Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Radical-Mediated Synthesis of γ-Lactones by Copper-Catalyzed Intermolecular Carboesterification of Alkenes with α-Carbonyl Alkyl Bromides and H_2O

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Supporting Information

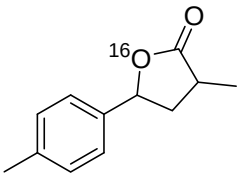
List of Contents

(A) Typical experimental procedure

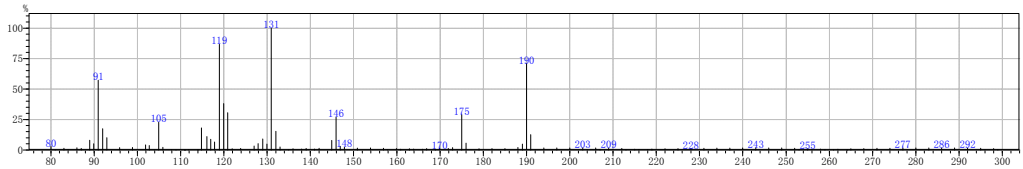
(a) Typical experimental procedure

To a Schlenk tube were added alkenes **1** (0.2 mmol), α-carbonyl alkyl bromides **2** (0.4 mmol), $Cu(OTf)_2$ (0.02 mmol), 1,10-phenanthroline (0.04 mmol), Ag_2CO_3 (0.2 mmol), H_2O (4 equiv) and MeCN (2 mL), Then the tube was charged with argon, and was stirred at 120 ^oC for 16 h until complete consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the reaction mixture was concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the desired product **3**.

(b) The O^{18}-Labelled Control Experiment:



Chemical Formula: $C_{12}H_{14}O^{16}O$
Exact Mass: 190.10



[MS Spectrum]

# of Peaks 121

Raw Spectrum 9.960 (scan : 1293) Base Peak m/z 130.95 (Inten : 21,937)

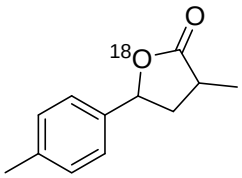
Background 10.250 (scan : 1351)

m/z Absolute Intensity Relative Intensity

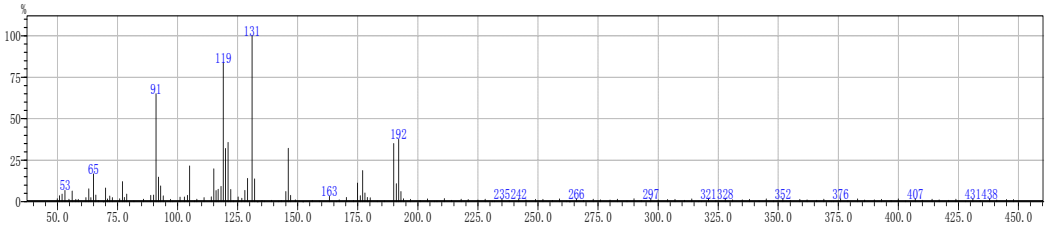
80.00	615	2.80	89.90	1098	5.01
81.00	42	0.19	90.95	12559	57.25
83.00	302	1.38	92.00	3828	17.45
84.00	60	0.27	92.95	2205	10.05
86.00	386	1.76	95.90	406	1.85
87.00	260	1.19	98.90	446	2.03
89.00	1754	8.00	101.90	914	4.17

102.75	789	3.60	144.95	1719	7.84	203.00	356	1.62
103.90	31	0.14	145.95	5884	26.82	204.00	60	0.27
104.95	4983	22.72	146.90	637	2.90	206.00	343	1.56
105.90	460	2.10	147.90	452	2.06	207.00	120	0.55
107.90	35	0.16	150.90	319	1.45	209.00	309	1.41
108.90	42	0.19	153.90	359	1.64	210.00	47	0.21
110.90	3	0.01	156.90	290	1.32	212.00	267	1.22
111.90	90	0.41	157.90	34	0.15	213.00	63	0.29
113.90	1	0.00	159.90	226	1.03	215.00	152	0.69
114.85	3974	18.12	160.90	155	0.71	216.00	103	0.47
116.05	2427	11.06	162.90	204	0.93	218.00	90	0.41
116.95	1910	8.71	163.90	184	0.84	219.00	174	0.79
117.85	1417	6.46	165.90	156	0.71	222.00	191	0.87
118.95	19015	86.68	166.90	242	1.10	225.00	239	1.09
119.95	8350	38.06	168.90	78	0.36	228.00	258	1.18
120.90	6706	30.57	169.90	276	1.26	231.00	289	1.32
121.90	208	0.95	171.90	249	1.14	234.00	316	1.44
123.90	257	1.17	172.90	398	1.81	237.00	322	1.47
127.00	697	3.18	175.00	6358	28.98	240.00	348	1.59
127.95	1141	5.20	176.00	1218	5.55	243.00	364	1.66
129.00	1979	9.02	179.00	204	0.93	246.00	338	1.54
129.95	1052	4.80	182.00	239	1.09	249.00	354	1.61
130.95	21937	100.00	185.00	247	1.13	252.00	297	1.35
132.00	3359	15.31	**188.00**	**425**	**1.94**	253.00	60	0.27
133.00	522	2.38	**189.05**	**1053**	**4.80**	255.00	216	0.98
135.00	61	0.28	**190.00**	**15538**	**70.83**	256.00	79	0.36
136.00	207	0.94	**190.95**	**2779**	**12.67**	258.00	168	0.77
138.00	169	0.77	**192.00**	**23**	**0.10**	259.00	110	0.50
139.00	255	1.16	194.00	351	1.60	261.00	60	0.27
141.00	185	0.84	197.00	343	1.56	262.00	151	0.69
142.00	298	1.36	200.00	345	1.57	264.00	1	0.00

265.00	215	0.98	280.00	340	1.55	295.00	295	1.34
268.00	228	1.04	283.00	342	1.56	296.00	36	0.16
271.00	286	1.30	286.00	356	1.62	298.00	231	1.05
274.00	294	1.34	289.00	348	1.59	299.00	73	0.33
277.00	326	1.49	292.00	332	1.51			



Chemical Formula: $C_{12}H_{14}O^{18}O$
Exact Mass: 192.10



[MS Spectrum]

# of Peaks 166

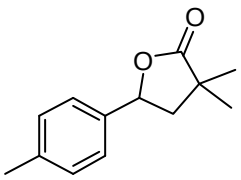
Raw Spectrum 11.390
(scan : 1579) Base Peak m/z 131.00
(Inten : 51,268)

Background 11.485
(scan : 1598)

m/z	Absolute Intensity	Relative Intensity
49.90	906	1.77
50.85	1829	3.57
51.90	2312	4.51
53.10	3479	6.79
54.75	768	1.50
56.05	3272	6.38
57.60	682	1.33
58.60	685	1.34
61.80	1211	2.36
63.00	3947	7.70
63.90	1201	2.34
64.90	8560	16.70
65.95	2068	4.03
67.00	271	0.53
69.00	3	0.01
70.05	4199	8.19
70.80	839	1.64
71.80	1712	3.34
72.80	1233	2.41
75.85	875	1.71
77.00	6176	12.05
77.80	1270	2.48
78.80	2331	4.55
81.80	124	0.24
85.80	720	1.40
88.80	1879	3.67
90.05	2068	4.03
91.00	33368	65.09
92.00	7554	14.73
92.95	4801	9.36
94.00	1740	3.39
97.00	701	1.37
98.00	98	0.19
101.00	1335	2.60
102.85	1402	2.73
104.05	1929	3.76
105.00	11032	21.52
107.00	225	0.44
108.00	770	1.50
111.00	1225	2.39
114.00	1519	2.96
115.05	10119	19.74
115.95	3421	6.67
116.90	3821	7.45
118.05	4710	9.19
119.00	42923	83.72
119.95	16378	31.95
121.00	18319	35.73
122.15	3763	7.34
123.15	223	0.43
125.20	1412	2.75
126.70	1044	2.04
128.00	3462	6.75
129.10	7148	13.94
131.00	51268	100.00
132.05	6993	13.64
133.10	34	0.07
135.10	263	0.51
136.10	140	0.27
139.10	402	0.78
142.10	130	0.25
143.10	242	0.47
145.00	3147	6.14
146.05	16448	32.08
147.00	1922	3.75
149.00	556	1.08
152.00	9	0.02
156.00	352	0.69
159.00	144	0.28
163.15	1626	3.17
166.20	81	0.16
167.20	518	1.01
170.20	1303	2.54
173.20	358	0.70
174.90	5688	11.09
176.00	1853	3.61
177.05	9561	18.65
177.95	2654	5.18
179.00	1301	2.54
180.15	1143	2.23
183.10	130	0.25
186.10	**192**	**0.37**
190.00	**17986**	**35.08**
191.05	**5492**	**10.71**

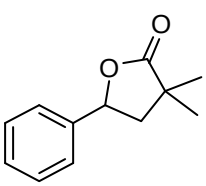
192.00	**19265**	**37.58**	228.00	725	1.41	266.00	902	1.76
192.95	**3139**	**6.12**	231.00	69	0.13	269.00	317	0.62
194.00	870	1.70	232.00	148	0.29	273.00	756	1.47
197.00	678	1.32	235.00	883	1.72	276.00	547	1.07
201.00	268	0.52	238.00	299	0.58	280.00	470	0.92
204.00	846	1.65	242.00	833	1.62	283.00	741	1.45
207.00	61	0.12	245.00	454	0.89	287.00	188	0.37
208.00	143	0.28	249.00	657	1.28	290.00	864	1.69
211.00	923	1.80	252.00	657	1.28	293.00	254	0.50
214.00	383	0.75	256.00	313	0.61	297.00	919	1.79
218.00	735	1.43	259.00	829	1.62			
221.00	586	1.14	262.00	141	0.28			
225.00	478	0.93	263.00	13	0.03			

(B) Analytical data:



3,3-Dimethyl-5-(*p*-tolyl)dihydrofuran-2(3*H*)-one (3aa):

Colorless oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.24-7.17 (m, 4H), 5.43-5.39 (m, 1H), 2.47-2.42 (m, 1H), 2.35 (s, 3H), 2.09-2.03(m, 1H), 1.35 (s, 3H), 1.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.7, 138.1, 136.3, 129.3, 125.3, 77.6, 46.0, 40.8, 24.9, 24.1, 21.1; LRMS (EI, 70 eV) *m/z* (%): 204 (M^+, 22), 160 (29), 145 (100); HRMS *m/z* (ESI) calcd for $C_{13}H_{17}O_2$ ($[M+H]^+$) 205.1223, found 205.1230.

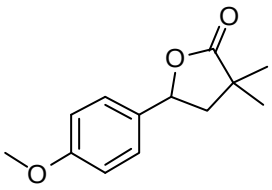


3,3-Dimethyl-5-phenyldihydrofuran-2(3*H*)-one (3ba):[1]

Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.40-7.26 (m, 5H), 5.47-5.43 (m, 1H), 2.50-2.46 (m, 1H), 2.10-2.05 (m, 1H), 1.37 (s,

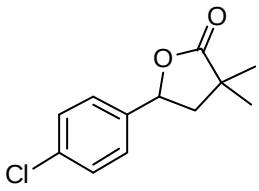
3H), 1.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.6, 139.4, 128.7, 128.3, 125.3, 77.6, 46.0, 40.7, 24.9, 24.2; (EI, 70 eV) *m/z* (%): 190 (M^+, 26), 160 (31), 145 (100); HRMS *m/z* (ESI) calcd for $C_{12}H_{15}O_2$ ($[M+H]^+$) 191.1067, found 191.1074.

5-(4-Methoxyphenyl)-3,3-dimethyldihydrofuran-2(3*H*)-one (3ca):



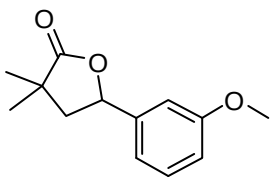
White solid, mp 79.7-80.5 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.27 (d, *J* = 8.0 Hz, 2H), 6.91 (d, *J* = 8.0 Hz, 2H), 5.42-5.38 (m, 1H), 3.81 (s, 3H), 2.45-2.40 (m, 1H), 2.11-2.05 (m, 1H), 1.36 (s, 3H), 1.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.7, 159.7, 131.1, 127.0, 114.0, 77.6, 55.3, 45.9, 40.8, 24.9, 24.1; LRMS (EI, 70 eV) *m/z* (%): 220 (M^+, 46), 161 (100), 135 (58); HRMS *m/z* (ESI) calcd for $C_{13}H_{17}O_3$ ($[M+H]^+$) 221.1172, found 221.1179.

5-(4-Chlorophenyl)-3,3-dimethyldihydrofuran-2(3*H*)-one (3da):[2]



White solid, mp 54.1-55.5 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.36 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.8 Hz, 2H), 5.44-5.40 (m, 1H), 2.51-2.46 (m, 1H), 2.05-2.00 (m, 1H), 1.37 (s, 3H), 1.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.3, 138.0, 134.2, 129.0, 126.7, 76.8, 46.0, 40.7, 24.9, 24.2; LRMS (EI, 70 eV) *m/z* (%): 224 (M^++2, 7), 224 (M^+, 22), 165 (100), 145 (75); HRMS *m/z* (ESI) calcd for $C_{12}H_{14}ClO_2$ ($[M+H]^+$) 225.0677, found 225.0670.

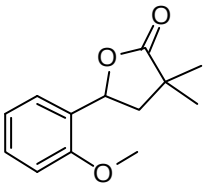
5-(3-Methoxyphenyl)-3,3-dimethyldihydrofuran-2(3*H*)-one (3ea):



Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.32-7.26 (m, 1H), 6.91-6.86 (m, 3H), 5.45-5.41 (m, 1H), 3.82 (s, 3H), 2.51-2.46 (m, 1H), 2.10-2.04 (m, 1H), 1.37 (s, 3H), 1.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ:

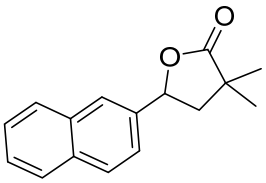
181.6, 159.9, 141.2, 129.8, 117.4, 113.8, 110.7, 77.4, 55.3, 46.0, 40.7, 25.0, 24.2; LRMS (EI, 70 eV) *m/z* (%): 220 (M^+, 63), 161 (100), 136 (31); HRMS *m/z* (ESI) calcd for $C_{13}H_{17}O_3$ ($[M+H]^+$) 221.1172, found 221.1180.

5-(2-Methoxyphenyl)-3,3-dimethyldihydrofuran-2(3*H*)-one (3fa):[3]



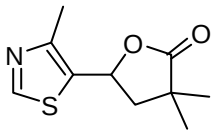
White solid, mp 77.1-79.3 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.38 (d, *J* = 7.6 Hz, 1H), 7.29 (t, *J* = 7.8 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 1H), 6.89 (d, *J* = 8.2 Hz, 1H), 5.72-5.68 (m, 1H), 3.84 (s, 3H), 2.59-2.54 (m, 1H), 2.02-1.97 (m, 1H), 1.37 (s, 3H), 1.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 182.1, 156.0, 129.0, 128.1, 125.5, 120.7, 110.4, 74.1, 55.3, 44.5, 40.4, 25.1, 24.7; LRMS (EI, 70 eV) *m/z* (%): 220 (M^+, 48), 161 (100), 121 (34); HRMS *m/z* (ESI) calcd for $C_{13}H_{17}O_3$ ($[M+H]^+$) 221.1172, found 221.1165.

3,3-dimethyl-5-(naphthalen-2-yl)dihydrofuran-2(3*H*)-one (3ga):



White solid, mp 122.1-123.1 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.89-7.83 (m, 4H), 7.51-7.49 (m, 2H), 7.41 (d, *J* = 8.4 Hz, 1H), 5.64-5.60 (m, 1H), 2.58-2.53 (m, 1H), 2.19-2.13 (m, 1H), 1.41 (s, 3H), 1.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.8, 136.8, 133.2, 133.1, 128.8, 128.0, 127.8, 126.6, 126.4, 124.4, 123.0, 77.7, 46.1, 40.8, 25.0, 24.4; LRMS (EI, 70 eV) *m/z* (%): 240 (M^+, 69), 181 (100), 156 (57); HRMS *m/z* (ESI) calcd for $C_{16}H_{17}O_2$ ($[M+H]^+$) 241.1223, found 241.1215.

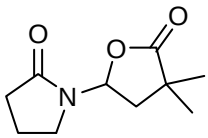
3,3-Dimethyl-5-(4-methylthiazol-5-yl)dihydrofuran-2(3*H*)-one (3ha):



Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.72 (s, 1H), 5.72-5.68 (m, 1H), 2.58-2.53 (m, 1H), 2.50 (s, 3H), 2.18-2.12 (m, 1H), 1.39 (s, 3H), 1.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.5, 151.7, 151.6, 129.7, 100.0, 71.3, 46.4, 40.8, 24.9, 24.0, 15.4; LRMS (EI, 70 eV) *m/z* (%): 211(M^+, 31),

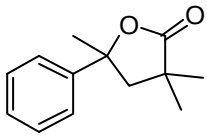
152 (100), 125 (66); HRMS *m/z* (ESI) calcd for $C_{10}H_{14}NO_2S$ ($[M+H]^+$) 212.0740, found 212.0748.

1-(4,4-Dimethyl-5-oxotetrahydrofuran-2-yl)pyrrolidin-2-one (3ia):



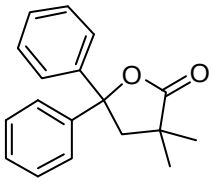
White solid, mp 78.2-79.3 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.40-6.36 (m, 1H), 3.48-3.41 (m, 2H), 2.47 (t, *J* = 8.0 Hz, 2H), 2.23-2.18 (m, 1H), 2.14-2.05 (m, 3H), 1.34 (s, 6H); ^{13}C NMR (100MHz, $CDCl_3$) δ: 179.7, 176.2, 79.0, 41.4, 41.0, 39.4, 31.3, 24.7, 24.6, 17.8; LRMS (EI, 70 eV) *m/z* (%): 197 (M^+, 4), 153 (48), 138 (100); HRMS *m/z* (ESI) calcd for $C_{10}H_{16}NO_3$ ($[M+H]^+$) 198.1125, found 198.1134.

