Electronic Supplementary Material (ESI) for RSC Advances.

Cobalt-catalyzed annulation of styrenes with α-bromoacetic acids

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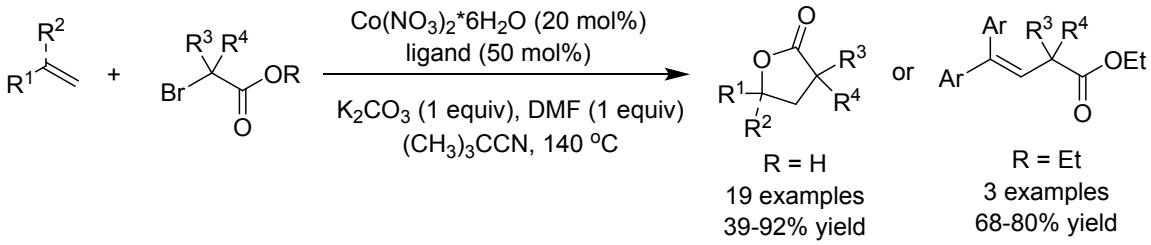


Supporting information

1. Materials and instrumentation

All reagents and starting materials were obtained commercially from Sigma-Aldrich, Acros and Merck, and were used as received without any further purification unless otherwise noted. Gas chromatographic (GC) analyses were performed using a Shimadzu GC 2010-Plus equipped with a flame ionization detector (FID) and an SPB-5 column (length = 30 m, inner diameter = 0.25 mm, and film thickness = 0.25 μm). GC-MS analyses were analyzed on a Shimadzu GCMS-QP2010Ultra with a ZB-5MS column (length = 30 m, inner diameter = 0.25 mm, and film thickness = 0.25 μm). The ^{1}H NMR and ^{13}C NMR were recorded on Bruker AV 500 spectrometers using residual solvent peak as a reference. Without notice, chemical shifts are reported in ppm and referenced to the residual peak for $CDCl_3$ (δ = 7.26 ppm for ^{1}H NMR and δ = 77.16 ppm for ^{13}C NMR). Splitting is reported with the following symbols: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, td = triplet of doublets, dt = doublet of triplets, and m = multiplet. Coupling constants (*J*) are reported in Hertz. HR-MS spectra were recorded by an Agilent HPLC 1200 Series coupled to Bruker micrOTOF-QII.

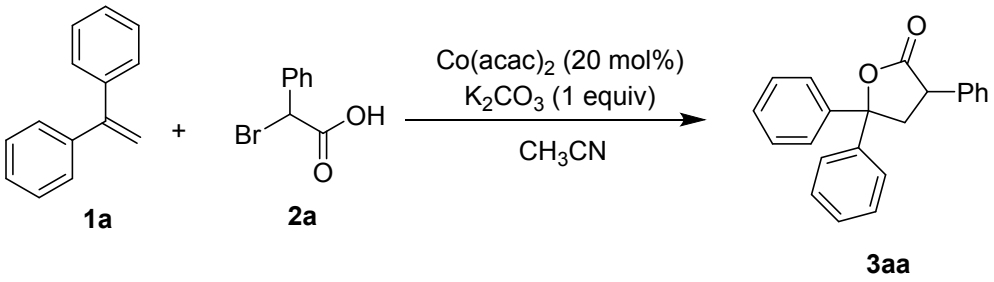
2. Optimization of reaction conditions

2.1. General procedure

To a 10 mL Schlenk tube equipped with a magnetic stir bar was added 1,1-diphenylethylene **1a** (0.1 mmol, 18 mg), α-bromophenylacetic acid **2a**, cobalt catalyst, ligand, base and dried solvent (1 mL). The reaction tube was flushed with argon for 5 min, capped, then placed into a preheated bath. After being cooled to room temperature, the crude mixture was added diphenyl ether (0.2 mmol, 34 mg) internal standard. An aliquot of the crude mixture was withdrawn and diluted with ethyl acetate (2 mL) and

brine (3 mL). The aqueous layer was extracted with ethyl acetate (2 mL x 3). The combined organic layers were washed with saturated $NaHCO_3$ (aqueous solution, 1 mL), dried over Na_2SO_4, filtered, and analyzed with GC with respect to the internal standard.

2.2. Effect of reaction temperature

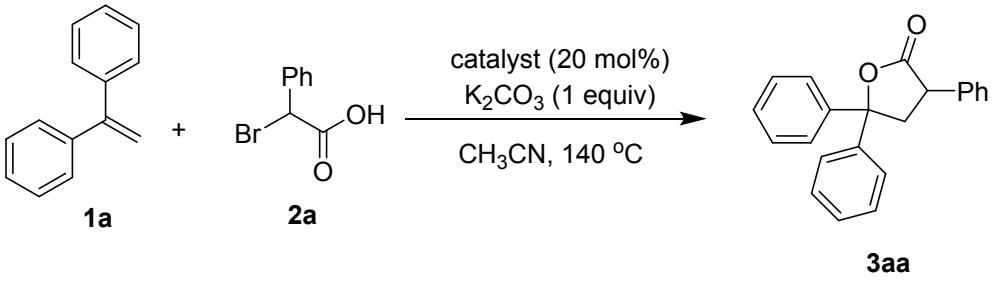


1a **2a** **3aa**

Entry	temperature, °C	yield of **3aa**, %
1	80	trace
2	100	12
3	120	37
4	**140**	**69**

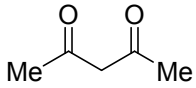
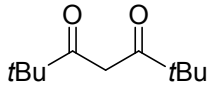
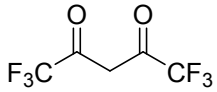
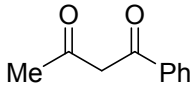
Reaction conditions: **2a** (0.2 mmol), $Co(acac)_2$ (0.02 mmol), K_2CO_3 (0.1 mmol), CH_3CN (1 mL), under argon, for 24 h. Yields are GC yields.

2.3. Effect of catalyst



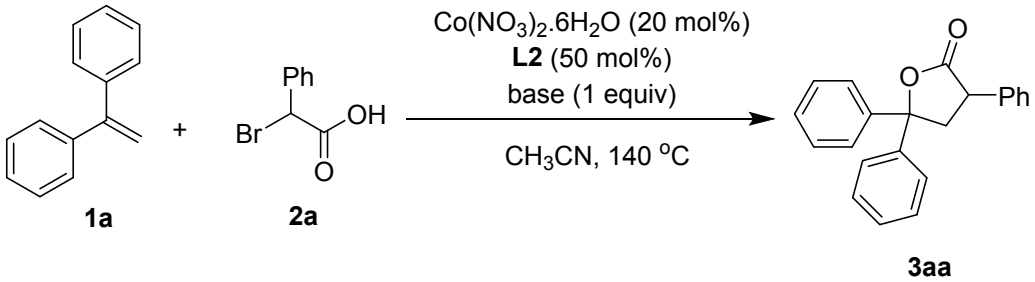
1a **2a** **3aa**

Entry	catalyst	ligand	yield of **3aa**, %
1	$Co(acac)_2$	-	69

2	$Co(acac)_3$	-	trace
3	$Co(OAc)_2$	-	57
4	$CoCl_2$	-	trace
5	$Co(NO_3)_2.6H_2O$	-	trace
6	$Co(NO_3)_2.6H_2O$	 **L1**	70
7	**$Co(NO_3)_2.6H_2O$**	 **L2**	**78**
8	$Co(NO_3)_2.6H_2O$	 **L3**	27
9	$Co(NO_3)_2.6H_2O$	 **L4**	52

Reaction conditions: **2a** (0.2 mmol), catalyst (0.02 mmol), ligand (0.05 mmol), K_2CO_3 (0.1 mmol), CH_3CN (1 mL), under argon, for 24 h. Yields are GC yields.

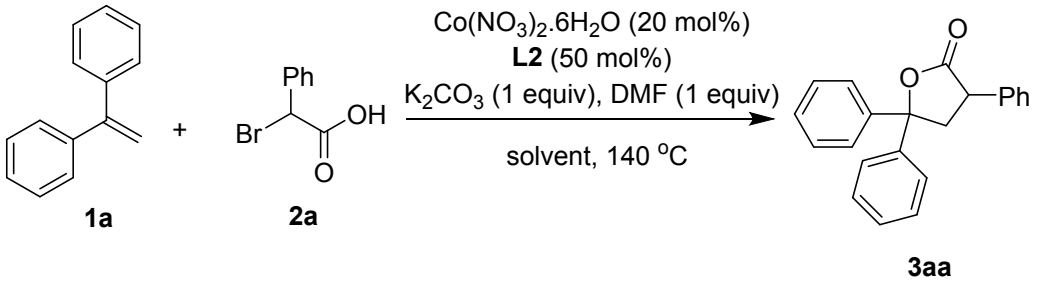
2.4. Effect of base



Entry	base	yield of **3aa**, %
1	K_2CO_3	78
2	Na_2CO_3	67
3	Cs_2CO_3	trace
4	KOAc	trace
5	K_3PO_4	32
6	KOPiv	trace
7	$KHCO_3$	62
8[a]	**K_2CO_3 + DMF**	**84**

Reaction conditions: **2a** (0.2 mmol), $Co(NO_3)_2.6H_2O$ (0.02 mmol), **L2** (0.05 mmol), base (0.1 mmol), CH_3CN (1 mL), under argon, for 24 h. Yields are GC yields. [a] K_2CO_3 (0.1 mmol) and DMF (0.1 mmol) were used as bases.

2.5. Effect of solvent



Entry	base	yield of **3aa**, %
1	CH_3CN	78
2	1,4-dioxane	75
3	DMF	81

4	DMSO	trace
5	toluene	trace
6	**$(CH_3)_3CCN$**	**91**
7	PhCN	trace
8	CF_3CH_2OH	trace
9	$(CF_3)_2CHOH$	trace

Reaction conditions: **2a** (0.2 mmol), $Co(NO_3)_2.6H_2O$ (0.02 mmol), **L2** (0.05 mmol), K_2CO_3 (0.1 mmol), DMF (0.1 mmol), solvent (1 mL), under argon, for 24 h. Yields are GC yields.

3. Annulation of styrenes and α-bromoacetic acids/ α-bromoacetates

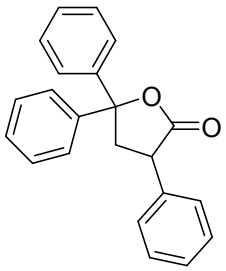
3.1. General procedures

Procedure 1 (Schemes 2 and 4): To a 25 mL Schlenk tube equipped with a magnetic stir bar was added a styrene derivative (0.5 mmol), an α-bromoacetic acid or ethyl ester, $Co(NO_3)_2.6H_2O$ (0.1 mmol, 18 mg), **L2** (0.25 mmol, 26 mg), K_2CO_3 (0.5 mmol, 69 mg), DMF (0.5 mmol, 37 mg), and $(CH_3)_3CN$ (5 mL). The reaction tube was flushed with argon for 5 min, capped, then placed into a preheated bath (at 140 °C). After being cooled to room temperature, the crude mixture was diluted with ethyl acetate (5 mL) and brine (10 mL). The aqueous layer was extracted with ethyl acetate (5 mL x 3). The combined organic layers were washed with saturated $NaHCO_3$ (aqueous solution, 10 mL), then dried over Na_2SO_4, and filtered. After concentrated under vacuum, the crude product was purified by column chromatography using hexanes/ethyl acetate eluent to afford the desired γ-lactone or the vinyl acetate.

Procedure 2 (for Scheme 3): similar to procedure 1, except: (i) no DMF was used, and (ii) reactions was run at 100 °C.

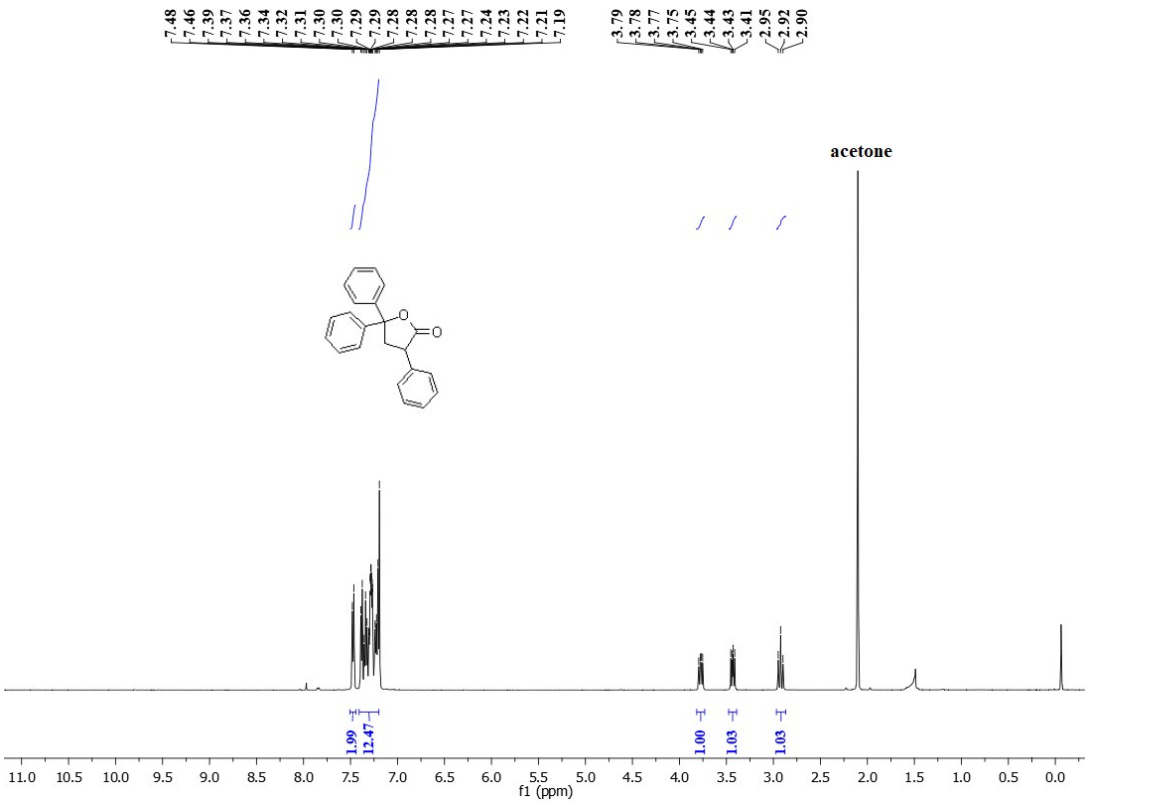
3.2. Characterization data of products

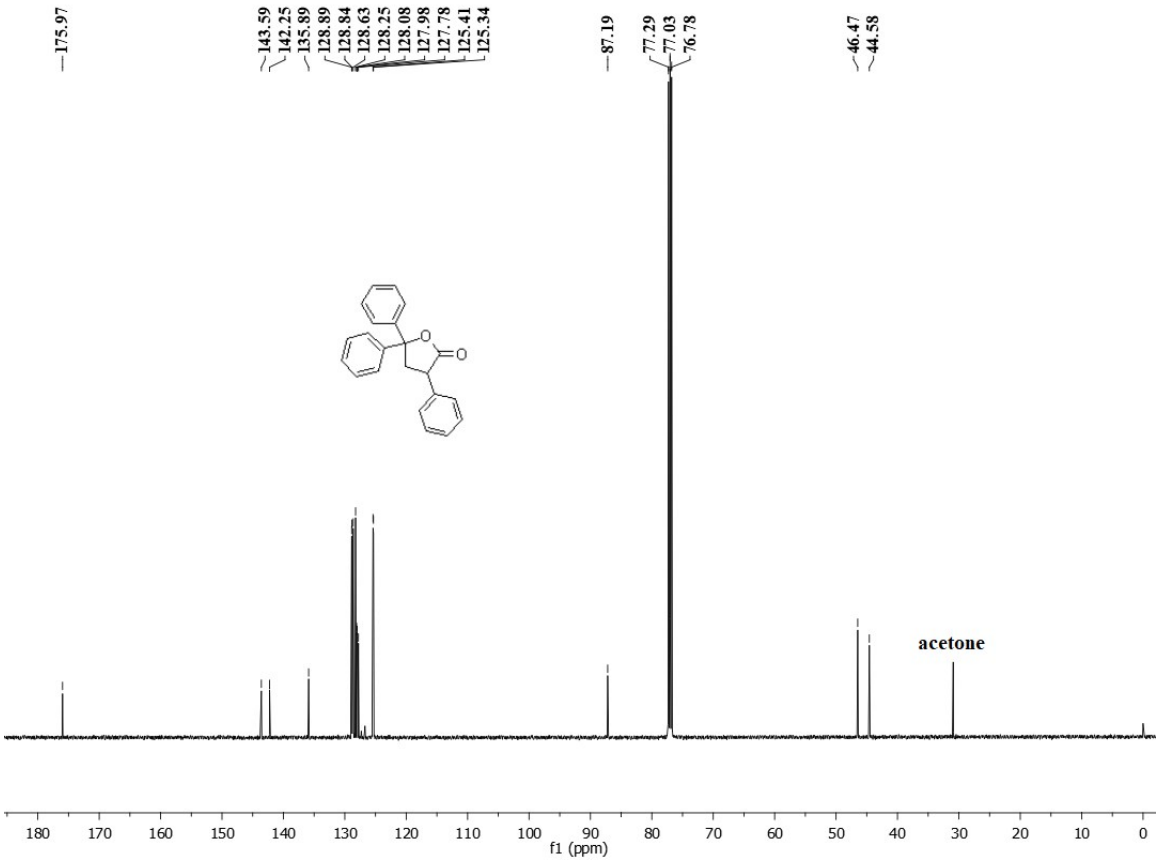
3,5,5-Triphenyldihydrofuran-2(3*H*)-one (3aa)[1]



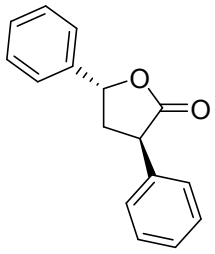
134 mg (85%); white solid. M.p. 108-110 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.47 (d, *J* = 7.8 Hz, 2H), 7.41 – 7.20 (m, 13H, overlapping with $CHCl_3$ signal), 3.77 (dd, *J* = 12.8, 8.0 Hz, 1H), 3.43 (dd, *J* = 12.7, 8.0 Hz, 1H), 2.92 (t, *J* = 12.8 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$, ppm) δ 175.9, 143.6, 142.2, 135.9, 128.9, 128.8, 128.6, 128.2, 128.1, 128.0, 127.8, 125.4, 125.3, 87.2, 46.5, 44.6.

[1] Wei, X.-J.; Yang, D.-T.; Wang, L.; Song, T.; Wu, L.-Z.; Liu, Q. *Org. Lett.*, **2013**, *15*, 6054.

