

Supporting Information

The Pyridine-Catalyzed Ring-Opening Reaction of Cyclopropenone with Bromomethyl Carbonyl Compounds toward Furan-2(5*H*)-ones

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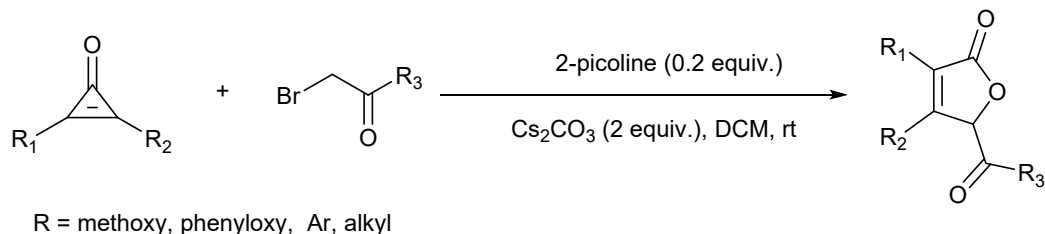
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1. General Information

All reagents and solvents were purchased from TCI, Sigma-Aldrich, Alfa Aesar, Acros and Meryer. All reactions were conducted using standard Schlenk techniques. Column chromatography was performed using EM silica gel 60 (300 – 400 mesh). ^1H NMR and ^{13}C NMR spectra were measured on a 400 MHz Bruker AVANCE spectrometer, using $\text{DMSO-}d_6$ or CDCl_3 as the solvent with tetramethylsilane (TMS) as the internal standard at room temperature. Chemical shifts were reported in ppm. ^1H NMR spectra were referenced to CDCl_3 (7.26 ppm) or $\text{DMSO-}d_6$ (2.50 ppm), and ^{13}C NMR spectra were referenced to CDCl_3 (77.0 ppm) or $\text{DMSO-}d_6$ (39.5 ppm). Peak multiplicities were designated by the following abbreviations: s, singlet; d, doublet; t, triplet; m, multiplet; q, quartet. Chemical shifts are given in δ relative to TMS, the coupling constants J are given in Hz. Analysis of crude reaction mixture was done on the Varian 4000 GC/MS and Agilent 7890A/5975C. High-resolution mass spectra were recorded on a microTOF-Q II 10410 mass spectrometer. Unless otherwise noted, all reagents and solvents were obtained commercially and used without further purification. The cyclopropenones¹ were prepared according to corresponding literature procedures.

2. General Experimental Procedures

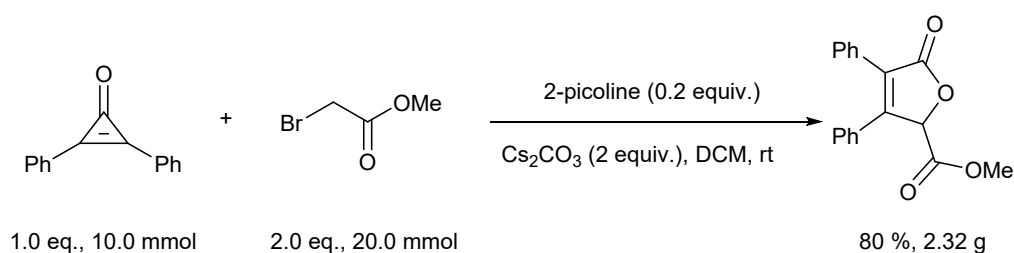
(1) General procedure (3aa-3ao, 3bl-3fl)



Scheme S1

A 10 mL pressure tube equipped with a stir bar was charged with cyclopropenones (0.1 mmol, 1.0 equiv.), bromomethyl carbonyl compounds (0.2 mmol, 2.0 equiv.), 2-picoline (0.02 mmol, 0.2 equiv.), Cs₂CO₃ (0.2 mmol, 2.0 equiv.) and DCM (2 mL). The reaction mixture was stirred at room temperature under air atmosphere for 16 h. After completion of the reaction, the reaction mixture was concentrated under reduced pressure, then 2.0 ml of water was added, and the reaction solution was extracted with ethyl acetate (3 x 2 mL), the combined organic layers were dried over anhydrous magnesium sulfate, filtered and evaporated under reduced pressure. The residue was then purified by flash chromatography on silica gel to provide the corresponding product.

(2) Procedure for 10.0 mmol scale

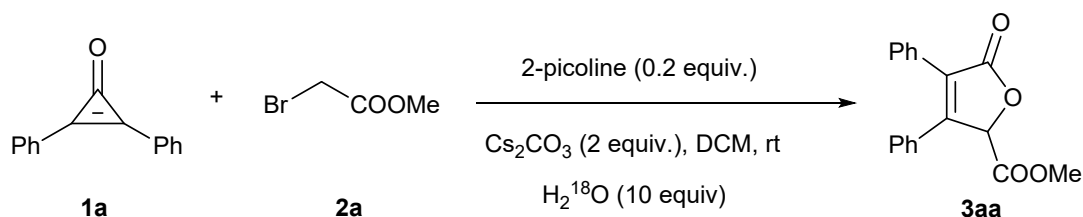


Scheme S2

A 150 mL pressure tube equipped with a stir bar was charged with cyclopropenones (10.0 mmol), bromomethyl carbonyl compounds (20.0 mmol, 2.0 equiv.), 2-picoline (0.2 equiv.), Cs₂CO₃ (2.0 equiv.) and DCM (2 mL). The reaction mixture was stirred at room temperature under air atmosphere for 16 h. After completion of the reaction, the reaction mixture was concentrated under reduced

pressure, then 30 ml of water was added, and the reaction solution was extracted with ethyl acetate (3 x 30 mL), the combined organic layers were dried over anhydrous magnesium sulfate, filtered and evaporated under reduced pressure. The remaining residue was purified by column chromatography on silica gel to afford **3aa** (2.32 g, 80%).

3. Mechanism study

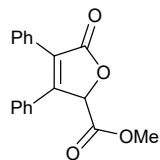


Scheme S3

A 10 mL pressure tube equipped with a stir bar was charged with 1,2-diphenylcyclopropenone (0.1 mmol, 1.0 equiv.), methyl bromoacetate (0.2 mmol, 2.0 equiv.), 2-picoline (0.02 mmol, 0.2 equiv.), Cs₂CO₃ (0.2 mmol, 2.0 equiv.), H₂¹⁸O (1mmol, 10 equiv.) and DCM (2 mL). The reaction mixture was stirred at room temperature under N₂ atmosphere for 12 h. After completion of the reaction, the reaction mixture was concentrated under reduced pressure. The residue was then purified by flash chromatography on silica gel to provide the corresponding product **3aa** (25.0 mg, 85%).

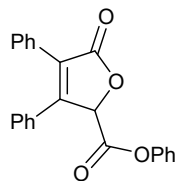
4. Characterization of Products in Details

methyl 5-oxo-3,4-diphenyl-2,5-dihydrofuran-2-carboxylate (**3aa**)



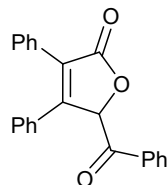
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a yellow oil (24.1 mg, 82%), ¹H NMR (400 MHz, chloroform-d) δ 7.27 – 7.20 (m, 6H), 7.17 – 7.14 (m, 4H), 5.57 (s, 1H), 3.96 (s, 3H). ¹³C NMR (126 MHz, chloroform-d) δ 162.82, 161.25, 157.21, 138.30, 134.13, 131.11, 128.68, 128.55, 128.24, 127.92, 127.18, 115.43, 84.66, 56.08. HRMS: (ESI) calculated for C₁₈H₁₅O₄ [M+H]⁺ 295.0965, found 295.0969.

phenyl 5-oxo-3,4-diphenyl-2,5-dihydrofuran-2-carboxylate (3ab)



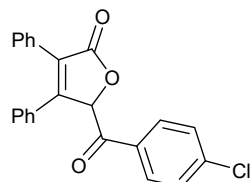
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a white solid (30.3 mg, 85%), Melting Point: 128.4 – 129.1 °C. **¹H NMR** (400 MHz, chloroform-d) δ 7.49 (t, $J = 7.7$ Hz, 2H), 7.35 – 7.34 (m, 1H), 7.32 – 7.30 (m, 2H), 7.28 – 7.25 (m, 5H), 7.24 – 7.22 (m, 3H), 7.10 (d, $J = 7.3$ Hz, 2H), 5.52 (s, 1H). **¹³C NMR** (126 MHz, chloroform-d) δ 161.79, 161.18, 156.38, 152.29, 137.88, 133.93, 131.09, 130.36, 128.84, 128.60, 128.31, 128.03, 127.45, 126.52, 120.54, 117.36, 87.65. HRMS: (ESI) calculated for C₂₃H₂₇O₄ [M+H]⁺ 357.1121, found 357.1120.

5-benzoyl-3,4-diphenylfuran-2(5H)-one (3ac)



Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a yellow solid (24.5 mg, 72%), Melting Point: 181.1 – 182.6 °C. **¹H NMR** (400 MHz, chloroform-d) δ 7.96 – 7.94 (m, 2H), 7.52 – 7.50 (m, 3H), 7.31 – 7.30 (m, 3H), 7.28 – 7.25 (m, 5H), 7.23 – 7.22 (m, 2H), 6.89 (s, 1H). **¹³C NMR** (126 MHz, chloroform-d) δ 162.73, 158.30, 152.81, 137.84, 133.89, 131.41, 130.94, 130.81, 129.01, 128.80, 128.75, 128.44, 128.02, 127.74, 125.64, 123.14, 105.05. HRMS: (ESI) calculated for C₂₃H₁₇O₃ [M+H]⁺ 341.1172, found 341.1167.

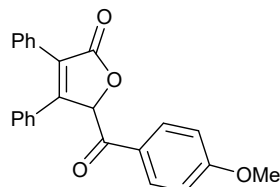
5-(4-chlorobenzoyl)-3,4-diphenylfuran-2(5H)-one (3ad)



Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a yellow oil (23.6 mg, 63%), **¹H NMR** (400 MHz, chloroform-d) δ 7.88 (d, $J = 8.7$ Hz, 2H), 7.48 (d, $J = 8.6$ Hz, 2H), 7.31 – 7.29 (m, 3H), 7.28 – 7.23 (m, 5H), 7.21 – 7.19 (m, 2H), 6.86 (s, 1H). **¹³C NMR** (126 MHz, chloroform-d) δ 162.44, 157.09,

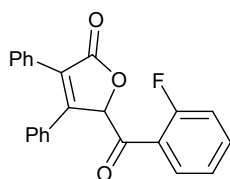
152.63, 137.66, 136.90, 133.70, 130.87, 129.88, 129.30, 128.86, 128.70, 128.46, 128.03, 127.84, 126.87, 123.48, 105.22. HRMS: (ESI) calculated for $C_{23}H_{15}ClO_3Na$ $[M+Na]^+$ 397.0602, found 397.0602.

5-(4-methoxybenzoyl)-3,4-diphenylfuran-2(5H)-one (3ae)



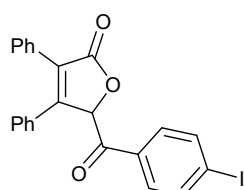
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a yellow oil (25.9 mg, 70%), 1H NMR (500 MHz, chloroform-d) δ 7.85 (d, J = 8.5 Hz, 2H), 7.25 – 7.22 (m, 3H), 7.20 – 7.17 (m, 5H), 7.16 – 7.14 (m, 2H), 6.97 (d, J = 8.5 Hz, 2H), 6.72 (s, 1H), 3.86 (s, 3H). ^{13}C NMR (126 MHz, chloroform-d) δ 162.90, 161.78, 158.51, 153.16, 138.06, 134.03, 130.96, 128.71, 128.66, 128.36, 127.95, 127.57, 127.33, 123.97, 121.94, 114.41, 103.60, 55.51. HRMS: (ESI) calculated for $C_{24}H_{19}O_4$ $[M+H]^+$ 371.1278, found 371.1278.

5-(2-fluorobenzoyl)-3,4-diphenylfuran-2(5H)-one (3af)



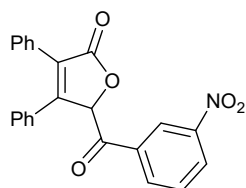
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a yellow solid (26.9 mg, 75%), Melting Point: 106.3 – 107.2 °C. 1H NMR (500 MHz, chloroform-d) δ 7.96 (t, J = 7.7 Hz, 1H), 7.32 (q, J = 6.9 Hz, 1H), 7.17 – 7.12 (m, 4H), 7.12 – 7.09 (m, 5H), 7.07 – 7.03 (m, 3H), 6.98 (s, 1H). ^{13}C NMR (126 MHz, chloroform-d) δ 162.43, 160.17 (d, J = 254.7 Hz), 152.74, 137.68, 133.74, 132.01 (d, J = 9.1 Hz), 130.90, 128.82, 128.42, 128.04, 127.84, 124.8 (d, J = 3.8 Hz), 119.7, (d, J = 10.0 Hz), 116.6 (d, J = 22.7 Hz), 110.10 (d, J = 15.6 Hz). ^{19}F NMR (471 MHz, chloroform-d) δ -111.29. HRMS: (ESI) calculated for $C_{23}H_{15}FO_3Na$ $[M+Na]^+$ 381.0897, found 381.0890.

5-(4-iodobenzoyl)-3,4-diphenylfuran-2(5H)-one (3ag)



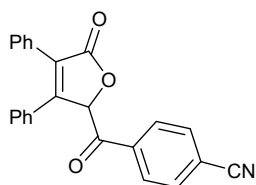
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a yellow solid (31.7 mg, 68%), Melting Point: 153.9 – 154.6 °C. ¹H NMR (500 MHz, chloroform-d) δ 7.70 (d, *J* = 8.2 Hz, 2H), 7.52 (d, *J* = 8.3 Hz, 2H), 7.17 – 7.15 (m, 3H), 7.13 – 7.09 (m, 5H), 7.06 – 7.05 (m, 2H), 6.74 (s, 1H). ¹³C NMR (126 MHz, chloroform-d) δ 162.42, 157.24, 152.58, 138.21, 138.15, 137.63, 133.70, 130.87, 128.88, 128.73, 128.48, 128.05, 127.86, 127.04, 123.66, 105.28, 97.41. HRMS: (ESI) calculated for C₂₃H₁₆IO₃ [M+H]⁺ 467.0139, found 467.0145.

5-(3-nitrobenzoyl)-3,4-diphenylfuran-2(5H)-one (3ah)



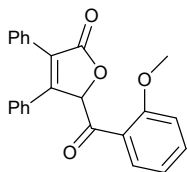
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a yellow solid (22.3 mg, 58%), Melting Point: 176.3 – 176.8 °C. ¹H NMR (500 MHz, chloroform-d) δ 8.62 (s, 1H), 8.22 – 8.20 (d, *J* = 7.5 Hz, 1H), 8.17 – 8.15 (d, *J* = 7.8 Hz, 1H), 7.61 (t, *J* = 8.1 Hz, 1H), 7.21 (m, 3H), 7.17 – 7.14 (m, 3H), 7.11 – 7.08 (m, 4H), 6.88 (s, 1H). ¹³C NMR (126 MHz, chloroform-d) δ 161.96, 155.36, 152.23, 148.79, 137.26, 133.36, 133.13, 131.12, 130.80, 130.26, 129.07, 128.70, 128.56, 128.10, 124.99, 124.73, 120.35, 106.55. HRMS: (ESI) calculated for C₂₃H₁₆NO₅ [M+H]⁺ 386.1023, found 386.1021.

4-(5-oxo-3,4-diphenyl-2,5-dihydrofuran-2-carbonyl)benzonitrile (3ai)



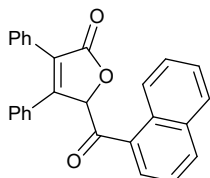
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a green solid (22.3 mg, 61%), Melting Point: 193.2 – 194.5 °C. ¹H NMR (500 MHz, chloroform-d) δ 7.91 (d, *J* = 8.3 Hz, 2H), 7.66 (d, *J* = 8.2 Hz, 2H), 7.20 – 7.13 (m, 6H), 7.12 – 7.06 (m, 4H), 6.86 (s, 1H). ¹³C NMR (126 MHz, chloroform-d) δ 161.99, 155.68, 152.15, 137.29, 135.29, 133.36, 132.76, 130.79, 129.06, 128.68, 128.56, 128.10, 125.97, 124.90, 118.20, 113.92, 107.11. HRMS: (ESI) calculated for C₂₄H₁₅NO₃Na [M+Na]⁺ 388.0944, found 388.0942.

4-(5-oxo-3,4-diphenyl-2,5-dihydrofuran-2-carbonyl)benzonitrile (3aj)



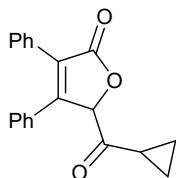
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a green solid (24.8 mg, 67%), Melting Point: 160.7 – 161.1. ¹H NMR (500 MHz, chloroform-d) δ 7.96 (d, *J* = 7.8 Hz, 1H), 7.32 (t, *J* = 7.9 Hz, 1H), 7.21 (s, 1H), 7.17 – 7.14 (m, 3H), 7.13 – 7.10 (m, 5H), 7.09 – 7.07 (m, 2H), 7.00 (t, *J* = 7.6 Hz, 1H), 6.91 (d, *J* = 8.3 Hz, 1H), 3.82 (s, 3H). ¹³C NMR (126 MHz, chloroform-d) δ 163.02, 157.48, 155.33, 153.08, 138.28, 134.10, 131.64, 130.97, 129.21, 128.86, 128.54, 128.31, 127.95, 127.58, 122.91, 121.01, 120.23, 111.51, 110.10, 55.71. HRMS: (ESI) calculated for C₂₄H₁₉O₄ [M+H]⁺ 371.1278, found 371.1279.

5-(1-naphthoyl)-3,4-diphenylfuran-2(5H)-one (3ak)



Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a yellow solid (20.7 mg, 53%), Melting Point: 168.6 – 169.3 °C. ¹H NMR (500 MHz, chloroform-d) δ 8.37 (s, 1H), 7.82 – 7.79 (m, 1H), 7.73 (m, 3H), 7.44 – 7.39 (m, 2H), 7.17 – 7.12 (m, 4H), 7.12 – 7.09 (m, 4H), 7.08 – 7.05 (m, 2H), 6.81 (s, 1H). ¹³C NMR (126 MHz, chloroform-d) δ 162.79, 158.18, 152.85, 137.89, 134.29, 133.91, 133.17, 130.97, 129.06, 128.81, 128.78, 128.46, 128.05, 127.81, 127.78, 127.04, 126.10, 123.19, 122.04, 105.45. HRMS: (ESI) calculated for C₂₇H₁₉O₃ [M+H]⁺ 391.1329, found 391.1332.