3,3,5-Trimethyl-5-phenyldihydrofuran-2(3*H*)-one (3ja):[4]



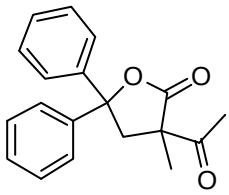
Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.40-7.34 (m, 4H), 7.27 (t, *J* = 6.8 Hz, 1H), 2.56 (d, *J* = 12.8 Hz, 1H), 2.36 (d, *J* = 12.8 Hz, 1H), 1.71 (s, 3H), 1.34 (s, 3H), 0.97 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.7, 145.8, 128.5, 127.3, 123.9, 83.4, 50.6, 40.8, 32.0, 26.6, 25.8; LRMS (EI, 70 eV) *m/z* (%): 204 (M^+, 7), 145 (72), 105 (100); HRMS *m/z* (ESI) calcd for $C_{13}H_{17}O_2$ ($[M+H]^+$) 205.1223, found 205.1230.

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



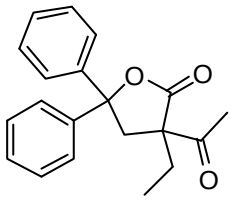
White solid, mp 73.5-75.1 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.46 (d, *J* = 8.0 Hz, 4H), 7.32 (t, *J* = 7.6 Hz, 4H), 7.23 (t, *J* = 7.2 Hz, 2H), 2.92 (s, 2H), 1.14 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.2, 144.7, 128.5, 127.5, 124.9, 86.1, 49.9, 40.5, 25.7; LRMS (EI, 70 eV) *m/z* (%): 266(M^+, 39), 183 (49), 105 (100); HRMS *m/z* (ESI) calcd for $C_{18}H_{19}O_2$ ($[M+H]^+$) 267.1380, found 267.1369.

3-Acetyl-3-methyl-5,5-diphenyldihydrofuran-2(3*H*)-one (3kg):[6]



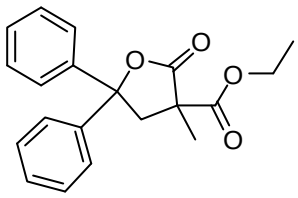
White solid, mp 71.3-73.2 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.35 (t, *J* = 8.0 Hz, 4H), 7.28-7.16 (m, 6H), 3.72 (d, *J* = 13.6 Hz, 1H), 2.64 (d, *J* = 13.2 Hz, 1H), 2.04 (s, 3H), 1.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 203.1, 175.9, 143.7, 142.7, 128.6, 128.5, 127.9, 127.9, 125.3, 124.9, 87.1, 57.9, 45.1, 25.7, 22.4; LRMS (EI, 70 eV) *m/z* (%): 294 (M^+, 12), 207 (100), 105 (54); HRMS *m/z* (ESI) calcd for $C_{19}H_{19}O_3$ ($[M+H]^+$) 295.1329, found 295.1338.

3-Acetyl-3-ethyl-5,5-diphenyldihydrofuran-2(3*H*)-one (3kh):



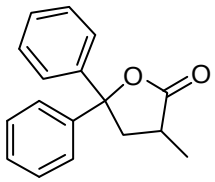
Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.34-7.33 (m, 4H), 7.28-7.16 (m, 6H), 3.71 (d, *J* = 13.2 Hz, 1H), 2.62 (d, *J* = 13.2 Hz, 1H), 2.03 (s, 3H), 1.92-1.85 (m, 1H), 1.76-1.69 (m,1H), 0.77 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 202.1, 175.4, 143.9, 142.7, 128.6, 128.5, 127.9, 127.8, 125.4, 125.0, 87.2, 63.1, 41.5, 29.1, 25.8, 8.98; LRMS (EI, 70 eV) *m/z* (%): 308 (M^+, 11), 221 (100), 105 (81); HRMS *m/z* (ESI) calcd for $C_{20}H_{21}O_3$ ($[M+H]^+$) 309.1485, found 309.1478.

Ethyl 3-methyl-2-oxo-5,5-diphenyltetrahydrofuran-3-carboxylate (3ki):[5]



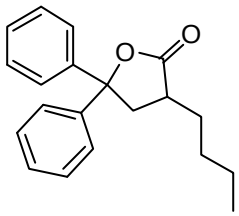
Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.45 (d, *J* = 7.6 Hz, 2H), 7.41 (d, *J* = 8.0 Hz, 2H), 7.35-7.30 (m, 4H), 7.27-7.22 (m, 2H), 3.92-3.84 (m,1H), 3.77- 3.67 (m, 2H), 2.82 (d, *J* = 13.2 Hz, 1H), 1.48 (s, 3H), 0.98 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.3, 170.6, 143.8, 142.5, 128.6, 128.4, 127.8, 127.8, 125.5, 124.9, 87.0, 62.0, 51.7, 47.5, 21.5, 13.5; LRMS (EI, 70 eV) *m/z* (%): 324 (M^+, 49), 182 (46), 105 (100); HRMS *m/z* (ESI) calcd for $C_{20}H_{21}O_4$ ($[M+H]^+$) 325.1434, found 325.1441.

3-Methyl-5,5-diphenyldihydrofuran-2(3*H*)-one (3kj):[5]



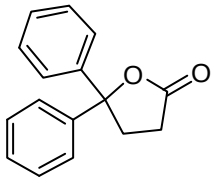
Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.44 (d, *J* = 8.0 Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 7.34-7.29 (m, 4H), 7.26-7.24 (m, 2H), 3.24-3.19 (m, 1H), 2.68-2.61 (m, 1H), 2.48-2.42 (m, 1H), 1.26 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 178.5, 143.8, 142.5, 128.5, 128.4, 127.7, 127.7, 125.2, 125.2, 87.1, 43.8, 35.0, 14.5; LRMS (EI, 70 eV) *m/z* (%): 252 (M^+, 20), 183 (67), 105 (100); HRMS *m/z* (ESI) calcd for $C_{17}H_{17}O_2$ ($[M+H]^+$) 253.1223, found 263.1233.

3-Butyl-5,5-diphenyldihydrofuran-2(3*H*)-one (3kk):



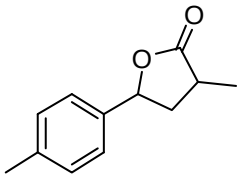
Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.44 (d, *J* = 7.6 Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 7.36-7.30 (m, 4H), 7.28-7.23 (m, 2H), 3.21-3.16 (m, 1H), 2.62-2.53 (m,1H), 2.46 (t, *J* = 12.0 Hz, 1H), 1.95-1.87 (m, 1H), 1.50-1.41 (m,1H), 1.36-1.26 (m, 4H), 0.89 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 178.1, 144.0, 142.7, 128.6, 128.5, 127.8, 127.7, 125.3, 125.2, 87.33, 41.9, 40.2, 29.6, 29.5, 22.4, 13.8; LRMS (EI, 70 eV) *m/z* (%): 294 (M^+, 15), 183 (65), 105 (100); HRMS *m/z* (ESI) calcd for $C_{20}H_{23}O_2$ ($[M+H]^+$) 295.1693, found 295.1685.

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



White solid, mp 76.3-78.4 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.42 (d, *J* = 8.0 Hz, 4H), 7.34 (t, *J* = 7.4 Hz, 4H), 7.29-7.26 (m, 2H), 2.91 (t, *J* = 7.8 Hz, 2H), 2.58 (t, *J* = 7.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 176.1, 145.0, 143.0, 128.6, 128.4, 127.9, 127.6, 125.6, 125.3, 89.7, 35.6, 29.0; LRMS (EI, 70 eV) *m/z* (%): 238 (M^+, 56), 183 (100), 105 (84); HRMS *m/z* (ESI) calcd for $C_{16}H_{15}O_2$ ($[M+H]^+$) 239.1067, found 239.1074.

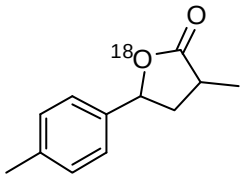
3-Methyl-5-*p*-tolyldihydrofuran-2(3*H*)-one (3aj): d.r. =1.2:1



Colourless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.23-7.12 (m, 4H), 5.56-5.53 (m, 1H), 2.79-2.67 (m, 1H), 2.79-2.69 (m, 1H), 2.36-2.29 (m, 4H), 1.33 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 179.9, 138.0, 136.7, 129.4, 125.0, 78.4, 38.4, 33.7, 21.1, 15.4; LRMS (EI, 70 eV) *m/z* (%): 190 (M^+, 56), 175 (21), 131 (80), 119 (100).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.26-7.18 (m, 4H), 5.34-5.30 (m, 1H), 2.85-2.73 (m, 2H), 2.36 (s, 3H), 1.89-1.80 (m, 1H), 1.33 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 179.2, 138.4, 136.0, 129.3, 125.5, 79.3, 39.9, 36.4, 21.1, 15.0; LRMS (EI, 70 eV) *m/z* (%): 190 (M^+, 81), 175 (33), 131 (98), 119 (100).

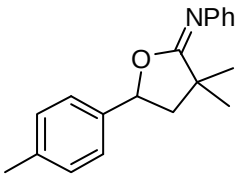
3-Methyl-5-*p*-tolyldihydrofuran-2(3*H*)-one (3aj-18O):



d.r. =1.2:1

Colourless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.24-7.17 (m, 4H), 5.56-5.53 (m, 1H), 2.79-2.69 (m, 1H), 2.47-2.40 (m, 1H), 2.35-2.32 (m, 4H), 1.33 (d, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 179.96, 179.93, 138.01, 136.71, 129.37, 124.98, 78.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.26-7.18 (m, 4H), 5.32 (m, 1H), 2.85-2.73 (m, 2H), 2.36 (s, 3H), 1.89-1.80 (m, 1H), 1.33 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 179.23, 179.19, 138.34, 135.96, 129.33, 125.54, 79.25, 39.92, 36.40, 21.13, 14.95.



***N*-(3,3-Dimethyl-5-*p*-tolyldihydrofuran-2(3*H*)-ylidene)aniline (3am):**

White solid. mp: 87.2-88.4 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.28-7.25 (m, 2H), 7.19-7.11 (m, 6H), 7.02 (t, J = 7.2 Hz, 1H), 5.39-5.35 (m, 1H), 2.42-2.34 (m, 4H), 2.02 (t, J = 12Hz, 1H), 1.44 (s, 3H), 1.41 (s, 3H); ^{13}C NMR (400 MHz, $CDCl_3$) δ: 168.1, 147.2, 137.9, 137.2, 129.2, 128.5, 125.5, 123.4, 122.7, 80.0, 47.2, 41.8, 29.7, 26.3, 26.2, 21.1; LRMS (EI, 70 eV) *m/z* (%): 279 (M^+, 60), 264 (25), 159 (48), 145 (100); HRMS *m/z* (ESI) calcd for $C_{19}H_{22}NO$ ($[M+H]^+$) 280.1696, found 280.1703.

(C) References

[1] S. Sathyamoorthi and J. D. Bois, *Org. Lett.,* 2016, **18**, 6308.

[2] T. Sakai, A. Yamawaki, T. Katayama, H. Okada, M. Utaka and A. Takeda, *B. Chem. Soc. Jpn.*, 1987, **60**, 1067.

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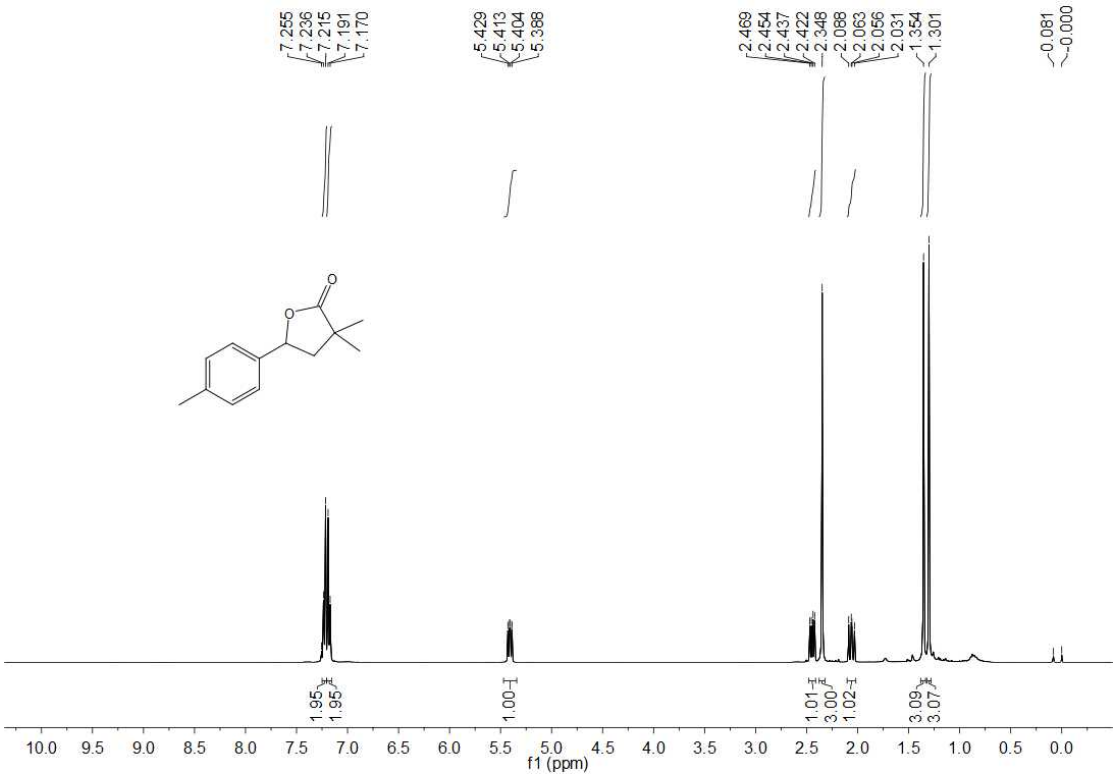
[4] I. M. Pastor and M. Yus, *Tetrahedron Lett.*, 2000, **41**, 5335.

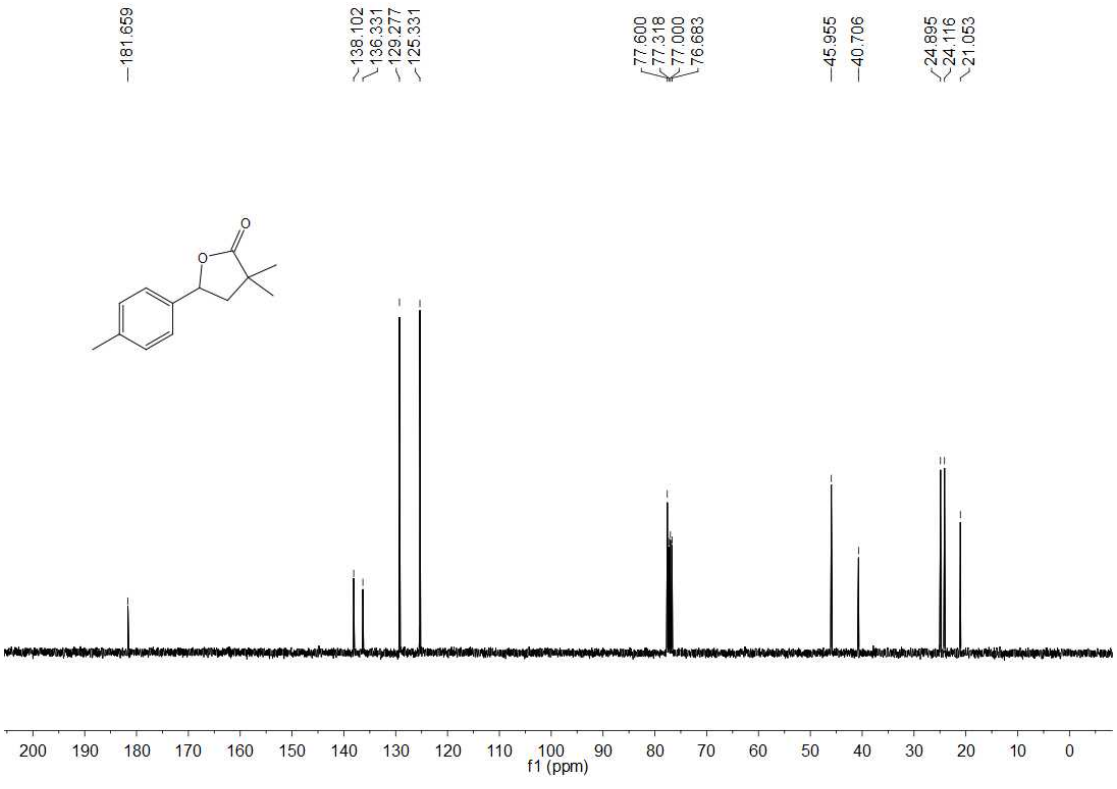
[5] X.-J. Wei, D.-T. Yang, L. Wang, T. Song, L.-Z. Wu and Q. Liu, *Org. Lett.*, 2013, **15**, 23.

[6] G. E. M. Moussa, *Egypt. J. Chem.*, 1988, **30**, 63.