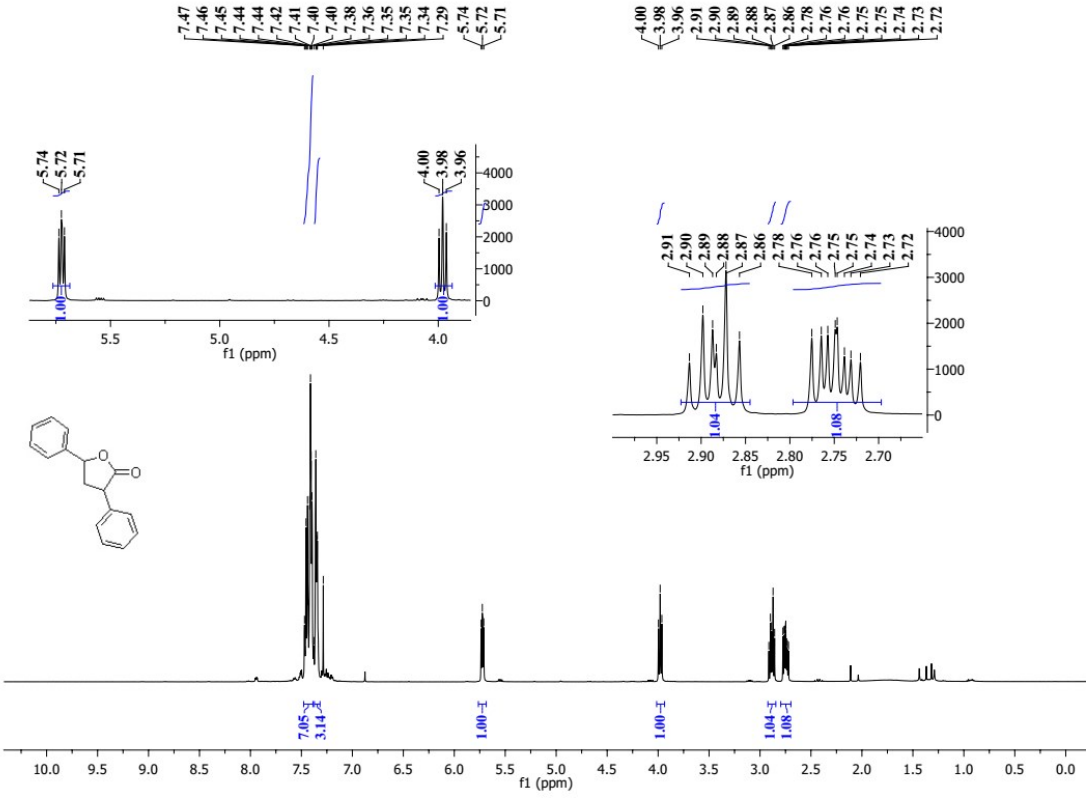

7.48
7.46
7.39
7.37
7.36
7.34
7.32
7.31
7.30
7.30
7.29
7.29
7.28
7.28
7.28
7.27
7.27
7.24
7.23
7.22
7.21
7.19
3.79
3.78
3.77
3.75
3.45
3.44
3.43
3.41
2.95
2.92
2.90
acetone
1.99
12.47
1.00
1.03
1.03
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


175.97
143.59
142.25
135.89
128.89
128.84
128.63
128.25
128.08
127.98
127.78
125.41
125.34
87.19
77.29
77.03
76.78
46.47
44.58
acetone
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

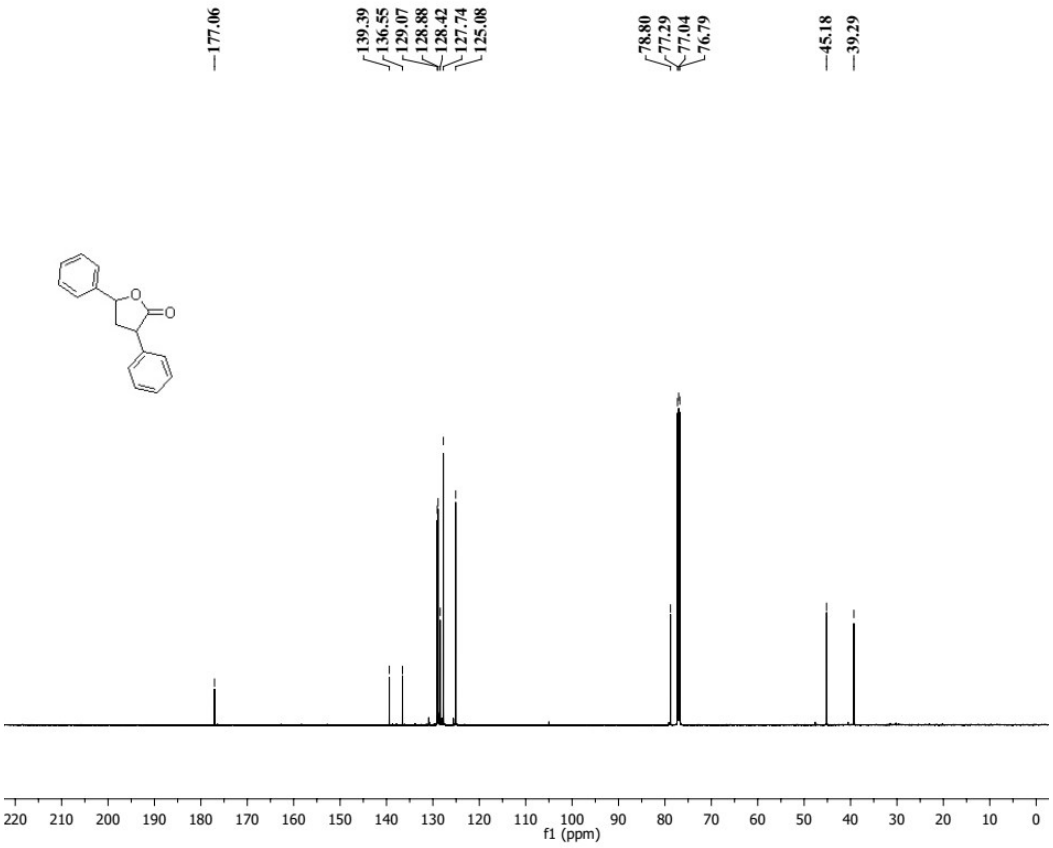
3,5-Diphenyldihydrofuran-2(3*H*)-one (3ab)[2]



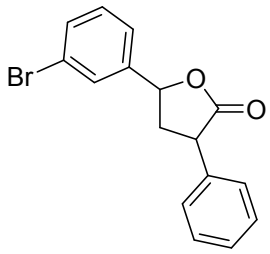
96 mg (81%); colorless oil which slowly solidified under air. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.48 – 7.38 (m, 7H), 7.36 – 7.29 (m, 3H), 5.75 – 5.69 (m, 1H), 3.98 (t, J = 8.4 Hz, 1H), 2.88 (dt, J = 13.1, 7.6 Hz, 1H), 2.75 (ddd, J = 13.1, 9.0, 5.4 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$, ppm) δ 177.1, 139.4, 136.5, 129.1, 128.9, 128.4, 127.7, 125.1, 78.8, 45.2, 39.3. One carbon signal could not be located.



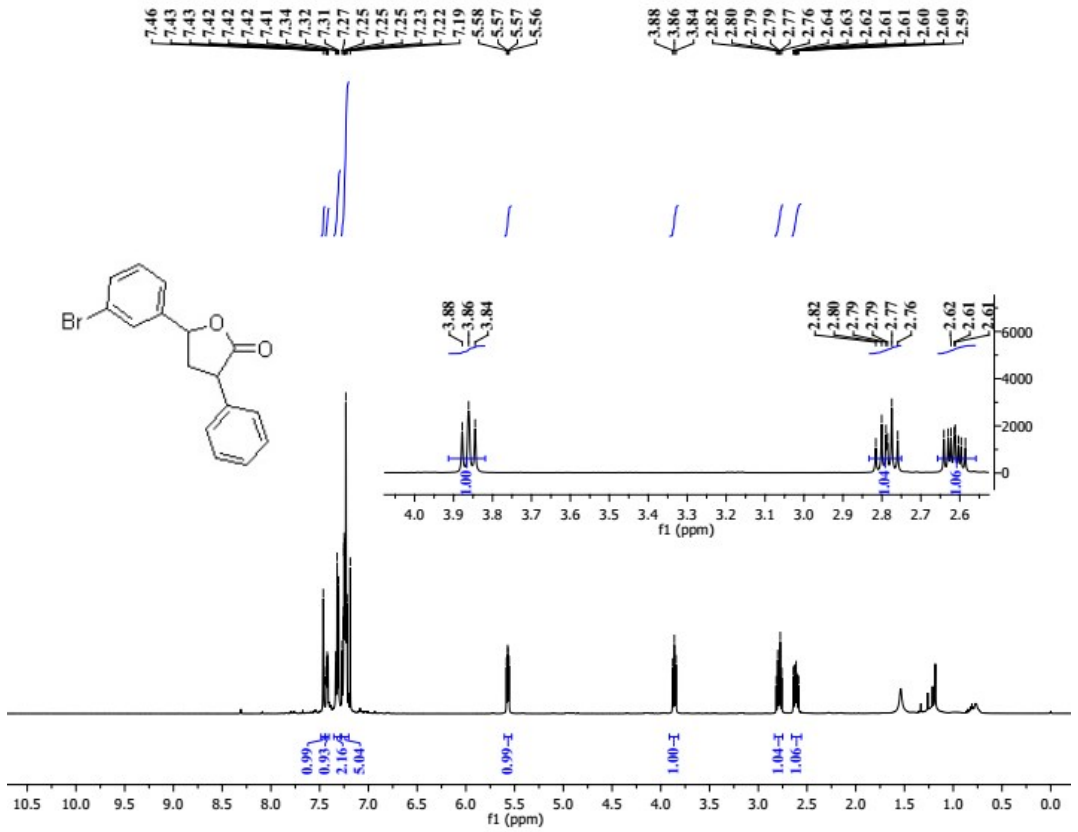
[2] Domingo, L. R.; Gil, S.; Parra, M.; Segura, J. *Molecules* **2018**, *13*, 1303.

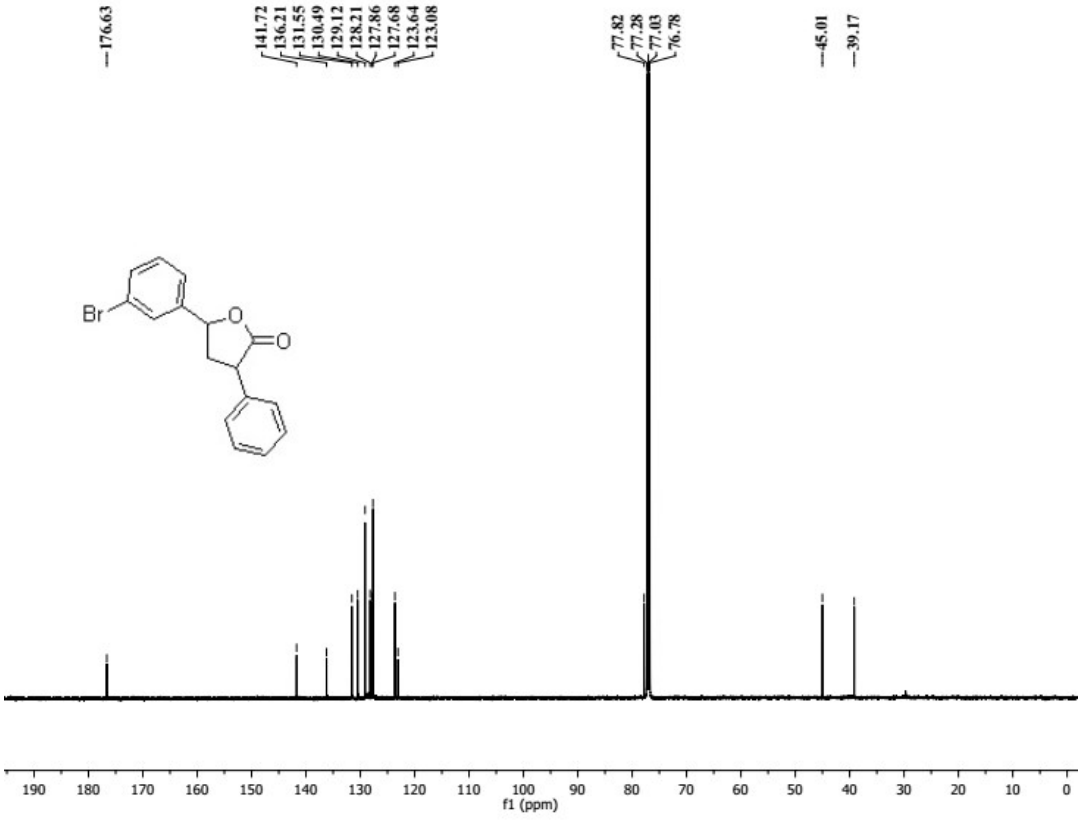


5-(3-Bromophenyl)-3-phenyldihydrofuran-2(3*H*)-one (3ca)

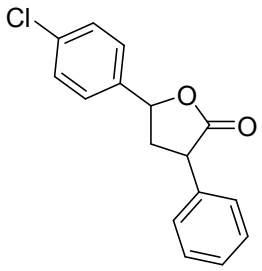


122 mg (77%); colorless oil which slowly solidified under air. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.46 (s, 1H), 7.44 – 7.41 (m, 1H), 7.32 (t, *J* = 7.3 Hz, 2H), 7.24 (dt, *J* = 15.1, 6.8 Hz, 5H), 5.57 (dd, *J* = 7.2, 5.7 Hz, 1H), 3.90 – 3.82 (m, 1H), 2.79 (dt, *J* = 13.1, 7.5 Hz, 1H), 2.61 (ddd, *J* = 13.1, 9.0, 5.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$, ppm) δ 176.6, 141.7, 136.2, 131.5, 130.5, 129.1, 128.2, 127.9, 127.7, 123.6, 123.1, 77.8, 45.0, 39.2. HRMS (ESI) calcd. for $C_{16}H_{14}{}^{79}BrO_2$ $[M+H]^+$: 317.0172; found: 317.0167.

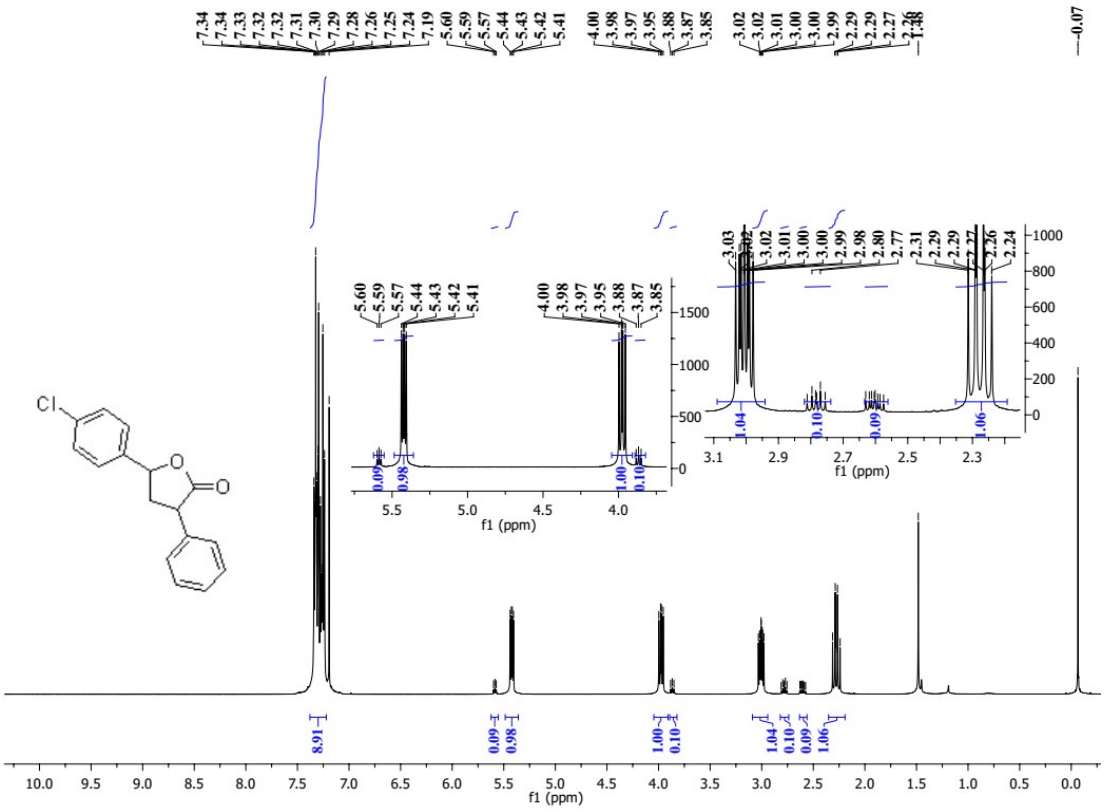

Br
O
O
f1 (ppm)
f1 (ppm)

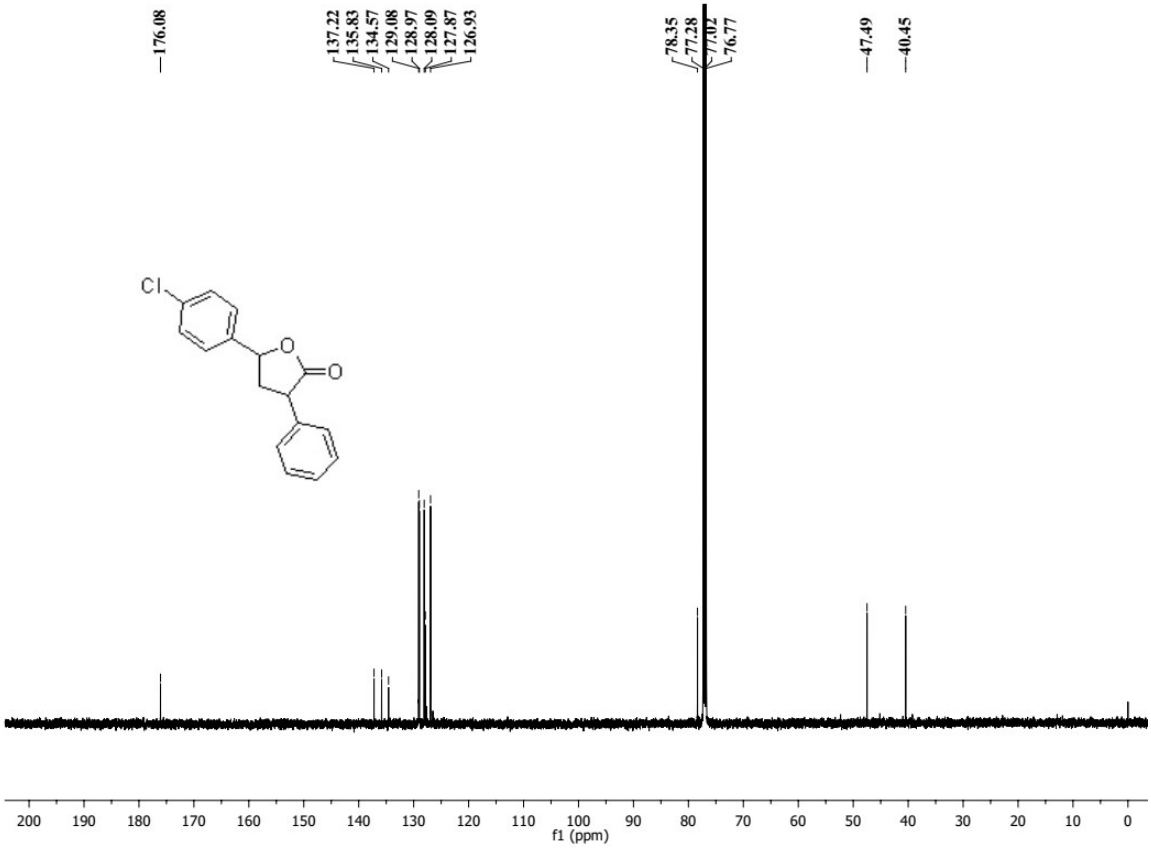

Br
O
O
f1 (ppm)

5-(4-Chlorophenyl)-3-phenyldihydrofuran-2(3*H*)-one (3da)

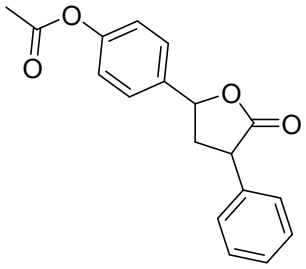


124 mg (91%); colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.38 – 7.22 (both isomers, m, 9H), 5.60 – 5.56 (minor, m, 1H), 5.42 (major, dd, J = 10.7, 5.6 Hz, 1H), 3.98 (major, dd, J = 12.8, 8.4 Hz, 1H), 3.89 – 3.84 (minor, m, 1H), 3.01 (major, ddd, J = 13.0, 8.4, 5.7 Hz, 1H), 2.78 (minor, dt, J = 13.2, 7.4 Hz, 1H), 2.60 (minor, ddd, J = 13.1, 9.0, 5.6 Hz, 1H), 2.28 (major, td, J = 12.8, 10.8 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$, ppm, only major isomer was shown) δ 176.1, 137.2, 135.8, 134.6, 129.1, 129.0, 128.1, 127.9, 126.9, 78.3, 47.5, 40.4. HRMS (ESI) calcd. for $C_{16}H_{14}{}^{35}ClO_2$ $[M+H]^+$: 273.0677; found: 273.0671.

