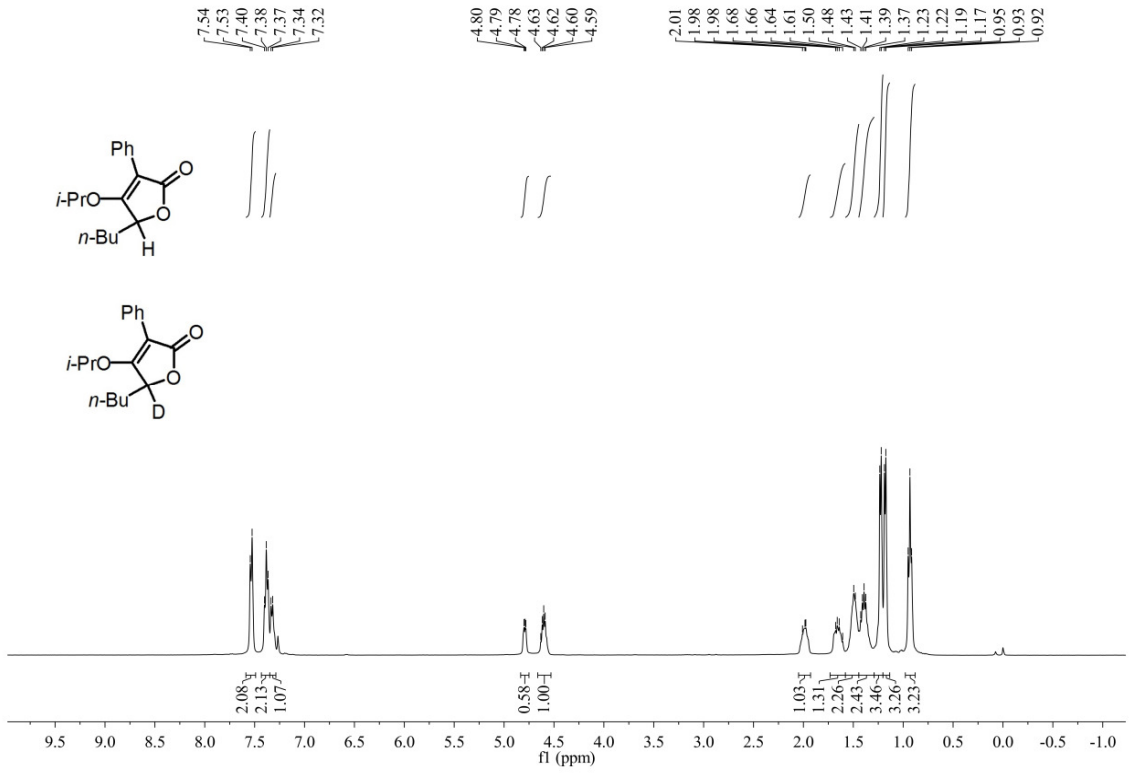


5.2 Nickel-Catalyzed Ring-Opening of 1s with PPh_3 or Xantphos as the Ligand:

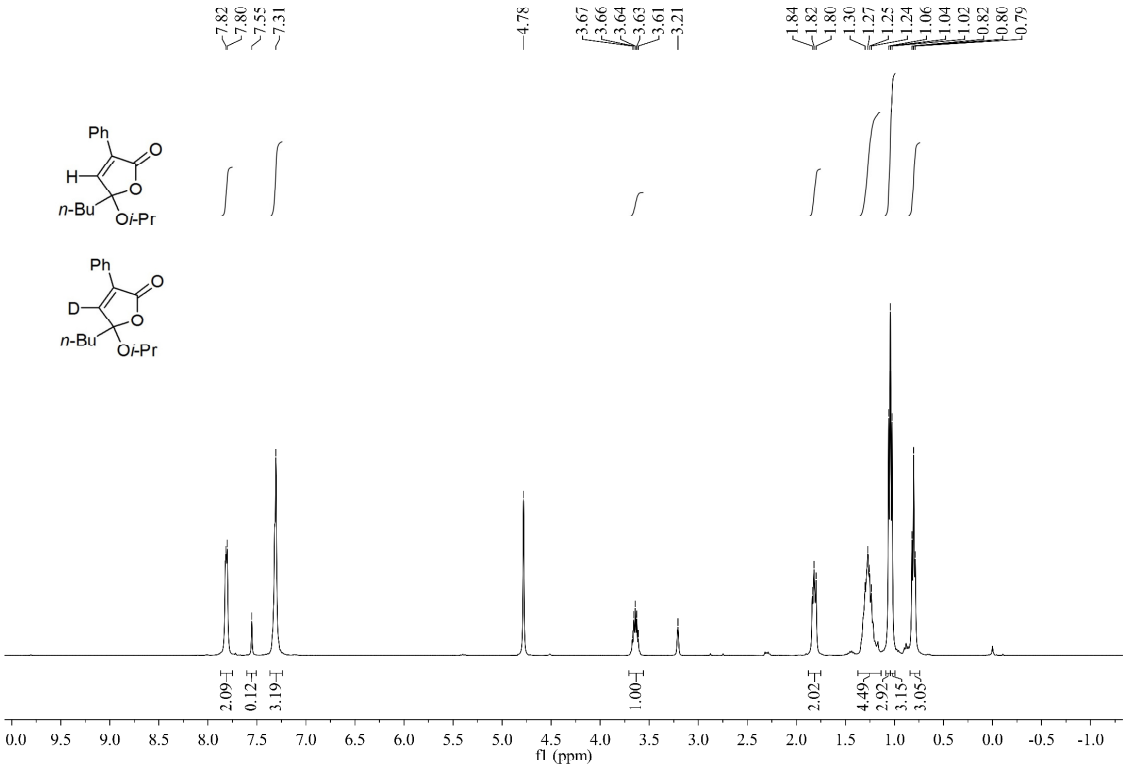
