Ligand-Enabled Double γ-C(sp^3)–H Functionalization of Aliphatic Acids: One-Step Synthesis of γ-Arylated γ-Lactones

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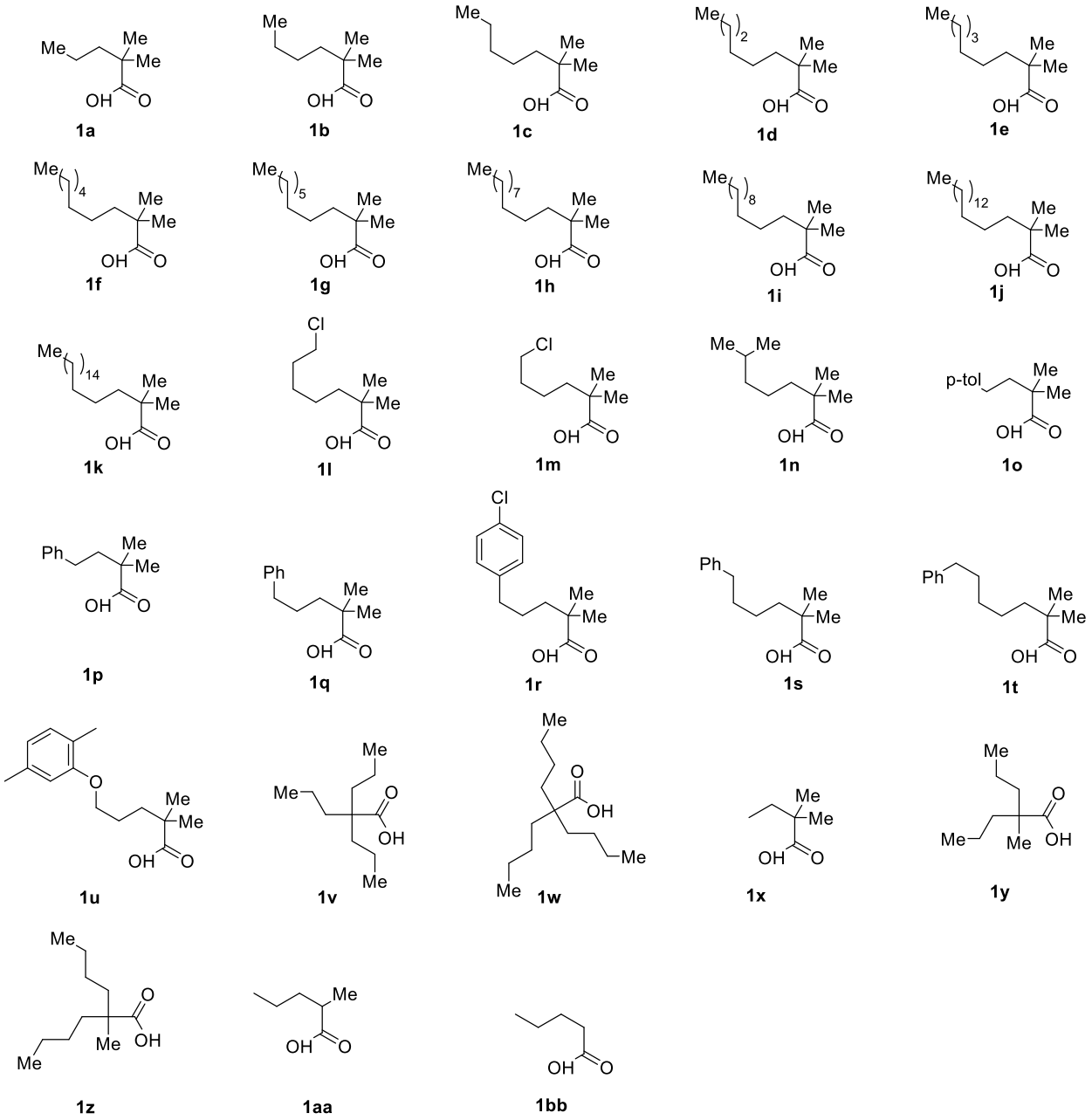
Table of Contents

1. General Information

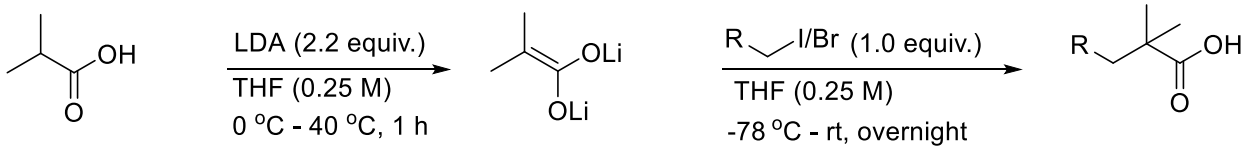
$Pd(OAc)_2$ was purchased from Sigma-Aldrich. Ag_3PO_4 was purchased from Strem. Solvents were obtained from Sigma-Aldrich, Acros and Oakwood, and used directly without further purification. Aryl iodides were obtained from commercial sources. Carboxylic acids were obtained from commercial sources or synthesized following literature procedures. Other reagents were purchased of the highest commercial quality and used without further purification, unless otherwise stated. Analytical thin layer chromatography was performed on 0.25 mm silica gel 60F254. Visualization was carried out with UV light. ^1H NMR spectras were recorded on Bruker DRX-600 instrument. Chemical shifts were quoted in parts per million (ppm) referenced to 0.00 ppm for TMS. The following abbreviations (or combinations thereof) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Coupling constants, J, were reported in Hertz unit (Hz). ^{13}C NMR spectra were recorded on Bruker DRX-600 was fully decoupled by broad band proton decoupling. Chemical shifts were reported in ppm referenced to the centre line of a triplet at 77.16 ppm of $CDCl_3$. Column chromatography was performed using E. Merck silica (60, particle size 0.043–0.063 mm), and preparative thin layer chromatography (pTLC) was performed on Merck silica plates (60F-254). High-resolution mass spectra (HRMS) were recorded on an Agilent Mass spectrometer using ESI-TOF (electrospray ionization-time of flight).

2. Scope of Aliphatic Linear α-Quaternary Carboxylic Acid:



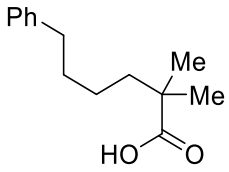
1a, 1b, 1c, 1d, 1e, 1g, 1g, 1q, 1s, 1x, 1aa, and 1bb were obtained from the commercial sources. Others were synthesized using reported procedure. All the substrates except 1s and 1t have been previously reported.

General Procedure for the Preparation of Corresponding *α*-Quaternary Carboxylic Acids Substrates:



To a stirring solution of diisopropylamine (22 mmol, 3.1 mL) in THF (40 mL) was added 2.5 M n-BuLi solution (22 mmol, 8.8 mL) dropwise at -78 °C. The resulting solution was stirred for 30 min at 0 °C. Isobutyric acid (10 mmol, 910 uL) was added dropwise at the same temperature, and the reaction mixture was stirred for 1 h at 40 °C. After cooling to -78 °C, corresponding alkyl bromide (10 mmol) was added dropwise. The reaction mixture was slowly warmed to room temperature overnight. The reaction mixture was quenched with water and diluted with EtOAc. The layers were separated, and the organic layer was extracted with water. The aqueous layer was combined and acidified to pH < 4 with 3 M HCl (aq.). Then, the aqueous layer was extracted with EtOAc. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuo. The crude carboxylic acid was purified by flash chromatography to get the pure carboxylic acid.

Full Characterization of New Compounds:



1s

2,2-dimethyl-6-phenylhexanoic acid (1s)

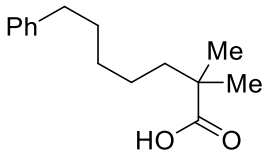
Substrate **1s** was synthesized following the general procedure (eluent: hexane/ethyl acetate = 4/1). The product was obtained as a white solid (1.65g, 75% yield). R_f = 0.4; ^{1}H NMR (600 MHz, $CDCl_3$) δ 11.24 (b s, 1H), 7.29 – 7.26 (m, 2H), 7.18-7.15 (m, 3H), 2.64 – 2.59 (m, 2H), 1.65 – 1.57 (m, 4H), 1.38 – 1.32 (m, 2H), 1.20 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 184.29, 142.73, 128.52, 128.41, 125.79, 42.25, 42.22, 40.42, 35.90, 32.08, 25.11, 24.72.

HRMS (ESI-TOF) Calcd for $C_{14}H_{19}O_2$ $[M-H]^-$: 219.1385; found: 219.1376.

Melting point: 61 °C to 65 °C

IR (cm^{-1}) data: 2935, 1692, 1452, 1254, 1195, 942, 737, 718, 695.



1t

2,2-dimethyl-7-phenylheptanoic acid (1t)

Substrate **1t** was synthesized following the general procedure (eluent: hexane/ethyl acetate = 4/1). The product was obtained as a white solid (1.68g, 72% yield). R_f = 0.4 ^{1}H NMR (600 MHz, $CDCl_3$) δ 11.17 (b s, 1H), 7.29 – 7.25 (m, 2H), 7.17 (t, *J* = 7.3 Hz, 3H), 2.61 – 2.56 (m, 2H), 1.62 (dt, *J* = 15.0, 7.2 Hz, 2H), 1.56 – 1.50 (m, 2H), 1.36 – 1.26 (m, 4H), 1.19 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 184.38, 142.87, 128.55, 128.38, 125.75, 42.23, 42.19, 40.58, 36.05, 31.44, 29.82, 25.11, 25.10, 24.84.

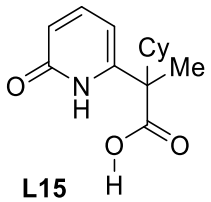
HRMS (ESI-TOF) Calcd for $C_{15}H_{11}O_2$ $[M-H]^-$: 233.1542; found: 233.1532.

Melting point: 58 °C to 62 °C

IR (cm^{-1}) data: 2930, 1696, 1474, 1195, 942, 744, 697.

3. Preparation of Ligands:

Notice: Ligands in the ligand screening table were obtained from the commercial sources or synthesized following literature procedures. [1-6] All ligands except L15 have been previously reported. L15 was synthesized using the same reported procedure as L14.[6]



L15

^{1}H NMR (600 MHz, $CDCl_3$) δ 11.59 (b s, 1H), 7.51 (dd, *J* = 9.0, 7.2 Hz, 1H), 6.55 (dd, *J* = 9.0, 0.7 Hz, 1H), 6.36 – 6.32 (m, 1H), 2.21 – 2.13 (m, 1H), 1.82 – 1.71 (m, 3H), 1.70 – 1.63 (m, 1H), 1.52 (s, 3H), 1.47 – 1.42 (m, 1H), 1.33 – 1.21 (m, 2H), 1.13 – 0.96 (m, 3H).

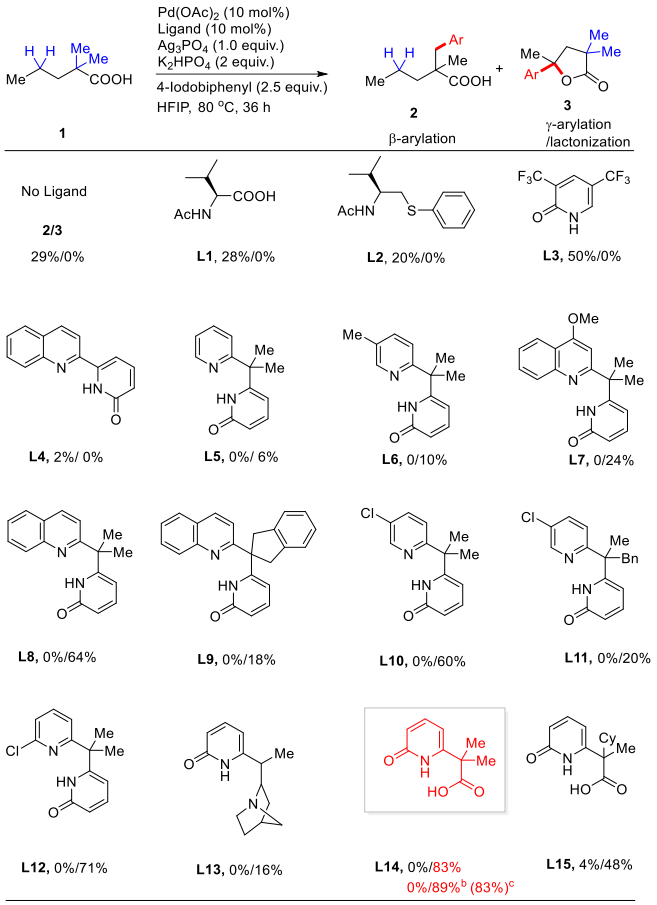
^{13}C NMR (151 MHz, $CDCl_3$) δ 177.93, 164.84, 149.40, 142.12, 117.97, 107.12, 53.79, 46.03, 28.91, 27.89, 26.91, 26.81, 26.41.

HRMS (ESI-TOF) Calcd for $C_{14}H_{20}NO_3$ $[M+H]^+$: 250.1438; found: 250.1440.

IR (cm^{-1}) data: 2927, 2362, 1650, 1589, 1275, 1262, 749, 671.

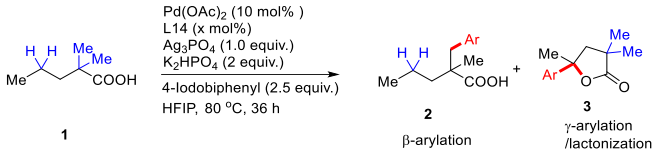
4. Optimization of Reaction:

Table S1. Ligand Optimization[a]



[a]The reaction was performed with carboxylic acid **1a** (0.1 mmol), $Pd(OAc)_2$ (10 mol%), ligand (10 mol%), Ag_3PO_4 (1.0 equiv.), and K_2HPO_4 (2.0 equiv.) in HFIP (1.0 mL) at 80 °C under air for 36 h. 20 mol% ligand used for **L1** and **L3**. [1]H NMR yields, determined using CH_2Br_2 as an internal standard. [b]15 mol% ligand used. [c] Isolated yield.

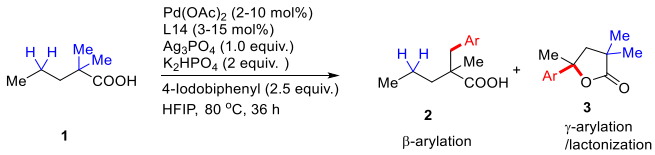
Table S2. Ligand Loading Optimization



Catalyst loading	Yield (2/3)
8 mol%	0%/79%
10 mol%	0%/83%
15 mol%	0%/89%
20 mol%	0%/85%

The reaction was performed with carboxylic acid **1a** (0.1 mmol), $Pd(OAc)_2$ (10 mol%), L14 (8 to 20 mol%), Ag_3PO_4 (1.0 equiv.), and K_2HPO_4 (2.0 equiv.) in HFIP (1.0 mL) at 80 °C under air for 36 h. ^1H NMR yields, determined using CH_2Br_2 as an internal standard.

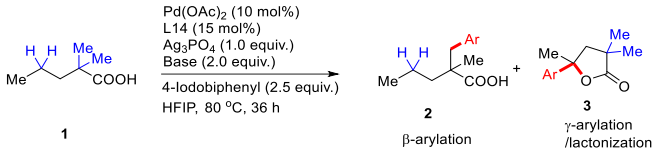
Table S3. Catalyst Loading Optimization



Catalyst loading	Yield (2/3)
2 mol%	0%/11%
4 mol%	0%/29%
6 mol%	0%/51%
8 mol%	0%/74%
10 mol%	0%/89%

The reaction was performed with carboxylic acid **1a** (0.1 mmol), $Pd(OAc)_2$ (2 to 10 mol%), L14 (3 to 15 mol%), Ag_3PO_4 (1.0 equiv.), and K_2HPO_4 (2.0 equiv.) in HFIP (1.0 mL) at 80 °C under air for 36 h. ^1H NMR yields, determined using CH_2Br_2 as an internal standard.

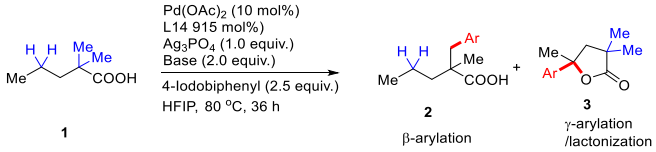
Table S4. Reaction Time Optimization



Time	Yield (2/3)
10 min.	0%/5%
30 min.	0%/8%
1 h	0%/16%
2 h	0%/25%
3 h	0%/35%
6 h	0%/44%
8 h	0%/48%
16 h	0%/76%
36 h	0%/89%

The reaction was performed with carboxylic acid **1a** (0.1 mmol), $Pd(OAc)_2$ (10 mol%), L14 (15 mol%), Ag_3PO_4 (1.0 equiv.), and K_2HPO_4 (2.0 equiv.) in HFIP (1.0 mL) at 60 – 90 °C under air for 24 h. ^1H NMR yields, determined using CH_2Br_2 as an internal standard.

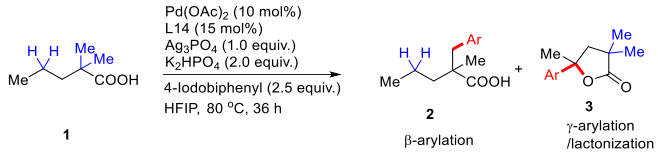
Table S5. Temperature Optimization



Temperature	Yield (2/3)
60 °C	0%/68%
70 °C	0%/80%
80 °C	0%/89%
90 °C	0%/89%

The reaction was performed with carboxylic acid **1a** (0.1 mmol), $Pd(OAc)_2$ (10 mol%), L14 (15 mol%), Ag_3PO_4 (1.0 equiv.), and K_2HPO_4 (2.0 equiv.) in HFIP (1.0 mL) at 60 – 90 °C under air for 36 h. ^1H NMR yields, determined using CH_2Br_2 as an internal standard.

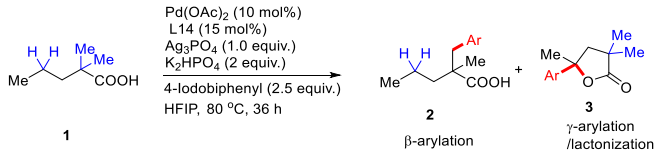
Table S6. Solvent Optimization



Solvent	Yield (2/3)
HFIP	0%/89%
TFE	0%/18%
THF	7%/0%
1,4-Dioxane	20%/0%
DCE	2%/0%
CH_3CN	No reaction
DMF	No reaction

The reaction was performed with carboxylic acid **1a** (0.1 mmol), $Pd(OAc)_2$ (10 mol%), L14 (15 mol%), Ag_3PO_4 (1.0 equiv.), and K_2HPO_4 (2.0 equiv.) in HFIP (1.0 mL) at 80 °C under air for 36 h. ^1H NMR yields, determined using CH_2Br_2 as an internal standard.

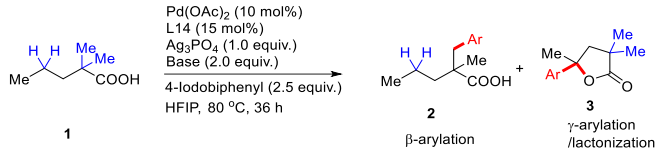
Table S7. of Ag-Salt Optimization



Ag-salt	Yield (2/3)
AgOAc	0%/34%
Ag_2CO_3	0%/43%
$AgNO_3$	0%/10%
$AgOCOCF_3$	0%/3%
Ag_2O	0%/30%
Ag_3PO_4 (2.0 equiv.)	0%/44%
Ag_3PO_4 (1.5 equiv.)	0%/62%
Ag_3PO_4 (1.0 equiv.)	0%/89%

The reaction was performed with carboxylic acid **1a** (0.1 mmol), $Pd(OAc)_2$ (10 mol%), L14 (15 mol%), Ag_3PO_4 (1.0 equiv.), and K_2HPO_4 (2.0 equiv.) in HFIP (1.0 mL) at 80 °C under air for 36 h. ^1H NMR yields, determined using CH_2Br_2 as an internal standard.

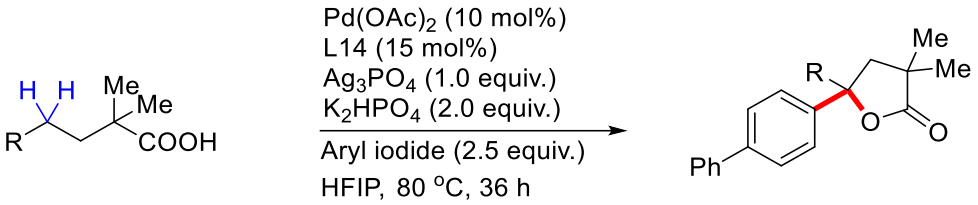
Table S8. Base Optimization



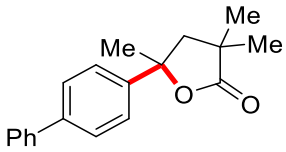
Base	Yield (2/3)
K_2CO_3	0%/70%
$KHCO_3$	0%/68%
K_3PO_4	0%/52%
Na_2CO_3	0%/33%
NaOAc	0%/44%
Cs_2CO_3	0%/66%
K_2HPO_4	0%/89%
1.5 equiv. K_2HPO_4	0%/81%

The reaction was performed with carboxylic acid **1a** (0.1 mmol), $Pd(OAc)_2$ (10 mol%), L14 (15 mol%), Ag_3PO_4 (1.0 equiv.), and base (2.0 equiv.) in HFIP (1.0 mL) at 80 °C under air for 36 h. ^1H NMR yields, determined using CH_2Br_2 as an internal standard.

5. Substrate Scope of the γ-arylation-γ-lactonization Free *α*-quaternary Aliphatic Acids:



General procedure for *β*-methylene C(sp^3)–H of free *α*-quaternary carboxylic acids: $Pd(OAc)_2$ (10 mol%), L14 (15 mol%), Ag_3PO_4 (1.0 equiv.), K_2HPO_4 (2.0 equiv.), Ar-I (2.5 equiv.), and HFIP (1.0 ml) were added to a reaction vial (8 ml) and stir at rt for 5 minutes. Then Carboxylic acid (0.1 mmol) was added. Next the vial was capped under air and closed tightly. Then the reaction mixture was stirred at 80 °C for 36 hours. After cooling to room temperature, the mixture was acidified with HCOOH (50 µL) and filtered through a pad of celite with ethyl acetate as the eluent to remove the insoluble precipitate. The resulting solution was concentrated and purified by preparative thin-layer chromatography to afford the desired product.



5-([1,1'-biphenyl]-4-yl)-3,3,5-trimethyldihydrofuran-2(3H)-one (3a)

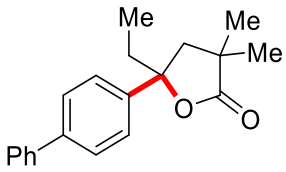
Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as an off white solid (23.2 mg, 83% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.57 (m, 4H), 7.47 – 7.42 (m, 4H), 7.37 – 7.33 (m, 1H), 2.60 (d, *J* = 13.0 Hz, 1H), 2.38 (d, *J* = 13.0 Hz, 1H), 1.75 (s, 3H), 1.36 (s, 3H), 1.03 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.91, 145.04, 140.52, 140.39, 128.95, 127.59, 127.43, 127.17, 124.62, 83.51, 50.82, 41.04, 32.19, 26.88, 26.18.

HRMS (ESI-TOF) Calcd for $C_{19}H_{21}O_2$ $[M+H]^+$: 281.1542; found: 281.1544.

Melting point: 114 ºC to 117 ºC

IR (cm^{-1}) data: 2973, 1764, 1487, 1385, 1248, 1068, 767, 697.



5-([1,1'-biphenyl]-4-yl)-5-ethyl-3,3-dimethyldihydrofuran-2(3H)-one (3b)

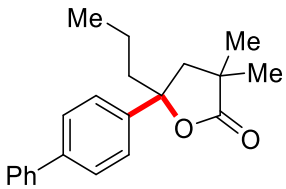
Substrate **1b** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (19.1 mg, 65% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.58 (m, 4H), 7.46 – 7.42 (m, 2H), 7.42 – 7.40 (m, 2H), 7.37 – 7.33 (m, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.38 (d, *J* = 13.1 Hz, 1H), 2.04 – 1.92 (m, 2H), 1.34 (s, 3H), 0.99 (s, 3H), 0.84 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.05, 143.23, 140.51, 140.23, 128.95, 127.57, 127.23, 127.14, 125.35, 86.26, 49.32, 40.64, 37.34, 26.70, 26.33, 8.41.

HRMS (ESI-TOF) Calcd for $C_{20}H_{23}O_2$ $[M+H]^+$: 295.1698; found: 295.1697.

Melting point: 110 ºC to 114 ºC

IR (cm^{-1}) data: 2971, 1765, 1487, 1235, 1085, 766, 698.



5-([1,1'-biphenyl]-4-yl)-3,3-dimethyl-5-propyldihydrofuran-2(3H)-one (3c)

Substrate **1c** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (19.1 mg, 62% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.58 (m,

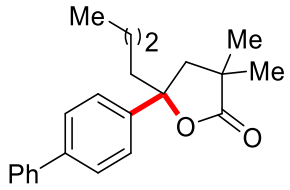
4H), 7.45 – 7.42 (m, 2H), 7.42 – 7.39 (m, 2H), 7.37 – 7.33 (m, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.38 (d, *J* = 13.0 Hz, 1H), 1.92 (dddd, *J* = 34.9, 14.0, 11.6, 4.8 Hz, 2H), 1.45-1.36 (m, 1H), 1.34 (s, 3H), 1.14-1.05 (m, 1H), 0.98 (s, 3H), 0.85 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.07, 143.54, 140.51, 140.20, 128.94, 127.56, 127.23, 127.14, 125.24, 85.98, 49.86, 46.79, 40.53, 26.73, 26.30, 17.37, 14.18.

HRMS (ESI-TOF) Calcd for $C_{21}H_{25}O_2$ $[M+H]^+$: 309.1855; found: 309.1855.

Melting point: 83 °C to 85 °C

IR (cm^{-1}) data: 2962, 1765, 1487, 1212, 1171, 1097, 768.



5-([1,1'-biphenyl]-4-yl)-5-butyl-3,3-dimethyldihydrofuran-2(3H)-one (3d)

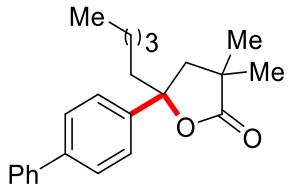
Substrate **1d** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (17.7 mg, 55% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.58 (m, 4H), 7.45 – 7.42 (m, 2H), 7.42 – 7.39 (m, 2H), 7.37 – 7.33 (m, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.38 (d, *J* = 13.0 Hz, 1H), 2.00-1.88 (m, 2H), 1.39-1.35 (m, 1H), 1.34 (s, 3H), 1.30 – 1.19 (m, 2H), 1.09 – 1.01 (m, 1H), 0.98 (s, 3H), 0.83 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.08, 143.60, 140.51, 140.18, 128.95, 127.57, 127.24, 127.14, 125.25, 85.98, 49.82, 44.35, 40.57, 26.74, 26.32, 26.07, 22.82, 14.02.

HRMS (ESI-TOF) Calcd for $C_{22}H_{27}O_2$ $[M+H]^+$: 323.2011; found: 323.2012.

Melting point: 75 °C to 78 °C

IR (cm^{-1}) data: 2959, 1764, 1487, 1240, 1033, 755, 697.



5-([1,1'-biphenyl]-4-yl)-3,3-dimethyl-5-pentyldihydrofuran-2(3H)-one (3e)

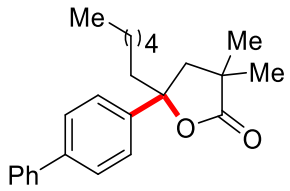
Substrate **1e** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (23.2 mg, 69% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.58 (m, 4H), 7.46 – 7.42 (m, 2H), 7.42 – 7.39 (m, 2H), 7.37 – 7.33 (m, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.37 (d, *J* = 13.1 Hz, 1H), 1.99-1.87 (m, 2H), 1.43 – 1.35 (m, 1H), 1.34 (s, 3H), 1.26 – 1.17 (m, 4H), 1.10 – 1.02 (m, 1H), 0.98 (s, 3H), 0.82 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.08, 143.60, 140.51, 140.16, 128.95, 127.56, 127.23, 127.13, 125.24, 86.00, 49.82, 44.56, 40.56, 31.89, 26.74, 26.31, 23.61, 22.55, 14.10.

HRMS (ESI-TOF) Calcd for $C_{23}H_{29}O_2$ $[M+H]^+$: 337.2168; found: 337.2170.

Melting point: 74 °C to 77 °C

IR (cm^{-1}) data: 2932, 1765, 1487, 1235, 1211, 1106, 767, 739, 697.



5-([1,1'-biphenyl]-4-yl)-5-hexyl-3,3-dimethyldihydrofuran-2(3H)-one (3f)

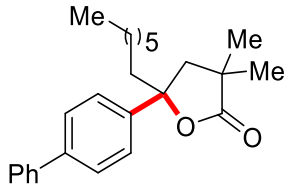
Substrate **1f** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (18.2 mg, 52% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.58 (m, 4H), 7.44 (ddd, *J* = 8.0, 6.3, 1.7 Hz, 2H), 7.41 – 7.39 (m, 2H), 7.37 – 7.33 (m, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.37 (d, *J* = 13.1 Hz, 1H), 1.98 – 1.87 (m, 2H), 1.41 – 1.34 (m, 1H), 1.34 (s, 3H), 1.24 – 1.16 (m, 6H), 1.08 – 1.01 (m, 1H), 0.98 (s, 3H), 0.83 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.08, 143.60, 140.51, 140.16, 128.94, 127.56, 127.23, 127.13, 125.23, 86.00, 49.81, 44.60, 40.56, 31.72, 29.38, 26.73, 26.31, 23.89, 22.67, 14.15.

HRMS (ESI-TOF) Calcd for $C_{24}H_{31}O_2$ $[M+H]^+$: 351.2324; found: 351.2317.

Melting point: 70 °C to 73 °C

IR (cm^{-1}) data: 2932, 1767, 1240, 1211, 1108, 842, 738, 697.



5-([1,1'-biphenyl]-4-yl)-5-heptyl-3,3-dimethyldihydrofuran-2(3H)-one (3g)

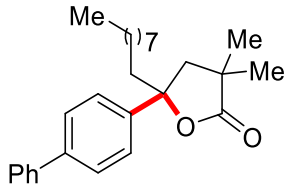
Substrate **1g** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (22.6 mg, 62% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.62 – 7.58 (m, 4H), 7.46 – 7.43 (m, 2H), 7.42 – 7.39 (m, 2H), 7.37 – 7.33 (m, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.37 (d, *J* = 13.0 Hz, 1H), 1.99-1.87 (m, 2H), 1.41 – 1.34 (m, 1H), 1.34 (s, 3H), 1.27 – 1.16 (m, 8H), 1.10 – 1.02 (m, 1H), 0.98 (s, 3H), 0.84 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.08, 143.60, 140.51, 140.16, 128.94, 127.56, 127.23, 127.13, 125.24, 86.00, 49.81, 44.60, 40.56, 31.87, 29.68, 29.19, 26.73, 26.31, 23.94, 22.71, 14.18.

HRMS (ESI-TOF) Calcd for $C_{25}H_{33}O_2$ $[M+H]^+$: 365.2481; found: 365.2482.

Melting point: 62 °C to 67 °C

IR (cm^{-1}) data: 2929, 1767, 1487, 1485, 1237, 1046, 767, 697.



5-([1,1'-biphenyl]-4-yl)-3,3,5-trimethyldihydrofuran-2(3H)-one (3h)

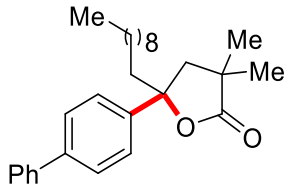
Substrate **1h** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (23.5 mg, 60% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.60 (ddt, *J* = 8.1, 6.1, 1.8 Hz, 4H), 7.46 – 7.42 (m, 2H), 7.42 – 7.39 (m, 2H), 7.37 – 7.33 (m, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.37 (d, *J* = 13.1 Hz, 1H), 1.99-1.87 (m, 2H), 1.42 – 1.34 (m, 1H), 1.34 (s, 3H), 1.27 – 1.17 (m, 12H), 1.11 – 1.03 (m, 1H), 0.98 (s, 3H), 0.86 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.08, 143.60, 140.50, 140.16, 128.94, 127.56, 127.22, 127.13, 125.24, 86.00, 49.80, 44.60, 40.56, 31.96, 29.71, 29.60, 29.51, 29.37, 26.73, 26.30, 23.93, 22.78, 14.22.

HRMS (ESI-TOF) Calcd for $C_{27}H_{37}O_2$ $[M+H]^+$: 393.2794; found: 393.2798.

Melting point: 59 °C to 62 °C

IR (cm^{-1}) data: 2926, 2853, 1767, 1457, 1239, 1211, 1007, 922, 767, 739, 697.



5-([1,1'-biphenyl]-4-yl)-5-decyl-3,3-dimethyldihydrofuran-2(3H)-one (3i)

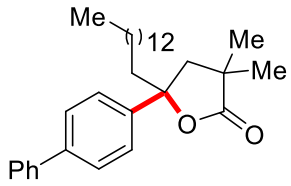
Substrate **1i** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as an off white solid (22.8 mg, 56% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.60 (ddt, *J* = 9.1, 6.3, 1.7 Hz, 4H), 7.45 – 7.43 (m, 2H), 7.42 – 7.39 (m, 2H), 7.35 (tt, *J* = 6.9, 1.1 Hz, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.37 (d, *J* = 13.0 Hz, 1H), 1.99-1.87 (m, 2H), 1.41 – 1.34 (m, 1H), 1.33 (s, 3H), 1.29 – 1.13 (m, 16H), 1.08 – 1.03 (m, 1H), 0.98 (s, 3H), 0.86 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.09, 143.61, 140.52, 140.17, 128.95, 127.56, 127.23, 127.14, 125.24, 86.01, 49.82, 44.61, 40.57, 32.01, 29.73, 29.68, 29.66, 29.53, 29.41, 26.74, 26.32, 23.94, 22.80, 14.24.

HRMS (ESI-TOF) Calcd for $C_{28}H_{39}O_2$ $[M+H]^+$: 407.2950; found: 407.2957.

Melting point: 57 °C to 61 °C

IR (cm^{-1}) data: 2924, 2853, 1767, 1487, 1239, 1211, 1007, 922, 767, 739, 697.



5-([1,1'-biphenyl]-4-yl)-3,3-dimethyl-5-tetradecyldihydrofuran-2(3H)-one (3j)

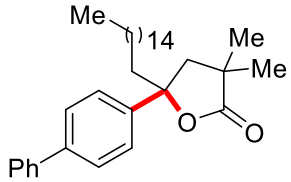
Substrate **1j** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as an off white solid (33.3 mg, 72% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.62 – 7.58 (m, 4H), 7.46 – 7.42 (m, 2H), 7.42 – 7.39 (m, 2H), 7.37 – 7.33 (m, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.37 (d, *J* = 13.1 Hz, 1H), 1.99-1.87 (m, 2H), 1.43 – 1.34 (m, 1H), 1.33 (s, 3H), 1.29 – 1.18 (m, 22H), 1.09 – 1.01 (m, 1H), 0.98 (s, 3H), 0.88 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.08, 143.61, 140.51, 140.17, 128.95, 127.56, 127.23, 127.14, 125.24, 86.01, 49.81, 44.61, 40.56, 32.06, 29.82, 29.80, 29.78, 29.76, 29.73, 29.67, 29.53, 29.49, 26.74, 26.31, 23.94, 22.83, 14.26.

HRMS (ESI-TOF) Calcd for $C_{32}H_{47}O_2$ $[M+H]^+$: 463.3576; found: 463.3572.

Melting point: 52 °C to 55 °C

IR (cm^{-1}) data: 2923, 2853, 1768, 1487, 1459, 1238, 1211, 841, 766, 739, 697.



5-([1,1'-biphenyl]-4-yl)-5-hexadecyl-3,3-dimethyldihydrofuran-2(3H)-one (3k)

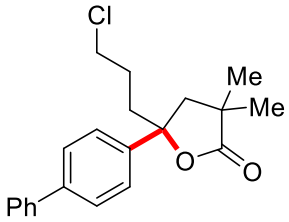
Substrate **1k** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (34.8 mg, 71% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.62 – 7.58 (m, 4H), 7.44 (t, *J* = 6.8 Hz, 2H), 7.42 – 7.39 (m, 2H), 7.35 (t, *J* = 6.8 Hz, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.37 (d, *J* = 13.0 Hz, 1H), 1.99 – 1.86 (m, 2H), 1.42 – 1.35 (m, 1H), 1.34 (s, 3H), 1.30 – 1.17 (m, 28H), 1.10 – 1.05 (m, 1H), 0.98 (s, 3H), 0.88 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.07, 143.61, 140.51, 140.17, 128.94, 127.56, 127.23, 127.13, 125.24, 86.00, 49.81, 44.61, 40.56, 32.06, 29.84, 29.83, 29.82, 29.81, 29.79, 29.76, 29.73, 29.67, 29.53, 29.50, 26.74, 26.31, 23.94, 22.83, 14.27.

HRMS (ESI-TOF) Calcd for $C_{34}H_{51}O_2$ $[M+H]^+$: 491.3889; found: 491.3908.

Melting point: 52 °C to 56 °C

IR (cm^{-1}) data: 2923, 2852, 1770, 1481, 1464, 1238, 766, 739, 697.



5-([1,1'-biphenyl]-4-yl)-5-(3-chloropropyl)-3,3-dimethyldihydrofuran-2(3H)-one (3l)

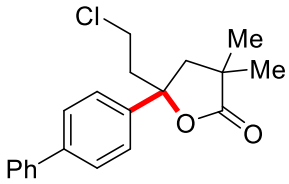
Substrate **1l** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as an off solid (18.9 mg, 55% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.60 (ddd, *J* = 7.8, 5.2, 1.5 Hz, 4H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.42 – 7.39 (m, 2H), 7.38 – 7.34 (m, 1H), 3.47 (t, *J* = 6.4 Hz, 2H), 2.57 (d, *J* = 13.1 Hz, 1H), 2.40 (d, *J* = 13.1 Hz, 1H), 2.19 (ddd, *J* = 14.2, 11.4, 4.6 Hz, 1H), 2.07 (ddd, *J* = 14.1, 11.1, 4.7 Hz, 1H), 1.93 – 1.85 (m, 1H), 1.53 (dddt, *J* = 14.1, 11.3, 6.6, 4.7 Hz, 1H), 1.35 (s, 3H), 0.99 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.79, 142.79, 140.52, 140.34, 128.97, 127.67, 127.47, 127.14, 125.14, 85.27, 50.42, 44.97, 41.81, 40.49, 27.27, 26.77, 26.30.

HRMS (ESI-TOF) Calcd for $C_{21}H_{24}ClO_2$ $[M+H]^+$: 343.1465; found: 343.1468.

Melting point: 98 °C to 102 °C

IR (cm^{-1}) data: 2965, 1766, 1487, 1452, 1211, 1137, 1055, 922, 766, 738, 698.



5-([1,1'-biphenyl]-4-yl)-5-(2-chloroethyl)-3,3-dimethyldihydrofuran-2(3H)-one (3m)

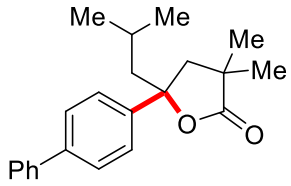
Substrate **1m** was arylated and lactonized following the general procedure with little modification by using 20 mol% $Pd(OAc)_2$ and 20 mol% ligand L14 (eluent: hexane/ethyl acetate = 5/1). The product was obtained as an off white solid (8.5 mg, 26% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.63 – 7.58 (m, 4H), 7.47 – 7.43 (m, 2H), 7.42 – 7.40 (m, 2H), 7.38 – 7.35 (m, 1H), 3.58 (td, *J* = 10.9, 5.1 Hz, 1H), 3.12 (td, *J* = 10.8, 5.5 Hz, 1H), 2.60 (d, *J* = 13.1 Hz, 1H), 2.50 (ddd, *J* = 14.1, 11.0, 5.6 Hz, 1H), 2.46 – 2.40 (m, 2H), 1.35 (s, 3H), 0.99 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.42, 141.63, 140.95, 140.22, 129.02, 127.79, 127.71, 127.17, 125.01, 84.35, 50.55, 47.21, 40.32, 39.28, 26.78, 26.26.

HRMS (ESI-TOF) Calcd for $C_{20}H_{22}ClO_2$ $[M+H]^+$: 329.1308; found: 329.1312.

Melting point:104 °C to 106 °C

IR (cm^{-1}) data: 2972, 1772, 1487, 1453, 1216, 1174, 1131, 1044, 767, 733, 697.



5-([1,1'-biphenyl]-4-yl)-5-isobutyl-3,3-dimethyldihydrofuran-2(3H)-one (3n)

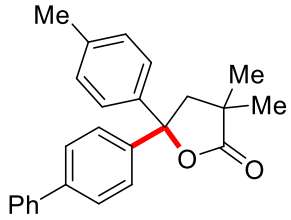
Substrate **1n** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (25.4 mg, 79% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 (ddd, *J* = 8.3, 4.2, 1.6 Hz, 4H), 7.46 – 7.41 (m, 4H), 7.37 – 7.33 (m, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.34 (d, *J* = 13.0 Hz, 1H), 1.98 (dd, *J* = 14.6, 5.0 Hz, 1H), 1.81 (dd, *J* = 14.6, 7.3 Hz, 1H), 1.53 – 1.46 (m, 1H), 1.33 (s, 3H), 0.95 (s, 3H), 0.94 (d, *J* = 6.6 Hz, 3H), 0.74 (d, *J* = 6.7 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.18, 143.54, 140.44, 140.12, 128.94, 127.57, 127.23, 127.11, 125.32, 86.24, 53.03, 51.41, 40.12, 26.78, 26.17, 24.68, 24.27, 23.68.

HRMS (ESI-TOF) Calcd for $C_{22}H_{27}O_2$ $[M+H]^+$: 323.2011; found: 323.2010.

Melting point: 110 °C to 114 °C

IR (cm^{-1}) data: 2937, 1785, 1487, 1385, 1233, 1210, 1104, 1032, 923, 760, 697.



5-([1,1'-biphenyl]-4-yl)-3,3-dimethyl-5-(p-tolyl)dihydrofuran-2(3H)-one (3o)

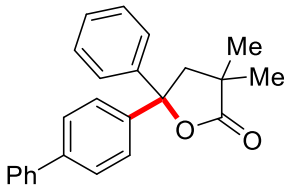
Substrate **1o** was arylated and lactonized following the general procedure with little modification by using 20 mol% $Pd(OAc)_2$ and 20 mol% ligand L14 (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (13.5 mg, 38% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.56 – 7.53 (m, 4H), 7.52 – 7.50 (m, 2H), 7.44 – 7.40 (m, 2H), 7.39 – 7.36 (m, 2H), 7.35 – 7.32 (m, 1H), 7.17 – 7.14 (m, 2H), 2.97 – 2.91 (m, 2H), 2.32 (s, 3H), 1.19 (s, 3H), 1.16 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.45, 144.10, 141.87, 140.51, 140.49, 137.55, 129.47, 128.92, 127.59, 127.43, 127.17, 125.54, 125.10, 86.24, 50.08, 40.82, 26.06, 25.97, 21.13.

HRMS (ESI-TOF) Calcd for $C_{25}H_{25}O_2$ $[M+H]^+$: 357.1855; found: 357.1856.

Melting point: 130 °C to 134 °C

IR (cm^{-1}) data: 2972, 1772, 1511, 1486, 1386, 1386, 1236, 1154, 1049, 760, 697.



5-([1,1'-biphenyl]-4-yl)-3,3-dimethyl-5-phenyldihydrofuran-2(3H)-one (3p)

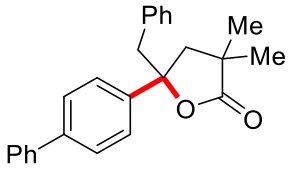
Substrate **1p** was arylated and lactonized following the general procedure with little modification by using 20 mol% $Pd(OAc)_2$ and 20 mol% ligand L14 (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (9.6 mg, 28% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.57 – 7.53 (m, 6H), 7.52 – 7.49 (m, 3H), 7.44 – 7.40 (m, 2H), 7.37 – 7.32 (m, 3H), 7.28 – 7.25 (m, 2H), 2.96 (s, 2H), 1.19 (s, 3H), 1.16 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.35, 144.82, 143.83, 140.61, 140.44, 128.94, 128.82, 127.78, 127.63, 127.45, 127.18, 125.62, 125.15, 86.19, 50.11, 40.79, 26.04, 25.96.

HRMS (ESI-TOF) Calcd for $C_{24}H_{23}O_2$ $[M+H]^+$: 343.1698; found: 343.1701.

Melting point: 118 ºC to 122 ºC

IR (cm^{-1}) data: 2972, 1774, 1488, 1237, 1154, 1046, 731, 697.



5-([1,1'-biphenyl]-4-yl)-5-benzyl-3,3-dimethyldihydrofuran-2(3H)-one (3q)

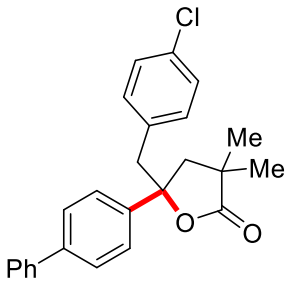
Substrate **1q** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (17.8 mg, 50% yield). R_f = 0.25; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.60 (dt, *J* = 8.2, 1.6 Hz, 2H), 7.58 – 7.55 (m, 2H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.38 – 7.33 (m, 3H), 7.21 – 7.16 (m, 2H), 7.02 – 6.97 (m, 2H), 3.23 (d, *J* = 14.1 Hz, 1H), 3.10 (d, *J* = 14.1 Hz, 1H), 2.55 (d, *J* = 13.1 Hz, 1H), 2.43 (d, *J* = 13.1 Hz, 1H), 1.12 (s, 3H), 0.96 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.53, 143.06, 140.49, 140.32, 133.73, 133.12, 132.27, 128.98, 128.36, 127.68, 127.20, 127.13, 125.41, 85.29, 49.41, 47.85, 40.57, 26.36, 26.20.

HRMS (ESI-TOF) Calcd for $C_{25}H_{25}O_2$ $[M+H]^+$: 357.1849; found: 357.1856.

Melting point: 129 ºC to 131 ºC

IR (cm^{-1}) data: 2971, 1767, 1489, 1244, 1142, 1030, 841, 766, 740, 698.



5-([1,1'-biphenyl]-4-yl)-5-(4-chlorobenzyl)-3,3-dimethyldihydrofuran-2(3H)-one (3r)

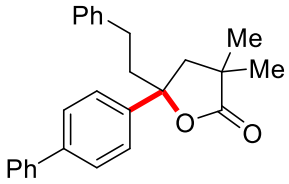
Substrate **1r** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as an off white solid (17.6 mg, 45% yield). R_f = 0.25; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.58 (m, 2H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.37 – 7.32 (m, 3H), 7.19 (d, *J* = 8.4 Hz, 2H), 6.99 (d, *J* = 8.4 Hz, 2H), 3.23 (d, *J* = 14.1 Hz, 1H), 3.10 (d, *J* = 14.1 Hz, 1H), 2.55 (d, *J* = 13.1 Hz, 1H), 2.43 (d, *J* = 13.1 Hz, 1H), 1.12 (s, 3H), 0.96 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.53, 143.06, 140.48, 140.31, 133.73, 133.12, 132.27, 128.97, 128.35, 127.68, 127.20, 127.13, 125.41, 85.29, 49.40, 47.84, 40.57, 26.36, 26.20.

HRMS (ESI-TOF) Calcd for $C_{25}H_{24}ClO_2$ $[M+H]^+$: 391.1465; found: 391.1467.

Melting point: 130 °C to 132 °C

IR (cm^{-1}) data: 2971, 1766, 1489, 1452, 1245, 1141, 1040, 841, 766, 739, 697.



5-([1,1'-biphenyl]-4-yl)-3,3-dimethyl-5-phenethyldihydrofuran-2(3H)-one (3s)

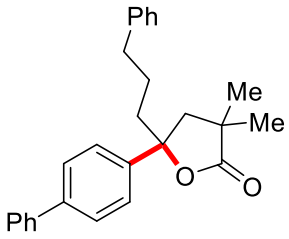
Substrate **1s** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (28.5 mg, 77% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 – 7.61 (m, 4H), 7.49 – 7.44 (m, 4H), 7.39 – 7.35 (m, 1H), 7.27 – 7.22 (m, 2H), 7.16 (t, *J* = 7.4 Hz, 1H), 7.09 (d, *J* = 7.1 Hz, 2H), 2.75 (td, *J* = 13.0, 4.7 Hz, 1H), 2.59 (d, *J* = 13.1 Hz, 1H), 2.43 (s, 1H), 2.37 – 2.20 (m, 3H), 1.36 (s, 3H), 1.02 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.99, 143.08, 141.42, 140.43, 128.98, 128.56, 128.39, 127.64, 127.45, 127.16, 126.09, 125.22, 85.51, 50.23, 46.61, 40.56, 30.46, 26.80, 26.34.

HRMS (ESI-TOF) Calcd for $C_{26}H_{27}O_2$ $[M+H]^+$: 371.2011; found: 371.2007.

Melting point: 108 °C to 112 °C

IR (cm^{-1}) data: 2970, 1765, 1488, 1211, 1142, 1075, 1047, 766, 739, 698.



5-([1,1'-biphenyl]-4-yl)-3,3-dimethyl-5-(3-phenylpropyl)dihydrofuran-2(3H)-one (3t)

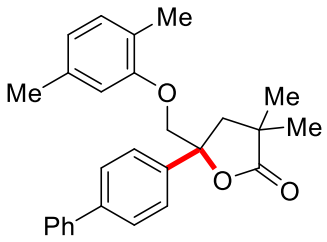
Substrate **1t** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (30.8 mg, 80% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 – 7.59 (m, 4H), 7.49 – 7.45 (m, 2H), 7.43 – 7.40 (m, 2H), 7.40 – 7.36 (m, 1H), 7.29 – 7.25 (m, 2H), 7.21 – 7.17 (m, 1H), 7.13 – 7.09 (m, 2H), 2.62 (ddd, *J* = 14.9, 8.8, 6.4 Hz, 1H), 2.59-2.52 (m, 2H), 2.37 (d, *J* = 13.0 Hz, 1H), 2.06-1.95 (m, 2H), 1.80-1.74 (m, 1H), 1.46-1.41 (m, 1H), 1.34 (s, 3H), 1.00 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.98, 143.29, 141.80, 140.46, 140.25, 128.95, 128.50, 128.45, 127.59, 127.30, 127.13, 125.98, 125.22, 85.80, 49.99, 43.95, 40.51, 35.73, 26.71, 26.27, 25.59.

HRMS (ESI-TOF) Calcd for $C_{27}H_{29}O_2$ $[M+H]^+$: 385.2168; found: 385.2172.

Melting point: 105 °C to 108 °C

IR (cm^{-1}) data: 2935, 1765, 1487, 1453, 1235, 1138, 1047, 920, 752, 698.



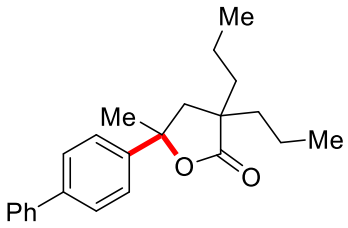
5-([1,1'-biphenyl]-4-yl)-5-((2,5-dimethylphenoxy)methyl)-3,3-dimethyldihydrofuran-2(3H)-one (3u)

Substrate **1u** was arylated and lactonized following the general procedure with little modification by using 30 mol% $Pd(OAc)_2$ and 30 mol% ligand L14 (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (10.0 mg, 25% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.3 Hz, 2H), 7.62 – 7.56 (m, 4H), 7.46 (t, *J* = 7.7 Hz, 2H), 7.37 (t, *J* = 7.4 Hz, 1H), 7.01 (d, *J* = 7.5 Hz, 1H), 6.69 (d, *J* = 7.5 Hz, 1H), 6.56 (s, 1H), 4.11 (d, *J* = 10.0 Hz, 1H), 4.06 (d, *J* = 10.0 Hz, 1H), 2.97 (d, *J* = 13.0 Hz, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.27 (s, 3H), 2.19 (s, 3H), 1.42 (s, 3H), 1.09 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.51, 156.27, 141.27, 140.71, 140.40, 136.80, 130.73, 129.01, 127.76, 127.52, 127.24, 125.76, 123.80, 121.78, 112.38, 84.22, 73.66, 44.60, 40.80, 26.06, 21.43, 16.15.

HRMS (ESI-TOF) Calcd for $C_{27}H_{29}O_3$ $[M+H]^+$: 401.2117; found: 401.2112.

IR (cm^{-1}) data: 2971, 2926, 1775, 1510, 1453, 1260, 1130, 1069, 767, 698.



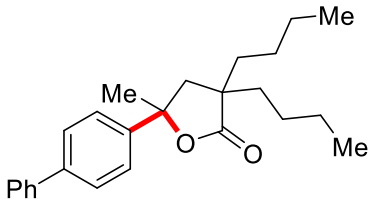
5-([1,1'-biphenyl]-4-yl)-5-methyl-3,3-dipropyldihydrofuran-2(3H)-one (3v)

Substrate **1v** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (22.2 mg, 66% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.58 (m, 4H), 7.46 – 7.42 (m, 4H), 7.37 – 7.34 (m, 1H), 2.52 (d, *J* = 13.4 Hz, 1H), 2.41 (d, *J* = 13.3 Hz, 1H), 1.73 (s, 3H), 1.67 – 1.57 (m, 2H), 1.54 – 1.44 (m, 1H), 1.37 – 1.25 (m, 3H), 1.21 – 1.02 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.69 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 180.86, 145.74, 140.58, 140.19, 128.95, 127.56, 127.31, 127.16, 124.50, 83.39, 48.83, 45.98, 39.48, 32.59, 17.97, 17.62, 14.61, 14.42.

HRMS (ESI-TOF) Calcd for $C_{23}H_{29}O_2$ $[M+H]^+$: 337.2168; found: 337.2171.

IR (cm^{-1}) data: 2959, 2932, 1769, 1487, 1452, 1074, 767, 734, 697.



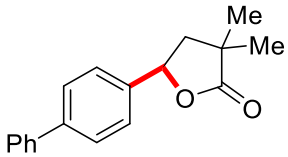
5-([1,1'-biphenyl]-4-yl)-3,3-dibutyl-5-ethyldihydrofuran-2(3H)-one (3w)

Substrate **1w** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (30.3 mg, 80% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.57 (m, 4H), 7.46 – 7.42 (m, 2H), 7.41 – 7.38 (m, 2H), 7.37 – 7.33 (m, 1H), 2.46 (d, *J* = 13.3 Hz, 1H), 2.41 (d, *J* = 13.3 Hz, 1H), 2.04 – 1.93 (m, 2H), 1.68 – 1.57 (m, 2H), 1.46 – 1.21 (m, 6H), 1.11 – 0.97 (m, 4H), 0.94 (t, *J* = 7.2 Hz, 3H), 0.83 (t, *J* = 7.3 Hz, 3H), 0.65 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.07, 143.78, 140.65, 140.08, 128.93, 127.51, 127.14, 127.10, 125.32, 86.22, 48.19, 44.77, 37.74, 36.79, 36.50, 26.80, 26.36, 23.27, 22.85, 14.13, 13.74, 8.55.

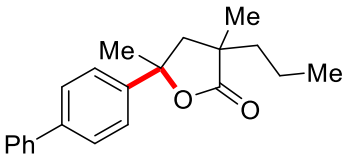
HRMS (ESI-TOF) Calcd for $C_{26}H_{35}O_2$ $[M+H]^+$: 379.2637; found: 379.2636.

IR (cm^{-1}) data: 2955, 2932, 1763, 1461, 1098, 968, 765, 734.



5-([1,1'-biphenyl]-4-yl)-3,3-dimethyldihydrofuran-2(3H)-one (3x)

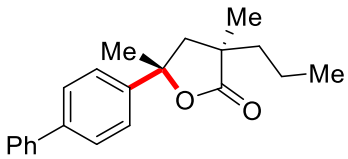
Substrate **1x** was tried arylation and lactonization following the general procedure. No reaction observed.



5-([1,1'-biphenyl]-4-yl)-3,5-dimethyl-3-propyldihydrofuran-2(3H)-one (3y)

Substrate **1y** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 7/1). The products (two diastereomers) were obtained as a gummy liquid (diastereomer-1; 14.1 mg, 46% yield & diastereomer-2; 8.9 mg, 29% yield). Overall yield of two diastereomers were 75%.

diastereomer-1; R_f = 0.4

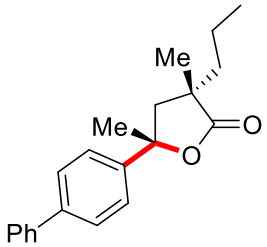


^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.58 (m, 4H), 7.47 – 7.42 (m, 4H), 7.37 – 7.33 (m, 1H), 2.46 (d, *J* = 2.6 Hz, 2H), 1.73 (s, 3H), 1.63 – 1.58 (m, 2H), 1.53 – 1.45 (m, 1H), 1.33 – 1.24 (m, 1H), 1.00 – 0.95 (m, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.59, 145.26, 140.51, 140.33, 128.94, 127.57, 127.40, 127.15, 124.62, 83.55, 48.05, 44.81, 41.25, 32.22, 24.33, 17.92, 14.52.

HRMS (ESI-TOF) Calcd for $C_{21}H_{25}O_2$ $[M+H]^+$: 309.1855; found: 309.1861.

IR (cm^{-1}) data: 2964, 2932, 1763, 1487, 1451, 1234, 1061, 955, 841, 766.



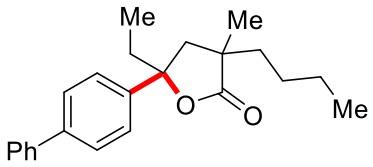
diastereomer-1; R_f = 0.3

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.58 (m, 4H), 7.47 – 7.42 (m, 4H), 7.36 (tt, *J* = 6.9, 1.2 Hz, 1H), 2.63 (d, *J* = 13.3 Hz, 1H), 2.29 (d, *J* = 13.2 Hz, 1H), 1.76 (s, 3H), 1.41 – 1.35 (m, 4H), 1.34 – 1.27 (m, 1H), 1.27 – 1.19 (m, 1H), 1.15 – 1.05 (m, 1H), 0.74 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.54, 145.39, 140.57, 140.30, 128.95, 127.57, 127.36, 127.16, 124.45, 83.49, 48.22, 44.79, 40.67, 32.31, 25.03, 17.67, 14.34.

HRMS (ESI-TOF) Calcd for $C_{21}H_{25}O_2$ $[M+H]^+$: 309.1855; found: 308.1861.

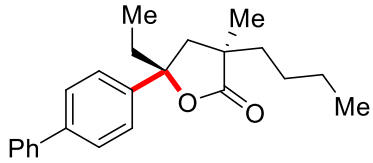
IR (cm^{-1}) data: 2962, 2932, 1764, 1487, 1217, 1071, 766, 733, 697.



5-([1,1'-biphenyl]-4-yl)-3-butyl-5-ethyl-3-methyldihydrofuran-2(3H)-one (3z)

Substrate **1z** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 6/1). The products (two diastereomers) was obtained as a gummy liquid (diastereomer-1; 14.1 mg, 42% yield & diastereomer-2; 10.4 mg, 31% yield). Overall yield of two diastereomers were 73%.

diastereomer-1; R_f = 0.4



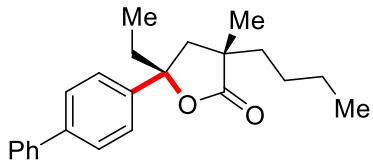
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.57 (m, 4H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.41 (d, *J* = 8.5 Hz, 2H), 7.35 (t, *J* = 7.4 Hz, 1H), 2.45 (d, *J* = 13.1 Hz, 1H), 2.40 (d, *J* = 13.1 Hz, 1H), 1.98 (ddt, *J* = 24.3, 14.2, 7.2 Hz, 2H), 1.64 – 1.58 (m, 2H), 1.45 – 1.32 (m, 3H), 1.28 – 1.20 (m, 1H), 0.96 – 0.91 (m, 6H), 0.83 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.75, 143.47, 140.53, 140.19, 128.95, 127.56, 127.22, 127.15, 125.38, 86.34, 46.49, 44.34, 38.69, 37.39, 26.82, 24.49, 23.19, 14.13, 8.47.

HRMS (ESI-TOF) Calcd for $C_{23}H_{29}O_2$ $[M+H]^+$: 337.2168; found: 337.2172.

IR (cm^{-1}) data: 2958, 2930, 1767, 1459, 1225, 1079, 766, 695, 616.

diastereomer-1; R_f = 0.3

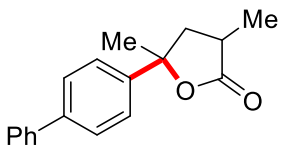


^{1}H NMR (600 MHz, $CDCl_3$) δ 7.59 (td, *J* = 6.7, 1.6 Hz, 4H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.41 – 7.37 (m, 2H), 7.37 – 7.33 (m, 1H), 2.60 (d, *J* = 13.3 Hz, 1H), 2.28 (d, *J* = 13.2 Hz, 1H), 2.06 – 1.94 (m, 2H), 1.36 – 1.29 (m, 4H), 1.28 – 1.20 (m, 2H), 1.16 – 0.99 (m, 4H), 0.84 (t, *J* = 7.3 Hz, 3H), 0.67 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.78, 143.52, 140.64, 140.17, 128.95, 127.54, 127.17, 127.16, 125.24, 86.29, 46.95, 44.32, 37.97, 37.61, 26.38, 24.61, 22.78, 13.76, 8.48.

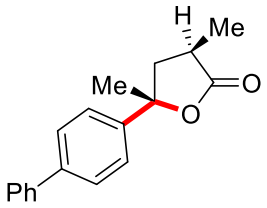
HRMS (ESI-TOF) Calcd for $C_{23}H_{29}O_2$ $[M+H]^+$: 337.2168; found: 337.2169

IR (cm^{-1}) data: 2961, 2932, 1764, 1487, 1450, 1206, 969, 842, 766, 697.



5-([1,1'-biphenyl]-4-yl)-3,5-dimethyldihydrofuran-2(3H)-one (3aa)

Substrate **1aa** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The products (two diastereomers) were obtained as a gummy liquid (diastereomer-1; 2.4 mg, 9% yield & diastereomer-1; 1.6 mg, 6% yield).



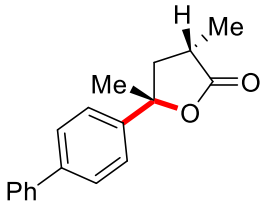
diastereomer-1; R_f = 0.3

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.58 (ddd, *J* = 7.4, 5.8, 1.3 Hz, 4H), 7.44 (t, *J* = 7.9 Hz, 4H), 7.37 – 7.35 (m, 1H), 2.81 (dd, *J* = 12.5, 8.2 Hz, 1H), 2.61 – 2.54 (m, 1H), 2.07 (t, *J* = 12.4 Hz, 1H), 1.77 (s, 3H), 1.28 (d, *J* = 7.1 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 179.44, 143.00, 140.76, 140.53, 128.99, 127.65, 127.46, 127.22, 124.89, 84.65, 45.20, 35.22, 30.46, 14.86

HRMS (ESI-TOF) Calcd for $C_{18}H_{19}O_2$ $[M+H]^+$: 267.1385; found: 267.1385.

IR (cm^{-1}) data: 2978, 2931, 1769, 1487, 1225, 1140, 952, 766, 697.



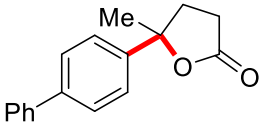
diastereomer-2; R_f = 0.25

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.59 (ddd, *J* = 8.1, 6.7, 1.4 Hz, 4H), 7.47 – 7.43 (m, 4H), 7.38 – 7.34 (m, 1H), 2.96 (ddq, *J* = 10.8, 8.6, 7.2 Hz, 1H), 2.74 (dd, *J* = 12.6, 8.8 Hz, 1H), 2.14 (dd, *J* = 12.5, 11.0 Hz, 1H), 1.72 (s, 3H), 1.28 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 178.97, 144.48, 140.66, 140.64, 128.97, 127.61, 127.44, 127.23, 124.55, 84.52, 44.18, 35.46, 28.93, 15.60.

HRMS (ESI-TOF) Calcd for $C_{18}H_{19}O_2$ $[M+H]^+$: 267.1385; found: 267.1385.

IR (cm^{-1}) data: 2975, 2935, 1769, 1487, 1308, 1215, 1154, 951, 842, 767.



5-([1,1'-biphenyl]-4-yl)-5-methyldihydrofuran-2(3H)-one (3bb)

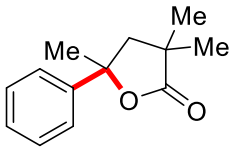
Substrate **1bb** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (2.2 mg, 9% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.58 (m,

4H), 7.47 – 7.44 (m, 4H), 7.36 (t, *J* = 7.3 Hz, 1H), 2.70 – 2.63 (m, 1H), 2.60 – 2.51 (m, 2H), 2.49 – 2.42 (m, 1H), 1.77 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 176.63, 143.40, 140.79, 140.56, 128.98, 127.65, 127.48, 127.23, 124.76, 87.06, 36.36, 29.57, 29.15.

HRMS (ESI-TOF) Calcd for $C_{17}H_{17}O_2$ $[M+H]^+$: 253.1223; found: 253.1228.

IR (cm^{-1}) data: 2979, 1773, 1486, 1240, 1133, 1076, 841, 766, 733, 698.



3,3,5-trimethyl-5-phenyldihydrofuran-2(3H)-one (4a)

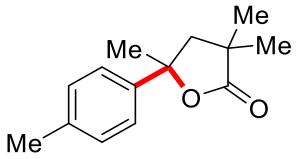
Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (14.7 mg, 72% yield). R_f = 0.4; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.42 – 7.32 (m, 4H), 7.29 – 7.25 (m, 1H), 2.56 (d, *J* = 13.0 Hz, 1H), 2.35 (d, *J* = 13.0 Hz, 1H), 1.71 (s, 3H), 1.34 (s, 3H), 0.97 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.93, 146.04, 128.73, 127.46, 124.11, 83.58, 50.84, 40.99, 32.19, 26.85, 26.06.

HRMS (ESI-TOF) Calcd for $C_{13}H_{17}O_2$ $[M+H]^+$: 205.1229; found: 205.1226.

Melting point: 48 °C to 51 °C

IR (cm^{-1}) data: 2976, 1766, 1447, 1250, 1218, 1083, 1062, 951, 702.



3,3,5-trimethyl-5-(p-tolyl)dihydrofuran-2(3H)-one (4b)

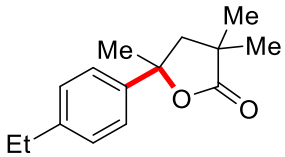
Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (15.3 mg, 70% yield). R_f = 0.4; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.28 – 7.25 (m, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.35 – 2.30 (m, 4H), 1.69 (s, 3H), 1.33 (s, 3H), 0.98 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.02, 143.10, 137.14, 129.37, 124.05, 83.65, 50.82, 41.03, 32.26, 26.86, 26.09, 21.10.

HRMS (ESI-TOF) Calcd for $C_{14}H_{19}O_2$ $[M+H]^+$: 219.1385; found: 219.1389.

Melting point: 58 °C to 60 °C

IR (cm^{-1}) data: 2974, 2930, 1766, 1513, 1384, 1250, 1068, 950, 819.