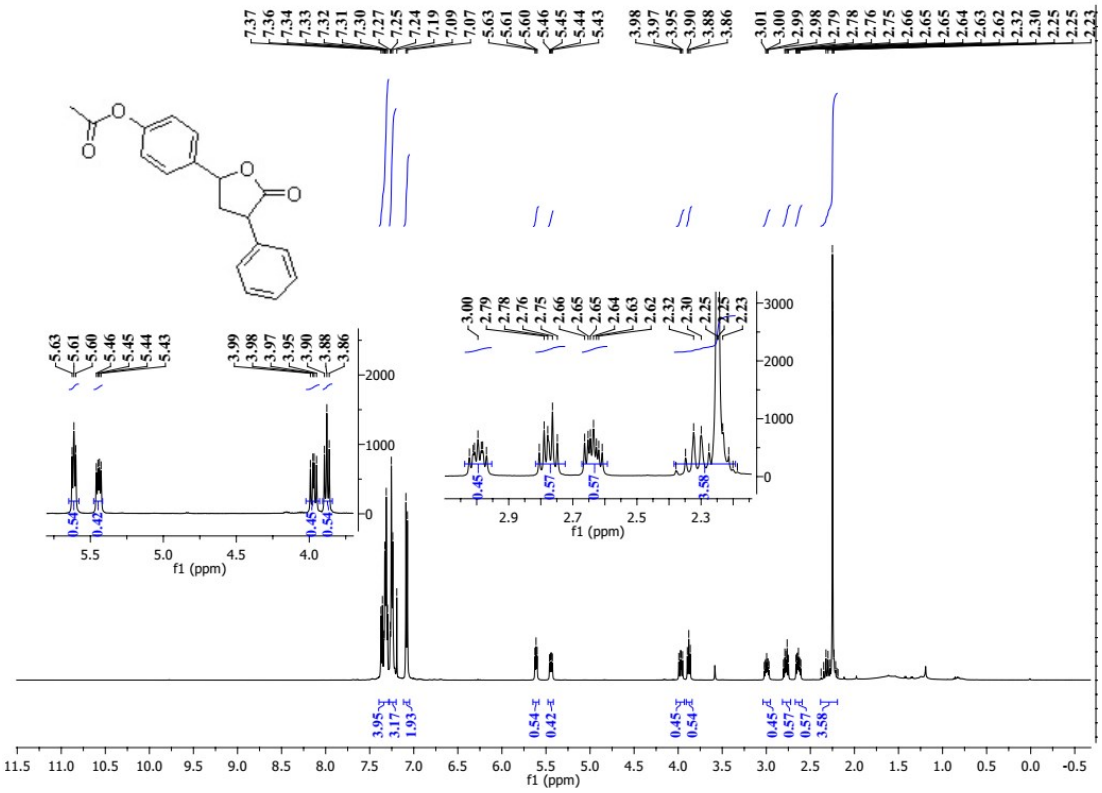

f1 (ppm)

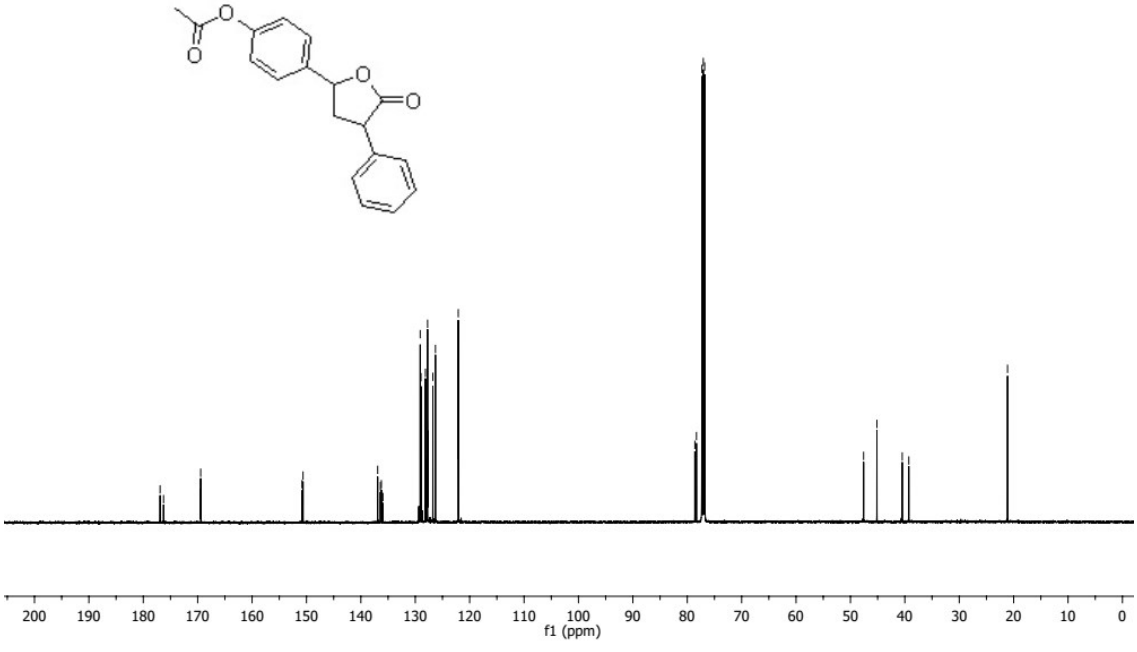

f1 (ppm)

4-(5-Oxo-4-phenyltetrahydrofuran-2-yl)phenyl acetate (3ea)

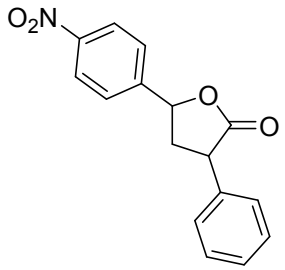


116 mg (78%); colorless oil which slowly solidified under air. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.40 – 7.29 (both isomers, m, 4H), 7.27 – 7.24 (both isomers, m, 3H), 7.08 (both isomers, d, J = 8.5 Hz, 2H), 5.65 – 5.57 (major, m, 1H), 5.44 (minor, dd, J = 10.7, 5.6 Hz, 1H), 3.97 (minor, dd, J = 12.8, 8.4 Hz, 1H), 3.88 (major, t, J = 8.3 Hz, 1H), 3.04 – 2.95 (minor, m, 1H), 2.83 – 2.72 (major, m, 1H), 2.68 – 2.59 (major, m, 1H), 2.38 – 2.21 (minor, m, 1H), 2.25 (both isomers, s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm, possible peaks for both isomers are listed) δ 176.9, 176.3, 169.45, 169.40, 150.8, 150.6, 136.9, 136.4, 136.2, 135.9, 129.4, 129.1, 128.9, 128.6, 128.1, 127.8, 127.8, 127.7, 126.8, 126.3, 122.10, 122.07, 78.6, 78.3, 47.6, 45.1, 40.5, 39.3, 21.1. HRMS (ESI) calcd. for $C_{18}H_{17}O_4$ $[M+H]^+$: 297.1121; found: 297.1120.

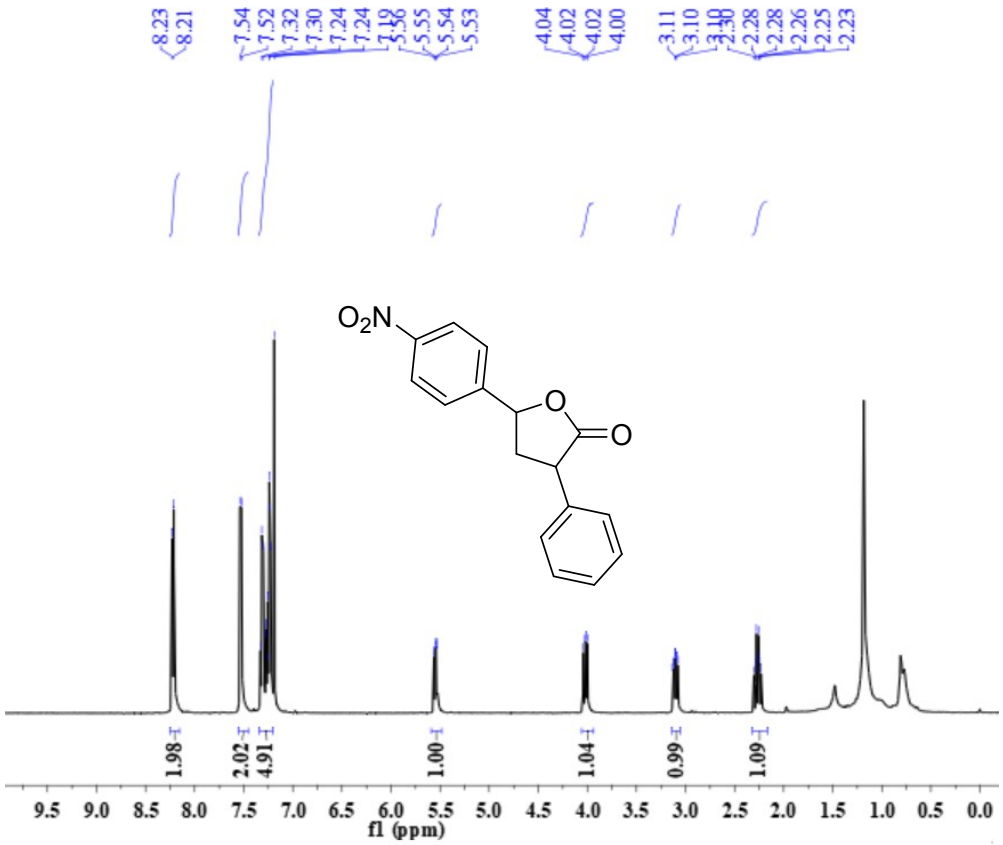

f1 (ppm)

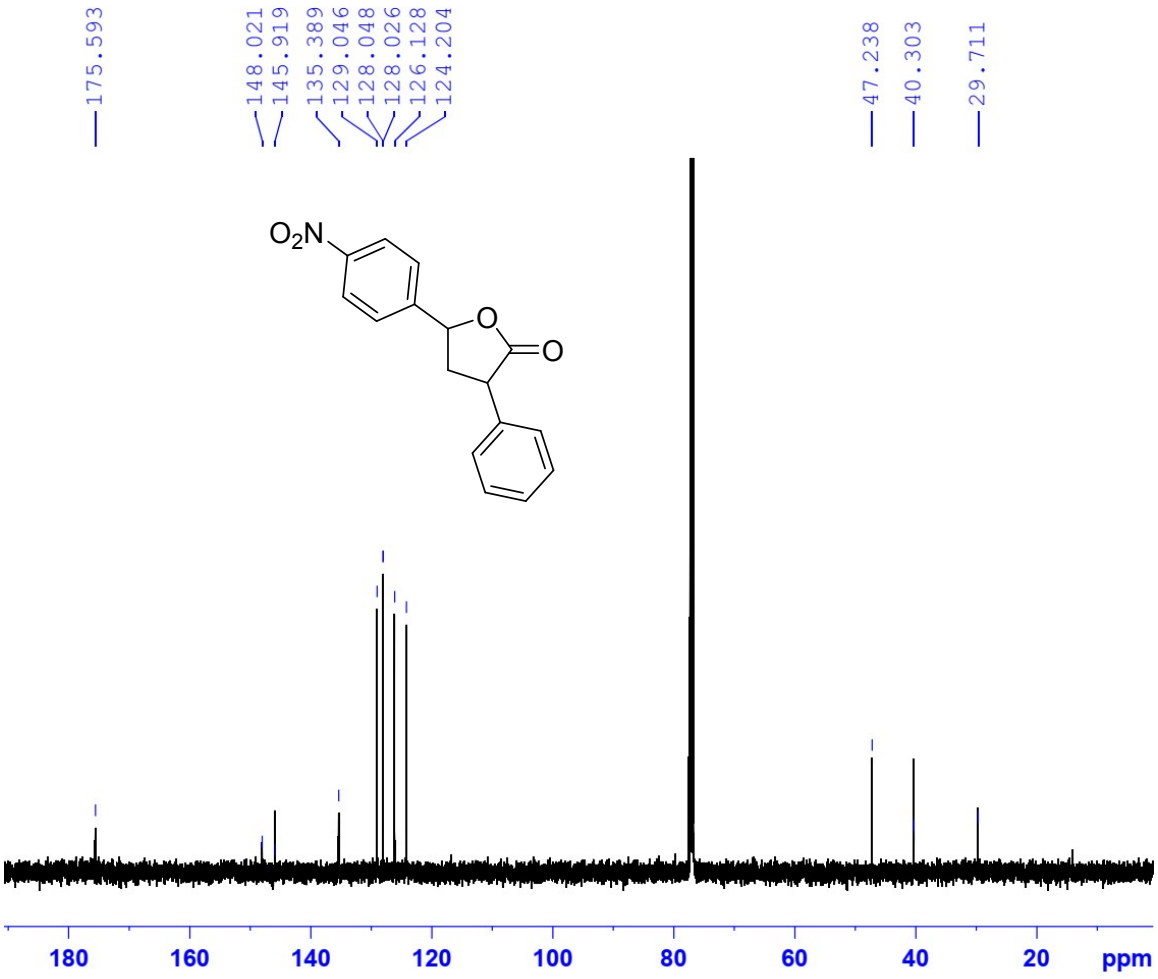

f1 (ppm)

5-(4-Nitrophenyl)-3-phenyldihydrofuran-2(3*H*)-one (3fa)

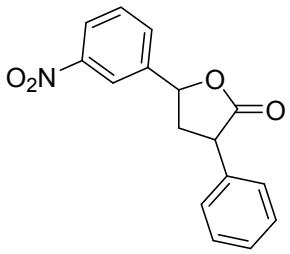


86 mg (61%); colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 8.22 (d, *J* = 8.7 Hz, 2H), 7.53 (d, *J* = 8.7 Hz, 2H), 7.34 – 7.20 (m, 5H), 5.55 (dd, *J* = 10.7, 5.8 Hz, 1H), 4.02 (dd, *J* = 12.7, 8.5 Hz, 1H), 3.11 (ddd, *J* = 12.9, 8.5, 5.8 Hz, 1H), 2.27 (td, *J* = 12.8, 10.8 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm) δ 175.6, 148.0, 145.9, 135.4, 129.0, 128.05, 128.02, 126.1, 124.2, 47.2, 40.3, 29.7. HRMS (ESI) calcd. for $C_{16}H_{14}NO_4$ $[M+H]^+$: 284.0917; found: 284.0911.



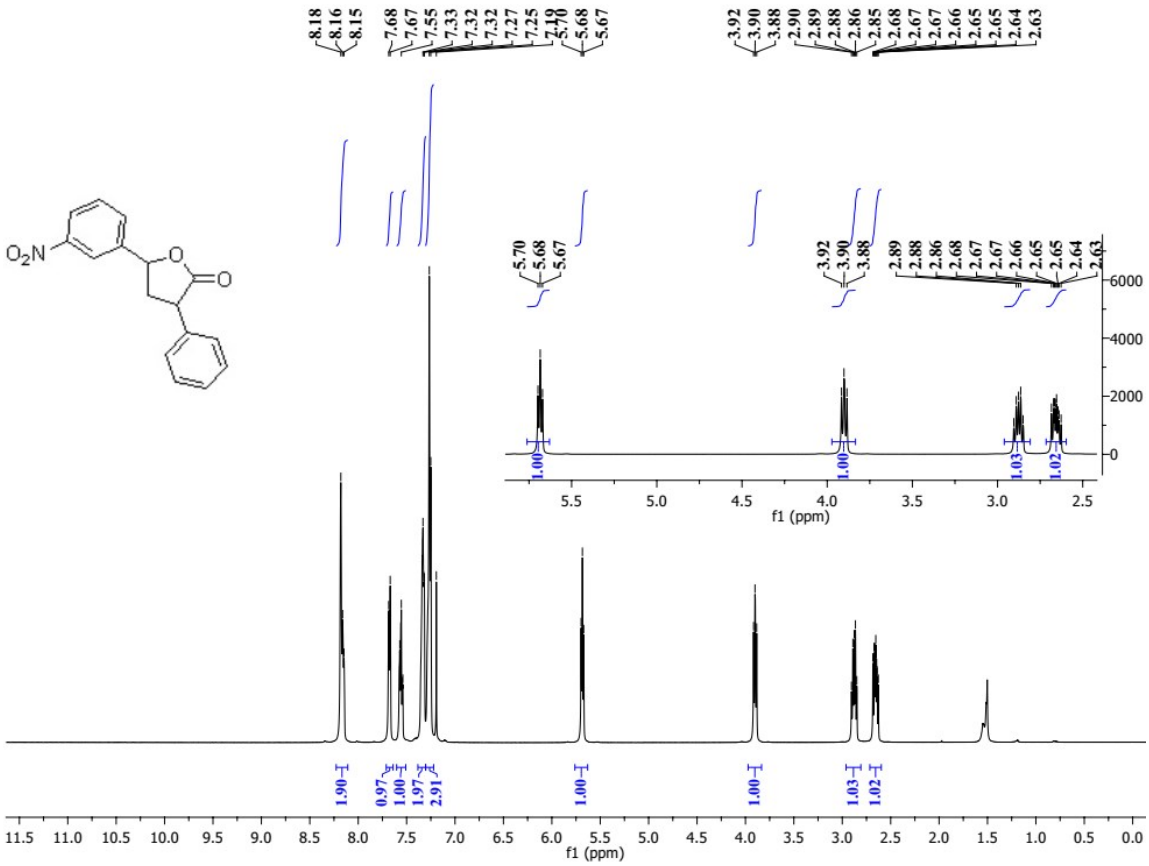


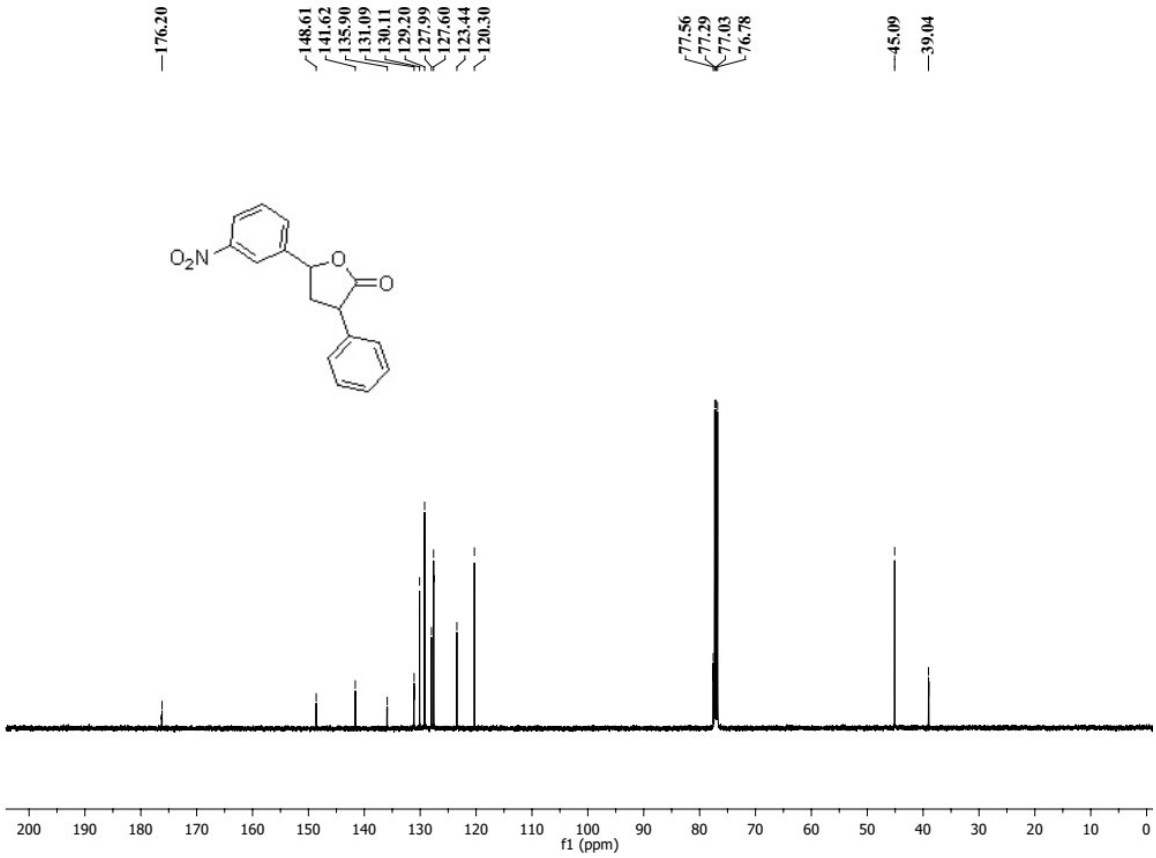
5-(3-Nitrophenyl)-3-phenyldihydrofuran-2(3*H*)-one (3ga)



