Supporting Information

MnO_2-Promoted Carboesterification of Alkenes with Anhydrides: A Facile Approach to γ-Lactones

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Contents

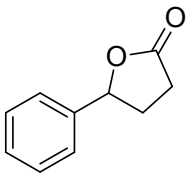
1. General methods

All solvents and commercialized reagents were used without further purifications. ^{1}H, ^{13}C and ^{19}F NMR data were recorded on a Bruker DRX-400 NMR Spectrometer with $CDCl_3$ as the solvent. ^{1}H shifts were referenced to $CDCl_3$ at 7.26 ppm. ^{13}C shifts were referenced to $CDCl_3$ at 77 ppm. IR spectra were obtained with an infrared spectrometer on either potassium bromide pellets or liquid films between two potassium bromide pellets. GC−MS data were obtained on Thermo using electron ionization. HRMS was carried out on a high-resolution mass spectrometer (LCMS-IT-TOF). TLC was performed using commercially available 100−400 mesh silica gel plates (GF_{254}).

2. General procedure for hydration of alkynes

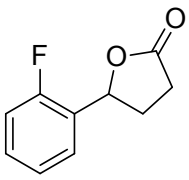
To a mixture of alkene (0.5 mmol), MnO_2 (1.2 equiv.), NaOAc (1.0 equiv.) and LiBr (0.2 equiv.) was added Ac_2O (1.0 mL) in a test tube (10 mL) equipped with a magnetic stirring bar. The mixture was stirred at 120 °C for 3 h. After the reaction was completed, the residue was directly subjected to silica gel column chromatography or purification of the mixture on a preparative TLC to afford the desired product.

3. Characterization data for all products



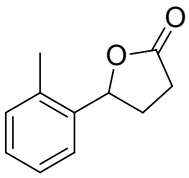
5-Phenyldihydrofuran-2(3*H*)-one (2a) [1]

Yield: 92% (74.5 mg); Red thick oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.34 (m, 5H), 5.52 (t, J = 7.0 Hz, 1H), 2.70-2.64 (m, 3H), 2.27-2.15 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 176.7, 139.4, 128.8, 128.5, 125.3, 81.3, 30.9, 28.9; IR (KBr) ν_{max} (cm^{-1}) 3032, 2930, 2358, 1775, 1174, 1025, 757; MS (EI) m/z 56, 77, 107, 117, 162.



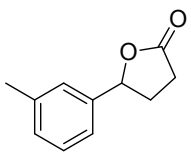
5-(2-Fluorophenyl)dihydrofuran-2(3*H*)-one (2b) [2]

Yield: 85% (76.5 mg); Red thick oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (t, J = 8.0 Hz, 1H), 7.33 (d, J = 4.0 Hz, 1H), 7.17 (t, J = 4.0 Hz, 1H), 7.08 (t, J = 8.0 Hz, 1H), 5.74 (t, J = 8.4 Hz, 1H), 2.76-2.70 (m, 1H), 2.68-2.64 (m, 2H), 2.24-2.15 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.7, 159.2 (d, J = 245 Hz), 130.0 (d, J = 8.1 Hz), 126.9 (d, J = 12.3 Hz), 126.5 (d, J = 4.3 Hz), 124.5 (d, J = 4.1 Hz), 115.6 (d, J = 21.6 Hz), 76.3, 29.7, 28.5; IR (KBr) ν_{max} (cm^{-1}) 3537, 3067, 2940, 2358, 2356, 1781, 1492, 1184, 1033, 761; MS (EI) m/z 56, 95, 123, 135, 180.



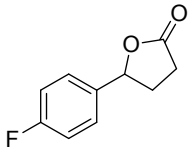
5-(*o*-Tolyl)dihydrofuran-2(3*H*)-one (2c) [1]

Yield: 86% (75.7 mg); White solid, M.p. 60-60.5ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, J = 4.4 Hz, 1H), 7.24-7.13 (m, 2H), 7.19 (m, 1H), 5.71 (t, J = 6.4 Hz, 1H), 2.69-2.64 (m, 3H), 2.34 (s, 3H), 2.16-2.10 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.1, 137.6, 134.2, 130.7, 128.2, 126.5, 124.2, 78.9, 29.6, 28.7, 19.0; IR (KBr) ν_{max} (cm^{-1}) 3529, 2928, 2360, 1775, 1459, 1177, 1022, 754; MS (EI) m/z 65, 91, 121, 131, 161, 176.



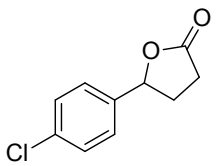
5-(*m*-Tolyl)dihydrofuran-2(3*H*)-one (2d) [3]

Yield: 89% (78.3 mg); Orange thick oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.29 (m, 1H), 7.18-7.13 (m, 3H), 5.50 (t, J = 8.0 Hz, 1H), 2.67 (d, J = 4.8 Hz, 3H), 2.39 (s, 3H), 2.24-2.19 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.0, 139.4, 138.6, 129.2, 128.7, 125.9, 122.4, 81.3, 30.9, 28.9, 21.4; IR (KBr) ν_{max} (cm^{-1}) 2927, 1776, 1181, 1030, 904, 786; MS (EI) m/z 56, 91, 121, 131, 161, 176.



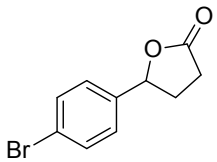
5-(4-Fluorophenyl)dihydrofuran-2(3*H*)-one (2e) [1]

Yield: 81% (72.9 mg); Red thick oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.33 (m, 2H), 7.12-7.07 (m, 2H), 5.50 (t, J = 8.0 Hz, 1H), 2.70-2.66 (m, 3H), 2.22-2.13 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 162.7(d, J = 246.1 Hz), 135.1 (d, J = 2.4 Hz), 127.2 (d, J = 9.1 Hz), 115.7(d, J = 21.6 Hz), 80.7, 31.0, 29.0; IR (KBr) ν_{max} (cm^{-1}) 3532, 2931, 1779, 1511, 1226, 1026, 836; MS (EI) m/z 56, 95, 125, 135, 180.



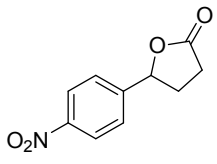
5-(4-Chlorophenyl)dihydrofuran-2(3*H*)-one (2f) [1]

Yield: 84% (78.4 mg); Red thick oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.35 (m, 2H), 7.28-7.26 (m, 2H), 5.48 (t, J = 7.2 Hz, 1H), 2.69-2.63 (m, 3H), 2.18-2.11 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 176.6, 137.9, 134.3, 129.0, 126.7, 81.5, 30.9, 28.9; IR (KBr) ν_{max} (cm^{-1}) 3533, 2932, 2361, 1781, 1492, 1175, 1021, 812; MS (EI) m/z 56, 77, 117, 141, 161, 196.



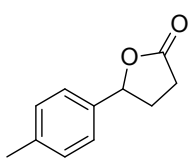
5-(4-Bromophenyl)dihydrofuran-2(3*H*)-one (2g) [1]

Yield: 84% (96.4 mg); Yellow brown solid, M.p. 80-81 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (s, 1H),7.50 (s, 1H), 7.22 (s, 1H), 7.20 (s, 1H), 5.45 (t, J = 8.0 Hz, 1H), 2.67-2.62 (m, 3H), 2.17-2.11 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 176.6, 138.5, 131.9, 127.0, 122.4, 80.5, 30.9, 28.9; IR (KBr) ν_{max} (cm^{-1}) 3532, 2927, 1782, 1489, 1174, 1018, 810; MS (EI) m/z 56, 77, 115, 161, 184, 241.



5-(4-Nitrophenyl)dihydrofuran-2(3*H*)-one (2h) [1]

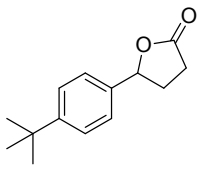
Yield: 79% (81.7 mg); Yellow solid, M.p. 81-82ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (d, J = 8.4 Hz, 2H), 7.53 (d, J = 8.4 Hz, 2H), 5.60 (t, J = 7.2 Hz, 1H), 2.78-2.68 (m, 3H), 2.19-2.14 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 175.9, 147.9, 146.6, 125.9, 124.1, 79.6, 30.9, 28.6; IR (KBr) ν_{max} (cm^{-1}) 3062, 2923, 1782, 1522, 1346, 1170, 740; MS (EI) m/z 56, 91, 121, 149, 159, 175, 204, 207.



5-(*p*-Tolyl)dihydrofuran-2(3*H*)-one (2i) [1]

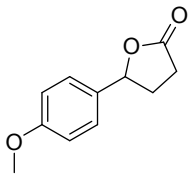
Yield: 89% (78.3 mg); White solid, M.p. 76-78 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28-7.20 (m, 4H), 5.50 (t, J = 7.2 Hz, 1H), 2.68-2.62 (m, 3H), 2.38 (s, 3H), 2.24-2.16 (m, 1H); ^{13}C NMR ($CDCl_3$, 100

MHz) δ 177.0, 138.3, 136.4, 129.4, 125.4, 81.4, 30.9, 29.0, 21.2; IR (KBr) ν_{max} (cm^{-1}) 3505, 2928, 1770, 1455, 1180, 940; MS (EI) m/z 56, 91, 121, 132, 161, 176.



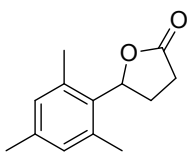
5-(4-(*tert*-Butyl)phenyl)dihydrofuran-2(3*H*)-one (2j) [1]

Yield: 92% (100.3 mg); Yellow solid, M.p. 56-57ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (s, 1H), 7.43 (s, 1H), 7.31 (s, 1H), 7.28 (s, 1H), 5.52 (t, J = 6.8 Hz, 1H), 2.69-2.65 (m, 3H), 2.26-2.20 (m, 1H), 1.35 (s, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 177.0, 151.6, 136.3, 125.7, 125.2, 81.3, 34.6, 31.3, 30.8, 29.1; IR (KBr) ν_{max} (cm^{-1}) 3700, 2960, 2359, 1779, 1175, 1022, 835; MS (EI) m/z 57, 85, 115, 161, 175, 203, 218.



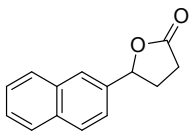
5-(4-Methoxyphenyl)dihydrofuran-2(3*H*)-one (2k) [1]

Yield: 91% (87.4 mg); Yellow solid, M.p. 53-54ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.24 (m, 2H), 6.91 (d, J = 8.4 Hz, 2H), 5.47-5.44 (m, 1H), 3.81 (s, 3H), 2.64-2.58 (m, 3H), 2.21-2.17 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 159.8, 131.2, 126.9, 114.1, 81.4, 55.4, 30.9, 29.2; IR (KBr) ν_{max} (cm^{-1}) 3527, 2940, 2360, 1775, 1514, 1249, 1027, 834; MS (EI) m/z 56, 77, 109, 137, 148, 161, 192.



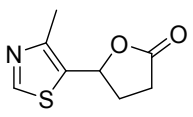
5-Mesityldihydrofuran-2(3*H*)-one (2l) [4]

Yield: 88% (89.8 mg); Red thick oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.84 (s, 2H), 5.87 (dd, J = 10.4, 7.2 Hz, 1H), 2.73-2.69 (m, 2H), 2.56-2.47 (m, 1H), 2.32 (s, 6H), 2.28 (s, 1H), 2.25 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 177.2, 137.9, 135.9, 131.2, 130.4, 79.5, 29.6, 29.0, 20.8, 20.5; IR (KBr) ν_{max} (cm^{-1}) 3524, 2966, 1774, 1462, 1182, 1033, 857; MS (EI) m/z 56, 91, 105, 121, 149, 159, 175, 204.



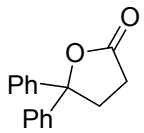
5-(Naphthalen-2-yl)dihydrofuran-2(3*H*)-one (2m) [1]

Yield: 87% (92.2 mg); Yellow solid, M.p. 117-118ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88-7.80 (m, 4H), 7.50 (d, J = 5.4 Hz, 2H), 7.40 (d, J = 8.4 Hz, 1H), 5.66 (d, J = 7.6 Hz, 1H), 2.74-2.66 (m, 3H), 2.29-2.25 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 136.7, 133.2, 133.1, 128.8, 128.1, 127.8, 126.6, 126.5, 124.3, 122.9, 81.3, 30.9, 28.9; 2923, 2359, 1756, 1196, 1015, 901, 756; IR (KBr) ν_{max} (cm^{-1}) MS (EI) m/z 56, 128, 157, 167, 212.



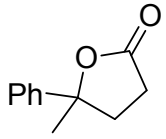
5-(4-Methylthiazol-5-yl)dihydrofuran-2(3*H*)-one (2n)

Yield: 72% (65.9 mg); Red thick oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (s, 1H), 5.79-5.75 (m, 1H), 2.76-2.69 (m, 3H), 2.50 (s, 3H), 2.31-2.24 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.6, 151.7, 151.6, 129.7, 74.9, 31.4, 29.1, 15.4; IR (KBr) ν_{max} (cm^{-1}) 3081, 2928, 2361, 1778, 1414, 1144, 931, 809; MS (EI) m/z 56, 100, 128, 139, 156, 183; HRMS-ESI (m/z) calcd for $C_8H_9NNaO_2S$ $[M + Na]^+$ 206.0246, found 206.0248.



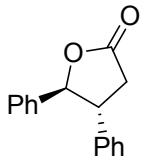
5,5-Diphenyldihydrofuran-2(3*H*)-one (2o) [5]

Yield: 88% (104.7 mg); Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 7.6 Hz, 4H), 7.37 (t, J = 7.6 Hz, 4H), 7.30 (t, J = 7.6 Hz, 2H), 2.94 (t, J = 7.6 Hz, 2H), 2.61 (t, J =7.6 Hz, 2H); ^{13}C NMR (100 Hz, $CDCl_3$) δ 175.8, 143.0, 128.6, 127.9, 125.4, 89.7, 35.7, 29.1; IR (KBr) ν_{max} (cm^{-1}) 3529, 2928, 2360, 1175, 1459, 1177, 1022, 754; MS (EI) m/z 77, 105, 115, 161, 183, 238.



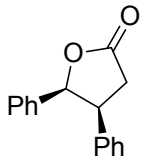
5-Methyl-5-phenyldihydrofuran-2(3*H*)-one (2p) [1]

Yield: 95% (83.6 mg); Red thick oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, J = 8.0 Hz, 4H), 7.27-7.31 (m, 1H), 2.59-2.67 (m, 1H), 2.41-2.53 (m, 3H), 1.72, (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 144.3, 128.6, 127.6, 124.1, 87.0, 36.2, 29.4, 29.0; IR (KBr) ν_{max} (cm^{-1}) 3527, 2932, 2361, 1773, 1454, 1161, 1014, 703; MS (EI) m/z 77, 107, 117, 158, 176.



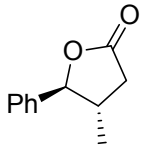
***trans*-4,5-Diphenyldihydrofuran-2(3*H*)-one (2q-1)** [6]

Yield: 48% (57.1 mg); White solid, M.p. 109-110 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.28 (m, 6H), 7.19 (t, J = 7.2 Hz, 4H), 5.43 (d, J = 8.4 Hz, 1H), 3.60 (dd, J = 19.2, J = 8.4 Hz, 1H), 3.07 (dd, J = 17.6, J = 8.4 Hz, 1H), 2.92 (dd, J = 17.6, J = 10.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 175.3, 138.0,137.8, 129.1, 128.7, 127.9, 127.4, 125.7, 87.5, 50.6, 37.2; IR (KBr) ν_{max} (cm^{-1}) 3359, 2922, 2361, 1783, 1456, 1198, 996, 698; MS (EI) m/z 77, 91, 105, 115, 161, 183, 238.



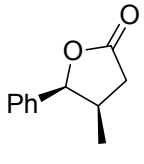
***cis*-4,5-Diphenyldihydrofuran-2(3*H*)-one (2q-2)** [6]

Yield: 43% (51.2 mg); White solid, M.p. 106-108 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13-7.08 (m, 6H), 6.90 (dd, J = 6.4, J = 2.8 Hz, 2H), 6.81 (dd, J = 6.4, J = 3.2 Hz, 2H), 5.83 (d, J = 6.8 Hz, 1H), 4.06 (dd, J = 14.8, J = 6.8 Hz, 1H), 3.05 (dd, J = 17.6, J = 8.0 Hz, 1H), 2.94 (dd, J = 17.6, 6.4 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 176.7, 136.7,135.5, 128.3, 128.0, 127.9, 127.9, 127.4, 125.7, 84.7, 46.9, 34.9; IR (KBr) ν_{max} (cm^{-1}) 3358, 2923, 2361, 1780, 1173, 697; MS (EI) m/z 77, 91, 105, 115, 161, 183, 238.



***trans*-4-Methyl-5-phenyldihydrofuran-2(3*H*)-one (2r-1)** [5]

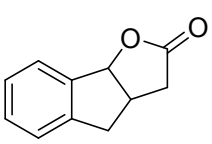
Yield: 54% (47.5 mg); Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.36 (m, 2H), 7.34-7.26(m, 3H), 5.58 (d, J = 4.8 Hz, 1H), 2.76-2.71 (m, 1H), 2.43-2.32 (m, 2H), 1.33 (d, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.9, 139.8, 128.8, 128.2, 124.9, 78.4, 38.4, 33.6, 15.4; IR (KBr) ν_{max} (cm^{-1}) 2928, 2359, 1780, 1212, 999, 702; MS (EI) m/z 77, 91, 107, 117, 131, 158, 176.



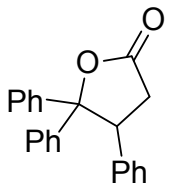
***cis*-4-Methyl-5-phenyldihydrofuran-2(3*H*)-one (2r-2)** [5]

Yield: 33% (29.0 mg); Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.34 (m, 5H), 5.36 (t, J = 7.6 Hz, 1H), 2.81 (dd, J = 12.0, J = 8.4 Hz, 2H), 1.85 (d, J = 10.4 Hz, 1H), 1.33 (d, J = 4.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.2, 139.1, 128.7, 128.5, 125.5, 79.2, 39.9, 36.4, 15.0; IR (KBr) ν_{max}

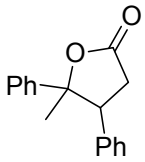
(cm^{-1}) 2928, 2359, 1780, 1212, 999, 702; MS (EI) m/z 77, 91, 107, 117, 131, 158, 176.



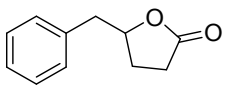
3,3*a*,4,8*b*-Tetrahydro-2*H*-indeno[1,2-*b*]furan-2-one (2s) [7]
Yield: 83% (72.2 mg); Yellow solid. M.p. 67-68ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, J = 7.6 Hz, 1H), 7.58-7.35 (m, 1H), 7.32-7.28(t, J = 7.6 Hz, 2H), 5.90 (d, J = 4.0 Hz, 1H), 3.38-3.30(m, 2H), 2.95-2.88 (m, 2H), 2.40 (dd, J = 18.0, J = 5.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.0, 142.6, 138.8, 129.9, 127.6, 126.4, 125.4, 87.7, 37.9, 37.3, 35.7; IR (KBr) ν_{max} (cm^{-1}) 3532, 2930, 2357, 1775, 1462, 1178, 1014, 751; MS (EI) m/z 63, 89, 114, 130, 145, 174.



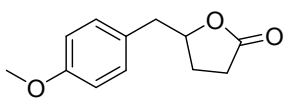
4,5,5-Triphenyldihydrofuran-2(3*H*)-one (2t) [8]
Yield: 75% (117.7 mg); White solid, M.p. 162-163 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, J = 7.8 Hz, 2H), 7.40 (t, J = 7.6 Hz, 2H), 7.32 (t, J = 7.2 Hz, 1H), 7.12 (d, J = 2.4 Hz, 3H), 7.06 (s, 5H), 6.95 (s, 2H), 4.49 (dd, J = 7.6, J = 4.8 Hz, 1H), 2.99 (dd, J = 17.6, J = 8.0 Hz, 1H), 2.80 (dd, J = 17.6, J = 4.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.8, 143.2, 139.9, 138.5, 128.7, 128.6, 128.4, 128.2, 127.7, 127.3, 127.2, 126.3, 126.1, 92.9, 51.0, 37.5; IR (KBr) ν_{max} (cm^{-1}) 3061, 2359, 1777, 1450, 1221, 981, 699; MS (EI) m/z 77, 91, 104, 165, 183, 256, 314.



5-Methyl-4,5-diphenyldihydrofuran-2(3*H*)-one (2u) [9]
Yield: 81% (102.1 mg); White solid. M.p. 112-113 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.32 (m, 8H), 7.15-7.13 (m, 2H), 3.76 (t, J = 7.8 Hz, 1H), 2.90 (dd, J = 8.0, J = 3.2 Hz, 2H), 1.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.9, 144.3, 137.3, 128.8, 128.6, 128.2, 127.9, 127.8, 124.5, 89.6, 52.9, 35.2, 24.1; IR (KBr) ν_{max} (cm^{-1}) 3031, 2929, 2361, 1777, 1450, 1224, 1055, 761; MS (EI) m/z 77, 91, 104, 121, 179, 224, 252.

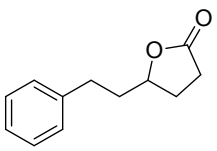


5-Benzyldihydrofuran-2(3*H*)-one (2v) [6]
Yield: 82% (72.2 mg); Red thick oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.30 (m, 2H), 7.27-7.22 (m, 3H), 4.73 (t, J = 8.0 Hz, 1H), 3.07 (dd, J = 14.0, J = 6.0 Hz, 1H), 2.93 (dd, J = 14.0, J = 6.0 Hz, 1H), 2.40-2.38 (m, 2H), 2.27-2.22 (m, 1H), 1.98-1.92 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 177.0, 135.9, 129.5, 128.7, 127.0, 80.8, 41.3, 28.7, 27.1; IR (KBr) ν_{max} (cm^{-1}) 3524, 3029, 2926, 2361, 1771, 1454, 1174, 1022, 915, 701; MS (EI) m/z 57, 85, 91, 176.



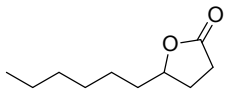
5-(4-Methoxybenzyl)dihydrofuran-2(3*H*)-one (2w) [10]
Yield: 83% (85.5 mg); Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (d, J = 8.0 Hz, 2H), 6.85 (d, J = 8.4 Hz, 2H), 4.73-4.66 (m, 1H), 3.79 (s, 3H), 3.00 (dd, J = 14.0, J = 6.0 Hz, 1H), 2.88 (dd, J = 14.0, J = 6.0 Hz, 1H), 2.51-2.40 (m, 1H), 2.40-2.29 (m, 1H), 2.29-2.19 (m, 1H), 2.00-1.91 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 177.1, 158.7, 130.5, 127.8, 114,1, 81.0, 55.3,

40.4, 28.7, 27.0; IR (KBr) ν_{max} (cm^{-1}) 2929, 2841, 2359, 1772, 1512, 1250, 1176, 1028, 740; MS (EI) m/z 57, 85, 121, 206.



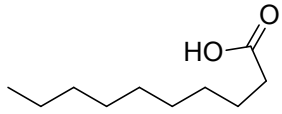
5-Phenethyldihydrofuran-2(3*H*)-one (2x) [11]

Yield: 82% (77.9 mg); Red thick oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.27 (m, 2H), 7.21-7.18 (m, 3H), 4.45 (dd, J = 6.4, J = 5.2 Hz, 1H), 2.81-2.78 (m, 1H), 2.75-2.70 (m, 1H), 2.53-2.49 (m, 2H), 2.31-2.26 (m, 1H), 2.04-2.00 (m, 1H), 1.91-1.84 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 177.3, 140.8, 128.6, 128.5, 126.2, 79.9, 37.4, 31.7, 28.9, 28.0; IR (KBr) ν_{max} (cm^{-1}) MS 3524, 2934, 2357, 1774, 1452, 1177, 751; (EI) m/z 65, 91, 117, 130, 190.



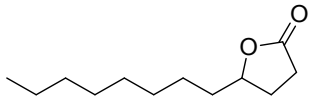
5-Hexyldihydrofuran-2(3*H*)-one (2y) [1]

Yield: 63% (53.5 mg); Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.51-4.47 (m, 1H), 2.53 (t, J = 8.0 Hz, 2H), 2.32 (dd, J = 12.4, 6.4 Hz, 1H), 1.93-1.81 (m, 1H), 1.81-1.67 (m, 1H), 1.67-1.54 (m, 1H), 1.46-1.27 (m, 8H), 0.88 (d, J = 5.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.4, 81.1, 35.6, 31.6, 28.9, 28.9, 27.9, 25.2, 22.5, 14.0; IR (KBr) ν_{max} (cm^{-1}) 2930, 2860, 2361, 1775, 1461, 1180, 912, 652; MS (EI) m/z 55, 85, 95, 126, 170.



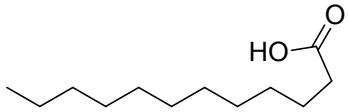
Decanoic acid (2y-1) [13]

Yield: 30% (25.8 mg); Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.95 (s, 1H), 2.37 (t, J = 7.2 Hz, 2H), 1.65 (d, J = 6.8 Hz, 2H), 1.29 (s, 14H), 0.90 (d, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.3, 34.1, 31.9, 29.7, 29.4, 29.2, 29.1, 24.7, 22.7, 14.1; IR (KBr) ν_{max} (cm^{-1}) 2926, 2858, 2361, 1711, 1459, 1286, 937, 671; MS (EI) m/z 65, 84, 98, 112, 130, 155, 172.



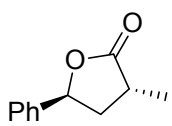
5-Octyldihydrofuran-2(3*H*)-one (2z) [12]

Yield: 61% (60.4 mg); Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.48 (dd, J = 13.2, 6.5 Hz, 1H), 2.52 (t, J = 8.0 Hz, 2H), 2.32 (m, 1H), 1.82-1.76 (m, 1H), 1.74-1.69 (m, 1H), 1.61-1.54 (m, 1H), 1.46-1.27 (m, 14H), 0.88 (t, J = 5.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.3, 81.1, 35.5, 31.8, 29.4, 29.3, 29.1, 28.8, 27.9, 25.2, 22.6, 14.0; IR (KBr) ν_{max} (cm^{-1}) MS 3534, 2927, 2857, 2361, 1777, 1460, 1179, 982, 651; (EI) m/z 55, 85, 96, 128, 151, 180, 198.



