

## Supporting Information

### Synthesis of 1,2-dicarbonyls from five-membered cyclic enamines and arylglyoxal hydrates under metal-free condition

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R. China

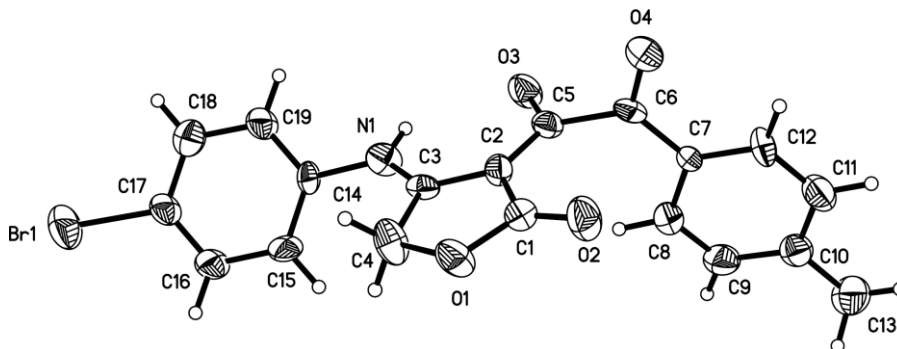
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## Experimental

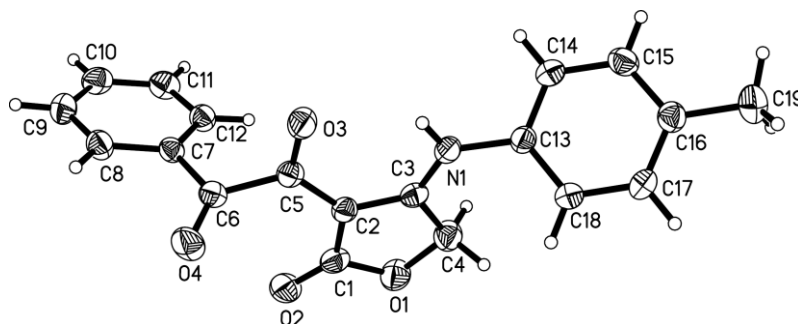
### General Information

Microwave irradiation was carried out with Initiator 2.5 Microwave Synthesizers from Biotage, Uppsala, Sweden. Melting points were determined in open capillaries and were uncorrected. IR spectra were taken on a FT-IR-Tensor 27 spectrometer in KBr pellets and reported in  $\text{cm}^{-1}$ .  $^1\text{H}$  NMR ( $^{13}\text{C}$  NMR) spectra were measured on a Bruker DPX 400 MHz spectrometer in  $\text{DMSO-}d_6$  with chemical shift ( $\delta$ ) given in ppm relative to TMS as internal standard [(s = singlet, d = doublet, t = triplet, brs = broad singlet, m = multiplet), coupling constant (Hz)]. HRMS (ESI) was determined by using microTOF-QII HRMS/MS instrument (BRUKER). X-Ray crystallographic analysis was performed with a Siemens SMART CCD and a Siemens P4 diffractometer.



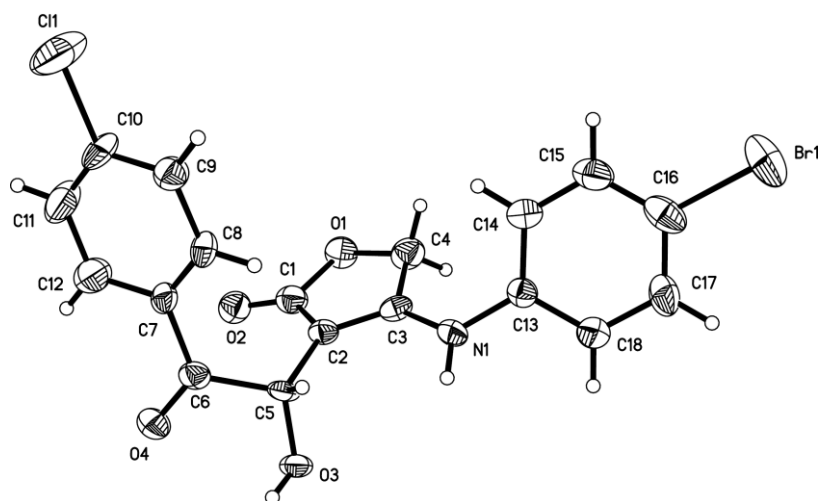
**Fig 1, X-ray Structure of 3m**

Crystal data for **3m** (CCDC: 2231949):  $\text{C}_{19}\text{H}_{14}\text{BrNO}_4$ ,  $M_r = 400.22$ , Monoclinic,  $a = 10.9020(8)$  Å,  $b = 6.2300(5)$  Å,  $c = 25.290(2)$  Å,  $U = 1668.5(2)$  Å<sup>3</sup>,  $T = 293(2)$  K, space group  $P2(1)/c$ ,  $Z = 4$ , 8126 reflections measured, 2943 unique ( $R_{\text{int}} = 0.1963$ ) which were used in all calculation. The final  $wR(F_2)$  was 0.1296 (all data)



**Fig 2, X-ray Structure of 3t**

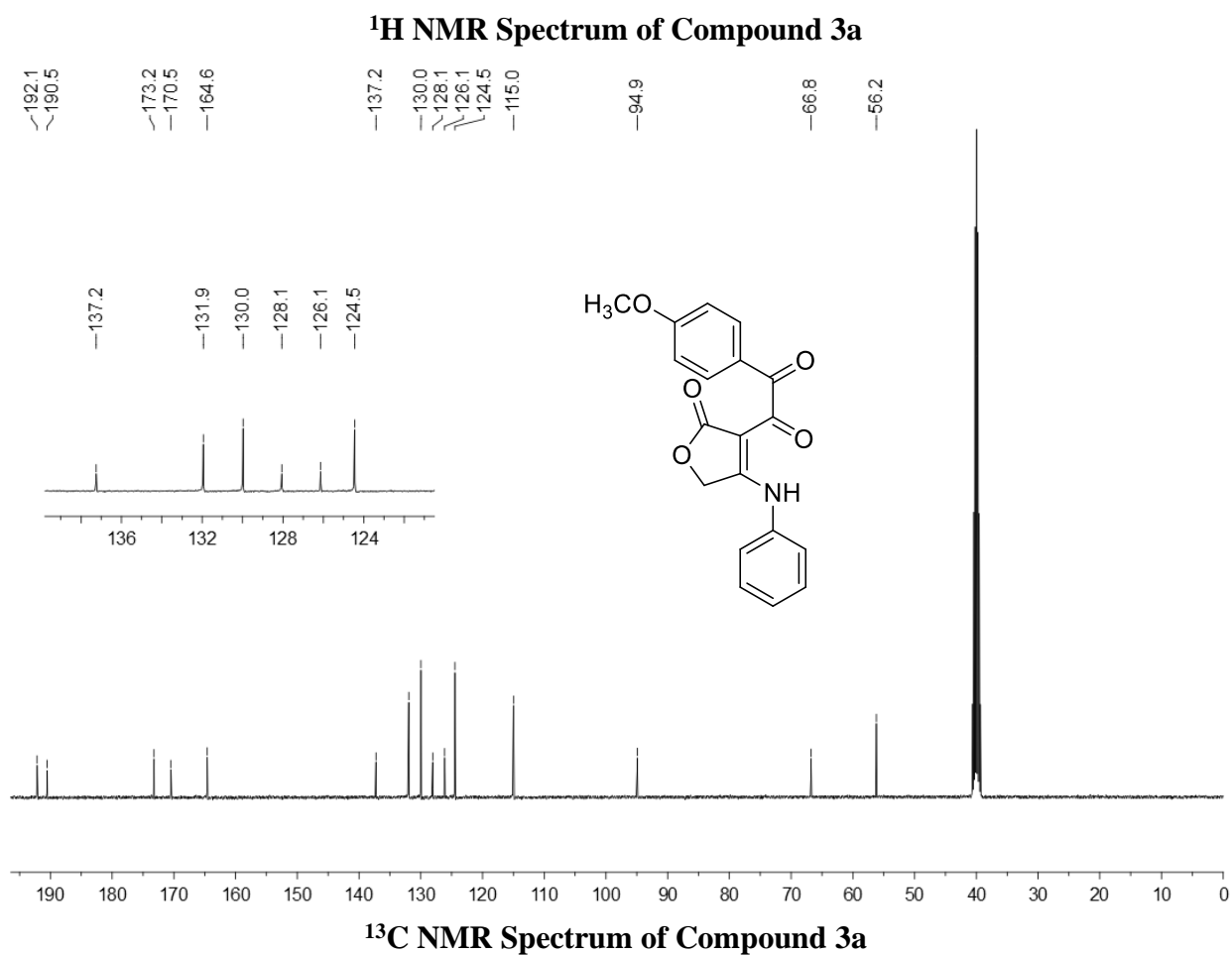
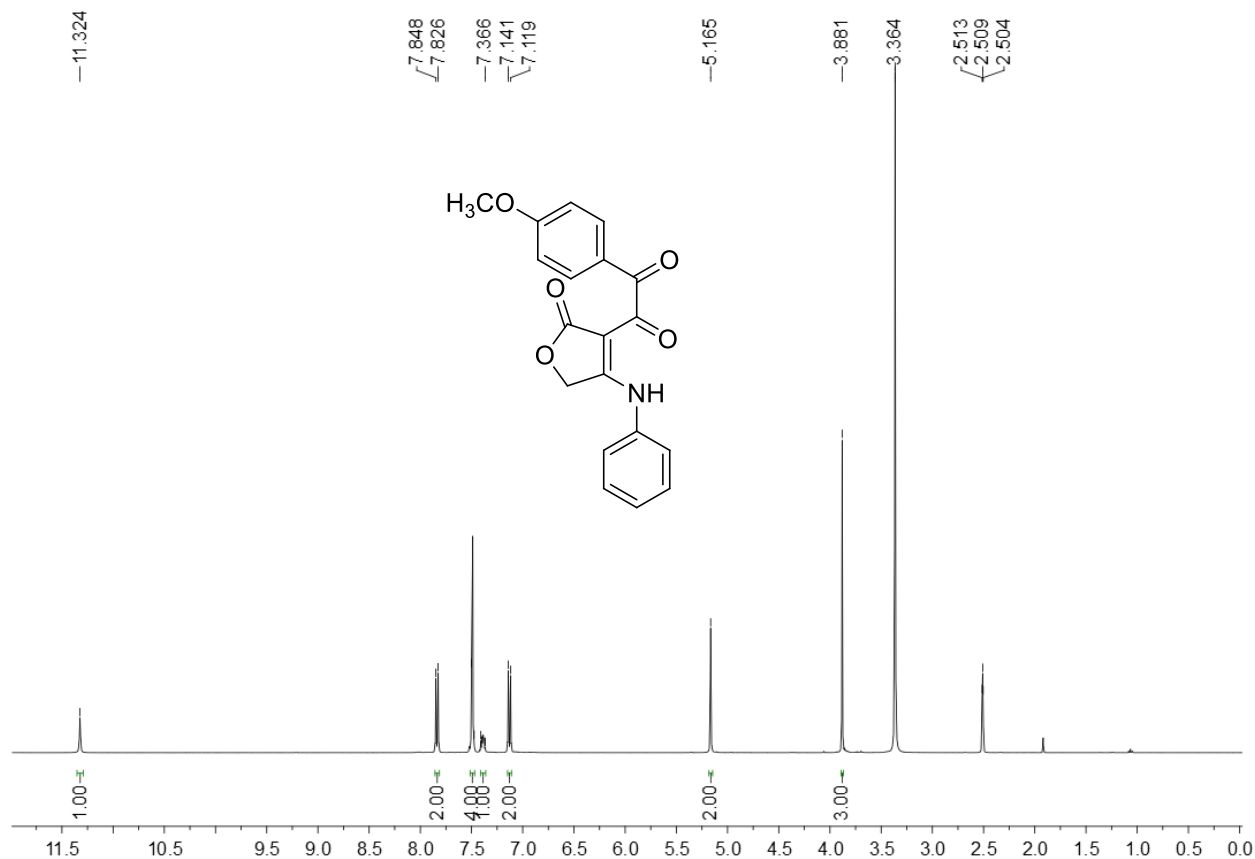
Crystal data for **3t** (CCDC: 2231948):  $\text{C}_{19}\text{H}_{15}\text{NO}_4$ ,  $M_r = 321.32$ , Monoclinic,  $a = 11.0238(9)$  Å,  $b = 5.9101(4)$  Å,  $c = 24.313(2)$  Å,  $U = 1568.4(2)$  Å<sup>3</sup>,  $T = 298(2)$  K, space group  $P2(1)/c$ ,  $Z = 4$ , 7459 reflections measured, 2725 unique ( $R_{\text{int}} = 0.0315$ ) which were used in all calculation. The final  $wR(F_2)$  was 0.1094 (all data)



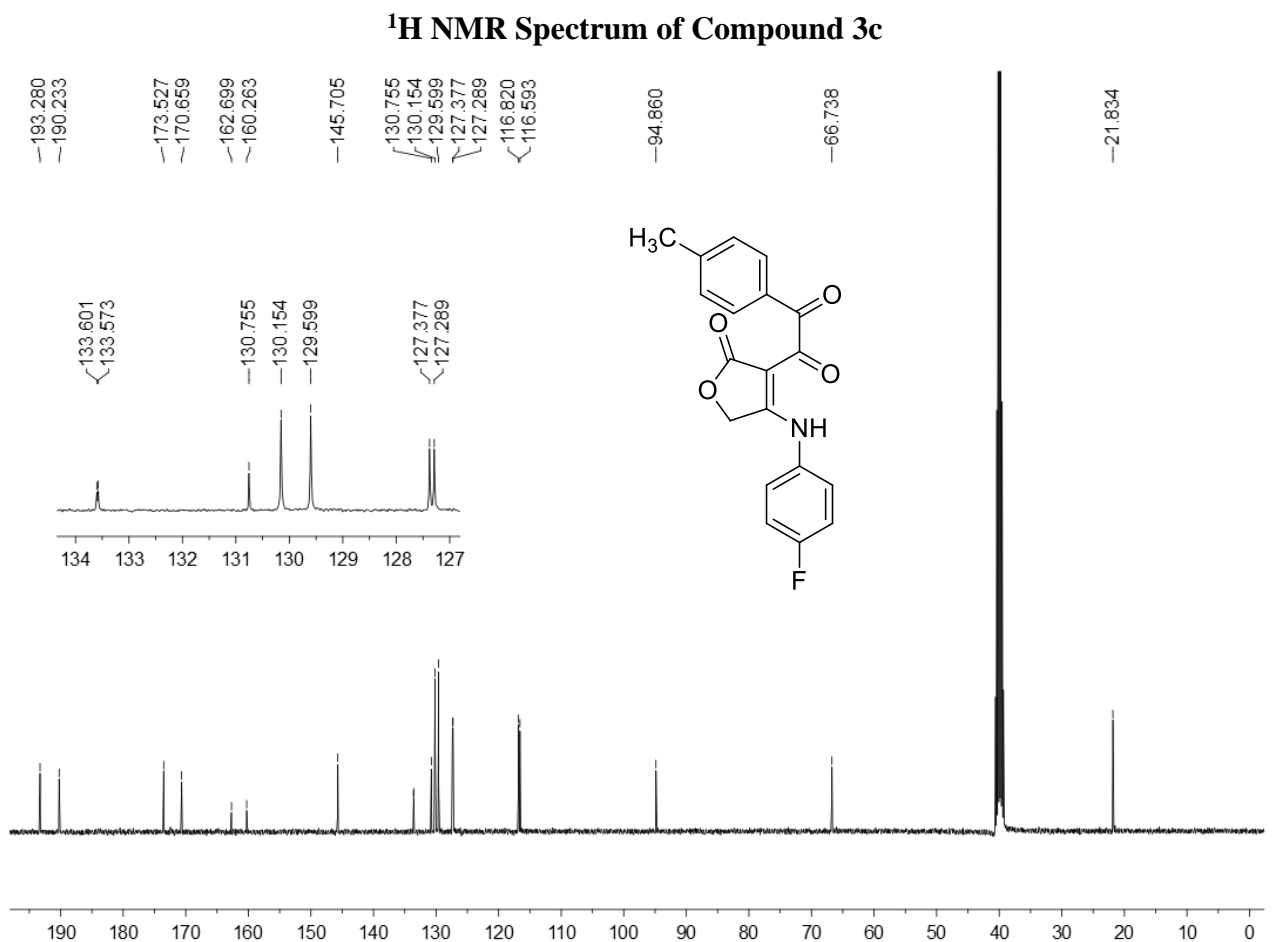
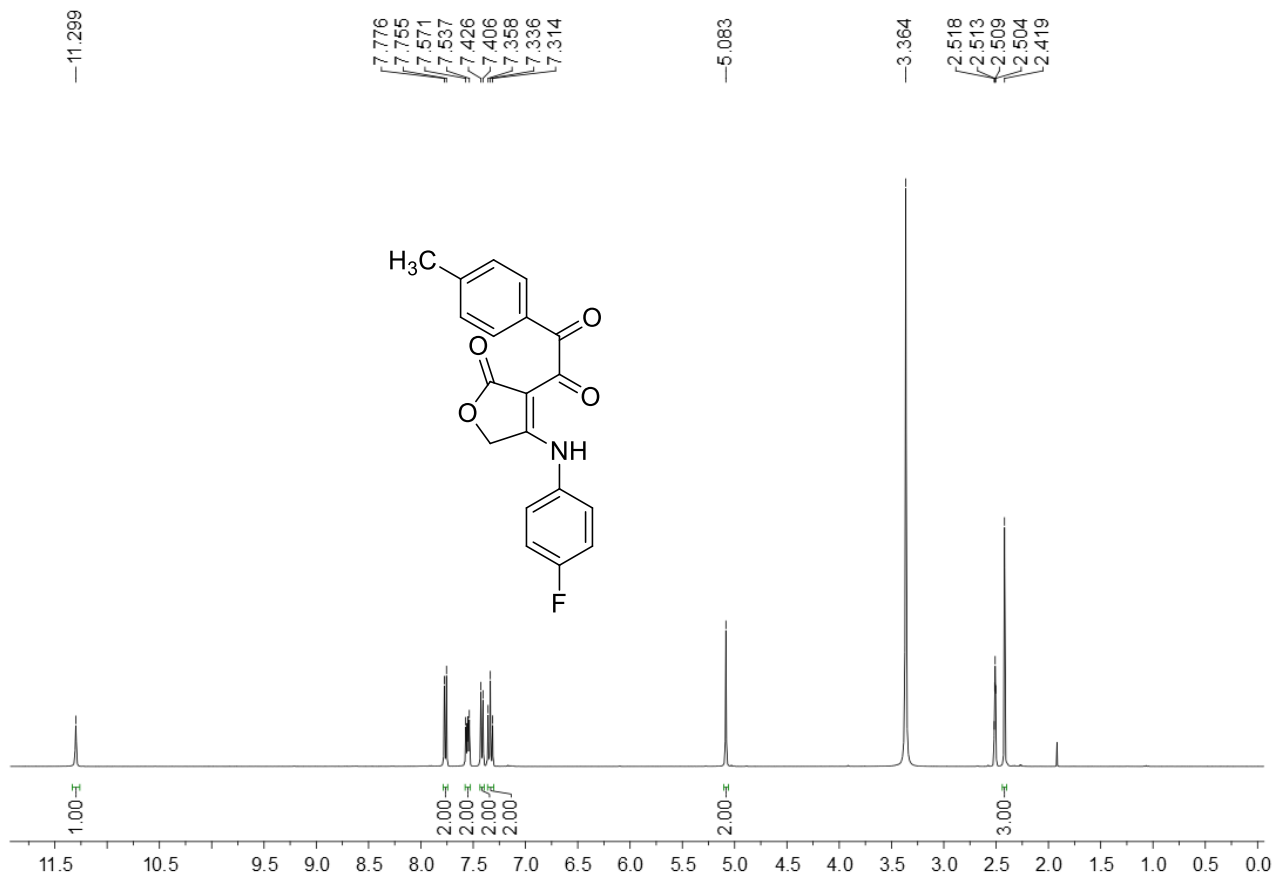
**Fig 3, X-ray Structure of intermediate A**

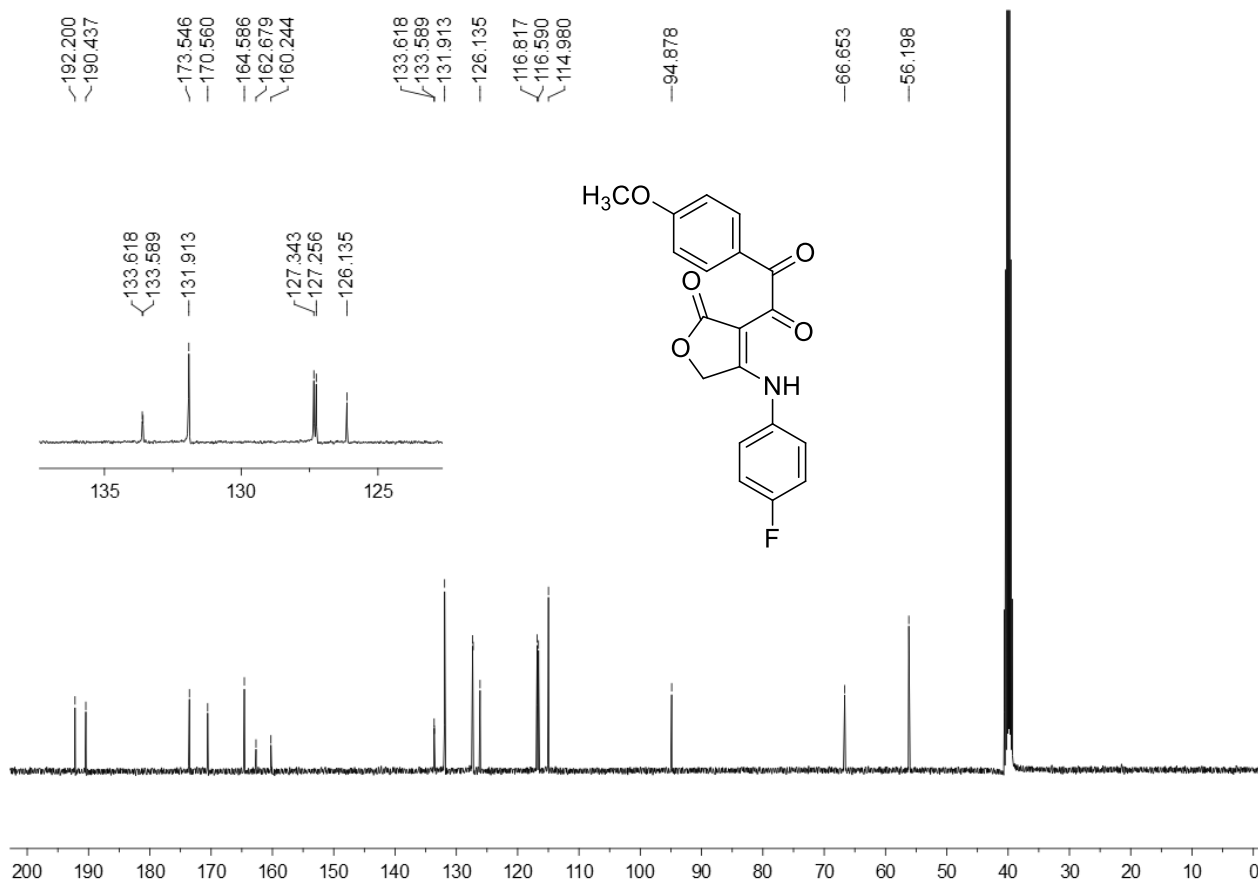
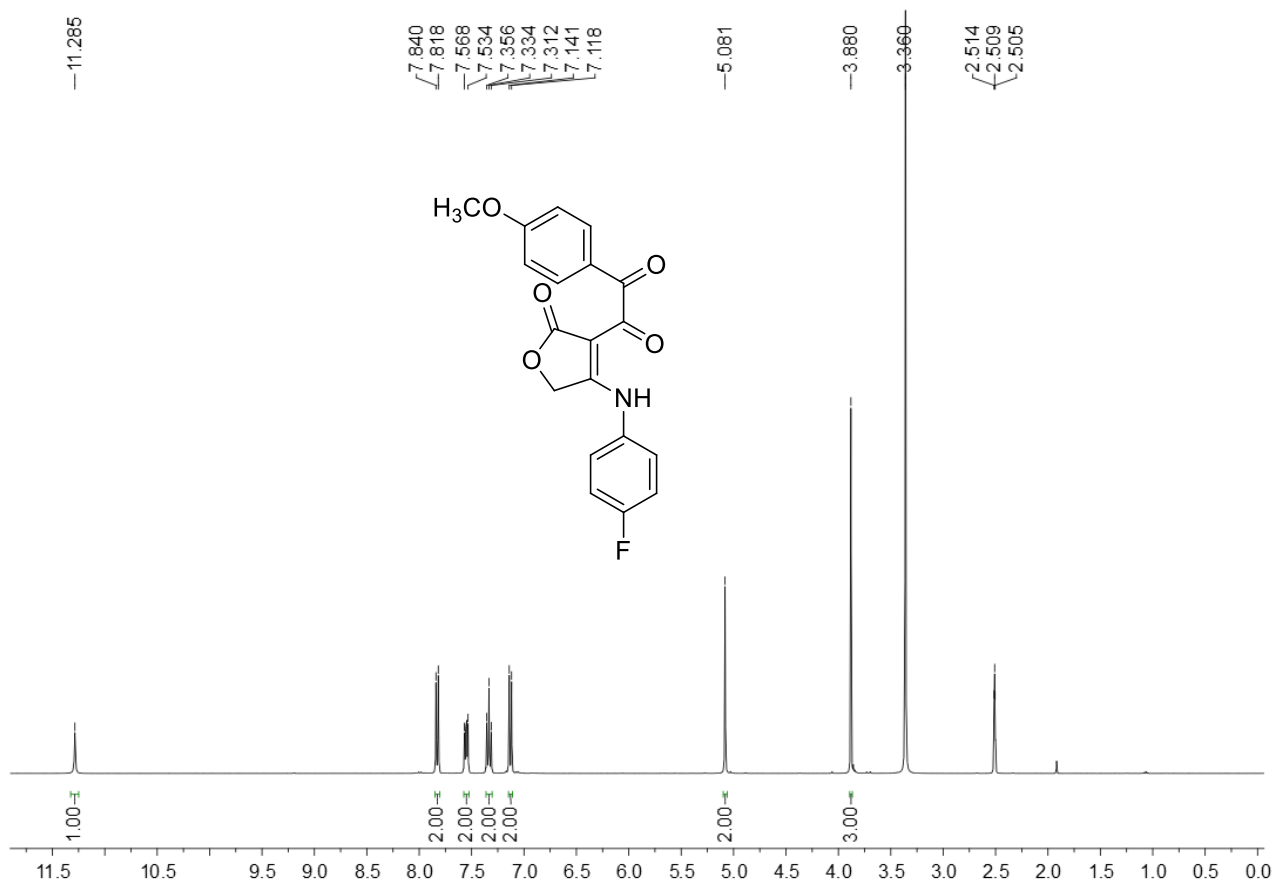
Crystal data for intermediate A (CCDC: 2232494):  $C_{18}H_{13}BrClNO_4$ ,  $M_r = 422.65$ , Monoclinic,  $a = 25.531(2) \text{ \AA}$ ,  $b = 11.3558(11) \text{ \AA}$ ,  $c = 12.1460(12) \text{ \AA}$ ,  $U = 3478.4(6) \text{ \AA}^3$ ,  $T = 293(2) \text{ K}$ , space group  $C2/c$ ,  $Z = 8$ , 9003 reflections measured, 3077 unique ( $R_{\text{int}} = 0.1907$ ) which were used in all calculation. The final  $wR(F_2)$  was 0.2132 (all data)