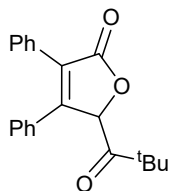
5-(cyclopropanecarbonyl)-3,4-diphenylfuran-2(5H)-one (3al)



Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a yellow solid (26.8 mg, 88%), Melting Point: 133.3 – 134.7 °C. ¹H NMR (500 MHz, chloroform-d) δ 7.10 – 7.03 (m, 6H), 7.03 – 6.99 (m, 2H), 6.96 – 6.95 (m, 2H), 6.10 (s, 1H), 1.72 – 1.67 (m, 1H), 1.05 – 1.04 (m, 2H), 0.87 – 0.85 (m, 2H). ¹³C NMR

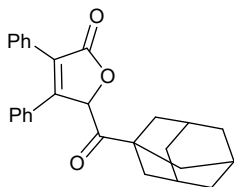
(126 MHz, chloroform-d) δ 164.50, 163.11, 153.09, 137.78, 134.23, 130.96, 128.69, 128.63, 128.29, 127.92, 127.41, 120.98, 105.16, 14.38, 8.32. HRMS: (ESI) calculated for $C_{20}H_{16}O_3Na$ $[M+Na]^+$ 327.0992, found 327.0997.

3,4-diphenyl-5-pivaloylfuran-2(5H)-one(3am)



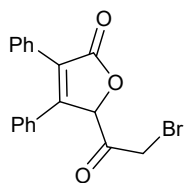
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a white solid (18.5 mg, 58%), Melting Point: 196.4 – 196.8 °C. 1H NMR (500 MHz, chloroform-d) δ 7.22 – 7.19 (m, 6H), 7.18 – 7.13 (m, 2H), 7.13 – 7.08 (m, 2H), 6.19 (s, 1H), 1.35 (s, 9H). ^{13}C NMR (126 MHz, chloroform-d) δ 170.57, 163.34, 152.67, 138.08, 134.06, 130.89, 128.70, 128.57, 128.29, 127.94, 127.52, 122.11, 103.13, 36.13, 28.03. HRMS: (ESI) calculated for $C_{21}H_{21}O_3$ $[M+H]^+$ 321.1485, found 321.1482.

5-(adamantane-1-carbonyl)-3,4-diphenylfuran-2(5H)-one (3an)



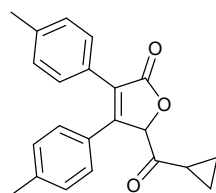
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a white solid (22.7 mg, 57%), Melting Point: 203.6 – 204.3°C. 1H NMR (500 MHz, chloroform-d) δ 7.16 – 7.08 (m, 6H), 7.06 – 7.04 (m, 2H), 7.01 – 6.99 (m, 2H), 6.03 (s, 1H), 2.02 (s, 3H), 1.90 (s, 6H), 1.74 – 1.64 (m, 6H). ^{13}C NMR (126 MHz, chloroform-d) δ 170.54, 163.46, 152.80, 138.16, 134.15, 130.91, 128.73, 128.55, 128.27, 127.93, 127.48, 122.06, 103.16, 39.71, 37.84, 36.51, 28.05. HRMS: (ESI) calculated for $C_{27}H_{27}O_3$ $[M+H]^+$ 399.1955, found 399.1955.

5-(2-bromoacetyl)-3,4-diphenylfuran-2(5H)-one (3ao)



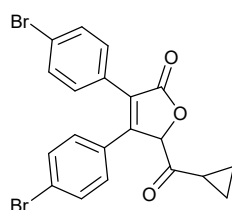
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a white solid (20.0 mg, 56%), Melting Point: 158.6 – 159.9 °C. ¹H NMR (500 MHz, chloroform-d) δ 7.17 – 7.15 (m, 6H), 7.09 – 7.07 (m, 2H), 7.02 (d, *J* = 7.4 Hz, 2H), 6.41 (s, 1H), 4.17 (s, 2H). ¹³C NMR (126 MHz, chloroform-d) δ 162.23, 156.48, 151.71, 136.89, 133.32, 130.72, 129.00, 128.68, 128.45, 128.09, 128.03, 124.90, 108.91, 26.99. HRMS: (ESI) calculated for C₁₈H₁₄BrO₃ [M+H]⁺ 357.0121, found 357.0123.

5-(cyclopropanecarbonyl)-3,4-di-*p*-tolylfuran-2(5*H*)-one(3bl)



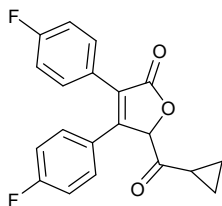
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a yellow oil (39.6 mg, 92%), ¹H NMR (500 MHz, chloroform-d) δ 6.94 – 6.92 (m, 6H), 6.91 – 6.89 (m, 2H), 6.11 (s, 1H), 2.20 (s, 3H), 2.19 (s, 3H), 1.75 – 1.70 (m, 1H), 1.08 – 1.06 (m, 2H), 0.90 – 0.88 (m, 2H). ¹³C NMR (126 MHz, chloroform-d) δ 163.96, 163.43, 152.61, 138.57, 137.02, 134.97, 131.33, 130.71, 128.96, 128.73, 128.65, 120.76, 105.18, 21.29, 21.27, 14.30, 8.12. HRMS: (ESI) calculated for C₂₂H₂₁O₃ [M+H]⁺ 333.1485, found 333.1481.

3,4-bis(4-bromophenyl)-5-(cyclopropanecarbonyl)furan-2(5*H*)-one (3cl)



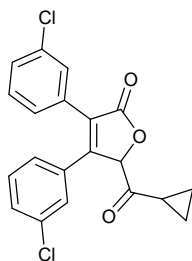
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a yellow solid (38.8 mg, 84%), Melting Point: 171.1 – 171.7 °C. ¹H NMR (500 MHz, chloroform-d) δ 7.29 (d, *J* = 8.3 Hz, 2H), 7.26 (d, *J* = 7.8 Hz, 2H), 6.90 (d, *J* = 8.6 Hz, 2H), 6.88 (d, *J* = 7.7 Hz, 2H), 6.09 (s, 1H), 1.77 – 1.72 (m, 1H), 1.10 – 1.08 (m, 2H), 0.95 – 0.93 (m, 2H). ¹³C NMR (126 MHz, chloroform-d) δ 165.30, 162.54, 152.03, 136.32, 132.77, 132.56, 131.77, 131.34, 130.24, 123.29, 121.90, 119.83, 104.67, 14.46, 8.58. HRMS: (ESI) calculated for C₂₀H₁₄Br₂O₃Na [M+Na]⁺ 482.9202, found 482.9206.

5-(cyclopropanecarbonyl)-3,4-bis(4-fluorophenyl)furan-2(5*H*)-one (3dl)



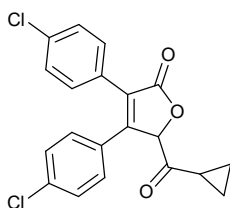
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a green solid (29.6 mg, 87%), Melting Point: 172.3 – 173.0 °C. $^1\text{H NMR}$ (400 MHz, chloroform-d) δ 7.12 – 7.07 (m, 4H), 6.95 – 6.89 (m, 4H), 6.22 (s, 1H), 1.89 – 1.82 (m, 1H), 1.19 – 1.18 (m, 2H), 1.04 – 1.01 (m, 2H). $^{13}\text{C NMR}$ (101 MHz, chloroform-d) δ 164.91, 162.70 (d, $J = 250.9$ Hz), 162.95, 162.00 (d, $J = 248.4$ Hz), 152.23, 133.62 (d, $J = 3.5$ Hz), 132.72 (d, $J = 8.2$ Hz), 130.71 (d, $J = 8.3$ Hz), 129.90 (d, $J = 3.5$ Hz), 119.98, 115.63 (d, $J = 21.2$ Hz), 115.25 (d, $J = 21.6$ Hz), 104.94, 14.39, 8.43. $^{19}\text{F NMR}$ (471 MHz, chloroform-d) δ -111.65, -113.94. HRMS: (ESI) calculated for $\text{C}_{20}\text{H}_{15}\text{F}_2\text{O}_3$ $[\text{M}+\text{H}]^+$ 341.0984, found 341.0990.

3,4-bis(3-chlorophenyl)-5-(cyclopropanecarbonyl)furan-2(5H)-one (3el)



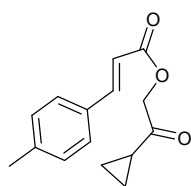
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a green solid (29.8 mg, 80%), Melting Point: 138.5 – 139.6 °C. $^1\text{H NMR}$ (500 MHz, chloroform-d) δ 7.17 – 7.10 (m, 1H), 7.09 – 7.03 (m, 5H), 6.86 (d, $J = 7.7$ Hz, 1H), 6.82 (d, $J = 7.7$ Hz, 1H), 6.10 (s, 1H), 1.78 – 1.73 (m, 1H), 1.11 – 1.10 (m, 2H), 0.96 – 0.94 (m, 2H). $^{13}\text{C NMR}$ (126 MHz, chloroform-d) δ 165.62, 162.40, 152.09, 139.08, 135.48, 134.48, 133.89, 130.84, 129.71, 129.29, 129.13, 129.00, 128.43, 127.92, 126.94, 119.96, 104.56, 14.48, 8.65. HRMS: (ESI) calculated for $\text{C}_{20}\text{H}_{15}\text{Cl}_2\text{O}_3$ $[\text{M}+\text{H}]^+$ 373.0393, found 373.0395.

3,4-bis(4-chlorophenyl)-5-(cyclopropanecarbonyl)furan-2(5H)-one (3fl)



Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a green oil (27.2 mg, 73%), **¹H NMR** (500 MHz, chloroform-d) δ 7.14 (d, J = 8.6 Hz, 2H), 7.11 (d, J = 8.3 Hz, 2H), 6.98 (d, J = 1.9 Hz, 2H), 6.95 (d, J = 8.1 Hz, 2H), 6.10 (s, 1H), 1.79 – 1.73 (m, 1H), 1.12 – 1.10 (m, 2H), 0.96 – 0.95 (m, 2H). **¹³C NMR** (101 MHz, chloroform-d) δ 165.27, 162.69, 152.07, 135.90, 135.02, 133.63, 132.33, 132.28, 130.03, 128.83, 128.43, 119.91, 104.76, 14.48, 8.58. HRMS: (ESI) calculated for C₂₀H₁₅Cl₂O₃ [M+H]⁺ 373.0393, found 373.0394.

2-cyclopropyl-2-oxoethyl (*E*)-3-(*p*-tolyl)acrylate

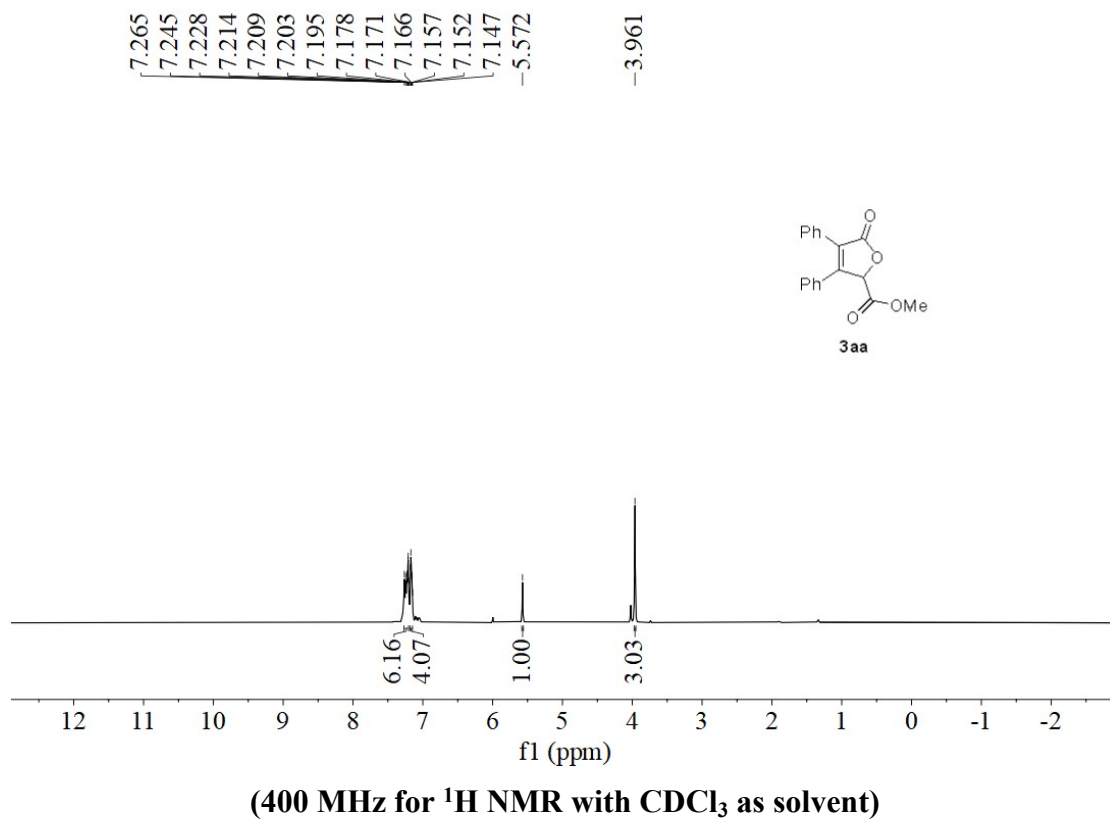


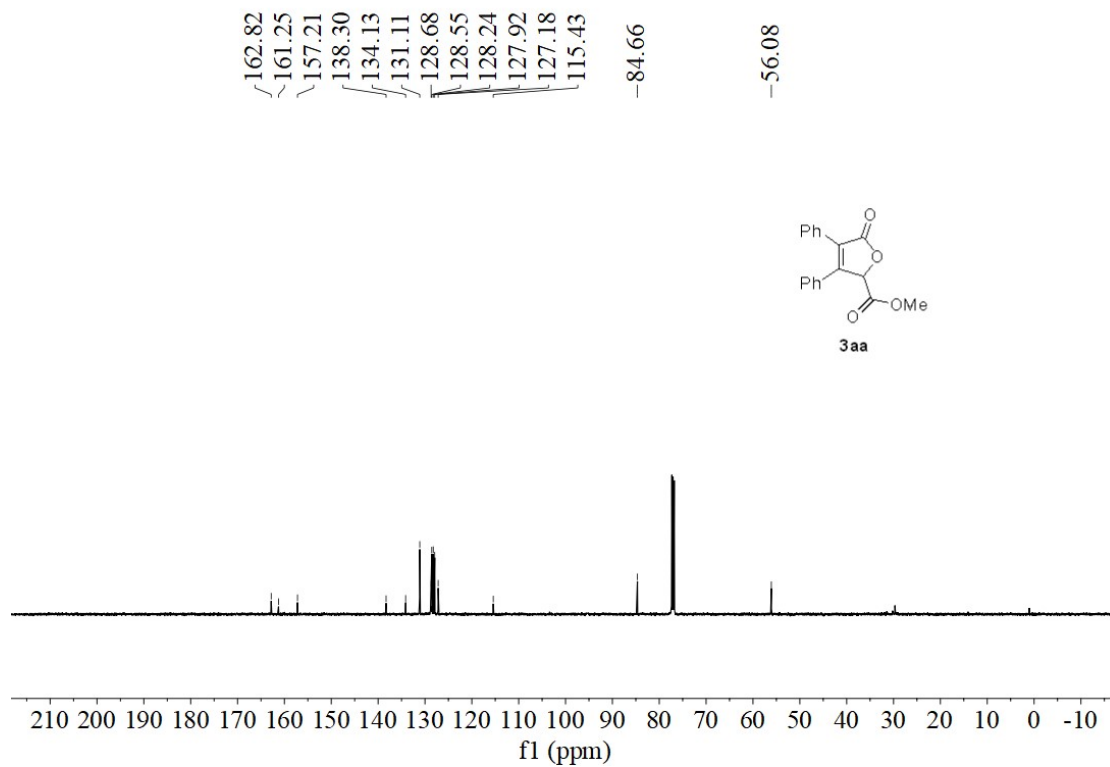
Following the general procedure, using 10/1 petroleum ether/EtOAc as the eluant afford a yellow oil (7.9 mg, 32%), **¹H NMR** (400 MHz, chloroform-d) δ 7.77 – 7.73 (m, 1H), 7.43 (d, J = 7.8 Hz, 2H), 7.20 (d, J = 7.8 Hz, 2H), 6.51 – 6.48 (m, 1H), 4.94 (s, 2H), 2.37 (s, 3H), 2.02 – 1.99 (m, 1H), 1.16 – 1.14 (m, 2H), 1.00 – 0.97 (m, 2H). **¹³C NMR** (126 MHz, chloroform-d) δ 204.12, 166.36, 146.18, 141.07, 131.52, 129.76, 129.35, 128.26, 126.89, 115.82, 106.75, 68.45, 21.50, 17.27, 11.44. HRMS: (ESI) calculated for C₁₅H₁₆O₃Na [M+Na]⁺ 267.0992, found 267.0990.

Reference

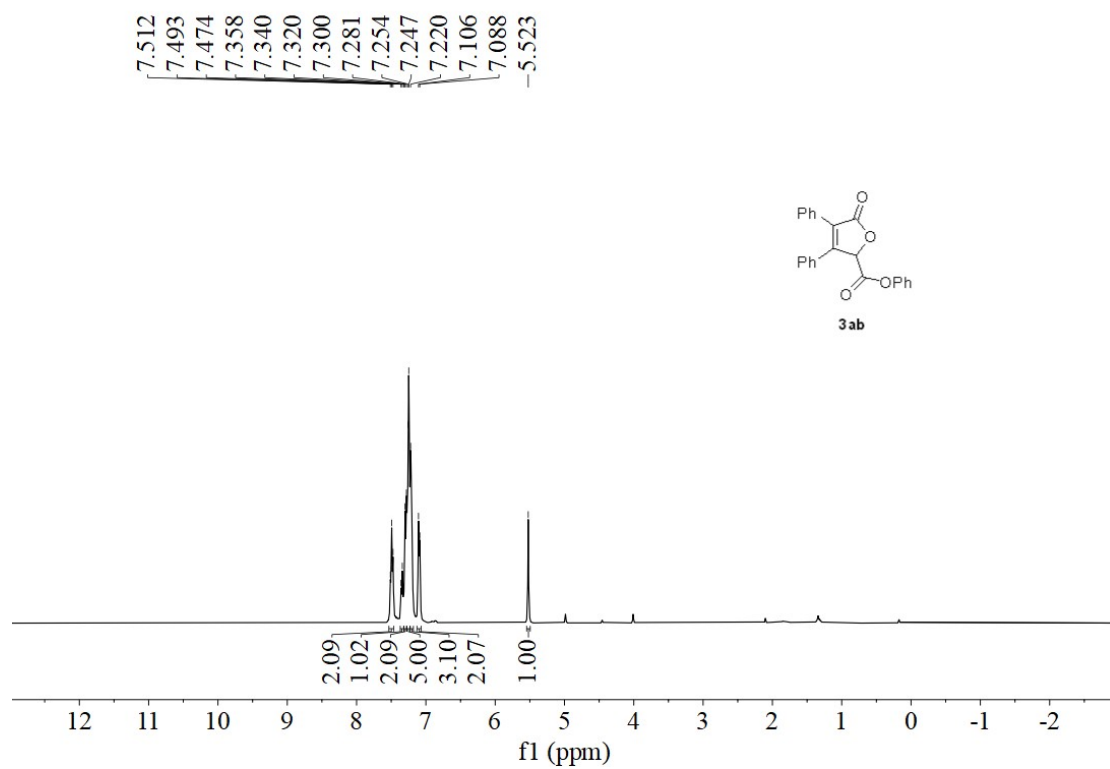
- (a) J. Zhou, X. Shi, H. Zheng, G. Chen, C. Zhang, X. Liu and H. Cao, *Org. Lett.* 2022, **24**, 3238-3243; (b) T. Nanda, P. Biswal, B. V. Patim S. K. Banjare and P. C. Ravikumar, *J. Org. Chem.* 2021, **86**, 2682-2695; (c) J. Chen, B. Tang, X. Liu, G. Lv, Y. Shi, T. Huang, H. Xing, X. Guo, L. Hai and Y. Wu, *Org. Chem. Front.* 2020, **7**, 2944-2949; (d) R. Ando, Y. Morinaka, H. Tokuyama, M. Isaka and E. Nakamura, *J. Am. Chem. Soc.* 1993, **115**, 1174-1175; (e) L. Yao, Q. Hu, L. Bao, W. Zhu and Y. Hu, *Org. Lett.*, 2021, **23**, 4971-4975; (f) W. Miao, W. Gao, X. Huang, M. Liu, Y. Zhou and H. Wu, *Org. Lett.*, 2021, **23**, 9425–9430.