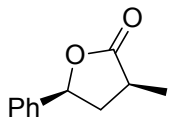
Dodecanoic acid (2z-1) [13]

Yield: 27% (27.0 mg); Yellow solid, M.p. 53-54ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.88 (s, 1H), 2.34 (t, J = 7.3 Hz, 2H), 1.63 (t, J = 4 Hz, 2H), 1.26 (s, 16H), 0.88 (t, J = 4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.5, 34.1, 31.9, 29.6, 29.4, 29.3, 29.2, 29.1, 24.7, 22.7, 14.1; IR (KBr) ν_{max} (cm^{-1}) 2926, 2857, 2361, 1710, 1272, 936, 738; MS (EI) m/z 84, 98, 112, 140, 183, 200.



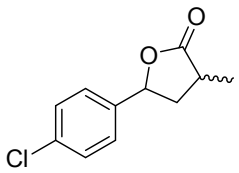
***trans*-3-Methyl-5-phenyldihydrofuran-2(3*H*)-one (3a-1)** [1]

Yield: 38% (33.4 mg); Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.36 (m, 2H), 7.34-7.26 (m, 3H), 5.58 (d, J = 4.9 Hz, 1H), 2.76-2.71 (m, 1H), 2.43-2.32 (m, 2H), 1.33 (d, J = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.9, 139.8, 128.8, 128.2, 124.9, 78.4, 38.4, 33.6, 15.4; IR (KBr) ν_{max} (cm^{-1}) 2933, 2881, 2361, 1774, 1456, 1172, 1012, 700; MS (EI) m/z 77, 91, 105, 117, 132, 176.



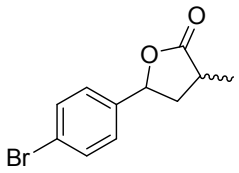
cis-3-Methyl-5-phenyldihydrofuran-2(3*H*)-one (3a-2) [1]

Yield: 40% (35.2 mg); Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.34 (m, 5H), 5.41-5.30 (m, 1H), 2.81 (dd, J = 11.8, 8.6 Hz, 2H), 1.85 (d, J = 10.4 Hz, 1H), 1.33 (d, J = 5.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.2, 139.1, 128.7, 128.5, 125.5, 79.2, 39.9, 36.4, 15.0; IR (KBr) ν_{max} (cm^{-1}) 2932, 2879, 2361, 1773, 1454, 1161, 1014, 753; MS (EI) m/z 77, 91, 105, 117, 132, 176.



5-(4-Chlorophenyl)-3-methyldihydrofuran-2(3*H*)-one (3b) [1]

Yield: 83% (87.1 mg); Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23-7.36 (m, 4H), 5.53 (q, J = 6.8 Hz, 0.44H), 5.32 (q, J = 5.6 Hz, 0.55H), 2.68-2.87 (m, 1.5H), 2.35-2.40 (m, 1H), 1.75-1.85 (m, 0.5H), 1.32 (dd, J = 4.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ179.6, 178.9, 138.3, 137.7, 134.2, 134.0, 128.91, 128.89, 126.9, 126.5, 78.4, 77.7, 39.9, 36.3, 38.2, 33.6, 15.4, 14.9; IR (KBr) ν_{max} (cm^{-1}) 3491, 2921, 2336, 1772, 1439, 1198, 1089, 756; MS (EI) m/z 75, 91, 111, 115, 131, 151, 175, 230, 210.



5-(4-Bromophenyl)-3-methyldihydrofuran-2(3*H*)-one (3c) [1]

Yield: 73% (92.7 mg); White solid, M.p., 95-97 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17-7.51 (m, 4H), 5.51 (q, J = 6.8 Hz, 0.55H), 5.31 (q, J = 5.6 Hz, 0.45H), 2.68-2.81 (m, 1.47H), 2.35-2.38 (m, 1.87H), 1.31-1.33 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.6, 178.9, 138.9, 138.2, 131.9, 131.8, 127.2, 126.8, 122.3, 122.1, 78.4, 77.7, 39.9, 38.2, 36.3, 33.6, 15.4, 14.9; IR (KBr) ν_{max} (cm^{-1}) 3362, 2921, 2362, 1771, 1486, 1196, 756; MS (EI) m/z 77, 91, 116, 131, 175, 185, 210, 254.

References:

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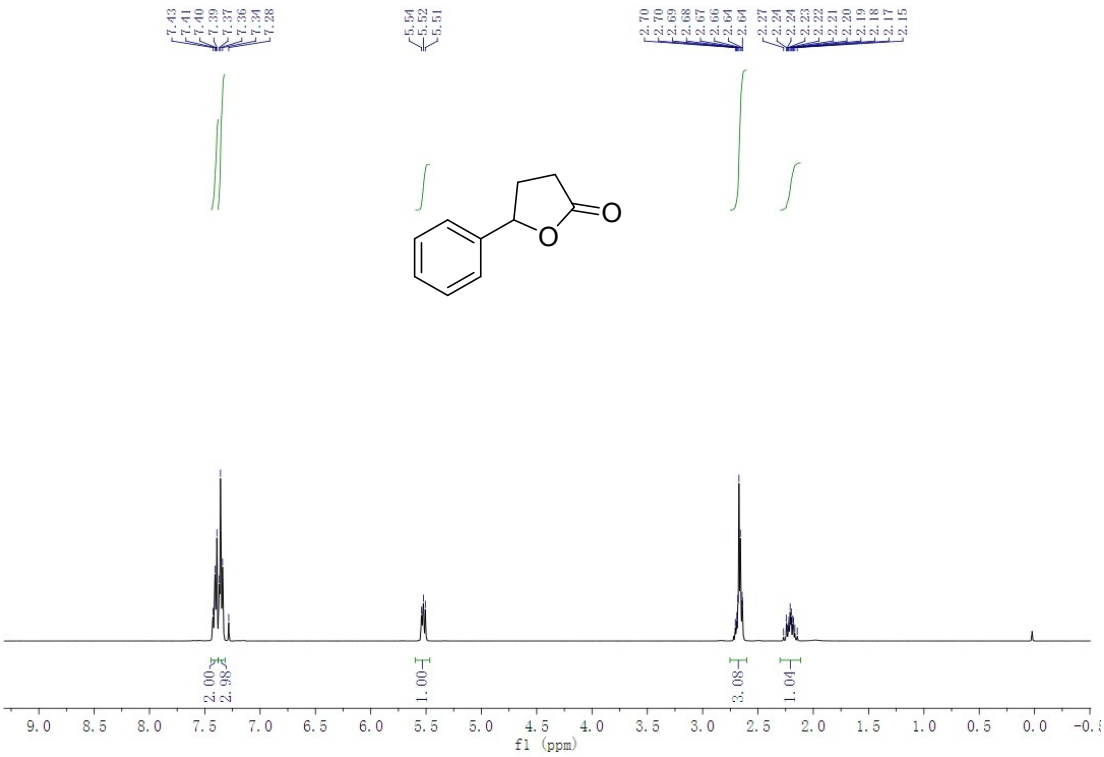
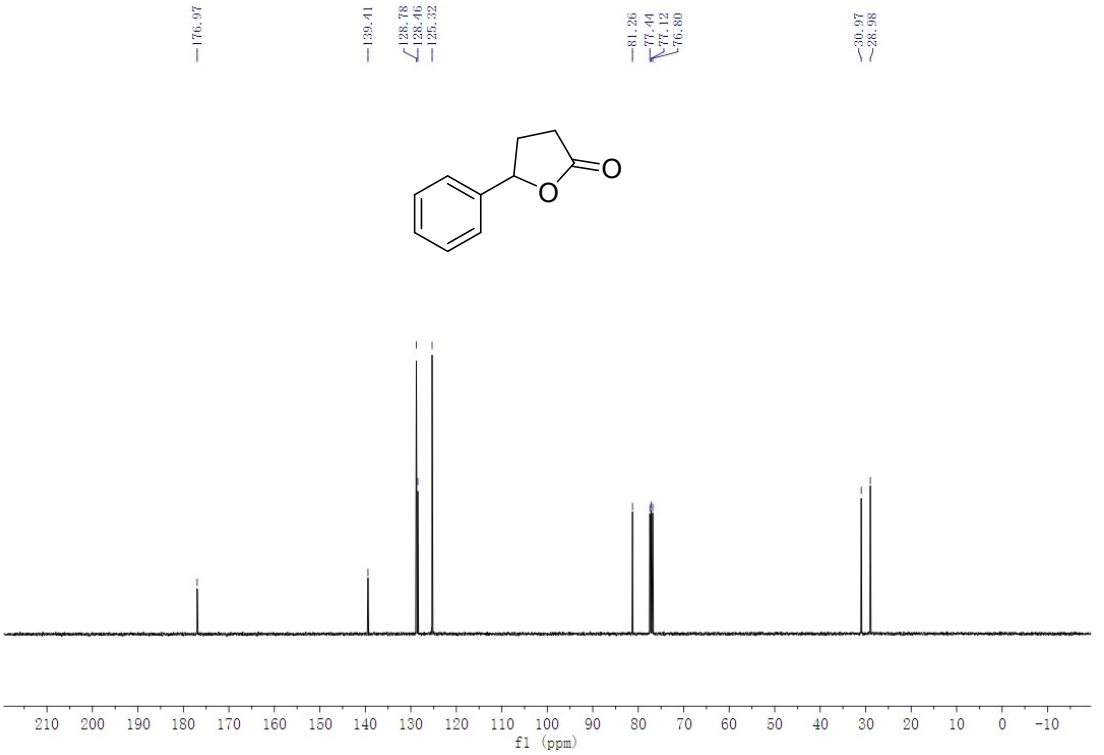
4. Copies of NMR spectra



^{1}H NMR of **2a**



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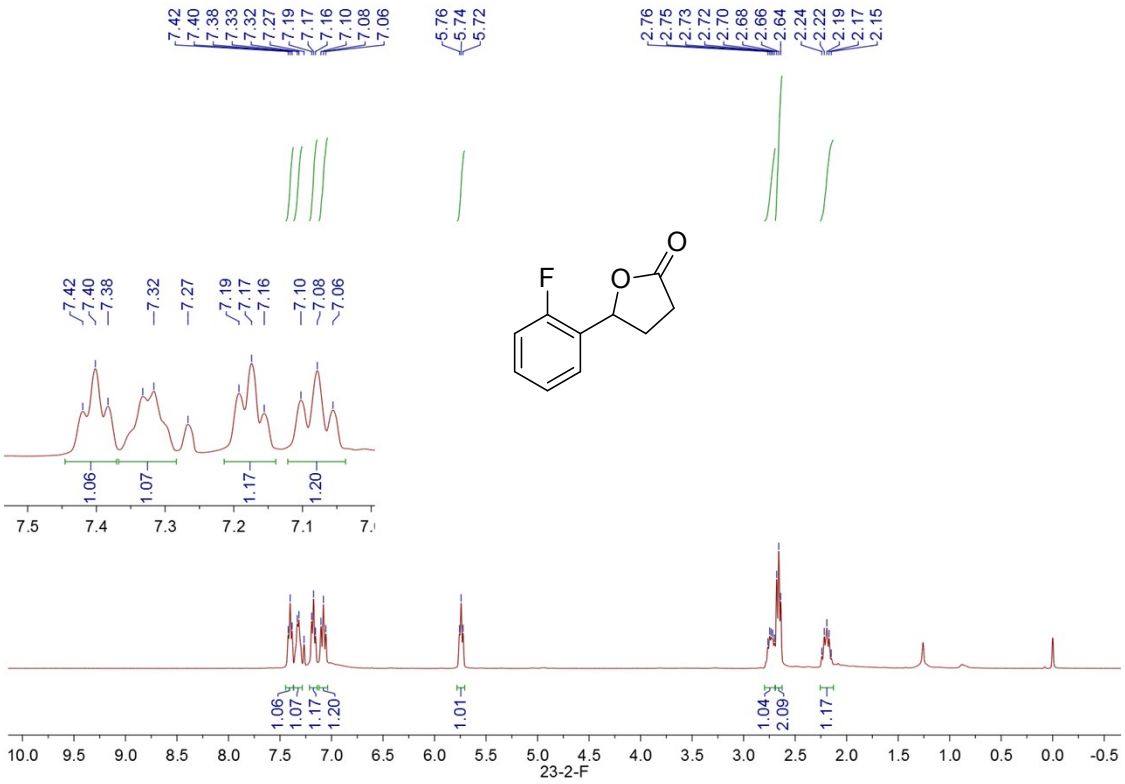
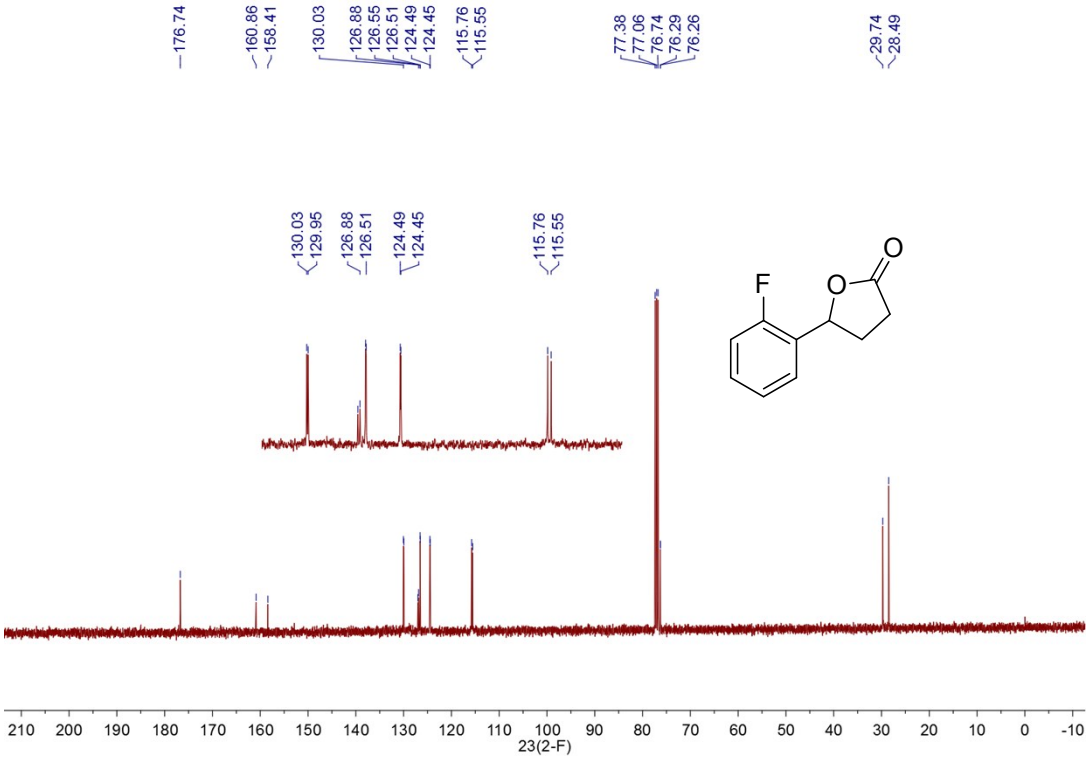


^{1}H NMR of **2b**



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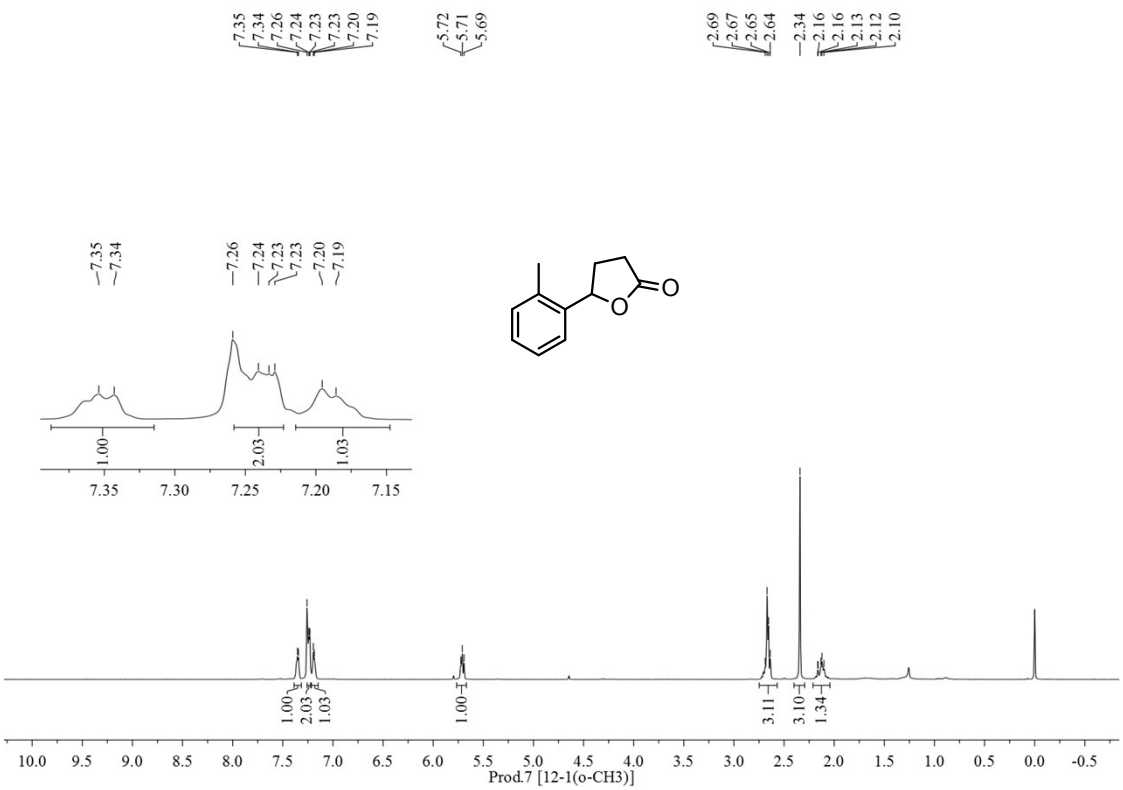
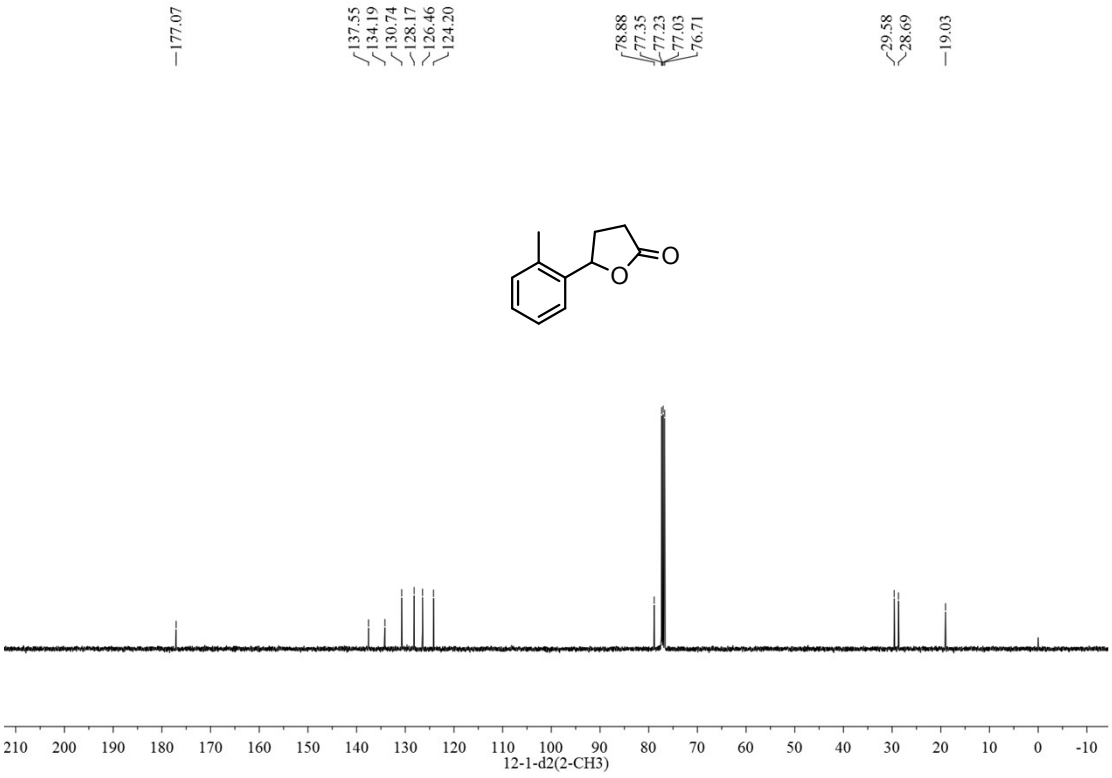


^{1}H NMR of **2c**



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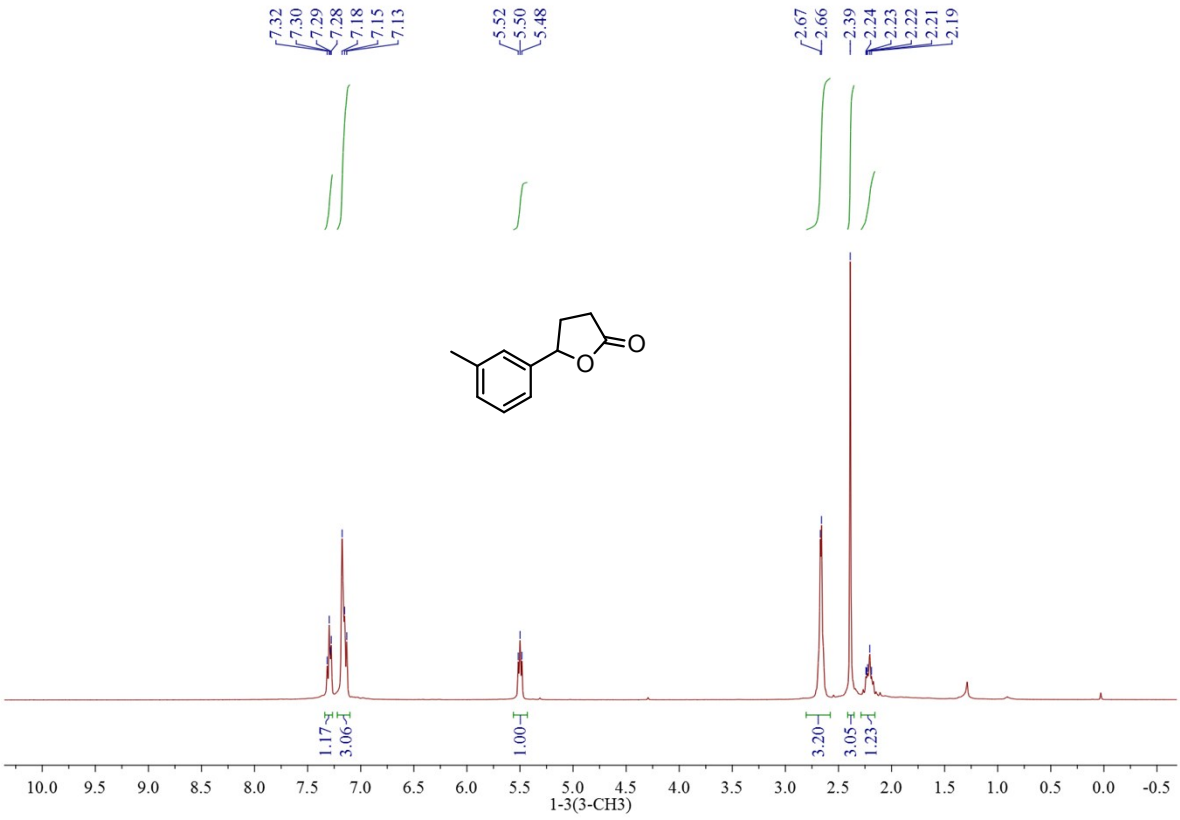
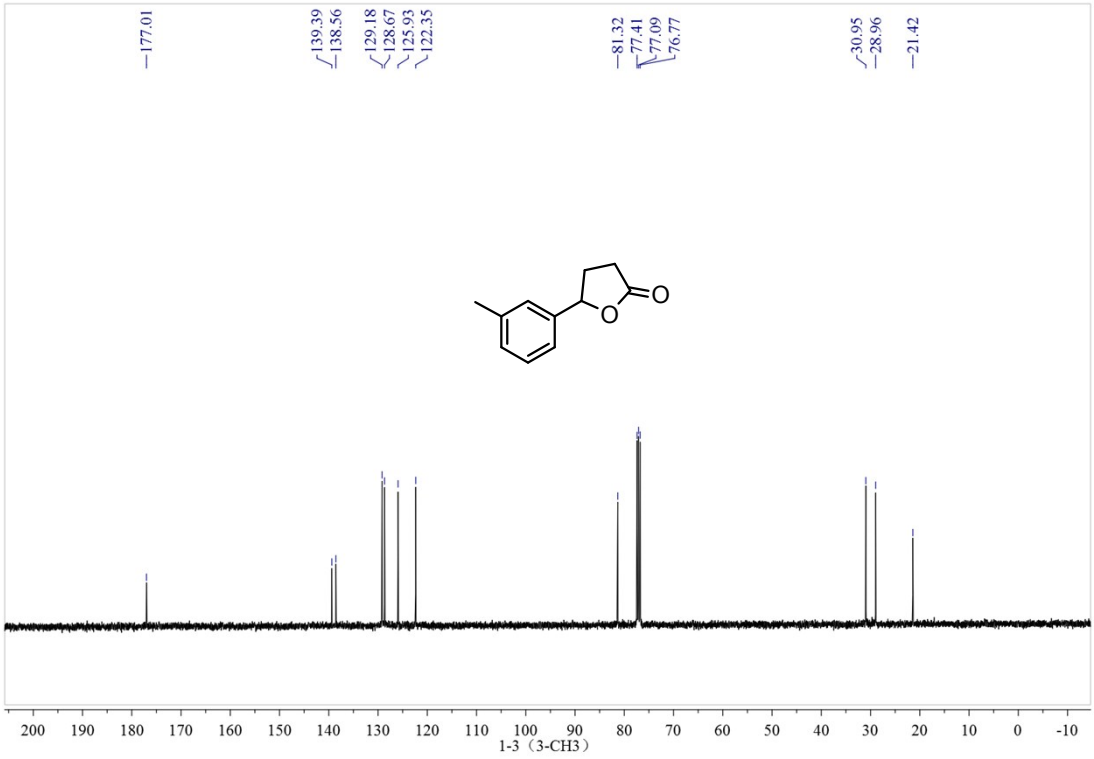
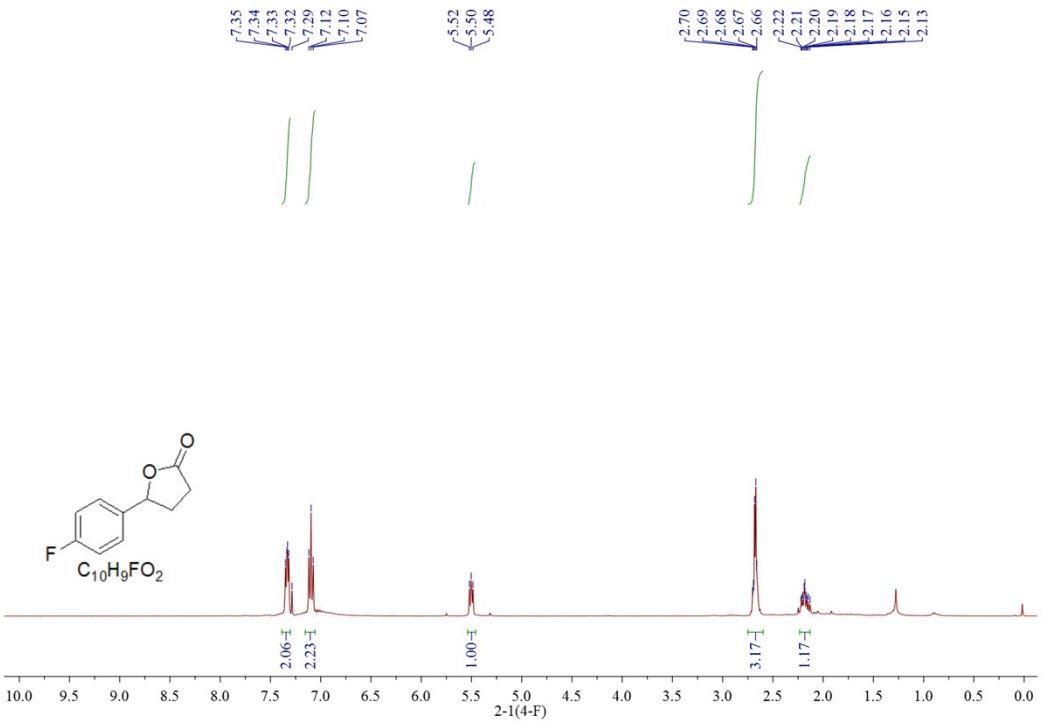


