

**Copper-Catalyzed Intermolecular Oxidative [3+2] Cycloaddition
between Alkenes and Anhydrides: A New Synthetic Approach to
 γ -Lactones**

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A. General method

¹H and ¹³C NMR spectra were recorded on BRUKER DRX-400 spectrometer using CDCl₃ as solvent and TMS as an internal standard. Gas chromatograph mass spectra were obtained with a SHIMADZU model GCMS-QP5000 spectrometer.

B. Screening reaction conditions and mechanistic experiments

To a Schlenk tube were added styrene (**1a**, 1 mmol), Cu catalyst (0.1 mmol), additive (0.3 mmol), base (1 mmol), and Ac₂O (2 mL). Then the tube was charged with O₂ (1 atm), and was stirred at 120 °C (oil bath temperature) for 12 hours. The reaction was monitored by gas chromatography (Table 1). The pure sample **2a** was obtained by isolation via silica gel column chromatography.

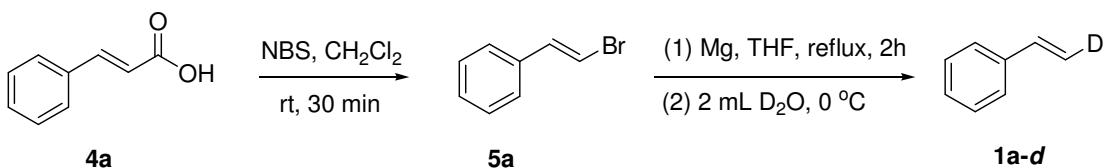
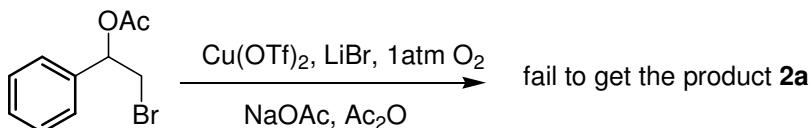
Table 1. Screening of Reaction Conditions

entry	catalyst	additive	base	solvent	temp (°C)	yield (%)
1	Cu(OTf) ₂	LiBr	NaOAc	Ac ₂ O	120	88
2	Cu(OAc) ₂	LiBr	NaOAc	Ac ₂ O	120	43
3	CuCl ₂	LiBr	NaOAc	Ac ₂ O	120	47
4	CuBr ₂	LiBr	NaOAc	Ac ₂ O	120	54
5	CuCl	LiBr	NaOAc	Ac ₂ O	120	43
6	CuBr	LiBr	NaOAc	Ac ₂ O	120	55
7	CuI	LiBr	NaOAc	Ac ₂ O	120	trace
8 ^c	Cu(OTf) ₂	LiBr	NaOAc	Ac ₂ O	120	86
9	Cu(OTf) ₂	LiBr	-	Ac ₂ O	120	41
10	Cu(OTf) ₂	LiBr	K ₂ CO ₃	Ac ₂ O	120	trace
11	Cu(OTf) ₂	LiBr	CsCO ₃	Ac ₂ O	120	46
12	Cu(OTf) ₂	LiBr	DBCAO	Ac ₂ O	120	51
13	Cu(OTf) ₂	LiBr	NaOAc	DMF	120	np
14	Cu(OTf) ₂	LiBr	NaOAc	NMP	120	np
15	Cu(OTf) ₂	LiBr	NaOAc	DMSO	120	np
16	Cu(OTf) ₂	LiBr	NaOAc	toluene	120	np
17	Cu(OTf) ₂	LiBr	NaOAc	dioxane	120	np

18 ^a	Cu(OTf) ₂	TEMPO	NaOAc	Ac ₂ O	120	np
19 ^b	Cu(OTf) ₂	LiBr	NaOAc	Ac ₂ O	120	66
120 ^c	Cu(OTf) ₂	LiBr	NaOAc	Ac ₂ O	120	62

^a the additive (1 equiv). ^b 1 equiv of TEMPO was added. ^c 1 equiv of 2β-di-tert-butyl-4-methylphenol (BHT) was added.

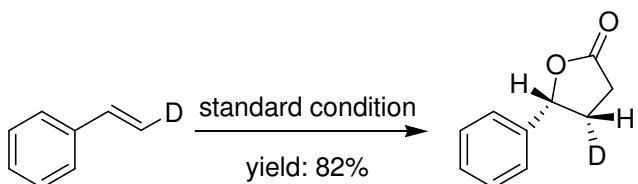
Mechanistic Experiments



To a solution of cinnamic acid (1.148 g, 10 mmol) in methylene chloride triethylamine (0.10 mL, 0.5 mmol) was added at room temperature and stirred for five minutes. N-bromosuccinimide (2.13 g, 12 mmol) was added in one portion and stirred for 1h. The solvent was removed under reduced pressure. The crude was washed by hexanes three times to afford 1.33 g of colorless oil for a 73% yield.

Grignard reagent was prepared from **5a** (1.33 g, 7.3 mmol) magnesium turnings, I₂ (0.127g, 0.5mmol) and in anhydrous THF (5 mL) refluxed 2h. And then add the reaction mixture to 2 mL D₂O (0 °C) to get the labeled styrene **2a-d** (0.52 g, 5 mmol).

To a Schlenk tube were added **1a-d** (1 mmol), Cu(OTf)₂ (0.1 mmol), LiBr (0.3 mmol), NaOAc (1 mmol), and Ac₂O (2 mL). Then the tube was charged with O₂ (1 atm), and was stirred at 120 °C (oil bath temperature) for 12 hours. The reaction was monitored by gas chromatography, and the pure sample **2a-d** was obtained by isolation via silica gel column chromatography.



1a-d

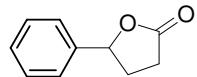
2a-d

C. General procedure for substrate 1a-1p

To a Schlenk tube were added alkene (1 mmol), Cu(OTf)₂ (0.1 mmol), LiBr (0.3 mmol), NaOAc (1 mmol), and Ac₂O (2 mL). Then the tube was charged with O₂ (1 atm), and was stirred at 120 °C (oil bath temperature) for the desired reaction time. After the reaction was finished, the reaction mixture was cooled to room temperature, diluted in diethyl ether, and wash with NaHCO₃. The aqueous phase was re-extracted with diethyl ether. The combined organic extracts were dried over MgSO₄ and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography using light petroleum ether/ethyl acetate as eluent to afford the desired product.

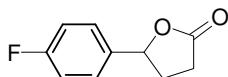
D. Analytical data for 2a-2o, 3a-3e

5-Phenyl-dihydro-furan-2-one (2a)



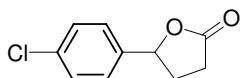
¹H NMR (CDCl₃, 400 MHz) δ 2.16-2.23 (m, 1H), 2.63-2.69 (m, 3H), 5.51 (dd, *J*₁ = 6.8 Hz, *J*₂ = 7.2 Hz, 1H), 7.32-7.41 (m, 5H); ¹³C NMR (CDCl₃, 100 MHz) δ 29.0, 31.0, 81.2, 125.3, 128.5, 128.8, 139.4, 176.9 ppm; MS (EI, 70 eV) *m/z* (%): 162 (M⁺, 100), 107 (60), 117 (43), 77 (31).

5-(4-Fluoro-phenyl)-dihydro-furan-2-one (2b)



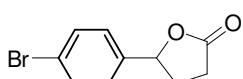
¹H NMR (CDCl₃, 400 MHz) δ 2.11-2.19 (m, 1H), 2.60-2.70 (m, 3H), 5.48 (dd, *J*₁ = 6.8 Hz, *J*₂ = 6.8 Hz, 1H), 7.07 (t, *J* = 8.4 Hz, 2H), 7.31 (t, *J* = 5.6 Hz, 2H); ¹³C NMR (CDCl₃, 100 MHz) δ 29.0, 31.0, 80.7, 115.6, 115.8, 127.2, 127.3, 135.1, 135.2, 161.4, 163.9, 176.7 ppm; MS (EI, 70 eV) *m/z* (%): 180 (M⁺, 69), 135 (62), 125 (100), 95 (37), 75 (19).

5-(4-Chloro-phenyl)-dihydro-furan-2-one (2c)



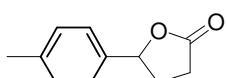
¹H NMR (CDCl₃, 400 MHz) δ 2.11-2.18 (m, 1H), 2.62-2.69 (m, 3H), 5.47 (dd, *J*₁ = 7.6 Hz, *J*₂ = 6.4 Hz, 1H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.35 (d, *J* = 8.4 Hz, 2H); ¹³C NMR (CDCl₃, 100 MHz) δ 28.9, 30.9, 80.5, 126.8, 129.0, 134.2, 137.9, 176.6 ppm; MS (EI, 70 eV) *m/z* (%): 196 (M⁺, 50), 198 (16), 161 (39), 141 (87), 117 (56), 56 (100).

5-(4-Bromo-phenyl)-dihydro-furan-2-one (2d)



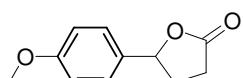
¹H NMR (CDCl₃, 400 MHz) δ 2.12-2.18 (m, 1H), 2.63-2.68 (m, 3H), 5.46 (dd, *J*₁ = 6.4 Hz, *J*₂ = 7.2 Hz, 1H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.52 (d, *J* = 8.4 Hz, 2H); ¹³C NMR (CDCl₃, 100 MHz) δ 29.0, 30.9, 80.5, 122.4, 127.0, 131.9, 138.5, 176.6 ppm; MS (EI, 70 eV) *m/z* (%): 240 (M⁺, 11), 185 (26), 161 (35), 115 (15), 28 (100).

5-*p*-Tolyl-dihydro-furan-2-one (2e)



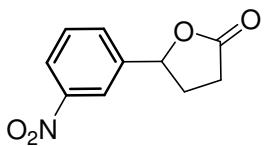
¹H NMR (CDCl₃, 400 MHz) δ 2.13-2.23 (m, 1H), 2.35 (s, 3H), 2.58-2.65 (m, 3H), 5.47 (dd, *J*₁ = 6.4 Hz, *J*₂ = 7.6 Hz, 1H), 7.20 (dd, *J* = 8.4 Hz, 4H); ¹³C NMR (CDCl₃, 100 MHz) δ 21.2, 29.1, 31.0, 81.4, 125.4, 129.4, 136.4, 138.3, 177.1 ppm; MS (EI, 70 eV) *m/z* (%): 176 (M⁺, 22), 163 (26), 134 (54), 121 (100), 93 (15).

5-(4-Methoxy-phenyl)-dihydro-furan-2-one (2f)



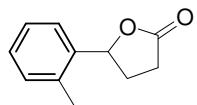
¹H NMR (CDCl₃, 400 MHz) δ 2.14-2.23 (m, 1H), 2.57-2.65 (m, 3H), 3.80 (s, 3H), 5.44 (dd, *J*₁ = 6.8 Hz, *J*₂ = 7.6 Hz, 1H), 6.91 (d, *J* = 8.8 Hz, 2H), 7.26 (d, *J* = 8.8 Hz, 2H); ¹³C NMR (CDCl₃, 100 MHz) δ 29.2, 30.9, 55.3, 81.4, 114.1, 127.0, 131.2, 159.8, 177.0 ppm; MS (EI, 70 eV) *m/z* (%): 193 (M+1, 11), 176 (14), 161 (29), 133 (100), 105 (87).

5-(3-Nitro-phenyl)-dihydro-furan-2-one (2g)



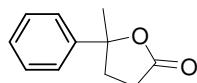
¹H NMR (CDCl₃, 400 MHz) δ 2.20-2.27 (m, 1H), 2.71-2.79 (m, 3H), 5.62 (dd, *J*₁ = 8 Hz, *J*₂ = 6.8 Hz, 1H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.72 (d, *J* = 7.6 Hz, 1H), 8.21 (d, *J* = 8.8 Hz, 2H); ¹³C NMR (CDCl₃, 100 MHz) δ 28.8, 30.9, 79.7, 120.4, 123.4, 130.0, 131.4, 141.7, 148.5, 176.2 ppm; MS (EI, 70 eV) *m/z* (%): 207 (M⁺, 8), 163 (9), 115 (41), 107 (95), 56 (100).

5-*o*-Tolyl-dihydro-furan-2-one (2h)



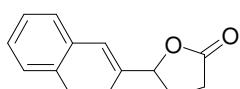
¹H NMR (CDCl₃, 400 MHz) δ 2.07-2.19 (m, 1H), 2.34 (s, 3H), 2.64-2.69 (m, 3H), 5.71 (dd, *J*₁ = 6.8 Hz, *J*₂ = 7.2 Hz, 1H), 7.19 (t, *J* = 8.4 Hz, 1H), 7.23-7.26 (m, 2H), 7.35 (d, *J* = 4.8 Hz, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 19.0, 28.7, 29.6, 78.9, 124.2, 126.5, 128.2, 130.7, 134.2, 137.5, 177.1 ppm; MS (EI, 70 eV) *m/z* (%): 176 (M⁺, 100), 161 (22), 131 (50), 121 (69), 91 (59).

5-Methyl-5-phenyl-dihydro-furan-2-one (2i)



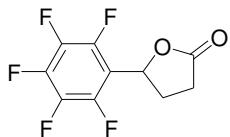
¹H NMR (CDCl₃, 400 MHz) δ 1.72, (s, 3H), 2.41-2.53 (m, 3H), 2.59-2.67 (m, 1H), 7.27-7.31 (m, 1H), 7.37 (d, *J* = 8.0 Hz, 4H); ¹³C NMR (CDCl₃, 100 MHz) δ 29.0, 29.4, 36.2, 87.0, 124.1, 127.6, 128.6, 144.3, 176.5 ppm; MS (EI, 70 eV) *m/z* (%): 176 (M⁺, 5), 161 (100), 121 (31), 105 (38), 77 (22).

5-Naphthalen-2-yl-dihydro-furan-2-one (2j)



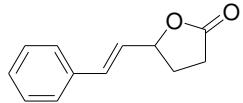
¹H NMR (CDCl₃, 400 MHz) δ 2.22-2.29 (m, 1H), 2.64-2.72 (m, 3H), 5.66 (dd, *J*₁ = 8 Hz, *J*₂ = 6.8 Hz, 1H), 7.38 (dd, *J* = 6.8 Hz, 1H), 7.48-7.51 (m, 2H), 7.79-7.87 (m, 4H); ¹³C NMR (CDCl₃, 100 MHz) δ 28.9, 30.9, 81.3, 122.9, 124.3, 126.5, 126.6, 127.8, 128.1, 128.9, 133.1, 133.2, 136.7, 177.0 ppm; MS (EI, 70 eV) *m/z* (%): 212 (M⁺, 27), 167 (11), 155 (20), 127 (14), 28 (100).

5-Pentafluorophenyl-dihydro-furan-2-one (2k)



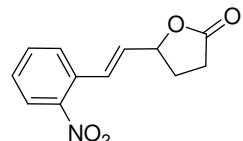
¹H NMR (CDCl₃, 400 MHz) δ 2.39-2.48 (m, 1H), 2.68-2.85 (m, 3H), 5.83 (dd, *J*₁ = 7.6 Hz, *J*₂ = 7.6 Hz, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ 27.6, 28.1, 71.1, 113.0, 136.5, 139.0, 140.4, 142.9, 143.8, 146.3, 175.6 ppm; MS (EI, 70 eV) *m/z* (%): 152 (M⁺, 45), 208 (85), 181 (79), 117 (18), 56 (100).