Copies of Spectra for Products **3a-3ff**

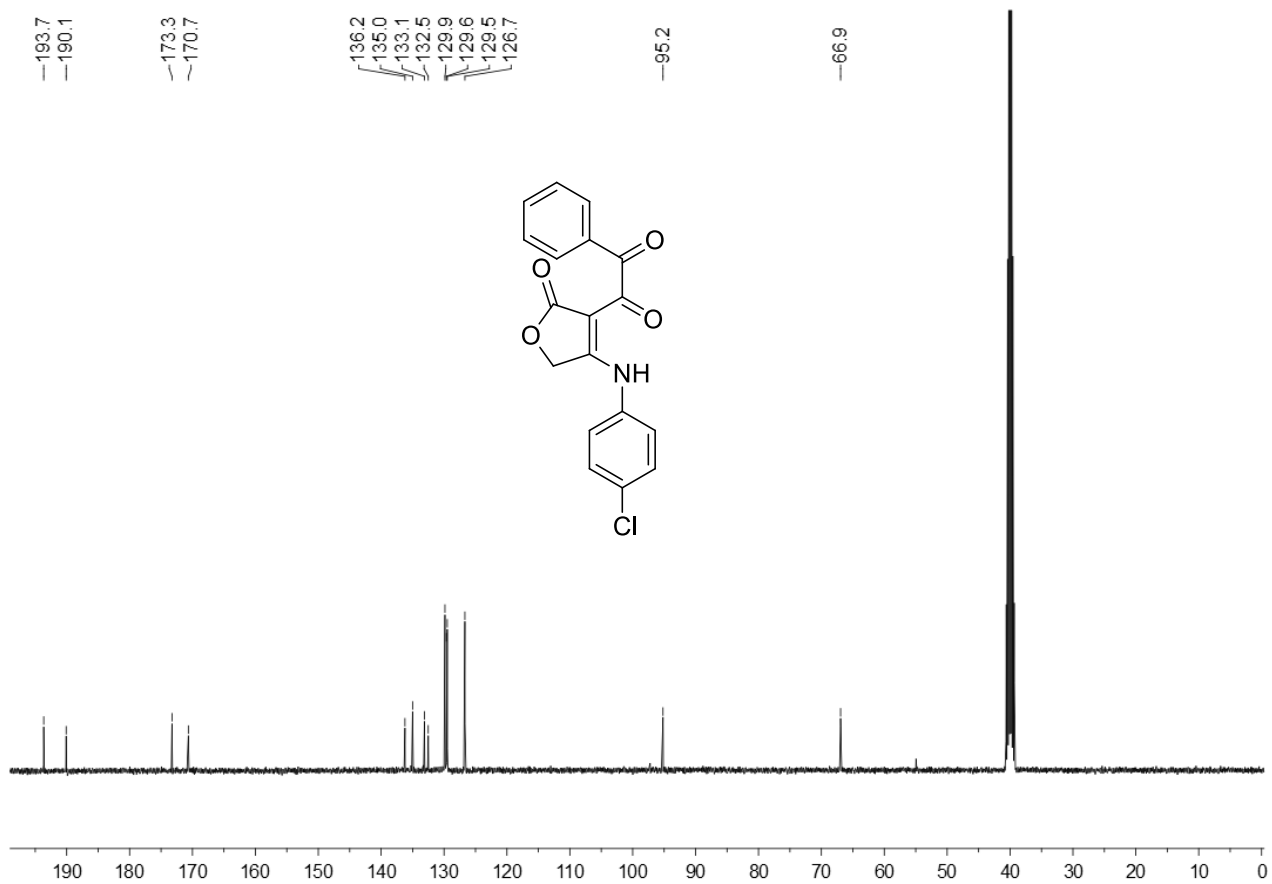
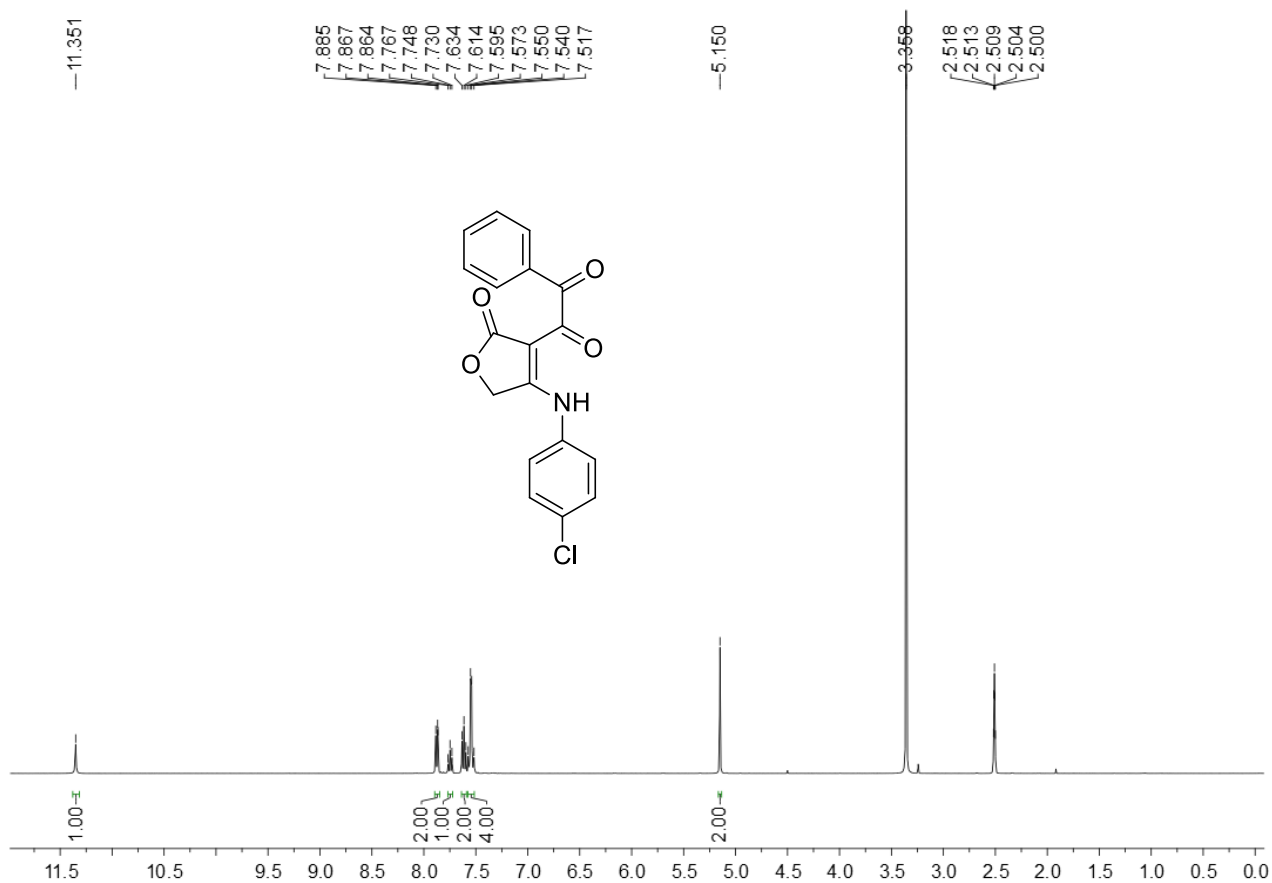


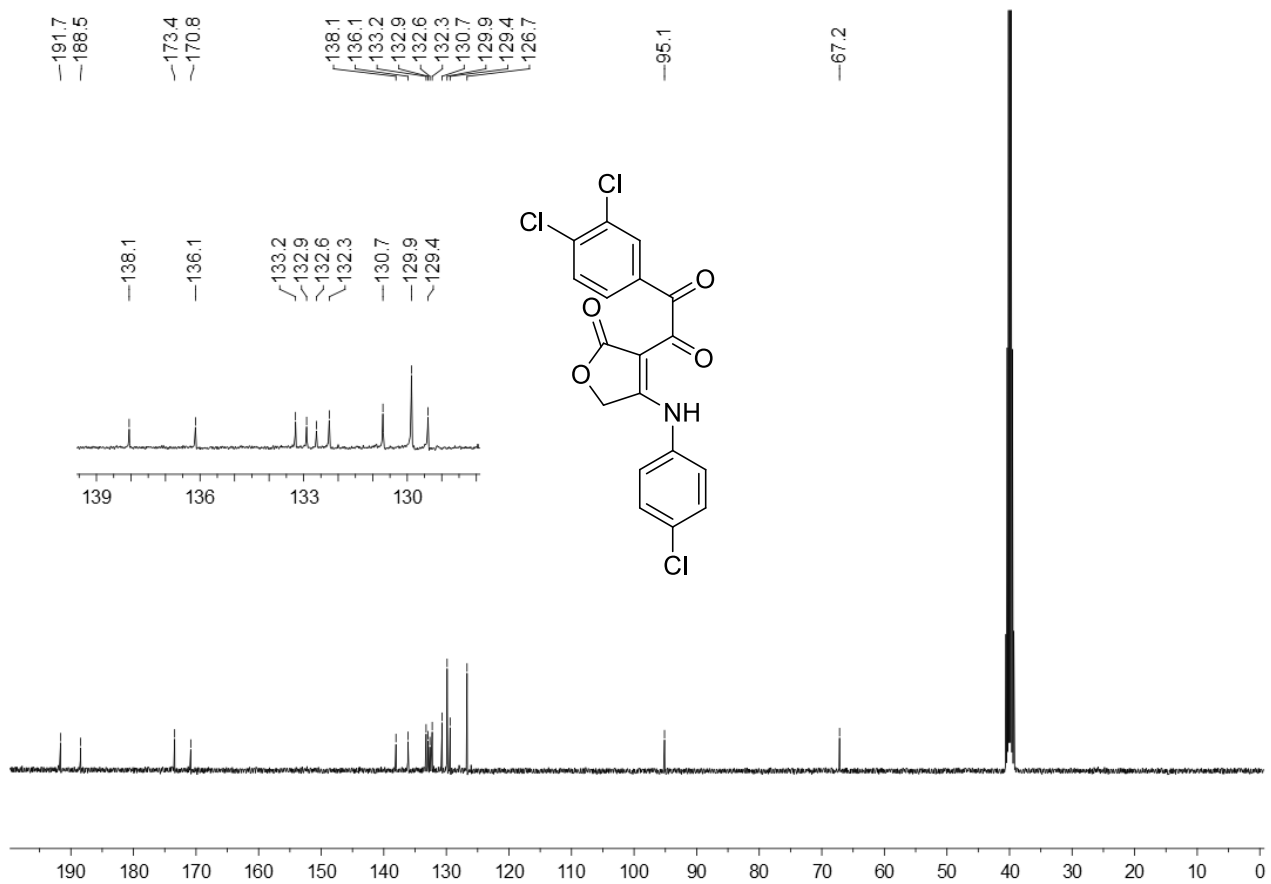
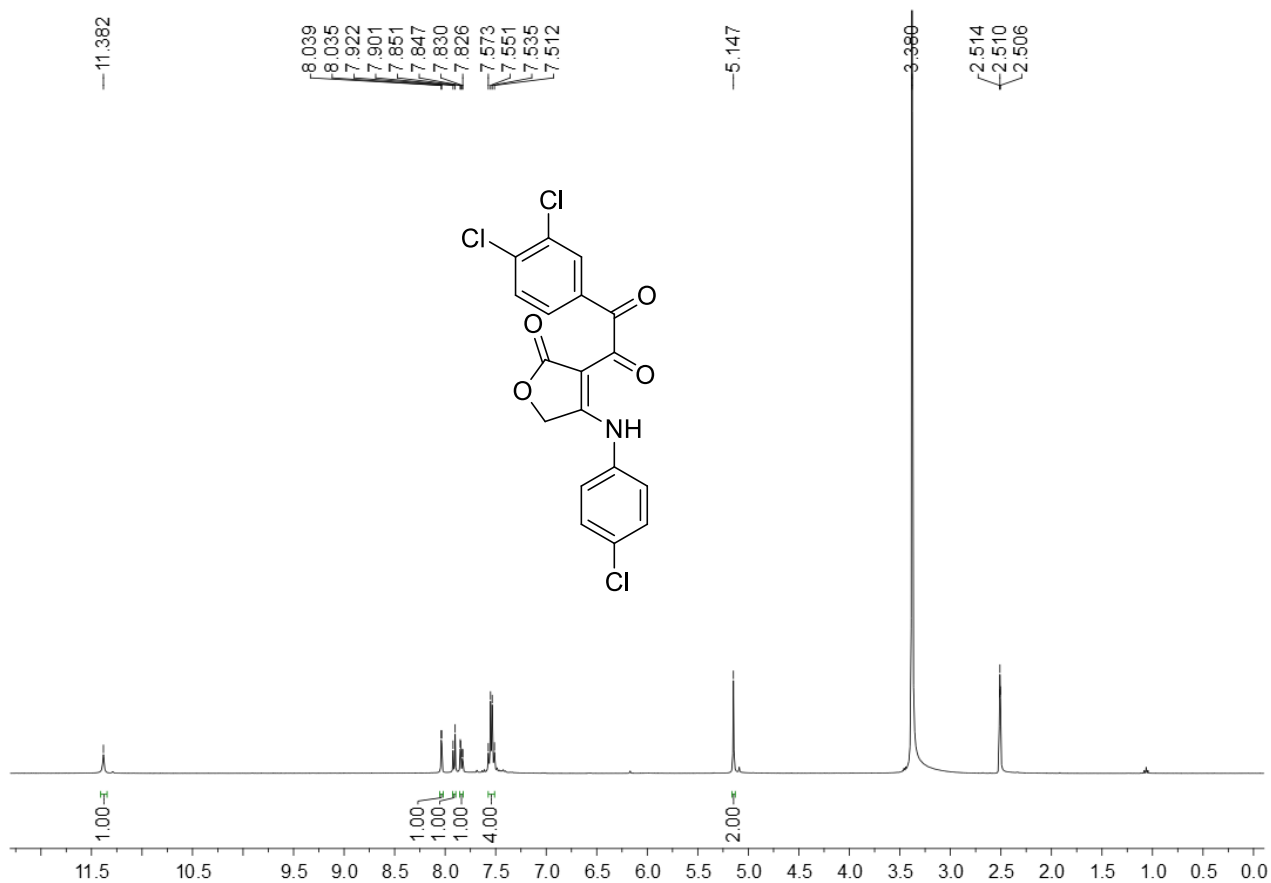




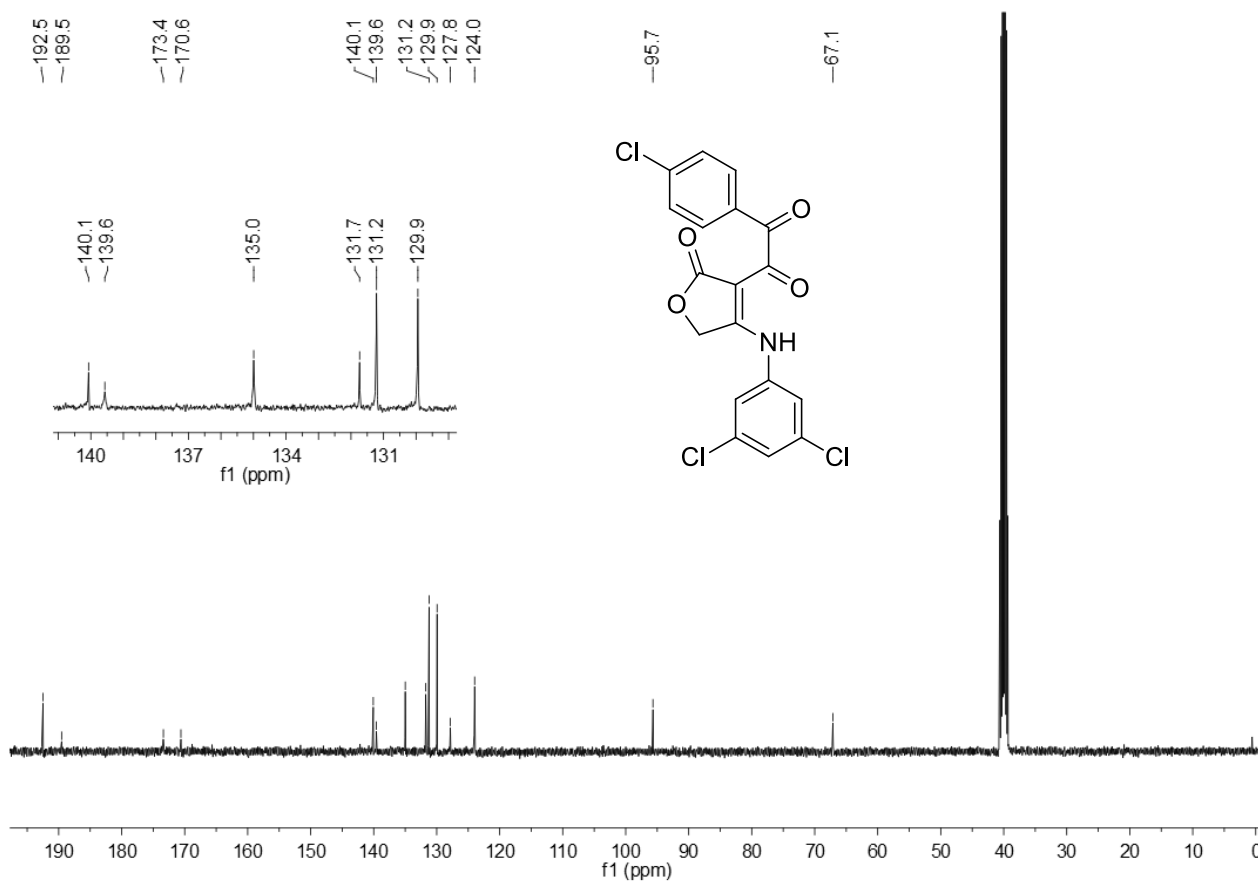
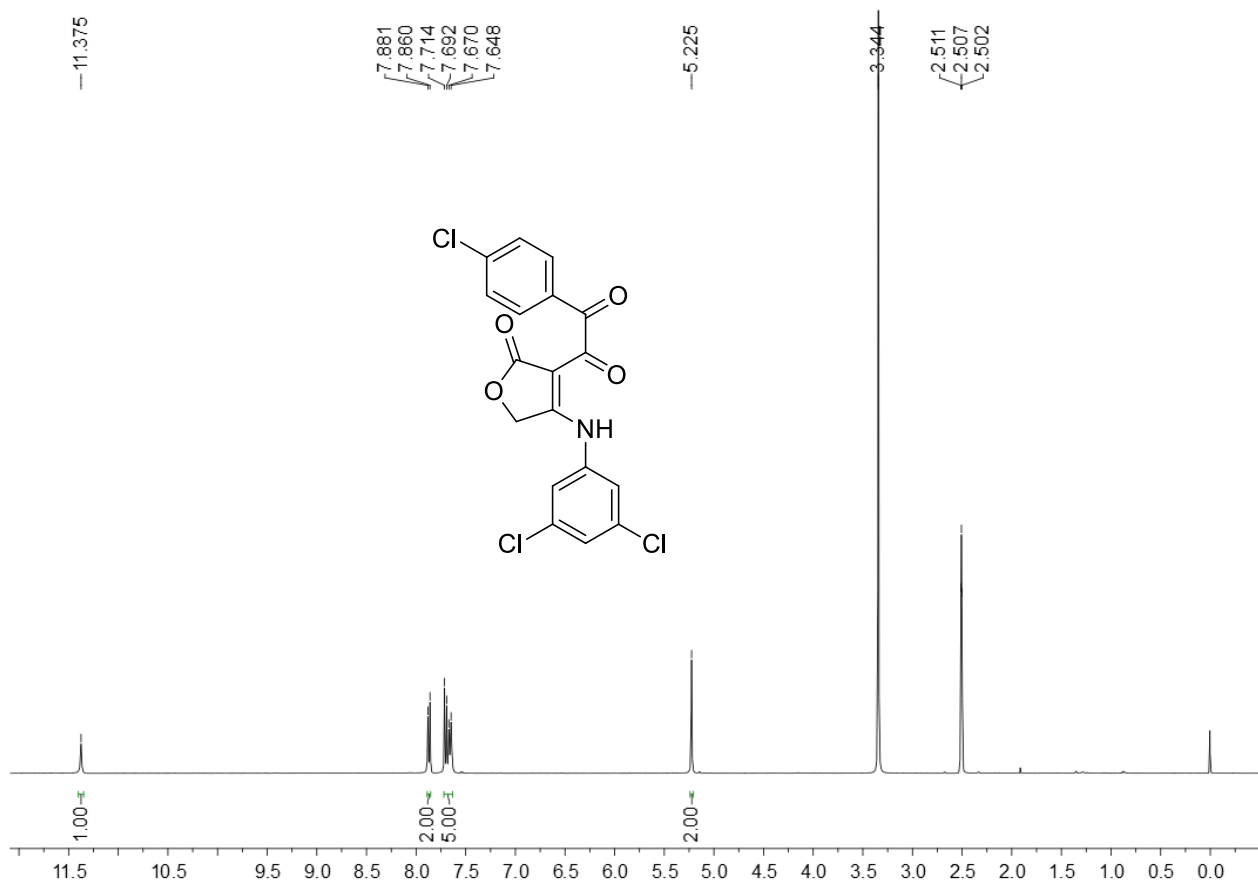


**<sup>13</sup>C NMR Spectrum of Compound 3d**

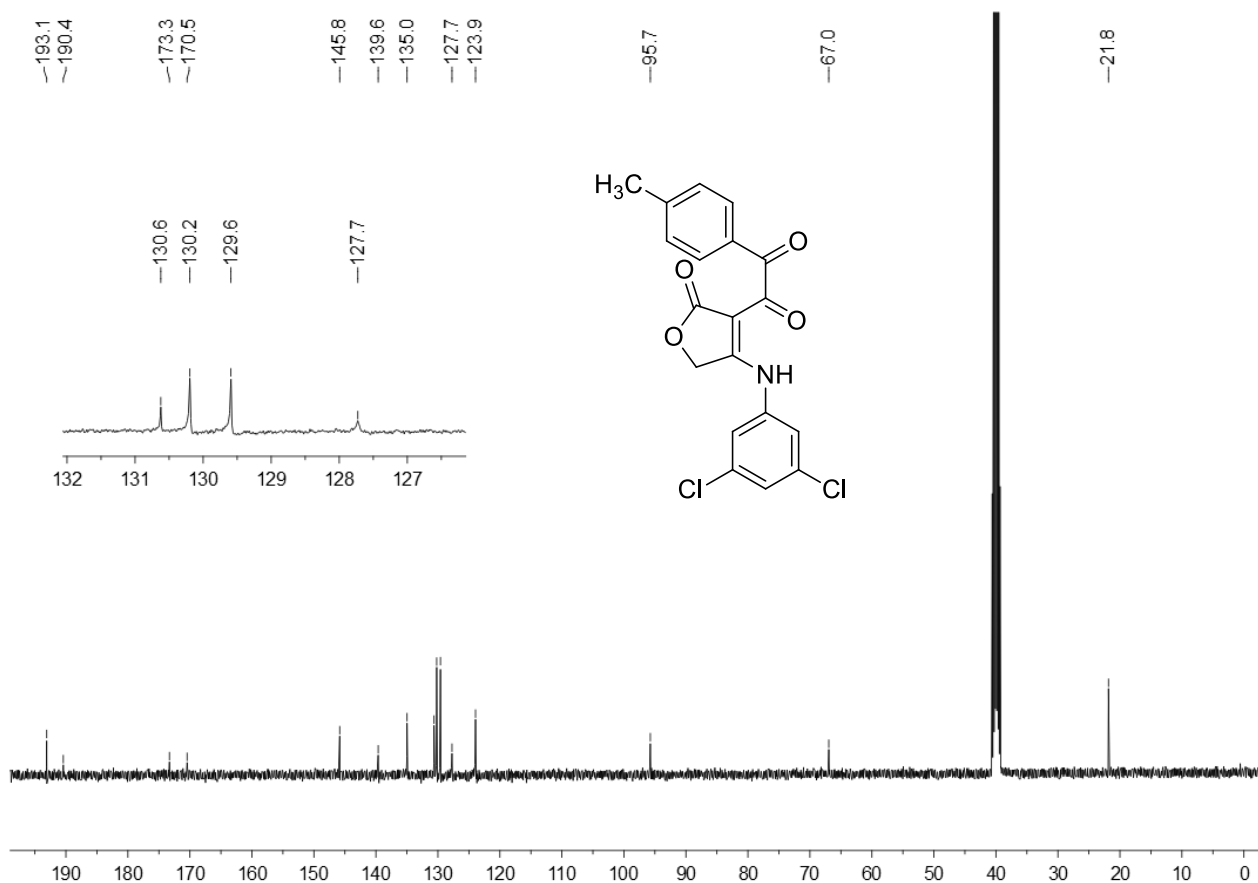
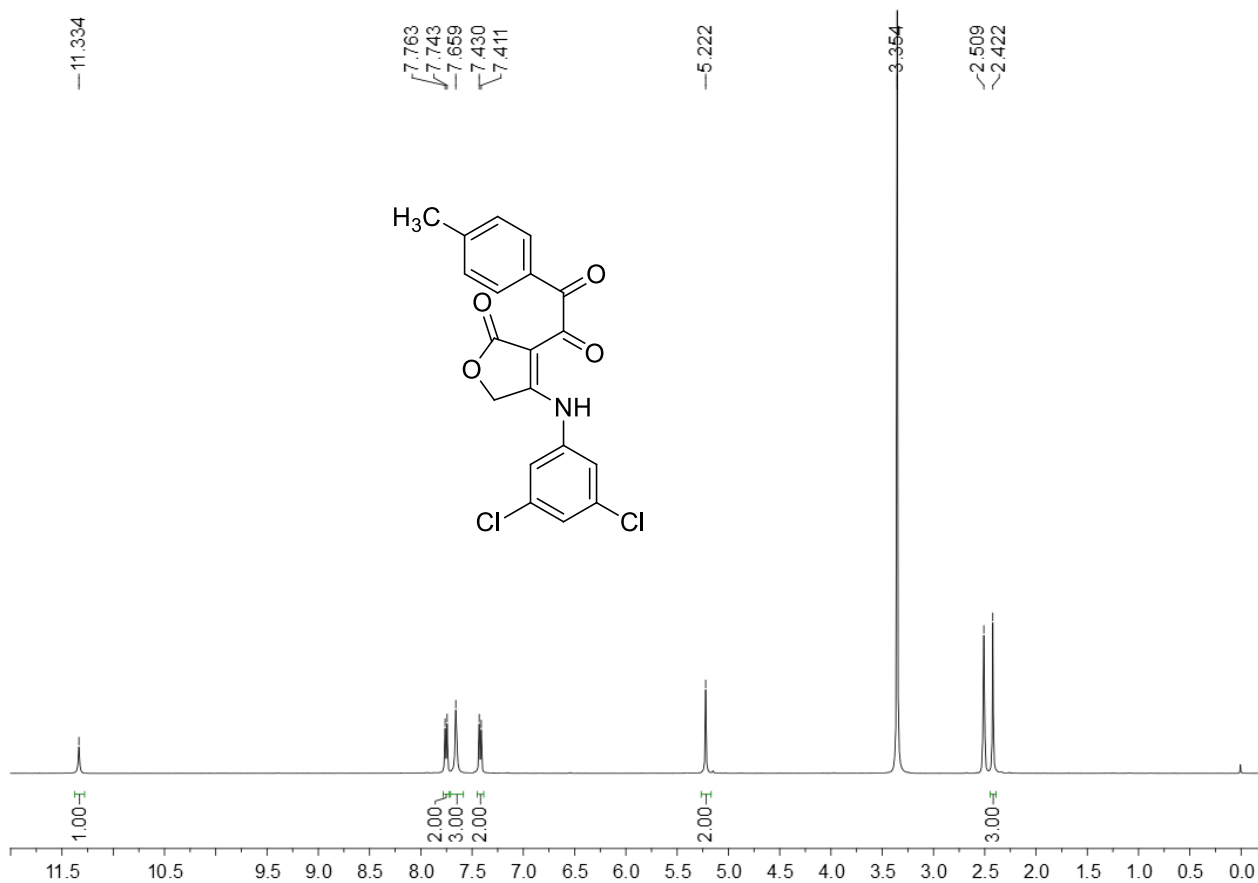