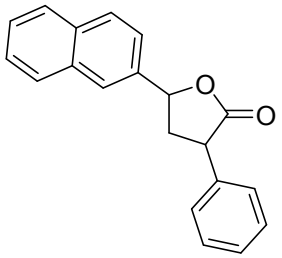
91 mg (64%); light yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 8.22 – 8.13 (m, 2H), 7.68 (d, J = 7.6 Hz, 1H), 7.55 (dd, J = 10.5, 5.3 Hz, 1H), 7.38 – 7.30 (m, 2H), 7.26 (d, J = 7.5 Hz, 3H), 5.68 (t, J = 6.7 Hz, 1H), 3.94 – 3.85 (m, 1H), 2.93 – 2.78 (m, 1H), 2.66 (ddd, J = 13.3, 9.0, 6.1 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm) δ 176.2, 148.6, 141.6,

135.9, 131.1, 130.1, 129.2, 128.0, 127.6, 123.4, 120.3, 77.6, 45.1, 39.0. HRMS (ESI) calcd. for $C_{16}H_{14}NO_4$ $[M+H]^+$: 284.0917; found: 284.0921.



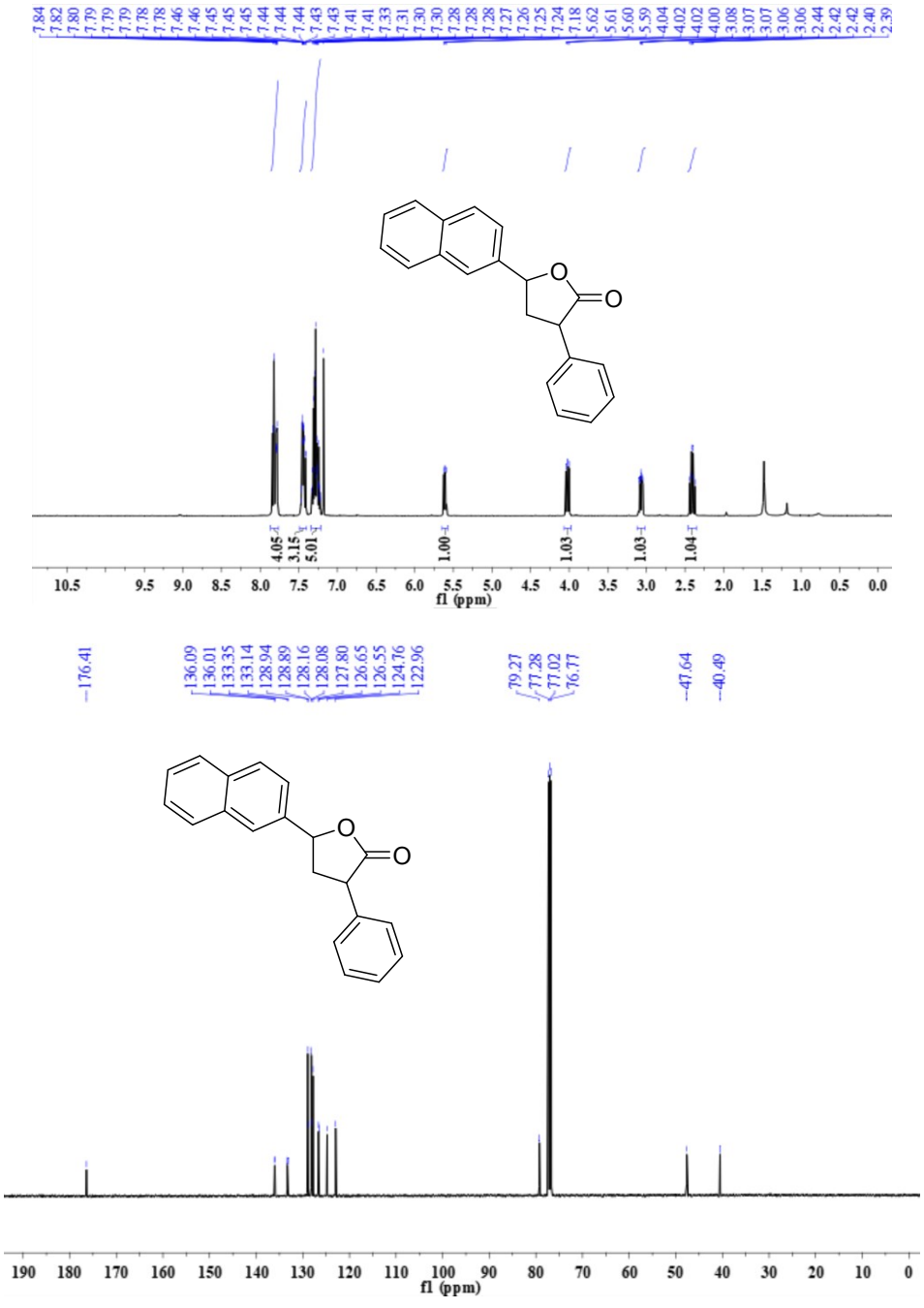


5-(Naphthalen-2-yl)-3-phenyldihydrofuran-2(3*H*)-one (3ha)

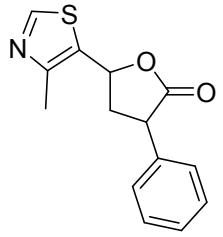


127 mg (88%); colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.84 – 7.78 (m, 4H), 7.49 – 7.40 (m, 3H), 7.34 – 7.21 (m, 5H), 5.61 (dd, *J* = 10.7, 5.7 Hz, 1H), 4.02 (dd, *J* = 12.8, 8.5 Hz, 1H), 3.07 (ddd, *J* = 12.9, 8.5, 5.7 Hz, 1H), 2.41 (td, *J* = 12.8, 10.8 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm) δ 176.4, 136.1, 136.0, 133.3, 133.1, 128.94, 128.89, 128.2, 128.1, 127.8, 126.6, 126.5, 124.8, 123.0, 79.27, 47.64, 40.49. One carbon signal

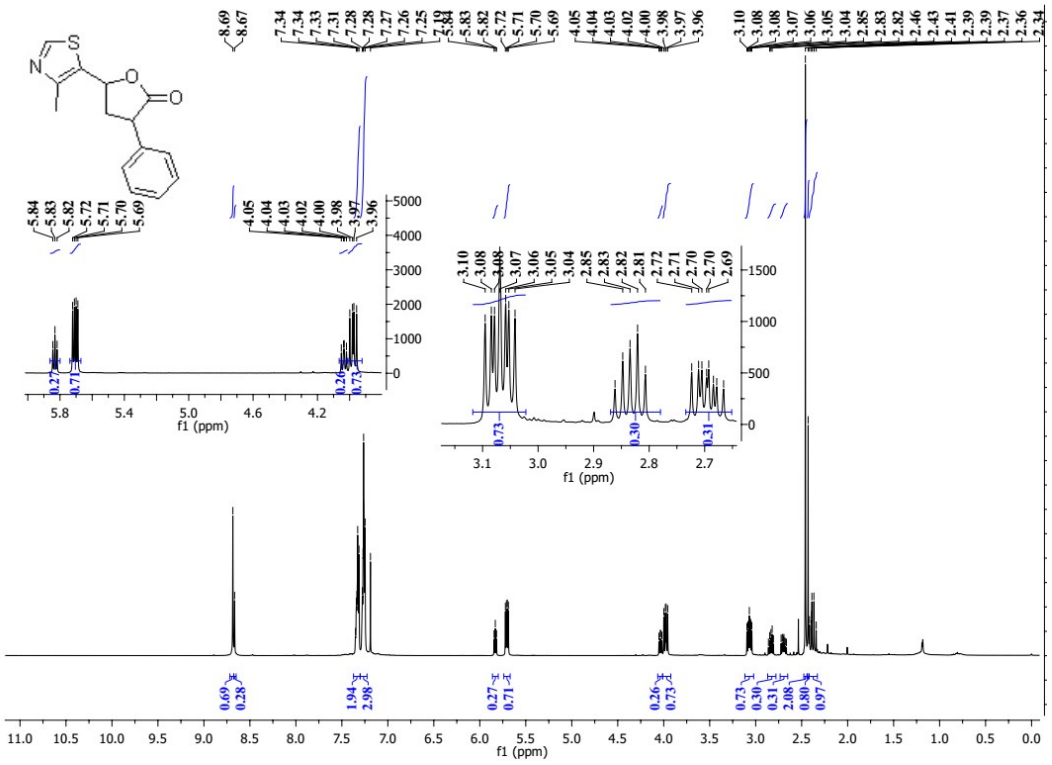
could not be located. HRMS (ESI) calcd. for $C_{20}H_{17}O_2$ $[M+H]^+$: 289.1223; found: 289.1231.

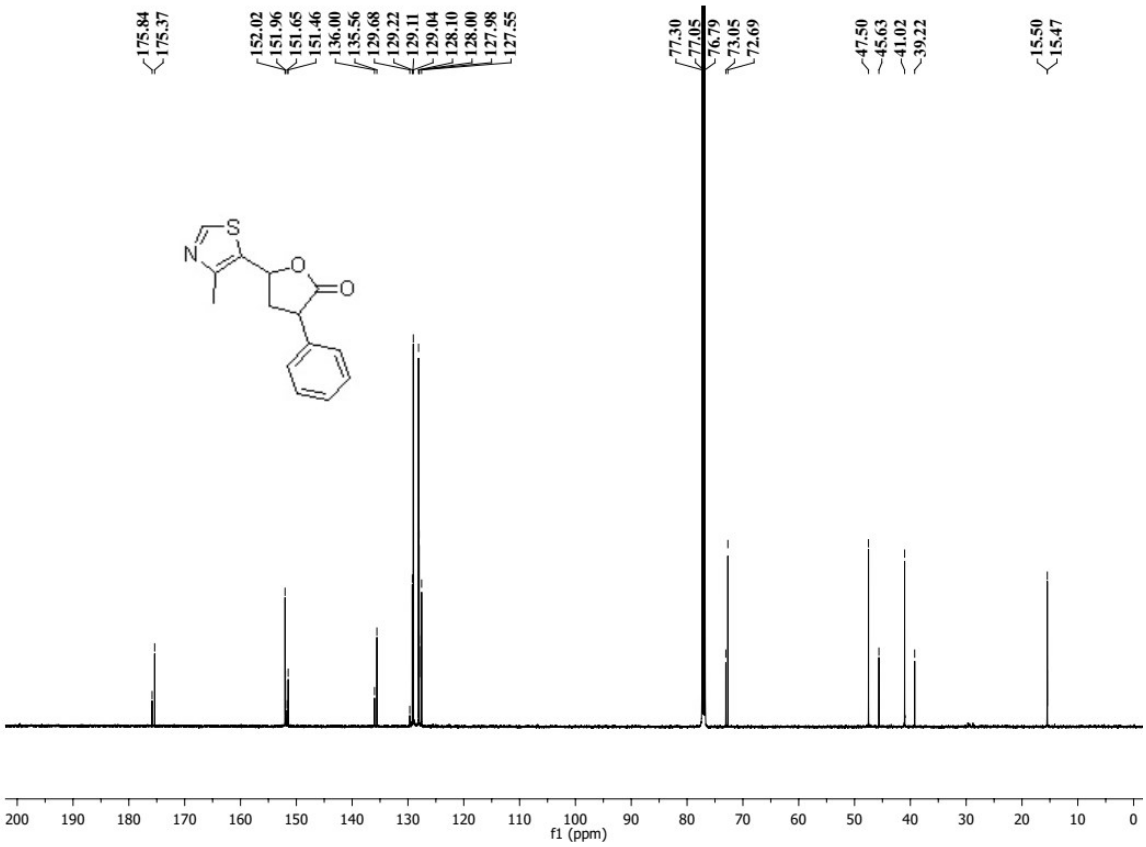


5-(4-Methylthiazol-5-yl)-3-phenyldihydrofuran-2(3*H*)-one (3ja)

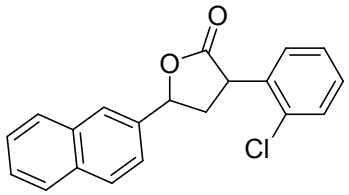


84 mg (65%); brown oil which slowly solidified under air. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 8.69 (major, s, 1H), 8.67 (minor, s, 1H), 7.37 – 7.30 (both isomers, m, 2H), 7.29 – 7.21 (both isomers, m, 3H), 5.83 (minor, t, J = 6.7 Hz, 1H), 5.71 (major, dd, J = 10.8, 5.5 Hz, 1H), 4.04 (minor, dd, J = 9.1, 6.9 Hz, 1H), 3.98 (major, dd, J = 12.7, 8.4 Hz, 1H), 3.07 (major, ddd, J = 13.7, 8.4, 5.6 Hz, 1H), 2.83 (minor, dt, J = 13.9, 7.0 Hz, 1H), 2.69 (minor, ddd, J = 13.4, 9.2, 6.2 Hz, 1H), 2.46 (major, s, 3H), 2.43 (minor, s, 3H), 2.41 – 2.34 (both isomers, m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$, ppm) δ 175.8 (minor), 175.4 (major), 152.02 (major), 151.96 (minor), 151.6 (minor), 151.5 (major), 136.0 (minor), 135.6 (major), 129.7 (minor), 129.2 (major), 129.1 (minor), 129.0 (major), 128.1 (major), 128.00 (major), 127.98 (minor), 127.5 (minor), 73.0 (minor), 72.7 (major), 47.5 (major), 45.6 (minor), 41.0 (major), 39.2 (minor), 15.50 (minor), 15.47 (major). HRMS (ESI) calcd. for $C_{14}H_{14}NO_2S$ $[M+H]^+$: 260.0740; found: 260.0732.

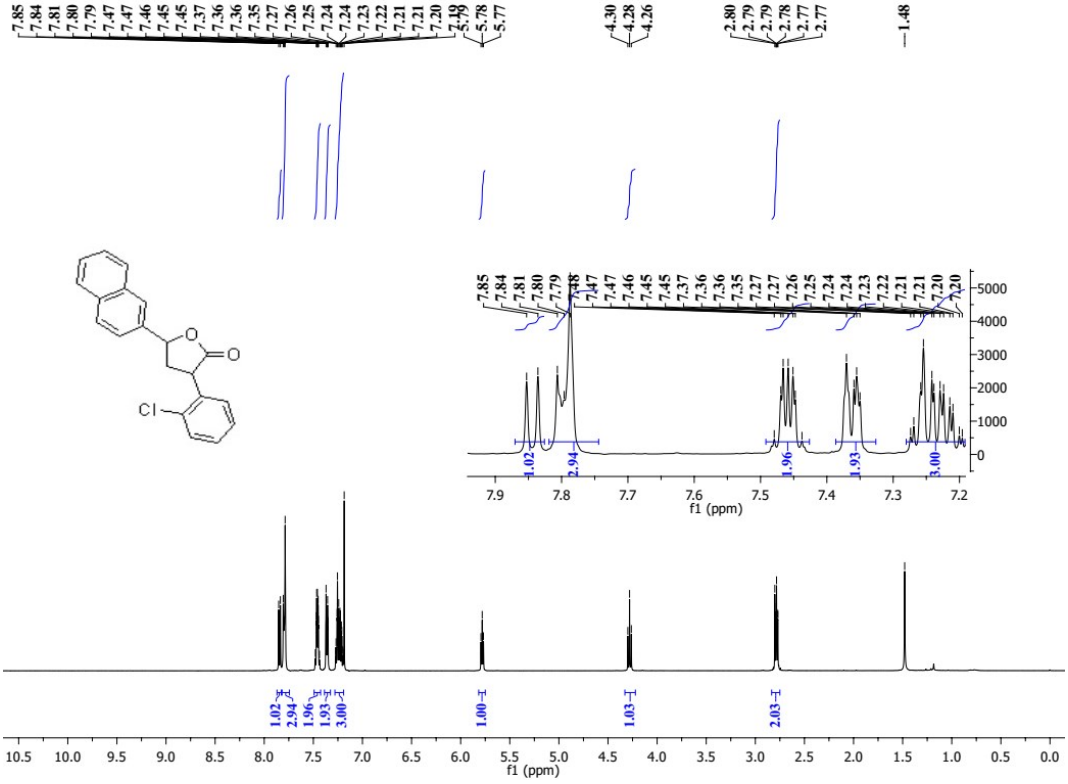

f1 (ppm)

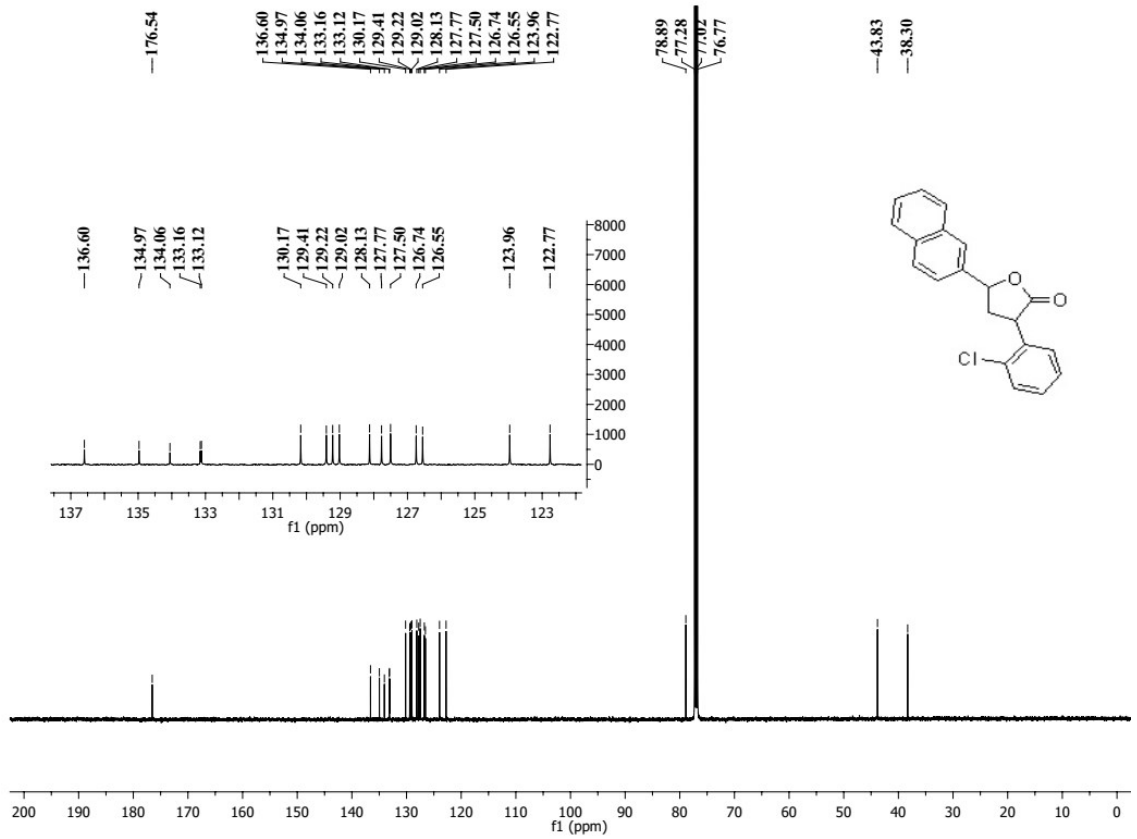

f1 (ppm)

3-(2-Chlorophenyl)-5-(naphthalen-2-yl)dihydrofuran-2(3*H*)-one (3hb)

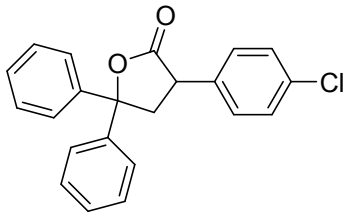


63 mg (39%); white amorphous solid. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.84 (d, *J* = 8.5 Hz, 1H), 7.82 – 7.77 (m, 3H), 7.49 – 7.43 (m, 2H), 7.36 (dd, *J* = 6.1, 4.1 Hz, 2H), 7.28 – 7.19 (m, 3H), 5.78 (t, *J* = 6.3 Hz, 1H), 4.28 (t, *J* = 8.8 Hz, 1H), 2.81 – 2.76 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$, ppm) δ 176.5, 136.6, 135.0, 134.1, 133.16, 133.12, 130.2, 129.4, 129.2, 129.0, 128.1, 127.8, 127.5, 126.7, 126.5, 124.0, 122.8, 78.9, 43.8, 38.3. HRMS (ESI) calcd. for $C_{20}H_{16}{}^{35}ClO_2$ $[M+H]^+$: 323.0833; found: 323.0827.