^{1}H NMR Spectrum of **2s** (44% deuteration, 400 MHz, CD_3OD)



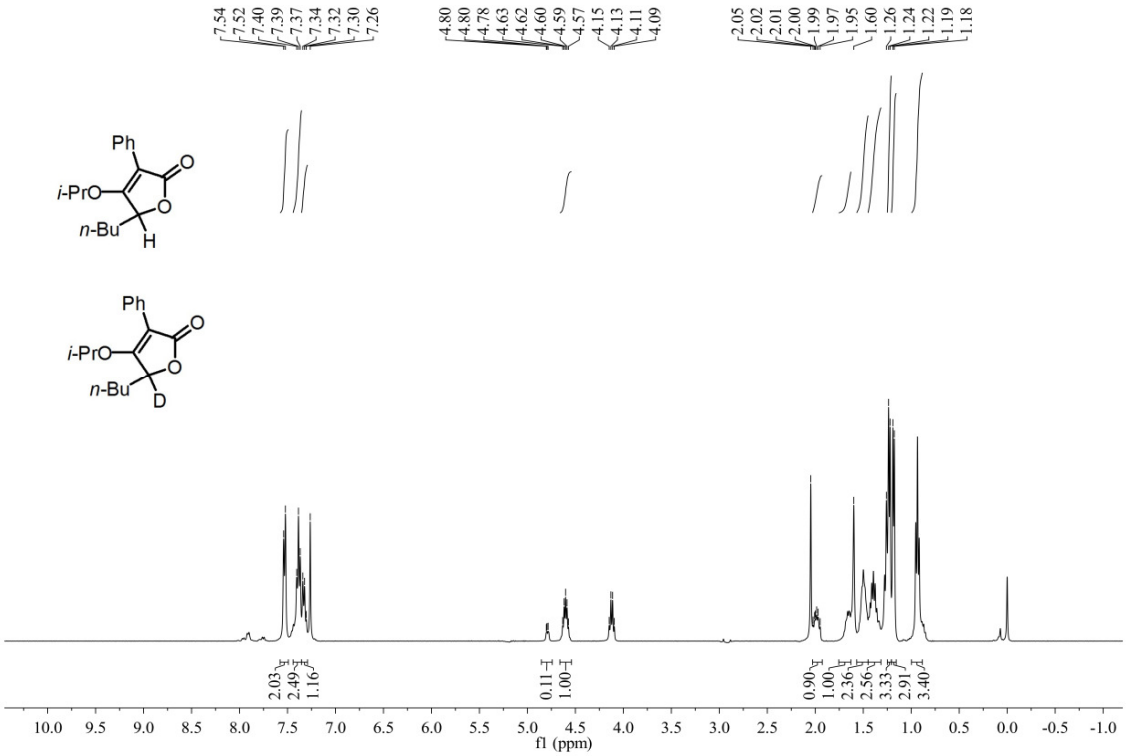
^{1}H NMR Spectrum of **3s** (42% deuteration, 400 MHz, $CDCl_3$)