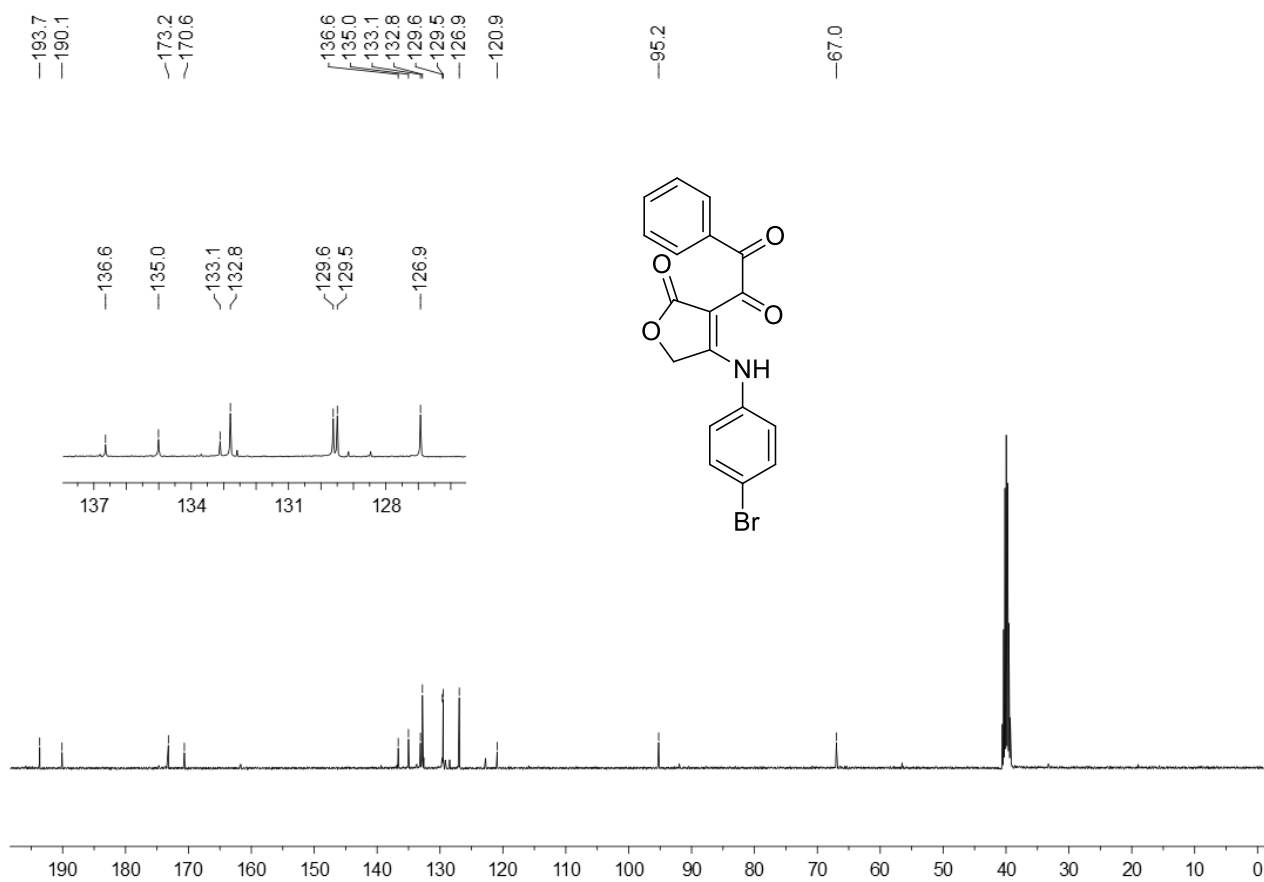
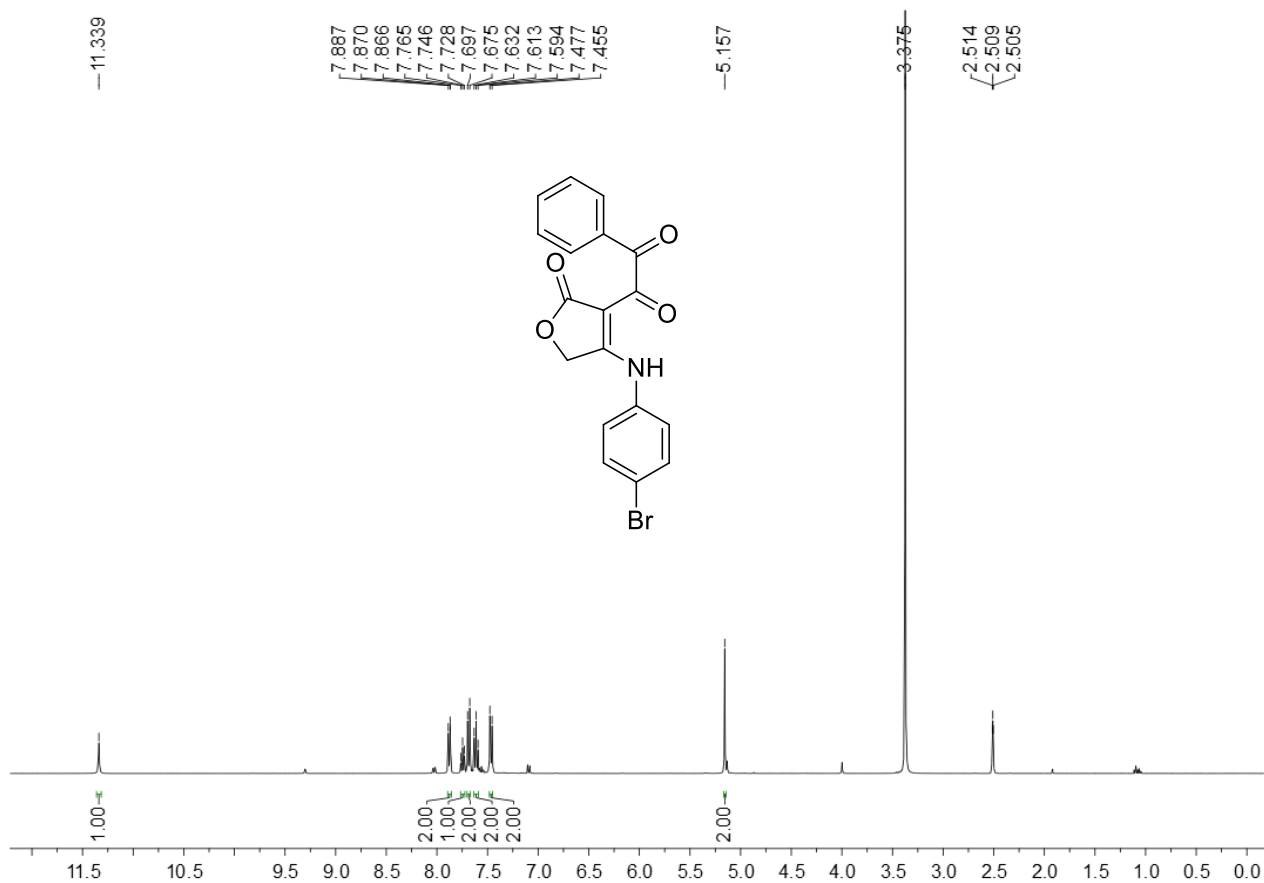


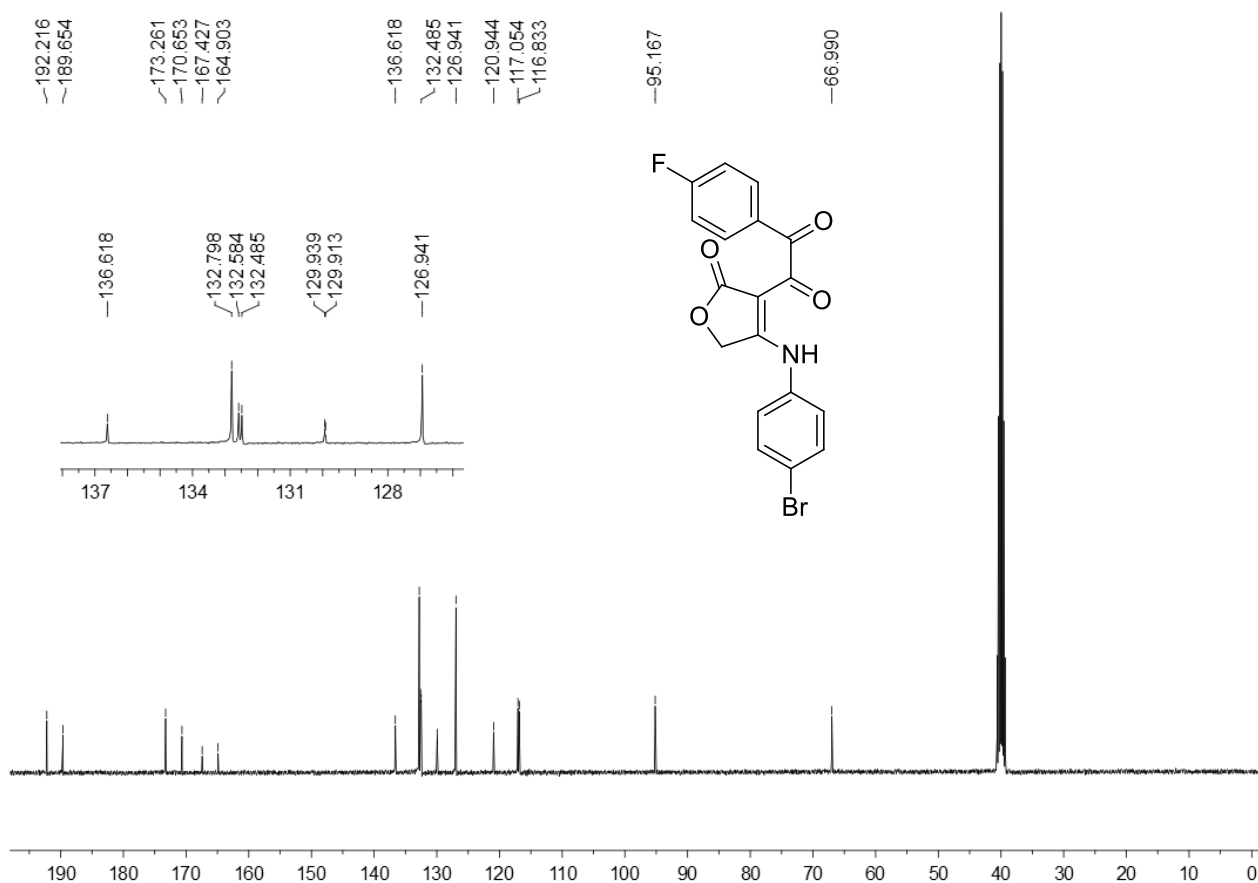
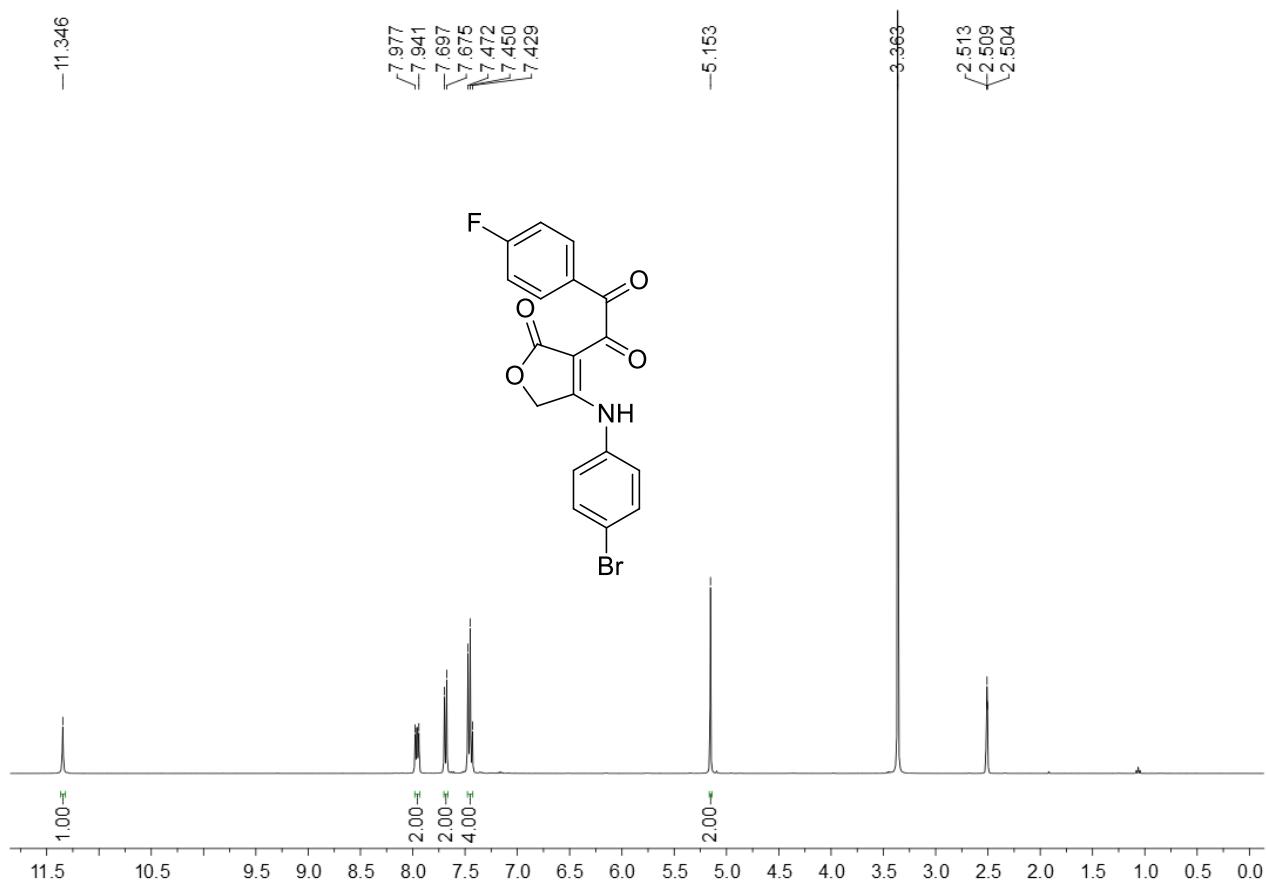
**<sup>13</sup>C NMR Spectrum of Compound 3f**



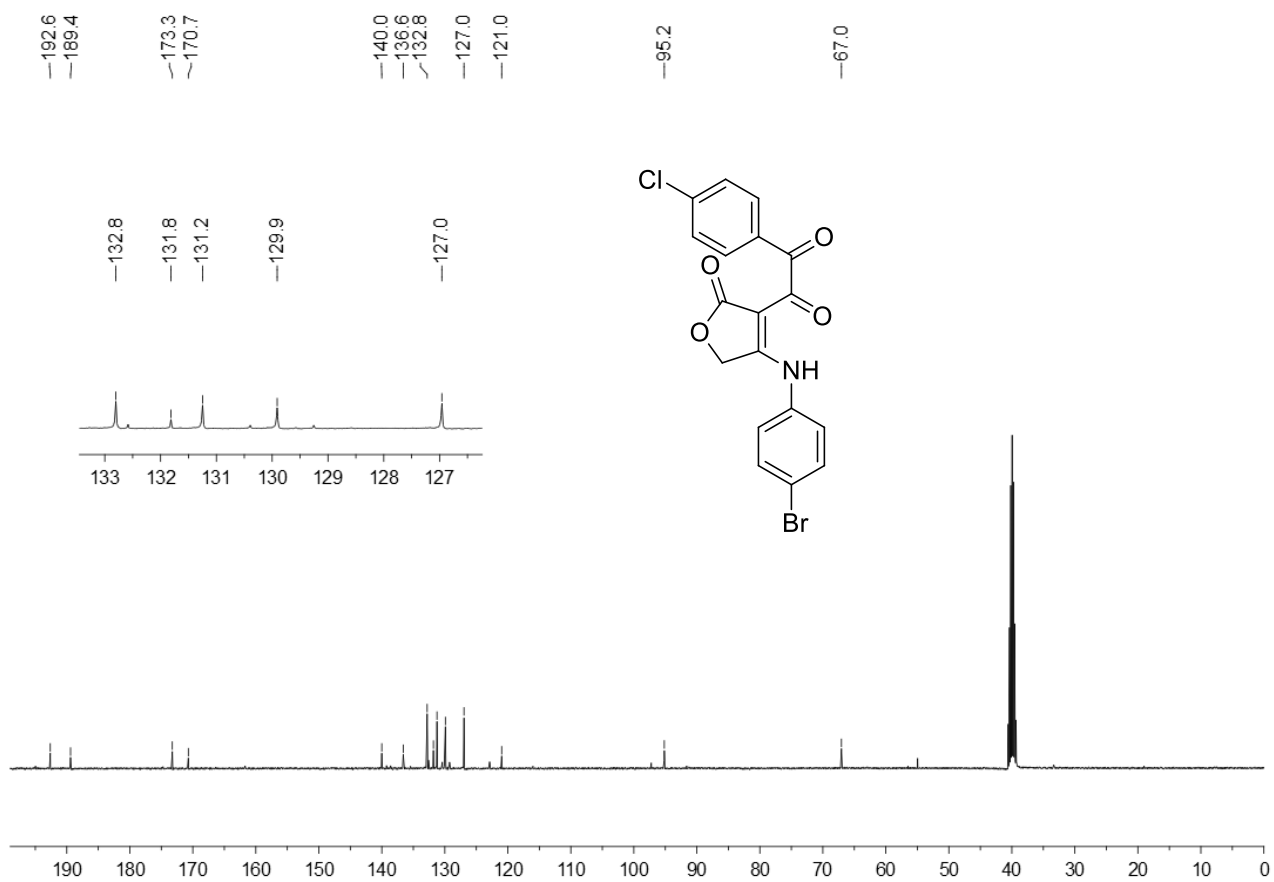
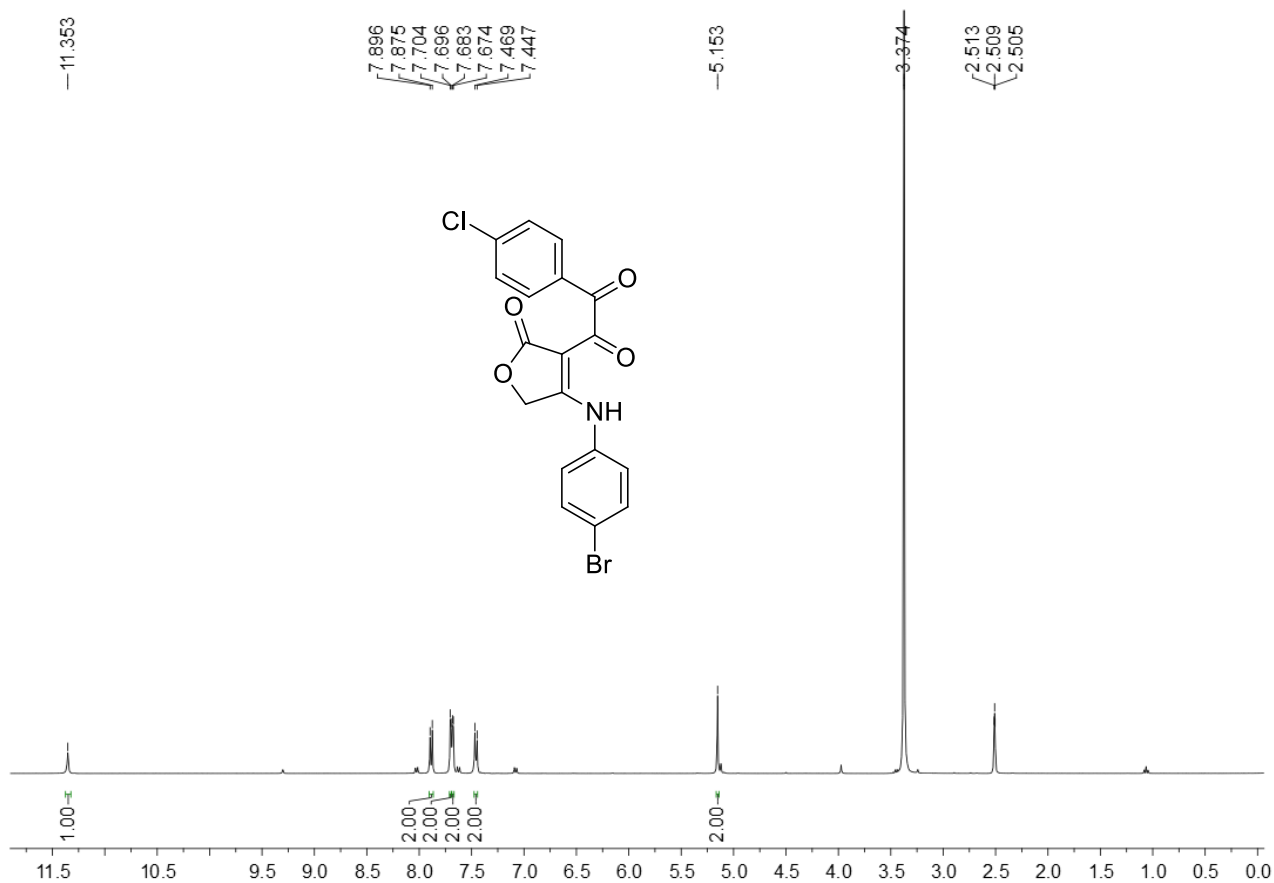
**<sup>13</sup>C NMR Spectrum of Compound 3g**