5-(4-ethylphenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4c)

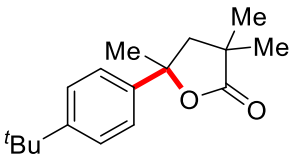
Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (19.7 mg, 85% yield). R_f = 0.4; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32 – 7.27 (m, 2H), 7.18 (d, *J* = 8.2 Hz, 2H), 2.64 (q, *J* = 7.6 Hz, 2H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.33 (d, *J* = 13.0 Hz, 1H), 1.69 (s, 3H), 1.33 (s, 3H), 1.23 (t, *J* = 7.6 Hz, 3H), 0.98 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.03, 143.44, 143.27, 128.14, 124.08, 83.66, 50.79, 41.02, 32.23, 28.48, 26.88, 26.12, 15.54.

HRMS (ESI-TOF) Calcd for $C_{15}H_{21}O_2$ $[M+H]^+$: 233.1542; found: 233.1544.

Melting point: 61 °C to 65 °C

IR (cm^{-1}) data: 2971, 2932, 1765, 1454, 1295, 1250, 1217, 1066, 950, 834.



5-([1,1'-biphenyl]-4-yl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4d)

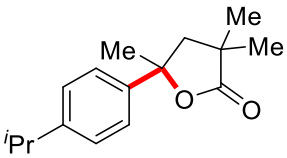
Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (17.7 mg, 68% yield). R_f = 0.4; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.38 – 7.34 (m, 2H), 7.31 – 7.28 (m, 2H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.33 (d, *J* = 13.0 Hz, 1H), 1.70 (s, 3H), 1.34 (s, 3H), 1.31 (s, 9H), 1.00 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.05, 150.36, 142.94, 125.55, 123.81, 83.63, 50.72, 41.02, 34.59, 32.18, 31.45, 26.94, 26.21.

HRMS (ESI-TOF) Calcd for $C_{17}H_{25}O_2$ $[M+H]^+$: 261.1855; found: 261.1856.

Melting point: 85 °C to 89 °C

IR (cm^{-1}) data: 2966, 1768, 1463, 1251, 1069, 950, 633.



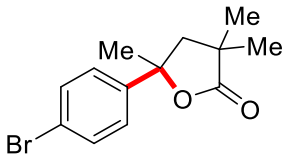
5-(4-isopropylphenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4e)

Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (18.5 mg, 75% yield). R_f = 0.4; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 – 7.27 (m, 2H), 7.20 (d, *J* = 8.2 Hz, 2H), 2.90 (sept, *J* = 6.9 Hz, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.33 (d, *J* = 13.0 Hz, 1H), 1.69 (s, 3H), 1.34 (s, 3H), 1.24 (d, *J* = 7.0 Hz, 6H), 0.99 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.05, 148.06, 143.34, 126.70, 124.06, 83.67, 50.76, 41.02, 33.77, 32.21, 26.92, 26.17, 24.06.

HRMS (ESI-TOF) Calcd for $C_{16}H_{23}O_2$ $[M+H]^+$: 247.1698; found: 247.1697.

IR (cm^{-1}) data: 2976, 1749, 1456, 1385, 1252, 1068, 950, 836, 611.



5-(4-bromophenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4f)

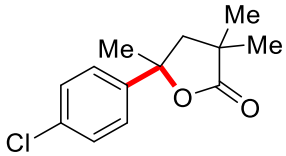
Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (13.0 mg, 46% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.50 – 7.46 (m, 2H), 7.27 – 7.24 (m, 2H), 2.49 (d, *J* = 13.1 Hz, 1H), 2.34 (d, *J* = 13.1 Hz, 1H), 1.67 (s, 3H), 1.33 (s, 3H), 0.98 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.56, 145.16, 131.88, 125.98, 121.46, 83.06, 50.71, 40.94, 32.08, 26.77, 26.11.

HRMS (ESI-TOF) Calcd for $C_{13}H_{16}BrO_2$ $[M+H]^+$: 283.0334; found: 283.0340.

Melting point: 83 °C to 86 °C

IR (cm^{-1}) data: 2974, 1765, 1487, 1248, 1248, 1066, 1008, 826.



5-(4-chlorophenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4g)

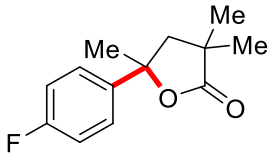
Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (10.5 mg, 44% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.37 – 7.29 (m, 4H), 2.50 (d, *J* = 13.1 Hz, 1H), 2.35 (d, *J* = 13.1 Hz, 1H), 1.68 (s, 3H), 1.33 (s, 3H), 0.98 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.59, 144.63, 133.39, 128.93, 125.64, 83.06, 50.76, 40.96, 32.13, 26.78, 26.11.

HRMS (ESI-TOF) Calcd for $C_{13}H_{16}ClO_2$ $[M+H]^+$: 239.0839; found: 239.0844.

Melting point: 68 °C to 72 °C

IR (cm^{-1}) data: 2975, 1766, 1491, 1249, 1067, 952, 830.



5-(4-fluorophenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4h)

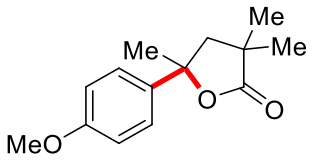
Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (9.3 mg, 42% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.38 – 7.32 (m, 2H), 7.07 – 7.01 (m, 2H), 2.51 (d, *J* = 13.1 Hz, 1H), 2.34 (d, *J* = 13.0 Hz, 1H), 1.68 (s, 3H), 1.34 (s, 3H), 0.98 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.69, 162.05 (d, *J* = 246.4 Hz), 141.88 (d, *J* = 3.2 Hz), 125.91 (d, *J* = 8.0 Hz), 115.60 (d, *J* = 21.6 Hz), 83.19, 50.83, 40.98, 32.32, 26.82, 26.08.

^{19}F NMR (376 MHz, $CDCl_3$) δ -115.21.

HRMS (ESI-TOF) Calcd for $C_{13}H_{16}FO_2$ $[M+H]^+$: 223.1134; found: 223.1136.

IR (cm^{-1}) data: 2976, 1768, 1604, 1501, 1229, 1068, 951, 838.



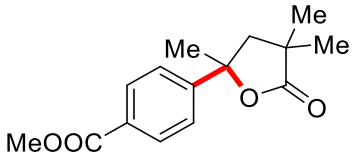
5-(4-methoxyphenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4i)

Substrate **1a** was arylated and lactonized following the general procedure with little modification by using 20 mol% $Pd(OAc)_2$ and 20 mol% ligand L14 (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (13.6 mg, 58% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 – 7.27 (m, 2H), 6.90 – 6.86 (m, 2H), 3.80 (s, 3H), 2.52 (d, *J* = 13.0 Hz, 1H), 2.31 (d, *J* = 13.0 Hz, 1H), 1.68 (s, 3H), 1.33 (s, 3H), 0.98 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.02, 158.86, 138.11, 125.37, 114.01, 83.54, 55.43, 50.79, 41.06, 32.34, 26.88, 26.07.

HRMS (ESI-TOF) Calcd for $C_{14}H_{19}O_3$ $[M+H]^+$: 235.1334; found: 235.1333.

IR (cm^{-1}) data: 2974, 1765, 1512, 1248, 1181, 1068, 834, 605.



methyl 4-(2,4,4-trimethyl-5-oxotetrahydrofuran-2-yl)benzoate (4j)

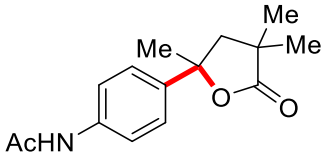
Substrate **1a** was arylated and lactonized following the general procedure with little modification by using 20 mol% $Pd(OAc)_2$ and 20 mol% ligand L14 (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (12.1 mg, 46% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.03 (d, *J* = 8.6 Hz, 2H), 7.46 (d, *J* = 8.5 Hz, 2H), 3.92 (s, 3H), 2.54 (d, *J* = 13.1 Hz, 1H), 2.38 (d, *J* = 13.1 Hz, 1H), 1.71 (s, 3H), 1.34 (s, 3H), 0.96 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.54, 166.76, 151.02, 130.16, 129.50, 124.26, 83.25, 52.33, 50.80, 40.94, 31.93, 26.75, 26.05.

HRMS (ESI-TOF) Calcd for $C_{15}H_{19}O_4$ $[M+H]^+$: 263.1283; found: 263.1285.

Melting point: 81 °C to 85 °C

IR (cm^{-1}) data: 2975, 1768, 1722, 1437, 1280, 1247, 1106, 1068, 755.



N-(4-(2,4,4-trimethyl-5-oxotetrahydrofuran-2-yl)phenyl)acetamide (4k)

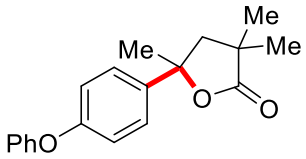
Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 3/1). The product was obtained as an off white solid (16.7 mg, 64% yield). R_f = 0.2; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.6 Hz, 2H), 7.36 (s, 1H), 7.32 (d, *J* = 8.6 Hz, 2H), 2.53 (d, *J* = 13.0 Hz, 1H), 2.33 (d, *J* = 13.0 Hz, 1H), 2.18 (s, 3H), 1.68 (s, 3H), 1.33 (s, 3H), 0.97 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 182.03, 168.54, 141.79, 137.26, 124.86, 120.06, 83.53, 50.71, 41.05, 32.19, 26.84, 26.08, 24.71.

HRMS (ESI-TOF) Calcd for $C_{15}H_{19}NO_3$ $[M+H]^+$: 262.1443; found: 262.1443.

Melting point: 90 °C to 93 °C

IR (cm^{-1}) data: 2976, 2361, 1762, 1670, 1602, 1533, 1318, 1252, 1068, 838.



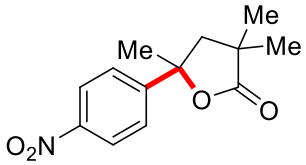
3,3,5-trimethyl-5-(4-phenoxyphenyl)dihydrofuran-2(3H)-one (4l)

Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (26.7 mg, 90% yield). R_f = 0.2; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.31 (m, 4H), 7.14 – 7.10 (m, 1H), 7.03 – 6.96 (m, 4H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.34 (d, *J* = 13.0 Hz, 1H), 1.71 (s, 3H), 1.35 (s, 3H), 1.02 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.85, 156.97, 156.70, 140.72, 129.95, 125.64, 123.67, 119.21, 118.74, 83.41, 50.78, 41.03, 32.29, 26.88, 26.15.

HRMS (ESI-TOF) Calcd for $C_{19}H_{20}O_3$ $[M+H]^+$: 297.1491; found: 297.1492.

IR (cm^{-1}) data: 2974, 1765, 1589, 1506, 1488, 1233, 1067, 870, 693.



3,3,5-trimethyl-5-(4-nitrophenyl)dihydrofuran-2(3H)-one (4m)

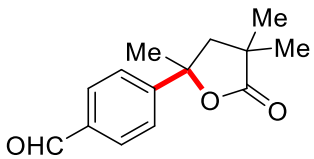
Substrate **1a** was arylated and lactonized following the general procedure with little modification by using 20 mol% $Pd(OAc)_2$ and 20 mol% ligand L14 (eluent: hexane/ethyl acetate = 4/1). The product was obtained as a light-yellow solid (13.7 mg, 55% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.26 – 8.20 (m, 2H), 7.60 – 7.55 (m, 2H), 2.53 (d, *J* = 13.2 Hz, 1H), 2.44 (d, *J* = 13.2 Hz, 1H), 1.73 (s, 3H), 1.36 (s, 3H), 0.98 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.07, 153.21, 147.38, 125.28, 124.17, 82.76, 50.73, 40.89, 31.89, 26.69, 26.18.

HRMS (ESI-TOF) Calcd for $C_{13}H_{16}NO_4$ $[M+H]^+$: 250.1079; found: 250.1078.

Melting point: 85 °C to 88 °C

IR (cm^{-1}) data: 2977, 1769, 1520, 1387, 1348, 1068, 856, 701.



4-(2,4,4-trimethyl-5-oxotetrahydrofuran-2-yl)benzaldehyde (4n)

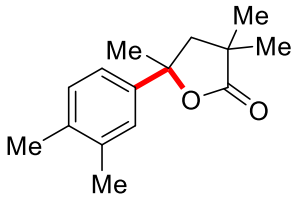
Substrate **1a** was arylated and lactonized following the general procedure with little modification by using 20 mol% $Pd(OAc)_2$ and 20 mol% ligand L14 (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (16.3 mg, 70% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.02 (s, 1H), 7.92 – 7.87 (m, 2H), 7.59 – 7.55 (m, 2H), 2.55 (d, *J* = 13.1 Hz, 1H), 2.41 (d, *J* = 13.1 Hz, 1H), 1.73 (s, 3H), 1.36 (s, 3H), 0.97 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 191.74, 181.39, 152.70, 135.74, 130.32, 124.92, 83.13, 50.79, 40.93, 31.90, 26.75, 26.12.

HRMS (ESI-TOF) Calcd for $C_{14}H_{17}O_3$ $[M+H]^+$: 233.1178; found: 233.1177.

Melting point: 132 °C to 136 °C

IR (cm^{-1}) data: 2975, 1767, 1700, 1610, 1250, 1215, 1068, 953, 617.



5-(3,4-dimethylphenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4o)

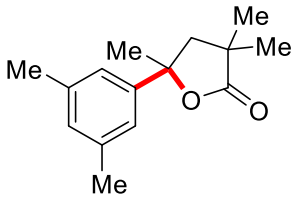
Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (19.7 mg, 85% yield). R_f = 0.4; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.15 (s, 1H), 7.13 – 7.06 (m, 2H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.32 (d, *J* = 13.0 Hz, 1H), 2.27 (s, 3H), 2.24 (s, 3H), 1.68 (s, 3H), 1.33 (s, 3H), 0.99 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.09, 143.53, 136.95, 135.74, 129.87, 125.32, 121.47, 83.64, 50.76, 41.03, 32.31, 26.86, 26.15, 20.08, 19.46.

HRMS (ESI-TOF) Calcd for $C_{15}H_{21}O_3$ $[M+H]^+$: 233.1542; found: 233.1545.

Melting point: 60 °C to 65 °C

IR (cm^{-1}) data: 2973, 1764, 1450, 1385, 1253, 1220, 1070, 950, 824.



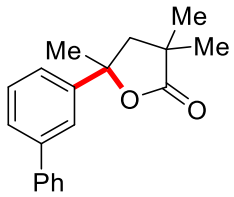
5-(3,5-dimethylphenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4p)

Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (20.2 mg, 87% yield). R_f = 0.4; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.00 – 6.95 (m, 2H), 6.92 – 6.89 (m, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.33 – 2.30 (m, 7H), 1.68 (s, 3H), 1.33 (s, 3H), 1.00 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.09, 146.06, 138.30, 129.02, 129.02, 121.87, 121.86, 83.69, 50.80, 41.01, 32.27, 26.88, 26.18, 21.57.

HRMS (ESI-TOF) Calcd for $C_{15}H_{21}O_2$ $[M+H]^+$: 233.1542; found: 233.1542.

IR (cm^{-1}) data: 2974, 1764, 1605, 1450, 1227, 1071, 942, 851, 709.



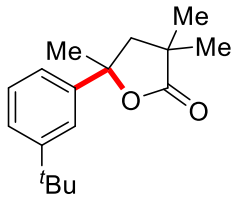
5-([1,1'-biphenyl]-3-yl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4q)

Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (23.8 mg, 85% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 – 7.57 (m, 3H), 7.53 – 7.50 (m, 1H), 7.47-7.42 (m, 3H), 7.37 (t, *J* = 7.4 Hz, 2H), 2.62 (d, *J* = 13.0 Hz, 1H), 2.40 (d, *J* = 13.0 Hz, 1H), 1.76 (s, 3H), 1.36 (s, 3H), 1.03 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.87, 146.66, 141.78, 140.90, 129.20, 128.95, 128.94, 127.70, 127.37, 127.36, 127.35, 126.30, 126.29, 123.06, 122.88, 83.60, 50.86, 41.00, 32.29, 26.84, 26.18.

HRMS (ESI-TOF) Calcd for $C_{19}H_{21}O_2$ $[M+H]^+$: 281.1542; found: 281.1548.

IR (cm^{-1}) data: 2974, 1763, 1479, 1452, 1237, 1065, 951, 758, 702.



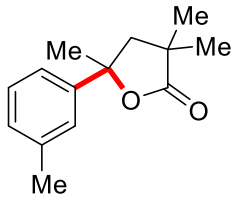
5-(3-(tert-butyl)phenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4r)

Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (19.5 mg, 75% yield). R_f = 0.4; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.41-7.39 (m, 1H), 7.32 – 7.27 (m, 2H), 7.17 (dt, *J* = 6.6, 2.0 Hz, 1H), 2.55 (d, *J* = 13.0 Hz, 1H), 2.35 (d, *J* = 13.0 Hz, 1H), 1.71 (s, 3H), 1.34 (s, 3H), 1.32 (s, 9H), 0.98 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.02, 151.75, 145.71, 128.37, 124.32, 121.33, 120.95, 83.87, 50.93, 40.99, 34.99, 32.25, 31.50, 31.49, 31.48, 26.90, 26.10.

HRMS (ESI-TOF) Calcd for $C_{17}H_{25}O_2$ $[M+H]^+$: 261.1855; found: 261.1853.

IR (cm^{-1}) data: 2967, 1768, 1460, 1241, 1068, 951, 798, 709.



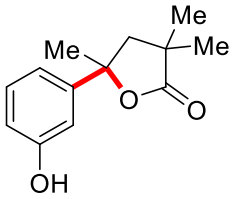
3,3,5-trimethyl-5-(m-tolyl)dihydrofuran-2(3H)-one (4s)

Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (17.5 mg, 80% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.57 (m, 4H), 7.47 – 7.42 (m, 4H), 7.37 – 7.33 (m, 1H), 2.60 (d, *J* = 13.0 Hz, 1H), 2.38 (d, *J* = 13.0 Hz, 1H), 1.75 (s, 3H), 1.36 (s, 3H), 1.03 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.91, 145.04, 140.52, 140.39, 128.95, 127.59, 127.43, 127.17, 124.62, 83.51, 50.82, 41.04, 32.19, 26.88, 26.18.

HRMS (ESI-TOF) Calcd for $C_{14}H_{19}O_2$ $[M+H]^+$: 219.1385; found: 219.1382.

IR (cm^{-1}) data: 2974, 1764, 1456, 1384, 1257, 1228, 1066, 951, 789, 705.



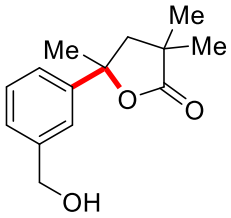
5-(3-hydroxyphenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4t)

Substrate **1a** was arylated and lactonized following the general procedure with little modification by using 20 mol% $Pd(OAc)_2$ and 20 mol% ligand L14 (eluent: hexane/ethyl acetate = 4/1). The product was obtained as a gummy liquid (13.6 mg, 62% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.22 (t, *J* = 7.9 Hz, 1H), 6.94 – 6.91 (m, 1H), 6.88 (ddd, *J* = 7.8, 1.7, 0.9 Hz, 1H), 6.76 (ddd, *J* = 8.1, 2.5, 0.9 Hz, 1H), 5.70 (s, 1H), 2.54 (d, *J* = 13.0 Hz, 1H), 2.32 (d, *J* = 13.0 Hz, 1H), 1.69 (s, 3H), 1.33 (s, 3H), 1.00 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.45, 156.22, 147.83, 130.11, 116.33, 114.48, 111.28, 83.76, 50.77, 41.11, 31.99, 26.80, 26.01.

HRMS (ESI-TOF) Calcd for $C_{13}H_{17}O_3$ $[M+H]^+$: 221.1178; found: 221.1182.

IR (cm^{-1}) data: 2974, 1764, 1456, 1384, 1257, 1228, 1066, 951, 789, 705.



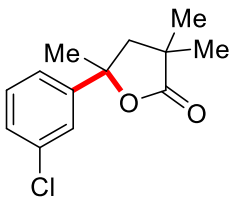
5-(3-(hydroxymethyl)phenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4u)

Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 4/1). The product was obtained as a gummy liquid (13.6 mg, 58% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.39 - 7.38 (m, 1H), 7.36 (t, *J* = 7.6 Hz, 1H), 7.32 – 7.27 (m, 2H), 4.71 (d, *J* = 5.0 Hz, 2H), 2.56 (d, *J* = 13.1 Hz, 1H), 2.36 (d, *J* = 13.0 Hz, 1H), 1.70 (s, 3H), 1.34 (s, 3H), 0.98 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.92, 146.46, 141.51, 129.01, 126.03, 123.39, 122.60, 83.59, 65.29, 50.78, 41.00, 32.23, 26.84, 26.15.

HRMS (ESI-TOF) Calcd for $C_{14}H_{19}O_3$ $[M+H]^+$: 235.1334; found: 235.1338.

IR (cm^{-1}) data: 2974, 1764, 1684, 1358, 1264, 1233, 1065, 952, 698.



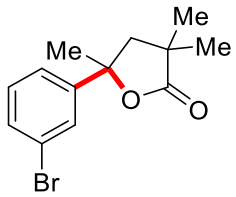
5-(3-chlorophenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4v)

Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (16.0 mg, 67% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.39 – 7.37 (m, 1H), 7.31 – 7.28 (m, 1H), 7.28 – 7.24 (m, 2H), 2.52 (d, *J* = 13.1 Hz, 1H), 2.35 (d, *J* = 13.1 Hz, 1H), 1.69 (s, 3H), 1.34 (s, 3H), 1.00 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.52, 148.18, 134.83, 130.13, 127.74, 124.55, 122.35, 82.90, 50.70, 40.91, 32.09, 26.77, 26.14.

HRMS (ESI-TOF) Calcd for $C_{13}H_{16}ClO_2$ $[M+H]^+$: 239.0839; found: 239.0837.

IR (cm^{-1}) data: 2975, 1765, 1469, 1244, 1064, 952, 789, 701.



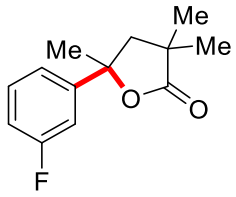
5-(3-bromophenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4w)

Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (24.4 mg, 86% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.54 (t, *J* = 1.8 Hz, 1H), 7.41 (ddd, *J* = 7.9, 1.9, 1.0 Hz, 1H), 7.31 (ddd, *J* = 7.9, 1.7, 1.0 Hz, 1H), 7.23 (t, *J* = 7.9 Hz, 1H), 2.51 (d, *J* = 13.1 Hz, 1H), 2.35 (d, *J* = 13.1 Hz, 1H), 1.69 (s, 3H), 1.34 (s, 3H), 1.00 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.51, 148.41, 130.70, 130.40, 127.42, 123.03, 122.82, 82.83, 50.69, 40.92, 32.13, 26.77, 26.16.

HRMS (ESI-TOF) Calcd for $C_{13}H_{16}BrO_2$ $[M+H]^+$: 283.0334; found: 283.0338.

IR (cm^{-1}) data: 2974, 1766, 1468, 1385, 1244, 1096, 1064, 952, 788, 698.



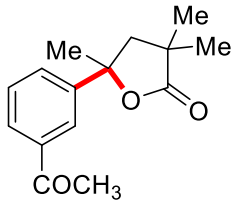
5-(3-fluorophenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4x)

Substrate **1a** was arylated and lactonized following the general procedure with little modification by using 20 mol% $Pd(OAc)_2$ and 20 mol% ligand L14 (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (15.6 mg, 70% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.33 (td, *J* = 8.0, 5.9 Hz, 1H), 7.15 (ddd, *J* = 7.8, 1.8, 0.9 Hz, 1H), 7.12 – 7.08 (m, 1H), 6.97 (tdd, *J* = 8.3, 2.6, 0.9 Hz, 1H), 2.52 (d, *J* = 13.1 Hz, 1H), 2.35 (d, *J* = 13.1 Hz, 1H), 1.69 (s, 3H), 1.34 (s, 3H), 0.99 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.57, 163.04 (d, *J* = 246.9 Hz), 148.76 (d, *J* = 6.8 Hz), 130.45 (d, *J* = 8.2 Hz), 119.78 (d, *J* = 3.0 Hz), 114.44 (d, *J* = 21.1 Hz), 111.58 (d, *J* = 23.1 Hz), 82.96 (d, *J* = 2.0 Hz), 50.76, 40.93, 32.01, 26.78, 26.08.

^{19}F NMR (376 MHz, $CDCl_3$) δ -111.8.
HRMS (ESI-TOF) Calcd for $C_{13}H_{16}FO_2$ $[M+H]^+$: 223.1134; found: 223.1136.

IR (cm^{-1}) data: 2976, 1765, 1589, 1441, 1255, 1222, 1068, 952, 881, 789.



5-(3-acetylphenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4y)

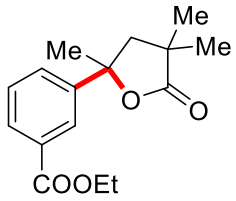
Substrate **1a** was arylated and lactonized following the general procedure with little modification by using 20 mol% $Pd(OAc)_2$ and 20 mol% ligand L14 (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a white solid (14.8 mg, 60% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.95 (t, *J* = 1.7 Hz, 1H), 7.88 – 7.85 (m, 1H), 7.64 (ddd, *J* = 7.8, 2.0, 1.1 Hz, 1H), 7.47 (t, *J* = 7.8 Hz, 1H), 2.62 (s, 3H), 2.58 (d, *J* = 13.1 Hz, 1H), 2.40 (d, *J* = 13.1 Hz, 1H), 1.72 (s, 3H), 1.35 (s, 3H), 0.97 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 197.99, 181.58, 146.86, 137.59, 129.19, 128.82, 127.71, 123.80, 83.21, 50.69, 40.94, 32.25, 26.89, 26.77, 26.16.

HRMS (ESI-TOF) Calcd for $C_{15}H_{19}O_3$ $[M+H]^+$: 247.1334; found: 247.1329.

Melting point: 74 °C to 76 °C

IR (cm^{-1}) data: 2974, 1764, 1684, 1358, 1264, 1233, 1065, 952, 698.



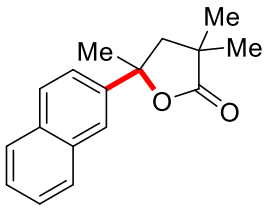
ethyl 3-(2,4,4-trimethyl-5-oxotetrahydrofuran-2-yl)benzoate (4z)

Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (14.1 mg, 51% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.01 (t, *J* = 1.7 Hz, 1H), 7.96 (dt, *J* = 7.7, 1.4 Hz, 1H), 7.63 (ddd, *J* = 7.8, 2.0, 1.2 Hz, 1H), 7.44 (t, *J* = 7.8 Hz, 1H), 4.39 (qd, *J* = 7.1, 1.5 Hz, 2H), 2.58 (d, *J* = 13.1 Hz, 1H), 2.39 (d, *J* = 13.1 Hz, 1H), 1.72 (s, 3H), 1.40 (t, *J* = 7.1 Hz, 3H), 1.35 (s, 3H), 0.97 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 181.61, 166.43, 146.53, 131.11, 128.92, 128.73, 128.50, 125.32, 83.22, 61.36, 50.69, 40.94, 32.24, 26.76, 26.13, 14.48.

HRMS (ESI-TOF) Calcd for $C_{16}H_{21}O_4$ $[M+H]^+$: 277.1440; found: 277.1442.

IR (cm^{-1}) data: 2976, 1767, 1716, 1451, 1268, 1235, 1066, 953, 757, 700.



3,3,5-trimethyl-5-(naphthalen-2-yl)dihydrofuran-2(3H)-one (4aa)

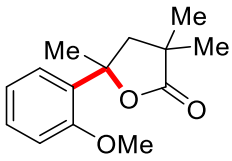
Substrate **1a** was arylated and lactonized following the general procedure (eluent: hexane/ethyl acetate = 5/1). The product was obtained as an off white solid (20.8 mg, 82% yield). R_f = 0.3; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.92 – 7.89 (m, 1H), 7.87 – 7.82 (m, 3H), 7.50 (m, 2H), 7.43 (dd, *J* = 8.6, 1.9 Hz, 1H), 2.67 (d, *J* = 13.1 Hz, 1H), 2.42 (d, *J* = 13.1 Hz, 1H), 1.79 (s, 3H), 1.37 (s, 3H), 0.98 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.01, 143.24, 133.18, 132.59, 128.76, 128.34, 127.69, 126.68, 126.35, 122.60, 122.56, 83.68, 50.69, 41.05, 32.11, 26.88, 26.16.

HRMS (ESI-TOF) Calcd for $C_{17}H_{19}O_2$ $[M+H]^+$: 255.1385; found: 255.1384.

Melting point: 108 ºC to 112 ºC

IR (cm^{-1}) data: 2974, 1764, 1454, 1380, 1233, 1070, 952, 821, 752.



5-(2-methoxyphenyl)-3,3,5-trimethyldihydrofuran-2(3H)-one (4ab)

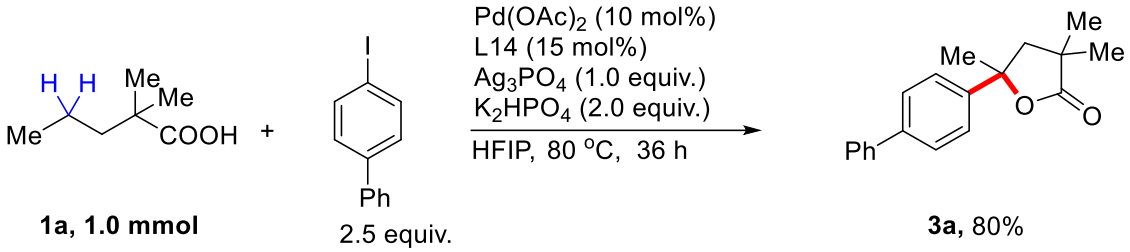
Substrate **1a** was arylated and lactonized following the general procedure with little modification by using 30 mol% $Pd(OAc)_2$ and 30 mol% ligand L14 (eluent: hexane/ethyl acetate = 5/1). The product was obtained as a gummy liquid (11.2 mg, 48% yield). R_f = 0.2; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.51 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.29 – 7.25 (m, 1H), 6.95 (td, *J* = 7.6, 1.1 Hz, 1H), 6.91 (dd, *J* = 8.2, 1.0 Hz, 1H), 3.86 (s, 3H), 2.65 (d, *J* = 13.5 Hz, 1H), 2.33 (d, *J* = 13.5 Hz, 1H), 1.75 (s, 3H), 1.33 (s, 3H), 1.01 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 182.43, 155.19, 133.70, 128.90, 125.21, 120.87, 111.34, 83.21, 55.28, 49.37, 40.56, 28.85, 27.19, 26.44.

HRMS (ESI-TOF) Calcd for $C_{14}H_{19}O_3$ $[M+H]^+$: 235.1334; found: 235.1329.

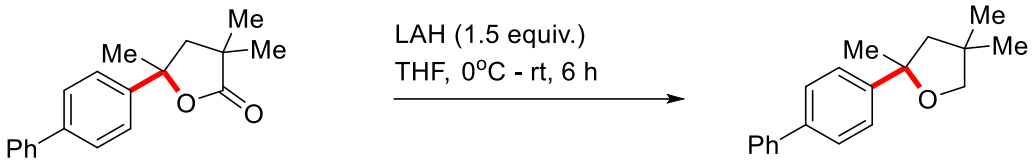
IR (cm^{-1}) data: 2972, 1764, 1489, 1463, 1242, 1058, 1025, 951, 755.

6. 1.0 mmol Scale Reaction and Further Transformation to Substituted Tetrahydrofuran Derivatives:



1a, 1.0 mmol 2.5 equiv. **3a,** 80%

$Pd(OAc)_2$ (22.5 mg, 10 mol%), Ligand L14 (27.2 mg, 15 mol%), Ag_3PO_4 (41.9 mg, 1.0 equiv.), K_2HPO_4 (348 mg, 2.0 equiv.), 4-iodobiphenyl (700 mg, 2.5 equiv.), and HFIP (10.0 ml) were added to a pressure tube (30 ml) and stir at rt for 5 minutes. Then Carboxylic acid **1a** (130.0 mg, 1.0 mmol) was added. Next the pressure tube was capped under air and closed tightly. Then the reaction mixture was stirred at 80 °C for 36 hours. After cooling to room temperature,) and filtered through a pad of celite with ethyl acetate as the eluent to remove the insoluble precipitate. The resulting solution was concentrated and purified by flash column chromatography (SiO_2, 5:1 hexane: EA) to afford the desired product **3a** (224.3 mg, 80%) as off white solid.



3a **5a**, 76%

The lactone **3a** (140.1 mg, 0.5 mmol) was dissolved in THF and cooled to 0 °C, and 2.0 (M) Lithium aluminum hydride solution in THF (375 µl, 1.5 equiv.) was added dropwise to it. Then the reaction mixture stirred for 6 hours at room temperature. After stirring for 6 h, the reaction mixture was carefully quenched with H_2O (0.2 mL), 10% NaOH (0.2 mL), and H_2O (0.5 mL) at 0 °C and filtered through a pad of Celite. The filtrate was extracted with EtOAc, washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography (SiO_2, 10:1 hexane: EtOAc) to give substituted tetrahydrofuran derivative **5a** (101.2 mg, 76%) as an off white solid.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.62 – 7.59 (m, 2H), 7.58 – 7.55 (m, 2H), 7.53 – 7.50 (m, 2H), 7.45 – 7.41 (m, 2H), 7.35 – 7.32 (m, 1H), 3.44 (d, *J* = 10.9 Hz, 1H), 3.35 (d, *J* = 10.9 Hz, 1H), 2.02 (d, *J* = 15.1 Hz, 1H), 1.96 (d, *J* = 15.1 Hz, 1H), 1.62 (s, 3H), 0.93 (s, 3H), 0.47 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 147.75, 140.89, 139.28, 128.89, 127.32, 127.13, 126.83, 125.42, 75.16, 71.47, 53.30, 36.77, 34.21, 28.87, 25.82.

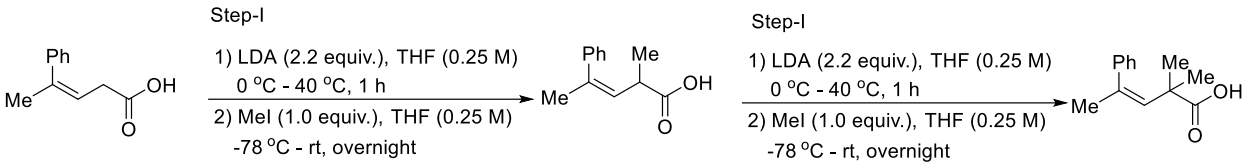
HRMS (ESI-TOF) Calcd for $C_{19}H_{13}O$ $[M+H]^+$: 267.1473; found: 267.1477.

Melting point: 41 °C to 44 °C

IR (cm^{-1}) data: 2962, 1486, 1077, 1043, 841, 755, 734, 697.

7. Control Experiments for Mechanistic Investigation:

Synthesis of 6d:



To a stirring solution of diisopropylamine (11 mmol, 1.55 mL) in THF (20 mL) was added 2.5 M n-BuLi solution (11 mmol, 4.4 mL) dropwise at -78 °C. The resulting solution was stirred for 30 min at 0 °C. (Z)-4-phenylpent-3-enoic acid (5 mmol, 881 mg) in 2mL was added dropwise at the same temperature, and the reaction mixture was stirred for 1 h at 40 °C. After cooling to -78 °C, methyl iodide (5 mmol, 332 µL) was added dropwise. The reaction mixture was slowly warmed to room temperature overnight. The reaction mixture was quenched with water and diluted with EtOAc. The layers were separated, and the organic layer was extracted with water. The aqueous layer was combined and acidified to pH < 4 with 3 M HCl (aq.). Then, the aqueous layer was extracted with EtOAc. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuo. The crude carboxylic acid was directly used in the next step.

To a stirring solution of diisopropylamine (11 mmol, 1.55 mL) in THF (40 mL) was added 2.5 M n-BuLi solution (11 mmol, 4.4 mL) dropwise at -78 °C. The resulting solution was stirred for 30 min at 0 °C. crude acid of step-I was added dropwise at the same temperature, and the reaction mixture was stirred for 1 h at 40 °C. After cooling to -78 °C, corresponding methyl iodide (5 mmol, 332 µL) was added dropwise. The reaction mixture was slowly warmed to room temperature overnight. The reaction mixture was quenched with water and diluted with EtOAc. The layers were separated, and the organic layer was extracted with water. The aqueous layer was combined and acidified to pH < 4 with 3 M HCl (aq.). Then, the aqueous layer was extracted with EtOAc. The combined organic layers were dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo. The crude carboxylic acid was purified by flash chromatography to get the pure carboxylic acid 632 mg (62%) as white solid.

^{1}H NMR (600 MHz, $CDCl_3$) δ 11.54 (b s, 1H), 7.38 (dd, *J* = 8.2, 1.1 Hz, 2H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.27 – 7.23 (m, 1H), 5.77-5.75 (m, 1H), 2.04 (d, *J* = 1.2 Hz, 3H), 1.47 (s, 6H).

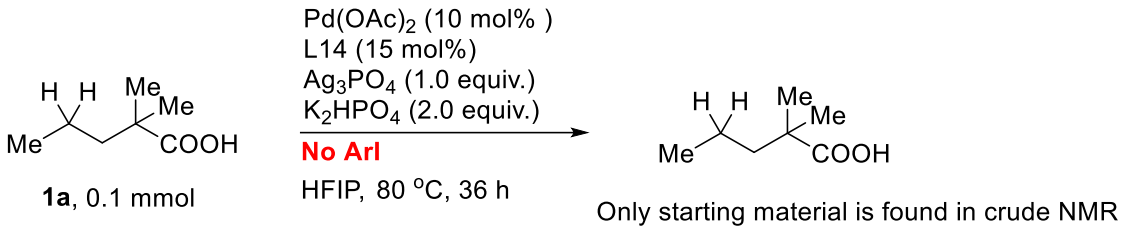
^{13}C NMR (151 MHz, $CDCl_3$) δ 184.02, 144.06, 137.64, 132.79, 128.32, 127.20, 126.04, 43.13, 27.21, 17.03, 17.02.

HRMS (ESI-TOF) Calcd for $C_{13}H_{17}O_2$ $[M+H]^+$: 205.1229; found: 205.1227.

Melting point: 119 °C to 125 °C

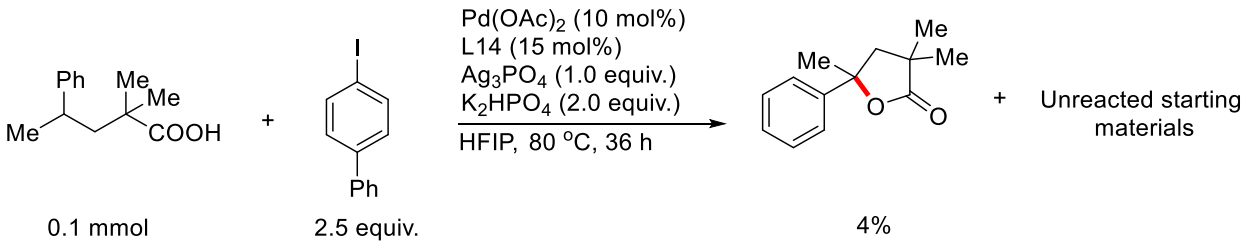
IR (cm^{-1}) data: 2989, 1693, 1285,1170, 933.

7.a) Controlled reaction with 2,2-dimethylpentanoic acid in absence of aryl iodide:



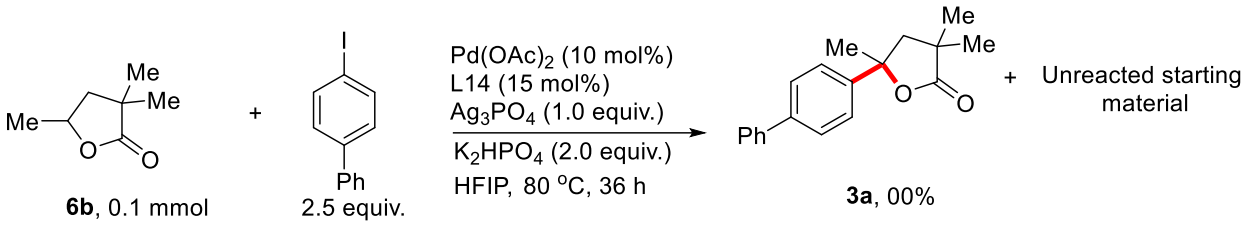
In a sealed tube equipped with a magnetic stir bar was charged with $Pd(OAc)_2$ (2.2 mg, 10 mol%), L14 (2.7 mg, 15 mol%), Ag_3PO_4 (41.9 mg, 1 equiv.), K_2HPO_4 (34.8 mg, 2 equiv.), and HFIP (1.0 mL) were then added and stir for 5 minutes. Then 2,2-dimethylpentanoic acid **1a** (0.1 mmol) was added. The reaction mixture was then stirred at the rate of 600 rpm at 80 °C for 36 h. After being allowed to cool to room temperature, the mixture was diluted with ethyl acetate and acidified with 0.1 mL of HCOOH. The mixture was passed through a pad of Celite with acetone as the eluent to remove any insoluble precipitate. The resulting solutions were concentrated, and crude NMR taken, and results were shown in the scheme.

7.b) Controlled reaction with 2,2-dimethyl-4-phenylpentanoic acid (6a) substrate:



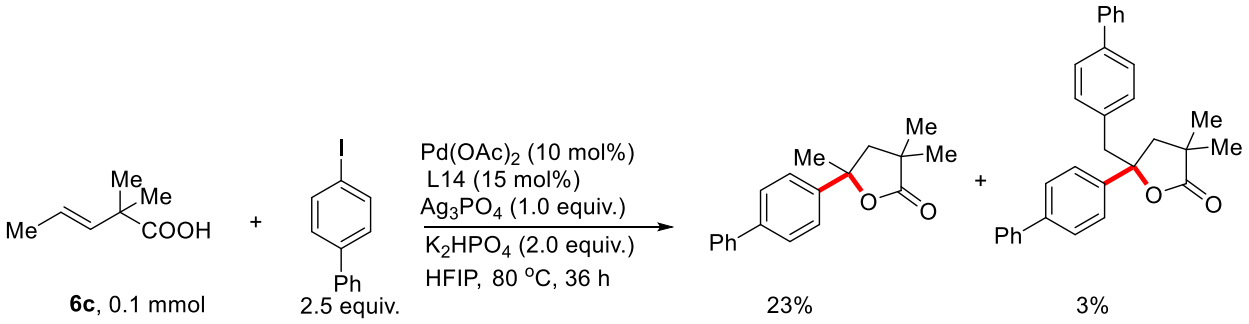
$Pd(OAc)_2$ (10 mol%), Ligand L14 (15 mol%), Ag_3PO_4 (1.0 equiv.), K_2HPO_4 (2.0 equiv.), 4-Iodo biphenyl (2.5 equiv.), and HFIP (1.0 ml) were added to a reaction vial (8 ml) and stir at rt for 5 minutes. Then 2,2-dimethyl-4-phenylpentanoic acid (**5a**) (0.1 mmol) was added. Next the vial was capped under air and closed tightly. Then the reaction mixture was stirred at 80 °C for 36 hours. After cooling to room temperature, the mixture was acidified with HCOOH (50 µL) and filtered through a pad of celite with ethyl acetate as the eluent to remove the insoluble precipitate. The resulting solution was concentrated, and the yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as the internal standard. Crude NMR analysis shows only 4% NMR yield of the desired γ-arylative γ-lactonization product.

7.c) Controlled reaction with 3,3,5-trimethyldihydrofuran-2(3H)-one (6b) substrate:



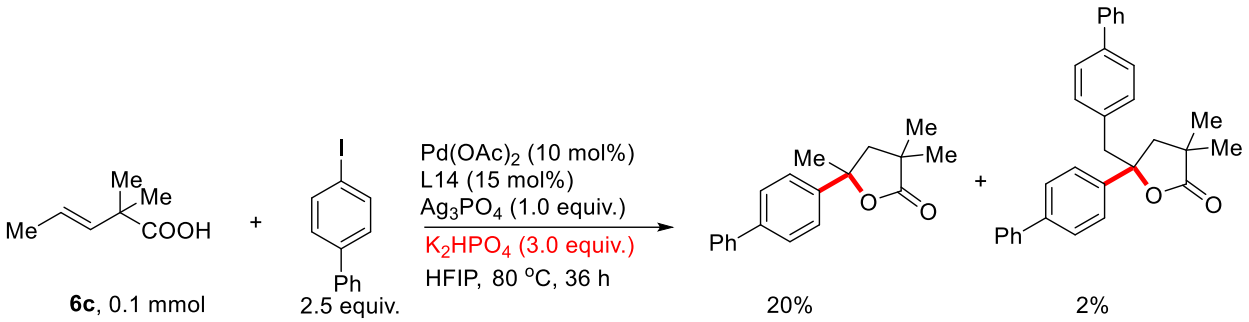
$Pd(OAc)_2$ (10 mol%), Ligand L14 (15 mol%), Ag_3PO_4 (1.0 equiv.), K_2HPO_4 (2.0 equiv.), 4-Iodo biphenyl (2.5 equiv.), and HFIP (1.0 ml) were added to a reaction vial (8 ml) and stir at rt for 5 minutes. Then 3,3,5-trimethyldihydrofuran-2(3H)-one (**5b**) (0.1 mmol) was added. Next the vial was capped under air and closed tightly. Then the reaction mixture was stirred at 80 °C for 36 hours. After cooling to room temperature, the mixture was acidified with HCOOH (50 µL) and filtered through a pad of celite with ethyl acetate as the eluent to remove the insoluble precipitate. The resulting solution was concentrated, and the yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as the internal standard. Crude NMR analysis shows only unreacted starting material, no product formation.

7.d) Controlled reaction with (E)-2,2-dimethylpent-3-enoic acid (6b) substrate:



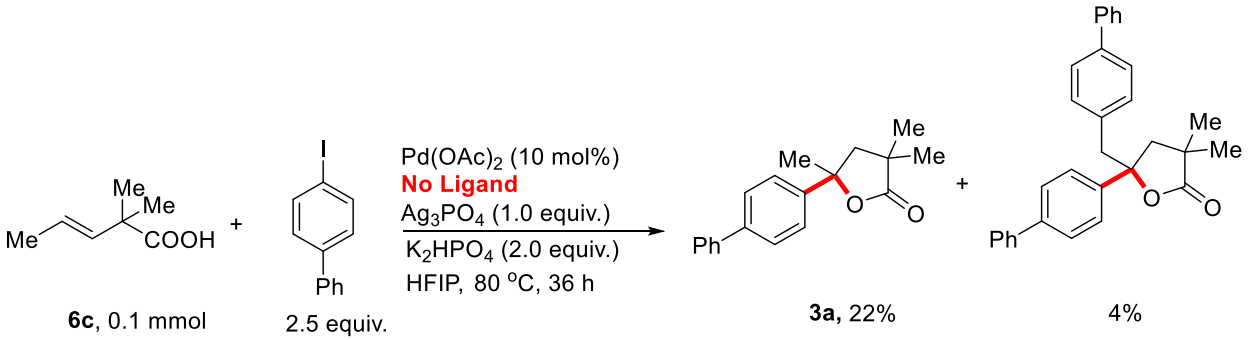
$Pd(OAc)_2$ (10 mol%), Ligand L14 (15 mol%), Ag_3PO_4 (1.0 equiv.), K_2HPO_4 (2.0 equiv.), 4-Iodo biphenyl (2.5 equiv.), and HFIP (1.0 ml) were added to a reaction vial (8 ml) and stir at rt for 5 minutes. Then (E)-2,2-dimethylpent-3-enoic acid (**6c**) (0.1 mmol) was added. Next the vial was capped under air and closed tightly. Then the reaction mixture was stirred at 80 °C for 36 hours. After cooling to room temperature, the mixture was acidified with HCOOH (50 µL) and filtered through a pad of celite with ethyl acetate as the eluent to remove the insoluble precipitate. The resulting solution was concentrated, and the yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as the internal standard. Crude NMR analysis shows only 23% NMR yield of the desired γ-arylative γ-lactonization product and 3% di isomer.

7.e) Controlled reaction with (E)-2,2-dimethylpent-3-enoic acid (6b) substrate with 3.0 equiv. base:



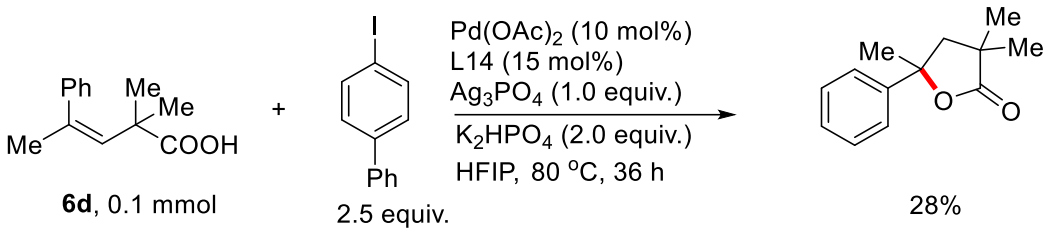
$Pd(OAc)_2$ (10 mol%), Ligand L14 (15 mol%), Ag_3PO_4 (1.0 equiv.), K_2HPO_4 (3.0 equiv.), 4-Iodo biphenyl (2.5 equiv.), and HFIP (1.0 ml) were added to a reaction vial (8 ml) and stir at rt for 5 minutes. Then (E)-2,2-dimethylpent-3-enoic acid (**6c**) (0.1 mmol) was added. Next the vial was capped under air and closed tightly. Then the reaction mixture was stirred at 80 °C for 36 hours. After cooling to room temperature, the mixture was acidified with HCOOH (50 µL) and filtered through a pad of celite with ethyl acetate as the eluent to remove the insoluble precipitate. The resulting solution was concentrated, and the yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as the internal standard. Crude NMR analysis shows only 20% NMR yield of the desired γ-arylative γ-lactonization product and 2% di isomer.

7.f) Controlled reaction with (E)-2,2-dimethylpent-3-enoic acid (6c) substrate:



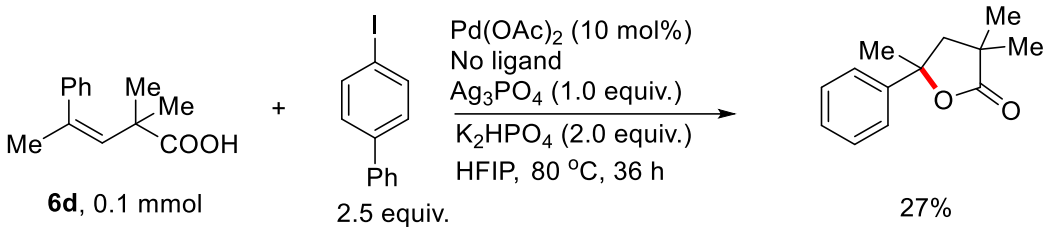
$Pd(OAc)_2$ (10 mol%), Ag_3PO_4 (1.0 equiv.), K_2HPO_4 (2.0 equiv.), 4-Iodo biphenyl (2.5 equiv.), and HFIP (1.0 ml) were added to a reaction vial (8 ml) and stir at rt for 5 minutes. Then (E)-2,2-dimethylpent-3-enoic acid (**6c**) (0.1 mmol) was added. Next the vial was capped under air and closed tightly. Then the reaction mixture was stirred at 80 °C for 36 hours. After cooling to room temperature, the mixture was acidified with HCOOH (50 µL) and filtered through a pad of celite with ethyl acetate as the eluent to remove the insoluble precipitate. The resulting solution was concentrated, and the yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as the internal standard. Crude NMR analysis shows 22% γ-arylative γ-lactonization product and 4% di isomer.

7.g) Controlled reaction with (Z)-2,2-dimethyl-4-phenylpent-3-enoic acid (6d) substrate:



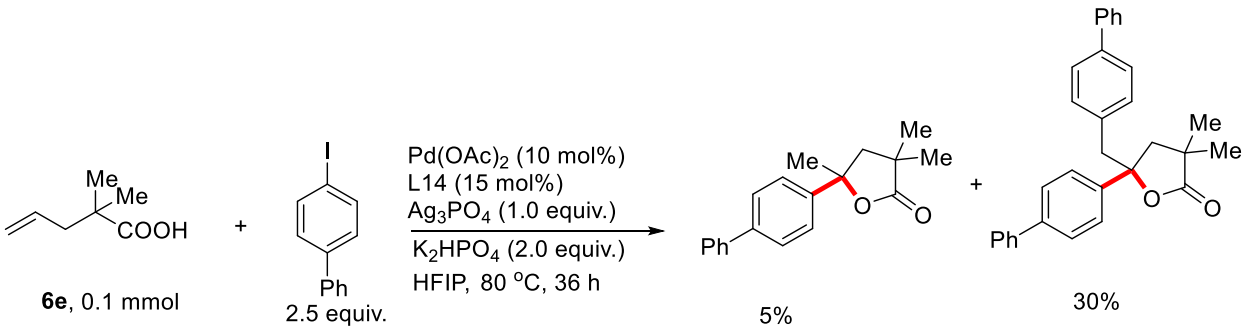
$Pd(OAc)_2$ (10 mol%), Ligand L14 (15 mol%), Ag_3PO_4 (1.0 equiv.), K_2HPO_4 (2.0 equiv.), 4-Iodo biphenyl (2.5 equiv.), and HFIP (1.0 ml) were added to a reaction vial (8 ml) and stir at rt for 5 minutes. Then (Z)-2,2-dimethyl-4-phenylpent-3-enoic acid (**6d**) (0.1 mmol) was added. Next the vial was capped under air and closed tightly. Then the reaction mixture was stirred at 80 °C for 36 hours. After cooling to room temperature, the mixture was acidified with HCOOH (50 µL) and filtered through a pad of celite with ethyl acetate as the eluent to remove the insoluble precipitate. The resulting solution was concentrated, and the yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as the internal standard. Crude NMR analysis shows 28% yield of the desired γ-arylative γ-lactonization product.

7.h) Controlled reaction with (Z)-2,2-dimethyl-4-phenylpent-3-enoic acid (6d) substrate:



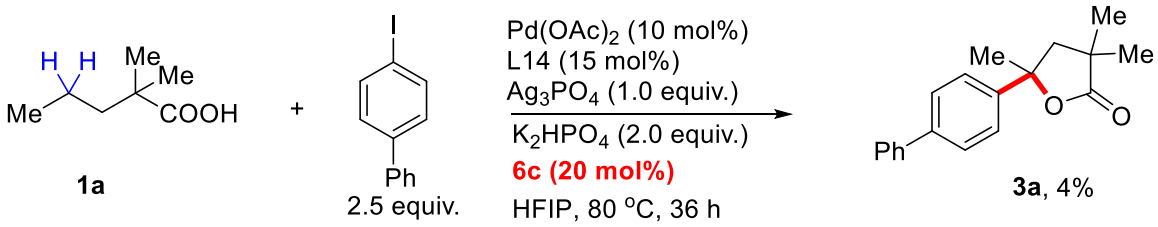
$Pd(OAc)_2$ (10 mol%), Ag_3PO_4 (1.0 equiv.), K_2HPO_4 (2.0 equiv.), and HFIP (1.0 ml) were added to a reaction vial (8 ml) and stir at rt for 5 minutes. Then (Z)-2,2-dimethyl-4-phenylpent-3-enoic acid (**6d**) (0.1 mmol) was added. Next the vial was capped under air and closed tightly. Then the reaction mixture was stirred at 80 °C for 36 hours. After cooling to room temperature, the mixture was acidified with HCOOH (50 µL) and filtered through a pad of celite with ethyl acetate as the eluent to remove the insoluble precipitate. The resulting solution was concentrated, and the yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as the internal standard. Crude NMR analysis shows 27% yield of the desired γ-arylative γ-lactonization product.

7.i) Controlled reaction with 2,2-dimethylpent-4-enoic acid (6e) substrate:



$Pd(OAc)_2$ (10 mol%), Ligand L14 (15 mol%), Ag_3PO_4 (1.0 equiv.), K_2HPO_4 (2.0 equiv.), 4-Iodo biphenyl (2.5 equiv.), and HFIP (1.0 ml) were added to a reaction vial (8 ml) and stir at rt for 5 minutes. Then 2,2-dimethylpent-4-enoic acid (6e) (0.1 mmol) was added. Next the vial was capped under air and closed tightly. Then the reaction mixture was stirred at 80 °C for 36 hours. After cooling to room temperature, the mixture was acidified with HCOOH (50 µL) and filtered through a pad of celite with ethyl acetate as the eluent to remove the insoluble precipitate. The resulting solution was concentrated, and the yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as the internal standard. Crude NMR analysis shows only 5% γ-arylative γ-lactonization product 30% yield of the di product.

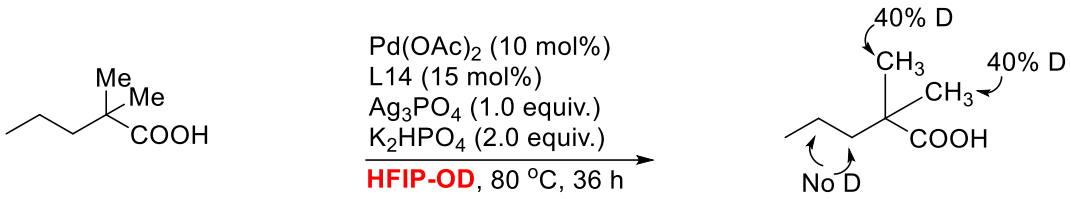
7.j) Controlled reaction with 6c as additive:



$Pd(OAc)_2$ (10 mol%), Ligand L14 (15 mol%), Ag_3PO_4 (1.0 equiv.), K_2HPO_4 (2.0 equiv.), 4-Iodo biphenyl (2.5 equiv.), and HFIP (1.0 ml) were added to a reaction vial (8 ml) and stir at rt for 5 minutes. Then 2,2-dimethylpentanoic acid (**1a**) (0.1 mmol) and **6c** (20 mol%) were added. Next the vial was capped under air and closed tightly. Then the reaction mixture was stirred at 80 °C for 36 hours. After cooling to room temperature, the mixture was acidified with HCOOH (50 µL) and filtered through a pad of celite with ethyl acetate as the eluent to remove the insoluble precipitate. The resulting solution was concentrated, and the yield was determined by ^1H NMR analysis of the crude product using CH_2Br_2 as the internal standard and results are shown in the scheme.

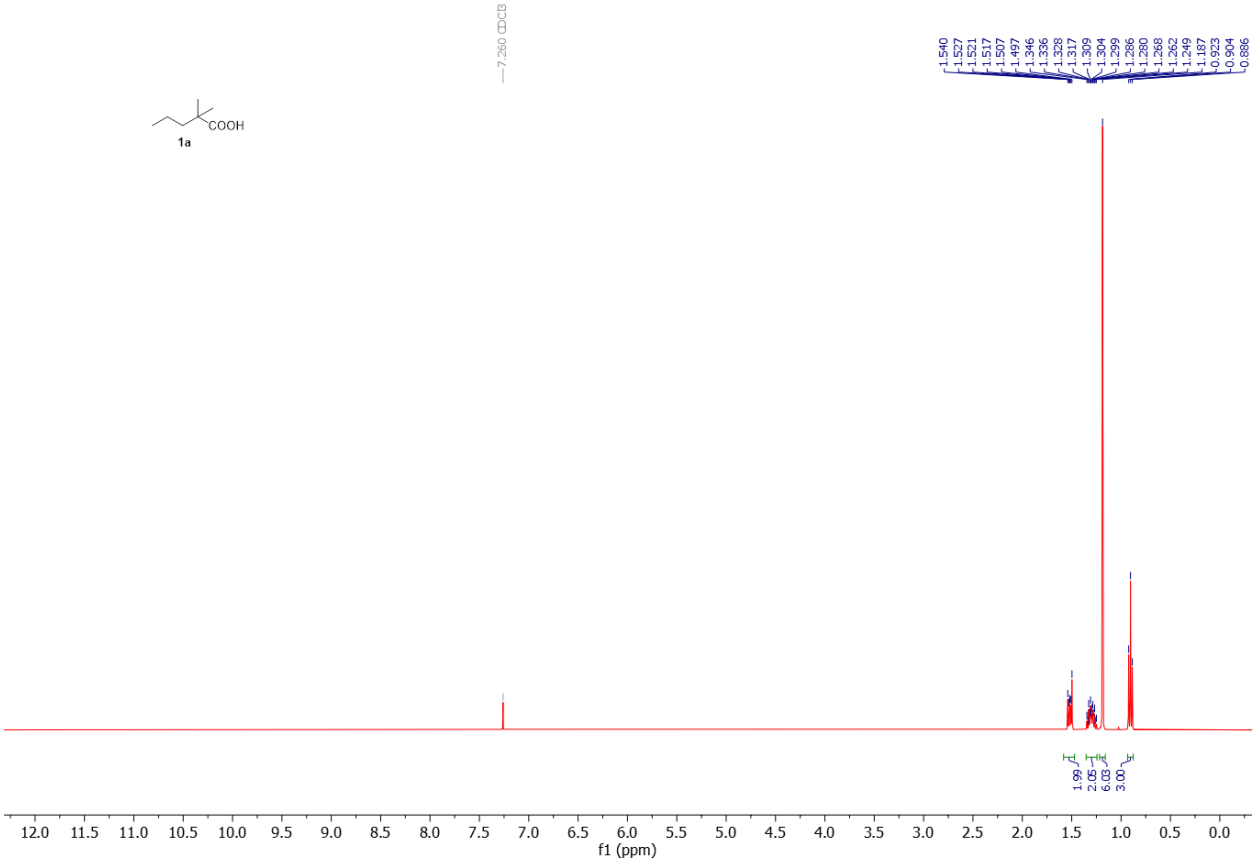
8. H/D exchange experiments:

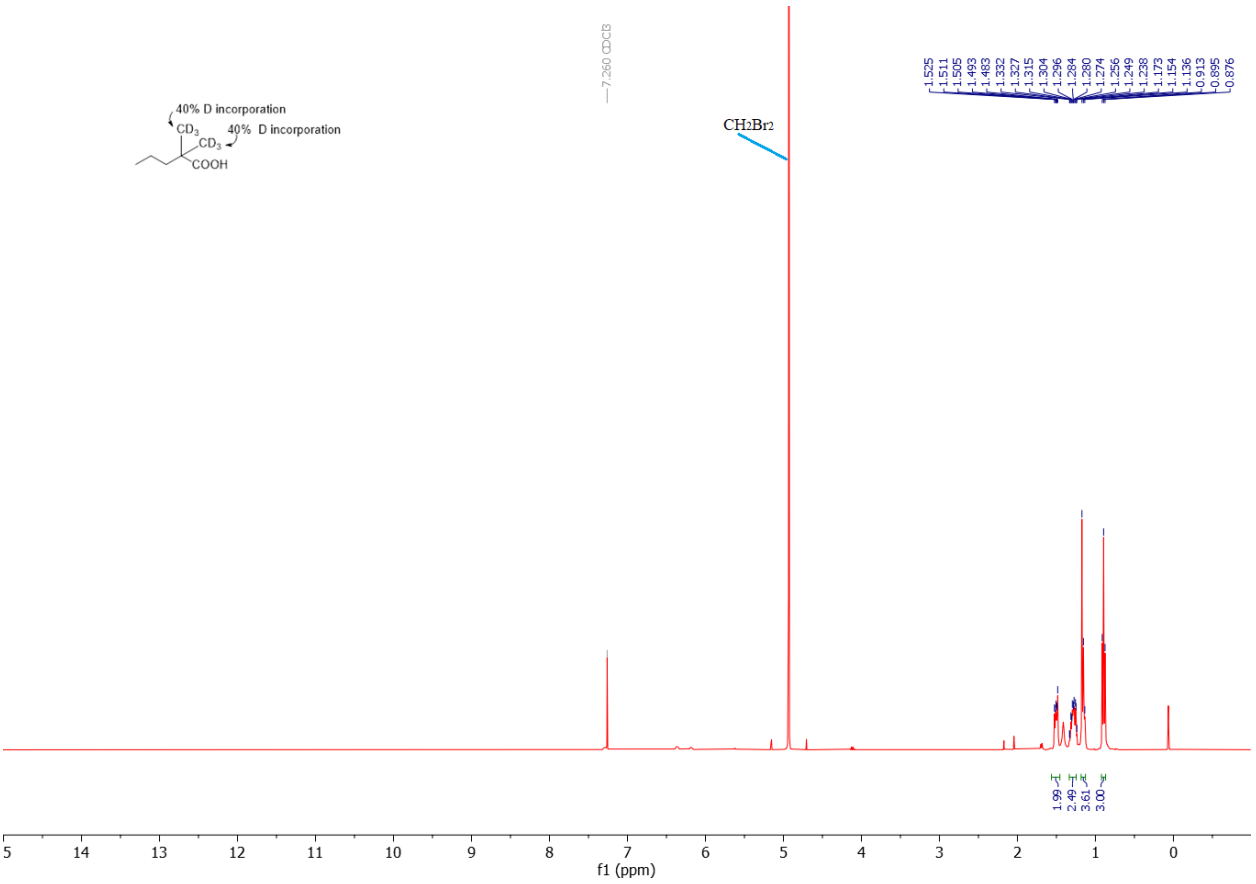
8. a. H/D exchange experiments of 2,2-dimethylpentanoic acid in absence of aryl iodide:



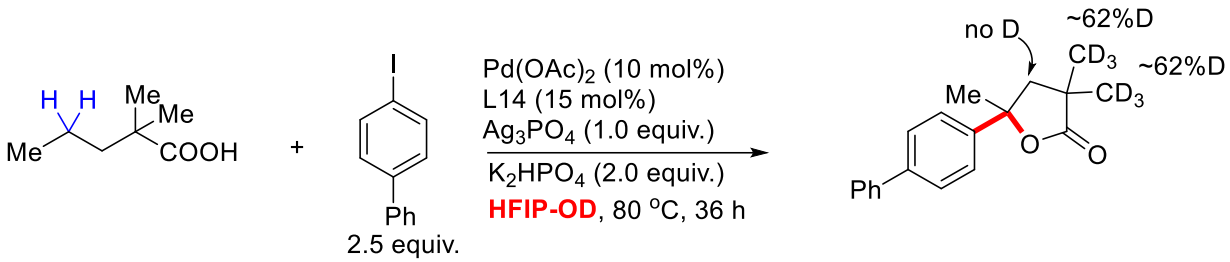
0.1 mmol

In a sealed tube equipped with a magnetic stir bar was charged with $Pd(OAc)_2$ (2.2 mg, 10 mol%), L14 (2.7 mg, 15 mol%), Ag_3PO_4 (41.9 mg, 1 equiv.), K_2HPO_4 (34.8 mg, 2 equiv.), and HFIP-OD (1.0 mL) were then added and stirred for 5 minutes. Then 2,2-dimethylpentanoic acid **1a** (0.1 mmol) was added. The reaction mixture was then stirred at the rate of 600 rpm at 80 °C for 36 h. After being allowed to cool to room temperature, the mixture was diluted with ethyl acetate and acidified with 0.1 mL of HCOOH. The mixture was passed through a pad of Celite with acetone as the eluent to remove any insoluble precipitate. The resulting solutions were concentrated, and crude NMR taken to determine the deuterium incorporation.

