Alkyltin Fluorides as Alkylating Reagent in Aminoalkylation of Maleimides

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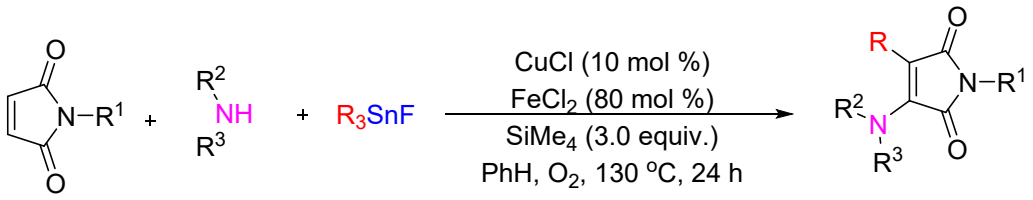
General Information

Maleimides[1] and trimethyltin fluoride[2] were prepared according to the reported procedures. ^{1}H and ^{13}C spectra of known compounds were in accordance with those described in the literatures. All other reagents were purchased from TCI, Sigma-Aldrich, Alfa Aesar, Acros, and Meryer and used without further purification. ^{1}H NMR (500 MHz), ^{13}C NMR (125 MHz) and ^{19}F NMR (470 MHz) spectra were recorded in $CDCl_3$ and DMSO-D6 solutions using a Burker AVANCE 500 spectrometer. High-resolution mass spectra were recorded on an ESI-Q-TOF mass spectrometer. Analysis of crude reaction mixture was done on the Varian 4000 GC/MS and 1200 LC. All reactions were conducted using standard Schlenk techniques. Column chromatography was performed using EM silica gel 60 (300−400 m).

General Experimental Procedures

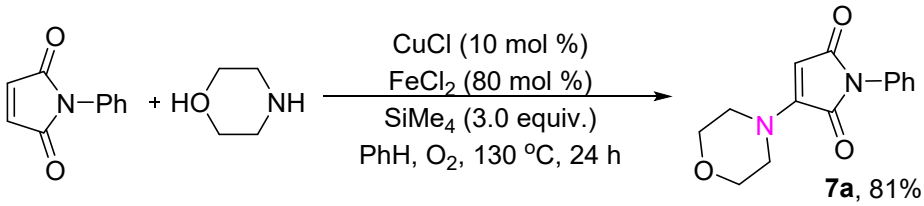
General Procedure of Aminoalkylation of Maleimides with Alkylamines and R_3SnF:



3-6

A 25 mL Schlenk tube equipped with a stir bar was charged with maleimide (0.2 mmol), secondary amines (0.6 mmol), organotin fluoride compounds (0.6 mmol), CuBr (10 mol %), $FeCl_2$ (80 mol %), $SiMe_4$ (0.6 mmol) and 2.0 mL PhH. The tube was fitted with a rubber septum, and then it was evacuated and refilled with dioxygen three times, then the septum was replaced by a Teflon screwcap under oxygen flow. The reaction mixture was stirred at 130 °C for 24 h. After cooling down, the reaction mixture was diluted with 10 mL of ethyl ether, filtered through a pad of silica gel, followed by washing the pad of the silica gel with the same solvent (20 mL), concentrated under reduced pressure. The residue was then purified by flash chromatography on silica gel to provide the corresponding product.

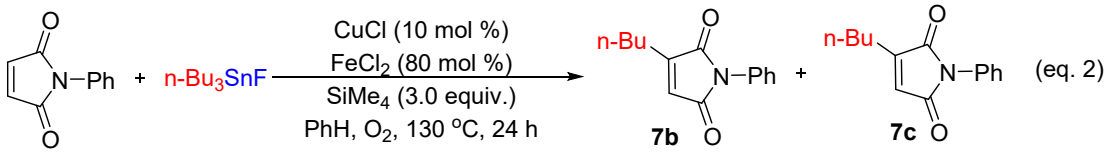
Mechanistic Studies



7a, 81%

(eq. 1)

A 25 mL Schlenk tube equipped with a stir bar was charged with *N*-phenyl maleimide (0.2 mmol), morpholine (0.6 mmol), CuBr (10 mol %), $FeCl_2$ (80 mol %), $SiMe_4$ (0.6 mmol) and 2.0 mL PhH. The tube was fitted with a rubber septum, and then it was evacuated and refilled with dioxygen three times, then the septum was replaced by a Teflon screwcap under oxygen flow. The reaction mixture was stirred at 130 °C. After stirring for 24 h, the reaction mixture was cooled to room temperature and the reaction was filtered through a pad of Celite and diluted with ethyl acetate (10 mL), aminated maleimide **7a** was isolated in 81% yield.

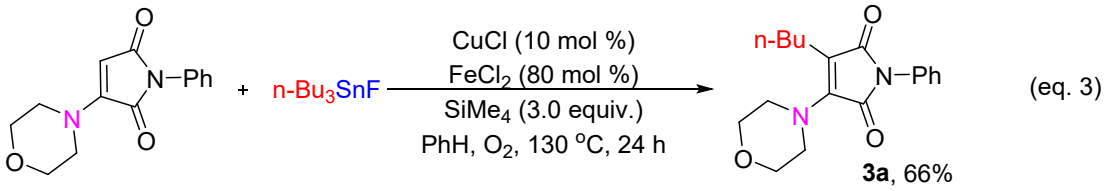


7b **7c**

(eq. 2)

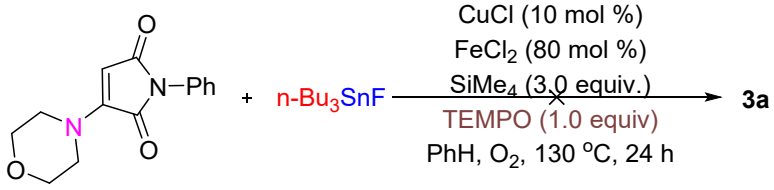
7b, **7c** were not detected by HRMS

A 25 mL Schlenk tube equipped with a stir bar was charged with *N*-phenyl maleimide (0.2 mmol), fluorotributyltin (0.6 mmol), CuBr (10 mol %), $FeCl_2$ (80 mol %), $SiMe_4$ (0.6 mmol) and 2.0 mL PhH. The tube was fitted with a rubber septum, and then it was evacuated and refilled with dioxygen three times, then the septum was replaced by a Teflon screwcap under oxygen flow. The reaction mixture was stirred at 130 ℃. After stirring for 24 h, the reaction mixture was cooled to room temperature and the reaction was filtered through a pad of Celite and diluted with ethyl acetate (10 mL), oxidative alkylated **7b** or hydroalkylated maleimide **7c** were not detected by HRMS.



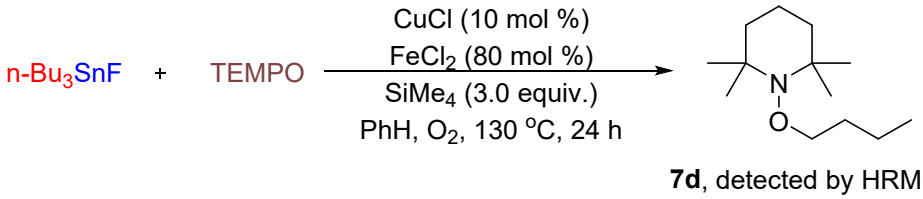
(eq. 3)

A 25 mL Schlenk tube equipped with a stir bar was charged with 3-morpholino-1-phenyl-1H-pyrrole-2,5-dione (0.2 mmol), fluorotributyltin (0.6 mmol), CuBr (10 mol %), $FeCl_2$ (80 mol %), $SiMe_4$ (0.6 mmol) and 2.0 mL PhH. The tube was fitted with a rubber septum, and then it was evacuated and refilled with dioxygen three times, then the septum was replaced by a Teflon screwcap under oxygen flow. The reaction mixture was stirred at 130 ℃. After stirring for 24 h, the reaction mixture was cooled to room temperature and the reaction was filtered through a pad of Celite and diluted with ethyl acetate (10 mL), the butylated product **3a** was isolated in 66%.



(eq. 4)

A 25 mL Schlenk tube equipped with a stir bar was charged with 3-morpholino-1-phenyl-1H-pyrrole-2,5-dione (0.2 mmol), TEMPO (0.2 mmol), fluorotributyltin (0.6 mmol), CuBr (10 mol %), $FeCl_2$ (80 mol %), $SiMe_4$ (0.6 mmol) and 2.0 mL PhH. The tube was fitted with a rubber septum, and then it was evacuated and refilled with dioxygen three times, then the septum was replaced by a Teflon screwcap under oxygen flow. The reaction mixture was stirred at 130 ℃. After stirring for 24 h, the reaction mixture was cooled to room temperature and the reaction was filtered through a pad of Celite and diluted with ethyl acetate (10 mL), no reaction was observed.

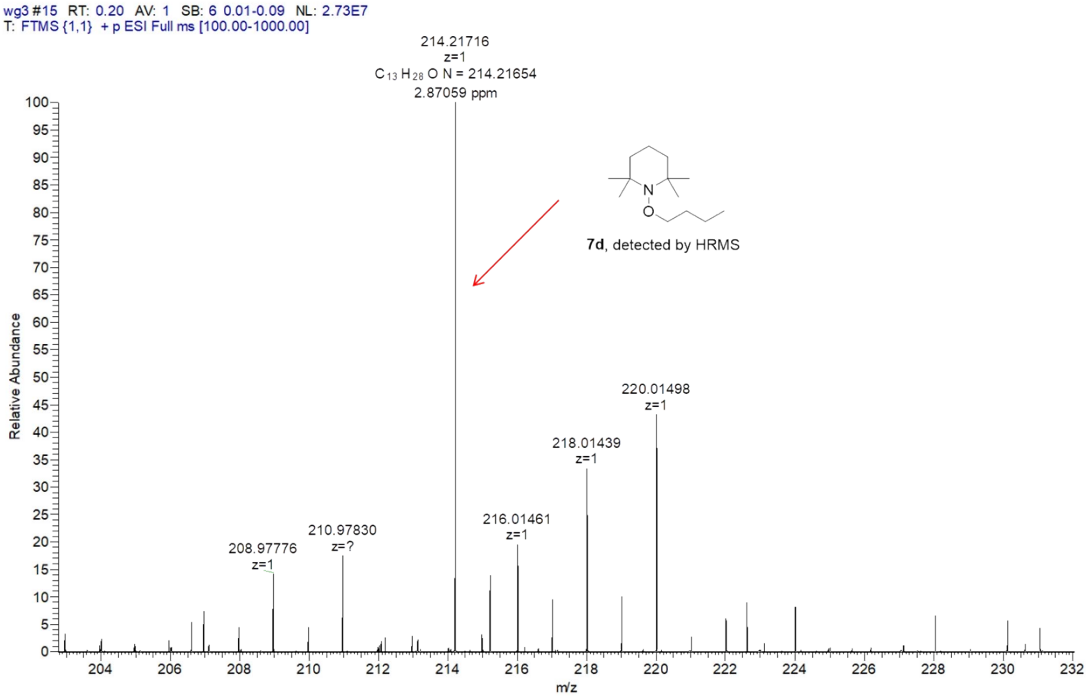


7d, detected by HRMS

(eq. 5)

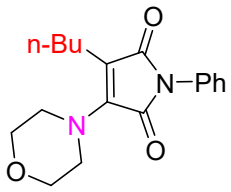
A 25 mL Schlenk tube equipped with a stir bar was charged with TEMPO (0.2 mmol), fluorotributyltin (0.6 mmol), CuBr (10 mol %), $FeCl_2$ (80 mol %), $SiMe_4$ (0.6 mmol) and 2.0 mL PhH. The tube was fitted with a rubber septum, and then it was evacuated and refilled with dioxygen three times, then the septum was replaced by a Teflon screwcap under oxygen flow. The

reaction mixture was stirred at 130 ℃. After stirring for 24 h, the reaction mixture was cooled to room temperature and the reaction was filtered through a pad of Celite and diluted with ethyl acetate (10 mL), the radical trapped product **7d** was detected by HRMS.



Characterization of Products in Details:

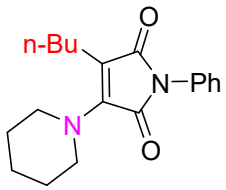
3-butyl-4-morpholino-1-phenyl-1H-pyrrole-2,5-dione



3a

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (46.5 mg, 74% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.46 (t, *J* = 7.8 Hz, 2H), 7.38-7.32 (m, 3H), 3.87-3.84 (m, 4H), 3.78-3.75 (m, 4H), 2.52-2.48 (m, 2H), 1.54 (tt, *J* = 7.9, 5.9 Hz, 2H), 1.43 (dt, *J* = 14.7, 7.4 Hz, 2H), 0.99 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.21, 167.37, 143.54, 132.02, 128.90, 127.28, 126.09, 108.97, 67.03, 48.93, 32.76, 23.27, 22.77, 13.94. **HRMS** (ESI): calcd for $C_{18}H_{23}N_2O_3$ $[M + H]^+$ 315.1709, found 315.1712.

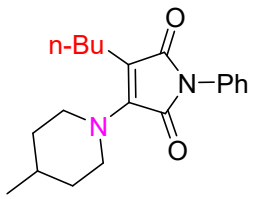
3-butyl-1-phenyl-4-(piperidin-1-yl)-1H-pyrrole-2,5-dione



3b

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (44.3 mg, 71% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.47-7.43 (m, 2H), 7.39-7.37 (m, 2H), 7.34-7.30 (m, 1H), 3.70 (d, *J* = 4.8 Hz, 2H), 2.51-2.47 (m, 2H), 1.73 (d, *J* = 3.4 Hz, 6H), 1.54 (tt, *J* = 7.7, 5.9 Hz, 2H), 1.46-1.41 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.56, 167.47, 144.40, 132.32, 128.79, 127.00, 126.09, 106.55, 50.10, 32.63, 26.49, 24.24, 23.45, 22.78, 13.95. **HRMS** (ESI): calcd for $C_{19}H_{25}N_2O_2$ $[M + H]^+$ 313.1916, found 313.1915.

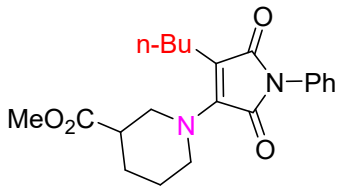
3-butyl-4-(4-methylpiperidin-1-yl)-1-phenyl-1H-pyrrole-2,5-dione



3c

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (45.6 mg, 70% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.46-7.42 (m, 2H), 7.39-7.36 (m, 2H), 7.34-7.30 (m, 1H), 4.34-4.30(m, 2H), 3.09 (td, *J* = 12.6, 2.5 Hz, 2H), 2.49 (dd, *J* = 8.6, 6.9 Hz, 2H), 1.80-1.75 (m, 2H), 1.67 (td, *J* = 11.3, 9.6, 5.7 Hz, 1H), 1.53 (qd, *J* = 7.8, 7.3, 4.0 Hz, 1H), 1.46-1.31 (m, 4H), 1.03-0.96 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.55, 167.48, 144.31, 132.32, 128.79, 127.01, 126.10, 106.65, 49.40, 34.69, 32.63, 30.77, 23.45, 22.78, 21.88, 13.95. **HRMS** (ESI): calcd for $C_{20}H_{27}N_2O_2$ $[M + H]^+$ 327.2073, found 327.2080.

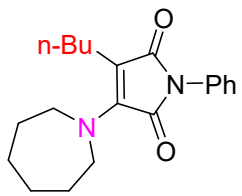
methyl 1-(4-butyl-2,5-dioxo-1-phenyl-2,5-dihydro-1H-pyrrol-3-yl)piperidine-3-carboxylate



3d

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (48.8 mg, 66% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.46-7.42 (m, 2H), 7.37-7.32 (m, 3H), 4.22 (dd, *J* = 13.4, 4.1 Hz, 2H), 3.74 (s, 3H), 3.26 (ddd, *J* = 13.6, 10.9, 2.9 Hz, 2H), 2.62 (tt, *J* = 10.5, 4.1 Hz, 1H), 2.50-2.46 (m, 2H), 2.07-2.02 (m, 2H), 1.90 (dtd, *J* = 14.3, 10.8, 3.9 Hz, 2H), 1.53 (tt, *J* = 7.8, 5.9 Hz, 2H), 1.45-1.40 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 174.63, 171.36, 167.37, 143.90, 132.16, 128.83, 127.13, 126.09, 107.92, 51.96, 48.29, 40.48, 32.51, 28.49, 27.90, 26.89, 23.42, 22.78, 13.92. **HRMS** (ESI): calcd for $C_{21}H_{27}N_2O_4$ $[M + H]^+$ 371.1971, found 371.1980.

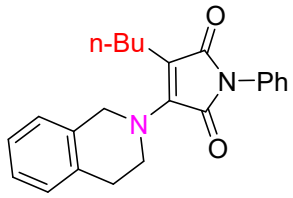
3-(azepan-1-yl)-4-butyl-1-phenyl-1H-pyrrole-2,5-dione



3e

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (32.6 mg, 50% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.45 (t, *J* = 7.7 Hz, 2H), 7.39-7.37 (m, 2H), 7.34-7.31 (m, 1H), 3.80 (t, *J* = 6.0 Hz, 4H), 2.52-2.48 (m, 2H), 1.87-1.84 (m, 4H), 1.70-1.63 (m, 4H), 1.53-1.49 (m, 2H), 1.45-1.38 (m, 2H), 0.97 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.72, 167.03, 143.35, 132.39, 128.77, 126.95, 126.15, 102.51, 52.07, 33.77, 29.01, 26.84, 23.59, 22.67, 13.99. **HRMS** (ESI): calcd for $C_{20}H_{27}N_2O_2$ $[M + H]^+$ 327.2073, found 327.2083.

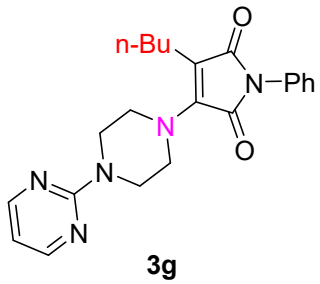
3-butyl-4-(3,4-dihydroisoquinolin-2(1H)-yl)-1-phenyl-1H-pyrrole-2,5-dione



3f

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (47.5 mg, 66% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.49-7.45 (m, 2H), 7.41-7.38 (m, 2H), 7.36-7.32 (m, 1H), 7.27-7.25 (m, 2H), 7.23-7.20 (m, 1H), 7.16-7.14 (m, 1H), 4.94 (s, 2H), 4.07 (t, *J* = 5.9 Hz, 2H), 3.06 (t, *J* = 5.9 Hz, 2H), 2.60-2.56 (m, 2H), 1.59 (tt, *J* = 7.6, 5.9 Hz, 2H), 1.48 (p, *J* = 7.2 Hz, 2H), 1.01 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.46, 167.35, 143.66, 134.24, 133.26, 132.19, 128.96, 128.87, 127.16, 127.01, 126.56, 126.20, 126.13, 106.79, 50.76, 46.71, 33.10, 29.27, 23.52, 22.81, 13.99. **HRMS** (ESI): calcd for $C_{23}H_{25}N_2O_2$ $[M + H]^+$ 361.1916, found 361.1921.

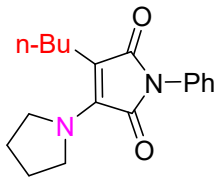
3-butyl-1-phenyl-4-(4-(pyrimidin-2-yl)piperazin-1-yl)-1H-pyrrole-2,5-dione



3g

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (63.4 mg, 81% yield), Mp = 92-93℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.38 (d, *J* = 4.8 Hz, 2H), 7.48-7.44 (m, 2H), 7.39-7.32 (m, 3H), 6.59 (t, *J* = 4.8 Hz, 1H), 4.02-4.00 (m, 4H), 3.85-3.83 (m, 4H), 2.55-2.51 (m, 2H), 1.56 (ddd, *J* = 9.9, 6.4, 2.7 Hz, 2H), 1.47-1.41 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.27, 167.44, 161.55, 157.89, 143.76, 132.07, 128.89, 127.25, 126.10, 110.59, 108.77, 48.49, 43.97, 32.74, 23.39, 22.80, 13.93. **HRMS** (ESI): calcd for $C_{22}H_{26}N_5O_2$ $[M + H]^+$ 392.2087, found 392.2083.

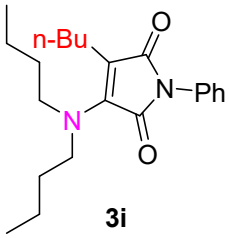
3-butyl-1-phenyl-4-(pyrrolidin-1-yl)-1H-pyrrole-2,5-dione



3h

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (32.2 mg, 54% yield), Mp = 44-45℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.47-7.42 (m, 2H), 7.40-7.37 (m, 2H), 7.33-7.31 (m, 1H), 3.88-3.85 (m, 4H), 2.56-2.52 (m, 2H), 2.00-1.97 (m, 4H), 1.57-1.49 (m, 2H), 1.45-1.38 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 172.02, 166.94, 142.16, 132.49, 128.77, 126.87, 126.07, 101.37, 50.44, 34.84, 25.40, 22.89, 22.69, 14.04. **HRMS** (ESI): calcd for $C_{18}H_{23}N_2O_2$ $[M + H]^+$ 299.1760, found 299.1761.

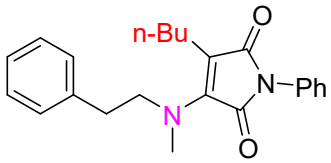
3-butyl-4-(dibutylamino)-1-phenyl-1H-pyrrole-2,5-dione



3i

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (49.2 mg, 69% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.47-7.43 (m, 2H), 7.40-7.37 (m, 2H), 7.34-7.32 (m, 1H), 3.60-3.56 (m, 4H), 2.46-2.42 (m, 2H), 1.69-1.61 (m, 4H), 1.53-1.34 (m, 8H), 1.02-0.98 (m, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.58, 167.23, 142.95, 132.39, 128.80, 126.97, 126.19, 103.21, 51.76, 33.52, 31.24, 23.46, 22.73, 19.94, 14.01, 13.97. **HRMS** (ESI): calcd for $C_{22}H_{33}N_2O_2$ $[M + H]^+$ 357.2542, found 357.2550.

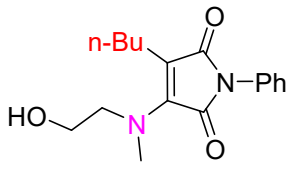
3-butyl-4-(methyl(phenethyl)amino)-1-phenyl-1H-pyrrole-2,5-dione



3j

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (43.5 mg, 60% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.48-7.44 (m, 2H), 7.37-7.26 (m, 8H), 3.98-3.94 (m, 2H), 3.26 (s, 3H), 2.99 (dd, *J* = 8.6, 6.5 Hz, 2H), 2.53-2.49(m, 2H), 1.53-1.45 (m, 2H), 1.43-1.36 (m, 2H), 0.97 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.43, 167.08, 143.61, 138.36, 132.18, 128.96, 128.77, 128.60, 127.03, 126.61, 126.14, 104.67, 55.27, 40.41, 35.05, 33.71, 23.24, 22.63, 13.92. **HRMS** (ESI): calcd for $C_{23}H_{27}N_2O_2$ $[M + H]^+$ 363.2073, found 363.2083.

3-butyl-4-((2-hydroxyethyl)(methyl)amino)-1-phenyl-1H-pyrrole-2,5-dione

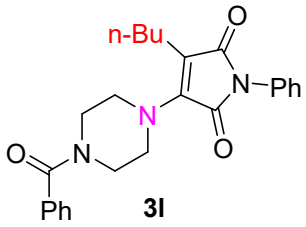


3k

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a

yellow liquid (39.3 mg, 65% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.47-7.44 (m, 2H), 7.37-7.32 (m, 3H), 3.90-3.85 (m, 4H), 3.30 (s, 3H), 2.58-2.54 (m, 2H), 2.36 (t, *J* = 5.1 Hz, 1H), 1.58-1.51 (m, 2H), 1.46-1.41 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.33, 168.12, 144.54, 132.04, 128.89, 127.29, 126.16, 106.13, 60.84, 55.18, 40.12, 33.82, 23.23, 22.70, 13.97. **HRMS** (ESI): calcd for $C_{17}H_{23}N_2O_3$ $[M + H]^+$ 303.1709, found 303.1721.

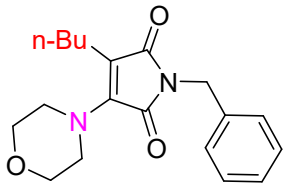
3-(4-benzoylpiperazin-1-yl)-4-butyl-1-phenyl-1H-pyrrole-2,5-dione



3l

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (58.4 mg, 70% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.48-7.44 (m, 7H), 7.37-7.34 (m, 3H), 3.94-3.75 (m, 8H), 2.51-2.47 (m, 2H), 1.53 (qd, *J* = 7.7, 7.3, 4.0 Hz, 2H), 1.43 (q, *J* = 7.3 Hz, 2H), 0.98 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.04, 170.67, 167.33, 143.42, 135.13, 131.91, 130.23, 128.94, 128.75, 127.37, 127.24, 126.05, 110.21, 48.81, 48.69, 32.60, 23.35, 22.81, 13.93. **HRMS** (ESI): calcd for $C_{25}H_{28}N_3O_3$ $[M + H]^+$ 418.2131, found 418.2143.

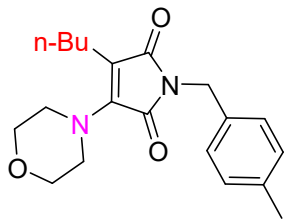
1-benzyl-3-butyl-4-morpholino-1H-pyrrole-2,5-dione



4a

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (47.9 mg, 73% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.39-7.29 (m, 5H), 4.63 (s, 2H), 3.80 (t, J = 4.7 Hz, 4H), 3.69 (t, J = 4.7 Hz, 4H), 2.43-2.39 (m, 2H), 1.48-1.35 (m, 4H), 0.97-0.94 (m, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 172.20, 168.31, 143.67, 137.00, 128.63, 128.50, 127.61, 108.50, 66.96, 48.71, 41.34, 32.89, 23.17, 22.75, 13.92. **HRMS** (ESI): calcd for $C_{19}H_{25}N_2O_3$ $[M + H]^+$ 329.1865, found 329.1870.

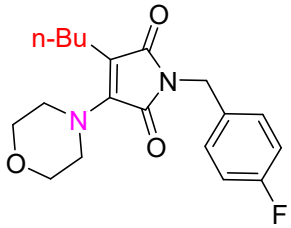
3-butyl-1-(4-methylbenzyl)-4-morpholino-1H-pyrrole-2,5-dione



4b

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (51.3 mg, 75% yield), Mp = 50-51℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.28 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 7.9 Hz, 2H), 4.59 (s, 2H), 3.81-3.78 (m, 4H), 3.69-3.67 (m, 4H), 2.42-2.38 (m, 2H), 2.35 (s, 3H), 1.47-1.33 (m, 4H), 0.97-0.94 (m, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 172.24, 168.32, 143.67, 137.32, 134.05, 129.29, 128.54, 108.51, 66.96, 48.70, 41.07, 32.88, 23.16, 22.75, 21.19, 13.92. **HRMS** (ESI): calcd for $C_{20}H_{27}N_2O_3$ $[M + H]^+$ 343.2022, found 343.2022.

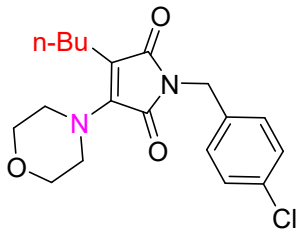
3-butyl-1-(4-fluorobenzyl)-4-morpholino-1H-pyrrole-2,5-dione



4c

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (42.2 mg, 61% yield), Mp = 65-66℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.37-7.33 (m, 2H), 7.03-6.99 (m, 2H), 4.59 (s, 2H), 3.81-3.79 (m, 4H), 3.70-3.67 (m, 4H), 2.42-2.38 (m, 2H), 1.47-1.35 (m, 4H), 0.95 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 172.12, 168.25, 162.29 (d, *J* = 245.6 Hz). 143.69, 132.82, 130.37 (d, *J* = 8.0 Hz), 115.46 (d, *J* = 21.4 Hz), 108.45, 66.94, 48.71, 40.61, 32.88, 23.16, 22.74, 13.90. **^{19}F NMR** (375 MHz, $CDCl_3$) δ -114.77; **HRMS** (ESI): calcd for $C_{19}H_{24}N_2O_3F$ $[M + H]^+$ 347.1771, found 347.1759.

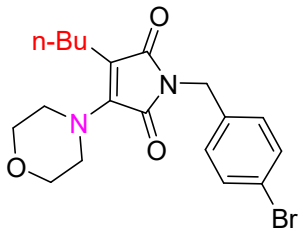
3-butyl-1-(4-chlorobenzyl)-4-morpholino-1H-pyrrole-2,5-dione



4d

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (45.6 mg, 63% yield), Mp = 75-76℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.30 (brs, 4H), 4.58 (s, 2H), 3.81-3.79 (m, 4H), 3.70-3.67 (m, 4H), 2.42-2.38 (m, 2H), 1.46-1.37 (m, 4H), 0.97-0.93 (m, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 172.05, 168.21, 143.69, 135.43, 133.53, 129.98, 128.79, 108.42, 66.94, 48.70, 40.67, 32.87, 23.17, 22.74, 13.91. **HRMS** (ESI): calcd for $C_{19}H_{24}N_2O_3Cl$ $[M + H]^+$ 363.1475, found 363.1470.

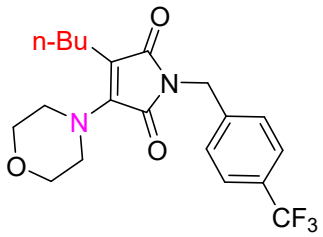
1-(4-bromobenzyl)-3-butyl-4-morpholino-1H-pyrrole-2,5-dione



4e

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (53.6 mg, 66% yield), Mp = 82-83℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.45 (d, J = 8.4 Hz, 2H), 7.25 (d, J = 8.4 Hz, 2H), 4.57 (s, 2H), 3.81-3.79 (m, 4H), 3.70-3.67 (m, 4H), 2.42-2.38 (m, 2H), 1.47-1.35 (m, 4H), 0.95 (t, J = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 172.03, 168.19, 143.69, 135.94, 131.75, 130.32, 129.98, 128.78, 121.67, 108.40, 66.93, 48.70, 40.72, 32.86, 23.16, 22.74, 13.91. **HRMS** (ESI): calcd for $C_{19}H_{24}N_2O_3Br$ $[M + H]^+$ 407.0970, found 407.0971.

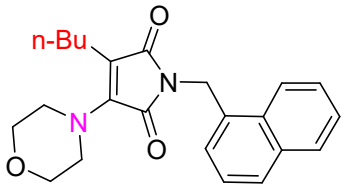
3-butyl-4-morpholino-1-(4-(trifluoromethyl)benzyl)-1H-pyrrole-2,5-dione



4f

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (55.4 mg, 70% yield), Mp = 79-80℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.60 (d, *J* = 8.0 Hz, 2H), 7.47 (d, *J* = 8.0 Hz, 2H), 4.67 (s, 2H), 3.82-3.80 (m, 4H), 3.71-3.69 (m, 4H), 2.44-2.40 (m, 2H), 1.49-1.38 (m, 4H), 0.98-0.094 (m, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.98, 168.18, 143.70, 140.81, 129.90 (d, *J* = 32.3 Hz), 128.72, 125.64 (q, *J* = 3.7 Hz), 124.05 (d, *J* = 271.0 Hz), 108.38, 66.93, 48.71, 40.86, 32.86, 23.18, 22.73, 13.89. **^{19}F NMR** (375 MHz, $CDCl_3$) δ -62.56 (3F); **HRMS** (ESI): calcd for $C_{20}H_{24}N_2O_3F_3$ $[M + H]^+$ 397.1739, found 397.1745.

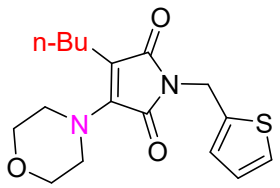
3-butyl-4-morpholino-1-(naphthalen-1-ylmethyl)-1H-pyrrole-2,5-dione



4g

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (53.7 mg, 71% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.34 (d, *J* = 8.4 Hz, 1H), 7.89 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.83 (d, *J* = 8.2 Hz, 1H), 7.57 (tt, *J* = 14.2, 7.0 Hz, 3H), 7.49-7.45 (m, 1H), 5.11 (s, 2H), 3.81-3.79 (m, 4H), 3.69-3.67 (m, 4H), 2.45-2.41 (m, 2H), 1.49-1.37 (m, 4H), 0.96 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 172.34, 168.54, 143.67, 133.81, 132.16, 131.34, 128.73, 128.49, 127.45, 126.45, 125.80, 125.39, 123.69, 108.56, 66.95, 48.71, 39.35, 32.90, 23.21, 22.75, 13.93. **HRMS** (ESI): calcd for $C_{23}H_{27}N_2O_3$ $[M + H]^+$ 379.2022, found 379.2036.

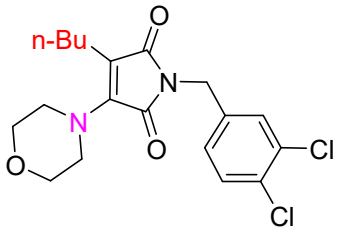
3-butyl-4-morpholino-1-(thiophen-2-ylmethyl)-1H-pyrrole-2,5-dione



4h

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (33.4 mg, 50% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.23 (dd, *J* = 5.1, 1.3 Hz, 1H), 7.08 (t, *J* = 2.2 Hz, 1H), 6.96 (dd, *J* = 5.1, 3.5 Hz, 1H), 4.80 (s, 2H), 3.82-3.80 (m, 2H), 3.71-3.68 (m, 2H), 2.43-2.39 (m, 2H), 1.48-1.38 (m, 4H), 0.98-0.94 (m, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.70, 167.90, 143.72, 138.90, 127.27, 126.86, 125.60, 108.51, 66.95, 48.70, 35.48, 32.84, 23.14, 22.72, 13.90. **HRMS** (ESI): calcd for $C_{17}H_{23}N_2O_3S$ $[M + H]^+$ 335.1429, found 335.1439.

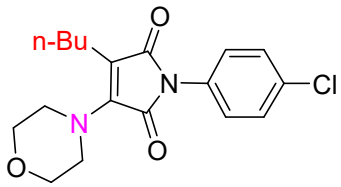
3-butyl-1-(3,4-dichlorobenzyl)-4-morpholino-1H-pyrrole-2,5-dione



4i

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (40.4 mg, 51% yield), Mp = 99-100℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.45 (d, *J* = 2.1 Hz, 1H), 7.40 (d, *J* = 8.2 Hz, 1H), 7.21 (dd, *J* = 8.2, 2.1 Hz, 1H), 4.56 (s, 2H), 3.82-3.80 (m, 4H), 3.71-3.69 (m, 4H), 2.43-2.39 (m, 2H), 1.48-1.36 (m, 4H), 0.95 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.87, 168.10, 143.71, 137.06, 132.63, 131.84, 130.61, 130.48, 127.97, 108.35, 66.93, 48.71, 40.25, 32.84, 23.18, 22.73, 13.90. **HRMS** (ESI): calcd for $C_{19}H_{23}N_2O_3Cl_2$ $[M + H]^+$ 397.1086, found 397.1095.

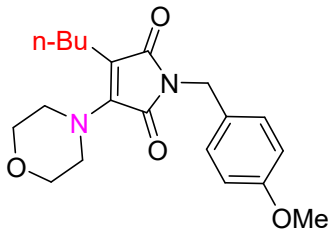
3-butyl-1-(4-chlorophenyl)-4-morpholino-1H-pyrrole-2,5-dione



4j

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (46.6 mg, 67% yield), Mp = 80-81℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.42 (d, *J* = 8.8 Hz, 2H), 7.33 (d, *J* = 8.8 Hz, 2H), 3.86-3.83 (m, 4H), 3.77-3.74 (m, 4H), 2.50-2.46 (m, 2H), 1.54-1.39 (m, 4H), 0.98 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 170.85, 167.04, 143.59, 132.77, 130.57, 129.04, 127.09, 108.88, 66.99, 48.92, 32.71, 23.26, 22.76, 13.92. **HRMS** (ESI): calcd for $C_{18}H_{22}N_2O_3Cl$ $[M + H]^+$ 349.1319, found 349.1333.

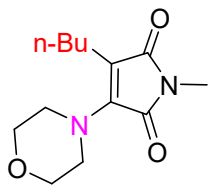
3-butyl-1-(4-methoxybenzyl)-4-morpholino-1H-pyrrole-2,5-dione



4k

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (50.1 mg, 70% yield), Mp = 62-63℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.33 (d, *J* = 7.1 Hz, 2H), 6.87 (d, *J* = 6.9 Hz, 2H), 4.57 (s, 2H), 3.82-3.79 (m, 7H), 3.70-3.68 (m, 4H), 2.42-2.38 (m, 2H), 1.46-1.38 (m, 4H), 0.98-0.94 (m, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 172.26, 168.33, 159.08, 143.68, 130.00, 129.28, 113.95, 108.50, 66.96, 55.30, 48.70, 40.77, 32.89, 23.15, 22.75, 13.91. **HRMS** (ESI): calcd for $C_{20}H_{27}N_2O_4$ $[M + H]^+$ 359.1971, found 359.1982.

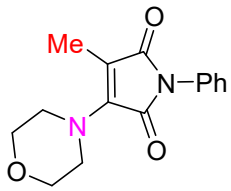
3-butyl-1-methyl-4-morpholino-1H-pyrrole-2,5-dione



4l

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (37.3 mg, 74% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 3.82-3.80 (m, 4H), 3.70-3.68 (m, 4H), 2.96 (s, 3H), 2.42-2.39 (m, 2H), 1.48-1.35 (m, 4H), 0.95 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 172.69, 168.78, 143.77, 108.73, 66.98, 48.75, 32.92, 23.64, 23.08, 22.70, 13.92. **HRMS** (ESI): calcd for $C_{13}H_{21}N_2O_3$ $[M + H]^+$ 253.1552, found 253.1556.

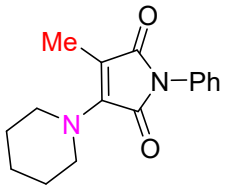
3-methyl-4-morpholino-1-phenyl-1H-pyrrole-2,5-dione



5a

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (35.9 mg, 66% yield), Mp = 96-97℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.48-7.44 (m, 2H), 7.36-7.33 (m, 3H), 3.86-3.81 (m, 8H), 2.11 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.35, 167.19, 143.95, 132.01, 128.94, 127.35, 126.16, 103.22, 67.14, 48.85, 9.28. **HRMS** (ESI): calcd for $C_{15}H_{17}N_2O_3$ $[M + H]^+$ 273.1239, found 273.1248.

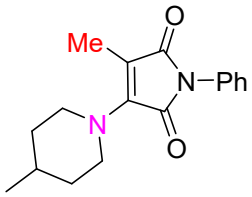
3-methyl-1-phenyl-4-(piperidin-1-yl)-1H-pyrrole-2,5-dione



5b

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (34.6 mg, 64% yield), Mp = 71-72℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.47-7.43 (m, 2H), 7.38-7.31 (m, 3H), 3.77-3.74 (m, 4H), 2.10 (s, 3H), 1.74-1.72 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 167.30, 144.86, 132.32, 128.84, 127.07, 126.16, 100.92, 49.98, 26.72, 24.29, 9.37. **HRMS** (ESI): calcd for $C_{16}H_{19}N_2O_2$ $[M + H]^+$ 271.1447, found 271.1453.

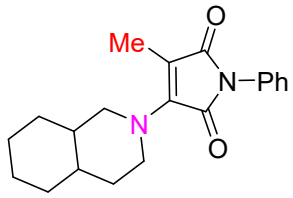
3-methyl-4-(4-methylpiperidin-1-yl)-1-phenyl-1H-pyrrole-2,5-dione



5c

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (34.7 mg, 61% yield), Mp = 65-66℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.47-7.43 (m, 2H), 7.38-7.31 (m, 3H), 4.41 (dt, *J* = 13.2, 2.3 Hz, 2H), 3.12 (td, *J* = 13.1, 12.7, 2.5 Hz, 2H), 2.10 (s, 3H), 1.78 (dd, *J* = 13.3, 2.7 Hz, 2H), 1.69 (ddq, *J* = 11.0, 6.8, 3.6 Hz, 1H), 1,40-1,32 (m, 2H), 1.02 (d, *J* = 6.5 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.68, 167.31, 144.79, 132.31, 128.85, 127.09, 126.17, 101.04, 49.28, 34.90, 30.83, 21.89, 9.37. **HRMS** (ESI): calcd for $C_{17}H_{21}N_2O_2$ [M + H]$^+$ 285.1603, found 285.1600.

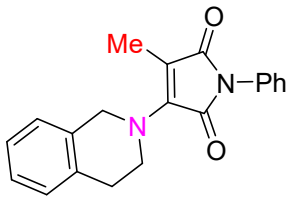
3-methyl-4-(octahydroisoquinolin-2(1H)-yl)-1-phenyl-1H-pyrrole-2,5-dione



5d

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (45.4 mg, 70% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.48-7.44 (m, 2H), 7.39-7.31 (m, 3H), 3.43 (ddq, *J* = 18.1, 12.3, 5.9, 5.5 Hz, 2H), 3.26 (td, *J* = 10.3, 3.1 Hz, 1H), 2.06 (s, 3H), 1.94-1.90 (m, 1H), 1.82-1.71 (m, 6H), 1.52-1.9 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.30, 168.02, 146.55, 132.14, 128.91, 127.20, 126.07, 104.46, 63.30, 48.96, 40.81, 32.97, 32.31, 29.45, 26.11, 25.26, 25.05, 9.11. **HRMS** (ESI): calcd for $C_{20}H_{25}N_2O_2$ [M + H]$^+$ 325.1916, found 325.1908.

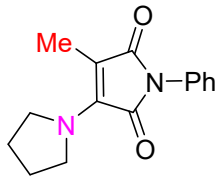
3-(3,4-dihydroisoquinolin-2(1H)-yl)-4-methyl-1-phenyl-1H-pyrrole-2,5-dione



5e

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (37.5 mg, 59% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.48-7.44 (m, 2H), 7.39-7.34 (m, 3H), 7.27-7.20 (m, 3H), 7.16-7.14 (m, 1H), 5.00 (s, 2H), 4.08 (t, *J* = 5.9 Hz, 2H), 3.06 (t, *J* = 5.9 Hz, 2H), 2.19 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.60, 167.19, 144.24, 134.12, 133.36, 132.20, 129.01, 128.91, 127.23, 127.00, 126.58, 126.20, 101.17, 50.68, 46.54, 29.43, 9.33. **HRMS** (ESI): calcd for $C_{20}H_{19}N_2O_2$ $[M + H]^+$ 319.1447, found 319.1442.

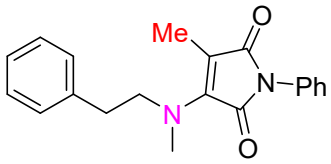
3-methyl-1-phenyl-4-(pyrrolidin-1-yl)-1H-pyrrole-2,5-dione



5f

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (25.6 mg, 50% yield), Mp = 96-97℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.47-7.43 (m, 2H), 7.39-7.36 (m, 2H), 7.34-7.32 (m, 1H), 3.93-3.90 (m, 4H), 2.17 (s, 3H), 1.99-1.96 (m, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 172.23, 166.78, 149.03, 132.48, 128.82, 126.96, 126.13, 95.79, 50.42, 25.38, 8.39. **HRMS** (ESI): calcd for $C_{15}H_{17}N_2O_2$ $[M + H]^+$ 257.1290, found 257.1289.