5-Styryl-dihydro-furan-2-one (2l)



¹H NMR (CDCl₃, 400 MHz) δ 2.05-2.15 (m, 1H), 2.46-2.51 (m, 1H), 2.58-2.62 (m, 2H), 5.13 (dd, *J*₁ = 6.8 Hz, *J*₂ = 6.8 Hz, 1H), 6.20 (q, *J* = 6.8 Hz, 1H), 6.68 (d, *J* = 16 Hz, 1H), 7.26-7.40 (m, 5H); ¹³C NMR (CDCl₃, 100 MHz) δ 28.6, 28.9, 80.7, 126.4, 126.7, 128.4, 128.7, 132.9, 135.7, 176.9 ppm; MS (EI, 70 eV) *m/z* (%): 212 (M⁺, 27), 167 (11), 155 (20), 127 (14), 28 (100).

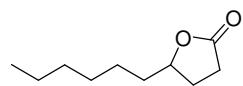
5-[2-(2-Nitro-phenyl)-vinyl]-dihydro-furan-2-one (2m)



¹H NMR (CDCl₃, 400 MHz) δ 2.12-2.20 (m, 1H), 2.52-2.66 (m, 3H), 5.17 (dd, *J*₁ = 6.8 Hz, *J*₂ = 6.8 Hz, 1H), 6.18 (dd, *J* = 9.2 Hz, 1H), 7.18 (d, *J* = 15.6 Hz, 1H),

7.44-7.48 (m, 1H), 7.58-7.64 (m, 2H), 7.79 (d, J = 8 Hz, 1H); ^{13}C NMR (CDCl₃, 100 MHz) δ 28.5, 28.6, 80.0, 124.7, 128.7, 128.9, 129.0, 131.6, 131.7, 133.4, 147.8, 176.6 ppm; MS (EI, 70 eV) m/z (%): 149 (16) 132 (24), 120 (42), 91 (23), 85 (100).

5-Hexyl-dihydro-furan-2-one (2n)



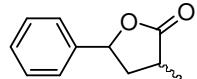
^1H NMR (CDCl₃, 400 MHz) δ 0.888 (t, J = 6.0 Hz, 3H), 1.29-1.46 (m, 8H), 1.57-1.63 (m, 1H), 1.70-1.75 (m, 1H), 1.83-1.88 (m, 1H), 2.28-2.37 (m, 1H), 2.53 (q, J = 6.8 Hz, 2H), 4.46-4.52 (m, 1H); ^{13}C NMR (CDCl₃, 100 MHz) δ 14.0, 22.5, 25.2, 28.0, 28.9, 29.0, 31.6, 35.6, 81.1, 177.4 ppm; MS (EI, 70 eV) m/z (%): 128 (6), 85 (79), 58 (7), 43 (11), 28 (100).

5-Phenylethynyl-dihydro-furan-2-one (2o)



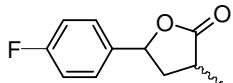
^1H NMR (CDCl₃, 400 MHz) δ 2.37-2.43 (m, 1H), 2.52-2.65 (m, 2H), 2.69-2.75 (m, 1H), 5.36 (dd, J_1 = 6.8 Hz, J_2 = 6.4 Hz, 1H), 7.30-7.35 (m, 3H), 7.43-7.45 (m, 2H); ^{13}C NMR (CDCl₃, 100 MHz) δ 27.9, 29.9, 69.7, 85.0, 87.4, 121.6, 128.4, 129.1, 131.8, 176.2 ppm; MS (EI, 70 eV) m/z (%): 186 (M⁺, 11), 158 (8), 141 (14), 115 (10), 28 (100).

3-Methyl-5-phenyl-dihydro-furan-2-one (3a)



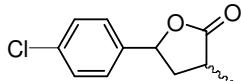
^1H NMR (CDCl₃, 400 MHz) δ 1.31 (s, 1.42H), 1.33 (s, 1.48H), 1.82-1.89 (m, 0.4H), 2.31-2.45 (m, 1.2H), 2.71-2.84 (m, 1.4H), 5.35 (q, J = 5.6 Hz, 0.3H), 5.56 (q, J = 4.8Hz, 0.7H), 7.29-7.40 (m, 5H); ^{13}C NMR (CDCl₃, 100 MHz) δ 15.0, 15.4, 33.6, 36.4, 38.3, 40.0, 78.4, 79.2, 125.0, 125.5, 128.2, 128.5, 128.7, 128.8, 139.1, 139.8, 179.2, 180.0 ppm; MS (EI, 70 eV) m/z (%): 176 (M⁺, 21), 132 (22), 117 (57), 105 (28), 28 (100).

5-(4-Fluoro-phenyl)-3-methyl-dihydro-furan-2-one (3b)



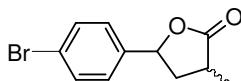
¹H NMR (CDCl₃, 400 MHz) δ 1.34 (d, *J* = 7.2 Hz, 3H), 2.32-2.41 (m, 2H), 2.69-2.83 (m, 1H), 5.33 (q, *J* = 5.6 Hz, 0.3H), 5.55 (t, *J* = 6.4 Hz, 0.7H), 7.05-7.35 (m, 4H); ¹³C NMR (CDCl₃, 100 MHz) δ 14.9, 15.4, 33.7, 36.4, 38.3, 40.0, 77.8, 78.6, 115.6, 115.8, 126.8, 126.9, 127.4, 127.5, 135.50, 135.53, 161.3, 163.7, 178.9, 179.6 ppm; MS (EI, 70 eV) *m/z* (%): 194 (M⁺, 27), 150 (50), 135 (100), 123 (64), 109 (23).

5-(4-Chloro-phenyl)-3-methyl-dihydro-furan-2-one (3c)



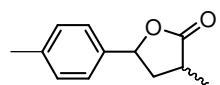
¹H NMR (CDCl₃, 400 MHz) δ 1.32 (dd, *J* = 4.0 Hz, 3H), 1.75-1.85 (m, 0.5H), 2.35-2.40 (m, 1H), 2.68-2.87 (m, 1.5H), 5.32 (q, *J* = 5.6 Hz, 0.55H), 5.53 (q, *J* = 6.8 Hz, 0.44H), 7.23-7.36 (m, 4H); ¹³C NMR (CDCl₃, 100 MHz) δ 14.9, 15.4, 33.6, 36.3, 38.2, 39.9, 77.7, 78.4, 126.5, 126.9, 128.89, 128.91, 134.0, 134.2, 137.7, 138.3, 178.9, 179.6 ppm; MS (EI, 70 eV) *m/z* (%): 210 (M⁺, 42), 212 (15), 175 (46), 151 (50), 131 (100).

5-(4-Bromo-phenyl)-3-methyl-dihydro-furan-2-one (3d)



¹H NMR (CDCl₃, 400 MHz) δ 1.31-1.33 (m, 3H), 2.35-2.38 (m, 1.87H), 2.68-2.81 (m, 1.47H), 5.31 (q, *J* = 5.6 Hz, 0.45H), 5.51 (q, *J* = 6.8 Hz, 0.55H), 7.17-7.51 (m, 4H); ¹³C NMR (CDCl₃, 100 MHz) δ 14.9, 15.4, 33.6, 36.3, 38.2, 39.9, 77.7, 78.4, 122.1, 122.3, 126.8, 127.2, 131.85, 131.86, 138.2, 138.9, 178.9, 179.6 ppm; MS (EI, 70 eV) *m/z* (%): 254 (M⁺, 41), 256 (41), 185 (58), 175 (75), 131 (89), 116 (100).

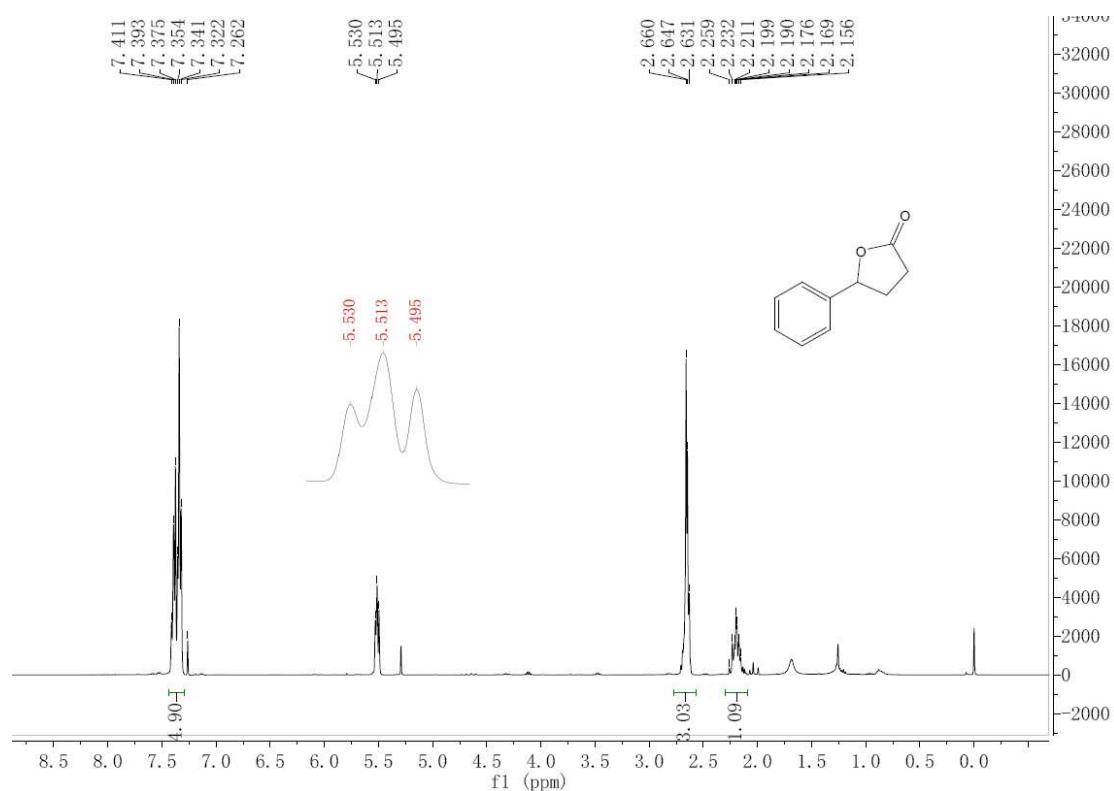
Dihydro -3-methyl-5-*p*-tolylfuran-2-(3*H*)-one (3e)

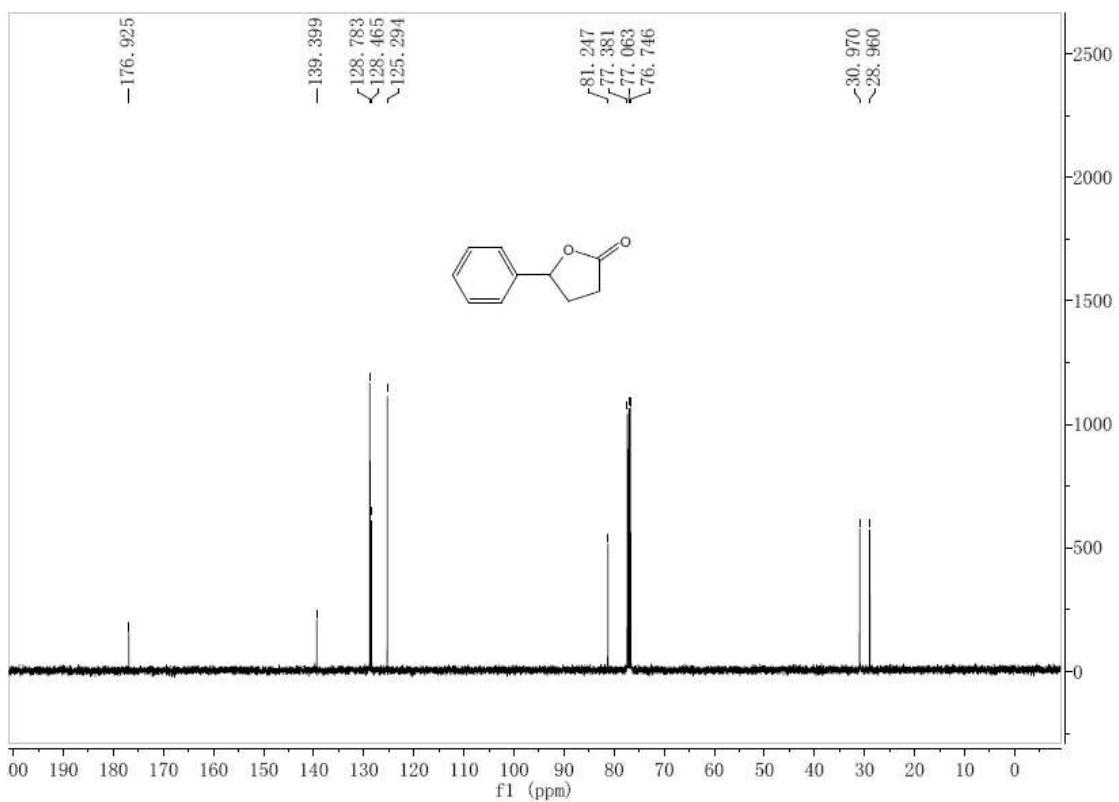


¹H NMR (CDCl₃, 400 MHz) δ 1.31 (d, *J* = 7.2 Hz, 3H), 1.84 (q, *J* = 10.8 Hz, 0.6H), 2.34 (d, *J* = 1.6 Hz, 3H), 2.38-2.45 (m, 0.6H), 2.68-2.84 (m, 1.6H), 5.28 (q, *J* = 5.6 Hz, 0.56H), 5.50 (t, *J* = 5.2 Hz, 0.44H), 7.17-7.24 (m, 4H); ¹³C NMR (CDCl₃, 100 MHz) δ 15.0, 15.4, 21.1, 21.2, 33.7, 36.5, 38.3, 39.4, 78.5, 79.3, 125.1, 125.6, 129.36, 129.39, 136.0, 136.8, 138.0, 138.4, 179.4, 180.1 ppm; MS (EI, 70 eV) *m/z* (%): 190 (M⁺, 74), 175 (31), 146 (30), 131 (98), 119 (100), 91 (59), 65 (16), 42 (24).