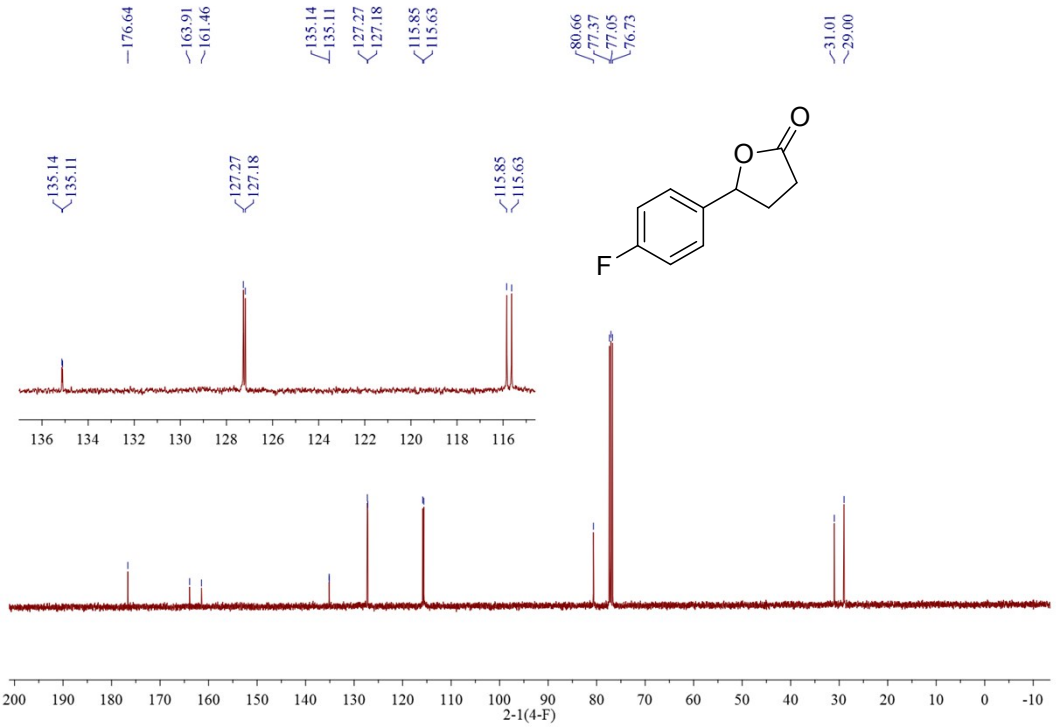
^{1}H NMR of **2d**



^{13}C NMR of **2d**



^{1}H NMR of **2e**



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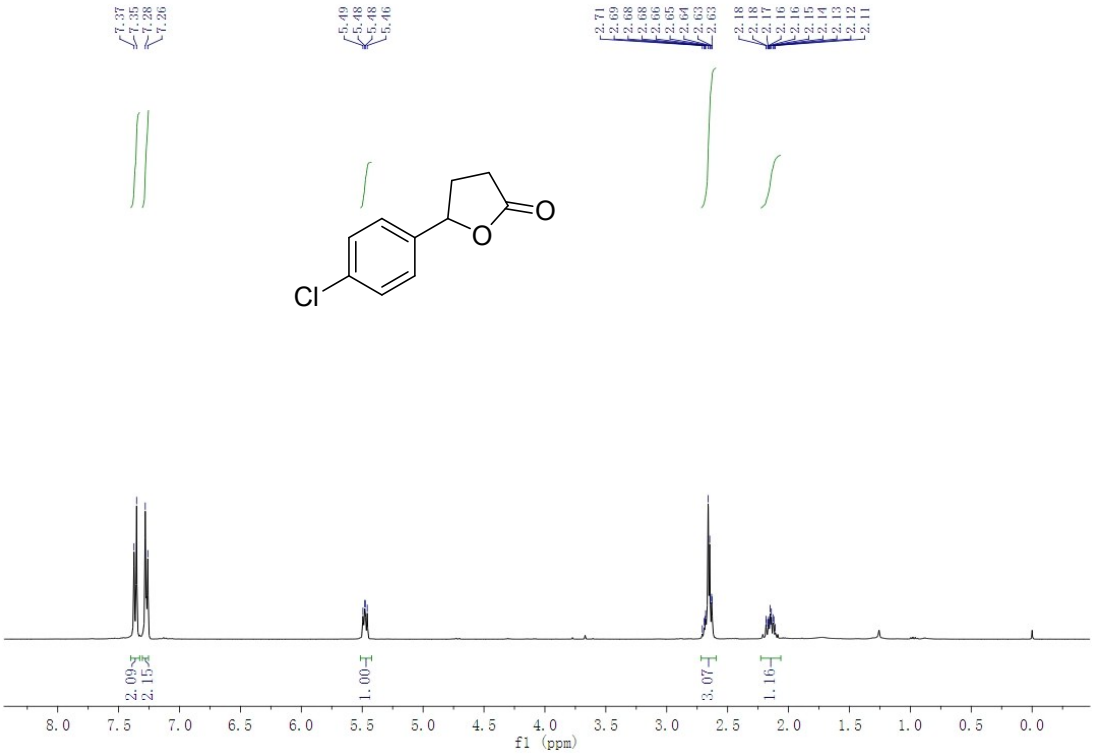

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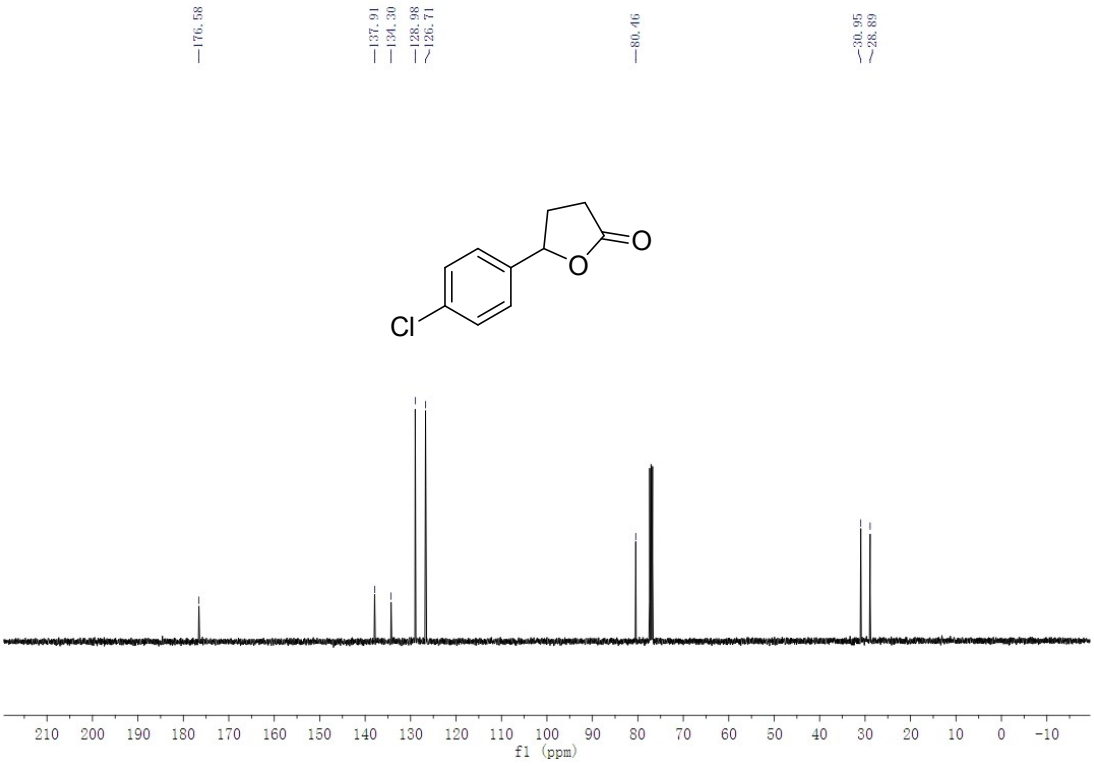
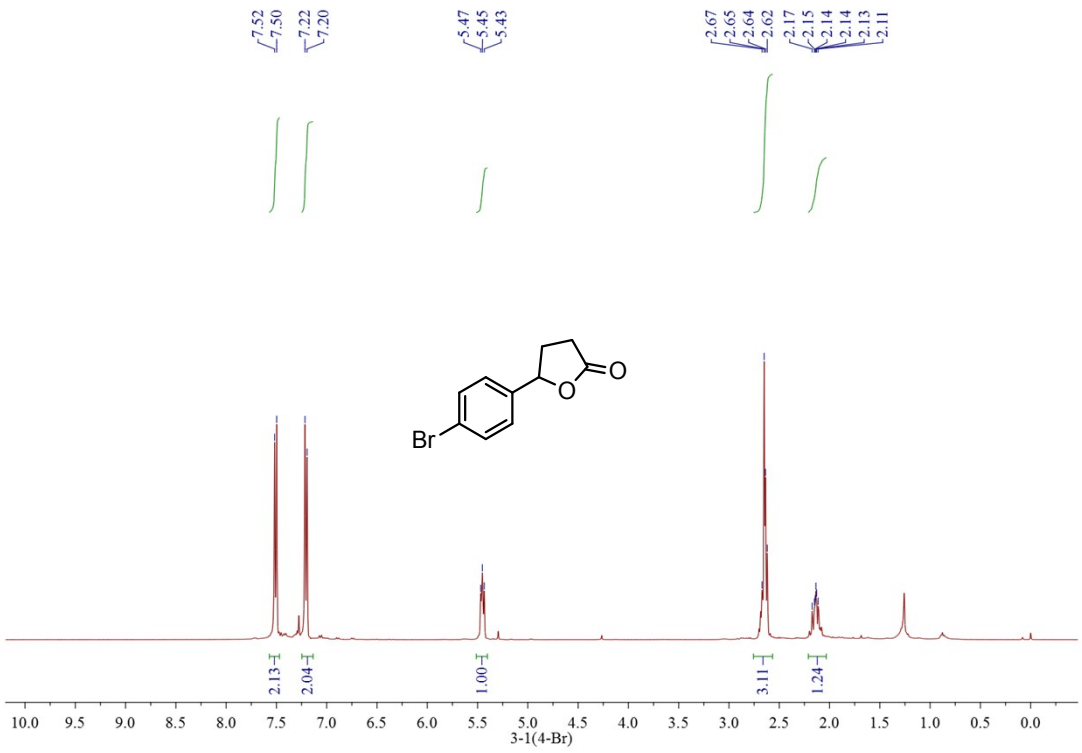
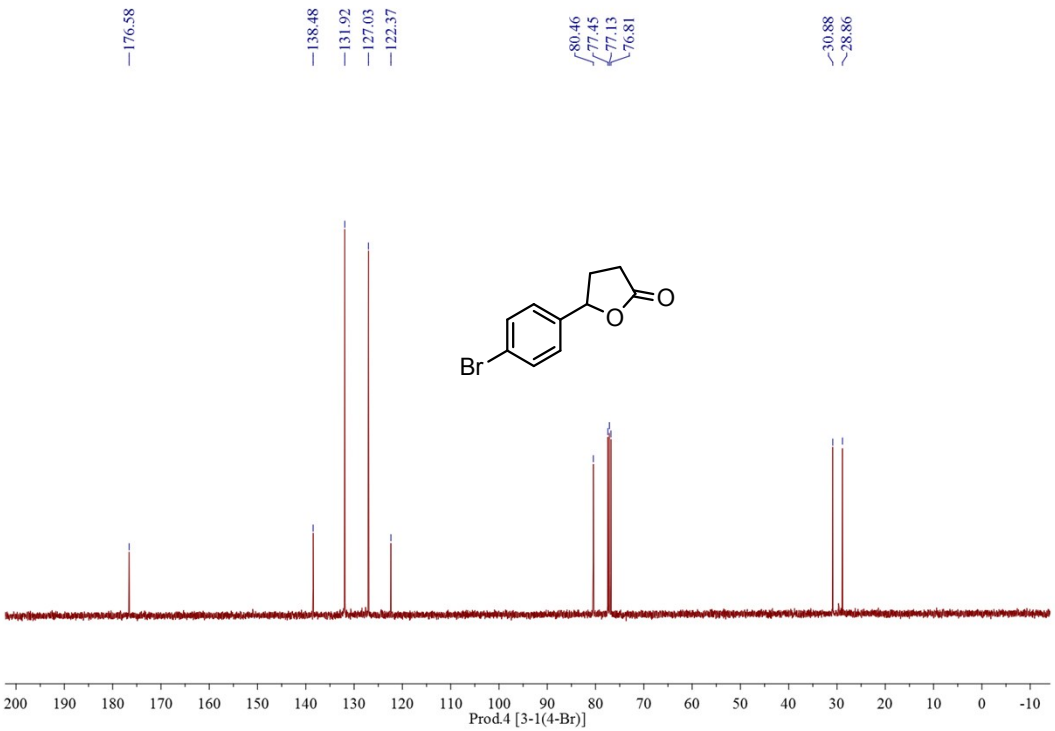


^{13}C NMR of **2f**



^{1}H NMR of **2g**



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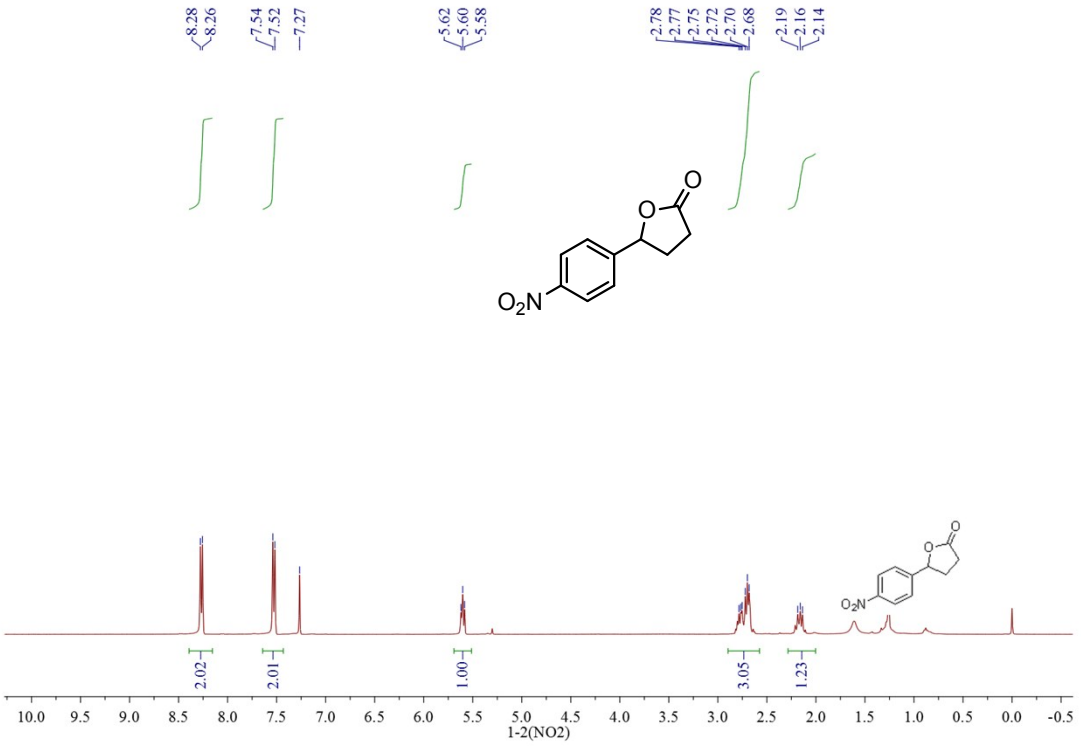

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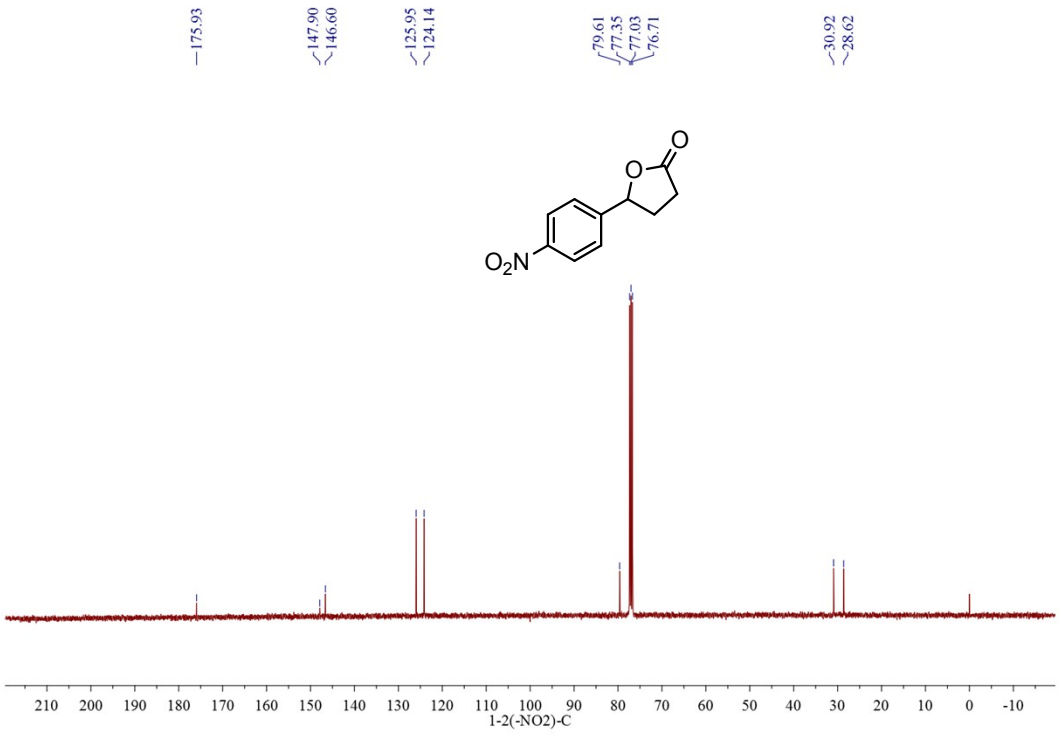

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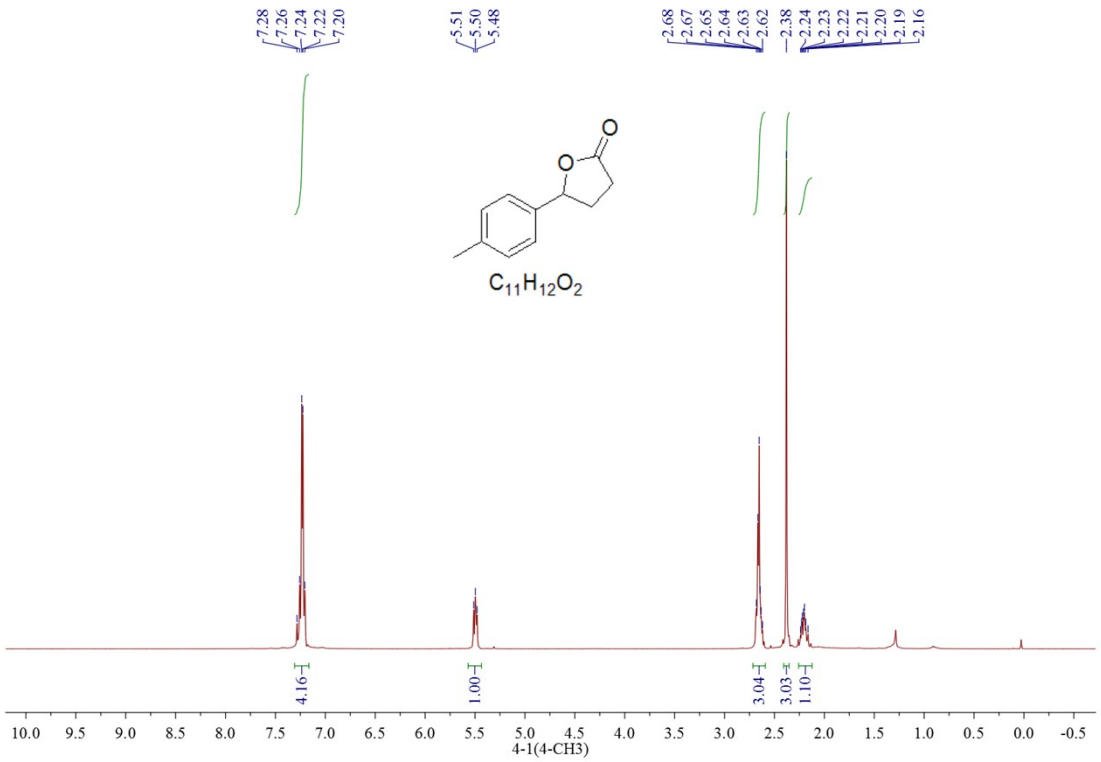

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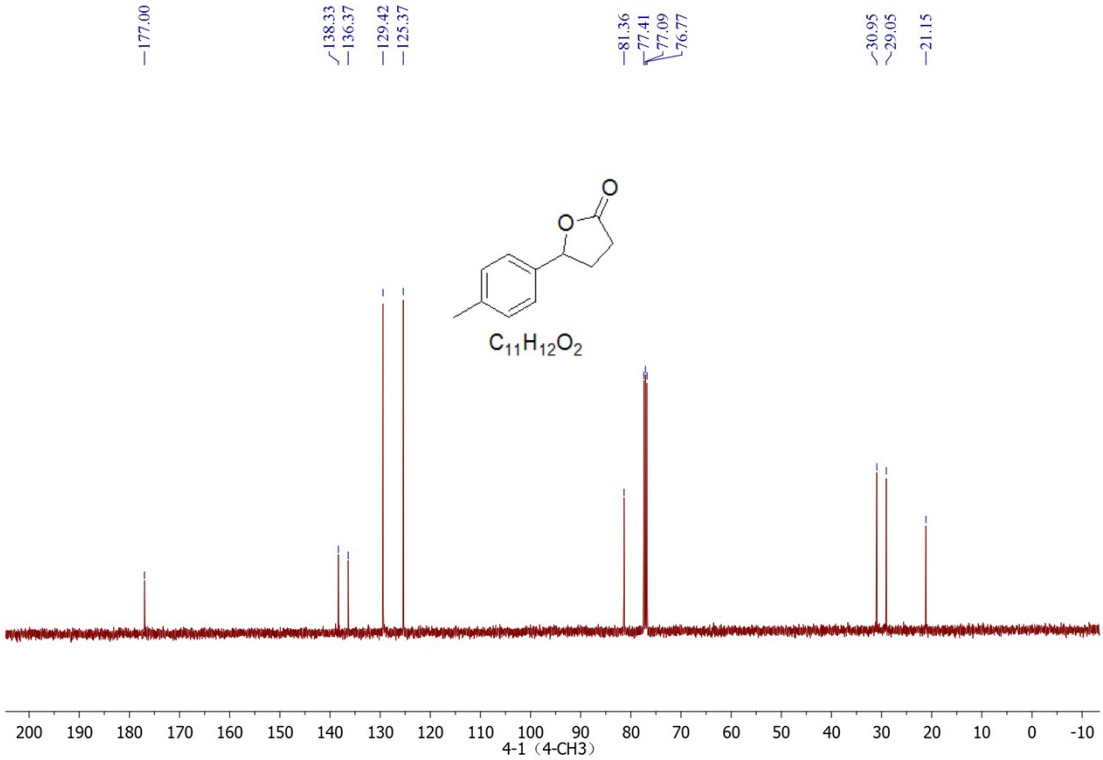