5. NMR Spectra

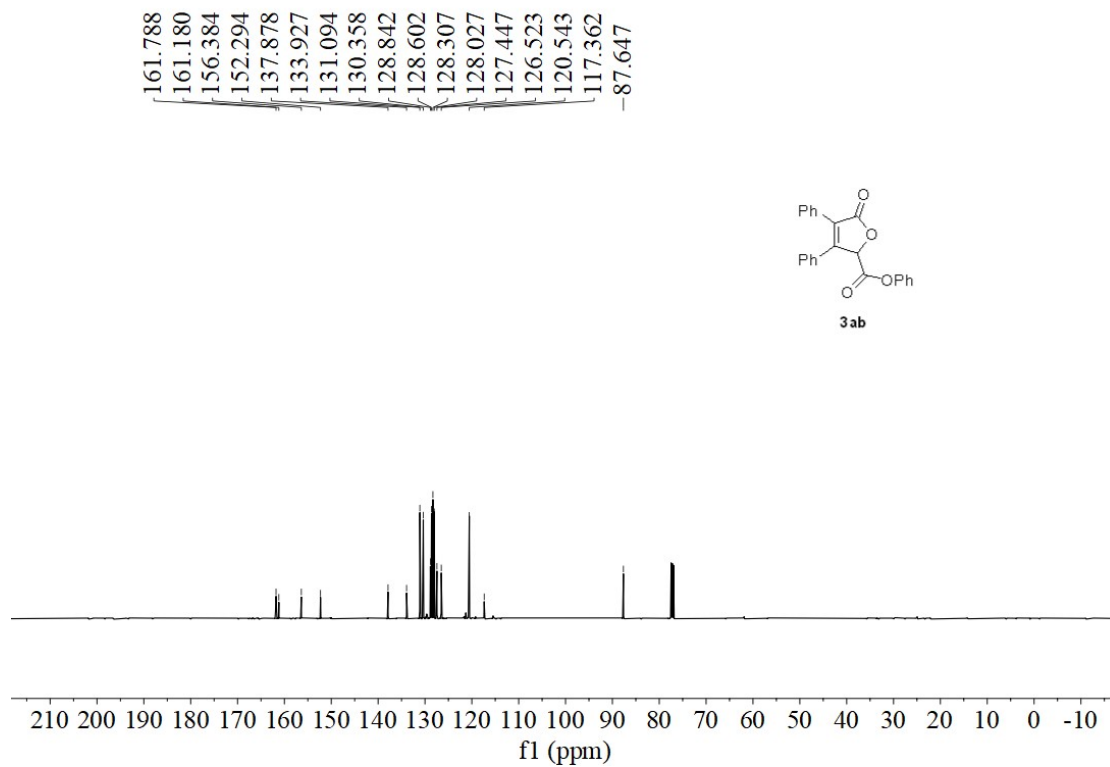




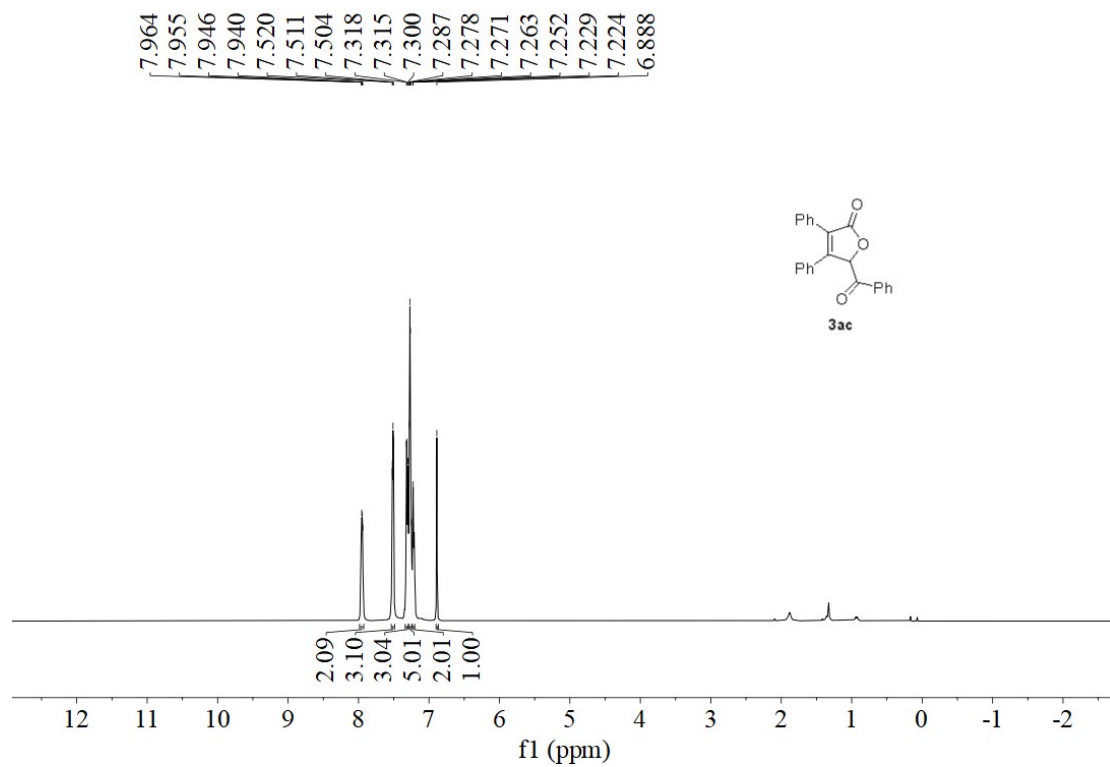
(126 MHz for ^{13}C NMR with CDCl_3 as solvent)



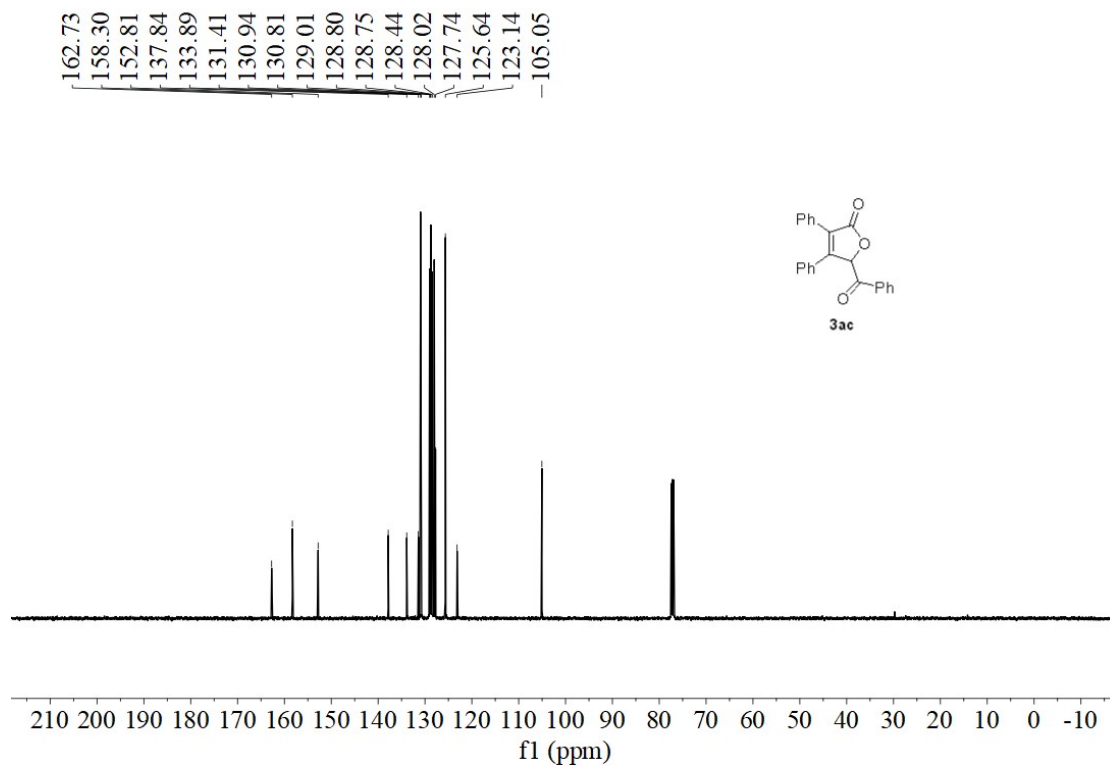
(400 MHz for ^1H NMR with CDCl_3 as solvent)



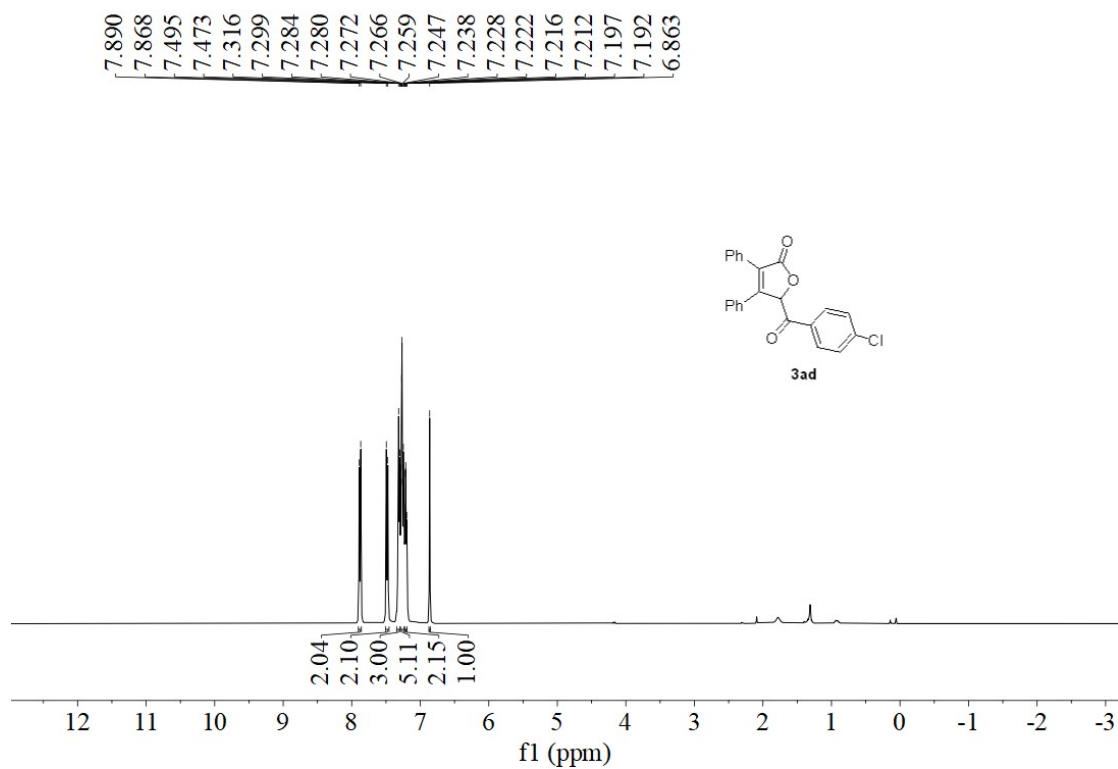
(126 MHz for ^{13}C NMR with CDCl_3 as solvent)



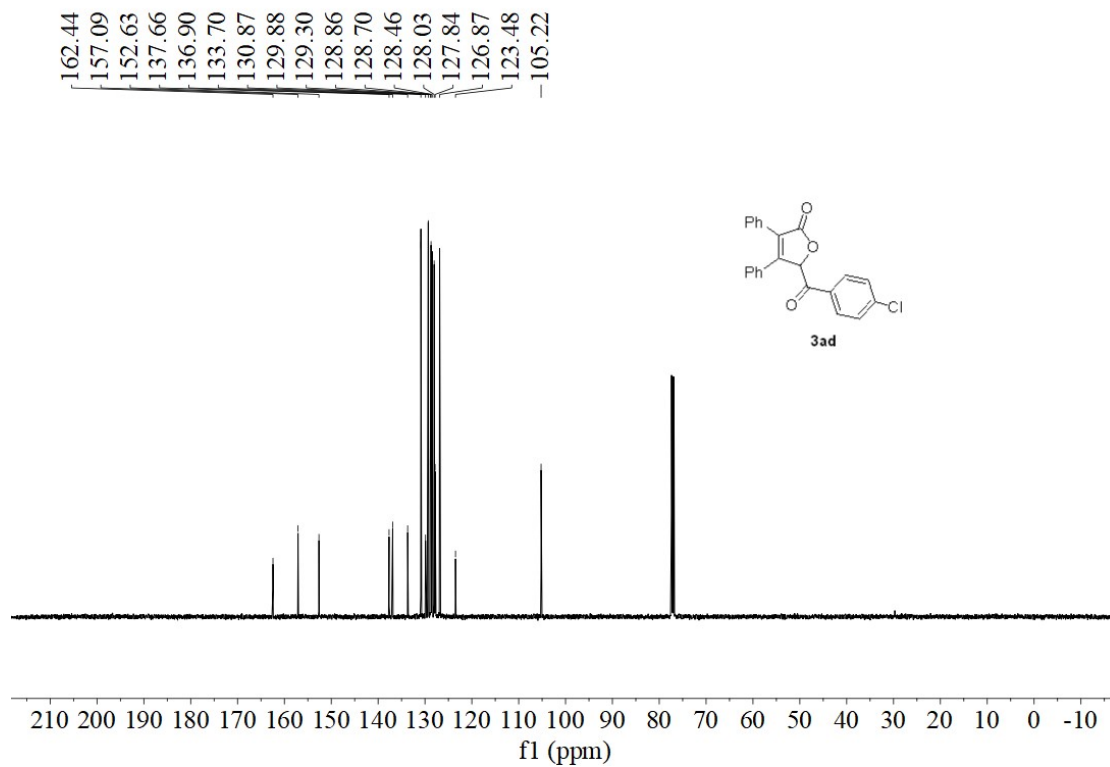
(400 MHz for ^1H NMR with CDCl_3 as solvent)



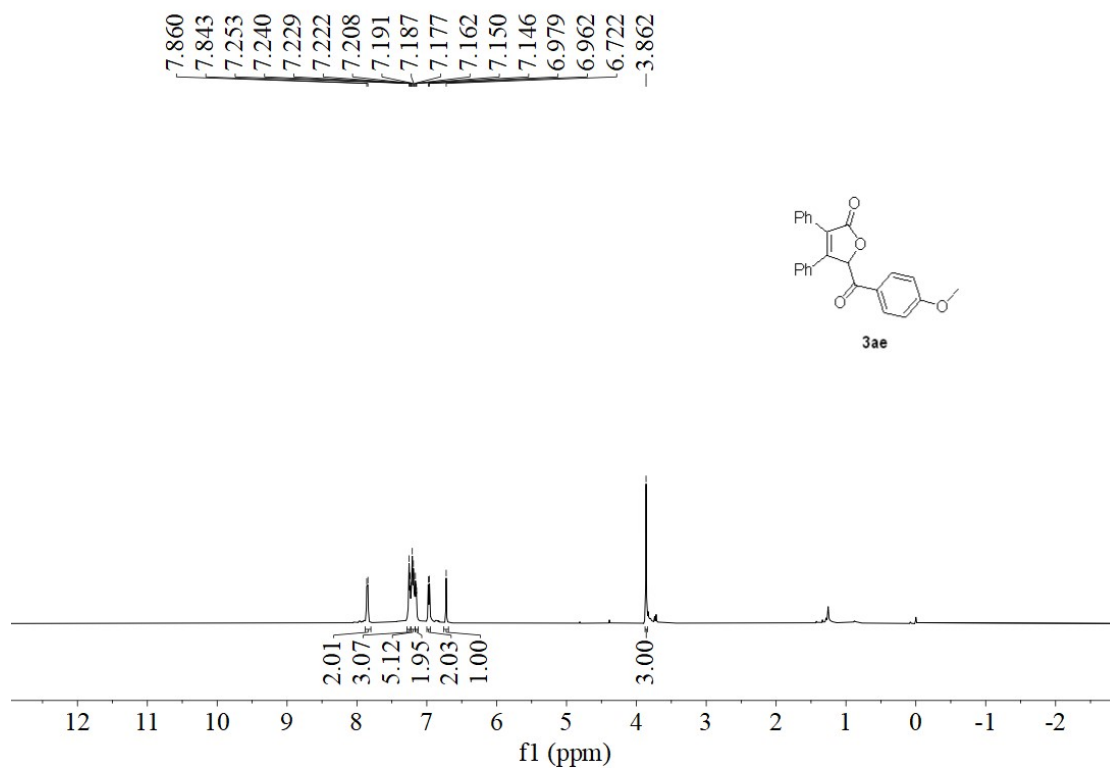
(126 MHz for ^{13}C NMR with CDCl_3 as solvent)