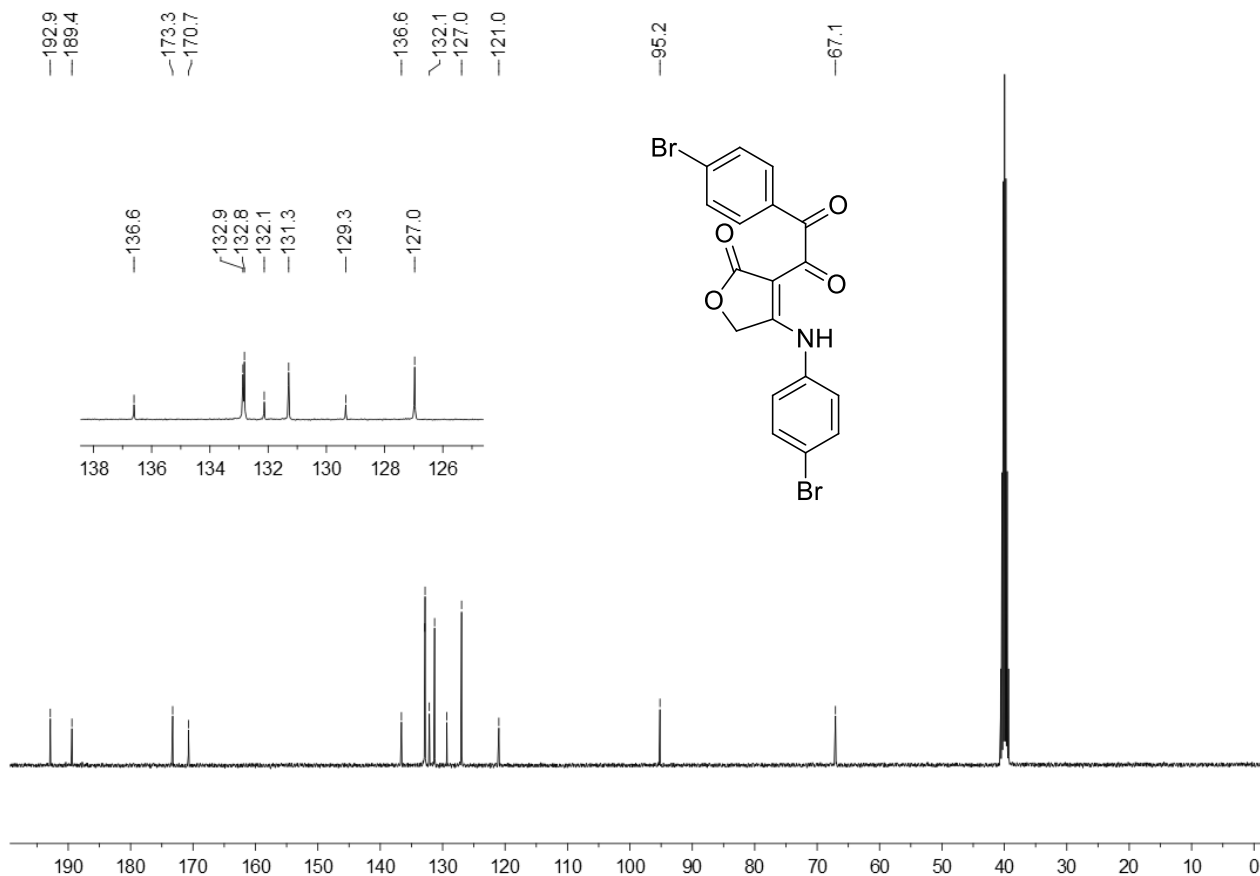
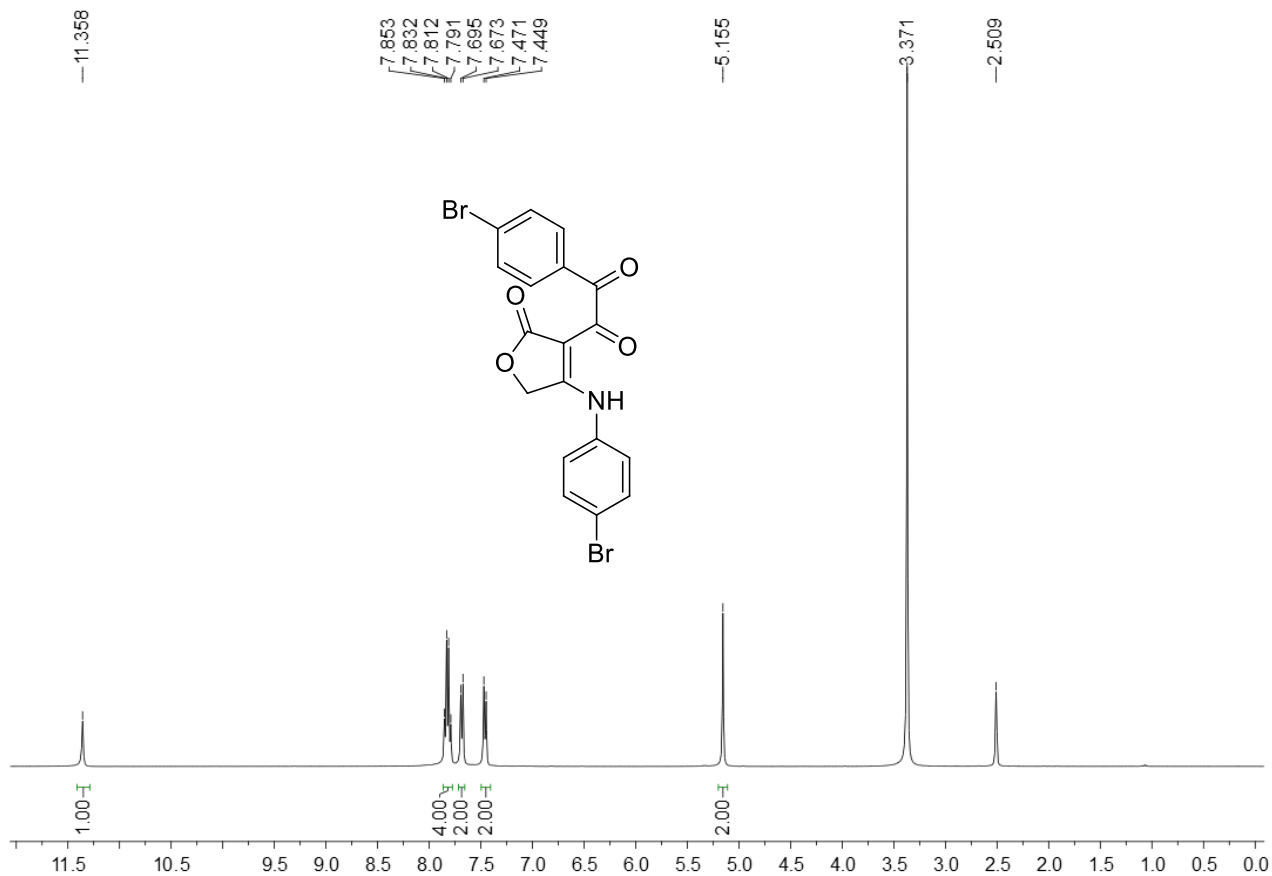




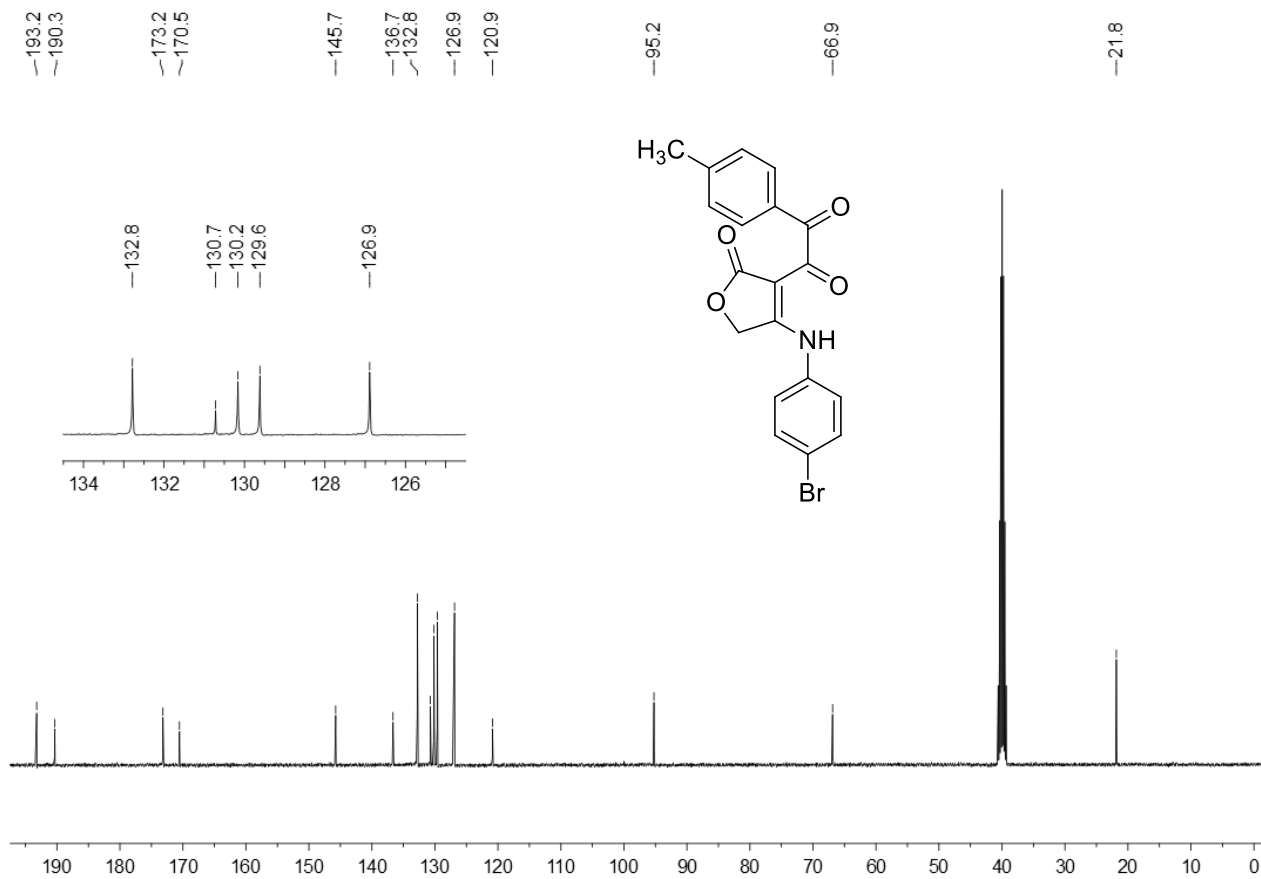
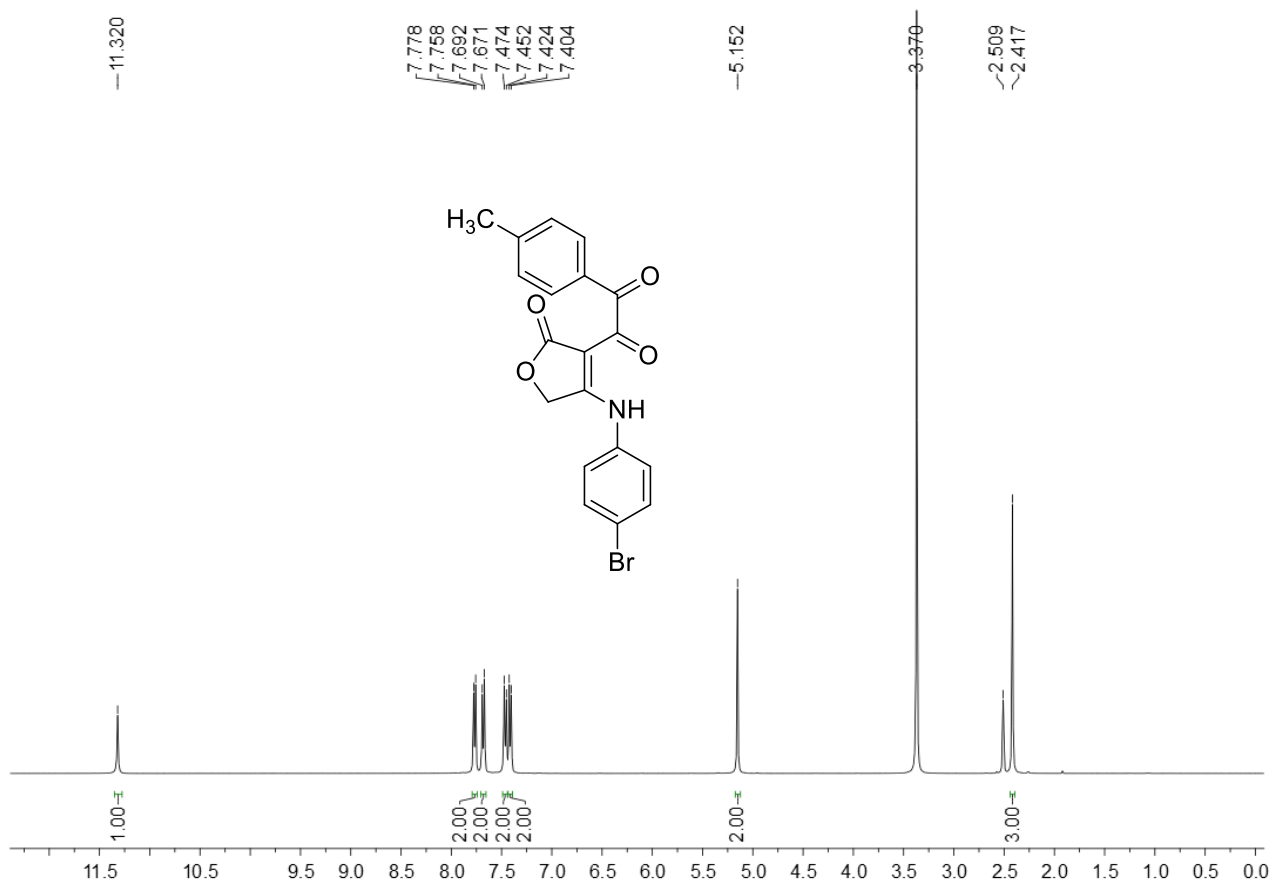


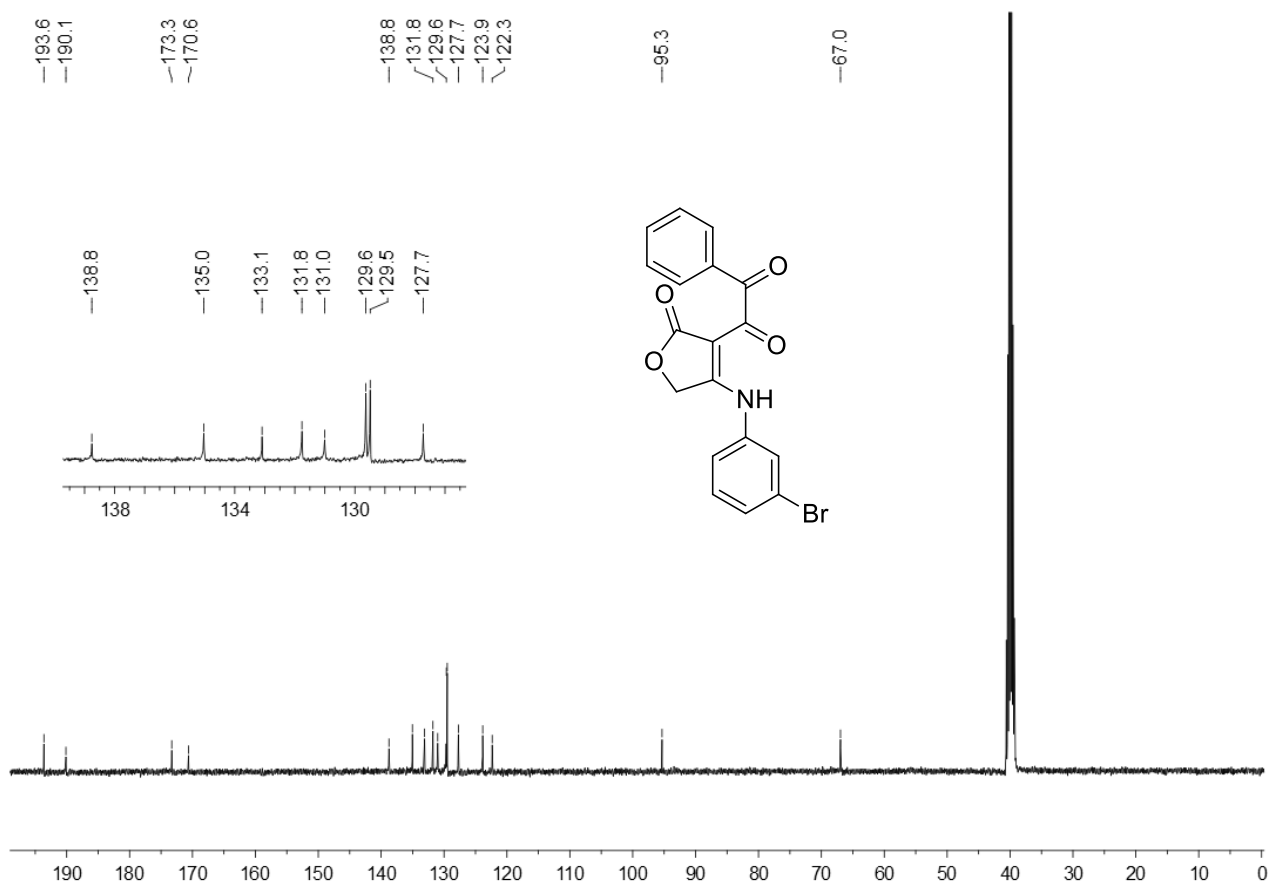
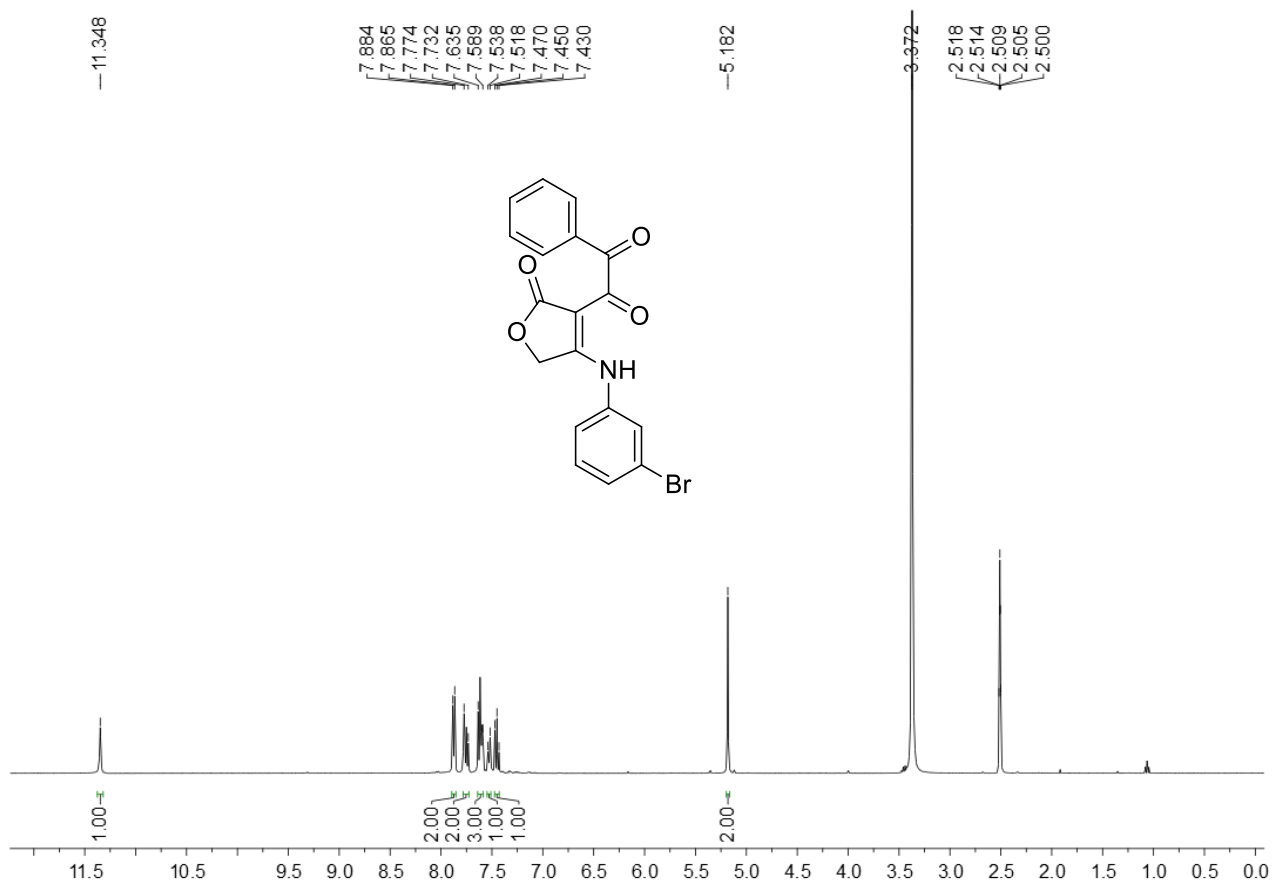
**<sup>13</sup>C NMR Spectrum of Compound 3j**



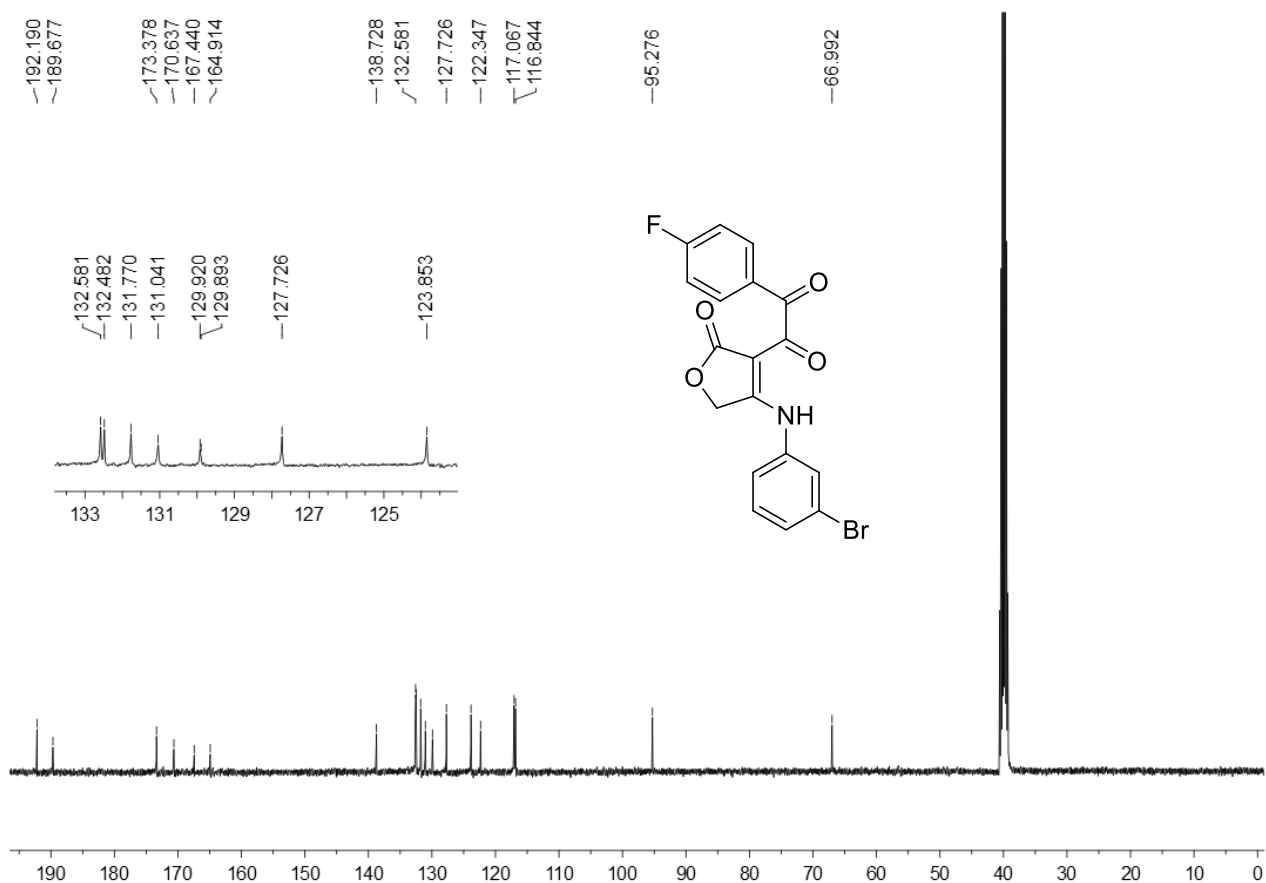
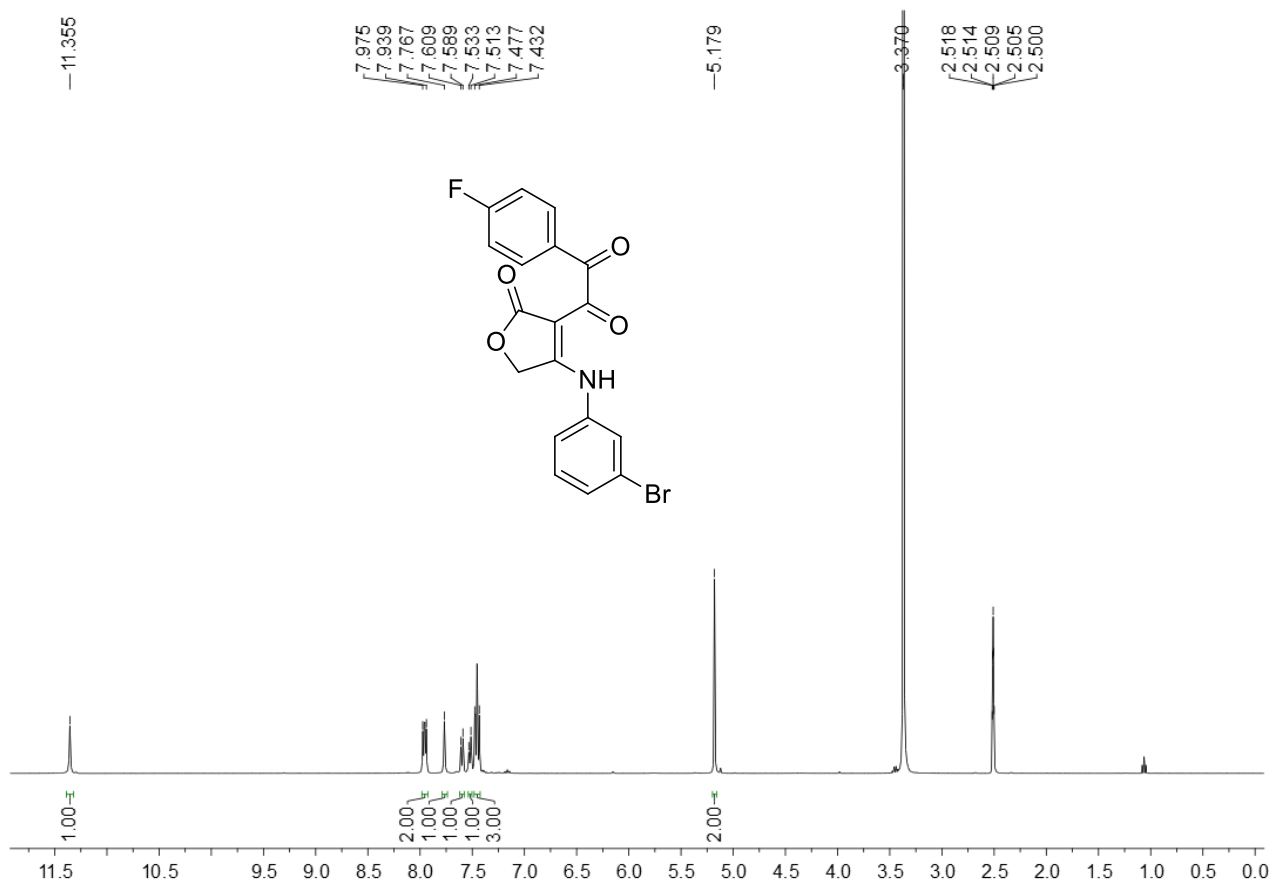