3-methyl-4-(methyl(phenethyl)amino)-1-phenyl-1H-pyrrole-2,5-dione

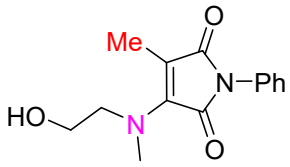


5g

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a

yellow solid (37.1 mg, 58% yield), Mp = 68-69℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.49-7.45 (m, 2H), 7.36-7.32 (m, 5H), 7.30-7.27 (m, 3H), 3.95 (t, *J* = 8.6 Hz, 2H), 3.31 (s, 3H), 3.00 (t, *J* = 8.6 Hz, 2H), 2.12 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.64, 167.01, 144.46, 138.41, 132.26, 129.01, 128.90, 128.69, 127.18, 126.75, 126.27, 99.33, 55.19, 40.40, 35.21, 9.17. **HRMS** (ESI): calcd for $C_{20}H_{21}N_2O_2$ $[M + H]^+$ 321.1603, found 321.1602.

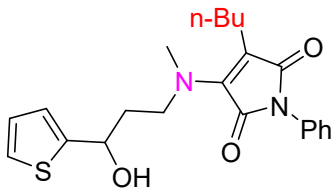
3-((2-hydroxyethyl)(methyl)amino)-4-methyl-1-phenyl-1H-pyrrole-2,5-dione



5h

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (31.7 mg, 61% yield), Mp = 84-85℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.48-7.44 (m, 2H), 7.36-7.32 (m, 3H), 3.91-3.87 (m, 4H), 3.36 (s, 3H), 2.26 (brs, 1H), 2.17 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.50, 167.90, 145.08, 132.05, 128.92, 127.34, 126.22, 100.38, 60.84, 54.98, 40.19, 9.19. **HRMS** (ESI): calcd for $C_{14}H_{17}N_2O_3$ $[M + H]^+$ 261.1239, found 261.1243.

3-butyl-4-((3-hydroxy-3-(thiophen-2-yl)propyl)(methyl)amino)-1-phenyl-1H-pyrrole-2,5-dione

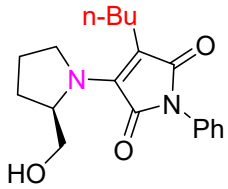


6a

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (54.1 mg, 68% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.48-7.43 (m, 2H), 7.38-7.31 (m, 3H), 7.30-7.28 (m, 1H), 7.03-6.99 (m, 2H), 5.07 (t, *J* = 6.4 Hz, 1H), 3.93 (ddd, *J* = 14.2, 8.1, 7.1 Hz, 1H), 3.75 (ddd, *J* = 13.9, 8.2, 5.3 Hz, 1H), 3.27 (s, 3H), 2.53 (td, *J* = 7.1, 1.9 Hz, 2H), 2.26-2.19 (m, 2H), 1.54-1.47 (m, 2H), 1.46-1.38 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.45, 167.51, 148.04, 144.09, 132.12, 128.86, 127.20, 126.81, 126.19, 124.80, 123.75, 105.62, 67.65, 50.43, 39.77, 37.62, 33.86, 23.26, 22.70, 14.01. **HRMS** (ESI): calcd for

$C_{22}H_{27}N_2O_3S$ $[M + H]^+$ 399.1742, found 399.1748.

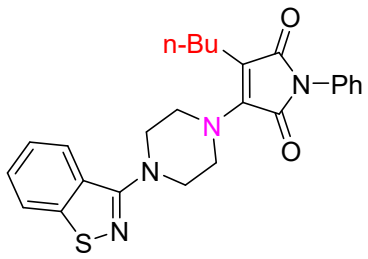
(R)-3-butyl-4-(2-(hydroxymethyl)pyrrolidin-1-yl)-1-phenyl-1H-pyrrole-2,5-dione



6b

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (47.2 mg, 72% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.47-7.43 (m, 2H), 7.38-7.31 (m, 3H), 4.88-4.83 (m, 1H), 3.94-3.90 (m, 1H), 3.72-3.55 (m, 3H), 2.63-2.56 (m, 1H), 2.51-2.44 (m, 1H), 2.07-1.98 (m, 5H), 1.55-1.39 (m, 4H), 0.97 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.67, 167.59, 142.15, 132.23, 128.84, 127.14, 126.18, 103.26, 65.25, 61.11, 50.62, 34.51, 27.77, 23.18, 23.05, 22.69, 14.02. **HRMS** (ESI): calcd for $C_{19}H_{25}N_2O_3$ $[M + H]^+$ 329.1865, found 329.1870.

3-(4-(benzo[d]isothiazol-3-yl)piperazin-1-yl)-4-butyl-1-phenyl-1H-pyrrole-2,5-dione



6c

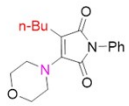
Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (61.6 mg, 69% yield), Mp = 59-60℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.96 (d, *J* = 8.2 Hz, 1H), 7.88 (d, *J* = 8.2 Hz, 1H), 7.56-7.52 (m, 1H), 7.46 (q, *J* = 7.4, 7.0 Hz, 3H), 7.42-7.33 (m, 3H), 4.01-3.99 (m, 4H), 3.73-3.71 (m, 4H), 2.58-2.54 (m, 2H), 1.61-1.55(m, 2H), 1.49-1.44 (m, 2H), 1.00 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.27, 167.44, 163.38, 152.99, 143.66, 132.07, 128.91, 127.86, 127.27, 126.13, 124.24, 123.70, 120.78, 108.96, 50.35, 48.41, 32.67, 23.43, 22.83, 13.97. **HRMS** (ESI): calcd for $C_{25}H_{27}N_4O_2S$ $[M + H]^+$ 447.1855, found 447.1850.

References:

(1) (a) Ding, G.; Li, C.; Shen, Y.; Lu, B.; Zhang, Z.; Xie, X. *Adv. Synth. Catal.* **2016**, *358*, 1241-1250. (b) Matuszak, N.; Muccioli, G. G.; Labar, G.; Lambert, D. M. *J. Med Chem*. **2009**, *52*, 7410-7420.

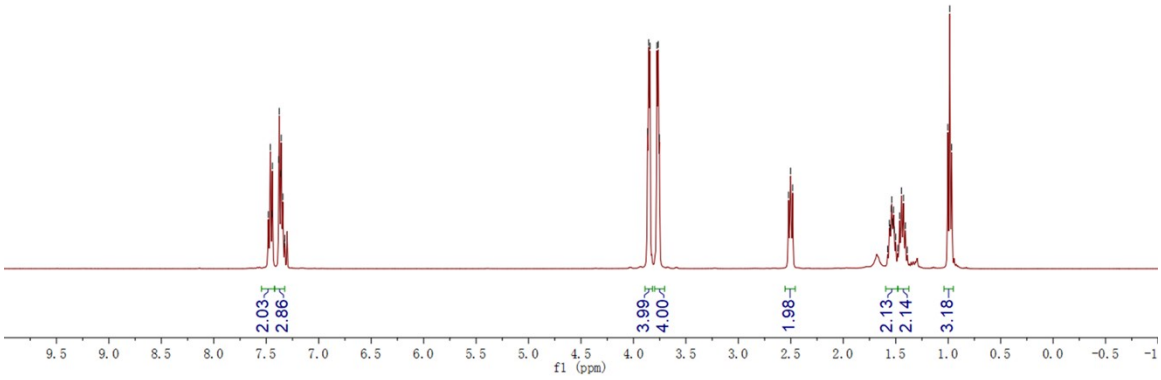
(2) K. Takahashi, Y. Ogiwara, N. Sakai, Chem. Asian J. 2018, 13, 809.

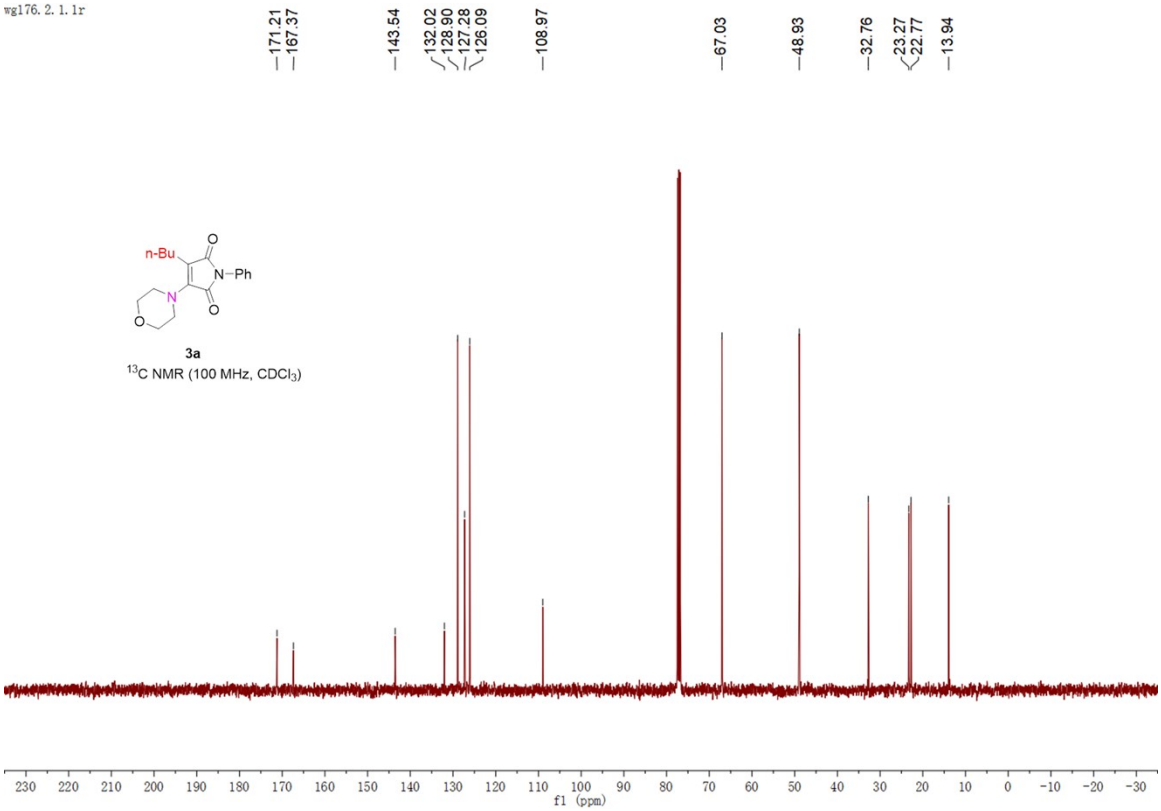
^{1}H, ^{13}C and ^{19}F NMR spectra of products



3a

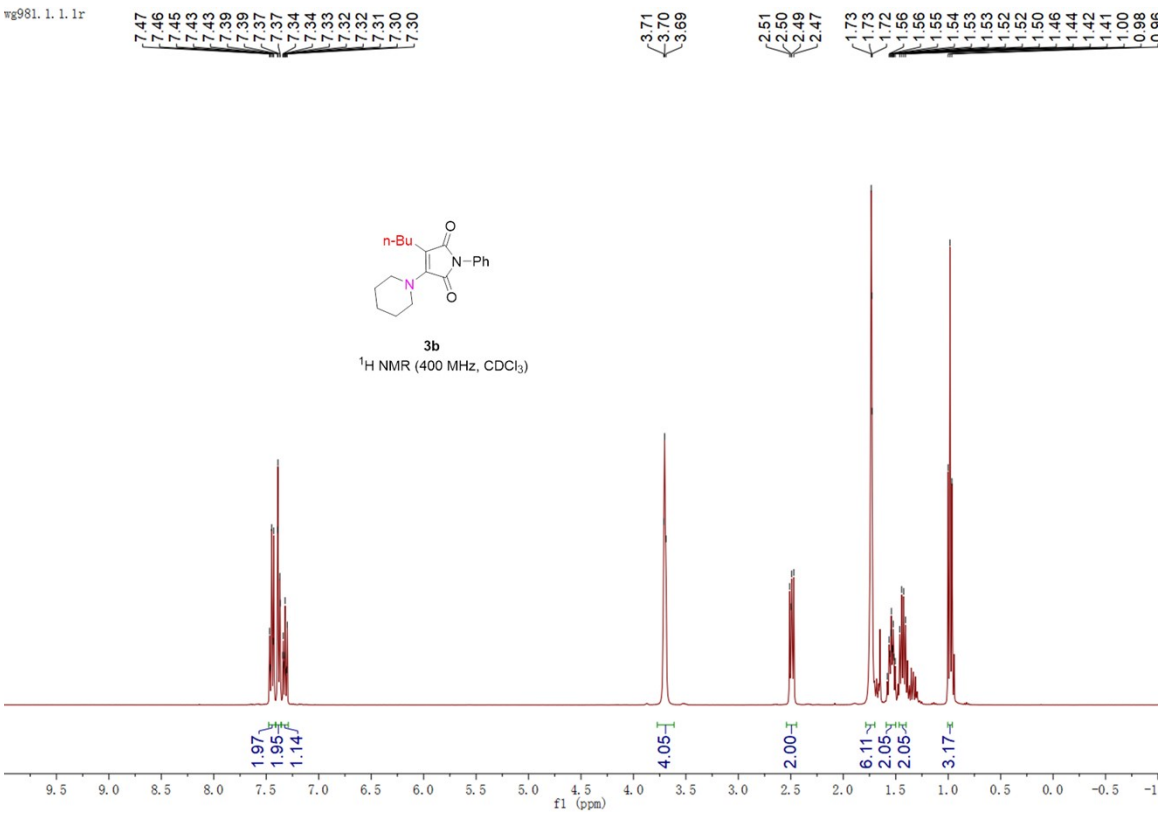
^{1}H NMR (400 MHz, $CDCl_3$)





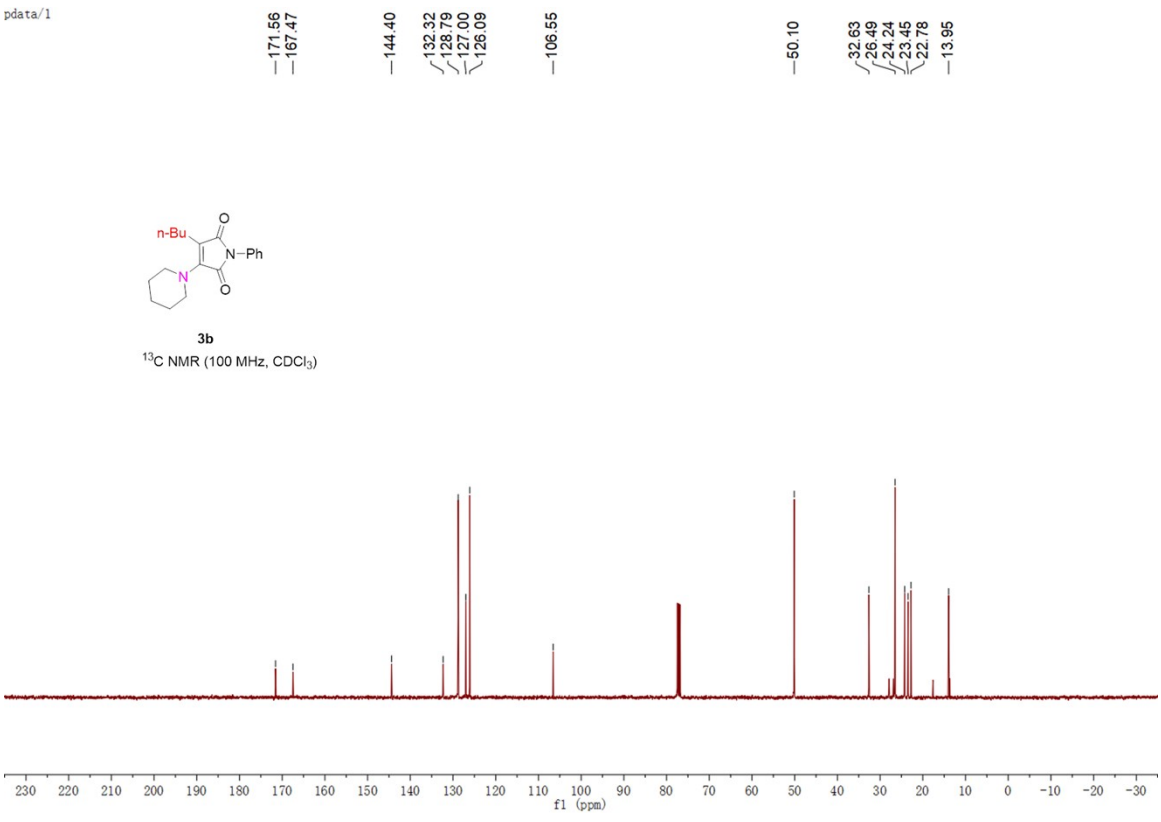
3a

^{13}C NMR (100 MHz, $CDCl_3$)



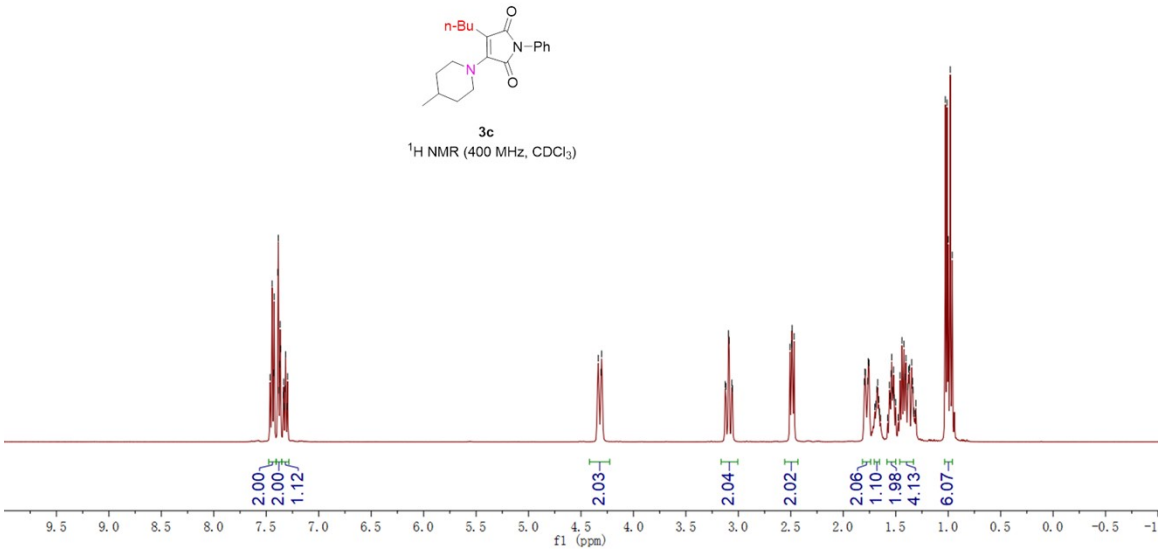
3b

^{1}H NMR (400 MHz, $CDCl_3$)



3b

^{13}C NMR (100 MHz, $CDCl_3$)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O
N
3c
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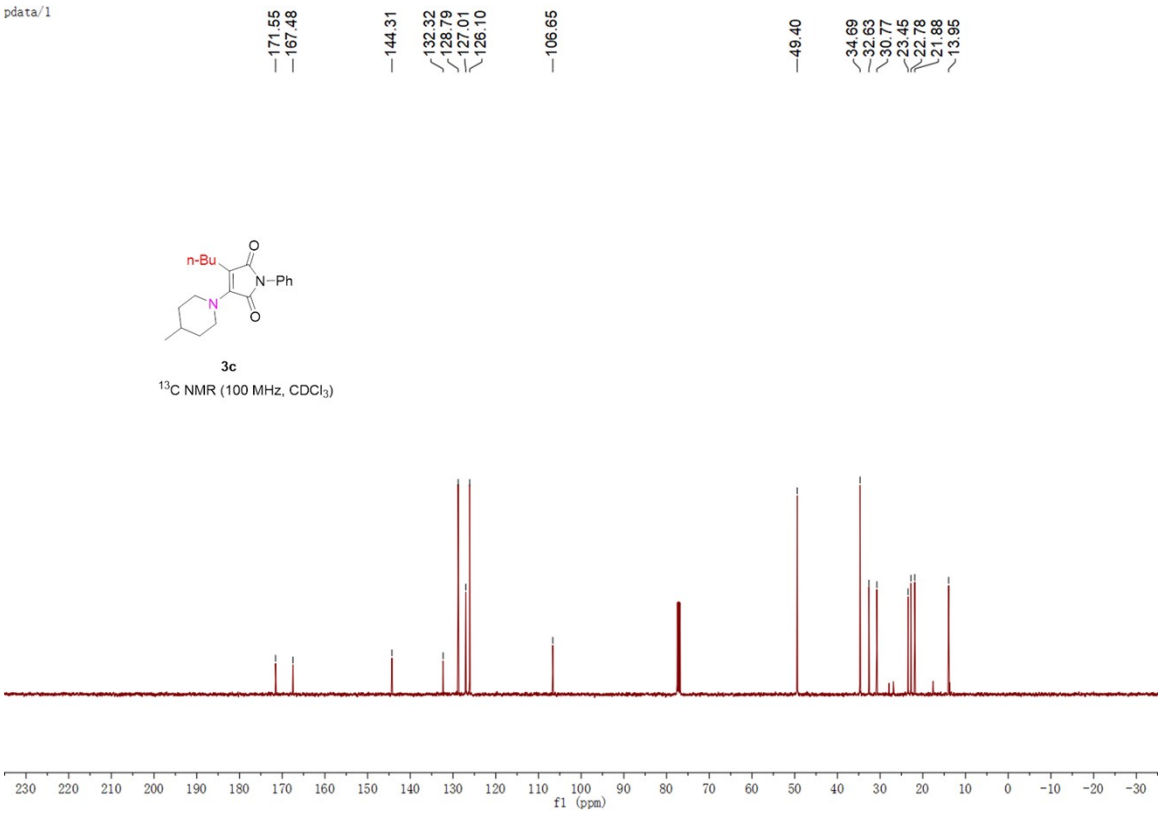
2.04
2.02
2.06
1.10
1.98
4.13
6.07
f1 (ppm)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49.40
34.69
32.63
30.77
23.45
22.78
21.88
13.95
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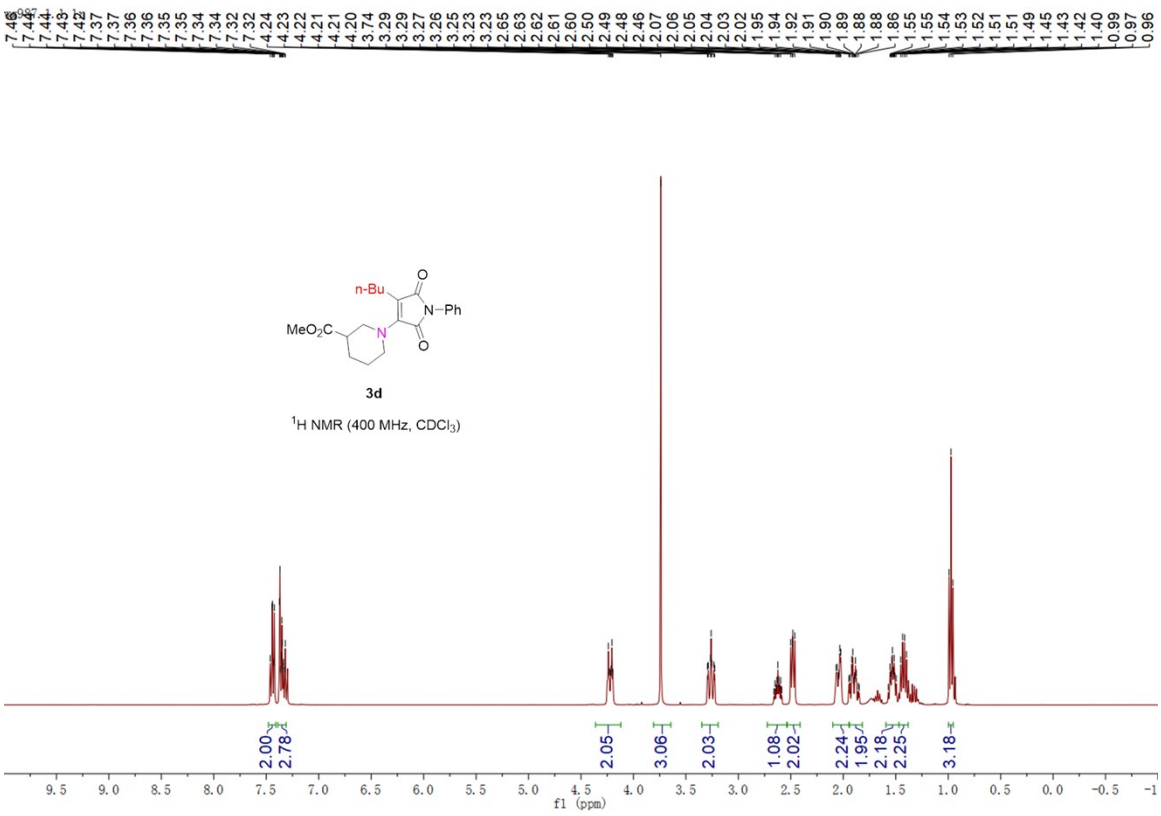
N–Ph
O
O
N
3c
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)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N–Ph
MeO2C
3d
1H NMR (400 MHz, CDCl3)
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2.05
3.06
2.03
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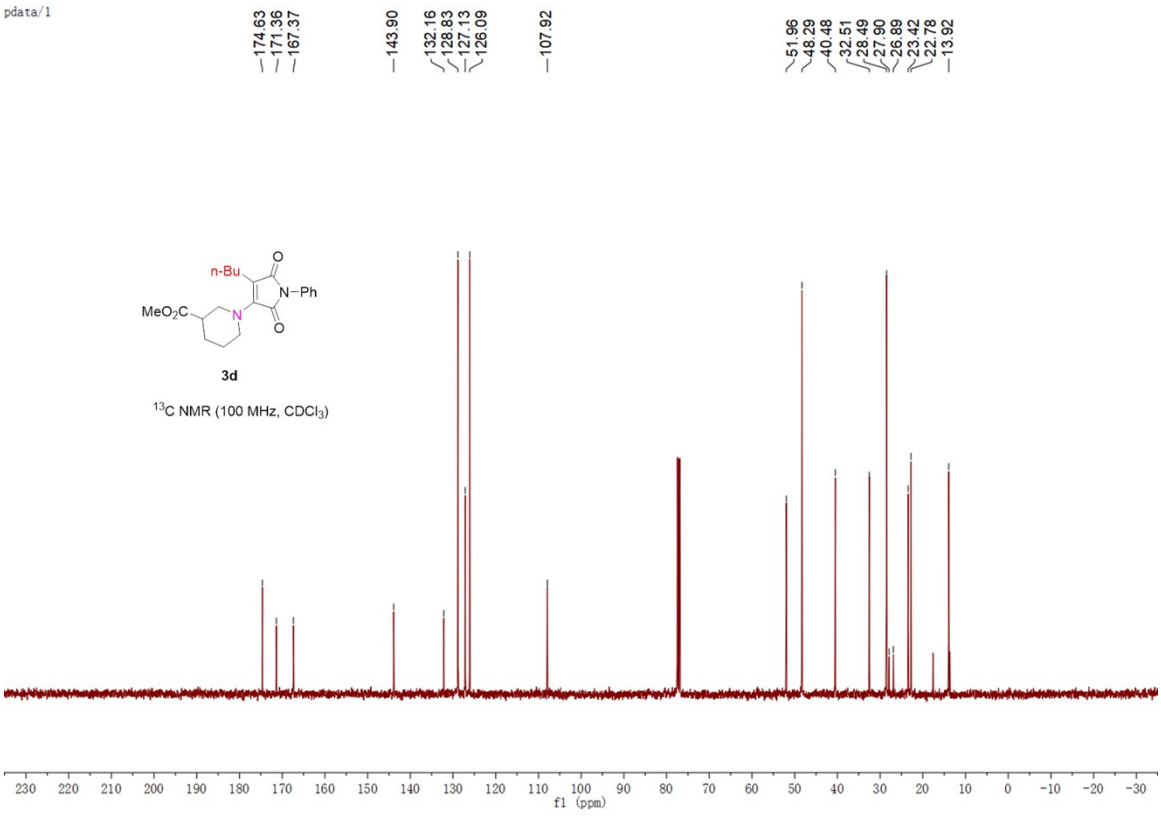
2.24
1.95
2.18
2.25
3.18
f1 (ppm)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27.90
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13.92
n-Bu
N–Ph
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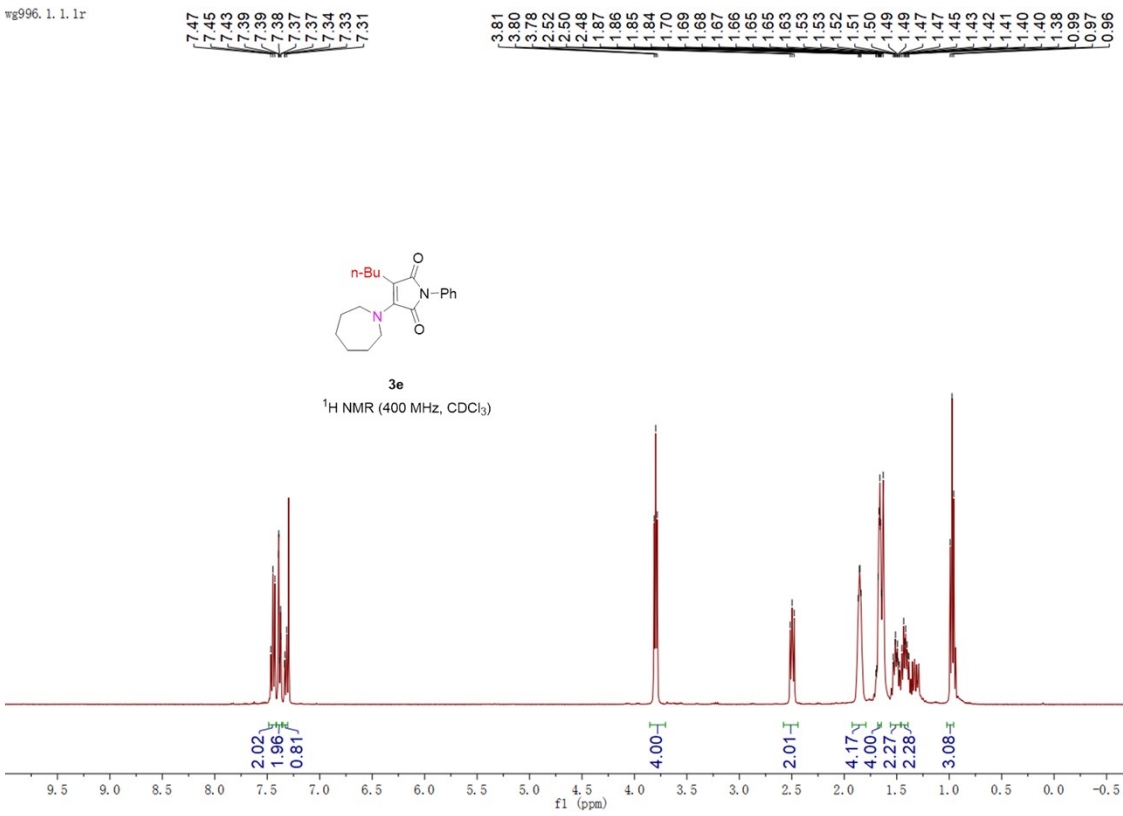
3d
13C NMR (100 MHz, CDCl3)
f1 (ppm)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3.80
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3e
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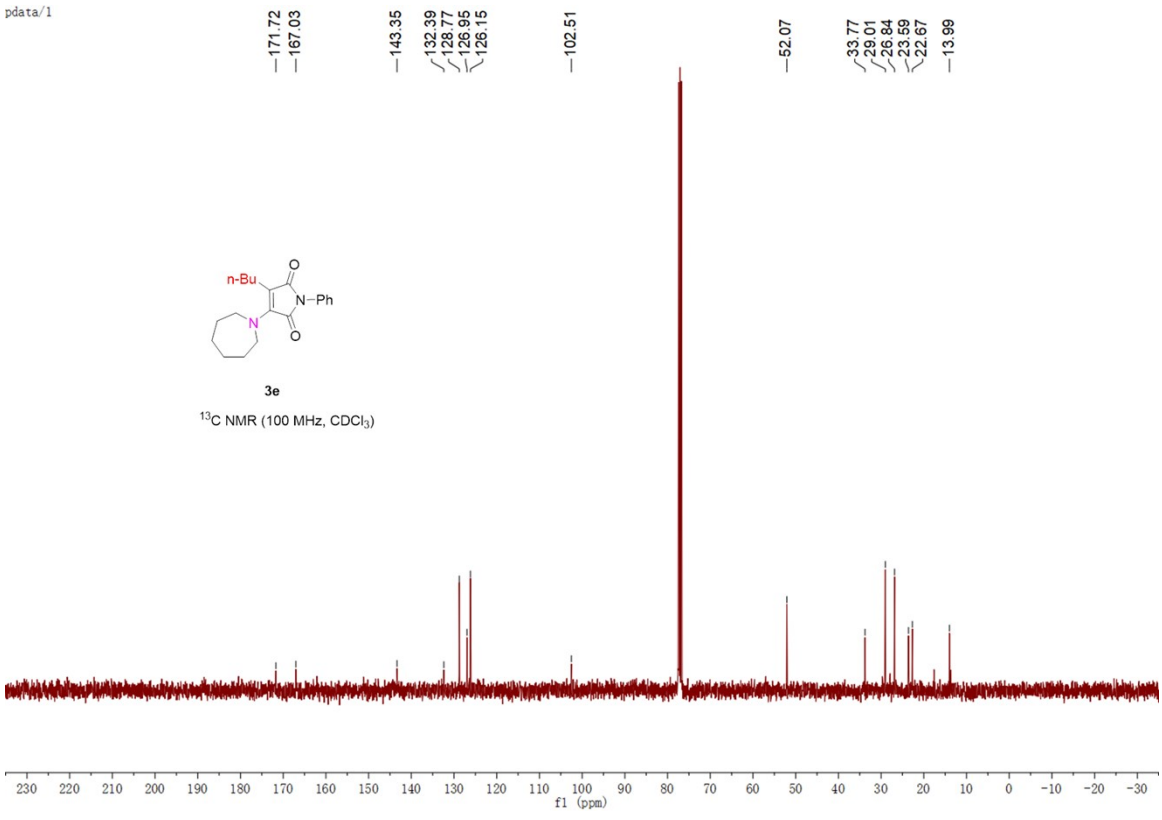
f1 (ppm)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3e
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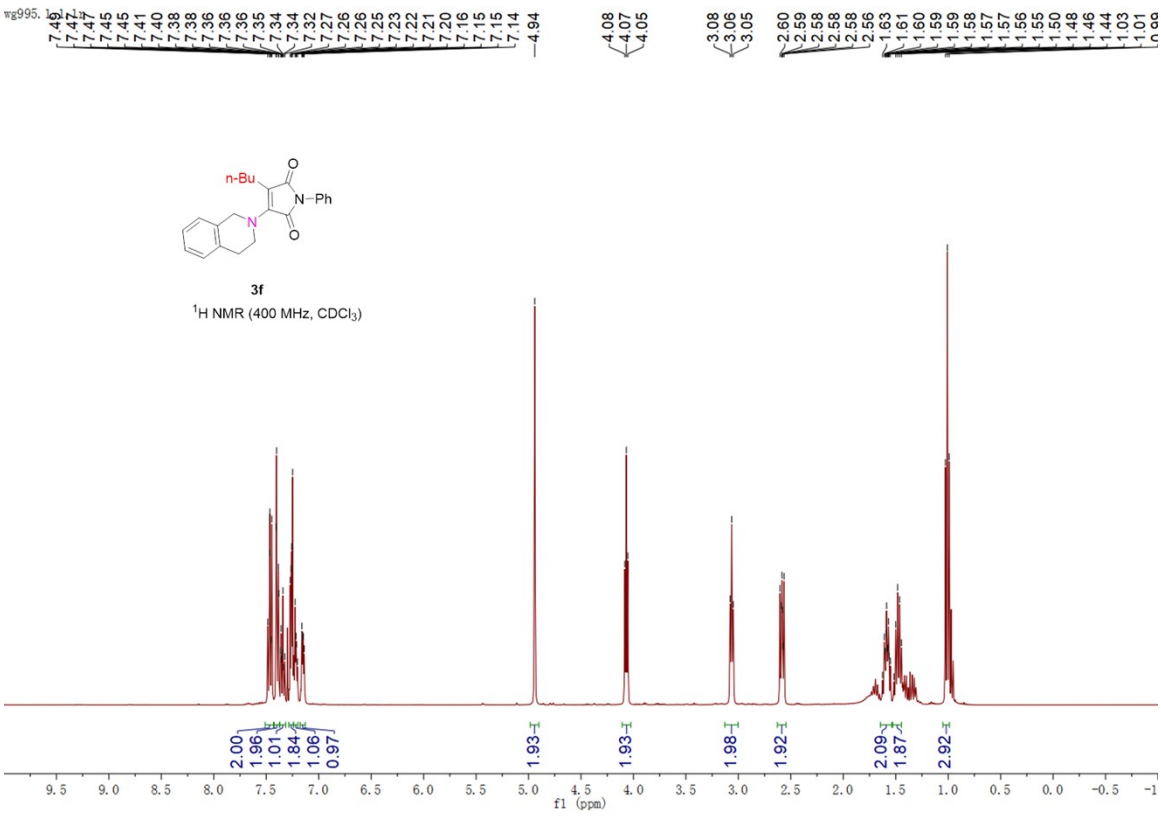
f1 (ppm)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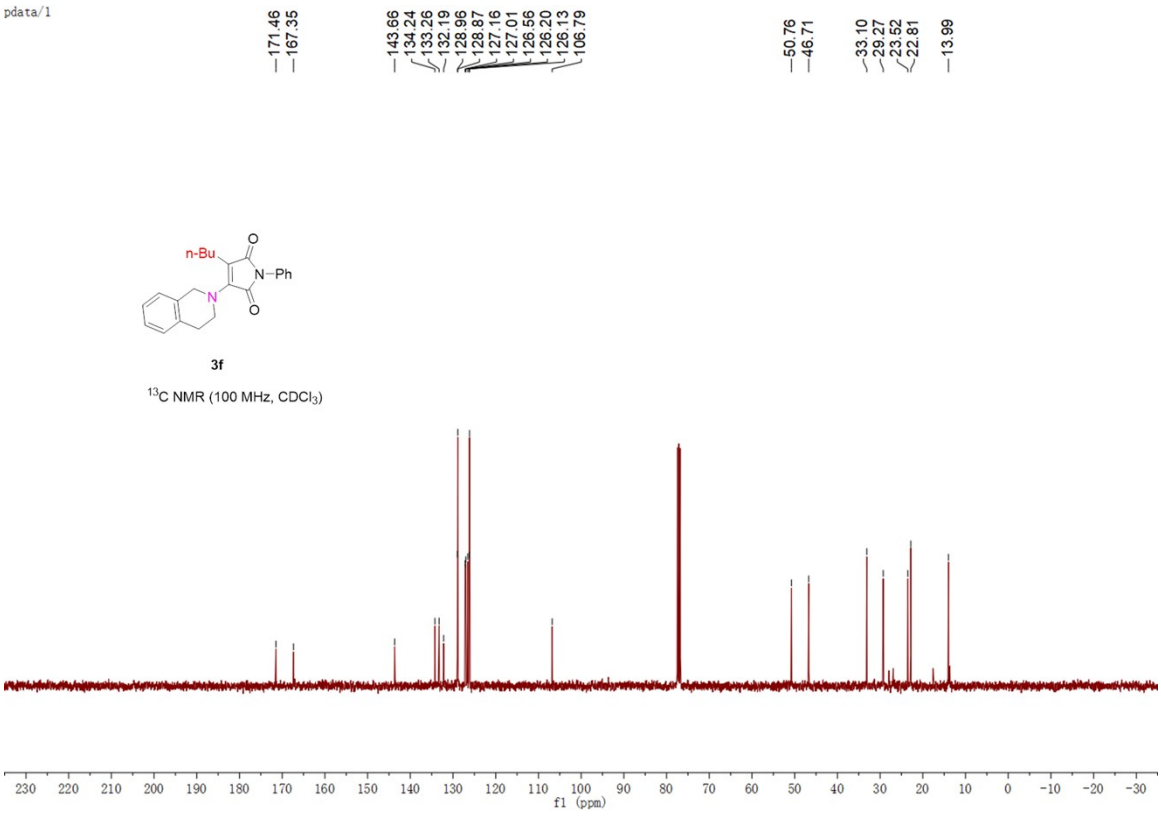
f1 (ppm)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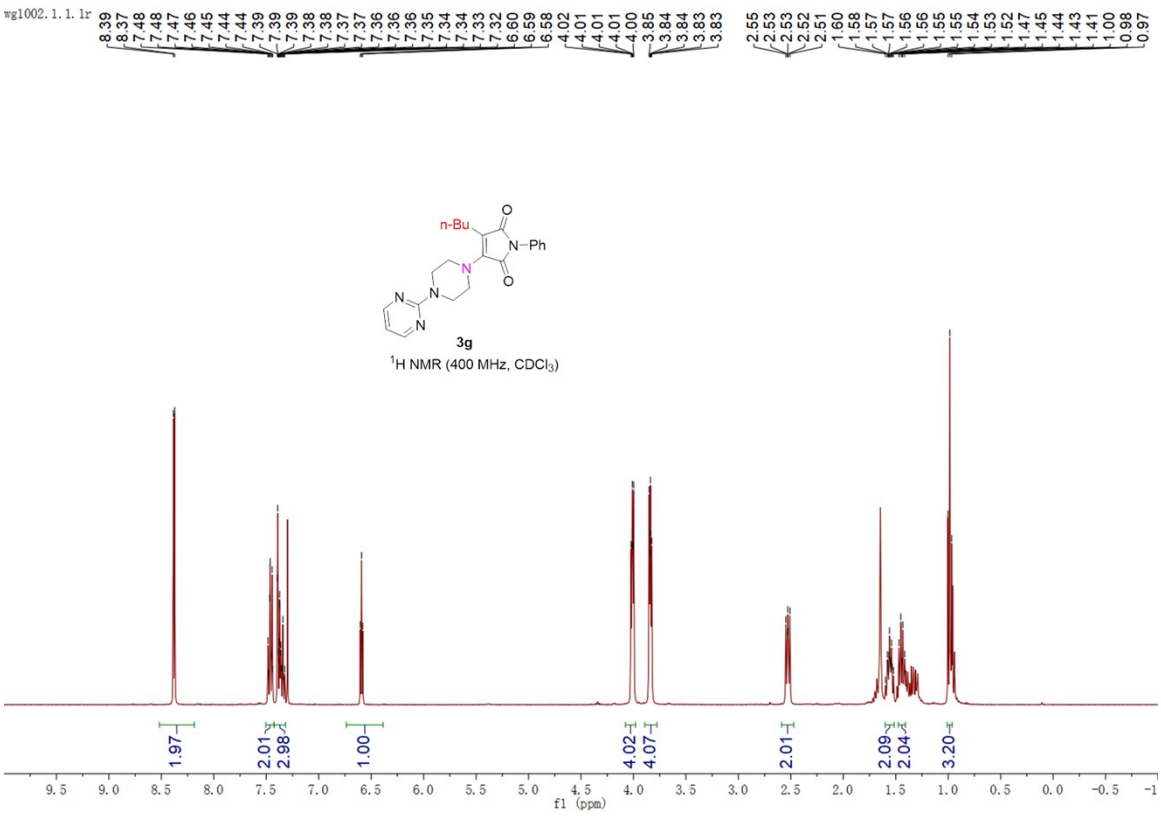
f1 (ppm)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n-Bu
N–Ph
3g
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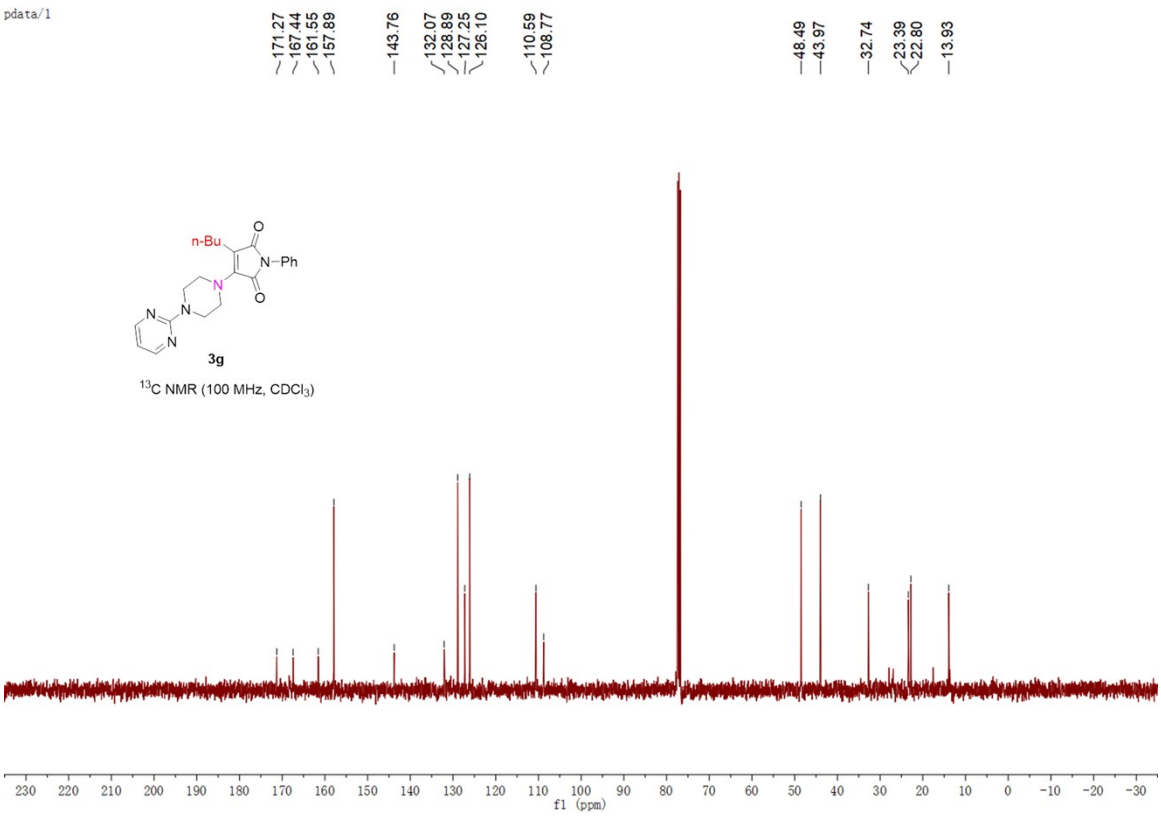
f1 (ppm)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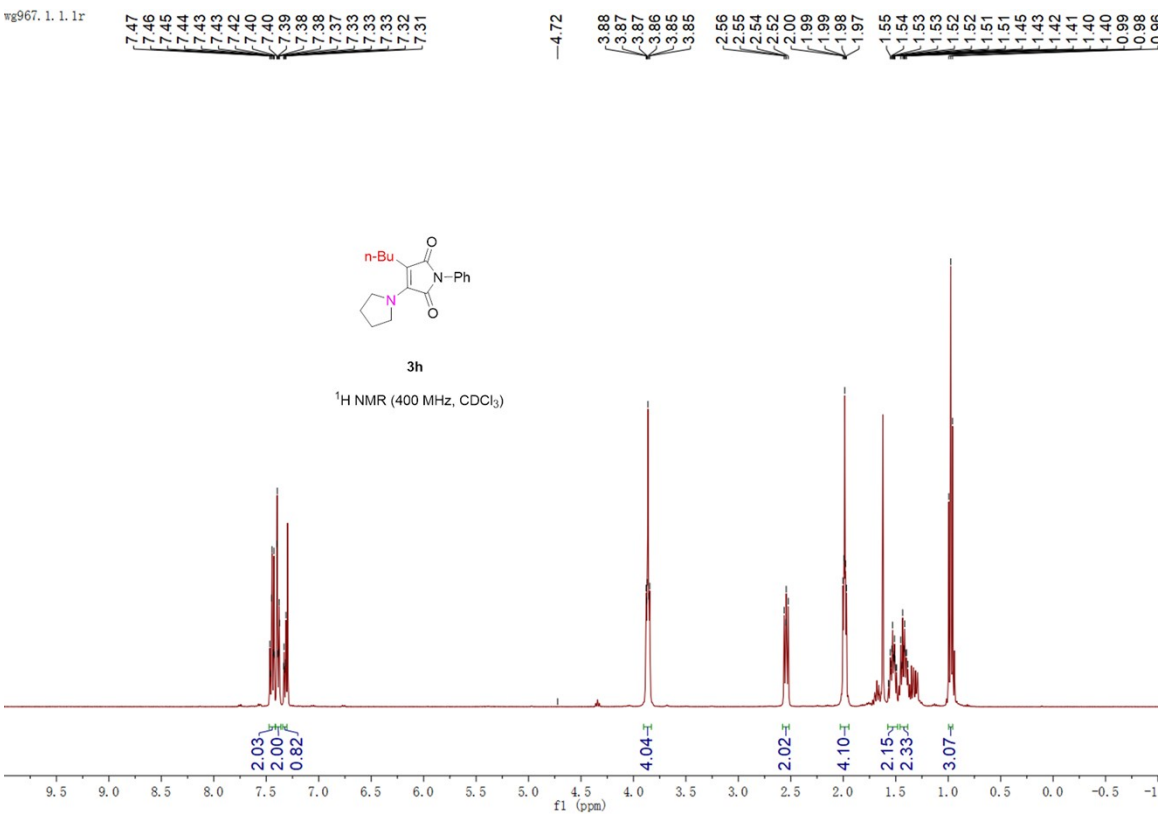
n-Bu
N–Ph
3g
13C NMR (100 MHz, CDCl3)
f1 (ppm)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2.52
2.00
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1.53
1.53
1.52
1.52
1.51
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1.42
1.41
1.40
1.40
0.99
0.98
n-Bu
N
Ph
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O
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3h
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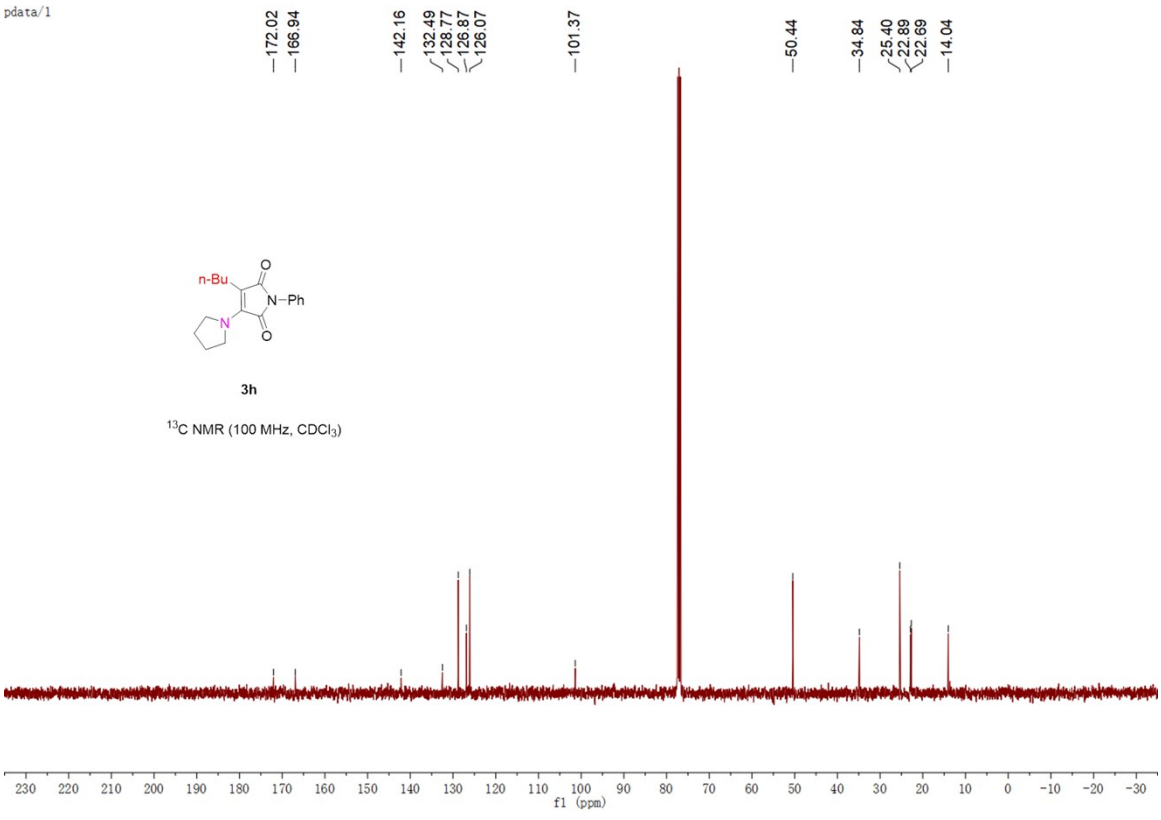
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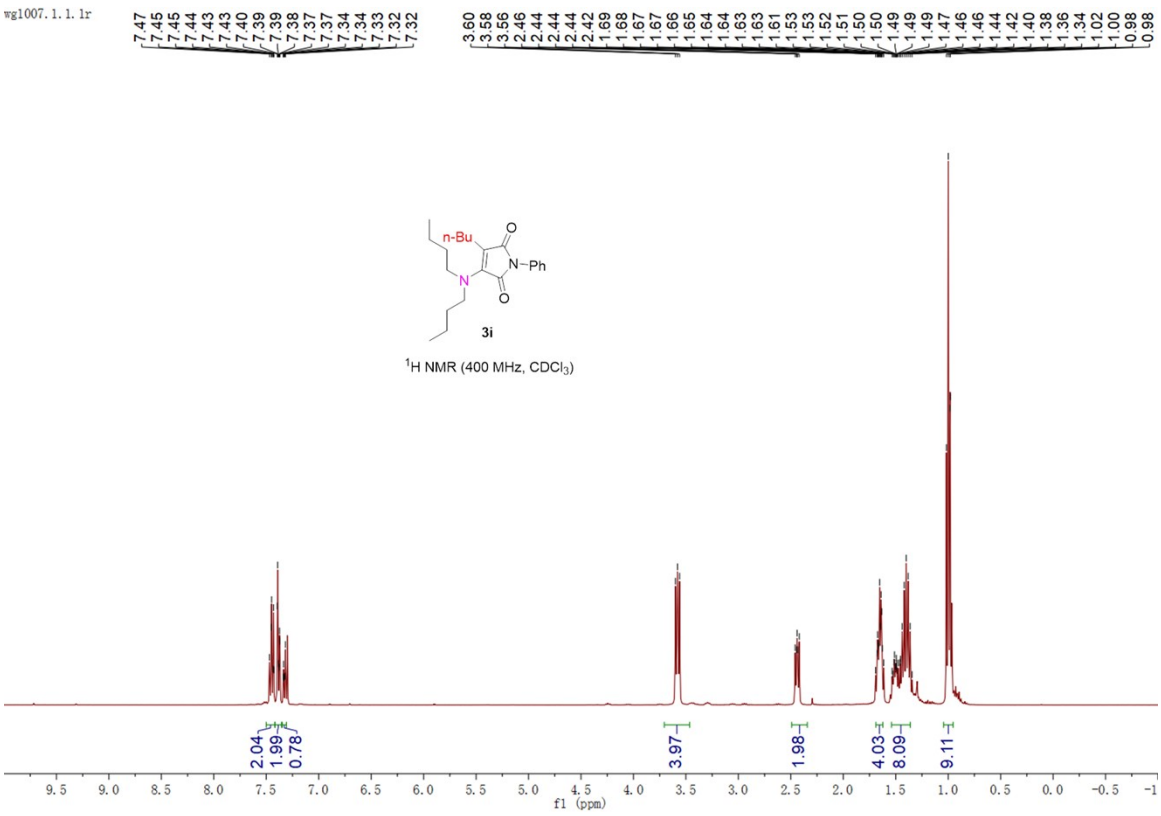
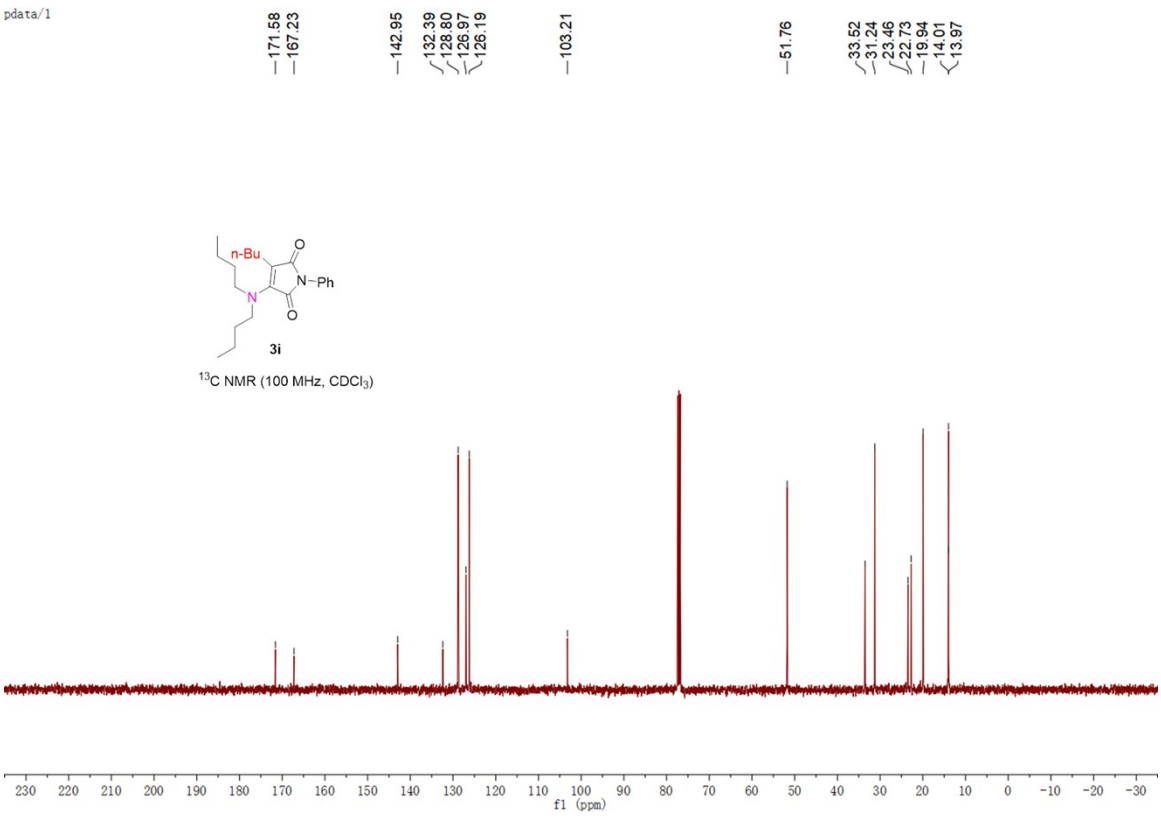
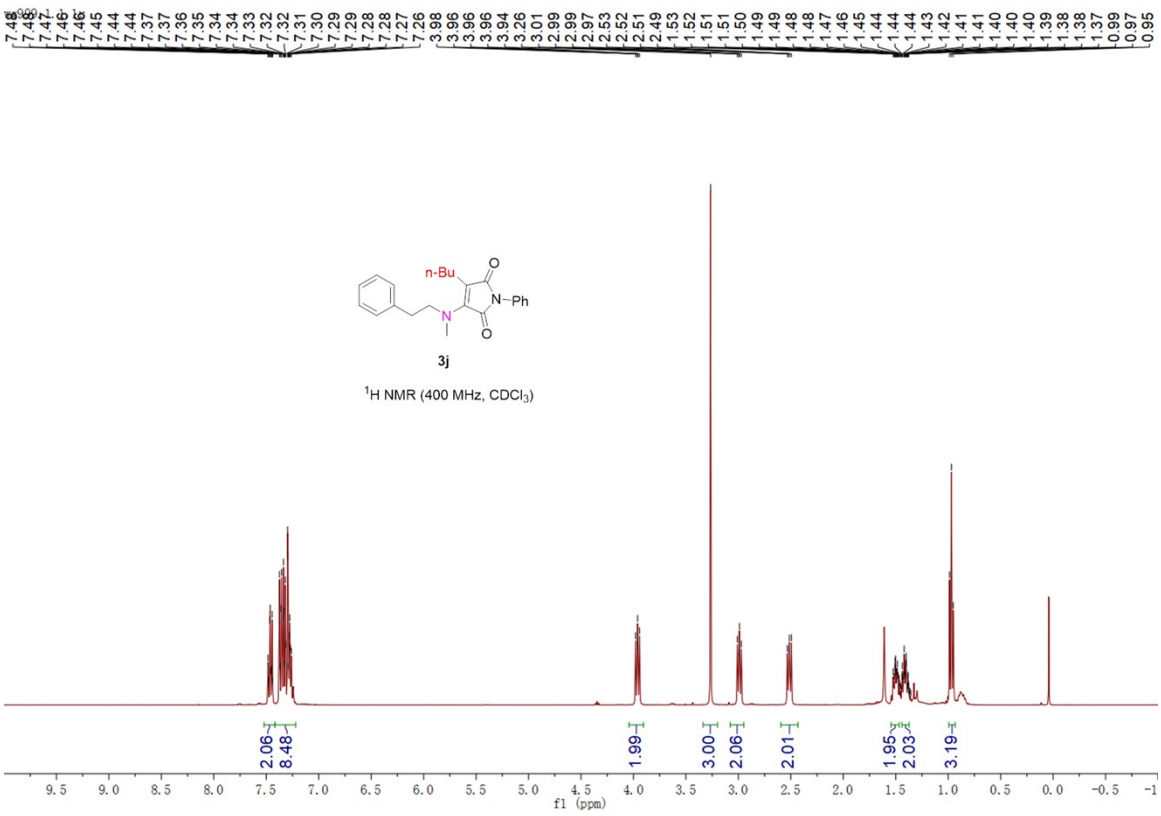
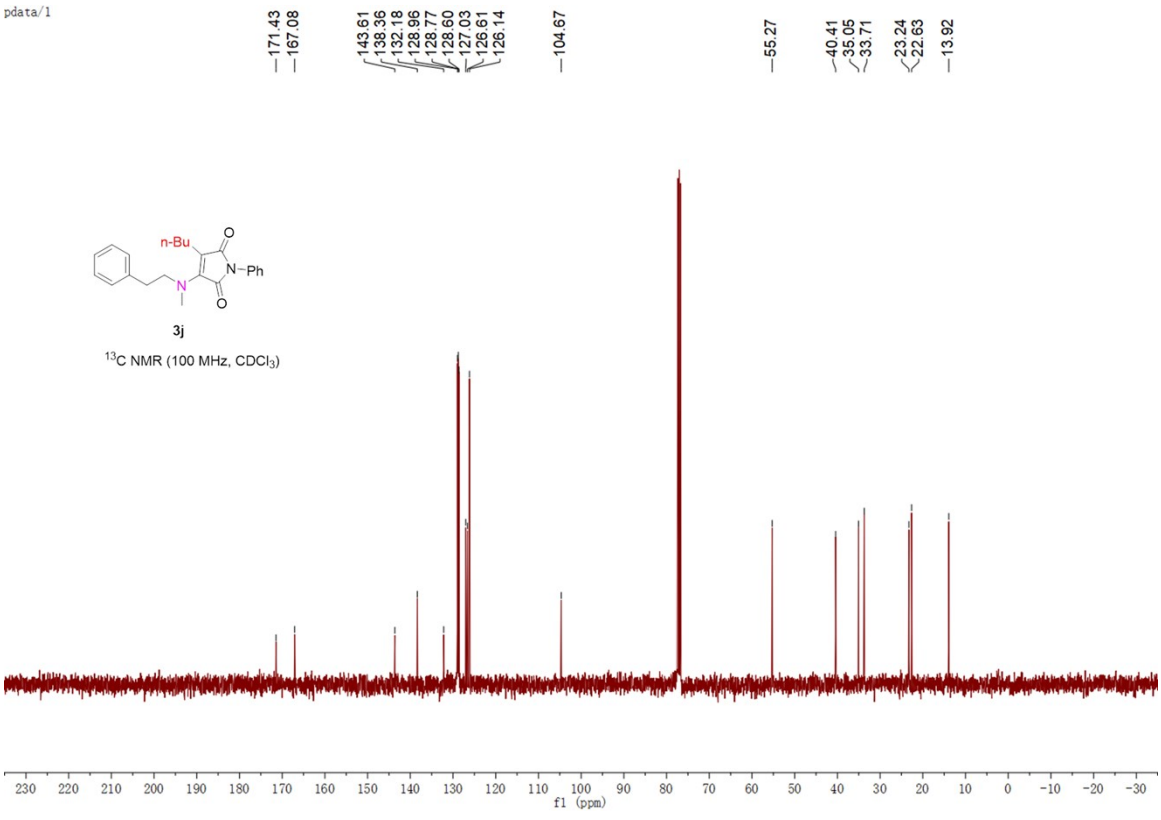
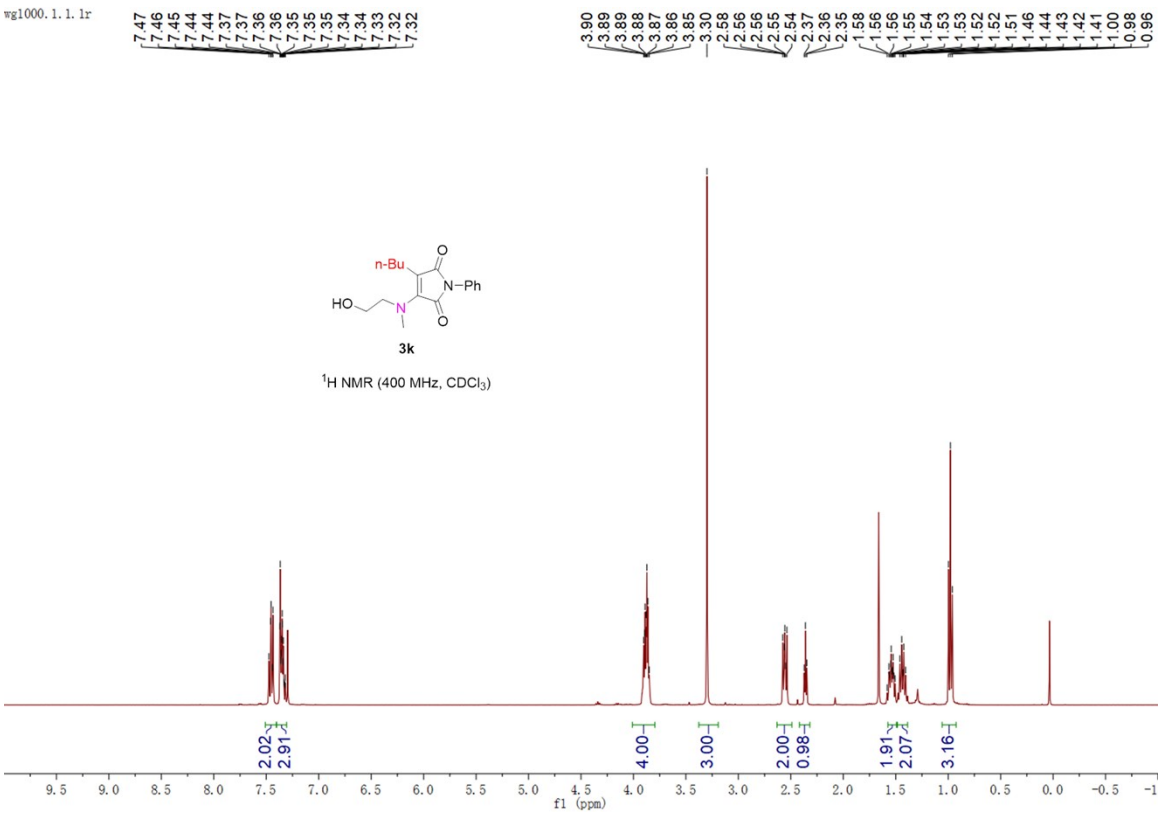
14.04
n-Bu
N
Ph
O
O
N
3h
13C NMR (100 MHz, CDCl3)
f1 (ppm)