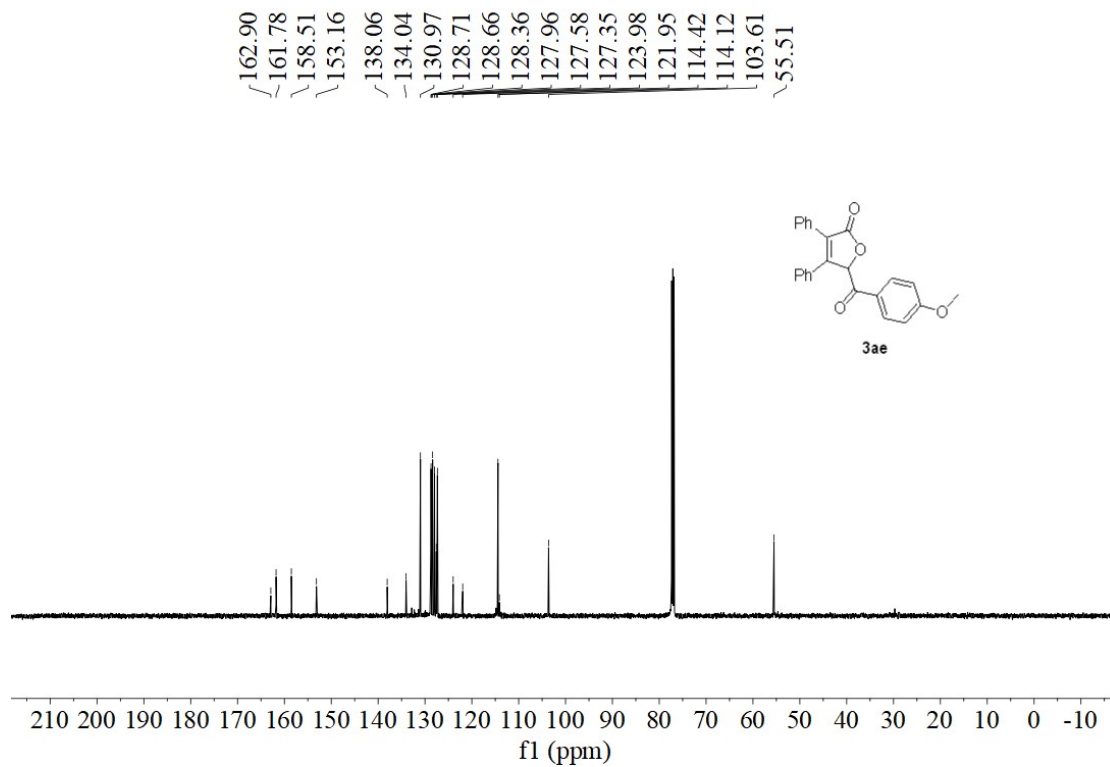
(400 MHz for ^1H NMR with CDCl_3 as solvent)



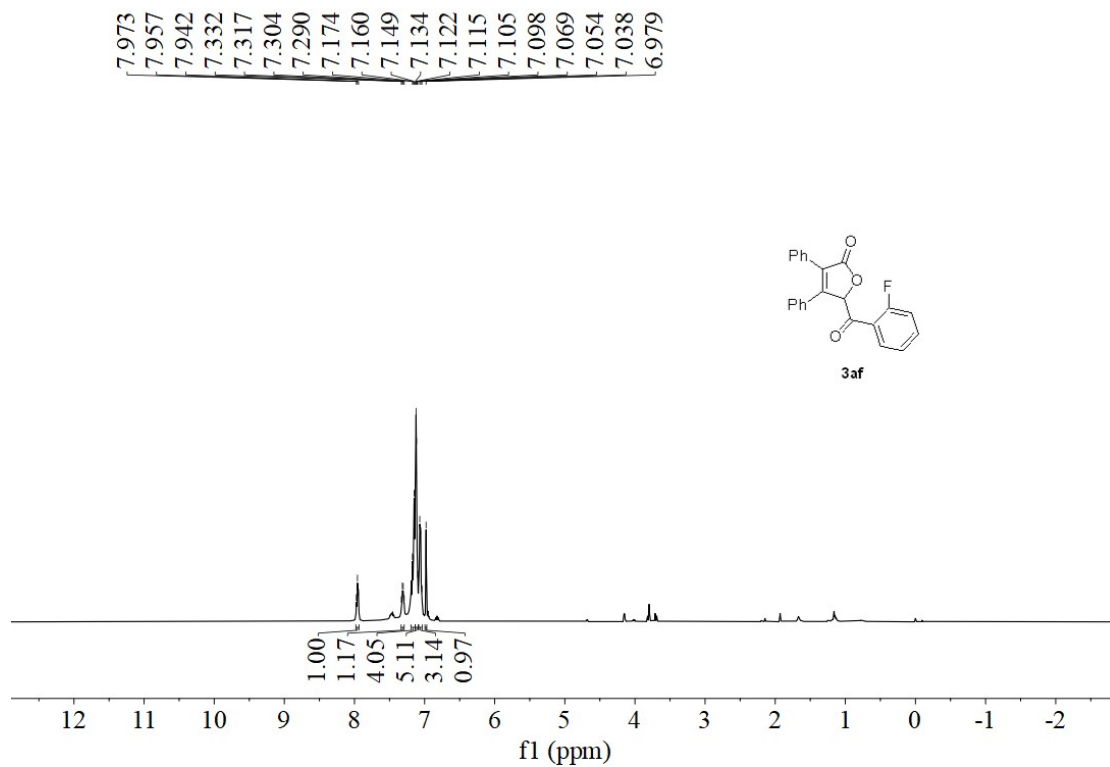
(126 MHz for ¹³C NMR with CDCl₃ as solvent)



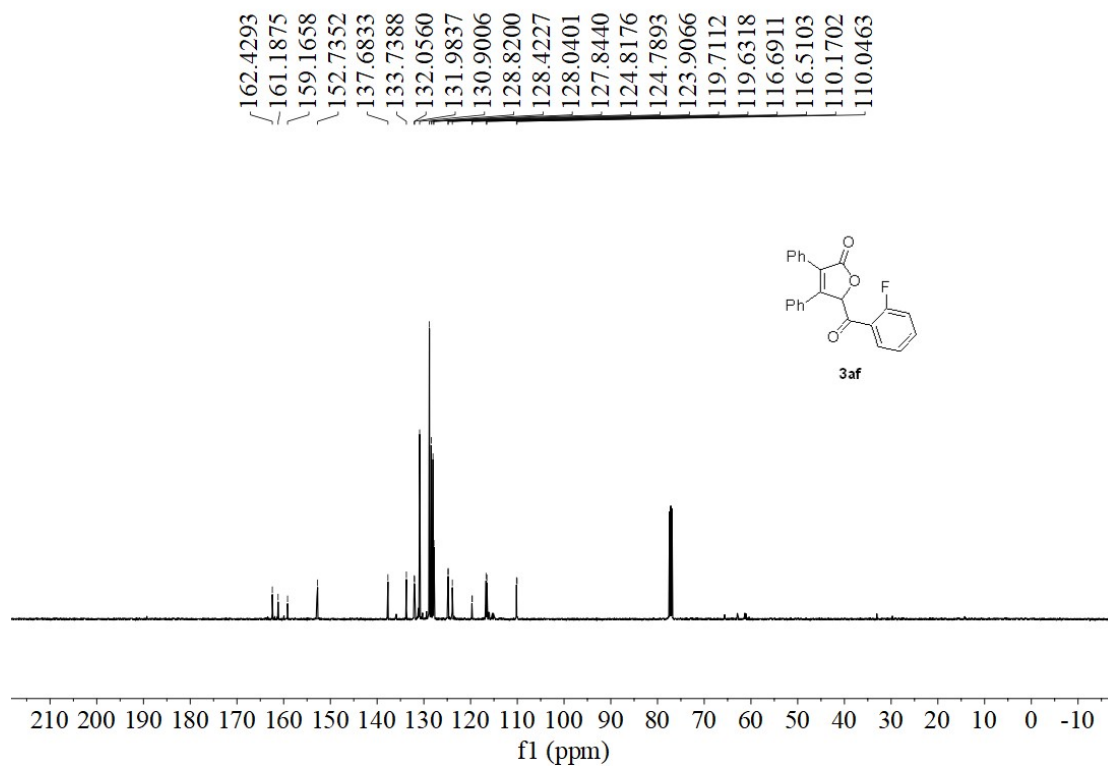
(500 MHz for ¹H NMR with CDCl₃ as solvent)



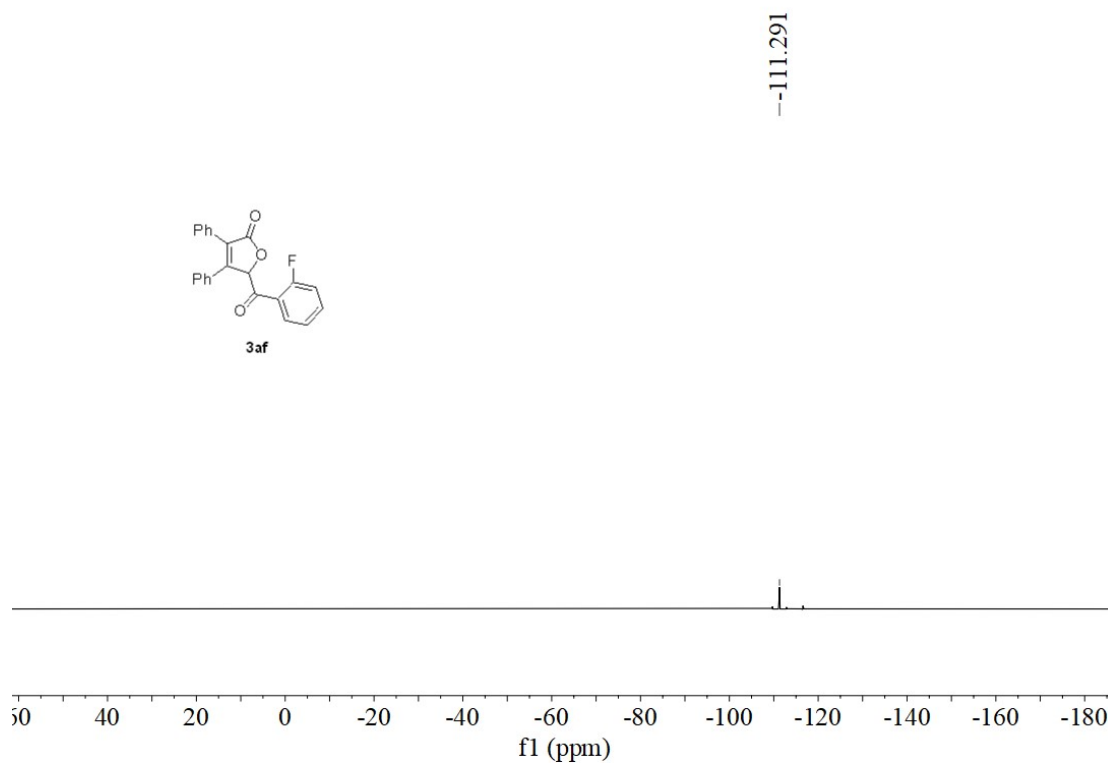
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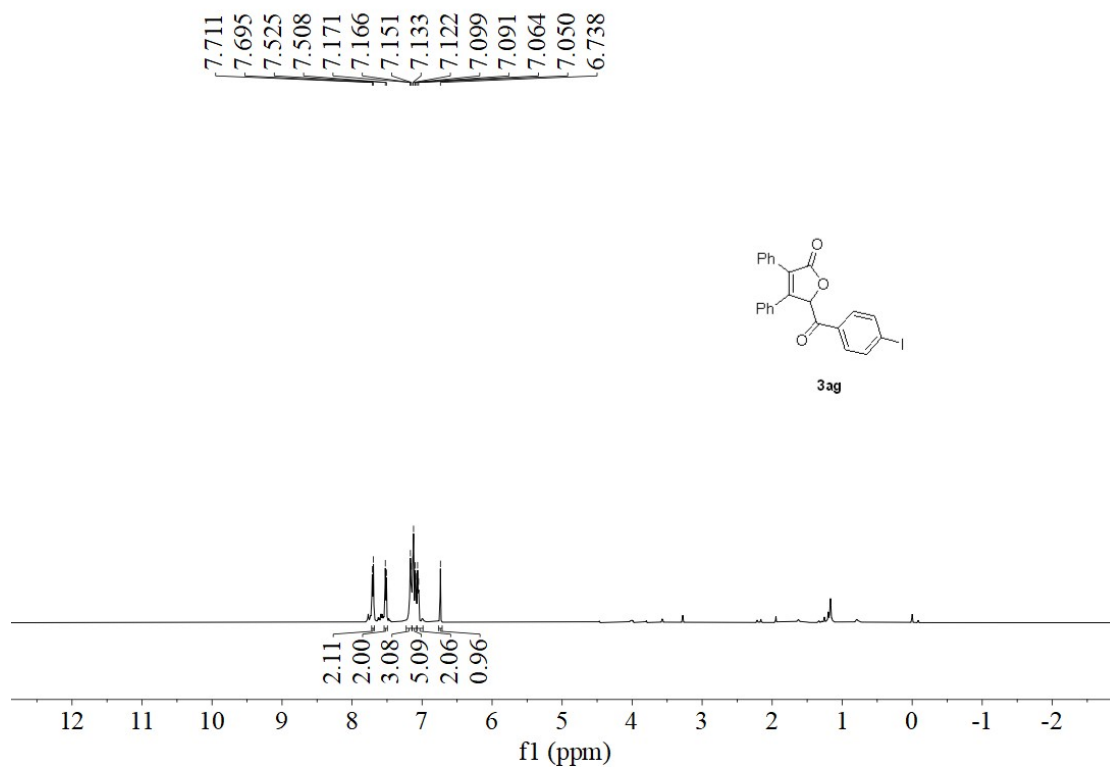
(500 MHz for ^1H NMR with CDCl_3 as solvent)



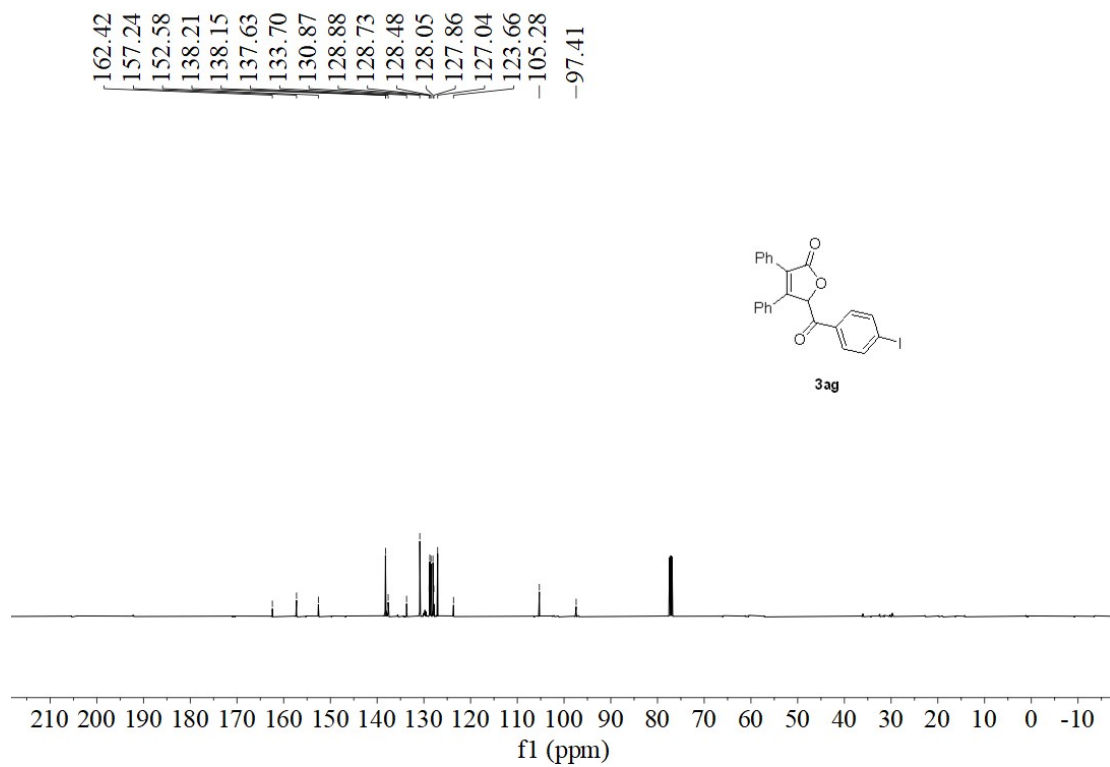
(126 MHz for ^{13}C NMR with CDCl_3 as solvent)



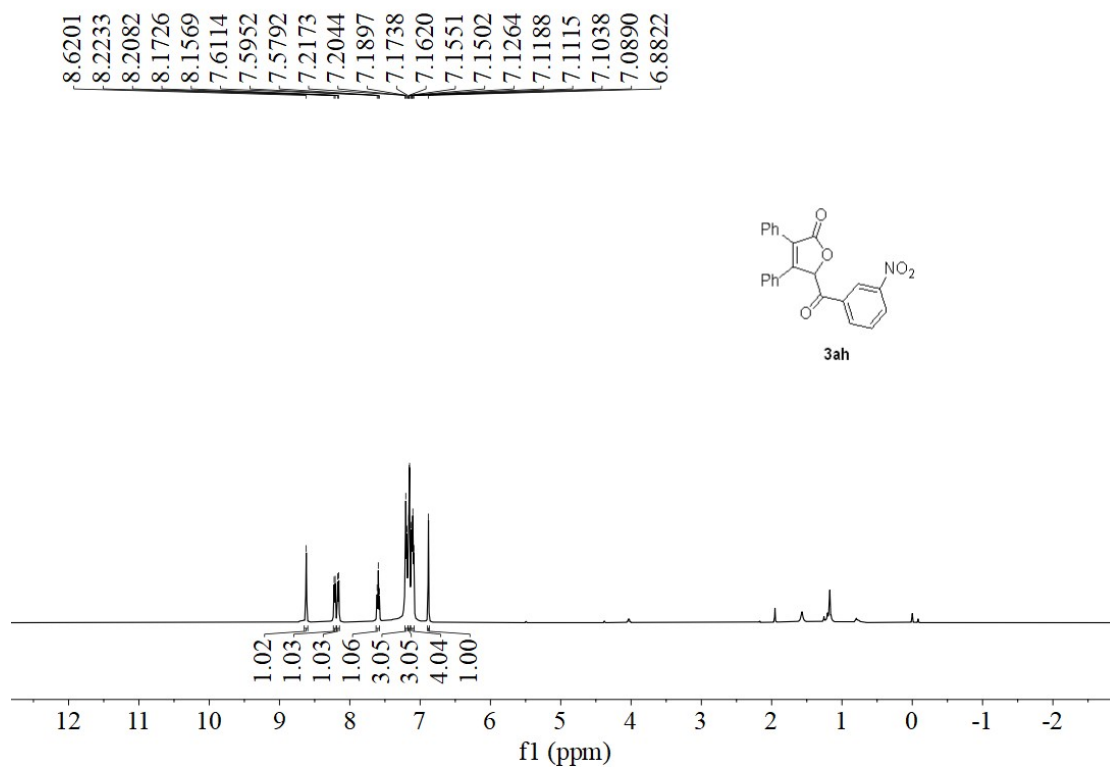
(471 MHz for ^{19}F NMR with CDCl_3 as solvent)