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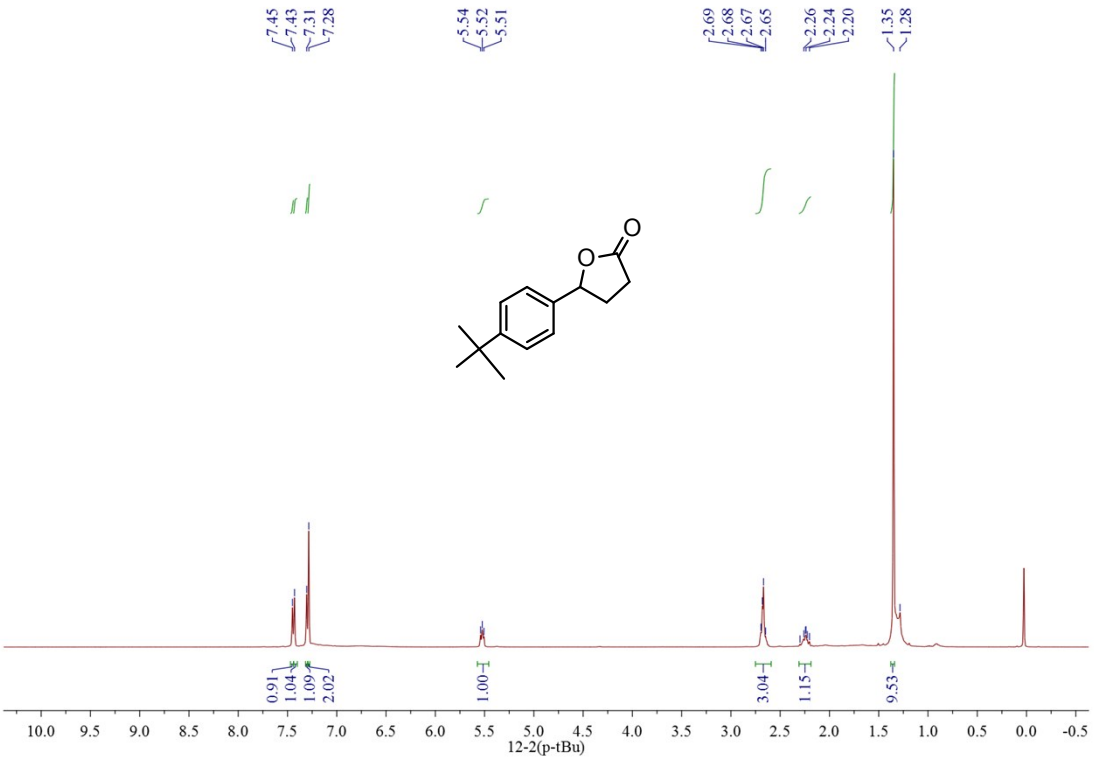

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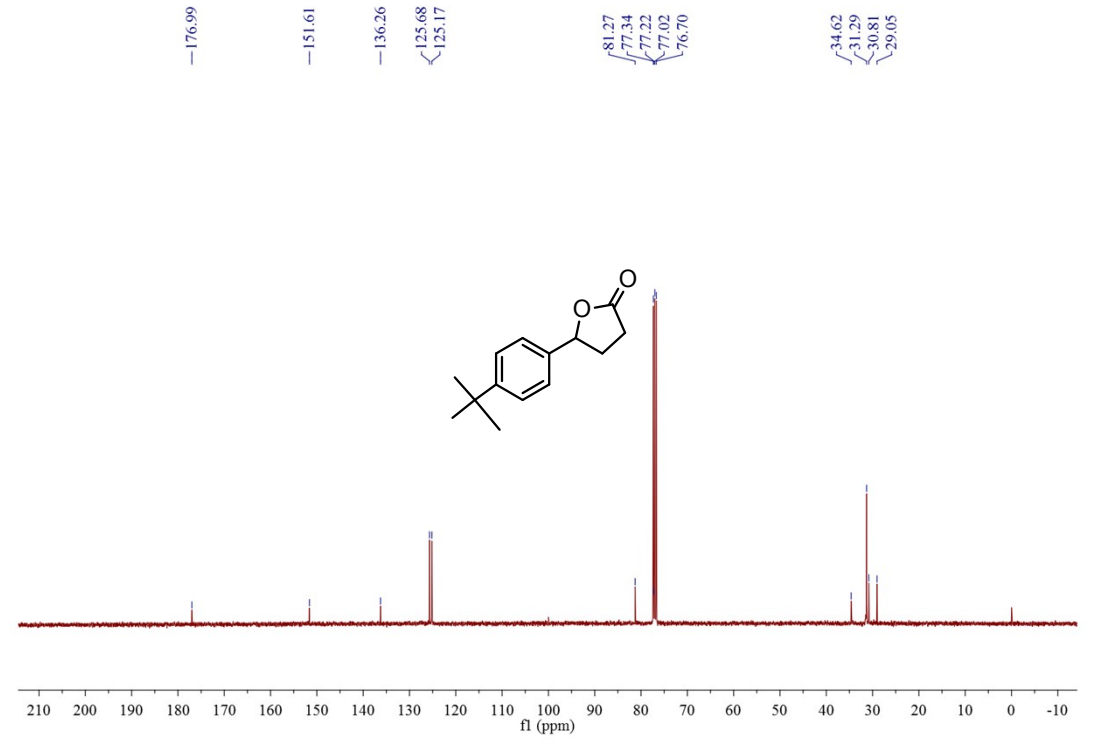

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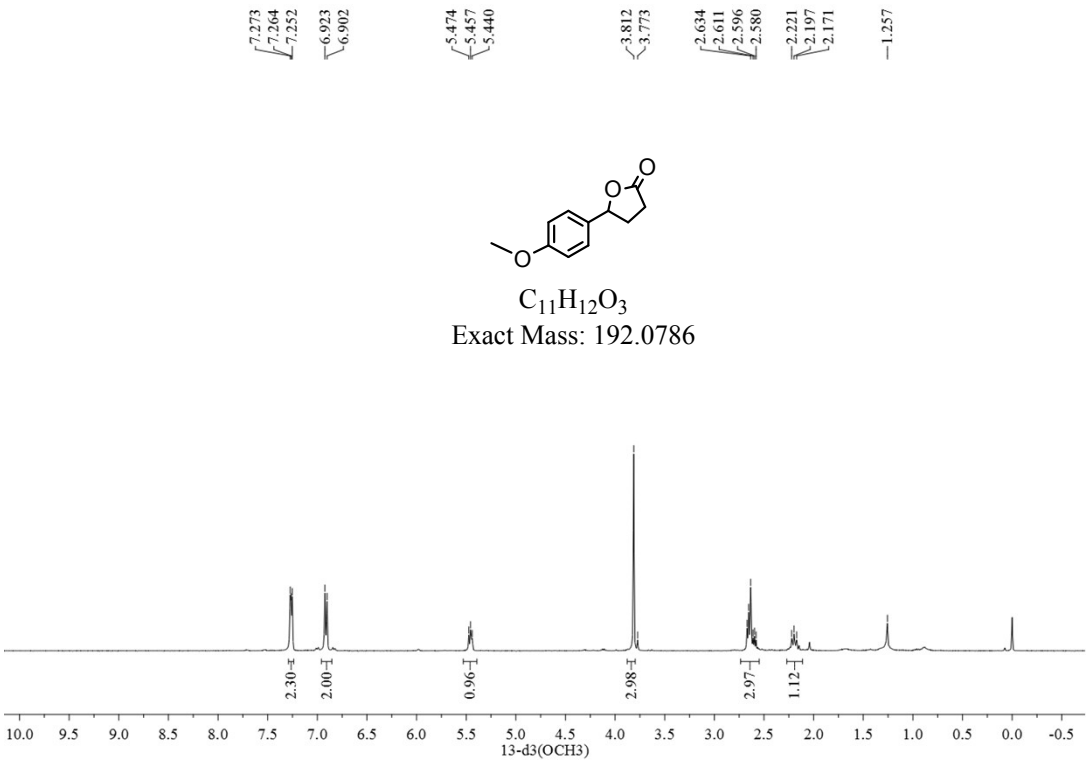
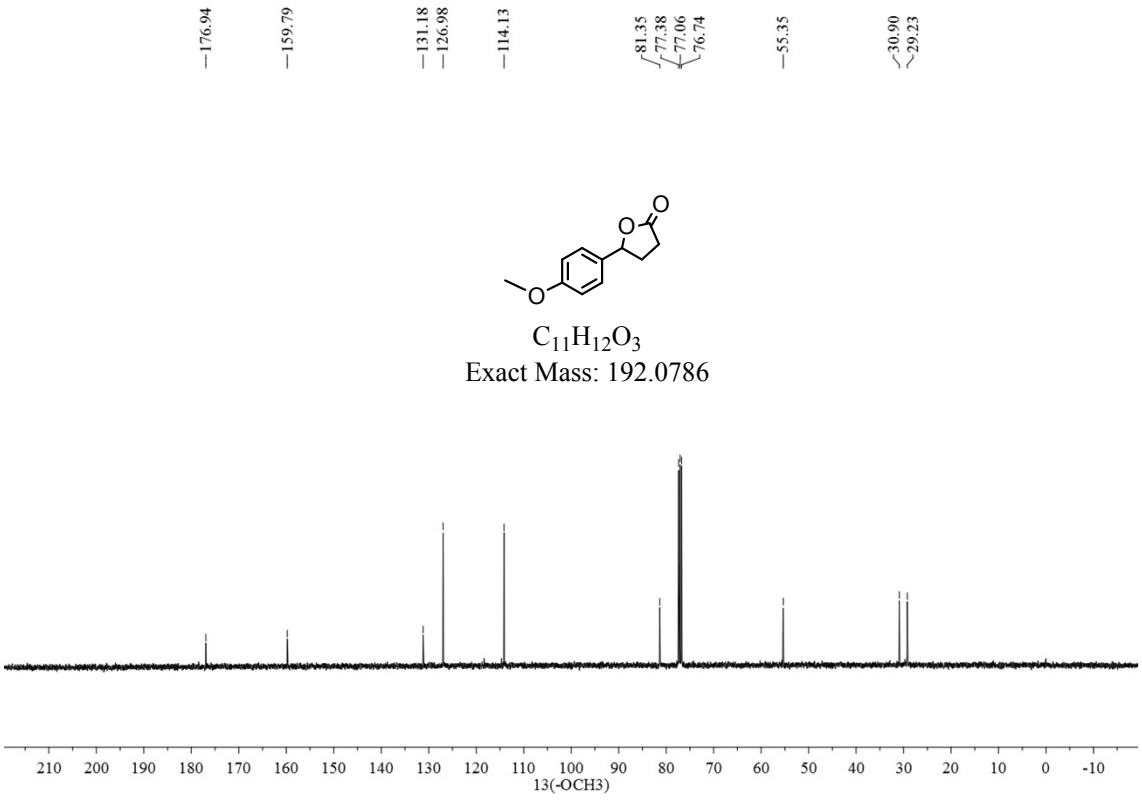


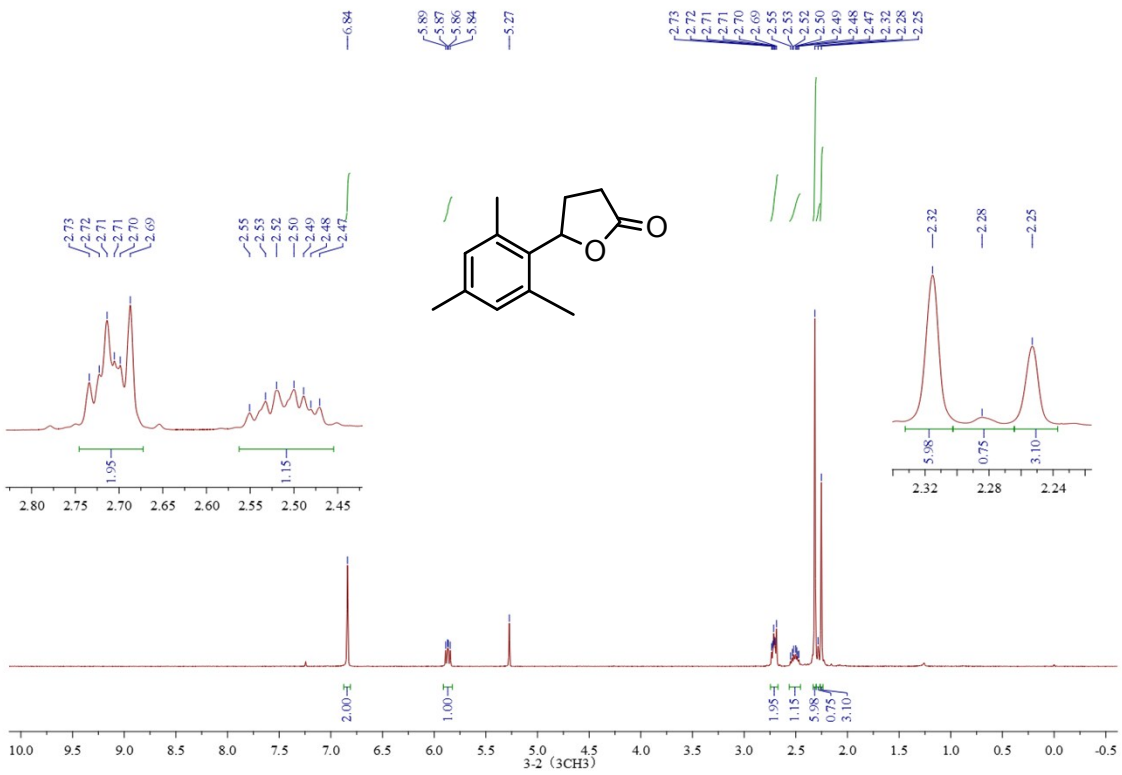
$C_{11}H_{12}O_3$
Exact Mass: 192.0786

^{1}H NMR of **2k**



$C_{11}H_{12}O_3$
Exact Mass: 192.0786

^{13}C NMR of **2k**



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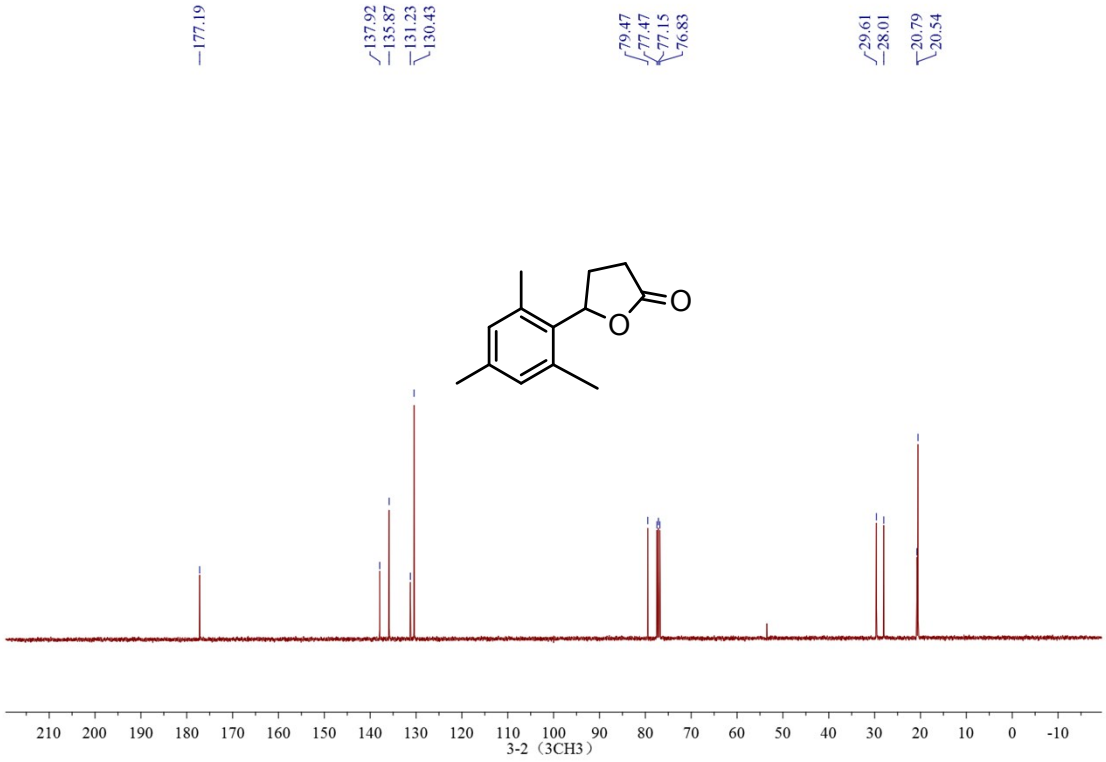

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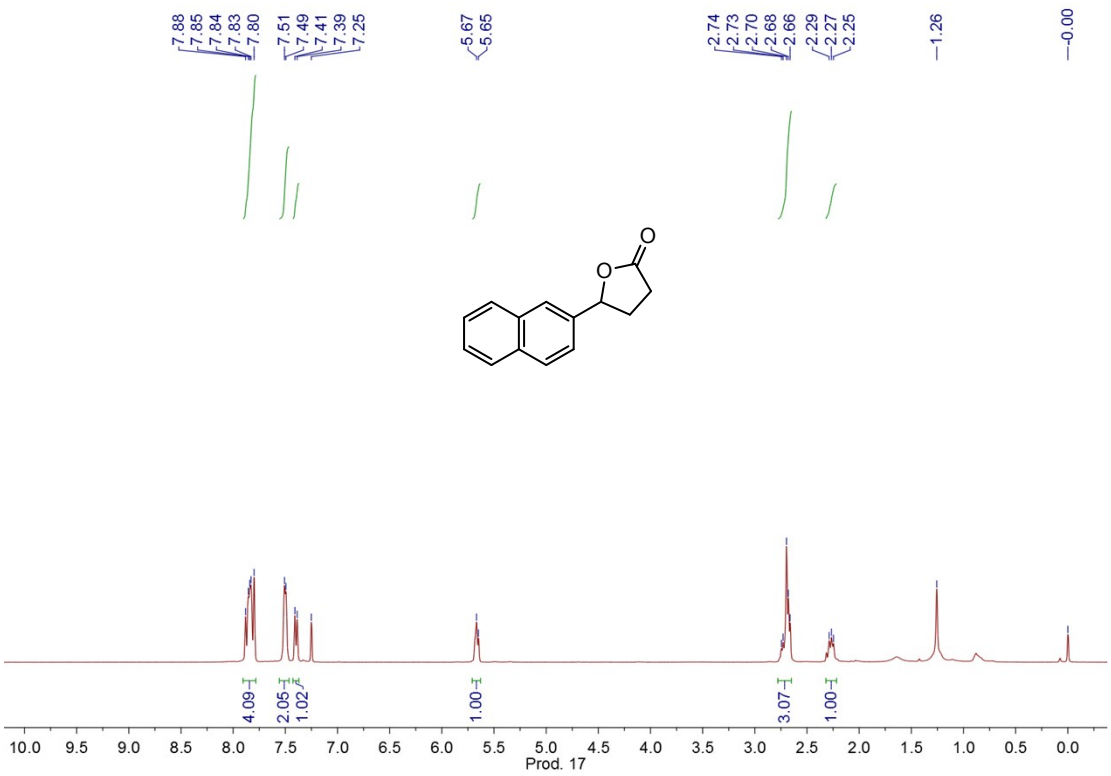

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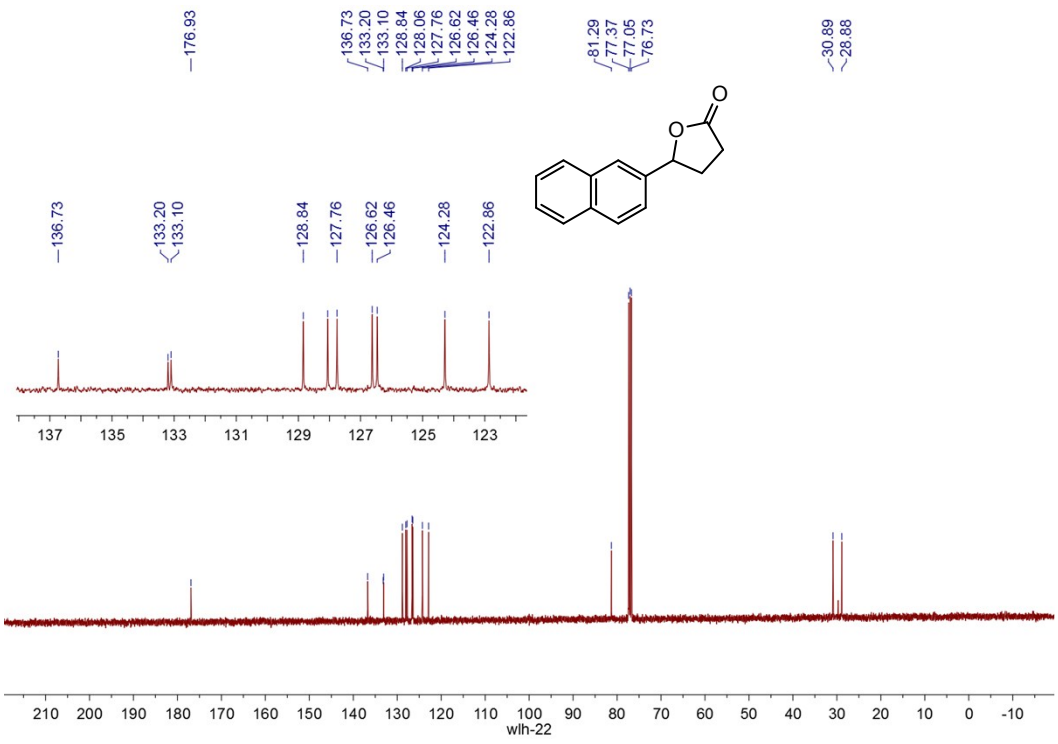

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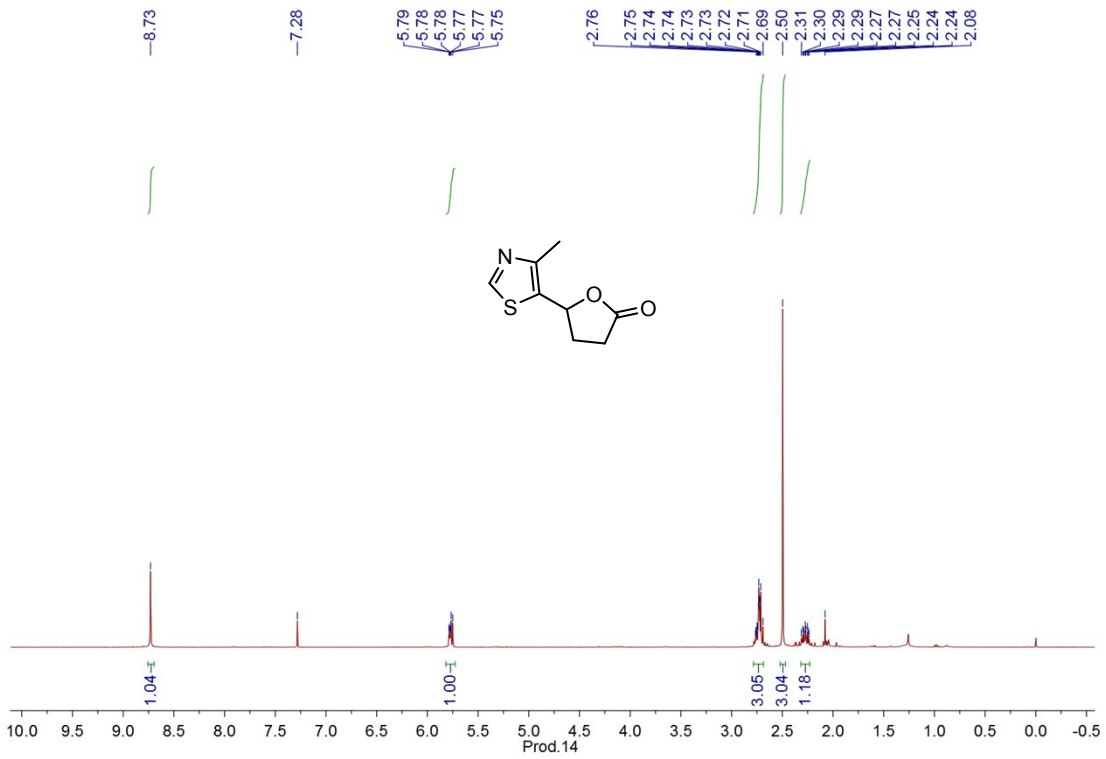