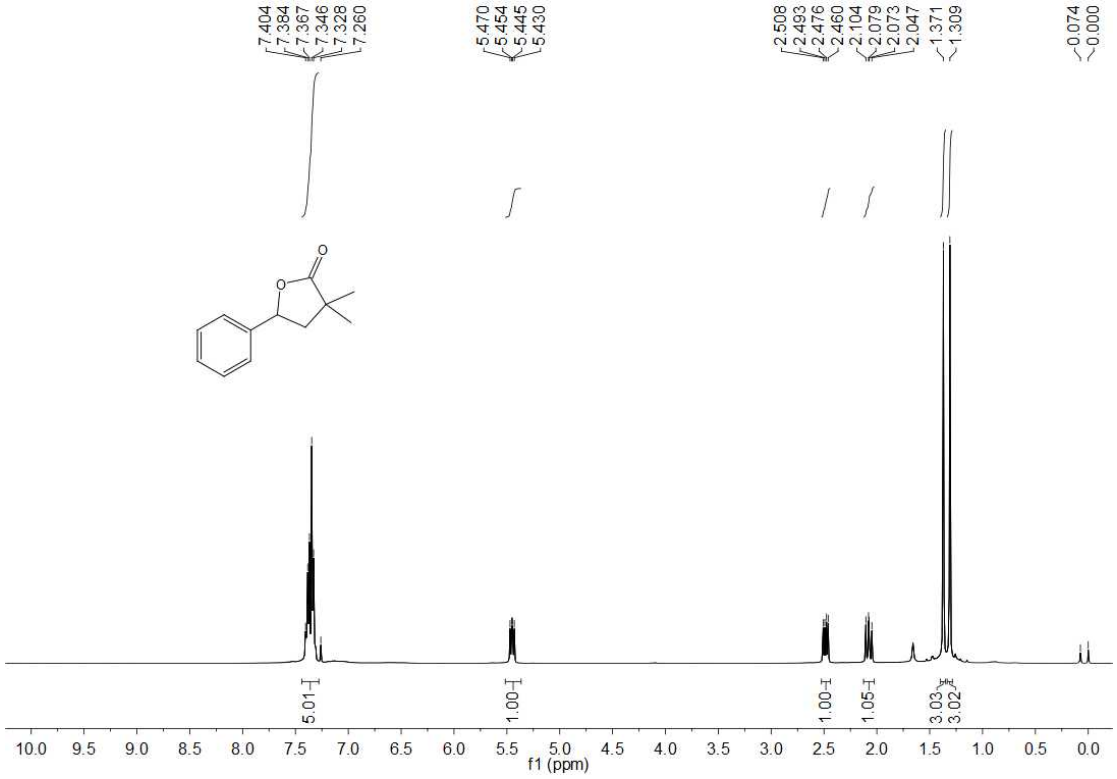
(D) Spectra

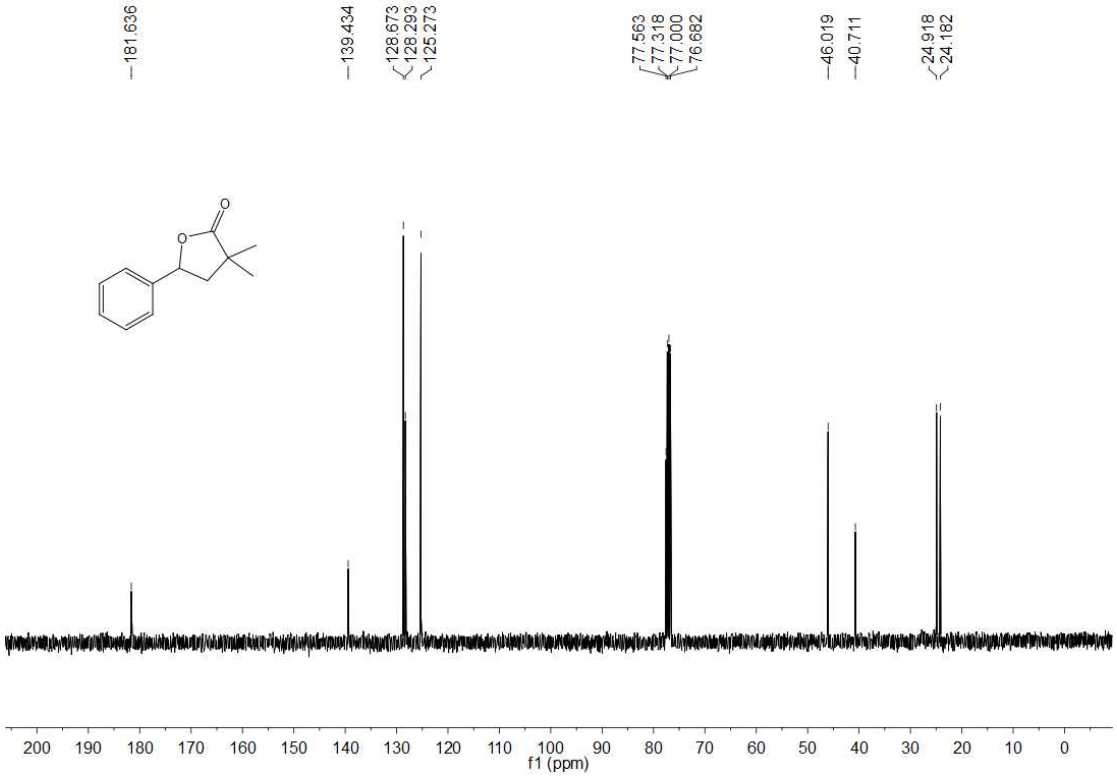
3,3-Dimethyl-5-(*p*-tolyl)dihydrofuran-2(3*H*)-one (3aa)



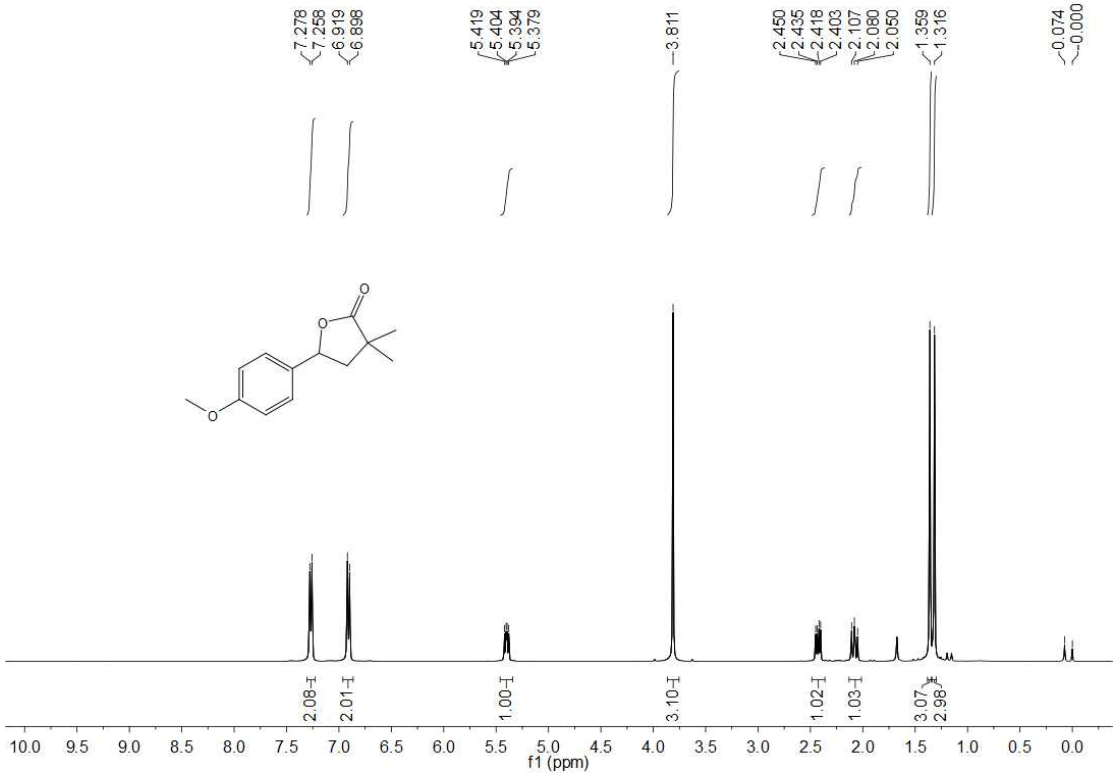


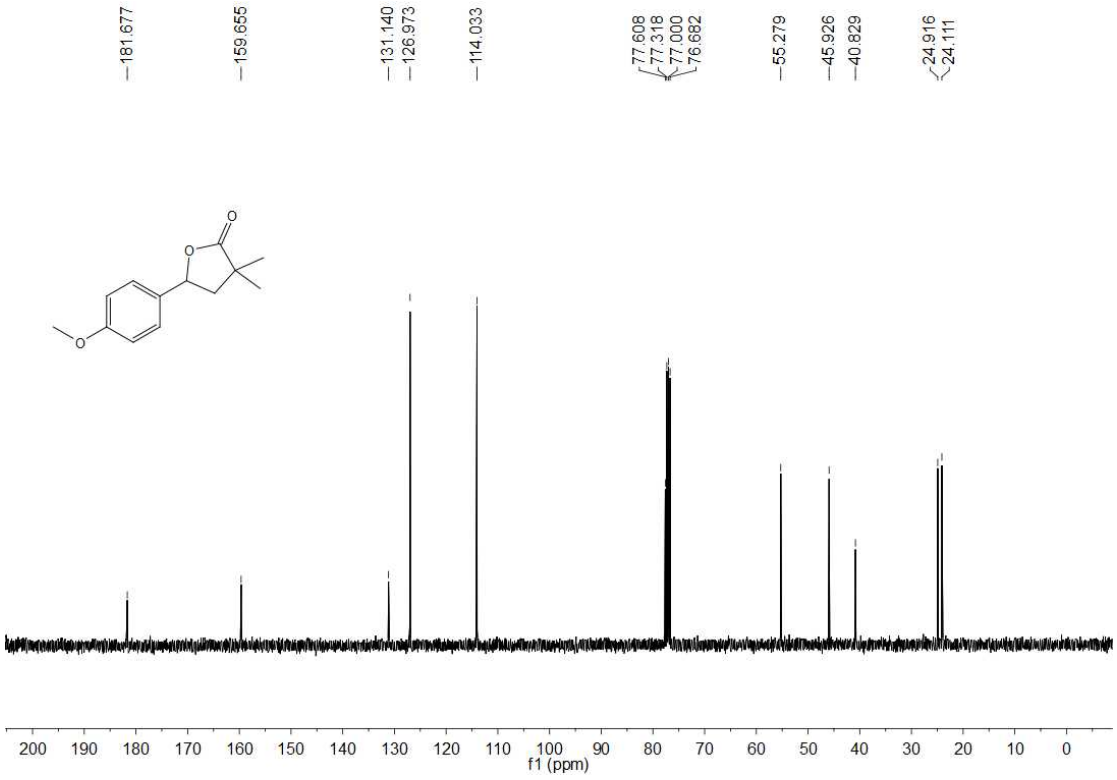
3,3-Dimethyl-5-phenyldihydrofuran-2(3*H*)-one (3ba)



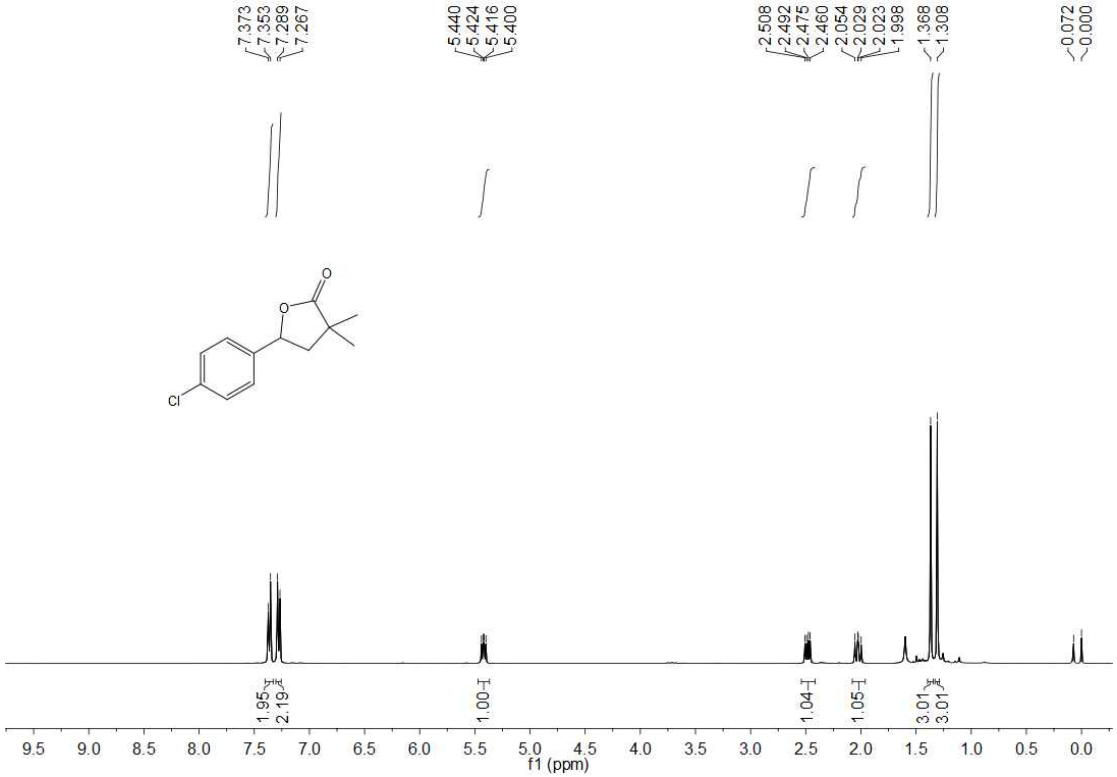


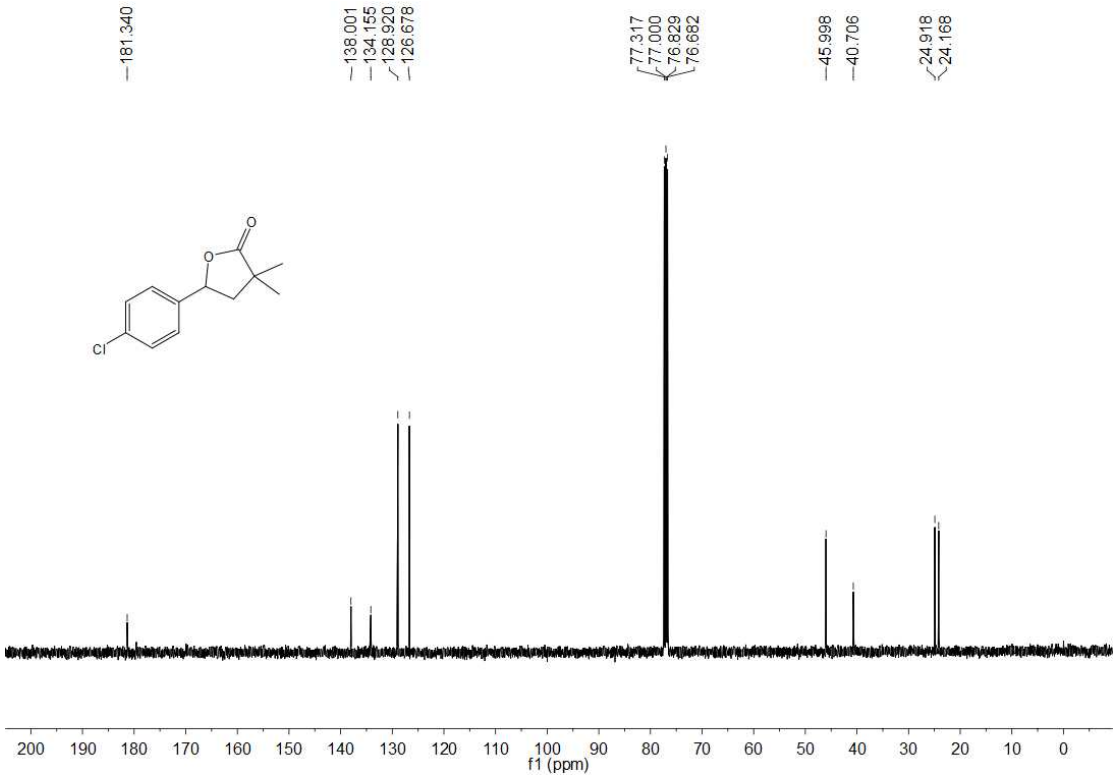
5-(4-Methoxyphenyl)-3,3-dimethyldihydrofuran-2(3*H*)-one (3ca)



