Electronic Supplementary Material (ESI) for Green Chemistry.

Electrochemical Oxidative [4 + 2] Annulation of Tertiary Anilines and Alkenes for the Synthesis of Tetrahydroquinolines

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Table of Contents

General information

All glassware was oven dried at 110 ℃ for hours and cooled down under vacuum. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. The instrument for electrolysis is dual display potentiostat (DJS-292B) (made in China). Both of the anode electrode and cathode electrode are platinum electrodes (1.5 cm×1.5 cm×0.3 mm). Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (bp. 60-90 ℃). Gas chromatographic analyses were performed on SHIMADZU GC-2014 gas chromatography instrument with a FID detector and biphenyl was added as internal standard. GC-MS spectra were recorded on Varian GC MS 3900-2100T or SHIMADZU GC MS-2010. ^{1}H and ^{13}C NMR data were recorded with Bruker Advance III (400 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. All chemical shifts are reported relative to tetramethylsilane and d-solvent peaks (77.00 ppm, chloroform), respectively. EPR spectra were recorded on a Bruker X-band A200 spectrometer.

Experimental procedure

Typical procedure for preparation of enamines: The mixture of ketoxime (5 mmol), acetic anhydride (10 mmol, 1.02 g), $NaHSO_3$ (1.5 mmol, 780.5 mg) and CuI (10 mol%, 95.5 mg) was stirred in 1,2-dichloroethane (DCE, 50 mL) at 120 °C under Ar. After completion of the reaction (detected by TLC), the reaction mixture was cooled to room temperature, diluted with EtOAc (25 mL) and washed with NaOH (2N, 20 mL) and brine (20 mL). The organic layers were dried over anhydrous Na_2SO_4 and evaporated in vacuo. The desired enamines were obtained after purification by flash chromatography on silica gel with hexanes/ethyl acetate as the eluent.

General procedure for the synthesis of tetrahydroquinolines: In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, enamine (0.25 mmol), tertiary aniline (1.25 mmol), $^{n}Bu_4NBF_4$ (65.8 mg, 0.2 mmol) and CH_3CN/AcOH (4 mL/1 mL) were combined and added. The bottle was equipped with platinum electrodes (1.5 cm×1.5 cm×0.3 mm) as both the anode and cathode and was then charged with nitrogen. The reaction mixture was stirred and electrolyzed at a constant current of 3 mA under room temperature for 6 h. When the reaction finished, the solvent was removed with a rotary evaporator. The pure product was obtained by flash chromatography on silica gel using petroleum ether and ethyl acetate as the eluent.

Procedure for gram scale synthesis: In an oven-dried undivided three-necked bottle (250 mL) equipped with a stir bar, enamine (6.0 mmol), tertiary aniline (30.0 mmol), $^{n}Bu_4NBF_4$ (789.6 mg, 2.4 mmol) and CH_3CN/AcOH (96 mL/24 mL) were combined and added to a bottle. The bottle was equipped with platinum electrodes (1.5 cm×1.5 cm×0.3 mm) as both the anode and cathode and was then charged with nitrogen. The reaction mixture was stirred and electrolyzed at a constant current of 6 mA under room temperature for 72 h. When the reaction was finished, the solvent was removed with a rotary evaporator. The pure product was obtained by flash column chromatography on silica gel using petroleum ether and ethyl acetate as the eluent.

General procedure for cyclic voltammetry (CV): Cyclic voltammetry was performed in a three-electrode cell connected to a schlenk line under nitrogen at room temperature. The working electrode was a platinum disk electrode, the counter electrode a platinum wire. The reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution, and separated from reaction by a salt bridge. 8 mL/ 2 mL of CH_3CN/AcOH containing 0.1 M $^{n}Bu_4NBF_4$ were poured into the

electrochemical cell in all experiments. The scan rate is 0.1 V/s, ranging from 0 V to 2.5 V.

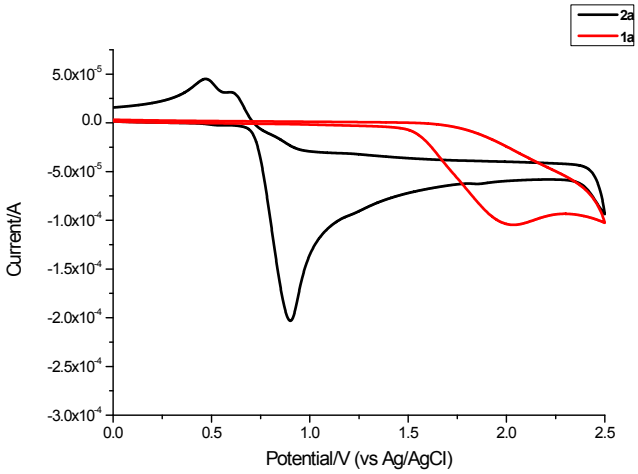
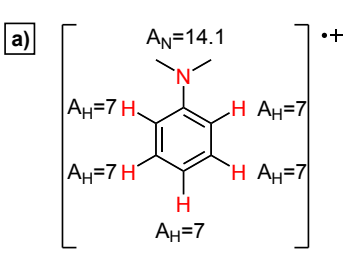
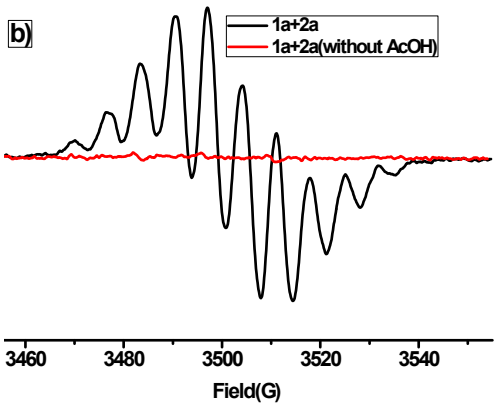


Figure S1. Cyclic voltammetry of **1a**(*N*-(1-phenylvinyl)acetamide) and **2a**(*N,N*-dimethylaniline) in CH_3CN/AcOH with $^nBu_4NBF_4$ (0.1 M) under nitrogen at a platinum-disk electrode at a scan rate of v=0.1 V/s.

EPR experiments: Under constant current conditions, a dried three-necked flask equipped with a stir bar was loaded with **1a** (0.25 mmol), **2a** (1.50 mmol), $^nBu_4NBF_4$ (0.20 mmol) in 5.0 mL CH_3CN or CH_3CN/AcOH (4 mL / 1 mL) was stirred under an N_2 atmosphere at 25 ºC. After 3 h, the solution sample was taken out into a small tube and analyzed by EPR. EPR spectra was recorded at room temperature on EPR spectrometer operated at 9.824452 GHz. Typical spectrometer parameters are shown as follows, scan range: 100.00 G; center field set: 3505.61 G. In our fitting result (g = 2.0038, A_N = 14.1 G, A_H = 7 G, A_H = 7 G, A_H = 7 G, A_H = 7 G and A_H = 7 G), there are five H-atom and one N-atom make an influence on conjugation radical system. A proposed structure was showed in Figure S2a. The difference between fitting results and experiment results may be caused by linear distortion.





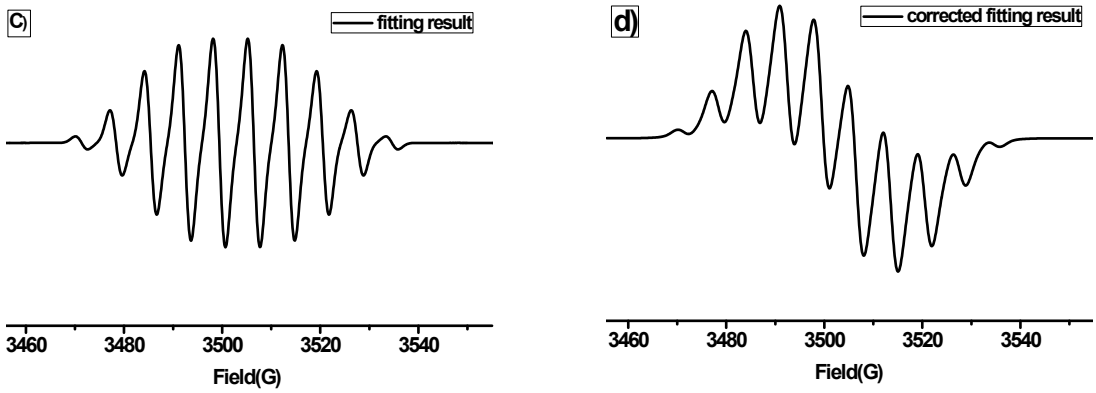
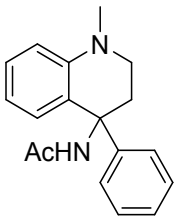
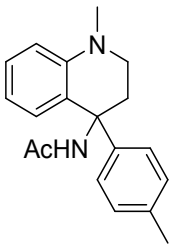


Figure S2. EPR measurements of a solution in CH_3CN/AcOH (4.0 mL/1.0 mL) or CH_3CN (5.0 mL) of $^nBu_4NBF_4$, **1a**, **2a** under constant current conditions for 3h.

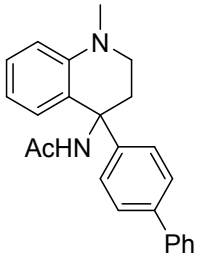
Detail descriptions for products



***N*-(1-methyl-4-phenyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3aa):[1]** yellow solid was obtained in 72% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 2H), 7.26 – 7.20 (m, 3H), 7.16 – 7.10 (m, 1H), 6.64 – 6.57 (m, 2H), 6.52 – 6.45 (m, 1H), 5.83 (s, 1H), 3.42 – 3.29 (m, 2H), 3.14 – 3.07 (m, 1H), 2.93 (s, 3H), 2.38 – 2.29 (m, 1H), 1.98 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$)δ168.85, 145.84, 144.16, 128.93, 128.72, 128.02, 126.92, 126.72, 125.85, 115.65, 111.01, 60.92, 47.27, 38.84, 31.46, 24.31.

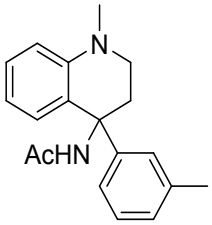


***N*-(1-methyl-4-(*p*-tolyl)-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ab):[1]** yellow solid was obtained in 60% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 – 7.10 (m, 5H), 6.66 – 6.59 (m, 2H), 6.53 – 6.46 (m, 1H), 5.76 (s, 1H), 3.42 – 3.29 (m, 2H), 3.14 – 3.07 (m, 1H), 2.93 (s, 3H), 2.37 – 2.28 (m, 4H), 2.01 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.81, 145.86, 141.23, 136.34, 128.91, 128.79, 128.64, 126.82, 125.97, 115.66, 111.01, 60.74, 47.35, 38.86, 31.46, 24.40, 20.87.

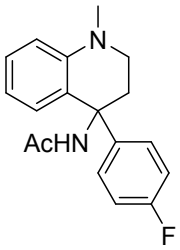


***N*-(4-([1,1'-biphenyl]-4-yl)-1-methyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ac):[1]** yellow solid was obtained in 60% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 – 7.52 (m, 4H), 7.46 – 7.38 (m, 2H), 7.37 – 7.29 (m, 3H), 7.21 – 7.12 (m, 1H), 6.72 – 6.61 (m, 2H), 6.56 – 6.46 (m, 1H), 5.75 (s, 1H), 3.48 – 3.35 (m, 2H), 3.24 – 3.13 (m, 1H), 2.97 (s, 3H), 2.46 – 2.35 (m, 1H), 2.07 (s,

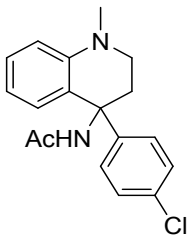
3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ168.93, 145.92, 143.18, 140.68, 139.61, 129.18, 128.95, 128.68, 127.43, 127.15, 127.01, 126.86, 125.97, 115.81, 111.17, 60.93, 47.37, 38.95, 31.44, 24.53.



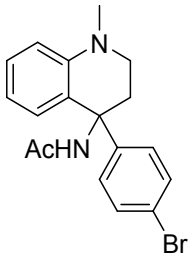
***N*-(1-methyl-4-(*m*-tolyl)-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ad):** yellow solid was obtained in 44% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.18 (m, 1H), 7.18 – 7.12 (m, 1H), 7.09 – 7.02 (m, 3H), 6.66 – 6.59 (m, 2H), 6.53 – 6.47 (m, 1H), 5.71 (s, 1H), 3.45 – 3.32 (m, 2H), 3.17 – 3.10 (m, 1H), 2.95 (s, 3H), 2.40 – 2.31 (m, 4H), 2.04 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.80, 145.91, 144.12, 137.67, 129.01, 128.79, 127.96, 127.67, 127.51, 126.08, 124.23, 115.75, 111.07, 61.02, 47.42, 38.93, 31.39, 24.52, 21.69. HRMS (ESI) calculated for $C_{19}H_{23}N_2O$ $[M+H]^+$: 295.1805; found: 295.1801.



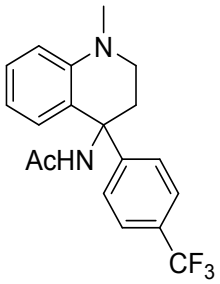
***N*-(4-(4-fluorophenyl)-1-methyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ae):[1]** white solid was obtained in 65% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.18 (m, 2H), 7.18 – 7.12 (m, 1H), 7.03 – 6.95 (m, 2H), 6.63 (dd, J = 8.4, 1.2 Hz, 1H), 6.59 – 6.54 (m, 1H), 6.52 – 6.47 (m, 1H), 5.77 (s, 1H), 3.40 – 3.31 (m, 2H), 3.18 – 3.09 (m, 1H), 2.95 (s, 3H), 2.37 – 2.27 (m, 1H), 2.02 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 168.91, 161.56(d, J_{CF} = 243.9 Hz), 145.80, 139.78(d, J_{CF} = 3.2 Hz), 129.21, 128.76, 128.61(d, J_{CF} = 8.0 Hz), 125.80, 115.75, 114.85(d, J_{CF} = 21.2 Hz), 111.17, 60.60, 47.24, 38.86, 31.52, 24.40. ^{19}F NMR (377 MHz, $CDCl_3$) δ -116.42.



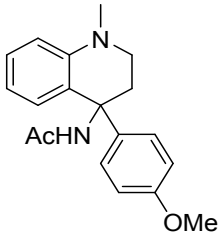
***N*-(4-(4-chlorophenyl)-1-methyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3af):[1]** white solid was obtained in 67% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.26 (m, 2H), 7.22 – 7.12 (m, 3H), 6.63 (dd, *J* = 8.4, 1.2 Hz, 1H), 6.57 – 6.52 (m, 1H), 6.52 – 6.46 (m, 1H), 5.72 (s, 1H), 3.42 – 3.31 (m, 2H), 3.19 – 3.12 (m, 1H), 2.95 (s, 3H), 2.38 – 2.27 (m, 1H), 2.04 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 168.93, 145.80, 142.61, 132.55, 129.32, 128.88, 128.48, 128.21, 125.60, 115.82, 111.23, 60.67, 47.20, 38.88, 31.39, 24.41.



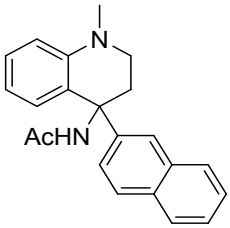
***N*-(4-(4-bromophenyl)-1-methyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ag):[1]** yellow solid was obtained in 64% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.39 (m, 2H), 7.19 – 7.09 (m, 3H), 6.66 – 6.59 (m, 1H), 6.58 – 6.50 (m, 1H), 6.53 – 6.44 (m, 1H), 5.74 (s, 1H), 3.42 – 3.28 (m, 2H), 3.20 – 3.09 (m, 1H), 2.95 (s, 3H), 2.37 – 2.25 (m, 1H), 2.02 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.92, 145.78, 143.16, 131.13, 129.32, 128.89, 128.85, 125.48, 120.71, 115.81, 111.21, 60.69, 47.16, 38.87, 31.34, 24.37.



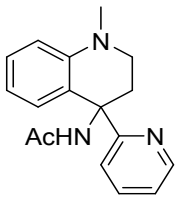
***N*-(1-methyl-4-(4-(trifluoromethyl)phenyl)-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ah):[1]** yellow solid was obtained in 68% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.32 (d, *J* = 8.0 Hz, 2H), 6.13 (d, *J* = 8.0 Hz, 2H), 5.94 – 5.87 (m, 1H), 5.40 (d, *J* = 8.0 Hz, 1H), 5.27 – 5.19 (m, 2H), 4.51 (s, 1H), 2.19 – 2.05 (m, 2H), 1.97 – 1.88 (m, 1H), 1.72 (s, 3H), 1.17 – 1.06 (m, 1H), 0.80 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.04, 148.04, 145.81, 129.51, 129.06, 128.95(q, J_{CF} = 32.2 Hz), 127.45, 125.34, 125.08(q, J_{CF} = 3.8 Hz), 124.13(q, J_{CF} = 270.3 Hz), 115.93, 111.35, 60.94, 47.10, 38.90, 31.39, 24.37. ^{19}F NMR (377 MHz, $CDCl_3$) δ -62.39.



***N*-(4-(4-methoxyphenyl)-1-methyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ai):** yellow solid was obtained in 30% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 – 7.12 (m, 3H), 6.88 – 6.83 (m, 2H), 6.67 – 6.60 (m, 2H), 6.54 – 6.48 (m, 1H), 5.71 (s, 1H), 3.80 (s, 3H), 3.42 – 3.30 (m, 2H), 3.16 – 3.07 (m, 1H), 2.94 (s, 3H), 2.37 – 2.27 (m, 1H), 2.04 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.83, 158.32, 145.90, 136.31, 129.01, 128.62, 128.09, 126.10, 115.71, 113.48, 111.08, 60.61, 55.21, 47.43, 38.91, 31.52, 24.54. HRMS (ESI) calculated for $C_{19}H_{23}N_2O_2$ $[M+H]^+$: 311.1754; found: 311.1752.

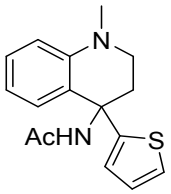


***N*-(1-methyl-4-(naphthalen-2-yl)-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3aj):[1]** yellow solid was obtained in 66% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.75 (m, 3H), 7.72 (d, J = 2.0 Hz, 1H), 7.49 – 7.41 (m, 2H), 7.35 – 7.29 (m, 1H), 7.18 – 7.11 (m, 1H), 6.66 (d, J = 8.4 Hz, 1H), 6.63 – 6.57 (m, 1H), 6.50 – 6.42 (m, 1H), 5.82 (s, 1H), 3.51 – 3.35 (m, 2H), 3.20 – 3.11 (m, 1H), 2.96 (s, 3H), 2.53 – 2.43 (m, 1H), 2.08 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.94, 145.97, 141.40, 132.95, 132.35, 129.17, 129.07, 128.25, 127.89, 127.32, 125.97, 125.86, 125.85, 125.24, 115.82, 111.18, 61.26, 47.37, 38.94, 31.29, 24.57.

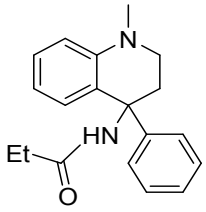


***N*-(1-methyl-4-(pyridin-2-yl)-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ak):[1]** yellow oil was obtained in 60% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 – 8.53 (m, 1H), 7.64 – 7.54 (m, 1H), 7.29 (s, 1H), 7.20 – 7.10 (m, 2H), 7.04 (dd, J = 8.0, 1.2 Hz, 1H), 6.83 – 6.75 (m, 1H), 6.67 (d, J = 8.4 Hz, 1H), 6.59 – 6.51 (m, 1H), 3.36 – 3.26 (m, 1H), 3.24 – 3.07 (m, 2H), 2.96 (s, 3H), 2.33 –

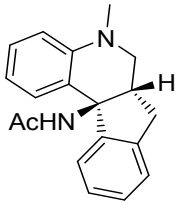
2.21 (m, 1H), 2.07 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.60, 163.24, 148.33, 146.54, 136.30, 128.61, 127.53, 124.29, 122.85, 121.99, 116.32, 111.42, 60.28, 47.61, 39.25, 32.33, 24.49.



***N*-(1-methyl-4-(thiophen-2-yl)-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3al):[1]** white solid was obtained in 62% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (dd, *J* = 5.2, 1.2 Hz, 1H), 7.19 – 7.13 (m, 1H), 6.96 – 6.92 (m, 2H), 6.84 (dd, *J* = 3.2, 1.2 Hz, 1H), 6.61 (dd, *J* = 8.0, 1.0 Hz, 1H), 6.59 – 6.54 (m, 1H), 5.93 (s, 1H), 3.52 – 3.45 (m, 1H), 3.37 – 3.30 (m, 1H), 3.20 – 3.13 (m, 1H), 2.92 (s, 3H), 2.39 – 2.31 (m, 1H), 1.99 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.70, 150.00, 145.30, 129.29, 127.54, 126.64, 125.28, 125.00, 124.51, 115.69, 111.19, 58.46, 47.29, 38.85, 32.77, 24.34.

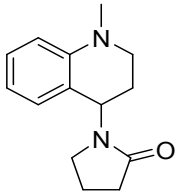


***N*-(1-methyl-4-phenyl-1,2,3,4-tetrahydroquinolin-4-yl)propionamide (3am):[1]** yellow solid was obtained in 65% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.29 (m, 2H), 7.27 – 7.21 (m, 3H), 7.17 – 7.12 (m, 1H), 6.63 (d, *J* = 8.4 Hz, 1H), 6.59 (dd, *J* = 7.6, 1.6 Hz, 1H), 6.52 – 6.47 (m, 1H), 5.68 (s, 1H), 3.47 – 3.30 (m, 2H), 3.17 – 3.09 (m, 1H), 2.95 (s, 3H), 2.40 – 2.32 (m, 1H), 2.31 – 2.23 (m, 2H), 1.18 (t, J = 7.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.54, 145.94, 144.31, 129.03, 128.75, 128.12, 126.95, 126.79, 126.13, 115.74, 111.11, 60.82, 47.37, 38.91, 31.57, 30.65, 9.99.

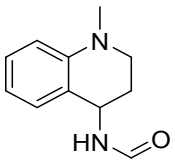


***N*-((6aS,11bS)-5-methyl-5,6,6a,7-tetrahydro-11bH-indeno[2,1-c]quinolin-11b-yl)acetamide (3an):[1]** white solid was obtained in 30% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.34 (m, 2H), 7.22 – 7.07 (m, 4H), 6.69 (t, *J* = 7.6 Hz, 1H), 6.63 (d, *J* = 8.2 Hz, 1H), 5.96 (s, 1H), 3.78 –

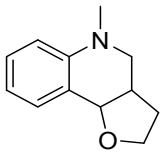
3.68 (m, 1H), 3.43 (dd, *J* = 12.0, 3.6 Hz, 1H), 3.08 – 2.99 (m, 2H), 2.95 – 2.84 (m, 4H), 1.94 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.19, 146.55, 145.90, 141.32, 128.46, 128.42, 127.56, 126.39, 125.35, 124.25, 123.28, 117.29, 112.01, 64.94, 50.88, 42.96, 39.58, 33.13, 24.18.



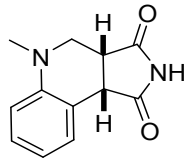
1-(1-methyl-1,2,3,4-tetrahydroquinolin-4-yl)pyrrolidin-2-one (3ao):[2] yellow solid was obtained in 49% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 – 7.10 (m, 1H), 6.90 – 6.84 (m, 1H), 6.70 – 6.60 (m, 2H), 5.42 (dd, *J* = 9.2, 6.0 Hz, 1H), 3.38 – 3.30 (m, 1H), 3.27 – 3.17 (m, 2H), 3.16 – 3.09 (m, 1H), 2.88 (s, 3H), 2.52 – 2.45 (m, 2H), 2.21 – 2.09 (m, 1H), 2.09 – 1.95 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.38, 147.40, 128.45, 127.46, 119.69, 116.68, 111.71, 49.45, 47.81, 43.44, 39.25, 31.44, 26.65, 18.20.



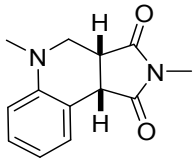
***N*-(1-methyl-1,2,3,4-tetrahydroquinolin-4-yl)formamide (3ap):** yellow solid was obtained in 55% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 – 8.08 (m, 1H), 7.25 – 7.06 (m, 2H), 6.77 – 6.56 (m, 2H), 5.87 (s, 1H), 5.25 – 5.07 (m, 1H), 3.34 – 3.12 (m, 2H), 2.91 (s, 3H), 2.22 – 1.98 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.07, 146.57, 129.21, 129.18, 120.71, 116.54, 111.56, 47.08, 44.83, 39.06, 28.40. HRMS (ESI) calculated for $C_{11}H_{15}N_2O$ $[M+H]^+$: 191.1179; found: 191.1180.



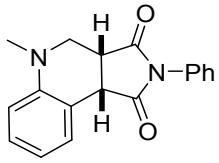
5-methyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3aq):[3] colorless liquid was obtained in 61% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (dd, *J* = 7.5, 1.7 Hz, 1H), 7.23 – 7.16 (m, 1H), 6.79 – 6.73 (m, 1H), 6.70 (dd, *J* = 8.3, 1.0 Hz, 1H), 4.58 (d, *J* = 5.4 Hz, 1H), 3.98 – 3.90 (m, 1H), 3.85 – 3.76 (m, 1H), 3.04 – 2.96 (m, 1H), 2.88 (s, 3H), 2.78 (t, *J* = 11.1 Hz, 1H), 2.57 – 2.47 (m, 1H), 2.30 – 2.19 (m, 1H), 1.79 – 1.71 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.10, 131.11, 129.00, 121.59, 117.36, 111.78, 75.79, 65.09, 52.49, 39.27, 35.88, 30.00.



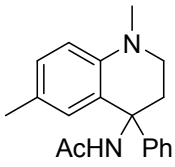
(3aR,9bS)-5-methyl-3a,4,5,9b-tetrahydro-1H-pyrrolo[3,4-c]quinoline-1,3(2H)-dione (3ar):[4] white solid was obtained in 64% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (s, 1H), 7.47 – 7.38 (m, 1H), 7.25 – 7.19 (m, 1H), 6.93 – 6.86 (m, 1H), 6.72 (dd, *J* = 8.3, 1.1 Hz, 1H), 4.03 (d, *J* = 9.5 Hz, 1H), 3.50 (dd, *J* = 11.5, 2.6 Hz, 1H), 3.45 – 3.39 (m, 1H), 3.06 – 2.98 (m, 1H), 2.81 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.83, 176.82, 148.46, 130.05, 128.68, 119.67, 118.34, 112.53, 50.36, 44.76, 43.21, 39.38.



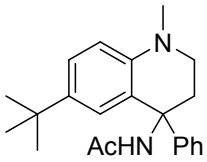
(3aR,9bS)-2,5-dimethyl-3a,4,5,9b-tetrahydro-1H-pyrrolo[3,4-c]quinoline-1,3(2H)-dione (3as):[4] yellow solid was obtained in 53% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.44 (m, 1H), 7.26 – 7.16 (m, 1H), 6.89 (m, 1H), 6.70 (dd, *J* = 8.2, 1.1 Hz, 1H), 4.00 (d, *J* = 9.4 Hz, 1H), 3.54 (dd, *J* = 11.5, 2.4 Hz, 1H), 3.37 (m, 1H), 3.03 (dd, *J* = 11.5, 4.5 Hz, 1H), 2.99 (s, 3H), 2.79 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.76, 176.80, 148.36, 130.13, 128.54, 119.57, 118.65, 112.43, 50.43, 43.56, 41.98, 39.36, 25.33.



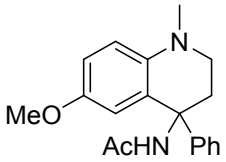
(3aR,9bS)-5-methyl-2-phenyl-3a,4,5,9b-tetrahydro-1H-pyrrolo[3,4-c]quinoline-1,3(2H)-dione (3at):[4] white solid was obtained in 65% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 – 7.51 (m, 1H), 7.46 – 7.39 (m, 2H), 7.38 – 7.33 (m, 1H), 7.29 – 7.20 (m, 3H), 6.94 – 6.87 (m, 1H), 6.74 (dd, *J* = 8.3, 1.1 Hz, 1H), 4.16 (d, *J* = 9.5 Hz, 1H), 3.61 (dd, *J* = 11.4, 2.8 Hz, 1H), 3.57 – 3.50 (m, 1H), 3.12 (dd, *J* = 11.5, 4.4 Hz, 1H), 2.84 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.70, 175.75, 148.47, 131.92, 130.29, 128.97, 128.65, 128.48, 126.32, 119.63, 118.49, 112.50, 50.62, 43.55, 42.09, 39.41.