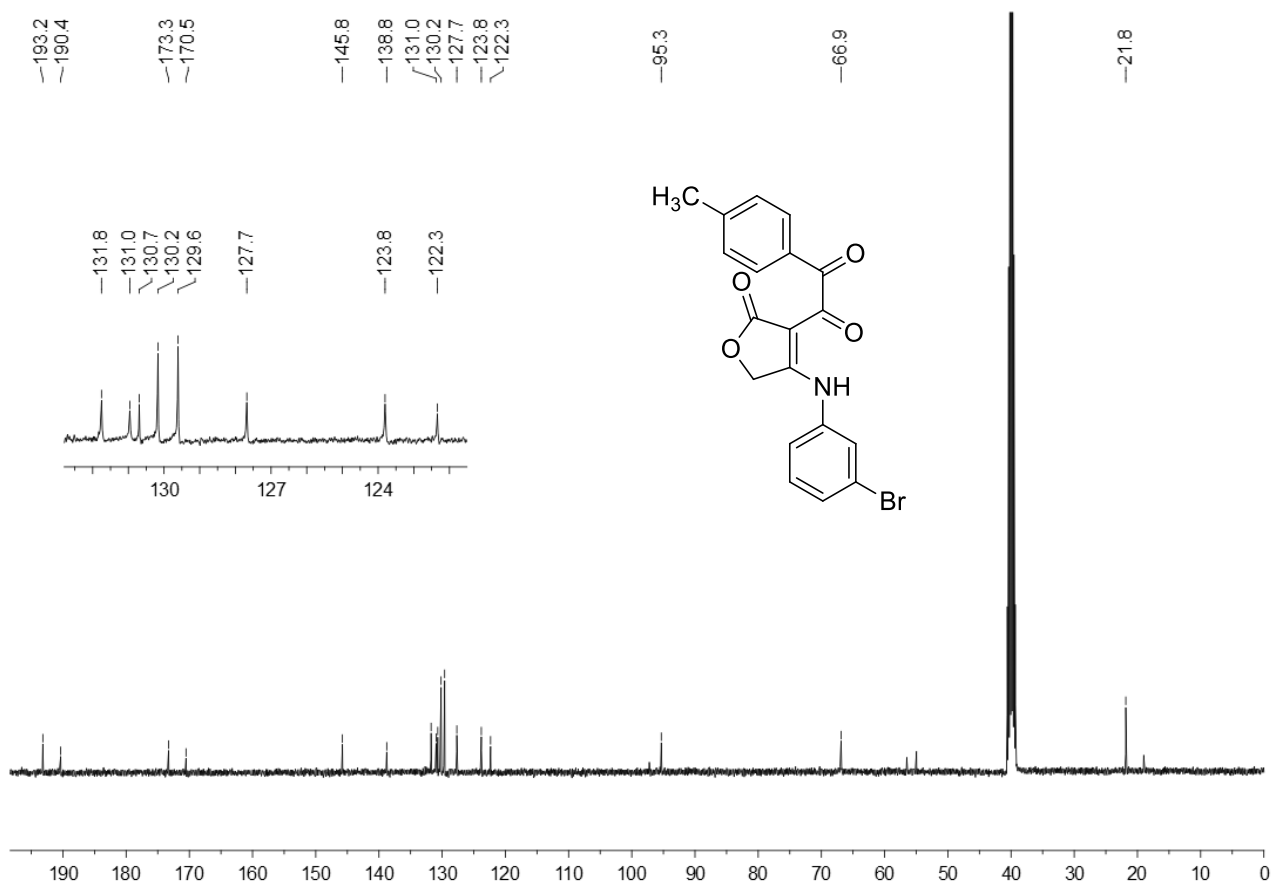
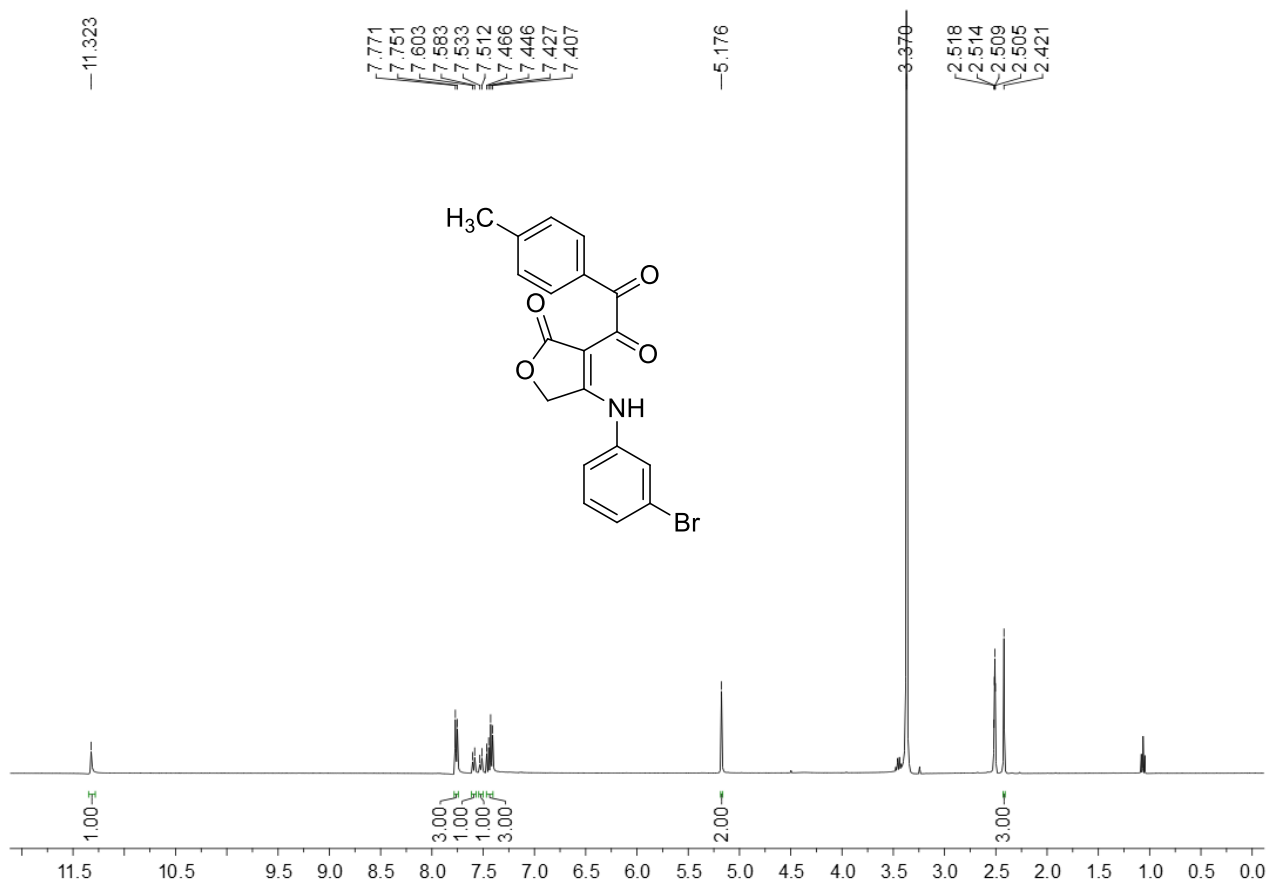
**<sup>13</sup>C NMR Spectrum of Compound 31**

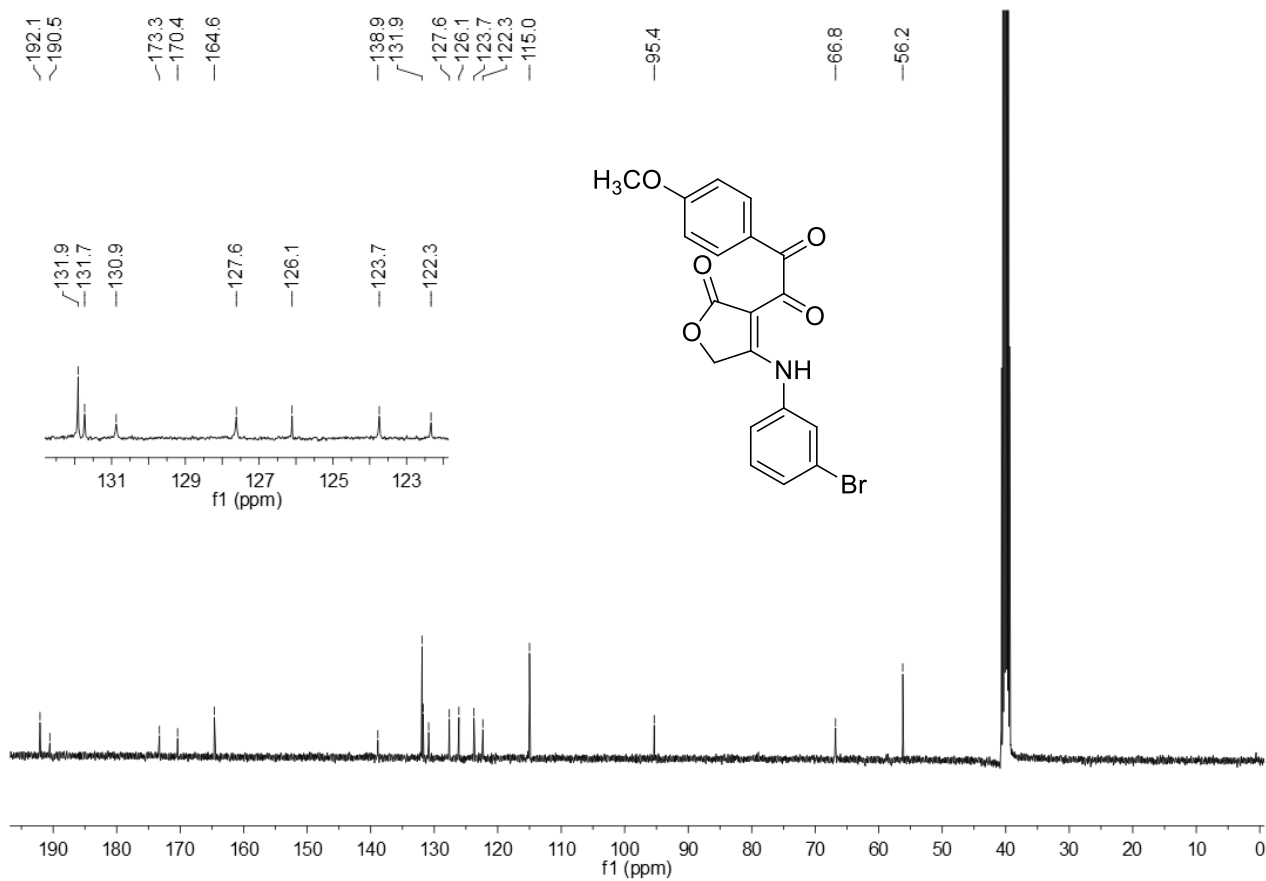
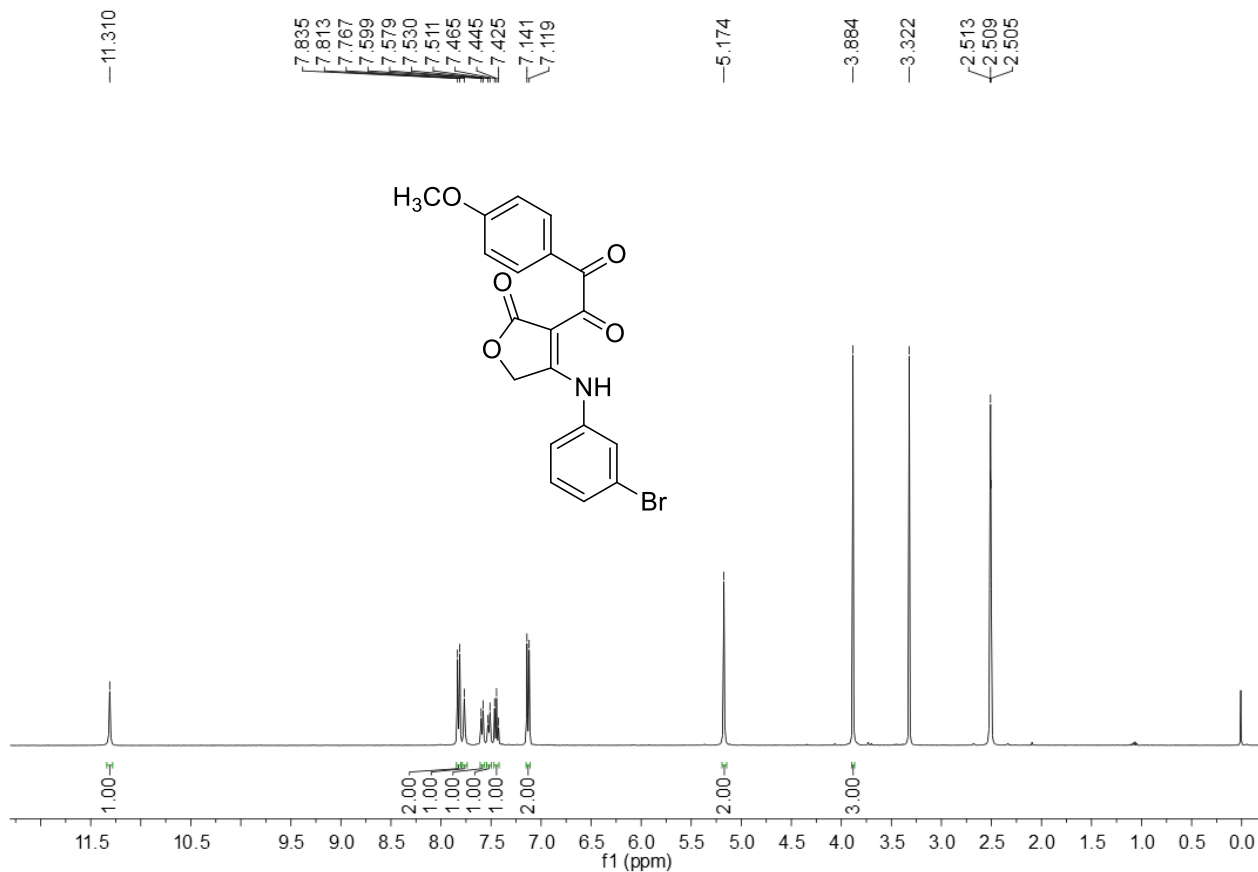




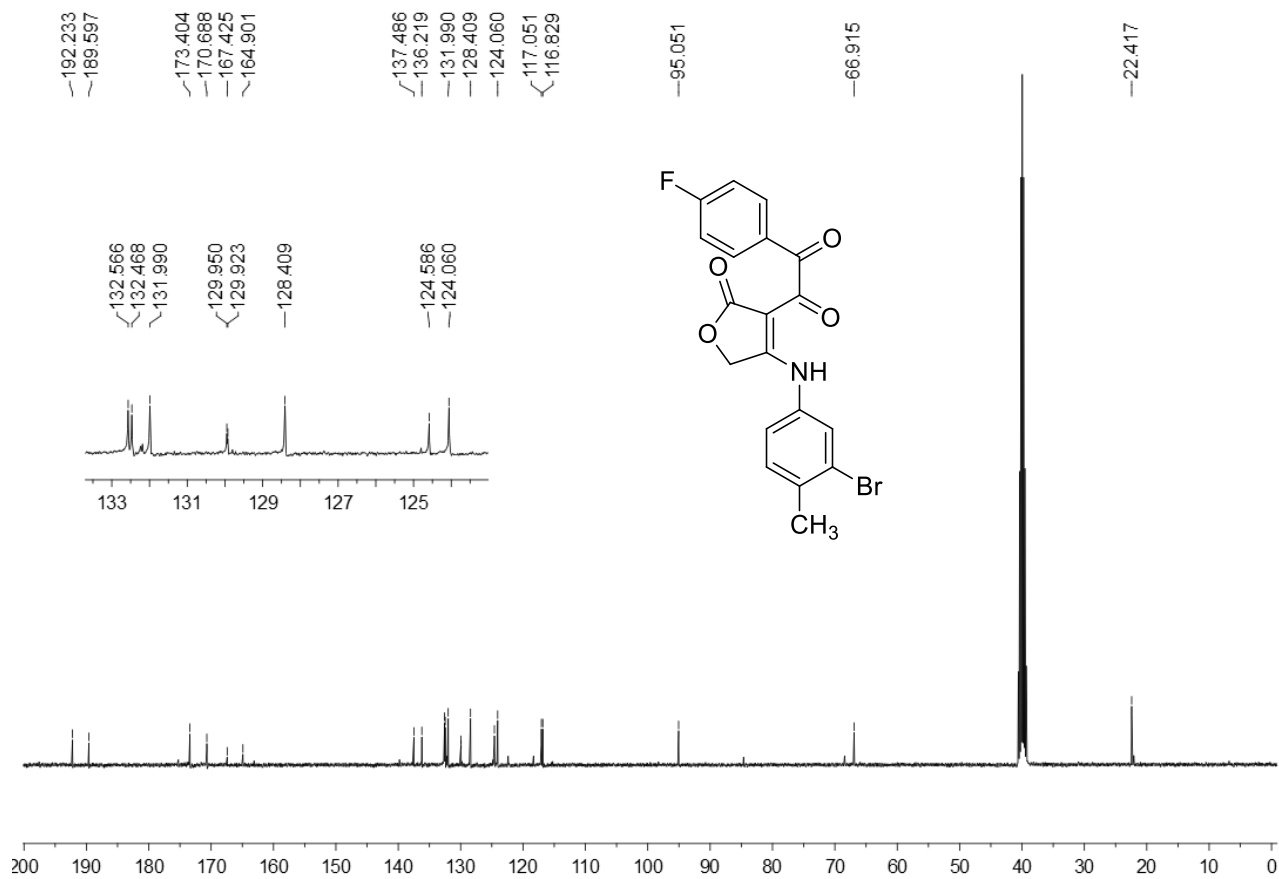
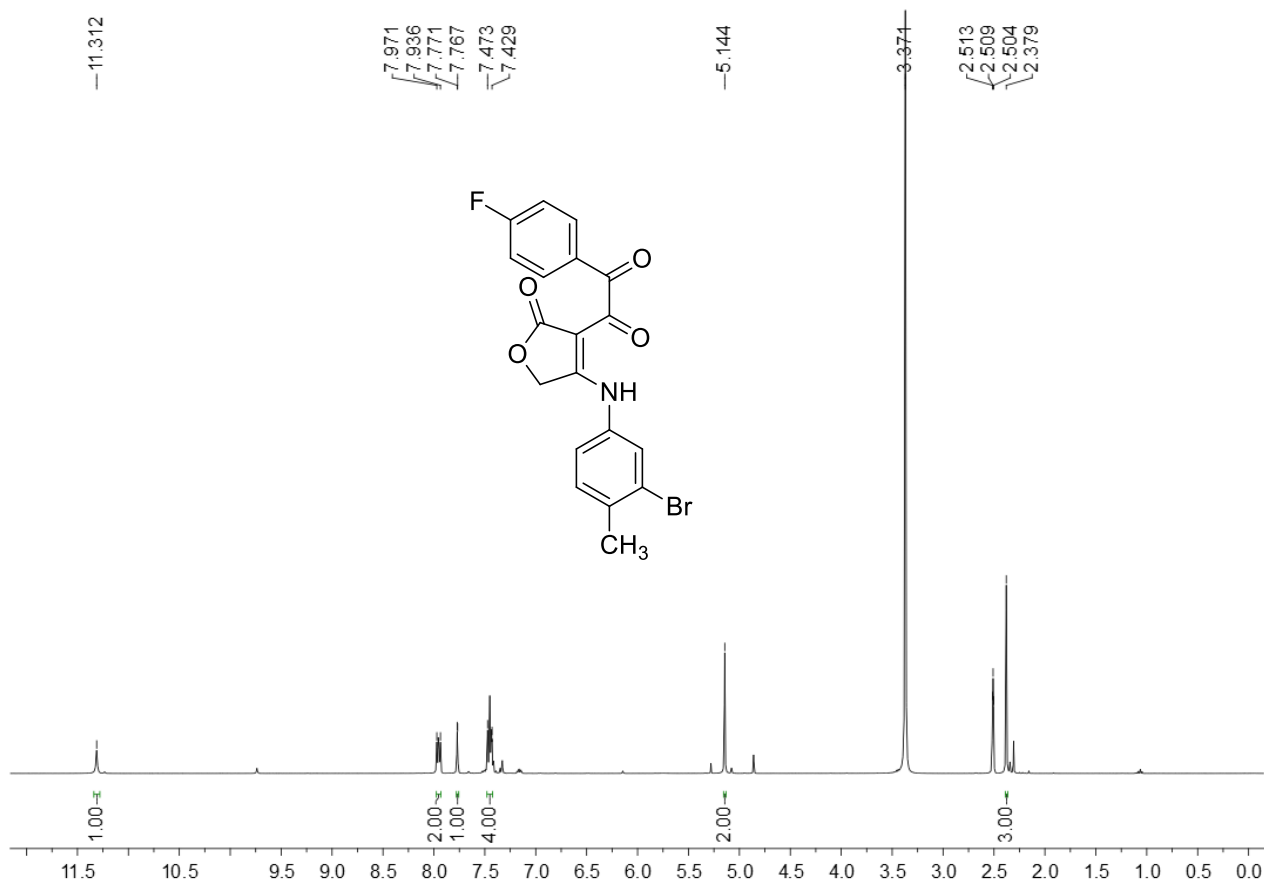
**<sup>13</sup>C NMR Spectrum of Compound 3n**

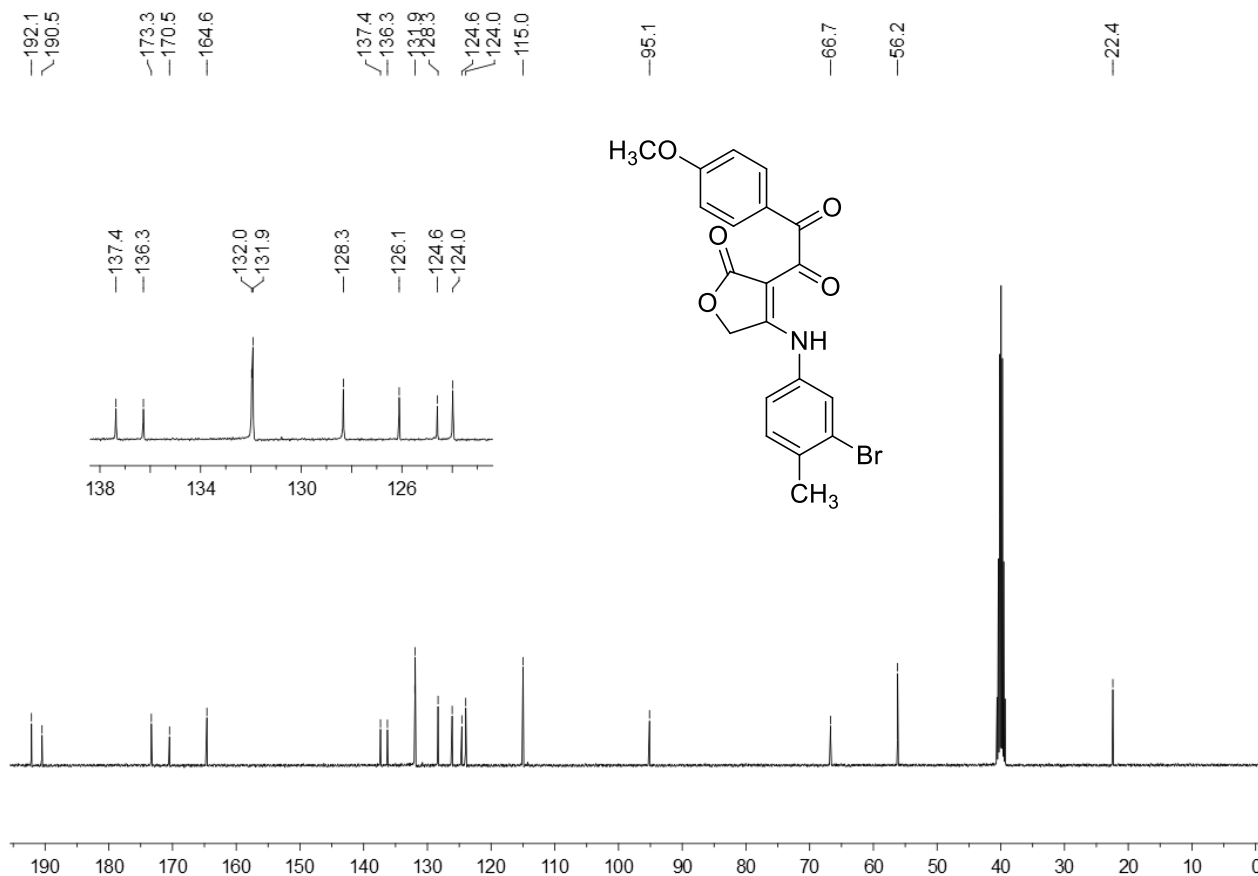
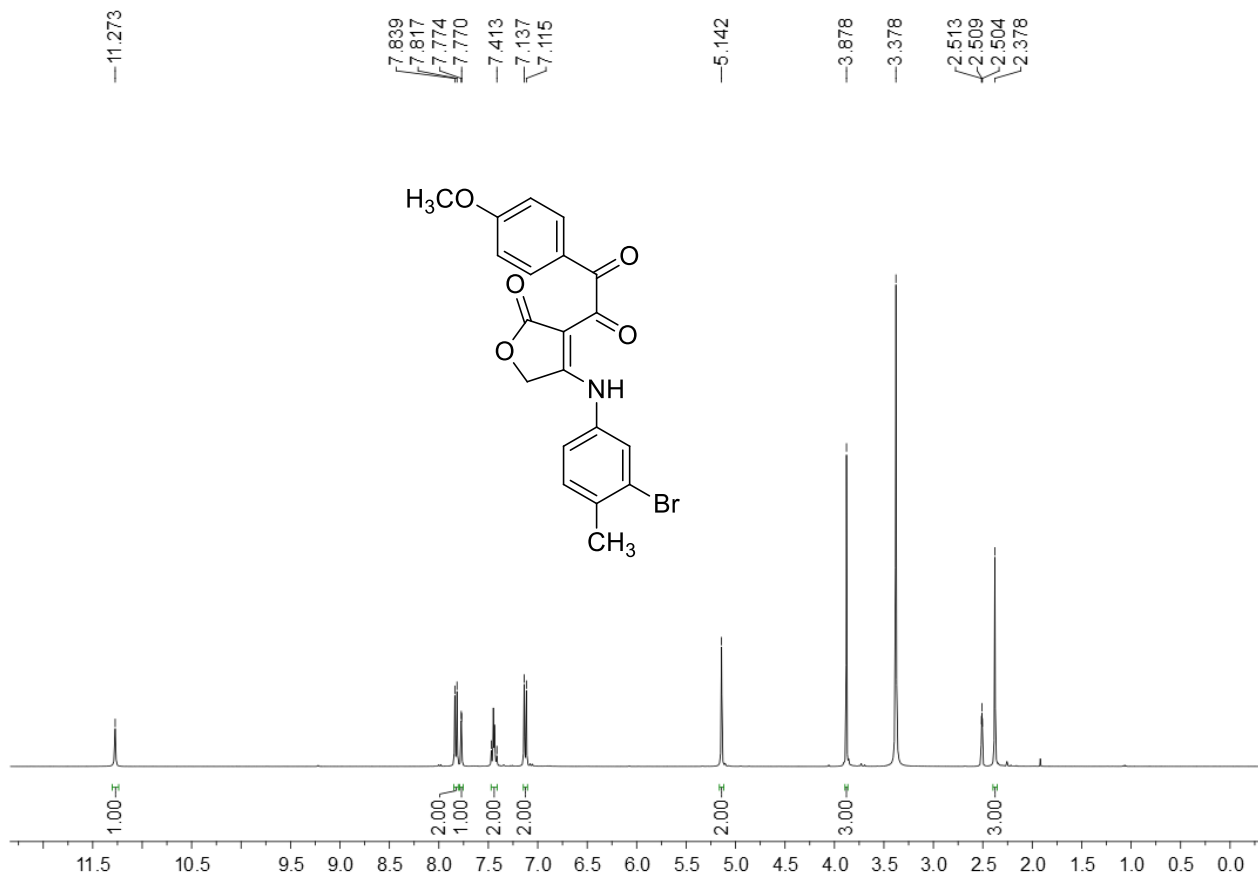