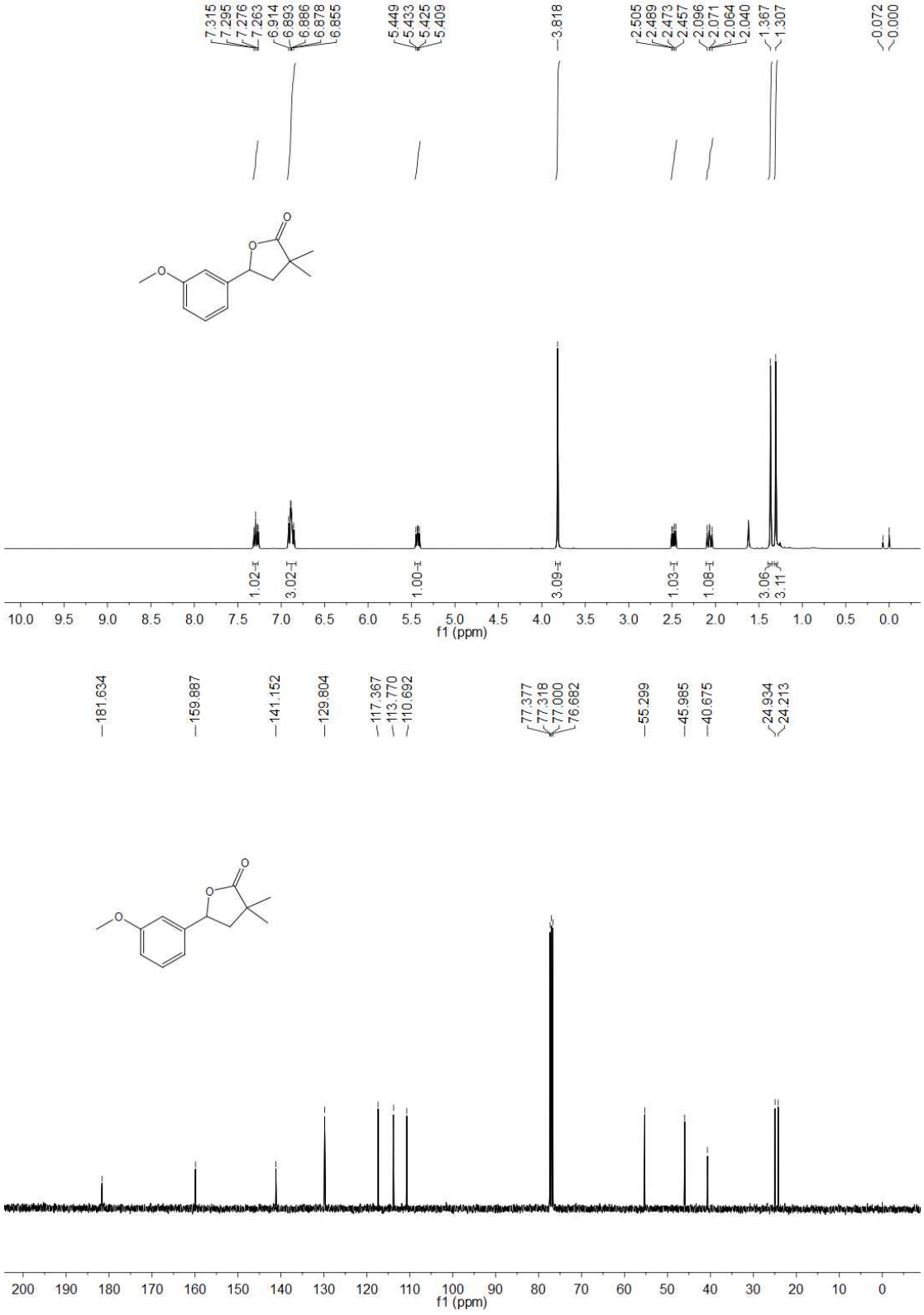


5-(4-Chlorophenyl)-3,3-dimethyldihydrofuran-2(3*H*)-one (3da)

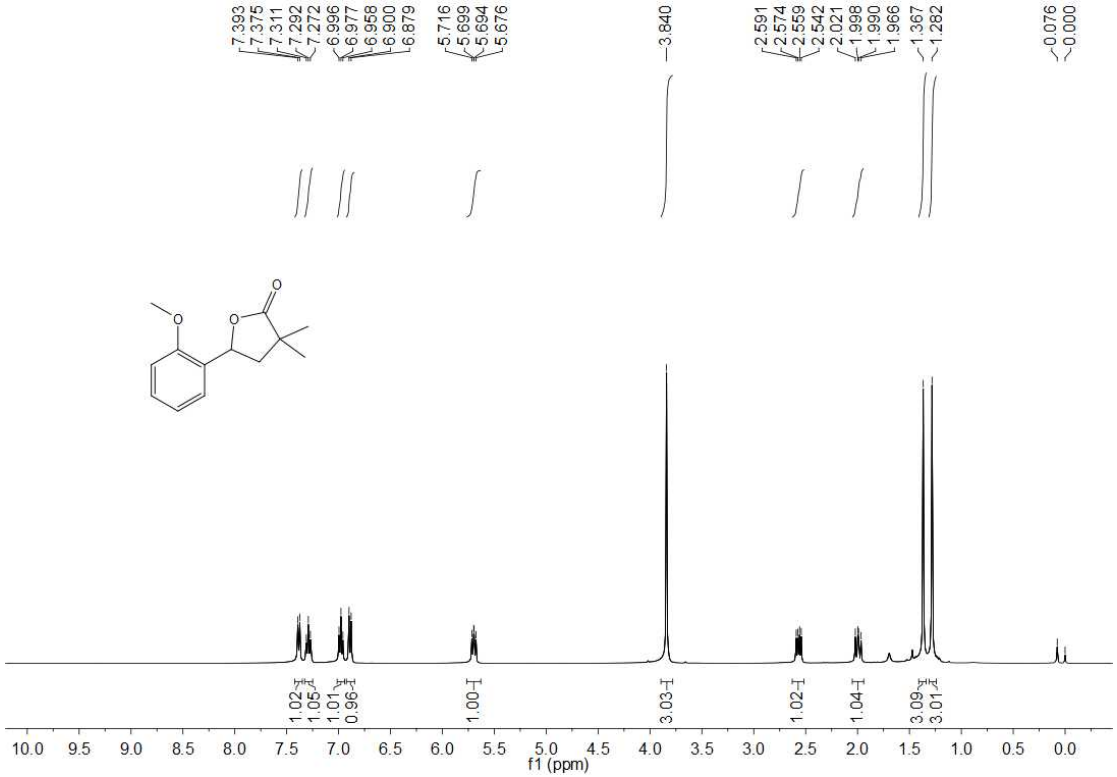


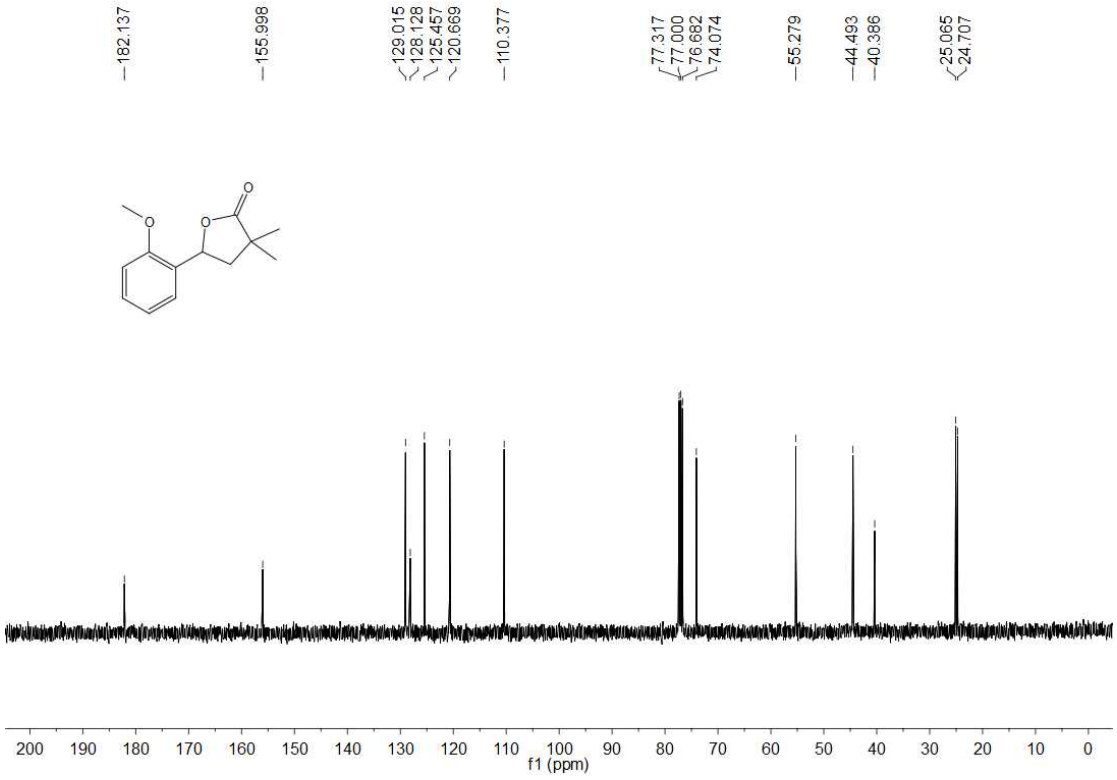


5-(3-Methoxyphenyl)-3,3-dimethyldihydrofuran-2(3*H*)-one (3ea)

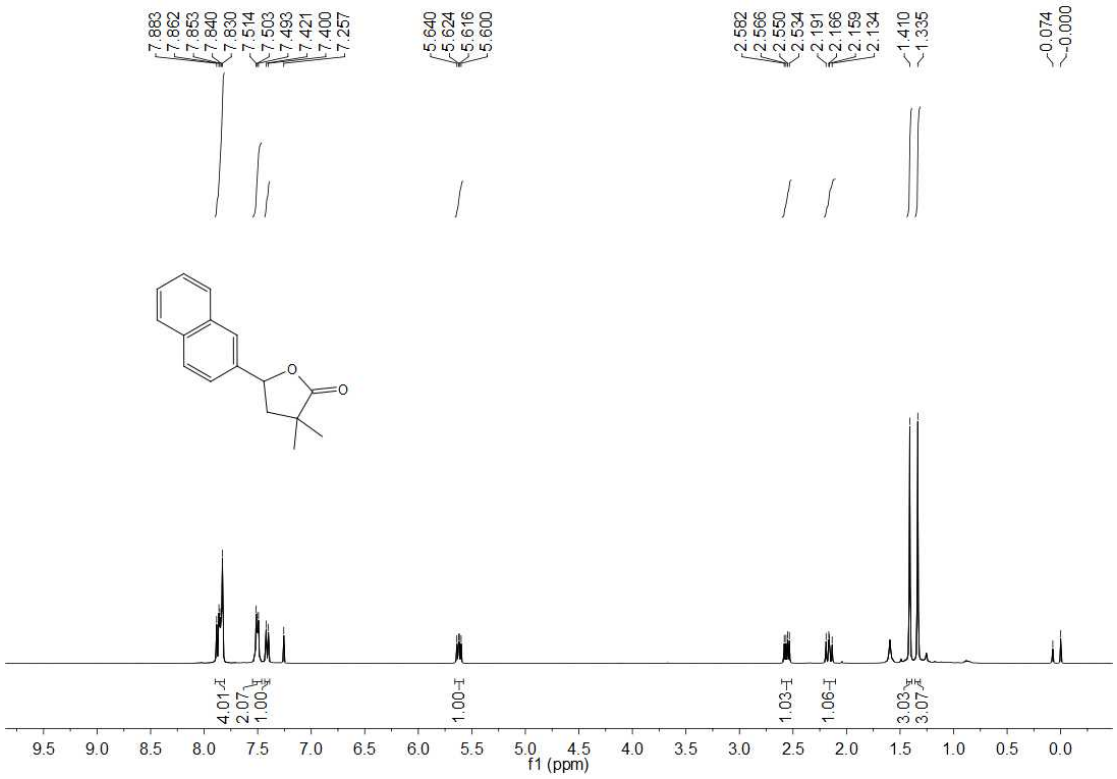


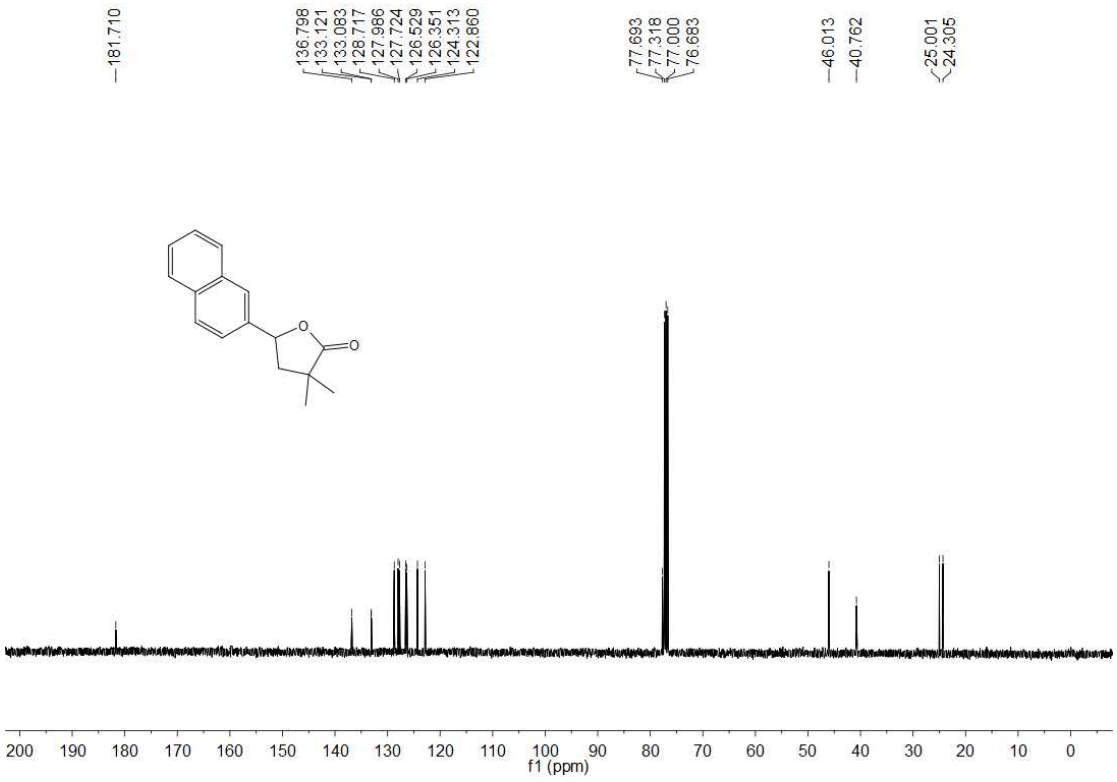
5-(2-Methoxyphenyl)-3,3-dimethyldihydrofuran-2(3*H*)-one (3fa)



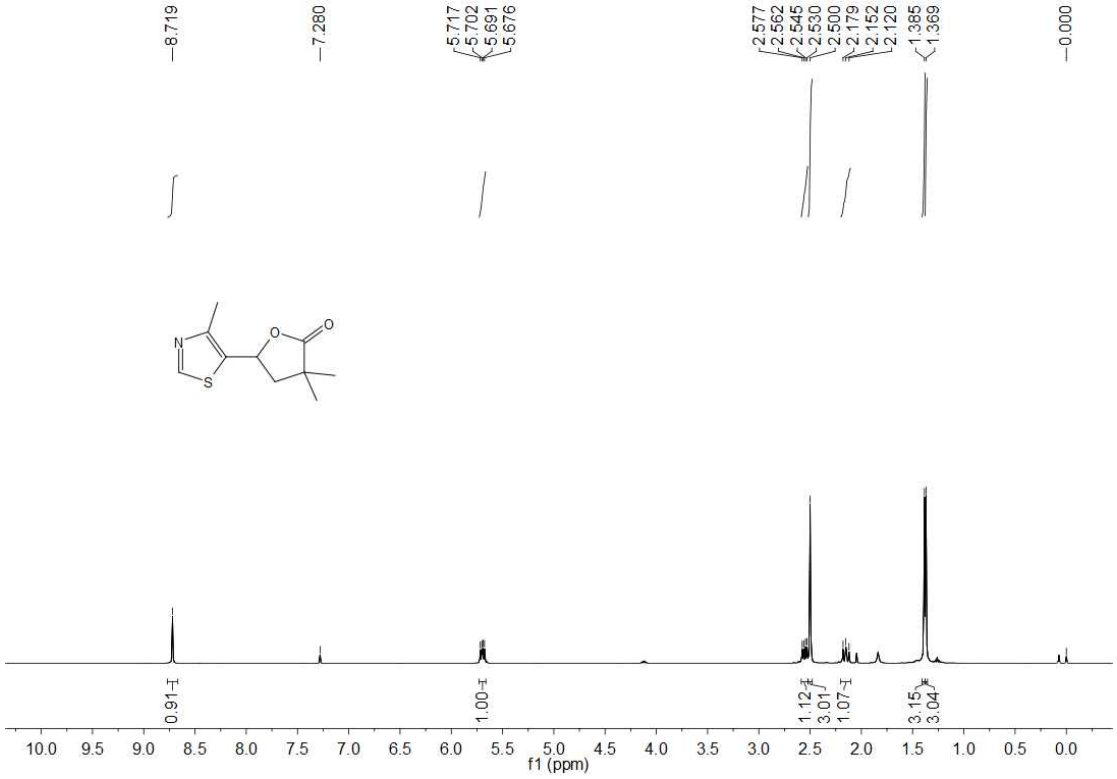


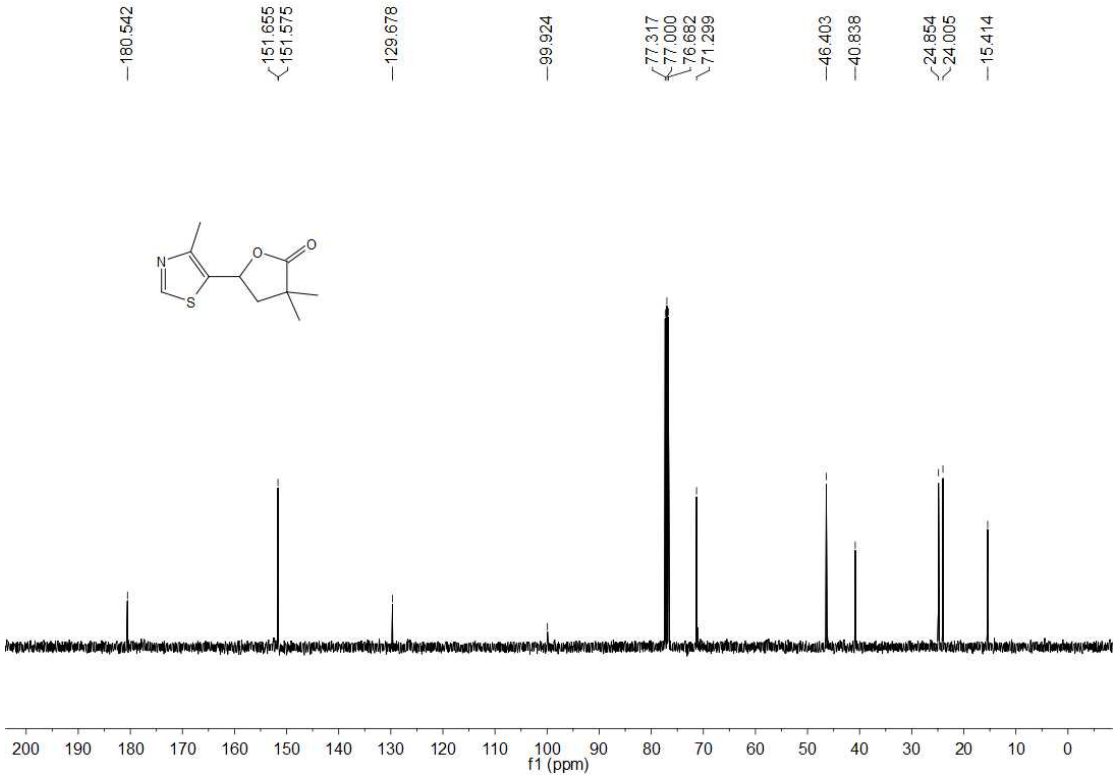
3,3-Dimethyl-5-(naphthalen-2-yl)dihydrofuran-2(3*H*)-one (3ga)