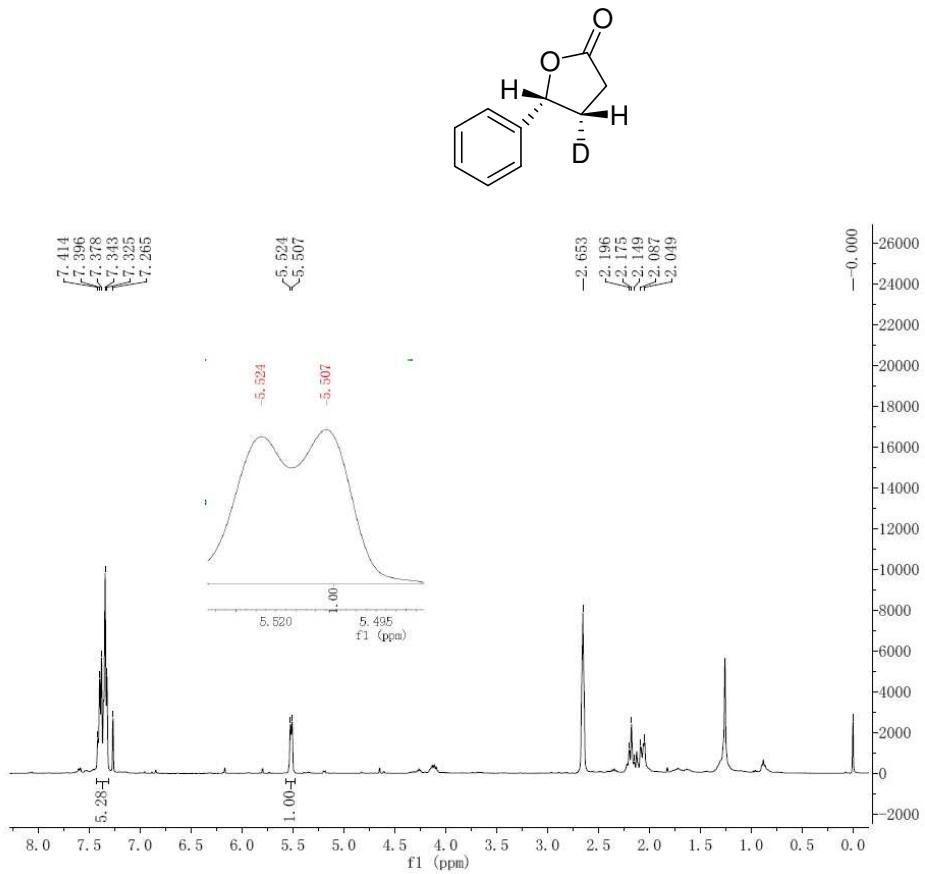
E. NMR Spectra

5-Phenyl-dihydro-furan-2-one (2a)

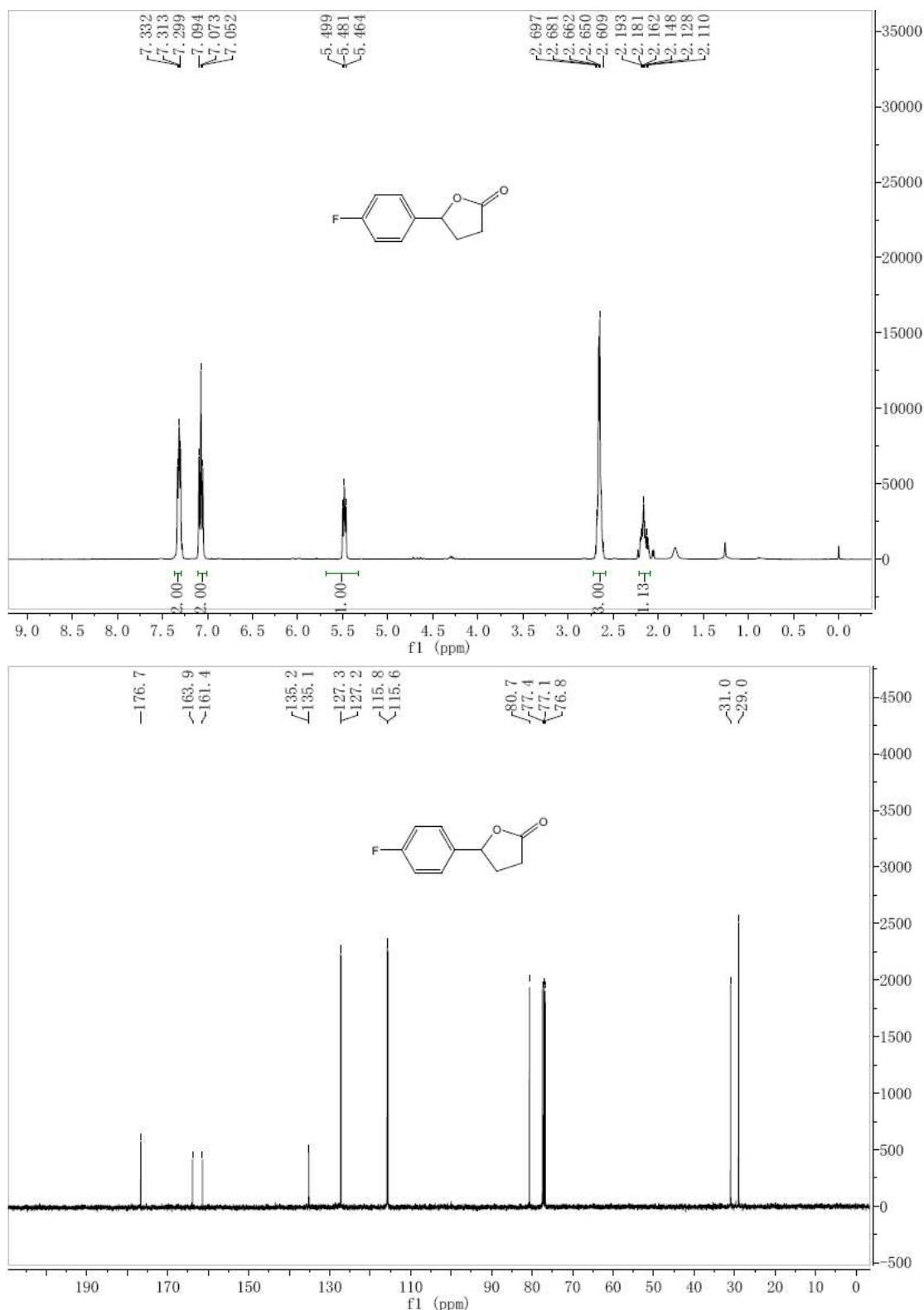




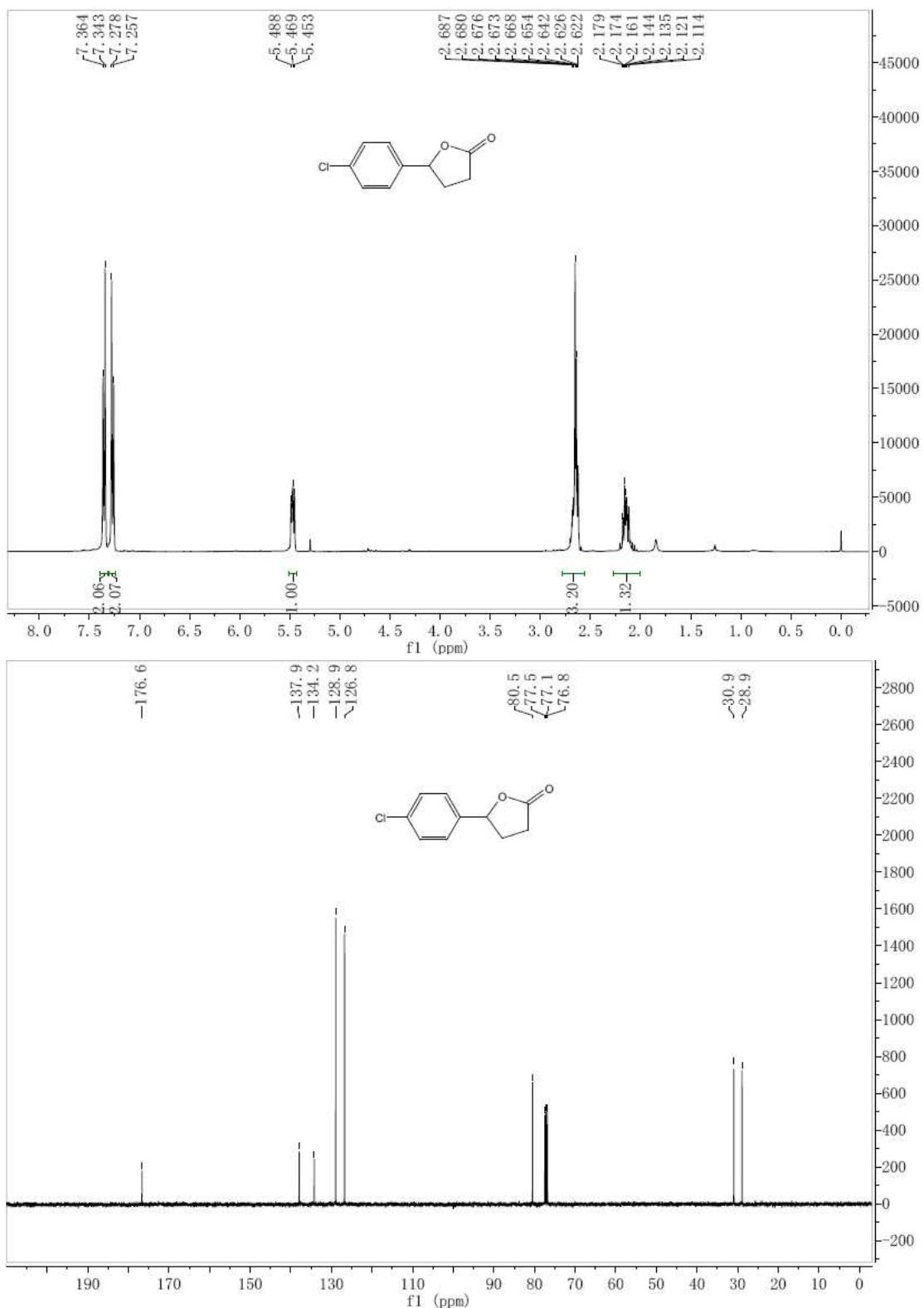
4-D-5-Phenyl-dihydro-furan-2-one (4-D-2a)



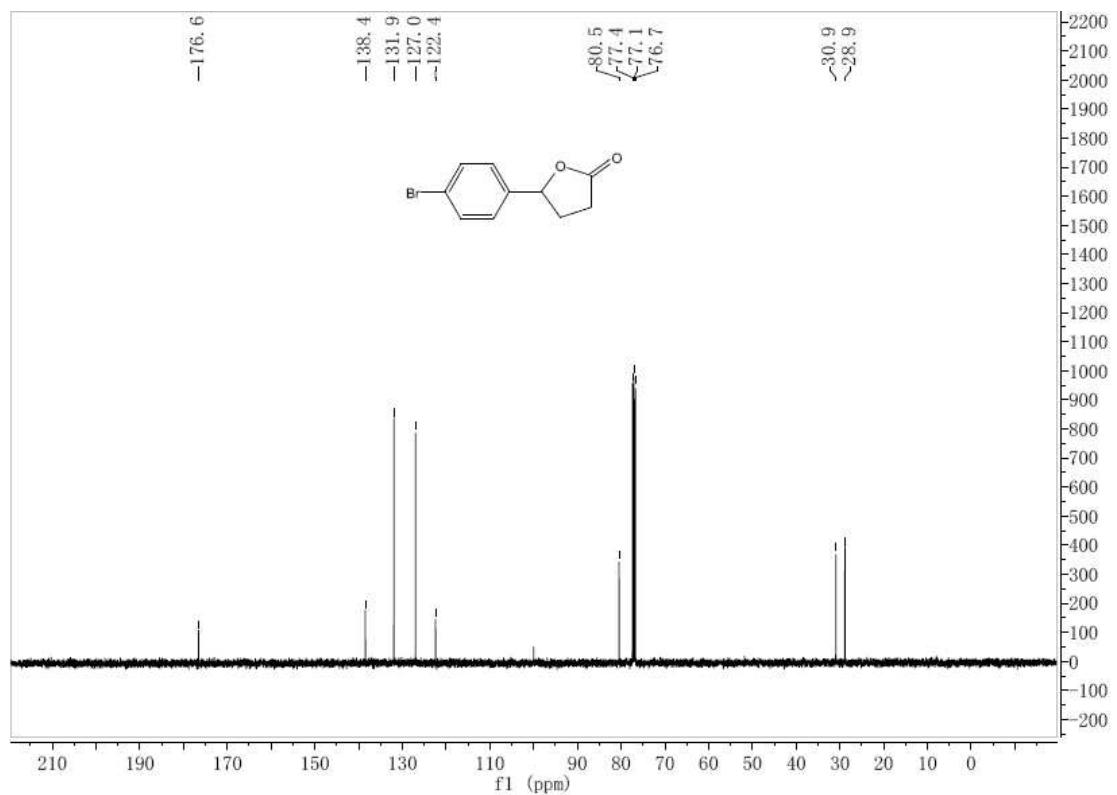
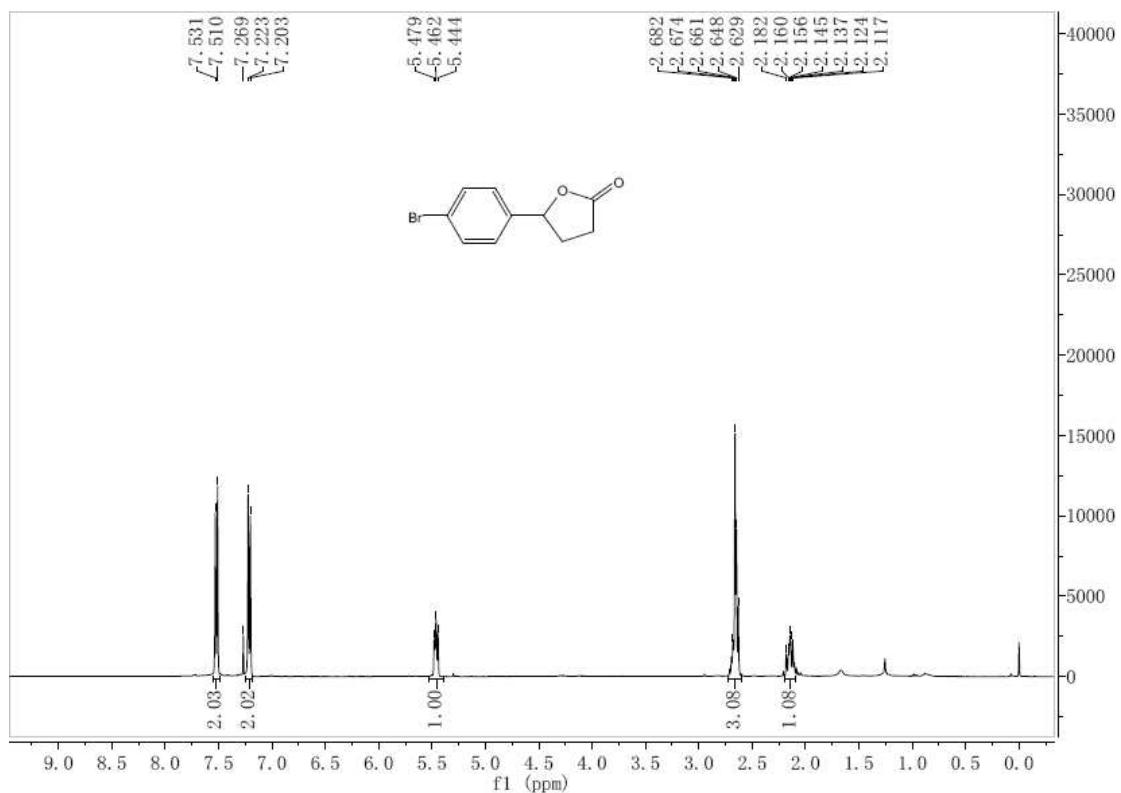
5-(4-Fluoro-phenyl)-dihydro-furan-2-one (2b)