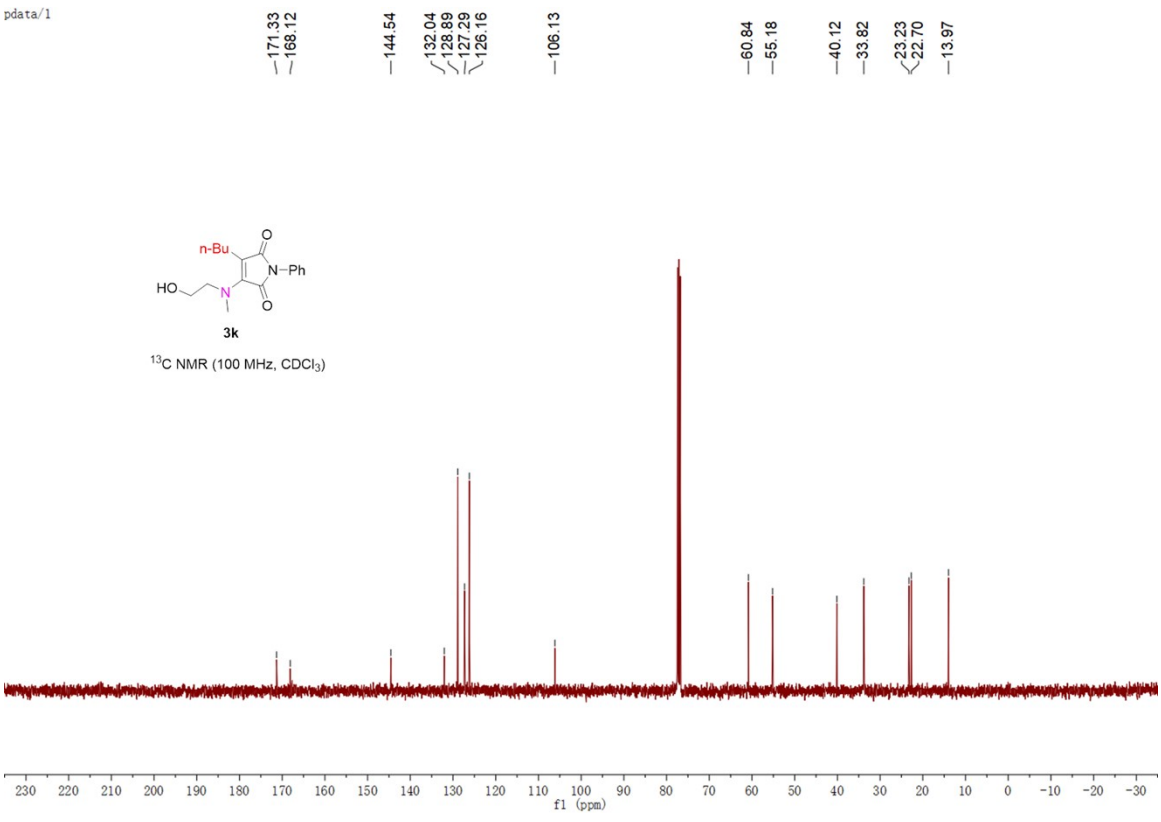

wg1007.1.1.1r
7.47 7.45 7.45 7.44 7.43 7.43 7.40 7.39 7.39 7.38 7.37 7.37 7.34 7.34 7.33 7.32 7.32
3.60 3.58 3.56 2.46 2.44 2.44 2.42 1.69 1.68 1.67 1.67 1.66 1.65 1.64 1.64 1.63 1.63 1.61 1.53 1.53 1.52 1.51 1.50 1.50 1.49 1.49 1.49 1.47 1.46 1.46 1.44 1.42 1.40 1.38 1.36 1.34 1.02 1.00 0.98 0.98
n-Bu
N
N–Ph
O
O
3i
^{1}H NMR (400 MHz, CDCl$_3$)
2.04 1.99 0.78 3.97 1.98 4.03 8.09 9.11
f1 (ppm)

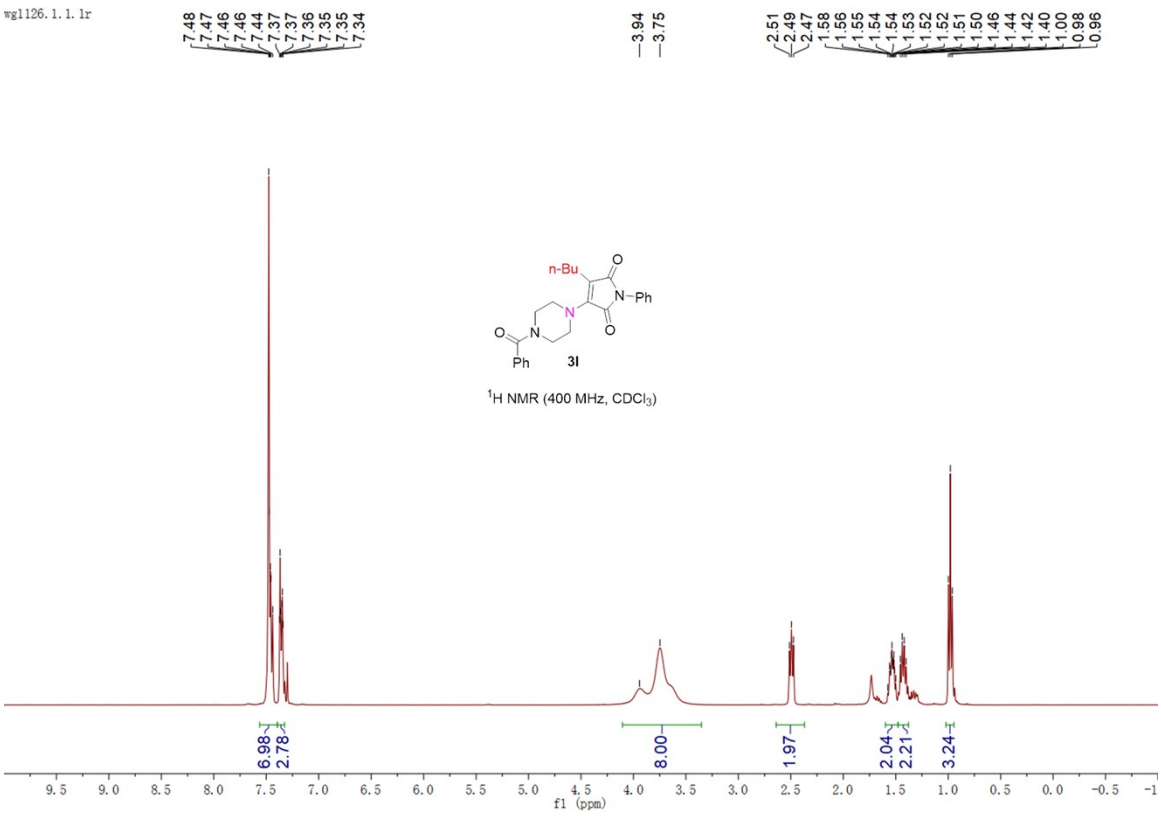

pdata/1
171.58 167.23 142.95 132.39 128.80 126.97 126.19 103.21 51.76 33.52 31.24 23.46 22.73 19.94 14.01 13.97
n-Bu
N
N–Ph
O
O
3i
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)


n-Bu
N
N–Ph
O
O
3j
^{1}H NMR (400 MHz, CDCl$_3$)
2.06
8.48
1.99
3.00
2.06
2.01
1.95
2.03
3.19
f1 (ppm)

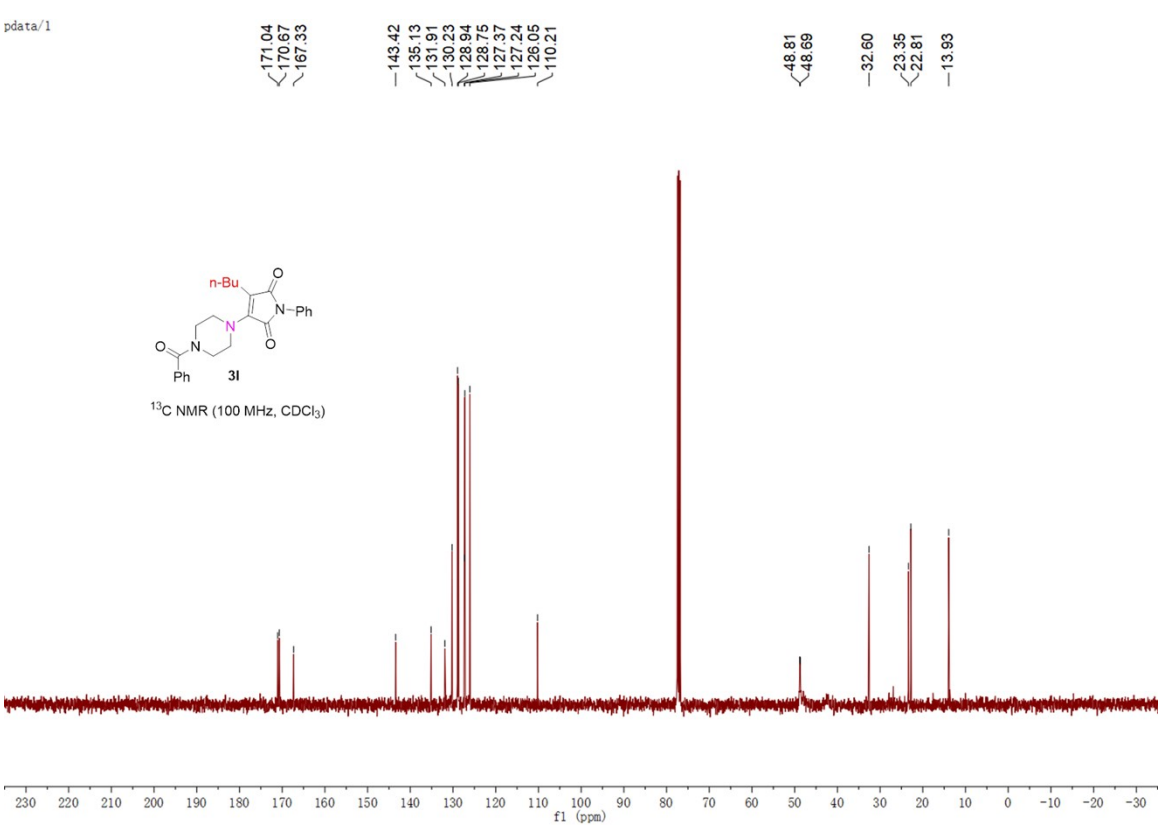

pdata/1
171.43
167.08
143.61
138.36
132.18
128.96
128.77
128.60
127.03
126.61
126.14
104.67
55.27
40.41
35.05
33.71
23.24
22.63
13.92
n-Bu
N
N–Ph
O
O
3j
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)


wg1000.1.1.1r
7.47 7.46 7.45 7.44 7.44 7.37 7.37 7.36 7.36 7.35 7.35 7.35 7.34 7.34 7.33 7.32 7.32
3.90 3.89 3.89 3.88 3.87 3.86 3.85 3.30 2.58 2.56 2.56 2.55 2.54 2.37 2.36 2.35 1.58 1.56 1.56 1.55 1.54 1.53 1.53 1.52 1.52 1.51 1.46 1.44 1.43 1.42 1.41 1.00 0.98 0.96
n-Bu
N–Ph
HO
N
O
O
3k
1H NMR (400 MHz, CDCl3)
2.02 2.91 4.00 3.00 2.00 0.98 1.91 2.07 3.16
f1 (ppm)

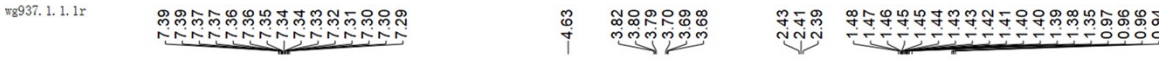

pdata/1
171.33 168.12 144.54 132.04 128.89 127.29 126.16 106.13 60.84 55.18 40.12 33.82 23.23 22.70 13.97
n-Bu
N–Ph
HO
N
O
O
3k
13C NMR (100 MHz, CDCl3)
f1 (ppm)

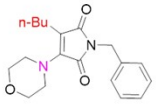


^{1}H NMR (400 MHz, $CDCl_3$)



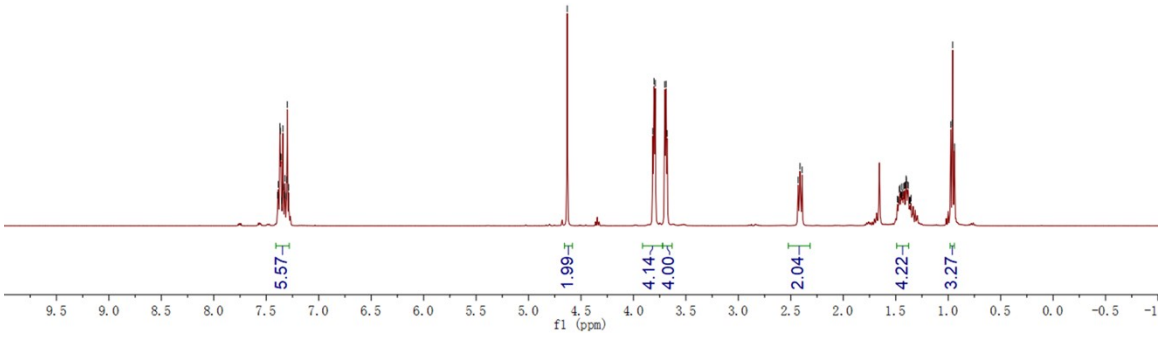
^{13}C NMR (100 MHz, $CDCl_3$)

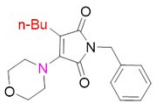




4a

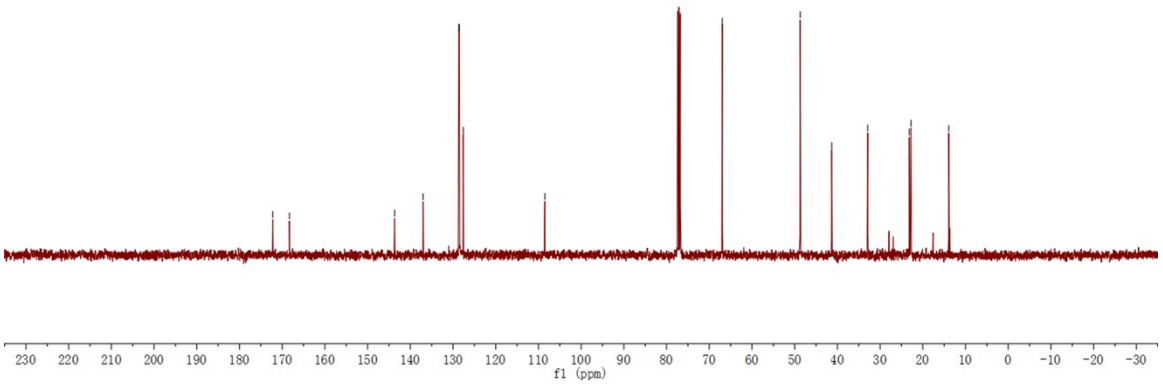
^{1}H NMR (400 MHz, $CDCl_3$)





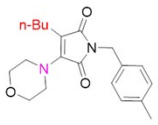
4a

^{13}C NMR (100 MHz, $CDCl_3$)



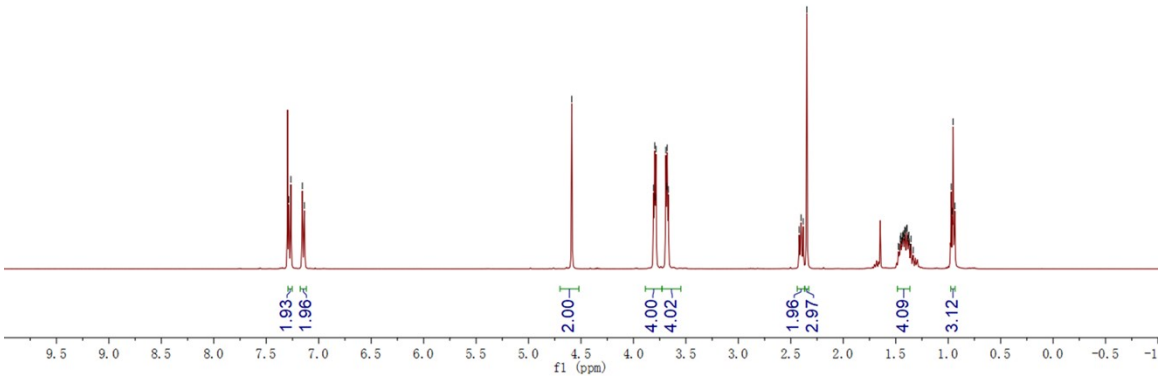
wg959.1.1.1r

7.29 7.27 7.16 7.14 4.59 3.81 3.80 3.79 3.78 3.69 3.68 3.68 3.67 2.42 2.40 2.38 2.35 1.46 1.45 1.43 1.43 1.43 1.42 1.41 1.41 1.40 1.39 1.39 1.38 1.37 1.35 0.97 0.96 0.95



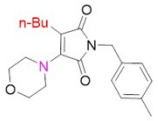
4b

^{1}H NMR (400 MHz, $CDCl_3$)



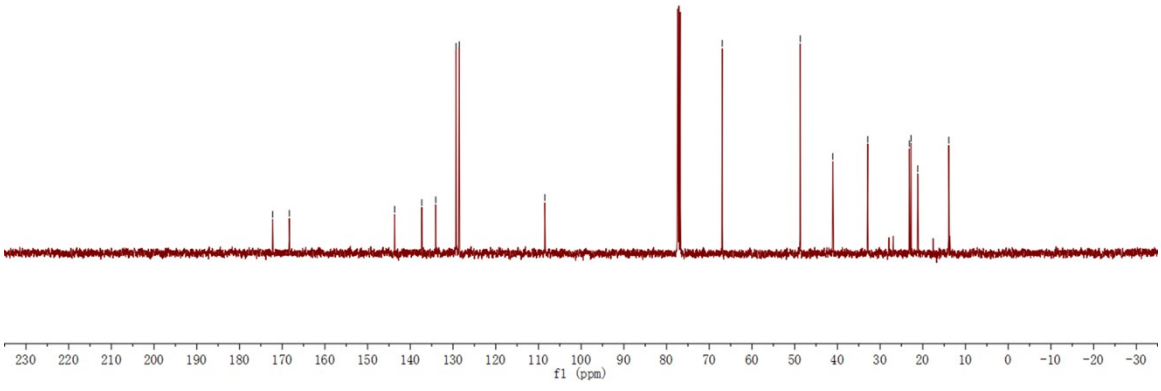
pdata/1

172.24 168.32 143.67 137.32 134.05 129.29 128.54 108.51 66.96 48.70 41.07 32.88 23.16 22.75 21.19 13.92