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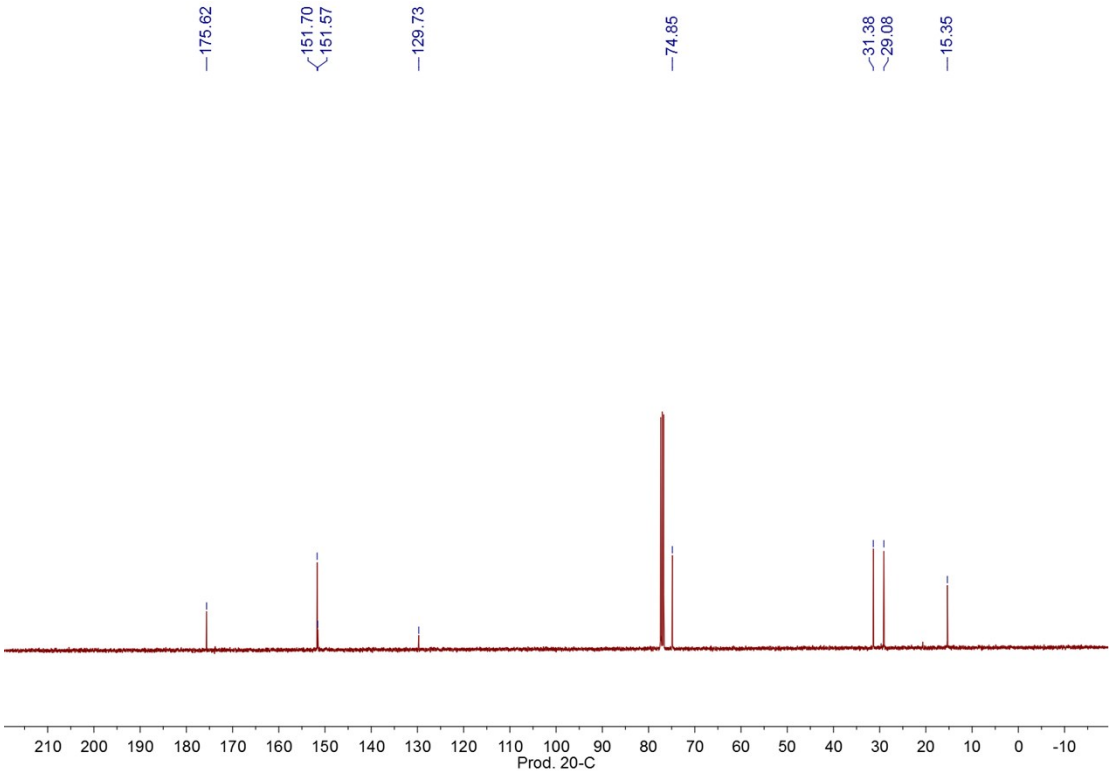

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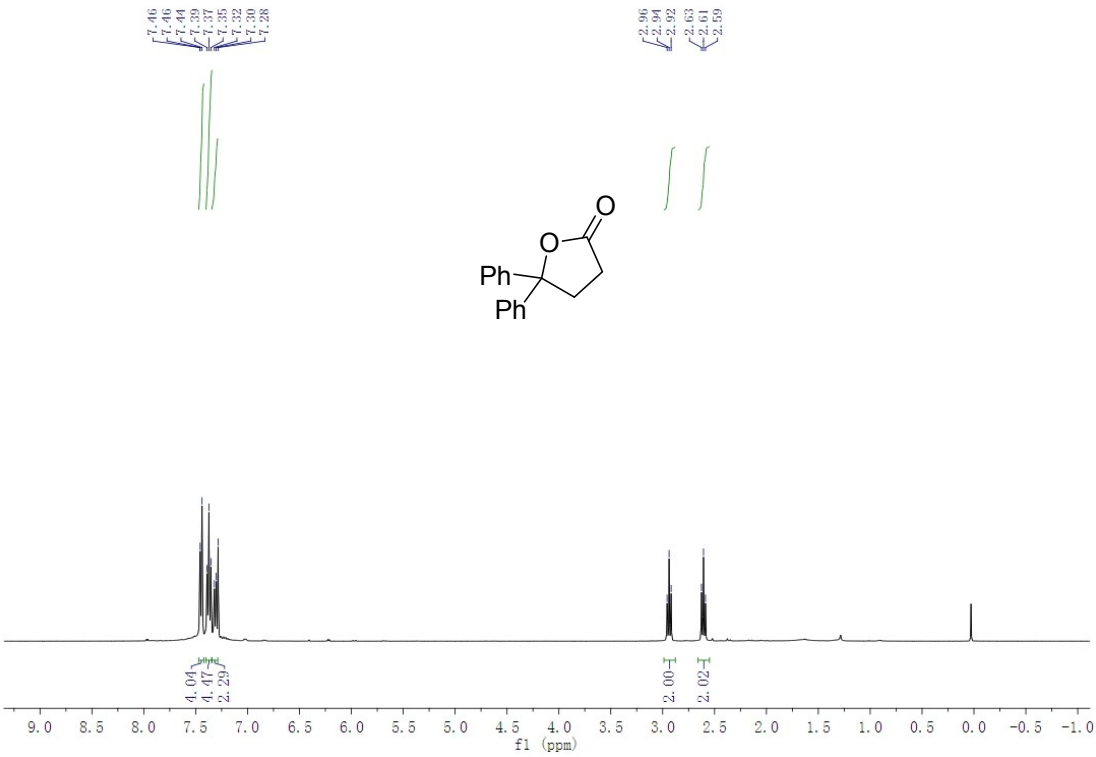

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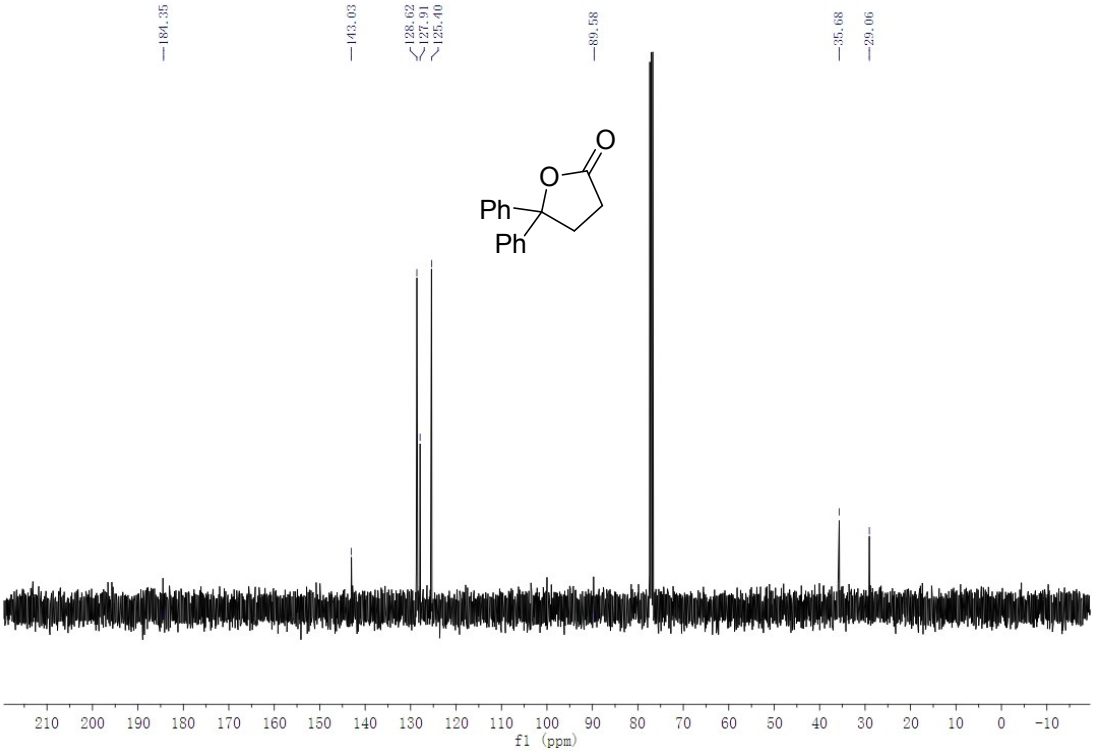

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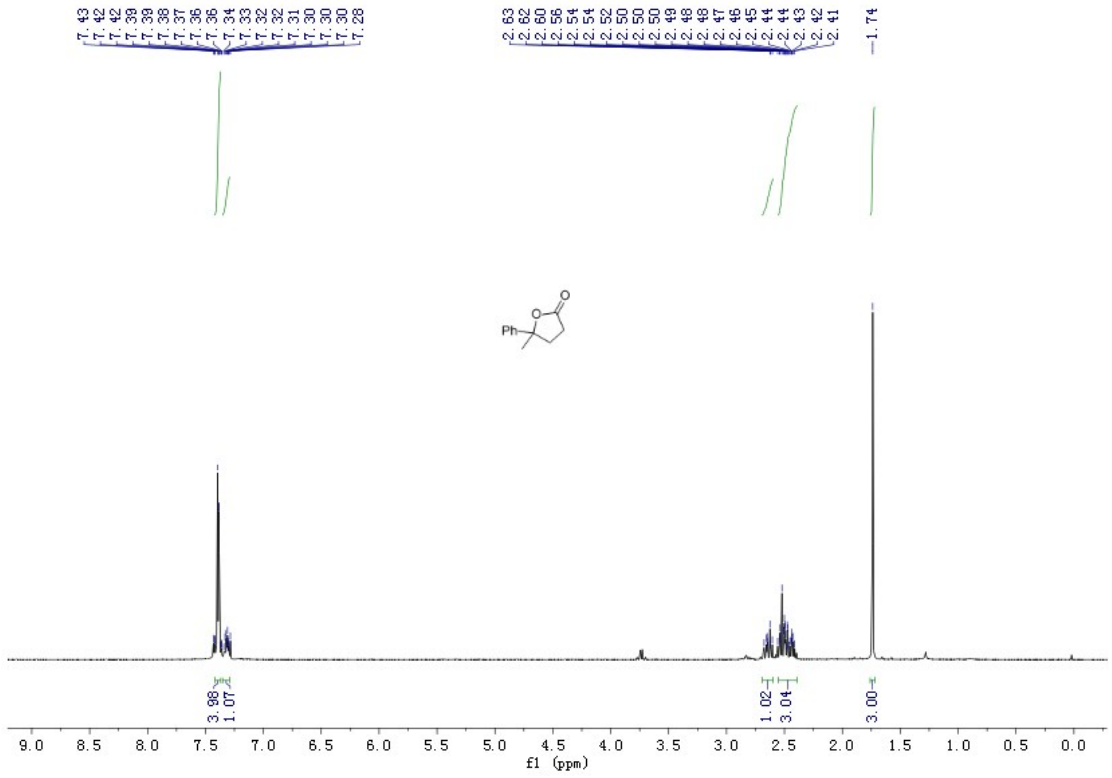

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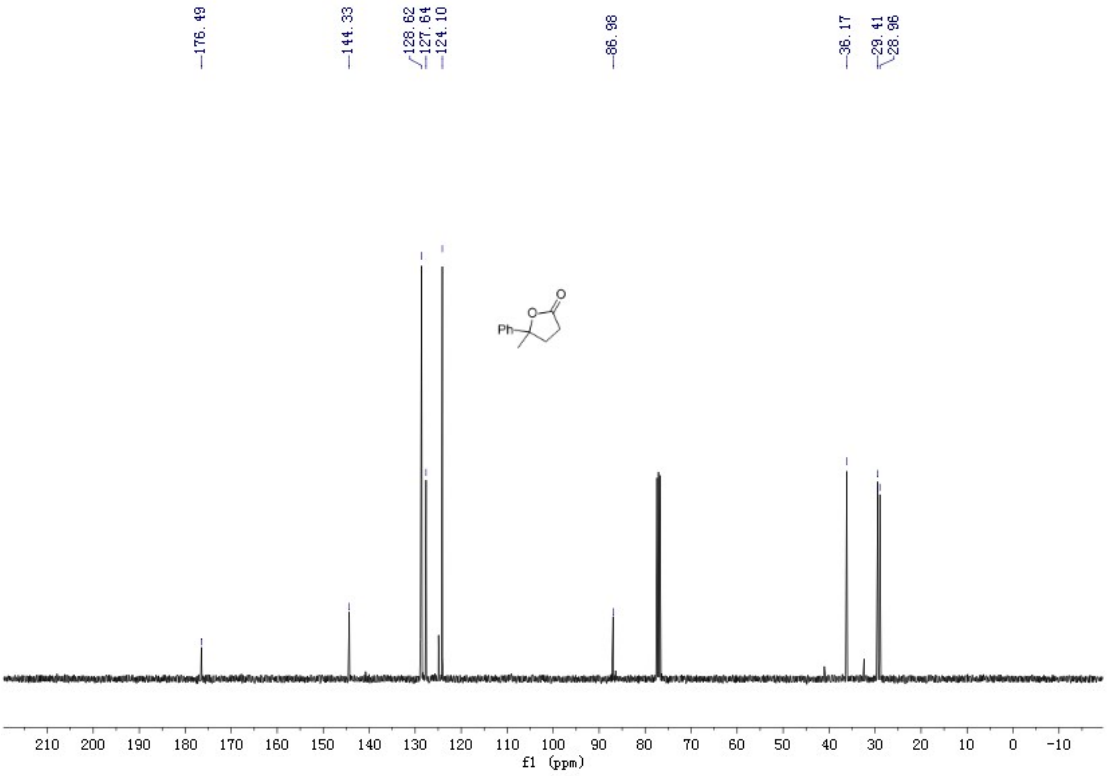
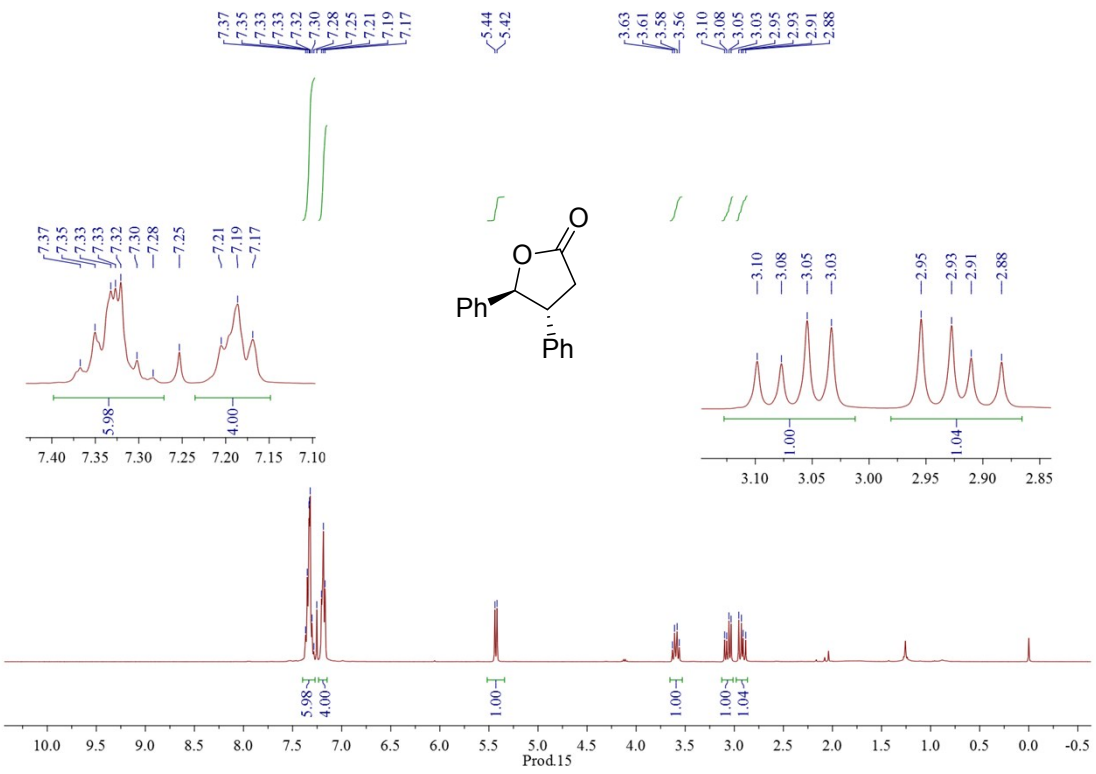
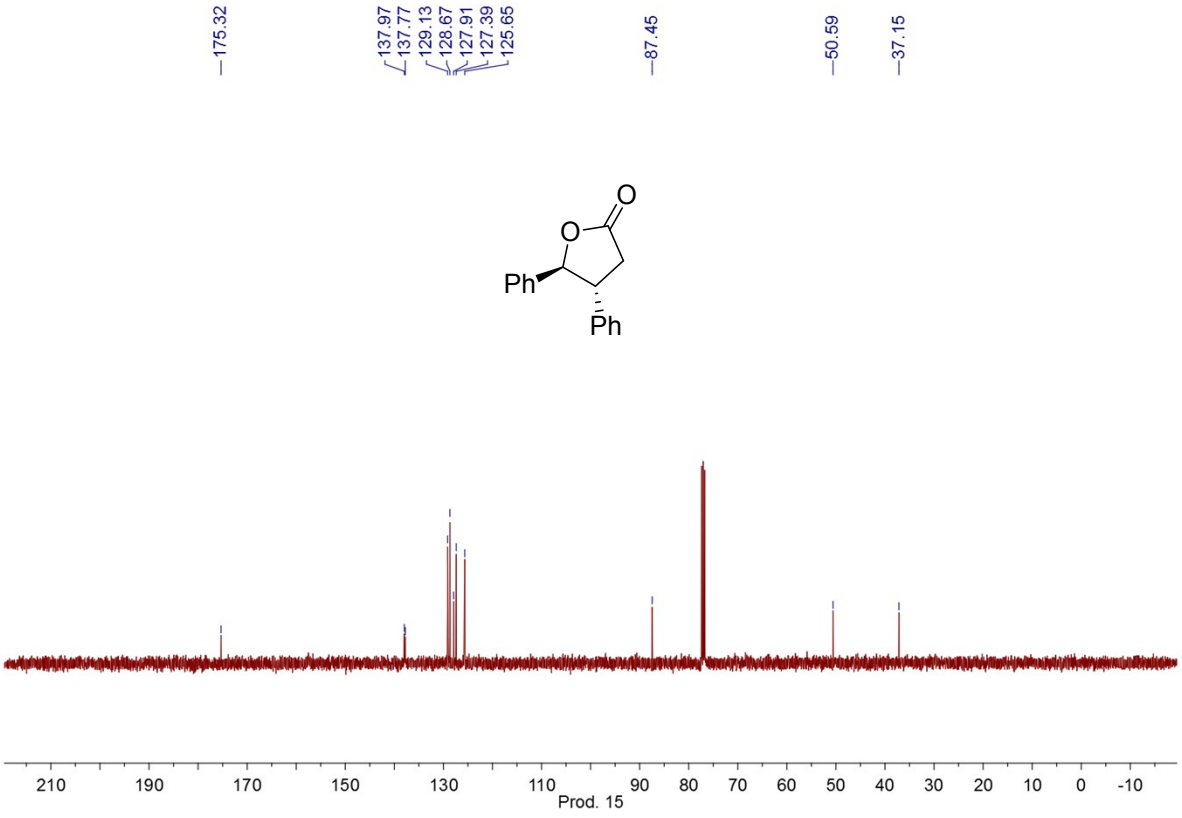


^{13}C NMR of **2p**



^{1}H NMR of **2q-1**



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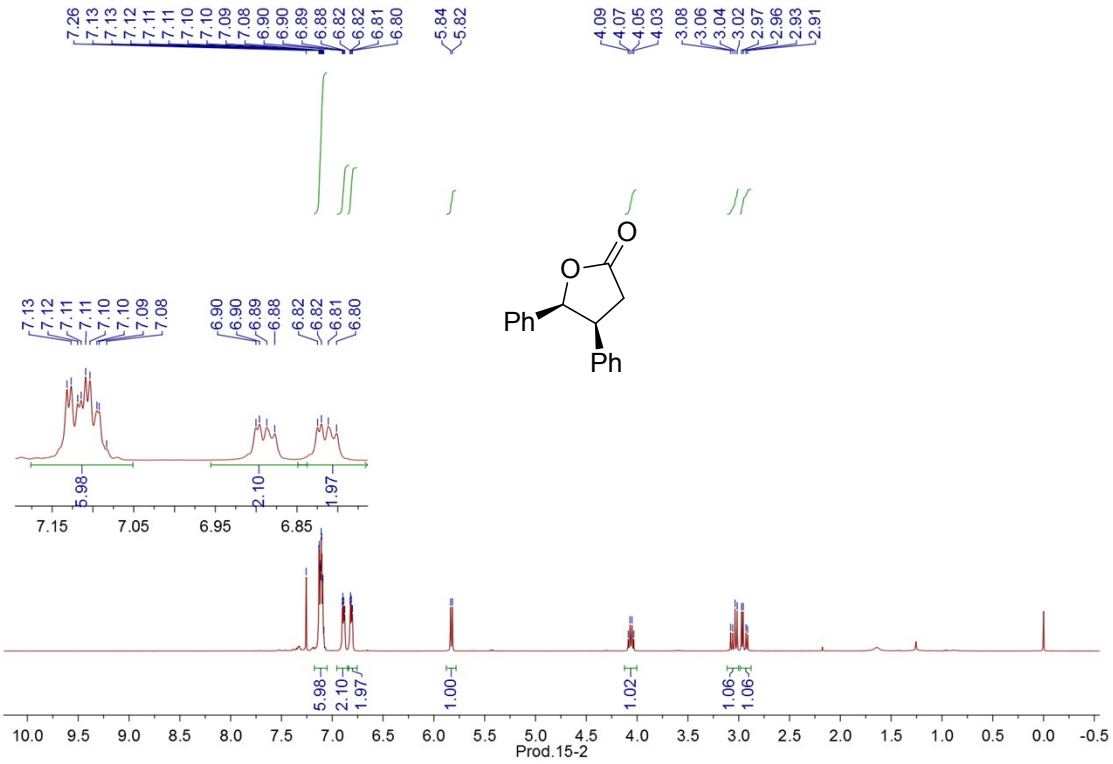
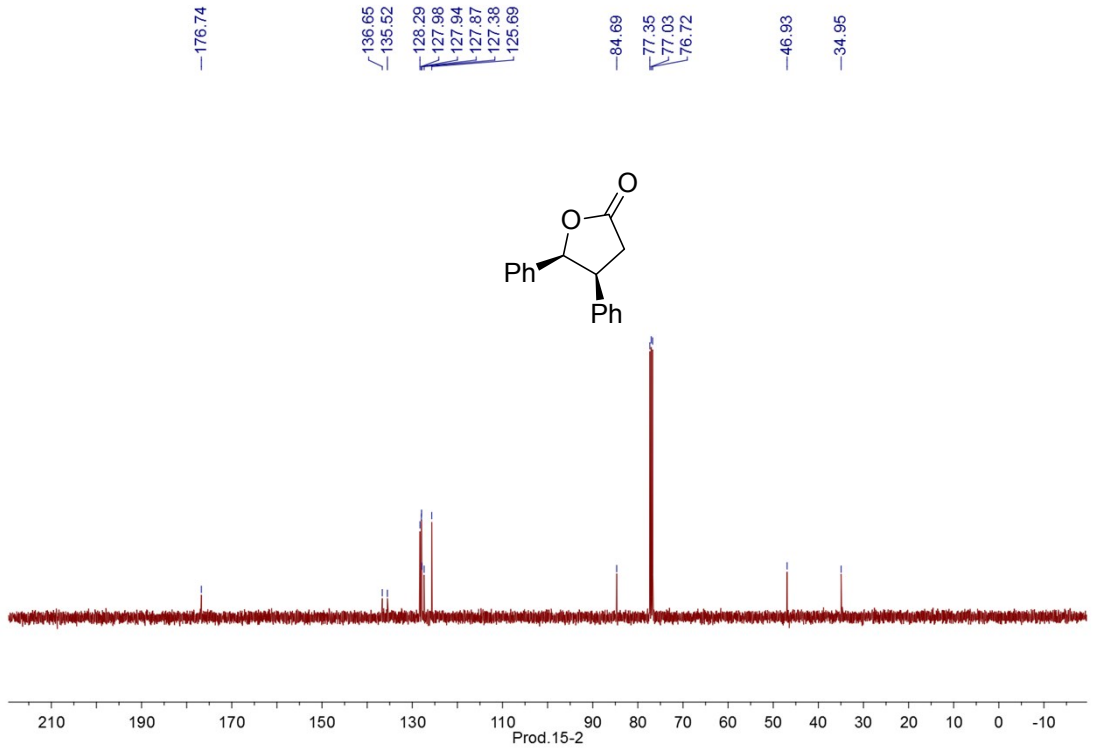


^{1}H NMR of **2q-2**



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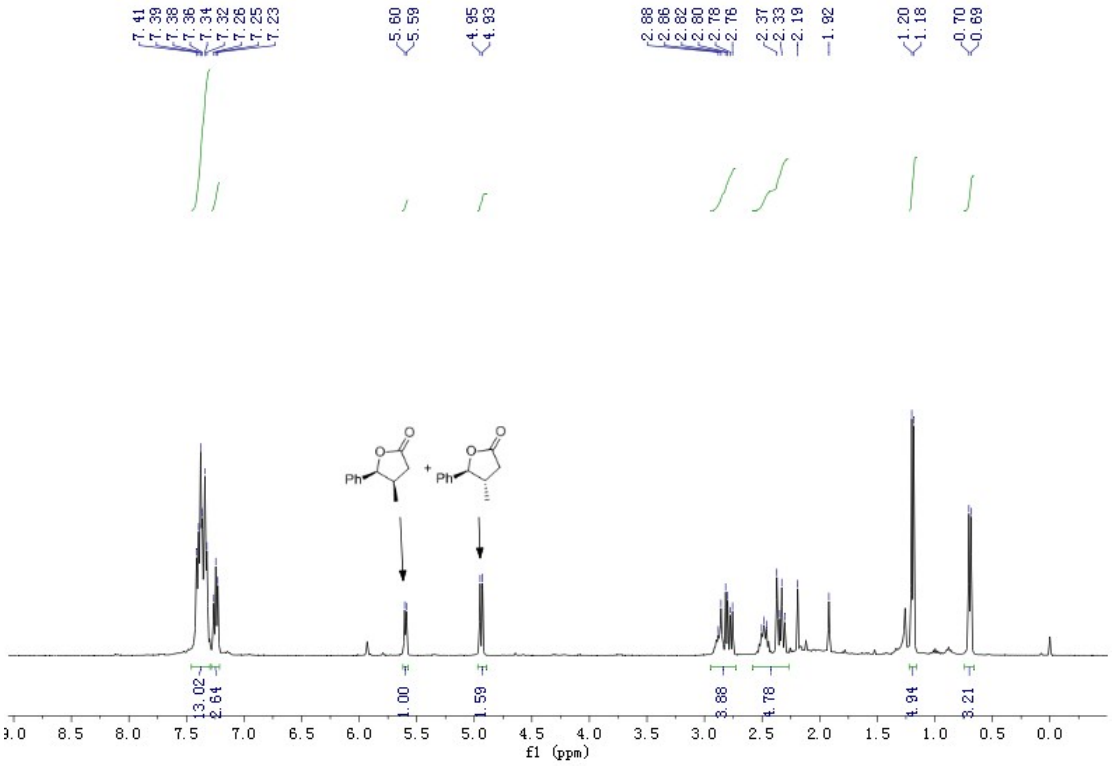