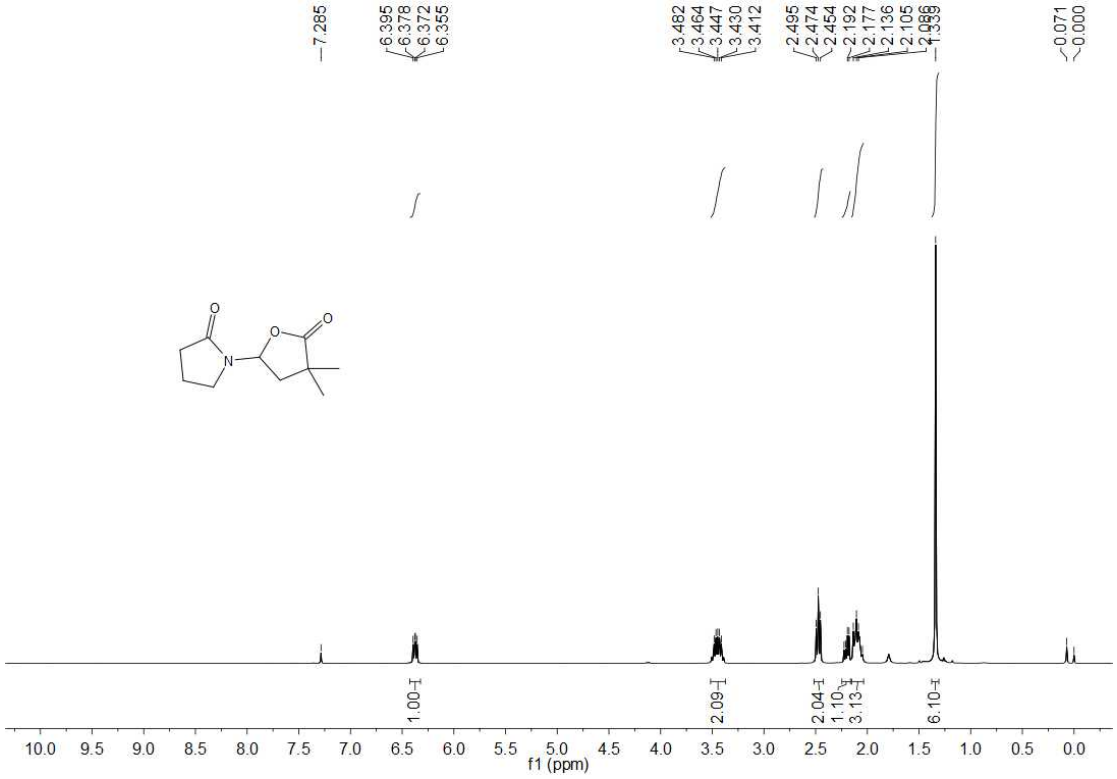


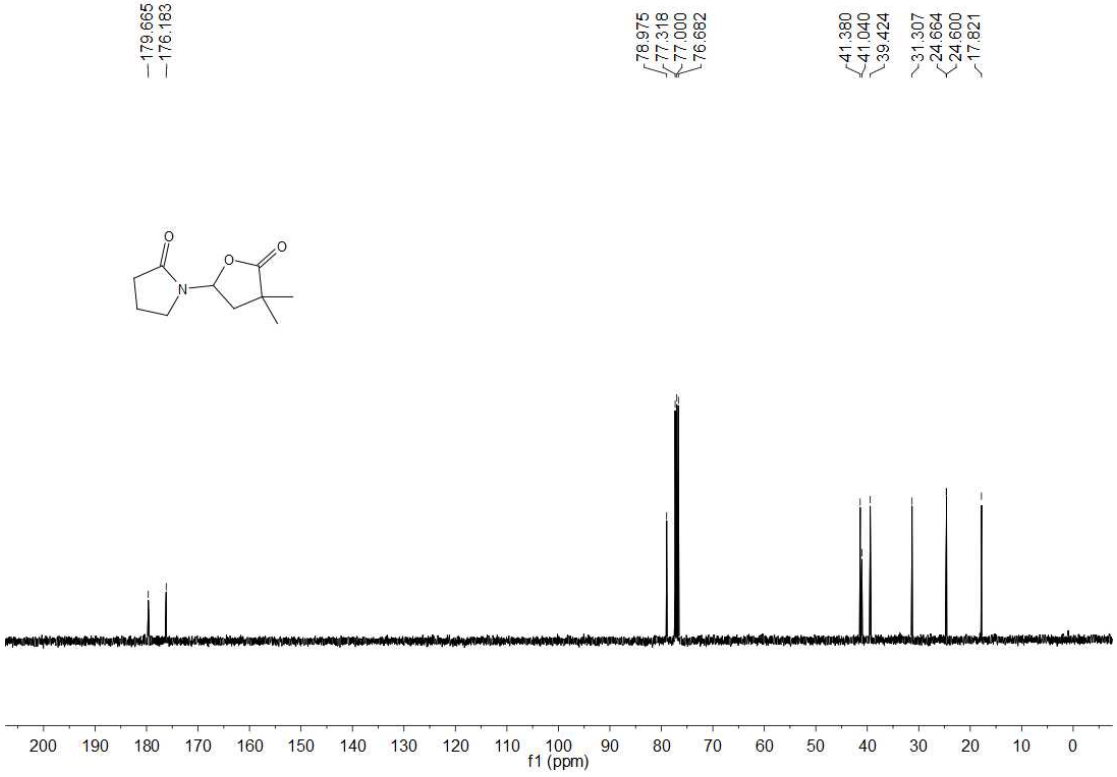
3,3-Dimethyl-5-(4-methylthiazol-5-yl)dihydrofuran-2(3*H*)-one (3ha)



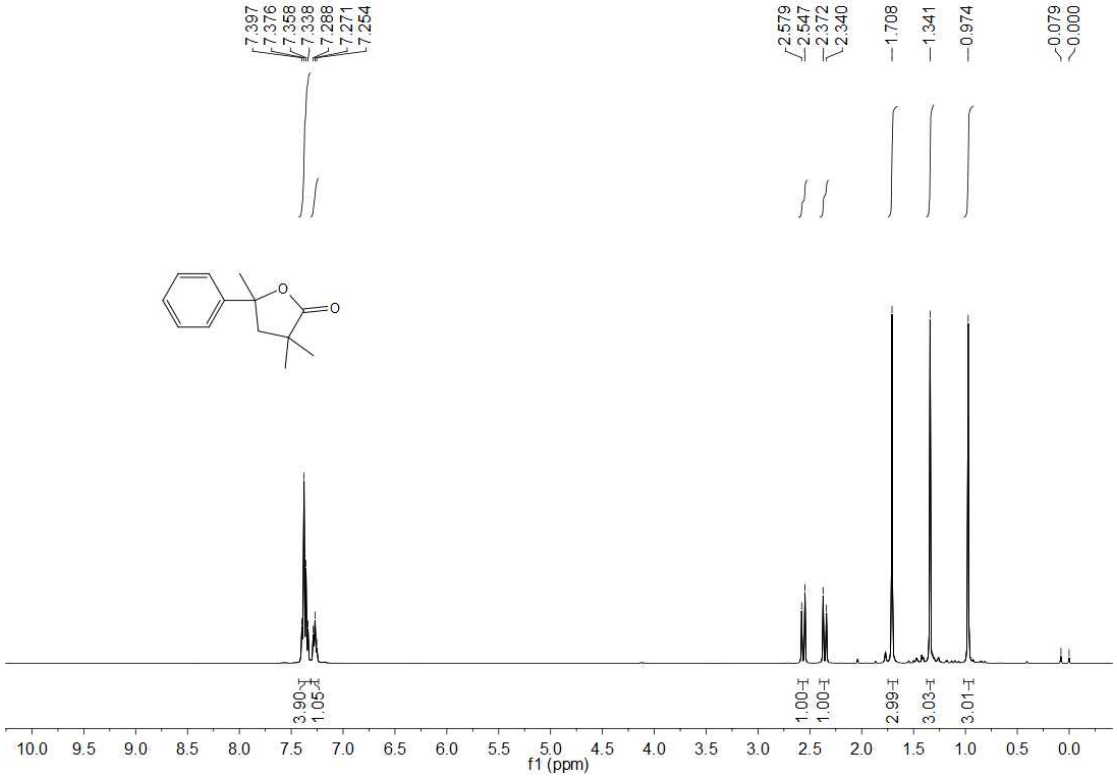


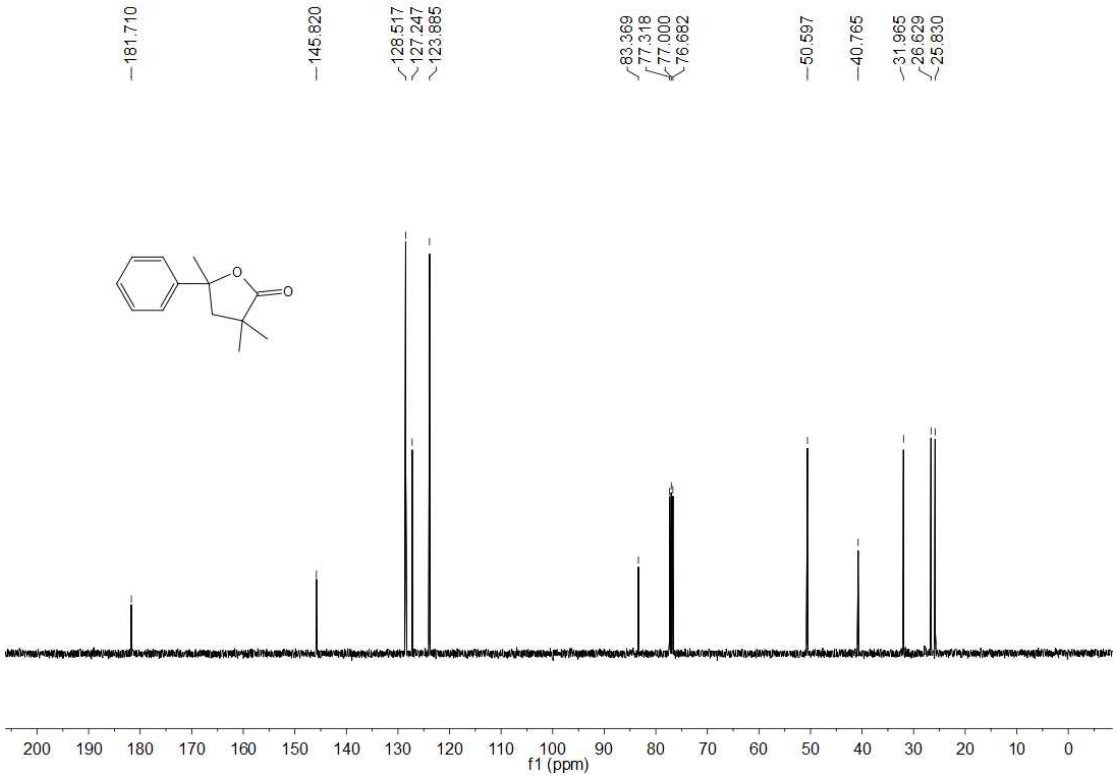
1-(4,4-Dimethyl-5-oxotetrahydrofuran-2-yl)pyrrolidin-2-one (3ia)



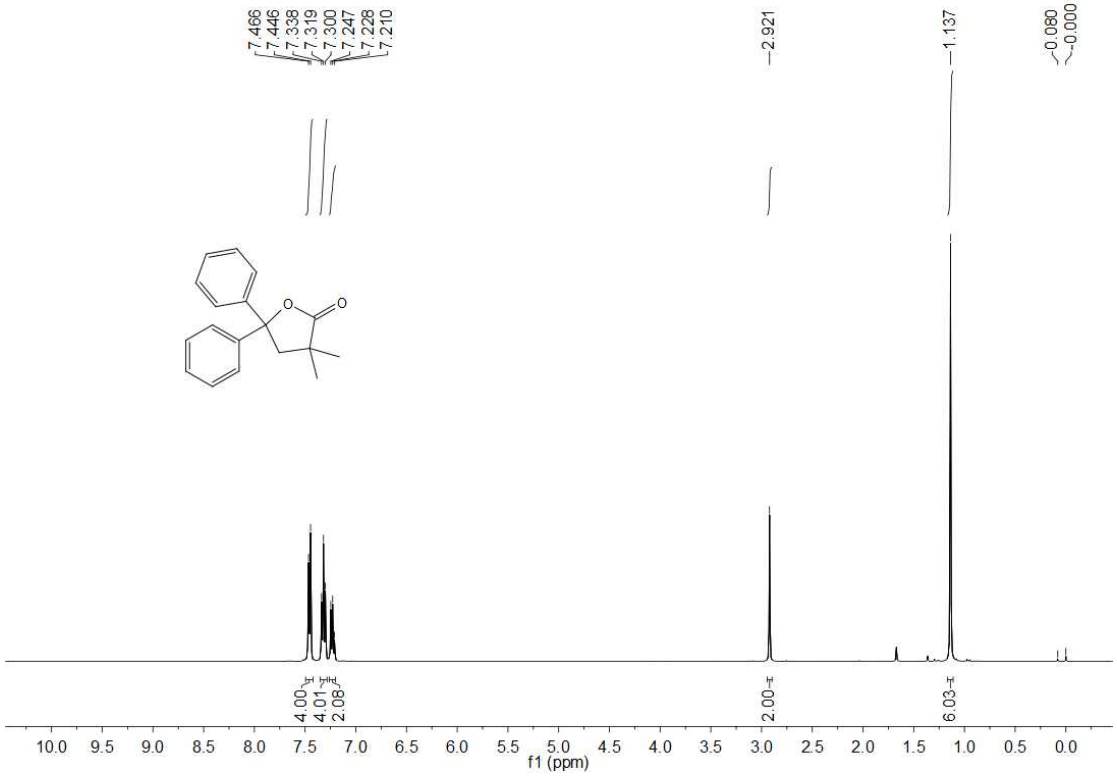


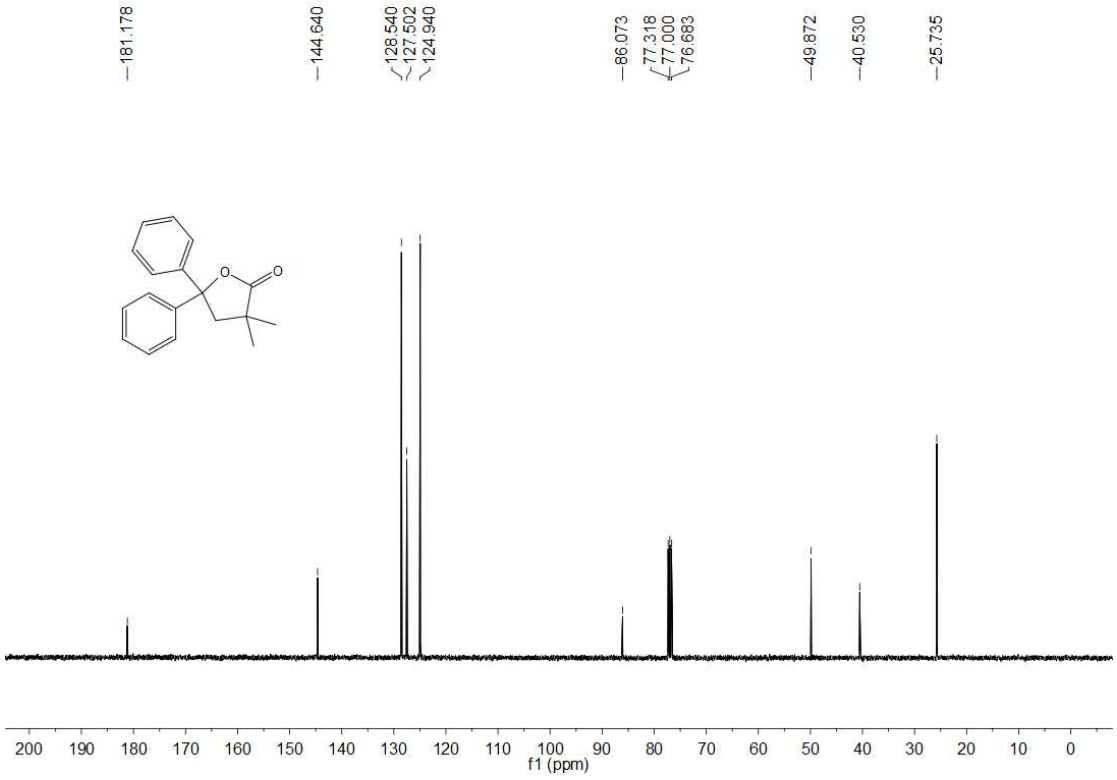
3,3,5-Trimethyl-5-phenyldihydrofuran-2(3*H*)-one (3ja)