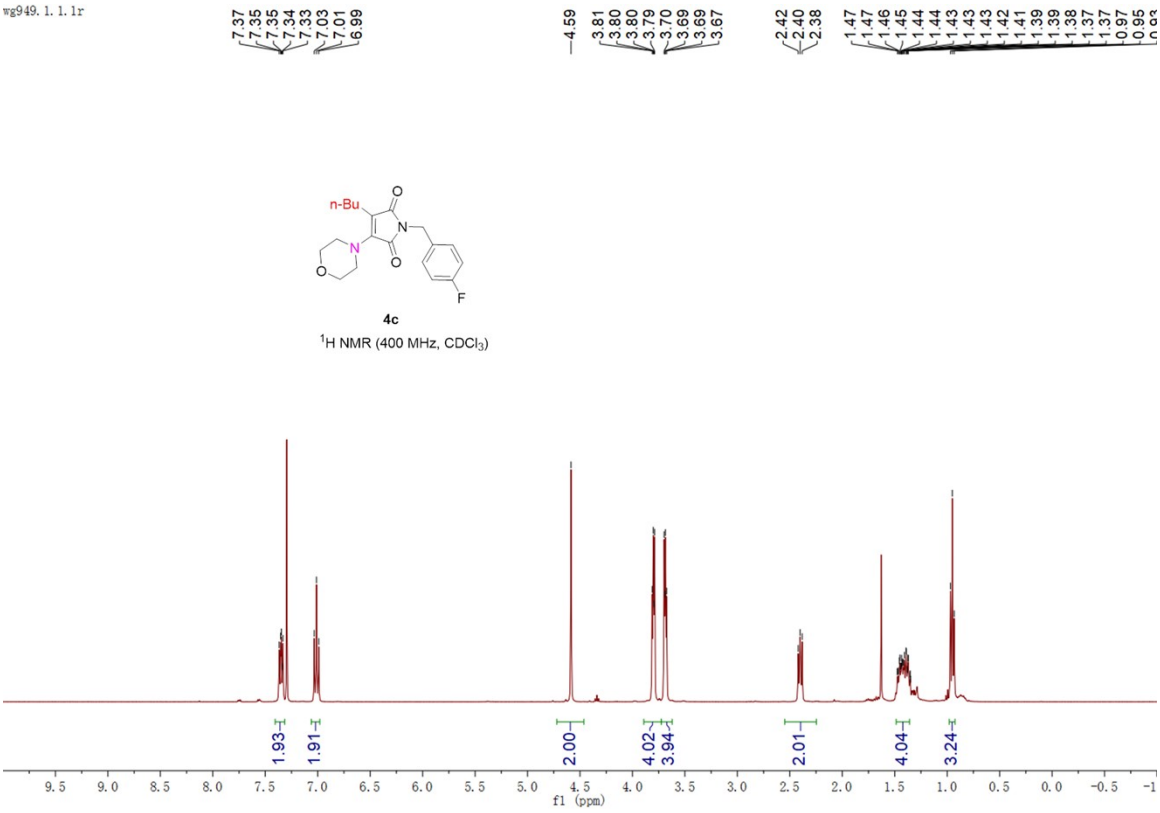


4b

^{13}C NMR (100 MHz, $CDCl_3$)



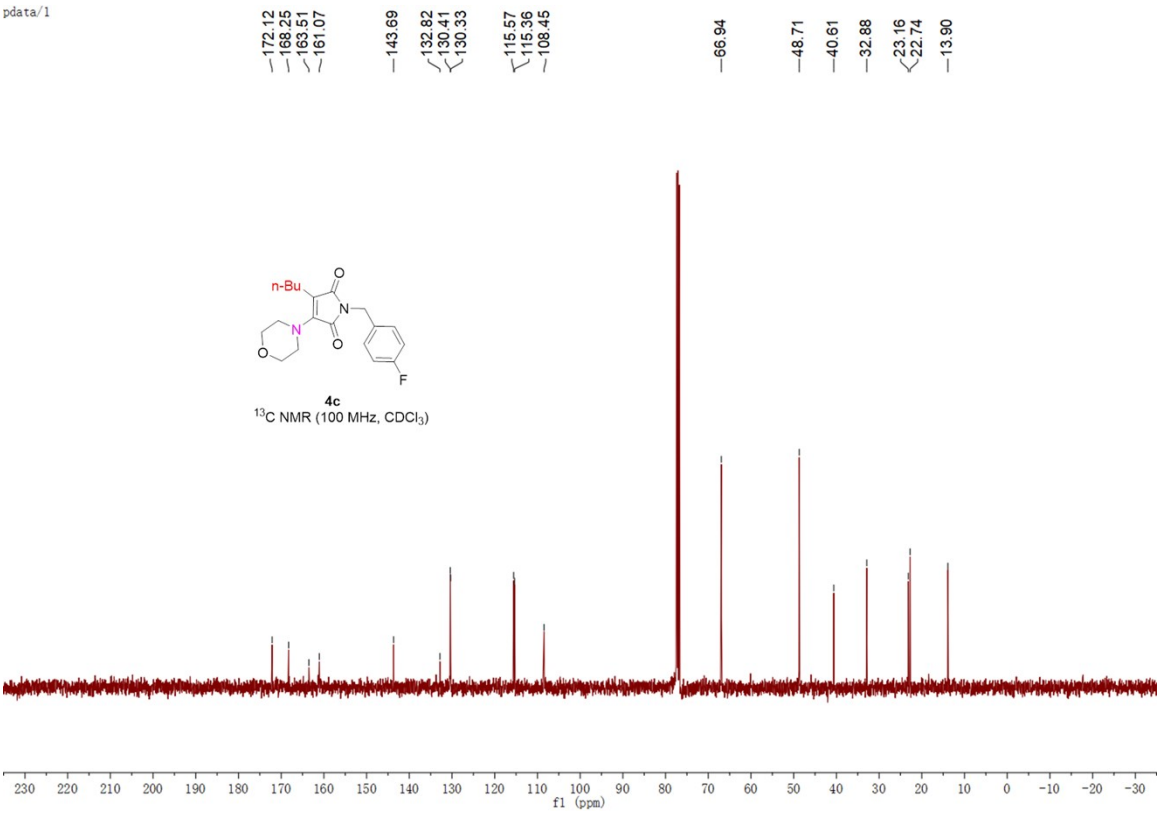
wg949.1.1.1r



4c

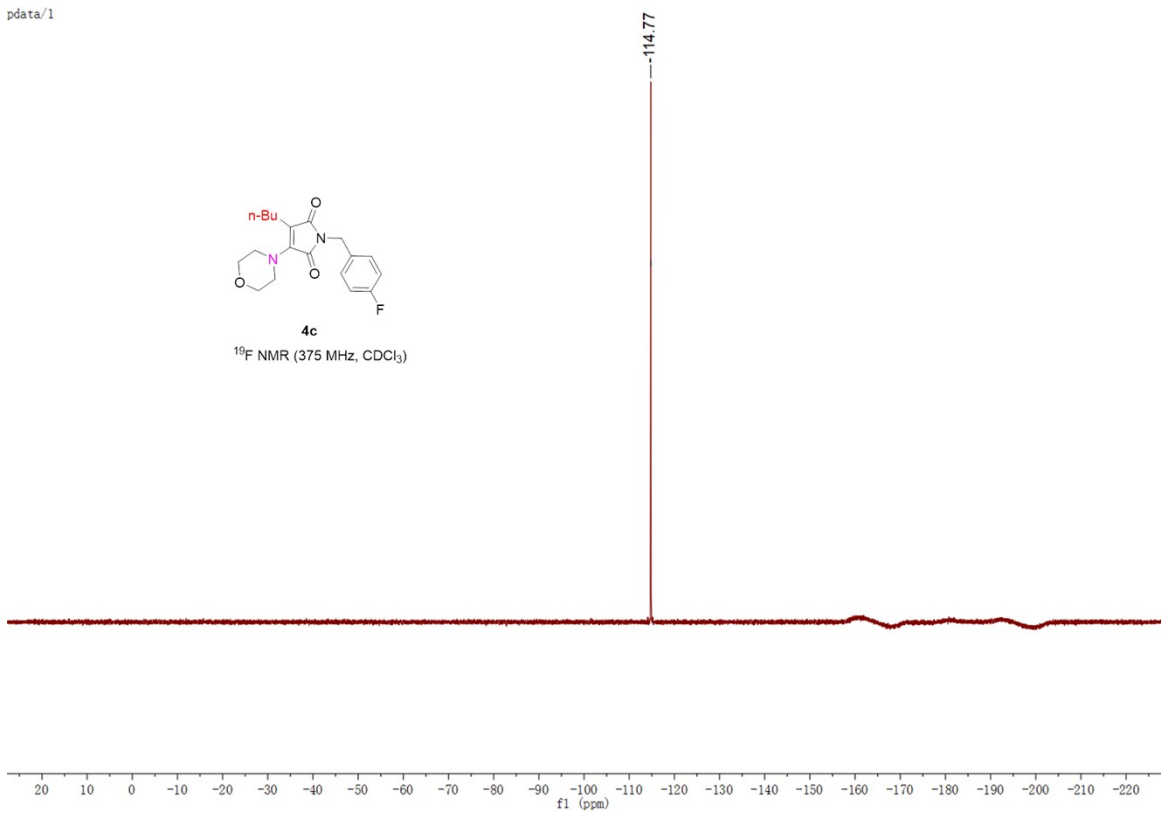
^{1}H NMR (400 MHz, $CDCl_3$)

pdata/1



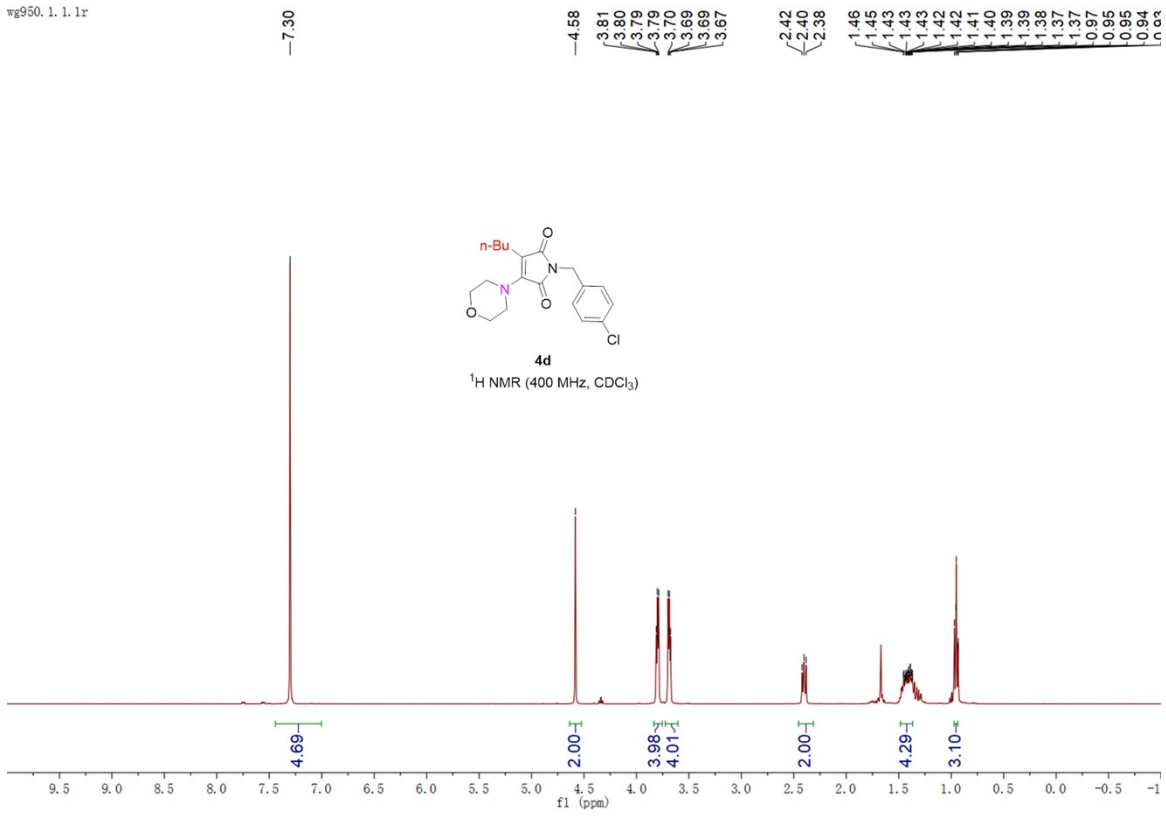
4c

^{13}C NMR (100 MHz, $CDCl_3$)



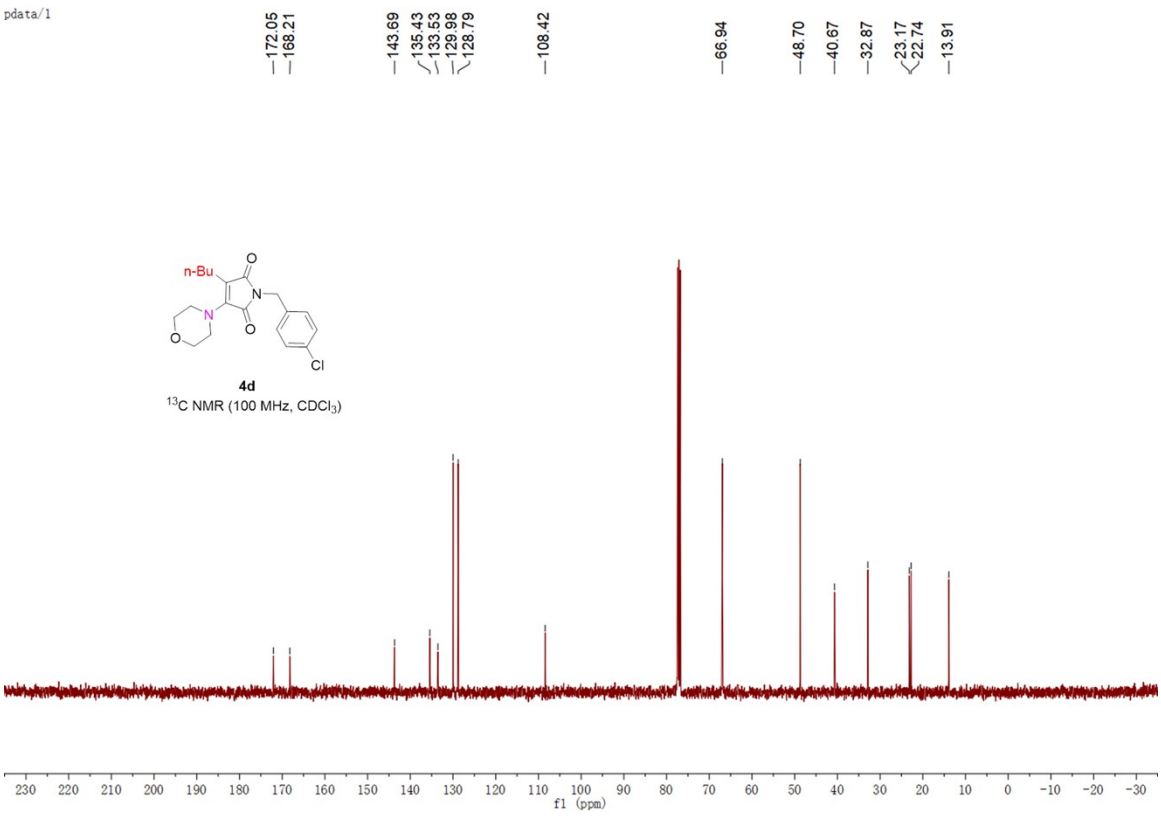
4c

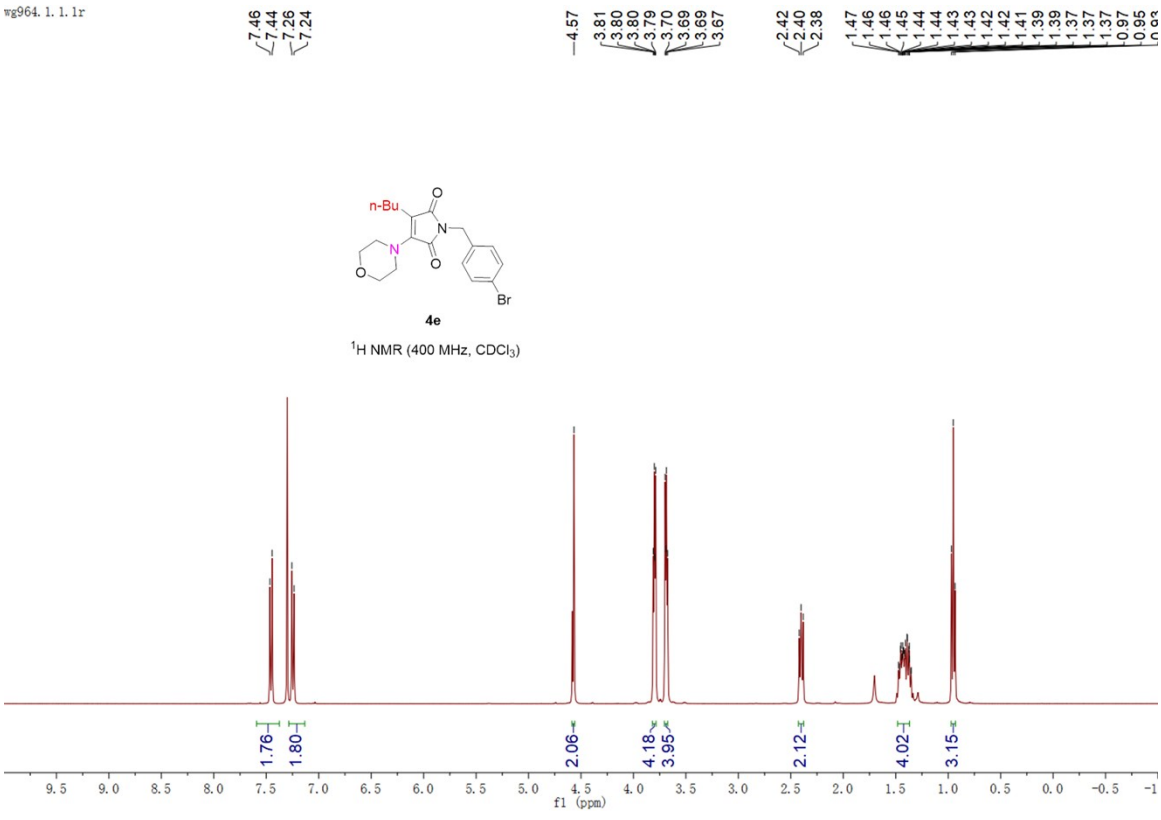
^{19}F NMR (375 MHz, $CDCl_3$)

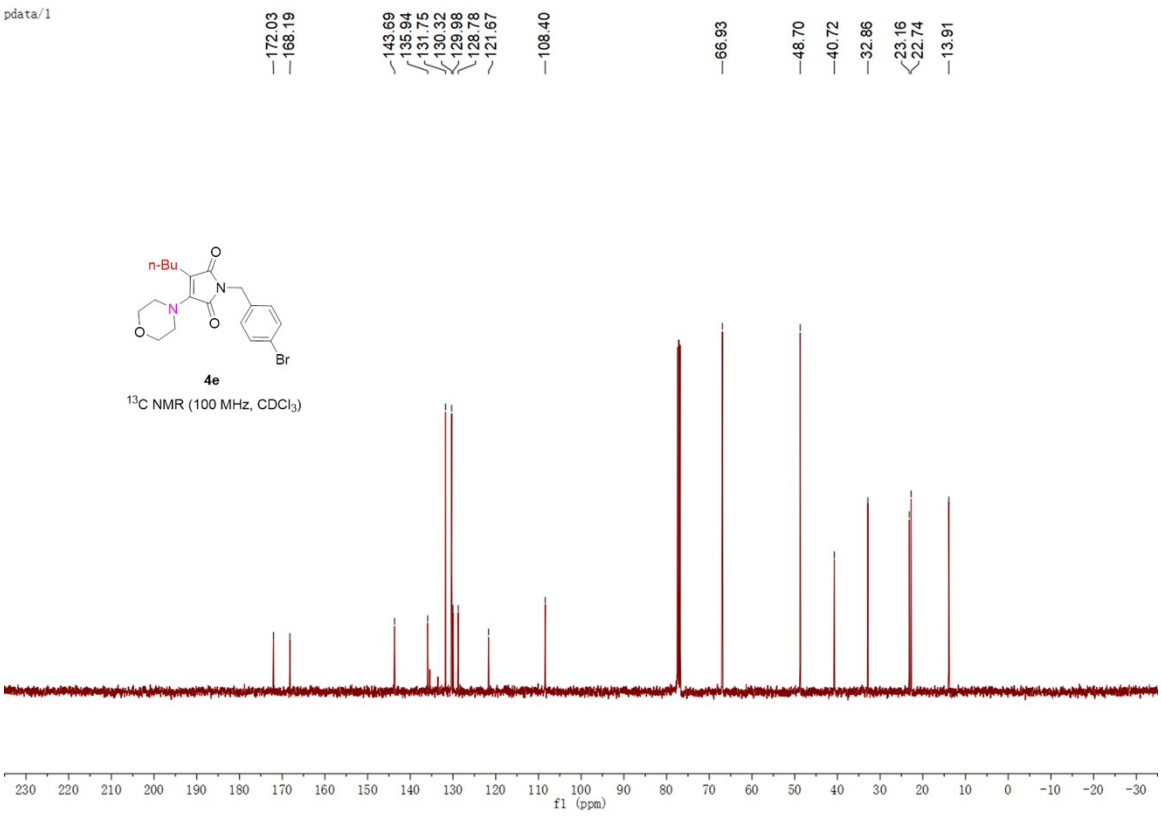


4d

^{1}H NMR (400 MHz, $CDCl_3$)

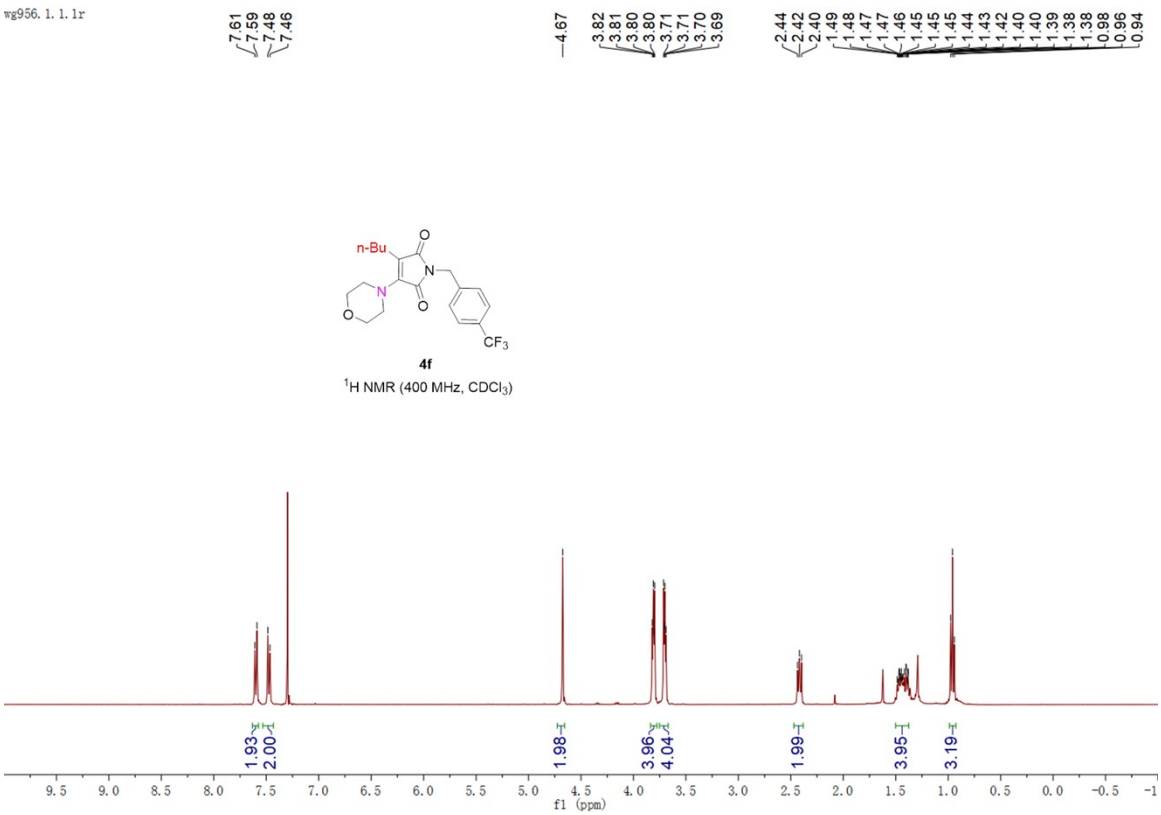

pdata/1
172.05
168.21
143.69
135.43
133.53
129.98
128.79
108.42
66.94
48.70
40.67
32.87
23.17
22.74
13.91
n-Bu
N
O
O
N
O
Cl
4d
13C NMR (100 MHz, CDCl3)
f1 (ppm)


wg964.1.1.1r
7.46
7.44
7.26
7.24
4.57
3.81
3.80
3.80
3.79
3.70
3.69
3.69
3.67
2.42
2.40
2.38
1.47
1.46
1.46
1.45
1.44
1.44
1.43
1.43
1.42
1.42
1.41
1.39
1.39
1.37
1.37
1.37
0.97
0.95
n-Bu
N
O
O
N
O
Br
4e
1H NMR (400 MHz, CDCl3)
1.76
1.80
2.06
4.18
3.95
2.12
4.02
3.15
f1 (ppm)



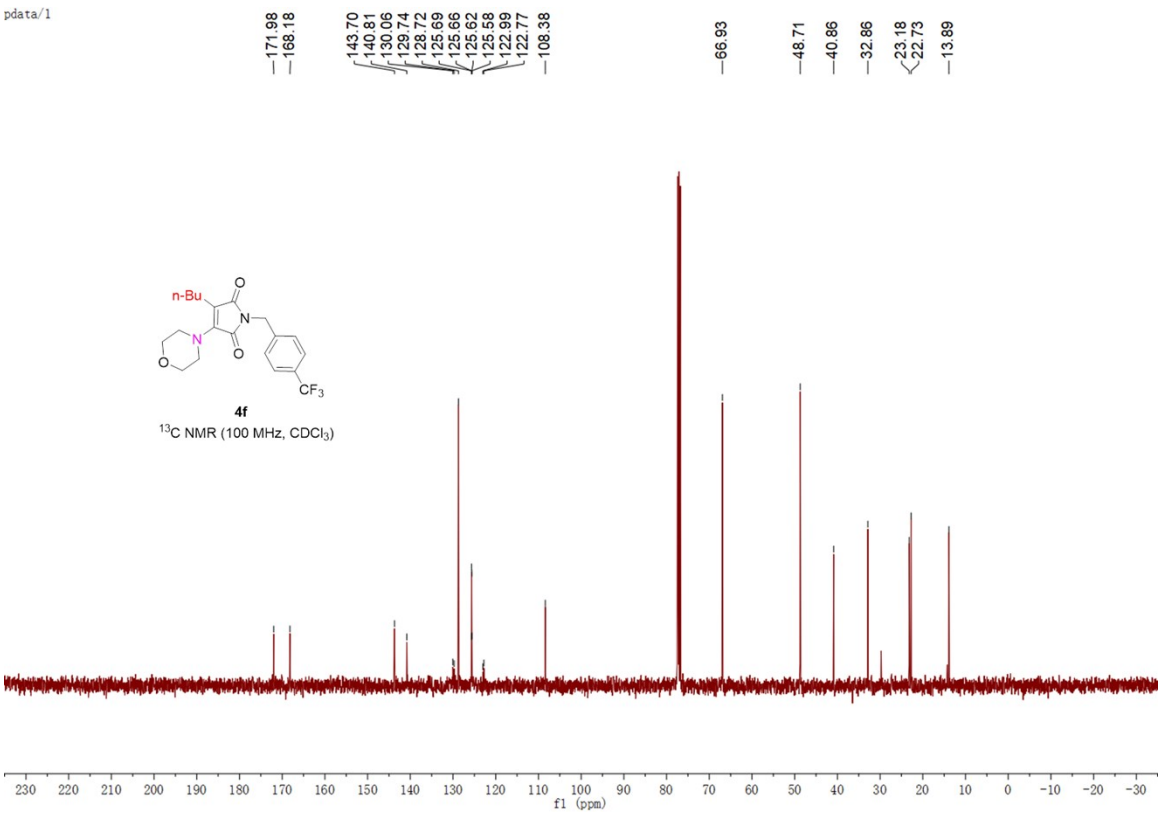
4e

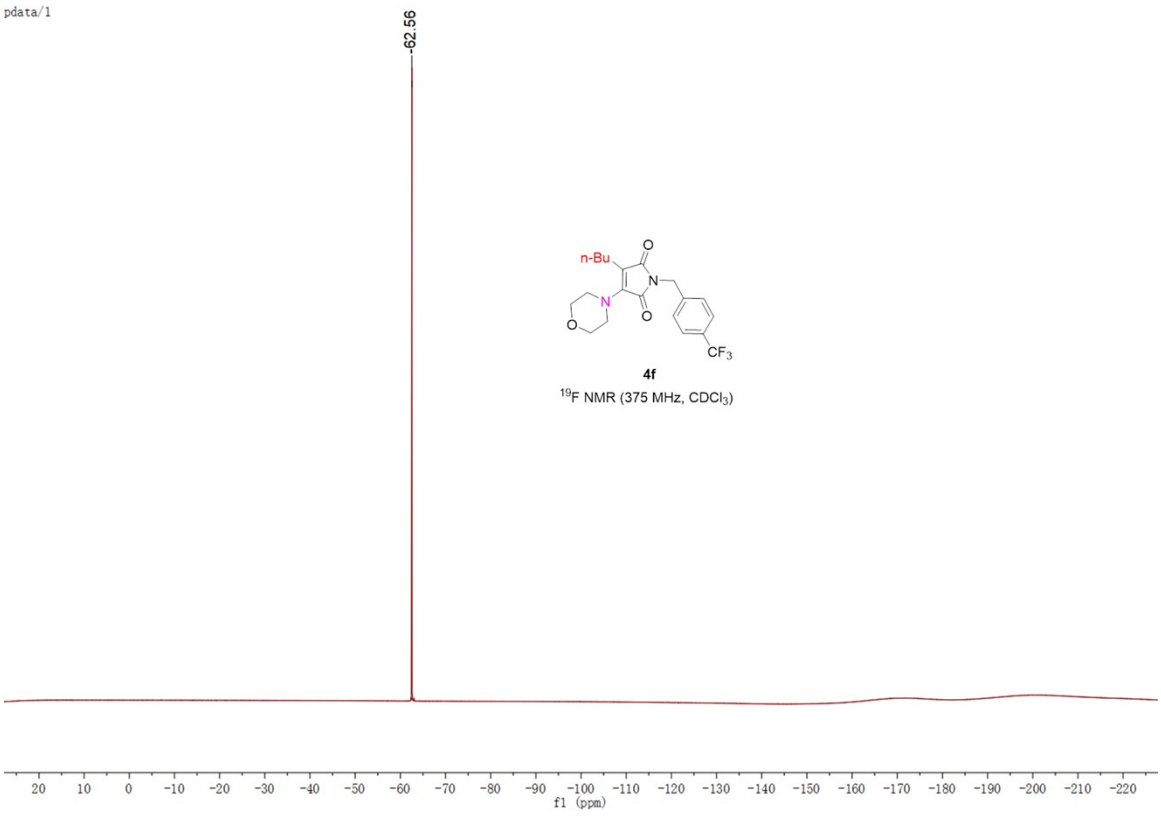
^{13}C NMR (100 MHz, $CDCl_3$)

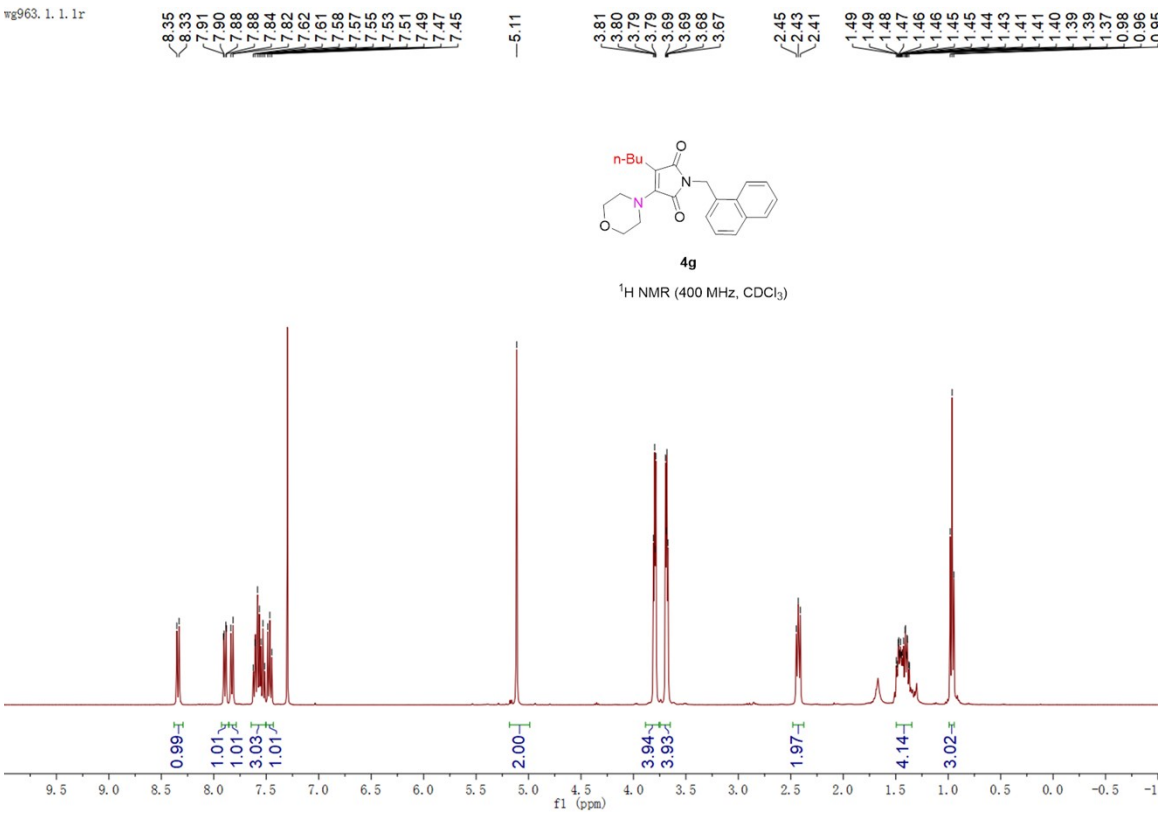


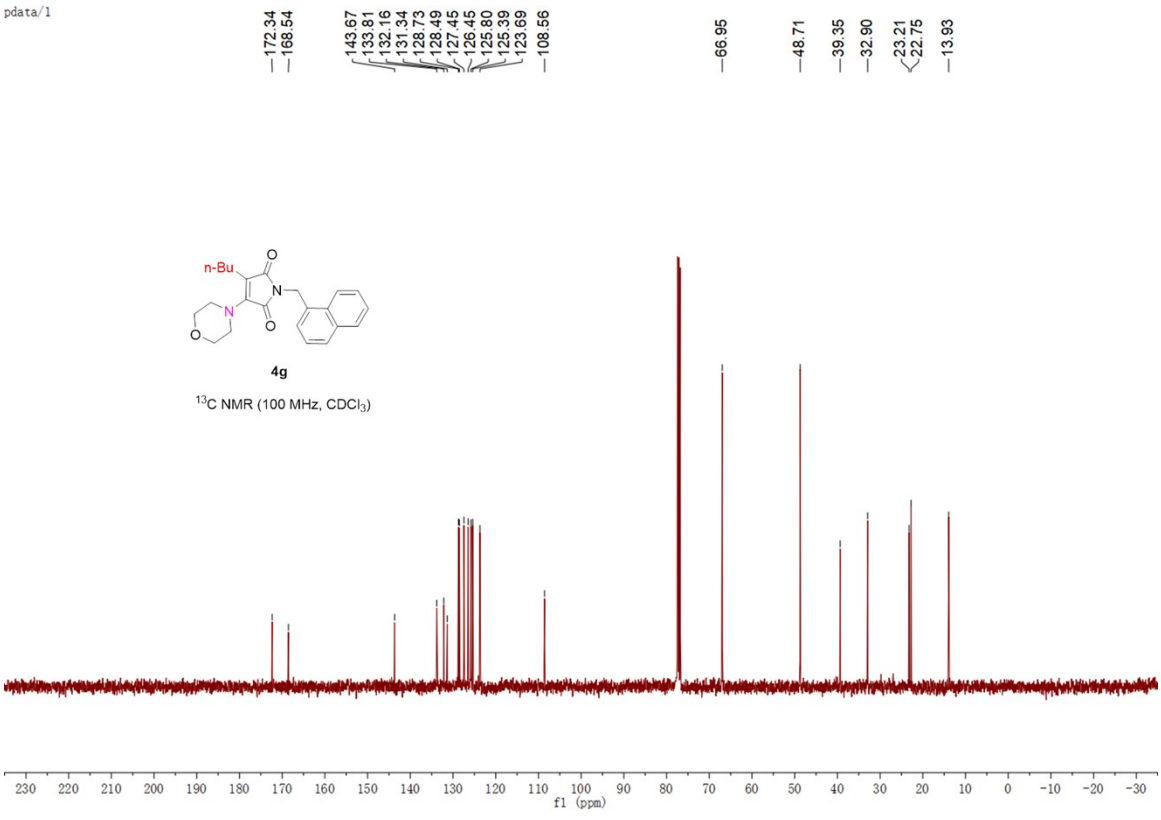
4f

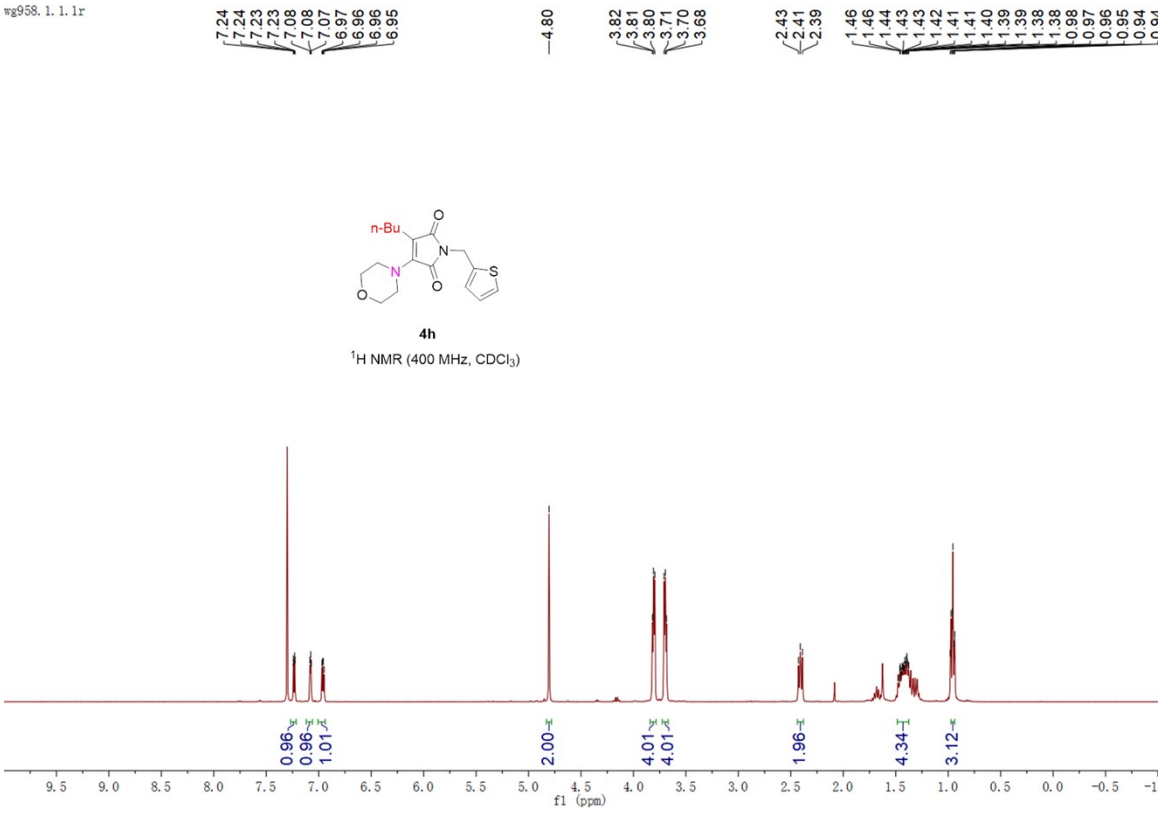
^{1}H NMR (400 MHz, $CDCl_3$)

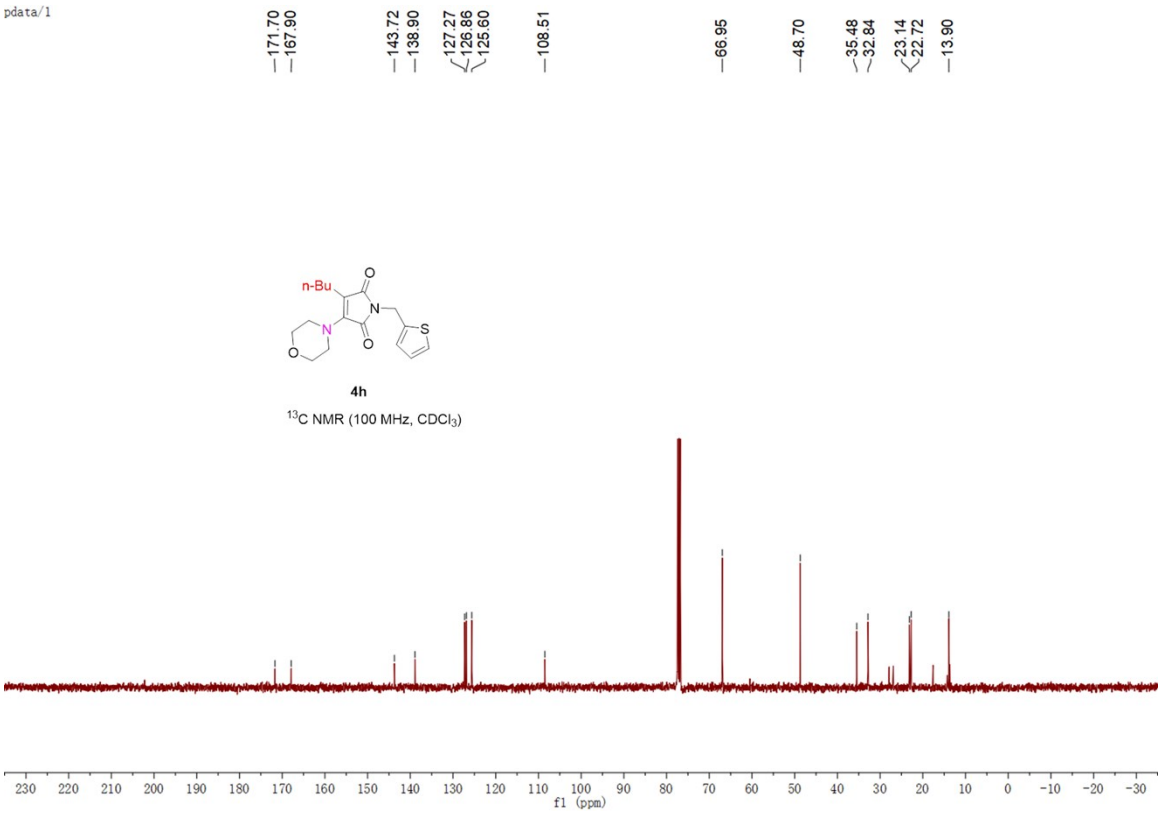

pdata/1
171.98
168.18
143.70
140.81
130.06
129.74
128.72
125.69
125.66
125.62
125.58
122.99
122.77
108.38
66.93
48.71
40.86
32.86
23.18
22.73
13.89
n-Bu
O
N
N
O
O
CF3
4f
13C NMR (100 MHz, CDCl3)
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30
f1 (ppm)