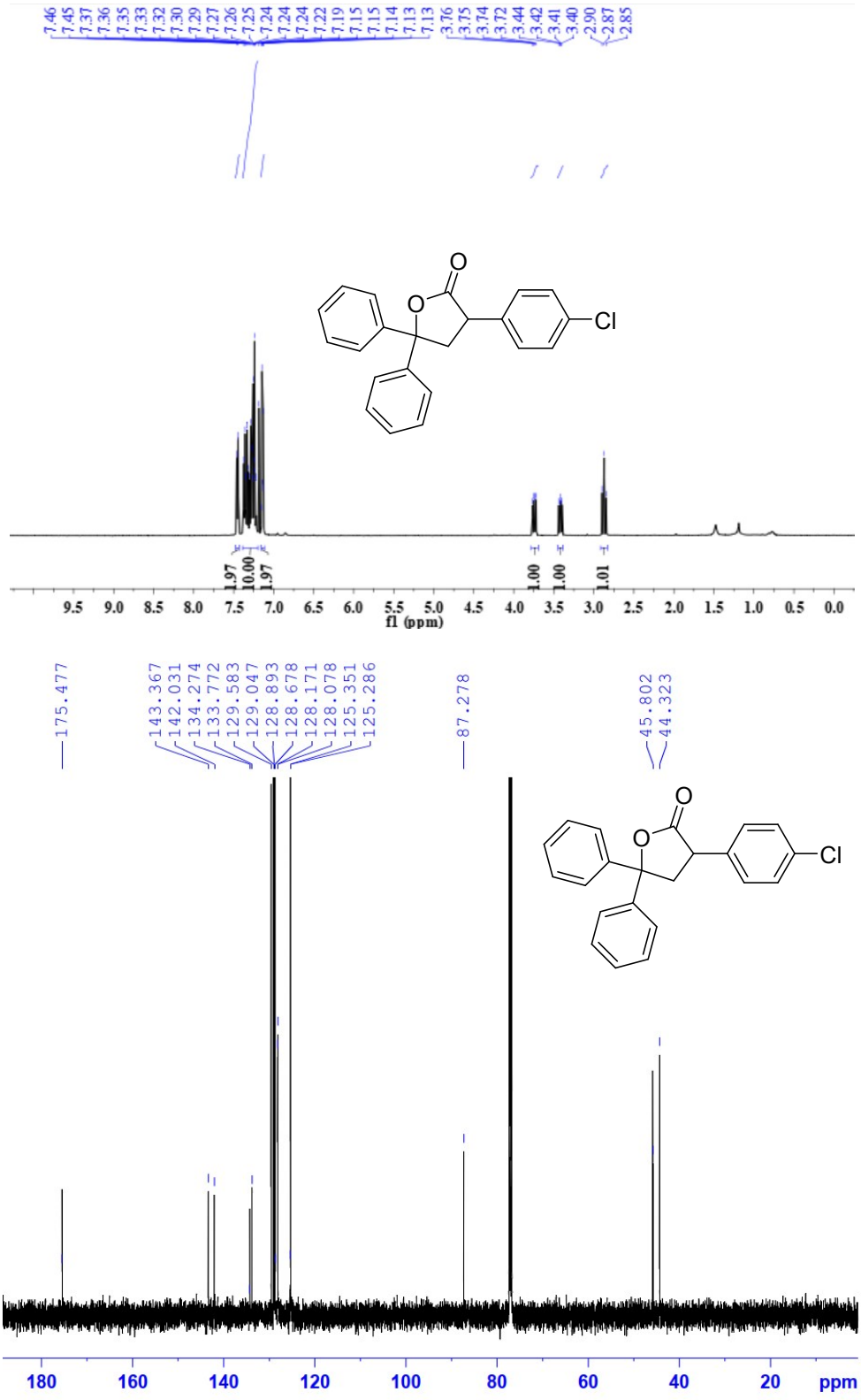




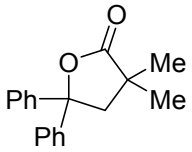
3-(4-Chlorophenyl)-5,5-diphenyldihydrofuran-2(3*H*)-one (3ac)



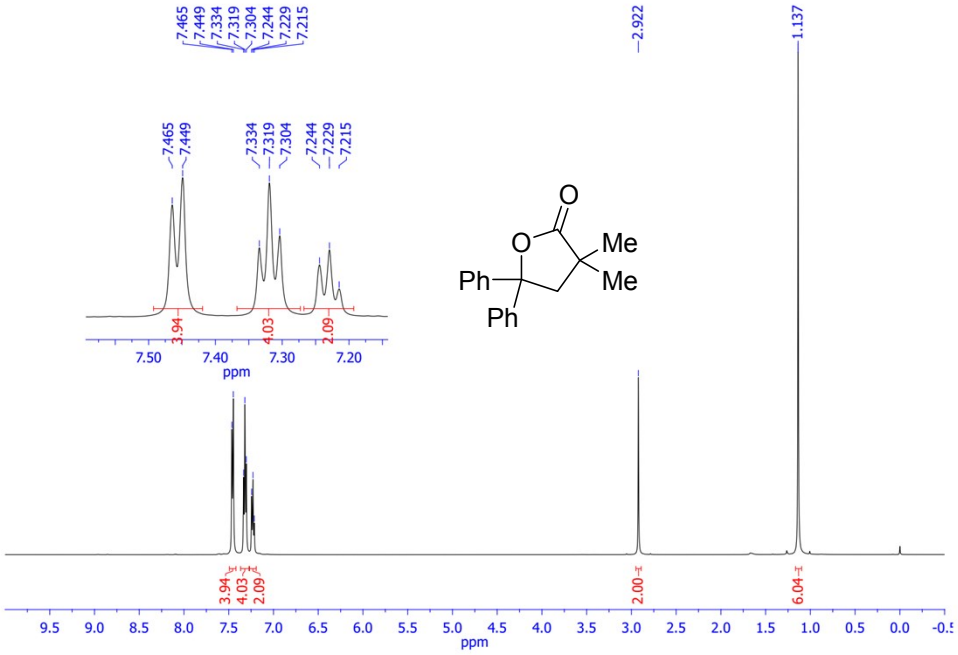
122 mg (70% yield); colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.45 (d, J = 7.8 Hz, 2H), 7.39 – 7.20 (m, 10H), 7.16 – 7.11 (m, 2H), 3.74 (dd, J = 12.9, 7.9 Hz, 1H), 3.42 (dd, J = 12.6, 7.9 Hz, 1H), 2.87 (t, J = 12.8 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm) δ 175.5, 143.4, 142.0, 134.3, 133.8, 129.6, 129.0, 128.9, 128.7, 128.2, 128.1, 125.4, 125.3, 87.3, 45.8, 44.3. HRMS (ESI) calcd. for $C_{22}H_{18}{}^{35}ClO_2$ $[M+H]^+$: 349.0990; found: 349.0997.



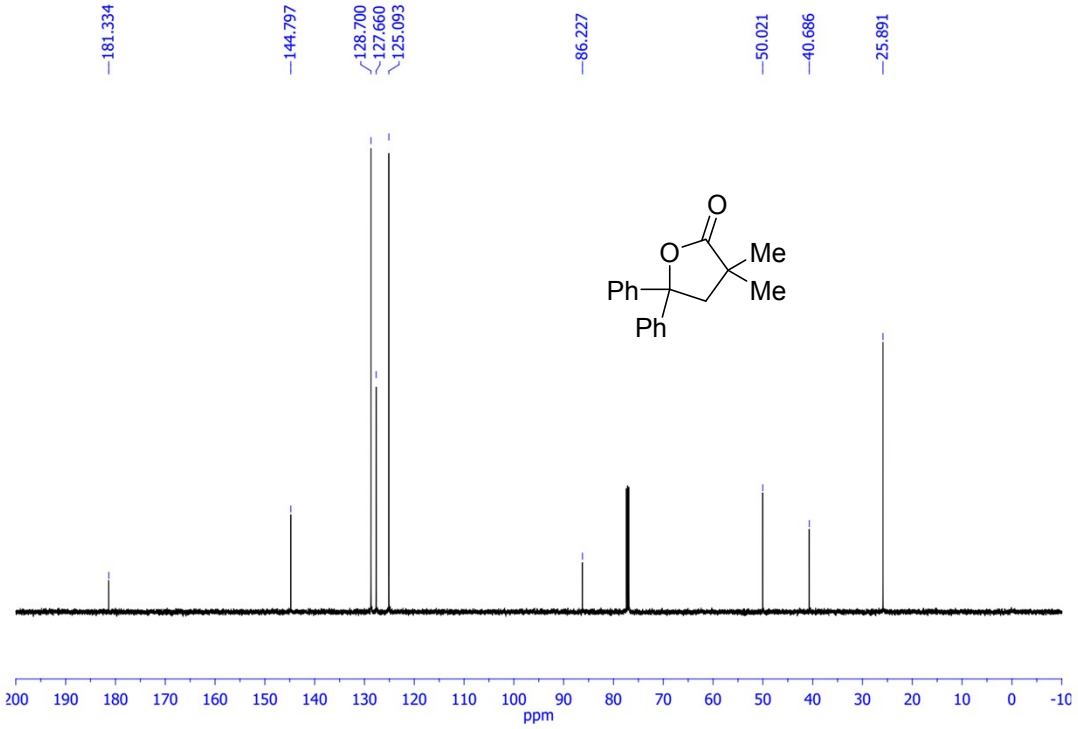
3,3-Dimethyl-5,5-diphenyldihydrofuran-2(3*H*)-one (5a)[3]



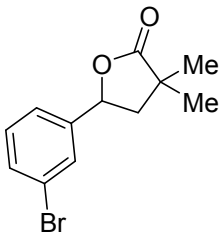
116 mg (87%); colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.46 (d, J = 8.0 Hz, 4H), 7.32 (t, J = 7.5 Hz, 4H), 7.23 (t, J = 7.5 Hz, 2H), 2.92 (s, 2H), 1.14 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm) δ 181.3, 144.8, 128.7, 127.7, 125.1, 86.2, 50.0, 40.7, 25.9.



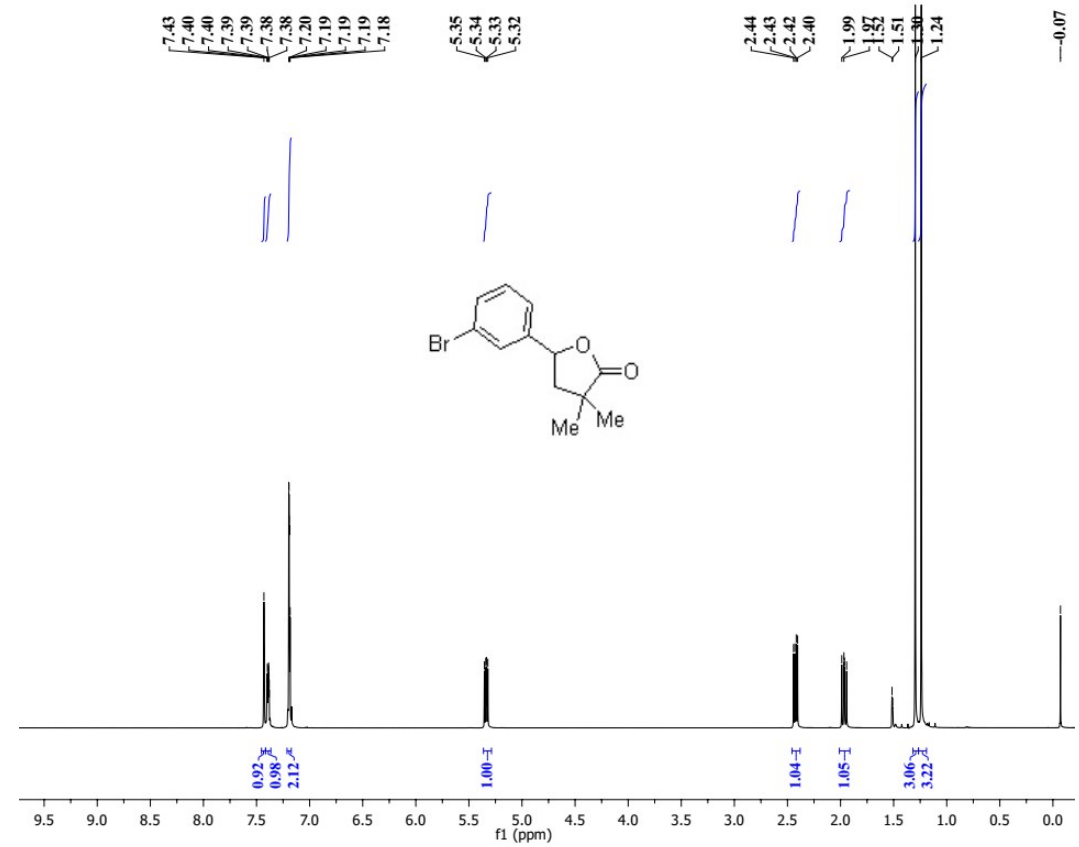
[3] Iwasaki, M.; Miki, N.; Ikemoto, Y.; Ura, Y.; Nishihara, Y. *Org. Lett.* **2018**, *20*, 3848.

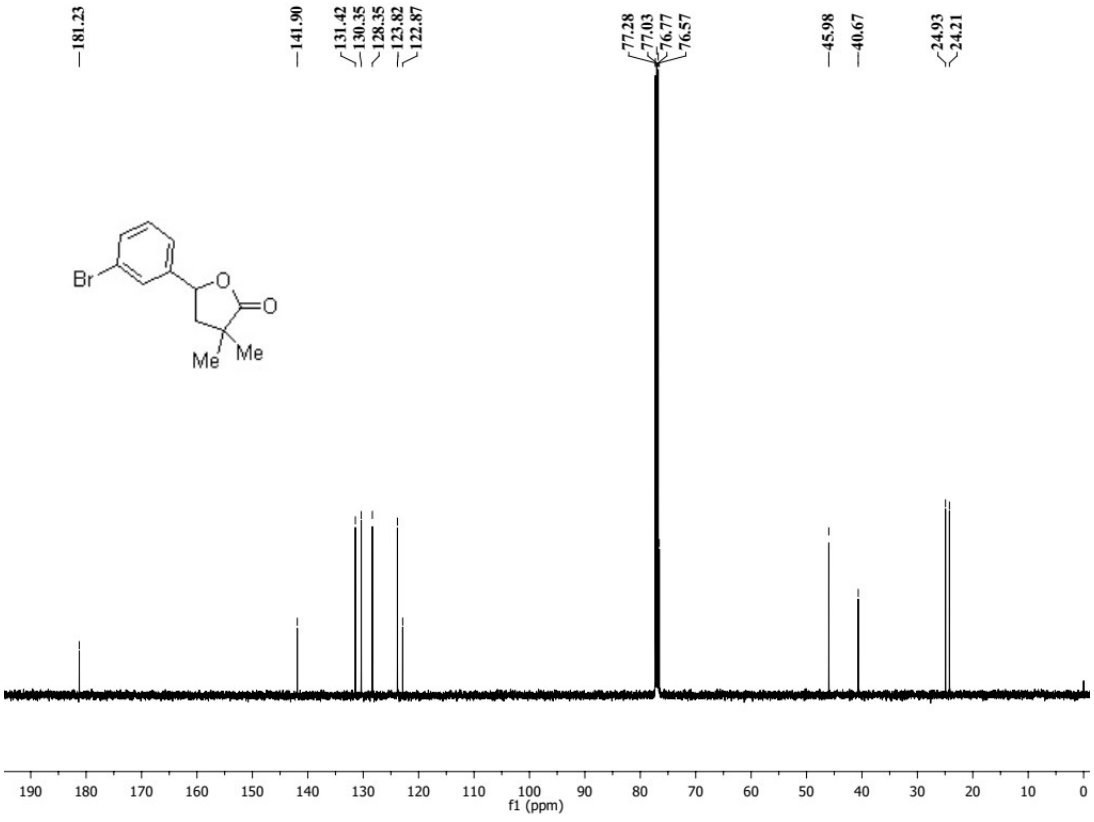


5-(3-Bromophenyl)-3,3-dimethyldihydrofuran-2(3*H*)-one (5c)

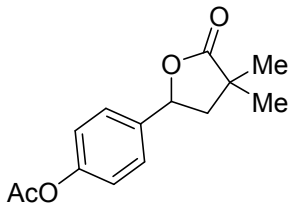


104 mg (78%); colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.43 (s, 1H), 7.41 – 7.38 (m, 1H), 7.21 – 7.18 (m, 2H), 5.34 (dd, J = 9.9, 6.4 Hz, 1H), 2.42 (dd, J = 12.9, 6.3 Hz, 1H), 1.97 (dd, J = 12.8, 9.9 Hz, 1H), 1.30 (s, 3H), 1.24 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 181.2, 141.9, 131.4, 130.3, 128.3, 123.8, 122.9, 76.6, 46.0, 40.7, 24.9, 24.2. HRMS (ESI) calcd. for $C_{12}H_{14}{}^{79}BrO_2$ $[M+H]^+$: 269.0172; found: 269.0178.