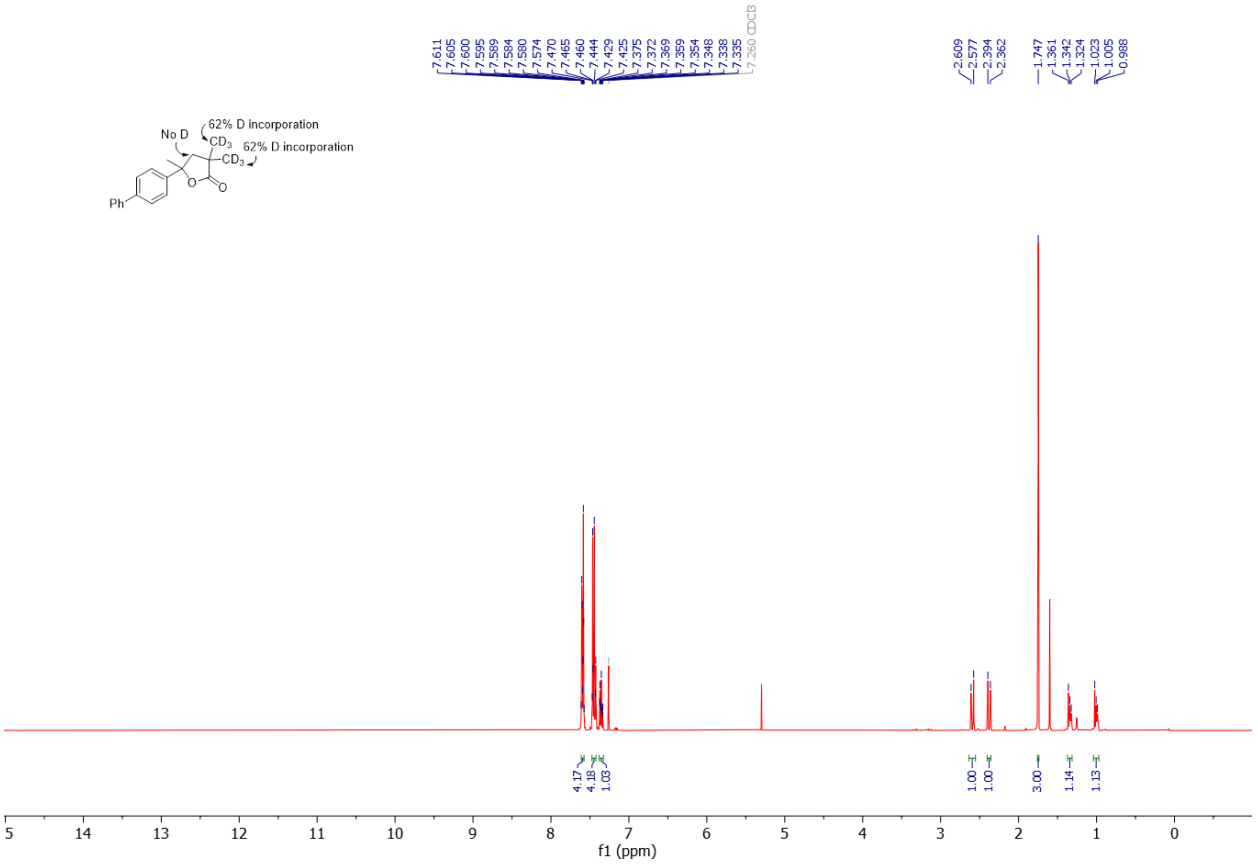




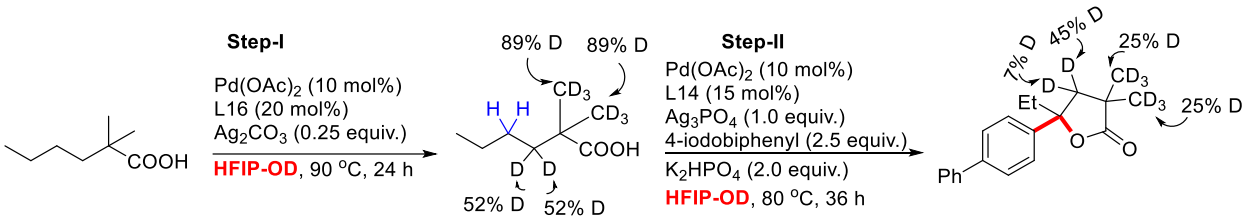
8. b. H/D exchange experiments of 2,2-dimethylpentanoic acid in absence of aryl iodide:



In a sealed tube equipped with a magnetic stir bar was charged with $Pd(OAc)_2$ (2.2 mg, 10 mol%), L14 (2.7 mg, 15 mol%), Ag_3PO_4 (41.9 mg, 1 equiv.), K_2HPO_4 (34.8 mg, 2 equiv.), and HFIP-OD (1.0 mL) were then added and stir for 5 minutes. Then 2,2-dimethylpentanoic acid **1a** (0.1 mmol) was added. The reaction mixture was then stirred at the rate of 600 rpm at 80 °C for 36 h. After being allowed to cool to room temperature, the mixture was diluted with ethyl acetate and acidified with 0.1 mL of HCOOH. The mixture was passed through a pad of Celite with acetone as the eluent to remove any insoluble precipitate. The resulting solutions was concentrated, and the residual mixture was isolated using pTLC (hexane/ethyl acetate = 6/1). The ^{1}H-NMR taken to determine the deuterium incorporation.



8. c. H/D exchange experiments with β-methylene deuterated substrate:

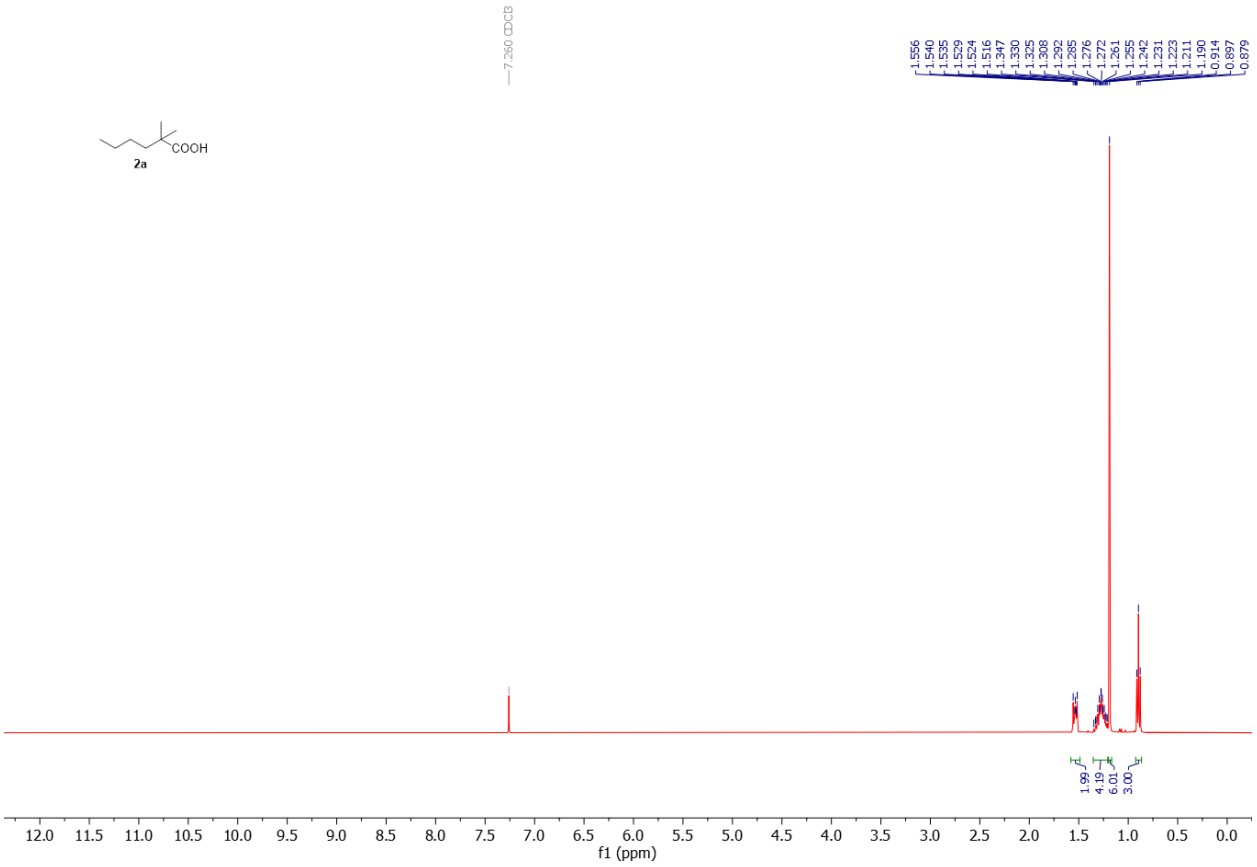


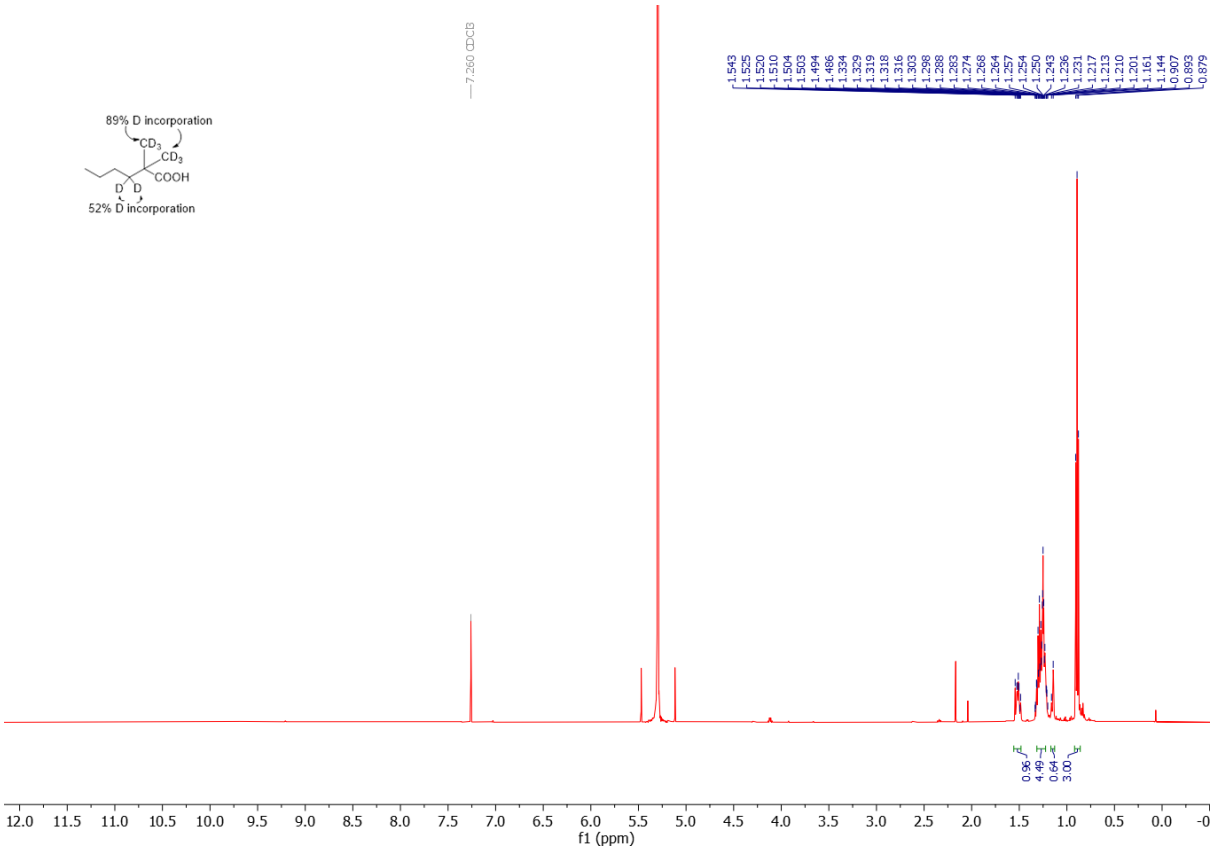
Step-I:

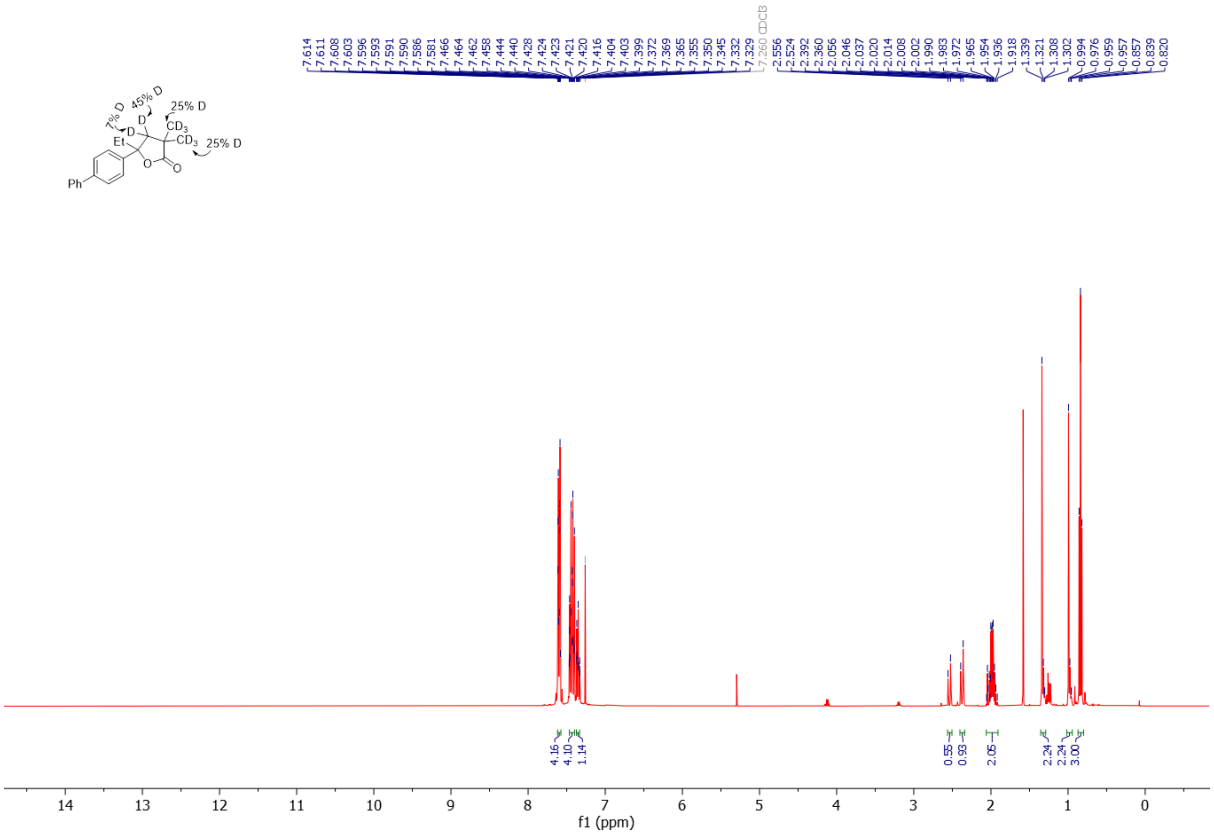
In a sealed tube equipped with a magnetic stir bar was charged with $Pd(OAc)_2$ (2.2 mg, 10 mol%), *N*-(2-(dimethylamino)ethyl)-2,4,6-triisopropylbenzamide ligand (L16) (6.4 mg, 20 mol%), Ag_2CO_3 (6.9 mg, 0.25 equiv.), and HFIP-OD (2.0 mL) were then added and stir for 5 minutes. Then 2,2-dimethylhexanoic acid **1b** (0.1 mmol) was added. The reaction mixture was then stirred at the rate of 600 rpm at 90 °C for 24 h. After being allowed to cool to room temperature, the mixture was diluted with ethyl acetate and acidified with 0.1 mL of HCOOH. The mixture was passed through a pad of Celite with acetone as the eluent to remove any insoluble precipitate. The resulting solution was concentrated, and crude NMR taken to determine the deuterium incorporation and found 89% deuterium -incorporation at the β-methyl position and 52% deuterium-incorporation at the β-methylene position. Then this crude product was used for the next step.

Step-II:

In a sealed tube equipped with a magnetic stir bar was charged with $Pd(OAc)_2$ (2.2 mg, 10 mol%), L14 (2.7 mg, 15 mol%), Ag_3PO_4 (41.9 mg, 1 equiv.), K_2HPO_4 (34.8 mg, 2 equiv.), 4-iodobiphenyl (70.0 mg, 2.5 equiv.), and HFIP-OD (1.0 mL) were then added and stir for 5 minutes. Then carboxylic acid substrate (0.1 mmol) was added. The reaction mixture was then stirred at the rate of 600 rpm at 80 °C for 36 h. After being allowed to cool to room temperature, the mixture was diluted with ethyl acetate and acidified with 0.1 mL of HCOOH. The mixture was passed through a pad of Celite with acetone as the eluent to remove any insoluble precipitate. The resulting solutions were concentrated, and the residual mixture was isolated using pTLC (hexane/ethyl acetate = 6/1).

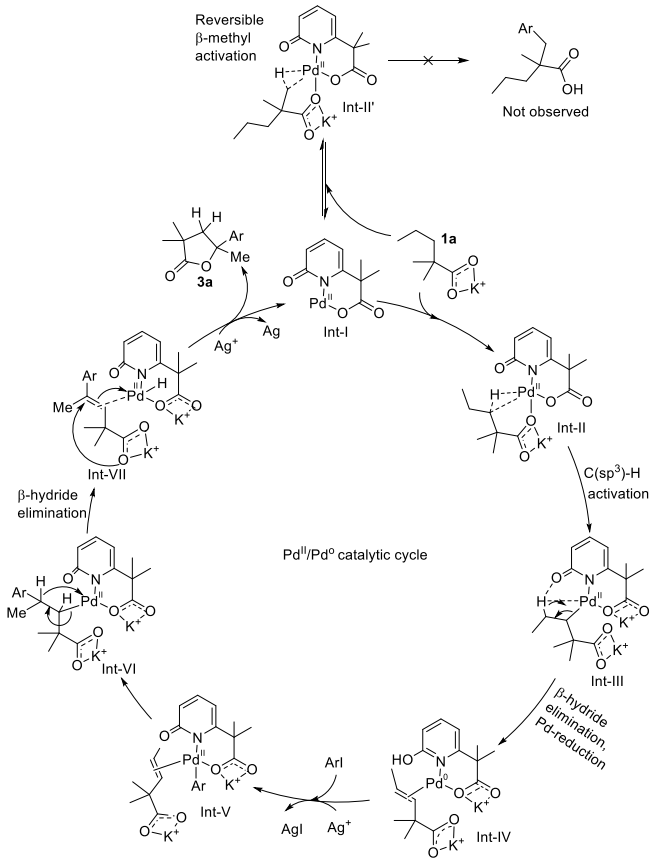



89% D incorporation
CD3
CD3
COOH
D D
52% D incorporation
7.260 CDCl3
1.543
1.525
1.520
1.510
1.504
1.503
1.494
1.486
1.334
1.329
1.319
1.318
1.316
1.303
1.298
1.288
1.283
1.274
1.268
1.264
1.257
1.254
1.250
1.243
1.236
1.231
1.217
1.213
1.210
1.201
1.161
1.144
0.907
0.893
0.879
0.96
4.49
0.64
3.00
12.0 11.5 11.0 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
f1 (ppm)


7% D
45% D
25% D
D
D
Et
CD3
CD3
25% D
O
O
Ph
7.614
7.611
7.608
7.603
7.596
7.593
7.591
7.590
7.586
7.581
7.466
7.464
7.462
7.458
7.444
7.440
7.428
7.424
7.423
7.421
7.420
7.416
7.404
7.403
7.399
7.372
7.369
7.365
7.355
7.350
7.345
7.332
7.329
7.260 CDCl3
2.556
2.524
2.392
2.360
2.056
2.046
2.037
2.020
2.014
2.008
2.002
1.990
1.983
1.972
1.965
1.954
1.936
1.918
1.339
1.321
1.308
1.302
0.994
0.976
0.959
0.957
0.857
0.839
0.820
4.16
4.10
1.14
0.55
0.93
2.05
2.24
2.24
3.00
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0
f1 (ppm)

9. Proposed General Mechanism:

"A general mechanism has been proposed in Scheme. Upon generation of the active Ligand-Metal complex **Int-I**, coordination of the model substrate **1a** to Pd forms **Int-IIa**, followed by pyridone-assisted CMD-type regioselective cyclopalladation of the β-methylene C-H bond to form **Int-III**. Then dissociation of the substrate carboxylate creates a vacant site for coordination of the adjacent C-H bond and subsequent β-hydride elimination and Pd reduction deliver **Int-IV**. Then oxidative addition of the ArI to Pd and subsequent reaction with Ag-salt generate **Int-V**. Next Aryl insertion occurs at the γ-position through Int-VI, followed by β-hydride elimination generate **Int-VII**. Subsequent cyclization delivered the γ-arylated γ-lactone product (**3a**) and while the Pd(II) species is regenerated by a Ag(I) oxidant, closing the catalytic cycle. Beside the main catalytic cycle, a reversible but unproductive β-methyl C–H activation (**Int-IIb)** also occurs in the reaction, but it fails to deliver any product under the reaction condition.



Proposed General Mechanism

10. References:

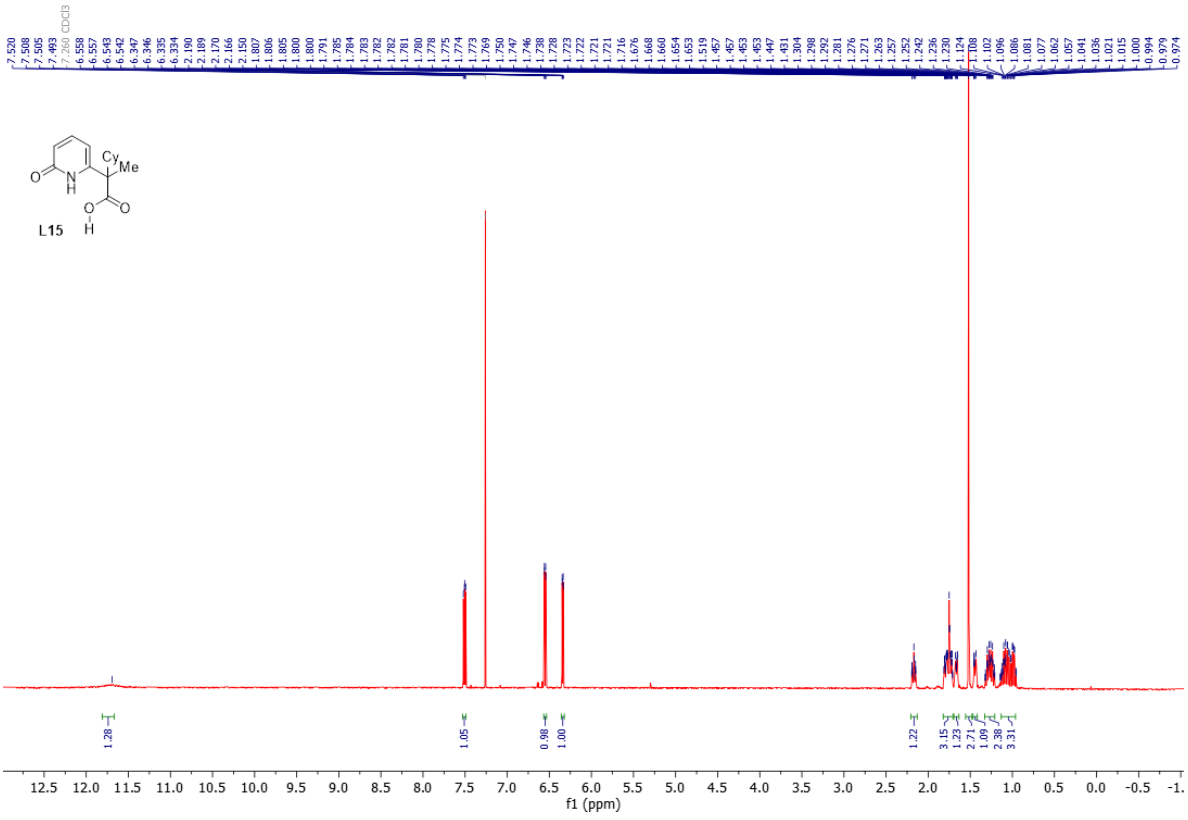
1. Z. Li, Z. Wang, N. Cheksin, S. Q. Qian, J. X. Qiao, P. Cheng, K.-S. Yeung, W. R. Ewing, J.-Q. Yu, *Science* **2021**, *372*, 1452–1457.

2. Z H. S. S. Chan, J. -M. Yang, J. -Q. Yu, *Science* **2022**, *376*, 1481−1487.

4. Z. Zhuang, C. -B. Yu, G. Chen, Q. -F. Wu, Y. Hsiao, C. L. Joe, J. X. Qiao, M. A. Poss, J. -Q. Yu, *J. Am. Chem. Soc.* **2018**, *140*, 10363−10367.

(5) G. Kang, D. A. Strassfeld, T. Sheng, C. -Y. Chen, J. -Q. Yu, *Nature* **2023**, *618*, 519–525.
(6) Z. Li, H. S. Park, J. X. Qiao, K. -S. Yeung, J.-Q. Yu *J. Am. Chem. Soc.* **2022**, *144*, 18109–18116.

11. NMR Spectra: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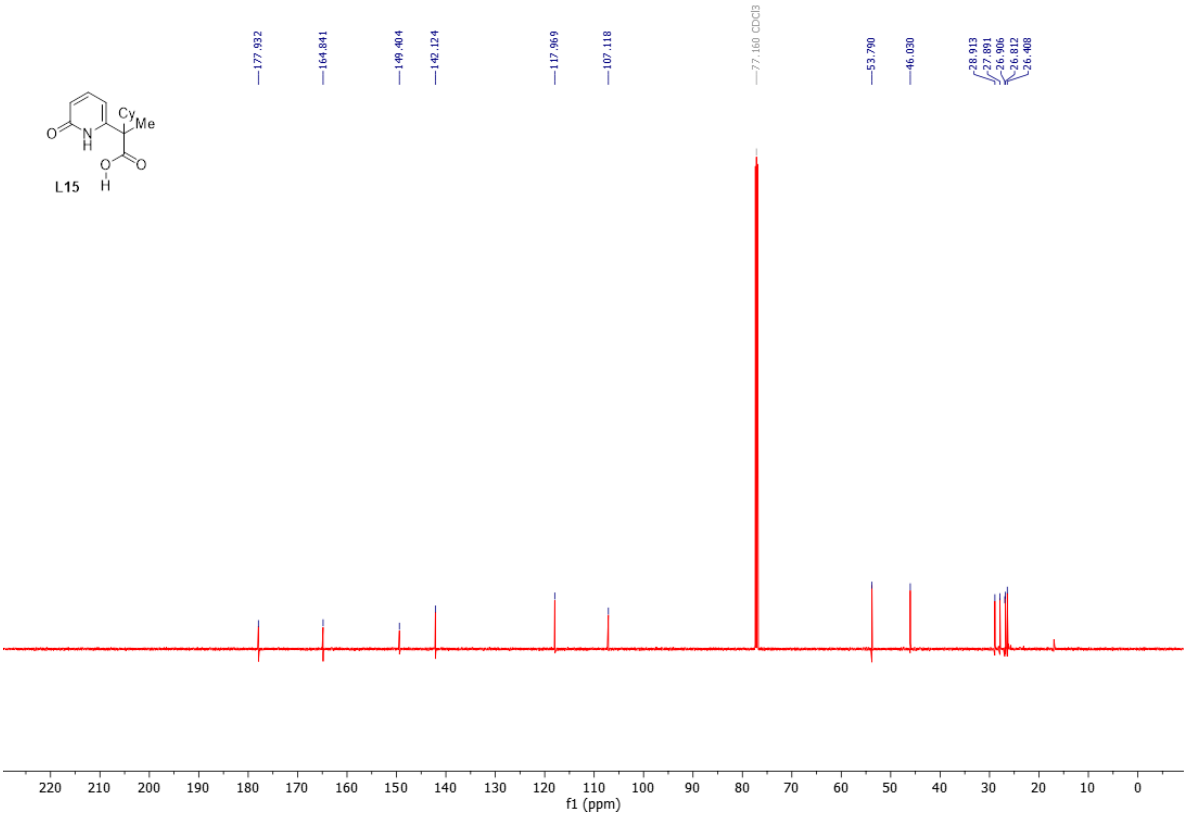
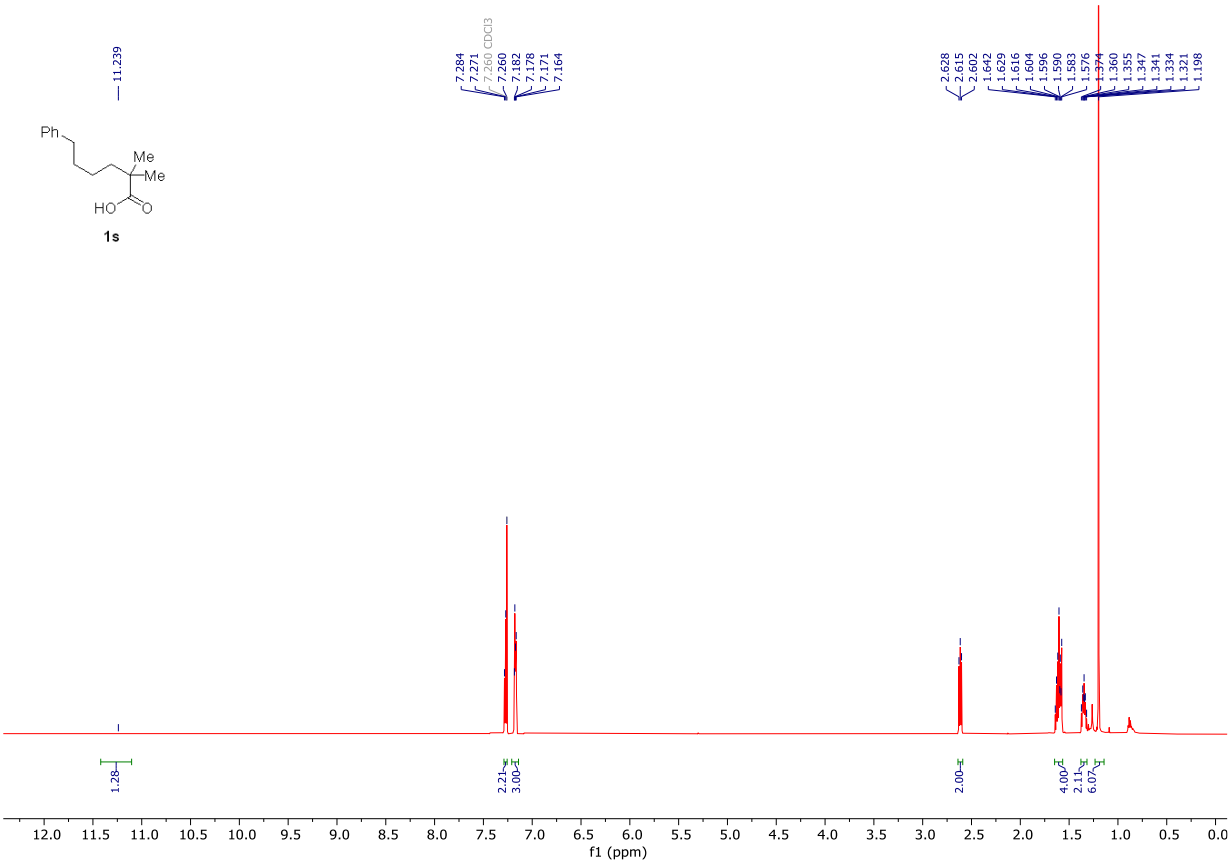
f1 (ppm)