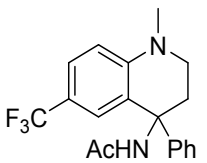
***N*-(1,6-dimethyl-4-phenyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ba):[1]** yellow solid was obtained in 68% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.28 (m, 2H), 7.26 – 7.20 (m, 3H), 6.99 – 6.90 (m, 1H), 6.56 (d, *J* = 8.4 Hz, 1H), 6.41 (d, *J* = 2.4 Hz, 1H), 5.77 (s, 1H), 3.44 – 3.36 (m, 1H), 3.34 – 3.25 (m, 1H), 3.12 – 3.04 (m, 1H), 2.91 (s, 3H), 2.38 – 2.29 (m, 1H), 2.07 (s, 3H), 2.01 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.77, 144.39, 143.97, 129.55, 129.26, 128.01, 126.98, 126.68, 126.19, 124.93, 111.43, 61.12, 47.48, 39.15, 31.94, 24.45, 20.23.



***N*-(6-(tert-butyl)-1-methyl-4-phenyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ca):** yellow solid was obtained in 50% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.29 (m, 2H), 7.28 – 7.21 (m, 3H), 7.18 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.63 – 6.56 (m, 2H), 5.75 (s, 1H), 3.46 – 3.31 (m, 2H), 3.17 – 3.08 (m, 1H), 2.93 (s, 3H), 2.41 – 2.31 (m, 1H), 2.04 (s, 3H), 1.09 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.84, 144.16, 143.72, 138.30, 127.96, 126.94, 126.72, 126.13, 125.76, 125.70, 110.71, 61.30, 47.36, 38.93, 33.61, 31.54, 31.23, 24.46. HRMS (ESI) calculated for $C_{22}H_{28}N_2NaO$ $[M+Na]^+$: 359.2094; found: 359.2099.

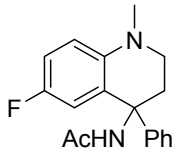


***N*-(6-methoxy-1-methyl-4-phenyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3da):[1]** yellow oil was obtained in 52% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.28 (m, 2H), 7.27 – 7.21 (m, 3H), 6.76 (dd, *J* = 8.8, 3.2 Hz, 1H), 6.61 (d, *J* = 8.0 Hz, 1H), 6.28 (d, *J* = 2.8 Hz, 1H), 5.78 (s, 1H), 3.59 (s, 3H), 3.46 – 3.39 (m, 1H), 3.29 – 3.21 (m, 1H), 3.10 – 3.01 (m, 1H), 2.90 (s, 3H), 2.41 – 2.29 (m, 1H), 2.05 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.77, 150.73, 144.36, 140.96, 128.17, 127.34, 126.97, 126.92, 115.23, 114.19, 112.45, 61.32, 55.60, 47.72, 39.59, 32.14, 24.54.

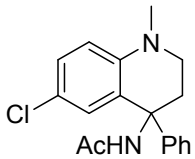


***N*-(1-methyl-4-phenyl-6-(trifluoromethyl)-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ea):** white solid was obtained in 62% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.26 (m, 4H),

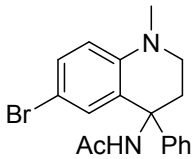
7.23 – 7.14 (m, 2H), 6.92 (d, J = 2.4 Hz, 1H), 6.62 (d, J = 8.4 Hz, 1H), 5.76 (s, 1H), 3.54 – 3.43 (m, 1H), 3.40 – 3.31 (m, 1H), 3.16 – 3.05 (m, 1H), 2.98 (s, 3H), 2.34 – 2.23 (m, 1H), 2.06 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.01, 148.05, 143.53, 128.55, 127.54, 126.86, 126.17（q, J_{CF} = 7.5 Hz）, 125.15 （q, J_{CF} = 7.5 Hz）,124.80（q, J_{CF} = 146.8 Hz）, 124.62, 116.80 （d, J_{CF} = 32.4 Hz）, 110.35, 60.79, 47.29, 38.77, 31.10, 24.48. ^{19}F NMR (377 MHz, $CDCl_3$) δ -60.75. HRMS (ESI) calculated for $C_{19}H_{19}F_3N_2NaO$ $[M+Na]^+$: 371.1342; found: 371.1345.



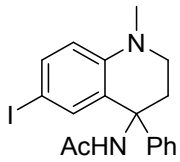
***N*-(6-fluoro-1-methyl-4-phenyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3fa):[1]** white solid was obtained in 64% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.29 (m, 2H), 7.29 – 7.24 (m, 1H), 7.23 – 7.16 (m, 2H), 6.90 – 6.82 (m, 1H), 6.55 (dd, J = 9.2, 4.8 Hz, 1H), 6.46 (dd, J = 9.6, 3.2 Hz, 1H), 5.85 (s, 1H), 3.45 – 3.36 (m, 1H), 3.29 – 3.20 (m, 1H), 3.07 – 2.97 (m, 1H), 2.89 (s, 3H), 2.34 – 2.24 (m, 1H), 2.01 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.88, 154.52(d, J_{CF} = 232.8 Hz), 144.13, 142.67, 128.32, 127.19, 126.88, 126.47 (d, J_{CF} = 5.8 Hz), 115.36 (d, J_{CF} = 21.9 Hz), 114.95 (d, J_{CF} = 23.0 Hz), 111.95 (d, J_{CF} = 7.1 Hz), 60.97, 47.45, 39.33, 31.84, 24.39. ^{19}F NMR (377 MHz, $CDCl_3$) δ -128.90.



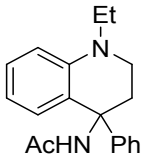
***N*-(6-chloro-1-methyl-4-phenyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ga):[1]** white solid was obtained in 66% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.31 (m, 3H), 7.30 – 7.24 (m, 1H), 7.23 – 7.16 (m, 2H), 6.89 (d, J = 2.0 Hz, 1H), 6.39 (d, J = 8.8 Hz, 1H), 5.76 (s, 1H), 3.46 – 3.37 (m, 1H), 3.34 – 3.25 (m, 1H), 3.11 – 3.02 (m, 1H), 2.90 (s, 3H), 2.32 – 2.20 (m, 1H), 2.04 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.89, 144.59, 143.80, 128.78, 128.41, 128.00, 127.30, 126.89, 126.80, 120.30, 112.31, 60.92, 47.32, 38.99, 31.53, 24.50.



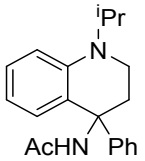
***N*-(6-bromo-1-methyl-4-phenyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ha):**[1] yellow solid was obtained in 60% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.31 (m, 2H), 7.30 – 7.24 (m, 1H), 7.24 – 7.17 (m, 3H), 6.75 (d, *J* = 2.4 Hz, 1H), 6.49 (d, *J* = 8.8 Hz, 1H), 5.74 (s, 1H), 3.47 – 3.39 (m, 1H), 3.34 – 3.25 (m, 1H), 3.10 – 3.02 (m, 1H), 2.91 (s, 3H), 2.32 – 2.23 (m, 1H), 2.05 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.91, 144.97, 143.74, 131.66, 130.76, 128.43, 127.32, 127.29, 126.89, 112.80, 107.33, 60.91, 47.30, 38.92, 31.47, 24.51.



***N*-(6-iodo-1-methyl-4-phenyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ia):** yellow solid was obtained in 46% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.31 (m, 3H), 7.30 – 7.24 (m, 1H), 7.22 – 7.15 (m, 2H), 6.89 (d, *J* = 2.4 Hz, 1H), 6.39 (d, *J* = 8.8 Hz, 1H), 5.78 (s, 1H), 3.47 – 3.37 (m, 1H), 3.35 – 3.25 (m, 1H), 3.10 – 3.01 (m, 1H), 2.90 (s, 3H), 2.31 – 2.21 (m, 1H), 2.03 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.93, 145.49, 143.70, 137.49, 136.45, 128.38, 127.92, 127.28, 126.87, 113.45, 60.74, 47.23, 38.79, 31.38, 24.47. HRMS (ESI) calculated for $C_{18}H_{19}IN_2NaO$ $[M+Na]^+$: 429.0434; found: 429.0445.

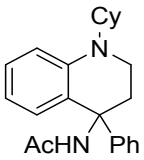


***N*-(1-ethyl-4-phenyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ja):**[1] yellow oil was obtained in 70% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.30 (m, 2H), 7.29 – 7.21 (m, 3H), 7.18 – 7.08 (m, 1H), 6.70 – 6.63 (m, 1H), 6.63 – 6.57 (m, 1H), 6.53 – 6.39 (m, 1H), 5.72 (s, 1H), 3.49 – 3.30 (m, 4H), 3.18 – 3.08 (m, 1H), 2.38 – 2.28 (m, 1H), 2.06 (s, 3H), 1.17 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.92, 144.47, 144.10, 129.06, 129.00, 128.15, 126.99, 126.87, 125.69, 115.07, 110.94, 61.11, 45.23, 44.45, 31.29, 24.48, 10.85.

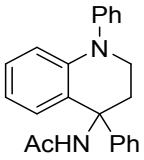


***N*-(1-isopropyl-4-phenyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ka):** yellow solid was obtained in 66% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.29 (m, 2H), 7.28 – 7.22 (m,

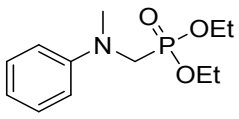
3H), 7.16 – 7.10 (m, 1H), 6.76 (d, *J* = 8.0 Hz, 1H), 6.59 (dd, *J* = 7.6, 1.6 Hz, 1H), 6.48 – 6.40 (m, 1H), 5.75 (s, 1H), 4.22 – 4.08 (m, 1H), 3.42 – 3.32 (m, 1H), 3.23 – 3.06 (m, 2H), 2.32 – 2.19 (m, 1H), 2.04 (s, 3H), 1.21 (dd, *J* = 6.4, 3.6 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.09, 144.90, 143.96, 129.07, 128.85, 128.14, 126.94, 126.84, 126.40, 114.86, 111.20, 61.07, 47.13, 36.40, 31.27, 24.34, 18.91, 18.82. HRMS (ESI) calculated for $C_{20}H_{24}N_2NaO$ $[M+Na]^+$: 331.1781; found: 331.1788.



***N*-(1-cyclohexyl-4-phenyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3la):** yellow solid was obtained in 57% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.30 (m, 2H), 7.28 – 7.23 (m, 3H), 7.16 – 7.09 (m, 1H), 6.72 (d, *J* = 8.0 Hz, 1H), 6.57 (dd, *J* = 7.6, 1.6 Hz, 1H), 6.46 – 6.40 (m, 1H), 5.72 (s, 1H), 3.69 – 3.59 (m, 1H), 3.39 – 3.31 (m, 1H), 3.27 – 3.12 (m, 2H), 2.31 – 2.21 (m, 1H), 2.05 (s, 3H), 1.91 – 1.67 (m, 6H), 1.57 – 1.34 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.08, 144.86, 143.92, 129.05, 128.83, 128.16, 126.96, 126.86, 126.33, 114.72, 111.14, 61.04, 56.58, 37.93, 31.36, 29.55, 29.32, 26.22, 26.02, 25.90, 24.39. HRMS (ESI) calculated for $C_{23}H_{28}N_2NaO$ $[M+Na]^+$: 371.2094; found: 371.2091.



***N*-(1,4-diphenyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (3ma):** yellow solid was obtained in 33% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.31 (m, 6H), 7.31 – 7.25 (m, 3H), 7.18 – 7.12 (m, 1H), 7.03 – 6.96 (m, 1H), 6.79 – 6.71 (m, 2H), 6.66 – 6.58 (m, 1H), 5.90 (s, 1H), 3.74 – 3.65 (m, 1H), 3.58 – 3.43 (m, 2H), 2.49 – 2.40 (m, 1H), 2.08 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.99, 147.66, 144.28, 144.21, 129.61, 128.68, 128.32, 128.30, 127.18, 127.12, 127.03, 125.31, 124.55, 118.00, 115.87, 61.10, 47.40, 32.18, 24.53. HRMS (ESI) calculated for $C_{23}H_{22}N_2NaO$ $[M+Na]^+$: 365.1624; found: 365.1626.



Diethyl ((methyl(phenyl)amino)methyl)phosphonate (4):[5] brown liquid was obtained in 33% isolated yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.10 (m, 2H), 6.82 (d, J = 8.2 Hz, 2H), 6.75 (t, J = 7.2 Hz, 1H), 4.26 – 3.91 (m, 4H), 3.71 (d, J = 7.8 Hz, 2H), 3.04 (s, 3H), 1.26 (t, J = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.18 (d, J_{CP} = 2.2 Hz), 128.93, 117.36, 112.78 (d, J_{CP} = 1.1 Hz), 62.06 (d, J_{CP} = 7.0 Hz), 49.82 (d, J_{CP} = 61.0 Hz), 39.08, 16.37 (d, J_{CP} = 5.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 23.68.

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(2) Min, C.; Sanchawala, A.; Seidel, D. *Org. Lett.* **2014**, *16*, 2756.

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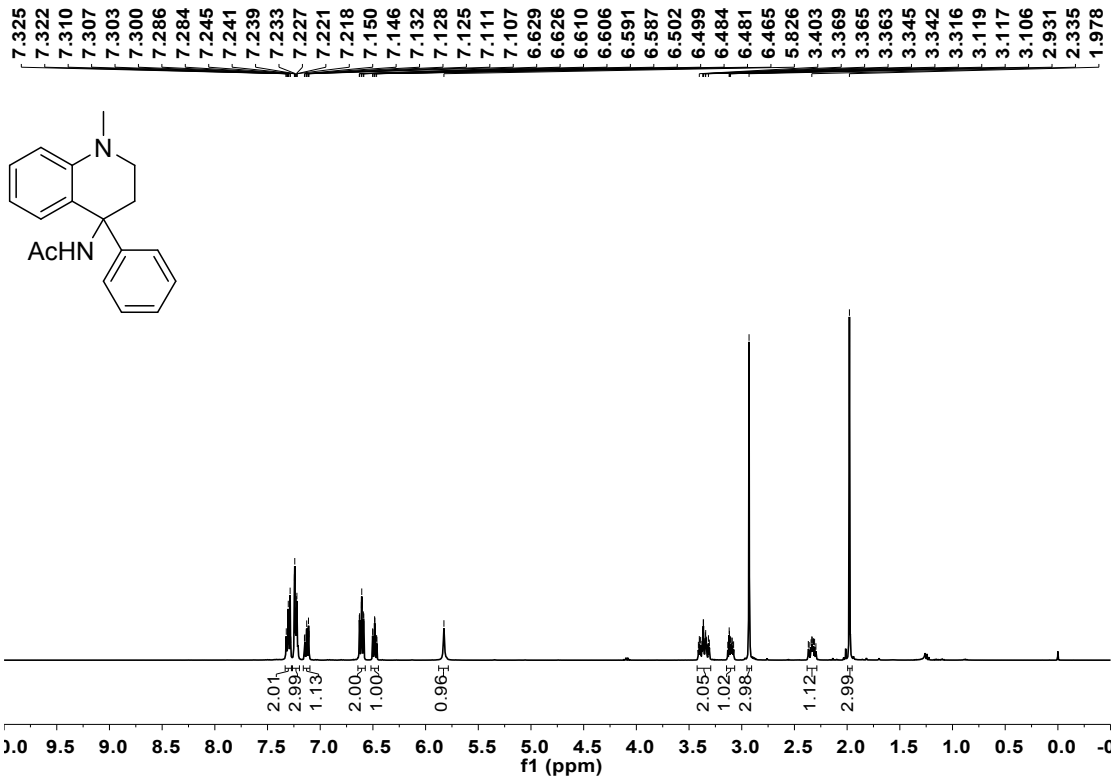
(4) Song, Z.; Antonchick, A.P. *Tetrahedron* **2016**, *72*, 7715.

(5) Niu, L.; Wang, S.; Liu, J.; Yi, H.; Liang, X.-A.; Liu, T.; Lei, A. *Chem. Commun.* **2018**, *54*, 1659.

Copies of product NMR Spectra

3aa

^{1}H NMR



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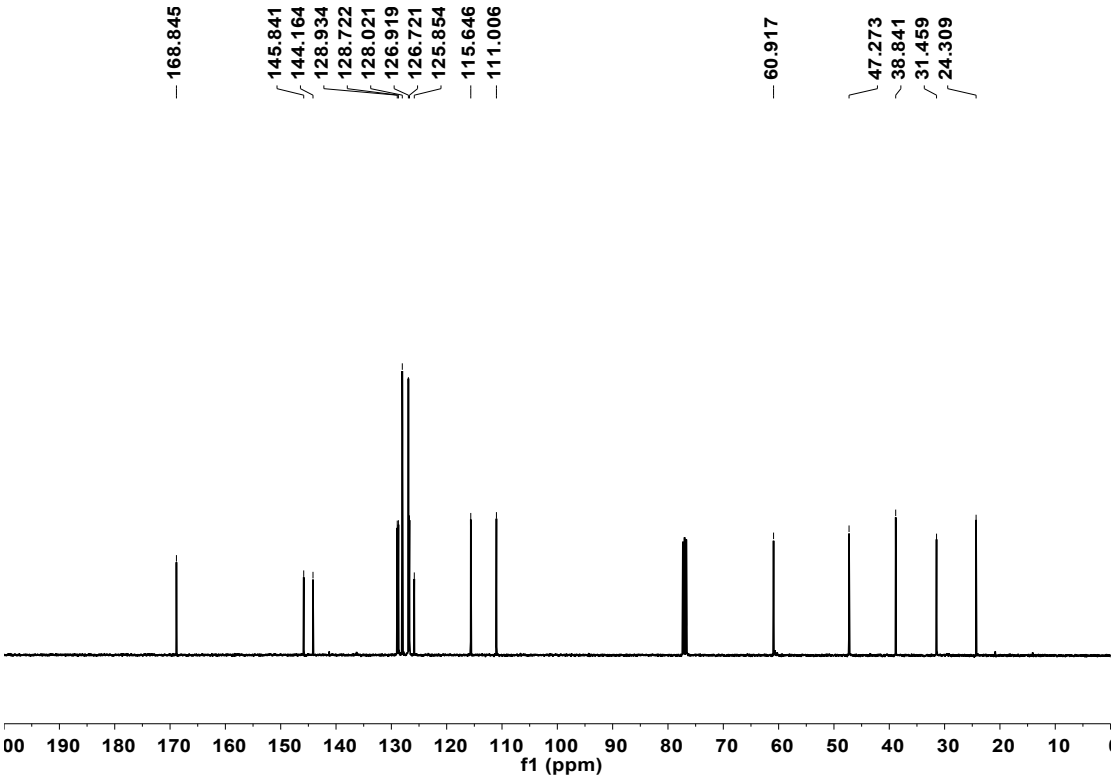

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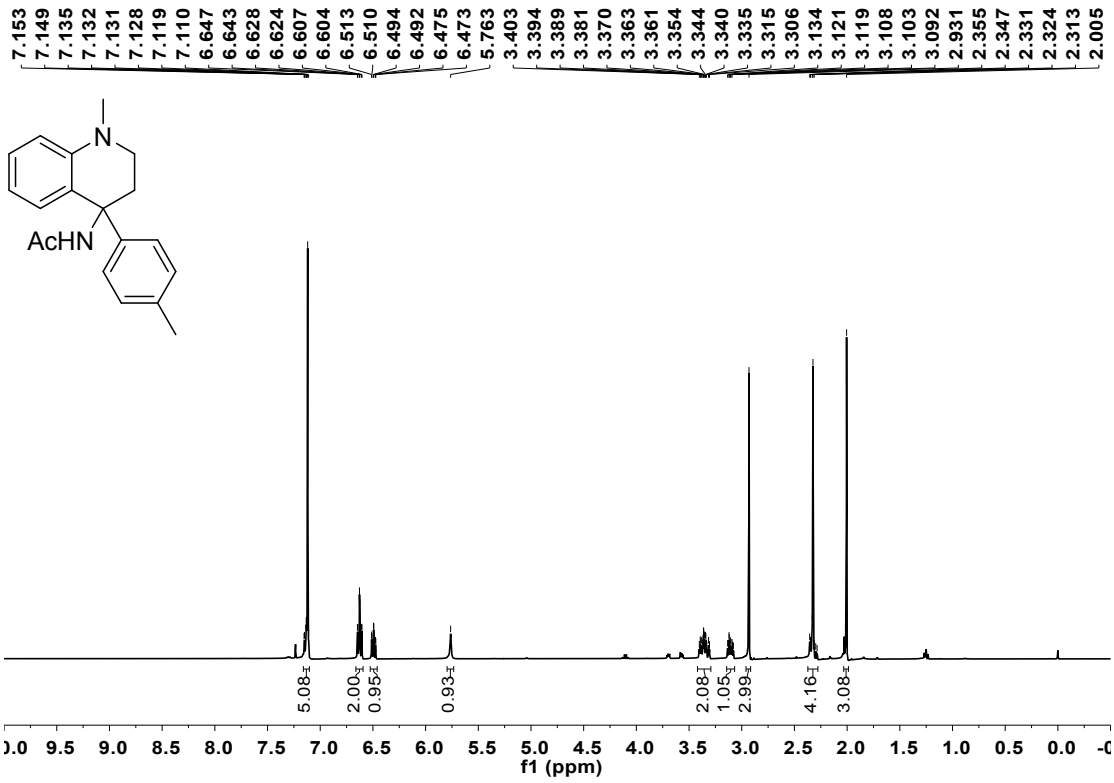

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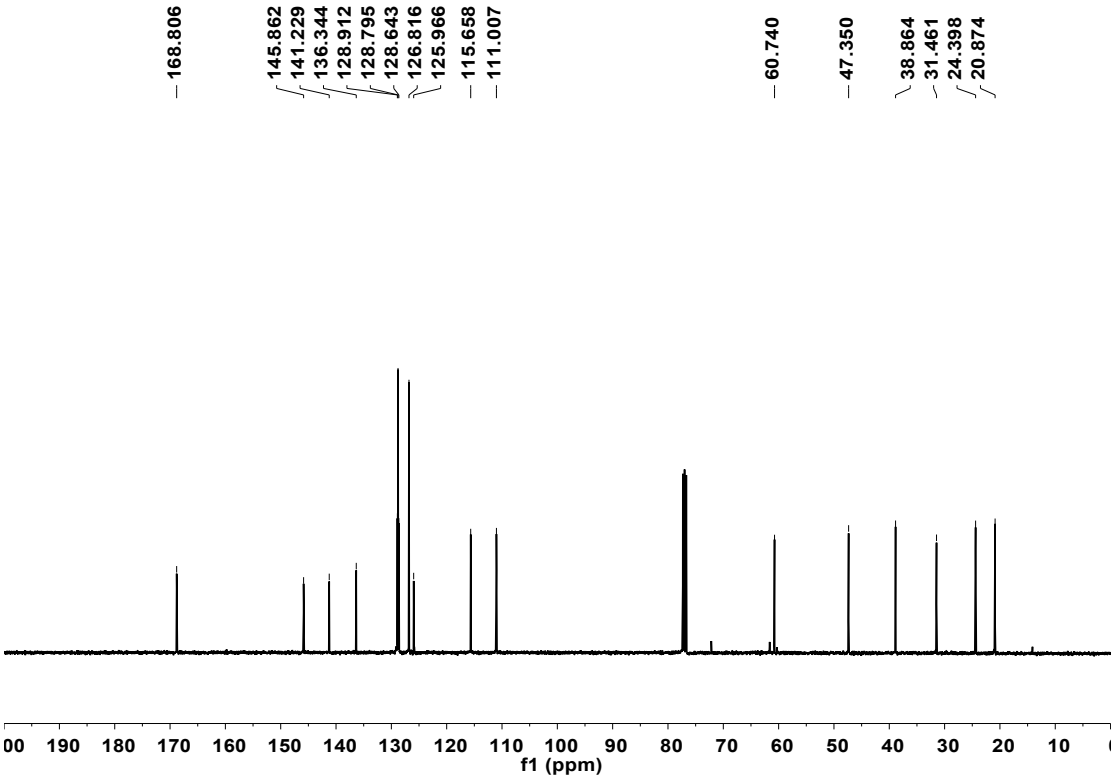

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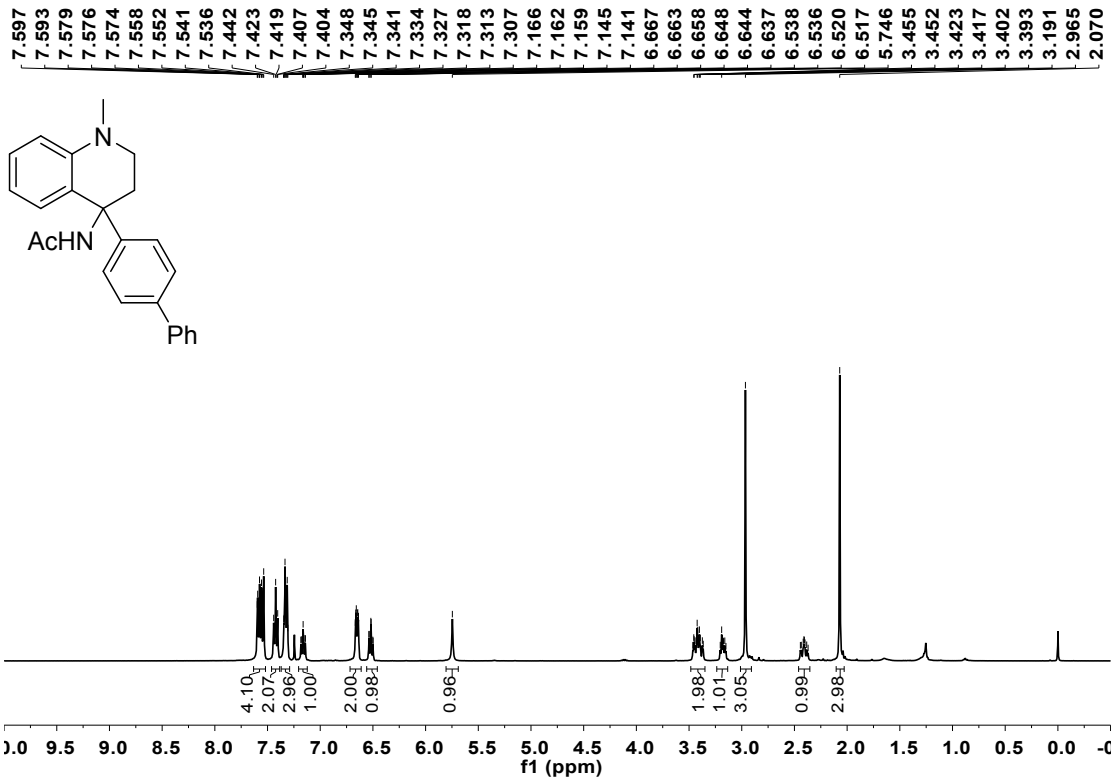

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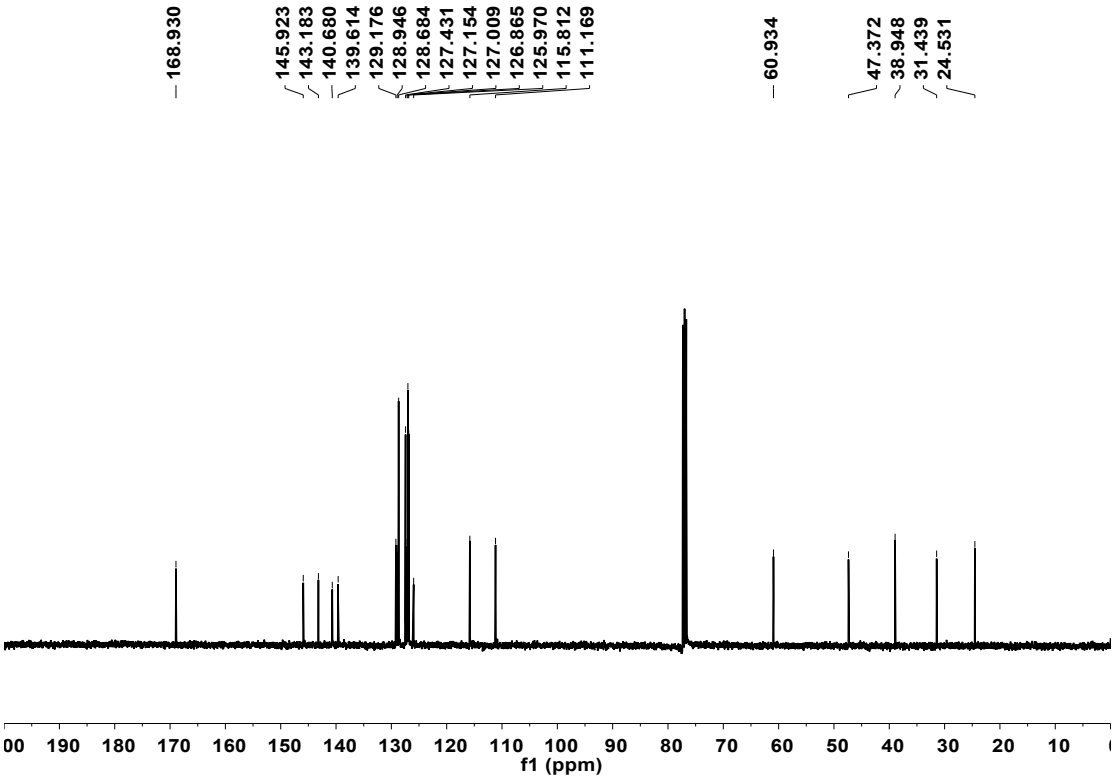