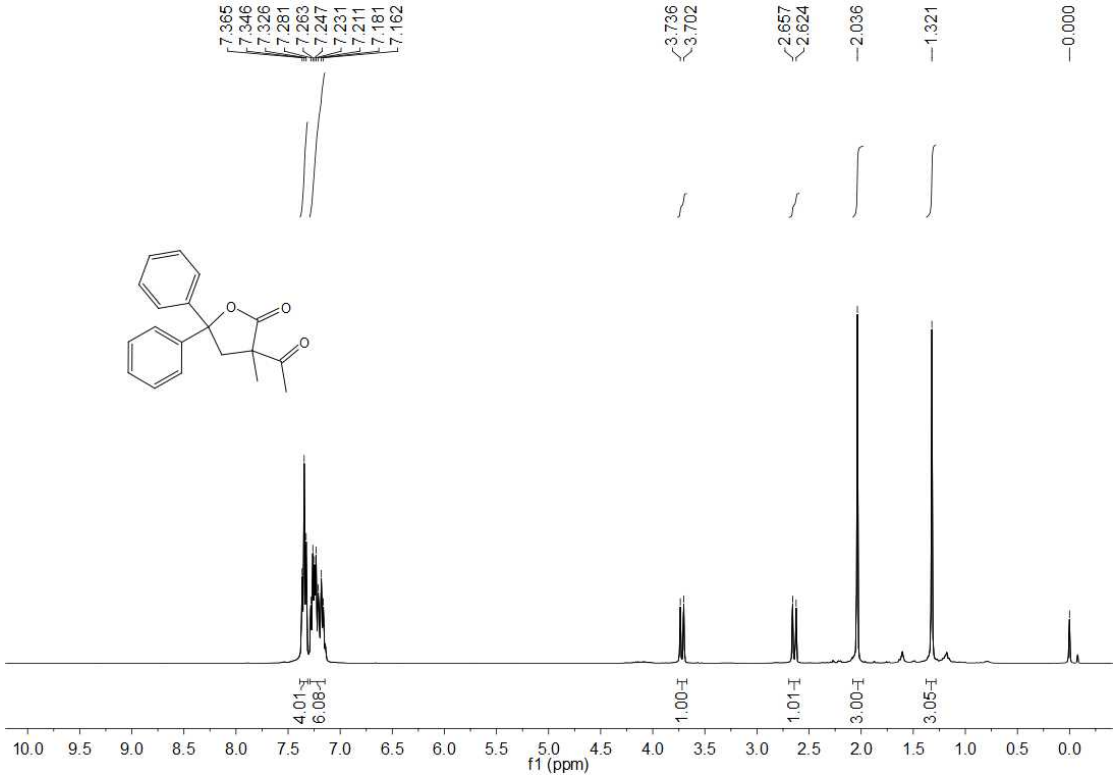


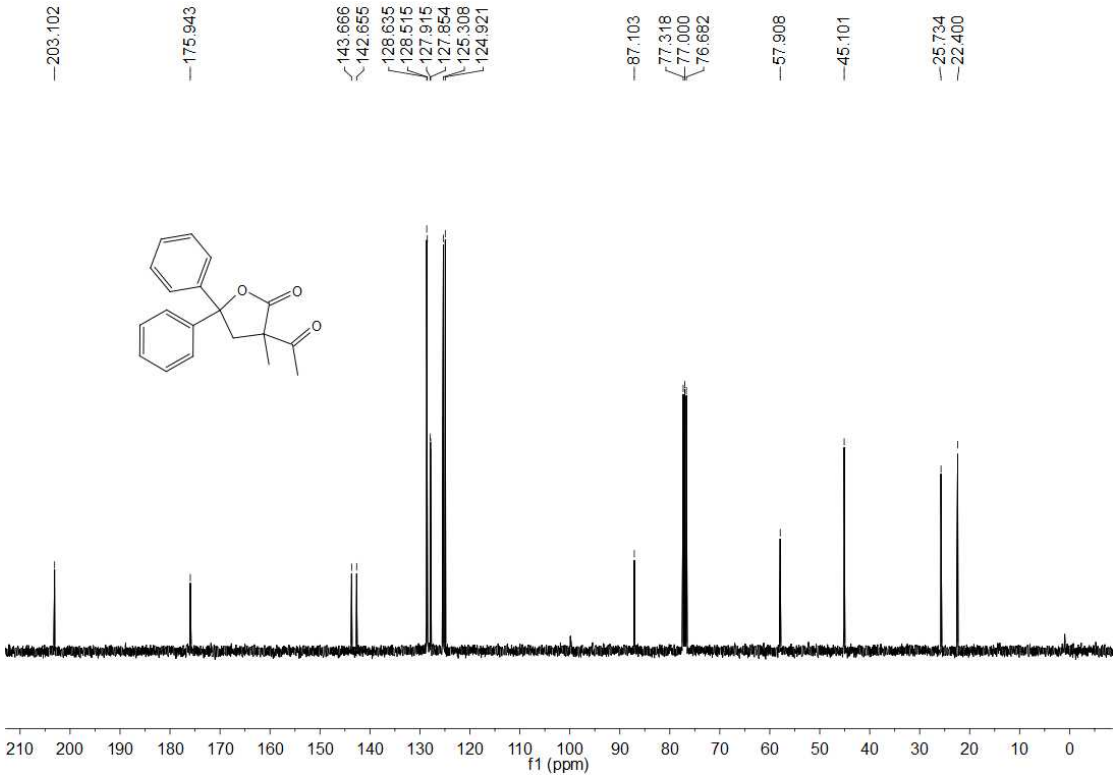
3,3-Dimethyl-5,5-diphenyldihydrofuran-2(3*H*)-one (3ka)



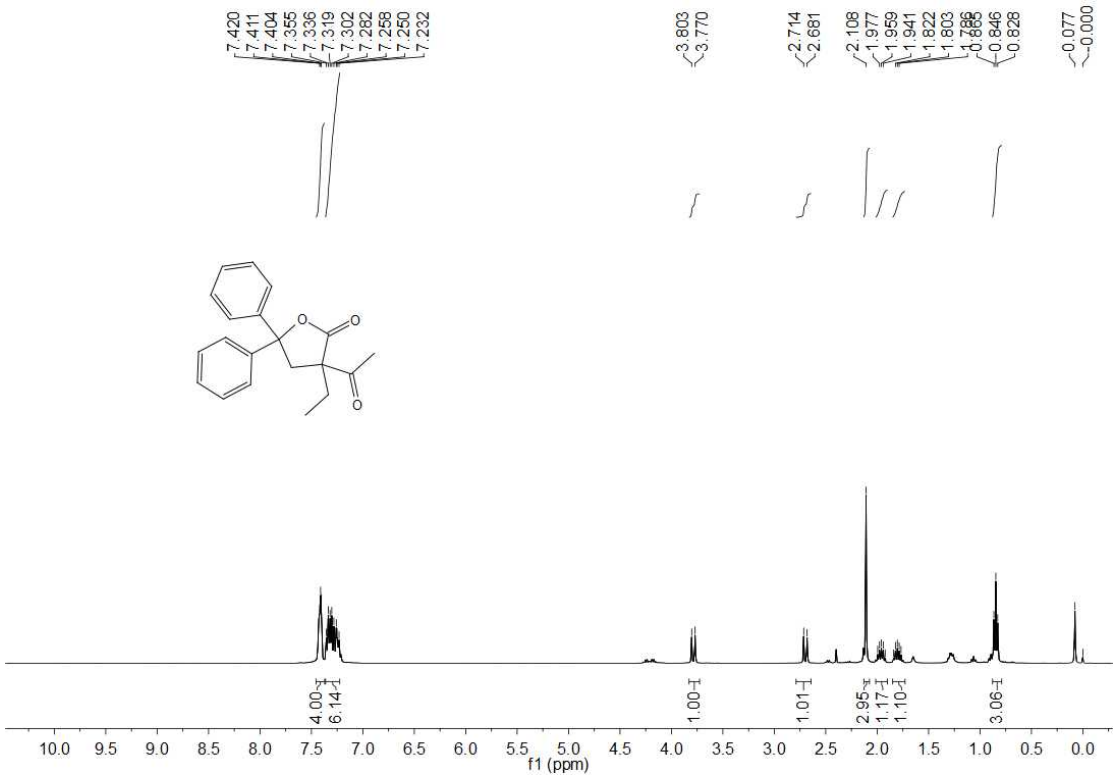


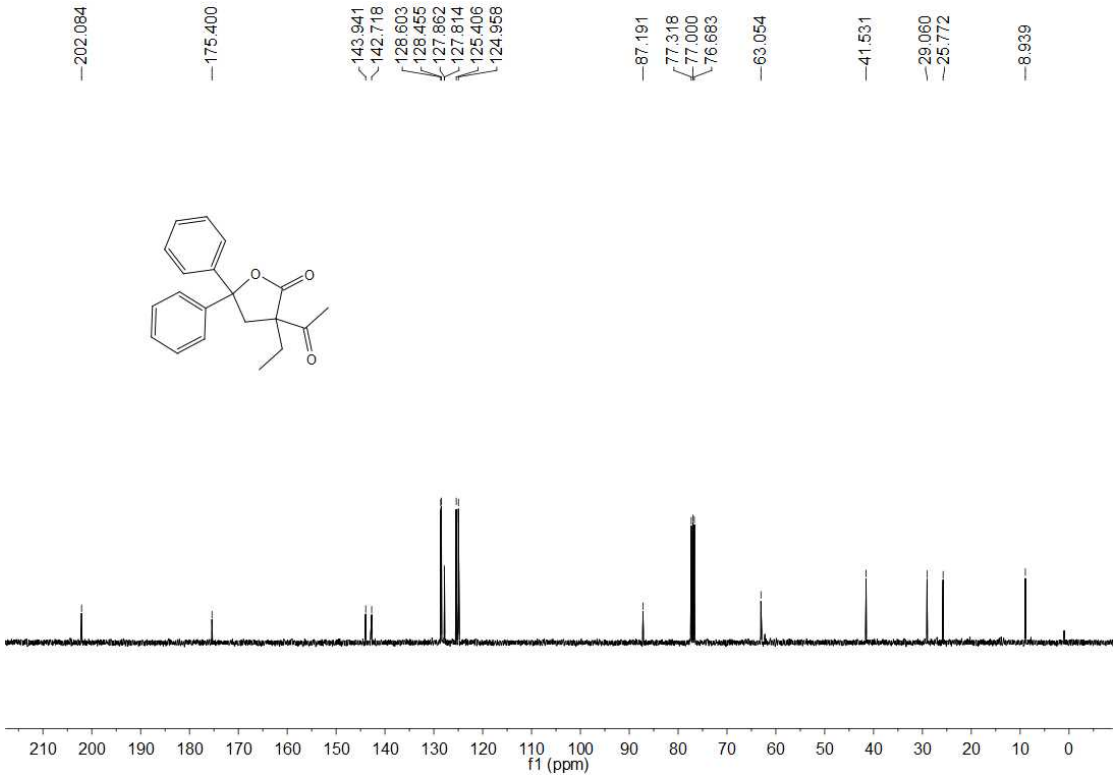
3-Acetyl-3-methyl-5,5-diphenyldihydrofuran-2(3*H*)-one (3kg)



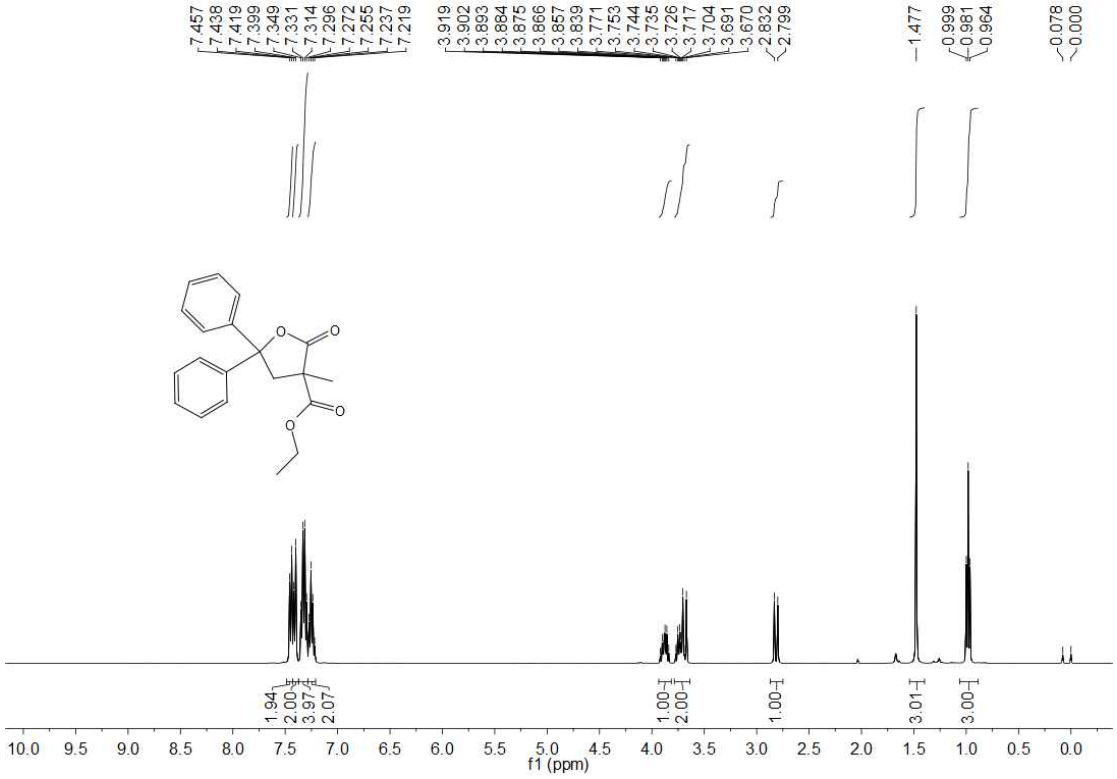


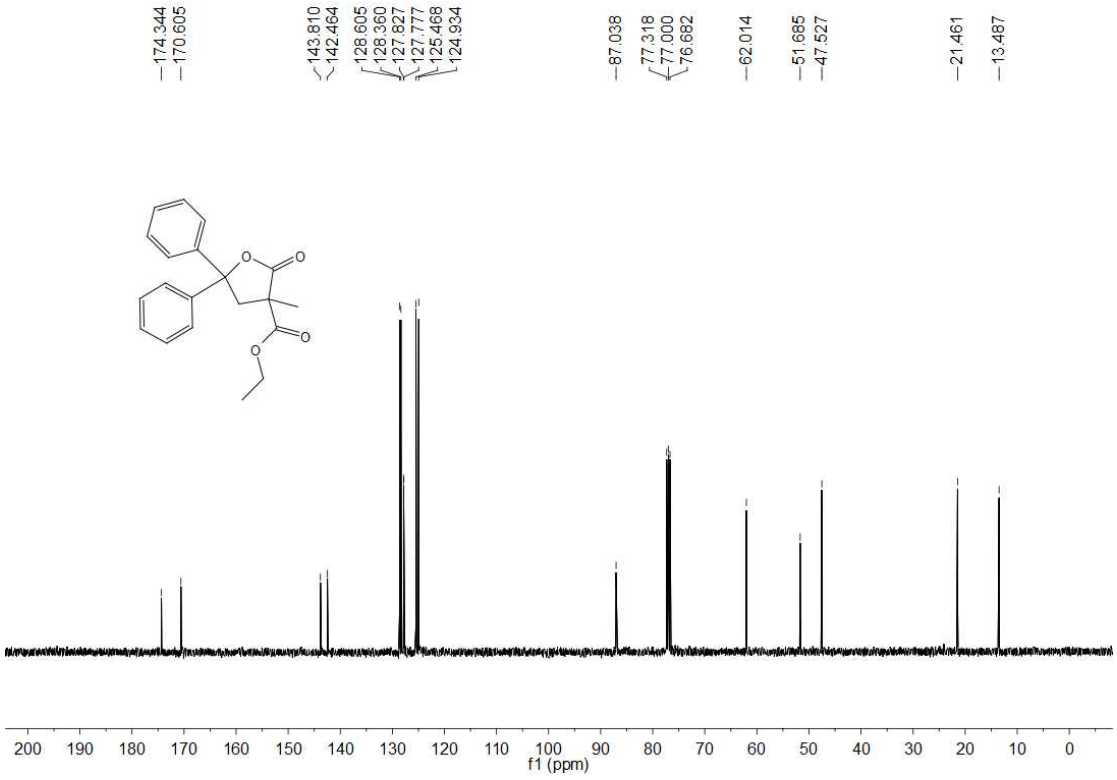
3-Acetyl-3-ethyl-5,5-diphenyldihydrofuran-2(3*H*)-one (3kh)