L15
f1 (ppm)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2.602
1.642
1.629
1.616
1.604
1.596
1.590
1.583
1.576
1.394
1.360
1.355
1.347
1.341
1.334
1.321
1.198
Ph
Me
Me
HO
O
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4.00
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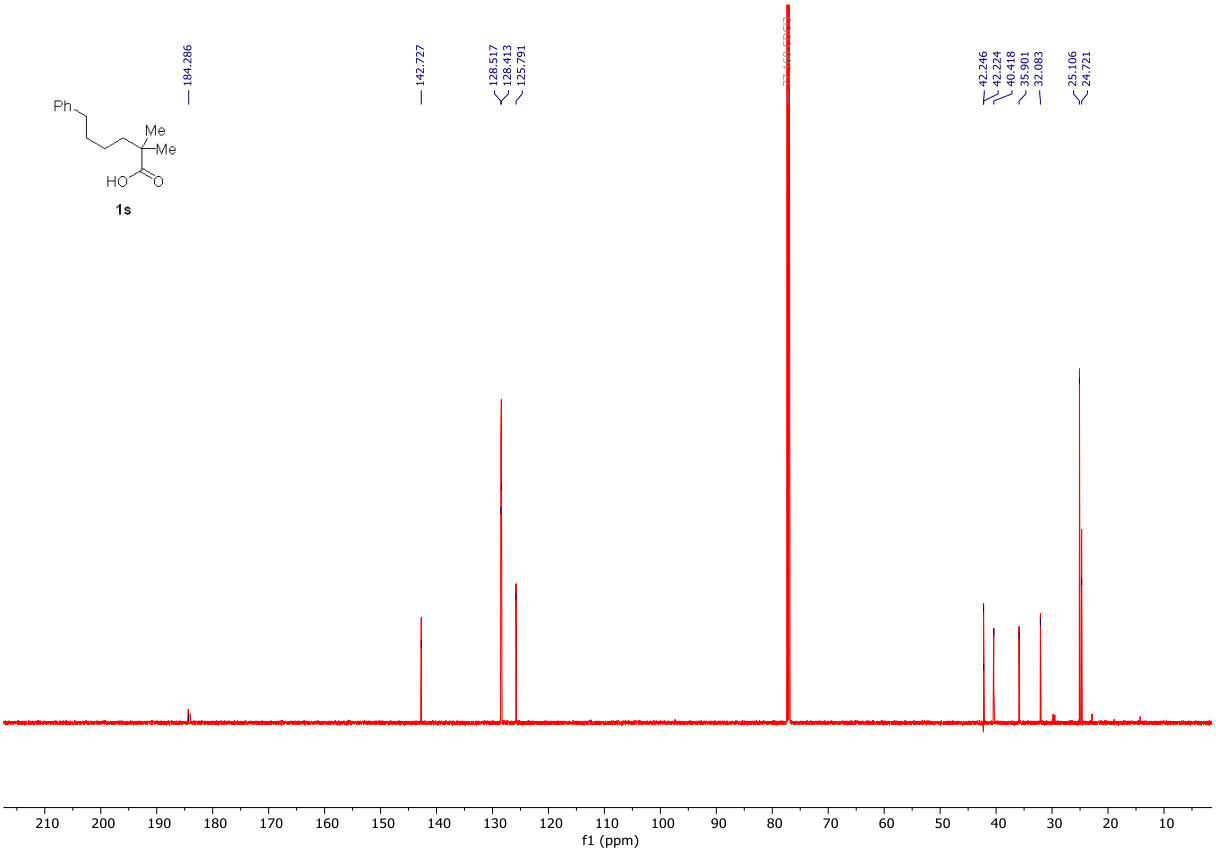
6.07
f1 (ppm)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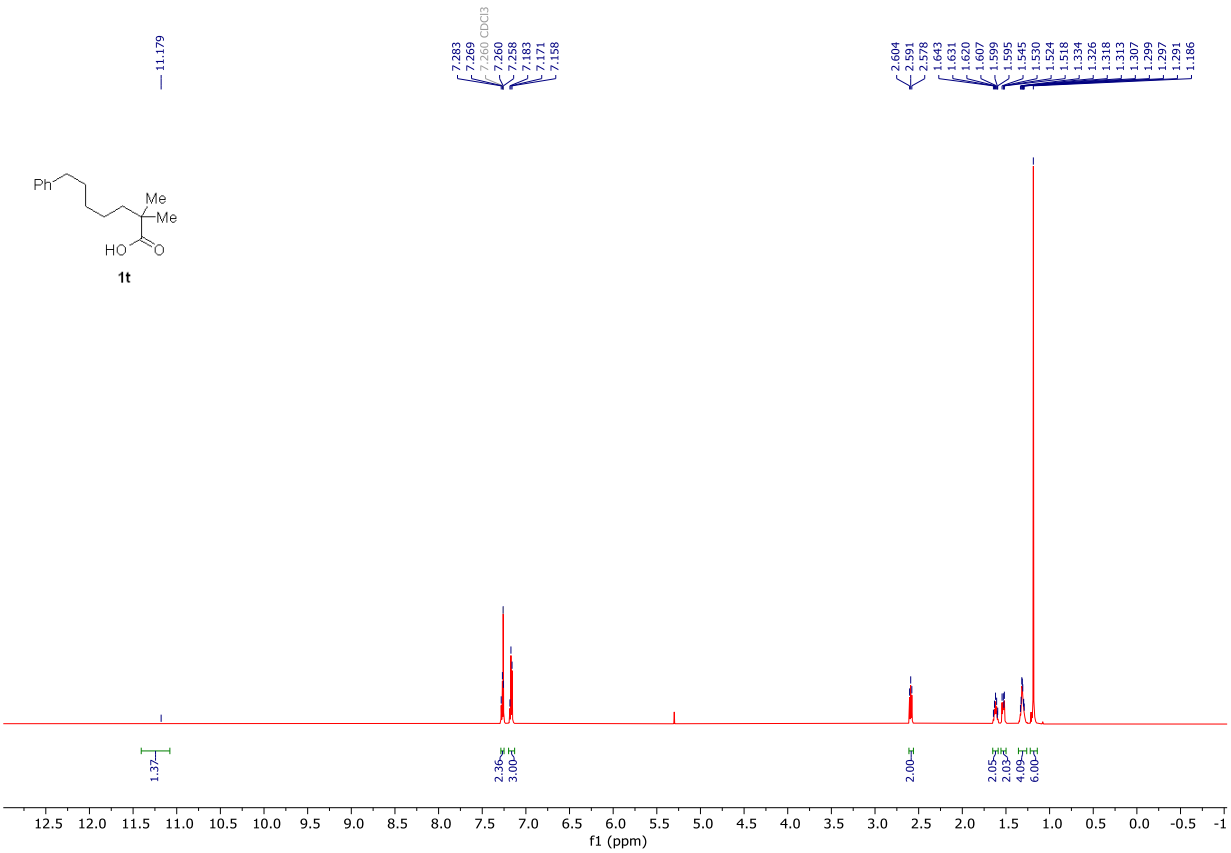
f1 (ppm)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1.599
1.595
1.545
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1.518
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1.307
1.299
1.297
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Ph
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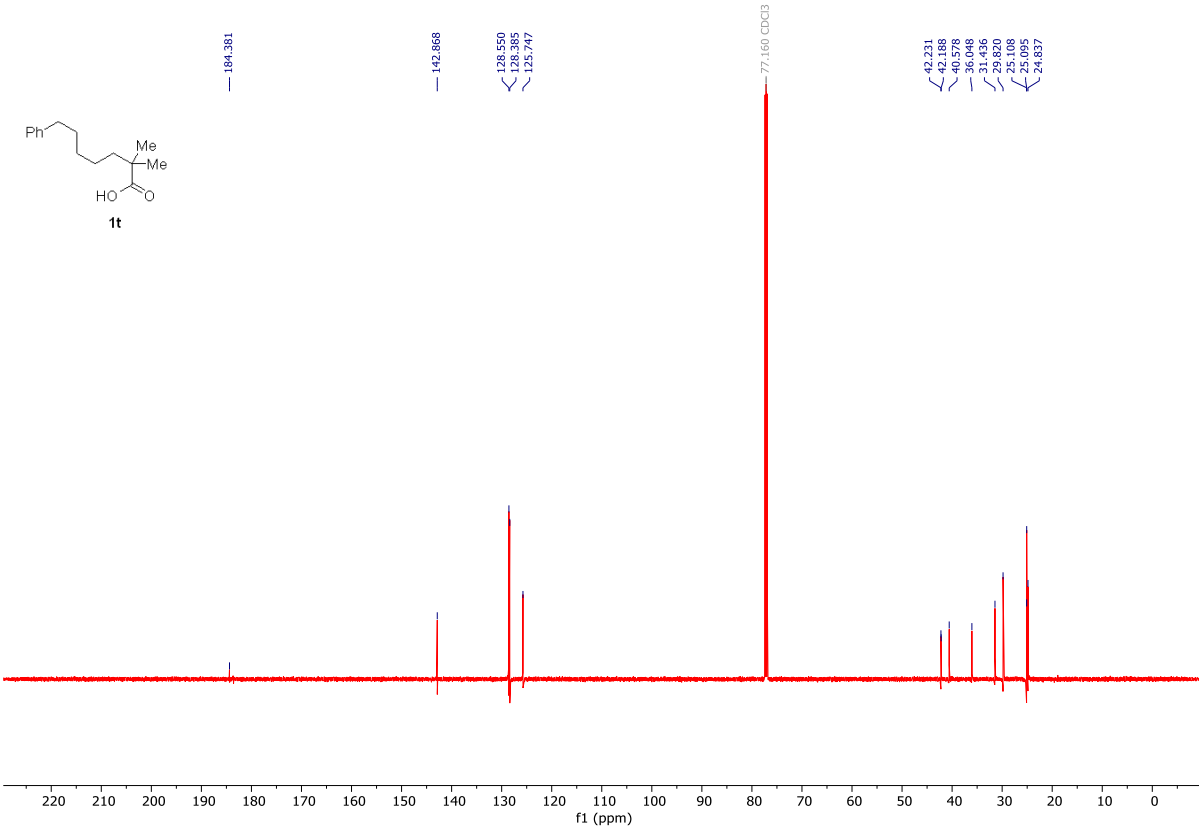
f1 (ppm)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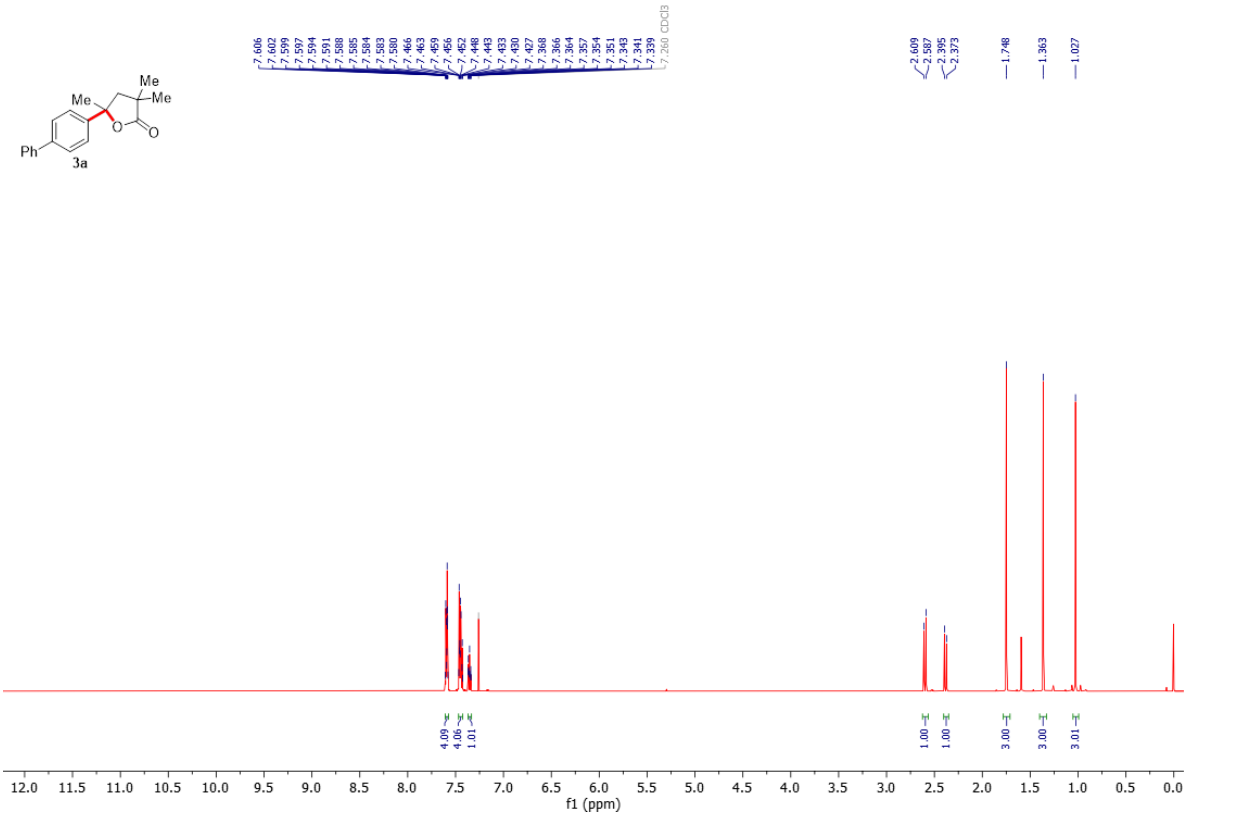
Ph
Me
Me
HO
O
1t
f1 (ppm)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7.343
7.341
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2.587
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1.363
1.027
4.09
4.08
1.01
1.00
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f1 (ppm)
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Me
Me
O
O
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124.617
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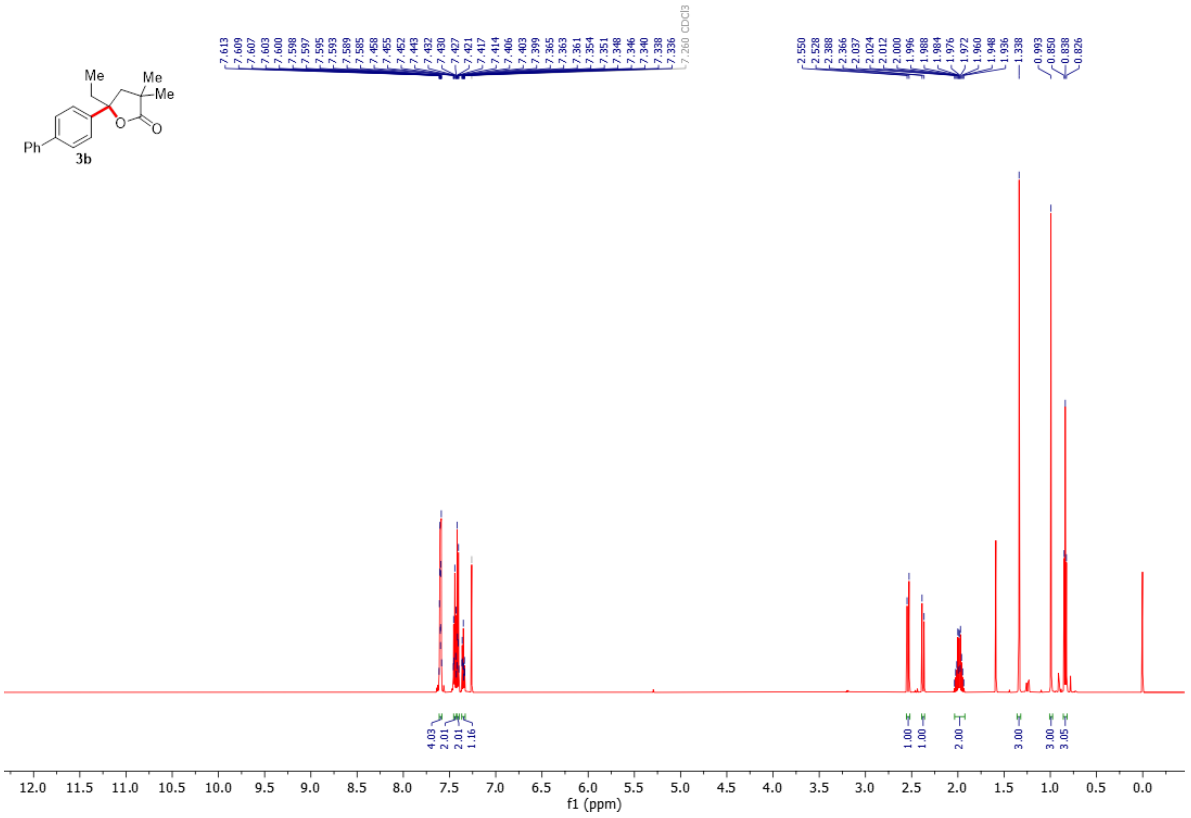
f1 (ppm)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7.348
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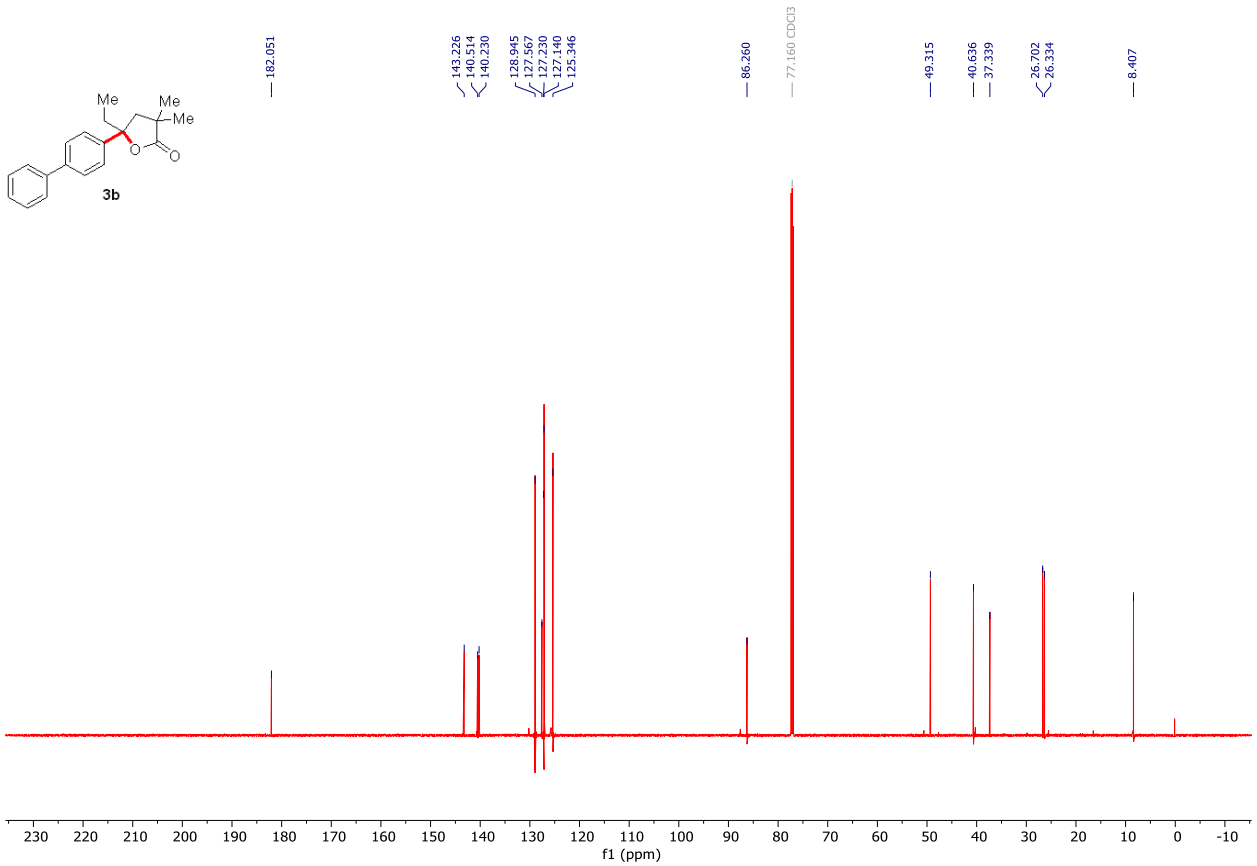
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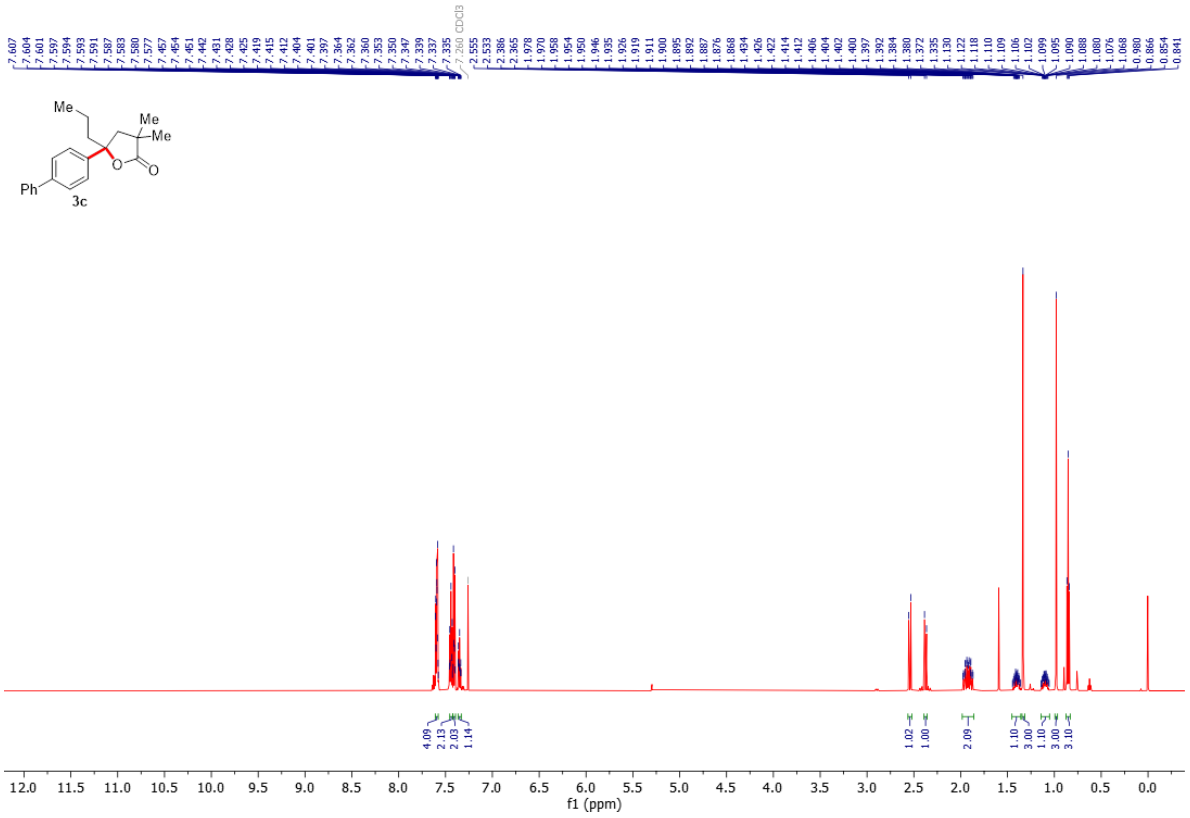
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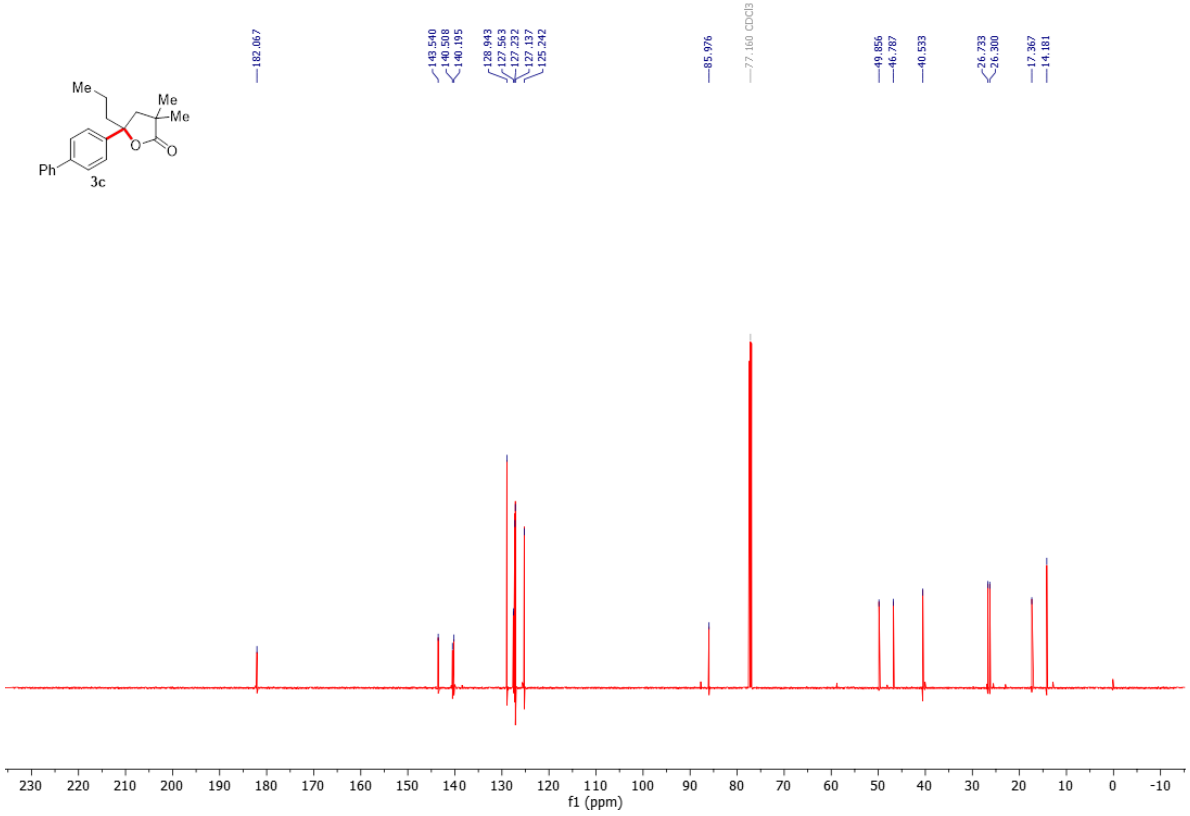
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Ph
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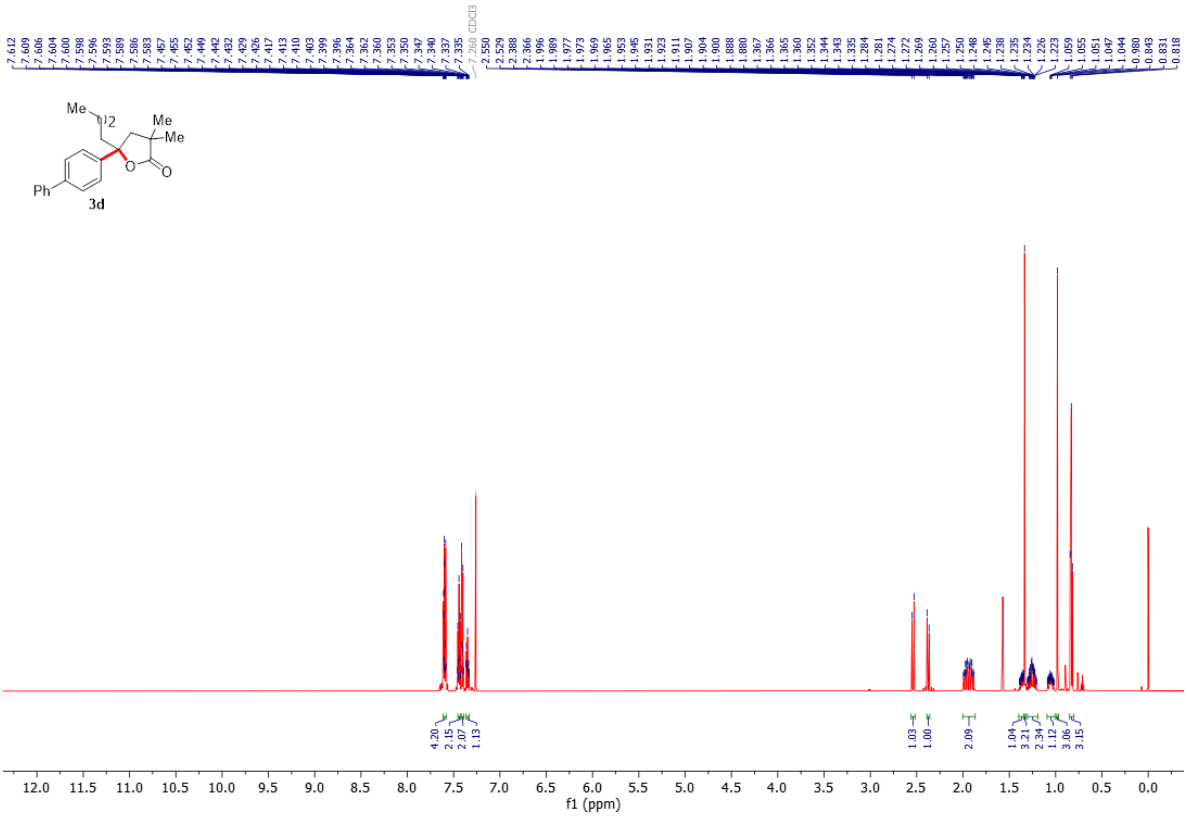
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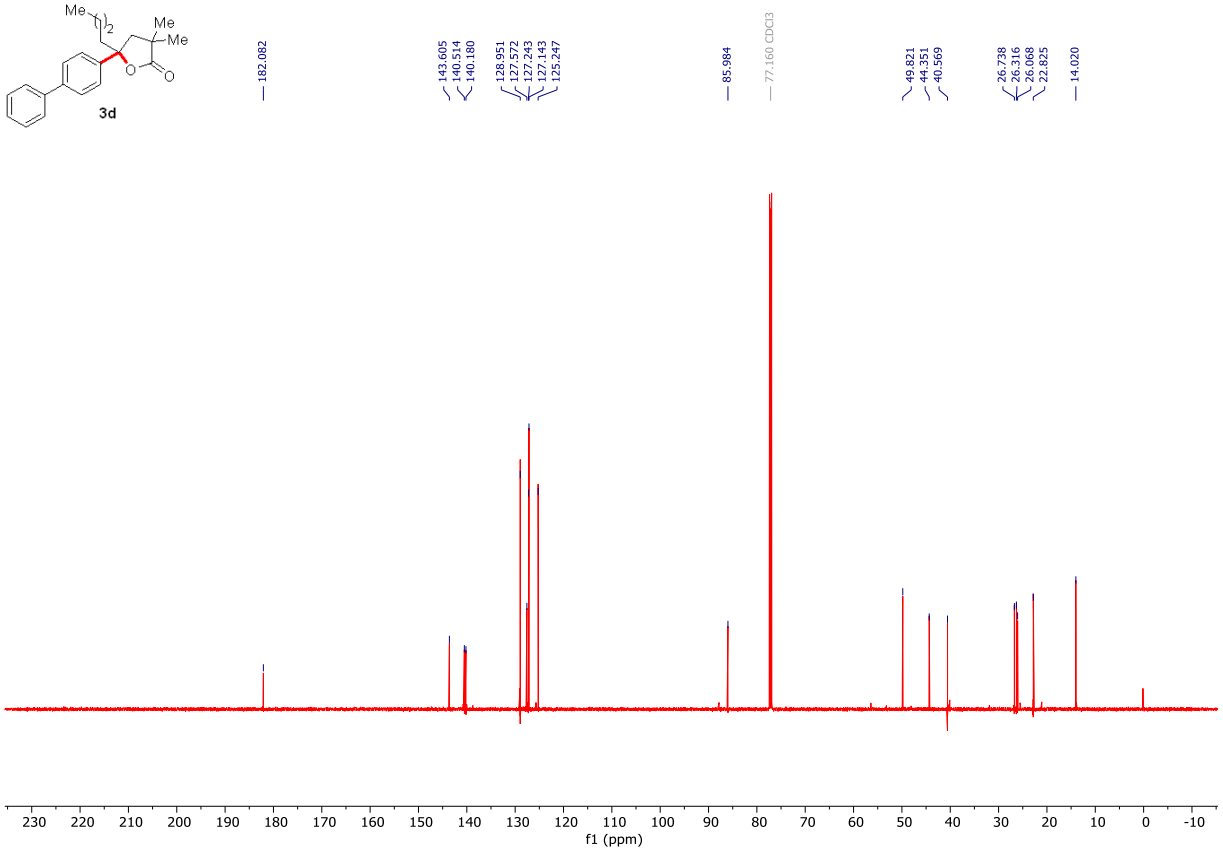
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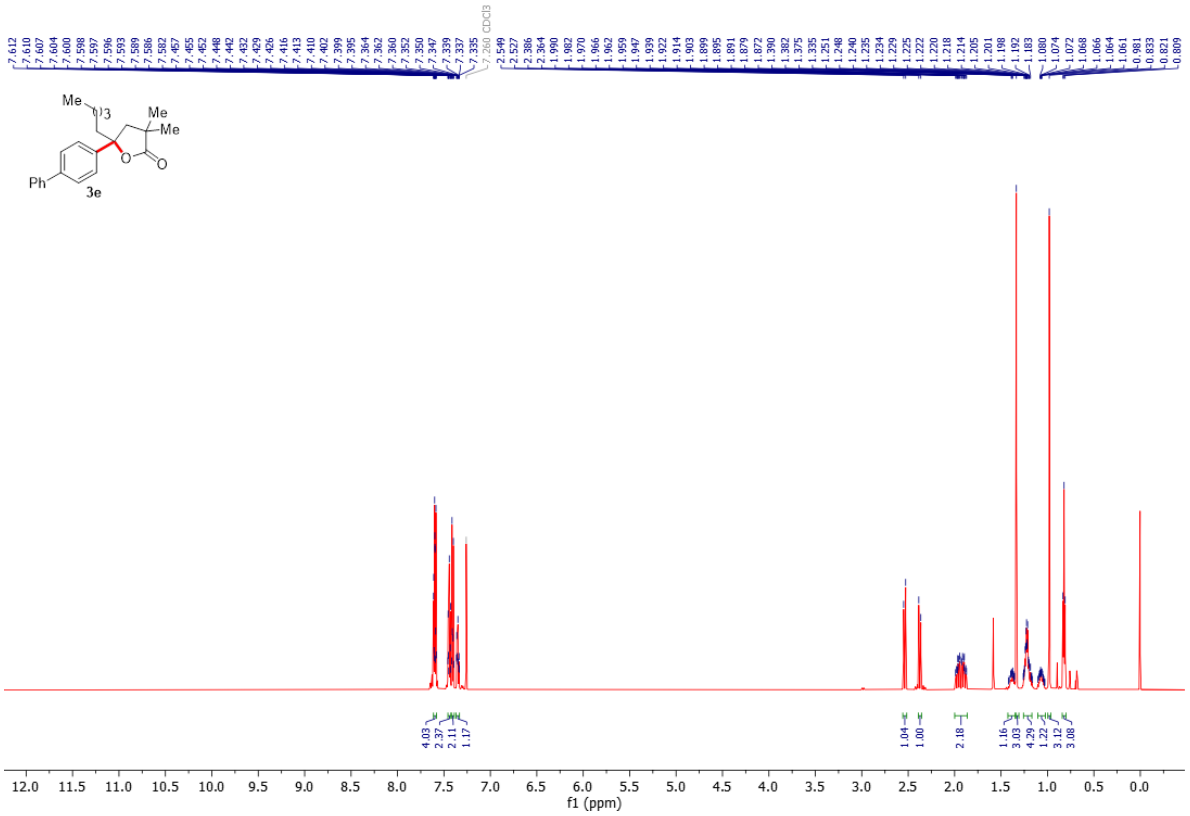
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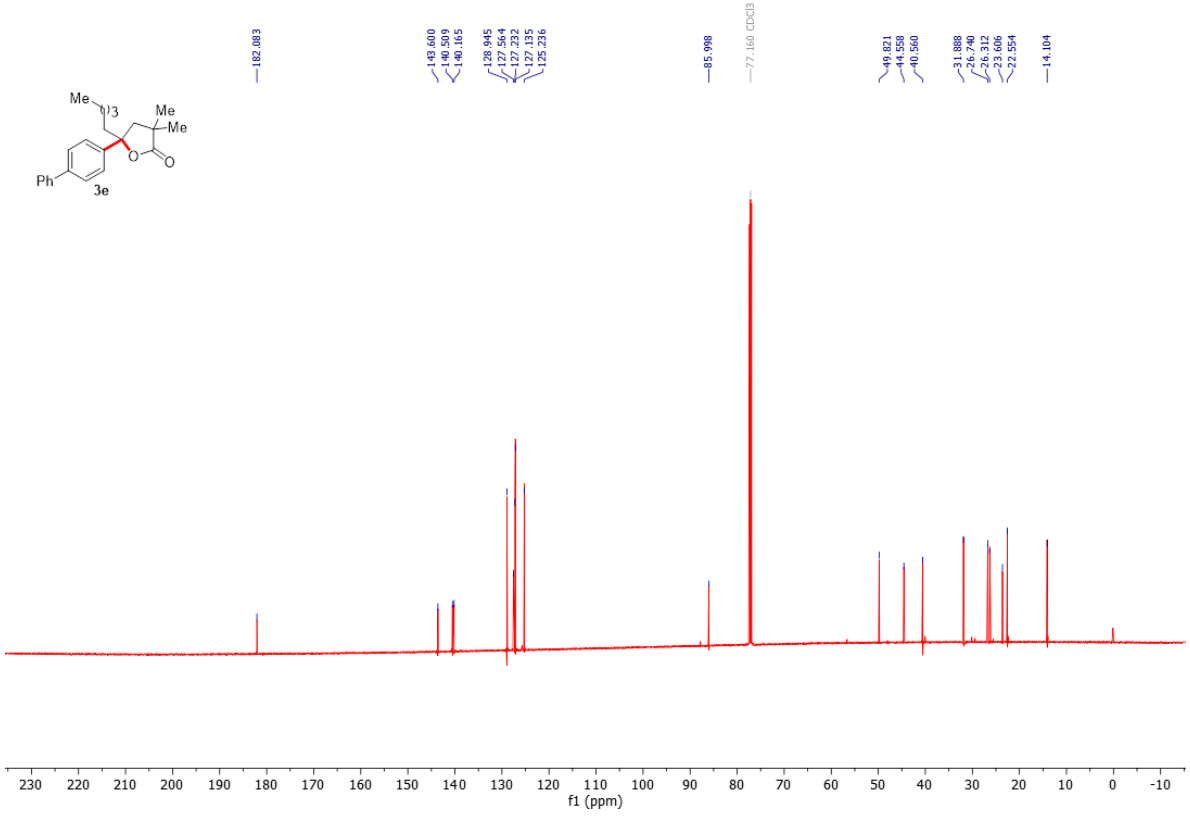
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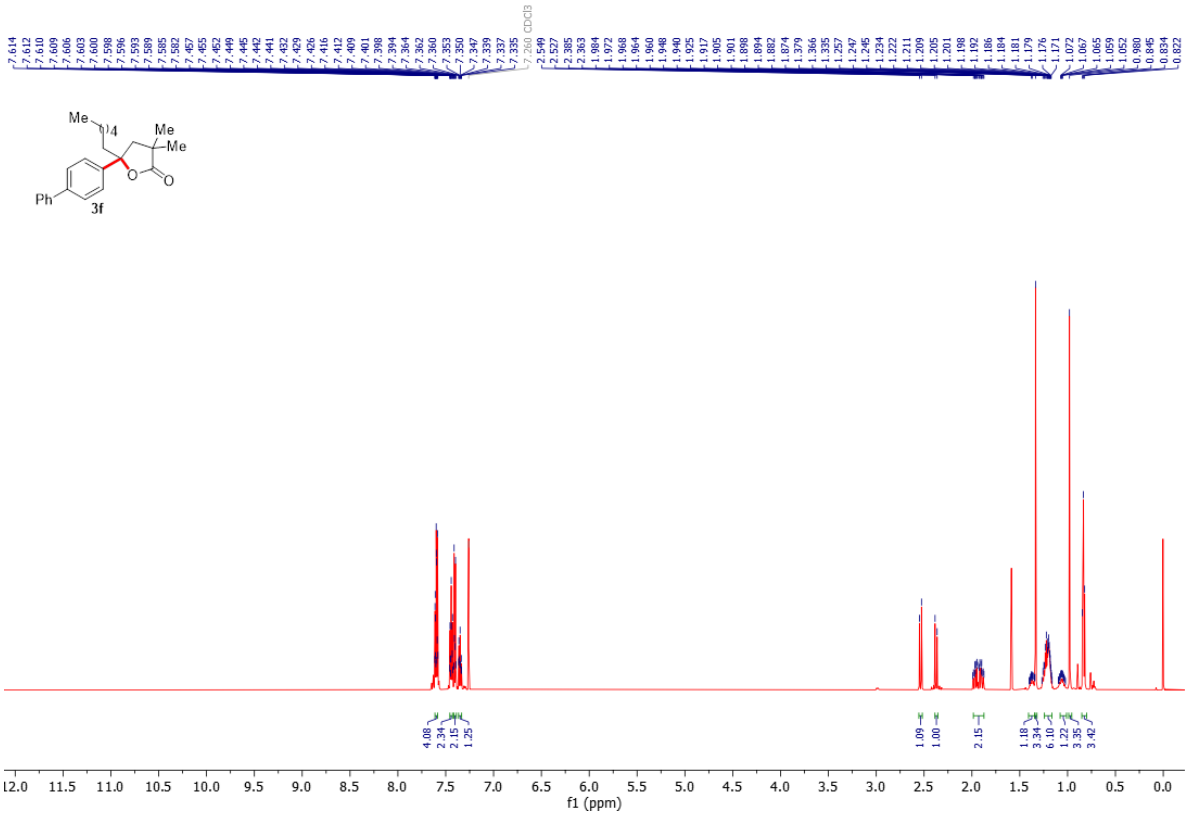
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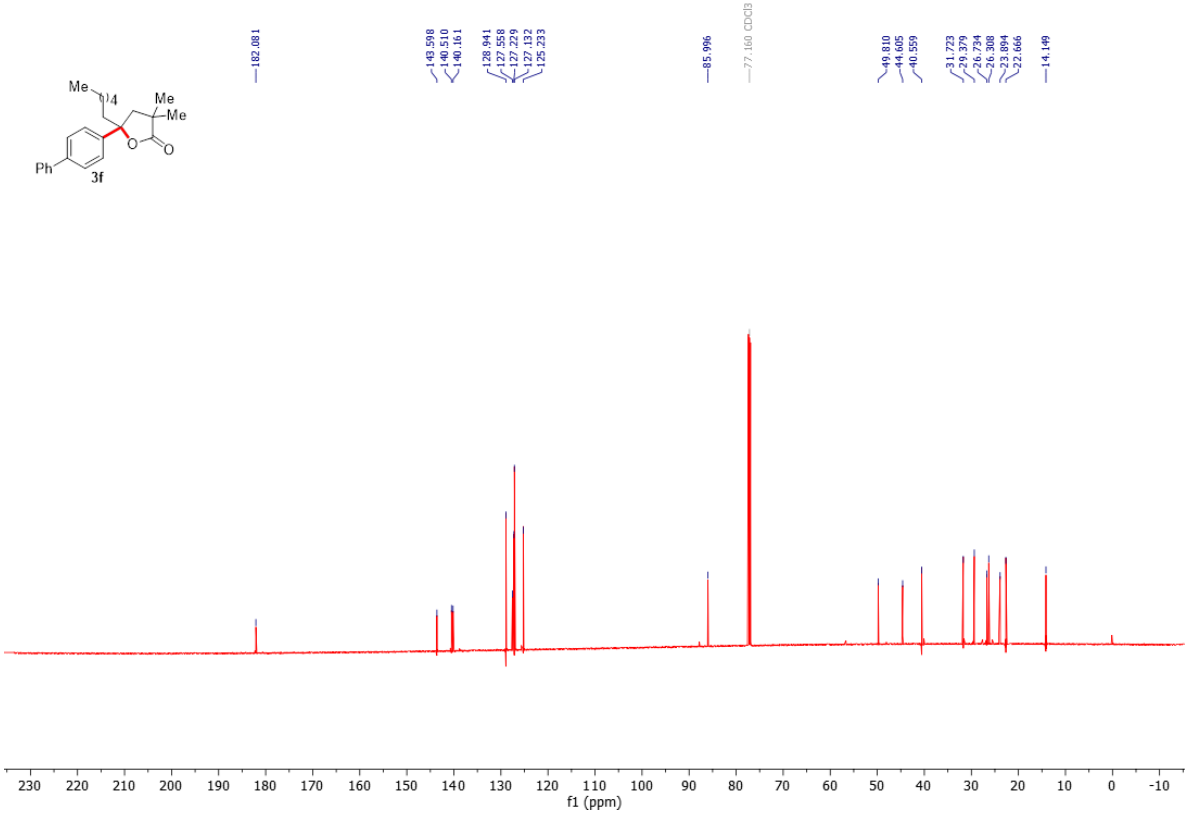
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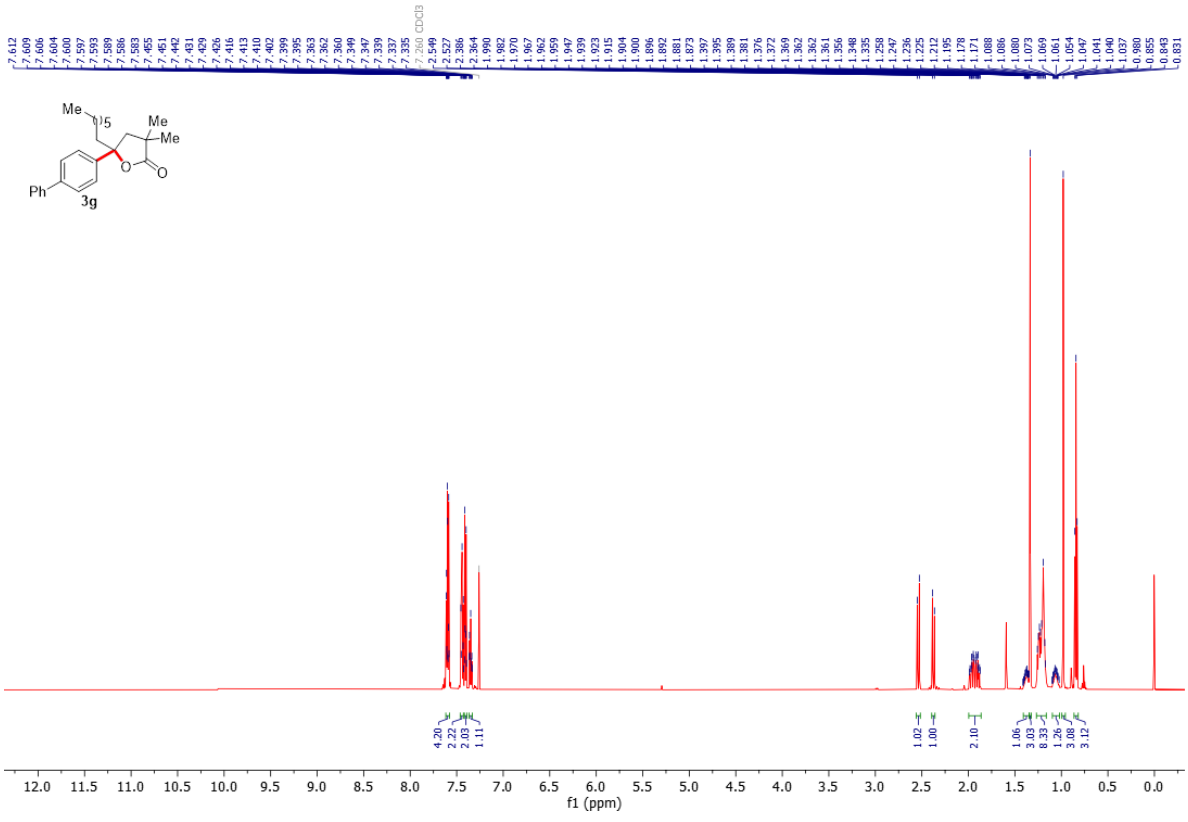
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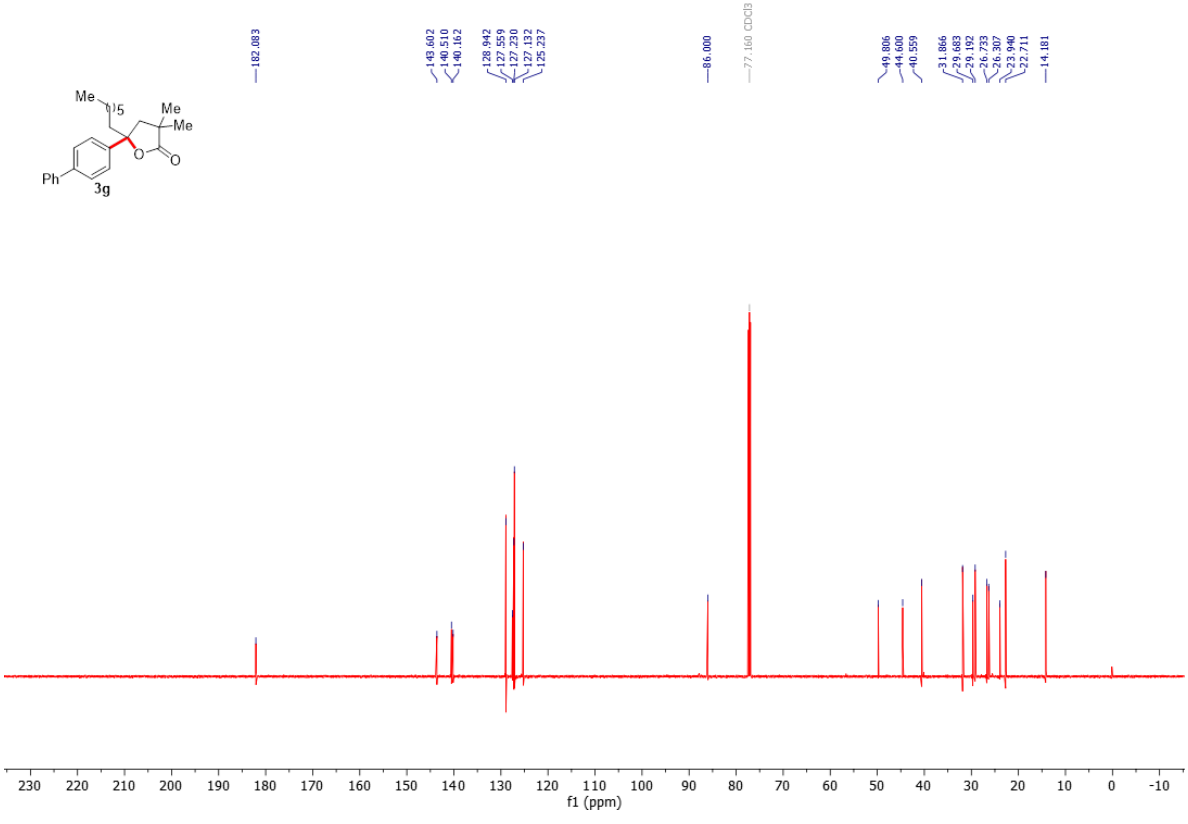
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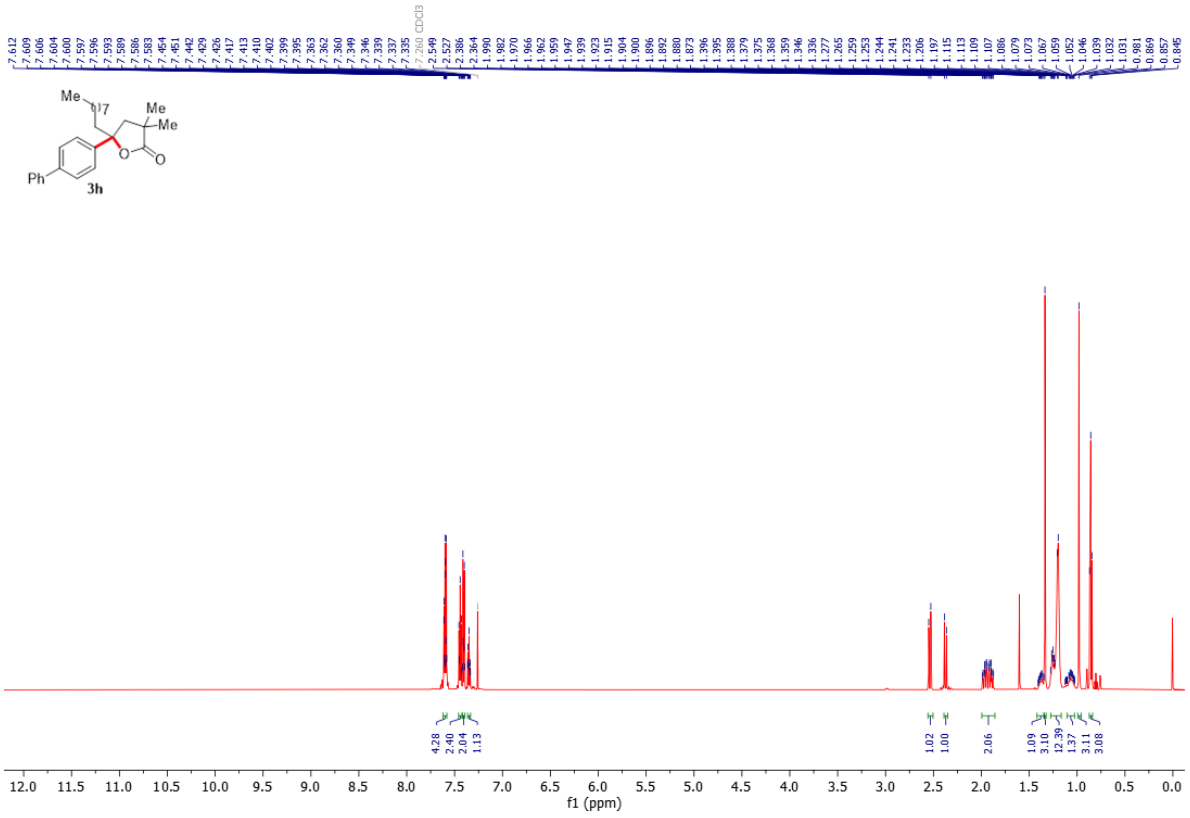
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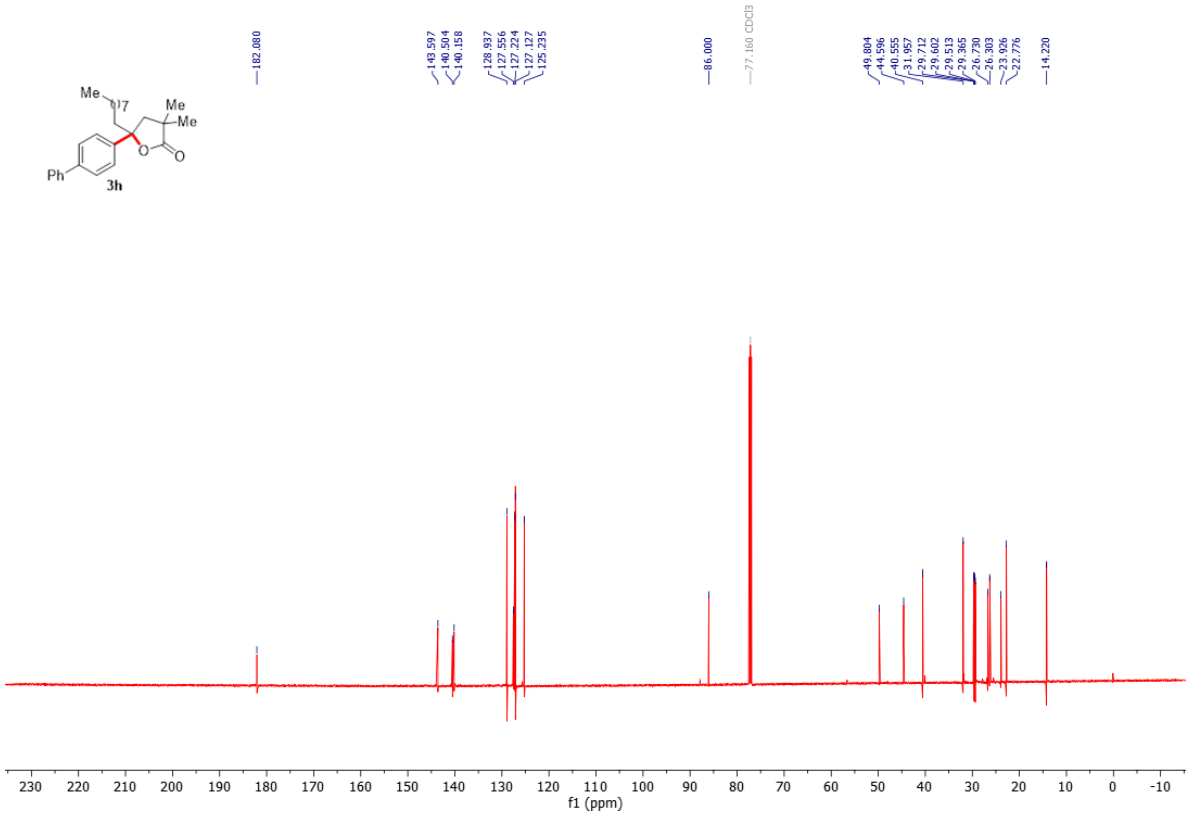
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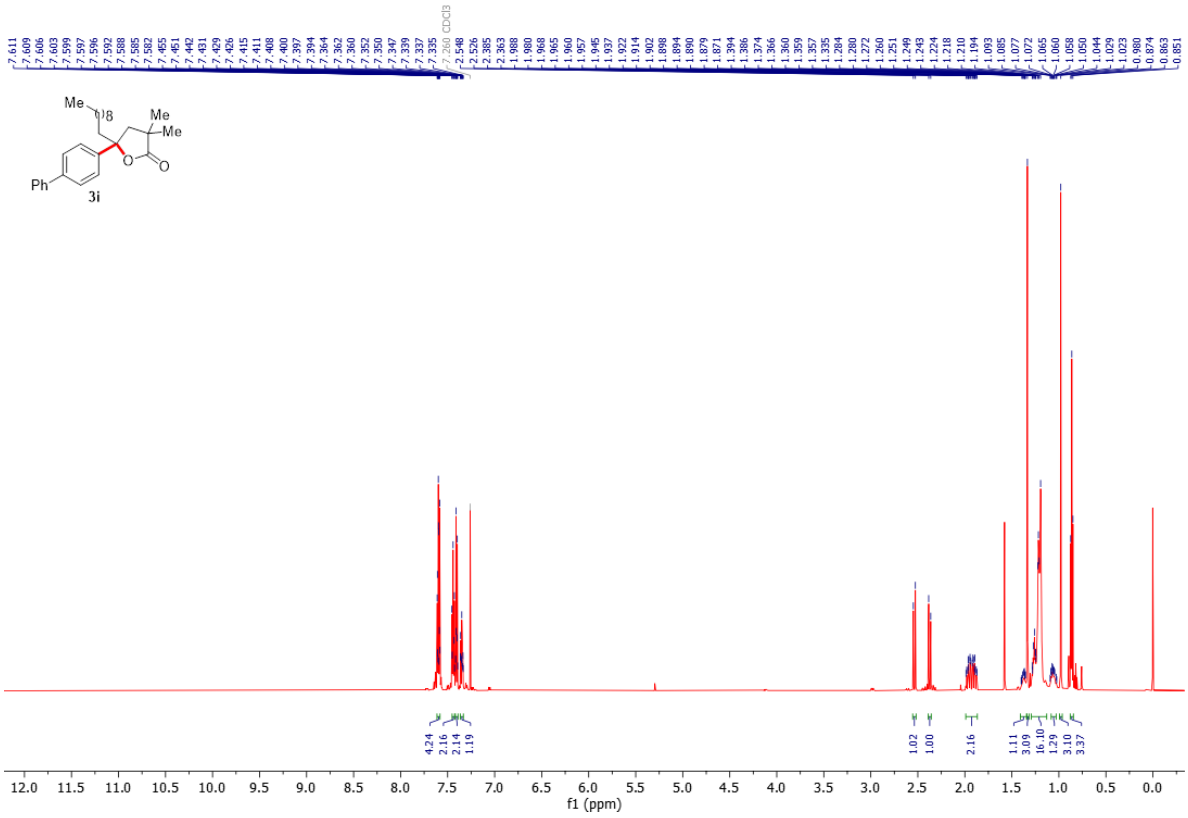
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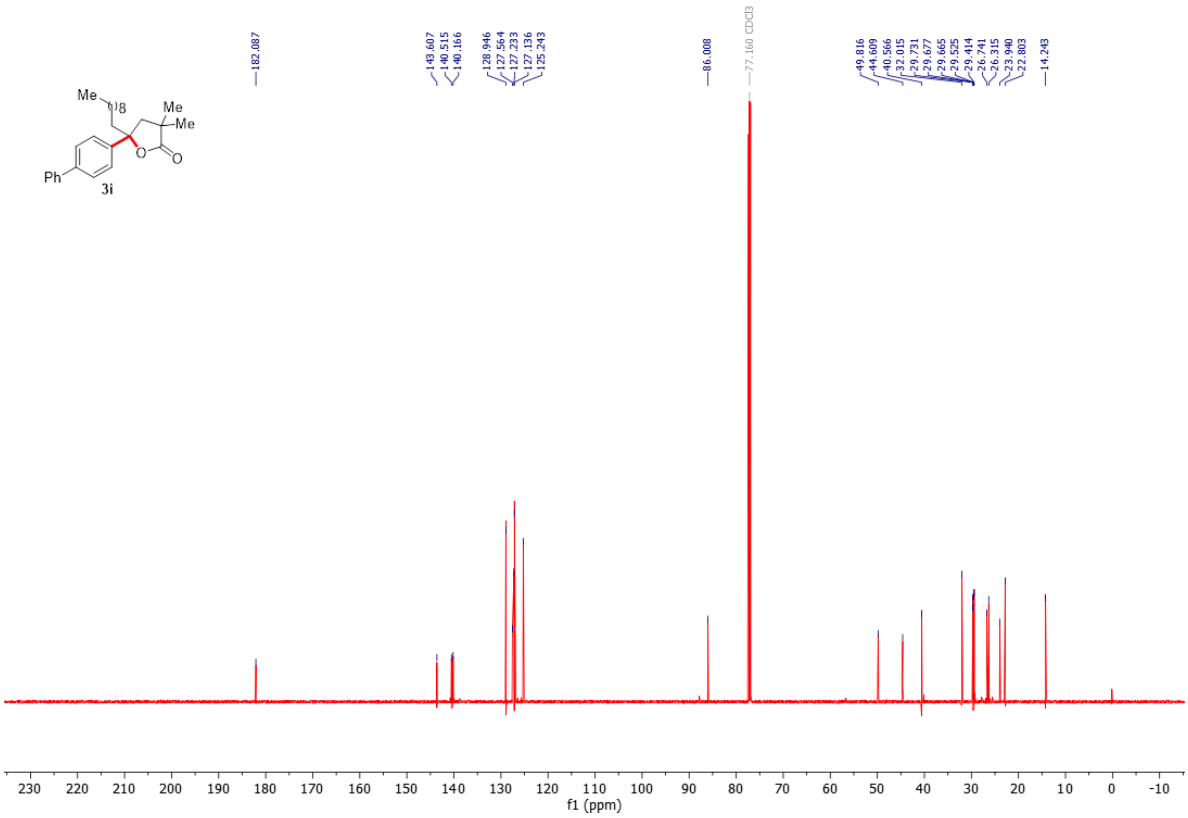
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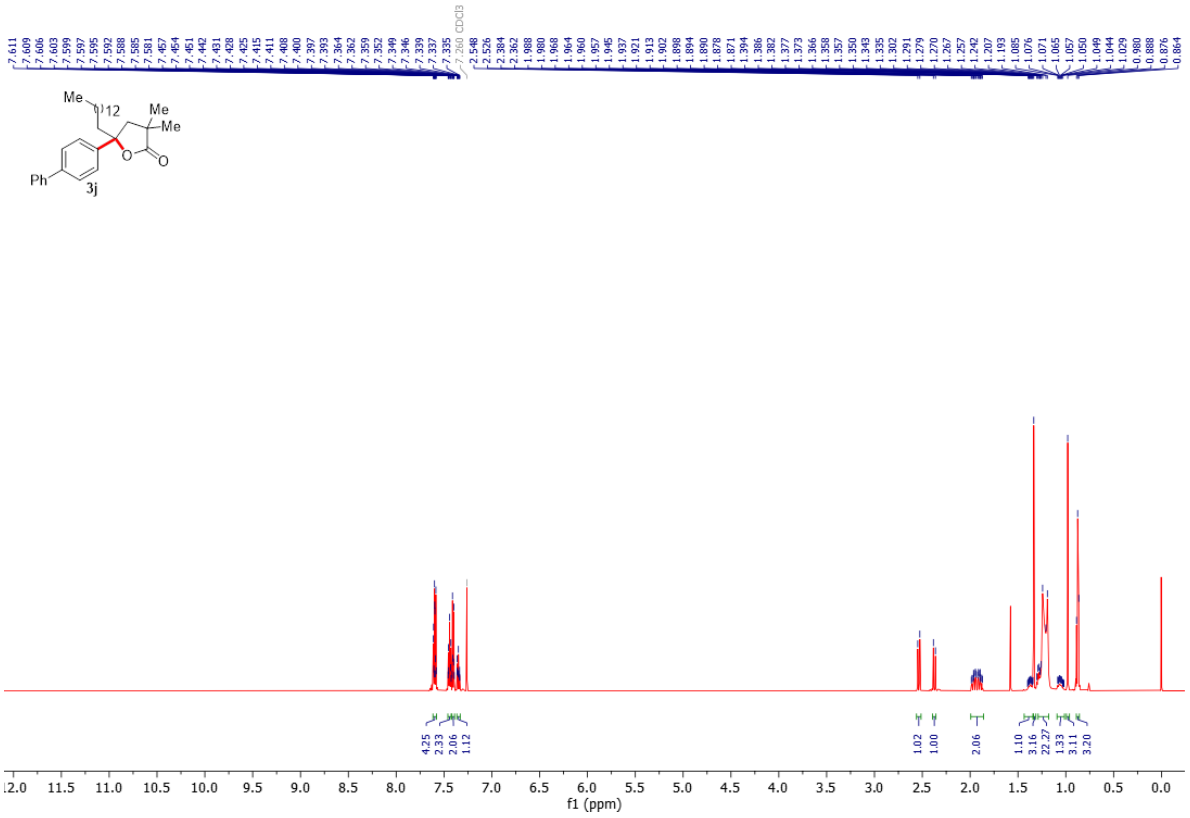
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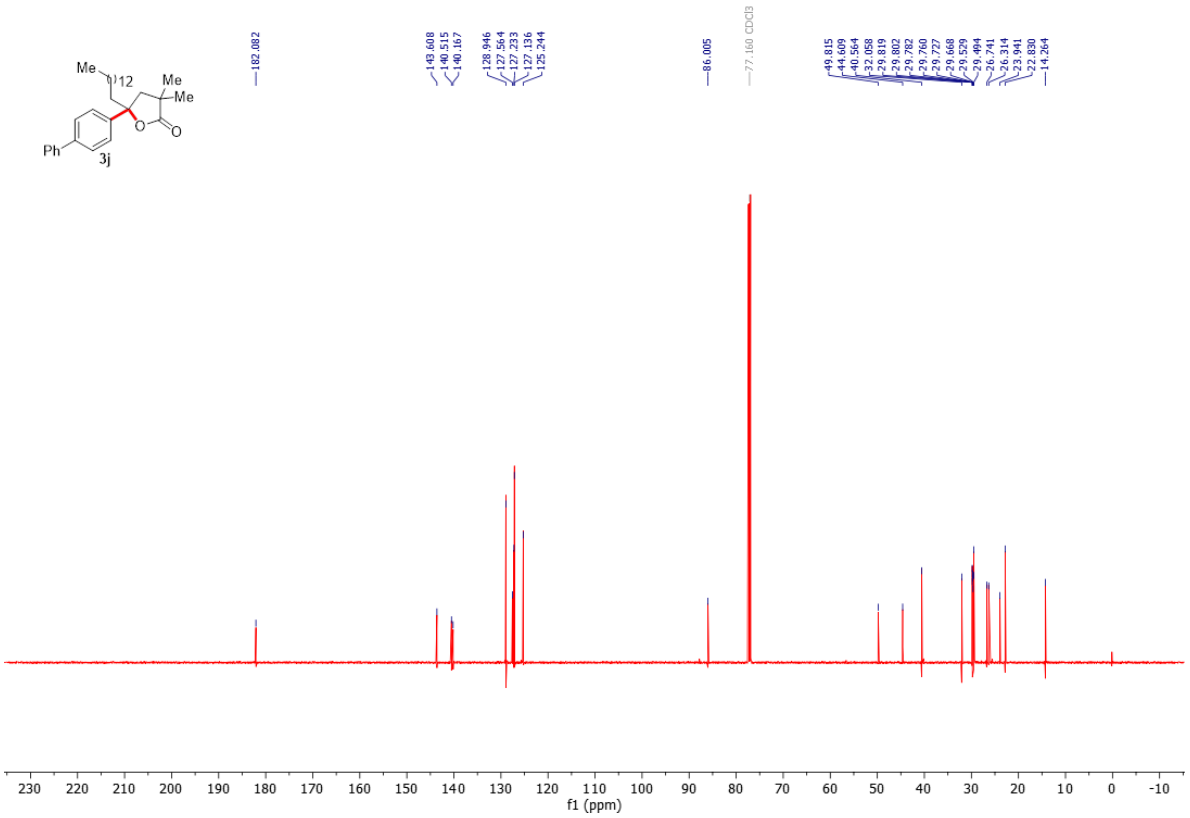
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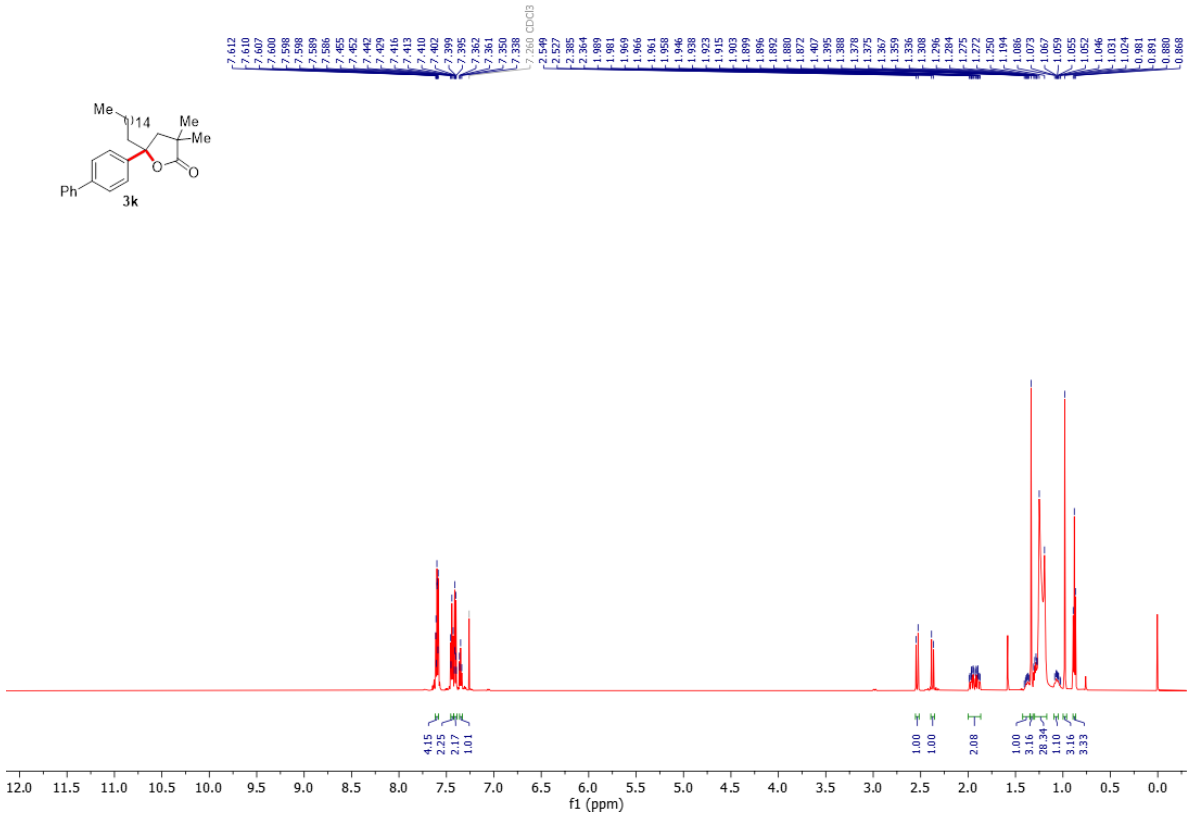
3j
f1 (ppm)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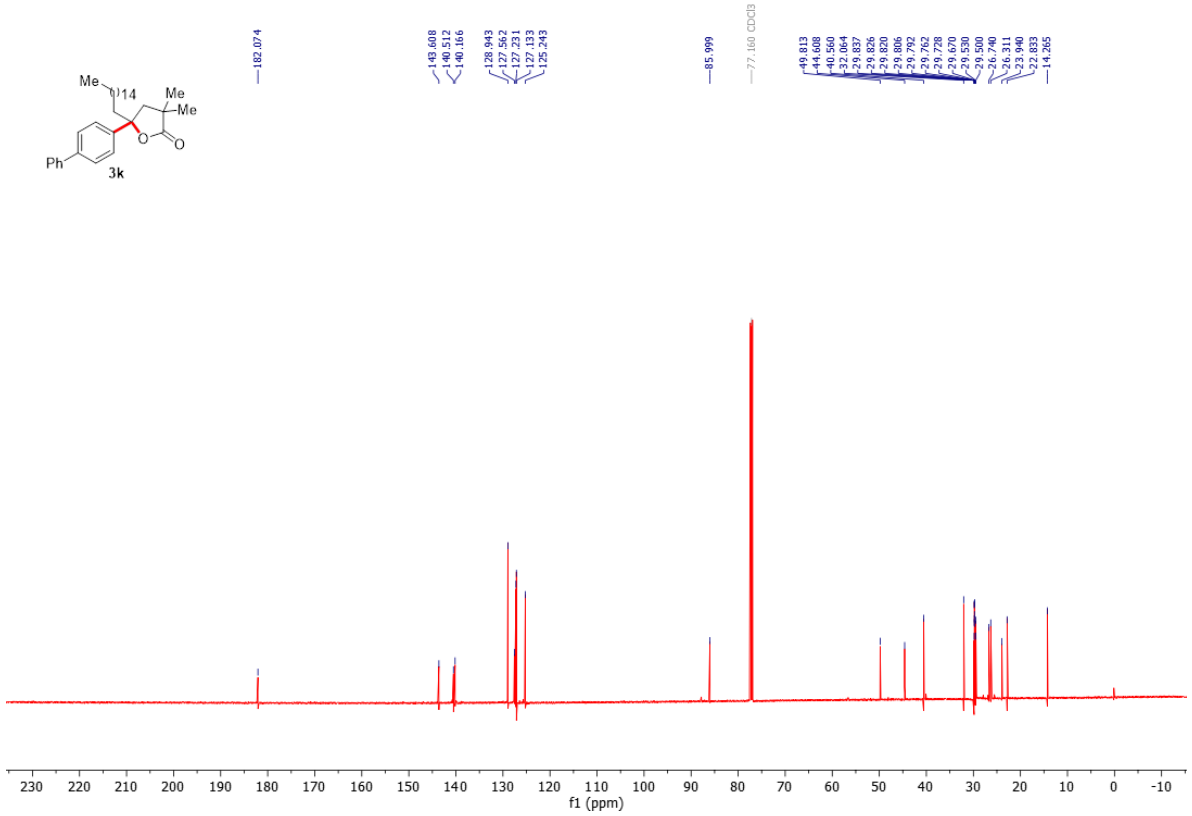
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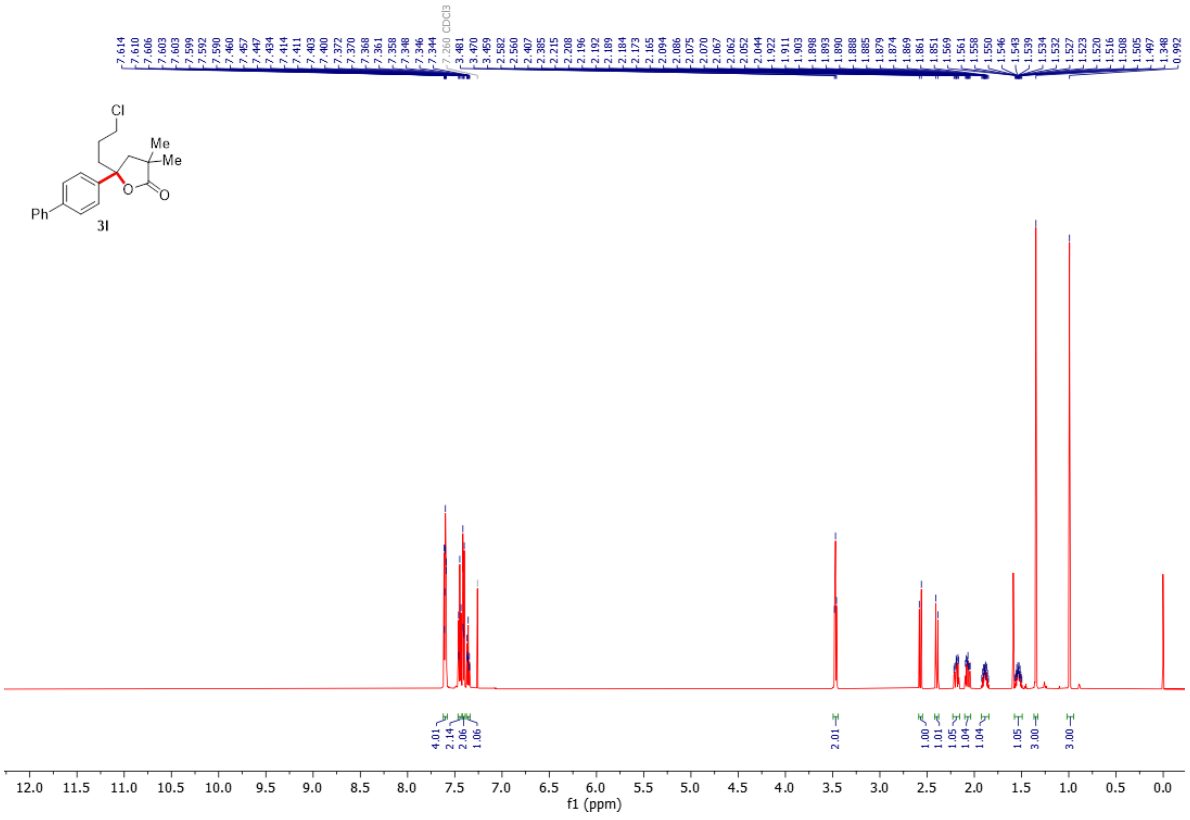
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7.457
7.447
7.434
7.414
7.411
7.403
7.400
7.372
7.370
7.368
7.361
7.358
7.348
7.346
7.344
7.260 CDCl3
3.481
3.470
3.459
2.582
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2.165
2.094
2.086
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2.067
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2.044
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1.903
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1.532
1.527
1.523
1.520
1.516
1.508
1.505
1.497
1.348
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4.01
2.14
2.06
1.06
2.01
1.00
1.01
1.05
1.04
1.04
1.05
3.00
3.00
12.0 11.5 11.0 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
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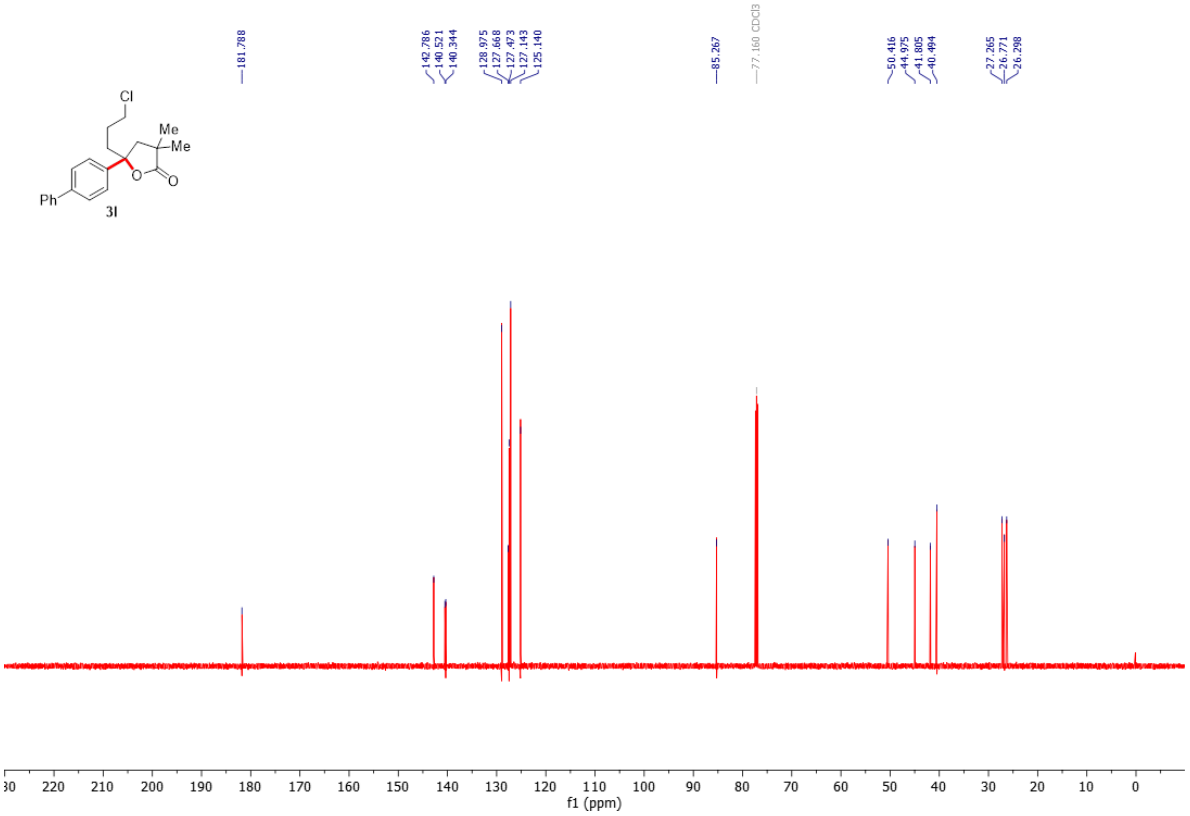
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127.143
125.140
85.267
77.160 CDCl3
50.416
44.975
41.805
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26.771
26.298
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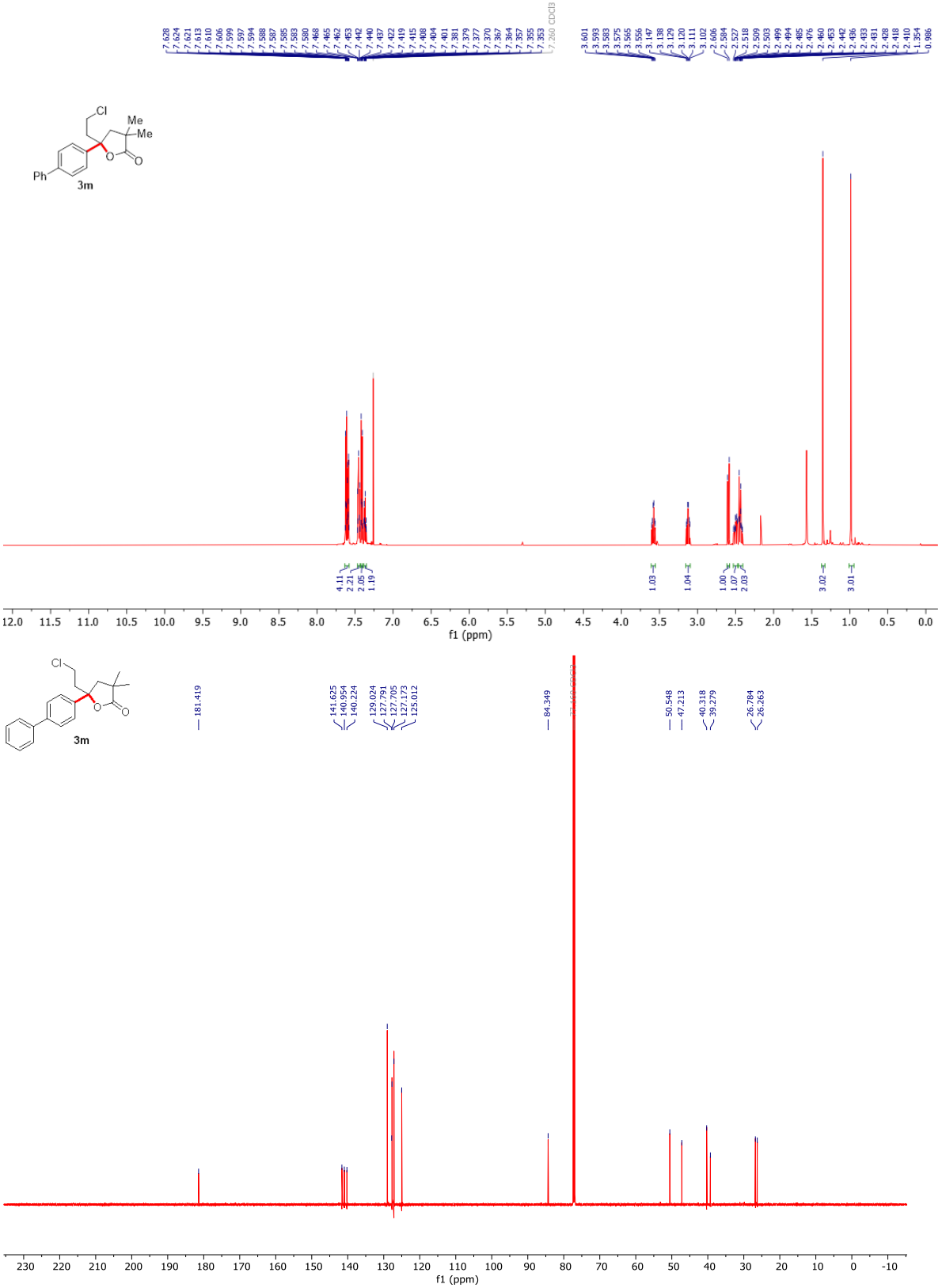
f1 (ppm)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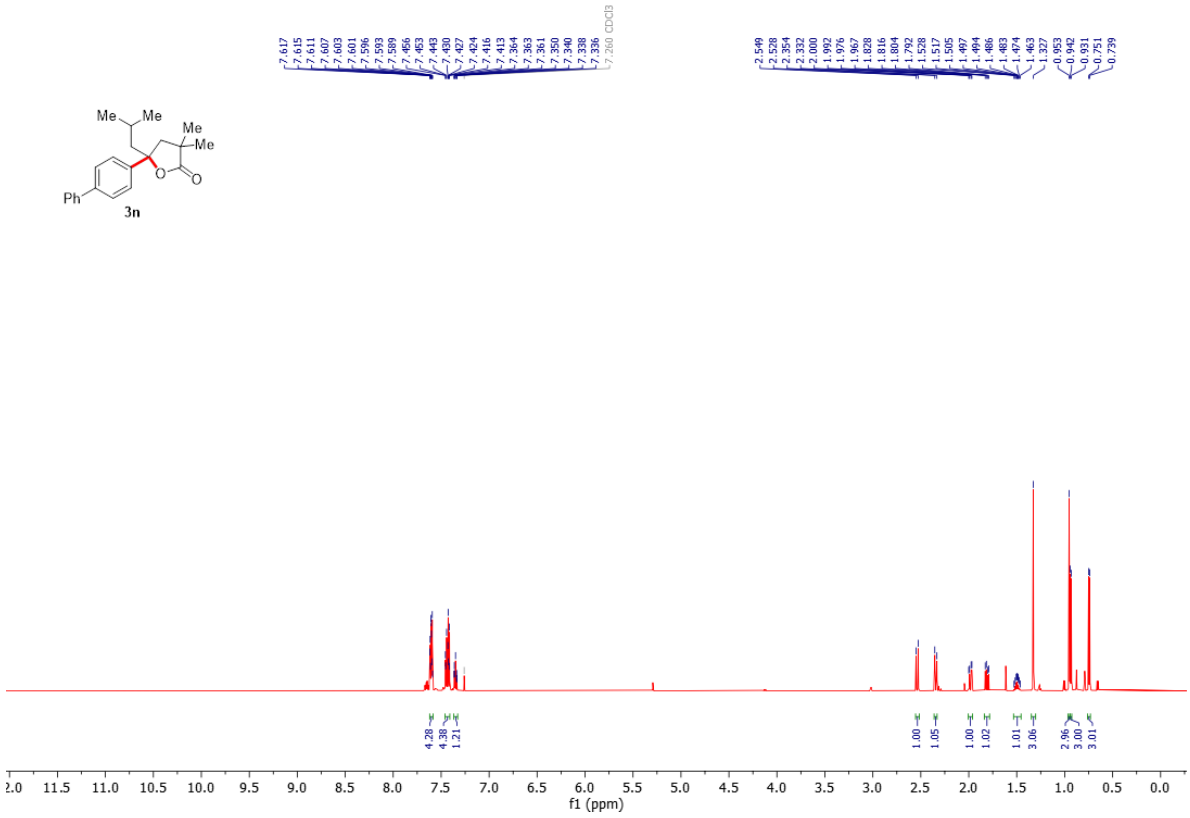
7.367
7.364
7.357
7.355
7.353
7.260 CDCl3
3.601
3.593
3.583
3.575
3.565
3.556
3.147
3.138
3.129
3.120
3.111
3.102
2.606
2.584
2.527
2.518
2.509
2.503
2.499
2.494
2.485
2.476
2.460
2.453
2.442
2.436
2.433
2.431
2.428
2.418
2.410
1.354
0.986
4.11
2.21
2.05
1.19
1.03
1.04
1.00
1.07
2.03
3.02
3.01
12.0
11.5
11.0
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
f1 (ppm)
Cl
O
O
3m
181.419
141.625
140.954
140.224
129.024
127.791
127.705
127.173
125.012
84.349
77.460 CDCl3
50.548
47.213
40.318
39.279
26.784
26.263
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
f1 (ppm)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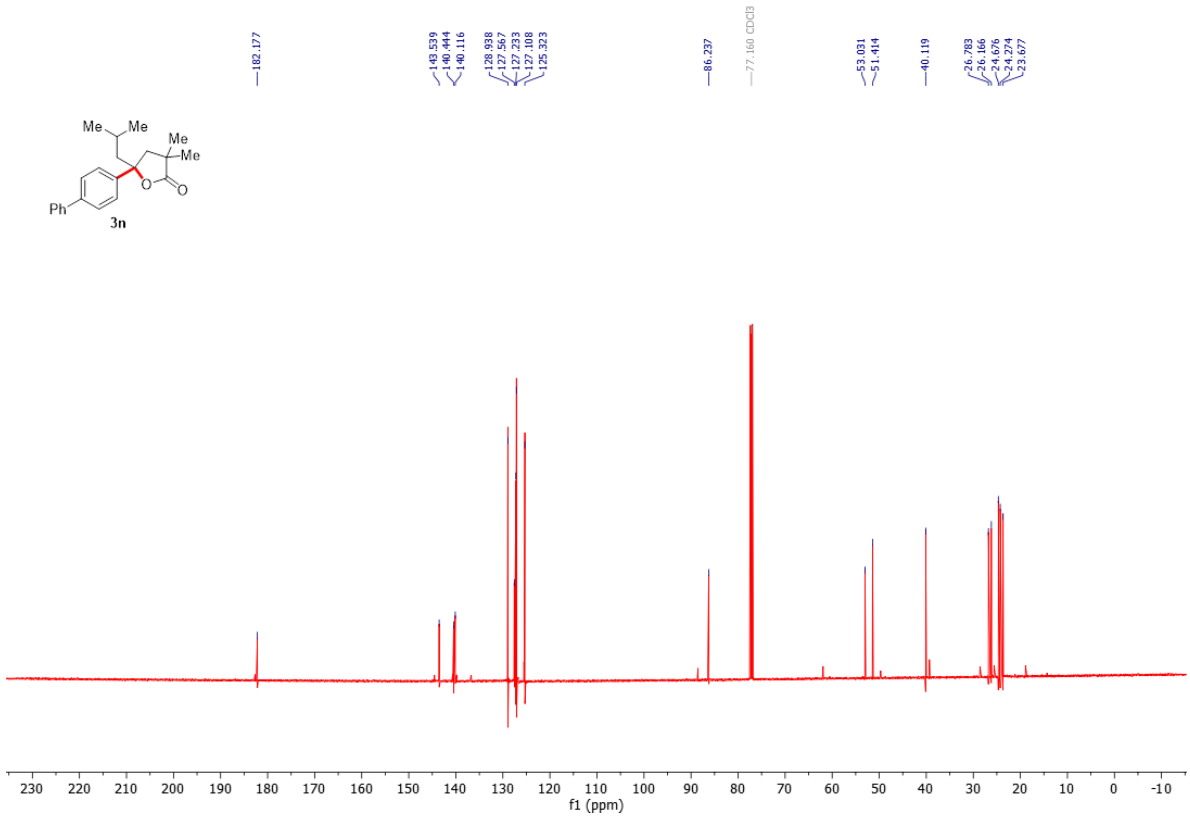
7.615
7.611
7.607
7.603
7.601
7.596
7.593
7.589
7.466
7.463
7.448
7.430
7.427
7.424
7.416
7.413
7.364
7.363
7.361
7.350
7.340
7.338
7.336
7.260 CDCl3
2.549
2.528
2.354
2.332
2.000
1.992
1.976
1.967
1.828
1.816
1.804
1.792
1.528
1.517
1.505
1.497
1.494
1.486
1.483
1.474
1.463
1.327
0.953
0.942
0.931
0.751
0.739
Me
Me
Me
Me
O
O
Ph
3n
4.28
4.38
1.21
1.00
1.05
1.00
1.02
1.01
3.06
2.96
3.00
3.01
f1 (ppm)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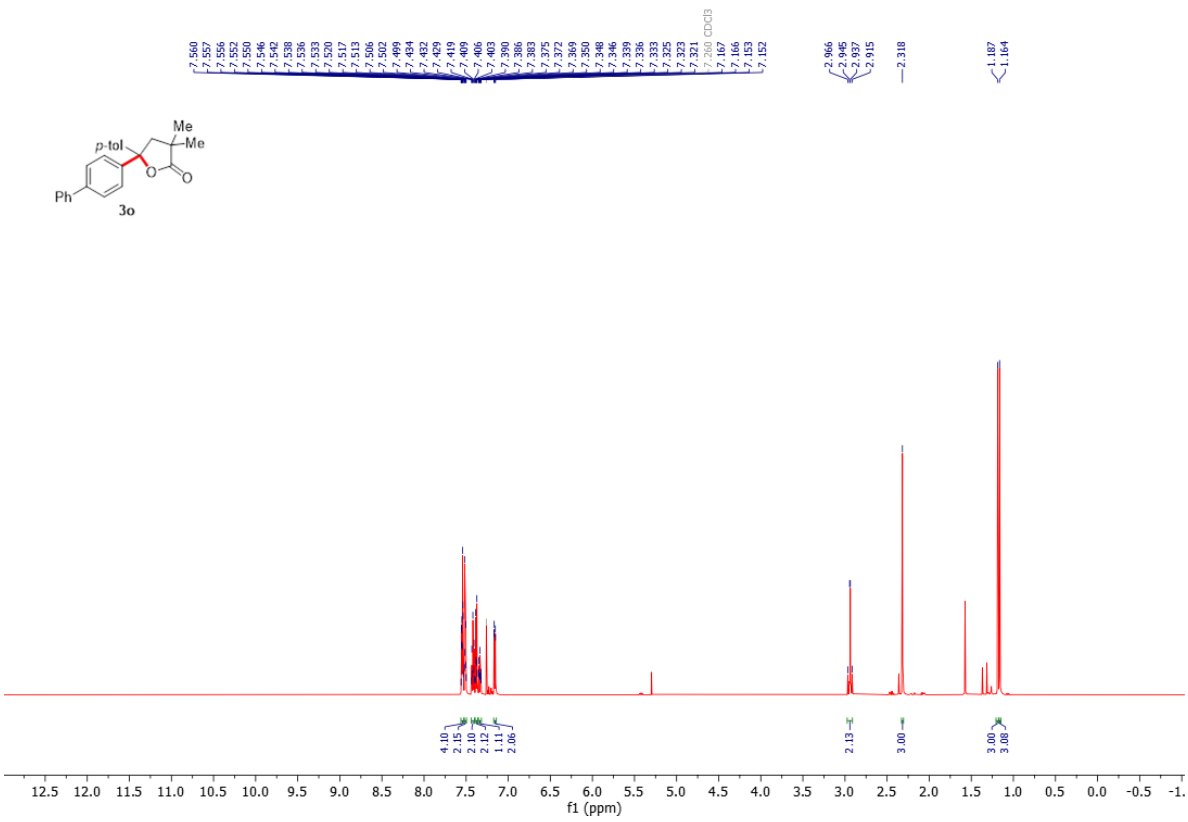
149.539
140.444
140.116
128.838
127.567
127.233
127.108
125.323
86.237
77.160 CDCl3
53.031
51.414
40.119
26.783
26.166
24.676
24.274
23.677
Me
Me
Me
Me
O
O
Ph
3n
f1 (ppm)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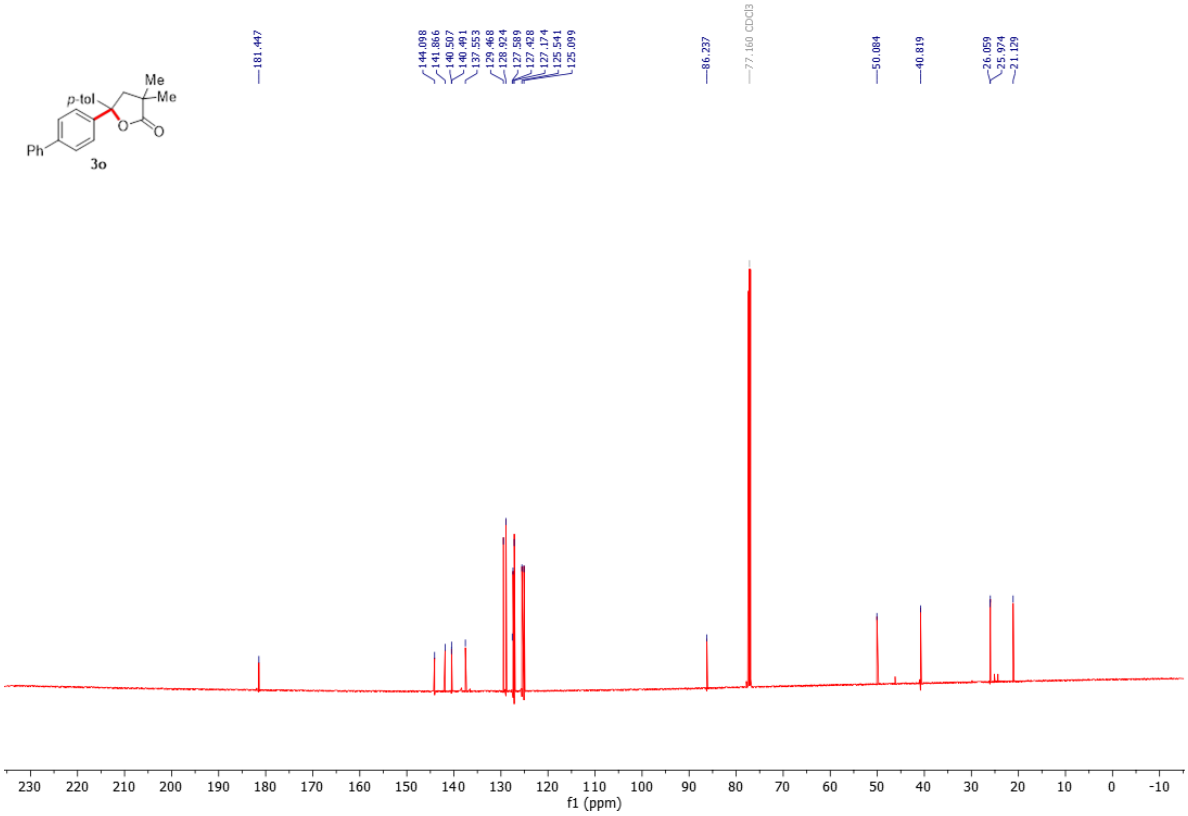
7.557
7.556
7.553
7.550
7.546
7.542
7.538
7.536
7.533
7.520
7.517
7.513
7.506
7.502
7.499
7.434
7.432
7.429
7.419
7.409
7.406
7.403
7.390
7.386
7.383
7.375
7.372
7.369
7.350
7.348
7.346
7.339
7.336
7.333
7.325
7.323
7.321
7.260 CDCl3
7.167
7.166
7.153
7.152
2.966
2.945
2.937
2.915
2.318
1.187
1.164
Me
Me
p-tol
O
O
Ph
3o
4.10
2.15
2.10
2.12
1.11
2.06
2.13
3.00
3.00
3.08
12.5 12.0 11.5 11.0 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.
f1 (ppm)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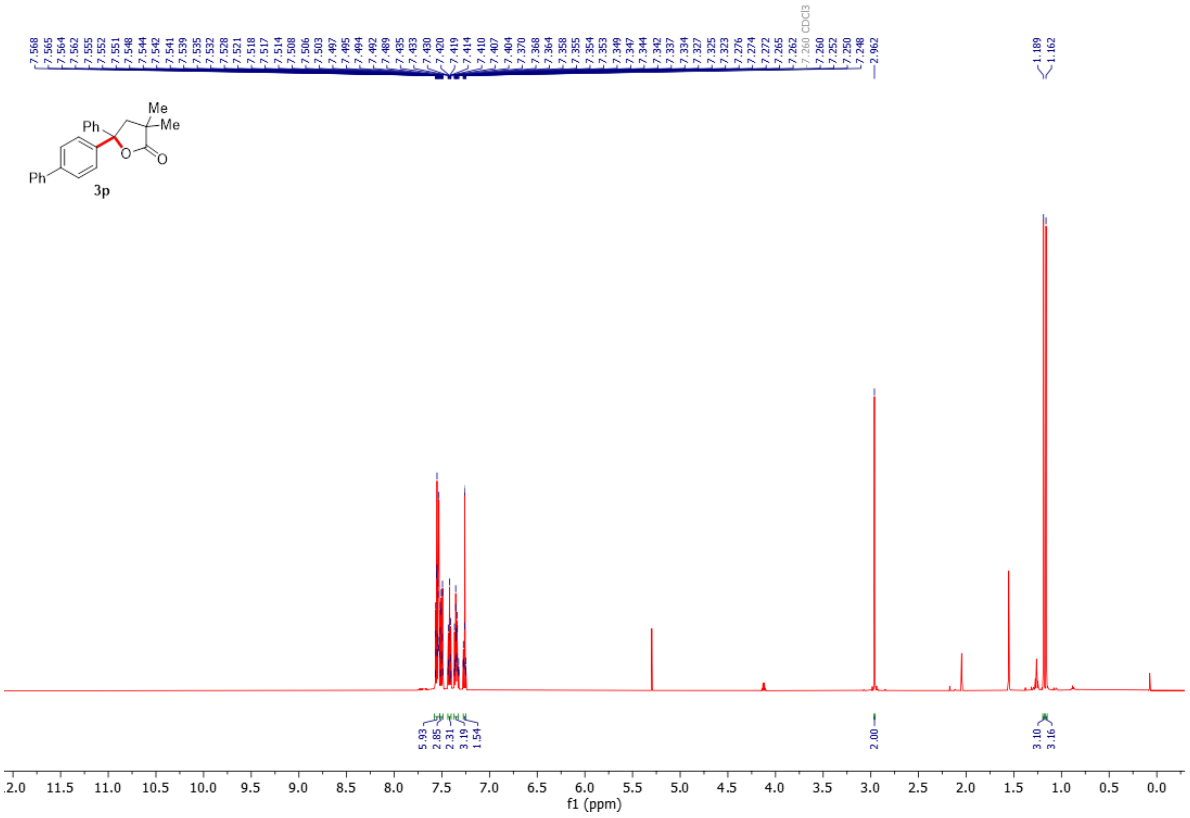
144.098
141.886
140.507
140.491
137.553
129.468
128.924
127.589
127.428
127.174
125.541
125.099
86.237
77.160 CDCl3
50.084
40.819
26.059
25.974
21.129
Me
Me
p-tol
O
O
Ph
3o
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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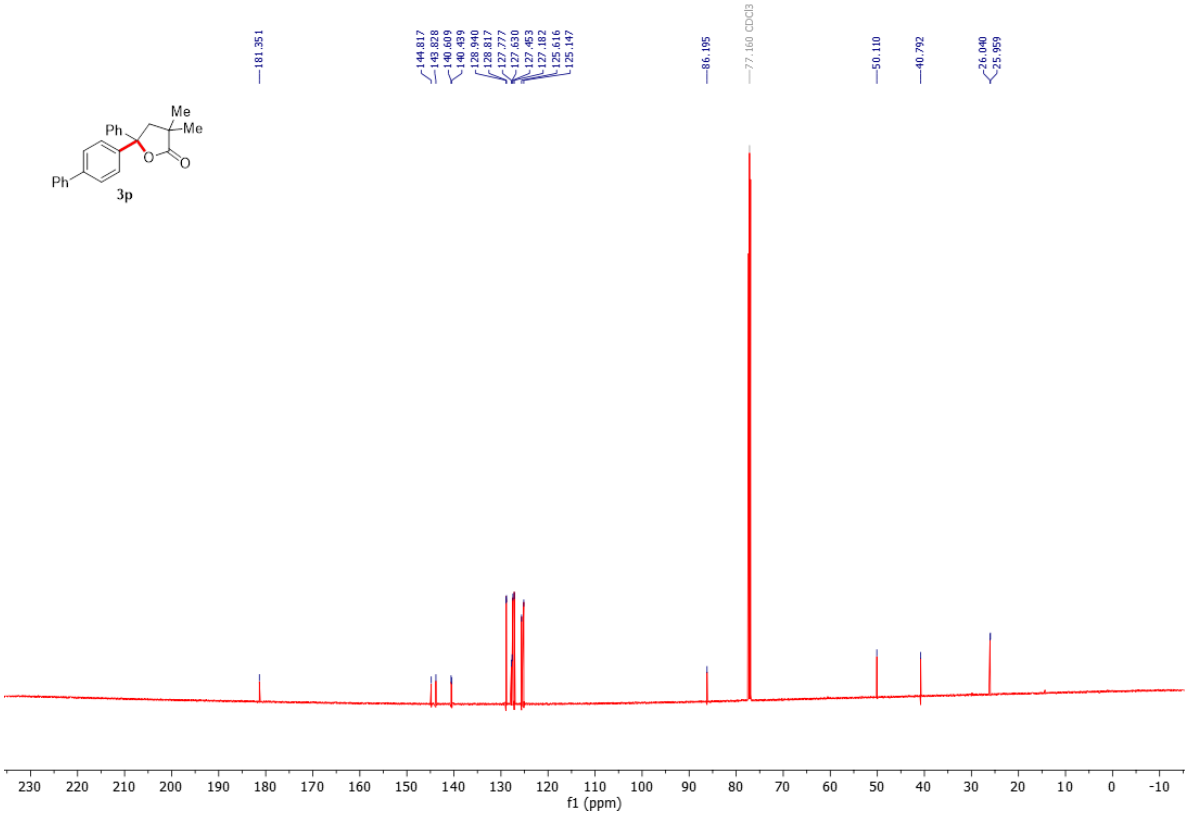
7.565
7.564
7.562
7.555
7.552
7.551
7.548
7.544
7.542
7.541
7.539
7.535
7.532
7.528
7.521
7.518
7.517
7.514
7.508
7.506
7.503
7.497
7.495
7.494
7.492
7.489
7.435
7.433
7.430
7.420
7.419
7.414
7.410
7.407
7.404
7.370
7.368
7.364
7.358
7.355
7.354
7.353
7.349
7.347
7.344
7.342
7.337
7.334
7.327
7.325
7.323
7.276
7.274
7.272
7.265
7.262
7.260 CDCl3
7.252
7.250
7.248
2.962
1.189
1.162
Me
Me
Ph
O
O
Ph
3p
5.93
2.85
2.31
3.19
1.54
2.00
3.10
3.16
2.0 11.5 11.0 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
f1 (ppm)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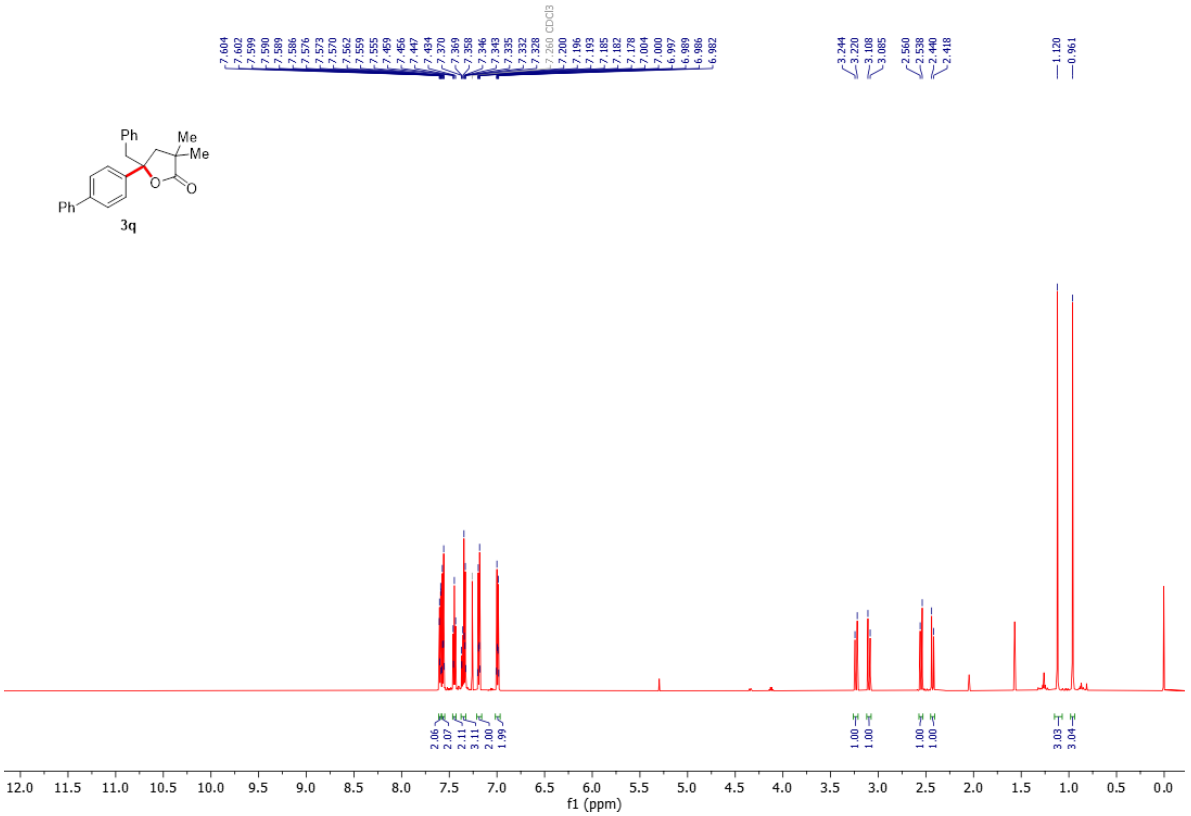
144.817
143.828
140.609
140.439
128.940
128.817
127.777
127.630
127.453
127.102
125.616
125.147
86.195
77.160 CDCl3
50.110
40.792
26.040
25.959
Me
Me
Ph
O
O
Ph
3p
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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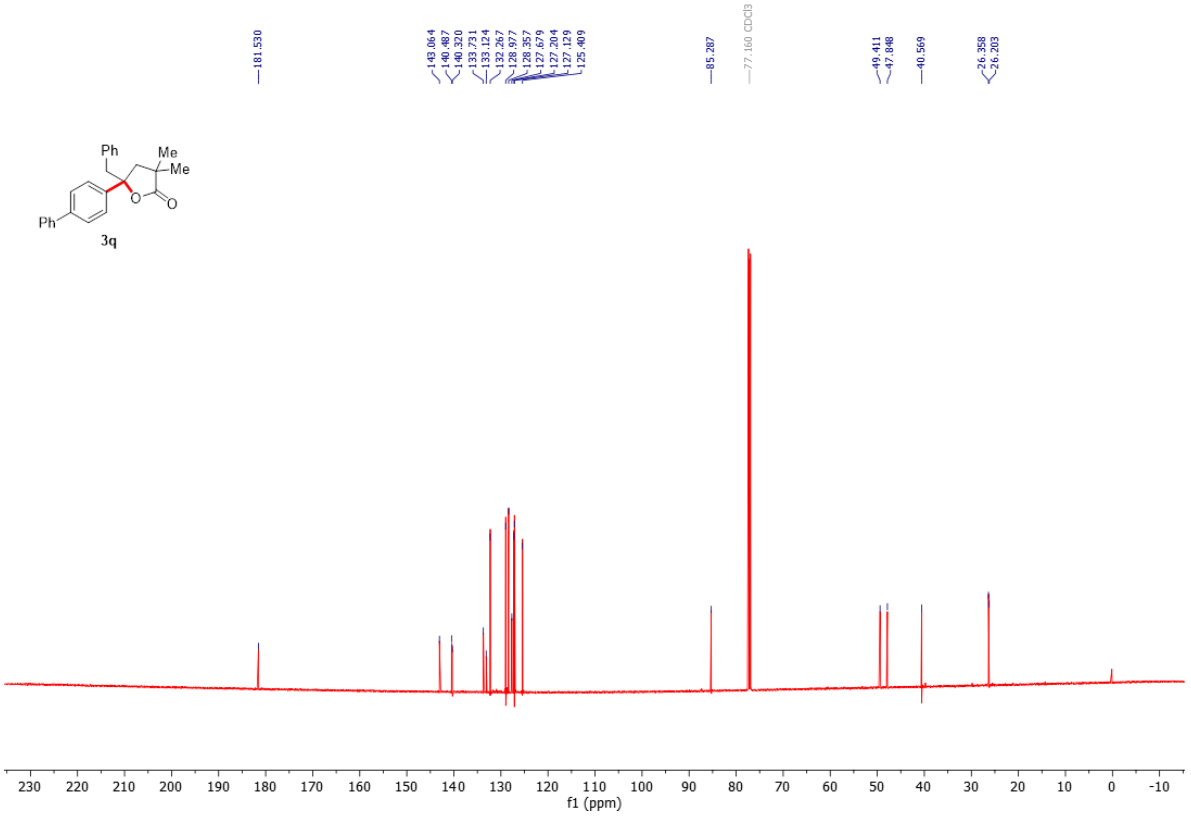
7.602
7.599
7.590
7.589
7.586
7.576
7.573
7.570
7.562
7.559
7.555
7.469
7.466
7.447
7.434
7.370
7.369
7.358
7.346
7.343
7.335
7.332
7.328
7.260 CDCl3
7.200
7.196
7.193
7.185
7.182
7.178
7.004
7.000
6.997
6.989
6.986
6.982
3.244
3.220
3.108
3.085
2.560
2.538
2.440
2.418
1.120
0.961
Ph
Me
Me
O
O
Ph
3q
2.06
2.07
2.11
3.11
2.00
1.99
1.00
1.00
1.00
1.00
3.03
3.04
12.0
11.5
11.0
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
f1 (ppm)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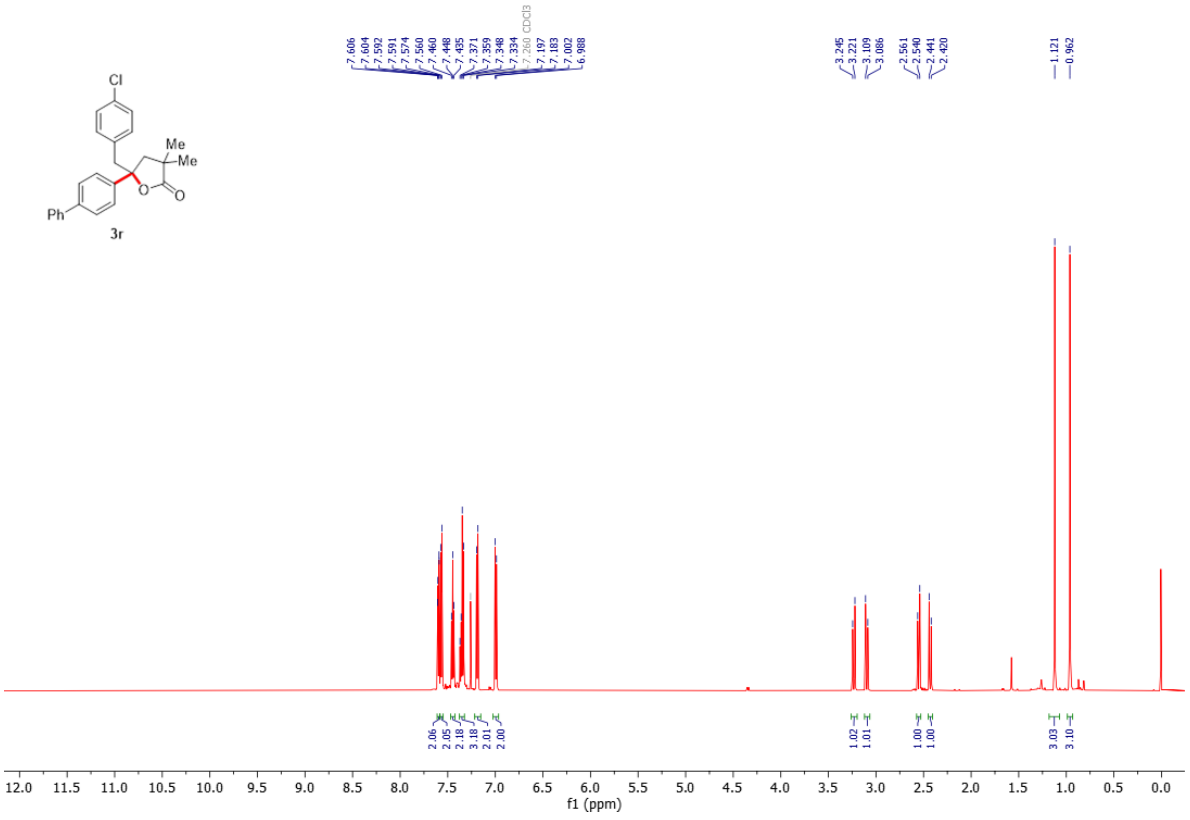
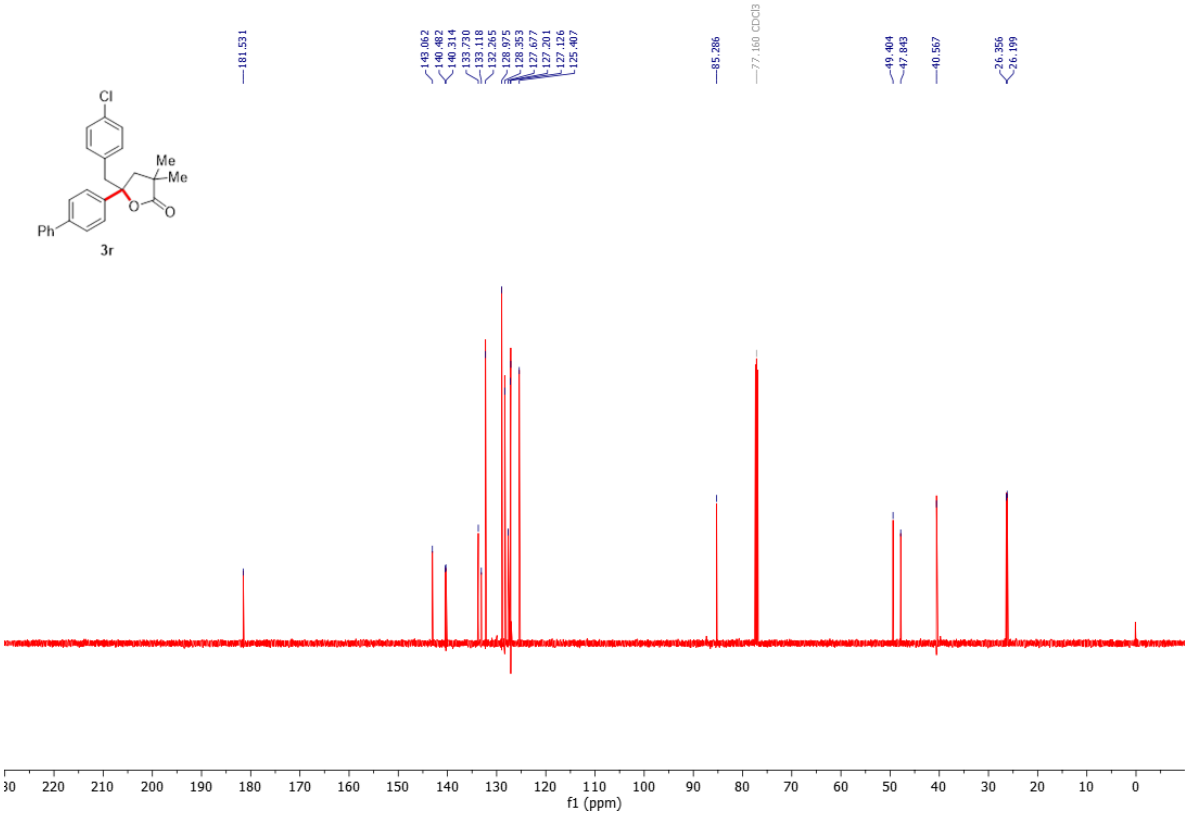
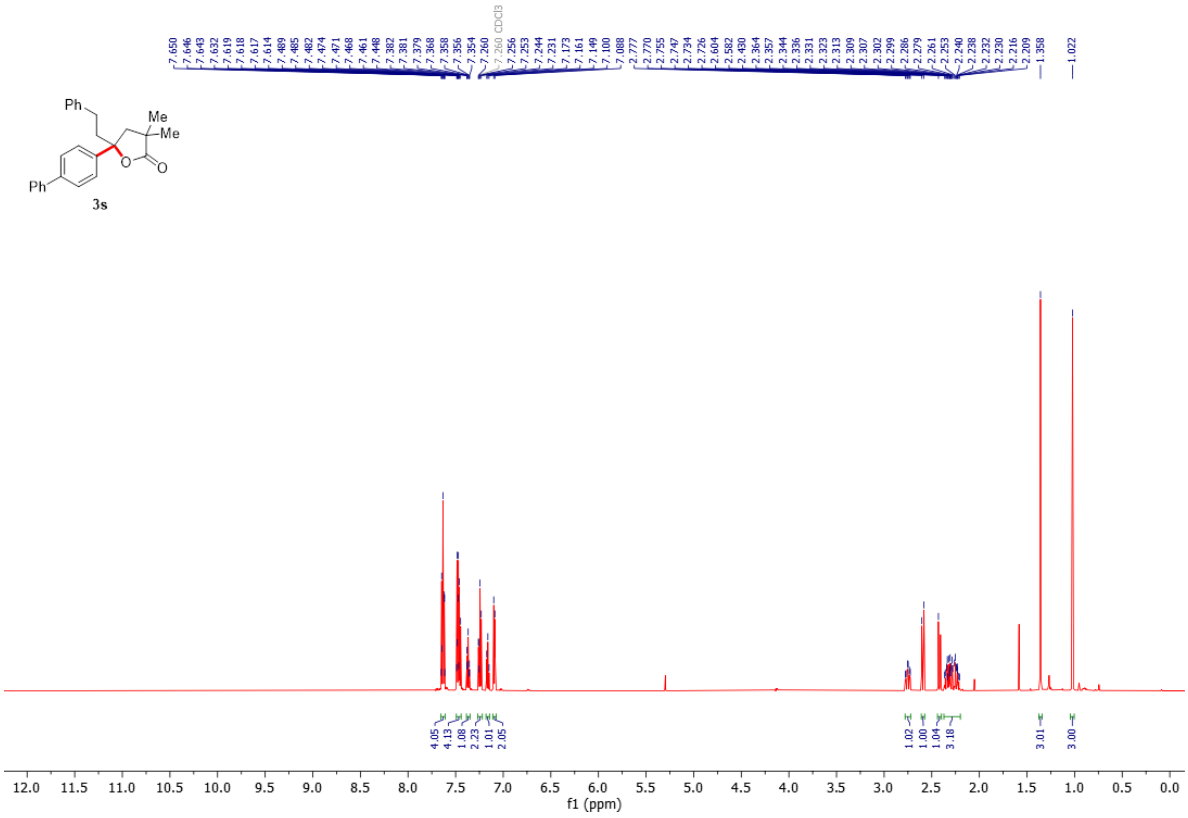
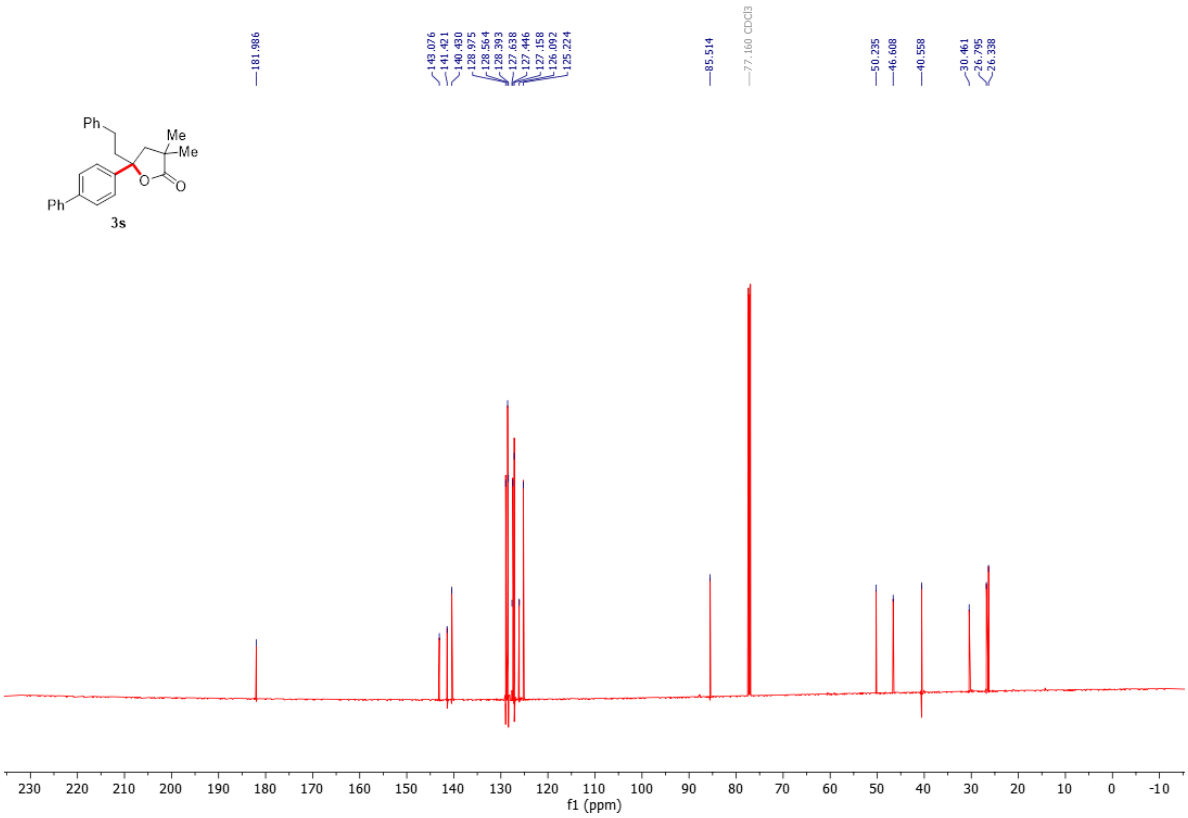
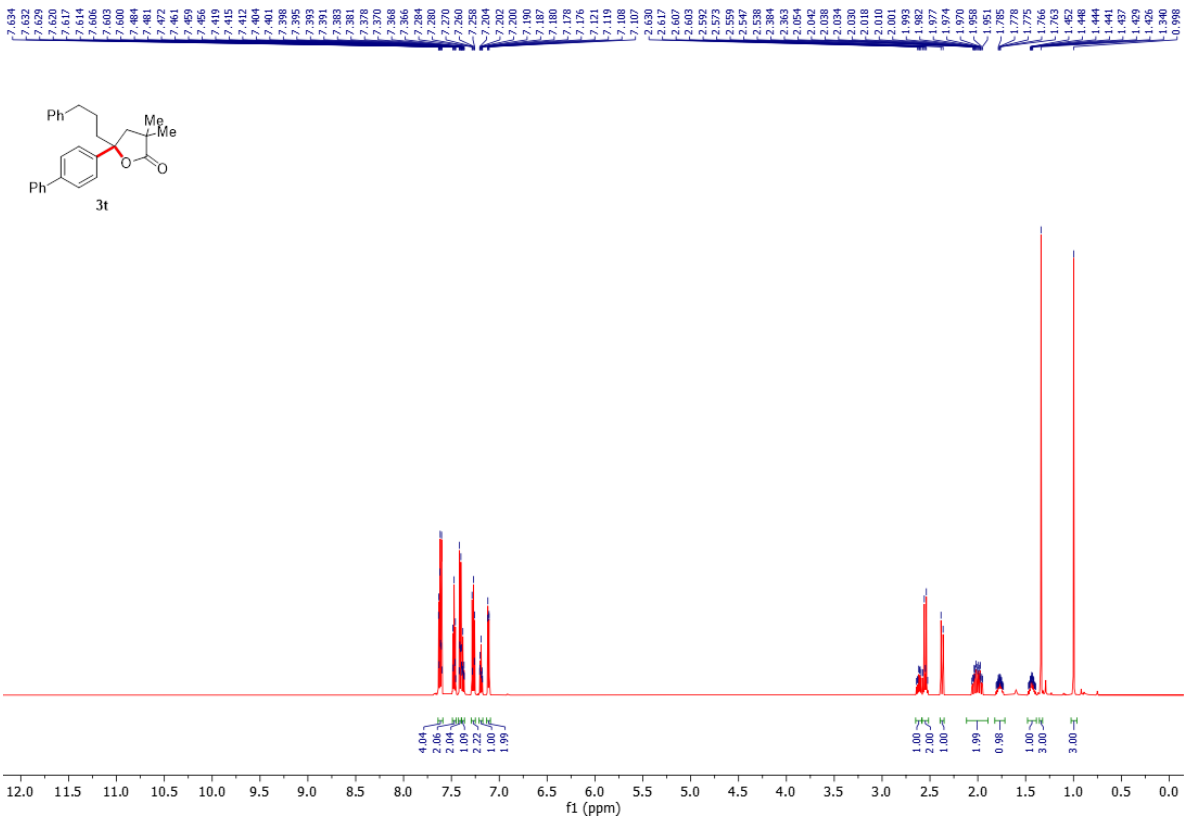
140.487
140.320
133.731
132.264
128.977
128.357
127.679
127.204
127.129
125.409
85.287
77.160 CDCl3
49.411
47.848
40.569
26.358
26.203
Ph
Me
Me
O
O
Ph
3q
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