^{1}H NMR Spectrum of **2s** (88% deuteration, 400 MHz, CD_3OD)

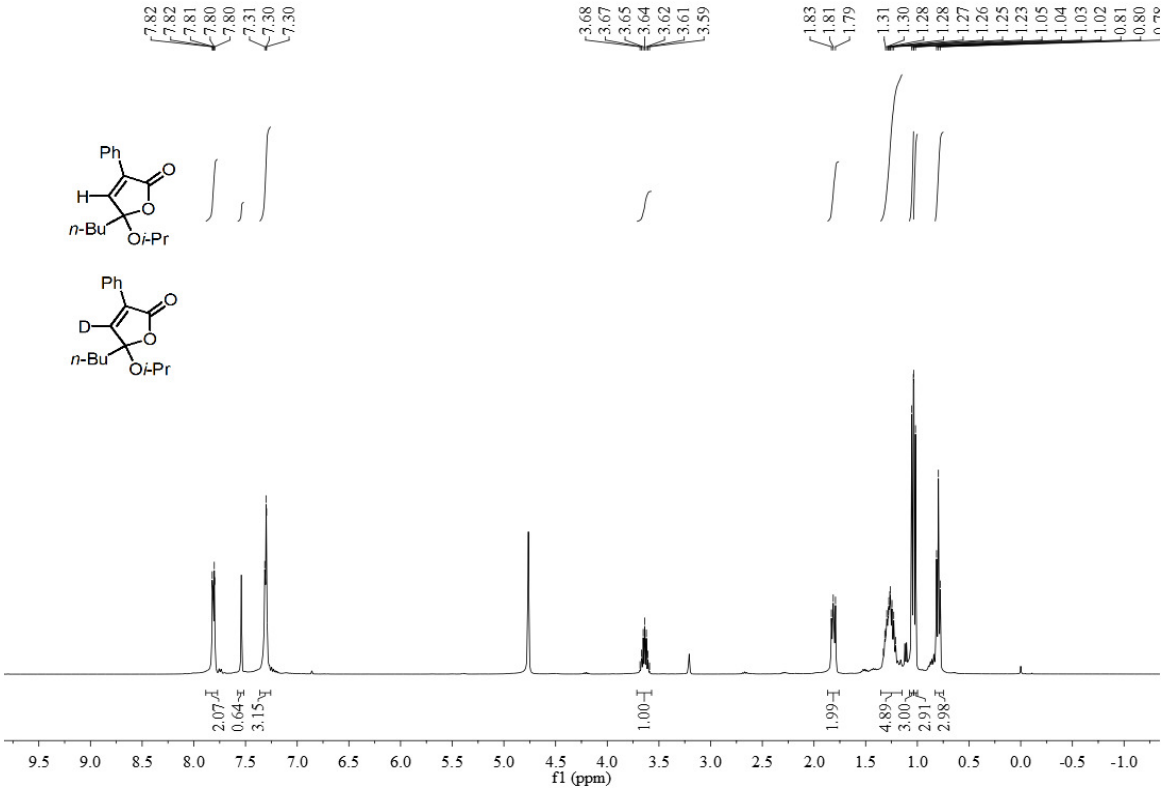


^{1}H NMR Spectrum of **3s** (89% deuteration, 400 MHz, $CDCl_3$)