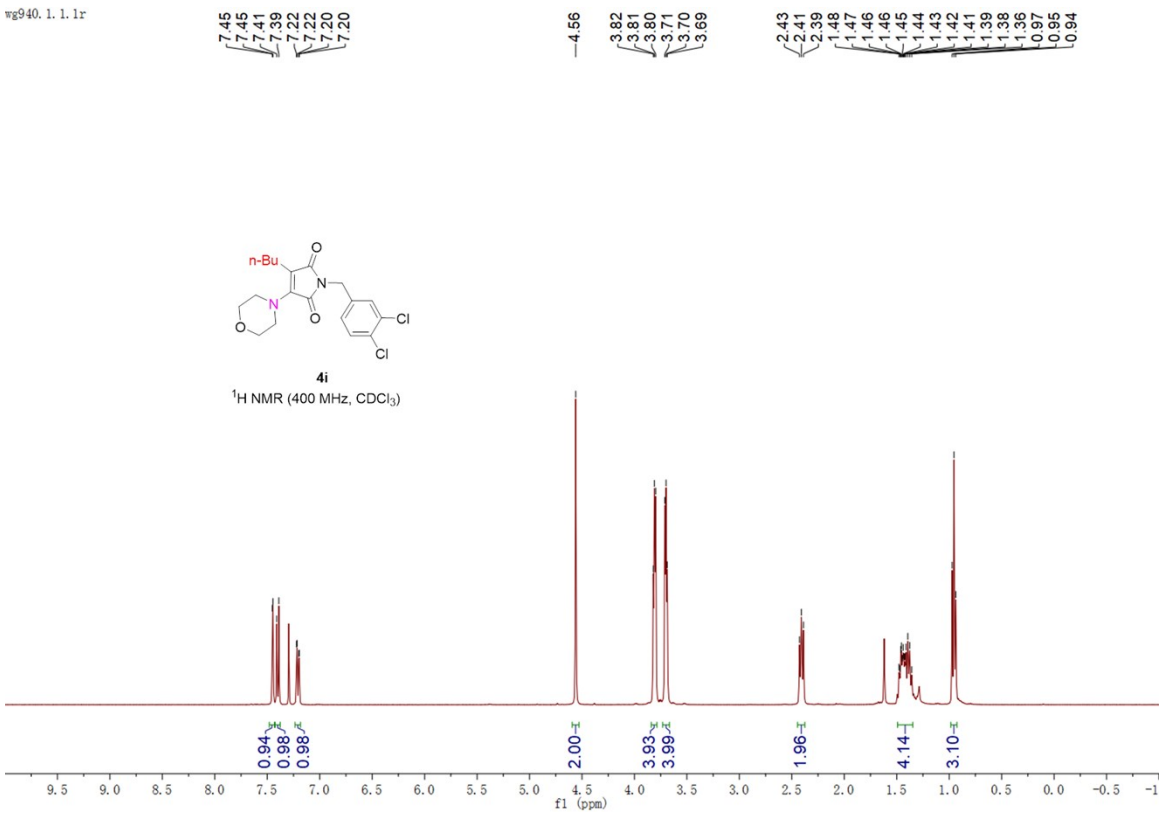

pdata/1
-62.56
n-Bu
O
N
N
O
O
CF3
4f
19F NMR (375 MHz, CDCl3)
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

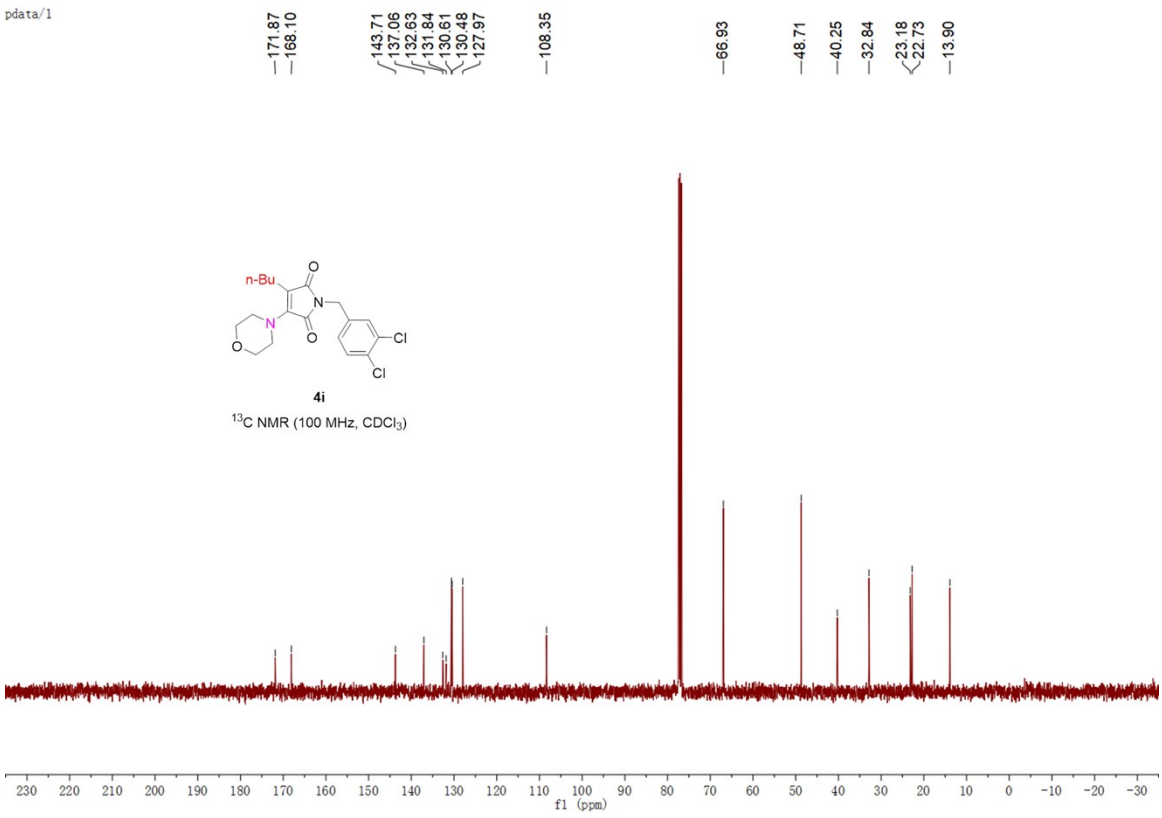

wg963.1.1.1r
8.35
8.33
7.91
7.90
7.88
7.88
7.84
7.82
7.62
7.61
7.58
7.57
7.55
7.53
7.51
7.49
7.47
7.45
5.11
3.81
3.80
3.79
3.79
3.69
3.69
3.68
3.67
2.45
2.43
2.41
1.49
1.49
1.48
1.47
1.46
1.46
1.45
1.45
1.44
1.43
1.41
1.41
1.40
1.39
1.39
1.37
0.98
0.96
n-Bu
O
N
N
O
O
4g
1H NMR (400 MHz, CDCl3)
0.99
1.01
1.01
3.03
1.01
2.00
3.94
3.93
1.97
4.14
3.02
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

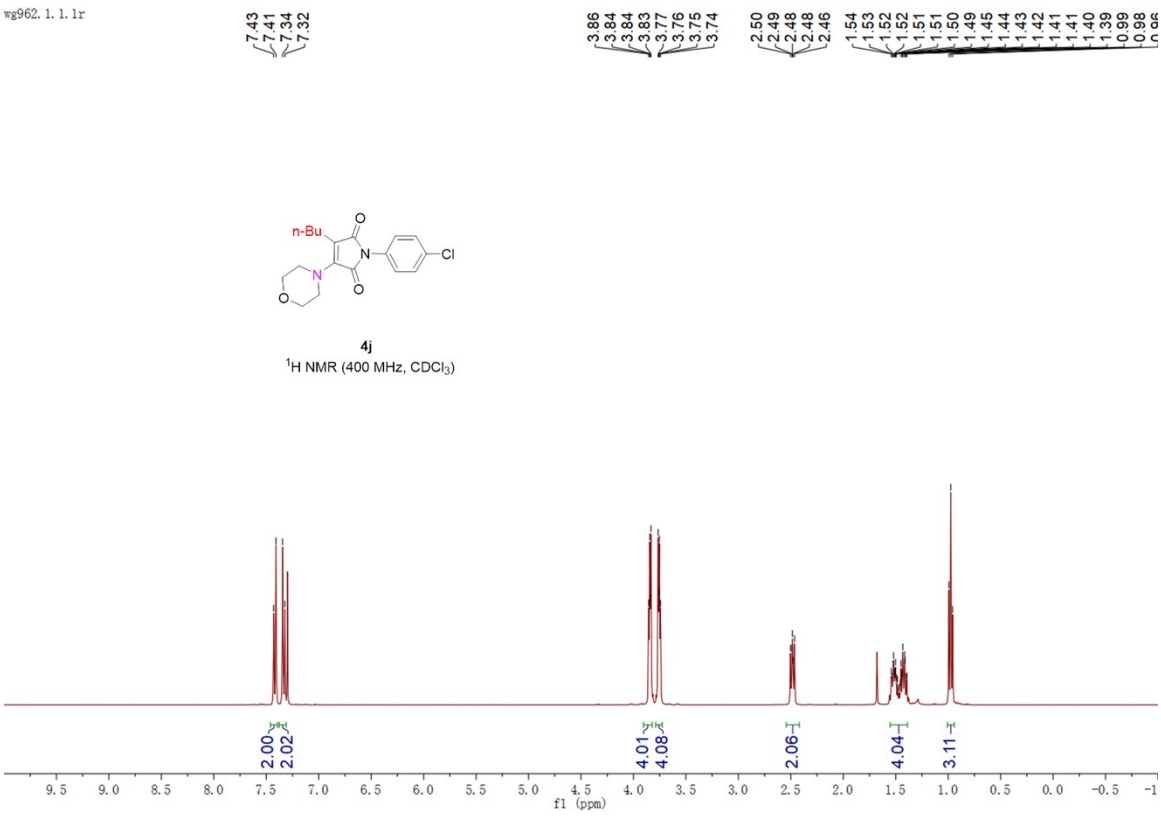

pdata/1
172.34
168.54
143.67
133.81
132.16
131.34
128.73
128.49
127.45
126.45
125.80
125.39
123.69
108.56
66.95
48.71
39.35
32.90
23.21
22.75
13.93
n-Bu
O
N
N
O
O
4g
13C NMR (100 MHz, CDCl3)
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30
f1 (ppm)

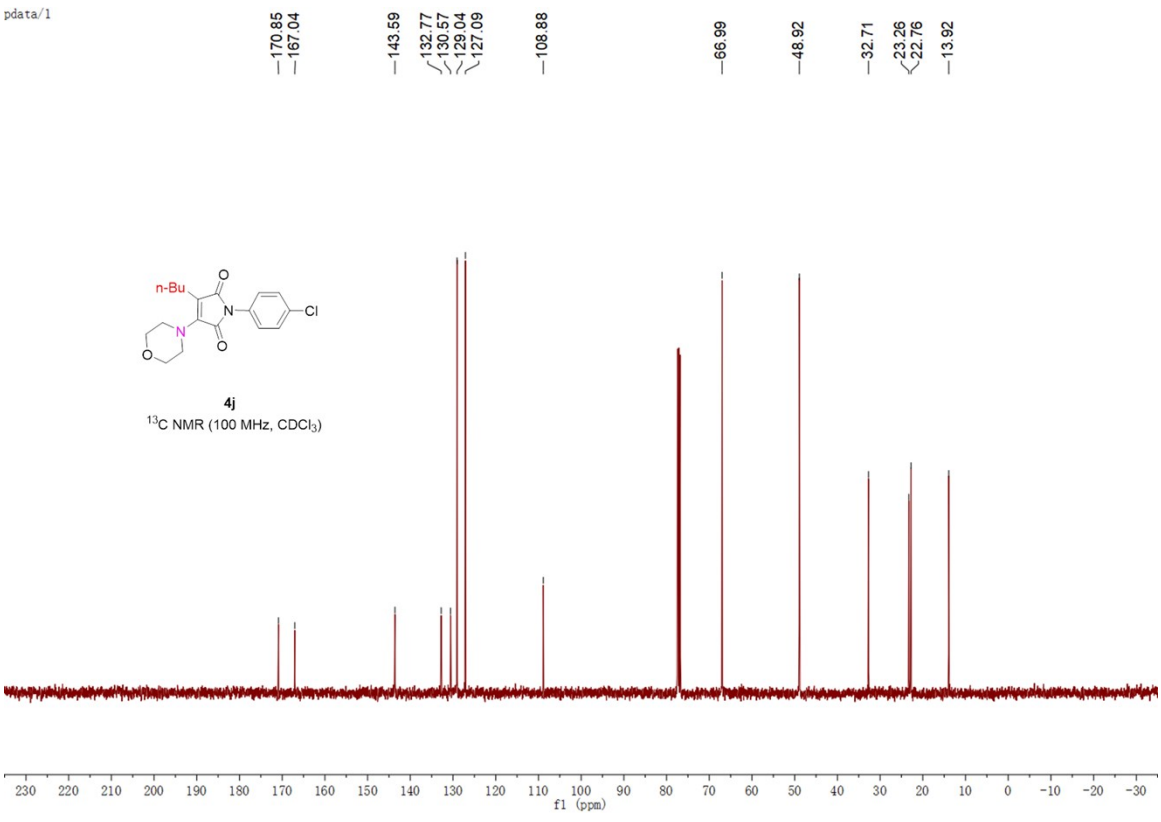

wg958.1.1.1r
7.24
7.24
7.23
7.23
7.08
7.08
7.07
6.97
6.96
6.96
6.95
4.80
3.82
3.81
3.80
3.71
3.70
3.68
2.43
2.41
2.39
1.46
1.46
1.44
1.43
1.43
1.42
1.41
1.41
1.40
1.39
1.39
1.38
1.38
0.98
0.97
0.96
0.95
0.94
n-Bu
4h
1H NMR (400 MHz, CDCl3)
0.96
0.96
1.01
2.00
4.01
4.01
1.96
4.34
3.12
f1 (ppm)

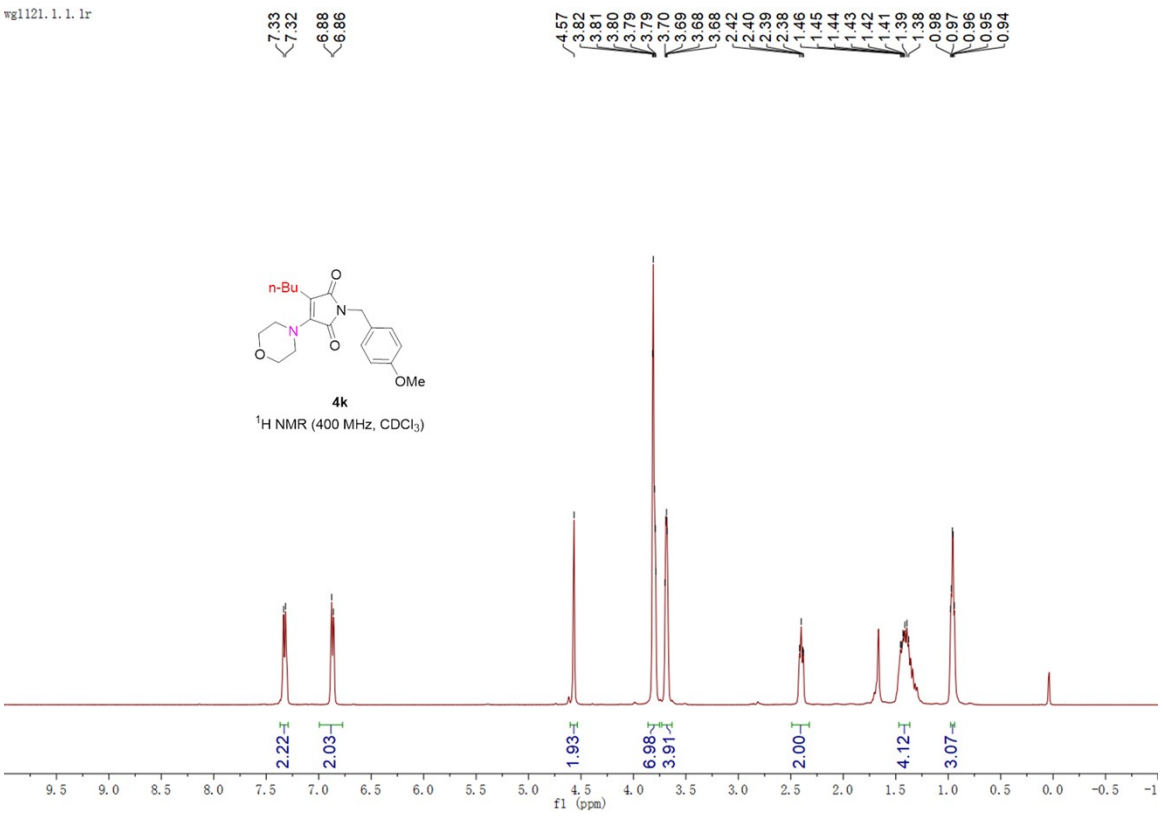

pdata/1
171.70
167.90
143.72
138.90
127.27
126.86
125.60
108.51
66.95
48.70
35.48
32.84
23.14
22.72
13.90
n-Bu
4h
13C NMR (100 MHz, CDCl3)
f1 (ppm)

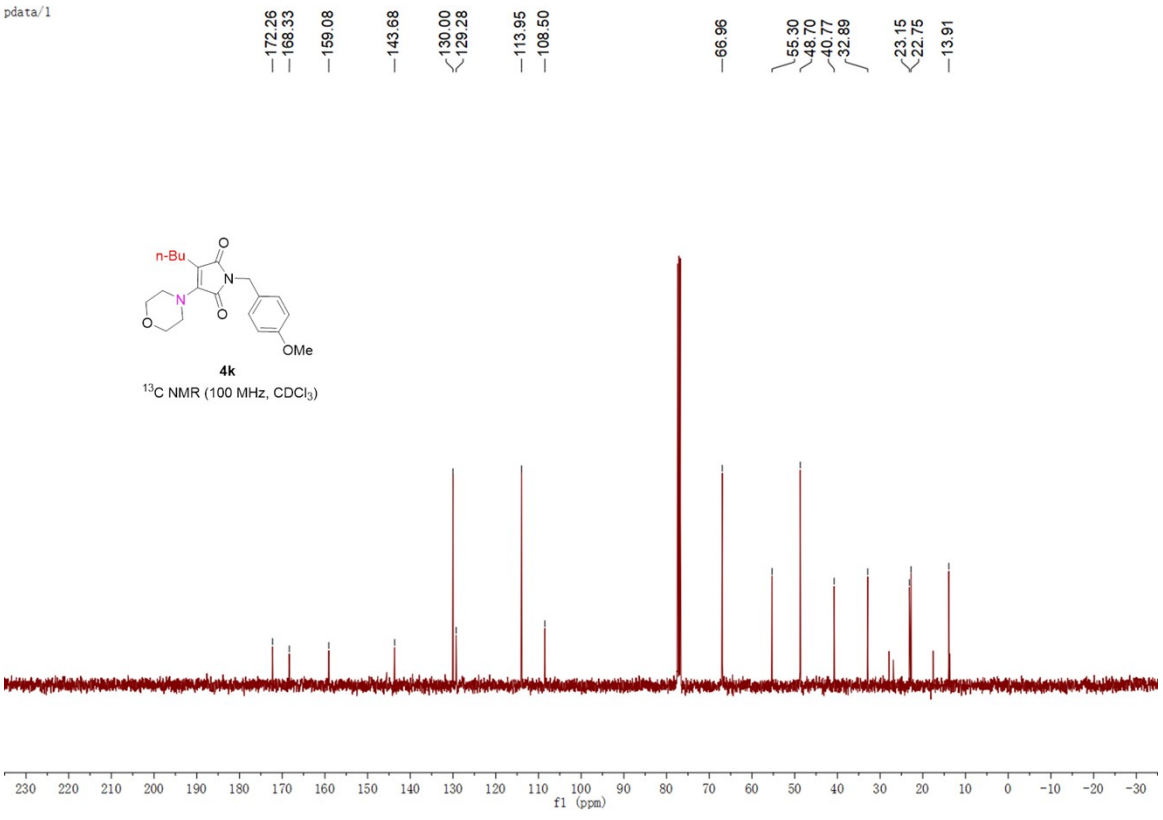

wg940.1.1.1r
7.45
7.45
7.41
7.39
7.22
7.22
7.20
7.20
4.56
3.82
3.81
3.80
3.71
3.70
3.69
2.43
2.41
2.39
1.48
1.47
1.46
1.46
1.45
1.44
1.43
1.42
1.41
1.39
1.38
1.36
0.97
0.95
0.94
n-Bu
O
N
O
N
O
Cl
Cl
4i
1H NMR (400 MHz, CDCl3)
0.94
0.98
0.98
2.00
3.93
3.99
1.96
4.14
3.10
f1 (ppm)

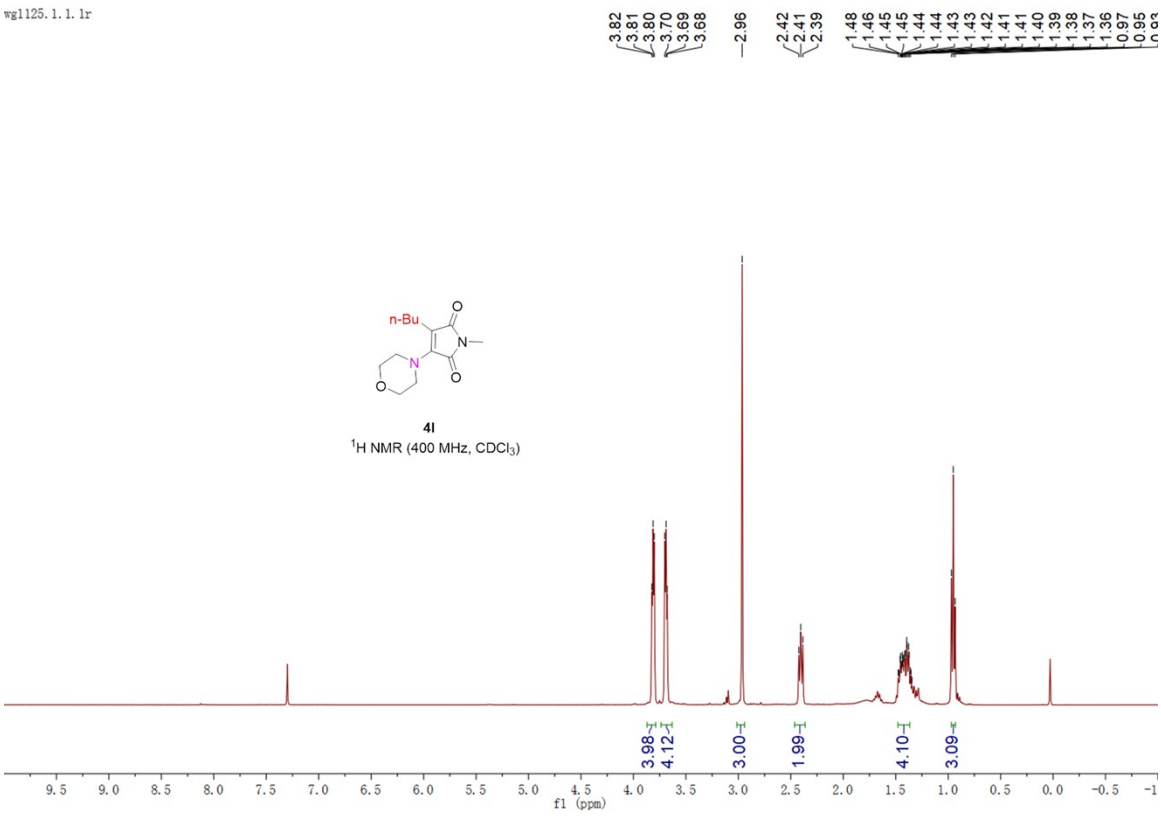

pdata/1
171.87
168.10
143.71
137.06
132.63
131.84
130.61
130.48
127.97
108.35
66.93
48.71
40.25
32.84
23.18
22.73
13.90
n-Bu
O
N
O
N
O
Cl
Cl
4i
13C NMR (100 MHz, CDCl3)
f1 (ppm)

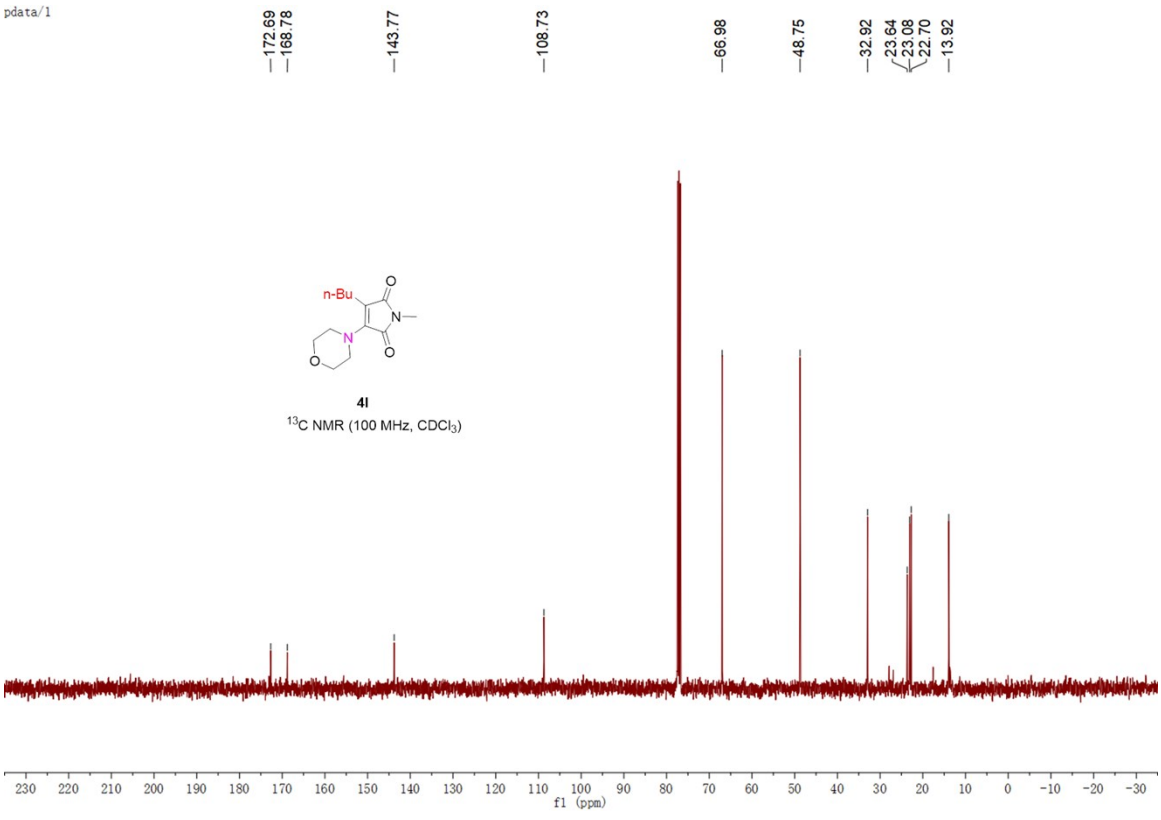

wg962.1.1.1r
7.43
7.41
7.34
7.32
3.86
3.84
3.84
3.83
3.77
3.76
3.75
3.74
2.50
2.49
2.48
2.48
2.46
1.54
1.53
1.52
1.52
1.51
1.51
1.50
1.49
1.45
1.44
1.43
1.42
1.41
1.41
1.40
1.39
0.99
0.98
n-Bu
N
O
O
Cl
4j
1H NMR (400 MHz, CDCl3)
2.00
2.02
4.01
4.08
2.06
4.04
3.11
f1 (ppm)

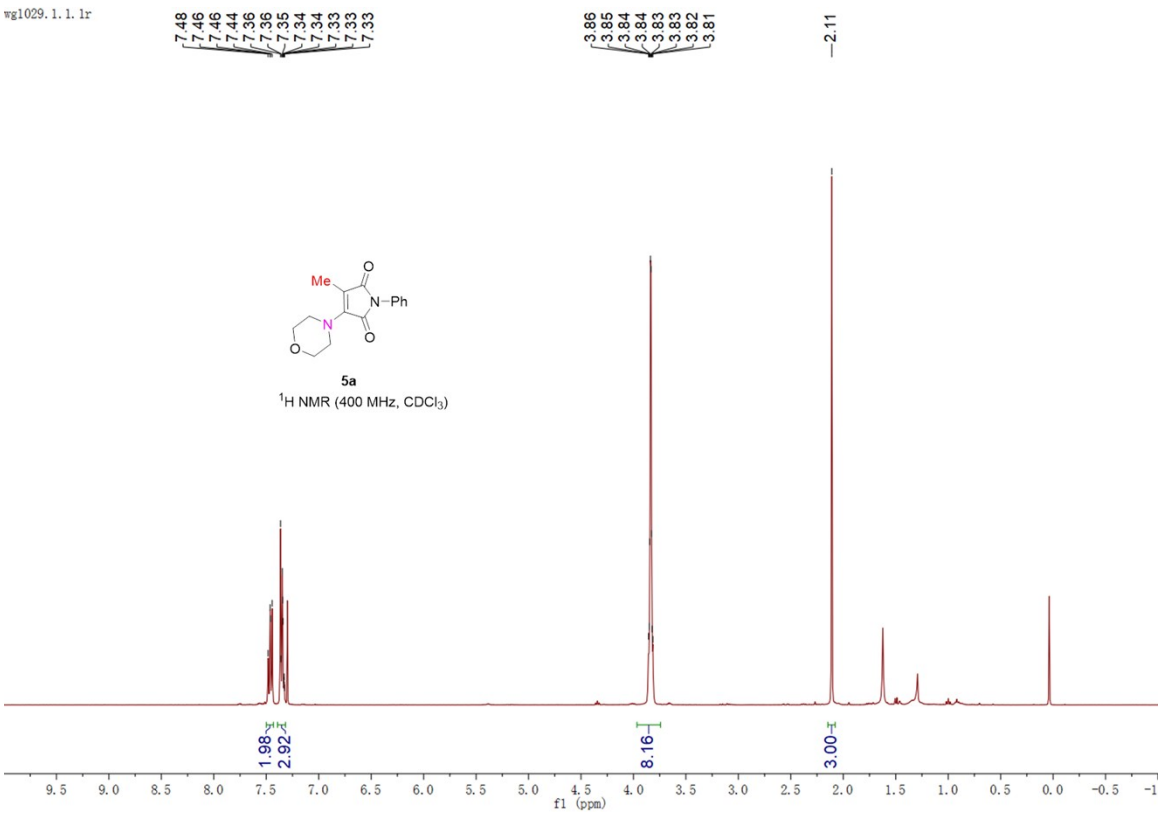

pdata/1
170.85
167.04
143.59
132.77
130.57
129.04
127.09
108.88
66.99
48.92
32.71
23.26
22.76
13.92
n-Bu
N
O
O
Cl
4j
13C NMR (100 MHz, CDCl3)
f1 (ppm)


wg1121.1.1.1r
7.33
7.32
6.88
6.86
4.57
3.82
3.81
3.80
3.79
3.79
3.70
3.69
3.68
3.68
2.42
2.40
2.39
2.38
1.46
1.45
1.44
1.43
1.42
1.41
1.39
1.38
0.98
0.97
0.96
0.95
0.94
n-Bu
N
O
O
N
O
OMe
4k
1H NMR (400 MHz, CDCl3)
2.22
2.03
1.93
6.98
3.91
2.00
4.12
3.07
f1 (ppm)


pdata/1
172.26
168.33
159.08
143.68
130.00
129.28
113.95
108.50
66.96
55.30
48.70
40.77
32.89
23.15
22.75
13.91
n-Bu
N
O
O
N
O
OMe
4k
13C NMR (100 MHz, CDCl3)
f1 (ppm)

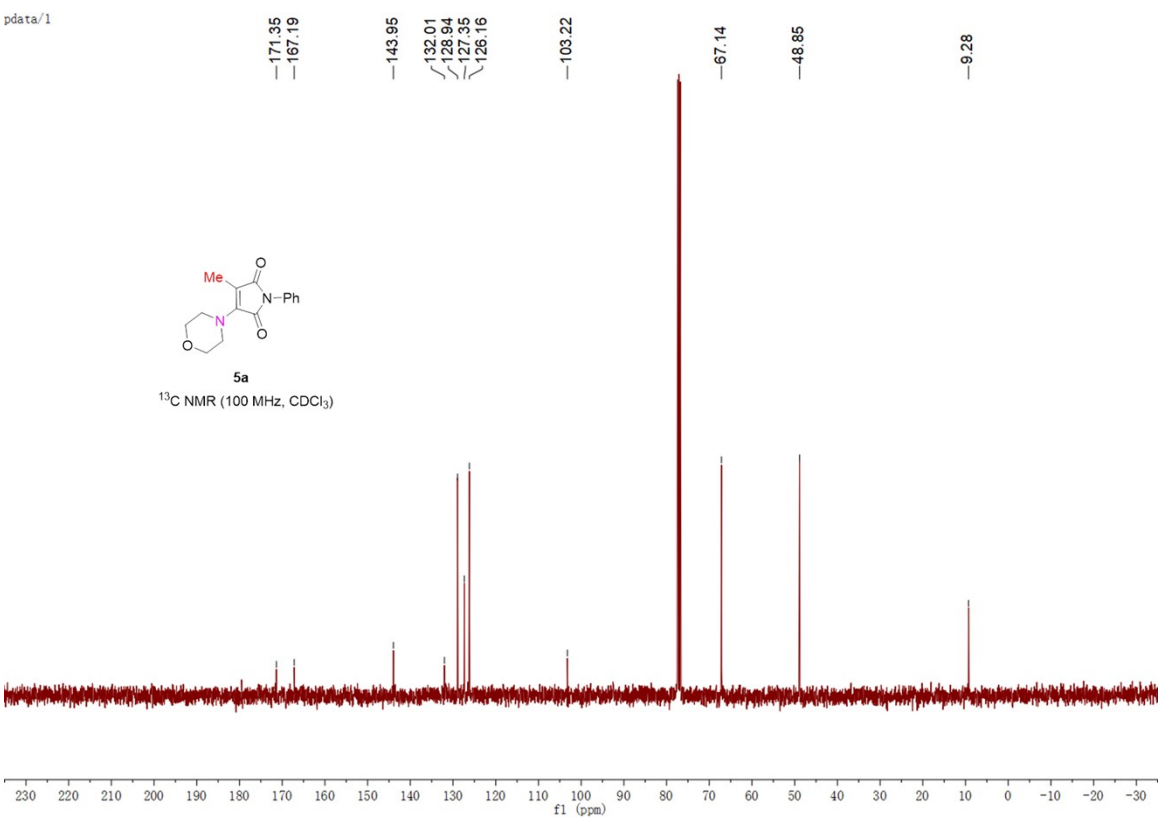

wgl125.1.1.1r
3.82
3.81
3.80
3.70
3.69
3.68
2.96
2.42
2.41
2.39
1.48
1.46
1.45
1.45
1.44
1.44
1.43
1.43
1.42
1.41
1.41
1.40
1.39
1.38
1.37
1.36
0.97
0.95
n-Bu
N
O
O
O
4l
1H NMR (400 MHz, CDCl3)
3.98
4.12
3.00
1.99
4.10
3.09
f1 (ppm)


pdata/1
172.69
168.78
143.77
108.73
66.98
48.75
32.92
23.64
23.08
22.70
13.92
n-Bu
N
O
O
O
4l
13C NMR (100 MHz, CDCl3)
f1 (ppm)



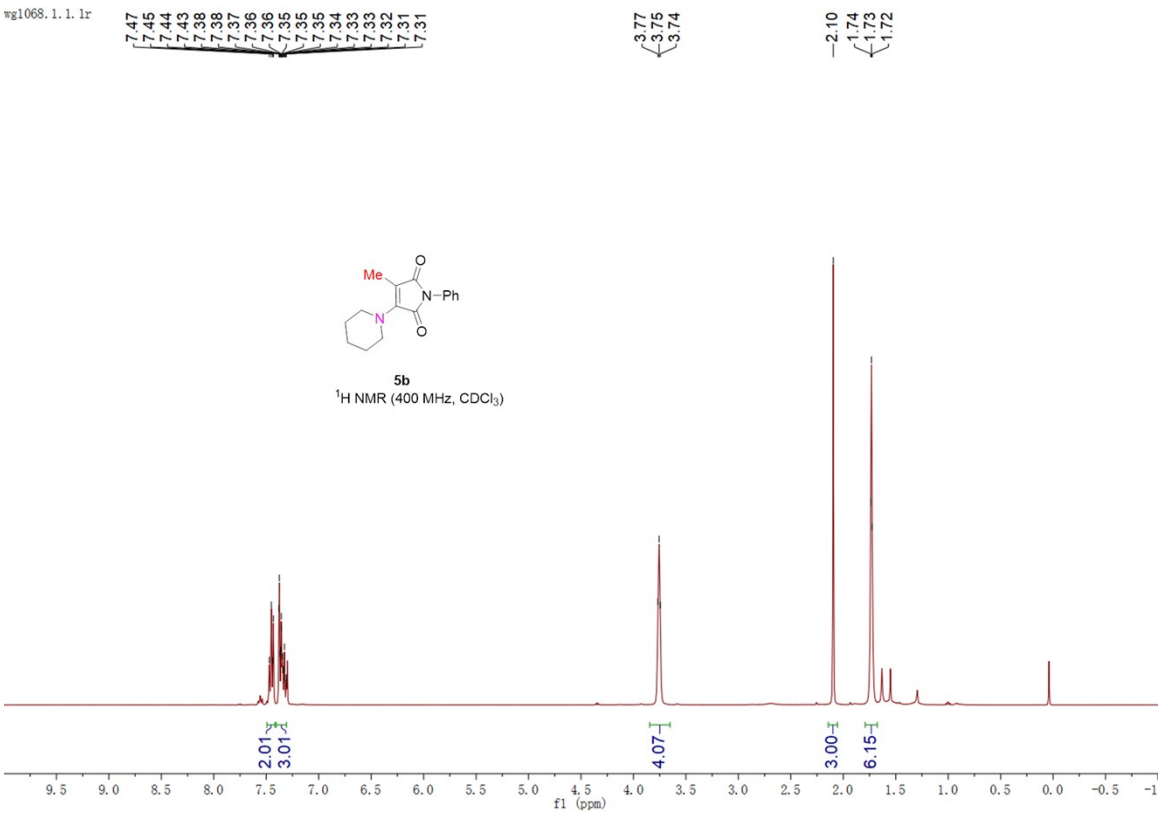
5a

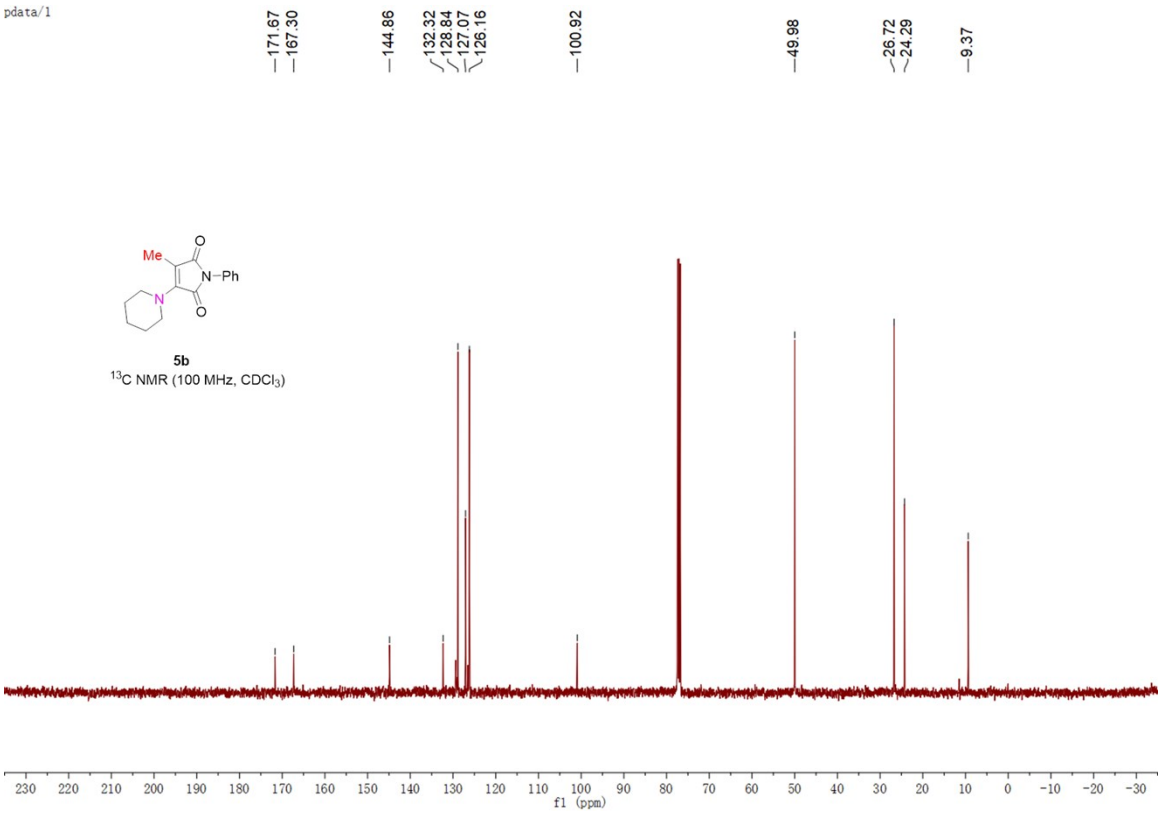
^{1}H NMR (400 MHz, $CDCl_3$)