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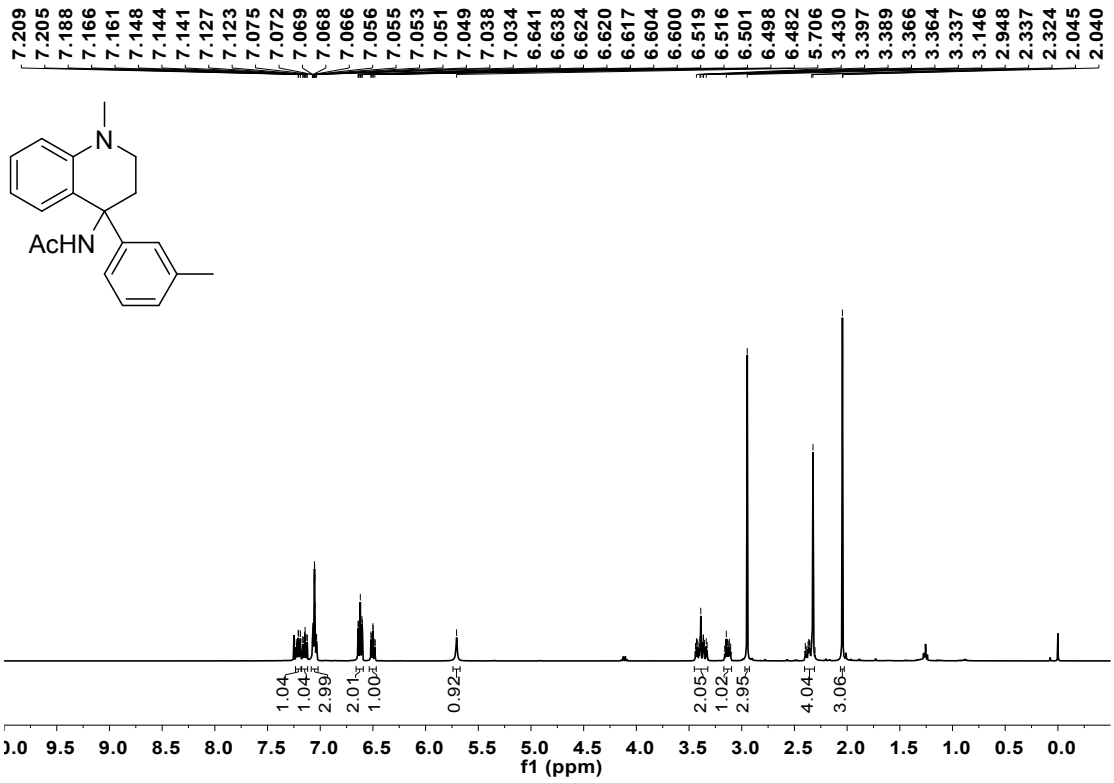

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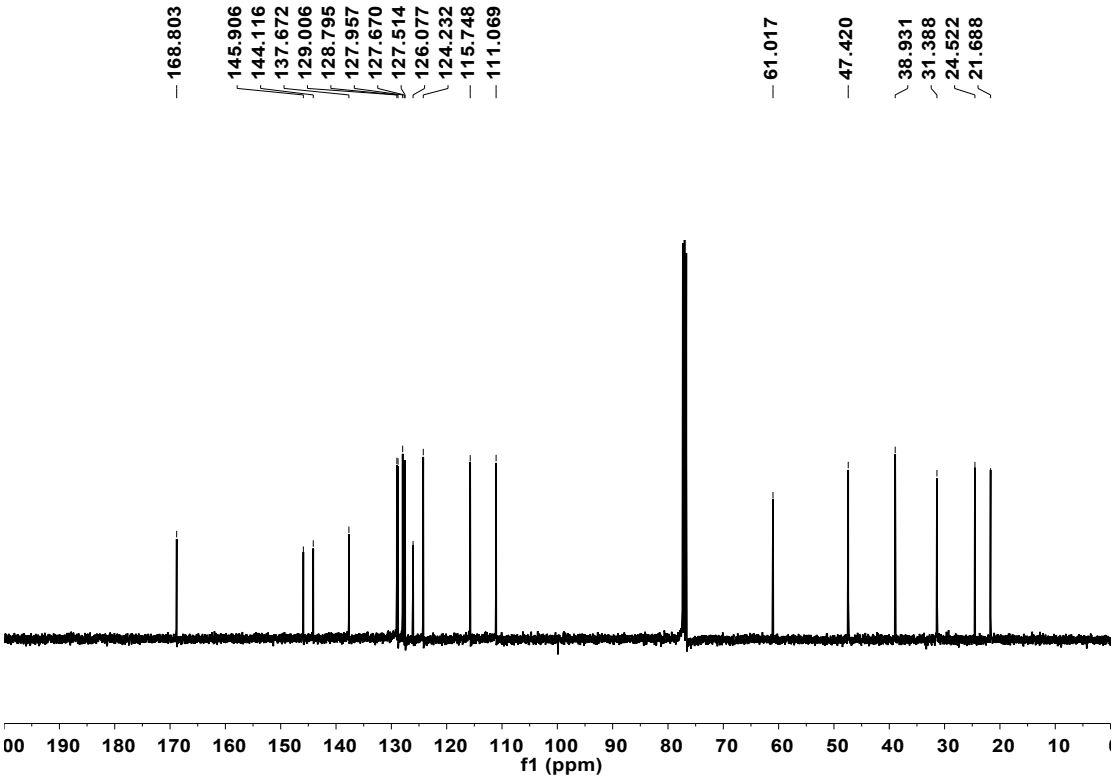

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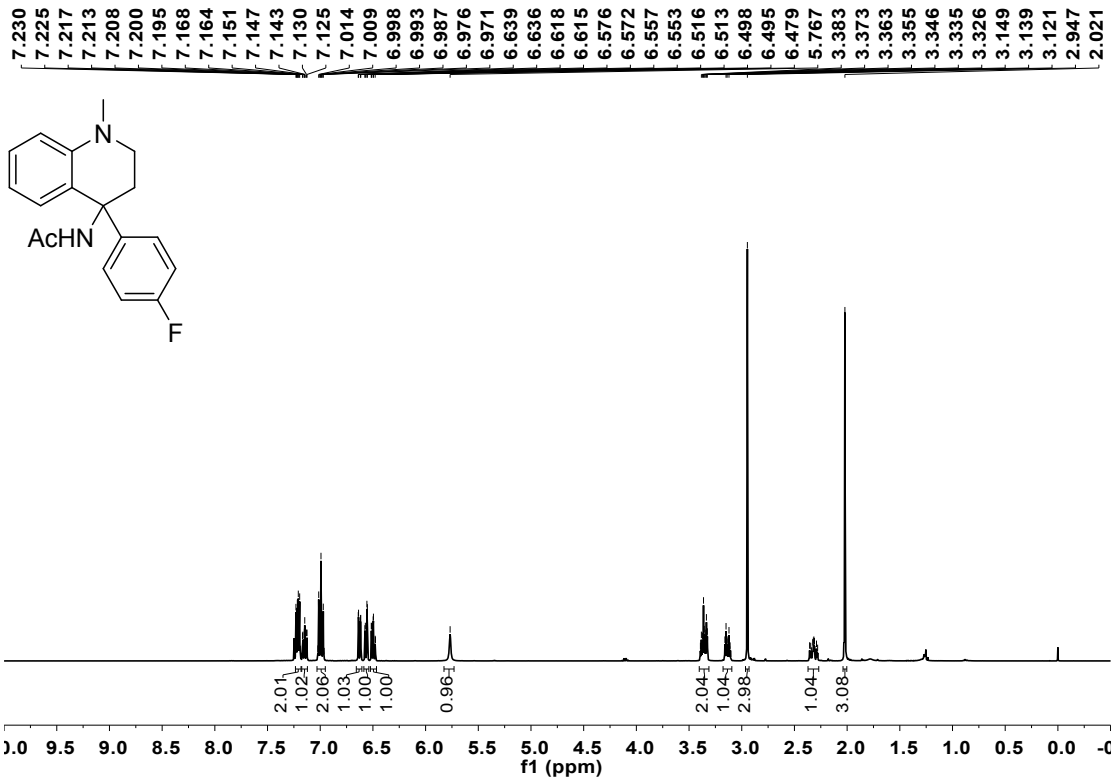

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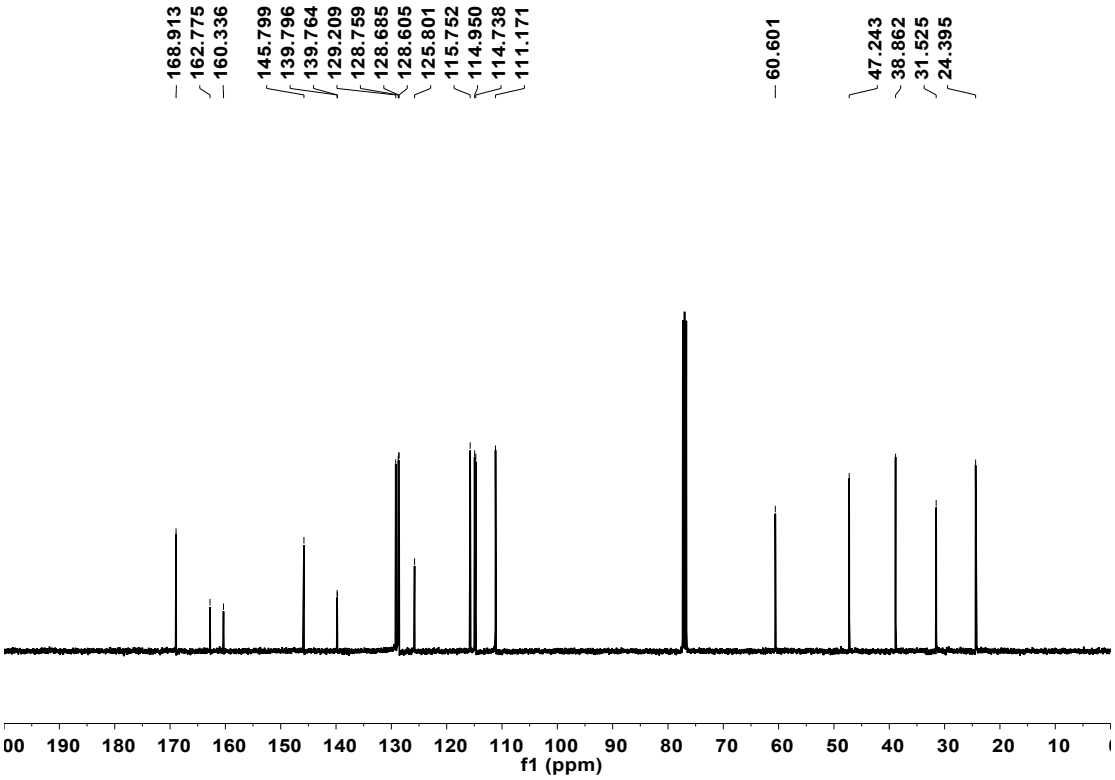

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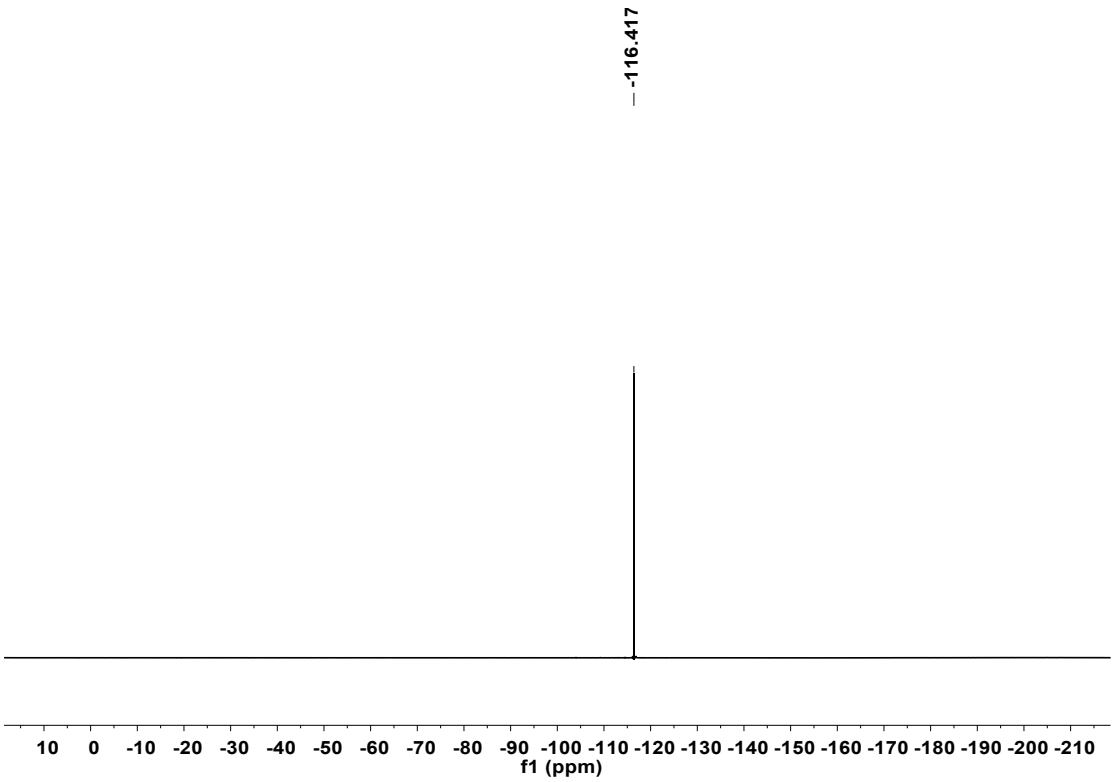
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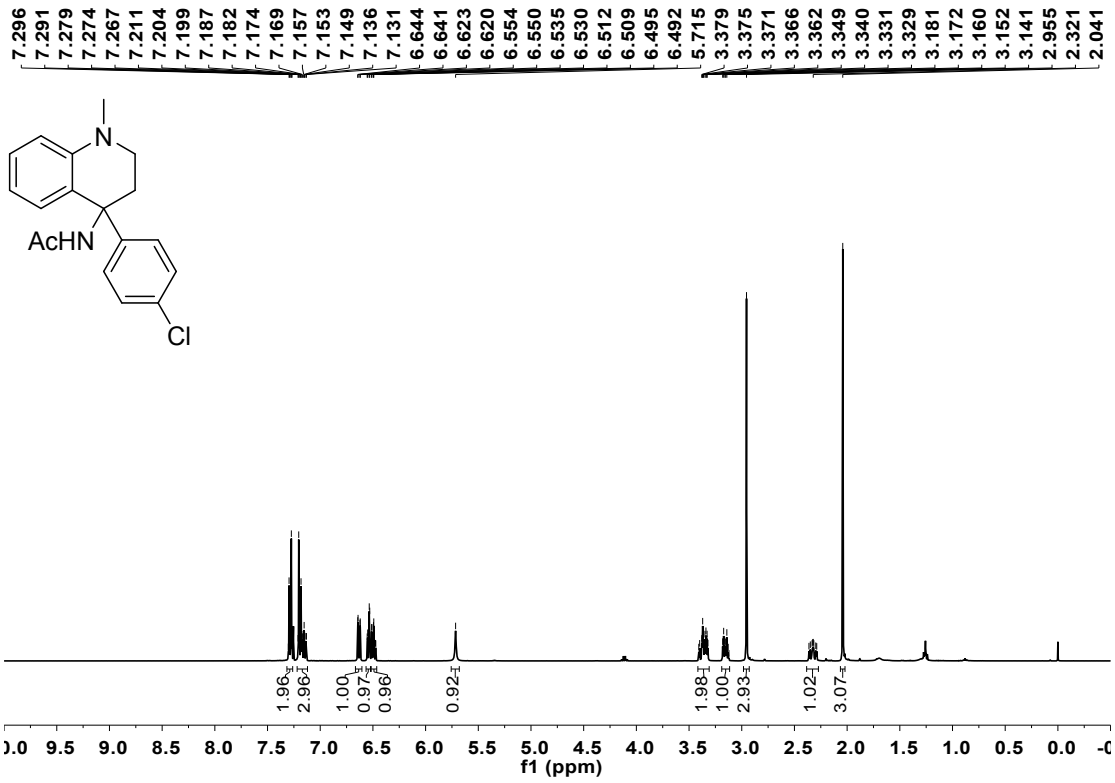
f1 (ppm)

3af

^{1}H NMR



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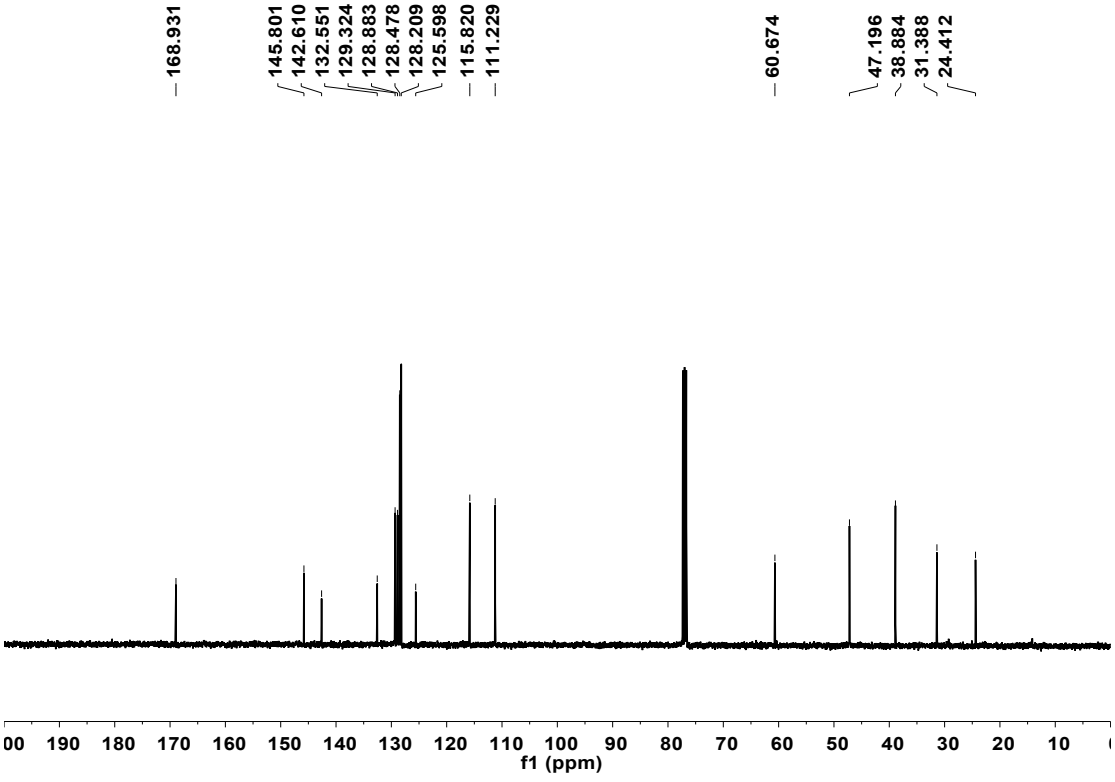

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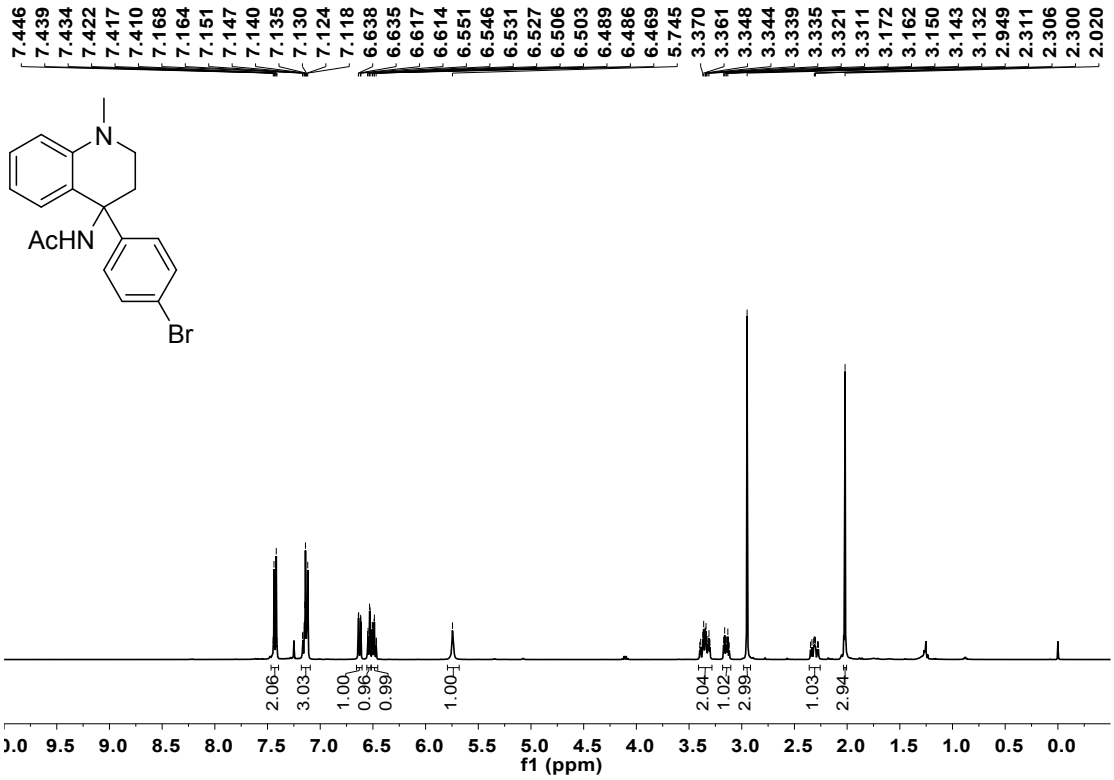

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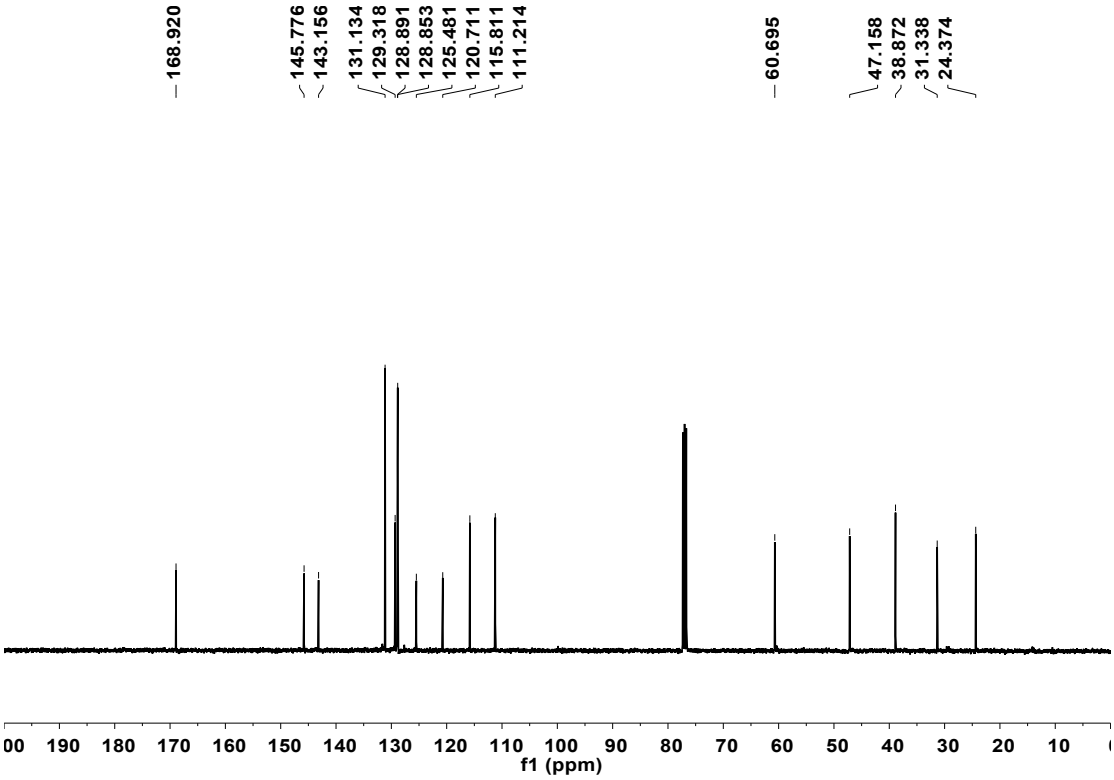

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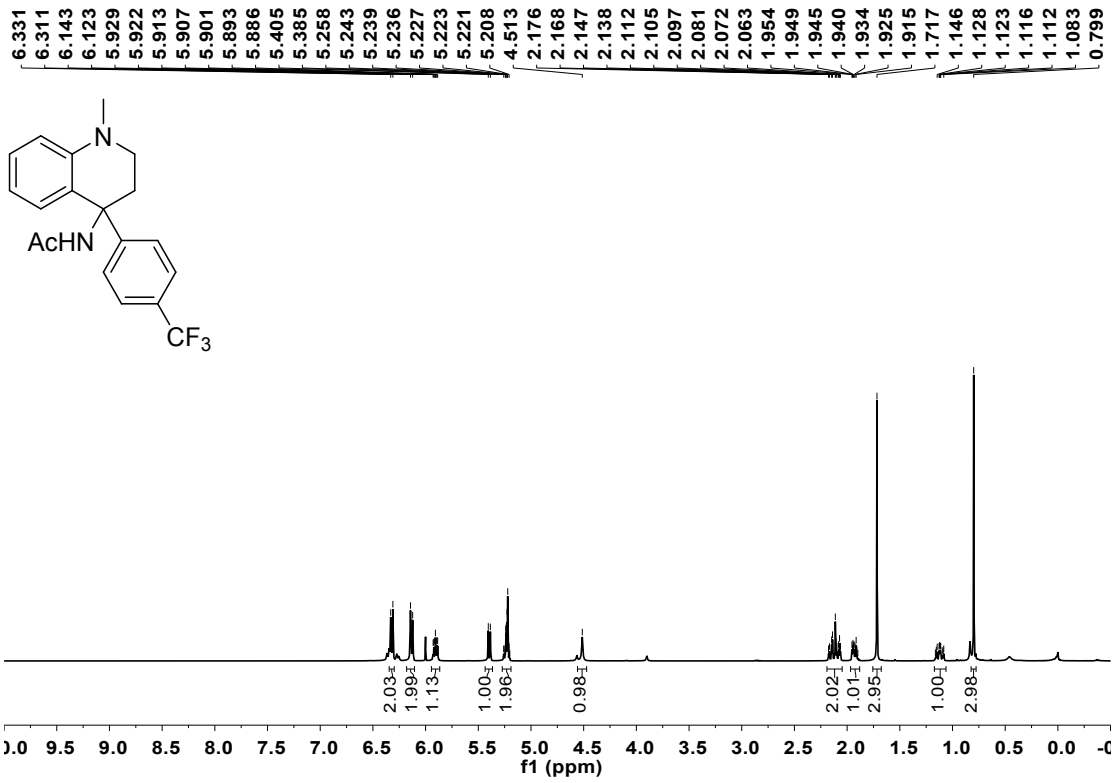

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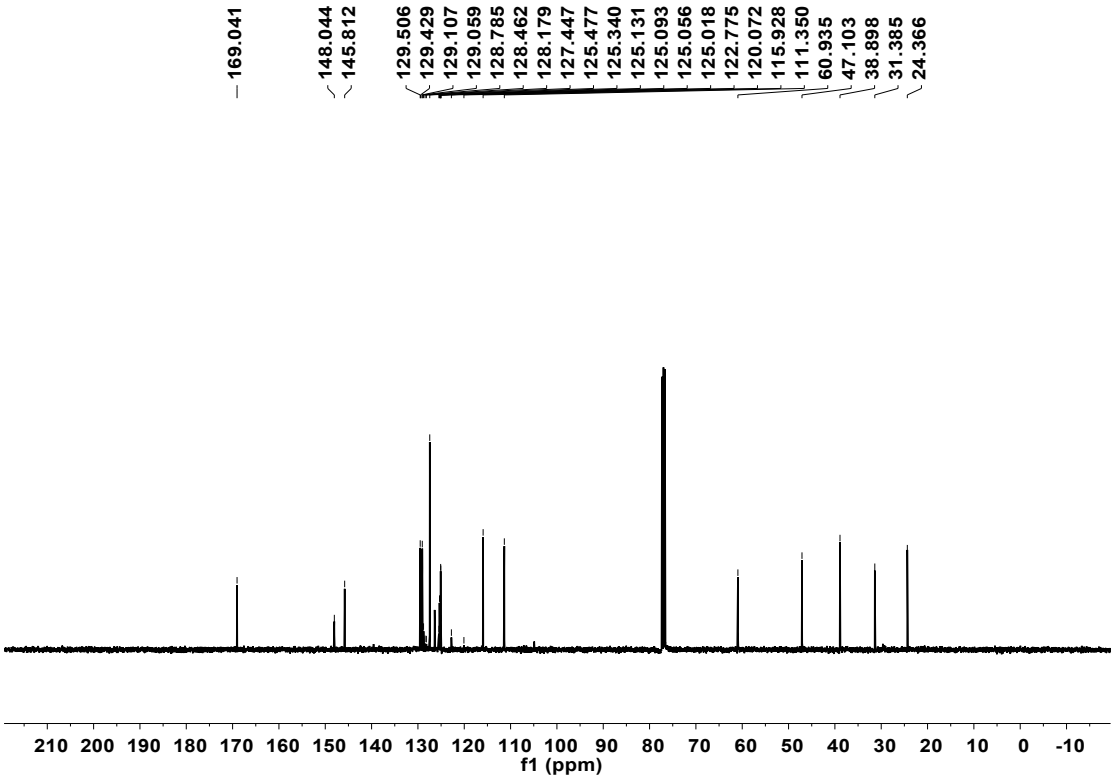