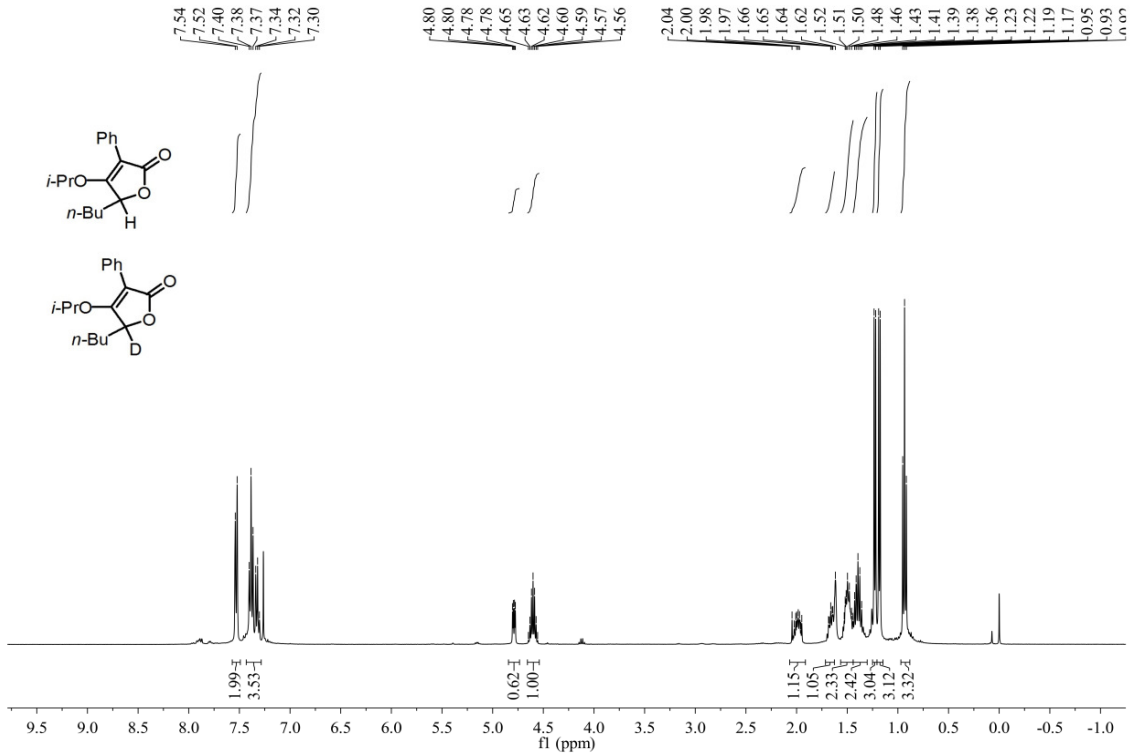


5.3 Related Control Experiments:

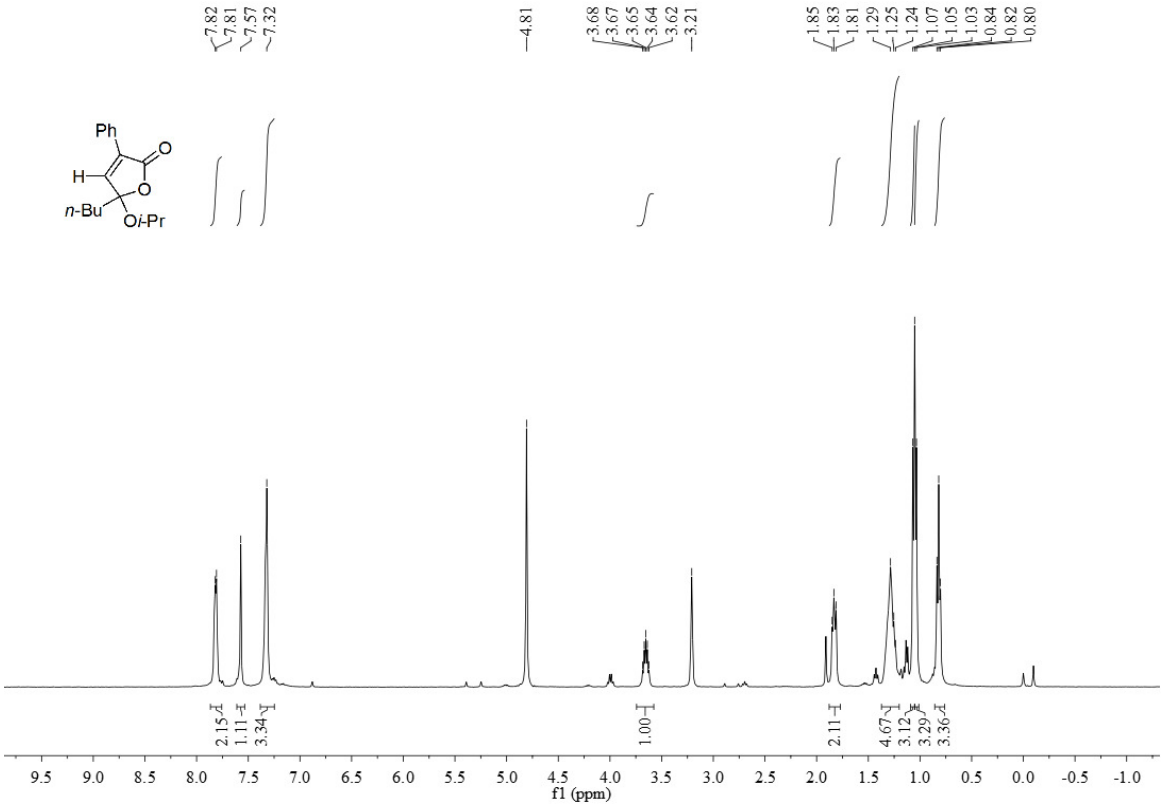
^{1}H NMR Spectrum of **2s** (36% deuteration, 400 MHz, CD_3OD)