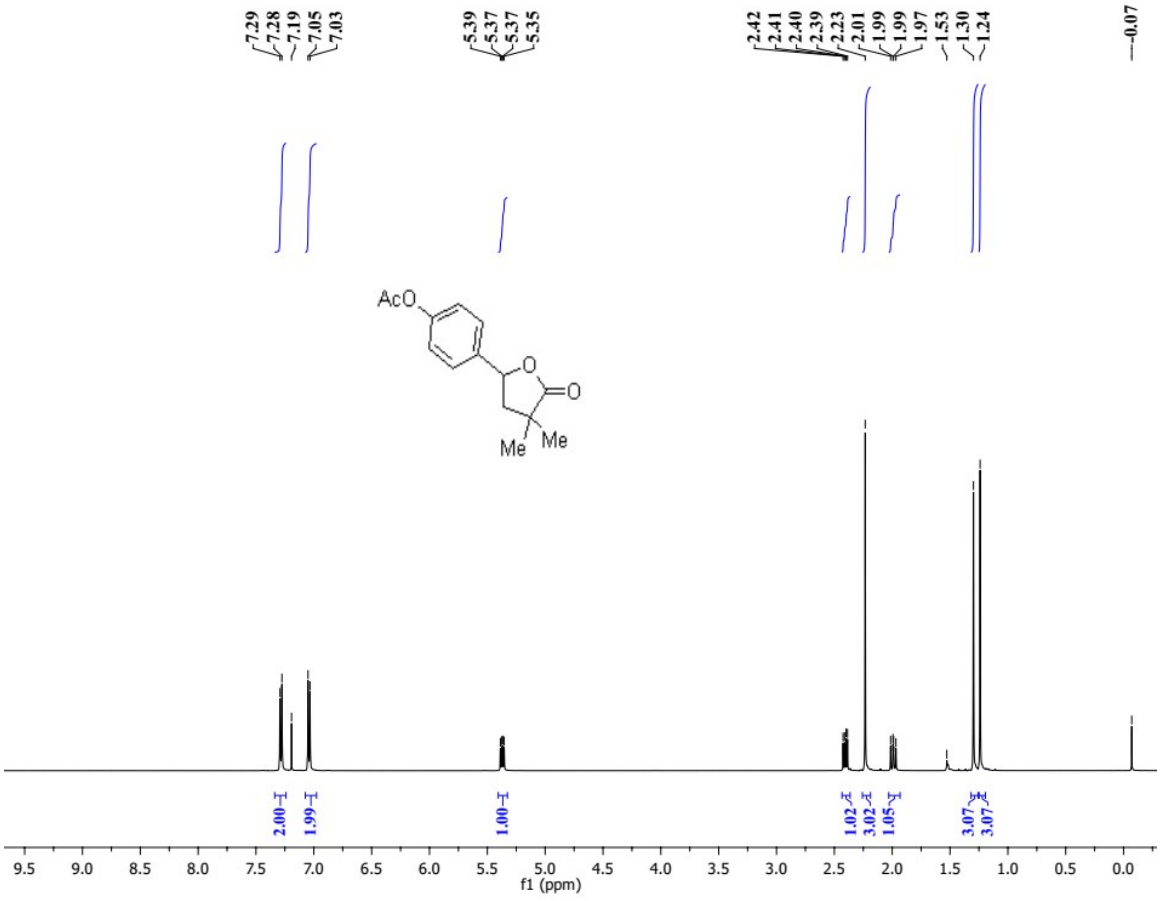

7.43
7.40
7.40
7.39
7.39
7.38
7.38
7.20
7.19
7.19
7.19
7.18
5.35
5.34
5.33
5.32
2.44
2.43
2.42
2.40
1.99
1.97
1.52
1.51
1.30
1.24
0.07
Br
O
O
Me
Me
0.92
0.98
2.12
1.00
1.04
1.05
3.06
3.22
f1 (ppm)

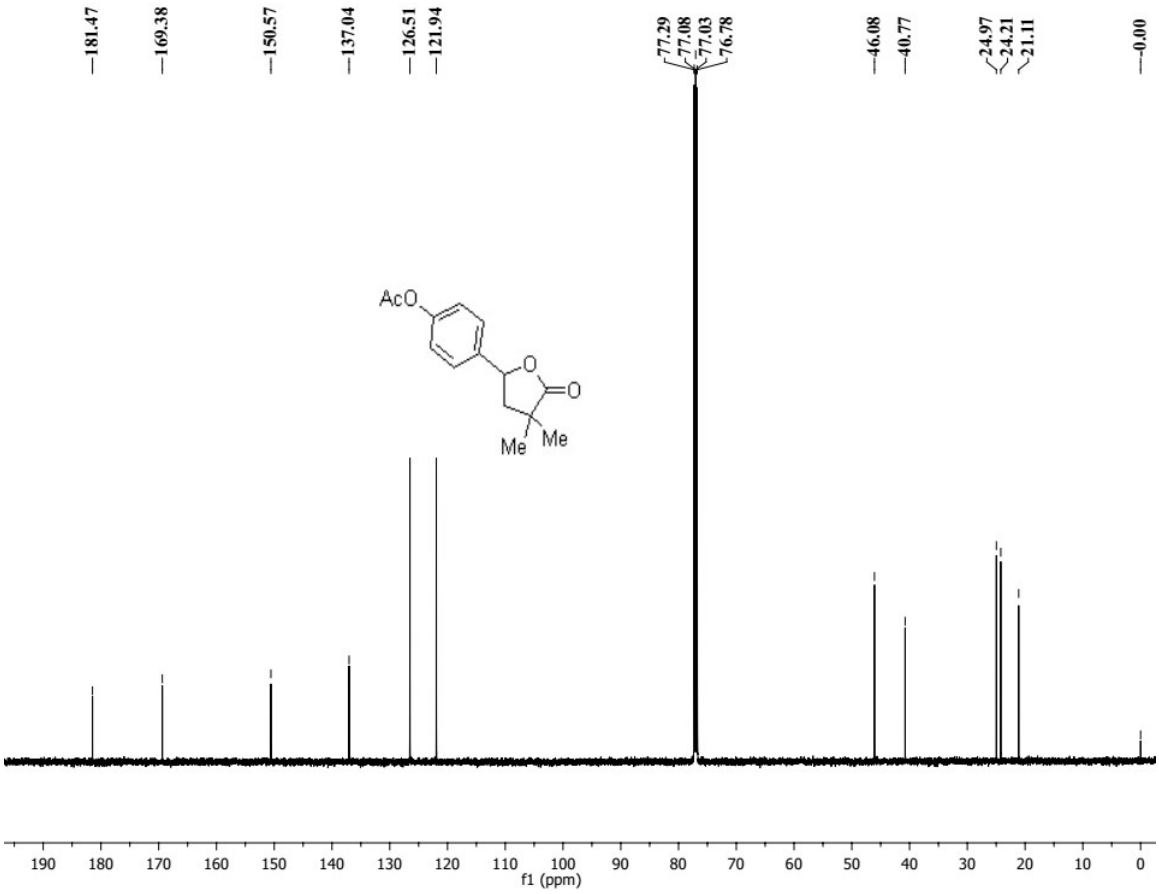

181.23
141.90
131.42
130.35
128.35
123.82
122.87
77.28
77.03
76.77
76.57
45.98
40.67
24.93
24.21
Br
O
O
Me
Me
f1 (ppm)

4-(4,4-Dimethyl-5-oxotetrahydrofuran-2-yl)phenyl acetate (5e)

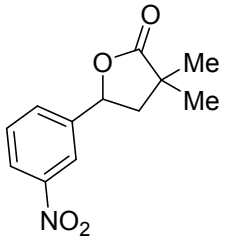


95 mg (77%); colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.28 (d, *J* = 8.4 Hz, 2H), 7.04 (d, *J* = 8.6 Hz, 2H), 5.37 (dd, *J* = 10.0, 6.3 Hz, 1H), 2.41 (dd, *J* = 12.9, 6.3 Hz, 1H), 2.23 (s, 3H), 1.99 (dd, *J* = 12.8, 10.1 Hz, 1H), 1.30 (s, 3H), 1.24 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 181.5, 169.4, 150.6, 137.0, 126.5, 121.9, 77.1, 46.1, 40.8, 25.0, 24.2, 21.1. HRMS (ESI) calcd. for $C_{14}H_{17}O_4$ $[M+H]^+$: 249.2855; found: 249.2849.

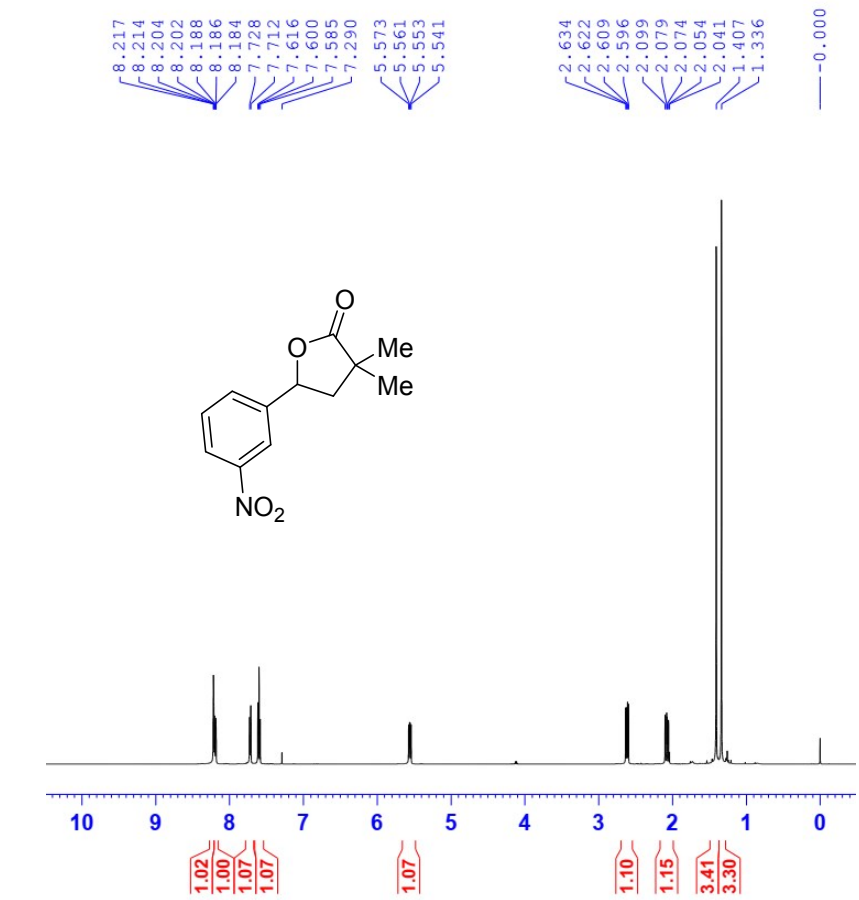


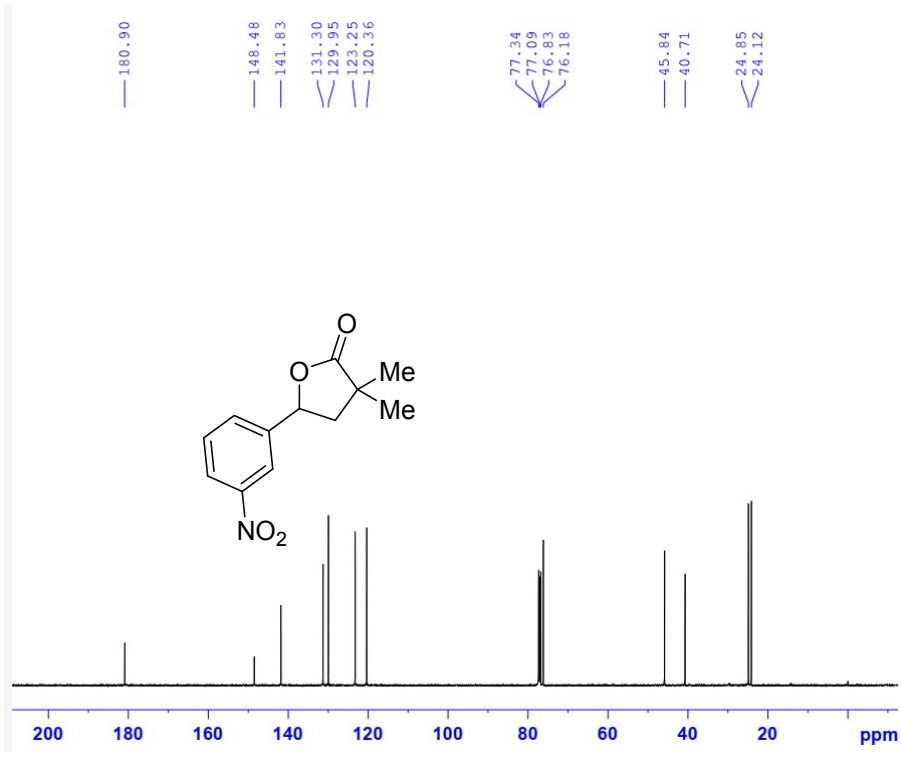


3,3-Dimethyl-5-(3-nitrophenyl)dihydrofuran-2(3*H*)-one (5f)

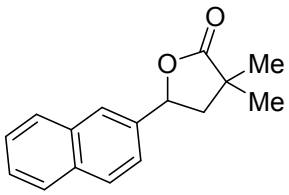


56 mg (48%); light yellow oil which slowly solidified under air. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 8.22 – 8.21 (m, 1H), 8.20 – 8.18 (m, 1H), 7.72 (d, *J* = 8.0 Hz, 1H), 7.60 (t, *J* = 8.0 Hz, 1H), 5.56 (dd, *J* = 10.0, 6.0 Hz, 1H), 2.61 (dd, *J* = 12.5, 6.0 Hz, 1H), 2.07 (dd, *J* = 12.5, 10.0 Hz, 1H), 1.41 (s, 3H), 1.34 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm) δ 180.9, 148.5, 141.8, 131.3, 129.9, 123.2, 120.4, 76.2, 45.8, 40.7, 24.8, 24.1

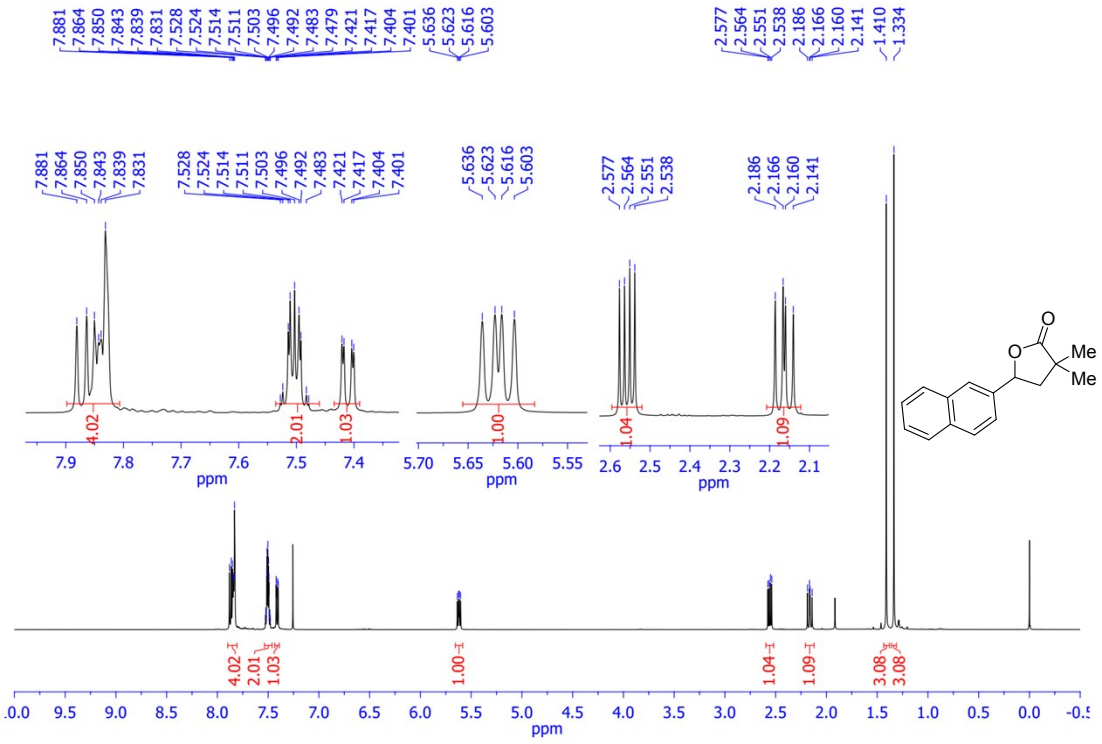

8.217
8.214
8.204
8.202
8.188
8.186
8.184
7.728
7.712
7.616
7.600
7.585
7.290
5.573
5.561
5.553
5.541
2.634
2.622
2.609
2.596
2.099
2.079
2.074
2.054
2.041
1.407
1.336
0.000
O
O
Me
Me
NO2
10
9
8
7
6
5
4
3
2
1
0
1.02
1.00
1.07
1.07
1.07
1.10
1.15
3.41
3.30

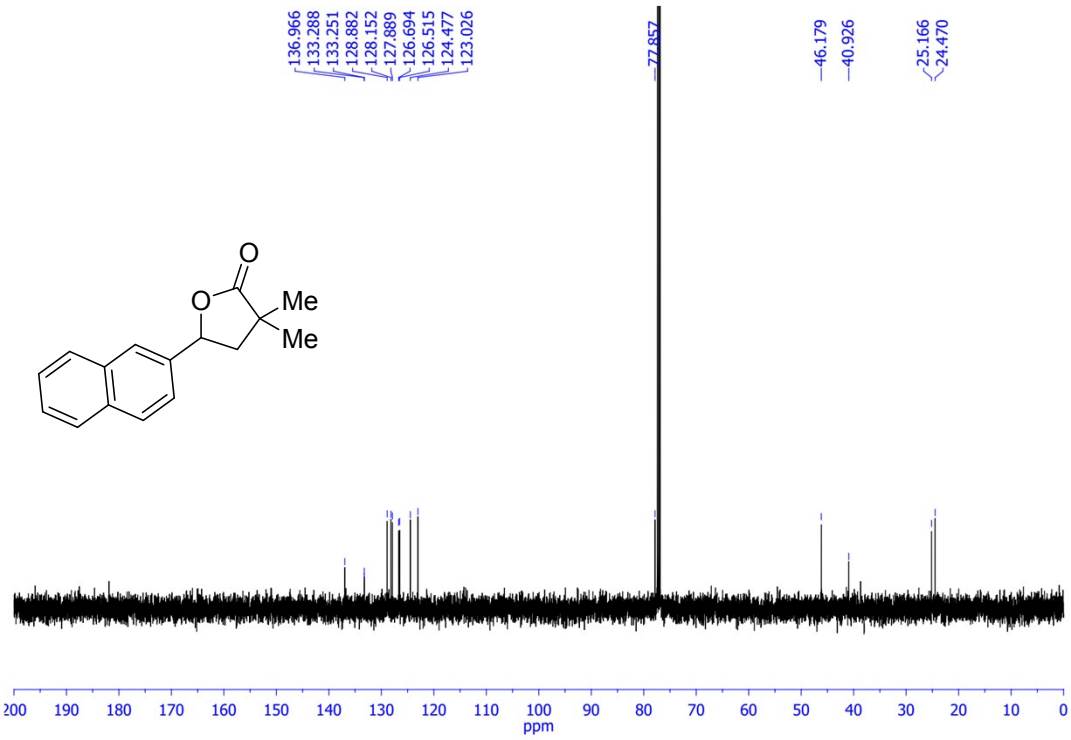

180.90
148.48
141.83
131.30
129.95
123.25
120.36
77.34
77.09
76.83
76.18
45.84
40.71
24.85
24.12
O
O
Me
Me
NO2
200
180
160
140
120
100
80
60
40
20
ppm

3,3-Dimethyl-5-(naphthalen-2-yl)dihydrofuran-2(3*H*)-one (5h)[3]

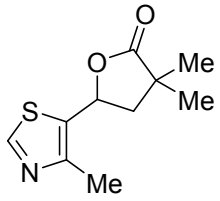


110 mg (92%); white solid. M.p. 120-122 ºC. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.90 – 7.81 (m, 4H), 7.54 – 7.46 (m, 2H), 7.41 (dd, *J* = 8.5, 1.5 Hz, 1H), 5.62 (dd, *J* = 10.0, 6.5 Hz, 1H), 2.56 (dd, *J* = 13.0, 6.5 Hz, 1H), 2.16 (dd, *J* = 13.0, 10.0 Hz, 1H), 1.41 (s, 3H), 1.33 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm) δ 137.0, 133.3, 133.2, 128.9, 128.2, 127.9, 126.7, 126.5, 124.5, 123.0, 77.9, 46.2, 40.9, 25.2, 24.5.