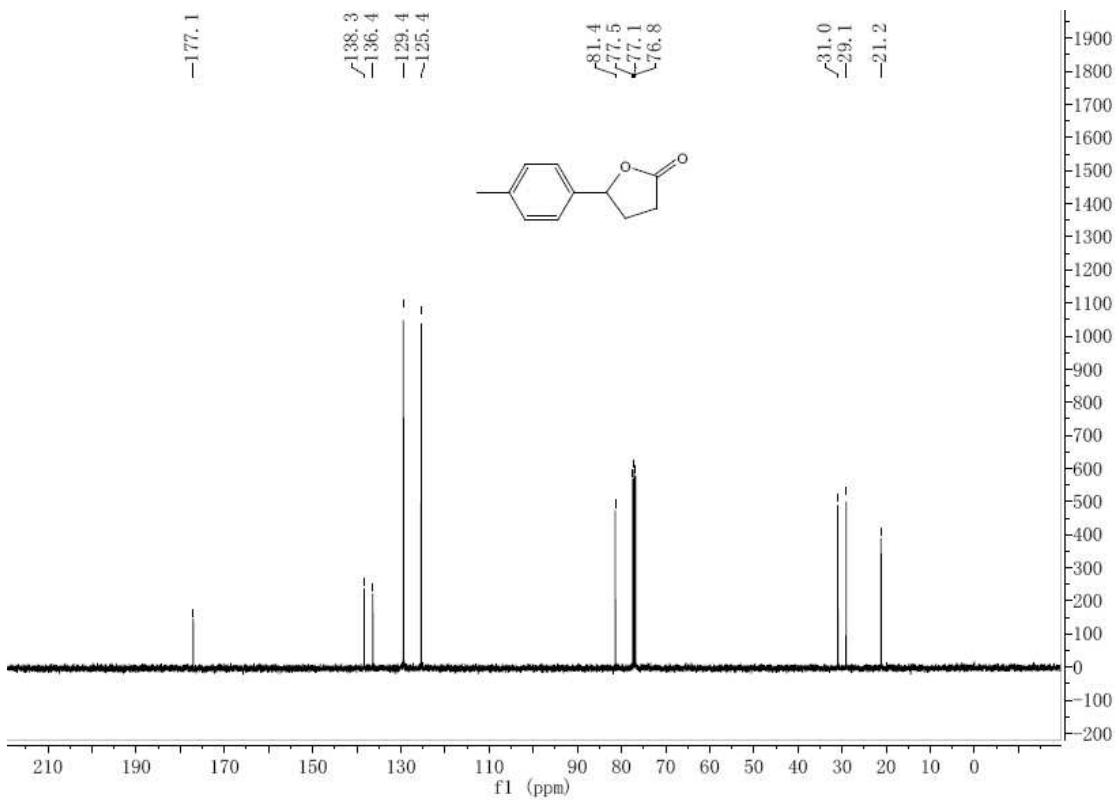
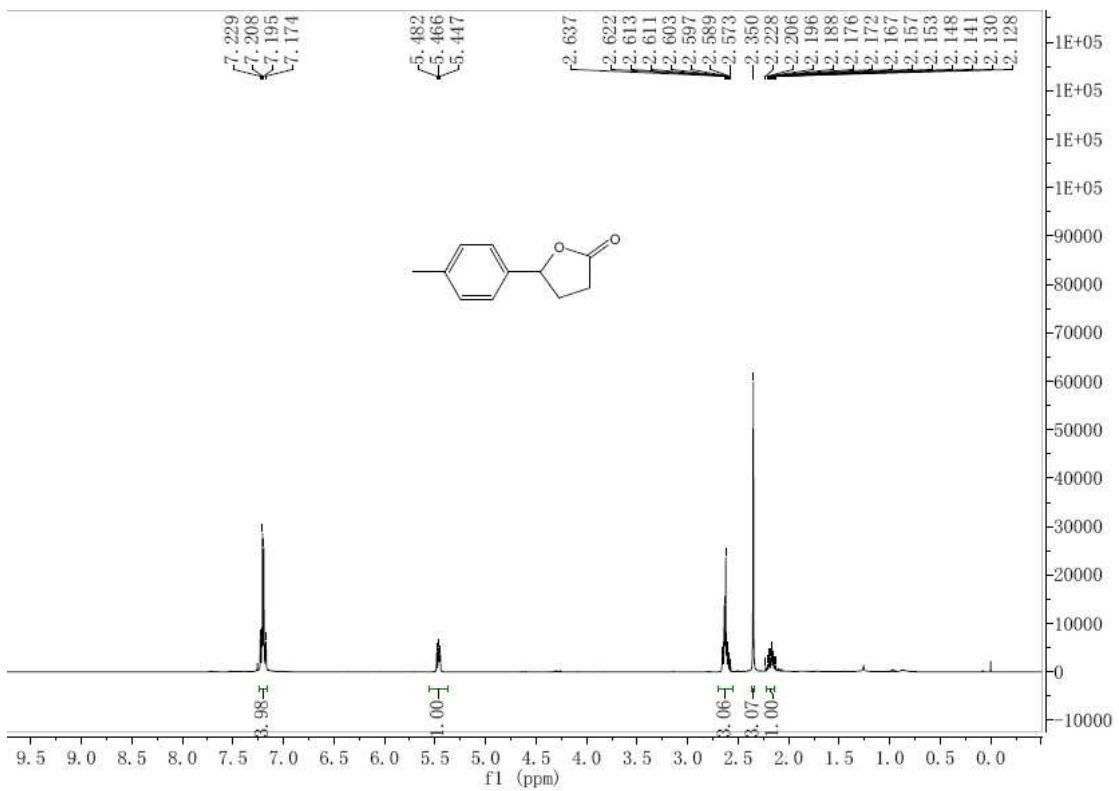
5-(4-Chloro-phenyl)-dihydro-furan-2-one (2c)



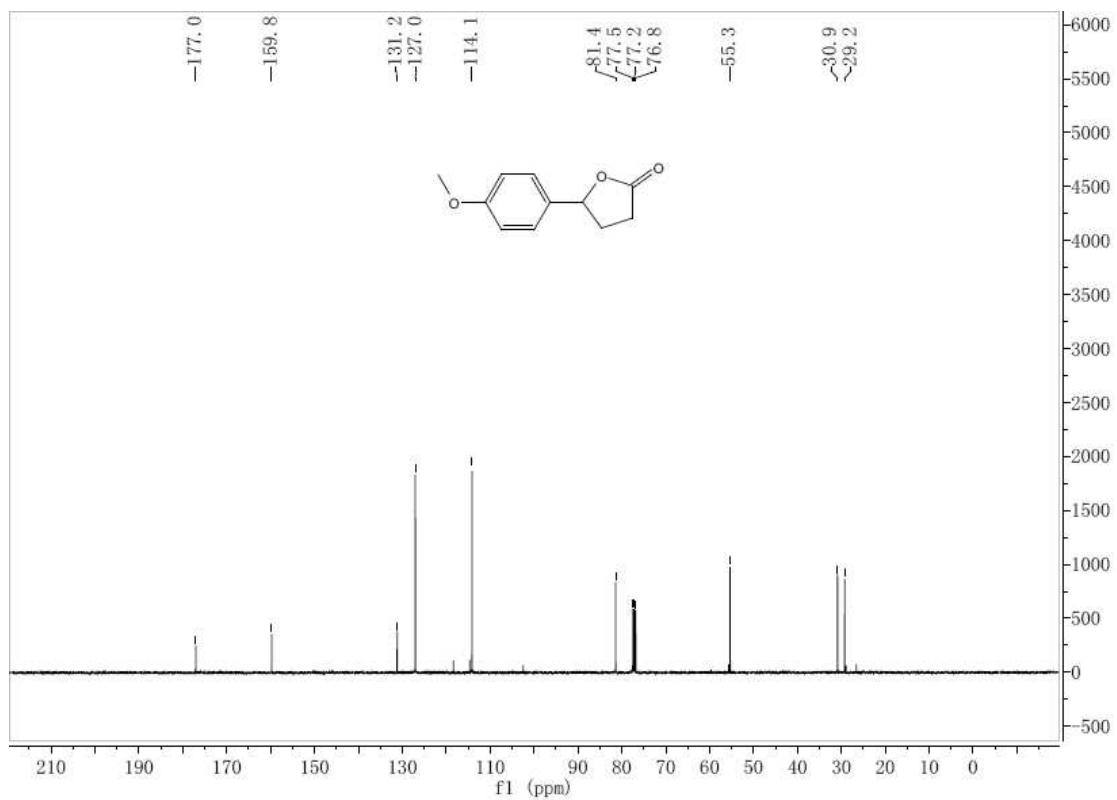
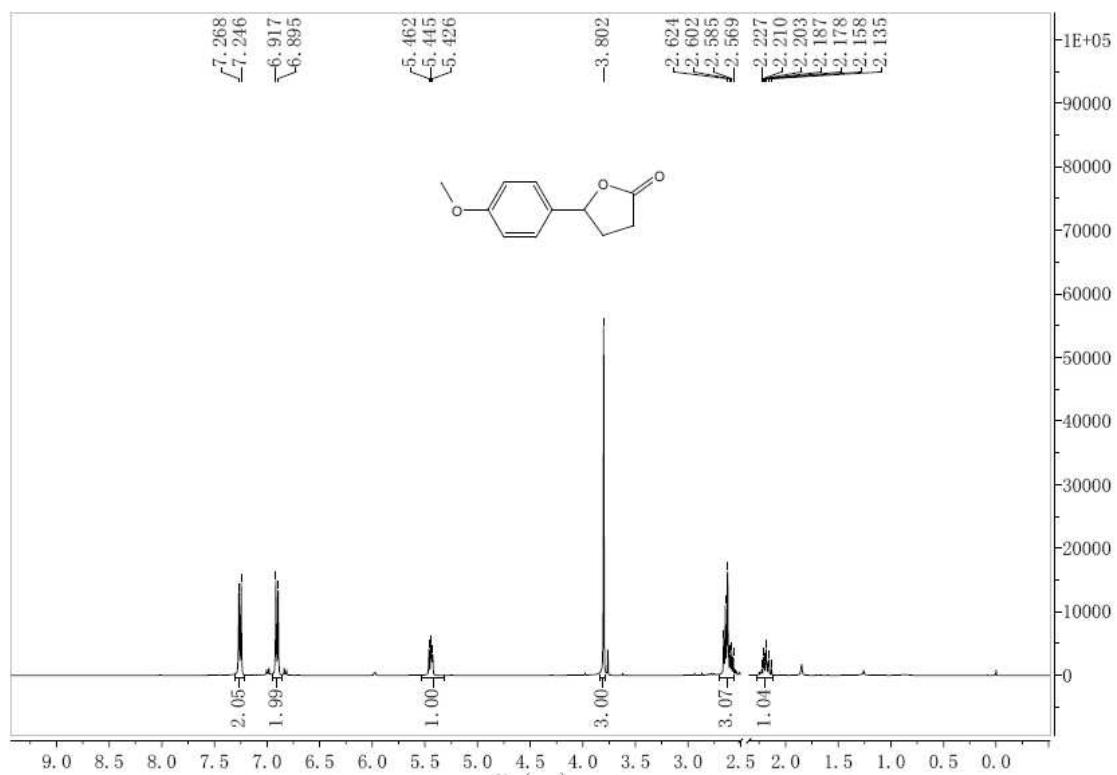
5-(4-Bromo-phenyl)-dihydro-furan-2-one (2d)



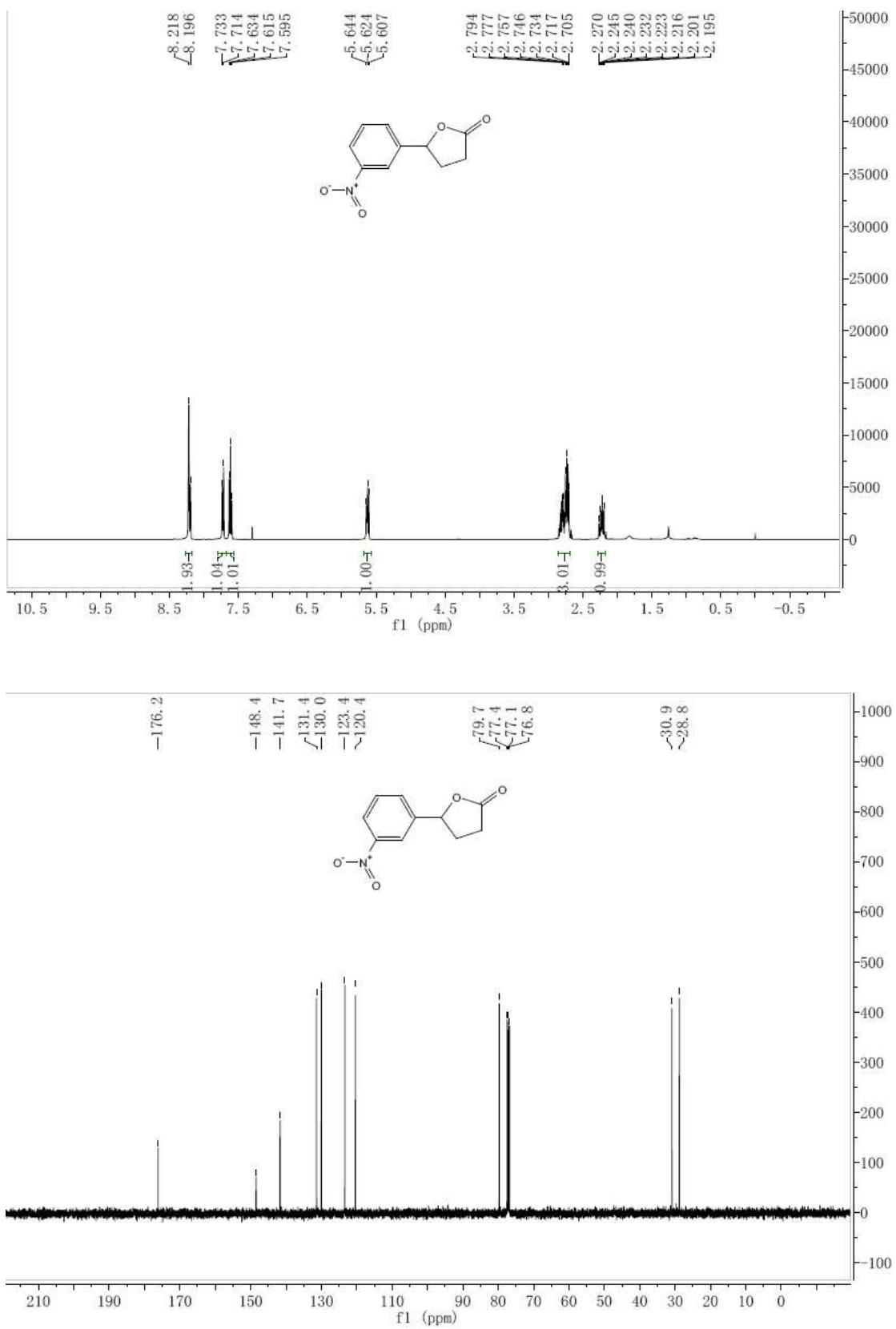
5-p-Tolyl-dihydro-furan-2-one (2e)