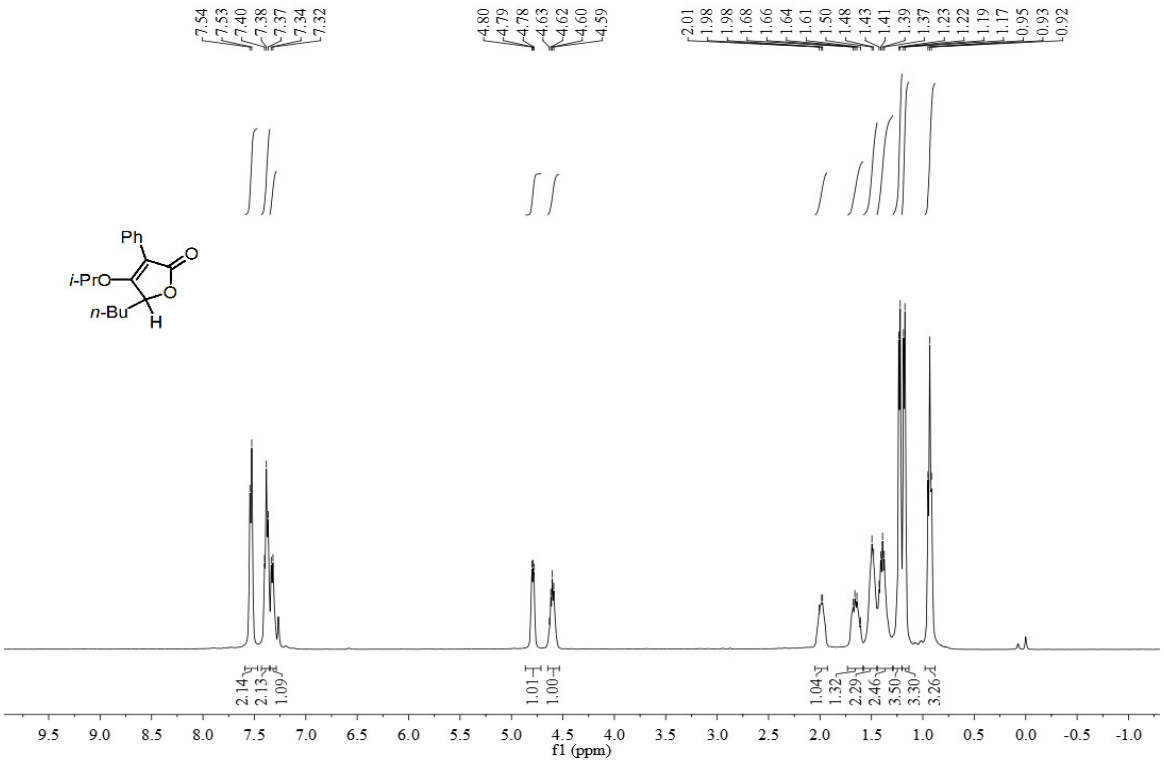
^{1}H NMR Spectrum of **3s** (38% deuteration, 400 MHz, $CDCl_3$)



^{1}H NMR Spectrum of **2a** (400 MHz, CD_3OD)