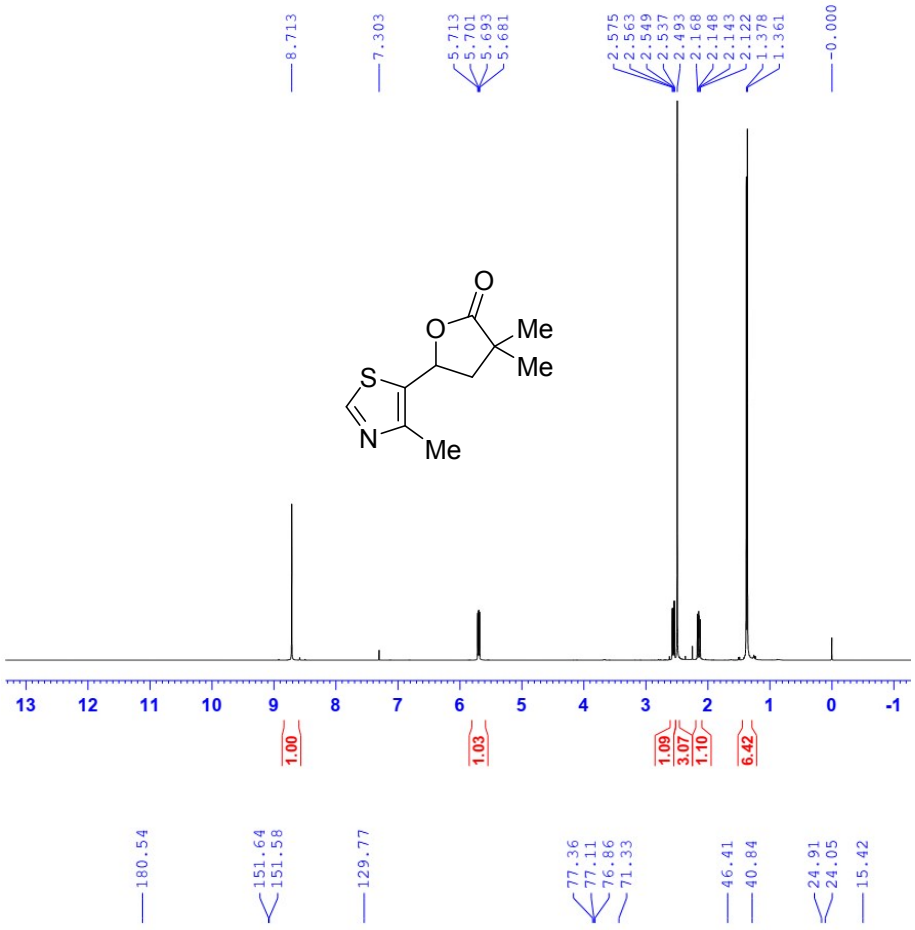


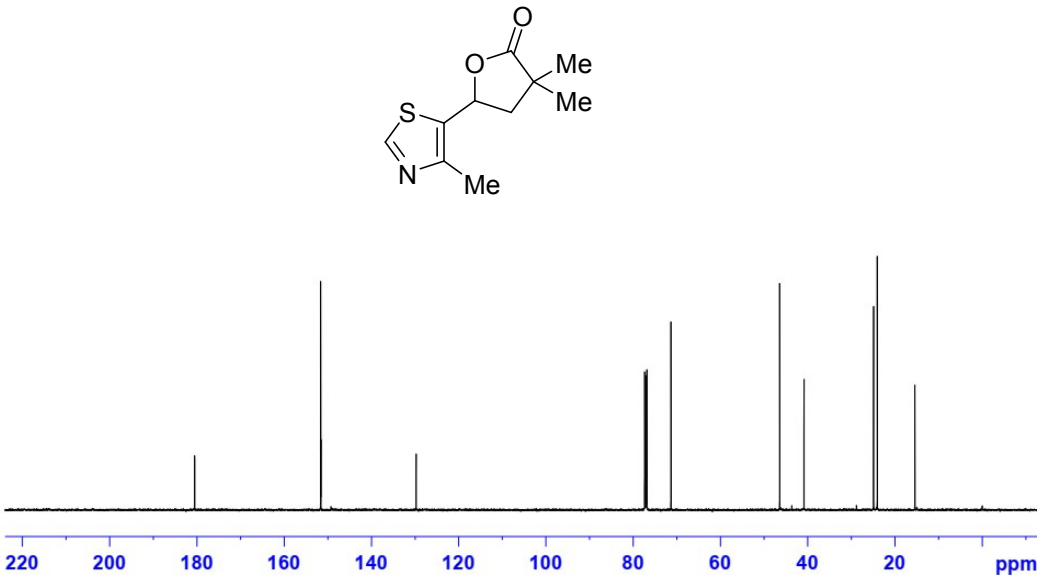
3,3-Dimethyl-5-(4-methylthiazol-5-yl)dihydrofuran-2(3*H*)-one (5i)[4]



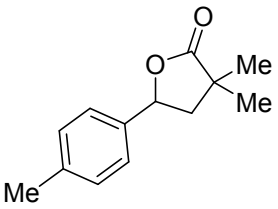
64 mg (61%); colorless oil which slowly solidified under air. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 8.71 (s, 1H), 5.70 (dd, J = 10.0, 6.0 Hz, 1H), 2.56 (dd, J = 12.5, 6.0 Hz, 1H), 2.49 (s, 3H), 2.14 (dd, J = 12.5, 10.0 Hz, 1H), 1.38 (s, 3H), 1.36 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm) δ 180.5, 151.64, 151.58, 129.8, 71.3, 46.4, 40.8, 24.9, 24.0, 15.4.

[4] Pan, G.-H.; Song, R.-J.; Li, J.-H. *Org. Chem. Front.* **2018**, *5*, 179.

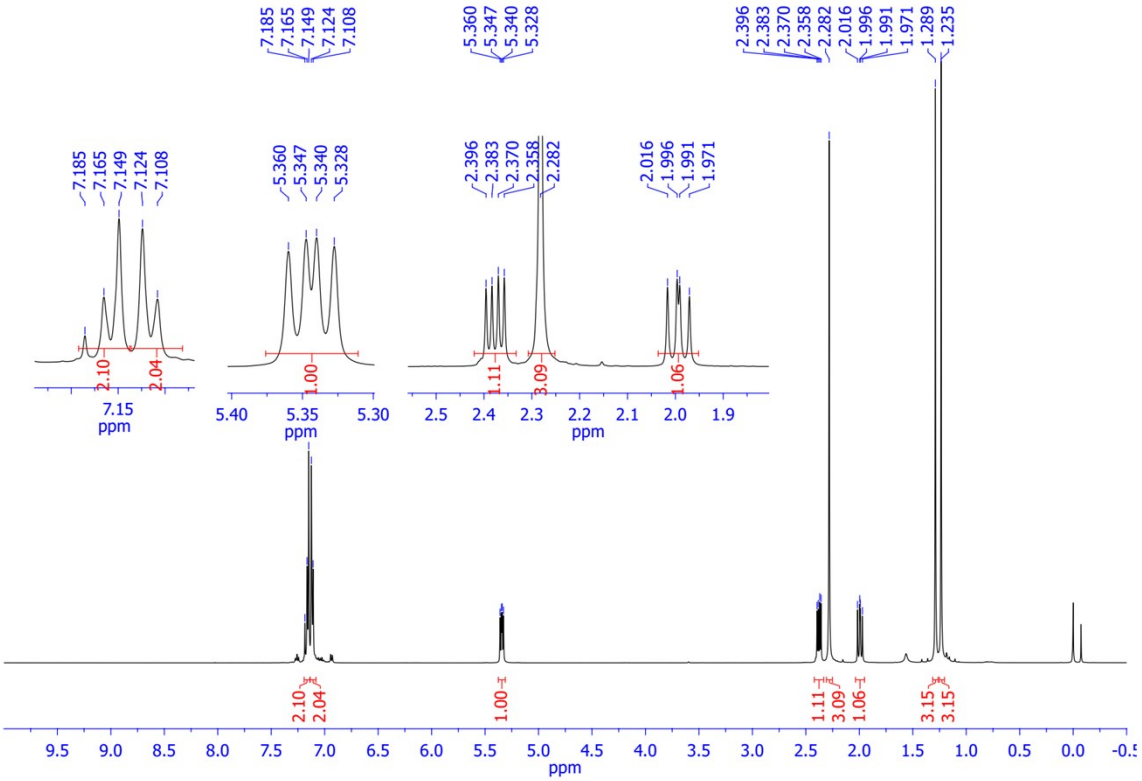

8.713
7.303
5.713
5.701
5.693
5.681
2.575
2.563
2.549
2.537
2.493
2.168
2.148
2.143
2.122
1.378
1.361
0.000
O
O
Me
Me
S
N
Me
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
1.00
1.03
1.09
3.07
1.10
6.42
180.54
151.64
151.58
129.77
77.36
77.11
76.86
71.33
46.41
40.84
24.91
24.05
15.42

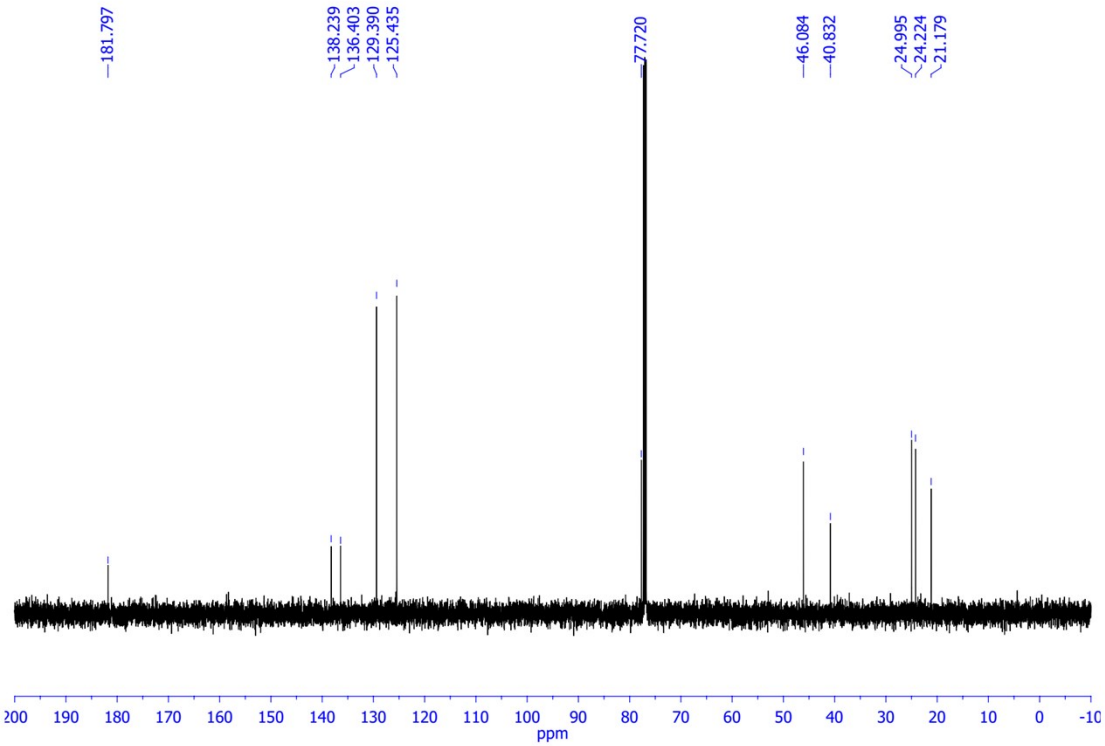

O
O
Me
Me
S
N
Me
220 200 180 160 140 120 100 80 60 40 20 ppm

3,3-Dimethyl-5-(*p*-tolyl)dihydrofuran-2(3*H*)-one (5k)[3]

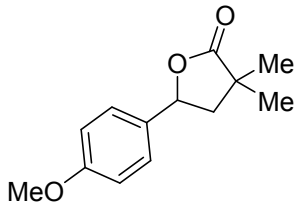


82 mg (80%); colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.19 – 7.14 (m, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 5.34 (dd, *J* = 10.0, 6.0 Hz, 1H), 2.38 (dd, *J* = 13.0, 6.0 Hz, 1H), 2.28 (s, 3H), 1.99 (dd, *J* = 13.0, 10.0 Hz, 1H), 1.29 (s, 3H), 1.23 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm) δ 181.8, 138.2, 136.4, 129.4, 125.4, 77.7, 46.1, 40.8, 25.0, 24.2, 21.2.

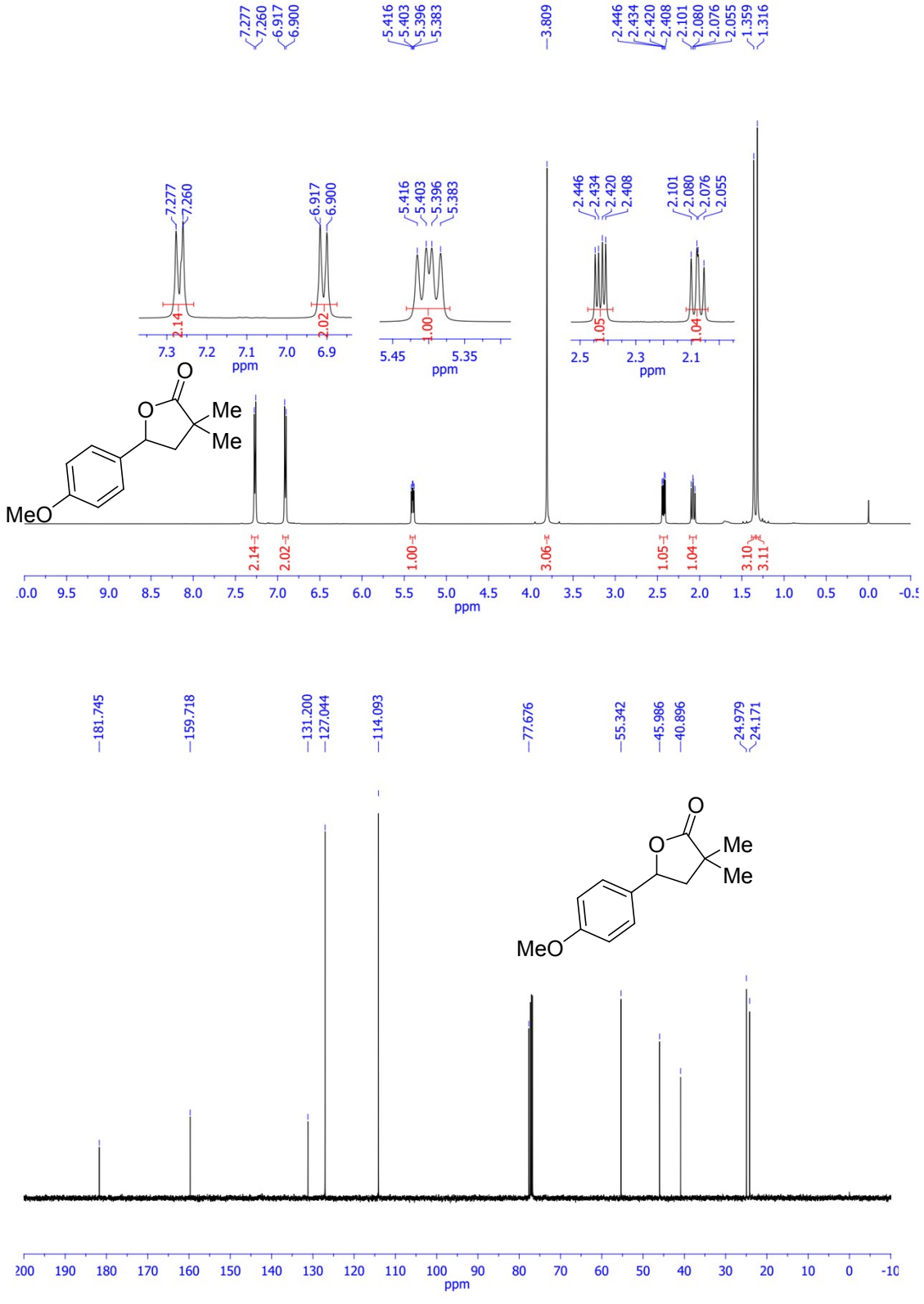




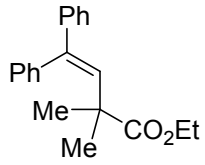
5-(4-Methoxyphenyl)-3,3-dimethyldihydrofuran-2(3*H*)-one (3cj)[3]



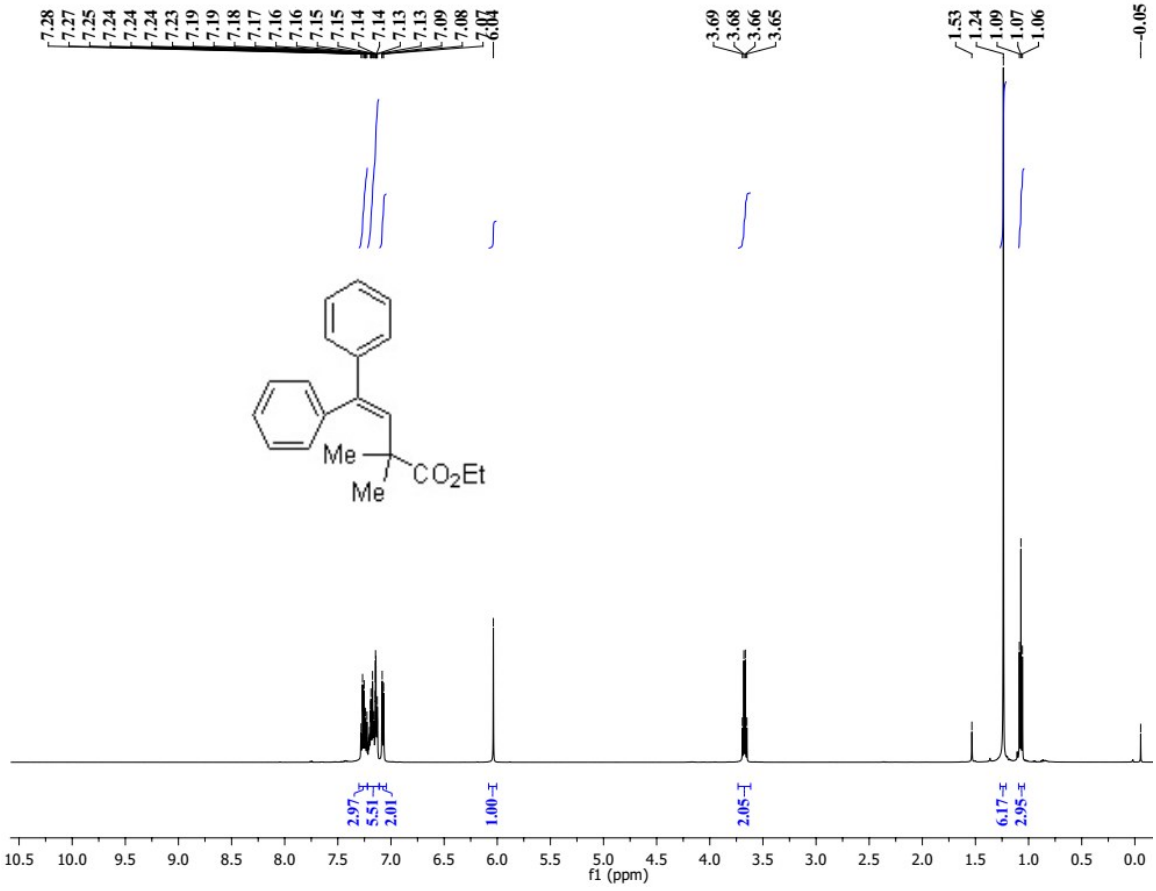
92 mg (84%); colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.27 (d, *J* = 8.5 Hz, 2H), 6.91 (d, *J* = 8.5 Hz, 2H), 5.40 (dd, *J* = 10.5, 6.0 Hz, 1H), 3.81 (s, 3H), 2.43 (dd, *J* = 12.5, 6.0 Hz, 1H), 2.08 (dd, *J* = 12.5, 10.5 Hz, 1H), 1.36 (s, 3H), 1.32 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm) δ 181.7, 159.7, 131.2, 127.0, 114.1, 77.7, 55.3, 46.0, 40.9, 25.0, 24.2.