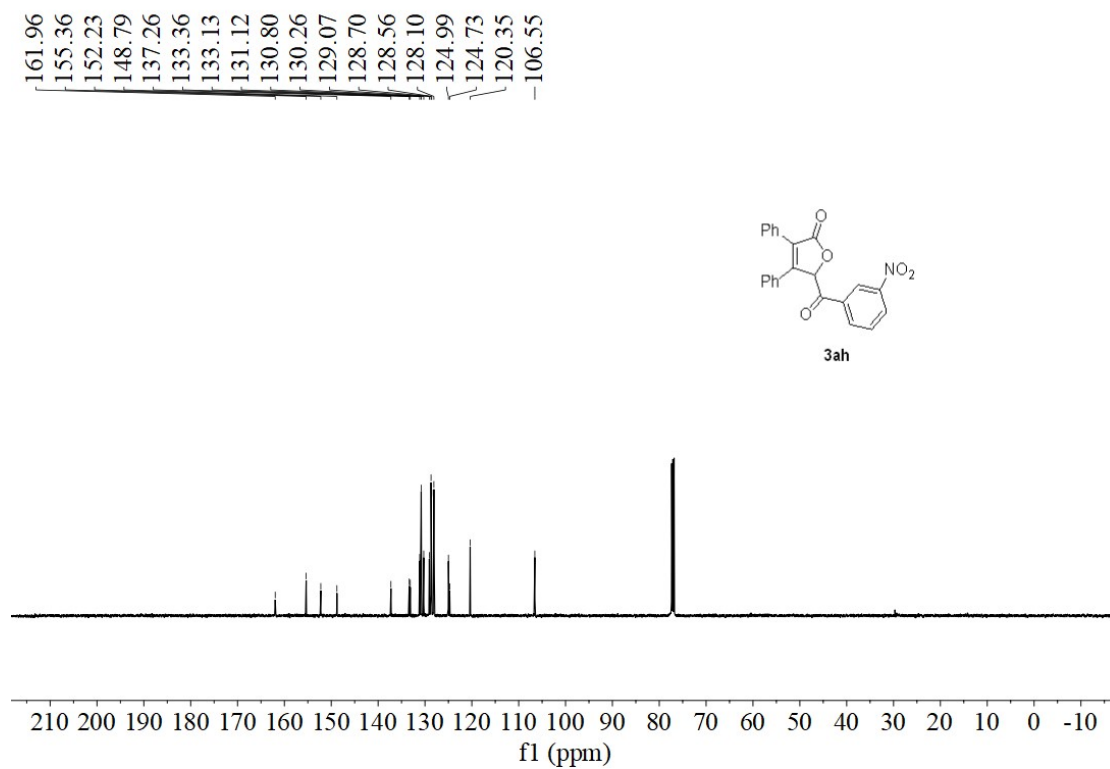
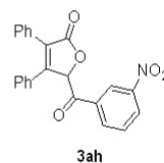
(500 MHz for ^1H NMR with CDCl_3 as solvent)



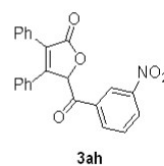
(126 MHz for ^{13}C NMR with CDCl_3 as solvent)



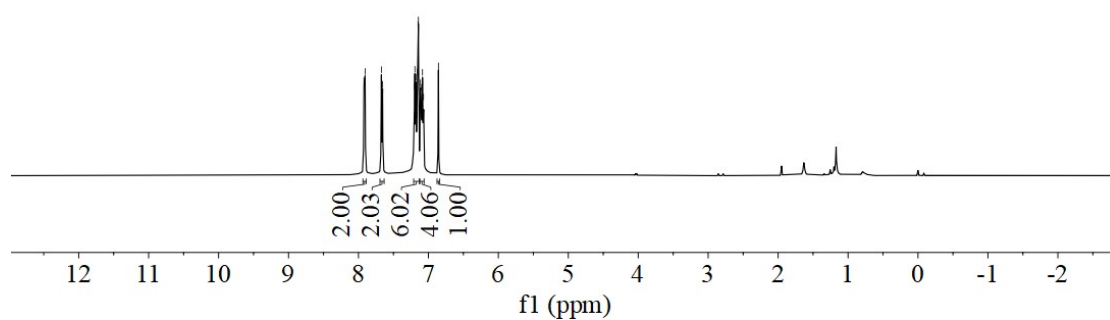
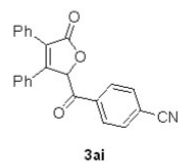
(500 MHz for ^1H NMR with CDCl_3 as solvent)



(126 MHz for ^{13}C NMR with CDCl_3 as solvent)

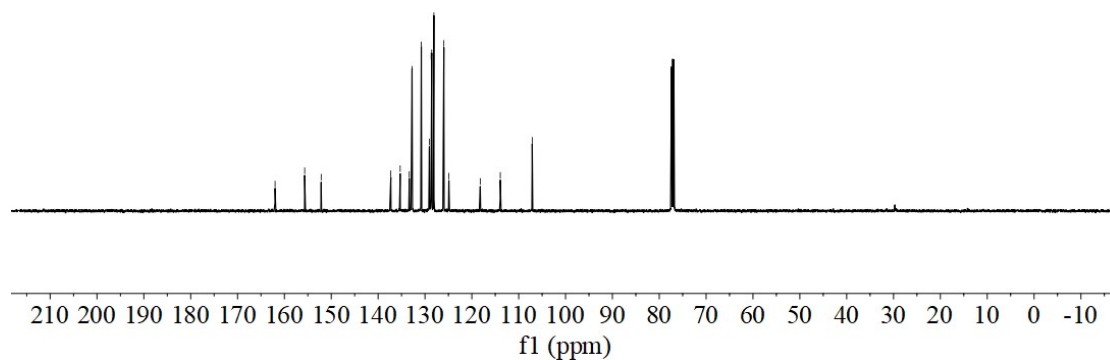
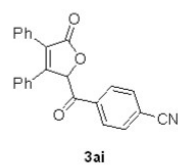


7.921
7.905
7.672
7.656
7.201
7.198
7.193
7.179
7.165
7.155
7.148
7.143
7.114
7.106
7.099
7.094
7.085
7.070
6.857

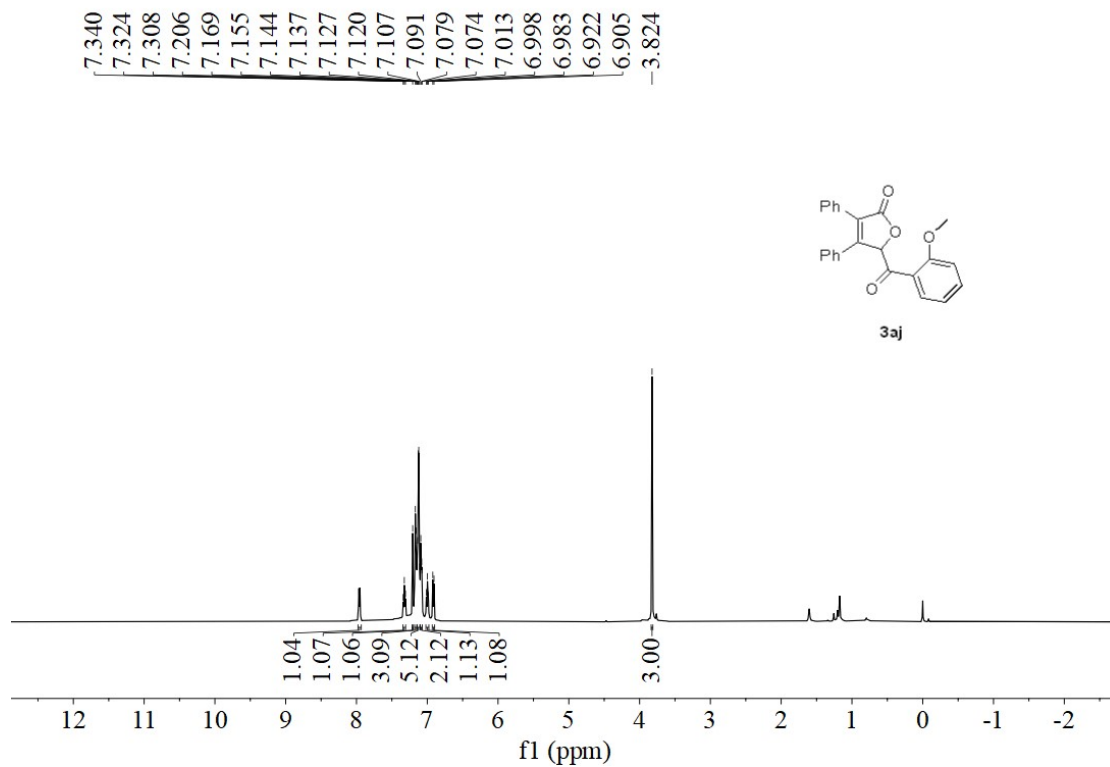


(500 MHz for ¹H NMR with CDCl₃ as solvent)

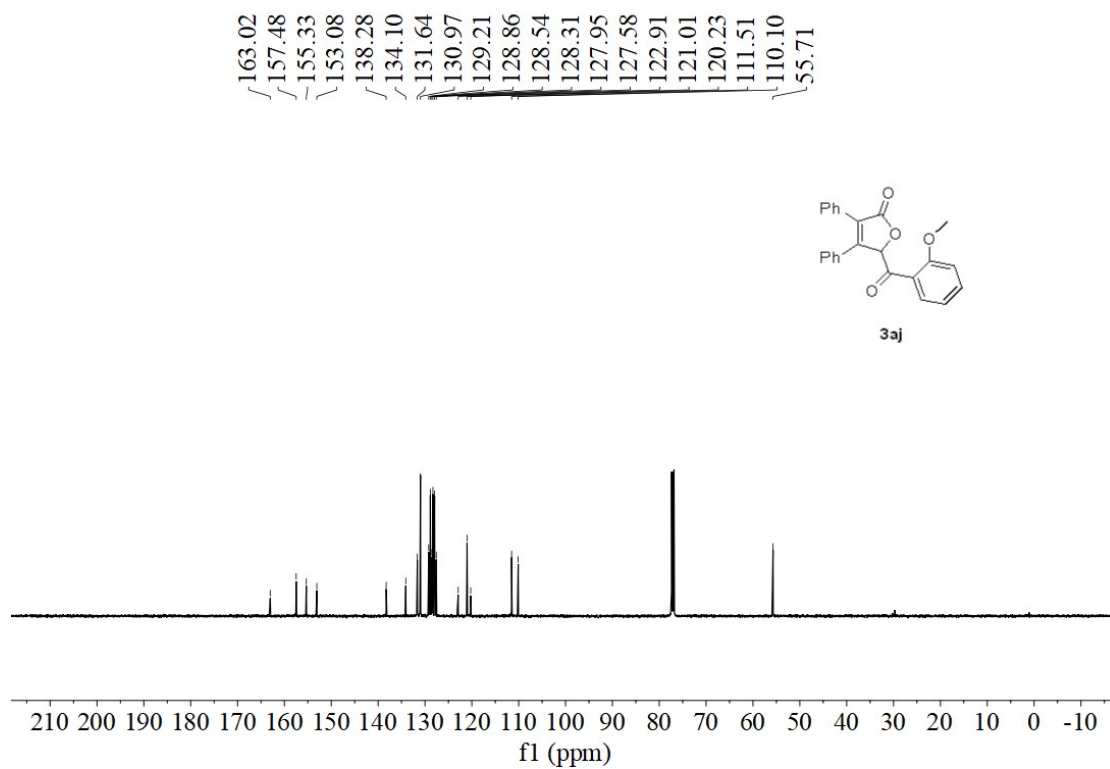
161.99
155.68
152.15
137.29
135.29
133.36
132.76
130.79
129.06
128.68
128.59
128.56
128.10
125.97
124.90
118.20
113.92
107.11



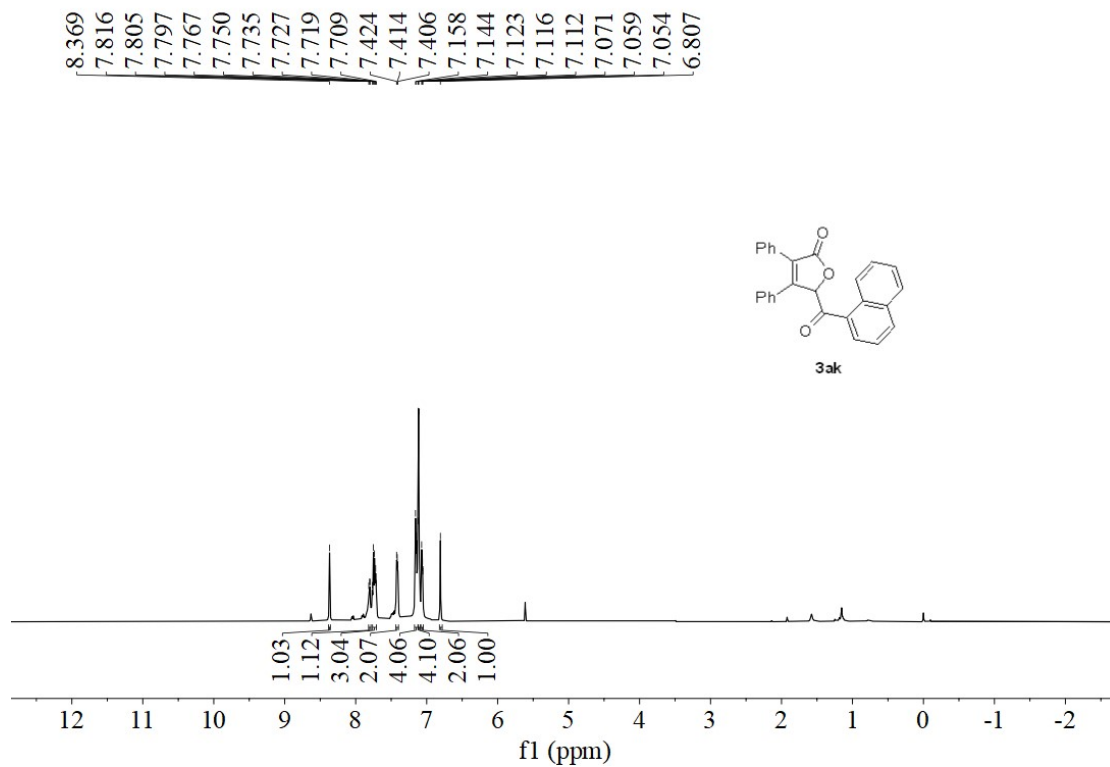
(126 MHz for ¹³C NMR with CDCl₃ as solvent)



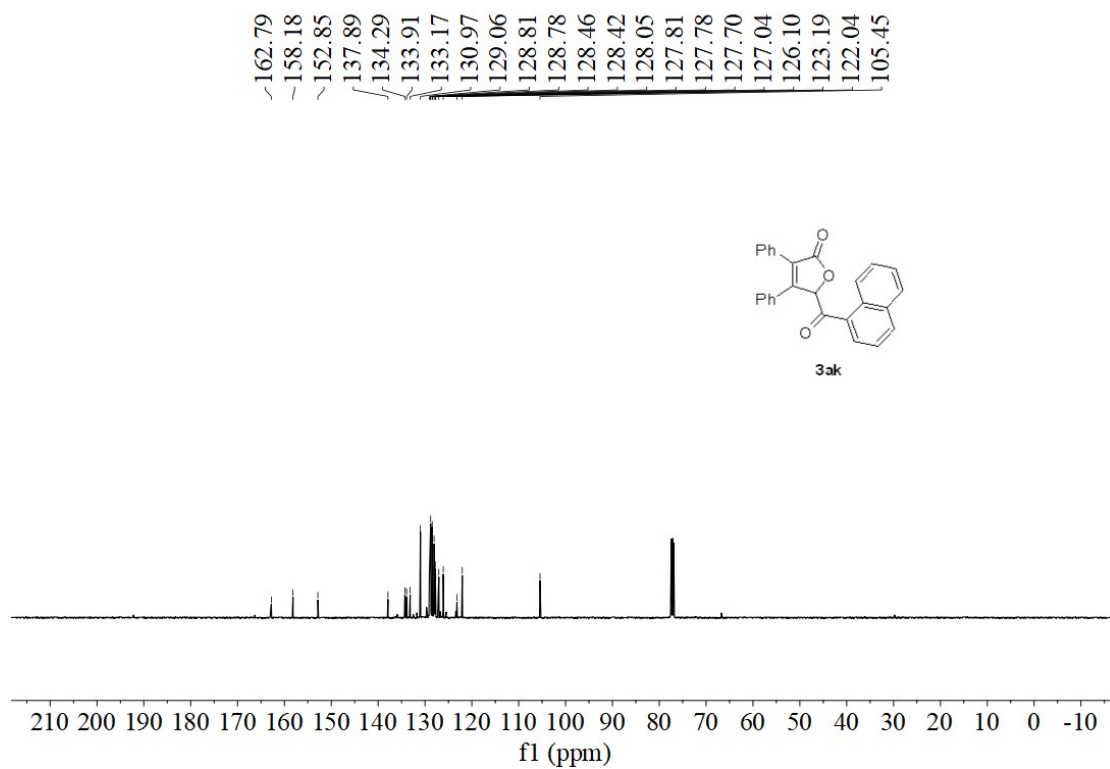
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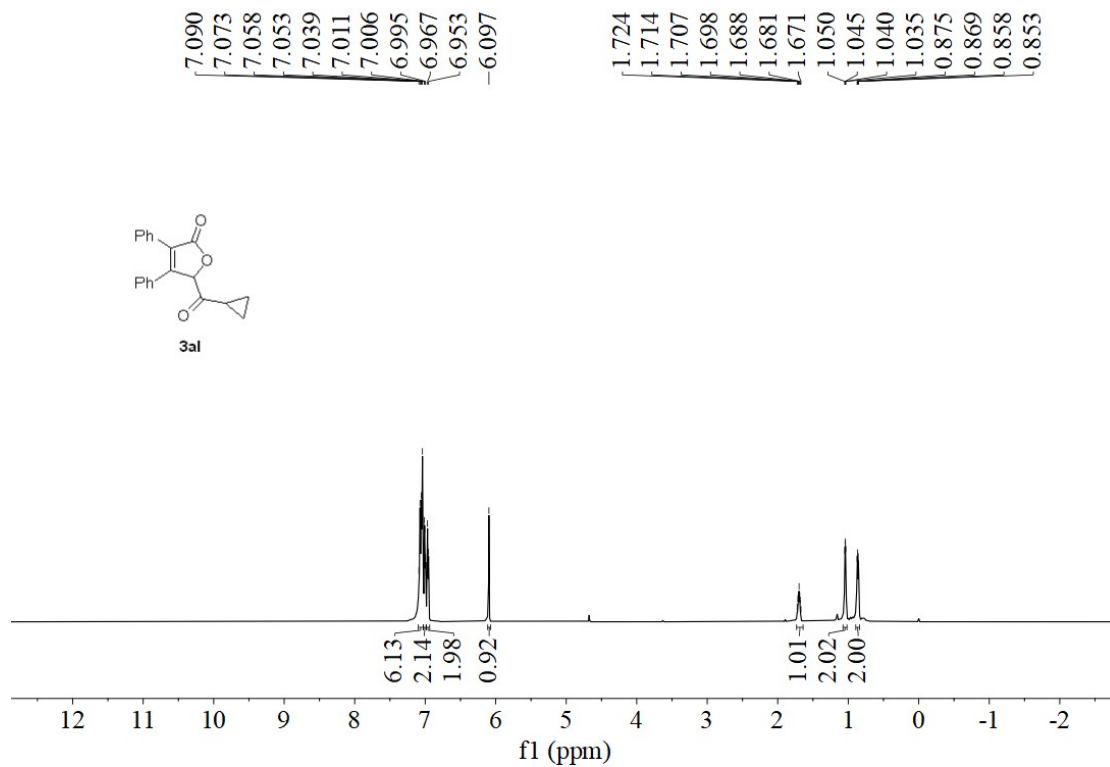
(126 MHz for ^{13}C NMR with CDCl_3 as solvent)