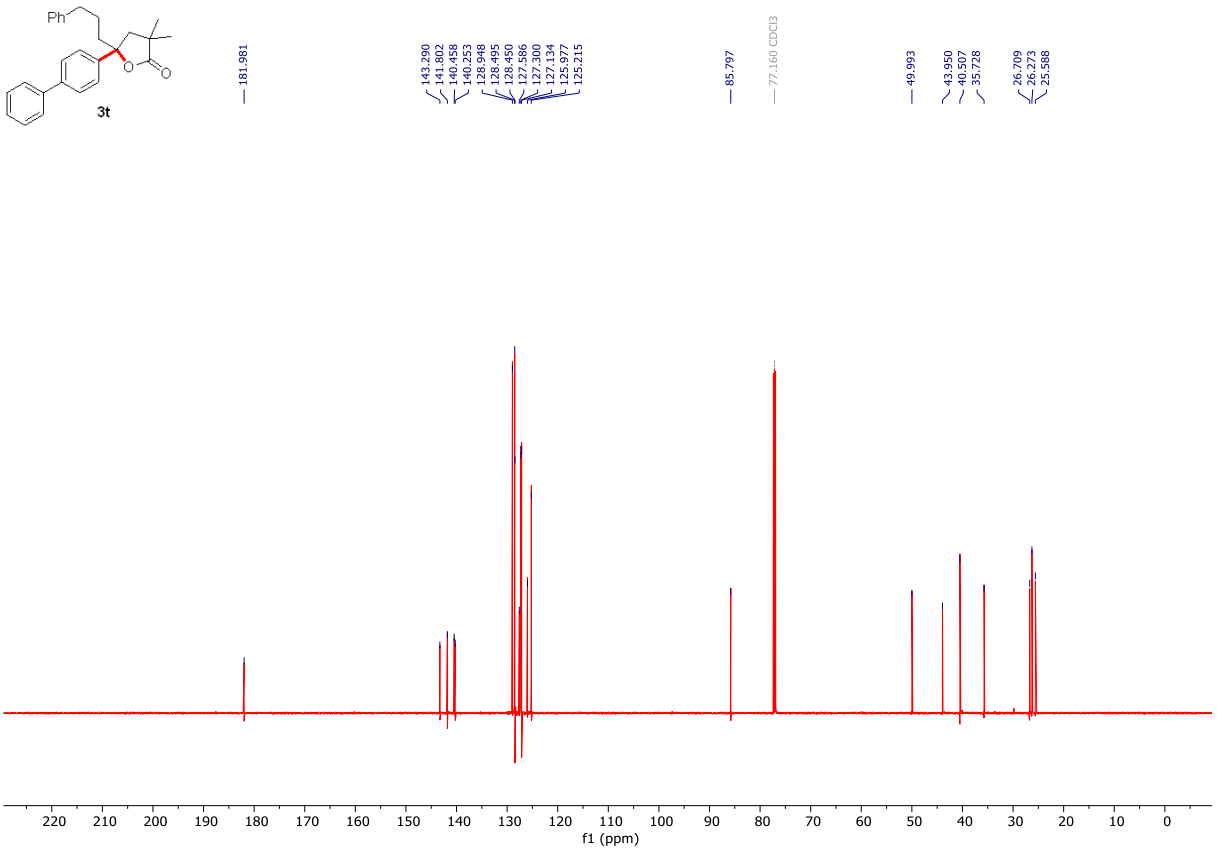

7.606
7.604
7.592
7.591
7.574
7.560
7.460
7.448
7.435
7.371
7.359
7.348
7.334
7.260 CDCl3
7.197
7.183
7.002
6.988
3.245
3.221
3.109
3.086
2.561
2.540
2.441
2.420
1.121
0.962
Cl
Me
Me
O
O
Ph
3r
2.06
2.05
2.18
3.18
2.01
2.00
1.02
1.01
1.00
1.00
3.03
3.10
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 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
f1 (ppm)


181.531
143.062
140.482
140.314
133.730
133.118
132.265
128.975
128.353
127.677
127.201
127.126
125.407
85.286
77.160 CDCl3
49.404
47.843
40.567
26.356
26.199
Cl
Me
Me
O
O
Ph
3r
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Ph
Me
Me
O
O
Ph
3s
f1 (ppm)


Ph
Me
Me
O
O
Ph
3s
f1 (ppm)


Ph
Me
Me
O
O
Ph
3t
f1 (ppm)


Ph
O
O
3t
181.981
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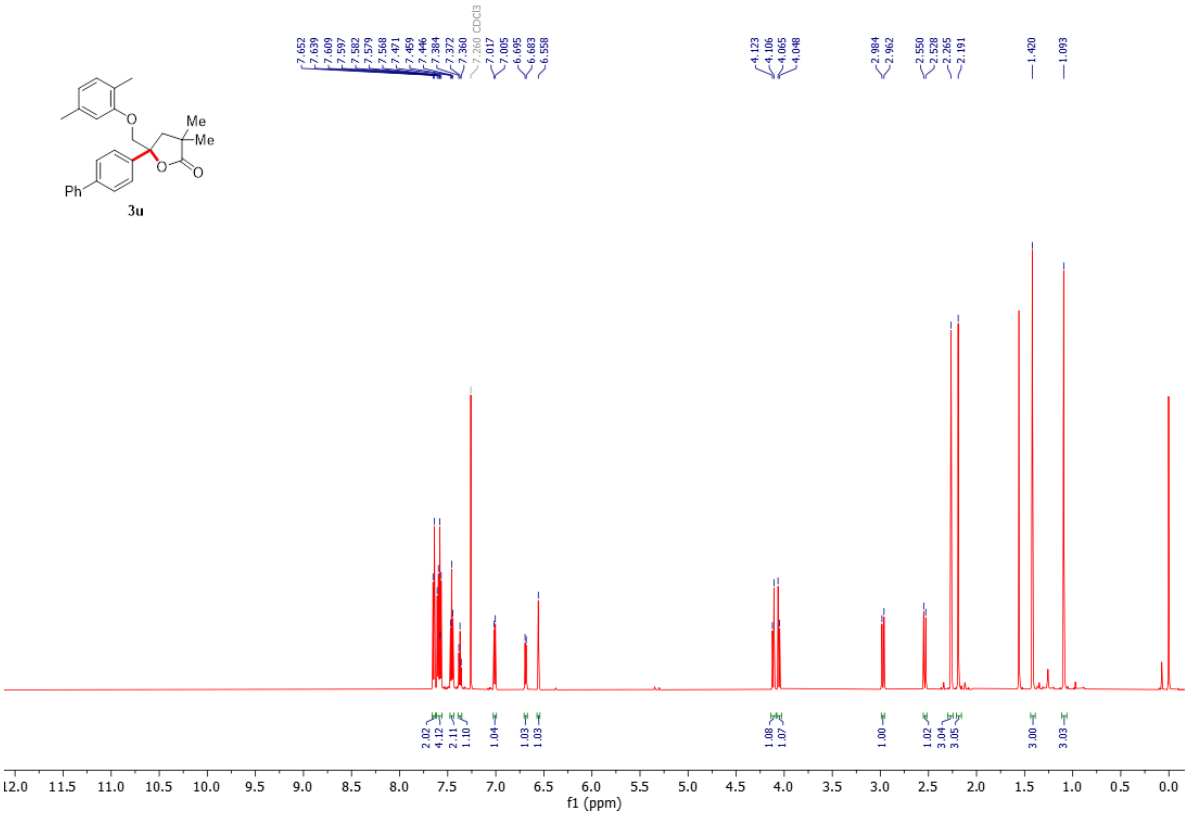
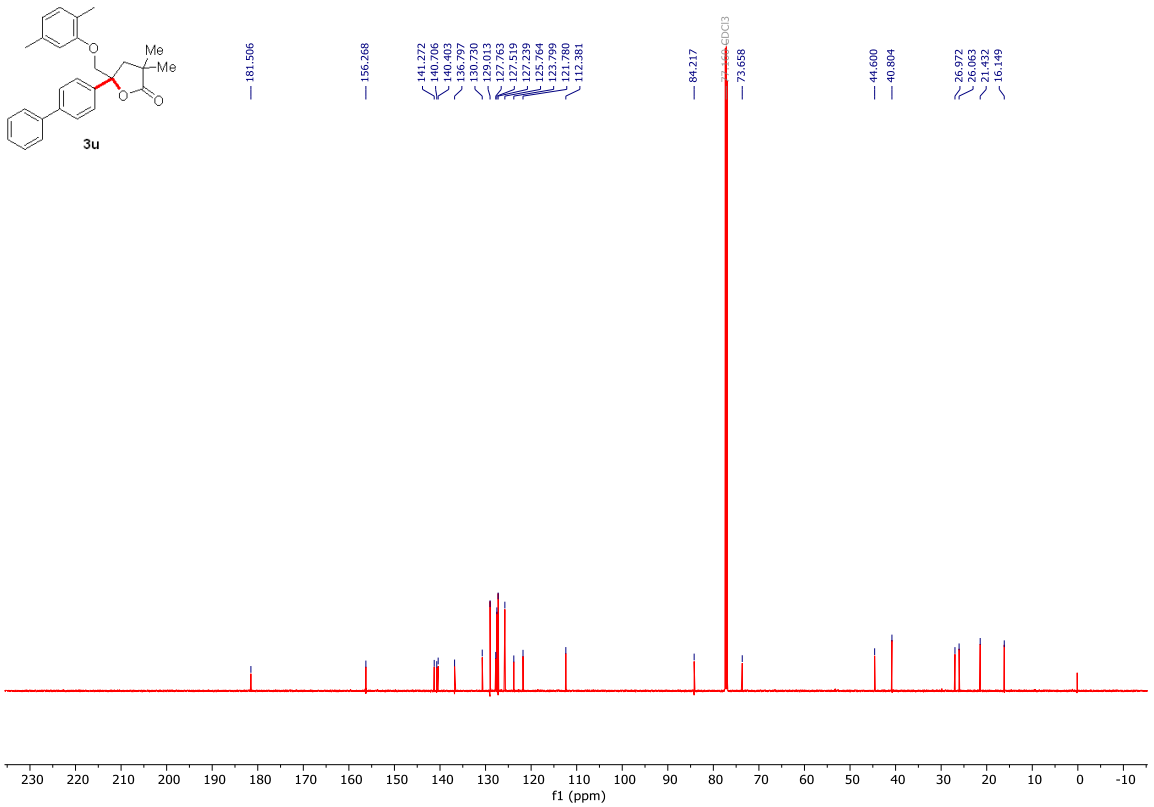
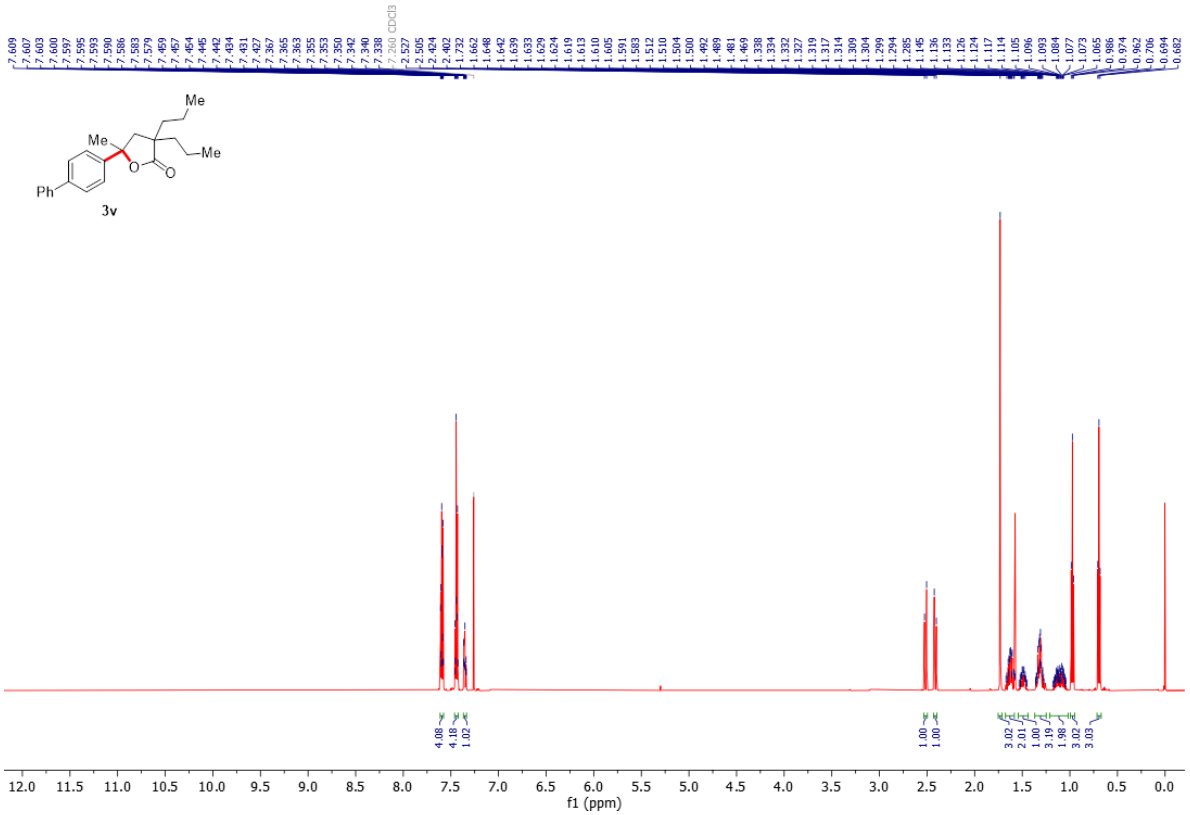
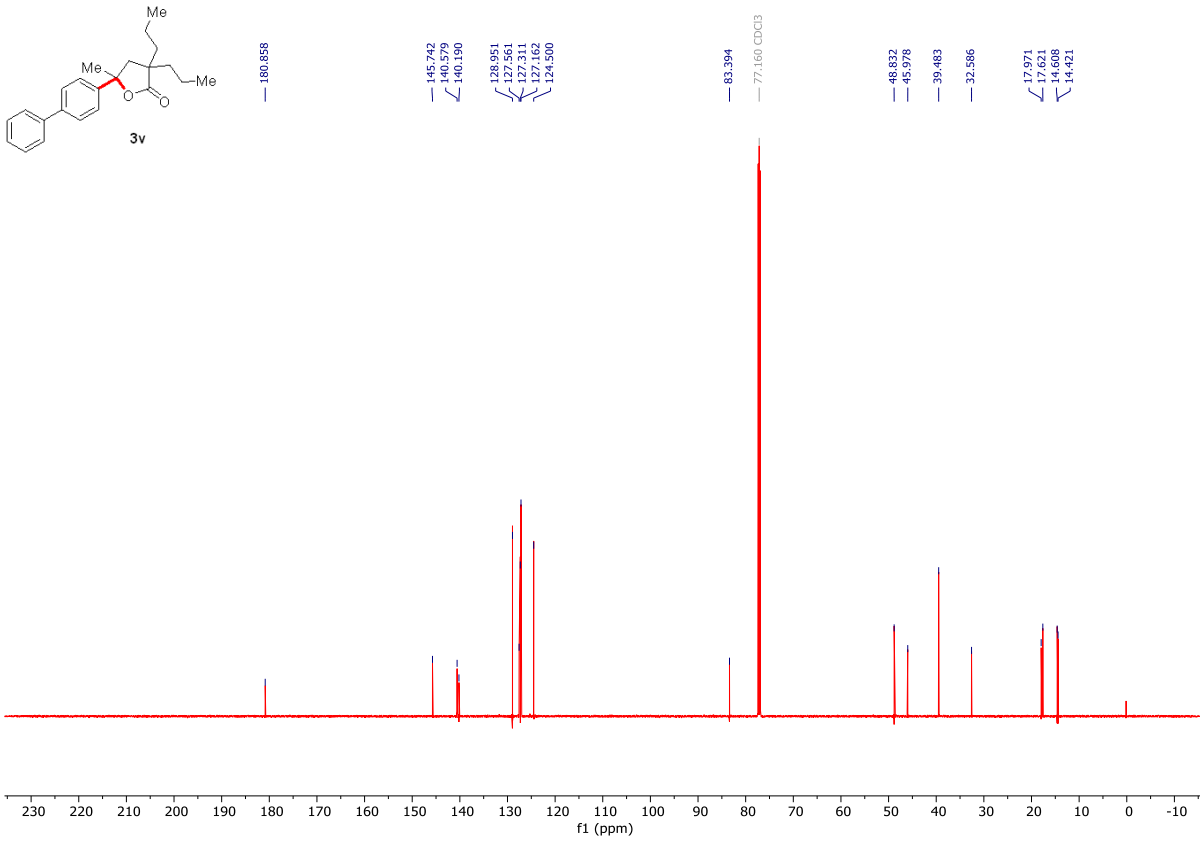
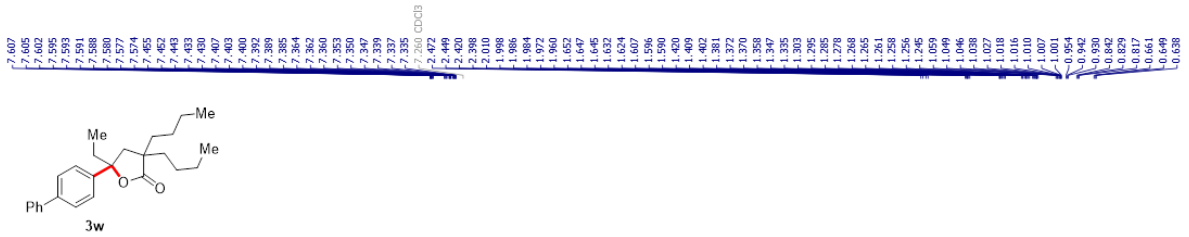
141.802
140.458
140.253
128.948
128.495
128.450
127.586
127.300
127.134
125.977
125.215
85.797
77.160 CDCl3
49.993
43.950
40.507
35.728
26.709
26.273
25.588
f1 (ppm)


Me
Me
O
O
O
Ph
3u
7.652
7.639
7.609
7.597
7.582
7.579
7.568
7.491
7.459
7.446
7.384
7.372
7.360
7.260 CDCl3
7.017
7.005
6.695
6.683
6.558
4.123
4.106
4.065
4.048
2.984
2.962
2.550
2.528
2.265
2.191
1.420
1.093
2.02
4.12
2.11
1.10
1.04
1.03
1.03
1.08
1.07
1.00
1.02
3.04
3.05
3.00
3.03
f1 (ppm)


Me
Me
O
O
O
3u
181.506
156.268
141.272
140.706
140.403
136.797
130.730
129.013
127.753
127.519
127.239
125.764
123.799
121.780
112.381
84.217
77.260 CDCl3
73.658
44.600
40.804
26.972
26.063
21.432
16.149
f1 (ppm)

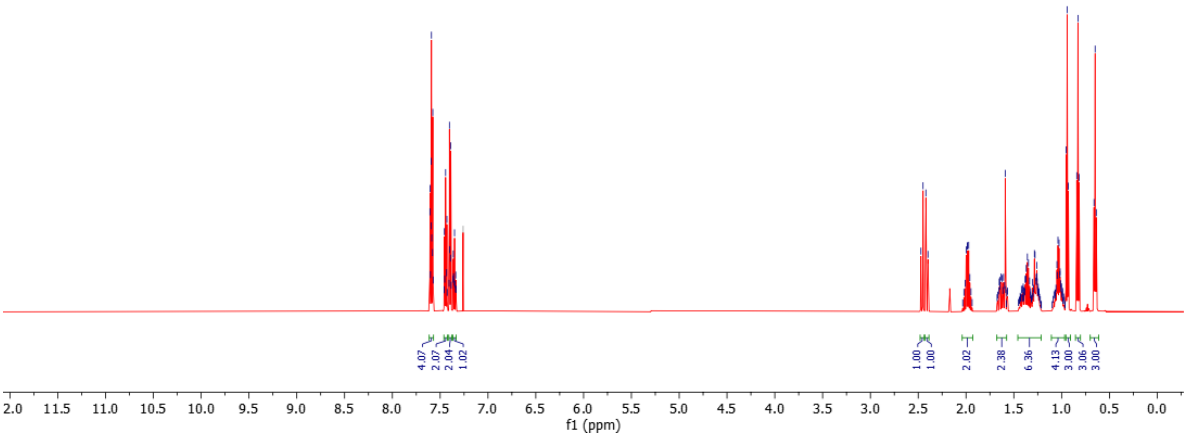

Me
Me
Me
O
O
Ph
3v
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

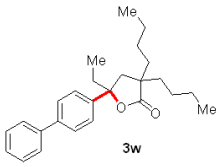

Me
Me
Me
O
O
3v
180.858
145.742
140.579
140.190
128.951
127.561
127.311
127.162
124.500
83.394
77.160 CDCl3
48.832
45.978
39.483
32.586
17.971
17.621
14.608
14.421
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.607 7.605 7.602 7.595 7.593 7.591 7.588 7.580 7.577 7.574 7.455 7.452 7.443 7.433 7.430 7.407 7.403 7.400 7.392 7.389 7.385 7.364 7.362 7.360 7.353 7.350 7.347 7.339 7.337 7.335 7.260 CDCl3 2.472 2.449 2.420 2.398 2.010 1.998 1.986 1.984 1.972 1.962 1.652 1.647 1.645 1.632 1.624 1.607 1.596 1.590 1.420 1.409 1.402 1.381 1.372 1.370 1.358 1.347 1.335 1.303 1.295 1.285 1.278 1.268 1.265 1.261 1.258 1.256 1.245 1.059 1.046 1.038 1.027 1.018 1.016 1.010 1.007 1.001 0.954 0.942 0.930 0.842 0.829 0.817 0.661 0.649 0.638

Me
Me
Me
O
O
Ph

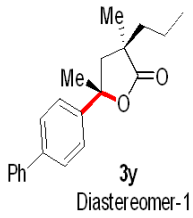
3w


4.07
2.07
2.04
1.02
1.00
1.00
2.02
2.38
6.36
4.13
3.00
3.06
3.00
2.0 11.5 11.0 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
f1 (ppm)


Me
Me
Me
O
O

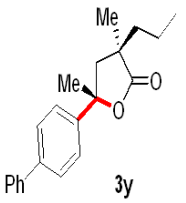
3w

181.067
143.785
140.646
140.082
128.934
127.512
127.139
127.102
125.315
86.217
77.160 CDCl3
48.186
44.767
37.740
36.794
36.502
26.799
26.358
23.266
22.846
14.134
13.738
8.548
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
f1 (ppm)



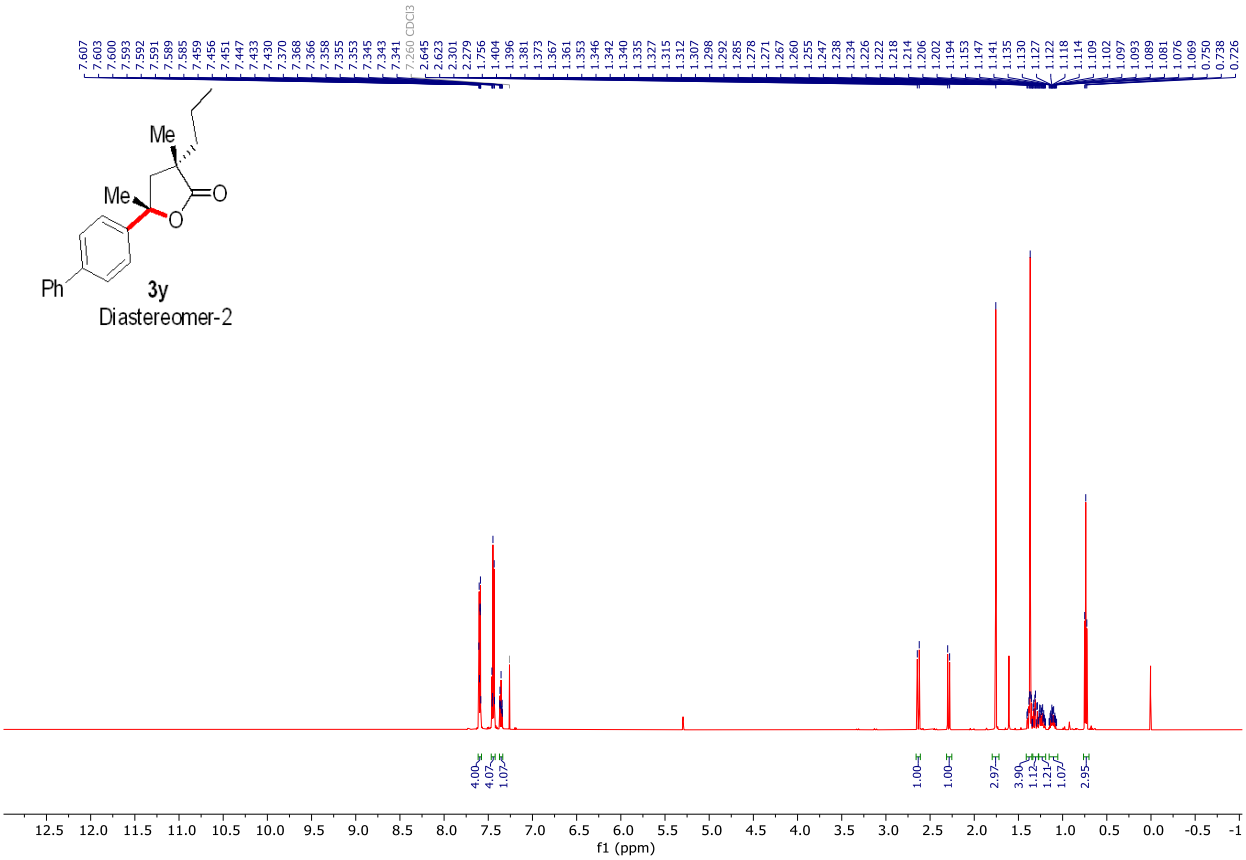
3y

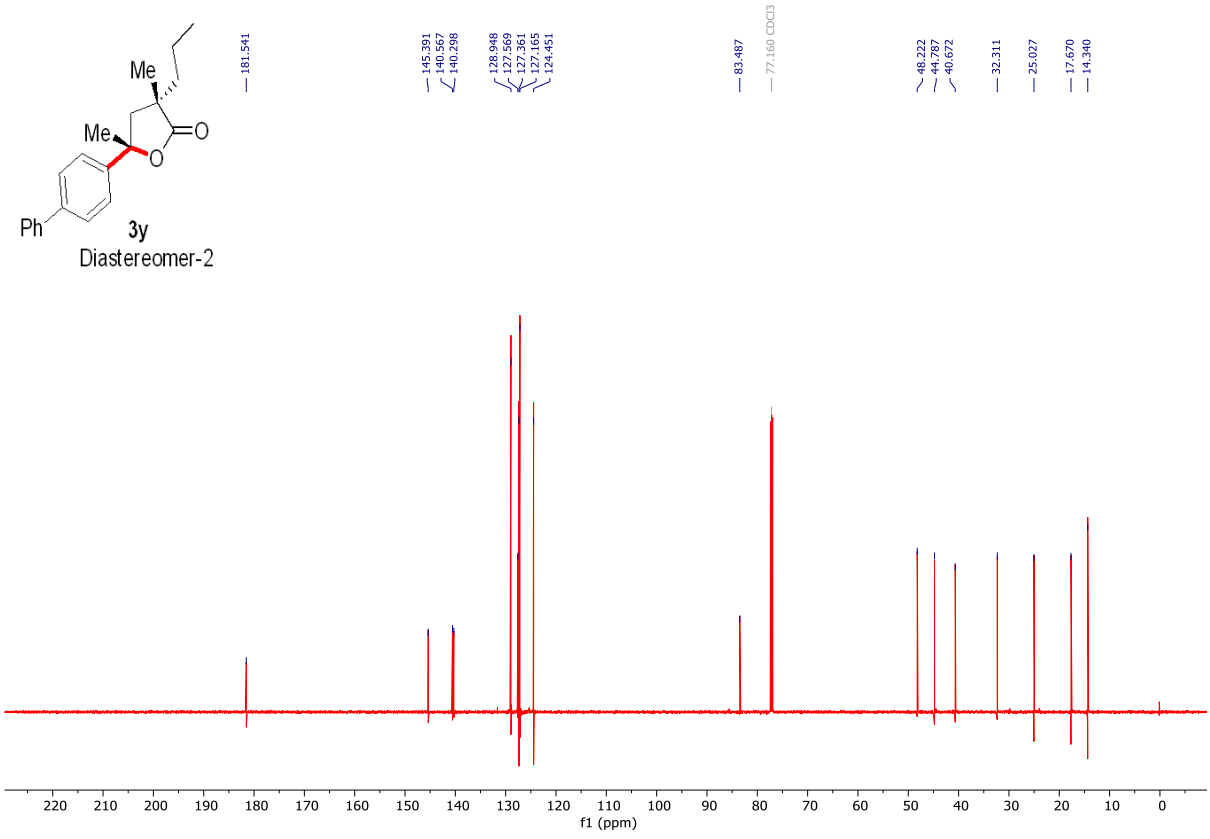
Diastereomer-1

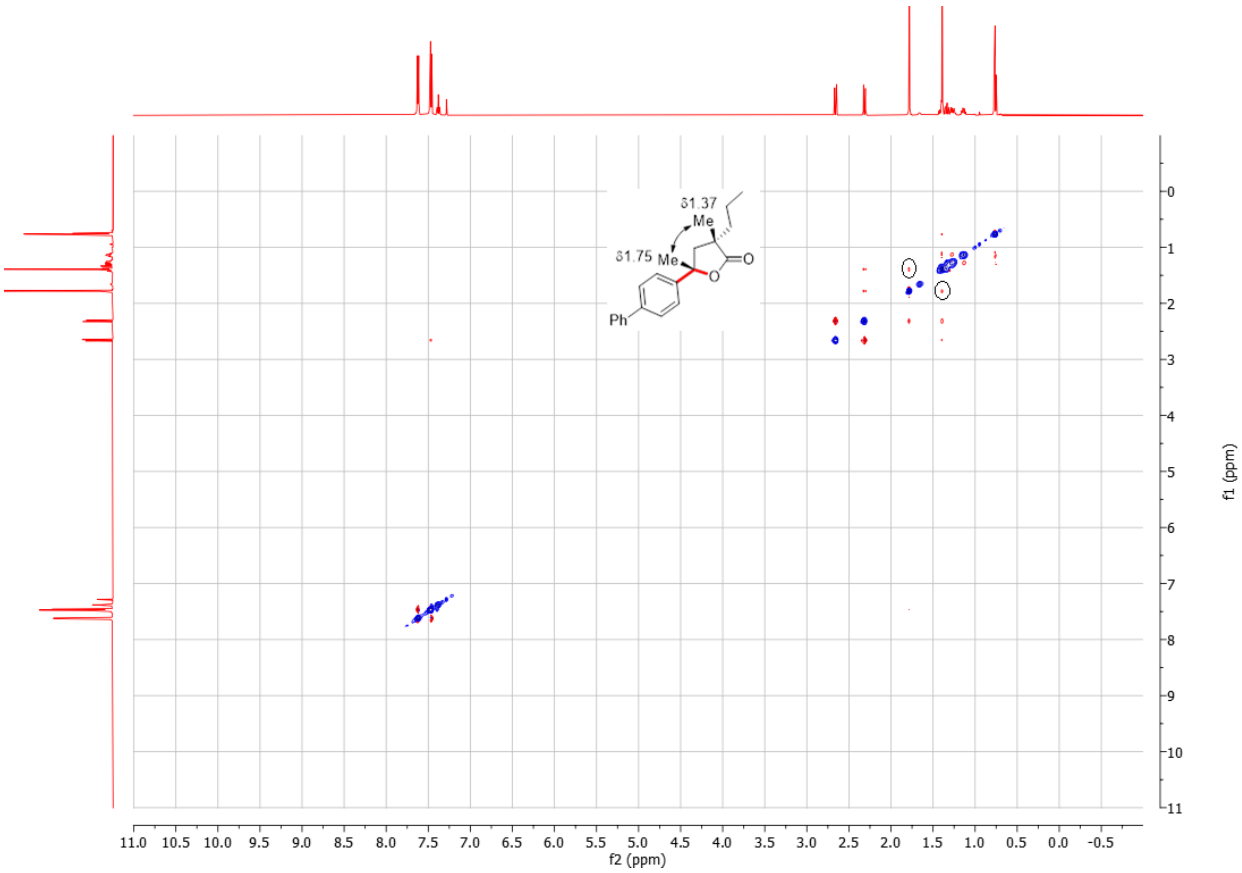


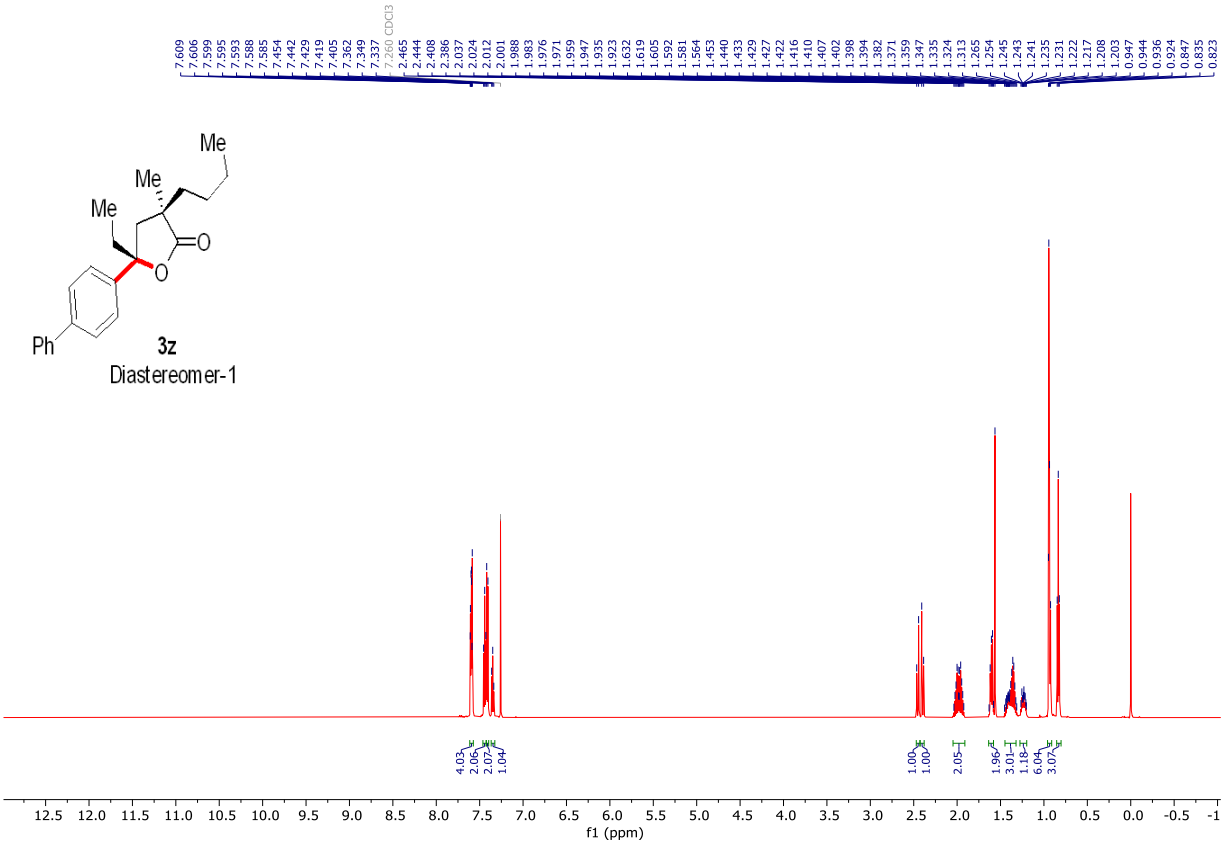
3y

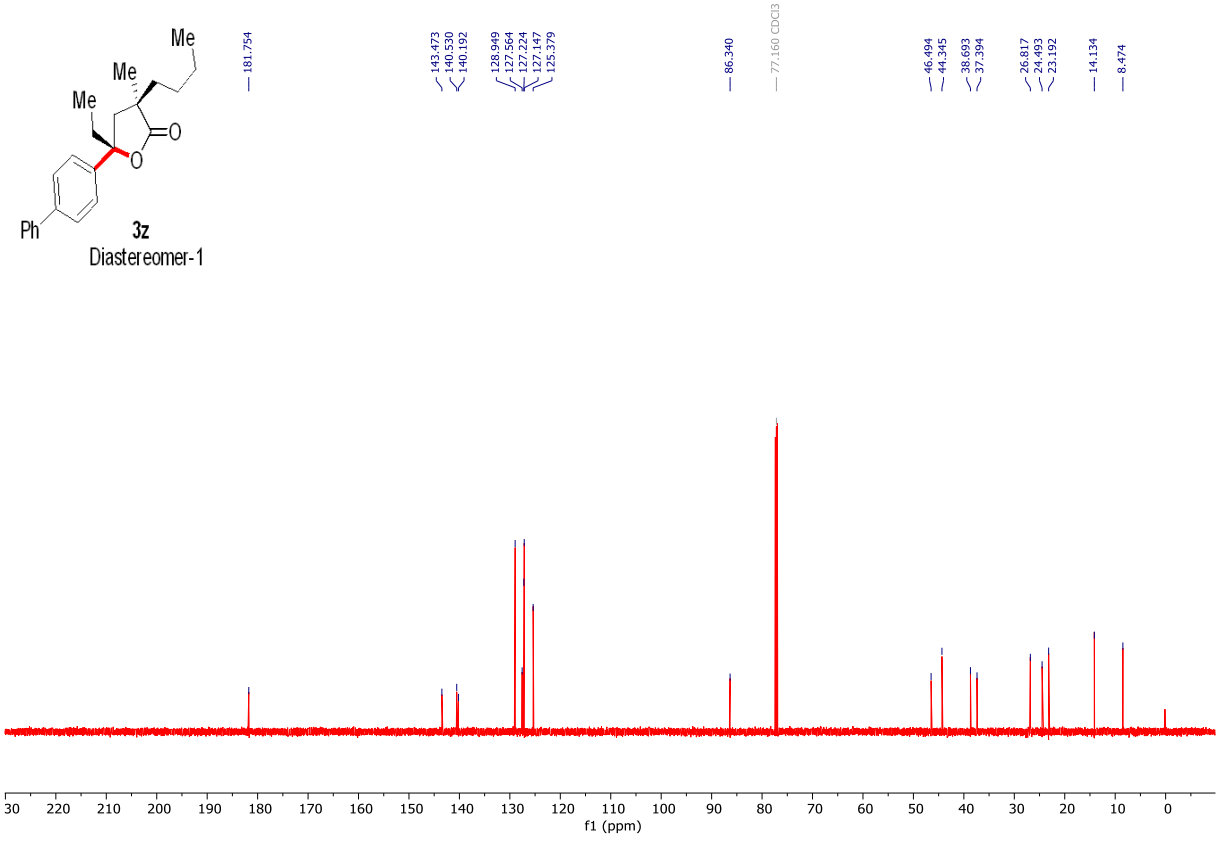
Diastereomer-1

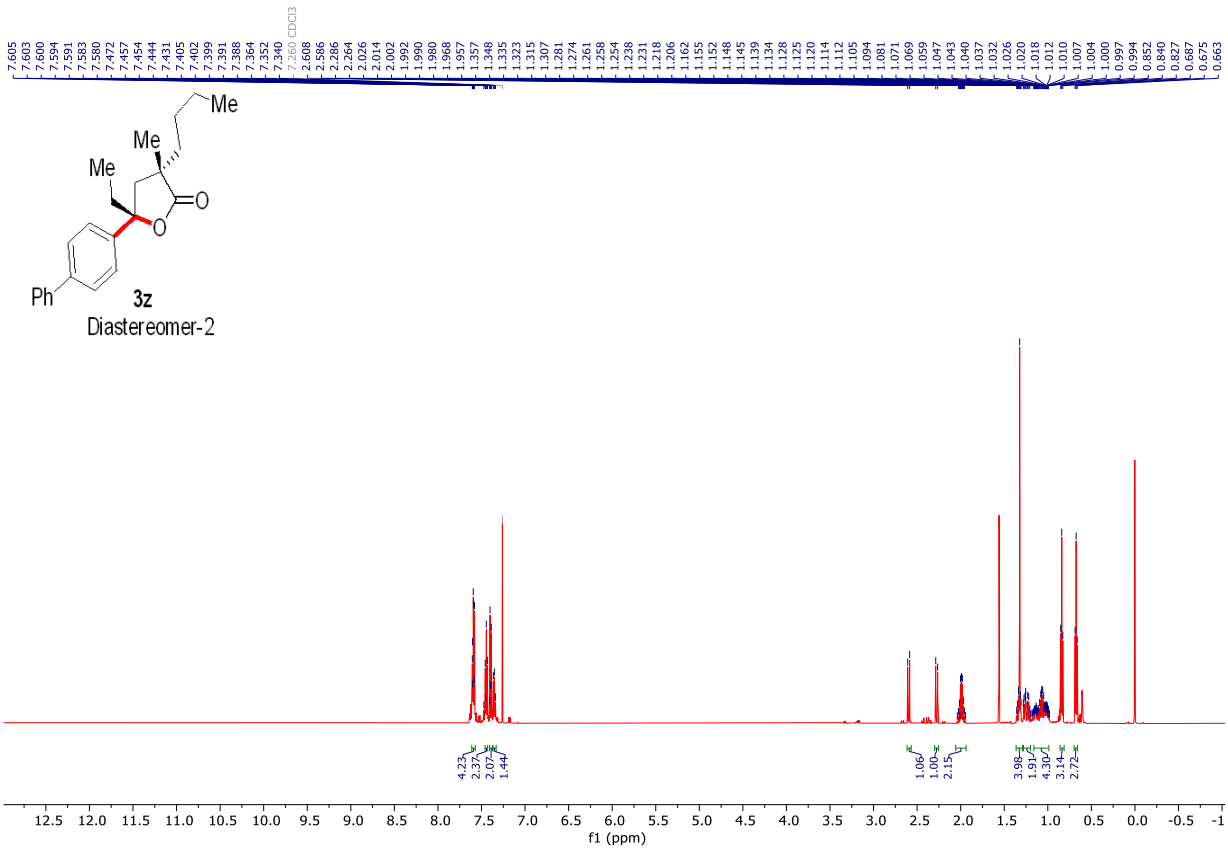

Me
Me
O
O
Ph
3y
Diastereomer-2
f1 (ppm)