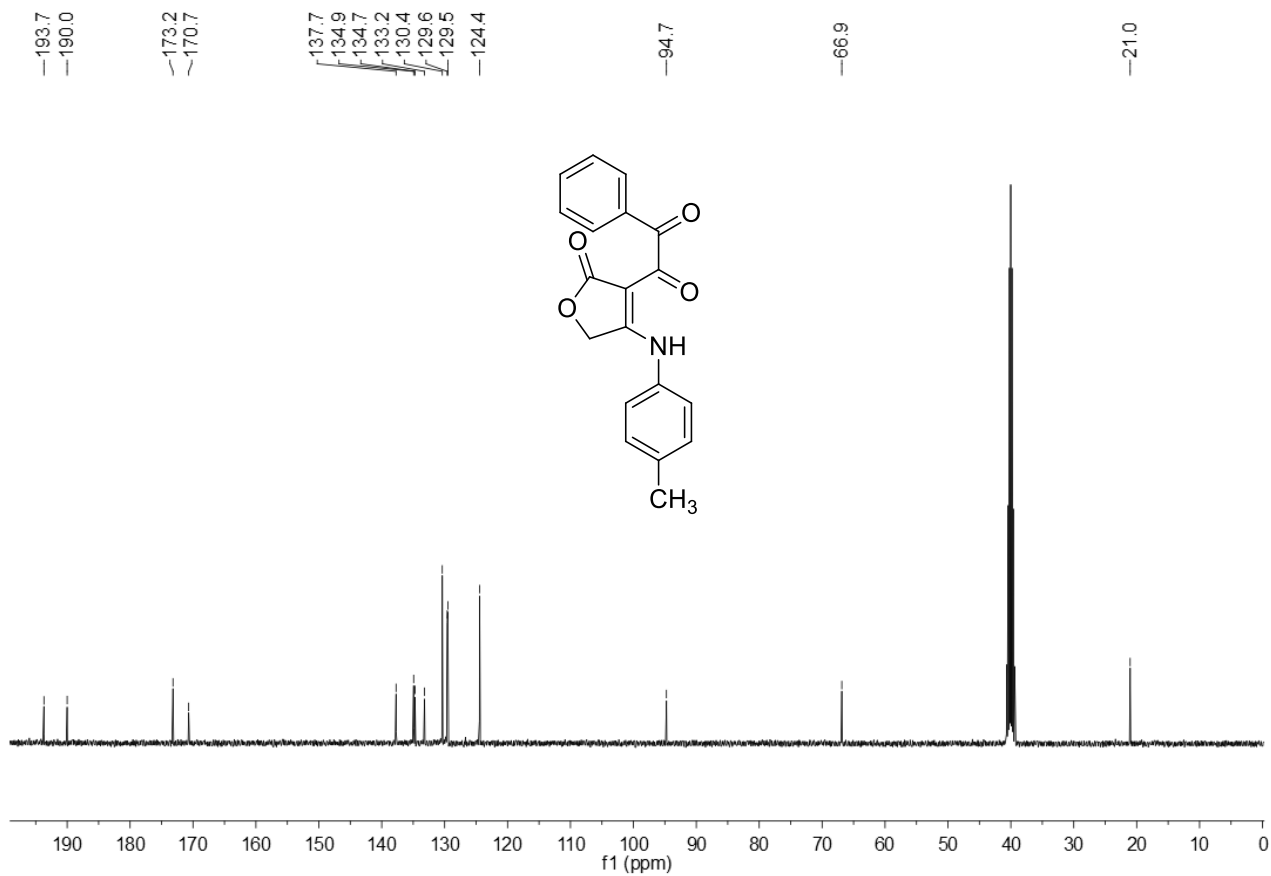
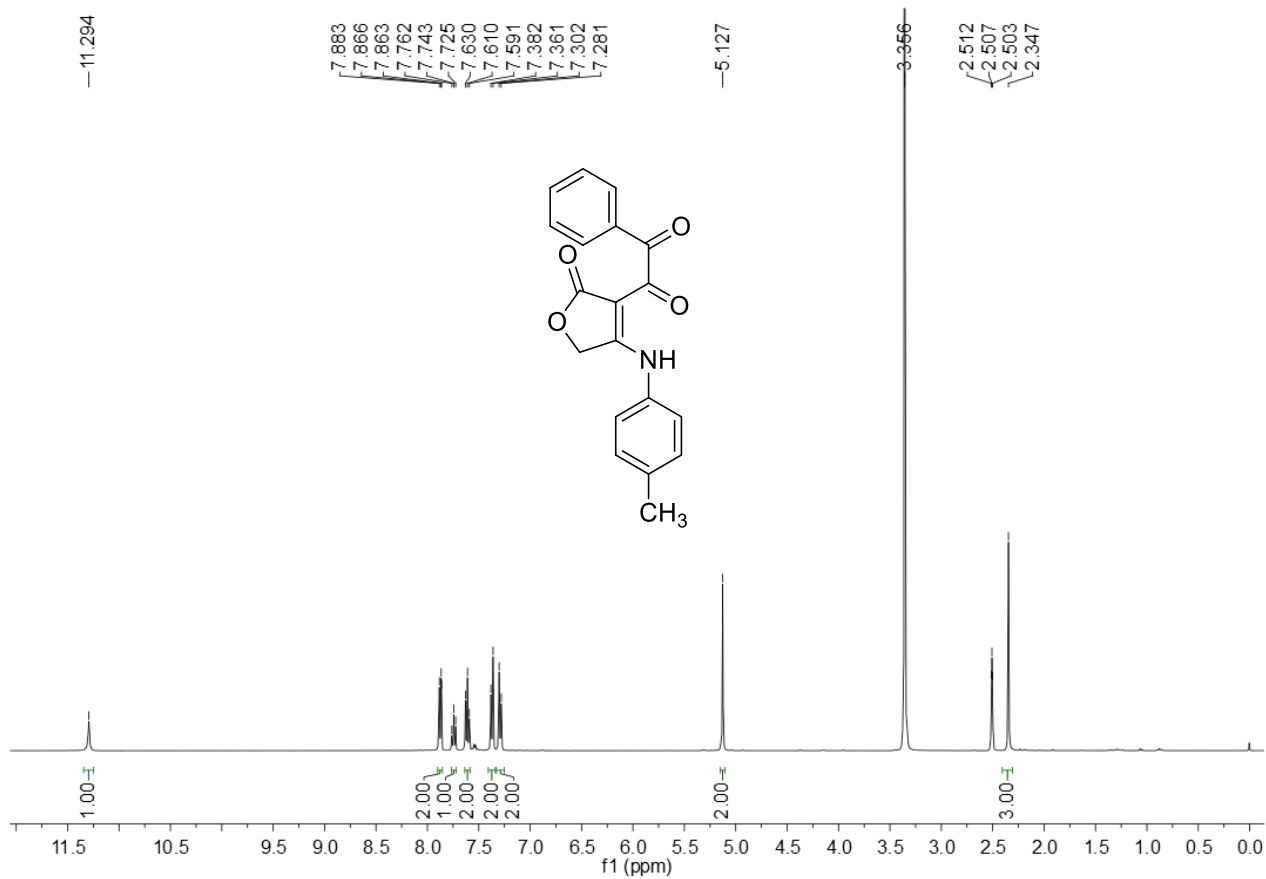


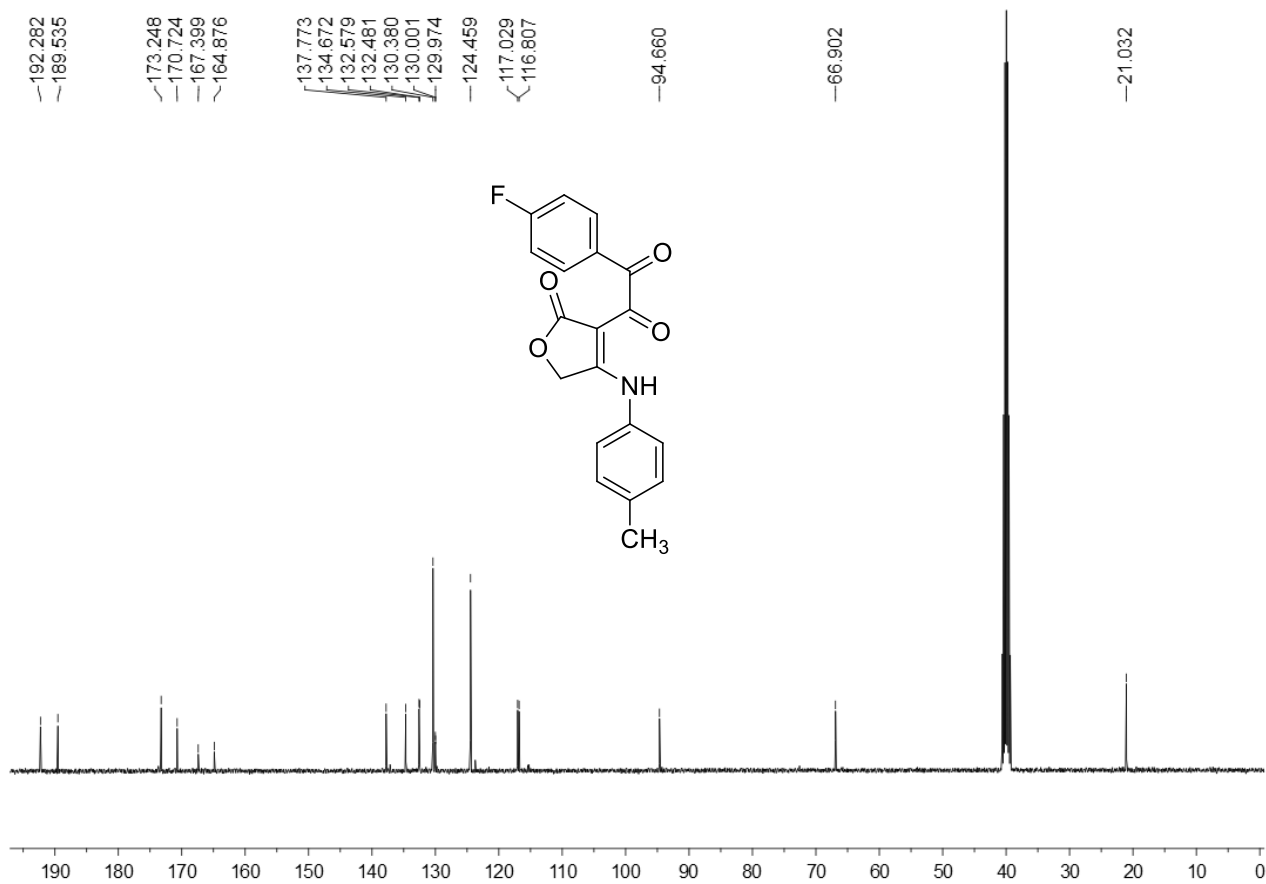
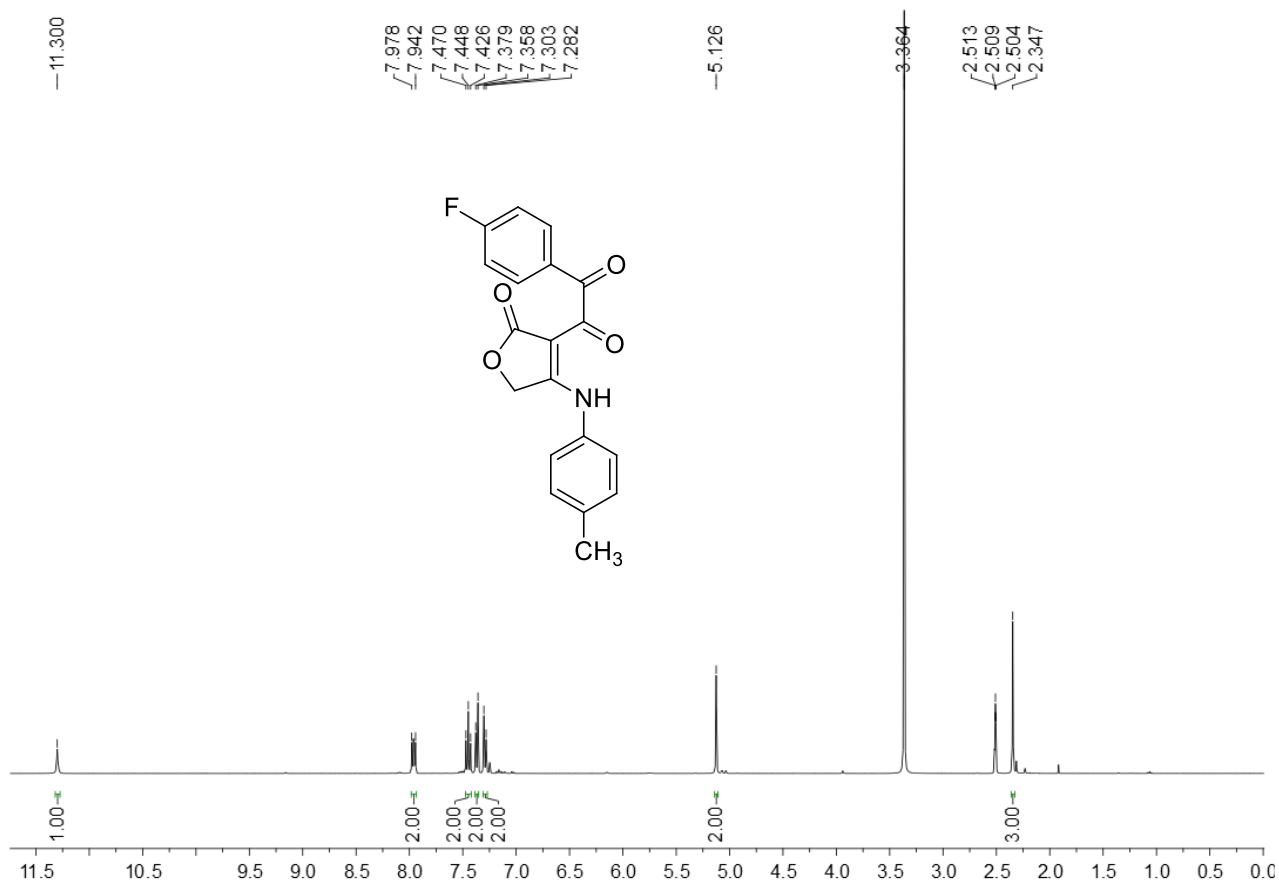


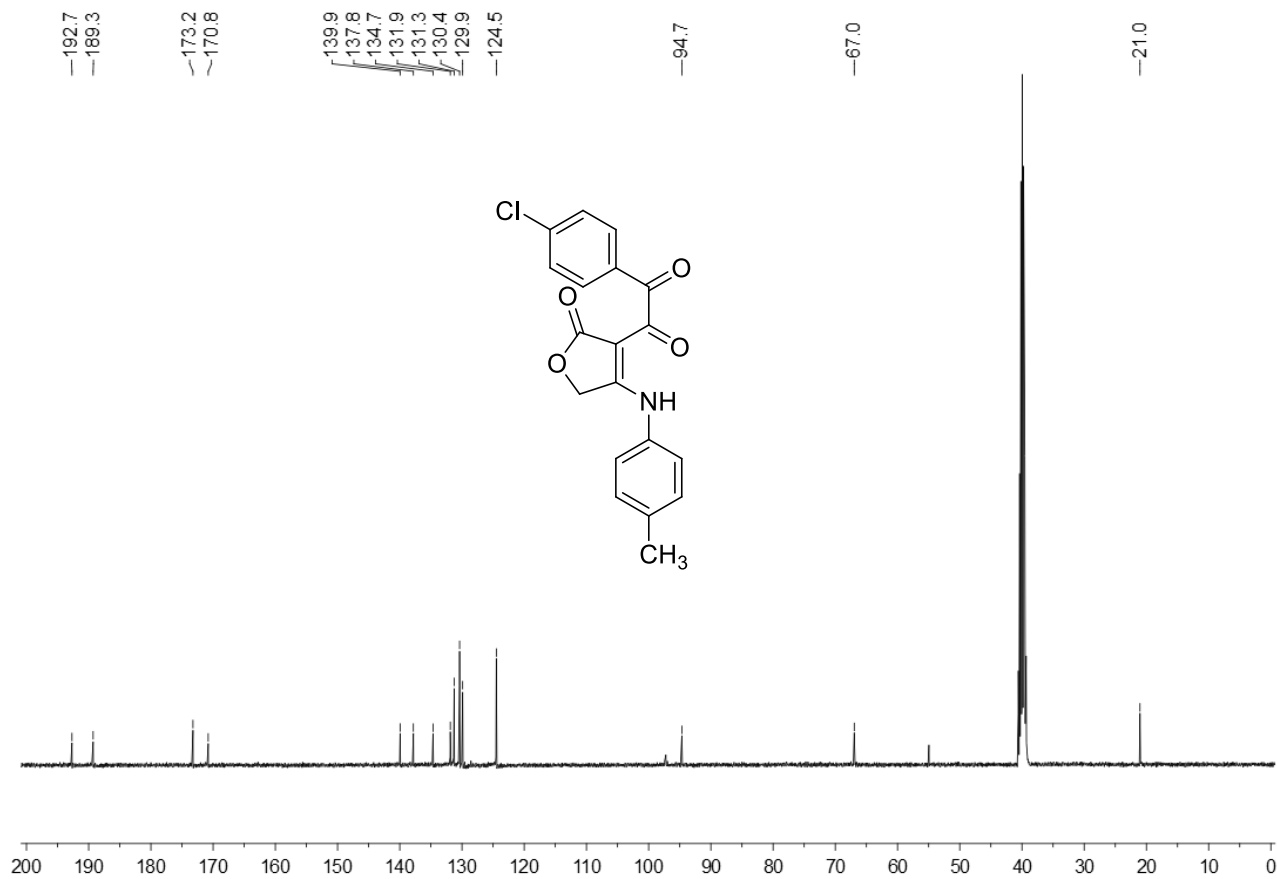
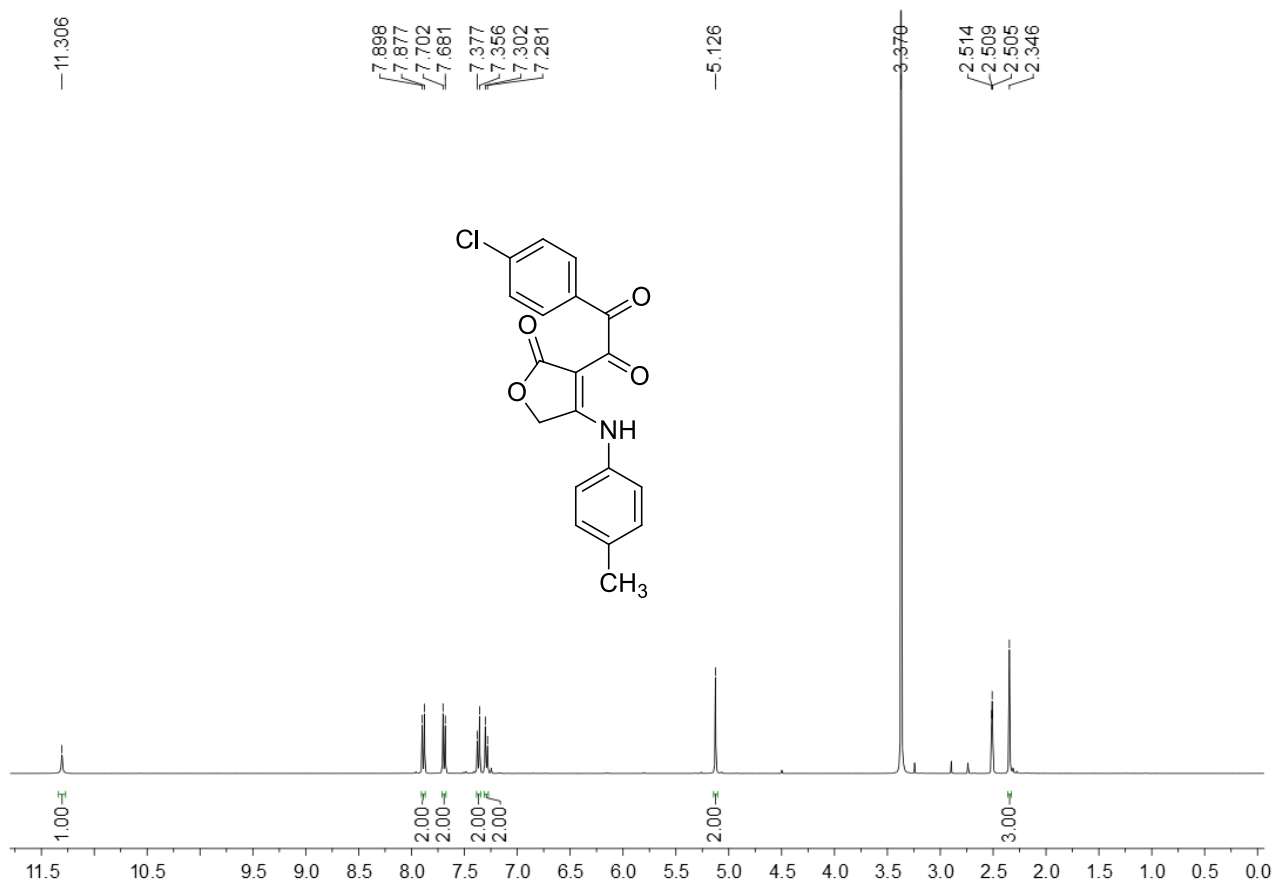
**<sup>13</sup>C NMR Spectrum of Compound 3q**

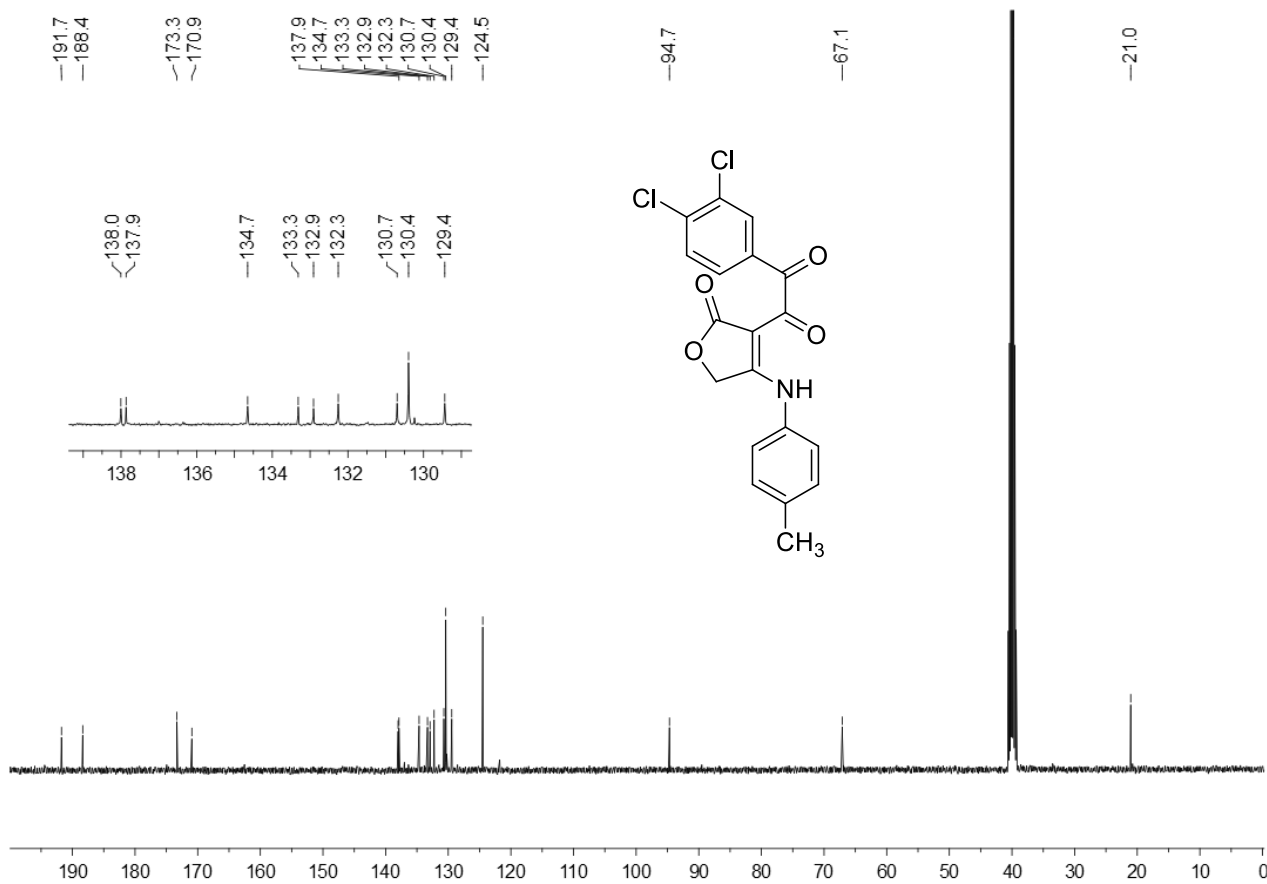
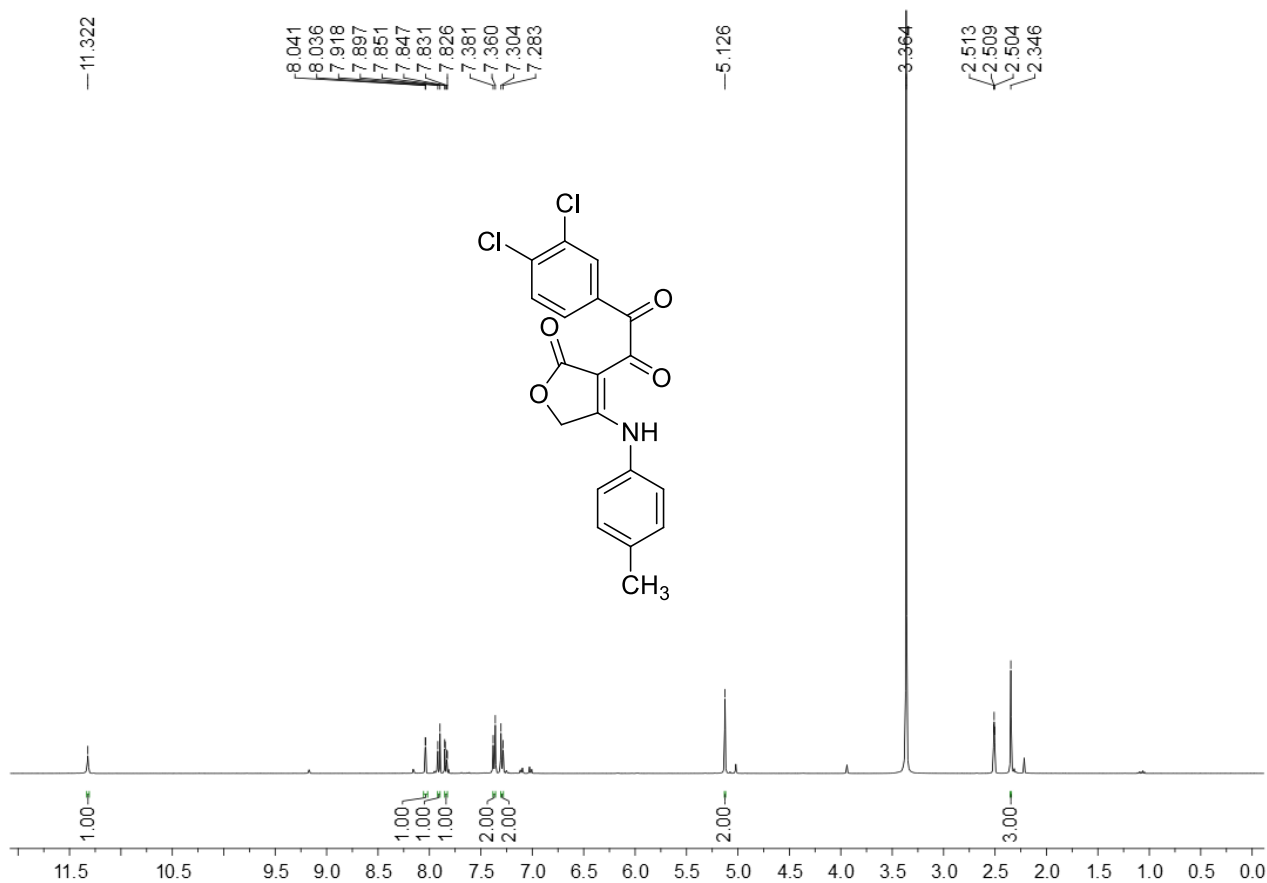