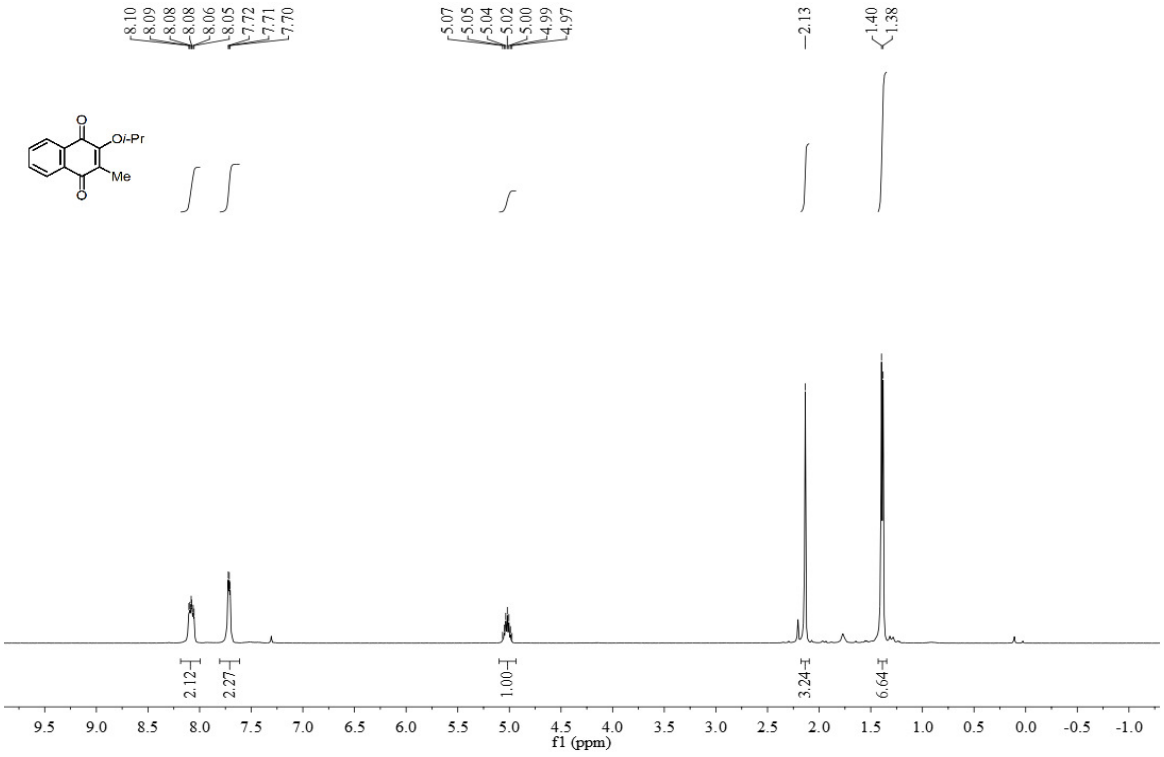


^{1}H NMR Spectrum of **3a** (100 MHz, $CDCl_3$)