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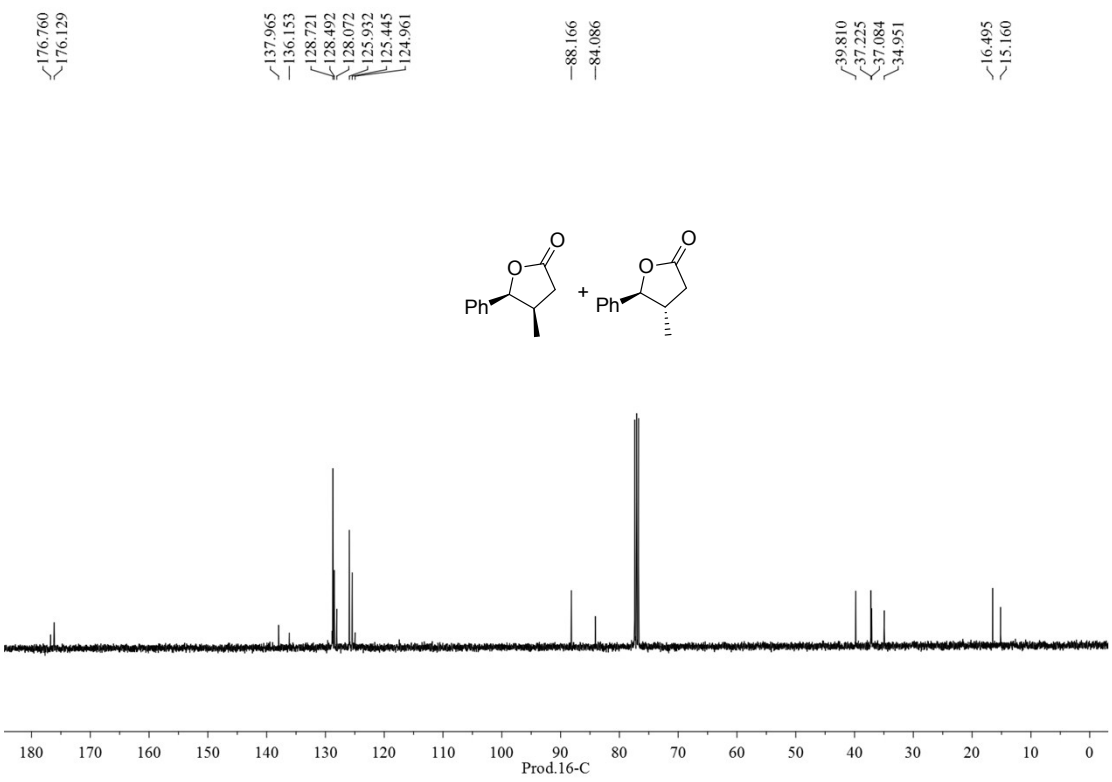

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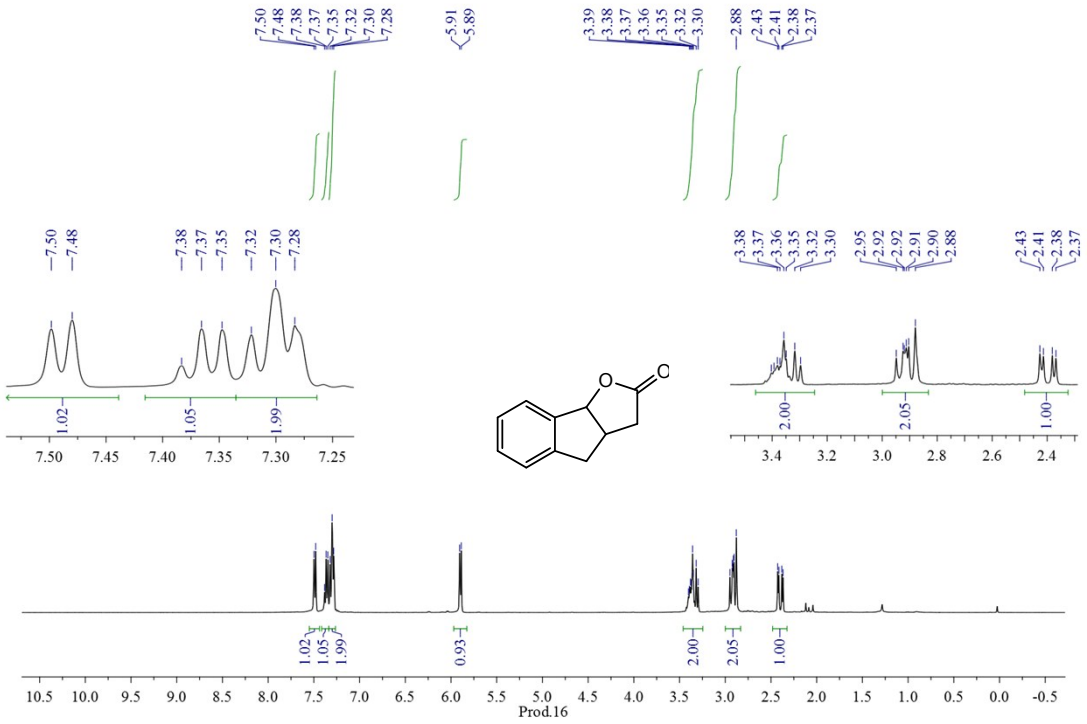

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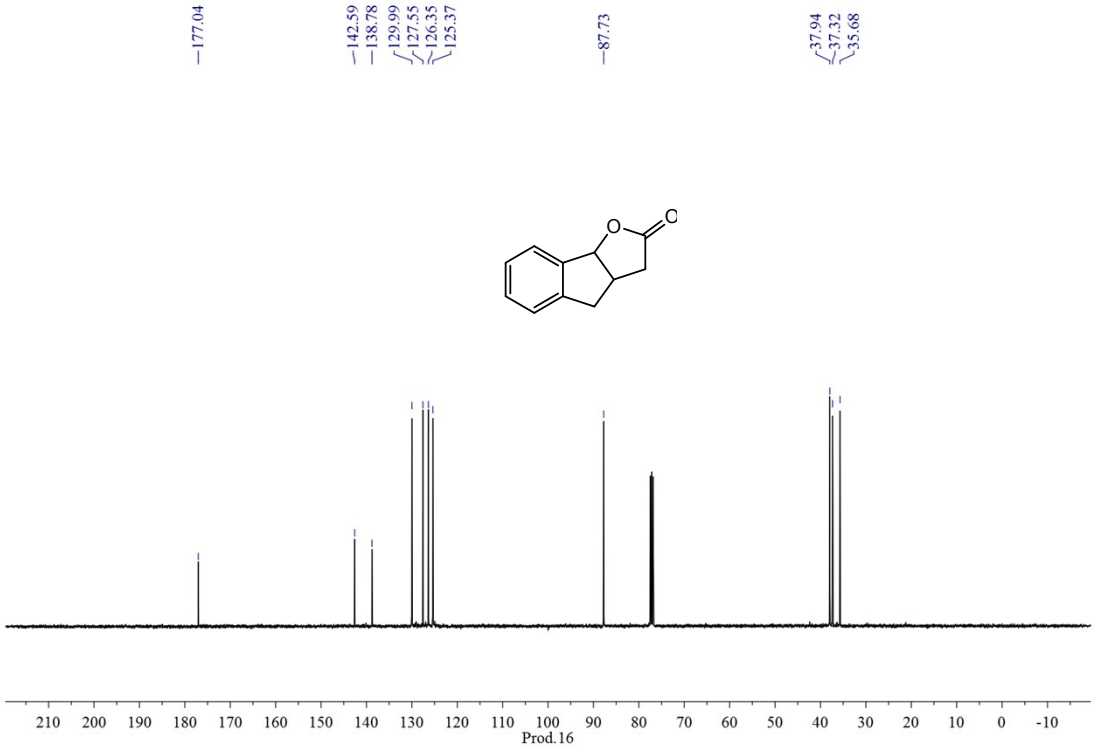
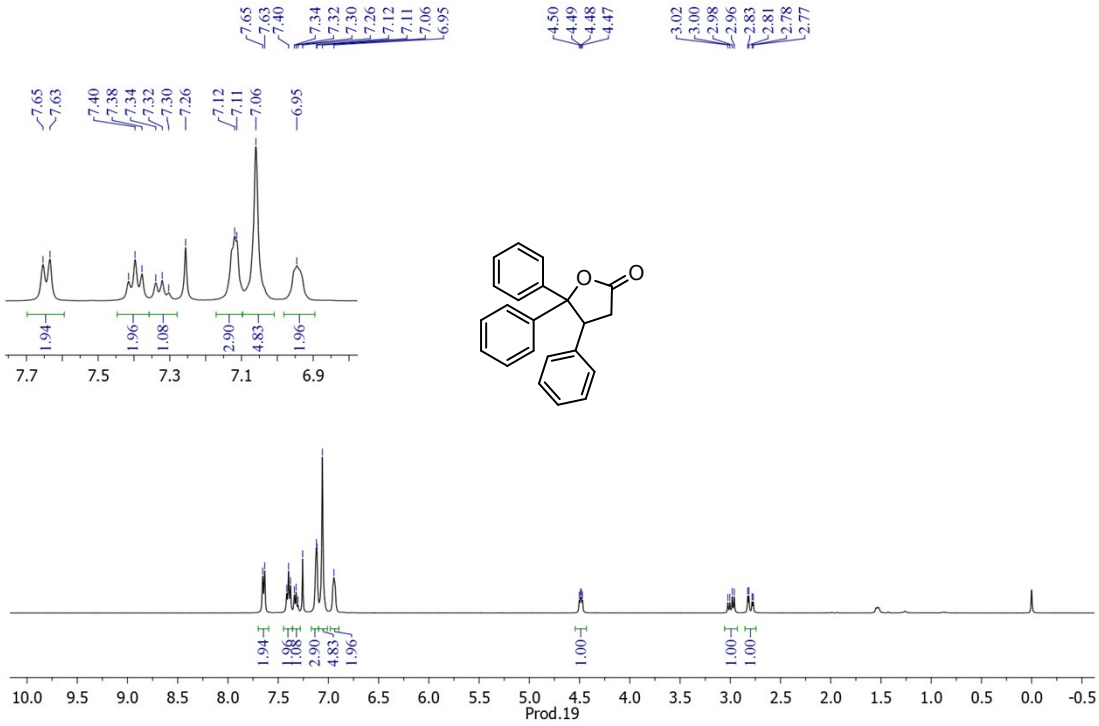


^{13}C NMR of **2s**



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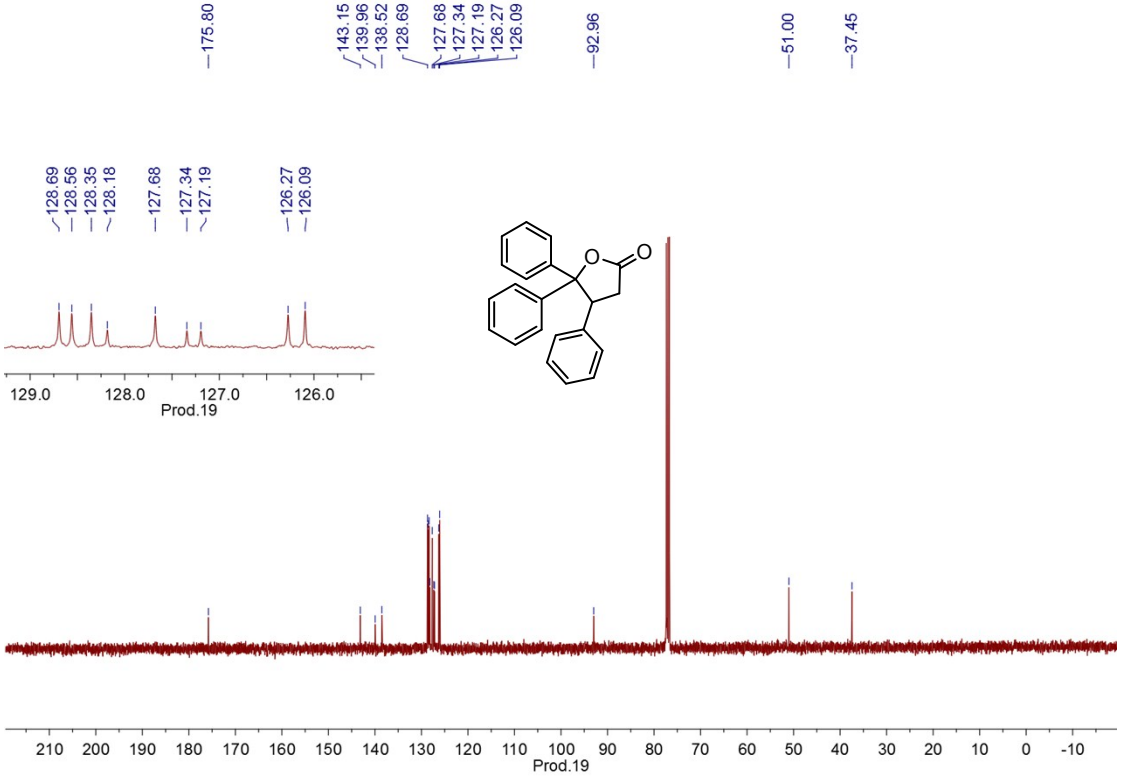

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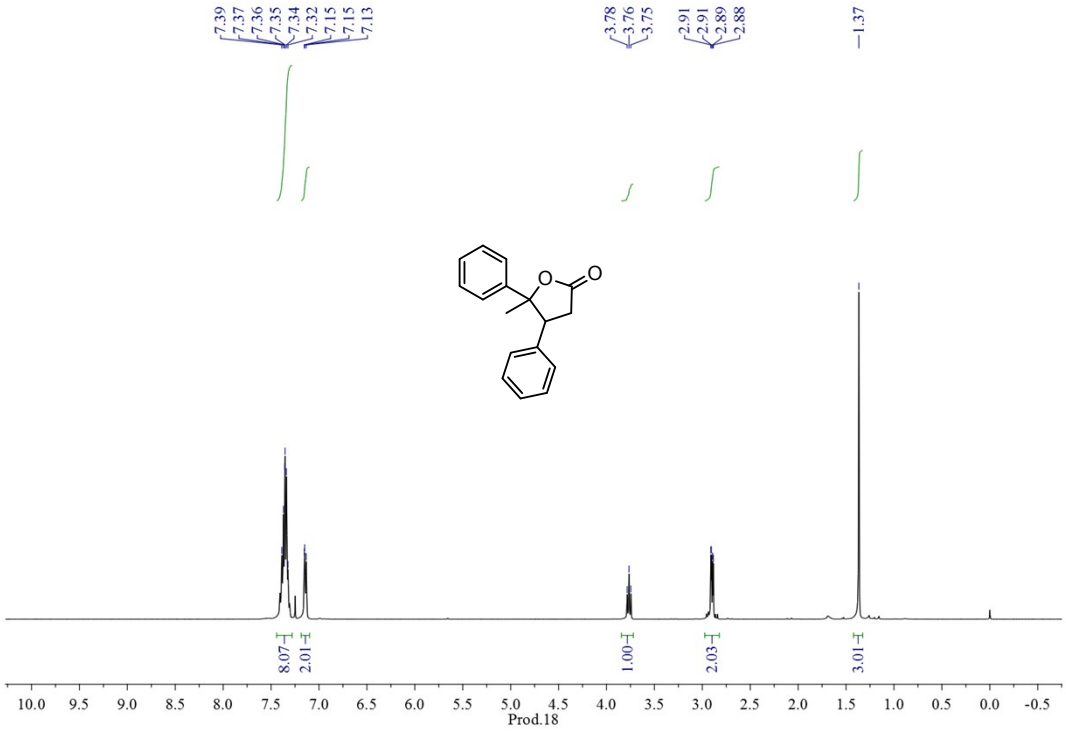
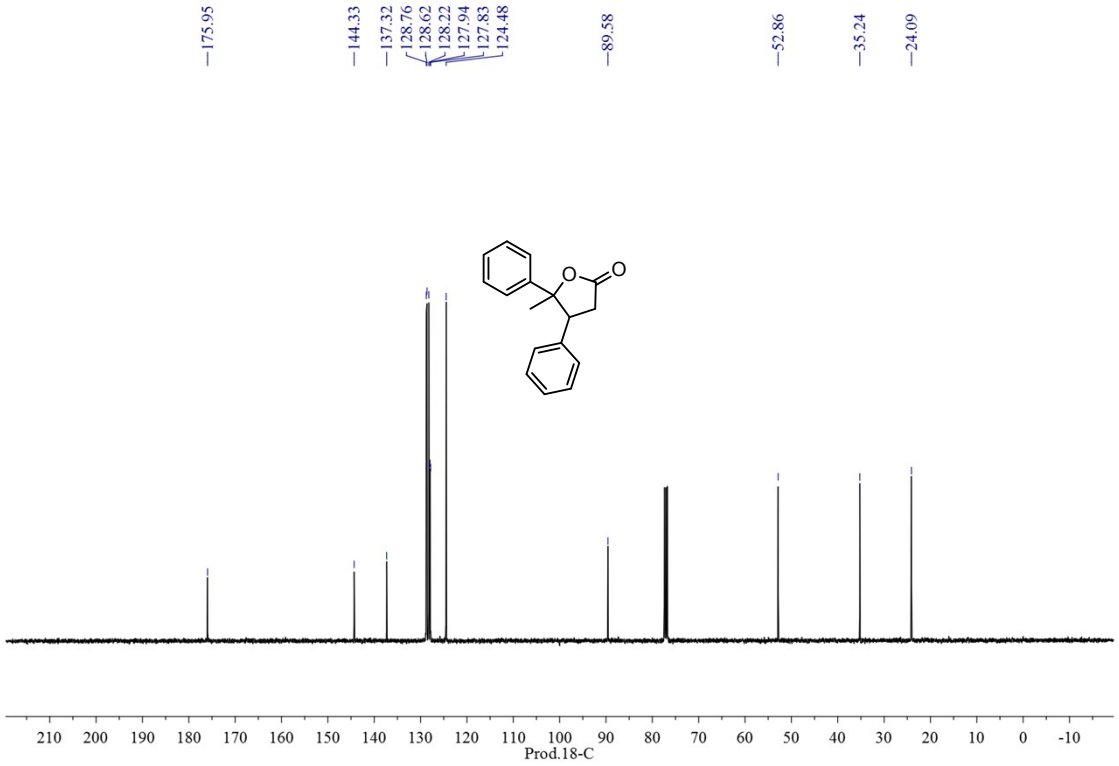
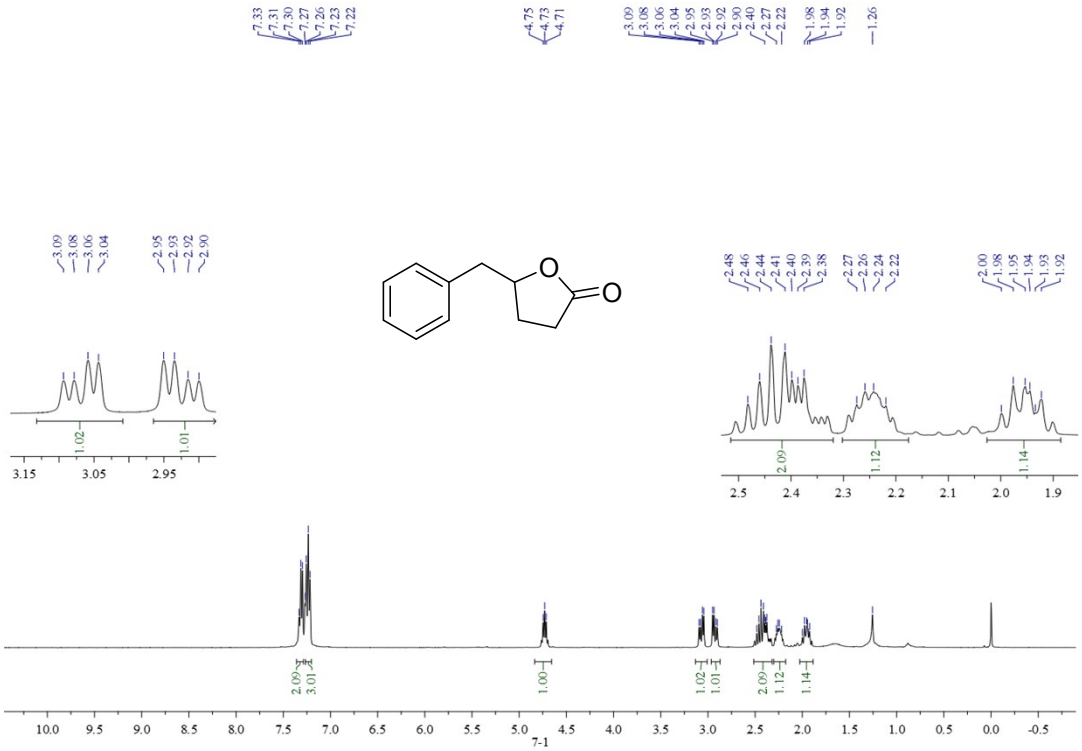