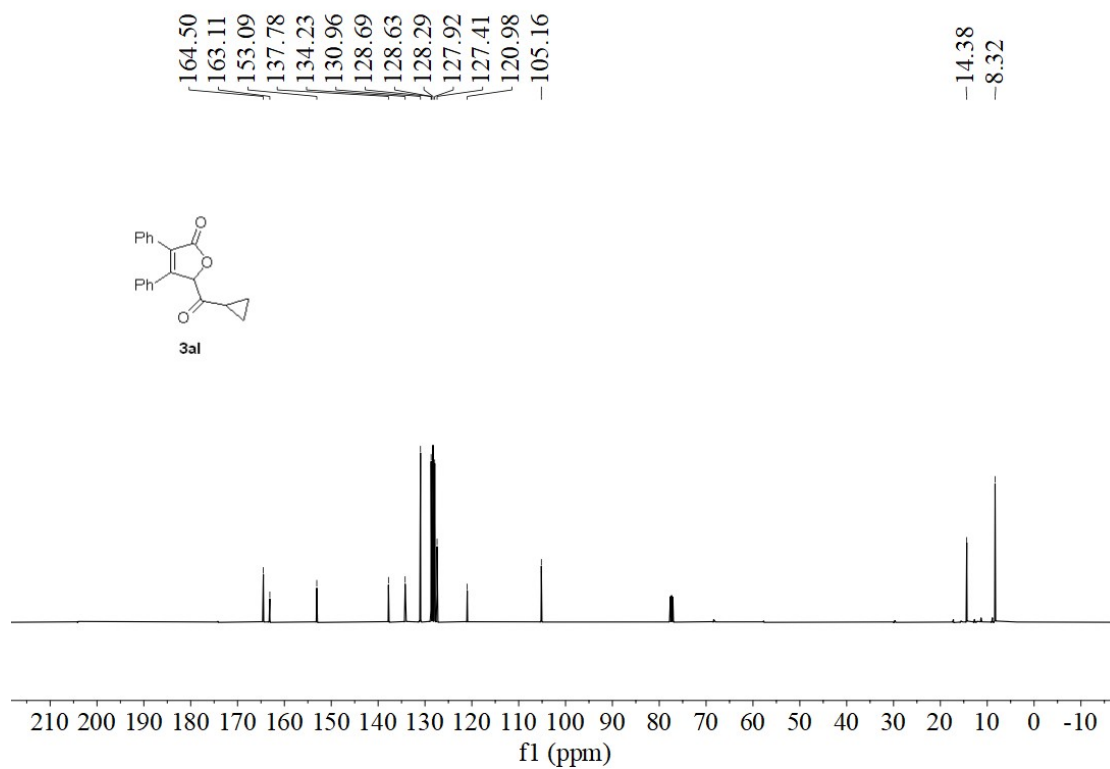
(500 MHz for ^1H NMR with CDCl_3 as solvent)



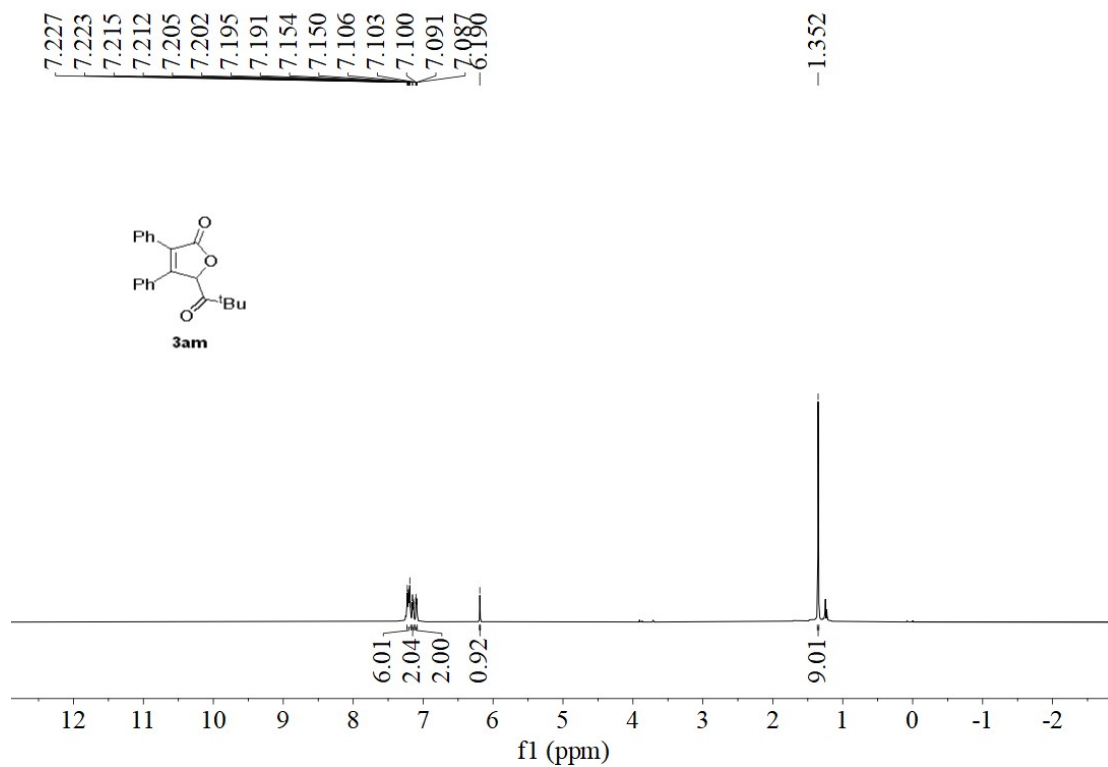
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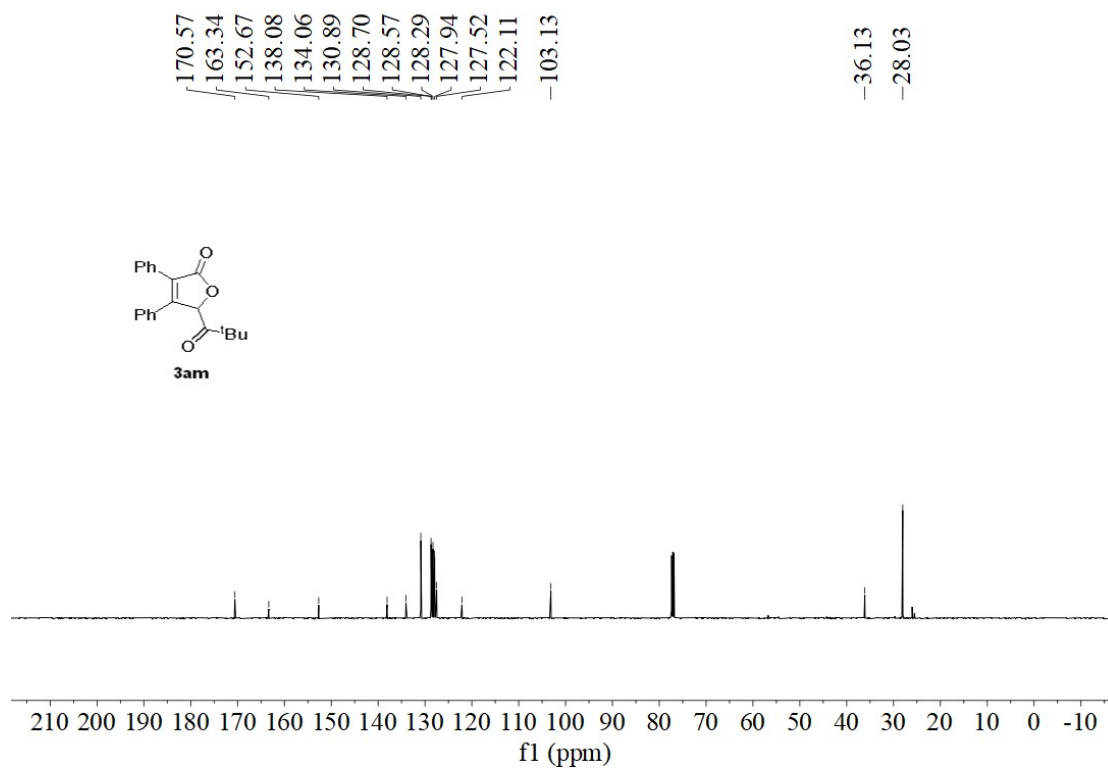
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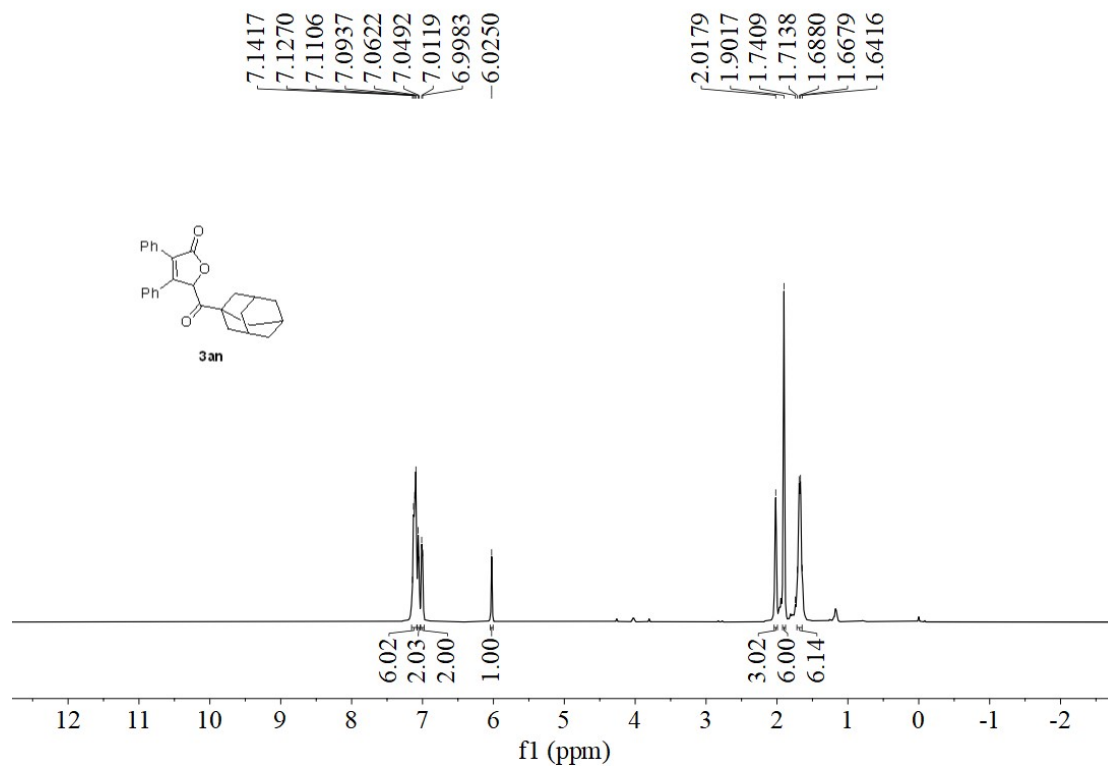
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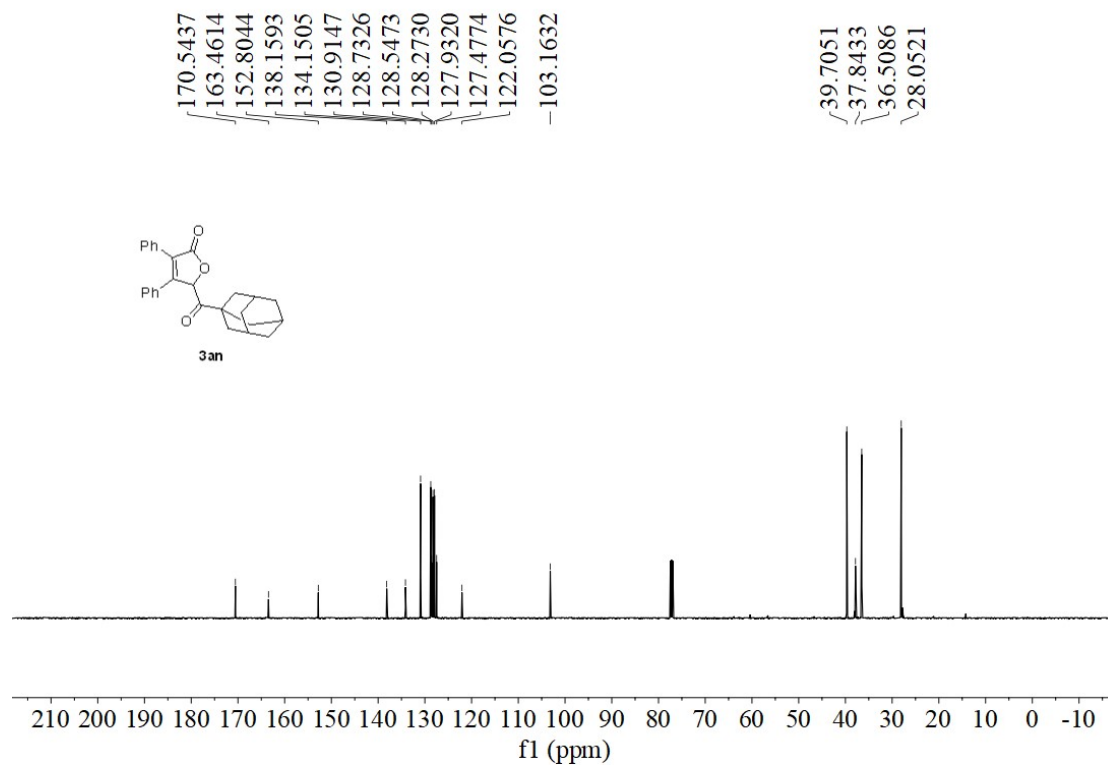
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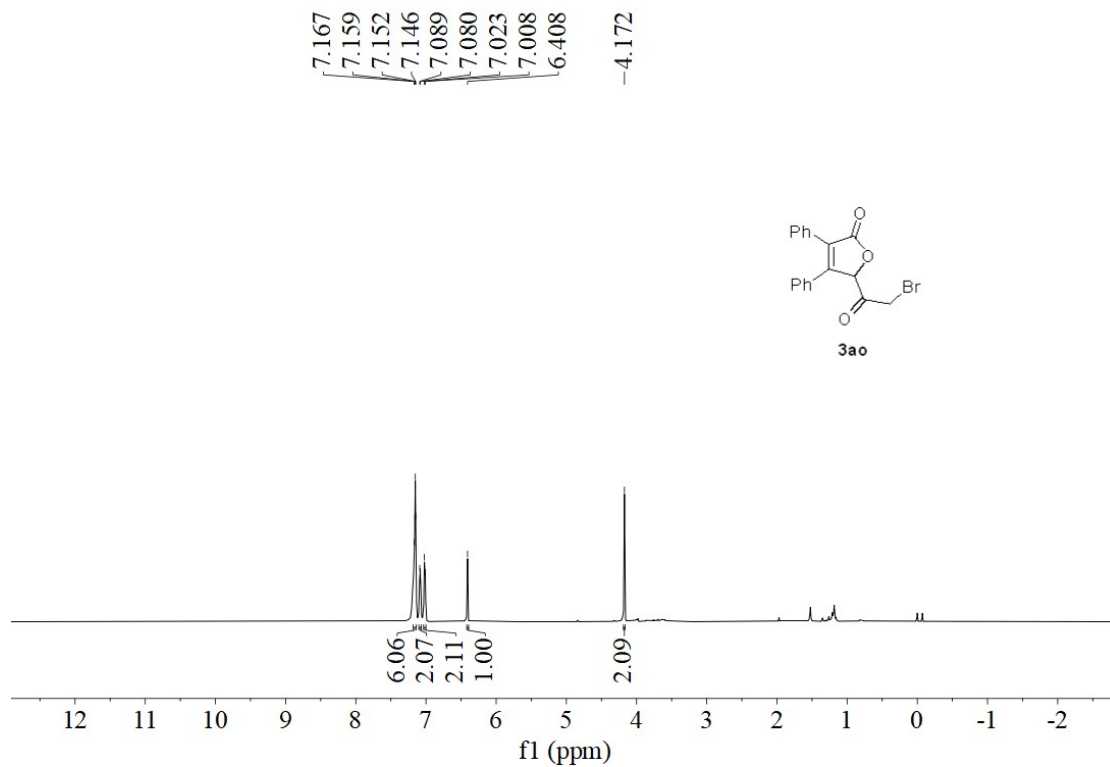
(126 MHz for ¹³C NMR with CDCl₃ as solvent)