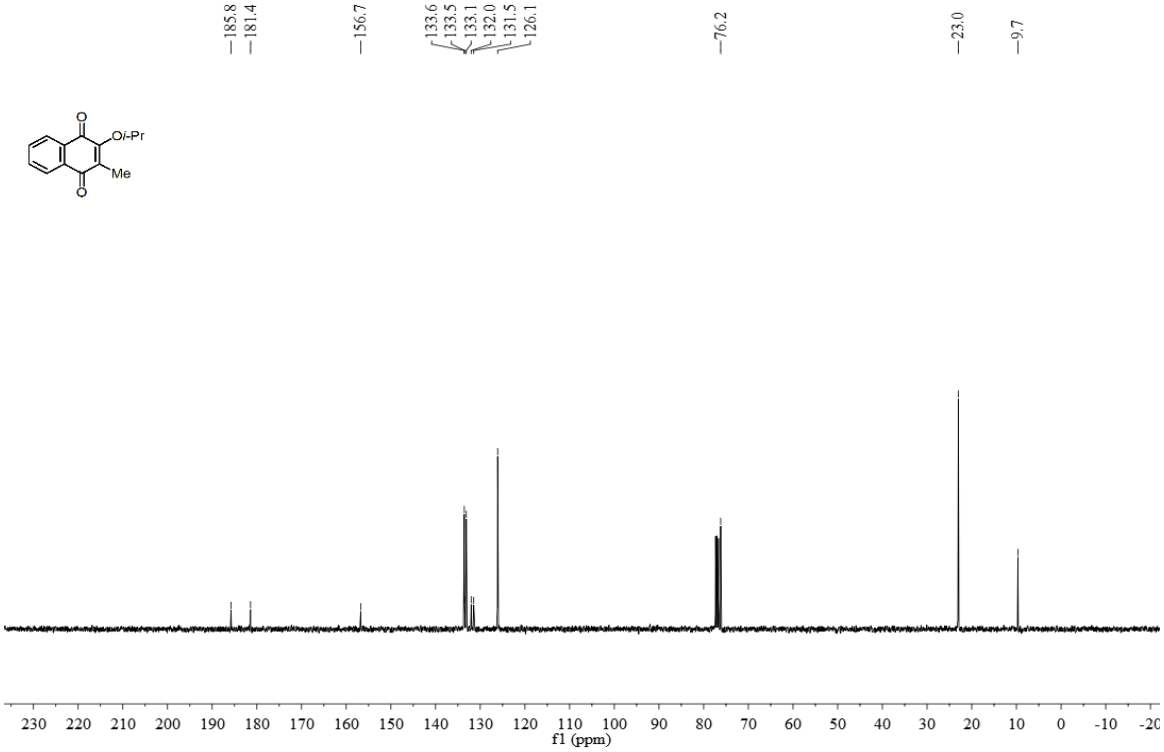


9. NMR Spectrum from Nickel-Catalyzed Ring-Opening of α-Hydroxylcyclotenones Bearing Phenyl or Propenyl group as the R^3 Substituent with PPh_3 or Xantphos as the Ligand

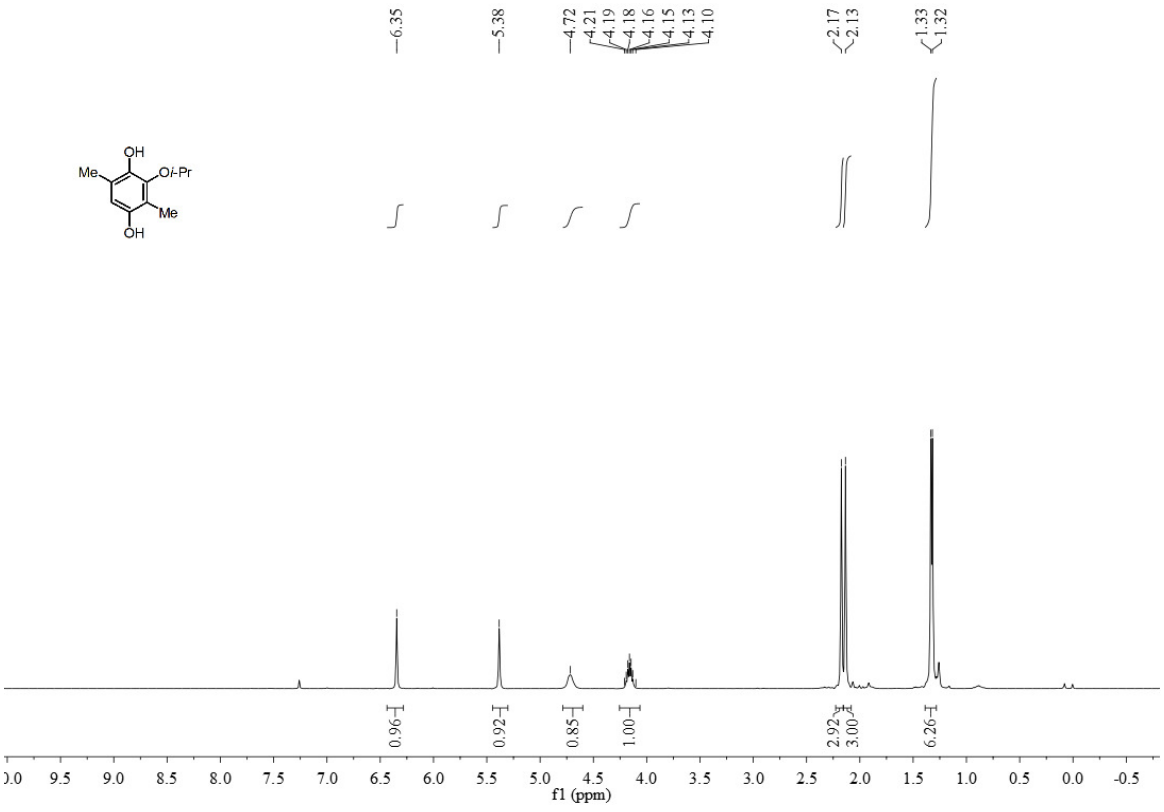
^{1}H NMR Spectrum of **4** (400 MHz, $CDCl_3$)