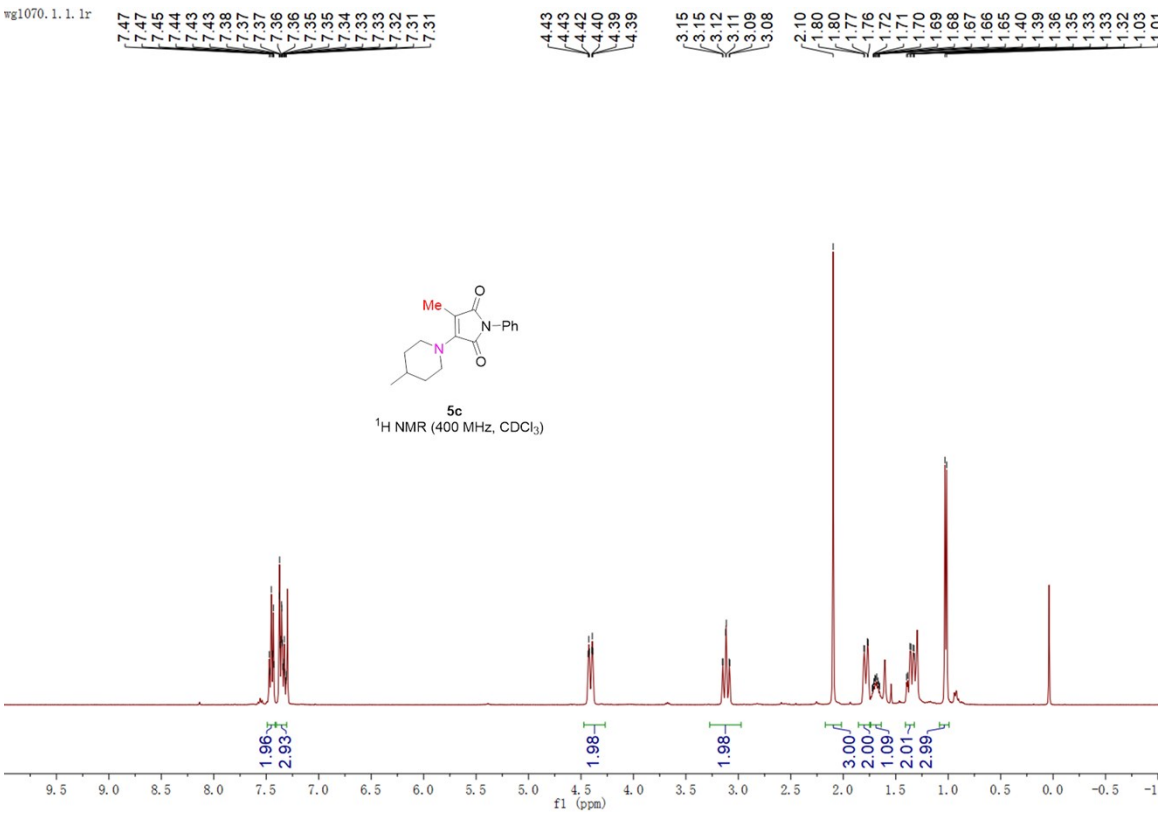


5a

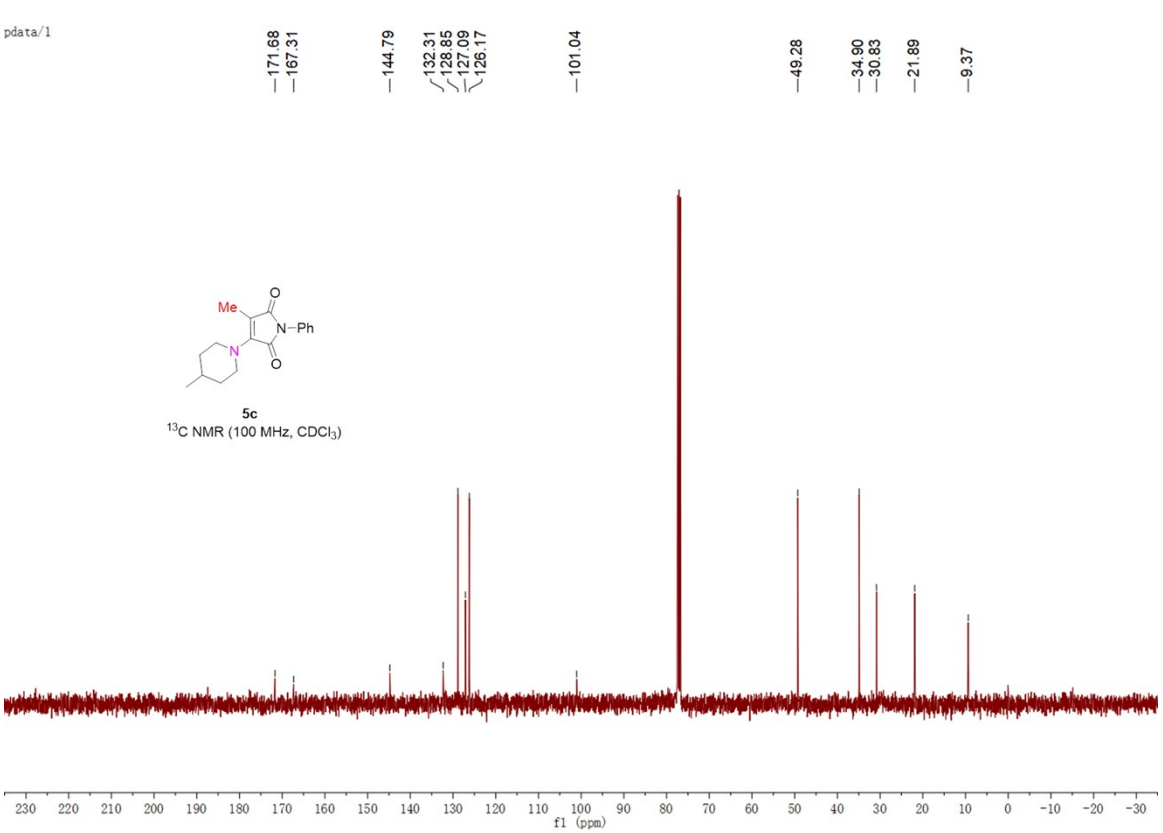
^{13}C NMR (100 MHz, $CDCl_3$)


wg1068.1.1.1r
7.47
7.45
7.44
7.43
7.38
7.38
7.37
7.36
7.36
7.35
7.35
7.35
7.34
7.33
7.33
7.32
7.31
7.31
3.77
3.75
3.74
2.10
1.74
1.73
1.72
Me
N–Ph
O
O
N
5b
1H NMR (400 MHz, CDCl3)
2.01
3.01
4.07
3.00
6.15
f1 (ppm)

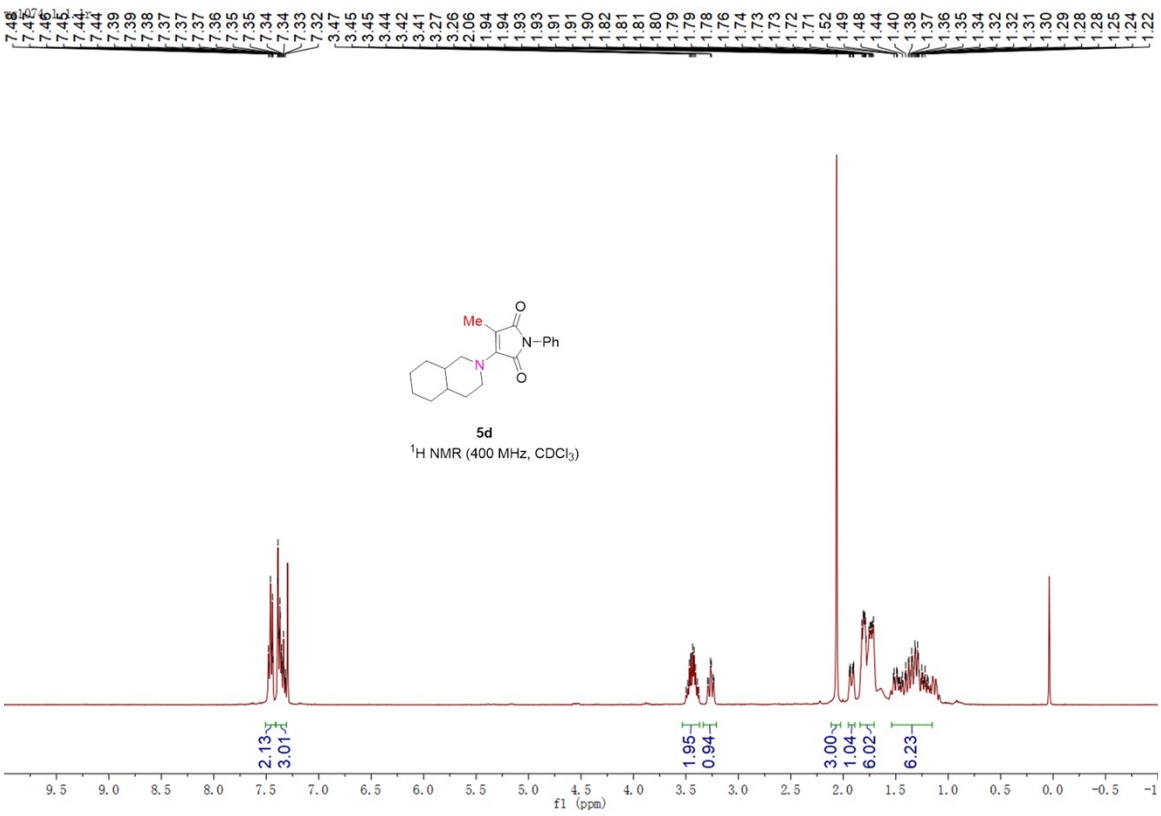

pdata/1
171.67
167.30
144.86
132.32
128.84
127.07
126.16
100.92
49.98
26.72
24.29
9.37
Me
N–Ph
O
O
N
5b
13C NMR (100 MHz, CDCl3)
f1 (ppm)

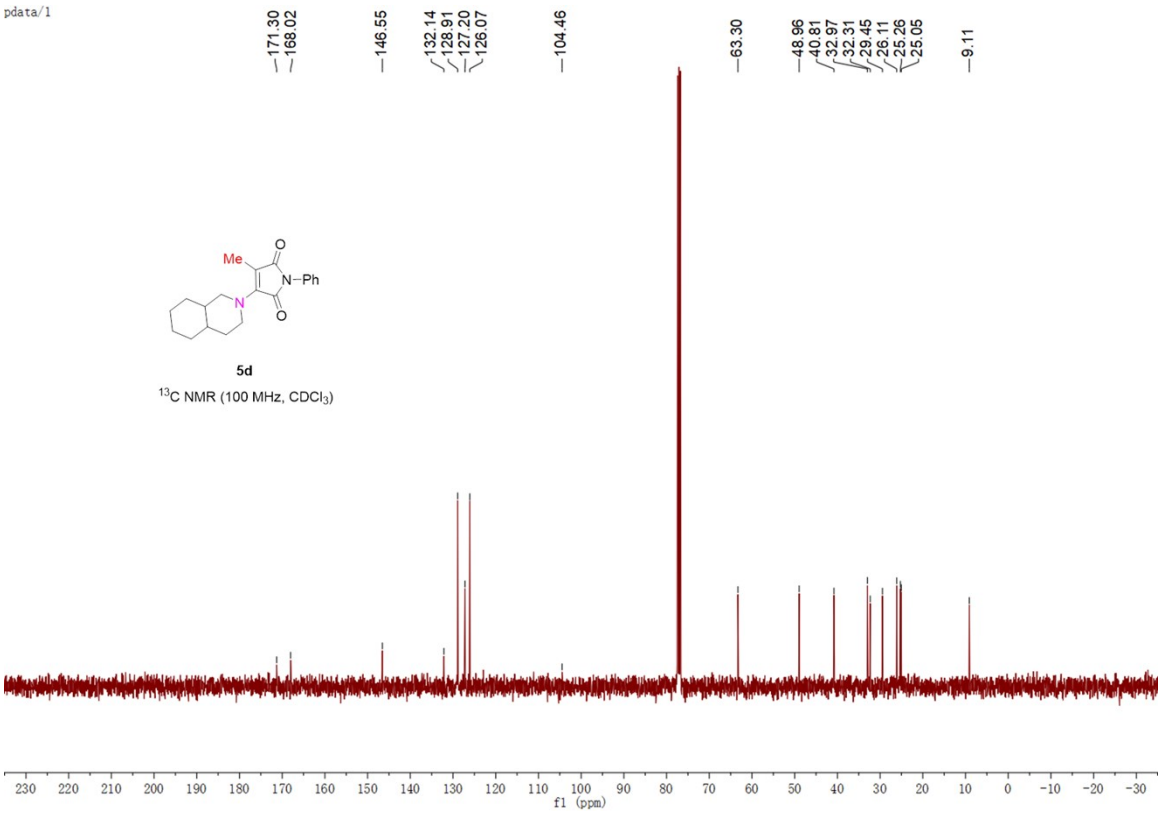


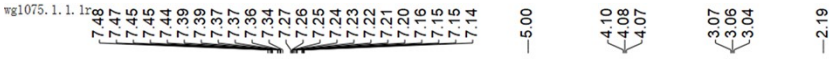
5c
^{1}H NMR (400 MHz, $CDCl_3$)

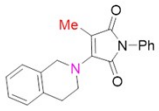


5c
^{13}C NMR (100 MHz, $CDCl_3$)


Me
N–Ph
O
O
N
5d
1H NMR (400 MHz, CDCl3)
2.13
3.01
1.95
0.94
3.00
1.04
6.02
6.23
f1 (ppm)

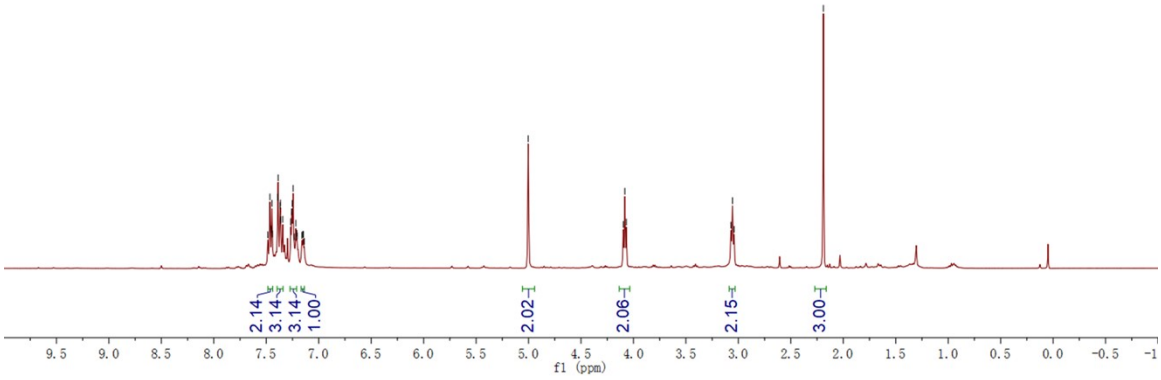

pdata/1
171.30
168.02
146.55
132.14
128.91
127.20
126.07
104.46
63.30
48.96
40.81
32.97
32.31
29.45
26.11
25.26
25.05
9.11
Me
N–Ph
O
O
N
5d
13C NMR (100 MHz, CDCl3)
f1 (ppm)

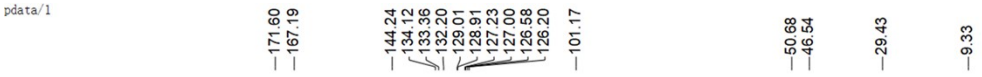


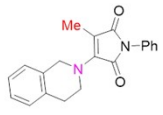


5e

^{1}H NMR (400 MHz, $CDCl_3$)

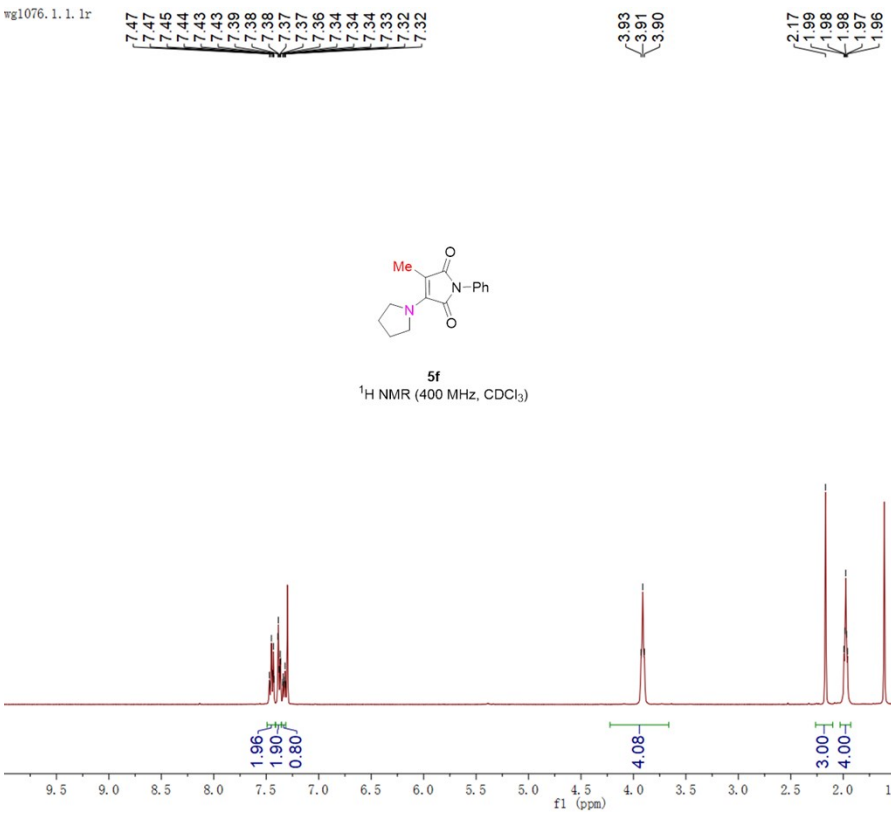


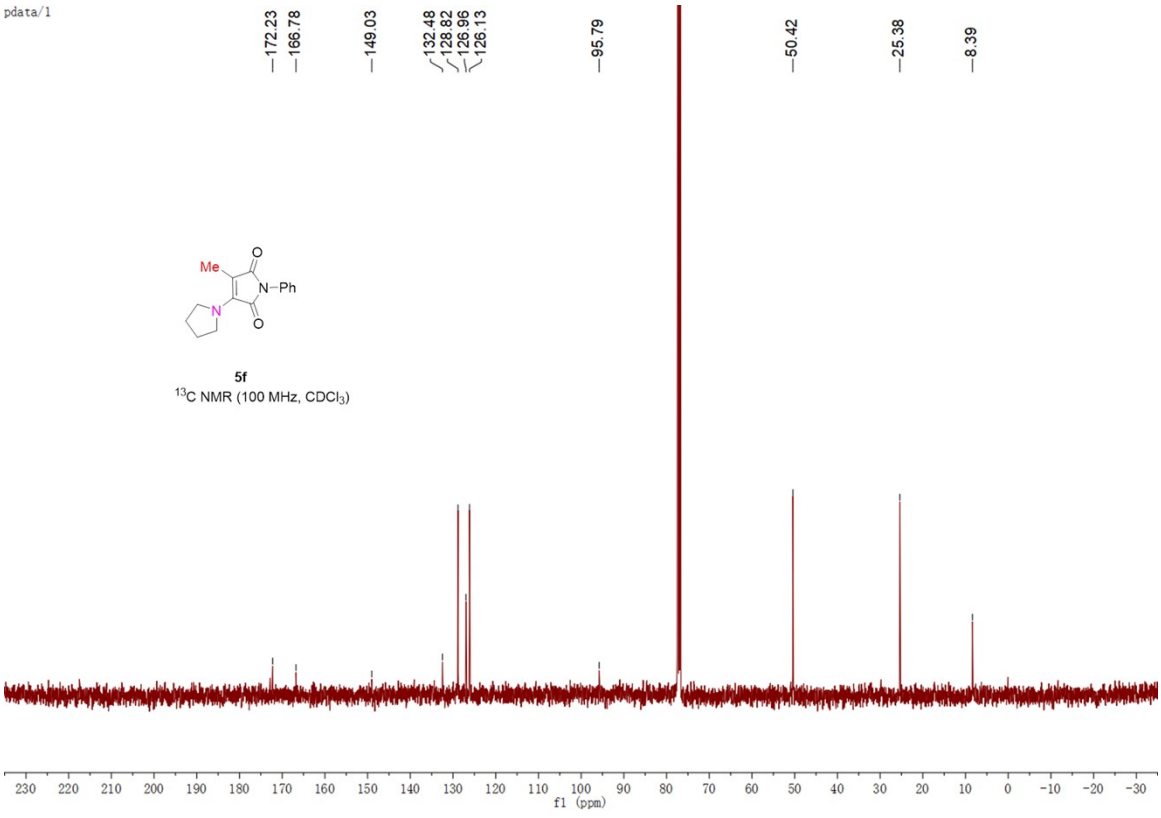


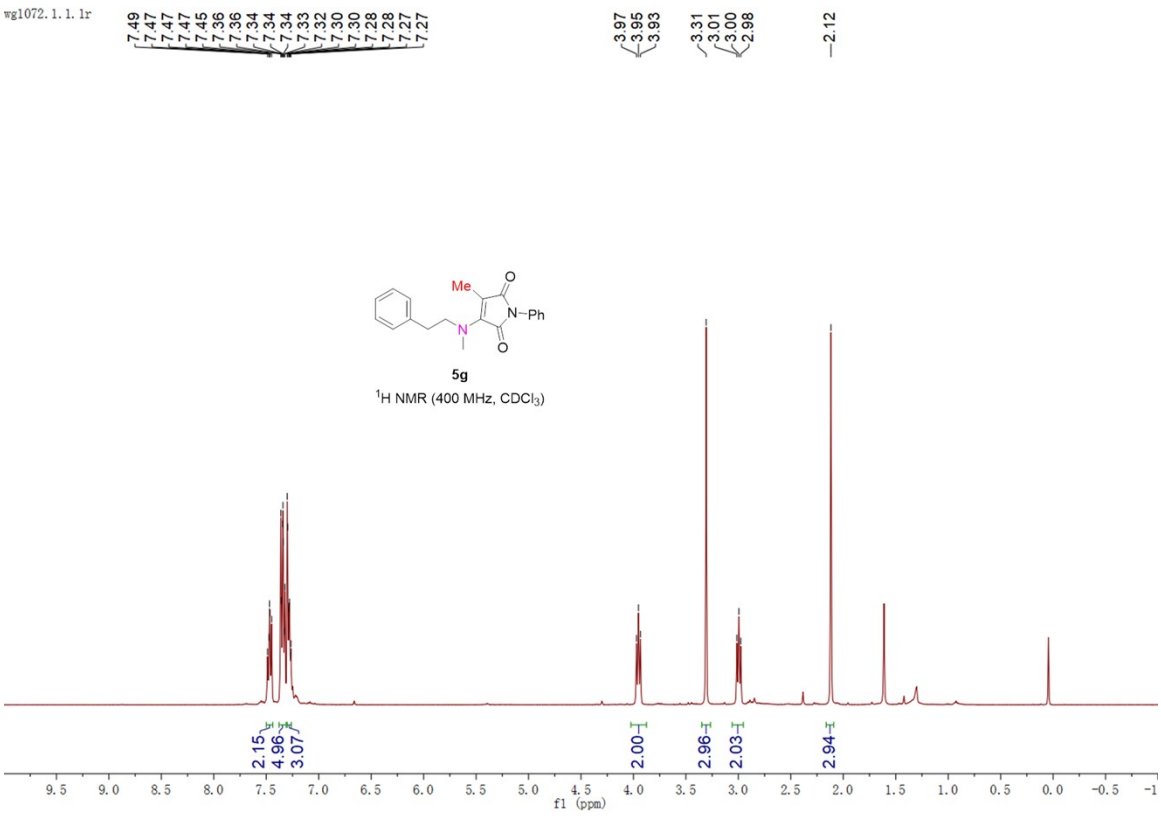


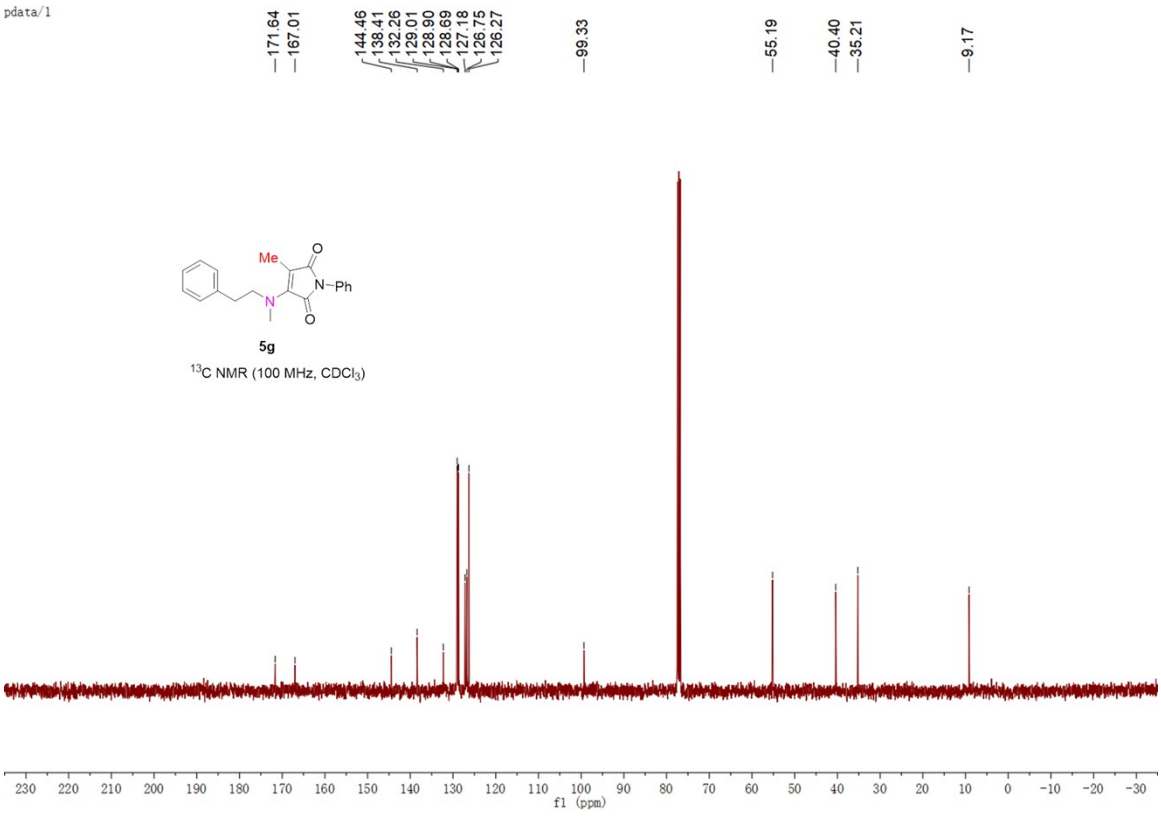
5e

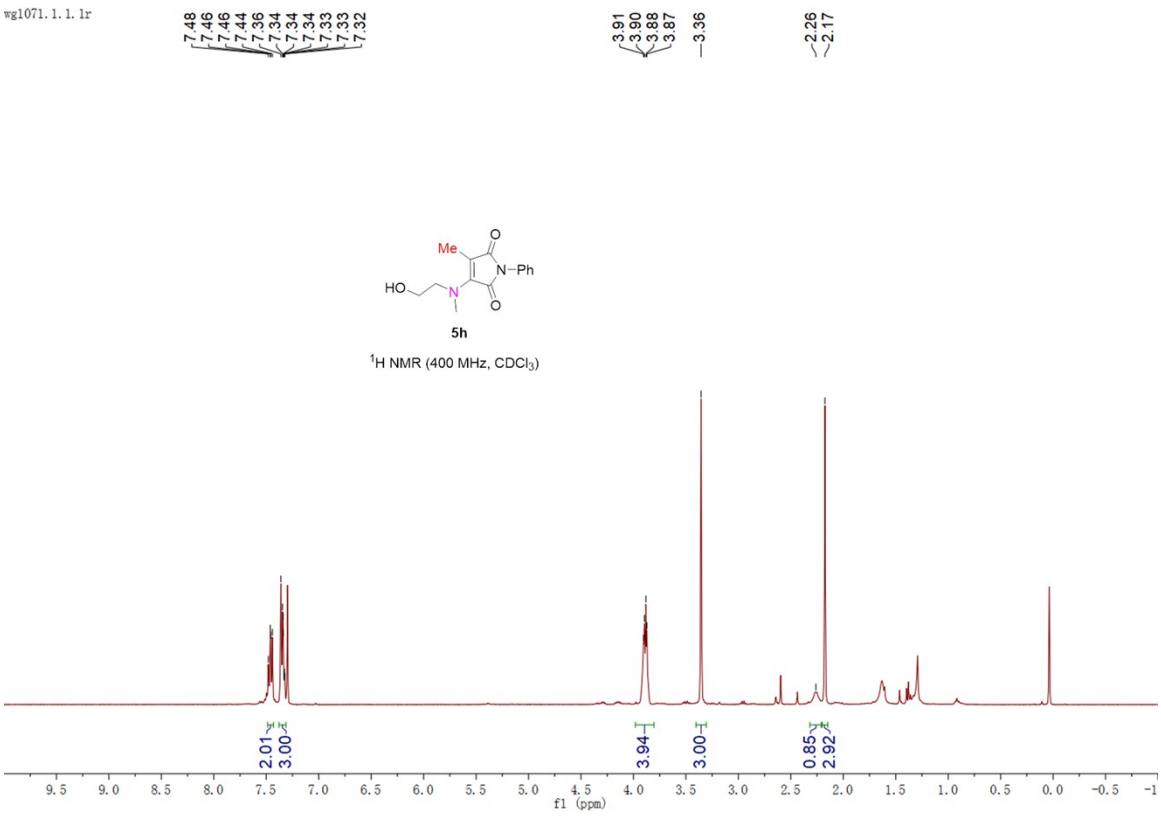
^{13}C NMR (100 MHz, $CDCl_3$)

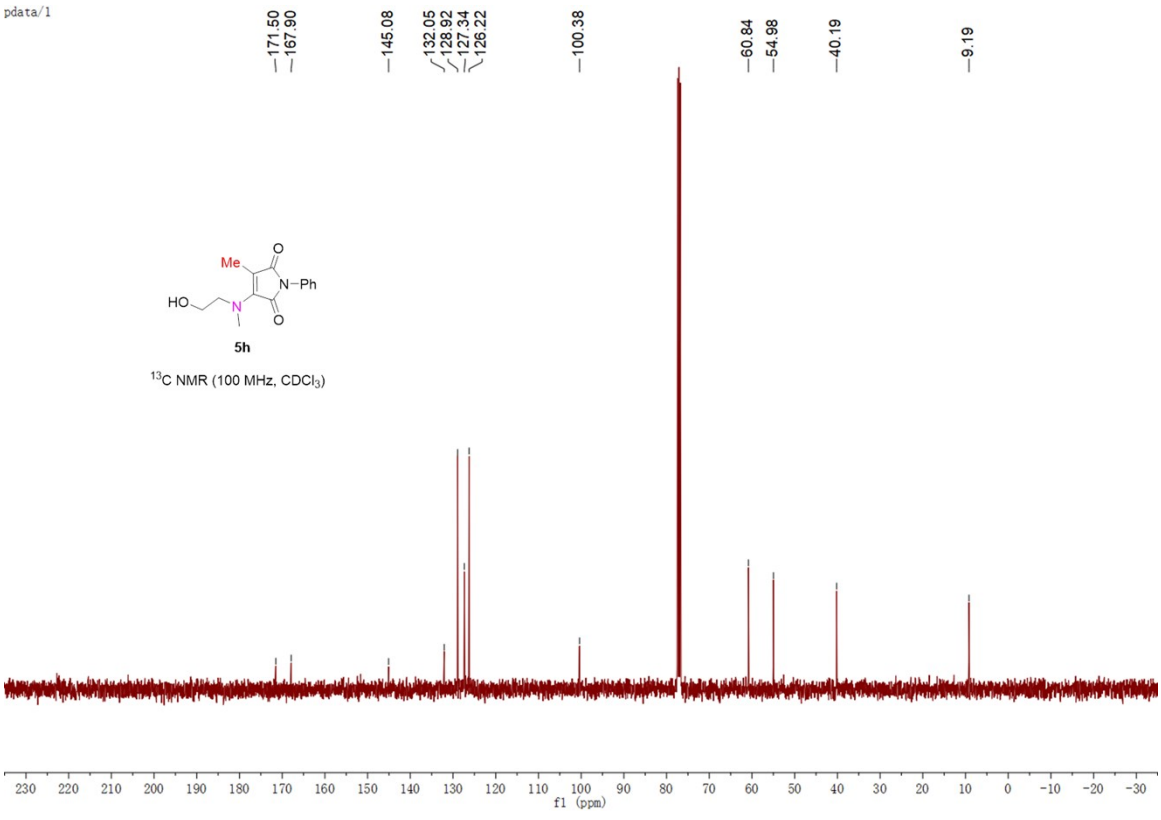

wg1076.1.1.1r
7.47
7.47
7.45
7.44
7.43
7.43
7.39
7.38
7.38
7.37
7.37
7.36
7.34
7.34
7.33
7.32
7.32
3.93
3.91
3.90
2.17
1.99
1.98
1.98
1.97
1.96
Me
N–Ph
N
O
O
5f
1H NMR (400 MHz, CDCl3)
1.96
1.90
0.80
4.08
3.00
4.00
f1 (ppm)

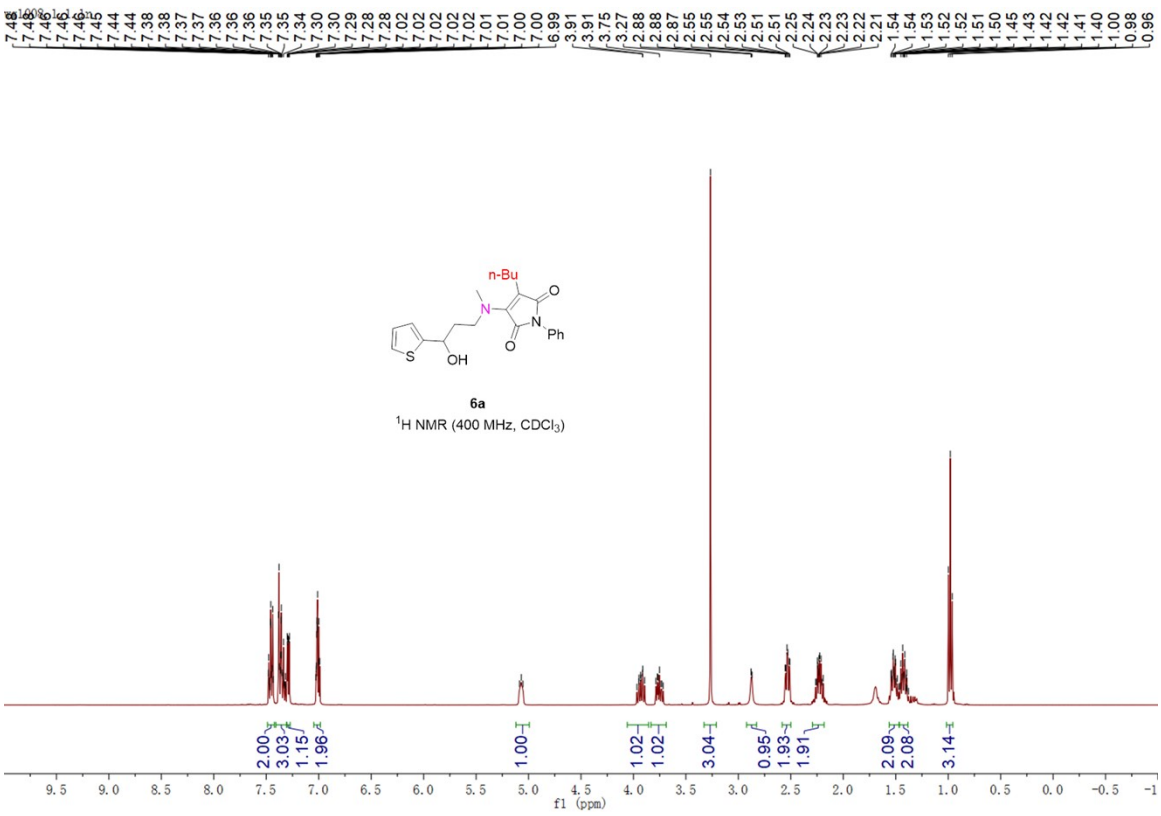

pdata/1
172.23
166.78
149.03
132.48
128.82
126.96
126.13
95.79
50.42
25.38
8.39
Me
N–Ph
N
O
O
5f
13C NMR (100 MHz, CDCl3)
f1 (ppm)

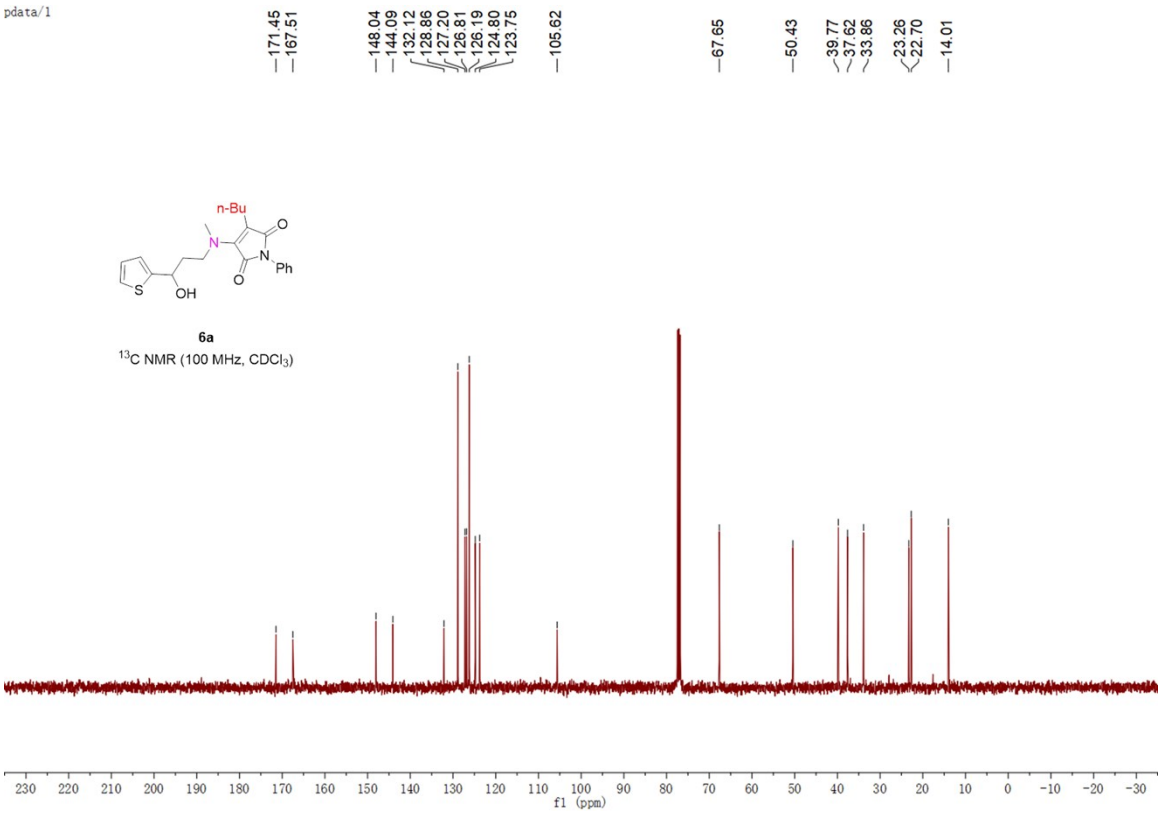

wg1072.1.1.1r
7.49
7.47
7.47
7.47
7.45
7.36
7.36
7.34
7.34
7.33
7.32
7.30
7.30
7.28
7.27
7.27
3.97
3.95
3.93
3.31
3.01
3.00
2.98
2.12
Me
O
N–Ph
N
O
5g
1H NMR (400 MHz, CDCl3)
2.15
4.96
3.07
2.00
2.96
2.03
2.94
f1 (ppm)

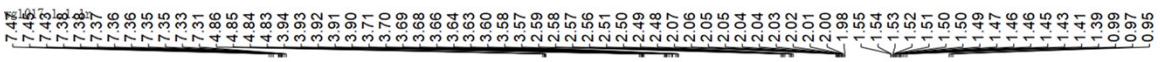

pdata/1
171.64
167.01
144.46
138.41
132.26
129.01
128.90
128.69
127.18
126.75
126.27
99.33
55.19
40.40
35.21
9.17
Me
O
N–Ph
N
O
5g
13C NMR (100 MHz, CDCl3)
f1 (ppm)

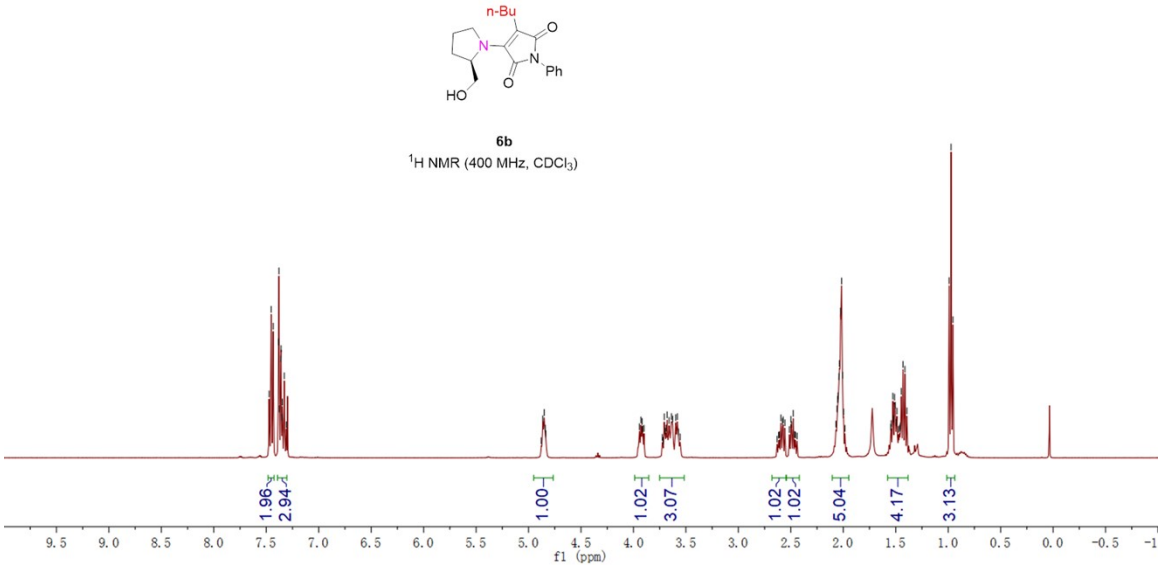

wg1071.1.1.1r
7.48
7.46
7.46
7.44
7.36
7.34
7.34
7.34
7.33
7.33
7.32
3.91
3.90
3.88
3.87
3.36
2.26
2.17
Me
O
N–Ph
HO
N
O
5h
1H NMR (400 MHz, CDCl3)
2.01
3.00
3.94
3.00
0.85
2.92
f1 (ppm)


pdata/1
171.50
167.90
145.08
132.05
128.92
127.34
126.22
100.38
60.84
54.98
40.19
9.19
Me
O
N–Ph
HO
N
O
5h
13C NMR (100 MHz, CDCl3)
f1 (ppm)


n-Bu
N
O
N
Ph
O
S
OH
6a
1H NMR (400 MHz, CDCl3)
2.00
3.03
1.15
1.96
1.00
1.02
1.02
3.04
0.95
1.93
1.91
2.09
2.08
3.14
f1 (ppm)

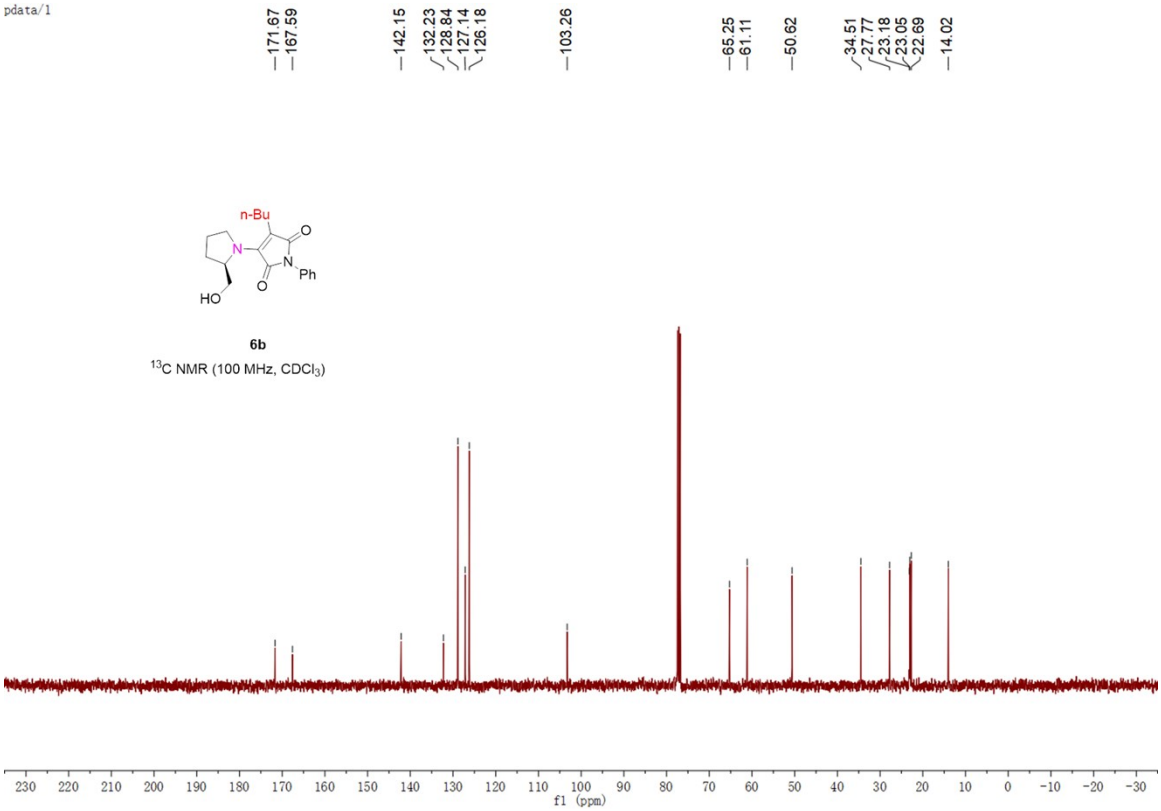

pdata/1
171.45
167.51
148.04
144.09
132.12
128.86
127.20
126.81
126.19
124.80
123.75
105.62
67.65
50.43
39.77
37.62
33.86
23.26
22.70
14.01
n-Bu
N
O
N
Ph
O
S
OH
6a
13C NMR (100 MHz, CDCl3)
f1 (ppm)