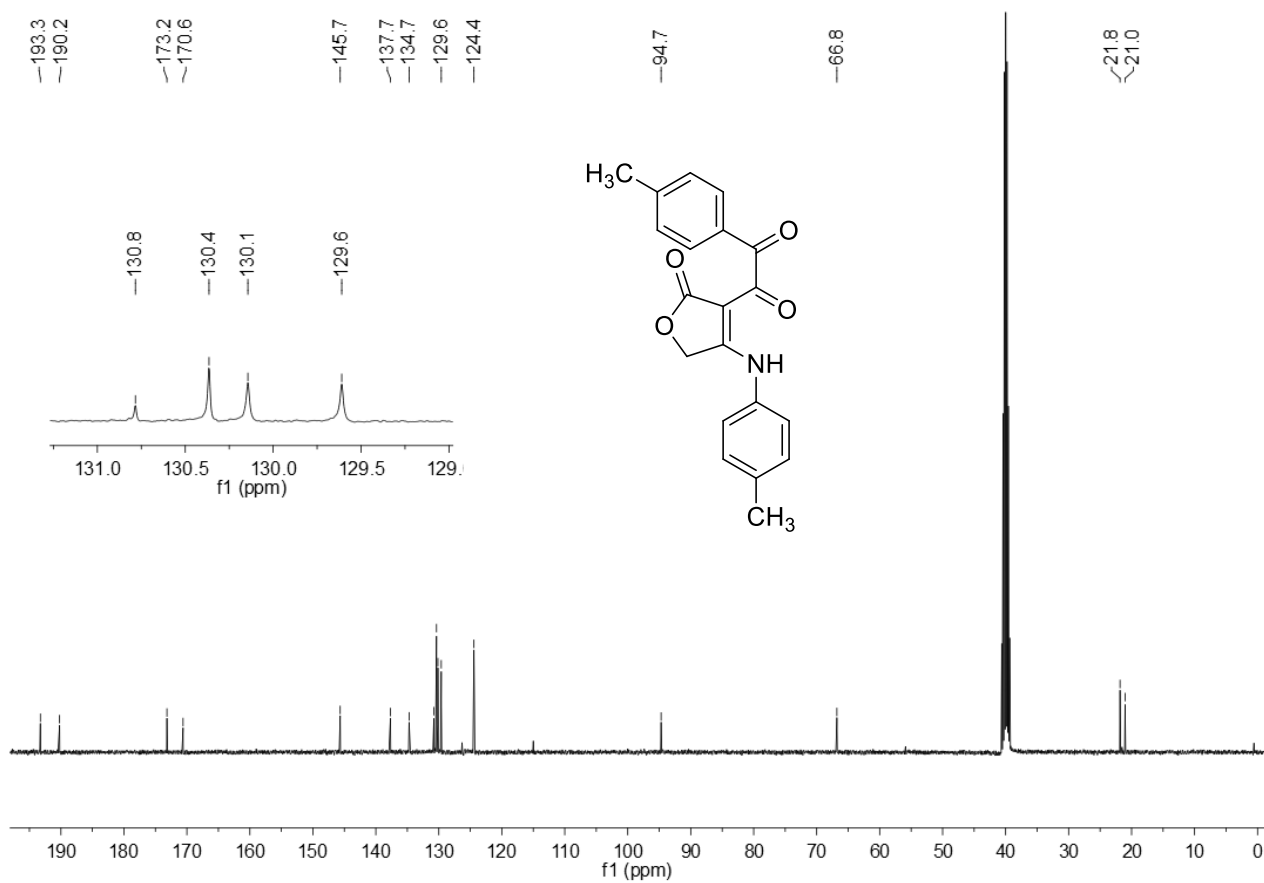
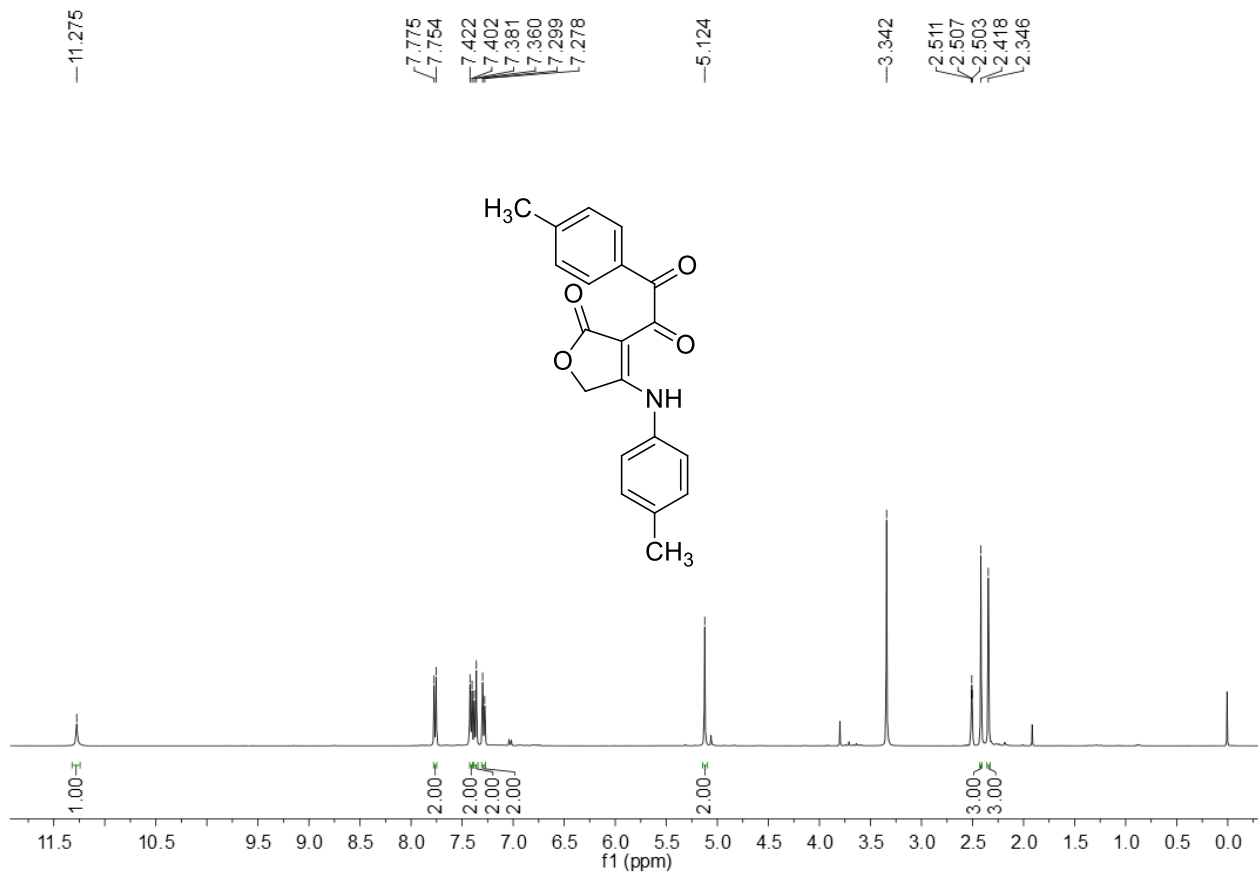


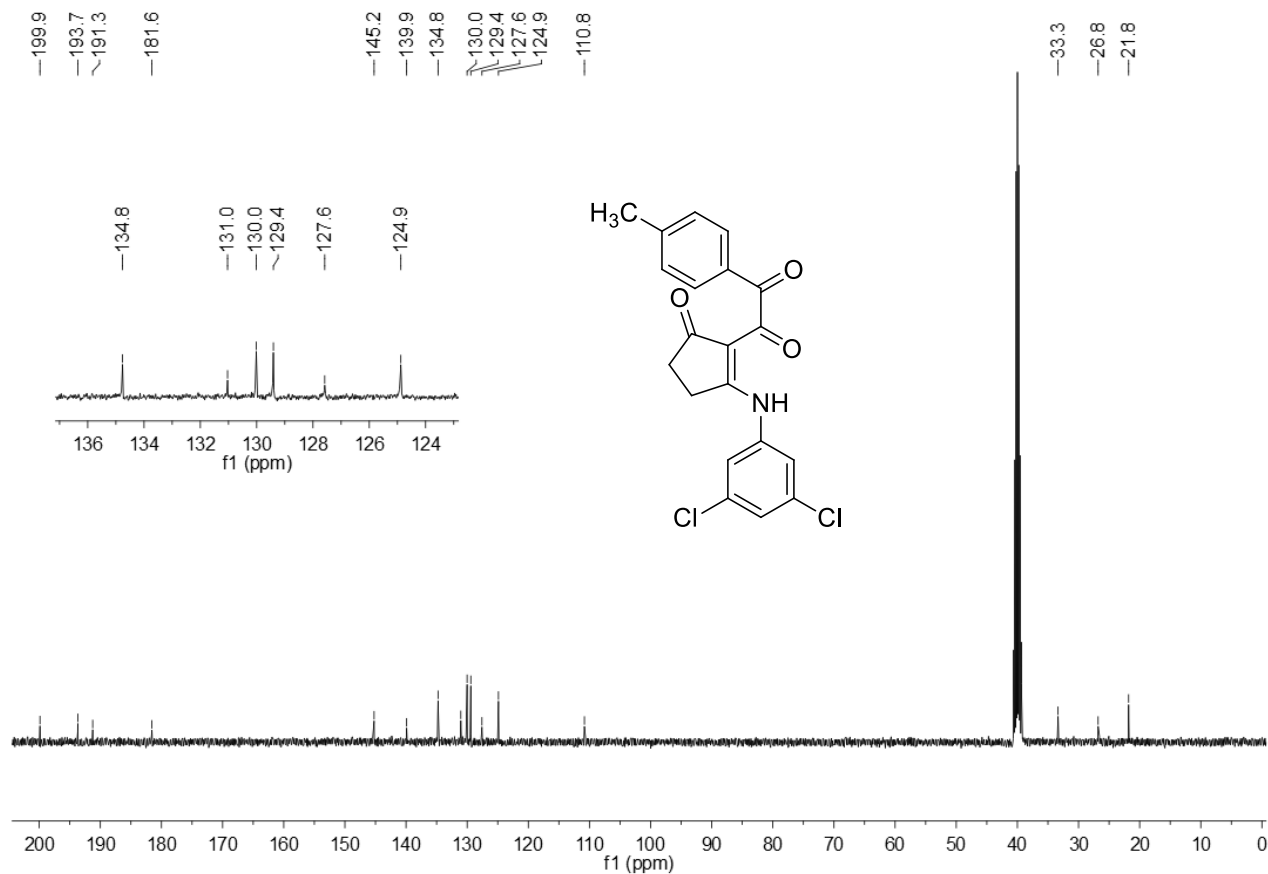
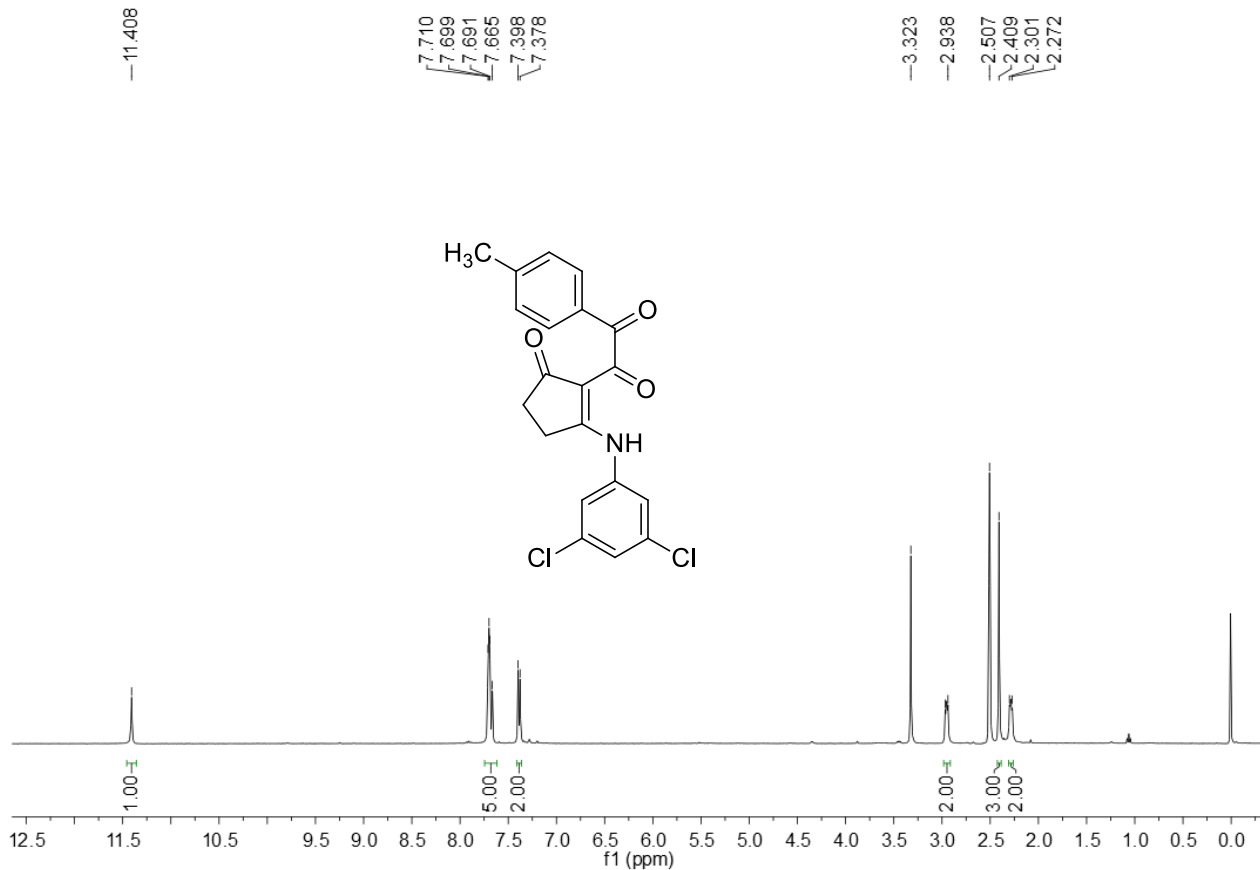