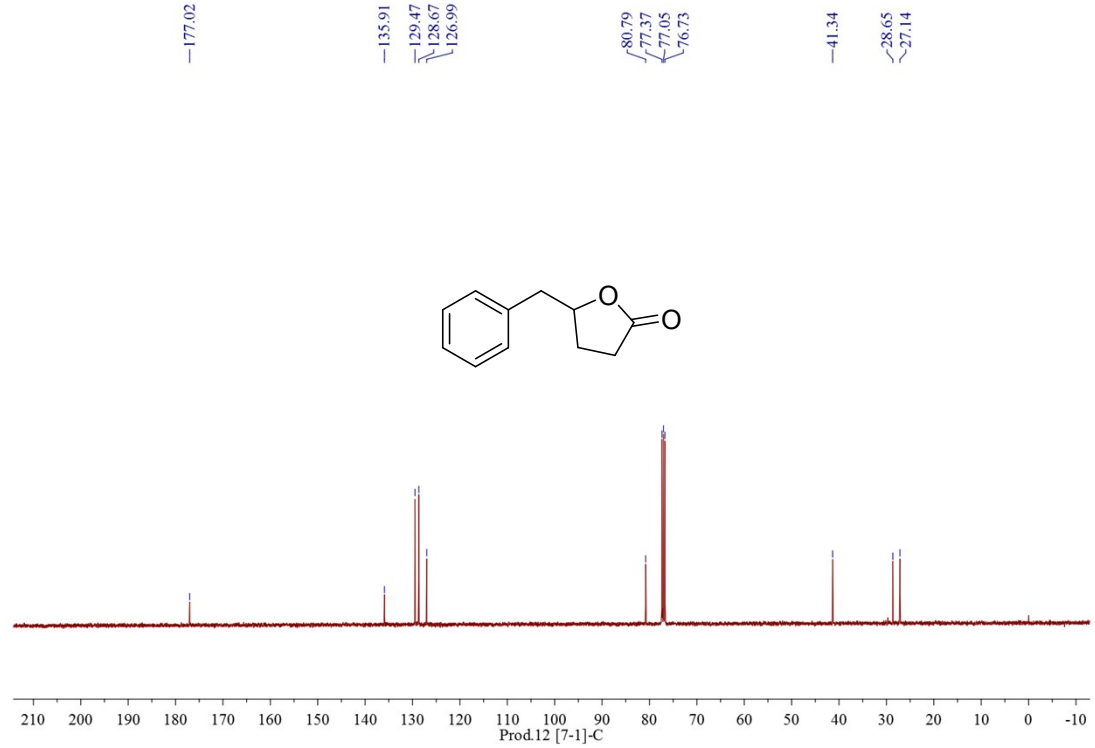
^{1}H NMR of **2u**



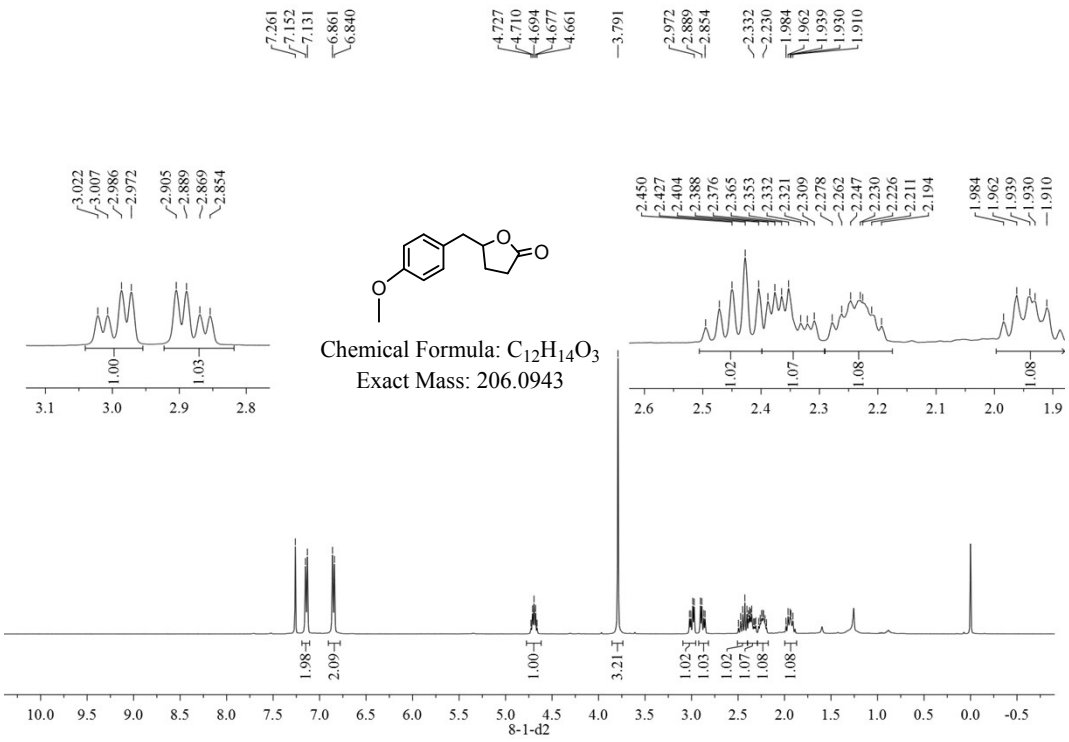
^{13}C NMR of **2u**



^{1}H NMR of **2v**



^{13}C NMR of **2v**



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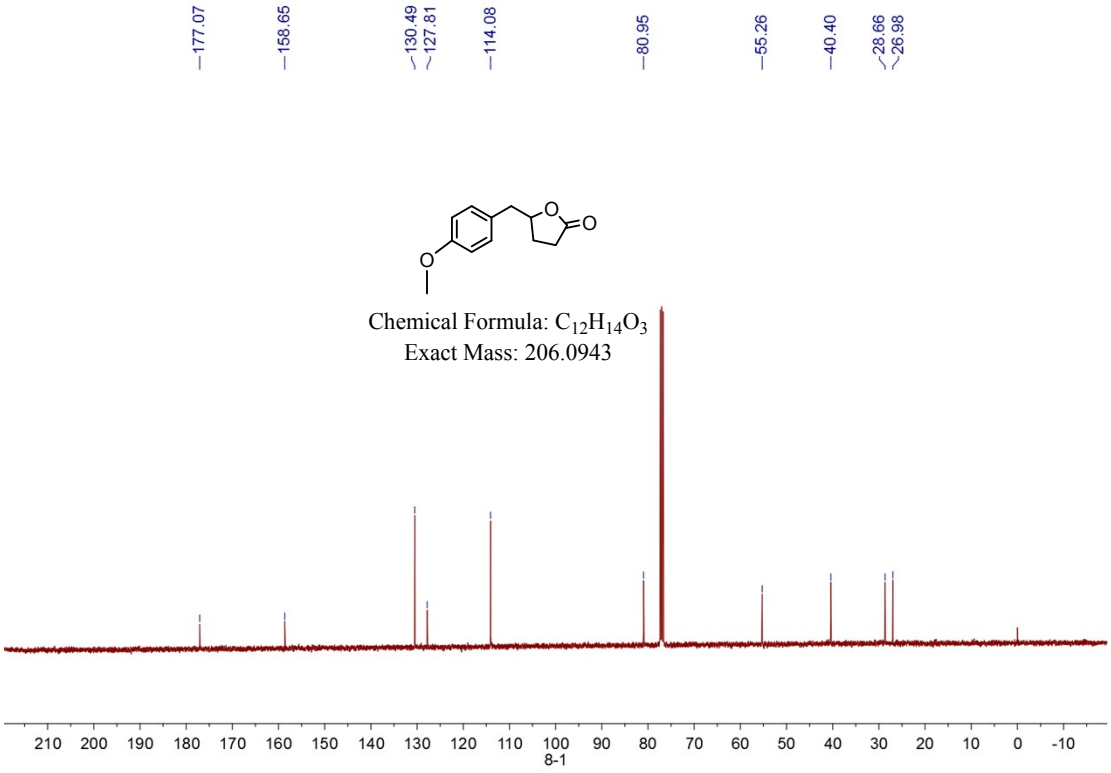
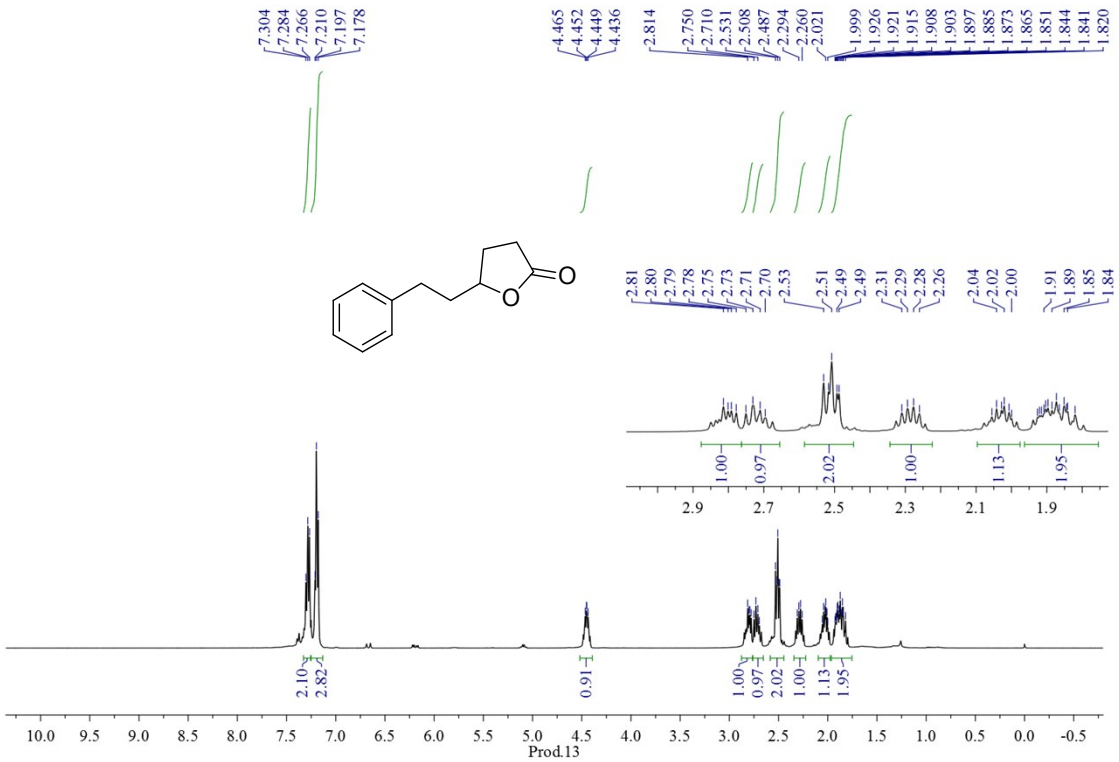
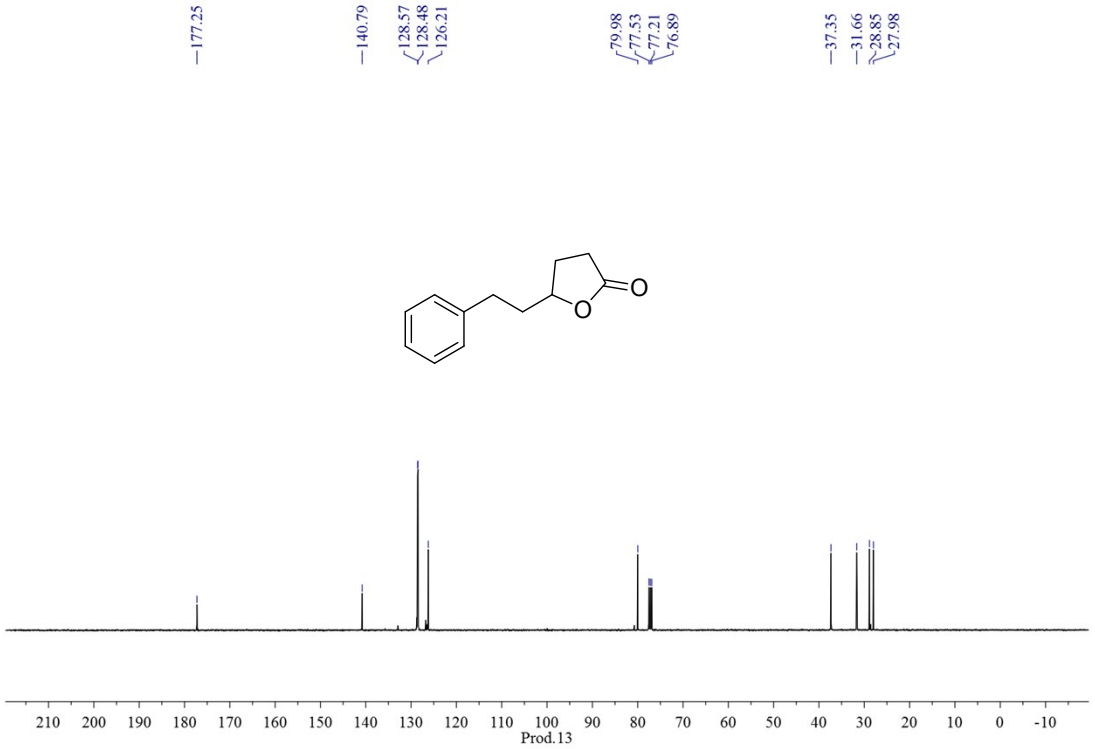