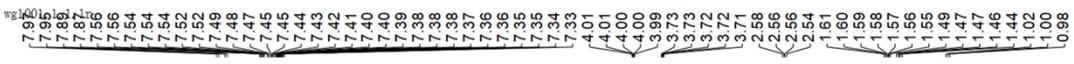
6b

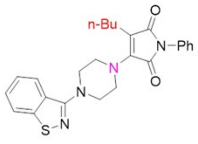
^{1}H NMR (400 MHz, $CDCl_3$)



6b

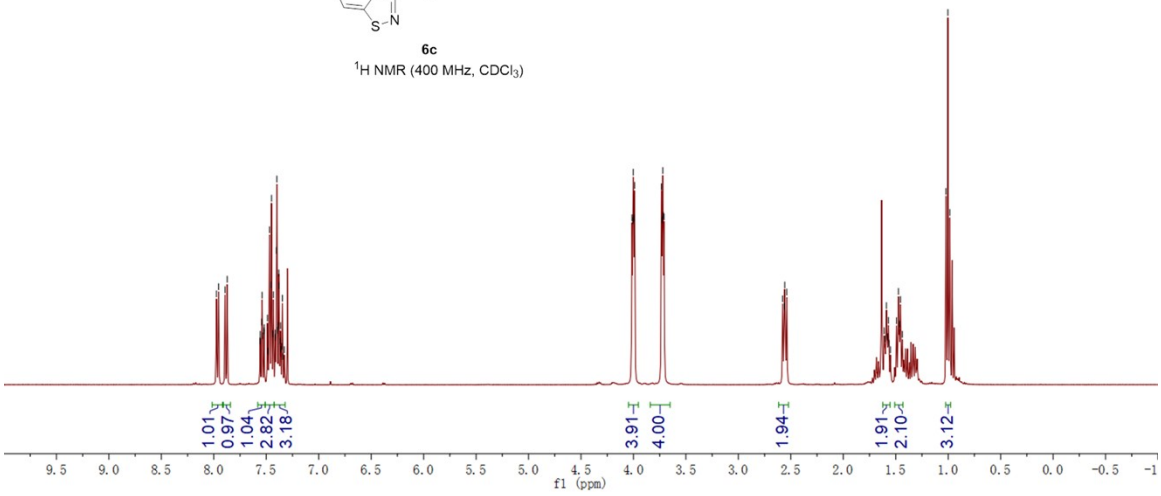
^{13}C NMR (100 MHz, $CDCl_3$)

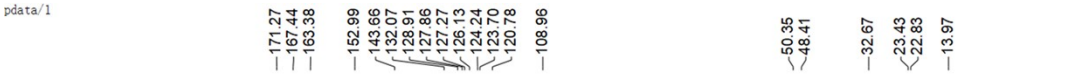


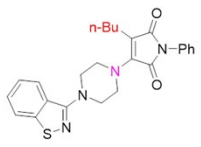


6c

^{1}H NMR (400 MHz, $CDCl_3$)

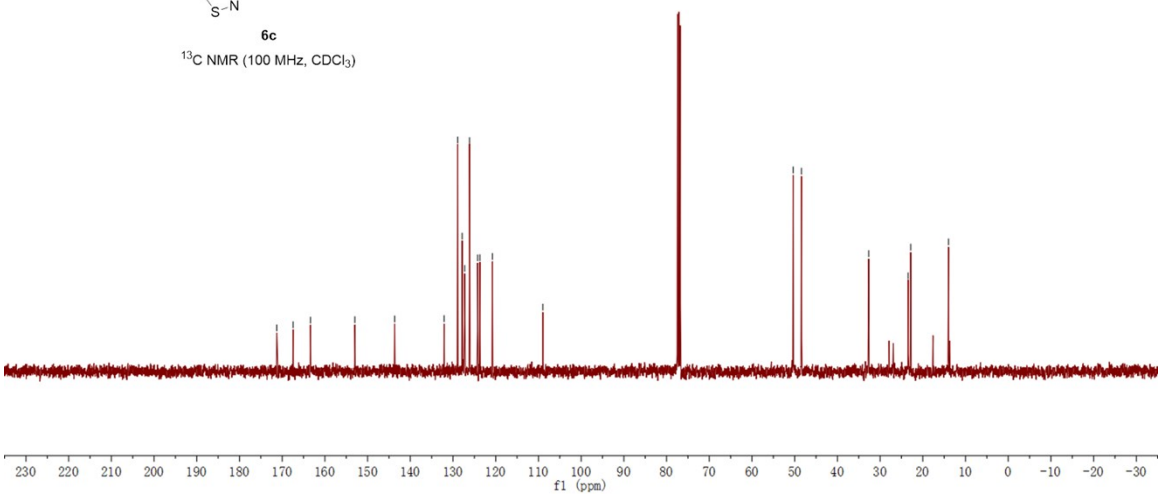




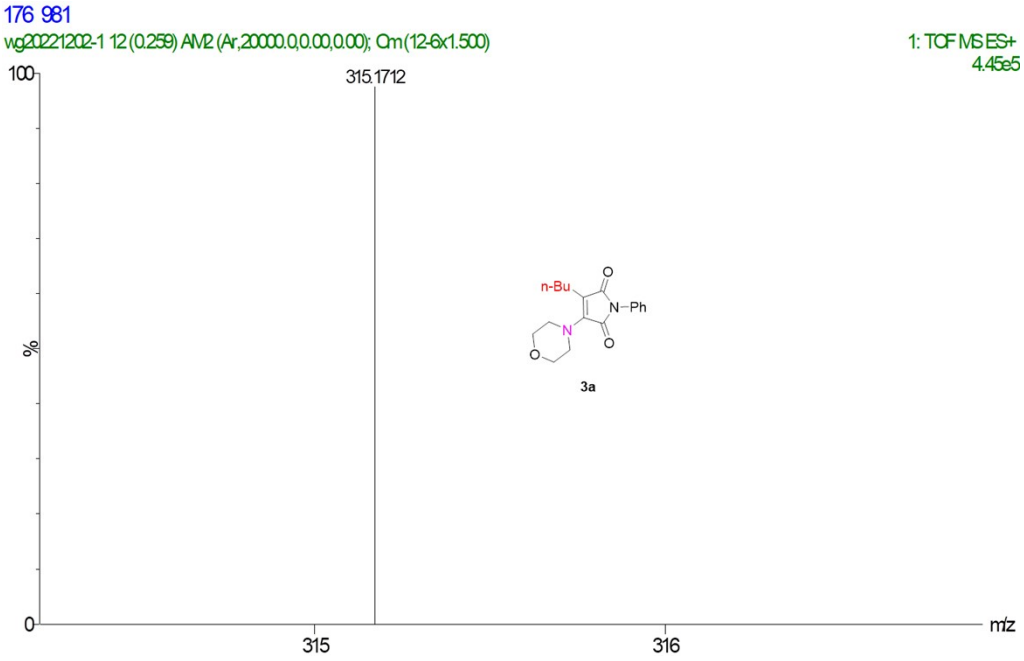


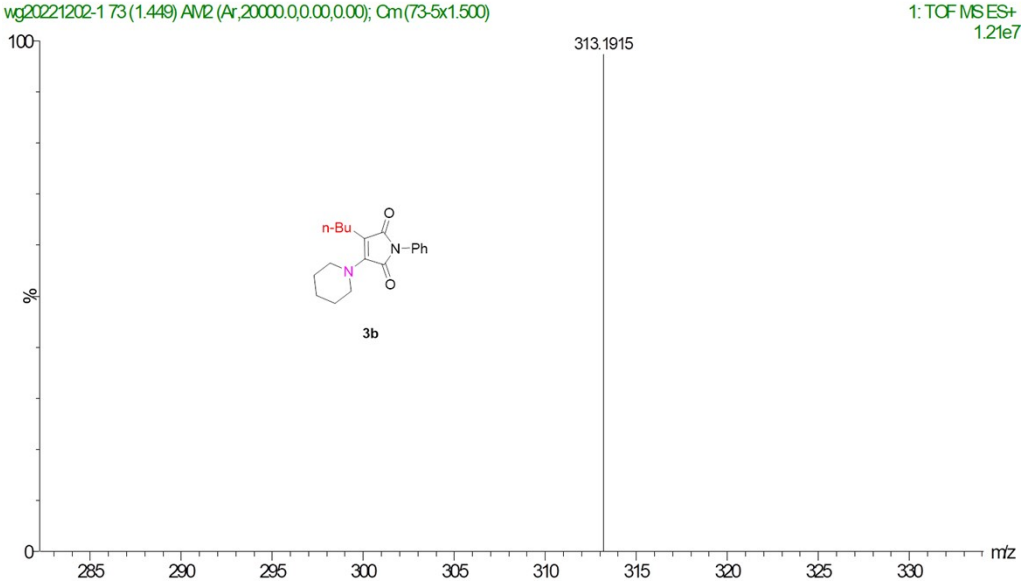
6c

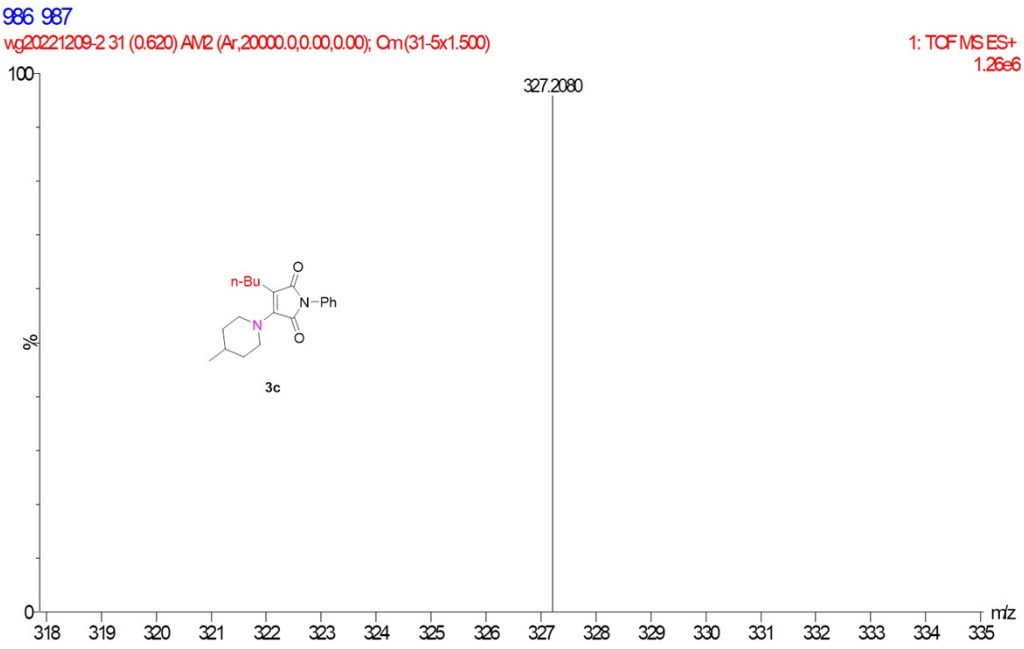
^{13}C NMR (100 MHz, $CDCl_3$)

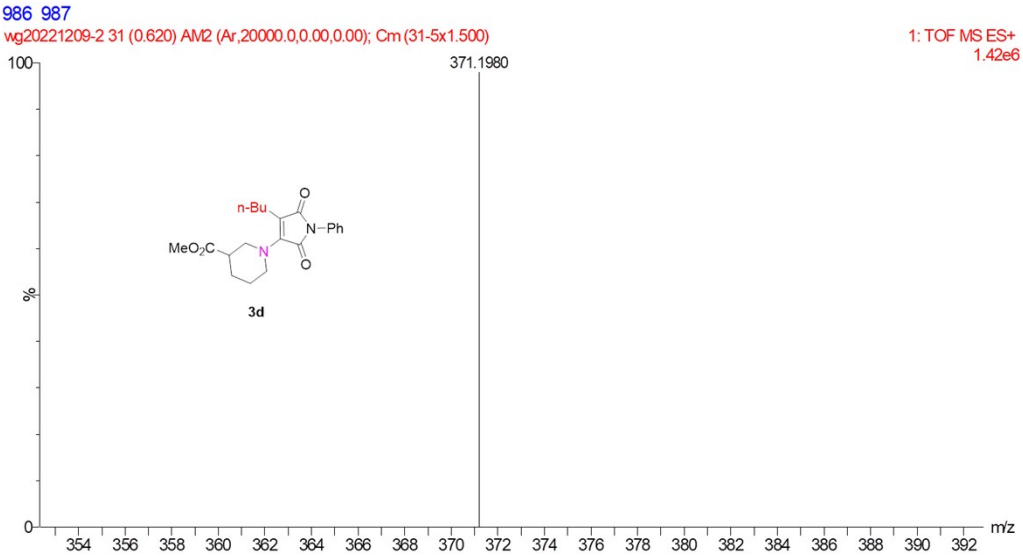


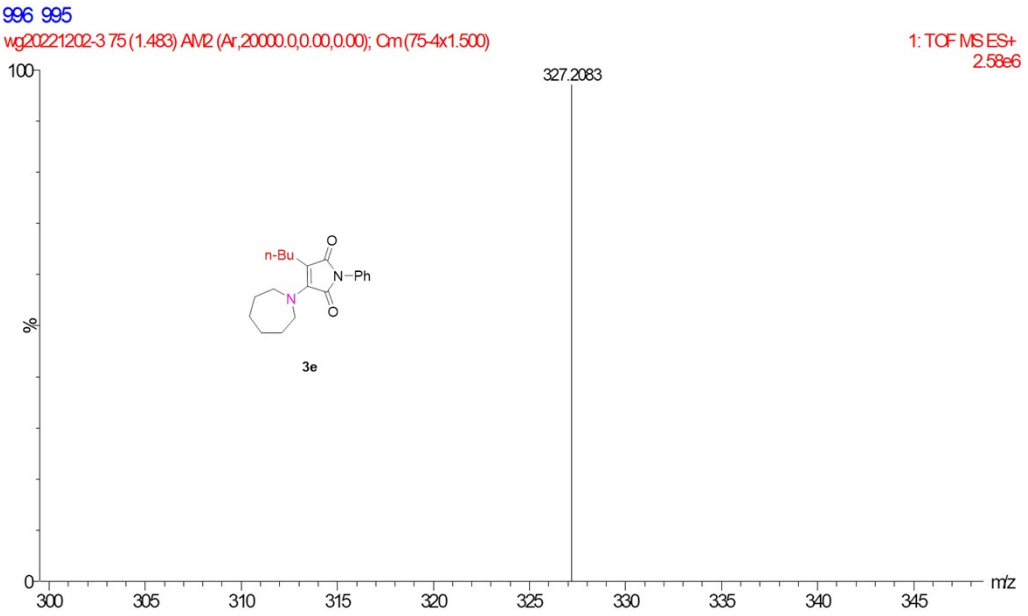
HRMS of Products

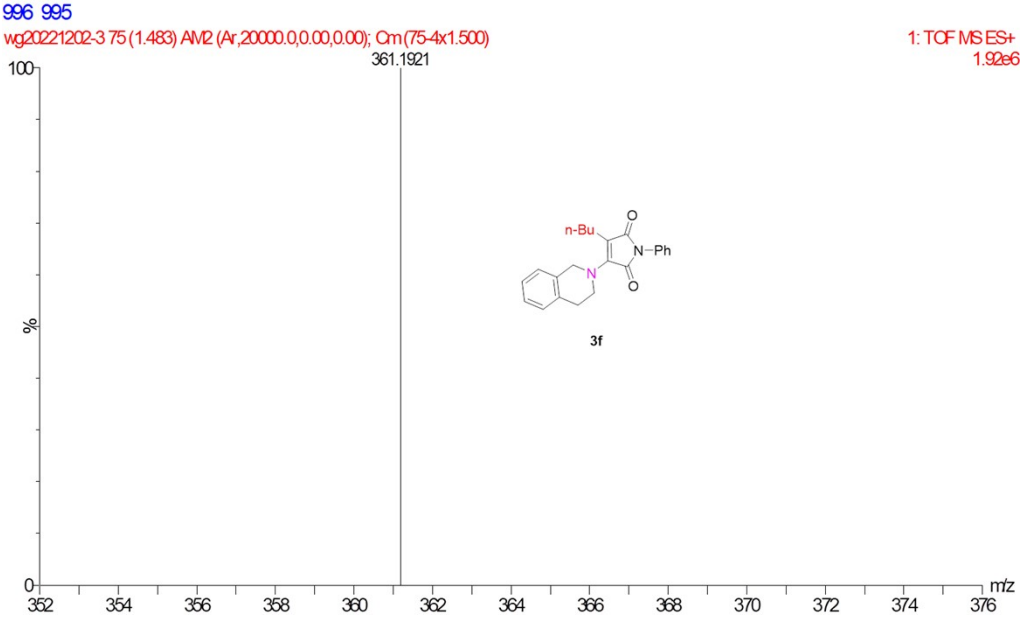


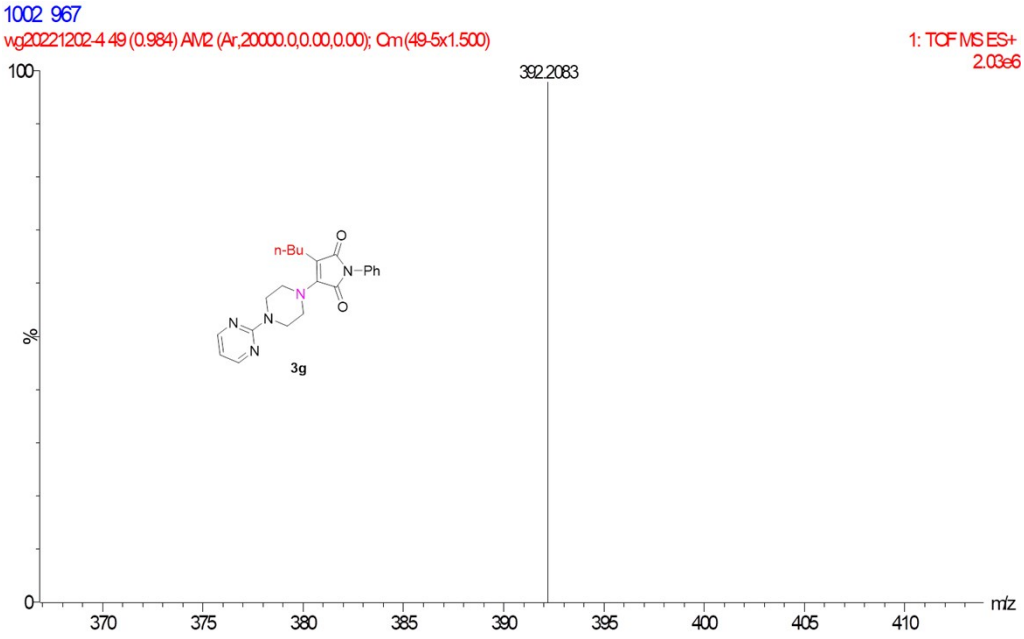


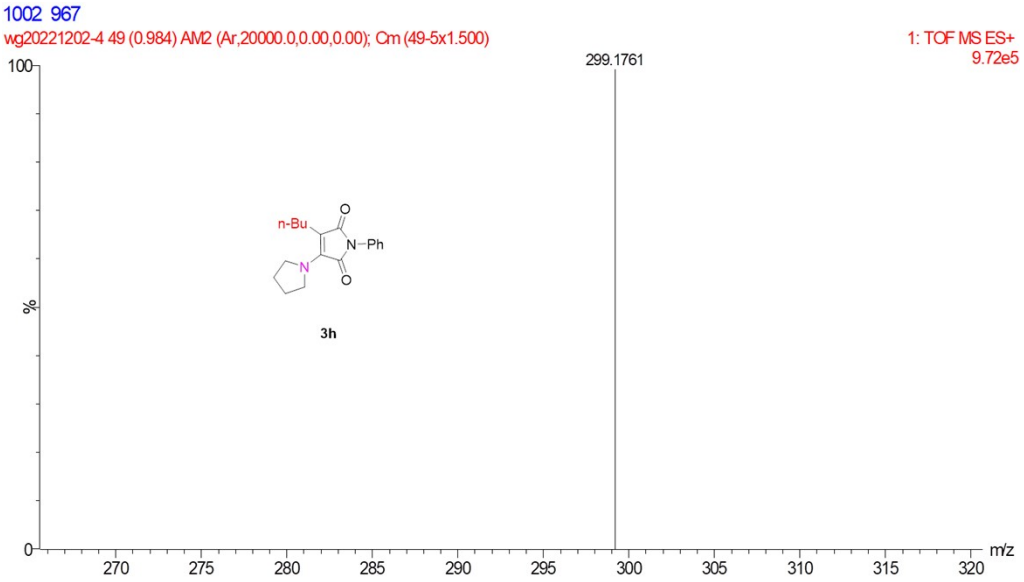

986 987
wg20221209-2 31 (0.620) AM2 (Ar,20000.0,0.00,0.00); Cm (31-5x1.500)
1: TOF MS ES+
1.26e6
327.2080
100
%
0
n-Bu
N–Ph
O
O
N
3c
318 319 320 321 322 323 324 325 326 327 328 329 330 331 332 333 334 335
m/z

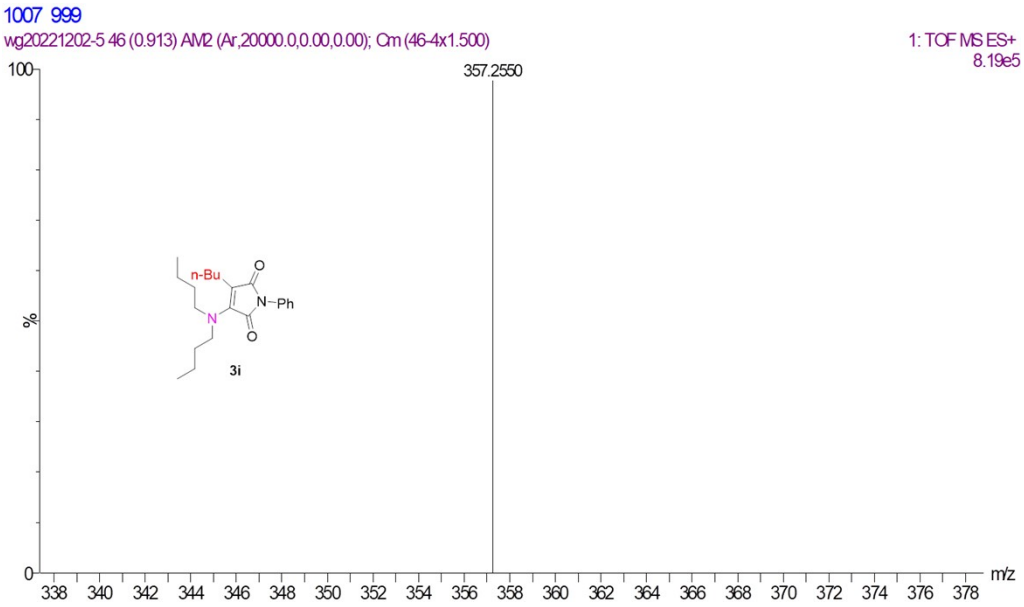

986 987
wg20221209-2 31 (0.620) AM2 (Ar,20000.0,0.00,0.00); Cm (31-5x1.500)
1: TOF MS ES+
1.42e6
371.1980
100
%
0
n-Bu
N–Ph
MeO2C
N
O
O
3d
354 356 358 360 362 364 366 368 370 372 374 376 378 380 382 384 386 388 390 392
m/z

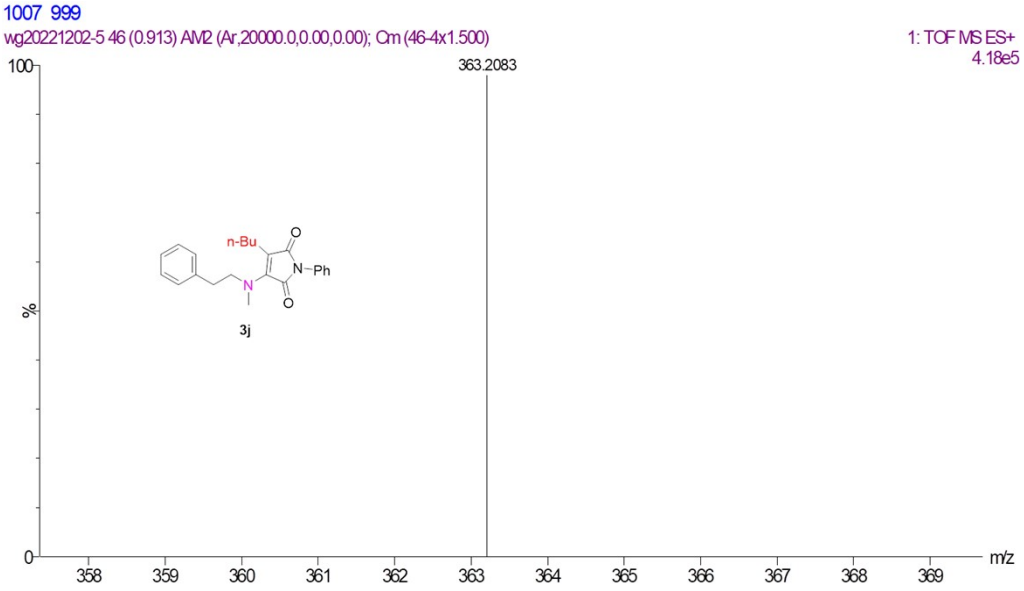

996 995
wg20221202-3 75 (1.483) AM2 (Ar,20000.0,0.00,0.00); Cm (75-4x1.500)
1: TOF MS ES+
2.58e6
327.2083
100
%
0
n-Bu
N
N–Ph
O
O
3e
300
305
310
315
320
325
330
335
340
345
m/z

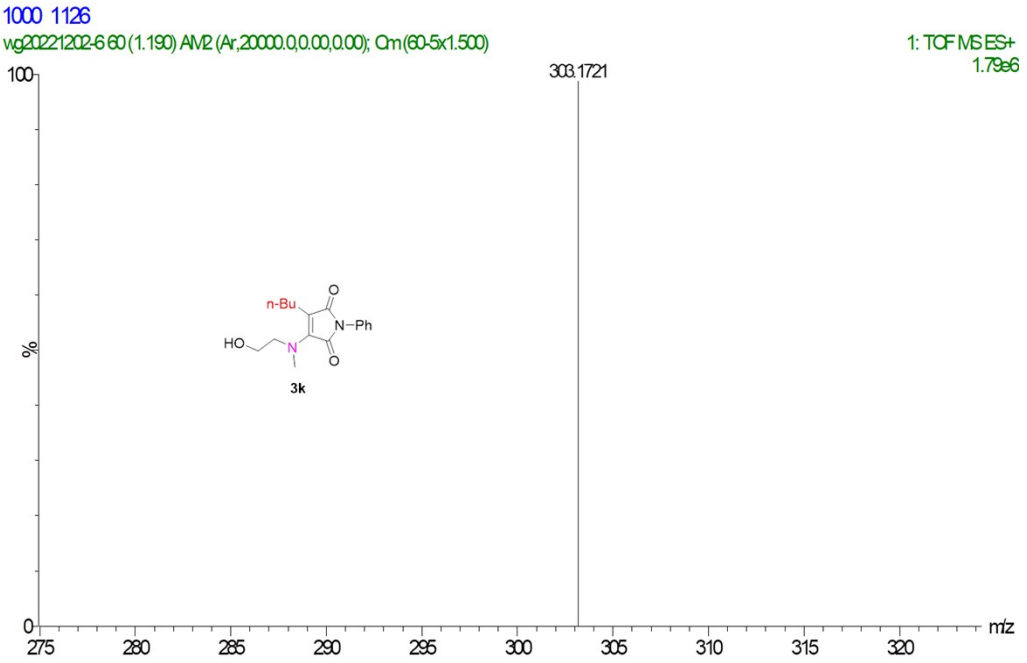

996 995
wg20221202-3 75 (1.483) AM2 (Ar,20000.0,0.00,0.00); Cm (75-4x1.500)
1: TOF MS ES+
1.92e6
361.1921
100
%
0
n-Bu
N
N–Ph
O
O
3f
352
354
356
358
360
362
364
366
368
370
372
374
376
m/z

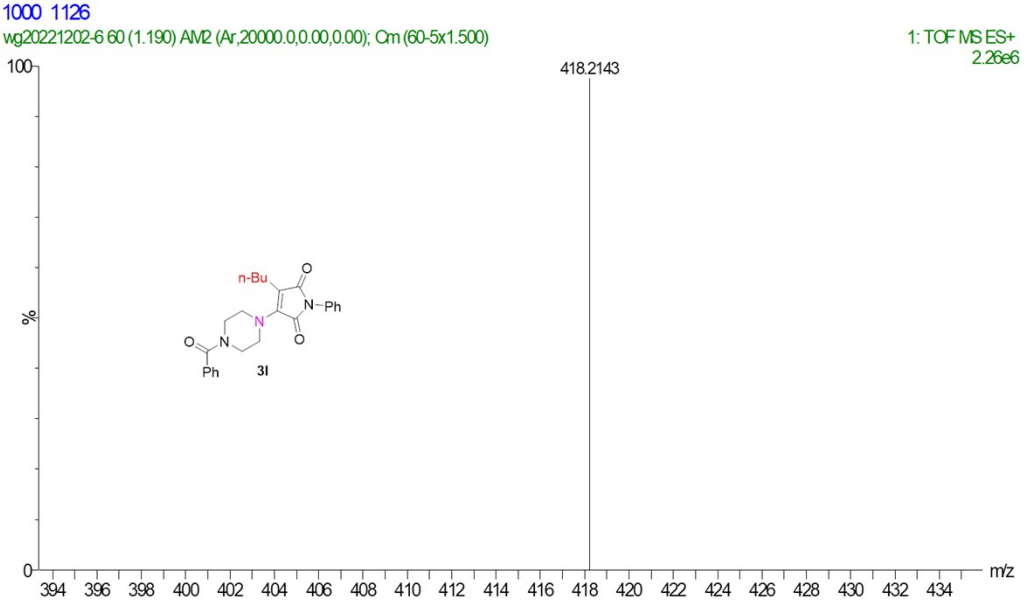

1002 967
wg20221202-4 49 (0.984) AM2 (Ar,20000.0,0.00,0.00); Cm (49-5x1.500)
1: TOF MS ES+
2.03e6
392.2083
100
%
0
n-Bu
N–Ph
O
O
N
N
N
3g
370
375
380
385
390
395
400
405
410
m/z

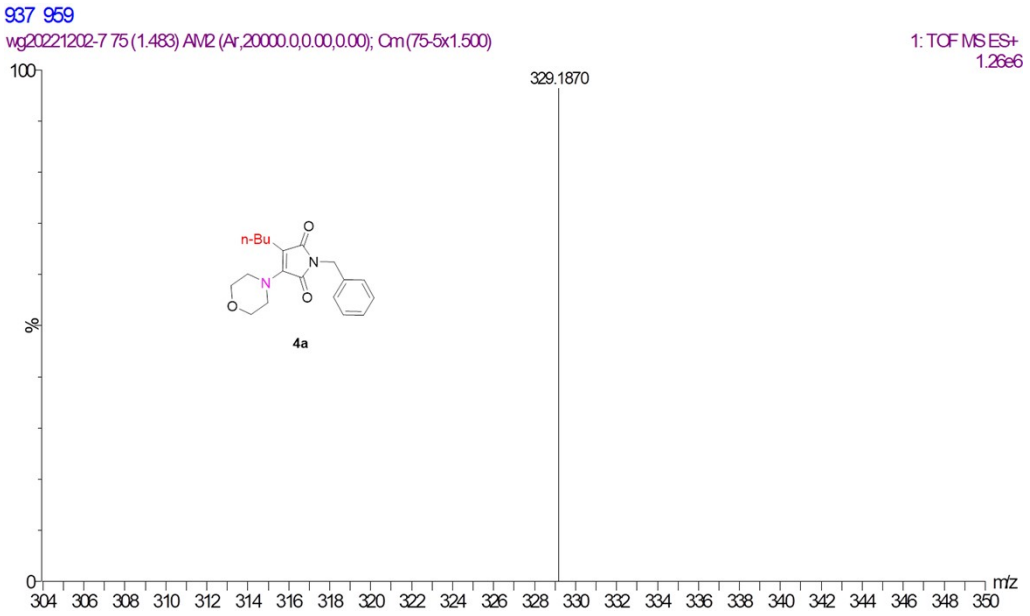

1002 967
wg20221202-4 49 (0.984) AM2 (Ar,20000.0,0.00,0.00); Cm (49-5x1.500)
1: TOF MS ES+
9.72e5
299.1761
100
%
0
n-Bu
N–Ph
O
O
N
3h
270
275
280
285
290
295
300
305
310
315
320
m/z

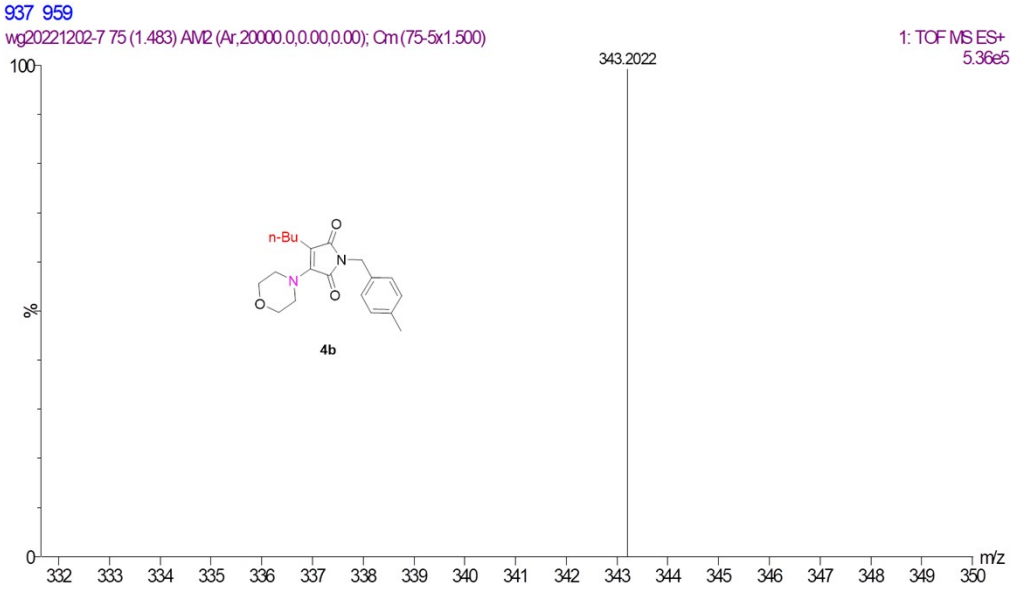

1007 999
wg20221202-5 46 (0.913) AM2 (Ar,20000.0,0.00,0.00); Cm (46-4x1.500)
1: TOF MS ES+
8.19e5
357.2550
100
%
0
n-Bu
N
N–Ph
O
O
3i
338 340 342 344 346 348 350 352 354 356 358 360 362 364 366 368 370 372 374 376 378
m/z

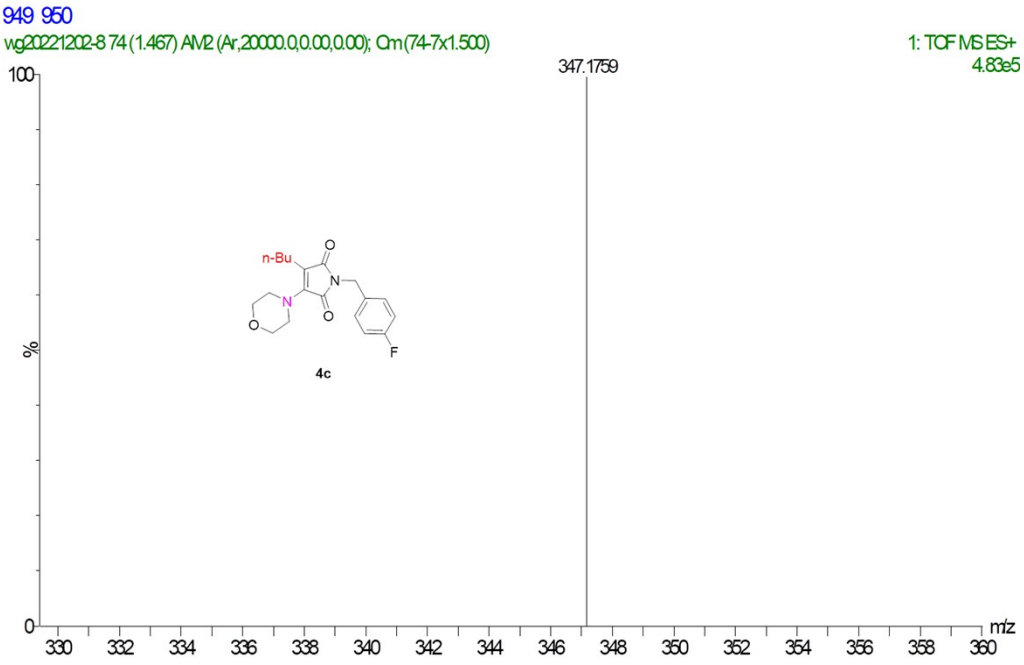

1007 999
wg20221202-5 46 (0.913) AM2 (Ar,20000.0,0.00,0.00); Cm (46-4x1.500)
1: TOF MS ES+
4.18e5
363.2083
100
%
0
n-Bu
N
N–Ph
O
O
3j
358 359 360 361 362 363 364 365 366 367 368 369
m/z

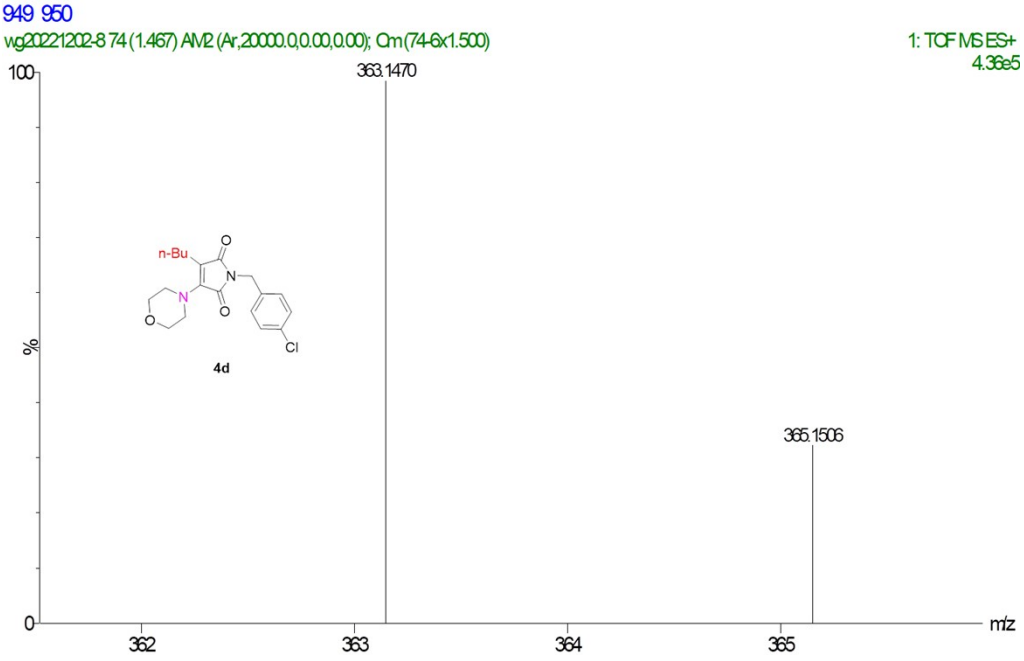

1000 1126
wg20221202-6 60 (1.190) AM2 (Ar,20000.0,0.00,0.00); Cm (60-5x1.500)
1: TOF MS ES+
1.79e6
303.1721
100
%
0
n-Bu
HO
N
N–Ph
O
O
O
3k
275
280
285
290
295
300
305
310
315
320
m/z


1000 1126
wg20221202-6 60 (1.190) AM2 (Ar,20000.0,0.00,0.00); Cm (60-5x1.500)
1: TOF MS ES+
2.26e6
418.2143
100
%
0
n-Bu
N
N–Ph
O
O
O
N
O
Ph
3l
394
396
398
400
402
404
406
408
410
412
414
416
418
420
422
424
426
428
430
432
434
m/z


937 959
wg20221202-7 75 (1.483) AM2 (Ar,20000.0,0.00,0.00); Cm (75-5x1.500)
1: TOF MS ES+
1.26e6
329.1870
100
%
0
n-Bu
4a
304 306 308 310 312 314 316 318 320 322 324 326 328 330 332 334 336 338 340 342 344 346 348 350
m/z


937 959
wg20221202-7 75 (1.483) AM2 (Ar,20000.0,0.00,0.00); Cm (75-5x1.500)
1: TOF MS ES+
5.36e5
343.2022
100
%
0
n-Bu
4b
332 333 334 335 336 337 338 339 340 341 342 343 344 345 346 347 348 349 350
m/z

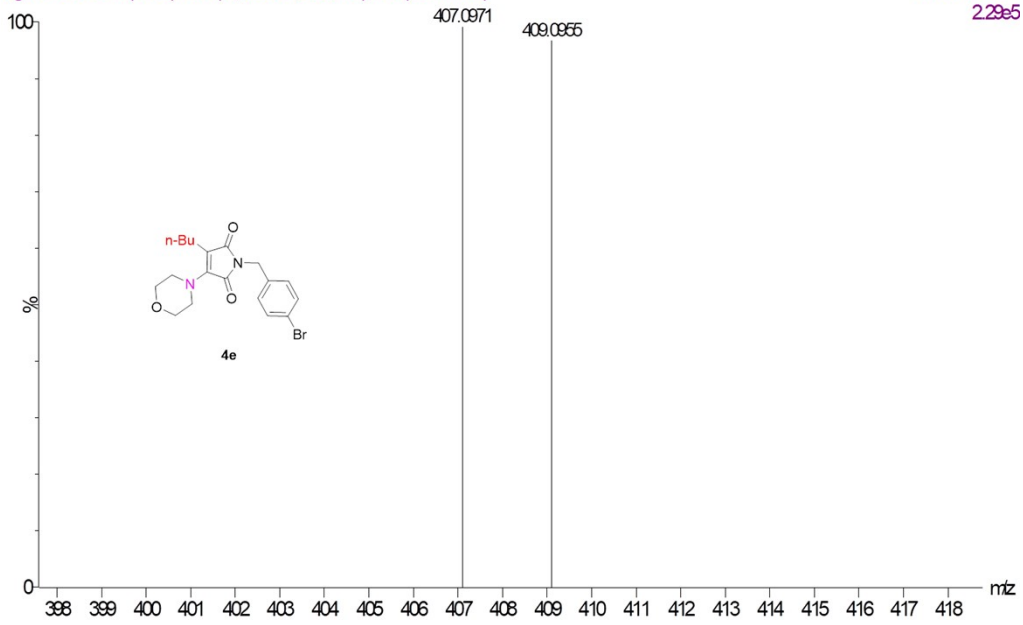

949 950
wg20221202-8 74 (1.467) AM2 (Ar,20000.0,0.00,0.00); Cm (74-7x1.500)
1: TOF MS ES+
4.83e5
347.1759
100
%
0
n-Bu
O
N
O
N
O
F
4c
330
332
334
336
338
340
342
344
346
348
350
352
354
356
358
360
m/z


949 950
wg20221202-8 74 (1.467) AM2 (Ar,20000.0,0.00,0.00); Cm (74-6x1.500)
1: TOF MS ES+
4.36e5
363.1470
365.1506
100
%
0
n-Bu
O
N
O
N
O
Cl
4d
362
363
364
365
m/z