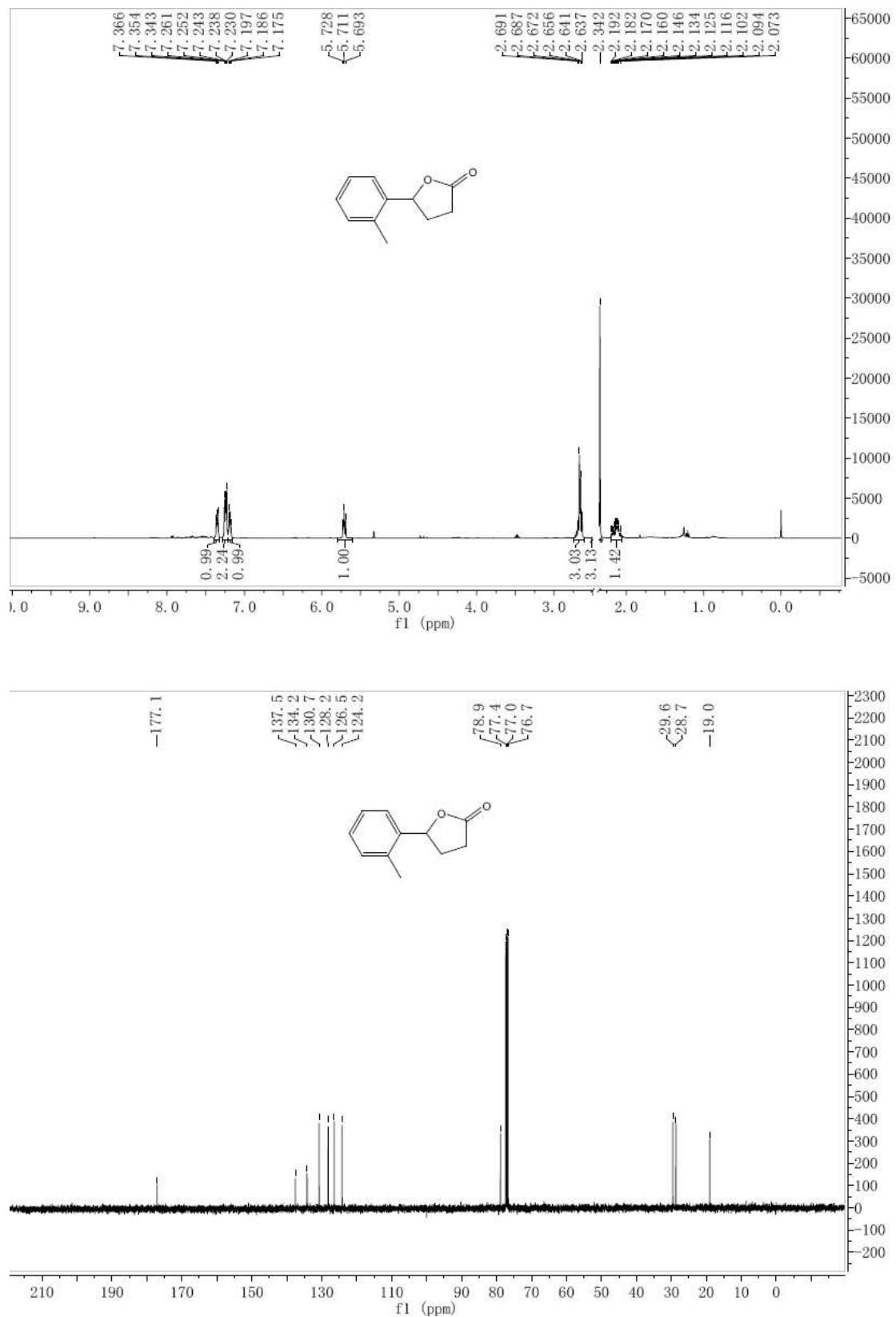
5-(4-Methoxy-phenyl)-dihydro-furan-2-one (2f)



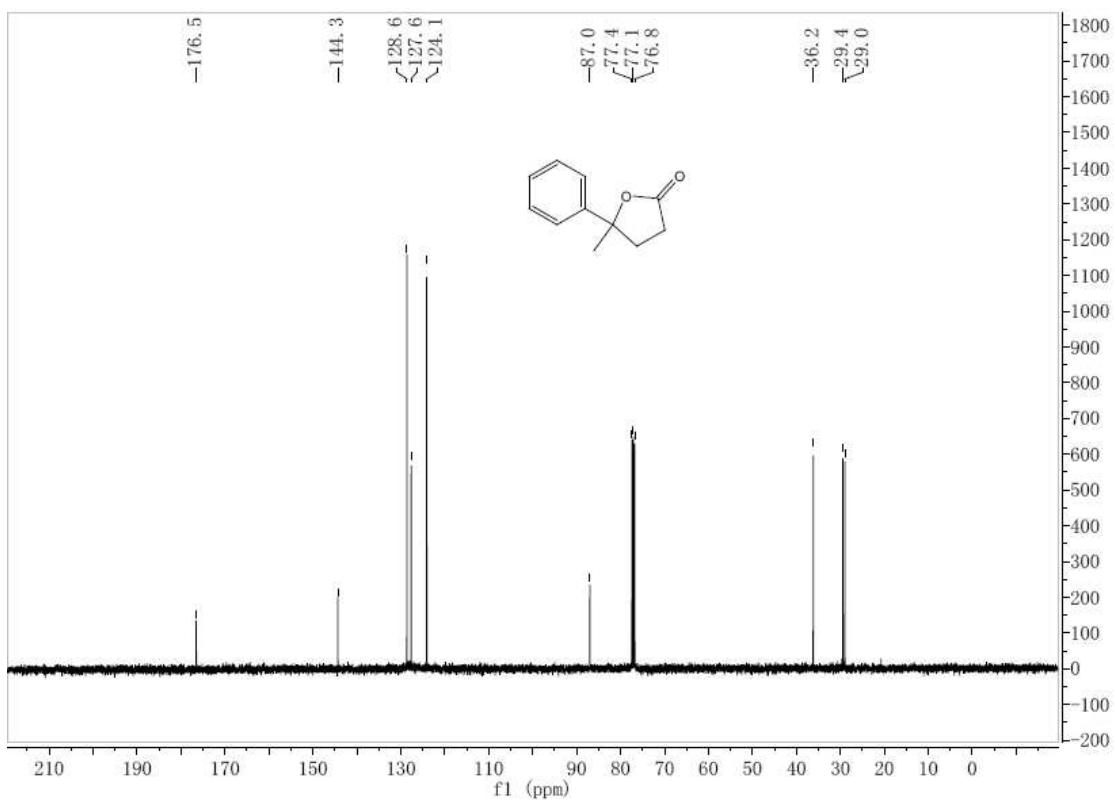
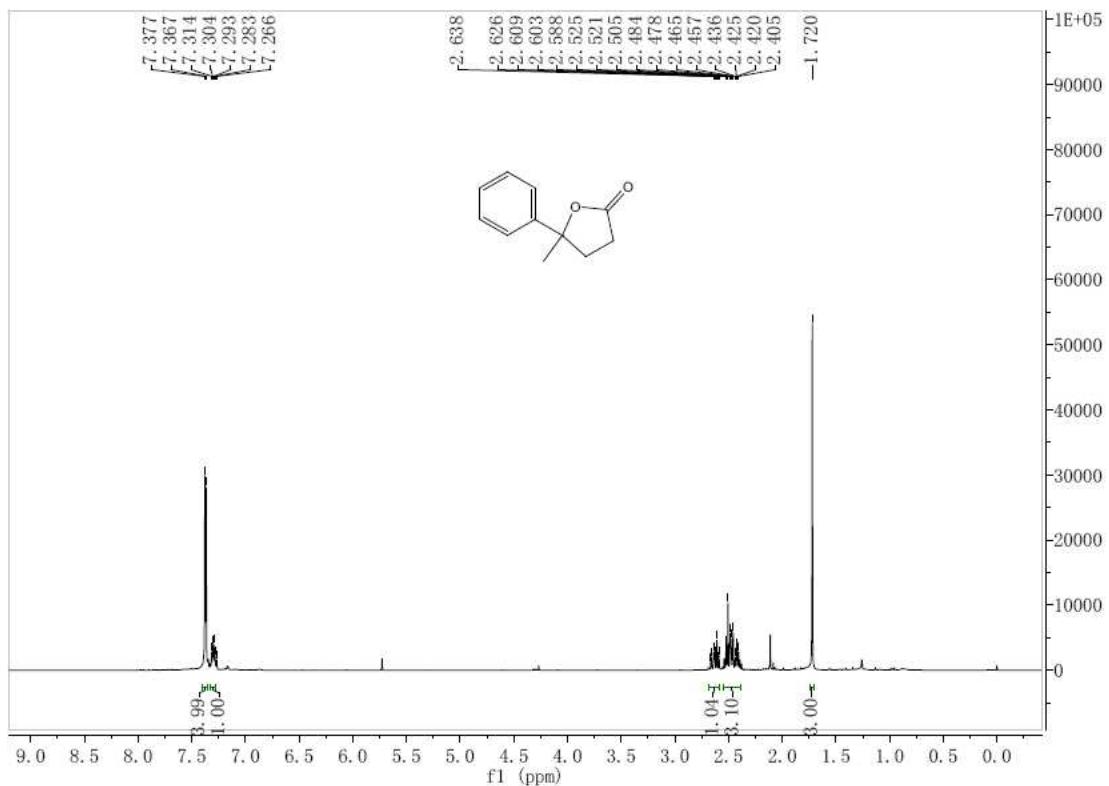
5-(3-Nitro-phenyl)-dihydro-furan-2-one (2g)



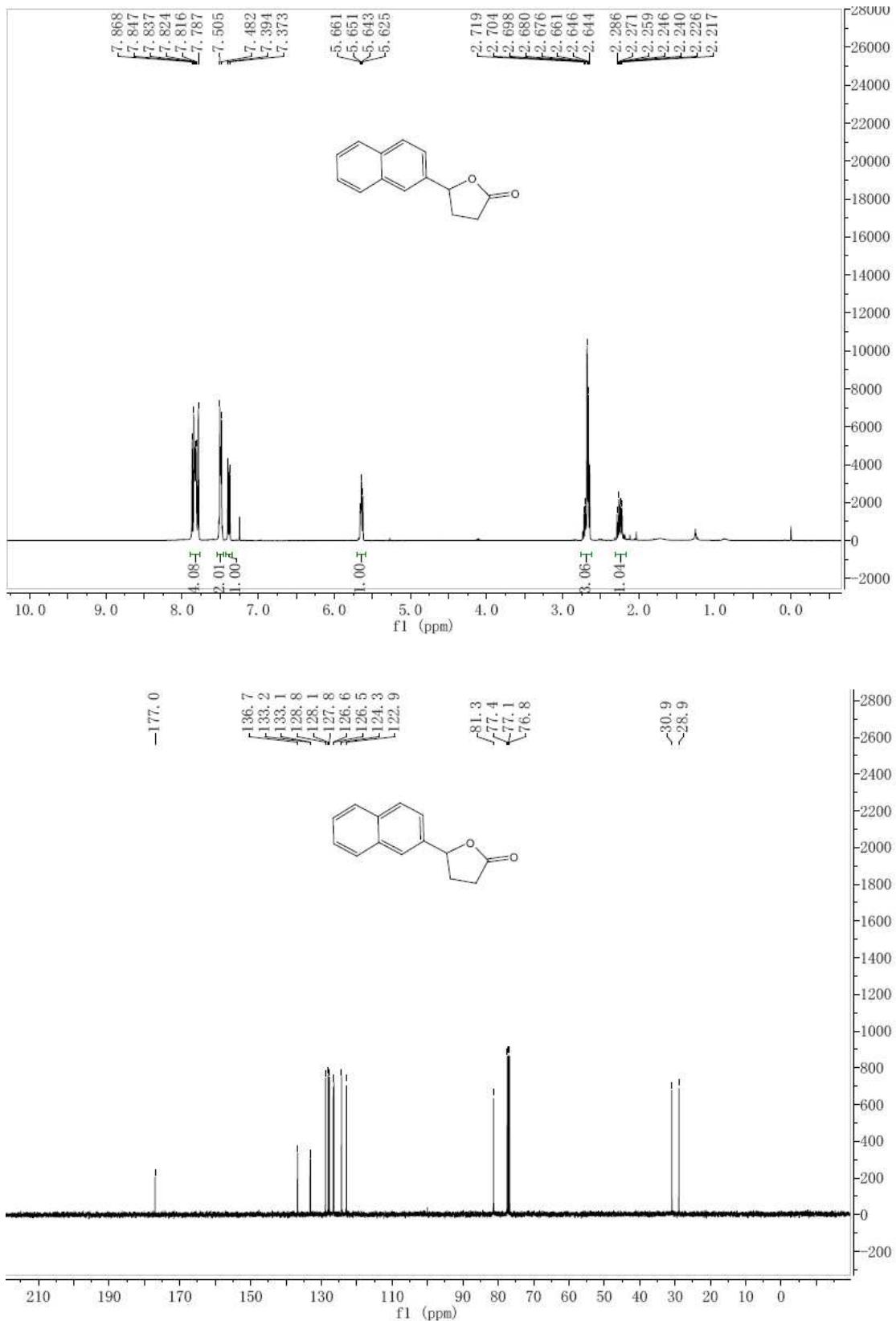
5-o-Tolyl-dihydro-furan-2-one (2h)