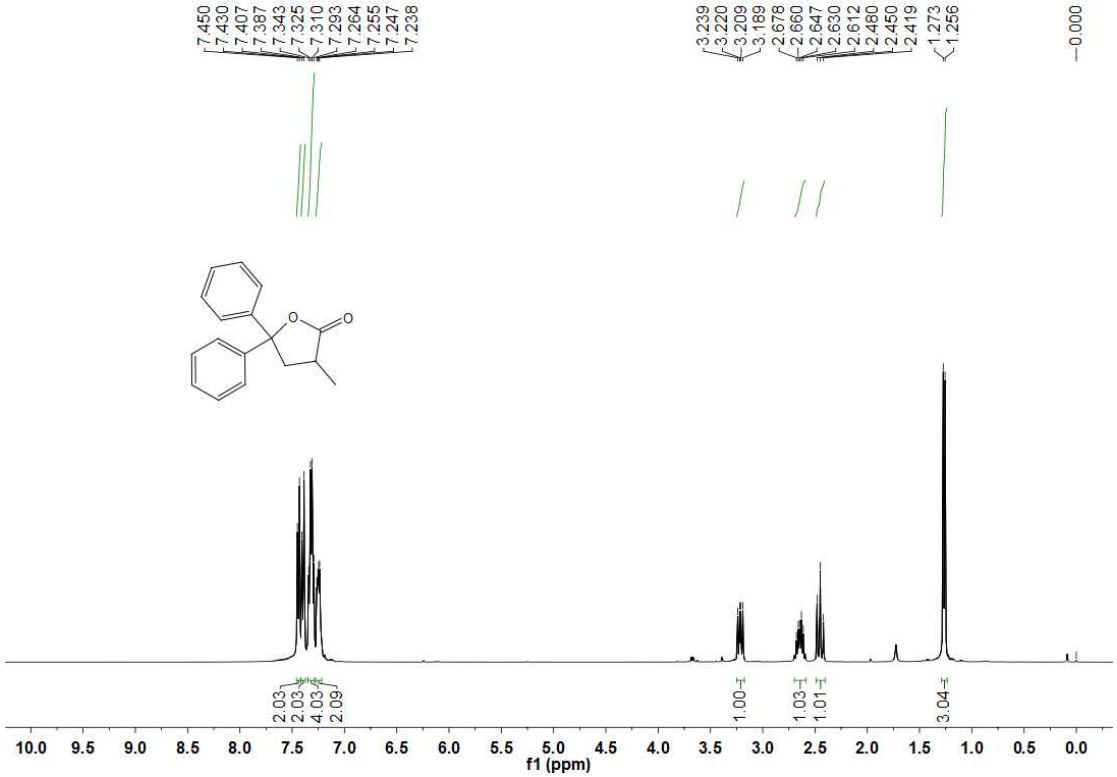


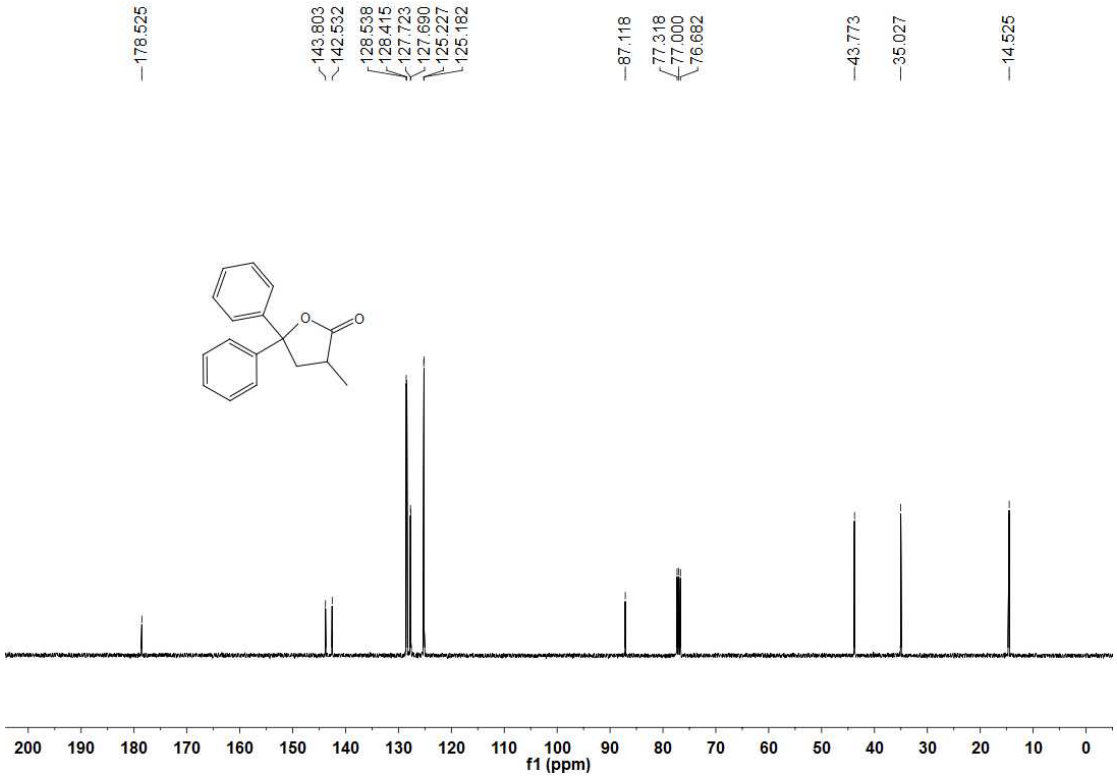
Ethyl 3-methyl-2-oxo-5,5-diphenyltetrahydrofuran-3-carboxylate (3ki)



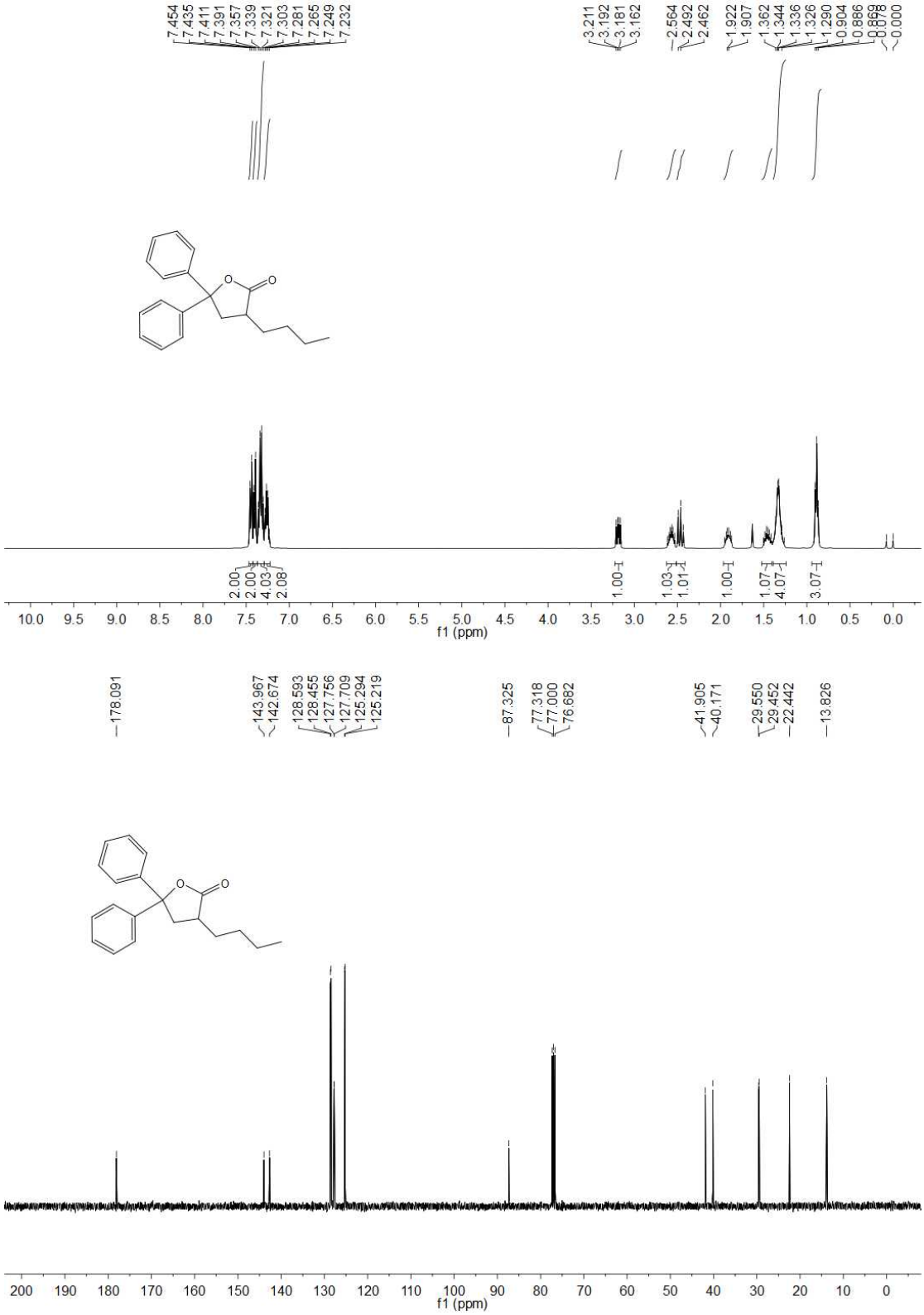


3-Methyl-5,5-diphenyldihydrofuran-2(3*H*)-one (3kj)

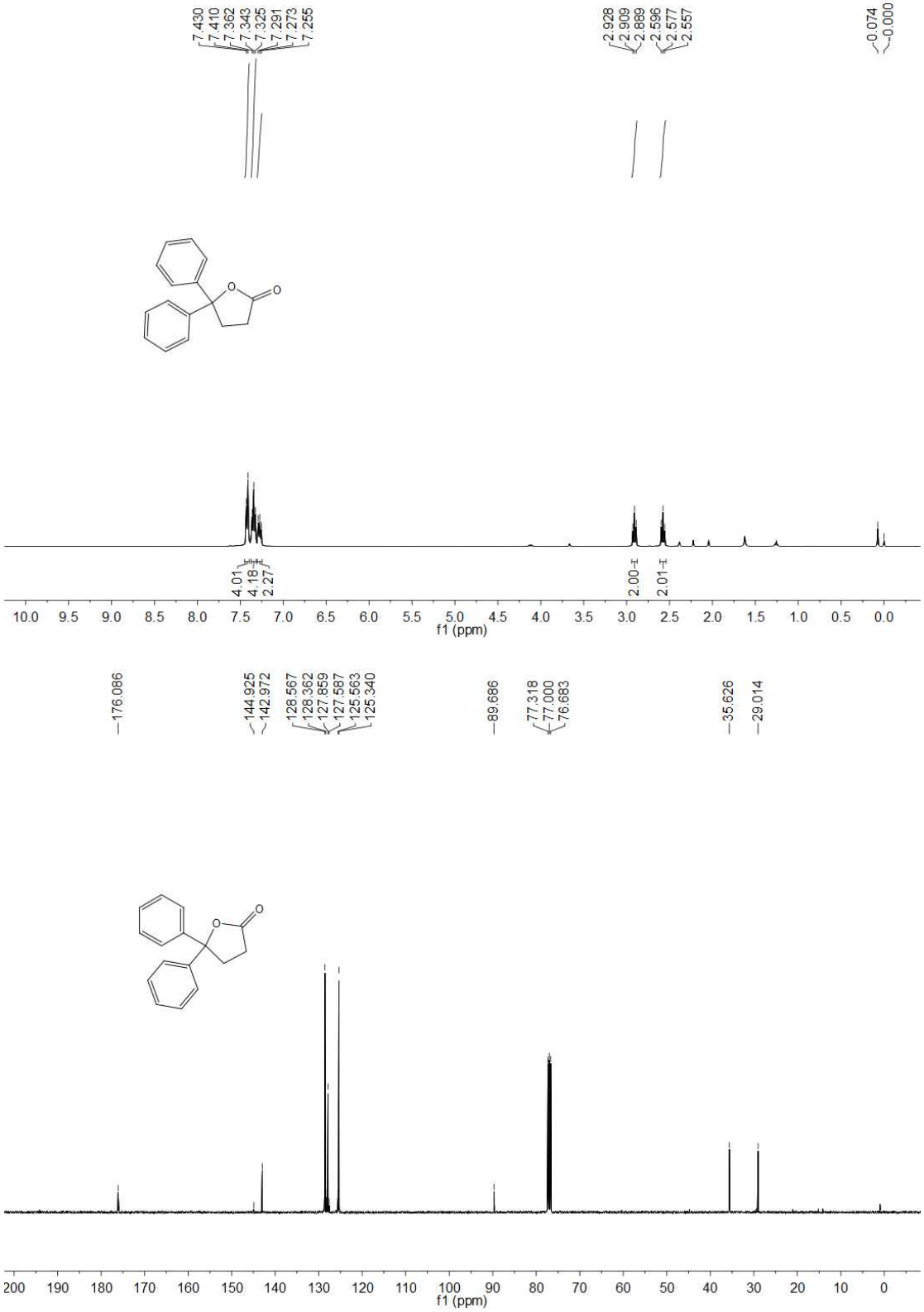




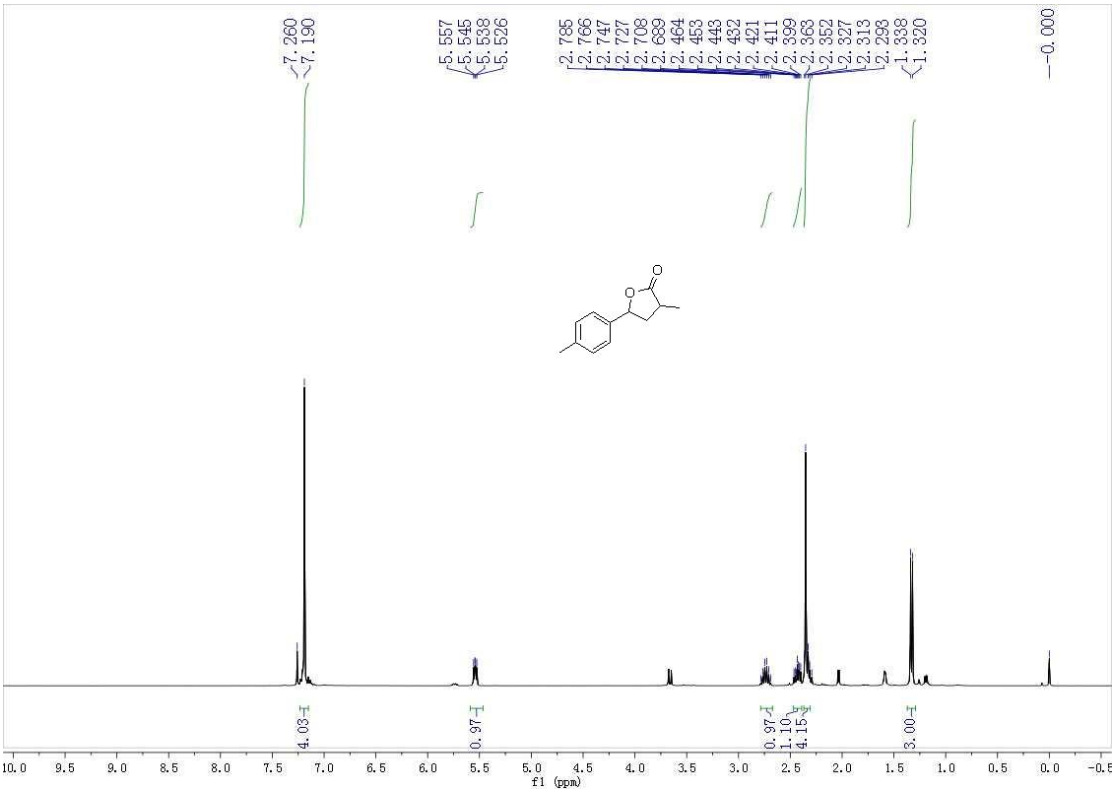
3-Butyl-5,5-diphenyldihydrofuran-2(3*H*)-one (3kk)

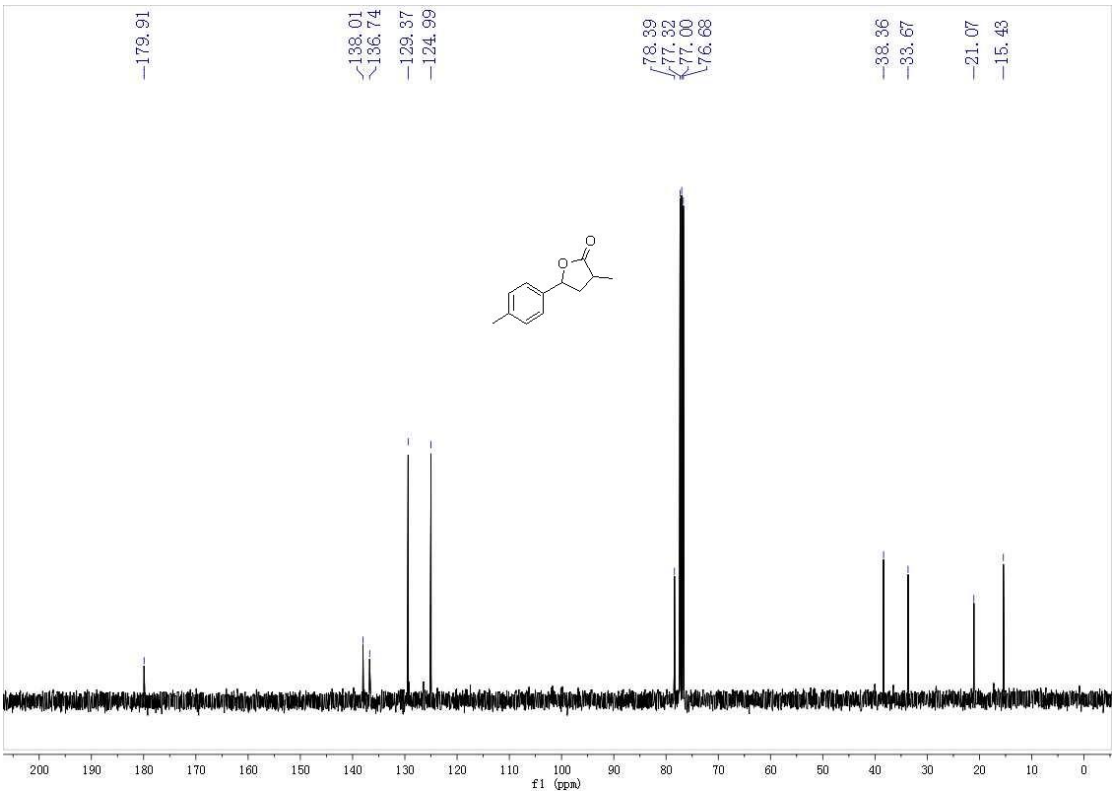


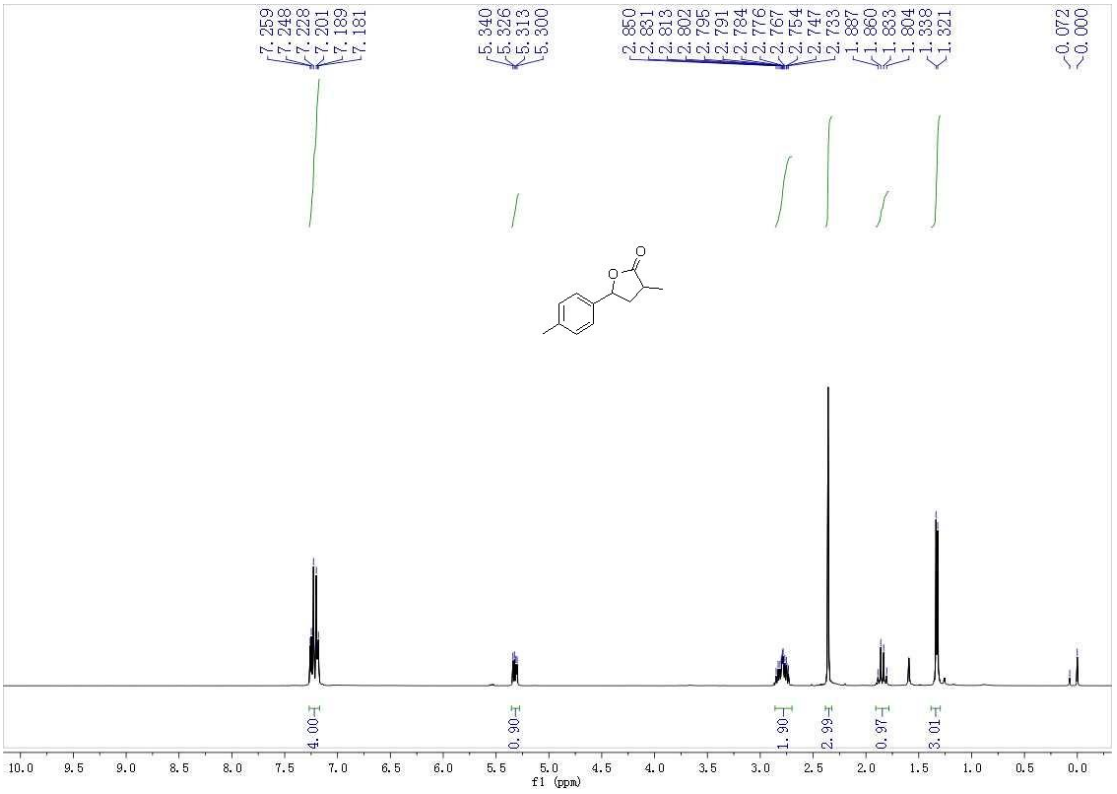
5,5-Diphenyldihydrofuran-2(3*H*)-one (3kl)