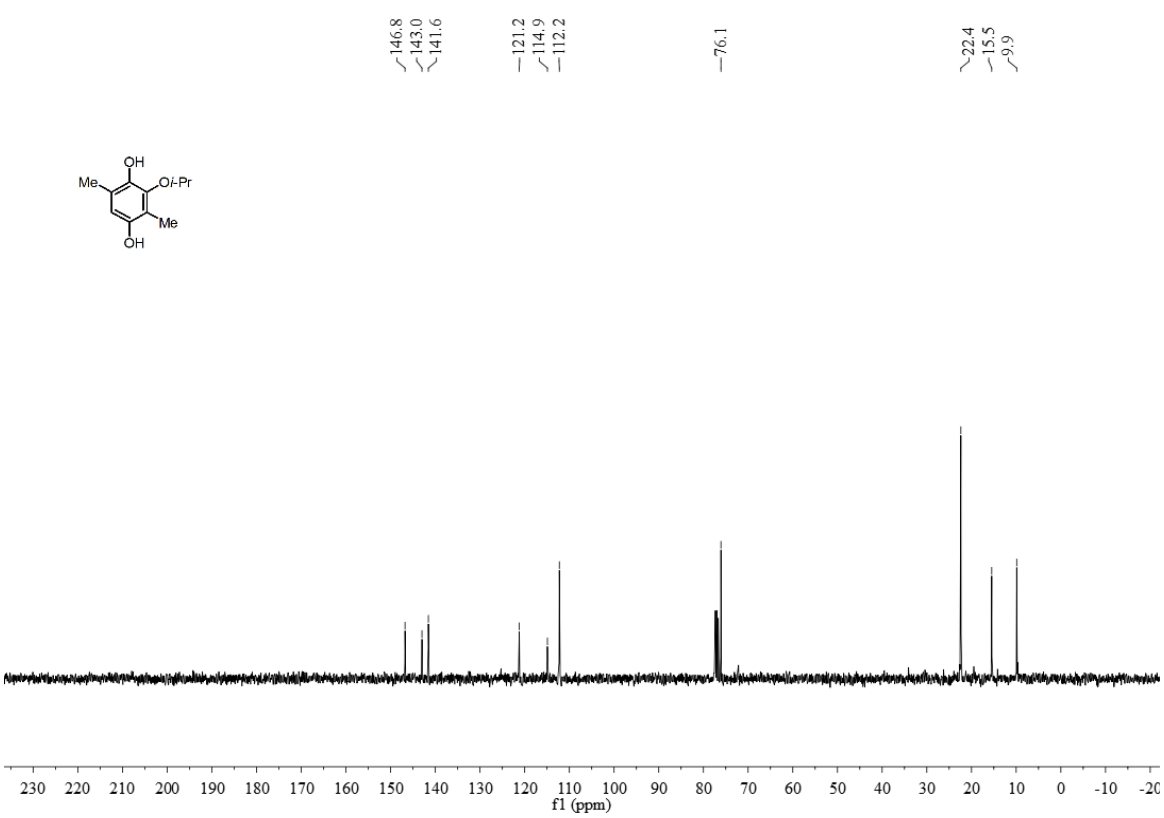
^{13}C NMR Spectrum of **4** (100 MHz, $CDCl_3$)



^{1}H NMR Spectrum of **5** (400 MHz, $CDCl_3$)



^{13}C NMR Spectrum of **5** (100 MHz, $CDCl_3$)


146.8
143.0
141.6
121.2
114.9
112.2
76.1
22.4
15.5
9.9
OH
Me
O*i*-Pr
Me
OH
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)