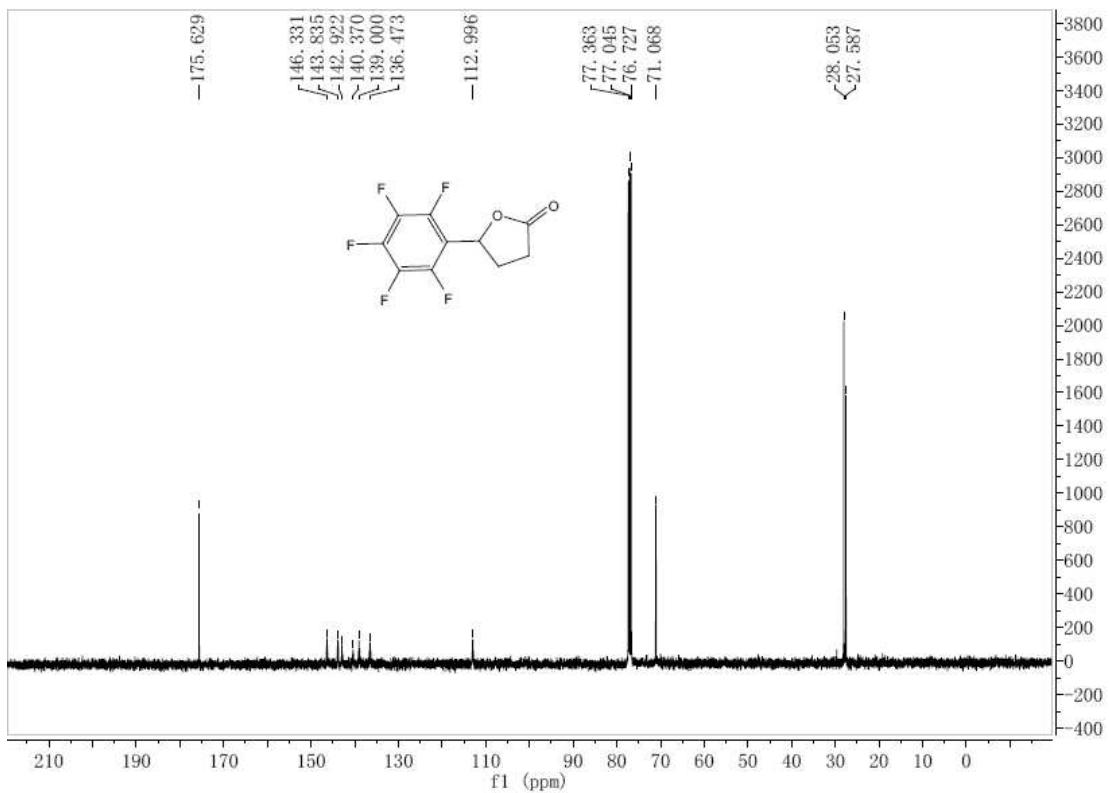
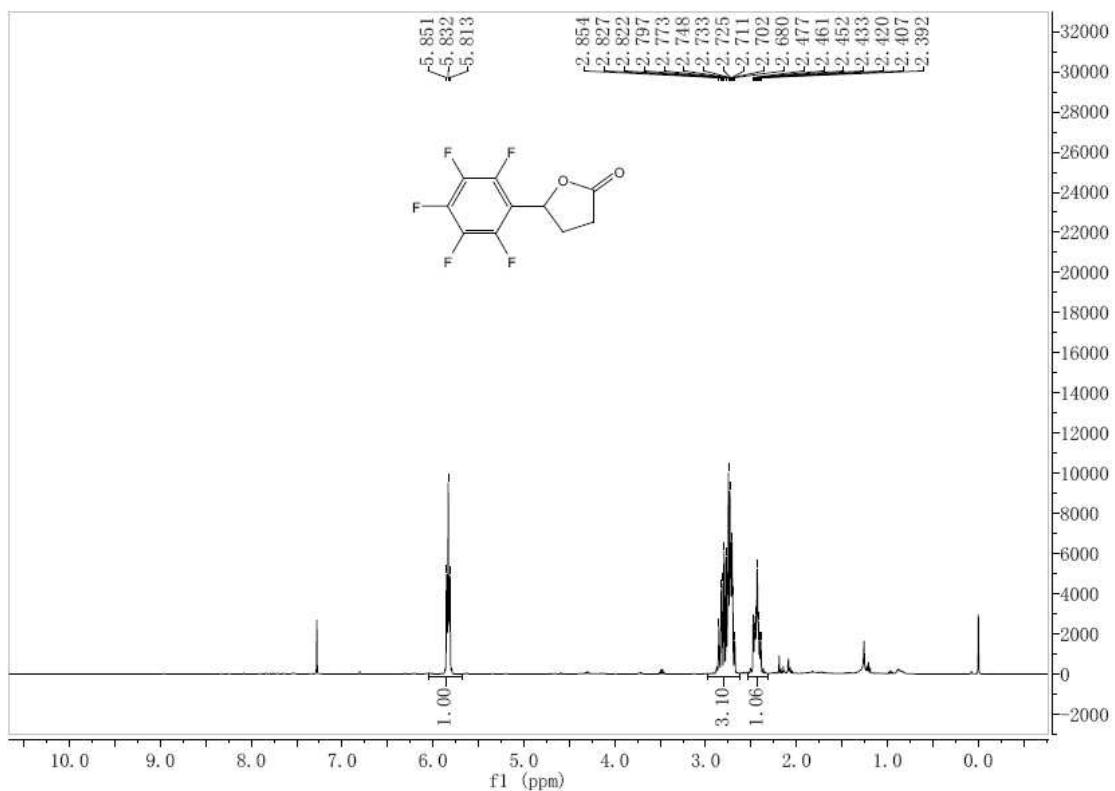
5-Methy-5-phenyl-dihydro-furan-2-one (2i)



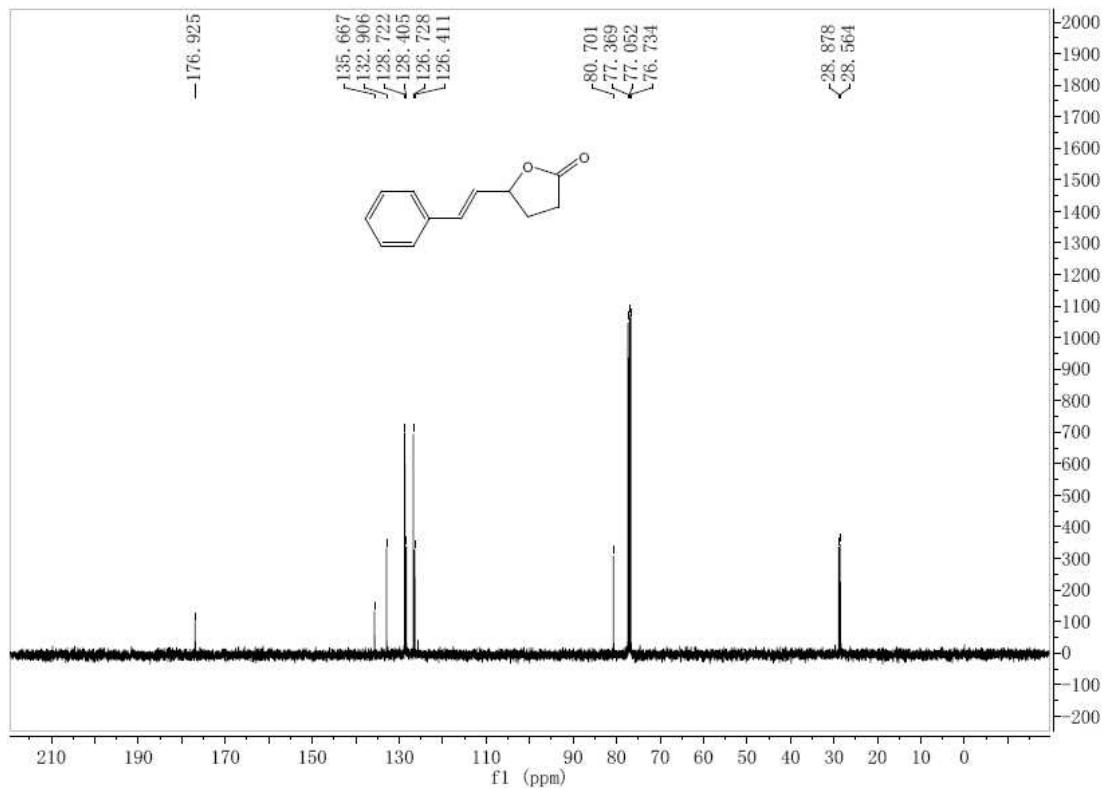
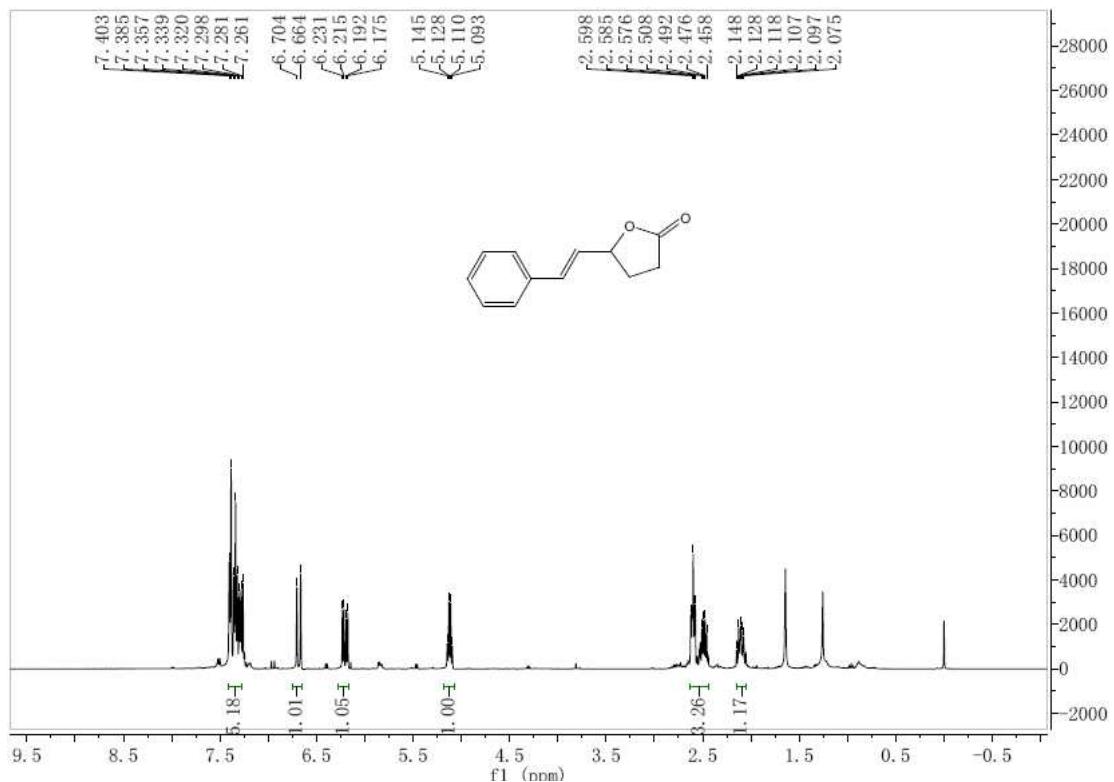
5-Naphthalen-2-yl-dihydro-furan-2-one (2j)



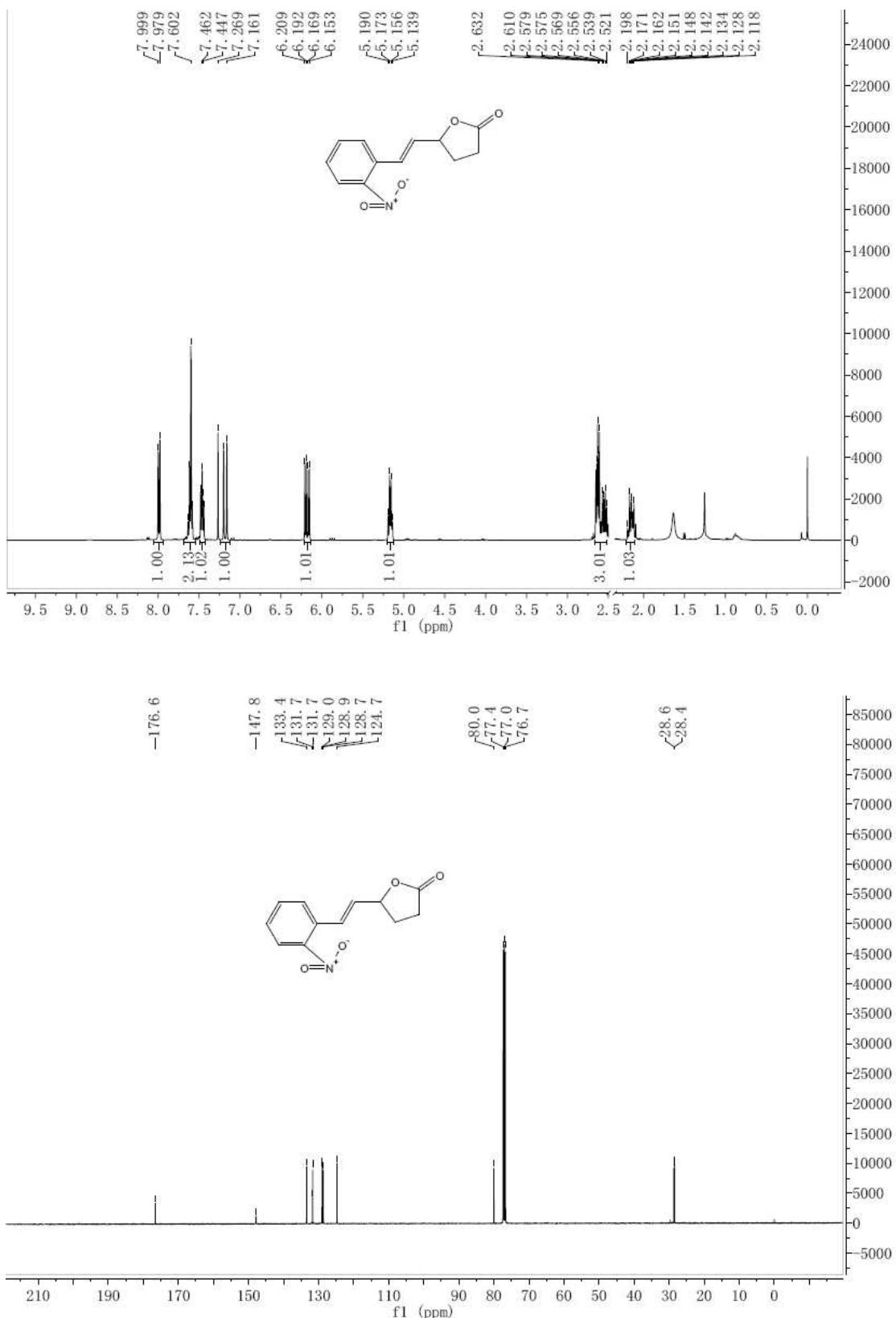
5-Pentafluorophenyl-dihydro-furan-2-one (2k)