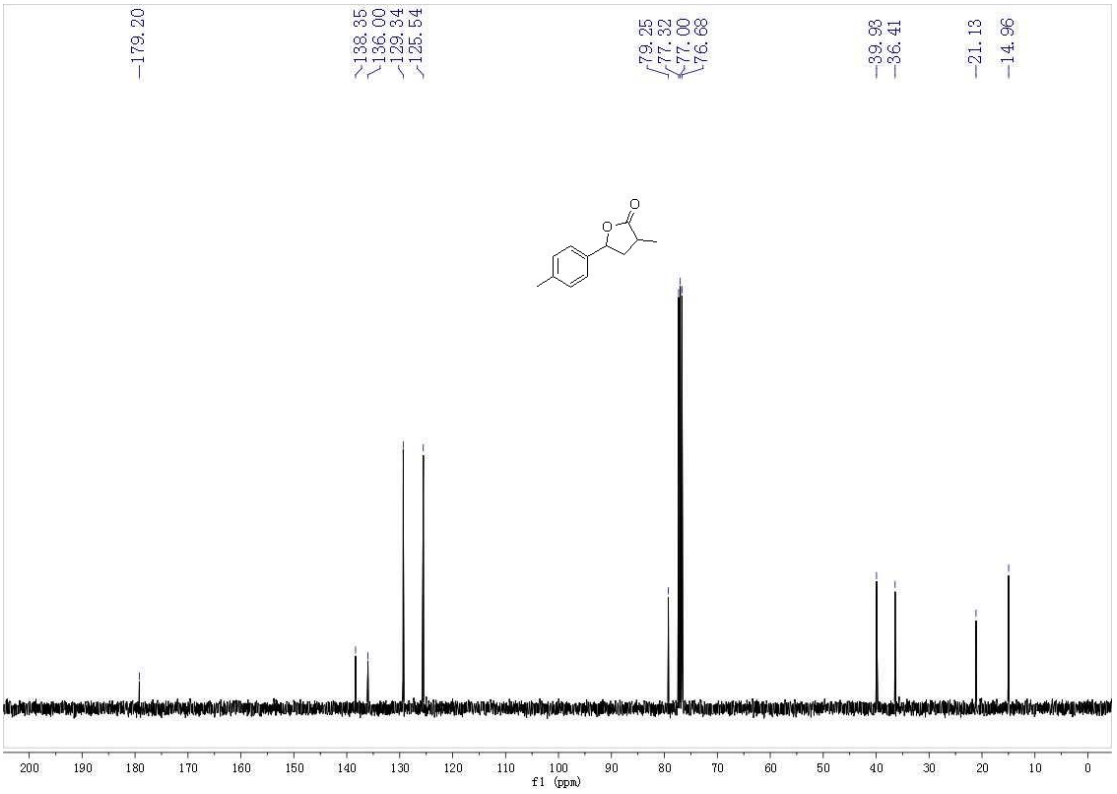


3-Methyl-5-*p*-tolyldihydrofuran-2(3*H*)-one (3aj): d.r. = 1.2:1

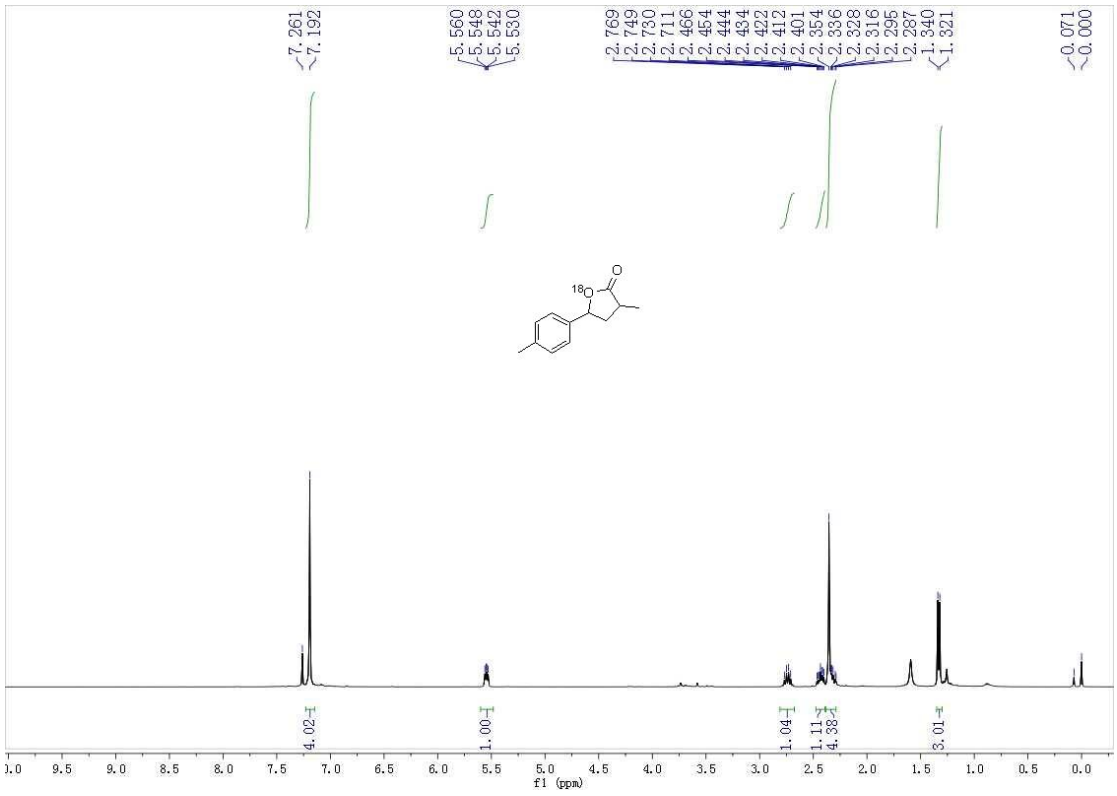


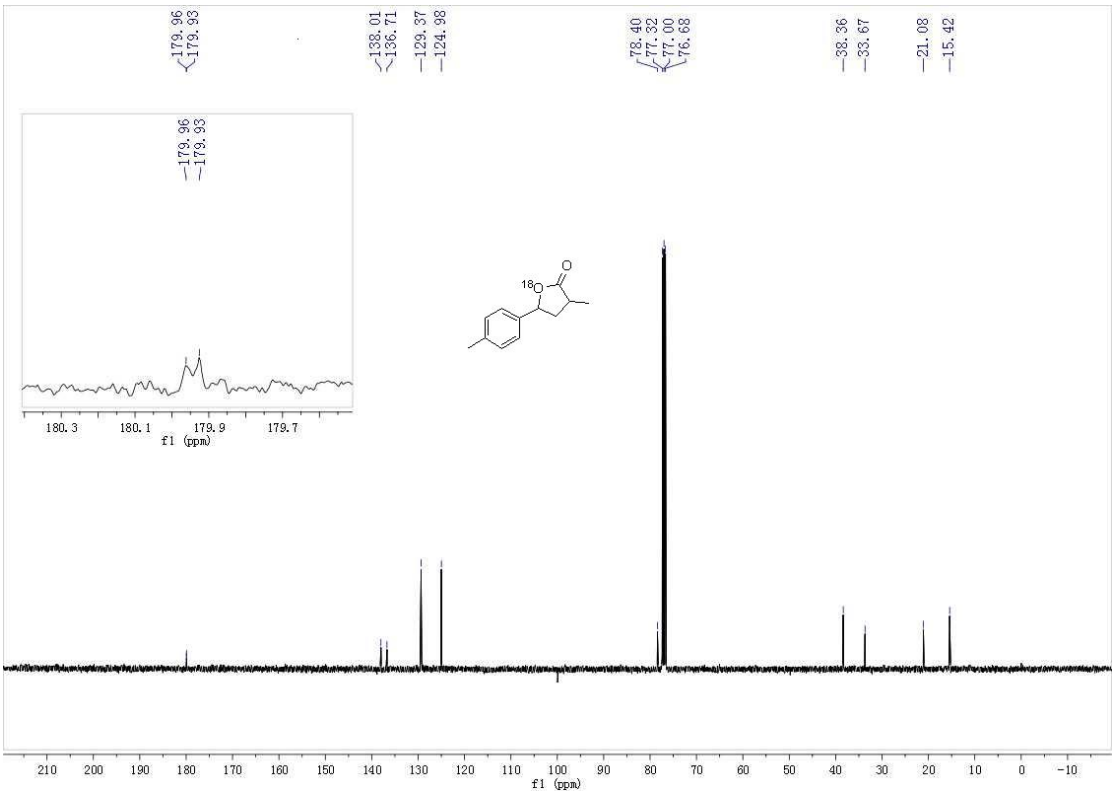


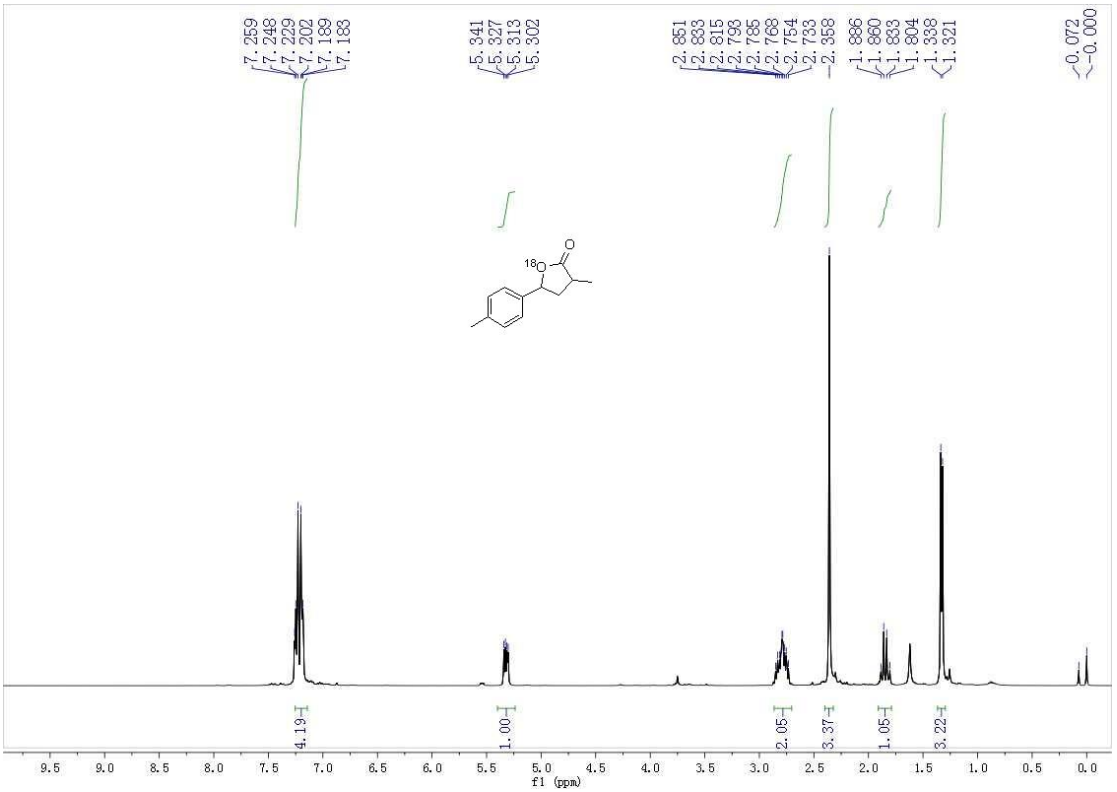

7.259
7.248
7.228
7.201
7.189
7.181
5.340
5.326
5.313
5.300
2.850
2.831
2.813
2.802
2.795
2.791
2.784
2.776
2.767
2.754
2.747
2.733
1.887
1.860
1.833
1.804
1.338
1.321
0.072
0.000
4.00
0.90
1.90
2.99
0.97
3.01
f1 (ppm)

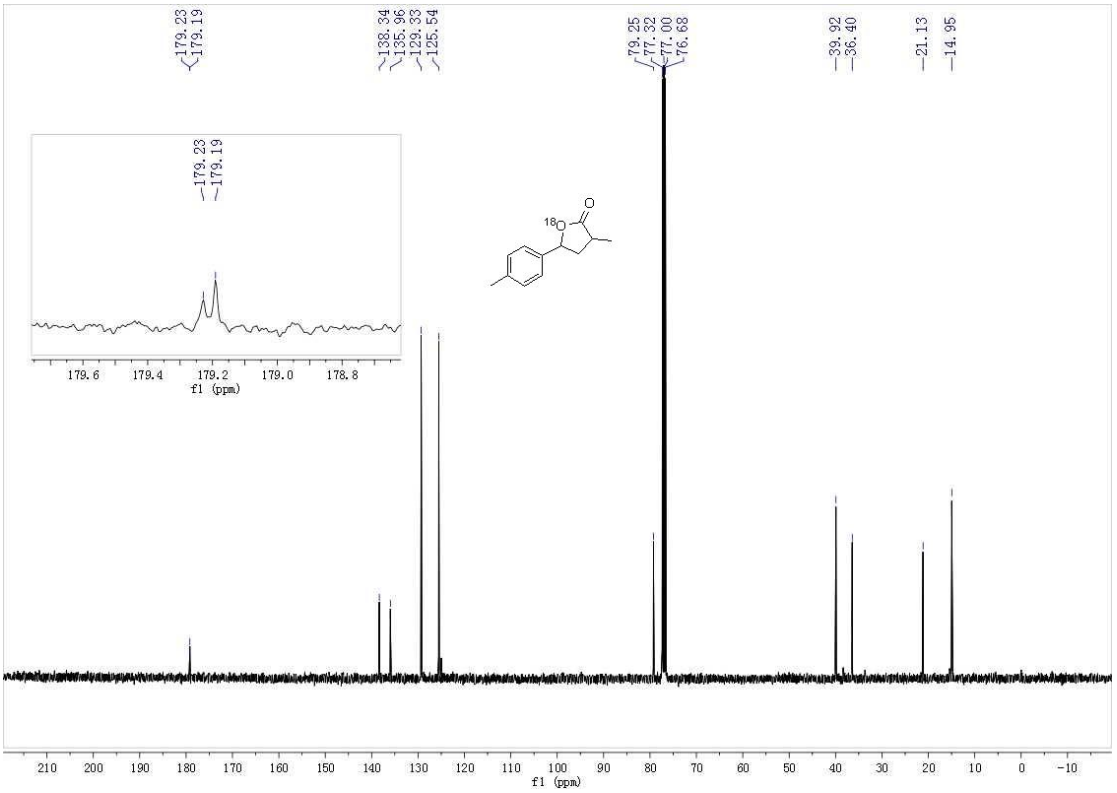

179.20
138.35
136.00
129.34
125.54
79.25
77.32
77.00
76.68
39.93
36.41
21.13
14.96
f1 (ppm)

3-Methyl-5-*p*-tolyldihydrofuran-2(3*H*)-one (3aj-18O): d.r. = 1.2:1






7.259
7.248
7.229
7.202
7.189
7.183
5.341
5.327
5.313
5.302
2.851
2.833
2.815
2.798
2.785
2.768
2.754
2.733
2.358
1.886
1.860
1.833
1.804
1.338
1.321
0.072
0.000
18O
O
4.19
1.00
2.05
3.37
1.05
3.22
f1 (ppm)


179.23
179.19
138.34
135.96
129.33
125.54
79.25
77.32
77.00
76.68
39.92
36.40
21.13
14.95
18O
O
f1 (ppm)
f1 (ppm)

_N_-(3,3-Dimethyl-5-_p_-tolyldihydrofuran-2(3_H_)-ylidene)aniline (3am)