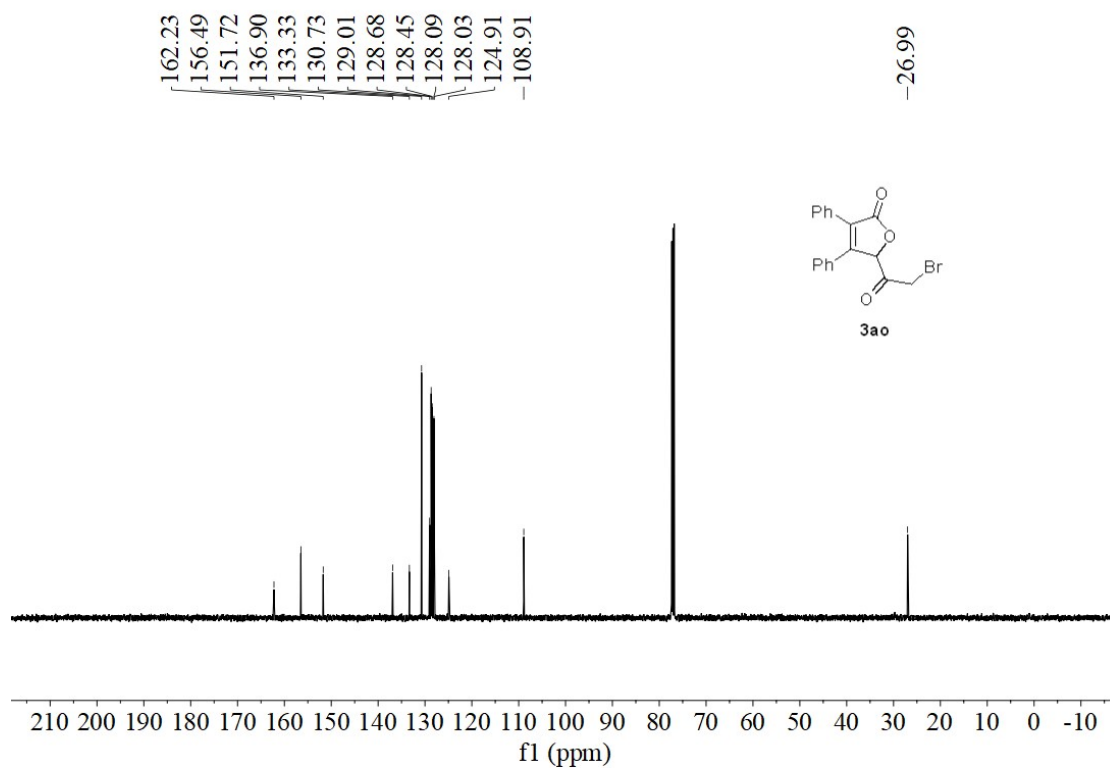
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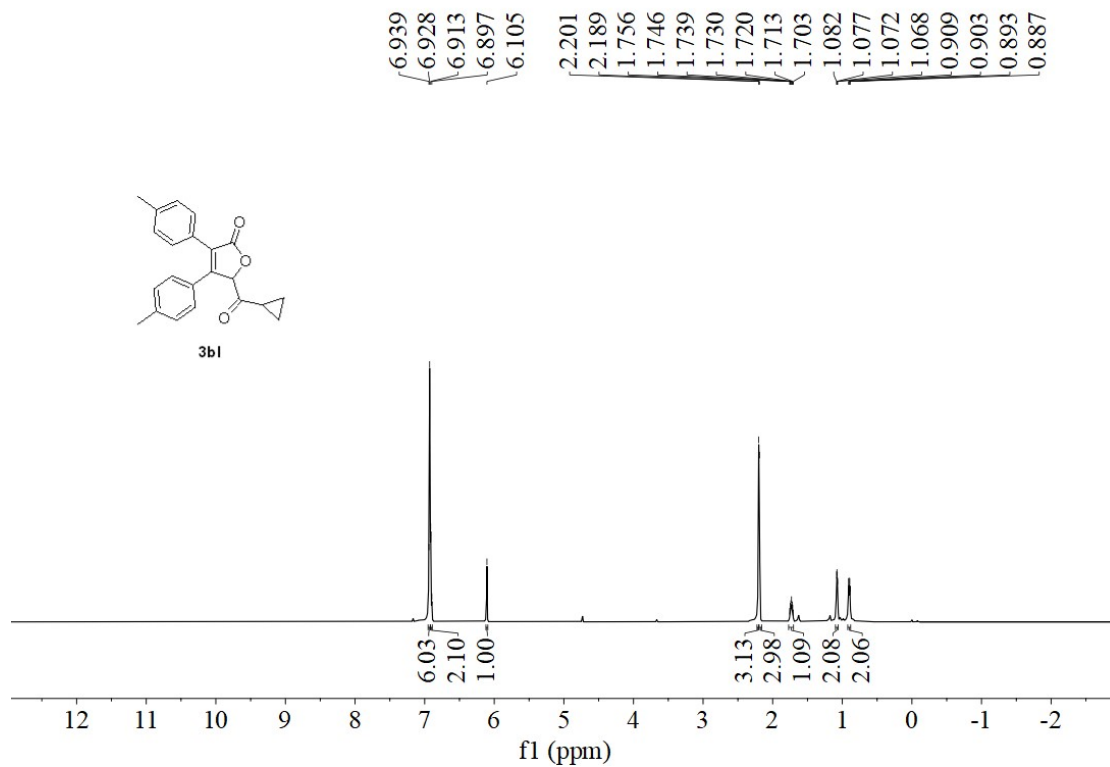
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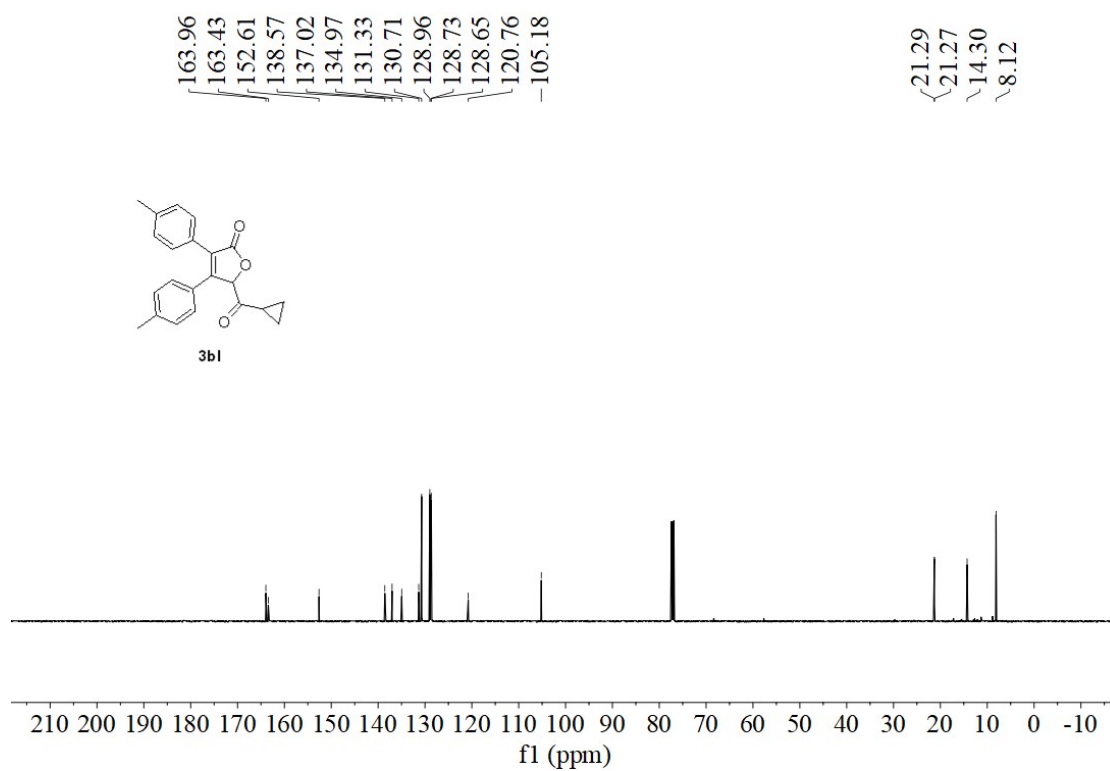
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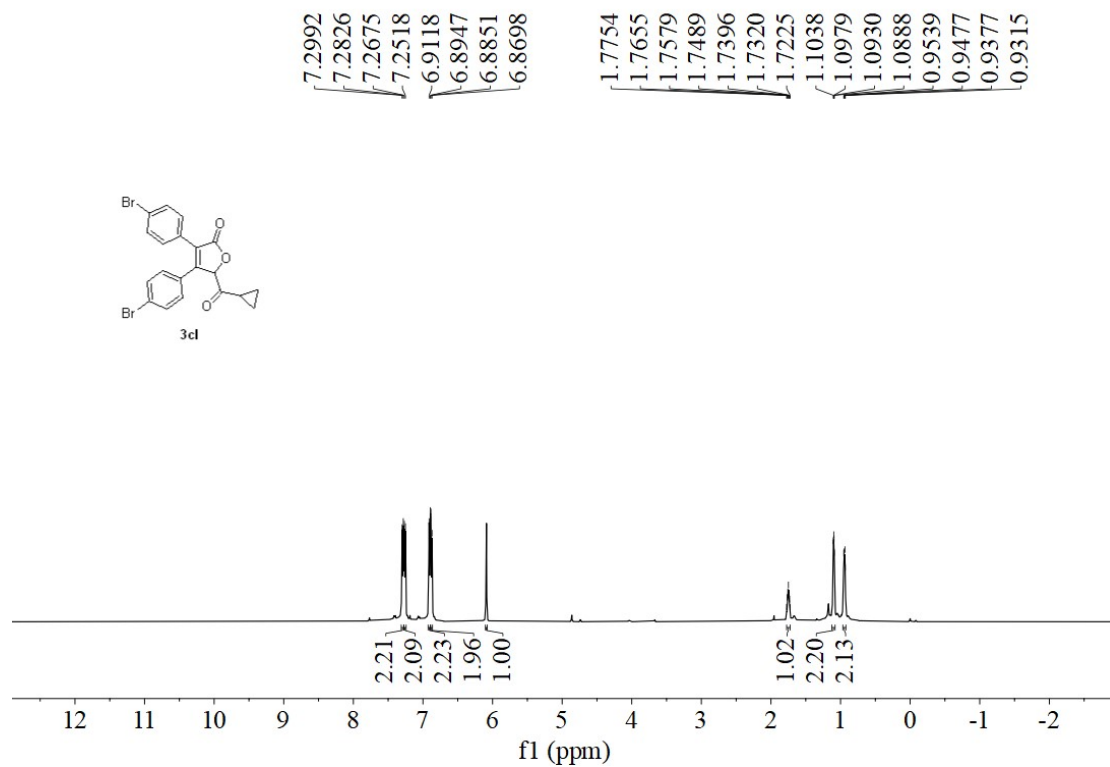
(126 MHz for ^{13}C NMR with CDCl_3 as solvent)



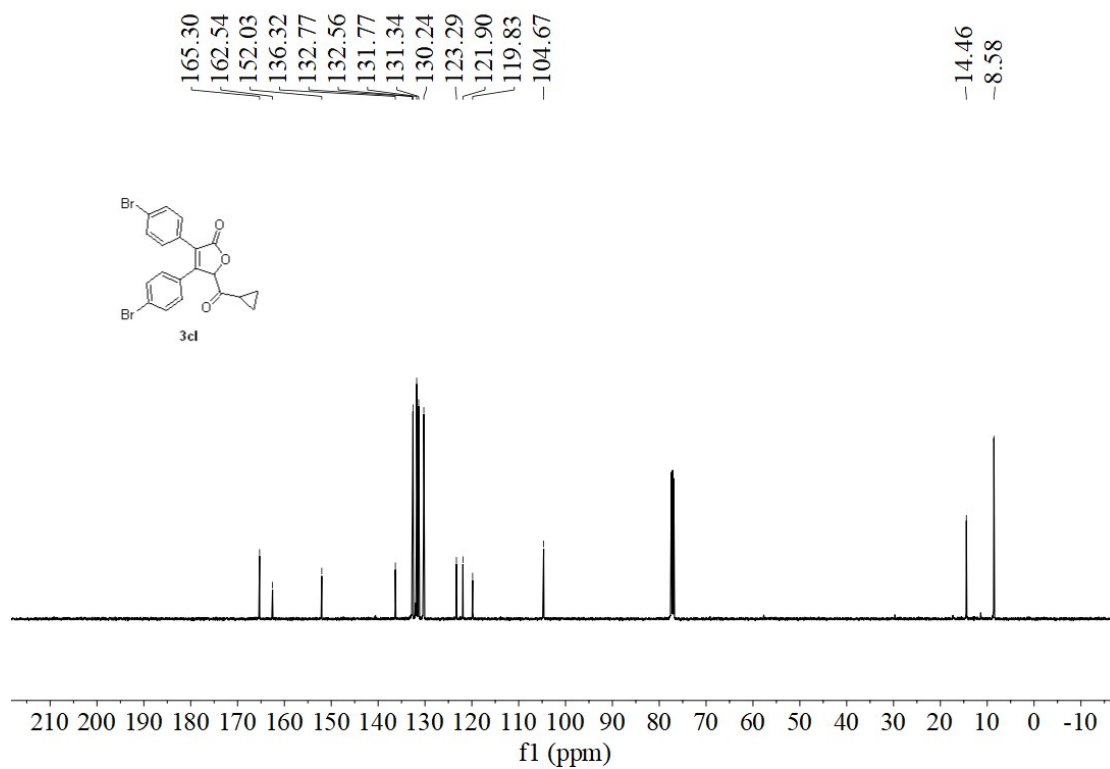
(500 MHz for ¹H NMR with CDCl₃ as solvent)



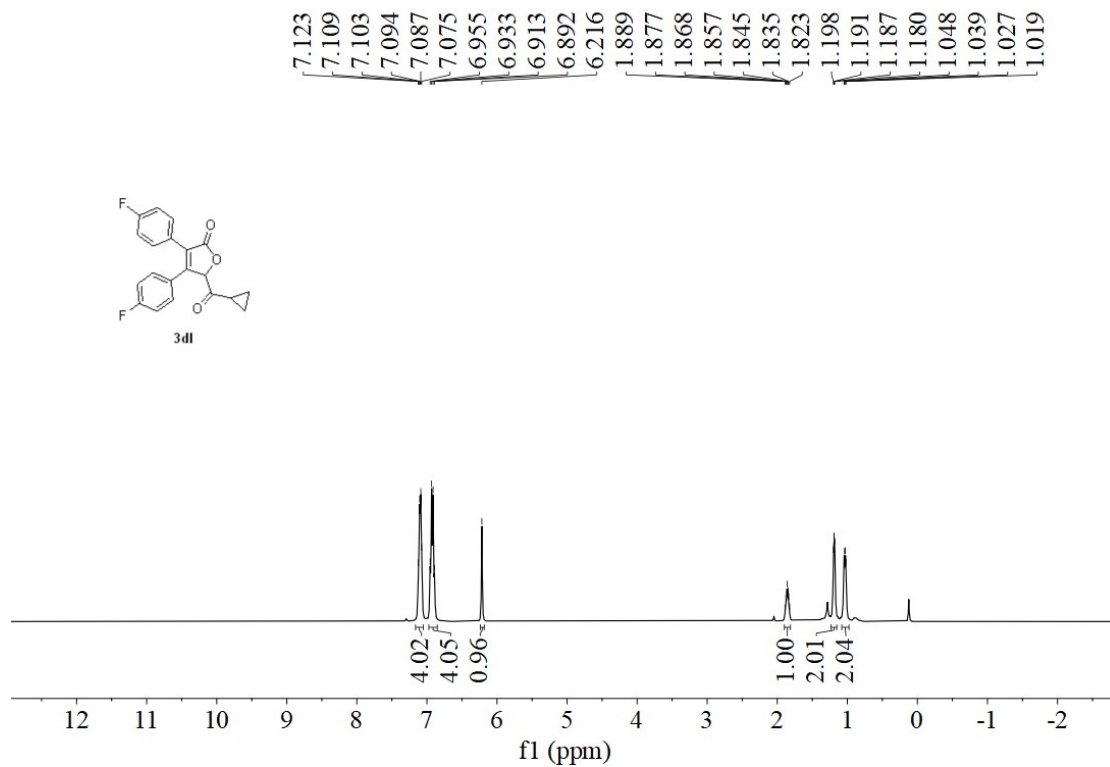
(126 MHz for ¹³C NMR with CDCl₃ as solvent)



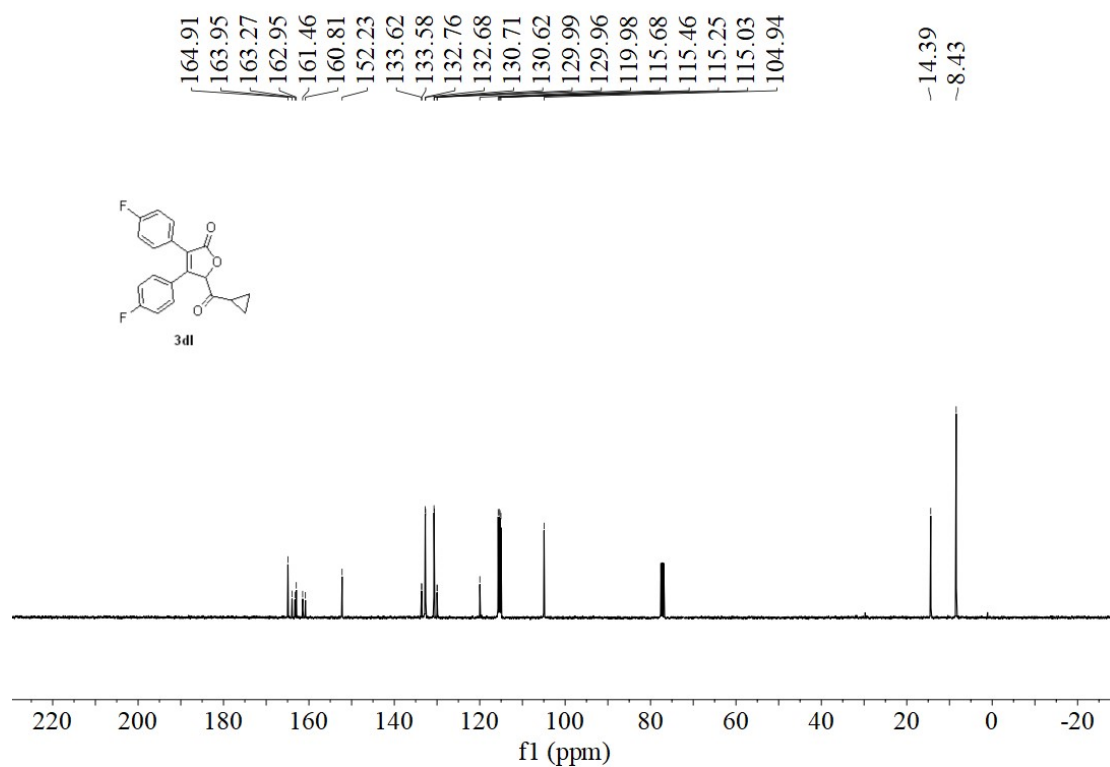
(500 MHz for ¹H NMR with CDCl₃ as solvent)