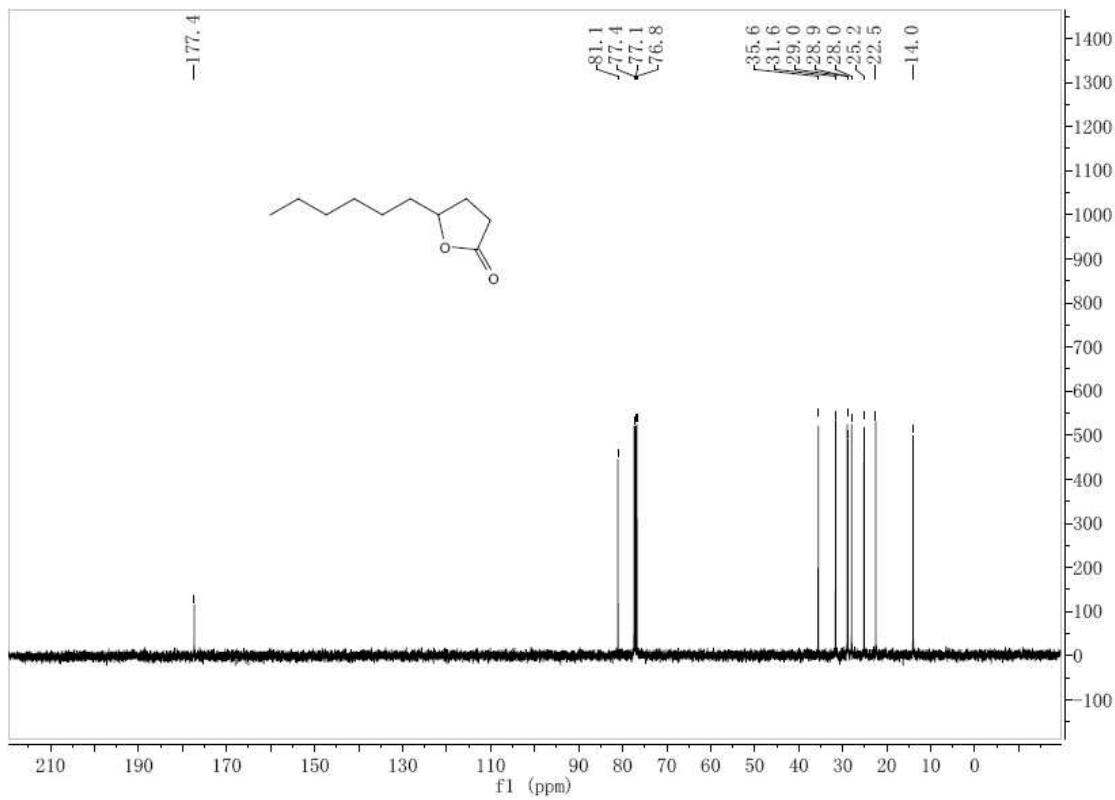
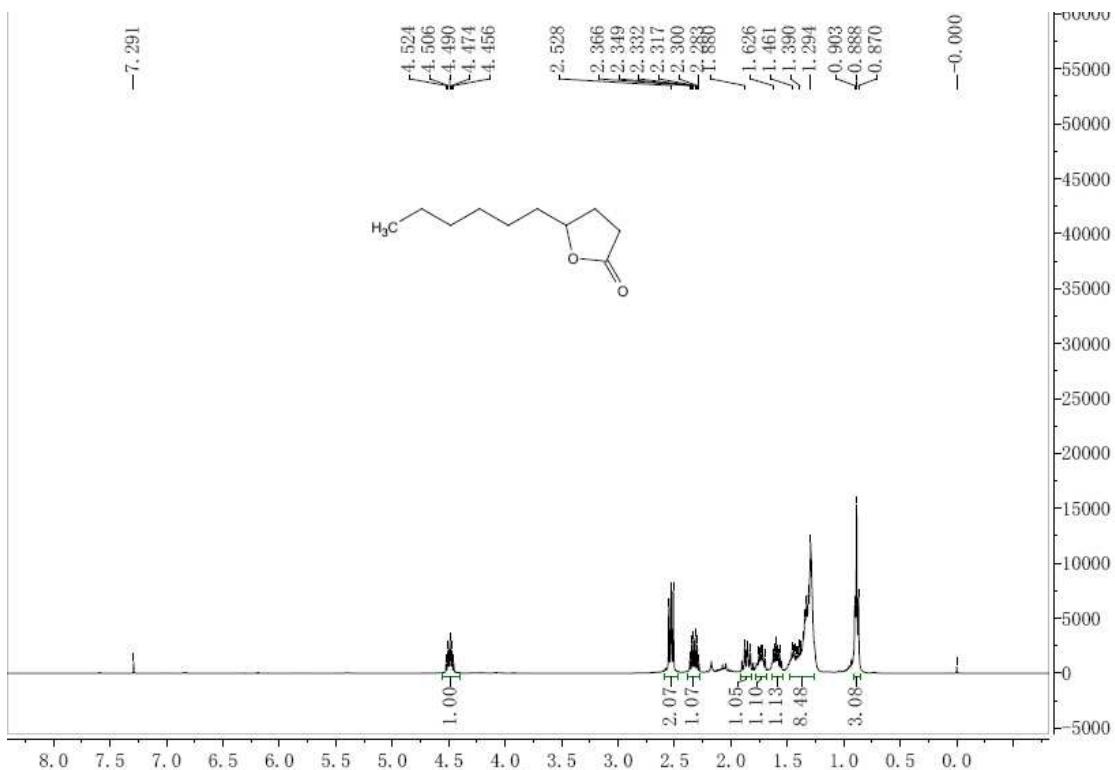
5-Styryl-dihydro-furan-2-one (2l)



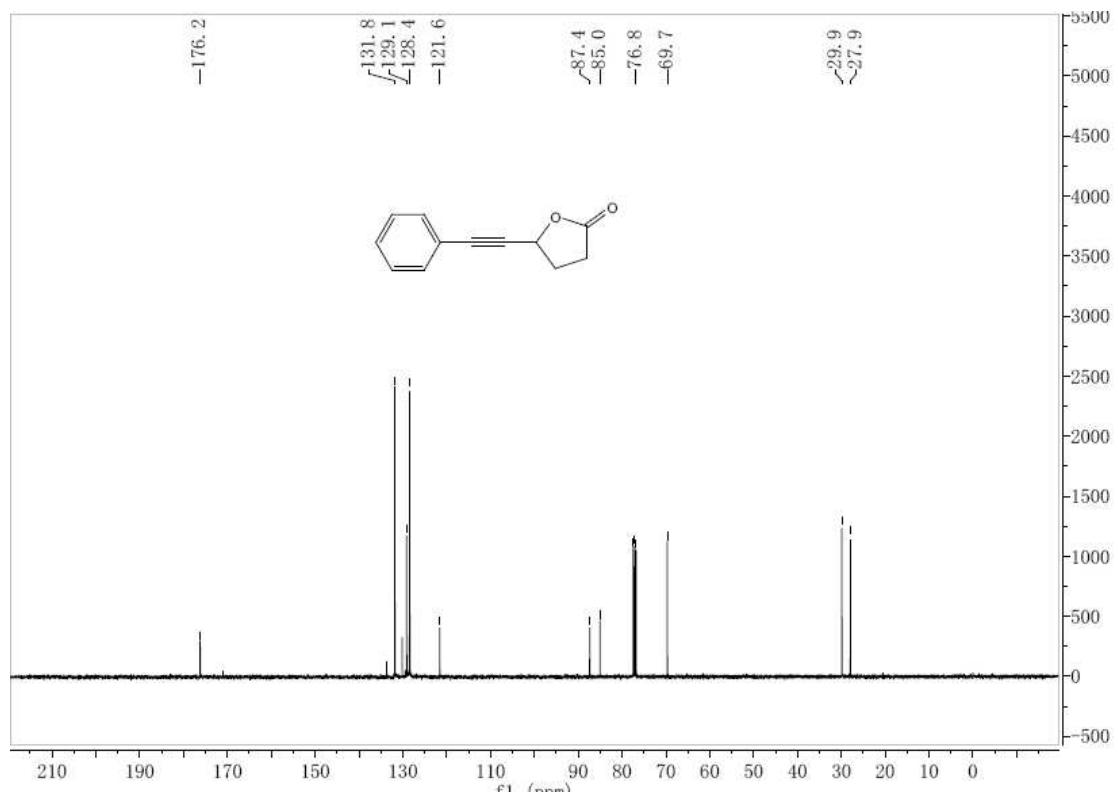
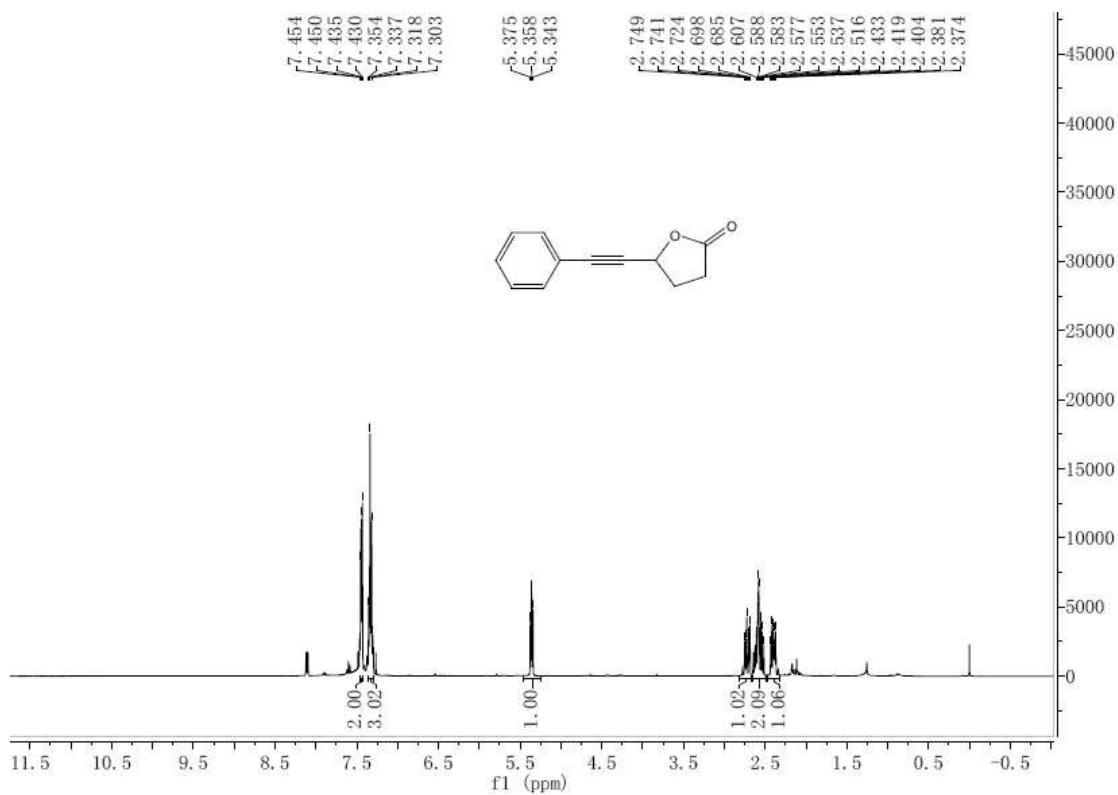
5-[2-(2-Nitro-phenyl)-vinyl]-dihydro-furan-2-one (2m)



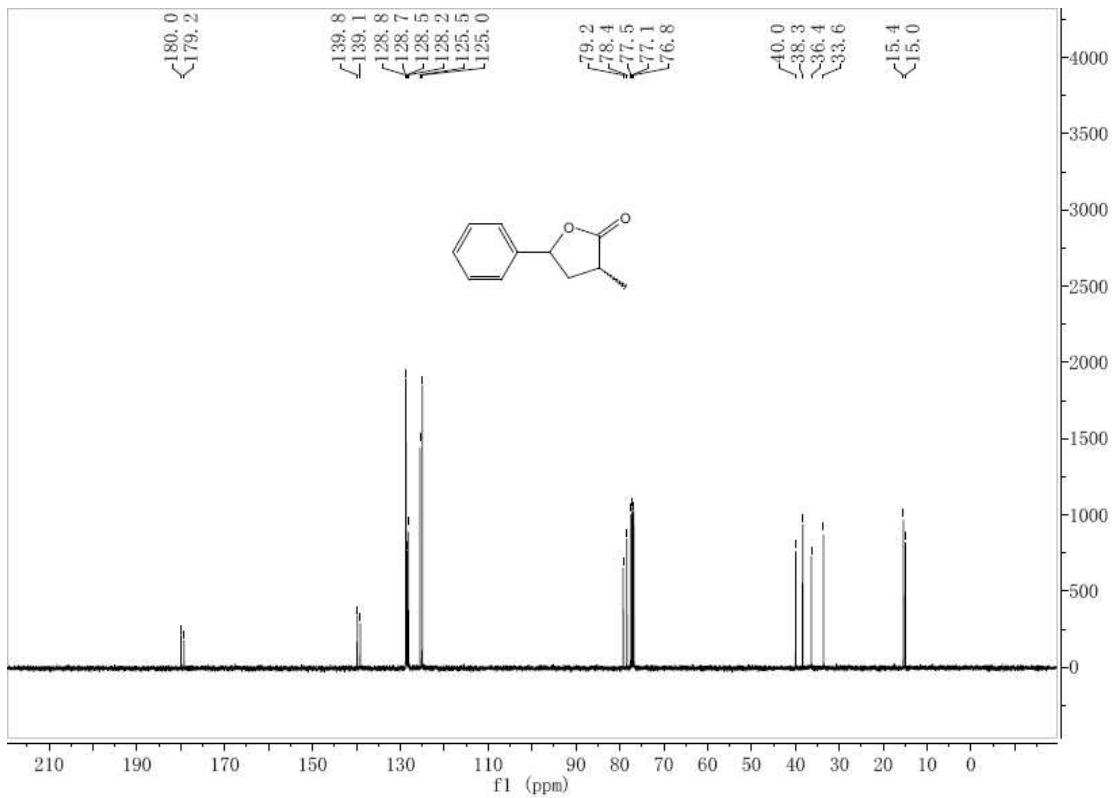
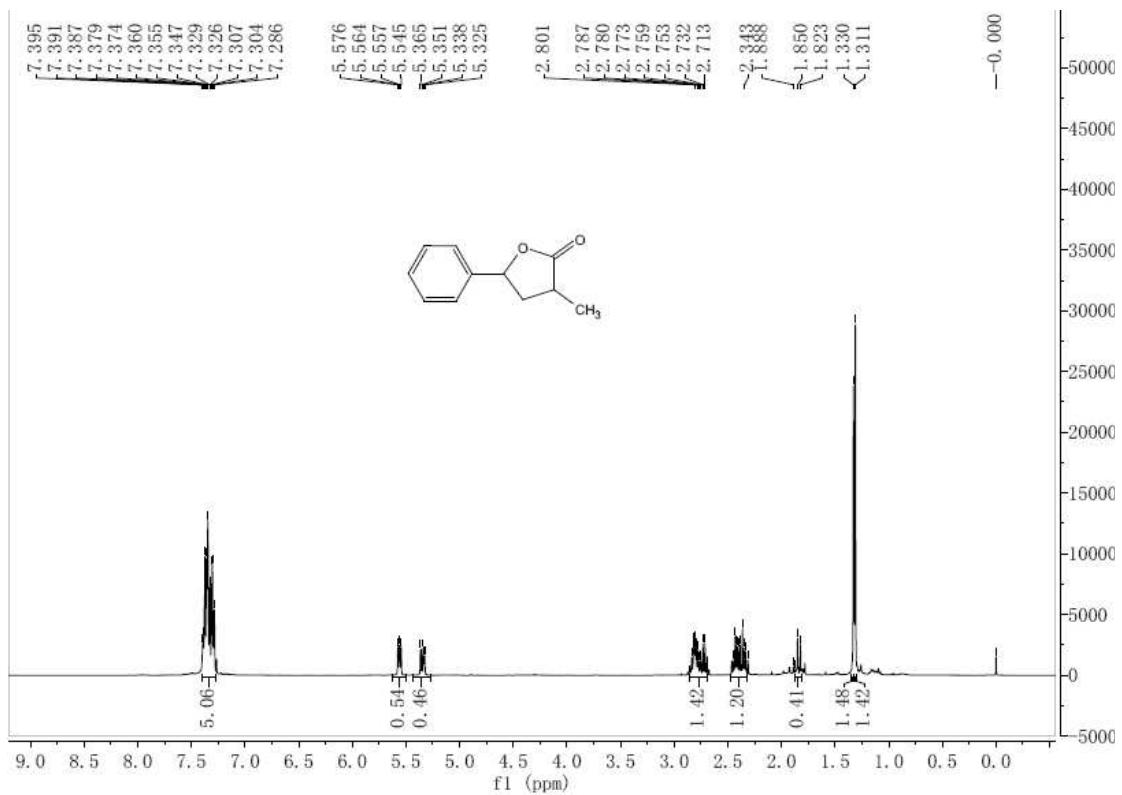
5-Hexyl-dihydro-furan-2-one (2n)