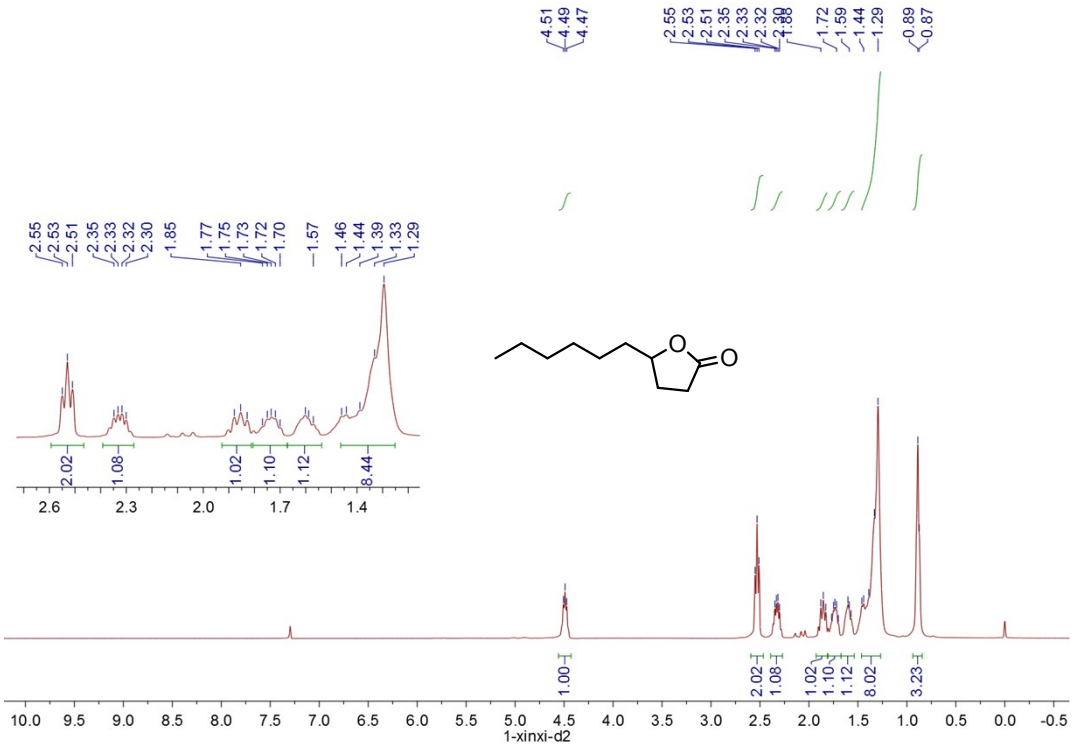
^{13}C NMR of **2w**



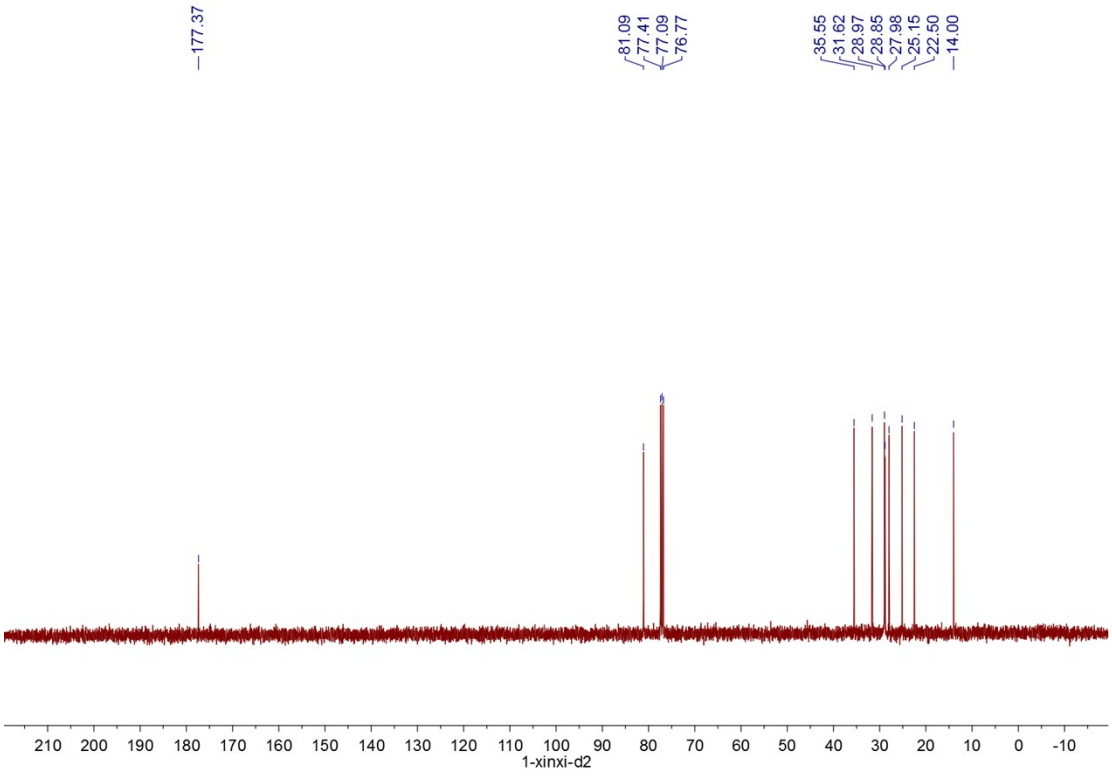
^{1}H NMR of **2x**



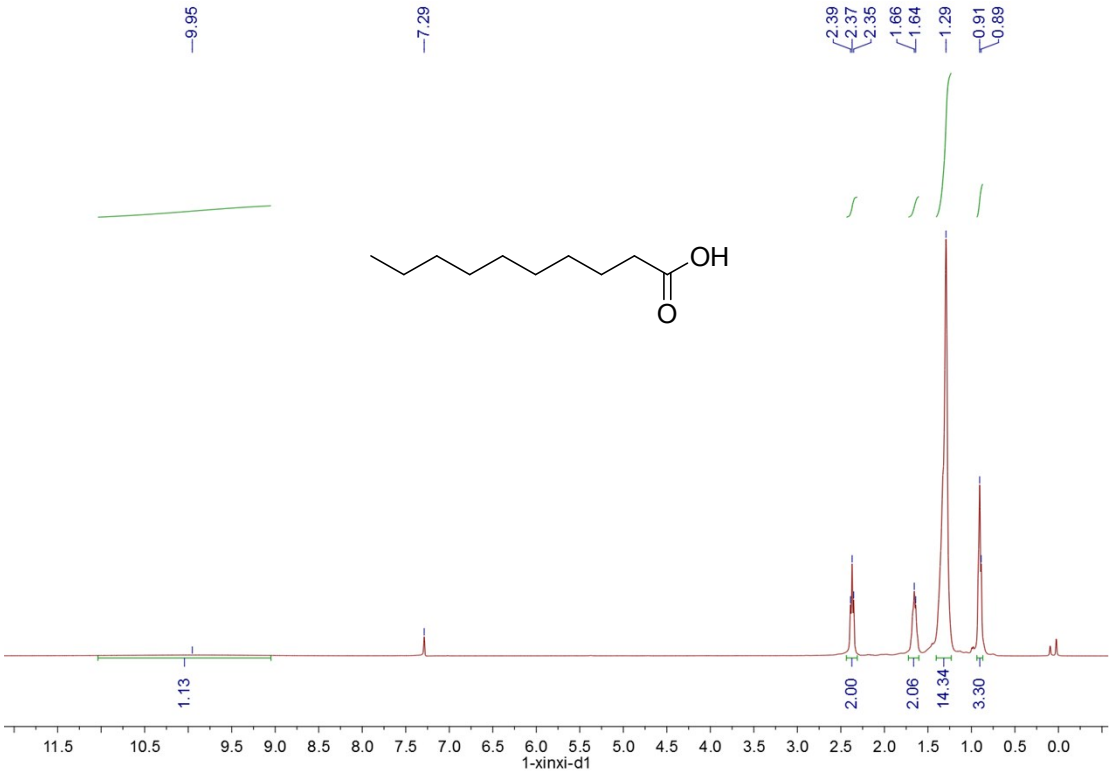
^{13}C NMR of **2x**



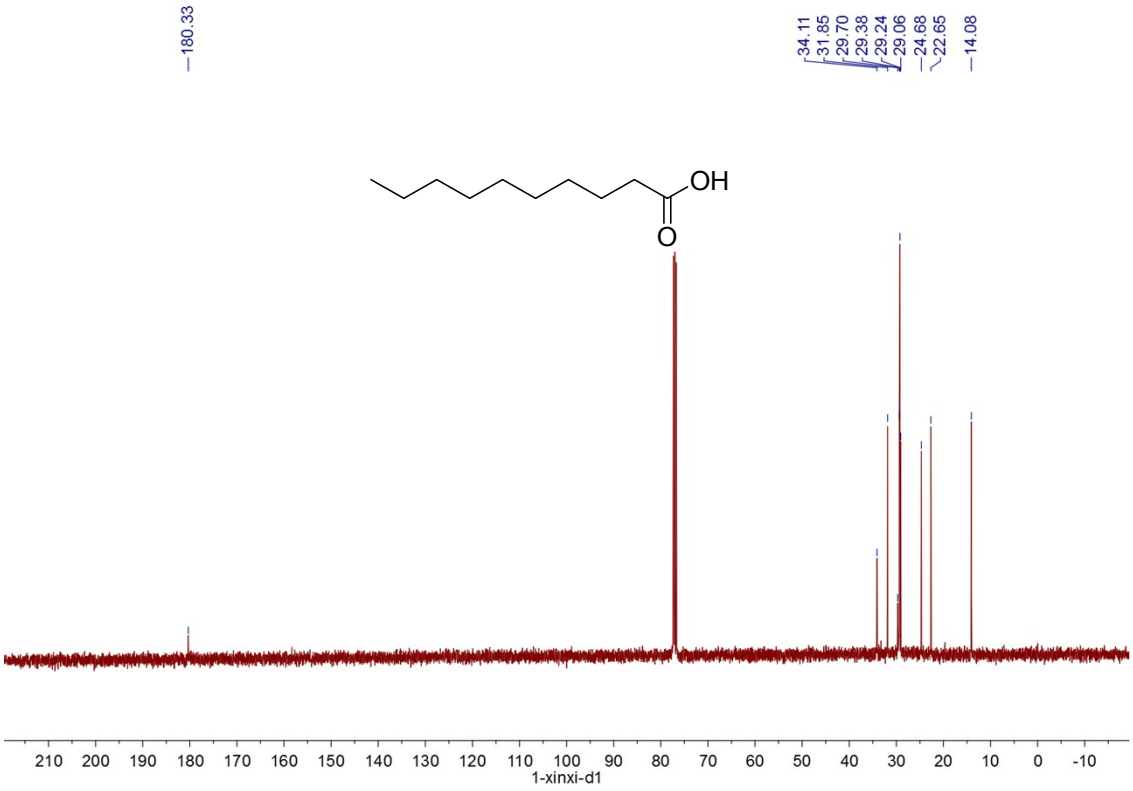
^{1}H NMR of **2y**



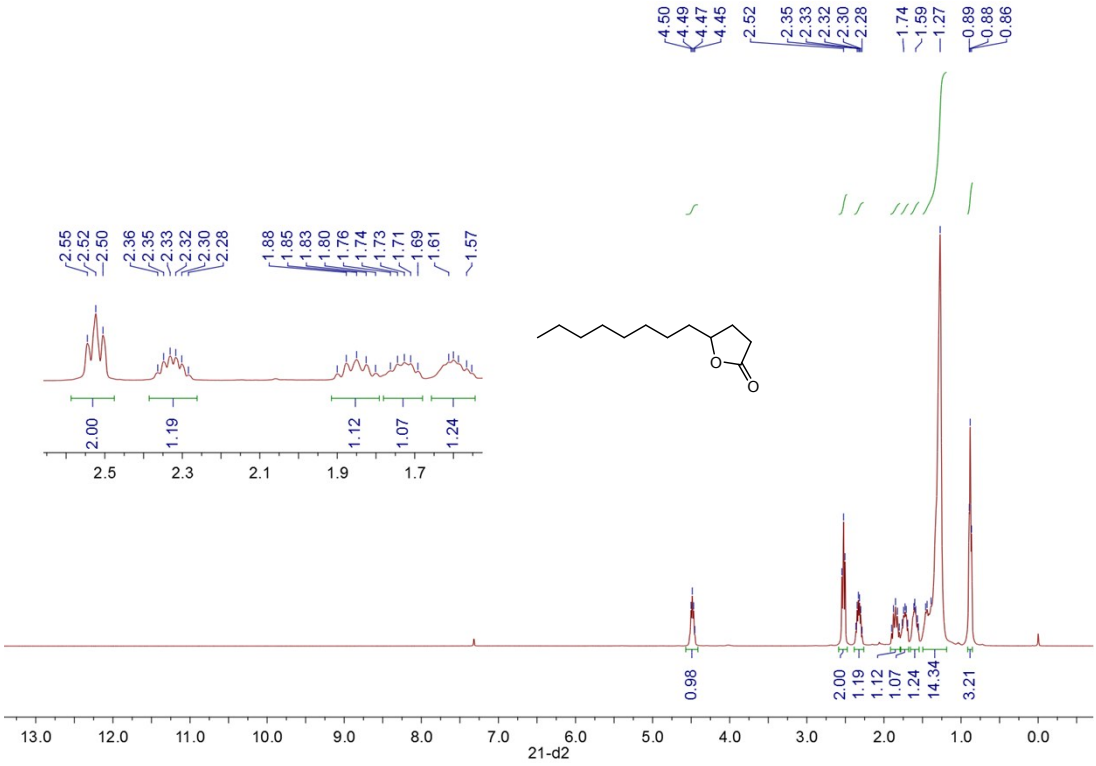
^{13}C NMR of **2y**



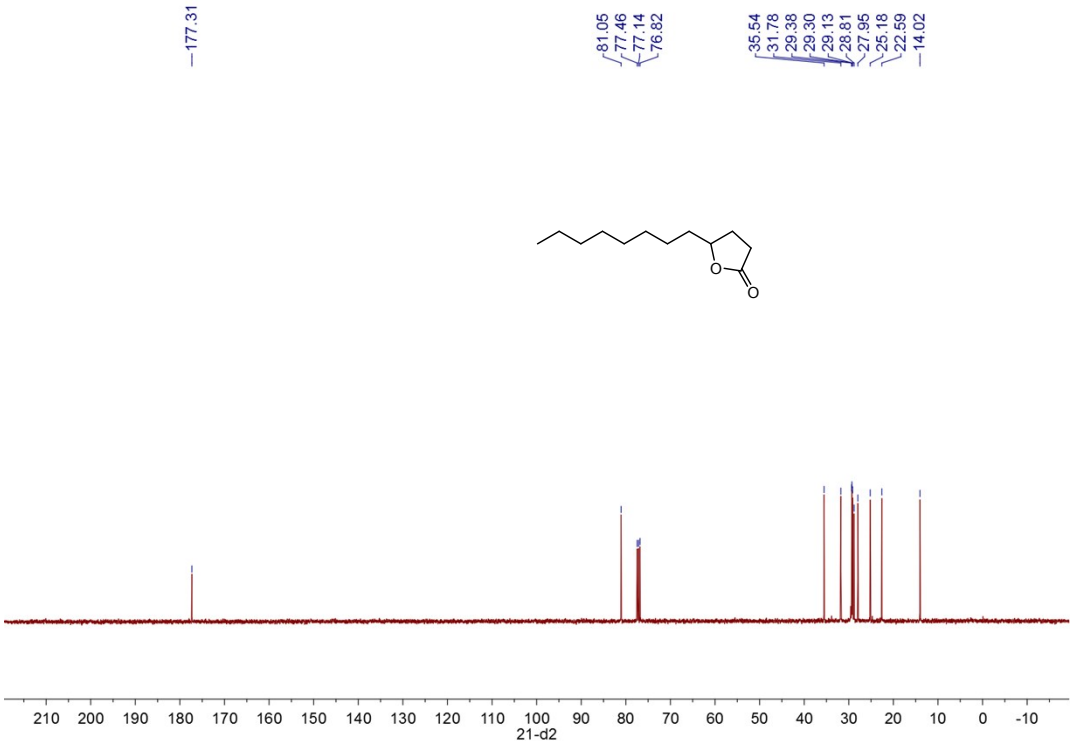
^{1}H NMR of **Decanoic acid (2y-1)**



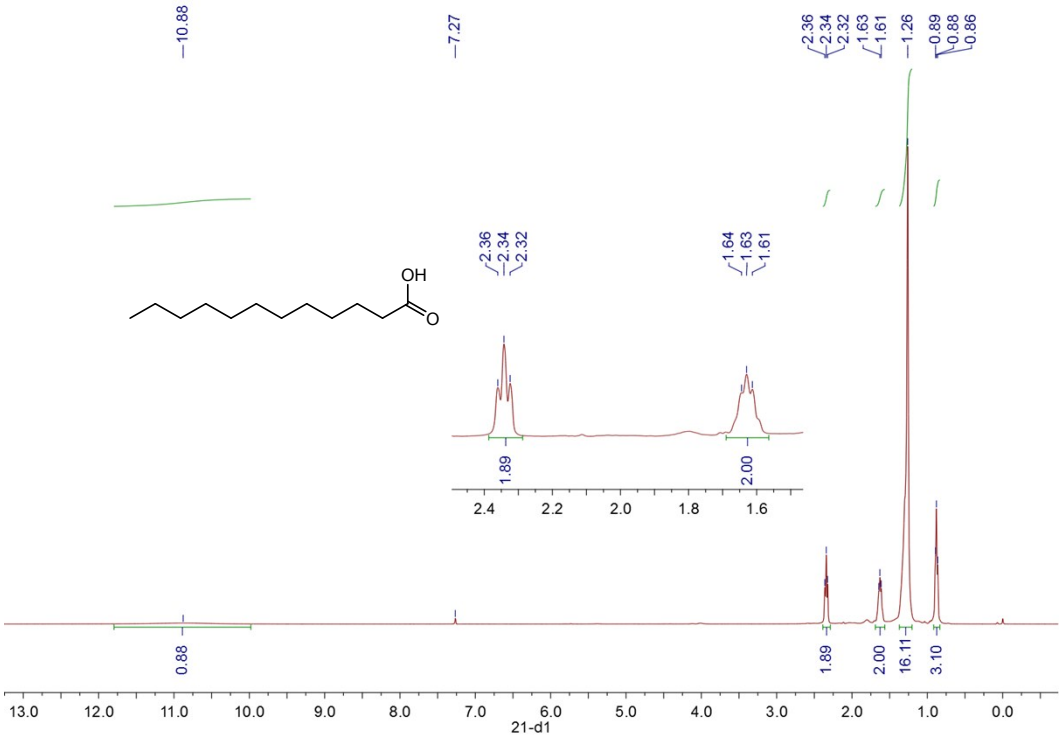
^{13}C NMR of **Decanoic acid (2y-1)**



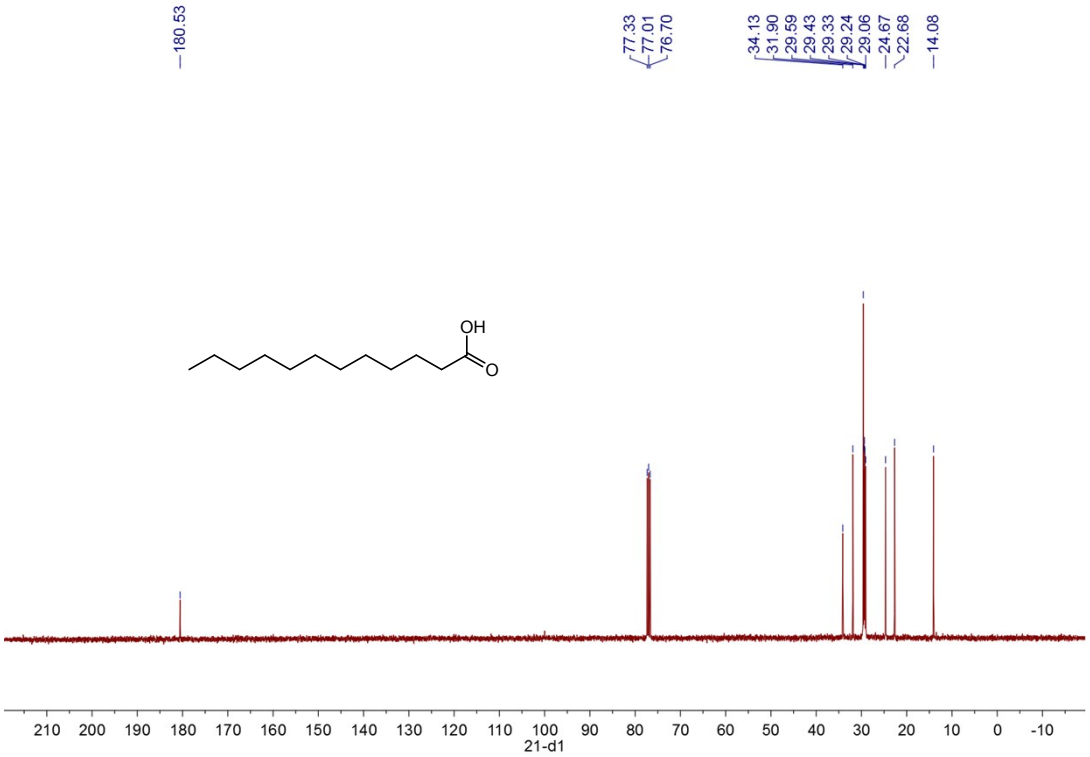
^{1}H NMR of **2z**



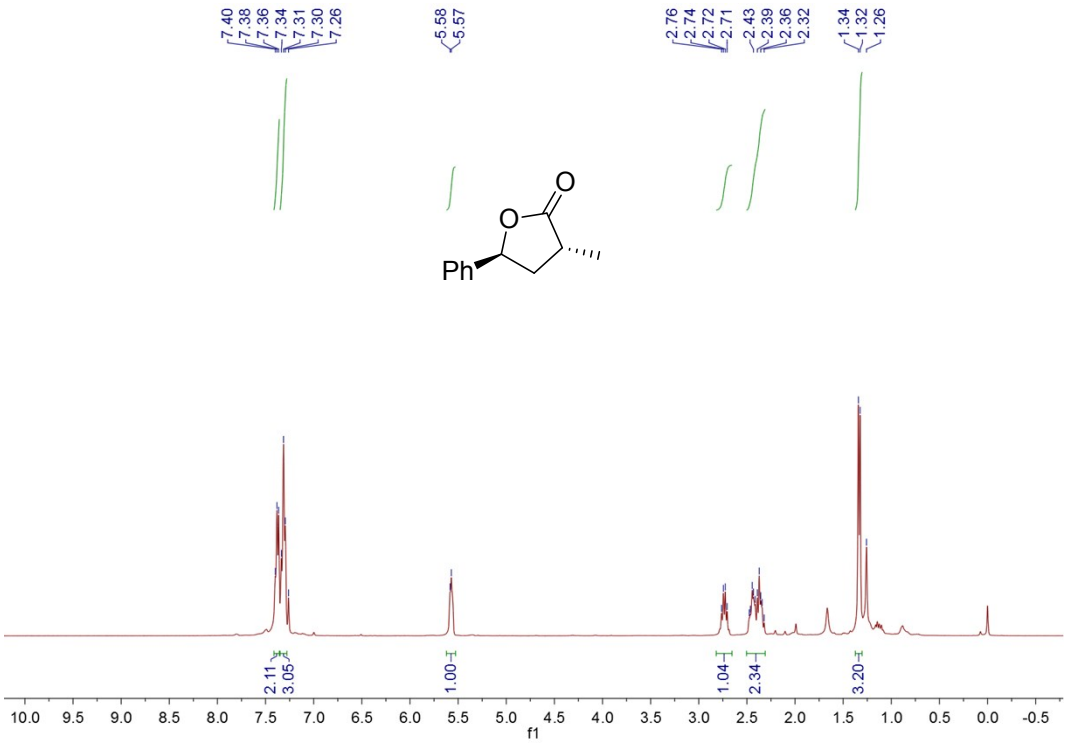
^{13}C NMR of **2z**



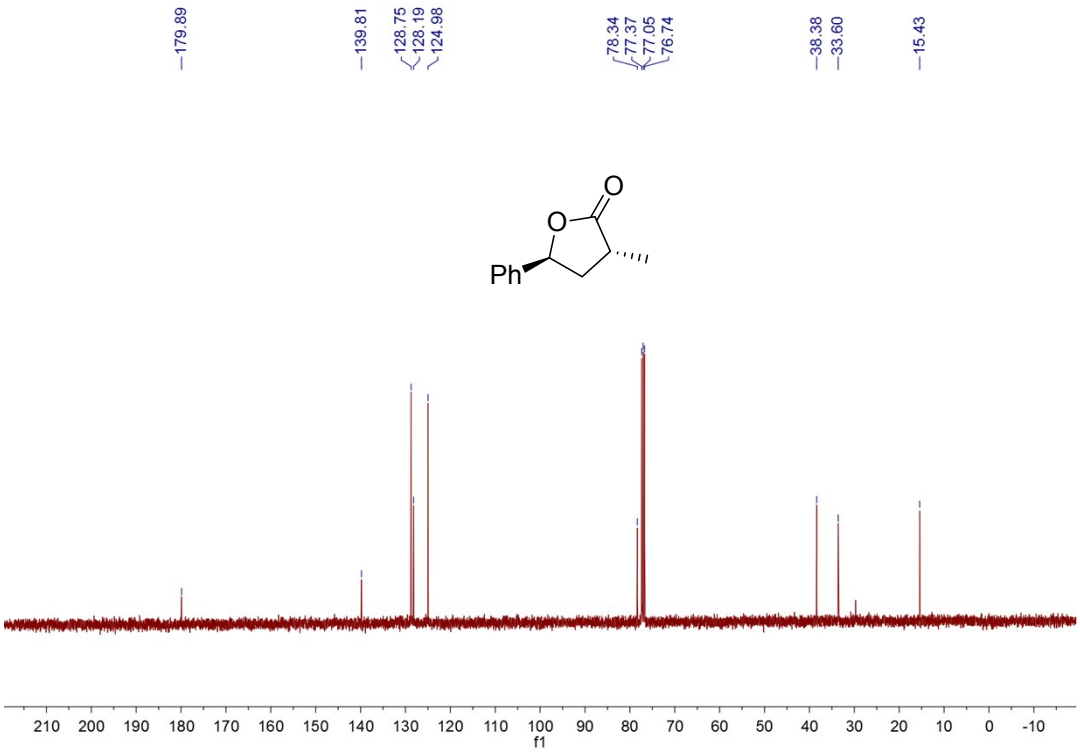
^{1}H NMR of **Dodecanoic acid (2z-1)**



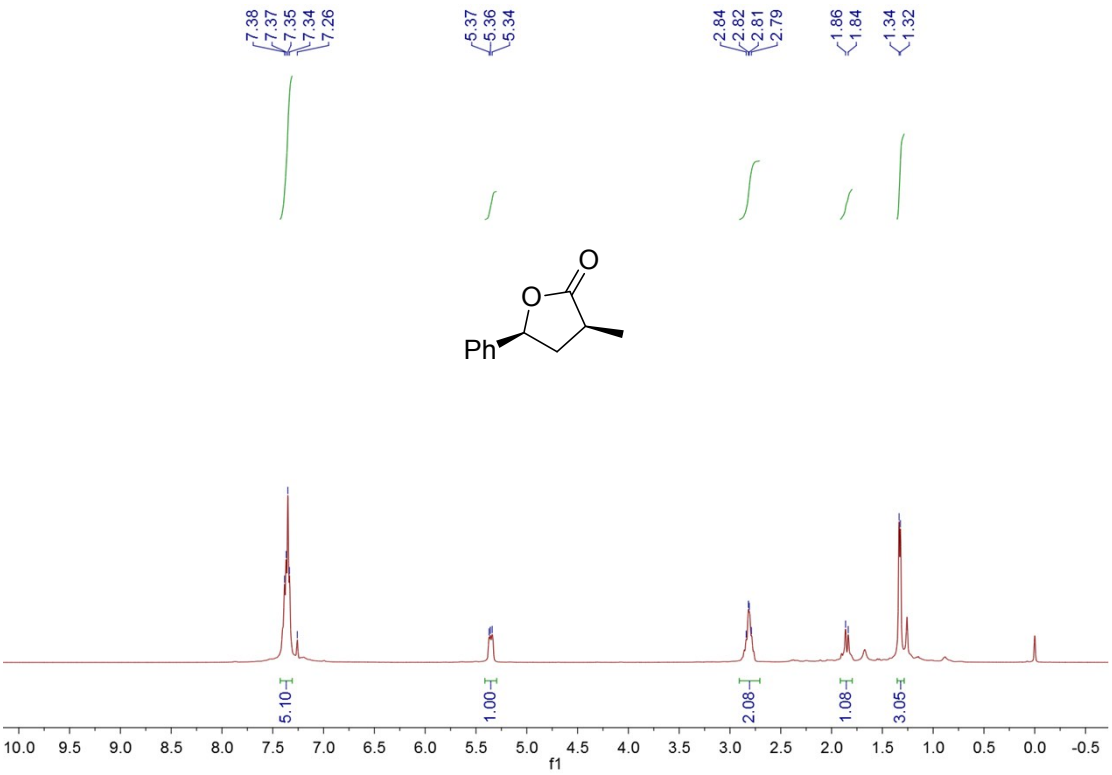
^{13}C NMR of **Dodecanoic acid (2z-1)**



^{1}H NMR of **3a-1**



^{13}C NMR of **3a-1**



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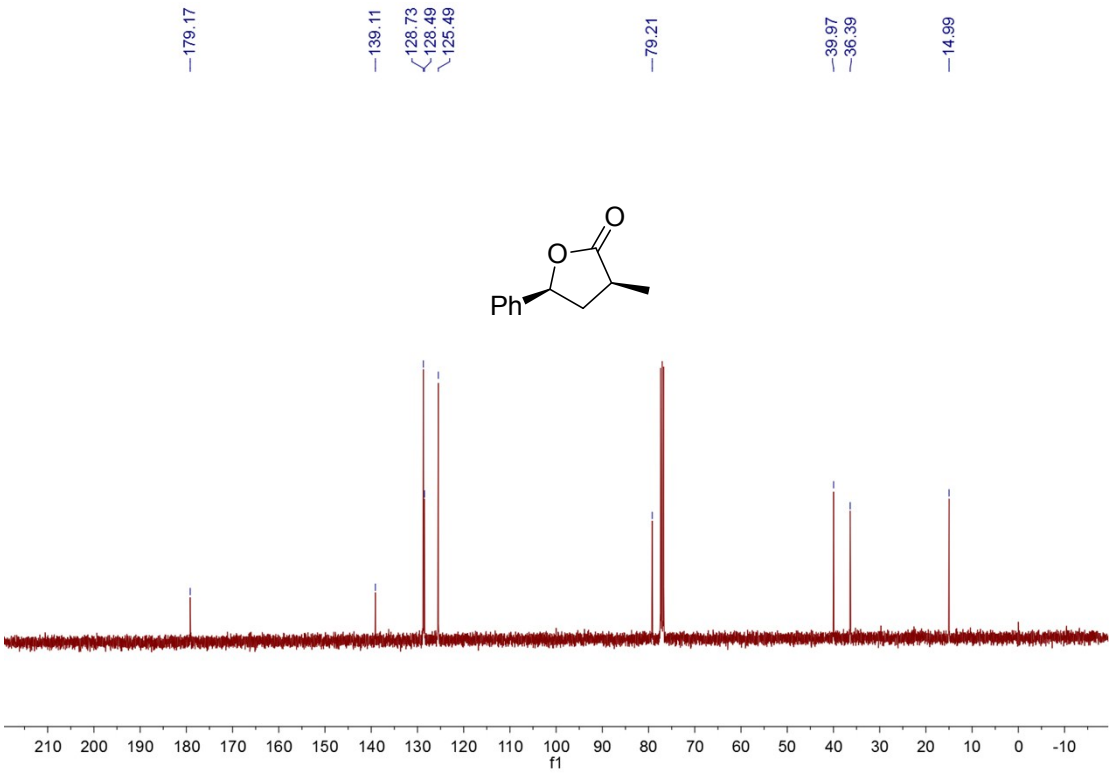
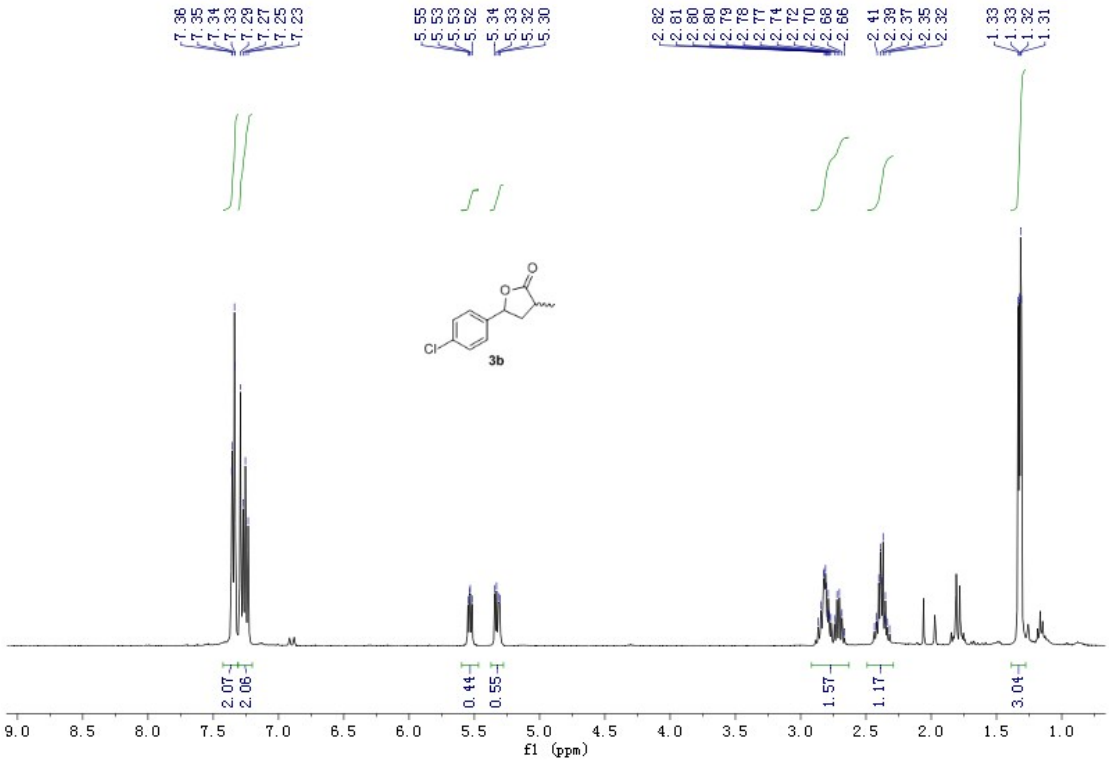
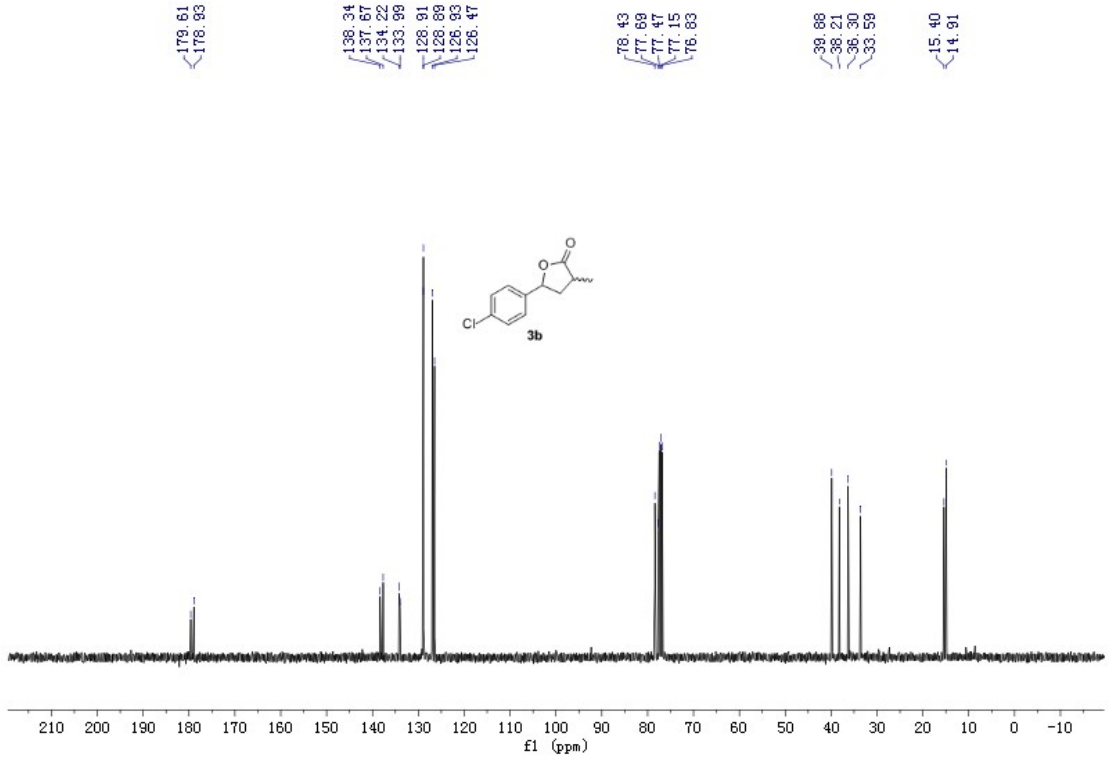


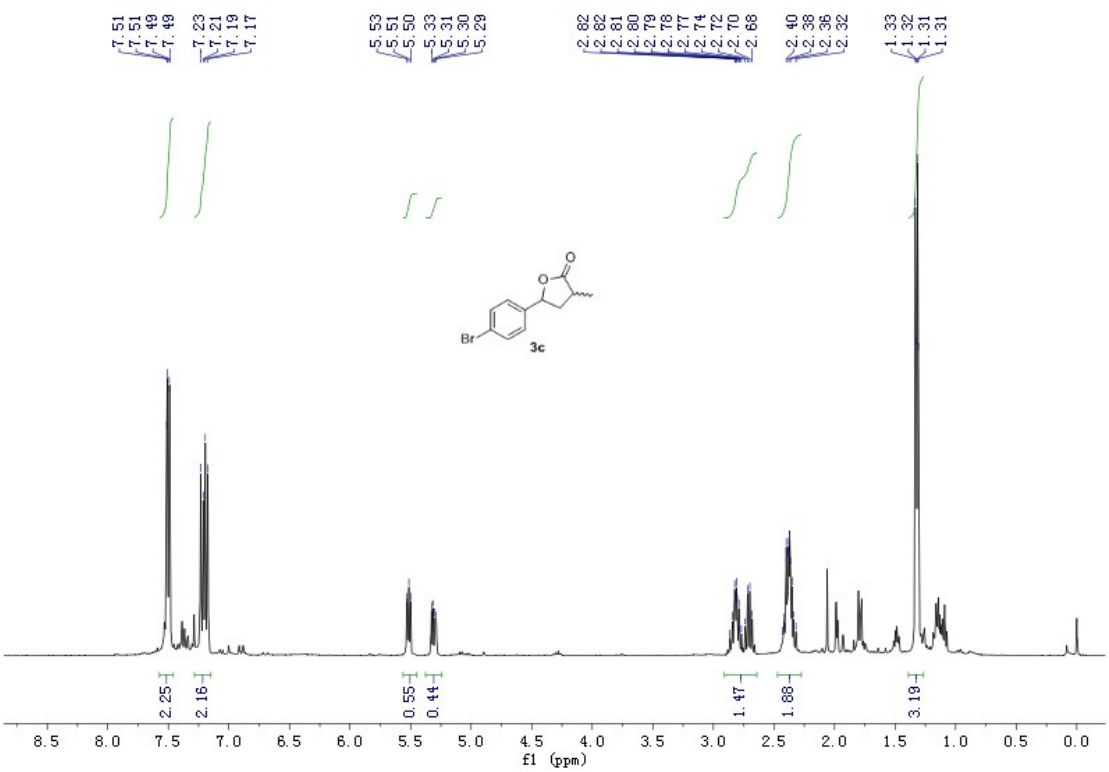
^{13}C NMR of **3a-2**



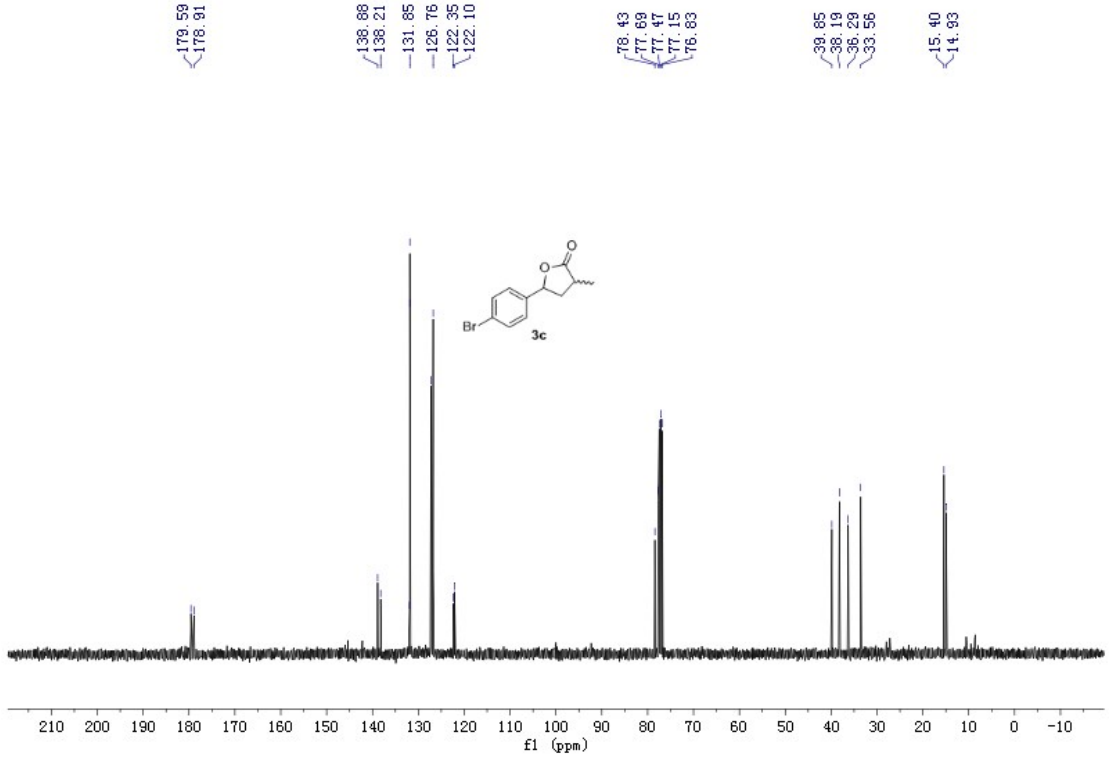
^{1}H NMR of **3b**



^{13}C NMR of **3b**



^{1}H NMR of **3c**



^{13}C NMR of **3c**