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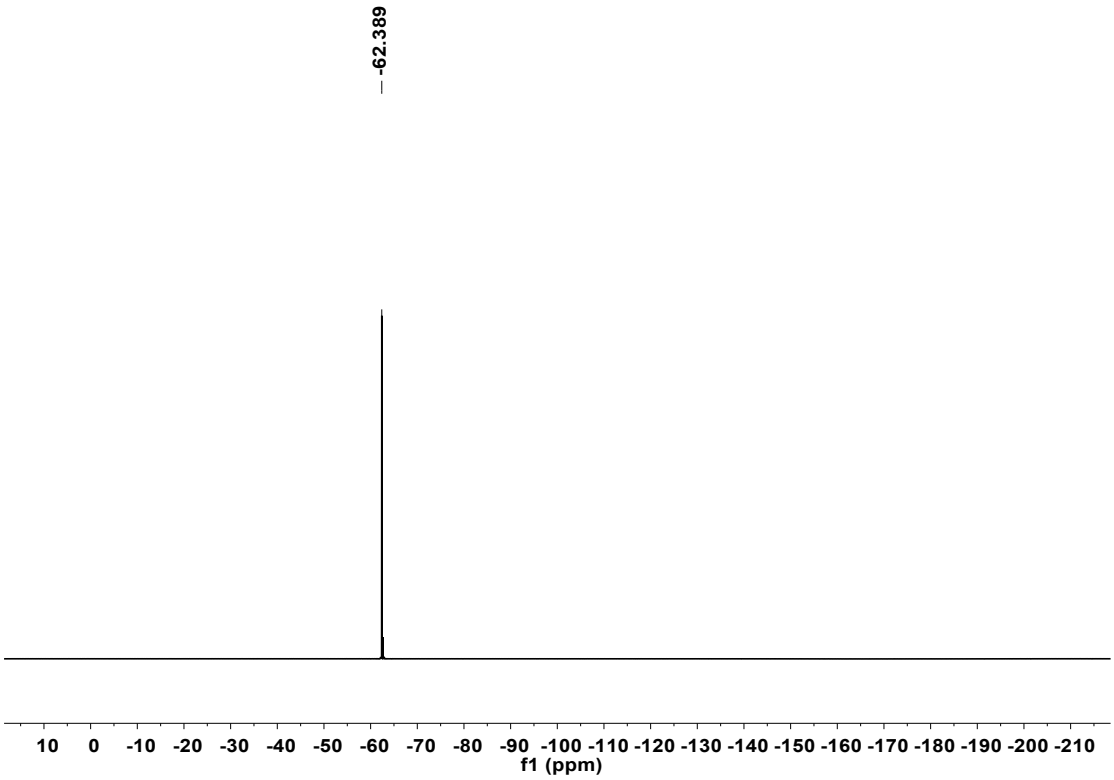
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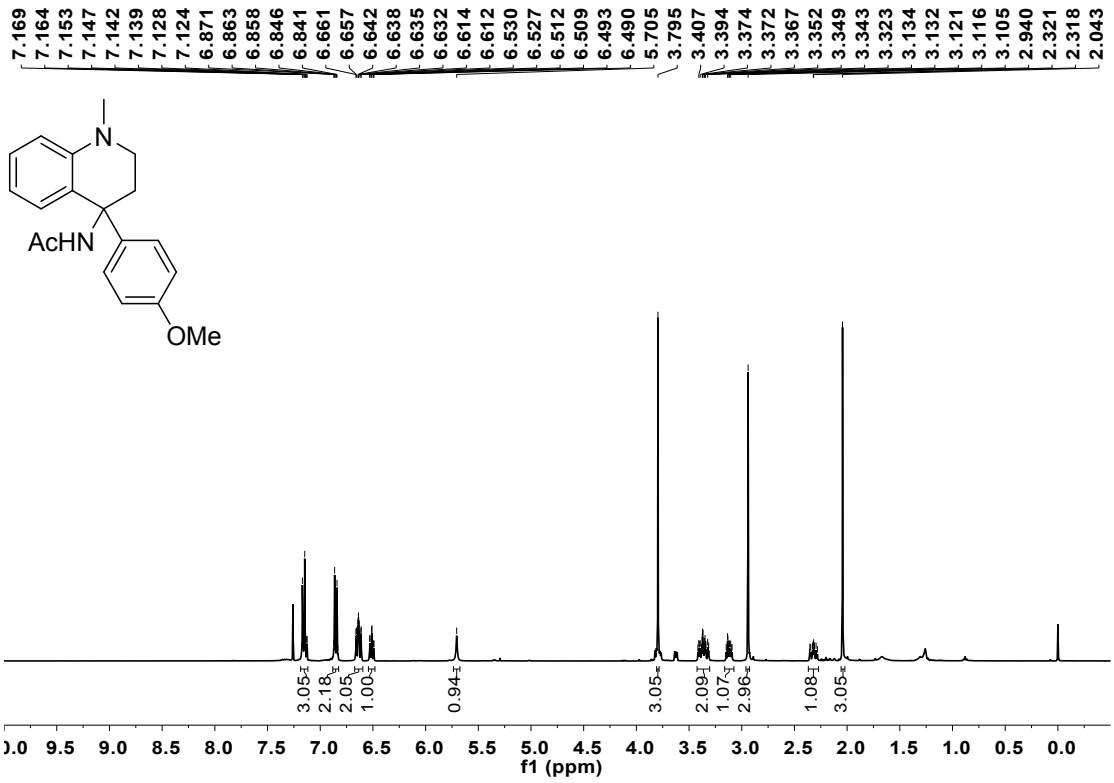

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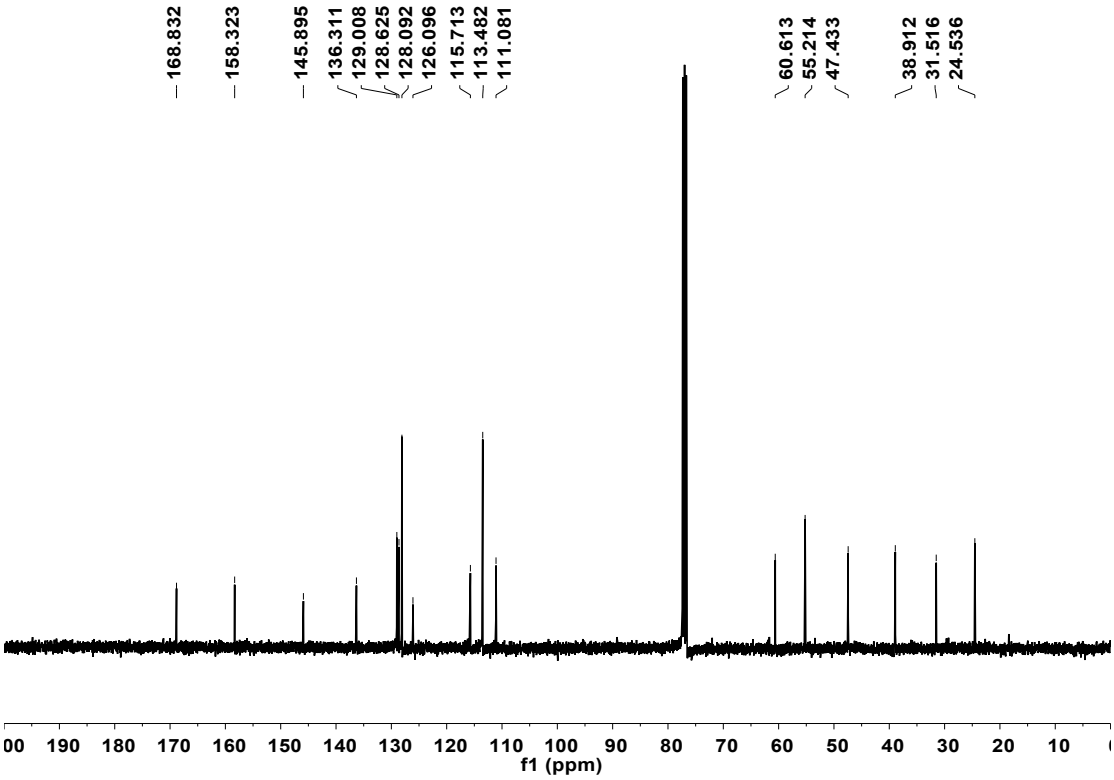

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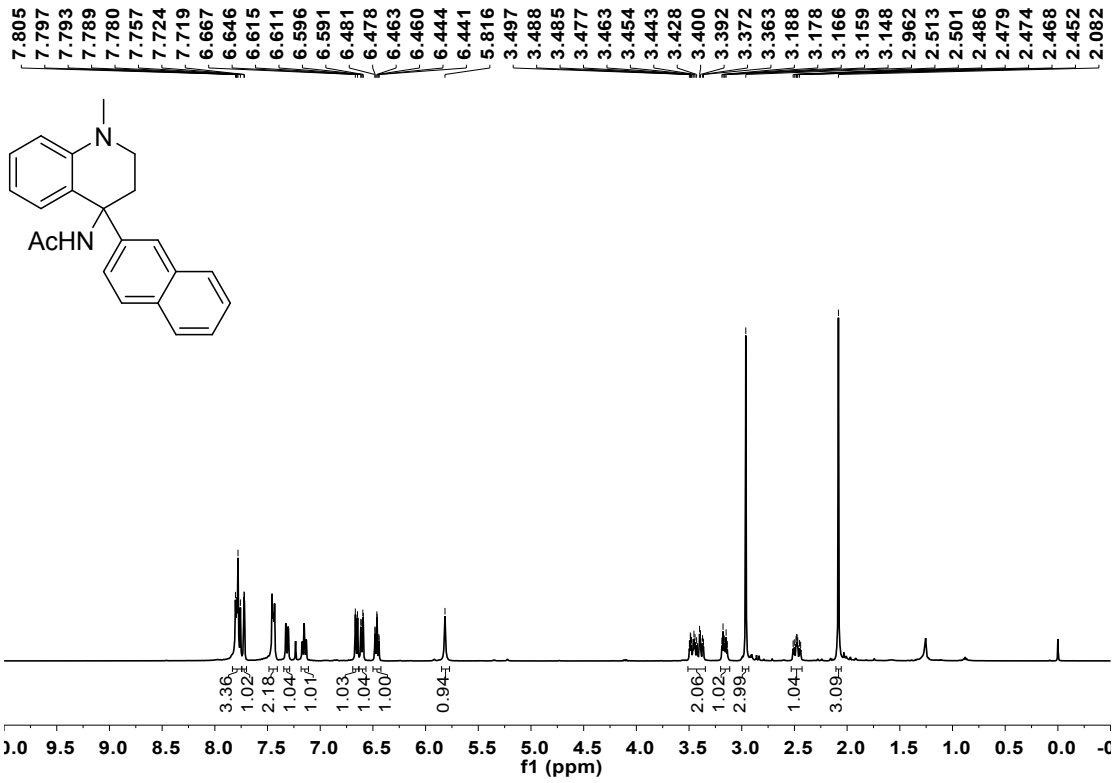

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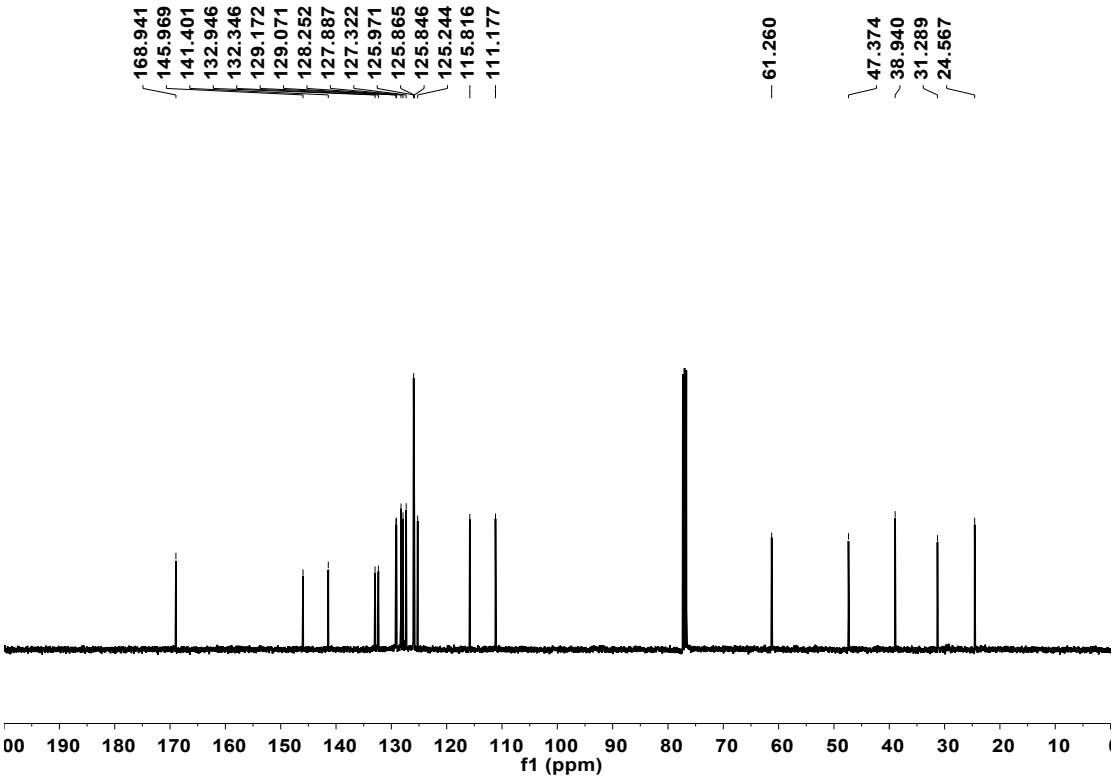

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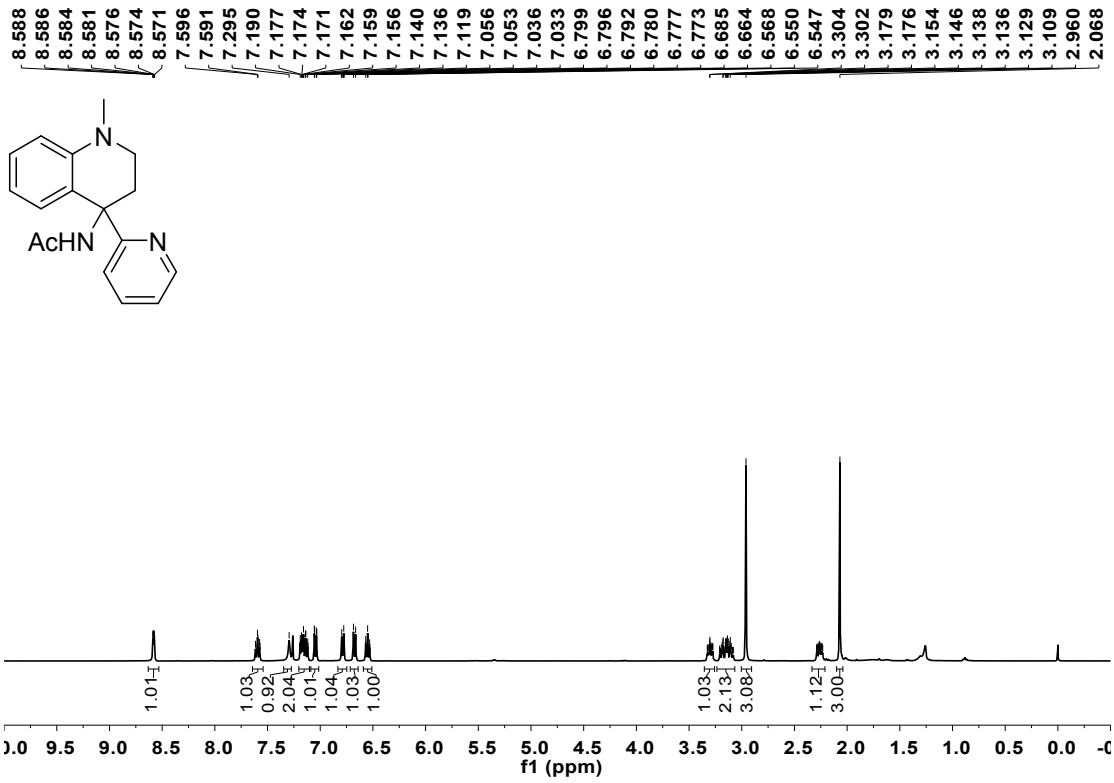

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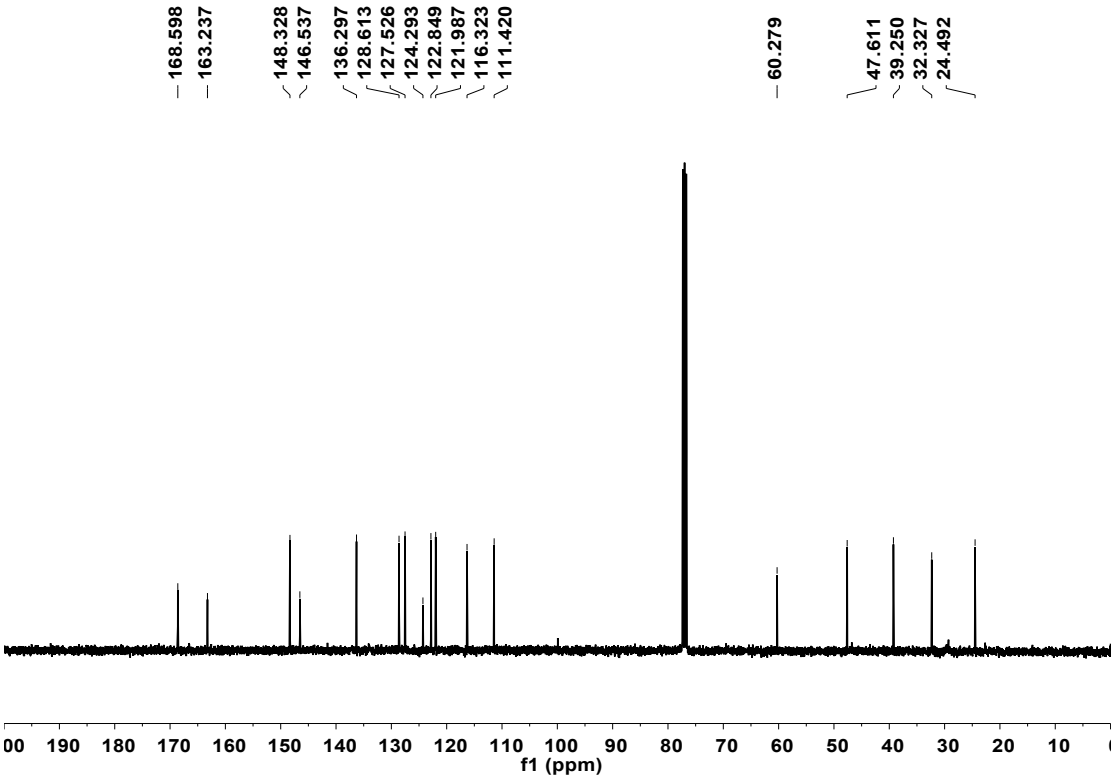

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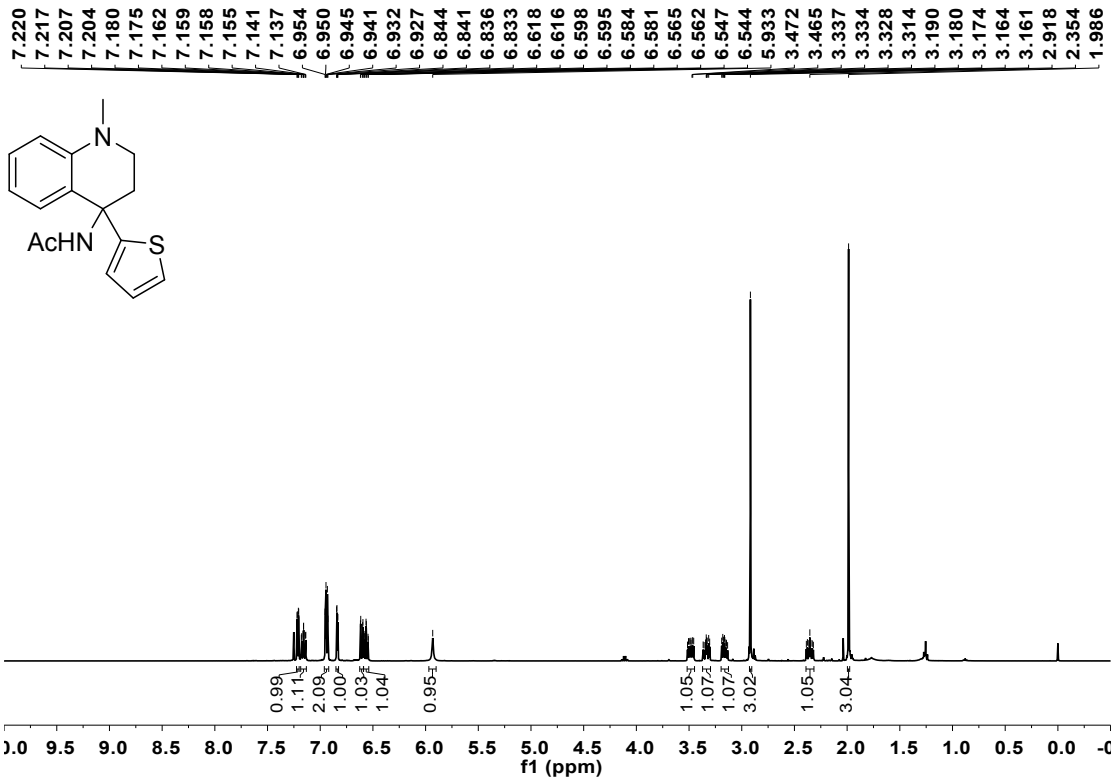

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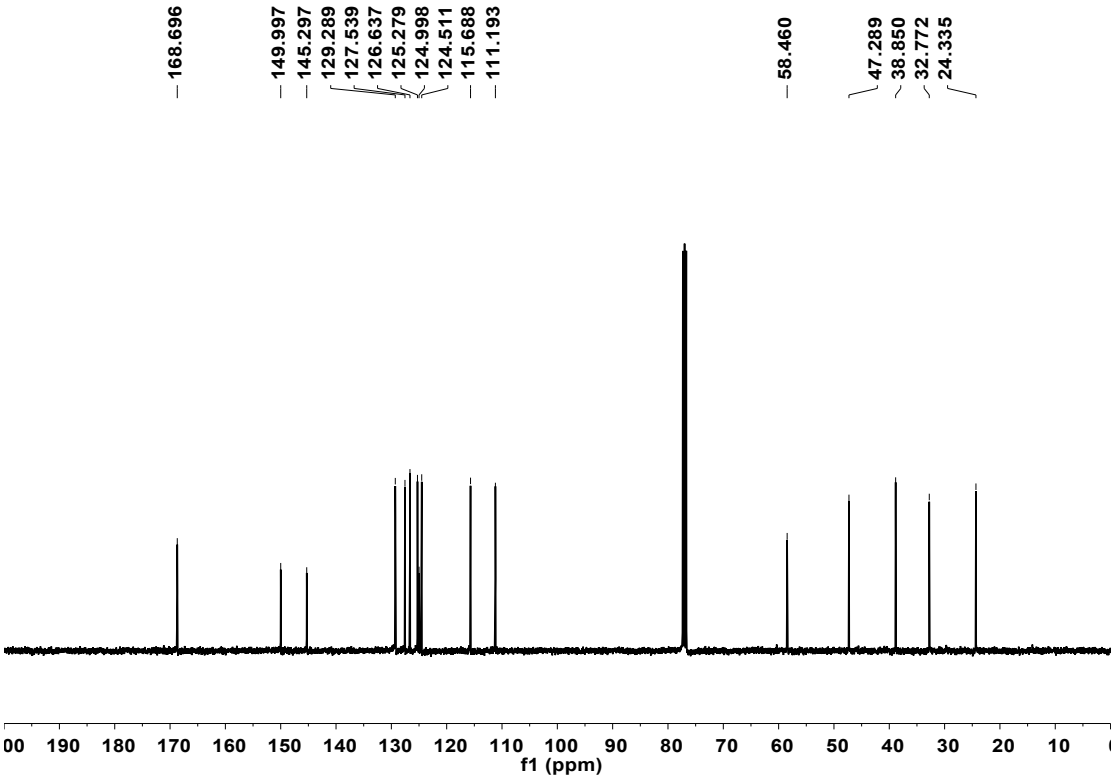

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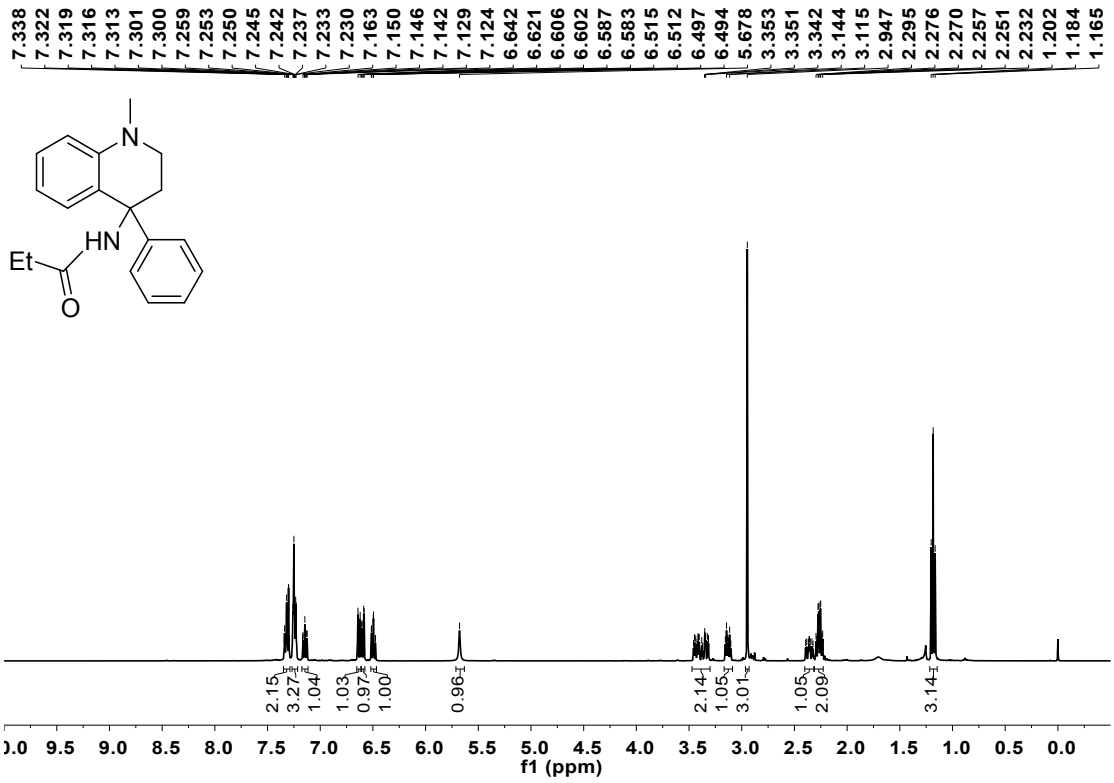