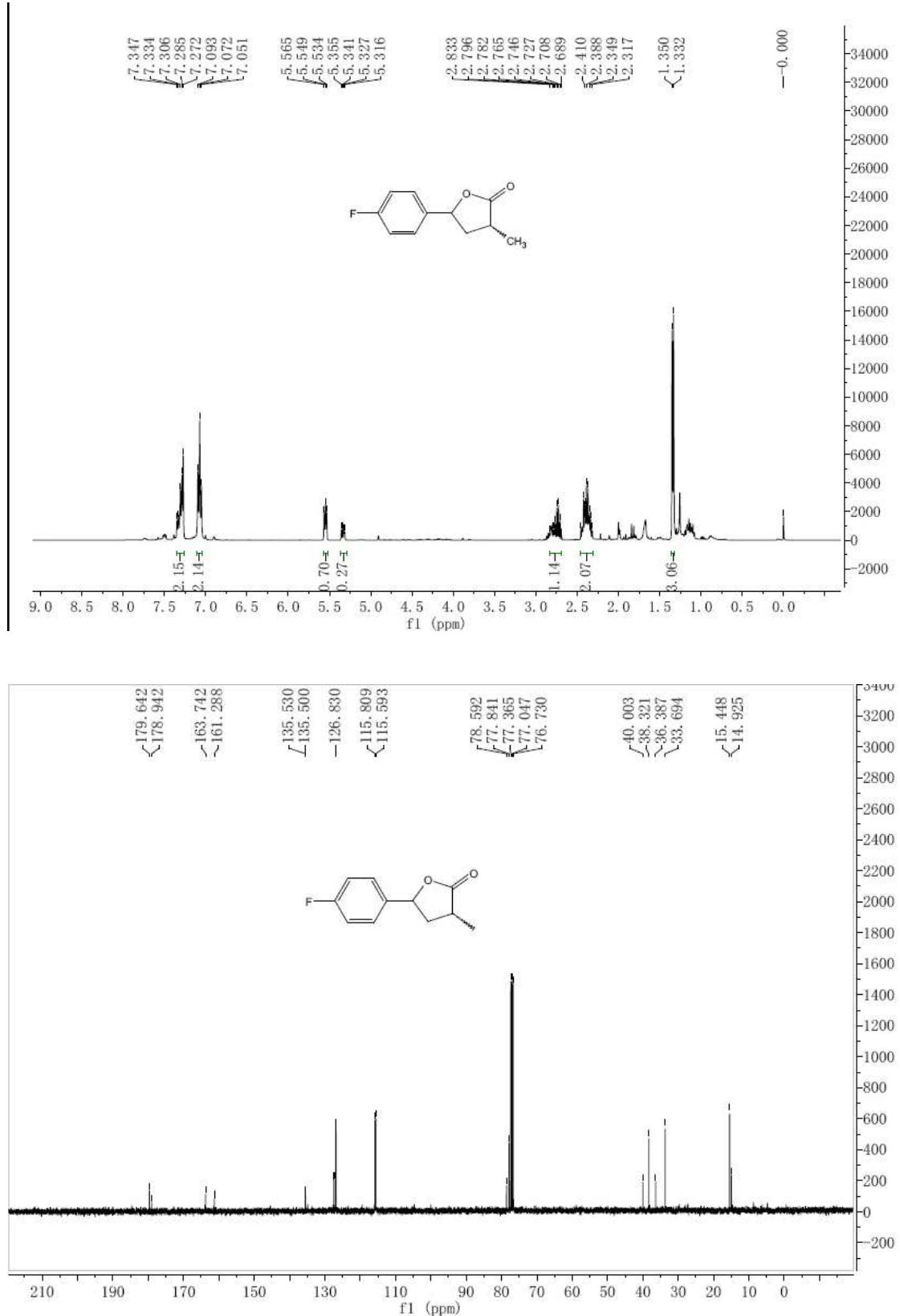
5-Phenylethynyl-dihydro-furan-2-one (2o)



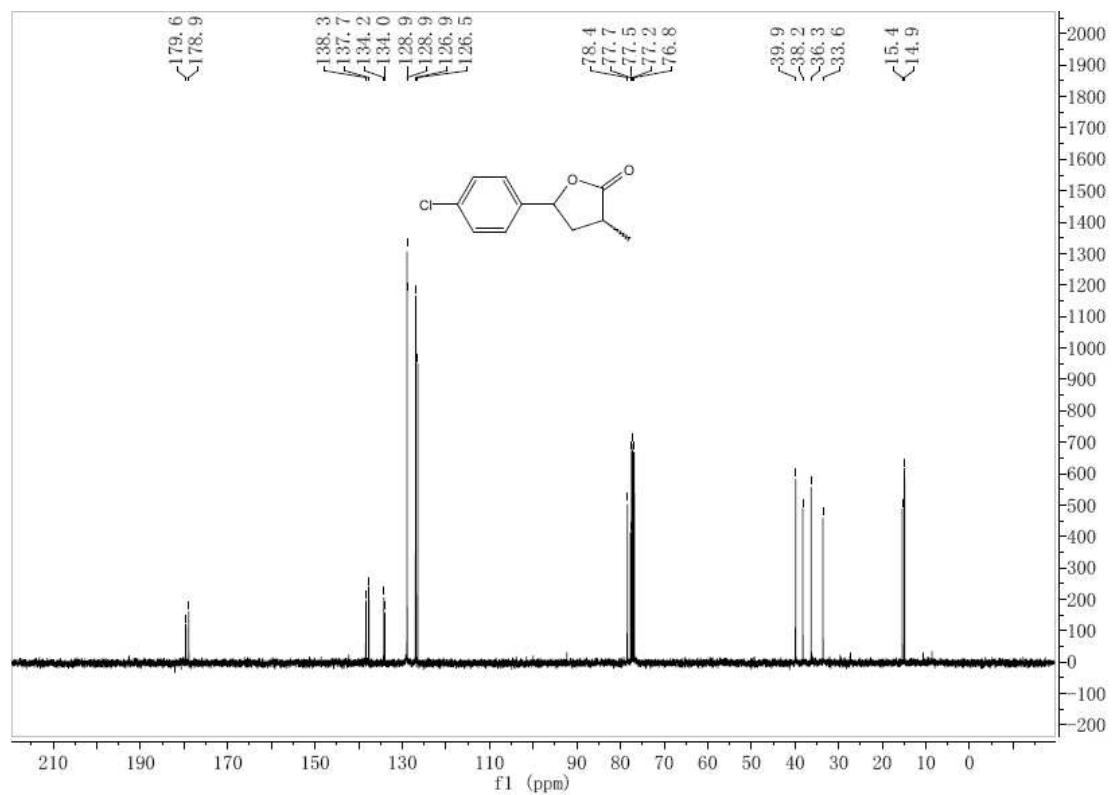
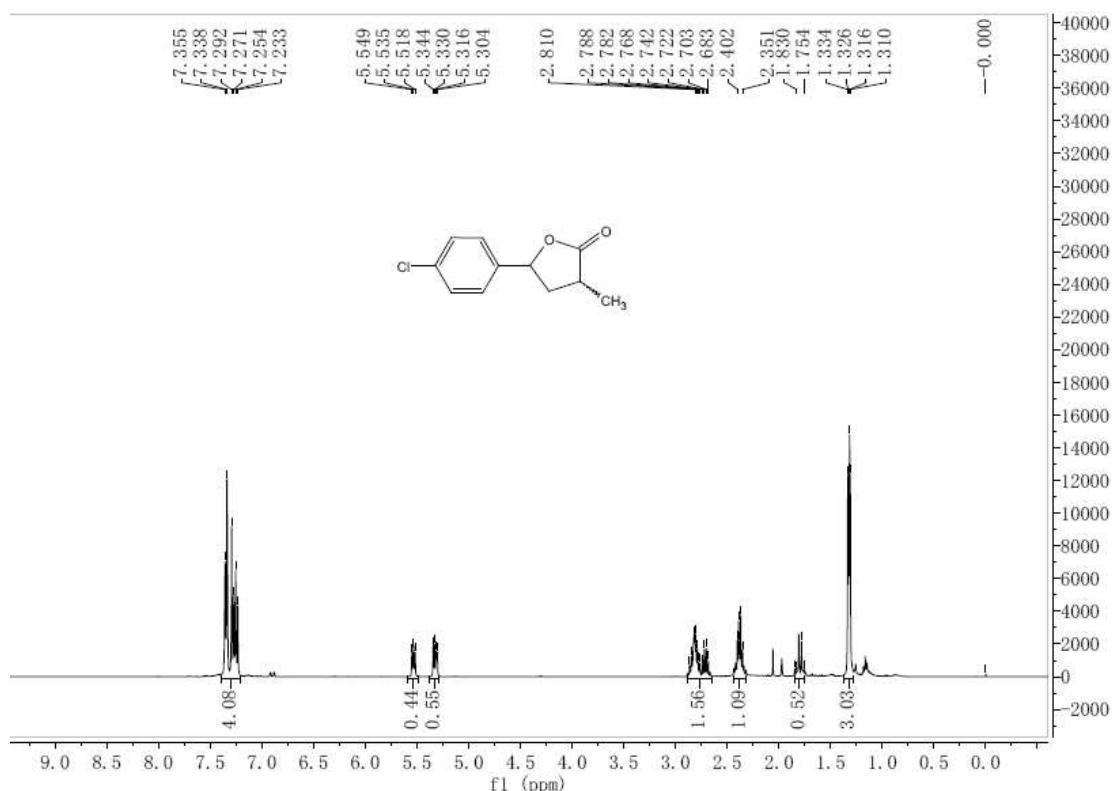
3-Methyl-5-phenyl-dihydro-furan-2-one (3a)



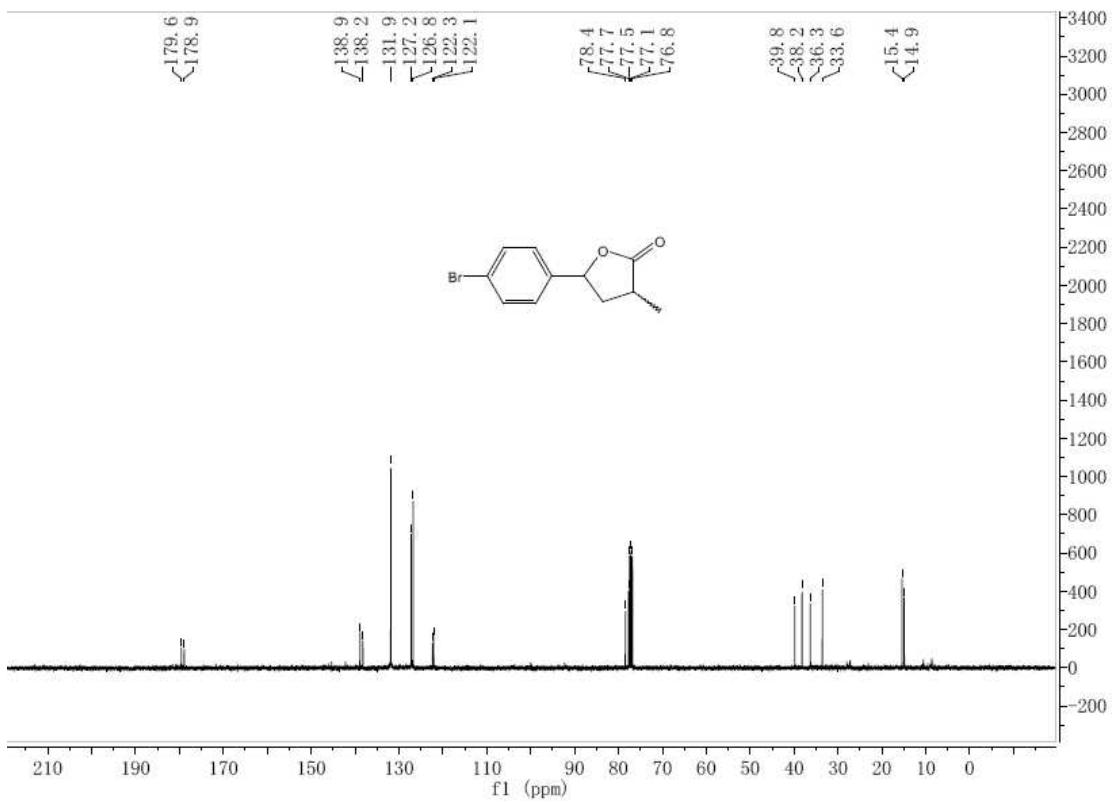
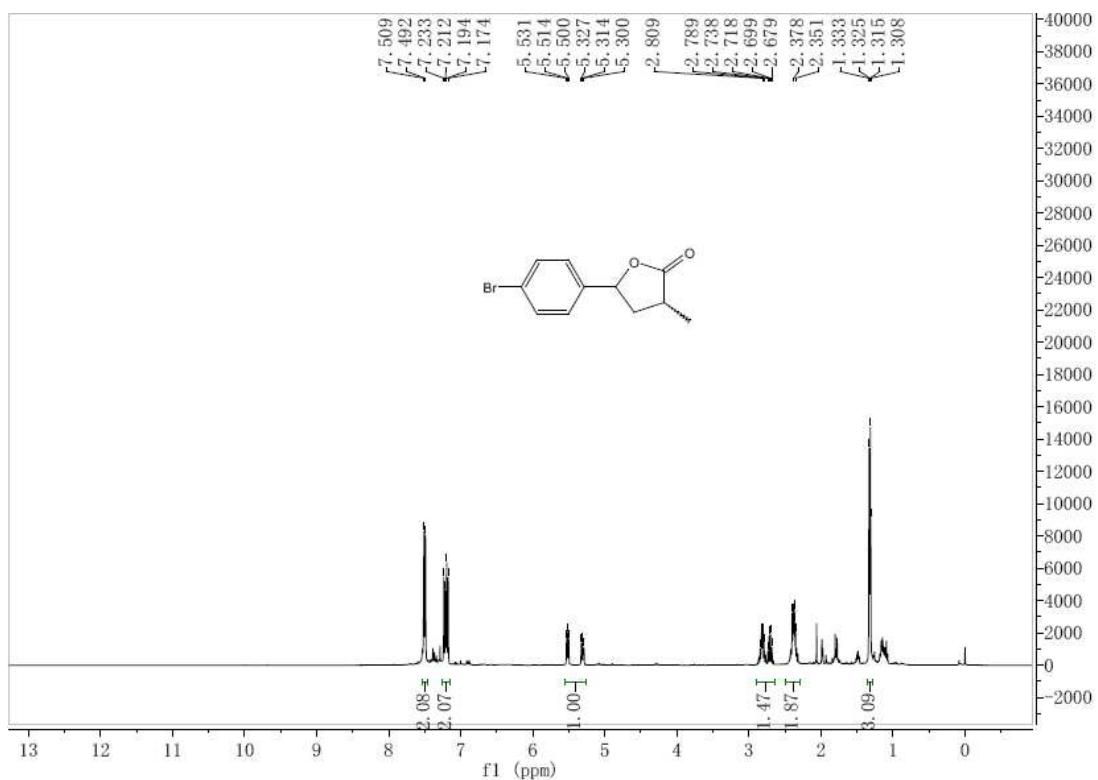
5-(4-Fluoro-phenyl)-3-methyl-dihydro-furan-2-one (3b)



5-(4-Chloro-phenyl)-3-methyl-dihydro-furan-2-one (3c)



5-(4-Bromo-phenyl)-3-methyl-dihydro-furan-2-one (3d)



Dihydro -3-methyl-5-p-tolylfuran-2-(3H)-one (3e)