964 956

wg20221202-9 74 (1.466) AM2 (Ar,20000.0,0.00,0.00); Cm(74-6x1.500)

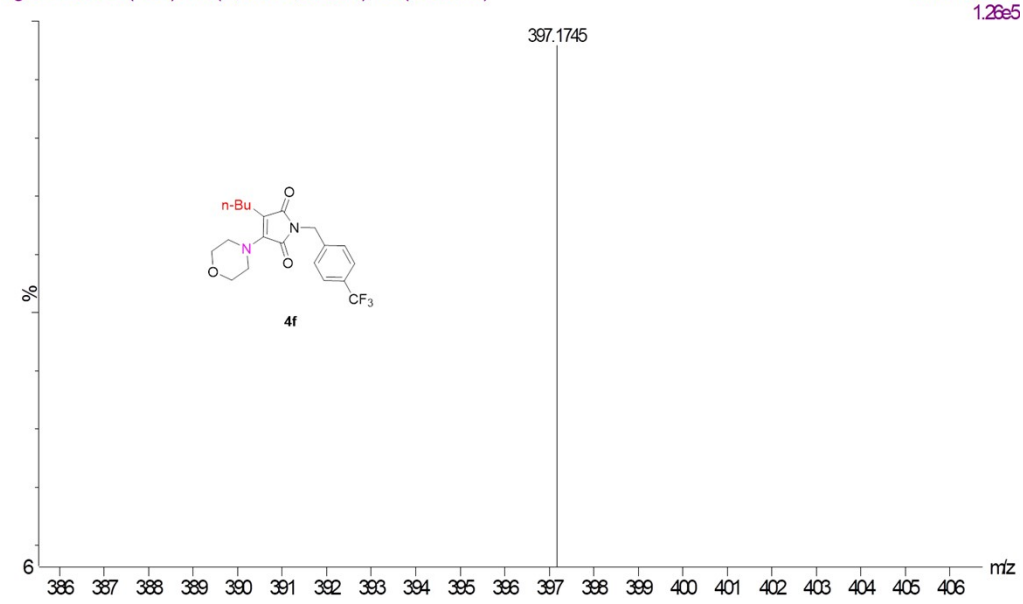
1: TOF MS ES+
2.29e5



964 956

wg20221202-9 74 (1.466) AM2 (Ar,20000.0,0.00,0.00); Cm(74-6x1.500)

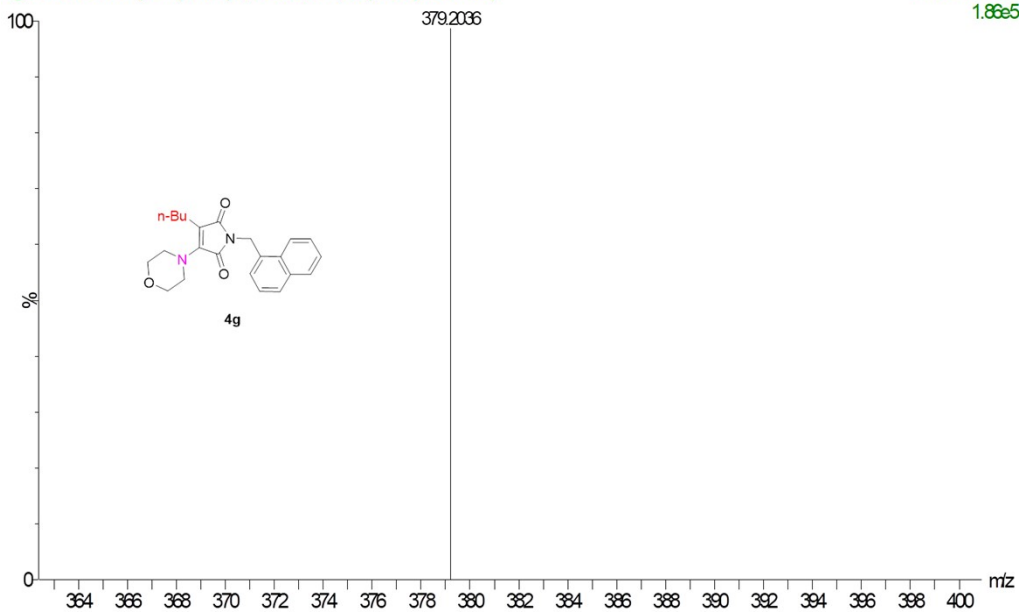
1: TOF MS ES+
1.26e5



963 958

wg20221209-10 37 (0.741) AM2 (Ar,20000.0,0.00,0.00); Cm (37-6x1.500)

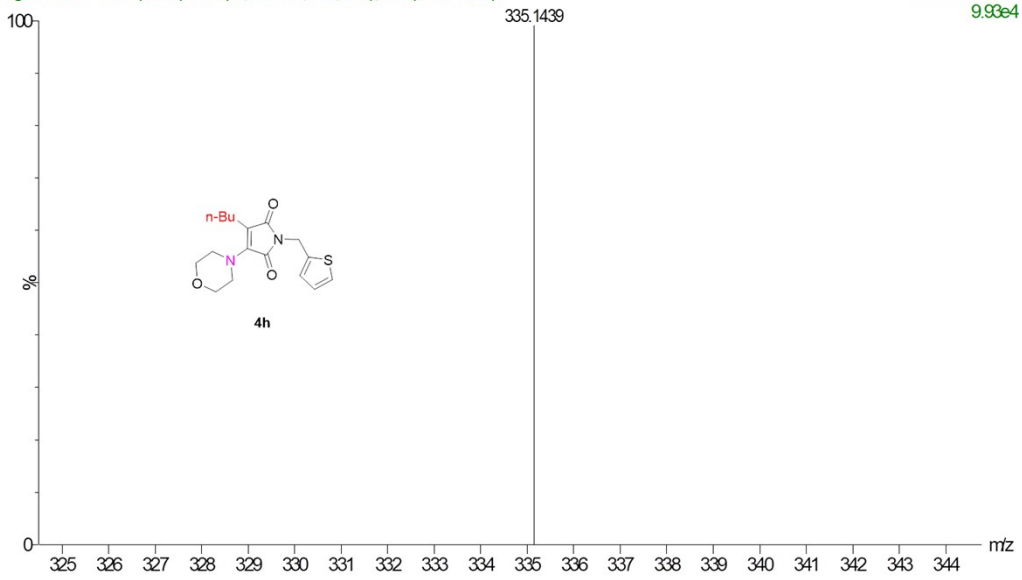
1: TOF MS ES+
1.86e5

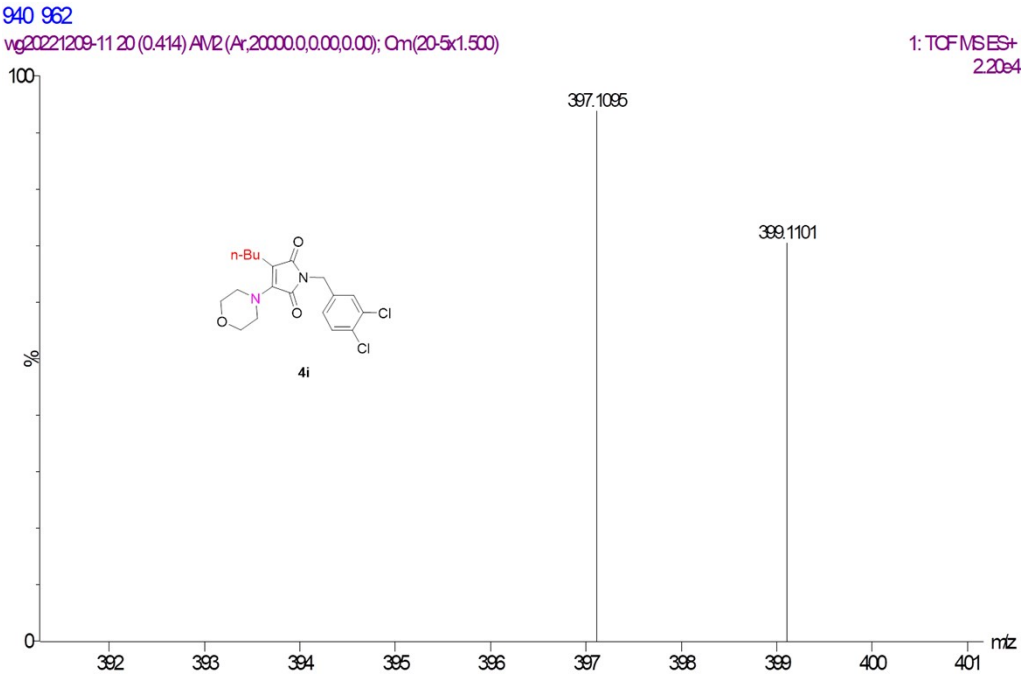


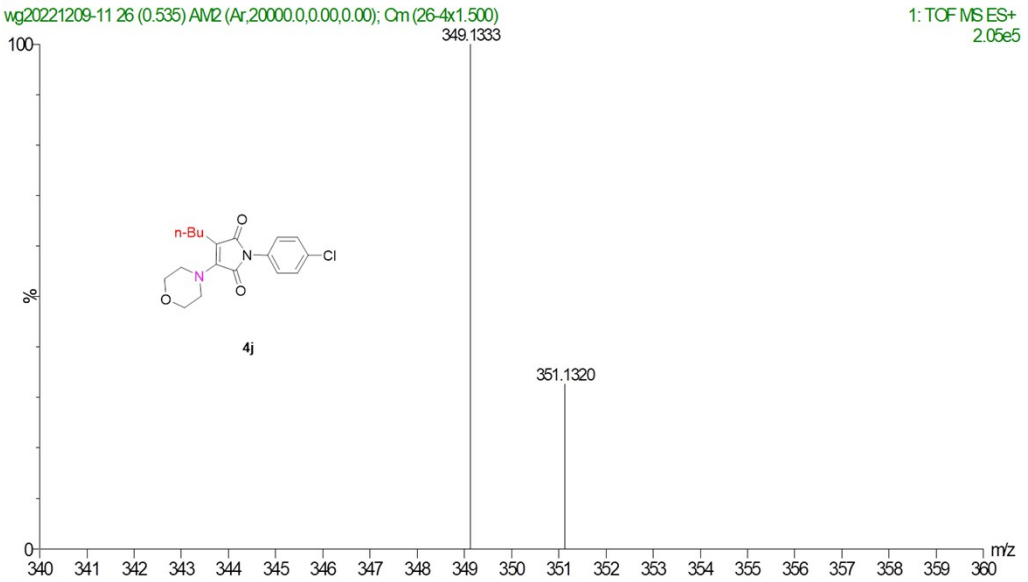
963 958

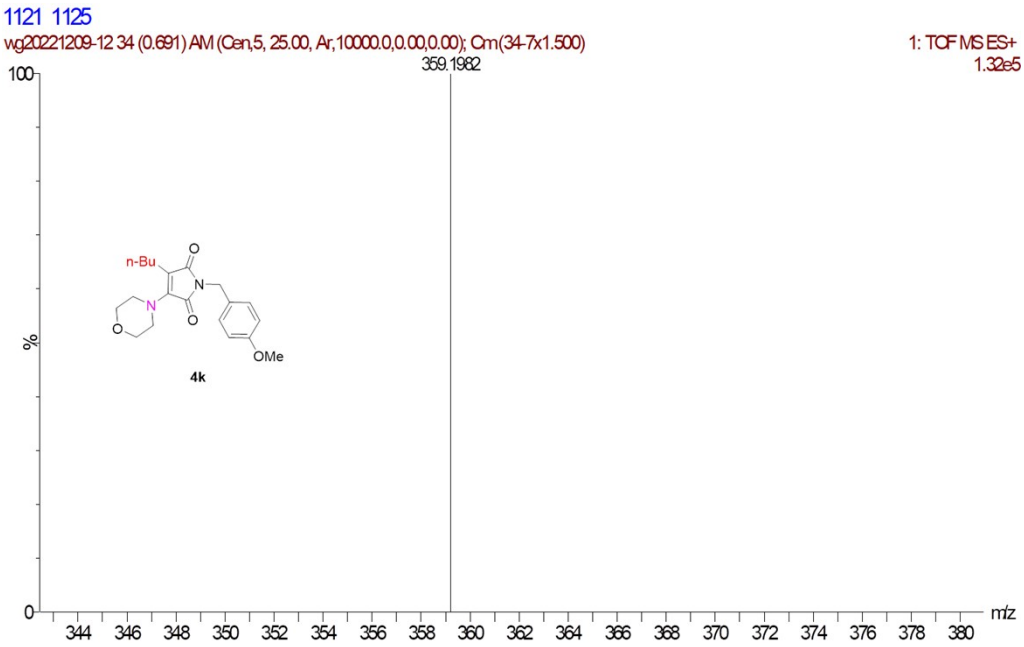
wg20221209-10 37 (0.741) AM2 (Ar,20000.0,0.00,0.00); Cm (37-6x1.500)

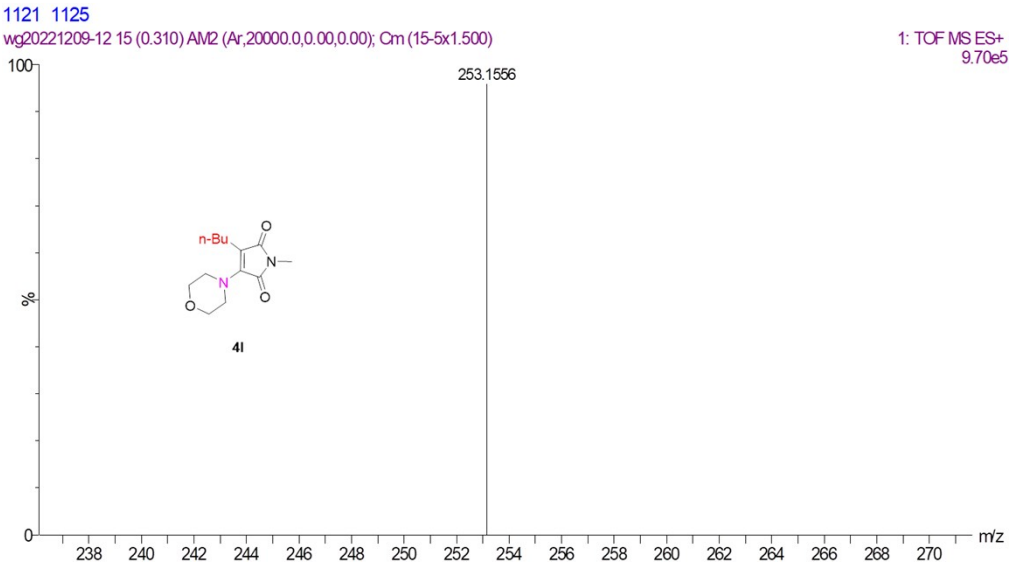
1: TOF MS ES+
9.93e4

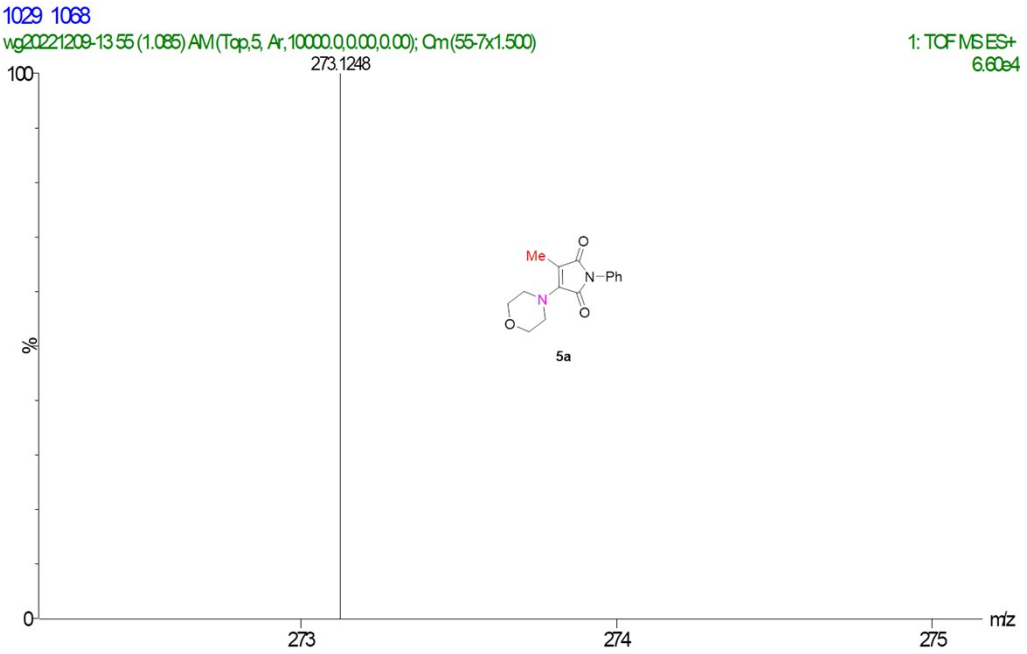


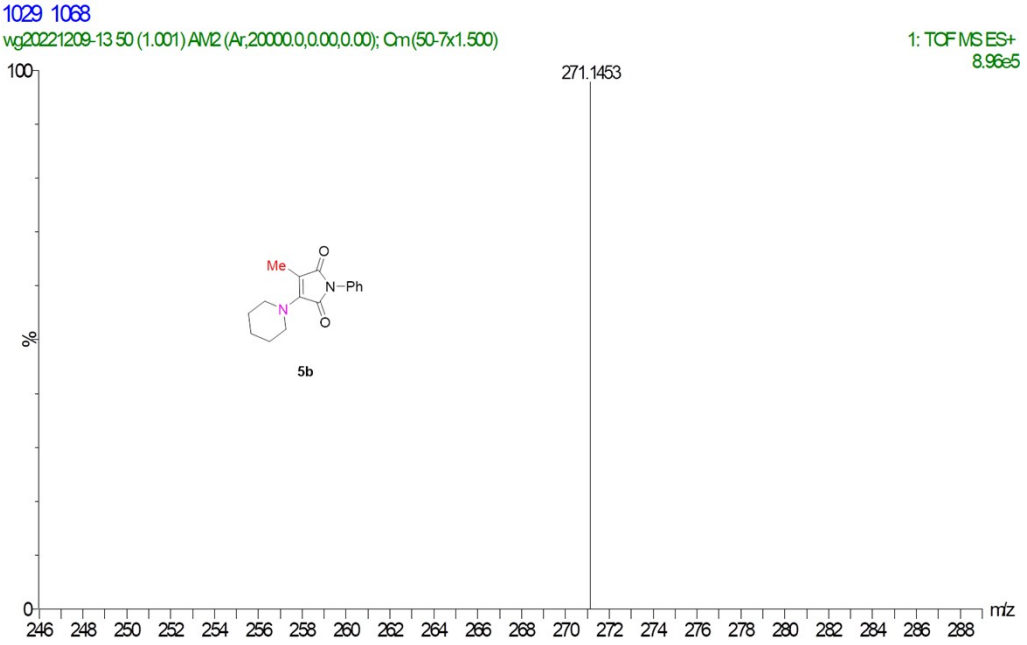

940 962
wg20221209-11 20 (0.414) AM2 (Ar,20000.0,0.00,0.00); Cm(20-5x1.500)
1: TOF MS ES+
2.20e4
100
%
0
397.1095
399.1101
n-Bu
O
N
O
N
O
Cl
Cl
4i
392
393
394
395
396
397
398
399
400
401
m/z

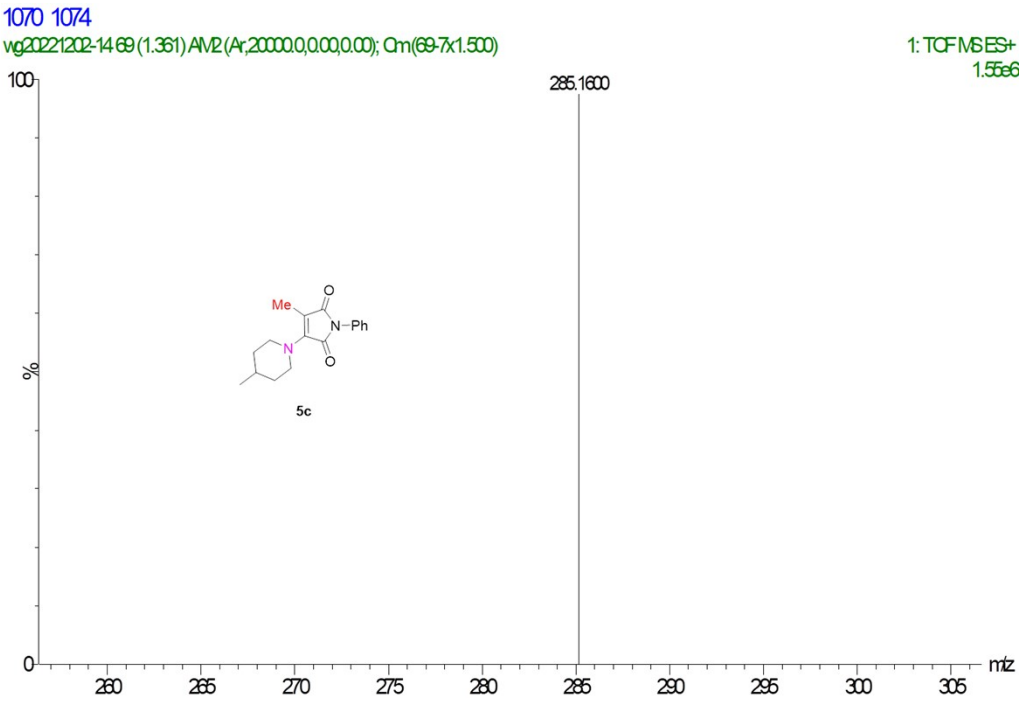

940 962
wg20221209-11 26 (0.535) AM2 (Ar,20000.0,0.00,0.00); Cm (26-4x1.500)
1: TOF MS ES+
2.05e5
100
%
0
349.1333
351.1320
n-Bu
O
N
O
N
O
Cl
4j
340
341
342
343
344
345
346
347
348
349
350
351
352
353
354
355
356
357
358
359
360
m/z

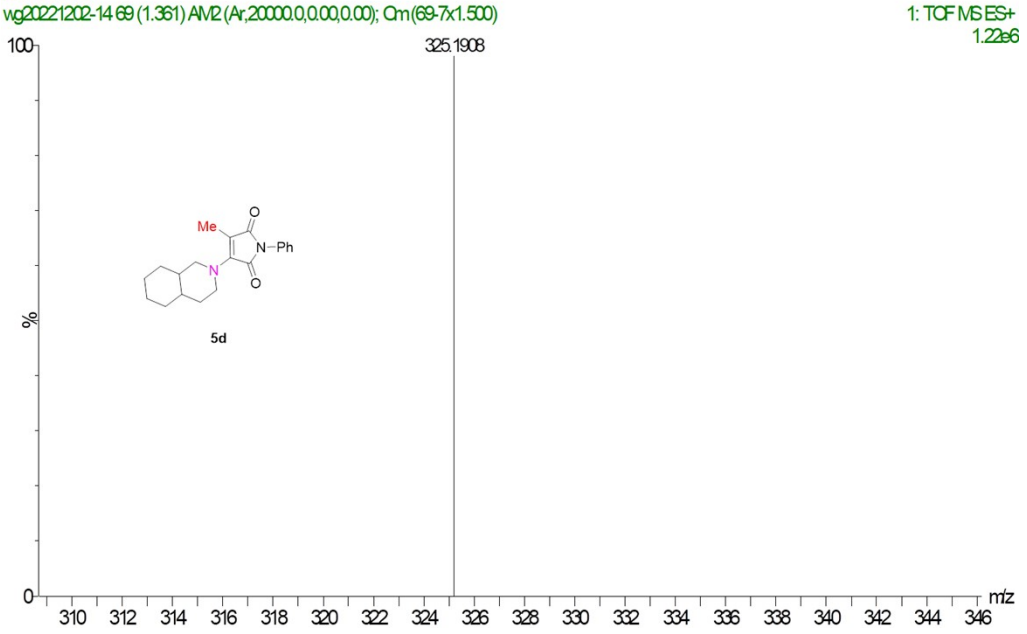

1121 1125
wg20221209-12 34 (0.691) AM (Cen,5, 25.00, Ar,10000.0,0.00,0.00); Cm (34-7x1.500)
1: TOF MS ES+
1.32e5
359.1982
100
%
0
n-Bu
N
O
O
N
O
OMe
4k
344 346 348 350 352 354 356 358 360 362 364 366 368 370 372 374 376 378 380
m/z

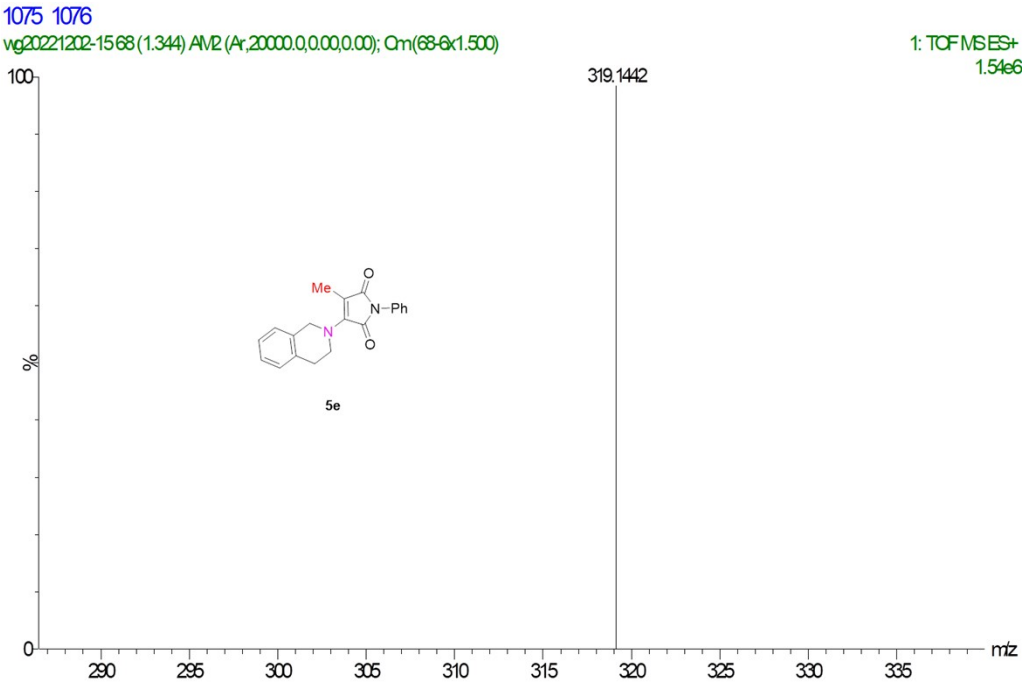

1121 1125
wg20221209-12 15 (0.310) AM2 (Ar,20000.0,0.00,0.00); Cm (15-5x1.500)
1: TOF MS ES+
9.70e5
253.1556
100
%
0
n-Bu
N
O
O
N
O
4l
238 240 242 244 246 248 250 252 254 256 258 260 262 264 266 268 270
m/z

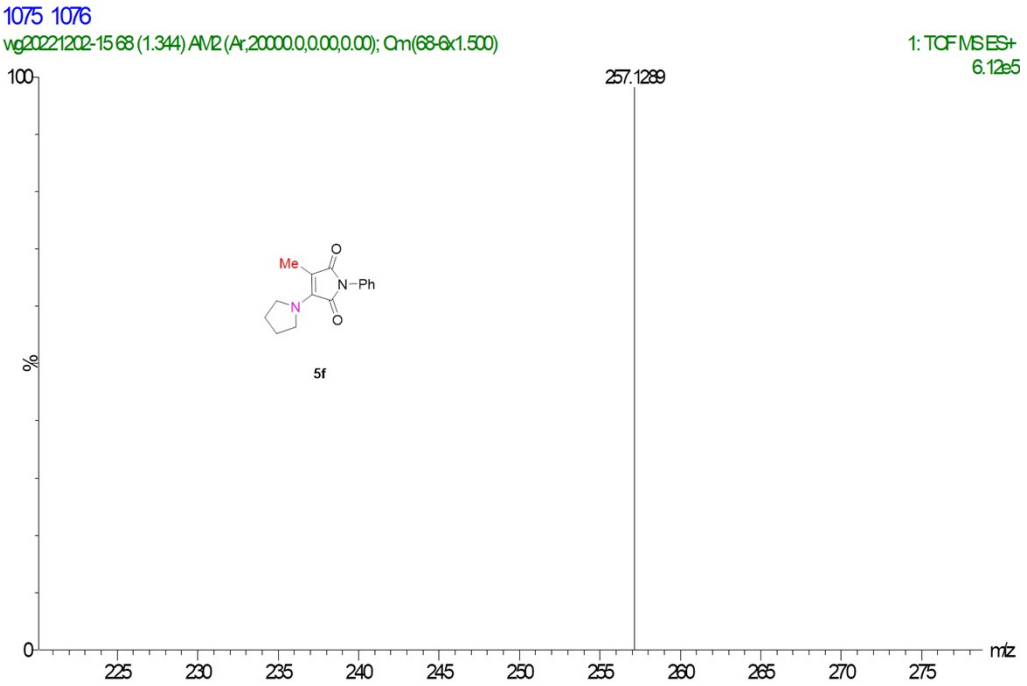

1029 1068
wg20221209-13 55 (1.085) AM (Top,5, Ar,10000.0,0.00,0.00); Cm (55-7x1.500)
1: TOF MS ES+
6.60e4
273.1248
100
%
0
Me
N
O
N–Ph
O
O
5a
273
274
275
m/z

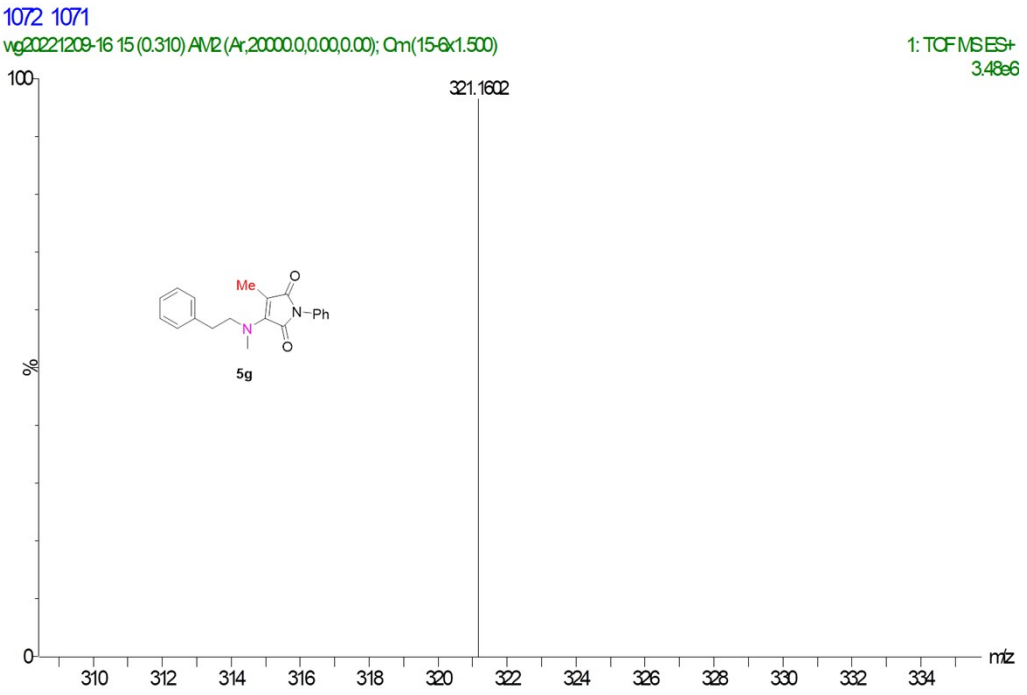

1029 1068
wg20221209-13 50 (1.001) AM2 (Ar,20000.0,0.00,0.00); Cm (50-7x1.500)
1: TOF MS ES+
8.96e5
271.1453
100
%
0
Me
N
O
N–Ph
O
5b
246 248 250 252 254 256 258 260 262 264 266 268 270 272 274 276 278 280 282 284 286 288
m/z

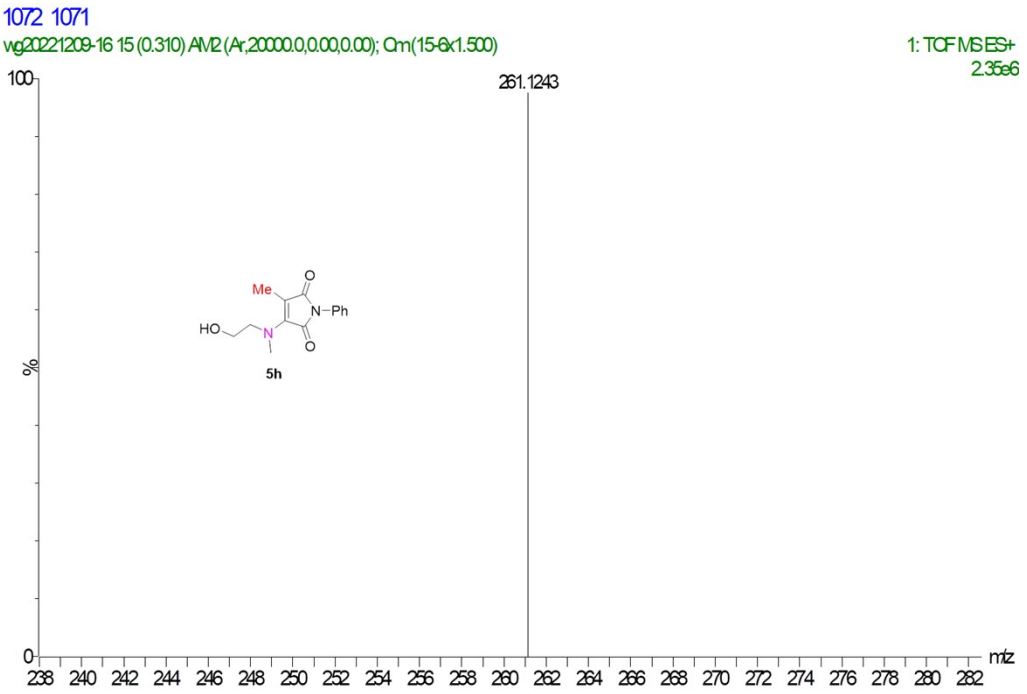

1070 1074
vg20221202-1469 (1.361) AM2 (Ar,20000.0,0.00,0.00); Cm(69-7x1.500)
1: TOF MS ES+
1.55e6
285.1600
100
%
0
Me
N
N–Ph
O
O
O
5c
260
265
270
275
280
285
290
295
300
305
m/z

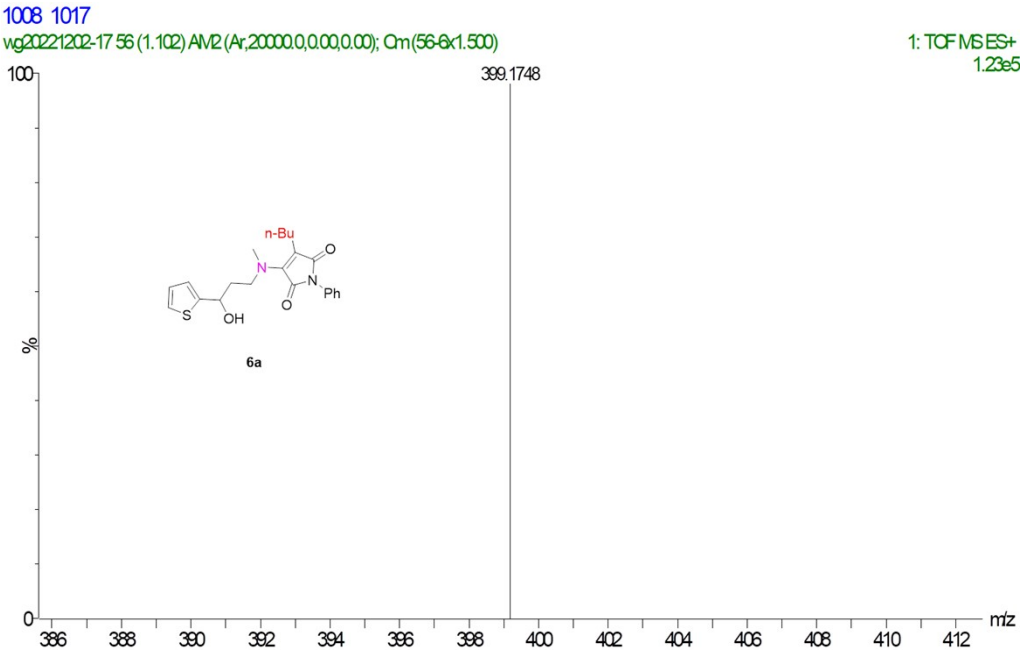

1070 1074
vg20221202-1469 (1.361) AM2 (Ar,20000.0,0.00,0.00); Cm(69-7x1.500)
1: TOF MS ES+
1.22e6
325.1908
100
%
0
Me
N
N–Ph
O
O
O
5d
310
312
314
316
318
320
322
324
326
328
330
332
334
336
338
340
342
344
346
m/z

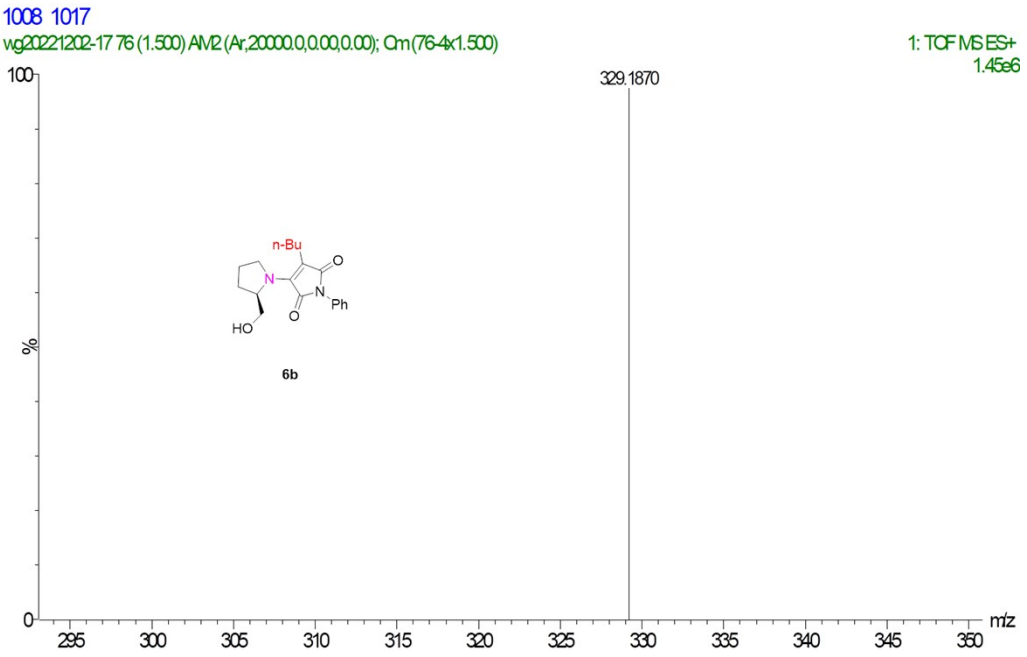

1075 1076
wg20221202-1568 (1.344) AM2 (Ar,20000.0,0.00,0.00); Cm(68-6x1.500)
1: TOF MS ES+
1.54e6
319.1442
100
%
0
290
295
300
305
310
315
320
325
330
335
m/z
Me
N–Ph
O
O
N
5e

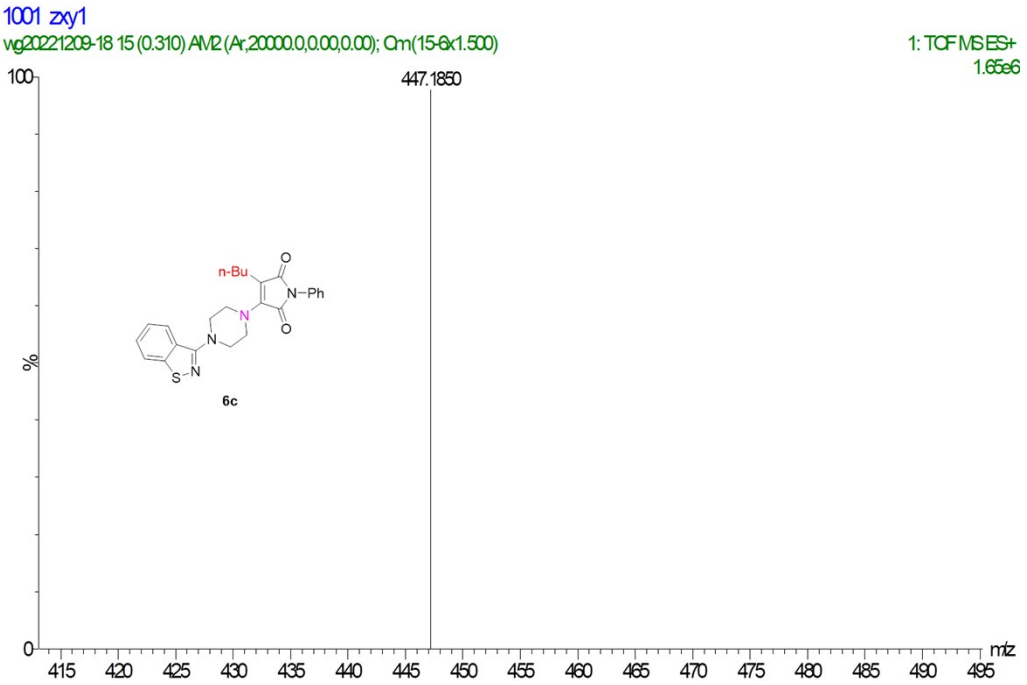

1075 1076
wg20221202-1568 (1.344) AM2 (Ar,20000.0,0.00,0.00); Cm(68-6x1.500)
1: TOF MS ES+
6.12e5
257.1289
100
%
0
225
230
235
240
245
250
255
260
265
270
275
m/z
Me
N–Ph
O
O
N
5f


1072 1071
vg20221209-16 15 (0.310) AM2 (Ar,20000.0,0.00,0.00); Cm(15-6x1.500)
1: TOF MS ES+
3.48e6
321.1602
100
%
0
Me
O
N–Ph
N
O
5g
310 312 314 316 318 320 322 324 326 328 330 332 334
m/z


1072 1071
vg20221209-16 15 (0.310) AM2 (Ar,20000.0,0.00,0.00); Cm(15-6x1.500)
1: TOF MS ES+
2.35e6
261.1243
100
%
0
Me
O
N–Ph
HO
N
O
5h
238 240 242 244 246 248 250 252 254 256 258 260 262 264 266 268 270 272 274 276 278 280 282
m/z


1008 1017
wg20221202-17 56 (1.102) AM2 (Ar,20000.0,0.00,0.00); Cm (56-6x1.500)
1: TOF MS ES+
1.23e5
399.1748
n-Bu
N
O
N
Ph
O
S
OH
6a
%
m/z
100
0
386
388
390
392
394
396
398
400
402
404
406
408
410
412


1008 1017
wg20221202-17 76 (1.500) AM2 (Ar,20000.0,0.00,0.00); Cm (76-4x1.500)
1: TOF MS ES+
1.45e6
329.1870
n-Bu
N
O
N
Ph
O
HO
6b
%
m/z
100
0
295
300
305
310
315
320
325
330
335
340
345
350


1001 zxy1
wg20221209-18 15 (0.310) AM2 (Ar,20000.0,0.00,0.00); Cm (15-6x1.500)
1: TOF MS ES+
1.65e6
100
%
0
447.1850
n-Bu
N–Ph
O
O
N
N
S
N
6c
415
420
425
430
435
440
445
450
455
460
465
470
475
480
485
490
495
m/z