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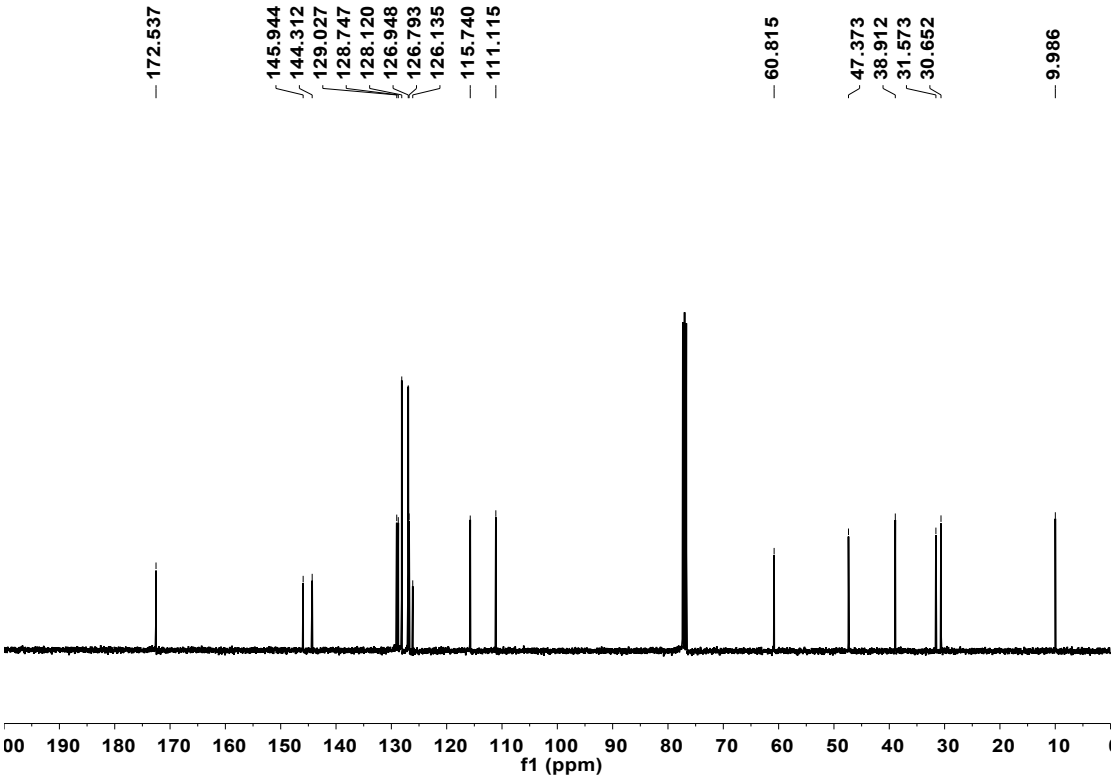

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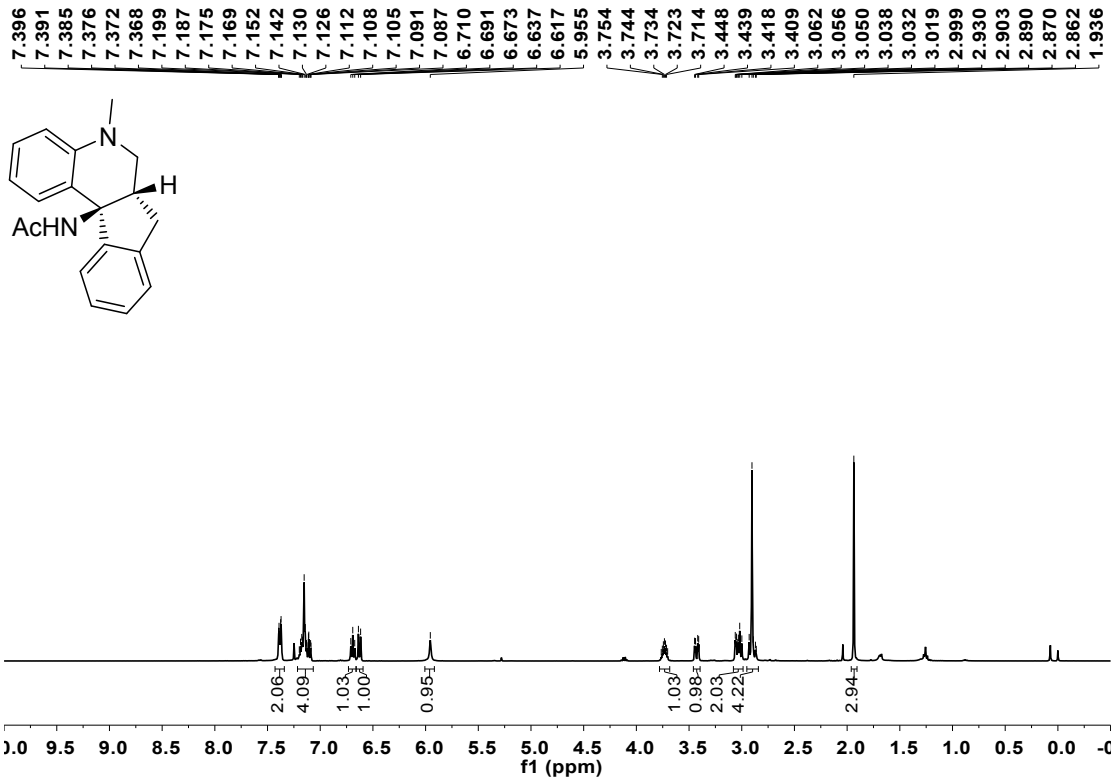

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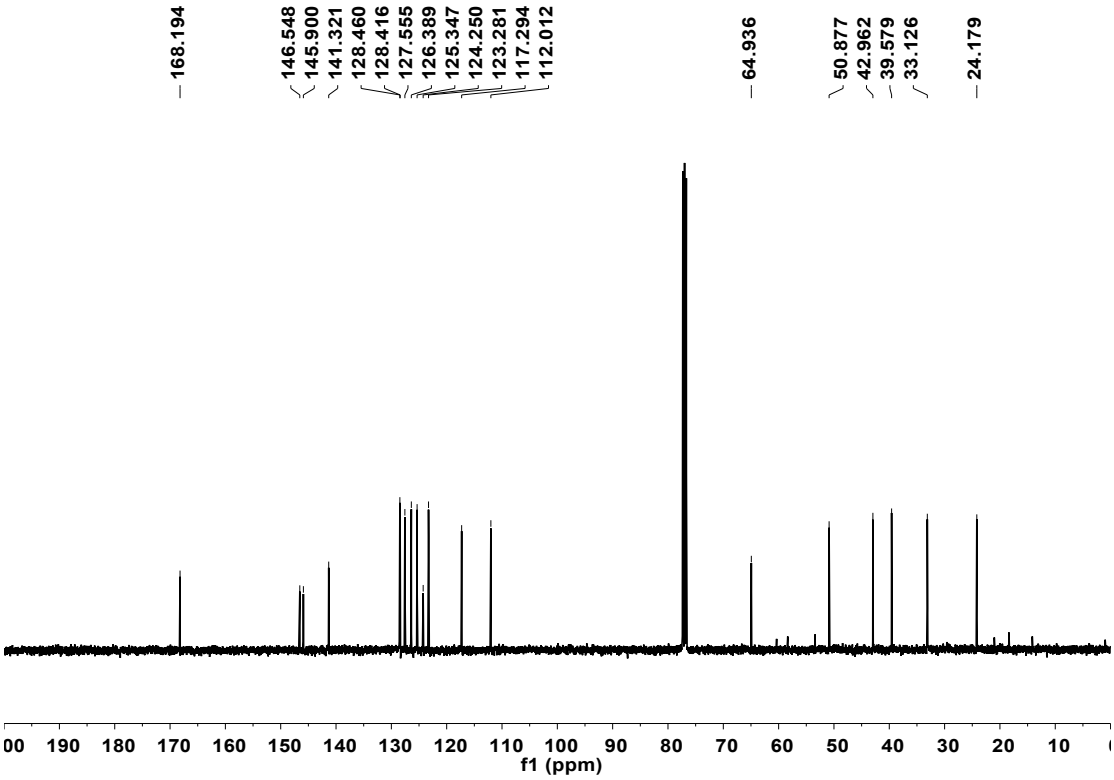

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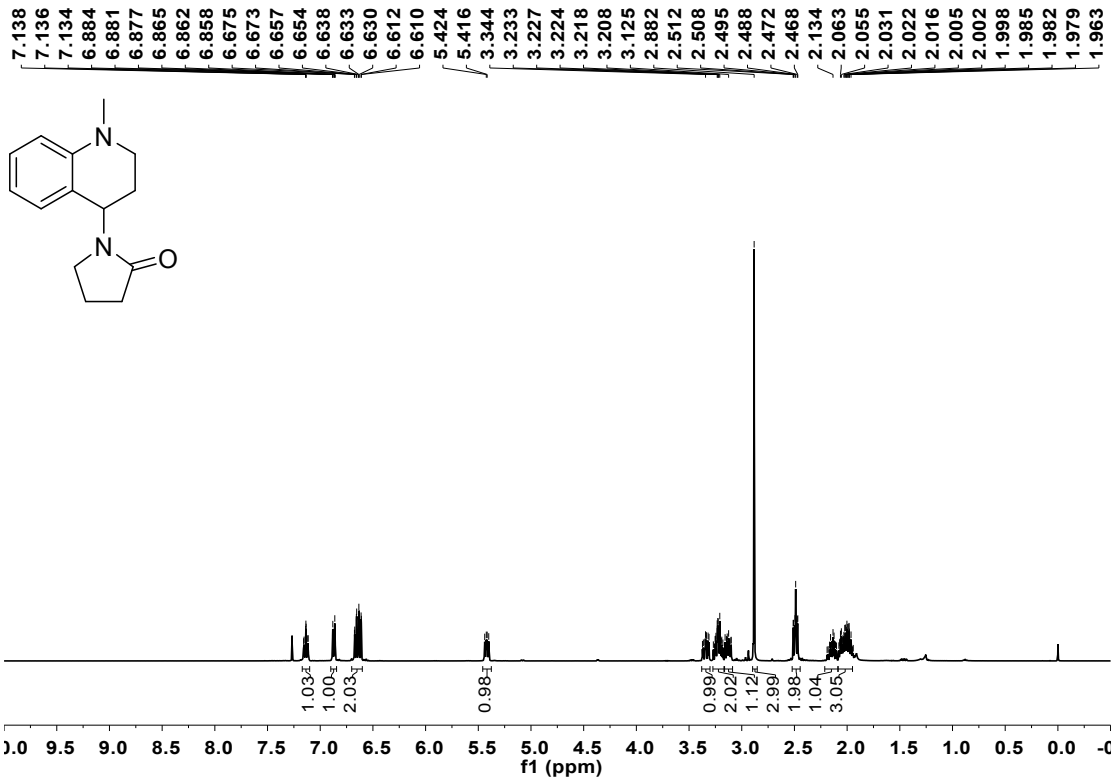
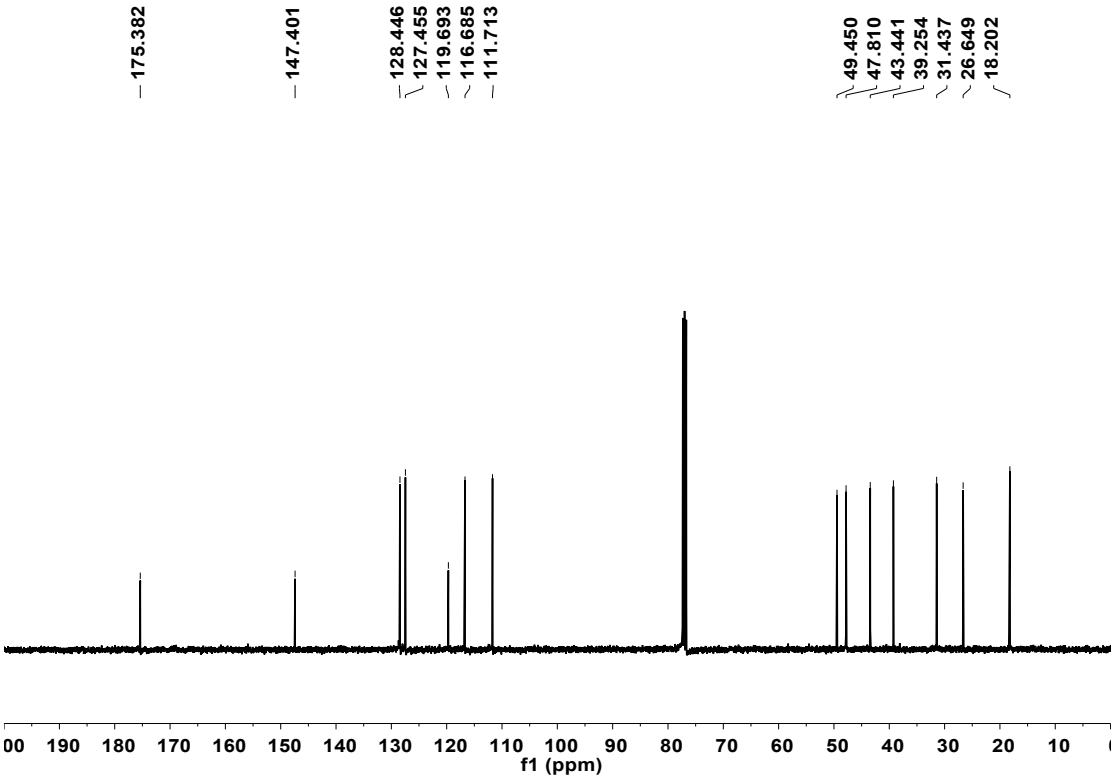


^{13}C NMR



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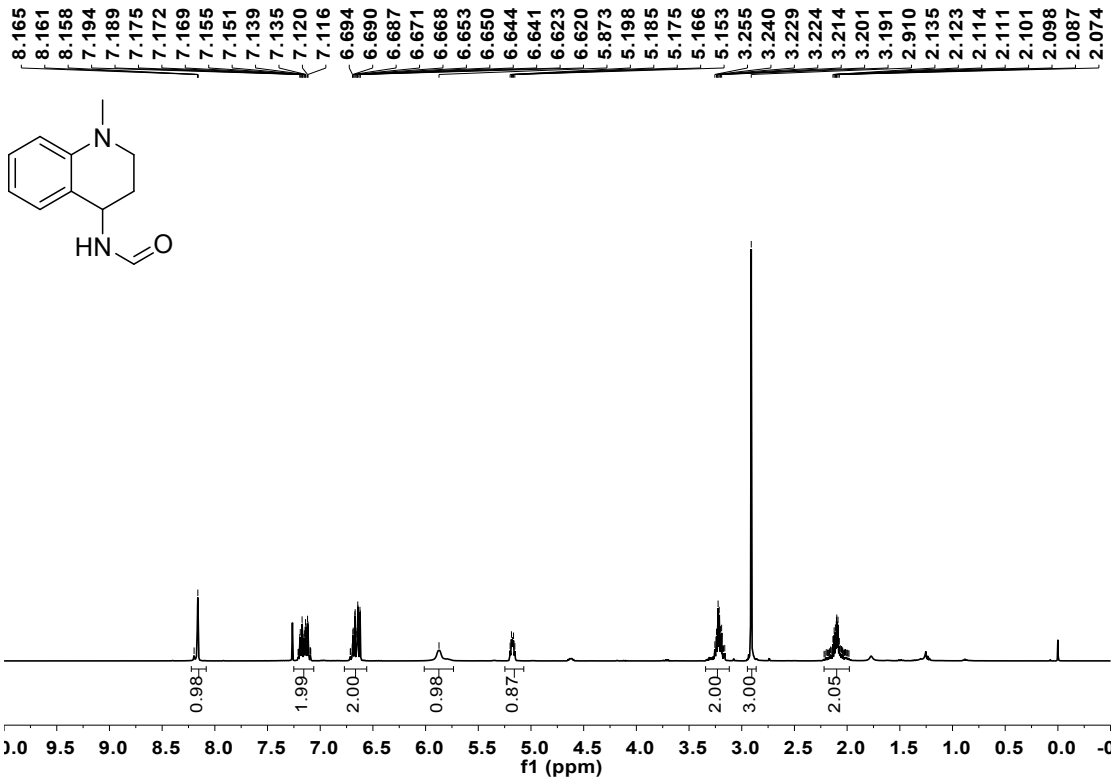

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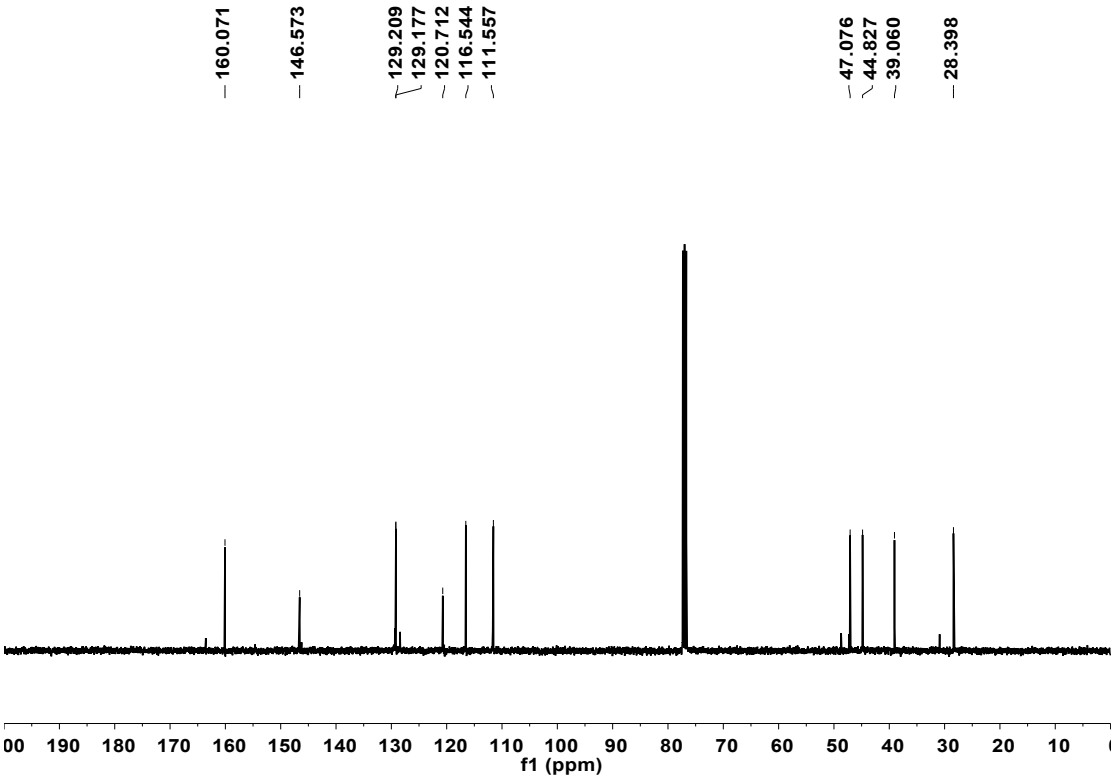

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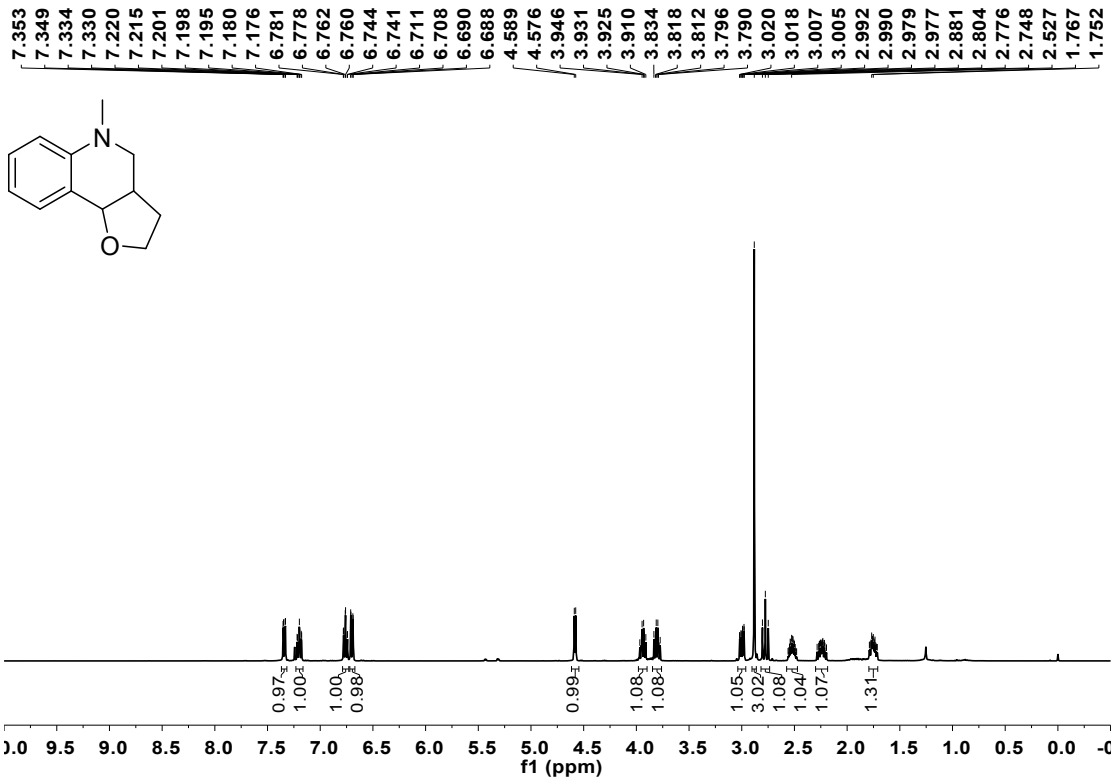

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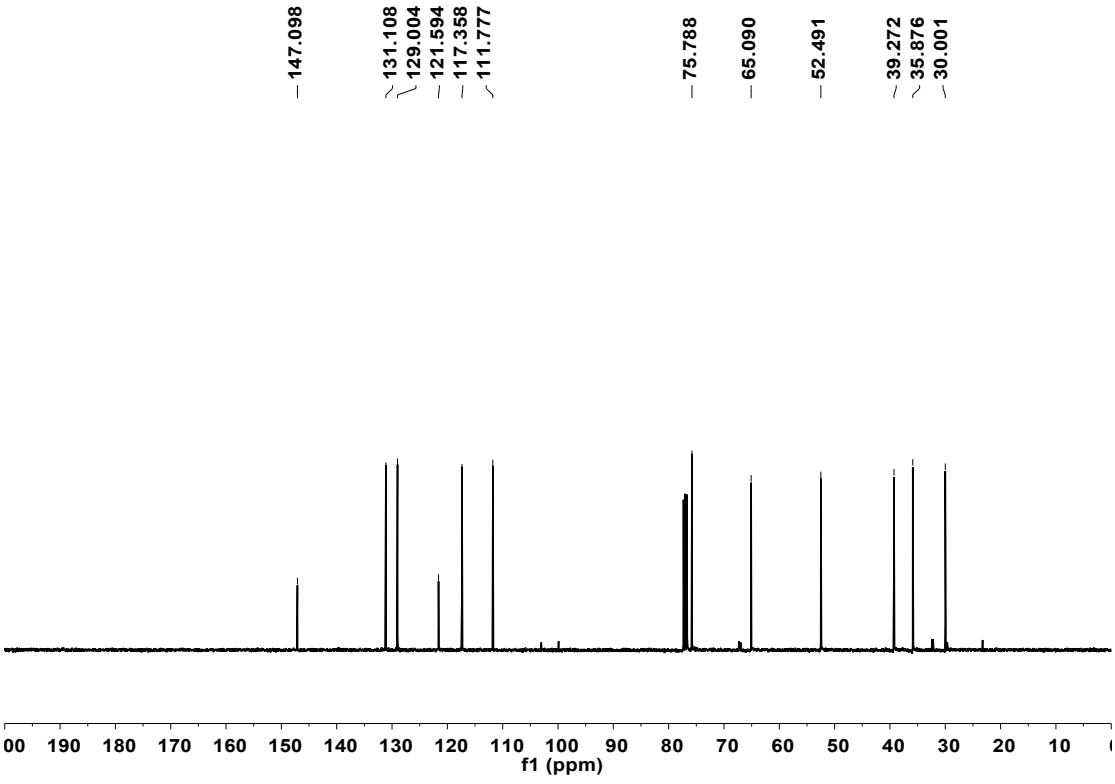

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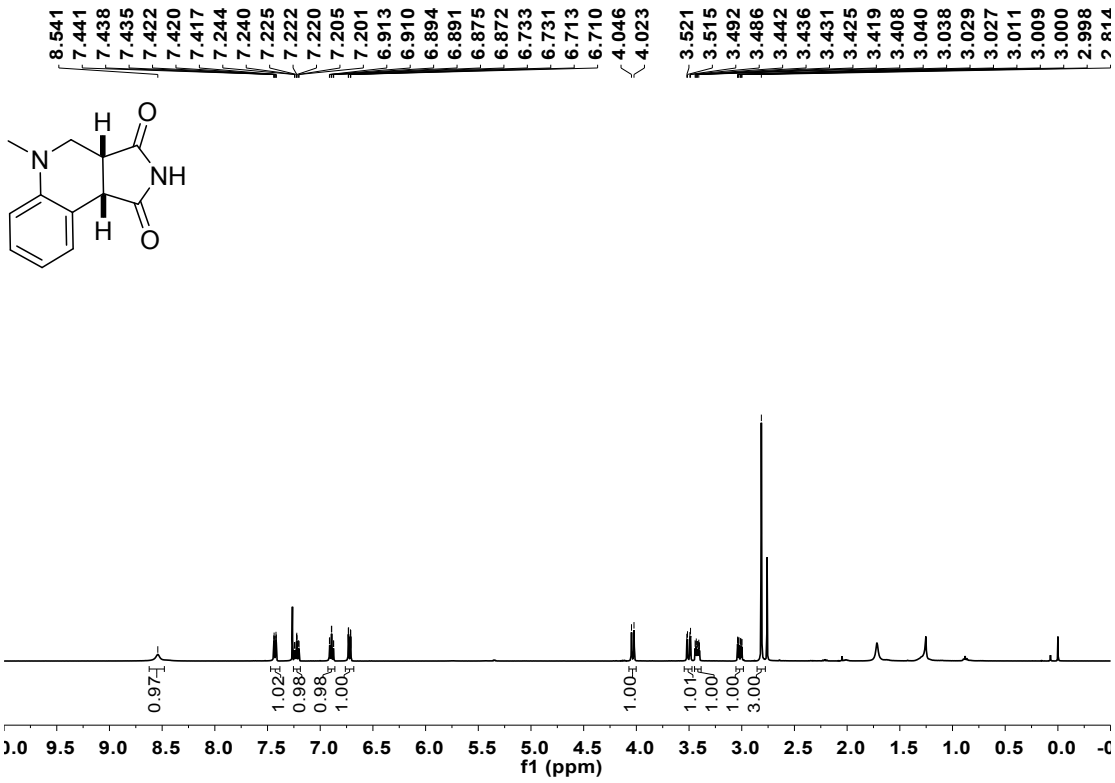

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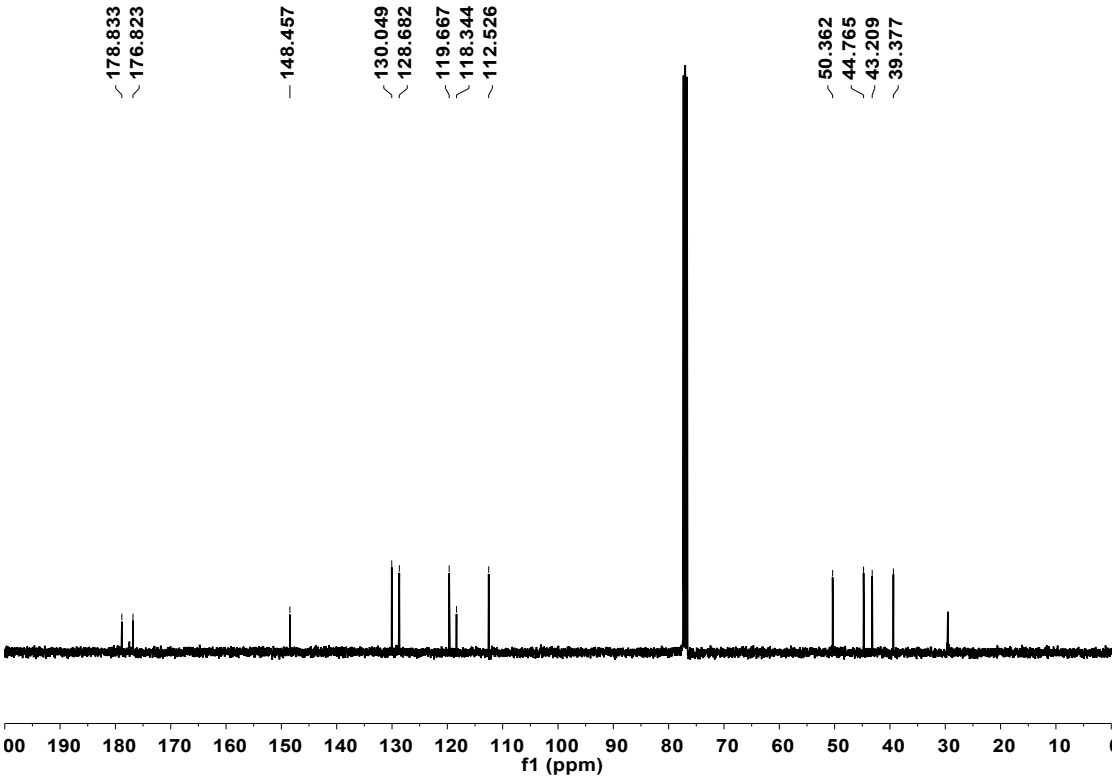