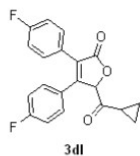
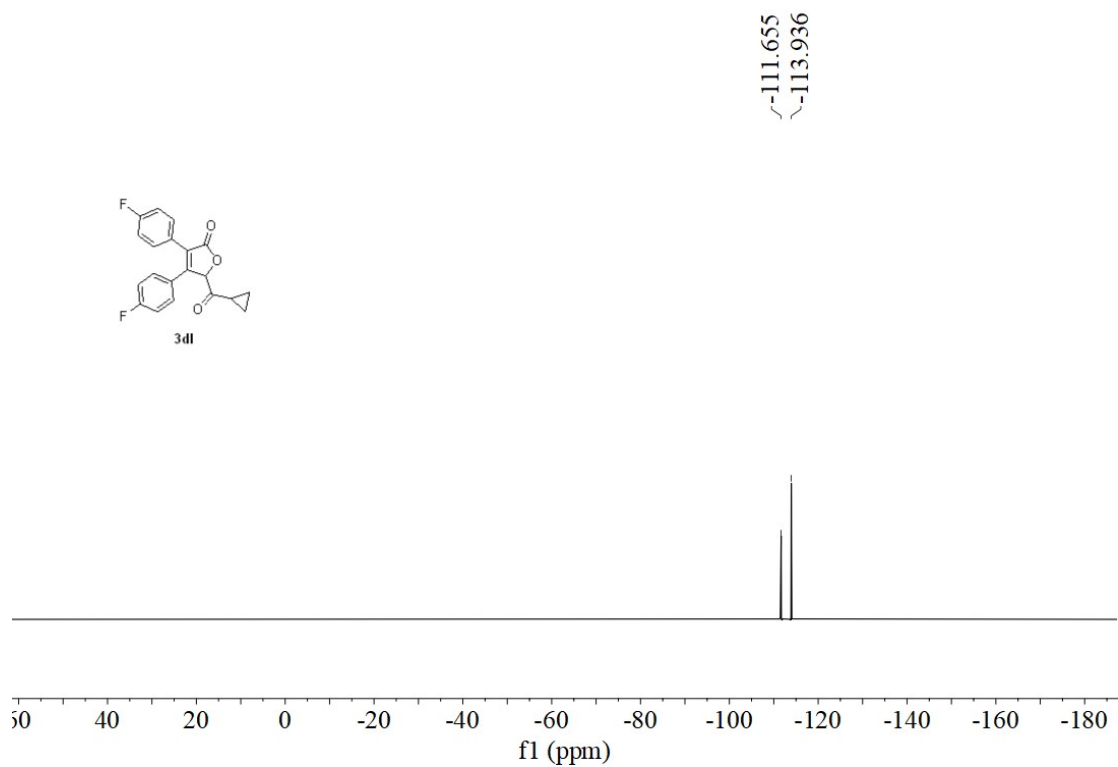
(126 MHz for ¹³C NMR with CDCl₃ as solvent)



(500 MHz for ¹H NMR with CDCl₃ as solvent)

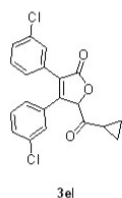
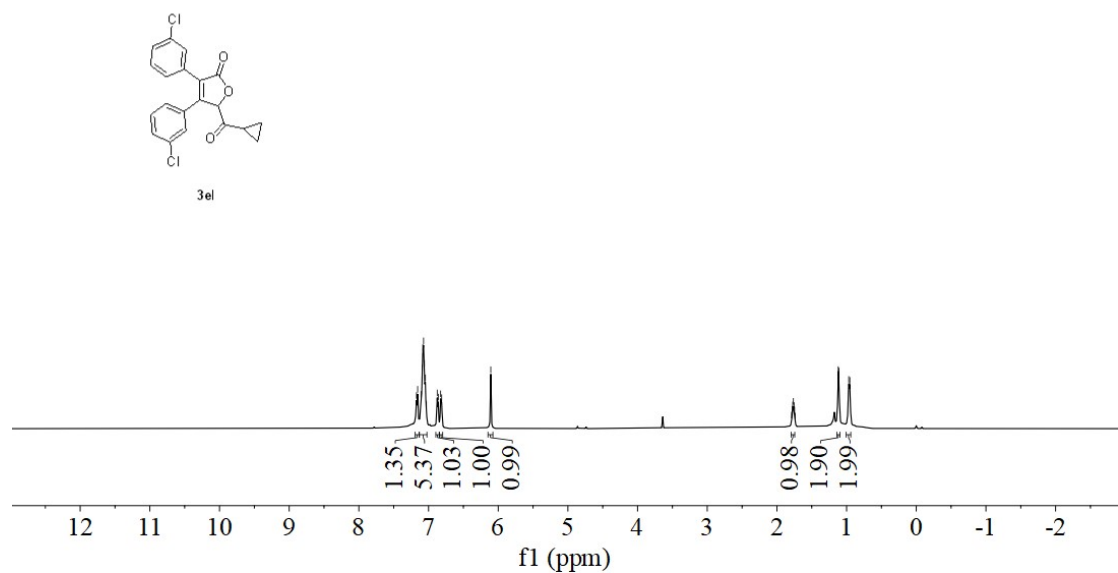


(101 MHz for ¹³C NMR with CDCl₃ as solvent)

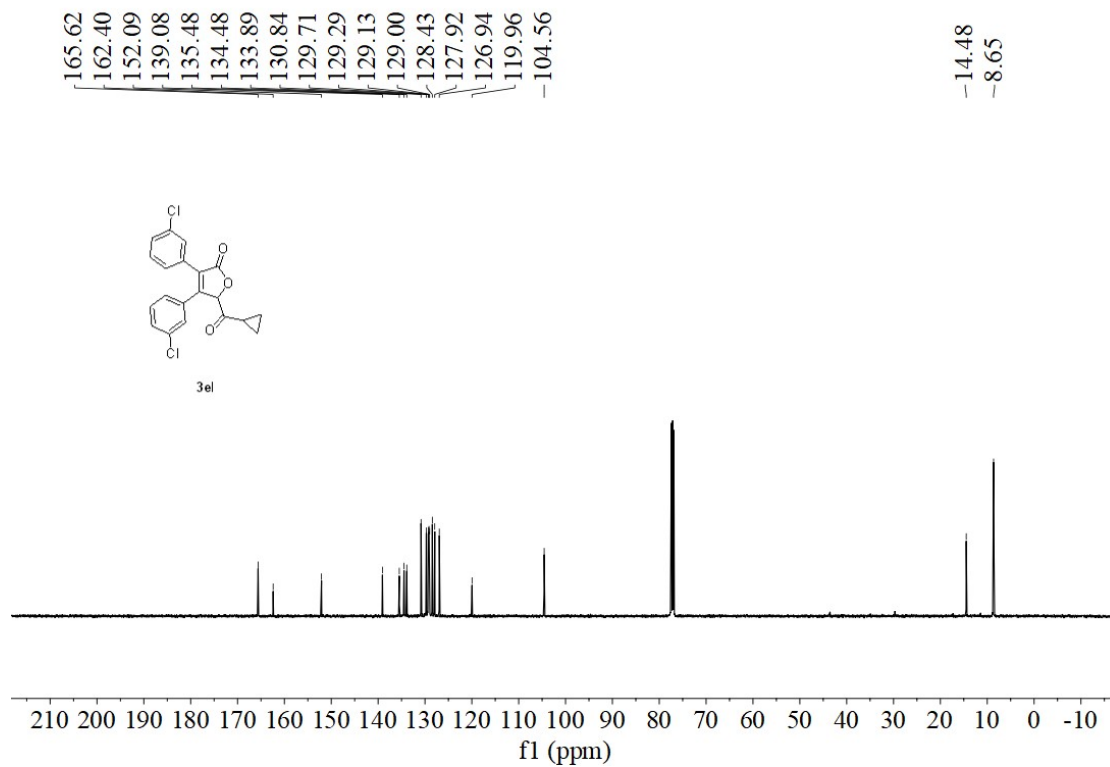


(471 MHz for ^{19}F NMR with CDCl_3 as solvent)

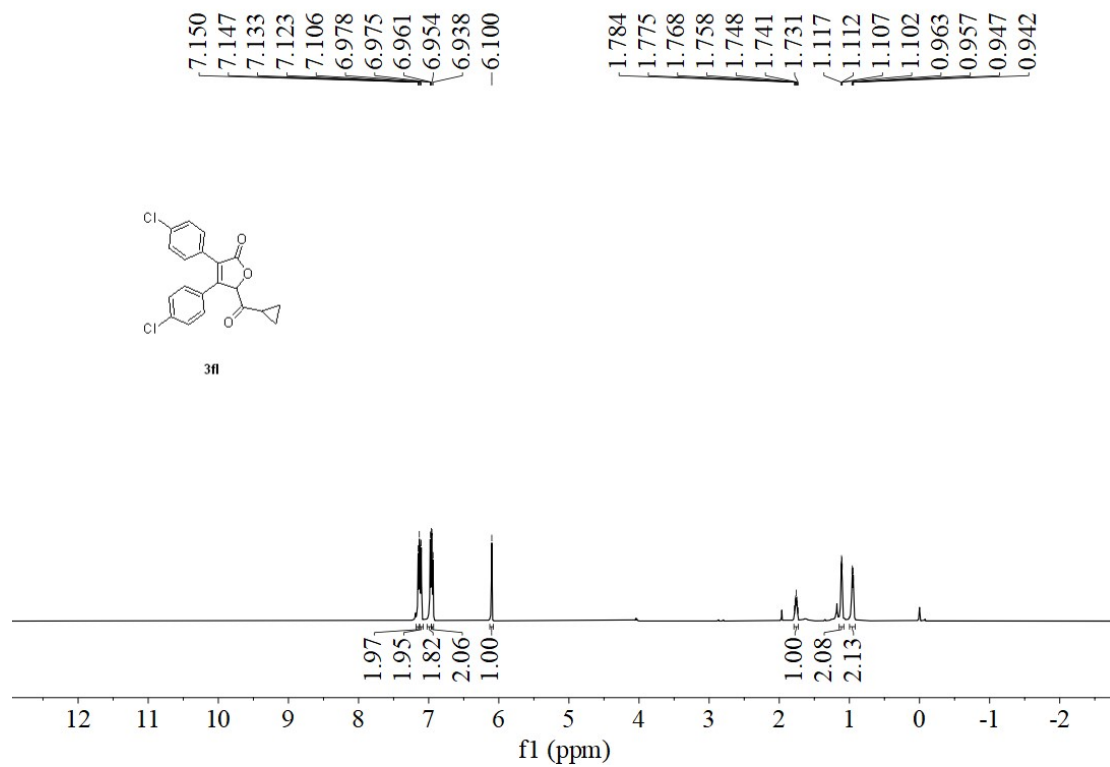
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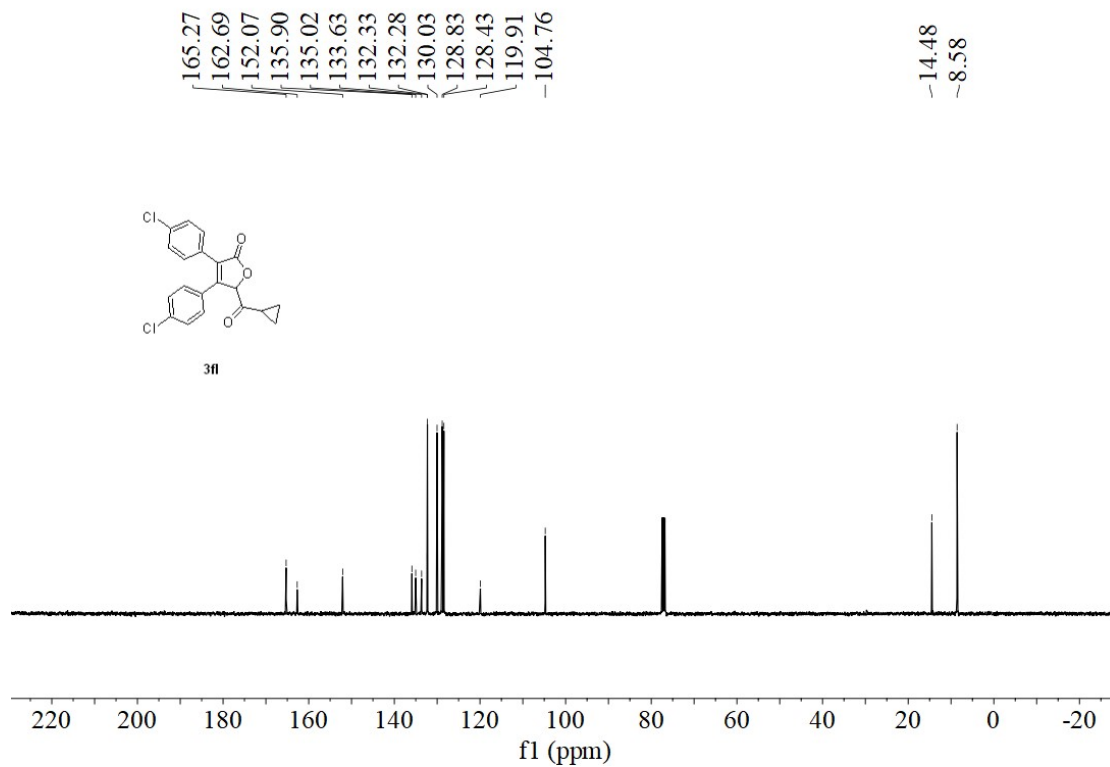
(500 MHz for ^1H NMR with CDCl_3 as solvent)



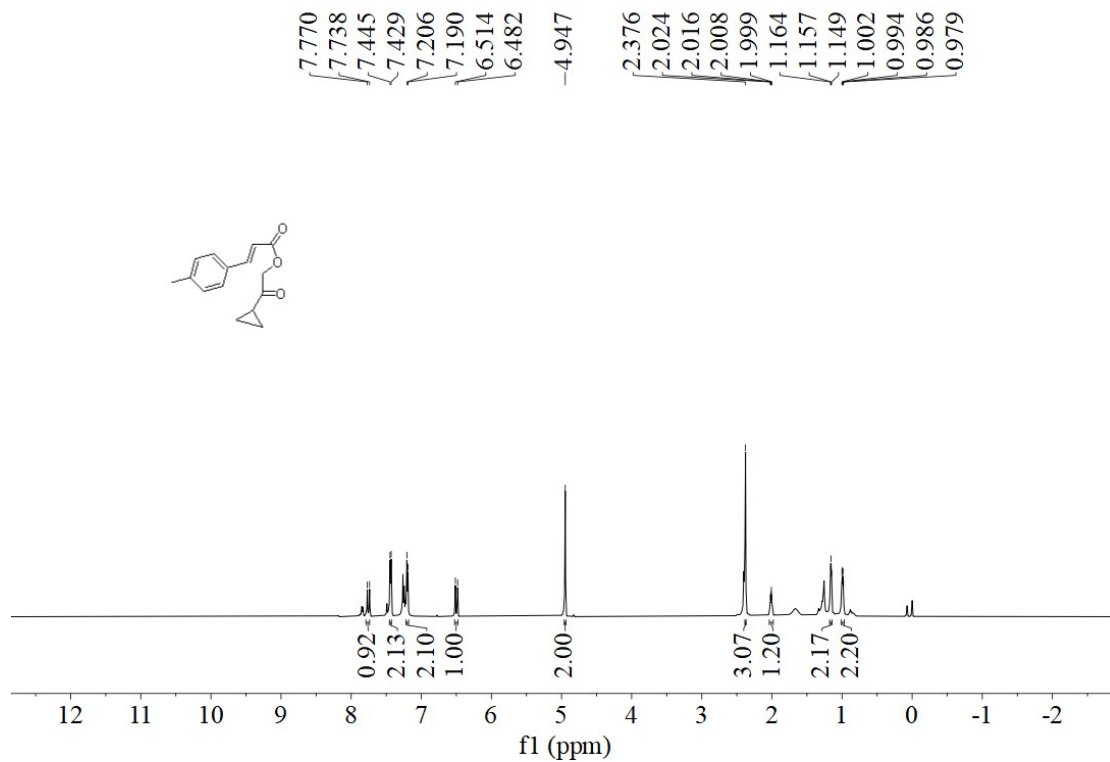
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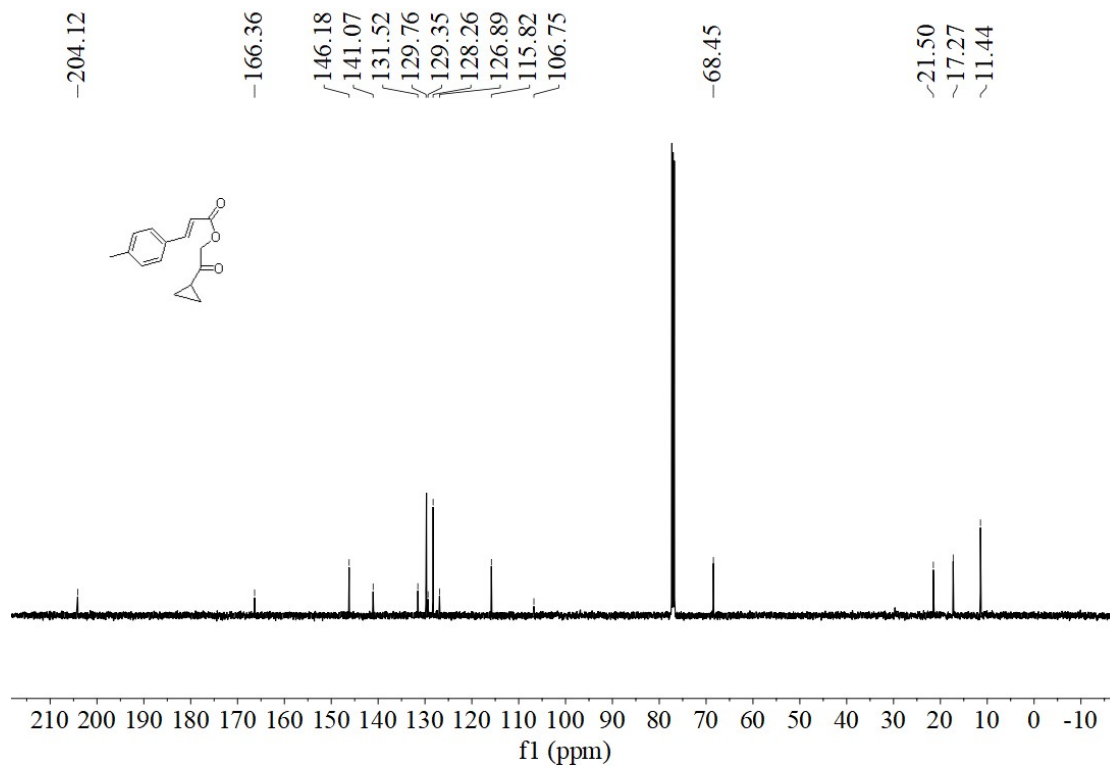
(500 MHz for ^1H NMR with CDCl_3 as solvent)



(101 MHz for ^{13}C NMR with CDCl_3 as solvent)



(400 MHz for ^1H NMR with CDCl_3 as solvent)



(126 MHz for ^{13}C NMR with CDCl_3 as solvent)