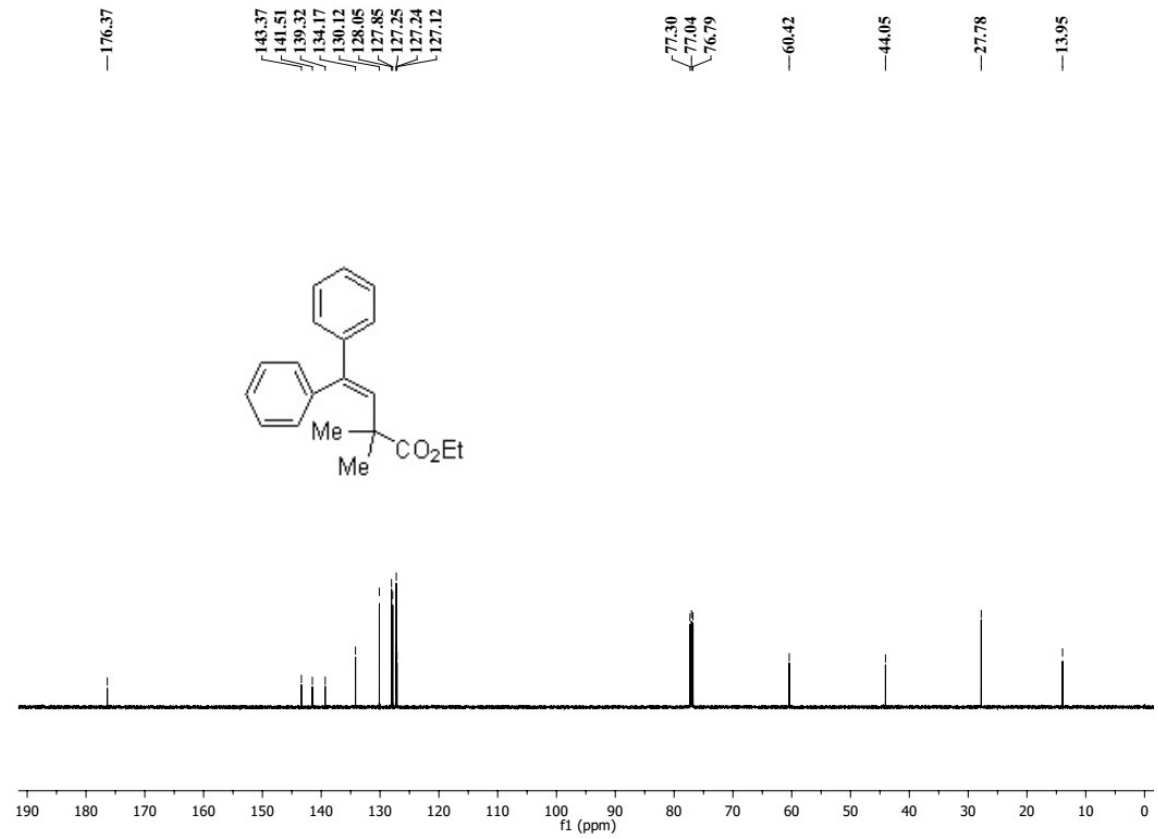
Ethyl 2,2-dimethyl-4,4-diphenylbut-3-enoate (7a)[5]



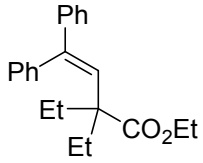
100 mg (68%); colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.30 – 7.22 (m, 3H), 7.22 – 7.11 (m, 5H, overlapping with $CHCl_3$ signal), 7.11 – 7.05 (m, 2H), 6.04 (s, 1H), 3.67 (q, *J* = 7.1 Hz, 2H), 1.24 (s, 6H), 1.07 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$, ppm) δ 176.4, 143.4, 141.5, 139.3, 134.2, 130.1, 128.0, 127.8, 127.25, 127.24, 127.1, 60.4, 44.0, 27.8, 13.9.



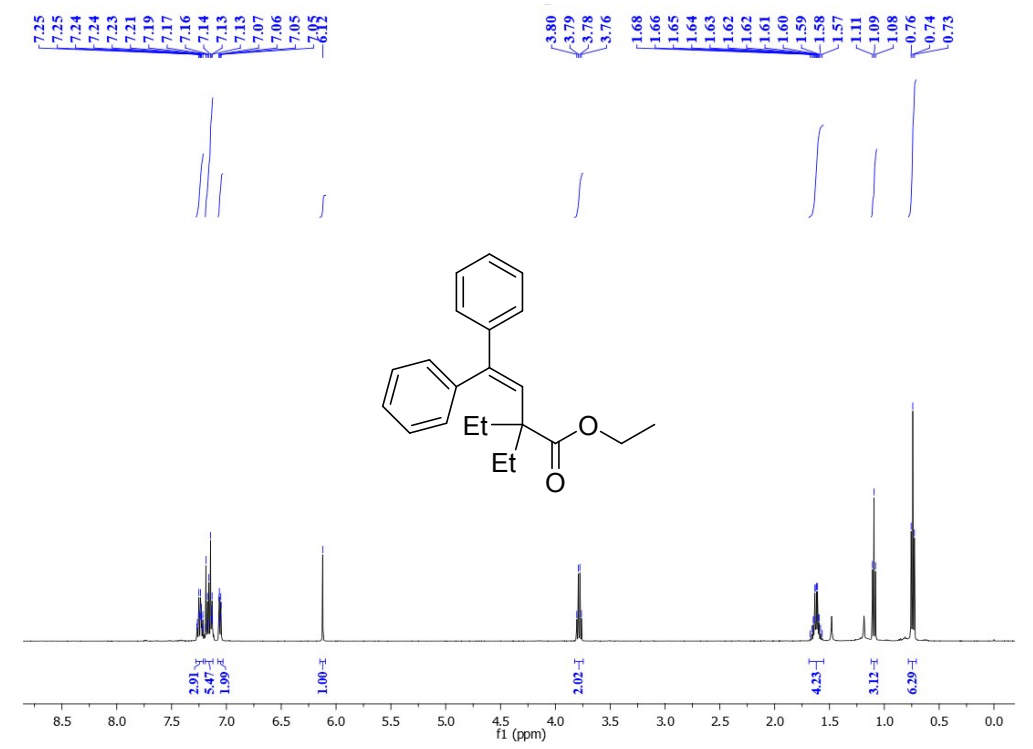
[5] Liu, C.; Tang, S.; Liu, D.; Yuan, J.; Zheng, L.; Meng, L.; Lei, A. *Angew. Chem. Int. Ed.* **2012,** *51*, 3638.

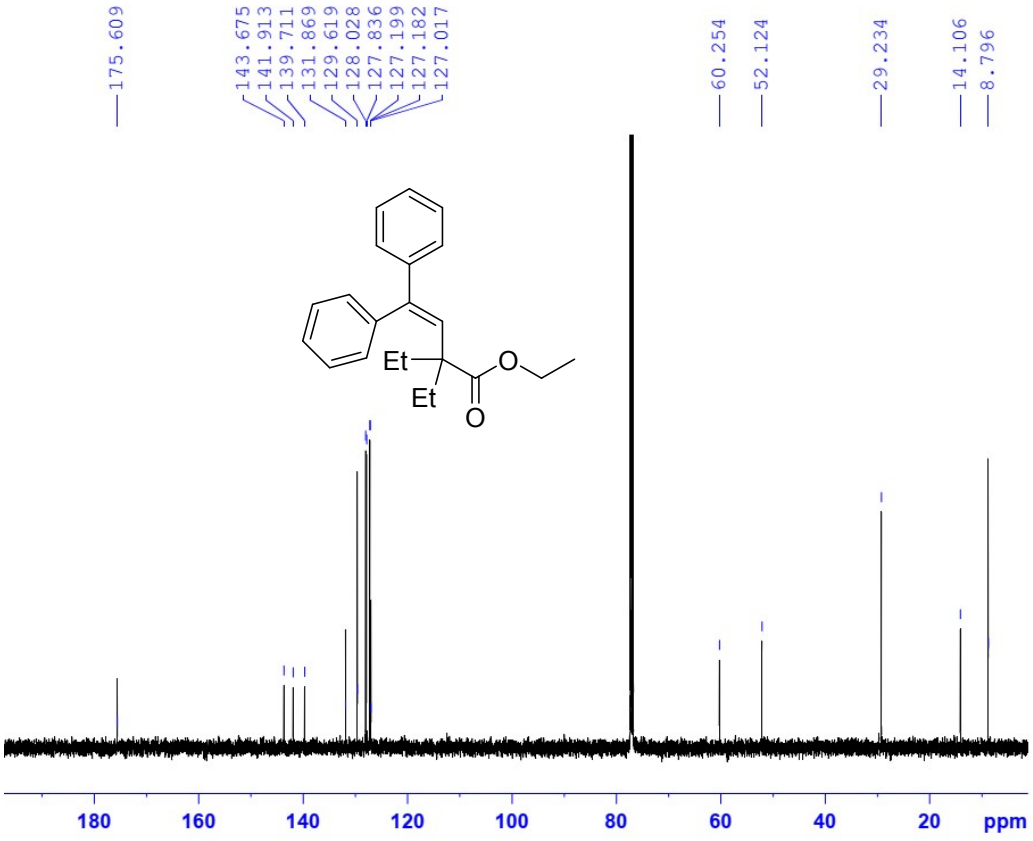


Ethyl 2,2-diethyl-4,4-diphenylbut-3-enoate (7b)

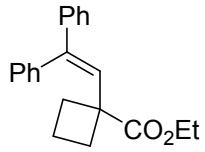


114 mg (71%); colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.28 – 7.21 (m, 2H), 7.19 – 7.12 (m, 5H, overlapping with $CHCl_3$ signal), 7.08 – 7.03 (m, 2H), 6.12 (s, 1H), 3.78 (q, J = 7.1 Hz, 2H), 1.69 – 1.56 (m, 4H), 1.09 (t, J = 7.1 Hz, 3H), 0.74 (t, J = 7.5 Hz, 6H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm) δ 175.6, 143.6, 141.9, 139.7, 131.8, 129.6, 128.0, 127.8, 127.19, 127.18, 127.0, 60.2, 52.1, 29.2, 14.1, 8.8. HRMS (ESI) calcd. for $C_{22}H_{27}O_2$ $[M+H]^+$ 323.2006; found: 323.2012.

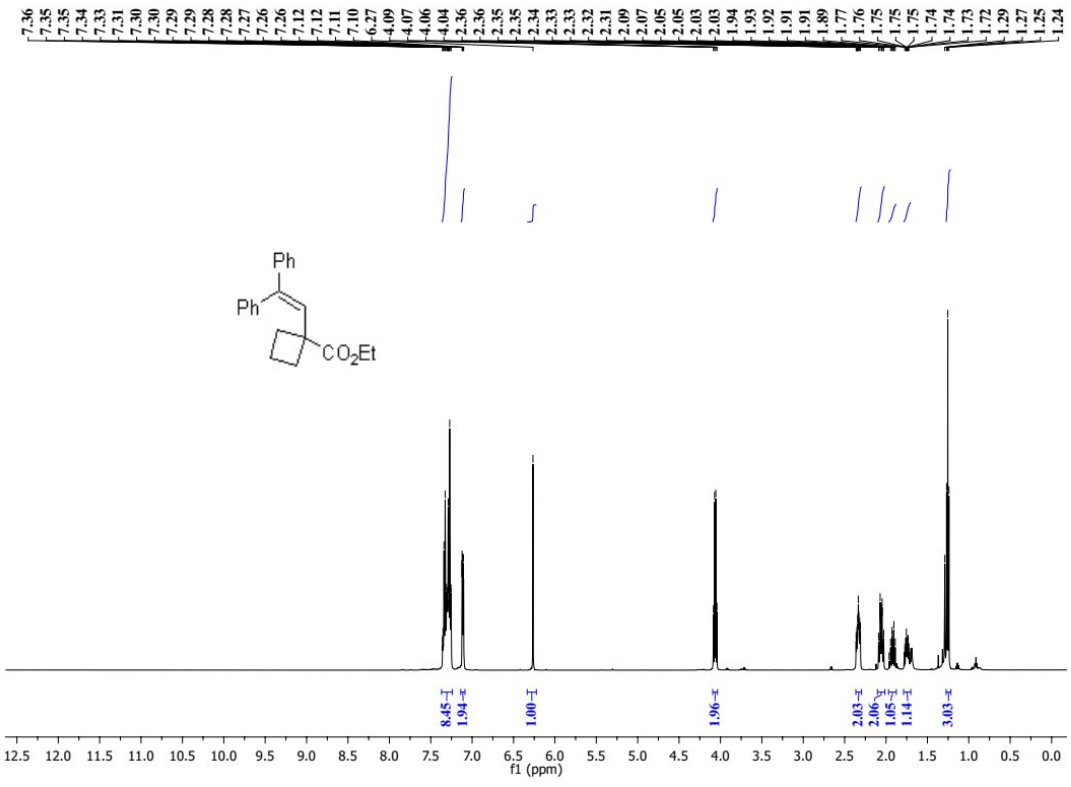

7.25
7.25
7.24
7.24
7.23
7.21
7.19
7.17
7.16
7.14
7.13
7.13
7.07
7.06
7.05
7.05
6.12
3.80
3.79
3.78
3.76
1.68
1.66
1.65
1.64
1.63
1.62
1.62
1.61
1.60
1.59
1.58
1.57
1.11
1.09
1.08
0.76
0.74
0.73
Et
O
Et
O
2.91
5.47
1.99
1.00
2.02
4.23
3.12
6.29
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

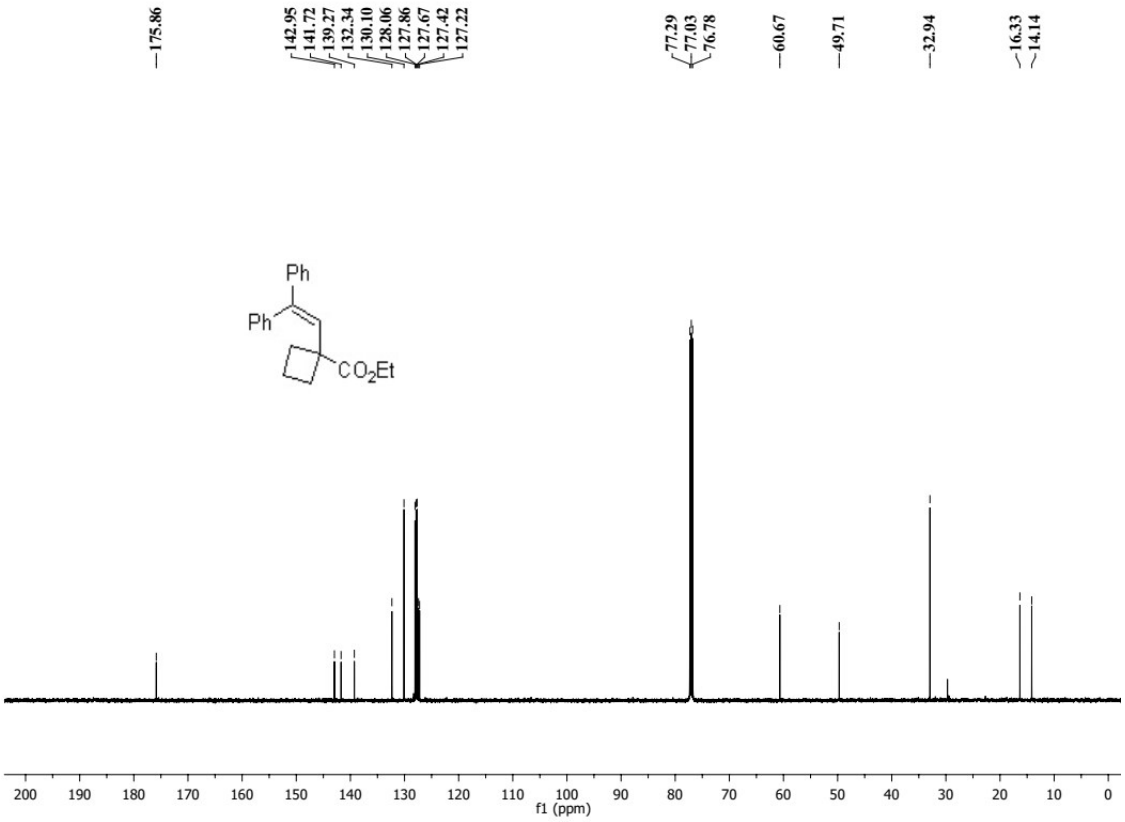

175.609
143.675
141.913
139.711
131.869
129.619
128.028
127.836
127.199
127.182
127.017
60.254
52.124
29.234
14.106
8.796
Et
O
Et
O
180
160
140
120
100
80
60
40
20
ppm

Ethyl 1-(2,2-diphenylvinyl)cyclobutane-1-carboxylate (7c)



122 mg (80%); colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.37 – 7.24 (m, 8H, overlapping with $CHCl_3$ signal), 7.11 (dd, *J* = 7.7, 1.5 Hz, 2H), 6.27 (s, 1H), 4.06 (q, *J* = 7.1 Hz, 2H), 2.37 – 2.29 (m, 2H), 2.05 (dt, *J* = 9.4, 5.6 Hz, 2H), 1.93 (ddd, *J* = 17.3, 10.9, 8.8 Hz, 1H), 1.74 (dtd, *J* = 15.5, 9.2, 4.4 Hz, 1H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$, ppm) δ 175.9, 142.9, 141.7, 139.3, 132.3, 130.1, 128.1, 127.9, 127.7, 127.4, 127.2, 60.7, 49.7, 32.9, 16.3, 14.1. HRMS (ESI) calcd. for $C_{21}H_{23}O_2$ $[M+H]^+$ 307.1693; found: 307.1690.




175.86
142.95
141.72
139.27
132.34
130.10
128.06
127.86
127.67
127.42
127.22
77.29
77.03
76.78
60.67
49.71
32.94
16.33
14.14
Ph
Ph
CO2Et
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)