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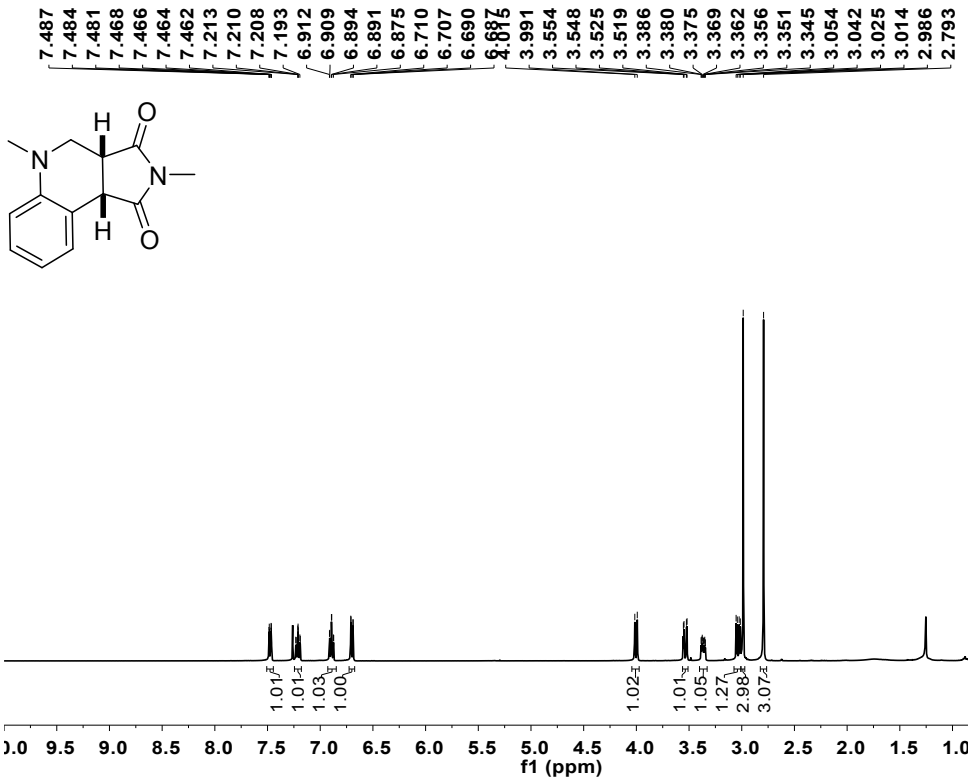

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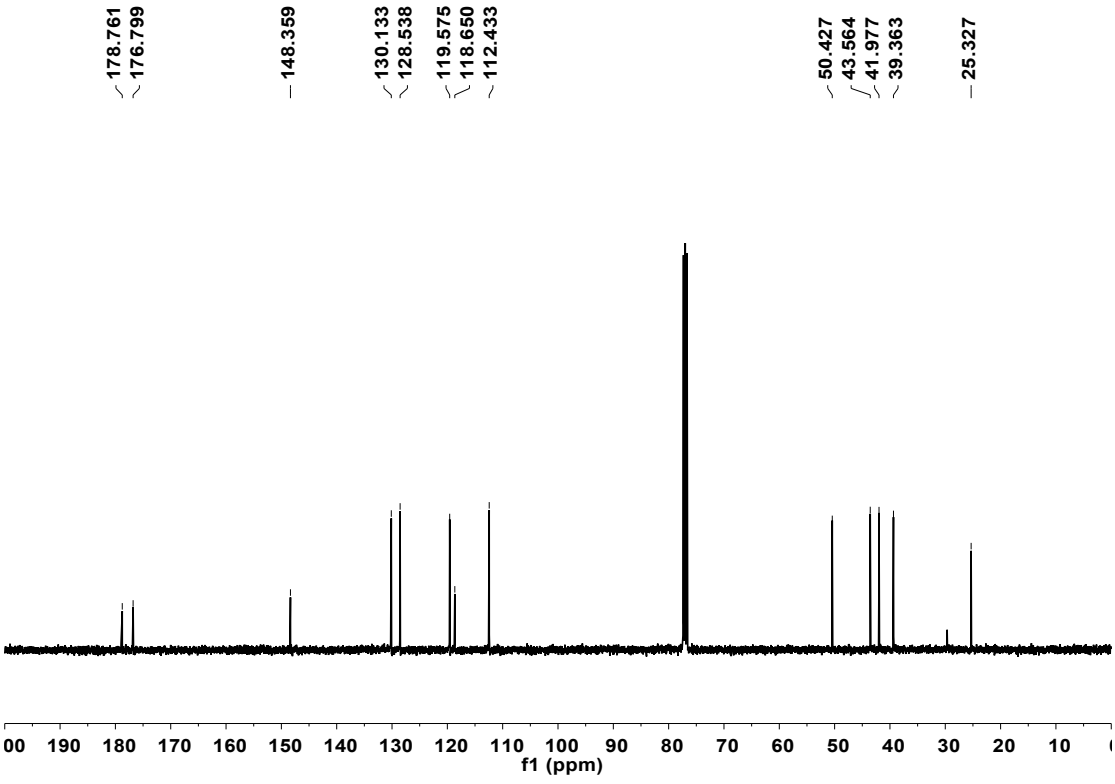

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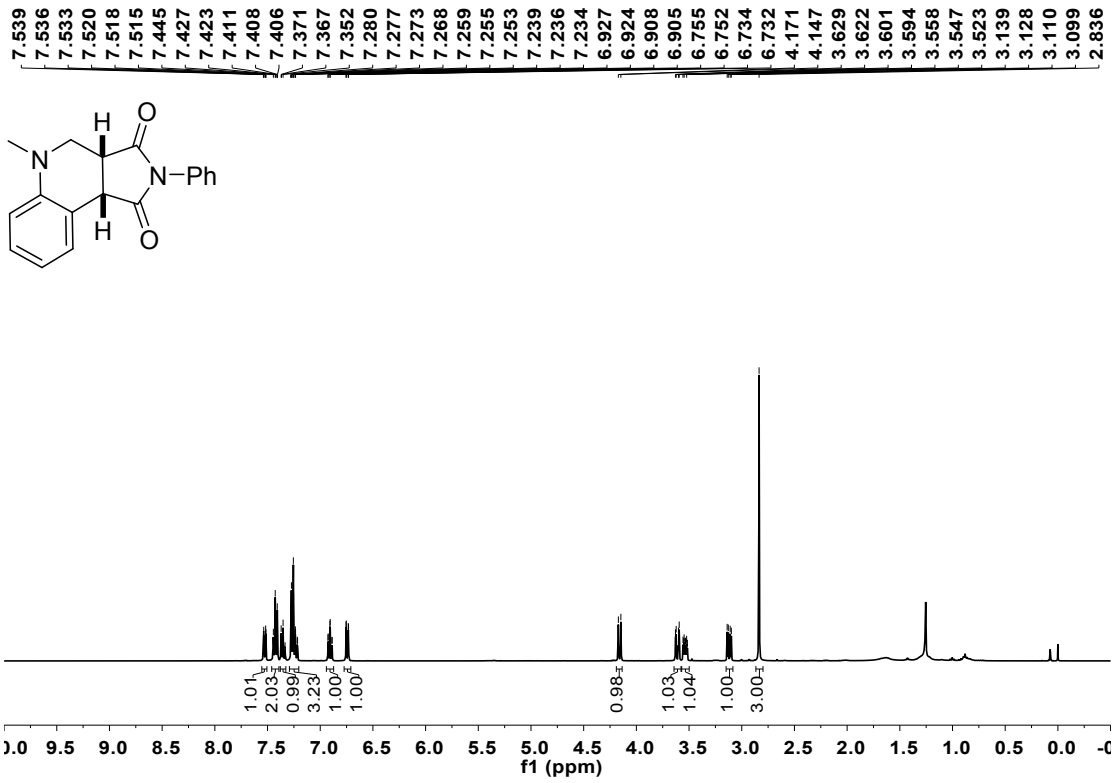

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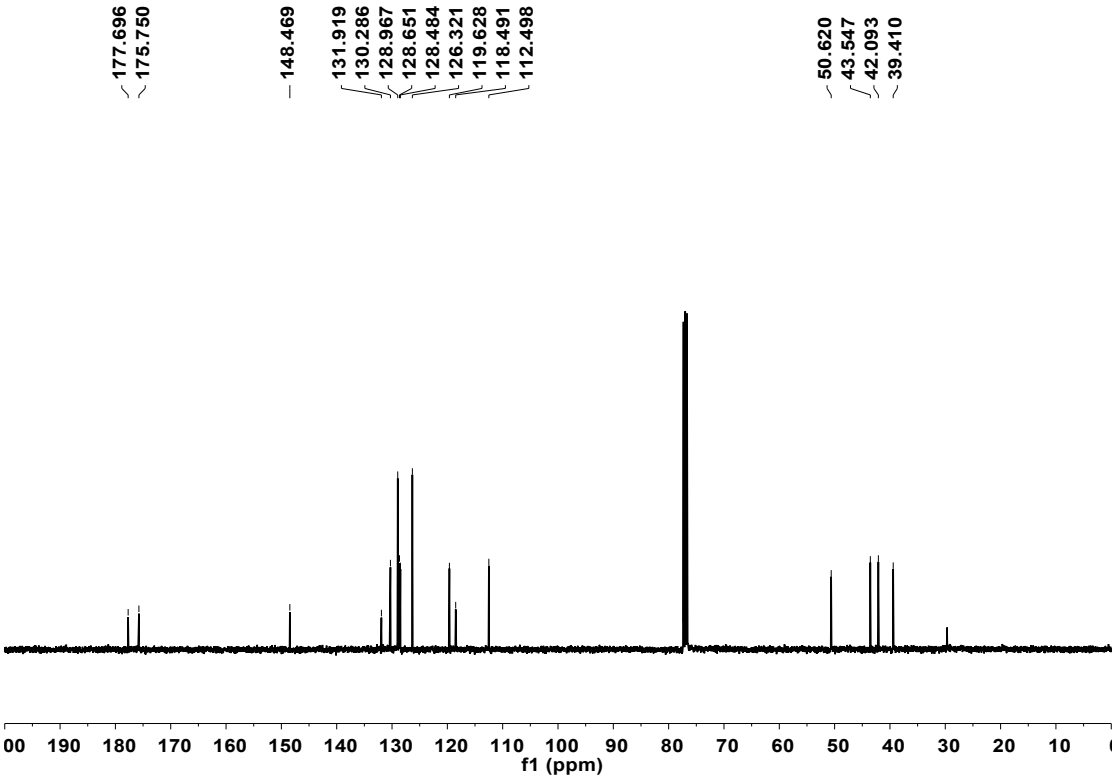

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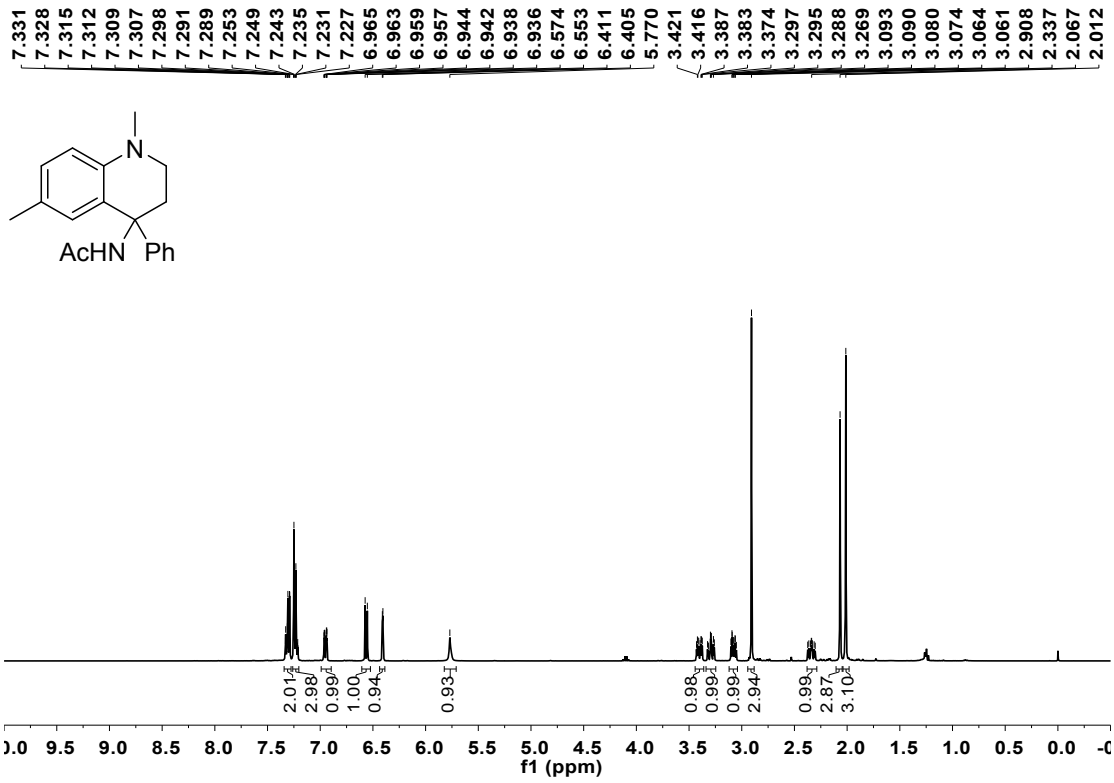
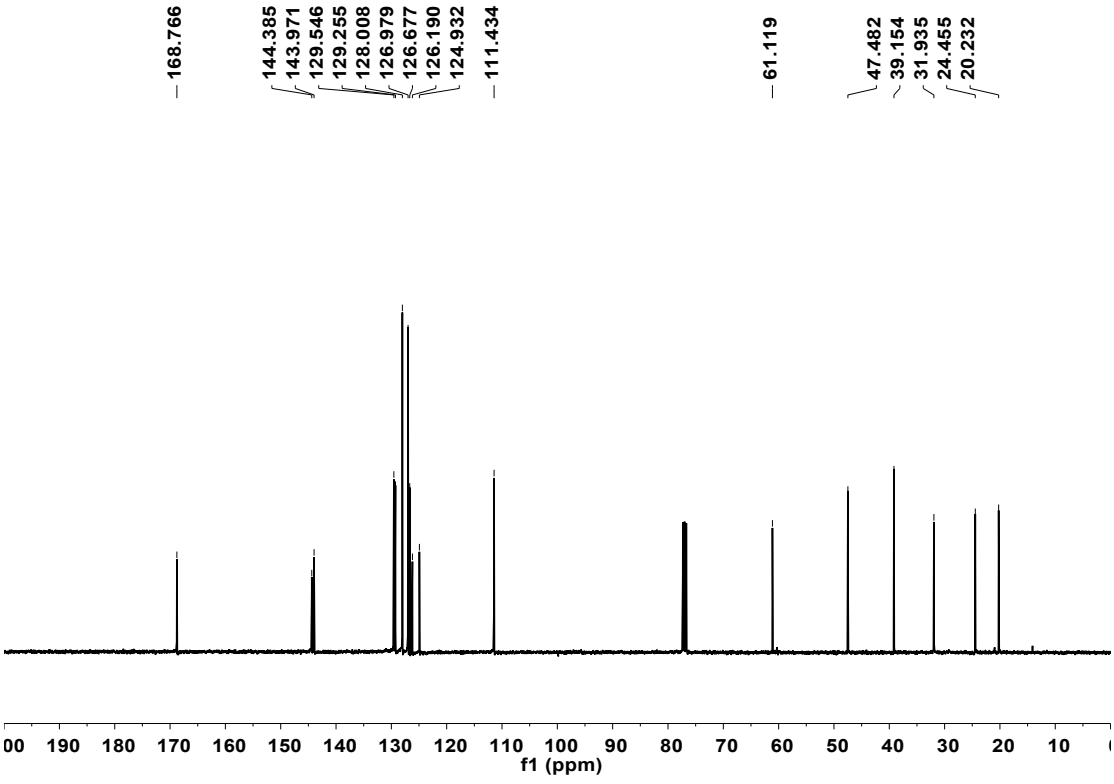
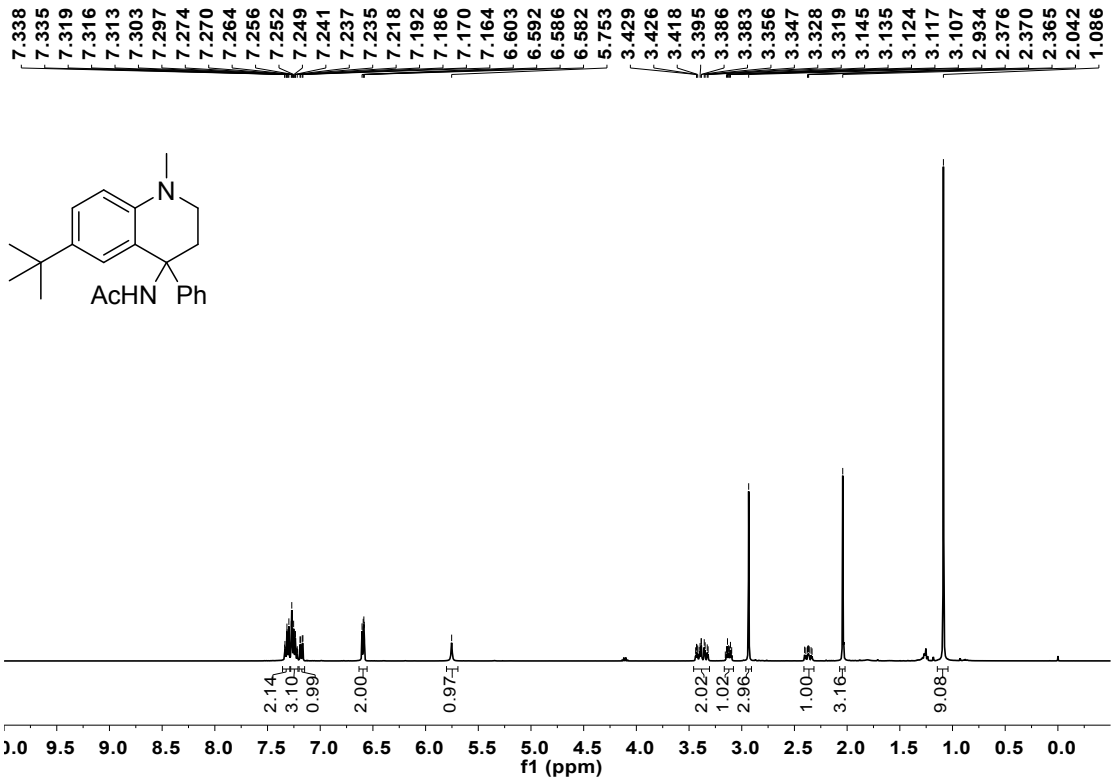