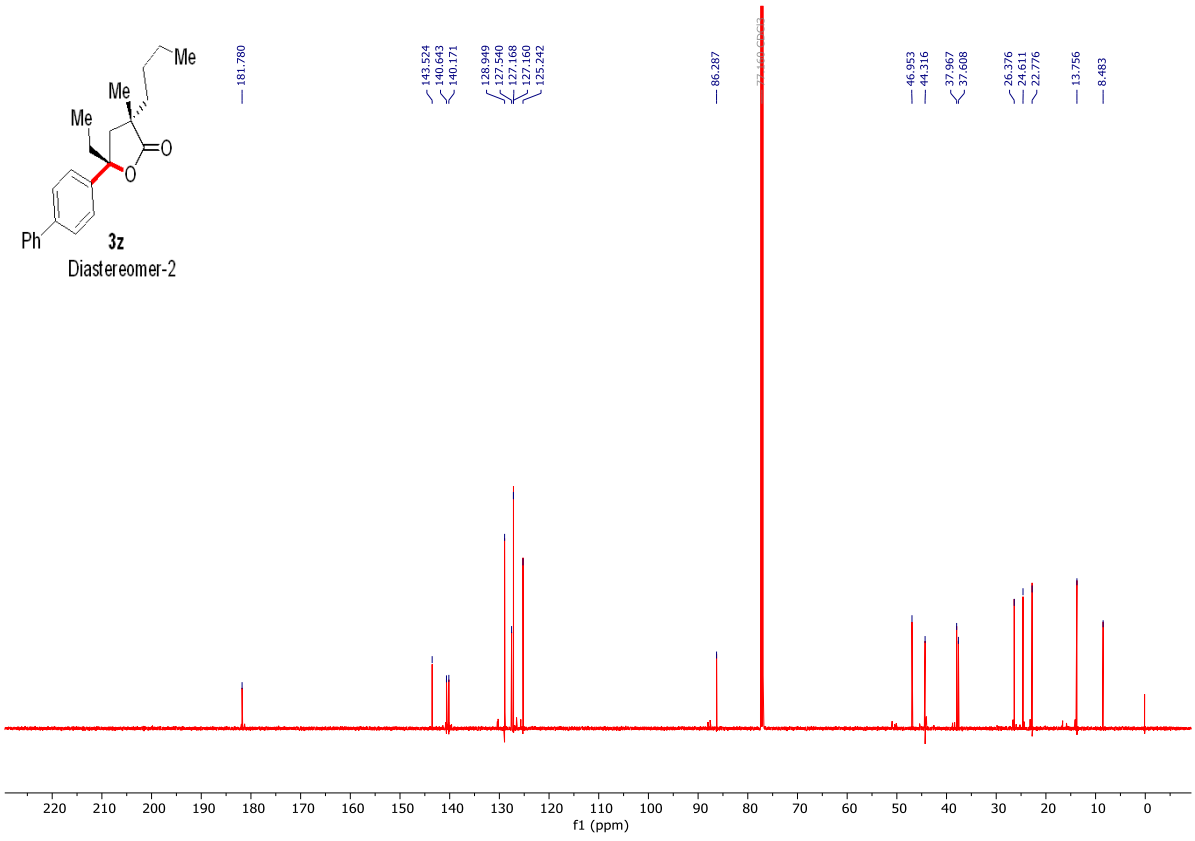

Me
Me
O
O
Ph
3y
Diastereomer-2
f1 (ppm)

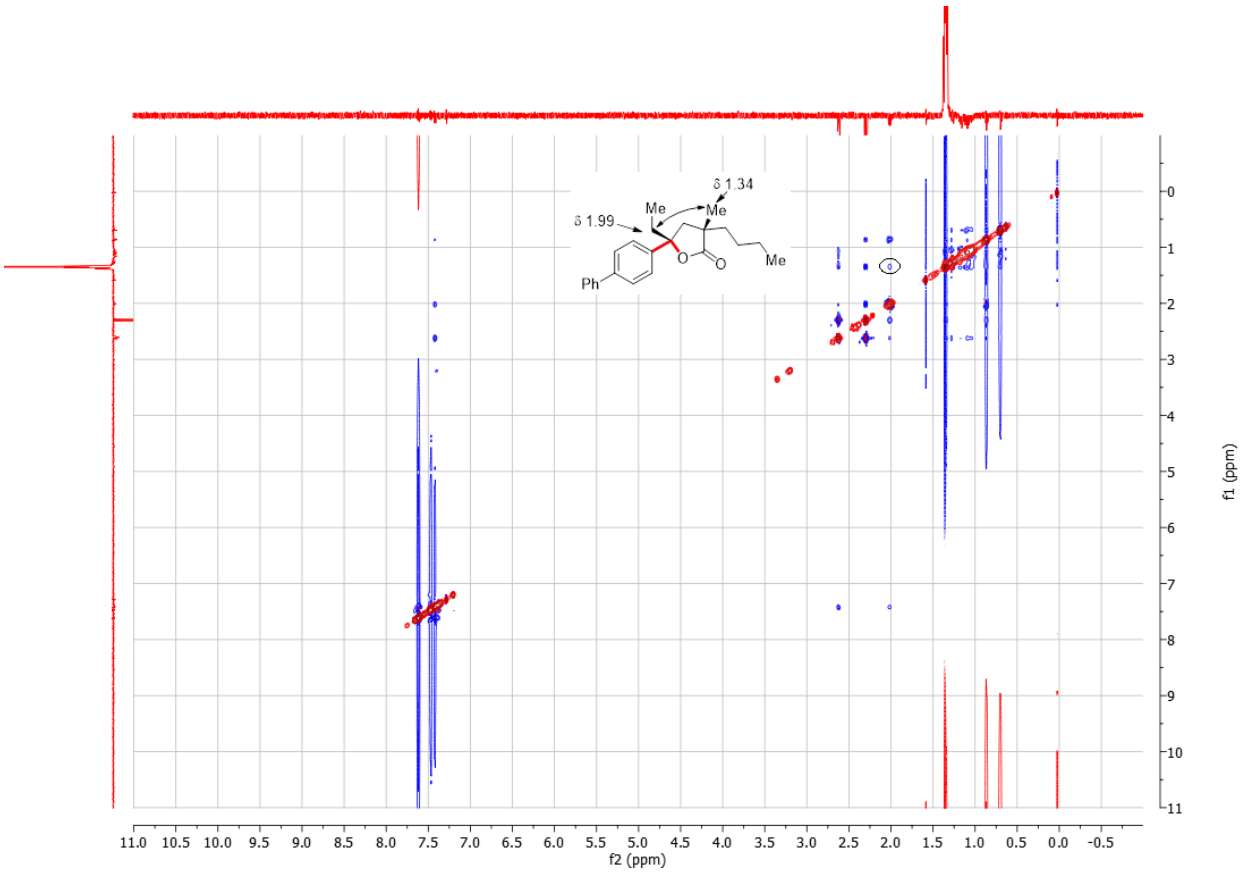

δ1.37
Me
δ1.75
Me
O
O
Ph
f1 (ppm)
f2 (ppm)

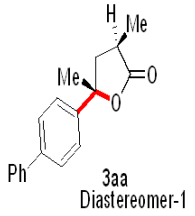

Me
Me
Me
O
O
Ph
3z
Diastereomer-1
f1 (ppm)


Me
Me
Me
O
O
Ph
3z
Diastereomer-1
f1 (ppm)

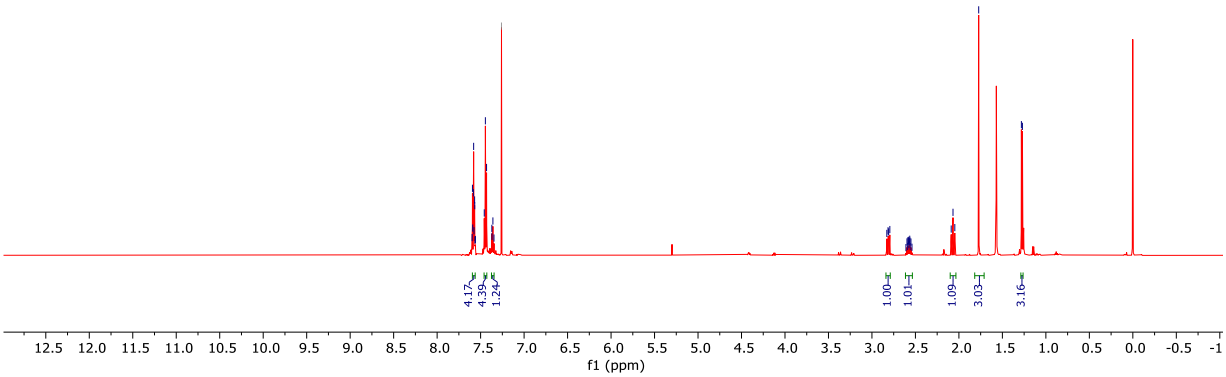

Me
Me
Me
O
O
Ph
3z
Diastereomer-2
f1 (ppm)

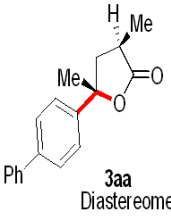

Me
Me
Me
O
O
Ph
3z
Diastereomer-2
f1 (ppm)


δ 1.34
Me
Me
δ 1.99
Ph
O
O
Me
f2 (ppm)
f1 (ppm)

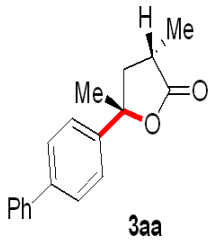


3aa
Diastereomer-1



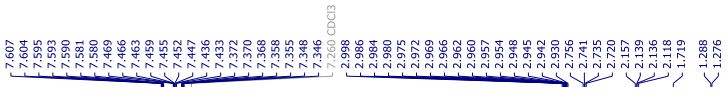


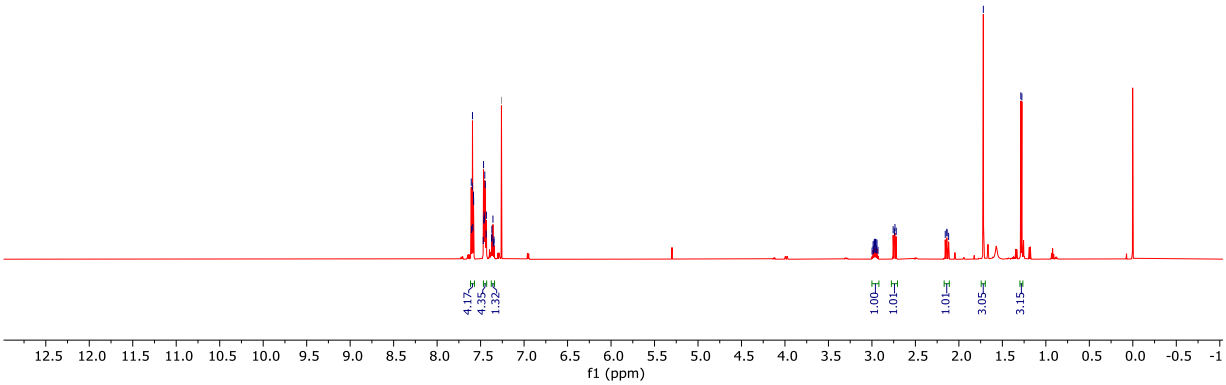
3aa
Diastereomer-1

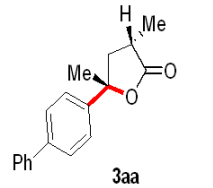


3aa

Diastereomer-2

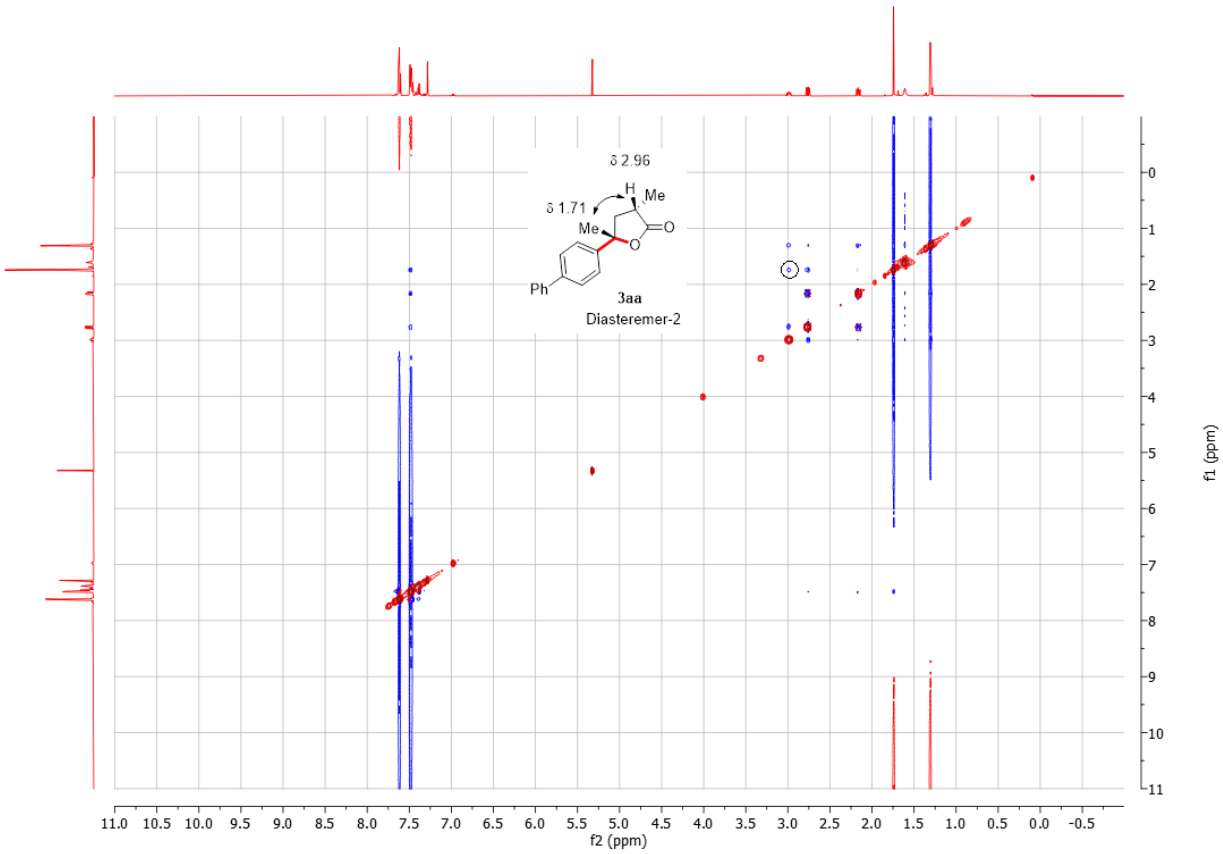


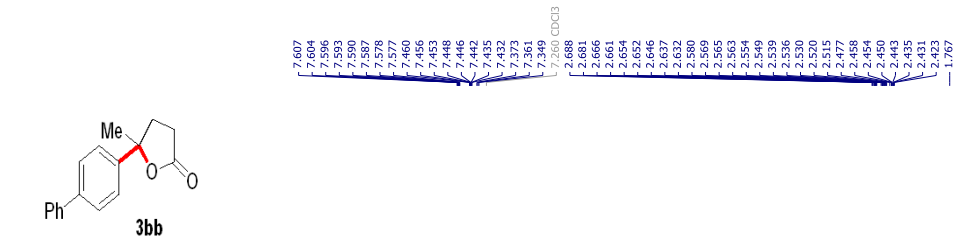




3aa

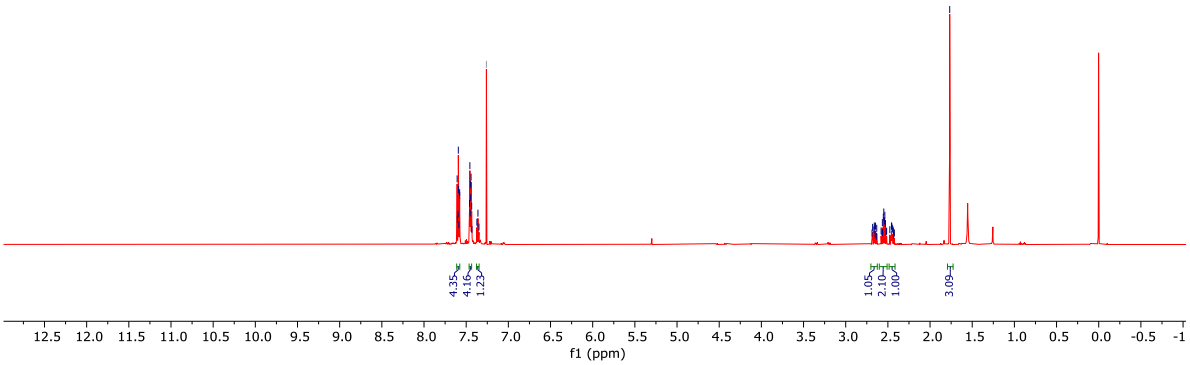
Diastereomer-2

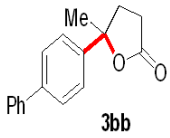

δ 2.96
δ 1.71
H
Me
Me
O
O
Ph
3aa
Diasteremer-2
f1 (ppm)
f2 (ppm)


7.607
7.604
7.596
7.593
7.590
7.587
7.578
7.577
7.460
7.456
7.453
7.448
7.446
7.442
7.435
7.432
7.373
7.361
7.349
7.260 CDCl3
2.688
2.681
2.666
2.661
2.654
2.652
2.646
2.637
2.632
2.580
2.569
2.565
2.563
2.554
2.549
2.539
2.536
2.530
2.520
2.515
2.477
2.458
2.454
2.450
2.443
2.435
2.431
2.423
1.767

Me
O
O
Ph

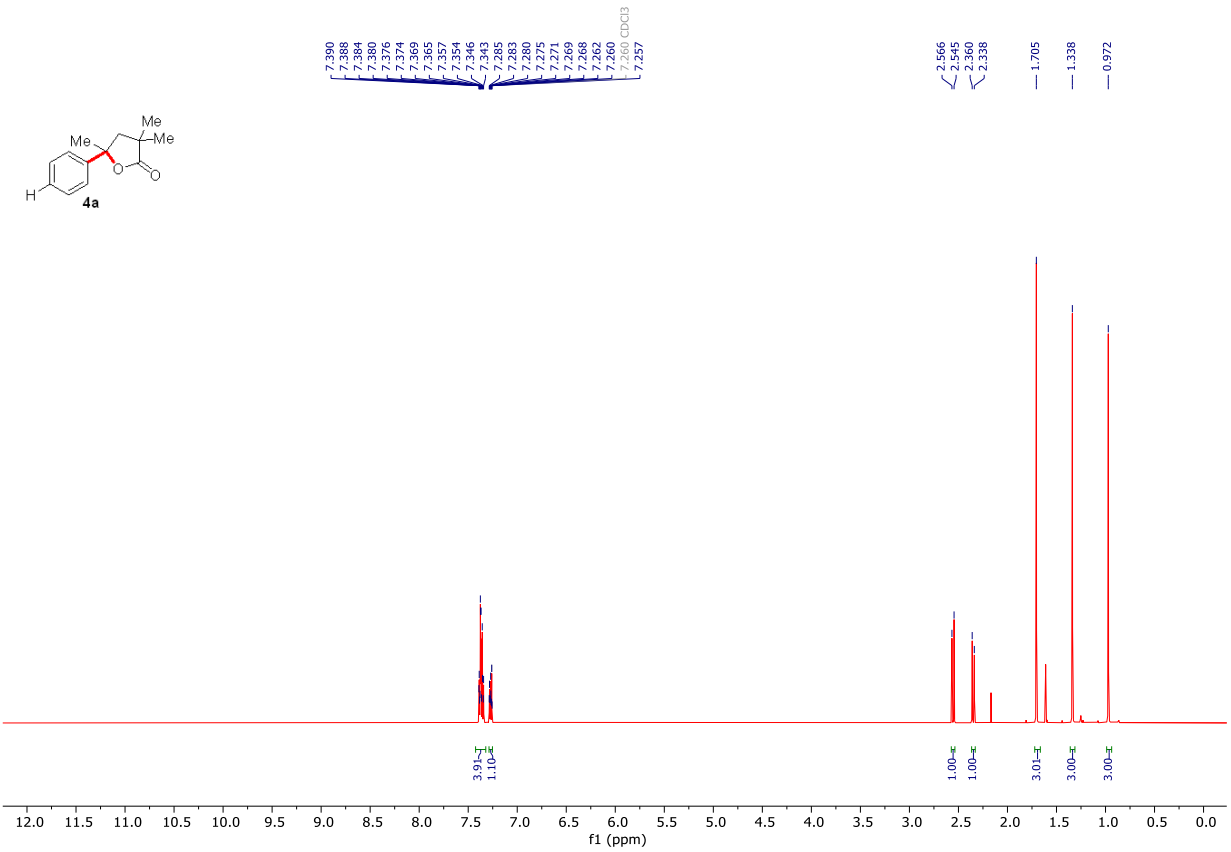
3bb

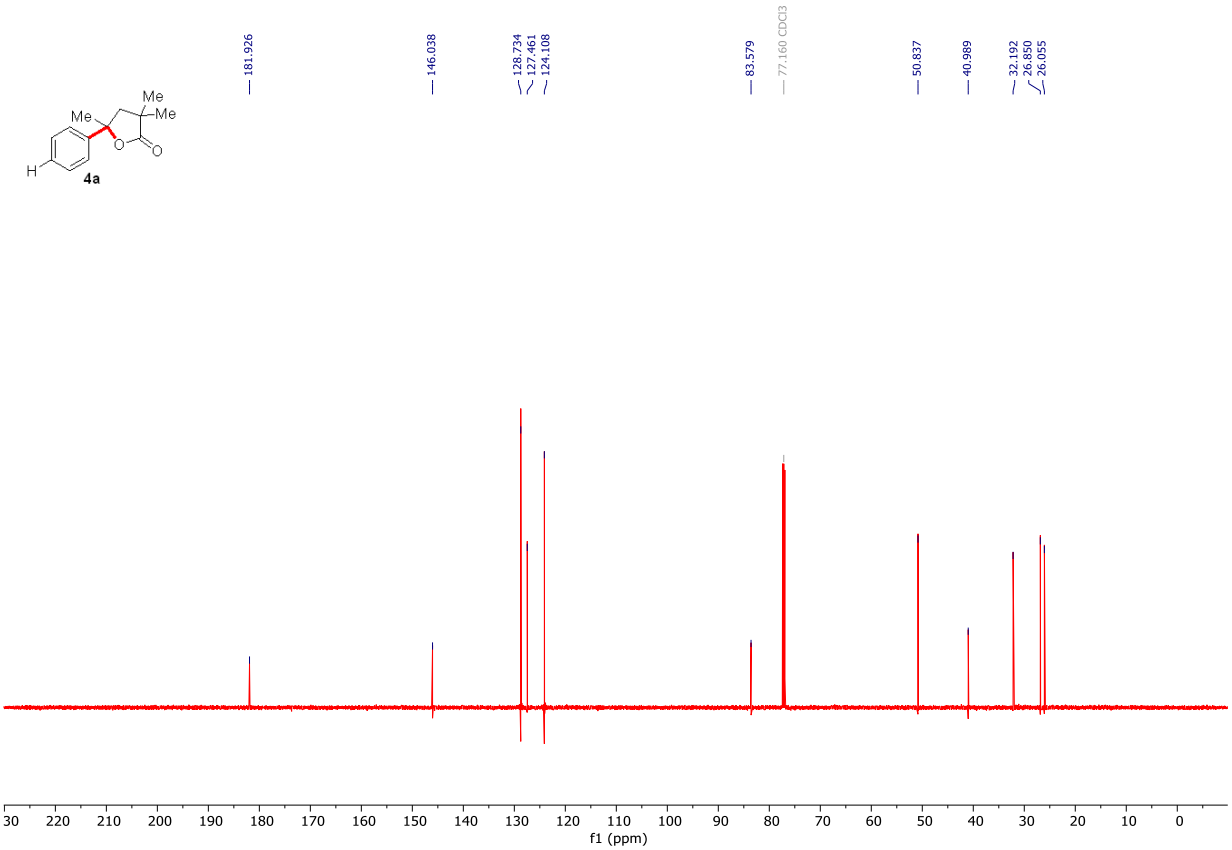

4.35
4.16
1.23
1.05
2.10
1.00
3.09
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

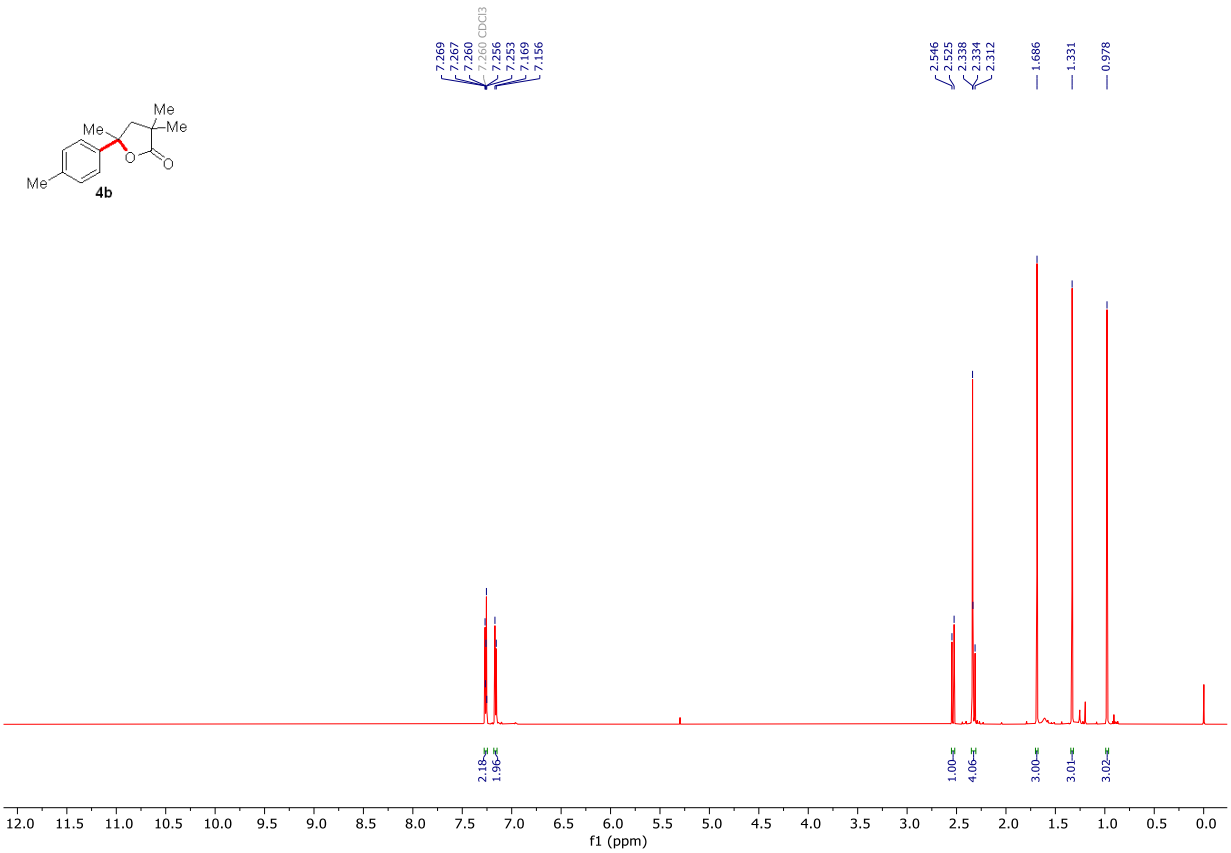

Me
O
O
Ph

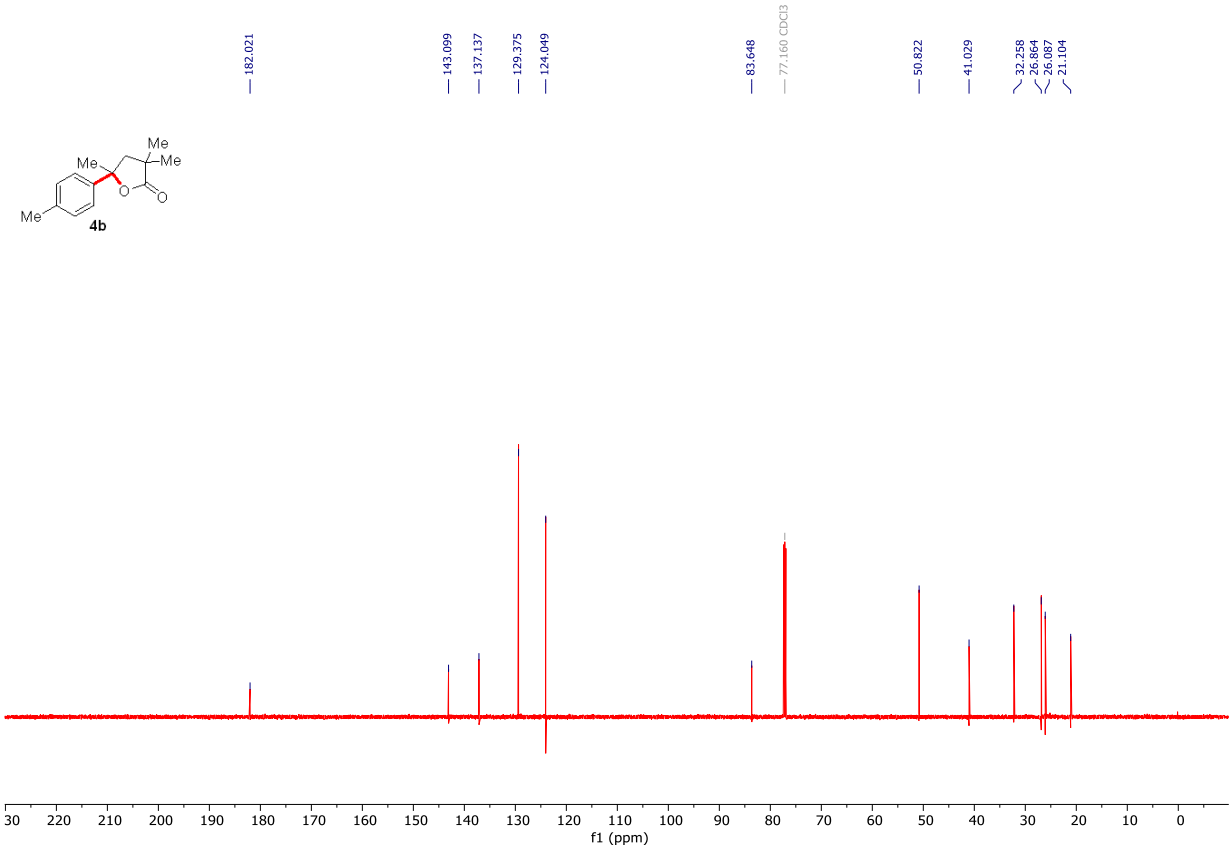
3bb

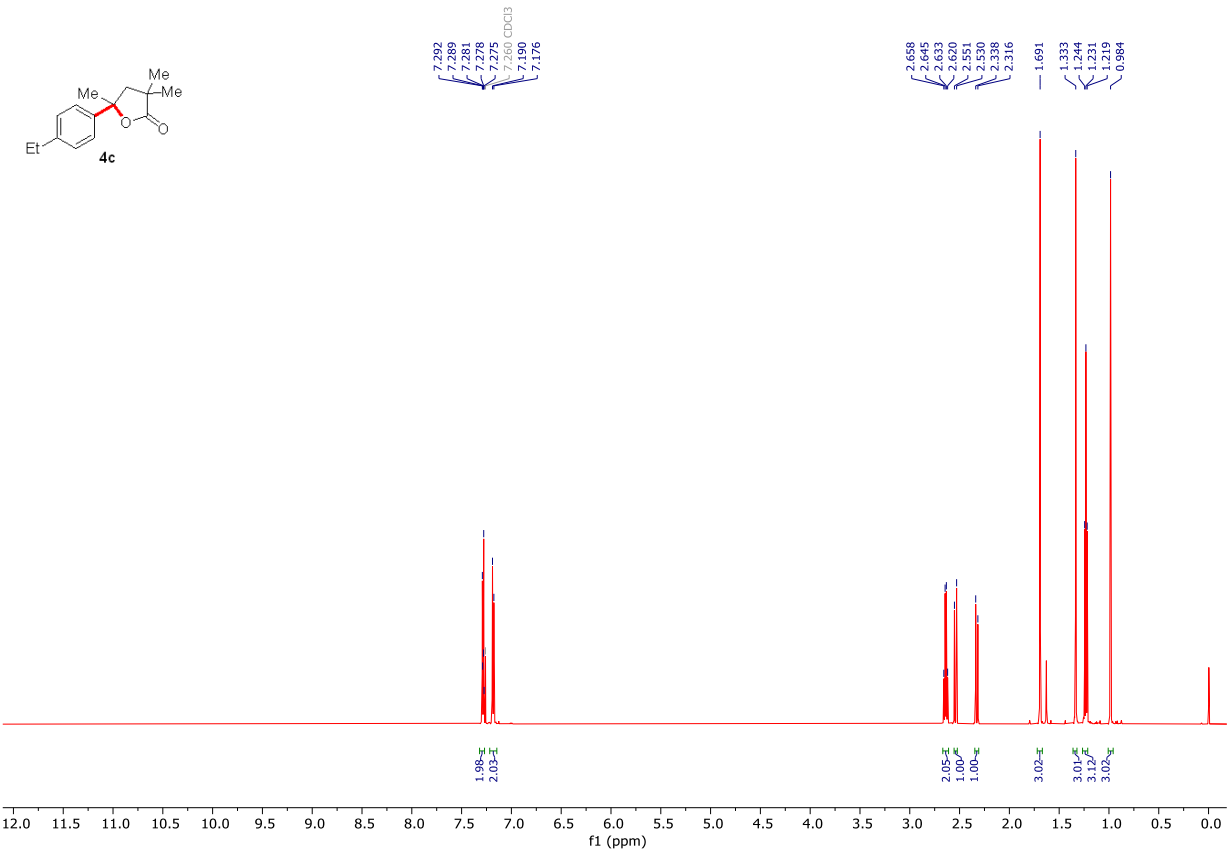
176.633
143.404
140.790
140.558
128.984
127.653
127.482
127.230
124.763
87.060
77.160 CDCl3
36.361
29.572
29.153
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

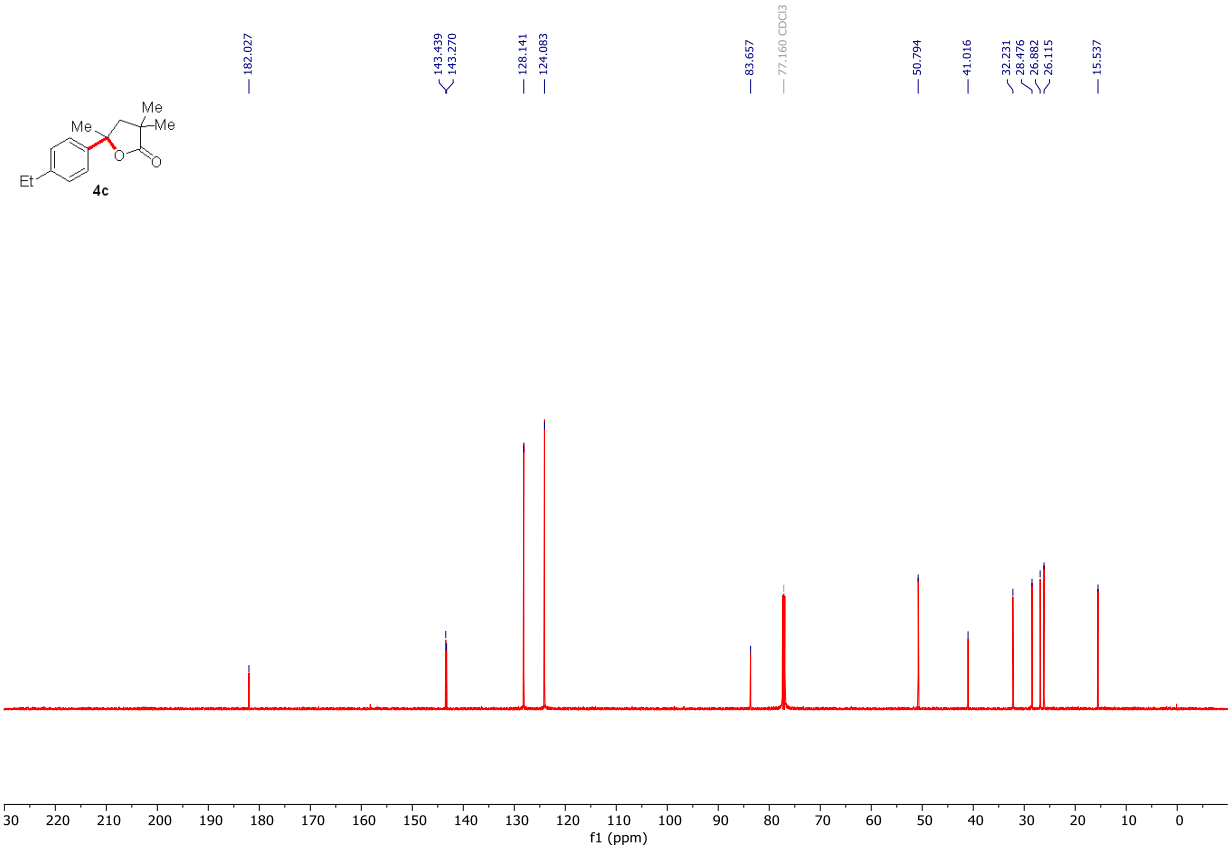

7.390
7.388
7.384
7.380
7.376
7.374
7.369
7.365
7.357
7.354
7.346
7.343
7.285
7.283
7.280
7.275
7.271
7.269
7.268
7.262
7.260
7.260 CDCl3
7.257
2.566
2.545
2.360
2.338
1.705
1.338
0.972
Me
Me
Me
O
O
H
4a
3.91
1.10
1.00
1.00
3.01
3.00
3.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

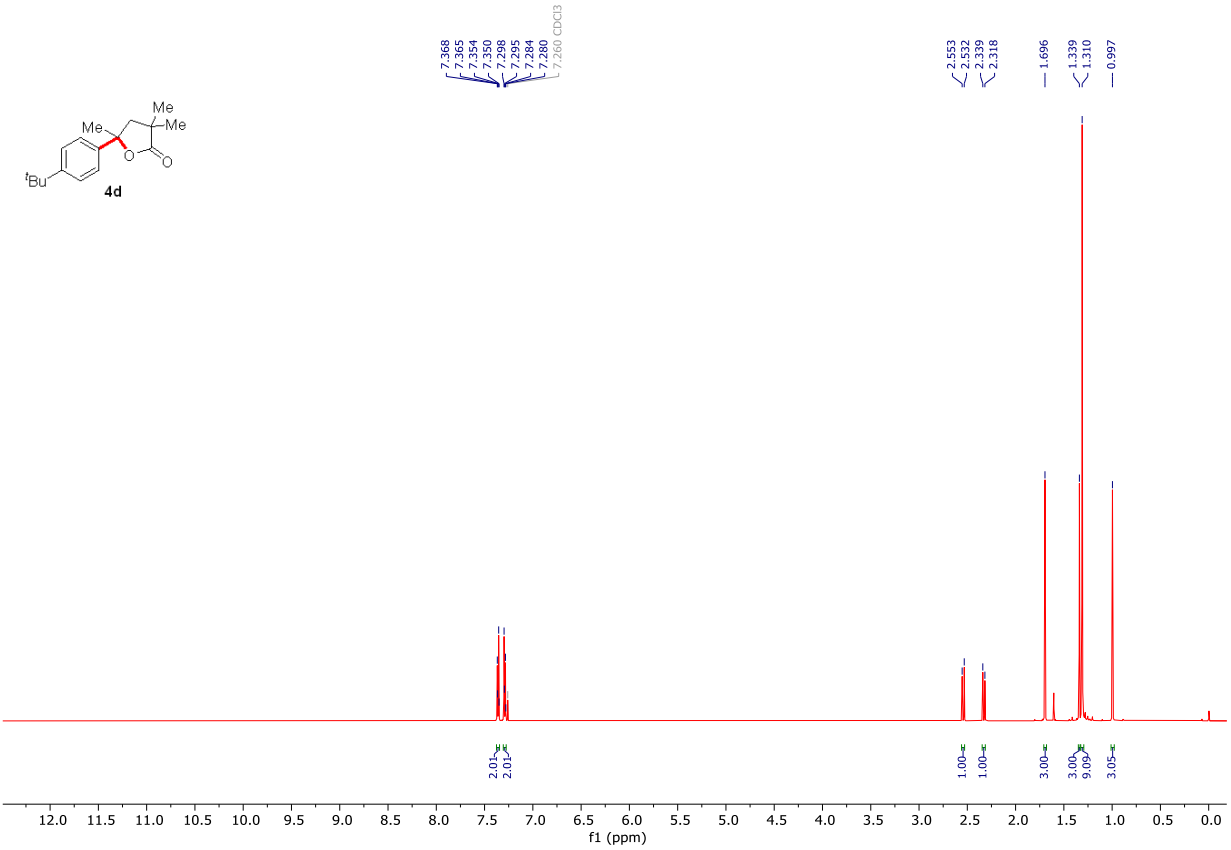

181.926
146.038
128.734
127.461
124.108
83.579
77.160 CDCl3
50.837
40.989
32.192
26.850
26.055
Me
Me
Me
O
O
H
4a
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

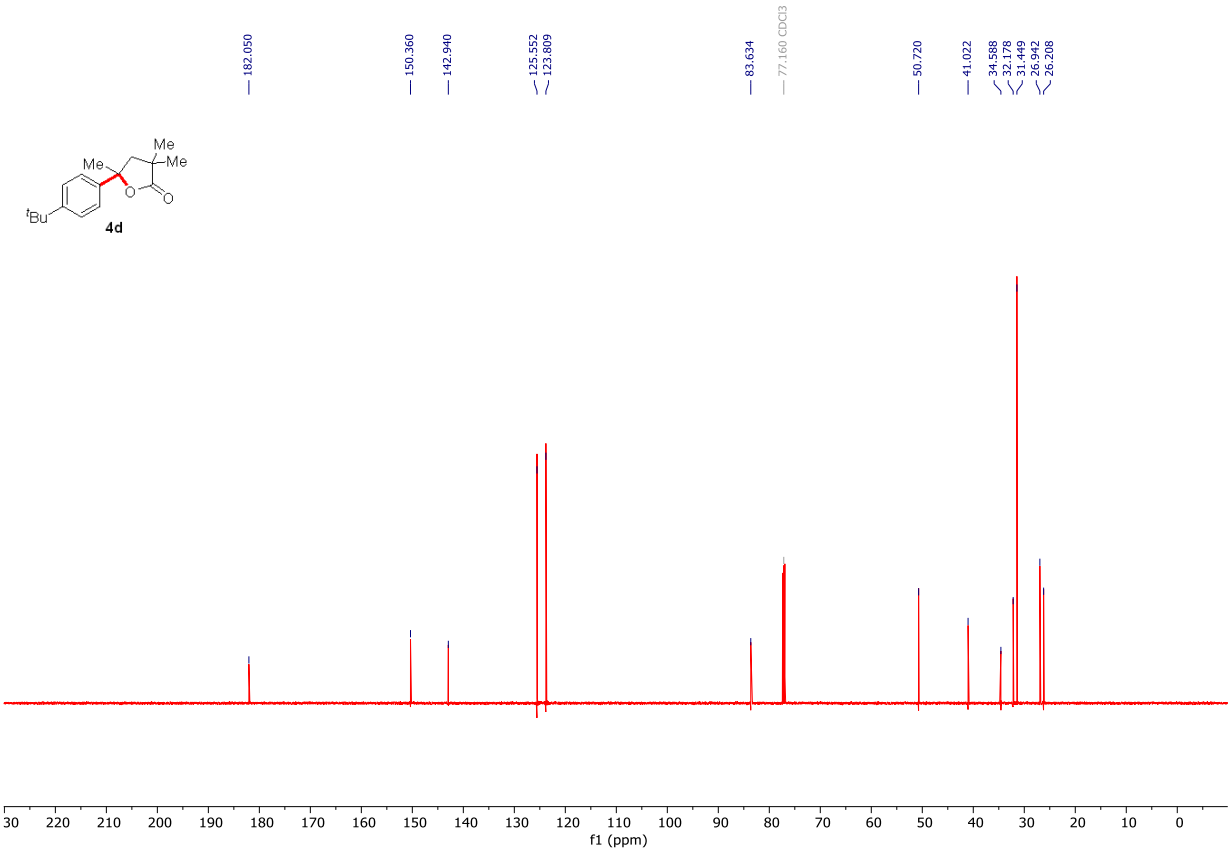

7.269
7.267
7.260
7.260 CDCl3
7.256
7.253
7.169
7.156
2.546
2.525
2.338
2.334
2.312
1.686
1.331
0.978
Me
Me
Me
Me
O
O
4b
2.18
1.96
1.00
4.06
3.00
3.01
3.02
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

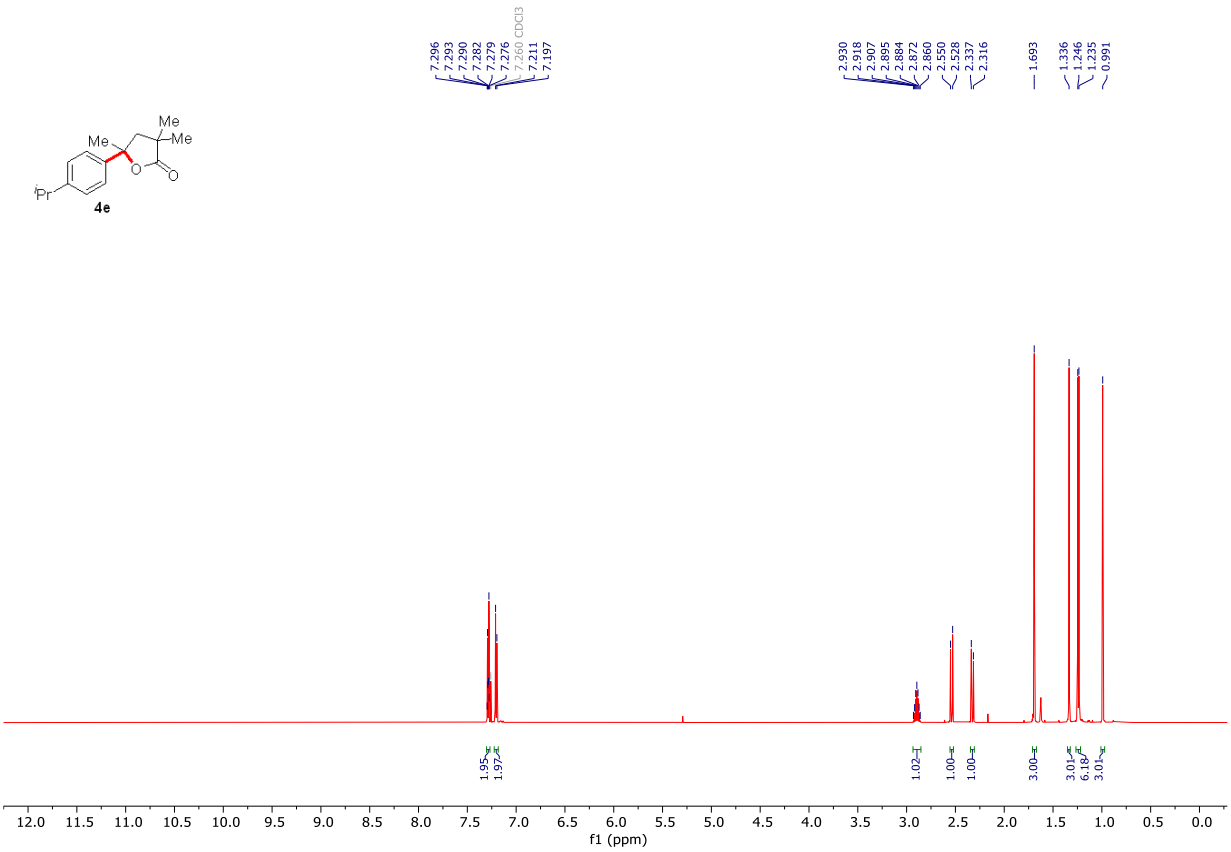

182.021
143.099
137.137
129.375
124.049
83.648
77.160 CDCl3
50.822
41.029
32.258
26.864
26.087
21.104
Me
Me
Me
Me
O
O
4b
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

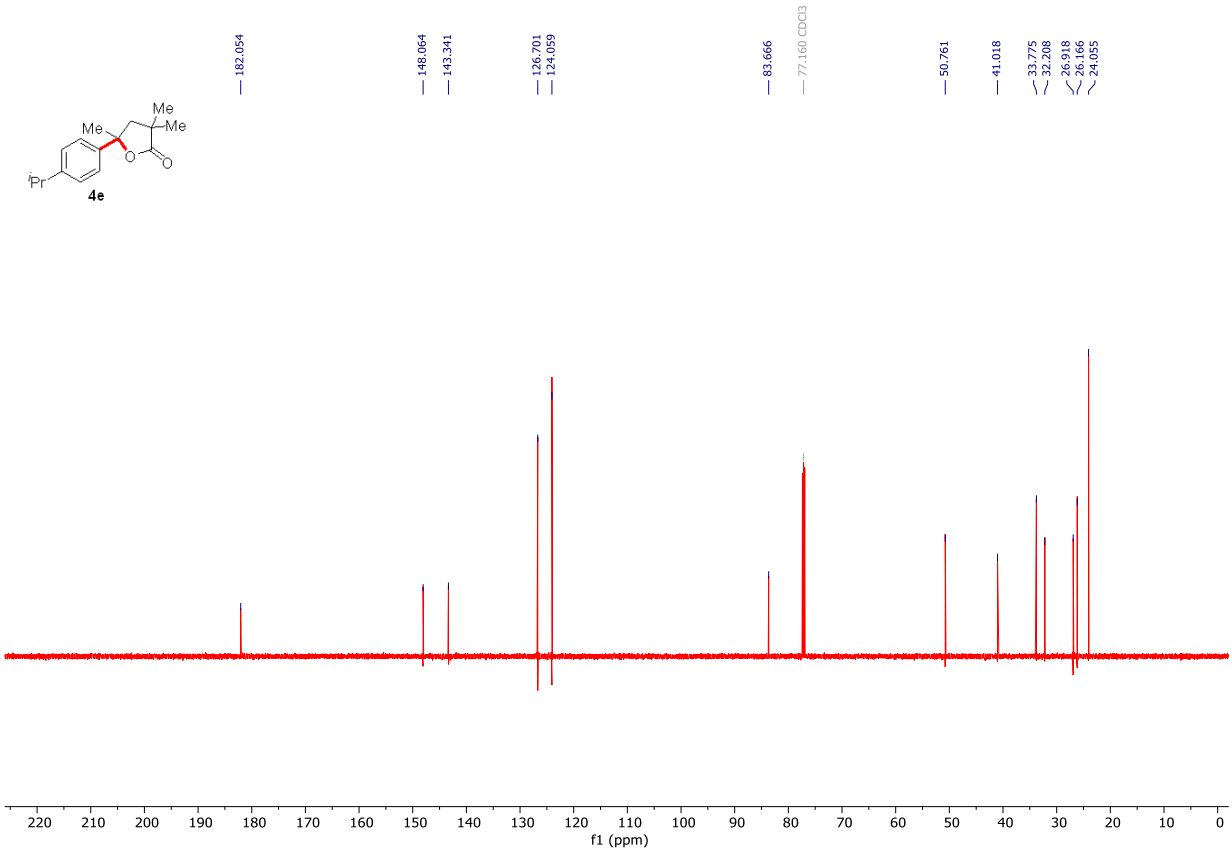

Me
Me
Me
O
O
Et
4c
7.292
7.289
7.281
7.278
7.275
7.260 CDCl3
7.190
7.176
2.658
2.645
2.633
2.620
2.551
2.530
2.338
2.316
1.691
1.333
1.244
1.231
1.219
0.984
1.98
2.03
2.05
1.00
1.00
3.02
3.01
3.12
3.02
f1 (ppm)

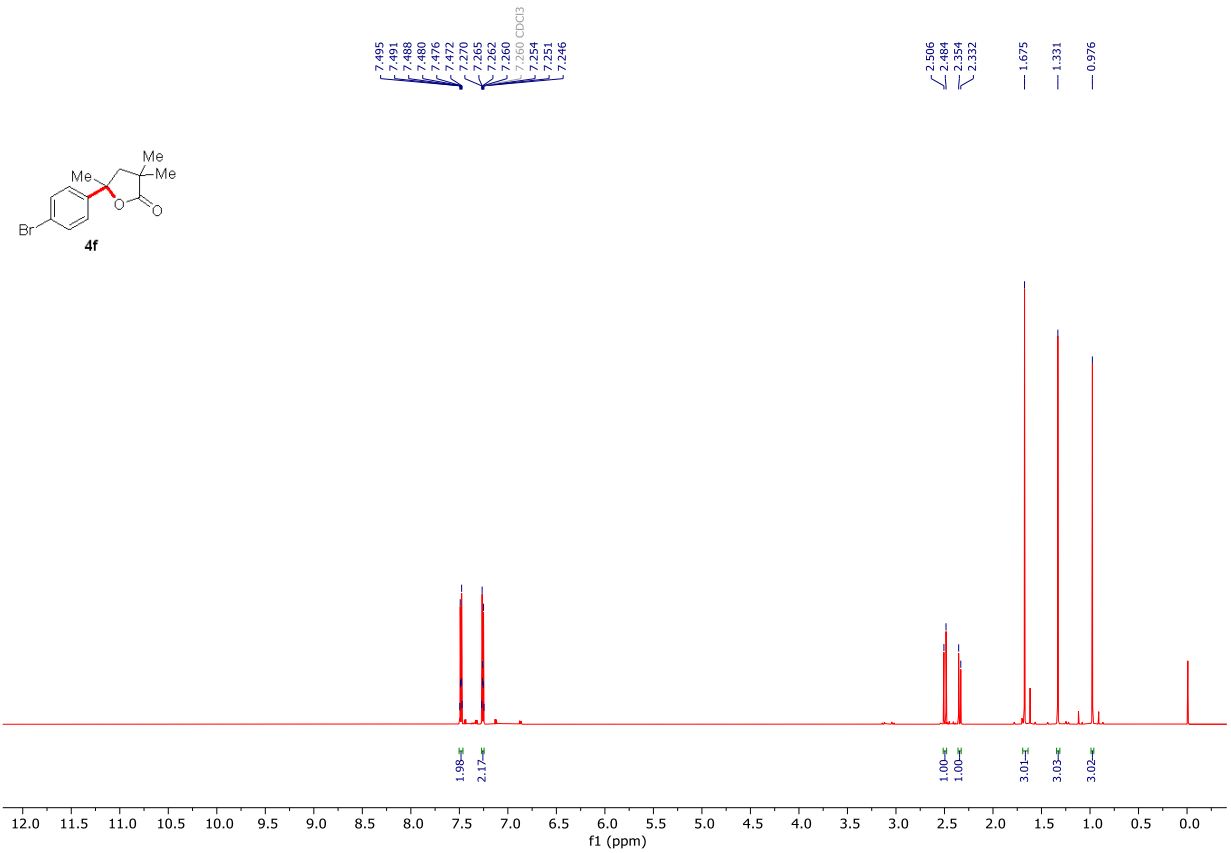

Me
Me
Me
O
O
Et
4c
182.027
143.439
143.270
128.141
124.083
83.657
77.160 CDCl3
50.794
41.016
32.231
28.476
26.882
26.115
15.537
f1 (ppm)

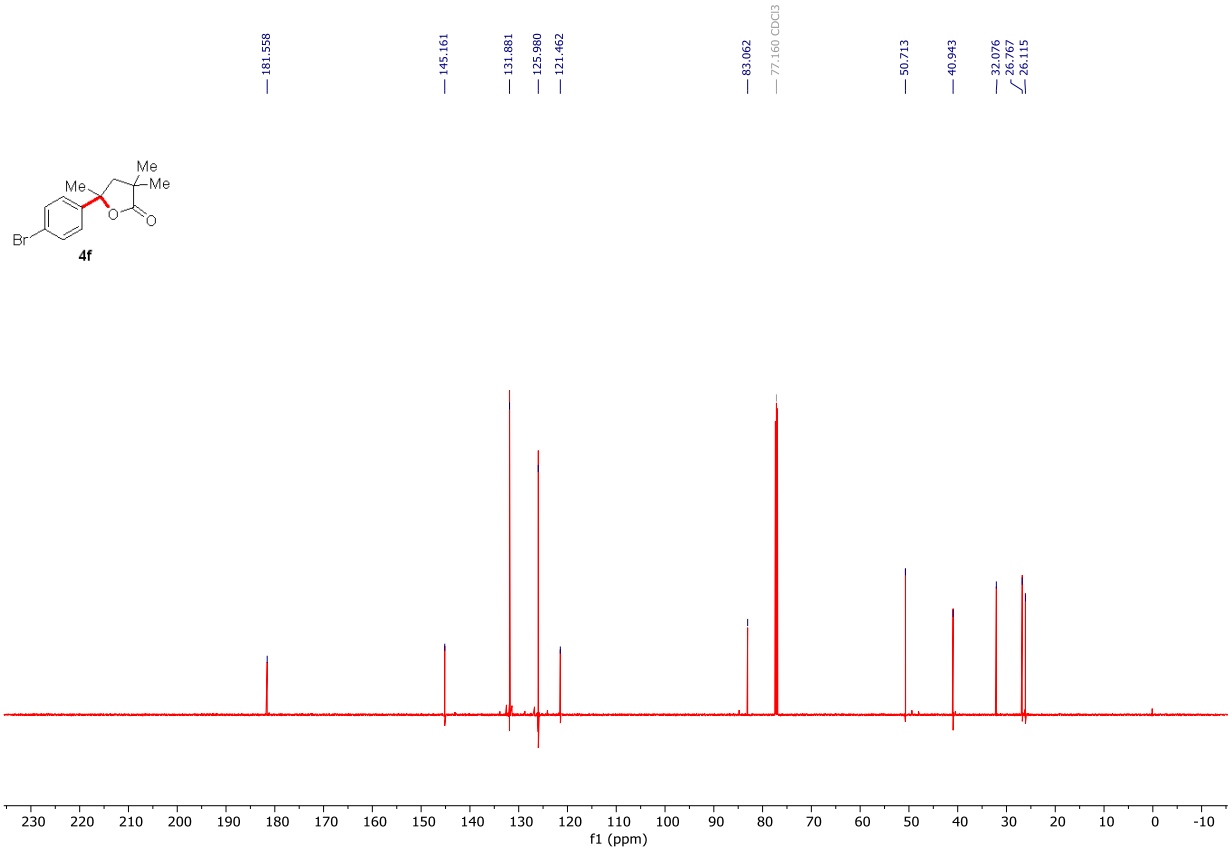

7.368
7.365
7.354
7.350
7.298
7.295
7.284
7.280
7.260 CDCl3
2.553
2.532
2.339
2.318
1.696
1.339
1.310
0.997
Me
Me
Me
O
O
tBu
4d
2.01
2.01
1.00
1.00
3.00
3.00
9.09
3.05
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

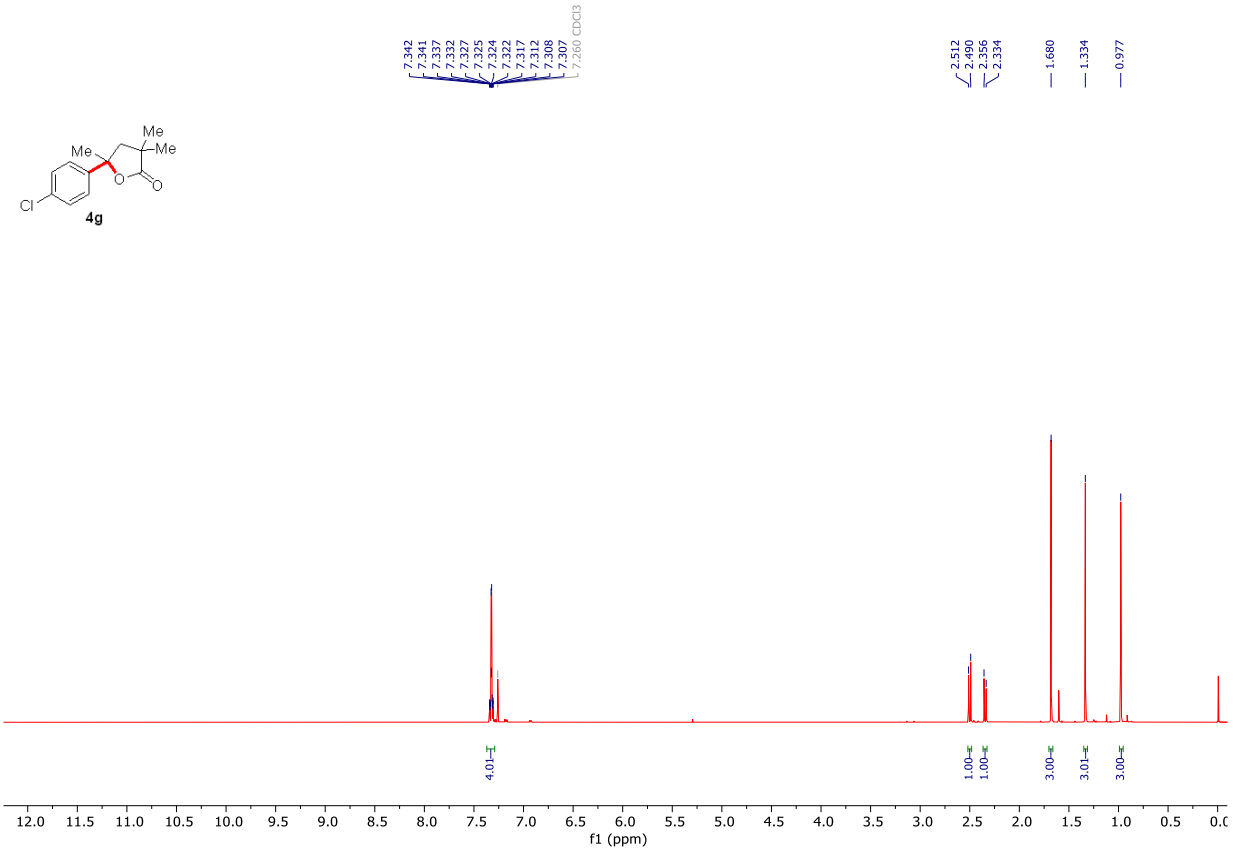

182.050
150.360
142.940
125.552
123.809
83.634
77.160 CDCl3
50.720
41.022
34.588
32.178
31.449
26.942
26.208
Me
Me
Me
O
O
tBu
4d
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

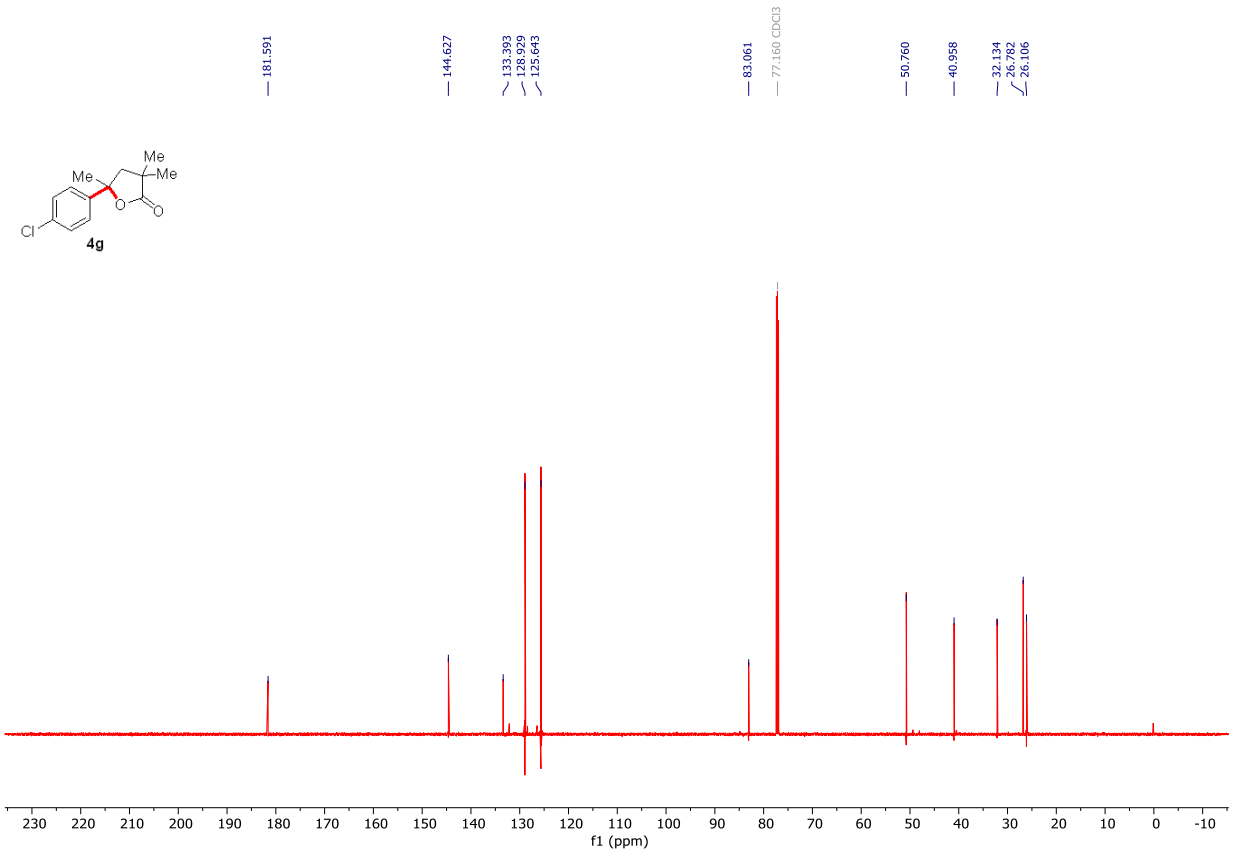

7.296
7.293
7.290
7.282
7.279
7.276
7.260 CDCl3
7.211
7.197
2.930
2.918
2.907
2.895
2.884
2.872
2.860
2.550
2.528
2.337
2.316
1.693
1.336
1.246
1.235
0.991
Me
Me
Me
O
O
iPr
4e
1.95
1.97
1.02
1.00
1.00
3.00
3.01
6.18
3.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