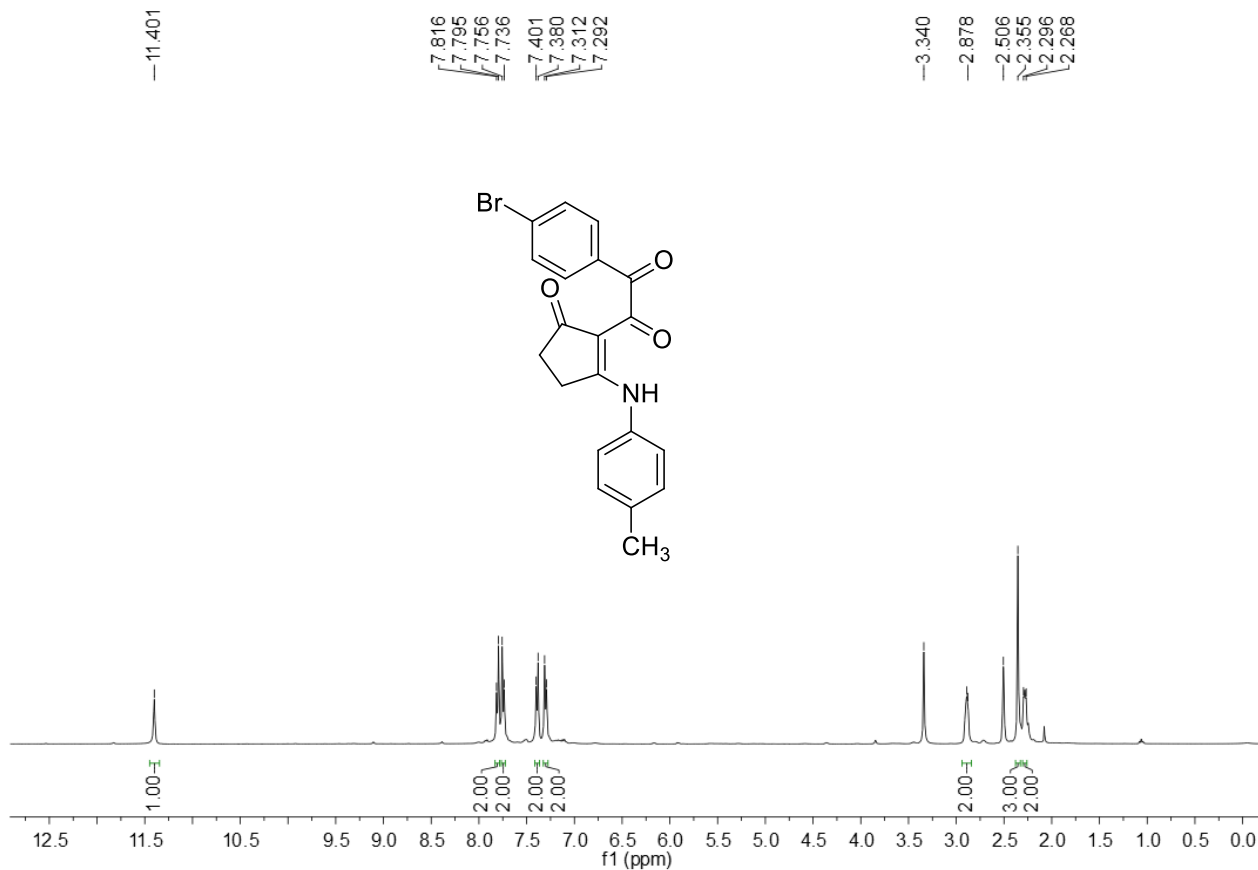




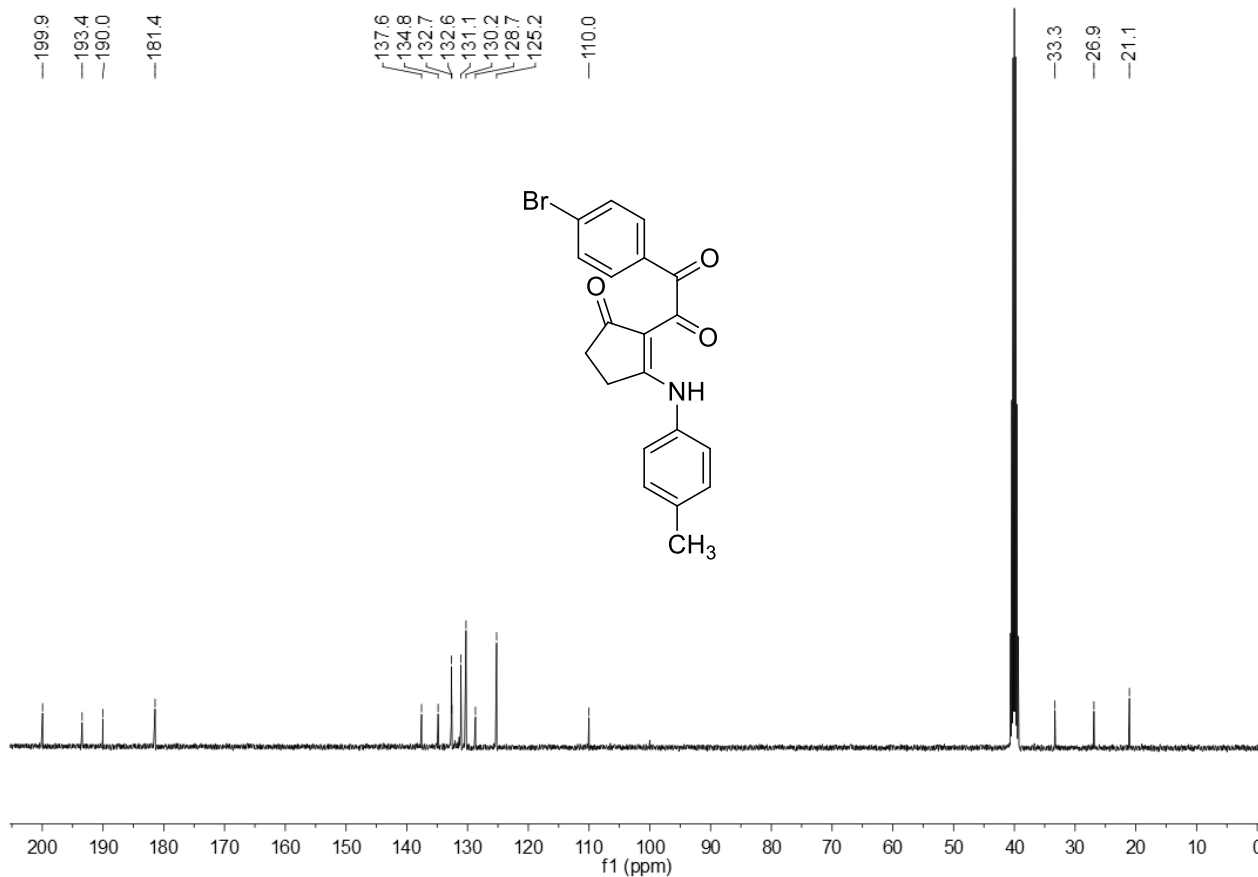




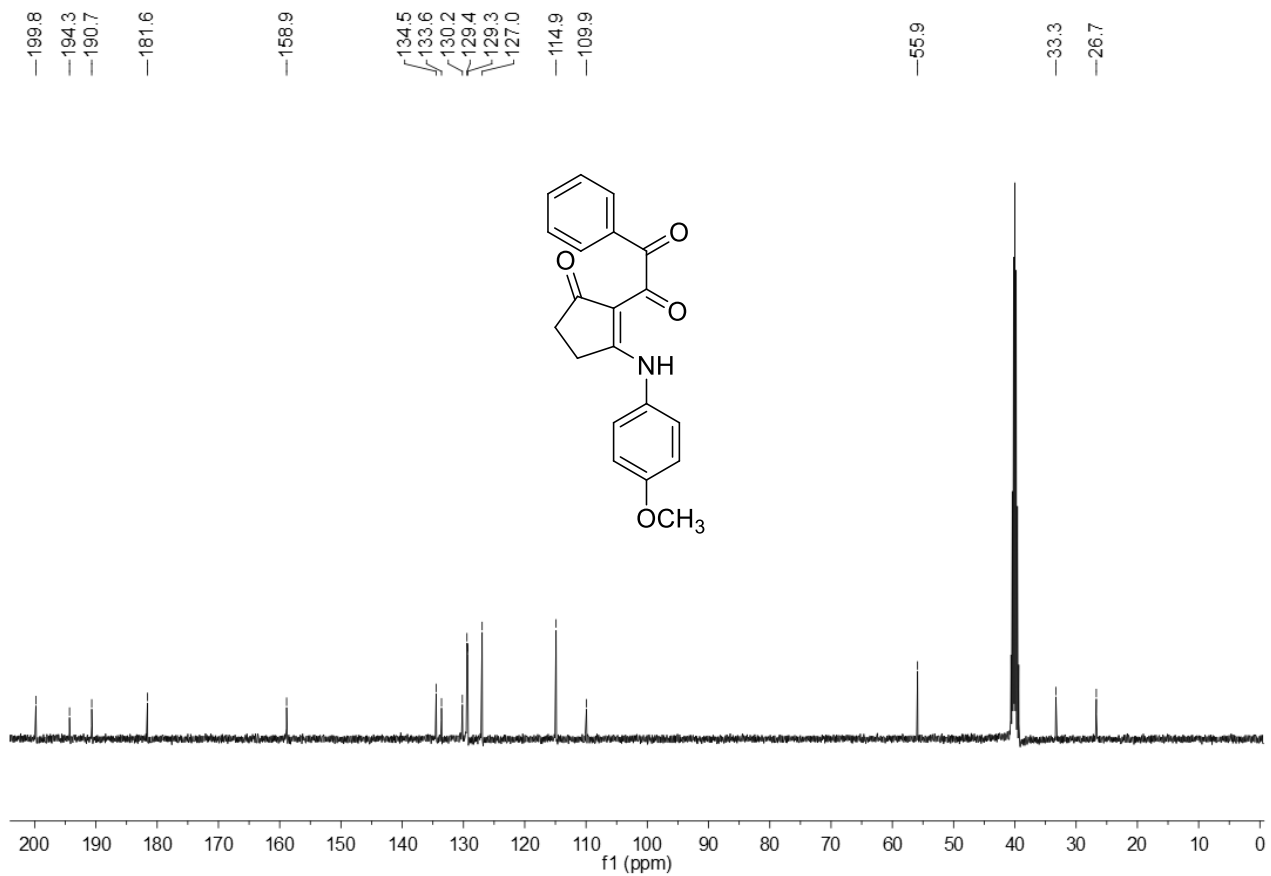
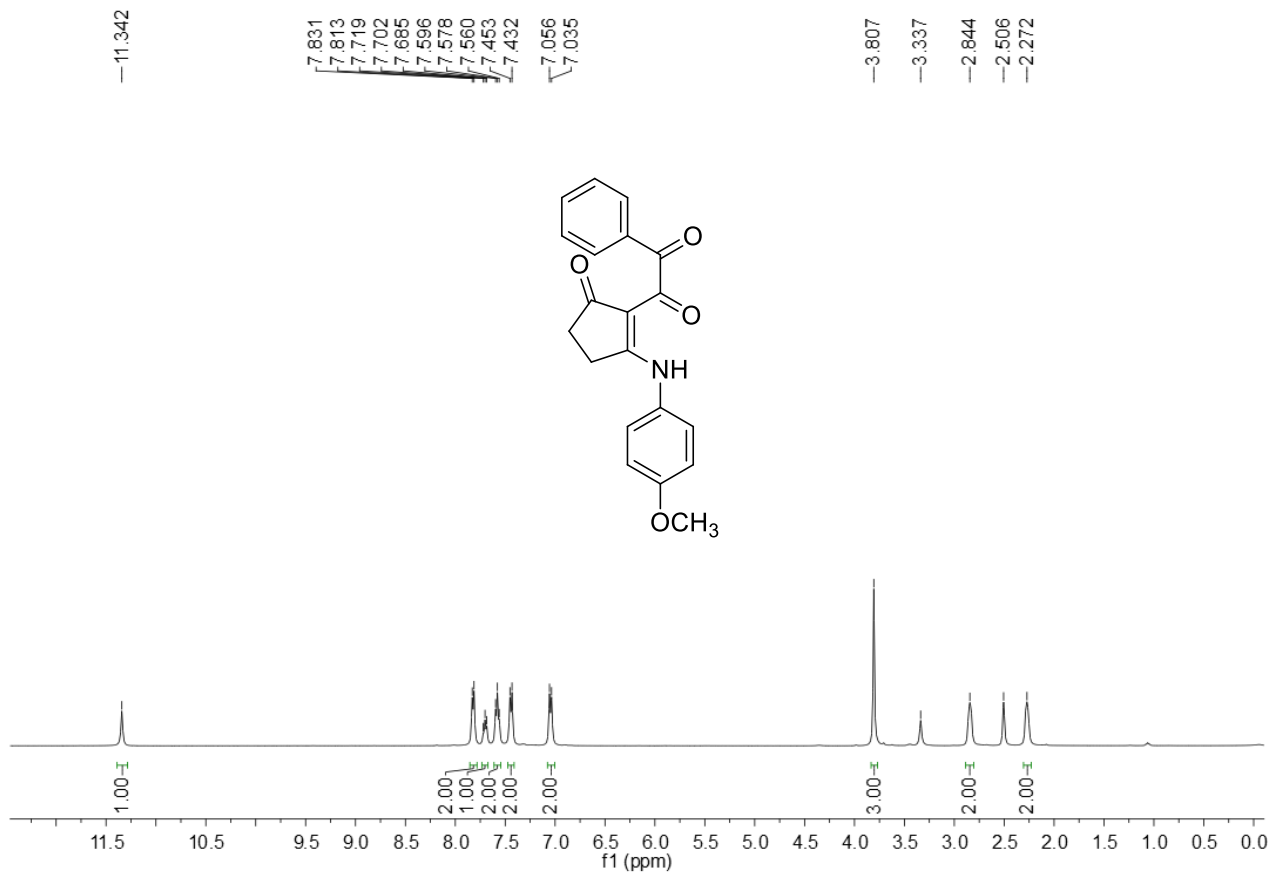




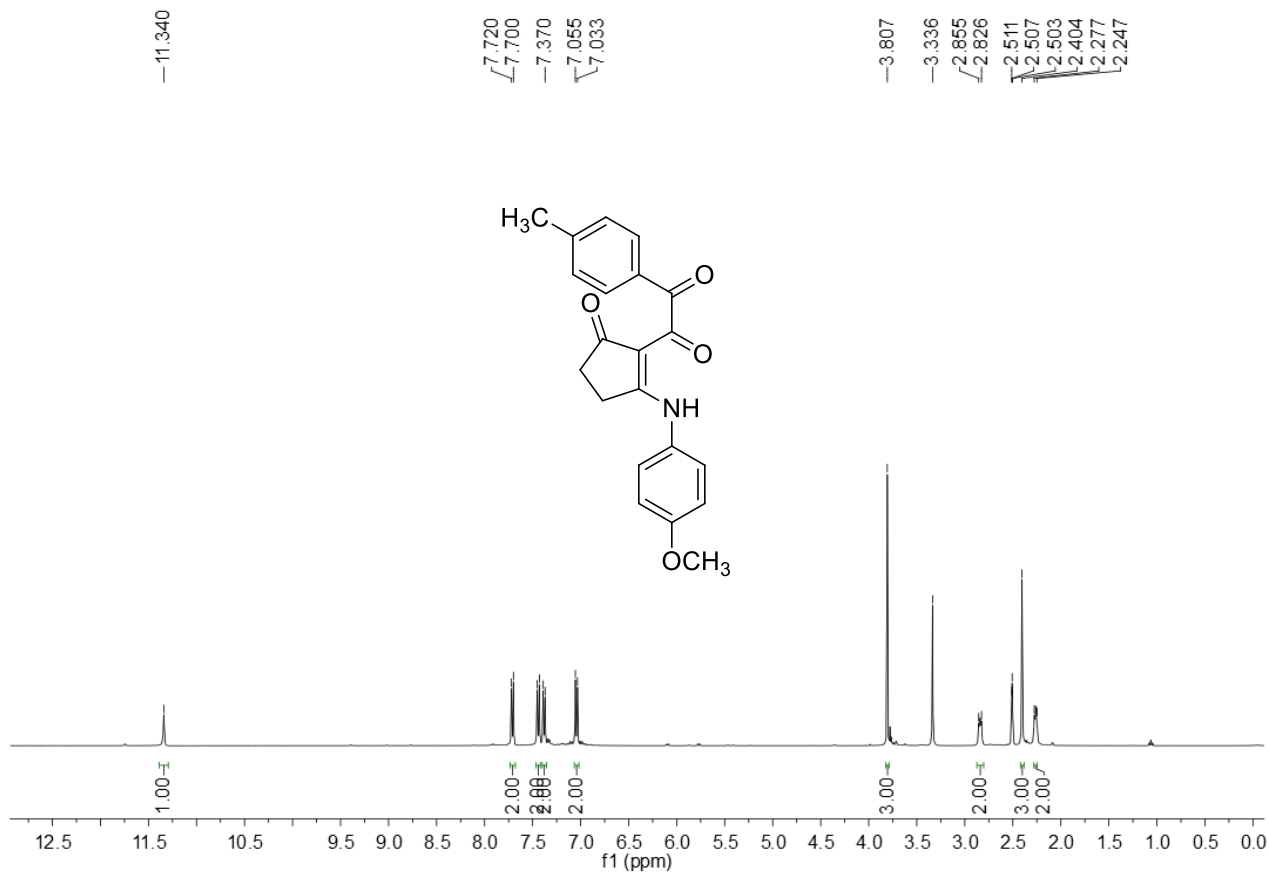
**<sup>1</sup>H NMR Spectrum of Compound 3z**



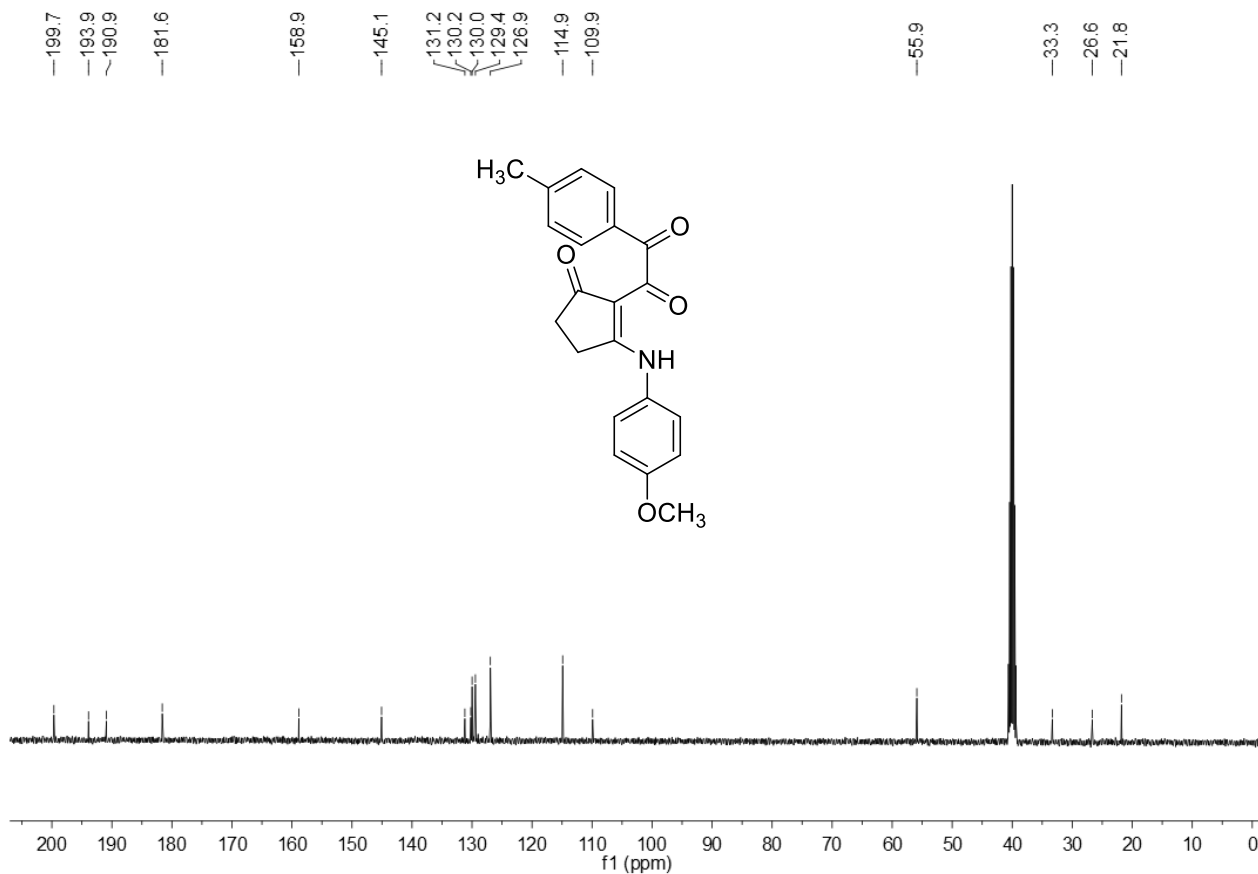
**<sup>13</sup>C NMR Spectrum of Compound 3z**



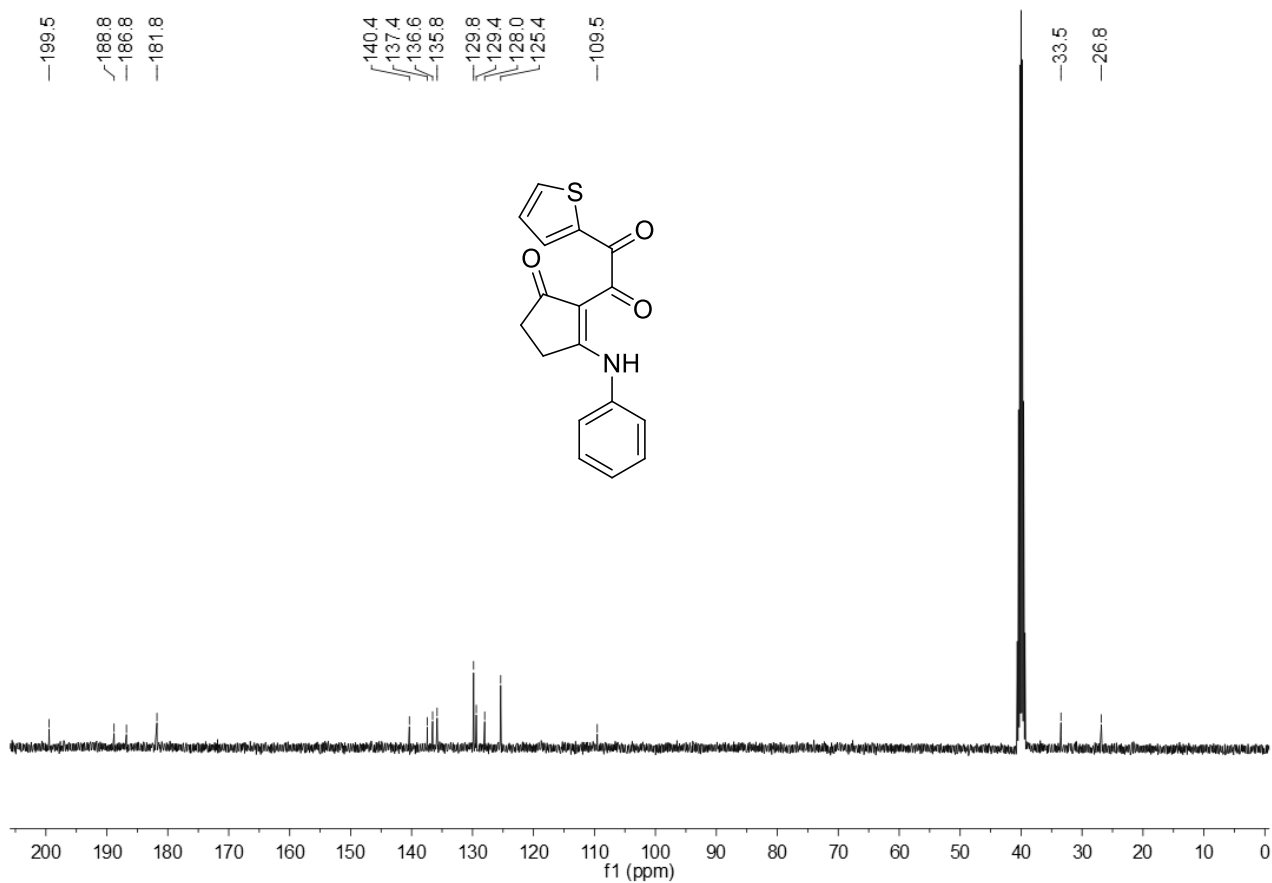
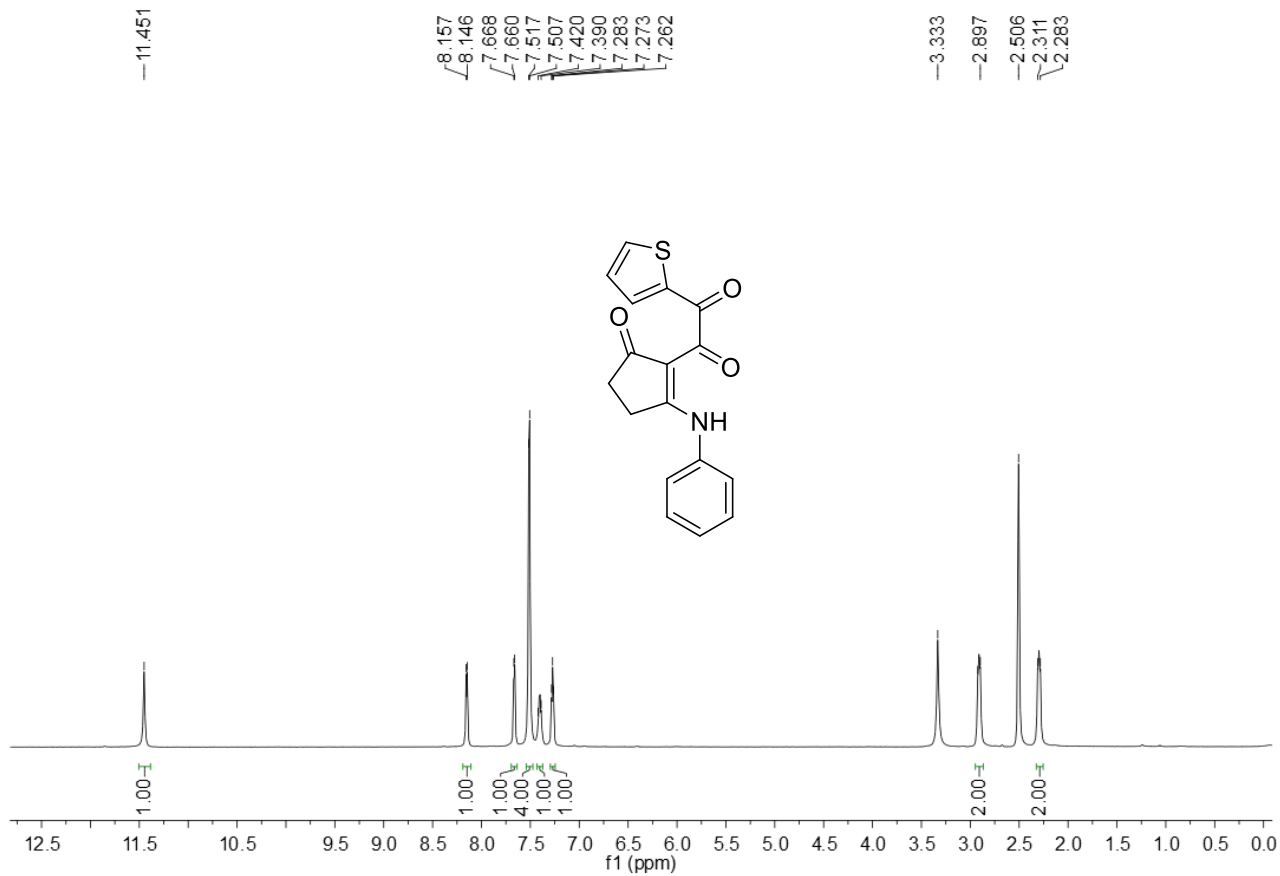
**<sup>13</sup>C NMR Spectrum of Compound 3aa**



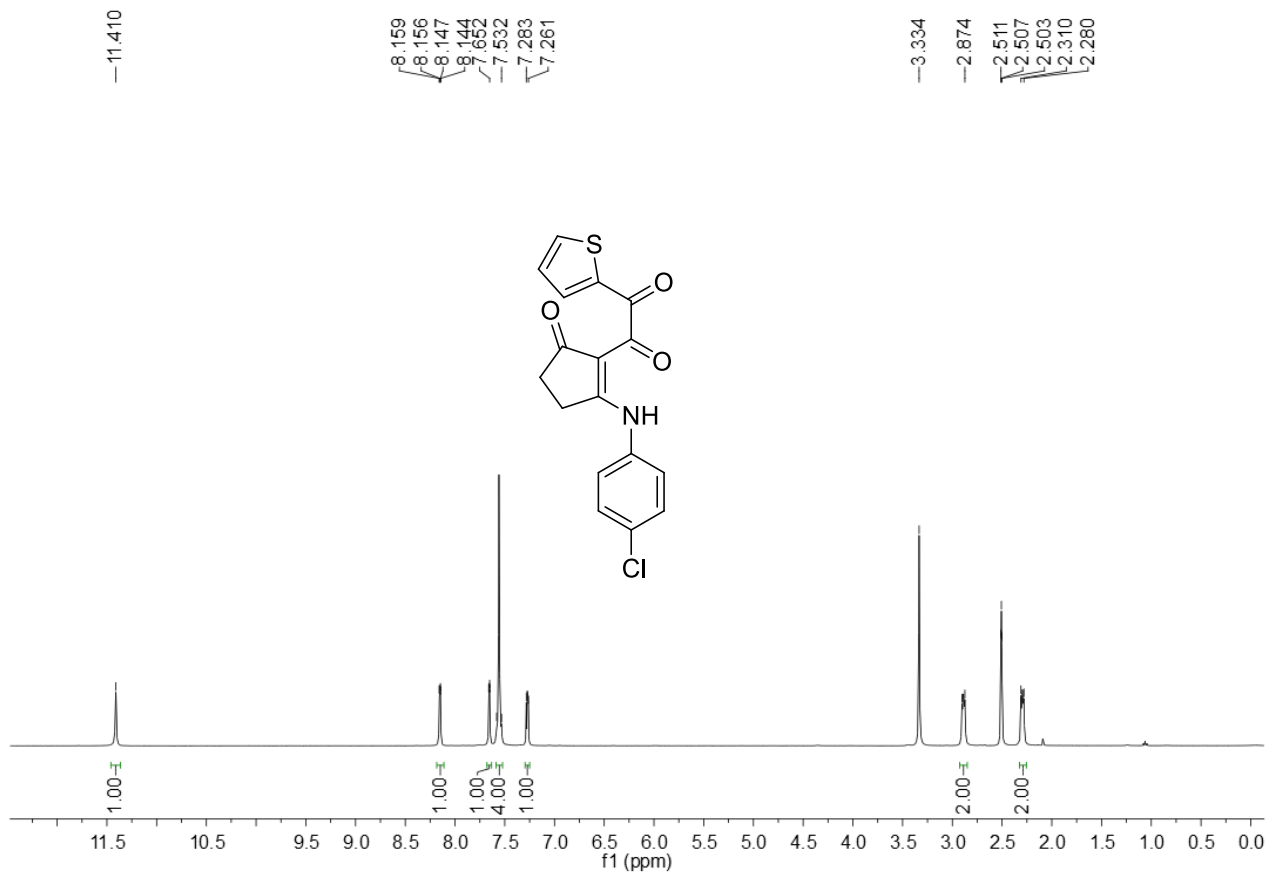
<sup>1</sup>H NMR Spectrum of Compound 3bb



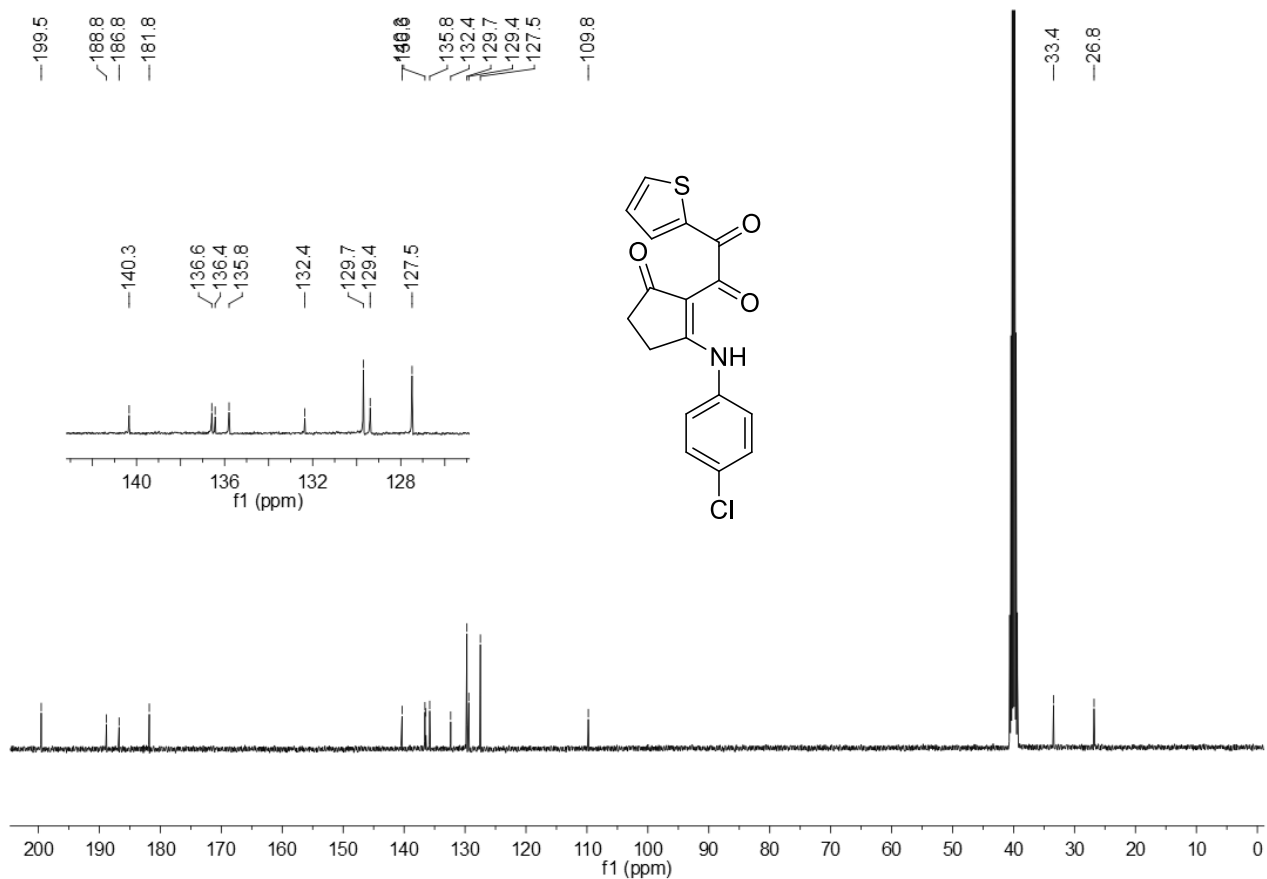
<sup>13</sup>C NMR Spectrum of Compound 3bb



**<sup>13</sup>C NMR Spectrum of Compound 3cc**



**<sup>1</sup>H NMR Spectrum of Compound 3dd**



**<sup>13</sup>C NMR Spectrum of Compound 3dd**