^{13}C NMR

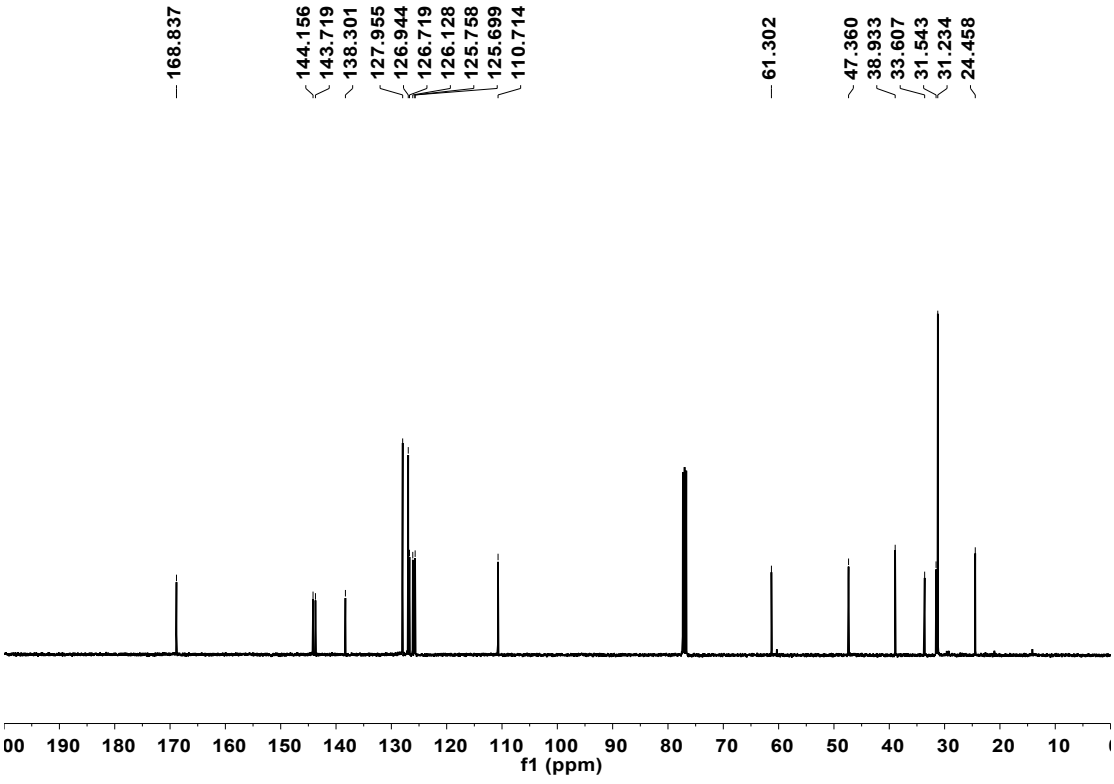


3ca

^{1}H NMR

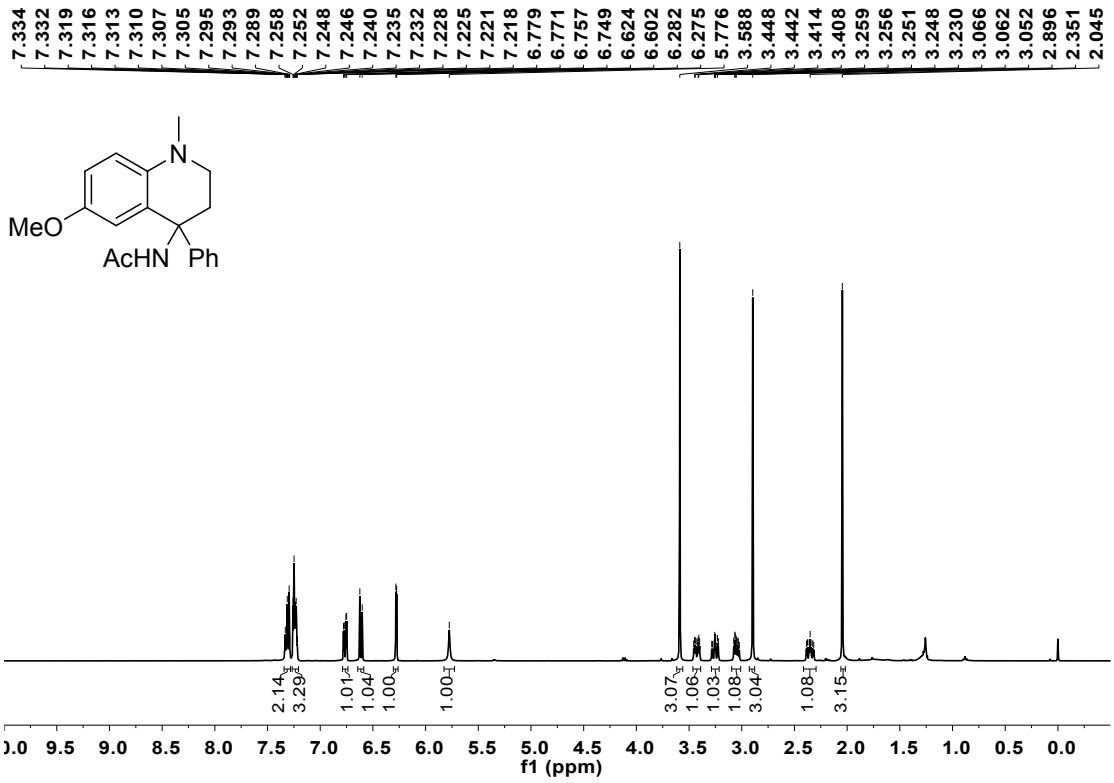


^{13}C NMR

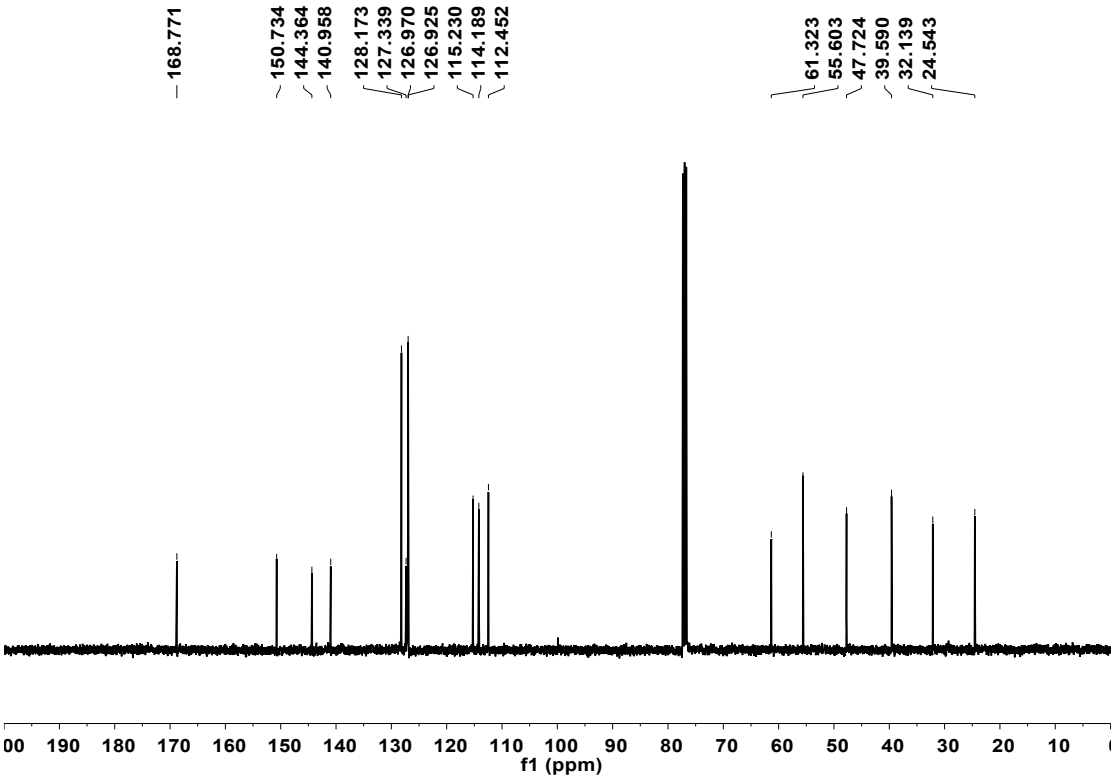


3da

^{1}H NMR

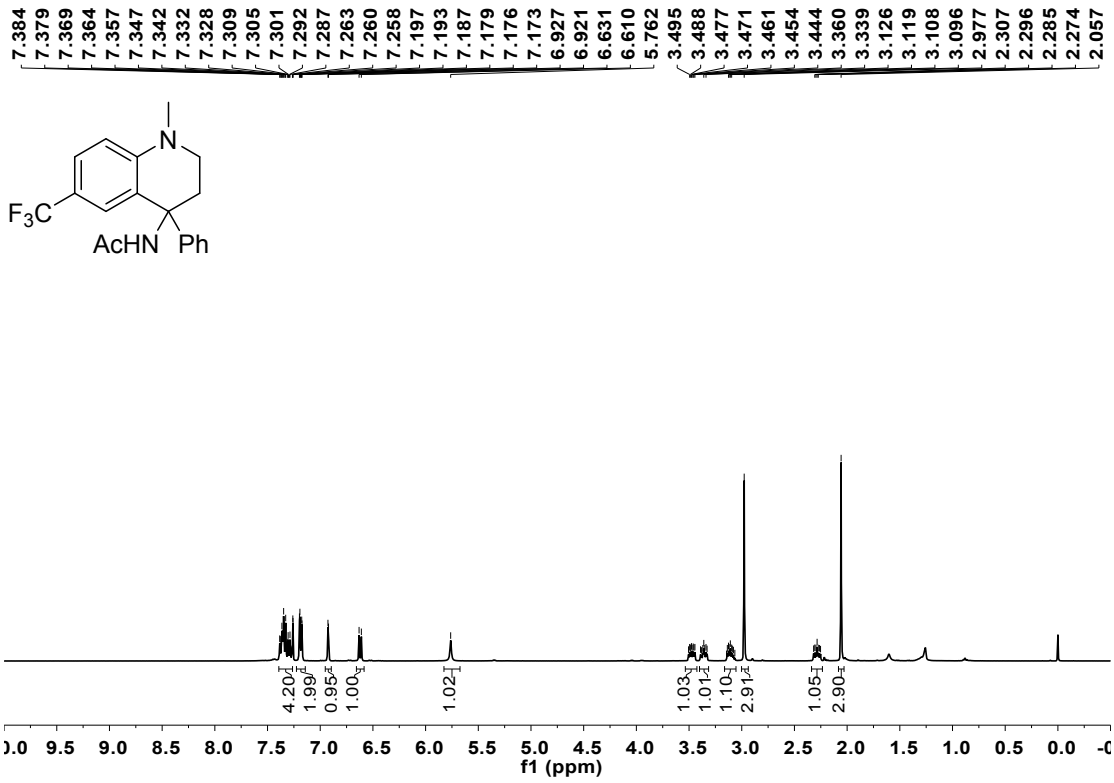


^{13}C NMR

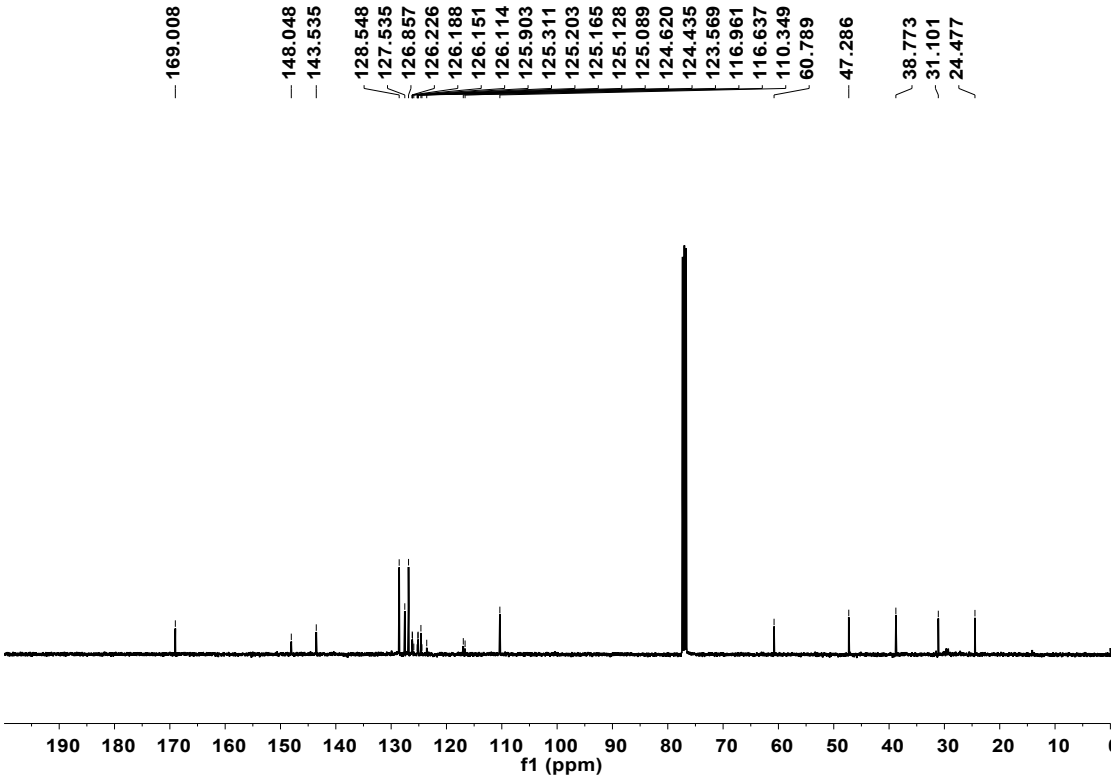


3ea

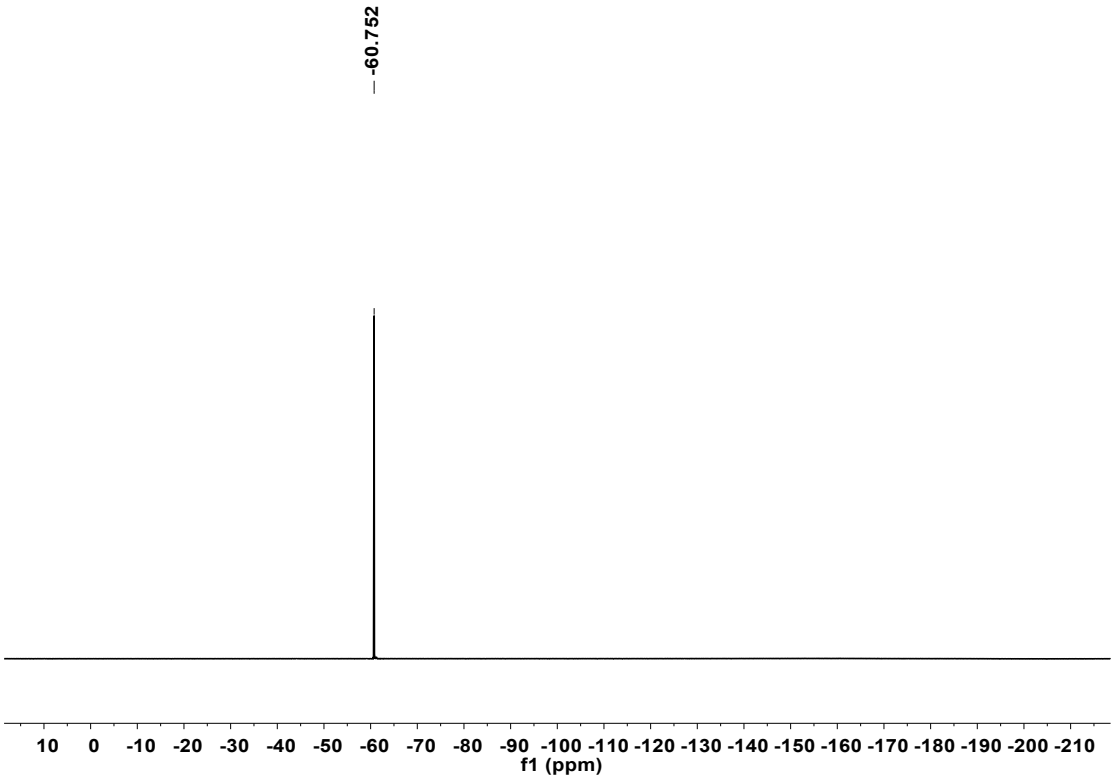
^{1}H NMR



^{13}C NMR

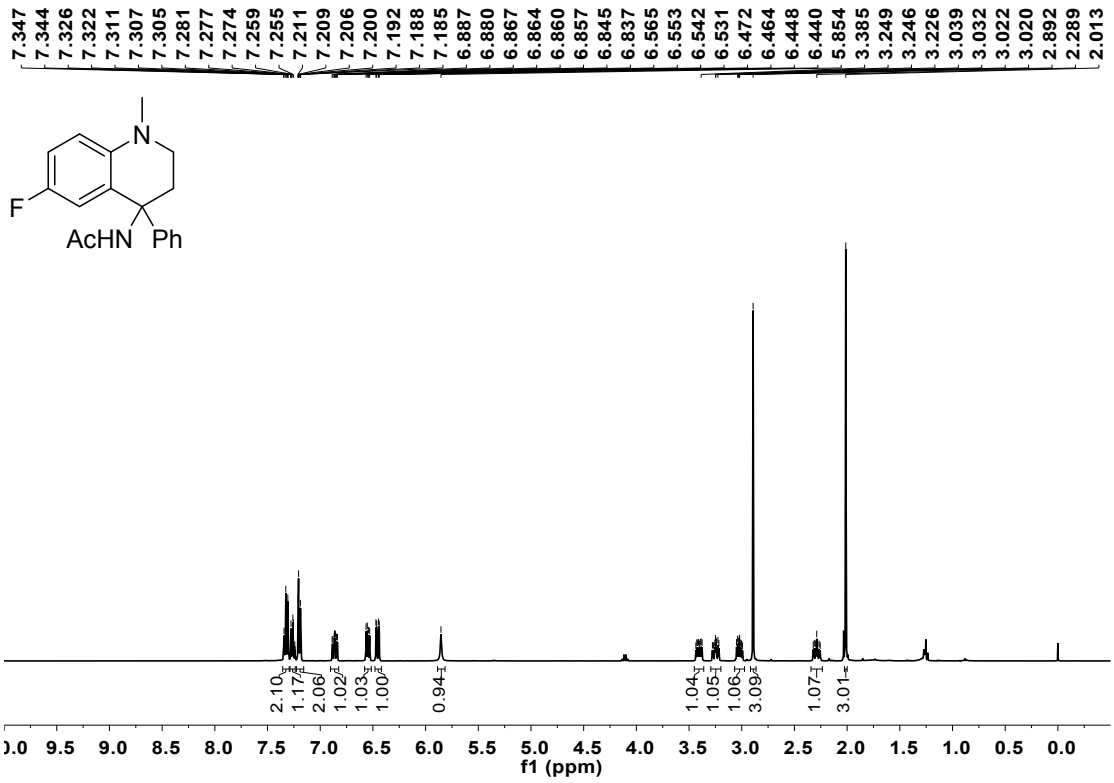


^{19}F NMR

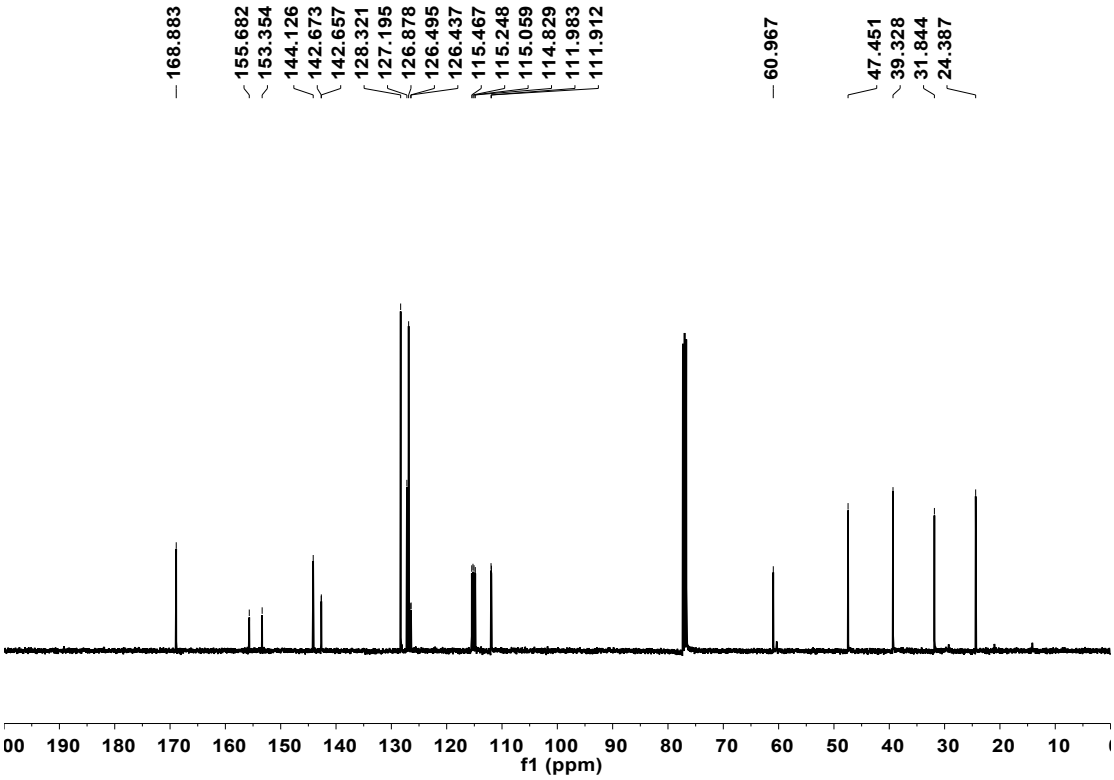

-60.752
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

3fa

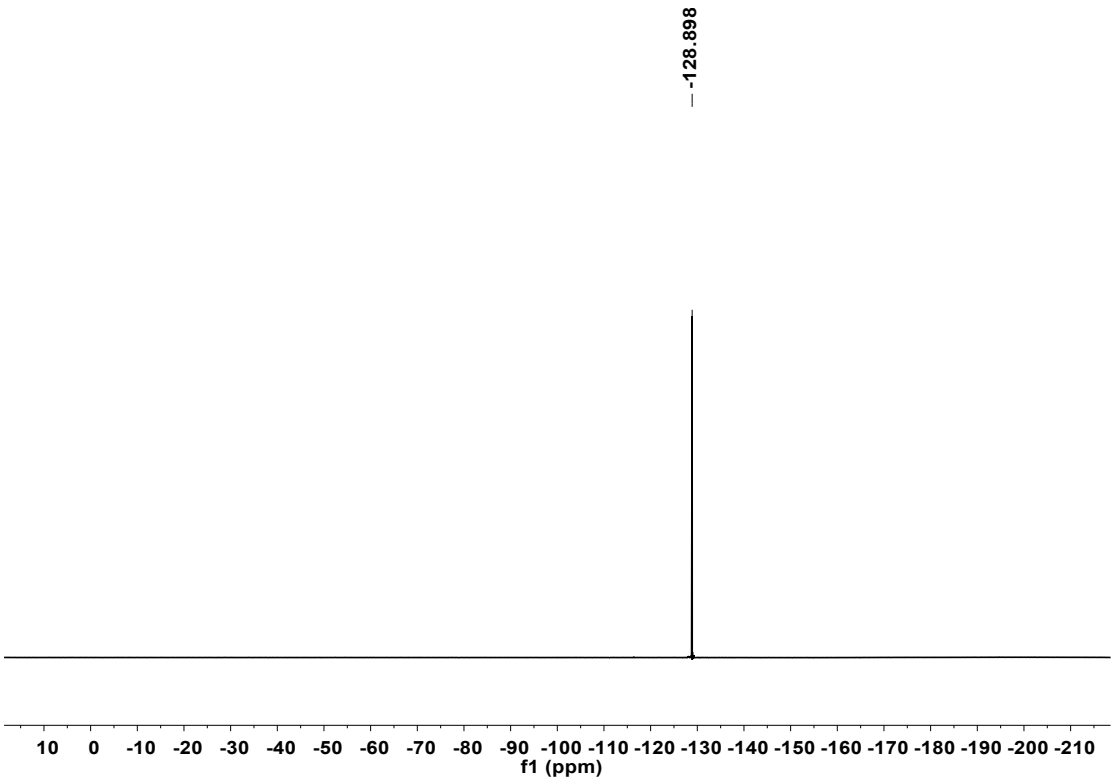
^{1}H NMR



^{13}C NMR

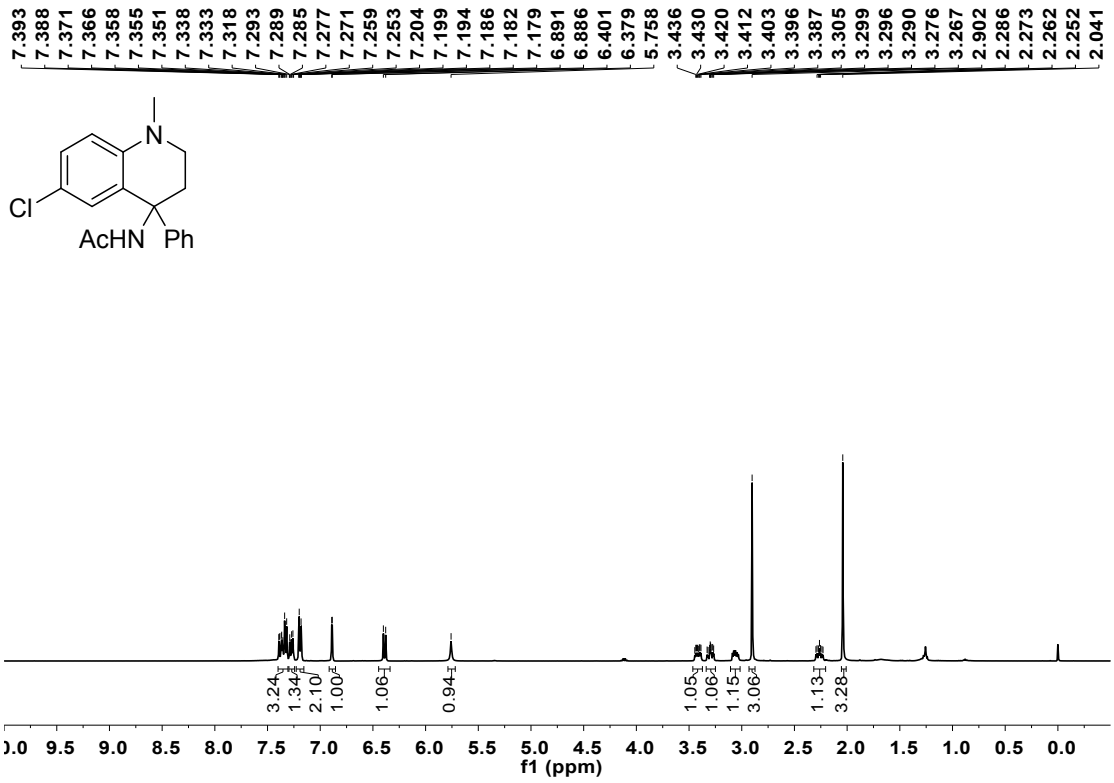


^{19}F NMR

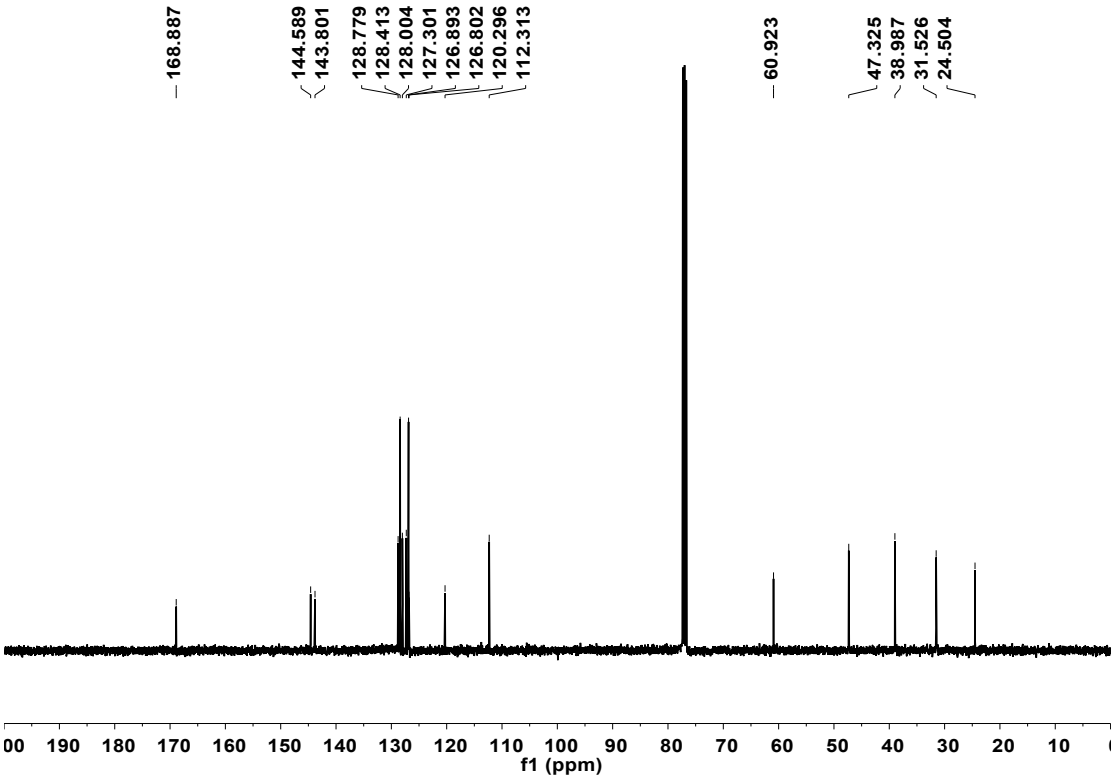

-128.898
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

3ga

^{1}H NMR

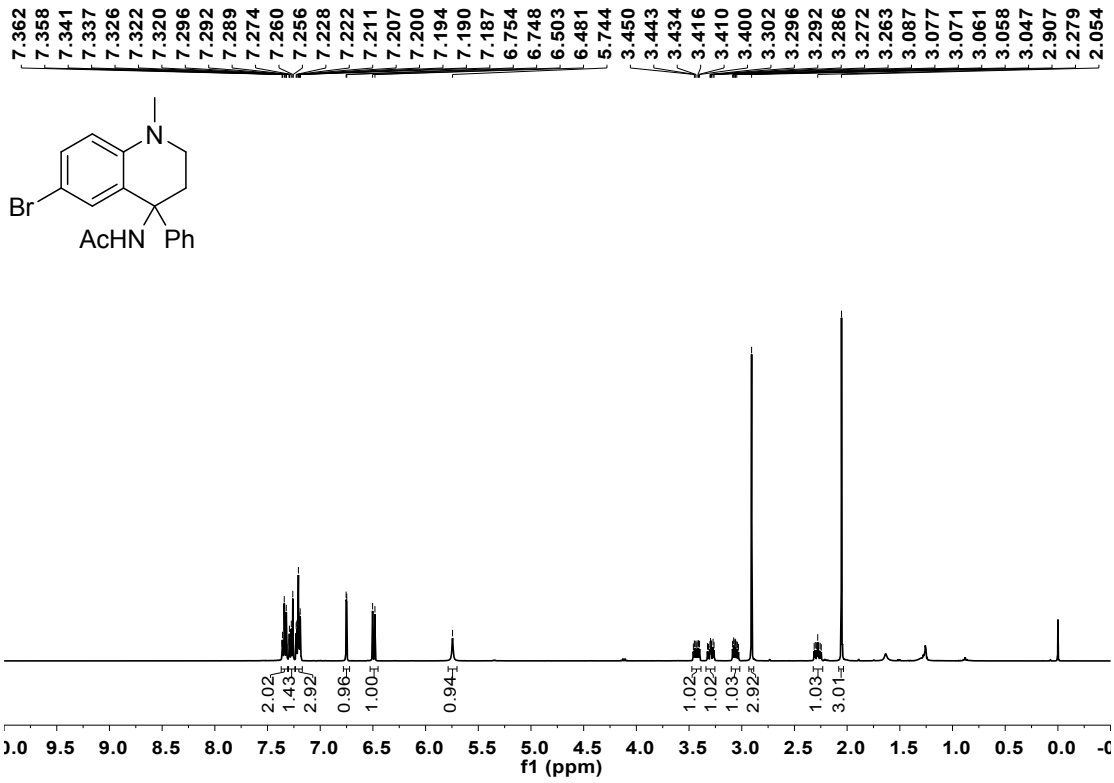


^{13}C NMR

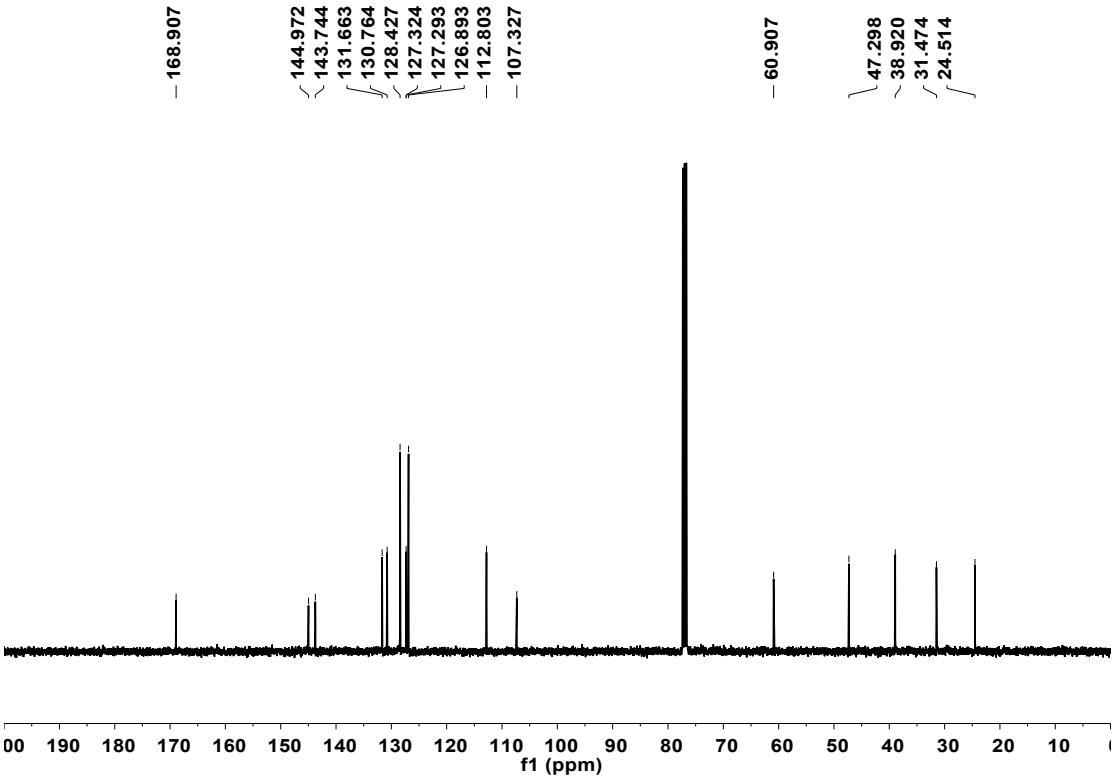