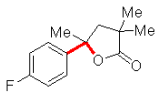

182.054
148.064
143.341
126.701
124.059
83.666
77.160 CDCl3
50.761
41.018
33.775
32.208
26.918
26.166
24.055
Me
Me
Me
O
O
iPr
4e
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.495
7.491
7.488
7.480
7.476
7.472
7.270
7.265
7.262
7.260
7.260 CDCl3
7.254
7.251
7.246
2.506
2.484
2.354
2.332
1.675
1.331
0.976
Me
Me
Me
O
O
Br
4f
1.98
2.17
1.00
1.00
3.01
3.03
3.02
12.0 11.5 11.0 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
f1 (ppm)


181.558
145.161
131.881
125.980
121.462
83.062
77.160 CDCl3
50.713
40.943
32.076
26.767
26.115
Me
Me
Me
O
O
Br
4f
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
f1 (ppm)

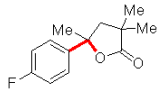

7.342
7.341
7.337
7.332
7.327
7.325
7.324
7.322
7.317
7.312
7.308
7.307
7.260 CDCl3
2.512
2.490
2.356
2.334
1.680
1.334
0.977
Me
Me
Me
O
O
Cl
4g
4.01
1.00
1.00
3.00
3.01
3.00
f1 (ppm)


181.591
144.627
133.393
128.929
125.643
83.061
77.160 CDCl3
50.760
40.958
32.134
26.782
26.106
Me
Me
Me
O
O
Cl
4g
f1 (ppm)


Me
Me
Me
O
O
F

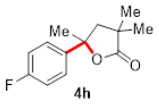
4h

7.367
7.362
7.358
7.353
7.350
7.349
7.347
7.342
7.338
7.333
7.260 CDCl3
7.062
7.057
7.054
7.048
7.046
7.043
7.039
7.037
7.032
7.028
7.023
2.524
2.502
2.356
2.334
1.685
1.337
0.980
2.00
2.00
1.00
1.00
3.00
3.02
3.01
12.0 11.5 11.0 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
f1 (ppm)


Me
Me
Me
O
O
F

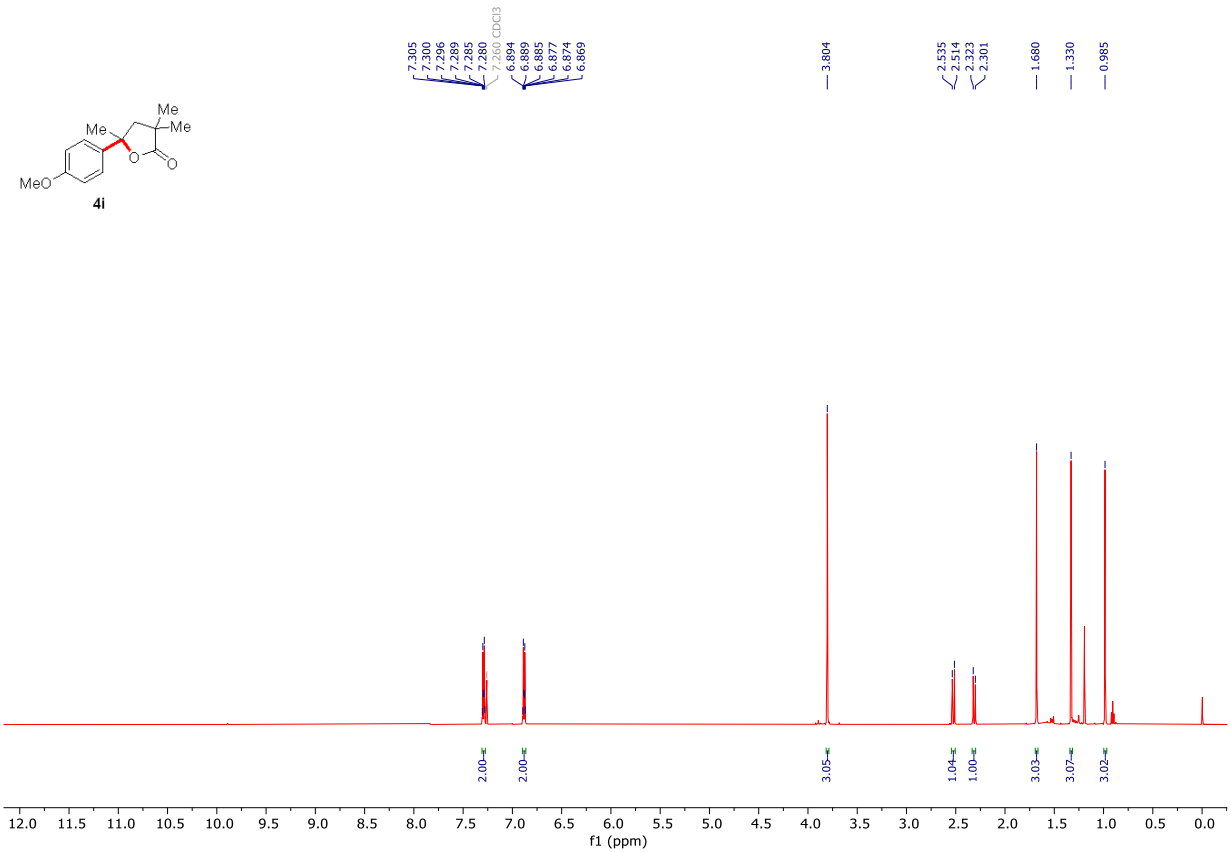
4h

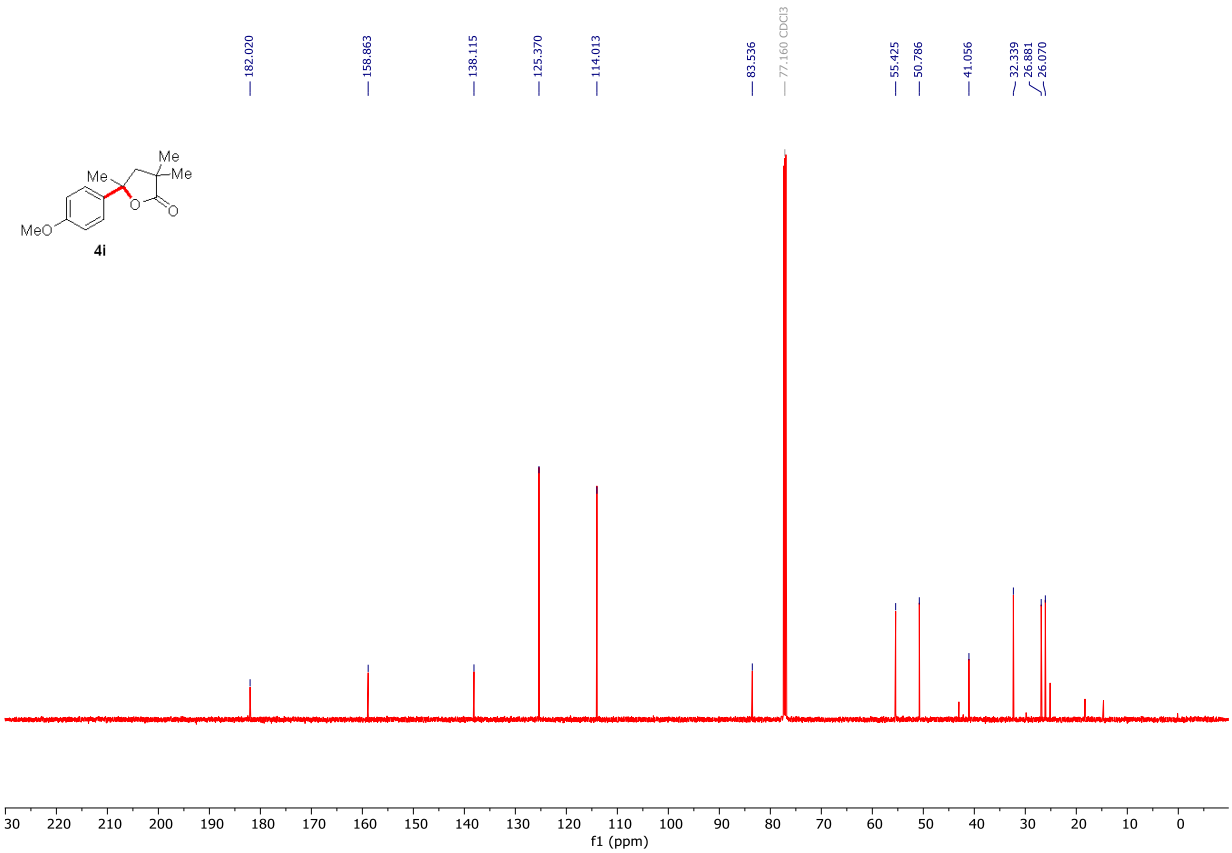
181.691
162.866
161.234
141.889
141.868
125.940
125.887
115.674
115.531
83.187
77.160 CDCl3
50.834
40.983
32.315
26.823
26.082
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

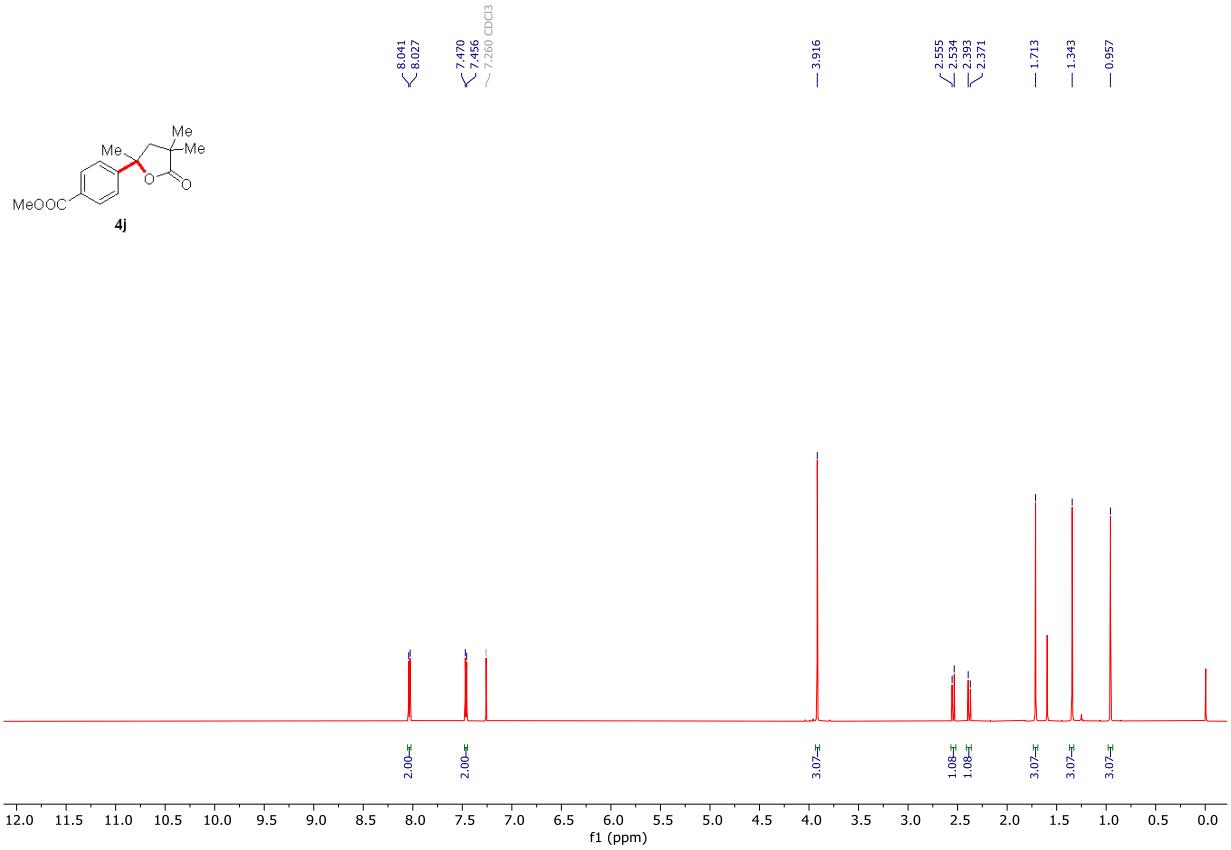

Me
Me
Me
O
O
F

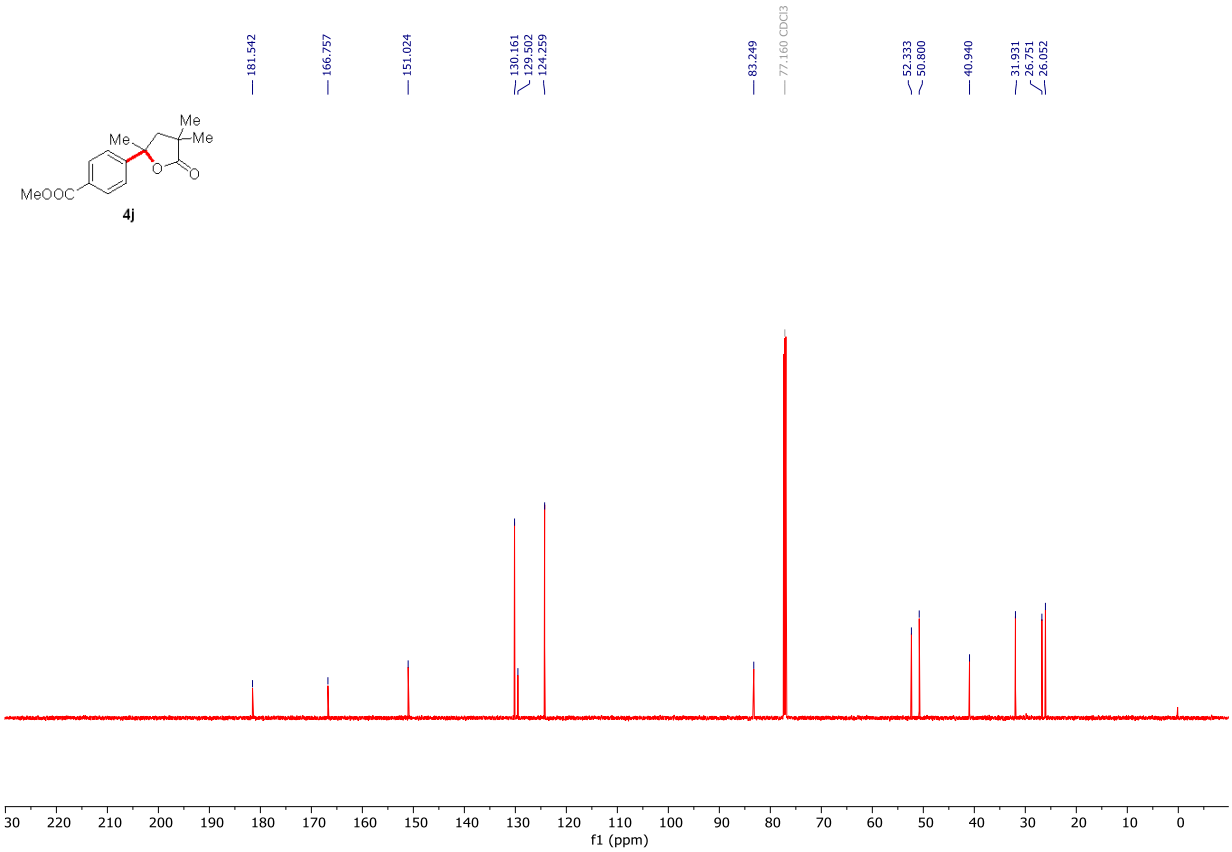
4h

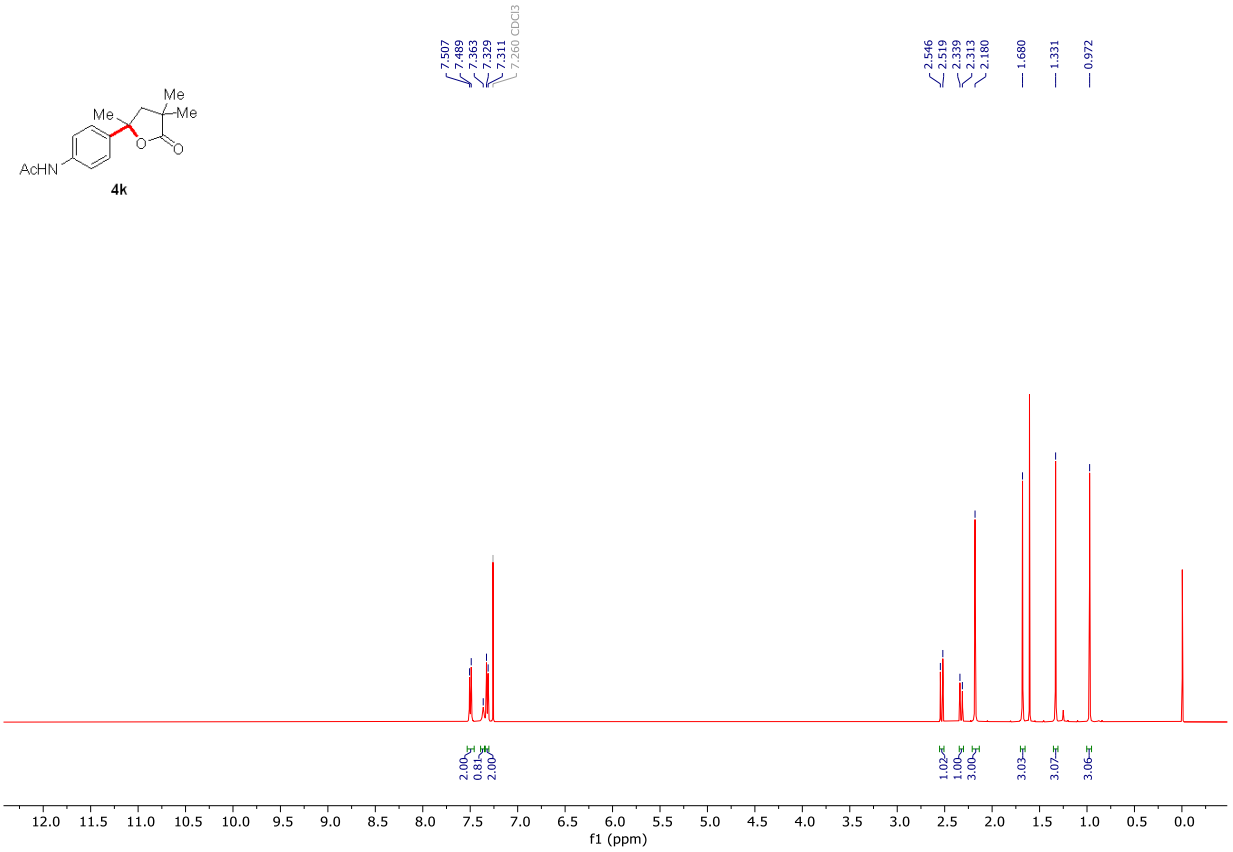
-115.169
-115.183
-115.192
-115.197
-115.206
-115.215
-115.220
-115.228
-115.242
A (s)
-115.21
1.00
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
-120
f1 (ppm)

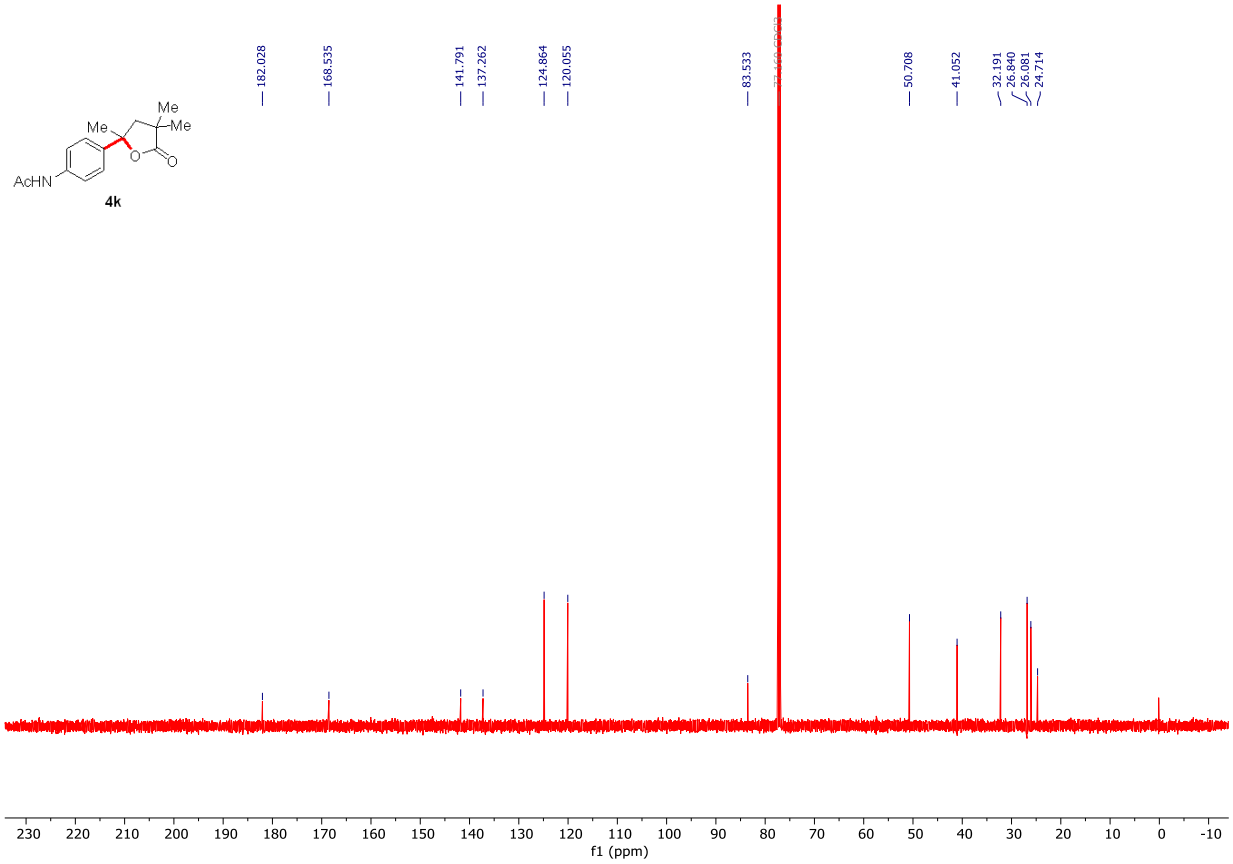

7.305
7.300
7.296
7.289
7.285
7.280
7.260 CDCl3
6.894
6.889
6.885
6.877
6.874
6.869
3.804
2.535
2.514
2.323
2.301
1.680
1.330
0.985
Me
Me
Me
O
O
MeO
4i
2.00
2.00
3.05
1.04
1.00
3.03
3.07
3.02
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

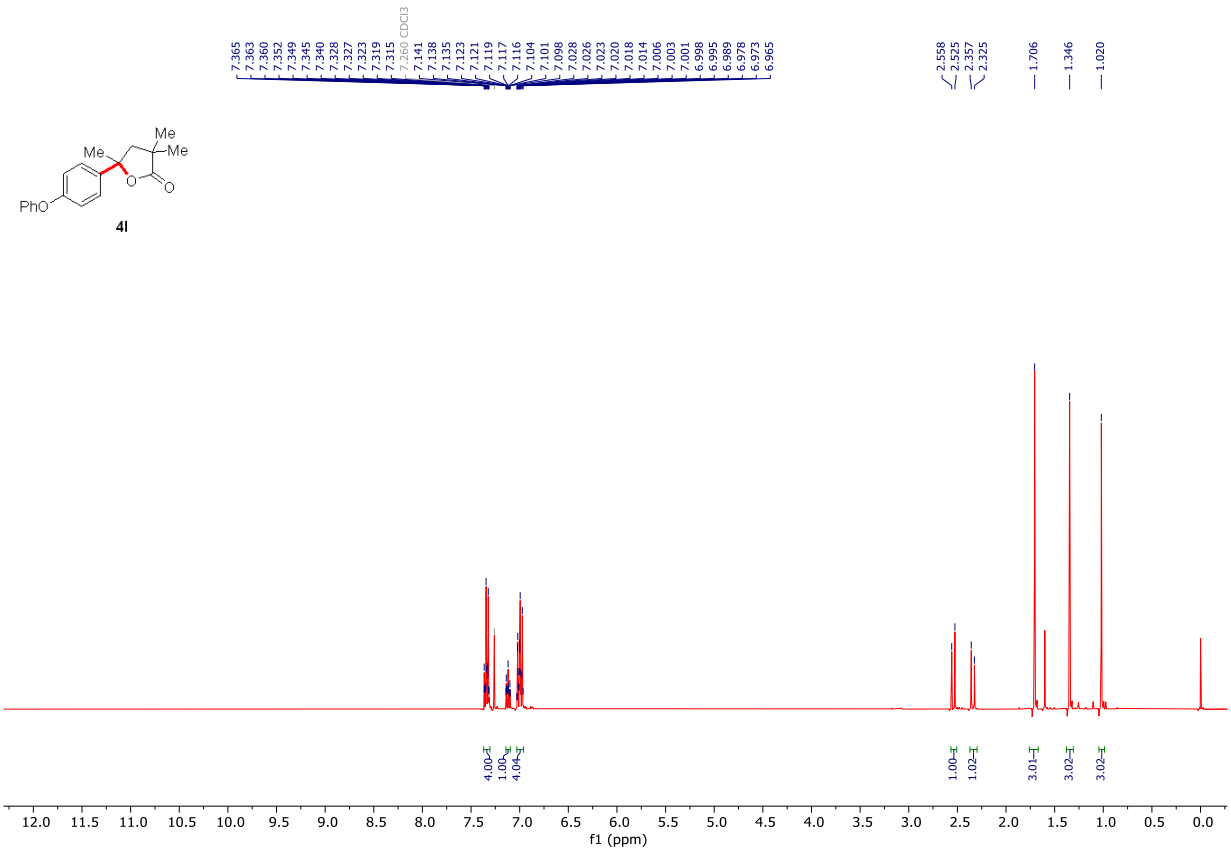

182.020
158.863
138.115
125.370
114.013
83.536
77.160 CDCl3
55.425
50.786
41.056
32.339
26.681
26.070
Me
Me
Me
O
O
MeO
4i
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.041
8.027
7.470
7.456
7.260 CDCl3
3.916
2.555
2.534
2.393
2.371
1.713
1.343
0.957
Me
Me
Me
O
O
MeOOC
4j
2.00
2.00
3.07
1.08
1.08
3.07
3.07
3.07
12.0 11.5 11.0 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
f1 (ppm)

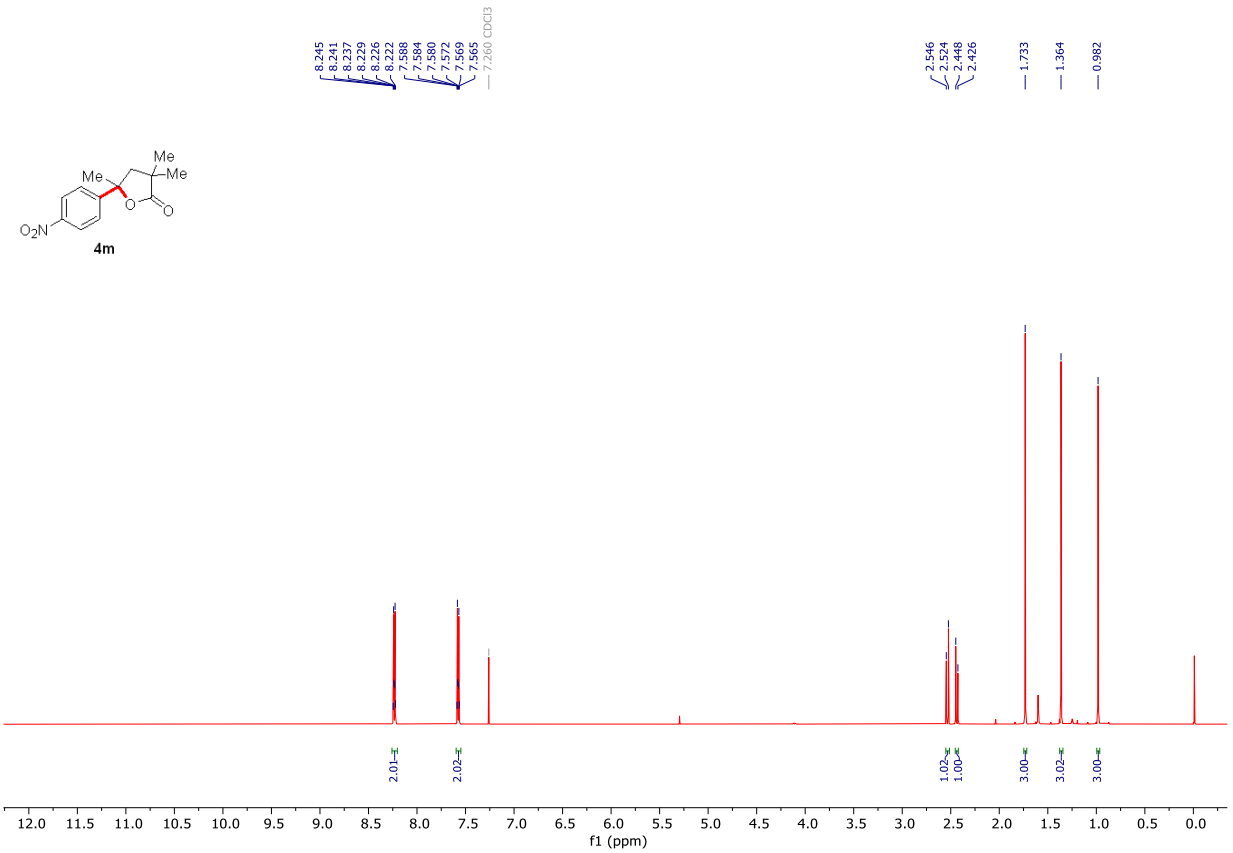

181.542
166.757
151.024
130.161
129.502
124.259
83.249
77.160 CDCl3
52.333
50.800
40.940
31.931
26.751
26.052
Me
Me
Me
O
O
MeOOC
4j
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

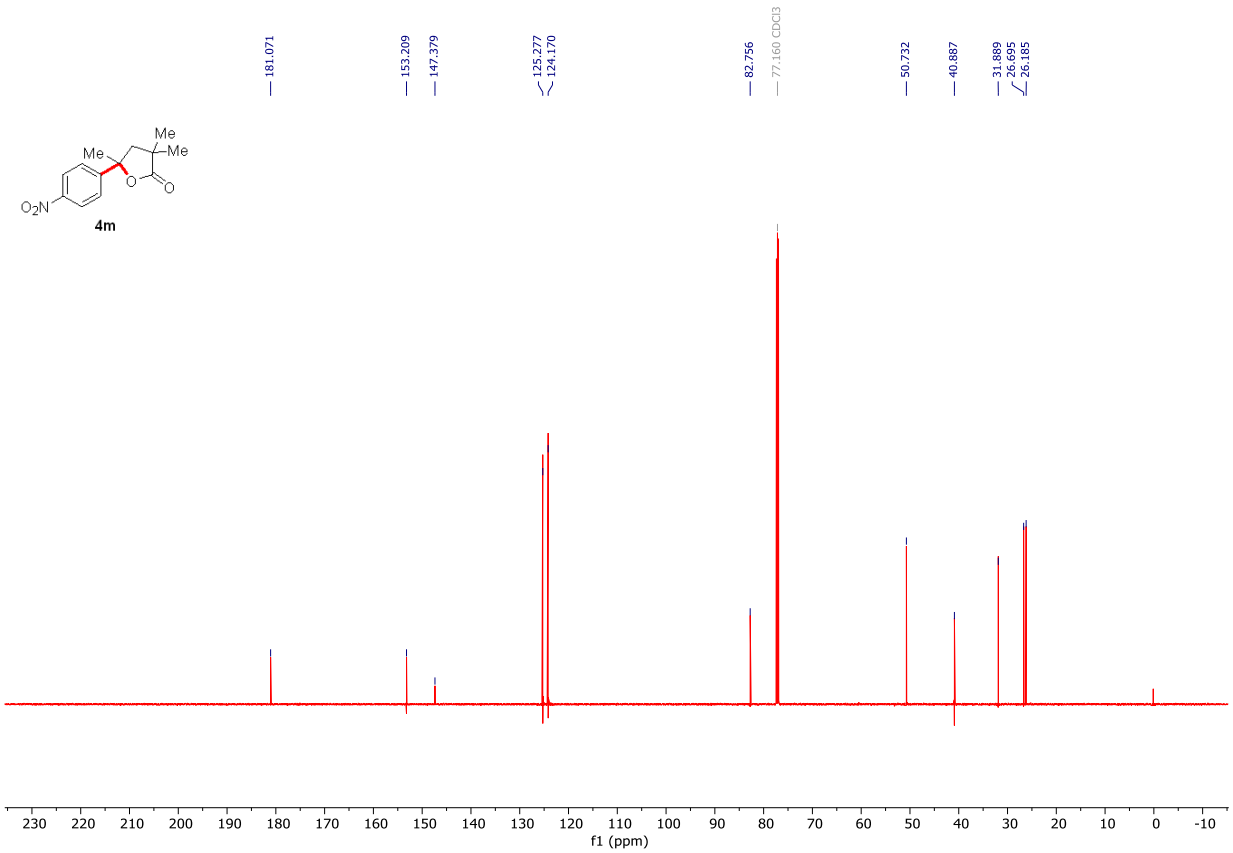

7.507
7.489
7.363
7.329
7.311
7.260 CDCl3
2.546
2.519
2.339
2.313
2.180
1.680
1.331
0.972
Me
Me
Me
O
O
AcHN
4k
2.00
0.81
2.00
1.02
1.00
3.00
3.03
3.07
3.06
f1 (ppm)

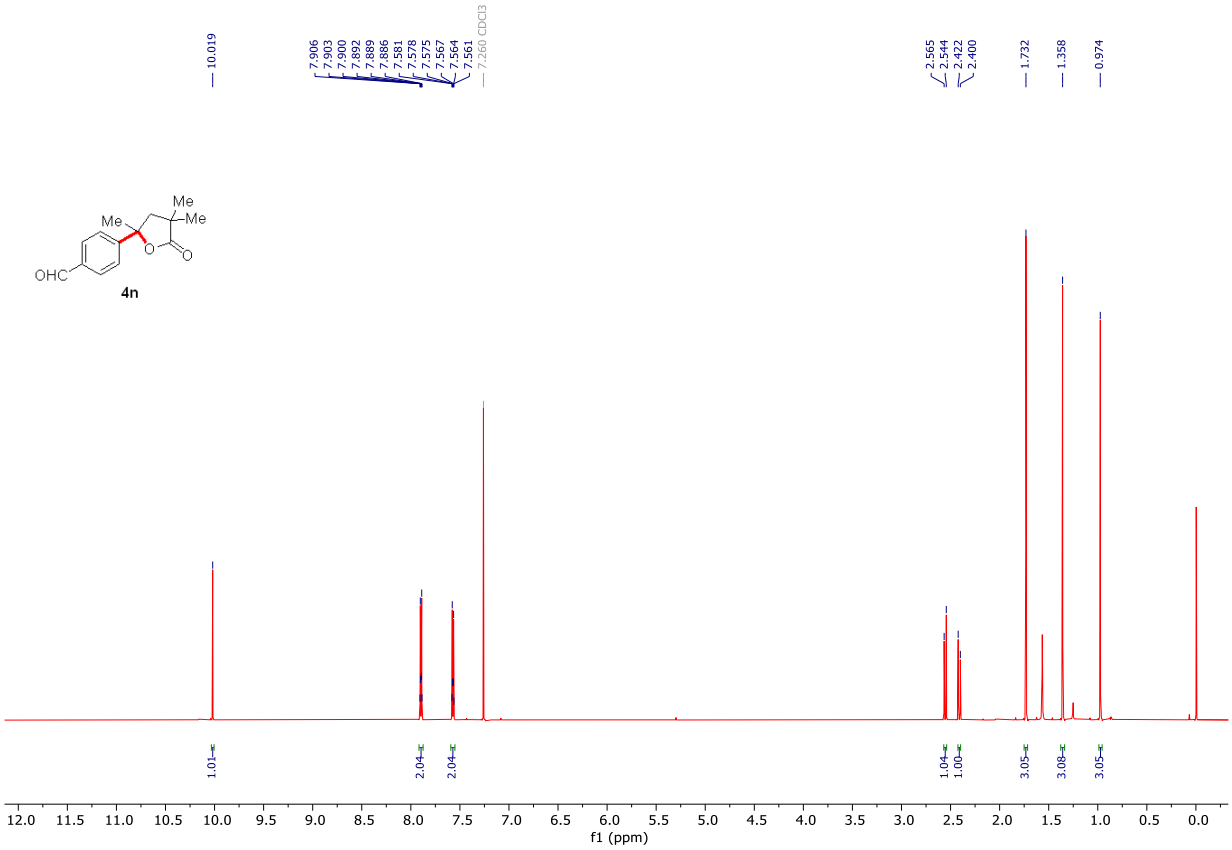

182.028
168.535
141.791
137.262
124.864
120.055
83.533
77.460 CDCl3
50.708
41.052
32.191
26.840
26.081
24.714
Me
Me
Me
O
O
AcHN
4k
f1 (ppm)

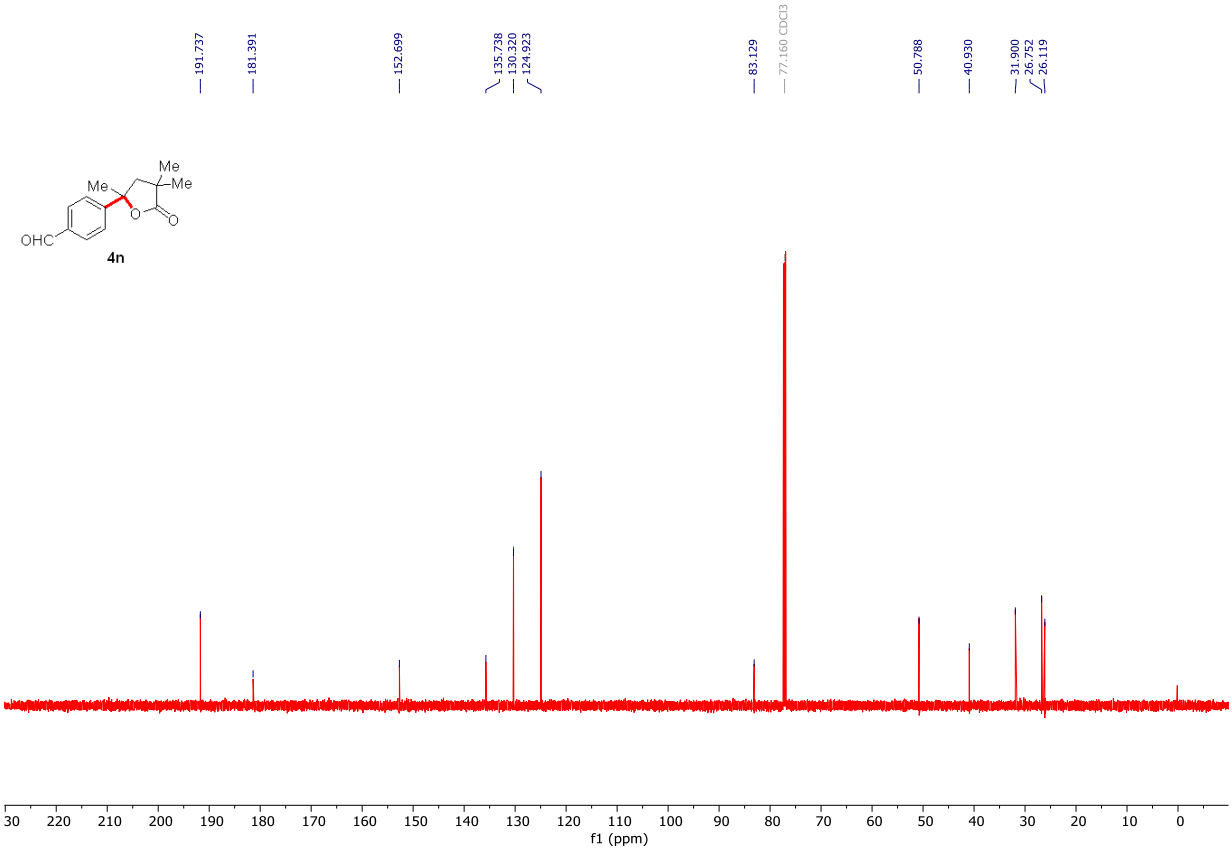

7.365
7.363
7.360
7.352
7.349
7.345
7.340
7.328
7.327
7.323
7.319
7.315
7.260 CDCl3
7.141
7.138
7.135
7.123
7.121
7.119
7.117
7.116
7.104
7.101
7.098
7.028
7.026
7.023
7.020
7.018
7.014
7.006
7.003
7.001
6.998
6.995
6.989
6.978
6.973
6.965
2.558
2.525
2.357
2.325
1.706
1.346
1.020
Me
Me
Me
O
O
PhO
4l
4.00
1.00
4.04
1.00
1.02
3.01
3.02
3.02
f1 (ppm)

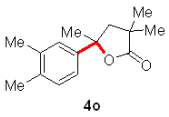

181.852
156.973
156.699
140.719
129.948
125.637
123.674
119.207
118.740
83.410
77.160 CDCl3
50.782
41.027
32.294
26.879
26.151
Me
Me
Me
O
O
PhO
4l
f1 (ppm)


8.245
8.241
8.237
8.229
8.226
8.222
7.588
7.584
7.580
7.572
7.569
7.565
7.260 CDCl3
2.546
2.524
2.448
2.426
1.733
1.364
0.982
Me
Me
Me
O
O
O2N
4m
2.01
2.02
1.02
1.00
3.00
3.02
3.00
f1 (ppm)


181.071
153.209
147.379
125.277
124.170
82.756
77.160 CDCl3
50.732
40.887
31.889
26.695
26.185
Me
Me
Me
O
O
O2N
4m
f1 (ppm)

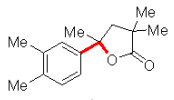

10.019
7.906
7.903
7.900
7.892
7.889
7.886
7.581
7.578
7.575
7.567
7.564
7.561
7.260 CDCl3
2.565
2.544
2.422
2.400
1.732
1.358
0.974
Me
Me
Me
O
O
OHC
4n
1.01
2.04
2.04
1.04
1.00
3.05
3.08
3.05
f1 (ppm)


191.737
181.391
152.699
135.738
130.320
124.923
83.129
77.160 CDCl3
50.788
40.930
31.900
26.752
26.119
Me
Me
Me
O
O
OHC
4n
f1 (ppm)


Me
Me
Me
Me
Me
O
O

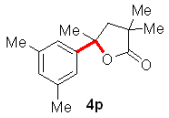
4o

7.260 CDCl3
7.147
7.116
7.103
7.089
7.086
7.076
7.073
2.553
2.531
2.327
2.365
2.267
2.243
1.681
1.331
0.991
0.99
2.00
1.00
1.06
2.96
3.01
3.01
3.05
3.04
12.0 11.5 11.0 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
f1 (ppm)


Me
Me
Me
Me
Me
O
O

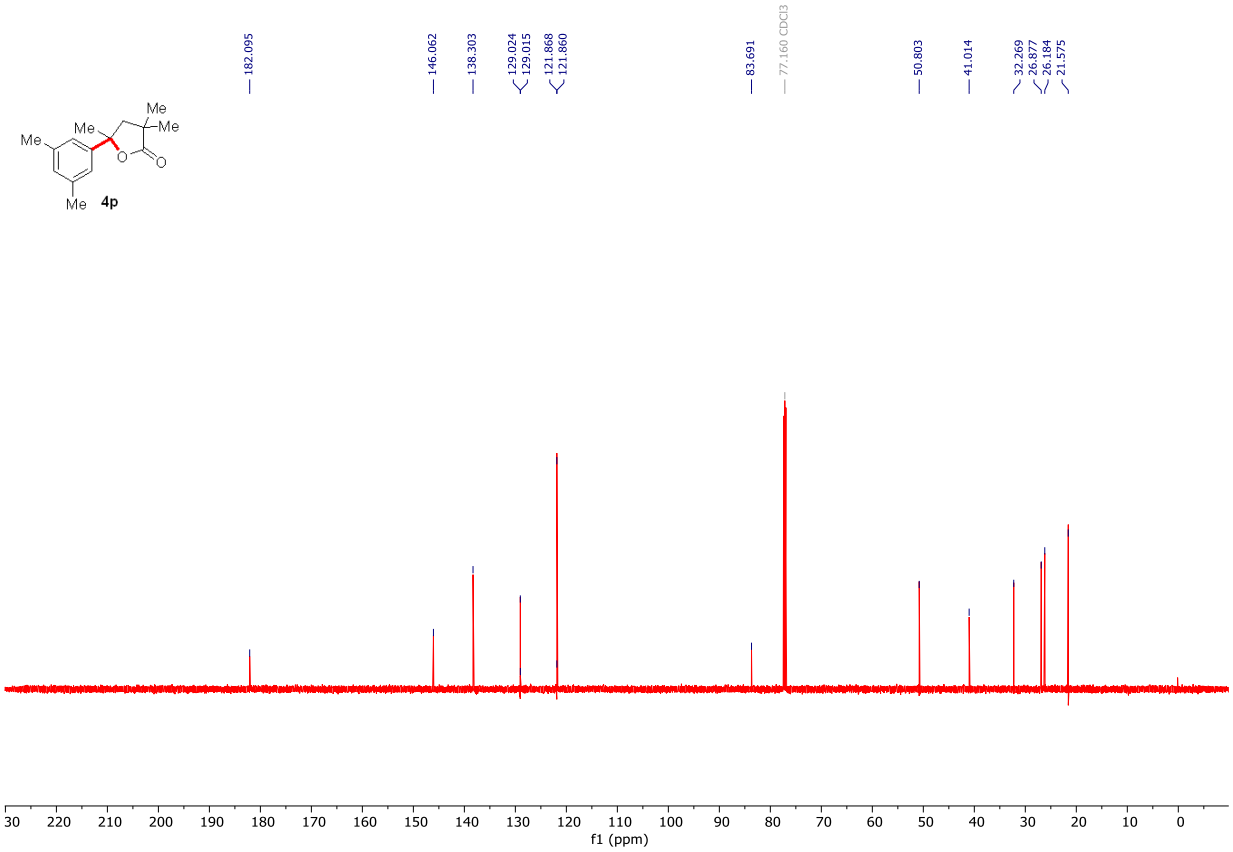
4o

182.090
143.526
136.950
135.744
129.869
125.321
121.472
83.636
77.160 CDCl3
50.758
41.030
32.313
26.859
26.150
20.076
19.457
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Me
Me
Me
Me
O
O
Me

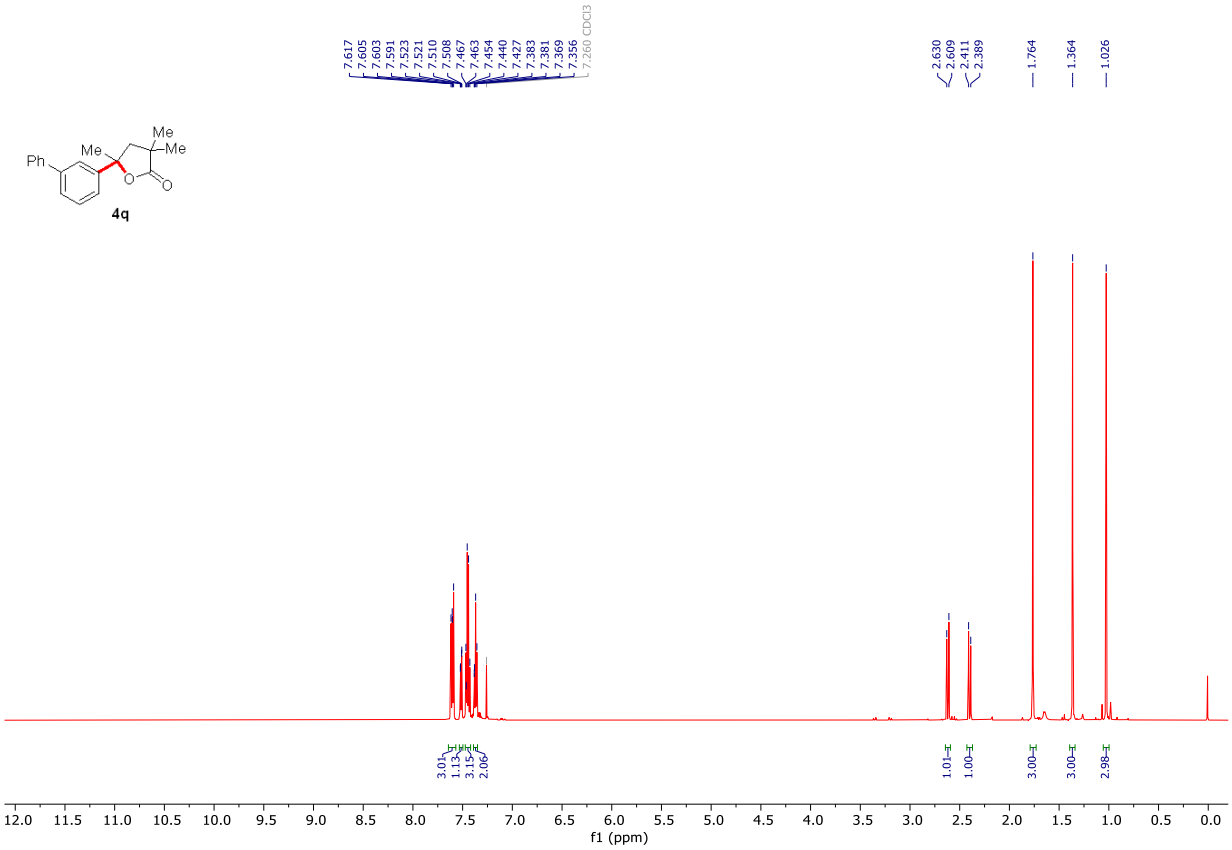
4p

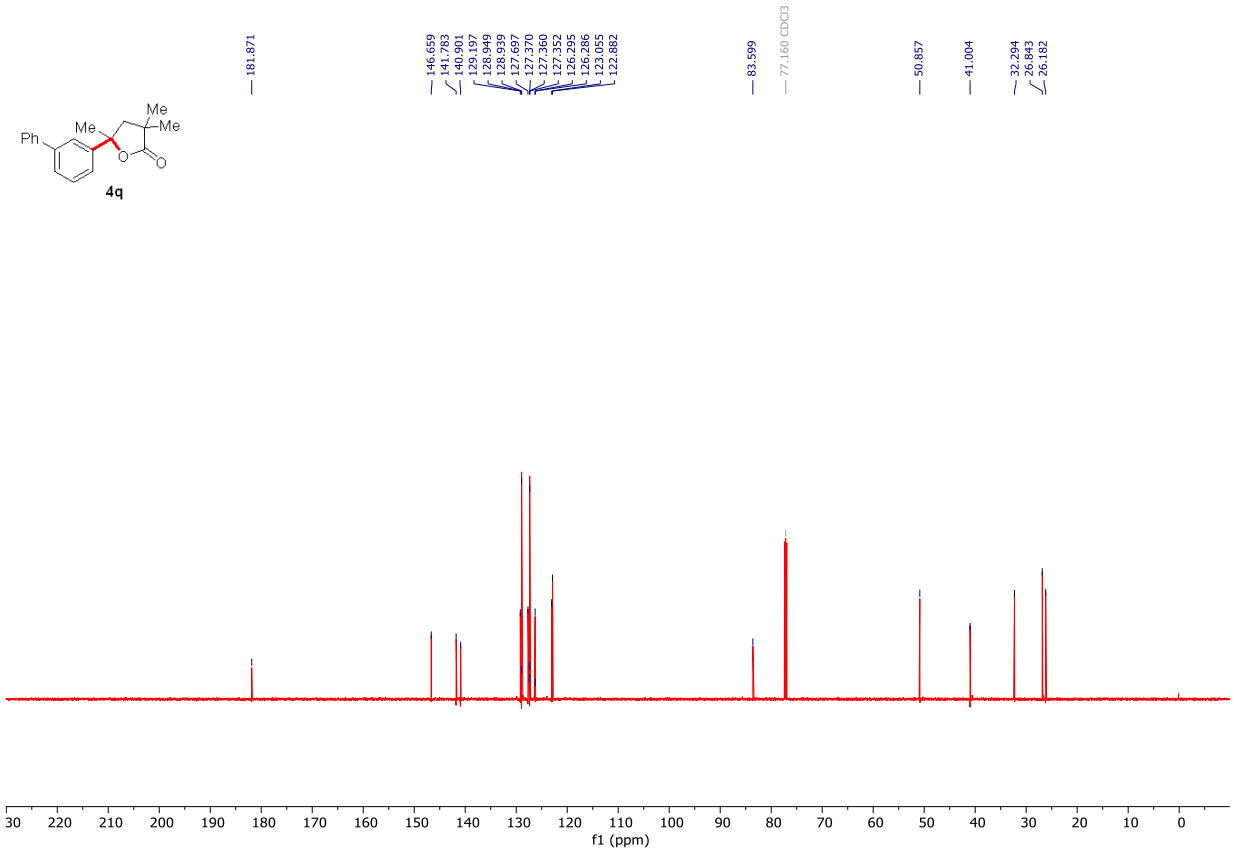
7.260 CDCl3
6.978
6.977
6.902
6.901
6.899
2.551
2.530
2.327
2.315
2.314
2.306
1.680
1.334
1.000
1.97
0.99
1.00
7.05
3.01
3.04
3.00
f1 (ppm)

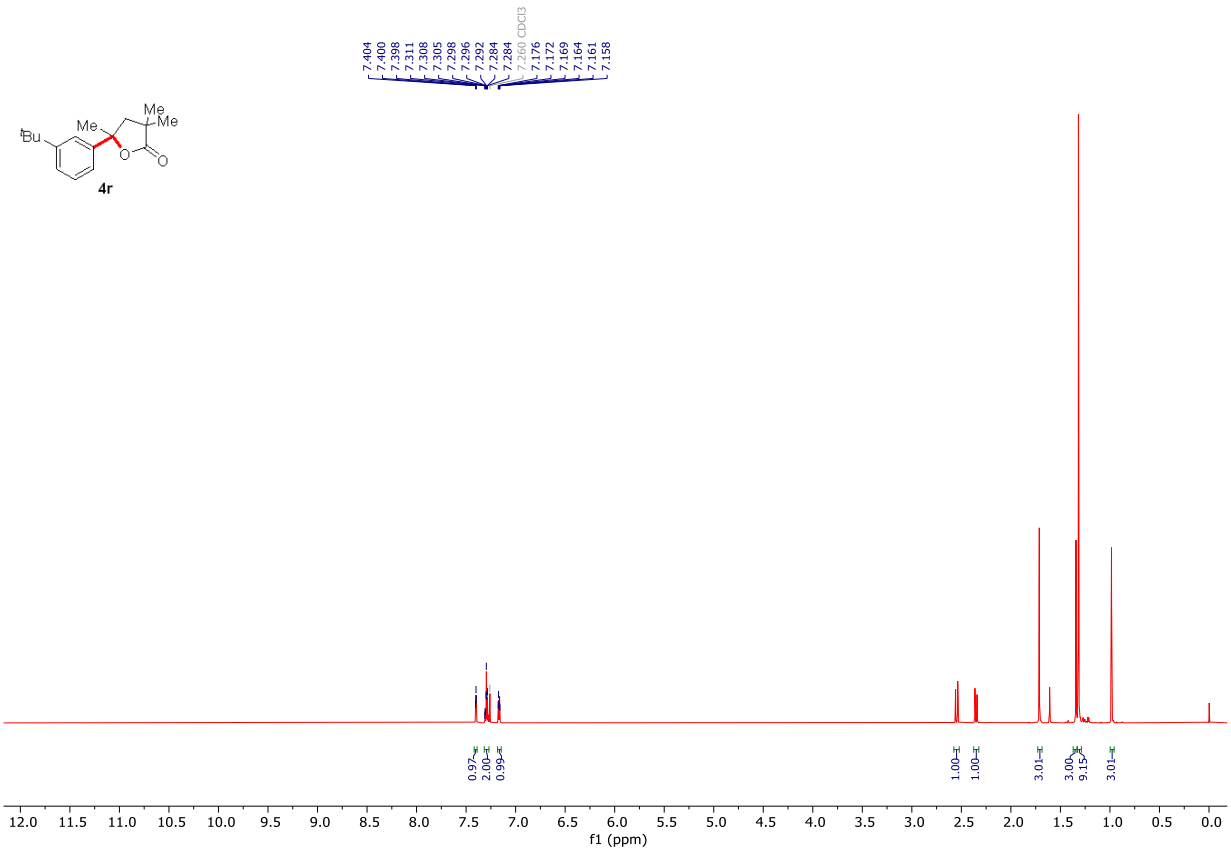

182.095
146.062
138.303
129.024
129.015
121.868
121.860
83.691
77.160 CDCl3
50.803
41.014
32.269
26.877
26.184
21.575
f1 (ppm)

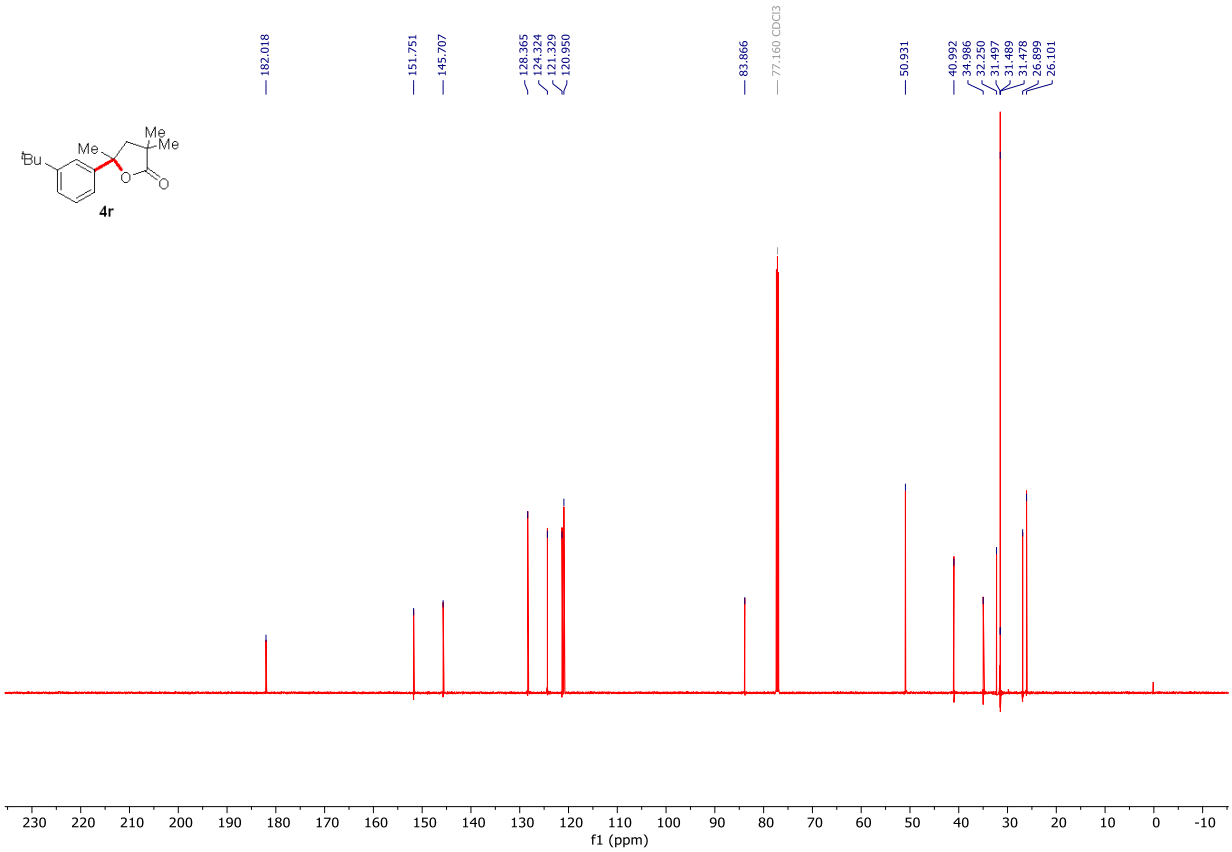
Me
Me
Me
Me
O
O
Me

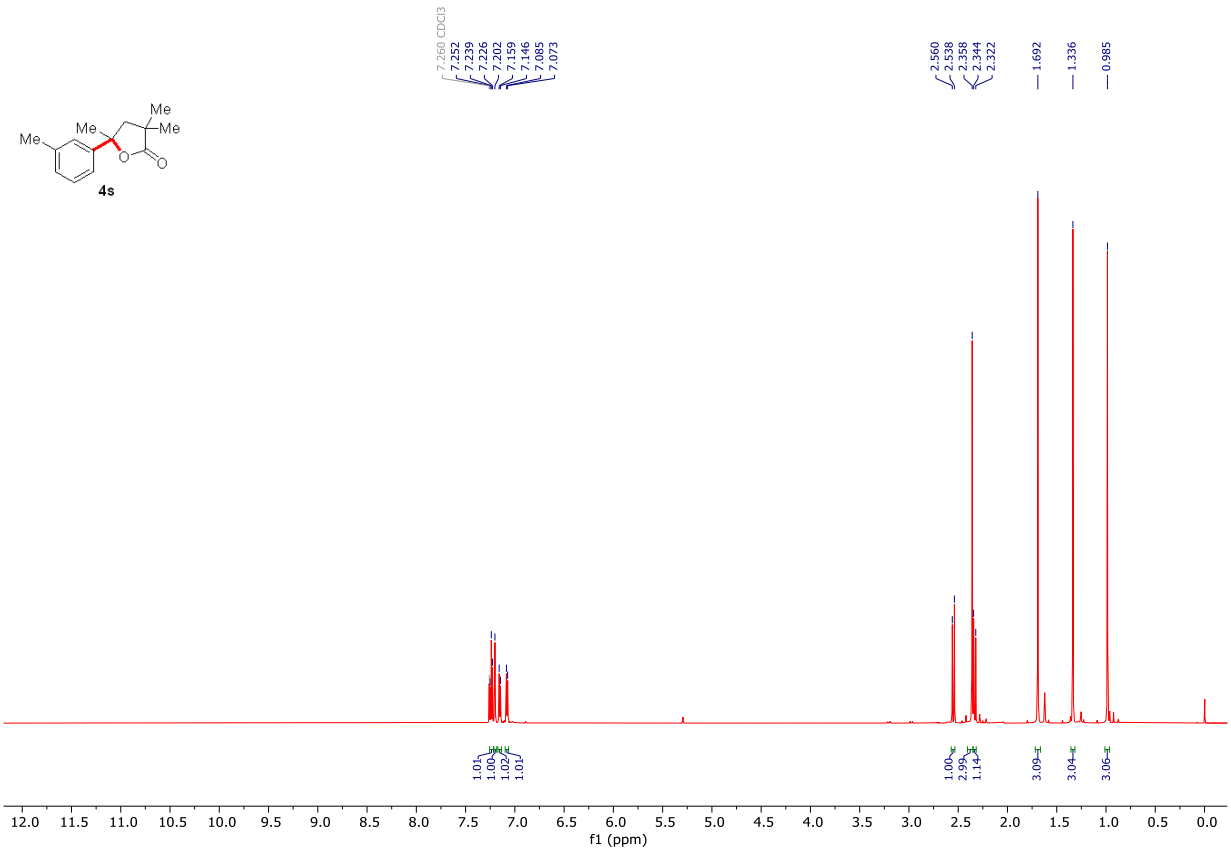
4p


7.617
7.605
7.603
7.591
7.523
7.521
7.510
7.508
7.467
7.463
7.454
7.440
7.427
7.383
7.381
7.369
7.356
7.260 CDCl3
2.630
2.609
2.411
2.389
1.764
1.364
1.026
Me
Me
Me
Ph
O
O
4q
3.01
1.13
3.15
2.06
1.01
1.00
3.00
3.00
2.98
12.0 11.5 11.0 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
f1 (ppm)

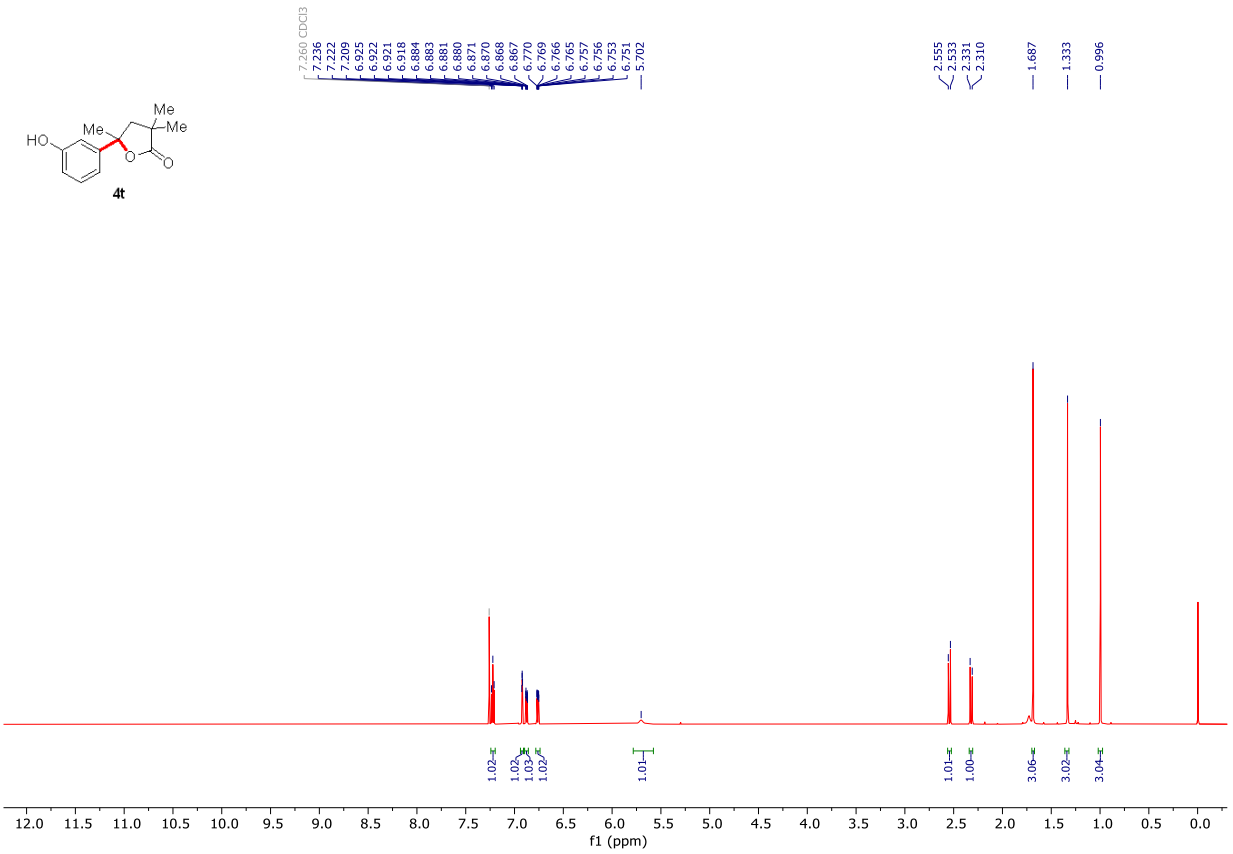

181.871
146.659
141.783
140.901
129.197
128.949
128.939
127.697
127.370
127.360
127.352
126.295
126.286
123.055
122.882
83.599
77.160 CDCl3
50.857
41.004
32.294
26.843
26.182
Me
Me
Me
Ph
O
O
4q
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