3ha

^{1}H NMR

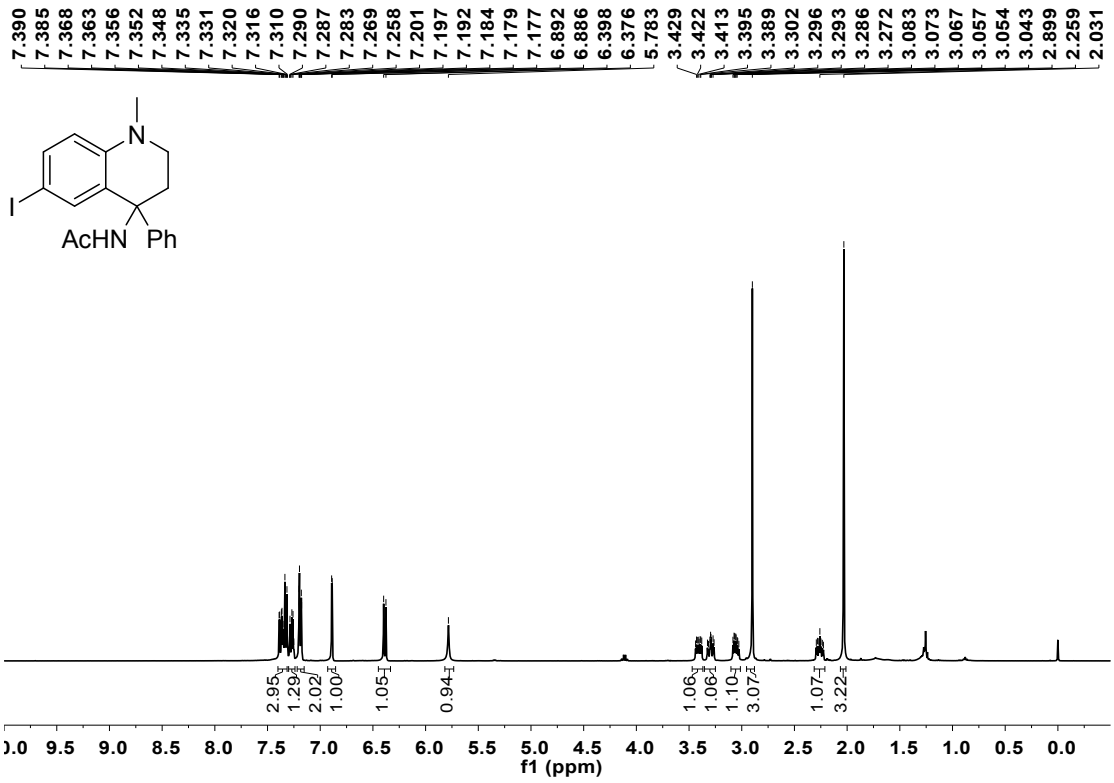


^{13}C NMR

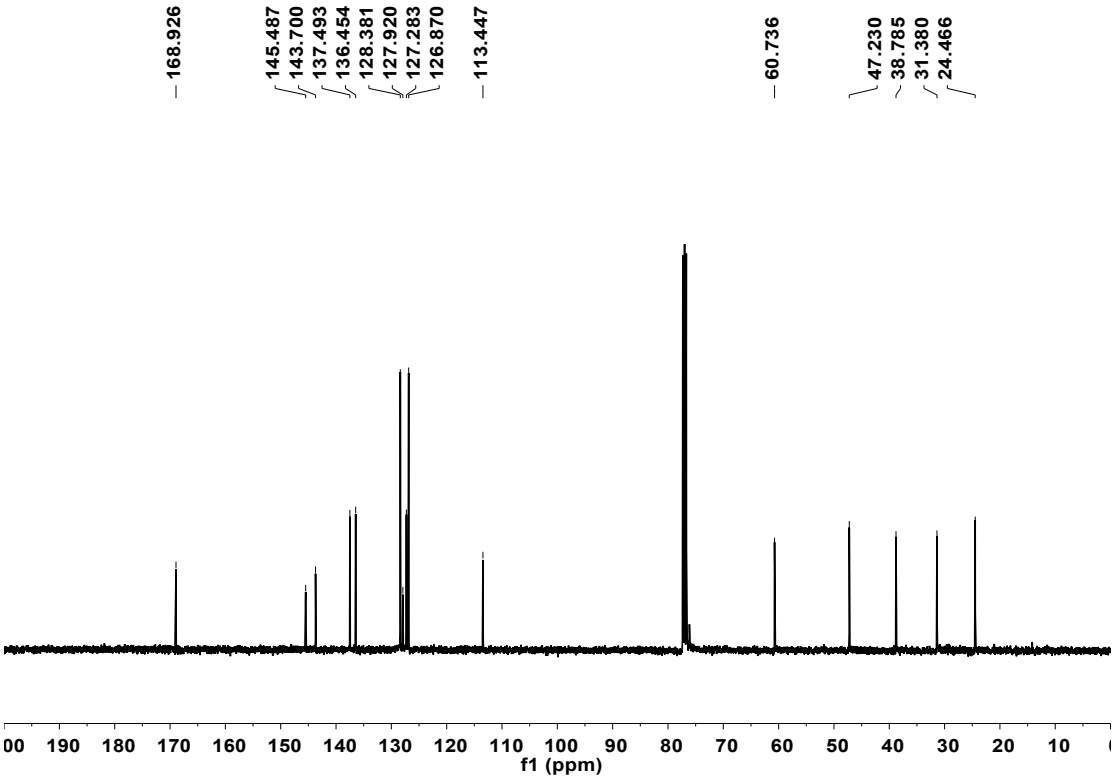


3ia

^{1}H NMR

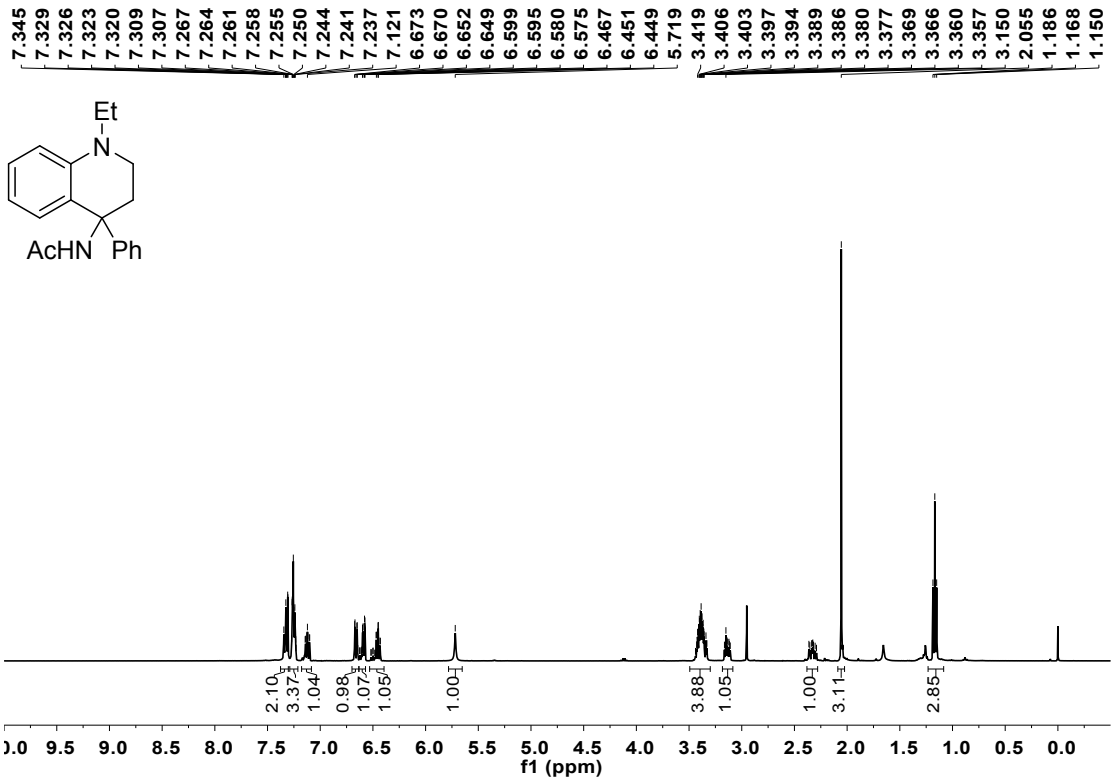


^{13}C NMR

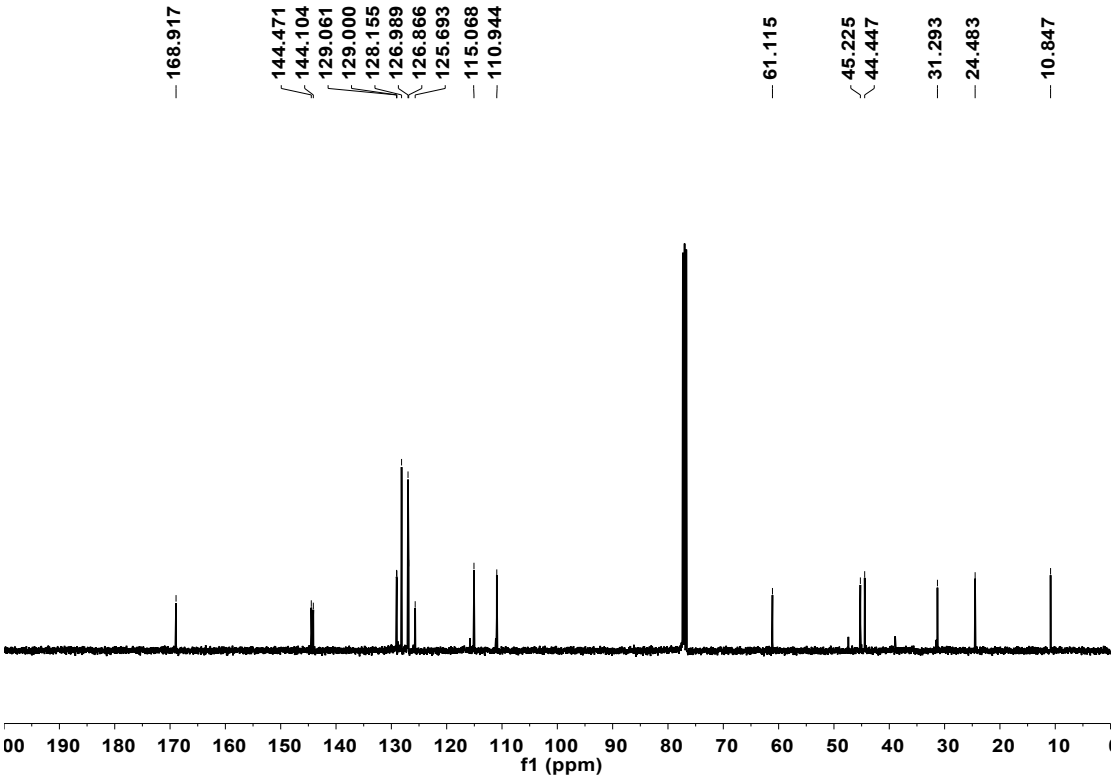


3ja

^{1}H NMR

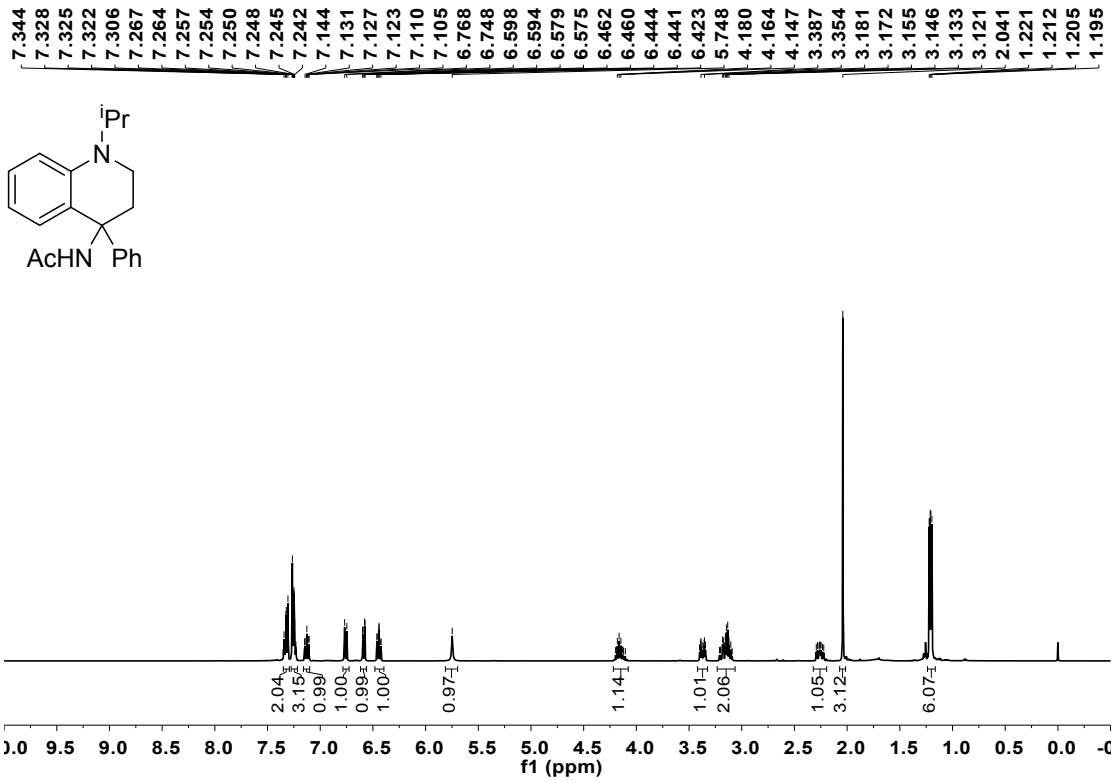


^{13}C NMR

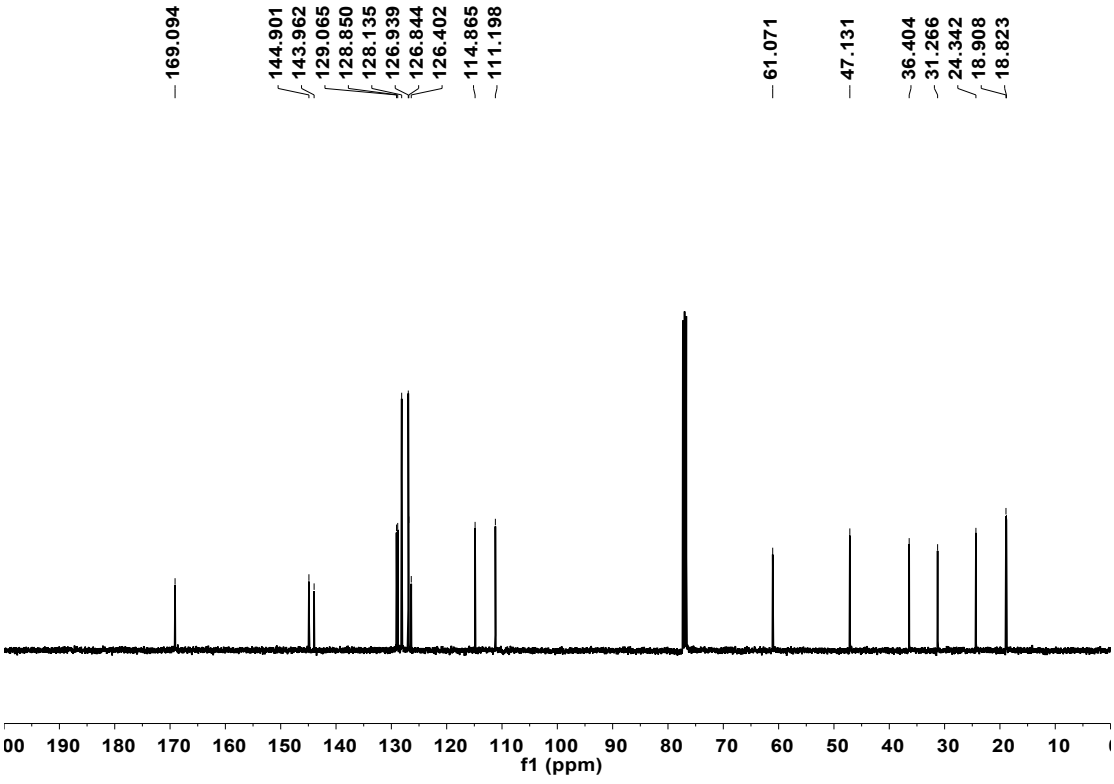


3ka

^{1}H NMR

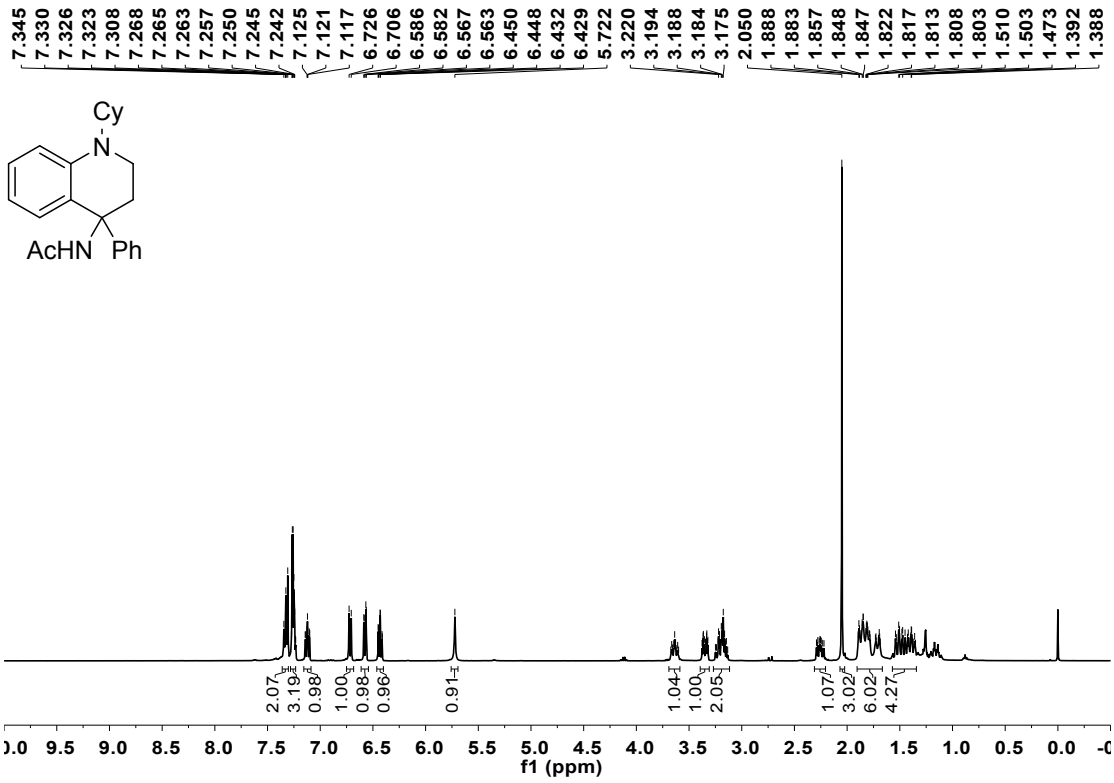


^{13}C NMR

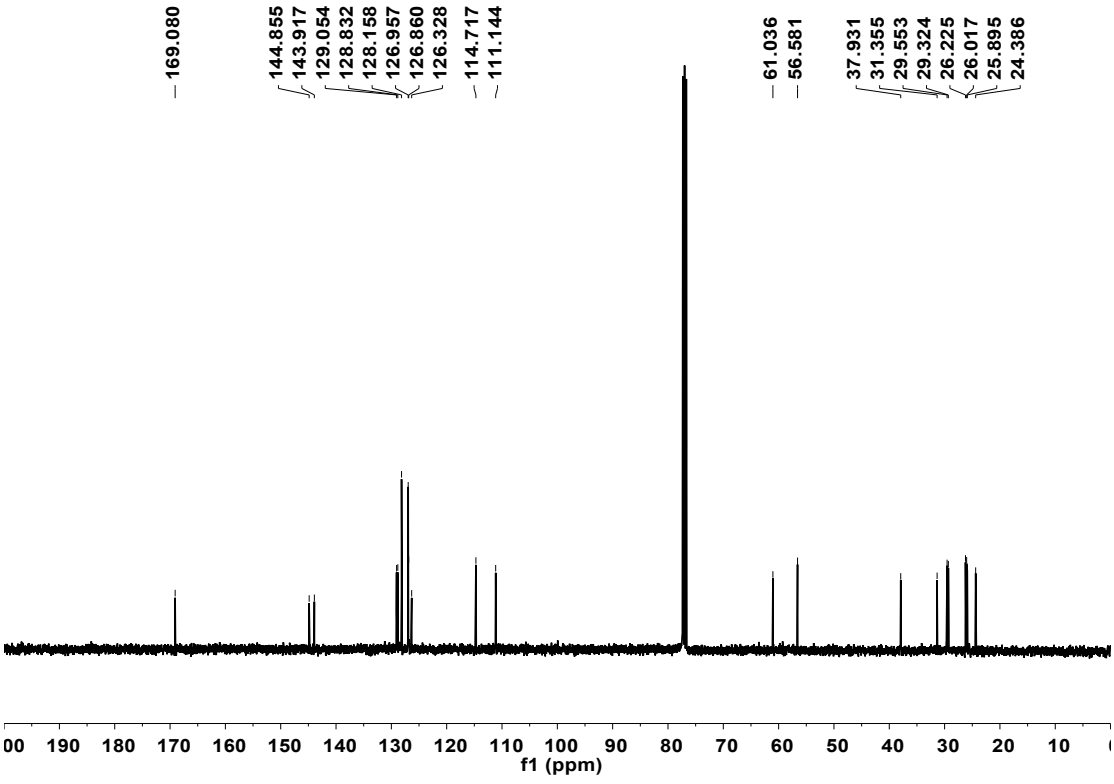


3la

^{1}H NMR

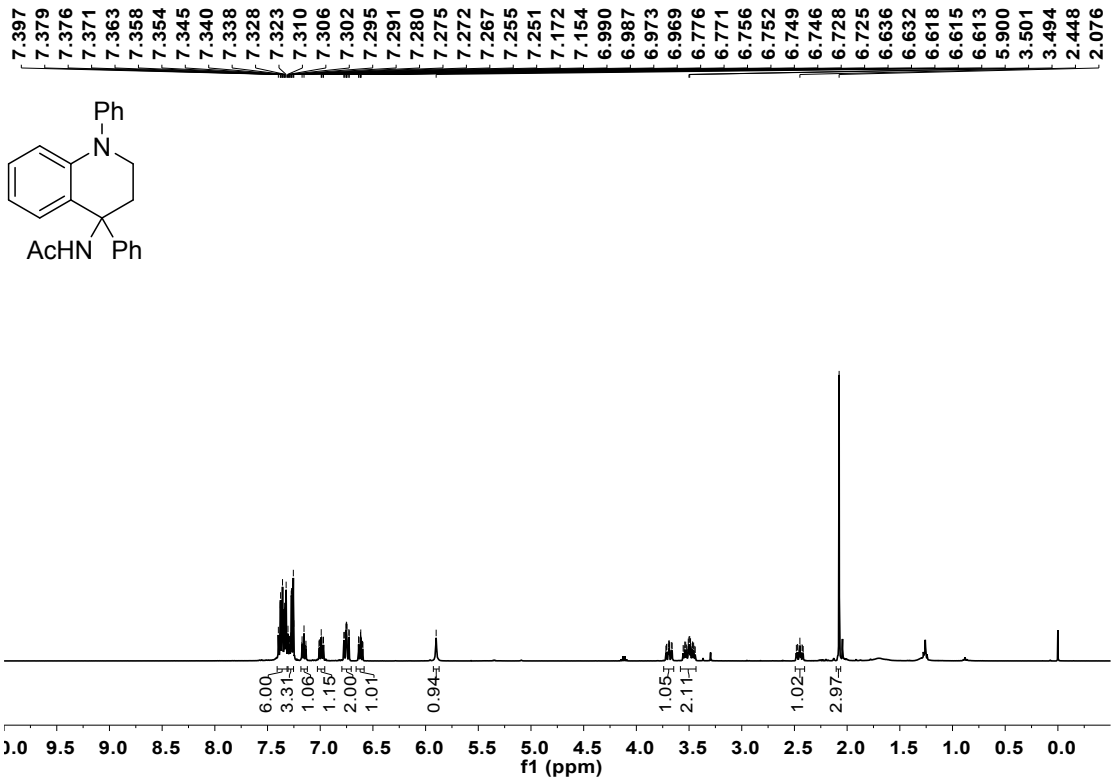


^{13}C NMR

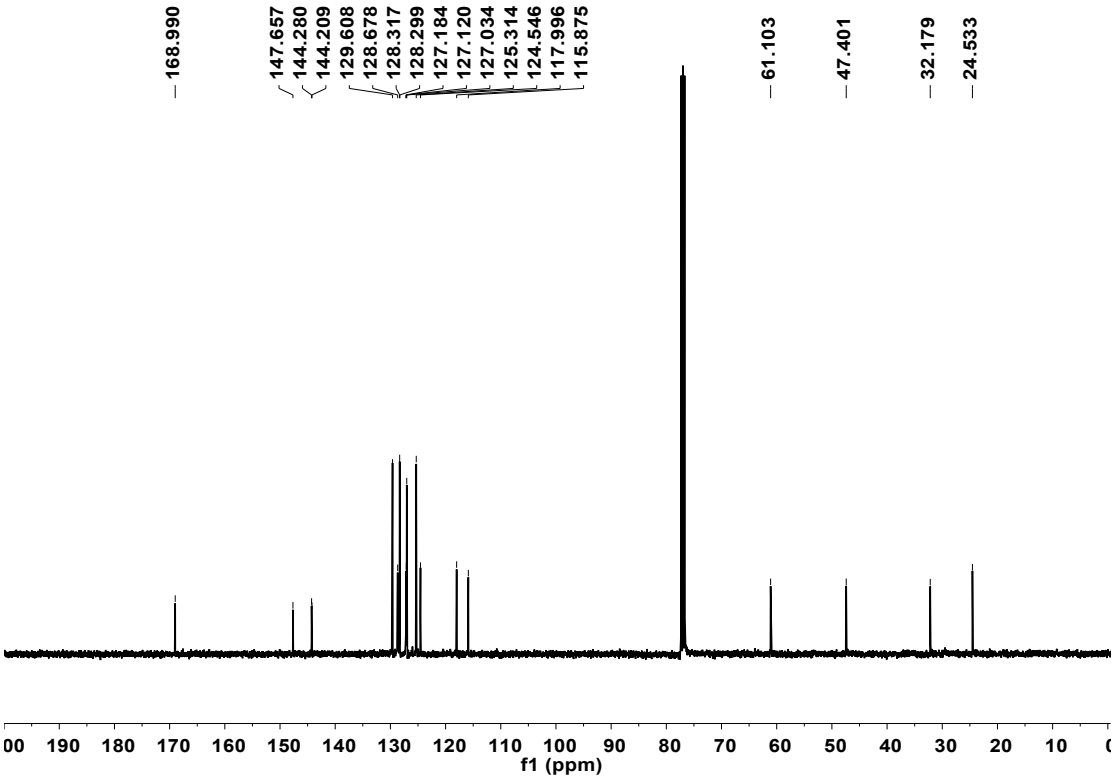


3ma

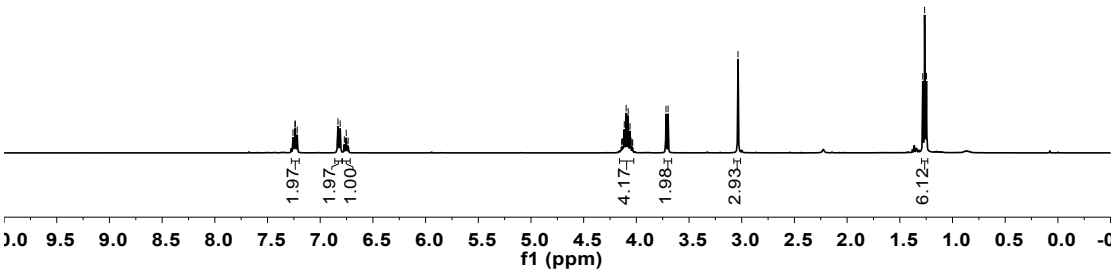
^{1}H NMR



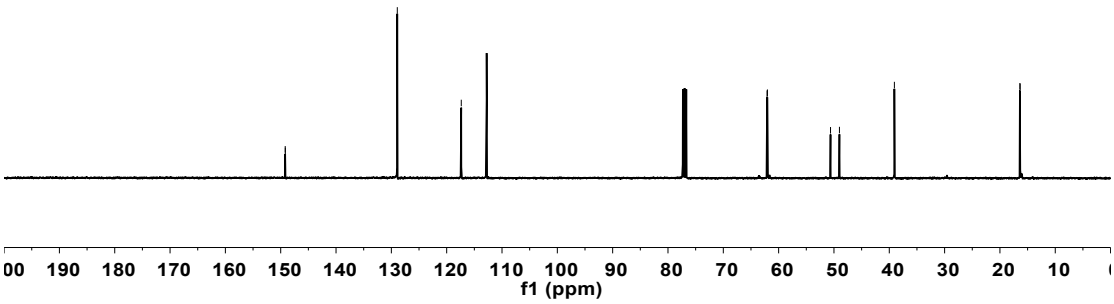
^{13}C NMR



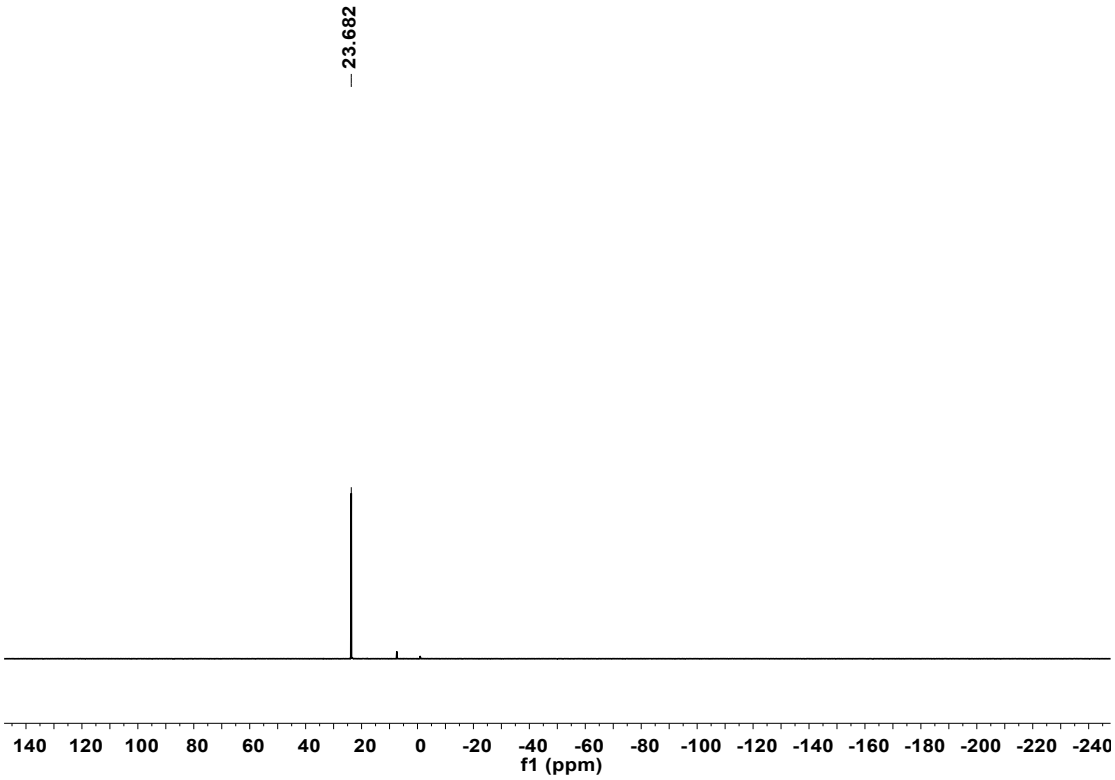
^{1}H NMR



^{13}C NMR



^{31}P NMR


23.682
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240
f1 (ppm)