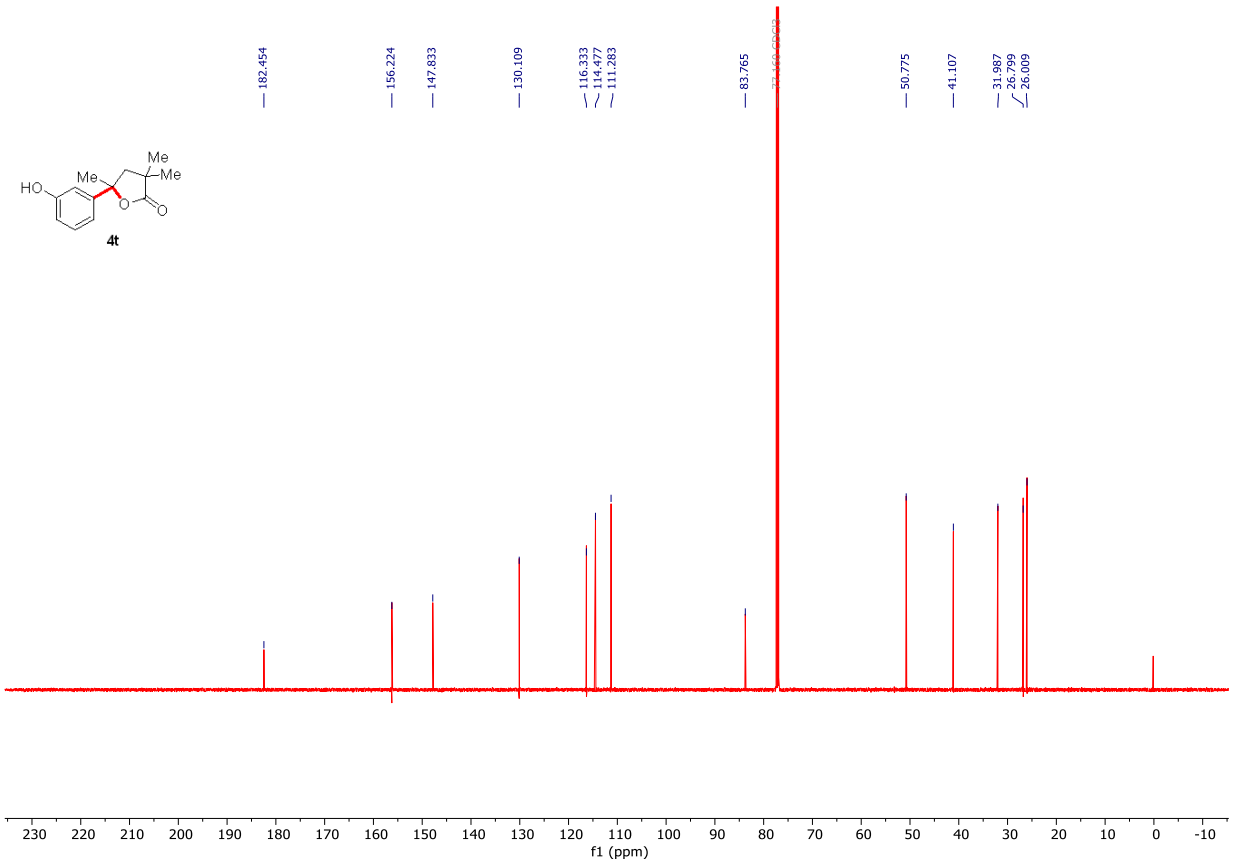

7.404
7.400
7.398
7.311
7.308
7.305
7.298
7.296
7.292
7.284
7.284
7.260 CDCl3
7.176
7.172
7.169
7.164
7.161
7.158
Me
Me
Me
tBu
O
O
4r
0.97
2.00
0.99
1.00
1.00
3.01
3.00
9.15
3.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

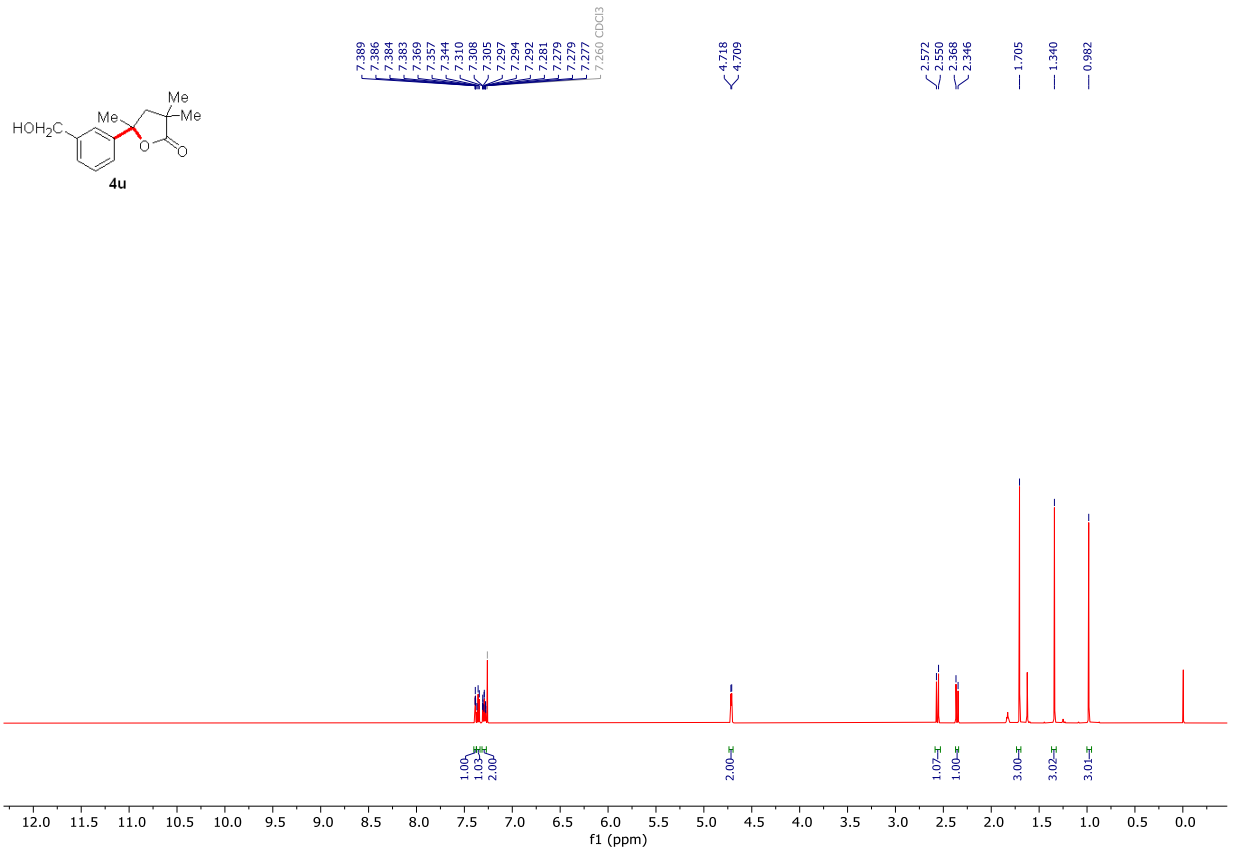

182.018
151.751
145.707
128.365
124.324
121.329
120.950
83.866
77.160 CDCl3
50.931
40.992
34.986
32.250
31.497
31.489
31.478
26.899
26.101
Me
Me
Me
tBu
O
O
4r
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

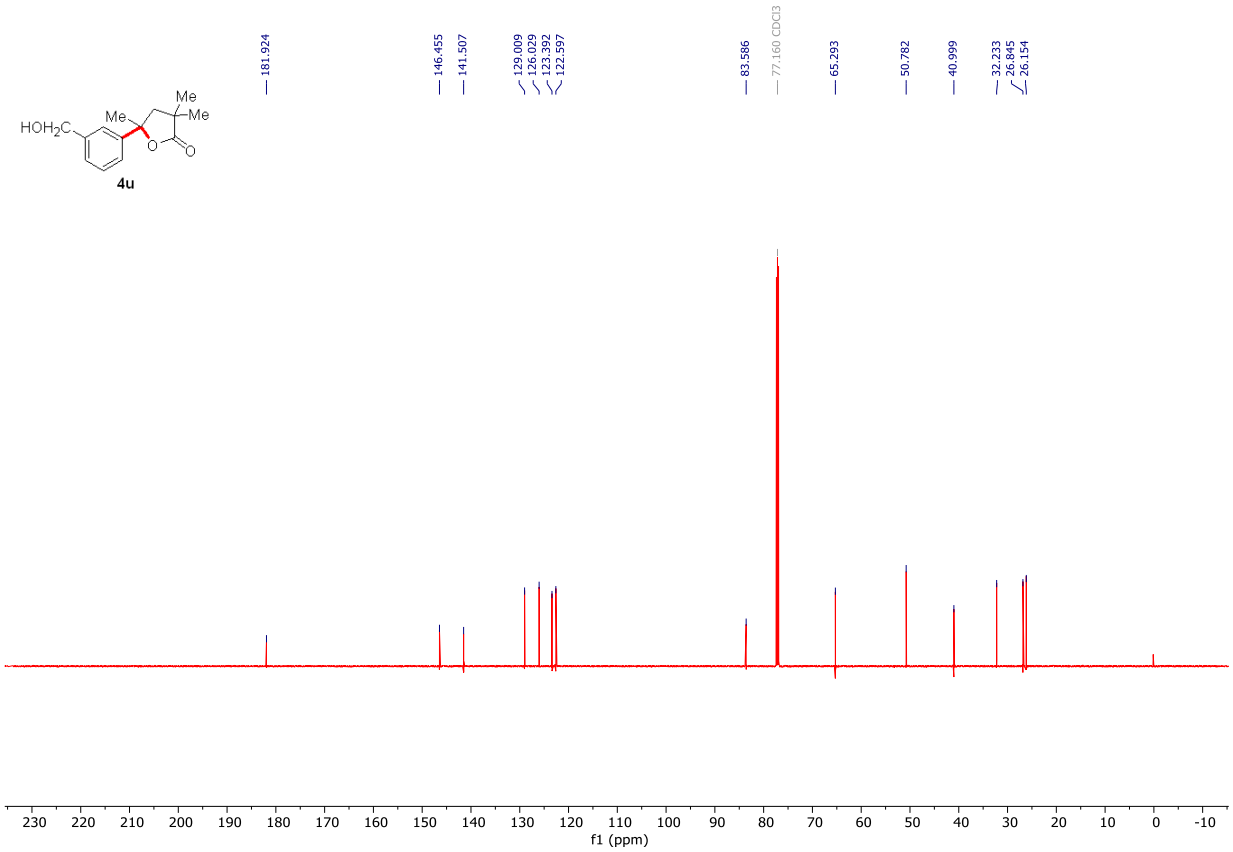

7.260 CDCl3
7.252
7.239
7.226
7.202
7.159
7.146
7.085
7.073
2.560
2.538
2.358
2.344
2.322
1.692
1.336
0.985
Me
Me
Me
Me
O
O
4s
1.01
1.00
1.02
1.01
1.00
2.99
1.14
3.09
3.04
3.06
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

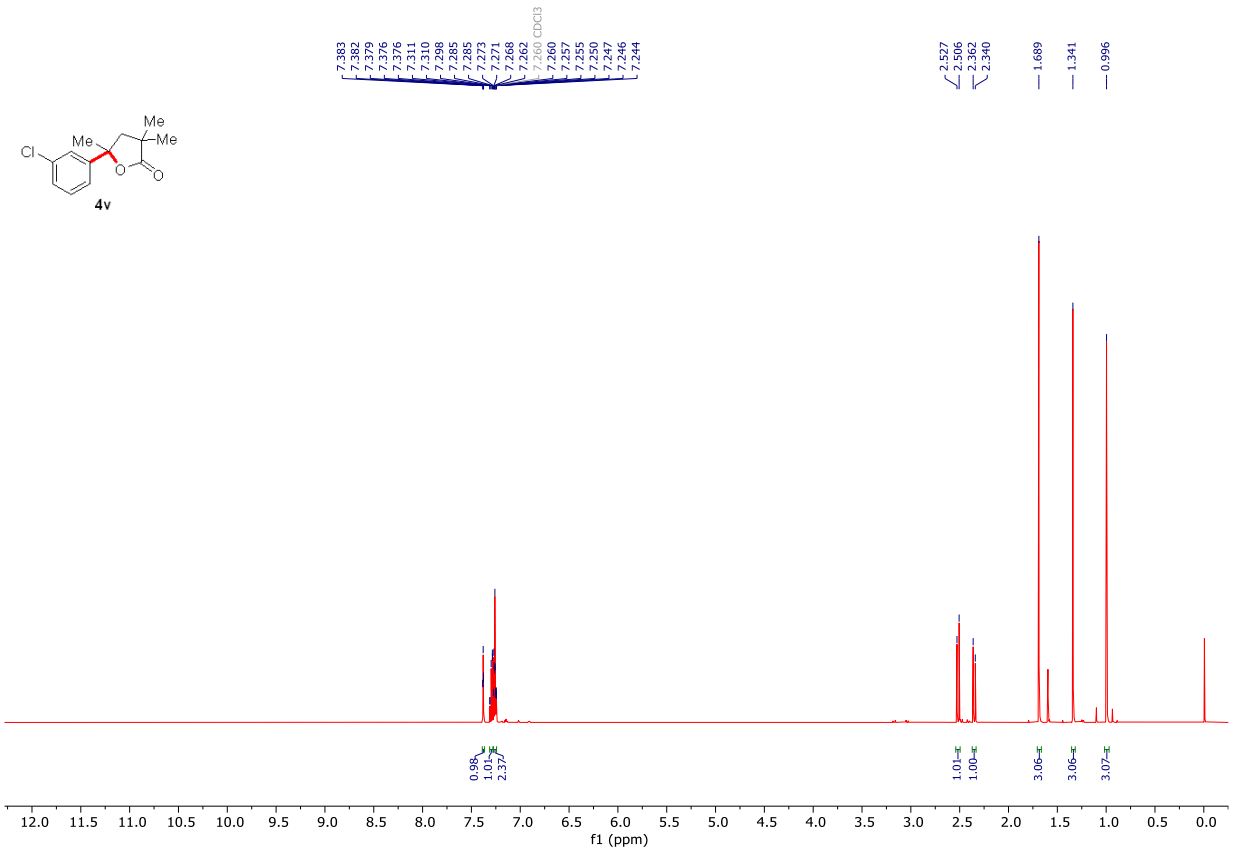

182.006
146.022
138.444
128.598
128.165
124.748
121.172
83.631
77.160 CDCl3
50.803
40.995
32.228
26.852
26.107
21.695
Me
Me
Me
Me
O
O
4s
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

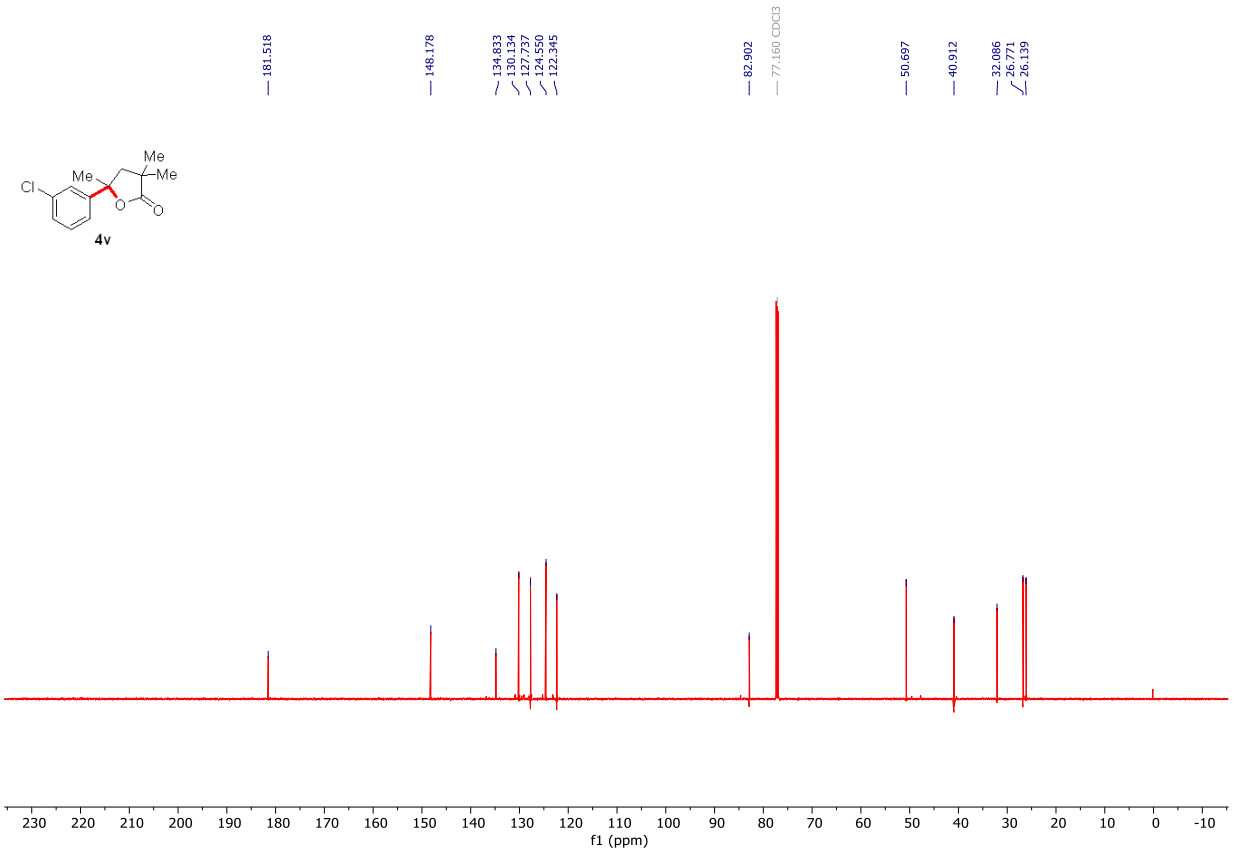

7.260 CDCl3
7.236
7.222
7.209
6.925
6.922
6.921
6.918
6.884
6.883
6.881
6.880
6.871
6.870
6.868
6.867
6.770
6.769
6.766
6.765
6.757
6.756
6.753
6.751
5.702
2.555
2.533
2.331
2.310
1.687
1.333
0.996
Me
Me
Me
HO
O
O
4t
1.02
1.02
1.03
1.02
1.01
1.01
1.00
3.06
3.02
3.04
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 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
f1 (ppm)

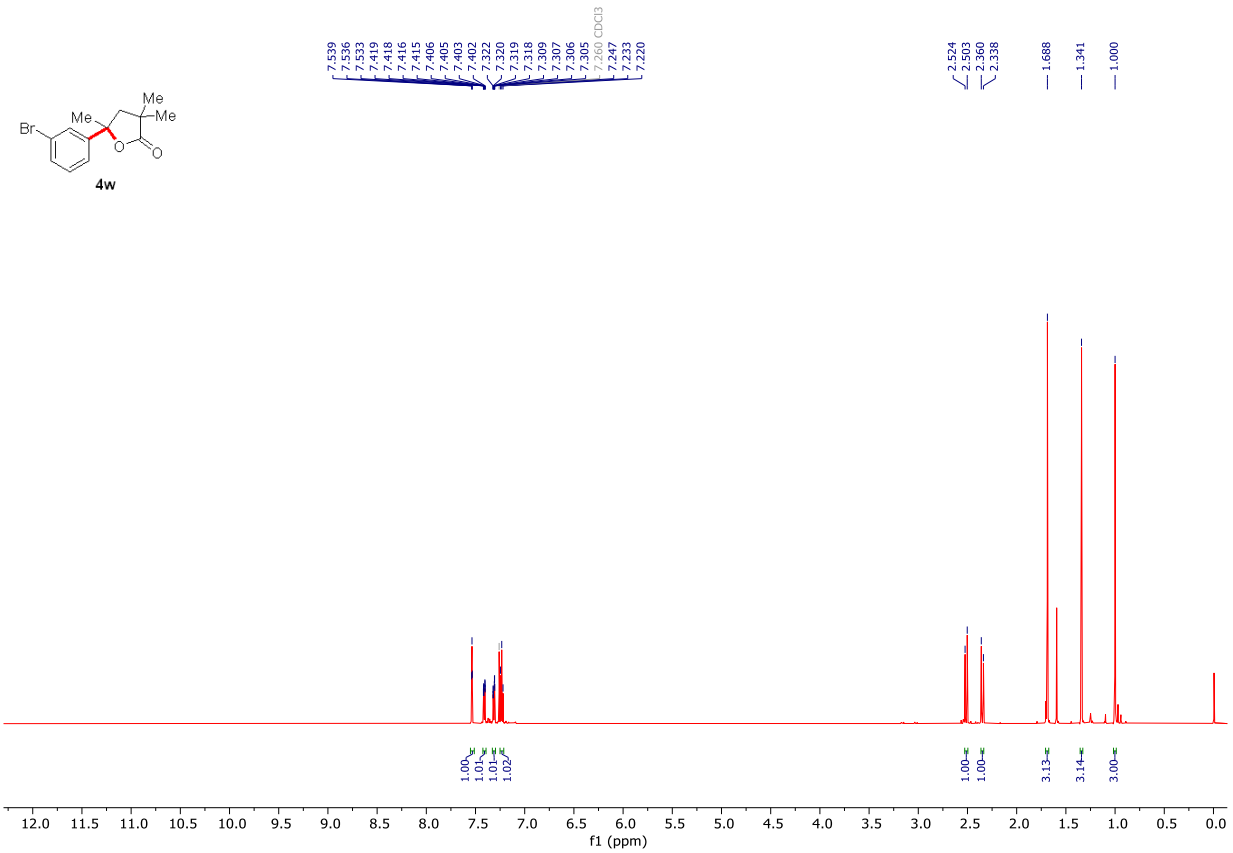

182.454
156.224
147.833
130.109
116.333
114.477
111.283
83.765
77.160 CDCl3
50.775
41.107
31.987
26.799
26.009
Me
Me
Me
HO
O
O
4t
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

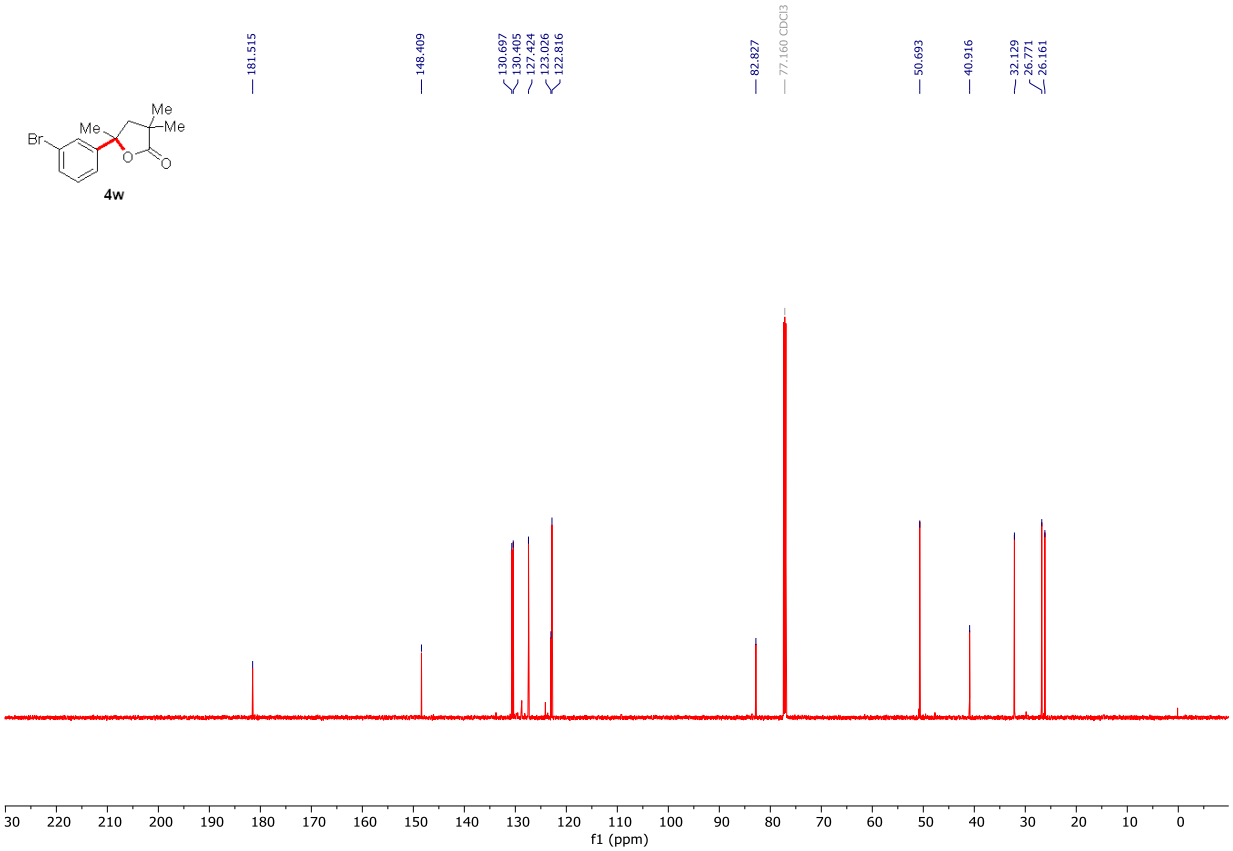

7.389
7.386
7.384
7.383
7.369
7.357
7.344
7.310
7.308
7.305
7.297
7.294
7.292
7.281
7.279
7.279
7.277
7.260 CDCl3
4.718
4.709
2.572
2.550
2.368
2.346
1.705
1.340
0.982
HOH2C
Me
Me
Me
O
O
4u
1.00
1.03
2.00
2.00
1.07
1.00
3.00
3.02
3.01
f1 (ppm)

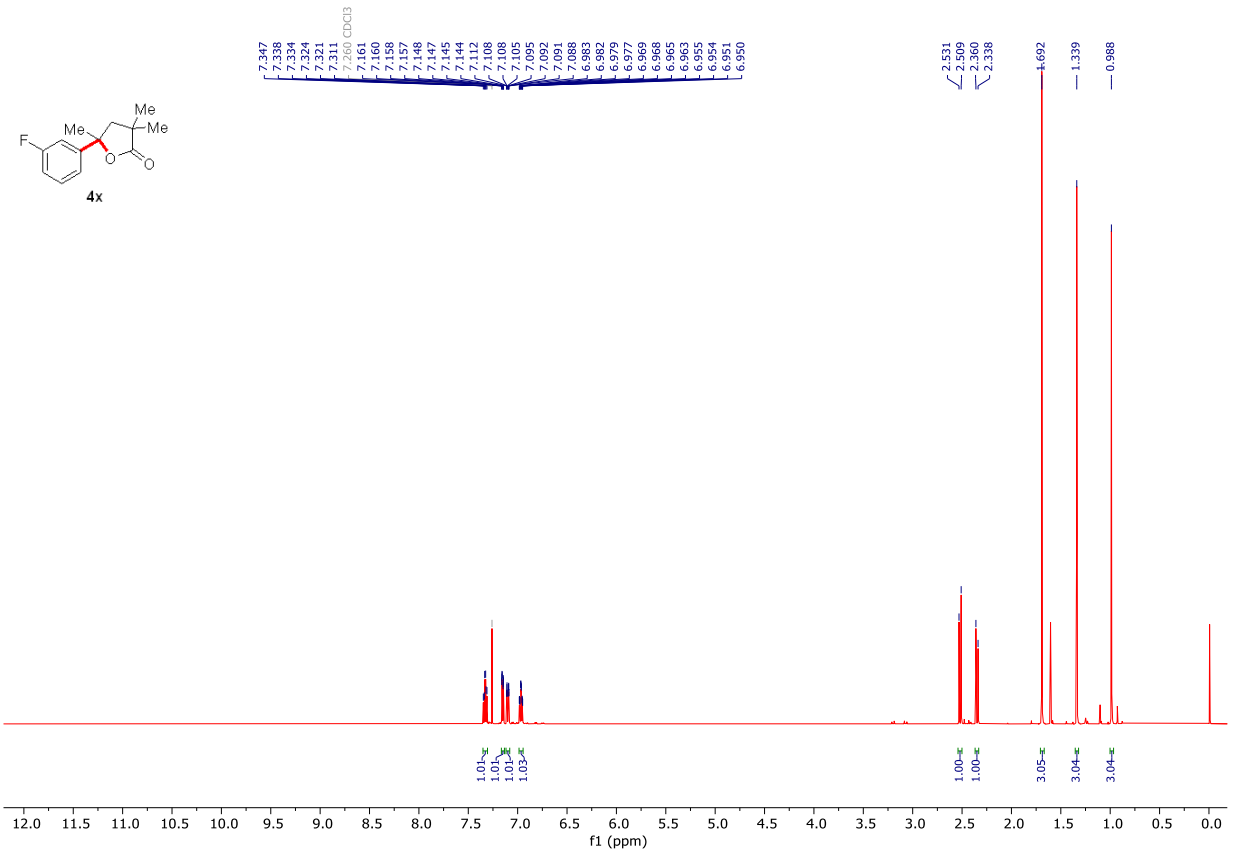

181.924
146.455
141.507
129.009
126.029
123.392
122.597
83.586
77.160 CDCl3
65.293
50.782
40.999
32.233
26.845
26.154
HOH2C
Me
Me
Me
O
O
4u
f1 (ppm)


7.383
7.382
7.379
7.376
7.376
7.311
7.310
7.298
7.285
7.285
7.273
7.271
7.268
7.262
7.260 CDCl3
7.257
7.255
7.250
7.247
7.246
7.244
2.527
2.506
2.362
2.340
1.689
1.341
0.996
Me
Me
Me
Cl
O
O
4v
0.98
1.01
2.37
1.01
1.00
3.06
3.06
3.07
12.0 11.5 11.0 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
f1 (ppm)

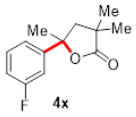

181.518
148.178
134.833
130.134
127.737
124.550
122.345
82.902
77.160 CDCl3
50.697
40.912
32.086
26.771
26.139
Me
Me
Me
Cl
O
O
4v
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.539
7.536
7.533
7.419
7.418
7.416
7.415
7.406
7.405
7.403
7.402
7.322
7.320
7.319
7.318
7.309
7.307
7.306
7.305
7.260 CDCl3
7.247
7.233
7.220
2.524
2.503
2.360
2.338
1.688
1.341
1.000
Me
Me
Me
Br
O
O
4w
1.00
1.01
1.01
1.02
1.00
1.00
3.13
3.14
3.00
12.0 11.5 11.0 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
f1 (ppm)

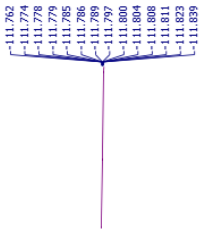

181.515
148.409
130.697
130.405
127.424
123.026
122.816
82.827
77.160 CDCl3
50.693
40.916
32.129
26.771
26.161
Me
Me
Me
Br
O
O
4w
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

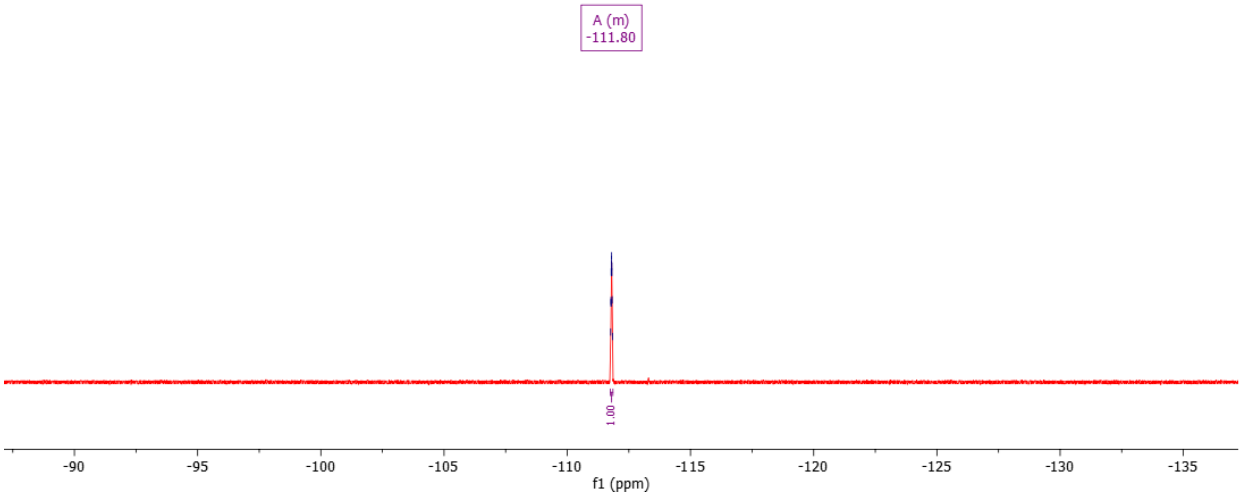

7.347
7.338
7.334
7.324
7.321
7.311
7.260 CDCl3
7.161
7.160
7.158
7.157
7.148
7.147
7.145
7.144
7.112
7.108
7.108
7.105
7.095
7.092
7.091
7.088
6.983
6.982
6.979
6.977
6.969
6.968
6.965
6.963
6.955
6.954
6.951
6.950
2.531
2.509
2.360
2.338
1.692
1.339
0.988
Me
Me
Me
F
O
O
4x
1.01
1.01
1.01
1.03
1.00
1.00
3.05
3.04
3.04
f1 (ppm)

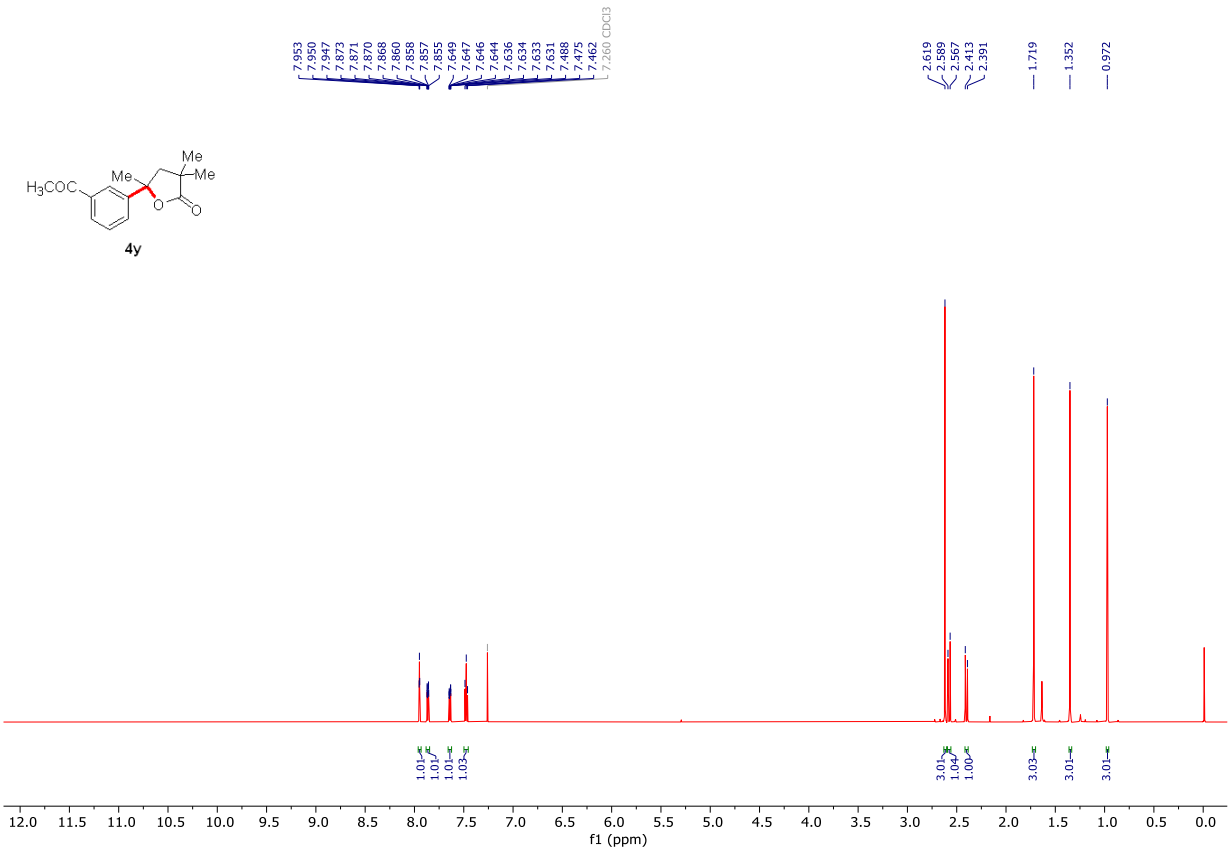

181.571
163.862
162.227
148.791
148.746
130.483
130.429
119.797
119.777
114.509
114.369
111.654
111.501
82.965
82.952
77.160 CDCl3
50.762
40.926
32.014
26.775
26.077
Me
Me
Me
F
O
O
4x
f1 (ppm)

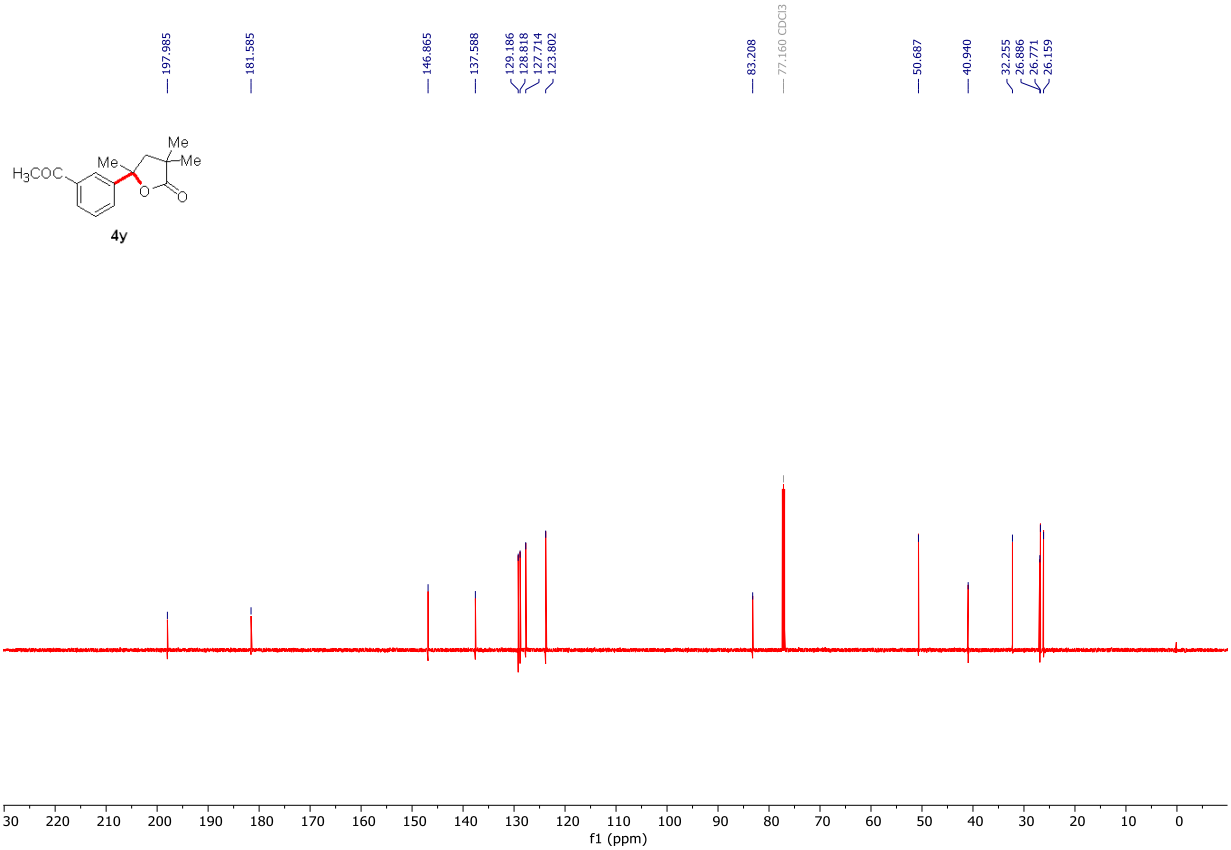

Me
Me
Me
O
O
F

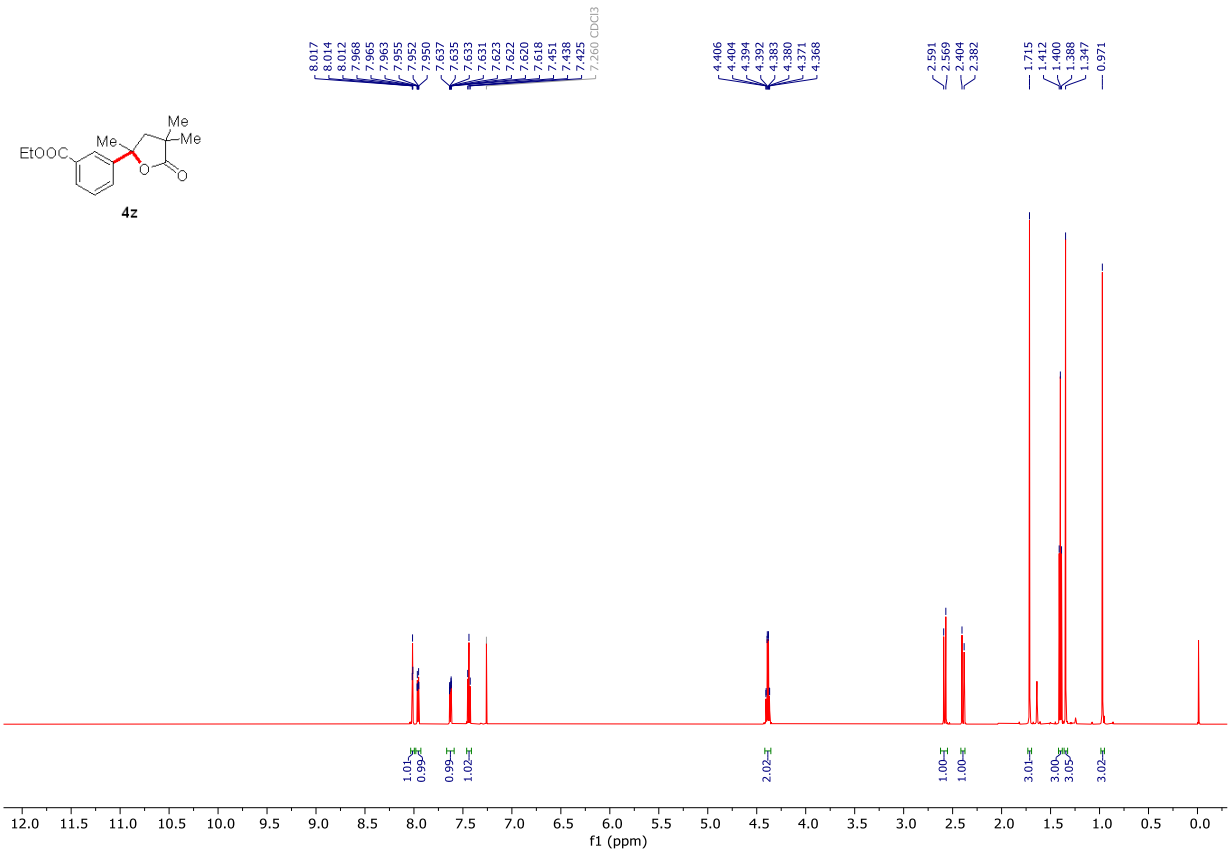
4x


-111.762
-111.774
-111.778
-111.779
-111.785
-111.786
-111.789
-111.797
-111.800
-111.804
-111.808
-111.811
-111.823
-111.839

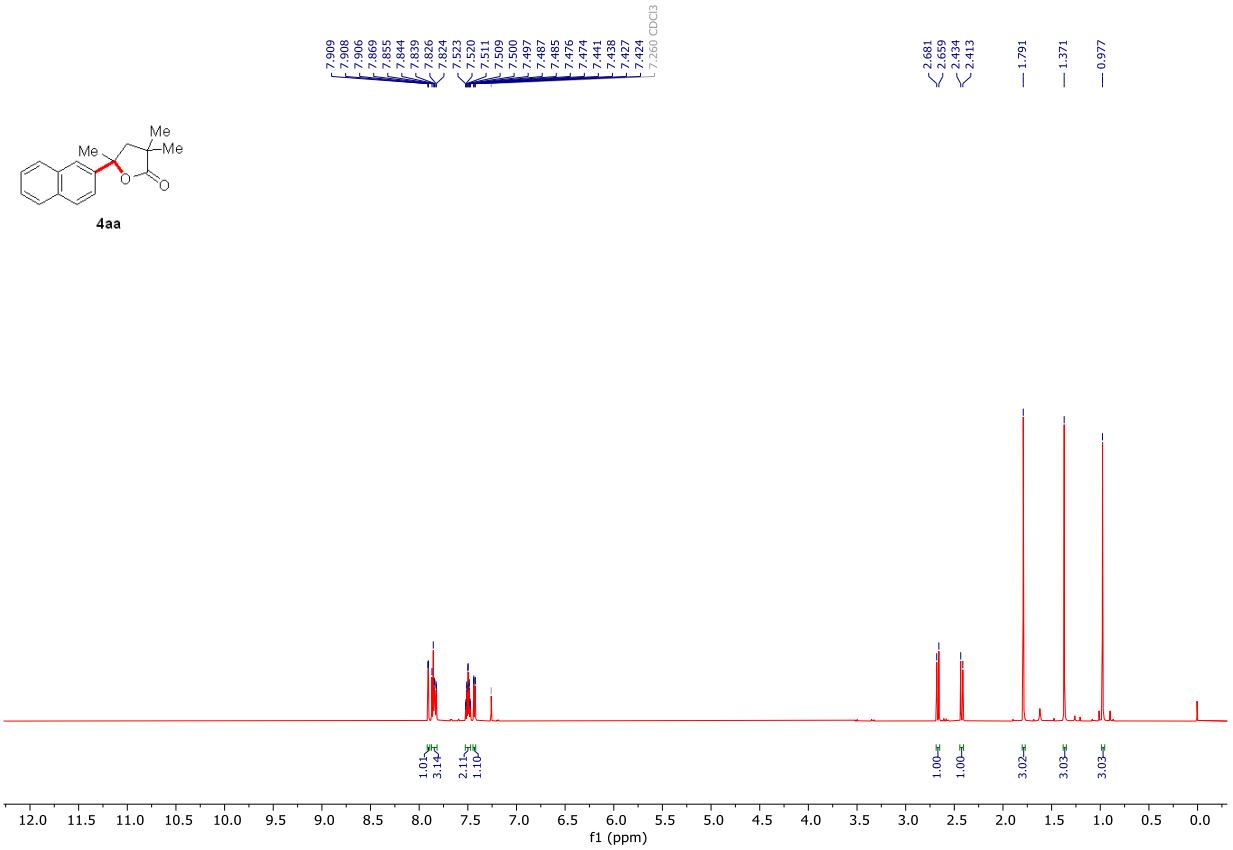

A (m)
-111.80
1.00
-90
-95
-100
-105
-110
-115
-120
-125
-130
-135
f1 (ppm)

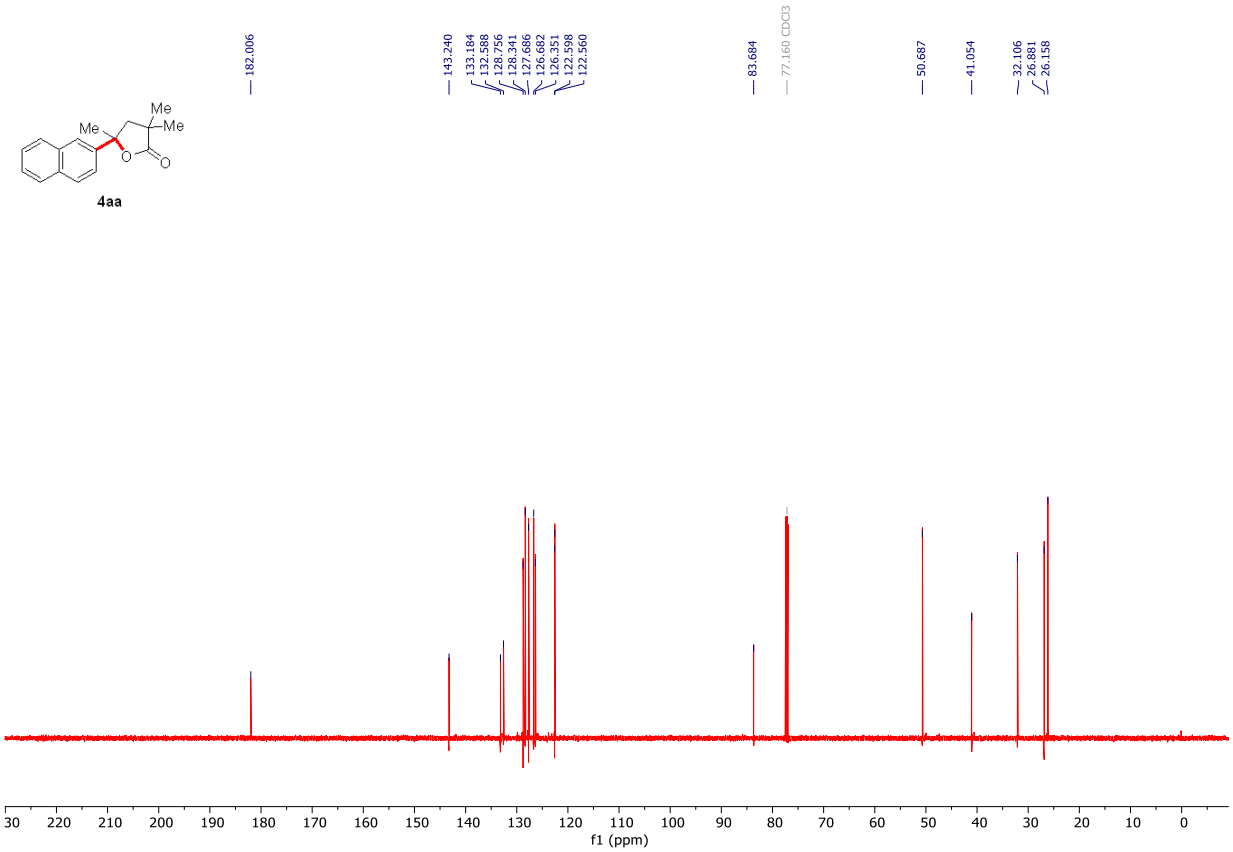

7.953
7.950
7.947
7.873
7.871
7.870
7.868
7.860
7.858
7.857
7.855
7.649
7.647
7.646
7.644
7.636
7.634
7.633
7.631
7.488
7.475
7.462
7.260 CDCl3
2.619
2.589
2.567
2.413
2.391
1.719
1.352
0.972
Me
Me
Me
H3COC
O
O
4y
1.01
1.01
1.01
1.03
3.01
1.04
1.00
3.03
3.01
3.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 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
f1 (ppm)

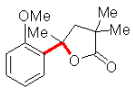

197.985
181.585
146.865
137.588
129.186
128.818
127.714
123.802
83.208
77.160 CDCl3
50.687
40.940
32.255
26.886
26.771
26.159
Me
Me
Me
H3COC
O
O
4y
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.017
8.014
8.012
7.968
7.965
7.963
7.955
7.952
7.950
7.637
7.635
7.633
7.631
7.623
7.622
7.620
7.618
7.451
7.438
7.425
7.260 CDCl3
4.406
4.404
4.394
4.392
4.383
4.380
4.371
4.368
2.591
2.569
2.404
2.382
1.715
1.412
1.400
1.388
1.347
0.971
Me
Me
Me
EtOOC
O
O
4z
1.01
0.99
0.99
1.02
2.02
1.00
1.00
3.01
3.00
3.05
3.02
f1 (ppm)


181.614
166.429
146.528
131.115
128.916
128.728
128.502
125.317
83.223
77.160 CDCl3
61.356
50.695
40.939
32.245
26.763
26.128
14.475
Me
Me
Me
EtOOC
O
O
4z
f1 (ppm)


7.909
7.908
7.906
7.869
7.855
7.844
7.839
7.826
7.824
7.523
7.520
7.511
7.509
7.500
7.487
7.485
7.476
7.441
7.438
7.427
7.424
7.260 CDCl3
2.681
2.659
2.434
2.413
1.791
1.371
0.977
Me
Me
Me
O
O
4aa
1.01
3.14
2.11
1.10
1.00
1.00
3.02
3.03
3.03
12.0 11.5 11.0 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
f1 (ppm)


182.006
143.240
133.184
132.588
128.756
128.341
127.686
126.682
126.351
122.598
122.360
83.684
77.160 CDCl3
50.687
41.054
32.106
26.881
26.158
Me
Me
Me
O
O
4aa
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


OMe
Me
Me
Me
O
O

4ab

7.513
7.510
7.500
7.497
7.286
7.283
7.273
7.272
7.270
7.269
7.260 CDCl3
7.260
7.257
6.964
6.962
6.951
6.950
6.939
6.937
6.916
6.914
6.902
6.900
3.858
2.665
2.642
2.343
2.320
1.752
1.331
1.011
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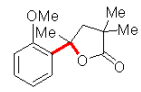
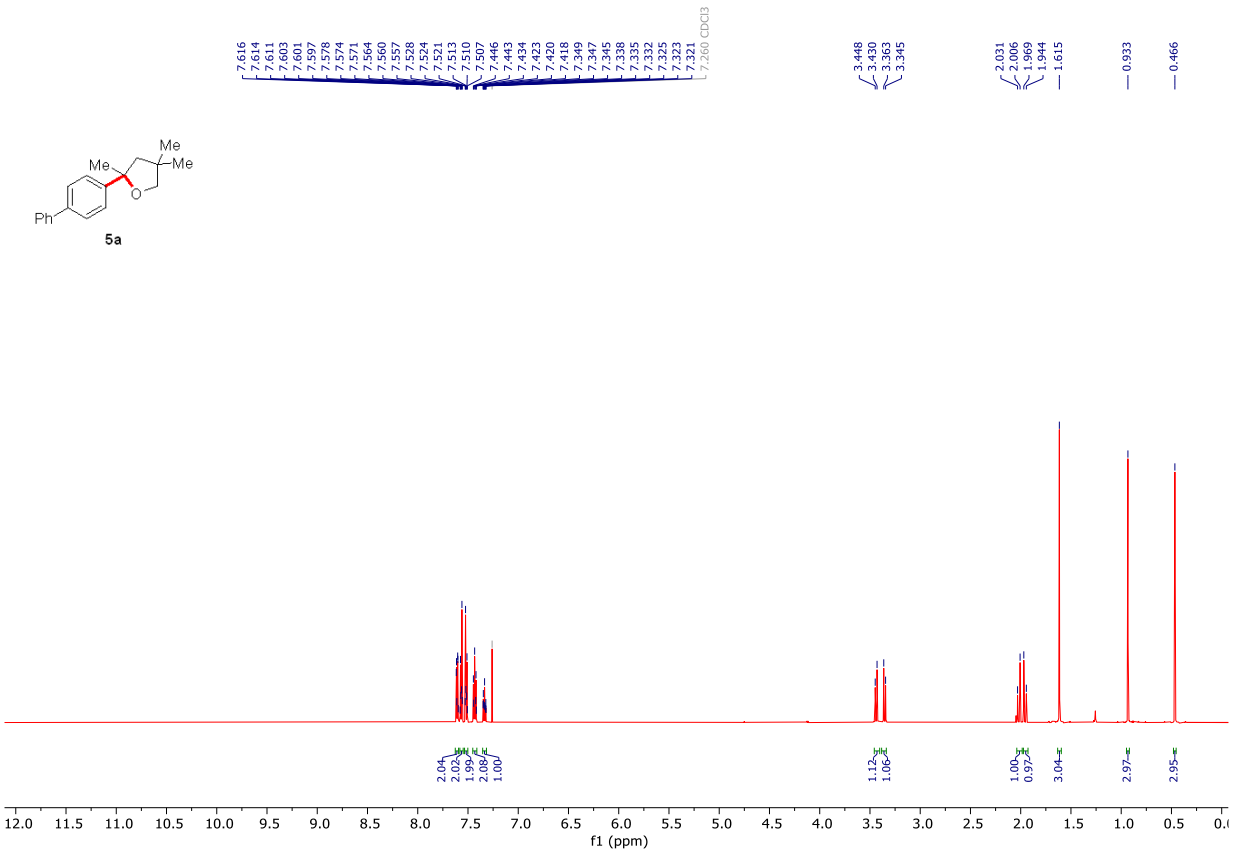
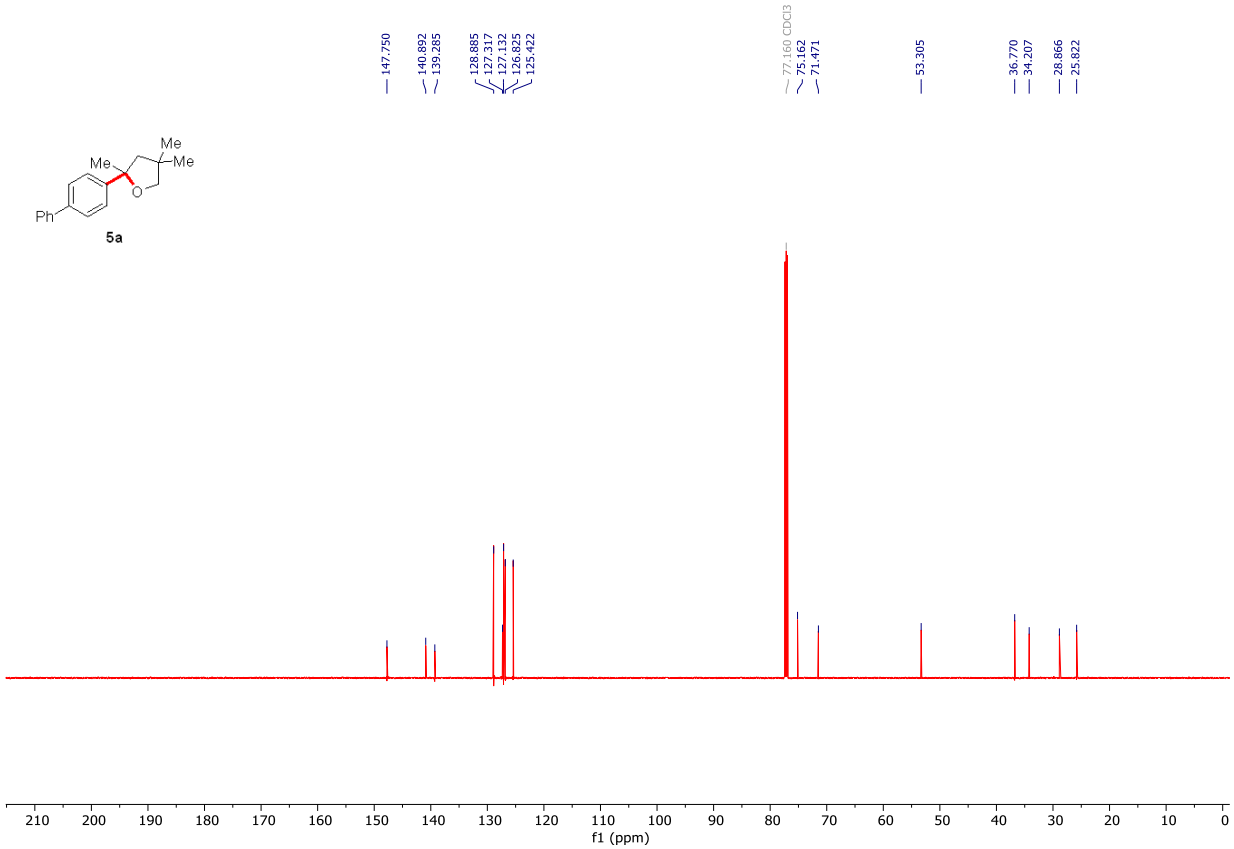
1.01
1.00
3.03
3.03
3.04
f1 (ppm)

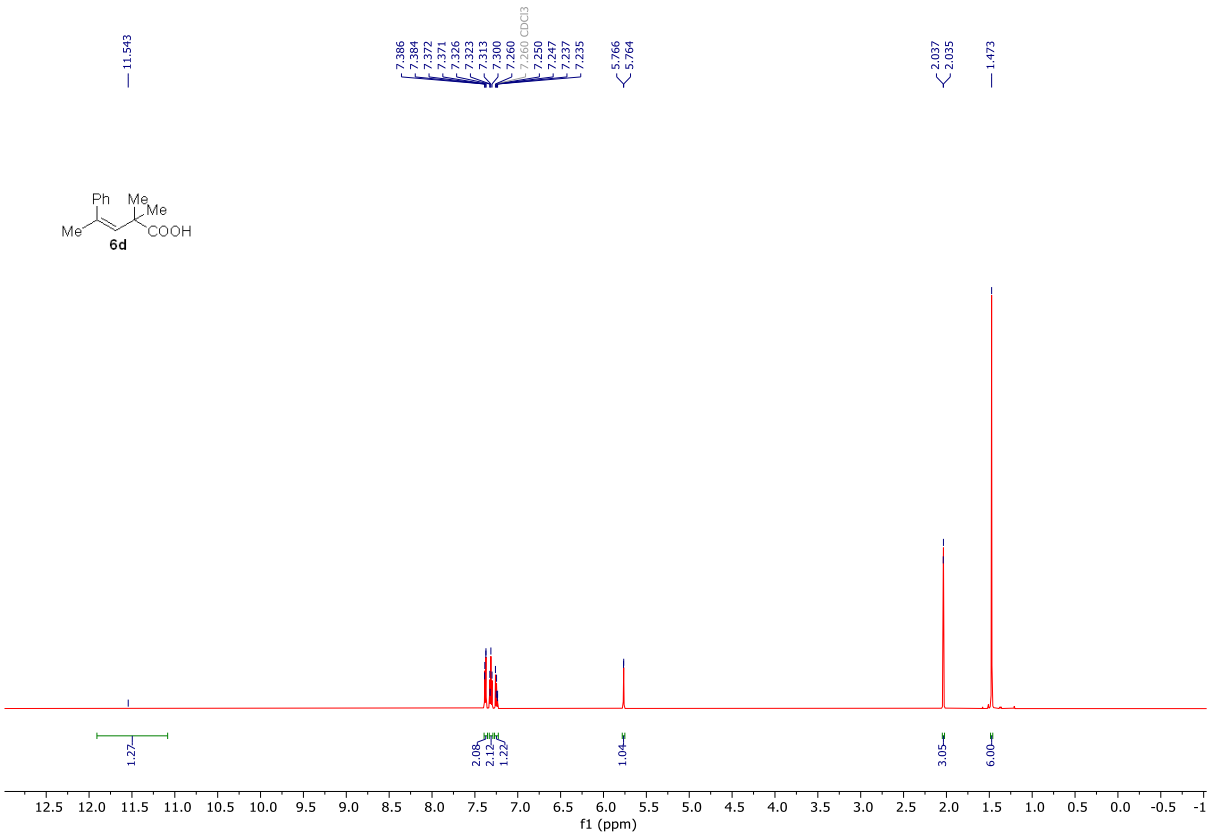

OMe
Me
Me
Me
O
O

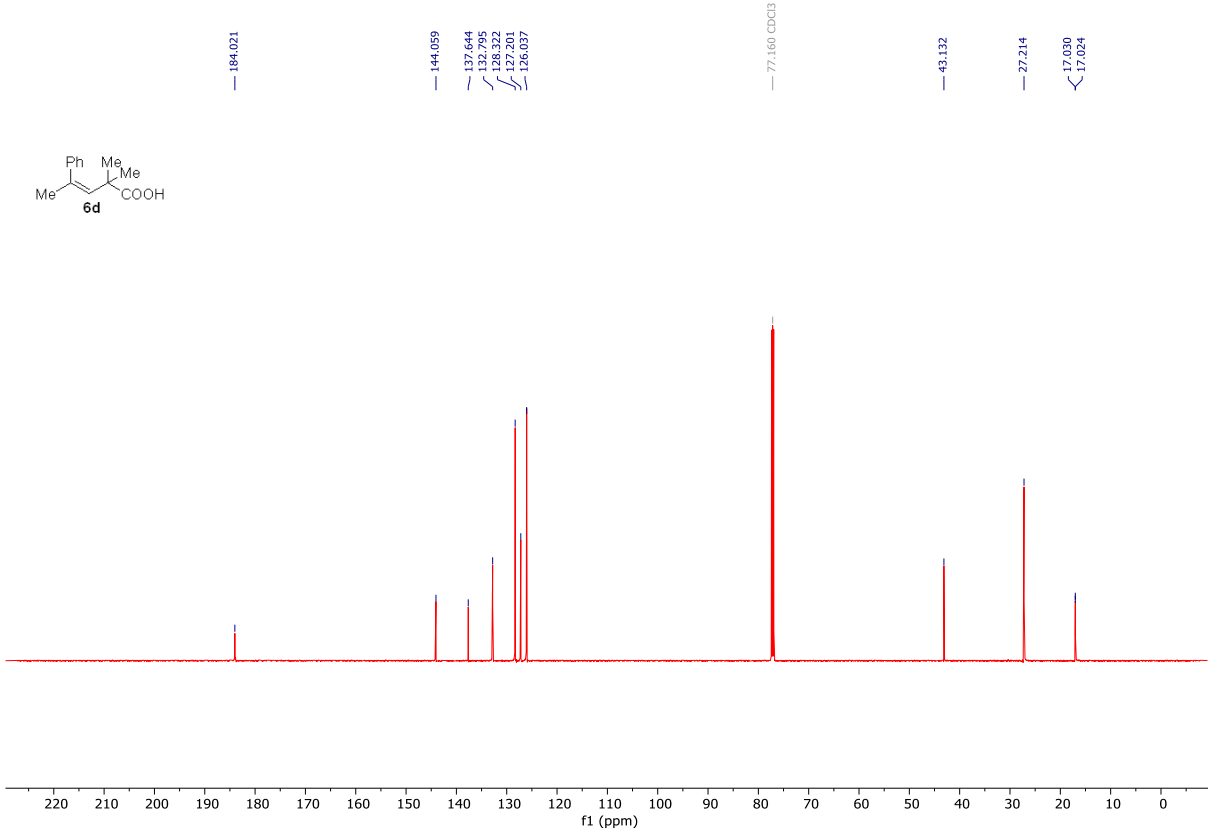
4ab

182.427
155.187
133.705
128.898
125.214
120.866
111.341
83.211
77.160 CDCl3
55.282
49.366
40.559
28.850
27.188
26.445
f1 (ppm)


7.616
7.614
7.611
7.603
7.601
7.597
7.578
7.574
7.571
7.564
7.560
7.557
7.528
7.524
7.521
7.513
7.510
7.507
7.446
7.443
7.434
7.423
7.420
7.418
7.349
7.347
7.345
7.338
7.335
7.332
7.325
7.323
7.321
7.260 CDCl3
3.448
3.430
3.363
3.345
2.031
2.006
1.969
1.944
1.615
0.933
0.466
Me
Me
Me
O
Ph
5a
2.04
2.02
1.99
2.08
1.00
1.12
1.06
1.00
0.97
3.04
2.97
2.95
12.0 11.5 11.0 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
f1 (ppm)


147.750
140.892
139.285
128.885
127.317
127.132
126.825
125.422
77.160 CDCl3
75.162
71.471
53.305
36.770
34.207
28.866
25.822
Me
Me
Me
O
Ph
5a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


11.543
7.386
7.384
7.372
7.371
7.326
7.323
7.313
7.300
7.260
7.260 CDCl3
7.250
7.247
7.237
7.235
5.766
5.764
2.037
2.035
1.473
Ph
Me
Me
Me
COOH
6d
1.27
2.08
2.12
1.22
1.04
3.05
6.00
f1 (ppm)


184.021
144.059
137.644
132.795
128.322
127.201
126.037
77.160 CDCl3
43.132
27.214
17.030
17.024
Ph
Me
Me
Me
COOH
6d
f1 (ppm)